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VACUUM PROBLEMS AND TECHNIQUES

By

C. E. Normand

Frank A. Knox

G. W. Monk

Alan J. Samuel

W. R. Perret

January, 1950

Clinton Engineer Works—
Tennessee Eastman Corporation
Oak Ridge, Tennessee

Technical Information Service, Oak Ridge, Tennessee

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AEC, Oak Ridge, Tenn., W46254

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C. E. Normand

Frank A. Knox

G. W. Monk

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PREFACE

Routine production of high vacuum in large systems on a scale never previously undertaken was required in the operation of the electromagnetic separation process. The problems encountered were a consequence of the magnitude of the undertaking and of the fact that the systems were designed and their component parts were chosen on the basis of little pilot-plant experience. Nevertheless, the equipment was found to be operable essentially as planned without major alterations. The paramount problems encountered were of an operational nature.

With this in mind, the first objective of this report is to describe the original vacuum equipment and evaluate its performance in view of the extensive experience gained.

The second objective is to present the most significant improvements in efficiency which resulted from changes in operating techniques. Since there was no previous experience for developing such techniques, it is felt that the "know how" gained represents a considerable advancement in the field of industrial high vacuum.

The third objective of this report is to bring together in an orderly fashion information which, if at all available, is widely spread throughout the literature and is not usually in a form directly applicable to the solution of vacuum problems. New and special designs for certain component parts of the equipment are appropriately treated.

C. E. Normand

January, 1950

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Chapter 1

PRODUCTION-PLANT VACUUM SYSTEMS

By C. E. Normand

1. INTRODUCTION

Operation of the plant for the separation of uranium isotopes by the electromagnetic process required the routine production of high vacuum in large systems on a scale never previously undertaken. The problems met in such large-scale high-vacuum operation arose in part from the magnitude of the undertaking, and in part from the fact that the systems were designed and their component parts were chosen or developed on the basis of very limited experience over a comparatively short period of time. Much of this experience was gained in connection with the developmental work carried out at the Radiation Laboratory of the University of California at Berkeley, and is covered in that laboratory's reports. It should be realized, however, that the initial units of the plant were designed, constructed, and put into operation without benefit of pilot-plant experience in the true sense. This being the case, the fact that operating experience has indicated the desirability of altering certain details of the original systems and equipment is much less surprising than the fact that the systems were operable essentially as planned and without major alterations.

Improvements that have been made fall generally into one or more of the following categories:

1. Changes initiated to eliminate or minimize operational errors.
2. Changes made to improve performance of existing equipment.
3. Introduction of equipment that was unknown or unproved at the time of designing the system.
4. Alteration and redesign of original equipment to remedy major or minor defects.

5. Changes made for economy and to avoid material shortages.

Less tangible, but not less significant than the improvements in equipment and materials, has been the development of techniques applicable to the operation of large-scale high-vacuum systems. These involve not only improvements in techniques applicable to the operation of any large all-metal vacuum system, but also procedures that apply particularly to multiple-unit systems which must be carried through recurrent cycles of operation and reevacuation on a more or less rigorously fixed schedule.

Since it is the object of this volume to present the experience of the operating company in so far as the vacuum phase of its work is concerned, developments both in equipment and techniques will be treated.

As far as possible only those problems arising, and developments undertaken, during the course of plant operation will be dealt with. Considerations that determined the types of systems and equipment provided in various parts of the plant are presented in the reports by the UCRL and by Stone & Webster Engineering Corporation. It is, however, the belief of the operating company that the vacuum phase of its work would have presented much more serious, and possibly insuperable, problems but for the excellence of design and construction of equipment and systems.

2. TYPES OF VACUUM SYSTEMS

As eventually built, the Y-12 plant makes use of three types of production vacuum systems and numerous auxiliary and servicing systems. The three types of production systems may be designated as the "Alpha I," "Beta," and "Alpha II" types. Although certain features are common to all three types of systems, and although minor variations occur within systems of each type, a familiarity with a typical system of each type should serve as an adequate basis for examining the various developments that have occurred during the course of plant operation.

Since the auxiliary systems utilize, in large measure, equipment and principles of design that are standard in the various production vacuum systems, any discussion of these will, likewise, be simplified by familiarity with the standard production systems.

In all three types of vacuum system the fundamental production unit is the individual "process tank" or "bin." It is in this tank, placed in a suitable magnetic field, that the ion beam is formed and the constituent ions are separated and collected. Since operation is cyclic, requiring a complete shutdown of each unit after a period of operation

for the removal of collected material and recharging, it is necessary that provision be made for evacuating and maintaining the vacuum in any tank independent of all others. Thus each tank must be provided with individual diffusion pumps that are capable of producing and maintaining operating pressure. A single mechanical pump may be supplied for backing the diffusion pumps on each tank or, if the operating cycle is sufficiently long, the diffusion pumps of two or more adjacent tanks may be connected to a common header pumped by one or more mechanical pumps. Such ganging of diffusion pumps on a common backing or finishing header gives rise to a secondary unit of the vacuum system, the finishing-header section.

During the period of evacuating a tank from atmospheric pressure to a pressure at which diffusion pumping becomes effective, high mechanical-pumping speed is required. This constitutes such a small part of the total operating cycle, however, that there is hardly any choice but to gang a number of tanks on a common roughing header served by one or more large-capacity mechanical pumps. All those tanks served by a common roughing header constitute a third natural division of the total vacuum system, the roughing-header section.

Finally, the complete production vacuum system is considered as comprising all those units and sections associated with all the tanks of one magnet or track, since the track is the natural operating unit of the plant.

In addition to the components mentioned above — tanks, diffusion pumps, mechanical pumps, and headers — numerous valves, cold traps, and instruments are necessary parts of each vacuum system.

2.1 Alpha I Type Vacuum System. Original plans called for the construction of five tracks of the type designated as "Alpha I." These tracks were to be housed two in the first, two in the second, and one in the third building to be constructed. Identical vacuum systems were planned for these five tracks; however, certain alterations indicated by experience in operating the first tracks completed were incorporated in later tracks.

The vacuum system associated with a typical Alpha I tank is shown schematically in Fig. 1.1. The arrangement of valves allowing the tank to be let down to air, roughed down, or pumped by the diffusion pumps is clearly shown. Also shown is the crossover valve allowing the roughing pump to be used for backing the diffusion pumps during early stages of diffusion pumping.

The grouping of tanks on headers and the provisions for mechanical pumping and vapor trapping are shown in the flow diagrams, Fig. 1.2.

Fig. 1.1 — Alpha I process tank and diffusion pump.

This diagram covers one quadrant of the track that comprises 96 process tanks.

Plans for the fifth Alpha I track were so altered during the course of earlier construction that it was finally completed as a hybrid track having the magnet, tanks, and vacuum system of an Alpha I track but operating with four beams per tank as in the later Alpha II units. This fact may be ignored for the present except for pointing out that the vacuum requirements arising from four-beam operation were more severe than in the earlier Alpha I tracks.

2.2 Beta Type Vacuum System. Concurrent with the designing and construction of the Alpha I buildings, work was proceeding on two Beta, or refining, buildings. Each of these buildings housed two tracks of 36 tanks each. Because of the smaller size of the tanks, the shorter operating cycle, and greater emphasis on material recovery in the refining stage of processing, the Beta-plant vacuum requirements differ from those in the Alpha process. These differences are reflected in the higher ratio of pumping capacity to volume and in the more effective high-vacuum vapor trapping provided in the Beta system. Figures 1.3 and 1.4 show a Beta tank with its associated vacuum system and a flow diagram of the roughing and finishing system of a section of a track.

With the expansion of the electromagnetic separation and gaseous diffusion programs, two additional Beta buildings were required. The vacuum systems in these buildings incorporate improvements based on experience in operating the earlier systems but mark no radical departure from the original design.

2.3 Alpha II Type Vacuum System. The Alpha II plant, consisting of four tracks of 96 tanks each housed in two buildings, was designed after considerable experience had been gained operating the earlier Alpha I tracks. The changes made in the vacuum system are due in part to a clearer firsthand knowledge of vacuum needs of the process and in part to the anticipated need of even greater pumping capacity in the Alpha II plant than in the Alpha I plant. This expected need of greater pumping capacity in the Alpha II system results from the fact that four rather than two ion beams per tank are maintained.

An Alpha II tank and its diffusion pumps are shown in Fig. 1.5. A flow diagram of a section of an Alpha II track showing mechanical pumps, headers, and valving arrangement is given in Fig. 1.6.

3. VACUUM REQUIREMENTS

In its construction and operation there are certain basic demands that each type of production vacuum system must meet:

Fig. 1.3—Beta tank and pump assembly.

Fig. 1.4—Early Beta vacuum system flow diagram.

1. The system must produce and maintain a pressure suitable for sustained operation. Pressures between 10^{-5} and 5×10^{-5} mm Hg may be considered normal operating pressures. Ion scattering and electric drains become excessive at pressures higher than this, and beam interaction is likely to occur at lower pressures.

Fig. 1.5 — Alpha II tank and diffusion pump assembly.

2. Starting with the system at atmospheric pressure, the average time required to establish operating conditions must be a small part of the total operating cycle. In the Alpha I processes in which the operating cycle is ten to fourteen days, the allowable start-up time is longer than in the Beta process in which the operating cycle is only two to three days. Obviously the time required for bringing a given system into operation is influenced by many factors. Some factors, such as the total volume to be evacuated and the pumping speeds of roughing and finishing pumps, are definite and determinate; others, such as leak troubles and extent and rate of outgassing of the system,

Fig. 1.6—Alpha II vacuum system flow diagram.

are highly indeterminate except as statistical averages. As operating techniques, and to some extent equipment, have been improved, operation at 90 per cent innage or better has been attained in both Alpha and Beta processes.

3. The system must give continuous service, free from interruption and proof against operational errors to the highest possible degree.

4. The system must be designed and operated for optimum production. Under wartime conditions this meant maximum production with little regard to economy. Changes that would obviously effect economies were not undertaken if they were likely to reduce output. Since the end of the war, however, it has become possible to consider overall economy of production, and some changes that reduce output appear to be justified because of a proportionately greater reduction in operating cost.

4. TYPE OF CONSTRUCTION

All production vacuum systems were of welded-steel construction. Flange and gasket joints and closures were used as conditions demanded. Brazed, soldered, or threaded joints were used in making some small connections, but arc welding was the principal method of fabrication.

Component parts such as tanks, diffusion pumps, valves, traps, and piping sections were tested for leaks by the manufacturer according to specifications of the contractor. The results were surprisingly good and very few leaks were found in these pretested parts. This indicates that for vacuum equipment of this type pretesting under adequate specifications provides effective proof of vacuum-worthiness. Also, such welded construction is free from injury due to strains that arise in the course of shipping and normal handling.

Welds and joints made in the field were pressure-tested using soap solution, and as a final test for acceptance each unit of a system was reduced to working pressure. Naturally in systems as large and complex as those dealt with here there were vacuum troubles in the start-up period of each track. Time was consumed looking for leaks that did not exist, and real leaks defied detection for days on end. That such problems were far from insuperable is clearly shown by the fact that a whole track could be brought into production within a few weeks after the completion of construction.

Once a system has been rendered vacuum-worthy little difficulty is experienced because of leaks at permanent welds or joints. A few weld cracks due to strain have occurred, and a few weld leaks have

appeared where no appreciable leak previously existed. It would be remarkable if this were not the case in a system containing a total of several miles of weld. On the whole, however, week after week and month after month units remain tight and leaks occur only at points where seals have been manipulated or where wear has occurred in the process of operation.

It should be clearly recognized at this point that it is possible to obtain and maintain working pressures in systems such as these, and to arrive at such pressures without too much difficulty on a production schedule, owing to (1) the very moderate pressure requirements and (2) the high pumping speed provided.

If lower pressures were required, or if less pumping speed were available, the whole vacuum problem would be considerably more difficult. Leaks that are now negligible would have to be found and eliminated, probably requiring more sensitive leak detection methods. Outgassing would remain prohibitive for a longer time, and pumps capable of lower base pressures than those now used might have to be substituted.

5. MECHANICAL PUMPS

5.1 Types and Performance. Kinney high-vacuum pumps were chosen for mechanical pumping in all production and auxiliary vacuum systems.

At one time several type-PR Beach-Russ pumps of 375 cfm capacity were installed in each of several Alpha vacuum systems. The resulting diversity of equipment proved troublesome to production personnel, and, consequently, these pumps were replaced by Kinney pumps when they became available. The Beach-Russ pumps were transferred from production systems to vacuum test docks, where uniformity of equipment was of less importance, and served quite satisfactorily in this capacity.

Figures 1.7 and 1.8 show the operating principles of Kinney and Beach-Russ pumps, respectively, and Table 1.1 lists significant information relative to the various sizes of these pumps used in the plant.

After extensive overhaul, and occasionally during operation, it is necessary to make certain simple performance tests on mechanical vacuum pumps. The tests commonly made are (1) blanked-off pressure attained and (2) pumping speed at one or more intake pressures.

The blanked-off pressure is usually measured with a McLeod gauge and is a function of the oil-metering valve setting. With proper oil

adjustment a blanked-off pressure of $10 \mu \text{ Hg}$ or less is usually considered acceptable. Higher blanked-off pressures are frequently caused by water in the oil. If high pressure persists after several changes of oil, the cause may be (1) air leaks, (2) closed oil line, (3) defective valve, or (4) some other mechanical defect.

Fig. 1.7—Kinney vacuum pump.

Pumping speed is determined by noting the pressure rise produced by the introduction of an air leak of known rate.

$$\text{Pumping speed} = \frac{\text{leak rate} \times \text{atmospheric pressure}}{\text{pressure elevation}}$$

where speed is in liters per second, leak rate is in milliliters per second, atmospheric pressure is in millimeters of mercury, and

pressure elevation is in microns of mercury. If the metered leak is adjustable to a range of values, pumping speeds over a range of pressures may be determined. In the case of pumps in service it is fre-

Fig. 1.8—Beach-Russ vacuum pump.

quently sufficient to determine the pumping speed at one significant pressure. Pumping speed at any pressure is also, to some extent, a function of the rate of oil flow, a higher flow rate being required for maximum speed at higher pressure.

In adjusting oil-flow rate for minimum blanked-off pressure or maximum pumping speed it is convenient to use a Pirani or thermocouple gauge, because of their continuous reading, rather than a McLeod gauge. Pressures indicated by these gauges are usually somewhat higher than corresponding McLeod gauge pressures unless they have been calibrated in an atmosphere similar to that existing above the mechanical pump.

Figure 1.9 is a typical Kinney-pump volumetric-efficiency curve showing the ratio of pumping speed to theoretical displacement throughout the working pressure range. Tests made on pumps of various sizes gave results that were in substantial agreement with the values shown here provided the pumps were in good condition and properly adjusted.

Table 1.1 — Mechanical Vacuum-pump Data

Pump type	Model	Normal speed,* rpm	Theoretical displacement at normal speed, cfm	Required motor hp	Inlet pipe size, in.
Kinney	VSD-556	430	12.47	0.5	1¼
Kinney	VSD-778	350	26.2	1.5	1¼
Kinney	VSD-8811	330	43.0	2	1½
Kinney	DVD-8810	430	105.2	5	2
Kinney	DVD-12814	400	210	10	5
Kinney	DVD-14918	350	303	15	6
Beach-Russ	RP-D-375	200	375	15	6

*Later bulletins from the Kinney Manufacturing Company showed that their pumps were being run at slightly higher speeds with resulting proportionate increase of 3 to 9 per cent in theoretical displacement.

All sizes of Kinneys are capable, under optimum conditions, of attaining blanked-off pressures of 10 μ Hg or better. Pressures of 5 μ Hg are not unusual. Common causes of high base pressure are: (1) contamination of the sealing oil, especially with water; (2) improper oil-flow adjustment; and (3) high temperature, especially in the case of the small air-cooled types of pumps.

It should also be noted that, because of low volumetric efficiency at low pressures, a comparatively low rate of leakage or outgassing in a system being evacuated will result in a considerable elevation of the base pressure.

