

Modern Vacuum Practice in Electronics

The entire electronics industry is dependent upon the electron tube which in turn is dependent upon the production of high vacuum. In this article the author discusses modern vacuum technique and the application of recently developed synthetic organic compounds to diffusion pumps

THE production of high vacuum appears to be one of the fundamental problems inherent to the manufacture of almost every electronic device. The actual final pressures required may vary from a fraction of a millimeter for a neon sign to less than 10^{-8} mm in the case of a high voltage cathode ray tube.

Mercury diffusion pumps developed by Gaede¹, Langmuir², and others³ have for many years been standard equipment for the commercial exhaustion of x-ray, cathode-ray, and radio tubes, photo-electric cells, etc. Because of the relatively high vapor pressure of mercury at room temperatures, *i.e.*, approximately 10^{-3} mm, and the tendency of a mercury vapor to remain supersaturated, pumps of this type can generally be employed only with the aid of cold traps employing refrigerants such as liquid air or solid carbon dioxide. In addition to the expense and inconvenience of providing such refrigerants, the more common traps materially reduce the available pumping speed, generally by a factor of from 4 to 10. Although mercury has the advantage of being an inexpensive and stable material, it is subject to contamination and the vapor tends to wander. For optimum performance, mercury pumps and attendant pipe lines may require frequent cleaning.

Burch⁴ advanced the art a significant step by suggesting the use of low vapor pressure fractions of hydrocarbon oils as a substitute for mercury. These fluids, commercially known as Apiezon oils, permit the attainment of relatively low pres-

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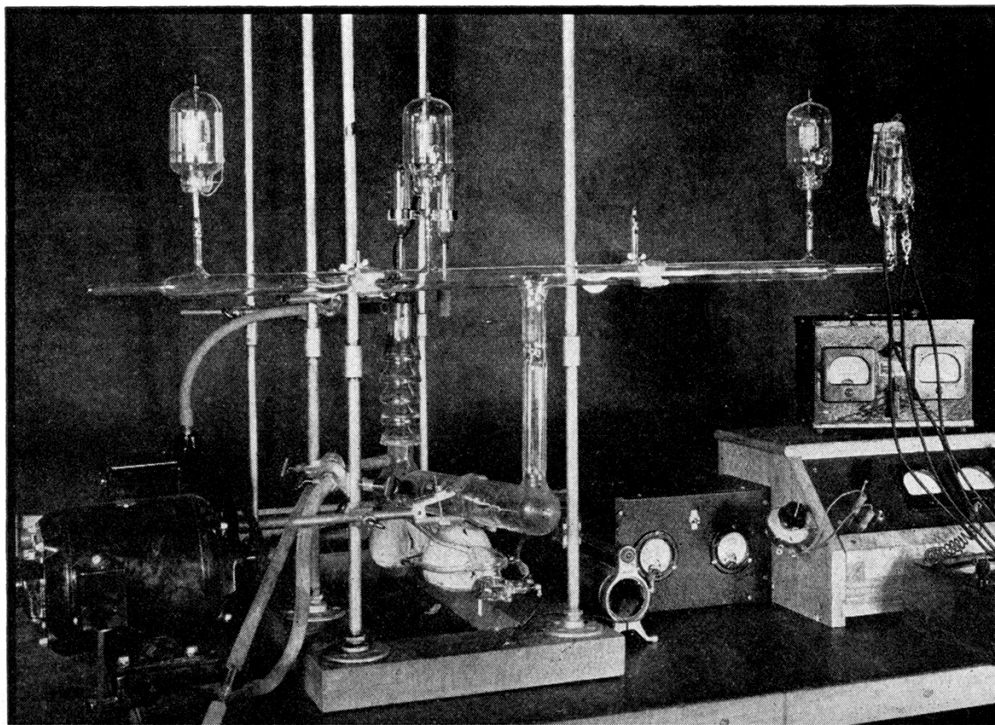
sures, less than 10^{-6} mm, without a cold trap of any type. Although with such oils a considerably lower fore pressure is required than with the older mercury pumps, units charged with the Apiezon oils soon found limited usefulness abroad in the broadcasting industry where they provided the vacuum for demountable transmitting tubes. However, in spite of their high pumping efficiency as compared with mercury, these materials have not found wide application in the commercial field in the United States. This is probably due to three causes, the most significant being that pumps of a type capable of bringing out their best features were not available. Hydrocarbon fluids also tend to crack, breaking down into higher

vapor pressure components, particularly if air is admitted when the pump is hot.

