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DISCUSSION ON OPTIMIZATION OF LARGE OIL-PUMPED ULTRA-HIGH VACUUM SYSTEMS

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ABSTRACT

Discussion is directed toward eventual optimization of the largest diffusion-pump systems. Less than 100 diffusion pump fluid molecules per cm^2/sec are possible to detect using an accumulation method. Optimization discussed demands highest possible system speed compatible with the above contamination rate. Bakeable oil diffusion pump systems, with equal orifice valve-trap units, without conventional baffling, permit theoretical through-put speeds up to 0.3 of the system orifice. The average backstreaming rate of oil in two unconventionally baffled commercial pumps of 6-in. and 10-in. size is reduced to $< 3 \times 10^{-4}$ g/cm^2/24 hr, including heating and cooling the pump boiler. A Ho factor of >0.4 is maintained. Results on trapping with activated alumina, retained by a wire mesh, at both liquid-nitrogen and room temperature are included. A variety of pumping speed measurements and bakeable combination valve-trap units are discussed.

INTRODUCTION

Unless optimization is applied to a restricted class of parameters the term "optimum" can have little or no meaning. In this paper we wish to optimize system pumping speed, negligible contamination rate by the pumping

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means, and the size or extent of the vacuum pumping system. Our principal aim is to pump hydrogen, its isotopes, and helium in large amounts under transient and steady-state conditions at pressures less than 1×10^{-8} torr. Our general vacuum objective[1-3] stems from the controlled thermonuclear research program at Livermore. Before proceeding to experimental results, consider an illustration, some definitions, and brief comments on other means of pumping.

Consider a vacuum chamber. Molecules, under molecular flow conditions, impinge on the wall surfaces of the chamber. Perfect vacuum pumping would occur if each molecule in the vacuum chamber could be made to instantaneously disappear. However, in practice the impingement frequency of molecules on the wall surfaces establishes the maximum pumping speed we can associate with a given vacuum chamber. A Maxwellian free molecular gas in the vacuum chamber has an average velocity $\bar{v}$. The projected area of the walls of the chamber is A. Then the maximum pumping speed S associated with any chamber of projected area A is given by $S = A\bar{v}/4$. Since this maximum speed S is independent of pressure under molecular flow conditions we can use this as our standard of reference. For any chamber, the system pumping speed divided by the maximum theoretical[4] pumping speed gives a figure of merit for the pumping system. Consider the case of a diffusion pump hooked up to the chamber.

We wish to hook up the fastest possible diffusion pump system to our vacuum chamber. At best a diffusion pump system can utilize only all of one end, side or bottom of a vacuum chamber. Accordingly, the system speed can be compared with the speed of the opening of the pumping system. If we define the speed efficiency of the vacuum pumping system, beware. Generally for thermalized gas we are not interested in little holes leading to large pumps.

Rather, systems with entrance opening and pump mouth diameter nearly equal are wanted. Now consider some definitions.

First, let us define the wall condition of our vacuum chamber. Cleanliness is a loaded word. We are not so much interested in atomically clean surfaces as we are in thermally stable surfaces. We do not want loosely bound surface molecules. Immediately after bake-out all surfaces can physisorb at room temperature. Accordingly, we are very sensitive to the rate of contamination delivered by the pumping means. As is well known, steady-state pressure measurements are an unreliable measure of contamination. To define wall conditions we must know the rate of contamination by the pumping means, the kind of contamination, and the average bond strength of the contamination on the wall surface.

Here are some state-of-the-art remarks about pumping methods. Remember, we require rapid continuous and transient pumping of hydrogen and helium at pressures less than 1×10^{-8} torr.

1. Cryopumping. The vapor pressure of solid hydrogen[5] and helium offers the main difficulty here. Multilayers of these gases haven't been pumped at high speeds at 1×10^{-8} torr.
2. Mechanical pumping. Molecular pumping based upon momentum transfer from a rotating or oscillating solid surface to gas molecules must cope with the room temperature velocity of hydrogen and helium. The mechanical strength of materials limits surface speeds of rotors to less than about 5×10^4 cm/second. Contamination by surface mechanism is a second objection.
3. Ion pumping. Continuous ion pumping at pressures less than 1×10^{-8} torr for large amounts of hydrogen has not been demonstrated.

