AECD - 1814

UNITED STATES ATOMIC ENERGY COMMISSION

VACUUM TESTING AT SAM LABORATORIES

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This document consists of 31 + 1 pages
Date Declassified: March 22, 1948

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Technical Information Division, Oak Ridge Directed Operations
AEC, Oak Ridge, Tenn., 10-14-48--1500-18453

Printed in U.S.A.
PRICE 15 CENTS

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I GENERAL THEORY OF LEAK TESTING

A. Definitions

In work which deals with fluids, it is often necessary to confine certain fluids to certain regions of space and to exclude them more or less completely from other regions. This requires the use of some sort of wall. A wall may be composed of solid matter, or in rare cases even of liquid or gas; two regions may be insulated from each other by an intermediate region filled with flowing liquid or gas, or with a very viscous liquid at rest, or with a liquid confined under surface tension constraints. All our leak testing work has dealt with solid walls.

The rate, under given conditions, at which a given fluid passes through a wall or system of walls is an inverse measure of the tightness of the wall or system. Walls which are not tight or which have selective tightness are variously called porous, leaky, or semipermeable. Porosity denotes lack of tightness over a considerable area. Leak is the term used when the lack of tightness is sharply localized. A leak is usually visualized as a hole or a crack.

Leak testing, i.e., the determination of tightness, involves, in general, the application of a suitable fluid to one side of the wall and the observation of its rate of passage through the wall to the other side. This is called the leak rate. From this one might calculate the leak rate for some fluid under other conditions. Usually, however, one chooses a test fluid and test conditions for which the leak rate will certainly be worse than it will be for the operating fluid and operating conditions.

Because of its low viscosity, a gas is generally used as the test fluid. It is known as the probe gas.

The unit commonly used to measure the leak rates is the micron cubic foot per hour (MFH), a micron being a unit of pressure defined as that pressure which is capable of supporting a column of mercury one micron (10^{-3} mm) in height. The micron cubic foot per hour is defined to be an hourly flow of that quantity of gas which at a pressure of one micron and at room temperature occupies a volume of one cubic foot. It will be seen that the leak rate in this unit is proportional to the number of molecules per hour or to the number of moles per hour.

B. Pressure Testing and Vacuum Testing

In a sense, all leak testing is pressure testing, since the only efficient way to make a fluid pass through a wall is to apply different pressures to the two sides. If, however, one of these two pressures is less than atmospheric pressure, the method is commonly known as vacuum testing method, otherwise, as a pressure testing method. The two methods have some features in common. Each requires that the wall to be tested be made a part of a system of walls which forms an enclosure. In one case this enclosure has its pressure raised above atmospheric and in the other case reduced below atmospheric. Each requires some means for observing flow, either directly or indirectly.

C. Methods of Measuring Flow

The direct method of measuring flow would require that a flowmeter be placed in the pressure line in one case or in the vacuum exhaust line in the other case. In both cases, the steady-state flow would be taken as the leak rate, but the sensitivity would be far higher with the flowmeter in the vacuum line, since for a given mass flow rate, the flow velocity is inversely proportional to the pressure at the place where it is measured.

In the indirect method for observing flow, we observe either the rate at which gas disappears from the high pressure side or the rate at which it appears on the low pressure side. That is, we observe the rate of change of the pV product on one side or the other, and since the temperature is constant, this is proportional to the rate of change of mass or leak rate. Changes in pV can be observed in either of two ways: we can observe the rate of change of p at constant known V or the rate of V at constant known p.

The first of these (constant V) is known as the leak-up method when applied to the vacuum sic and the leak-down method when applied to the pressure side. In both cases the sensitivity will obviously be inversely proportional to V. In general, the sensitivity will be far higher for the vacuum method, since the lower the pressure the smaller the detectable changes in pressure. The type of vacuum pressure gauge selected will depend on the vacuum which can be attained. It will be noted that in neither case is any gas to be introduced in the pressure side or withdrawn from the vacuum side during the test.

In the constant p method the volume changes may be observed in various ways. The case where V is on the high pressure side is exemplified by the well-known scheme for measuring flow rates through a needle valve. Here a drop of mercury is drawn through a capillary tube at a rate proportional to dV/dt. A well-known case where dV/dt is observed on the low pressure side is that of bubble-testing. Here the pressure inside a vessel is raised (but not more than the vessel can safely withstand) and the vessel is submerged in water or painted with a soap film. Bubbles will emerge at points where leakage exists. The rate of leakage at each of these points may be measured by collecting the bubbles in an inverted graduate filled initially with water in order to determine their volume, dividing by the elapsed time in appropriate units, and multiplying by the atmospheric pressure. For objects which can be immersed, this is an excellent method, for with a high enough pressure and a keen observer it not only shown up small leaks, but shows exactly where they are, provided the leak point is not concealed. It has the added advantage of requiring very little equipment.

Another common method is the pump and gauge method. If a vacuum pump which is known to be capable of pumping a tight object to a low pressure refuses to pump some test object to a comparably low pressure, we conclude that the test object leaks. Now the speed S of a vacuum pump is defined as the volume rate of flow into the pump at the inlet pressure p, and, in general, is a function of p. The pump may be thought of as a vessel whose volume keeps expanding at a rate $dV/dt = S$. If we know the gauge reading p, we can look up S from the S-p curve for the pump. The leakage is then p dV/dt or pS. We see that this also is in reality a constant p method, with the difference that dV/dt is obtained somewhat indirectly.

D. The Effect of Outgassing

When gas is pumped out of a closed system of walls, with a resultant lowering of pressure inside the system, it is found that the reduction of pressure is not as great as that predicted by Boyle's Law. It appears as though gas were flowing in from some source, even when it is known that no leaks exist. The effect is known as outgassing and is more marked at low pressures, the apparent rate of inflow increasing as the pressure is lowered. The rate may be obtained by shutting off the pump and observing the rate of pressure rise or leak-up rate.

The outgassing rate depends on the material of which the walls are made, the kind and amount of dirt adhering to them, and the kind of gas or vapor with which they have been in contact. Thus, it appears that the source of gas is the walls themselves. Some of the possible processes by which gas is evolved from the walls are: evaporation, sublimation, decomposition, and the inverse of the processes of adsorption, absorption, occlusion, and solution.

The rate of evolution of gas increases steeply with rising temperature. Early in the outgassing process the preponderance of the gas given off is normally water. In general, metal surfaces outgas worse than glass surfaces.

In all of the vacuum methods mentioned in Section C, the result is a determination merely of the apparent or virtual leakage. Due to outgassing, a virtual leak may be observed even though no real leak (inleak) exist. If no means is at hand for distinguishing between inleakage and outgassing, one cannot safely conclude that the inleak rate is less than the measured virtual leak rate. That is, the best our measurements are able to do is to establish an upper limit for the inleakage, viz., the virtual leak rate. This upper limit we shall call the leak limit.

E. Use of Gas Analyzers

If as probe gas we employ a gas which can be readily distinguished from the gases given off by the walls of the vessel, we have the possibility of measuring the actual inleakage.

Thus an extension of the leak-up method (constant-V vacuum method) would consist in flooding the leaks in question with probe gas for a measured length of time Δt, taking the gases accumulated in V, and analyzing them to determine the molar fraction f of the total gas which is probe gas. If p is the pressure in V after the interval Δt, and the units are microns, cubic feet, and hours, the inleakage of probe gas is:

$$\frac{f\ p\ V}{\Delta t} \quad \text{micron ft}^3/\text{hr}$$

To conform with the optimum operating conditions of the analyzer one may take either all or part of the accumulated gas and may either compress or expand it or leave its pressure unchanged, all without affecting the result. Obviously, the amount of gas in V at the beginning of Δt is immaterial provided the resulting value of f is not below the threshold of the analyzer. The method will be at its most sensitive when the vessel is pumped down to the lowest attainable pressure before starting the test.

A gas analyzer can be applied to the constant-p "pump and gauge" method as follows. When the vessel has been pumped down to constant pressure, the rate at which gas flows through the pump line is then equal to the leak rate plus the outgassing rate. If the pump pumps all gases with the same speed, it follows that the ratio of the partial pressure of probe gas to the total pressure is equal to the ratio of the flow of probe gas (leak rate) to the total flow. If, then, we divert part of the flow into an analyzer and determine the molar fraction f of probe gas present, we can compute the inleak f p S, where p is the pressure at a gauge placed at some point of the pumping line past which all gas from the vessel must flow, and S is the pumping speed at this point for the pressure p. If the pressure is less than about 1 mm, it is unnecessary actually to divert part of the flow into the analyzer; the analyzer may be placed at the end of a short closed line branching off from the main line. Diffusion will be rapid enough so that the gas composition in the analyzer will be the same as that in the main line. A variation of this method would consist in measuring the virtual leakage as V dp/dt (leak-up method) instead of p S. This is a more useful method, since a pump does not hold its calibration very well.

Perhaps the most easily applied method of determining inleakage is the standard leak method. Here an artificial leak of known size is inserted in the exhaust line and is used to calibrate the gas analyzer in terms of inleakage. In almost all cases, the indications of the analyzer are proportional to the size of leak, so that an unknown leak may be calculated by a simple proportion, provided that the conditions under which the probe gas is applied are the same in both cases.

If the analyzer is of the type which gives a continuous indication of the composition of the gas, we have an arrangement which is well adapted to the localization of leaks. One moves a fine jet of probe gas over the areas to be tested, meanwhile observing the indications of the gas analyzer. When the jet comes into the immediate neighborhood of a leak, the analyzer will, after a slight delay, indicate the presence of probe gas in the system. The duration of this delay depends on a number of factors to be discussed later. The sensitivity with which leaks can be localized by probing with a jet is called the dynamic sensitivity.

There are a number of gas analyzers which can be adapted to vacuum leak testing. Other things being equal, the lower the pressure at which the analyzer will work, the more suitable it is. The reason for this is that, since all analyzers ultimately are indicators of the partial pressure of probe gas, the lower the partial pressure at which the analyzer begins to indicate, the shorter the time required to build up this pressure in a given volume for a given size of leak. This limitation can be mitigated by compressing the gas into as small a volume as possible after it has been pumped out of the test vessel. For very small leaks, however, it may not be possible to make this volume small enough, so that to attain the required dynamic sensitivity, the only alternative is to go to an analyzer which can operate at a lower pressure.

Without much doubt, the gas analyzer which is operable at the lowest pressures is the mass spectrometer; this is therefore the best instrument for dynamic leak testing.

F. Use of the Mass Spectrometer

The probe gas used in conjunction with the mass spectrometer for leak testing is helium. The principal advantage of helium is its inertness. It neither combines chemically nor adsorbs to any appreciable extent. Because of this fact, it is not present in the gases evolved from the walls of the system, so that there is no "background" of probe gas to allow for. Besides, it is present in air to only about 1 part in 250,000. Next, its m/e value is low, so that it is easily resolved in a relatively compact mass spectrometer. Besides, it is nonflammable, nonpoisonous, and has about the same viscosity as air, so that it flows through a leak at about the same rate. The use of helium presents two slight disadvantages. Its cross section for ionization by electrons is low, and its diffusion coefficient is roughly a factor of 3 higher than that of air. Due to this latter fact, the percentage in a system which is being pumped at low pressure is somewhat lower than would be the case for a heavier probe gas.

Figure 1 is a schematic diagram of a complete leak testing system using a mass spectrometer. This is the system which has been used in this laboratory. It is to be noted that there are two complete diffusion pumping systems. The one associated with the spectrometer tube is for the purpose of maintaining a pressure of about 10^{-4} mm in the tube regardless of the pressure in the test vessel. Its diffusion pump should have a cutoff pressure of less than 10^{-5} mm. In the main pumping system we have a diffusion pump of the "booster" type with high speed at moderately high pressures and a cutoff pressure of about 10^{-4} mm. G_1 is a Pirani gauge which indicates the pressure in the exhaust line in the range 1 to 500 microns. G_2 is an ion gauge which indicates tube pressure in the range 10^{-5} to 10^{-3} mm. G_3 is a thermocouple gauge. It is useful in the early stages of getting a vacuum in the tube.

The various traps are for the purpose of protecting certain parts from vapors and to aid the pumps in producing a high vacuum. T_1 protects the test vessel from the oil vapor present in the mechanical pump and protects the mechanical pump from volatile matter which might be pumped out of the test vessel and which might condense in the mechanical pump, impairing its efficiency. T_2 protects the test vessel from oil vapors present in the diffusion pump. T_3 removes condensable matter which would otherwise go into the spectrometer tube, reducing the sensitivity of the test and possibly contaminating the tube. T_4 protects the tube from oil vapor from the diffusion pump.

