

INDUSTRIAL HIGH VACUUM

By FRANK B. JEWETT JR.

THE development of any tool or technique is always of interest to technically trained men for its potential impact on their individual fields. Recent developments in high vacuum equipment and practice have indicated an ever-increasing sphere of commercial usage. Consequently, it is felt that a brief resume of the field may prove to be of considerable interest even to those not now involved in low-pressure work.

Industrial high vacuum may be defined as the use on a large scale of total pressures less than a few millimeters of mercury. Rarely do industrial applications require pressures less than 10^{-5} millimeters. In general, high vacuum represents to each industry the utilization of pressures lower by a large factor than that previously believed possible or practical.

Prior to 1940 industrial use of high vacuum was confined to a limited number of specialized applications despite the fact that very low pressures had been common in the laboratory for more than a generation. Up to that time, no commonly accepted standards or equipment and, probably more important, no large-scale demonstration that low pressure could effectively be used were available to the engineer seeking to use it in his process. The inevitable result was that where applications were made the design was usually done by men primarily interested in other phases of the work;

consequently, the vacuum equipment represented in each case pioneering work in high-vacuum engineering and technique. As might be expected, the results were of varied quality; but it is fair to say that all suffered from the lack of contributions by organizations whose primary business was the production of the best in engineering design, equipment and technique. Despite these handicaps, however, high vacuum was early used in the production of electronic tubes and electrical capacitors, later in the distillation of marine and vegetable oils for vitamin content, and in the small-scale dehydration of blood plasma. Much of its later success hinges on these early applications.

Industrial high vacuum as a field may be dated from about 1940, when the military needs of the impending war first began to make themselves felt. Its rapid development during the war years was, in a large measure, the result of demands made by the magnesium, penicillin, atom bomb, and other military projects. These programs not only fostered the development of rugged, dependable, large-scale equipment and demonstrated conclusively that low pressures, when properly applied, could be used economically on a commercial scale, but also brought into existence a large body of technical and business personnel familiar with the possibilities and limitations of high vacuum. Also extremely important was the growth during the war of several organizations specializing in the design, construction, and application of vacuum equipment. Whereas previously it was necessary to develop and construct all pumps and equipment for a particular job, now it is possible to secure catalogs of standard equipment and expert advice from any of several competent organizations. Now it is unnecessary for the process engineer to dilute his efforts by considerations of vacuum equipment design and construction.

VACUUM TECHNOLOGY

A brief description of some of the standard equipment or common practices of modern high-vacuum engineering may lend credence to the statement that engineering, fabrication techniques, and equipment are ready to handle any vacuum problem on whatever scale industry may pose. No longer need the development