4. Getter-ion pumping. The same remark for ion pumping applies here to helium at pressures less than 1×10^{-8} torr.

5. Diffusion pumps. Presently these pumps offer the longest life at the highest speeds at the lowest pressures. If a high-speed elemental-fluid diffusion pump system is perfected, such as a mercury system,[6] there will be no need to use oil. At present oil pumps still offer a higher system speed without contamination than do mercury pumps.

Let's look at a diffusion pump system:

At the system entrance we need a bakeable, high conductance valve. Next we require an isolation trap to keep contamination from entering the vacuum chamber in detectable amounts. Then a baffle stage is needed to limit the rate at which pump fluid molecules reach the trapping surface of the isolation trap. Below the baffle stage are the jets of the diffusion pump. The diffusion pump exhausts to a foreline pump. Our main concern begins at the entrance to the diffusion pumping system and ends at the foreline. We must maximize the probability that particles in the vacuum chamber will reach the atmosphere through the pumping system. We must minimize the probability of contamination molecules reaching the vacuum chamber from the pumping means; and we must not make the system size too large[4] in relation to the opening to the system. To do this, we must know how the system scaling laws are related to pumping speed and contamination rate.

Scaling laws[4] treating optimized conductances are given in this volume. This related paper shows that in order to optimize the conductance of a complete diffusion pump system, the conductance of the assembled parts must be studied. Accurate calculation of the optimized system conductance cannot be made from the conductance of its separate parts within a factor of two or three. Our efforts to optimize system speed depend on a study of conductance scaling laws for the complete diffusion pump system.

EXPERIMENTAL RESULTS

No completely optimized system is yet finished. Preliminary consideration of results is given separately for pumps, traps and valves. Four possible schemes for complete diffusion pump systems are then discussed.

Presently, diffusion pumps need to be improved to be used in an optimized system. A diffusion pump must be considered from a complete thermodynamic view. In addition to mass transfer of the working fluid of the pump, chemical and physical reactions between working fluid and gases being pumped as well as thermal decomposition of the working fluid must be considered. The following points are important in pump design:

1. Mass flow of high velocity vapor through the top jet of the pump must be adequate; top jet design is especially important for pumping light gases.
2. Proper boiler design is necessary to eliminate eruptive boiling and thermal cracking of oil. Transfer of foreline gases into the vapor jet stack must also be considered.
3. Backstreaming of oil from the pump to the surfaces of the isolation trap must be reduced to less than 1×10^{-5} g/cm^2/hr.
4. The distance between the bottom of the isolation trap and the lower edge of the top jet must be made as small as possible. Perhaps there is even a possibility of raising the top jet into the isolation trap.
5. Perhaps some day a noncontaminating stage can be placed above the top jet of a diffusion pump, thereby eliminating the isolation trap impedance.

Progress on the above points is being made as follows.

Figure 1 shows how oil backstreaming from the top jet of a diffusion pump is reduced. This jet cap modification permits the pump barrel above the top jet to be shortened. For pumps larger than about six inches in diameter, the conventional top jet cap can profitably be improved by heating it, after placing over it a water-cooled cover that extends down into the fringe of the vapor stream. Work at the Oak Ridge National Laboratory[7] led to our present investigation. Without our prior knowledge, results[8] with a water-cooled cap alone have been described.

Referring to Fig. 1, an electric heater is fixed to the top jet cap. Over this heated top jet cap and insulated from it is a water-cooled cover. By bringing the bottom edge of this water-cooled cover down at least 0.6 in. and extending the foot of the cover at least 0.6 in. radially outward, below the bottom of the heated jet cap, backstreaming oil can almost be eliminated. Details of this action of the water-cooled cover are given in Refs. 8 and 9. Supplying heat to the jet cap raises the pumping speed slightly and stops oil backstreaming during starting, running and stopping the pump. Also, the water-cooled cover and heated cap combination allows the barrel of the pump to be shortened nearly down to the level of the bottom edge of the water-cooled cap. Access to the jets by molecules is thereby greatly improved.

