

July 26, 1966

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3,262,279

EXTREME HIGH VACUUM APPARATUS

Filed Oct. 9, 1964

3 Sheets-Sheet 1

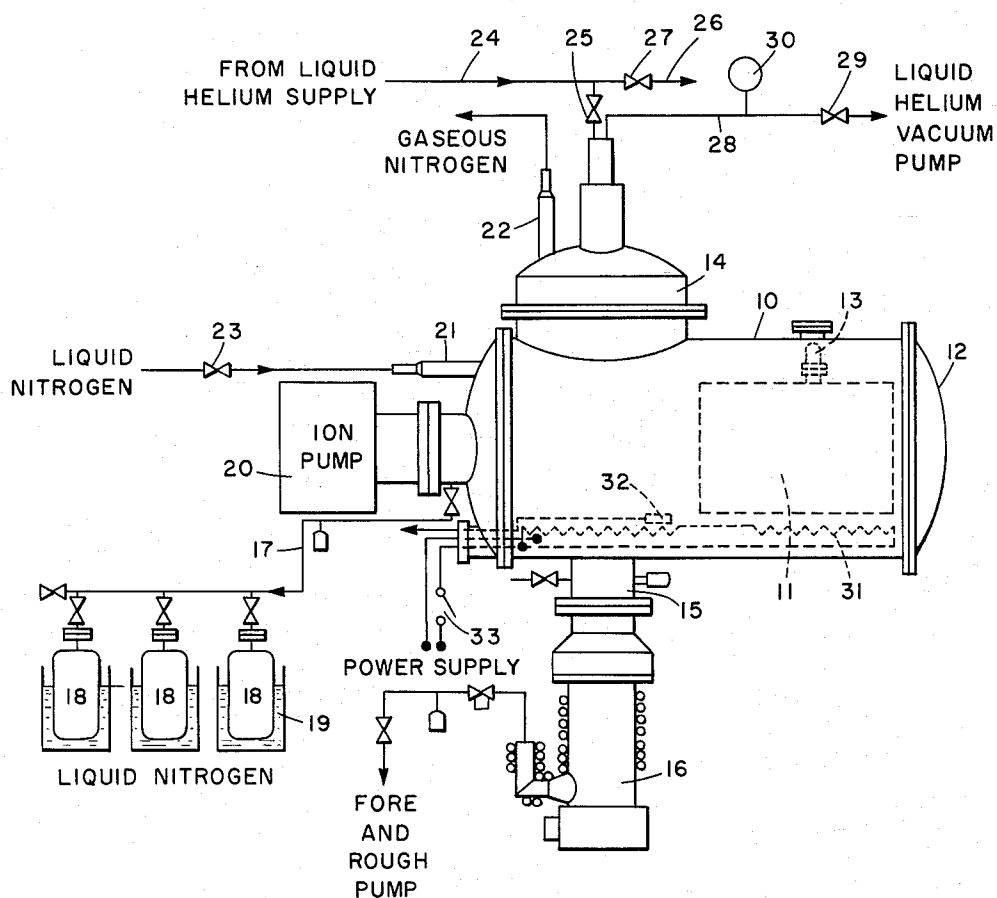


Fig. 1

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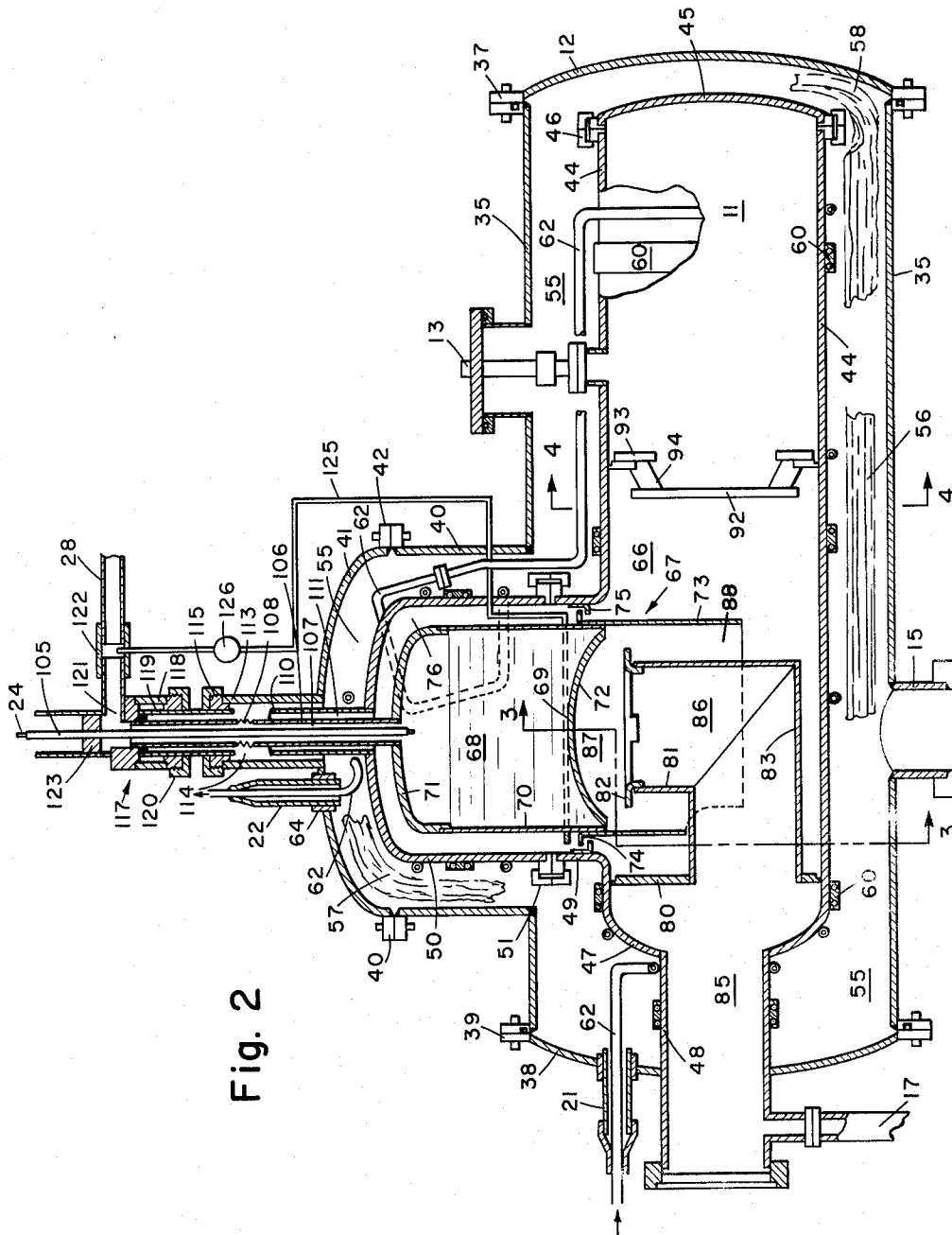
