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Design, Fabrication and Testing of Porous Tungsten Vaporizers for Mercury Ion Thrusters

Ralph Zavesky and Erich Kroeger
Lewis Research Center
Cleveland, Ohio

and

Seiji Kami
Hughes Research Laboratories
Malibu, California

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Ralph Zavesky and Erich Kroeger
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

and

Seiji Kami
Hughes Research Laboratories
Malibu, California 90265

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SUMMARY

The dispersions in the characteristics, performance and reliability of vaporizers for early model 30-cm thrusters were investigated. The purpose of the paper is to explore the findings and to discuss the approaches that were taken to reduce the observed dispersion and present the results of a program which validated those approaches. The information that is presented includes porous tungsten material specifications, a discussion of assembly procedures, and a discription of a test program which screens both material and fabrication processes. There are five appendixes providing additional detail in the areas of vaporizer contamination, nitrogen flow testing, bubble testing, porosimeter testing, and mercury purity. Four neutralizers, seven cathodes, and five main vaporizers were successfully fabricated, tested, and operated on thrusters. Performance data from those devices is presented and indicates extremely repeatable results from using the design and fabrication procedures.

INTRODUCTION

Electron bombardment mercury ion thrusters have been under development since 1959. Their specific impulse in excess of 2000 seconds makes them significantly more fuel efficient than competing propulsion systems for high energy mission applications. Space flight tests have been conducted. The SERT I suborbital test of a 10-cm mercury ion thruster in 1964 proved the feasibility of the device (ref. 1). The SERT II orbital test (ref. 2) of two 15-cm mercury ion thrusters in 1970 demonstrated the long life capability. A technology readiness program was recently completed to demonstrate the readiness of the 30-cm mercury ion thrust subsystem which is useful for primary propulsion (ref. 3). A program is underway to perform a spaceflight test of two 8-cm mercury ion thrusters useful for auxiliary propulsion of spacecraft (ref. 4).

The vaporizer is one of the key components of mercury ion thrusters. It serves as a flow control device to meter the very low mercury propellant flow rates required for thruster operation. Flow as low as 0.045 gram per hour is required. A typical vaporizer assembly is comprised of a porous tungsten element, welded into a holder and heated by an external heater that is brazed to the holder (fig. 1). This paper will only discuss the porous

tungsten material, the design and fabrication of the porous tungsten welded into a holder, and the subsequent testing of that component. Feasibility of this design concept for control of mercury flow was verified by the excellent long life operation of vaporizers that was demonstrated during the SERT II program. However, during the 30-cm technology readiness program, large dispersions in the characteristics, performance and reliability of vaporizers were noted.

Flow varied during vaporizer calibration, 50-hour acceptance testing, and during thruster testing. There were numerous reported intrusions of vaporizers while running on thrusters. Flow blockage was caused by contamination (see appendix A). Variations between purchased lots of vaporizer material caused vaporizer flow rate dispersion.

This range of performance of early model 30-cm thrusters was investigated. It is not the purpose of this report to explore those findings in detail. It is sufficient to point out that the performance dispersion prompted design and processing changes which resulted in improved performance and yield.

This paper presents information on porous tungsten material specifications. A discussion of assembly procedures, and a description of a test program which screens both material and fabrication processes is presented. There are also five appendixes providing additional detail in the areas of vaporizer contamination, nitrogen flow testing, bubble testing, porosimeter testing, and mercury purity. The information presented will facilitate reliable and predictable fabrication of mercury vaporizers for future programs.

VAPORIZER DESIGN FEATURES

Before discussing the details of vaporizer materials and processes, it is worthwhile to review the major design features of a typical vaporizer and discuss their importance. Detailed design of vaporizers must account for the use of dissimilar materials that will undergo wide temperature excursions, typically 243 K (-30° C) to 723 K (450° C), repeatedly during useful life. Also the devices are assembled from refractory metals in various configurations using unusual processes that must be contamination free. Vaporizer temperature during operation is typically 573 K (300° C).

Mercury Flow Design Considerations

A typical vaporizer design shown in figure 1 consists of a porous tungsten plug that is electron beam welded into a tantalum holder. A heater is brazed to the outside of the holder. The feed system applies enough pressure to the liquid mercury to overcome the partial vapor pressure of the mercury and keep the liquid mercury in contact with the porous tungsten plug under normal operating conditions.

In normal operation, mercury does not wet tungsten and therefore the capillary forces prevent liquid mercury from entering or passing through the small pores of the porous tungsten. The pores are large enough to allow vapor mercury to flow through the porous tungsten. The amount of flow of vaporized mercury is primarily controlled by the temperature at the surface of the liquid mercury and the properties of the porous material. The following material parameters are of importance: (1) pore size of the tungsten,

(2) area of the plug, and (3) thickness of the plug. The intrusion pressure or pressure at which liquid mercury can be forced into the pores and eventually through the plug is controlled by the pore size and the fact that mercury does not wet porous tungsten.

Factors Affecting Intrusion Pressure and Flow

Porous tungsten was selected for the vaporizer material because it can be fabricated with a small pore structure, on the order of 1.5 microns. When coupled with the contact angle that mercury makes with tungsten, this allows the material to be used to block the flow of liquid mercury at pressures and temperatures consistent with propellant system requirements. Smaller uniform pore size provides higher intrusion pressure, but more resistance to vapor flow.

The design of a vaporizer starts with the mercury flow requirement for a particular application, and a selected temperature of operation. The transmission coefficient (see appendix B for definition) of the porous tungsten material must be known for a given material thickness. Knowing the flow required, the temperature range and the transmission coefficient, the area of the porous tungsten can be calculated.

Sizing of the area of the vaporizer is dependent on all of the flow being directed through the thickness of the plug. If some of the flow is allowed to take a short path as shown in figure 2, the transmission coefficient for the assembly would be higher and the amount of flow would not be predictable. In order to direct the flow through the plug the cylindrical outside surface of the plug must be sealed. This sealing is accomplished by "washing" the surface with an electron beam welder to close the pore structure. This 100 percent dense outside surface also improves the ability to join the plug to the holder by electron beam welding.

Thermal Mechanical Design Considerations

The ideal vaporizer thermal design requires the vaporizer plug to be the hottest component in the system. The heater placement is important to this requirement. The heater should be positioned so that the heat has a direct conductive path to the vaporizer plug as shown in figure 1.

Tantalum was selected as the plug holder material because its' coefficient of expansion of 2.00×10^{-6} per °C closely matches the value of 1.38×10^{-6} per °C of tungsten. Tantalum also has good ductility. Even with these favorable characteristics, strain relief was added to the design by thinning down the walls in the vicinity of the porous tungsten plug to reduce the stress on the porous tungsten due to welding and thermal cycling. Tantalum is relatively easy to machine, easy to electron beam weld to the porous tungsten, and can be brazed to stainless steel with brazes that are compatible with mercury.

Assembly and Testing Considerations

The design must consider the vaporizer screening tests. The subassembly of a porous plug welded into a holder is an important configuration that allows many reliability and performance tests to be accomplished. The design

must allow visual observation of the downstream side of the plug for bubble testing (see appendix C) and welding inspection. The holder should be adaptable to mounting in a nitrogen flow test apparatus (see appendix B for detailed discussion) and be capable of accepting a slip-on heater to measure mercury flow. The reason for the above design considerations is that it is economically advantageous to assess the condition of the vaporizer as early as possible before extensive fabrication resources are applied.

The design configuration also allows the assembly sequence to minimize contamination of the liquid side of the plug during fabrication. Figure 3 shows the electron beam weld of the plug into the holder, and shows how the liquid side of the plug is shielded from material that is evaporated when the electron beam weld is made.

The open pore structure on the liquid side of the plug is more important than the vapor side because it controls the liquid vapor interface. If a small amount of vaporized material from welding blocks a few pores on the vapor side of the plug, a new path of least resistance to flow will be found.

POROUS TUNGSTEN

Background

The porous tungsten material is the most important vaporizer element. Obtaining acceptable porous tungsten is more difficult than initially imagined. Manufacture of the sintered porous tungsten stock varies from vendor to vendor and contains many proprietary nuances or "trade secrets." Processes and people change so it is difficult to assure that a vendor will make repeatable batch-to-batch material.

Consequently, it was decided that the most practical method to obtain usable porous tungsten was to concentrate on being able to screen and select good material from bad material as purchased, rather than concentrate on determining all the production details of a given vendor who may change the product or personnel by the time the next batch is purchased. The information that follows then, is in that spirit; namely, purchase a quantity of porous tungsten from a number of sources, give the vendors maximum flexibility to use their own processes, and then, upon delivery, select usable material by inspection and test.

Material Requirements

Two different types of porous tungsten materials are required for the 30-cm thruster program. The neutralizer and cathode vaporizers were sized using 80 percent dense material. The flow rate required for the neutralizer at 573 K (300° C) is nominally 0.035 amp[1], and for the cathode is 0.1 amp. A nominal flow rate of 1.25 amps is required for the main vaporizer at 573 K (300° C). Because of this high flow rate 73 percent dense porous tungsten was selected for the main vaporizer in order to obtain a higher transmission coefficient and keep the diameter of the plug small for ease of fabrication and greater structural strength.

[1]One equivalent ampere equals 6.25×10^{18} mercury atoms per second or 7.48 grams per hour of mercury at room temperature.

Manufacturing Specifications

Table I shows the porous tungsten manufacturing specification. The specification was made broad enough to cover all known porous tungsten supplier processes. The following is a discussion of some of the items of the specification. The numbers in parentheses correspond to line items of the specification.

Powder (1). - The 4 to 5 micron average powder diameter was specified because most manufacturers use this size to make 80 percent dense material. The powder was classified to eliminate large agglomerates, and produce a material with a uniform pore size. A powder certification was requested with each batch of porous tungsten. The average particle diameter, the particle size distribution, the chemical analysis, and the lot number was required on the certification. A sample of the data received is shown in table II.

Powder shape (2). - The powder shape, spherical or angular, was left to the discretion of the manufacturer. Spherical powder requires extra processing and cost. Scanning electron microscope (SEM) pictures of porous tungsten material made from spherical powder show more uniform structure, but past experience showed no difference in vaporizer performance.

Density (3). - Powder size, shape, compaction of the powder, sintering time and temperature all affect the density. As the density increases, the pore size decreases, which causes the transmission coefficient to decrease, and the intrusion pressure to increase. The manufacturer should have the capability of measuring the density in an "as-sintered" or bulk state of the porous tungsten to a tolerance of ±1 percent. The density should also be measured on each delivered part, and meet the ±2 percent manufacturing specification. If the density is not held to these tolerances the material will yield a large dispersion of flow transmission coefficient from vaporizer to vaporizer.

Sintering (4). - Most manufacturers of porous tungsten consider the sintering time and temperature as well as powder compaction before sintering as company proprietary information. Some manufacturers mix the powder with a filler material that is used as a vehicle to obtain the proper compaction prior to sintering in order to obtain specific porous tungsten densities. The filler material is driven off during the sintering process, leaving only the porous tungsten. Sintering is done in vacuum or in a hydrogen atmosphere so that the tungsten does not oxidize.

Handling (5). - After sintering, the material is in the cleanest and probably best condition for vaporizer use. However, further processing is required to assemble the porous tungsten into a vaporizer component. The material should be handled with clean lint free gloves from this point on.

Machining (6). - Before the plug can be machined from the sintered raw material, it must be filled with a medium that acts as a backing or stiffener to the brittle porous tungsten. The filler material makes the porous tungsten machinable with standard type tooling. A single point cutting tool such as used on a lathe is the best, although the material has also been drilled successfully. Methyl methacrylate is commonly used because of the ease of infiltrating and removing it without residue at fairly low temperature. Copper has also been used as the infiltrant. The removal of copper is somewhat more difficult because of the higher temperatures required.

Polish etch (7). - All machining operations leave the surface of the porous tungsten with smeared over pores, as shown in figure 4. In order to

regain an open pore structure at the surface, the material is etched. Any acid solution (such as Murakami) that can remove the excess tungsten material is acceptable. This process is done with the filler material still in the porous tungsten. The material can be inspected from time to time under at least 10X magnification to determine the right amount of etching. The proper amount of etching has been accomplished when the machining marks in the surface disappear.

Filler removal (8). - Six to eight hours at 618 K (345° C) is used to remove the bulk of the methyl methacrylate, and 10 to 15 minutes in the hydrogen reducing atmosphere is used to clean the porous tungsten of any oxide residue that might be present.

Inspection-identification-packaging (9, 10, 11). - The items are self explanatory. It is good practice to carry the identification of the porous tungsten through the fabrication of the vaporizer.

Material Evaluation

An evaluation of the porous tungsten that was purchased to the above specificiations should be made prior to processing the material for assembly into vaporizers. Microscopic inspection at 5x to 40x magnification will reveal unacceptable surface defects such as cracks (fig. 5), chips, gross surface contamination (fig. 6), and improper surface etching (fig. 4). Acceptable material will not contain any of the above defects.

Approximately 10 percent of each batch of porous tungsten should be subjected to a scanning electron microscope (SEM) inspection at a magnification range of 50x to 2000x. This test is primarily used to evaluate the bonding of the tungsten powder during the sintering operation. Some examples of acceptable bonding of both 73 and 80 percent dense porous tungsten are shown in figures 7 and 8. The powder particles are heavily fused to a point that it is hard to distinguish the individual powder particle. Some examples of unacceptable bonding of 80 and 58 percent dense porous tungsten material are shown in figures 9 and 10. The powder particles are not fused or joined to each other as in the acceptable bond. The SEM can also be used to further investigate surface contamination particles (fig. 11), and density variations (fig. 12).

PROCESSING

Processing includes the procedures and screening tests that are required to complete the fabrication and testing of a porous tungsten vaporizer to a state where the plug has been successfully welded into a holder. Processing steps are covered by detailed documentation. Following are comments about some of the key parameters.

The 30-cm thruster vaporizer processing sequence is shown in table III. In-process inspections and screening tests are used to identify unacceptable units as soon as possible. The numbers in the parenthesis in the titles below refer to steps in the procedure outlined in table III.

Cleaning vaporizer parts (2). - All of the tantalum parts of the vaporizer are vacuum baked at 1273 K (1000° C) for 1 hour as a cleaning and outgassing procedure to remove contaminants prior to assembly.

Porosimeter testing (4). - Every porous tungsten vaporizer plug is porosimeter tested as a nondestructive test to investigate the internal structure. See appendix D for a detailed discussion of porosimeter theory and testing. Basically, a porosimeter test measures the porous tungsten internal void volume which is open to the outside surfaces. A plot is made of the amount of mercury filled void volume against the pressure at which the voids were filled. Because of the known contact angle of mercury with tungsten the pore size can be directly related to the pressure. An acceptable range for porosimeter data is shown in figure 13.

The reasons for the shape of the cross hatched area are:

A. In the range marked A, materials with large pores are minimized by limiting the total porosity. This criteria rejects material that would have low intrusion pressure.