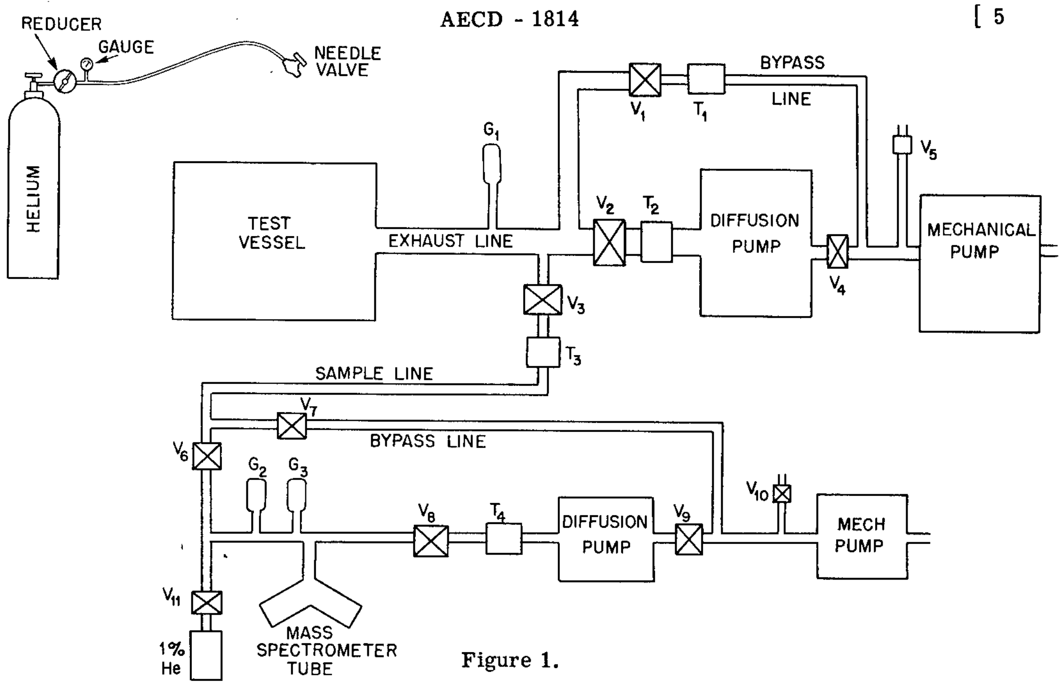


Figure 1.

V_1 is the main pumping by-pass valve. It remains open until the pressure in the test vessel has been reduced to 300 or 400 microns. During this time the high-vacuum valve, V_2, and the fore-vacuum valve, V_4, remain closed to protect the oil in the diffusion pump from the oxidation and cracking which occur when hot oil comes in contact with air. When the diffusion pumping is started, it is important that V_1 be closed before either V_2 or V_4 is opened, and that V_4 be opened before V_2, since it sometimes happens that the pressure in the closed-off diffusion pump has built up, resulting in a sudden burst of oil-laden gas when the valve is opened, which must not be allowed to get into the test vessel. V_5 is a relief valve for breaking the vacuum in the mechanical pump when shutting down the apparatus and after closing V_1 and V_4. V_3 is used mainly as a shutoff valve to protect the sample line trap, T_3, from vapor during the rough pumping of the test vessel. It is used also as a throttle valve when testing at pressures above 100 microns. At these high pressures, if V_3 were opened and the sample were bled in through V_6, helium would diffuse too slowly through the long column of stagnant gas in the sample line. If, however, the sample is bled in by just cracking V_3 and keeping V_5 open, the pressure in the sample line will be very low and the helium will diffuse through it almost instantaneously. V_3 is placed as close as practicable to the current of gas in the exhaust line, so that the helium arrives at it from the test vessel almost entirely by convection. V_7 is the by-pass valve, used for pumping out the tube after it has been at atmospheric pressure, as for replacing a burned out filament. V_7 is also used for rough pumping of the sample line preparatory to using V_3 to bleed in the sample. The high-vacuum valve, V_8, and the fore-vacuum valve, V_9, are used to keep a vacuum in the diffusion pump when the by-pass is in use or when it is necessary to let atmospheric pressure into the tube. Thus the pump can be kept hot while the tube is being repaired or replaced with a spare, and considerable cooling and heating time is thereby saved. The relief valve, V_{10}, performs a function similar to that of V_5.

The traps are cooled by immersion in vacuum bottles which contain either liquid nitrogen or a mixture of dry ice and some liquid, such as trichlorethylene or acetone. Liquid nitrogen is the more convenient of the two, but is expensive and is absolutely necessary only in T_4 and then only when the diffusion pump is one using mercury. Its use in T_3 often results in considerably improved sensitivity, since the outgassed matter is largely water vapor which is effectively removed at the temperature of liquid nitrogen.

The mass spectrometer may be thought of as an ion gauge which measures the partial pressure of helium in the presence of other partial pressures. The essential difference lies in the interposition of a magnetic analyzer between ion source and collector. The effect of this analyzer is that only helium ions are collected. The number collected is proportional to the number formed in the source, which, in turn, is proportional to the partial pressure of helium in the source.

Besides the tube and the vacuum system, the spectrometer has several electrical parts. There is a power supply which supplies stabilized voltages to the various electrodes and which contains means for automatic regulation of the space current of electrons from the filament. Regulation is accomplished by means of an amplifier which controls the filament temperature. The ion accelerating voltage is variable by means of a manual control, so that the mass spectrum may be observed by causing the dispersed ion beam to move across the collector slit. The collected ion current is observed by means of a DC amplifier with high current gain. The first tube of this amplifier is located near the collector in order to avoid the difficulties which would otherwise result from the fact that the input resistance is of the order of 10^{11} ohms. Currents as small as 10^{-14} ampere are detectable, and are observed on an ordinary milliammeter at the output of the amplifier.

Closely associated with the spectrometer is the ion gauge. This indicates substantially the total pressure in the ion source of the spectrometer, and when the pressure rises high enough to endanger the filaments, a relay in the control circuit of the ion gauge automatically cuts off the current to the filaments. An automatic emission control similar to that of the spectrometer is part of this circuit.

Calibration of the spectrometer is accomplished by use of a standard mixture of helium and air, say 1 per cent helium by pressure, contained in a can at a pressure anywhere in the range from one atmosphere to half an atmosphere. The sensitivity of the spectrometer is specified as the output reading when the mixture is bled in at a rate which causes a pressure rise of say 10^{-4} mm in the ion gauge. If it is desired to measure the percentage of helium in an unknown sample, the standard mixture is bled in to the same pressure that prevailed when the sample was in the tube. The percentages will then be in the same ratio as the meter readings.

II DEVELOPMENT OF THE PUMPING UNIT

A. General Problem

Most of the equipment tested at SAM was sufficiently small in volume so that a 1-inch pumping line gave adequate pumping speed. Since 1-inch metal hose was about the largest which could be handled conveniently, it was decided to standardize on this size for most of the pumping equipment. Six pumping units were built using 1-inch piping and other parts in keeping. It was possible to build these units small and mobile with a maximum of convenience for operation as well as maintenance, and with sufficient flexibility to permit their use under a variety of conditions in crowded laboratories.

There were, however, a few systems at SAM which required a pumping speed as high as that corresponding to a 4-inch line. Two units of this size were constructed, part of the fabrication being done by an outside shop at the same time that similar units were built for the Vacuum Engineering School which was at that time being set up at SAM. Less mobility and flexibility were required of these units, since their use was limited and rather specialized.

B. The 1-Inch Pumping Unit, Preliminary Design

The first 1-inch units had to be built in great haste and with whatever parts which were immediately available. A great many lessons were learned from them which proved helpful in the later design.

Housing—The framework in which the unit was housed was made of welded angle iron and mounted on rubber-tired wheels with brakes. The space provided for the parts was far more than necessary, the result being an unwieldy piece of apparatus.

Method of Assembly — All the metal parts were soldered into the assembly, certain demountable parts being soft-soldered and the rest silver-soldered. It was reasoned that the fewer demountable joints present, the fewer would be the leakage troubles. This soon proved to have been a serious mistake. The assembly could be tested only as a whole, and it was frequently necessary to remove the entire piping assembly including the diffusion pump (whose oil had to be drained), in order to repair a leak.

Pumps — A nominally 6 CFM International mechanical pump was used for backing and for rough pumping through the by-pass. This was connected into the system by a short length of rubber tubing which was attacked by the oil and frequently started to leak. The diffusion pump was a Distillation Products MB-15 with rated speed of 15 liters per second and ultimate vacuum of 10^{-4} mm.

Valves — Because Kerotest diaphragm-sealed valves could not be procured in time, it was decided to use Nordstrom greased-plug valves. These were of cast steel and much difficulty was experienced in soldering them into the system in a leak-tight manner. Their only advantage lay in the fact that they could be opened or closed with a 90° motion of the handle. On the other hand, they were sometimes very hard to operate, they needed frequent regreasing in order to keep them tight, the grease accumulated in the port, eventually blocking it, and every time the valve was operated a slug of air was released from the grease, causing a momentary spoiling of the vacuum.

Traps — It was desired to make the traps of glass in order to avoid waste of liquid nitrogen as well as to provide visibility. They were of the coaxial tube type, with the lower parts of type 705 glass so that they could be sealed onto Kovar rings, which in turn were soft-soldered into the system. Whenever it became necessary to remove the outer glass tube for cleaning, the entire piping assembly had to be removed and inverted in order to make a satisfactory soft-solder joint. These traps were rather fragile and sometimes broke from thermal shock, and since the parts were not interchangeable, serious delays resulted.

Gauges — Two gauges were used, a mercury manometer and a Distillation Products Pirani gauge. These were connected into the system with short lengths of rubber tubing.

Cooling — The cooling water for the diffusion pump was obtained from the nearest cold water tap and returned to the nearest drain by way of rubber or Saran lines. There was a great deal of trouble from the leaking of these lines and from the accidental turning off of the water valve. Occasionally this had serious results, such as the distilling of mercury from the diffusion pump over into the spectrometer tube.

C. The 1-Inch Pumping Unit, Final Design

Housing — Figures 2A and 2B will give a good idea of the welded angle iron framework. Strapped to the sloping front are the valves which support the piping. On the upper deck is mounted the mechanical pump and on the lower deck the refrigerator, water reservoir, water pump, vacuum bottle supports, and diffusion pump. Although this unit contains more parts than the first design, it is smaller and better adapted to use in a crowded laboratory.

Method of Assembly — An effort was made to standardize the assembly and to make many of the subassemblies interchangeable and demountable. Rubber was freely used in making the vacuum joints, most of which were of the flat gasket flange type. One tongue-and-groove flange, several Dresser couplings, and one flare coupling were used. In the freon and water circulating systems, all the demountable joints were of the flare type. All electrical connections to demountable parts were made with cable connectors. All this greatly facilitated the work of maintenance as well as that of initial construction, and reduced the out-of-service time to one or two per cent.