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Filed Oct. 9, 1964

3 Sheets-Sheet 2



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3 Sheets-Sheet 3

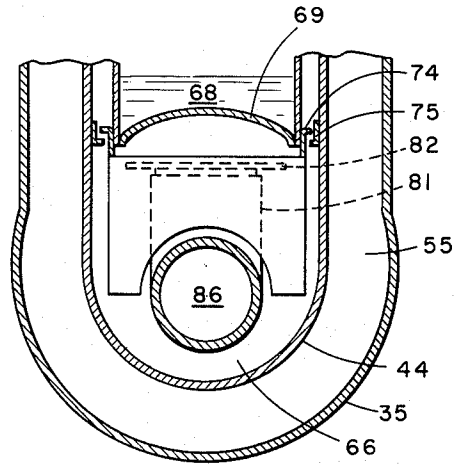


Fig. 3

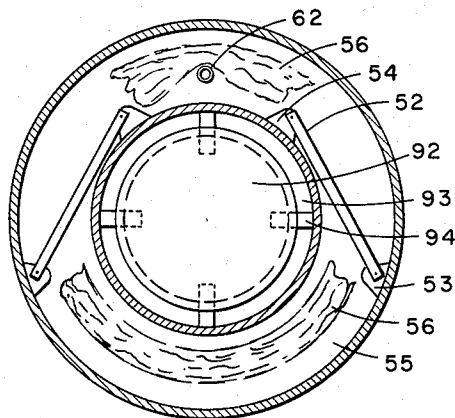


Fig. 4

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EXTREME HIGH VACUUM APPARATUS