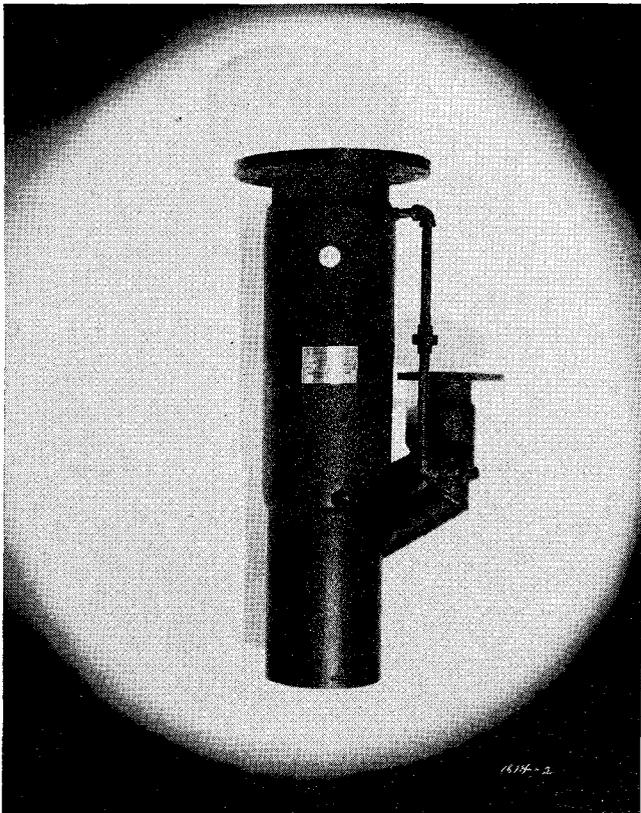
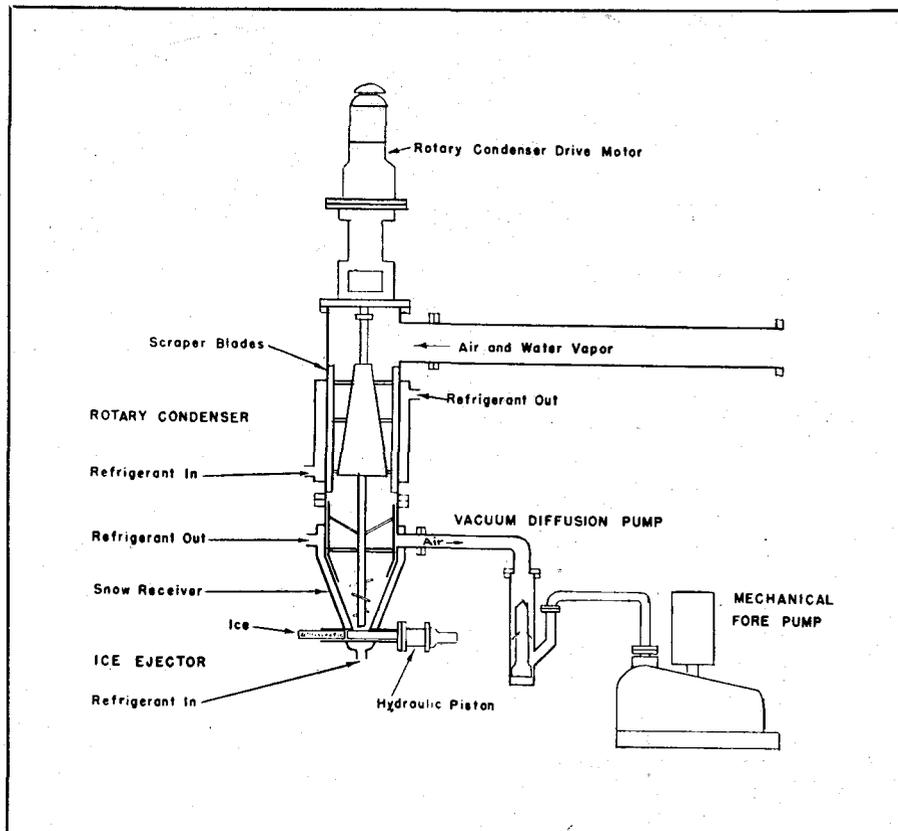


Fig. 1 Typical three-stage oil diffusion pump, 10-inch diameter inlet.

Fig. 2 Schematic diagram of Rotary Condenser unit for pumping water vapor at low pressures.



man discard a high-vacuum process merely because it involves low pressures on a large scale.

Diffusion pumps, similar to the 10-inch model illustrated in Fig. 1, up to 32 inches in diameter with speeds up to 30,000 cubic feet per minute at 0.1 micron* are standard items with several manufacturers. Electrically heated metal pumps have almost completely supplanted all other types. Diffusion pumps fall into two broad classifications, and each has its proper place: (1) The high-vacuum type usually consists of three umbrella stages in a cylindrical, water-cooled casing. It is designed to have its maximum speed at about 0.1 micron and blank off at 0.001 micron or less and to operate against a forepressure of 100 to 500 microns. This is the most common diffusion pump. (2) The booster type is designed to have its maximum speed between 10 and 500 microns and to blank off between 0.1 and 1.0 micron. It will operate against forepressures of 1000 microns or more.

It is interesting to note that diffusion pumps can be used either to produce low pressures or to reduce the number or size of mechanical pumps required to achieve a given speed at pressures in the micron range. No diffusion pump will exhaust to atmosphere directly, and so some sort of backing pump, either mechanical or steam ejector, must be provided.