In 1930, Hickman and Sanford⁵ suggested replacing the hydrocarbon oils with highly purified synthetic organic compounds such as esters of phthalic acid and in particular butyl phthalate.

More recently the development of compounds of much lower vapor pressure⁶ have rendered n-butyl phthalate obsolete except in cases where the lowest pressures are not of primary importance. By employing some of the newest esters such as Octoil and Octoil-S it is possible to obtain pressures at room temperature which are as low or lower than those made possible with the best mercury pumps employing liquid air traps.

In operating any type of diffusion pump regardless of whether one employs mercury, hydrocarbons, or synthetic oils, the question



Experimental vacuum system to exhaust transmitting tubes without the aid of a cold trap

of contamination of the fluid is of prime importance. Gaede⁷ has shown that the lowest pressure attainable with a vapor pump corresponds to the vapor pressure of the working liquid. It must also be remembered that this pressure may depend upon contaminating materials even though present in minute quantities.

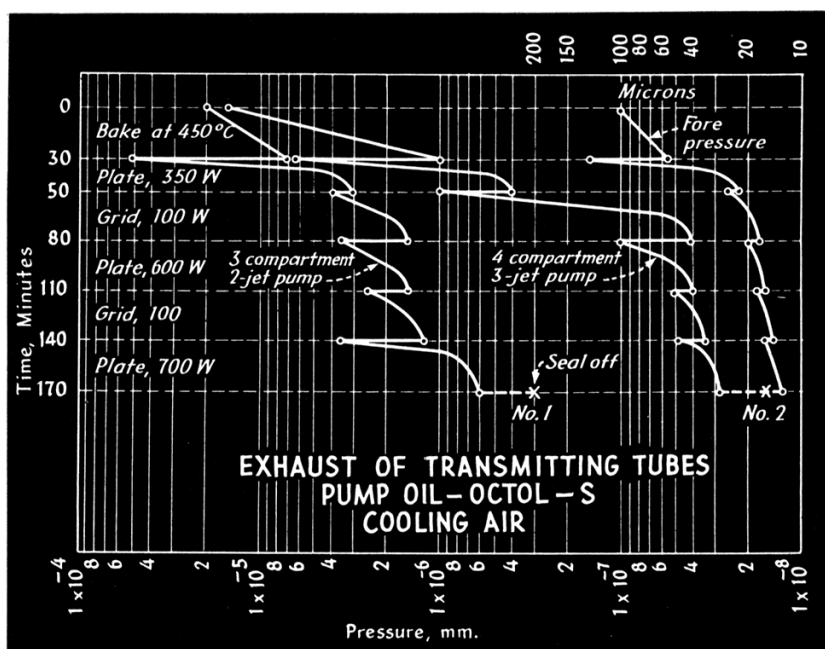
Hickman⁸ has investigated this general problem and has demonstrated that through the use of fractionating pumps the lowest pressures commensurate with the pumping fluid are assured at all times even though reactive and relatively high vapor pressure materials are being handled by the system. These findings have been confirmed by others⁹, and the term "Fractionating pump"¹⁰ has come into use for any type of diffusion pump in which the pumping fluid is continuously purified during operation, the volatile constituents being rejected or segregated at the fore pressure end of the system and only the lowest vapor pressure materials being allowed to reach the high vacuum jets.