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Filed Oct. 9, 1964, Ser. No. 402,898

8 Claims. (Cl. 62—45)

This invention relates to high vacuum chambers and more particularly to high vacuum chambers in which pressures below 10^{-12} torr may be attained.

There is now a growing need for vacuum systems which are capable of attaining pressure levels in the range of 10^{-13} or even 10^{-14} torr. This need is brought about through the desire to study the effects of the extremely low pressures of deep space on systems which must work in this environment (where pressures of about 10^{-14} torr are thought to exist) and by the increasing interest of material research in studying "clean" surfaces. Work of this type requires a vacuum system which meets two basic technical requirements. First, in order to maintain "clean" surfaces, it should not contaminate test specimens with "foreign" molecules emanating from the vacuum system; and second, in order to produce "clean" surfaces, it should rapidly remove gas molecules emanating from the test specimen.

The extreme high vacuum chamber must be unusually "clean." The cleanliness of a vacuum system without experiment is characterized by its ultimate pressure when empty. The rate of molecular bombardment of a surface in the chamber is directly proportional to the pressure. At 1×10^{-10} torr enough molecules strike a surface so that, if each one was sorbed, a complete surface coverage would be produced in about 4 hours. Thus, for example, if one wishes to maintain a "clean" specimen surface for 400 hours, it is necessary to insure that the partial pressure of any gas specie that might be strongly sorbed is very small compared to 10^{-12} torr, say 10^{-14} torr.

The ability of the vacuum system to produce clean surfaces is dependent on its pumping speed relative to the size of the specimen. The importance of this factor is not well recognized. Most ultra-high vacuum systems now used do not provide very high pumping speeds because it would be expensive to do so. Speeds of 500 to 1,000 liters/second for air are common in 1-3 feet diameter systems. Accurate simulation of space for sizable specimens would require pumping speeds which are orders of magnitude higher. However, even a speed of 1,000 liters/second can provide useful space simulation and can produce clean surfaces in a reasonable time if the test specimen is small.

Current practices to meet these requirements include bakeout of the chamber (to reduce its outgassing rate and lower the achievable ultimate pressure), cryogenic baffling of pumps (to prevent backstreaming of pump fluid), the use of metal seals around ports (to eliminate contamination from elastomer outgassing), and the use of higher speed pumps (diffusion, ion or sublimation types). The all-metal or glass chamber construction which can be baked and which have common pumping means can attain empty chamber pressures down to about 1×10^{-11} torr in a reasonably short time.

The removal of molecules from an atmosphere by condensing them on a cold surface, i.e., by the process of cryopumping, has been employed for several years in the attainment of higher vacuums. However, in itself cryopumping is not the solution to the attainment of pressures of the order of 10^{-13} or 10^{-14} torr. It is rather an important adjunct and in the extreme high vacuum chamber of this invention it is used as such.

To attain extreme high vacuum it is desirable to maintain high pumping speeds at very low pressure levels.

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The vacuum chamber of this invention is specifically designed to achieve such high pumping speeds within a system which itself does not contribute to the contamination of the sample contained therein. The vacuum chamber to be described makes extensive use of cryogenic technology in two ways as will be apparent from the description of the apparatus. First outgassing from the walls of the vacuum chamber is made negligible by cooling them to near 80°K .; and second, high pumping speeds at the low pressures required are provided in the chamber by cryopumping surfaces maintained at temperatures from about 2.5° to 4.2°K . The flow path of the residual gases is so designed to provide ready flow of gases from the work space to the cryopumping region and at the same time to make it difficult for gases from the warm end of the system to reach the work space.