B. The range marked B is the area where all the pores (1.5 to 1.0 microns) should lie for acceptable material. This area determines where the gross intrusion will take place 827 kPa to 1379 kPa (120 to 200 psia). An ideal curve would be a straight vertical line at 827 kPa (120 psia).

C. The pores in the range marked C would give the material a high intrusion pressure but would limit vapor flow. If material in this region was used, the transmission coefficient would be low, and the plug area required would be large. The porous tungsten should have low percentage of pores in this range.

Examples of a porosity determination for acceptable materials are shown in figure 14. Unacceptable material is shown in figure 15.

The porosimeter test results (void volume and total volume) can also be used as a check on the density of the material that was measured by the porous tungsten manufacturer. The porosimeter curve also shows the pressure at which total intrusion of the material will take place.

Porosimeter testing cannot determine if there is a large pore path that is connected through the thickness of the plug. For instance a piece of material that is in the form of a donut could appear as acceptable material in a porosimeter test, because the hole is not distinguishable.

Machining the outside diameter (6). - The porous tungsten is usually received in the shape of a plug, with approximately a 0.25 mm (0.010 in.) larger outside diameter than specified in the vaporizer design. This requires the outside diameter to be turned to size the plug, and smear the outside edge material. The smeared material is desireable because it improves the electron beam wash of the outside diameter of the plug.

Electron beam washing (11). - The electron beam washing of the outside surface of the plug is required to close off short flow paths, and force the flow of mercury through the plug as previously discussed. The electron beam washing is made just heavy enough to close the pores. The electron beam wash is done with the plug mounted between two copper heat sinks. The assembly is rotated as shown in figure 16. The electron beam is focused at the center of the outside surface. The plug is slowly heated by the beam until it has just started to melt the tungsten. The beam is moved to an outside edge of the plug where melting is started. The beam is then swept across the plug melting the outside surface. After the beam reaches the other edge, it is again moved toward the center of the plug and shut down. This procedure should leave the plug with sides that are square, not rounded, as shown in figure 17. The outside diameter of the plug should not be reduced by more than 0.0254 mm (0.001 in.).

Vacuum firing (15). - Because the porous tungsten was subjected tc possible contaminants in the previous operations the plugs are vacuum fired at 1923 K (1650° C) for 1 hour. The time and temperature were selected because the temperature is high enough to remove most of the contaminants (even oxides) but not high enough or long enough to cause major changes in the tungsten sintering.

Electron beam welding (17). - Kerslake (ref. 5) has shown three methods of electron beam welding the porous tungsten to the holder. The method shown in figure 18 is the preferred method. The counter-bore depth in the holder is about 0.254 mm (0.010 in.) less than the thickness of the plug. The weld is made by heating both parts, and then melting the tantalum with a light weld into the tungsten, while the parts are being rotated with the beam directed as shown in figure 18. A heat sink is clamped to the plug holder close to the weld in order to reduce the tantalum thermal expansion.

Bubble testing (19, 22). - This is the first screening test that is used in the procedure. It is used to inspect the integrity of the weld and the plug after welding. For a detailed discussion of bubble testing, see appendix C.

Thermal cycling (20). - The thermal cycling test is included to verify that the plug and weld can withstand the cyclic stress.

Nitrogen flow test (23). - Kerslake (ref. 5) showed the correlation between nitrogen flow and mercury flow through a vaporizer plug. A nitrogen flow test was used to verify that the transmission coefficient is satisfactory. He also showed that the most accurate method of calibrating vaporizers is with mercury flow. Because of the difficulty associated with measuring small increases in pressure, the nitrogen flow method of calibration should only be used as a screening test for material acceptance. However, it is a very useful test when used for this purpose. For a detailed discussion of nitrogen flow testing see appendix B.

Furnace firing in dry air. - The furnace firing in dry air that was used for SERT II vaporizers to oxidize the plug, was eliminated from the 30-cm thruster procedure. The mono-layers of oxide that are formed at near ambient conditions are adequate to assure the mercury does not wet the tungsten.

Assembly (25). - The plug holder assembly that has passed all previous tests is now assembled into a complete vaporizer. The final screening tests are done on complete units. Note that testing up to this point has not involved mercury.

Mercury Intrusion testing (26). - The reservoir system as shown in figure 19 must be tested for "compressibility" before each mercury intrusion test. The flow calibration tube is filled by opening the valve to the reservoir. The valve is closed when the mercury level has reached a satisfactory level. The test is started by slowly increasing the pressure in 34.5 kPa (5 psi) increments to 118.4 kPa (15 psi), and recording the flow tube level after each increase in pressure. The volume of mercury in the calibration tube should not change by more than 0.0015 ml.

If the system compressibility is satisfactory, the intrusion test is started by slowly increasing the pressure in 118.4 kPa (15 psi) increments. The incremental pressure increases are reduced to 34.5 kPa (5 psi) above 517 kPa (75 psi), and the test is terminated if 862 kPa (125 psi) is reached. The pressure should be maintained at each level for at least 30 seconds without the level in the flow tube changing. If the flow tube level continues to change after 30 seconds, the intrusion limit has been reached.

Another method for determining intrusion pressure has also been used. After the system compressibility test has been satisfied, the pressure is

increased in increments as in the standard intrusion test, but the pressure is also removed after reaching a new pressure level and holding for 30 seconds. The reservoir level is then compared to the original reservoir level to determine the volume of the plug that has been intruded. This second method is preferred because it yields data for a plot of volume of intrusion against pressure which shows the characteristics of intrusion.

Mercury bake-out (27). - After intrusion testing, the mercury must be removed from the plug to restore it to a usable condition. This is done by removing the pressure on the system and heating the vaporizer to 623 K (350° C) for 30 minutes with a plug in a vacuum environment of less than 10^{-5} torr.

Mercury flow calibration (28). - The equipment, shown schematically in figure 19, that is used to calibrate vaporizers and to measure mercury propellant flow rate to thrusters has been a source of confusion in determining vaporizer flow rates and mercury utilization of thrusters. Three areas that have given variable vaporizer flow characteristics are: (1) leakage in valves and fittings; (2) air in mercury lines; and (3) size and/or calibration of flow measuring tubes.

Valves and fittings should be helium leak checked when the system is assembled. Valves selected should be a type that have zero displacement when they are actuated. If the valve does displace mercury when operated, it will mechanically force mercury into the porous tungsten causing intrusion.

Valve designs should be such that they minimize the possibility of trapping air. Both valves and fittings should have the capability of being pressurized without changing volume or allowing mercury to enter a cavity at pressures up to 1034 kPa (150 psi).

Air in mercury lines is probably the biggest source of error in vaporizer flow calibration. A "compressibility" test as previously described in the section "Mercury Intrusion Testing," should be repeated before each flow calibration.

Mercury flow is usually expressed in cubic centimeters per hour or in equivalent amps. The value that is commonly used to correlate the two units for vaporizer calibration is 1.810 amp-hr/cm^3.[2]

The glass flow tubes are purchased with precision bores. The glass tubes that are used to measure the mercury flow are purchased with a ±0.01 mm (0.0004 in.) tolerance on the bore diameter. The bore should either be measured or the tube should be calibrated by filling with mercury, draining a known amount, and weighing the dispensed mercury. After confidence has been achieved in the facility, vaporizers are calibrated at four different flow rates, 593 K (320° C), 573 K (300° C), 553 K (280° C), and 533 K (260° C). Results of flow calibrations will be discussed later.

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[2]The factor is calculated as follows:

$$\left[6.0228\times10^{23}\ \frac{\text{atom}}{\text{mole}}\right] \times \left[\frac{1}{200.6}\ \frac{\text{mole}}{\text{gram}}\right] \times \left[1.6018\times10^{-19}\ \frac{\text{C}}{\text{atom}}\right]$$

$$\times \left[\frac{1\ \text{amp}}{1\ \frac{\text{C}}{\text{sec}}}\right] \times \left[13.55\ \frac{\text{gram}}{\text{cm}^3}\right] \times \left[\frac{1\ \text{hr}}{3600\ \text{sec}}\right] = 1.810\ \frac{\text{amp-hr}}{\text{cm}^3}$$

50-Hour acceptance test (29). - After completion of flow calibration, a 50-hour acceptance test is performed. The cathode and neutralizer vaporizers are run at 414 kPa (60 psi) and 623 K (350° C). The main vaporizers are run at 414 kPa (60 psi), and 563 K (290° C). The flow is calibrated at the beginning, middle, and end of the 50-hour test. A flow change of more than 5 percent is reason for rejection.

Mercury high temperature intrusion test (30). - A hot intrusion test at 673 K (400° C) is attempted. This test has proven to be very difficult to perform. In the case of the main vaporizer it is difficult to distinguish between the high flow rate and the place where intrusion takes place by visually monitoring the calibration tube.

Mantenieks (ref. 6) has shown that intrusion pressure drops about 25 percent between room temperature and 573 K (300° C). This is caused in part by the change in mercury properties such as viscosity and surface tension. Since the results of this test are imprecise it could be eliminated as a screening test. However, the lower intrusion pressure with increased temperature should be considered when selecting the operating pressure of the propellant system.

TEST RESULTS AND DISCUSSION

Vaporizers were fabricated for 30-cm thrusters by Hughes Research Labs to the previously described designs, material requirements, and fabrication procedures. Four neutralizer vaporizers, seven cathode vaporizers, and five main vaporizers were fabricated. Screening and acceptance test results for these vaporizers are shown in tables IV, V, and VI and the mercury flow data is plotted in figures 20, 21, and 22.

In these plots the slope of the mass flow rate, when plotted as a function of the temperature on log-linear paper, should be a constant. The theoretical slope can be shown by starting with the Clausius-Clapeyron equation.

$$\frac{d \ln P}{dT} = \frac{\Delta H_V}{RT^2} \tag{1}$$

Integrating between P_1 and P_2 and T_1 and T_2 gives:

$$\ln \frac{P_1}{P_2} = - \frac{\Delta H_V}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right)$$

where

P	vapor pressure
T	temperature
ΔH_V	heat of vaporization
R	gas constant

In a constant temperature, constant volume process, partial pressure is proportional to mass flow. Therefore, P_1/P_2 can be replaced by $\dot{M}_2/\dot{M}_1$.

$$\ln \frac{\dot{M}_2}{\dot{M}_1} = - \frac{\Delta H_V}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right)$$

where $\dot{M}$ is mass flow.

In order to get this in the form of the plot the logarithm conversion can be made:

$$\frac{\log \dot{M}_2 - \log \dot{M}_1}{\left(\frac{1}{T_2} - \frac{1}{T_1} \right)} = - \frac{\Delta H_V}{(2.303)R}$$

using

H_V 59727.6 J/mole
R 8.325 J/deg - mole

The theoretical slope then is -3115 K.

Since for $T_2 \equiv T_1$

$$\left(\frac{1}{T_2} - \frac{1}{T_1} \right) = \frac{(T_2 - T_1)}{(T_2 \times T_1)} \approx (T_2 - T_1)$$

It is more practical to plot log $\dot{M}$ against T. Although this is not exactly linear when plotted on log-linear paper it is approximately true over the range of interest and is more easily interpreted.

Neutralizer Vaporizer Data

The neutralizer vaporizer acceptance and screening test data are shown in table IV. The calibration flow data were taken at four temperatures. The flow rate data had very low dispersion. The intrusion pressures of all four vaporizers were greater than 862 kPa (125 psi). The flow rate changes during the 50-hour acceptance test were insignificant, and the flow rates at the 623 K (350° C) and 414 kPa (60 psia) were consistent with the flow rates at the lower temperatures and pressure. The mercury flow coefficient was calculated from the calibration data at 573 K (300° C). The neutralizer calibration flow data is plotted in figure 20. The normal range of mass flow rate is shown for a typical thruster operation. Based on this range the mean required neutralizer vaporizer calibration curve for 573 K (300° C) operation is drawn on figure 20. It intercepts the 573 K (300° C) line at a mass flow rate of 0.035 amp. The plot shows that the flow data is very consistent, and that the neutralizer vaporizer is correctly sized for this application.

Cathode Vaporizer Data

The cathode vaporizer data is shown in table V. The calibration flow data were taken at the same temperatures as the neutralizer data and were very consistent. The cold intrusion data of CV 904 is suspect because hot intrusion data showed a higher value. The values for these two tests are normally reversed. The 50-hour acceptance test data was consistent with the flow data except for CV 902. Although the calibration data was excellent the flow changed drastically during the 50-hour test for CV 902. There probably is a crack in this vaporizer, and the addition of 414 kPa (60 psi) for the test caused a flow change and subsequent higher flow during operation. Unfortunately there was no reported nitrogen flow data on CV 902 to compare it with the other vaporizers. This vaporizer was not used on a thruster. The nitrogen flow data for the other vaporizers was consistent. The mercury flow coefficient was calculated from the mercury flow calibration data at 573 K (300° C). The cathode calibration flow data is plotted in figure 21. The range of mass flow rate is shown for typical thruster operation. Based on this range, the mean required cathode vaporizer calibration curve for 573 K (300° C) operation is drawn on figure 21. This line intercepts the 573 K (300° C) line at a mass flow rate of 0.1 amp. It is obvious that the cathode vaporizer is not correctly sized for the 30-cm thruster.

The design of the cathode plug in a holder is identical to the neutralizer. Because the plug size is identical, the flow calibration data are identical to the neutralizer data. The data from cathode and neutralizer vaporizers had very low dispersion, with the a mean mass flow rate of 0.035 amp at 573 K (300° C).

Because the cathode flow requirement is different than the neutralizer, the cathode should be resized by first finding the flow per area of the plugs as they are presently sized, and then finding the new plug area for the desired flow. The present neutralizer/cathode plug is 0.447 cm (0.176 in.) diameter. The new cathode plug diameter using the same porous tungsten should be 0.762 cm (0.300 in.) diameter. This is a very simple design change to incorporate.

Main Vaporizer Data

The main vaporizer data is shown in table VI. The calibration flow data was taken at the same four temperatures as the neutralizer and cathode vaporizer data. The flow data for MV 901, MV 903, and MV 904 was consistent. The mercury flow data for MV 902 was extremely high; however, the nitrogen flow data for this vaporizer was in the family with the other main vaporizers. That is indicative of a possible calibration error in mercury flow tests. The vaporizer was recalibrated while running on a thruster, and did fall in the family of flow calibrations with vaporizers MV 901, MV 903 and MV 904. The nitrogen flow test proved to be valuable. Figure 22 shows the flow data of MV 903 running on a thruster is a continuation of MV 903 flow calibration at a higher temperature.

It is obvious that the calibration data for MV 909 is incorrect. The slope of the curve of mass flow against temperature does not follow the evaporation rate for mercury as previously discussed. A review of the raw data taken during the calibration revealed that the compressibility test for air pockets in the system did not meet the compressibility requirement. Trapped

air that expands due to temperature changes during flow calibration is an explanation of the erroneous data. The erroneous data on MV 902 and MV 909 is presented in the data set for illustration. If the data had been plotted and evaluated during the test instead of afterwards, such anomalies would have been noticed and investigated before the test completion.

