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FINAL REPORT

RANKINE CYCLE TEST FACILITY STUDY

Technical Data

Edited
By
R. H. WETTACH

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NASw-943

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

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August 1966

Technical Management
NASA - Lewis Research Center
Nuclear Power Technology Branch
Joseph P. Joyce, Technical Manager

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

FOREWORD

Contract NASw-943, Rankine Cycle Test Facility Study, was negotiated between the National Aeronautics and Space Administration Headquarters and the General Electric Company on April 15, 1964. It provided for an analytical study of the facility requirements for testing an advanced liquid metal Rankine cycle energy conversion system. Various parameters of power level, maximum temperature and chamber vacuum level were established by the contract for initial review. One set of conditions was agreed upon at the end of Phase I and a detailed study of these conditions was made.

Mr. Robert H. Wettach was Project Manager for this study. Mr. William W. Shoemaker was the technical engineer responsible for the loop design. Dr. J. W. Semmel, the metallurgical and vacuum engineering consultant, contributed significantly in these areas. Mr. J. L. Markwalter was responsible for instrumentation.

A major portion of the study effort, primarily relating to the vacuum chamber and the supporting buildings and utilities, was subcontracted to the Bechtel Corporation. Mr. Donald Furlong was Program Manager for the Bechtel effort. He was assisted by Mr. David J. Goerz who developed the vacuum and cryogenic systems and by Mr. Kenneth R. Broome.

The Project Manager expresses his appreciation for the guidance of Mr. Joseph P. Joyce, NASA Technical Program Manager.

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ABSTRACT

A preliminary analytical study was performed to determine facility requirements for testing liquid metal Rankine cycle components. The facility was to be capable of testing individual components or a group of components comprising a complete test loop. After an initial survey, the following final conditions were selected for the study:

1. Maximum electrical output - 2 MW (Thermal input - 15 MW)
2. Maximum fluid temperature - 2200°F
3. Three-loop system
   a. Heating loop fluid - Lithium (Li)
   b. Working loop fluid - Potassium (K)
   c. Heat rejection loop fluid - Sodium - Potassium Eutectic (NaK)
4. High temperature containment material to be a refractory material such as columbium alloy.
5. The Space Power Chamber at NASA Lewis Research Center to be modified for the vacuum chamber.

Principal results and conclusions derived from this preliminary analytical study are:

1. Conversion of the Space Power Chamber at NASA-Lewis is feasible for this project and presents a reasonable approach to providing a vacuum chamber for testing a 15-MW (2 MW electrical output), 3-loop Rankine cycle test facility.
2. The vacuum requirements of 5×10^{-9} torr steady state test condition for all condensable gasses and 1×10^{-5} for 300 hours maximum allowable hydrogen pressure during startup are attainable.
3. FS-85 columbium alloy is recommended as the liquid metal containment material.
4. Some development programs are necessary before initiating final design. The most important of these programs is an evaluation of material outgassing so that the calculations prerequisite to selection of materials to meet vacuum level requirements can be accomplished with reasonable assurance.
5. The total time required to design, build and install this facility would be 48 months after selection of loop containment material.

LIST OF SYMBOLS
(Unless otherwise specified in particular equations)

c	Allowable contamination by weight, ppm
d	Density, gm/cm^3
f	Sticking factor
H	Hydrogen gas load, torr-liters
l	Liters
L	Weight, pounds
M	Molecular weight
P	Partial pressure, torr
Q	Throughput, torr-liters per second
s	Second
S	Pumping speed, liters per second
t	Time, hours
T	Temperature, ^oK
w	Wall thickness, inches

I. INTRODUCTION

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The work scope of this contract was divided into three phases. Phase I required a feasibility study and cost analysis of the following parameters:

1. A 30 MW heat source compared to a 50 MW heat source.
2. A 10^{-8} torr vacuum system compared to a 10^{-9} torr vacuum system.
3. A primary loop with lithium as the heating fluid at its optimum temperature compared to sodium as the heating fluid at its optimum temperature.

In addition, Phase I required an investigation of the feasibility of utilizing a single loop to test and develop flight-type components by making provisions for bypassing workhorse components already in the loop.

The following Phase I recommendations were made at a formal presentation to the NASA Lewis Research Center on June 26, 1964.

1. Select a 30 MW heat source.
2. Establish the following vacuum capabilities:
    a) 2×10^{-9} torr for O_2 and N_2
    b) 1×10^{-9} torr for water vapor
    c) 1×10^{-8} torr for H_2
    d) 500,000 Liters/second H_2 pumping speed
3. Utilize a lithium primary heating loop at 2200°F maximum temperature.
4. Do not plan on bypassing components with a valving system but rather plan on welding components into place as needed.

A. Test Facility Description

After considering the Phase I recommendations, the NASA Project Manager established the following facility description for the guidelines of Phases II and III.

1. The test facility shall have the capability of testing full scale Rankine cycle components singly, with the assistance of facility "workhorse" components, or together, to simulate hardware of one Rankine cycle power generator. This capability will not include the reactor and radiator.

Specifically, a capability for development and endurance testing of the following items are required.

a) Turbines
b) Boilers
c) Condensers
d) Turboalternator packages
e) Circulating pumps
f) Condensate pumps

2. The facility shall be designed to achieve a high degree of versatility with a useful life expectancy of 10 years. The facility shall have a design objective of meeting test requirements for a wide range of Rankine cycle power plants from a moderate temperature, 100 kilowatt electric power output conversion system to a very high temperature, 2 megawatt electric power output conversion system.

3. The test facility shall include a 3-loop system comprised of a liquid-lithium heating loop, a two-phase potassium boiling loop, and a NaK heat rejection loop. A vacuum chamber shall house the 3-loop system and provide the required high temperature loop environment. The facility shall have the following characteristics:

a) Location	Lewis Research Center, Cleveland, Ohio
b) Life	10 years of intermittent operation with a minimum of 30,000 hours operation at design temperatures.
c) Thermal input	The thermal input shall be determined by the requirement for a maximum electrical power output of 2 MW.
d) Heating fluid maximum temperature	2200°F
e) High-temperature loop environment	(1) 5×10^{-9} torr as a steady state test condition for all condensable gasses. (2) 1×10^{-5} torr for period of 300 hours, maximum hydrogen pressure during startup.
f) Heat source	Electric

g)	High temperature containment material	Alloys of columbium. Heat rejection loop to be stainless steel.
h)	Heat rejection temperature	1500°F maximum
i)	Heating fluid	Li
j)	Working fluid	K
k)	Heat rejection fluid	NaK

4. The three-loop test system is defined to include facility "workhorse" components designed with present technology. No development components shall be considered during this study. It will be possible to remove "workhorse" components in such a way that development test components can be welded into the loop in place of the facility component. A turbine simulator shall be provided with sufficient space for testing of future turbine and turbine-generator packages. The construction of the weld joints shall be such that the facility component can be placed back into the loop for further testing of some other test component.
5. The Space Power Chambers (SPC), formerly the Altitude Wind Tunnel located at NASA Lewis Research Center, Cleveland, Ohio, shall be considered for modification as a vacuum chamber. Equipment and facilities, which are currently part of the Altitude Wind Tunnel facility shall be used whenever practical. Adjacent buildings shall be considered potentially available for this project.
6. NASA Lewis shall be responsible for providing the electrical power to the DC acyclic generator drive motor.

B. Study Tasks

Phase II was divided into three tasks as defined below:

1. Task 1

Perform a Feasibility Design Study of modifying the Space Power Chambers for the vacuum chamber required to house the three-loop test system defined above. The areas of study shall include:

a) Analyze present NASA facility drawings.

b) Conduct detailed site survey.

c) Conduct a hazards analysis.

d) Evaluate access to site.

e) Evaluate chamber volume to accommodate test loop.

f) Evaluate site and building for possible relocation of equipment, control room and work space.

g) Evaluate the operation of the chamber at a vacuum level in the 10^{-9} torr range.

h) Analyze site utilities such as cooling water, waste disposal, shop air, heating, power, etc.

i) Evaluate structure of present chamber.

2. <u>Task 2</u>

Identify significant technological problem areas associated with the construction, instrumentation and operation of the Test Facility described above which require solution prior to construction of such a Test Facility. These problem areas covered shall include but shall not be limited to the following:

a) The probability, time and expense required to provide D-43 (X-110) and FS-85 advanced columbium base alloys in sizes and shapes for facility fabrication.

b) The minimum required wall cooling compatible with the chamber vacuum defined above (compare refrigerants, their installation and operating costs).

c) Improvement of facility control by utilizing a preheater in the boiling loop.

d) Determine maximum amount of hydrocarbon backstreaming that can be expected from facility vacuum pumps.

3. <u>Task 3</u>

Prepare preliminary design drawings and specifications for the test facility as defined above. A preliminary design as used herein is defined as follows:

a) Instrumentation flow schematic.

b) Drawing showing location of major components and test loop configuration.

c) Requirements and general specifications of major components requiring development which would include but not be limited to:

(1) Electrical heat source
(2) Instrumentation
(3) High temperature liquid metal valves
(4) Liquid metal pumps

Phase III consisted of the development of a construction schedule, setting forth milestones by major task areas which include time required for any research and development necessary to permit construction of the test facility.

C. Summary of Work Effort and Results

1) Considerable attention was given to an analysis of the reactive gases in establishing the vacuum requirements. The outgassing of stainless steel, extrapolated to high temperatures, was considered with respect to potential chamber pumping capacity and limiting the contamination of refractory alloys. These studies lead to the recommendation that the partial pressures be held to 2.5×10^{-9} torr for oxygen, 1.25×10^{-9} torr for nitrogen, and 1.25×10^{-9} torr for carbon containing gases, which results in a 5×10^{-9} torr requirement for reactive gases in general. For this particular facility, it is desirable to maintain a lower water vapor pressure to prevent the reduction of the water vapor by the refractory alloy and the resulting absorption of oxygen. Overall, it is evident that the test facility and the vacuum pumping systems can be properly matched to maintain the contamination of refractory alloys at conservative levels.

2) Fabrication of the refractory alloy to construct the 15 MW facility will require a scale-up program to produce the basic mill products and to join large sections. The heat rejection portion of the liquid metal facility can be constructed of stainless steel. Expectations are that satisfactory performance will be obtained from bimetallic loops with consideration of several precautions. These include the following:

1. Using Type 316 L SS to maintain the carbon content below 300 ppm.
2. Designing the refractory alloy bimetallic loop components and their location in the facility so as to minimize the difficulty and cost of their replacement.

3. Including in-line hot traps of titanium just ahead of the refractory alloy portion of the loop.
4. Restricting low temperature operation (below about 1200°F) where gettering is relatively slow.

3) The test facility consists of three liquid metal loops. The primary loop, a lithium liquid loop in which the lithium is heated to 2200°F, has a columbium alloy as the containment material, and the entire loop is enclosed within the vacuum chamber. The secondary loop is a potassium vapor loop in which the vapor is generated at 2050°F. The containment material is a columbium alloy, and the entire loop is also enclosed within the vacuum chamber. The radiator loop is a NaK liquid loop in which the liquid is heated to 1395°F, the containment material is Type 316 L stainless steel, and the major portion of the loop is outside the vacuum chamber. The loop components, both inside and outside the vacuum chamber, have been designed for long life and a wide range of operating conditions. A workable design of the loop both inside and outside the chamber was prepared.

4) A review was made of existing NASA drawings and a site survey was conducted to evaluate the facility and buildings for use and relocation of equipment, work spaces, and control room. The access to the site and the availability of utilities, as well as restrictive limitations such as building and stack heights, were determined.

Studies were made of the utilization of the wind tunnel and its present pumping systems to provide the necessary vacuum environment and to provide services for the test loops with limited structual modifications or additions. Alternative gearing arrangements were considered for use of the wind tunnel motor as a drive for the primary heater acyclic generators.

Vacuum pumping systems were considered and a combined system using the existing equipment with the addition of ion and cryo-pumps was selected to provide the most practicable pumping capacity consistent with the chamber size and intended service. Refrigeration systems were also evaluated and sized to remove the test loop radiated heat and to provide cold walls for cryo-pumping of 100°K (-279°F) condensable gases. A NaK heat rejection loop radiator and blower system was sized and located. A scrubber was provided to remove atmospheric contaminants from selected spaces as required.

The auxiliary systems were located consistent with the limits imposed by site restrictions. Refrigeration and electric equipment was located to minimize cryogenic piping length and the high-current, low-voltage bus bars.

Modifications to the existing wind tunnel are proposed for providing structural support for an inner chamber, vacuum pumping systems, test loops, and improved chamber access. Penetrations for electrical bus bars, view ports, instrumentation leads, and cryogenic piping were conceptually designed. These and a number of other areas were examined and drawings prepared to delineate the arrangements and concepts selected.

5) The instrumentation for the test facility is broken into three categories; namely, facility, vacuum system and loop. The facility and vacuum system instruments are standard types employed in the process and vacuum industry. The control panels for the facility and vacuum systems have two types of graphic displays. One is a block-diagram presentation showing system operation by illustrating main vessels, equipment, and line layout. The other displays a sequence of events and illuminates each event in turn as the sequence is followed.

The loop instrumentation consists of instruments currently being used in liquid metal systems and a discussion of the problems associated with the specific type of instrument. The position of the loop instrumentation for monitoring and control is shown on a loop schematic drawing. The output of each transducer is amplified to a voltage level in the order of one volt for control, storage and display purposes. The meters are located on two graphic display boards depicting the loop system: one board (which also contains the controls) for loop operation; the other board displays engineering data as required. The recording of data is in a digital and numerical (visual) form. The recorded data are on magnetic tape and punched paper tape. The visual numerical data are printed on adding machine tape. The punched and magnetic tapes are suitable for computer data reduction operations and for data storage. A typical instrumentation data handling system for this facility is included.

D. Conclusions

Results of this study are enumerated below.

1) The design and construction of a 15 Megawatt (thermal) Test Facility for testing Rankine Cycle components as presented in this study is feasible and entirely within the present day state of the art and capability of industry. The design presented in this study represents one solution to providing the facility. There are other equally possible approaches and this approach needs additional engineering to arrive at the optimum system.
2) The total length of time required for this program is 48 months after the selection of materials. It is expected, however, that a development program on the outgassing of materials will require at least 6 months before the selection of materials can be finally established.
3) A steady state vacuum of 5×10^{-9} torr for all condensable gases is required in the vacuum tank during testing. If the test time is 30,000 hours the time pressure product for the condensable gases in the chamber would be:

   2.5×10^{-9} torr x 30,000 hrs. for oxygen
   1.25×10^{-9} torr x 30,000 hrs. for nitrogen
   1.25×10^{-9} torr x 30,000 hrs. for carbon containing gases

   A time pressure product of 1×10^{-5} torr for 300 hrs. maximum is required and attainable in this facility for hydrogen during startup.
4) The liquid metal portion of the study consists of 3 loops. The primary loop is a lithium liquid loop, the secondary loop is a potassium vapor loop and the tertiary loop is a NaK liquid loop. The primary and secondary loops are totally contained within the vacuum chamber and the containment material is FS-85, a columbium alloy. The tertiary loop is located outside of the vacuum chamber and the containment material is Type 316 L stainless steel.
5) The conversion of the Space Power Chamber at NASA Lewis for this project is feasible and presents a reasonable approach to providing a vacuum chamber for testing a 15 MW, 3 loop, Rankine cycle test facility.

6) The major problems associated with this project are as follows:

a) An evaluation of material outgassing so that the calculations leading to the selection of materials to meet the vacuum level requirements can be accomplished with a reasonable amount of assurance. This program would take approximately 12 months.

b) Space Power Chamber structural integrity, high current bus connections, decontamination and leak detecting. This program would take approximately 6 months.

c) Material handling of test components within the chamber. This program would take approximately 6 months.

d) The refractory alloy scale up, property determination and joining. This program would take approximately 21 months.

e) Valve development. This program would take approximately 18 months.

f) Liquid metal pump development. This program would take approximately 6 months.

II. VACUUM ENVIRONMENTAL REQUIREMENTS

II. VACUUM ENVIRONMENTAL REQUIREMENTS

Columbium and tantalum alloys react with oxygen, nitrogen and carbon which are residual gases in high vacuum systems, and the gradual absorption of these constituents by the alloys can substantially reduce their ductility and weldability.(1) This contamination can also proceed to such an extent that the material resistance to corrosion by alkali metals is markedly reduced.(2) In addition to the contamination or gettering action of the alloys, they can also present a significant gas load on the vacuum system by outgassing substantial quantities of hydrogen, particularly during the initial periods of facility operation. The procedures used to define the hydrogen pumping requirements and the vacuum requirements for protecting the test loops from excessive contamination are outlined below.

Loop construction, support structures, and instrumentation also require the use of materials other than refractory alloys, mostly stainless steel. The outgassing of stainless steel, particularly at relatively high temperatures, is considered with respect to limiting the contamination of the refractory alloys. Definitive data on stainless steel outgassing are lacking and assumptions must be made. These are described along with the needs for experimental work to define more accurately stainless outgassing rates, refractory alloy contamination in high vacuum environments, and the influence of contamination on material performance.

A. Reactive Gas Pressure Limitations

The most thoroughly studied case of contamination is the reaction of oxygen with Cb-1Zr. Under NASA Contracts NAS3-1903 and NAS3-4169, performed by the General Electric Company, Cb-1Zr specimens have been exposed to predominately oxygen environments in the 1×10^{-5} to 1×10^{-7} torr pressure range for 236-hour periods at 1800° and 2000°F. A preliminary evaluation of the data by C. A. Barrett of the NASA-Lewis Research Center has led to the following tentative conclusions. At low pressures the extent of contamination is proportional to the number of oxygen molecules which collide with the Cb-1Zr surface. Calculation of the

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1) Data being developed (unavailable at this time) by Westinghouse Electric under contract to NASA.

2) ORNL-3424. Corrosion Mechanisms in Refractory Metal-Alkali Metal Systems. Aug. 30, 1963 by E. E. Hoffman and J. R. DiStafano.

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number of collisions from the kinetic theory of gases reveals that only a small fraction of these collisions result in sticking and absorption of the oxygen by the Cb-1Zr. The probability that an oxygen molecule will stick, which is also called the sticking factor, is believed to be less than 0.10 for the conditions examined.(1) At higher pressures or at relatively high levels of contamination, the rate of contamination can decrease with the exposure time; however, for the amounts of contamination which are of interest here, it is appropriate to assume that the contamination rate remains independent of the exposure time. For the purpose of defining pressure requirements for the test facility, the conclusions from the contamination experiments can be expressed in the following way:

$$P = 1.2 \times 10^{-8} \frac{cwd}{ft} \frac{1}{\sqrt{M/T}} \qquad (2)$$

where:

P is the allowable oxygen partial pressure in torr

c is the allowable contamination in ppm by weight-200 ppm for columbium alloys and 100 ppm for tantalum alloys

f is the sticking factor - 0.10 for oxygen on Cb-1Zr

w is the minimum tube wall or sheet metal thickness in inches - 0.100 inch

d is the density in gm/cm^3 - 8.6 for columbium and 16.6 for tantalum

t is the exposure time in hours - 30,000 for the test facility

M is the molecular weight of the gas - 32 for oxygen

T is the temperature of the ambient gas (gauge) in $^{\circ}$K, about 400°K (260°F)

This leads to an allowable oxygen pressure of 2.5×10^{-9} torr for columbium alloy. (See Table 1.) The pressure can, of course, exceed this value for short periods, such as during start-up; the critical requirement being that the time-partial pressure product for oxygen should not exceed the 30,000-hour-2.5×10^{-9} torr product during the test facility operating life. Table 1 shows the allowable partial pressure for the other reactive gases.

A number of assumptions must be made in applying the existing data to the projected test facility condition. It is assumed that the experimentally observed sticking factor remains unchanged when: (1) the pressure is decreased by about a

1) Private cummunication with Mr. C. A. Barrett of the NASA-Lewis Research Center.

2) Equate the total allowable contaminant in gms/cm^2 to the wt. of contaminant striking a unit surface (Vacuum Technique by Dushman, Page 17, Eq. 6b) and solve for pressure.

TABLE 1

Allowable Partial Pressure (Torr) for Reactive Gases for 30,000 Hours Operation

Containment Material	Density gm/cm^3	Reactive Gases	Mol. Wt.	Contamination PPM	Sticking Factor	Allowable Press. (Torr)
Cb Alloy	8.6	Oxygen	32	200	.1	2.43×10^{-9}
	8.6	Nitrogen	28	100	.1	1.3×10^{-9}
	8.6	Carbon	12	100	.1	2×10^{-9}
Ta Alloys	16.6	Oxygen	32	100	.1	2.35×10^{-9}
	16.6	Nitrogen	28	50	.1	1.25×10^{-9}
	16.6	Carbon	12	50	.1	1.93×10^{-9}

factor of 40, (2) the exposure time is increased by about a factor of 100, (3) the exposure temperatures are outside of the 1800° to 2000°F range, (4) gases other than oxygen are present in substantial quantities, (5) columbium or tantalum alloys other than Cb-1Zr are involved, and (6) that the sticking factor is independent of the various surface conditions and treatments which may be applied to the alloy. No direct experimental evidence has been found to support this assumption and some critical tests should be undertaken. While some of these assumed items undoubtedly influence the sticking factor, it is not probable that they would greatly increase the sticking factor. With regard to the sticking factor in general, it should be noted that the largest possible increase would be a factor of 10 which would lead to a pressure requirement of 2.5×10^{-10} torr. Items (3) to (5) are most deserving of experimental investigation.

The other significant assumptions which are made concern the allowable contamination of the refractory alloys are as follows: 1) An increase of 200 ppm has been selected for columbium alloys and an approximately equivalent increase on an atomic basis has been chosen for tantalum alloys (100 ppm), both averaged across a material thickness of 0.10 inch. (As will be mentioned later, additional allowances must be made for contamination by nitrogen and carbon), 2) The oxygen allowance has been set about as low as considered practical, mainly to assure that the facility can be modified after use by welding under constrained conditions. It is presently assumed that highly strengthened refractory alloy containment materials would be used and that weldability in the contaminated condition is the major factor in establishing permissible levels of contamination, (There has been no systematic investigation of the effect of prior contamination on the weldability of the stronger containment alloys under constraint. The matter should be examined experimentally, and the material performance in this regard should be considered an important criterion for the alloy selection. In general, the allowable contamination level can be increased, possibly by a factor of five, by resorting to more weldable and usually weaker alloys; a major objective, however, is to obtain a reasonable balance between strength and weldability in complex situations). 3) While it is also true that contamination can reduce the corrosion resistance to alkali metals, it is assumed that the containment alloy will have a substantial alloying addition of zirconium or hafnium which will effectively combine with oxygen and provide adequate corrosion resistance at oxygen contamination levels on the order of 1000 ppm. It should be noted that the selection of 100 to 200 ppm oxygen contamination limitations could not be reduced very substantially, inasmuch as a lower, but still comparable, amount of contamination must be expected in the processing and heat treatment of the initial mill products.

As mentioned earlier, the reaction of oxygen with Cb-1Zr has been examined in some detail. In contrast, the contamination by gases containing carbon and nitrogen has received substantially less attention, making it necessary to establish pressure requirements in the following assumed fashion. It is assumed that sticking factors for the various residual gases in the vacuum system will not substantially exceed the value of 0.10 used for oxygen. The effect of contamination by carbon and nitrogen on weldability is assumed to be equal to or more severe than the effect of oxygen. As a matter of safety, the allowable pressures of nitrogen and carbon containing gases are set at 1.25×10^{-9} torr which is more restrictive than the requirement for oxygen by a factor of two. Assuming equal sticking factors of 0.10, the total contamination for 0.100 inch thick material has been set at about 200 ppm oxygen, 100 ppm carbon, and 100 ppm nitrogen for columbium alloys and half of these values for tantalum alloys. Needless to say, the reaction of nitrogen and carbon containing gases with columbium and tantalum alloys and the influence of contamination by these constituents on the material performance requires investigation.

In summary, it is recommended that the partial pressures be held to 2.5×10^{-9} torr for oxygen, 1.25×10^{-9} torr for nitrogen, and 1.25×10^{-9} torr for carbon containing gases, which results in a 5×10^{-9} torr requirement for reactive gases in general. For this particular facility, it is desirable to maintain a lower water vapor pressure, as will become evident in the following discussion.

B. Hydrogen Outgassing From Refractory Alloys

Columbium and tantalum alloys contain hydrogen which can outgas for long periods of time during loop start-up and operation. While this probably does not influence the alloy behavior, it presents an operational problem. The pressure exceeds the customary 10^{-8} to 10^{-9} torr limitations, and although monitoring with a mass spectrometer will show that most of the gas is hydrogen, there is some uncertainty as to whether the appearance of hydrogen merely results from columbium and tantalum outgassing or whether it results from the detrimental reaction of water vapor (and possibly other gases containing hydrogen) with the refractory alloys. (Experiments conducted under NASA Contract NAS3-1903 have shown that when water vapor is admitted to a high vacuum system containing hot Cb-1Zr, the water is reduced, the oxygen is absorbed, and a high hydrogen pressure is observed with a mass spectrometer). It then becomes desirable to provide an adequate pumping capacity to remove the hydrogen which outgasses from the refractory alloys, and it is essential that the vacuum system design and operation is such that substantial amounts of water vapor are not permitted to be present. With the high pumping speeds for water vapor that is provided by the cryopanels, there is little reason for a water vapor reaction producing hydrogen to be confused with hydrogen.

The hydrogen outgassing load from the refractory alloys and the desired pumping speed has been estimated in the following way, using a columbium alloy as the example:

$$H = L\ (c \times 10^{-6})\ 454\ \frac{\text{gms}}{\text{lb}}\ 0.5\ \frac{\text{Moles}}{\text{gm}}\ 22.4\ \frac{\text{liters}}{\text{mole}}\ \frac{338^{\circ}\text{K}}{273^{\circ}\text{K}}\ 760\ \text{torr}$$

$$H = 4.8\ Lc$$

where:

H is the hydrogen gas load in torr-liters at 150°F

L is the weight of columbium alloy in pounds - assume 70,000 pounds for the test facility

c is the hydrogen content of the alloy in ppm by weight - assume 15 ppm for columbium alloys

$$S = \frac{H}{3600tP} = 1.33 \times 10^{-3}\ \frac{Lc}{tP} \quad (1)$$

where:

S is the required pumping speed in liters per second

P is the maximum allowable hydrogen pressure in torr - assume 1×10^{-5} torr

(1) Equation (4 - 7), Modified High Vacuum Engineering by A. E. Barrington, p. 49.

t is the time in hours permitted to outgas the facility during startup - assume 300 hours.

This leads to a pumping speed requirement of about 5 x 10^5 liters per second (Ref. Fig. 1) which is economically practical, as was planned in advance. It should be carefully noted that the time-pressure product of 300 hours-1 x 10^{-5} torr exceeds the allowable time-pressure product for oxygen by a factor of 40, which illustrates the necessity for assuring that the hydrogen outgassing is not confused with the reaction of water vapor or other hydrogen containing gases with the refractory alloy. Increasing the pumping speed for hydrogen by a factor of 40 just to minimize this situation is economically impractical.

If a tantalum alloy were chosen as the example, the total weight of the alloy would remain essentially the same (the allowable stress for a tantalum alloy is higher but the density is higher also) and the hydrogen content of the alloy, in ppm by weight would be 10 ppm, which is two-thirds of that assumed for the columbium alloy. Therefore, the pumping requirement for hydrogen would be two-thirds of that chosen for the columbium alloy.

Little is known of the fine details of hydrogen outgassing and the conditions which arise upon heating large refractory alloy loops to the test temperature. For example, the 300 hour-1 x 10^{-5} torr time-pressure product might have been presented just as well by assuming 3,000 hours and 1 x 10^{-6} torr and then requiring slower initial heating of the test facility. It would be desirable for somewhat academic reasons to know enough about the effects of time, temperature, pressure, concentration, etc. on the outgassing rate to define the hydrogen gas load during system operation. It is recognized that this gas load may remain sufficiently large, even after a nominal start-up period, that it may be the controlling factor with respect to establishing the total pressure in the system throughout most or all of the 30,000-hour operating period. It is doubtful that there is sufficient information available to predict the hydrogen gas load as a function of time for such a complex facility, and as a result it may be impossible to predict the total system pressure after prolonged operation. However, this is not a serious limitation, even if the pressure exceeds the 10^{-9} torr range, provided that there is adequate assurance that the hydrogen does not arise from the reaction of other hydrogen bearing gases with the refractory alloy facility. Water vapor would be the major hydrogen containing gas, but the very high pumping speed for water vapor (7.1×10^7 liters per second) should maintain its pressure at a suitably low value as is illustrated on the following pages.

C. Outgassing From Stainless Steel and Thermal Insulation

Calculation of the gas loads from the vacuum chamber, flanges, and feedthroughs is fairly straightforward, and with the planned pumping capacity a clean, dry, and empty chamber capability of 5×10^{-10} torr is expected.* Also, estimates of the gas load from the stainless steel supporting structure for the 15 MW loop can be made reasonably well at temperatures below about 500°F, and surface areas in excess of 100,000 ft^2 are acceptable.* However, higher operating temperatures and the need to open the system occasionally require an estimate of the gas load from the alkali metal loops and supporting materials to assure that the test facility and pumping capacity are reasonably well matched with respect to avoiding excessive contamination of the refractory alloys. Table 2 shows a summary of outgassing calculations which are discussed in detail below.

Ideally, it would be desirable to know the outgassing rate of several materials, particularly stainless steel at temperatures up to about 1400°F, which would permit a calculation of the partial pressures as a function of facility operating time and temperature conditions. The required data are not available; however, it is possible to estimate lower limits for the amount of material that can be used for facilities construction without presenting an excessive gas load.

The outgassing of stainless steel at elevated temperatures is an important case to consider because it is used for cooling loops in significant quantities at 1200° to 1400°F. See Figures 2 and 3 for typical stainless steel outgassing. At these temperatures, interstitial elements can diffuse from the bulk material to the surface and provide a significant gas load. Carbon is usually the major interstitial element constituent in stainless steel, and it will be assumed that the average carbon concentration is 300 ppm. With a pumping speed of 3.3×10^6 liters per second for carbon containing gases, it can be shown that about 2,500 pounds of stainless steel could supply sufficient carbon to exceed the 30,000-hour-1.25×10^{-9} torr time-pressure product limitation which was set for carbon contamination of the refractory alloys. An examination of the preliminary facility design indicates that about 4,500 pounds of stainless steel have been used above 600°F, mainly in the construction of cooling loops for 1200° to 1400°F service.

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*See Section V

TABLE 2

SUMMARY OF OUTGASSING CALCULATIONS

Source of Gas	Surface Area or Wt. of Material	Outgassing Rate or PPM of Gas	Time Pressure Product Hour-Torr	Pumping Speed Required	Pumping Speed Available	Remarks
H_2 in Cb Alloy	70,000 #	15 PPM	300 x 10^{-5}	5 x 10^5 1/s	5 x 10^5 1/s (Fig. 1)	
H_2 in Stn. Steel	12,000 ft.^{2}	3 x 10^{-10} torr liters Sec^{-1} cm^{-2} (Fig. 3)	3000 x 10^{-8}	360,000 1/s	1300,000 1/s (Fig. 1)	At 500°K & after 100 hrs.
Carbon in Stn. Steel	4,500 # above 600°F	300 PPM	30,000 x 1.25 x 10^{-9}	3.4 x 10^6 1/s	3.3 x 10^6 1/s	
Condensables (CO_2-N_2-O_2-A) in Stn. Steel	12,000 ft.^{2}	3 x 10^{-10} torr liters Sec^{-1} cm^{-2} (Fig. 3)	3000 x 5 x 10^{-9}	6.8 x 10^5 1/s	3.3 x 10^6 1/s	At 500°K & after 100 hours
Water Vapor due to opening chamber	50,000 ft.^{2}	Open 50 times 100 molecular layers of H_2O	30,000 x 1 x 10^{-9}	6.66 x 10^7 1/s	7.1 x 10^7 1/s	3 x 10^{-8} cm spacing
Other Reactive Gases (O_2-N_2-CO_2) due to opening chamber	50,000 ft.^{2}	20 molecular layers of gases	30,000 x 5 x 10^{-9}	2.68 x 10^6 1/s	3.3 x 10^6 1/s	3 x 10^{-8}cm spacing

TABLE 2 (Continued)

H_2 in Cb Alloy

$H = 4.8\ LC$

H = hydrogen gas load in torr-liters at 150°F

L = weight of columbium alloy in lbs; assume 70,000 lbs for the test facility

C = hydrogen content of the alloy in ppm by weight; assume 15 ppm for columbium alloys

$$S = \frac{H}{3600\ tP} = 1.33 \times 10^{-3} \frac{LC}{tP}$$

S = required pumping speed in liters per second

P = maximum allowable H_2 pressure in torr; assume 1×10^{-5} torr

t = time, in hours, permitted to outgas the facility during startup; assume 300 hours

$$S = \frac{1.33 \times 10^{-3} \times 70{,}000 \times 15}{300 \times 1 \times 10^{-5}} = 4.65 \times 10^{5}\ \text{l/s}$$

Figure 1 shows an available pumping speed for hydrogen of approximately 5×10^{5} l/s

H_2 in Stn. Stl.

$$S = \frac{O_r\ A}{P}$$

O_r = outgassing rate for stn. stl. in torr-liters per second - cm^2 - taken as 3×10^{-10} torr - Ll/second - cm^2 from Figure 3 at 500°K (440°F) and after 100 hours

A = surface area of stn. stl; assume 5000 ft^2 of reflective insulation, 5000 ft^2 of pipe and supports and 2000 ft^2 of miscellaneous material = 12,000 ft^2

P = H_2 pressure in torr; assume 1×10^{-8} torr because the partial pressure of H_2 is relatively unimportant (discussion in text) and pumping speed at 1×10^{-8} torr is optimum for system (Figure 2)

$$S = 3 \times 10^{-10} \times 12{,}000 \times 929 = 334{,}000\ \text{l/s}$$

Figure 1 shows an available pumping speed of approximately 1,300,000 l/s at 1×10^{-8} torr.

Carbon in Stn. Stl.

$C = .343\ Lc$

C = carbon gas load in torr-liters at 150°F assuming gas is evolved as carbon monoxide

L = weight of stn. stl. in lbs at a temperature above 600°F; assume 4500 lbs

c = carbon content of the stn. stl. in ppm by weight; assume 300 ppm

$S = \frac{H}{3600\ tP} = .095 \times 10^{-3}\ \frac{LC}{tP}$

tP = time-pressure product - 30,000 hrs x 1.25 x 10^{-9} torr

$$S = \frac{.095 \times 10^{-3} \times 4500 \times 300}{30{,}000 \times 1.25 \times 10^{-9}} = 3.4 \times 10^{6}\ \mathrm{l/s}$$

Figure 4 of the test shows an available pumping speed for condensables of 3.3 x 10^{6} l/s

Condensables (CO_2 - N_2 - O_2 - A) in Stn. Stl.

$S = \frac{O_r\ A}{P}$

O_r = outgassing rate of condensables for stn. stl. in torr-liters per second - cm^2 - taken as 3 x 10^{-10} torr - Ll/second - cm^2 from Figure 3 at 500°K (440°F) after 100 hours

A = surface area - 12,000 ft^2

P = condensable partial pressure - 5 x 10^{-9} torr

$$S = \frac{3 \times 10^{-10} \times 12{,}000 \times 929}{5 \times 10^{-9}} = 6.7 \times 10^{5}\ \mathrm{l/s}$$

Figure 4 shows an available pumping speed for condensables of 3.3 x 10^{6} l/s.

Water Vapor Caused by Opening Chamber

Assumptions:

a. System will be opened 50 times.

b. 100 molecular layers of water will be absorbed on all surfaces.

c. Spacing between molecules will be 3 x 10^{-8} cm.

$$\text{Molecules }/cm^2 = \frac{1}{3 \times 10^{-8}} \times \frac{1}{3 \times 10^{-8}} = .11 \times 10^{16}$$

$$\text{Molecules }/ft^2 = .11 \times 10^{16} \times 927 = 102 \times 10^{16}$$

$$\text{Molecules }/ft^2 - 100\ \text{layers} = 1.02 \times 10^{20}$$

$$\text{Liters }/ft^2 = \frac{1.02 \times 10^{20} \times 22.4}{6.023 \times 10^{23}} = 3.8 \times 10^{-3}$$

$$\text{Torr liters }/ft^2 = 3.8 \times 10^{-3} \times 760 - 2.88$$

$$\text{Torr Ll }/ft^2 - 50\ \text{openings} = 144$$

$$S = \text{gas load per } ft^2 \times \frac{A}{P\ t}$$

A = facility surface area = 50,000 ft^2
P = 1 x 10^{-9} torr
t = 30,000 hours

$$S = \frac{144 \times 50{,}000}{3600 \times 30{,}000 \times 1 \times 10^{-9}} = 6.66 \times 10^7 \ 1/s$$

Figure 4 shows an available pumping speed for water vapor of 7.1 x 10^7 1/s.

Other Reactive Gases (O_2 - N_2 - CO_2) Caused by Opening Chamber

Assumptions:

Same as water vapor above except that 20, rather than 100, molecular layers will be absorbed. Therefore, gas load per $ft^2 = \frac{1}{5} \times 144 = 29$ torr 1/ft^2.

$$S = \text{gas load per ft}^2 \times \frac{A}{Pt}$$

A = facility surface area = 50,000 ft^2

P = 5 x 10^{-9} torr

t = 30,000 hours

$$S = \frac{29 \times 50{,}000}{3600 \times 30{,}000 \times 5 \times 10^{-9}} = 2.68 \times 10^6 \ 1/s$$

Figure 4 of the text shows an available pumping speed for condensables of 3.3 x 10^6 1/s.

While this amount of stainless steel could be reduced substantially by substituting a refractory alloy at increased costs, the similarity between the estimated lower limit and amount used in design identifies a need to understand more clearly the outgassing of stainless steel.

Although, as illustrated by the above estimate, the system has sufficient pumping capacity to handle the outgassing from a limited amount of stainless steel, the restricted use of stainless steel will tend to increase costs, and there will be difficulty in predicting the reactive gas pressure as a function of loop operating conditions. It should be recognized that the outgassing will probably occur more rapidly during the early periods of operation and the pressure limitations would be exceeded substantially for several thousand hours if the maximum use of stainless steel is made. As another example of the influence on cost, in addition to the cooling loops, there are about 5,500 pounds of reflective insulating foil which operate between 500° to 2200°F. Much of this material, below about 1500°F, could be stainless steel instead of refractory alloy if it were not for the desire to limit outgassing (and also to take advantage of the gettering effect of refractory alloy foil at intermediate temperatures). The outgassing values for stainless steel used in Table 2 are based on outgassing rates after 100 hrs. at 500°K (440°F). If the rates are projected to higher temperatures by using appropriate factors from Figure 2 the outgassing becomes prohibitive. As an example, the outgassing rate at 1160°F is a factor of 1×10^6 higher than the rate at 440°F. In order to make the maximum use of stainless steel for high temperature components and to permit a prediction of the partial pressure of reactive gases during the first few thousand hours of operation, it is recommended that the outgassing characteristics of stainless steel be determined at temperatures up to about 1500°F. (See Problem Areas, Section IX).

Excluding surface effects involving the offgassing of adsorbed gases, which will be discussed later, it is not believed that the other materials which are used with the test facility will present problems as severe as stainless steel with respect to the outgassing of absorbed constituents from the bulk material. Alumina and beryllia insulation are used in small quantities and should not present a problem if material of high density and high purity is used properly. It is assumed that refractory alloys, which are used in large quantities, will not outgas from the bulk material (except hydrogen). This is an assumption that is well substantiated for alloys such as Cb-1Zr at temperatures near 2200°F, pressures near 1 x 10^{-8} torr, and times up to 10,000 hours.(1) At higher temperatures and lower pressures, however, there are indications that outgassing can occur. Expectations are that indirect experiments, such as corrosion tests and creep tests conducted in high vacuums at several laboratories, will help clarify this situation. Ultimately, it may be desirable to examine the matter by more direct experiments but, for the present purposes, it is assumed that the refractory alloys will, in general, act as getters rather than sources of outgassing.(1)

In addition to the gas load arising from the bulk of the materials, it is necessary to consider the release of adsorbed gases from material surfaces, particularly with respect to the requirement for occasional opening and modification of the facilities. Evaluation of the preliminary facilities design indicates that the surface area of the loops and supporting equipment will be approximately 50,000 ft^2, excluding the cryogenically cooled portion of the vacuum chamber. This area consists of the following: 2,000 ft^2 of refractory alloy piping and loop components, 43,000 ft^2 of reflective insulation (38,000 ft^2 of Cb-1Zr, for example) and 5,000 ft^2 of stainless steel and small amounts of miscellaneous materials. By making the following assumptions it can be shown that a surface area of 50,000 ft^2 and the available pumping capacity are consistent with the requirements for limiting the contamination of the refractory alloy loop components:

1. The system will be opened 50 times.
2. One hundred molecular layers of water will be absorbed on all surfaces; the spacing between water molecules being 3 x 10^{-8} cm.(2)
3. A total of 20 molecular layers of other reactive gases will be absorbed on all surfaces, also with a spacing of 3 x 10^{-8} cm.(2)

Assumptions 2 and 3 are believed to be conservative and the amount of absorbed gas on a previously evacuated surface could be substantially less. The above assumptions lead to a total gas load of about 150 torr-liters of water vapor per ft^2 of surface area and 30 torr-liters of other reactive gases per ft^2. Setting the allowable time-pressure product at the lower average value of

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(1) Private communication from Mr. David T. Bourgette of O.R.N.L.
(2) Engineering estimate by D. J. Goerz - No known data available.

30,000 hours-1 x 10^{-9} torr and using the pumping speed of 7.1 x 10^{7} liters per second for water vapor, it can be shown that a surface area of about 50,000 ft^2 can be tolerated. Similarily, the 30,000 hour-5 x 10^{-9} torr, time-pressure product requirement for other reactive gases, combined with the appropriate pumping speed of 3.3 x 10^{6} liters per second, indicates that a surface area of about 60,000 ft^2 will be acceptable.

The above estimates illustrate several important features of the system. Primarily, the pumping capacity and the test facility are compatible. The similarity between the area obtained from the preliminary design and that which was estimated as acceptable should not be a cause for great concern because of the conservative nature of the assumptions regarding the limits on refractory alloy contamination and the amount of adsorbed gas. In addition, the fact that much of the adsorbed gas would be released upon heating to moderate temperatures, below the temperature at which refractory alloys are effective getters, has been neglected. This has the effect of allowing the facility to be outgassed at temperatures that will not affect the time-pressure product that has been established. Nevertheless, the requirements to avoid unnecessary increases in surface area and to maintain a clean system are apparent. As was discussed earlier, it is desirable to maintain the water vapor at a particularly low value in order to avoid the possibility of confusing the reaction of water vapor and refractory alloys with the hydrogen outgassing from refractory alloys. In this regard, the above estimates indicate that it should be possible to maintain a time-pressure product for water vapor below 30,000 hours-1 x 10^{-9} torr.

It is apparent that the major source of surface area is the reflective insulation, and any final design will require detailed investigations to minimize costs. There are the conflicting technical requirements to use many layers of insulation to reduce heat losses and to use as few layers as possible to reduce the gas load, with their corresponding influence on cooling costs and vacuum pumping costs. There is also the desire to reduce material costs by using less insulation overall and by using proportionately smaller amounts of refractory alloy and larger amounts of less expensive materials, such as stainless steel, in the cooler regions. Many layers of insulation could be canned to reduce the apparent surface area, but manufacturing costs would increase. It is desirable to vent the insulation to increase the gas conductance to the vacuum chamber pumping surfaces, but without significantly increasing the heat losses. All of these trade-offs influence the gas load and pumping requirements and, ultimately, more detailed investigations will be required to optimize the insulation with respect to cost and technical requirements. (See Problem Areas, Section IX).

III. LOOP AND FACILITY MATERIALS

III. LOOP AND FACILITY MATERIALS

A. Alkali Metals

It is proposed to adhere to alkali metal purification and analytical procedures which are similar to those in general use with smaller test facilities. Examples of such specifications (01-0030-00-B, 01-0033-00-B, 01-0034-00-B, 01-0049-00-A, 01-0050-00A), which are used at General Electric for the purchase and hot trapping of lithium, potassium, and NaK for smaller facilities, are presented in the Appendix. It would be advisable to prepare detailed specifications and procedures for the alkali metal procurement and purification with particular reference to this facility. The referenced specifications deal with quantities of liquid metal up to 200 pounds. The proposed facility requires quantities far in excess of these values. The primary loop requires 2080 lbs. of lithium, is contained in a refractory alloy loop rather than stainless steel, and the lithium is not readily hot trapped for oxygen with zirconium. The secondary loop requires 2450 pounds of potassium and is contained in a refractory alloy loop rather than stainless steel. The radiator loop requires 12,250 pounds of NaK and lends itself better than lithium or potassium to cold trapping for oxygen. The suggested approach for the 15 MW facility primary and secondary loops would be to buy the purest liquid metal obtainable, then after flushing the loops, hot trap and cold trap the liquid metals in the dump tanks provided. The suggested approach for the NaK radiator loop would be the same as the primary and secondary loop except that cold trapping would be done continuously during loop operation. Provisions should be made to sample and analyze the respective alkali metals during loop operation. Impurities should be similar to those mentioned in the specifications listed above and in the Appendix.

It is anticipated that the possible leakage of alkali metals and the need to decontaminate the vacuum chamber and loop hardware may require new procedures in addition to shoveling, distillation, and chemical leaching, with the usual concern to avoid the hydrogen embrittlement of refractory alloys. In particular, the wide distribution of even small amounts of alkali metal over the cryogenically cooled vacuum chamber walls could be troublesome because of the large surface area involved. The current practice with smaller test facilities is to use bakeable vacuum chambers with water cooling coils, which usually makes cleaning easy after small potassium leaks because the potassium accumulates along the cooling coils. However, for cryogenically pumped systems which cannot be baked, it would be desirable to convert the alkali metal to more easily handled compounds by controlled gaseous reactions. For example, conversion of the alkali metal to its carbonate by the controlled addition of water vapor and carbon dioxide should be considered. It is recommended that the kinetics of such reactions be examined experimentally in order to develop additional decontamination procedures specifically for the 15 MW facility. In Section V under Safety Systems, a procedure is outlined for decontamination of the chamber and the components.

B. Refractory Alloys

The candidate materials for construction of the high temperature loops consist primarily of the stronger, weldable, columbium and tantalum alloys which contain an internal getter in the form of a zirconium or hafnium alloying addition. The FS-85, D-43, T-111, and T-222 alloys are representative examples, and the preliminary design study was conducted largely with FS-85 as the typical material. Material specifications for FS-85 are included in the Appendix. Densities and temperature capabilities of the various alloys are listed in Table 3. The long time strength properties which were used tentatively for preliminary design considerations are presented in Figure 5. These estimated properties have been derived from tests conducted for about 1,000 hours or less at various commercial and government laboratories.

Fabrication of the materials to construct the 15 MW facility will require a scale-up program to produce the basic mill products and to join large sections. The scale-up activities were discussed with two refractory alloy producers to determine the requirements for development programs, equipment limitations, anticipated material costs, and the effect on the overall program schedule. A listing of the refractory alloy materials required for construction and insulation of the preliminary facility design is shown in Table 4. In general, the scale-up requirements from experimental quantities to the quantities and sizes shown in Table 4 are not overpowering with respect to the total 15 MW facility program. It is anticipated that the largest ingot diameter required would be 22 inches to make the 36-inch diameter x 9-inch long headers by forging (Table 4).

Accompanying the scale-up work, vacuum annealing equipment should be installed. The largest furnace dimensions would be about 5-foot x 6-foot x 16-foot. Requirements for the 24 and 30-foot long, 24-inch diameter rolled and welded dump tanks (Table 4) should be reduced in design to avoid the requirement for a very large furnace for the post-weld heat treatment.

TABLE 3

ESTIMATED TEMPERATURE CAPABILITY CORRESPONDING TO A MINIMUM* RUPTURE STRESS OF 5,000 PSI IN 10,000 HOURS

Alloy	Temperature Capability, °F	Estimated Alloy Density, lbs/in^3
Cb-1Zr	1700 (+)	0.31
D-43	2200 (-)	0.33
FS-85	2200 (+)	0.38
T-111	2300 (±)	0.61
T-222	2500 (+)	0.61

* The minimum rupture stress was taken as 75% of the average rupture stress values because the data are generally insufficient to define minimum properties.

Nominal Compositions:

D-43: 10W-1Zr-0.1C-bal. Cb
FS-85: 28Ta-10.5W-0.9Zr-bal. Cb
T-111: 8W-2Hf-bal. Ta
T-222: 9.6W-2.4Hf-0.01C-bal. Ta

TABLE 4

15 MW FACILITY REFRACTORY ALLOY REQUIREMENTS
AS DESIGNED WITH THE COLUMBIUM FS-85 ALLOY

Seamless Tubing

6.25 - inch OD x 0.375-inch wall x 5-foot long
6.25 - inch OD x 0.225-inch wall x 202-foot long (total)
6.25 - inch OD x 0.125-inch wall x 289-foot long (total)
2.5 - inch OD x 0.25 -inch wall x 80-foot long (total)
2.5 - inch OD x 0.10 -inch wall x 100-foot long (total)
1.25 - inch OD x 0.060-inch wall x 70-foot long (total)
1.0 - inch OD x 0.065-inch wall x 4940-foot long (total)
1.0 - inch OD x 0.049-inch wall x 7500-foot long (total)
0.375- inch OD x 0.035-inch wall x 8500-foot long (total)

Rolled and Welded Pipe

34-inch OD x 1.0 - inch wall x 20-inch long
32-inch OD x 1.5 - inch wall x 12-foot long
37-inch OD x 1.0 - inch wall x 7.5-foot long, 2 pieces
24-inch OD x 0.25- inch wall x 30-foot long
24-inch OD x 0.25- inch wall x 24-foot long

Elliptical Dished Heads

24-inch OD x 0.25 - inch wall x 6.25-inch height, 6 pieces
37-inch OD x 1.0 - inch wall x 11.25-inch height, 2 pieces
34-inch OD x 1.0 - inch wall x 12.0 -inch height

Plate, Sheet, and Bar

37-inch dia. cylinder x 3.0-inch height, 4 pieces
34-inch dia. cylinder x 3.0-inch height
30-inch dia. cylinder x 3.0-inch height
24-inch dia. cylinder x 3.0-inch height
18-inch dia. cylinder x 3.0-inch height
36-inch dia. cylinder x 9.0-inch height, 2 pieces
30-inch wide x 1.5 - inch thick x 4.5-foot long, 6 pieces
42-inch wide x 0.125- inch thick x 9 -foot long, 2 pieces
12-inch wide x 1.0 - inch thick x 4.5-foot long, 6 pieces
3-inch wide x 0.25 - inch thick x 20 -foot long (total)
18-inch wide x 0.50 - inch thick x 14 -foot long, 2 pieces
12-inch wide x 0.25 - inch thick x 100 -foot long (total)
12-inch wide x 0.50 - inch thick x 100 -foot long (total)
2.5-inch dia. x 18-inch long, 4 pieces
4 -inch dia. x 24-inch long, 2 pieces
6 -inch dia. x 24-inch long
9 -inch dia. x 18-inch long, 4 pieces (casting acceptable)
15 -inch dia. x 24-inch long, 4 pieces (casting acceptable)

TABLE 4 (Continued)

Forgings

6-inch x 12-inch Std. Wt. Pipe Reducer
4-inch Sch. 10 Pipe Cap, 12 pieces
0.75-inch Sch. 5 Pipe Cap, 650 pieces

Wire and Rod

0.040-inch dia. x 5,000-feet long (total)(Cb-1Zr alloy acceptable)
0.063-inch dia. x 20,000-feet long (total)
0.125-inch dia. x 40,000-feet long (total)

Foil and Sheet (Cb-1Zr Alloy Acceptable)

6-inch wide x 0.005-inch thick x 10,000-feet long (total)
24-inch wide x 0.025-inch thick x 2,000-feet long (total)

C. Stainless Steel-Bimetallic Loops

A NaK-filled, Type 316 L SS loop will remove heat from the 15 MW facility. This results in a bimetallic loop and bimetallic joints which result in material mass transfer. Interstitial elements will transfer from Type 316 L SS to the refractory alloy, resulting in contamination levels easily in excess of 1,000 ppm. The refractory alloy ductility will be decreased, and the ability to make welds for repairs and modifications may be greatly reduced. Control of the alkali metal purity becomes particularly difficult and critical. Operation at low temperatures, below the point where the refractory alloy can serve as an effective getter and purify the NaK, could lead to extensive mass transfer of the refractory alloy as a result of the presence of impurities such as oxygen in the NaK.(1) Although no easy or certain remedy has been developed to solve these problems, there are a number of improvements which may arise in the materials area, and there are some loop design and operation precautions which should be taken.

With regard to materials improvements, there is a possibility that the use of an internally gettered stainless steel such as Type 321 may transfer interstitial elements more slowly, and this is being investigated at General Electric under NASA Contract NAS3-6012. Also, it is possible that the interstitial element content of the stainless steel could be reduced to a very low value by heat treatment in a vacuum or in alkali metal with a getter. The development of bimetallic tubing of stainless steel and the refractory alloy, along with suitable joining procedures, could also provide a solution. This would be a rather lengthy and expensive development and, while it may eventually be desirable for flight-type systems, it has not been included as a program specifically for the 15 MW facility.

Assuming that none of the above hypothetical and expensive material developments are brought to completion in the relatively near future, there are several precautions which should be taken to minimize the bimetallic loop problems in the final design of the 15 MW facility. These include the following:

1. Using Type 316 L SS to maintain the carbon content below 300 ppm.

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(1) ORNL-3424. Corrosion Mechanisms in Refractory Metal-Alkali Metal Systems, Aug. 30, 1963 by E. E. Hoffman and J. R. DiStafano

2. Designing the refractory alloy bimetallic loop components and their location in the facility so as to minimize the difficulty and cost of their replacement.
3. Including in-line hot traps of titanium just ahead of the refractory alloy portion of the loop.
4. Restricting low temperature operation (below about 1200°F) where gettering is relatively slow.

With reasonable precautions, expectations are that satisfactory performance should be obtained from the bimetallic loops.

D. Loop Insulation

The loop piping inside of the vacuum chamber must be insulated to prevent excessive heat loss to the cryo walls of the chamber. The insulation will consist of layers of foil, approximately .005 inch thick separated by .040 diameter wire. The foil material will be Cb-1Zr in areas where the temperature is above 1200°F. The lower temperature areas will be wrapped with 316 stainless steel foil provided that the outgassing of the stainless does not become prohibitive. The wire material will be the same as the foil. Table 4 lists the quantity of .005 inch Cb-1Zr foil required. The foil will be spiral wrapped in the opposite direction to the wire to allow a path between the layers of insulation for outgassing.

The loop piping outside of the vacuum chamber will be insulated with a high temperature ceramic fiber covered with a lower temperature mineral fiber and then protected from damage by covering with screen on metal foil.

E. Chamber Material

The Space Power Chamber is constructed of mild steel and in its present condition is not capable of containing a high vacuum due to its surface condition. The chamber will be lined with a 304 L stainless steel liner that will be vacuum tight and contain integral cooling coils through which liquid argon will be circulated to remove heat and pump water vapor. The outer surface of the liner is polished to reduce the emissivity to below 0.1. The inner surface is painted with Parson's optical black lacquer or similar material to improve absorption to an emissivity greater than 0.9. Figure 6 shows the stainless steel superstructure that supports the new inner liner shroud and in turn attaches to the Space Power Chamber.

Helium cryopanels made of 304 stainless steel are suspended within the envelope of the liner and are further shielded from the loops by liquid argon cryopanel shields of 304 L stainless. All surfaces of both cryopanels and shields are electro-polished to obtain an emissivity less than 0.1.

The liner will be an all welded construction which precludes the use of O-ring seals. The flanges on the Space Power Chamber will be sealed with butyl O-rings. Pyrex glass windows are used for all viewing ports. Structural material within the chamber for support of the loop piping will be 304 stainless steel. Connections between the columbium alloy loop material and the stainless steel structural material will be made by bolting, hook and eye, or brazing.

IV. LOOP DESIGN

IV. LOOP DESIGN

The alkali metal loop configuration presented as a result of this study and the accompanying drawings, schematics, and tables evolved from established guidelines of the contract and other assumptions and calculations.

A. Loop Schematic

The loop flow schematic, Figure 7, shows the major components, general flow path and the portions of the loops inside and outside of the chamber. With the requirement of a maximum electrical output of 2 MW, a thermal input of 15 MW was established. Since two 8 MW acyclic generators were available, the thermal input is shown as such on the schematic. A 15 MW thermal input for 2 MW electric output was arrived at in a series of previous studies of Rankine cycle space power plants using lithium as the primary fluid and potassium as a secondary or vaporizing fluid. Overall thermal efficiencies of such power plants based on 2 and 3 loop systems range from 15 to 17 percent.(1)

The maximum temperature of the primary loop was set by the guidelines of the study contract and the upper limit of the containment material with regard to stress, time and temperature. A Δ T of 100°F was chosen as a minimum value across the I^2R heater. Lower Δ T's would cause space power plant reactors to become large or pressure drops and resulting pumping power to become excessive for the established reactor output power. Higher I^2R heater Δ T's are possible with resulting reductions in primary fluid flows.

A secondary loop maximum temperature (turbine inlet temperature) of 2050°F was selected to have a minimum of 50°F Δ T between primary and secondary temperatures to insure 100% quality in a co-current (parallel) flow boiler design. A co-current flow boiler design was chosen because of the higher outlet vapor temperatures that are possible with this design.(2) The turbine exit temperature was chosen as 1420°F. The 1420°F represents 75% of the turbine inlet temperature (expressed in °R) and was evolved from previous Rankine cycle studies of turbine exit temperature versus minimum radiator size for space power systems.(3)

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(1) Powerplant Heat Cycles for Space Vehicles by D.B. Mackay, North American Aviation, Inc. IAS Paper No. 59-104.

(2) Boiler Design Parameters Leading to Lowest Weights in a Space Power System. Part 1, April 7, 1964, By J. Longo, Jr., Space Power & Propulsion Section, General Electric Co., Internal Memorandum.

(3) Thermodynamics of Space Power Plants by G. C. Wesling and H. Brown. Flight Propulsion Laboratory, General Electric Co., R59AGT16.

The secondary loop condensate temperature is sub-cooled 75°F to allow for reasonable pressure drops in the condenser and condensate lines to the pump. The maximum NaK cooling temperature of 1395°F was chosen to give reasonable average condensing ΔT's and to allow for the use of stainless steel in the radiator loop. A minimum ΔT of 150°F for the NaK cooling loop was chosen to prevent excessive NaK flows and resulting pressure drop and to keep condenser sizing reasonable. Higher NaK ΔT's are possible in the facility proposed but may not be consistent with optimum heat rejection for space systems and may affect the mass transport phenomena in a bimetallic system. The temperatures, pressures, and flows shown on Figure 7 represent the maximum design conditions. The primary loop temperature and flow ranges are unlimited below the design values. The secondary loop temperature range is unlimited below the design value but the flow range is limited by the pressure drop associated with the higher specific volume of the vapor at lower temperatures. The radiator loop temperature and flow ranges are again unlimited below the design values.

Figure 7 shows a minimum number of valves, and of the valves that are included, an attempt was made to place them in the cooler locations in the loops. General Electric's experience with liquid metal valves has been that they fail quite often and valve life is inversely proportional to operating temperature. The schematic shows two condensers since a single condenser would be rather large in size and weight and present difficulties in mounting in the space chamber. An auxiliary condensate heater is shown in the secondary loop. This heater will permit variations in the boiler inlet temperature independent of condensing temperatures and reduce thermal stresses and thermal shocking in the boiler. The condensate heater does not boil potassium. The primary and secondary loop dump tanks are shown within the vacuum chamber. These dump tanks could be located outside of the vacuum chamber and be constructed of stainless steel at a reduction in cost, heat load and outgassing within the chamber. The dump tanks are located within the chamber, however, to eliminate bimetallic systems in the primary and secondary loops and to allow for emergency dumping of the loops at temperature.

B. 15 MW Study Design Data

After the loop schematic was constructed and the corresponding loop temperature selected, a data sheet was developed to show flows, pressure drops, etc. The tabulated data are shown in Table 5.

TABLE 5

15 MW STUDY DESIGN DATA

Primary Loop - Lithium - FS-85 Cb Alloy

Heater Out Temp., $^{\circ}$F			2200
Heater In Temp., $^{\circ}$F			2100
Li Flow, lb/sec		Ref. 1	154.4
Li Flow, gpm			2600
Pipe O.D., inches			6.25
Pipe Thickness, inch			.225
Heater and Pipe Vel., f.p.s.			31.8
Heater Length, ft.		Refs. 2, 3	92.8
ΔP/ft, psi			.13
Vel. Hd., psi			2.9
Pump Inlet Press.,psia			20
Pump Outlet Press.,psia			70
Stress, 1% Creep - 30,000 hrs @ 2200°F-(Factor of Safety = 2)			950 ψ
Heat Loss, BTU/hr ft of Pipe (20 Layers)			750
Outer Skin Temp., $^{\circ}$F			600
Dump Tank Dia., inches (O.D.)			24
Dump Tank Thickness, inch			.25
Loop Lithium Volume, ft^3			70
Dump Tank Volume, ft^3	10% H & 15 F.B. (H = Head & F.B. = Freeboard)		87.5
Dump Tank Line Size, OD" x W"			2.5x.1
Length of 6.25 OD x .225 W Pipe, ft			202
Length of 2.5 OD x .1 W Pipe, ft			35
Length of 1.25 OD x .1 W Pipe, ft			40
Lithium Inventory, lbs @ 27 lb/ft^3			2080

Secondary Loop - Potassium - FS-85 Cb Alloy

Boiler Out Temp., $^{\circ}$F	P_{sat} 179 psia	Ref. 4	2050
Turbine Out Temp., $^{\circ}$F	P_{sat} 16.4 psia		1420
Boiler In Temp., $^{\circ}$F	P_{sat} 10.9 psia		1345
K Flow lb/sec		Ref. 4	17.7
K Flow, gpm			190.5
Boiler Out Pipe O.D.,inches			6.25
Boiler Out Pipe Thickness, inch			.375
Boiler Out Vapor Vel., f.p.s.	M= .14	Ref. 4	284
ΔP/ft, psi			.18
Vel. Hd., psi			3.17
Stress, 1% Creep - 30,000 hrs @ 2050°F - (Factor of Safety = 2)			1450 ψ
Ht Loss, BTU/hr ft of Pipe (17 Layers)			750
Outer Skin Temp., $^{\circ}$F			600
Length of 6.25 OD x .375 W Pipe ft			5

TABLE 5
15 MW STUDY DESIGN DATA (Continued)

Condenser Out Pipe O.D., inches		2.5
Condenser Out Pipe Thickness, inch		.1
Condenser Out Vel., f.p.s.		14
Condenser Out Δ P/ft		.12
Condenser Out Vel. Hd		.845
Stress - 1% Creep - 30,000 hrs @ 1500°F - (Factor of Safety = 2)		5000 ψ
Ht Loss - BTU/hr ft of Pipe (6 Layers)		315
Outer Skin Temp., °F		600
Length of 2.50 OD x .1 W Pipe, ft		34
Preheater Pipe O.D., inches		2.5
Preheater Pipe Thickness, inch		.30
Preheater Vel., f.p.s.		21
Preheater Δ P/ft		.27
Preheater Vel. Hd		1.8
Preheater Δ T, °F		850
Preheater Power MW		3
Preheater Length, ft	Ref. 2	14
Heat Loss - BTU/hr ft of Pipe (17 Layers)		315
Outer Skin Temp., °F		600
Length of 2.5 OD x .25 W Pipe ft		80
Turbine Exit Pipe O.D., inches (4 Req'd)		6.25
Turbine Exit Pipe Thickness, inch		.125
Turbine Exit Pipe Vel., f.p.s.		550
Turbine Exit Δ P/ft		.05
Turbine Exit Pipe Mach No.		.33
Turbine Exit Pipe Vel. Hd., psi		1.1
Ht Loss - BTU/hr ft of Pipe (6 Layers)		750
Outer Skin Temp., °F		600
Length of 6.25 OD x .125 W Pipe, ft		32
Pump Inlet Press, psia (minimum)		10.9
Pump Outlet Press, psia		250
Dump Tank Dia. O.D., inches		24
Dump Tank Thickness, inch		.25
Loop Potassium Volume, ft^3		57
Dump Tank Volume, ft^3 10% H & 15% F.B.		71
Dump Tank Line Size - OD" x W"		2.5x.1
Length of Dump Line, ft (2.5 x.1W)		31
Length of 1.25 OD x .1 W Pipe, ft		30
Potassium Inventory, lbs @ 39 lb/ft^3		2450

TABLE 5
15 MW STUDY DESIGN DATA (Continued)

Radiator Loop - NaK (78/22) - Stainless Steel

Condenser Out Temp., °F			1395
Condenser In Temp., °F			1245
NaK Flow, lb/sec		Ref. 5	452
NaK Flow, gpm			4825
Pump Inlet Press, psia			25
Pump Outlet Press, psia			75
Pipe O.D., inches	Cb (2 Req'd)		6.25
Pipe Thickness, inch			.125
Pipe Vel., f.p.s.			27.5
Pipe Δ P/ft, psi			.18
Pipe Vel. Head, psi			3.43
Stress, 1% Creep - 30,000 hrs. @ 1500°F - (Factor of Safety = 2)			5000ψ
Ht Loss, BTU/hr ft of Pipe	(5 Layers)		750
Outer Skin Temp., °F			600
Length of 6.25 OD x .125 W Pipe, ft			257
Pipe O.D., inches	Stainless Steel (2 Req'd)		8.625
Pipe Thickness, inch		Ref. 6	.322
Pipe Vel., f.p.s.			15
Pipe Δ P/ft, psi			.04
Pipe Vel. Head, psi			1.02
Stress Code @ 1500°F		Ref. 6	1500 ψ
Ht Loss, BTU/hr ft of Pipe	(5 Layers)		1040
Outer Skin Temp., °F			600
Length of 8" Pipe (In Vac. Tank) ft			129
Dump Tank Dia. (O.D.) inch			48
Dump Tank Thickness, inch		Ref. 7	.5
Loop NaK Volume, ft^3			357
Dump Tank Volume, ft^3	10% H & 10% F.B.		430
NaK Inventory lbs @ 44 lb/ft^3			17,250

(1) Properties of Inorganic Working Fluids & Coolants for Space Applications Part II Liquid Metals WADC TR 59-598

(2) Deem, H. W., and Matolich, J. Jr., "The Thermal Conductivity and Electrical Resistivity of Liquid Potassium and the Alloy Niobium-1 Zirconium," Report BATT-4673-T6, Battelle Memorial Institute, 1963

(3) Kapelner, S. M., "Electrical Resistivity of Lithium and 46NaK," Report PWAC349, United Aircraft Corp., 1961

(4) Thermodynamic Properties of Potassium Calculated from Experimental Data in the Temperature Range of 1200 to 2700°F. General Electric Co. R63FPD375 by T. A. Phillips and M. E. McCarthy

(5) Liquid Metals Handbook, Sodium NaK Supplement, Atomic Energy Commission and Dept. of the Navy, Washington, D. C. (1955) C. B. Jackson, Editor.

(6) Code for Pressure Piping, ASA B31.1-1955

(7) Unfired Pressure Vessel Code, Section VIII

The reasons for the selection of columbium alloy (FS85) as the material for the piping loops and components are explained in Section III of this report. The respective loop liquid metal flows were the result of the required power output and the selected temperatures shown in Figure 7. The selection of the pipe sizes to accommodate the loop flows was somewhat arbitrary; however, the following conditions were considered:

1. A maximum size was selected that was within the capability of industry to produce by extrusion from existing ingots with the proper area reduction.
2. A minimum number of sizes was selected to minimize tooling costs.
3. The outside diameter of the piping was fixed and the wall thickness permitted to vary with operating pressure. The outside diameter was chosen because field welding will be necessary and such welding will require a field erected welding chamber that will clamp and seal on the OD of the pipe. The smaller number of pipe outside diameters will simplify the welding chamber design.
4. Fluid velocities were maintained that were consistent with reasonable pressure drops.
5. Sizes were selected where possible to permit maximum flexibility for loop expansion.

The pump inlet pressure in the primary and radiator loops was set at a value well above the vapor pressure of the liquid metal at the pump inlet temperature. The pump outlet pressure in the primary and radiator loops was chosen by estimating the pressure losses required to pump the fluids through the piping and components. The pressure losses can be substantiated after completion of the loop layout and conceptual component design. The pump inlet pressure in the secondary loop was taken as potassium vapor pressure at 1345°F. The actual pressure at the pump inlet will have to be above this value to prevent vapor lock. The discharge pressure was set at 250 psia which is equal to the vapor pressure of potassium at 2050°F plus an allowance for line, valve, and boiler pressure drop.

Loop pipe thicknesses were established based on maximum loop pressures, temperatures, and stress values for the alloy chosen. Figure 5 shows estimated average 1% creep stress versus a time temperature parameter for FS-85. With 30,000 hrs. as the operating time at temperature, loop operating temperatures from the schematic, and pump outlet pressures, the pipe wall thicknesses were determined. A design stress for the piping was taken as the 1% creep value at time and temperature with a factor of safety of 2. The pipe thickness of the columbium alloy was determined by setting the tangential hoop stress equal to the design stress. The determination of the pipe thickness for the columbium loop based on the hoop stress and a factor of safety of 2 based on 1% creep is only a first approximation of the final pipe thickness. A much more detailed analysis of the loop piping must be undertaken to establish the operating stresses and the safe design stress. There is no reason to believe that the safe design stress cannot be taken as the 1% creep at time and temperature. The operating stresses would be a combination of all the stresses imposed on the piping, that is, stresses caused by pressure, pipe weight, temperature differences, and discontinuities. The determination of all the loop stresses is best handled by a computer after a final layout is established. Past experience at General Electric has indicated that pipe thicknesses based on hoop stresses equal to one-half the design stress are good approximations. The pipe thickness of the stainless steel pipe outside of the vacuum chamber was determined in accordance with applicable power piping practices.

The primary and auxiliary heater piping lengths were calculated based on input power to the heaters, pipe and fluid resistivity, and acyclic generator current ratings. As mentioned above, the input power to the primary heater was taken as 16 MW rather than 15 MW because of the availability of the higher power with existing generators. The input power to the auxiliary heater was taken as 20% of 15 MW. This is sufficient power to heat the design condensate flow from 1345 to 2050°F.

Piping heat loss per foot of pipe was determined by using loop temperatures radiating to liquid argon cryopanels with radiation shielding of layers of columbium - 1% zirconium foil. The number of layers of foil was determined by setting the outer foil temperature at 600°F. This temperature was chosen to facilitate the outgassing of water vapor from the surfaces of the insulation. Preliminary heat transfer calculations of all the piping and components within the chamber, based on pipe lengths from the layout, show total heat losses to be approximately 3% of the primary heater power. A general heat balance of the loop piping and vacuum chamber appears in Section V, Paragraph D.

C. Component Design

1. Boiler

A conceptual design of the boiler is shown in Figure 8 and the conditions governing the design of this boiler are given in Specification No. S-1. (See Appendix.) The boiler design is, for purpose of this study, a maximum envelope, facility type, workhorse boiler. Average heat fluxes are 50,000 BTU/hr. ft.^{2} With the above heat flux, the temperature differences between primary and secondary fluids can be reduced considerably below the design conditions. The boiler is designed to operate as a horizontal boiler because of the length available in the vacuum chamber and the closer approximation to a zero gravity condition.

2. Simulator

A conceptual design of the turbine simulator is shown in Figure 9. It will be designed to simulate a typical 2 MW electric turbine; conditions governing its design are given in Specification No. S-2. (See Appendix.) Turbine exit quality may be varied by varying the NaK cooling flow to the simulator but the power removal capabilities of the simulator, other than for design conditions, will depend largely on the operating ranges of the other system components. The flow area of the simulator is not variable.

3. Condenser

A conceptual design of the condenser is shown in Figure 10. Conditions governing its design are given in Specification No. S-3. (See Appendix.) The condenser design is, for the purposes of this study, a maximum envelope facility type, workhorse condenser.

Average heat fluxes are 25,000 BTU/hr. ft.^{2}. The length of the condensers may be reduced in the final design due to pressure drop considerations. Two condensers are proposed and shown on the layout due to size and subsequent handling problems in the vacuum chamber. The condensers are designed to operate as horizontal condensers because of the length available in the vacuum chamber and the closer approximation to a zero gravity condition.

4. Pumps

The proposed pumps both inside and outside of the loops are flat linear electromagnetic polyphase induction pumps. The pumps inside of the vacuum chamber will have canned stators and, if possible, all cooling will be accomplished with an inert gas. The inert gas would prevent contamination of the loop piping in the event of a leak into the chamber. Specification No. S-5 (Appendix) lists the flows, heads, and other conditions governing the design of the liquid metal pumps within the chamber. The cooling required for the pumps appears in the general heat balance in Section V, Subsection D.

5. Valves

All liquid metal valves within the vacuum chamber will be remotely operated with argon gas. The flow, size and operating pressures of each valve within the chamber are listed in Specification No. S-4. (See Appendix.) Valves outside of the chamber will be stainless steel, with operators where required.

6. Flowmeters

All flowmeters, both inside and outside of the vacuum chamber, are of the electromagnetic type. The principle of operation of the flowmeter is to induce a magnetic field normal to the flowing fluid and measure the resulting induced voltage caused by the flowing fluid cutting the magnetic field. The conditions governin the design of the flowmeters and the flow range of interest for the flowmeters within the chamber are given in Specification No. S-6. (See Appendix.)

D. Liquid Metal Loops Layout (Inside Vacuum Chamber)

The layout of the liquid metal loops inside of the vacuum chamber is shown in Figure 11. The layout takes the loop schematic (Figure 7), the design data listed in Table 5, and the major components and develops them into a working system within the vacuum chamber. The boiler, condenser and simulator were sized from the conceptual drawings and such items as pumps and flowmeters were sized from overall dimensions established by respective vendors. The chamber envelope, into which the loops were designed, was established by assuming an imaginary working volume inside of the cryopanel shields of the chamber.

The net available workspace established in the Conversion of the Space Power Chamber [See Section V, Subparagraph (B)(4)(e), of this report] is 82 ft. long and has diameters of 16 ft. at one end and 20 ft. at the other. The layout of the liquid metal loops, except for penetrations, was accomplished in a length of 65 ft. and has diameters of 15 ft. at the condenser end and 19 ft. at the primary heater end. The difference between the 82 ft. length of chamber and 65 ft. length of loop is available for growth provided the chamber is not vacuum limited due to loop outgassing area.

The loop layout is based on the following design conditions:

1. The primary and secondary heater terminals are fixed with respect to the chamber.
2. The boiler and inlet and outlet connections of the radiator loop are fixed with respect to the chamber.
3. The turbine simulator and condensers are free to move horizontally away from the boiler during heat up.
4. Pumps, flowmeters, and pipe loops will be suspended from the top of the chamber and be free to swing.
5. The dump tanks will be fixed with respect to the chamber and supported from the base of the chamber.
6. Component cooling lines, sub-system penetrations, actuation lines, and loop instrumentation will be flexible.

The design conditions of fixing the heater terminals and inlet and outlet connections of the radiator loop, from the standpoint of the layout, are a desirable set of conditions because the loop expansion and resulting forces are independent of the equipment outside of the chamber. Unfortunately, neither the new inner vacuum chamber nor the modified outer vacuum chamber is capable of absorbing high localized forces and moments. The seemingly simple task of supporting the dump tanks from the base of the chamber becomes complicated because the inner and outer vacuum chambers will not accept localized loads. Section V, Sub-paragraph (B),(4)(c) describes a method of supporting the weight of the loop piping through longitudinal rails suspended from new box ring girders on the outside of the chamber. Figure 6 shows the location of the rails and girders. The conditions of expansion, reactions, and relative moments of the chambers and piping are a possible problem area that will require further study. (See Section IX.)

The NaK cooling flow pipes, to and from the condensers and simulator, double back toward the boiler before leaving the vacuum chamber. The doubling back of the NaK piping not only serves to bring the fluid to the heat rejection facility located within the wind tunnel loop but could eliminate the thermal expansion problem in the tunnel length direction. With all loops operating at design temperature and the boiler or primary heater fixed, the pipe reactions caused by differential thermal expansion could be reduced by the proper location of the bimetallic joints and by taking advantage of the higher expansion rate of stainless steel over columbium.

An attempt has been made to design considerable flexibility into the piping to accommodate vertical movement. Due to the large weight of pumps and flowmeters (Table 6 shows a loop component weight estimate), it would be advantageous to support these components with swinging rods rather than spring or constant support hangers. A swinging rod approach to hanging components in a temperature changing system is feasible if there is sufficient flexibility in the accompanying piping.

All pipe lengths shown in Table 5 have been scaled from the layout as it appears in Figure 11. The pipe lengths were used for heat transfer calculations, pipe material costs, system weights, outgassing areas and insulation

TABLE 6

Loop Component Weight Estimate

	Component	Weight (lbs)
1)	Primary Heater Electrodes	2,000
2)	Primary Heater	2,000
3)	Boiler	10,000
4)	Primary Piping & Valves	6,000
5)	Surge Tank (Pri.)	2,000
6)	E.M. Pump (Pri.)	5,000
7)	Flow Meter (Pri.)	3,000
8)	Dump Tank (Pri.)	7,000
9)	Insulation & Line Heaters	1,500
10)	Secondary Heater Electrodes	500
11)	Secondary Heater	1,000
12)	Secondary Piping & Valves	4,000
13)	Turbine Simulator	4,000
14)	Head Tank (Sec.)	1,500
15)	E.M. Pump (Sec.)	4,000
16)	Flow Meter (Sec.)	2,000
17)	Dump Tank (Sec.)	5,000
18)	Insulation & Line Heaters	1,000
19)	Condenser (2)	10,000
20)	Radiator Loop Piping Inside Chamber	5,000
21)	Insulation & Line Heaters	3,500
22)	Liquid Metal	9,000
23)	Hangers & Support Structure	29,000
	Total Loop Wt. in Tank	120,000

and installation costs.

E. Radiator Loop Layout (Outside Vacuum Chamber)

The layout of the radiator loop outside of the vacuum chamber is shown in Figure 12. The layout takes the loop schematic (Figure 7), the design data listed in Table 5, the major components, and the available area within the wind tunnel loop and develops them into a working system. The design of the radiator enclosure, blower and motor is included in the Conversion of the Space Power Chamber (See Section V of this report.)

The radiator consists of an upper and lower cylindrical header connected by a series of 1 1/8" OD finned tubes. Cooling air enters through the center of 2 banks of tubes, splits, and flows over each bank of tubes. The NaK flows inside the 1 1/8" OD tube. The cooling air flow is split to balance aerodynamic loading on the radiator. The upper drum of the radiator is assumed to be fixed and all expansion of the piping will be absorbed in expansion loops between the radiator and the vacuum chamber. This design simplifies the design of the air flow through the radiator; however, if pipe reactions become large, the radiator tube bundle may have to float. A more detailed pipe analysis will be required before the final design.

NaK flow from the radiator passes through a single pump before being separated into cooling flows for the simulator and two condensers. The separate flows are controlled and measured before entering the vacuum chamber. A portion of the NaK flow is by-passed around the pump and passed through a cold trap and plugging indicator before being returned to the main stream. The dump tank is located over the pit in the existing pump house floor.

F. Auxiliary Loop Connections (Outside Vacuum Chamber)

The layout of the auxiliary loop connections outside of the vacuum chamber are shown in Figure 13. The auxiliary systems are located west of and adjacent to the vacuum chamber, inside of the existing wind tunnel loop. Grouped into the auxiliary systems are the following:

1. The primary loop cold trapping components. These components consist of an EM pump, flowmeter, and cold trap.
2. The primary loop vapor separators and liquid nitrogen cold trap filters for both the dump tank and surge tank.

3. The secondary loop vapor separator and liquid nitrogen cold trap filter for the dump tank and a cold trap filter for the loop vacuum line.
4. The radiator loop vapor separators and liquid nitrogen cold trap filters for both the dump tank and surge tank.
5. All loop fill and sample lines.
6. Vacuum pumping equipment and argon purge headers for evacuating and purging the various loops.

The auxiliary systems are necessarily grouped in one area to facilitate the piping of services to the systems. Such services as liquid nitrogen, high purity argon, cooling air and vacuum headers can be readily piped to the area chosen.

G. Loop Welding and Leak Checking

The liquid metal loop design cannot be considered complete without a discussion and understanding of the methods of welding and leak checking the liquid metal portion of the facility.

1. Loop Welding

In the detail design of the loops, field welding of the components and pipes will play an important part. A program must be undertaken to develop material joining techniques both in the shop and in the field. These points are covered under Status of Refractory Alloys, Paragraph 6, in Problem Areas, Section IX. The timing for the development of joining techniques of FS-85 is shown in the Development Program Schedule, Figure 45.

The major portion of the welding should be done at the place of fabrication either in an inert chamber or by the electron beam process. Field welding should be kept to a minimum and preferably in straight sections of pipe. A clamp-on type welding chamber will have to be designed to cover the field weld and provide the clean environment required for welding. The cover gas, both inside of the clamp-on chamber and inside of the pipe to be welded will have to be of a purity similar to that required for welding Columbium 1% Zirconium listed in Specification 03-0005-00-A in the Appendix. The type of weld groove and the inspection of the finished weld will be similar to that shown in General Electric Company Specifications 03-0005-00-A and 03-0015-00-A in the Appendix.

2. Leak Checking of the Loop Piping

After the loop components and piping have been welded into the chamber, inspected, and heat treated (if required) the complete piping system should be leak tested. Leak testing should include any pressure tests to determine system integrity but the final test should be a Mass Spectrometer Leak Test similar to Specification 03-0013-00-B in the Appendix. If Mass Spectrometer Leak Testing is conducted on the components and piping sub-assemblies in the fabricating shop, the leak checking in the Space Power Chamber can be minimized to the testing of the field welds only. Each welded joint should be bagged and filled with helium on one side of the joint and the leak detection connected to the other side of the joint.

V. CONVERSION OF SPACE POWER CHAMBER

A. Proposed Conversion General Description

The principal effort in the study was devoted to the definition of means to provide the necessary high-vacuum environment for the test loops. Optimization studies could not be made within the study scope for various systems and arrangements of auxiliaries, existing equipment, or building utilization.

The Space Power Test Facility consists of a vacuum chamber in which refractory alloy components and loops can be tested in a suitable environment with a minimum of atmospheric contamination. The Altitude Wind Tunnel, presently the Space Power Chamber (SPC), is utilized as a vacuum chamber. (See Figure 14.) Available space is in the form of two cylinders joined by the frustrum of a cone. Overall inside length is 99 ft., the inside diameter of the large end is 31 ft. and the inside diameter of the small end is 27 ft.

To attain the required vacuum of 5×10^{-9} torr, an inner chamber is required that has an ultra-high vacuum pumping system and that will contain the test loops. This inner chamber parallels the form of the outer chamber. It has a length of 82 ft. and end diameters of 26 and 22 ft. Constructed of stainless steel plate coil, it is suspended within the outer chamber. Longitudinal rails in the inner chamber support the electrical resistance heaters, test loops, and components. A series of stiffening rings and new pier foundations have been designed to support the weight of the inner chamber, pumping equipment, and test loops.

Vacuum equipment consists of the existing roughing train and the existing diffusion pumps taking suction on the outer chamber. High-vacuum pumping for the inner chamber consists of 20 getter-ion pumps supported by the outer chamber and penetrating through the inner chamber wall.* The inner and outer chambers are roughed with the existing pumping system.

*Figure 1 shows the ability of the pumps to attain the 1×10^{-5} torr pressure with a gas load of 5×10^{5} li/sec. as established in Table 2.

Liquid argon at 95°K (-290°F) is circulated through the inner chamber wall as well as through shielding panels which cover approximately 1300 sq ft of 20°K (-423°F) dense-gas helium cryo-pumping surface. Refrigeration equipment consists of 1200 kw of liquid nitrogen sub-cooler capacity and 3 kw of gaseous helium at 20°K. (-423°F). The argon is sub-cooled by boiling off liquid nitrogen.

Power for the primary heater loop is provided at 60 vdc by two 8 MW acyclic generators(1) driven at 3600 rpm through a speed increasing gear train by the existing 450 rpm, 19,750-hp motor formerly used to drive the wind tunnel fan.

Power for the potassium loop boiler-preheater is provided at 26 volts by two 1.5 MW acyclic generators driven in tandem by a 4,250-hp motor. Primary and preheater electric power is conducted through copper bus bars through four penetrations for each heater in the outer and inner chambers.

Heat rejected from the NaK loop is removed in an air-cooled radiator. A 500-hp motor operates the fan. In the event of fire or leakage of liquid metals, the blower-discharge will be directed to a scrubber before being discharged to the stack.

To provide adequate weather protection for equipment and personnel and space for auxiliary systems and services, the tunnel is enclosed in a new high-bay building. The receipt, handling, preparation, installation and removal of test components is facilitated by an adequate laydown area with overhead crane service. A mezzanine floor is constructed at the level of a new door in the north end of the east tunnel leg. The high-bay building houses liquid nitrogen, argon, and helium systems and vacuum pumping systems. Other auxiliaries for the chamber and for the test loop are arranged below and on either side of the tunnel. The shelter around the existing diffusion and rough pumping systems is removed.

A decontamination room is provided for the cleaning of components after service in the test loop. The NaK radiator dump tank and scrubber are located along the west side of the tunnel leg. The former pump house foundation is

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(1) Bulletin GED-4471 Large Motor & Generator Dept., General Electric Co., Schenectady, N. Y.

retained and the pit used as a sump for contaminated discharge from the scrubber or from the steam ejection system used for chamber cleaning.

A central control room is provided in the former exhauster building for facility and test loop monitoring and control. Local startup is planned for equipment. The Facility and its system are described in greater detail in the following sections.

B. Civil Structural Design

1. Existing Facilities

The Altitude Wind Tunnel (See Figure 14) at the Lewis Research Center is shown on the plot plan in Figure 15. It is an outdoor structure surrounded on three sides by buildings housing associated equipment; the Icing Tunnel is on the fourth. A pump house for circulating cooling water occupies part of the inside of the tunnel loop. The east leg of the tunnel has previously been converted into a vacuum chamber by the addition of bulkheads at the east ends of the north and south legs, an access dome on top, and a vacuum pump house and pumping equipment below. Capable of vacuums up to 1×10^{-5} torr, the chamber has been used for environmental testing of the Centaur Stage.

The north, east, and south legs of the tunnel, the adjacent Exhauster Building,Pump House, and the east end of the Office and Shop Building were indicated at the site inspection visit of October 30, 1964 as being available for the proposed conversion.

Access is available to the east leg of the tunnel from the north by Ames Road and Durand Road from the south. Yard space is available at the south end of the leg. The large outdoor substation to the south occupies about four times the space required by a modern substation of equal capacity and could be replaced if the need justified the considerable cost. It can supply about 20,000 KW more than the wind-tunnel drive motor requires.

There are adequate steam and water supply systems and stormwater and sanitary drainage. Underground cable and pipe tunnels permit ready access for repairs and modifications to main services.

Extensive exhauster, refrigeration, and cooling water circulation capacity is available from wind tunnel mechanical systems now surplus to requirements. Utilization of some of this equipment is discussed in Mechanical Systems of this section.

The 20,000-hp wind tunnel fan motor and its supporting control and auxiliary equipment are available for conversion to a prime mover to generate dc power for loop heating as described later in this subsection and in Electrical, Subsection E.

Supporting facilities and services available from NASA-Lewis include administrative and engineering office space, receiving and storage warehouses, plant engineering and maintenance facilities, workshops, emergency equipment, parking lots and cafeteria.

2. Site Layout

The original requirements for operation as a wind tunnel resulted in a layout of equipment and service buildings clustered around the tunnel as closely as possible. The only allowance for access to the outside of the tunnel was for maintenance purposes and there was no need for protection from the weather. As can be seen from later paragraphs, layout requirements of a facility for full scale testing of Advanced Rankine Systems are numerous and complex. The majority of these can be met by the conversion layout shown in Figure 15.

a) Site Layout Requirements

Requirements include the following:

(1) Access to the chamber should permit convenient transfer of loop components and piping from an

assembly and preparation area. This area should be about 5000 sq ft and be accessible by truck.

(2) A component decontamination area of about 1000 sq ft should be readily accessible from the chamber acess door and yet separable from the assembly area.

(3) Heat rejection equipment must be located outside the chamber, as close as possible to the NaK loop, and yet separated from all other equipment and operations.

(4) Vacuum system, cryogenic pumping, and chamber cooling equipment must be either mounted in, on, or as close to the chamber as possible.

(5) Power supply equipment for the loop heaters must be located as close as possible to the loop connection points in order to reduce the length of costly bus bars to a minimum.

(6) The control room should be close to the chamber and all facility equipment.

(7) Decontamination of the chamber under vacuum requires a five-stage steam ejector connected to it and discharging to a sump.

(8) A water wash scrubber is required to decontaminate exhaust from all areas where liquid metal may be exposed to oxidation.

(9) Storage of 560,000 gallons of liquid nitrogen, a 4-day supply, as close to the chamber cooling equipment as possible.

(10) Truck transfer points for receiving liquid nitrogen must adjoin the storage tanks.

b) Alternate Layouts

The only feasible locations for access doors to the chamber that will permit convenient installation and removal of loop component assemblies are the north and south ends of the east leg of the tunnel. Use of the existing dome access door in the top of the tunnel would require both vertical and horizontal transfer of components, a much higher crane lift and building height, and would prevent any possibility of removing completed test loops.

Since access door locations are possible at either end of the chamber, and adequate space for assembly and preparation areas is available at each, two layouts are feasible.

Operationally, there will be very little difference between access at the north or south ends of the chamber. An access door into the north end of the chamber would have a clear diameter of 22 ft, which is more than adequate; a door into the south end, 26 ft. A south access would require the additional cost of building a power supply at the north end and relocating the existing substation and replacing it with modern equipment. With north end access, part of the existing Office and Shop Building has to be demolished but this has a replacement value of less than $50,000 compared to more than $200,000 for the higher cost of electrical systems with the south access.

c) Description of Selected Layout

The layout selected uses an access to the chamber from the north with a high-bay building housing the assembly and preparation area next to this end. Approximately 20 ft of the Office and Shop Building has to be demolished to provide sufficient space for the construction of the Space Power Building.

The Decontamination Building is located in the angle of the Office and Shop Building immediately west of the Space

Power Building so that components withdrawn from the chamber can be readily transferred by dolly through automatic fire doors from the Space Power Building.

The Heat Rejection Facility is located within the wind tunnel loop in a lean-to building immediately west of the Space Power Building. This location has poor access but is suitable for housing the NaK piping,air blast heat exchanger, and NaK auxiliaries.

The chamber vacuum pumping equipment and associated cryogenic systems can all be located alongside and below the chamber, or, in the case of ion pumps, mounted on the chamber. These systems require protection from the weather and, since they cannot be given normal aisle space for installation and maintenance, require overhead crane service.

The existing wind-tunnel fan motor is planned for conversion to drive the acyclic generators required to provide the loop heater power supply. The motor is presently located on the centerline of the tunnel about 30 ft above ground level, at the south end of the Exhauster Building. At this height, there is not enough space between the motor and the tunnel to install the gear drive and generators and so it is planned to lower the motor to ground level where the same control system and switchgear can be used and where there is sufficient space for the generators. The centerline of heater connections is about 12 ft north of the motor-generator centerline which reduces bus lengths practically to a minimum.

The control room is located within the existing Exhauster Building on the first floor. Sufficient space is available here for present and future control and instrumentation requirements together with locations for the addition of future office space and for housing of mechanical and electrical utility equipment. Sleeping quarters for test personnel could be located in the exhauster building either on the mezzanine above the wind tunnel drive motor switch gear or on the first floor in part of the area shown as potential office in Figure 16. Some additional toilet facilities may be needed which could be located adjacent to the existing toilets on the first floor level or immediately above them on the mezzanine.

The steam ejector is located at the south end of the Heat Rejection Facility convenient to the chamber and uses the wall of the building for structural support.

The scrubber is placed in the center of the tunnel loop where it can be fed conveniently from the Decontamination Building and Heat Rejection Facility and discharge into the existing pit under the north leg of the tunnel.

Storage tanks for liquid nitrogen, liquid argon, and gaseous helium are shown in the yard space to the south of the Altitude Wind Tunnel where they can be filled from tank trucks backing up to racks on the south wall of the Space Power Building and where supply lines are relatively short to points of use.

d) Future Expansion

No significant allowance appears to exist for future expansion either in power rating or test loop accommodation at this facility. It would appear that the physical and economic limit has been reached with the 15-MW test loop presently proposed and that additional facility requirements will have to be met elsewhere.

3. Buildings

The existing layout and the requirements of the proposed facility determined which of the buildings can be used without modification, which required conversion and which have to be demolished to make way for new construction. The size and types of construction of the buildings proposed for the project are shown in Figures 16 through 19. A detailed description of each building is in later paragraphs. The types of construction proposed for new buildings are selected on the basis of fire resistive rating, cost, and compatibility with existing materials. Durability and need for maintenance is equivalent to existing construction.

Construction access is very restricted especially inside the wind tunnel loop and it is expected that space will be needed for material storage and crane erection outside the building areas during construction.

a) Demolition of Existing Buildings and Removal of Equipment

The east end of the existing Office and Shop Building interferes with construction of the high-bay assembly area in the new Space Power Building and approximately 20 ft, or an area of 1700 sq ft, will have to be demolished. A new wall can be built either before or immediately following demolition to minimize interference with activity in the remainder of the building.

The Pump House within the wind tunnel loop is inadequate for housing the proposed heat rejection equipment and the roof level is too high for mounting a radiator. Its location, however, is the only suitable place to install the heat rejection equipment so this building must be demolished. The floor and pit can be reused but all existing pumping and control equipment should be removed and stored.

The heat exchanger located beneath the north leg of the wind tunnel will interface with duct work connecting the scrubber to the Decontamination Building. This heat exchanger and its piping and valves should be placed in storage. The pit under the exchanger will be used as a reservoir for the scrubber.

All outdoor exhaust lines and support frames within the area to be occupied by the Space Power Building must be removed. Reinstalling systems required for existing operations is not included within the scope of this study or cost estimate.

The Centaur test program is expected to be out of the tunnel prior to start of the demolition work. It is assumed that all equipment will be removed from the chamber, that the dome cover will be left unsealed, and that the skid-mounted nitrogen tanks used for the program will be removed.

The existing Vacuum Equipment Building under the east leg of the tunnel will no longer be required for weather protection after completion of the new Space Power Building and since it interferes with equipment access, it is proposed that the building be demolished.

The present location of the tunnel drive motor (its centerline is about 30 ft above the ground) is unsuitable for conversion to a generator drive so it is proposed to support it on cribbing, demolish the support piers, build a new foundation, and lower it to the ground level for reinstallation.

b) Utilization and Conversion of Existing Buildings

(1) Converting the Exhauster Building to a Control Room

The Exhauster Building has been stripped of mechanical equipment and pits in the floor filled to provide a flush surface. The south end of this building contains the wind-tunnel drive motor and associated control auxiliaries and switchgear. Minimum toilet facilities for men and women are located at the south end on the first floor. The structure is in sound condition;

it has brick walls, wood roof trusses, a wood roof deck, and a wood mezzanine floor at the south end.

It is proposed to convert part of this building into a control room. A raised computer-type floor is proposed to allow easy rerouting of cables as test and data acquisition needs change. A suspended acoustic ceiling will contain lights and air-conditioning diffusers. A separate air system supplied underfloor and exhausted overhead will cool electronic equipment racks. Steel stud and plaster board walls will complete the enclosure. Existing window openings require filling to 4-hour fire resistive standards and two class "A" fire doors are indicated to provide separation from areas where liquid metals are handled.

An area at the north end of the building is shown partitioned off to form an equipment room for mechanical and electrical utility equipment.

(2) Utilization of Office and Shop Building

The high-bay area of the Office and Shop Building is proposed for use as a Receiving and Storage Area in support of the project. The 10-ton crane has a hook height of approximately 50 ft over a floor area of about 40 ft by 50 ft.

The two-story office area at the east end of this building has a total area of approximately 4500 sq ft after allowing for the area that has to be demolished. This includes toilet facilities for about 40 men and 20 women.

c) New Construction

(1) Space Power Building

This building is to provide weather protection for the Space Power Chamber, Assembly Area, Clean Room, and associated equipment. It also provides support for the 10-ton overhead bridge crane, lighting, ventilation, and space heating equipment.

Foundations for this building will probably require underpinning of some existing footings in the adjacent Exhauster Building and Office and Shop Building and some existing underground utility lines will have to be rerouted.

Trenches with removable covers are shown for new pipelines and cable runs between mechanical and electrical equipment and the chamber.

The floor slab is depressed for an area of 1,800 sq ft in front of the chamber access door and a special "grate" floor built over it. Liquid metals may be inadvertently spilled in this area. This flooring will prevent spread of spilled liquids and will suppress their burning by limiting the oxygen needed for combustion. A mezzanine floor with a concrete deck provides working space at an elevation of 17 ft 6 in. above the first floor which is just below the lower edge of the chamber access door. This floor is 33 ft. wide and about 80 ft long and has a designed live load of 150 psf. Main support beams and columns are designed to allow erection of support frames for test loop rail extensions if removal of test loops from the chamber becomes practical. Two steel stairways provide access to the first floor and a fire door connects the mezzanine to the second floor office area.

Walls adjacent to other facilities are used as fire separation walls to contain fires and their fumes. Other walls are insulated to lower the cost of heating the building. Buildings constructed along Ames Road are designed to match the appearance of adjacent buildings.

Steel rollup doors provide access for equipment at each end of the building and automatic sliding fire doors protect openings to the Decontamination and Control Room.

The roof, supported by steel purlins, tapered steel beams, and steel columns, consists of builtup composition roofing on rigid insulation and steel decking. Crane-rail support girders have three long span sections to bridge the north and south tunnel legs and the wind tunnel drive motor house.

The height of the building is limited to 65 ft to conform to present FAA regulations governing obstructions to aircraft in the vicinity of the airport. As can be seen from Figures 18 and 19, the bridge crane is unable to pass the dome on top of the chamber or to lift any large object out of the chamber through the opening under the dome. This limitation prevents crane service to equipment handled through the south door and maintenance of equipment located south of the dome. It is recommended that permission be sought for an increase in height of this building to at least 75 ft to enable the dome to be removed and more than 75 ft if long objects are likely to be handled through the dome opening.

One 10-ton overhead electric bridge-crane with pendant control for standard industrial service is required for handling loop components outside the chamber and for installation and maintenance of facility equipment in the Space Power Building.

Steam unit heaters supported from column brackets will provide $70^{\circ}F \pm 5^{\circ}$ air in the winter with 10 minute air changes induced by power roof ventilators. No cooling or humidity control is proposed. The safety exhaust system is described in Subsection V(C).

General lighting is provided by mercury vapor lights mounted under the roof to maintain a level of 25 ft-c on the floor. Fluorescent lighting under the mezzanine provides 50 ft-c for assembly of components. Local lighting for special equipment can be provided.

Sprinklers are not installed in this building since water increases the burning rate of liquid metals. Portable fire fighting equipment and breathing apparatus are expected to be provided as part of the operating equipment inventory.

The assembly area is located at the north end of the Space Power Building at the first floor and mezzanine levels.

The first floor area is about 60 ft. by 75 ft. long, the east half of which has bridge crane service and direct access through the main door for receiving and dispatching equipment. This area is intended for preparation and sub-assembly of major components such as boilers and turbine simulators.

The west half of the first floor area has headroom limited by the mezzanine to about 15 ft. clear. This area is intended for preparation and sub-assembly of small components for piping, wiring and instrumentation systems. A portion of this area,20 ft. by 30 ft. at the north end,would be a suitable location for the clean room. Fresh air intake and exhaust discharge ducts could be located in the north wall without interference with the overhead bridge crane and the air conditioning units could be mounted on the mezzanine immediately above the clean room.

The mezzanine portion of the assembly area is located at approximately the same elevation as the floor of the inner test chamber and is intended for final assembly of test articles prior to insertion in the chamber. The bridge crane handles components from the first floor to the mezzanine. The test articles may then be built up while suspended from trolleys running on extensions of the overhead supporting rails in the chamber. Sections of the test article can then be transferred into the chamber or withdrawn on conclusion of the test.

The Clean Room would be lined with plaster on steel lath and studs on walls and ceiling. An air lock with slight positive pressure inside the room would prevent contamination due to door opening. The floor would have a vinyl tile surface with all corners coved. Lighting of up to 100 f.c. intensity at bench height would be through sealed translucent ceiling panels. Air conditioning would maintain temperature at $72^\circ \pm 1^\circ F$ with humidity at 40% + 5%. Filters would remove airborne particulate matter to whatever standards of cleanliness are required for up to 20 changes per hour and up to 100% make-up air depending on the nature and hazard of any materials likely to be released in the room atmosphere.