Another much smaller source of oil backstreaming originates in the boiler of the pump when bubbling oil can be seen from the top of the pump. We observe small droplets of oil to be thrown upward by the violent action of bubbling oil in the boiler. These upward rising drops can be eliminated by blocking the line of sight to the bubbling oil in the boiler. With the water-cooled cover and heated jet cap combination, 10°C cooling water on the upper portion of the diffusion pump barrel, and with line of sight to the agitated boiler oil blocked, the following average conditions were achieved:

a. Starting the pump with jet cap preheated yielded 2×10^{-4} g/cm^2 of oil.

b. Running the pump with jet cap hot yielded 5×10^{-6} g/cm^2/hour.

c. Stopping the pump, boiler cooled first, yielded 1.8×10^{-4} g/cm^2.

These results were achieved using Narcol 40 oil and an H-10-P 10-inch diffusion pump made by the National Research Corporation. The unmodified pump gave a steady-state backstreaming rate 1000 times larger than the modified pump.

These total amounts of oil backstreaming are determined by catching the oil on a liquid-nitrogen-cooled surface. Upon warming the surface the oil was washed off with acetone into a weighing boat and the acetone evaporated. Not more than 3 mg of oil are lost during each washing and rapid evaporation. By collecting oil samples of from 35 to 150 mg reasonable accuracy is maintained. When the oil collecting surface was maintained at 20°C or higher, less than milligram amounts of oil are collected. Our weighing scheme could not distinguish between the amount of oil collected at −20°C and the amount of oil collected at liquid-nitrogen temperature.

Pumping speed measurements were next undertaken. The 10-inch pump with the above modified jet cap combination was hooked up to a 2400-liter-volume vacuum chamber. Three aspects are studied: 1) The Ho factor, i.e., the ratio of pumping speed to the measured conductance down to the lower edge of the top jet cap. 2) The Ho factor as a function of the molecular weight of the gas pumped.[10] 3) Pumping speed as a function of jet cap and boiler temperature.

Pumping speed was not reduced by using the jet cap combination in Fig. 1. Both steady-state and dp/dt measurements are used, the dp/dt measurement always yielding a value about 15% lower. In the measured range from 1×10^{-4} torr to 5×10^{-7} torr, the pumping speeds were essentially constant. Diffusion-pump speed is reported to be independent of pressure to at least 1×10^{-9} torr. For nitrogen 1940 liters per second is achieved, and 2400 liters per second for helium in steady state. To account for these low values, the conductance from the chamber to the lower edge of the top jet is determined in the apparatus described in Ref. 4, by use of a scale model. In Fig. 1 the ratio of the distance from the chamber to the top jet to the diameter of the pump is 9/10.6. The ratio of the cross-sectional area of the jet cap combination to the cross-sectional area of the pump is 0.3. From the model of the geometry in Fig. 1, a Clausing factor (see Ref. 4) of 0.35 was measured.

The entrance to the pump was then restricted by a thin diaphragm. The ratio of the diameter of the hole in the diaphragm to the diameter of the pump is 6.4/10.6. Measurement, using the above model with this latter restricted access to the top jet yields a Clausing factor of 0.65.

Armed with these two true conductances from the vacuum chamber to the lower edge of the top jet cap, we determined the true Ho coefficient of the 10-in. diffusion pump. By using dp/dt measurements, calibration of the ionization gauges is avoided. In this way, only the vapor stream issuing from the top jet is shown to be responsible for the poor pumping performance with helium. Our reasoning follows: Without the diaphragm in place over the entrance to the pump, the Ho factor for N_2 is 0.84 and for He it is 0.40. With the diaphragm in place, the Ho factor for N_2 is 0.89 and 0.48 for He. With the diaphragm in place, once molecules entered through the hole in the diaphragm they entered the top jet more times before going back into the vacuum chamber. Therefore, the probability of molecules being entrained by the top jet is increased by placing an impedance between the top jet and the vacuum chamber. This impedance leading from the vacuum chamber to the top jet is undesirable, but offers an explanation for the inverse dependence on square root of the mass we previously observed[1] in 1958. Our task in the future is to increase the mass flow in the top jet of the pump and to remove the unnecessary barrel above the plane of action of the top jet.

Results with isolation trap geometries are also encouraging to the achievement of an optimum system. Following Biondi's[11] announcement in 1958 of his work with activated alumina and artificial zeolite in isolation traps we began testing these traps. Figure 2 shows a cross section of our isolation trap test.