The main vaporizer calibration flow data is plotted in figure 22. A range of mass flow rate is shown for a typical thruster operation, which is the expected throttling range of the thruster. Based on this range the mean required main vaporizer curve for 573 K (300° C) operation is also drawn on figure 22.

The intrusion pressure for the 73 percent dense main vaporizer porous tungsten is lower than the 80 percent dense porous tungsten used for the neutralizer and cathode vaporizers. The data is well above the expected propellant storage pressure of 345 kPa (50 psi) that would be considered for system operation. The cold intrusion of MV 903 appears low and could be in error because as previously discussed, the hot intrusion value should be lower than the cold intrusion pressure. The only 50-hour acceptance data that falls where expected with respect to flow calibration data is on MV 901. It is consistent and did not change with time.

The flow rate for the main vaporizer as originally sized is also too low to meet the throttling flow requirements of the thruster. The flow at 573 K (300° C) for a median set of data is 0.60 amp (see fig. 22). The main vaporizer plug should be larger to provide adequate flow at the 573 K (300° C) design point. The present diameter of the plug is 1.56 cm (0.616 in.). A flow of 1.25 amps is required at 573 K (300° C) to throttle from 0.75 to 2 amps (see fig. 22). This would require a vaporizer plug of 2.17 cm (0.854 in.) diameter. This is larger in diameter than any vaporizer that has been previously fabricated; however, it is thought to be feasible. An alternative to making the plug larger is to operate at higher temperature to achieve required flow. This is what has been successfully done in practice.

Flow Coefficients

The nitrogen and mercury flow coefficients were calculated and are shown in tables IV, V, and VI. The ratio of nitrogen to mercury flow coefficients should be 0.52 as shown in appendix B. If an average is taken of the measured values of nitrogen and mercury flow coefficients and the ratio C_{N_2}/C_{Hg} computed the result is:

	$C_{N_2} \times 10^{-5}$	$C_{Hg} \times 10^{-5}$	C_{N_2}/C_{Hg}
NIV	3.62	6.26	0.58
CIV	3.02	5.44	.56
MIV	4.86	8.5	.57
		Avg.	0.57

The average C_{N_2}/C_{Hg} ratio 0.57 found by testing is slightly higher than the 0.52 calculated value. This is due to the fact that the test pressure

levels used to determine C_{N_2} and C_{Hg} are slightly out of the region of free molecular flow. The result is a slightly higher C_{N_2} value than C_{Hg}.

In general the C_{N_2}/C_{Hg} ratios do agree very closely. Also the individual C values for each family of plugs are in good agreement. This information can be used with a high degree of confidence to give a preliminary evaluation of a porous tungsten plug performance.

CONCLUSIONS

The 30-cm vaporizer program has been successful. Most of the anomalies with early vaporizers that resulted in performance dispersion were investigated, and identified. Required features that affect mercury flow, intrusion, assembly and testing, and thermal-mechanical design were incorporated in the design. Porous tungsten was purchased to the manufacturing requirements. Vaporizers were successfully fabricated, tested, and operated on thrusters. This produced items with little dispersion in mercury flow from vaporizer to vaporizer.

The present vaporizer design point requires vaporizer operation at 573 K (300° C) for thruster operation. That being the case, the neutralizer has been sized correctly but cathode and main vaporizers must be larger to meet the thruster flow requirements at 573 K (300° C).

APPENDIX A

VAPORIZER CONTAMINATION

Some past vaporizer flow anomalies have been attributed to contamination. The incidents were not many but they warrant discussion.

Types of Vaporizer Anomalies Due to Contamination

Mercury contamination has been considered a cause of failure by two different types of vaporizer malfunctions. One type of contaminant (such as copper, silver, or gold amalgamated in mercury) can cause mercury to wet the porous tungsten causing liquid mercury penetration (intrusion) of the pores at low pressures. Kerslake (ref. 5) discusses this type of contamination. Only one case of this type of vaporizer malfunction has been documented (ref. 7). One percent silver impurity caused the liquid mercury to wet the tungsten and intrude the vaporizer after 24 hours of operation. No recent vaporizer intrusion has been attributed to mercury wetting. The effect of a wetting contaminant is higher flow rate and lower intrusion pressure.

The second type of mercury contaminant causes blockage. The blockage type of contaminant results in lower flow rates and higher intrusion pressures. To insure that the mercury does not contain high levels of contaminants it should meet a specification such as the Lewis Research Center Specification 101 "Standards for High Purity Mercury" contained in appendix E.

Vaporizer Blockage

There have been at least three different vaporizer blockage incidents identified.

High iron content. - The first one was identified as a flow blockage after many hours of operation.

Samples were taken from a mercury supply system that caused blockage of a vaporizer. A spectrographic analysis of the samples revealed iron contamination. In this case distilled water had been used to cover the mercury in the reservoir to prevent mercury oxides from forming during thruster life tests. The water contained small amounts of iron. As the water evaporated the iron accumulated on top of the mercury. When the mercury level was allowed to drop in the supply tube, iron was fed into the vaporizers causing a blockage.

High silver content. - The second known blockage occurred during testing of 8-cm thruster vaporizers. The failure was identified as blockage or a change in flow rate due to a white substance in the vaporizer plug. The white substance was identified as silver. An analysis of the mercury showed an extremely high silver content of 33 ppm. Instead of causing wetting this large amount of silver was being filtered out in the porous tungsten. When the contaminated mercury was replaced the problem disappeared.

Porous tungsten oxidation. - Porous tungsten oxidation is a vaporizer blockage malfunction that is not attributed to mercury contamination. It is, however, considered as a contamination of the porous tungsten. The third blockage occurred during testing of 30-cm vaporizers. After approximately 40 hours of "baking" a main vaporizer to remove mercury after a series of intrusion tests, the calibration flow rate reduced considerably. Subsequent,

additional intrusion testing exhibited higher intrusion pressure. It also required higher nitrogen pressure to achieve a bubble pattern during bubble testing.

Figure 23 is a 30x picture of the liquid side of the plug. What appears to be particles on the liquid surface (shown by the arrow) were found to be tungsten oxide crystals. Figure 24 is a magnified picture of the same area.

SEM (Scanning Electron Microscope) photographs of the plug vapor and liquid surfaces were taken in order to compare the change that took place. The SEM photographs of the vapor side of the plug are shown in figures 25 and 26. The porous tungsten looks as it did when it was received. The SEM photographs of the liquid side of the plug are shown in figures 27 and 28. In comparing the liquid and vapor sides of the plug it is obvious that the sintered powder has changed. The tungsten is not joined on the liquid side as it is on the vapor side of the plug. Attempts were made to focus in on the particles on the surface and to identify any foreign material with an Energy Dispersing Analysis using X-Rays (EDAX) measurement. The EDAX results were interpreted as tungsten and its oxides. No other foreign material was identified. The conclusion is that the particles that can be seen on the liquid side surface are tungsten oxide crystals.

In order to check the above results an X-ray diffraction analysis of the surface was performed. This analysis also indicated that the particles were tungsten oxide (WO_3).

Cause and effect of oxidation. - Flow and intrusion testing has shown that the oxidation of the porous tungsten took place during the procedure that is used to "bake" the mercury from the plugs after intrusion testing. Air from a leak in the system was reaching the liquid side of the plug. The following chronology (see table VII). of the vaporizer testing shows how this conclusion was reached.

Step 1 is the initial flow calibration test, and Step 2 is the initial intrusion test. Step 3 is a one point check on the flow calibration. Even though the plug had been intruded and not baked out, this point still fell on the initial calibration curve.

The vaporizer characteristics started to change after Step 4 which was the first "bake."

There was an increase in the intrusion pressure (Step 5) after the vaporizer had been baked in Step 4. However, there was no change in flow from the original calibration (Step 6). The oxidation was apparently increasing the contact angle of the mercury on the porous tungsten causing higher intrusion pressure, but was still not affecting the pore openings.

The vaporizer was again baked in Step 7. The intrusion pressure was not tested, but the flow was calibrated at various pressures (see Step 8). The flow calibrations at 199.9 kPa (29 psi) and 308.2 kPa (44.7 psi) were the same as the original calibration, but the flows at 411.6 kPa (59.7 psi) and 446.2 kPa (64.7 psi) were higher. This could be attributed to a condition where the plug was partially intruded from the applied pressure resulting in higher flow.

The vaporizer was again drained and baked (see Step 9). This step caused a dramatic change in intrusion pressure (see Step 10) and flow calibration (see Step 11). This final data shows that the plug was being oxidized and that the oxidation blocked the flow paths and resulted in increased intrusion pressure and decreased flow.

The data also shows that two "bakes" for 6 hours each did not affect the flow, but did increase the ability of the material to withstand intrusion

pressure. Some oxidation is not only acceptable but helpful. Too much oxidation, however, is unacceptable.

Bubble testing. - Bubble testing of the vaporizer after the long-time-baking showed that some flow paths through the porous plug were closed. This plug had been previously bubble tested during processing. The original gas flow pressures were 41.4 kPa (6 psi) for the first bubbles, and 82.7 kPa (12 psi) for a full bubble flow pattern. After the long bake-out the vaporizer was again bubble tested. The first bubbles appeared at 62.0 kPa (9 psi) and the full bubble flow pattern was at 103.4 kPa (15 psi).

APPENDIX B

NITROGEN FLOW TESTING

A useful s creening test for porous tungsten vaporizer subassemblies is the determination of the nitrogen gas flow coefficient. It can be used on a relatively inexpensive subassembly to verify that the ability to flow vapor through the plug is in an acceptable range. It involves a simple test at room temperature and avoids the contamination of mercury flow tests requiring operation at high temperature.

Figure 31 is a schematic of the test installation used to measure the flow coefficient. The governing equation is as follows. It will be derived and justified later in this appendix.

$$C = \frac{2.74 \times 10^{-4} \, \Delta p V \sqrt{M}}{tPA \sqrt{T}} \qquad \text{(B-1)}$$

where

C flow coefficient
ΔP change in pressure on downstream side (dyne/cm^2)
V volume on downstream side (cm^2)
M molecular weight
t time (sec)
P pressure on upstream side (dyne/cm^2)
A area of plug (cm^2)
T temperature of gas (K)

Some of the required terms are constants determined by test geometry and the gas used. For one facility, they are as follows:

V volume of vacuum reservoir = 10574 cc
A plug area = 0.1570 cm^2 for cathodes and neutralizer
= 1.9227 cm^2 for main
M molecular weight = 28 for N_2

The remaining terms are experimentally determined when operating the test facility through the following procedural steps:

1 valve A closed
2 valves B and C opened
3 system evacuated to <0.5 torr
4 valves B and C closed
5 valve A opened to allow pressure to increase in nitrogen reservoir to between 3.44×10^4 and 6.89×10^5 dyne/cm^2 (0.5 and 10 psia).

This results in a large pressure gradient across the plug. Pressure readings are taken on gage B for several time increments. The pressure of the nitrogen reservoir is held constant by manual control of valve A during this time interval. Thus the final unknowns (Δp, T, t, and P) in equation (B-1) are obtained and C can be calculated for each value of P (3.44×10^4 to 6.89×10^5 dyne/cm^2) (0.5 to 10 psia) selected. A typical curve of P

against C shown in figure 32. A summary of the nitrogen and mercury flow coefficients is shown in tables IV, V, and VI.

It is possible to correlate the nitrogen flow coefficients obtained above with coefficients that would be determined in lot tests with mercury.

The mercury transmission coefficient can be calculated using the equation from Kerslake's (ref. 5) report.

$$j_o = C\left[\frac{1}{e}\sqrt{\frac{N_o}{2\pi k}}\right]\frac{P_V}{\sqrt{TM}} \qquad \text{(B-2)}$$

where

j_o gas flow leaving plug, equivalent A/cm^2
C transmission coefficient
P_V upstream vapor pressure, dynes/cm^2
T upstream vapor temperature, K
e 6.24×10^{18} atoms/sec per equivalent ampere
N_o Avogadro's number, 6.022×10^{23} atoms/mole
k Boltzmann constant, 1.38×10^{-16} dyne-cm/K
M gas molecular weight, 200.6 for Hg

Substituting the constants into equation (B-2) results in:

$$j_o = 4.22\ C\ \frac{P_V}{\sqrt{TM}} \qquad \text{(B-3)}$$

Since C was calculated at 573 K (300°C) and P_V for Hg is 3.3×10^5 dyne/cm^2, equation (B-3) reduces to:

$$j_o = 4.1\times10^3\ C \qquad \text{(B-4)}$$

Solving for C:

$$C = 2.4\times10^{-4}\ j_o \qquad \text{(B-5)}$$

Equation (B-5) and the measured mercury flow at 573 K (300°C) were used to calculate the C values for mercury flow shown in tables IV, V, and VI.

The ratio of the nitrogen to mercury flow coefficient was calculated using equation (B-1). Assuming that Δp, V, t, P and A are the same, the ratio is:

$$\frac{C_{N_2}}{C_{Hg}} = \frac{\sqrt{\frac{M_{N_2}}{T_{N_2}}}}{\sqrt{\frac{M_{Hg}}{T_{Hg}}}} \qquad \text{(B-6)}$$

where

C_{N_2} nitrogen flow coefficient
C_{Hg} mercury flow coefficent nitrogen
M_{N_2} nitrogen molecular weight, 28
T_{N_2} nitrogen gas temperature, 294 K
M_{Hg} mercury molecular weight, 200.6
T_{Hg} mercury vapor temperature, 573 K

For the above given conditions, equation (B-6) reduces to:

$$\frac{C_{N_2}}{C_{Hg}} = 0.52 \tag{B-7}$$

Following is the derivation of the nitrogen flow coefficient expression of equation (B-1). The coefficient is defined as follows:

$$C = \frac{N}{vt} \frac{\text{number of gas molecules leaving downstream surface}}{\text{number of gas molecules arriving at upstream surface}} \tag{B-8}$$

Since N is the total number of molecules, let N_X be the number of molecules at time t. Now

$$N = N_1 - N_0 \tag{B-9}$$

where

N_0 number of molecules at t = 0.
N_1 number of molecules at t = X.

From Charles' law we know that

$$N = \frac{P_X V}{kT} \tag{B-10}$$

where

P_X pressure (dyne/cm^2)
V volume (cm^3)
k Boltzmann constant (1.38×10^{-16} dyne -cm/K)
T temperature (K)

Substituting equation (B-10) into (B-9):

$$N = \frac{P_1 V}{kT} - \frac{P_0 V}{kT} = \Delta p \frac{V}{kT} \tag{B-11}$$

where $\Delta p = P_1 - P_0$.