5.2 Operational and Servicing Requirements. The rate of flow of sealing oil is the only commonly required adjustment in an operating pump. This adjustment is not very critical but may be quite different depending on the quantity being optimized. The flow rate for minimum blank-off pressure is less than for maximum pumping speed at some higher pressure. Also, the metering-valve setting for optimum performance varies somewhat from one pump to another.

Provision is made for adjustment of valve tension on all but the smallest size of these pumps. Since this adjustment is not critical over a wide range of tensions, however, it is seldom made except in cases of major overhaul or defective valve springs.

Fig. 1.9— Typical volumetric efficiency curve for Kinney vacuum pump.

Pump sealing oil is subject to contamination, especially by water, chlorine, hydrochloric acid, and perhaps other chlorides, and must be replaced when this contamination is sufficient to affect pump performance adversely. How often such a change is required depends on the rate at which contaminants enter the pump. More water vapor is

present during roughing and bake-out than during later operation, whereas chlorine is more abundant during operation.

Three distinct effects of oil contamination are observed as follows:

1. High base pressure. This appears to be due largely to the presence of water in the oil. A simple change of oil is usually effective, though in cases of severe contamination two or more changes may be required for complete restoration of optimum performance.

2. Collection of ferric chloride. This is a cumulative effect resulting, after a year or more of operation, in the partial or total plugging of oil feed and drain lines by an accumulation of ferric chloride crystals, and requiring the removal and cleaning of these lines and the separator tank. This ferric chloride apparently comes from the main vacuum system and not from corrosion of the pump parts since pumps show only slight evidence of corrosion after a year or more of service.

3. "Freezing" of pumps. The occasional freezing or sticking of pumps has presented one of the most persistent problems encountered in mechanical pump operation. Such freezing results from the formation of varnishlike films on the bearing surfaces of shafts, cams, and pistons and is usually associated with a high degree of chlorination of the pump oil. The seriousness of this problem lies in the outage and man-hours required for complete dismantling and reservicing of the pump that has frozen. The pump seems to suffer no mechanical injury other than that which may result from the forceful methods that are required for separating the frozen parts.

A survey of plant-wide experience with this difficulty indicates that pump freezing is more prevalent in Alpha than in Beta type systems and that its occurrence has been somewhat epidemic. Each track appears to have experienced a maximum of pump freezing a month or two after start-up. The frequency of occurrence then decreased steadily with time. The large (303 cfm) pumps appear to be wholly free from this trouble.

Although no complete solution to the freezing problem has been found, progress in reducing the frequency of occurrence has been made along several lines. Operating the pumps continuously, since most cases of freezing occur when the pump is stopped, has been partially effective. Frequent changing of the oil to minimize chlorination and the choice of an oil producing a minimum of wax and sludge have also helped.

A number of high-grade turbine-type lubricating oils have proved satisfactory as pump oils, except that they possibly contribute to pump freezing. Socony vacuum-pump oil, Texaco URSA P-10 (Navy symbol 2110), and Gulf Crest C vacuum-pump oil have had extensive

plant use at one time or another. Of other oils tested experimentally, a few are demonstrably poorer than these, none is clearly better, and a number appear about equally good. An oil of SAE weight 10 or 20 is preferred, but lighter or heavier oils may be used. Since pump oil is repeatedly purified and reused, oil containing essential additives that may be removed in the purification process should be avoided.

Mechanical pumps of the production vacuum systems are connected in groups into a number of oil-reclaiming systems. Each of these reclaiming systems consists of an overhead storage tank for clean oil, a sump into which used oil from the pumps is drained, a Hilco oil reclaiming unit, and the necessary connecting pipes and valves. Oil from the sump is pumped to the reclaimer, where it is preheated and passed, under vacuum, through a filter of absorbent clay and paper onto the surface of a heated vaporizer and then to the clean oil storage tank. Usual plant practice has been to operate the oil-reclaiming systems on a batch basis. Used oil is drained from any pump, and reclaimed oil is introduced as required by established procedure or pump performance. The reclaiming unit is operated only as required to maintain an adequate supply of clean oil in the storage tank. Continuous flow operation was carried out for some time on one reclaiming system without showing any clear-cut operating advantage.

The number of oil purifying units required in actual plant operation proved to be less than anticipated in the earliest plant designs. Consequently some of the units installed in the first tracks were later removed and used in subsequent construction. As finally operated, four units of 50 gal/hr capacity serve the forty-eight 303-cfm pumps of each Alpha I track. Likewise, four units operate in each Beta track. Each Alpha II track has twelve units serving sixteen 303-cfm pumps and forty-eight 105-cfm pumps. Since batch reclamation is standard practice, most present installations represent considerable surplus capacity.

Various schedules and procedures for oil changing have been followed at different times and in different parts of the plant, but no one procedure stands out as definitely superior to all others. Typical practice at the time when the plant was in full operation was to change oil in all finishing pumps on a two-day schedule; to change oil in roughing pumps when the dry-ice trap was changed, that is, about once a week; and to change oil in any pump when poor performance indicated the necessity.

Recent tests have demonstrated that the frequency of oil changes can be greatly reduced and the use of the dry-ice trap to protect the pump oil from vapors can be completely eliminated. These improvements may be effected by operating with a stream of dry air passing

through the valve chamber of the pump and out through the separator tank. Dry air at the rate of about $\frac{1}{2}$ cfm for each 180-cfm capacity for finishing pumps, and at a considerably higher rate for roughing pumps, is introduced into the valve chamber of the pump and passed out through the exhaust line. Under these conditions the average oil-change cycle has been extended to a week or more. At the time of this writing all Beta tracks were operating with air-drying of the pump oil.

Two problems are presented by the elimination of dry ice in the above procedure:

1. The possible loss of U 235 previously recovered from the dry-ice trap and now carried out, at least in part, by the exhaust air.
2. Overloading of the backing pumps during the bake-out period caused by loss of the pumping action of the trap for condensable vapors.

Tests show that material carried out the exhaust can be recovered by suitable exhaust scrubbers. It is questionable, however, whether the amount of material recoverable is sufficient to justify its recovery. It is found also that the unrefrigerated trap is almost as effective as the refrigerated trap in the collection of U 235. To compensate for the loss of pumping action of cold traps for condensable vapors it is necessary to use the large-capacity roughing pumps for backing the diffusion pumps during the bake-out period, and to this end roughing-to-finishing cross ties are being installed wherever necessary. Once this period of heavy outgassing is past, the small finishing pumps have adequate capacity to carry the load.

The mechanical servicing of pumps has been handled in various ways at different times and in different parts of the plant. Initially, servicing of the pumps in each building was carried out by crews in that building. In some cases all pumps were removed from service and completely overhauled on a six-month schedule, routine checks and minor repairs being made in the meantime on pumps in position. In other buildings overhaul was given only as performance demanded. Typical of the repairs required during this period is the condensed tabulation on the following page of all pump repairs in one Alpha I building during the first three months of 1945. At that time this building had been in operation almost a year.

It will be noted that most of these repairs are of a minor nature, and the total of major repairs is quite low—five valve replacements, three major parts replacements, and five complete overhauls.

Mechanical servicing of vacuum pumps by individual buildings proved unsatisfactory in several respects, but principally because

the servicing demands per building were not sufficient to justify the maintaining of highly skilled specially trained crews for this work. Early in 1945 a department was organized for handling all major servicing of mechanical pumps and oil purifiers. Placed in a central location and provided with adequate shop facilities, test equipment, and pickup and delivery services, this specialized group has greatly improved the mechanical servicing situation. Minor repairs on pumps

Pump Repairs, January to March, 1945

Type of repair	No. of times
303-cfm pumps (48)	
By-pass valve repacked or replaced	8
Metering valve repaired or replaced	9
Shaft gland repacked	8
Sight glass repaired or replaced	10
Solenoid valve replaced	1
Three-way oil valve repaired	1
Poppet valve replaced	2
Shaft replaced	2
Cam replaced	1
Routine overhaul	2
Smaller pumps	
Feather valve replaced	3
Pump freed after locking	2
Routine overhaul	1

in service are still made by the operating crews in the individual buildings. Pumps requiring major repairs or overhaul go to the central servicing shop. All mechanical pumps and oil reclaiming units are now being overhauled on a twelve-month schedule. Parts showing wear that might affect operation during the following year are repaired or replaced. Each unit is thoroughly cleaned, tested, and returned to the building from which it came. The shop operates 24 hr per day, seven days per week, and with present curtailed plant operation approximately one pump or reclaimer is serviced per day on regular schedule plus whatever incidental servicing may be required.

5.3 Experience with Multiple-tank Header Systems. Experience in operating the first Alpha I tracks indicated clearly that for greater efficiency it would be desirable to (1) provide roughing pumps of greater capacity and (2) increase the number of header sections, thereby reducing the number of tanks operating on a single header.

These changes were made in the earlier tracks and were incorporated in the initial construction of later tracks.

The substitution of larger roughing pumps was brought about largely by recognition of the fact that the actual average roughing time, under operating conditions, exceeded the theoretical roughing time by a larger factor than had been anticipated in designing the system. This discrepancy existed, and still exists, in spite of the fact that individual instances are observed in which roughing is accomplished in essentially theoretical time. The difficulty seems to be that this happens much too seldom. Outgassing and other extraneous causes usually extend the roughing time well beyond that required for the simple removal of free air from a tight system. Theoretically, an Alpha I tank, having a volume of 8,800 liters, can be evacuated from atmospheric pressure to 50 μ Hg in about 40 min if a 105-cfm pump is used, and in about 14 min if a 303-cfm pump is used. With the larger pump, pump-down time of 18 min or less is occasionally obtained; however, the average time required, even after deletion of those very long pump-down times due to leaks or operational errors, is between 1 and 2 hr. With the smaller roughing pumps the situation is even worse, and by substituting 303-cfm pumps for all roughing service a small but cumulative reduction in interrupt outage is realized.

Another argument for the large-capacity roughing pumps is their ability to serve for backing the diffusion pumps through the cross-over line during the early stages of diffusion pumping and tank bake-out. In this way the danger of overloading a finishing header into which a number of operating tanks are working is reduced.

In the Alpha I vacuum system as originally designed there were eight roughing header sections. Each section served twelve tanks and was pumped by one 105-cfm Kinney. There were four finishing header sections each serving 24 tanks and being pumped by six 303-cfm Kinneys. Adjacent sections of both roughing and finishing headers were joined by sectionalizing valves permitting interchange of pumping between sections should occasion require. With the substitution of 303-cfm roughing pumps, the roughing systems were quite operable. The 24 tank finishing sections were objectionable, however, because of the danger of interruption of operation in a large number of tanks as a result of vacuum failure in one tank or of a single error in vacuum operation. Quite early, therefore, finishing headers were further sectionalized into twelve sections of eight tanks each. At the same time that this was done, four additional 303-cfm roughing pumps were provided, and the roughing system was similarly divided into twelve sections of eight tanks each. Finishing header sections, in groups of

three, were connected by sectionalizing valves, thus allowing the operating tanks on any header to be carried by the pumps on the neighboring headers during periods of servicing individual finishing pumps or dry-ice traps. Sectionalizing valves between adjacent sections of the roughing header were not considered essential since roughing-pump demands are intermittent. Routine roughing-pump and cold-trap servicing can be so scheduled as not to conflict with roughing requirements, and failure of a roughing pump affects only those few tanks that might be waiting for roughing while pump repair or replacement is made.

The flow diagram, Fig. 1.2, shows the pump and header arrangement in one quadrant of a typical Alpha I track as eventually modified.

As shown in Fig. 1.4, plans for the first four Beta tracks called for six finishing header sections and six roughing header sections each serving six tanks. Both finishing and roughing header sections were joined in groups of three by sectionalizing valves. Each roughing header section was pumped by a 105-cfm Kinney, and each finishing header section by a 303-cfm pump. Because of the smaller size of Beta tanks and units, pumping requirements were not so severe in Beta as in Alpha systems. More than neutralizing this advantage, however, was the short Beta operating cycle, which made it imperative that interrun pump-down time be much less than that allowed in Alpha. No significant changes were found necessary in these systems, and at time of writing they were being operated as designed.

Changes that were considered desirable, though not necessary for operation, were incorporated in the design of the last four Beta tracks. These changes, as shown in Fig. 1 10, were:

1. Substitution of a 303-cfm roughing pump for the 105-cfm pump on each six-tank roughing section, thus realizing a small but consistent reduction in roughing time.
2. The elimination of finishing headers by providing one 43-cfm pump for backing the diffusion pumps of each tank. This change was made in order that each tank, once brought into operation, might be independent of all other tanks. Thus, the ever-present danger of having production interrupted in six units because of a single vacuum failure or operating error was eliminated.
3. Elimination of all roughing-to-finishing crossover connections. The elimination of crossovers simplified vacuum operation and presented no serious limitations as long as full vapor trapping was provided. Later experiments testing the possibility of operating without vapor traps, however, indicated that, although the 43-cfm finishing pumps were adequate for backing a trapped system or one in which

steady operation had been established, they were probably inadequate during the bake-out period of an untrapped system.

In such a case, crossover connections that would allow the use of the large roughing pumps for backing purposes during the initial stages of diffusion pumping would appear to be a practical solution of the difficulty. Crossover connections would also provide for the emergency that arises when there is a failure of the backing pump on an operating unit. At present, operation is continued under such conditions by installing a temporary crossover of rubber vacuum tubing between existing instrument valves on the finishing and roughing lines. If the operating tank is tight and well outgassed, the diffusion pumps will continue operating for half an hour or more into the closed off fore-vacuum line. Thus, short servicing outages of the backing pump are possible without the provision of substitute backing.

The four Alpha II vacuum systems embodied improvements over the Alpha I systems both in equipment and design. The tanks were larger, they operated with four rather than two ion beams per tank, and much higher diffusion pumping speed was provided.

Original plans provided a 303-cfm roughing pump per 12-tank header section, with four header sections joined by sectionalizing valves. One 105-cfm pump backed the diffusion pumps of two tanks, and pairs of adjacent finishing headers were connected by a cross-tie line. Roughing-to-finishing crossover connections were also provided.

Operating experience very soon indicated a need for greater roughing capacity, and the roughing headers were further divided into six-tank sections. Each section was pumped by a 303-cfm pump, and the eight sections comprising a half track were joined by sectionalizing valves. This increase in the number of roughing pumps gave more efficient roughing, since it was frequently possible to utilize the pumps of two or more adjacent sections in roughing a single tank; but, of even greater importance, it allowed the use of the large-capacity roughing pumps for backing purposes during the bake-out stage of finishing. This accelerated pump-down and also reduced the danger of interference with the established operation in one tank by the heavy outgassing in another.

A 105-cfm pump proved barely adequate for backing the six large diffusion pumps on two tanks, especially during early stages of operation of one of the tanks. One of several steps taken to alleviate this condition was to increase the capacity of the mechanical pumps by driving them at higher than normal speed. Extensive tests (see Fig. 1.11) indicate that 15 per cent increase in pumping speed, with small

Fig. 1.10—Late Beta vacuum system flow diagram.

Fig. 1.11 — Pumping speed vs. pressure curves for Kinney pump driven at various speeds.

loss in volumetric efficiency, can be realized by driving the pumps at about 25 per cent overspeed. This change was made on one Alpha II track and was partially, but not wholly, effective in meeting the situation. No adverse effects were noted in operating at this higher speed over a period of several months.

A survey of plant-wide experience in vacuum operation revealed certain rather well-defined trends in the changes that had been found necessary or desirable in so far as mechanical-pump and header requirements were concerned. In analyzing these trends as possible indications of principles to be applied in future design it should be remembered that, up to the time when the last plant unit was well under construction, maximum output rather than most economical production was the first aim of design and operation. Allowance must be made for this unusual condition in evaluation of the various plant experiences.

In evaluating the rough vacuum requirements of multiple-unit vacuum systems such as those here described, it is now clear that pumping requirements for three distinct stages of evacuation must be considered. These stages may be designated as roughing, prefinishing, and finishing.

Roughing comprises that stage of evacuation during which the pressure in the system is being reduced from atmospheric pressure to a pressure such that diffusion pumping may be advantageously used. If the system being exhausted is relatively dry, the bulk of the gas removed is free air, and the time required for roughing a given system is approximately proportional to the volume to be evacuated and inversely proportional to the roughing-pump capacity. If roughing time is to be a minimum, it follows, therefore, that pumps of the greatest practical capacity should be used.