It is therefore a primary object of this invention to provide an extreme high vacuum chamber which can attain pressure levels of the order of 10^{-13} torr or even lower. It is another object of this invention to provide a high vacuum chamber of the character described which may be used as a ready working tool and which is fundamentally simple to use and operate. It is another object of this invention to provide such a vacuum chamber which can be readily placed in operating condition, which can contain samples of relatively large size, and which is flexible in its use. Other objects of the invention will in part be obvious and will in part be apparent hereinafter.

The invention accordingly comprises the features of construction, combination of elements and arrangement of parts which will be exemplified in the constructions hereinafter set forth, and the scope of the invention will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention reference should be had to the following detailed description taken in connection with the accompanying drawings in which

FIG. 1 is a schematic layout of the high vacuum chamber along with the attendant equipment;

FIG. 2 is a longitudinal cross-section of the high vacuum chamber of this invention;

FIG. 3 is a cross-sectional view taken along line 3—3 of FIG. 2; and

FIG. 4 is a cross-sectional view taken along line 4—4 of FIG. 2.

A schematic layout of the extreme high vacuum chamber along with the attendant pumps and feedlines is shown in FIG. 1. Within the high vacuum chamber, generally indicated by the numeral 10 is a test chamber 11 to which access is had by means of a removable door 12. Associated with the test chamber is a suitable vacuum gauge 13, preferably of the cold cathode type to minimize any outgassing from the cathode at the very low pressures in the test chamber. Within the vertical portion 14 is the cryopumping system which will be described in detail with reference to FIG. 2. A suitable conduit 15 leads to a diffusion pump 16 (with its attendant valves, etc.) used to pump out the guard vacuum about the inner vessel as detailed in the description of FIG. 2. A roughing line 17, with appropriate valving, communicates between the inner vessel and three sorption roughing units 18, immersed during use in liquid nitrogen 19. There is also provided an ion pump 20 further to pump down the inner vessel.

The inner vessel is cooled by circulating liquid nitrogen through coils which are in thermal contact with its outer walls. This liquid nitrogen is introduced to the coil through a conduit entering the system through extension 21 and gaseous nitrogen is removed through a suitable conduit leaving the system through extension 22. Valv-

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ing means 23 are provided to control the rate of liquid nitrogen flow.

Liquid helium is supplied from a suitable source to the cryopump section by way of transfer line 24, controlled by valve 25, and boiled off helium gas during cool down is removed through line 26 controlled by valve 27. The liquid transfer line is illustrated in detail in FIG. 2. In order to provide a partial vacuum over the liquid helium contained in the cryopumping section a line 28, controlled by valve 29 and incorporating a pressure gage 30, leads to a liquid helium vacuum pump.

For baking out the inner vessel there is supplied a heating system diagrammatically represented as a heater 31 which has associated with it a suitable temperature-sensing means 32 and switch 33.

The extreme high vacuum chamber is illustrated in detail as a longitudinal cross-sectional view in FIG. 2. It is in essence a double-walled vessel defining a guard vacuum between the walls. The outer vessel will be seen to consist of a cylindrical section 35 and a test-chamber end piece 12 which is fastened to the cylindrical section by means of suitable vacuum-tight flange sealing means 37. On what may be termed the warm end of the chamber is an end piece 38 fastened in vacuum-tight relationship to cylindrical section 35 through similar flange sealing means 39. Sealed or welded to the cylindrical section 35 is a vertical portion 40 which has a top piece 41 fixed to the vertical section 40 by a vacuum-tight flange seal 42. Thus it will be seen that the interior of the outer vessel is readily accessible for insertion of equipment and that it can be easily assembled.

The inner vessel comprises a cylindrical section 44 and a test chamber end piece 45 which is affixed to the cylindrical section through sealing means 46. On the warm end the inner vessel terminates in an end piece 47 into which a warm end extension 48 is sealed. As will be apparent this warm end extension 48 extends beyond the outer vessel to make contact with the ion pump 20 (FIG. 1) and with the roughing line 17. The inner vessel has a vertical portion positioned within the vertical portion of the outer vessel and this consists of a cylindrical section 49, a top section 50, and suitable joining means 51. It will be seen that the inner and outer vessels can be readily dismantled and assembled and that the test chamber 11 is readily accessible by the removal of outer vessel end 12 and inner vessel end 45.