With the exception of some conditions where very high forepressures are required, oil has replaced mercury as a pump fluid. Several types of oils make proper selection for particular applications possible. For instance dioctyl phthalate is useful where low back-streaming and high ultimate vacuum are required. It has good speed characteristics, but is quite heat-sensitive. Sili-

cone types are also useful for low-pressure work and are heat-stable, but have relatively low pumping speeds. Chlorinated hydrocarbons, while not useful for very low pressures, have excellent speed characteristics at about 0.1 micron and are reasonably stable.

The pumping of condensable gases like water vapor offers interesting problems to the vacuum engineer. When one considers that to move 10 pounds of water per hour at 100 microns requires a pumping speed of 25,000 cubic feet per minute, it is obvious that diffusion pumps are unsatisfactory. Mechanically refrigerated cold traps have been developed into efficient water-vapor pumps for the lower micron range. The rotary condenser (Fig. 2) is an interesting example. In this case refrigerant at -70° F. to -85° F. is circulated through the jacket. Water vapor condenses in the form of ice on the inner surface of the condenser and is continuously removed from that surface by a rotating cutter blade. The resulting snow is customarily removed from an ice-receiver pot at the end of the run, but may be continuously ejected to the atmosphere. By virtue of its cold surface temperature, this type of unit can be used to produce ultimate water-vapor pressures down to 1 micron,* and it has the advantage of always presenting an ice-free surface for maximum condensing efficiency not possessed by the static trap. Within limits, the pumping capacity of any given rotary condenser is dependent upon the tonnage of refrigeration applied to it, and obviously the pressure varies with the condensing temperature. More recent developments indicate that liquid absorber units using a fluid like lithium chloride may replace the rotary condenser on large installations because of lower operating cost. For pressures of 1000 microns or higher, multi-stage steam ejectors are more

*One millimeter of mercury equals 1000 microns. One micron equals 0.0000194 pounds per square inch absolute.

*Equilibrium temperature of ice at 1 micron approximately -100° F.

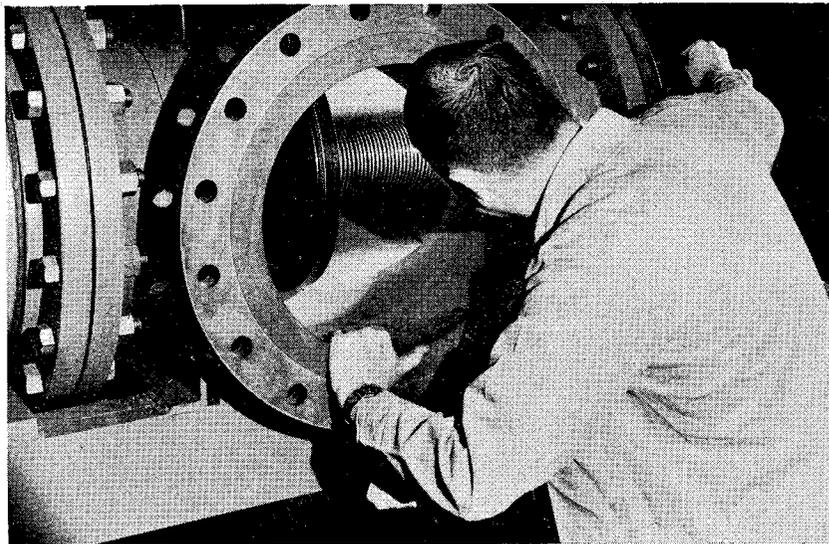


Fig. 3 Modern high-vacuum valve showing metallic bellows.

economical to use. Comparative operating costs for pumping water at 250 microns show 2.5 cents per pound for the rotary condenser, 1.5 cents per pound for liquid absorbers, and 25 cents per pound for steam ejectors.

Accurate vacuum measurement is difficult and is influenced by mixture and composition of gas, presence of condensable vapors, contamination, temperature, and location of gauge. Probably one of the greatest difficulties encountered by persons utilizing high vacuum is the discrepancy between gauge readings and the actual pressure in the system. This error can be many hundred per cent and often leads to dangerous misinterpretation of result. Many types of vacuum gauges are now on the market. Each has its field and its own peculiar limitations.