Substituting k into equation (B-11):

$$N = \frac{7.244 \times 10^{15} \Delta pV}{T} \qquad \text{(B-12)}$$

From the kinetic theory of gases the total number of molecules striking the upstream side of the plug is:

$$\upsilon t = 1/4\ n\bar{v}\ At \qquad \text{(B-13)}$$

where

υ molecules/sec
t time (sec)
n molecules/cm^3
$\bar{v}$ average velocity (cm/sec)
A plug area (cm^2)

From the Maxwell-Boltzmann distribution equation:

$$\bar{v} = \sqrt{\frac{8R_0T}{\pi M}} \qquad \text{(B-14)}$$

where

R_0 gas constant (8.3×10^7 dyne-cm/K/molecule)
T temperature (K)
M molecular weight (28 for N_2)
π constant (3.1416)

Substituting into equation (B-14) one gets:

$$\bar{v} = 14551 \sqrt{\frac{T}{M}} \qquad \text{(B-15)}$$

from the gas laws:

$$n = \frac{P}{kT} \qquad \text{(B-16)}$$

where

P pressure (dynes/cm^2)
k Boltzmann constant (1.38×10^{-16} dyne-cm/K)
T temperature (K)

Substituting equations (B-15) and (B-16) into (B-13) gives:

$$\upsilon t = \frac{1}{4}\left(\frac{P}{kT}\right)\left(14551\sqrt{\frac{T}{M}}\right)At = 2.64\times10^{19}\,\frac{PAt}{\sqrt{TM}} \qquad \text{(B-17)}$$

Substituting equations (B-12) and (B-17) into equation (B-8):

$$C = \frac{N}{\upsilon t} = \frac{\dfrac{7.244\times10^{15}\,\Delta pV}{T}}{\dfrac{2.64\times10^{19}\,PAt}{\sqrt{TM}}}$$

Which reduces to equation (B-1):

$$C = 2.74\times10^{-4}\,\frac{\Delta pV}{PAt}\sqrt{\frac{M}{T}}$$

APPENDIX C

BUBBLE TESTING

A simple test that can be made on the porous tungsten plug - tantalum housing subassemblies is a bubble test. This test can show gross flow deficiencies such as blockage, lack of uniformity caused by contamination, cracks, weld cracks, or unetched surfaces.

After welding and visual inspection, a bubble test should be done. A bubble test setup is shown in figure 33. The test setup allows each porous tungsten subassembly to be completely immersed under alcohol or to have a thin film of alcohol on the surface. The nitrogen pressure can range from 0 to 172.4 kPa (0 to 25 psi).

To run the bubble test, the sample is mounted in the holder. A piece of vacuum hose or other hose of proper diameter makes an adequate holder. The nitrogen pressure is set at 34.4 kPa (5 psi). The nitrogen pressure is applied before the alcohol to prevent intrusion of the plug by the alcohol.

After the nitrogen pressure is applied, a thin film of alcohol is applied to the sample's surface using a squeeze bottle. The bubble pattern is then noted. The nitrogen pressure is increased in 1 psi increments (alcohol applied at each increment) and the bubble pattern observed and noted either by photograph or sketch. Each sample is tested, increasing the pressure until the bubble flow becomes so vigorous that one can no longer see a well defined pattern, and a foam appears. This maximum pressure varies from sample to sample.

To inspect the weld area on the sides of the sample, the sample is immersed by pushing it down into the holder so the sample's surface is approximately 1.5 mm (0.06 in.) below the top of the holder. The nitrogen pressure and then alcohol are applied. The weld area is inspected for leaks at various nitrogen pressures.

Figures 34, 35, 36, and 37 are a sequence of photographs of a main vaporizer being bubble tested with increasing nitrogen pressure. This vaporizer was selected because it shows both acceptable and unacceptable flow areas. The center of the vaporizer is contaminated.

Figure	N_2 kPa (psi)	Observation
34	55 (8)	First bubbles.
35	62 (9)	A bubble pattern is starting in the non-contaminated area.
36	69 (10)	An excellent acceptable bubble pattern is shown around the outside. If the entire plug had this pattern it would be acceptable. The center is blocked by the contaminant.

37	76 (11)	The bubble pattern around the outside is almost a foam. Not much can be learned by increasing the pressure. The center has some bubbles which shows the contaminant is not completely blocking the pores. This sample would be rejected, but could possibly be cleaned by vacuum firing.

Figure 38 is also a main vaporizer with a small amount of contamination. This buble pattern shows some areas have small pores and do not allow flow. There are also two lines of bubbles which could be an indiction of a small crack, or a line of different density as shown in figure 12.

APPENDIX D

POROSIMETER TESTING THEORY

The pore size distribution and pore volume in the porous tungsten vaporizer plugs can be measured using a porosimeter. That makes porosimeter testing a very useful process for screening porous tungsten. A nonwetting liquid like mercury can be forced into the pores of a porous material. The pressure required to fill the pore completely is inversely proportional to the size of the pore. The general equation for this relationship is:

$$D = \frac{-4\sigma \cos \theta}{p} \qquad \text{(D-1)}$$

where

D diameter of pore, microns
p mercury pressure, kPa
θ wetting or contact angle of mercury with solid, deg
σ surface tension of mercury, N/m

The value of the contact angle, θ, varies with different materials. An average value of 130° was used in the porous tungsten measurements. The value used for the mercury surface tension, σ, was 0.473 newton/meter at 25° C. Substituting the above values and suitable conversion factors into equation (D-1) results in:

$$D = \frac{1.216 \times 10^6}{p} = \frac{175}{P_{psia}} \qquad \text{(D-2)}$$

where

D pore diameter in microns
p absolute mercury pressure in kPa
P_{psia} absolute mercury pressure in psia

Thus, if one places a porous sample in a volume of mercury and measures the change in the mercury volume while increasing the pressure on the mercury and sample, a plot of pressure against volume change can be made. Since the relationship of pressure against pore size is known (eq. D-2), the plot of pressure against volume change can be converted into pore size against volume of pores.

Porosimeter Operation

The porosimeter measurements were made using an Aminco JS-7121-B, 103 mPa (15000 psi) porosimeter. This porosimeter is capable of measuring pore diameters from 100 microns to 0.012 micron (assuming a 130° contact angle) and pore volumes from 0.0001 cc to 0.21 cc.

To make a porosimeter measurement a porous tungsten vaporizer plug is placed in a sample holder (penetrometer). A vacuum (<50 microns) is pulled on the sample and penetrometer. The penetrometer is then filled with mercury. The penetrometer containing the sample surrounded by mercury is now slowly pressurized. Readings of mercury volume changes are taken at various pressure levels. This information (pressure against porous volume) can be plotted on a graph as shown in figure 39.

This data allows determination of the following plug characteristics.

Pore size distribution is obtained directly by labeling the pressure axis with pore size using the relation of D-2.

Intrusion pressure of the completed vaporizer is indicated by the vertical section of the curve.

APPENDIX E

MERCURY PURITY

NASA LeRC - No. 101
May 1, 1965

LEWIS RESEARCH CENTER

NATIONAL AERONATUCIS AND SPACE ADMINISTRATION

CLEVELAND, OHIO

MERCURY STANDARDS FOR HIGH-PURITY

NASA SPECIFICATION FOR

1.0 SCOPE

1.1 This specification defines the standards for high-purity mercury.

2.0 APPLICABLE DOCUMENTS

2.1 The following specifications and standards, of the issue in effect on the date of invitation for bids, form a part of this specification to the extent specified herein.

STANDARDS

Federal Standard No. 102 - Preservation, Packaging and Packing Levels

Federal Standard No. 123 - Marking for Domestic Shipment (Civilian Agencies)

(Copies of documents required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the Contracting Officer.)

3.0 REQUIREMENTS

3.1 High-purity mercury shall be defined as metallic mercury (quicksilver) that meets the standards as determined by the requirements embodied herein.

3.2 Appearance. High-purity mercury shall give no evidence of oxidation and have a bright mirror-like surface free from film or scum.

3.3 Nonvolatile Material. Nonvolatile material shall not exceed 0.025 parts per million of residue by weight. Individual particle size of the nonvolatile material shall not exceed 2.5 microns (0.0001 in.).

3.4 Volatile Material. The total quantity of volatile matter shall be no greater than 0.1 parts per million.

3.5 Shrinkage. Weight loss, due to processing and handling, shall not exceed the limits as outlined below.

Shrinkage Limits

Net weight (lb)	Percent of loss	Net weight (lb)	Percent of loss
0 - 25	2.00	201 - 400	0.50
26 - 50	1.50	401 - 700	.30
51 - 100	1.00	701 - 1000	.25
101 - 200	.75	1001 +	.22

Unless otherwise specified in the procurement document, shrinkage will not be compensated for with additional mercury.

4.0 SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 Inspection Responsibility. The supplier is responsible for the performance of inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities or services acceptable to the government. Inspection records of the examination and tests shall be kept complete and available to the government. The government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to insure that material and services conform to requirements of this specification.

4.2 Certification. The processor shall submit with each shipment written certification that the requirements of this specification have been met.

4.3 Test Requirements

4.3.1 Visual Test. Sample(s) of high-purity mercury shall be drawn, prior to bottling, and examined under a bright light. The sample size shall be not less than one-half pound for each 500 pounds of mercury. Sound practices for random sampling shall prevail in this test. Any evidence of oxidation, film, scum or discoloration shall be cause for rejecting the entire shipment.

4.3.2 Contamination Test. Samples of high-purity mercury shall be drawn prior to bottling, and placed in a thoroughly cleaned, dry 250 milliliter beaker. The sample size shall be one-half pound for each 500 pounds of mercury. Sound practices for random sampling shall prevail in this test. Rotate the beaker so as to impart a circular motion to the mercury. Pour off the mercury without shaking the beaker. If mercury adheres to the beaker walls the entire shipment shall be rejected.

4.3.3 Nonvolatile Residue Test. A sample (under 1200 lb) or a composite of two samples (over 1200 lb or excess of), drawn prior to bottling, of 2000 grams per each 1200 pounds shall be evaporated completely in a vacuum of 100 microns at 500 degrees F. When the sample is reduced to a 10 gm button, it shall be placed in a crucible and completely evaporated under vacuum and heat as above. The amount of residue shall not exceed the requirements of Section 3.3.

5.0 PREPARATION FOR DELIVERY

5.1 Packaging and packing levels shall meet the requirements of Fed. Std. No. 102, Level C.

5.2 Packaging. High-purity mercury shall be placed in containers which will not cause or promote oxidation nor contamination to the mercury.

5.3 Markings. In addition to any special markings required by contract or order, unit packages and exterior shipping containers shall be marked in accordance with Fed. Std. No. 123.

5.4.1 The unit containers shall include the following markings:

1. Federal Stock Number
2. Item Identification
3. Contract or Purchase Order No.
4. Contractor's Name and Address
5. Net Weight
6. Lot Number

5.4.2 The shipping container shall be limited to the following markings:

1. Consignee's Address
2. Consignor's Address
3. Federal Stock No. of Item
4. Net Weight
5. Gross Weight
6. Quantity
7. Contract or Purchase Order No.

NOTE: The item identification shall not appear on the shipping container.

6.0 NOTES

6.1 Intended Use. Standards for high-purity mercury are specified to insure a quality product for use in research and development relative to the space effort.

6.2 Ordering Data. Procurement documents should specify the following:

a. Title, No. and date of this specification.
b. Complete item description.
c. Federal stock No.
d. Quantity in pounds (net weight).

Notice: When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the government may have formulated, furnished, or in any way supplied the said drawings, specifications or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian:

NASA Lewis Research Center

Preparing Activity:

Lewis Research Center

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TABLE I. - POROUS TUNGSTEN MANUFACTURING SPECIFICATION

1. POWDER: 4 to 5 micron average powder diameter. Particle sizes to be normally distributed with less than 2 percent by weight of sizes under 1 micron and classified to eliminate particles and agglomerates above 10 microns.

2. POWDER SHAPE: Angular or spherical.

3. DENSITY: 73 ±2 percent for main material. Measure and provide density of each part. 80 ±2 percent for cathode and neutralizer material.

4. SINTERING: Provide time and temperature in hydrogen atmosphere or vacuum.

5. HANDLING: Handle with clean gloves after sintering.

6. MACHINING: Machine to final size after filling with polymethyl methacrylate or other suitable medium. The manufacturer shall identify the filler medium used. The name of the medium shall be noted on tags packed with the porous plug blank.

7. POLISH: Polish etch with fresh Murakami solution to completely remove smeared-over pores. Microscopically inspect at 500x or better. Reetch if necessary.

8. FILLER REMOVAL: Remove filler medium by firing 6 to 8 hours at 618 K (345° C) followed by firing 10 to 15 minutes in hydrogen atmosphere at 1673 K (1400° C). Cool to room temperature in hydrogen.

9. INSPECTION: Visually inspect at 10x or more mag. for complete removal of medium and all contaminants. Verify no imperfections, e.g. cracks, chips or density differences.

10. IDENTIFICATION: Each part must be identified with a serial number.

11. PACKAGING: Individually packaged in a clean polyethylene bag with P/N, S/N and Rev/N.

TABLE II. - TUNGSTEN POWDER LOT CERTIFICATION

Customer	Customer Order Number	Lot Number
	Quantity Shipped	Date Shipped

AVERAGE PARTICLE DIAMETER BY FISHER SUB-SIEVE SIZER (Per ASTM B-330)		
Powder Condition (see ASTM Bi430)	As supplied	Lab milled
A.P.D. (microns)	4.45	4.05
Porosity Value	.630	.450

PARTICLE SIZE DISTRIBUTION BY TURBIDIMETRIC SEDIMENTATION (Per ASEM B-430)	
Powder Condition:	Lab Milled
Micron Range	Weight Percent
0 - 1	.8
1 - 2	7.8
2 - 3	15.3
3 - 4	19.7
4 - 5	20.4
5 - 6	16.5
6 - 7	10.5
7 - 8	6.3
8 - 9	2.0
9 - 10	.8
10 - 11	
11 - 12	
12 - 13	
13 - 14	
14 - 15	
15 - 20	
20 - 25	

CHEMICAL ANALYSIS			
Element	ppm	Element	ppm
Al	-2	Mg	4
Ca	5	Sn	-2
Si	-5	O2	290
Mo	216		
Fe	18		
Cr	-3	W (By differences and on a gas-free basis) 99.9 percent minimum	
Ni	32		
Cu	-3		
Mn	-2		

TABLE III. - 30 CM VAPORIZER PROCESSING AND TESTING SEQUENCE

1. Clean vaporizer parts (except porous W plug) in freon.
2. Bake out all tantalum parts at 1273 K (1000° C) for 15 minutes at 10^{-5} torr ion pumped.
3. Visual inspection of all plugs with 10X magnification.
4. Porosimeter test W plug. To be acceptable, results must fall above and to the right of curve shown in figure 13.
5. Bake out Hg in W plug 563 K (290° C), 100 microns, 15 hours.
6. Turn the outside diameter of the porous tungsten plugs.
7. Visual inspection, 10X magnification. (Reject cracked or scratched plugs.)
8. Ultrasonically clean in trichloroethylene or equivalent.
9. Clean in freon TF[1] vapor.
10. Furnace dry at 400 K (127° C) for 1 hour in dry GN_2 atmosphere.
11. EB wash edge of W plug. 99 percent of surface shall be covered after wash.
12. Visual inspection, 10X magnification.
13. Ultrasonically clean in trichloroethylene or equivalent.
14. Clean in freon TF[1] vapor.
15. Vacuum fire plug, 1923 K (1650° C), 10^{-5} torr, ion pumped 1 hour (support plug at edges only with W holder).
16. Visual inspection, 10X magnification.
17. EB weld W plug in Ta housing.
18. Visual inspection of assembly 30X magnification.
19. Bubble test. (Use ultrahigh purity nitrogen - 99.997 percent.)
20. Thermal cycle. 373 K (100° C) to 723 K (450° C), 25 times, 10^{-5} torr.
21. Visual inspection of assembly, 30X magnification.
22. Bubble test.
23. N_2 flow test. Cathode-Neutralizer = C = 3.0 to 4.0 x 10^{-5} at 250 torr pressure; Main C = 4.0 to 5.0 x 10^{-5} at 250 torr.