Exhaustion of Transmitting Tubes

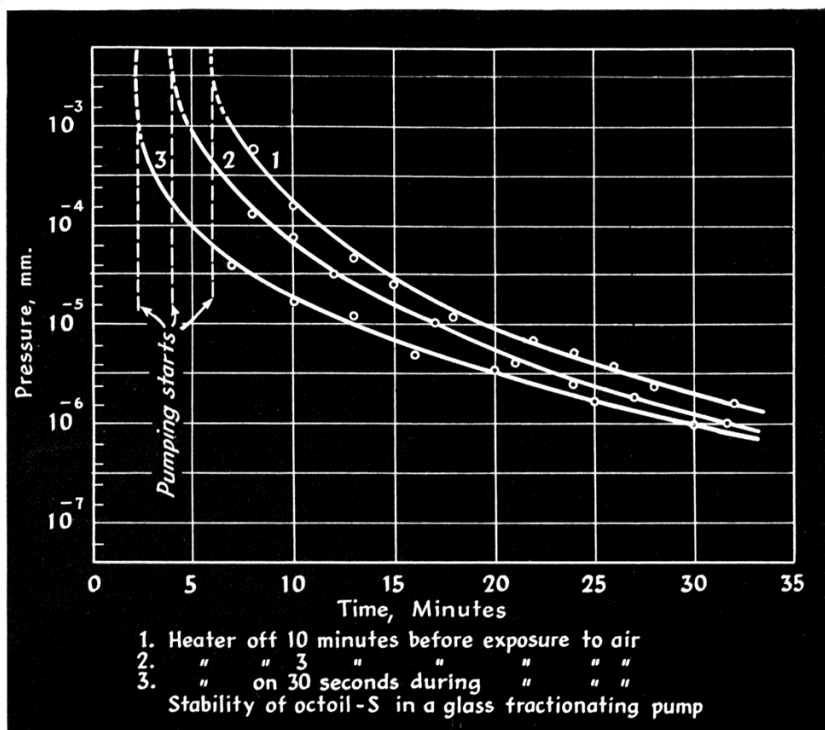
In spite of the extremely low pressures which have been consistently obtained with fractionating oil pumps by many scientific workers equipment of this type has only recently been used for commercial purposes. In order to demonstrate the effectiveness of this newly developed apparatus, several types of standard transmitting tubes have been evacuated according to schedules generally used with mercury pumps employing liquid air traps.

A typical arrangement of work of this type is shown on page 33. The diffusion pump is a 3-compartment, 2-jet all-glass fractionating unit and as can be seen, direct connection is made to the manifold without the usual high impedance trap or cold sink.

During the exhaust schedules of various types of transmitting tubes, continuous pressure readings were made on the manifold with an ionization tube (Western Electric Type D79512). The fore pressure was also measured with the aid of a Pirani type gauge. Typical results are shown in the upper diagram, the schedules as given approximating



Exhaust curves of medium power transmitting tubes illustrating the difference between a two jet pump with air cooling and a three jet pump with ice cooling



Curves showing the ability of the synthetic organic ester pump fluid, Octoil-S, to withstand exposure to the atmosphere while hot

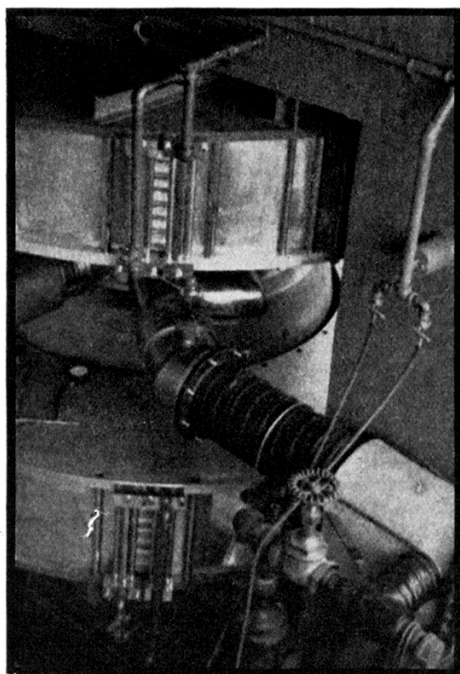
those employed by several manufacturers of medium power transmitting tubes.