All vacuum solder joints were either of "Easyflo" or "Silfos" solder. It was easy to make such joints tight, and they stayed tight in contrast to soft-soldered joints. All joints except copper-to-copper ones were made with Easyflo and Handyflux in subassemblies which could be turned on the bench to take advantage of gravity, and which, after soldering, could be cleaned in an acid bath. The

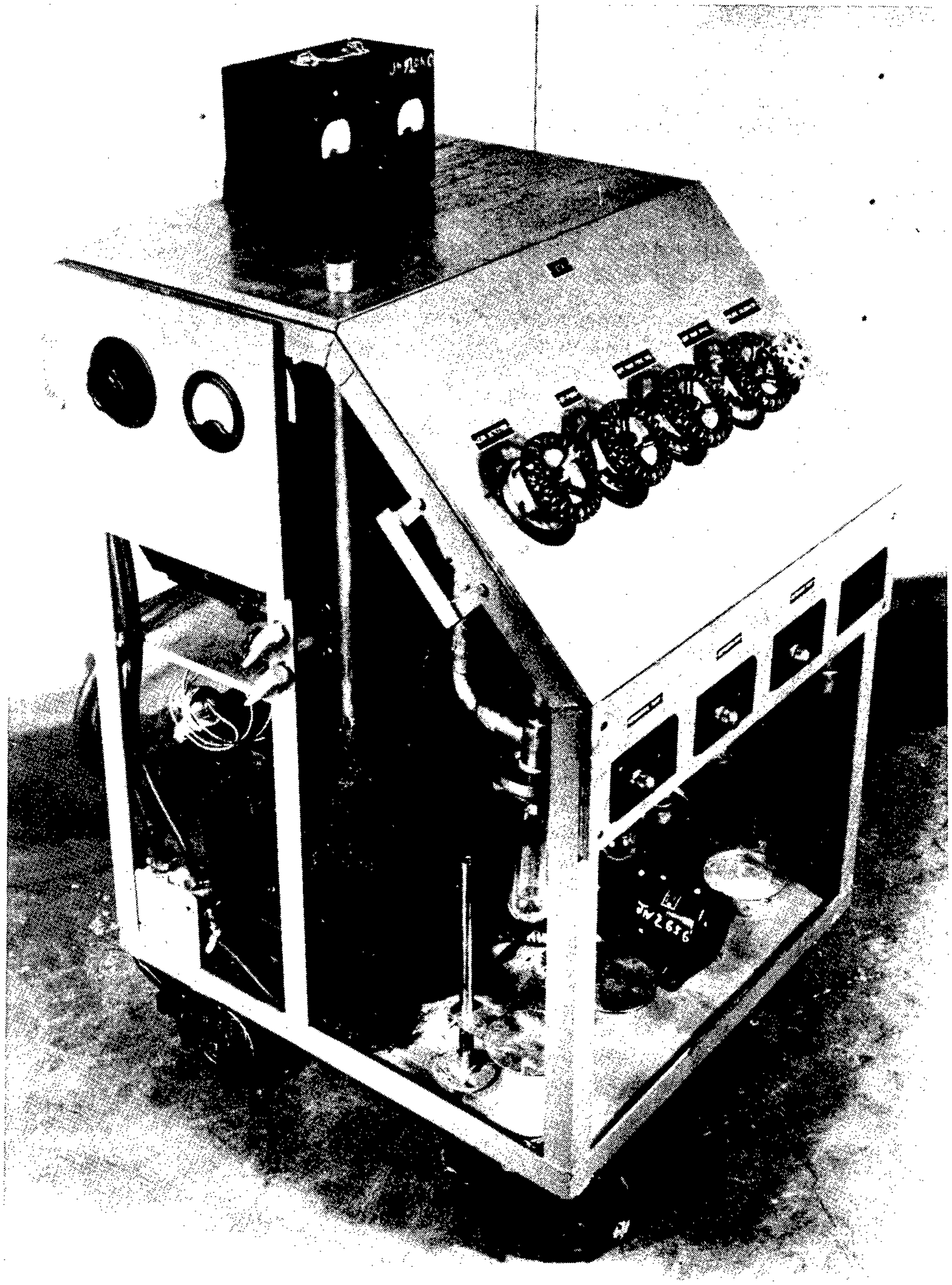


Figure 2A.

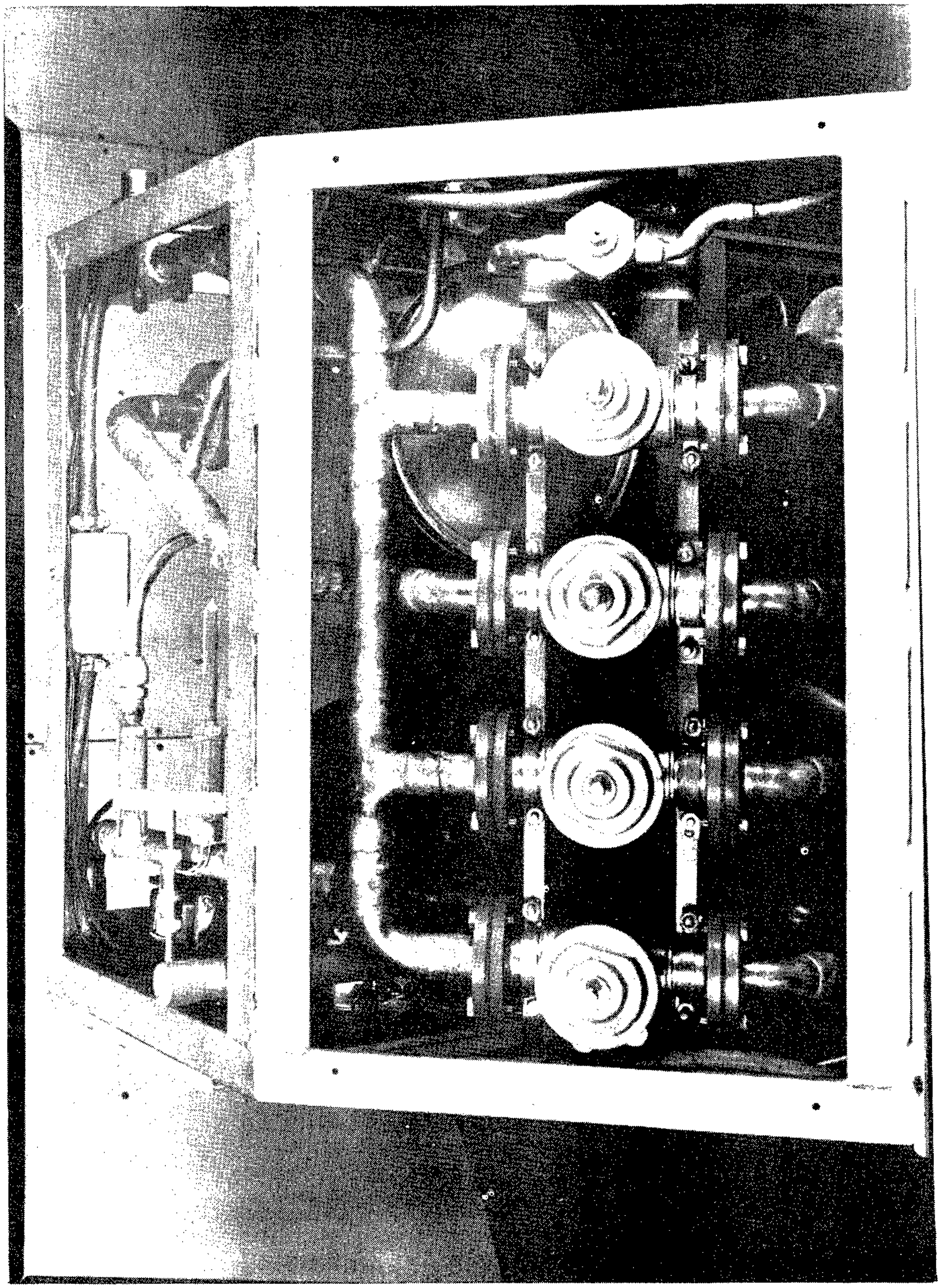


Figure 2B.

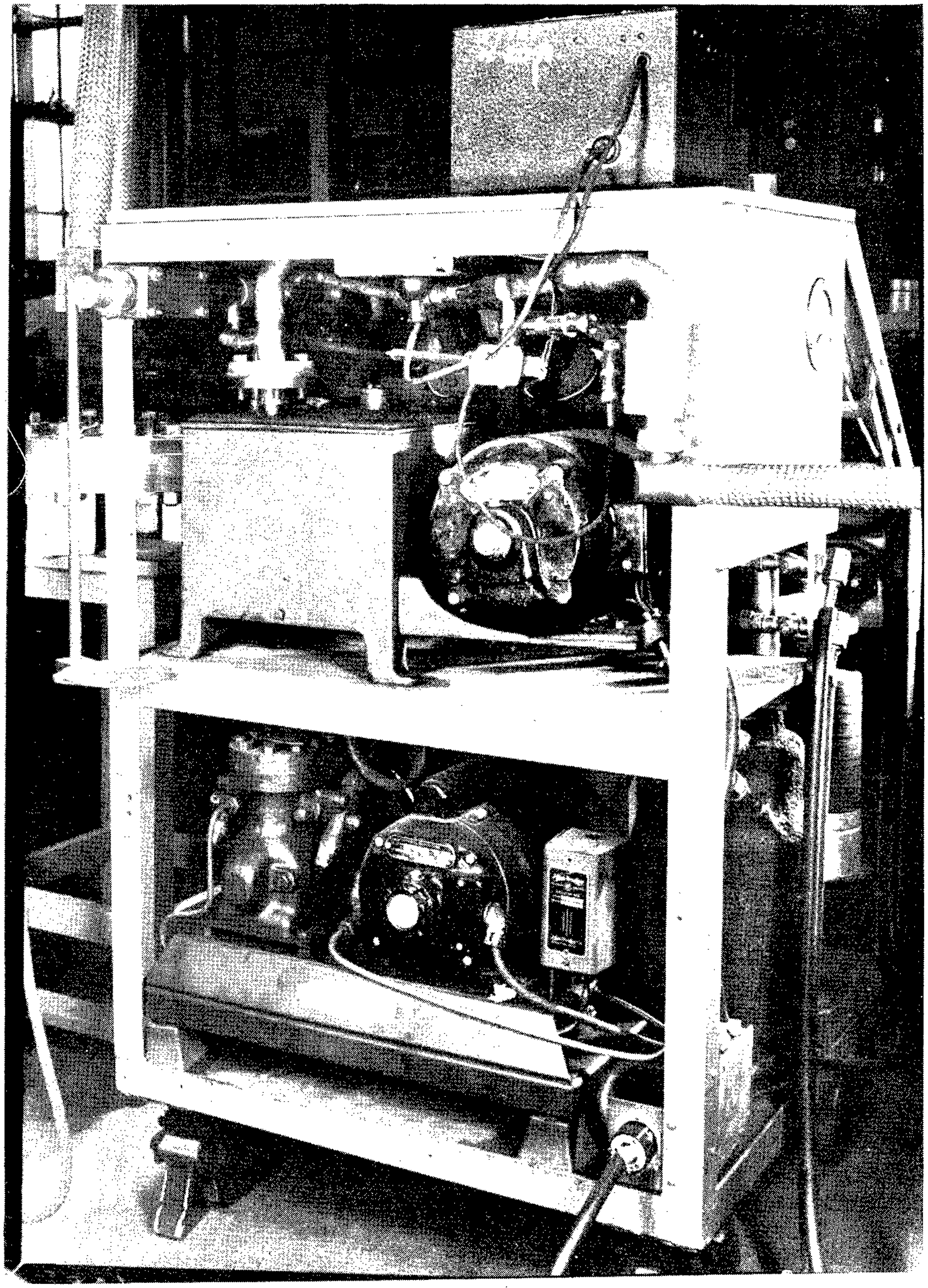


Figure 3.

only joints which were done at final assembly were copper-to-copper. These were made with Silfos without flux and with an inert or reducing gas flowing through the pipes. Either nitrogen or 95 per cent nitrogen 5 per cent hydrogen is suitable. Pure hydrogen may be used if it is allowed to burn at the point where it leaves the system and if it is purged out with an inert gas after the pipes have cooled.

Pumps—As in the preliminary design, the mechanical pump is a 6 cfm International and the diffusion pump an MB-15. These are equipped with flanges and electrical connectors so as to be readily demountable. The flange on the diffusion pump inlet is large enough so that the jet assembly can be pulled out for cleaning. The fluid used initially in this pump was butyl sebacate, but this decomposed and left a heavy deposit of carbon. It was replaced with Aroclor 1254, purchased from the Monsanto Chemical Corporation. This fluid stands up well even under abuse and appears to pump just as efficiently as the less stable butyl sebacate.

Valves—The valves are Kerotest with a number of modifications. For vacuum tightness the Bakelite seat was replaced by a neoprene disc bonded directly to the stem. Rubber diaphragms are now used in place of the metal diaphragms which required that an excessive torque be applied to seal the bonnet and which broke frequently. The bonnet was suitably modified as shown in Figure 4. The 1-inch valves were equipped with flanges as illustrated. An important feature is the pump-out channels without which it would take a long time to get a good vacuum because of air trapped in pockets and leaking out through the threads.

Cold traps—Use was made of standard Pyrex flanged pipe in the demountable glass trap. This pipe is very heavy and the breakage was almost negligible. The glass parts could be removed by means of a wrench in a minute or two, so that cleaning was an easy matter. Details of the construction are shown in Figure 5.

Vacuum Couplings—One of the aims was to provide enough flexibility in the set-up arrangements so that the apparatus could be used in any laboratory situation. The use of metal hose as the pumping line was a means to this end. A further means was the use of a double pumping connection consisting of two Dresser couplings with their axes horizontal and at right angles to each other. By plugging the right-angle end of a metal hose into such a coupling, it was possible to have the hose lead off in any direction in either of two planes, and so prolong hose life by obviating sharp bends. Into the other coupling there was inserted either a plug, a standard leak, or a portable McLeod gauge for checking the Pirani gauge calibration. The sample line between the pumping unit and the spectrometer unit was a short horizontal metal hose with right-angle ends plugged into vertical Dresser couplings. By this means it was possible to set up the units in various relative positions.