(2) Decontamination Facility

The function of this building is to house component-decontamination operations. After disassembly from test loops, components and piping retain a film of liquid metal on inside surfaces and may contain substantial amounts trapped in cavities that cannot be drained at operating attitudes. Prior to reuse, liquid metal contamination must be removed by reaction or melting, cleaning, washing, and drying. The sequence is explained in Safety Systems, Subsection V(C).

The building, about 27 ft by 40 ft, is located in the angle of the existing Office and Shop Building on the west side of the Space Power Building. The site is unoccupied except for a small substation which has to be relocated. Openings in the existing walls have to be filled to maintain a 4-hour fire separation and the doorway in the new brick wall to the Space Power Building requires automatic fire doors. The south wall and roof will be asbestos cement sheeting on a steel frame. The roof must be high enough to support a 10-ton overhead bridge crane that can lower a 14-ft object into an enclosed decontamination booth. The floor is covered with a steel grating and perforated plate.

Two booths are shown. (See Figs. 16, 17 & 18.) One is for decontaminating major components. It is completely enclosed and is equipped for heating, purging, washing, and drying by remote control. The other is an open-front booth designed for a high face-velocity exhaust system to decontaminate pipe and small components having surface films of alkali metal. Both boots are exhausted directly to the scrubber, and the room is ventilated at a rate of up to two air changes a minute. Each booth has connections for steam, water, compressed air, gaseous argon and carbon dioxide, and electricity. A pit under the work stand in the component booth contains sand in a removable bucket for absorption of metals melted out of components.

Building services include mercury vapor lights to provide 25 ft-c at floor level. Radiant heaters and steam coils in supply ventilating ducts maintain comfortable conditions despite high rates of airflow in the ventilating systems.

(3) Heat Rejection Facility

This building contains the NaK radiator, dump tank, piping and loop auxiliaries which, together with the air blower and motor, form the Heat Rejection System. It also houses the induced-draft fan for the scrubber and the five-stage steam ejector for chamber of decontamination. Normally, the building will not be occupied continuously.

The building is located inside the wind tunnel loop on the site of the Pump House that has to be demolished. Approximately 40 ft square by 50 ft high at the eaves, it is a steel frame building with cement asbestos roof and wall sheeting. The first floor slab extends the existing Pump House floor and pit which is retained as a catch pit for NaK spillage.

A concrete mezzanine supports the NaK radiator and piping loops and provides overhead protection to personnel servicing auxiliary systems on the first floor.

Two steel stairways serve the mezzanine and a lifting well provides equipment access with a 5-ton monorail hoist suspended from the roof structure.

The 500-hp blower and drive motor for the NaK radiator are mounted on a foundation pad at ground level. This blower provides 329,000 pounds per hour of air to the radiator. The 160-hp fan for the scrubber is located on the mezzanine. The NaK dump tank is located over the existing pit which now requires a steel grating cover. The stack is self-supporting as an extension of the radiator enclosure. The steam ejector is supported by the south wall columns and wall framing.

Ventilation is normally provided by power roof vents but, in the event of NaK leakage, smoke detectors initiate closure of roof vents and smoke is exhausted through the scrubber. No space heater is provided although power outlets will permit the use of portable electric heaters for maintenance workers during winter shutdowns.

Mercury vapor lights at roof level provide general illumination with incandescent lighting below the mezzanine.

(4) Space Power Chamber

(a) Condition as Modified for the Centaur Program

As modified for the Centaur test program, the east leg of the former Altitude Wind Tunnel is capable of maintaining a vacuum of 5 x 10^{-5} torr and has supported internal loads of about 13,000 lb from the Centaur vehicle. The addition of the dome access door amounted to about 160,000 lb extra dead load to be supported by the chamber, ring supports, piers and foundations.

An access door, 15-ft clear internal diameter, was provided in the bulkhead installed in the north leg of the tunnel. The inside surface of the chamber has been painted with Rustoleum and the outside with aluminum paint. Ten diffusion pumps have been mounted in pairs along the bottom surface of the chamber. The chamber appears to be in good condition and a review of the calculations and drawings indicates a suitable design was made for the Centaur modifications.

(b) Space Power Chamber Requirements for Testing Advanced Rankine Systems

A vacuum of 5×10^{-9} torr is required to minimize contamination of loop components. The estimated total weight of loop components and liquid metals in operating condition is 60 tons. Table 6 shows the estimated loop weights. The loop components will be distributed through an overall length of about 60 ft. and can be contained within a cone of 15-ft diameter at the north end and 19-ft diameter at the south end. It is preferable to suspend the test loops to allow for thermal movement. Access is required at one end of the chamber for installation and removal of loop components.

A cooling system is required to dispose of an estimated 800 kw of energy radiated from the test loops to the enclosing surface. (See General Heat Balance, Table 8, Subsection V(D).

View ports are provided for visual observation of the following four points of interest:

- Lithium heater connections
- Potassium boiler
- Turbine simulator
- Boiler pre-heater connections

(c) Selection of Design Method for Chamber Modification

Vacuum and Cooling Requirements. The high vacuum requirement of 5×10^{-9} torr is not economically attainable in a mild steel chamber due to excessive outgassing. With the restricted surface area available for mounting pumps and cryopanels and the need for providing as much pumping capacity as possible, it is of vital importance to reduce outgassing from the chamber walls to a minimum.

It is, therefore, proposed to use a double-walled chamber with the inner wall serving as the cooling shroud as well as the non-structural membrane separating the inner high vacuum chamber from the outer intermediate vacuum of 5×10^{-5} torr. The inner wall is formed of stainless steel plate coil with liquid argon circulated through the passages to absorb the heat radiation.

Load Carrying Requirements. It is assumed that the existing chamber forming the outer wall is structrually capable of withstanding the vacuum loading and of carrying the existing dead loads. Additional loading of 60 tons from the test loops and supports, plus about 50 tons from the shroud and internal cryopanels and shields, and 25 tons from Ion Pumps is considered too great for the existing chamber structure to carry without strengthening, especially since the Centaur modification added some 80 tons without any alterations to piers and foundations. Any safety margin that may have existed is now probably gone.

New supports could be provided independent of the existing chamber by isolating pier penetrations with bellows to permit relative thermal and stress movements. It is preferable, however, for both test loop and shroud loads to be carried in suspension from above rather than to impose considerable restraint on these elements from bending in columns supporting them from below or by adding the complication of sliding or rolling supports.

The method selected is to add two new box ring girders to the outside of the existing chamber with new piers and foundations to support them on the ground. Loads from the test loop and shroud are suspended from the new box rings and the two existing corner box rings. Slide bearings between new box rings and piers permit thermal and stress movements in the chamber relative to the ground. The new rings, one of which carries nearly all the load of the dome, relieve a large part of the existing load currently carried by the corner rings so it does not appear necessary to strengthen them. Figure 20 shows a load diagram of the chamber. A complete stress analysis of the chamber modifications under combined vacuum and equipment loading should be made prior to preparation of working drawings.

(d) Chamber Modifications

Modifications to the existing chamber, shown in perspective in Figure 6, are as follows:

New Box Ring Girders and Supports. Two box-section ring girders are welded to the outside of the chamber centered on existing stiffeners. One is a complete circular ring; the other is located at the dome and is in the form of a U with a horizontal beam closing the top legs and crossing the dome opening. Both rings are supported on concrete cantilever piers by brackets aligned tangentially to the rings. Sliding plate bearings, similar to existing Altitude Chamber bearings, permit horizontal movement in any direction relative to the piers. The piers are supported by a new combined footing in the shape of a rectangle.

The rings carry point loads from the test article support rails and from suspenders carrying the shroud and are designed to assume their tributary share of the existing chamber weight.

Doorway at North End of Chamber. A new circular doorway is required at the north end on the centerline of the chamber for test component access. The largest size that can be installed without cutting into the existing corner support ring girder is 22-ft clear diameter. A short cylindrical transition section must be joined to the tunnel to provide the frame support necessary for the new door. The stiffeners and plate in the area of the transition have to be strengthened considerably to compensate for the portions of both cylinders that are missing at the transition.

The door itself is designed to be removed by the overhead bridge crane rather than to open on hinges. This will enable it to be taken away from the vicinity of the chamber access opening and stored where it will not interfere with operations. It is designed as a concave inward tension membrane for minimum weight and cost and to project less into the work area when closed. A circular port, 7 ft. in diameter, is located in the center of the main door for personnel access after the main door has been closed.

The inside surface of the existing mild-steel chamber requires sand blasting to remove the Rustoleum paint finish and a treatment with glass beads to achieve a reflective surface preserved by a silicone finish equivalent to Dow-Corning DC 705. Surface emissivity should be 0.2 or less with the above treatment and was assumed as such in the calculation of chamber heat loads. See Table 8 of Subsection V(D).

Installation and maintenance access is needed to the space between inner and outer chamber walls. The majority of this space will be about 2 ft. 6 in. wide with reductions at stiffening rings and beams to a minimum of 1 ft. 10 in. Brackets will be located to support portable scaffolding and ladders. The existing door in the north leg of the tunnel can be used for access to this space.

(e) Shroud Design

The shroud or inner chamber, shown in Figure 6, is an optically dense and vacuum-tight membrane separating the high vacuum and intermediate vacuum zones. It is designed to withstand a 10 inch water column pressure differential; flap-type safety valves operate automatically in the event of pressure buildup in either direction. The other main function of the shroud is to act as a cooled wall to absorb heat radiated from test loops. The shroud is not required to simulate a "space" environment. Eighty-two ft long, it varies from 22 ft to 26 ft diameter inside.

The shroud panels, fabricated from stainless steel type 304 L as a single embossed plate coil, present a clean inside surface and permit circulation of liquid argon through embossed channels on the outside. The outer surfaces require electro-polishing to reduce emissivity below 0.1. The inner surface is blackened to improve absorption to an emissivity greater than 0.9. Typical paints for emissivities greater than 0.9 would be Parson's Optical Black Lacquer(1) or "Black Velvet".(2) The shroud panels are prefabricated in quadrants of a length suitable for manufacture and shipping. They will be installed inside the chamber to a frame consisting of circular ribs at approximately 10 ft. on center with main longitudinal beams at mid height and secondary longitudinal stiffeners top and bottom. The secondary longitudinals also serve as supply and return headers for liquid argon.

(1) Eppley Laboratory, Newport, R. I.

(2) 3-M Co., St. Paul, Minn.

The main longitudinal beams are supported by hangers from proposed chamber stiffening girders and from bridle beams which transfer the load to the center of the existing corner stiffening girders.

Each end closure is a flat surface made of stainless steel plate coil similar to the cylindrical walls. The north end closure is removable to the full 22-ft diameter. A 7-ft port in the center for personnel access matches the port in the outer chamber door.

Helium cryopanels are formed of double-embossed stainless steel plate coil and are supported from the shroud stiffener rings by insulated brackets (Fig. 6). The brackets are continued through the cryopanels to support shields which protect the cryopanels from loop radiation. The cryopanels shields are formed of stainless steel single-embossed plate coil. All surfaces of both cryopanels and shields are electro-polished to obtain an emissivity less than 0.1.

The spacing of cryopanels and shields is designed for efficient operation with minimum encroachment on test-article workspace. The net available workspace is 82 ft long and has diameters of 16 ft minimum and 20 ft maximum. The operating temperature will be from 85 (-305°F) to 115°K (-252°F). The outgassing rate will be less than 10^{-11} torr liters/sec/CM^2 at 100°K (-280°F), Figure 2, which is two orders of magnatude less than the rate for stainless steel at room temperature and therefore can be neglected. All permanent penetrations in the shroud are welded. Openable joints are flanged and bolted.

(f) Other Modifications

Penetrations through both chamber and shroud are required for ion pumps, power feedthroughs, liquid metal pipes, control and instrumentation lines, and viewports. A schedule of penetrations is shown in Table 7.

Table 7

SPACE POWER CHAMBER PENETRATION SCHEDULE

TYPE OF PENETRATION			SIZE & LOCATION				
SERVICE	TYPE	FIG. NO.	Outer Chamber	Inner Chamber	Individual Conductors or Tubes	No. per Group or Connectors	No. of Penet Groups
LIQUID METAL:			All Liquid Metal has Schedule 40 Pipe & Bellows:				
NaK Loop	Vacuum & Foil Insulated Pipe	--	8"	8"	6	1	6
Dump Tank Vap. Sep.	Vacuum & Foil Insulated Pipe	--	2"	2"	1	1	1
Sec. Sample & Fill	Vacuum & Foil Insulated Pipe	--	1"	1"	2	1	2
Pri. Sample & Fill	Vacuum & Foil Insulated Pipe	--	1"	1"	2	1	2
Primary Cold Trap	Vacuum & Foil Insulated Pipe	--	2"	2"	2	1	2
Primary Dump Tank Vapor Sep.	Vacuum & Foil Insulated Pipe	--	2"	2"	1	1	1
Pri. & Sec. Vac. Connect.	Vacuum & Foil Insulated Pipe	--	2"	2"	2	1	2
ELECTRICAL							
Pri. Heater Conn.	Copper Bar 150,000 amps	41	15" OD Bellows	15" OD Bellows	4	1	4
Sec. Heater Conn.	Copper Bar 57,000 amps	41	9" OD Bellows	9" OD Bellows	4	1	4
Line Heaters & Lighting Circuits	20 amp Calrod or Serpentine Htrs.	23	--	--	300	30	10/3
Primary Pump Power	523 kw, 440 v, 3 phase	23	--	--	3	3	1
Secondary Pump Power	104 kw, 440 v, 3 phase	23	--	--	3	3	1
INSTRUMENTATION							
Loop Thermocouple Wires	30 MIL	24	--	--	1250	37	34/3
Facility Thermocouple Wires	30 MIL	24	--	--	150	25	6/3
Liquid Level Gage Lead	--	6	1/8" OD Tube	1/8" OD Tube	10	5	2
Vacuum Gage Connections	--	6	2" Tube	2" Tube	10	1	10

Table 7 (Cont'd)

SPACE POWER CHAMBER
PENETRATION SCHEDULE
(CONTD.)

TYPE OF PENETRATION			SIZE & LOCATION				
SERVICE	TYPE	FIG. NO.	Outer Chamber	Inner Chamber	Individual Conductors or tubes	No. per Group or Connector	No. of Penet Groups
ACCESS AND VIEWPORTS							
Equipment Access Door, existing	Dome Top, east leg	20	22' ID	--	1	1	1
Equipment Access Door, existing	West Side, east leg	--	15' ID	--	1	1	1
Equipment Access Door, new	North End, east leg	20	22' ID	22' ID	1	1	1
Personnel Access Door, new	North End, east leg	20	7' ID	7' ID	1	1	1
Viewports, Existing	West Side, east leg	--	18"	--	6	1	6
Viewports, new	East Side, east leg	22	12" ID	4"	4	1	4
VACUUM PUMPS							
Diffusion Pumps	CVC-PMC 50,000 Lit/Sec	--	36" D Flange	--	10	1	10
Ion Pumps	Ultek 50,000 Lit/Sec	21	36" D Flange	36" D Bellows	20	1	20
GAS AND LIQUID SERVICES							
Argon primary pump cooling	SS Pipe	--	4" ID	4" ID	2	1	2
Argon secondary pump cooling	SS Pipe	6	2" ID	2" ID	2	1	2
Argon-valve actuation gas	Bulkhead tube connector	6	1/2" OD tube	1/2" OD tube	12	6	2
Argon Cover Gas	SS Pipe	6	2" ID	2" ID	4	1	1
Liquid Argon	SS Pipe	--	4" ID	4" ID	16	4-6	3
Dense Gas Helium	SS Pipe	6	2" ID	2" ID	6	2	3
Chamber Ventilation	MS Pipe	--	24" ID	24" ID	1	1	1
Chamber Reaction Gas	SS Pipe	--	6" ID	6" ID	1	1	1
Chamber Decontamination and Exhaust Gas	MS Pipe	--	40" D Flange	40" D Bellows	1	1	1
Helium for welding	Bulkhead tube connector	6	1/2" OD	1/2" OD	3	1	3

Vacuum-tight connections are required in the outer chamber wall since it is subject to most of the 14.7 psia pressure differential. Since the wall operates at ambient temperature conditions, organic gasket materials can be used. The shroud wall operates at 85°K (-305°F) to 115°K (-252°F) which precludes use of O-ring seals. Wherever possible welded joints are used with bolted flange connections for removable parts.

Allowance must be made for relative movement between the ground, the outer chamber, the shroud, and test loops due to temperature changes and stress variations. A detailed study is required to identify the maximum relative movements that can develop between these four elements under varying operating and shutdown conditions.

Gas. Figure 21 shows a group of 6 gas penetrations in a single flange bolted to the outer wall. An O-ring gasket, bulkhead-type connectors, and flare fittings are used and an expansion loop provides for relative movement. Several good O-ring materials are available. Butyl rubber is adequate for room temperature or reduced temperature. The loading on the seals is critical and should be in the range of 150-500 pounds per linear inch if ultra-high vacuum is necessary. For this application, 50 pounds per linear inch is adequate. Neoprene is adequate at 10^{-5} torr. For high temperature (above 100°F) use Viton A. The tubes are continuous through the inner wall and have a sealweld to an insulating thimble.

Ion Pumps. Details of an ion pump penetration are shown in Figure 22. A complete description of this item is given in Mechanical Systems, Subsection V(D).

View Ports. A typical view port penetration designed for maximum angle of vision is shown in Figure 23. The glass window in the outer wall is sealed with an O-ring in a flanged connection. A lens sealed to a cone extension of the shroud provides the optimum spacing for wide angle vision. The seal is made by joining Corning 7052 glass to Kovar. This window is made by standard well known techniques and could be procured without special attention. Four such view ports are provided.

Medium Current. Connection for 110-v and 480-v power circuits are shown in Figure 24 grouped in a typical penetration. Applicable to currents up to about 500 amps in each circuit, as many terminations as possible (to a maximum of about 200) should be located at each penetration in the space available between stiffeners. An O-ring joint provides a hermetic seal between the outer chamber and the bolted flange carrying plastic-sleeve insulated connectors. Flexible cables connect terminals at inner and outer walls. Ceramic materials provide thermal and electrical insulation at the inner wall. Brazed joints seal each junction.

Thermocouple and Instrumentation. Figure 25 illustrates the plug-in type of connectors selected for the thermocouple and instrumentation penetrations. Connectors and plugs are available for up to 37 terminations each and up to 200 connectors can be located in each plate. Use of this type of connector will avoid the need for soldering connections in the annular space between the inner and outer walls. O-ring seals maintain the vacuum at the outer wall and at the inner wall, and a welded joint connects the mounting plate to the shroud.

(5) Materials and Equipment Handling and Storage

(a) Receiving and Storage of Liquid Nitrogen

Bulk deliveries of liquid nitrogen by truck are anticipated in 3000 gal loads. The maximum design boil-off rate of 5400 gph, Table 8, Section V(D), requires a 4-day supply of 560,000 gallons allowing some spare capacity. This amount can be contained in two cylindrical tanks 27 ft in diameter by 65 ft high. Double-wall construction is used, the inner of stainless steel and the outer of mild steel, with perlite insulation in the cavity. A vacuum is drawn on the cavity to minimize boil-off. It is assumed that the gas supplier will furnish storage tanks as required.

(c) Receiving and Storage of Loop Components

Components that can be used immediately are received within the Space Power Building and unloaded by the 10-ton overhead bridge crane. Items that are not required immediately can be unloaded in the high-bay of the existing Office and Shop Building by the 10-ton bridge crane.

(d) Handling of Loop Components during Preparation for Installation

The 10-ton bridge crane in the Space Power Building is designed to handle all components of the test loops during preparation and checkout before installation. Special workstands and dollies will be required to support components after removal from crates. Handling capability under the mezzanines is restricted to movable dollies although hoists may be mounted from main beams if required. Hoists and dollies are not included in the estimate.

(e) Removal of Loop Components for Decontamination

After withdrawal from the chamber, the overhead bridge crane transfers components to a dolly that moves them into the Decontamination Building. Another 10-ton crane in this building lifts them into the booth for decontamination.

(6) Utilities

The following maximum estimates are based on preliminary equipment selections and tentative operational procedures.

(a) Water Distribution System

Process requirements include make-up water for the scrubber system and cooling water for mechanical systems. Domestic needs are not expected to surpass present usage. Estimated process demand is 300 gpm. No fire-fighting requirements exists for water.

(b) Industrial Waste System

Discharge will vary up to a maximum of approximately 600 gpm when the scrubber reservoir, 50,000-gal capacity, is emptied. Prior to discharge, the alkaline solutions will require neutralization.

(c) Sanitary Waste System

No increase is expected over existing discharge.

(d) Storm-Water Drainage System

No increase is expected over existing discharge. Minor changes in collection piping will be made to suit new downspout locations.

(e) Steam and Condensate Systems

Saturated steam at 100 psig has been assumed for space heating and steam ejector operation with demand estimated as follows:

- Space heating, 3500 lb/hr
- Steam ejector, 4500 lb/hr for a maximum of 8 hr

Steam required for start-up of the NaK radiator (about 2000 lb/hr) is less than required for the steam ejector. They will not be operated simultaneously.

(f) Compressed Air

Air is needed for remote valve operation, air tool operation, and general cleaning. Estimated demand is 500 scfm @ 100 psig.

(g) Electric Power

Power is needed for loop heating, vacuum and cryogenic system operation, utility systems and lighting. Assuming that all systems will be operated simultaneously for short periods, the maximum demand will equal the connected load. From an average load standpoint, a diversity factor of approximately 75% can be used. A one-line diagram is shown in Figure 26.

Existing switchgear installed for the wind tunnel drive motor and vacuum pumping equipment is assumed to remain. New transformers and switchgear are provided for all other loads.

A detailed breakdown is given in Subsection V(E) to support the following summary:

Existing Wind Tunnel Drive Motor	17,500 kva
Existing Vacuum Pump Equipment	500 kva
Total Existing	18,000 kva
New Load	7,000 kva
Overall Total	25,000 kva

C. Safety Systems

The general approach to facility systems safety during testing of Advanced Rankine Systems is to prevent the development of hazardous conditions by using redundant equipment and "fail-safe" design, to monitor conditions with devices that actuate automatic safety systems where necessary, and to contain and suppress any malfunction in order to limit its effects. A hazardous condition includes loop system integrity being adversely affected by temperature and pressure.

The most critical system is the electrical power supply. A failure in the Lewis Research Center powerline, transformers, or switch gear would cause the Space Power Chamber vacuum to decay. If the vacuum decayed faster than the loop temperature decayed, a portion of the allowable time-pressure product of the refractory alloy would be used up. An emergency power system is therefore provided. Redundancy is also found in various mechanical systems.

The safety systems in this proposal include emergency power, monitoring, purge, ventilation, scrubber, and fire containment and suppression.

1. Emergency Power System

This system provides emergency electricity to equipment, such as the chamber vacuum system, which must not stop while a test is in progress. This electricity will be supplied by a 1600-kw, 2000-kva, 4160-volt, 3-phase, synchronous generator that is engine driven, spark ignited, and uses natural gas.

This emergency system starts automatically upon failure of the 4160 main supply to the facility. The existing and proposed vacuum systems then restart automatically and power is supplied to selected instrumentation and controls; other systems require manual restart.

A transfer switch is provided so that the existing diffusion pump vacuum pumping system can be transferred automatically to the new 4160-volt supply system. If there is a failure in this new 4160-volt main supply, the emergency generator will also supply power to the existing vacuum pumping system through the transfer switch. See Figure 26 for a one-line diagram of the system. The primary and secondary heaters and pumps would not be operated during the power outrage.

The following systems will continue to operate in the event of a power failure.

a) Vacuum system, including existing diffusion, backing, and roughing pumps, and the proposed getter-ion vacuum pumps.
b) Ventilation system
c) Argon system, at reduced capacity
d) Helium system, at reduced capacity
e) Selected instrumentation and controls
f) Emergency lighting
g) Auxiliary argon system for valve actuation and pump cooling

Significant effort will be required, during the final design of this facility, to evaluate all the ramifications connected with a total power failure. Each control component of each subsystem should be designed to fail safe based on the remainder of the loop continuing to operate, if at all possible, because complete shut downs are costly and time consuming. If this design philosophy is followed for each subsystem component, a complete power failure may not initiate the proper or desired action. Therefore, it is felt that most of the instrumentation and controls should receive emergency power, both gas and electrical, to allow for a controlled action by the operators of the facility. The present thinking, for the loop in the event of a total power failure, is as follows:

a) Keep the vacuum and cryopumping systems in operation.
b) Allow the heaters, NaK cooler blower, and EM pumps to stop.
c) Allow the liquid metals to freeze in their respective loops or at least cool to a point where they could be dumped without shocking the system.

The above system will use a minimum of auxiliary power.

2. Monitoring

a) Vacuum Chamber Leak Detection

Liquid metal leaks from the piping and components into the vacuum chamber may be difficult to detect. Liquid metal gages, as described in Subparagraph VI (B)(4)(e), in the various loops appear to offer the most immediately available and reliable method of detecting large leaks in the lithium, potassium, and NaK loops. It is unlikely that vacuum gages in the chamber will detect small liquid metal leaks due to rapid freezing on the cryopanels. No commercial equipment is available that can detect small leaks under the proposed operating conditions. One answer might be a system which incorporates a remotely controlled infrared sensitive TV camera and a viewing screen for operator observation located in the central control room. A leak would show as a change in radiation intensity against a stable background. See Possible Problem Areas, Subparagraphs IX (B) (9).

b) Building Leak Detection

Liquid metal leaks outside the chamber are detected by a (Pyr-A-Larm) system that reacts to fire and smoke. The system has two ionization chambers and a cold-cathode gas discharge tube. One chamber is open and exposed to the atmosphere requiring monitoring; the other is sealed and is used as a reference. Each contains minute sources of alpha-emitting material which ionizes the air in the chambers making it electrically conductive. Normally, the chambers are electrically balanced. When fire or smoke occurs, the electrical conductivity of the open chamber decreases due to the ionized particles in the smoke. The resulting voltage across the chamber drops, the voltage on the starter electrode of the gas discharge tube increases, the tube fires, and an alarm is sounded.

3. Chamber Decontamination and Purge System

If a liquid metal leak inside the chamber is detected, this procedure will be followed:

a) Cut-off loop power, dump loop, and flood loop with Argon
b) Cool down loop and shut down chamber high-vacuum pumping systems
c) Startup steam ejection system
d) Purge chamber with CO_2 and water vapor
e) Repressurize chamber with air after liquid metal has been carbonated
f) Repair leak
g) Clean chamber with water
h) Dry

The purge system proposed for decontaminating the chamber (Figure 27) entails the controlled addition of carbon dioxide and water vapor to react with loop metals condensed on the internal chamber surfaces. The reaction products formed are solids such as sodium carbonate and noncondensables such as hydrogen. This procedure, carried out at moderate temperature and vacuum, controls the effects of reaction between the metals and oxidants. The reaction rate can be controlled by predetermined combinations of chamber temperature and vacuum. It is possible that the upper pressure of the reaction products may be high enough to allow for gradual vaporization of the materials from the chamber surfaces without going above the materials triple-point, thereby preventing the formation of undesirable reactive or corrosive liquid-solid mixtures. An experimental program is necessary to verify the effectiveness of this method.

It is undesirable to pump these gases and metallic fumes through the mechanical pumping system as the potential of damage to the system exists from condensation, contamination and further reactions from heat and pressure. The chamber will be evacuated during these decontamination and purging operations by a five-stage, barometric condensing, steam-jet vacuum pump. Pump blank-off pressure is 6×10^{-2} torr. The pump is capable of evacuating 100 pounds of air per hour at 1.5×10^{-1} torr using 100 psig steam and 75°F condensing water. The ejector is made of carbon steel. The steam requirement is approximately 4500 lbs/hr and the condensing water requirement is approximately 650 gpm.

The operating procedure would be for the steam ejector to be turned on and attain blank-off pressure. With the chamber at slightly above blank-off vacuum having been filled with venting or purge gases, the valve between the chamber and ejector would be opened. The ejector has been located in the Heat Rejection Building to eliminate steam or water leaks inside the Space Power Building. It is also desirable to raise the chamber panels slowly to ambient temperature under vacuum in the final stages of decontamination before opening the chamber.

The low rates of vaporization or reaction of metallic fume from the chamber under these conditions allows the steam ejector to act safely as a final steam-fume reaction system. Fumes are scrubbed in the two barometric condensers. There are no significant problems in discharging the final stage ejector outlet directly to the atmosphere.

A major rupture of any of the liquid metal loops will require manual removal of spillage after cool-down and repressurization of the chamber. It is not considered feasible to provide drains from inner and outer chambers since solidification would have occurred prior to repressurization being safely accomplished.

4. Building Ventilation

In addition to the normal requirements for building heating and ventilation, special provision must be made for ventilation of fumes from the work areas using roof ventilators and hoods. Three stainless steel hoods are provided as shown in Figure 27, one surrounding the upper chamber opening to capture fumes from the chamber and two located in the decontamination room. The 20 by 10 by 20 foot decontamination room hood is completely enclosed and equipped for argon inerting. The other decontamination room hood is 5 by 25 feet with a 10-foot open clearance for use while cleaning small parts, etc. The face velocities of the hoods are approximately 100 fpm when operating at a combined full capacity of 40,000 scfm. The duct work is designed for velocities of less than 4000 fpm and each hood system is equipped with a separate control damper, as individual requirements will vary with the work flow.

The Space Power Building is equipped with standard roof ventilators to give approximately one room air change per minute over the chamber opening work area. This system will be used during operations when fumes evolve from the chamber or when test articles are being transferred from the chamber to the decontamination room. A similar roof ventilation system is installed in the decontamination room.

5. Scrubber

The scrubber is used to remove particles of liquid metal reaction compounds from gases exhausted from building areas where leakage and spills are likely to occur. It will also be connected to the NaK radiator enclosure as an emergency fume scrubber with the main blower operating at reduced power. The system is shown in Figure 27.

The design flow-rate is estimated to be 40,000 scfm based on the assumption that ventilation requirements for the building areas will not be at a maximum simultaneously. It does not appear likely that high temperature gas or liquid metal fumes will reach the scrubber before cooling and oxidation and there will therefore be no requirement for humidification or condensation equipment.

The system contains a two-stage Aerotec 400-FB, sub-micron scrubber. It is about 10 feet in diameter and 33 feet high. Of floating bed design and using 1 1/2 in. diameter polypropylene spheres, this unit can handle very high fume loads and recirculation liquid flow rates without fouling. The collection efficiency of 0.01 to 1 micron particles should be above 97 percent by weight. The pressure drop through the unit is 10 in. of H_2O at a liquid flow rate of 200 gpm. The recirculating scrubbing liquid can be either from the adjacent pit or directly from the scrubber base.

6. Containment and Suppression

Buildings are separated by fire walls and contain automatic fire doors for fire containment and control. In areas where liquid metals could be spilled, floor construction consists of a steel grating covered by perforated steel plates. Any spillage will be trapped in cells of the grating where the limited supply of oxygen through the plate perforations will suppress any tendency toward combustion.

All pipes carrying liquid metals are contained within ventilated sheet metal ducts to protect personnel and permit fumes to be exhausted without entering building air spaces. Areas where considerable quantities of liquid metals can be emptied from ruptured containers, such as under the NaK dump tank, are designed to contain the spillage in a pit and discharge a blanket of inert-gas over it.

7. Component Decontamination

Components from the liquid metal test loops will require decontamination after having been drained in the chamber while in operating position. Considerable quantities of liquid metal may be trapped and frozen in components such as the boiler and condensers. The largest single item to be handled is the boiler. It is 14 ft long by 3 ft in diameter and may weigh as much as 18,000 lb.

The procedure for decontamination is as follows:

a) Move component to the decontamination area
b) Strip insulation
c) Heat and remove liquid metal
d) Purge with CO_2, water vapor, and inert gas
e) Clean with water
f) Dry

The facilities provided for decontaminating components and piping are described in Civil Structural Design, Subsection V (B).

D. Mechanical Systems

This section describes the mechanical portion of the design.

1. Environmental Test Requirements

To meet test objectives, the chamber must provide the following:

a) A high vacuum environment during high temperature loop-operation to limit refractory metal corrosion. An allowable partial pressure for reactive gases as listed in Table 1 for columbium alloys and discussed in Section II. A time pressure product of 1×10^{-5} torr for 300 hours maximum for hydrogen during startup.

b) A low temperature heat sink completely surrounding the loop which will remove heat radiated from the loop and provide a pumping surface for water vapor. The heat to be removed from the facility is shown in the General Heat Balance, Table 8.

c) A clean, dry, and empty chamber pressure with a pressure of less than 5 x 10^{-10} torr for all gases to insure that the loop won't be contaminated by the chamber outgassing or by the pump backstreaming.

2. Chamber Vacuum System

a) General

The existing chamber vacuum system consists of a two-stage roughing system, and a noncondensable, high-vacuum system consisting of ten existing 35-inch diffusion pumps and associated vacuum lines and valves. The inner chamber ultra-high vacuum system will consist of twenty 36-inch combination sputter and getter ion pumps for hydrogen pumping and 1300 sq ft of helium cryopaneling for pumping nitrogen, oxygen, and carbon compounds. The system is shown in Figure 28.

The two-stage roughing system, connected to the chamber through the diffusion pumps, will evacuate the chamber to a pressure of 1 x 10^{-2} with a gas inleak of 5 torr liters/sec. When this pressure is reached, the ten 50,000 liters/sec diffusion pumps are started and cooling of the thermal shroud begins. The roughing system is used to back up the diffusion pumps and liquid argon cooled baffles located above the diffusion pumps prevent oil backstreaming. When the thermal shrouds have been cooled to 110°K, the shielded cryopanels are cooled. With the diffusion pumps operating, the thermal shrouds at 115°K (-252°F) and the cryopanels at 20°K (-423°F), the ultimate pressure with a clean, dry, and empty chamber will be less than 5 x 10^{-10} torr. System performance is shown on Figure 4 .

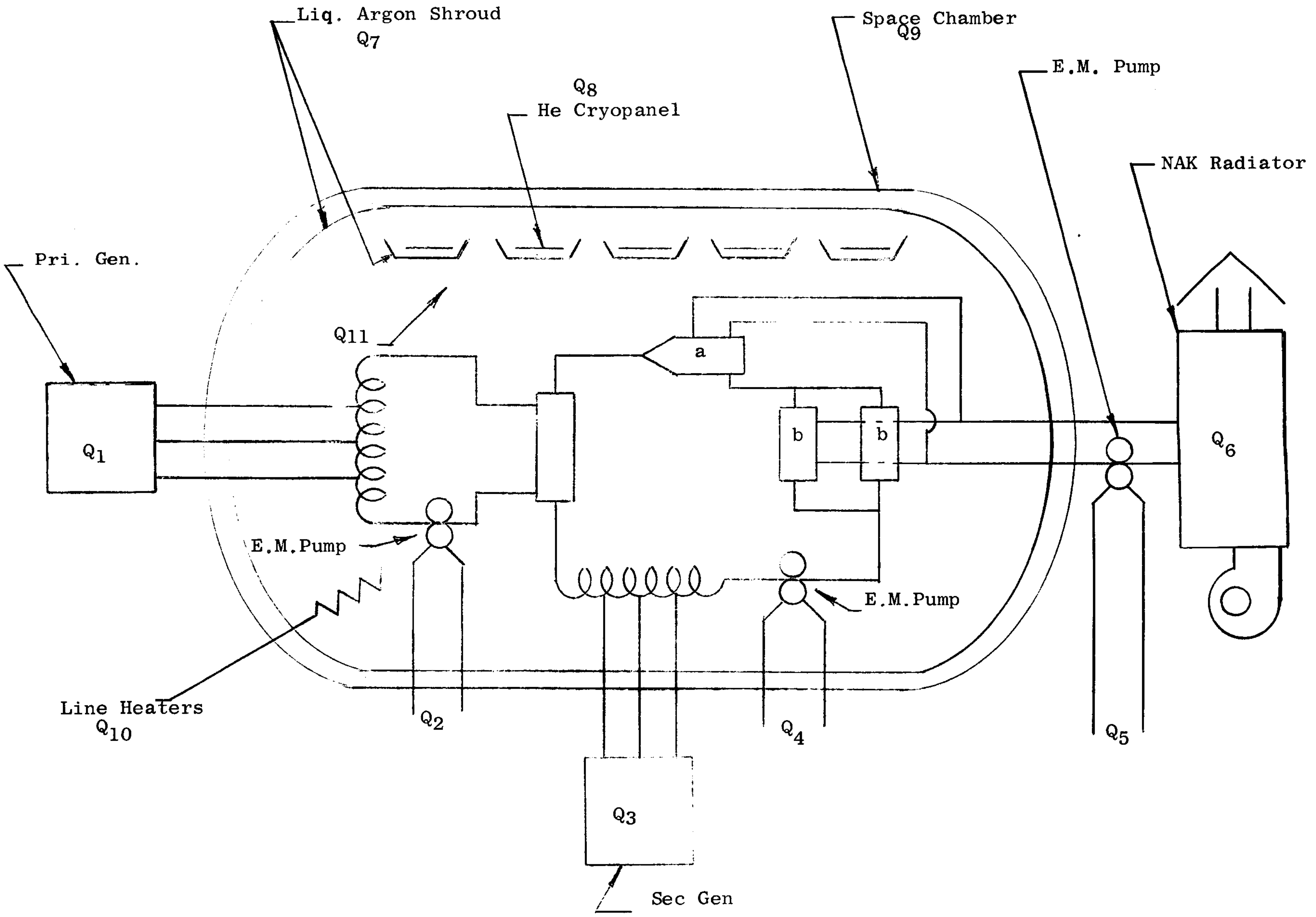
TABLE 8
GENERAL HEAT BALANCE

No*	Description	Heat Added or Removed	Max. Quantity KW	Remarks
Q_1	Primary Acyclic Generator	Added	16000	Into chamber as electrical power
Q_2	Primary E.M. Pump Cooling	Removed	393	Argon Gas @ 3.75#/sec. flow
Q_2	Primary E.M. Pump Power	Added	523	Electrical Power
Q_3	Secondary Acyclic Generator	Added	3000	Electrical Power
Q_4	Secondary E.M. Pump Cooling	Removed	95	Argon Gas @ .95#/sec. flow
Q_4	Secondary E.M. Pump Power	Added	130	Electrical Power
Q_5	Radiator E.M. Pump Cooling	Removed	446	Air @ 2.2#/sec. flow
Q_5	Radiator E.M. Pump Power	Added	597	Electrical Power
Q_6	NaK Radiator	Removed	15000	Air @ 92#/sec. flow
Q_{6a}	Turbine Simulator	Removed	2350	NaK @ 71#/sec. flow
Q_{6b}	Condenser	Removed	7500	NaK @ 226#/sec. flow
Q_7	Liquid Argon Shroud	Removed	900	Vaporizing LN_2 @ 5400 GPH (Indirect) Heat from Q_9 and Q_{11} & sources external to chamber
Q_8	Helium Cryopanels	Removed	2	Heat from Argon shrouds

* Nos. referred to sketch immediately following Table 8.

TABLE 8 (Cont'd)
GENERAL HEAT BALANCE

No.*	Description	Heat Added or Removed	Max. Quantity KW	Remarks
Q_9	Space Chamber	Added	23	
Q_{10}	Line Heaters	Added	350	Electrical Power
Q_{11}	Loop Ht. Loss	Removed	800	



GENERAL HEAT BALANCE SKETCH
(Refer to Table 8)

Two 36-in. flapper valves connect the inner chamber to the outer chamber for evacuation to 10^{-5} torr. Below 10^{-5} torr the chambers are isolated. If a differential of more than 10 in. of water exists between chambers, the valves will open automatically.

b) Roughing System Description

The rough pumping system is a two-stage system. Vent lines, containing relief valves set at atmospheric pressure, are located on the discharge of the first stage. During chamber evacuation, the first stage is bypassed to 0.75 torr. The roughing system is used as is currently installed. System performance is shown in Figure 29 and 30.

(1) Stage One

The first stage is a Roots-Connersville 10 x 24 RGS vacuum booster having a free air displacement of 2700 cfm at 1160 rpm. The booster is direct-coupled to a 1200-rpm induction motor.

(2) Second Stage

The second stage has two Stokes model 912-H mechanical pumps in parallel. Each has a free air displacement of 728 cfm and is driven by a V-belt with a 30-hp motor.

(3) Roughing System Safety Considerations

Temperature sensors will be located on the discharge from the booster. The sensors will actuate an alarm in the event of pump overheating and will stop any booster after three minutes if it remains overheated.

Nitrogen-gas ballast will be supplied to each mechanical pump. As an additional safety measure, oil mist filters will be included on the mechanical pumps.

c) Outer Chamber Vacuum Pumping

The outer chamber is evacuated and maintained at less than 1 x 10^{-5} torr by the ten 35-in diffusion pumps and backing train. Providing an intermediate vacuum in the space between the outer chamber and the shroud relieves the shroud of any significant structural load from vacuum conditions, reduces heat leakage from the shroud, and simplifies penetration seals subject to low temperature.

(1) Diffusion Pumps

Ten nominal 35-in. Consolidated Vacuum Corporation Model PMC-50000 diffusion pumps will be used. These pumps are standard production items and replacement parts are readily available. They also have a low backstreaming rate if the crossover range is limited to 10^{-2} to 10^{-3} torr. The backstreaming is less than 0.17 cc/hr.(1) These pumps are currently installed.

(2) Pump Fluid

The diffusion pumps will be filled with Dow Corning DC 705 fluid. This fluid provides the highest resistance against oxidation and fire in the event of repressurization.(2) The DC 705 fluid also has a higher decomposition temperature than other fluids and will reduce hydrogen backstreaming from the pump and minimize the gas load at chamber working vacuum.

(3) Baffle

A 35-in. liquid argon cooled chevron baffle will be used above the pumps to prevent oil backstreaming. The baffles are currently installed but will require minor modification to permit use with liquid argon.

d) Inner Chamber Vacuum Pumping

To provide the high pumping capacity required to remove large gas loads expected from operation of the test loop at elevated

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(1) NRC Catalogue

(2) Vapor Pressure-Fire Point Characteristics of Diffusion Pump Fluids in Appendix

temperatures, a combination pumping system is proposed. The system consists of surfaces operating at approximately 110°K (-261°F) to pump water vapor, at 20°K (-423°F) to pump oxygen, nitrogen, argon, methane, etc., and a combination sputter and getter ion pump (BoostiVac 20-900, or equivalent) to pump hydrogen, neon, and helium.

Since the inner chamber is completely isolated from the outer chamber during operation, no backstreaming from oil pumps is expected in the inner chamber. The inner chamber consists of a shroud operating at 110°K (-261°F) to remove all moisture and reduce wall outgassing to a negligible amount.

By gas specie, the inner chamber will have the following performance characteristics (Ref. Figure 4):

Water -110°K (-261°F) condensable- 71 x 10^6 liters/sec
O_2, N_2, A, CH_2, etc.-20°K (-423°) condensable- 3.3 x 10^6 liters/sec
H_2 - noncondensable - 1.3 x 10^6 liters/sec

(1) Ion Pumping

It is proposed to use the Ultek Model 20-900 BoostiVac pump, or equivalent, for pumping hydrogen. The pump combines the advantages of clean ion pumping with titanium sublimation. The pump is mounted external to the chamber with the cylindrical ion pump external and the filament holder extended concentrically into a liquid argon cooled cylinder. This argon cooled cylinder provides a surface for titanium sublimation. Thirty-eight titanium elements are provided on the holder for optimum sublimation performance. The pump is designed for continuous operation from 10^{-8} torr through 10^{-12} torr, and is capable of pumping the chamber down from 10^{-5} to 10^{-8} torr. The ion pump elements draw current directly proportional to the pressure in the pump and may be used to measure vacuum.

The ion pumping elements have a 40,000-hr life rating for continuous operation at 10^{-6} torr and 400,000 hr at 10^{-7} torr. The BoostiVac filament's life is a constant 4 to 6 hr per filament, regardless of pressure and its effective life in the system, and is controlled by cycling time.(1) Preliminary tests give values of approximately 50 torr-liters/titanium filament at 10^{-7} torr. By proper cycling, this value can be maintained at lower pressures to give over 10,000 hr of rated pumping at 10^{-9} and 10^{-10} torr from the 38 filaments available. Cycle on-times will vary as a function of pressure. Optimum results are achieved from continuous filament operation at 10^{-7} and 10^{-8} torr; 2 minutes on and 30 minutes off operation at 10^{-9} torr; and 2 minutes on, 5 to 10 hours off at 10^{-10} torr.

The Model 20-900 pump requires three Model 60-154 power supplies for operation of the ion pump elements and two Model 60-501 power supplies for BoostiVac filament operation. Only electric power is required: 230 v, single phase, 60 cycle, ac. A maximum of 18.3 kva is required during starting. Less than 2.3 kva is required for continuous operation below 10^{-7} torr. The installation of the pump is shown in Figure 22.

Advantages of Ion pumping include the following:

(a) Oils are not used as a pumping medium, eliminating any possibility of hydrocarbon contamination of the system. This insures more reliable and accurate test results.

(1) Bulletin: B-1221. Ultek Corporation

(b) During high vacuum operation, the system is completely sealed from atmosphere. Forepumping is eliminated and a power failure cannot destroy vacuum in the system. Pumping will start again when power is reapplied.

(c) Power consumption is low, averaging less than 2 KW during high vacuum operation, as compared with over 20 KW for a diffusion pump and its forepump.

(d) Vacuum system design is simplified because the pumps will operate in any position and do not have to be located vertically.

(e) A full 50,000 liters/sec of nitrogen and 70,000 liters/sec of hydrogen pumping capacity is available at the chamber orifice.

(2) Cryogenic Pumping

O_2 and N_2 pumping is provided by 1300 sq ft of shielded cryopanel operating at 20°K (-423°F). This array is estimated to have a speed of 3.3×10^6 liters/sec from 10^{-5} torr to 10^{-9} torr. The cryopanel is shielded from the loop by liquid argon cooled shields to reduce the heat load to the panel. The estimated heat load to the panels is 2 KW. The 2 KW is a judgment factor. The actual heat load will depend on cryopanel array chosen and emissivity of panels. The heat load also depends on loop temperature and area. The cryopanel array is shown in Figure 6.

e) Chamber Performance

The chamber is evacuated from ambient to 10^{-2} torr using the two-stage roughing train. At 10^{-2} torr, the diffusion pump heaters are turned on and the liquid argon is let into the shroud. When the diffusion pumps reach operating temperature, the chamber is evacuated to an operating pressure of 10^{-5} torr.

The roughing system and diffusion pump performance is shown in Figure 30, chamber evacuation time is 4 hr. The time vs pressure curve is shown in Figure 29.

Pumpdown performance of roughing system is given in Table 9.

TABLE 9

CALCULATED TIME TO EVACUATE CHAMBER

(Clean, dry and empty - outer chamber pumping only)

P_1 torr	P_2 torr	Calculated Time (min) Incremental	Calculated Time (min) Cumulative
760	1	45	45
1	10^{-2}	145	190
Diffusion Pump Warm Up		60	250
10^{-2}	10^{-3}	42	292
10^{-3}	10^{-5}	2	294

The hydrogen pumping speed is shown in Figure 1 which illustrates the importance of operating below 10^{-7} torr. The total performance of the chamber for all gases is shown in Figure 4 where throughput vs pressure is given for various gas species.

The ultimate pressure, P in torr, attainable in any vacuum vessel, is given by:

$$P = \frac{\sum Qi}{S}^{(1)}$$

where Qi denotes the amount of gas influx from all sources in torr liters/sec, and S denotes the system pumping speed in liters/sec.

The gas influx will come from 6 general sources: (1) true leaks; (2) virtual leaks; (3) specific influx from the walls; (4) seals; (5) noncondensable cracking products from the ion pumps; and (6) the test article.

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(1) Equation (4-7), pg. 49, High Vacuum Engineering by A. E. Barrington

In the inner chamber, the shroud is cooled to 110°K, thus reducing the outgassing rate to less than 10^{-12} torr liters/sec; virtual leaks will be essentially eliminated and true leaks will be less than 10^{-7} torr liters/sec because of the double wall construction. No organic seals will be present in the chamber, because of the possibility of contamination. The ion pumps have demonstrated their ability to operate at 10^{-12} torr and should not be a source of backstreaming.(1) The operating pressure for the inner chamber is less than 5×10^{-10} torr. Since the expected working pressure will be 5×10^{-9} torr or greater, the base pressure gas load will be less than 10% of the total gas load and can be neglected within experimental errors. The working or operating pressure is therefore determined by outgassing of the test loop.

The outgassing rate of a material is determined by the material, its previous history and operating temperature. Figure 2 shows the outgassing rate for stainless steel as a function of temperature after 24 hr at temperature. Figure 3 shows the outgassing rate of stainless steel by gas specie as a function of time for stainless steel at 500°K (440°F).

Chamber performance is determined by the gas load. The chamber is designed to have the largest feasible gas throughput.

Figure 31 shows the allowable square feet of surface area for a given partial pressure as a function of various outgassing rates.

f) <u>System Limitations</u>

<u>Backstreaming</u>

It is extremely difficult to completely eliminate backstreaming; however, it can be reduced to a very low level. Backstreaming can occur from the mechanical pumps, the Roots-type pumps, and the diffusion pumps. The backstreaming

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(1) Ultek literature and papers on Ion Pumps

from any source is pressure dependent.

In the high pressure or viscous flow region, diffusion gas sweeping occurs and hence contributes very little to the backstreaming. In the transition region, backstreaming is most significant. For the roughing system this would occur from about 10 torr to 10^{-2} torr. It is difficult to trap backstreaming gas in the transition region by mechanical methods because the mean system dimension is so small that it would limit the conductance. Therefore, some other technique must be used. Backstreaming will occur from the diffusion pumps in the 10^{-2} to 10^{-4} torr region and operation in this pressure region should be limited. Liquid argon-cooled, optically-dense baffles will be used to reduce backstreaming in the molecular region.

The regime of flow for control of backstreaming is shown on Figure 32. The backstreaming rate through a baffle is shown on Figure 33. To trap oil backstreaming in the transition region, an ion baffle (See Figure 34) is proposed. This consists of a cylinder with an electrode operating at high voltage located axially. The oil is decomposed into low molecular weight molecules which are evacuated by the mechanical pump. The typical performance of the ion baffle is shown in Figure 35, indicating a significant improvement in backstreaming with an ion baffle operating.

3. Loop Radiation Heat Removal

a) Thermal Shroud. A panel coil type shroud will be constructed to form an optically dense and essentially vacuum tight inner chamber. The shroud will form both a means for condensing water vapor, heat removal, and vacuum closure with very low outgassing. The shroud material will be Type 304L stainless steel to be compatible with the liquid metals.

The proposed arrangement of the thermal shroud is shown in Figure 6 . The shroud is welded in place and is free to contract longitudinally. The large end is clamped closed, making a metal-to-metal low conductance seal. All feedthroughs are welded in place. There is adequate clearance around the shroud for maintenance and cleaning without removal.

The shroud system comprises 8 zones; 6 around the circumference of the chamber; one each at the closed end, and the removable end. Each of these is cooled by an independently controlled liquid argon circuit. The shields for the helium cryopanels are zone controlled and interlocked with the helium refrigerators.

The entire interior surface of the shroud is coated to provide a radiation absorption coefficient of greater than 0.9 to reduce reflected radiation to the cryopanels. The outside surface of the shroud and the shield panels are electropolished to reduce heat absorption. The absorption coefficient will be less than 0.1.

The thermal shroud will have a leak rate of less than 5×10^{-7} torr liters/sec. The panels will be designed to operate up to 150 psi. The shroud will be cooled with liquid argon to a surface temperature of $115^{\circ}K$ ($-252^{\circ}F$) within 2 hrs.

The shroud temperature will be measured at three points on each zone. Temperatures will be recorded in multipoint chart recorders and used to control independently the temperature of each zone.

b) Refrigeration System. The argon cooling system provides a subcooled liquid argon to the thermal shroud. Liquid nitrogen is supplied to the subcoolers from a liquid storage facility. The argon flow rate and temperature are controlled to vary the surface temperature of the thermal shroud within the required range. The system is shown schematically in Figure 36.

Since argon is used as a coolant, it is necessary to provide a pressurized system to prevent freezing. A nitrogen-argon phase diagram is shown in Figure 37.

The primary liquid argon system for continuous heat loading is composed of a subcooler and circulation unit, control instrumentation, and necessary transfer and distribution lines and valves. The subcooler and circulation unit operates on a closed-cycle, single-phase principle in which the heat is removed from the circulating stream by heat exchange with nitrogen boiling at essentially 35 psia pressure. The unit is composed of a Dewar subcooler with integral heat exchanger, variable speed pump, insulated valve manifolds, and a control panel. Insulation is Linde Division, Union Carbide Corporation, Super-Insulated VIP and/or foam-insulated polyurethane. Valves and instrumentation are of proven design for remote operation.

The primary liquid nitrogen system consists of: (1) a 20-hp, variable-speed, centrifugal pump with a 300-gpm capacity, (2) a Dewar subcooler with integral heat exchangers, (3) insulated valve manifolds and (4) a remote instrument and control panel. The liquid nitrogen subcooler, including the subcooler Dewar and control console, is a complete package. Four complete packages are provided such that one unit will provide redundancy and backup. The schematic layout of the equipment is shown in

Figure 36. Subcooler vents are insulated and plumbed to the exterior of the mechanical equipment building. The system is located in the mechanical equipment area.

Each of the 20-hp pumps is capable of supplying approximately 300 gpm of liquid nitrogen. This gives both flexibility of operation and necessary standby capability for long term tests. Each unit consists of a horizontal centrifugal pump (Byron Jackson Company, or equal) coupled to a two-speed, double-winding electrical motor. Each pumping unit is individually controlled.

The Dewar subcooler consists of a vacuum, powder-insulated vessel containing a heat exchanger and a reservoir of liquid nitrogen at atmospheric pressure. Both the outer casing and the inner vessel are designed in accordance with the ASME code for pressure vessels. The heat exchanger is a braced-aluminum plate, fin-type unit, to be selected in final design, (Trane Company, or equal) and is mounted vertically within the inner vessel. The inner vessel is supported within the outer casing, using a low heat-leak method of suspension and thereby minimizing product loss. Both inner and outer vessels are of welded construction. All welding on shell seams of the inner tank will be made by the helium or argon shielded arc method.

A vacuum line, with valve, is provided for evacuation of the annular space of the vessel. A suitable gauge for monitoring annular space will be provided.

The remote control panel of the subcooler provides a graphic representation of the unit. Controls will be located for simplicity of operation and monitoring. The panel includes pressure and temperature indicators, indicator lights, a Dewar level indicating controller, required pump controls, and push-buttons.

Normal liquid argon inlet temperature to the shrouds will be 85°K (-306°F). Outlet temperature will be 103°K (-274°F). The complete system is designed to maintain subcooled liquid nitrogen at an approximate pressure of 100 psig in the return line from the chambers to minimize two-phase potential. The design working pressure is 150 psig.

The primary system is capable of stable operation with constant heat loads from 300 to 1200 KW. Fluid makeup to the pressurized circulation loop is obtained from the NASA-furnished storage and supply system to prevent loss of subcooling. The inherent nature of the subcooler system, as specified, necessitates continuous uninterrupted flow at the inlet.

The nitrogen level in the Dewar subcooler is automatically maintained by a level-control (to be selected in final design) which senses the static 'test of liquid and activates a control valve in the makeup line from the storage system.

The pumps and heat exchanger can be placed in operation about 1/2 hr. after initiation of cooldown. The system is capable of cooling the thermal shrouds from ambient temperature to a maximum 115°K surface temperature in approximately two hours.

(1) Heat Load. The primary liquid argon system is designed to maintain the thermal shroud at 115°K (-252°F). The total design load is shown in Table 8, Section V(D), and was determined as follows:

Heat from Loop	800 kw
Heat from Chamber Structure	23
Heat from Pipe & Valves	14
Heat from Other Sources (Safety Factor)	63
Total Design Load	900 kw

The Linde Division, Union Carbide Corporation, Super-Insulated VIP pipe, with vacuum preservation, is used for all straight sections, 20 ft. or longer, outside of the chamber. This pipe, with vacuum preservation, is the best known cryogenic insulation system available at a reasonable price. Shorter lines outside the chamber, which continuously handle liquid nitrogen, are insulated with polyurethane foam, applied in two layers with staggered joints and covered with a suitable moisture barrier. This system averages less than 1.0 Btu/hr/ft heat leak for straight sections; valves and fittings average higher. The liquid argon lines within the chamber are not insulated because chamber vacuum provides sufficient insulation.

The liquid argon system is sized to handle the 900 kw. The argon will enter the chamber at approximately 110 psia and 85°K (-305°F) and leave at 90 psia and approximately 103°K.(-274°F). The design working pressure is 150 psig.

Signals for monitoring and controlling operation and performance are provided in the central control room. All components, such as the subcooler, will be started at the central control room.

Distribution of liquid argon through the shroud zones of a chamber is accomplished by remote operation of the control valves in the discharge lines (Ref. Fig. 36). The valves provided for operating at cryogenic temperatures are designed to assure good performance and to minimize heat leak into the system during continuous cold service operation.

(2) <u>Choice of Coolant.</u> Water, nitrogen, and argon were considered for the shroud cooling system. Because of the possibility of shroud failure and the resulting rapid corrosion of the test loop in the presence of nitrogen or oxygen, the fluid recommended is liquid argon. The argon will be circulated under pressure and heat removed in a subcooler using liquid nitrogen as the refrigerant. Liquid nitrogen consumption is 6 gal/kwh, which would correspond in this case to 5400 gal/hr for operation at 900 kw.

(3) Temperature Requirements. The temperature of 115°K (-252°F) is chosen as the operating temperature of the panels between fins because of need to operate at the temperature where argon, the selected medium, is a liquid. A liquid is desirable because of the heat transfer characteristics.

Liquid nitrogen is selected as a convenient refrigerant because of its high latent heat capacity (86 Btu's/#) and relative low cost.

(4) Use of Existing Equipment. A preliminary investigation has been conducted to determine whether converting the existing wind tunnel refrigeration system for service in support of the space lower chamber merits further study. This preliminary investigation indicates that a more detailed study will be worthwhile. The facts that lead to this conclusion are presented in the following discussion.

The main purpose of the existing Carrier refrigeration system was to cool the low pressure air circulating in the Altitude Wind Tunnel. A secondary York system cooled the combustion air fed to test engines.

The Carrier system consists of 14 centrifugal compressors driven by 1500-hp motors, each rated from 250 to 550 tons of refrigeration, depending on the temperature and pressure conditions under which they are operated. Performance limits of the system as it is now installed appear to be a refrigerant temperature range having a lower limit of -65°F. The total refrigeration tonnage at this temperature is approximately 3500. At higher temperatures, tonnage capacity increases to a maximum of about 7700, assuming an 87°F heat rejection temperature.

The cooling requirements of the Space Power Chamber conversion is approximately 300 tons at -300°F.

It may be possible to modify the existing system to accept 300 tons of refrigeration at -120°F and reject this heat at 87°F. The modification would probably consist of compounding the existing machines, changing their operating speed and changing the refrigerant and replacing some of the cycle components (such as heat exchangers) with components made of more suitable materials. This expansion of the existing system's operating temperature range reduces the systems capacity to such an extent that it may be necessary to add an additional unit in order to obtain an 87°F heat rejection temperature.

Another additional refrigeration cycle will have to be purchased. This cycle will have to accept 300 tons of heat at -300°F and reject it to the modified existing equipment at approximately -120°F.

This preliminary investigation indicates that even if a more suitable refrigerant is employed, the present equipment is not capable of operating down to -300°F.

It is recommended that the matter of converting the existing refrigeration system be studied in more detail. The object of further study would be to balance the capital and operating costs of modifying or adding to the existing plant versus corresponding costs of alternative heat removal proposals. The two alternative plans that would be considered are (a) removing the total heat by using it to boil vendor supplied liquid nitrogen and (b) constructing a new, more advanced refrigeration system to handle the total load.

The lowest initial cost is obtained by using purchased nitrogen to cool liquid argon that is used as the circulating fluid in a closed loop. This is the system assumed in the present study.

(5) System Layout. The System Layout is shown in Figure 36.

4. Dense Gas Helium System

a) System Description

The dense gas helium system consists of three helium refrigerators, each with a nominal capacity of one kw at 20°K (-423°F) or lower, and a cold gas distribution system. These components and all required auxiliary piping, valves, and controls are shown schematically in Figure 38.

The three refrigerators are used to provide flexibility of operation, redundancy, and reduced operating costs during periods of low refrigeration usage. A manifolding system allows any of the three units to be removed from service without affecting operation of the other two.

The cryogenic helium system is operated and monitored from the control room. Control panels indicate pressures and temperatures at key points in the system and the positions of the remote operating helium distribution valves. To minimize steady state heat leak and cooldown loads imposed on the refrigerators, the cryogenic helium gas distribution piping is vacuum super-insulated pipe.

b) Panel Configuration

The cryopanels will provide condensing surfaces for gases which are condensable at about 20°K (-423°F) and thus serve as effective pumps for all but a few gases. The cryopanels are shielded from the test article and chamber structure and any other heat sources within the chamber by liquid argon cooled panels. The cryopanels are supported longitudinally on low conductivity supports and have a high reflectivity to minimize heat inleakage. The cryopanels are cooled by a helium refrigeration system.

The cryopanels are fabricated from Type 304L stainless steel and are arranged in three zones around the circumference of the chamber. Each zone has independently controlled helium cooling. This cryopanel array provides a shielded work zone 16 to 20 ft in diameter. The array is shown in Figures 6 and 28.

All cryopanels are located in the cylindrical part of the chamber so that the chamber access end may be removed and the access door may be opened without breaking any helium piping connections.

The panel configuration has been selected to maximize available work area and maintain high pumping speed with a slow heat loss.

c) Cryopanel Performance

The refrigerator is sized to maintain a maximum surface temperature of 20°K (-423°F) with the design heat load of 800 kw from the test loop radiated to the argon cooled shroud. The heat load is estimated at 2 kw. Three kw in three separate units provide redundancy since, if the panels are inadvertently warmed up, all gases which were pumped will be released.

The pumping speed is estimated at 3.3×10^{-6} liters/sec for oxygen and nitrogen at pressures from 10^{-5} to 10^{-9} torr.

The panel leak rate will be maintained less than 5×10^{-7} torr liters/sec for the entire cryopanel system as installed.

The cryopanel temperature will be measured in each zone. Temperatures are used to control the panel temperature of each zone independently.

d) Helium Refrigerators

The helium refrigerators (Linde, Cryovac or equal) are furnished as a complete skid mounted package. The systems are located in the mechanical equipment area under the chamber.

The refrigerator consists of expanders, heat exchangers, helium purifiers, gas reservoirs, integral piping, control and other auxiliary equipment.

Pressure and temperature sensors will be installed at three points within each cryopanel zone. Temperature sensors would be thermocouples prepared to NBS standards. The pressure gauges could be supplied by Cryovac or equal.

The refrigerators/compressor system is designed to maintain a continuous sub 20°K (-423°F) flow of cold helium gas at a minimum pressure of 30 psig to the cryopanels. The system is designed to operate continuously for a period of up to 1000 hr. without maintenance.

The system is a closed cycle type and is designed to recover the helium for storage on panel warm up.

The helium system also provides for controlled warmup of the helium panels from 20°K (-423°F) to 100°K (-279°F).

5. Heat Rejection System

The function of the NaK loop is to remove heat from the potassium loop. Heat enters the NaK loop in the potassium condensers and the turbine simulator and leaves the NaK loop through the NaK-air heat exchanger. This portion of the report describes the air system which removes the heat rejected by the NaK loop. The air system consists of two subsystems. The major subsystem removes up to 51.2 million Btu/hr (15 MW) from the NaK-air heat exchanger. The minor subsystem removes the 280,000 Btu/hr heat loss from the NaK lines located outside the space chamber, plus the heat rejected from the exterior surfaces of the major subsystem. These subsystems are discussed separately in the following paragraphs.

a) NaK-Air Heat Exchanger Air System

The primary function of the NaK-air heat exchanger air system is to remove 15 MW from the NaK in the NaK-to-air heat exchanger. This air system also serves to heat the heat exchanger in preparation for NaK loop startup. Figure 39 includes a schematic of the NaK-air heat exchanger air system.

(1) Normal Operation. During normal operation, air enters the NaK-air heat exchanger at 200°F and leaves at 850°F. The 200°F inlet air temperature is obtained by recycling some of the heat exchanger exhaust air and mixing it with ambient air prior to introduction to the blower inlet.

Valves 1, 2 and 5 (Figure 39) are modulating valves which are automatically controlled to provide the proper amount of 200°F air required to handle any NaK cooler load at any facility ambient temperature. Valves 3, 4 and 6 are closed during normal operation. The NaK cooler exhaust air that is not used to heat blower inlet air is exhausted through the stack to the atmosphere. The top of the stack is 65 feet above ground level.

During normal operation at the 15 MW heat rejection level, 329,000 pounds per hour of 200°F air is required by the NaK cooler. On a cold day, about 75 weight percent of this air will be drawn from the atmosphere. The remaining 25 percent will be recycle air taken from the NaK cooler discharge. The blower total pressure rise will be approximately 20 in. of water column.

There are advantages and disadvantages to a single NaK cooler blower system and the alternative dual parallel blower system. The relative merits of each will be weighed again before definition of final design is complete. It is possible that a dual blower design may be used for the NaK cooler as a result of this later analysis.

(2) Start-Up. During start-up, the air system supplies 80°F air to heat the NaK-air heat exchanger core. The heat for this duty will be supplied by 100 psig saturated steam in the heat exchanger located between valves 3 and 4 in Figure 39.

During start-up operation, valves 1, 3 and 5 (Figure 39) are modulated automatically to maintain an 80°F air temperature at the NaK cooler inlet. Valve 4 is open but does not modulate. Valves 2 and 6 are closed. Warm air that has passed through the steam-air heat exchanger is mixed with ambient air to provide the required air temperature at NaK-cooler inlet. After passing through the NaK cooler, some of the air is recycled back through the steam heater while the remainder goes out through the stack to the atmosphere.

The steam-air heat exchanger has been tentatively sized to provide two million Btu/hr for system heating. The design philosophy of the start-up system is to make it as small as possible, consistent with the start-up requirements, because it is used much less than other cooling system components. The size is dictated by the amount of metal to be heated, the temperature to which this metal must be heated, and the time allowed for heating. In a later phase of the design when these criteria have been defined, the start-up system will be resized with an eye toward further reducing its size.

With the presently defined system, if the ambient air temperature is -10°F, then 40,000 lb/hr of air is drawn from the NaK cooler discharge and passes through the steam-air heat exchanger. The resulting hot air is mixed with 53,000 lb/hr of cold air which is drawn from the atmosphere. Mixing takes place upstream of the blower, and the total flow passes through the blower and NaK cooler.

With the proper combination of design criteria, it would be possible to eliminate the start-up system altogether. In this case, initial heating would be accomplished utilizing the fan temperature rise and passing full flow through the blower with valve 1 closed, valve 2 open, and valve 5 open only enough to vent the pressure caused by expansion of the air. This would be a boot-strap operation, with the fan inlet temperature being increased with each recirculation cycle. When the fan discharge temperature reaches the desired level, valve 1 could be opened to allow cooling via mixing with ambient air.

(3) Emergency. The two emergency conditions that affect the NaK-air heat exchanger air system are (1) a NaK leak into the air stream and (2) a power failure. Both of these eventualities are automatically covered. As soon as a predetermined NaK or NaK combustion product concentration is detected by sensors in the stack, valve 6 (Figure 39) opens, valve 5 closes, and power input to the primary loop and the NaK flow rate are cut to a level which is consistent with the reduced cooling capacity of the NaK-Air Heat Exchanger Air System. The emergency capacity of the NaK-Air Heat Exchanger Air System varies from 3 MW to 4 MW depending on ambient temperature. All of the NaK cooler exhaust air is discharged through valve 6 and the scrubber before it is exhausted to the atmosphere. During this type of emergency operation, valves 1 and 2 still modulate automatically to maintain $200^{o}F$ inlet air temperature to the NaK cooler.

In the event of a power failure, the auxiliary power supply will automatically start and supply electricity for the NaK Air Heat Exchanger Air Systems and other essential space chamber systems, until a safe shutdown can be accomplished. Any failure in the NaK-air heat exchanger air moving system equipment will be sensed by automatically monitoring NaK-cooler discharge air temperature. In the event that this temperature exceeds a predetermined limit, the power input to the primary test loop heater and the NaK flow rate will be cut. Valve 6 will be automatically opened, valves 5 and 2 will close and valve 1 will go wide open. The scrubber blower, running on power supplied by the auxiliary power unit, will draw cool air through the NaK cooler until a safe shutdown can be accomplished.

b) Heat Rejection Facility Ventilating System.

All NaK lines and containers outside the space chamber including the NaK-air heat exchanger air system are enclosed in an essentially air-tight building. These lines, containers and the air system transfer approximately 600,000 Btu/hr to the air in the heat rejection building. The primary function of the ventilating system is to remove the resulting hot air from the heat rejection facility building. The ventilating system consists of two 17,000 cfm fans located on the outside wall of the building, two corresponding remote controlled air inlet louvres, two remote controlled exit louvres, and a remote controlled valve connecting the dump tank building to the air scrubber suction line.

During normal operation, the inlet and discharge louvres are open, the air scrubber line valve is closed, and the fans move 34,000 cfm of air through the building. The air temperature inside the building is maintained at a level approximately 20°F higher than the ambient air temperature.

In the event of a NaK leak or NaK fire in the building, the scrubber line valve is automatically opened and the exhaust louvres are automatically closed, hence all contaminated air from the building is directed through the scrubber before it is exhausted to the atmosphere. The inlet louvres remain open and the fans remain in operation so that the flow of fresh air is not interrupted.

If the scrubber is already operating at capacity, or if the extent of the NaK leak is so great that the introduction of fresh air is undesirable, then the intake louvres and scrubber line valve can be closed from the control center and the air-tight building can be flooded with inert gas to extinguish the fire.

In the event of a general power failure, the auxiliary power unit supplies electricity to operate the fans, louvres, and valves as discussed in the preceding paragraphs.

In the event of a fan malfunction, the scrubber line valve can be opened from the control center and the scrubber blower used to draw air through the building. Opening both the inlet and discharge louvres will establish a natural convection air flow which will augment the cooling capacity available from the scrubber blower.

6. Auxiliary Systems

a) Chamber Ventilation

The ventilation system will provide for exhaust ventilation of the chamber during servicing of the test article under ambient conditions and chamber cleaning. No provision is made for heating or cooling of the ventilation air. The system provides an air flow rate of 7000 scfm through the chamber, i.e., approximately five changes of air per hour.

The ventilation blower is located outside the chamber. It exhausts to the scrubber or directly to the 65-ft stack provided on the facility.

Intakes and ducts are of standard ventilation design. The duct penetration through the chamber wall is of 24-inch nominal diameter. The ventilation isolation valve is of carbon steel.

b) Chamber Lighting

The chamber is illuminated by eight 110-v lighting fixtures located near the top of the chamber. The lamps are 500-w, tungsten filament, tubular quartz lamps similar to those in thermal simulators. These lamps provide sufficient illumination for test loop set up and visual observation through the test ports. Special lighting may be required if television or photographic cameras are used for test surveillance.

c) Auxiliary Argon and Helium Systems

Argon for auxiliary systems will be supplied from vaporization of liquid argon from a storage tank. The liquid argon will be fed to a vaporizer and then to a compressor where it is pressurized to 100 psig in a 100-cf accumulator. The gas is available for use in a number of systems from the accumulator. A flow schematic is shown in Figure 40. The following systems are connected to the accumulator:

(1) Valve operation at one lb/sec up to 100 psi.

(2) Chamber repressurization at 30 psig inlet pressure.

(3) EM pump cooling. Make up is provided to the recirculating system giving 5 lb/sec at 100 psig and 100°F. A heat exchanger is provided to remove 560 Btu/sec.

(4) Cover gas. Cover gas is provided for the Li dump tank, Li surge tank, K dump tank, NaK tank and NaK surge tank. The typical impurities contained in liquid argon are as follows: Oxygen - less than 5 PPM, Hydrogen-nil, Nitrogen-less than 25 PPM, and carbonaceous gases-nil.(1) It is felt that, since the cover gas is stagnant and a small percentage of gas weight to respective liquid metal weight, the gas impurities will be acceptable.

A high purity inert environment is necessary for welding refractory metals. A helium purification system is included to provide 100 cfm of gas at 30 psia with an active impurity level below 5 parts per million. The system uses high temperature zirconium to purify commercial high purity helium.

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(1) Product Specifications for Liquid Argon from Air Products and Chemicals, Inc.

E. Electrical

a) Site Electrical

The existing switch gear and control system installed for the wind tunnel drive motor will remain and supply power to the primary loop acyclic power generator. The power will be supplied at 4160 volts, and peak power required for this drive is 17,500 kva to generate 15 MW at 67 vdc. The existing 480-v switch gear will be used for the outer chamber vacuum pumping system. This will require 500 kva.

New switch gear will be installed to handle power for the remainder of the new facility. The 4160-v line is assumed available at the site. The new loads require the service characteristics listed in Table 10.

The one-line diagram of the system is shown in Figure 26.

One transformer is supplied for 480-v test power and one for 208/120-v line heaters. A second 480-v transformer will supply power to equipment added to the facility.

b) Primary Loop Heater

Power for the lithium-loop heater is supplied by two new 8 MW, 60-vdc acyclic generators(1) driven by an existing 19,750-hp wound-rotor motor through a speed increaser. Current output is controlled by varying the dc current to the field coil; fields are controlled in unison to obtain the same voltage and current from both generators. Loop temperature will be controlled by regulating input power by varying the field current. Field current will be regulated by a closed-loop feed-back control which senses loop temperature.

The dc output from the acyclic generators will be transmitted to the chamber through a system of four air-cooled copper bus bars. Each bus bar is made of twenty 3/4-inch copper bars ten inches wide. The individual bars are connected to a single copper bar 15 in. in diameter, with provision for liquid argon cooling, before penetrating the outer wall (see Figure 41). This bar is brazed with 65 Cu/35 Au alloy to a columbium-1/zirconium alloy casting. The lithium loop is passed through the casting and welded.

(1) Bulletin GED-4471 Large Motor & Generator Dept., General Electric Co., Schenectady, N. Y.

TABLE 10

NEW ELECTRICAL LOAD SERVICE CHARACTERISTICS

Boiler Preheater	-	3,000 kva	-	4160 v	-	3 phase
EM Pump Power	-	1250 kva	-	480 v	-	3 phase
NaK Dump Tank Heater	-	75 kva	-	208 v	-	3 phase
Instrumentation	-	50 kva	-	120 v	-	1 phase regulated
Line Heaters	-	350 kva	-	208/120 v	-	3 phase intermittent and controlled
Ion Pumps	-	366 kva	-	480 v	-	3 phase intermittent and controlled
Chamber Heat Removal and Helium Refrigeration	-	850 kva	-	480 v	-	3 phase
NaK Cooler Fans	-	500 kva	-	4160 v	-	3 phase
Scrubber Fan	-	200 kva	-	480 v	-	3 phase
Air Conditioning	-	50 kva	-	480 v	-	3 phase
Lighting	-	75 kva	-	208/120 v	-	3 phase
	-	100 kva	-	480 v	-	3 phase
Ventilation Fans	-	50 kva	-	480 v	-	3 phase
Unit Heaters	-	10 kva	-	480 v	-	3 phase

SUMMARY

Existing	17,500 kva,	4160 volts
	500 kva,	480 volts
New Load	7,000 kva,	4160 volts
Total	25,000 kva	

c) Conversion of Existing Wind Tunnel Fan Motor

The existing 19,750 HP motor referred to in the previous paragraph was originally installed to drive the Altitude Wind Tunnel Fan. The maximum speed is 450 rpm and it can be controlled down to 300 rpm.

It is mounted on an elevated foundation with its centerline coincident with that of the wind tunnel, about 30 ft. above the ground as shown in Figure 41. The 9-ft distance between the coupling face and the outer surface of tunnel stiffeners is insuffecent for installation of both an in-line type of gear-shaped increaser and the acyclic generators.

Several alternative solutions to this problem were explored.

(1) Replace the existing motor with a new one installed at ground level and designed to operate at the 3600-rpm speed of the generators.

(2) Use a double right angle drive gear-speed increaser with generators located alongside the wind tunnel.

(3) Relocate the existing motor horizontally in a position where adequate space for the in-line speed increaser and generators can be provided.

(4) Lower the existing motor to ground level where adequate space exists under the wind tunnel for the gear drive and generators.

Alternative (4) is the least expensive by a considerable margin and is the recommended solution unless a more compact gearing and generator arrangement can be developed that can fit into the existing 9-ft. space.

The existing switchgear and control systems would continue to be used for motor startup, but it is not proposed to operate at other than maximum speed. Generator output would be regulated solely by variation in field current.

d) Boiler Preheater

Two 1500-KW acyclic generators(1) driven by a single 4250-HP, 4000-v,60-cycle synchronous motor will provide input power to the boiler

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(1) Bulletin GED-4471 Large Motor & Generator Dept., General Electric Co., Schenectady, N.Y.

preheater. The heater will operate at 26 vdc to reduce interference with instrumentation. Acyclic generators will be used to eliminate ac ripple. The output will be controlled by varying the current to the field coils. The bus system will be of similar construction for the lower (1500-KW) capacity.

e) Loop Heating

During startup, auxiliary heat will be supplied to the loop by resistance heaters. A total of 146 (20-amp 120-vac) circuits will be provided for this purpose.. Each circuit will be supplied with temperature sensing and on-off control.

f) Auxiliary NaK Dump Tank Heater

75-kva, 208-v, 3-phase power, controlled by a temperature sensor, will be available for the NaK dump tank heater. On-off control is provided.

g) Emergency Power

To maintain a safe environment during failure of the electric power system, an emergency generator will maintain power to the vacuum equipment and other facilities requiring continuous operation. The unit proposed is a 1600-KW, 2000-kva, 4160-v, 3-phase 60-cycle, natural gas fueled, spark ignited, engine driven, synchronous generator. The unit will be started automatically on failure of normal power to the 4160-v bus. The proposed vacuum system, as well as the existing vacuum system, will be restarted automatically. However, other drives required in operation will have to be restarted by the operators. Power will be provided to selected instrument and control panels as discussed under Safety, Sub-paragraph V(C)(1).

VI. INSTRUMENTATION

A. FACILITY INSTRUMENTATION

1) Electronic Instruments

Wherever possible, the instruments used for facility and vacuum control are standard types employed in the process industry. Transducers, located at various places in the chamber and in the operating systems, generate low voltage signals representing operating conditions (pressure, temperature, flow, etc.) which are routed to the control room.

2) Local Instruments

Local instruments for air, argon gas and liquid, liquid nitrogen and dense helium consist of conventional pressure gages, temperature gages, and local pneumatic controls.

3) Control Panels

The control panels for the facility are modular types using standard relay racks. The panels in the main control room have two types of graphic displays. One is a block-diagram presentation showing system operation by illustrating main vessels, equipment and line layout. The other displays a sequence of events and illuminates each event in turn as the sequence is followed. A countdown console, using this type of display, will be located in a central position in the main control room to coordinate control of the facility and the test article. The controls and indicators will be grouped below the graphic display and the alarm annunciators will be above. Lights on the graphic display will provide status information. The control set points are set by the operator.

4) Vacuum System

Control of the vacuum system consists mainly of on-off switching and monitoring functions. These functions include: startup of mechanical pumps, blowers, oil diffusion pumps, and ion pumps; monitoring of utilities such as cooling water and lube and sealing oil; regulation of gas ballasting; and operation of high vacuum valves. All systems are appropriately interlocked.

Instruments are provided for monitoring test chamber pressures from atmospheric to 1×10^{-12} torr and are connected to equipment for transmitting these data to the remote control room. These instruments are as follows:

(1) Three diaphragm-type pressure transducers to monitor pressures from 760 torr to 1 torr.

(2) Four Alphatron ionization gages, with alpha particle sources, capable of monitoring pressures from 1 to 1×10^{-4} torr (two in chamber; two in foreline).

(3) Six Bayard-Alpert type, dual-filament, non-burnout, ionization gages located in the chamber for measuring pressures below 1×10^{-4} torr. These gages will be Veeco RG-75, or equal.

Filament switching for the ionization gages will be accomplished from the control room. Indications from the pressure-monitoring equipment will be used for control. Two mass spectrometers will be used to monitor gas leaks within the chamber. These spectrometers will be capable of measuring a partial pressure of 1×10^{-10} torr for gaseous species between 1 and 40 AMU. These measurements will be used as a guide.

The main control panels have facilities for activating all valves and operating all pumps. However, specific malfunction alarms for each subsystem will be summarized into one alarm at the main panel. Pressures and important temperatures are indicated and recorded on the main control panel.

5) Time vs. Vacuum Pressure Product Meter

An instrumentation system is required to provide data corresponding to the integrated loop contamination. For this purpose, an instrumentation system is envisioned which will integrate pressure vs. time during loop operation at temperatures over 500°F. The system does this by integrating the logarithmic output of the mass spectrometer. Loop thermocouples determine the integrating time.

6) Cyrogenic Systems

(1) Argon System The temperature of each thermal shroud zone is controlled by the manual regulation of liquid argon flow rate. The liquid argon temperature at each zone element is recorded on a multipoint recorder. Density detectors on the inlet and outlet of each of the liquid argon headers to and from the chamber indicate incipient two-phase flow. An electronic-pressure indicating controller maintains back pressure on the common return header from all zones to control system pressure in order to prevent two-phase flow or freeze up. If zone pressure or temperature varies beyond established test conditions, an alarm is activated.

(2) Nitrogen System A nitrogen system is used to absorb the heat from the liquid argon shroud cooling system. The nitrogen is sent from a storage and pumping system through four subcoolers. The liquid nitrogen is evaporated from 35 psia to atmospheric pressure through a control valve.

(3) Dense Gas Helium System A temperature control loop similar to the argon system control loop regulates the dense gas helium flow through each control zone. One resistance element is located at the outlet of each zone. Enough information from the helium refrigation, e.g., expander turbine inlet and outlet pressure and temperature, is transmitted to the central control room for surveillance and control of the refrigeration.

7) Leak Detection

Vacuum chamber - liquid level gages in surge tank extension pipes appear to offer the most immediately available and reliable method of detecting leaks in the lithium, potassium, and NaK loops. Another answer might be a system which incorporates a remotely controlled infrared sensitive TV camera and a viewing screen for operator observation located in the central control room. A leak would show as a change in radiation intensity against a stable background. No commercially available equipment is known to exist that can perform this function and a considerable research and development program would be necessary to assure satisfactory performance and reliability. It is considered unlikely that vacuum gages will detect liquid metal leaks due to rapid freezing on the cryopanels.

8) Emergency Instrumentation and Control

In a facility as complicated as this, it is impossible to design the controls for all emergencies. However, certain safety features will be designed into the instrumentation and controls to provide for various emergencies.

a) A power failure will require the automatic activation of a standby power supply. This power supply is described under Safety, Subparagraph V(C)(1).

b) Certain control instrumentation will contain warning lights or buzzers to indicate over or under temperature or pressure.

c) Each controlled actuator (valves, etc.) will be designed to fail open or closed on loss of power depending on its function in the system.

d) Critical control operations will be determined during the final design of the system and will be interlocked to prevent actuation at an inappropriate time.

c) A series of panic buttons will be provided on the control panel that, when energized, will sequentially shut down the facility. More than one panic button will be provided because different emergencies require different shut down procedures. The facility operator will be required to decide which shut down is appropriate after a review of the failure.

B. LOOP INSTRUMENTATION

1. Purpose

The loop instrumentation has the following purposes:

a) Loop control

b) Loop component control and performance

2. Data Gathered

Primary data, directly gathered, includes :

a) Electrical input quantities: voltage, current, power, frequency

b) Thermodynamic quantities: pressure, temperature, liquid flow rate

c) Working fluid quantities: liquid level

Secondary data directly gathered or measurable:

a) Heating fluid, working fluid and heat rejection fluid sampling and contamination

b) Infrared television presentation of system temperature distribution

c) Chamber vacuum level and contamination

3. Data Presentation, Handling and Storage

All transducers integral to the loop provide dc analogs, either directly or from associated electronics, of the measured quantities. The various quantities are displayed by meters calibrated directly in units of the quantities sensed, e.g., temperature, pressure, etc. The meters are located on two graphic display boards depicting the loop: one board for loop operation also contains the controls; the other board displays the quantities of engineering significance used by data taking personnel. Data measured on any test device(s) would be displayed by indicators added to the latter board. Where required, indicators on the loop control graphic board have adjustable limit contacts for initiating alarm and automatic safety procedures. Figure 42 shows a typical instrumentation data handling system for this facility.

All data (analog) is continuously scanned in a 4-input per second cycle, digitized in sequence and recorded in digital form on magnetic and paper tape; it is also printed out numerically on paper tape. The format of the magnetic tape recording is set up for ready assimilation by any available general purpose digital computer. Temperature and pressure data are directly utilized in system control and in alarm actuation. The output of each transducer is amplified to a voltage level in the order of one volt full scale to preclude the possibility of error due to thermal emf's or circulating currents. Each item is made available at control room jack panels; this allows convenient selection and routing of data for chart recording.

4. Loop Control

Independent quantities are automatically controlled, with the levels being manually adjustable. An example is control of heating and working fluid temperatures to fixed values through proportional servo control of heater power input. Dependent quantities, e.g., pressure, while not controlled, will cause corrective action to be initiated if preset levels are exceeded, in this case, by power reduction to the heater(s).

a. Sensing and Display

Figure 43 shows the loop instrumentation. The type and location of loop instrumentation was chosen from the standpoint of facility monitoring and control rather than for component test data. It is assumed that test components, such as boilers and condensers, will contain performance instrumentation integral with the component and be of such a type as to measure accurately the parameters desired.

b. Heaters

Although the heaters are not part of the instrumentation system, their design is influenced by instrumentation considerations (1) that practically no current circulate in the piping external to the heater and (2) that magnetic field strength be very low everywhere beyond the immediate environs of the heater conductors.

The first is accomplished by supplying current to the heater at the ends and at its center, with current flow in opposite directions about the center point so that the net voltage across the heater is zero. Cancellation of external magnetic fields comes about from opposed current flow in the two halves of the heater helix, both of which are coiled in the same direction. Heater power is controlled by the voltage level applied to the heaters, in both the primary and preheater systems. Each heater half is supplied from a separate DC generator. In each of the heater systems, one of the two generators is proportionally controlled in accordance with sensed temperature at the heater outlet; the other generator voltage is slaved to that of the temperature-controlled generator such that end-to-end voltage across the heater is maintained at zero. The principle of applying power directly to the two halves of a liquid metal loop heater, in such a manner as to prevent current flow external to the heater, is an established practice now being used in single phase ac powered systems at the General Electric Evendale facility.

c. Temperature Measurement

Measurement of all temperatures is through ungrounded-junction thermocouples immersed in wells. Within the wells and the radiation shielded volume, the thermocouple leads are insulated with BeO beads; alumina beads are used external to the radiation shielding. Experience with refractory metal loops has shown outgassing from ceramic beads not to be a serious problem.

The highest temperature encountered in the loop is nominally 2200°F and all loop temperatures within the vacuum chamber are above 1400°F. Best calibration stability in this temperature range is normally provided by the tungsten-rhenium alloy and platinum-rhodium alloy thermocouple systems. Of the two, the tungsten-rhenium alloy system is preferred, being used in the General Electric 100 KW and corrosion loops because of less chance of reactivity with columbium alloys.(1) The particular alloys used are tungsten-3% rhenium vs. tungsten-26% rhenium. Unfortunately, the tungsten-

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(1) ORNL-TM-883 High-Temperature High-Vacuum Thermocouple Drift Tests by J. W. Hendricks & D. L. McElroy.

rhenium alloy wires become brittle at the junction because of recrystallization from the welding operation. It is necessary that mechanical stress be kept from the thermocouples by anchoring the bead insulators with foil straps near the junction ends.

Experience at General Electric has indicated that frequency of failures due to brittleness is directly related to temperature cycling, particularly when the thermocouple junction is made directly to a columbium pipe wall. However, the number of failures even in the absence of temperature cycling usually becomes quite high during extended operating periods. The development of better high temperature thermocouple alloys, amendable for direct attachment to columbium or enclosure in columbium sheathing, should be considered.

At temperature pickup positions outside the vacuum chamber, stainless steel or Inco sheathed platinum vs. platinum-10% rhodium thermocouples are used in the range 1200 - 1400°F. Sheathed chromel-alumel thermocouples are used at lower temperatures since in the 1200 - 1400°F range drift is higher than with platinum vs. platinum-10% rhodium. Table 11 summarizes the thermocouple characterics.

Three identical thermocouples are located at each station and the output of each is brought to a terminal board outside the chamber. Only one thermocouple is connected into the instrumentation system at a time. The output of the three thermocouples at each pickup point are compared at intervals during loop operation and if all agree, any one of the group may be connected into the system. If two of the three thermocouples agree, both would be assumed correct, and either could be connected into the system. Finally, if all three disagree, the choice of using none or if the temperature of one compares reasonably with adjacent indicated temperatures, it might be used, depending upon circumstances. This method of sensor comparison and selection is used generally in the system where redundant pickup is employed. Experience at General Electric indicates redundant pickup to be desirable for long period operation.

TABLE II

Thermocouple Characteristics

Thermocouple Material	Signal	Applicable Temp. Range	Compatibility	Remarks
Tungsten Tungsten-Rhenium	10 $\mu v/^{o}F$	1000 to 4100^{o}F	No Problem with Columbium	Thermocouples become brittle with temperature cycling.
Platinum Platinum-Rhodium (10%)	6 $\mu v/^{o}F$	1000 to 2900^{o}F	Possibility of Reaction with Columbium above 2000^{o}F	Possibility of thermocouple contamination in a vacuum with ceramic insulators.
Platinum Platinum-Rhodium (10%)	6 $\mu v/^{o}F$	1000 to 2900^{o}F	No Problem with Stainless Steel	Low output signal & tends to drift with time.
Chromel Alumel	20 $\mu v/^{o}F$	600 to 2100^{o}F	No Problem with Stainless Steel	

d. Pressure Measurement

Currently, the most stable pressure transducer capable of continuous operation with high temperature alkali metal is the slack-diaphragm type.(1) The inert gas efflux system, equally good relative to calibration stability does not, however, measure continuously or allow simultaneous, continuous observation of a multiplicity of points. The slack diaphragm system lacks the rapid response necessary for observing fractional-second transient phenomena, and if transient data are desired, a suitable system must be developed. Although each transducer system has particular merit, the slack-diaphragm type pressure transducer is recommended for this application. For reliability, three slack-diaphragm pickups are recommended at each pressure measurement point with only one transducer active in the instrumentation system at a time.

e. Liquid Level Measurement

Liquid level is measured in the surge and dump tanks. J-tube resistance gage systems(2) are employed for reliability and economy. Two sensors are installed at each pickup point to afford some redundancy and to provide a cross checking feature. Normally, J-tube level sensors are custom designed for the application and present no significant engineering risk. Flow data are measured with permanent magnet EM flowmeters. No practical need exists for backing up the EM flowmeters with other EM flowmeters, since reliability has been proved.(3) To avoid a loss of electrical contact with the pipe wall, the connections are made redundant.

The accuracy attainable with the EM flowmeter is about ±1% when all contributing parameters are known. That actually attainable over extended periods, however, is about ±5% due to variation of the

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(1) Taylor Pressure Transmitter with NaK filled element. Taylor Instrument Company, Rochester, N. Y.

(2) Development of Liquid Metal Level Probes - NASA-307.

(3) Calibration and Testing of 2-N3.5-inch magnetic flowmeter for high temp. NaK Service. ORNL 2793.

electrical characteristics of the pipe wall, contact between it and the flowing liquid, and other factors not readily determinable for correction of indicated flow. (See Problem Areas, Section IX of this report.) Accuracy reliable to ±1% or better is reported with the ultrasonic flowmeter(1) and its use may be considered. If the ultrasonic flowmeter is used, each position should be backed up with an EM flowmeter for reliability. The EM flowmeter should be calibrated against the ultrasonic flowmeter and its data used pending failure of the ultrasonic flowmeter. Ultrasonic flowmeters are custom designed for the application; some development work would probably be necessary to provide for cooling the piezoelectric element holder.

f. Vapor Flow

No provision has been made for measuring vapor flow in the loop system, although this is a quantity useful in connection with devices under test. It is suggested that vapor flowmeters be designed appropriate to requirements of the test device. Orifice or venturi flow devices, with slack diaphragm pressure transducers, would be appropriate.

g. Quality Measurement

No provision has been made for measuring vapor quality in the system, although this is a pertinent quantity in testing developmental devices, particularly turbines. Inlet qualities would likely always be high, probably above 98%; exit qualities may be as low as 80%. For the higher range, a throttling calorimeter, now used in the General Electric 3000 KW loop, would be a good choice; it is not, however, capable of measuring qualities much below about 98%. The heating calorimeter adequately measures quality over 80 - 100% range but it is not as accurate as the throttling calorimeter at the high end of the scale. The heating calorimeter would require that a rather large heater capable of operating at 2000°F (or higher) be developed.

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(1) Ultrasonic Flowmeters by Gulton Industries, Inc., 212 Durham Ave., Metuchen, N.J.

The separating calorimeter(1) appears to be the best all-around approach in terms of wide quality range capability and simplicity. A further advantage is the fact that the liquid and vapor components of the two-phase mixture can be measured with EM flowmeters; the latter, after condensation. It is unnecessary for the liquid and vapor components to be measured to better than ±5% accuracy each for ±1/2% accuracy in final results; it is necessary, however, that separation be done precisely. Separating calorimeters are available for steam systems and it is probable that some existing design could be adapted for potassium operation. Two components, a separator and a sampler, would require development.

h. Electrical Voltage, Current and Power Measurement

All data are measured as dc electrical analogs or are translated into dc electrical analogs for convenience of indication and digitizing and, ultimately, for computer assimilation. The electrical loop heating voltage, being dc, is inherently in the desired form and merely requires dividing down in a resistor network. The measurement of current and power is through Hall-effect transducers, which provide a dc signal directly for indication, and does not require the use of current shunts or current ratioing devices. A Hall-effect single phase ac wattmeter(2) is in service in General Electric's Corrosion Loop System. If shunts were used at the current levels of the subject system, 5 KW would be lost in each for a 50 millivolt drop at 100,000 amperes. So-called dc current transformers, used in conjunction with conventional ammeters and wattmeters, would be satisfactory from the measurement standpoint but would provide no electrical analog of power for data recording and processing. A dc analog could be derived from the deflection of an electrodynamic wattmeter movement, but the overall system would be far more complex than the Hall-effect counterpart.

(1) Mark's Handbook, Lionel S. Marks, ed., p. 2112, McGraw Hill, 1941.

(2) Model 150C61-YM Wattmeter, Scientific Columbus, Inc., 1035 W. 3rd Ave., Columbus, Ohio.

Hall-effect ammeters and wattmeters are presently custom designed for the application in higher current ranges, which is the case for currents in the order of 100,000 amperes. This is not an engineering problem and requires, mainly, designing a magnetic yoke to surround the conductor through which the current flows. The Hall-effect element, a wafer of semiconductor material, operates in a gap in the magnetic yoke.

i. Signal Transmission and Display

The outputs of the various sensors vary widely as to level and output impedance. Because of this and the desirability of circuit isolation, each active sensor output is applied to a chopper type dc amplifier where it is amplified to the order of one volt. In this level range, the signals are applied directly to D'Arsonval movement meter indicators on the graphic display boards and are also of a convenient level for digitizing (or transmitting as raw analog data, if desired). Measured data, e.g., loop temperatures, pressures, flows, heater electrical quantities, etc., are displayed by meters on the graphic display boards calibrated directly in the units measured, as stated previously. The graphic display board has gained wide acceptance particularly in the process industries.

The recording of data is in digital and numerical (visual) form. The recorded data are on magnetic tape and punched paper tape; the visual numerical data are printed on 'adding machine' tape. The punched and magnetic tapes are suitable for computer data reduction operations and for data storage. This system has been in use at General Electric for some time and has proved its worth in terms of data capacity vs. cost. It would not be practical, and probably of no great advantage, to record all data simultaneously and continuously. The graphic board indicators (meters) present the quantities simultaneously and continuously, however. Digital recording is done in a sequence of four points per second, or faster. Quantities which would most desirably be taken simultaneously can be grouped together in the time sequence by connecting sensor amplifier outputs to the scanner inputs, or the jack panels, in the numerical sequence desired.

VII. LOOP START-UP PROCEDURE

VII. LOOP START-UP PROCEDURE

A. Start-Up Conditions

The loop start-up described here will be the first start-up of the system. The following conditions will be assumed:

1. All sub-systems such as generators, line heaters, argon, loop vacuum, etc., and all components such as EM pumps, valves, and flowmeters have been checked out and operate satisfactorily.
2. The vacuum chamber and associated pumping systems operate in a satisfactory manner.
3. All liquid metal loops have been installed, leak tested, line traced and insulated.

B. Operating Procedure

The liquid metal piping has been laid out in such a manner that, in order to minimize expansion stresses, all three loops (Primary, Secondary and Radiator) should be brought up to temperature at the same time. It will be necessary to have all loop piping and components within the chamber at a temperature of 400 - 500°F prior to flowing liquid argon through the cryopanels. The pipe temperature can be obtained with the line heaters. With all line and component heaters on and the chamber at an acceptable vacuum, the loops are ready for filling.

A reference to the flow schematic, Figure 7, will assist the reader in following the start-up procedure.

1. With all valves to the various loops closed and the dump tank heaters operating, the dump tanks should be alternately evacuated and back filled with argon until the tanks are free of air. The respective liquid metal may be blown or sucked into the dump tanks through the fill lines.

2. The respective loops should be alternately evacuated, back-filled with argon and evacuated to remove all air. By opening the dump valves the loops can be filled with liquid either by sucking or pressurizing liquid metal from the dump tanks. There is sufficient liquid metal in the respective dump tanks to completely fill each loop. With the respective loops held at 400 - 600°F by the line heaters, the liquid metal pumps should be started to flow liquid metal through the loops. This operation will perform the final clean up of the loop and components by using the liquid metals as the cleaning fluid. It may not be possible to bring the radiator loop temperature up to 400°F because the portion of this loop, outside of the vacuum chamber, is not line traced. By preheating the radiator cooling air with the steam heat exchanger and controlling the radiator liquid metal flow at a low value, the temperature of this loop can be held at a sufficient temperature for flushing and proper pumping.

3. The respective loops should be dumped, after flushing, and the liquid metal hot trapped in the dump tanks. Samples of the liquid metal may be taken from the dump tank and analyzed for purity after hot trapping. A method of extracting a sample for analysis is described in the Appendix. The first dumping of the respective loops, that occurs during this cleanup operation, will be a good time to record the time required to empty the loops of liquid metal.

4. The primary loop should be refilled with lithium at 400 - 600°F to a low level in the surge tank. The primary pump should be started and power applied to the primary heater. Continuous monitoring of the liquid level gage in the surge tank will be required because, as the lithium is heated, it will expand and may tend to flood the surge tank. If additional lithium is required in the loop it may be blown in from the dump tank. If lithium must be removed from the loop it may be returned to the dump tank by opening the equalizing valve and jogging the dump valve. Cover gas pressure is applied and maintained on the liquid metal through the argon valve above the surge tank.

5. The radiator loop is filled in the same manner as the primary loop. As mentioned previously, all loops should be brought up to operating temperature at the same time. This requirement will necessitate either extensive automatic control on the heaters and radiator or three groups of operators (one group per loop) working together. By flowing small quantities of NaK and by flowing the radiator air through the by-pass valve provided, the radiator loop can be made to follow the secondary loop up to design temperature.

6. The secondary loop is probably the most difficult to start because of the absence of a simulator inlet valve, the presence of two-phase flow and the horizontal boiler and condenser. The secondary loop should be filled to an indicated level in the head tank. With the potassium at a temperature of 500 - 600°F the pump should be started and potassium pumped around the loop. Potassium will have to be fed from the dump tank to the loop to maintain a level in the head tank. After a period of time at a given pumping speed, the level in the head tank will stabilize. The temperature of the liquid potassium should be brought up either by heat transfer from the boiler or heat input from the secondary I^2R heater. A cover gas pressure should be maintained on the potassium liquid to suppress boiling during heatup.

   At a predetermined temperature of the potassium liquid the cover gas pressure should be reduced slowly. The lowering of the cover gas pressure will cause boiling of the potassium. By slowly applying additional heat and monitoring the head tank level the secondary loop can be brought up to design temperature. This method of startup of the secondary loop has the following disadvantages: (1) large temperature changes can occur in the piping during the time of initial boiling and (2) the simulator may experience slugging from potassium liquid until sufficient heat is applied to the boiler to dry out the vapor.