Curve No. 1 is representative of an exhaust procedure followed with the 2-jet pump as illustrated. A rise in pressure was, of course, noted each time that bombardment of one of the electrodes took place. Because of the relatively high ca-

capacity of the pump employed and the fact that no trap with its corresponding high impedance was placed between the manifold and the high vacuum jet, the pressure was reduced very rapidly after each evolution of gas. Problems of ionization during the early stages in the tube were, therefore, practically eliminated.

In an effort to obtain the lowest possible pressures additional tubes of several types were evacuated with a 4-compartment, 3-jet fractionating pump and the connecting tube between the manifold and the pump maintained at 0°C with ice. Typical results of this work are shown by Curve No. 2 and it can be seen that the final pressure attainable was of the order of 1.5×10^{-6} mm.

Although usually the speed of exhaustion is limited by the diameter and length of the seal-off tube, in many cases considerably higher pumping speeds could well be used on the exhaust manifold itself. The more commonly employed mercury pumps rarely have speeds in excess of one to three liters per second at 10^{-4} mm. Assuming, therefore, a



Metal oil diffusion pump exhausting the cyclotron without the aid of a cold trap at M.I.T.

trap efficiency of 20 per cent, the available pumping speeds may well be below 0.2–0.6 liters per second.

The 4-compartment, 3-jet and 3-compartment, 2-jet pumps used during these tests have speeds of 15 liters per second and 12 liters per second respectively for all pressures below 10^{-4} mm. This capacity is adequate to handle several large tubes and it has been demonstrated that one such pump is capable of simultaneously exhausting ten nine-inch cathode-ray tubes.

Data is also given for the fore pressures normally encountered under average conditions. As can be seen, these readings offer an excellent indication as to the operation of the pump and the pressure in the system under exhaust.

These do not represent the best, or by any means the shortest, procedures which might be employed, as the tests were conducted merely to obtain a direct comparison between tubes exhausted with mercury pumps and those evacuated with the simple setup as shown using only air cooling.

Commercial tubes made according to both of the above procedures have been submitted to life test and gas current measured under recommended operating conditions. In all cases, the final products were found to be as good or better than tubes made with mercury pumps employing liquid air.

Stability of Pumping Fluid

For commercial applications of oil pumps the question of decomposition of the pumping fluid under adverse operating conditions must be considered. In employing the pumps for production work, there is always the possibility of exposing the fluid to atmospheric pressure because of breakage of a tube under exhaust, mishandling, etc. In order to demonstrate the stability of the newer low vapor pressure fluids, particularly Octoil and Octoil-S, a series of tests were made with the 4-compartment, 3-jet all glass fractionating pump.

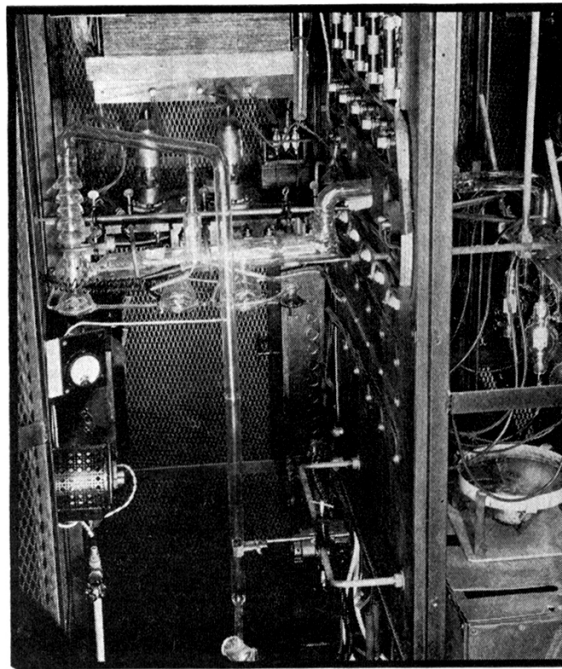
After the pump was operated for several hours, a study was made of the effect of exposing the hot oil to atmospheric pressure. The results of tests of this type are shown on page 34 and it can be seen that even when the heaters, which are immersed directly in the oil, were operating and the pump was exposed to atmospheric pressure for thirty seconds the same ultimate vacuum was attained. As a matter of fact, with this particular condition lower pressures were reached for any given time after pumping action began. In this case the fluid was sufficiently hot to permit pumping action during the initial stages of exhaust although decomposition was detected by a rise in fore pressure.