Gauges—For pressures from 160 mm down to 1 mm there was a mercury manometer which was coupled into the pumping line by means of a Dresser coupling. For pressures from 500 microns down to 1 micron there was a Distillation Products Pirani gauge, coupled into the pumping line by means of a flare fitting, with a wax joint between the glass tube and the fitting. The calibration of this Pirani gauge was checked frequently by means of a portable plug-in McLeod gauge and was found to change rather rapidly. This aging is thought to be due to the blackening of the filaments.

Measurement of Volume—Sometimes it is desirable to be able to measure the volume of the test vessel. For this purpose a can of .013 cubic foot volume was permanently connected into the pumping line by way of a small valve. Also connected to the can was a small valve opening to atmosphere. With a vacuum in the test vessel and the pump valves closed, .013 cu ft. of air at one atmosphere was released into the system and the final pressure was read on the manometer. The unknown volume was a function of this pressure and could be read off from a chart. This volume calibrator also served as a channel for the admission of a dry inert gas into the test vessel after the test was finished, a requirement often met with. It was more often used for breaking the vacuum with atmospheric air.

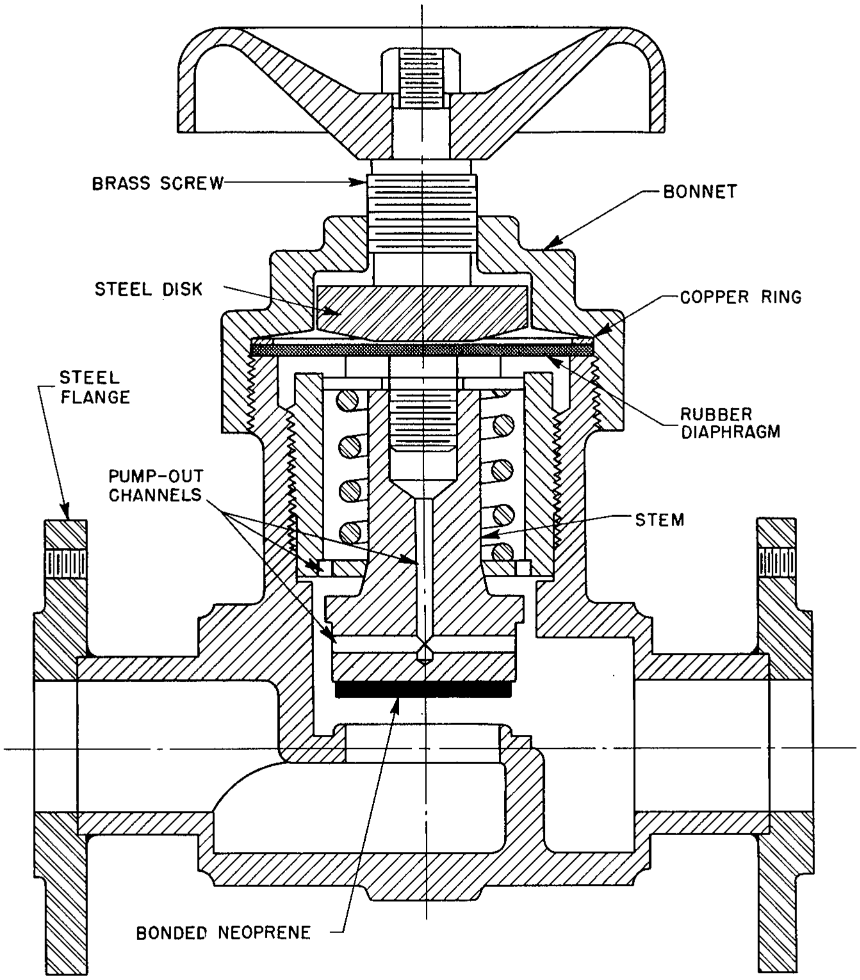


Figure 4.

Cooling System—In order to make setting-up easier and quicker, and to avoid serious floods, a closed cooling system was built into the pumping unit. This consisted of a water circulating system and a freon circulating system (refrigeration cycle). The water was circulated by a centrifugal pump from a reservoir through the water jackets of the diffusion pumps and back to the reservoir. The freon "12" was circulated by a compressor from the receiver, through the expansion valve, through a copper evaporator coil inside the water reservoir, through the compressor, through the air-cooled condenser, and back into the receiver. The freon circulates intermittently, the compressor being controlled by a thermal switch which is soldered to the outlet line from the water reservoir and adjusted so that the water in the reservoir is kept at about 15°C.

Safety Features—To avoid serious breakdowns due to inattention on the part of the operator or due to failure of some part of the equipment, a number of safely devices were used.

In case of failure of the cooling system, a thermal switch soldered to the cooling coil of the diffusion pump heats up and breaks the heater circuit.

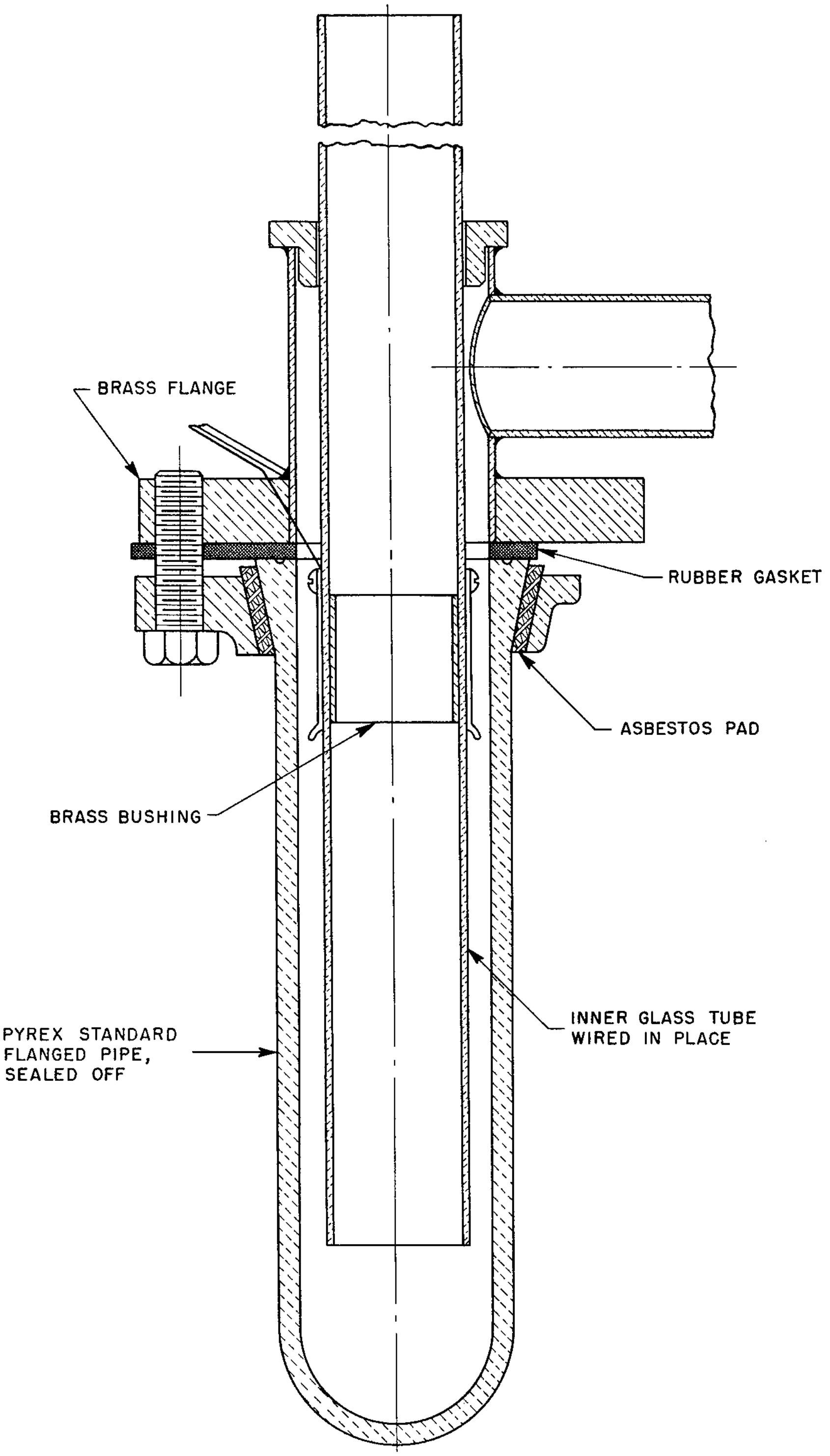

BRASS FLANGE
RUBBER GASKET
ASBESTOS PAD
BRASS BUSHING
INNER GLASS TUBE WIRED IN PLACE
PYREX STANDARD FLANGED PIPE, SEALED OFF

Figure 5.

Another thermal switch of different type is soldered to the boiler of the diffusion pump about an inch below the cooling coil. When the boiler is warm this switch is closed and thereby keeps the cooling system running until the boiler has cooled off, regardless of whether the manual control of the cooling system is on or off. In this way it is unnecessary for the operator to stay with the machine until the pumps have cooled off in order to stop the cooling system.

At one time a very frequent type of accident was the backing up of oil from the mechanical pump into the vacuum system. This occurred when the operator stopped the pump and forgot to break the vacuum in the pump by opening the relief valve. To break the vacuum automatically was not feasible since it would involve also the closing of the by-pass and fore-vacuum valves. Instead, an alarm system was installed. This consisted of a buzzer connected in series with a battery, the contacts of a relay, and a microswitch arranged to be closed when the relief valve was closed. The coil of the relay was connected in parallel with the pump motor so that when the pump was shut off the relay contacts closed and the buzzer buzzed until the operator opened the relief valve.

D. The 4-Inch Pumping Unit

Figure 6 is a photograph of the 4-inch pumping unit. It is a mobile unit but considerably larger and heavier than the 1-inch unit.

Method of assembly was similar to that of the 1-inch unit except that in the main pumping line use was made of electrically welded standard steel pipe and flanges. The by-pass and sample lines were of 1-inch copper pipe construction as previously described.

Pumps—The mechanical pump was nominally a 16 cfm International and the diffusion pump a Distillation Products MB-200 with a rated speed of 200 liters a second and a cutoff pressure of 10^{-4}. At pressures of the order of several microns the actual speed at the pumping line connection was probably about 200 cfm. Aroclor oil was used.

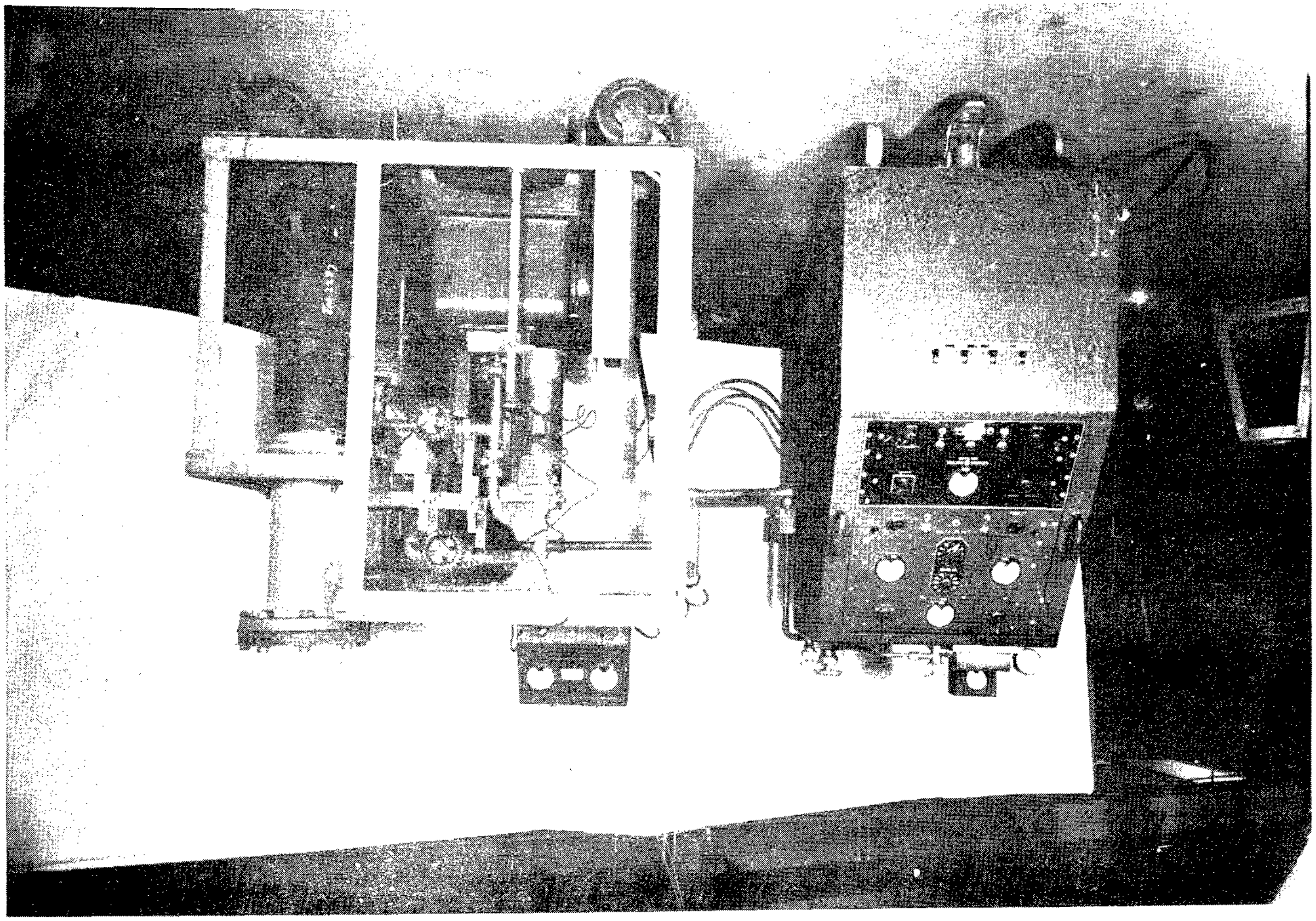
Valves—The high vacuum diffusion pump valve was a specially designed 4-inch bellows-sealed, rubber-seated angle valve, and is illustrated in Figure 7. All the other valves were of the modified 1-inch Kerotest type previously described.