Because of the high cost and low utilization of the large pumps required to rough-down tanks in a small fraction (1 per cent or less) of the operating cycle, it is highly uneconomical to provide each tank with its individual roughing pump. If, however, a single large-capacity pump is used for roughing any one of a number of tanks, fewer pumps are required, each is more fully utilized, and short roughing time is economically realized.

Thus the roughing-header type of system is justified, and the question arises as to how many units may be operated from a single header. If the above ratio of roughing time to operating cycle is accepted, 96 tanks, or a whole Alpha track, could be served by a single roughing header provided operating cycles could be rigorously scheduled and provided the roughing pumps performed no service other than roughing. Actually rigorous scheduling of the operating cycle is not prac-

tical, and 12 tanks per roughing section is the largest number attempted.

To assure continuity of service no roughing section should be solely dependent on a single pump. This is avoided by the use of sectionalizing valves between adjacent header sections or by the providing of more than one pump for each section.

Any need for additional roughing-pump capacity may be met by substituting pumps of a larger size, by increasing the number of pumps on a header section, or by installing additional pumps and further sectionalizing. It is this last plan that has usually been adopted, partly because greater flexibility of operation results, but mainly because such an arrangement makes it possible to use the roughing pumps not only for roughing but also for backing the diffusion pumps during the prefinishing stage of evacuation.

Prefinishing refers to that stage of evacuation from the turning on of the diffusion pumps until the pressure in the system is reduced to a value such that the diffusion pumps will operate at their full rated speed when backed by the finishing pumps provided. Since roughing is usually terminated at an arbitrary pressure of 25 to 50 μ Hg, and since the full diffusion pumping speed from a tank is several thousand liters per second, it is quite out of the question to provide backing pumps of sufficient capacity to maintain the fore pressure required for full diffusion pumping until the pressure within the system has been reduced to a small fraction of this value. During this period the diffusion pumps operate at such a speed as to maintain the fore pressure at their dynamic fore-pressure limit, and the quantity of gas removed from the system per second is proportional to the speed of the fore pump at this pressure. It follows that high backing-pump capacity or high dynamic fore-pressure limit is required if the prefinishing period is not to be unduly prolonged. This requirement is even more severe because of the heavy normal and induced outgassing that occurs during this stage of pump-down, for it is at this time that preliminary heating and initiation of the arc and beam occur.

To provide each unit with a finishing pump of sufficient capacity to meet the needs of prefinishing is out of the question, since the backing requirements during the remaining 90 per cent or more of the operating cycle are quite small. In the first Alpha I systems three large (303 cfm) pumps on a 24-tank header were provided. Since little of this capacity is required to carry the fully operating tanks, there remains a large reserve capacity for backing those tanks in the section that might be in the prefinishing stage. Fully operating tanks are unlikely to be adversely affected by the large gas output of tanks being prefinished on the same header because of the high limit-

ing fore pressure of a diffusion pump through which little gas is passing. The main objection to this system is, therefore, the ever-present danger that 24 units may be thrown out of production because of a single vacuum failure or operational error.

To reduce greatly the number of tanks per finishing section and still maintain an adequate reserve pumping capacity for handling prefinishing requires an unnecessarily large number of large-size pumps. Since large-capacity pumps are also required for efficient roughing, the most practical solution found to the prefinishing problem has been the use of small roughing-header sections serving six to eight tanks, each pumped by a 303-cfm pump. By means of cross-over connections this pump is used for both roughing and prefinishing. The tank is not thrown on the finishing pump until outgassing is greatly reduced, steady operation is attained, and the backing requirements are quite moderate. With this arrangement the large roughing pumps are utilized more fully than would otherwise be possible, and the finishing-pump demand is reduced to such a point that pumps of small size may be used. If more rigorous scheduling of operation were possible, operation of many more tanks per roughing-header section would be practicable.

Finishing may be considered as beginning with full diffusion pumping and continuing throughout the remainder of the run. If the same fore pump is used for prefinishing and finishing, the first operation passes imperceptibly into the second. If the roughing pump is used for prefinishing, the finishing is usually well advanced before the system is switched to the finishing pump. In this case, and throughout the period of full operation in any case, the pumps act to maintain operating pressure only, and they handle very little gas. The fore-pump demands are, therefore, very moderate during operation, and many tanks could be operated from a single finishing header except for the danger of large-scale production loss due to accidents as previously mentioned. This danger is so real that the tendency has been to rely on a large number of small finishing pumps, each serving one or two tanks, rather than on multiple tank headers using larger pumps.

Perhaps the chief drawback to systems of the type just described lies in the complexity of their operation. Elaborate valving arrangements are required, and the valving procedures that must be followed if the system is to be used to its full capacity cannot be rigorously formulated. Thus costly mistakes in operation are likely to occur unless operating crews are highly trained and alert.

6. DIFFUSION PUMPS

6.1 Diffusion-pump Requirements. Basically the function of the diffusion pumps in a typical production vacuum system is to reduce the pressure in the system from the lowest value readily attainable with mechanical pumps to the much lower value required for operation, and to maintain this required pressure throughout the period of operation. Furthermore, this must be accomplished in a time that is

Table 1.2—Physical Data of Typical Process Tanks

	Alpha I	Beta	Alpha II
Total units	480	288	384
Evacuated volume per unit:			
Cu ft	312	86.5	462
Liters	8,840	2,450	13,100
Exposed surface area per unit, sq ft	461	176.5	652
Exposed weld length per unit, ft	200	125	210
Closure gasket length per unit, ft	75	60	140

short relative to the length of the operating cycle, and attempts at evacuation must result in the attainment of operating pressures in a high percentage of cases.

Physical data relative to the various units that require evacuation are given in Table 1.2.

The volume of a system fixes definitely the quantity of free gas to be removed in reducing the pressure through any given range. The remaining factors listed contribute to residual leakage, outgassing, or both. It is not, however, safe to assume that the surface outgassing in two systems will be proportional to their exposed surface areas, since rate and extent of outgassing depends also on surface condition, temperature, and other variables of operation. Nor would it be of particular value to know, much less to assume, that the average residual leakage is proportional to weld and gasket closure length, since individual variations in leakage are great.

A simple calculation shows that diffusion pumps of very moderate capacity are necessary to produce the required reduction in pressure

in systems of the volumes here dealt with, provided nothing more is involved than the removal of free air from the system. In the presence of residual leakage, outgassing, and gas evolution due to the production process, however, the pressure eventually attained is a function of the rate at which gas enters the system, the rate of its removal, and the base pressure attainable by the pumps. Since the base pressure attainable by a typical oil diffusion pump is of the order of $0.005 \mu \text{ Hg}$ and operating pressure is below $0.05 \mu \text{ Hg}$, it follows that the net rate of combined leakage, outgassing, and gas evolution, in micron-liters per second must be less than $0.045 \times S$, where S is the speed of the pump in liters per second, if operating pressure is to be attained. Since the rate at which gas enters a system is a highly indeterminate quantity, varying widely from system to system and from time to time, it follows that the greatest hope of consistently attaining operating pressure lies in making S , the pumping speed, large enough to cope with any situation likely to arise. Limited experience in operating systems similar to those designed for the plant indicated that the highest pumping speed available at the time of initial plant design would not be excessive in plant operation.

This policy of providing pumping speed far in excess of what might be considered minimum requirements has been amply justified. The whole vacuum phase of the electromagnetic separation problem would have been infinitely more difficult but for this high pumping speed provided.

6.2 Diffusion-pump Installations. Typical diffusion-pump installations in Alpha I, Beta, and Alpha II systems are shown in Figs. 1.1 to 1.6. The practice of ganging two or more diffusion pumps in parallel on a single production tank was established as feasible in preliminary operation, and extensive plant experience confirms this. The practicability of operating many diffusion pumps into a single backing header is limited by the nature of the process and the competence of operating personnel. The trend in this plant has been in the direction of smaller numbers of diffusion pumps per backing header.

6.3 Diffusion-pump Types. Oil diffusion pumps of five distinct types have been used extensively in the Y-12 plant. Four of these types are large multiple-stage pumping units that were developed and manufactured specifically for this job. The fifth type is a small pump that was commercially available prior to the designing of the plant. Units of the first four types, though differing in size, performance, and details of construction, have certain points of similarity:

1. Each is a complete diffusion pumping unit consisting of a large high-speed primary pump backed by a booster pump.

2. All are vertical-type multiple-stage pumps.
3. All pumps are of welded-steel construction.
4. Clamped-on ring-type electric heaters are used in all cases.
5. Pump side walls and high-vacuum inlet baffles are water-cooled.

(a) Westinghouse 20-8. Pumps of this type were developed at UCRL and manufactured for plant use by the Westinghouse Electric Manufacturing Company in their East Pittsburgh plant. Both Westinghouse and Stone and Webster contributed to details of design of the final production model.

As shown schematically in Fig. 1.12 this is a diffusion pumping unit consisting of a primary and booster pump in series. The barrel of the primary pump is 20 in. in diameter, flaring near the top to allow higher conductance past the pump-head baffle. The booster pump is 8 in. in diameter and discharges through a 4-in. arm. Over-all height of the unit is approximately 62 in.

Pumps of this type were installed, two units per process tank, in all Alpha I tracks and in tracks 1, 2, 5, and 6 of Beta systems.

(b) D.P.I. 20-6. In the early stages of plant design Distillation Products, Inc., was asked to design and build a number of units of the same general size as the Westinghouse 20-8 units and with performance characteristics equal to or better than these. The result was a unit (see Fig. 1.13) consisting of a three-stage 20-in. primary pump, now designated by the manufacturer as MC-7000, backed by a 6-in. booster pump (MB-200). Although the performance characteristics of this pump are somewhat better than those of the Westinghouse pump, the latter embodies several superior features of mechanical design and is generally of a more rugged construction.

Pumps of the D.P.I. 20-6 type are used in Beta tracks 3 and 4.

(c) Westinghouse 32-8. When plans were being made for construction of the Alpha II systems, Westinghouse was asked to design pumping units of still greater size and capacity to meet the anticipated needs arising from four-beam operation. The resulting unit (see Fig. 1.14) consists of a three-stage primary pump built in a barrel of 30-in. base diameter flaring slightly to a 32-in. intake opening, backed by an 8-in. booster in which the jets have been redesigned to provide handling capacity for backing this large primary pump. The over-all height of the unit is 80 in.

Each Alpha II production tank is equipped with three of these units.

(d) Westinghouse-D.P.I. 20-8. Although the fifth Alpha I track was originally designed for two-beam operation, it operated from the first as a four-beam, or Alpha II, system. Thus an abnormally heavy load was thrown on the pumping system in this track, and the need for

higher pumping speed was soon apparent. To attain this end without radically altering the vacuum system, three changes in the diffusion pumps were made. These were (1) removal of the baffle from the head of the 20-in. pump, (2) substitution of a 20-in. jet assembly of

Fig. 1.12 — Westinghouse 20-8 diffusion pump.

the MC-7000 type, and (3) substitution of an 8-in. jet assembly modeled after that of the MB-200.

The combined effect of the first two changes was an increase of approximately 60 per cent in pumping speed, and the third change gave a 20 per cent increase in the limiting fore pressure.

Prior to the closing down of all Alpha I tracks, these changes had been made throughout track 5, and on an experimental basis in a few other Alpha I units.

Fig. 1.13 — Distillation Products 20-6 diffusion pump.

When the seventh and eighth Beta tracks were planned advantage was taken of the above experience and the pumps provided were standard Westinghouse 20-in. and 8-in. barrels equipped with D.P.I. jet assemblies. MC-7000 jets were used in the 20-in. pump, but the baffle was retained because of the nearness of the pump to the process

tank and the greater need for protecting the operating unit from oil vapor. The jets used in the 8-in. pump were of a type developed after conversion had been made in the fifth Alpha I track and have even

Fig. 1.14 — Westinghouse 32-8 diffusion pump.

better operating characteristics than those used in this conversion.

(e) D.P.I. MC-500. These small pumps were being manufactured by Distillation Products prior to the initiation of this project, and are used on a number of small test systems in various Beta buildings.

They are three-stage pumps of 6-in. barrel diameter and differ from other pumps in the plant in their use of internal heaters and aluminum jet assemblies.

Plant-wide distribution of pumps of the various types is summarized in Table 1.3.

Table 1.3—Diffusion-pump Distribution by Types

Type system	Total no. of diffusion pumps				
	Westinghouse 20-8	D.P.I. 20-6	Westinghouse 32-8	Westinghouse- D.P.I. 20-8	MC-500
Alpha I	960*				
Tracks 1-5	(768)			(192)*	
Aux. systems	77				
Beta					
Tracks 1, 2, 5, 6	288				
Tracks 3, 4		144			
Tracks 7, 8				144	
Aux. systems	6	6		3	120
Alpha II					
Tracks 1-4			1152		
Aux. systems	72				
Total	1,403 (1,211)	150	1152	147 (339)	120

*192 pumps on track 5 converted to Westinghouse-D.P.I. 20-8 in the fall of 1944.

6.4 Comparison of Design and Structural Details. Before proceeding to a consideration of the operating characteristics of the various types of diffusion pumps, certain details of design and construction will be reviewed in the light of operating experience.

(a) Pump Barrels. The over-all dimension of a diffusion-pump barrel is dictated primarily by three considerations:

1. The speed of the pump for a given jet geometry is roughly proportional to the cross-sectional area of the barrel in the plane of the first-stage jet.

2. The quantity of vapor available for operation, if the oil temperature is to remain within permissible limits, is proportional to the area of oil surface. Also, if external electric heaters are used, the area of the pump bottom limits the power that can be applied without exceeding the heater rating.

3. The minimum height of the barrel is determined by the number of diffusion stages, the distance required between stages, and the baffle-space requirements.

Enlarging the upper part of the pump barrel, either by flaring as in the Westinghouse pumps or by a step as in the D.P.I. 20-6 type, is desirable when a baffle is to be used. By such construction reasonably tight baffling without excessive impedance is secured. If no baffle is to be used it is doubtful whether the gain from this construction would justify its expense. Of the two methods of providing enlarged pump openings, the flared barrel is preferable because of simpler construction, less welding, and probably greater admittance. The resulting design is also better in appearance.

In the primary pumps of all larger pumping units, adequate vapor is supplied by applying heat at a rate less than 15 watts per square inch of pump base. This is well within the safe operating range for commercial ring-type heaters. In the booster pumps, however, the power input and therefore the limiting pressure at which the pumps will operate is limited by the area of the pump base and the heater manufacturer's recommended limit of 30 watts heat dissipation per square inch. The 8-in. pumps are operated at 2,000 watts or less although operation at higher power offers distinct advantages. At 2,000 watts heat is dissipated at a rate of 40 watts per square inch of heater contact area, and this exceeds the dissipation rate recommended by heater manufacturers. Possible methods of correcting this condition are discussed in a later section dealing with pump heaters.

It cannot be claimed that the optimum barrel length for a pump of given diameter and number of stages has been determined. It appears probable, however, that the ratio of barrel length to diameter can be somewhat reduced whenever economy of space demands. It is true that jet assemblies having one more diffusion stage than was originally provided in both the 20-in. and 32-in. Westinghouse barrels have been successfully operated with resulting improvements in performance. But in both cases the new jet assemblies encroach, to some extent, on the space originally provided for the baffle.

(b) Interpump Connection. Although no special criticism can be made of the D.P.I. type of interpump connection, the type of connection used on all Westinghouse pumps is preferred on the basis of ruggedness and appearance.

Whatever the type of interpump connection, it should be so designed as to bring the bases of the two pumps into the same plane in order that the oil levels in the boilers of the two pumps may be simply equalized if this proves necessary.

(c) Cooling Coils. The use of copper tubing of generous size and relatively heavy walls for pump cooling is favored because such construction minimizes the chance of stoppage, assures adequate flow at

moderate pressure drop, and is able to withstand mechanical maltreatment. The $\frac{1}{2}$ - and $\frac{5}{8}$ -in. tubing used on Westinghouse 20-8 and 32-8 units, respectively, has been quite free from operating trouble.

Soft-soldering the tubing to the barrel wall, especially if the tube has been slightly flattened on one side to provide broad contact, has proved not only satisfactory but in some respects preferable to hard-soldering. Repairs are more easily made, if they become necessary, and the danger of overheating the tubing during the soldering process is reduced.