Vacuum measurement has often held an entirely different meaning for the engineer in industrial applications from what it has for the scientist in his laboratory. Where a repetitive process is involved, cycles are frequently worked out by trial and error using some form of gauge which gives consistent, but not necessarily accurate, results. Consequently, the comparison of operating conditions between two installations must always be preceded by a resolution of the methods of vacuum measurement. The proper use of new vacuum gauges will do much to eliminate these errors.

Representative of these developments is the Alpha-tron Gauge. It is actually an ionization gauge in which alpha particles emanating from a small radium source, rather than electrons from a hot filament, act as the ionizing agent. Not susceptible to filament failure, the gauge may be operated at atmospheric pressure and covers the range between 1 micron and 10 millimeters in three linear scales. For all practical purposes, it has an instantaneous response. Where water vapor is present, the instrument is of special value because of the small variations in calibration between water vapor and air.

A complete new chapter in valve requirements has been written by the demands of high vacuum where impedance at low pressures and inleakage are so important. All good vacuum valves must have exceptionally wide openings to reduce impedance. Generally they should be as short as possible. Sealing of the operating mechanism is accomplished either by a metal bellows (Fig. 3) or by some variation of a rubber gasket-vacuum grease seal.

Anyone familiar with vacuum equipment undoubtedly has visions of hours, days, or weeks spent immersed in castor oil, acetone, and various waxes and greases, while one searches for hard-to-find leaks. Of necessity, those whose business is the production of vacuum equipment have solved this problem partly by improved design and production methods and partly by vastly superior testing methods. The old technique of spark-testing glass tubing still lingers on, pressure-testing with soap bubbles has its place, and acetone or ether-spray-testing has changed only in that it is more frequently done with a radium-type ionization gauge. But by far the best method, the most accurate and quickest way of locating even minute leaks, involves the use of a mass spectrometer. A tracer gas such as helium diffuses through a leak and into a pre-set mass spectrometer tube. The ion current registers not only the leak but, with special care, the magnitude of the leak as well. Suffice it to say that in this manner leaks which would allow a volume of one cubic foot to show a pressure change of 0.1 micron per hour can be located within a small fraction of an inch.

From the brief summary presented above it is possible to visualize the tremendous progress made in the art of producing very low pressures. The refinements in vacuum equipment, the tools for producing vacuum, would, however, be meaningless by themselves. The end result of these developments must lie in commercially profitable operations if they are to be of interest and importance. Although new applications are constantly coming to the fore, some indication of the direction of motion of the whole field may be gained from areas already well explored.

LOW-PRESSURE DEHYDRATION

Among the larger vistas in which vacuum is used, dehydration deserves a major rating. Low-pressure dehydration is usually required for the drying of heat-sensitive materials. Such materials may be dried either to save shipping weight and make distribution more economical or to prevent decomposition and spoilage in the liquid form. The actual mechanism of low-pressure drying will apply to any particular product for one or more of the following reasons: (a) to reduce or eliminate thermal decomposition of the product during the

drying operation because of low equilibrium temperature, (b) to obtain a low final-moisture content, (c) to produce a lyophilic (easily rehydrated) structure in the dry product, or (d) to improve the appearance of the dry product.

Perhaps the most interesting dehydration operation from the engineering point of view is that of the orange juice plant at the Vacuum Foods Corporation at Plymouth, Florida. Briefly the plant (Fig. 4) operates as follows. Oranges are squeezed in a juicing plant at an average rate of 500 boxes per hour. The juice is then passed to either of two banks of falling-film concentrators, where approximately 86 per cent of the water (75 per cent of total mass) is removed by steam ejectors at a pressure of 12 millimeters. In this stage more than 2000 gallons of product per hour are handled. Subsequently a portion of the output is canned and marketed as a frozen orange juice concentrate, while the remainder is dried to a powder in the vertical dryers at a pressure of about 100 microns. The water is pumped by large rotary condensers. This stage is interesting in that the product is introduced as a liquid and removed as a powder without breaking the vacuum. Tests have shown that the mean retention of ascorbic acid in both concentrate and powder is more than 96 per cent of the fresh juice content. The size of this plant, probably one of the largest commercial vacuum installations in the world, may be judged by comparing the size of the man and the equipment.