We lined the walls of a right-angle elbow with artificial zeolite pellets, type 13X made by the Linde Company. These pellets were contained by stainless-steel wire screen of No. 10 mesh. A highly restricted water baffle was used over an MCF300 pump using Octoil S. The rate at which Octoil S molecules were delivered to the trapping surfaces was therefore determined by the vapor pressure of Octoil S at about 18°C. The purpose of this test was to show that the wire screen holding the beads in place did not permit small angle scatters of the oil molecules to pass through the trap. Our results with Octoil S type-13X artificial zeolite and the MCF300 pump are very similar to results reported by Biondi[11] in this volume.

The maximum number of oil particles passing through the zeolite trap per unit time is estimated as follows. Referring to Fig. 2, this test using artificial zeolite 13X lasted 18 months. A history of the test follows: First the zeolite material must be activated by baking it at high temperature in place in the vacuum system. In this trap, approximately two layers of 1/8-in.-diam by 1/8-in.-long zeolite pellets are used. Forty-eight hours of bake-out and 450°C removed approximately 28 grams of water from 250 grams of zeolite beads. After cooling the outside of the metal trap to room temperature, the pressure registered on a GIC 100 B-A type gauge steadily went down over several hours' time, and equilibrium pressure was finally established at 3×10^{-10} torr. This pressure gradually rose over a period of two weeks until 1×10^{-9} torr was indicated by the gauge. After six more weeks the indicated pressure was 1×10^{-8} torr. After one year's time the indicated pressure was still about 1 × 10^{-8} torr when the test was ended. During this year's time power failures occurred and the diffusion pump was turned off as a test.

With the fine pressure reading less than 1 × 10^{-9} torr, a rubber seat valve was closed in the foreline of the system and then the diffusion pump power was turned off. Twenty-four hours later the diffusion pump was heated up and then the foreline valve was opened. The pressure indicated by the ionization gauge recovered to 2 × 10^{-9} torr within 35 minutes after starting the diffusion pump. Another test was made where the power to the diffusion pump was off for 11 days. Again the ionization gauge recovered its former reading within about 45 minutes from the time the pump was started. During the time the diffusion pump was off, the pressure in the system was not higher than about 2 × 10^{-4} torr. Under these conditions the oil was apparently still being trapped by the zeolite material.

An attempt to evaluate the upper limit for oil particles passing through the isolation trap was based upon two ideas. Having only an ionization gauge at our disposal, in the first method we used a shutter in front of the entrance to the gauge as well as an accumulation and desorption procedure. By moving a zeolite-covered shutter in front of the opening to the gauge we could increase the number of collisions which the oil particles made before entering the tabulation to the gauge. No difference in pressure reading over many days with the shutter in place in front of the gauge and with the shutter removed could be seen.

In the second method, qualitative information was gotten by turning the gauge off for varying periods of time. If the gauge is left off for longer and longer time periods the amount of gas desorbed upon turning the gauge on should increase linearly with time if oil is entering the gauge. Using this method, no positive indication that oil molecules were entering the gauge was obtained. The procedure was as follows:

By cooling the envelope of the gauge with liquid nitrogen and then allowing the walls to desorb the molecules they accumulated while cold, an idea of the heat of liquefaction of molecules picked up on the cold walls could be obtained. Oil, having a high heat of liquefaction, should be adsorbed on cold surfaces in multilayers. If the background gases were very light fractions of cracked oil or other gases with low heats of liquefaction only single monolayers can be formed on the liquid-nitrogen-cooled surface. Without furnishing conclusive proof, this adsorption, desorption, pressure measurement method indicates that the slow rise in pressure starting from the base pressure of the system may not be due to transmission of oil molecules into the ionization gauge.

The above pressure behavior evidence is not adequate for our purposes. Therefore, to assess contamination accurately, a mass spectrometer combined with surface adsorption, desorption technique is proposed. A similar technique to that used with the ion gauge will be used. Contaminant material issuing from a diffusion pump system will be collected on a large surface at liquid-helium temperature. This surface will then be isolated from the pump by a valve. Material collected on the cold surface will be desorbed by heating the surface. A sensitive mass spectrometer will sample the resulting pressure rise. Our mass spectrometer will have a steady-state sensitivity of about 1×10^{-11} torr. Using this spectrometer, with the above desorption technique as few as 100 contaminant particles issuing out of the diffusion pump system per square centimeter of cross section per second may be detected. Quantitative description of the thermal stability of surfaces in contact with our vacuum will thus be obtained.