VIII. SCHEDULE

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VIII. SCHEDULE

The Test Facility Schedule is shown in Figure 44. Starting with the material selection, the total elapsed time required before the test loop can be filled with liquid metal is 48 months. This schedule allows sufficient time for preliminary engineering and the solution of all anticipated problems, except for the basic feasibility study required to determine the outgassing rates of materials to be used in both the loop and chamber construction. As described in Problem Areas and Development Programs, Section IX, this problem must be solved and understood before proceeding with the loop and chamber design. A more detailed review of the schedule follows.

A. Material Selection

The 48 month time cycle begins with the final selection of the high temperature, liquid metal loop containment material. Based on present data, FS-85 columbium alloy is recommended. The material selection is necessary at this point since both the preliminary design work and the development programs must be based on this choice.

B. Development Programs

The work to be performed in the Development Programs is covered in Problem Areas and Development Programs, Section IX. A supplementary schedule, Figure 45, shows the interrelationship of these programs. Some of the questions raised by these programs may be settled before work on the Test Facility has begun, in which case the work would not be considered part of this effort.

C. Chamber

The overall time schedule for the chamber is 34 months. At this point it could be checked out clean, dry and empty. This schedule is based on releasing the engineering and construction work on a phase basis. The partial packages selected are as follows and as shown in Figure 44.

1) Demolition. Selective removal of equipment and structure to clear the site for new construction.

2) Buildings. Includes subgrade work and superstructure, building foundations, major equipment foundations, cable and pipe trenches, and building floors (less high-bay mezzanine).

3) Vacuum Chamber. Includes structural modification to east leg of Space Power Chamber to provide new support, access, reinforcement, penetrations, inner chamber, and test loop supports. Removal of present interior coating of chamber and recoating is also included.

4) Long Lead Equipment Procurement Package. Includes selected items of equipment for which procurement specifications can be prepared early in the final design period. Such equipment involves, in some cases, substantial detail design on the part of vendors.

Item	From time of award, months
(1) Reduction gear (speed increaser)	12
(2) Acyclic generators	12
(3) Helium refrigerators	9
(4) Argon subcoolers	6-9
(5) Ion pumps	9
(6) Gas storage tanks	9

5) General, Mechanical, Electrical, Piping, Building Completion. Includes responsibility for all functions not specifically assigned in the preceding packages, the procurement of material, the installation and checkout of all components, equipment, systems controls, instrumentation, and the completion of the buildings.

D. Loop

The Test Loop Schedule is tied closely to the Development Program Schedule and the Chamber Schedule. (Refer to Figure 44 and 45 respectively.)

1. Preliminary Design - 6 months

   The preliminary design will cover all of the basic conceptual design work including the study and analysis necessary for optimization of the loop and chamber design. Material would be selected based on results of the outgassing development program. Material handling techniques would be finalized and integrated with the chamber design.

2. Component and Loop Design - 18 months

   After completion of the Preliminary Design phase, the detailed component and loop design would begin. This design phase would take 18 months with specific long lead procurement items released ahead of that time as required. A thorough and final loop stress analysis would be made early in this phase of the schedule.

3. Material Procurement - 9 months

   The 9 month material procurement cycle overlaps considerably with the Final Design phase of the program but, more importantly, it bears a direct relationship to the Development Program phase. The schedule concept presented here calls for processing and manufacturing techniques to be developed during the Development Program phase of this facility. Materials and Components being procured (to be included in the loop) will be the second or third part produced rather than the first part produced with all of the normal, time requiring, development effort.

4. Installation, Leak Check and Insulation - 19 months

   The installation schedule is based both on the completion of the fabricated components and the availability of the test chamber. The techniques required for field welding will have been fully developed.

   Leak checking of the entire loop will be required before the insulation is applied to the loop piping and components.

5. Check-Out - 4 months

A check-out period of 4 months is provided before filling the loop with liquid metal. This point is cited as the completion of the project even though it is fully realized that a continuing check-out period will be required. It is a significant point, however, in that all systems must be operative before filling the loops with liquid metal.

IX. PROBLEM AREAS AND DEVELOPMENT PROGRAMS

IX. PROBLEM AREAS & DEVELOPMENT PROGRAMS

During the course of this study contract several problem areas and possible problem areas were uncovered. A problem area is defined as an area that could affect basic feasibility or an area in which a separate development program should be undertaken to obtain basic design information prior to or in the early stages of the design of the system. A possible problem area is defined in this report as an area that:

1. Will require a more detailed analysis that is beyond the scope of this study.
2. Will affect the economics of the design and/or operation.
3. Will affect the life or endurance capability of the facility.

The solution of possible problem areas should evolve during the course of the preliminary design of the facility or through separately funded development programs.

A. Problem Areas Affecting Basic Feasibility

In a high vacuum system, the pumping required is largely a function of the surface area and type of material within the chamber. The surface area of the materials of the loops and reflective foil used for insulation is estimated to be 50,000 sq. feet. If all the surface area were taken as stainless steel, the pumping speed for condensable gases after 100 hours per Figure 3, at 5×10^{-9} torr, would have to be 14×10^{6} liters per second. If the outgassing rate for stainless steel is extrapolated to an average temperature of 1000^{o}F, which would more nearly approximate the operating temperature of all the stainless, from Figure 2, the pumping speed would have to be 70×10^{10} liters per second. The pumping speed available at 5×10^{-9} torr for condensable gases is 3.3×10^{6} liters per second. Further study is required to optimize the vacuum and cryogenic systems in conjunction with further refinement of test loop and component outgassing characteristics. Beyond some optimum point, the cost of pumping increases sharply and, in the case of the Space Power Chamber, the feasibility of adding pumps to the structure becomes questionable. There are two requirements that have conflicting solutions and continued study is needed to reach a satisfactory compromise. First, a particular environment must be developed. This requires free circulation to permit pumping of outgassed materials. Second, the loop must be adequately insulated so that temperature losses will not develop to degrade loop performance.

The particular environment required is a function of the chamber pumping capacity. The insulation of the liquid metal loops and components affects not only heat loss but also loop outgassing, chamber pumping capacity and environment. To reduce the heat loss from the liquid metal loops within the vacuum chamber, the pipes will be wrapped with a reflective foil, as many as 20 layers on some of the pipe that operates at high temperature.

A possible solution might be the use of columbium foil as insulation. Current literature and experience show that hot columbium will pump certain gases (O_2, N_2 and CO_2) that are pump speed limited by the present cyro pumping system. The columbium foil on the outer surfaces may not run at a temperature high enough to pump the marginal gases. These outer layers of insulation may have to be canned to prevent contamination of the loop from their outgassing products.

A canned foil insulation may present additional problems. For the canning to be effective, as far as outgassing is concerned, the cans must be made leak tight. The forming, welding and leak testing of the canned insulation will be expensive. For the canning to be effective, as far as vacuum insulation is concerned, the vacuum in the cans must remain high. If the can is not evacuated and sealed in the hot condition, outgassing of the foil within the can may reduce insulating effectiveness when the can temperature is raised during loop operation.

In summary, before a final design of this facility can be initiated, the problems of material outgassing must be resolved.

B. Possible Problem Areas Affecting Cost, Facility Life or Endurance Testing

1. Material Handling - A possible problem area is the ability to install, service, and remove components of the various loops in and from the chamber. The handling of the components in and out of the chamber will be difficult due to the fact that (1) the working volume for the loops has been dictated

by the volume in the chamber remaining after all cryopanels are installed, (2) flexibility must be provided in the piping to reduce stresses and reactions,and (3) large components of workhorse variety are required initially. To minimize downtime during change-over of components, certain compromises will have to be made in the chamber and loop design and extensive tooling and material handling equipment will be required. The chamber modification and loop layout should proceed hand in hand to minimize the material handling problem. A scale model of the chamber and loop should be constructed to study material handling problems.

2. Valves - Valves are a possible problem area. Stainless steel valves operating in liquid metal loops at 1500° - 1600°F are a reality but their life is short and mortality rate high. A columbium alloy valve operating at the same temperature, or higher, and in a vacuum atmosphere may incur more problems than its stainless steel counterpart. Problem areas that should be studied are:

a) Seizing due to self welding or high friction.
b) Erosion due to liquid metal flow over the seat and plug.
c) Bellows failure due to pressure and cycling.
d) Leaking due to thermal distortion and wear.
e) Actuation and control due to the high vacuum environment of the valve.

The development of liquid metal columbium alloy valves will probably have to be a joint venture between a competent valve manufacturer and someone familiar with liquid metals, columbium fabrication and welding , and the friction characteristics of materials in a liquid metal environment. The final valve selection should be made only after extensive testing under actual operating conditions.

3. Altitude Wind Tunnel Structure - The structural modification and conversion of the Altitude Wind Tunnel requires further study. The tunnel is presently in use as a vacuum chamber for Centaur and its feasibility for that purpose is established. For the proposed use, however, additional

supports are required for the new loads imposed by ion pumps, the inner chamber, and test loops. The following should be considered in greater detail:

a) Reinforcement of existing structure to accommodate new penetrations required for ion pumps, access door, electrical feedthroughs, etc.

b) Relative thermal expansion resulting from weather and cryogenic systems and from test loop heat between the inner and outer chambers and between the outer chamber and fixed installations.

c) Coatings for the interior surface of the tunnel or outer chamber to provide rust protection, reflectivity and low outgassing.

d) Support and mounting of the test loop, loop components, and dump tanks in the inner chamber, giving consideration to the problem of thermal expansion, the location and arrangement of piping and other auxiliary systems.

4. High Current Bus Connectors - High-current bus connectors and feedthroughs require additional study. Arrangements must be considered for providing structural supports to the lithium and potassium heaters to avoid loading the soft copper conductors. Supports must also be provided to the bus external to the chamber and relative movement of inner and outer chambers must be considered. Electromagnetic forces must also be considered.

5. Status of Refractory Alloys - The preliminary design study was conducted with F S-85 alloy as the typical material. Although documentation of the metallurgical characteristics and design data are rather limited for this alloy, experimental programs are underway to obtain much of the information which will eventually be needed to conduct a detailed facility design. Fabrication of the materials to construct the 15 MW facility will require a scale-up program to produce the basic mill products and to join large sections. The fabrication and installation of the components of the 15 MW facility will require large vacuum annealing furnaces and fixed position welding techniques.

Development programs to assure the capability of producing the basic mill products would require about one year, and joining programs to establish procedures, inspection techniques, and specifications would be conducted concurrently and end about six months later. The investment in new capital equipment on the part of industry should not exceed about one and one-half million dollars, and this could be considerably less, depending upon the alloy selection, final facility design, and time at which the program is begun. The major expenditure would be required for larger vacuum annealing furnaces, with increased melting capacity and special welding facilities requiring much smaller expenditures. On the average, columbium alloy costs would be less than $50 per pound and tantalum costs less than $100 per pound. The materials procurement could be completed in about two years after beginning the scale-up development programs.

The scale-up development program should begin with a study of the effects of heat treatment on the phase morphology of the alloy. The extent of this study would depend upon the complexity of the alloy selected and the knowledge of its behavior at the time the program is initiated. In general, it has been assumed that considerable documentation of the selected alloy, its mechanical properties, weldability, thermal stability, and behavior in alkali metals, will be obtained from existing programs and such basic work will not be required as a specific part of 15 MW facility program. Scale-up should include melting, extrusion, forging, rolling, bending, and tube reducing optimization studies.

The joining scale-up program should include studies of thick section welding, joining tubes to headers, fixed position pipe welding in the field, and inspection procedures. It is assumed that the refractory alloy will require a post-weld heat treatment, and procedures for annealing 6.25-inch diameter piping in the field should be established. Diffusion bonding should be investigated as the method of joining the 6.25-inch diameter containment piping to the 9 and 15-inch diameter electrodes. In order to make the bimetallic loop transition joints, present methods of joining Cb-1Zr tubing to stainless steel tubing should be extended to include the stronger refractory alloys in sizes up to 6.25 inches in diameter.

As a final part of the scale-up program, specifications should be prepared for the procurement of materials and for the fabrication procedures. Examples of such specifications, which are used at General Electric for the procurement of the FS-85 alloy and the welding of columbium alloys, are included in the Appendix.

6. Stainless Steel-Bimetallic Loops - Present plans are to use NaK filled, Type 316 L SS loops to remove heat from the 15 MW facility. This results in bimetallic loops which present materials problems that will require detailed consideration in the final design. Interstitial elements will transfer from Type 316 L SS to the refractory alloy, resulting in contamination levels easily in excess of 1,000 ppm. The refractory alloy ductility will be decreased, and the ability to make welds for repairs and modifications may be greatly reduced. Control of the alkali metal purity becomes particularly difficult and critical. Operation at low temperatures, below the point where the refractory alloy can serve as an effective getter and purify the NaK, could lead to extensive mass transfer of the refractory alloy as a result of the presence of impurities such as oxygen in the NaK. In order to make the bimetallic loop transition joints, present methods of joining Cb-1Zr tubing to stainless steel tubing should be extended to include the stronger refractory alloys in sizes up to 6.23 inches in diameter. The ability of these joints to absorb pipe loads and moments should be investigated.

7. <u>Decontamination</u> - It is anticipated that the possible leakage of alkali metals and the need to decontaminate the vacuum chamber and loop hardware may require new procedures in addition to mechanical removal, distillation, and chemical leeching, with the usual concern to avoid the hydrogen embrittlement of refractory alloys. In particular, the wide distribution of even small amounts of alkali metal over the cryogenically cooled vacuum chamber walls could be troublesome because of the large surface area involved. The current practice with smaller test facilities is to use bakeable vacuum chambers with water cooling coils, which usually makes cleaning easy after small potassium leaks because the potassium accumulates along the cooling coils. However, for cryogenically pumped systems which cannot be baked, it would be desirable to convert the alkali metal to more easily handled compounds by controlled gaseous reactions. For example, conversion of the alkali metal to its carbonate by the controlled addition of water vapor and carbon dioxide should be considered. It is recommended that the kinetics of such reactions be examined experimentally in order to develop additional decontamination procedures specifically for the 15 MW facility.

8. <u>Leak Detection</u> - A problem area that will require additional study is the detection of liquid metal leaks in the piping or components within the vacuum chamber. Large liquid metal leaks can be detected by:

a) A loss of pressure within the liquid metal loop or component.

b) A rise in chamber vacuum or chamber heat load.

c) A lowering of liquid level in the head or surge tanks.

Small liquid metal leaks may go undetected until they become large leaks for the following reasons:

a) The primary and radiator loop have argon gas as a cover gas to maintain pressure and the secondary loop is a two-phase loop. Small leaks will not reflect in a significant pressure change.

b) The liquid nitrogen and helium cryopanels have large pumping capacities for liquid metal vapors so that a loss of vacuum may be undetectable.

c) Liquid level gages in the head and surge tanks, which have large surface areas, may not be sensitive enough to detect level chances associated with small liquid level leaks.

d) A visual observation of the loops during operation is impractical because of the limited number of sight ports available. An added handicap is the fact that there will be no tell-tale smoke associated with the leak.

e) A continuous monitoring system for traces of liquid metal in the pumped gases of the vacuum chamber may not detect the leak. Since the leaking flow will be molecular and the inside of the vacuum chamber is lined with cryopanels with a high sticking factor, the leakage may never find its way to the detector. A suitable system for detecting small liquid metal leaks in large vacuum chambers should be developed.

9. Expansion - A possible problem area could arise due to the high and changing temperature of the pipes. The expansion associated with the changing temperature may cause (1) openings to occur in the insulation, (2) pipe reactions and resulting high loads and stresses.

a) Because the insulation around the pipe runs cooler than the pipe wall, it is conceivable that gaps may occur in the insulation. If the hot pipe radiates its heat directly to the liquid argon cryopanels, the resulting heat load to the panel may cause the argon to vaporize in the cryopanel and cause vapor lock.

b) The thermal expansion of the loop piping and the associated loads and stresses presents a possible problem that is complicated by the following:

   (1) The I^2R heater feedthroughs are copper and can take very little mechanical load.

   (2) The present vacuum chamber is not capable of absorbing high loads locally.

   (3) Pipes and high current feedthroughs must penetrate the chamber as high vacuum seals.

   (4) The chamber is a double wall construction and the inner and outer walls do not move in the same direction at the same time.

   (5) Loop expansion and resulting forces and movements change with changing operating conditions of the cycle.

10. Pumps and Flowmeters - Pumps: The pumps proposed for this facility, both inside and outside of the vacuum chamber, are electromagnetic polyphase induction pumps. The EM pumps for outside of the vacuum chamber should present no new problems as pumps of this type have been built and operated successfully for long periods of time. The EM pumps for inside of the vacuum chamber (primary and secondary loop pumps) could present problems. The electromagnetic portion of the design lends itself well to calculations and should present no problem. The structural portion of the design will require additional study because (1) conventional polyphase motor material is contemplated and therefore will have to be canned for the vacuum atmosphere, and (2) the present thinking is to have the stator offer structural support to the pump duct.

A fabrication problem may arise due to the welding of columbium alloys but this problem is common to the other components of the loops and must be solved independently of the pump. The thermal design is unique only from the standpoints that an inert gas is proposed for cooling and the temperature is high compared to existing technology. Large flow, medium head, canned pumps operating with water cooling have been built for the Sea Wolf program and small flow, high head, cooled pumps operating in a vacuum have been studied (NASA-CR-54019) but high flow, high head, refractory alloy pumps operating in a vacuum at high temperatures have not been built at this time.

Flowmeters: The possible problem areas associated with the proposed electromagnetic flowmeters are:

a) Pole face cooling.

b) Pipe to magnet gap.

c) Calibration.

The magnets used in the proposed flowmeters are permanent magnets whose pole face should operate below 800°F. In order to avoid the cost of auxiliary cooling of the pole faces it may be possible to insulate between the hot pipes and the pole faces and take the reduced output signal inherent with this design.

The gap between the flow measured pipe and the pole face of the magnet must remain constant to prevent errors in flow readings. To avoid a possible reading error the magnet must track the pipe through any movement due to temperature and loads.

The calibration curves for the proposed flowmeters will be quite accurate under ideal conditions, but the conditions under which they will operate in the vacuum chamber will be far from ideal. Changes in pipe, pole, and lead wire temperature and changes in air gap will cause deviations from the standard calibration curves. It is suggested that a system be devised which will permit flowmeter calibration at operating temperatures prior to operating the loops for test data.

11. Sliding Friction - The present thinking on the loop design is to fix the horizontal boiler in the chamber and allow the simulator and condensers to slide in a horizontal plane during expansion. Any relative movement between components and mounting structure, whether it is linear or rotational, will present a sliding friction problem. The sliding friction problem is complicated by the difference between breakaway and sliding frictional values and the self welding tendencies of material in a high vacuum.

Fortunately, the sliding velocities of the friction points are low and with proper design the unit loading and surface temperature can be maintained at a low value.

12. Vacuum Pump Back Streaming - It is impossible to eliminate backstreaming completely; however, it can be reduced to a very low level. Backstreaming can occur from the mechanical pumps, the Roots-type pumps, and the diffusion pumps. The backstreaming from any source is pressure dependent.

In the high pressure of viscous flow region, diffusion gas sweeping occurs and hence contributes very little to the backstreaming. In the transition region, backstreaming is most significant. For the roughing system this would occur from about 10 torr to 10^{-2} torr. It is difficult to trap backstreaming gas in the transition region by mechanical methods because the mean system dimension is so small that it would limit the conductance. Therefore, some other technique must be used. Backstreaming will occur from the diffusion pumps in the 10^{-2} to 10^{-4} torr region and operation in this pressure region should be limited. Liquid argon cooled, optically dense baffles will be used to reduce backstreaming in the molecular region.

The proposed vacuum system with its baffles and traps should have very low backstreaming. The amount of backstreaming and the effects of this backstreaming on loop performance can only be obtained by future testing programs. It is felt that tests of this nature should be undertaken by NASA prior to the design of the facility. It is suggested that the existing roughing and diffusion vacuum pumping systems be tested for backstreaming either with or without the addition of the liquid argon cooled baffle. If the backstreaming is appreciable, steps should be taken to do further testing with the ion baffle control and other liquid argon cooled baffles.

13. Thermocouples - The tungsten - 3% rhenium vs. tungsten 26% rhenium thermocouple system is mechanically deficient because of brittleness. The tungsten - rhenium alloy wires unfortunately become brittle at the junction because of recrystallization from the welding operation. It has been the experience at General Electric that frequency of failures is directly related to temperature cycling, particularly when the thermocouple junction is made directly to a columbium pipe wall. However, the number of failures even in the absence of temperature cycling usually becomes quite high during extended operating periods. The development of better high temperature thermocouple alloys, amendable for direct attachment to columbium or enclosure in columbium sheathing, should be considered.

C. Development Programs

Any problem area that affects the basic feasibility of the facility will require a development program. In this case the development program should provide sufficient information to evaluate the feasibility or non-feasibility of the proposed design concept and, if feasible, develop the information into useful forms for design data.

The possible problem areas, listed under Paragraph B above, fall into two categories: the first requiring separate development programs to obtain useful design information; and the second only requiring more design effort, which would follow in a preliminary design phase of the overall facility design. The decision, as to whether or not a possible problem area will require a development program, is not clear cut. Many of the possible problem areas may be solved by the continuing development programs being conducted in industry and at NASA today.

In the course of this study the development programs that were felt to be the most important from the standpoint of building this facility in the near future are listed below. The relative timing of the development programs appears in Figure 45.

1. Material Outgassing - A development program should be undertaken to establish the following:

   a) Outgassing data for columbium foil with temperature and time as the parameters.

   b) Outgassing data for stainless steel at 500°C (932°F) and above with particular emphasis on time temperature phenomenon and interstitial element diffusion.

   c) The effectiveness of canned insulation as a thermal barrier at high temperature.

   d) Insulation costs with method of manufacture, heat loss, outgassing and conductance as parameters.

2. <u>Chamber Preliminary Design</u> - A development program should be undertaken to consider in greater detail:

a) The reinforcement of the existing chamber to accommodate new penetrations and loads.

b) Thermal expansions resulting from weather and cryogenic systems.

c) Coating to reduce outgassing for the inside surface of the outer chamber.

d) Supports and mounting of the test loop in the chamber and the resulting loads.

e) Decontamination of the chamber in the event of a liquid metal leak.

f) Leak detection within the chamber.

g) High current feedthroughs.

h) Motor drive systems.

3. <u>Status of Refractory Metals</u> - A development program should be undertaken to do the following:

a) Assure the capability of producing the basic mill products.

b) Study the effects of heat treating on the phase morphology of the alloy.

c) Assure the capability of rolling, bending, welding and heat treating the refractory alloy in the sizes and shapes anticipated for this facility.

d) Prepare specifications for the procurement of materials and for the fabrication procedures.

e) Procure materials for trial fabrication.

f) Determine the tolerable limits of refractory alloy contamination with respect to welding under constraint.

4. Valves - A development program should be undertaken to study:

   a) Seizing due to self welding or high friction.

   b) Erosion due to liquid metal flow over the seat and plug.

   c) Bellows failure due to pressure and cycling.

   d) Leaking due to thermal distortion and wear.

   e) Actuation and control due to the high vacuum environment of the valve.

5. Diffusion Pump Backstreaming - A development program should be undertaken at NASA-Lewis to do the following:

   a) Determine backstreaming data on the existing chamber pumping system at various vacuum levels and for varying times at vacuum.

   b) Determine the improvements in backstreaming with the addition of baffles, both liquid argon cooled and ion baffles.

   c) Determine the deleterious effects of backstreamed pump products on the loop material.

ILLUSTRATIONS

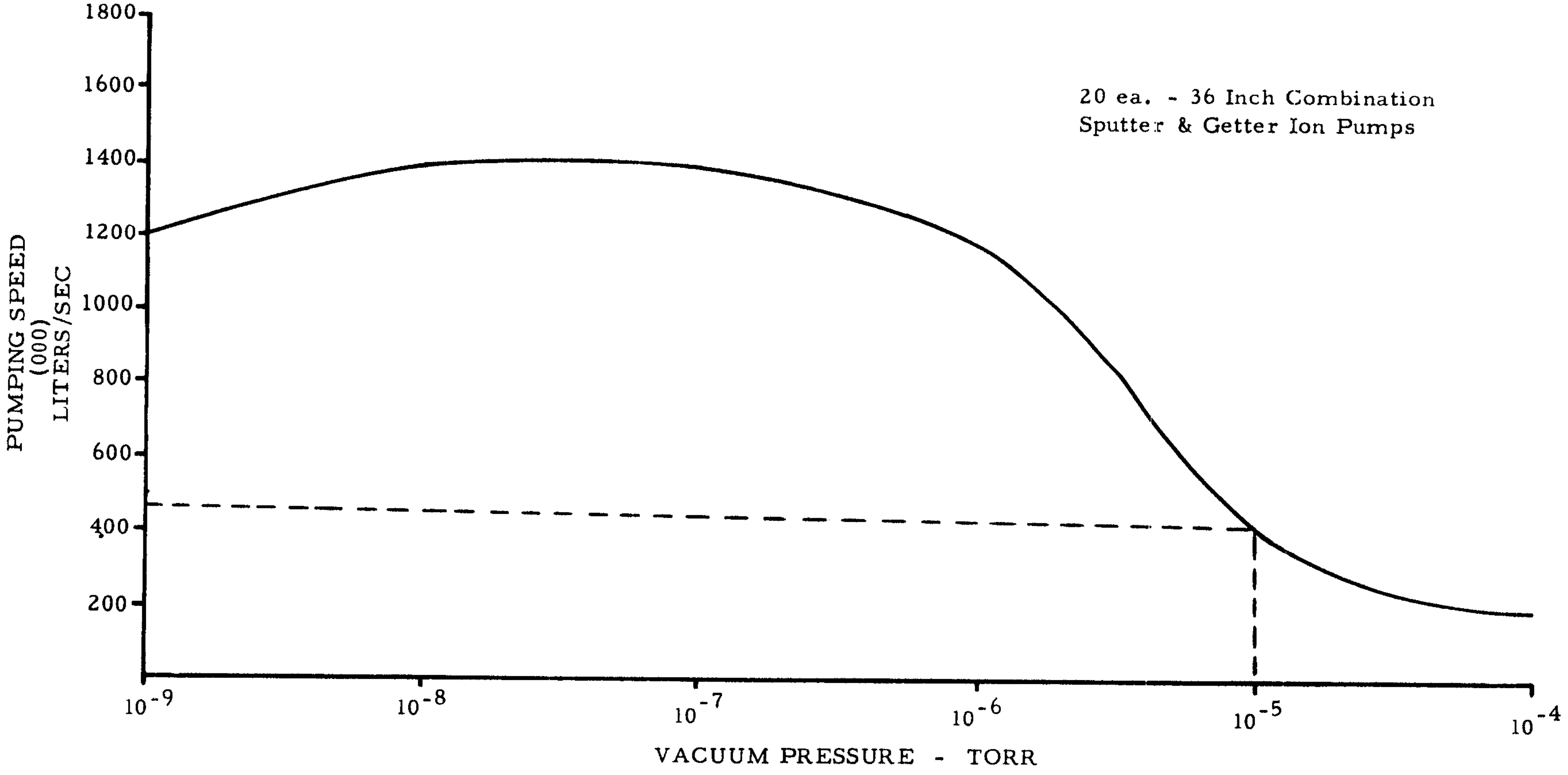

Figure 1. Hydrogen Pumping Speed vs Vacuum Pressure.

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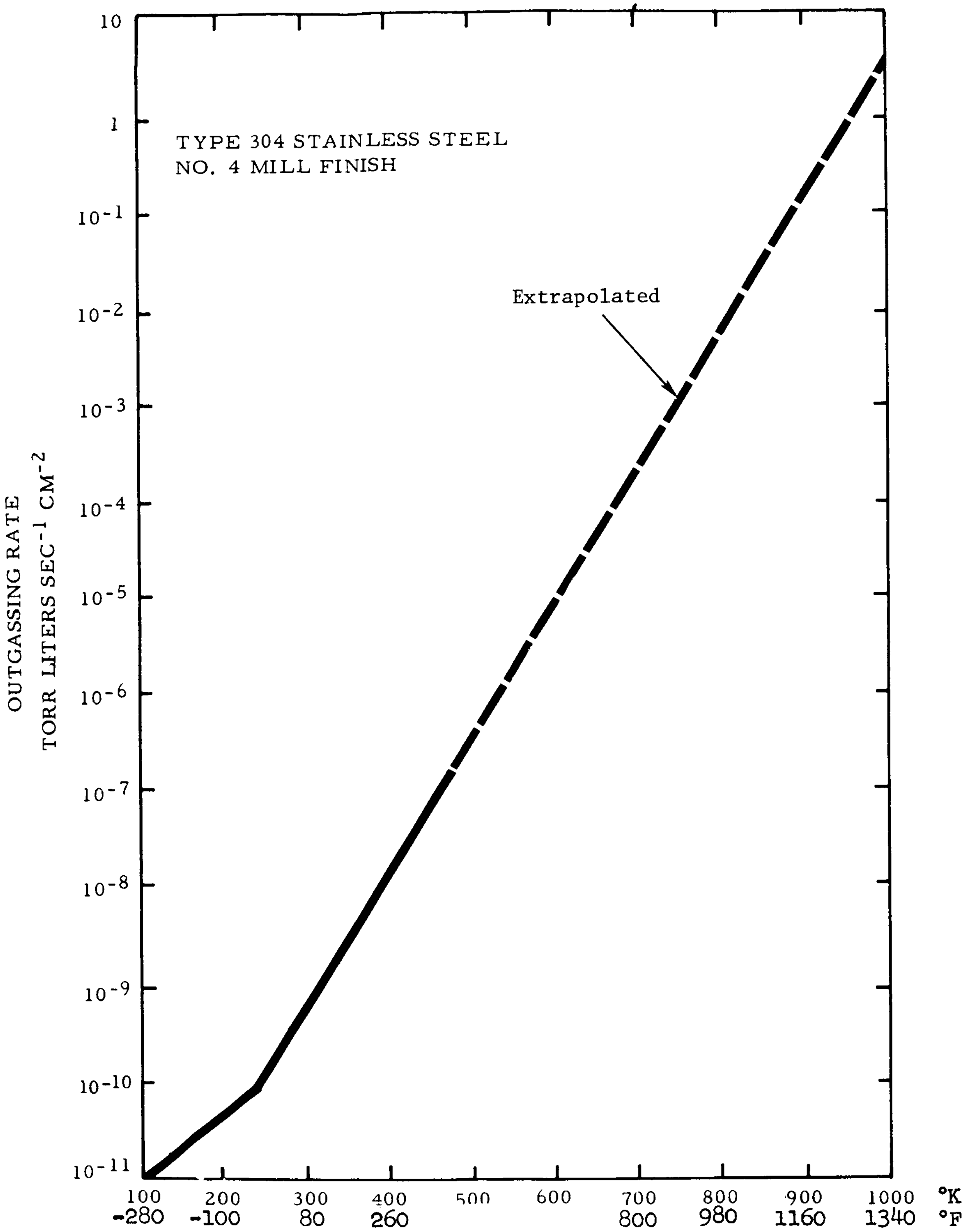


Figure 2. Stainless Steel Outgassing Rate vs Temperature, 24 Hour.

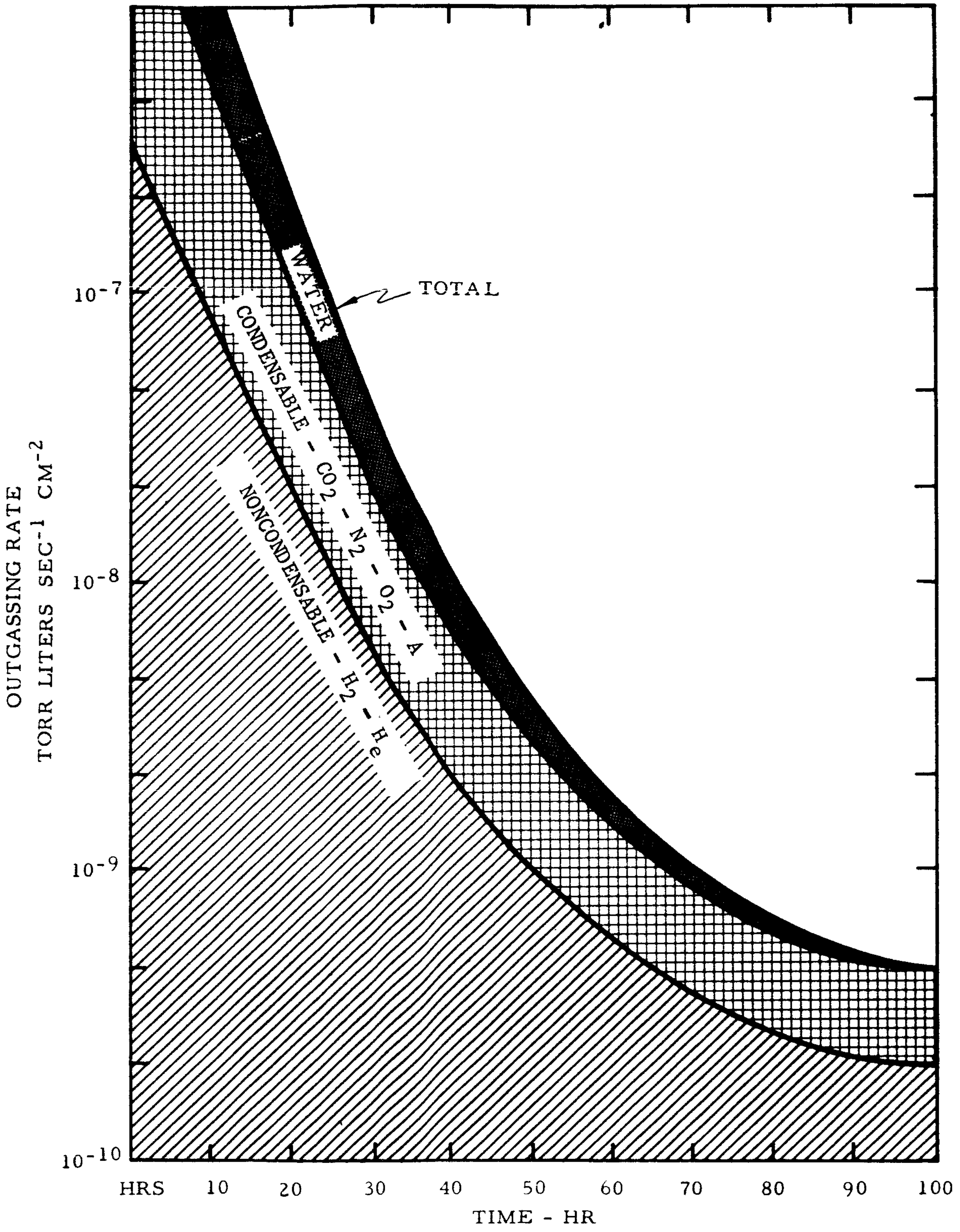


Figure 3. **Stainless Steel Outgassing Rate vs Time, 500°K (440°F).**

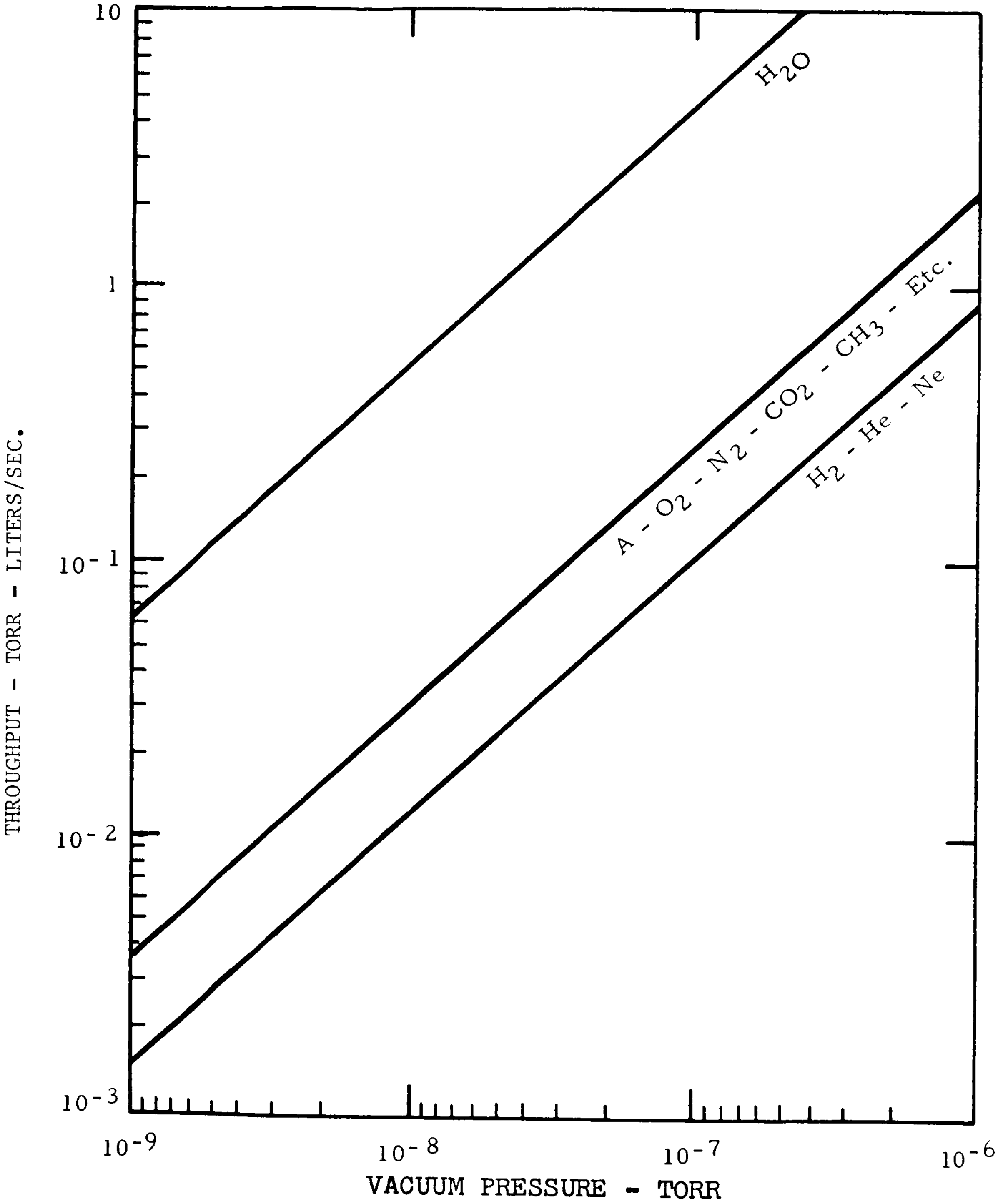


Figure 4. Pumping Rate vs Vacuum Pressure.

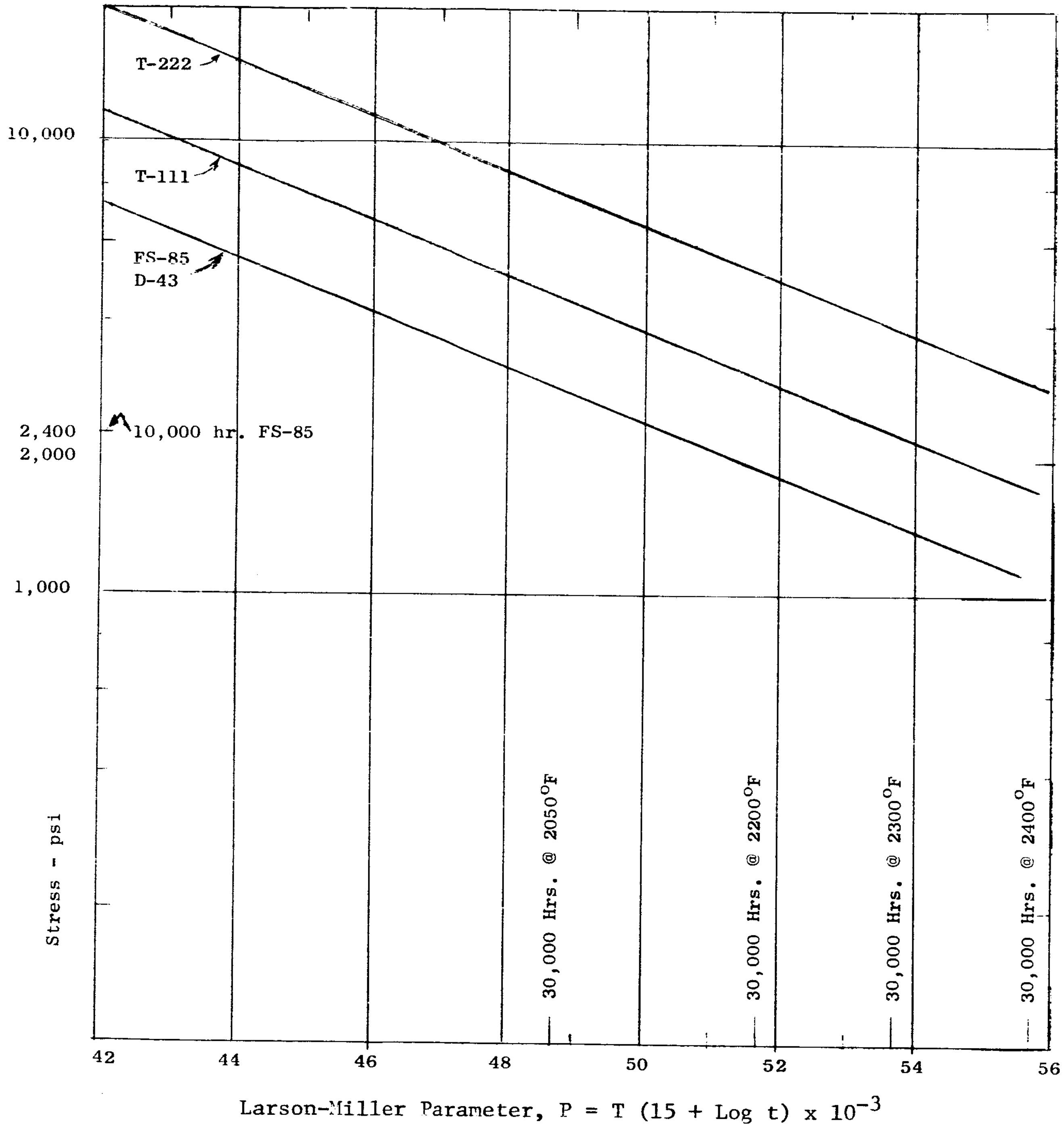
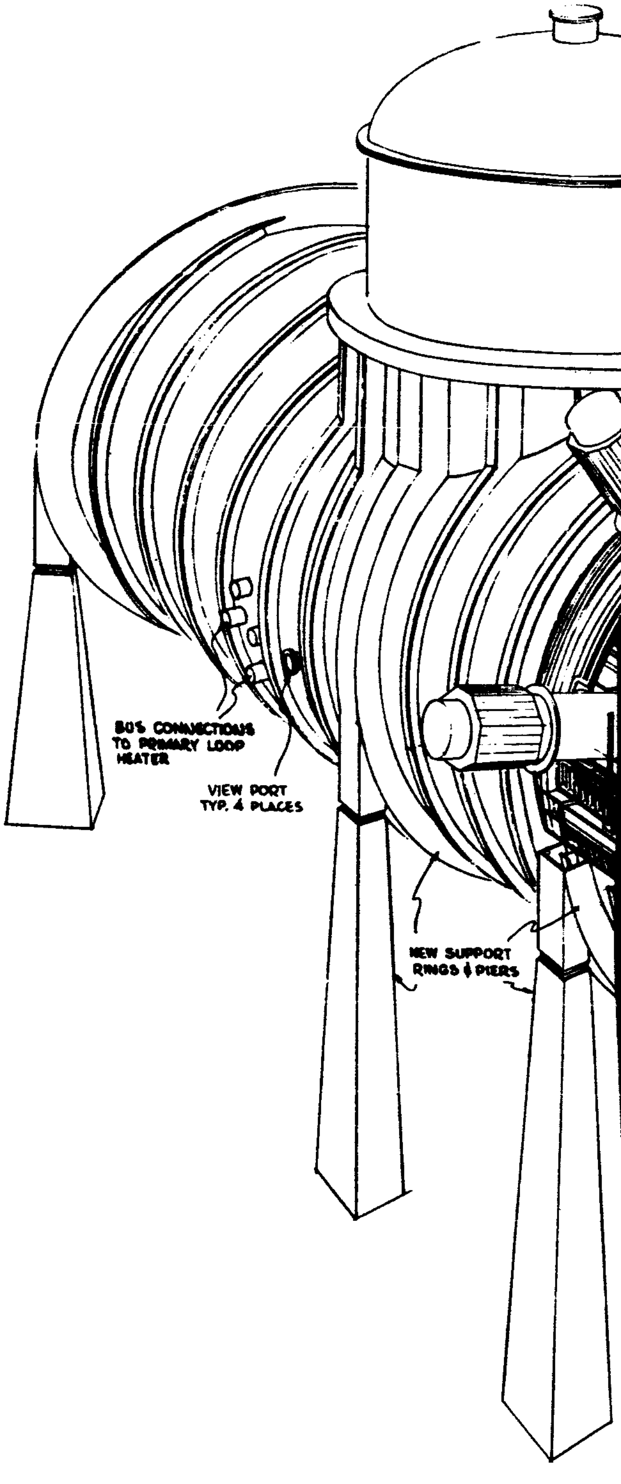
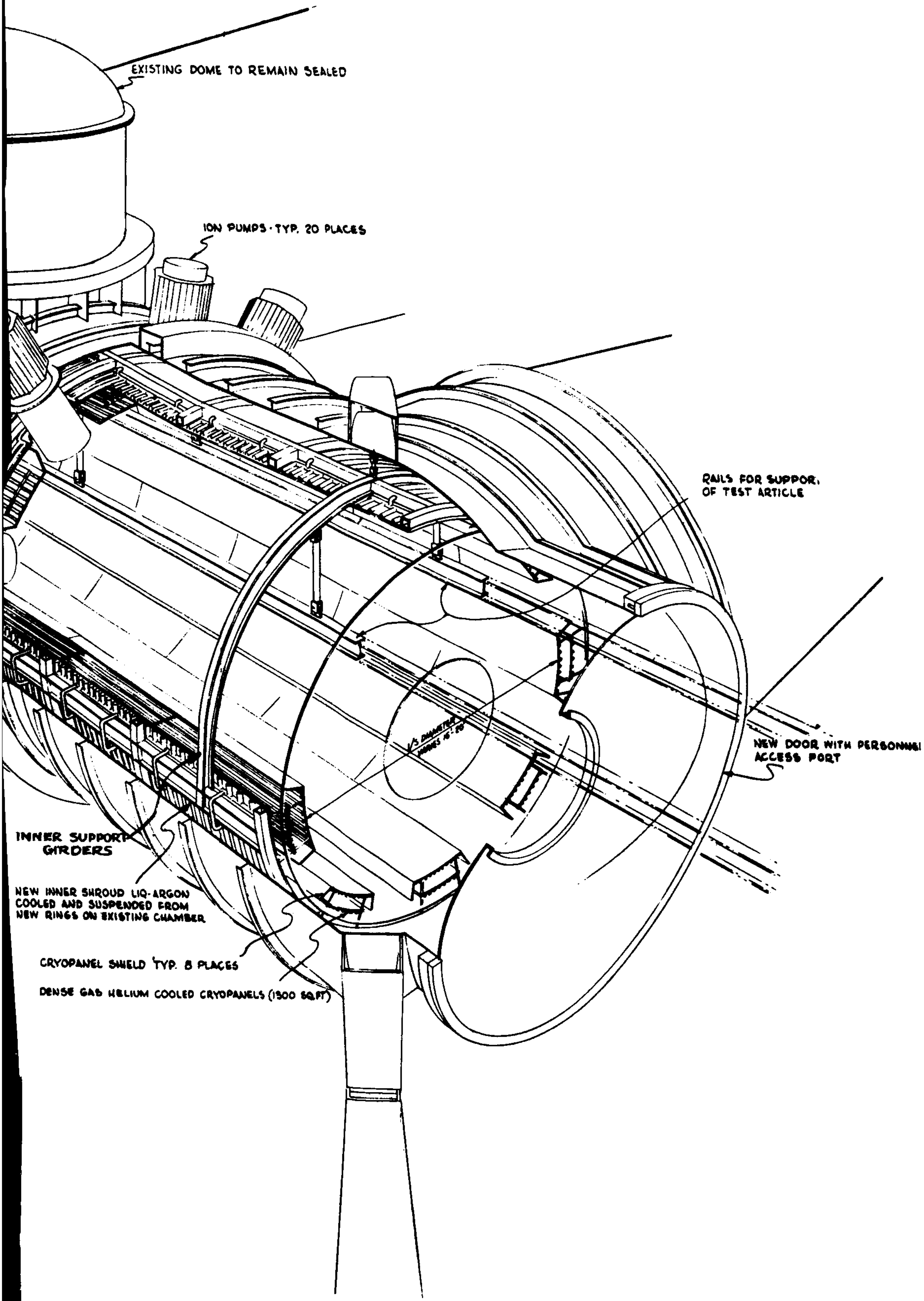


Figure 5. Estimated Average 1% Creep Strength for Columbium and Tantalum Alloys.


BUS CONNECTIONS TO PRIMARY LOOP HEATER
VIEW PORT TYP. 4 PLACES
NEW SUPPORT RINGS & PIERS



igure 6. Chamber Perspective.

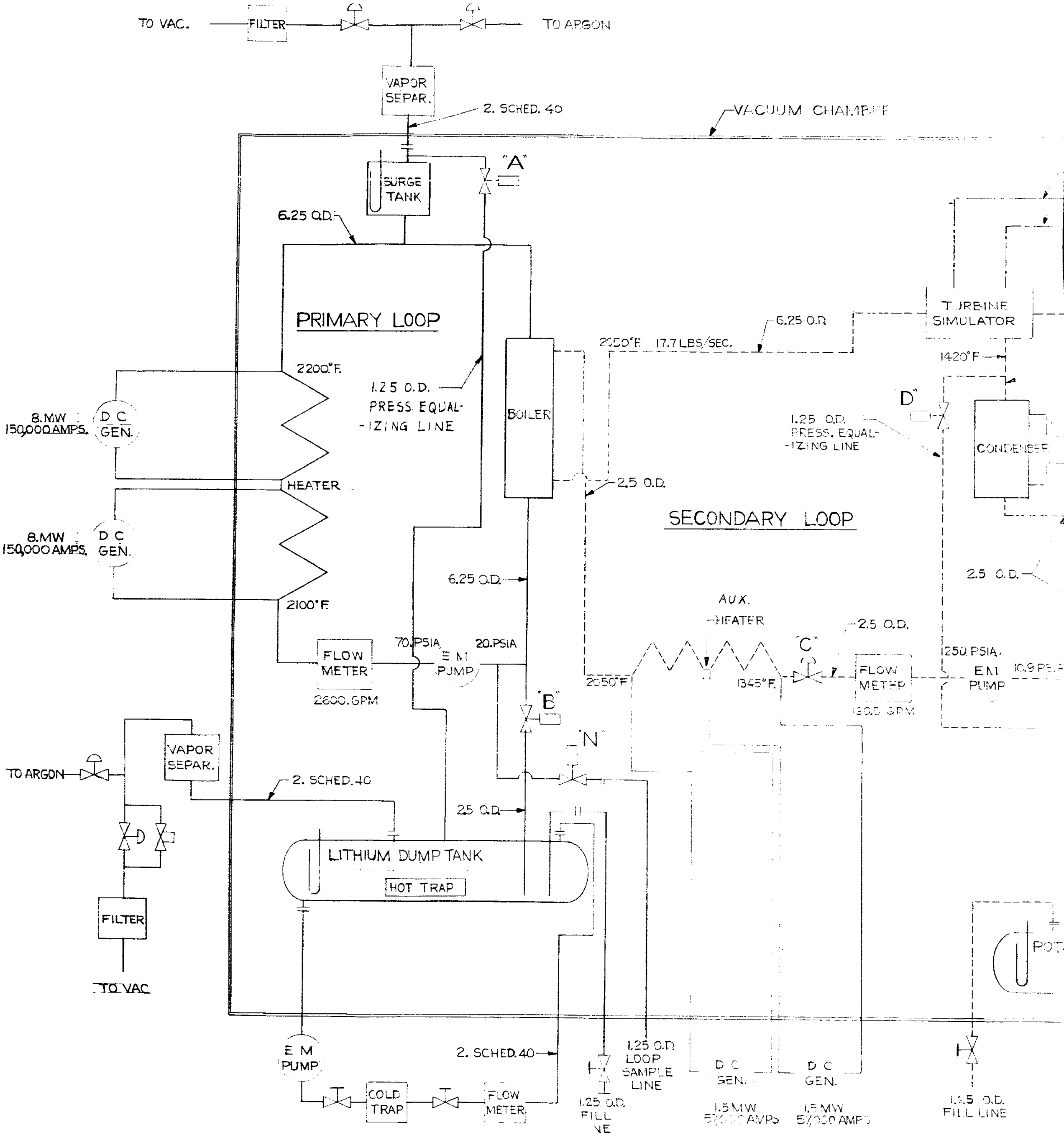
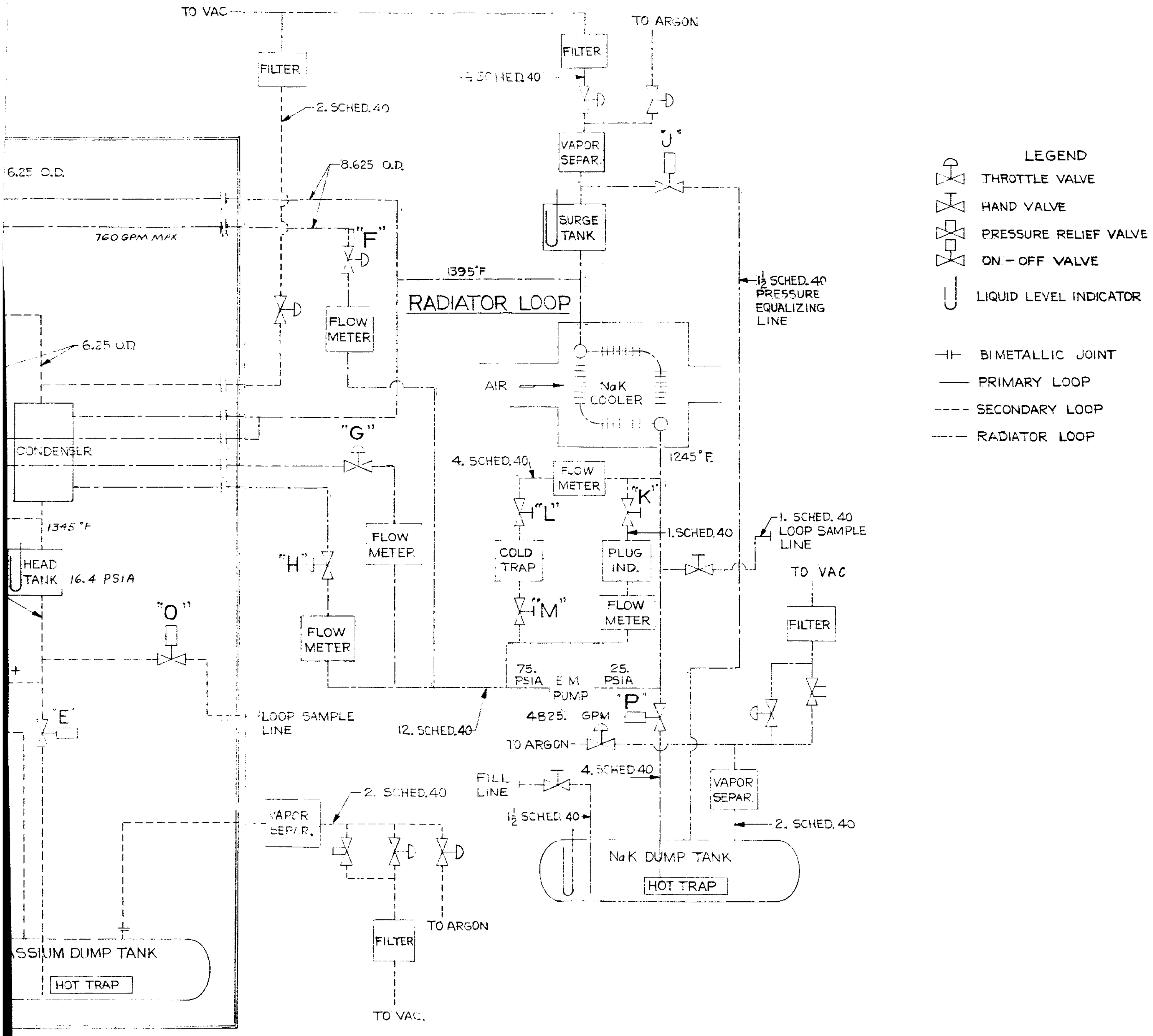


Figure 7. Flow Schematic, 15 MW


TO VAC
FILTER
2. SCHED. 40
FILTER
4 SCHED. 40
TO ARGON
VAPOR SEPAR.
"J"
6.25 O.D.
8.625 O.D.
760 GPM MAX
SURGE TANK
"F"
1395°F
RADIATOR LOOP
1½ SCHED. 40 PRESSURE EQUALIZING LINE
FLOW METER
6.25 O.D.
AIR
NaK COOLER
CONDENSER
"G"
1245°F
4. SCHED. 40
FLOW METER
"K"
"L"
1345°F
FLOW METER
COLD TRAP
1. SCHED. 40
PLUG IND.
1. SCHED. 40 LOOP SAMPLE LINE
HEAD TANK
16.4 PSIA
"H"
TO VAC
"O"
"M"
FLOW METER
FLOW METER
FILTER
75. PSIA
E M PUMP
25. PSIA
"E"
LOOP SAMPLE LINE
12. SCHED. 40
4825. GPM
"P"
TO ARGON
FILL LINE
4. SCHED. 40
VAPOR SEPAR.
2. SCHED. 40
VAPOR SEPAR.
1½ SCHED. 40
2. SCHED. 40
NaK DUMP TANK
HOT TRAP
FILTER
TO ARGON
SSIUM DUMP TANK
HOT TRAP
TO VAC.
LEGEND
THROTTLE VALVE
HAND VALVE
PRESSURE RELIEF VALVE
ON.-OFF VALVE
LIQUID LEVEL INDICATOR
BIMETALLIC JOINT
PRIMARY LOOP
SECONDARY LOOP
RADIATOR LOOP

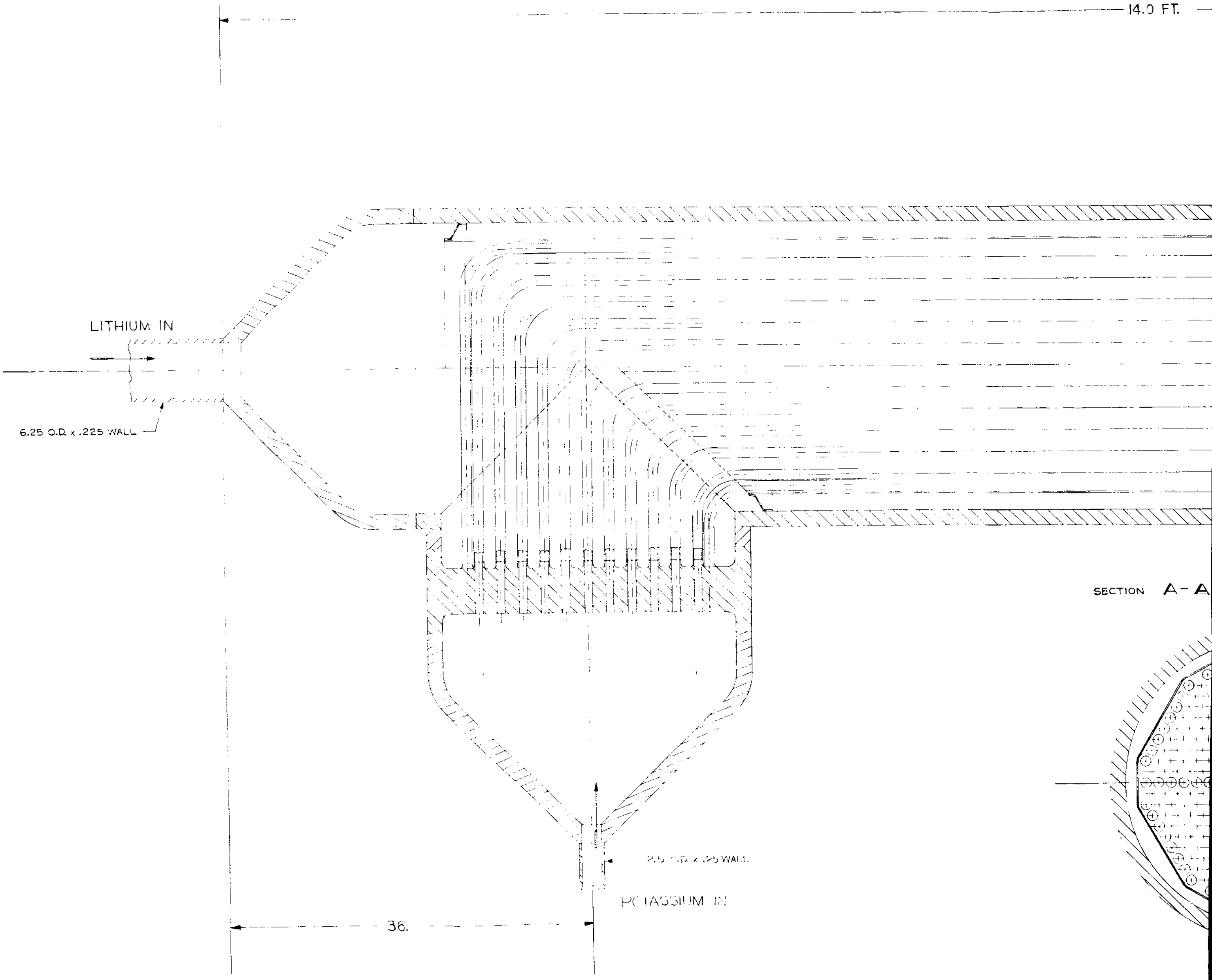
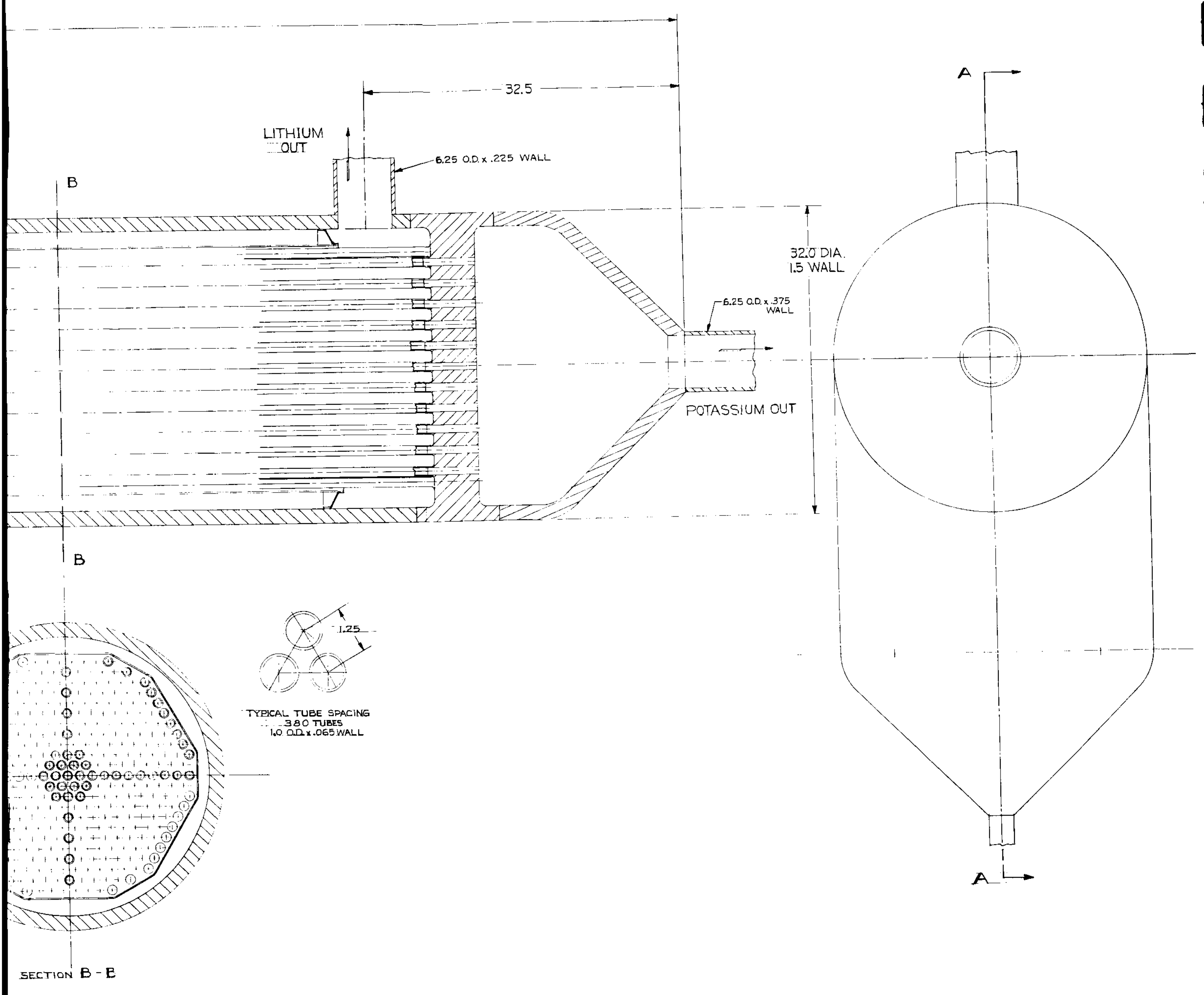


Figure 8. 15 MW Bo



ler (Conceptual).

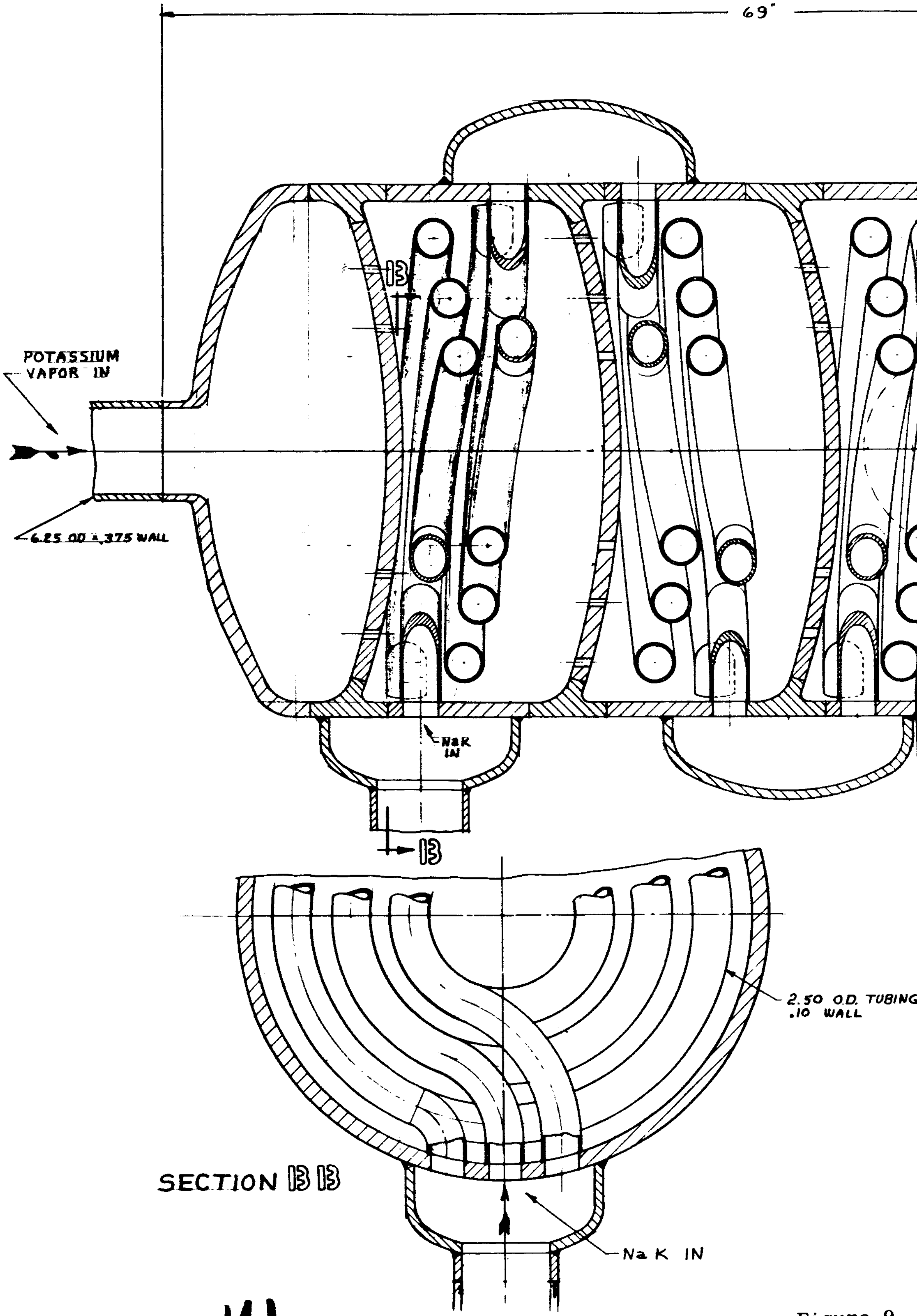
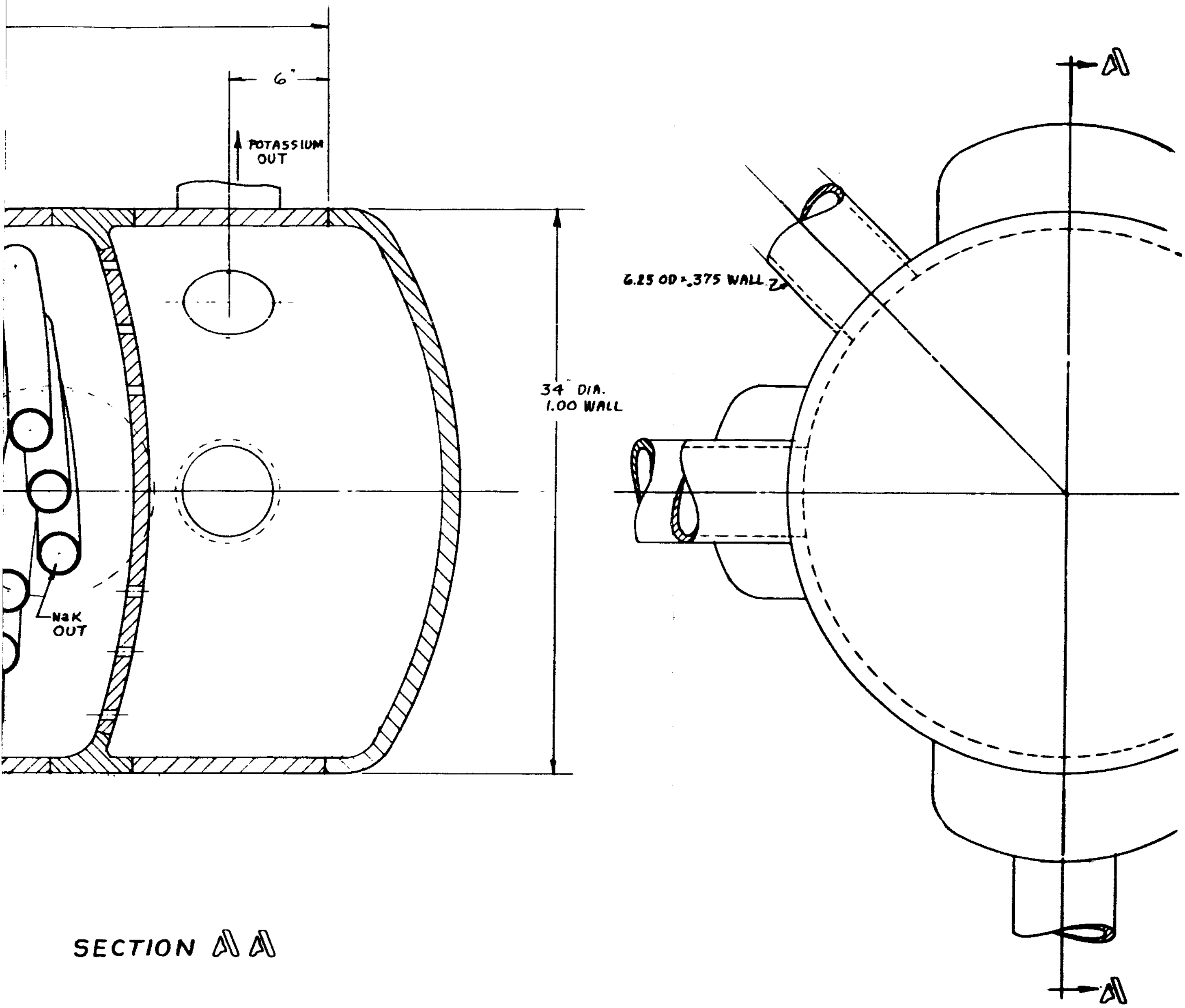

69"
POTASSIUM VAPOR IN
6.25 OD x .375 WALL
B
B
NaK IN
SECTION B B
2.50 O.D. TUBING
.10 WALL
Na K IN

Figure 9.



15 MW Turbine Simulator (Conceptual).

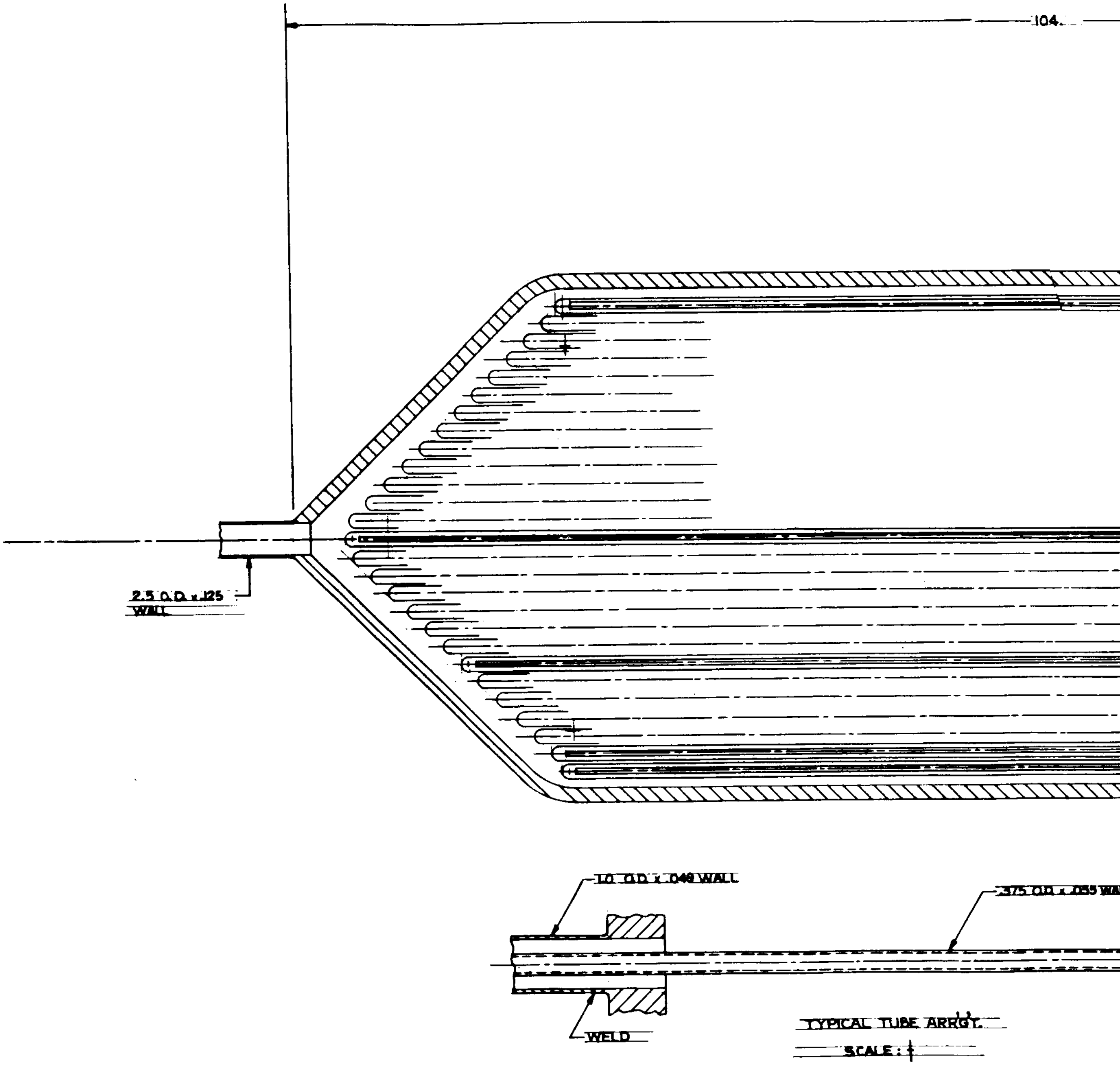
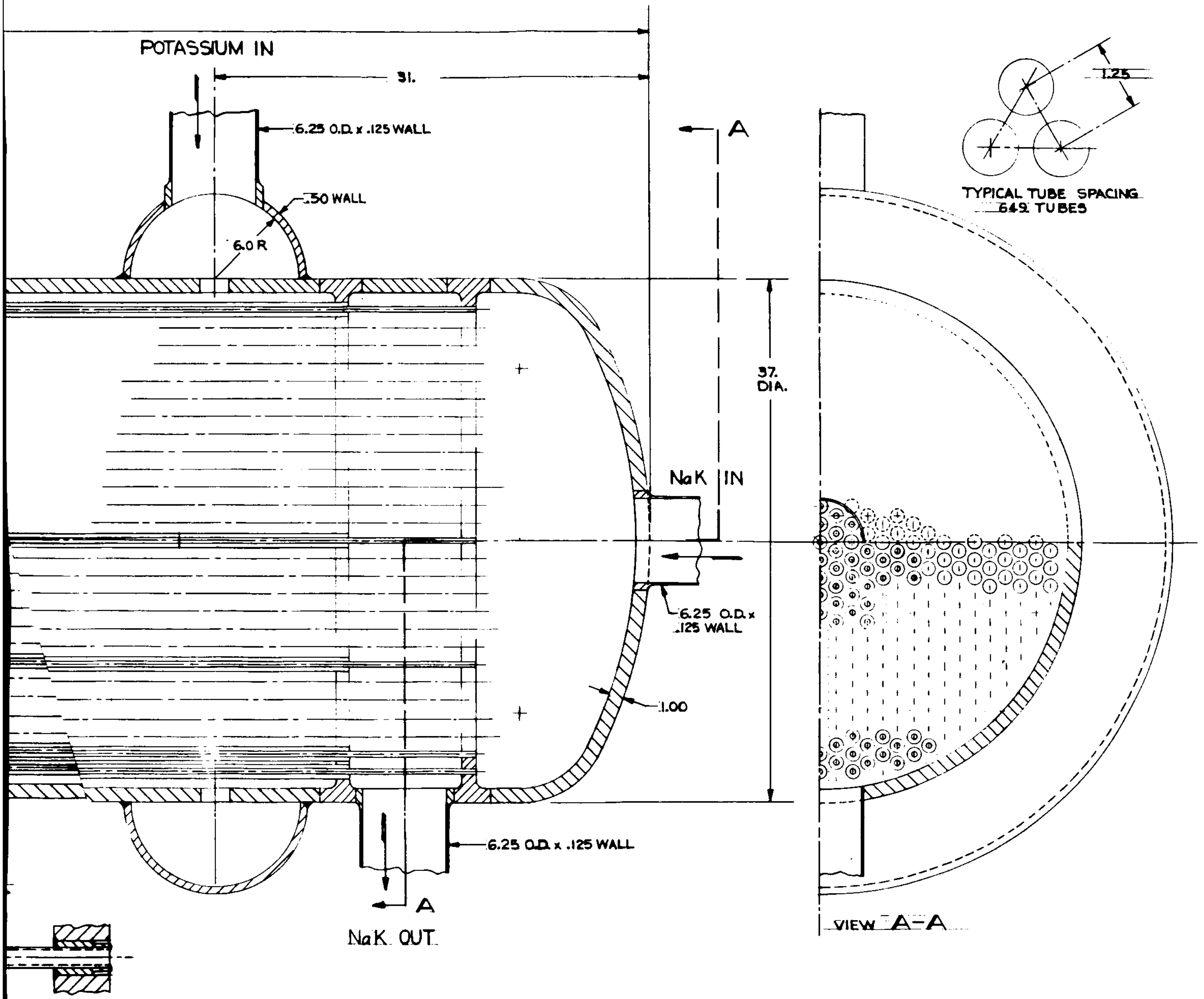
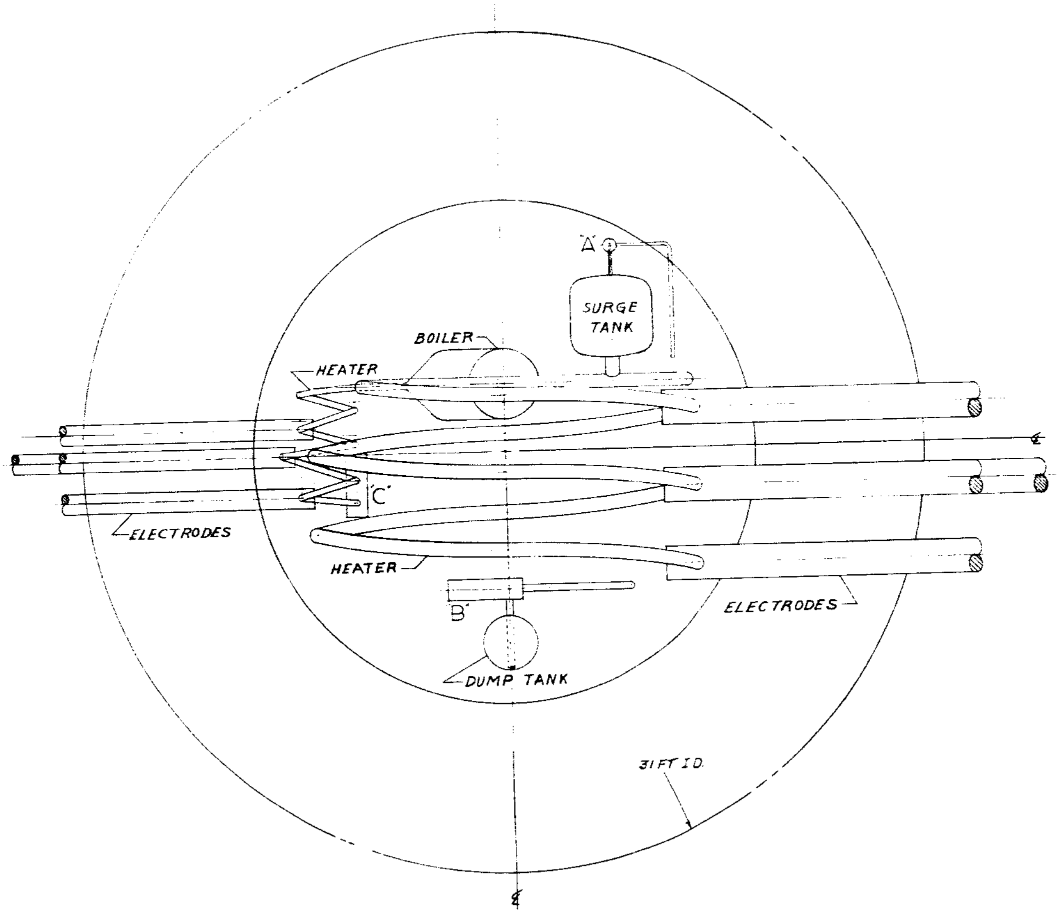


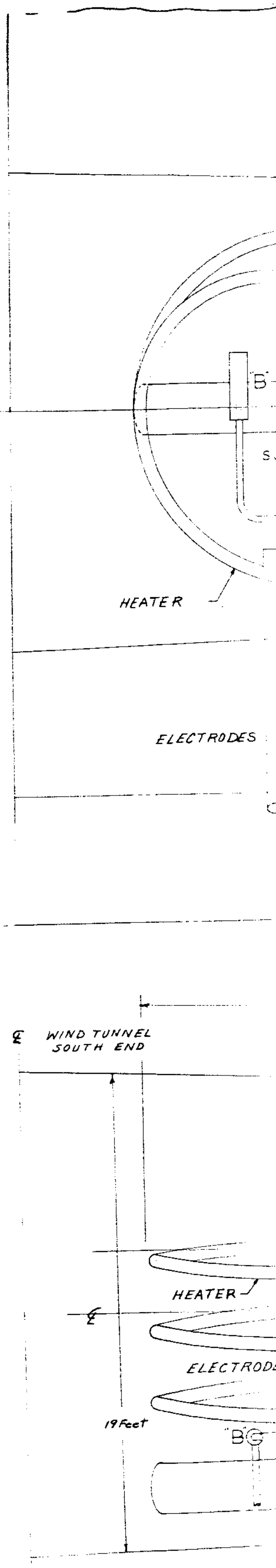
Figure 10. 15 MW Co



denser, Bayonet Type (Conceptual).



END VIEW
HEATER ELECTRODE
PENETRATION



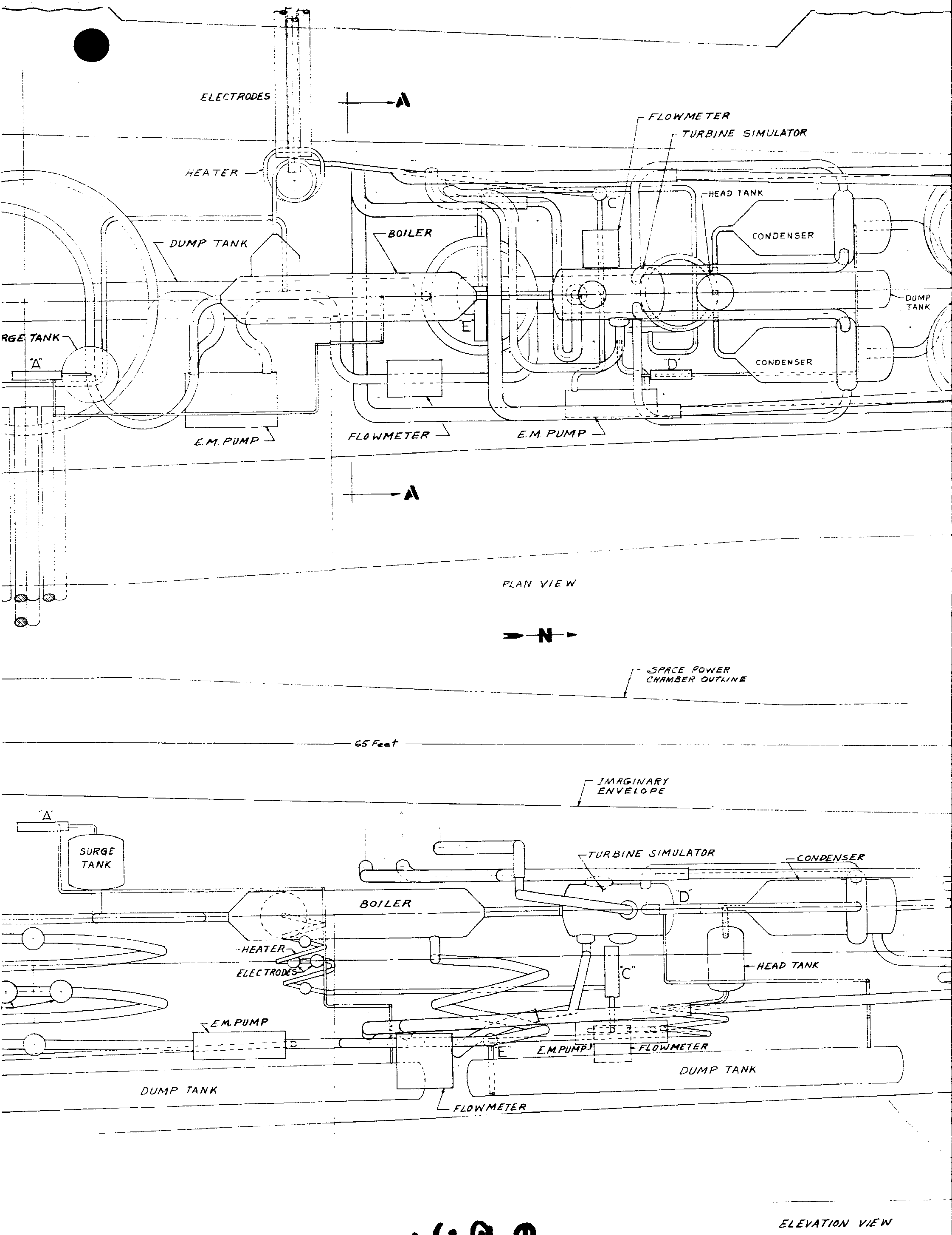
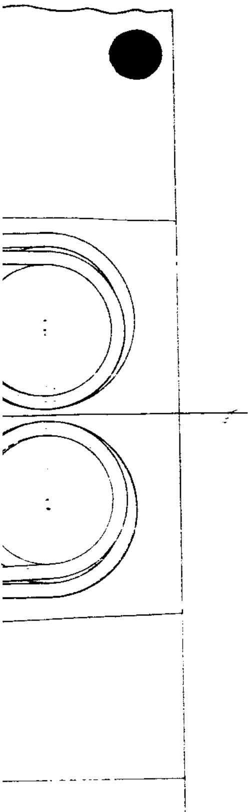
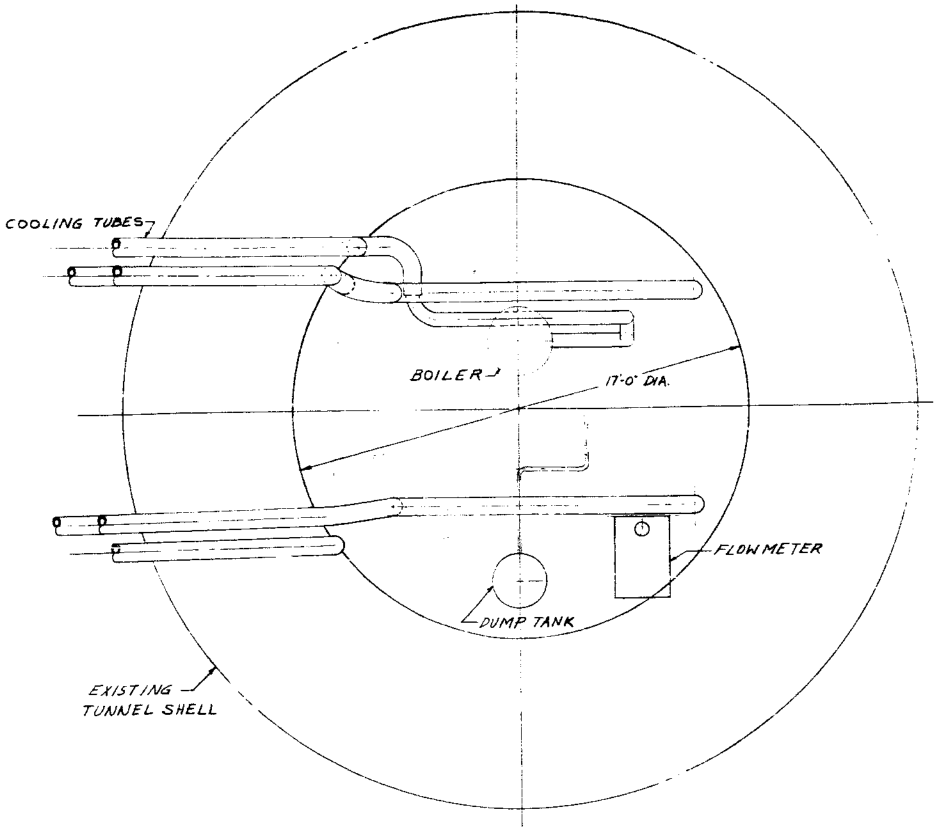
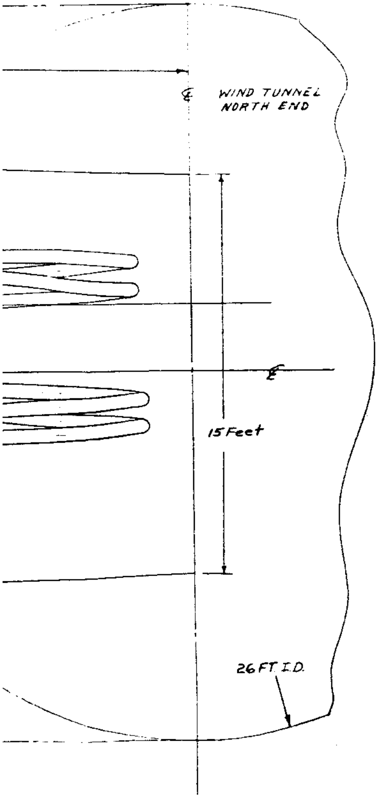


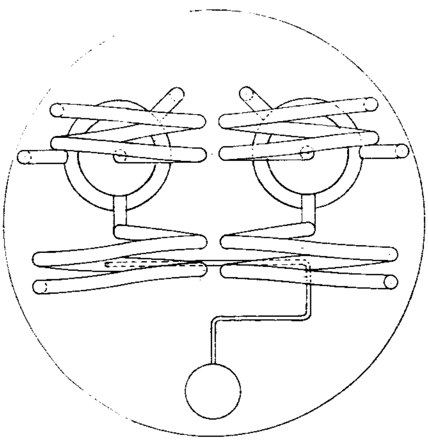
Figure 11. Layout Liquid Metal Loop, Inside Vacuum Chamber.





SECTION **AA**
COOLING TUBE
PENETRATION





END VIEW

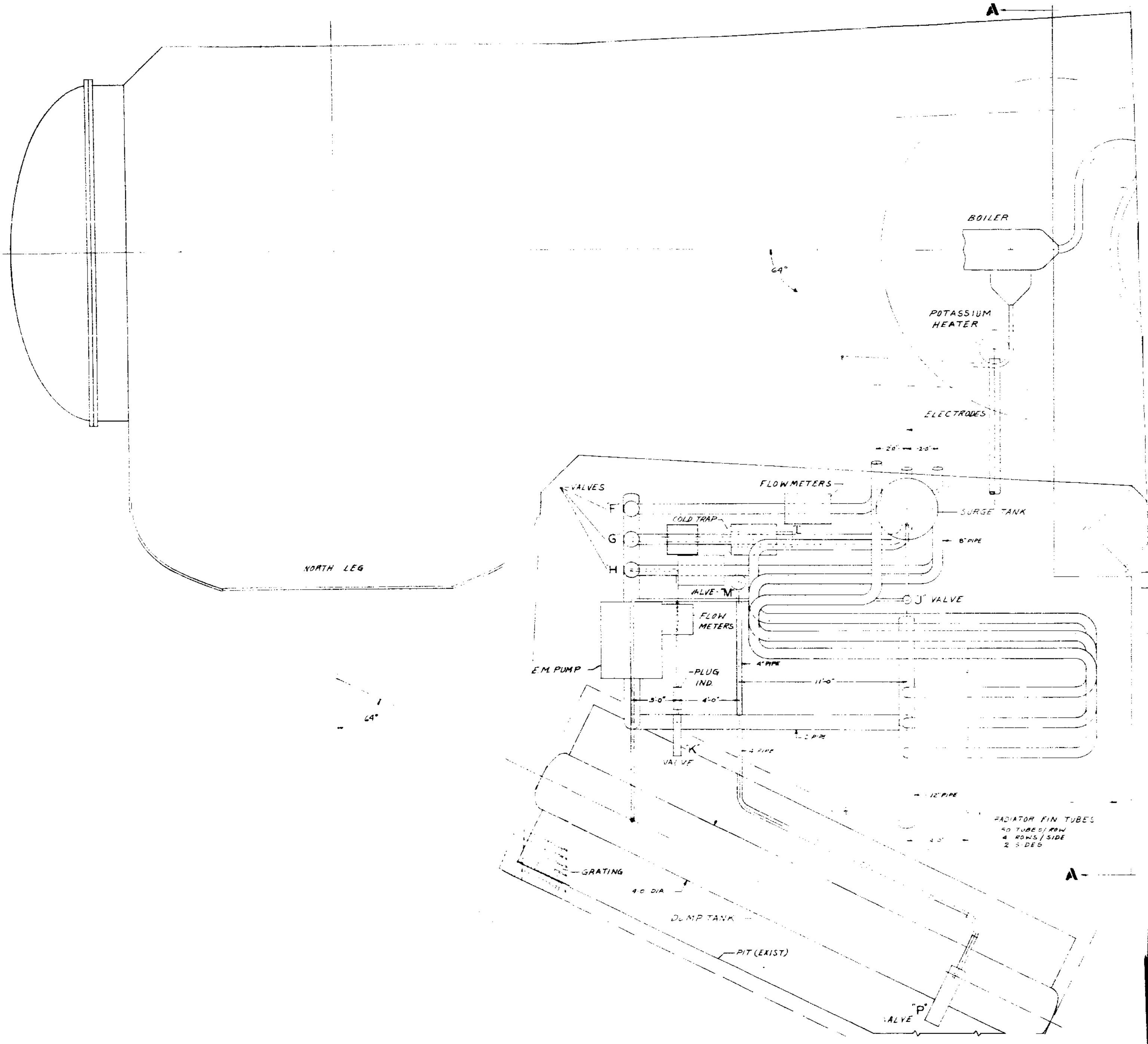
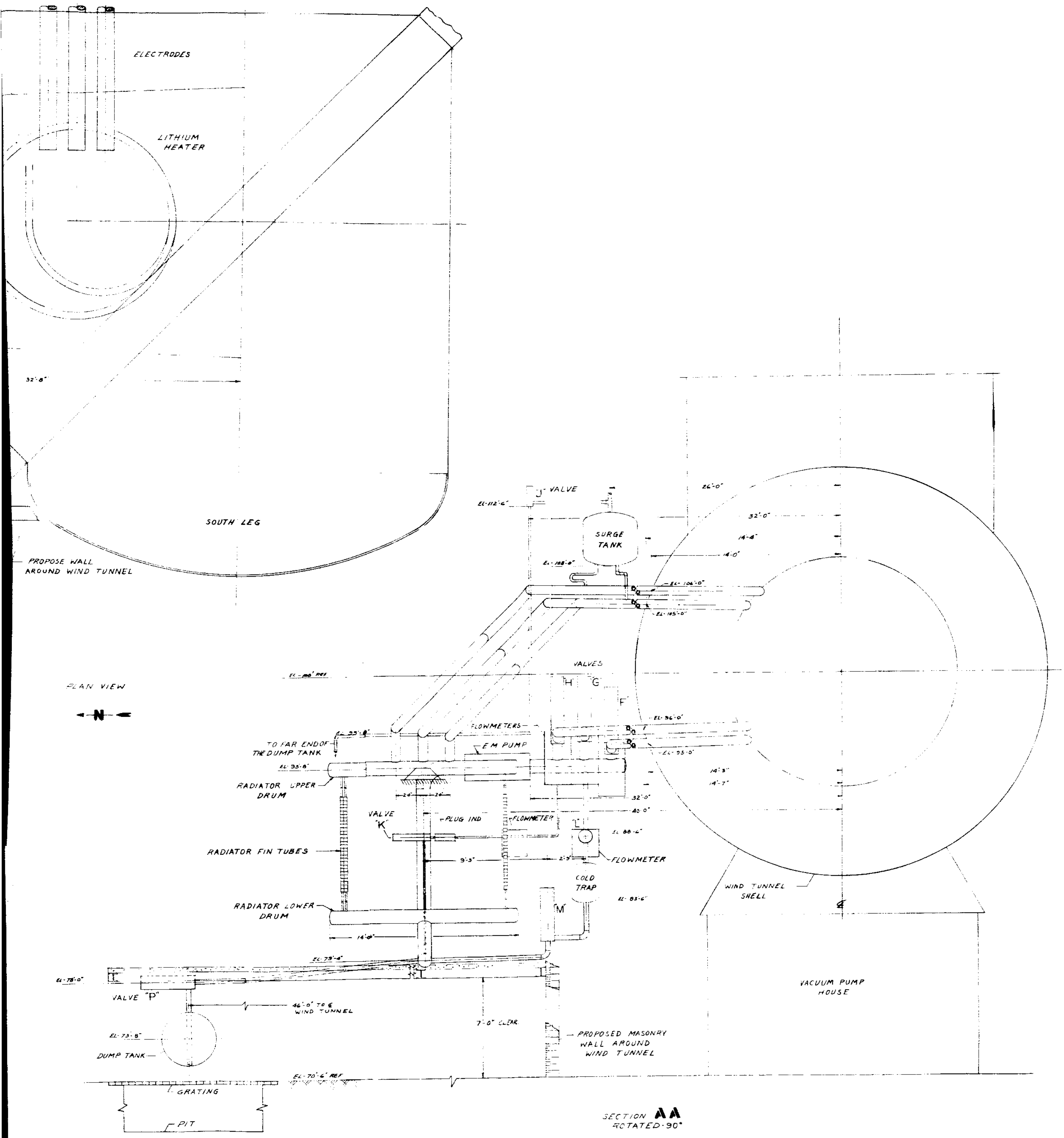


Figure 12. Layout Liquid Met

191-1



al Loop, Outside Vacuum Chamber.