The provision of fixed cooling-coil terminals to which standard pipe may be connected is definitely preferable to free ends to which SAE or similar fittings are to be applied.

Since barrel-cooling requirements are, on the whole, quite moderate, it appears probable that adequate cooling would be provided by coils of wider spacing than those used in the present pumps.

(d) Baffles. In instances in which a water-cooled baffle is connected in series with the pump-cooling line, the baffle-cooling tube should not be smaller in size than that used on the pump barrel.

The degree of baffling required in a given situation is difficult to determine since it depends on the tolerance of the operating system for oil vapor, the inherent back-streaming characteristics on the pump jet assembly, the rate of heating and degree of cooling, and on the geometry of path from pump to tank. Baffling obviously should be held to a minimum, thereby assuring maximum conductance and pumping speed. The baffles supplied with the standard pumping units result in a reduction of 25 per cent or more in pumping speed. There is little doubt that more open baffles or no baffles at all would suffice in most installations.

(e) Pump Heaters. All pumps except the MC-500 are heated by one or more nests of ring-type electric heaters clamped firmly to the pump bottom. Gas heating would undoubtedly be practical, but gas is not available at the plant. Three principal reasons can be given for the choice of external- rather than immersion-type heaters:

1. The external heating system is independent of the vacuum system, and replacement of defective heater elements is the simplest possible procedure.

2. The presence of immersion heaters interferes with the simple design of fractionating jet assembly adopted in early pumps.

3. The multiple-unit feature of large external heaters makes it easy to provide a series of heating rates for each pump.

On the basis of the very limited experience in large diffusion-pump operation at the time the pumps were designed, the choice of external heaters was a logical one. In plant operation the service rendered by

these heaters has been even better than was anticipated. Replacements, after a year or more of continuous operation were at an estimated rate of less than 0.2 per cent per month. Heat losses, which were expected to be large, have been greatly reduced in some installations by insulating the heater compartments.

If new pumps were being designed, however, present experience indicates that a change to immersion heaters would be desirable. The long life of external heaters, even when operated above the recommended heat-transfer rate as in the case of most booster pumps, indicates that immersion heaters, chosen to provide the required heat while operating below their rated power, would have extremely long life. Ease of heater replacement then becomes a secondary consideration. The tendency in later jet-assembly design to dispense with full fractionation resolves the problem of interference between immersion heaters and compartmentalized boilers. The provision of a range of heating rates could be provided with immersion heaters, but is unnecessary in most installations.

On the positive side, greater heat efficiency could be obtained with heaters of the immersion type. Also, the rate of applying heat to a pump would not be limited by the area of the pump base. Finally, construction would be simplified.

(f) Oil Drain, Refill, and Equalization. With pumps of the size here considered and under the exigencies of industrial operation, it is essential that means be provided for changing the oil in pumps without their removal from the system and with as little interruption in service as possible. In providing for this a compromise must be made between complicated arrangements that would be most convenient to use and simple arrangements that might prove less convenient but would be less likely to result in leakage. Leakage at a point below the normal oil level in a pump is particularly difficult to detect and should be avoided if possible. It should also be recognized that oil change, under proper operating conditions, should seldom be required, particularly if pumps are removed for cleaning and reservicing on a properly adjusted schedule.

A simple type of drain and refill fixture designed for the first pumps manufactured for the plant and incorporated in most later models is shown in Fig. 1.15a. This device had the advantage of simplicity and had at time of this writing given little trouble as a leak source. Its chief defects are that oil changing requires considerable time and that the closing fittings have a tendency to stick because of prolonged high-temperature operation.

Oil changing has been speeded up by improved techniques and the development of special oil-changing equipment. With this equipment

Fig. 1.15 — (a) Diffusion pump drain and refill assembly. (b),(c),(d) Oil equalizer line types.

the pump is both drained and filled through the refill tube. Thus only one vacuum closure is broken, and, since the refill cap is less prone to stick than is the drainage plug, difficulties arising from sticking fittings are reduced. Pipe fittings of steel were substituted for brass after a number of the less rugged brass fittings had been broken in service.

More significant than any improvements in methods and equipment for oil changing, however, have been the improvements in vacuum-operating technique with a resulting decrease in demands for oil change.

Plant operation showed considerable oil migration occurring from primary to booster pump or vice versa. As a result the oil supply in the booster pump became excessive or deficient. Of several possible methods of rectifying this condition, the one most readily applicable to the pumps then in service (Westinghouse 20-8) appeared to be the installation of oil-equalizer lines connecting the 20-in. and 8-in. boilers. This was a comparatively simple addition to make since the pump bases were at the same level and since drain and refill openings in the bases were already present. Equalizer lines of a number of types have been used more or less extensively throughout the plant. Three of these are shown in Figs. 1.15b, c, and d. Type b, using copper tubing and SAE or similar fittings, has the advantage of being easily removed for pump servicing but is not recommended because of the considerable difficulty experienced in securing vacuum-tight seals with such fittings. Type c, in which the copper equalizing line is joined to the pump fittings by hard-soldered joints, is free of serious leakage problems and is not difficult to remove or replace in servicing. It is even possible to service the booster pump without breaking the equalizer line seals by carefully bending the copper connecting tube. Type d, the simplest of all, consists of a $\frac{3}{8}$ -in. iron pipe with plugged side outlet for oil changing welded directly to the pump bases. However, this line must be cut and rewelded to permit servicing of the booster pump.

Various proposals to incorporate valves, sight glasses, and other complicating features into the oil change-equalizer system have been made from time to time. Generally the advantages offered have been outweighed by possible weaknesses, and they have not been adopted. An obvious proposal to use valves in place of the pipe plugs or caps at the drain or refill openings has been frequently made. Such valves must form vacuum-tight closures, withstand indefinitely the action of hot oil, and provide wide enough openings to allow quick oil change. No valve was known to meet all these requirements, and experimenting on plant equipment was necessarily discouraged because of the

strong possibility of losing operating time. Incidentally, tests made outside the production plant indicate that refrigeration type (Kero-test) valves having phenolic laminated disks meet these requirements very well and may be safely used.

(g) Protective Devices. In large vacuum systems there appears to be no practical means of protecting diffusion pumps against their greatest hazard, namely, the admission of air to the pumps while they are hot. Pressure- or temperature-actuated switches set to cut off heating power with rise of pressure or temperature in the pump might prevent extreme damage but could give only partial protection. Only meticulous attention to vacuum-system operation can eliminate this hazard.

As a protection against overheating in the case of cooling water failure, mercoid switches controlling the heater power are provided on each pumping unit. These switches are mounted on the side of the main pump barrel two turns above the bottom of the cooling coil. Although there is considerable time lag in their response, this is not serious, since moderate overheating of the oil under vacuum causes no harmful effects. On the whole, this type of protection appears to be more positive than that provided by flow switches in the water lines.

(h) Jet Assemblies. The performance characteristics of a diffusion pump are determined by the design of its jet assembly. The early Westinghouse pumps performed very well considering the limited experience behind their design, though there was never reason to doubt that this performance could be improved. Size for size, assemblies designed by Distillation Products have surpassed in performance those built by Westinghouse. This superior performance appears to be the result of minor variations in proportions and geometry* and of an increase in the number of diffusion stages rather than of any radically different design principles. Aside from differences in geometry the most significant difference between the two types of jet assemblies lies in the multiple-boiler construction of the Westinghouse jets as contrasted with the single-boiler jets by Distillation Products. Actually this difference does not appear to be very significant. The fractionation provided by the concentric boiler compartments is certainly not necessary for obtaining the required operating pressures, whereas use of a single boiler from which vapor may find its way to any jet removes all necessity of balancing vapor distribution to the various jets. Also, the single-boiler construction is some-

*Throughout this volume the term "geometry" is used to mean the size, shape, and relative dimensions of component parts together with their orientation and spacing in an assembly.

what simpler and facilitates, as has been pointed out, the use of the immersion-type heater.

Some 32-in. jets, recently designed on an experimental basis but never installed in plant pumps, incorporate a feature designed to reduce heat loss by radiation from the rising vapor stream to the cooled pump walls. Since the surface area of the chimney conducting vapor to the jets is very large, especially in the section between the boiler and lower jet, considerable heat is lost by radiation. To reduce this loss a radiation shield is placed around this large chimney section. The effectiveness of this shielding has not been definitely determined, although operating characteristics of these jets indicate an appreciable improvement in thermal efficiency.

In the design of jet assemblies account should be taken of the fact that such assemblies may occasionally require thorough cleaning. The assembly should, therefore, be readily separable at sufficient points to give free access to all parts. It is advantageous to have parts permanently joined by spot-welding, brazing, or welding wherever access does not require a separable joint.

6.5 Operating Characteristics of Diffusion Pumps. The significant operating characteristics of the various types of diffusion pumps have been determined on many occasions both by laboratory and plant tests. From the operating point of view, the significant characteristics of a pump may be limited to

1. The pumping speed at, and immediately above, normal operating pressure.
2. The minimum pressure attainable.
3. The limiting fore pressure, both at no load and at full load.
4. The optimum heating power for the service required.
5. Such minor factors as cooling requirements and oil vapor migration.

A knowledge of these characteristics is sufficient for evaluating and maximizing the performance of a given pump or pumping unit. In the case of an integral unit of two or more pumps, it is the over-all performance of the unit, and not that of the individual elements, that is of importance. More detailed characteristics, which might be significant in altering or redesigning a pump to improve its performance, are not considered at this point.

Experience with large numbers of oil diffusion pumps of various types indicates that, under similar conditions of operation, individual pumps of a given type have characteristics that are identical within the limits of the methods commonly used in determining these characteristics. Characteristics of a type may be accepted, therefore, as applying to all pumps of that type.

(a) Pumping Speed. The pumping speed of a multistage oil diffusion pump is essentially constant throughout the normal pressure range if its speed is defined by

$$S = \frac{rB}{P - P_0}$$

where S is the pumping speed in liters per second, r is the rate at which air at atmospheric pressure is admitted to the pump in milliliters per second, B is the barometric pressure in millimeters of mercury, and $P - P_0$ is the pressure rise, in microns of mercury, produced by the air admitted.

(b) Ultimate Pressure. Although the minimum pressure attainable by a pump may never be determined, it is important to know that the pump is capable of producing pressures substantially lower than the required operating pressure.

(c) Limiting Fore Pressure. The limiting fore pressure of a multistage pump, as indicated by loss of high vacuum at the pump head, occurs usually after unsteady pumping in the final stages has set in. As a result, the fore pressure is fluctuating, perhaps widely, when final breakdown occurs and the value of fore-pressure limit established under these conditions is necessarily an average and approximate value.

(d) Heating Requirements. In general, and within rather wide limits, the following conditions hold:

1. The speed of a diffusion pump is essentially independent of the power supplied.
2. Lower ultimate pressures are attained at lower heating rates.
3. Limiting fore pressures increase as the heating rate increases.

The general range of heating within which a given type of pump will operate is readily determined. In view of the above effect of heating rate on operating characteristics, a primary pump is usually operated in the lower part of its range and a booster pump in the upper part of its range.

(e) Tabulation of Characteristics. With the above facts in mind, typical significant operating characteristics of the various types of plant pumping units are presented in Table 1.4.

Pump speeds given in this table are speeds with baffles except in the case of the converted pumps of Alpha I, track 5, where baffles were removed. In general, removal of baffles results in an increase in pumping speed of 25 to 35 per cent.

Fore-pressure limits both at no load and at full load are the values, conservatively stated, corresponding to normal operating power. In

almost every case higher fore-pressure limits can be attained by operating the booster pump at higher power input.

At the high pumping speeds provided by these pumps, high-vacuum flow resistance between tank and pumps becomes appreciable even if

Table 1.4—Operating Characteristics of Various Types of Pumps*

Type	Speed at 10^{-4} mm Hg, liters/sec	No-load fore- pressure limit, μ Hg	Full-load fore-pressure limit, μ Hg	Normal operating power, watts
Westinghouse 20-8	3,750	170	130	4,000
D.P.I. 20-8	5,000	200	150	1,800
				3,500
Westinghouse 32-8	8,000	120	90	600
				7,000
				2,000
Westinghouse-	5,000	280†	150†	4,000
D.P.I. 20-8	6,300‡	300§	160§	2,200
MC-500	400	200	150	600

* Minimum pressure for all pumps was 5×10^{-6} mm Hg or lower, using Litton Molecular-C or Myvane oil.

† In Alpha I, track 5.

‡ Without baffle as in Alpha I, track 5.

§ In Beta, tracks 7 and 8.

ducts of large cross sections are used. Consequently, the actual speed from the various process tanks in comparison with combined speed of operating pumps is as given in Table 1.5. These net pumping speeds are averages based on both calculated and experimentally determined values, and apply to empty tanks. If liners and baffles are an integral part of the operating units, the speed of pumping from within these is even further reduced.

The maximum fore-pump speeds given in Table 1.5 assume a mechanical-pump efficiency of 50 per cent at the limiting fore pressure. No allowance is made for the possibility of using two or more finishing pumps, or large-capacity roughing pumps, for backing purposes during the early stages of diffusion pumping, nor for the fact that large-capacity finishing pumps are always used on headers serving a number of units.

From the data given in Table 1.4, additional significant operating limits may be derived:

1. Maximum rate of gas evolution permitting the attainment of operating pressure. Assume the base pressure attainable by all pumps to be 0.005μ Hg and maximum operating pressure to be 0.05

Table 1.5—Pumping Characteristics of Alpha I, Beta, and Alpha II Production Units

Production unit	Gross vol. of system, liters	No. and type of pumps	Combined speed of pumps, liters/sec	Net pump-ing speed from tank, liters/sec	Full-load fore-pres-sure limit, μ Hg	Max. fore-pump speed, liters/sec
Alpha I Tracks 1-5	8,840	2, Westing-house 20-8	7,500 12,600*	6,300 10,000*	130 150*	71
Beta Tracks 1,2	2,450	2, Westing-house 20-8	7,500	4,500	130	71
Tracks 5,6	2,560	2, Westing-house 20-8	7,500	4,500	130	10
Tracks 3,4	2,560	2, D.P.I. 20-8	10,000	5,500	150	71
Tracks 7,8	2,560	2, Westing-house-D.P.I. 20-8	10,000	5,500	160	10
Alpha II Tracks 1-4	13,000	3, Westing-house 32-8	24,000	14,000	90	25

*Track 5 after conversion.

Table 1.6—Speed-Pressure Characteristics of Typical Systems

Tracks	Net pumping speed from tank, liters/sec	Full load fore-pump limit, μ Hg	Max. fore-pump speed, liters/sec	$q_{\max}$, μ -liters/sec	ΔP , μ Hg	$P_{\max}$, μ Hg
Alpha I						
1-5	6,300	130	71	283	4.0	1.47
5 converted	10,000	150	71	450	6.3	1.06
Beta						
1, 2	4,500	130	71	202	2.8	2.05
3, 4	5,500	150	71	248	3.5	1.94
5, 6	4,500	130	10*	202	20.2	.29
7, 8	5,500	150	10*	248	24.8	.27
Alpha II						
1-4	14,000	90	25†	630	25.2	.16

*One tank per finishing pump.

†Two tanks per finishing pump.

μ Hg. Then for a pumping speed from the tank of 6,300 liters/sec the maximum permissible rate of gas evolution, including residual leakage, outgassing, and gas liberation by the production process, is given by

$$q = (P - P_0) S$$

where q is in micron-liters per second. Thus for a typical Alpha I system

$$\begin{aligned} q &= (0.05 - 0.005) \times 6,300 \\ &= 0.045 \times 6,300 \\ &= 283 \mu\text{-liters/sec} \end{aligned}$$

This value must be accepted as an approximation only since the speed of pumping from various regions of the operating tank is further reduced by the impedance of baffles, shields, etc., and since the pumping action of cold traps for condensable vapors has been neglected.