Figures 3a and 3b show four possible cross sections of optimized diffusion pump systems. The system conductance for each of these schemes down to the lower edge of the top jet is given in another paper in this volume.[4] There it is shown that Clausing factors for the system between 0.3 and 0.5 are possible. A valve action is included in each of the isolation traps without paying for the additional impedance of a valve. The blocking plate in each of the isolation trap schemes can be moved in front of the entrance to the diffusion pump system. Let us consider this valve action.

The valve action is created by gripping the edge of a solid metal valve disc by hydraulic force acting in the plane of the disc. The hydraulic force is introduced into either of two metal bladders coupled together at right angles around the edge of the disc. The vacuum seal is created by one bladder acting in the plane of the disc radially inward and outward. Inflating the other bladder which acts normal to the surface of the disc deflates the bladder around the edge of the disc. By the combination of these two bladders an expanding and contracting pressure-actuated valve seal is created. Work on a practical realization of this valve idea is continuing.

Clearly, Fig. 3 does not exhaust all the possible designs for optimized systems. For our own particular requirements, these four schemes come very close to the theoretically possible maximum established by the conductance measurements.

CONCLUSION

An outline for one class of optimized, ultra-high vacuum systems using oil diffusion pumps has been given. Preliminary results for parts of these idealized systems indicate the practical possibility of the optimized schemes presented. Much future work is necessary to bring these plans to successful conclusion.

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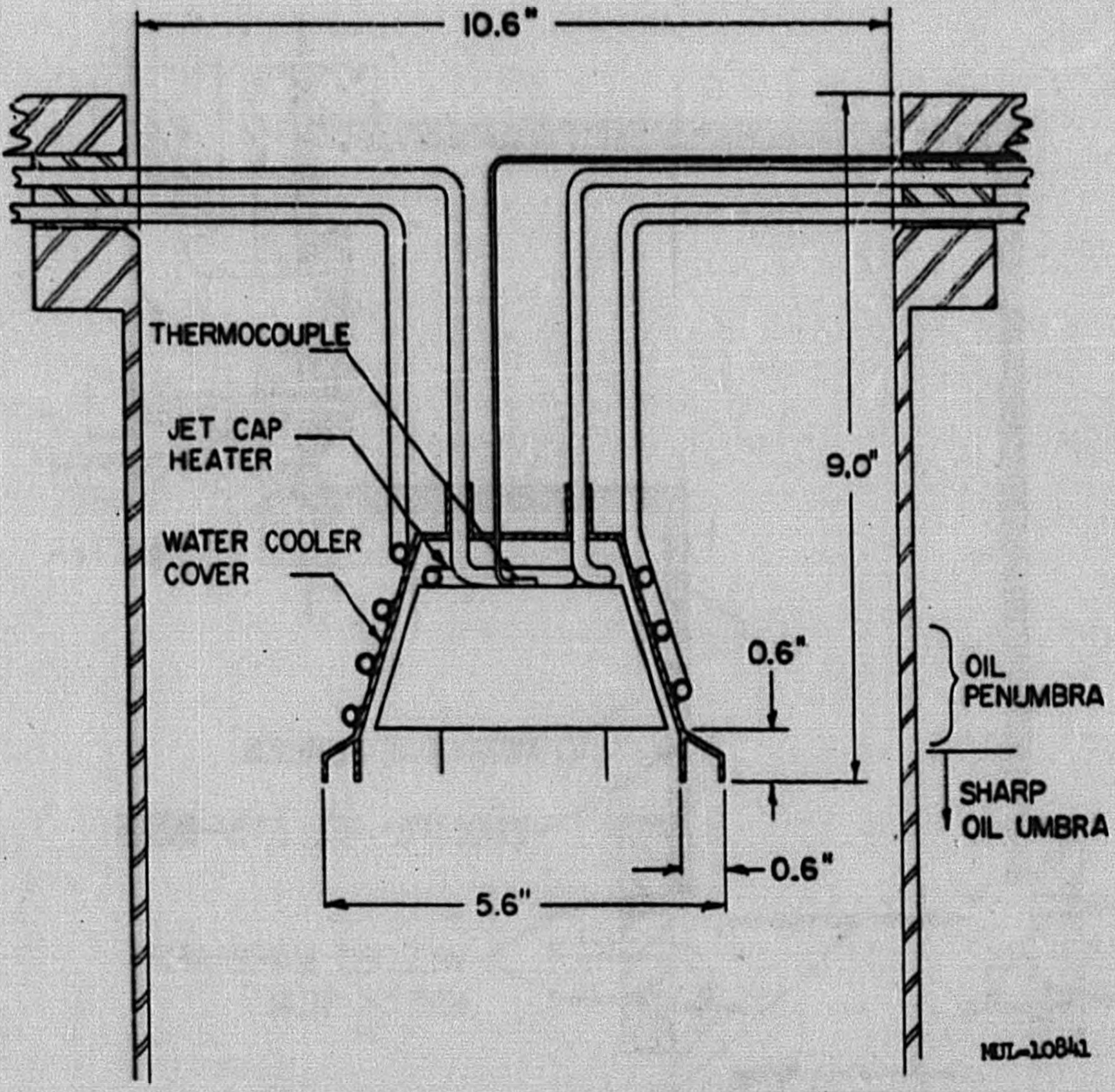