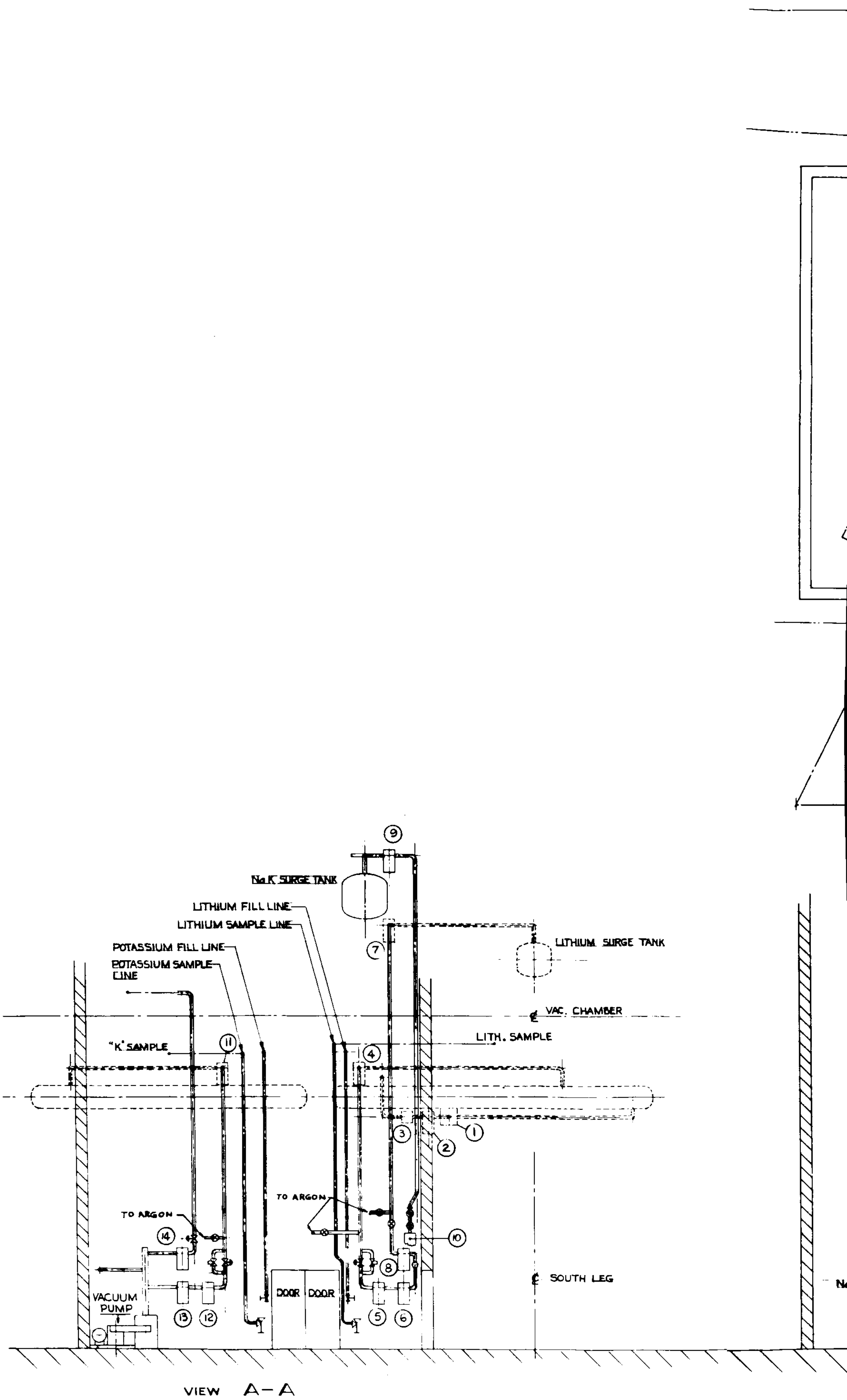
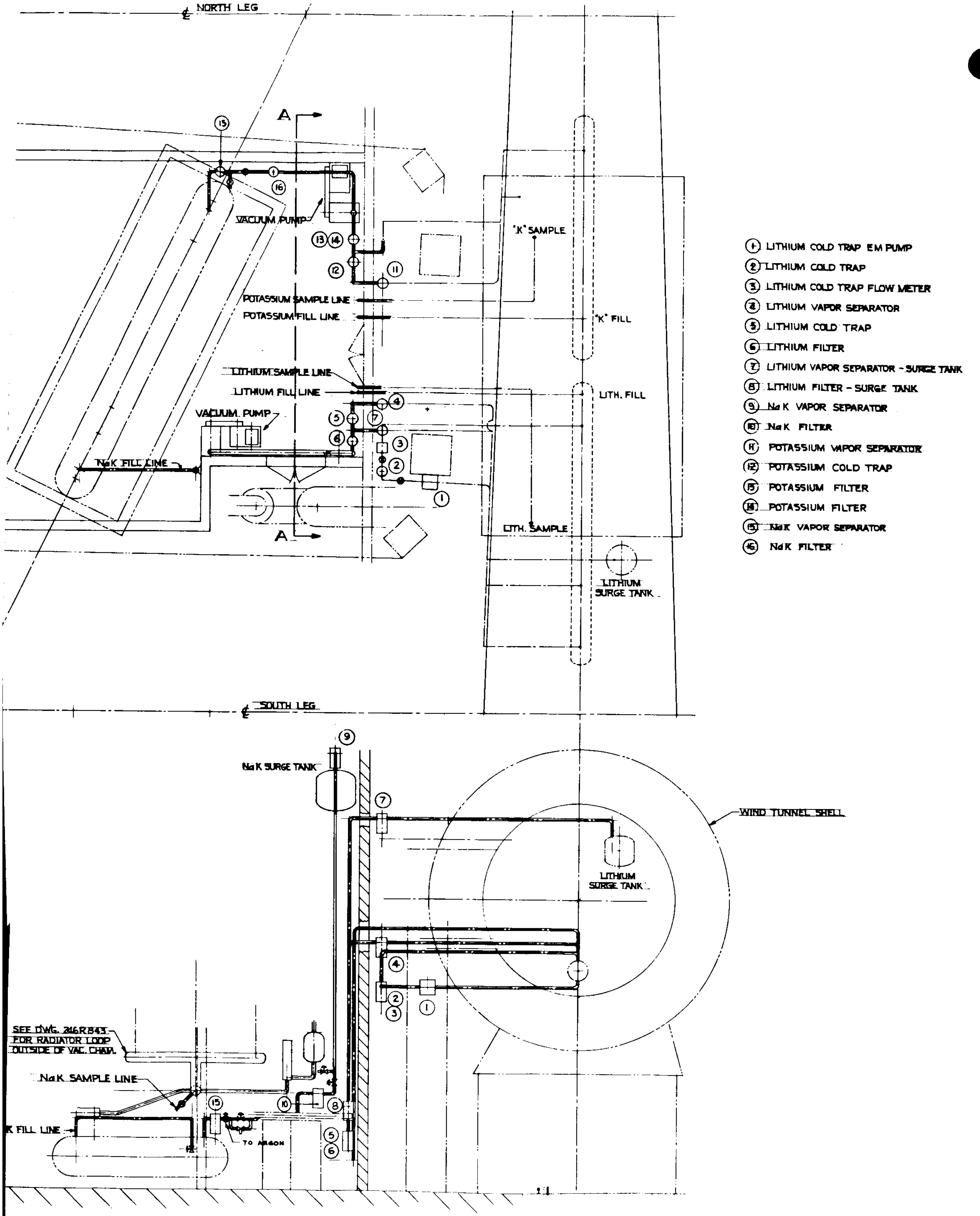


Figure 13. Auxiliary



Loop Connections, Outside Vacuum Chamber.

192-2

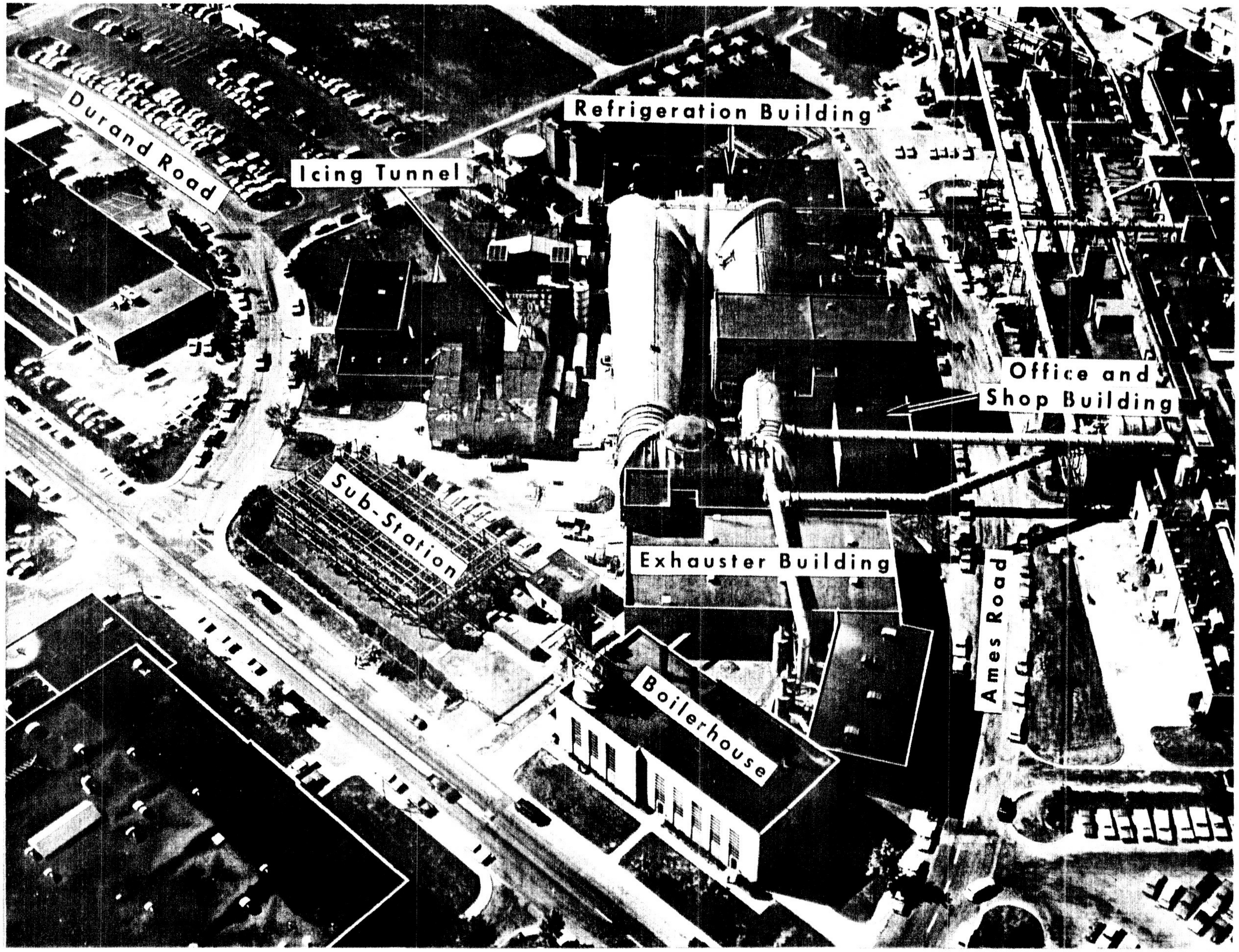
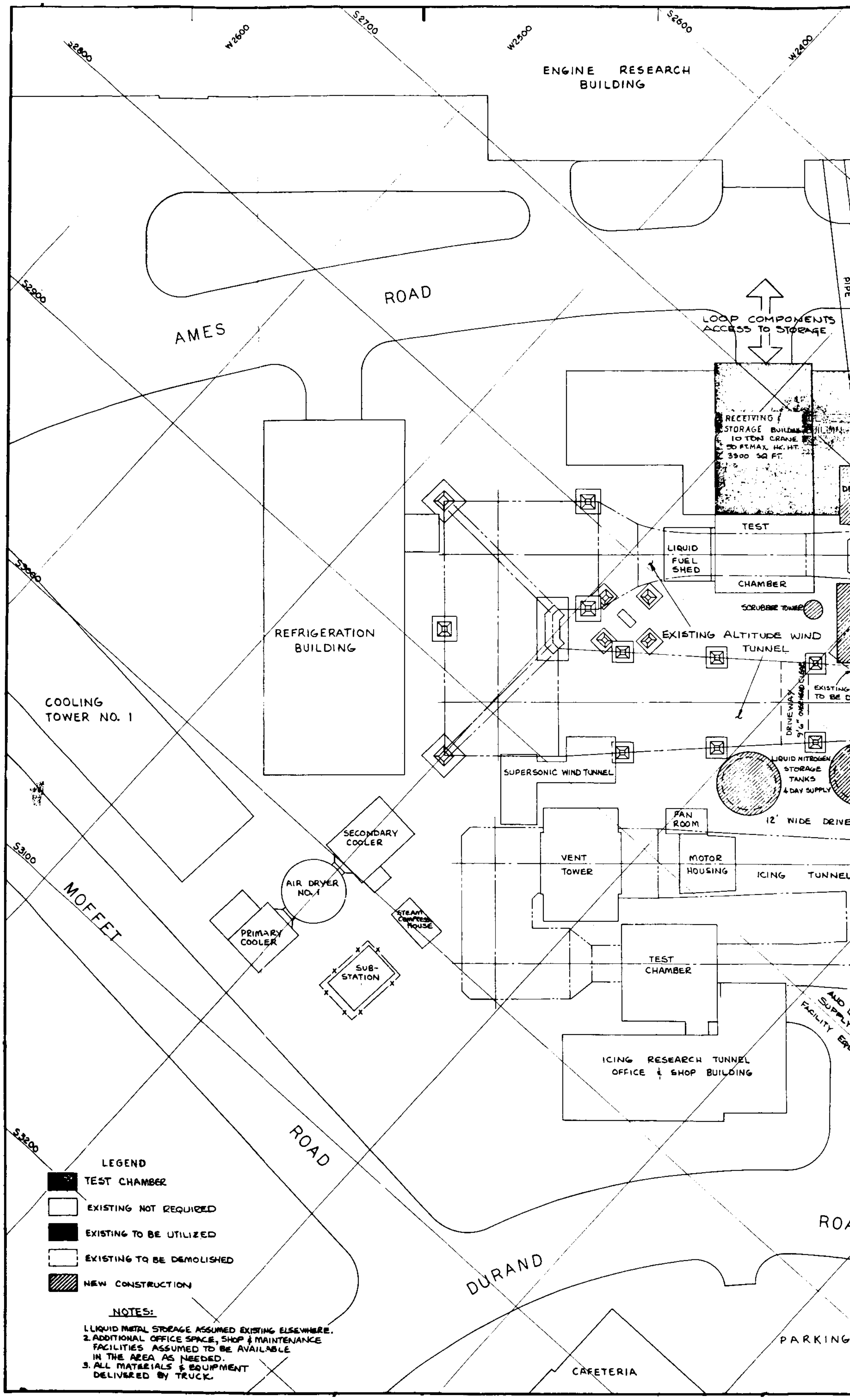


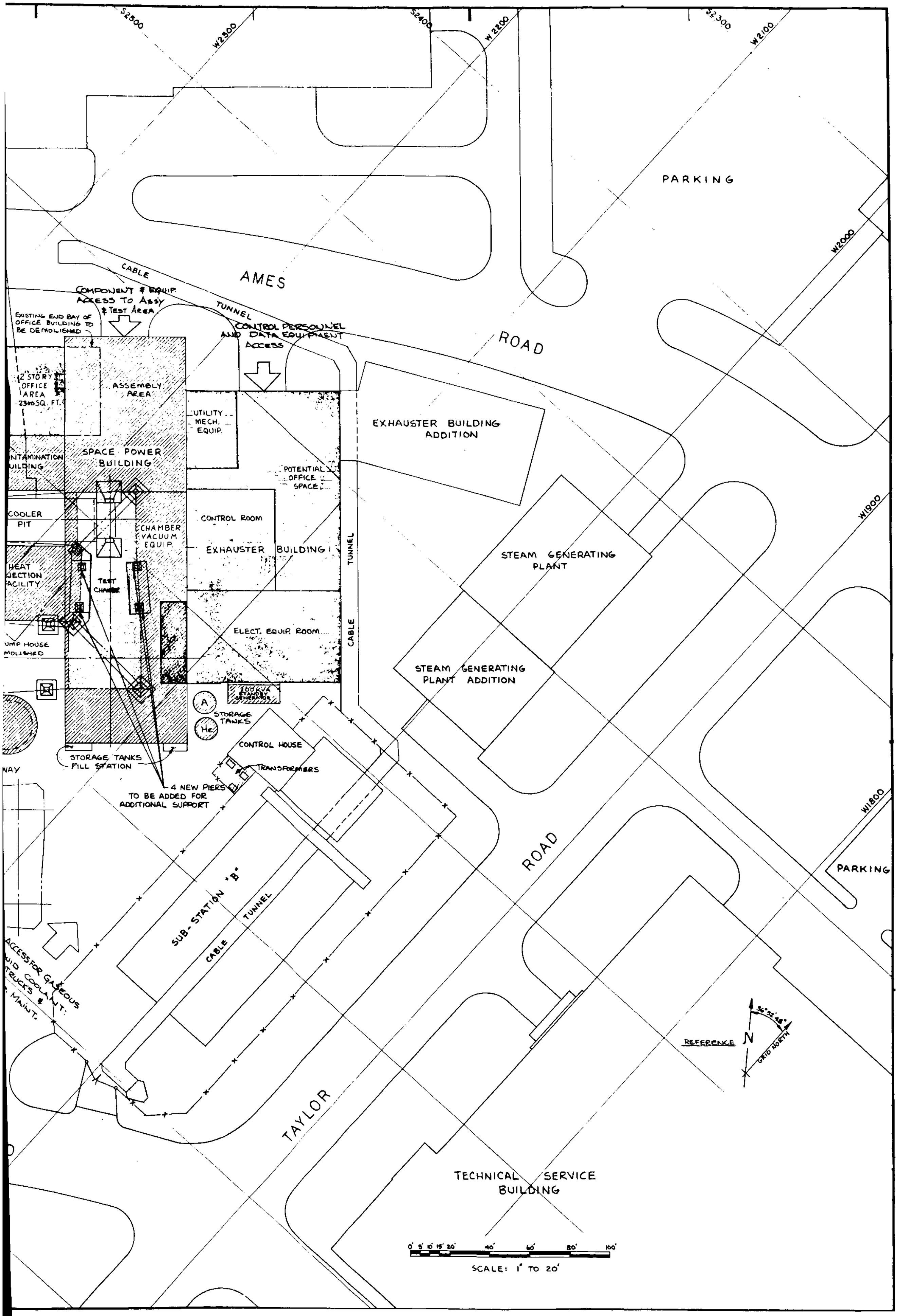
Figure 14. Altitude Wind Tunnel NASA-Lewis.



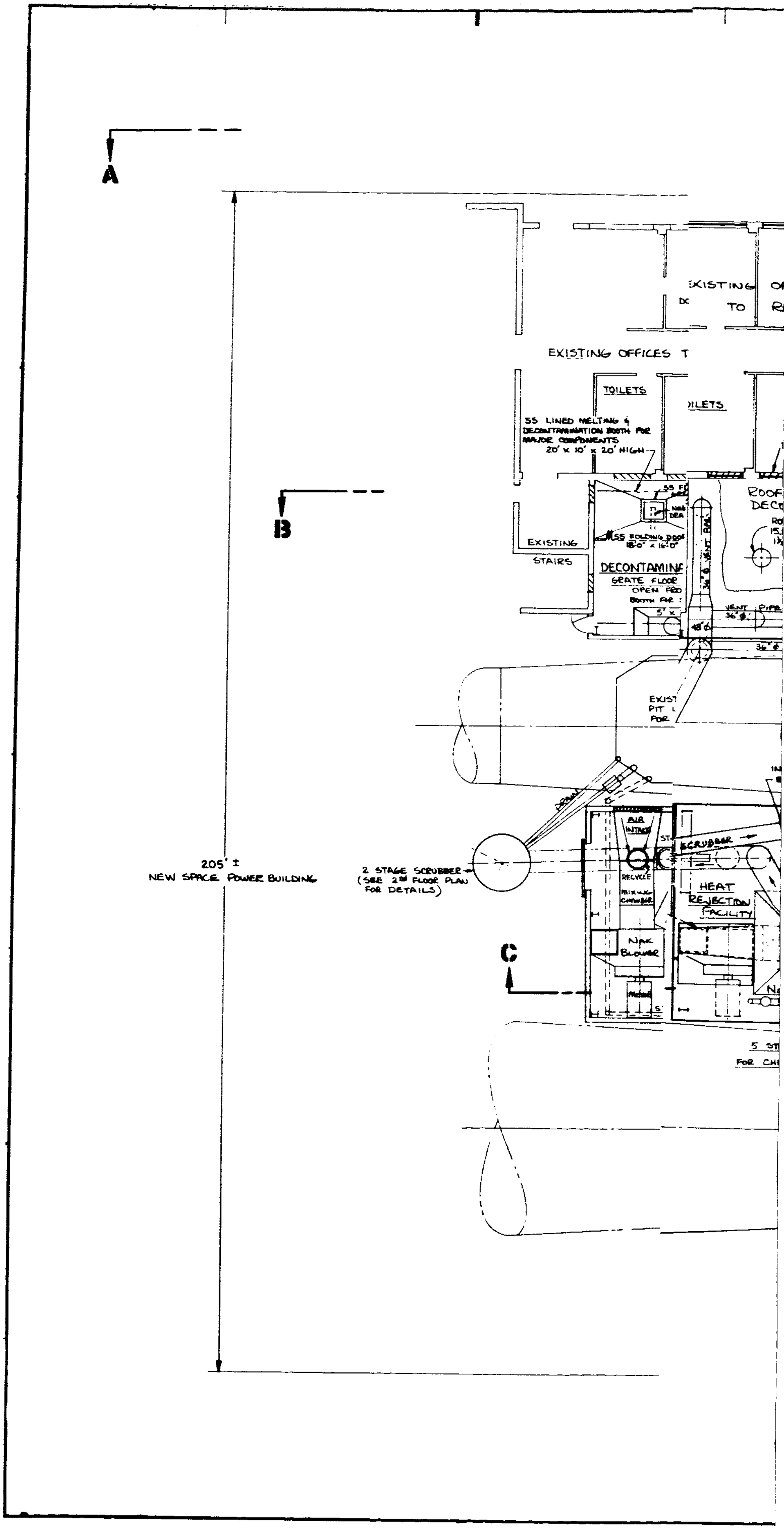
NOTES:

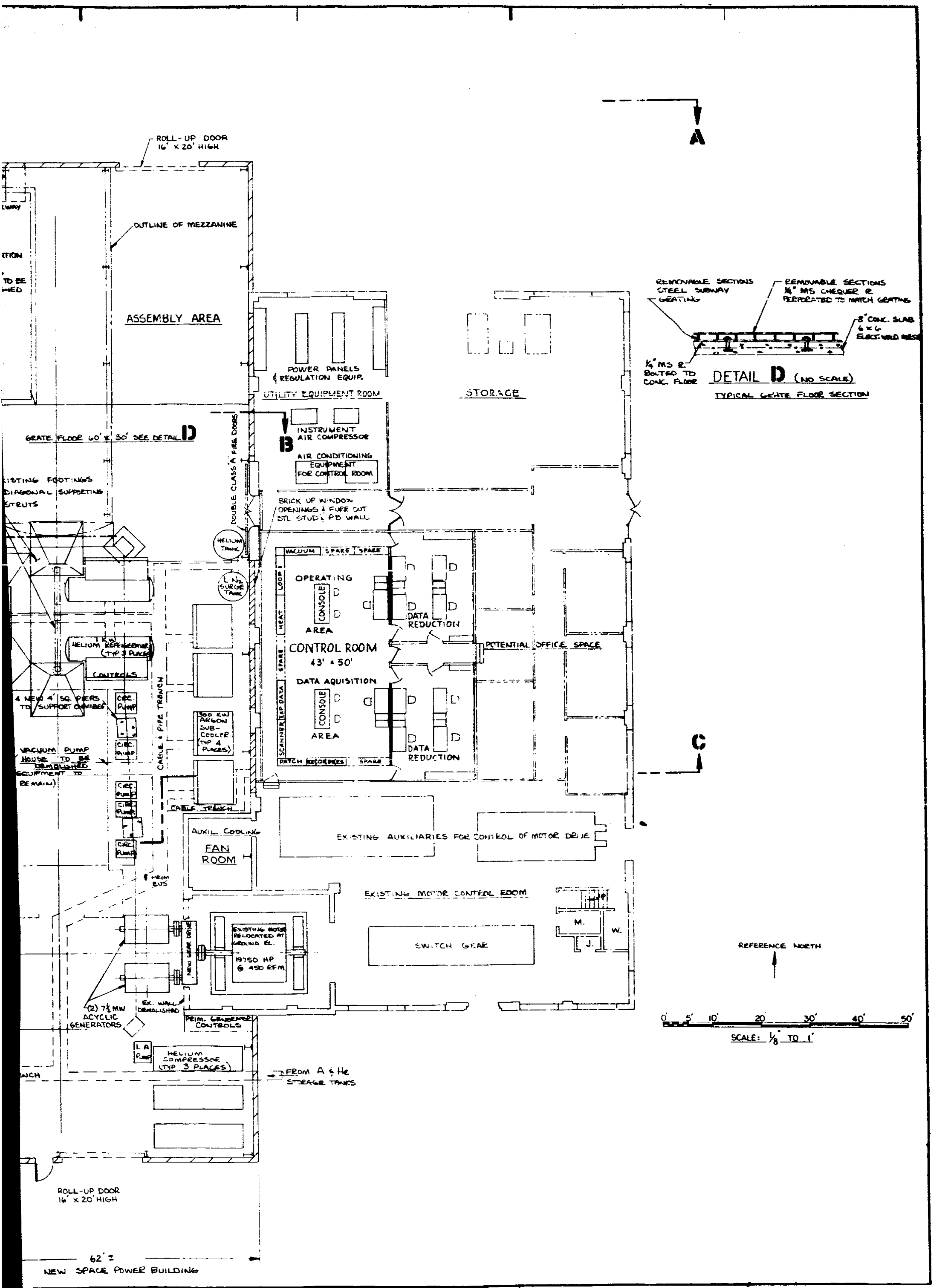
1. LIQUID METAL STORAGE ASSUMED EXISTING ELSEWHERE.
2. ADDITIONAL OFFICE SPACE, SHOP & MAINTENANCE FACILITIES ASSUMED TO BE AVAILABLE IN THE AREA AS NEEDED.
3. ALL MATERIALS & EQUIPMENT DELIVERED BY TRUCK.

Figure

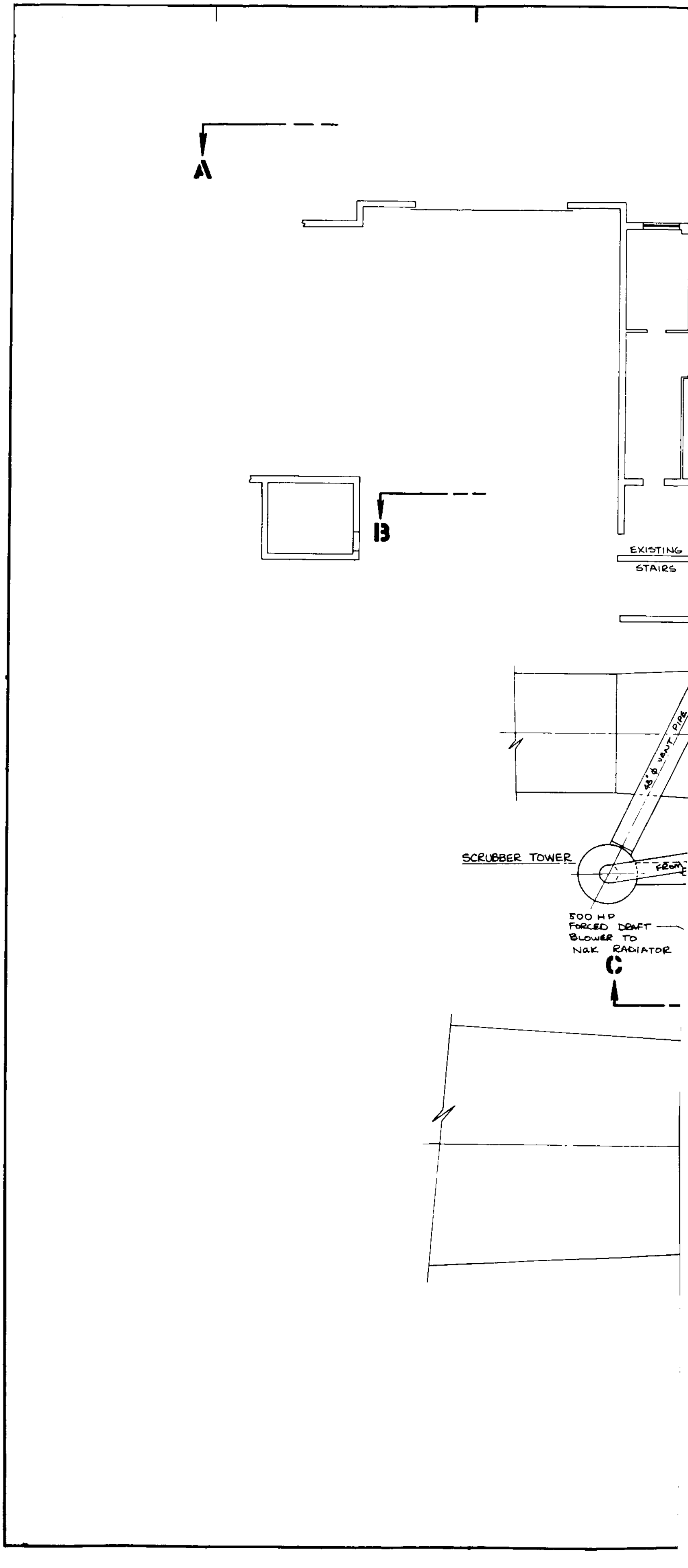

S2500
W2300
S2400
W2200
S2300
W2100
PARKING
W2000
AMES
ROAD
CABLE
TUNNEL
COMPONENT & EQUIP.
ACCESS TO ASSY
& TEST AREA
EXISTING END BAY OF
OFFICE BUILDING TO
BE DEMOLISHED
CONTROL PERSONNEL
AND DATA EQUIPMENT
ACCESS
2 STORY
OFFICE
AREA
2300 SQ. FT.
ASSEMBLY
AREA
UTILITY
MECH.
EQUIP.
EXHAUSTER BUILDING
ADDITION
NTAMINATION
UILDING
SPACE POWER
BUILDING
POTENTIAL
OFFICE
SPACE
COOLER
PIT
CONTROL ROOM
CHAMBER
VACUUM
EQUIP.
EXHAUSTER BUILDING
TUNNEL
STEAM GENERATING
PLANT
W1900
HEAT
JECTION
ACILITY
TEST
CHAMBER
ELECT. EQUIP. ROOM
CABLE
UMP HOUSE
MOLISHED
STEAM GENERATING
PLANT ADDITION
200 KVA
STANDBY
GENERATOR
A
He
STORAGE
TANKS
CONTROL HOUSE
TRANSFORMERS
STORAGE TANKS
FILL STATION
NAY
4 NEW PIERS
TO BE ADDED FOR
ADDITIONAL SUPPORT
W1800
ROAD
PARKING
SUB-STATION "B"
CABLE
TUNNEL
ACCESS FOR GASEOUS
UID COOLANT
TRUCKS &
MAINT.
REFERENCE
N
36°52'48"
GRID NORTH
TAYLOR
TECHNICAL SERVICE
BUILDING
0' 5' 10' 15' 20' 40' 60' 80' 100'
SCALE: 1" TO 20'

5. Plot Plan.

A
B
C
EXISTING OF
TO R
EXISTING OFFICES T
TOILETS
OILETS
SS LINED MELTING & DECONTAMINATION BOOTH FOR MAJOR COMPONENTS 20' x 10' x 20' HIGH
SS FOLDING DOOR 18'-0" x 16'-0"
EXISTING STAIRS
DECONTAMINA
GRATE FLOOR
OPEN FRO
BOOTH FOR
5' x
36" Φ VENT PIPE
VENT PIPE 36" Φ
48" Φ
36" Φ
ROOF
DECO
EXIST PIT FOR
205' ±
NEW SPACE POWER BUILDING
2 STAGE SCRUBBER (SEE 2nd FLOOR PLAN FOR DETAILS)
DRAIN
AIR INTAKE
ST
SCRUBBER
RECYCLE
MIXING CHAMBER
HEAT REJECTION FACILITY
NaK BLOWER
MOTOR
5 ST
FOR CH



16. First Floor Plan.


A
B
EXISTING
STAIRS
48" φ VENT PIPE
SCRUBBER TOWER
FROM
500 HP
FORCED DRAFT
BLOWER TO
NaK RADIATOR
C

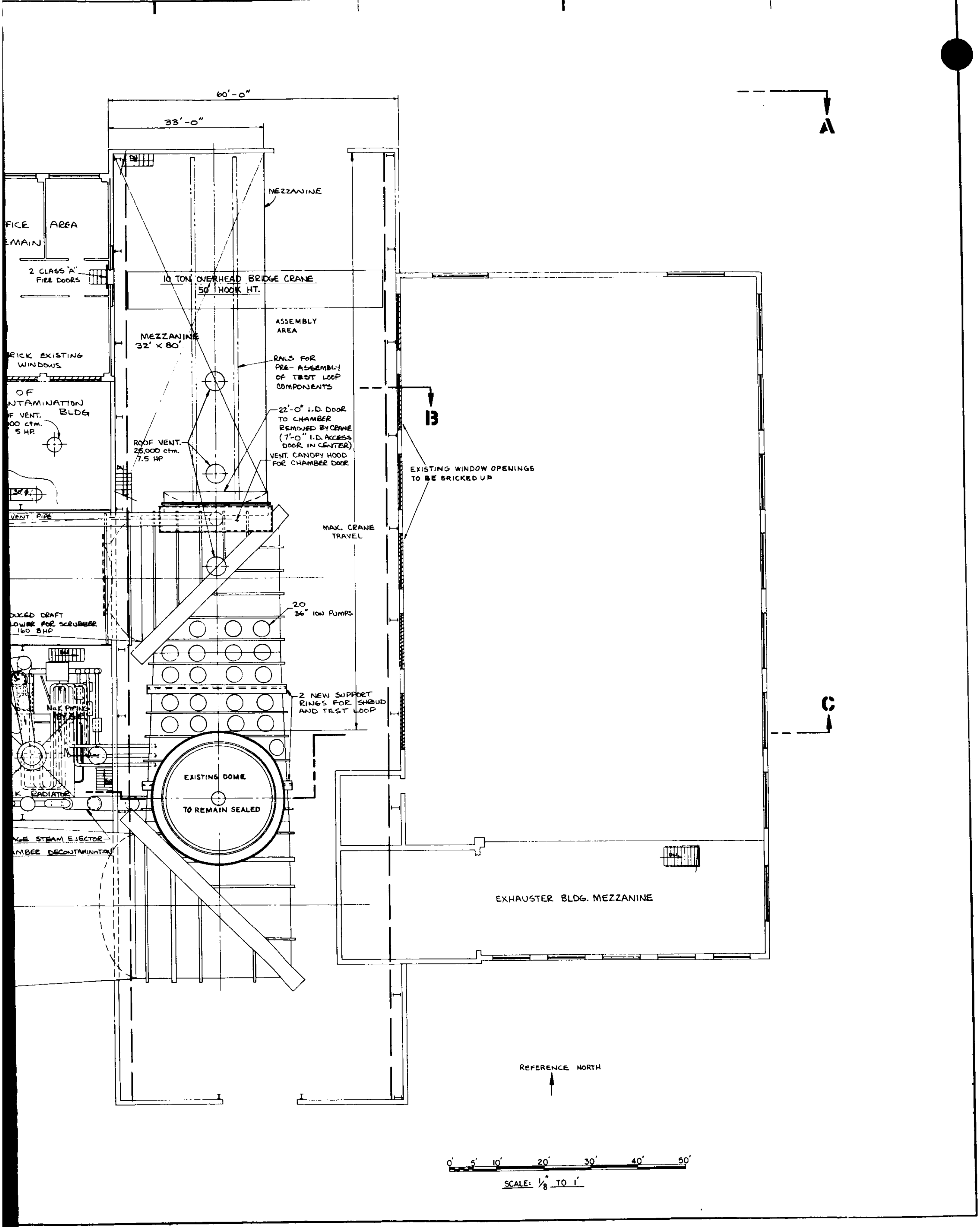


Figure 17. Second Floor Plan.

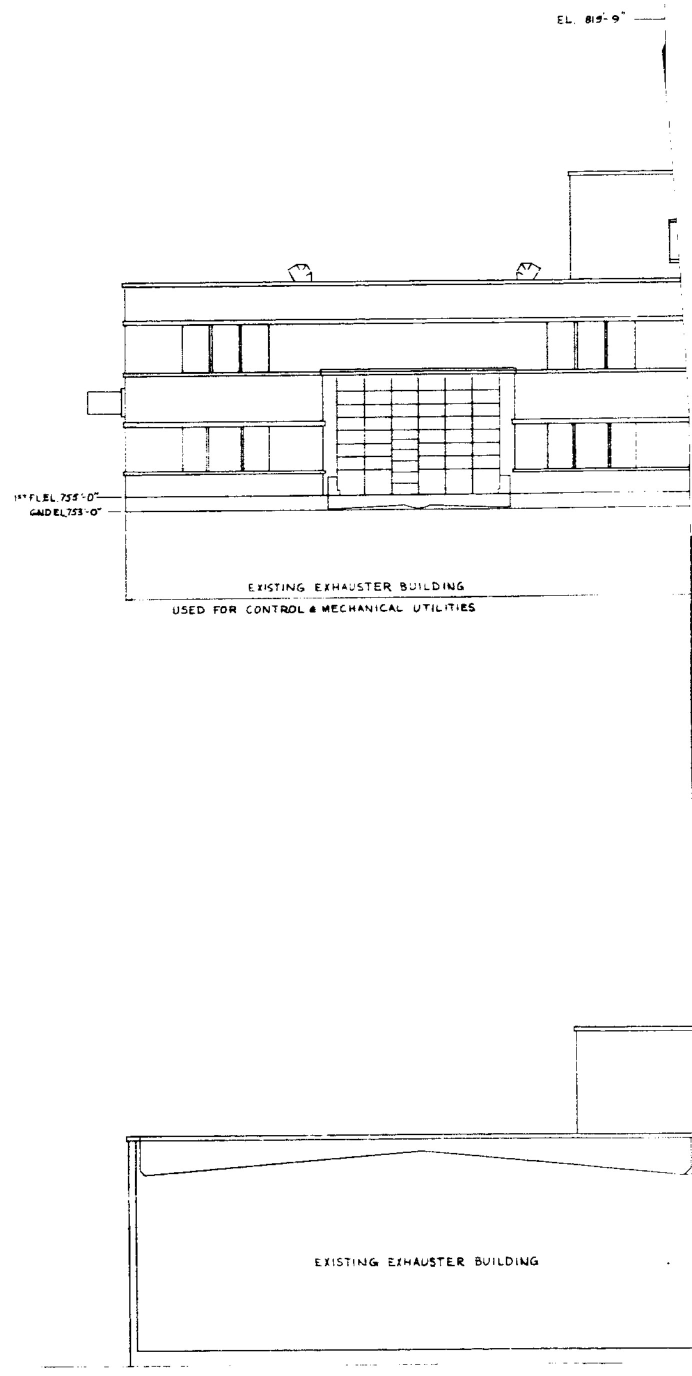
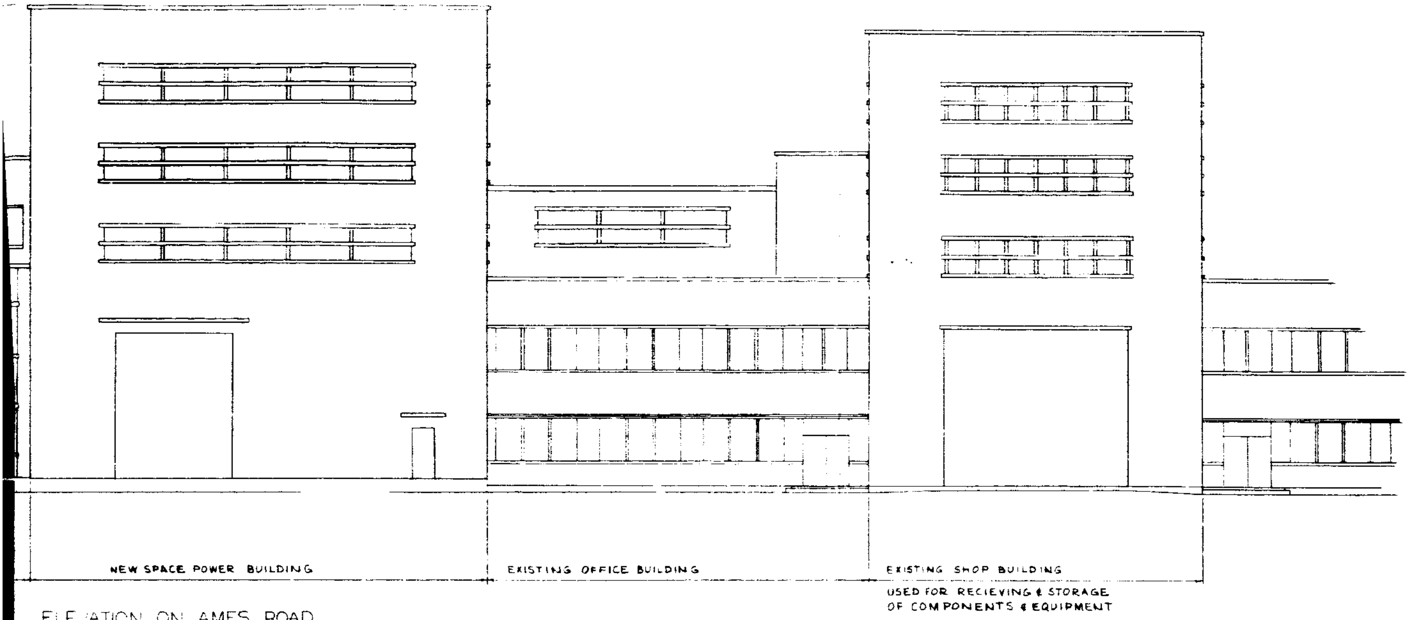

EL. 819'-9"
1st FL EL 755'-0"
GND EL 753'-0"
EXISTING EXHAUSTER BUILDING
USED FOR CONTROL & MECHANICAL UTILITIES
EXISTING EXHAUSTER BUILDING

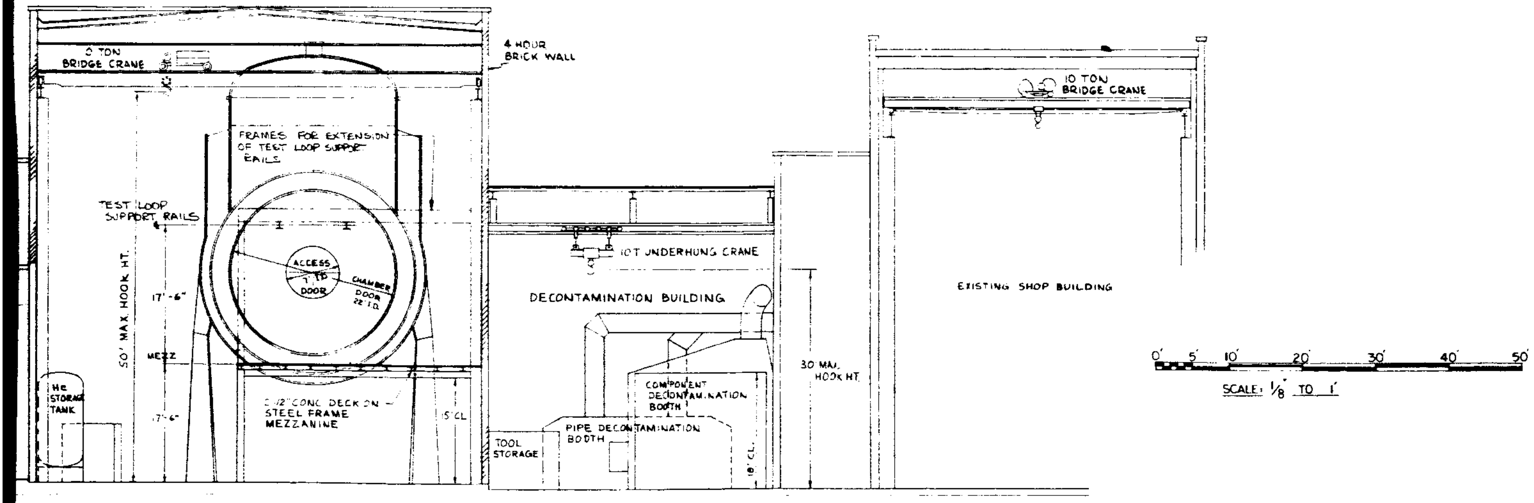
Figure 18.



ELEVATION ON AMES ROAD

VIEW **A-A**

SCALE 1/8" = 1'-0"



SECTION **B-B**

SCALE 1/8" = 1'-0"

ilding Elevation and Section Through Assembly Area.

2

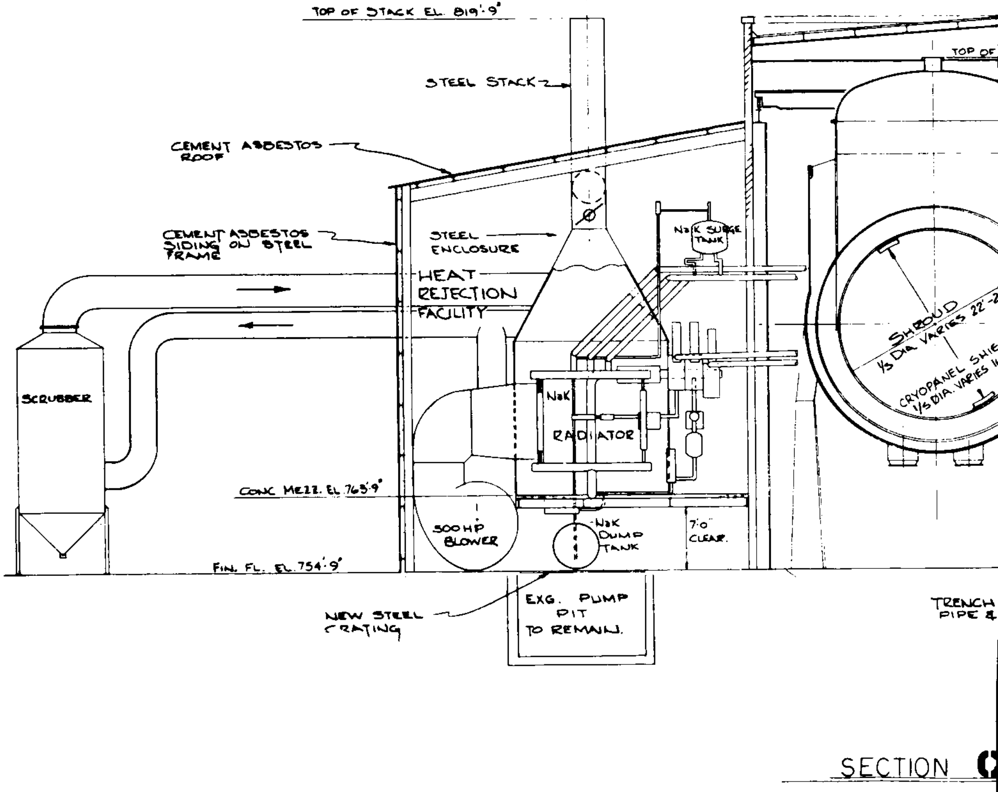
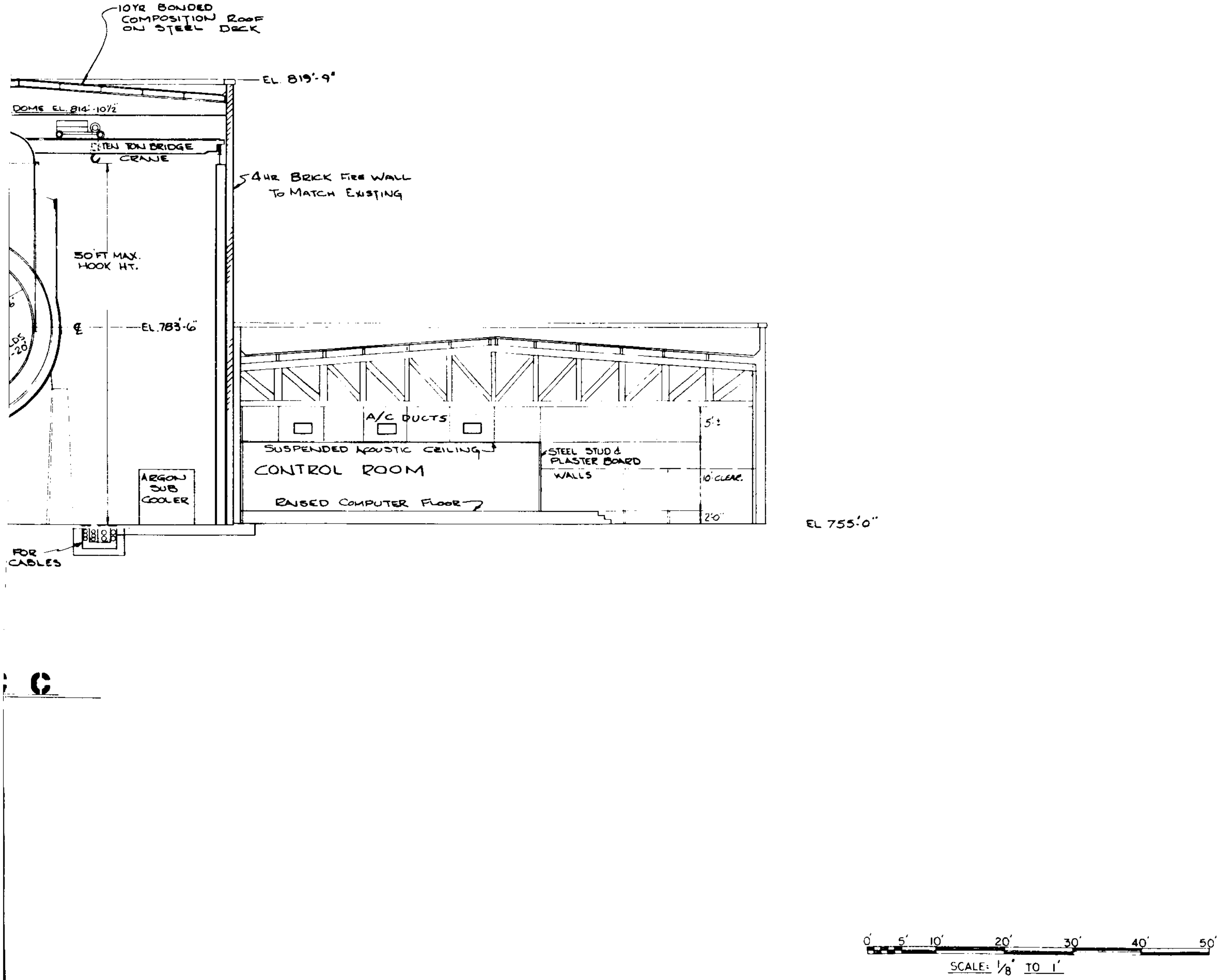
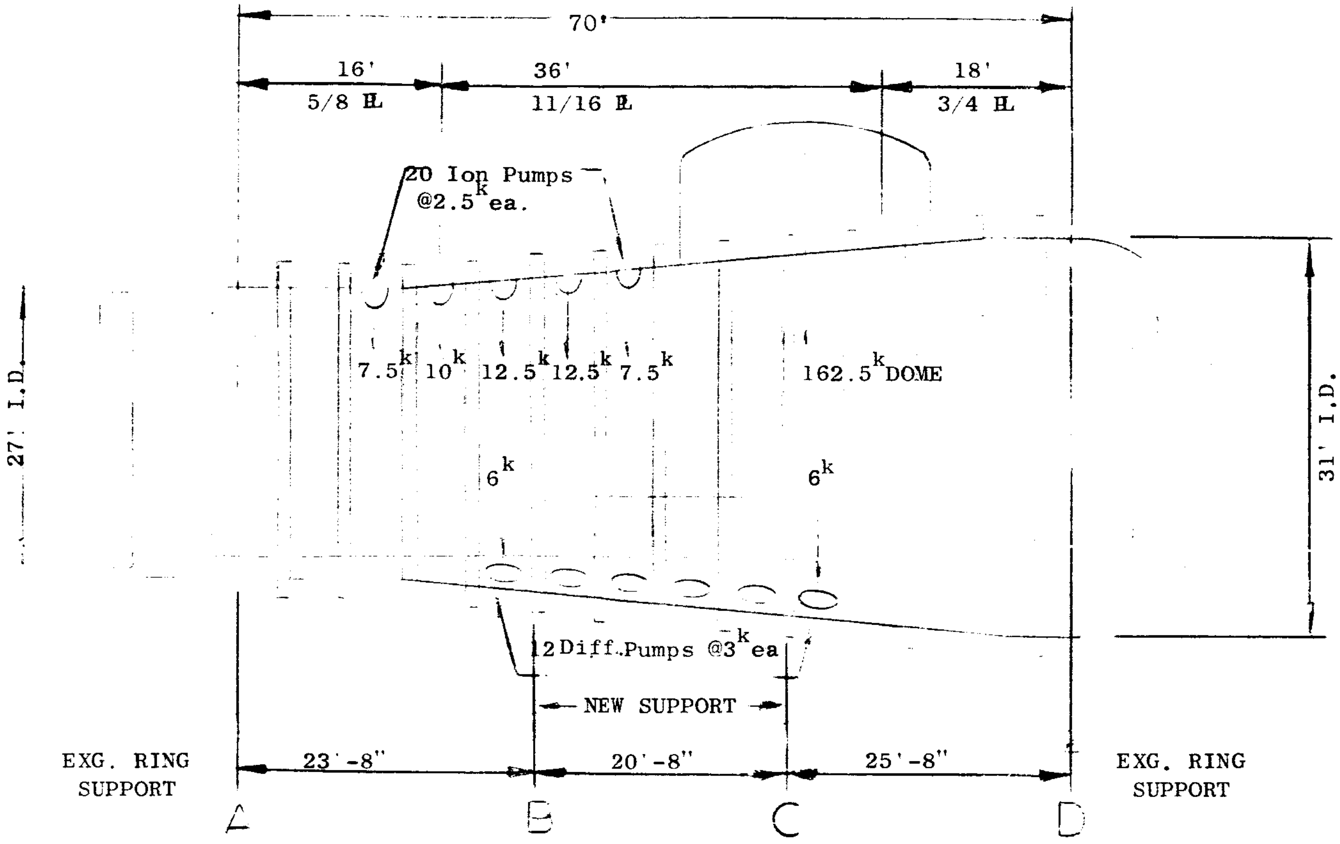


Figure 19. Section Thro



ugh Test Chamber and Heat Rejection Facility.

198-2



Assume tunnel infinitely stiff & all loads transferred to existing & new supports in prop. to dist. from supports.

LOADS (1000 lbs):	A	B	C	D	TOTAL^{k}
1. Exg. Tunnel	35.0	64.9	75.6	44.5	220.0
2. Diff Pumps	0.6	17.4	17.4	0.6	36.0
3. Ion Pumps	8.7	36.9	4.4	0.0	50.0
4. Dome	0.0	0.0	146.0	16.5	162.5
*5. Shroud	30.6	33.4	36.0	38.0	138.0
*6. Test Loop	20.0	30.0	35.0	35.0	120.0^{k}
	94.9	182.6	314.4	134.6	726.5

*Assume the Shroud & Test Loop are supported only at the 4 chamber support points.

Figure 20. Load Diagram for Space Power Chamber

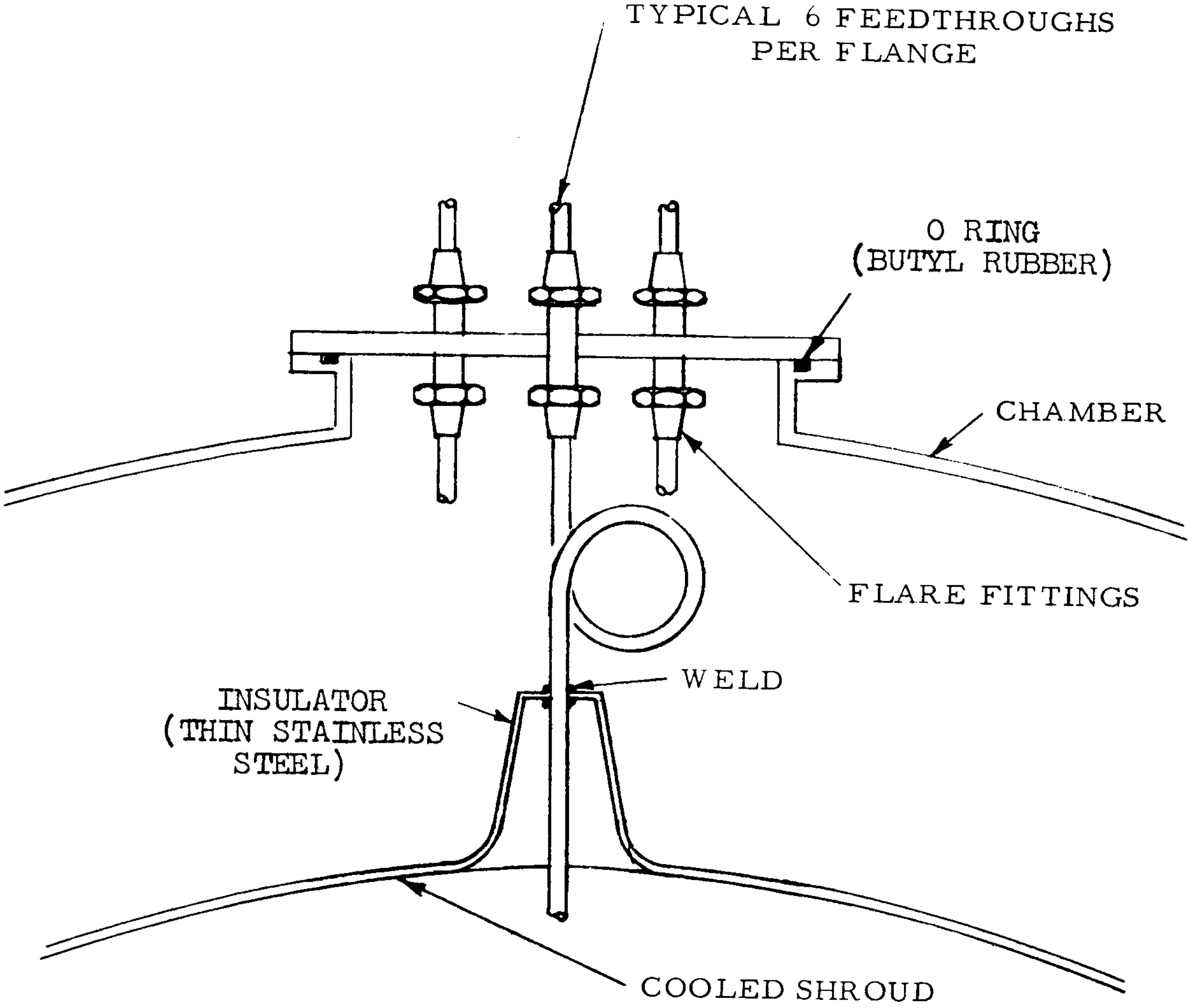


Figure 21. Gas Feedthrough.

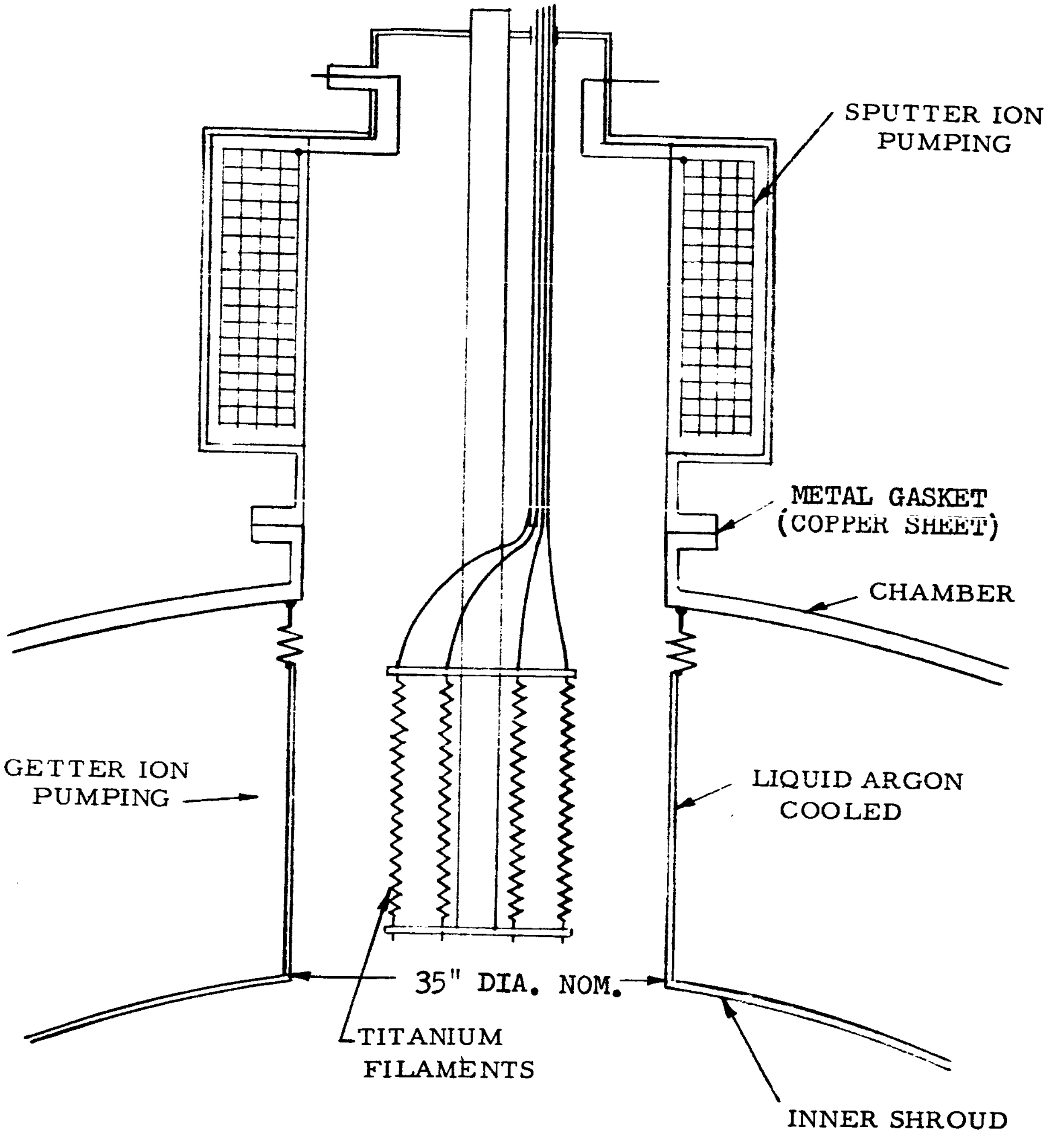


Figure 22. Ion Pump Mounting.

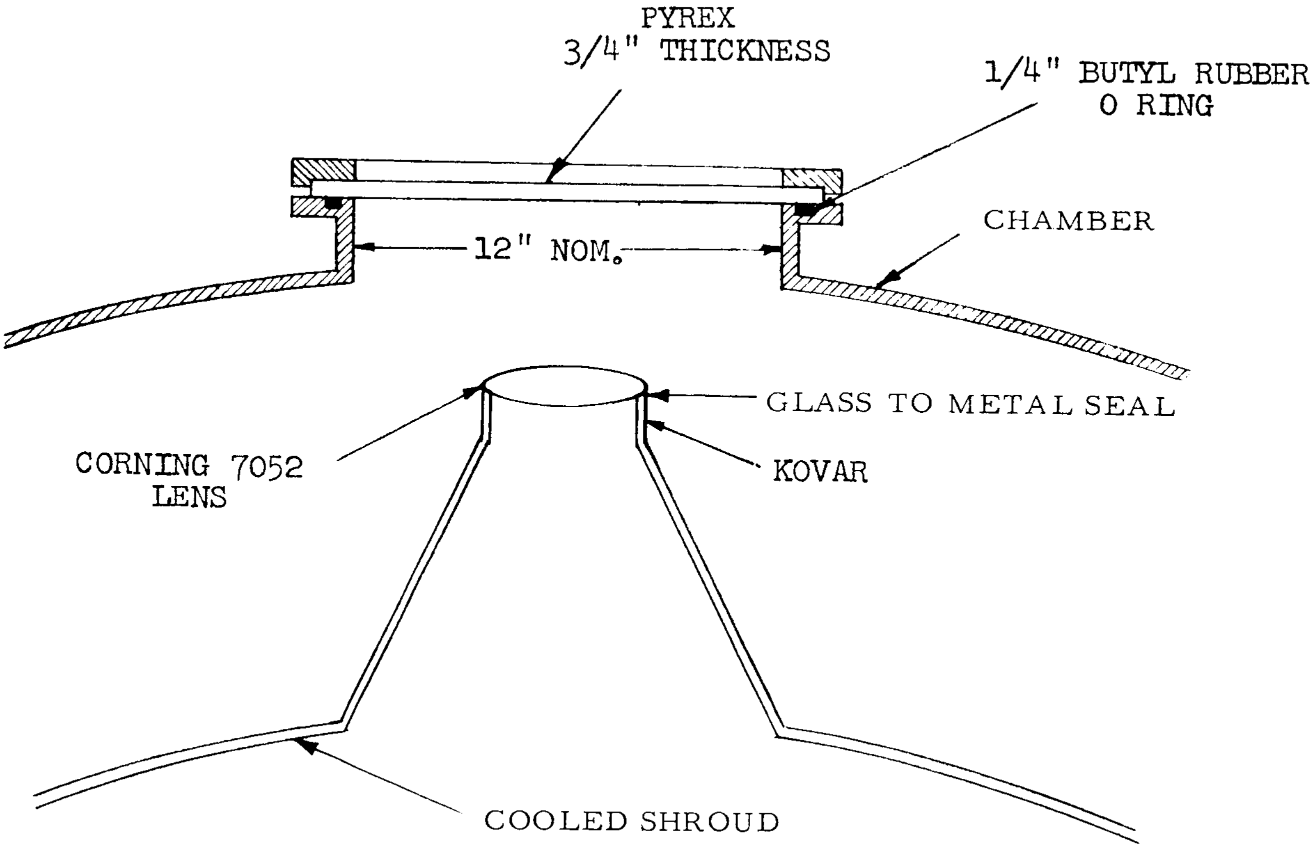


Figure 23. View Port.

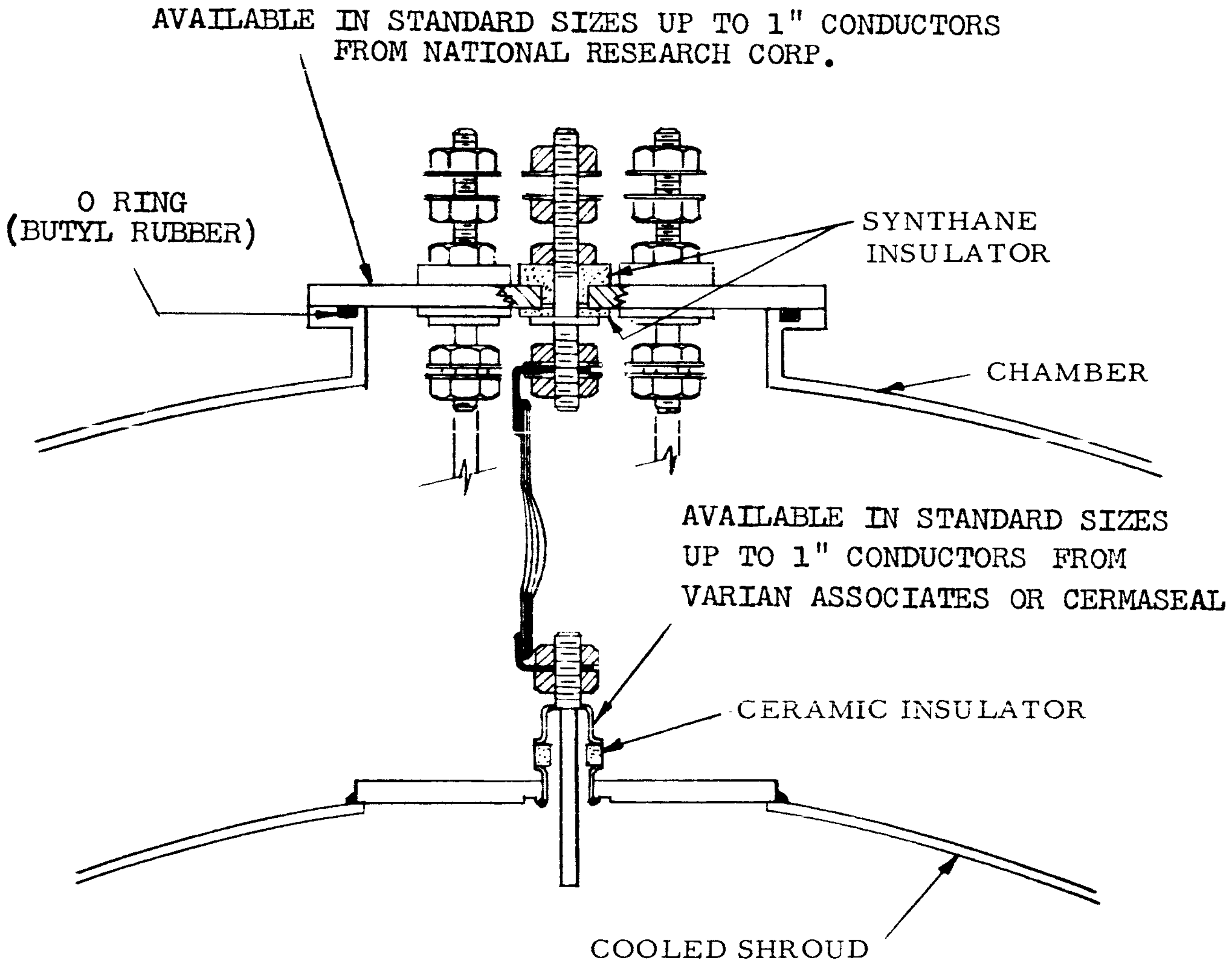


Figure 24. Medium Current Feedthrough.

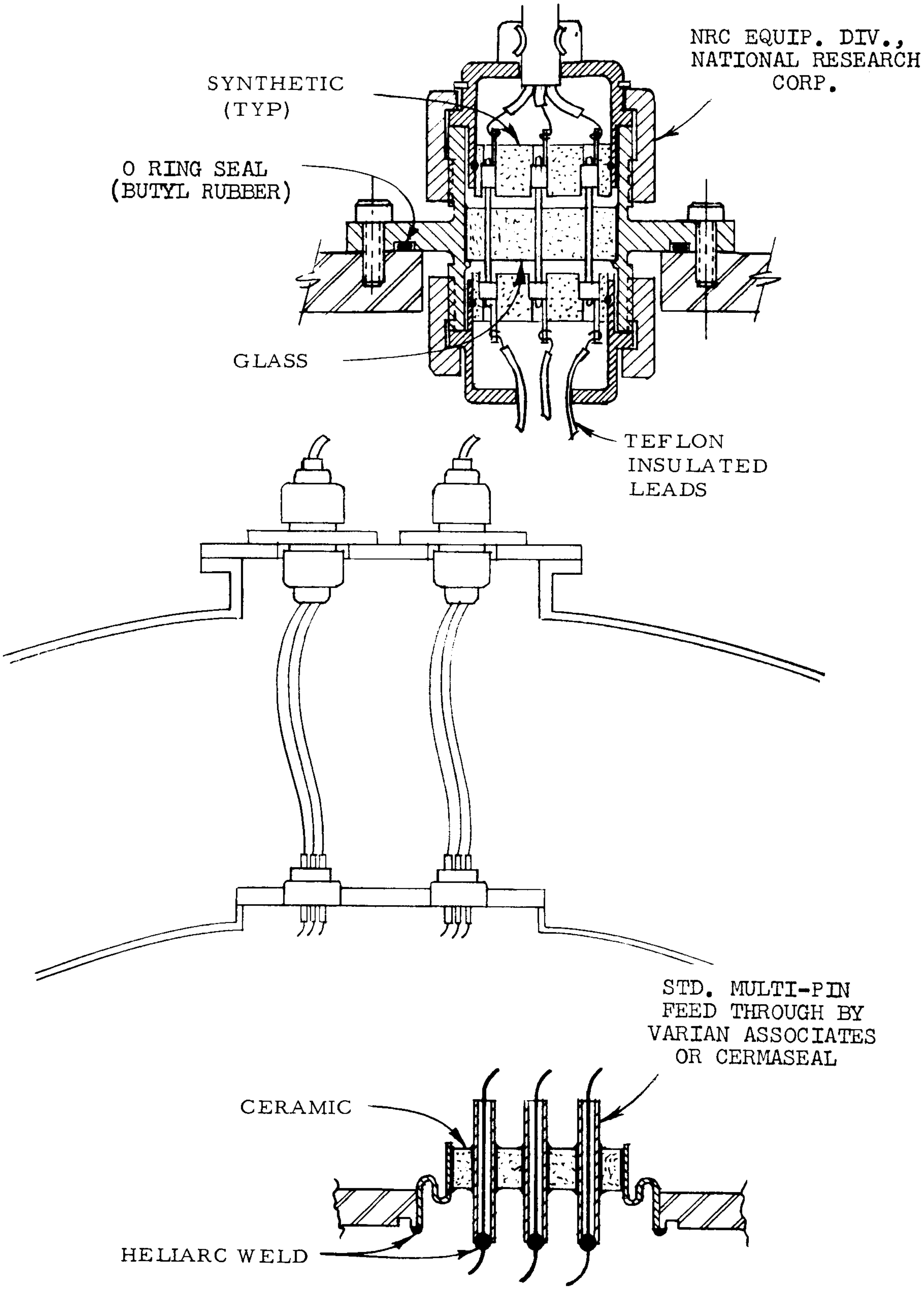
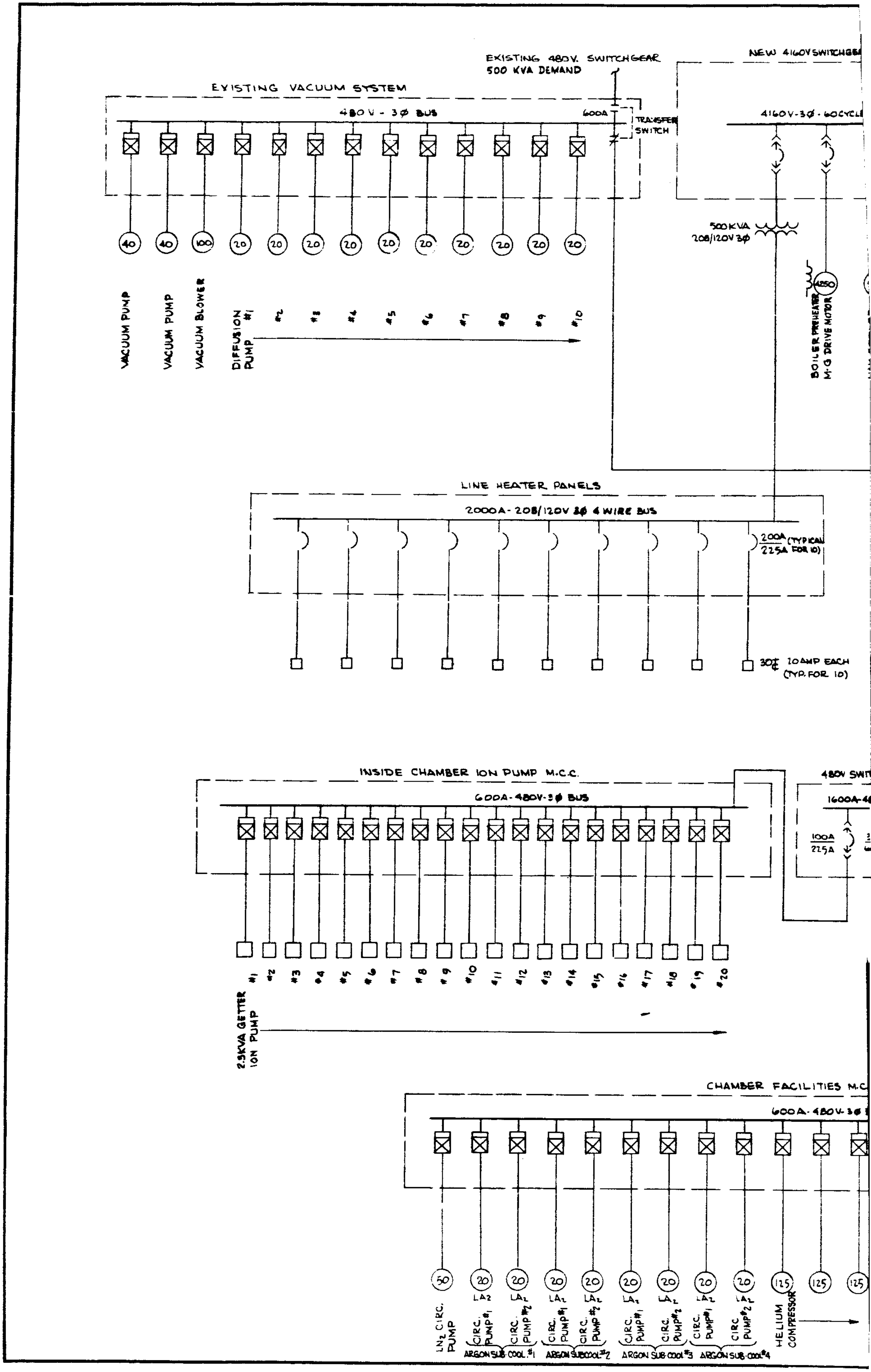
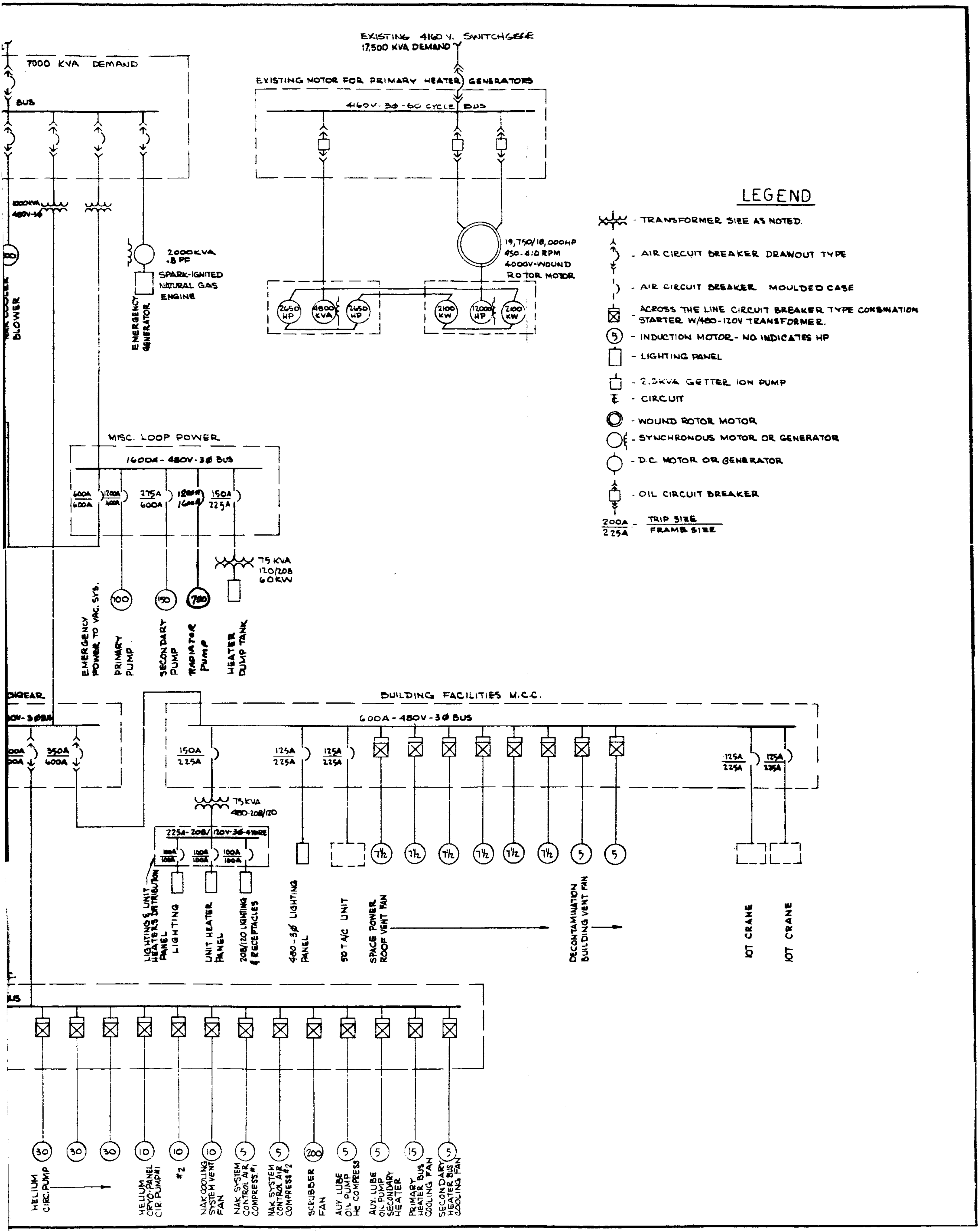


Figure 25. Thermocouple and Instrumentation Feedthrough.


EXISTING 480V. SWITCHGEAR
500 KVA DEMAND
EXISTING VACUUM SYSTEM
480V - 3Ø BUS
600A
TRANSFER SWITCH
40
40
100
20
20
20
20
20
20
20
20
20
20
VACUUM PUMP
VACUUM PUMP
VACUUM BLOWER
DIFFUSION PUMP #1
#2
#3
#4
#5
#6
#7
#8
#9
#10
NEW 4160V SWITCHGE
4160V-3Ø-60CYCL
500KVA
208/120V 3Ø
4250
BOILER PREHEATER
M-G DRIVE MOTOR
LINE HEATER PANELS
2000A-208/120V 3Ø 4 WIRE BUS
200A/225A (TYPICAL FOR 10)
30Ø 20AMP EACH (TYP. FOR 10)
INSIDE CHAMBER ION PUMP M.C.C.
600A-480V-3Ø BUS
#1
#2
#3
#4
#5
#6
#7
#8
#9
#10
#11
#12
#13
#14
#15
#16
#17
#18
#19
#20
2.5KVA GETTER ION PUMP
480V SWI
1600A-4
100A/225A
CHAMBER FACILITIES M.C
600A-480V-3Ø
50
20
20
20
20
20
20
20
20
125
125
125
LA2
LA2
LA2
LA2
LA2
LA2
LA2
LA2
LN2 CIRC. PUMP
CIRC. PUMP #1
CIRC. PUMP #2
ARGON SUB-COOL #1
CIRC. PUMP #1
CIRC. PUMP #2
ARGON SUBCOOL #2
CIRC. PUMP #1
CIRC. PUMP #2
ARGON SUB-COOL #3
CIRC. PUMP #1
CIRC. PUMP #2
ARGON SUB-COOL #4
HELIUM COMPRESSOR

205-1

Figu



e 26. One Line Diagram.

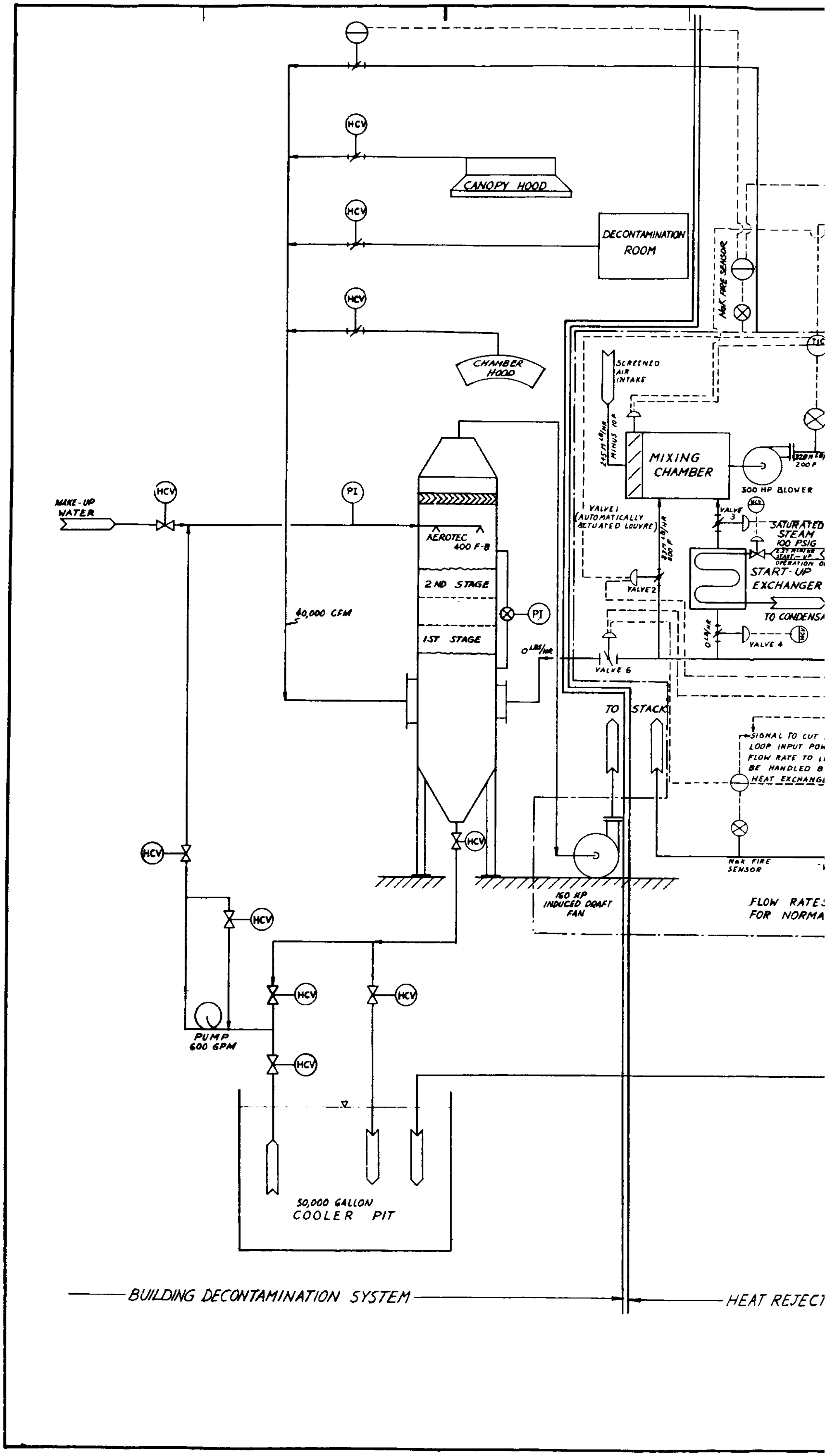
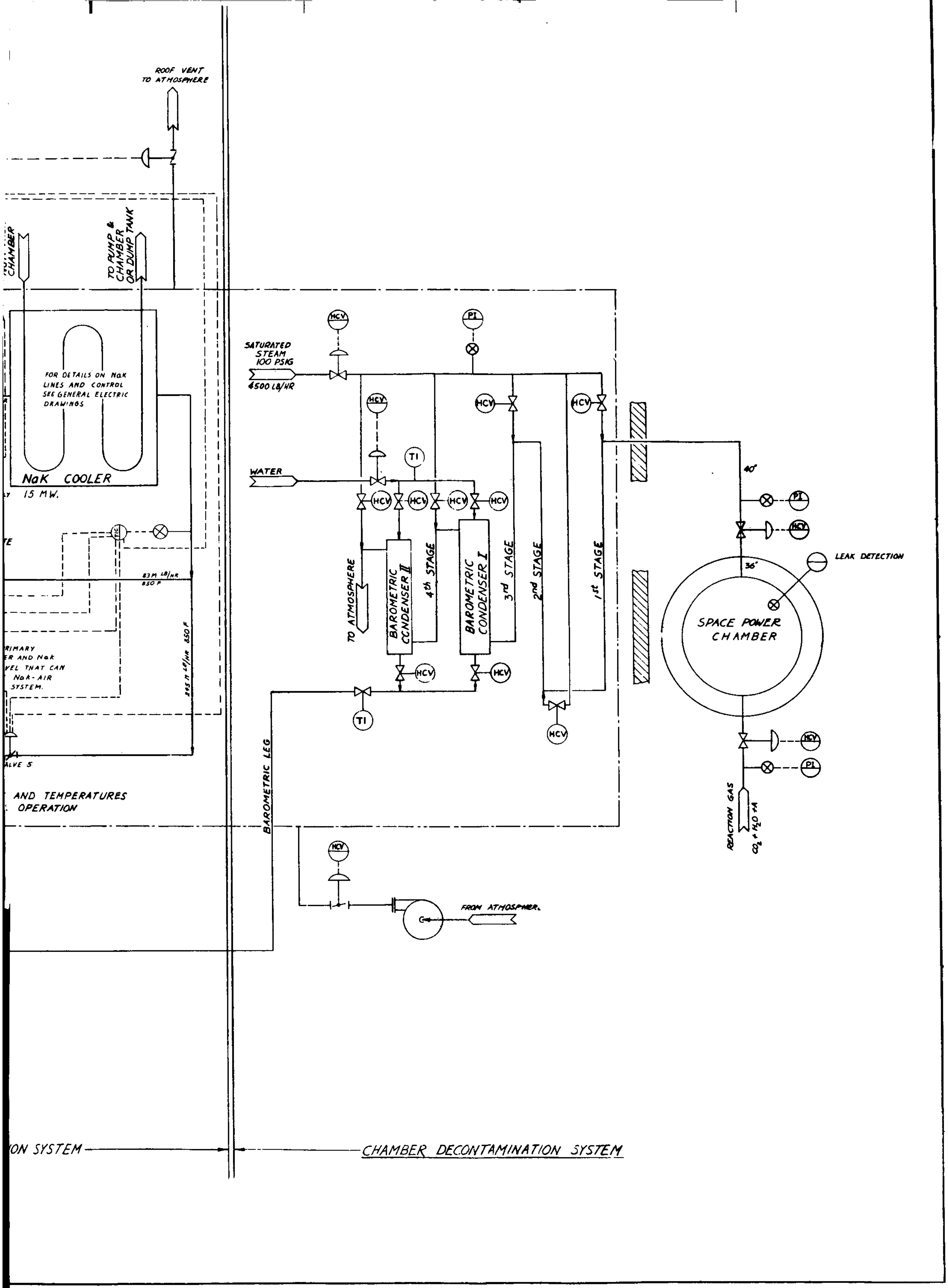

CANOPY HOOD
DECONTAMINATION ROOM
HCV
HCV
HCV
NaK FIRE SENSOR
TIC
CHAMBER HOOD
SCREENED AIR INTAKE
245 M LB/HR MINUS 10 F
MIXING CHAMBER
328 M LB/
200 F
300 HP BLOWER
VALVE 1 (AUTOMATICALLY ACTUATED LOUVRE)
83 M LB/HR 800 F
VALVE 3
HCV
SATURATED STEAM 100 PSIG
2.27 M LB/HR START-UP OPERATION O
START-UP EXCHANGER
TO CONDENSA
VALVE 2
0 LB/HR
VALVE 4
HCV
MAKE-UP WATER
HCV
PI
AEROTEC 400 F-B
2ND STAGE
1ST STAGE
PI
40,000 CFM
0 LBS/HR
VALVE 6
TO STACK
SIGNAL TO CUT
LOOP INPUT POW
FLOW RATE TO L
BE HANDLED B
HEAT EXCHANG
NaK FIRE SENSOR
HCV
HCV
160 HP INDUCED DRAFT FAN
FLOW RATES
FOR NORMA
HCV
PUMP 600 GPM
HCV
HCV
HCV
50,000 GALLON COOLER PIT
BUILDING DECONTAMINATION SYSTEM
HEAT REJECT

Figure 27.



Decontamination and Heat Rejection System - Flow Diagram.

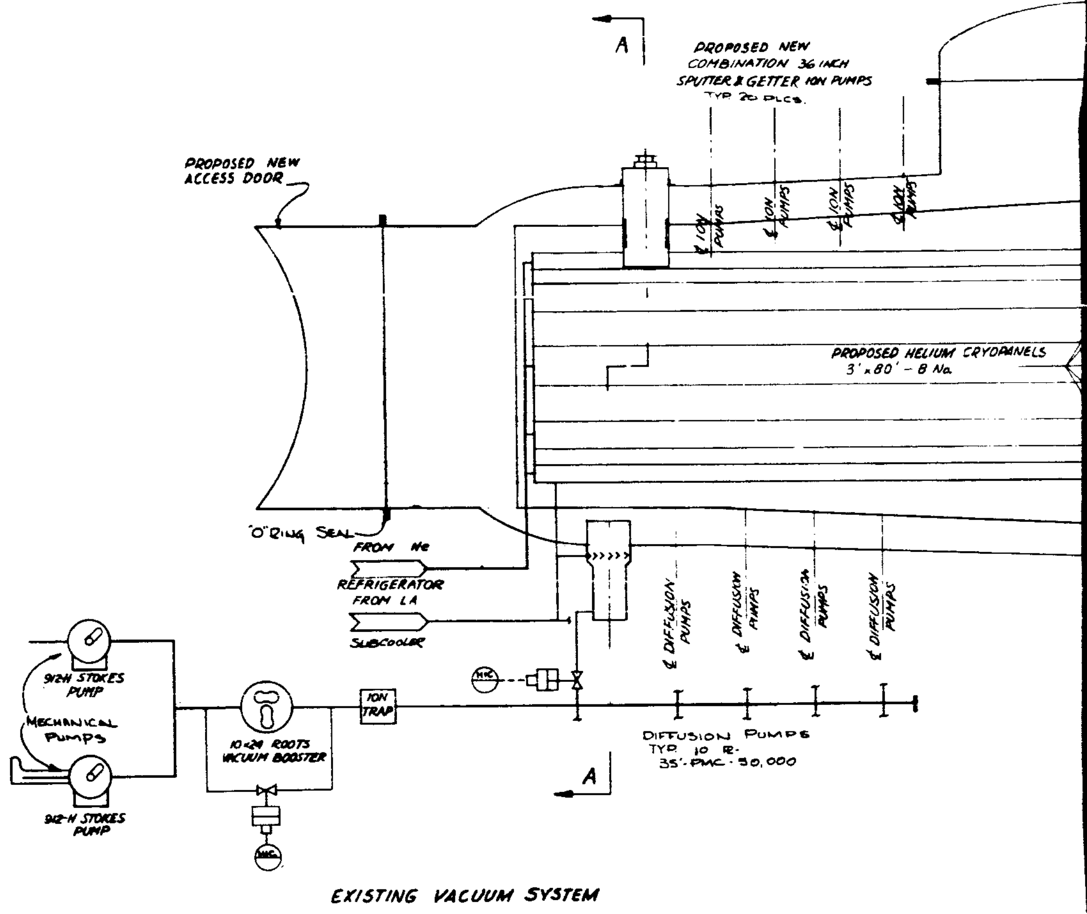
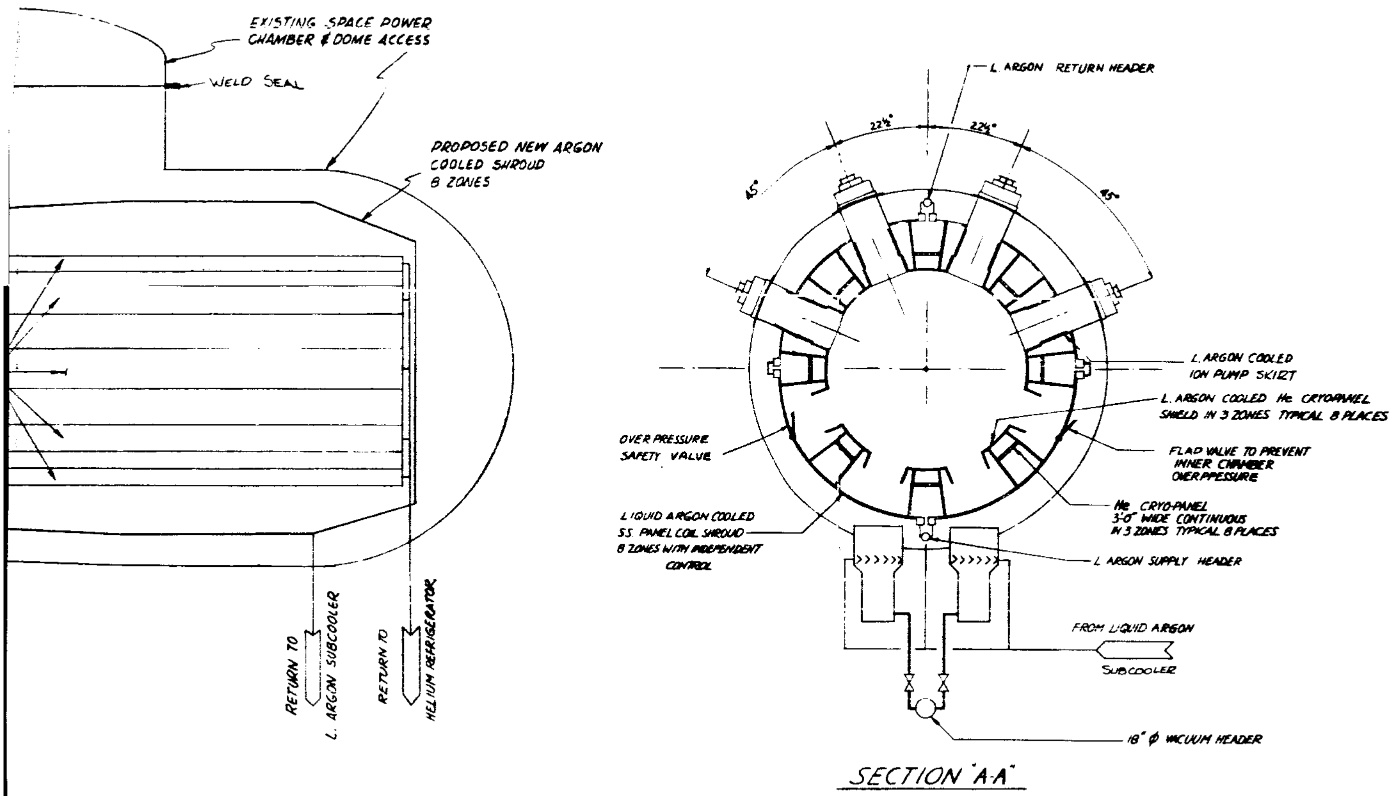


Figure 28. Cham



er Vacuum and Cryopanel System - Flow Diagram.