EVAPORATION OF METALS AND SALTS

One of the best-known applications of high vacuum developed rapidly during the war. Starting with the deposition of one-quarter-wave-length films of magnesium fluoride and other substances on glass to reduce surface light reflection, this field has now expanded to include the deposition of metallic films on a wide variety of materials. A typical wartime optics coating installation depicted in Fig. 5, and a modern, high-speed

evaporating tank in Fig. 6. Machines have been developed for applying metal films to continuous rolls of plastics, paper, and other materials. These machines open up the whole electrical capacitor field where small, self-healing units can be constructed from zinc-coated paper.

By far the most interesting development, however, is the automatic metallizing unit (Fig. 7). This unit was designed to produce front-surface aluminum reflectors for the optical systems of television sets, but it is equally important for its revelation of what can be done with modern high-vacuum equipment. Clean pieces to be metallized approach the machine in jigs on a conveyor line from the left. They are automatically staged into the machine and moved from chamber to chamber until they reach the coating cell which remains constantly at a pressure of about 0.1 micron. Here aluminum, continuously evaporating from a controlled source, is deposited on the surface. The material is then staged out of the machine and back to the line. Complete controls and safety devices make the entire operation automatic, and labor is reduced to a minimum. Although this particular machine was designed for television applications, the general philosophy of the design will have a much wider application.

VACUUM METALLURGY

Vacuum metallurgy represents a field yet in its infancy but with unlimited possibilities. Four basic phenomena make high vacuum metallurgical processes possible. (1) High vacuum provides the only true inert atmosphere, and by its use metals may be protected during processing or heat treating. (2) At low pressures the boiling or subliming temperature of metals is reduced sometimes by as much as 1000° C. This phenomenon allows the purification of some metals by selective evaporation, the evaporation of metals for mirror work, and similar processes. (3) Operation in a vacuum tends to lower the reaction temperature by favoring any reaction

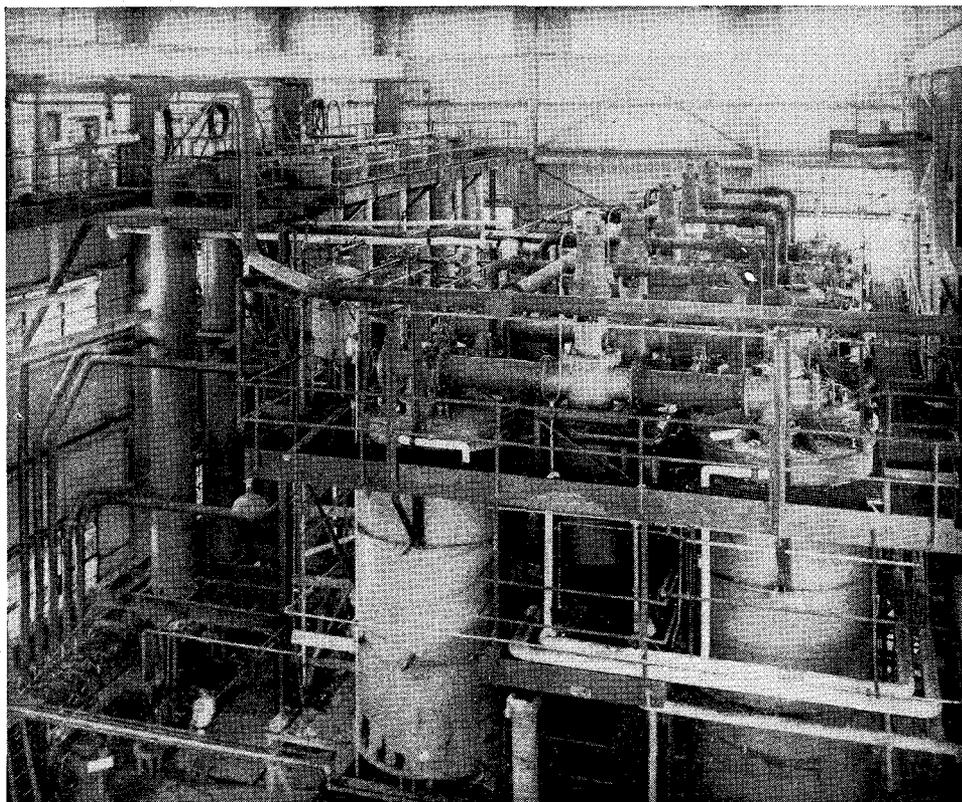


Fig. 4 Vacuum Foods Corporation drying plant for orange juice. Falling-film concentrators at left; two rows of powder dryers in foreground with rotary condensers between.

in which gas or volatile metal is produced from non-volatile constituents. (4) In many cases dissolved gases can be removed by melting the base metal in vacuum.