[1]DuPont registered trade mark for fluorocarbon compounds.

TABLE III. - Concluded.

24. Vacuum fire assy, 1923 K (1650° C), 10^{-5} torr, ion pumped 1 hour.
25. Visual inspection of assembly, 10 magnification.
26. Assemble into vaporizer assembly.
27. Hg intrusion. Ambient.
28. Bake out Hg 623 K (350° C), 10^{-5} microns, 30 minutes minimum.
29. Hg flow calibration at 543 K (270° C), 573 K (300° C), and 593 K (320° C).
30. 50-Hour Acceptance Test - 50 hour flow 413.6 kPa (60 psia) Hg at 623 K (350° C) for the Cathode-Neutralizer 413.6 kPa (60 psia) Hg at 563 K (290° C) for the Main
31. Hg high temperature intrusion test. 673 K (400° C).

TABLE IV. - NEUTRALIZER SCREENING TEST DATA

NV number	901	902	903	904
Calibration temperature	Mercury flow			
	amp	amp	amp	amp
593 K	0.057	0.064	0.057	0.054
573 K	.035	.039	.036	.034
553 K	.022	.024	.021	.019
533 K	.012	.007	-----	-----
Intrusion (kPa)				
Cold	>862	>862	>862	>862
Hot	>862	>862	>862	-----
Flow rate during 50-hour acceptance test (623 K - 414 kPa)	Mercury flow			
	amp	amp	amp	amp
Start	0.111	0.116	0.123	-----
Middle	.111	.115	.128	-----
End	.111	.119	.128	-----
Nitrogen flow coefficient $Cx10^{-5}$	3.6	3.7	3.7	3.5
Mercury flow coefficient at 573 K $Cx10^{-5}$	5.4	6.0	5.6	5.3

TABLE V. - CATHODE SCREENING TEST DATA

CV number	901	902	903	904	907	908	911
Calibration temperature	Mercury flow						
	amp	amp	amp	amp	amp	amp	amp
593 K	0.051	0.051	0.045	0.053	0.053	0.062	0.038
573 K	.041	.041	.032	.034	.035	.038	.022
553 K	.019	.019	.018	.022	.022	.025	.014
533 K	.010	.010	.010	.012	.012	.014	.008
Intrustion (kPa)							
Cold	>862	>862	>862	731	826	647	855
Hot	>862	>862	>862	>862	848	>862	>862
Flow rate during 50-hour acceptance test (623 K - 414 kPa)	Mercury flow						
	amp	amp	amp	amp	amp	amp	amp
Start	0.101	0.113	0.117	0.111	0.121	0.138	0.076
Middle	.103	.176	.125	.116	.122	.141	.077
End	.106	.209	.128	.119	.127	.151	.074
Nitrogen flow coefficient							
$Cx10^{-5}$	3.3	-----	3.2	-----	3.1	3.3	2.2
Mercury flow coefficient							
$Cx10^{-5}$	6.4	6.4	4.9	5.2	5.9	5.9	3.4

TABLE VI. - MAIN SCREENING TEST DATA

MV number	901	902	903	904	909
Calibration temperature	Mercury flow				
	amp	amp	amp	amp	amp
593 K	0.959	3.27	1.08	0.842	1.39
573 K	.595	1.99	.709	.508	1.088
553 K	.363	1.26	.424	.301	.838
533 K	.216	.72	.261	.192	.714
Intrusion (kPa)					
Cold	724	>862	614	779	731
Hot	662	744	841	793	703
Flow rate during 50-hour acceptance test (563 K - 414 kPa)	Mercury flow				
	amp	amp	amp	amp	amp
Start	0.497	2.27	-----	-----	1.83
Middle	.496	2.46	-----	-----	.514
End	.516	2.61	-----	-----	.528
Nitrogen flow coefficient $Cx10^{-5}$	4.6	4.6	4.9	4.2	6.0
Mercury flow coefficient $Cx10^{-5}$	7.5	N.A.	N.A.	6.4	13.8
Mercury flow coefficient running on a thruster $Cx10^{-5}$	-----	8.1	6.7	-----	-----

TABLE VII. - CHRONOLOGY OF VAPORIZER M-1 TESTING

1. Flow 1 (Fig. 29)
   Initial flow calibration at 623 K (350° C), 593 K (320° C), and 533 K (260°C) each at 1 atmosphere pressure.

2. Intrusion 1 (Fig. 30)
   Intrusion tested from 202.7 kPa (29.4 psi) to 475 kPa (68.9 psi) newtons/meter^2 at 298° (25° C).

3. Flow 2 (Fig. 29)
   Calibration at 533 K (260° C) at 1 atmosphere pressure.

4. Drained and baked the system at 638 K (365° C) for 6 hours.

5. Intrusion 2 (Fig. 30)
   Intrusion tested from 199.2 kPa (28.9 psi) to 579.8 kPa (84.1 psi) at 297 K (24° C).

6. Flow 3 (Fig. 29)
   Repeated the initial flow calibration.

7. Baked at 643 K (370° C) for 6 hours, followed by 533 K (260° C) for 24 hours.

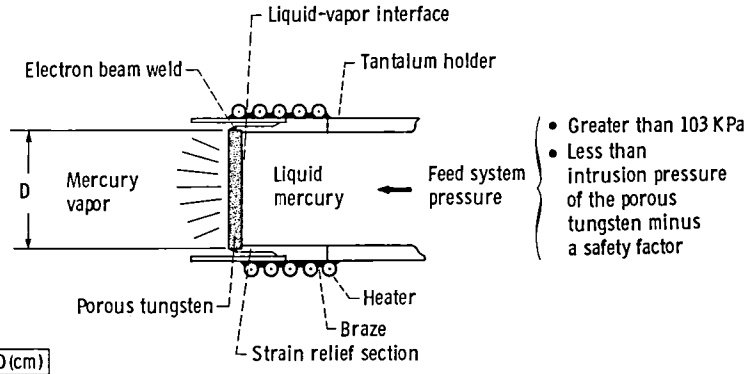
8. Flow 4 (Fig. 29)
   Pressure-flow calibration at 199.9 kPa (29 psi) and 308.2 kPa (44.7 psi) at each of 533 K (260° C), 563 K (290° C), 593 K (320° C), and 623 K (350 °C); then at 411.6 kPa (59.7 psi) at 533 K (260° C); and at 446.1 kPa (64.7 psi) at each of 563 K (290° C), 593 K (320° C), and 623 K (350° C).

9. Drained and baked system at 648 K (375° C) for 33 1/2 hours.

10. Intrusion 3 (Fig. 30)
    Intrusion tested from 199.9 kPa (29 psi) to 939 kPa (136.2 psi) at 298 K (25° C).

11. Flow 5 (Fig. 29)
    Flow calibration at 533 K (260°C), 563 K (290°C), 593 K (320°C), and 623 K (350°C) at one atmosphere.

    1 psi = 6.8947 kPa



Vaporizer	D(cm)
Main	1.565
Cathode Neutralizer	.447

Figure 1. - Typical vaporizer design.

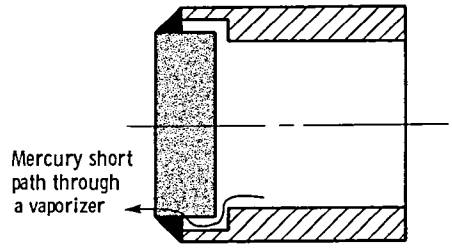


Figure 2. - Possible short path through a vaporizer.

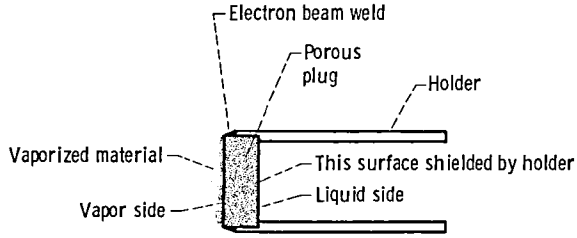


Figure 3. - Preventing vaporized material on the liquid to vapor interface.

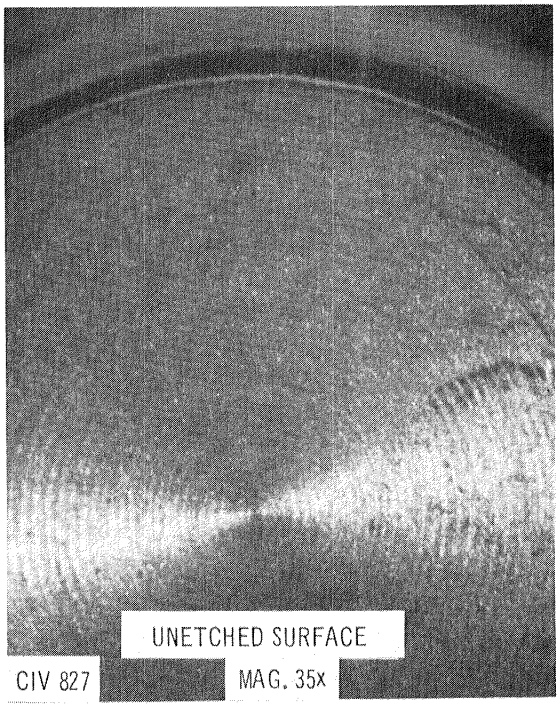


Figure 4. - Porous tungsten plug - unetched surface.

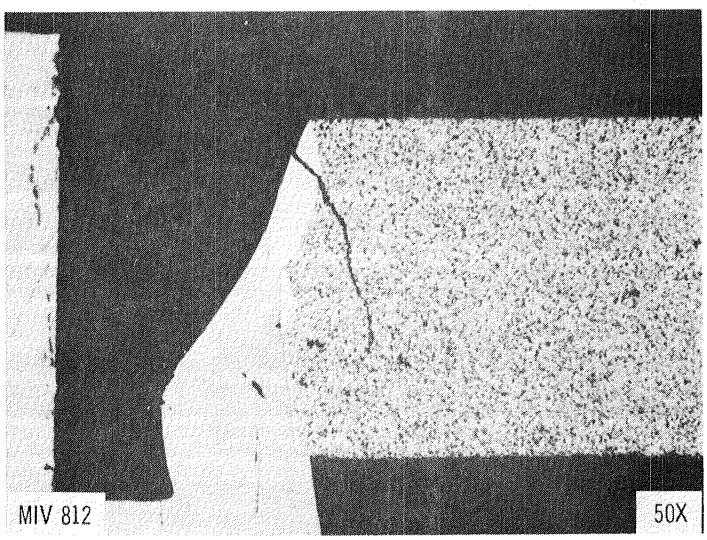


Figure 5. - Porous tungsten plug - cracked.

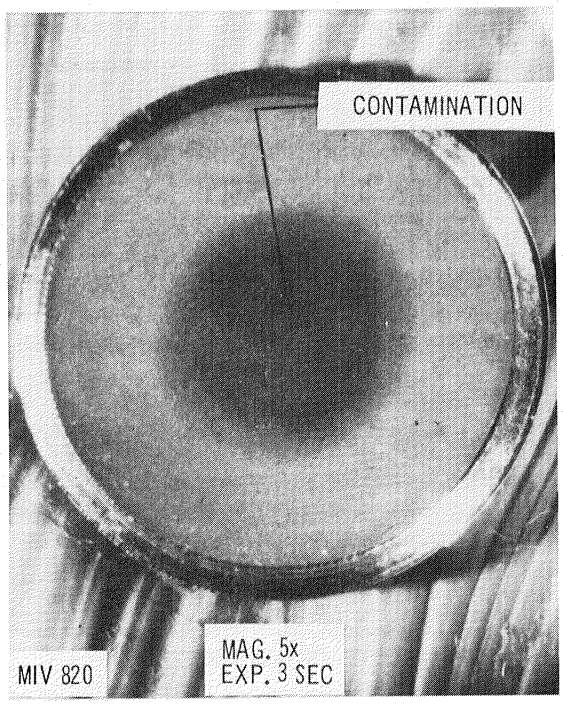


Figure 6. - Porous tungsten plug - contaminated.

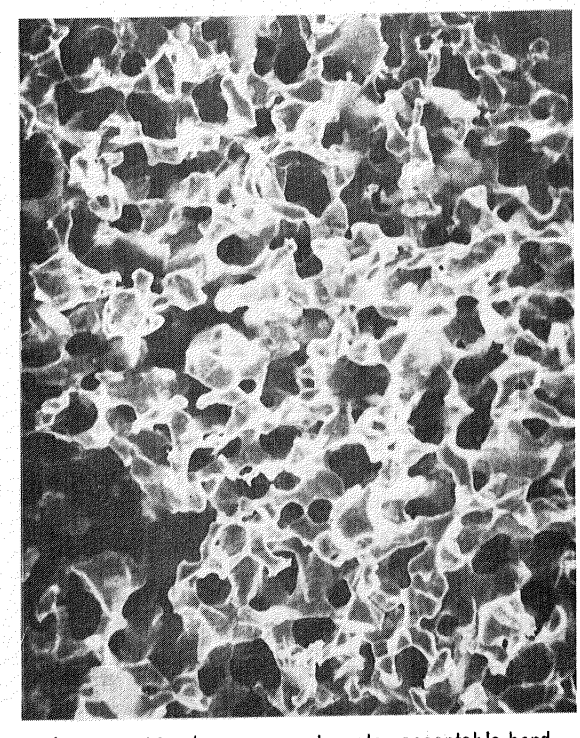


Figure 7. - 73% dense porous tungsten acceptable bond (2000X).