Traps—The main trap was cooled with "dry ice." A 6 by 6 by 4-inch steel tee was welded onto the top of the diffusion pump, while the 4-inch end was flanged and bolted to the valve. The dry ice pot was of thin stainless steel welded into a flange which was bolted to the top of the tee so that the pot hung down into the tee. The by-pass and sample line traps were the 1-inch type previously described.

Couplings—The sample line coupling was a Dresser coupling placed at the same height and in the same relative position as the one on the 1-inch pump unit. The pumping connection was a standard 4-inch pipe flange with horizontal axis. The pumping line itself, external to the truck, was a 5-foot length of 4-inch copper pipe with opposed 90° elbows at the ends. To the elbows were silver-soldered brass rings which were made to match the sealing surface of standard 4-inch flanges, to which they were clamped by means of loose rings which were bolted through to the standard flange with a rubber gasket between the sealing faces. By clamping the pipe in the proper position it was possible to couple onto a system the height of whose outlet was anywhere from zero to eight feet above the floor.

Gauges—The gauges were of the same kind as those used in the smaller pumping outfit.

Cooling—Since this pumping system was quasi-stationary, it was not thought necessary to have a built-in cooling system. However, fittings were provided so that the spectrometer could be supplied with water from the pumping unit, the same as in the case of the small pumping units. The long external water lines were sometimes rigged overhead.



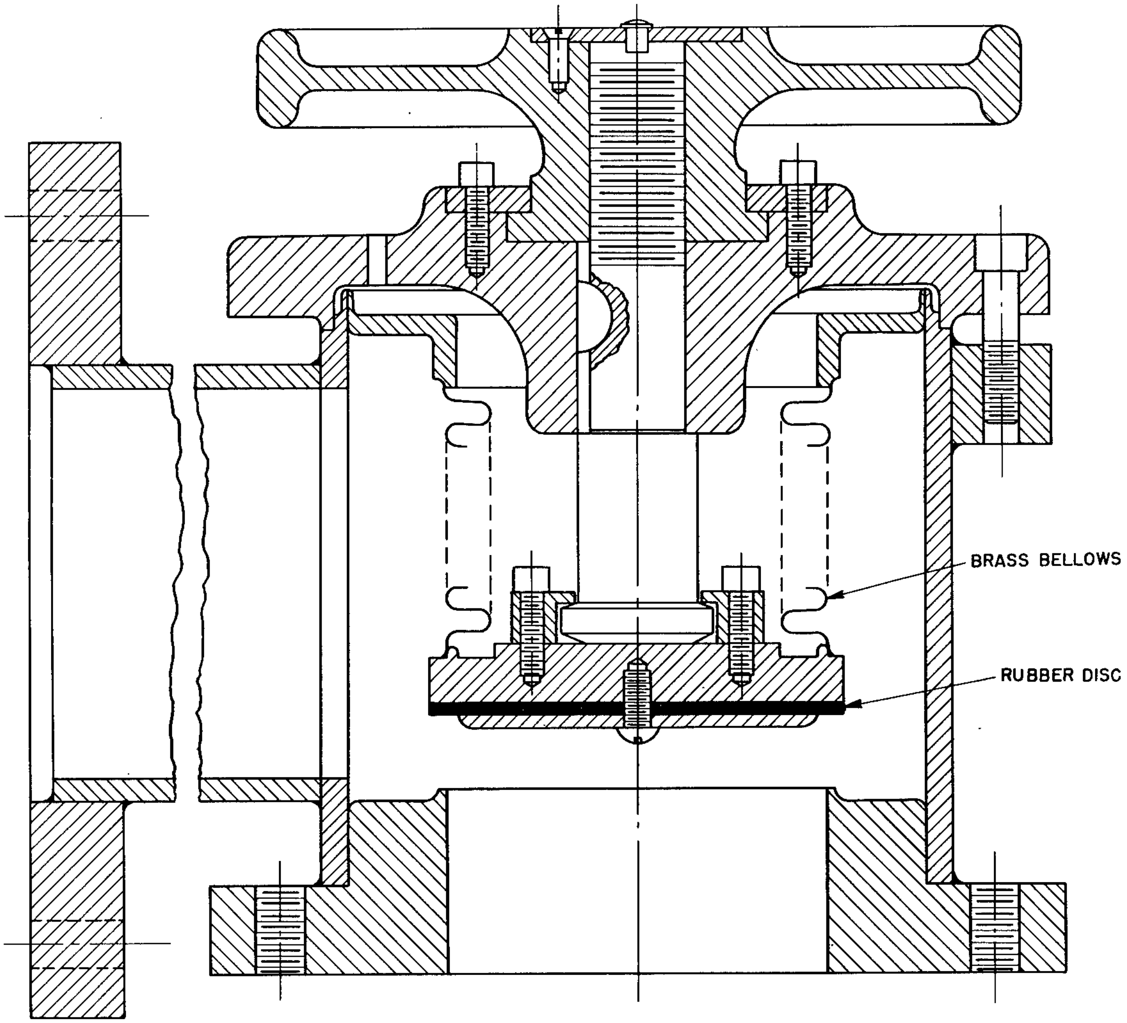


Figure 7.

III DEVELOPMENT OF COUPLING EQUIPMENT

The type of coupling equipment used with the 4-inch pumping unit has already been described. It could be adapted to other than 4-inch flanges by means of short reducing sections of pipe, etc. The need for flexibility was felt, mainly, however, in connection with the testing done by means of the smaller outfits. For use with these a stock of 1-inch metal hoses of various lengths was kept on hand. These terminated in 1 1/8-inch OD male fittings, to fit a standard 1 1/8-inch Dresser coupling. It was found that most of the testing involved coupling onto a copper or nickel tube of 1 1/8, 7/8, 5/8, 1/2, 3/8, 1/4 or 1/8 inch OD. For this purpose there were designed composite couplings having on one end one half of a standard 1 1/8-inch Dresser coupling and on the other a Dresser coupling or a "home-made" coupling of one of the previously mentioned sizes. A typical coupling of this sort is illustrated in Figure 8. About a dozen of each size were kept in stock. A number of caps similar to the couplings were made and used to blank off all the tubes except the one used for pumping.

The rubber gaskets were made from laboratory pressure tubing of good quality, live, black rubber. This tubing is made in ID's of 1/2, 3/8, 1/4, and 1/8 inch with sufficient precision. The 1 1/8, 7/8, and 5/8-inch sizes are standard and can be purchased. The outsides of the smaller gaskets are machined to size with a tolerance of about .003 inch. This is done in a lathe by means of a tool post grinder, the gaskets being held on an arbor. At one time considerable trouble was experienced in finding suitable rubber tubing. The principal defects were poor quality of rubber, out-of-roundness, and pitted inner surface.

The couplings were developed several months later; before that, the technique had been to use rubber tubing slipped over the end of a metal tube and sealed with grease. The new method proved easier, cleaner, safer, and more reliable from the point of view of vacuum technique. Also, the setting-up time was considerably shortened.

IV LEAK TESTING TECHNIQUE

A. General

The problem of how to do the leak testing work in the most effective manner — at SAM, at any rate — proved to be a rather broad one involving some hunam as well as physical factors. The technical development could be carried out with advantage only to a certain point; beyond that point the job reduced itself to an art.

Two main problems confronted the leak testers, the determination of tightness and the localization of leaks. The latter was much more difficult, and required far more art and judgment. The first question which had to be decided was which type of test ought to be made first. If the inherently lengthy localization test was made first and no leaks showed up, it would then appear that time could have been saved by making the overall tightness test in the beginning. On the other hand, in some types of system where slow-pumping pockets existed, it would be taking a considerable risk to make the overall test first, for if a large leak did exist, the system would be flooded with helium, and this would result in a low dynamic sensitivity which might persist for a long time. During this time the localization test would be blocked. Such questions had to be decided on the basis of the known structure of the system and an estimate of the probability of leaks. This estimate was based on the appearance of the workmanship on the joints, a knowledge of the man who did the job and of his past record, and on past experience with joints of the types present.

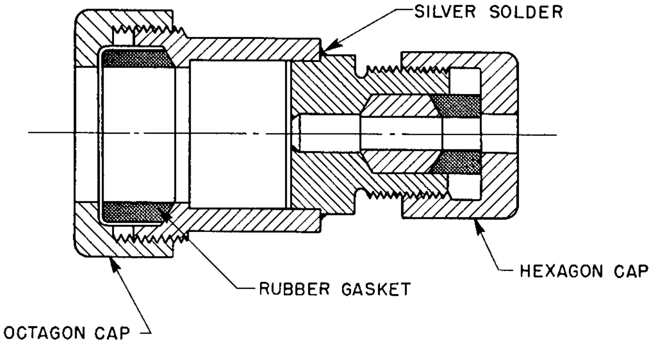


Figure 8.

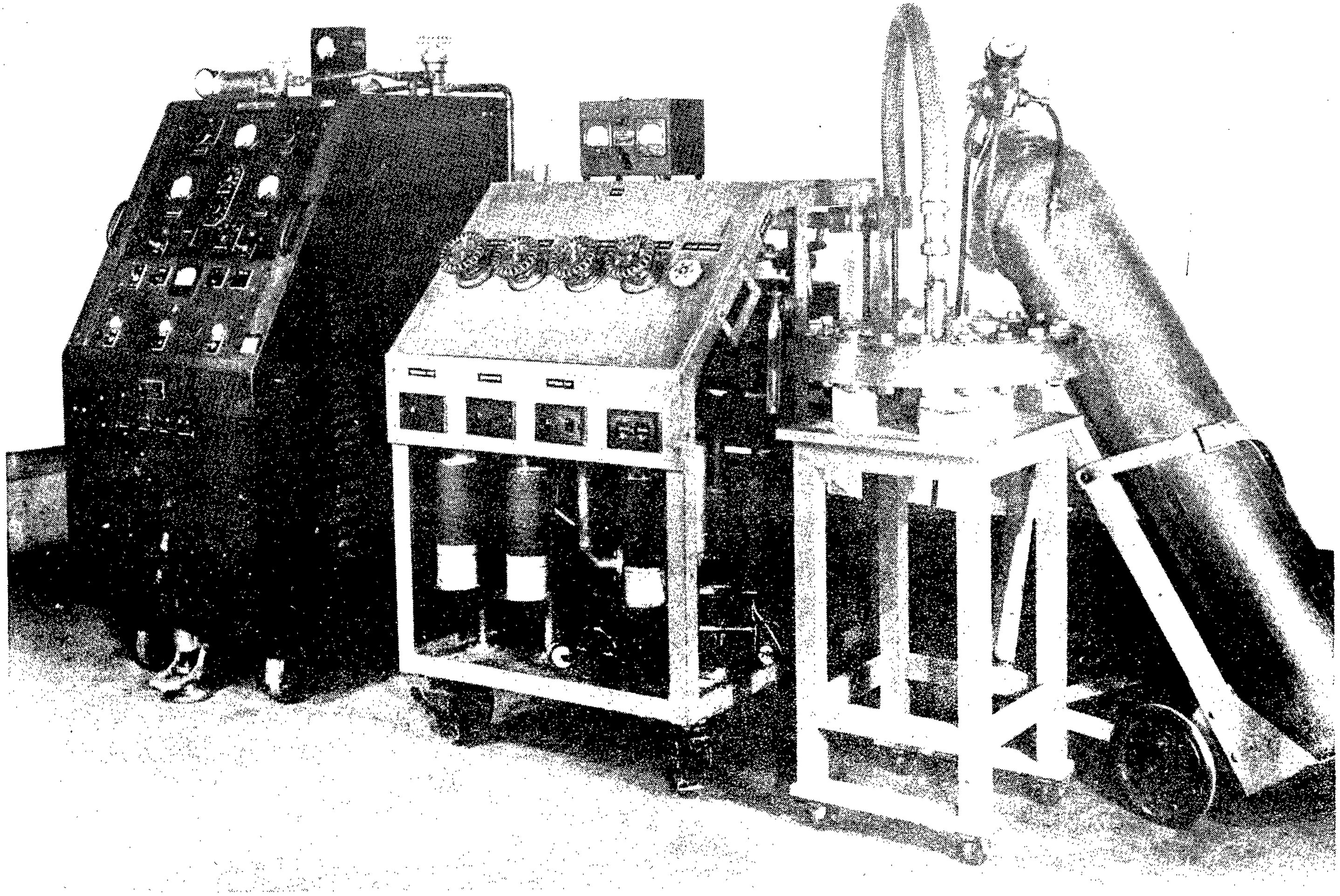


Figure 9.