Fig. 1. Cross section of heated jet cap with water-cooled cover.

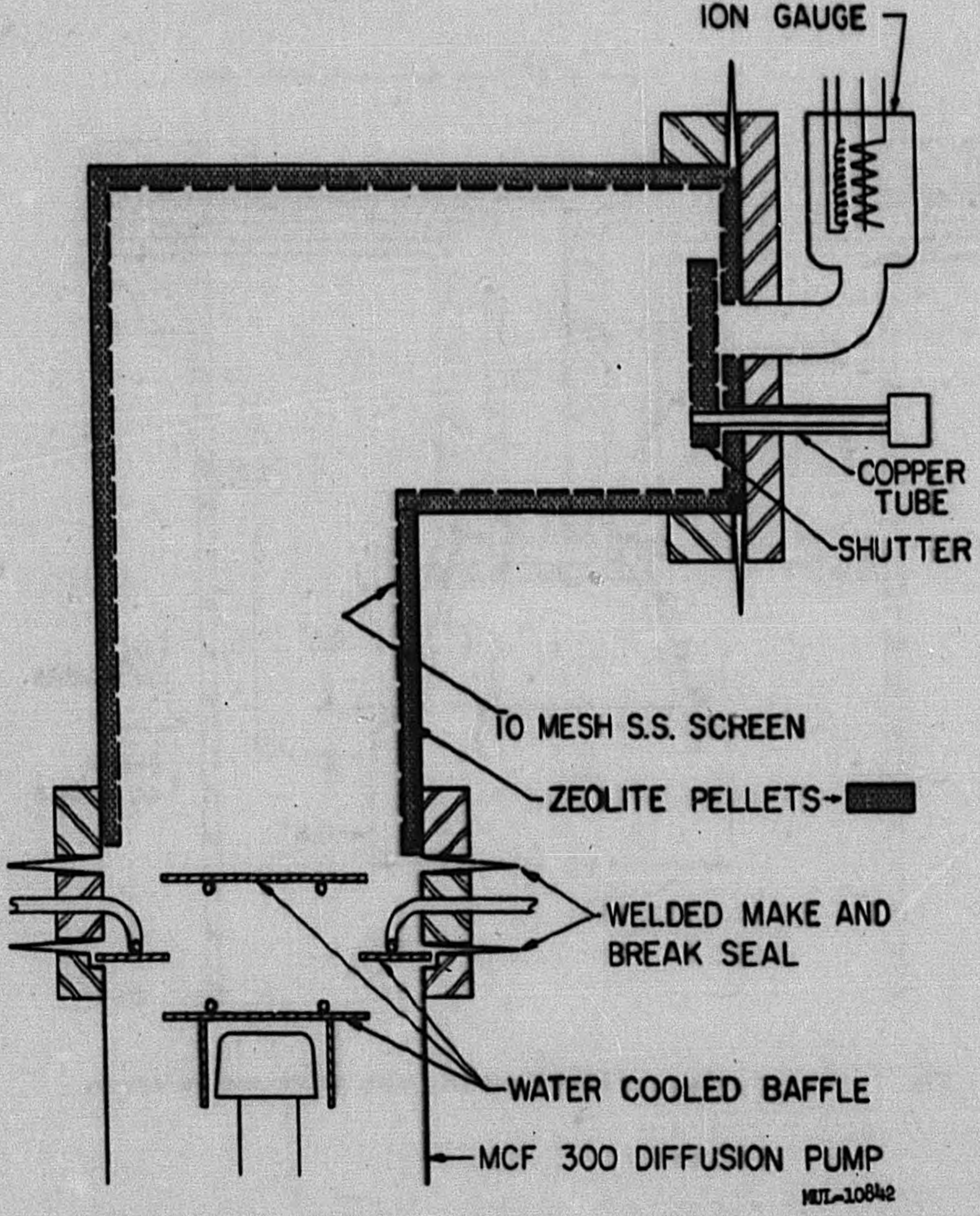

Fig. 2. Cross section of