In the spacing defined between the inner and outer vessels there is a guard vacuum 55 which is typically maintained at a pressure of about 10^{-5} torr or less by means of the diffusion pump 16 (FIG. 1) connected to the outlet 15 of the outer guard vessel. It is preferable to fill at least a portion of this guard vacuum space 55 with a suitable insulating material such as a quartz fiber batting shown in fragmentary sections in FIG. 2. It will be appreciated that this insulating material extends substantially around the entire inner vessel, there being a cylindrical section 56 and a conical hat-shaped section 57 which can be dropped over the vertical section in the process of assembling the vacuum chamber. The test chamber end portion of the guard vacuum 55 contains an insulating cap 58.

The inner vessel is supported within the outer vessel by suitable means such as by spaced sets of arms 52 (FIG. 4) which are attached through supports 53 to the inner wall of the outer vessel and through supports 54 to the outer wall of the inner vessel.

In order to remove the foreign gases which are associated with the inner wall of the inner vessel and thus to render it as clean as possible, it is desirable to be able to bake this inner vessel up to temperatures of about 750° F. for a sufficient period of time. To do this heaters are provided which are shown in FIG. 2 to be electric hermetically sealed tubular-type heaters 60 which are wound around both the horizontal cylindrical portion as well as the vertical top portion of the inner vessel.

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During operation of the test chamber the inner vessel is maintained at essentially liquid nitrogen temperature through the circulation of liquid nitrogen in coil 62 which is also wrapped around the horizontal and vertical portions of the inner vessel. The liquid nitrogen coil is brought in through the fixed extension 21, welded to the end 38, and leaves through the top of the outer vessel through extension 22 which in turn is sealed to the top through movable sealing means 64.

Within the volume 66 of the inner vessel which is that portion of the cylindrical section not occupied by the test chamber and extending up into the vertical section is the cryopumping portion 67. The cryopump will be seen to consist of a vessel 68 capable of containing liquid helium, this vessel comprising a convex bottom 69, a cylindrical side section 70, and a top 71. The exterior surface 72 of the bottom section 69, which is of course cooled by the liquid helium, presents a cold surface ranging in temperature between 2.5° and 4.2° K.

Thermally bonded to the bottom section of the cylindrical section 70 of the helium vessel is a copper skirt 73 which terminates at its upper end in a molecular flow shield 74. A second molecular flow shield 75 attached to the inner wall of the inner vessel provides some overlapping with the molecular flow shield 74 thus impeding the flow of any gases into the passage 76 which is defined between the helium vessel and the inner vessel. In place of the liquid helium vessel coils suitable for circulating liquid helium in heat exchange relationship with skirt 73 and a dished surface equivalent to vessel bottom 72 may be used to cool these cryopumping surfaces.

Within the inner vessel there is positioned a solid apertured plate 80 to which is sealed an elbow 81 extending up into and partially surrounded by copper skirt 73. As will be apparent from FIG. 3 the copper skirt does not make contact with the elbow. Around the top edge of the elbow 81 is placed a ring 82, the purpose of which will become apparent in the description of the operation of the cryopump. The interior surface 83 of the elbow is blackened to absorb radiation. It will be seen that there is a gas path from the warm end of the high vacuum chamber to the test chamber 11 which is comprised of the passage 85 defined by the warm end extension 48, passage 86 which is within the elbow 81, volume 87 which extends between the cold helium vessel bottom 72 and the upper portion of the copper skirt 73, and finally the annular passage 88 which is defined between elbow 81 and skirt 73. The geometry of this path is of particular importance since it is designed to prevent gases from reaching the test chamber 11 while at the same time it provides an easy means for gases to be removed from the work space. Any gas which is to reach the work space or test chamber 11 must do so by means of the cryopumping section. That is it must go by way of passages 85 and 86 and upon its entry into volume 87 it will strike the cold surface 72. If it is not condensed on this surface upon striking, it will be reflected back onto the ring 82 and then rereflected upon the cold surface 72. Thus the opportunities for the gas molecules to enter volume 66 and test chamber 11 are materially reduced.