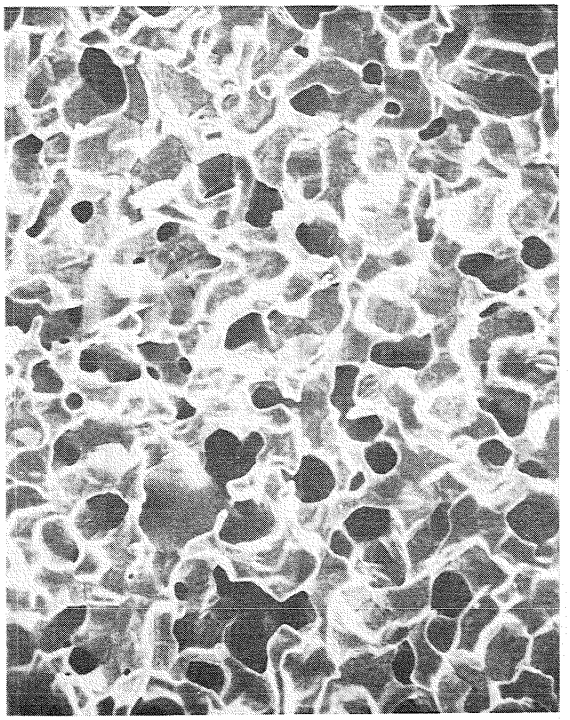


Figure 8. - 80% dense porous tungsten acceptable bond (2000X).

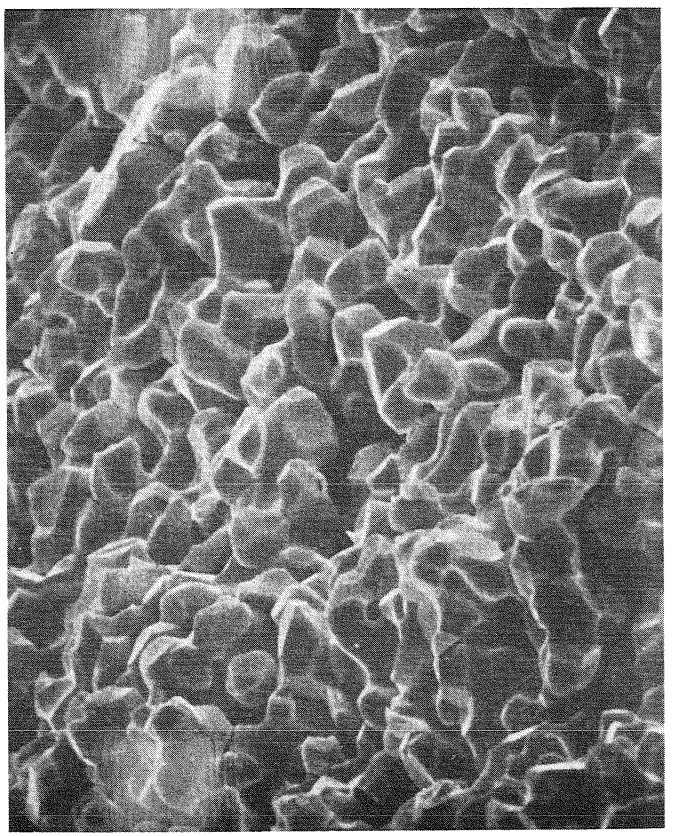


Figure 9. - 80% dense porous tungsten unacceptable bond (2000X).

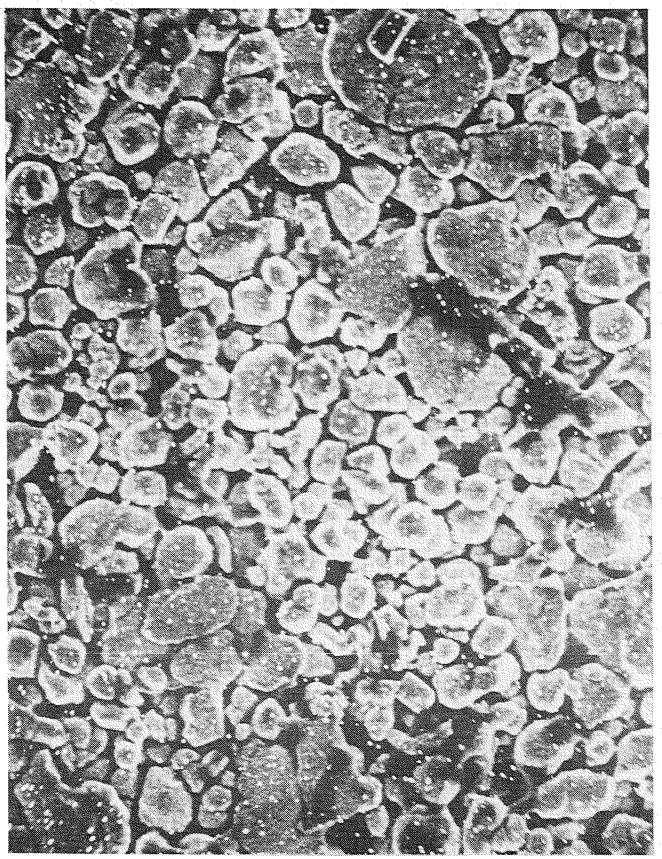


Figure 10. - 58% dense porous tungsten unacceptable bond (2000X).

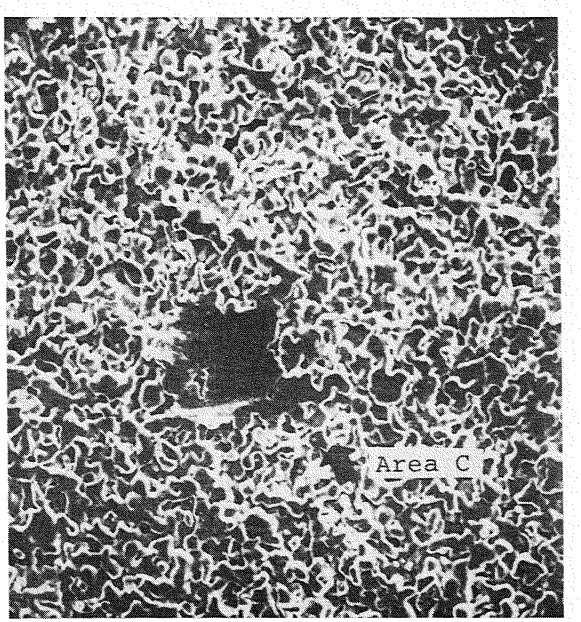


Figure 11. - SEM pictures of porous tungsten contamination (600X).

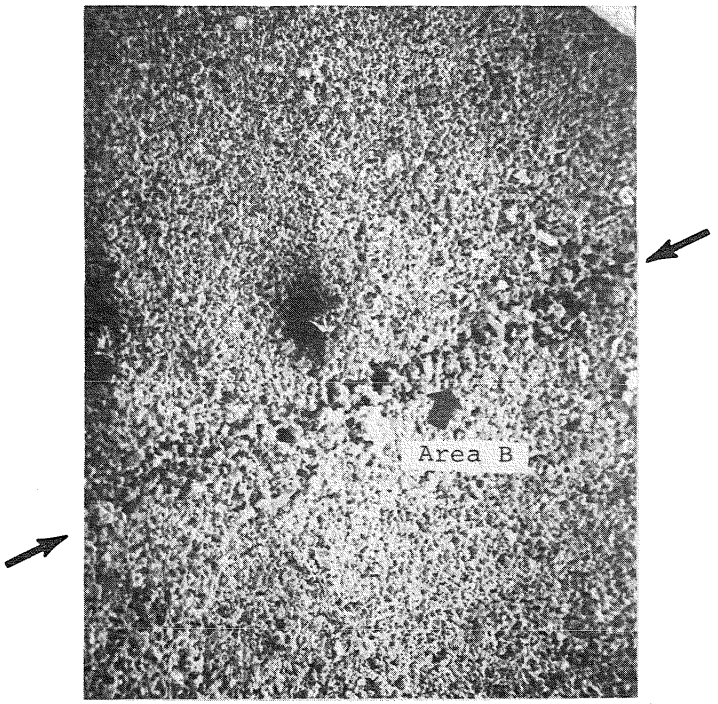


Figure 12. - Line of low density in porous tungsten (200X).

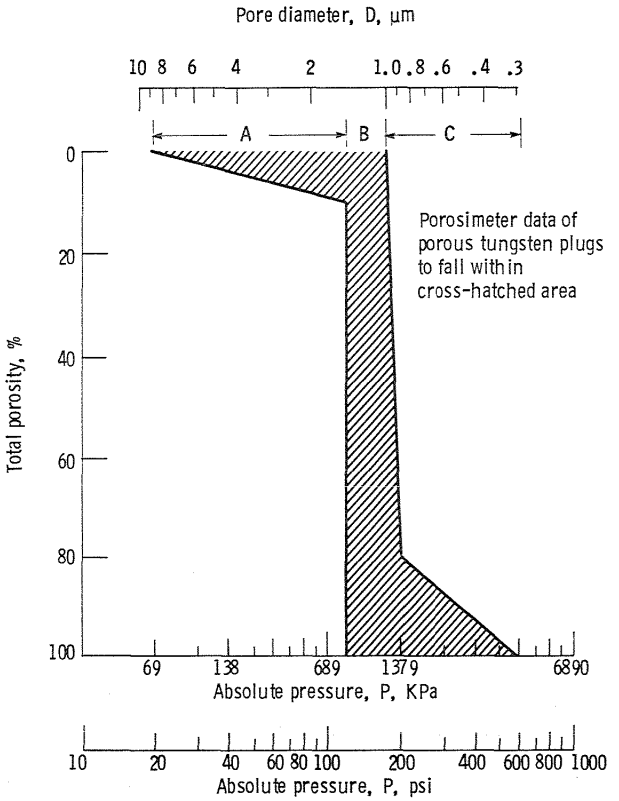


Figure 13. - Porosimeter curve - acceptable range.

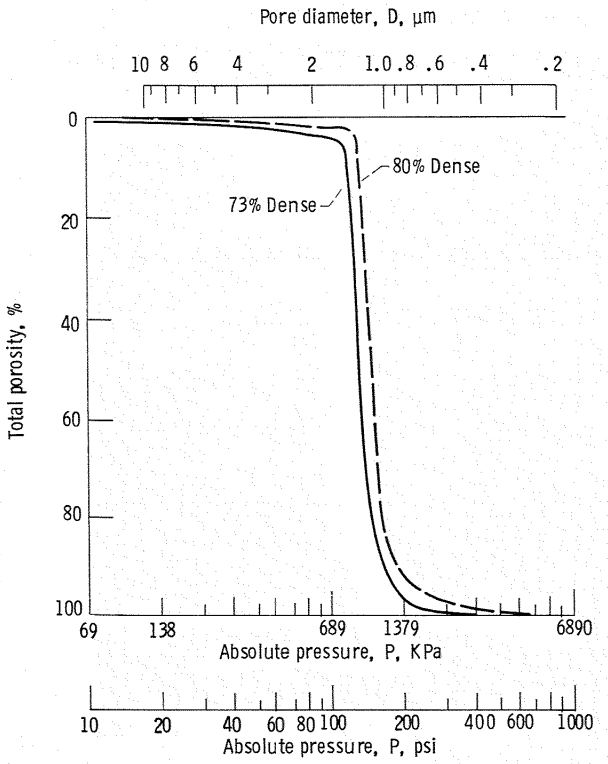


Figure 14. - Porosimeter curve - acceptable 73% and 80% dense material.

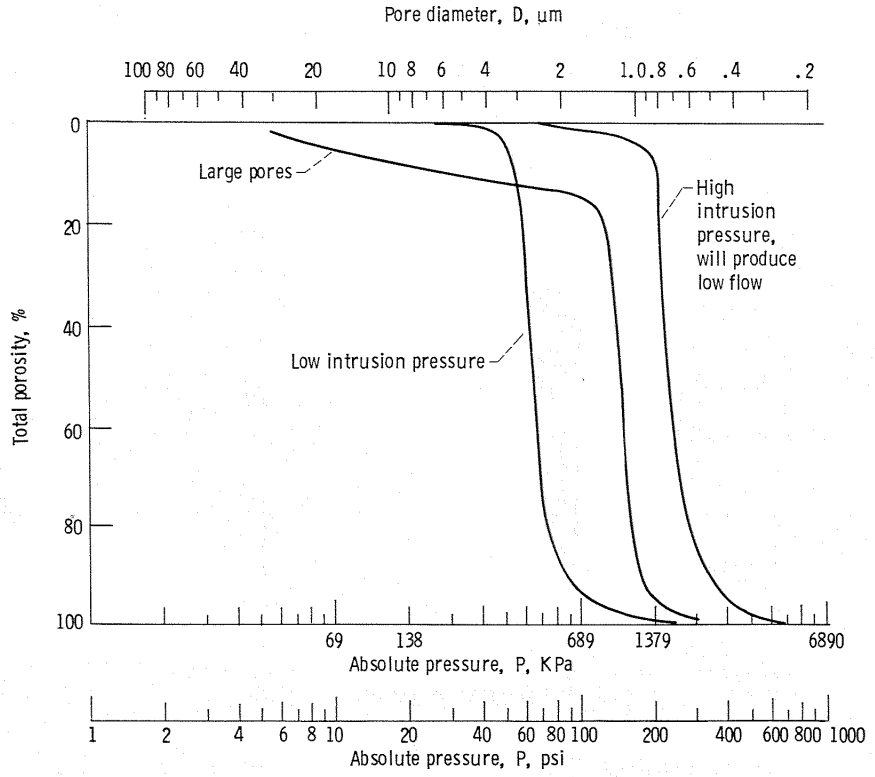


Figure 15. - Porosimeter curve - unacceptable material.

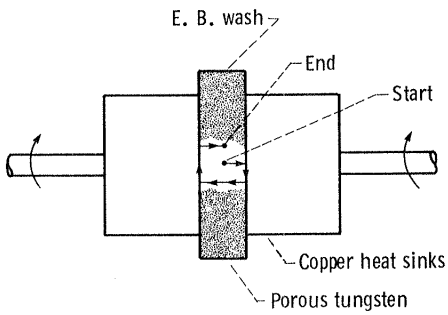


Figure 16. - Electron beam washing.

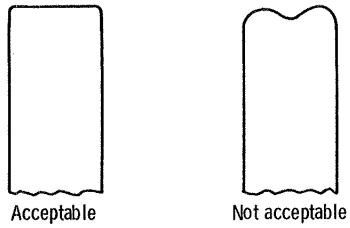


Figure 17. - Electron beam wash - acceptable, not acceptable.

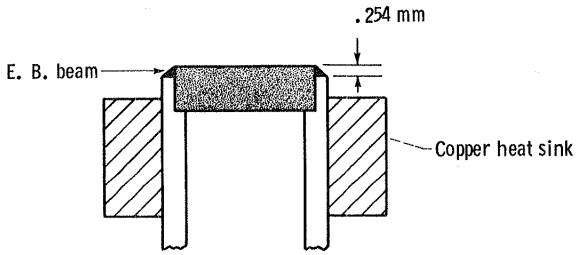


Figure 18. - Electron beam welding-plug to tantalum holder.