During the war these properties were put to use in the now-abandoned thermal-reduction process for producing magnesium. Although the economics of the process would not justify its use except for emergency operations it represents a typical application. Calcined dolomite and ferrosilicon are crushed, mixed, bricquetted, and charged into a vacuum retort. There, at a temperature of 1175°C . and at a pressure of 100 microns, magnesium vapor is condensed away from the other constituents in a very pure form.

In this reaction vacuum prevents the oxidation of magnesium, allows the metal to distil as rapidly as it is formed, and by lowering the boiling point of magnesium permits the reaction to proceed 500°C below the temperature required for reduction at atmospheric pressure. Similar processes are used for obtaining other metals in pure form.

The properties of gas-free metals are extremely interesting. In general they seem to show increased ductility, conductivity (both thermal and electrical), density, and permeability, and decreased hysteresis loss, blow holes, and gas evolution.

An interesting illustration of the use of vacuum furnaces lies in the hydrogen-atmosphere, heat-treating field. Despite the added cost of vacuum equipment, the process is quickly made more economical by virtue of the fact that only about 2 per cent of the customary amount of hydrogen is consumed. For use in this field, equipment has been developed capable of handling tons, not pounds, at temperatures up to 2000°C .

VACUUM DISTILLATION

Vacuum distillation has long been a field of great interest to the chemist and chemical engineer because low air pressures minimize oxidation, lower boiling points