These tests indicate that these pure organic esters, which have been

selected from a large number of less suitable compounds, represent a very real advance in pump fluids. Although with all fluids decomposition may take place under conditions of high temperature and pressure, the products coming from the new esters are more volatile than the esters themselves and are easily removed by the fore pump. The compound remaining in the pump is a relatively pure substance essentially the same as the original oil except for the development of a slight color under adverse conditions.

Advantages

Providing sufficiently low fore pressures can be maintained at all times oil pumps may, in almost all cases, be advantageously substituted for mercury units in order to pro-



Three-stage glass fractionating pump used to exhaust Raytheon getterless transmitting tubes

duce economically the lowest pressures. It is also interesting to note that the capacity of any mechanical pump may be effectively increased many times in the lower pressure regions through the aid of so-called "oil boosters." Units of this type operate against high fore pressures, *i. e.* greater than 0.5 mm and yet they can be designed with capacities of hundreds or even thousands of liters per second. If, for example, a booster is operated with a fore pressure of 0.5 mm and the system under

exhaustion is at 5×10^{-4} mm, the combination of mechanical pump and booster permits an equivalent pumping speed of 1000 times that of the mechanical pump alone.

In considering the use of fractionating oil diffusion pumps the following factors are of interest:

A. Use of Cold Traps. Because of the extremely low vapor pressures of the newer organic fluids oil pumps may be operated without the aid of cold traps of any type. Pumping speeds are thus not impaired, the inconvenience of maintaining liquid air or solid carbon dioxide is eliminated and considerable saving can usually be effected.

B. Speed. Because of the higher pumping efficiency and the unique types of design which may be used with oil pumps, extremely high pumping speed may be obtained. In general, these are many times that of common mercury pumps.

C. Cooling. Because of the low vapor pressure materials which are employed with the newer oil pumps, those of glass construction operate very satisfactorily with air cooling. The larger metal units generally require water but the heat to be dissipated for a given speed is relatively low.

Other Applications

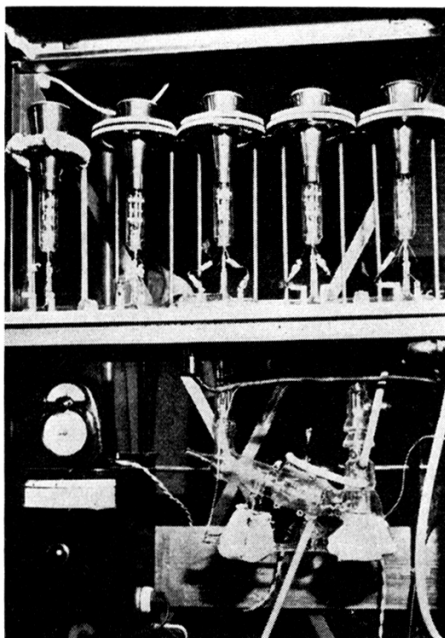
Although it has been demonstrated that fractionating oil pumps are particularly useful for the economical exhaustion of "getterless" transmitting tubes, there are many other electronic devices with which they may well be employed.

In spite of the many advances which have been made by the electronics industry in recent years, an improvement in vacuum technique appears to be peculiarly lacking. Exhaust schedules employed by different organizations in some cases differ from twenty minutes to four hours for the same type of tube. Obviously in the latter case, a substantial reduction in the manufacturing cost could be achieved by more appropriate design of exhaustion equipment and proper adherence to the fundamentals of high vacuum technology.