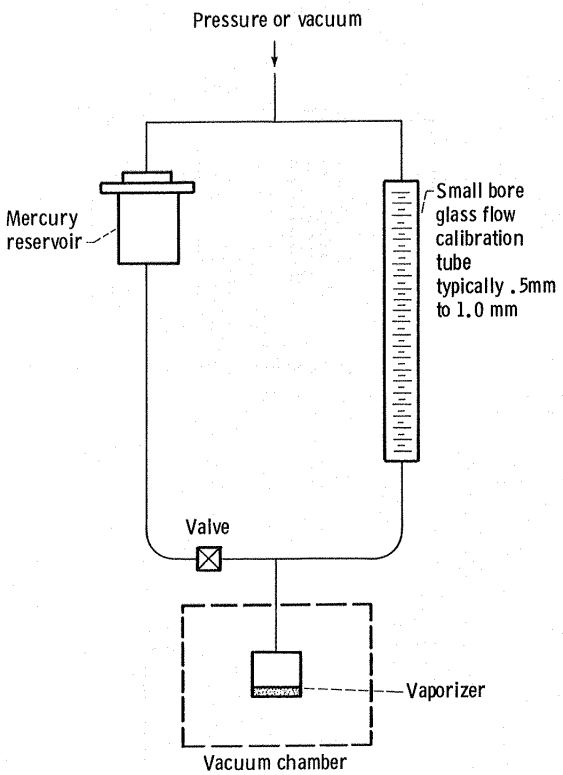


Figure 19. - Intrusion and flow test facility.

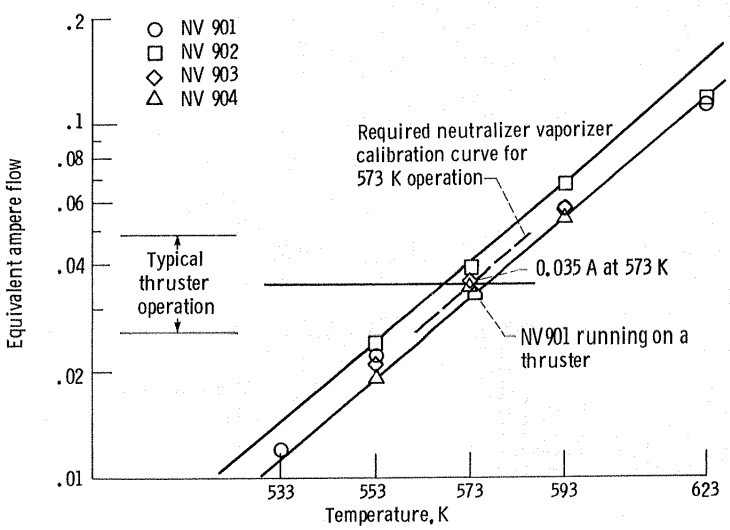


Figure 20. - Neutralizer vaporizer flow data.

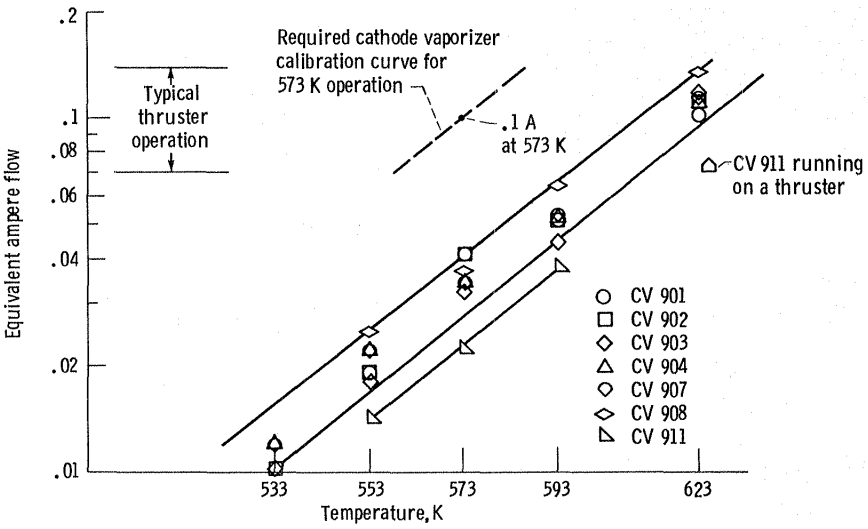


Figure 21. - Cathode vaporizer flow data.

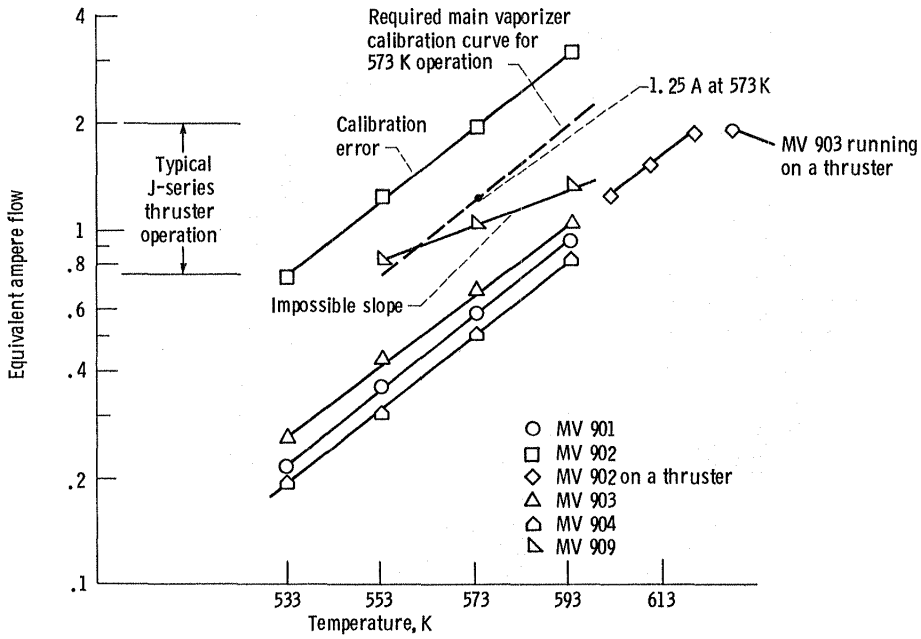


Figure 22. Main vaporizer flow data.

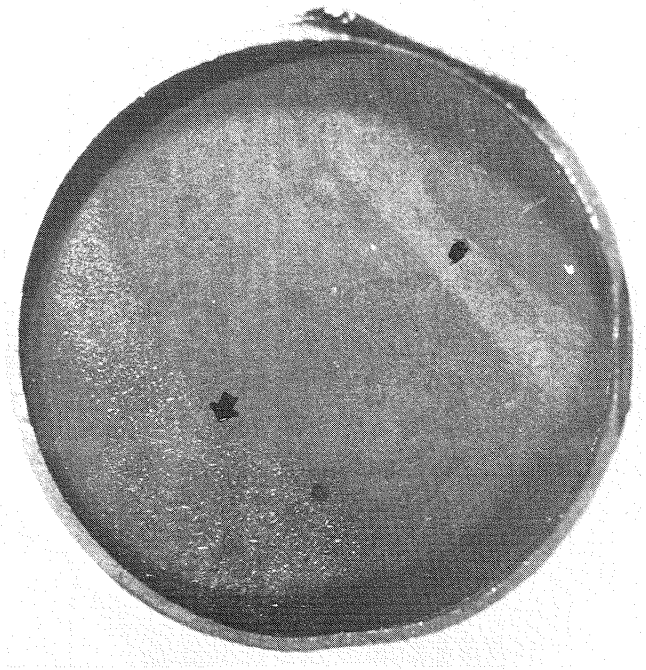


Figure 23. - Liquid side of M-1 plug.

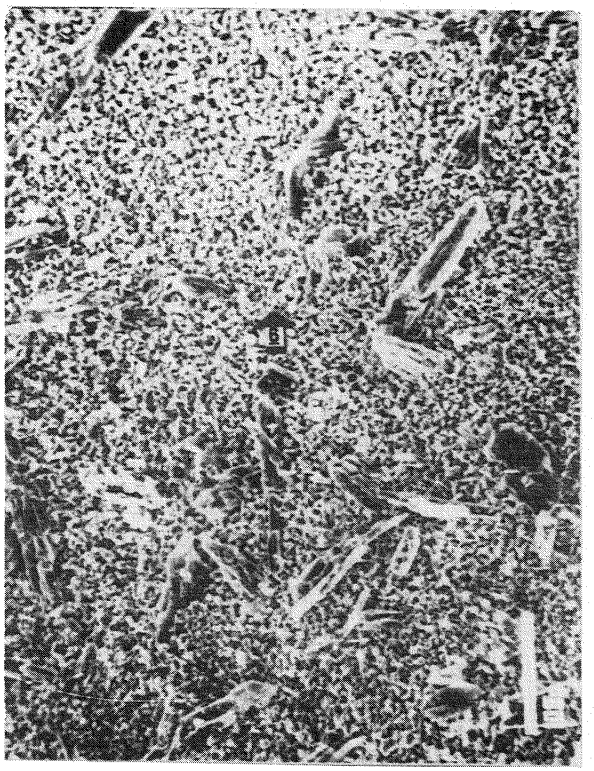


Figure 24. - Liquid side of M-1 plug.

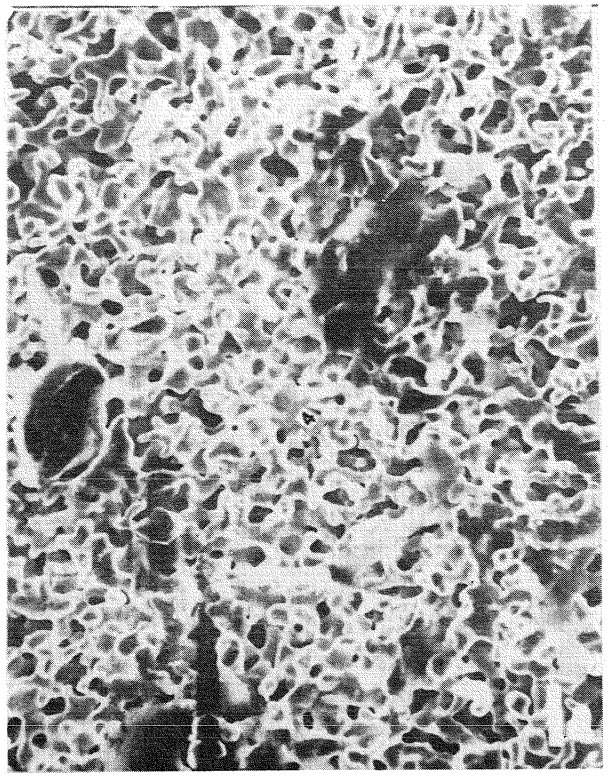


Figure 25. - Vapor side of M-1 plug.

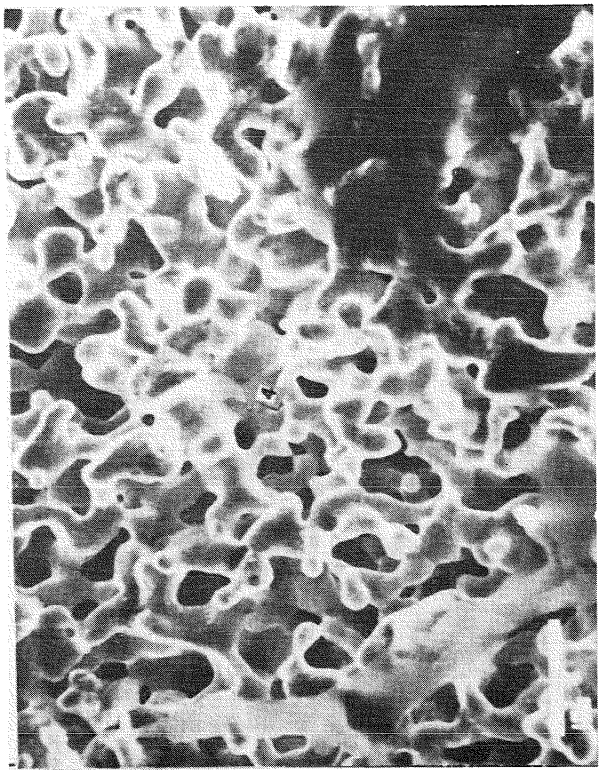


Figure 26. - Vapor side of M-1 plug.

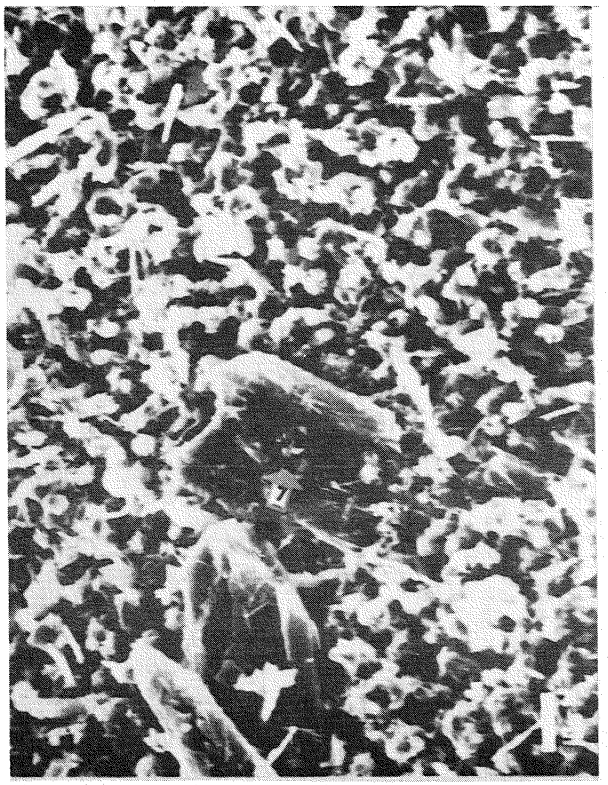


Figure 27. - Liquid side of M-1 plug.

Figure 28. - Liquid side of M-1 plug.

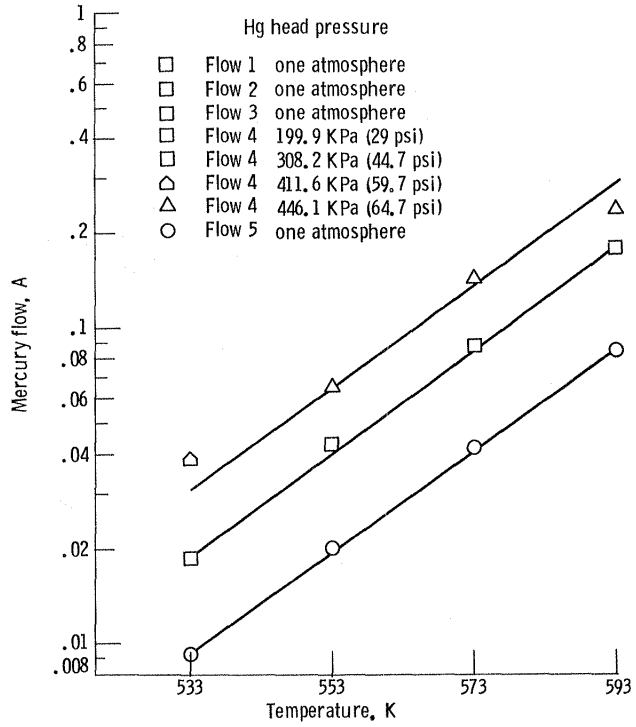


Figure 29. - Flow calibration plot of M-1.