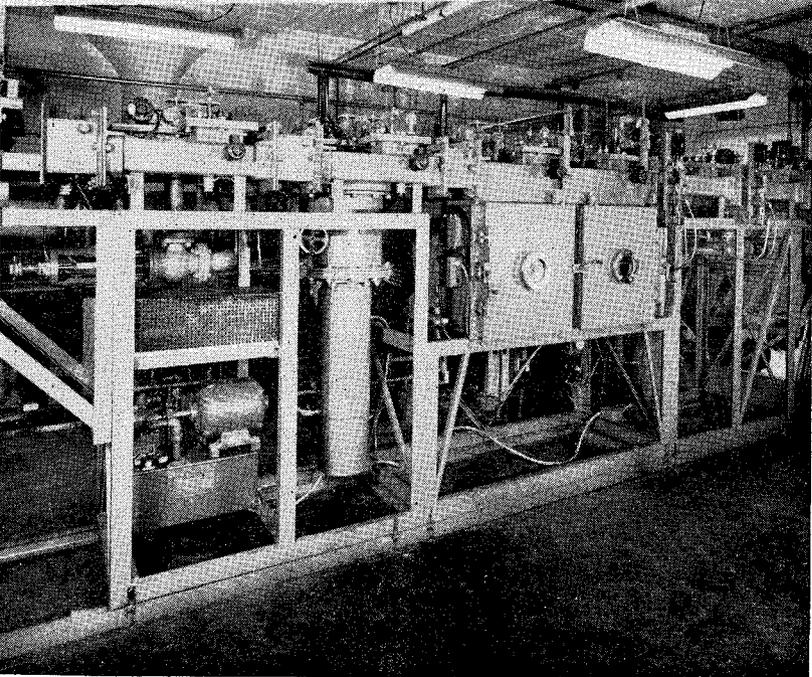
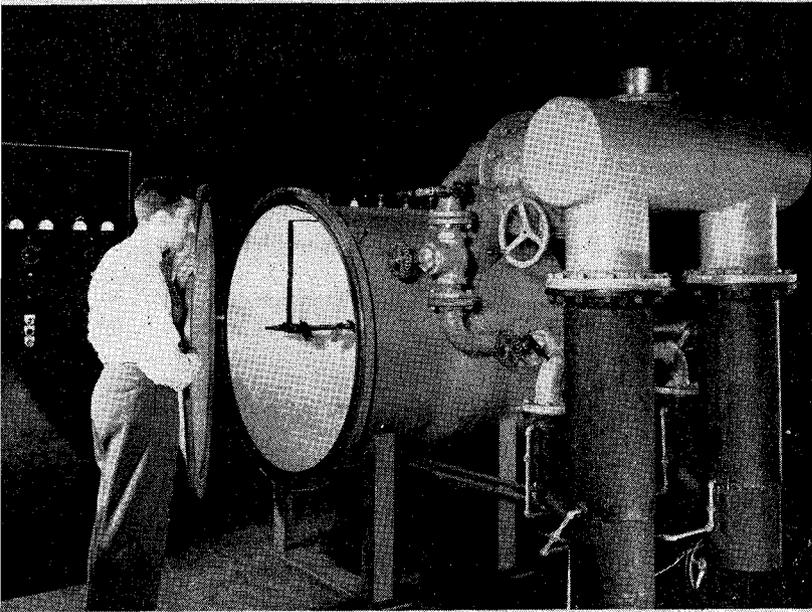
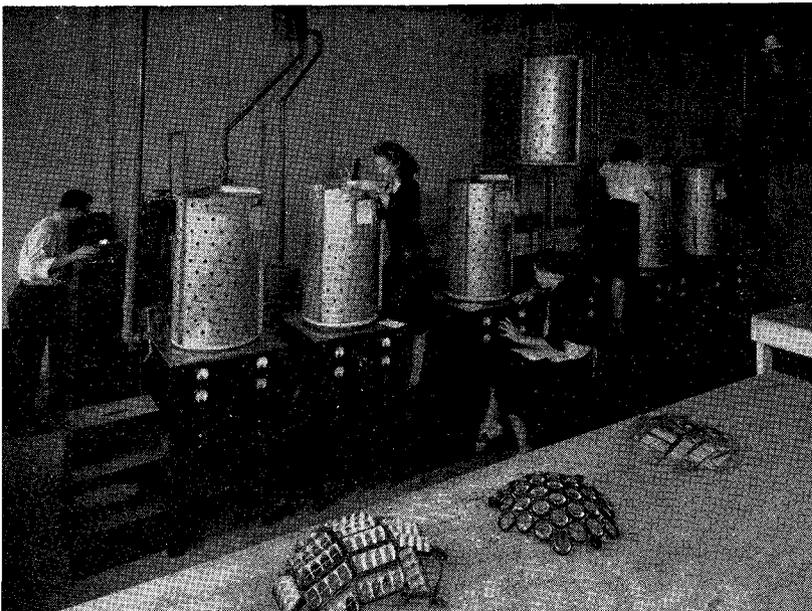
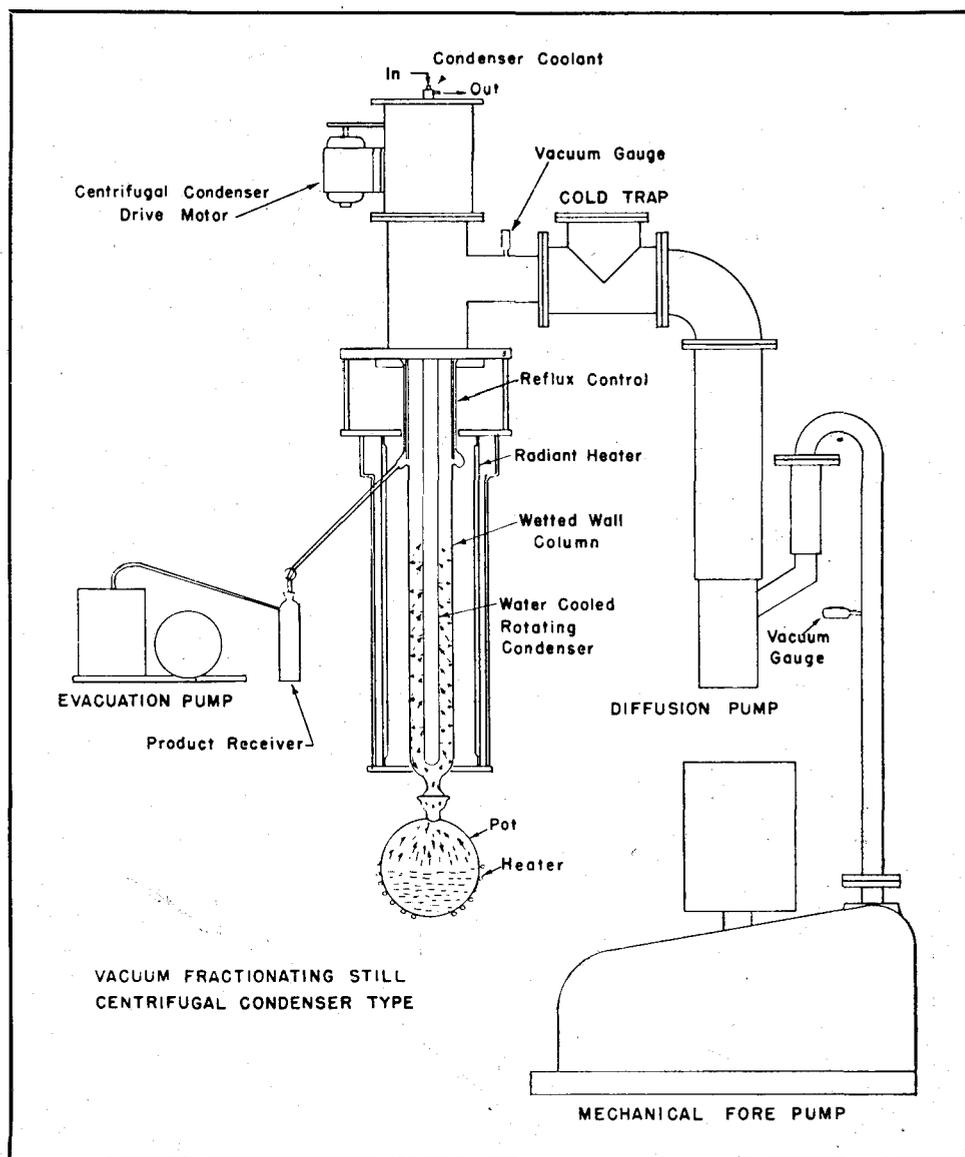