2. Maximum tank pressure for full-speed diffusion pumping. The diffusion pumps may be assumed to attain their full pumping speed when

$$\begin{aligned} \text{Tank pressure} \times \text{net pumping speed from tank} \\ = \text{limiting fore pressure} \times \text{fore-pump speed} \end{aligned}$$

Thus for a typical Alpha I system,

$$\begin{aligned} P \times 6,300 &= 130 \times 71 \\ P &= 1.47 \mu \text{ Hg} \end{aligned}$$

This value of P , like that of q above, is subject to the same approximations, and in addition, in the case of multiple-tank finishing headers, only a part of the fore-pump capacity is available for backing the pumps of a given unit.

These limits, as calculated for the various types of systems, are given in Table 1.6. In this table $q_{\max}$ is the maximum gassing rate for operating pressure, P is the fore-pressure elevation at maximum operating pressure, and $P_{\max}$ is the maximum tank pressure for full diffusion pumping speed.

Notice that the maximum gassing rate at which operating pressure may be attained is essentially constant for all Beta and two-beam Alpha units but is approximately twice as high for four-beam Alpha units. Expressed in terms of standard atmospheric leakage rates, these allowable rates of gassing vary from 0.25 to 0.83 ml/sec at atmospheric pressure.

In comparing the fore-pressure elevations resulting from tanks at maximum operating pressure, account must be taken of the number of tanks per finishing pump in the various systems. Thus in Alpha I systems with eight tanks per finishing pump the fore-pressure elevation, with all eight tanks operating, may be as high as 32μ Hg in tracks 1 to 4 and 50μ Hg in track 5. Likewise, in Beta tracks 1 to 4 the fore-pressure elevation due to six operating tanks may be as high as 17 (or 21) μ Hg. All of these values are well below the full-load fore-pressure limit of the diffusion pumps and may be considered safe. In Beta tracks 5 to 8 the 20 (or 25) μ Hg pressure elevations are also quite safe. With two Alpha II tanks pumped by a single 105-cfm fore pump, however, fore-pressure elevation with both tanks operating may be as high as 50μ Hg. Such elevation may bring the fore pressure dangerously close to the full-load fore-pressure limit for the Alpha II pumps, and allows a very narrow margin of operating safety.

For merely sustaining operating pressure small individual fore pumps as used in Beta tracks 5 to 8 are about equivalent to the one large pump per six tanks as in Beta tracks 1 to 4. For carrying a tank through its period of outgassing and reducing it to operating pressure, however, large fore-pump capacity offers obvious advantages. During this period the speed of removal of gas from the system is limited by the fore-pump speed until the tank pressure has been reduced to the designated value $P_{\max}$. Comparing Beta tracks 1 and 2 with tracks 5 and 6, in which conditions are identical except for the sizes of fore pumps and the number of tanks per pump, it is obvious that with the smaller pumps of tracks 5 and 6 the rate of removal of gas during this period is only one-seventh as great as in tracks 1 and 2, and in addition this condition persists until the tank pressure is reduced to a value only one-seventh as high as required in the case of the larger fore pumps. It is just this situation that has led to the use of the large-capacity roughing pumps for backing purposes during the initial pump-down period. Obviously, full advantage of large fore-pump capacity can be realized only to the extent that it is possible to maintain outgassing at such a rate that the tank pressure remains at, or above, $P_{\max}$. Likewise, sustained operation can be had only when the total rate of outgassing and gas evolution does not raise the tank pressure above $P_{\max}$.

6.6 Evaluation and Improvement of Diffusion-pump Performance.

In the preceding section operating characteristics of the various types of diffusion pumps used in the plant have been summarized without seriously considering the adequacy or inadequacy of these pumps for

the service demanded of them. Likewise, little attention has been given to the diffusion-pump improvements that have been introduced.

The present section deals with an evaluation of pump performance in the light of operating experience, and indicates significant changes that have been made both in the pumps themselves and methods of operating them. If in doing this there should be created an impression of serious inadequacy on the part of the pumps originally designed for the plant, let it be remembered that these were designed, produced, and put into operation in the shortest possible time and without benefit of extensive development. Although they were recognized as the best pumps immediately available, no one considered them the best pumps possible. They did, however, without significant change or improvement make possible the early operation of the plant, and this alone served to justify their use.

(a) Pumping Speed. All Alpha I and early Beta tanks were equipped with two Westinghouse 20-8 pumps per production tank. Only two pumps were used because of space limitations, and the Westinghouse pumps were of the highest speed available at the time of their installation. Thus each production unit was provided with the highest possible speed of pumping. Except for the fifth Alpha I track, in which the pumps were converted to yield higher pumping speed for reasons previously mentioned, these pumps have been considered satisfactory in so far as their speed is concerned. It is true that increased pumping speed would have resulted in some improvement in operation. When the value of the improvement in operation is weighed against the cost of conversion, however, the necessary changes have never seemed justified.

The higher speed D.P.I. 20-6 pumps found in the third and fourth Beta tracks were possible because a longer time was allowable for getting these tracks into operation. Thus, the manufacturers had, in addition to their considerable experience in diffusion-pump design, a longer period for developing and producing these pumps. The added speed provided by these pumps is definitely reflected in plant operation, and pumps of this type would probably have been used in the fifth and sixth Beta tracks but for the limited production capacity of the manufacturer.

When the seventh and eighth Beta tracks were designed as the last plant units to be constructed, Westinghouse-D.P.I. 20-8 pumps were chosen because they had the best operating characteristics, including pumping speed, of any known pump of this size.

The Westinghouse 32-8 pumps of the Alpha II tracks were designed primarily for high pumping speed, and because of a change in magnet design and tank location it was possible to put three pumps on each

production unit. That the resulting high pumping speed was advantageous in operation cannot be questioned. However, it appears that the returns from high pumping speed are definitely diminishing in this extreme case as is shown in the following comparison, Table 1.7, of typical Alpha I and Alpha II operation.

The low ratio of useful to total pumping speed in the Alpha II system is due, of course, to the impossibility of increasing tank-to-pump conductance in the same ratio that pump speed has been increased.

Table 1.7—Comparison of Alpha I and Alpha II Performance

	Alpha I	Alpha II
Total speed of pumps per tank, liters/sec	7,500	24,000
Net speed of pumping from tank, liters/sec	6,300	14,000
Ratio of useful speed/total speed	0.84	0.58
Total normal operating power, kw	11.6	27.0
		(33.0)*
Ratio of useful speed/operating power	543	520
		(424)*
Average pump-down time, hr	24	16
Net speed $\times$ pump-down time	151,000	224,000

* Power required for operation fully comparable with Alpha I.

This tank-to-pump conductance, which cannot be practically increased beyond a certain point because of spatial restrictions, limits the speed that can economically be utilized in any given system.

In comparing power economy as expressed by useful speed per kilowatt of power, the revised figures for Alpha II should probably be used, as explained later in dealing with problems of pump heating.

That the decrease in pump-down time is less than proportional to the increase in useful pumping speed is to be expected. Still it is primarily in this phase of operation that any advantage is to be gained from pumping speed in excess of that actually required to meet the demands of established operation, and for an increase more than threefold in the pumping speed provided, one might reasonably expect to obtain a decrease of more than 33 per cent in pump-down time. The fact that the Alpha II system has a greater volume than the Alpha I contributes to the small reduction in pump-down time, but since removal of free air is a minor part of the final pump-down process, this contribution is not great.

Late in 1944 improved jet assemblies for the Westinghouse 32-in. barrels were developed by Distillation Products, National Research

Corporation, and Westinghouse. Comparative tests of these assemblies, made at UCRL, showed the highest speed to be obtained with a four-stage assembly by Distillation Products. With these jets the present speed of 8,000 liters/sec with baffle is raised to a probable 15,000 liters/sec, and if it is possible to dispense with the baffle a speed of 17,500 liters/sec is attainable. This fact is mentioned as indicating the pump speeds now available, although conversion to high-speed 32-in. jets was never made in the plant.

There are a number of reasons that this conversion of Alpha II pumps to higher pumping speed was never made:

1. It was apparent that the relative increase in useful pumping speed would be much less than the increase of speed at the pump unless the tank-to-pump conductance could be simultaneously increased in comparable ratio. This appeared to be difficult, if not impossible, especially in the case of the two lower pumps. Even at the upper pump an extensive modification of the pump-head valve would have been necessary if any considerable part of the higher pumping speed was to be useful.

2. It was impossible to determine, except by plant experience, what production improvement would result from a given increase in useful pumping rate.

3. Any advantage arising from increased pump speed could be fully realized only if pronounced improvements could also be made in the booster fore-pump system.

4. Finally, an independent conversion program involving the installation of liners in the process tanks and promising to alter pumping requirements was under way at the same time that pump conversion was considered.

Enough of the improved jets to allow a plant test to be made were procured, but the Alpha II plant was closed down before their installation was completed.

To summarize,

1. Pumping units having speeds ranging from 375 liters/sec to 17,500 liters/sec are available.

2. Although higher pumping speed results in improved operation, the extent of improvement measured by shortened start-up diminishes at higher speeds.

3. As the speed of pumps increases the difficulty of providing connecting ducts and valves of comparable conductance also increases. In the end it is likely to be this factor that limits the speed of pumps which can be used economically.

4. The speed of the pumping systems originally provided in the various production units has been, in every case, adequate for sus-

taining operation. The need for higher pumping speed, when it has arisen, has been felt in the pump-down phase of the operating cycle.

(b) Limiting Fore Pressure. In tandem pump units such as those used in all production vacuum systems, the first requirement of the primary pump is that it provide the highest possible pumping speed. The first requirement of the secondary, or booster pump, is that it operate at the highest possible fore pressure. In addition, the limiting fore pressure of the primary pump and the speed of the booster pump must, of course, be such as to provide a gas-handling capacity at least equal to that of the fore pump, otherwise these inter-pump conditions will act to limit the pumping capacity of the unit. Assume for the moment that this last condition is satisfied, and consider the significance of limiting booster fore pressure.

The limiting static fore pressure of a given pump or combination of pumps may be defined as the maximum fore pressure at which the pump will maintain high vacuum when no appreciable gas is passing through the pump. Under these conditions the pump acts merely as a barrier between the high fore-pressure and the low head-pressure regions. Loss of high vacuum at the fore-pressure limit marks the failure, at least in part, of the barrier action, and some gas is allowed to pass back through the pump. Although it is frequently given as a cardinal characteristic of a pump, this factor is actually of very limited significance. A given pump may operate at a fore pressure of 300 μ Hg when no gas is passing through it, but under these conditions there is usually no difficulty in maintaining the fore pressure at a small fraction of this value. Exception should be noted in this connection in the case of several pumps operating into a common header. High static fore-pressure limit here serves to protect an evacuated unit against variations in header pressure arising from sources outside the operating unit.

Of far greater significance than the static fore-pressure limit in evaluating the performance of a pump is its corresponding dynamic limit. By dynamic fore-pressure limit is meant the highest fore pressure at which the pump will operate with full effectiveness when gas, in appreciable quantities, is passing through it. This limit, which is revealed primarily by a sharp dropping off in pumping speed, differs for different rates of gas passage. Therefore when the dynamic fore-pressure limit of a pump is expressed, it is necessary to specify the rate of gas handling at which this limit is observed.

Since, however, the limiting fore pressure is observed to decrease as the rate of passage of gas through the pump increases, and since, for a given fore pump the pressure in the fore system increases with increasing rate of gas delivery to it, there exists for any combination

of diffusion pump and fore pump a rate of gas flow for which the resulting fore pressure just equals the limiting fore pressure of the diffusion pump handling gas at this rate. This limiting pressure and the corresponding quantity of gas passing through the pumps per second are of particular significance in that they represent the dynamic fore-pressure limit at full load and the maximum gas handling capacity of the particular pump combination.

At all pressures below this critical value the diffusion pump speed is definite, and is generally essentially constant. Furthermore, the rate at which gas passes through the system may be expressed as

$$P_1 \times S_D = P_2 \times S_F$$

where P_1 is the high-vacuum pressure and P_2 is the fore pressure, S_D is the diffusion-pump speed and S_F the fore-pump speed, in any consistent system of units.

It follows that for a given pump combination the fore pressure P_2 can be less than the dynamic limit at full load only if the high-vacuum pressure, P_1 , is less than some determinate value. As P_1 exceeds this critical value the volumetric speed of the diffusion pump no longer remains constant but decreases at such a rate as to maintain an essentially constant difference between fore pressure and head pressure. As long as the high-vacuum pressure remains small compared with fore pressure, the latter remains essentially constant. Thus, the rate at which gas is removed from the system is no longer a function of head pressure and rated diffusion-pump speed but of limiting fore pressure and fore-pump speed.

Since the product of the limiting fore pressure at full load and the speed of the fore pump limits the quantity of gas that can be removed per second by a given combination of pumps, it is obvious that this product should be large, especially in those cases in which large quantities of gas are to be handled. In plant production systems the quantity of gas to be removed during established operation was not large and pump capacities are not taxed even in the most unfavorable cases. It is, therefore, during evacuation that limited pump capacity is felt, sometimes seriously.

Consider as an example an Alpha I test dock. This unit consists of a tank with a volume of approximately 9,000 liters evacuated by two Westinghouse 20-8 diffusion pumps backed by a 105-cfm Kinney. The combined pumping speed of the two pumps is about 7,500 liters/sec, and the full-load fore-pressure limit in this system may be taken as 120 μ Hg. Assuming 50 per cent efficiency at this pressure, the speed of the Kinney is 25 liters/sec. If now the diffusion pumps are cut in

when the tank pressure has been reduced by roughing to, for example, say 30 μ Hg, the fore pressure will jump to perhaps 140 μ Hg, settle shortly to 120 μ Hg, and remain at this value until the tank pressure has been reduced to a value, P_1 , such that

$$P_1 \times 7,500 = 120 \times 25$$
$$P_1 = 0.4 \mu \text{ Hg}$$

Throughout most of this period gas is being removed at a rate of only

$$q_1 = 120 \times 25 = 3,000 \mu\text{-liters/sec}$$

owing to fore-pressure limitations.

Except for these limitations pumping would have started at a rate

$$q_2 = 7,500 \times 30 = 225,000 \mu\text{-liters/sec}$$

With reduction in pressure this rate of removal would decrease, becoming 3,000 μ -liters/sec at 0.4 μ Hg head pressure and 120 μ Hg fore pressure. Beyond this point pumping would be identical in the two cases, and would be limited by diffusion-pump speed only.

Although the above assumption of no fore-pressure limitation is unjustified in actual practice, the advantage to be gained from any substantial increase in this limitation is obvious. If the full-load fore-pressure limit of the diffusion pump could be increased threefold, not only would the rate of gas removal during the initial diffusion pumping stages be increased by a factor of three or more, but also the tank pressure at which full pumping speed is attained would be three times as high. Increasing the speed of the fore pump would likewise help, but not to the same extent, since any increase in the rate of gas handling by the diffusion pump would act to reduce its full-load fore-pressure limit. Thus increasing the fore-pump speed from 25 to 75 liters/sec might well result in a reduction of the full-load pressure limit from 120 to 80 μ Hg, and the maximum gas handling capacity would be increased to only

$$q_3 = 75 \times 80 = 6,000 \mu\text{-liters/sec}$$

a twofold capacity increase from a threefold increase in fore-pump speed.

Realizing that the low full-load fore-pressure limit of plant pumping systems was prolonging pump-down time, various steps were taken to improve this condition. These steps are presented briefly

at this point; however, a full discussion of the pump-down problem is reserved for a later section.

(1) Increased Backing-pump Capacity. Within existing vacuum systems it was frequently possible to provide extra backing-pump capacity during the critical pump-down period by using large-capacity roughing pumps for backing purposes. This was standard practice, for example, in Alpha II systems in which a 303-cfm roughing pump could be substituted for, or placed in parallel with, the 105-cfm backing pump. Also, as has been mentioned, the capacity of the Alpha II finishing pumps was increased somewhat by driving them above normal speed.

(2) Booster Jets of Improved Performance. At the same time that jet assemblies were being designed to provide higher primary pumping speed, the development of improved booster jets suitable for use in the original Westinghouse 8-in. barrels and capable of operating at higher fore pressures was also undertaken. The net result of this program was the making available of booster jets that would raise fore-pressure limits from less than 200 μ Hg to above 300 μ Hg at no load, and from about 90 μ Hg to 150 μ Hg at full load. An additional increase of approximately 30 per cent in fore-pressure limits may be attained by increasing the heating rate from 2,000 to 2,500 watts. This increase is gained, however, at the sacrifice of heater life.