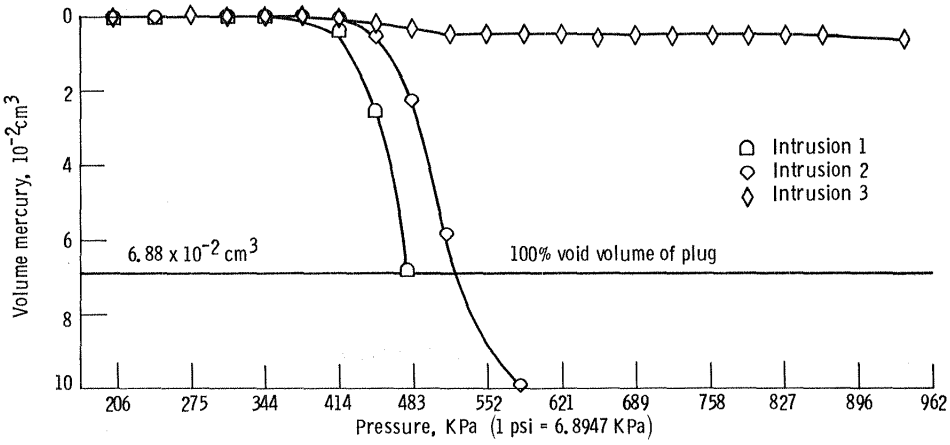


Figure 30. - Intrusion testing plot of M-1.

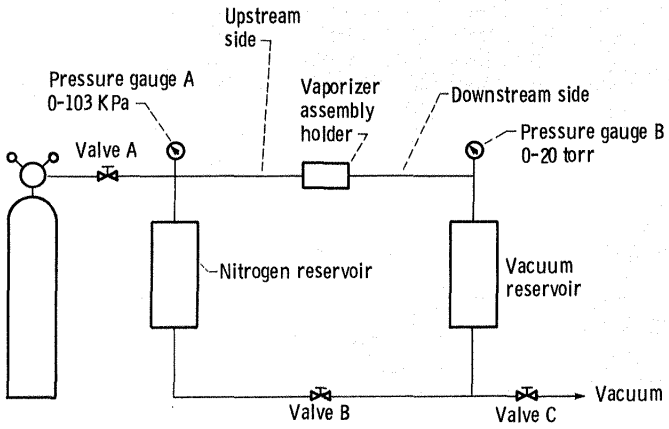


Figure 31. - Nitrogen flow test schematic.

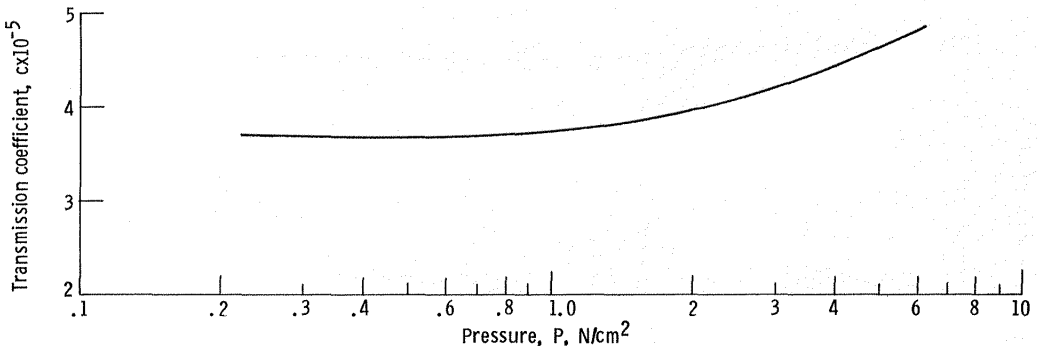


Figure 32. - Typical nitrogen flow test plot.

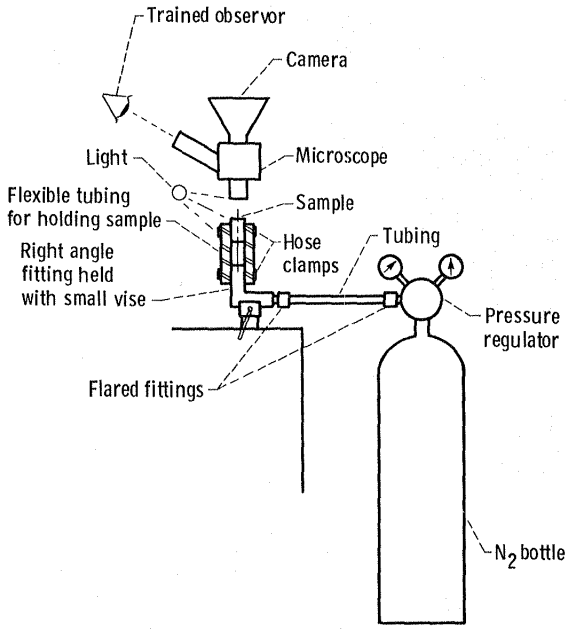


Figure 33. - Bubble test set-up.

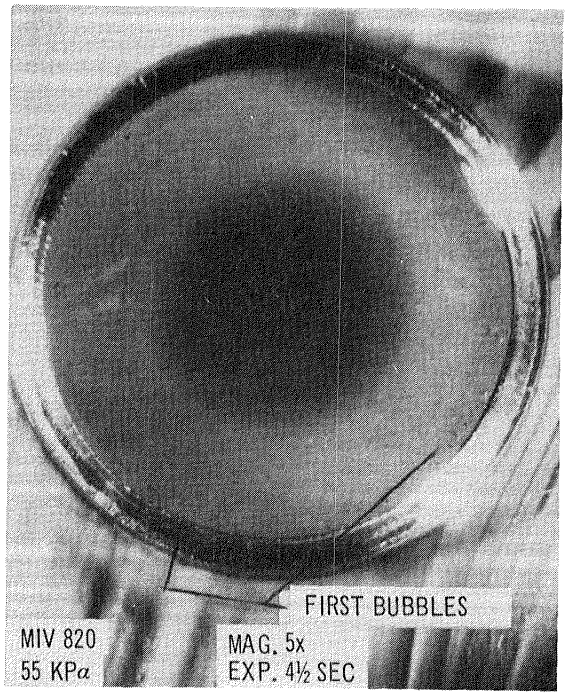


Figure 34. - Bubble testing of porous tungsten plugs.

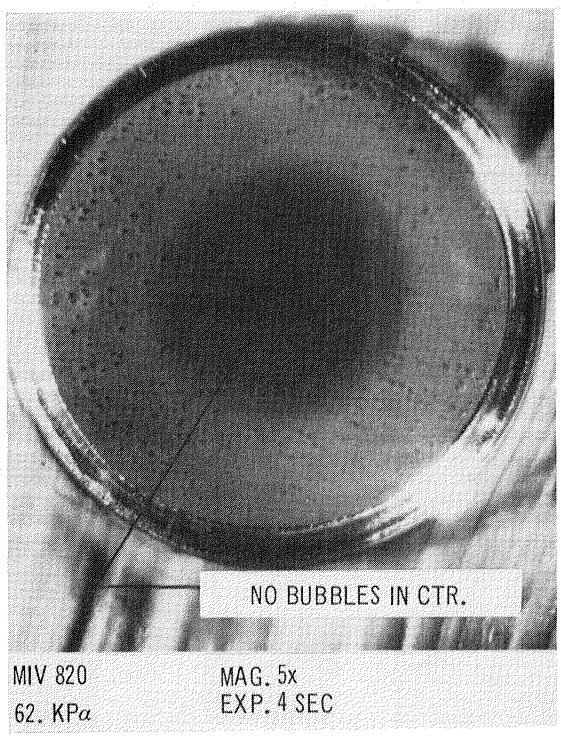


Figure 35. - Bubble testing of porous tungsten plugs.

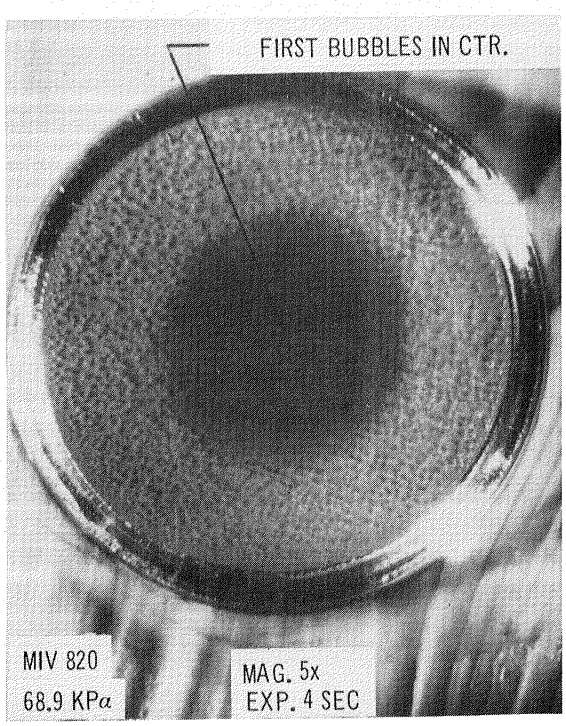


Figure 36. - Bubble testing of porous tungsten plugs.

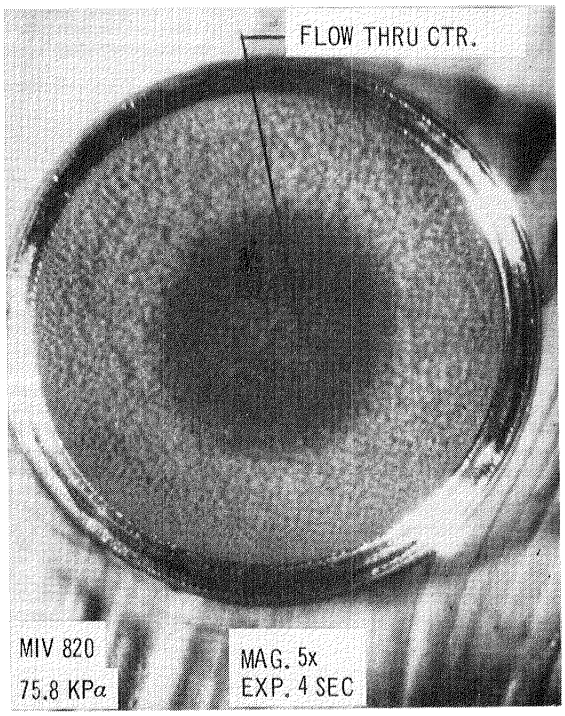


Figure 37. - Bubble testing of porous tungsten plugs.

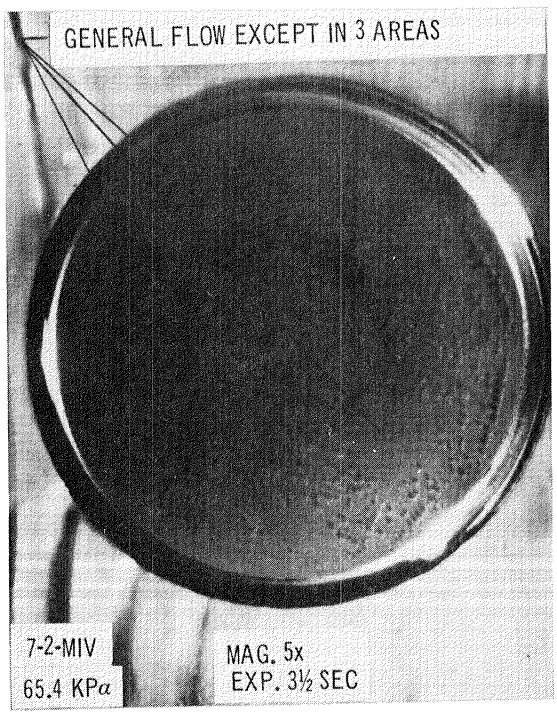


Figure 38. - Bubble testing of porous tungsten plugs.

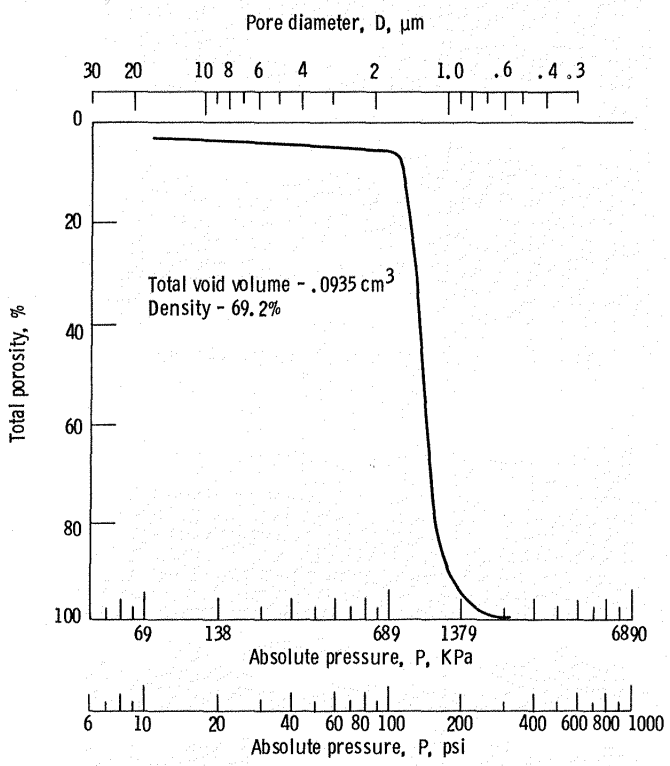


Figure 39. - Porosity determination .

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15. Supplementary Notes

Ralph Zavesky and Erich Kroeger, NASA Lewis Research Center, and Seiji Kami, Hughes Research Laboratories, Malibu, California 90265.

16. Abstract

The dispersions in the characteristics, performance and reliability of vaporizers for early model 30-cm thrusters were investigated. The purpose of the paper is to explore the findings and to discuss the approaches that were taken to reduce the observed dispersion and present the results of a program which validated those approaches. The information that is presented includes porous tungsten material specifications, a discussion of assembly procedures, and a discription of a test program which screens both material and fabrication processes. There are five appendixes providing additional detail in the areas of vaporizer contamination, nitrogen flow testing, bubble testing, porosimeter testing, and mercury purity. Four neutralizers, seven cathodes, and five main vaporizers were successfully fabricated, tested, and operated on thrusters. Performance data from those devices is presented and indicates extremely repeatable results from using the design and fabrication procedures.

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