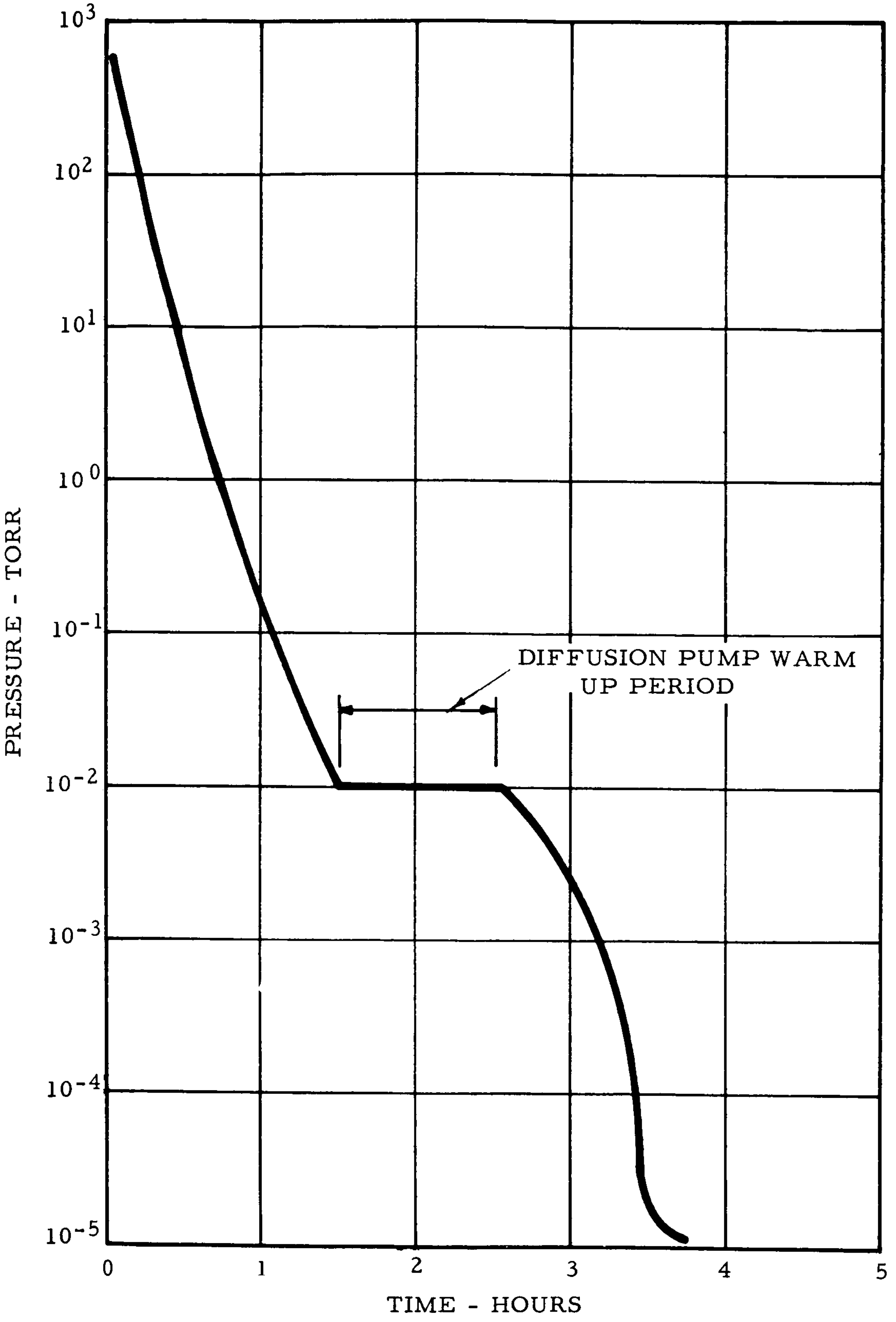


Figure 29. Outer Chamber System Performance.

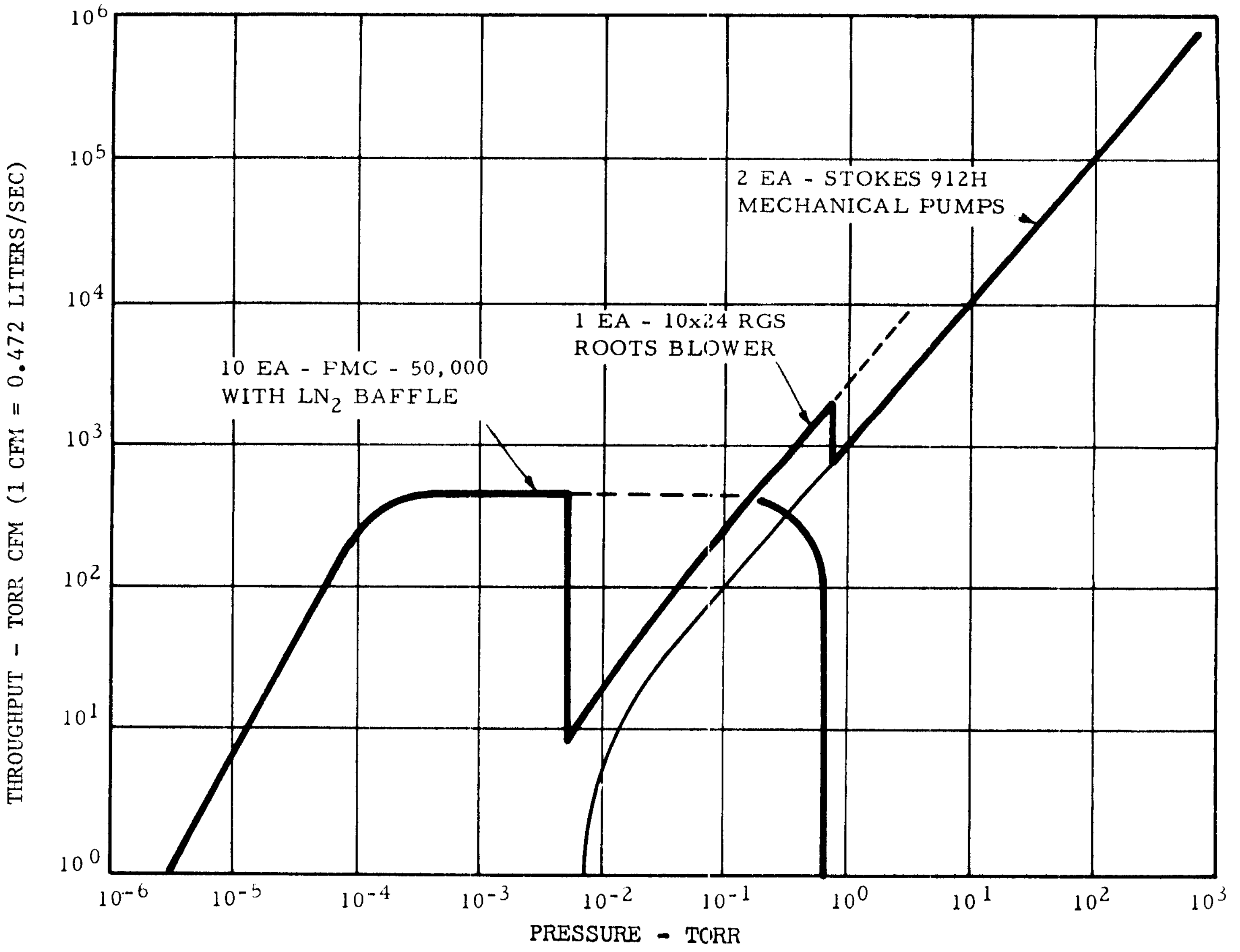


Figure 30. Outer Chamber System Performance.

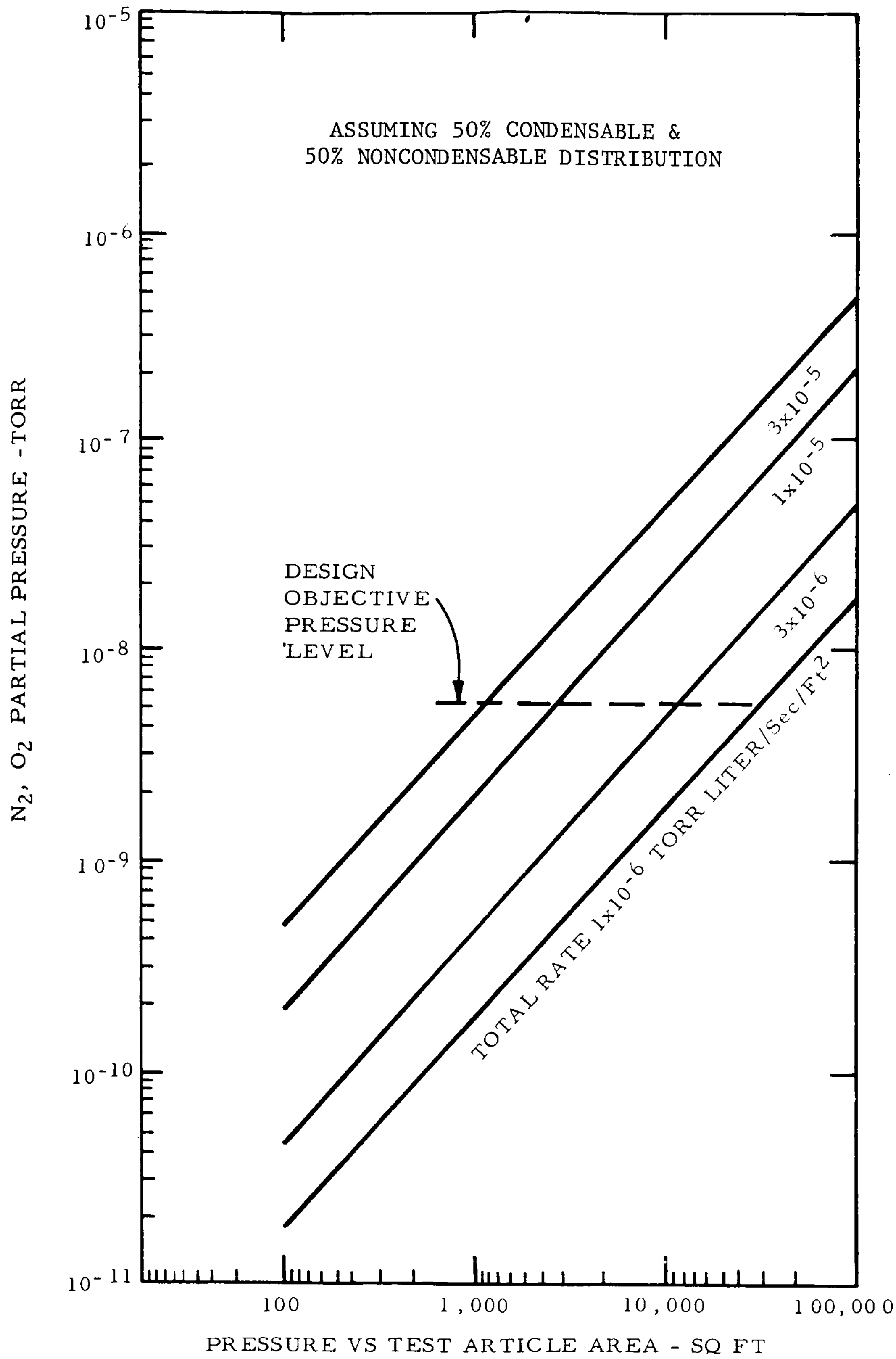


Figure 31. Condensable Partial Pressure vs Test Article Area.

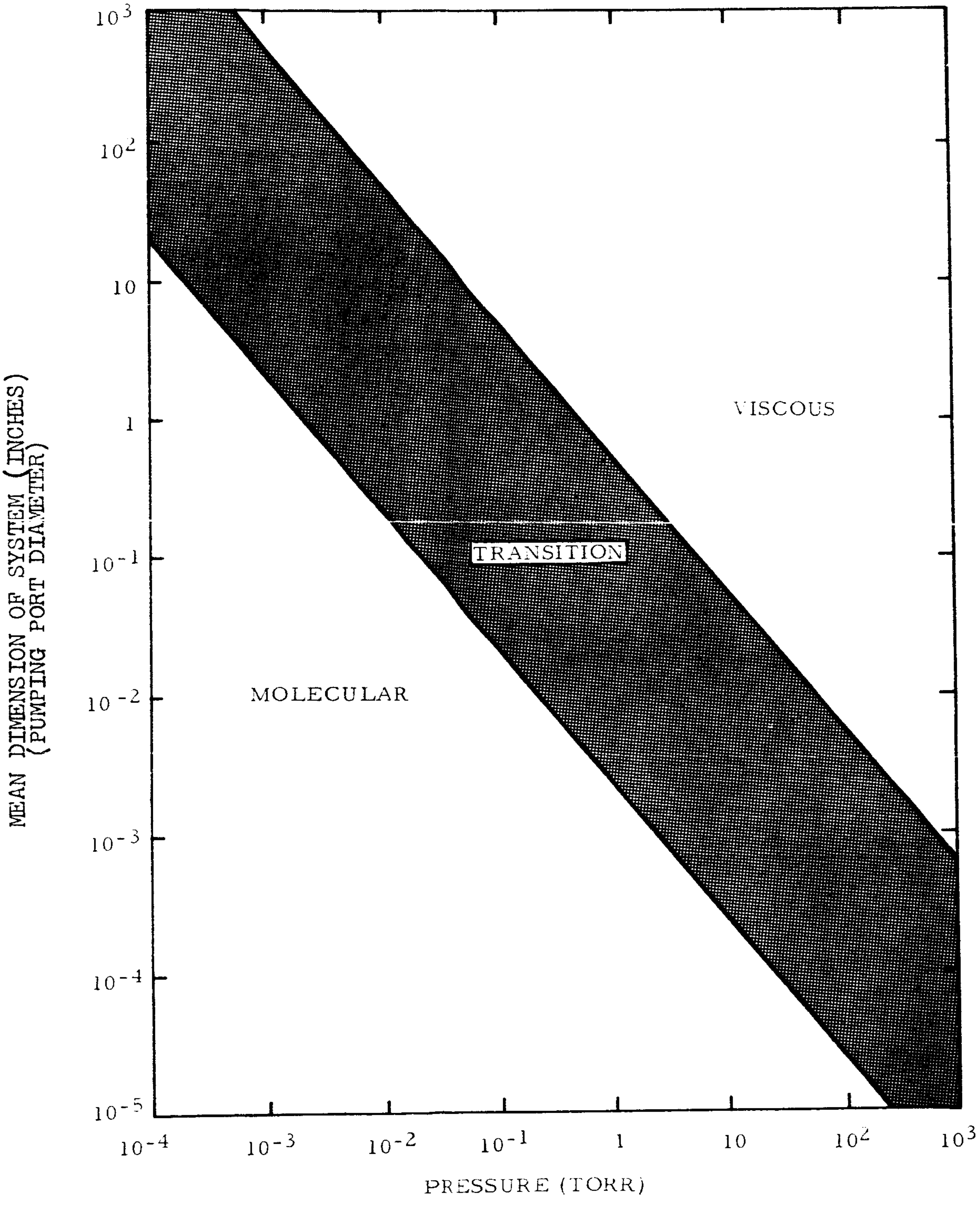


Figure 32. Regimes of Flow for Control of Backstreaming.

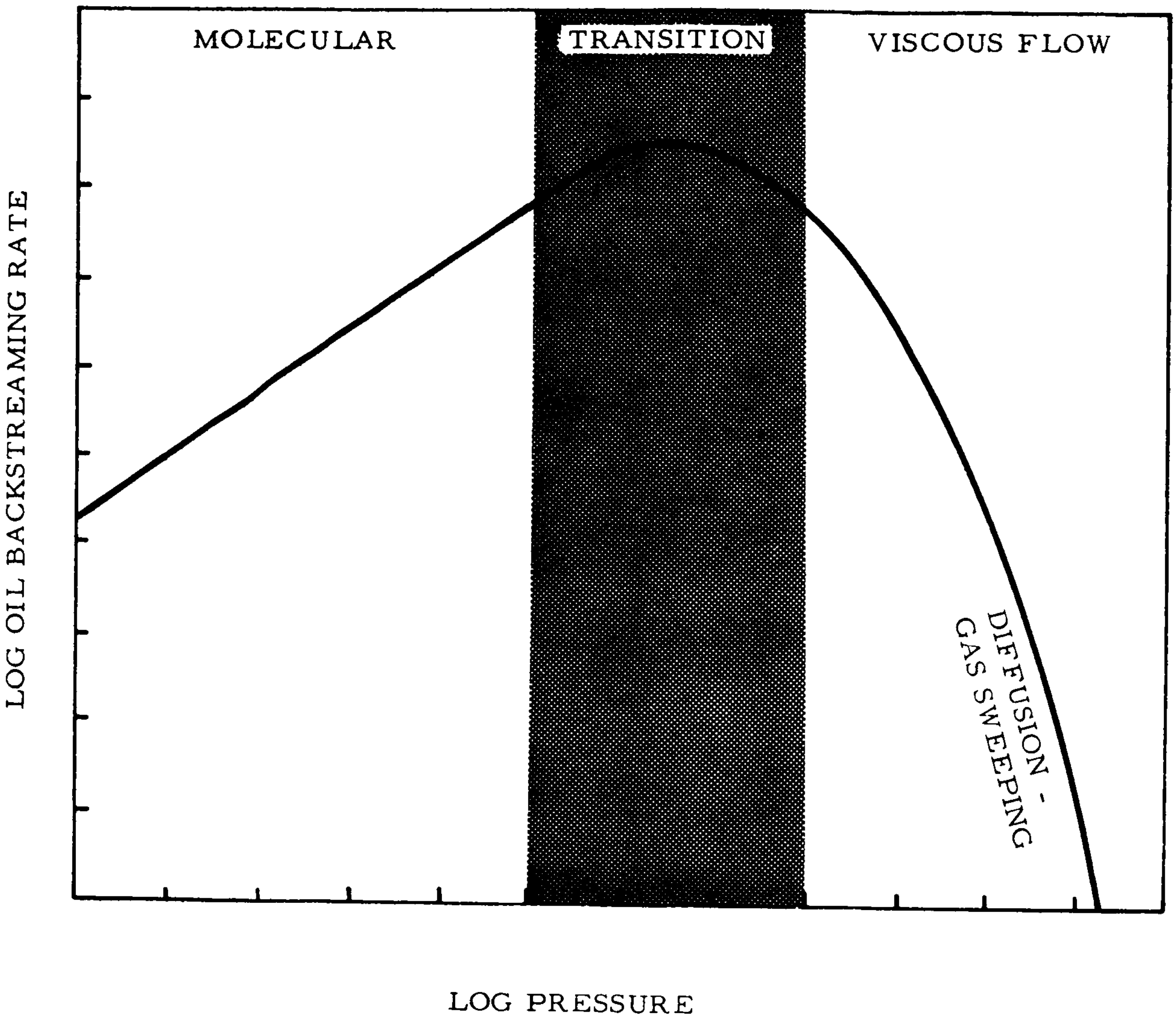


Figure 33. Characteristic Curve of Backstreaming Rate Through Baffle Illustrating Peaking at a Particular Pressure.

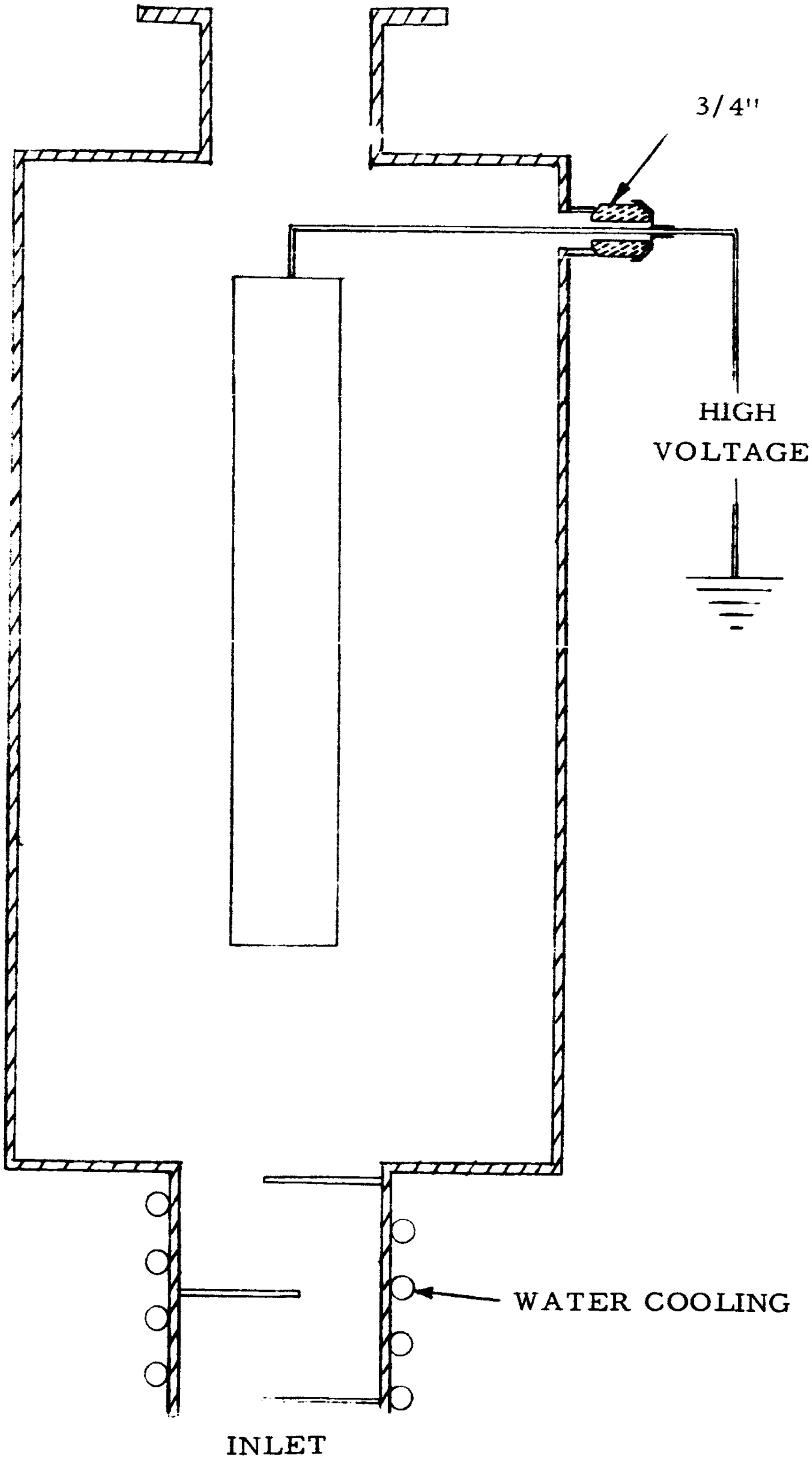
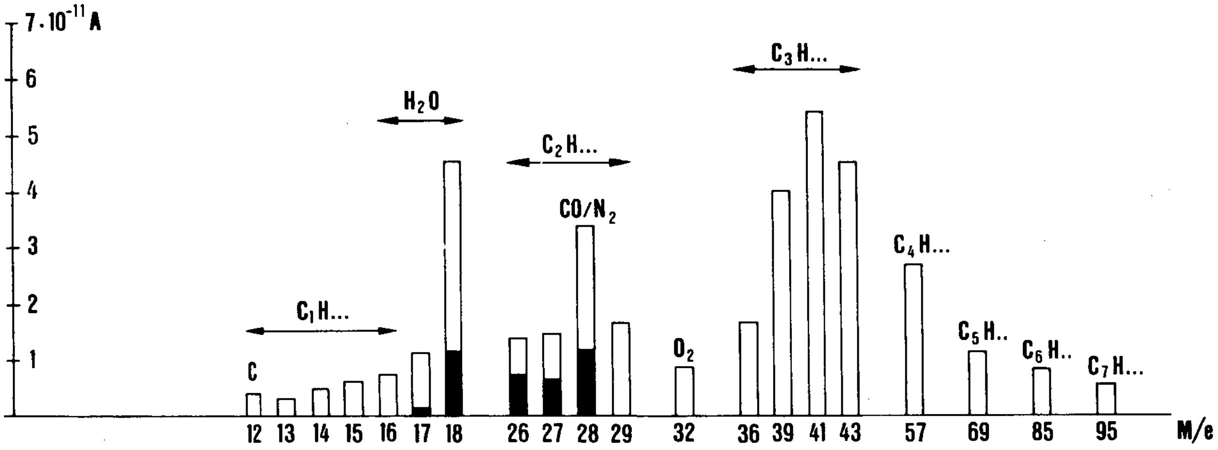


Figure 34. Ion Baffle.

References:

R.A. Haefer: The Ion Baffle, A Novel Electric Hydro Carbon Trap for Rotational Oil Pumps - 1961

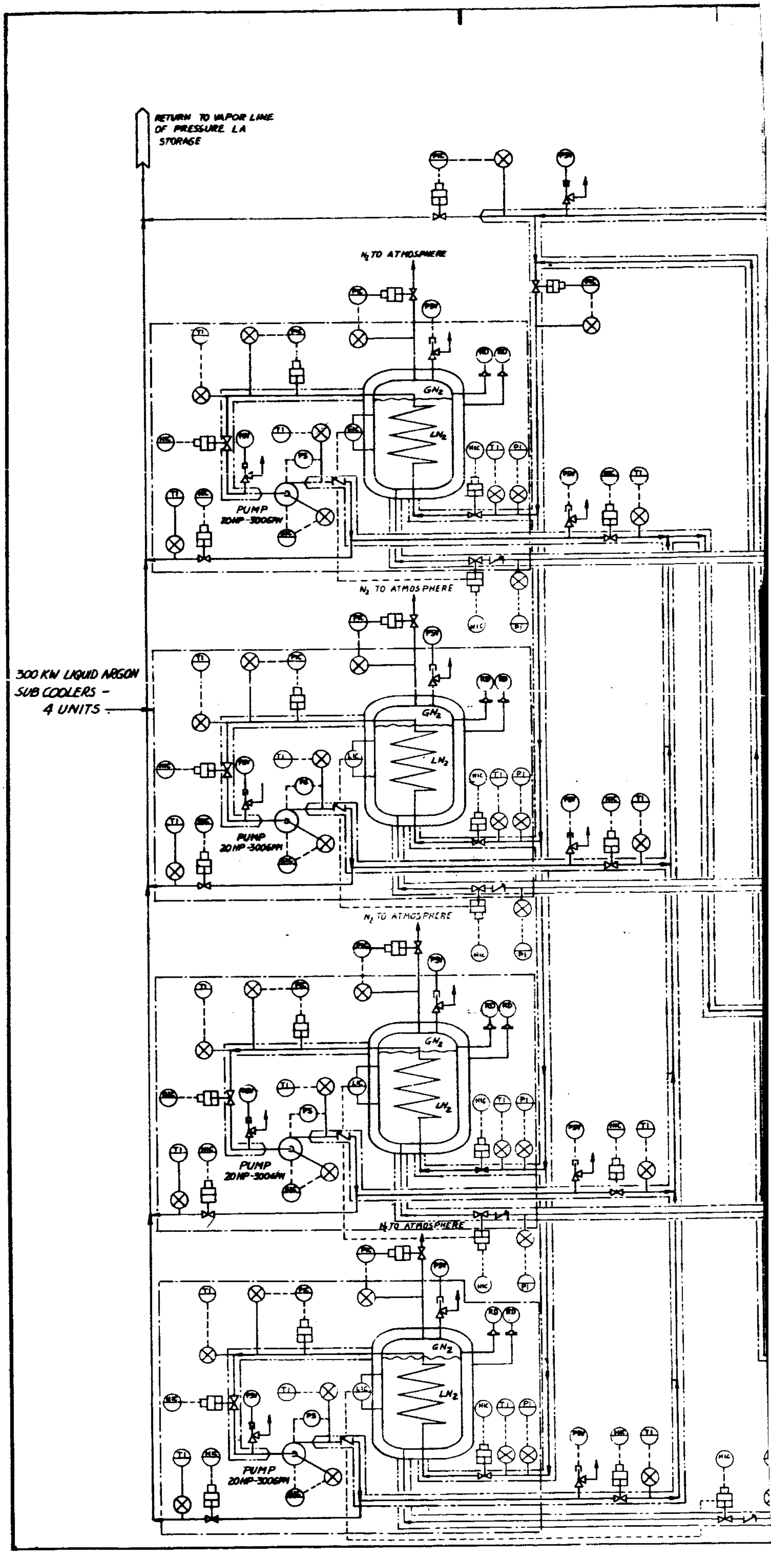
H.J. Schutzer, G. Heyge: Investigations on Applications of the Ion Baffle in the Manufacture of Electron Tubes - 1961



Mass spectrum for the oil-sealed rotary pump

▭ without Ion Baffle ▬ with Ion Baffle

Figure 35. Ion Baffle Control of Backstreaming.


RETURN TO VAPOR LINE OF PRESSURE LA STORAGE
N_2 TO ATMOSPHERE
300 KW LIQUID ARGON SUB COOLERS - 4 UNITS
GN_2
LN_2
PUMP 20HP-300GPM
N_2 TO ATMOSPHERE
GN_2
LN_2
PUMP 20HP-300GPM
N_2 TO ATMOSPHERE
GN_2
LN_2
PUMP 20HP-300GPM
N_2 TO ATMOSPHERE
GN_2
LN_2
PUMP 20HP-300GPM
TI
PIC
PSV
HIC
LIC
PS
PI
RD

2151

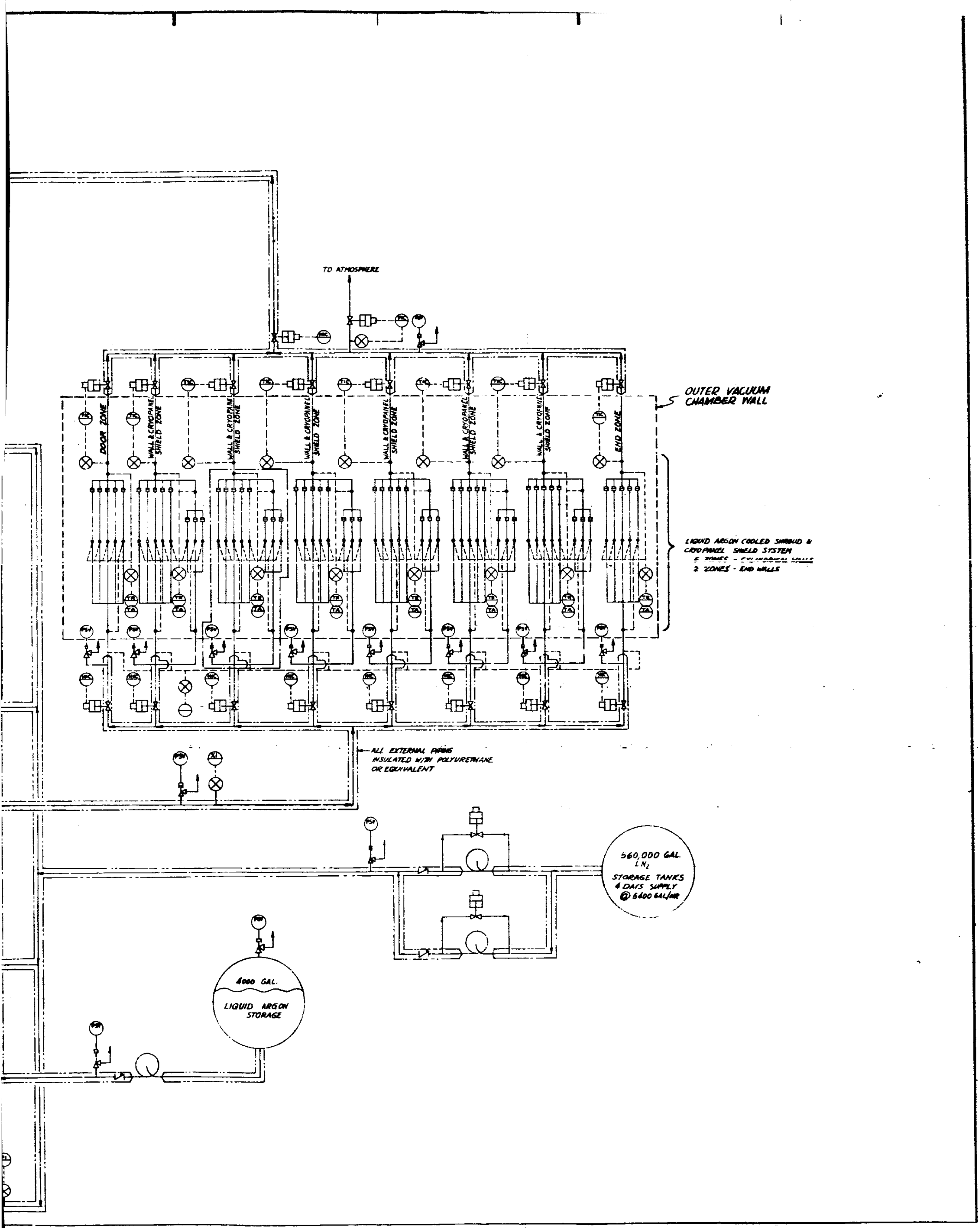


Figure 36. Liquid Argon Cooling System Flow Diagram.

REFERENCES

*1. V. J. Johnson, General Editor, "A Compendium of the Properties of Materials at Low Temperature (Phase 1)", **WADD** Tech. Report 60-56, Oct. 1960.

2. M. W. Kellog **Company

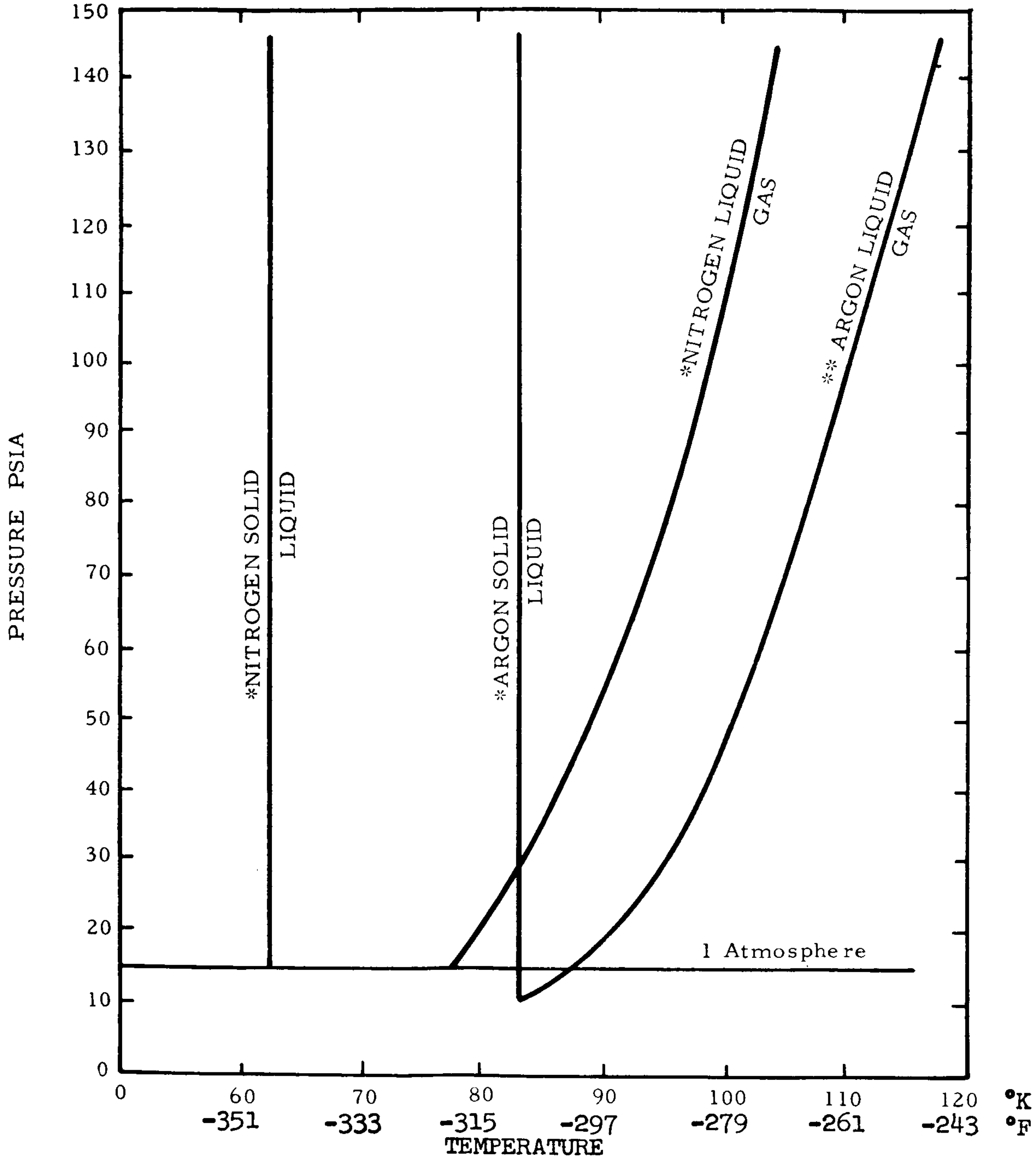
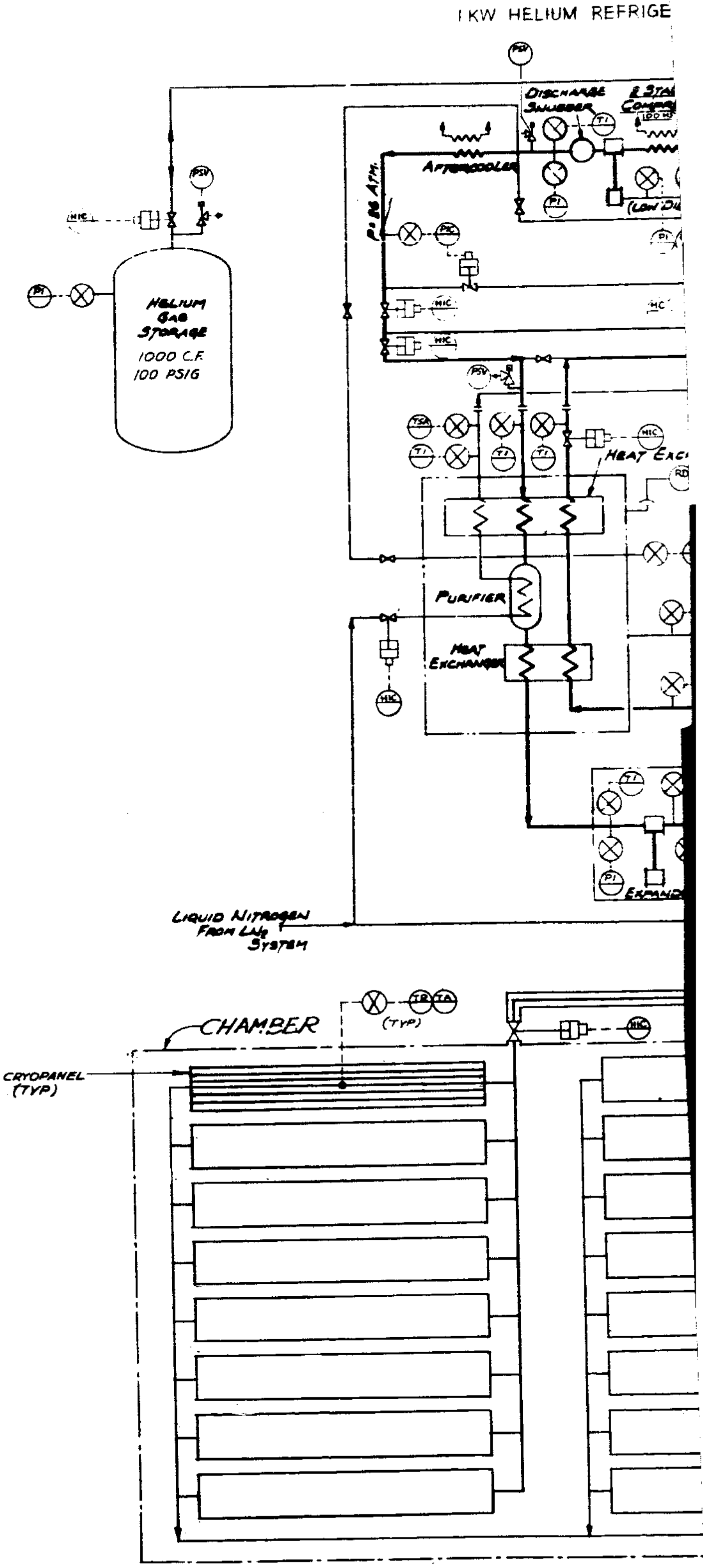
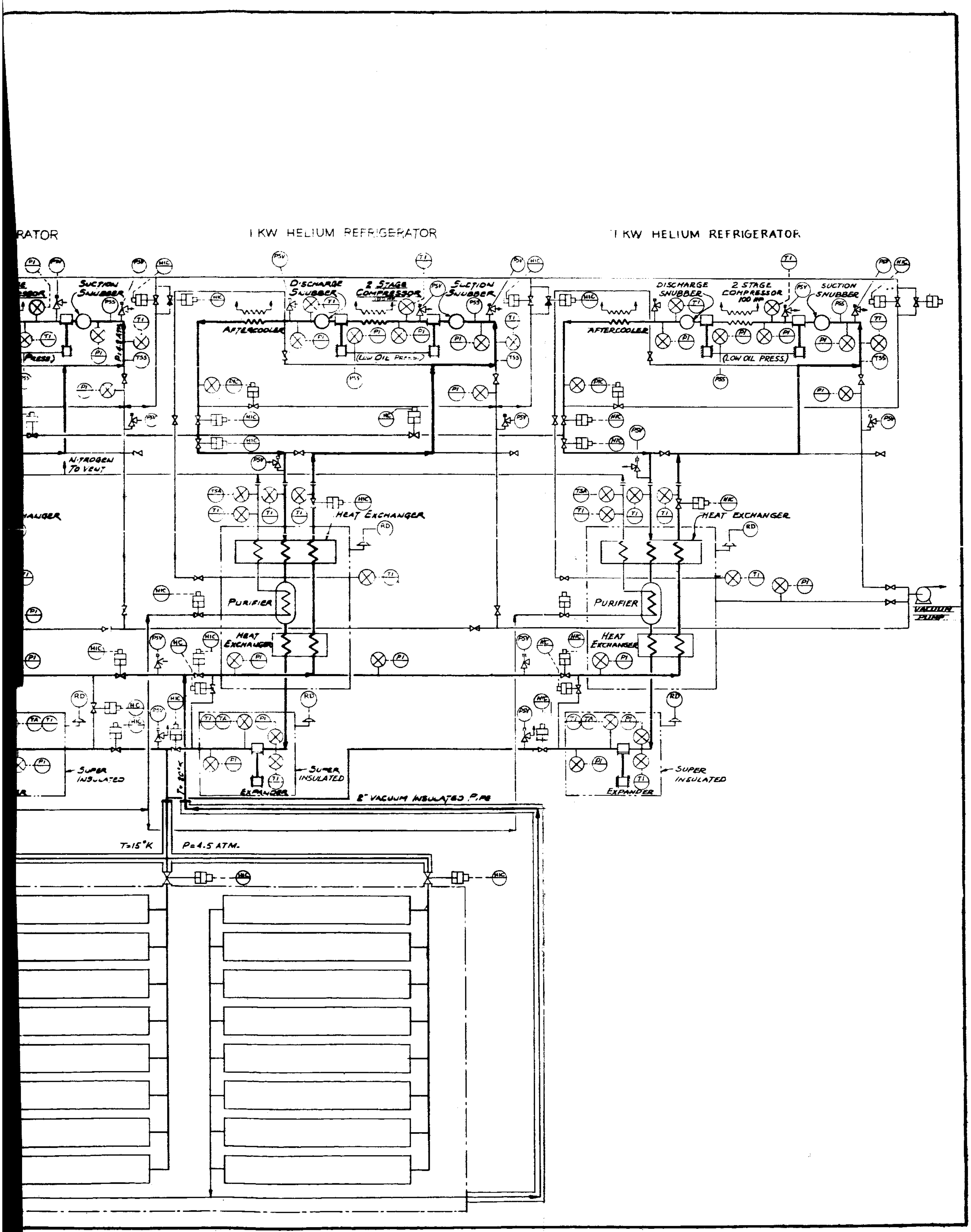


Figure 37. Nitrogen and Argon Phase Diagram.


1 KW HELIUM REFRIGE
PSV
DISCHARGE SNUBBER
TI
AFTERCOOLER
PI
P=86 ATM.
PIC
HIC
HC
HIC
HIC
PI
HELIUM GAS STORAGE
1000 C.F.
100 PSIG
PSV
TSA
TI
TI
TI
HIC
HEAT EXC
RD
PURIFIER
HEAT EXCHANGER
HIC
TI
PI
LIQUID NITROGEN FROM LN2 SYSTEM
CHAMBER
TR
TA
(TYP)
HIC
CRYOPANEL (TYP)

F

2171



igure 38. Helium System Flow Diagram.

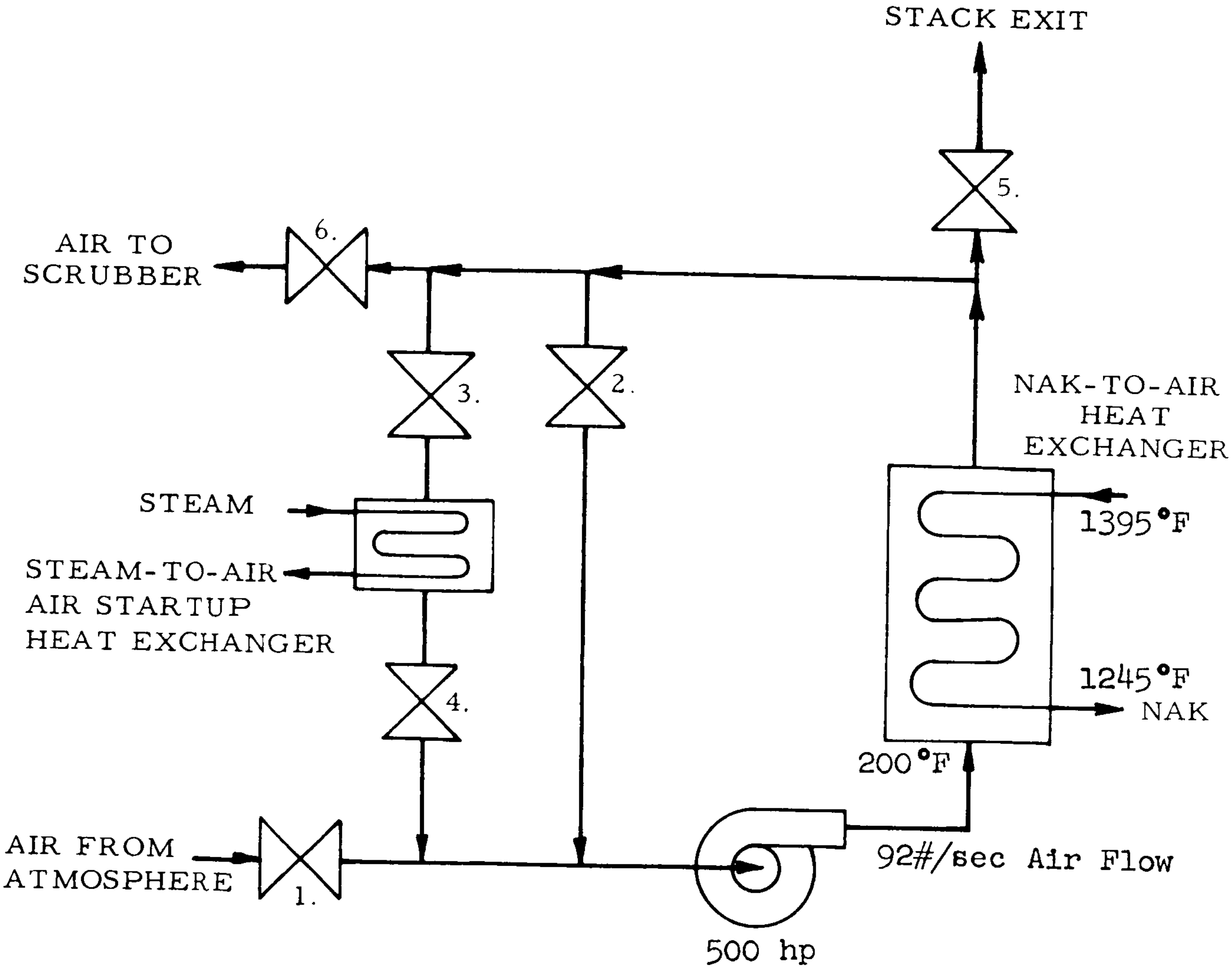
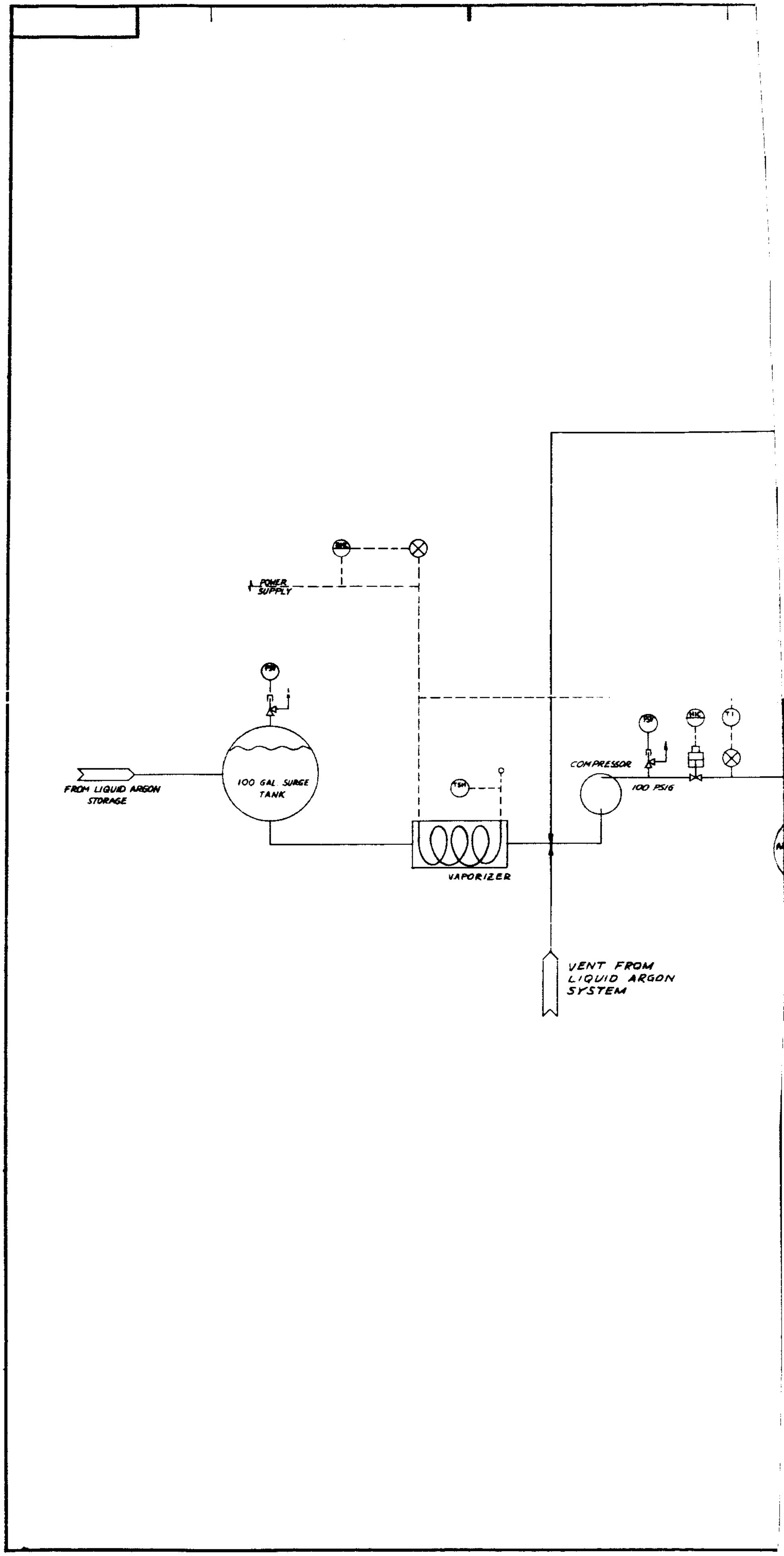


Figure 39. Heat Rejection System Flow Diagram.


POWER SUPPLY
TSH
TSH
FROM LIQUID ARGON STORAGE
100 GAL SURGE TANK
VAPORIZER
COMPRESSOR
100 PSIG
HIC
TI
VENT FROM LIQUID ARGON SYSTEM

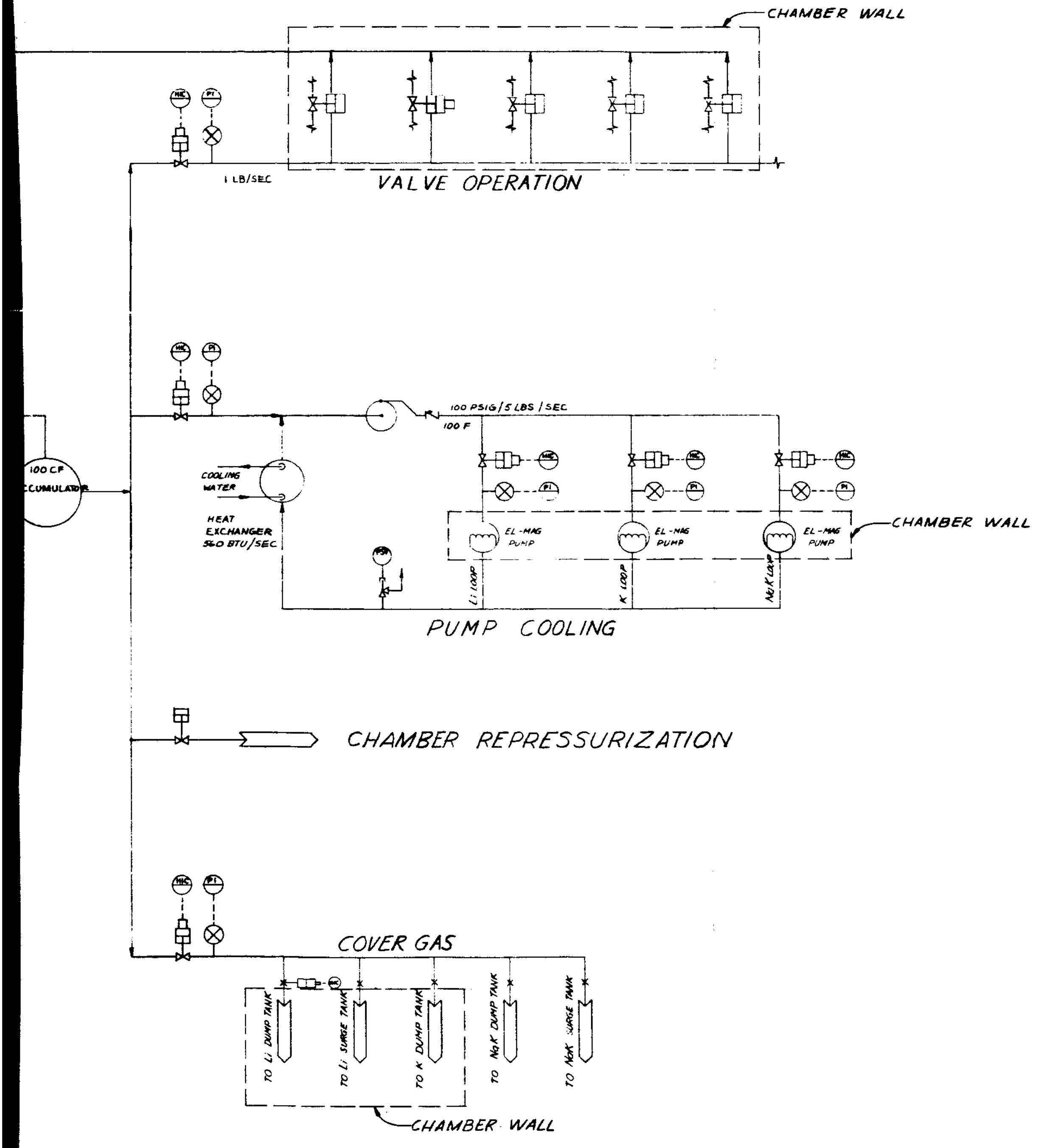


Figure 40. Gaseous Argon System Flow Diagram.

2

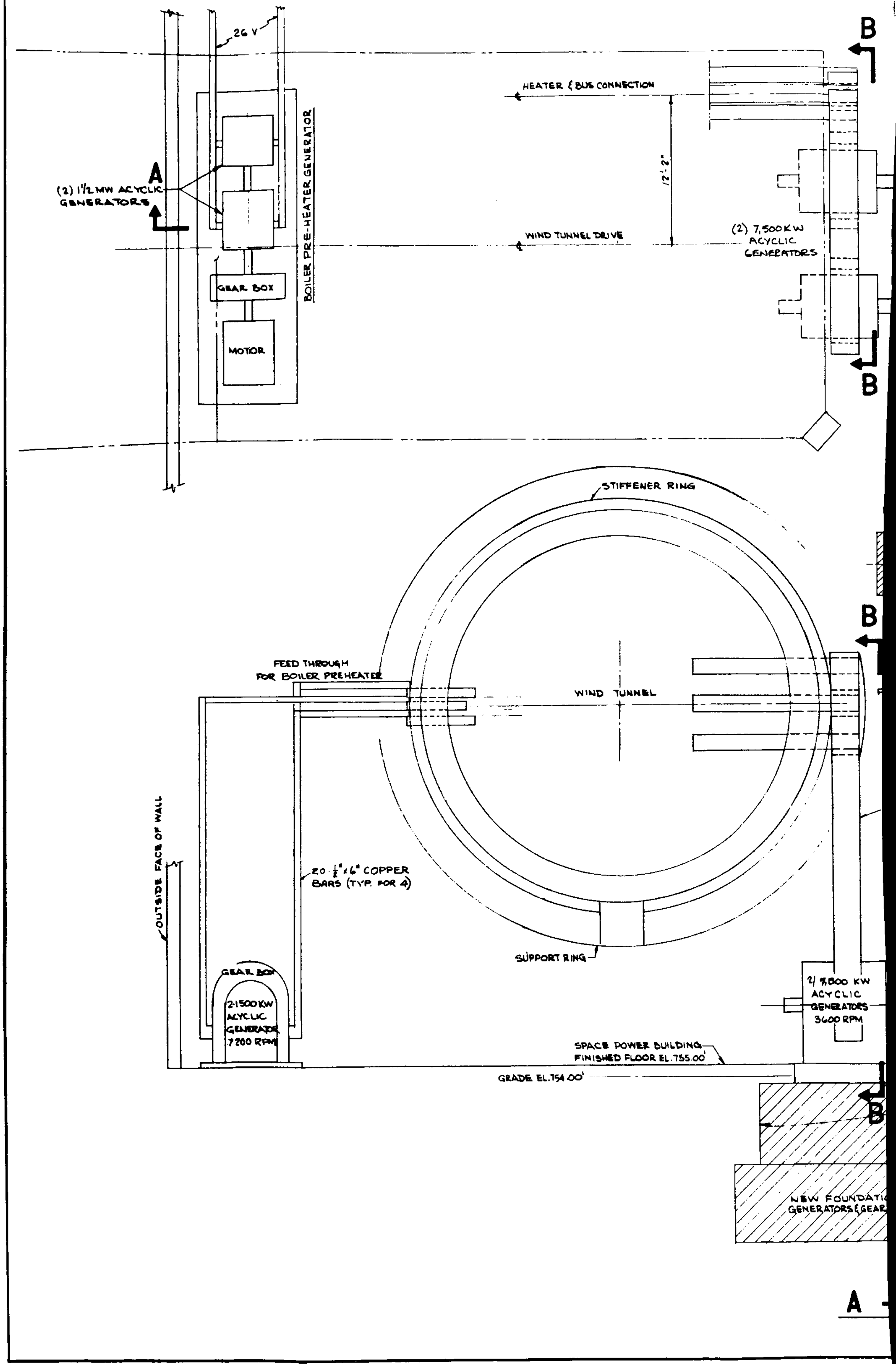
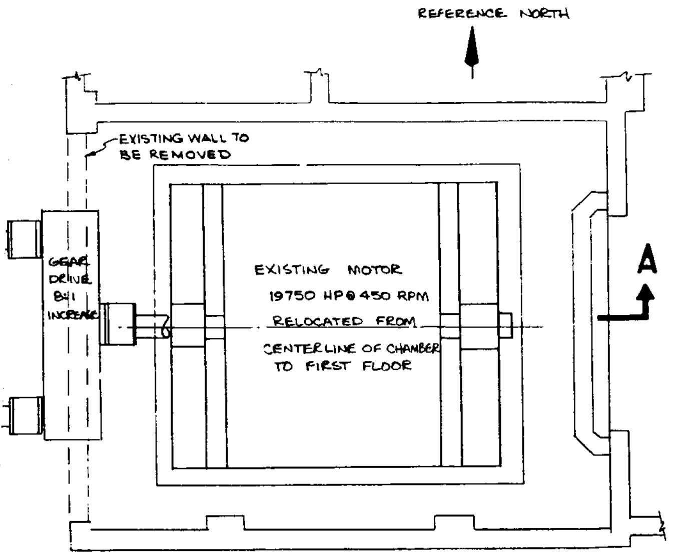
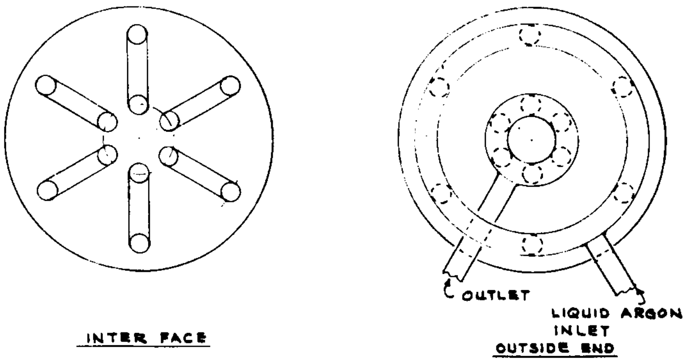

B
26 V
HEATER & BUS CONNECTION
12'-2"
A
(2) 1½ MW ACYCLIC GENERATORS
BOILER PRE-HEATER GENERATOR
WIND TUNNEL DRIVE
(2) 7,500 KW ACYCLIC GENERATORS
GEAR BOX
MOTOR
B
STIFFENER RING
B
FEED THROUGH FOR BOILER PREHEATER
WIND TUNNEL
OUTSIDE FACE OF WALL
20-½"x6" COPPER BARS (TYP. FOR 4)
SUPPORT RING
GEAR BOX
2-1500 KW ACYCLIC GENERATOR 7200 RPM
2/ 7,500 KW ACYCLIC GENERATORS 3600 RPM
SPACE POWER BUILDING FINISHED FLOOR EL. 755.00'
GRADE EL. 754.00'
B
NEW FOUNDATI
GENERATORS & GEAR
A -

Figure 41.



PLAN



BUS COOLING DETAIL

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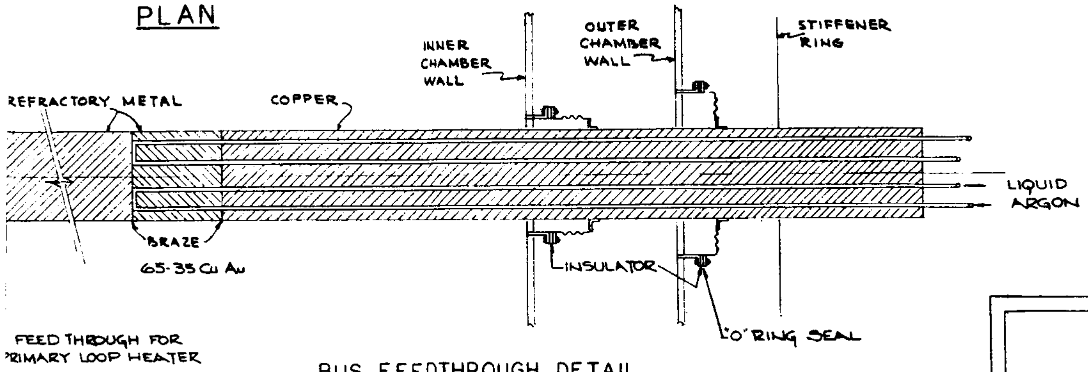
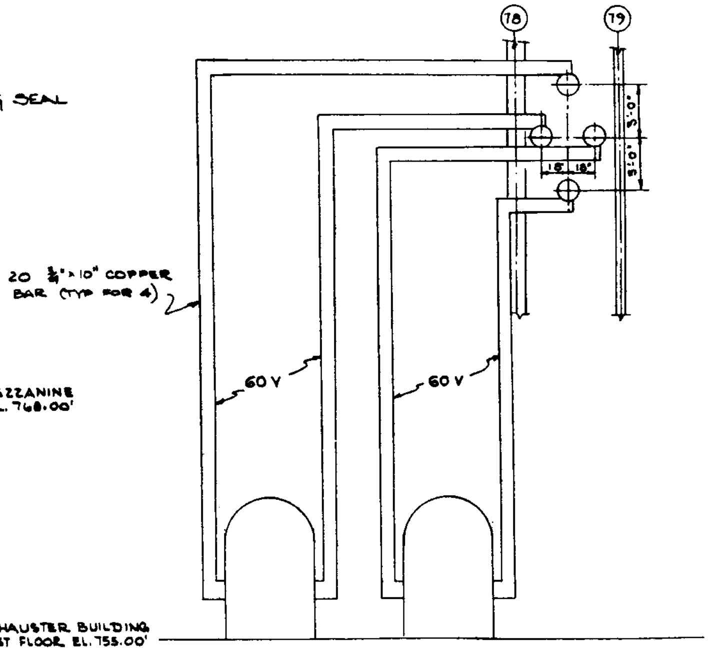
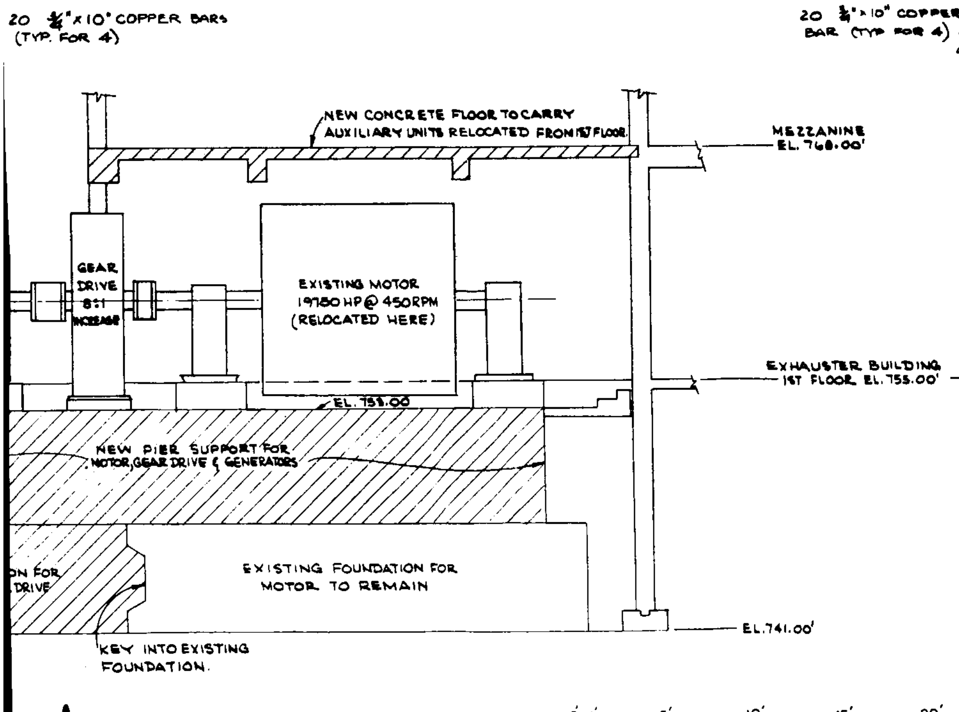


BUS FEEDTHROUGH DETAIL

NOT TO SCALE



B – B



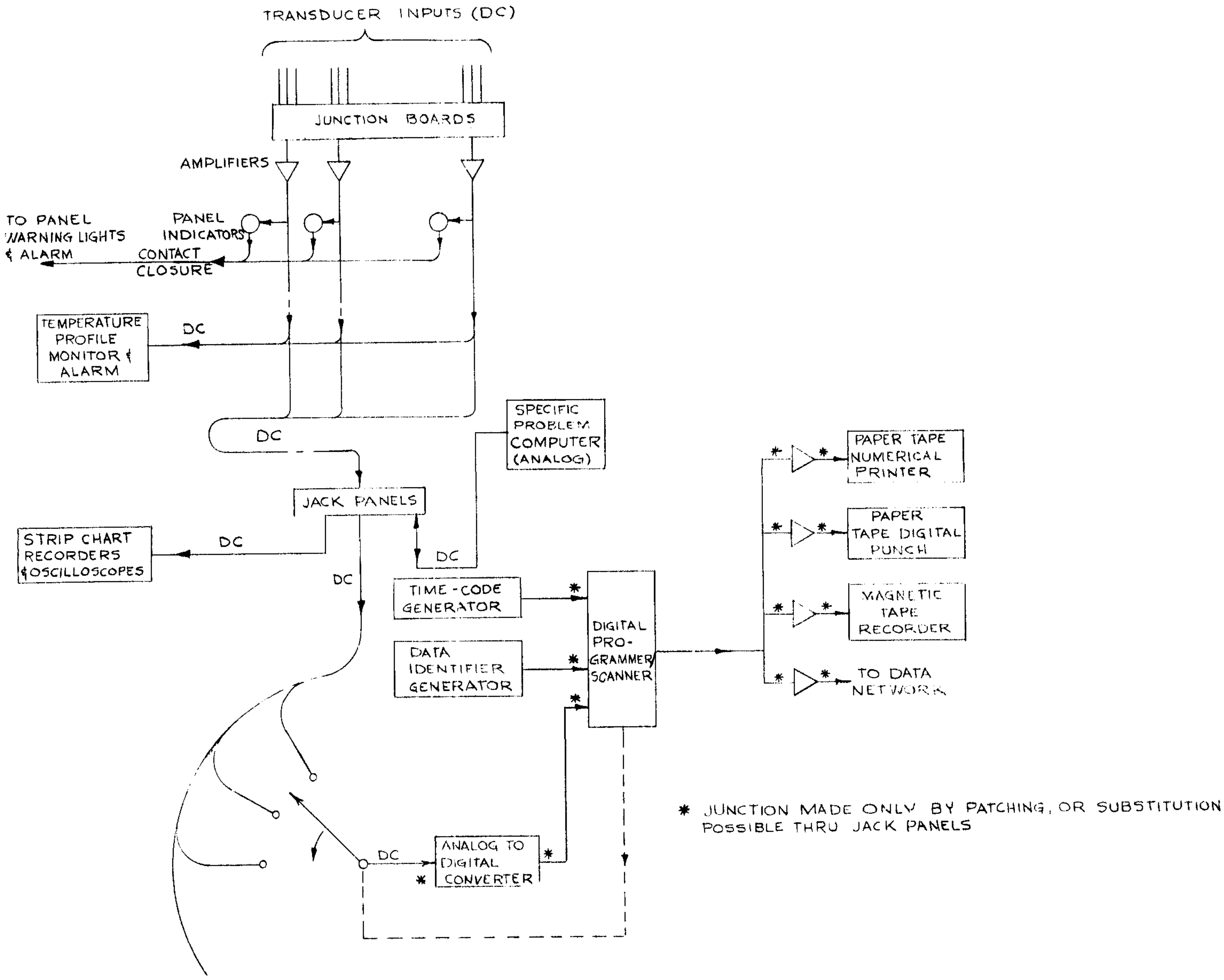
– A

0 1' 5' 10' 15' 20'

SCALE: ¼" TO 1'

Electrical Plan and Sections, Heater Power Supply.

220-2



* JUNCTION MADE ONLY BY PATCHING, OR SUBSTITUTION POSSIBLE THRU JACK PANELS

Figure 42. Instrumentation Data Handling System.

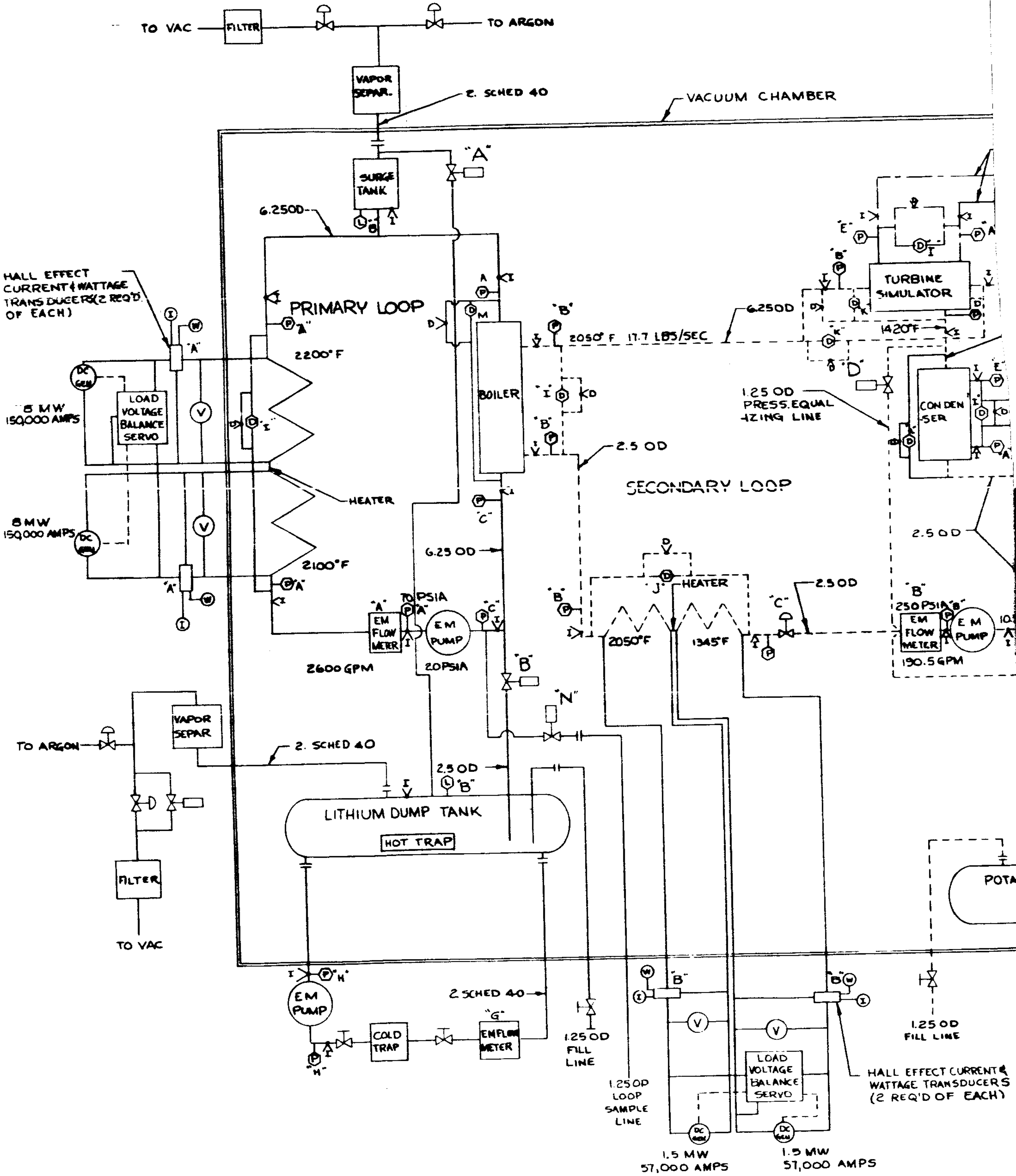
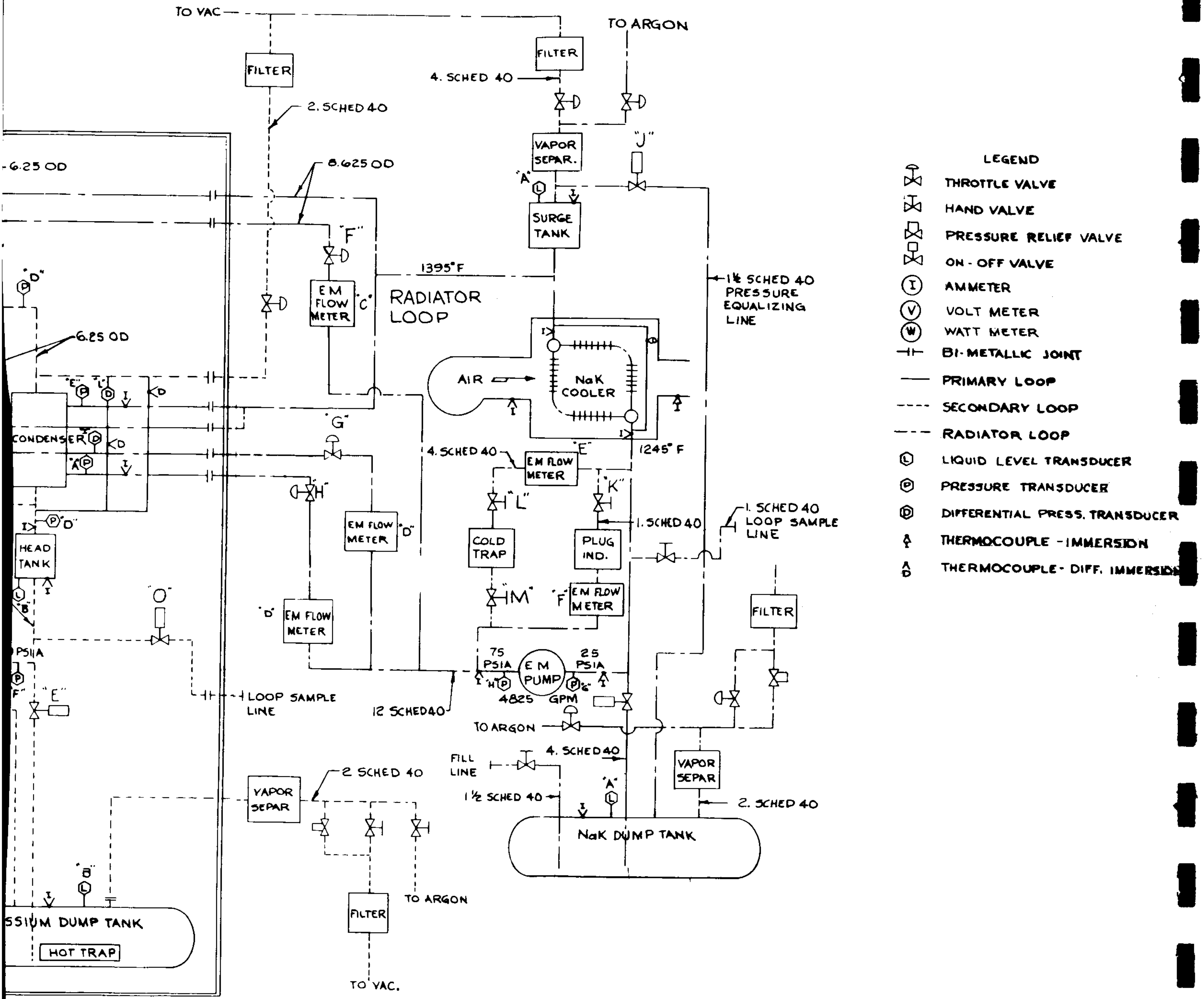


Figure 43. Flow and Loo



Instrumentation Schematic.

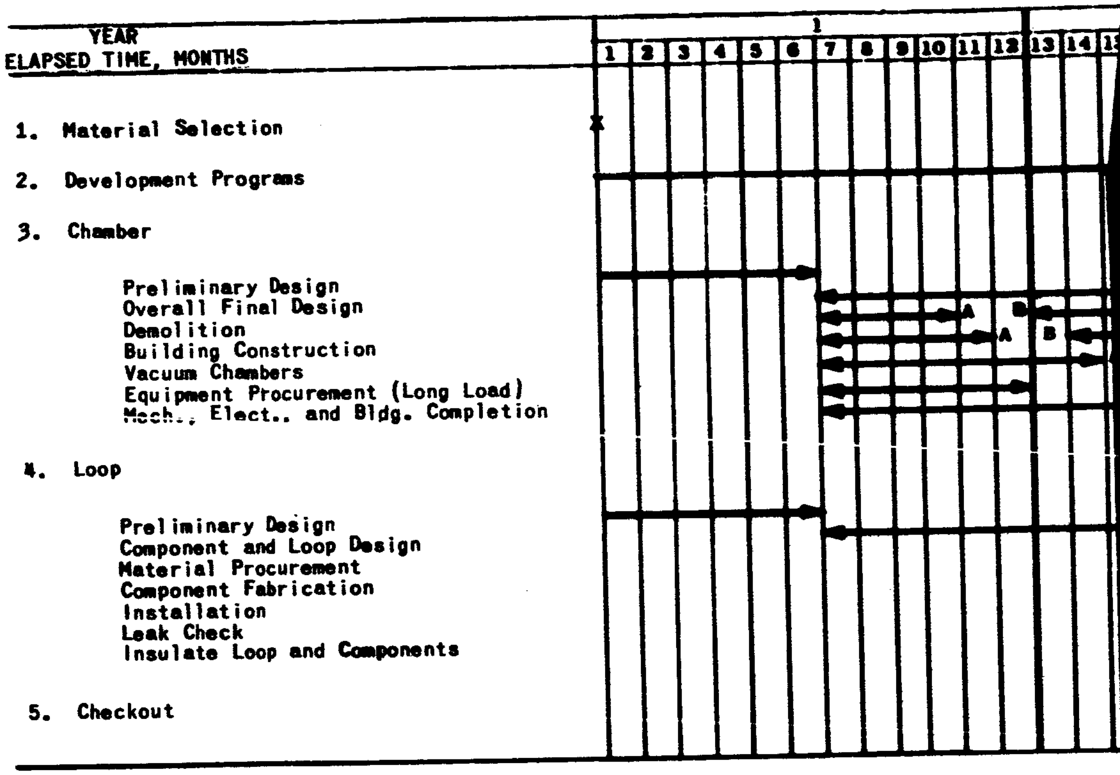
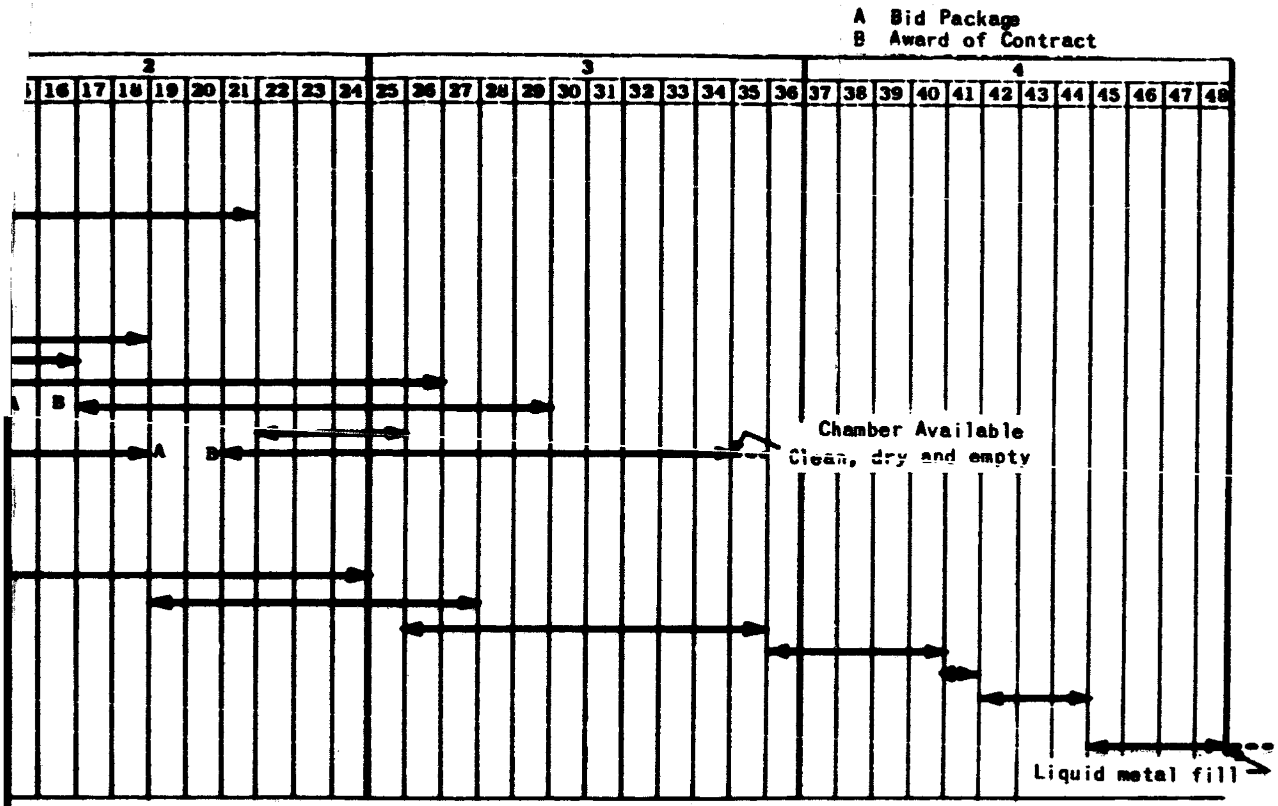


Figure 44. Test



Facility Schedule.

2

I. Basic Feasibility Programs

A. Material Outgassing

II. Problem Areas Requiring Development

A. Chamber Preliminary Design

1. Space Power Chamber Structural Analysis
2. High Current Bus Connection
3. Motor Drive System
4. Decontamination
5. Leak Detection

B. Material Handling

C. Status of Refractory Metals

1. Phase Morphology Study
2. Material Scale Up
3. Pipe Bending Study
4. Property Determination
5. Material Joining
6. Non-destructive Testing
7. Specification, Codes and Qualifications

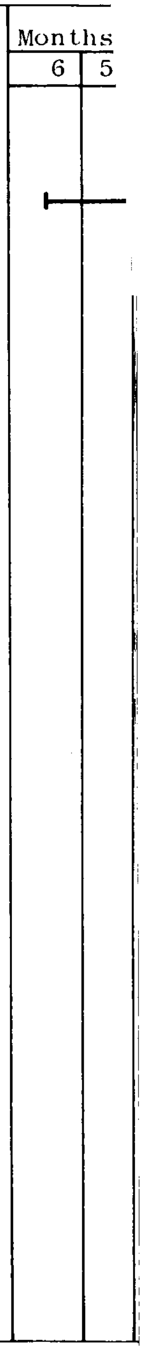
D. Valve Development

E. Loop Stress Analysis

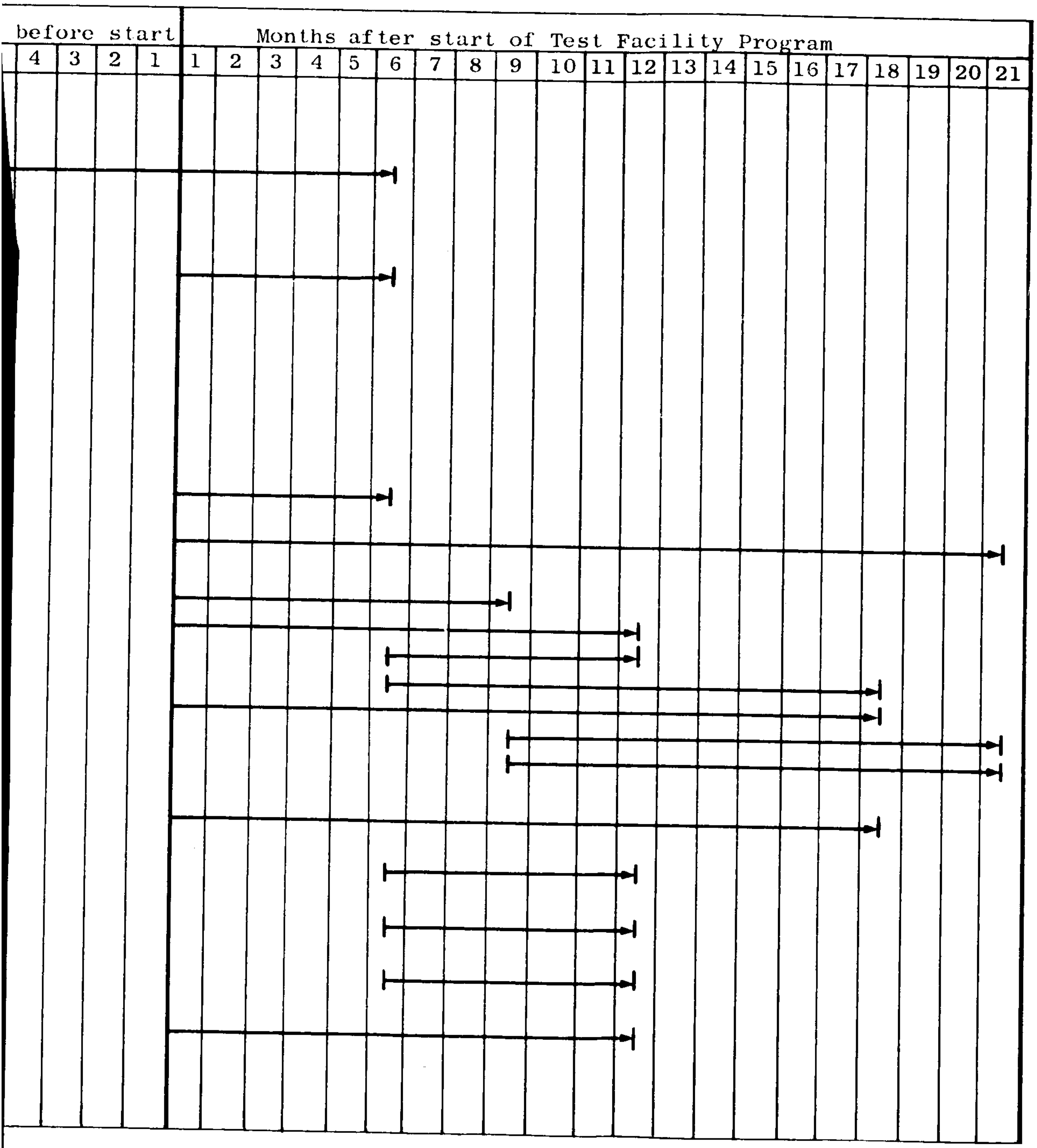
F. Pumps and Flowmeter

G. Sliding Friction

H. Diffusion Pump Backstreaming



Figur



45. Development Program Schedule.

APPENDIX

SPECIFICATIONS

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December 11, 1964
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PRELIMINARY SPECIFICATION

BOILER ASSEMBLY

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

PRELIMINARY SPECIFICATION

BOILER ASSEMBLY

1. SCOPE

1.1 Scope. This specification covers the facility boiler assembly for generation of potassium vapor to be used in testing advanced space power system components. The boiler assembly is intended for use in the 15 MW Space Power Test Facility to transport heat from the primary loop to the secondary loop under typical advanced power system operating conditions.

2. APPLICABLE DOCUMENTS

2.1 Government Documents. The following government documents of the issue in effect on the date of this specification form a part of the specification to the extent specified herein.

MIL-D-70327	Drawings, engineering and associated tests.
MIL-STD-129	Marking for shipment and storage.
MIL-STD-130	Identification marking of U.S. military property.
MIL-STD-810	Environmental test methods.
MIL-STD-143	Use of specification and standards.
MIL-STD-271C	(SHIPS)

2.2 Non-Government Documents

SPPS Spec. 03-0013-00-B (August 2, 1965)	Mass Spectrometric Leak Detection Using Helium
SPPS Spec. 01-0020-00-B (23 December 1964)	Sheet, Plate, and Strip: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy

SPPS Spec. 01-0021-00-B (23 December 1964)	Bar and Rod: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 01-0022-00-B (23 December 1964)	Seamless Tubing and Pipe: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 03-0010-00-B (26 July 1963)	Chemical Cleaning of Columbium and Columbium Alloy Products
SPPS Spec. 03-0005-00-A (24 September 1963)	Welding of Columbium-1% Zirconium Alloy by the Inert-Gas Tungsten Arc Process (Where Applicable)
SPPS Spec. 03-0012-00-A (24 January 1964)	Welding of Columbium-1% Zirconium Alloy by the Electron Beam Process (Where Applicable)
SPPS Spec. 03-0015-00-A (30 April 1964)	Arc Weld Groove Designs for Austenitic Stainless Steels, L-605, Columbium and Tantalum Alloys

ASME Boiler and Pressure Code, Section III, Rules for Construction of Nuclear Vessels (where applicable).

3. REQUIREMENTS

The boiler is required to generate dry saturated potassium vapor with up to 100°F of superheat at design outlet temperature. The design condition for full load is as follows:

Heating Fluid	Lithium (1)
Inlet temperature, °F	2200
Outlet temperature, °F	2100
Inlet pressure, psia	40
Outlet pressure, psia	25
Flow rate, lbs/sec	143
Boiling Fluid	Potassium (2)
Inlet temperature, °F	1345-2050
Outlet temperature, °F	2050
Inlet pressure, psia	225
Outlet pressure, psia	179
Flow rate, lbs/sec	17.7
Heat Load, KW	15,000
Design life, hrs.	30,000

The once-through L-tube boiler design shall be constructed as shown in Figure 8 (Illustrations). Lithium flows on the shell side and potassium will be vaporized inside the tube. An insert design based upon experimental results from NAS 3-2528 will be specified.

The boiler design will permit counter-current or parallel flow of fluids through the boiler, as may be desired by the NASA tests. Slight deviations in shell side temperatures to lower values will be permitted for the parallel flow case.

A complete mechanical analysis for structural integrity shall be prepared in accordance with the requirements of Section III of the ASME Boiler and Pressure Vessel code. The thermal performance of the boiler shall be analyzed over the heat load range from 1500 to 15,000 KW.

The material of construction for the boiler assembly will be FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy. The material to be supplied for this boiler will be in accordance with the applicable specifications (SPPS 01-0020-00-B, 01-0021-00-B, 00-0022-00-B). All welding will be in accordance with SPPS 03-0005-00-A and inspection by mass spectrometer, as described in specification SPPS 03-0013-00-B.

The operational environment for this facility will be 10^{-9} Torr within the space power chamber at the Lewis Research Center. It is imperative that heat losses be minimized to control cryogenic cooling requirements of the facility.

4. QUALITY ASSURANCE PROVISIONS

The general provisions for quality assurance contained in NASA document QA-2a and MIL-Q-9858 will be followed in design, manufacture and testing.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging

5.1.1 Cleaning - In order to meet the stringent life, performance and reliability requirements of the subsystem it is necessary that the utmost care be taken to assure that the component is free from actual or incipient contamination.

5.1.1.1 Part Degreasing - After all fabrication processes, where applicable the parts shall be degreased. The parts shall be inspected under an ultra violet lamp, 2500 to 3700 angstroms wave length, to verify that they have been degreased. Thereafter the parts shall be stored in plastic bags or other containers which are equally as clean until ready for assembly.

5.1.1.2 Temporary Parts - Temporary parts such as port closures, shipping bags, etc., shall be cleaned to the same level as the parts in the assembly.

5.1.1.3 Inspection - Prior to assembly and after fabrication processes have been completed, all parts which comprise the final assembly shall be inspected to assure freedom from burrs, feather edges, attached slivers or any other similar type particles which could subsequently become dislodged. This inspection shall also include checking the plating, coating or any other finish to assure that a satisfactory bond exists and that there is no danger of flaking, peeling or other possible surface deterioration which could constitute a contamination hazard.

5.1.1.4 Cleaning of the Assembly - The component including the shipping bag, port closures, etc., shall be thoroughly cleaned using procedures and equipment which will be clean to the level specified below. The gases and/or liquids used in this process shall have been passed, finally, through a 5 micron absolute filter. The parts shall be cleaned until the final rinse solution contains no more than the numbers of particles shown below per 100 ML of solution filtered through a standard HA millipore filter.

Particle Size - Microns	Max. Number of Particles
10-30	160
30-40	40
40-80	20
80-up	0

5.1.2 Packaging - Precautions shall be taken to assure that the component contains no residual cleaner either as a solid, liquid or gas. The component shall be double sealed in 2 polyethylene or nylon bags of a thickness of at least 5 mils and the ports shall be sealed.

REFERENCES:

(1) Properties of Inorganic Working Fluids and Coolants for Space Applications, Part II, Liquid Metals WADC TR59-598.

(2) Thermodynamic Properties of Potassium Calculated from Experimental Data in the Temperature Range of 1200 to 2700°R. General Electric Company R63FPD375 by T.A. Phillips and M.E. McCarthy.

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NASw-943
December 11, 1964
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PRELIMINARY SPECIFICATION

SIMULATOR ASSEMBLY

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER & PROPULSION SECTION

PRELIMINARY SPECIFICATION

SIMULATOR ASSEMBLY

1. SCOPE

1.1 Scope. This specification covers the facility turbine simulator assembly for power extraction from potassium vapor to be used in testing advanced space power system components. The turbine simulator is intended for use in the 15 MW Space Power Test Facility to extract power from the secondary loop to simulate a 2 MW (electric) output power turbine.

2. APPLICABLE DOCUMENTS

2.1 Government Documents. The following government documents of the issue in effect on the date of this specification form a part of the specification to the extent specified herein.

MIL-D-70327	Drawings, engineering and associated tests.
MIL-STD-129	Marking for shipment and storage.
MIL-STD-130	Identification marking of U.S. military property.
MIL-STD-810	Environmental test methods.
MIL-STD-143	Use of specification and standards.
MIL-STD-271C	(SHIPS)

2.2 Non-Government Documents

SPPS Spec. 03-0013-00-B (August 2, 1965)	Mass Spectrometric Leak Detection Using Helium.
SPPS Spec. 01-0020-00-B (23 December 1964)	Sheet, Plate, and Strip: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 01-0021-00-B (23 December 1964)	Bar and Rod: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 01-0022-00-B (23 December 1964)	Seamless Tubing and Pipe: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy

SPPS Spec. 03-0010-00-B (26 July 1963)	Chemical Cleaning of Columbium and Columbium Alloy Products
SPPS Spec. 03-0005-00-A (24 September 1963)	Welding of Columbium-1% Zirconium Alloy by the Inert-Gas Tungsten Arc Process (Where Applicable)
SPPS Spec. 03-0012-00-A (24 January 1964)	Welding of Columbium-1% Zirconium Alloy by the Electron Beam Process (Where applicable)
SPPS Spec. 03-0015-00-A (30 April 1964)	Arc Weld Groove Designs for Austenitic Stainless Steels, L-605, Columbium and Tantalum Alloys

ASME Boiler and Pressure Code, Section III, Rules for Construction of Nuclear Vessels (where applicable).

3. REQUIREMENTS

The simulator is required to extract heat from and reduce the pressure of potassium vapor from a potassium boiler. The design condition for full load is as follows:

Simulator Fluid	Potassium Vapor(1)
Inlet temperature, °F	2050
Outlet temperature, °F	1420
Inlet pressure, psia	179
Outlet pressure, psia	16.4
Flow rate, lbs/sec	17.7
Outlet quality	80%
Simulator Cooling Fluid	**NaK (78/22)(2)**
Inlet temperature, °F	1245
Outlet temperature, °F	1395
Inlet pressure, psia	75
Outlet pressure, psia	60
Flow rate, lbs/sec	71
Heat Load, KW	2350
Design life, hrs.	30,000

The turbine simulator design shall be constructed as shown in Figure 9 (Illustrations). Potassium vapor flows on the shell side and NaK flows inside the tubes. Heat is removed through NaK tubes. Pressure drop is accomplished by successive drops through orificed baffle plates.

The flow in the simulator is co-current. The NaK flow is manifolded from a 6" inlet and outlet pipe to 3 parallel flow, 2.5 OD x .1 wall, pipes within the simulator.

A complete mechanical analysis for structural integrity shall be prepared in accordance with Section III, ASME Boiler and Pressure Vessel code.

The material of construction for the simulator assembly will be FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy. The material to be supplied for this simulator will be in accordance with the applicable specifications (SPPS 01-0020-00-B, 01-0021-00-B, 00-0022-00-B). All welding will be in accordance with SPPS 03-0005-00-A and inspection by mass spectrometer, as described in specification SPPS 03-0013-00-B.

The operational environment for this facility will be 10^{-9} Torr within the space power chambers at the Lewis Research Center. It is imperative that heat losses be minimized to control cryogenic cooling requirements of the facility.

4. QUALITY ASSURANCE PROVISIONS

The general provisions for quality assurance contained in NASA document QA-2a and MIL-Q-9858 will be followed in design, manufacture and testing.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging

5.1.1 Cleaning - In order to meet the stringent life, performance and reliability requirements of the subsystem it is necessary that the utmost care be taken to assure that the component is free from actual or incipient contamination.

5.1.1.1 Part Degreasing - After all fabrication processes, where applicable the parts shall be degreased. The parts shall be inspected under an ultra violet lamp, 2500 to 3700 angstroms wave length, to verify that they have been degreased. Thereafter the parts shall be stored in plastic bags or other containers which are equally as clean until ready for assembly.

5.1.1.2 Temporary Parts - Temporary parts such as port closures, shipping bags, etc., shall be cleaned to the same level as the parts in the assembly.

5.1.1.3 Inspection - Prior to assembly and after fabrication processes have been completed, all parts which comprise the final assembly shall be inspected to assure freedom from burrs, feather edges, attached slivers or any other similar type particles which could subsequently become dislodged. This inspection shall also include checking the plating, coating or any other finish to assure that a satisfactory bond exists and that there is no danger of flaking, peeling or other possible surface deterioration which could constitute a contamination hazard.