Fig. 5 UPPER Installation of bell jar units for deposition of low-reflection films on optics.

Fig. 6 CENTER Large high-speed tank unit for evaporation of both metals and salts.

Fig. 7 LOWER Machine for continuous production of front-surface mirrors. Glass enters from conveyor on left; mirrors returned to conveyor on right.

Fig. 8 Schematic diagram of Rotary Condenser Still having high fractionating power.



with consequent reduction or elimination of thermal decomposition, and remove the effect of residual gas on distillation rate.

Butyl stearate may be used as a typical example of boiling-point depression with reduced pressure. At 1.0 millimeter the boiling point is 155°C., while at 10 microns the boiling point is reduced 55 degrees to 100°C. The effect of residual gas pressure on distillation rate is typified by data reported by K. C. D. Hickman for dioctyl phthalate. With partial pressure of the distilland held constant at 1 micron, the distillation rate was five times as great with a residual air pressure of 0.2 micron as it was with a residual air pressure of 50 microns.

To the organic chemist no development could be more important than the rotary fractionating still developed by Dr. J. R. Bowman of the Mellon Institute (Fig. 8). This unit operates on a unique principle and has demonstrated fractionating powers as high as 100 theoretical plates. Heat applied to the bottom pot evaporates the material initially. The vapor then condenses

on the outer surface of the rotating condenser and is subsequently thrown by centrifugal force to the outer wall. The thin film on the outer wall is re-evaporated, and the cycle is repeated. By adjusting the heat ratios between wall and pot, the number of re-evaporations and the amount of fractionation versus through-put can be controlled. Obviously the still requires a very large amount of heat input when the material is re-evaporated several times. Although its actual commercial potentialities are not yet known, this type of still will at least provide the research chemist with a valuable new analytical tool. Standard laboratory models are now under construction.

In conclusion, high vacuum may be regarded as a realm that has demonstrated its importance in many industrial fields. Past work forms a firm basis of departure for new. Its future expansion is subject to conjecture, but continued development of equipment and more wide-spread understanding of its possibilities and limitations cannot but make for wider application.