It must be remembered that the question of effectiveness of leak testing is inseparable from that of workmanship. It is axiomatic that "the best way to get the leak testing done is not to have any leaks." Conversely, a means to the development of good workmanship is good leak testing. This presupposes that good leak testing includes close liaison, so that the man who makes the joints will know without undue delay which techniques to discard and which to develop. It was found that this regenerative cycle led to a fairly rapid improvement in workmanship, and an increase in volume of leak testing output. To some extent also, the leak testing department acted as a clearing house for sealing techniques. Because confidence in the validity of the leak testing results was essential to good liaison, from the very first a special effort was made to build a reputation for reliability. The establishing of a reputation involved not only correct results but also the dissemination of information which would lead to confidence in the methods. For example, at times distrust was encountered based on comparison with methods capable of showing only virtual leaks.

The work of the vacuum leak testing department was considerably lightened in times of stress by the willingness of many laboratories to test their apparatus where possible by the pressure-bubble method in order to find gross leaks. This was especially helpful in cases where the test vessel had a large volume and only a small pumping connection, with a consequently low dynamic sensitivity.

B. Localization of Leaks

Leaks may be localized either by the isolation method or by the probe method. In the isolation method the surface to be tested is divided up into several mutually isolated regions and the helium is applied to each of these regions in turn. Although this method does not yield detailed information, it is often sufficient and is sometimes useful as a preliminary to probing.

Isolation can be complete where it pays to make special gas-tight jigs or partial, where various regions are masked by wrapping with rubberized fabric. Masking is usually effective enough to remove an uncertainly which may have existed during rough probing unless the dynamic sensitivity is extremely low.

It sometimes happens that a test object contains two or more naturally isolated regions, internal or external. Thus, in a system of pipes and valves, it is often possible by opening and closing valves to narrow a leak down to a certain section. Or, it may be that some of the external surface is in the form of a pocket which connects with the atmosphere by way of a narrow channel. If a channel exists at both ends of the pocket, the air can be purged out and displaced with helium. If this is not the case, the air will have to be pumped out and helium let in to take its place. In either case, it is easy to determine whether a leak exists in the pocket even in the presence of a low dynamic sensitivity.

Another aspect of the isolation method is subassembly testing. In the case of extensive assemblies, especially those which contained some part which could not be adequately tested after assembly or which could not be repaired after assembly, it was found to be a great saving to leak test the subassemblies. In general, the dynamic sensitivity was better on the subassembly than on the main assembly.

The probe method consists of exploring the exterior of the object with a jet of helium. In general, this is a tedious but fruitful method; at its best, leaks can be localized to within 1/8 inch. The higher the dynamic sensitivity, the more successful the probe method becomes. Good judgment is required in adjusting the helium jet. This jet should, in general, be small. A helium probe could be visualized as a region where helium is present in a concentration which varies rapidly as a function of radial distance from the orifice and less rapidly as a function of angular distance from the center line of the orifice. This will be the case if the rate of flow is small enough so that the helium mixes with the air solely by diffusion. For high flow rates, however, turbulence sets in and the jet breaks up into large fragments of relatively high concentration which may be wafted considerable distances and thus get into a leak outside of the region which the operator thinks he is probing. This sort of probe obviously is confusing and the use of it by inexperienced operators has proved to be one of the greatest time wasters.

It would probably be of advantage to have a flowmeter in the probe line, but since the flow rates are usually very small this might be too difficult to arrange. In practice, flow rates were estimated by immersing the orifice in water and observing the bubbles, and this proved to be a very satisfactory method. The diameter of a probe might be defined as the maximum diameter of that surface at all of whose points the concentration is 50 per cent (or some other suitable value). For an orifice of given diameter it is possible, within limits, to vary the diameter of the probe by varying the flow rate. Beyond these limits the size of the orifice must be changed. For example, if a large orifice is used with a very small flow rate, air will diffuse into the orifice fast enough to cause a very low concentration even directly in the orifice, where it should be 100 per cent. A great deal can be learned about probes by studying gas flames.

In probing a long seam, the speed with which the probe is moved along the seam will depend on the dynamic sensitivity of the system and on the size of leak which can be tolerated. If either is low, the probe will have to be moved slowly in order to give any indication when it passes over a leak. It frequently happens that an overall test shows a leak, but the product of leak size and dynamic sensitivity is so small that it becomes a very tedious or almost impossible task to localize the leak. The dynamic sensitivity should be high enough so that such leaks fall below the tolerance, unless economic conditions are such that it pays to discard leaky units rather than to repair the leaks.

C. Overall Tightness Testing

This type of testing is comparatively easy. The system to be tested is enclosed in a reasonably tight enclosure, which may be a housing or even a piece of canvas. Helium is allowed to flow in, displacing the air. The leak detector will show an indication of helium proportional to the inleakage. It remains only to determine the proportionality constant. This may be done most simply by having a standard leak of size comparable to the expected inleakage, inside the housing and connected to the system. If the reading with the leak capped is D_1 and if the reading when the leak is uncapped is D_2, the inleakage lx may be calculated by solving for lx in

$$\frac{D_1}{D_2} = \frac{lx}{lx + ls}$$

where ls is the value of the standard leak. It is not necessary that the atmosphere inside the housing be pure helium; the concentration should be sufficiently high to give a good indication on the leak detector and the helium should be thoroughly mixed with the air.

An elegant variation of this method was worked out by Kellex. During steady pumping conditions the housing is first placed over the standard leak and helium is run in until the leak detector reads directly the value of the standard leak. The concentration inside the housing is read off directly in percentage by means of a conductivity cell apparatus. The housing is then placed over the test object and the concentration is run up to its previous value. The inleakage of the test object may then be read off directly from the leak detector.

A method which was used considerably at SAM consisted in first measuring the virtual leakage of the system and then multiplying by the concentration of the gas in the system and by the concentration in the housing. The virtual leakage was obtained by observing the rate of pressure rise with pumps closed and multiplying this rate by the volume of the system. The helium concentration in the system was obtained by using a standard helium-air mixture to calibrate the leak detector in terms of percentage helium at the pressure of the test. The helium concentration inside the housing was either made 100 per cent by continued purging with helium or was calculated as explained in Appendix E.

APPENDIX A

CONDUCTANCE AND PUMPING SPEED

The flow of gases in a system is dependent on (1) the conductances of the component parts of the system and (2) the speed of the pumps. By the conductance of a pipe is meant the volume of a gas, measured at the inlet pressure, which will pass through the pipe in unit time under given conditions. By the speed of pump, or pumping speed, is meant the rate at which the pump removes gas from a vessel, the volume of gas being measured as described on page 11 of this document. Pumping speed is roughly constant over a wide range of pressures. Both pumping speed and conductance are frequently expressed in cm^3/sec, liters/sec, and ft^3/min or cfm.

If a vessel is being evacuated by a pump of speed S_p through a number of series connected pipes of conductances S_1, S_2, ..., the pumping speed S as measured at the vessel is given by

$$\frac{1}{S} = \frac{1}{S_1} + \frac{1}{S_2} + \cdots\cdots + \frac{1}{S_p} \qquad (1)$$

The resultant conductance S of the pipes arranged in parallel is

$$S = S_1 + S_2 + \cdots\cdots \qquad (2)$$

These formulas are analogous to the familiar electrical resistance laws which are derived from the formula $V_1 - V_2 = RI$; here, however, we have $p_1 - p_2 = 1/S\ Q$, where $p_1 - p_2$ is the difference in pressure, 1/S is the resistance of the pipe, and Q is the mass flow of gas.

The conductance of a pipe depends not only on its dimensions but also on the molecular weight and the viscosity of the gas flowing through it and on the value of the pressure at each end of the pipe; the relative importance of each of these factors in turn depends on the product of the pipe diameter and the average pressure (see equation 6).

At pressures such that the mean free path of the gas molecules is much smaller than the diameter of the pipe, intermolecular collisions are the predominant factor in determining the conductance. The flow is a function of the viscosity of the gas, and we may apply Poiseuille's law* for viscous flow, namely

$$S = \frac{Q}{p_1 - p_2} = \frac{\pi d^4 p}{128\ n\ L} \qquad (3)$$

where the conductance S is in cm^3/sec; Q is the amount of gas that passes per second (as measured by its pV value) in dyne-cm/sec; d and L are the diameter and length of the pipe in cm; n is the viscosity in poises; and p is the average pressure in microns Hg.

When the molecular mean free path is much greater than the diameter of the pipe, the flow is "molecular" or "diffusive," and the law which then applies is

$$S = \frac{\sqrt{2}\,\pi d^3}{6\sqrt{p_1}\,L} \qquad (4)$$

where p_1 is the density of the gas at a pressure of one dyne/cm^2.

An empirical formula due to Knudsen not only includes the previously mentioned cases but also covers the intermediate pressure region where the mean free path and the pipe diameter are of the same order of magnitude. This formula in general terms follows:*

$$S = \frac{\pi d^4 p}{128\ n\ L} + \frac{\sqrt{2}\,\pi d^3}{6\sqrt{p_1}\,L} \left[\frac{n + pd\sqrt{p_1}}{n + 1.24\,pd\sqrt{p_1}}\right] \qquad (5)$$

---

* Valid only for long straight pipes when the flow is nonturbulent.

For the specific case of the flow of air at 20°C, equation 5 reduces to

$$S = \frac{d^3}{L}\left[6.28\ dp + \frac{(165)\ (1 + 1.29\ dp)}{1 + 1.80\ dp}\right] \qquad (6)$$

where S is in cfm, d and L are in inches, and the average pressure p is in microns Hg.

The accompanying nomogram (Figure 10) is based on equation 6 and may be used in its place to determine conductance (conductivity) of a pipe. The conductance of a hole at such pressures that the flow is predominantly molecular may also be read from the chart.