Although these steps mark substantial gains in pump capacity they still fall far short of the gains that could be advantageously used. An additional tenfold increase in fore-pressure limits would not only accelerate pump-down to operating pressure but would permit the use of very small backing pumps throughout the long operating period.

It is doubtful, however, that such an increase in fore-pressure limits as is here contemplated can possibly be realized with the present type of booster pumps. It is likely rather that a complete redesign along lines allowing the use of ejector-type jets and high boiler-pressure operation will be required. Also, the booster should be so designed as to make possible the use of special booster pump fluids differing from the fluids required for the high-vacuum fore pumps.

Such extensive alterations in existing systems have not been seriously considered because of the expense and outage time that would be involved. However, the operating limitations imposed by the best present boosters are so serious as to strongly suggest the improvement of this situation in any future design. It can be safely said that the weakest link in present vacuum systems is the condition resulting from low dynamic fore-pressure limits.

(c) Heating Power. The requisite heating power for pumps of the various types and sizes was determined experimentally for a repre-

sentative number of early pumps of each type. To allow for possible variation in requirements arising from individual differences, i.e., differences in installation and differences in operating requirement, heaters were wired through switches mounted on the pump assembly in such a way as to provide two or more rates of heating for each pump.

Heating rates originally provided for the principal types of pumping units are listed in Table 1.8.

Table 1.8 — Diffusion-pump Heating Rates in Watts

	Westinghouse 20-8		D.P.I. 20-6		Westinghouse 32-8		Westinghouse- D.P.I. 20-8	
	20 in.	8 in.	20 in.	6 in.	32 in.	8 in.	20 in.	8 in.
Low	4,000*	1,800	2,700	600	5,000		4,000*	1,800
Medium			3,150	900	6,000	2,000*		
High	4,640	2,000*	3,520*	1,000*	7,000*		4,640	2,000*

* Normal operation.

Operating experience reveals no appreciable difference in heat requirements of different pumps of a given model as long as their conditions of operation are the same. Requirements may, however, differ appreciably with the extent to which heat is lost by radiation and convection. The optimum heating rate as determined for a pump mounted on a test stand with the pump base a few inches from the floor is likely to prove inadequate when the pump is installed in a position far enough above the floor so that radiation and convection losses are a maximum.

Although there is some advantage in being able to select one of several heating rates for a given pump, the advantage hardly seems sufficient to justify the added cost of multiple-tap heaters and range switches since power adjustment is neither critical nor subject to change in a given situation.

Adequate heating within the ranges provided was originally supplied in all pumps except the Westinghouse 32-in. models. The maximum power of 7 kw in these pumps was barely adequate to provide sufficient vapor for effective operation of all jets and was often inadequate in the case of pumps subject to considerable air cooling. In such cases the pumps developed only a fraction of their rated speed.

Temperature measurements made at the pump base showed these pumps to be operating at below 200°C; normal temperature for 20-in. pumps is 225°C.

Two steps were proposed to rectify this deficiency: (1) insulation of the heater compartment and pump base by the application of 2 to 3 in. of mineral wool held in place by a sheet-metal container and (2) the substitution of heater elements of higher power to provide up to 9 kw power input.

Insulation was applied to all 32-in. pumps, and this eliminated defective pumping speed completely. Since the resulting operating temperature was still low (210°C), additional heaters were being installed to provide a factor of operating safety at the time the Alpha II plant was shut down.

The 2 kw normally applied to the 8-in. booster pumps represents a relatively higher rate of heating than used in the primary pumps, as is indicated by their higher (250°C) operating temperature. Even higher heating rates would have increased limiting fore pressures in all cases. Such a change would, however, have resulted in the heaters being operated considerably above their rated temperature.

Although heater compartment insulation was applied only as a means of correcting a deficiency, it is apparent that considerable power economy could be effected by proper heat insulation of large pumps. The insulation applied to the 32-in. pumps was roughly equivalent to an additional 2 kw of power.

The use of pump-base temperature as a check of pump operation has proven, in many instances, a convenient and informative test. The practice of providing a thermometer well in each pump base is, therefore, to be recommended. Either mercury or metallic thermometers may be used, and since only relative temperatures are required, errors due to stem exposure are not important.

(d) Diffusion-pump Oil Problems. Two types of diffusion-pump oils have been used extensively in the plant. These are Litton molecular lubricant C from the Litton Laboratories, and Myvane Diffusion Pump Oil from Distillation Products. Both are petroleum products produced by taking a narrow cut from the vacuum distillation of a standard lubricating oil. In service these two oils behave quite the same. They are alike in the vacuum they will attain, the heating power required, the limiting fore pressure, the extent of back-diffusion, and in the extent of damage by exposure and contamination and in their recovery from such abuse.

Some months after the above work was started samples of a number of silicone oils were tested on an experimental scale as possible diffusion-pump oils. Some of these, because of high vapor pressure, were definitely not usable for high-vacuum purposes. Two samples, however, ZV-5289 from General Electric and type 703 Silicone from Dow Corning, proved suitable for use at the required pressures. The

one clear-cut advantage that these fluids have to offer over previous diffusion-pump fluids is their remarkable resistance to deterioration under the action of air. In systems provided with valving facilities for maintaining pumps under vacuum while other parts of the system are down to air, silicone fluids might be valuable as a protection against harm from operating errors, but realization of the full advantage of these fluids appears to await the design of systems from which valves have been largely eliminated and in which diffusion pumps will be let down to air along with other parts of the system.

Prices of these oils were such as to preclude their use on an industrial scale. Dow Chemical Company's quotation as of May 1945 was \$480 per gallon; in June 1946 the price was \$140 per gallon. There is no obvious reason why such fluids cannot be produced at even lower cost if demand warrants it.

The migration of oil between the two pumps of a unit and the introduction of oil-equalizer lines to compensate for this migration has been mentioned previously. While this has been a satisfactory solution of the migration problem in existing pumps, it should be noted that there are serious limitations to its general application. Such a plan is limited to units in which primary- and booster-pump bases are at the same level. The method is hardly applicable in the case of boosters that operate at high boiler pressure, and the equalizer line precludes the use of different fluids in primary and booster pumps.

If, owing to one or more of the above conditions, the use of an equalizer line is impractical, oil migration may be reduced to an allowable rate by the use of interpump baffles or an effective refluxing design of interpump connection.

Loss of diffusion-pump oil to the fore-vacuum line has also occurred to an objectionable extent in some systems. Here again there is a choice of baffling or refluxing the discharge vapors. A discharge line so designed that all vapor condensing in the first few feet flows back into the pump should be quite effective, especially if this section of line is provided with water cooling.

Back-diffusion of oil into the high-vacuum region is a function of jet design, heating and cooling rates, and the degree of pump-head baffling. The allowable rate of back-diffusion depends on the extent to which oil vapor is permissible in the system, the separation of the pumps from the operating region, and the resulting depletion of the pump oil. All primary pumps were originally equipped with water-cooled baffles of such effectiveness that back-diffusion was well below the allowable rate in all installations. In seeking higher pumping speeds the possibility of operating pumps without baffles was explored since it was known that the simple removal of baffles would result in

a 20 to 30 per cent speed increase. Back-diffusion from unbaffled 20-in. pumps was found to be quite small and easily permissible in all Alpha I systems. Whether or not this is true in Beta systems, in which the pump-to-tank distance is much smaller, has not been determined though it appears probable that baffles could be eliminated even here.

A somewhat detailed study of oil back-diffusion from 32-in. pumps with and without baffles and equipped with jet assemblies of various designs, indicates that moderately tight baffling resulting in a speed reduction of about 20 per cent is required to reduce back-diffusion to an arbitrary 0.5 ml/hr. Without baffles, back-diffusion in the case of the highest speed jets exceeds 4 ml/hr, and oil depletion becomes a matter of concern.

Unquestionably the greatest problem that has arisen in connection with diffusion-pump operation has been that of oil changing. The problem is aggravated by the lack of any reliable test that can be readily applied to a given pump, or a sample of oil from the pump, to determine whether or not a change of oil is required. Consequently, the decision to change or not to change is frequently a matter of individual judgment based on the past history of the pump and the past experience of the one making the decision. A further complication arises from the fact that oil which has been definitely injured by exposure or contamination will, in time, recover if continued in use. The time required and expense involved in an oil change must, in such cases, be weighed against the estimated time required for recovery in use; and here again a decision must be made by some individual on the basis of his experience.

In early plant operation the experience necessary for making such decisions was almost wholly lacking. Consequently, when a need for oil change was suspected, and almost any difficulty in attaining operating pressure was considered evidence of such a need, a change was made on the theory that it could do no harm and might possibly help. As a result, much time and expensive oil was undoubtedly wasted in making unwarranted oil changes.

With growth in experience and improvements in operating technique, the frequency of oil changes, both necessary and unnecessary, decreased. In the summer of 1944, about a year and a half after the beginning of plant operation, oil was being changed at an average rate of once in 30 weeks for each operating pump. At the time of writing diffusion pumps were being given a routine servicing on a 12-month schedule. Oil was removed at this time for purposes of recovery of U 235 although the oil might still have been serviceable. The number of oil changes between servicing was, at that time, quite nominal.

Three primary causes for oil change may be identified:

1. Depletion. Pumps used in the plant are normally filled with oil to a depth of about 1 in. If, owing to back-diffusion or loss through the fore line, this depth is reduced to much less than $\frac{1}{4}$ in. it is advisable to replenish the oil supply. Unless depletion has been severe it is unnecessary to drain off any residue. Oil may simply be added to reestablish the required level.

2. Contamination. Contamination of the diffusion-pump oil, especially by water or high-vapor-pressure oil, is a common cause of defective pumping. In either case the chief pumping deficiency is an inability to attain normally low base pressures. But since common air leaks, water leaks, and outgassing produce the same effect, oil contamination is not readily detected. Also, since considerable time is usually required to determine the pressure attainable by a pump or with a given sample of oil, and since oil will be freed of these contaminants by prolonged operation, no practical direct test is available.

If pump oil is known to contain water its removal by operation may be accelerated by reducing the rate of heating to such a degree that little oil vapor is produced. Water still vaporizes readily at this reduced temperature, but the return of oil-entrapped water to the boiler is minimized. To a reduced extent, the same principle may be applied to the rapid removal of high-vapor-pressure oils.

3. Exposure. If hot diffusion-pump oil is exposed to air, oxidation and dissociation occur to a degree depending on the extent of exposure. Highly volatile constituents result, and these impair the ultimate pressure attainable. As with other highly volatile contaminants, these are discharged in time, leaving the oil fully recovered. Whether time for this recovery should be allowed or an oil change should be made as soon as exposure has been discovered can be estimated but never rigorously decided.

Because of large-scale production made possible by large demand, the cost of diffusion-pump oil has been reduced from \$36 per gallon at the beginning of plant operation to a present \$4.80 per gallon.

6.7 Diffusion-pump Servicing. The principal aspects of diffusion-pump servicing have been discussed in preceding sections and will merely be summarized at this point.

(a) Heater Replacement. When large numbers of electric heaters are operated continuously at input near or even above their normal rating, eventual failures are inevitable. Such failures in plant operation have been less frequent than was anticipated, and replacement is comparatively simple. The most serious phase of the heater problem is that of detecting failures promptly. Failure of the single booster-pump heater results in a pumping failure that usually leads directly

to detection. In the case of the larger pumps, however, two or more parallel heating circuits are used and failure of one circuit may impair without completely destroying the action of the pump. It follows that such failures may be overlooked for a considerable time unless revealed by some fortunate diagnosis or routine checks. A periodic check of all pump heaters appears worth while. This may be a power check or a pump-temperature check. The latter test is more easily made and has proven adequate with heater systems of existing types. In future installations serious consideration should be given to greatly extending heater life or to providing pilot lights or test jacks in all heater circuits.

Once a defective heater element has been detected its replacement may be made with little interruption of service. An operating tank may normally be carried by one diffusion pump while heater replacement on the other is being made, or often it is possible to schedule the replacement for the interrun period when diffusion pumps are out of service.

(b) Oil Changing. The problem of oil changing has been attacked primarily from the direction of reducing the frequency of change to a minimum both by insisting that oil be changed only when really necessary and by reducing the errors of operation that are the most common causes of change being required. In this way outage is reduced, economy is realized, and the fact that oil changing is a tedious operation is not very serious because it is seldom required.

Routine change of oil was made in the Beta systems on an 18-month schedule as a phase of material recovery. There was no evidence that this was necessary on the basis of pump performance, though without it a scheduled replenishing of pump oil would appear to be necessary.

(c) Scheduled Overhaul. The advisability of removing all pumps from the systems at arbitrary intervals for complete cleaning and servicing cannot be definitely evaluated. Certainly it appears that, barring accidents of operation and assuming that oil level is maintained, such overhaul might well be at very long intervals. Continuous operation for two years, or even longer, might reasonably be expected.

6.8 Diffusion-pump Tests. For purposes of discussion, distinction is made between those relatively simple tests that may be required to determine the normalcy of operation of diffusion pumps in service, the more extensive tests required to determine the conditions of optimum operation of a given type of pump, and the even more detailed tests required as a basis for pump comparison and improvement.

(a) Scope of In-service Tests. For the moment consideration will be given to tests of the first type only. Pumps of a given type are assumed to be in service in a vacuum system. Standard operating con-

ditions, such as rate of heating and cooling and kind and quantity of oil, have been adopted, and normal, though not necessarily optimum, performance characteristics under these conditions of operation have been determined. The tests considered are not those which might be required for justifying a change in operating conditions or for improving operating characteristics, but merely those required for determining whether or not operation is normal.

Such tests being in the nature of checks need be only of such accuracy as is required for revealing significant abnormalities in operation conditions or performance. Since the tests are applied to pumps in service, however, it is important that they be easily applied and readily interpreted.

Checks of heating rate, cooling rate, oil level, and oil quality may be made as preventive measures. Deficiencies are thus noted and corrected before performance is seriously impaired. On the other hand, these checks may be required to establish the cause of some observed shortcoming in performance.

(b) Heating Rate. The periodic checking of heating rate as a means of detecting defective heaters has already been mentioned. Deficient heating is commonly indicated by low limiting fore pressure, and sometimes by a sharp reduction in pumping speed.

(c) Cooling Rate. Rate of cooling is not critical within wide limits. Its chief effect is a slight elevation of ultimate pressure with rising temperature of cooling water. Complete stoppage of cooling water results in loss of vacuum, deterioration of pump oil, and excessive diffusion of oil from the pump. Flow glasses installed in the water lines of all plant pumps provide a simple and adequate check of cooling. Temperature of the cooling coil near its discharge end as determined by touch is also an adequate test. The coil may be warm but should not be uncomfortably hot. Reliance should not be placed on the temperature of the discharge line beyond the pump since this may remain cool in case of complete stoppage.

(d) Oil Level. The maintenance of the oil level within the rather wide limits permissible is accomplished largely by routine replenishment on a schedule based on operating experience. In the Westinghouse type of pumps the oil level may be checked by inserting a feeler rod in the oil refill tube. This requires, however, that the pump be cooled and brought to atmospheric pressure. Add to this the time required for reheating, and such a test can hardly be made in less than 2 hr. The float-type visual indicators provided with the D.P.I. 20-6 pumps avoid this difficulty, but as now built these devices have been the source of considerable vacuum trouble.

(e) Oil Quality. The only conclusive test of the quality of diffusion-pump oil appears to be a determination of its ability or inability to pump normally, a fact that is obviously of little value in diagnosing problems of subnormal pumping.

(f) Boiler Temperature. Boiler temperature may be considered as a definite operating characteristic of a diffusion pump. This can be readily checked, and the results are often informative. It should be realized, however, that boiler temperature depends not only on the rate of heating, but on local radiation and convection conditions, on pressure inside the pump, and on the boiling point of the pump oil.

The commonly questioned performance characteristics of a pump are its pumping speed, limiting fore pressure, and ultimate pressure. Of these characteristics, pumping speed requires checking more frequently than any other, and various methods are available for making this test.