It will be seen from the construction of the liquid helium tank that even if the liquid helium level falls to a very low level, the bottom 72 and the skirt 73 in thermal contact with it will remain cold because a maximum quantity of any residual helium will always be in thermal contact with the skirt 73. The use of the elbow 81 also provides the maximum opportunity for preventing heat transfer through radiation inasmuch as radiation entering through extension 48 must be transmitted down through passage 85 and 86 where it strikes the blackened interior surface of the elbow. Thus it is apparent that there is provided the maximum protection against the passage of gas molecules toward the working area.

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Just ahead of the test chamber 11 there is placed a solid radiation shield 92 which is mounted to the internal wall of the interior of the inner vessel through a dual support ring 93 and radial support arms 94 (see also FIG. 4). The path of any gas molecules from the test chamber to the cryopump section of the ion pump is that which will provide the least obstructions for such gas flow. This path will of course be around the radiation shield 92 into area 66 and that portion which is not condensed on the copper skirt 73 is free to enter annular passage 88.

As indicated in FIG. 1 the liquid helium is introduced into the helium vessel 68 through line 24 which is controlled by valve 25. This liquid helium line is shown in detail in FIG. 2 along with the means by which it is inserted into the liquid helium vessel and the means by which the vessel may be pumped to reduce the pressure and hence the temperature of the liquid helium. As will be apparent from FIG. 2 a series of concentric tubes is employed. The first of these is the vacuum jacket tubing 105 which surrounds the liquid helium line 24 to form therebetween a fluid-tight space which is evacuated for insulation purposes. At the end of the vacuum jacketed helium delivery line which extends into the liquid helium vessel 68 the annular passage between jacket 105 and tube 24 is closed by a plug not shown. This annular passage of the vacuum jacketed helium delivery line is evacuated and sealed off.

A tubing 106 is sealed to the top 71 of the liquid helium vessel and extends upwardly beyond the point at which a T-joint is attached. This tubing 106 surrounds the vacuum jacket 105 and defines with it an annular passage 107 which is open to the horizontal arm of the T-joint and is thereby connected to line 28 which leads to the liquid helium vacuum pump (FIG. 1). This annular passage 107 is open to the interior of the liquid helium vessel 68 and hence is evacuated and exhibits a temperature gradient ranging from the temperature of liquid helium to the temperature of liquid nitrogen. In the central part of the tubing 106 there are located bellows 108 which furnish the necessary flexibility for readily joining the various tubing sections while still maintaining fluid-tight seals.

Extending from the top 50 of the inner vessel and welded to it is a second tubing 110 which terminates below the location of bellows 108 in tubing 106 so that a gas-tight weld might be formed to join tubings 106 and 110 and thereby provide the necessary support to suspend the liquid helium vessel 68 without effecting any additional connections between it and the remaining portion of the apparatus. There is defined between the lower section of tubing 106 and tubing 110 an annular gas passage 111 which, as will be seen in FIG. 2 is open to the interior 66 of the inner vessel through passage 76.

Extending from and attached to top portion 41 of the outer vessel is an outer tubing 113 which, by virtue of the fact that it is in communication with the guard vacuum 55, extends the guard vacuum up to that point where suitable sealing means close it at the juncture of the arm of the T-joint. It will be appreciated that in the drawing of FIG. 2 for the purpose of simplifying the detail, sealing rings and the like are not included. Suitable seal rings will of course be used in connection with the screw plugs 115 and 120. Wherever fluid-tight connections must be made suitable sealing means will be used. Associated with the T-joint, generally indicated at 117, is an outer protection sheath 118, means for screwing it onto the extending tube 119, and a horizontal arm 121 which, through suitable joining member 122, is attached to line 28 which is valve-controlled and leads to the liquid helium vacuum pump. With a suitable sealing means 123 it will be seen that any gaseous helium which boils off or is pumped off must be discharged through line 28. Associated with the helium vessel is a liquid level tube pressure tap 125 which communicates with pressure gauge 126 and then with line 128 leading to the liquid helium vacuum pump.