5.1.1.4 Cleaning of the Assembly - The component including the shipping bag, port closures, etc., shall be thoroughly cleaned using procedures and equipment which will be clean to the level specified below. The gases and/or liquids used in this process shall have been passed, finally, through a 5 micron absolute filter. The parts shall be cleaned until the final rinse solution contains no more than the numbers of particles shown below per 100 ML of solution filtered through a standard HA millipore filter.

Particle Size - Microns	Max. Number of Particles
10 - 30	160
30 - 40	40
40 - 80	20
80 - up	0

5.1.2 Packaging - Precautions shall be taken to assure that the component contains no residual cleaner either as a solid, liquid or gas. The component shall be double sealed in 2 polyethylene or nylon bags of a thickness of at least 5 mils and the ports shall be sealed.

REFERENCES:

(1) Thermodynamic Properties of Potassium Calculated from Experimental Data in the Temperature Range of 1200 to 2700°R. General Electric Company R63FPD375 by T.A. Phillips and M.E. McCarthy.

(2) Liquid Metals Handbook, Sodium NaK Supplement, Atomic Energy Commission and Dept. of the Navy, Washington, D.C. (1955) C.B. Jackson, Editor.

No. S-3
NASw-943
December 11, 1964
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PRELIMINARY SPECIFICATION

CONDENSER ASSEMBLY

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

PRELIMINARY SPECIFICATION

CONDENSER ASSEMBLY

1. SCOPE

1.1 Scope. This specification covers the facility condenser assembly for the condensation of potassium vapor to be used in testing advanced space power system components. The condenser assembly is intended for use in the 15MW Space Power Test Facility to transport heat from the secondary loop to the radiator loop under typical advanced power system operating conditions.

2. APPLICABLE DOCUMENTS

2.1 Government Documents. The following government documents of the issue in effect on the date of this specification form a part of the specification to the extent specified herein.

MIL-D-70327	Drawings, engineering and associated tests.
MIL-STD-129	Marking for shipment and storage.
MIL-STD-130	Identification marking of U.S. military property.
MIL-STD-810	Environmental test methods.
MIL-STD-143	Use of specification and standards.
MIL-STD-271C	(SHIPS)

2.2 Non-Government Documents

SPPS Spec. 03-0013-00-B (August 2, 1965)	Mass Spectrometric Leak Detection Using Helium
SPPS Spec. 01-0020-00-B (23 December 1964)	Sheet, Plate, and Strip: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 01-0021-00-B (23 December 1964)	Bar and Rod: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 01-0022-00-B (23 December 1964)	Seamless Tubing and Pipe: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 03-0010-00-B (26 July 1963)	Chemical Cleaning of Columbium and Columbium Alloy Products
SPPS Spec. 03-0005-00-A (24 September 1963)	Welding of Columbium-1% Zirconium Alloy by the Inert-Gas Tungsten Arc Process (Where Applicable)
SPPS Spec. 03-0012-00-A (24 January 1964)	Welding of Columbium-1% Zirconium Alloy by the Electron Beam Process (Where Applicable)
SPPS Spec. 03-0015-00-A (30 April 1964)	Arc Weld Groove Designs for Austenitic Stainless Steels, L-605, Columbium and Tantalum Alloys

ASME Boiler and Pressure Code, Section III, Rules for Construction of Nuclear Vessels, (where applicable).

3. REQUIREMENTS

The condenser is required to condense potassium vapor from a turbine or turbine simulator and subcool the resulting liquid a minimum of 75°F. Due to size, weight, and handling considerations, two condensers will be required. The design conditions of each condenser for full load is as follows:

Condensing Fluid	Potassium Vapor (1)
Inlet temperature, °F	1420
Outlet temperature, °F	1345
Inlet pressure, psia	16.4
Outlet pressure, psia	12
Flow rate, lbs/sec	8.85
Cooling Fluid	NaK (78/22) (2)
Inlet temperature, °F	1245
Outlet temperature, °F	1395
Inlet pressure, psia	75
Outlet pressure, psia	55
Flow rate, lbs/sce	226

Heat Load, KW	7,500 each
Design Life, Hrs.	30,000

The bayonet type condenser design shall be constructed as shown in Figure 10 (Illustrations). NaK flows down and back through the tubes and potassium will be condensed on the outside of the tubes.

A complete mechanical analysis for structural integrity shall be prepared in accordance with the requirements of Section III of the ASME Boiler and Pressure Vessel code. The thermal performance of each condenser shall be analyzed over the heat load range from 750 to 7,500 KW.

The material of construction for the condenser assembly will be FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy. The material to be supplied for this condenser will be in accordance with the applicable specifications (SPPS 01-0020-00-B, 01-0021-00-B, 01-0022-00-B). All welding will be in accordance with SPPS 03-0005-00-A and inspection by mass spectrometer, as described in Specification 03-0013-00-B.

The operational environment for this facility will be 10^{-9} Torr within the space power chamber at the Lewis Research Center. It is imperative that heat losses be minimized to control cryogenic cooling requirements of the facility.

4. QUALITY ASSURANCE PROVISIONS

The general provisions for quality assurance contained in NASA document QA-2a and MIL-Q-9858 will be followed in design, manufacture and testing.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging

5.1.1 Cleaning - In order to meet the stringent life, performance and reliability requirements of the subsystem it is necessary that the utmost care be taken to assure that the component is free from actual or incipient contamination.

5.1.1.1 Part Degreasing - After all fabrication processes, where applicable the parts shall be degreased. The parts shall be inspected under and ultra violet lamp, 2500 to 3700 angstroms wave length, to verify that they have been degreased. Thereafter the parts shall be stored in plastic bags or other containers which are equally as clean until ready for assembly.

5.1.1.2 Temporary Parts - Temporary parts such as port closures, shipping bags, etc., shall be cleaned to the same level as the parts in the assembly.

5.1.1.3 Inspection - Prior to assembly and after fabrication processes have been completed, all parts which comprise the final assembly shall be inspected to assure freedom from burrs, feather edges, attached slivers or any other similar type particles which could subsequently become dislodged. This inspection shall also include checking the plating, coating or any other finish to assure that a satisfactory bond exists and that there is no danger of flaking, peeling or other possible surface deterioration which could constitute a contamination hazard.

5.1.1.4 Cleaning of the Assembly - The component including the shipping bag, port closures, etc., shall be thoroughly cleaned using procedures and equipment which will be clean to the level specified below. The gases and/or liquids used in this process shall have been passed, finally, through a 5 micron absolute filter. The parts shall be cleaned until the final rinse solution contains no more than the numbers of particles shown below per 100 ML of solution filtered through a standard HA millipore filter.

Particle Size - Microns	Max. Number of Particles
10 - 30	160
30 - 40	40
40 - 80	20
80 - up	0

5.1.2 Packaging - Precautions shall be taken to assure that the component contains no residual cleaner either as a solid, liquid or gas. The component shall be double sealed in 2 polyethylene or nylon bags of a thickness of at least 5 mils and the ports shall be sealed.

REFERENCES:

(1) Thermodynamic Properties of Potassium Calculated from Experimental Data in the Temperature Range of 1200 to 2700°R. General Electric Company R63FPD375 by T.A. Phillips and M.E. McCarthy.

(2) Liquid Metals Handbook, Sodium NaK Supplement, Atomic Energy Commission and Dept. of the Navy, Washington, D.C. (1955) C.B. Jackson, Editor.

No. S-4
NASw-943
December 11, 1964
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PRELIMINARY SPECIFICATION

REFRACTORY ALLOY VALVES

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

PRELIMINARY SPECIFICATION

REFRACTORY ALLOY VALVES

1. SCOPE

1.1 Scope. This specification covers the design and fabrication of high temperature, refractory alloy valves for use in alkali liquid metal service in a high vacuum environment.

2. APPLICABLE DOCUMENTS

2.1 Government Documents. The following government documents of the issue in effect on the date of this specification form a part of the specification to the extent specified herein.

MIL-D-70327	Drawings, engineering and associated tests.
MIL-STD-129	Marking for shipment and storage.
MIL-STD-130	Identification marking of U.S. military property.
MIL-STD-810	Environmental test methods.
MIL-STD-143	Use of specification and standards.
MIL-STD-271C	(SHIPS)

2.2 Non-Government Documents

SPPS Spec. 03-0013-00-B (August 2, 1965)	Mass Spectrometer Leak Detection Using Helium
SPPS Spec. 01-0020-00-B (23 December 1964)	Sheet, Plate, and Strip: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 01-0021-00-B (23 December 1964)	Bar and Rod: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy

SPPS Spec. 01-0022-00-B (23 December 1964)	Seamless Tubing and Pipe: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 03-0010-00-B (26 July 1963)	Chemical Cleaning of Columbium and Columbium Alloy Products
SPPS Spec. 03-0005-00-A (24 September 1963)	Welding of Columbium-1% Zirconium Alloy by the Inert-Gas Tungsten Arc Process (Where Applicable)
SPPS Spec. 03-0012-00-A (24 January 1964)	Welding of Columbium-1% Zirconium Alloy by the Electron Beam Process (Where Applicable)
SPPS Spec. 03-0015-00-A (30 April 1964)	Arc Weld Groove Designs for Austenitic Stainless Steels, L-605, Columbium and Tantalum Alloys
SPPS Spec. 03-0014-00-B (13 September 1965)	Welding of Austenitic Stainless Steels
ASTM-A 182	
ASTM-A 213	
ASTM-A 240	

3. REQUIREMENTS

3.1 All valves and actuators shall be operated in a vacuum environment of 1×10^{-9} torr.

3.1.1 All heat rejection from the valves shall be by radiation to liq. N_2 cryopanels.

3.2 All stem seals shall be the metal bellows type.

3.3 A piston operator shall be required on all valves. Valve actuation shall be accomplished by argon gas lines connected to the piston operator; argon actuation pressure is 90 psig. On-off valves shall have remote position indicators. Throttle valves shall have positioners to prevent plug drift.

3.4 The design life of the valve and actuator shall be 30,000 hrs. (See specific valve for other operating conditions.)

3.5 Valve must be drainable, under the force of gravity, in the open position. Auxiliary drain traps are not permitted.

3.6 A means of determining bellows leakage, so that a failure can be immediately observed, shall be provided.

3.6.1 Valve bellows may be backed up with argon gas to reduce bellows stress during operation.

3.7 Where specific materials are cited and specifications are provided covering these materials or their fabrication, welding, etc., these specifications shall be compiled with. Where no material or fabrication, welding, etc. specifications are provided, the vendor shall delineate what specification will cover his material, inspections, welding, and fabrication.

3.7.1 The external finish of the valve and actuator shall be highly polished (electropolish or vendor approved equal) to reduce outgassing. The vendor shall select materials considering especially outgassing in the high vacuum atmosphere.

3.8 Valve "A". (See Illustrations, Figure 7, for location.)

3.8.1 Location: surge tank primary loop.

3.8.2 Line Size: 1.25 O.D. x .1 wall tubing-butt weld ends.

3.8.3 Controlled media: argon gas saturated with lithium vapor.

3.8.4 Pressure: 70 psia max.

3.8.5 Temperature: 1600°F

3.8.6 Flow area: inlet pipe area minimum.

3.8.7 Valve type: angle.

3.8.8 Operation: on-off, 5000 cycles, fail open.

3.8.9 Shut-off flow: minimum-to be specified by vendor with 70 psia against plug and 20 psia downstream of plug.

3.8.10 Valve material: See Paragraph 4.0.

3.8.11 Recommended design stress (stead state) for columbium alloy valve body: 5000 psi.

3.9 Valve "B". (See Illustrations, Figure 7, for location.)

3.9.1 Location: dump valve primary loop.

3.9.2 Line size: 2.5 O.D. x .1 wall tubing-butt weld ends.

3.9.3 Controlled media: liquid lithium.

3.9.4 Pressure: 70 psia max.

3.9.5 Temperature: 1400°F steady state; 2150°F transient.

3.9.6 Flow area: inlet pipe area minimum.

3.9.7 Valve type: angle.

3.9.8 Operation: on-off, 10,000 cycles, fail open.

3.9.9 Shut-off flow: minimum-to be specified by vendor with 40 psia against plug and 20 psia downstream of plug.

3.9.10 Valve material: See Paragraph 4.0.

3.9.11 Recommended design stress for columbium alloy valve body: 950 psi.

3.10 Valve "C". (See Illustrations, Figure 7, for location.)

3.10.1 Location: boiler feed secondary loop.

3.10.2 Line size: 2.5 O.D. x .3 wall tubing-butt weld ends.

3.10.3 Controlled media: liquid potassium.

3.10.4 Pressure: 250 psia max.

3.10.5 Temperature: 1500°F.

3.10.6 Flow: 190 gpm max @ 10 psi P max (flow prop to valve travel).

3.10.7 Valve type: angle.

3.10.8 Operation: throttling, fail open.

3.10.9 Shut-off flow: minimum.

3.10.10 Valve material: See Paragraph 4.0.

3.10.11 Recommended design stress for columbium alloy valve body: 5000 psi.

3.11 Valve "D". (See Illustrations, Figure 7, for location.)

3.11.1 Location: pressure equalizing line secondary loop.

3.11.2 Line size: 1.25 O.D. x .1 wall tubing-butt weld ends.

3.11.3 Controlled media: argon gas saturated with potassium vapor.

3.11.4 Pressure: 70 psia max.

3.11.5 Temperature: 1600°F.

3.11.6 Flow area: inlet pipe area minimum.

3.11.7 Valve type: angle.

3.11.8 Operation: on-off, 5000 cycles, fail open.

3.11.9 Shut-off flow: minimum-to be specified by vendor with 70 psia against plug and 20 psia downstream of plug.

3.11.10 Valve material: See Paragraph 4.0.

3.11.11 Recommended design stress (steady state) for columbium alloy valve body: 5000 psi.

3.12 Valve "E". (See Illustrations, Figure 7, for location.)

3.12.1 Location: dump valve secondary loop.

3.12.2 Line size: 2.5 O.D. x .1 wall tubing-butt weld ends.

3.12.3 Controlled media: liquid potassium.

3.12.4 Pressure: 70 psia max.

3.12.5 Temperature: 1400°F steady state; 2000°F transient.

3.12.6 Flow area: inlet pipe area minimum.

3.12.7 Valve type: angle.

3.12.8 Operation: on-off, 10,000 cycles, fail open.

3.12.9 Shut-off flow: minimum-to be specified by vendor with 40 psia against plug and 20 psia downstream of plug.

3.12.10 Valve material: See Paragraph 4.0.

3.12.11 Recommended design stress for columbium alloy valve body: 1450 psi.

3.13 Valve "N". (See Illustrations, Figure 7, for location.)

3.13.1 Location: primary loop sample line.

3.13.2 Line size: 1.25 O.D. x .1 wall tubing-butt weld ends.

3.13.3 Controlled media: liquid lithium.

3.13.4 Pressure: 50 psia max.

3.13.5 Temperature: 2100°F.

3.13.6 Flow area: .196 $in.^2$ minimum.

3.13.7 Valve type: angle.

3.13.8 Operation: intermittent sampling, 5000 cycles, fail closed.

3.13.9 Shut-off flow: minimum-to be specified by vendor with 50 psia against plug and zero psia downstream of plug.

3.13.10 Valve material: See Paragraph 4.0.

3.13.11 Recommended design stress for columbium alloy valve body: 950 psi.

3.14 Valve "O". (See Illustrations, Figure 7, for location.)

3.14.1 Location: secondary loop sample line.

3.14.2 Line size: 1.25 O.D. x .1 wall tubing-butt weld ends.

3.14.3 Controlled media: liquid potassium.

3.14.4 Pressure: 50 psia max.

3.14.5 Temperature: 1500°F.

3.14.6 Flow area: .196 $in.^2$ minimum.

3.14.7 Valve type: angle.

3.14.8 Operation: intermittent sampling, 5000 cycles, fail closed.

3.14.9 Shut-off flow: minimum-to be specified by vendor with 50 psia against plug and zero psia downstream of plug.

3.14.10 Valve material: See Paragraph 4.0.

3.14.11 Recommended design stress for columbium alloy valve body: 1450 psi.

4. MATERIALS

4.1 Materials that come in contact with the process fluid, excluding plug, valve seat and stem guide materials, shall conform to the following specifications:

a) Bar stock per SPPS Spec. 01-0021-00-B.
b) Tubing per SPPS Spec. 01-0022-00-B.
c) Plate, sheet and strip per SPPS Spec. 01-0020-00-B.

4.2 Materials that are not wetted by the process fluid, excluding seals and packing materials, shall conform to the following specifications:

a) Forgings per ASTM-A213.
b) Pipe and tube per ASTM-A213.
c) Plate and sheet per ASTM-A240.

4.3 Materials used for valve seats, plugs and stem guides shall be specified by the vendor considering the following requirements:

a) Compatibility with process fluid.
b) Galling, seizing and self welding at valve temperature and in the liquid metal atmosphere.

5. FABRICATION AND WELDING REQUIREMENTS

5.1 All welding will be in accordance with Spec. 03-0005-00-A and 03-0014-00-B; all weld groove designs, per Spec. 03-0015-00-A.

6. TESTING

6.1 Valve body and bellows shall pass a helium mass spectrometer leak test per SPPS Spec. 03-0013-00-B. Maximum leak rate shall be 5.0×10^{-10} std cc/sec of air.

6.2 Valve body shall pass a hydrostatic pressure test; the method and pressure used shall be determined by the vendor subject to the buyer's approval.

6.3 Valve seat leakage tests shall be specified by the vendor subject to the buyer's approval.

6.4 Valve actuator shall pass a helium mass spectrometer leak test per SPPS Spec. 03-0013-00-B. Maximum leakage rate shall be 5.0×10^{-10} std cc/sec of air.

6.5 Valve actuator shall pass a hydrostatic pressure test, the test pressure to be twice the maximum operating pressure.

6.6 Valve bellows tests, hydrostatic and cyclic, shall be conducted. The vendor shall determine the type of test, number of cycles, pressure, etc. subject to approval by the buyer.

7.0 QUALITY ASSURANCE PROVISIONS

7.1 The general provisions for quality assurance contained in NASA document QA-2a and MIL-Q-9858 will be followed in design, manufacture and testing.

8. CLEANING AND PACKAGING

8.1 Cleaning. To meet the stringent life, performance and reliability requirements of the subsystem, utmost care must be taken to assure that the component is free from actual or incipient contamination.

8.1.1 Part Degreasing. After all fabrication processes, where applicable the parts shall be degreased. The parts shall be inspected under an ultra violet lamp, 2500 to 3700 angstroms wave length, to verify that they have been degreased. Thereafter the parts shall be stored in plastic bags or other containers, equally as clean, until ready for assembly.

8.1.2 Temporary Parts. Temporary parts, such as port closures, shipping bags, etc., shall be cleaned to the same level as the parts in the assembly.

8.1.3 Inspection. Before assembly and after fabrication processes have been completed, all parts which comprise the final assembly shall be inspected to assure freedom from burrs, feather edges, attached slivers or any other similar type particles which could subsequently become dislodged. This inspection shall also include checking the plating, coating or any other finish to assure that a satisfactory bond exists and that there is no danger of flaking, peeling or other possible surface deterioration which could constitute a contamination hazard.

8.1.4 Cleaning the Assembly. The component, including the shipping bag, port closures, etc., shall be thoroughly cleaned using procedures and equipment which will be clean to the level specified below. The gases and/or liquids used in this process shall have been passed, finally, through a 5 micron absolute filter. The parts shall be cleaned until the final rinse solution contains no more than the numbers of particles shown below per 100 ML of solution filtered through a standard HA millipore filter.

Particle Size, Microns	Max. No. Particles
10 - 30	160
30 - 40	40
40 - 80	20
80 - up	0

8.2 Packaging. Precautions shall be taken to assure that the component contains no residual cleaner either as a solid, liquid or gas. The component shall be double-sealed in two polyethylene or nylon bags, at least 5 mils thick, and the ports shall be sealed.

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December 11, 1964
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PRELIMINARY SPECIFICATION

REFRACTORY ALLOY
ALKALI METAL FLOWMETER PUMP

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

PRELIMINARY SPECIFICATION

REFRACTORY ALLOY ALKALI METAL PUMP

1. SCOPE

1.1 This specification covers the design and fabrication of high temperature, refractory alloy pumps for pumping alkali liquid metals. The pumps must operate in a high vacuum environment.

2. APPLICABLE DOCUMENTS

2.1 Government Documents. The following government documents of the issue in effect on the date of this specification form a part of the specification to the extent specified herein.

MIL-D-70327	Drawings, engineering and associated tests.
MIL-STD-129	Marking for shipment and storage.
MIL-STD-130	Identification marking of U.S. military property.
MIL-STD-810	Environmental test methods.
MIL-STD-143	Use of specification and standards.
MIL-STD-271C	(SHIPS)

2.2 Non-Government Documents

SPPS Spec. 03-0013-00-B (August 2, 1965)	Mass Spectrometric Leak Detection Using Helium
SPPS Spec. 01-0020-00-B (23 December 1964)	Sheet, Plate, and Strip: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 01-0021-00-B (23 December 1964)	Bar and Rod: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 01-0022-00-B (23 December 1964)	Seamless Tubing and Pipe: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy

SPPS Spec. 03-0010-00-B (26 July 1963)	Chemical Cleaning of Columbium and Columbium Alloy Products
SPPS Spec. 03-0005-00-A (24 September 1963)	Welding of Columbium-1% Zirconium Alloy by the Inert-Gas Tungsten Arc Process (Where Applicable)
SPPS Spec. 03-0012-00-A (24 January 1964)	Welding of Columbium-1% Zirconium Alloy by the Electron Beam Process (Where Applicable)
SPPS Spec. 03-0015-00-A (30 April 1964)	Arc Weld Groove Designs for Austenitic Stainless Steels, L-605, Columbium and Tantalum Alloys

ASME Boiler and Pressure Code, Section III, Rules for Construction of Nuclear Vessels (where applicable).

3. REQUIREMENTS

Two pumps will be required - a primary loop and a secondary loop pump. The design conditions for each pump for full load is as follows:

3.1 Primary Loop

a) Pumped Fluid	Lithium
b) Fluid Temperature	2100°F
c) Fluid Flow (See flow map, Figure 1.)	2600 GPM Max.
d) Inlet Pressure	20 PSIA Min.
e) Outlet Pressure	70 PSIA
f) Pump Material	See Paragraph 3.3.1
g) Pipe Size	6.25 OD x .225 W
h) Design Stress	950 PSI
i) Design Life	30,000 Hrs. Min. at Temperature
j) Pump Environment	1 x 10^{-9} Torr Vacuum
k) Cooling Available	Liq. N_2 Cryopanels Radiation Only
l) Power Available	220 or 440V, 3Ø, 60 Cycle

3.2 Secondary Loop

a) Pumped Fluid	Potassium
b) Fluid Temperature	1500°F Max.
c) Fluid Flow (See Flow Map)	190.5 GPM
d) Inlet Pressure	10 PSIA Min.
e) Outlet Pressure	250 PSIA
f) Pump Material	See Paragraph 3.3.1
g) Pipe Size	2.5 OD x .3 W Outlet 2.5 OD x .1 W Inlet
h) Design Stress	5000 PSI
i) Design Life	30,000 Hrs. Min.
j) Pump Environment	1 x 10^{-9} Torr Vacuum
k) Cooling Available	Liq. N_2 Cryopanels Radiation Only
l) Power Available	220 or 440V, 3Ø, 60 Cycle

3.3 General

3.3.1 Materials that come in contact with the process fluid shall be in accordance with the following specifications:

a) Bar stock per SPPS Spec. 01-0021-00-B.
b) Tubing per SPPS Spec. 01-0022-00-B.
c) Plate, sheet and strip per SPPS Spec. 01-0020-00-B.

3.3.2 Welding of columbium alloys to be per SPPS Spec. 03-0005-00-A; weld groove designs, per SPPS Spec. 03-0015-00-A.

3.3.3 The external finish of the pump shall be highly polished (electro-polish or vendor approved equal) to reduce outgassing. The vendor shall pay particular attention to his choice of materials with regard to outgassing in the high vacuum atmosphere.

3.3.4 Vendor to specify:

a) Maximum pump envelope, port locations and pump weight.
b) Power required.
c) Pump control required.
d) Auxiliary cooling required (argon gas is available at 90 psig).
e) List of all materials.
f) Number and size of vacuum chamber penetrations.
g) Descriptive literature.
h) Problem areas.

3.3.4.1 A complete mechanical analysis for structural integrity shall be prepared by the vendor and submitted to the buyer for approval.

4.0 TESTING

4.1 All areas of the pump containing the pumped fluid shall be subject to and must pass a helium mass spectrometer leak test per SPPS Spec. 03-0013-00-B. Maximum leak rate to be 5.0×10^{-10} std. cc/sec of air.

4.2 All areas of the pump containing the pumped fluid shall be subject to and pass a hydrostatic pressure test. The method and pressure which shall be used shall be determined by the vendor subject to the buyer's approval.

4.3 All cooling passages and ducts shall be subject to and must pass a helium mass spectrometer leak test per SPPS Spec. 03-0013-00-B. Maximum leak rate to be 5.0×10^{-10} std. cc/sec of air.

4.4 All cooling passages and ducts shall be subject to and pass a hydrostatic pressure test. The method and pressure which shall be used shall be determined by the vendor subject to the buyer's approval.

5.0 QUALITY ASSURANCE PROVISIONS

The general provisions for quality assurance contained in NASA document QA-2a and MIL-Q-9858 will be followed in design, manufacture and testing.

6.0 PREPARATION FOR DELIVERY

6.1 Preservation and Packaging

6.1.1 Cleaning - In order to meet the stringent life, performance and reliability requirements of the subsystem it is necessary that the utmost care be taken to assure that the component is free from actual or incipient contamination.

6.1.1.1 Part Degreasing - After all fabrication processes, where applicable the parts shall be degreased. The parts shall be inspected under an ultra violet lamp, 2500 to 3700 angstroms wave length, to verify that they have been degreased. Thereafter the parts shall be stored in plastic bags or other containers which are equally as clean until ready for assembly.

6.1.1.2 Temporary Parts - Temporary parts such as port closures, shipping bags, etc., shall be cleaned to the same level as the parts in the assembly.

6.1.1.3 Inspection - Prior to assembly and after fabrication processes have been completed, all parts which comprise the final assembly shall be inspected to assure freedom from burrs, feather edges, attached slivers or any other similar type particles which could subsequently become dislodged. This inspection shall also include checking the plating, coating or any other finish to assure that a satisfactory bond exists and that there is no danger of flaking, peeling or other possible surface deterioration which could constitute a contamination hazard.

6.1.1.4 Cleaning of the Assembly - The component, including the shipping bag, port closures, etc., shall be thoroughly cleaned using procedures and equipment which will be clean to the level specified below. The gases and/or liquids used in this process shall have been passed, finally, through a 5 micron absolute filter. The parts shall be cleaned until the final rinse solution contains no more than the numbers of particles shown below per 100 ML of solution filtered through a standard HA millipore filter.

Particle Size - Microns	Max. Number of Particles
10 - 30	160
30 - 40	40
40 - 80	20
80 - up	0

6.1.2 Packaging - Precautions shall be taken to assure that the component contains no residual cleaner either as a solid, liquid or gas. The component shall be double sealed in 2 polyethylene or nylon bags of a thickness of at least 5 mils and the ports shall be sealed.

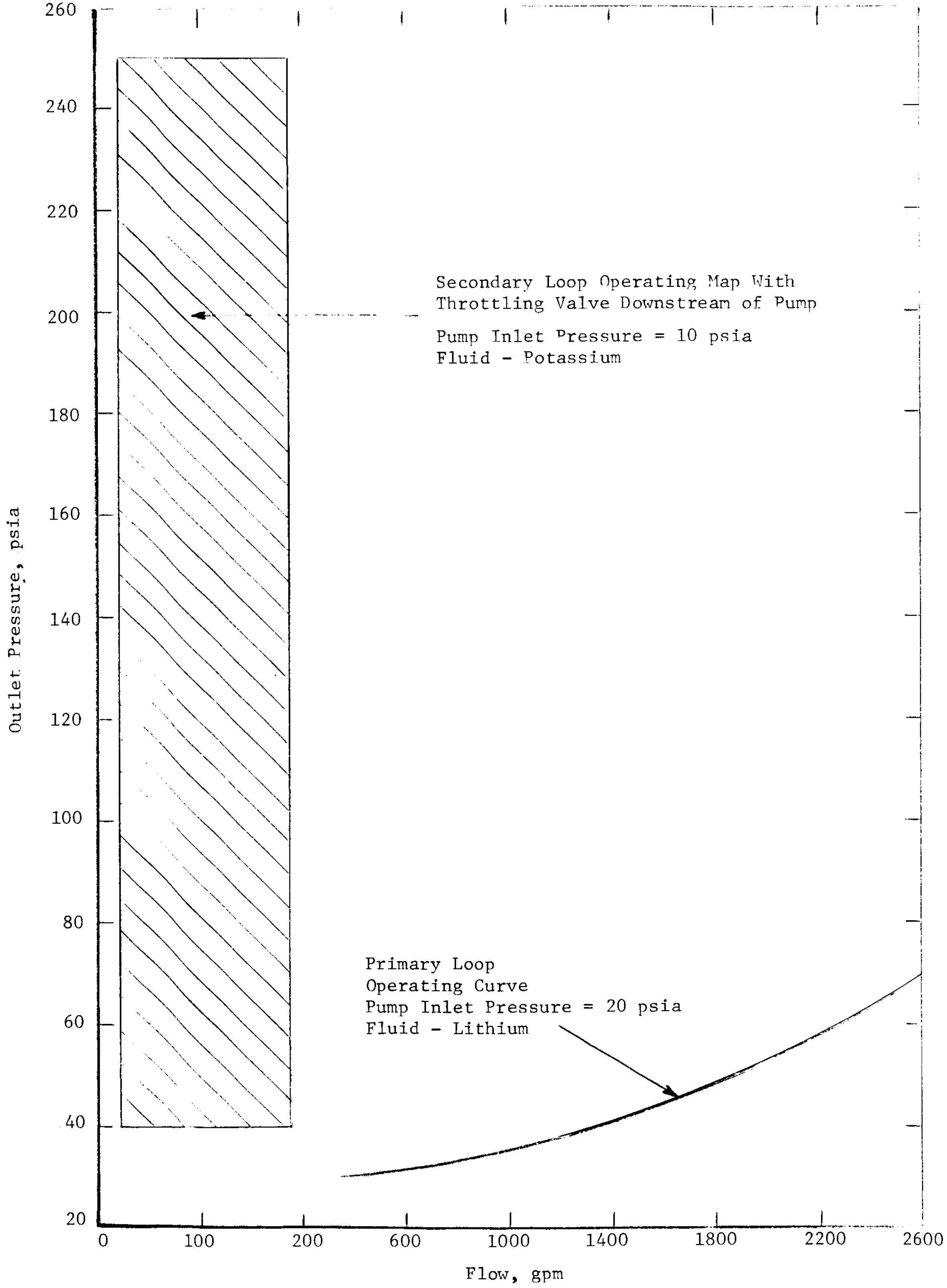

Figure 1. Primary and Secondary Loop Operating Conditions 15 M.W. Study.

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December 11, 1964
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PRELIMINARY SPECIFICATION

REFRACTORY ALLOY ALKALI METAL FLOWMETER

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

PRELIMINARY SPECIFICATION

REFRACTORY ALLOY ALKALI METAL FLOWMETER

1. SCOPE

1.1 This specification covers the design and fabrication of high temperature refractory alloy flowmeters for measuring the flow of alkali metals. The flowmeters will operate in a high vacuum environment.

2. APPLICABLE DOCUMENTS

2.1 Goverment Documents. The following government documents of the issue in effect on the date of this specification form a part of the specification to the extent specified herein.

MIL-D-70327	Drawings, engineering and associated tests.
MIL-STD-129	Marking for shipment and storage.
MIL-STD-130	Identification marking of U.S. military property.
MIL-STD-810	Environmental test methods.
MIL-STD-143	Use of specification and standards.
MIL-STD-271C	(SHIPS)

2.2 Non-Government Documents

SPPS Spec. 03-0013-00-B (August 2, 1965)	Mass Spectrometric Leak Detection Using Helium
SPPS Spec. 01-0020-00-B (23 December 1965)	Sheet, Plate, and Strip: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 01-0021-00-B (23 December 1965)	Bar and Rod: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy

SPPS Spec. 01-0022-00-B (23 December 1964)	Seamless Tubing and Pipe: FS-85 (Cb-28Ta-10.5W-0.9Zr) Alloy
SPPS Spec. 03-0010-00-B (26 July 1963)	Chemical Cleaning of Columbium and Columbium Alloy Products
SPPS Spec. 03-0005-00-A (24 September 1963)	Welding of Columbium-1% Zirconium Alloy by the Inert-Gas Tungsten Arc Process (Where Applicable)
SPPS Spec. 03-0012-00-A (24 January 1964)	Welding of Columbium-1% Zirconium Alloy by the Electron Beam Process (Where Applicable)
SPPS Spec. 03-0015-00-A (30 April 1964)	Arc Weld Groove Designs for Austenitic Stainless Steels, L-605, Columbium and Tantalum Alloys

ASME Boiler and Pressure Code, Section III, Rules for Construction of Nuclear Vessels (where applicable).

3. REQUIREMENTS

Two flowmeters will be required - a primary loop and a secondary loop flowmeter. The design conditions for each flowmeter for full load is as follows:

3.1 Primary Loop

Flow Range	260-2600 GPM
Temperature	2100°F
Pipe Size	6.25" OD x .225 Wall
Pipe Material	See Paragraph 3.3.2.
Fluid	Lithium
Fluid Velocity	3.2 to 32 F.P.S.
Flowmeter Environment	1×10^{-9} Torr Vacuum
Accuracy (Absolute)	± 5% of Reading
Repeatability	± 2% of Reading
Cooling Available	Liq. N_2 Cryopanels Radiation Only
Design Stress for Columbium Alloy	950 psi
Design Pressure	70 psia

3.2 Secondary Loop

Flow Range	19-190 GPM
Temperature	1400°F
Pipe Size	2.5" OD x .3 Wall
Pipe Material	See Paragraph 3.3.2.
Fluid	Potassium
Fluid Velocity	1.9 to 19 F.P.S.

3.2 Secondary Loop (Cont.)

Flowmeter Environment	1×10^{-9} Torr Vacuum
Accuracy (Absolute)	± 3% of Reading
Repeatability	± 1% of Reading
Cooling Available	Liq. N_2 Cryopanels, Radiation Only
Design Stress for Columbium Alloy	5000 psi
Design Pressure	70 psia

3.3 General

3.3.1 The design life on all flowmeters shall be a minimum of 30,000 hours at temperature.

3.3.2 Materials that come in contact with the process fluid shall be in accordance with the following specifications:

a) Bar stock per SPPS Spec. 01-0021-00-B
b) Tubing per SPPS Spec. 01-0022-00-B
c) Plate, sheet and strip per SPPS Spec. 01-0020-00-B

3.3.3 Welding of columbium alloys to be per SPPS Spec. 03-0005-00-A; weld groove design, per SPPS Spec. 03-0015-00-A.

3.3.4 The external finish of the flowmeter shall be highly polished (electropolish or vendor approved equal) to reduce outgassing. The vendor shall select materials considering especially the outgassing in the high vacuum atmosphere.

3.3.5 Vendor to specify:

a) Type flowmeter.
b) Maximum envelope, port locations and flowmeter weight.
c) Power required.
d) Type signal and readout.
e) Auxiliary cooling required (argon gas is available at 90 psig).
f) List of all materials.
g) Number of vacuum chamber penetrations.
h) Calibration curves at operating temperature and flow correction curves for lower temperatures if applicable.
i) Descriptive literature.

3.3.5.1 A complete mechanical analysis for structural integrity shall be prepared by the vendor and submitted to the buyer for approval.

4.0 TESTING

4.1 All areas of the flowmeter containing the metered fluid shall pass a helium mass spectrometer leak test per SPPS Spec. 03-0013-00-B, Maximum leakage rate to be 5.0×10^{-10} std. cc/sec of air.

4.2 All areas of the flowmeter containing the metered fluid shall pass a hydrostatic pressure test. The method and pressure used shall be determined by the vendor subject to the buyer's approval.

4.3 All cooling passages and ducts shall pass a helium mass spectrometer leak test per SPPS Spec. 03-0013-00-B. Maximum leakage rate to be 5.0×10^{-10} std. cc/sec of air.

4.4 All cooling passages and ducts shall pass a hydrostatic pressure test. The method and pressure used shall be determined by the vendor subject to the buyer's approval.

5.0 QUALITY ASSURANCE PROVISIONS

The general provisions for quality assurance contained in NASA document QA-2a and MIL-Q-9858 will be followed in design, manufacture and testing.

6.0 PREPARATION FOR DELIVERY

6.1 Preservation and Packaging

6.1.1 Cleaning - In order to meet the stringent life, performance and reliability requirements of the subsystem it is necessary that the utmost care be taken to assure that the component is free from actual or incipient contamination.

6.1.1.1 Part Degreasing - After all fabrication processes, where applicable the parts shall be degreased. The parts shall be inspected under an ultra violet lamp, 2500 to 3700 angstroms wave length, to verify that they have been degreased. Thereafter the parts shall be stored in plastic bags or other containers which are equally as clean until ready for assembly.

6.1.1.2 Temporary Parts - Temporary parts, such as port closures, shipping bags, etc., shall be cleaned to the same level as the parts in the assembly.

6.1.1.3 Inspection - Prior to assembly and after fabrication processes have been completed, all parts which comprise the final assembly shall be inspected to assure freedom from burrs, feather edges, attached slivers or any other similar type particles which could subsequently become dislodged. This inspection shall also include checking the plating, coating or any other finish to assure that a satisfactory bond exists and that there is no danger of flaking, peeling or other possible surface deterioration which could constitute a contamination hazards.

6.1.1.4 Cleaning of the Assembly - The component, including the shipping bag, port closures, etc., shall be thoroughly cleaned using procedures and equipment which will be clean to the level specified below. The gases and/or liquids used in this process shall have been passed, finally, through a 5 micron absolute filter. The parts shall be cleaned until the final rinse solution contains no more than the numbers of particles shown below per 100 ML of solution filtered through a standard HA millipore filter.

Particle Size - Microns	Max. No. Particles
10 - 30	160
30 - 40	40
40 - 80	20
80 - up	0

6.1.2 Packaging - Precautions shall be taken to assure that the component contains no residual cleaner either as a solid, liquid or gas. The component shall be double sealed in 2 polyethylene or nylon bags of a thickness of at least 5 mils and the ports shall be sealed.

01-0020-00-B
SPPS-28-R1
23 December 1964
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SPECIFICATION

SHEET, PLATE, AND STRIP: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

SHEET, PLATE, AND STRIP: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in sheet, plate, and strip form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T (26 December 1957)	Method of Tension Testing of Metallic Materials
ASTM Designation E29-58T (1958)	Recommended Practices for Designating Significant Places in Specified Limiting Values
ASTM Designation (Pending)	Methods for Chemical Analysis of Reactor and Commercial Columbium
ASTM E112-61 (1961)	Estimating Average Grain Size of Metals
AMS 2242A (1 December 1950)	Tolerances, Corrosion and Heat Resistant Sheet, Strip and Plate
AMS 2635 (15 August 1958)	Radiographic Inspection
AMS 2645 (1 March 1955)	Fluorescent Penetrant Inspection
AMS 2646 (1 March 1955)	Contrast Dye Penetrant Inspection

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal, shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I), except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

Element	Check Analysis Limits, Max., ppm	Permissible Variations in Check Analysis, ppm
Carbon	100	+ 10
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I

CHEMICAL COMPOSITION

FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

Element	Minimum Content ppm	Maximum Content ppm
Carbon	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26 $^{w}/_{o}$	29 $^{w}/_{o}$
Tungsten	10 $^{w}/_{o}$	12 $^{w}/_{o}$
Zirconium	0.6 $^{w}/_{o}$	1.1 $^{w}/_{o}$
Columbium	Remainder	-

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

Product Thickness, Inches	Minimum Allowable ASTM Grain Size No.	Allowable Spread in ASTM Grain Size Nos. in Any One Item	% R_x Minimum
0.010 to 0.060	6	2	100
0.060 to 0.125	4	2	100
0.125 to 0.187	4	2	100
0.187 to 0.500	3	3	95
0.500 to 1.0	3	3	95
Greater than 1.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F):

Ultimate Tensile Strength, ksi		0.2% Yield Strength, ksi		Elong., % in 2 Inches
Minimum	Maximum	Minimum	Maximum	Minimum
70	100	50	80	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

Test Temp., °F	Stress, ksi	Minimum Life Hours
2200	16	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--

less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.7.3. Bend Ductility. Representative samples of the materials in final form shall withstand the following bend test at room temperature without failure when tested according to procedures described in the most recent revision of the Materials Advisory Board report MAB-176M, "Evaluation Test Methods for Refractory Metal Sheet Materials." The samples shall be sectioned with the long axis of the bend specimens perpendicular to the final rolling direction.

3.7.3.1. Sheet 0.060 inch in thickness and under shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute and subsequently flattened for a total bend of 180°.

3.7.3.2. Sheet over 0.060 inch to 0.187 inch in thickness shall be bent over a 1T radius through 105° at a ram speed of 1 inch per minute.

3.8. Tolerances

3.8.1. Plate

3.8.1.1. Definition. Plate includes material 6 inches wide or over and 0.187 inch or more in thickness.

3.8.1.2. Dimensions. Plate dimensions shall conform to the following tolerances:

Thickness	Width	Length
± 0.025 inch or ± 5% whichever is less	± 0.125 inch	± 0.125 inch

3.8.1.3. Flatness. Flatness tolerance on plate shall conform to AMS 2242A, "Tolerances, Corrosion and Heat Resistant Sheet, Strip and Plate."

3.8.2. Sheet

3.8.2.1. Definition. Sheet includes material 6 inches wide or over and up to 0.187 inch in thickness.

3.8.2.2. Dimensions. Sheet dimensions shall conform to those presented in Table II.

3.8.2.3. Flatness. See paragraph 3.8.3.3.

3.8.3. Strip

3.8.3.1. Definition. Strip includes material less than 6 inches wide and up to 0.187 inch in thickness.

3.8.3.2. Dimensions. Strip dimensions shall conform to those presented in Table II.

3.8.3.3. Flatness. Total deviation from flatness of sheet and strip shall not exceed 6% as determined by the formula:

$$\frac{H}{L} \times 100 = \%\ \text{Flatness Deviation}$$

where

H = maximum distance from a flat reference surface

and

L = minimum distance from this point to the point of contact with the reference surface.

The actual values shall be reported. In determining flatness, the sheet shall not be subject to external pressure at any point but shall lie freely on a flat surface during measurement. Oilcanning will be reported. An estimate of the extent (area, height, etc.,) of these defects shall be made.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

TABLE II

DIMENSIONAL TOLERANCES FOR SHEET AND STRIP

Material Thickness, Inch	Width, Inches	Thickness Tolerances, Inch
0.010-0.019	to 24	± 0.001
0.020-0.039	to 24	± 0.0015
0.040-0.059	to 24	± 0.002
0.060-0.089	to 24	± 0.003
0.090-0.129	to 24	± 0.004
0.130-0.159	to 24	± 0.005
0.160-0.187	to 24	± 0.010

Material Thickness, Inch	Width Tolerances, Inch
0.010-0.059	+ 0.031, -0
0.060-0.125	+ 0.046, -0
0.126-0.187	+ 0.125, -0

Material Thickness, Inch	Length Tolerances, Inch
0.010-0.059	+ 0.046, -0
0.060-0.125	+ 0.062, -0
0.126-0.187	+ 0.125, -0

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness, whichever is smaller, shall be a minimum of 0.50 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken transverse to the final working direction from plate and sheet and from strip if of sufficient width. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analyses of Reactor and Commerical Columbium."

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

> Specimens shall be tested in a vacuum of 1 x 10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.
>
> Specimens shall be held for a half hour at the test temperature before application of load.
>
> Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Bend Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse shear.

Transducers for the shear wave inspection shall be focused, preferably cylindrically, to a beam no more than 0.125-inch wide in its smaller dimension (where it enters the material being inspected). Cylindrically-focused transducers shall not exceed 2 inches in length. The focal distance shall be adjusted when the transducer is beamed perpendicular to the surface of the calibration piece; then this focal distance shall be maintained throughout the actual inspection. After the focal distance is established, an appropriate shear wave angle shall be set and the calibration notch indication shall be set at 80% on the indication where the sound beam traverses one or two thicknesses of the sheet (depending on whether the notch is on the far side or incident side of the sheet). Calibration gain settings shall be recorded when the calibration defect is on both the incident and the far side of the sheet. If there is any difference in the indication, that gain setting giving an 80% indication from the side which produces the smaller indication shall be used for inspection. Calibration shall be done before and after the ultrasonic inspection, or at the beginning and end of each work shift. If the magnitude of indication from the calibration notch differs 10% or more from the previous calibration, all material inspected since then shall be reinspected.

5.7.1.2.2. Calibration of Plate. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes. The depth of the notches shall be 0.005 inch, the width shall be 0.005 inch and the length greater than the ultrasonic beam width. The notches shall be placed on the surface of the calibration piece perpendicular to the direction of the intended shear wave inspection, i.e., transverse and longitudinal and at least 1 inch from the edge of the plate. In addition, a 0.020-inch diameter hole shall be made in the calibration piece parallel to the surface to a depth of at least 0.750 inch at a point one-half the thickness of the plate. If the thickness of the plate exceeds 0.750 inch, similar holes shall also be made at points one-quarter and three-quarters of the plate thickness. Calibration settings to achieve 80% amplitude of the notches and holes, along with the magnitude of the other applicable calibration defects, shall be recorded. For example, on plate using a shear wave, the notch on the near surface should be set at 80% and the amplitude recorded for the indications from the hole and notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave inspection, only the 0.020-inch diameter holes shall be used for calibration.

5.7.1.2.3. Calibration of Sheet and Strip. The sheet shall be inspected by a shear wave beam pointed in both longitudinal and transverse directions. Calibration shall be done on notches cut perpendicular to the direction of the beam in pieces of sheet of the same material and thickness as that to be inspected. If that portion is later trimmed and scrapped, the calibration notches may be made on a section of the actual sheet. The depth of the calibration notches shall be 3% of the sheet thickness; width, no greater than the depth; length, no more than 1 inch. All notches shall be at least 1 inch from the edge of the sheet. Duplicate notches may be made on the opposite face of the sheet in locations where the sound beam will not intersect both notches in a single traverse, or the sheet may be turned over during calibration to determine the relative response from the calibration notch on both the incident and far side of the sheet.

5.7.1.2.4. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notch.

5.7.1.2.5. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.6. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITION

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

Test	Rounded-Off Unit for Observed or Calculated Value
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

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23 December 1964
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SPECIFICATION

BAR AND ROD: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

BAR AND ROD: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in bar and rod form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T (26 December 1957)	Method of Tension Testing of Metallic Materials
ASTM Designation E29-58T (1958)	Recommended Practices for Designating Significant Places in Specified Limiting Values
ASTM Designation (Pending)	Methods for Chemical Analysis of Reactor and Commercial Columbium
ASTM E112-61 (1961)	Estimating Average Grain Size of Metals
AMS 2635 (15 August 1958)	Radiographic Inspection
AMS 2645 (1 March 1955)	Fluorescent Penetrant Inspection
AMS 2646 (1 March 1955)	Contrast Dye Penetrant Inspection

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The amount of total reduction from the turned ingot to the finished product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the previous recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product shall be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6. All annealing shall be carried out in a vacuum less than 1 x 10^{-5} torr.

3.4.2. Heat Treatment. All mill products to be annealed shall be thoroughly degreased, chemically cleaned, and protected from furnace parts by a layer of fresh tantalum, columbium, or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1 x 10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or columbium-1% zirconium alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium, or columbium-1% zirconium alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross-sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

Element	Check Analysis Limits, Max., ppm	Permissible Variations in Check Analysis, ppm
Carbon	100	+ 10
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

TABLE I

CHEMICAL COMPOSITION

FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

Element	Minimum Content ppm	Maximum Content ppm
Carbon	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26 w/o	29 w/o
Tungsten	10 w/o	12 w/o
Zirconium	0.6 w/o	1.1 w/o
Columbium	Remainder	-

Product Diameter or Thickness, Inches	Minimum Allowable ASTM Grain Size No.	Allowable Spread in ASTM Grain Size Nos. in Any One Item	% R_x Minimum
0.125 to 0.250	4	2	100
0.250 to 0.500	4	2	100
0.500 to 1.0	4	2	100
1.0 to 2.0	4	2	95
Greater than 2.0	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

Ultimate Tensile Strength, ksi		0.2% Yield Strength, ksi		Elong., % in 4D
Minimum	Maximum	Minimum	Maximum	Minimum
70	100	50	80	20

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3).

Test Temp., °F	Stress, ksi	Minimum Life Hours
2200	16	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon	±	10 ppm
Oxygen	±	50 ppm
Nitrogen	±	50 ppm
Hydrogen	±	2 ppm

3.8. Tolerances

3.8.1. Rolled, Swaged, or Drawn Rounds

3.8.1.1. Definition. Rod - 3.5 inches in diameter or less.

3.8.1.2. Diameter. The permissible variation in diameter and the limits of out-of-roundness of descaled rounds shall not exceed those in Table II (refer to page 8).

3.8.1.3. Cut Lengths. Maximum length variation shall be 0.25-inch.

3.8.1.4. Straightness. Maximum deviation shall be 0.050 inch per foot in any length.

3.8.2. Square or Rectangular Bar

3.8.2.1. Definition. Bar - any straight product with a rectangular cross-section 0.187 inch or more thick and less than 5 inches wide.

3.8.2.2. Dimensions. Unless otherwise specified, forged or rolled square and rectangular shapes shall have the following tolerances:

Thickness	Length	Width
± 0.025 inch or ± 5%, whichever is less	± 0.125 inch	± 0.125 inch

3.8.2.3. Straightness of Bar. Maximum deviation shall be 0.050 inch per foot in any length.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

TABLE II

PERMISSIBLE DIMENSIONAL VARIATIONS FOR ROUND BAR

Diameter Inches	Diameter Variation Inch	Out-of-Roundness Inch
0.125 to 0.281	+ 0.002, -0.002	0.004
Over 0.281 to 0.406	∓ 0.010, -0.005	0.008
Over 0.406 to 0.625	+ 0.010, -0.005	0.012
Over 0.625 to 0.875	+ 0.015, -0.005	0.015
Over 0.875 to 1.000	+ 0.020, -0.005	0.015
Over 1.000 to 1.375	+ 0.020, -0.010	0.018
Over 1.375 to 1.500	+ 0.020, -0.015	0.020
Over 1.500 to 1.625	+ 0.025, -0.015	0.020
Over 1.625 to 2.000	+ 0.030, -0.030	0.025
Over 2.000 to 2.500	+ 0.032, -0.032	0.025
Over 2.500 to 3.250	+ 0.032, -0.032	0.027
Over 3.250 to 3.500	+ 0.045, -0.045	0.040
Centerless Ground Rounds		
0.0625 to 2.0	+ 0.002, -0.002	
Over 2.0	+ 0.003, -0.002	

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4.2. Porosity and Inclusions. Indications of internal porosity and non-metallic inclusions greater than 0.020 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Those indications in the range 0.010 inch to 0.020 inch or 2% of the thickness or diameter, whichever is smaller, shall be a minimum of 0.500 inch apart; those indications less than 0.010 inch shall be a minimum of 0.12 inch apart.

4.3. Surface Rework. All surface pores, gouges, and other defects deeper than 0.005 inch or 3% of the thickness, whichever is smaller, shall be unacceptable. Surface imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., samples may be taken transverse to the final working direction from bar of sufficient width or from bar greater than 2 inches in diameter. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The location of test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.05 inch, plus or minus 0.02 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

> Specimens shall be tested in a vacuum of 1 x 10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.
>
> Specimens shall be held for a half hour at the test temperature before application of load.
>
> Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. Whenever specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the technique described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so the exact position of each radiograph can be correlated with the specific area on the particular product.

5.7.1.2. Ultrasonic. All material 0.125-inch diameter and larger shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. The finished products shall be ultrasonically inspected by the immersed technique at 5 mc or above. Transducers shall be no larger than 0.75-inch diameter. Surface finishes shall be no rougher than 125 rms. Inspection shall be by longitudinal wave and by shear wave in two perpendicular directions, i.e., longitudinal and transverse and shall be with focused transducers appropriate to the diameter being inspected (360 degree transducers are allowable where appropriate). Automatic equipment which traverses a spiral path is satisfactory; but three traverses shall be made, one with the transducer in the circumferential shear position, one with the transducer in the axial shear position, and one with the transducer in the longitudinal wave position, unless otherwise specified.

5.7.1.2.2. Calibration of Bar and Rod. Calibration shall be on notches and holes in a segment of the material reserved solely for calibration purposes or in a calibration specimen of similar nature and shape. The depth of the notches shall be 3% of the bar thickness, 1.5% of the rod diameter, or 0.005 inch, whichever is smaller; the width, no greater than depth; the length, greater than beam width. The notches shall be placed perpendicular to the direction of the shear wave beam and perpendicular to the surface, e.g., axial and circumferential notches on bar. In addition to the notches, a 0.020-inch diameter hole shall be made at least 0.5-inch deep in the calibration piece parallel to the surface at a distance from the surface of 1/2 the thickness or diameter or, if the thickness exceeds 0.750 inch, 1/4, 1/2 and 3/4 the thickness. Calibration settings to achieve 80% amplitude of these notches or holes along with the magnitude of the other applicable calibration defects shall be recorded. For example, on bar with shear wave, the notch on the near surface should be set at 80% and the amplitudes recorded for the indications from the hole and the notch on the far surface. Gain settings should be recorded to achieve 80% as above and 80% on each of the other applicable calibration defects. For longitudinal wave, only the 0.020-inch diameter holes, with additional holes at 1/4 and 1/2 the thickness if the thickness exceeds 0.750-inch shall be used for calibration.

5.7.1.2.3. Evaluation. Evaluation during inspection shall be made against the appropriate calibration defect. For example, on bar with shear wave, the defects on or near the far surface shall be compared to the calibration from the far surface notch; defects near the center shall be compared to the calibration from the hole at the appropriate depth; defects on the near surface shall be compared to the calibration from the near surface notches.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings, and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.2.5. Rejection. The above procedure shall be followed, and indications of defects which exceed the magnitude obtained from the appropriate calibrated notch in the sample shall be cause for rejection, unless otherwise agreed by the purchaser and vendor.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

Test	Rounded-Off Unit for Observed or Calculated Value
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

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23 December 1964
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SPECIFICATION

SEAMLESS TUBING AND PIPE: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

SEAMLESS TUBING AND PIPE: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in tube and pipe form intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8-57T (26 December 1957)	Method of Tension Testing of Metallic Materials
ASTM Designation E29-58T (1958)	Recommended Practices for Designating Significant Places in Specified Limiting Values
ASTM Designation (Pending)	Methods for Chemical Analysis of Reactor and Commercial Columbium
ASTM Designation E112-61 (1961)	Estimating Average Grain Size of Metals
AMS 2635 (15 August 1958)	Radiographic Inspection
AMS 2645 (1 March 1955)	Fluorescent Penetrant Inspection
AMS 2646 (1 March 1955)	Contrast Dye Penetrant Inspection

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing and drawing equipment normally found in ferrous and non-ferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6 and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%. The amount of final reduction for each mill product, imparted just prior to the final vacuum heat treatment and the total reduction since the last recrystallization anneal shall be reported in the certificate of compliance.

3.4. Condition

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross-sectional area to the grain size range specified in paragraph 3.6.

3.4.2. Heat Treatment. All annealing shall be carried out in a vacuum of less than 1 x 10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1 x 10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.4.3. Contamination. All items are to be free of contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness increase not greater than 50 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050-inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.3. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analysis for products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Wall Thicknesses 0.020 Inch or Greater

Element	Check Analysis Limits, Max., ppm	Permissible Variations in Check Analysis, ppm
Carbon	100	+ 10
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I

CHEMICAL COMPOSITION

FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

Element	Minimum Content ppm	Maximum Content ppm
Carbon	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26.0 $^w/_o$	29.0 $^w/_o$
Tungsten	10.0 $^w/_o$	12.0 $^w/_o$
Zirconium	0.60 $^w/_o$	1.10 $^w/_o$
Columbium	Remainder	-

For Wall Thicknesses Less Than 0.020 Inch

Element	Check Analysis Limits, Max., ppm	Permissible Variations in Check Analysis, ppm
Carbon	150	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Grain Size. The grain size of the final products shall conform to the following limits:

Product Wall Thickness, Inches	Minimum Allowable ASTM Grain Size No.	Allowable Spread in ASTM Grain Size Nos. in Any One Item	% R_x Minimum
Less than 0.010	6	2	100
0.010 to 0.065	6	2	100
0.065 to 0.125	5	2	100
0.125 to 0.250	4	2	95
0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be capable of the following property limits at room temperature (65°-85°F).

Ultimate Tensile Strength, ksi		0.2% Yield Strength, ksi		Elong., %(1)
Minimum	Maximum	Minimum	Maximum	Minimum
70	100	50	80	20

(1) % Elongation in 4D for Threaded or Button-Head Test Specimens; in 2 Inches for Flat Test Specimens.

3.7.2. Stress-to-Rupture Tests. The material shall be capable of achieving the following stress-rupture life under suitable environmental conditions (see paragraph 5.4.3):

Test Temp., °F	Stress, ksi	Minimum Life Hours
2200	16	20

Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits: total increase in oxygen plus nitrogen content--less than 100 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to check analyses of the analytical results:

Carbon ± 10 ppm

Oxygen ± 50 ppm

Nitrogen ± 50 ppm

Hydrogen ± 2 ppm

3.7.3. Hydrostatic Test. Each tube, 1/8 inch or larger in outside diameter with a wall thickness of 0.015 inch or over, shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 12,000 psi. The test pressure, not to exceed 10,000 psi, shall be determinated by the equation ($P = 2St/D$), where:

P = hydrostatic test pressure in pounds per square inch;

S = 12,000 psi

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

3.7.4. Flare Test. A section of the heat treated tube shall be capable of being flared without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 15%.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 9).

3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8 inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4 inch will be permitted, unless otherwise specified.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600, unless otherwise agreed upon.

3.9. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and Inclusions. Indications with dimensions greater than 3% of the wall thickness shall be unacceptable. Indications with dimensions in the range of 1% to 3% of wall thickness must be a minimum of 0.50 inch apart. Indications with dimensions less than 1% of the wall thickness must be a minimum of 0.12 inch apart.

4.3. Surface Rework. Defects less than 3% of the nominal wall thickness detected by penetrant or ultrasonic inspection may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II (refer to page 9).

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

TABLE II

PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

Nominal OD Inches	OD Inch	ID Inch	Wall Thickness %
0.187 to but not incl. 0.625	± 0.004	± 0.004	± 10
0.625 to but not incl. 1.000	± 0.005	± 0.005	± 10
1.000 to but not incl. 2.000	± 0.0075	± 0.0075	± 10
2.000 to but not incl. 3.000	± 0.010	± 0.010	± 10
3.000 to but not incl. 4.000	± 0.0125	± 0.0125	± 10

NOTES: --

(1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.

(2) For tolerances applicable for very small tubes (less than 0.187-inch diameter) or very thin-wall tubes (less than 0.010-inch thick), the producer shall be consulted.

(3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.

(4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

5.2. Customer Review. The purchaser or his representative may witness the testing and inspection of the material. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement for purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operations.

5.3. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraphs 5.4.2 and 5.4.3 shall be mutually agreed upon by the vendor and purchaser prior to the placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commercial Columbium."

5.4.2. Tensile Test. The tension test shall be performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Tests. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested in a vacuum of 1 x 10^{-6} torr or better. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112-61, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Tensile Test - two per lot per ingot

Stress-Rupture Test - two per lot per ingot

Flare Test - two per lot per ingot

Grain Size - two per lot per ingot

Microhardness Traverse - one per lot per ingot

Hydrostatic Proof Test - 100%

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2 using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, the material shall be inspected ultrasonically.

5.7.1.2.1. Method and Equipment. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Inspection shall be by both circumferential and axial shear techniques with longitudinal wave being added when the wall thickness is greater than 0.150 inch. For longitudinal wave technique and for circumferential shear, transducers up to 2 inches long may be used with or without automatic equipment to rotate the tube past the transducer. If spiral pattern inspection traverse is not used, steps must be taken to assure that the ultrasonic beam remains in the same position relative to the tubing so the beam-to-tubing angle remains constant. For axial (longitudinal) shear, transducers must have no greater than 0.5 inch axial length. Transducers must be cylindrically focused for a diameter range which includes the tubing on which it is to be used.

5.7.1.2.2. Calibration. Calibration shall be on notches (a total of four, two axial and two circumferential), cut in the tube on both the outside and inside surface unless otherwise specified. The depth of the notches shall be 3% of the wall thickness to a minimum depth of 0.001 inch; the width, no greater than depth; the length, at least that of the ultrasonic beam with a maximum length of 1 inch. Material having a wall thickness greater than 0.150 inch shall also have a 0.020-inch diameter hole machined into the wall in the longitudinal direction at mid-point of the wall thickness. Focusing shall be done to maximize the indication from the inside diameter notch placed properly for the type of inspection contemplated. After focusing is completed, the inside diameter indication shall be set at 80% and gain setting recorded. Gain setting for 80% on the outside diameter notch shall also be recorded. Inspection shall be at the gain setting for the inside diameter indication. A distance corresponding to the wall thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. If calibration has changed (gain change greater than 5%), all inspections since the previous calibration shall be repeated.

5.7.1.2.3. Rejection. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication; i.e., inside diameter defects shall be compared to the indication from the notch on the inside diameter, and outside diameter defects shall be compared to the indication from the notch on the outside diameter. Defects less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the outside diameter calibration indication. Defects more than half the thickness from the incident surface or more than 0.150 inch from the surface shall be compared to the indications from the inside diameter calibration notch.

5.7.1.2.4. Reports. The ultrasonic inspection report shall contain the equipment serial numbers, calibration amplitudes and gain settings and the amplitude and location of each defect whose amplitude is 60% or greater.

5.7.1.3. Penetrant Inspection. The exterior surface of the product shall be penetrant inspected and found free of flaws as specified in paragraph 4.3 using AMS 2645, "Fluorescent Penetrant Inspection," or AMS 2646, "Contrast Dye Penetrant Inspection." All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of a report showing inspection results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

5.8. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. An analysis may be requested by the purchaser chaser of the metal, after it has been processed into finished mill forms, for the purpose of verifying the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29-58T, "Recommended Practices for Designating Significant Places in Specified Limiting Values."

Test	Rounded-Off Unit for Observed or Calculated Value
Chemical composition and dimensional tolerances (when expressed decimally)	Nearest unit in the last right-hand place of figures of the specified limit
Tensile strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture life	Nearest 0.1 hour

SPECIFICATION

HIGH PURITY LITHIUM METAL

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

1. SCOPE

1.1. Scope. This specification covers the requirements for the high purity grade of lithium metal.

2. APPLICABLE DOCUMENTS

2.1. Government Documents

MIL-STD 271C (Ships) 1 October 1963	Non-Destructive Testing
Interstate Commerce Commission Regulations Applying to Shippers, Part 73-206.	

2.2. Non-Government Documents

03-0013-00-B 2 August 1965	Mass Spectrometric Leak Detection Using Helium
03-0014-00-A 1 May 1964	Welding of Austenitic Stainless Steels
ASME Unfired Pressure Vessel Code, Section VIII, 1963	

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Chemical Composition

3.2.1. Product Composition. The Manufacturer's analysis on the product shall conform to the requirements in Table I.

3.2.2. Check Analysis. On receipt, the product will be sampled and reanalyzed, and shown to conform to the requirements on Table II. Non-conformance will be reason for rejection of the shipment.

4. QUALITY ASSURANCE PROVISIONS

4.1. Chemical Analysis

4.1.1. Certifications. The analysis made by the material manufacturers to determine the concentrations of elements stipulated in this specification shall be reported to the purchaser at the time of shipment in a certificate of test. The certification will be supplied in triplicate.

4.1.2. Vendor Analyses. The chemical analyses shall be performed in accordance with ASTM methods when available. When ASTM methods are not available, the analyses will be performed according to written procedures which result in data of known precision and accuracy. Such procedures will be supplied to the purchaser, and the vendor will reference such procedures on test certificates. If procedures are proprietary, the vendor will keep them on file for review with the government contractor, if requested.

4.1.3. Check Analyses. Check analyses will be performed by the purchaser and/or Nuclear Materials and Equipment Corporation, Apollo, Pa.

4.2. Transfer Methods

4.2.1. Environmental Conditions. Shipping containers will be fabricated from an austenitic stainless steel in accordance with Spec. 03-0014-00-A and ASME Unfired Pressure Vessel Code, Section VIII, 1963; and will be leak checked prior to use in accordance with either Spec. 03-0013-00-B or MIL-STD 271C (Ships). The maximum allowable leak rate will be 5 x 10^{-10} std. cc. of air per sec. The lithium will be transferred from storage to shipping containers through stainless steel lines and valves under conditions which do not result in a nitrogen contamination of greater than 20 ppm and an oxygen contamination of greater than 50 ppm. The lithium must not contact brazed or soldered connections or joints during the transfer operation. The maximum transfer temperature will be 500°F. Containers are to be pressurized to 5 psig argon pressure at ambient temperature. The purity of the argon used should be equivalent to or greater than commercial welding grade argon.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each shipping container shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order number, lot number and gross, net and tare weights.

5.2. Packing. Material will be shipped in accordance with Interstate Commerce Commission, Regulations Applying to Shippers, Part 73-206. Each shipment will be legibly and conspicuously marked, "Danger - Hazardous Material - Keep Away from Water."

6. NOTES. None

TABLE I

PURITY SPECIFICATION FOR HIGH PURITY LITHIUM

Element	Maximum Content (ppm)
Oxygen	150 (in Li)
Nitrogen	50 " "
Carbon	50 (in Li) total
Chlorine	50 (in Li)
Sodium	15 (in Li_2CO_3)
Potassium	25 " "
Aluminum	10 " "
Barium	10 " "
Beryllium	10 " "
Boron	25 " "
Calcium	10 " "
Chromium	10 " "
Cobalt	10 " "
Columbium	10 " "
Copper	10 " "
Iron	10 " "
Lead	10 " "
Magnesium	10 " "
Manganese	10 " "
Molybdenum	10 " "
Nickel	10 " "
Silicon	10 " "
Silver	10 " "
Strontium	10 " "
Tin	10 " "
Titanium	10 " "
Vanadium	10 " "
Zirconium	10 " "
Lithium	99.94%

TABLE II

CHECK ANALYSIS FOR HIGH PURITY LITHIUM

Element	Maximum Content (ppm)
Oxygen	200 (in Li)
Nitrogen	60 " "
Carbon	60 (in Li) total
Sodium	25 (in Li_2CO_3)
Potassium	50 " "
Aluminum	15 " "
Barium	15 " "
Beryllium	15 " "
Boron	25 " "
Calcium	15 " "
Chromium	15 " "
Cobalt	15 " "
Columbium	15 " "
Copper	15 " "
Iron	15 " "
Lead	15 " "
Magnesium	15 " "
Manganese	15 " "
Molybdenum	15 " "
Nickel	15 " "
Silicon	15 " "
Silver	15 " "
Strontium	15 " "
Tin	15 " "
Titanium	15 " "
Vanadium	15 " "
Zirconium	15 " "

SPECIFICATION

HIGH PURITY GRADE
POTASSIUM METAL

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

1. SCOPE

1.1. Scope. This specification covers the high purity grade potassium metal.

2. APPLICABLE DOCUMENTS

2.1. Government Documents

MIL - STD.-271C

Interstate Commerce Commission, Regulations Applying to Shippers, Part 73-206.

2.2. Non-Government Documents. None

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase acknowledgments.

3.2. Chemical Composition

3.2.1. Product Composition. The manufacturers analysis on the product shall conform to the requirements in Table I.

3.2.2. Check Analysis. On receipt, the product will be sampled and reanalyzed, and shown to conform to the requirements in Table II. Nonconformance will be reason for rejection of the shipments.

4. QUALITY ASSURANCE PROVISIONS

4.1. Chemical Analysis

4.1.1. Certifications. The analysis made by the material manufacturers to determine the percentage of elements stipulated in this specification shall be reported to the purchaser at the time of shipment. in a certificate of test. The certification will be supplied in triplicate.

4.1.2. Vendor Analyses. The chemical analyses shall be performed in accordance with ASTM methods when available When ASTM methods are not available, the analyses will be performed according to written procedures which result in data of known precision and accuracy. Such procedures will be supplied to the purchaser and the vendor will reference such procedures on test certificates. If procedures are proprietary, the vendor will keep them on file for review with the government contractor, if requested.

4.1.2.2. Check Analyses. Check analyses will be performed by the purchaser and/or by Nuclear Materials and Equipment Corporation, Apollo, Pa.

4.2. Transfer Methods

4.2.1. <u>Environmental Conditions</u>. Shipping containers will be mass spectrometer leak-checked per MIL-STD-271C. The potassium will be transferred from storage to shipping containers through stainless steel lines and valves under conditons which do not result in an oxygen contamination of greater than 5 ppm. The potassium must not contact brazed or soldered connections or joints during the transfer operation. The maximum transfer temperature will be 250°F. Containers are to be pressurized to 5 psig argon pressure after the potassium is frozen.

5. PREPARATION FOR DELIVERY

5.1. <u>Identification</u>. Each shipping container shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order number, lot number and gross, net and tare weights.

5.2. <u>Packing</u>. Material will be shipped in accordance with Interstate Commerce Commission, Regulations Applying to Shippers, Part 73-206. Each shipment will be legibly and conspicuously marked "Danger - Hazardous Materials - Keep Away from Water."

6. <u>NOTES</u>. None

TABLE I

PURITY SPECIFICATION FOR HIGH GRADE POTASSIUM

Element	Maximum Content (ppm)
Oxygen	50 (in K) as K_2O
Sodium	50 (in KCl)
Tin	5 "
Lead	5 "
Cobalt	5 "
Nickel	25 "
Iron	50 "
Boron	10 "
Manganese	10 "
Copper	30 "
Silver	5 "
Aluminum	10 "
Magnesium	8 "
Chromium	5 "
Silicon	25 "
Titanium	5 "
Molybdenum	3 "
Vanadium	5 "
Beryllium	5 "
Zirconium	10 "
Strontium	5 "
Barium	5 "
Calcium	25 "
Potassium	99.99% min.

TABLE II

CHECK ANALYSIS FOR HIGH PURITY GRADE POTASSIUM

Element	Maximum Content (ppm)
Oxygen	60 (in K) as K_2O
Sodium	50 (in KCl)
Tin	5 "
Lead	5 "
Cobalt	5 "
Nickel	25 "
Iron	50 "
Calcium	25 "
Manganese	10 "
Copper	30 "
Silver	5 "
Magnesium	8 "
Chromium	5 "
Silicon	25 "
Titanium	5 "
Aluminum	10 "
Zirconium	10 "
Molybdenum	5 "
Columbium	5 "

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SPPS-46-II
10 May 1965
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SPECIFICATION

HOT TRAPPED HIGH PURITY GRADE POTASSIUM METAL

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

1. SCOPE

1.1. Scope. This specification covers the hot trapping and purity of high purity grade potassium metal.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

01-0033-00-B 10 May 1965	High Purity Grade Potassium Metal
03-0018-00-A 15 June 1965	Alkali Metal Handling and Control Procedures
03-0014-00-A 1 May 1964	Welding of Austenitic Stainless Steels for Liquid Metal Systems

3. REQUIREMENTS

3.1. Hot Trapping Procedure

3.1.1. Outgassing. The alkali metal will be outgassed in the shipping container, as indicated below, prior to transfer to the hot trap. A vacuum system capable of achieving a blank-off pressure of 1×10^{-3} torr will be used. Trapping will be provided between the vacuum pump and the vacuum-inert gas manifold to prevent contamination of the system by back-streaming of pump oil vapors. The inert gases used (helium or argon) will be shown to contain less than 10 molar ppm combined water and oxygen. Referring to Figure 1, the procedure is:

a. Make sure that all valves are clean to the seats and then connect the vacuum-inert gas manifold as shown in Figure 1. (See Spec. 03-0018-00-A for valve cleaning.)

b. Heat the shipping container gas valve and the line connecting it to the shipping container to a temperature of 250°F plus or minus 25°F and pressurize the manifold to about 20 psia with inert gas.

c. Open the shipping container gas valve to blow down any alkali metal which it may contain.

d. Evacuate the shipping container through the gas valve, heat it to between 450°F and 600°F and monitor the pressure in the manifold.

e. When the pressure levels off or drops below 1×10^{-2} torr, close the valve at the trap and measure the pressure rise rate. Continue evacuating and checking pressure rise until the rate is less than 10^{-2} torr-liters per minute.

f. Cool the shipping container at a rate not exceeding 50°F per hour to a temperature which exceeds the melting point of the alkali metal by not more than 50°F.

3.1.2. Hot Trapping. The hot trap will be built of an austenitic stainless steel and will be capable of withstanding an internal pressure of 50 psig at 1500°F for at least 10,000 hours. It will be lined with titanium or zirconium sheet, not less than 0.01 inch thick, in a manner which prevents liquid metal between the liner and the hot trap body from returning to the interior of the liner when the hot trap is properly oriented. The getter shall be titanium, zirconium or 50% titanium-50% zirconium alloy sheet, turnings or chunks (not sponge), and the alkali metal weight to getter surface ratio shall not exceed 10 grams/square inch. When it is known that the oxygen content of the getter is greater than 10% by weight for titanium of 5% by weight for zirconium, the liner and getter will be replaced.

All valves used will be of austenitic stainless steel except plugs and/or seats which may be Stellite. They will be of the bellows type, sealed with metal gaskets or welded. Valves which contact the alkali metal will be angle pattern and will be oriented so that the bellows is between the hot trap and valve seat with one exception. The exception is the valve on the connecting line between the shipping container and hot trap. Here the bellows should be between the valve seat and the vacuum-inert gas manifold. All valves should be attached so that the plug axis is vertical and that the handle is toward the ground to facilitate cleaning. (See Spec. 03-0018-00-A for valve cleaning.)

All welding will be performed according to Spec. 03-0014-00-A .

Before use, the hot trap will be outgassed at a temperature exceeding 250°F until the pressure rise rate is less than 1×10^{-3} torr-liter per minute. It will be helium leak-checked at this temperature with a maximum allowable leak rate of 5×10^{-10} std. cm^3 of air per second. Hot traps will have a stainless steel filter having a nominal pore size of 5 microns on the dip leg between the hot trap and the valve.

Referring again to Figure 1, the procedure is:

a. Heat the connecting line between the shipping container and hot trap, including valves, to 250°F and evacuate through the vacuum-inert gas valve. Helium leak-check transfer line.

b. Pressurize manifold to 20 psia and open the hot trap gas valve to blow down alkali metal which may be in it.

c. Evacuate the hot trap and transfer line to less than one torr inert gas pressure and close the manifold valve.

d. Pressurize the shipping container to about 20 psia.

e. Heat all components to a temperature exceeding the melting point of the alkali metal. The temperature of the shipping container filter should not exceed the melting point of the alkali metal by more than 50°F.

f. Open the gas valve of the hot trap and then the dip leg valve of the shipping container and transfer the desired quantity of metal to the hot trap. (See Spec. 03-0018-00-A for methods for determining quantities transferred.)

g. Close the dip leg valve on the shipping container and the gas valve on the hot trap. Cool the transfer system to room temperature and remove it, and clean valves.

h. Reconnect the vacuum-inert gas valve to the hot trap gas valve and evacuate back to the seat of the gas valve.

i. Heat the gas valve to a temperature exceeding the melting point of the alkali metal and pressurize the manifold to about 17 psia.

j. Open the hot trap gas valve to pressurize the hot trap to about 17 psia and maintain this pressure during subsequent operations.

k. Heat the hot trap to a temperature between 1300°F and 1450°F and maintain it at temperature for at least 20 hours.

l. Cool to about 400°F and remove specimens for analysis. The specimen collecting technique is not specified, but one of a number of methods described in Spec. 03-0018-00-A must be used, depending on the analytical apparatus which applies.

3.2. Chemical Composition

3.2.1. Oxygen. The oxygen content shall be less than 20 ppm by weight as K_2O in sodium.

3.2.2. Metal Impurities. The metallic impurity levels shall conform to the requirements in Table I.

4. QUALITY ASSURANCE PROVISIONS

4.1. Certification. After completion of the analyses, a report will be prepared in quadruplicate indicating the lot number of the purified batch and referencing the notebook and page number covering the purification and analyses. This report will be distributed as follows:

1. Project Engineer in charge at facility in which the potassium will be used.
2. Quality Assurance Engineer
3. Alkali Metal Custodian
4. File

4.2. Chemical Analysis

4.2.1. Specimen Preparation. Analytical specimens will be prepared in accordance with techniques described in Spec. 03-0018-00-A.

4.2.2. Analytical Methods

4.2.2.1. Oxygen. Oxygen analyses will be performed by the methods described in Spec. 03-0018-00-A.

4.2.2.2. Metal Impurities. Analyses will be performed in accordance with methods found in Spec. 03-0018-00-A.

5. PREPARATION FOR DELIVERY. None

6. NOTES. None.

TABLE I

ANALYSIS OF HOT TRAPPED HIGH PURITY GRADE POTASSIUM METAL

Element	Maximum Content (ppm)
Tin	5 (in KCl)
Lead	5 " "
Cobalt	5 " "
Nickel	10 " "
Iron	10 " "
Calcium	25 " "
Manganese	10 " "
Copper	30 " "
Silver	5 " "
Magnesium	8 " "
Chromium	5 " "
Silicon	25 " "
Titanium	5 " "
Aluminum	10 " "
Zirconium	10 " "
Molybdenum	5 " "
Columbium	5 " "

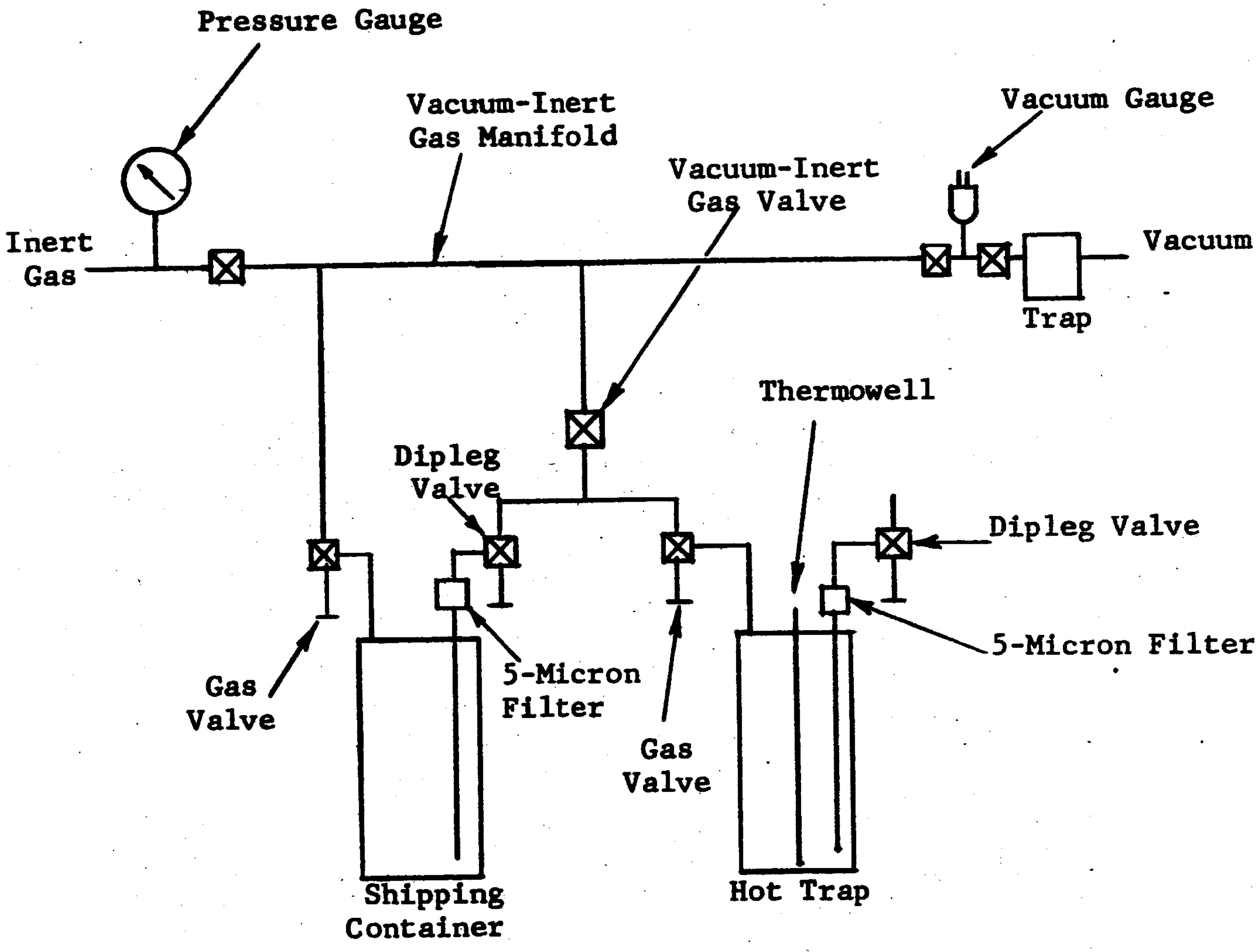

Figure 1. Schematic Drawing of the Apparatus for Outgassing and Hot Trapping Potassium

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SPPS-62
23 December 1964
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SPECIFICATION

FOIL: FS-85 (Cb-28Ta-10.5W-0.9Z r) ALLOY

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

FOIL: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in foil form intended for high temperature non-structural applications.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation (Pending)	Methods for Chemical Analysis of Reactor and Commercial Columbium

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition through the cross-sectional area. All annealing shall be carried out in a vacuum of less than 1 x 10-5 torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1 x 10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped of a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyses shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses for products supplied under this specification (Table I), except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. Finished product analysis shall not exceed the following limits or variations:

Element	Check Analysis Limits, Max., ppm	Permissible Variations in Check Analysis, ppm
Carbon	150	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Bend Ductility. Representative samples of the materials in final form shall withstand a 180° bend without failure.

3.7. Tolerances

3.7.1. Definition. Foil includes material less than 12 inches wide and up to and including 0.010-inch thick.

TABLE I

CHEMICAL COMPOSITION

FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

Element	Minimum Content ppm	Maximum Content ppm
Carbpn	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26 $^w/_o$	29 $^w/_o$
Tungsten	10 $^w/_o$	12 $^w/_o$
Zirconium	0.6 $^w/_o$	1.1 $^w/_o$
Columbium	Remainder	-

3.7.2. Dimensions. Foil dimensions shall conform to the following limits:

Material Thickness Inches	Thickness Tolerances Inch	Width Tolerance Inch
Less than 0.003	+0.0008, -0.0000	+0.031, -0.000
0.003 to 0.005	+0.001	+0.031, -0.000
0.005 to 0.010	+0.0015	+0.031, -0.000

3.8. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analyses of Reactor and Commercial Columbium."

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

Bend Test - two per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or other similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITION

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

01-0046-00-A
SPPS-66
23 December 1964
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SPECIFICATION

WIRE: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

WIRE: FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

1. SCOPE

1.1. Scope. This specification covers FS-85 (Cb-28Ta-10.5W-0.9Zr) alloy in wire form for use as weld filler material in fabricating components intended for high temperature structural applications and alkali metal containment.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation (Pending)	Methods for Chemical Analysis or Reactor and Commercial Columbium

3. REQUIREMENTS

3.1. Acknowledgments. The vendor shall mention this specification in all quotations and all purchase order acknowledgments.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been double vacuum melted by the electron beam and/or consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging and rolling equipment normally found in primary ferrous and nonferrous plants.

3.3. Processing. The starting stock size, processing temperatures, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor.

3.4. Condition. The finished product shall be supplied in the fully recrystallized condition throughout the cross-sectional area. All annealing shall be carried out in a vacuum less than 1 x 10^{-5} torr.

All mill products to be annealed shall be thoroughly degreased, chemically cleaned and protected from furnace parts by a layer of fresh tantalum, columbium, or Cb-1Zr alloy foil 0.002-inch thick or greater. When annealing is carried out in a vacuum greater than 1 x 10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned tantalum, columbium or Cb-1Zr alloy retort or wrapped in a minimum of two layers of fresh tantalum, columbium or Cb-1Zr alloy foil 0.002-inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots and billets for conversion to finished products shall conform to Table I (page 4). A minimum of four analyes shall be obtained as follows: ingot top-center, mid-radius and edge, and ingot bottom-center; all analyses must conform to ranges stated in Table I.

3.5.2. Final Product Composition. The manufacturer's ingot analyses shall be considered the chemical analyses of products supplied under this specification (Table I) except carbon, oxygen, nitrogen and hydrogen content which shall be determined on the finished product.

3.5.3. Check Analysis. The finished product analysis shall not exceed the following limits or variations:

For Material Greater than 0.030 Inch in Diameter

Element	Check Analysis Limits, Max., ppm	Permissible Variations in Check Analysis, ppm
Carbon	100	+ 10
Oxygen	200	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

TABLE I

CHEMICAL COMPOSITION

FS-85 (Cb-28Ta-10.5W-0.9Zr) ALLOY

Element	Minimum Content ppm	Maximum Content ppm
Carbon	-	100
Nitrogen	-	75
Oxygen	-	100
Hydrogen	-	10
Molybdenum	-	200
Nickel	-	50
Cobalt	-	50
Iron	-	50
Tantalum	26 $^w/_o$	29 $^w/_o$
Tungsten	10 $^w/_o$	12 $^w/_o$
Zirconium	0.6 $^w/_o$	1.1 $^w/_o$
Columbium	Remainder	-

For Material 0.030 Inch and Less in Diameter

Element	Check Analysis Limits, Max., ppm	Permissible Variations in Check Analysis, ppm
Carbon	150	+ 10
Oxygen	300	+ 20
Nitrogen	100	+ 10
Hydrogen	10	+ 2

3.6. Tolerances

3.6.1. Definition. Wire - material less than 0.125 inch in diameter.

3.6.2. Diameter. The permissible variation in diameter shall not exceed the following limits:

Diameter, Inch	Diameter Variation, Inch
0.005 to 0.009	± 0.0002
0.010 to 0.019	± 0.0003
0.020 to 0.029	± 0.0005
0.030 to 0.061	± 0.001
0.062 to 0.125	± 0.002

3.7. General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil, residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment, unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

4.2. Sample Selection. Care shall be exercised to insure that the samples selected for testing and chemical analyses are representative of the material and uncontaminated by the sampling procedure. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller.

4.3. Chemical Analysis. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and the spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM "Methods for Chemical Analysis of Reactor and Commerical Columbium."

4.4. Number of Tests Required. Representative test specimens from the finished product representing each ingot and each lot of material shall be taken to determine conformity to this specification. The minimum frequency of these tests shall be:

Finished Product Chemistry - one per lot per ingot

4.5. Reports. The manufacturer shall supply at least three copies of a report showing non-proprietary manufacturing methods, processing conditions, and test procedures and results for each lot of material in the shipment. The report shall also include the number of the specification and the purchase order or contract number.

4.6. Rejection. Material not conforming to this specification or to any authorized modification shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense if the purchaser does not receive other instructions for disposition within three weeks after notice of rejection.

4.7. Referee. If the manufacturer and the purchaser disagree about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each bundle, box, or carton shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carton consists of components from more than one ingot number or lot number, each component shall be identified individually.

5.2. Packing. Each individual item shall be wrapped in heavy gauge polyethylene film or similar material and packed in a manner assuring safe delivery when properly transported by any common carrier.

6. DEFINITIONS

6.1. Lot. A lot shall include all material of the same size, shape, condition and finish from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

6.2. Check Analysis. An analysis, made or requested by the purchaser of the metal after it has been processed into finished mill forms, to verify the composition within a heat or lot. Check analysis tolerances do not broaden the specified heat analysis requirements but rather cover variations between laboratories in the measurement of the chemical content.

SPECIFICATION

HIGH PURITY GRADE SODIUM POTASSIUM ALLOYS (NaK)

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

1. SCOPE

1.1. Scope. This specification covers the purity requirements for as-received sodium-potassium alloys.

2. APPLICABLE DOCUMENTS

2.1. Government Documents

MIL-STD 271C (Ships) 1 October 1963	Non-Destructive Testing

Interstate Commerce Commission
Regulations Applying to
Shippers, Part 73-206.

2.2. Non-Government Documents. None

3. REQUIREMENTS

3.1. Acknowledgments. The vendor will mention this specification in all quotations and all purchase acknowledgments.

3.2. Chemical Composition

3.2.1. Product Composition. The manufacturers analysis on the product will conform to the requirements in Table I. (See also Section 6).

3.2.2. Check Analysis. On receipt, the product will be sampled, reanalyzed and shown to conform to the requirements in Table I. Nonconformance will be reason for rejection of the product.

4. QUALITY ASSURANCE PROVISIONS

4.1. Chemical Analysis

4.1.1. Certifications. The results of the manufacturers analysis on the product will be reported in a certificate of test, in triplicate, to the purchaser at the time of product shipment.

4.1.2. Vendor Analyses. The chemical analyses will be performed in accordance with ASTM methods when such methods are available. Otherwise the analyses will be performed according to written procedures which result in data of known precision and accuracy. Such procedures will be supplied to the purchaser and the vendor will reference such procedures on the test certificates. If the procedures are proprietary, the vendor will keep them on file for review with the government contractor, if requested.

4.1.2.2. Check Analyses. Check analyses will be performed by the purchaser and/or by the Nuclear Materials and Equipment Corporation, Apollo, Pa.

4.2. Transfer Methods

4.2.1. Environmental Conditions. Shipping containers will be mass spectrometer leak-checked per MIL-STD-271C. The sodium-potassium alloy (NaK) will be transferred from storage to shipping containers through austenitic stainless steel lines and valves under conditions which do not cause an oxygen contamination of greater than 5 ppm. The NaK must not contact brazed or soldered joints or connections during the transfer operation. The maximum transfer temperature will be 200°F. Containers will be pressurized to 5 psig. argon pressure at ambient temperature.

5. PREPARATION FOR DELIVERY

5.1. Identification. Each shipping container will be legibly and conspicuously marked or tagged with the number of this specification, the purchase order number, the lot number and the gross, net and tare weights.

5.2. Packing. The product will be shipped in accordance with Interstate Commerce Commission Regulations Applying to Shippers, Part 73-206. Each shipment will be legibly and conspicuously marked "Danger-Hazardous Materials - Keep Away from Water".

6. NOTES. Purchase orders must specify the particular composition of the alloy regarding the potassium content except when the eutectic is indicated.

TABLE I

PURITY SPECIFICATION FOR HIGH PURITY GRADE NaK

Element	Maximum Content - ppm
Oxygen	50 (in NaK) as K_2O and Na_2O
Potassium (Eutectic NaK)	78 (+0%, -1%)
Potassium (All other compositions)	within ± 1% of specified
Iron	25 (in Chloride)
Boron	10 "
Cobalt	5 "
Manganese	10 "
Aluminum	10 "
Magnesium	8 "
Tin	5 "
Copper	30 "
Lead	5 "
Chromium	5 "
Silicon	25 "
Titanium	5 "
Nickel	10 "
Molybdenum	5 "
Vanadium	5 "
Beryllium	5 "
Silver	5 "
Zirconium	10 "
Strontium	5 "
Barium	5 "
Calcium	25 "
Carbon	50 (in NaK) total
Sodium + Potassium	99.92% min.

SPECIFICATION

HOT TRAPPED HIGH PURITY GRADE SODIUM POTASSIUM ALLOYS (NaK)

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

1. SCOPE

1.1. Scope. This specification covers the hot trapping and purity requirements for high purity grade sodium-potassium alloys.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

01-0049-00-A 15 March 1966	High Purity Grade Sodium-Potassium Alloys (NaK)
03-0018-00-B (In Preparation, Use 03-0018-00-A)	Alkali Metal Handling and Control
03-0014-00-A 1 May 1964	Welding of Austenitic Stainless Steels for Liquid Metal Systems
03-0013-00-B 2 August 1965	Mass Spectrometer Leak Detection Using Helium

3. REQUIREMENTS

3.1. Hot Trapping

3.1.1. Apparatus. The hot trap will be constructed of austenitic stainless steel and will be capable of withstanding an internal pressure of 50 psig. at 1500°F for 10,000 hours. It will be lined with titanium or zirconium sheet, not less than 0.01-inch thick, in a manner which prevents liquid metal between the liner and the hot trap body from returning to the interior of the liner when the hot trap is properly oriented. The getter material will be titanium, zirconium or 50% titanium-50% zirconium alloy sheet, turnings or chunks (not sponge), and the alkali metal weight to getter area ratio will not exceed 10 grams/square inch. When it is known that the oxygen content of the getter is greater than 10% by weight for titanium or 5% by weight for zirconium the liner and getter will be replaced.

All valves which contact alkali metal will be austenitic stainless steel except the plugs and/or seats which may be Stellite. These valves will be the bellows type, sealed with metal gaskets or by welding, and should be the angle pattern, oriented so that they may be cleaned to the valve seat. When the valve is located in a transfer line between a shipping container and a hot trap as shown in Figure 1, the valve seat should face the transfer line. All angle pattern valves should be attached seat upward to facilitate cleaning. (See Spec. 03-0018-00-B for valve cleaning).

The hot trap will have an austenitic stainless steel filter with a nominal pore size of 5 microns in the dip leg between the hot trap and the valve as shown in Figure 1.

Auxiliary apparatus will include a vacuum-inert gas manifold. The vacuum system will be capable of achieving a blank-off pressure of at least 10^{-3} torr. Trapping will be provided between the vacuum pump and the vacuum-inert gas manifold to prevent contamination of the system by back-streaming of pump oil vapors. The inert gas (helium or argon) used will be certified to contain less than 10 molar ppm of combined water and oxygen.

All welding will be performed according to Spec. 03-0014-00-A.

3.1.2. Outgassing. Before use, the hot trap will be outgassed at a temperature exceeding 250°F until the outgassing rate is less than 1×10^{-3} torr-liters/minute when no leaks greater than 5×10^{-10} std. cc/sec can be detected by helium leak-checking (See Spec. 03-0013-00-B for leak checking). In addition, the alkali metal will be outgassed in the shipping container prior to transfer to the hot trap. Referring to Figure 1, an acceptable procedure for outgassing the hot trap and alkali metal is:

a. Make sure that all valves are clean to the seats and then connect the shipping container, hot trap and vacuum-inert gas manifold as shown in Figure 1. (See Spec. 03-0018-00-B for valve cleaning).

b. Heat the hot trap to 250°F minimum and outgas until the rate is less than 1×10^{-3} torr-liters/minute with liquid nitrogen cooled trapping between the vacuum system and the hot trap.

c. Heat the shipping container gas valve and the line connecting it to the shipping container to 100 ± 25°F and pressurize the manifold to 20 psia with inert gas.

d. Open the shipping container gas valve and blow down any alkali metal which it may contain.

e. Evacuate the shipping container through the gas valve, heat the shipping container to 450-600°F and monitor the pressure in the manifold.

f. When the pressure levels off or drops below 1×10^{-2} torr, close the hot trap gas valve and measure the outgassing rate. Continue outgassing until the rate is less than 1×10^{-2} torr-liters per minute.

3.1.3. Procedure. Referring to Figure 1, the hot trapping procedure is:

a. Heat the connecting line between the shipping container and hot trap, including valves, to 100°F and evacuate the connecting line through the vacuum-inert gas valve. Helium leak check the transfer line (See Spec. 03-0013-00-B for leak checking).

b. Pressurize the manifold to 20 psia and open the hot trap gas valve to blow down any alkali metal in it.

c. Evacuate the hot trap and transfer line to less than 0.05 torr, inert gas pressure, and close the manifold valve.

d. Pressurize the shipping container to about 20 psia, inert gas pressure.

e. Heat all components to a temperature exceeding the melting point of the alkali metal. The filter temperature should be less than 50°F above the melting point of the alkali metal.

f. Open the hot trap gas valve and the shipping container dip leg valve and transfer the desired quantity of metal to the hot trap. (See Spec. 03-0018-00-B for determination of quantities transferred).

g. Close the shipping container dip leg valve, open the manifold valve and blow part of the alkali metal in the transfer line into the hot trap.

h. Close the hot trap gas valve, adjust the manifold pressure to 5 psi greater than the shipping container pressure, open the shipping container dip leg valve and blow as much remaining metal as possible from the transfer line back into the shipping container.

i. Close the shipping container dip leg valve and vacuum-inert gas valve and cool the shipping container and transfer line to room temperature. If liquid NaK remains in the transfer line, cool the transfer line to 0-5°F with dry ice or liquid nitrogen before disconnecting the transfer line.

j. Remove the transfer line, clean all valves exposed to alkali metal and reconnect the vacuum-inert gas manifold to the hot trap gas line.

k. Evacuate the line between the manifold and the hot trap and then pressurize the hot trap to 15-17 psia and maintain this inert gas pressure on the hot trap during subsequent operations.

l. Heat the hot trap to 1300°F ± 25°F and maintain the alkali metal at this temperature for at least 20 hours. Bleed pressure off during heat up to maintain 17 psia maximum in hot trap at all times during operation.

m. Cool the hot trap to 200-400°F and remove the alkali metal samples for analysis. Use the appropriate sampling apparatus and technique specified in Spec. 03-0018-00-B for the particular alkali metal and analyses required.

3.2. Chemical Composition

3.2.1. Oxygen. The oxygen content will be less than 20 ppm by weight as Na_2O and K_2O in the NaK alloy.

3.2.2. Carbon. The total carbon content will be less than 50 ppm by weight.

3.2.3. Metallic Impurities. The metallic impurity levels will meet the requirements presented in Table I.

4. QUALITY ASSURANCE PROVISIONS

4.1. Certification. After completion of the analyses, a report of the results will be prepared in quadruplicate indicating the sample identity and referencing the notebook and page number covering the purification and analytical operations. This report will be distributed as follows:

1. Project Engineer in charge at the facility in which the alkali metal will be used.
2. Quality Assurance Engineer
3. Alkali Metal Custodian
4. File

4.2. Chemical Analysis

4.2.1. Specimen Preparation. Analytical specimens will be prepared in accordance with techniques described in Spec. 03-0018-00-B.

4.2.2. Analytical Methods

4.2.2.1. Oxygen. Oxygen analyses will be performed by the methods described in Spec. 03-0018-00-B.

4.2.2.2. Carbon. Determination of total carbon will be done by the techniques described in Spec. 03-0018-00-B.

4.2.2.3. Metallic Impurities. Analyses for metallic impurities will be performed according to methods prescribed in Spec. 03-0018-00-B.

5. PREPARATION FOR DELIVERY. None

6. NOTES. None

TABLE I

PURITY SPECIFICATION FOR HOT TRAPPED HIGH PURITY GRADE NaK

Element	Maximum Content - ppm
Oxygen	20
Potassium (Eutectic NaK)	78 (+0%, -1%)
Potassium (All other compositions)	within ± 1% of specified
Iron	25 (in Chloride)
Boron	10 " "
Cobalt	5 " "
Manganese	10 " "
Aluminum	10 " "
Magnesium	8 " "
Tin	5 " "
Copper	30 " "
Lead	5 " "
Chromium	5 " "
Silicon	25 " "
Titanium	5 " "
Nickel	10 " "
Molybdenum	5 " "
Vanadium	5 " "
Beryllium	5 " "
Silver	5 " "
Zirconium	10 " "
Strontium	5 " "
Barium	5 " "
Calcium	25 " "
Carbon	50 (in NaK) total
Sodium + Potassium	99.92% min.