(g) Speed Test by Comparison. In systems such as those of the Alpha I and Beta plants in which each tank is equipped with two identical diffusion-pumping units, a simple though not always definite test of pumping speed may be made by comparing the action of one pump with that of the other. Assume that the pressure in the system has been reduced to a value well within the ionization-gauge range but still high relative to the ultimate pressure attainable by the pumps. If P_1 is the value of this essentially stable pressure, then on closing the head valve to one of the pumps a new equilibrium pressure P_2 characteristic of the reduced total pumping speed is established. On opening this valve and closing off the other pump, still a third equilibrium pressure P_3 is obtained. If P_2 is approximately equal to P_3 , the speeds of the two pumps are essentially equal, and although it does not necessarily follow that the speeds are normal the probability that two pumps will be equally deficient in pumping speed is so slight as to be practically negligible. If on the other hand P_2 is greater than P_3 , the speed of the second pump to be closed off is definitely deficient, and the need for further tests to determine the cause of this deficiency is indicated. While this test is also applicable to a vacuum system of the Alpha II type, interpretation of results are complicated by the unequal pumping speeds out of the tank of upper and lower pumps.

The fact that the pressure attained by two pumps is usually greater than half that attained by either pump operating alone can be traced to the pumping action of the cold trap for condensable vapors. This action continues at an essentially constant rate regardless of pump-valve manipulation.

As tank pressure approaches the blanked-off pressure attainable by the pumps, and this may be quite high in the case of badly damaged oil, this test becomes indeterminate in that little gas is being removed and little pressure change results from the closing off of either pump. In a similar way little response occurs when the tank pressure is due solely to the presence of water vapor for little permanent gas is being removed and the speed of either pump is small compared with the total speed of pumps and trap.

In either of the above cases the low rate of pressure rise observed when both diffusion-pump-head valves are closed indicates that the high pressure is not attributable to low pumping speed.

(h) Speed by Rate-of-rise Method. If an equilibrium pressure P_1 is maintained in a system of volume V by pumps having a total pumping speed S and if on isolating the system from the pumps the pressure is observed to rise at a rate R_1 , then

$$S(P_1 - P_0) = VR_1$$

where P_0 is the base pressure of the pumps and where S may be in liters per second, P_1 and P_0 in microns of mercury, V in liters, and R_1 in microns per second.

If now a small air leak is introduced into the system, a new equilibrium pressure P_2 and a new rate of rise R_2 will be established such that again

$$S(P_2 - P_0) = VR_2$$

Subtracting the first equation from the second and solving for S ,

$$S = V \frac{R_2 - R_1}{P_2 - P_1} = V \frac{\Delta R}{\Delta P}$$

If leaks of a series of rates are introduced and the resulting rates of rise are plotted against corresponding pressures, the curve obtained is a straight line if the pumping speed is constant, and the slope, M , of this line is

$$M = \frac{\Delta R}{\Delta P} = \frac{S}{V}$$

Virtues of this method of speed determination are its independence of base pressure and of absolute pressure units. Although it was

suggested that P and R be expressed in microns of mercury and microns per second, respectively, it is obvious that any arbitrary pressure unit may be used as long as the same unit is retained throughout.

A practical limitation imposed on this method is that the numerical values of speed and volume should be of the same order of magnitude. If speed is very great compared with volume, a leak of such rate as to produce an easily measurable pressure increase will result in a rate of rise that is too rapid for accurate measurement. Conversely, if the volume is great compared with the speed, a leak that would produce a desirable rate of rise would elevate the pressure beyond the gauge limit.

Fortunately the volume-to-speed ratios in production systems fall well within these limitations, and the rate-of-rise method serves as a standard means of determining pumping speeds of complete systems and of individual pumps. If both gauges and adjustable leak valves are provided as standard equipment on each tank, no special equipment is required for these determinations. When tanks are not equipped with leak valves, such a valve must be temporarily installed for making the tests.

If results of rate-of-rise speed tests are to be directly comparable, the relative locations of leak and pressure gauge should be the same in all cases, for it should be noted that the speed determined by this method is the speed at the point at which the pressure is measured or at the point at which the leak is introduced, whichever is nearer the pump intake. Thus, if both leak and pressure gauge are on the tank it is the net speed of pumping from the tank that is determined. If either is placed near the pump intake, it is the speed at this point that is indicated.

(1) Speed by Metered-leak Method. The metered-leak method of determining pumping speed is the most versatile of all the methods commonly used. For this reason, it is the method generally favored for making detailed studies of pumping speed. In the routine checking of pumps in service, however, one of the preceding methods is usually adopted as simpler in application and requiring less specialized equipment.

If an air leak of measured rate r is admitted to a system in which an initial equilibrium pressure P_0 is maintained by pumps of total speed S , a new and higher equilibrium pressure P_1 will be established. The increase in pressure $(P_1 - P_0)$ is directly proportional to the leak rate and inversely proportional to the pumping speeds,

$$(P_1 - P_0) = K \frac{r}{S}$$

If r and S are expressed in common units (liters or milliliters per second, cubic feet per minute), continuity of flow at pressure equilibrium requires that $K = P$, the pressure at which the leak rate is measured, and that this pressure be expressed in the same units used in measuring P_0 and P_1 .

$$P_1 - P_0 = P \frac{r}{S}$$

Since the leak rate is usually measured in milliliters per second at atmospheric pressure b (in millimeters of mercury), then P_1 and P_0 may be measured in microns of mercury, and speed will be given in liters per second;

$$S = \frac{br}{P_1 - P_0}$$

may be taken as the working equation for speed determinations by this method.

To apply this method to the determination of pumping speed it is necessary to provide (1) a method of introducing a controlled leak into the system, (2) a means of measuring the rate of this leak, and (3) a means of measuring the resulting equilibrium pressures.

In operating units of the production plant the required pressure measurements are taken care of by the gauge with which each unit is normally equipped.

Neither fine nor wide adjustment of the leak rate is required since a single speed determination at any pressure within the operating range of the pump provides an adequate check of its performance. Any one of a number of types of needle valves are satisfactory for this purpose. For descriptions see Sec. 7.4b. While the permanent installation of such a valve on each unit facilitates the making of tests, a suitable needle valve may be temporarily installed on any available instrument valve at no great inconvenience.

The rate of leakage is usually measured with an oil manometer, three types of which are shown in Fig. 1.16. While these instruments are admirably adapted to the more elaborate laboratory or test area tests, their use in the plant is often inconvenient, and it is not possible to utilize fully their wide range. For these reasons, consideration has been given to less cumbersome devices that might meet the limited requirement of plant testing.

A rotameter of suitably low range may be used in place of the oil manometer. The use of any timing device, and this is a complicating

factor when working near the plant magnet, is thus eliminated. By combining a suitable leak valve with the rotameter a compact instrument suitable for temporary attachment to any system is provided.

Unfortunately, however, experience with a number of these instruments shows that their indications are somewhat erratic at rates of flow below 0.5 ml/sec (S.T.P.) and that considerable care in their operation is required even at higher rates of flow.

Fig. 1.16—Oil manometers.

Use of a calibrated fixed leak of a suitably chosen rate offers obviously desirable features. Such a leak connected permanently or temporarily to a system through a simple instrument valve would admit air at its calibrated rate r and the only measurements necessary for a speed test would be an observation of equilibrium pressures with the leak open to the system and with it closed off. However, of the various types of fixed leaks that have been examined, none has been found that can be relied upon to hold its calibration indefinitely. Filtering the air passing into the leak and utilizing a porous plug rather than a single fine capillary or orifice reduce the

chance of calibration change, but still, frequent calibration checks appear necessary.

Speeds determined by the metered-leak method depend on the position of leak and gauge relative to the pump intake exactly as in the case of the rate-of-rise method.

In general, the speed checks made on operating diffusion pumps do not aim at any high degree of accuracy. Speed deficiencies of a few per cent are not significant; and fortunately, deficiencies that are significant are usually very great. Thus, the speed of a pump may be expected to be essentially normal or else very small, e.g., 25 per cent of normal or less. Obviously, a high degree of accuracy is not required to differentiate between these two extremes.

(j) Limiting Fore Pressure. In large vacuum systems, equipped with a full complement of gauges and valves, various methods of checking the limiting diffusion-pump fore pressure are available.

Perhaps the simplest method consists of observing, with a McLeod or Pirani gauge, the essentially constant value of fore pressure that is obtained during the initial period of evacuation of the tank by the diffusion pumps. This observed pressure is the limiting fore pressure of the pumps when operating at full load. To be conclusive the test must be made on individual pumps and not on pairs of parallel pumping units.

The same results may be obtained by introducing into an evacuated tank a series of leaks of increasing rates and noting the limiting fore pressure at which a sharp rise and instability of high-vacuum pressure occurs.

In a system that has been evacuated to a stable pressure, the limiting fore pressure of the diffusion pumps may be determined by closing the valve in the fore vacuum line at the fore pump, and again noting the fore pressure at which high-vacuum pressure rises sharply and becomes unstable. If the pressure in the tank is low and little gas is being removed by the pumps, the limiting fore pressure may not be reached for a number of minutes after the fore-vacuum valve is closed. In this case the static fore-pressure limit is indicated. If, on the other hand, the pressure in the tank is high and gas is being removed at a considerable rate, the pressure in the fore-vacuum region will reach the limiting value very quickly. It is even possible that partial closing of the fore-vacuum valve may so reduce the effective fore-pumping speed as to allow the fore pressure to rise to the limiting value. The fore-pressure limit, in such a case, is that appropriate to the particular rate at which gas is being handled by the pump.

Application of any but the first of these methods to large industrial installations is often rendered inconvenient when high-vacuum and fore-vacuum gauges are in different rooms or even on different floors. Since variations in high-vacuum pressure serve to indicate the attainment of limiting fore pressure, it is necessary, in such cases, to establish direct communication between observers of the two gauges.

The use of a properly calibrated Pirani or thermocouple gauge for measuring limiting fore pressure is recommended because these gauges are continuous reading and because they indicate approximately the total pressure due to both permanent gases and condensable vapors. McLeod-type gauges, on the other hand, must be read intermittently, and may be in error by 100 per cent or more if much water vapor is present.

(k) Ultimate Pressure Attainable. Perhaps the most difficult problem of diffusion-pump testing is that of determining whether or not the ultimate vacuum currently attainable by a pump is normal, or even adequate for producing the pressure required for operation. Usually this question arises after exposure of the pump oil to air, or some similar abuse, is known to have occurred, or after prolonged effort has failed in the attaining of working pressure in a system.

The fact that residual leakage and outgassing result in the establishment of an equilibrium pressure well above that attainable by the pump makes it difficult, if not impossible, to determine directly, and in a reasonable time, the pressure that a given pump is capable of attaining. The providing of an ionization gauge attachment below the pump-head valve and above the baffle allows pressure to be measured in the head of the isolated pump. This largely eliminates the effects of residual leakage and outgassing of the system being evacuated, but places the gauge in an exposed position relative to oil vapor. Moreover, considerable time is required for evacuating and outgassing the gauge if it is put into service only for making this test. Even if it were possible to determine by some simple method the pressure attainable by a pump, this would only partially solve the problem in case of subnormal performance. Defective ultimate pressure may usually be attributed to defective pump oil, and this is a defect from which recovery may be made in the process of normal operation. The time required for such recovery varies from a few minutes to several days, however, and a means of estimating the time required for recovery is needed if cases of defective pumping are to be handled most effectively.

Since direct methods of detecting and diagnosing poor ultimate pressure are lacking, these problems must be dealt with by indirect

methods. The results of such treatment are often indefinite and sometimes confusing. Poor ultimate pressure may be suspected in a system having a high equilibrium pressure associated with a low rate of rise. However, this condition is also symptomatic of a water leak in the system. It frequently happens that the only evidence of poor ultimate vacuum ever obtained is the improvement observed when some corrective measure is taken.

Although the demands for checks of pump performance may be imperative when they do arise, it should be realized that such demands are not frequent in a well-operated system. If not subjected to operational abuses, oil diffusion pumps may reasonably be expected to give continuous, trouble-free service and to maintain their rated characteristics for months or even years of operation.

7. VACUUM VALVES

In any large multiple-unit vacuum system designed for intermittent operation the vacuum valves necessary for efficient operation comprise a major equipment item. Prior to the time of designing the electromagnetic separation plant, few valves suitable for vacuum installation, and none of large size, were commercially available. Certain commercial types of valves, especially in the 2-in. size, had been modified for vacuum purposes, and shop-built valves in larger sizes had been designed to meet particular requirements. Although none of these valves were entirely adaptable to plant requirements, many details of their construction, such as the Wilson-type stem seals and the vulcanized disk washers, were carried over into the design of plant valves.

The various valves used in the plant vacuum systems may be designated according to their functions as (1) pump-head valves, (2) vacuum-line valves, (3) instrument valves, and (4) air-inlet valves. Within each of these categories, valves may be further classified according to size or type.

7.1 Pump-head Valves. These valves, placed on the intake side of all diffusion pumps, are necessary in order that the pumps may be maintained in operation during those periods for which the tank pressure is above the diffusion-pumping range. When closed they must provide a vacuum-tight seal and withstand a pressure difference of an atmosphere or more if the higher pressure is from above the valve disk. It is not necessary, however, that they withstand an equal pressure difference in the opposite direction; nor is it necessary that it be possible to open the valve against a large pressure from above. When opened, the high-vacuum conductance through the valve must be

large relative to the speed of the diffusion pump. Otherwise, effective pumping speed is substantially reduced. Mechanically the valve should open and close quickly and without undue effort on the part of the operator. Theoretically the valve need be operated only at the beginning and end of a run, but actually it is used much more frequently, as in taking rates of rise, and there are accidents of operation that require the closing of one or more of these valves as quickly as possible.

Two models of pump-head valves are used in the plant. They were designed for use with the 20-in. and 32-in. pumps, respectively, and are designated by their opening sizes. Both of these, and also the 6-in. vacuum-line valve described later, were designed by Stone and Webster in collaboration with UCRL, and all valves of these three types were built by the Chapman Valve Company.

(a) Chapman 20-in. Valves. Each of these units, see Fig. 1.17, consists of two disk-type valves mounted in a common housing. This housing forms the terminal section of the rectangular duct through which the tank is evacuated, and a 20-in. pump is connected directly beneath each of the valve openings.

The valve is actuated by the movement of an endless chain running over a grooved wheel keyed to a shaft projecting through the side of the valve housing. The rotation of this shaft is transmitted through bevel gears to a screw movement that raises or lowers the valve disk. Guide rods prevent rotation of the disk during travel and guide it into proper seating position. When the disk is seated, closure is formed between a Neoprene (Hycar in later models) insert, vulcanized into an annular groove machined in the lower disk face, and a matching seating land. A centrally spaced ridge running the length of the seating land provides high pressure, line contact, and assures positive sealing. The drive shaft projecting through the housing wall is equipped with a Wilson-type vacuum seal.

Several defects of these valves, as originally designed and built, became apparent with extensive use:

1. Owing to low screw pitch and drive-gear ratio, the travel was much too slow.
2. Higher lift and wider valve opening than necessary was provided.
3. Excessive bearing friction in some individual assemblies rendered operation difficult.
4. Need was felt, especially in the early period when operators were inexperienced, for some positive indication of valve setting.

The first three defects were mitigated, if not eliminated, in the later quick-operating models by the introduction of high-pitch screws and a higher gear ratio, by shortening the lift range, and by the sub-

Fig. 1.17—Chapman 20-in. angle valve.

stitution of needle and ball bearings for the plain bearings previously used. With these improvements, the valves became quite operable. Nevertheless, even more rapid operation was desirable.

At the same time that the above modifications were being worked out, a new and radically different valve mechanism was designed which might be substituted in existing housings to utilize many existing valve parts. A single model of this valve was built, and it has given very good service in the vacuum laboratory for the past two years or more. Plant adoption of this valve was never seriously considered because the modification described in the preceding paragraph gave adequate improvement and could be safely adopted without extensive experimental tests. Because of its possible value in future design, however, the new valve is illustrated in Fig. 1.18. Travel through the complete $7\frac{1}{2}$ -in. range is obtained by about 5 ft of chain travel. An additional foot of chain travel is required for seating or unseating, and it is at this stage only that any considerable force must be applied to the chain. Thus, this valve is both free and rapid in operation. Its chief limitation appears to be the limited lift obtainable.