right-angle isolation trap experiment.

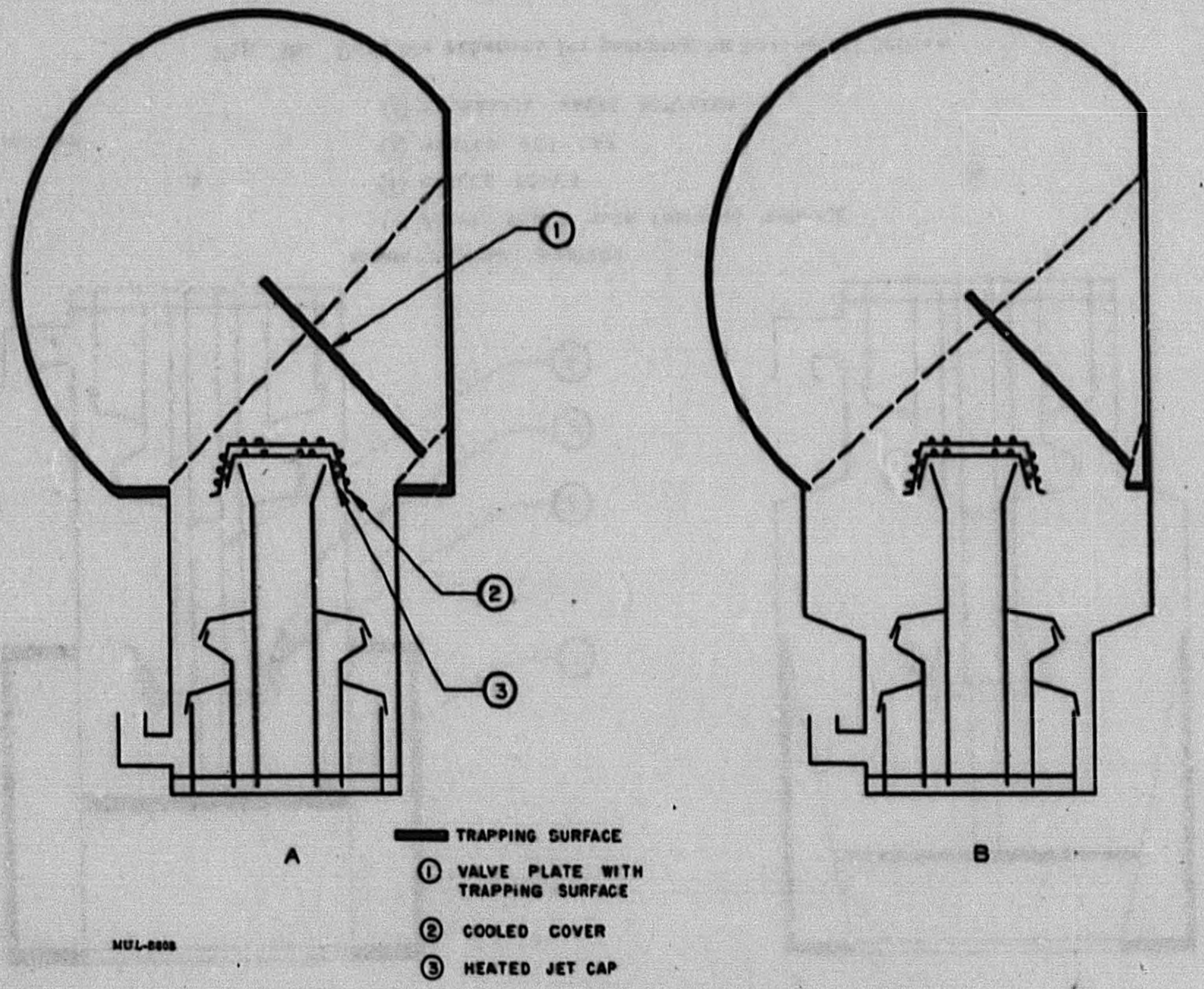

Fig. 3a. Possible schemes for pumping on vertical orifice.