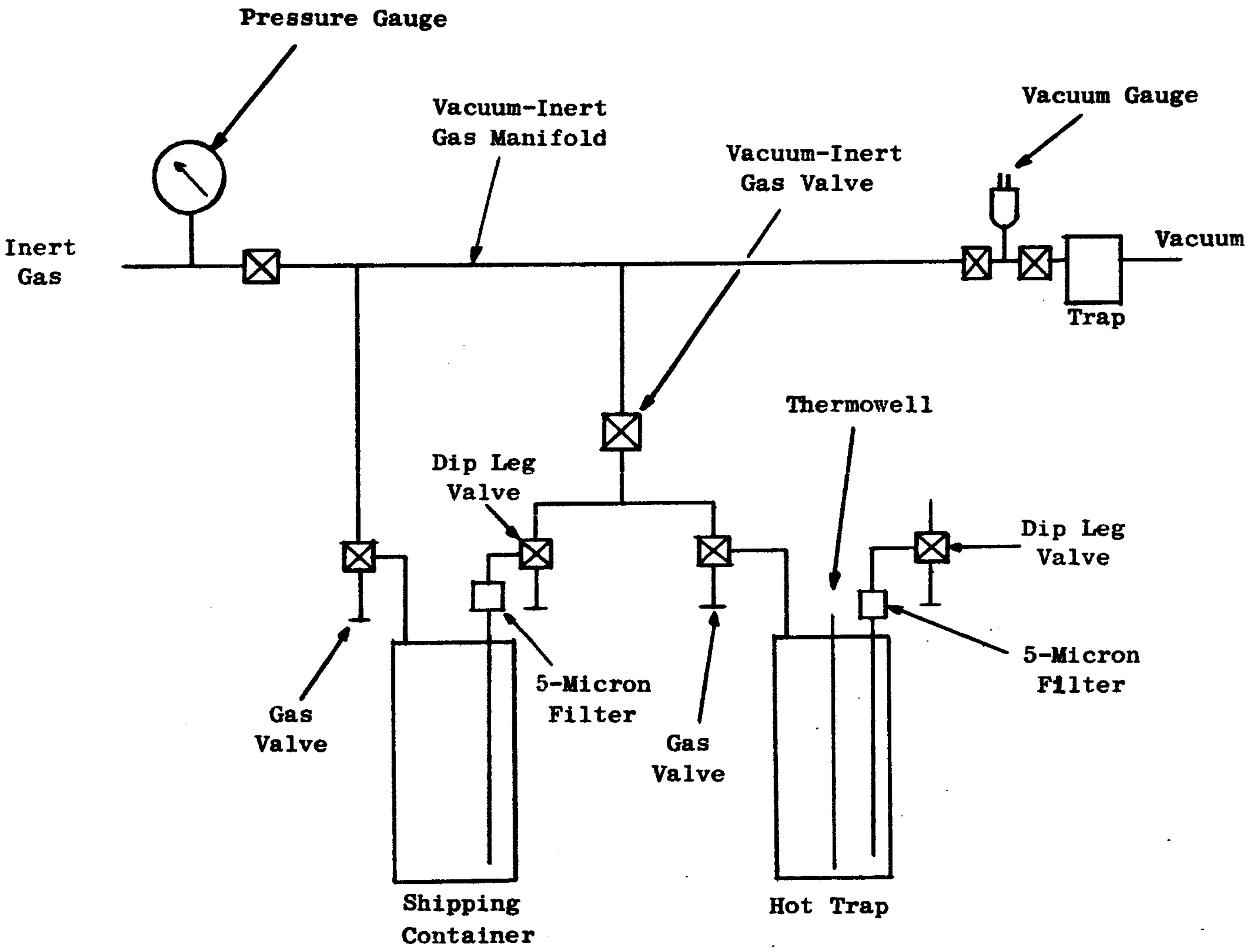

Figure 1. Schematic Drawing of the Apparatus for Outgassing and Hot Trapping Alkali Metal.

SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM ALLOY BY THE INERT-GAS TUNGSTEN ARC PROCESS

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM ALLOY BY THE INERT-GAS TUNGSTEN ARC PROCESS

1. SCOPE

1.1. Scope. This specification establishes the procedures, process substantiation, and quality requirements for fusion welding the columbium - 1% zirconium alloy by the inert-gas shielded tungsten arc-welding process. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements on the drawings supersede this specification.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents.

ASTM Designation B297-55T (1 February 1955)	Tungsten Arc-Welding Electrodes
AMS 2635 (15 August 1955)	Radiographic Inspection

3. REQUIREMENTS

3.1. Materials

3.1.1. Inert gases used shall be Bureau of Mines Grade A helium or argon, welding grade, 99.99% minimum purity, -80°F maximum dewpoint. The inert-gases shall be purified to contain less than 1 ppm active impurities by volume.

3.1.2. Tungsten electrodes, class EWTH-2, shall conform to ASTM Designation B297-55T, "Tungsten Arc-Welding Electrodes".

3.1.3. Welding filler material composition shall conform to the specification for the base metal.

3.2. Equipment

3.2.1. A direct current, arc-welding machine, equipped with a contactor and a foot-operated remote welding-current control, shall be used.

3.2.2. The welding shall be done in an enclosed chamber that can be evacuated to less than 1×10^{-5} torr. The leak rate shall result in a pressure increase of not more than 5 microns/hour starting at 1×10^{-3} torr pressure. The chamber shall be equipped with glove ports and neoprene rubber gloves that are sealed gas-tight to the ports.

3.2.3. The chamber shall be equipped with a water-cooled tungsten arc-welding torch. The water-cooling passage of the torch shall be permanently sealed. The water-cooled cable and hose connections shall be leak tight. The inert-gas shield line shall not be used.

3.3. Cleaning

3.3.1. The parts to be joined shall be free of dirt, grease, cutting compounds, or any extraneous material which will attack or react with the metal during welding or contaminate the inert-gas atmosphere. The metal adjacent to the weld shall be chemically cleaned to remove surface oxide and/or nitride with a solution of $20\%HF-20\%HNO_3-60\%H_2O$ by volume.

3.3.2. The welding filler-wire shall be washed with acetone and shall be chemically cleaned to remove dirt, oxide, grease, drawing compounds using the above solution.

3.4. Fixtures

3.4.1. Design. The fixtures used to position the parts shall hold the parts in proper alignment and minimize distortion.

3.4.2. Materials. The fixtures shall be made of materials that do not corrode or rust in air. To avoid metallic contamination, the fixture components that contact the columbium parts near the weld joint shall be made of molybdenum, tungsten, or columbium.

3.4.3. Cleaning. The fixture shall be clean and free of surface contamination.

3.5. Welding Procedures.

3.5.1. Welding of components shall be done using equipment and procedures which conform to Section 4. The parts to be welded and the required fixturing shall be loaded into the welding chamber, evacuated of air to 1×10^{-5} torr or better. The chamber shall then be filled with inert-gas containing less than 1 ppm active impurities by volume, evacuated to less than 1×10^{-5} torr, and refilled with inert gas. Before removing the glove port cover plates, the evacuation line to the glove ports shall be closed.

3.5.2. Direct current, straight-polarity power shall be used for welding. The welding torch shall be used without a gas cup and without gas flow.

3.5.3. Before welding the columbium parts, the purity of the welding atmosphere shall be checked by making fusion weld beads on titanium sheet or bar. If the titanium weld shows any discoloration, the inert atmosphere shall be considered unsuitable for welding columbium alloys.

3.5.4. Prior to the first piece welded and subsequent to the last piece welded in each inert gas environment, weld bend specimens shall be prepared using the same filler wire material used to weld the intervening pieces. Each specimen shall be from 0.04 to 0.08 inch thick, 1 inch wide (minimum), and 2 inches long (minimum), with a longitudinal weld bead weighing 2.5 grams (minimum).

The above specimens and 10-gram (minimum) samples taken from each heat of sheet and filler wire used to make the bend specimens shall be submitted with the finished parts. Specimens and samples shall be tagged with the drawing number, part number, and serial number of each part represented by the samples and specimens, and, when multiple welds on a part are made in separate environments, shall describe the particular welds for which the specimens and samples are representative.

Note: These specimens and samples shall be retained by quality assurance for subsequent chemical analysis should an investigation of welding contamination be desired.

3.5.5. Precautions shall be taken during welding to avoid contamination of the weld metal by the tungsten electrode.

3.5.6. The completed weldment shall be left in the chamber until it cools to below 400°F.

3.5.7. In repair welding, the weld defects shall be removed by machining or filing. Unless approved by the General Electric project engineer, grinding is prohibited. The area shall then be rewelded, according to the requirements of this specification, and reinspected.

3.6. Postweld Heat Treatment

3.6.1. The weld and adjacent heat-affected zone shall be postweld annealed at 2200°F, plus or minus 25°F, for one hour in a vacuum of 1×10^{-5} torr or better. The annealing may be local at the weld joint or the entire weldment may be annealed in a vacuum furnace. The procedures and equipment used shall conform to paragraph 4.3.

3.6.2. Localized heating shall be done using a heater having only refractory metals in the hot zone. Dense vitrified alumina (99.2% purity) shall be used as the heater electrical insulation in the cold zone of furnace. The temperature of the weld joint shall be measured by a Pt/Pt-Rh thermocouple that is resistance spot-tacked to the weld metal. After heat treatment the thermocouple spot-tack shall be filed off.

3.6.3. A columbium - 1% zirconium control specimen, weighing 5 grams or more, shall accompany the welded components or parts through postweld vacuum heat treatment.

The above specimens and a similar, but unheated, specimen of the control material shall be submitted with the finished parts. Specimens shall be tagged with the drawing number, part number, and serial number of each part represented by the specimens.

Note: These specimens shall be retained by quality assurance for subsequent chemical analysis, should an investigation of vacuum environment contamination be desired.

3.7. Defect Limits. The completed weldment shall be examined for defects visually and by ultrasonic or x-ray techniques, or both. Welds which do not meet the following quality requirements of this specification are unacceptable.

3.7.1. Visual and Radiographic. Welds shall be reasonably smooth and uniform in appearance and free of the following defects by visual and radiographic inspection:

1. Cracks in the weld and adjacent base metal,
2. Crater checks or cracks,
3. Surface inclusions and pores,
4. Cold layers in deposited weld metal,
5. Overlap of weld metal on base metal (not fused),
6. Undercutting of the base metal,
7. Depression of the weld below the base metal,
8. Unfilled weld craters or stops,
9. Damage to the weld and adjacent base metal by contamination from the shielding gas or from foreign materials or metals,
10. Lack of fusion of the weld deposit to the base metal,
11. Incomplete fusion and penetration to the root of the joint (back side of butt and groove welds),
12. Porosity in the weld metal,

13. Tungsten inclusions having
    a. the longest dimension greater than 0.010 inch in welds in material up to 0.10 inch thick and greater than 10% of the metal thickness in material 0.10 inch thick and above
    b. spacing between two adjacent tungsten inclusions less than three times the metal thickness
    c. more than three inclusions per inch of weld length.

3.7.2. Ultrasonic. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication. For tubing, inside diameter (or under side) defects shall be compared to the indication from the notch on the inside diameter (or under side), and outside diameter (or upper side) defects shall be compared to the indication from the notch on the outside diameter (or upper side). Indications less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the upper side (or outside diameter) calibration indication. Defects more than half the distance from the incident surface or more than 0.150 inch from the surface shall be compared to the indication from the inside diameter (or under surface) notch.

4. QUALITY ASSURANCE PROVISIONS

4.1. Weld Inspection Procedures. All welded joints shall be inspected for conformance to the quality requirements visually and by X-ray or ultrasonic techniques, or both.

4.1.1. X-ray Inspection

4.1.1.1. X-ray inspection shall be done according to AMS-2635, "Radiographic Inspection", Fine grain film shall be used for maximum sensitivity. The penetrameter thickness and hole sizes shall be based on the smaller section thickness being joined. The radiographic technique shall be considered satisfactory if the smallest hole in the penetrameter can be clearly distinguished on the radiograph.

4.1.1.2. The radiographs shall be taken in positions that will best delineate lack of weld penetration and fusion. Wherever possible, the film shall be placed on the back side of the weld joint.

4.1.1.3. When the back side of the weld is not accessible and the radiation must pass through a double thickness, only that portion of the weld next to the film shall be considered.

4.1.1.4. Radiographic interpretation for weld quality shall be done by the General Electric project engineer or his representative.

4.1.2. Ultrasonic Inspection

4.1.2.1. When required by the drawing, welded joints shall be ultrasonically rather than radiographically inspected.

4.1.2.2. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Shear wave technique shall be used with the beam traverse perpendicular to the weld centerline. Inspection shall be from both directions toward the weld. For welds in tubing, a transducer focused to the diameter of the tubing shall be used with a maximum axial length on the transducer of 0.5 inch. For welds in plate, a spherically focused transducer or a cylindrically focused transducer of less than 1.5 inch diameter focus and 0.5 inch or less long shall be used with its axis parallel to the weld centerline.

4.1.2.3. Calibration shall be on notches cut in a tube or plate of similar material, preferably near a weld of similar nature. These circumferential notches shall be: depth, 3% of the original wall or plate thickness; width, not more than the depth; length, at least one beam width. One notch shall be placed on the inside diameter or under side near the weld; the other notch shall be placed on the outside diameter or upper side near the weld but on the opposite side of the weld or at least two beam widths away (at the same distance from the weld) if on the same side of the weld. Focusing shall be done to maximize the indication from the notch on the under side or inside diameter. After focusing is completed, whichever indication is larger shall be set at 80% and the amplitude of the indication from the other notch shall be recorded. Distance corresponding to the wall or plate thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. All testing since previous calibration shall be repeated whenever the calibration gain changes by more than 10%.

4.2. Welding Equipment and Procedure

4.2.1. Before welding actual columbium alloy parts and components, the welding equipment and procedures shall be qualified by welding columbium - 1% zirconium alloy and testing as follows.

4.2.2. A butt-welded joint will be made in the vacuum purged welding chamber in the columbium - 1% zirconium alloy. The material thickness shall be approximately the same as the parts to be welded with a maximum thickness requirement of 1/8 inch. Welding filler material shall be sheared from the parent alloy.

4.2.3. Four strips shall be cut from the welded joint with the weld running transverse to the length. The minimum width of three strips shall be four times the base metal thickness or 1/2 inch, whichever is smaller.

4.2.4. Three welded strips shall be bend tested using a 75-degree V-block and mating V-punch with a radius three times the base metal thickness. Each weld is to be placed face down and centered under the punch radius. The reinforcement on the back side shall be removed before testing.

4.2.5. All three test strips shall withstand a transverse bend through 90 degrees without cracking. The weld and heat-affected zones shall be visually examined for cracks at a magnification of 10X. All three specimens must pass the bend test requirements.

4.2.6. Chemical analyses of the parent metal and weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the fourth strip. Gas analyses shall be by vacuum fusion techniques and the carbon shall be determined by the combustion method. These chemical analyses shall demonstrate that the degree of environmental contamination did not exceed the following limits: increase in oxygen content--less than 50 ppm; increase in nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the analytical results.

Carbon	±	10 ppm
Oxygen	±	30 ppm
Nitrogen	±	30 ppm
Hydrogen	±	2 ppm

4.3. Postweld Annealing Procedures and Equipment

4.3.1. Before annealing welded joints in components, the annealing procedures and equipment shall be qualified as described to insure that the weldments are not contaminated and embrittled during the operation.

4.3.2. A sample welded joint simulating the actual weldment shall be welded using qualified equipment and procedures. The weld shall be heat-treated at 2200°F, plus or minus 25°F, for one hour in a vacuum using the procedures and equipment that will be used for production welds. If localized heat-treating equipment is used, the temperature of the weld shall be measured by a Pt/Pt-Rh thermocouple which is resistance spot-tack to the part.

4.3.3. Three strips shall be cut from the joint. Two shall have a width four times the base metal thickness but not less than 1/2 inch. The third one shall be sufficiently wide to obtain a weld metal, chemical analysis sample. The weld shall run transverse to the length of the strip at midpoint.

4.3.4. One transverse weld bend strip shall be bend tested as annealed, and the other shall be bend tested after aging at 1500°F for fifty hours in a vacuum furnace at a pressure of 1×10^{-5} torr maximum. The bend test procedures shall be those described in paragraph 4.2. Both bend strips must pass the test requirements.

4.3.5. Chemical analysis of the weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the third strip. These analyses shall demonstrate that the degree of environmental contamination did not exceed the limits set forth in paragraph 4.2.

4.3.6. Qualifying both welding and annealing procedures and equipment simultaneously is permissible. The test results must meet the requirements of paragraphs 4.2 and 4.3.

4.4. Reports. A report shall be submitted with the finished parts and shall include all vacuum, temperature, and inert gas purity measurements applicable to welding and heating, the X-rays of the weld joints, and/or when required, location and approximate depth of ultrasonic indications found. Ultrasonic reports shall also include the ultrasonic equipment and transducer used and calibration data. The above reports and all accompanying X-rays, charts, etc., shall reference the drawing number, part number, and serial number of each part represented and, when multiple welds on a part are made in separate environments, the particular weld to which the information is applicable.

Note: These reports shall be retained by quality assurance, should subsequent weld evaluation be desired.

03-0010-00-B
SPPS-11A
26 July 1963

SPECIFICATION

CHEMICAL CLEANING OF COLUMBIUM AND COLUMBIUM ALLOY PRODUCTS

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

CHEMICAL CLEANING OF COLUMBIUM AND COLUMBIUM ALLOY PRODUCTS

1. SCOPE

1.1. This specification establishes the procedures for the chemical cleaning of columbium and columbium alloy products.

1.2. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements included on the drawings supersede this specification.

2. **APPLICABLE DOCUMENTS.** None.

3. **MATERIALS.** Materials required under this specification are hydrofluoric acid (48% solution) and nitric acid (70% solution).

4. **EQUIPMENT.** Necessary equipment includes:

(1) Acid tank resistant to hydrofluoric/nitric acid mixtures
(2) Wash tanks
(3) Cleaning racks resistant to hydrofluoric/nitric acid mixtures
(4) Safety equipment and clothing.

5. REQUIREMENTS

5.1. The solution required for the proper chemical cleaning of columbium materials is EXCEEDINGLY HAZARDOUS. Extreme caution, which shall be exercised in the preparation and use of the solution, requires that

(1) Protective clothing, as specified by the plant safety specialist, shall be mandatory.

(2) Proper warning signs shall be posted.

(3) Access to the facility shall be to authorized personnel only.

(4) An emergency center shall be established and shall store the proper acid antidotes and the equipment and directions for their usage, as specified and demonstrated by the plant industrial hygienist.

5.2. All surfaces of the columbium or columbium alloy products shall be free of grit, metallic chips, grease, oil, fingerprints or any organic markings.

5.3. The cleaned and dried products shall be transferred immediately to polyethylene bags. All hollow parts shall be sealed with suitable non-adherent plugs. Cleaned products shall be handled using clean nylon gloves.

6. PROCEDURE

6.1. The composition of the solution shall be:

Hydrofluoric acid (48% solution)	- 20% by volume
Nitric acid (70% solution)	- 20% by volume
Water	- 60% by volume

The quantity of cleaning solution shall be calculated so that ENTIRE container units of the acids are consumed in the preparation. Acids shall always be added to the water, NEVER the reverse. The prepared solution shall be thoroughly agitated to insure complete mixing and maintained at 100-125°F during pickling.

6.2. The cleaning rack shall be loaded so that the cleaning solution shall have free access to all surfaces of the charge. To assure uniform cleaning, rack loads shall consist of products with as similar dimensions as possible.

6.3. To maintain a minimum storage time for cleaned stock, the chemical cleaning operation shall be scheduled just before any fabrication process. The racked columbium products shall be totally immersed in the solution and agitated by gently raising and lowering the rack beneath the surface of the solution. If hollows (tubes, pipes, cylinders, etc.) are being cleaned, the agitation shall be achieved by moving the rack back and forth in the solution to insure a continuous supply of fresh solution to the internal surfaces. Depending on the mass and configuration of the rack loads, the immersion time will vary. The amount of stock removal required shall be specified by the requestor.

6.4. After the immersion, the rack shall be raised above the solution, tilted slightly and allowed to drain for 2-3 minutes. Then the rack shall be immersed immediately in clean, flowing water and agitated by a back and forth movement of the rack. Immersion in the water rinse shall be no less than thirty minutes with the water at a temperature above

125°F. A second rinse shall be conducted for a minimum of five minutes in distilled or de-ionized water at a temperature above 125°F. The rack load shall be allowed to drip-dry or shall be dried by blowing dry, clean, oil-free air over and through the load.

7. RECORDS. The part name, date of cleaning, and general observations shall be recorded as specified by the project manager or his representative.

03-0012-00-A
SPPS-14
24 January 1964
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SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM ALLOY BY THE ELECTRON BEAM PROCESS

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

WELDING OF COLUMBIUM - 1% ZIRCONIUM ALLOY BY THE ELECTRON BEAM PROCESS

1. SCOPE

1.1. Scope. This specification establishes the procedures, process substantiation, and quality requirements for electron beam welding of columbium-1% zirconium alloy. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements on the drawings supersede this specification.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents.

AMS 2635 (15 August 1955)	Radiographic Inspection
SPPS-11A (26 July 1963)	Chemical Cleaning of Columbium and Columbium Alloy Products

3. REQUIREMENTS.

3.1. Equipment

3.1.1. A high voltage electron beam welder, capable of 150 kilovolts at 20 milliamperes, shall be used.

3.1.2. The welding chamber shall be capable of evacuation to less than 5×10^{-5} torr and shall be leak-tight as determined by mass-spectrometer leak checks at a sensitivity of 5×10^{-10} std. cc/sec.

3.2. Cleaning

3.2.1. The parts to be joined shall be cleaned in accordance with specification SPPS-11A of 26 July 1963, for material thicknesses of 0.015 inch or more. Material thicknesses less than 0.015 inch shall be cleaned with reagent grade acetone.

3.3. Fixtures

3.3.1. The fixtures shall hold the parts in proper alignment and shall maintain the gap tolerance listed in Table I.

TABLE I

ALLOWABLE GAP TOLERANCES

Material Thickness (Inch)	Maximum Allowable Gap (Inch)
0.000 to and including 0.010	metal-to-metal contact
0.010 to and including 0.060	0.002
0.60 to and including 0.125	0.003
0.125 and greater	0.005

3.3.2. The fixtures shall be made from materials that do not corrode or rust in air. The fixture components that contact the columbium parts near the weld joint shall be made of molybdenum, tungsten or columbium.

3.3.3. The fixture shall be clean and free of surface contamination.

3.4. Welding Procedures

3.4.1. Welding of components shall be done using equipment and procedures which conform to Section 4. After loading of components and fixtures, the chamber shall be evacuated to 1×10^{-4} torr or better before the filament current is started and to 5×10^{-5} torr or better before the electron beam is activated.

3.4.2. Prior to welding the beam shall be sharply focused upon a tungsten target whose surface is at the same level as the surface of the material to be welded. This procedure may be modified in special cases where surface focusing would not allow complete fusion of the weld joint.

3.4.3. Prior to the first piece welded and subsequent to the last piece welded in each sequence of chamber evacuations, weld bend specimens shall be prepared. Each specimen shall be from 0.04 to 0.08 inch thick, 1 inch wide (minimum), and 2 inches long (minimum) with a longitudinal weld bead weighing 2.5 grams (minimum).

The above specimens shall be tested in accordance with paragraph 4.2. Specimens and samples shall be tagged with the drawing number, part number, and serial number of each part represented by the samples and specimens. These specimens shall be retained by quality assurance.

3.4.4. The completed weldment shall be left in the chamber until sufficient time has elapsed to allow complete cooling of the component to room temperature.

3.4.5. In repair welding, procedures shall be provided by the General Electric project engineer. The component shall then be welded, according to the requirements of this specification, and re-inspected.

3.5. Postweld Heat Treatment

3.5.1. The weld and adjacent heat-affected zone shall be postweld annealed at 2200°F, plus or minus 25°F, for one hour in a vacuum of 1×10^{-5} torr or better. The annealing may be local at the weld joint or the entire weldment may be annealed in a vacuum furnace. The procedures and equipment used shall conform to paragraph 4.3.

3.5.2. Localized heating shall be done using a heater having only refractory metals in the hot zone. Dense vitrified alumina (99.2% purity) shall be used as the heater electrical insulation in the cold zone of furnace. The temperature of the weld joint shall be measured by a Pt/Pt-Rh thermocouple that is resistance spot-tacked adjacent to the weld metal. After heat treatment the thermocouple spot-tack shall be filed off.

3.5.3. A columbium-1% zirconium control specimen of 0.040 to 0.080-inch thick sheet, weighing 5 grams or more, shall accompany the welded components or parts through postweld vacuum heat treatment.

The above specimens and a similar, but unheated, specimen of the control material shall be submitted with the finished parts. Specimens shall be tagged with the drawing number, part number, and serial number of each part represented by the specimens.

Note: These specimens shall be retained by quality assurance for subsequent chemical analysis, should an investigation of vacuum environment contamination be desired.

3.6. Defect Limits. The completed weldment shall be examined for defects visually and by ultrasonic or X-ray techniques, or both. Welds which do not meet the following quality requirements of this specification are unacceptable.

3.6.1. Visual and Radiographic. Welds shall be reasonably smooth and uniform in appearance and free of the following defects by visual and radiographic inspection:

1. Cracks in the weld and adjacent base metal
2. Crater checks or cracks
3. Surface inclusions and pores
4. Undercutting of the base metal
5. Depression of the weld below the base metal
6. Unfilled weld craters or stops
7. Damage to the weld and adjacent base metal by contamination from foreign materials or metals
8. Lack of fusion of the weld deposit to the base metal
9. Incomplete fusion and penetration to the root of the joint (back side of butt and groove welds)
10. Porosity in the weld metal

3.6.2. Ultrasonic. Rejection shall be by any indication which exceeds the amplitude of the respective calibration indication. For tubing, inside diameter (or under side) defects shall be compared to the indication from the notch on the inside diameter (or under side), and outside diameter (or upper side) defects shall be compared to the indication from the notch on the outside diameter (or upper side). Indications less than half the thickness from the surface or less than 0.150 inch from the surface, whichever is smaller, shall be compared to the upper side (or outside diameter) calibration indication. Defects more than half the distance from the incident surface or more than 0.150 inch from the surface shall be compared to the indication from the inside diameter (or under surface) notch.

4. QUALITY ASSURANCE PROVISIONS

4.1. Weld Inspection Procedures. All welded joints shall be inspected for conformance to the quality requirements visually and by X-ray or ultrasonic techniques, or both.

4.1.1. X-ray Inspection

4.1.1.1. X-ray inspection shall be done according to AMS 2635, "Radiographic Inspection". Fine grain film shall be used for maximum sensitivity. The penetrameter thickness and hole sizes shall be based on the smaller section thickness being joined. The radiographic technique shall be considered satisfactory if the smallest hole in the penetrameter can be clearly distinguished on the radiograph.

4.1.1.2. The radiographs shall be taken in positions that will best delineate lack of weld penetration and fusion. Wherever possible, the film shall be placed on the back side of the weld joint.

4.1.1.3. When the back side of the weld is not accessible and the radiation must pass through a double thickness, only that portion of the weld next to the film shall be considered.

4.1.1.4. Radiographic interpretation for weld quality shall be done by the General Electric project engineer or his representative.

4.1.2. Ultrasonic Inspection

4.1.2.1. When required by the drawing, welded joints shall be ultrasonically rather than radiographically inspected.

4.1.2.2. Ultrasonic inspection shall be by the immersed technique at 5 mc or higher frequency using focused transducers. Shear wave technique shall be used with the beam traverse perpendicular to the weld centerline. Inspection shall be from both directions toward the weld. For welds in tubing, a transducer focused to the diameter of the tubing shall be used with a maximum axial length on the transducer of 0.5 inch. For welds in plate, a spherically focused transducer or a cylindrically focused transducer of less than 1.5 inch diameter focus and 0.5 inch or less long shall be used with its axis parallel to the weld centerline.

4.1.2.3. Calibration shall be on notches cut in a tube or plate of similar material, preferably near a weld of similar nature. These circumferential notches shall be: depth, 3% of the original wall or plate thickness; width, not more than the depth; length, at least one beam width. One notch shall be placed on the inside diameter or under side near the weld; the other notch shall be placed on the outside diameter or upper side near the weld but on the opposite side of the weld or at least two beam widths away (at the same distance from the weld) if on the same side of the weld. Focusing shall be done to maximize the indication from the notch on the under side or inside diameter. After focusing is completed, whichever indication is larger shall be set at 80% and the amplitude of the indication from the other notch shall be recorded. Distance corresponding to the wall or plate thickness shall be marked on the oscilloscope. Focal distance to the part to be inspected shall be set to that used for the calibration piece before beginning inspection. Calibration shall be done both before and after the inspection or at the beginning and end of each work shift. All testing since previous calibration shall be repeated whenever the calibration gain changes by more than 10%.

4.2. Welding Equipment and Procedure

4.2.1. Before welding actual columbium alloy parts and components, the welding equipment and procedures shall be qualified by welding columbium-1% zirconium alloy and testing as follows.

4.2.2. A full penetration bead on sheet weld shall be made in the electron beam facility in the columbium-1% zirconium alloy. The material thickness shall be approximately the same as the parts to be welded with a maximum thickness requirement of 1/8 inch.

4.2.3. Four strips shall be cut from the welded joint with the weld running transverse to the length. The minimum width of three strips shall be four times the base metal thickness or 1/2 inch, whichever is larger,

4.2.4. Three welded strips shall be bend tested using a 75-degree V-block and mating V-punch with a radius three times the base metal thickness. Each weld is to be placed face down and centered under the punch radius. The reinforcement on the back side shall be removed before testing,

4.2.5. All three test strips shall withstand a transverse bend through 90 degrees without cracking. The weld and heat-affected zones shall be visually examined for cracks at a magnification of 10X. All three specimens must pass the bend test requirements.

4.2.6. Chemical analyses of the parent metal and weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the fourth strip. Gas analyses shall be by vacuum fusion techniques and the carbon shall be determined by the combustion method. These chemical analyses shall demonstrate that the degree of environmental contamination did not exceed the following limits: increase in oxygen content--less than 50 ppm; increase in nitrogen content--less than 50 ppm; increase in hydrogen content--less than 5 ppm; increase in carbon content--less than 10 ppm. The following limits shall apply to the analytical results.

Carbon	±	10 ppm
Oxygen	±	30 ppm
Nitrogen	±	30 ppm
Hydrogen	±	2 ppm

4.3. Postweld Annealing Procedures and Equipment

4.3.1. Before annealing welded joints in components, the annealing procedures and equipment shall be qualified as described to insure that the weldments are not contaminated and embrittled during the operation.

4.3.2. A sample welded joint simulating the actual weldment shall be welded using qualified equipment and procedures. The weld shall be heat-treated at 2200°F, plus or minus 25°F, for one hour in a vacuum using the procedures and equipment that will be used for production welds. If localized heat-treating equipment is used, the temperature of the weld shall be measured by a Pt/Pt-Rh thermocouple which is resistance spot-tacked to the part.

4.3.3. Three strips shall be cut from the joint. Two shall have a width four times the base metal thickness but not less than 1/2 inch. The third one shall be sufficiently wide to obtain a weld metal, chemical analysis sample. The weld shall run transverse to the length of the strip at midpoint.

4.3.4. One transverse weld bend strip shall be bend tested as annealed, and the other shall be bend tested after aging at 1500°F for fifty hours in a vacuum furnace at a pressure of 1×10^{5} torr maximum. The bend test procedures shall be those described in paragraph 4.2. Both bend strips must pass the test requirements.

4.3.5. Chemical analysis of the weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the third strip. These analyses shall demonstrate that the degree of environmental contamination did not exceed the limits set forth in paragraph 4.2.

4.3.6. Qualifying both welding and annealing procedures and equipment simultaneously is permissible. The test results must meet the requirements of paragraphs 4.2. and 4.3.

4.4. Reports. A report shall be submitted with the finished parts and shall include all vacuum, temperature, and inert gas purity measurements applicable to welding and heating, the X-rays of the weld joints, and/or when required, location and approximate depth of ultrasonic indications found. Ultrasonic reports shall also include the ultrasonic equipment and transducer used and calibration data. The above reports and all accompanying X-rays, charts, etc. shall reference the drawing number, part number, and serial number of each part represented and, when multiple welds on a part are made in separate environments, the particular weld to which the information is applicable.

Note: These reports shall be retained by quality assurance, should subsequent weld evaluation be desired.

SPECIFICATION

MASS SPECTROMETRIC LEAK DETECTION USING HELIUM

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

1. SCOPE

1.1. Scope. This specification describes the helium mass spectrometric method and quality requirements for leak detection in various types of containment equipment. Reference to this specification shall be made on engineering drawings whenever applicable, however, information or requirements on such drawings supercede this specification.

1.2. For other methods of leak detection, see MIL-STD 271C (Ships).

2. APPLICABLE DOCUMENTS

2.1. Government Documents

MIL-STD 271C (Ships) 1 October 1963	Non-Destructive Testing

2.2. Non-Government Documents. None

3. REQUIREMENTS

3.1. Equipment and Materials

3.1.1. Leak Detector. Final acceptance testing shall be accomplished using a mass spectrometric helium leak detector. Appropriate conversion factors are presented in Table I.

Table I

Leakage Units (Flow)

To Convert From	To	Multiply By
Std. cc/sec.	Micron-liters/sec.	7.6×10^{2}
	Micron cubic feet/hr.	9.66×10^{4}
	Torr-liters/sec.	7.60×10^{-1}
Micron-liters/sec.	Std. cc/sec.	1.32×10^{-3}
	Micron cubic feet/hr.	1.27×10^{2}
	Torr-liters/sec.	1.00×10^{-3}
Micron cubic feet/hr.	Std. cc/sec.	1.04×10^{-5}
	Micron-liters/sec.	7.87×10^{-3}
	Torr-liters/sec.	7.87×10^{-6}
Torr-liters/sec.	Std. cc/sec.	1.32
	Micron-liters/sec.	1.00×10^{3}
	Micron cubic feet/hr.	1.27×10^{5}

3.1.2. Sensitivity of the Instrument

3.1.2.1. This test, which serves to calibrate the instrument only, shall be performed with a calibrated leak attached directly to the leak detector with helium applied at atmospheric pressure. Recalibration will be performed upon change in setup or after moving the leak detector from one place to another. The sensitivity of the instrument shall be determined as follows:

3.1.2.2. Prior to leak detection (during calibration) random meter movement (oscillations) shall be less than 10% of the full scale value at the maximum sensitivity (the times 1 scale or equivalent) of the instrument.

3.1.2.3. During the calibration, the length of time for the output meter to go from zero to full scale on any sensitivity range shall not exceed one second.

3.1.2.4. The operator will assure himself that the output meter has risen to a maximum, constant level to be sure that equilibrium has been reached in the system.

3.1.3. Calculation of Sensitivity. The sensitivity shall be calculated as follows:

$$S = \frac{L}{CHD}$$

where S = Sensitivity

L = Calibrated leak (micron cubic feet per hour or standard cubic centimeters per second of air)

CHD = Deflection caused by helium during calibration (calibration helium deflection)

3.1.4. Helium. The helium used shall be a pure commerical grade which is not oil pumped.

3.2. Preliminary Conditions

3.2.1. Stages for Helium Leak Testing. When any part of a test piece cannot be reached for proper repair after final assembly, leak testing shall be performed during appropriate predetermined stages of fabrication.

3.2.2.1. One or both of the following tests may be conducted on the test piece during or after assembly to locate large leaks in order to reduce time for leak testing with the helium mass spectrometer.

3.2.2.2. The test piece shall be subjected to an internal gas pressure (air or inert gas, <u>not helium</u>) not to exceed the design pressure. All connections and welded joints shall then be examined for leaks by application of a soap solution or its equivalent. The joint or connection will be observed for the growth in size of bubbles or the obvious removal of the soap solution by the force of the gas through the leak.

3.2.2.3. Vacuum leak testing shall be performed with the test piece, including all welds and joints, unpainted and free of any type of coating or filler. The test piece shall be evacuated with the leak detector vacuum system to less than 300 microns of mercury. Where a pressure greater than 300 microns exists, an attempt will be made to reduce the pressure by tightening all connections and coating welds with a compound such as Apiezon "Q" putty. The location of large leaks will be noted when a large decrease in pressure occurs. Careful examination of the suspected area will usually disclose the defect, and repair procedures can be implemented. When a pressure of 300 microns or less can be attained, helium leak testing can be initiated.

3.2.3. <u>Cleanliness</u>. Whenever possible, the test piece shall be clean and free of water vapor, oil, grease and other contaminants which interfere with the mass spectrometer leak test.

3.2.4. <u>Adsorbed Gases</u>. Inert gases or inert gas mixtures used during fabrication should not contain helium. When fabrication procedures make it necessary to use helium the test piece shall be evacuated below a pressure of 50 microns. Whenever possible, test pieces will be warmed (150-250°F or more) to remove adsorbed gases to reduce the background within the leak detector to an insignificant level. On a relatively leak-free system, an insignificant background is indicated by an output reading on the most sensitive scale which is steady to the extent that the needle oscillations are less than 10% of the full scale deflection. In less ideal cases, where the output is high and/or unsteady due to previous exposure to helium, high outgassing rates or relatively large leaks, leaks may be detected; but the significance of a particular reading must be based on the interpretation of the operator.

3.2.5. Openings in the Test Piece. All unused openings in the test piece shall be sealed leak-tight. All leaks around these closures shall be sealed, using sealing wax, cements or other suitable material which can be readily and completely removed after testing.

3.2.6. Vacuum. The test piece shall be evacuated to 50 microns or less as determined by a thermocouple gauge, or its equivalent, which shall be placed in the vacuum line immediately adjacent to the test piece. If, after all preliminary testing, with all openings leak-tight, a system can be evacuated to only 300 microns, the leak detector may then be used with the following technique. The operator can determine if the instrument has enough sensitivity during leak checking to detect the leak. When the leak is detected and repaired, the test piece shall then be evacuated to 50 microns or less and rechecked, using the helium leak detector as described in paragraph 3.5.

3.3. Frequency of Calibration. The leak detector must be calibrated at the beginning and completion of any leak test, at least, and more frequently if the leak testing is prolonged.

3.4. Calculation of Actual Leak Rate. Leak rates shall be calculated as follows:

LR = S x THD

where LR = Leak rate (std. cc/sec. or micron cubic feet/hour air)

S = Sensitivity

THD = Deflection caused by helium during leak checking (test helium deflection)

This calculation applies only when the leak is exposed to helium at one atmosphere pressure and after the indicated level indication has risen to a maximum, constant value. In this specification it applies only to the Envelope Method described below.

3.5. Helium Leak Testing Methods

3.5.1. Envelope Method. The envelope method of helium leak detection shall be used to indicate the total leakage of the item (including portions of test pieces) after all large leaks have been repaired as indicated by an ability to evacuate the test piece to a pressure lower than 50 microns. The procedure is as follows:

a. Interconnect the calibrated leak detector with the item under test.

b. Evacuate to 50 microns or less as measured by a thermocouple gauge or its equivalent placed in the evacuation line adjacent to the test piece

c. Envelop the entire test piece or portion thereof with a suitable container such as a plastic bag.

d. Fill the space between the outside of the item under test and the container with helium at, or slightly above, atmospheric pressure.

e. With the inlet valve to the detector in a wide open position, the pumping speed of the detector reduced to a minimum, and no auxiliary pumping on the test piece, the leakage into the detector is measured on the output meter. This measurement of leakage is valid only after the deflection has risen to a steady, constant value.

3.5.2. Probe Method. When the actual leak rate exceeds the permissible value or when an over-all probe test is specified, all welds or other suspected areas shall be inspected by means of a fine jet of helium on one side of the weld or other joint and the leak detector on the other. After repair of all defects located by this method, the actual leak rate will be evaluated, if required, using the Envelope Method (paragraph 3.5.1.).

3.5.3. Sniffer Method. Where configuration or access makes probe or envelope testing impossible, the system under test will be pressurized with helium to a point within design specifications. All welds or suspected areas shall be inspected by the use of an evacuated probe which will draw helium from a leak into the leak detector. All leaks detected by this method must be repaired since it is not possible to measure the size of the leak using this method.

3.6. Allowable Leakage Rates

3.6.1. Categories. Two categories of acceptable leakage rates are specified for various applications: critical and non-critical.

3.6.2. Critical Applications. In critical applications a maximum, total leakage rate of 5.0×10^{-10} std. cc/sec. of air is specified unless otherwise noted on engineering drawings or directed by the design engineer.

3.6.3. Non-Critical Applications. When non-critical applications are noted on engineering drawings, the maximum, total, allowable leak rate will be 5.0×10^{-8} std. cc/sec. of air.

4. QUALITY ASSURANCE PROVISIONS

4.1. Certification. After completion of a helium leak test on a piece of test equipment or component, the leak detector operator will prepare a Helium Leak Test Process Control Record (Form SP1067) in duplicate. He will send the original to the person requesting the leak test and will retain the duplicate for the Helium Leak Test Record notebook.

5. PREPARATION FOR DELIVERY. Not applicable

6. NOTES. None

03-0014-00-B
13 September 1965
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SPECIFICATION

WELDING OF AUSTENITIC STAINLESS STEELS

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

1. SCOPE

1.1. Scope. This specification covers the requirements for welding austenitic stainless steels in the fabrication of austenitic stainless steel components, facilities, and test devices.

2. APPLICABLE DOCUMENTS

2.1. Government Documents

MIL-STD-271C (Ships) (1 October 1963)	Non-Destructive Testing Requirements for Metals

2.2. Non-Government Documents

ASME Boiler and Pressure Vessel Code, 1962 and Addenda	
ASTM A371-62T (1962)	Tentative Specification for Corrosion-Resisting Chromium and Chromium-Nickel Steel Welding Rods and Bare Electrodes (AWS A5.9-62T)
ASTM A298-62T (1962)	Tentative Specification for Corrosion-Resisting Chromium and Chromium-Nickel Steel Covered Electrodes (AWS A5.4-62T)
ASTM B297-55T (1955)	Tentative Specifications for Tungsten Arc-Welding Electrodes (AWS A5.12-55T)
ASTM E165-63 (1963)	Standard Methods for Liquid Penetrant Inspection
ASTM E142-64 (1964)	Controlling Quality of Radiographic Testing
ASTM E94-62T (1962)	Tentative Recommended Practice for Radiographic Testing
AWS A2.0-58 (1958)	Standard Welding Symbols (American Welding Society)
AWS A3.0-61 (1961)	AWS Definitions-Welding and Cutting (American Welding Society)

3. REQUIREMENTS

3.1. Operator Qualification. When the components, facilities, or piping are to be fabricated to a specific code, standard, or specification, the welders shall be qualified for proficiency as required by the applicable documents. The applicable documents shall be specified on the engineering drawings, specifications, or purchase order. When no code, standard, or specification is specified, the components shall be fabricated by welders who are proficient in the welding of austenitic stainless steels by the applicable welding process.

3.2. Welding Processes and Equipment

3.2.1. Welding Processes. The welding process used shall be that designated for the joint on the engineering drawing by welding symbols. The process designations are:

P8G - Gas tungsten arc
P8J - Gas metal arc
P8B - Shielded metal arc

3.2.2. Gas Tungsten Arc. Welding by this process, manual or automatic, shall be done using direct current, straight polarity. The torch gas cup shall be of adequate size to provide optimum gas coverage to the weld zone. Tungsten electrodes shall conform to ASTM B297-55T and shall be Class EWTh-2.

3.2.3. Gas Metal Arc. The welding equipment shall be in good condition and free of water and gas leaks. The gas shielding cup shall be of adequate size to provide optimum gas coverage to the weld zone.

3.2.4. Atmospheres. For gas tungsten arc welding, argon of 99.95% minimum purity shall be used for both torch shielding and weld joint backup protection. For gas metal arc welding, argon-1% oxygen shall be used through the welding head and 99.95% purity argon shall be used to protect the back side of the weld.

3.2.5. Welding Filler Wires and Electrodes

3.2.5.1. The welding filler wire and/or electrode shall be that specified on the engineering drawing, part specification, or welding procedure specifications. Root pass inserts, if used, shall be manufactured from material meeting the specifications for the welding filler metal.

3.2.5.2. Bare welding wires for P8G and P8J welding processes shall conform to ASTM A371-62T. Covered welding electrodes for the P8B process shall conform to ASTM A298-62T.

3.2.5.3. Coated welding electrodes used for shielded metal arc welding shall be purchased in commercial moisture-proof containers. Electrodes that have been exposed to ambient atmosphere for more than eight hours shall be baked at 350°F for two hours prior to use to drive off absorbed water vapor in the coating.

3.2.5.4. When the welding wire or electrode is not specified as described in paragraph 3.2.5.1., it shall be selected from Table I for the alloys to be joined.

3.3. Fabrication Techniques

3.3.1. Gas Shielding

TABLE I

AUSTENITIC STAINLESS STEEL WELDING FILLER MATERIALS

Alloys to be welded	302 304 308	304L	309	310	316	316L	321
302, 304, 308	ER308 E308	ER308L E308L	ER308 E308	ER308 E308	ER316 E316	ER316L E316L	ER316 E316
304L		ER308L E308L	ER308L E308L	ER308L E308L	ER316L E316L	ER316L E316L	ER316L E316L
309			ER309 E309	ER309 E309	ER316 E316	ER316L E316L	ER316 E316
310				ER310 E310	ER316 E316	ER316L E316L	ER316 E316
316					ER316 E316	ER316L E316L	ER316 E316
316L						ER316L E316L	ER316L E316L
321							ER321

Note: "ER" designates bare wire for P8G or P8J welding processes. "E" designates coated electrodes for P8B welding process.

3.3.1.1. Adequate shielding against air drafts shall be provided for all welding operations to avoid disturbance of the torch gas shielding and/or the welding arc.

3.3.1.2. The back side of the weld joint shall be protected by argon shielding in a manner that will protect the joint against contamination and oxidation.

3.3.1.3. All weld joints in pipe, tubing, tanks, and containers shall have the back side of the weld protected by argon. The interior of the pipe or container shall be purged of air at the joint by sealing off the interior on both sides of the joint or both ends of the pipe or container. No welding shall be done until the interior has been purged by a volume of argon equal to five times its volume. Systems already pressurized with argon need not be purged.

3.3.2. Joint Preparations

3.3.2.1. The edges of the parts shall be grooved for welding by machining, grinding, or filing as shown on the engineering drawing or specifications for the particular joint design and thickness.

3.3.2.2. When the surfaces to be welded are ground, the grinding wheel and techniques used shall not leave imbedded grit particles in the stainless steel.

3.3.2.3. The edges and surfaces to be joined and the adjacent base metal (about 2" on each side of the joint) shall be cleaned of all oil, grease, dirt and other foreign matter. The areas shall also be free of surface oxides and scale. No residual cleaning compounds shall be left on the surfaces.

3.3.2.4. Before applying weld metal on the second side to be welded, the root of double welded joints shall be prepared by machining, grinding, or filing to sound metal, free of cracks, laps, seams, and craters. The root of the weld shall be liquid penetrant inspected for defects. All inspection residue shall be removed before starting to weld.

3.3.3. Joint Positioning

3.3.3.1. Fixturing. The joints to be welded shall be positioned to provide proper alignment, match of parts, and root opening.

3.3.3.2. Alignment. The joint edges shall not be misaligned more than 20% of the thinner section being joined, or 1/16", whichever is less. When abutted ends of unequal internal diameters exceed the allowable misalignment, the part with the smaller inner diameter shall be machined to match that of the larger diameter. The internal taper shall not exceed 1:4.

3.3.3.3. Tack Welds. After the parts are properly positioned, tack welds may be used to maintain alignment during welding. The tack welds shall be cleaned of oxide with a stainless steel wire brush prior to making the first pass. Cracked tack welds shall be removed and retacked. Tack welds shall have complete fusion and penetration to the weld joint root.

3.3.3.4. Weld Inserts. Consumable weld inserts are permissible provided the welding procedures used insure complete consummation of the insert in the root pass. Backing rings shall not be used unless (1) specifically called out on the drawing or specifications, or (2) they are machined away after welding.

3.3.4. Welding

3.3.4.1. Each joint shall be made using accepted welding procedures for the particular joint type, position, and thickness of base metal.

3.3.4.2. All weld metal that will be in contact with liquid metals contained in test apparatus shall be deposited by P8G or P8J process. In multipass welds, the first two passes shall be deposited by the P8G or P8J process. Shielded metal arc welding (P8B) may be used for fill passes of the joints, except for welds joining Type 321 stainless steel. Welds joining Type 321 stainless steel must be made using either P8G or P8J process for all passes. The welds must meet the quality requirements of section 3.4.

3.3.4.3. Each weld pass shall be thoroughly cleaned with a stainless steel wire brush to remove surface oxidation. Weld deposits shall be free of flux, oxide, dirt, welding defects, etc. before welding over them or connecting to them.

3.3.4.4. Preheating shall not be used.

3.3.4.5. Interpass temperature shall not exceed 500°F.

3.3.4.6. Completed welds shall be cleaned and wire brushed for visual inspection.

3.4. Quality Requirements

3.4.1. General. Weld deposits shall be reasonably smooth and uniform in appearance, have complete fusion, and blend smoothly into the base metal. The welded joints shall be free of the following defects by any and all methods of inspection:

1. Cracks of any type or size in the weld and adjacent base metal,
2. Crater checks and cracks,
3. Surface holes,
4. Cold laps in and along the edge of the weld,
5. Overlap of weld metal on the base metal,
6. Undercutting along the edges of the weld or depression of the weld face below the adjacent base metal,
7. Weld craters,
8. Damage to the weld metal by oxidation,
9. Incorrect weld profile and size,
10. Lack of complete (100%) joint penetration in groove welds,
11. Incomplete fusion between weld metal and/or base metal.

3.4.2. Drop Through. Weld metal on the back side of the joint shall not protrude above the base metal surfaces by more than 25% of the base metal thickness or 1/16", whichever is the least.

3.4.3. Groove Weld Reinforcement. Weld reinforcement on the face side of the joint shall not exceed the following:

Base Metal Thickness (inch)	Height of Reinforcement (inch)
Up to 1/2	1/16
Over 1/2 to 1	3/32
Over 1 to 2	1/8

3.4.4. Fillet Weld Contour. The face of a fillet weld shall be at approximately equal angles to the sections it joins, unless specifically noted otherwise on the drawing. The weld face may be slightly convex, flat, or slightly concave. Convex welds shall have a maximum convexity of 0.1 S plus 0.03 inch where S is the average length (inch) of the two legs of the fillet weld.

3.4.5. Radiographic Welds - Porosity and Inclusions

3.4.5.1. The total area of porosity and inclusions, as determined from the radiographic film, shall not exceed 0.03T square inch in any six-inch length of weld, where T is the average thickness of the weld. If the weld joint is less than 6 inches long, the total allowable area of porosity and inclusions is reduced in proportion of weld length, inches/6.

3.4.5.2. The largest dimension of any single indication shall not exceed 0.15T or 3/32 inch, whichever is smaller.

3.4.5.3. In any 2T length of weld, indications of porosity and inclusions may be clustered to a concentration four times ($0.04T^2$) that permitted in paragraph 3.4.5.1. Such indications shall be included in the total area permitted in paragraph 3.4.5.1. including all of the cluster.

3.4.5.4. Any group of aligned indications having a summation of diameters greater than T in a weld length of 24T is unacceptable. If the weld joint is less than 24T long, the allowable summation is reduced in proportion to weld length/24T.

3.4.5.5. Any two indications separated by a distance less than six times the largest dimension of the larger indication is unacceptable.

3.4.6. Leaks in Welds

3.4.6.1. Each welded joint tested by hydrostatic, gas pressure, or pressure rise (vacuum) methods shall show no evidence of leakage.

3.4.6.2. Each welded joint tested using a helium mass spectrometer, shall have a leak rate of less than 5×10^{-10} standard cc/sec of air.

4. QUALITY ASSURANCE PROVISIONS

4.1. Visual Inspections. Visual inspections and measurements of the parts to be joined, joint preparation and alignment, and the welded joint shall be performed as required.

4.2. Radiographic Inspection

4.2.1. Applications. Welded joints shall be radiographed for soundness when radiographic inspection (RT) is called out on the engineering drawing or parts specification. Welds made in existing facilities and components that contain liquid metals at 300°F and above will be radiographed provided the radiographs will adequately delineate the welded joint. Radiographic procedures shall conform to ASTM E94-62T, Tentative Recommended Practices for Radiographic Testing.

4.2.2. Radiographic Quality Control. Radiographic quality control procedures used shall be those described in the specification called out on the engineering drawing or parts specification. If no specification is called out, the procedures described in the specifications listed below shall apply to the applicable types of components. Weld quality requirements are given in Section 3.4.

ASTM E142-64: Test components and equipment

ASME Boiler and Pressure Vessel Code: Facilities installations and components including piping, vessels, tanks, boilers, condensers, heat exchangers, and valves.

4.2.3. Penetrameters. Penetrameters shall be used for all radiographs and the penetrameter image will be employed to determine the radiographic quality. Penetrameter design and material shall be that given in the applicable specification.

4.2.3.1. Penetrameter Placement. Penetrameters shall be employed as required by the applicable specification. Normally, the penetrameter is placed on the source side of the weldment. When it is not practical to place the penetrameter on the section being examined, and in the plane normal to the radiation beam, it may be positioned on a block of radiographically similar material placed as close as possible to the area being radiographed. The block shall be the same thickness as the total weld thickness, and it shall be placed so that the penetrameter is at the same distance from the film as if it were placed on the source side of the weld joint being radiographed.

4.2.3.2. Double-Wall Radiography. When radiographing double-wall welds, such as joints in pipe or tubing, the thickness of the penetrameter employed shall be based upon the total thickness of material between the penetrameter and the film.

4.2.4. Weld Surface Preparation. Accessible surfaces of welds shall be prepared as necessary so that valleys between beads, weld ripples, and other surface irregularities are blended so that radiographic contrast due to surface condition cannot mask or be confused with that of any defect.

4.2.5. Pipe and Tube Joints. Unless otherwise specified on the engineering drawing, parts specification, or purchase order, welds in pipe and tube of 3 inches O.D. and smaller may be radiographed by the double wall technique and the weld zones in each wall viewed for acceptance. A minimum of two radiographs shall be taken 90° to each other. For welds in pipe and tubes having an O.D. larger than 3 inches, only the weld zone closest to the film shall be viewed for acceptance. A minimum of four radiographs shall be taken 90° to each other.

4.2.6. Source to Film Distance. Minimum recommended radiation source to film distance d_o (inches) is that calculated by the following formula:

$$d_o = 2.5\ Ft$$

where F = maximum effective radioisotope source or focal spot dimension, mm.

t = weld thickness, or pipe or tube O.D., inches

Where a gap exists between the weldment and the film holder, the minimum source to film distance should be increased by the ratio of:

$$\frac{t + \text{gap}}{t}$$

4.2.7. Identification of Radiograph Films. A system of positive identification of the film shall be used. The following information shall appear on each radiograph and in the records accompanying each film:

1. Organization making the radiograph.
2. Date of exposure.
3. Identification of (a) the component, facility, or system, (b) the weld joint, and (c) the viewing direction.
4. The radiograph is the original area or a repaired area.

4.2.8. Radiographic Quality Level. Unless otherwise specified on the engineering drawing or parts specification, the radiographic quality level shall be 2% (2+2T) or better.

4.2.9. Interpretation of Weldment Radiographs. The radiographs shall be examined for quality and for unacceptable weld defects described in paragraph 3.4. Final interpretation of radiographed welds for conformance to quality requirements is reserved by the General Electric Company.

4.2.10. Disposition of Radiographs. Radiographs of weldments shall be forwarded to SPPS Quality Control for filing, unless the fabricator is required to retain the radiographs by law or other instructions.

4.3. Liquid Penetrant Inspection

4.3.1. Application. Welds shall be liquid penetrant inspected for soundness when liquid penetrant inspection (PT) is called out on the engineering drawing or parts specification. Welds made in existing facilities and components that contain liquid metals at 300°F and above, and cannot be radiographed in a suitable manner to show weld defects, shall be liquid penetrant inspected.

4.3.2. Specifications. The applicable inspection specification shall be that called out on the engineering drawing or parts specification. If none is called out, the specification ASTM E165-63 shall apply for the particular process.

4.3.3. Surface Condition. As-welded surfaces, following the removal of surface oxides, or flux, shall be considered suitable for inspection without grinding. Shot, sand, grit, and vapor blasting shall not be done on surfaces to be inspected.

4.3.4. Penetrants and developers shall be sulfur free types.

4.3.5. Multiple Pass Welds. When more than one pass of weld metal is required to complete a weld joint, each layer of weld metal shall be inspected for defects. All indications shall be considered as described in paragraph 4,3,6. Prior to further welding, all penetrant and developer shall be cleaned from the joint and adjacent areas using a suitable solvent.

4.3.6. Relevant and Irrelevant Indications. All indications in the weld and along the fusion line between the base metal and weld metal are considered relevant and shall be further evaluated or repaired. The indication shall be explored by removing the surface roughness or other conditions believed to have caused the indication to determine if defects are present. The weld shall be reinspected. Any indications are considered relevant and shall be evaluated with respect to quality requirements of paragraph 3.4.

4.4. Leak Testing

4.4.1. Applications. Weldments shall be checked for leakage as specified on the engineering drawing or parts specification. All welds made in existing components and facilities that will contain liquid metal shall be leak checked.

4.4.2. Specifications. When leak testing using a helium mass spectrometer is required and no testing specification is given, the testing shall be done as described in MIL-STD-271C (Ships).

4.4.3. A final leak check shall be performed on the completed weld assembly using the specified method.

4.5. Surveillance. Representatives of the General Electric Company shall be permitted free access for inspection of welding operations, fabrication, materials, and testing. The vendor shall afford each representative all reasonable facilities to satisfy himself that the fabrication is being furnished in accordance with the specified requirements.

4.6. Rejection of Weldments. Weldments and components not conforming to the specifications shall be rejected.

5. PREPARATION FOR DELIVERY. Not applicable.

6. DEFINITIONS

6.1. Welding Symbols. Interpretation and meaning of welding symbols on engineering drawings and specifications are those in AWS A2.0-58 Standard Welding Symbols, unless specifically delineated otherwise on the drawings and specifications.

6.2 Welding Terminology. Welding terms in this specification are defined in AWS A3.0-61.

SPECIFICATION

ARC WELD GROOVE DESIGNS FOR AUSTENITIC STAINLESS STEELS, L-605, COLUMBIUM, AND TANTALUM ALLOYS

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

GENERAL ELECTRIC COMPANY
RE-ENTRY SYSTEMS DEPARTMENT
SPACE POWER AND PROPULSION SECTION

SPECIFICATION

ARC WELD GROOVE DESIGNS FOR AUSTENITIC STAINLESS STEELS, L-605, COLUMBIUM, AND TANTALUM ALLOYS

1. SCOPE

1.1. Scope. This specification defines the typical groove designs for inert gas shielded tungsten arc welded joints in liquid metal containment components, assemblies and facilities. It is applicable to austenitic stainless steels, L-605, columbium, and tantalum alloys.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None.

2.2. Non-Government Documents. Bulletin No. 214 - Specific Safety Requirements Covering the Installation of Pressure Piping Systems, Industrial Commission of Ohio, 1961. ASME Boiler and Pressure Vessel Code, 1962.

3. REQUIREMENTS

3.1. Joint Designs. The weld groove designs are shown in Figure 1. The groove dimensions for the four materials are tabulated. The groove designs are typical and they are applicable to most weldment designs.

3.2. Applications. The groove designs are to be utilized in the design of liquid metal containment facilities, components, and test devices. When welding procedures development shows the need for special groove designs to obtain the required weld quality, the modified design shall be incorporated on the engineering drawing with approval of SPPS Materials and Processes engineering and design engineering.

3.3. Groove Preparation. The edge preparation shall be machined, using procedures that will not (1) induce flaws in the metal, (2) leave embedded particles or other foreign matter on the groove surfaces, and (3) overheat or otherwise damage the metal metallurgically. After machining, all oil, cutting lubricants, chemicals, dirt, grinding deposits, etc. shall be cleaned off the parts.

3.4. Root Opening. The root openings (G), shown in Figure 1, are maximum values. The actual opening used should be that required to obtain full weld penetration and joint fusion. When the root opening is delineated on the drawing, its value shall be used for manufacture of the components.

3.5 Quality Requirements. The welding grooves shall be within dimensional tolerances shown on the engineering drawings. They shall have reasonably good surfaces consistent with good machining practices. Sharp corners shall be broken a minimum amount for safety only. Correction of machining errors by welding, or any other process of adding material, must be approved by SPPS Materials and Processes engineering.

3.6. Responsibility for Use. The use of the groove designs in this specification, or those delineated on the engineering drawing, does not relieve the manufacturer of components and facilities of the responsibility for producing weld joints to the quality requirements of the applicable welding specification.

4. QUALITY ASSURANCE PROVISIONS

4.1. Inspection. The edge preparations shall be inspected for dimensional requirements as shown in the engineering drawing.

5. PREPARATION FOR DELIVERY. Not applicable.

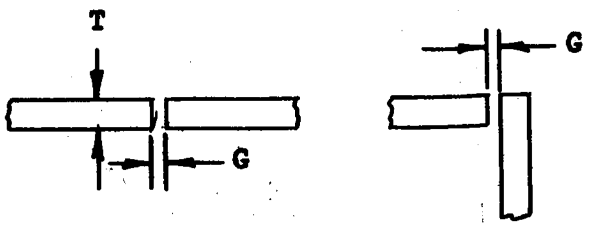
6. DEFINITIONS

6.1. Materials and Processes Engineering. SPPS materials engineer covering the applicable component.

6.2. Design Engineering. SPPS engineer responsible for the design of the applicable component.

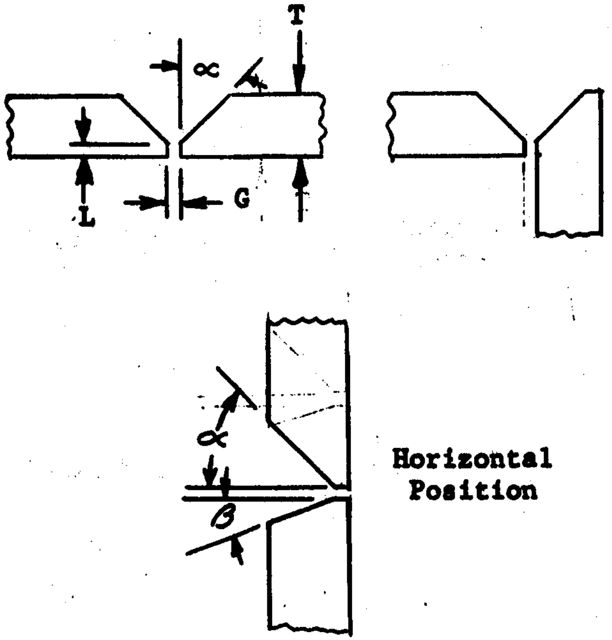
6.3. Applicable Welding Specification. SPPS specification covering the welding of the particular material of which the component is made.

FIGURE 1. WELD JOINT GROOVE DESIGNS



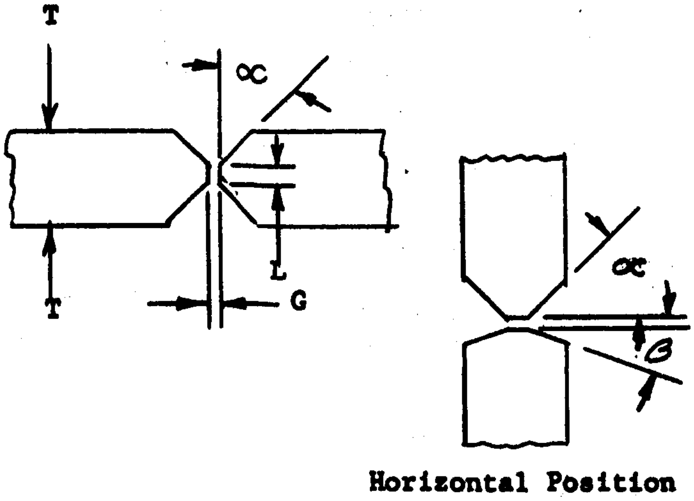
	Dimensions (inch, degree)		
	SS	L-605	Cb, Ta
T max	.13	.095	.06
G max	.06	.05	.03

a. Square Groove (one or both sides)



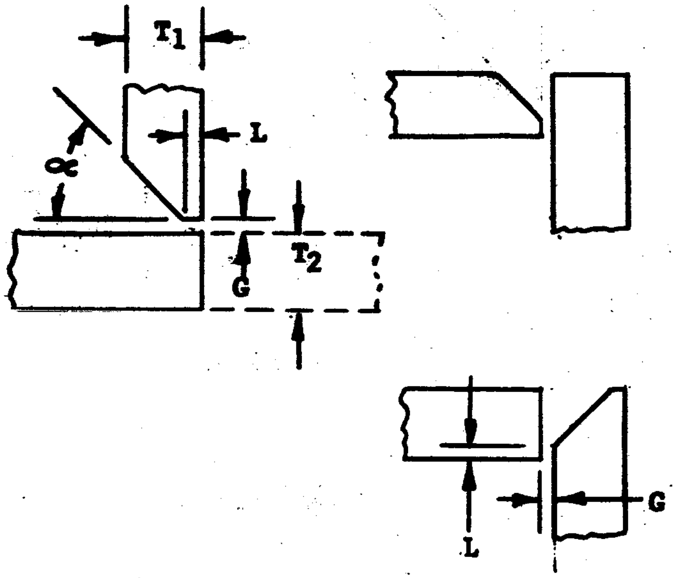
	SS	L-605	Cb, Ta
T min	.09	.09	.06
T max	.50	.28	.19
G max	.06	.06	.04
L	.05-.06	.04-.05	.03-.04
α	40	45	45
β	15	15	20

b. Single Vee (one or both sides)



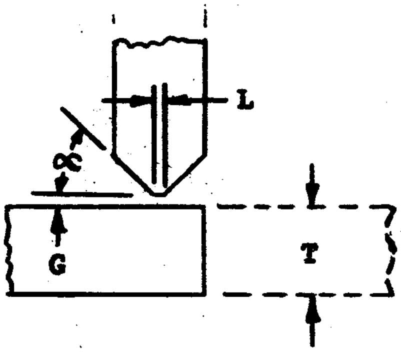
	SS	L-605	Cb, Ta
T min	.38	.25	.19
T max	1.0	.75	.50
G max	.06	.06	.04
L	.05-.07	.05-.06	.04-.05
α	40	45	45
β	15	15	20

c. Double Vee (both sides)



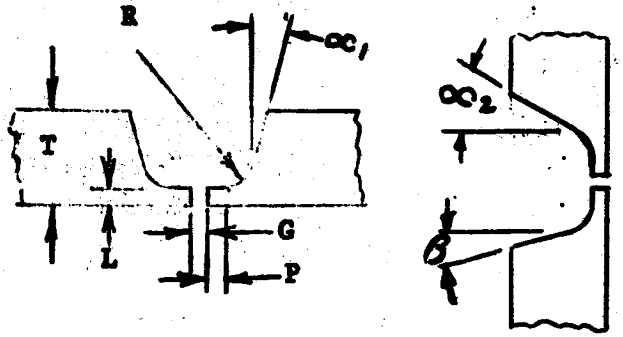
d. Single Bevel (one or both sides)

	Dimensions (inch, degree)		
	SS	L-605	Cb, Ta
T_1 min	.09	.09	.06
T_1 max	.38	.25	.19
T_2 min	.09	.09	.06
G max	.09	.06	.06
L	.04-.05	.04-.05	.03-.04
∝	45	50	50



e. Double Bevel (both sides)

T min	.25	.19	.19
T max	.75	.75	.50
G max	.13	.13	.09
L	.05-.07	.04-.05	.03-.04
∝	45	50	50

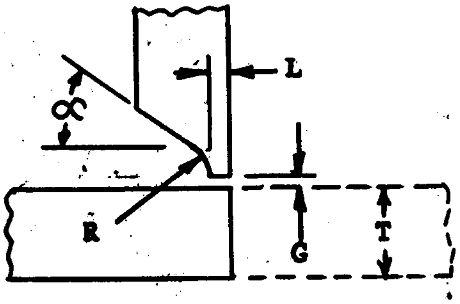


Horizontal Position

f. Single U (one or both sides)

T min	.50	.28	.19
G max	.06	.06	.04
L	.05-.06	.05-.06	.04-.05
P	.09	.06	.05
R	.19	.13	.13
α_1	15°	20°	20°
α_2	30°	30°	30°
β	15°	15°	15°

FIGURE 1. WELD JOINT GROOVE DESIGNS - continued



	Dimensions (inch, degree)		
	SS	L-605	Cb, Ta
T min	.44	.31	.22
G max	.09	.06	.06
L	.05-.06	.05-.06	.04-.05
R	.19	.16	.12
α	40°	40°	40°

g. Single J (one or both sides)

SPECIFICATIONS

ALKALI METAL HANDLING AND CONTROL PROCEDURES

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

1. SCOPE

1.1. Scope. This specification covers all technical phases of the procurement, surveillance, transfer, storage, purification, sampling, analysis, monitoring, usage and handling of alkali metals.

2. APPLICABLE DOCUMENTS

2.1. Government Documents

MIL-STD-271C (Ships) (1 October 1963)	Non-Destructive Testing Requirements for Metals
Interstate Commerce Commission Regulations Applying to Shipping, Part 73-206	

2.2. Non-Government Documents

03-0013-00-A 15 September 1964	Mass Spectrometer Leak Detection Using Helium
03-0014-00-A 1 May 1964	Welding of Austenitic Stainless Steels
01-0030-00-A 21 January 1965	High Purity Lithium Metal
01-0031-00-B 10 May 1965	Reactor Grade Sodium Metal
01-0032-00-B 10 May 1965	Hot Trapped Reactor Grade Sodium Metal
01-0033-00-B 10 May 1965	High Purity Grade Potassium Metal
01-0034-00-B 10 May 1965	Hot Trapped High Purity Grade Potassium Metal
01-0049-00-A Not yet written	High Purity Grade Eutectic NaK
01-0050-00-A Not yet written	Hot Trapped High Purity Grade Eutectic NaK
01-0051-00-A Not yet written	High Purity Grade Rubidium Metal
01-0052-00-A Not yet written	High Purity Grade Cesium Metal
03-0016-00-A 9 August 1964	Stainless Steel Conoseal Tube Unions

3. REQUIREMENTS

3.1. Procurement

3.1.1. Work Authorizations. Work Authorizations (Form GT 3099-A(4-62)) for the purchase of alkali metals will be initiated by personnel responsible for the operation of a given facility or by the alkali metal custodian when requested to write the Work Authorization by operating personnel. When a Work Authorization is initiated for the purchase of an alkali metal, it will be accompanied by an Alkali Metal Surveillance Record (Form SP 1061), an example of which is attached to this specification as Appendix A. The correct use of this record is explained in paragraph 3.2. of this specification.

3.1.2. Procurement Specifications. Alkali metals will be purchased in accordance with the appropriate specification. A listing of these specifications is given in paragraph 2.2. of this specification.

3.1.3. Approvals. No alkali metal will be purchased without the approval of the Alkali Metal Custodian. Approval for purchase will be indicated when both the Materials Specification and the Purification Specification indicated on the Alkali Metal Surveillance Record have been approved by the Alkali Metal Custodian.

3.2. Surveillance

3.2.1. Purpose. The purpose of the surveillance function is to provide continuous information concerning all alkali metals in the plant. It will yield immediate knowledge about the quantities, location and history of each lot of metal. Furthermore, it will help ensure that a particular lot of alkali metal is appropriate for the facility in which it is being used. The surveillance function is also designed to provide quality assurance information.

3.2.2. Alkali Metal Surveillance Record. The Alkali Metal Surveillance Record (Form SP 1061) will, when completed, provide the information required to accomplish the task set forth in the preceding paragraph. Referring to the example of this record shown as Appendix A, the method of use is as follows:

a. The requestor will at the time of initiation of a Work Authorization attach a copy of the Surveillance Record after filling in the following items: Requestor, Vendor, W/A Number, Intended Use, Material Specification, and Purification Specification.

b. The requestor will send the W/A and Surveillance Record to the Alkali Metal Custodian.

c. The Alkali Metal Custodian will review the W/A and Surveillance Record and approve the Material Specification and the Purification Specification, or will make necessary changes in the Specifications and then approve. The requestor will be informed of any changes.

d. The W/A will be revised, if necessary, approved by the Custodian and sent to Purchasing. The Surveillance Record will be retained by the Custodian.

e. Purchasing will, after approval by Finance, place the order and inform the Custodian of the order number.

f. On receipt of the material, the Custodian will arrange for its transfer to an approved place of storage or to an analytical facility where the material can be sampled.

g. After sampling, the Custodian will arrange for its transfer to an approved place of storage.

h. After receipt of the vendor's analysis and completion of the "in-house" analyses, the Custodian will review the Surveillance Record and approve the material for use if the material conforms to the Specification. If it does not meet the Specification, the Custodian will arrange to have the material returned to the vendor or will waive that part of the Specification involved and then approve the material for use and indicate why the material may be used despite non-conformance.

i. The Custodian will make the necessary arrangements to release the material from storage and the requestor will arrange for the transfer to the facility.

j. When testing is complete or the material is of no further use, it will be disposed of by burning or hydrolyzing, returned to storage, or transferred to shipping for return to the vendor. The requestor will make these arrangements and inform the Custodian by means of an autogram. The autogram must indicate the Material Designation Number and the planned disposition.

3.3. Transporation.

3.3.1. Interplant Transportation. Transfer of alkali metals by commercial carriers between plants will be in accordance with Interstate Commerce Commission, Regulations Applying to Shippers, Part 73-206.

3.3.2. Intraplant Transportation. Quantities of alkali metals in excess of one pound will be transported in steel, stainless steel, or other suitable metal containers which are sealed against water and atmosphere. Quantities in excess of ten pounds will be transported on fork lift trucks or on the beds of open body trucks. Containers containing less than ten pounds of alkali metal may be transported in a passenger automobile, but only with the approval of the Custodian or Safety Specialist. The only alkali metal which may be transported on any public transportation vehicle such as a plant bus will be analytical specimens weighing less than one pound.

Quantities of alkali metals weighing less than a pound may be transported in glass containers which are sealed against water and the atmosphere. Such containers must be enclosed in a metal container with a tightly fitting lid, such as a paint can. The glass container must be cushioned with glass wool, steel wool, or exploded mica (Vermiculite). Combustibles such as cotton, cloths, wood chips, etc. must not be used as a cushioning agent.

3.4. Storage and Inventories

3.4.1. Approved Storage Areas. At present there is one approved place of permanent storage for alkali metals. It is the storage room of Building 309.

3.4.2. Inventories. The total alkali metal inventory consists of that which is stored in Building 309 and those quantities which are in use or being kept temporarily at various approved locations. These approved locations, the quantities permitted in each, and the conditions governing storage and use are indicated in the following paragraph.

3.4.2.1. Permissible Quantities and Approved Locations. The locations where alkali metals may be used and stored, the quantities permitted in each, and the conditions governing storage and use are determined by consideration of probable damage to equipment. These regulations have been formulated in accordance with the desires of the insurance company involved, and a failure to follow them could result in a considerable increase in insurance rates.

Approved locations and permissible quantities are shown in the table in Appendix B of this specification.

3.4.2.2. Periodic Checking of Inventories. The Alkali Metal Custodian will, at quarterly intervals, take an inventory of all alkali metals. This inventory will verify the Surveillance Records and determine whether or not there are cases of non-conformance with the procedures specified herein.

3.4.3. Containment Vessels. Alkali metals weighing more than one pound must be stored in metal containers, sealed against atmosphere and water; the containers to be fabricated from a metal which is resistant to attack by the contained alkali metal; i.e., steel or stainless steel. Stainless steel packless valves with corrosion resistant bellows or diaphragms should be used. Such containers, excluding vendor shipping containers, should be welded in accordance with 03-0014-00-A and leak-checked, prior to use, in accordance with 03+0013-00-A or MIL-STD-271C (Ships). The requirements for vendor shipping containers will be found in the applicable Materials Specifications.

Glass containers should, in general, be avoided for the storage of alkali metals. However, quantities weighing less than one pound may be stored in glass containers which are sealed against the atmosphere and water. Such containers must be packed carefully in a cushioning material such as glass wool, steel wool or Vermiculite inside of a steel container which has a tightly fitting lid, such as a paint can.

All supply containers (such as shipping containers) must be labelled. The label should indicate the alkali metal, the Material Designation Number which appears on the Surveillance Record, the original requestor, the tare of the container, the amount of alkali metal remaining, the name of the person who determined the amount remaining, and the date of the determination. Such labels must be protected so that they are not defaced during handling and storage.

3.5. Purification. Purification procedures will differ for different alkali metals and for different facilities. Consequently, the approved purification procedures have been written up as separate specifications. These specifications are listed in paragraph 2.2. of this specification; for example, 01-0034-00-B gives the approved procedure for hot trapping potassium.

3.5.1. Waiver of Purification Specifications. Under certain circumstances it may be possible to waive a certain purification specification or a portion thereof or to modify it. Such changes can be made only with the approval of the Alkali Metal Custodian, and must be documented on the Surveillance Record before the material is approved for use.

3.6. Chemical Analytical Methods

3.6.1. Methods for Determining Oxygen in Alkali Metals

3.6.1.1. The Amalgamation Method for Oxygen In Potassium, Sodium and NaK

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3.6.1.1.6.2. Vacuum Operation

3.6.1.1.1. Introduction. The amalgamation method for oxygen in alkali metals consists of amalgamating the alkali metal with the mercury and allowing the insoluble "oxide" to separate from and float to the top of the amalgam, after which the "oxide" quantity is determined by titrimetric or flame photometric techniques. As with other analytical methods, the results produced by the amalgamation method are influenced by the specific techniques, apparatus and conditions used in employing the method. Consequently, the detailed procedures used in conducting the various operations involved in employing the method, as well as the equipment used, is presented as follows.

3.6.1.1.2. Mercury Purification

3.6.1.1.2.1. Cleaning Procedure. The initial amalgamation reaction involves one to five grams of alkali metal and about 1000 grams of mercury. The mercury, therefore, must be free of reducible oxides as well as sodium or potassium oxides if the amalgamation method is to give an accurate indication of the oxygen content of the alkali metal.

3.6.1.1.2.1.1. Preliminary Cleanup. The mercury is washed with hydrochloric acid and then drained through a separatory funnel to remove the acid. It is then washed with water and drained through a separatory funnel to remove the film coating the mercury and the water. This step is repeated until the mercury surface is free of surface film. Acetone is added to the drained mercury to remove the traces of water, using the separatory funnel. The mercury is drained into hexane and then into a dry separatory funnel. This washing process cleans the mercury of alkali metal salts, water, grease and other impurities.

3.6.1.1.2.1.2. Oxide Removal. The mercury is purified further by oxidizing the other metallic species in the mercury and then mechanically separating these oxides from the mercury by floatation and filtration. The apparatus used for this is a Bethlehem oxifier. The mercury is agitated in a revolving container for four hours with warm air bubbling through the mercury to oxidize the impurities. The mercury-oxide mixture is then allowed to sit in a separatory funnel for at least twenty-four hours during which the oxidized materials float to the top. The pure mercury is then drained from the bottom of the funnel through a gold-adhesion filter into a clean polyethylene bottle and stored for later use.

3.6.1.1.3. Amalgamation Analytical Apparatus. The apparatus used in employing the amalgamation method is shown in Figures 3.6.1.1. and 3.6.1.2. The basic components of the apparatus are:

a. a stainless steel extruder or a liquid sampling reservoir

b. an amalgamation reaction flask

c. a mercury supply reservoir

d. a stainless steel cutter assembly

e. a vacuum-argon manifold

f. a cold trap

g. a titanium boat to accept the waste sample

h. a nickel stirring bar and magnet

i. Viton O-ring seals

A sketch of the extruder is shown in Figure 3.6.1.3. All components are of stainless steel with Viton O-ring seals.

The liquid sampling reservoir and transfer system is shown in Figure 3.6.1.4.

3.6.1.1.3.1. Vacuum System. The vacuum system shown in Figure 3.6.1.1. consists of a Welch Duo-Seal roughing pump and a VEECO model VS-9 pumping system, which includes an air-cooled, oil diffusion pump and optically dense cold trap.

3.6.1.1.3.2. Inert Gas Purification System. The argon or helium used passes through a molecular sieve dryer (Linde type 13X) and a 1500°F hot trap containing titanium chips.

3.6.1.1.4. Equipment Preparation

3.6.1.1.4.1. Extrusion Sampling. An analytical sample of solid sodium or potassium is obtained by extruding the metal into the reaction section with the extruder system shown in Figure 3.6.1.3., and shown attached to the reaction assembly in Figures 3.6.1.1. and 3.6.1.2.

The extruder is designed to accept 0.5-inch outside diameter tubing only. The plunger diameter is 0,437 inch and requires that 20-mil wall tubing be used. The minimum sample tubing length is 12 inches if Swagelok fittings are used to seal the ends.

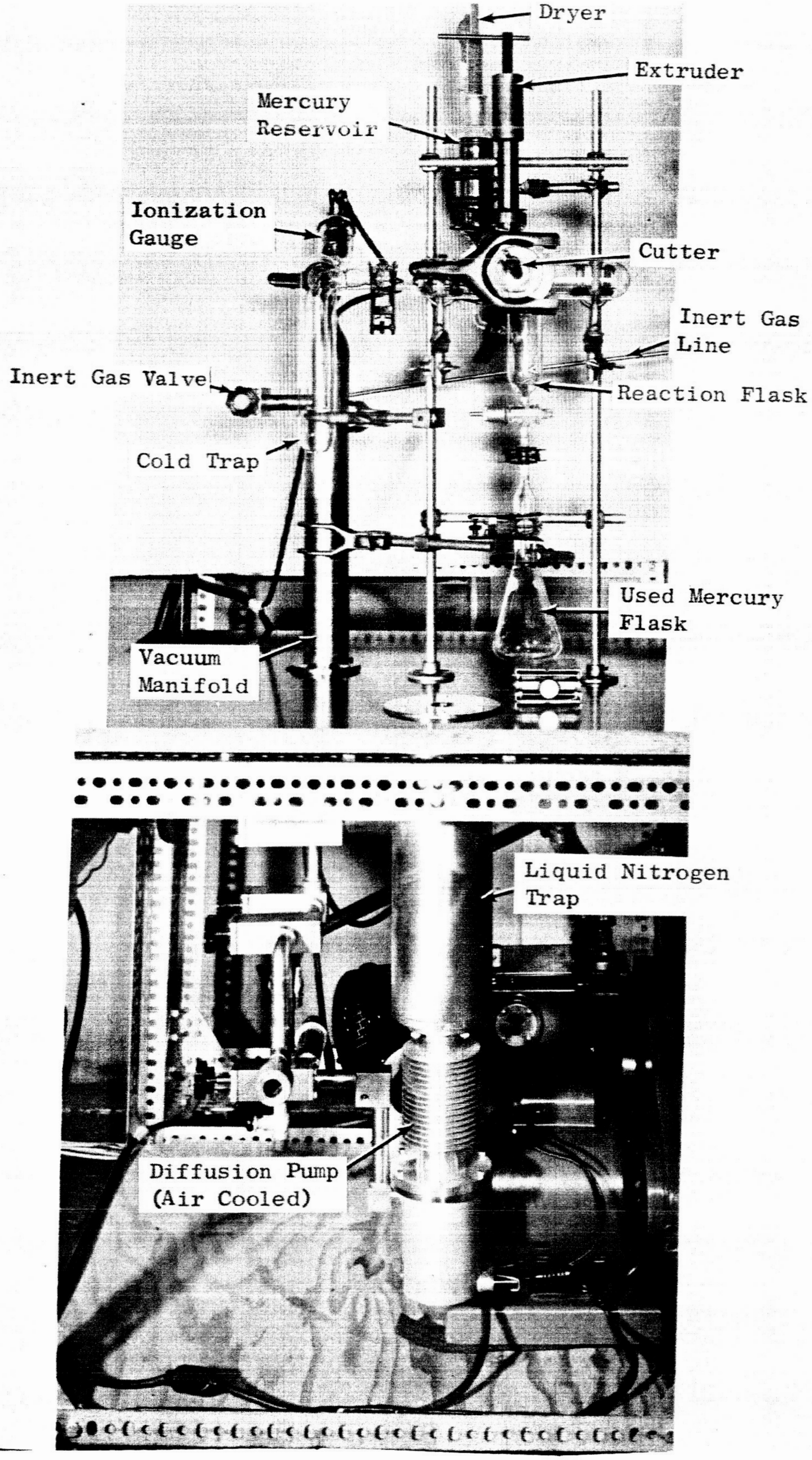


Figure 3.6.1.1. Vacuum-Inert Gas Amalgamation Apparatus Showing Vacuum System. (C64041572).

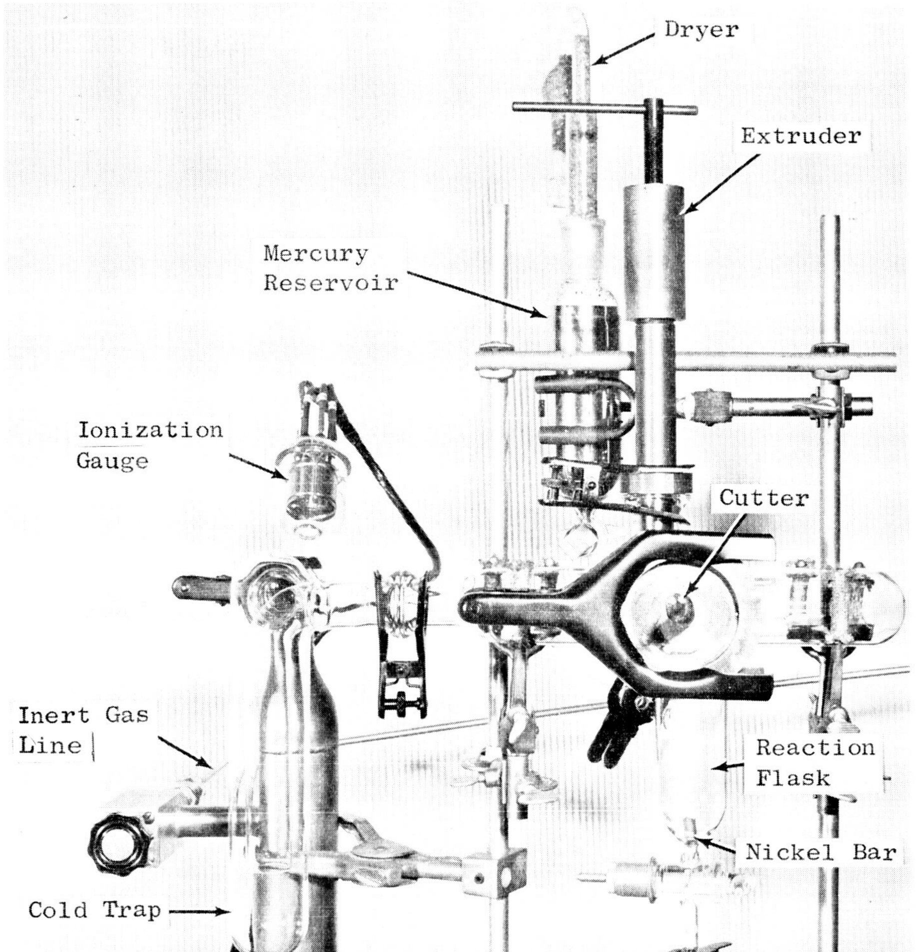


Figure 3.6.1.2. Vacuum-Inert Gas Amalgamation Apparatus. (C64041574).

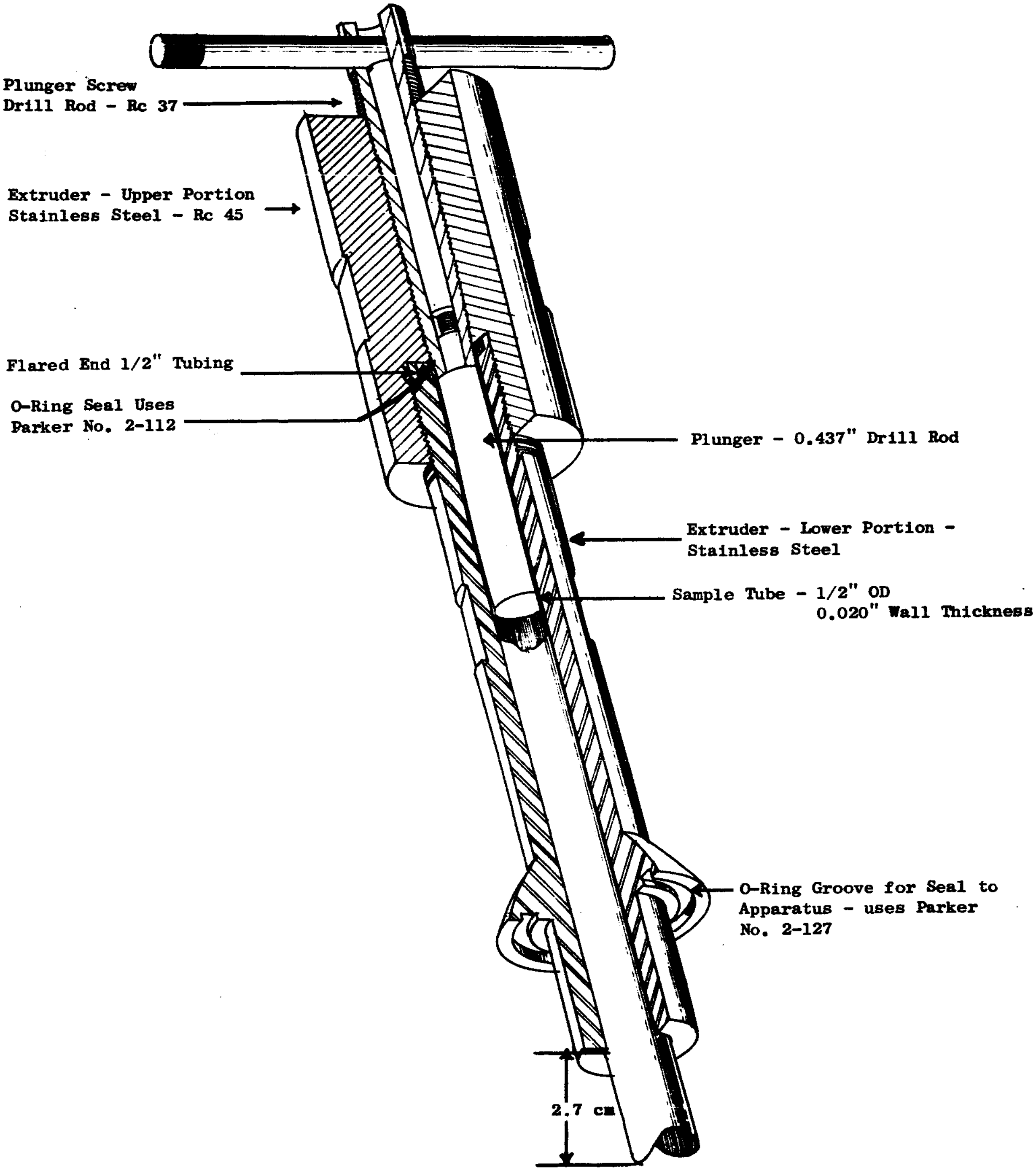
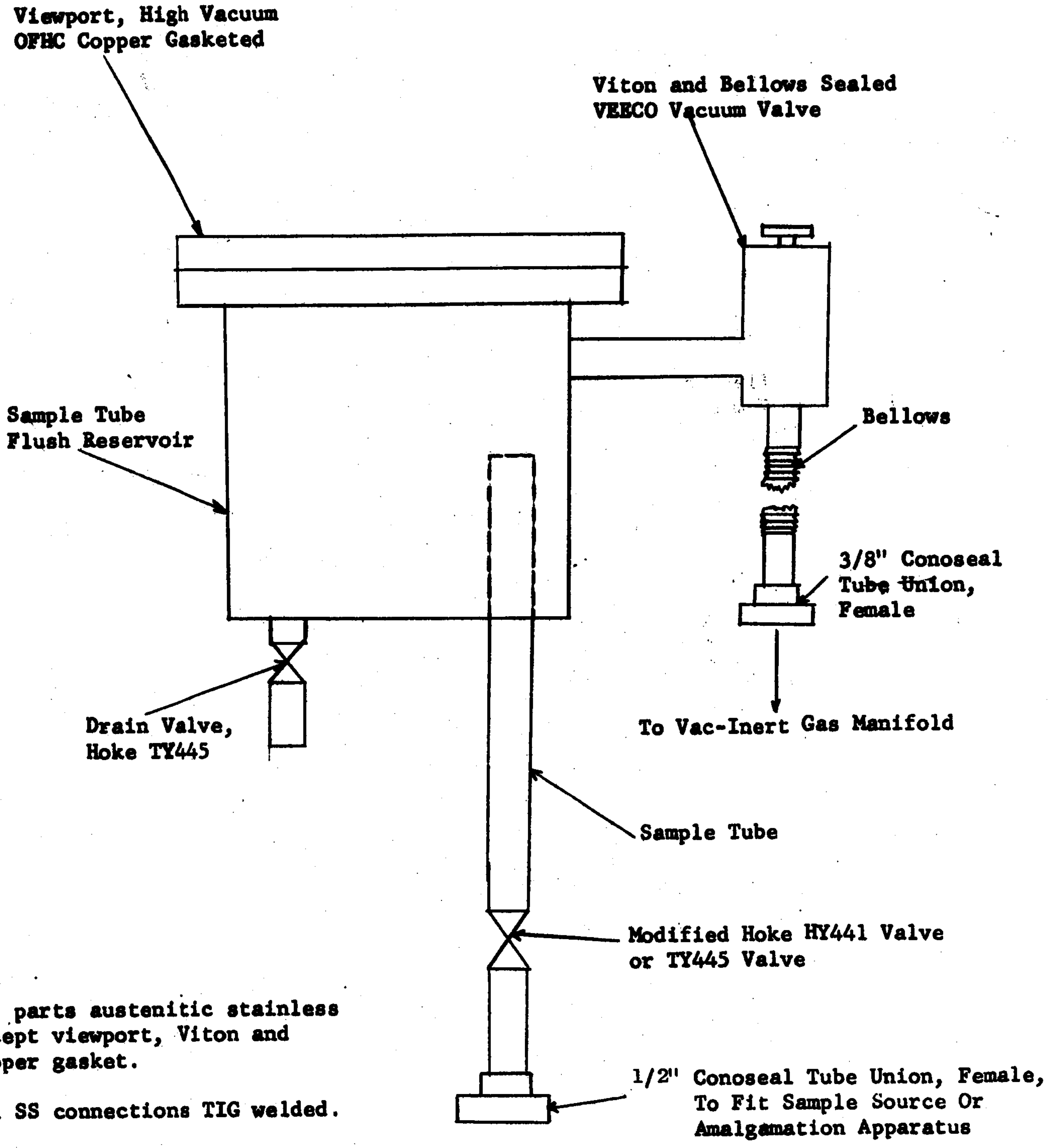


Figure 3.6.1.3. Alkali Metal Sample Extruder.



Notes:

1. All parts austenitic stainless except viewport, Viton and copper gasket.
2. All SS connections TIG welded.
3. Drawing not to scale.

Figure 3.6.1.4. Alkali Metal Sampling Reservoir.

3.6.1.1.4.1.1. Extruder Sample Preparation. One end of the sample tube is cut off with a tubing cutter as close to the Swagelok fitting as possible. This end of the tube is inserted into the bottom of the lower portion of the extruder and pushed up through the top. The O-ring (see Figure 3.6.1.3.) is placed over the tube, a small amount of alkali metal is removed, and the tube end is flared. The upper portion of the extruder is then screwed into place, forcing the flared tubing to seal against the top of the O-ring. Plunger(s) and plunger screw are then loaded and lowered until the potassium is contacted. The tube end protruding from the bottom is then cut off 2.7 cm below bottom of the lower position of the extruder. The extruder assembly is then attached to the reaction section of the analytical apparatus with a clamped O-ring joint.

3.6.1.1.4.1.2. Cutter Preparation. As shown in Figure 3.6.1.2., the cutter assembly is attached to the large glass O-ring fitting of the reaction flask. The cutter moves via a bellows for cutting samples and is positioned to place the cutter wire over the waste boat. The cutter wire is cleaned with water, hydrochloric acid, water, acetone, and air-dried, in this order. The cutter wire is heated by infrared lamps immediately prior to assembly.

3.6.1.1.4.1.3. Flask Preparation. The reaction flask is flushed thoroughly with hexane to remove all traces of mercury and grease. The flask is then washed in a "Sparkleen" solution, rinsed in water, then acetone, and then oven-dried. The stopcock is turned in with Apiezon "N" grease.

3.6.1.1.4.1.4. Stirring Bar Preparation. The nickel stirring bar is washed with nitric acid to remove mercury, rinsed with water, washed with hydrochloric acid, water, and acetone, and then dried in an oven.

3.6.1.1.4.1.5. Waste Boat Preparation. The titanium boat is cleaned of potassium waste metal, rinsed in hydrochloric acid, water, and acetone, and then oven dried.

3.6.1.1.5. Apparatus Assembly

3.6.1.1.5.1. Assembly. Referring to Figures 3.6.1.1. and 3.6.1.2., the apparatus is assembled as follows:

a. Attach the cold trap to the vacuum-argon manifold with inert gas flowing.

b. Insert the nickel stirring bar and titanium waste boat in the reaction flask and connect the reaction flask to the cold trap. The reaction flask must also be supported, as shown in Figure 3.6.1.1.

c. Install the extruder, containing the sample tube, on the reaction flask.

d. Attach the mercury reservoir to the reaction flask as shown in Figure 3.6.1.2. Support the mercury reservoir so that undue stress is not placed on the glassware.

e. Connect the cutter system to the reaction flask and check to see that the cutter can be positioned over the titanium waste boat.

f. Attach the amalgam receiver flask to the bottom of the reaction flask and support it with a lab jack.

g. Turn off the inert gas flow and evacuate the apparatus to begin outgassing. If leaks are suspected, find them with acetone and eliminate them.

h. Attach heating tapes and/or heating jackets to the apparatus in preparation for outgassing.

3.6.1.1.5.2. <u>Outgassing</u>. Referring to Figure 3.6.1.2., the glassware is heated by flaming with a gas burner to de-gas the interior surfaces of the flask and cold trap. The bellows and flange on the cutter assembly are heated by heat lamps. Due to the presence of solid alkali metal in the extruder, the outgassing temperature is limited to a temperature below the melting point of the metal to avoid melting it out of the sample tube.

3.6.1.1.5.2.1. <u>Inert Gas Operation</u>. For conduction of the analysis under inert gas, the system is outgassed until the pressure at the ion gauge shown in Figure 3.6.1.2. is 10^{-5} torr maximum while the system is cold. The pressure of $2\text{-}5 \times 10^{-6}$ torr is usually achieved within two hours.

3.6.1.1.5.2.2. <u>Vacuum Operation</u>. For analyses under vacuum, the system is outgassed until the pressure is 2×10^{-7} torr maximum and the pressure rise rate is less than 0.5 micron-liter per hour. This requires 24 to 48 hours of outgassing.

3.6.1.1.6. <u>Analytical Procedure</u>

3.6.1.1.6.1. <u>Inert Gas Operation</u>. When conducting the amalgamation analysis under inert gas, the cold trap between the analytical apparatus and the vacuum-inert gas manifold is put into use while the apparatus is cooling down after bakeout. (See Figure 3.6.1.1.). If argon is to be used, the cold trap is cooled by a dry-ice-acetone mixture or with liquid nitrogen if helium is to be used as the cover gas. With the cold trap in operation and the ion gauge indicating less than 5×10^{-6} torr pressure, the analytical apparatus is backfilled with inert gas to a pressure which exceeds atmospheric pressure by about 1/2 psi or one inch of mercury.

3.6.1.1.6.1.1. <u>Extrusion Sampling - Sodium and Potassium</u>. The solid alkali metal sample is obtained from the extruder as follows:

a. The cutter wire is positioned to the left of the sample tube.

b. About 1/2 inch of metal is extruded from the tube and cut off even with the end of the tube.

c. This waste piece is then positioned over and melted off into the waste boat by infrared heat lamps or resistance heating of the cutter wire.

d. The cutter is repositioned to the left of the sample tube, another 1/2-inch of metal is extruded, cut and melted into the waste boat.

e. The cutter is repositioned to the left of the sample tube and the sample for analysis is extruded. The sample size is normally varied between 1 and 5 gms. or 3/4 to 3 3/4 inches long. The longer samples must be cut off in small pieces which are dropped individually into the reaction flask.

f. The sample is cut, positioned over the center of the reaction flask, and melted off the wire into the bottom of the flask.

3.6.1.1.6.1.2. <u>Liquid Sampling - Sodium, Potassium and NaK</u>. For obtaining liquid samples, the reservoir shown in Figure 3.6.1.4. or a NaK container is connected to the analytical apparatus instead of the extruder.

3.6.1.1.6.1.2.1. Liquid Sampling Apparatus. Figure 3.6.1.5. shows the apparatus used in obtaining a liquid sample. The liquid container is attached to the transfer line by a Conoseal, Swagelok or equivalent connector. The waste boat is positioned over the reaction flask and the cutter is placed to the right of the waste boat.

3.6.1.1.6.1.2.2. Liquid Sampling Procedure. For sodium and potassium, the sampling reservoir must be heated to melt the metal. Sodium should be heated to 300°F plus or minus 10°F, and potassium should be heated to 200°F plus or minus 10°F. A one-hour residence time at the recommended temperature has proven adequate to equilibrate the system and provide a homogeneous liquid sample. The sampling procedure is as follows:

a. Open the valve on the reservoir (see Figure 3.6.1.4.) or the NaK container and flush the transfer line with 1 to 5 gms of liquid metal into the waste boat.

b. Push the waste boat to the left with the cutter rod until the transfer line outlet can see the bottom of the reaction flask.

c. Secure the cutter rod so that liquid does not touch it while taking the sample.

d. Add the sample to the reaction flask dropwise, again using 1 to 5 gms of metal, using the same valve used to control the flushing of the transfer line.

e. Allow sodium or potassium samples to cool and solidify before proceeding with the analysis.