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In a typical experimental run the specimen is located within test space 11, the ends 45 and 12 bolted on and valve 23 is closed. Operation of the extreme high vacuum chamber then begins with the evacuation of the guard vacuum 55 (FIG. 2) by means of first, the roughing pump (not shown) and then the diffusion pump 16 (FIG. 1). At the same time the inner vessel is roughed down with the sorption roughing units 18 immersed in liquid nitrogen 19 (FIG. 1) and operation of the ion pump 20 is begun. Bakeout of the inner vessel is started by closing switch 33 to turn on heaters 60 (FIG. 2) and is continued for several hours by maintaining the temperature of the inner vessel walls at up to 750° F. At the completion of this bakeout valve 23 is opened and the inner shell is cooled to about 80° K. by passing liquid nitrogen through coil 62. The liquid nitrogen is introduced at that end of coil 62 which enters through extension 21, then flows around the horizontal portion of the inner vessel, then around the vertical portion and finally is discharged as gaseous nitrogen through coil 62 passing up through extension 22.

The liquid helium vessel in the cryopump section is precooled by first filling the vessel with liquid nitrogen and then displacing the liquid with helium gas. After cool down the insulated liquid helium vacuum transfer line 105 (FIG. 2) is inserted and liquid helium is transferred into the vessel from a storage dewar or other suitable source. During the filling of the liquid helium vessel 68, valve 29 (FIG. 1) is open and it remains opened after filling is completed and valves 25 and 27 are closed. By the use of the mechanical vacuum pump it is possible to maintain the pressure over the liquid helium at a reduced level and the liquid helium temperature at about 2.5° K., thus materially improving the efficiency of the cryopump system.

At the completion of the test, the liquid helium vessel is brought to atmospheric pressure with gaseous helium and the liquid helium is displaced. The inner vessel and the helium vessel are warmed to room temperature with the bakeout heaters. The vacuum within the inner vessel and then the guard vacuum are released by admitting dry nitrogen gas. The ends of the outer and inner vessels may then be removed for access to the work chamber.

With the cryopump operating at 2.5° K., all gases except helium are cryopumped—that is they are condensed on the cryopump surface. Because the cryopump surface is immediately adjacent to the test chamber 11 and separated from it by only the high-conductance radiation shield 92 (FIG. 2) high pumping speeds at the test chamber area are realized. In addition, water vapor and less volatile constituents are cryopumped on the liquid nitrogen-cooled walls of the test chamber, resulting in extremely high speeds for these constituents. Typical pumping speeds attainable are 43,000 liters/second for water vapor, 1,400 liters/second for nitrogen, oxygen and carbon monoxide, and 5,200 liters/second for hydrogen. Helium gas is pumped by the ion pump at a speed of approximately 10 liters/second.

Pressure in the test chamber can be reduced to 1×10^{-13} torr or less. This extremely low pressure is made possible by the novel arrangement within the extreme high vacuum system. The guard vacuum reduces leakage into the inner vessel through feedthrough, seals and welds to a negligible level. Bakeout and cooling of the walls of the inner vessel reduce outgassing to an extremely low level. Finally, the liquid helium cryopump not only provides a high pumping speed immediately adjacent to the test chamber but also isolates the test chamber from the warm end of the system where wall outgassing may be appreciable. Since helium is not outgassed by metals and is not present to any measurable degree in the inner vessel any contribution of helium to the ultimate pressure is minimal when the

liquid helium vessel is constructed to eliminate leak-
ages.

It will thus be seen that the objects set forth above, among those made apparent from the preceding description, are efficiently attained and, since certain changes may be made in the above construction without departing from the scope of the invention, it is intended that all matter contained in the above description or shown in the accompanying drawings shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all of the generic and specific features of the invention herein described, and all statements of the scope of the invention which, as a matter of language, might be said to fall therebetween.