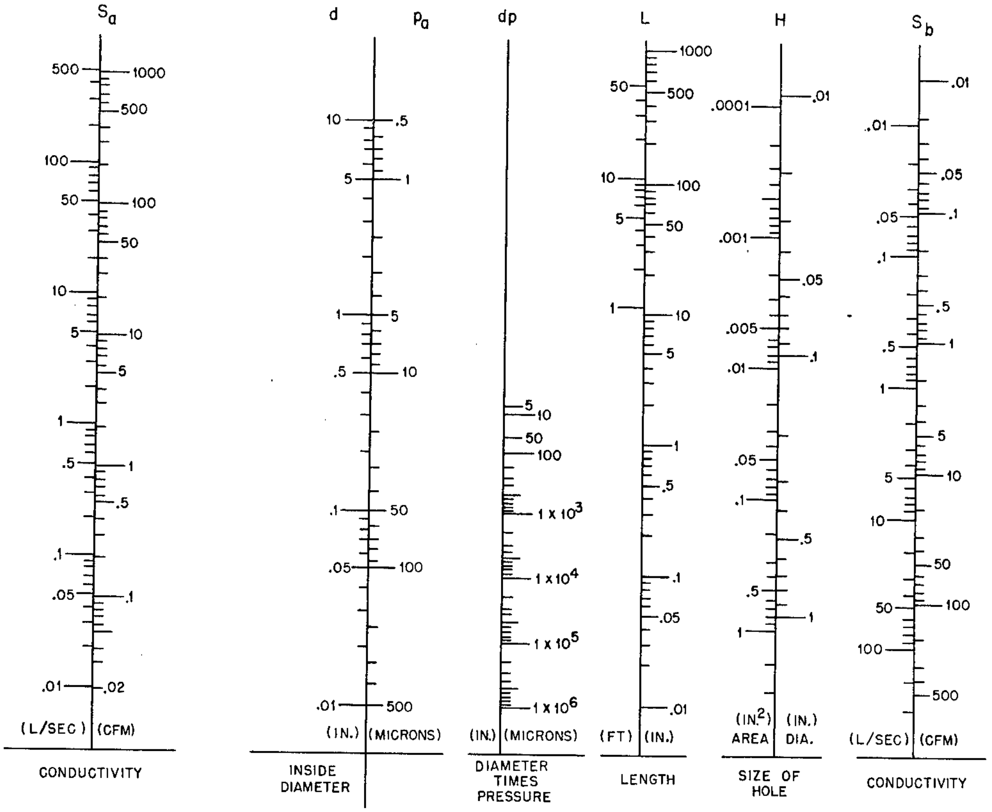


Figure 10. The flow of air through pipes and holes. The equation for the flow of air through a pipe at room temperature is

$$S = \frac{6.28\ d^4 p}{L} + \frac{165\ d^3}{L} \times \frac{1 + 1.29\ dp}{1 + 1.80\ dp}$$

where S is the conductivity (in cfm) of a pipe whose diameter d and length L are given in inches; p (in microns) represents the average of the inlet pressure and the outlet pressure.

To find the conductivity when L, d, and p are known, extend the line joining the appropriate points on the L and d scales to the S_a scale. Note the value on the p_a scale through which the line passes.

(a) If $p < p_a$, the intersection at the S_a scale represents the conductivity of the pipe.

(b) If $p > p_a$, multiply d (in inches) by p (in microns) and locate this value on the dp scale. Pass a line through the point on the S_a scale and the point on the dp scale; the intersection of this line with the S_b scale gives the conductivity of the pipe.

To find the conductivity at low pressures of a hole, read the S_b scale directly to the right of the point on the H scale which represents the diameter or the area of the hole.

APPENDIX B

REACTION TIME IN A VACUUM TESTING SYSTEM

Suppose that a system such as has been described has been set up. The system is pumped down until the pressure at each point is substantially constant, although not necessarily the same at all points. This steady state pressure will depend on the rate of outgassing of the various surfaces and on the pumping speed at the various points. The sensitivity with which leaks can be detected by exploring with probe gas will depend on a number of factors such as rate of outgassing, sensitivity of the gas analyzer, volume of the system, speed of pumping, length of pumping line, and pressure in the system. In a discussion of the dynamic sensitivity an important consideration is the reaction time. This may be defined as the time elapsed between the application of pure probe gas to the outside of a leak and the first definite (above-threshold) indication shown by the gas analyzer. The various factors affecting the reaction time will be taken up in order.

A. Pocket Effect

If the leak is simple, i.e., short, direct, and of uniform cross sectional area, the time required for the probe gas to penetrate into the vacuum is usually negligible. If. however, the leak path is very long or has one or more enlargements which form pockets, the delay may be considerable. The effect is similar to that encountered when an electrical signal is suddenly applied to one end of a filter or a long line. In the case of a leak with pockets there may be a further complicating effect. If one or more pockets contain air at a pressure in excess of about 1 mm,there will be a delay caused by the slow diffusion of the molecules of probe gas through the air. Frequently the construction of the apparatus is such that these atmospheric pockets exist. When such is the case, provision must be made either for pumping out or for purging out the air.

B. Charging Effect

Part of the delay in the response to a suddenly applied probe is analogous to the delay observed when a condenser is charged as part of a resistance network. Volume is analogous to capacity, pressure to voltage, and rate of gas flow to current.

As an example, we calculate the effect in the case of a system virtually all of whose volume is concentrated in a single vessel. We assume also that all gases entering the vessel are completely and instantaneously mixed. At low pressures this is very nearly true because of the high rates of diffusion.

Let V = volume (cu ft.) of the vessel

p = partial pressure (microns) of probe gas in the vessel

P = total pressure (constant) in vessel

l = inleakage (micron cu ft. per hour)

L = virtual leakage (micron cu ft. per hour)

S = pumping speed (cu ft. per min) at entrance to pumping line

t = time (min) elapsed since application (or removal) of probe

Probe gas enters the vessel at the rate l and leaves at the rate pS. The rate of accumulation of probe gas is therefore

$$l - pS = V \frac{dp}{d_t} \qquad (1)$$

The solution of this equation is

$$\frac{p}{P} = \frac{l}{L} (1 - e^{-St/V}, \qquad (2)$$

The time required to obtain a given probe gas concentration is plotted in Figure 11. It will be observed that the more probe gas is present, the more time is required to get a given rise in probe gas concentration. In practice this means that dynamic testing becomes increasingly difficult as the amount of probe gas in the system increases.

If, initially, the probe gas concentration of p/P and the probe is suddenly removed, the concentration will decay according to the equation

$$\frac{p}{P} = \frac{p_0}{p} e^{-St/V} \qquad (3)$$

It will be noticed that the probe gas concentration grows to the threshold value much faster than it decays to below the threshold value. In systems of large <u>time constant</u> (V/S) this can be very troublesome and has sometimes led to the erroneous conclusion that helium had been adsorbed inside the system.

C. Diffusive Propagation

As the concentration of helium builds up in the test vessel, it simultaneously builds up in the gas analyzer. Under ideal conditions, the concentration in the analyzer will at all times be the same as that in the vessel. This will be the case at low pressures where diffusion is rapid provided the volumes and speeds of the analyzer and the associated piping are such that the charging effect in these parts is negligible. At pressures from roughly 100 microns up, however, diffusion must be considered.

Consider the case of a long pipe containing air at uniform pressure p. The air is stagnant, i.e., the average or drift velocity is zero. At one end of the pipe is a valve on the other side of which is a vessel containing a mixture of air and helium at the pressure p and at concentration N_0. The valve is suddenly opened at time t = 0. The problem is to find the concentration N at any point of the pipe as a function of the distance x from the valve and the time t. The problem is analogous to the problem of describing the temperature variations in a metallic rod, initially at uniform temperature, one of whose ends is suddenly raised to and held at a higher temperature. The differential equation is

$$\frac{\delta N}{\delta t} = D \frac{\delta^2 N}{\delta x^2}$$

and its solution is

$$N = N_0 \left[1 - \frac{2}{\sqrt{\pi}} \int_0^{2} \frac{x}{\sqrt{tD}} e^{-\beta^2} d\beta \right]$$

For most molecules the constant D (coefficient of diffusion) is, in air at atmospheric pressure, about 0.1 cm²/sec. D varies inversely with pressure.

A related problem is that of the decay of concentration in a pipe in which the initial concentration was N_0 throughout, and one end of which is suddenly, at t = 0, connected to a vessel in which the gas has the same pressure but zero concentration. The solution is

$$N = N_0 \frac{2}{\sqrt{\pi}} \int_0 \frac{x}{2\sqrt{tD}} e^{-\beta^2} d\beta$$

The variation with respect to distance and time are plotted in Figure 12 and Figure 13.

D. Convection Propagation

It seldom happens that we have to rely on diffusive propagation alone. When the pressure is high enough so that diffusive propagation has to be considered, there is almost always a convection effect. The gas in the pipe may be considered to be moving as a whole, with the diffusive effect superimposed. This drift velocity is simply

$$v = S/A$$

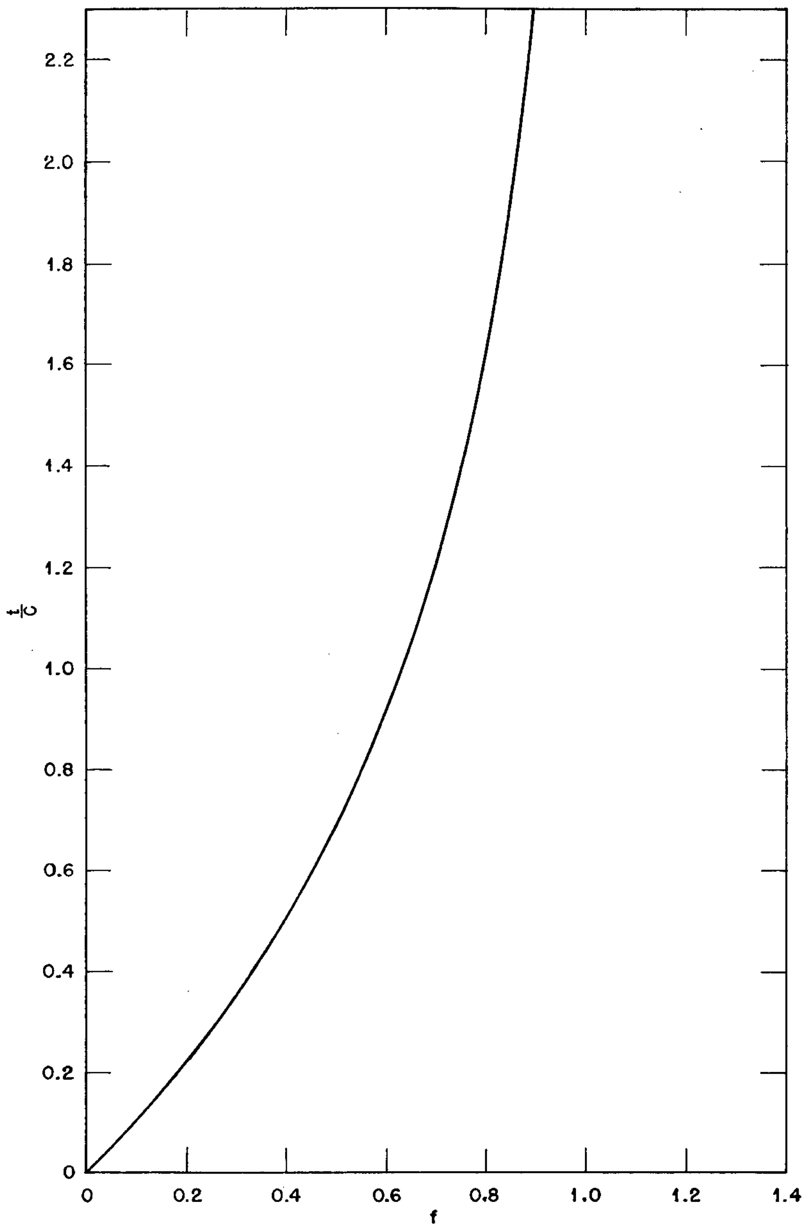


Figure 11. Time (t) required for concentration (C) in a reservoir to reach a fraction (f) of its steady state value

$$t = \frac{V}{S}$$

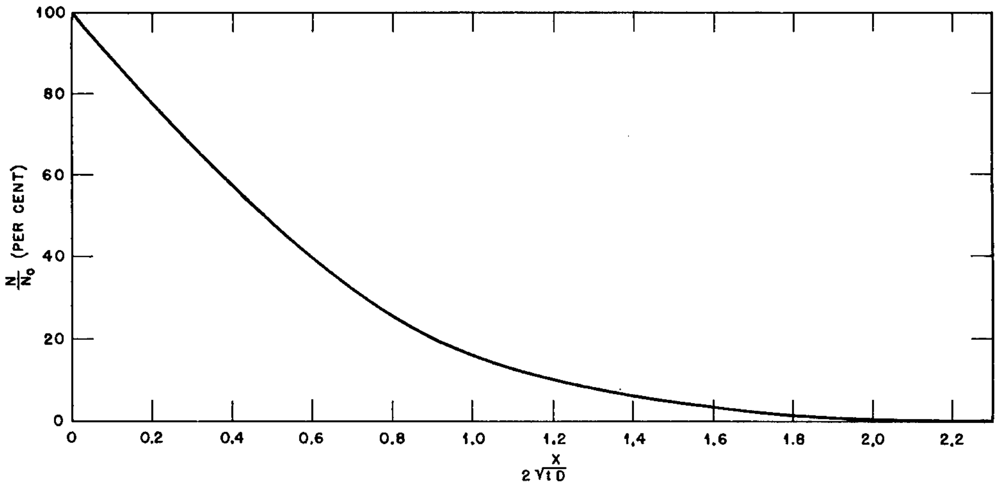


Figure 12. Diffusion propagation of a helium front in a long tube.

$$\frac{N}{N_0} = 1 - \frac{2}{\sqrt{\pi}} \int_0^{\frac{x}{2\sqrt{tD}}} e^{-\beta^2}\, d\beta$$

Where D = coefficient of diffusion of He in air at pressure in tube.