3.6.1.1.6.1.3. Sample Amalgamation. The sodium, potassium or NaK sample should be amalgamated with the mercury as follows:

a. Introduce mercury into the reaction flask slowly (to avoid smoking) until 40 ml of mercury have been added.

b. With solid potassium samples, heat the mercury gently until the metal begins to melt on top of the mercury, and then react the metal with the mercury by gentle manipulation of the sitrring bar. AVOID A VIOLENT REACTION of potassium with mercury by exercising care in heating the mercury.

c. With sodium or NaK samples the amalgamation reaction is instantaneous and quite vigorous. For these metals it is not possible to float the material on top of the mercury. Therefore, the mercury is added in small increments until no further smoking occurs and then heated gently without agitation until the reaction is complete, as evidenced by no detection of solid metal with the stirring bar.

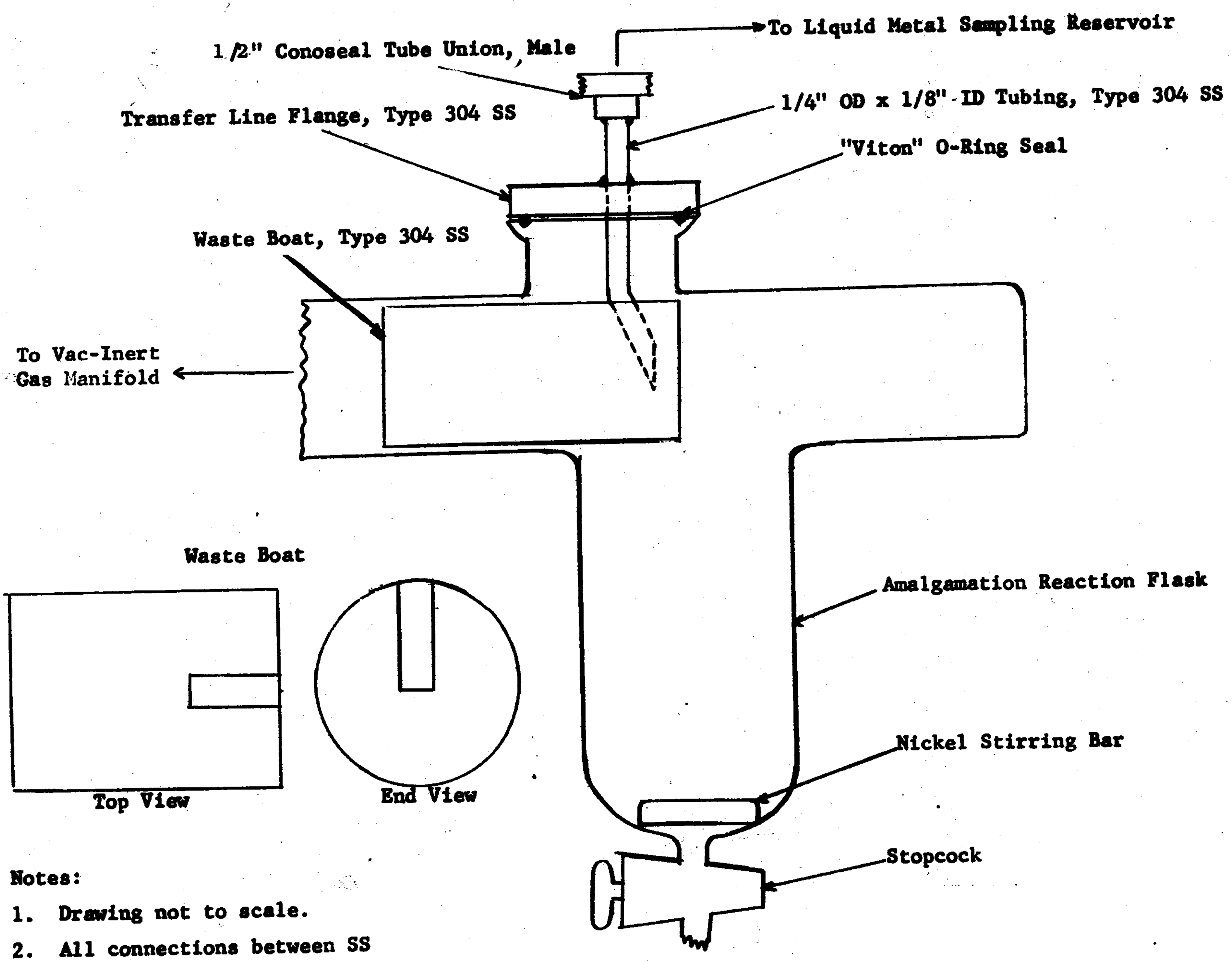
3.6.1.1.6.1.4. Extraction of the Alkali Metal from the Mercury

a. Drain the initial alkali metal-rich amalgam from the reaction flask into 1N HCl until 1 to 2 ml of amalgam remain in the reaction flask. The quantity of HCl needed will depend on the sample weight and should be in excess of that needed to convert all the free alkali metal to the chloride.

b. Add 20 ml of mercury to the reaction flask, heat gently to about 150°F and clean all metal droplets or particles from the flask walls with the nickel stirring bar.

c. Again drain the amalgam into the 1N HCl used in (a) above, leaving 1 to 2 ml.

d. Repeat steps (b) and (c) three more times; however, on the last extraction, the amalgam (mercury) is drained into 1 to 2 ml of distilled water containing 1 to 2 drops of phenolphthalein. If no red color results, it is assumed that all free alkali metal has been extracted from the reaction flask.



Notes:

1. Drawing not to scale.
2. All connections between SS parts are TIG welded.

Figure 3.6.1.5. Basic Elements of Apparatus for Liquid Alkali Metal Sampling for the Amalgamation Method.

3.6.1.1.6.1.5. "Oxide" Residue Removal. The "oxide" residue is then extracted from the reaction flask with distilled water of known pH and known volume. The water is sprayed over the interior walls of the flask and the stirring bar is used also to scrub the walls with water.

3.6.1.1.6.1.6. "Oxide" Residue Analysis. The residue solution is analyzed for oxygen content by both titrimetric and flame photometric methods.

3.6.1.1.6.1.6.1. Titrimetric Analyses

3.6.1.1.6.1.6.1.1. Titration Apparatus. Most analyses of alkali metals for oxygen by the amalgamation method are done on reasonably pure or purified metal. Thus the oxygen quantities normally found are small and require micro-techniques to obtain accurate titration data. The titration equipment used is described as follows:

The acid is added to the solution being titrated via a micro-buret syringe made by the Micro-Metric Instrument Co. The syringe has a volume displacement of 0.5 microliter of solution per each 0.001 inch of plunger travel and a total capacity of 0.5 ml of acid.

The syringe is operated by a Bird Kymograph which has speeds of 1, 0.2, 0.004, 0.008 and 0.0016 inch per minute.

The pH of the solution is measured by a Leeds and Northrup pH indicator, catalog No. 7664, which is connected in series to a Brown recorder where a trace of pH vs. time is recorded during the titration.

The solution being titrated is agitated with a Teflon covered stirring bar rotated by a magnetic stirrer.

3.6.1.1.6.1.6.1.2. Titration Procedure. The titration procedure is as follows:

a. The pH of the water used to extract the residue is first measured and the recorder is standardized.

b. The micro-buret syringe is filled with 0.0125 N HCl normally, although stronger acid may be used when the oxide content of the residue is high.

c. The acid transfer tube and the pH electrodes are washed thoroughly with distilled water, then immersed in the residue solution, and the stirrer is turned on.

d. The recorder and Kymograph are actuated simultaneously and acid is added to the solution until the pH decreases to below the pH of the extraction water.

e. The pH vs. time trace is removed from the recorder and the oxygen content of the residue solution is calculated.

3.6.1.1.6.1.6.1.3. Titration Calculation. The oxygen content of the residue solution is calculated as follows: The determination of the oxygen content is based on the following two equations, using potassium monoxide as the example:

$$K_2O + 2HCl \rightarrow 2KCl + H_2O \text{ ---------------------------(1)}$$

$$K_2CO_3 + 2HCl \rightarrow 2KCl + H_2O + CO_2 \text{ ------------------(2)}$$

From equation (1), one atomic weight of oxygen will react with two moles of HCl. From equation (2), one atomic weight of oxygen as K_2O from K_2CO_3 will react with two moles of HCl. Thus, although the carbonate may be present, the calculation of the oxygen is based on K_2O plus K_2O from K_2CO_3.

Therefore, the oxygen amount is calculated as follows:

$$\frac{8\ \text{gms O}}{\text{moles HCl}} \times \text{liters HCl} \times \frac{\text{moles HCl(N)}}{\text{liter}} = \text{gms O} \text{ -----------------(3)}$$

where N = normality (usually 0.0125N or 0.125N HCl)

To determine the volume of HCl, the following formula is employed:

$$\text{liters HCl} = \frac{C \times I \times T \times S}{1000} \text{ ----------------------------------(4)}$$

where: C = recorder chart speed, minutes/inch
I = distance between points on chart where pH is that of residue extraction water, inches
T = microburet plunger speed, inches/minute
S = microburet volume displacement, milliliters/inch

Inserting equation (4) into equation (3) gives:

$$\text{gms O} = 0.008 \times C \times I \times T \times S \times N \text{ -------------------------------(5)}$$

Therefore:

$$\text{ppm O} = \frac{0.008 \times C \times I \times T \times S \times N \times 10^6}{\text{sample weight, gms}} \text{ ----------------------(6)}$$

3.6.1.1.6.1.6.2. Flame Photometric Analysis. An alternate method of measuring the amalgamation residue makes use of flame emission photometry. Any commercial flame photometer capable of isolating and measuring the appropriate line emission of the alkali metal of interest may be used. The Beckman DU Spectrophotometer has been found to be quite satisfactory for this purpose. The general flame analytical procedure is as follows:

a. Calibration curves are prepared over various concentration ranges of NaCl and KCl in the distilled water used for residue extraction. The spectrographically pure NaCl and KCl used in making the known standard solutions are obtained from Johnson, Matthey & Co., Limited in London, England.

b. The total potassium and sodium contents of a given residue extraction solution are then determined by transmission measurements, and the calibration curves are based on known standards.

c. The assumption is then made that all potassium and sodium were originally present as monoxide and the oxygen contents are calculated on this basis.

3.6.1.1.6.1.7. Sample Weight Determination. The sample weight is determined by back titration of the acid solution produced by the reaction of the alkali metal amalgam with the 1N HCl as described in 3.6.1.1.6.1.4. The end point indicator used is phenolphthalein. 1N NaOH is used for the back titration. The sample is determined from the following formula:

$$\text{Sample weight, grams} = (\text{ml HCl} - \text{ml NaOH}) \; \frac{(\text{mol. wt. of alkali metal})}{1000}$$

3.6.1.1.6.2. Vacuum Operation. For vacuum operation, both cold traps shown on the apparatus in Figure 3.6.1.1. are cooled with liquid nitrogen. Otherwise, the analytical technique is the same as for inert gas with a few minor differences. The vacuum amalgamation technique has been shown to be capable of producing results for oxygen in potassium which compare fairly well with results obtained on similar samples by the amalgamation procedure run under inert gas; however, the vacuum technique does present the following problems. Mercury is deposited in the upper, colder regions of the reaction flask, making post-analytical cleanup a greater problem. The amalgamation reaction also tends to be more violent under vacuum. Any alkali metal hydrides may be partially decomposed at the amalgamation temperature under vacuum.

3.6.1.2. Determination of Oxygen in Lithium

3.6.1.2.1. The Fast Neutron Activation Method. At present the fast neutron activation technique is the only approved method for determining oxygen in lithium metal, and the only approved analytical laboratory is Activation Analysis Service, General Atomic Division, General Dynamics, P. O. Box 608, San Diego, California 92112.

3.6.1.2.1.1. Specimen Preparation. Specimens of lithium metal should be obtained as described in Sections 3.7.3.1., 3.7.4.1.1. and 3.7.4.2. of this specification. The stainless steel sample tube should be capped tightly with stainless steel Swagelok (or equivalent) caps prior to shipment.

3.6.1.3. Determination of Oxygen in Rubidium. Same as Section 3.6.1.2.

3.6.1.4. Determination of Oxygen in Cesium. Same as Section 3.6.1.2.

3.6.2. Preparation of Samples for Determination of Metallic Impurities in Alkali Metals.

3.6.2.1. Sodium, Potassium and NaK. Spectrographic analysis of potassium, sodium, or NaK for metallic impurities is done on the chloride salts of the alkali metals. The chloride salts are prepared as follows:

a. A platinum dish is cleaned by melting potassium pyrosulfate in the dish with agitation to assure that the entire platinum surface is contacted by the molten salt. The residue is then washed from the dish with distilled water and oven-dried at 100-120°C.

b. The alkali metal sample (1-2 gms) is then introduced into the clean platinum dish. The metal may be extruded, cut and placed in the dish as a solid, or it may be introduced in liquid form. In any case, the method of placing the metal specimen into the dish should be such that no metallic contaminants are added to the sample.

c. Cover the alkali metal sample with spectroquality hexane (DO NOT USE PRACTICAL GRADE HEXANE), and add absolute ethanol to the hexane until the alkali metal is converted to the ethoxide. FACE SHIELD AND GLOVES SHOULD BE USED DURING DISSOLUTION OF THE METAL FOR PERSONNEL PROTECTION.

d. Add concentrated reagent grade HCl to the ethoxide-ethanol-hexane solution dropwise to convert all ethoxide to the chloride. If HCl is ADDED TOO RAPIDLY, THE HEAT OF THE CONVERSION REACTION WILL IGNITE THE FLAMMABLE SOLUTION.

e. Place the chloride solution on a hot plate or steam bath and evaporate to dryness in a fume hood.

f. Store the resulting alkali metal chloride in a clean glass vial until the spectrographic analysis is done.

3.6.2.2. Lithium. Spectrographic analysis of lithium for metallic impurities is performed on the carbonate, Li_2CO_3. The carbonate is prepared as follows:

a. Same as Section 3.6.2.1. a

b. Same as Section 3.6.2.1. b

c. Same as Section 3.6.2.1. c

d. Add distilled water to the ethoxide-ethanol-hexane solution dropwise to hydrolyze the ethoxide. Then bubble CO_2 through the solution using a non-contaminating tube; i.e., platinum; or cover with CO_2 until the pH of the aqueous solution is reduced to 8 or 9. Dry ice is a convenient source of CO_2 and is sometimes used to convert LiOH to Li_2CO_3.

e. Evaporate to dryness.

f. Store in a clean, dry, glass vial

3.6.3. Spectrographic Methods for Determining Metallic Impurities in Alkali Metals

3.6.3.1. Potassium

3.6.3.1.1. Specimen Preparation. Metallic impurities in potassium metal are usually determined on the chloride salt of potassium and the impurity levels are reported as ppm impurity in potassium chloride. The procedure for converting potassium metal to potassium chloride is described in paragraph 3.6.2.1. of this specification.

3.6.3.1.2. Analytical Procedure. This method provides for the quantitative determination of 20 impurity elements in the range 25-250 ppm in potassium chloride prepared from potassium metal. The method can also be used for obtaining a semi-quantitative estimate of the 20 impurity elements in the range 1-25 ppm by varying conditions as outlined below.

3.6.3.1.2.1. Apparatus and Reagents

a. Jarrel-Ash 3.4-meter Wadsworth Mounting Spectrograph.

b. Excitation Source, NSL Spec - Power.

c. Neutral Density Filter, Steps % Transmission, 100-64.3-42.5-27.0-17.5-11.3-7.1.

d. Comparator microphtometer, Jarrell-Ash Model SA-200.

e. Photographic plate processing unit, NSL Spec. Processor.

f. Spectrographically pure KCl.

g. Spectrographic Standards, Jarrell-Ash Master SQ powder standards.

h. Graphite Electrodes.

3.6.3.1.2.2. Preparation of Standard Curves. Spectrographic Standards for establishing analytical curves are prepared by mixing Jarrell-Ash "Master SQ Standards" with spectrographically pure KCl. Fifteen milligrams of standards containing from 25 to 250 ppm of the impurities of interest are weighed directly into graphite cup electrodes. The loaded electrodes are dried at 115°C for 30 minutes. The dried, loaded electrodes are stored in a dessicator until arced. Details of the analytical apparatus and operating parameters are summarized in Table 3.6.3.1. Using the established conditions, the samples are arced for the designated times and the spectra recorded on photographic plates.

TABLE 3.6.3.1.

APPARATUS AND CONDITIONS

Spectrograph	Jarrell-Ash 3.4 m Wadsworth
Wavelength Range, A	2400-4250
Slit Width, μ	30
Slit Height, mm	2.8
Filter	Neutral density, steps %T, 100-64.3-42.5-27.0-17.5-11.3-7.1
Excitation Source	NSL Spec-Power
DC Amps	8.0
Electrodes	
Sample	UCP-105-S
Counter	UCP-2615
Analytical Gap, mm	4
Gas Mixture to Stallwood Jet	50-50 Argon-Oxygen, Burdett Oxygen Co.
Flowrate, SCFH	8
Exposure	
Pre-Burn	None
Exposure	Total Burn
Emulsion	Kodak SA-1, developed in D-19 for 4 minutes at 68°F

Intensity ratios based on the 3446.722A line of potassium are determined for the standards using the comparator-microphotometer and a Seidel calibration curve. The analytical line pairs for each of the elements of interest are given in Table 3.6.3.2. The final analytical curves are obtained by plotting the intensity ratios against impurity concentration on semi-log scales.

TABLE 3.6.3.2.

ANALYTICAL LINE PAIRS

FOR THE IMPURITY ELEMENTS SOUGHT

Element	Element Line, A[a]	Element	Element Line, A[a]
Boron	2497.733	Sodium	3302.323
Manganese	2801.064	Silver	3382.891
Magnesium	2852.129	Zirconium	3391.975
Lead	2833.069	Cobalt	3405.120
Silicon	2881.578	Nickel	3414.765
Iron	3020.640	Chromium	3605.333
Vanadium	3102.299	Titanium	3635.463
Molybdenum	3132.594	Aluminum	3961.527
Tin	3175.019	Calcium	3968.468
Copper	3247.540	Columbium	4058.938

[a] Potassium internal standard line 3446.722 A with filter, 11.6%T.

Impurity levels below 25 ppm yield lines of very low intensity and it is not practical to establish analytical curves for impurities below 25 ppm.

3.6.3.1.2.3. Analysis of Samples. Fifteen milligram samples of potassium chloride which have been converted from potassium metal according to the procedure detailed in 3.6.2.1. of this specification are loaded into graphite cup electrodes. The loaded electrodes are dried for 30 minutes at 115°C and stored in a dessicator until arced. The conditions for burning the unknown samples are identical to the conditions used to establish the analytical curves. Three exposures are required for each sample.

3.6.3.1.2.4. Calculations

a. Determine the average per cent transmission of the three exposures for each analytical line of interest.

b. Determine the concentration of each element based on KCl from the simultaneously prepared standard curve.

NOTE: For impurity levels from 1-25 ppm a semi-quantitative estimate of impurity concentration is possible by visually comparing line intensity of low level standards. This method should have an accuracy of ±5 ppm. Table 3.6.3.3. shows the detection limits and analytical line pairs for low level impurities.

TABLE 3.6.3.3.

SENSITIVITY OF DETECTION OF ELEMENTS IN POTASSIUM (AS CHLORIDE)

Element	Line Observed, A	Minimum Detected Amount
Mg	2802.70	<1
Mn	2794.82	<1
Cr	4254.35	<1
Sn	2863.33	1
Si	2516.12	<1
Ti	3653.50	1
Al	3082.16	<1
V	3102.30	5
Cu	3247.54	<1
Ag	3280.68	<1
Co	3405.12	1
Ni	3414.77	1
Zr	3601.19	5
Pb	3639.58	5
Mo	3902.96	<1
Ca	3933.67	<1
Cb	4079.73	1
B	2497.73	5
Fe	3719.94	1
Na	3302.99	1
Ba		Not detected up to 25 ppm

3.6.3.1.2.5. Precision and Accuracy. In order to evaluate the accuracy and precision associated with the method, a 100 ppm standard was prepared by mixing a Jarrel-Ash Master SQ standard with pure KCl. This standard was arced 4 times under the prescribed conditions and per cent standard deviation was calculated for each element of interest. These data are summarized in Table 3.6.3.4.

3.6.3.2. Sodium

3.6.3.2.1. Specimen Preparation. See paragraph 3.6.2.1. of this specification.

3.6.3.2.2. Analytical Procedure. This method is under development, but will be essentially the same as for potassium (See paragraph 3.6.3.1.2.). Until such time that the method is established, samples may be analyzed by Nuclear Materials Equipment Corporation, Apollo, Pa.

3.6.3.3. Lithium

3.6.3.3.1. Specimen Preparation. Metallic impurities are determined in lithium carbonate prepared from the lithium metal, and the impurity levels are reported as ppm in Li_2CO_3. The technique used to prepare the lithium carbonate is described in paragraph 3.6.2.2.

TABLE 3.6.3.4.

PRECISION AT 100 PPM LEVEL

Element	Per Cent Standard Deviation*	Element	Per Cent Standard Deviation
Boron	± 49.9	Sodium	± 32.6
Magnesium	± 7.7	Silver	± 17.1
Manganese	± 7.3	Cobalt	± 7.5
Lead	± 9.3	Nickel	± 13.4
Silicon	± 25.0	Chromium	± 13.2
Iron	± 15.9	Zirconium	± 11.6
Vanadium	± 25.5	Titanium	± 6.2
Molybdenum	± 12.2	Aluminum	± 38.4
Tin	± 15.5	Columbium	± 8.4
Copper	± 22.1	Calcium	± 50.0

*Per cent Standard Deviation in this method is calculated as follows:

$$\% \text{ Std. Dev.} = \frac{100}{C} \times \sqrt{\frac{\Sigma d^2}{n - 1}}$$

Where: C = Average concentration in ppm

d = Difference of the determination from the mean

n = Number of determinations

3.6.3.3.2. Analytical Procedure. This method is under development, but will be essentially the same as for potassium (see paragraph 3.6.3.1.2.). Until such time that the method is established, samples may be analyzed by Nuclear Materials Equipment Corporation, Apollo, Pa.

3.6.4. Analytical Methods for the Determination of Nitrogen in Alkali Metals. These methods are under development. Until such time that methods have been established samples may be analyzed by Ledoux and Company, Teaneck, New Jersey or by Mine Safety Appliance Research, Callery, Pennsylvania.

3.6.5. Analytical Methods for the Determination of Carbon in Alkali Metals. These methods are under development. Until such time that methods have been established samples may be analyzed by Ledoux and Company, Teaneck, New Jersey or by Mine Safety Appliance Research, Callery, Pennsylvania.

3.7. Alkali Metal Sampling

3.7.1. Sampling Objectives. The general objective sought in an alkali metal sample is the homogeneous, representative, uncontaminated specimen which can be analyzed as accurately as possible for the desired impurity(s) concentration. Specifically, the objectives of this sampling specification are:

a. To indicate the sampling criteria for obtaining alkali metal specimens to be analyzed for oxygen, carbon, nitrogen and metallic impurities.

b. To specify types of apparatus which can be used for taking samples

c. To present semi-detailed procedures for sample taking

3.7.2. Sampling Criteria

3.7.2.1. For Oxygen Analysis

a. When sampling high purity metal under inert gas, the purity of the gas must be such that the contamination of the sample by the gas does not exceed 1 ppm oxygen as the monoxide.

b. When sampling high purity metal under vacuum, the pressure rise rate of the sampling system must be such that the contamination of the sample therefrom does not exceed 1 ppm oxygen as monoxide.

c. When sampling liquid sodium or potassium with a suspected oxygen concentration higher than the solubility for oxygen of the metal at its melting point, it is recommended that the specimen be obtained horizontally if the analytical specimen is to be sampled later as a solid. Regardless of the sampling position used for this grade of metal, the entire sample should be analyzed and the results averaged to off-set possible uneven distribution of oxygen in the sample. Sampling temperature should be high enough to dissolve all oxide present in the metal source.

d. The transfer lines between the alkali metal source and the sampling system should be flushed with a quantity of metal twice the volume of the transfer line before sampling. The sample tube should be flushed with a quantity of metal twice the volume of the tube after the transfer line has been flushed.

e. All samples should be transferred and taken in austenitic stainless steel or other material which does not interfere with the analytical procedure and results.

f. Where high accuracy is not required or under other conditions where all the previously stated criteria cannot be met, the criteria may be waived with the approval of the person responsible for control and measurement of alkali metal purity (the Alkali Metal Custodian).

3.7.2.2. For Carbon Analysis. The essence of the criteria indicated for oxygen samples applies to samples to be analyzed for carbon except that such species as CO or CO_2 or other compounds which would contaminate the sample should not be present in the sampling system to the extent that 1 ppm carbon contamination of the sample would occur.

3.7.2.3. For Nitrogen Analysis. The essence of the criteria presented in paragraph 3.7.2.1. applies to samples for nitrogen analysis except that nitrogen or its compounds should be excluded from contact with the sample so that contamination by nitrogen does not exceed 1 ppm nitrogen.

3.7.2.4. For Metallics Analyses. The criteria by which samples should be obtained for metallic analyses by spectrographic techniques include:

a. Samples with oxygen content greater than 100 ppm should not be taken in austenitic stainless steel at temperatures in excess of 500°F if true concentrations of the elements in the sample tubing are desired, since corrosion of the austenitic stainless steel tubing could occur at higher temperatures.

b. Conversion of sodium, potassium or NaK to their chlorides should be done in a freshly pyrolyzed (potassium pyrosulfate) platinum dish with spectroquality hexane, absolute ethnaol, and reagent grade concentrated HCl. See paragraph 3.6.2.1.

3.7.3. Sampling Apparatus. The apparatus used for sampling sodium, potassium and NaK for oxygen, carbon and nitrogen analysis can be the same for all three metals except that NaK must be contained between valves since it is liquid at room temperature. The sodium and potassium may be introduced into the analytical apparatus in either solid or liquid form -- the NaK in liquid form only. The solid samples are extruded the liquid samples are taken from a leak-tight container. Thus, two types of sampling apparatus are required depending on whether solid or liquid samples are to be analyzed.

3.7.3.1. Apparatus for Solid Samples. Solid samples are obtained by extruding the metal from austenitic stainless steel tubing or by cutting metal containing sections from the tubing, For extrusion, the sample tube must be 1/2-inch outside diameter by 20-mil wall thickness and at least 10 inches long. Where sections of tubing are cut to obtain the metal sample, the tubing wall thickness is not important, but the other dimensions should approximate those used for extrusion samples.

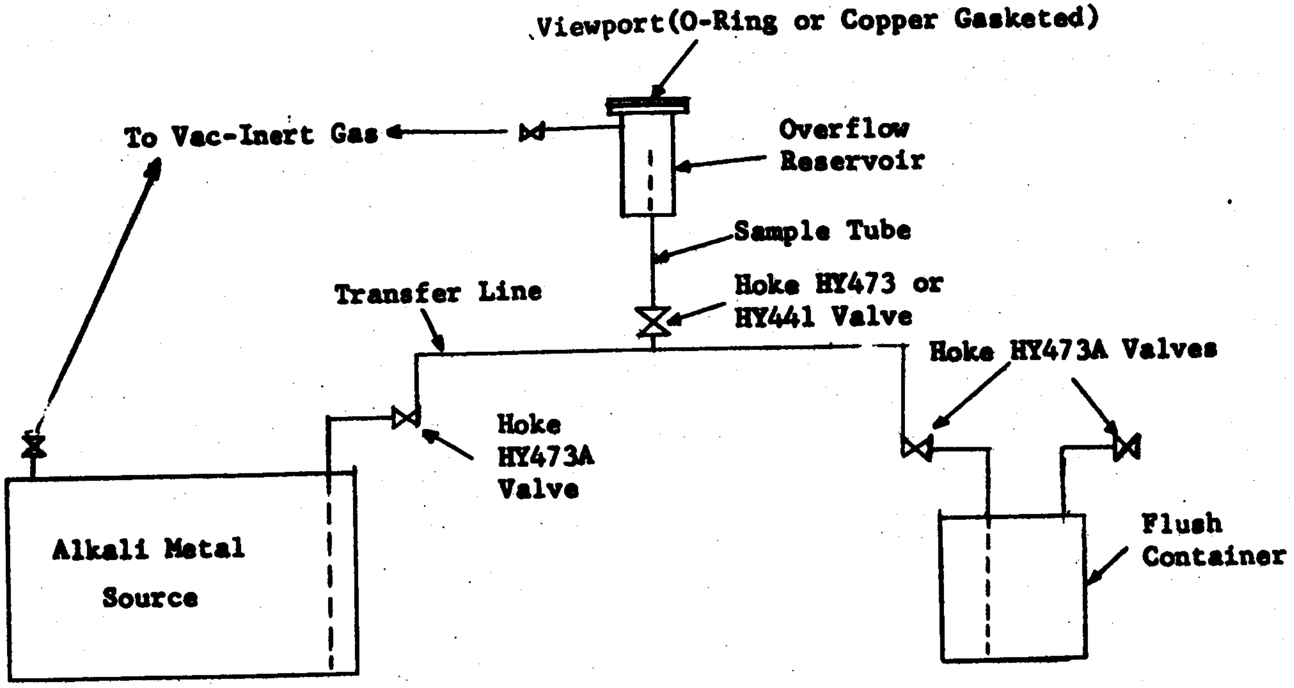
The apparatus system used for obtaining solid samples can be either of two types. The two types are shown schematically in Figure 3.7.3.1. The apparatus for sampling large alkali metal sources utilizes a flush container large enough to clear the transfer line, and the sampling system is positioned between the source and flush container as shown.

The apparatus used for small sources utilizes the sampler overflow volume as the flush container.

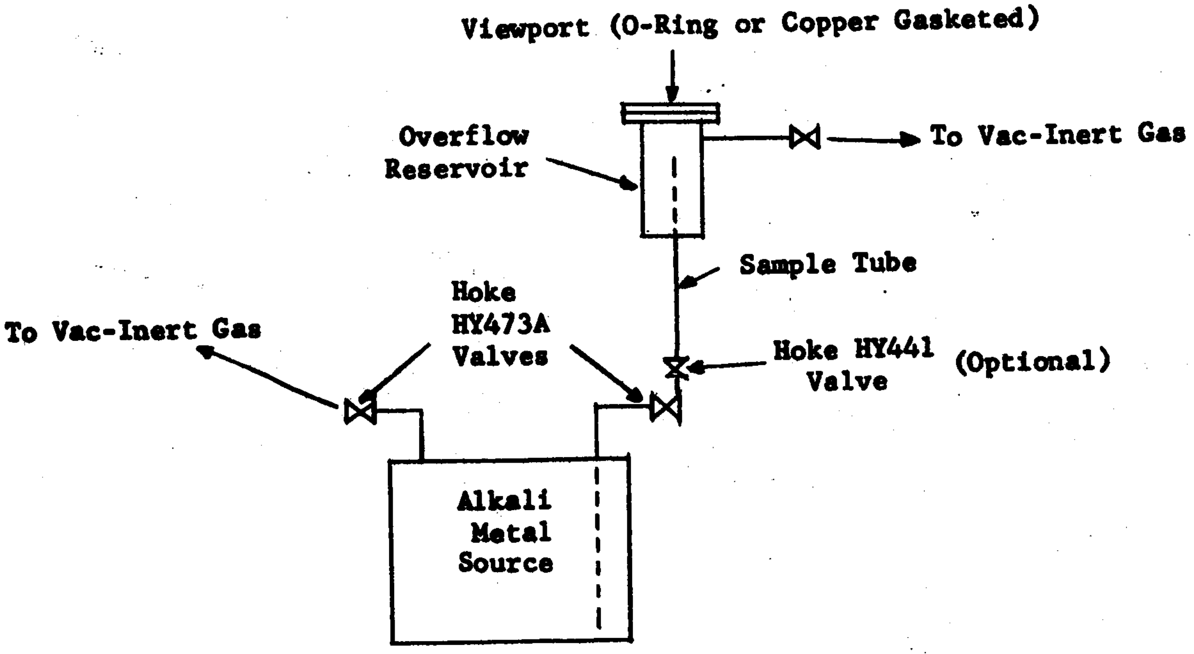
For both systems shown in Figure 3.7.3.1., all valves from which alkali metal must be cleaned are Hoke HY473A angle pattern, and the connecting fittings are either Swagelok, Gyrolok, Conoseal or equivalent type connectors. As shown, both systems are equipped with argon or helium and/or vacuum manifolds for obtaining a pure inert gas atmosphere or high vacuum for sampling.

3.7.3.2. Apparatus for Liquid Samples. The sampler from which liquid samples will be introduced into analytical apparatus will be placed in the same location shown in Figure 3.7.3.1. for solid samples. However, the liquid sampling containers must be completely isolated by valves after the sample is taken, and the valve which must be attached to the analytical apparatus must be cleanable up to the seat. Figure 3.6.1.5. shows the type of apparatus from which liquid samples will be taken.

It should be noted that the sampling criteria stated in paragraph 3.7.2. may be more difficult to meet when liquid samples are used, since the molten metal will more actively getter and dissolve contaminants present in the sample container. Therefore, extreme attention to cleanliness, of cover gas and sample tube, as well as to pressure rise rate under vacuum conditions, is necessary if the sample is to be introduced into the analytical apparatus in liquid form.



Sampling Rig Where Large Flush Quantities are Required



Sampling Rig Where Small Flush Quantities are Required

Figure 3.7.3.1. Alkali Metal Sampling Arrangements.

3.7.4. Sampling Procedures. The procedures used in taking samples for analysis will include preparation of apparatus and the operations required for obtaining representative samples for quantitative analyses.

3.7.4.1. Sample Apparatus Preparation

3.7.4.1.1. For Extrusion Sampling (Na and K). Referring to Figure 3.7.3.1. the following operations are required to prepare the sampling apparatus:

a. Degrease and pickle the sample tube, overflow reservoir and connecting fittings with 20% HCl for 10 to 15 minutes, rinse with water, acetone and dry.

b. Attach the sample tube and viewport cover to the overflow reservoir.

c. Attach the sampling assembly to the clean Hoke HY437A sampling valve or tee in the flush line and connect the inert gas-vacuum line to the gas valve on the reservoir.

d. Evacuate the sampling system to less than 10^{-3} torr and leak-check cold to meet the criteria stated in paragraph 3.7.2.

e. Heat the sampling system to 250-300°F (or higher, if necessary to obtain a representative sample) and outgas at less than 10^{-3} torr until the pressure rise rate meets the contamination criteria stated in paragraph 3.7.2.

3.7.4.1.2. For Liquid Sampling. Referring to Figure 3.6.1.5., the initial preparation (pickling) of the liquid sampling apparatus will be done as in paragraph 3.7.4.1.1., except that this system is designed for high vacuum, high temperature use. The sample tube proper is welded to the overflow reservoir and to a Hoke HW441 valve on the bottom. Thus, pickling and cleanup of the interior surfaces of the system will be done during fabrication.

Following assembly of this system, a helium leak-check is required with a maximum leakage of 5×10^{-10} std. cc/sec of air allowed.

Attach the sampler to the alkali metal source and outgas at less than 10^{-5} torr at the temperature at which the sample will be taken until the pressure rise rate meets the previously specified requirements.

3.7.4.2. Sampling Operations. With sampler attached, the sampling procedure will be as follows:

a. Flush the transfer line and sample tube with the required volume of metal at the required temperature. This operation may be done under high vacuum or pure inert gas for either extrusion or liquid samplers.

b. For extrusion samples, pressurize the sampler to 5 psig, cool to room temperature, remove the sampler and cap the end exposed to air.

c. For liquid samples, close the valve on the bottom of the sampler, pressurize to 5 psig, cool to room temperature and remove the sampler from the source. Clean the valve on the bottom of the sample tube to the seat as specified in paragraph 3.10.1.

3.8. Monitoring

3.8.1. Definition. Monitoring, as used in this specification, is the procedure(s) by which the purity of the alkali metal in use in a system is assured.

3.8.2. Purpose. The purpose of monitoring procedures is to maintain the impurity concentration levels in the alkali metals below certain specified limits.

3.8.3. Justification. Justification of the effort, time and money required by a monitoring program is based on the known or suspected deleterious effects which certain impurities may have on the structural integrity and/or operation of facilities or equipment using alkali metals as working fluids. Some specific effects due to contaminants are given below; however, insufficient data are available to ascertain whether or not certain impurities are harmful, and as a consequence, the overall policy is to use liquid metals of the highest practical purity in order to avoid the problem as far as is possible.

3.8.3.1. Deleterious Effects Due to Impurities

3.8.3.1.1. Oxygen. A number of effects have been noted which are due to oxygen contamination in the alkali metals.

3.8.3.1.1.1. Plugging. When the temperature in a portion of a system drops to a point where the solubility limit of a dissolved impurity is exceeded, precipitation will occur and may cause a plug. The oxide may be either the alkali metal oxide or a more complex oxide such as sodium columbate.

3.8.3.1.1.2. Corrosion. It is known that alkali metals containing oxygen react with containment materials such as stainless steel, nickel, columbium, etc. and with titanium and zirconium in a way which may lead to the dissolution or spalling of the metal surfaces. Such attack may weaken the containment material and/or yield particulate matter which may plug or abrade various components.

3.8.3.1.1.3. Mass Transfer. The presence of as little as 50 ppm oxygen in sodium has been associated with the mass transfer of carbon in austenitic and ferritic steel systems. It is also known that columbium may be transferred as a columbate when oxygen is present. Stainless steel loops circulating sodium at 650°C show dissolution losses to be ten times as great at 30 ppm oxygen in sodium as for 5 ppm oxygen in sodium.

3.8.3.1.1.4. Embrittlement. The presence of oxygen may lead to oxygen embrittlement in various alloys, especially refractory alloys.

3.8.3.1.2. Carbon. At least two effects have been noted which are associated with carbon in alkali metals.

3.8.3.1.2.1. Carburization. It has been noted that as little as 40 ppm carbon can lead to very rapid carburization of stainless steels at temperatures in the vicinity of 1200°F provided there is a source of carbon present. When no source is present, carburization ceases but the carbon content does not decrease drastically - perhaps to only 20 or 30 ppm. The mass transfer of stainless steel is also increased by the addition of carbon (10 to 15 ppm) to sodium.

3.8.3.1.2.2. Reduction of Getter Efficiency. The formation of carbide on the surfaces of titanium or zirconium getters in alkali metal systems has been found to reduce the efficiency of the getter for the removal of oxygen.

3.8.3.1.3. Nitrogen. Nitrogen contamination in the alkali metals can also lead to reduction of getter efficiency for oxygen removal by the formation of nitrides on titanium and zirconium surfaces. Nitrogen can lead to embrittlement of various metals. Sodium and potassium do not normally dissolve nitrogen to any great extent; however, lithium does.

3.8.3.1.4. Hydrogen. The presence of hydrogen in alkali metals can lead to hydrogen embrittlement, especially of refractory metals. However, the test temperatures of systems constructed of these materials generally exceed the decomposition temperatures of the hydrides of most materials and the hydrogen diffuses out of the system, so it is generally not a problem.

3.8.3.1.5. Calcium. The presence of calcium in alkali metals has been associated with nitrogen transfer and has lead to a less than 50 ppm specification on Reactor Grade Sodium.

3.8.3.1.6. Metals. The presence of other metals as contaminants in the alkali metals is generally associated with the presence of oxygen. Since these oxides are not detected by the usual analytical methods for oxygen in the alkali metals and since they may cause oxygen transfer or plugging it is necessary to analyze the alkali metals for other metals.

3.8.3.2. Purification Efficiency. Monitoring the impurity levels before and after purification operations, such as cold trapping or hot trapping, will indicate how effective the purification step was, and thus provide the operator with information which is of value in determining whether the cold trap or hot trap is saturated, or whether the temperatures or flow rates through these devices should be altered.

3.8.4. Monitoring Procedures. The specific procedure by which the impurity levels of the alkali metal(s) in a particular system will be monitored is the responsibility of the Program Manager; however, it will be necessary to comply with the following essential requirements.

3.8.4.1. Lithium, Sodium, Potassium, and NaK

a. Analyze the as-received metal, before transfer to the system, for oxygen and metallic impurities, and carbon also when it is of particular concern. Lithium must also be analyzed for nitrogen in addition to oxygen and metallic impurities.

If it does not meet the quality specification, it must be purified or replaced.

b. If the alkali metal must be purified, it must be re-analyzed afterward.

c. When a system is capable of being flushed prior to operation it should be flushed and the alkali metal re-analyzed for oxygen and metallics; and repurified or replaced if necessary. This process of flushing, purification and analysis must be continued until the impurity levels of the alkali metal fall below the maximum limits specified. Lithium must also be analyzed for nitrogen.

d. Whenever, an accident occurs or components are changed in a way which could possibly lead to the exposure of the alkali metal to the atmosphere or other contaminants, the alkali metal must be re-analyzed and repurified or replaced if necessary.

e. In the case of systems where the exposure of the alkali metal to oil or other contaminants is possible, analyses should always be made for this contaminant also.

f. The alkali metals used in working systems, such as heat transfer, turbine, and bearing and seal test rig loops should be analyzed periodically for oxygen and metallic impurities, and in the case of lithium for nitrogen. Such periodic analyses will generally be performed between testing periods. They will provide information on the condition of the loop such as excessive corrosion and leakage between primary and secondary circuits. Continuous oxygen monitoring devices such as plugging meters may be used when required for a particular facility.

3.8.4.2. Rubidium and Cesium. If rubidium and cesium are used in loop systems, the same monitoring procedures should be used as for the other alkali metals (see previous section). However, when they are used in such devices as ion engines, it is generally impractical to analyze the metal after its introduction to the feed system, but its purity should be established prior to the introduction. Historically, the vendor's analysis has been accepted and this practice will be continued except when there is some reason to suspect that the alkali metal has been contaminated. If it is discovered that contaminants which would be deleterious to the engine or the feed system are present, the metal should be purified or replaced.

3.8.4.3. Static Systems. Static systems include such devices as capsules, wick boilers, the alkali metal wetting study apparatus, etc. The purity of alkali metals used in such systems must be established before introduction to the device. When an inert gas chamber or vacuum chamber is used during the transfer of the metal, a sample should be obtained in the chamber during the transfer, under conditions which are as similar to the transfer conditions as is practical. The sample container should be sealed in the chamber and the sample analyzed to determine the quality of the metal placed in the device.

3.8.5. Maximum Allowable Impurity Concentrations. Several programs use alkali metals which are specially purified, for which the purity requirement is extremely high and for which special purification and purity specifications have been written. All other programs should maintain the purity of the alkali metals in use so that they comply with the following specifications.

3.8.5.1. Lithium. Specification 01-0030-00-A High Purity Lithium Metal, Table I.

3.8.5.2. Sodium. Specification 01-0032-00-B, Hot Trapped Reactor Grade Sodium Metal, except that the maximum allowable concentration for oxygen and carbon will be 50 ppm each.

3.8.5.3, Potassium. Specification 01-0034-00-B, Hot Trapped High Purity Grade Potassium Metal, except that the maximum allowable concentration for oxygen and carbon will be 50 ppm each.

3.8.5.3. Eutectic Nak. Specification 01-0050-00-A. Hot Trapped High Purity Grade Eutectic NaK except that the maximum allowable concentration for oxygen and carbon will be 50 ppm each.

3.8.5.4. Rubidium. Specification 01-0051-00-A High Purity Grade Rubidium Metal.

3.8.5.5. Cesium. Specification 01-0052-00-A, High Purity Grade Cesium Metal.

3.9. Handling

3.9.1. Definition. Handling, as used in this specification, refers to the methods and techniques used to transfer alkali metals from one container, apparatus or device to another and to determine the quantity transferred.

3.9.2. Purpose. The purpose of this section is to describe methods by which alkali metals may be transferred from one container to another with a minimum of contamination, and the ways in which the quantity transferred may be determined. It is not the intention to limit the methods used solely to those described here, but instead to provide those involved with alkali metals with several procedures which can serve as guides.

3.9,3. Transfer Methods

3.9.3.1. Transfer in Air. It is sometimes desirable to transfer small quantities of solid alkali metal at low temperatures without resorting to complex handling systems. This can be done by using an inert cover liquid of high purity which does not dissolve appreciable quantities of atmospheric constituents such as nitrogen, oxygen, carbon dioxide and water vapor. Hydrocarbon liquids such as high purity mineral oil or spectrographic grade hexane are generally used for this purpose. When the proper care is exercised, it is possible to handle alkali metals for brief periods under such liquids without excessive contamination. Care should be taken that these flammable liquids do not catch fire. Also, alkali metals which may be coated with peroxides or superoxides must not be placed in such solvents since they may react violently with such active oxides.

This technique is not recommended for use with liquid alkali metals. Also, it should be remembered that lithium is less dense than the hydrocarbon liquids and so will float.

3.9.3.2. Transfer in Inert Gas and Vacuum Chambers

3.9.3.2.1. Inert Gas. Inert gas chambers or glove boxes are often used to facilitate the transfer of both liquid and solid alkali metals. If it is important not to contaminate the alkali metal, it is generally concluded that the inert gas should contain no more than 1 molar ppm of any particular active impurity, and that even then the exposure time should not exceed a few hours. It is not adequate to put high purity gas in the chamber and use it statically because outgassing of the chamber walls and diffusion of the atmosphere through the gloves contaminates the gas to unacceptable levels within a few minutes. A continuous purification system should be used through which the gas is recirculated at a rate which has been proved to be rapid enough to maintain the gas at the acceptable impurity level.

3.9.3.2.2. Vacuum. Alkali metals may be transferred in a vacuum chamber using remote handling techniques at pressures up to 10^{-5} torr provided the exposure time is not too long. Capsule filling operations which required less than two hours at about 10^{-5} torr have resulted in oxygen contamination to the extent of 5 to 10 ppm on occasion.

A pressure of 10^{-5} torr is equivalent to an inert gas containing about 10^{-2} molar ppm of impurities. The reason that one can use an inert gas of much greater impurity level is that the rate of contamination is slowed because it is necessary for the impurities to diffuse to the surface of the alkali metal.

3.9.3.3. Transfer Using Inert Gas Pressure. This is the most used method for transferring liquid alkali metals. The technique is, in essence, that of pushing the liquid metal from one container to another through a connecting pipe. Usually the empty container is evacuated while the full container is pressurized to a few psig. Containers, valves and piping must be fabricated of corrosion resistant materials such as stainless steel, columbium, tantalum, etc. Valves should be of the bellows sealed, packless type; and if disconnects are to be made after the passage of alkali metal through them, they should have an angle pattern and should be oriented so that they may be cleaned back to the valve seat using the procedure described in paragraph 3.10.1 of this specification.

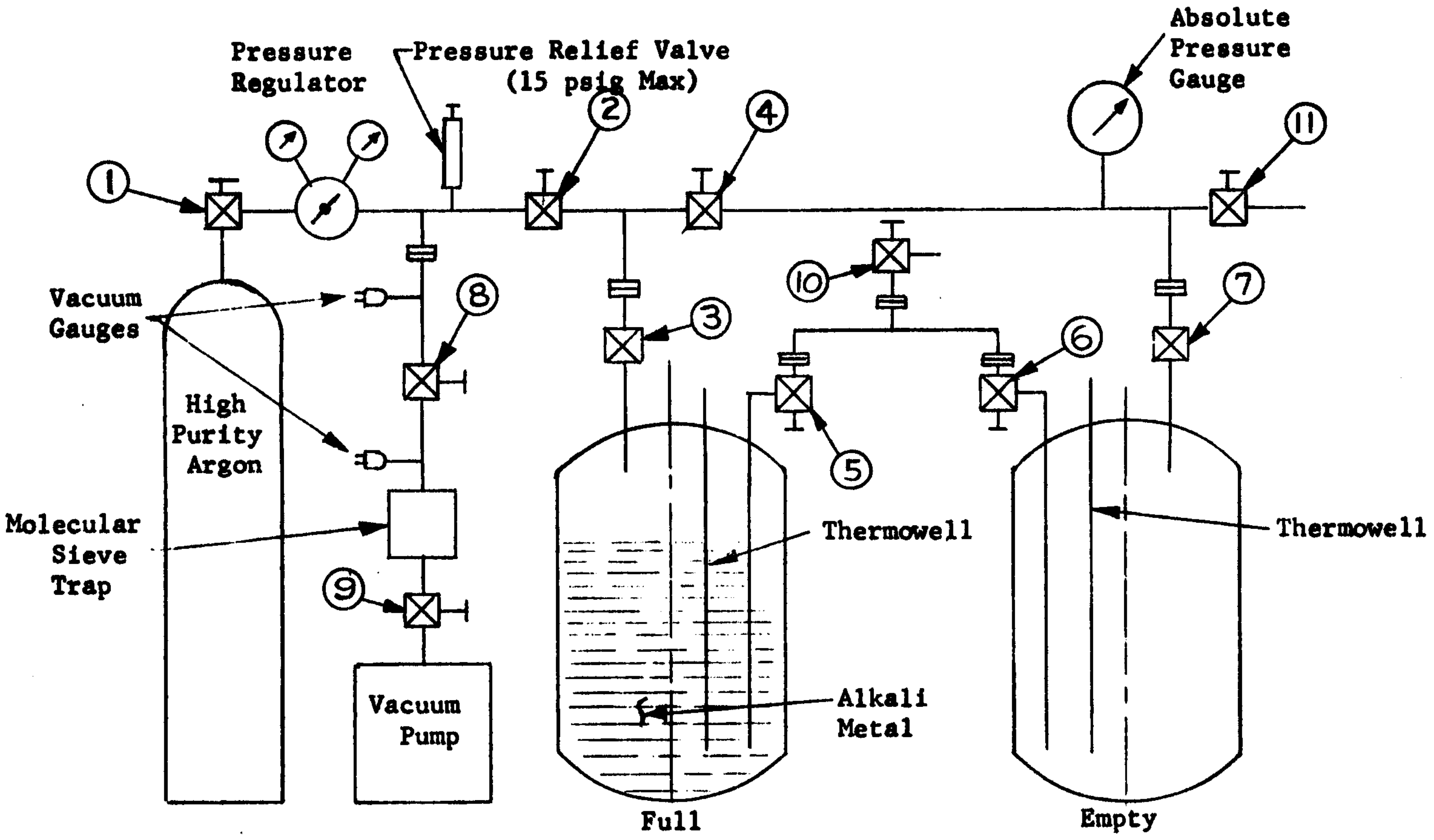
For critical applications where the highest purity is required, all parts of the system must be carefully cleaned by degreasing, pickling, rinsing with distilled water and drying under conditions which do not recontaminate the component prior to assembly. The components must be connected using approved welding techniques such as those described in Specifications 03-0005-00-A and 03-0014-00-A, or connecting joints which will meet the critical leak specification of less than 5 x 10^{-10} std. cc of air per second at the intended use temperature. After assembly, the complete system should be evacuated, baked out at a temperature above the intended use temperature until the pressure rise rate is less than 0.1 micron-liter of gas per gram of alkali metal to be transferred over the anticipated transfer time. The entire system should then be helium leal-checked according to Specification 03-0013-00-A.

A schematic of a generalized alkali metal transfer system for high purity alkali metal is shown in Figure 3.9.3.1. Valves 5, 6 and 10 are angle pattern bellows sealed valves oriented so that they may be easily cleaned back to the seat after contamination with alkali metal. All other valves, except valve 1, are also bellows sealed valves. The pressure relief valve provides protection against inadvertent overpressurization. The molecular sieve trap prevents contamination of the system by back diffusing of oil vapor. It should be baked out under vacuum with valve 8 closed before use. Valve 10 permits the transfer line between valves 5 and 6 to be evacuated and leak checked when the empty container has been used previously and may contain some alkali metal. Valve 11 permits leak checking of the system and may also be used to evacuate the transfer line between valves 5 and 6 through valve 10. The vacuum gauges should be compatible with the ultimate vacuum and pressure rise rate desired. Obviously, they should be ionization gauges, trigger discharge gauges or equivalent if a diffusion pump or getter-ion pump is used and should be backed up with a thermocouple gauge for measuring higher pressures.

The procedures to be used are given in the following paragraphs.

3.9.3.3.1. Leak Checking. Connect a mass spectrometer helium leak detector of adequate sensitivity to valve 11, close valve 11 and leak check the connection according to Specification 03-0013-00-A (Critical Applications). If the empty container is clean, open all valves except 1, 3, 5, 9 and 10, heat all components to between 200° and 250°C, evacuate and leak check according to Specification 03-0013-00-A (Critical Applications). Close valve 11 tightly and disconnect the leak detector.

Valves such as 10 and 11 which may not be leak checked across the seat should be tested before use, while closed and, ideally, should be back filled with argon and capped after disconnecting the leak detector.



╪ Indicates Swagelok, Conoseal, TIG weldment or other approved connection.

FIGURE 3.9.3.1. Transfer System for Use with High Purity Alkali Metals, Including the High Purity Inert Gas Supply and Vacuum System.

3.9.3.3.2. Outgassing. Connect valves 10 and 11 and open all valves except 1, 3 and 5. Evacuate through valve 9. Heat all components except valves 1 and 9, the pressure regulator, the pressure relief valve, the molecular sieve trap and the full container to between 200° and 250°C. When pressure has dropped to about 1 micron, close valve 8 and bake out the trap. Cool the trap, open valve 8 and continue bakeout and evacuation until the pressure rise rate, measured with valve 8 closed, is below the maximum allowable rate. Close valve 8, bake out the trap and close valve 9.

3.9.3.3.3. Transfer of Metal. At this point it is assumed that the entire system has been evacuated except for the full container which is presumed to be under a few psig of argon. Heat the full container, the empty container and the transfer line, including valves 5, 6 and 10 to the transfer temperature. Usually the transfer temperature should exceed the melting point of the alkali metal by about 50°F.

Close valves 7, 10 and 11, make sure that valve 8 is also closed, and remove the connection between valves 10 and 11. Valves 3 and 5 are already closed. Close the pressure regulator valve and open valve 1, then set the pressure regulator to give a system pressure of about 25 psia; then open valve 3. This will provide a pressure in the full tank of about 25 psia. If the pressure is too high, bleed down through valve 11 to the atmosphere, or through valves 8 and 9 by using the vacuum system forepump.

Close valve 2, then open valve 5 and transfer the desired quantity of alkali metal to the empty container and close valves 5 and 6.

Close the pressure regulator valve and open valves 2 and 7; then open the pressure regulator valve and pressurize both containers to a few psig; then close valves 3, 4, 5, 6, and 7. Cool to room temperature.

Disconnect above valves 5, 6 and 7. Clean valves 5, 6 and 10 immediately according to paragraph 3.10.1 of this specification. The reasons that the valves should be cleaned immediately is that the process is easier before the alkali metal becomes oxidized and that peroxides can form if exposed for long periods. The presence of peroxides is dangerous because they may react explosively with the underlying alkali metal or with the hexane and alcohol used during the cleaning operation.

It is generally easier to discard the transfer line; however, it must be cleaned by using one of the methods described in paragraph 3.10.2. of this specification.

CAUTION: If cesium, rubidium or an alloy like NaK which is liquid at room temperature is involved, the transfer line must be cleared before disconnecting it from the containers. Liquid alkali metals almost always catch fire when exposed to the atmosphere. The heat of reaction of cesium and rubidium with air is usually sufficient to liquify them.

The transfer line may be cleared by evacuating both tanks with valves 3 and 7 open and valves 5 and 6 closed, then closing valves 3 and 7 and blowing down with argon through valves 10 and 11 to clear valves 5 and 6.

3.9.4. Determination of Quantities Transferred

3.9.4.1. Transfers in Air. Rough determinations may be made of the quantity transferred using the inert liquid cover transfer method by weighing the receptacles before and after the transfer; however, an error is involved since the weight of the liquid adhering to the metal remains unknown. Where the surface area to volume ratio is small this error may be insignificant.

When this method is involved during the chemical analytical techniques, it is generally possible to determine the sample weight chemically.

3.9.4.2. Transfers in Inert Gas and Vacuum Chambers. When making transfers of alkali metals in inert gas and vacuum chambers the quantity transferred may be determined gravimetrically with a balance for solids and liquids, or volumetrically with a burette, hypodermic syringe, volumetric flask, graduated cylinder or some other volumetric device for liquids.

3.9.4.3. Transfers Using Inert Gas Pressure. A number of methods are available for determining the quantity of alkali metal transferred. They are indicated in the following paragrahs.

3.9.4.3.1. Gravimetric. Referring again to Figure 3.9.3.1., either the full or empty container may be weighed during the transfer operation by placing it on a scale. When this is done the transfer line should be flexible enough to permit the container to move without restraint.

Either container may be weighed before and after the transfer. In this case, one of the volumetric methods described in the next section should be used to estimate the quantity transferred during the transfer operation.

3.9.4.3.2. Volumetric. In all but one of the following methods, it is essential to know the change in internal volume as a function of height for the particular container involved. If it is necessary to know the mass transferred, then the temperature and the change in density with temperature must also be known.

3.9.4.3.2.1. Thermal Gradient Method. This is the simplest of the methods to be described. It consists of determining the vertical thermal gradient of some part of the container which is in contact with the alkali metal and through which heat may be transferred to the environment. Generally this is a thermowell such as is shown in Figure 3.9.3.1. A thermocouple is inserted into the thermowell and a temperature versus depth profile is developed. This profile will show a different rate of change of temperature with depth above and below the alkali metal surface and the point of intersection of the projections of these two lines defines the alkali metal surface.

This method is not applicable when the temperature difference between the alkali metal and the environment is less than about 200°F because the difference in the thermal gradients above and below the surface is not sufficiently great to give a sharp indication of the point of intersection.

3.9.4.3.2.2. Thermal Conduction Methods. This method is simple and is almost universally applicable. It consists of determining the point on some component of the container, which is in contact with both the alkali metal and the space above it, at which the thermal conduction through the wall of the component changes abruptly. For example, if a pipe which is partially filled with alkali metal is heated (preferably in the dark) in the vicinity of the alkali metal-free space interface it will become red hot opposite the free space first and the interface will be indicated by a sharp change between red hot and dark on the pipe wall. The same method may be applied to the outer wall of most containers. Obviously, the method fails when temperature equilibrium is reached and the system is isothermal.

Another method consists of inserting a small heater or cooler into a well which penetrates the alkali metal surface. A thermocouple is attached to the heater or cooler and makes sliding contact with the well wall. A temperature versus depth profile is developed and the liquid metal surface is defined as the point at which the rate of change of the slope of this curve is a maximum.

Still another method consists of sliding a thermowell containing a thermocouple through a double O-ring sealed feedthrough until contact with liquid is indicated by an abrupt change in temperature. This method is only applicable when an appreciable temperature difference exists between the alkali metal and the environment. It is not recommended for extremely high purity applications.

3.9.4.3.2.3. Electrical Contact Method. This method consists of inserting an electrically conducting probe through a sliding seal which electrically insulates the probe from the metal container until contact with the metal surface is indicated by the fact that the resistance between the probe and container suddenly drops from a very high value to a negligible value. The make point gives a more accurate determination of the level than the break point because surface forces cause the liquid metal to cling to the probe and form a conducting path even after the probe tip is above the liquid metal surface. Double O-ring seals have been used successfully for the insulating sliding seal; however, they are not recommended where the highest purity must be maintained. Stainless steel bellows, of the type used in ultrahigh vacuum valves could be used in high purity systems. The probe, in this case, would be an extension of a high vacuum, ceramic to metal electrical feedthrough. The bellows must be long enough to provide a distance between minimum and maximum extension which is equal to the maximum level change of interest. Also, the bellows should be given adequate support to prevent over-extension or inadvertent contraction during pressure changes in the container; e.g., a screw drive could be used. This method is not applicable when alkali metals may deposit on the insulating region, causing a short.

3.9.4.3.2.4. Electrical Resistance Methods. These methods are based on the change in electrical resistance of some conductor, which penetrates the surface of the liquid metal, as liquid level changes cause more or less of the conductor length to be out of contact with the metal; i.e., a change in resistance as more or less of the conductor is shorted out through the liquid metal to the container. The simplest method consists of measuring the resistance between the metal container and a wire which enters the container through a seal which insulates it from the container. The wire must be long enough to reach a point just above the bottom of the container. Raising or lowering the liquid metal level will then result in a corresponding change in the exposed length of the wire which will, in turn, produce a change in resistance. This technique has three serious disadvantages. The first is that incorporation of a ceramic or glass to metal seal in an alkali metal system limits the operating temperature and pressure because the insulating region may either react with the metal vapors or be rendered conducting by metal deposits. Second, such insulating seals are fragile and may be broken during operation thus exposing the metal to the atmosphere. Third, ideally, the wire should be of small diameter to give a large resistance per unit length; however, the metal may wet the wire and the conduction of the surface layer of liquid metal may have a significant, but unknown, effect on the measured resistance.

3.9.4.3.2.4.1. "J" Type Liquid Level Probe. The disadvantages indicated above which are due to the use of an insulating seal are avoided by the use of the "J" type probe.

3.9.4.3.2.4.1.1. <u>Description and Method of Use</u>. The complete level measuring system consists of three individual components:

a. Probe

b. Control and Monitoring Circuitry

c. Constant Voltage D.C. Power Supply

A drawing of the probe and a simplified electrical schematic is shown in Figure 3.9.4.1. The probe consists of three concentric conductors separated by ceramic insulating material except at the end which is exposed to liquid metal. At this end, all conductors are welded together in a fashion which completely eliminates exposure of the interior of the probe to liquid metal or to metal vapors. All layers are swaged (before welding the end) to give a tight, evenly supported and easily bendable probe length. After welding the end, the tube package is bent in the form of a "J" and welded into a flange which can be welded directly into the liquid metal container.

Copper leads are attached to the individual conductors at the exposed end,and connections between these leads and the control circuits are made with a total of nine individual conductors as shown in Figure 3.9.4.2.

Current is made to flow through the center tube (Figure 3.9.4.1.) and it passes through the welded tip into the outer tube (sheath) and then through the outer tube into the liquid metal which shorts that part of the tube which is submerged.

The IR drop across the sheath is measured and is proportional to the height of the liquid. The center conductor is used as a voltage lead and carries no current. The probe sheath and center conductor are set up in a bridge circuit so that the temperature-resisivity effects are self compensating. Figure 3.9.4.2. shows the circuit as it actually exists.

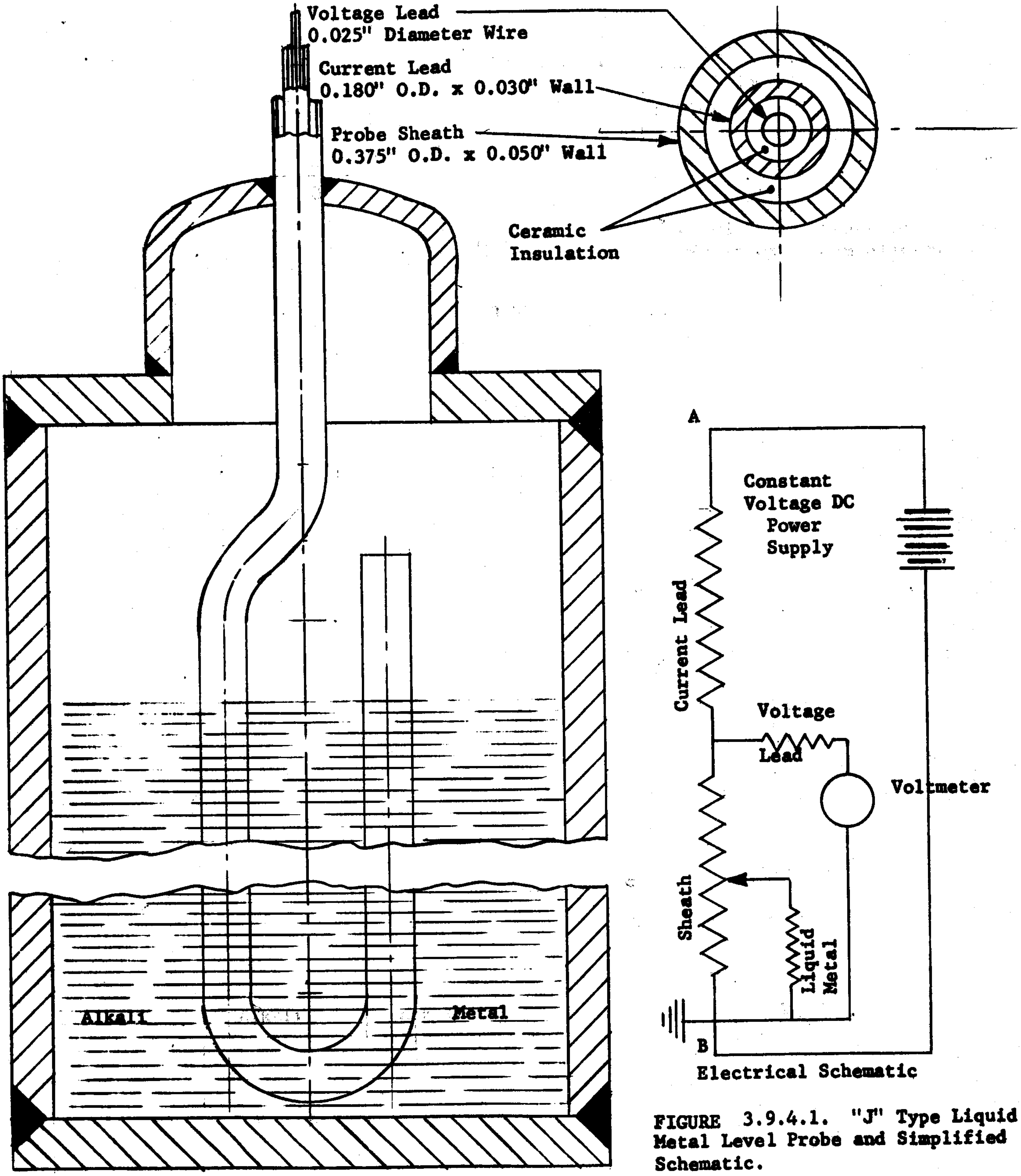
A constant D.C. voltage is maintained across points A and B by the power supply, so that the output voltage is proportional only to the height of liquid metal. The output voltage must be measured by a high impedance/null balance instrument to minimize or preferrably eliminate loading effects.

The control and monitoring circuit contains a 500-ohm, 10-turn potentiometer which may be used to adjust the signal level. It also contains terminal strips for access to output and input signals.

The D.C. power supply (a Harrison Lab Model 802B has been used) provides excellent load regulation at low voltage and has provision for remote sensing which is necessary to eliminate effects of lead wire voltage drops on voltage across the probe.

3.9.4.3.2.4.1.2. <u>Interconnecting Wiring</u>. Figure 3.9.4.3. shows the recommended wiring between the probe, the control circuit and the power supply. It is essential that all wires to be connected to the probe be grouped together and soldered to the copper leads at the exposed end of the probe at one location. Other types of configurations can be used, but temperature compensation may suffer.

3.9.4.3.2.4.1.3. <u>Calibration</u>. It must be realized that this type of level measuring system is not able to define an absolute level, but will provide a repeatable indication of <u>change</u> in level. Therefore, the probe must be calibrated in place after an initial soaking period at temperatures high enough to insure complete wetting.



Electrical Schematic

FIGURE 3.9.4.1. "J" Type Liquid Metal Level Probe and Simplified Schematic.

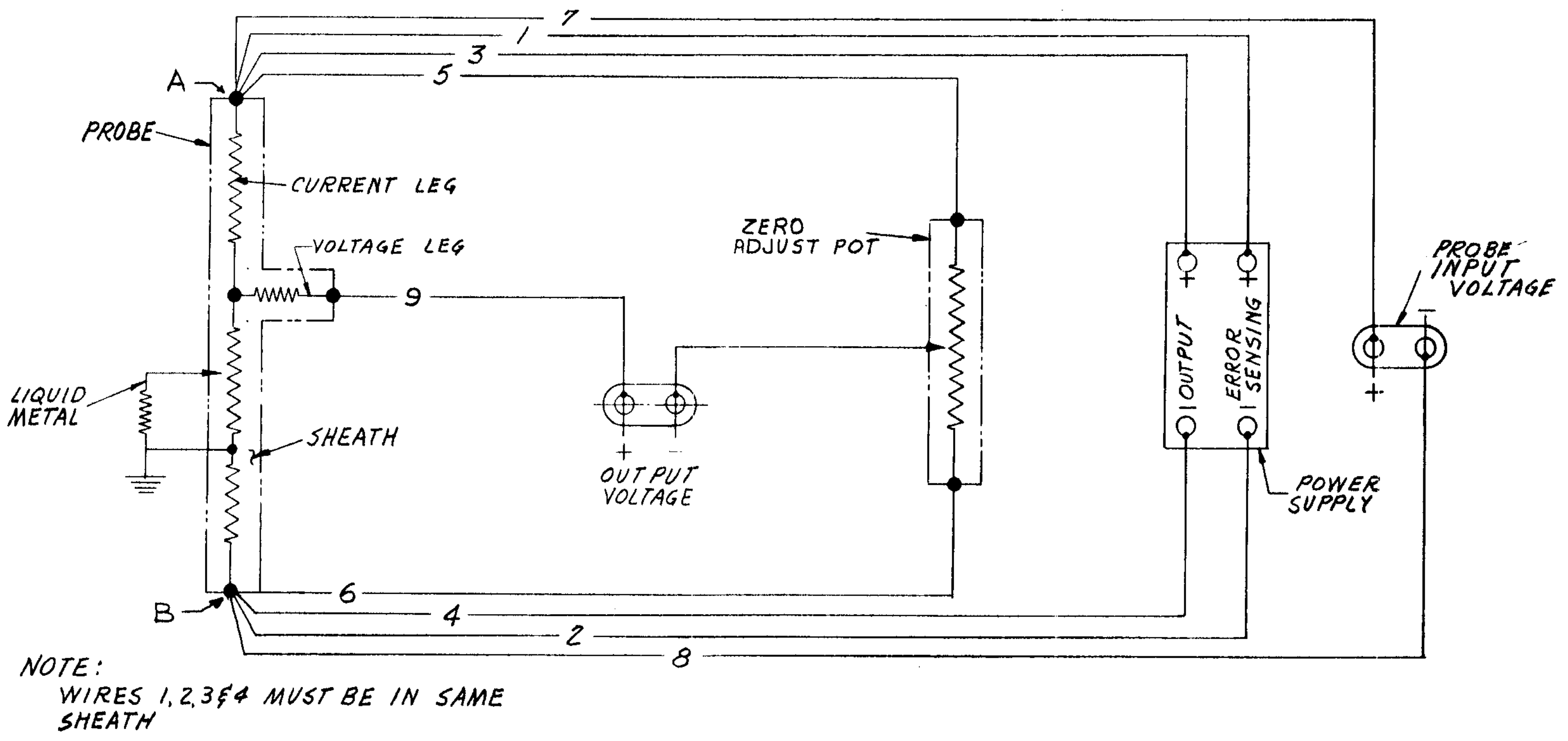


Figure 3.9.4.2. "J" Type Liquid Metal Level Probe - Electrical Schematic.

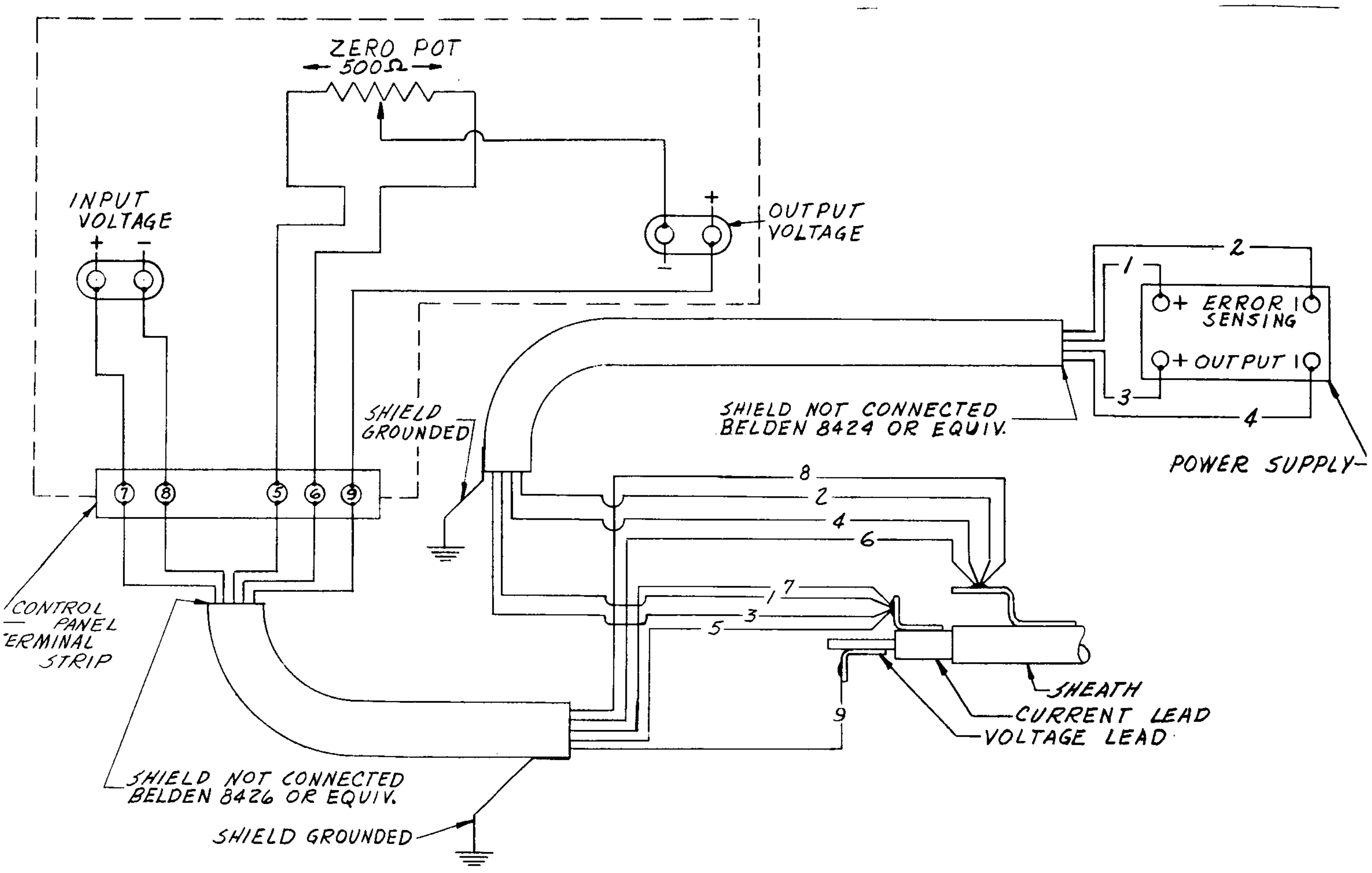


Figure 3.9:4.3. "J" Type Liquid Metal Level Probe - Electrical Schematic.

The quantity which changes as the level is varied is the effective resistance of the probe sheath as it is shunted by the liquid metal. The calculated resistance of the sheath at 1000°F is 0.000787 ohm per inch of length for the configuration shown in Figure 3.9.4.1. when fabricated from L-605 alloy. Using maximum current of (for the 802B power supply) 1.5 amps gives a theoretical sensitivity of 1.18 millivolts per inch change in level. There are a number of factors which tend to decrease this sensitivity and the following procedure will give an average sensitivity over the length of the probe.

a. After all connections have been made and the probe has been soaked at temperature to insure wetting, adjust the current by means of the power supply controls to 1.4 amps with the tank full of liquid metal (probe completely submerged).

NOTE: The values given below are valid for a 1.4-amp D.C. current through the probe. They will change if the probe current is increased by means of a bigger power supply.

b. Measure the input voltage at the control box terminals. This value will be approximately 100 millivolts.

c. Adjust the output voltage to a convenient level (approximately 10 millivolts) with the 10-turn potentiometer.

d. Lower the level in the tank until a sharp change in output voltage is noted. This occurs when the level drops beneath the bottom of the probe and the current path is doubled.

e. Raise the level in the tank until the liquid metal just touches the bottom of the probe. The output voltage at this point should be measured after the input voltage has been checked and adjusted to the original value, if necessary, using the power supply controls.

f. The change in voltage over the active length of the probe has now been determined. This should be linear.

g. It is possible to perform this operation in reverse order starting with the liquid metal in contact with the bottom of the probe. However, initial current setting should be somewhat less than 1.4 amps, since it will increase slightly as the level is raised, and it is important that the 1.5-amp rating of the power supply not be exceeded.

Theoretically, the only requirement for complete temperature compensation is that there be no temperature gradient across the cross section of the probe. Axial gradients along the probe are not harmful. However, due to the low level of signals, there are several factors which can cause shifts in output with temperature. Most prominent of these, are thermoelectric emfs between copper and L-605 junctions and liquid metal.

During operation it is necessary to adjust the probe input voltage from time to time to compensate for long term drift characteristics of the power supply. The entire circuit is based on maintaining a constant input voltage at the probe and any change in this voltage is reflected in the output signal.

For purposes of emphasis, it is necessary to measure all voltages with null balance or high impedance instruments to avoid loading effects on the circuit.

The sensitivity is directly proportional to the current through the probe and may be increased many times by using a bigger power supply. The only limit to the current through the probe is the heating of the probe elements. It is estimated that this would be insignificant up to 50 amps for the configuration shown in Figure 3.9.4.1. It is also possible to use an A.C. power supply, but practical types of read-out instruments would not be as accurate.

3.9.4.3.2.5. Electrical Induction Methods. The coefficient of self-inductance or simply the self-inductance, L, of a solenoid is a function of the relative permeability, Km, of its environment. $Km = \frac{\mu}{\mu_o}$, where μ and μ_o are the magnetic permeabilities of a material and a vacuum respectively. Consequently, when the environment of a solenoid is changed its self inductance will change. For example, if a small solenoid is placed inside a metal tube which penetrates a liquid metal surface and its self-induction is measured as a function of depth of insertion into the tube, it will be noted that a change in self-induction takes place as the solenoid moves past the liquid metal - free space interface. Such probes must be calibrated in actual liquid metal systems. Furthermore, the tube must be fabricated from a material which is diamagnetic or paramagnetic not ferromagnetic; and if a paramagnetic material is used its magnetic susceptibility should be lower than that of the alkali metal if possible.

Certain manufacturers market liquid metal level indicators which make use of this phenomenon. Mine Safety Appliance Research, Callery, Pennsylvania is one such manufacturer.

3.9.4.3.2.6. Radiation Methods. Beams of gamma rays are attenuated when they pass through material objects,and the amount of attenuation depends directly on the path length and the density of the material. If a radiation source is placed on one side of a liquid metal container and a radiation detector on the other side, the radiation which reaches the detector will be less when the container is full than when it is empty, and with properly positioned detectors it is possible to determine the location of the liquid metal surface.

Certain manufacturers market liquid level indicators which are based on this concept. One such manufacturer is The Ohmart Corporation, Cincinnati, Ohio.

3.9.4.3.2.7. Pressure-Volume Method. This method makes use of the ideal gas law, PV = nRT, to calculate a change in volume by determining a change in pressure. For example, if P_1 and V_1 represent the pressure and volume respectively of the gas in a container held at constant temperature and P_2 and V_2 represent the pressure and volume after the container is partially filled with a liquid without removing any of the gas, then nRT is constant and $P_1V_1 = P_2V_2$. It is then possible to calculate V_2 if P_1, V_1 and P_2 are known. The change in volume of the alkali metal in the container is $V_2 - V_1$. Referring to Figure 3.9.3.1., assume that the empty container is partially or completely evacuated after which valve 7 has been closed. Also, assume that the full container has been pressurized to P_1 as indicated on the absolute pressure gauge and that valves 2 and 11 are closed. The initial volume, V_1, is the free volume in the full container, V_{cl}, plus the volume of the gas manifold between valves 2, 11 and 7, V_{gm}, plus the volume of the pressure gauge, V_{pg}; i.e., $V_1 = V_{cl} + V_{gm} + V_{pg}$. After some liquid metal has been transferred to the empty container via valves 5 and 6, the pressure in the full container will be P_2 and the free volume of this container will now be V_{c2} and $V_2 = V_{c2} + V_{gm} + V_{pg}$. If the gas manifold and full container temperatures are equal and did not change during the transfer, then

$$P_1(V_{c1} + V_{gm} + V_{pg}) = P_2(V_{c2} + V_{gm} + V_{pg}) \tag{1}$$

Since V_{gm} and V_{pg} are both constant, let

$$V_{gm} + V_{pg} = K$$

Then

$$P_1(V_{c1} + K) = P_2(V_{c2} + K)$$

and

$$V_{c2} = \frac{P_1V_{c1}}{P_2} + \frac{(P_1 - P_2)K}{P_2} \tag{2}$$

Obviously, if K is small with respect to V_{c1} and V_{c2}, the second term on the right of the equation (2) may be neglected. Also, if K is small, the temperature of the inert gas manifold and valves need not be exactly the same as that of the full container.

The same method may, of course, be used to find the quantity of alkali metal added to the empty container.

This is the only volumetric method described which does not depend on a knowledge of the variation of volume with depth for the particular container used in order to calculate the volume of metal transferred.

It should be pointed out that this method may be used to find the free volume in any container. It is only necessary to allow the gas from a known volume to expand into the evacuated unknown volume. The two volumes should be comparable in size for the greatest accuracy.

3.9.4.3.2.8. Measured Volume Method. This method consists of equipping the transfer system with a tank (between valves) which has an accurately known volume, and from which the alkali metal may be displaced completely. This arrangement permits a known volume to be transferred from the tank to the facility. Gas pressure or gravity may be used to effect the transfer.

3.10. Miscellaneous Operations

3.10.1. Valve Cleaning Procedure. The Hoke HY473A valve is designed to permit complete removal of all alkali metal downstream from the valve seat. The location where the valve is cleaned will be free of water and moist surfaces. The cleaning procedure normally used for solids is as follows:

a. The person cleaning the valve should wear protective clothing, safety glasses, face shield and gloves. The valve MUST be cleaned immediately after use. Do not look into the valve at any time unless protected by a face shield and safety glasses.

b. Using a clean, dry drill bit of the largest diameter which will fit the access tube, remove the bulk of the alkali metal by drilling carefully down to the valve seat by hand. Remove the drill bit from the valve and scrape the metal off of the drill with a screwdriver or spatula (for example) before proceeding further.

c. Remove the metal from the fitting on top of the valve as completely as possible, using a screwdriver or spatula. If the alkali metal starts burning during steps a through c, let it burn itself out.

d. Fill the valve with practical grade hexane if--and only if--the valve is cool and there is no evidence that residual alkali metal is burning.

If a fire should develop during steps d through g, extinguish with a CO_2 fire extinguisher. CAUTION: Do not use a CO_2 fire extinguisher to extinguish alkali metal fires. In steps d through g, the fire will be due to hexane and alcohol which have been ignited by the reaction of the alcohol with very small quantities of residual alkali metal. Throwing sand on such a fire may cause splattering of the flaming hexane-alcohol mixture.

CAUTION: Do not leave bottles of hexane, alcohol or CO_2 fire extinguishers in an area where there is the possibility of an alkali metal fire. For example, do not leave the hexane and alcohol where the valve is. Also, only small quantities of hexane and alcohol are required. About 100 ml of each in separate, LABELED, polyethylene squeeze bottles should be sufficient. Larger quantities should be stored in an area which is cool and free of fire hazards.

Those portions of the cleaning operation which result in the generation of hydrogen by the reaction of ethanol with the alkali metal will be performed in an exhaust hood or other well-ventilated area to avoid accumulation of explosive mixtures of air and hydrogen.

e. Add ethanol to the hexane drop by drop until the hydrogen evolution becomes reasonably vigorous. As the hexane evaporates, add more hexane and alcohol to keep the valve full. Continue adding alcohol until no bubbling is observed in the solution.

f. Remove the hexane-alcohol-ethoxide solution and add fresh hexane and alcohol to the valve. If bubbling occurs, continue adding alcohol until all alkali metal is reacted.

g. Remove the solution from the valve again; an eyedropper is normally used. Add pure alcohol to the valve CAUTIONSLY. Swab the valve interior with cheesecloth until the alcohol and all solid material are removed.

h. Finally, add water to the valve when assured that no free metal remains. Add a few drops of 0.1-1.0 N HCl to the water to neutralize any basic solution.

i. Remove the acid solution from the valve and dry the valve thoroughly.

The cleaning procedure for liquid alkali metals is essentially the same except that the liquid may be poured out or sucked out of the valve rather than drilled out. Moreover, it is the general practice to drain or blow down such valves before disconnecting them from the system.

3.10.2. General Decontamination and Cleaning Procedures

3.10.2.1. Alkali Metal Containers. Containers such as shipping containers, hot traps, surge tanks, charge pots, still pots and receivers, etc. which contain alkali metals which must be removed may be cleaned by first transferring the bulk of the alkali metal to a disposal drum or shipping container, using one of the techniques described in paragraph 3.9 of this specification, then cutting the container open and completing the cleaning by using one of the following methods.

3.10.2.1.1. Steam Cleaning. This is the fastest method of getting rid of residual alkali metals. It consists of placing the component in a specially constructed room, usually called a "burn room", and then spraying the interior of the component with high pressure steam. The steam reacts with the alkali metal yielding the hydroxide and hydrogen. After conversion, the hydroxide is washed away with water. The air flow through the room is high enough to prevent the overall hydrogen-air ratio from eaching the explosion limits, although locally one may have hydrogen fires or small explosions.

This method is not recommended for delicate parts which may be damaged by the high local temperatures which may develop. Furthermore, it is not generally recommended for metals, such as columbium, which may become hydrogen embrittled; however, if the part is massive enough and the quantity of alkali metal is relatively small, the hydrogen uptake may be insignificant during the short time required for the cleaning operation. Appropriate protective clothing should be worn when applying this method. Stainless steels are subject to stress corrosion cracking when the steam temperature exceeds 150°F.

3.10.2.1.2. Reaction with Alcohols. Residual alkali metals may be removed from containers using alcohols such as ethanol or isopropanol. The part to be cleaned is first filled with hexane, kerosene or some other inert solvent, or it may be placed in another container containing such a solvent. The alcohol is then added slowly with agitation until all of the alkali metal has been converted to the alkoxide. It is preferable to perform this operation under a flowing inert gas cover to minimize the possibility of hydrogen fires and explosions.

When this method is used, it is essential that the alkali metal be freshly exposed so that the surface is not covered with peroxides or superoxides. Such oxides may react violently with organic materials such as the solvents and alcohols used.

This method permits the reaction which produces hydrogen to be controlled so that low temperatures may be maintained. It should be remembered that lithium metal will float on the solvents used, so it is essential that an inert cover gas be used and that the solvent-alcohol mixture be well agitated. The alcohol is best added through a pipe which reaches below the solvent surface in this case.

Fires which occur during the application of this method will generally be solvent fires and may be extinguished with a CO_2 extinguisher. CO_2 should not be used on burning alkali metals since they react to form the oxide and CO.

Appropriate protective clothing should be used while using this cleaning method.

3.10.2.2. Piping, Etc. Pipes, tubes and other similar types of plumbing which contain alkali metal should be drained as much as possible before final cleaning. They may then be cleaned, using techniques similar to those described in paragraphs 3.10.1., 3.10.2.1.1. and 3.10.2.1.2.

Another technique which may be used for short lengths of pipe, elbows, tees, etc. consists of placing them inside a container which may be evacuated to low pressures (about 10^{-5} torr) and heated to a temperature which is high enough to achieve a reasonable evaporation rate for the alkali metal concerned. The container must contain a condensing surface which operates at a temperature below the freezing point of the metal being distilled. The alkali metal may then be distilled from the component to the condensing surface. The rate at which such distillations take place will be dependent on the conductance of the component and the distillation system, and on the vapor pressure of the alkali metal.

3.10.2.3. Delicate Components. Delicate components should not be steam cleaned except as described below. Instead, the methods described in sections 3.10.1, 3.10.2.1.2 and 3.10.2.2. should be used. The method selected will depend on the shape, size and materials of construction of the components. It is quite possible that for some components none of these methods will be applicable, and will require the development of a new technique.

Certain components which permit the passage of gas through them may be cleaned by blowing argon or helium through them at a rate sufficient to keep them cool. Then steam or water vapor may be added to the flowing gas stream at a low rate. The inert gas will prevent hydrogen-air explosions by dilution of the hydrogen produced by the reaction of the alkali metal with the water vapor. One disadvantage is that it is difficult to know when all of the alkali metal has been converted to the hydroxide.

3.10.2.4. Vacuum and Inert Gas Chambers. Massive spills of alkali metals inside of vacuum or inert gas chambers may be partially cleaned up by back filling the chamber with an inert gas, opening the chamber and scooping up the metal and placing it in a bucket along with dry sand, then washing off the residual metal with water and finally a wet cloth, providing the residual amount is not too great.

Some large chambers have been equipped with water sprays and drains which may be used to convert the alkali metal to the hydroxide and wash it away under a flowing inert gas cover.

Another method which is especially useful for removing alkali metal surface deposits consists of flowing CO_2 - H_2O vapor mixtures through the chamber. Reactions occur which ultimately produce the carbonate which may be removed by washing away with water.

In the case of chambers that may be heated, the distillation and condensation method described in paragraph 3.10.2.2. may be used to advantage.

3.11. Disposal

3.11.1. Disposal Methods

3.11.1.1. Small Quantities. Small quantities of alkali metal such as that remaining in sample tubes are best disposed of by taking them to the "Burn Room" and converting them to the hydroxide with steam. Such quantities should be kept under inert gas cover until they can be transported to the "Burn Room".

Such quantities may also be disposed of by reaction with alcohols in an inert solvent as described in Section 3.10.2.1.2.

3.11.1.2. Large Quantities

3.11.1.2.1. Return to Vendor. Most vendors of alkali metals will dispose of alkali metals returned to them in shipping containers. Generally they make a small charge per pound for this service.

3.11.1.2.2. Burning. Alkali metals may be disposed of by burning in air; however, this requires a sizeable area far removed from human habitation, highways, etc. Such areas are not generally available. Much caustic smoke is produced during the process and this must be prevented from coming into contact with people, animals, houses, machinery, streams, etc.

Smaller quantities may be burned or hydrolyzed in a "Burn Room" which is equipped with a scrubber. The quantity disposed of during a given interval must not exceed the capacity of the scrubber for removing the oxide smoke prior to discharge to the atmosphere.

3.11.1.2.3. Hydrolysis. Large quantities of alkali metal may be disposed of by reaction with water, either in an isolated body of water such as a quarry or at sea. The method consists of opening or puncturing the container before or after throwing it into the water. Generally, a hydrogen explosion will take place in a short time which will tear the container to pieces, thereby bringing all of the alkali metal into contact with the water. The container may be punctured initially after throwing it into the water with a high-powered rifle or a small explosive charge.

Much smoke is generally produced and the explosions are violent, so this operation must be performed at a considerable distance from habitation, public roads, etc. and should only be performed by personnel who are familiar with the hazards involved.

3.11.1.3. Reaction of Alkali Metals with the Atmosphere. Although alkali metals which are exposed to the atmosphere for long periods will eventually be converted to oxides, hydroxides and carbonates, this is not an approved method for their disposal. The reason for this is that most of the alkali metals become covered with peroxides and superoxides. Subsequent handling may bring these higher oxides into contact with the underlying unreacted metal at which time an explosion may take place.

4. QUALITY ASSURANCE PROVISIONS. Quality assurance for alkali metals is covered in the individual procurement specifications. These are listed in paragraph 2.2. of this Specification. (Also see paragraph 3.2. of this Specification.)

5. PREPARATION FOR DELIVERY. The preparation for delivery requirements for the alkali metals are covered by the individual procurement specifications.

6. NOTES. None

APPENDIX A

ALKALI METAL SURVEILLANCE RECORD

The following form, SP 1061, is to be associated with each lot of alkali metal which is purchased.

PAGE 1 OF 3

SPACE POWER & PROPULSION SECTION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

ALKALI METAL SURVEILLANCE RECORD

WA NO. SP 11107

A

REQUESTER: R. A. Fuller

DATE: 4/24/65

PHONE: 1764

VENDOR: MSAR

INTENDED USE: 300 KW Secondary

MATERIAL SPECIFICATION: 01-0033-00-B, except that the maximum limit on oxygen is increased to 60ppm K will be hot trapped prior to use.

APPROVED: R. B. Hand

B

PURIFICATION SPECIFICATION: 01-0034-00-B, except that the hot trappings will be performed at 1200°F

APPROVED: R. B. Hand

C

RECEIVING DATE: 5/21/65

MATERIAL DESIGNATION NO.: DRUM D-80

D

INTRA-PLANT TRANSFER RECORD

FROM	TO	RECIPIENT	DATE
Bldg 200 Dock	Bldg 309 Storage	R. B. Hand	5/22/65
	Bldg 314 Analytical	L. E. Dotson	5/24/65
	Bldg 309 Storage	R. B. Hand	5/26/65
	300 KW Facility	R. A. Fuller	5/30/65
	Bldg 309 Storage	R. B. Hand	6/25/65
	Bldg 200 Dock	G. Van Walde	6/28/65
	Vendor for Disposal	MSAR	6/30/65

SP 1061

ALKALI METAL SURVEILLANCE RECORD - CONTINUED

WA NO. SP11107

E

VENDOR ANALYSIS

See attached certificate of compliance. Vendor claims that value for lead is due to contaminated analytical reagent. J. L. King, Buyer

F

INTERNAL ANALYSIS

INTERSTITIALS

O_2 as K_2O = 65 ppm

C = 3 ppm

ANALYSIS NO. 206

REMARKS Carbon analysis by Kallman Ledoux and Company — NO 743550, 5/28/65, PO # 030-122697

PERFORMED BY L. Paian

DATE 5/25/65

METALLICS

Na	35	Ca	15	Si	10
Sn	3	Mn	<1	Ti	2
Pb	<1	Cu	5	Al	5
Co	2	Ag	<1	Zr	<10
Fe	10	Cr	<1	Mo	<1

ANALYSIS NO. 304

REMARKS

PERFORMED BY R. Yoder

DATE 5/27/65

PAGE 3 OF 3

ALKALI METAL SURVEILLANCE RECORD - CONTINUED

WA NO.

G

REMARKS

Vendor high value fm lead evidently in error

L. E. Dotson

H

USE APPROVAL

SIGNATURE

DATE 6/29/65

REMARKS 60 ppm O_2 max per 01-0033-00-B is waived since K will be hot trapped prior to use.

SP 1061 B

APPENDIX B

AUTHORIZED ALKALI METAL STORAGE AND USE AREAS - PERMISSIBLE QUANTITIES AND OPERATING CONDITIONS

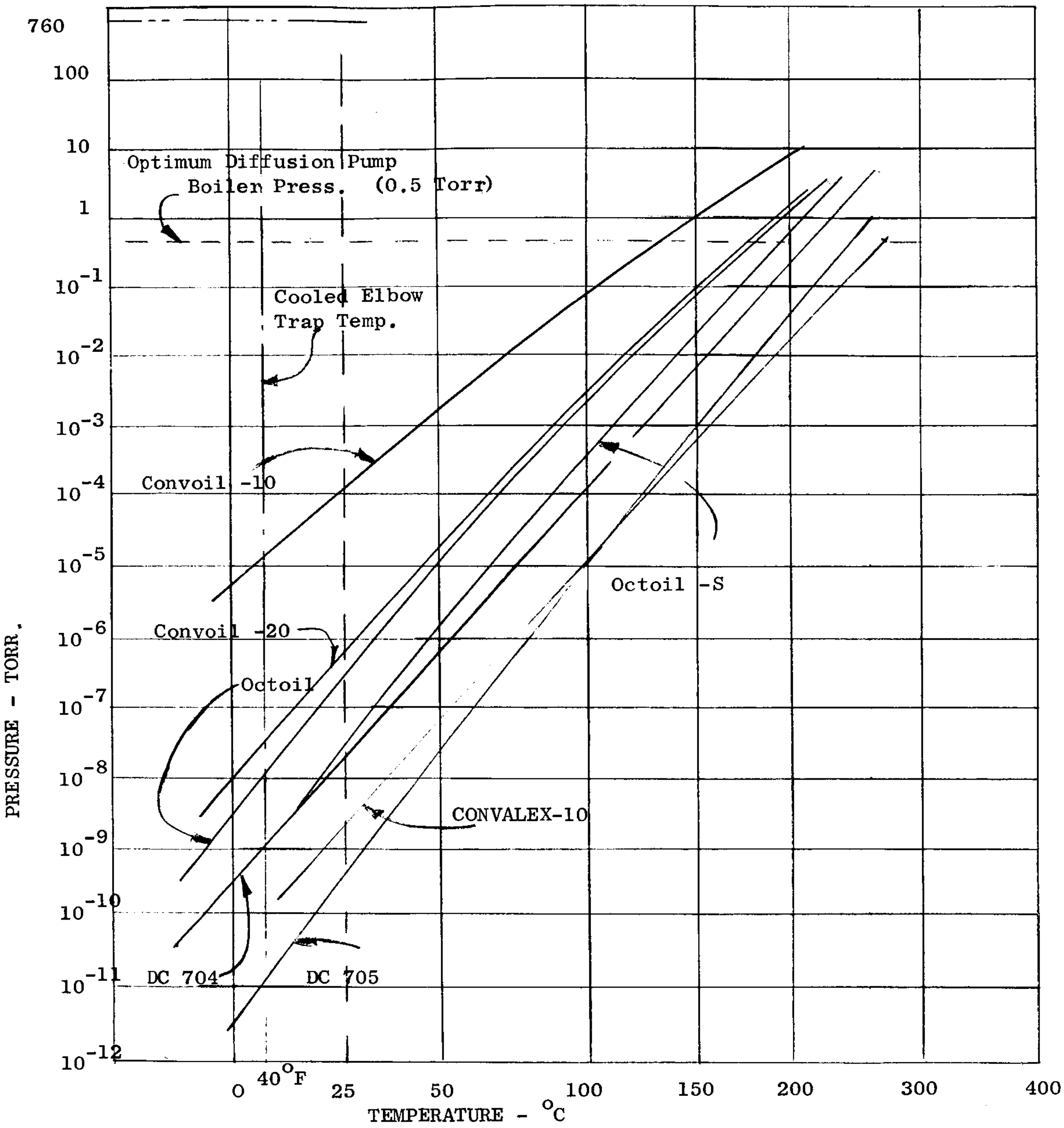
The following table shows the locations where alkali metals may be used, the maximum quantities permitted, and the extremes of temperature and pressure allowed in each. No other locations are authorized, except that access corridors may be used to transport alkali metals if they are at room temperature.

AUTHORIZED ALKALI METAL AREAS

Bldg. No.	Area Designation	Maximum Quant. - lbs.		Operating Conditions Upper Limits	
		In Use	Stored	Press. - psig	Temp. - °F
309	East Bay	1000	2000 (Permanent)	150	2500
	West Bay	1000	1000 (Temporary)	150	2000
	Control Room	None	0.5 (Temporary)	0	Rm. T.
314	North Bay	750	None	10	1500
	South Bay	400	None	150	2500
	Control Room	None	0.5 (Temporary)	0	Rm. T.
318	Facility	3500	None	20	1700
	Control Room	None	0.5 (Temporary)	0	Rm. T.
700	1st Floor - Area A	3	None	165	2500
	1st Floor - Area B	30	None	10	1500
	1st Floor - Area C	0.2	None (Temporary)	0	Rm. T.
	1st Floor - Area D	30	None (Temporary)	10	1600
	1st Floor - Fabrication	20	None	0	300
	1st Floor - Electric Propulsions Labs	2	None	0	700
700	2nd Floor - Physical Test Lab	0.2	None	0	1000
	2nd Floor - Analytical Chemistry Lab	0.5	1	0	300
	2nd Floor - Physical Chemistry Lab	0.5	None	10	1500
	2nd Floor - Ceramic Materials Lab	0.2	None	10	1600

VAPOR PRESSURE-FIRE POINT CHARACTERISTICS OF DIFFUSION PUMP FLUIDS

(Bechtel Corp., San Francisco, Calif., Oct. 31, 1963)



PUMP FLUID DATA	OCTOIL-S	OCTOIL	CONVOIL-10	CONVOIL-20	CONVALEX -10	DC 704	DC 705
vapor phase at 0.5 torr.	199°C	183°C	135°C	190°C	275°C (appx)	210°C	230°C
Boiler Liq. Temp.	204°- 214°C	188°- 198°C	140°- 150°C	195°- 205°C	280°- 290°C	225°-235°C	235°- 245°C
Molecular Weight	426.7	390.5	250	400	454 avg.	484	546
Pour Point	-56°C	-52°C	-23°C	-9°C	+5°C	-38°C	-15°C
Flash Point (open)	210°C	196°C	191°C	218°C	288°C	232°C	243°C
Fire Point	248°C	229°C	224°C	259°C	349°C	315°C	—

Figure 1. Vapor Pressure-Fire Point Characteristics of Diffusion Pump Fluids.

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