I claim:

1. A high-vacuum chamber, comprising in combination

- (a) a vacuum-tight horizontally oriented cylindrical inner vessel;
- (b) means for circulating a cryogenic fluid in heat transfer relationship with the outer wall of said inner vessel;
- (c) an experimental vacuum chamber located at one end of said inner vessel adapted to be accessible for insertion and withdrawal of an experimental body;
- (d) conduit means at the other end of said inner vessel and communicating with vacuum pump means;
- (e) cryopumping surface means located within said inner vessel between said experimental vacuum chamber and said conduit means;
- (f) means separate from (b) for bringing a cryogenic fluid in heat exchange relationship with said cryopumping surface means thereby to maintain them at a cryopumping temperature;
- (g) radiation shield means within said inner vessel between said cryopumping surface means and said experimental vacuum chamber; and
- (h) an outer vacuum-tight horizontally oriented cylindrical vessel immediately surrounding said inner vessel and defining an evacuable guard volume around said inner vessel.

2. A high-vacuum chamber in accordance with claim 1 wherein said means for bringing a cryogenic fluid in heat exchange relationship with said cryopumping surface means comprises a cryogenic liquid storage vessel having an inverted dish shaped bottom section forming a portion of said cryopumping surface means, whereby said cryopumping surface means will be maintained at cryopumping temperature irrespective of the level of said cryogenic fluid in said vessel.

3. A high-vacuum chamber in accordance with claim 1 wherein said means for bringing a cryogenic fluid in heat exchange relationship with said cryopumping surface means comprises coils adapted to circulate said cryogenic liquid.

4. A high-vacuum chamber in accordance with claim 1 further characterized by having heater means associated with said inner vessel.

5. A high-vacuum chamber in accordance with claim 1 further characterized by having a fibrous insulating material occupying at least a part of said guard volume.

6. A high-vacuum chamber, comprising in combination

- (a) a vacuum-tight inner vessel comprising a horizontal cylindrical section and a vertical cylindrical section integral with said horizontal section;
 - (b) means for circulating a cryogenic fluid in heat transfer relationship with the outer wall of said inner vessel;
 - (c) an experimental vacuum chamber located at one end of said horizontal section adapted to be accessible for insertion and withdrawal of an experimental body;
 - (d) first conduit means at the other end of said horizontal section and adapted to provide communication between said inner vessel and vacuum pumping means;
 - (e) cryopumping means positioned between said experimental vacuum chamber and said conduit means and comprising
 - (1) a liquid helium vessel extending into the vertical section of said inner vessel and having an inwardly dish bottom, and
 - (2) a metal skirt in thermal contact with the outer wall of said vessel, depending from the bottom thereof and defining with said bottom of said liquid helium vessel an enclosure having cryopumping surfaces maintained at near liquid helium temperatures;
 - (f) radiation shield means within said horizontal section positioned between said cryopumping means and said experimental chamber;
 - (g) second conduit means communicating with said first conduit means and extending up into and open within said enclosure of said cryopumping means whereby gas originating from walls of said pumping means and of said conduit means in flowing to said experimental chamber must follow a path which causes it to contact said cryopumping surfaces thereby minimizing the possibility of its return into said experimental chamber;
 - (h) heating means associated with said inner vessel;
 - (i) an outer vacuum-tight vessel comprising a horizontal cylindrical section and a vertical cylindrical section and surrounding said inner vessel and defining an evacuable guard volume around said inner vessel; and
 - (j) concentric tubular extensions of said liquid helium vessel and of said vertical sections of said inner and outer vessels, said tubular extensions being adapted to extend said guard volume, to receive a vacuum-jacketed liquid helium delivery line and to communicate with a liquid helium vacuum pump.
7. A high-vacuum chamber in accordance with claim 6 further characterized in that the inner wall of said second conduit means is blackened and that a gas reflecting ring is affixed to its open end within said enclosure of said cryopumping means.
8. A high-vacuum chamber in accordance with claim 6 further characterized by having a fibrous insulating material occupying at least a part of said guard volume.

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