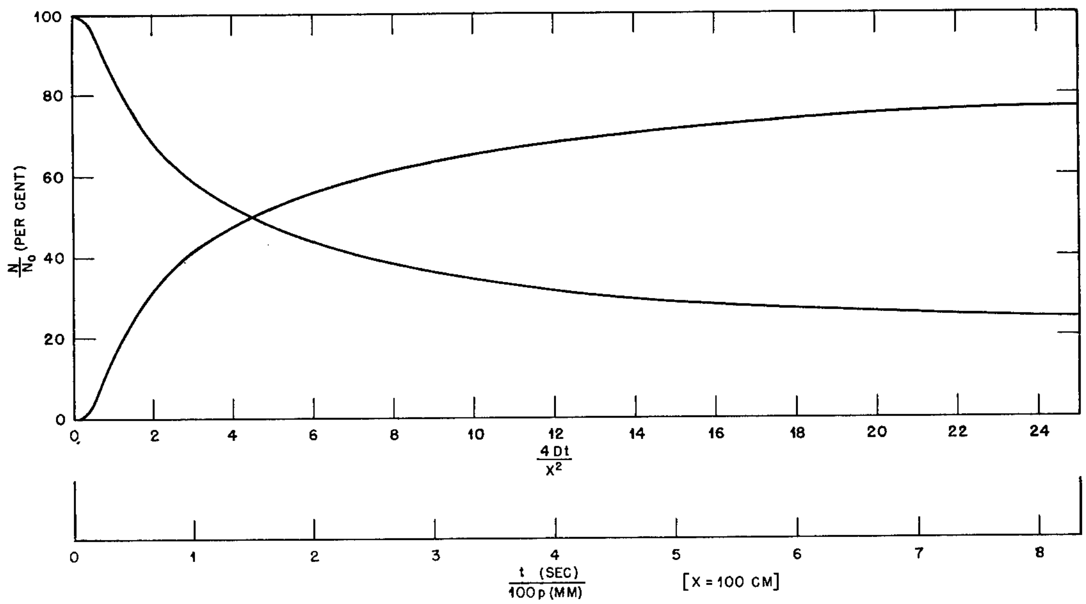


Figure 13. Diffusion propagation of a helium front in a long tube.

$$\frac{N}{N_0} = 1 - \frac{2}{\sqrt{\pi}} \quad \frac{x}{2\sqrt{tD}} \quad e^{-\beta^2} \quad d\beta \text{ (growth)}$$

$$\frac{N}{N_0} = \frac{2}{\sqrt{\pi}} \quad \frac{x}{2\sqrt{tD}} \quad e^{-\beta^2} \quad d\beta \text{ (decay)}$$

where S and A are, respectively, the pumping speed and the cross sectional area at the point in question in appropriate units. For example, the drift velocity in a pipe of .01 sq ft cross section when pumped at 10 cu ft per min would be 1000 ft per min. The practical significance of this effect is embodied in the rule that (especially at high pressures when only a relatively slow mechanical pump is being used) the pumping line should be no larger than necessary to utilize the full speed of the pump. Also, whenever a throttle valve is used in a line it should be at the upstream end of the line so as to achieve the greatest possible pumping speed in the line as well as the lowest possible pressure. It goes without saying that all lines, especially those in which the drift velocity is low, should be kept as short as possible.

E. Delay in Response of Gas Analyzer

In the case of the mass spectrometer the delay inherent in the instrument itself is negligible, the time constant being of the order of 1/10 second. With some other types of gas analyzer, however, the time constant may be large enough to be a serious factor. This is true where the indicator is a sensitive galvanometer. It is also true where a thermal detector such as a hot filament is used, since there is a practical limit to the extent to which the thermal capacity can be reduced. By the application of principles similar to those used in feedback amplifier design, it might be possible further to reduce the effective time constant.

APPENDIX C

DYNAMIC SENSITIVITY

We may define a figure of merit for leak localization, the <u>dynamic sensitivity</u> σ, as the reciprocal of the smallest leak detectable under given conditions of probing. In Appendix B we have already defined reaction time as the elapsed time between the sudden application of helium to the outside of a leak and the appearance on the instrument of the least detectable indication. The dynamic sensitivity depends on two other main factors: probe diameter D and probe velocity v. The length of time the leak remains within the probe is D/v. The leak whose reaction time is D/v is therefore the smallest leak which can be detected. There exists a functional relationship

$$l = f\,(\Theta) \tag{1}$$

between leak size and reaction time which in general is quite complicated, depending on the various effects discussed in Appendix B. For normal testing, however, the only effect of much importance is the charging effect, and we confine our discussion to it.

It has been shown that the concentration of helium varies with time according to

$$\frac{p}{P} = \frac{l}{L}\,(1 - e^{-St/V})$$

If $\left(\frac{p}{P\ \mathrm{min}}\right)$ is the least detectable concentration, equation 1 becomes:

$$= \frac{p}{P\ \mathrm{min}}\quad \frac{L}{1 - e^{-S\Theta/V}}$$

So that

$$\sigma = \frac{1}{l\ \mathrm{min}} = \frac{1 - e^{-\frac{S}{V}\,\frac{D}{V}}}{\dfrac{p}{P\ \mathrm{min}}\quad L} \tag{2}$$

As we might expect, the dynamic sensitivity is inversely proportional to the least detectable concentration and to the virtual leak rate (substantially the outgassing). It increases with pumping speed and with probe diameter and decreases with volume and probe velocity exponentially.

APPENDIX D

DIFFERENTIAL EFFECTS IN A SYSTEM PUMPING NEAR CUT-OFF PRESSURE

In the foregoing it was tacitly assumed that the vacuum pumps pumped all gases indiscriminately; that is, that over the entire pressure range all gases were pumped with the same speed. This is not strictly true. At low pressures, for instance, the light molecules will certainly diffuse out faster than the heavy ones. There has been observed another more important differential pumping effect, however, and an attempt will be made here to explain it.

It has been observed that when a mixture of helium and air is being pumped, the observed percentage of helium drops by a large factor when the system is opened to a diffusion pump. It is also observed that the drop in total pressure when the pump valve is opened is negligible, a fact which shows that the pump is working near its cut-off pressure. It thus appears that although at this pressure the pumping speed for the total gas is zero, nevertheless the pumping speed for the helium fraction is considerable.

The following is an attempt to explain this effect in the terms of back diffusion of nonhelium gases and vapors. The idea of back-diffusion will be used first to derive an approximate relation between pressure and speed in a simple diffusion pump and will then be extended to other cases.

A. Speed of a Simple Diffusion Pump

Diffusion pumping is a matter of statistics. A jet of high-speed, unidirectional molecules is set up in such a way as to divide the system into two regions such that a randomly moving gas molecule is much more likely to pass from region A to region B than in the opposite direction. That being the case, the actual net passage of molecules is from region A to region B, although individual molecules can and do pass in either direction. The number of molecules passing from A to B is proportional to the number of molecules, (i.e., the pressure) in region A. As this pressure becomes lower, the forward diffusion becomes smaller and the back diffusion (essentially constant) becomes more important.

Let P = backing pressure (constant)

p = inlet pressure

p_0 = inlet pressure at cut-off

W = a number proportional to the probability for a molecule to pass <u>forward</u> through the jet in a given time

w = a number proportional to the probability for a molecule to pass <u>backward</u> through the jet in a given time.

The net mass rate of flow is:

$$p\,W - P\,w$$

The net volume rate of flow or pumping speed is:

$$S = \frac{p\,W - P\,w}{p} \qquad (1)$$

By definition, as $p \to p_0$, $S \to o$.

$$o = p_0\,W - Pw \qquad (2)$$

For p sufficiently high, $p\,W \gg Pw$, and S approaches a constant maximum:

$$s_{max} = W$$

Substituting in equation 2,

$$s = \frac{p_0}{p} S_{max} \tag{3}$$

This describes fairly well the behavior of a diffusion pump operating with constant backing pressure.

B. Pumping of a Mixture by a Simple Diffusion Pump

We now consider the problem of the pumping of a mixture of two gases, say helium and air, such mixture being introduced in a fixed proportion, as through a leak or valve.

Let M = rate of mass flow of total gas

M' = rate of mass flow of helium

p' = helium partial pressure at inlet

P' = helium partial backing pressure

For simplicity we assume that the probabilities W and w are the same for helium as for air.

$$M = p\,W - P\,w$$

$$M' = p'\,W - P'w$$

$$\frac{M'}{M} = \frac{p' \frac{W}{w} - P'}{p \frac{W}{w} - P}$$

$$= \frac{p' \frac{P}{p_0} - p'}{p \frac{P}{p_0} - p}$$

$$= \frac{p' - p_0\, P'/P}{p - p_0}$$

$$p' = (p - p_0) \frac{M'}{M} + \frac{P'}{P} p_0$$

$$\frac{p'}{p} = (1 - \frac{p_0}{p}) \frac{M'}{M} + \frac{p_0}{p} \frac{P'}{P}$$

This equation shows the way in which the helium concentration p'/p depends on the pressure p, as well as on the concentration M'/M of the gas as introduced through the leak and on the concentration P'/P on the backing side of the pump. The pressure may be varied by varying the size of the leak. As $p \to p_0$, $\frac{p'}{p} \to \frac{P'}{P}$, which may be much smaller than M'/M if the backing pump also shows a differential effect.

While the effect has been investigated for a single stage diffusion pump only, it will be seen that it exists for all vacuum pumps. The treatment of a multistage diffusion pump is more involved since the backing pressures are variable. In the case of a mechanical pump it is thought that the ultimate vacuum is determined mainly by the vapor pressure of the oil rather than by leakage of atmospheric air back through the valve. Nonetheless this results in a differential pumping effect similar to the one already discussed when the pump is operated near its cut-off pressure.

APPENDIX E

CONCENTRATION OF PROBE GAS INSIDE A HOUSING

If helium is introduced into a moderately tight housing equipped with fans to provide stirring, it is possible to compute the percentage concentration.

Let v cubic feet of helium be introduced into a housing of volume V. Let f be the percentage concentration of helium. For each volume dv of helium added, an equal volume dv of helium-air mixture is displaced. The volume of helium (at atmospheric pressure) contained in this is $\frac{f\,dv}{100}$. The net addition of helium is therefore $(1 - \frac{f}{100})\,dv$. The resultant change in f is

$$df = \frac{100\left(1 - \frac{f}{100}\right)dv}{V}$$

$$= \frac{(100 - f)\,dv}{V}$$

$$\frac{df}{100 - f} = \frac{dv}{V}$$

The solution of this equation is

$$f = 100\,(1 - e^{-v/V})$$

The flow of helium into the housing must be large enough that dilution of the mixture inside the housing by diffusion through the cracks is negligible.

APPENDIX F

PROPOSALS FOR REDESIGN

Some thought was given to ways and means of making the leak detector more satisfactory as an industrial tool. Following is a list of suggested features which ought to be considered.

1. Combination of spectrometer and small pumping outfit into one compact mobile unit about the size of the present spectrometer unit.

2. Air-cooled diffusion pumps for convenience, simplicity, and compactness.

3. Use of stable pump oil, such as silicone.

4. Satisfactory operation without refrigerated traps, but easily-cleaned traps provided to be used with coolants in cases where ultimate sensitivity must be realized.

5. Vacuum system designed to use as few pumps and valves as possible.

6. Power operated valves. Pneumatic operation would be simple, long-lived, clean, and would facilitate the laying out of the vacuum system since the valves would not have to be accessible to the operator.

7. Quiet and clean mechanical pumps, such as the Duoseal.

8. Demountable vacuum parts.

9. Test manifold for small objects provided as part of machine. Also a table top on the machine for small test objects.

10. As few as possible ''loose'' auxiliaries which might get lost.

11. Simple and reliable quantitative method using standard leaks.

12. Pressure gauge which holds its calibration better than a Pirani. This might be a viscosity gauge such as an electromechanical vibrator forming an element of an AC bridge.

13. Protective interlocks in vacuum system to prevent misoperation of valves.

14. Elimination of all batteries.

15. Elimination of fragile parts.

16. Construction capable of withstanding rough handling in shipping, but not too heavy. Construction should be such as to require a minimum of disassembly for shipping.

17. Use of mass spectrometer tube which is easily disassembled for cleaning and which does not require frequent cleaning. For ease of disassembly, the semicircular focusing tube used in the Westinghouse leak detector is the best design known to us. For a description of the development of a tube intended to meet the above specifications, see a forthcoming report by F. Stein, et al.