Television and cathode ray tubes. In the exhaustion of both cathode ray and, in general, all types of "image" tubes, the highest possible vacuum is generally desirable. Be-

cause of the relatively large volumes involved, the problems of outgassing must be more seriously considered and relatively high pumping speeds are desirable. Glass fractionating pumps are now being used for work of this type and pressures are being consistently reported which are as low or lower than those attainable with the best mercury pumps employing liquid air traps.

Receiving tubes. In the manufacture of receiving tubes with automatic machines, it is questionable



Glass fractionating pump used in the exhaustion of commercial television picture tubes

whether extremely low pressures are necessary or in some cases even desirable. Because of the small exhaust ports, extremely high exhaust speeds are quite impossible. Providing that adequate fore pressures can be maintained, there appears to be some justification for replacing the generally employed mercury pumps with metal oil boosters in order to make somewhat lower pressures possible and obtain savings in both water and power consumption.

It is interesting to contemplate the possibility of attaching a high capacity oil pump to a large volume, this in turn being maintained at a pressure of perhaps 10^{-5} mm. Connections could then be made from this volume to various ports on the machine and the large number of mercury units which are generally employed would be eliminated. In this way, extremely high effective pumping speed would always be

available at each exhaust position. The question of breakage with subsequent loss of vacuum at any exhaust position could be adequately taken care of with the aid of properly designed automatic check valves.

X-ray tubes. The problems of exhaust with both the x-ray and cathode-ray tubes are quite similar, although in the former case somewhat higher pressures may be tolerated. Through the use of even the smallest glass oil pumps adequate pumping speed and sufficiently low pressures may be attained.

Neon signs, etc. In the exhaustion of neon and other types of gas discharge tubes, extremely low pressures are rarely required. Nevertheless relatively small oil pumps are being used by many manufacturers because of the ease with which they permit the rapid exhaustion down to pressures of 10^{-4} or below. Here again, particularly with the present interest in large size fluorescent tubing, higher pumping speeds are of interest.

Continuously evacuated systems. At the present time there is considerable interest both in this country and abroad in the development of high power demountable transmitting and x-ray tubes. Although high pumping speeds are not generally required, reliability of operation is of utmost importance and the questions of maintenance and operating cost must be considered. Although the design of proper baffle arrangements to prevent back streaming of the oil vapor should receive serious consideration, through the use of fractionating oil pumps, cold traps may be eliminated and assurance given that the lowest possible pressures will be maintained at all times even over long periods of operation.

References

- (1) Gaede, *Ann. de. Physik*, 46, 357-392 (1915); *Z. fur tech Physik*, 4, 337 (1923).
- (2) Langmuir, *G. E. Rev.*, 1060 (1916); *J. Frank. Inst.*, 182, 719 (1916).
- (3) Dushman, *J. Frank Inst.*, 211, 689-750 (1931); Williams, *Phys. Rev.*, 7, 583 (1916); Moltan, *Z. fur tech Physik*, 7, 377, 452 (1926); Crawford, *Phys. Rev.*, 10, 557 (1917).
- (4) Burch, *Nature*, 122, 729 (1928); *Proc. Royal Soc. London*, 123, 271 (1929).
- (5) Hickman - Sanford, *R.S.I.*, 2, 140 (1930); U. S. Patent No. 1,857,508.
- (6) U. S. Patent 2,147,479; U. S. Patent 2,147,488; Kipp, *Phys. Rev.*, 9, 311 (1917); Baker, *Phys. Rev.*, 10, 6422 (1917); Volmer, *Ber. Deut. Chem. Gess.*, 52, 804 (1919); 804 (er.Phys.RBak ev.,648804(1919); Ebert, *Z. of Phys.*, 19, 3, 206 (1923).
- (7) Gaede, *Ann. de. Physik*, 46, 357 (1915).
- (8) Hickman, *J. Frank. Inst.*, 221, 215-235 (1936).
- (9) Lockenwitz, *R.S.I.*, 8, 322-323 (Sept. 1937); Malter and Marcovitz, *R.S.I.*, 9, 92-95 (March 1938).
- (10) U. S. Patents 2,080,421; U. S. Patent 2,150,676; U. S. Patent 2,153,189.