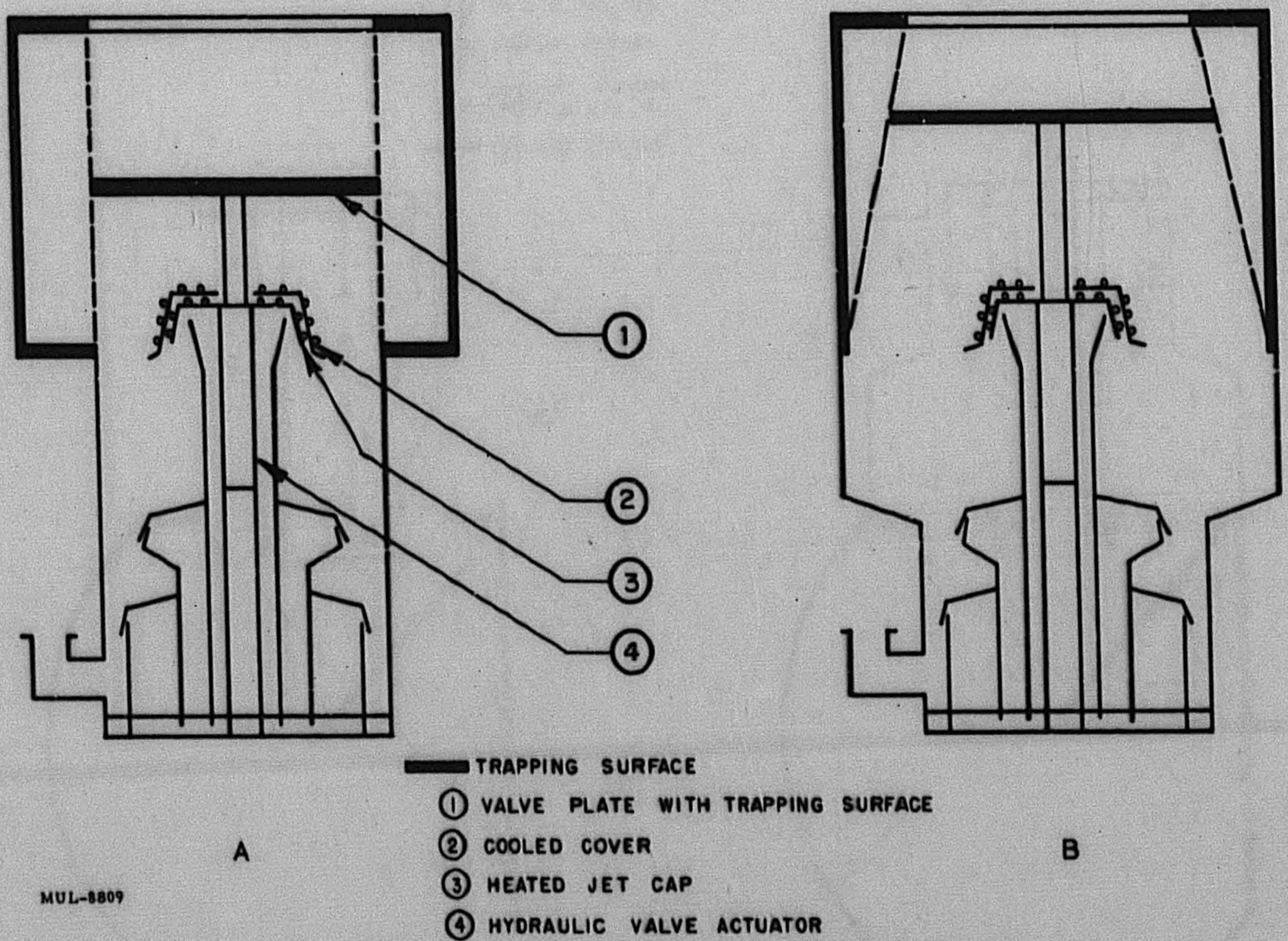

Fig. 3b. Possible schemes for pumping on horizontal orifice.