Various position-indicating devices have been proposed and installed on an experimental basis. The need for such devices diminished as operating procedures and experience on the part of operating personnel increased, and no general installation of these devices was ever undertaken.

All the 20-in. valves on one Beta track were converted to motor operation by the installation of Limitorque mechanisms in place of the chains and drive wheels. This arrangement has proved completely successful. All valves are controlled from a central station, operational errors are minimized, and personnel requirements are reduced. Plant-wide conversion along this line was not considered justified primarily because of the considerable cost involved and the indefinite time for which operation of the plant might be continued.

Mechanically, all the 20-in. valves have functioned very well. Servicing requirements are moderate and replacements have been minor. Gears and bearings are lubricated periodically with a low-vapor-pressure (diffusion-pump) oil or a graphite paste. Seating washers in the valve disks show some slight deformations with use, but no appreciable wear or rupture. Some early difficulty was experienced in servicing shaft seals, largely because of the close fit of metal seal parts in the seal recess. Increasing the tolerance of this fit has effectively relieved this condition. Use of the oil-sight glasses on these, as well as on other vacuum seals, has been largely discontinued.

(b) Chapman 27-in. Valves. This valve, see Fig. 1.19, was designed for use with 32-in. pumps in the Alpha II plant.

Some desirable features of its design are (1) freedom and speed of action, (2) absence of internal mechanism and adjustments, and (3) back seating at the stem seal.

The chief criticisms that can be brought against it are (1) limited lift, and (2) complexity of operating mechanism and resulting high costs.

In service these valves have performed very well. Stem seals require about the same attention as in other types of valves. Seating and back-seating adjustments are rarely required, and no instance of a major failure is known.

Shortly before the closing down of the Alpha II plant, thought was being given to the possibility of increasing the effective speed of the pumping systems by the installation of 32-in. jet assemblies of higher speed. In this connection it became apparent that only limited advantage would be gained from these high-speed jets unless higher conductance through the 27-in. valve could be provided, especially in the case of the upper pump. To this end the pneumatic lift shown in Fig. 1.20 was designed by Stone and Webster, and a single experimental model was built and tested in the vacuum laboratory. Designed with a view to easy installation on existing valves, this lift gives rapid, positive action, and has been quite free of operating trouble. Head room required for the pneumatic cylinder is the most obvious limitation to this type of lift, otherwise, the possibility of realizing quick action, high lift, remote control, and dependable operation in a relatively inexpensive lift mechanism makes it appear very attractive.

7.2 Vacuum-line Valves. This category includes all valves in the roughing, finishing, and crossover lines, and in the various vacuum headers. To be generally useful they must have vacuum-tight stem seals and must hold effectively against pressure differences of an atmosphere or more in either direction across them. Their size and distance of opening must be such that they do not seriously limit the conductance of the line in which they are installed.

(a) Chapman 6-in. Angle Valve. Valves of this type are, with a few exceptions noted later, used in all low-vacuum line installations. As shown in Fig. 1.21, the valve body is a standard 6-in. welding tee. High lift with slight stem travel is provided by the compound screw mechanism. Seating to back-seating movement is obtained by eight to nine turns of the stem. Valve-seat and disk-washer construction is similar to that used in the pump-head valves; also, the same type of

Fig. 1.18 — Crank arm lift for 20-in. valve.

Fig. 1.19—Chapman 27-in. valve.

Fig. 1.20 — Pneumatic lift for Chapman 27-in. valve.

Fig. 1.21 — Chapman 6-in. angle valve.

stem seal is employed. Hand-wheel or endless-chain operation is optional, depending on accessibility of the valve as installed.

Except for minor tolerance errors in a few of the first valves built, these valves have rendered excellent trouble-free service. Replacement and servicing requirements have been very moderate.

In early installations sufficient attention was not given to the removal of all free foreign particles from the vacuum-piping system, and the trapping of these particles in valves sometimes resulted in jamming of the screw mechanisms and marring of the disk washers. In later installations such failures were prevented by giving all lines and valves a thorough cleaning with a wire brush and compressed air prior to initial operation of the systems.

Basically, the design of these valves is sound. However, somewhat faster travel could be usefully provided and less massive construction of some of the parts appears desirable. The practice of welding valves directly into the vacuum lines, thus eliminating flange and gasket connections, is definitely commended.

In order to facilitate production this type of valve was built in the 6-in. size only. As a result, 6-in. valves are frequently used where a valve of smaller size would be adequate. For the most economical construction of vacuum systems of the present scale this or some similar type of valve should be available in a 3- or 4-in. size.

(b) Kinney 2-in. Globe Valve. A number of these valves were used in the vacuum systems of test docks and other secondary installations. As shown in Fig. 1.22a, a standard globe valve is modified for vacuum service by

1. Substitution of a sylphon bellows for the usual stem packing seal.
2. Use of a rubber or synthetic washer.
3. Provision of a lead gasket for making the bonnet-to-body seal.

Experience with these valves has not given very favorable results. Some of the disadvantages are:

1. Periodic tightening of the lead gasket seal is required.
2. The soldering of the bellows is sometimes defective.
3. The strain to which the light-weight bellows is subjected at the time of seating or unseating and especially the strain that results if the bonnet is tightened or loosened with the valve seated, is often sufficient to twist and rupture the bellows.

(c) Chapman 2-in. Angle Valve. This valve, shown in Fig. 1.22b, may be described as a standard 2-in. steel body equipped with a stem assembly of a modified 6-in. valve type. These valves were installed in all Alpha II roughing-to-finishing header cross-connect lines and their performance in service has been satisfactory in every respect.

The rugged construction of these valves as contrasted with valves of the type described in the preceding section, appears to be necessary for dependable industrial operation.

(d) Fulton Packless Valve, Model 304 N-V. This valve is a modification of a poppet-type globe valve originally manufactured for use in handling gasoline and other highly volatile liquids. The principal modification, as shown in Fig. 1.22c, is the substitution of a disk with Hycar washer, and a matching seat insert for the original poppet assembly. Bonnet assembly seals are metal to metal with shellac or glyptal coatings. The same type of bonnet-to-body seal has given some trouble; however, the design is such as to be readily adaptable to the use of a rubber gasket at this point. Use of a heavy-weight bellows and provision of a stem guide to relieve the bellows of torque makes this valve much more rugged than the similar bellows-sealed Kinney valve.

Although eventual rupture of the bellows in a valve of this type is certain, hundreds of these valves have been in service over a period of one to two years or more, and considerably less than 1 per cent replacements have been required as a result of all bellows failures, natural and otherwise. Valve-disk failures have been more frequent. Disks of oil-resistant material with a high durometer reading (Neoprene, Hycar, Koroseal, or Teflon), and not less than $\frac{3}{16}$ in. in thickness, are advised.

Valves of this type are available in $\frac{1}{2}$ -, 1-, and 2-in. sizes. The 2-in. size is used in Beta crossover lines and in various test system installations. The smaller sizes are used in test systems, experimental systems, and for various minor applications.

Except as noted above, the performance of these valves has been highly satisfactory. They represent, within their size range, the most economical type of vacuum valve that has been extensively used in the plant.

7.3 Instrument Valves. Included in this classification are all those valves provided for the attachment of gauges and other accessories to the vacuum system. The use of valves in making such attachments is recommended as providing the maximum freedom in installing, isolating, and removing such accessories, with a minimum disturbance of vacuum conditions in the system.

(a) Kerotest $\frac{3}{8}$ -in. Angle Valve (Type 797). These diaphragm-type refrigeration valves, Fig. 1.23a, were adopted for plant use on the basis of availability and their record of satisfactory service in earlier vacuum systems. The only modification required, and this was a matter of convenience rather than necessity, was the substitution of

hand-tightened fittings for the SAE-type fittings normally provided at the side outlet. Seat inserts of Micarta, Neoprene, and silver were used at one time or another. The silver inserts were unsatisfactory

Fig. 1.22a—Kinney 2-in. globe valve.

because of faulty sealing, a condition that became progressively worse with deformation of the silver insert by use. Seating with the Neoprene inserts was positive and required only moderate closing force; however, these inserts were damaged by excessive tightening, and their greater deformation imposes greater stress on the sealing dia-

Fig. 1.22b—Chapman 2-in. angle valve.

phragm. Consequently the Micarta inserts have come into almost universal use. These disks stand up well in service, withstand temperatures of 200°C or higher, and seldom fail to give a vacuum-tight seal. The great closing force sometimes required to give a tight seal

Fig. 1.22c—Fulton 2-in. packless valve.

with these disks is their most serious shortcoming. The substitution, on an experimental scale, of Teflon for Micarta washers appears to improve this condition without loss of the desirable features of the Micarta.

The average life of both disks and diaphragms is great. Many valves are now in their third year of continuous service. Also, replacements of these parts, when required, is a simple operation.

Within their capacity range and in applications in which the residual-leakage tolerance is moderately high as in plant systems, they are highly useful vacuum valves. Applied to low-speed high-vacuum systems, however, leakage both through and into these valves is often so great as to preclude their use. It is for this reason that Kerotest valves have been replaced by Fulton $\frac{1}{2}$ -in. valves in leak-detector systems.

(b) Chapman 1-in. Angle Valve. The installation of liquid-nitrogen traps at the ionization gauges required the placing of a valve between the trap and tank. Two types of valves were used for this purpose: the Fulton model 304 N-V valve previously described and a modified 1-in. angle valve designed and built by the Chapman Valve Company and shown in Fig. 1.23d.

Trouble with these valves as originally installed arose from two causes:

1. They were not thoroughly cleaned after being allowed to rust in storage.
2. The stem-packing material was not fully impregnated with vacuum grease.

In the Beta plant these causes of trouble were removed by cleaning and repacking all valves, and subsequent service has been fully equal if not superior to that obtained with valves equipped with the more elaborate Wilson stem seals. In the Alpha I buildings, on the other hand, use of the valves was discontinued after a short time partly because of these defects, which could have been overcome simply, and partly because of dissatisfaction with the use of the associated liquid-nitrogen traps. Thus from one part of the plant there comes a highly unfavorable report on a piece of equipment that has been shown, in another part of the plant, to render very satisfactory service.

7.4 Air-inlet Valves. The requirements applying to the air-inlet valve are much less severe than those imposed on the valves previously discussed. In general the air-inlet valve needs to be vacuum tight on its closed side only; stem sealing, beyond that normally obtainable with a gland packing, is not required. Thus any valve that can be closed to form a vacuum-tight seal and can be opened against atmospheric pressure meets the main requirements of such a valve.

(a) Dry-air Inlet Valves. For breaking the vacuum in a process tank, dry air is admitted through a 1-in. line. Any disk-type globe or angle valve with conventional gland packing is satisfactory for this purpose so long as the valve is installed with the stem side away from the vacuum. Poppet and gate-type valves, relying on metal-to-metal closure, are not satisfactory. Moreover, the valve washer should be

Fig. 1.23—See facing page for legend.

Fig. 1.23—(a) Kerotest $\frac{3}{8}$ -in. angle valve. (b) General purpose needle valve. (c) Leak detector calibration needle valve. (d) Chapman 1-in. angle valve.

moderately soft and oil resistant. Hard fiber washers frequently fail to seat tightly. The washer should be sealed into the disk to avoid air leakage past the back side. Valves having removable seat inserts must be examined for leakage along the insert thread. Valves having one-piece cast brass bodies have been found uniformly free of porous leaks within the requirements of the systems here considered.

(b) Leak Valves. There are a number of conditions under which it is desirable to introduce air, or some other gas, to a vacuum system at a controlled rate. Making speed measurements, adjusting operating pressure, and calibrating helium leak detectors are three examples of this. While the demands placed on the leak device may vary somewhat from one application to another there are certain characteristics which any good, all-purpose leak valve should have. The characteristics may be listed as

1. Air-tight closure without injury to the closing mechanism.
2. Fine adjustment over an adequate range with uniform variation of leakage rate with adjustment.
3. Positive opening and closing, and freedom from excessive backlash.
4. Close agreement with its calibration for a given leak rate.
5. Accessibility for cleaning.
6. Moderately tight, but not necessarily vacuum tight, stem seal.

These various requirements are more nearly met by some form of needle valve than by any other device. Although commercial needle valves such as those made by Hoke and Imperial Brass Company are of limited usefulness, they sometimes fail to close positively, and their adjustment is usually more coarse than is desired. Specially designed valves have in general given superior service. Figures 1.23b and c show two valves of this type developed by A. J. Samuel in the CEW-TEC Vacuum Laboratory. These are of recent design and while typical of a number of earlier models they incorporate certain improvements over these. The first is a wide-range all-purpose valve suitable for speed measurements and for pressure control. The second is a fine-adjustment low-leak-rate valve designed especially for leak-detector calibration but useful also for pressure adjustment. Provided with screwdriver adjustment, position-locking nuts, and inlet air filters, valves of this type have also been used as standard leaks adjustable at the time of calibration to any desired rate. Readjustment to calibration is simply provided, but periodic checking is required to determine need of calibration.

Generous stem size, effective stem seal, low pitch threads, and needle stop nuts are good design features of these valves. The use of

a stainless-steel needle is good from the standpoint of corrosion resistance, but a harder steel would be desirable. Everdur is preferable to brass for the valve body because it resists seat damage from the wiping action of the seating needle. Terminal fittings are, of course, optional.

For satisfactory performance, these valves must be precision built. Unless parts are machined to close tolerance, individual assembly and testing will be required. If properly built and assembled, however, smoothly controlled leaks ranging from several milliliters per second down to less than 0.005 ml/sec can be obtained.

The use of leak valves in speed measurements and in leak-detector calibration is discussed in other sections of this report. Their use in controlling operating pressure might well be outlined at this point.

The need to control operating pressures arose from the observation that beam interaction is much less common at operating pressures of the order of 0.04μ Hg than at lower pressure. Thus when interaction due to low pressure occurs, a leak of sufficient rate to raise the equilibrium pressure to its optimum value is introduced. In early installations these leaks were introduced through needle valves in the unit face plates. Valves similar to the one shown in Fig. 1.23b were used for this purpose in the Alpha I and Beta systems. Valves designed and built by Westinghouse were supplied with the Alpha II units.

These Westinghouse valves did not prove very satisfactory in service. The overtightening required to ensure closure often resulted in sticking of the needle or jamming of the stem thread. Most valves developed considerable backlash, and as a result individual valves operated quite differently.

Aside from the difficulties arising from defective needle valves, however, it soon became evident that pressure control by the leak method could never be fully effective until it was possible for the leak adjustment to be made by the same operator who watched the pressure gauge. That is, the leak must be controlled from the operating cubicle either by remote control or by placing the needle valve at the cubicle.

Throughout the Beta plant leak valves were installed at the operating cubicles. Valves were connected to their respective tanks by copper tubing of $\frac{1}{4}$ in. I.D. Preliminary experiments indicated that response time through 100 ft of tubing of this size is 40 sec, whereas for an equal length of $\frac{1}{2}$ - or $\frac{3}{8}$ -in. tubing it is approximately twice as great. Response time is here defined as the time elapsing between the opening or closing of the leak valve and the attaining of a pressure in the tank equal to 80 per cent of the equilibrium pressure.

As a safeguard against the opening of an excessive leak, a limiting fixed leak of approximately 1 ml/sec (S.T.P.) was installed between the needle valve and the tank. This limiting leak consisted of approximately 6 in. of No. 27 hypodermic-needle tubing, approximately 0.008 in. I.D.

In spite of the response lag due to the length of connecting tubing and the necessity for periodic cleaning of both valve and fixed leak, pressure control from the cubicle was quite practical and far preferable to the original two-man operation.

8. VACUUM JOINTS, CLOSURES, AND SEALS

8.1 Vacuum Joints. In the construction and assembly of large industrial vacuum systems, the component parts may be permanently joined together by welding, brazing, or soldering; or breakable joints, such as provided by threaded pipe fittings or gasketed flanges, may be employed. As a general rule, joints of the latter type should be avoided except as demanded by operating and servicing requirements.

(a) Permanent Joints. In systems built of steel, arc welding is the favored method of forming permanent joints, although oxyacetylene welding can give equally good results. Such welds are mechanically strong, and can be quite free of leaks if skillfully made. Testing and correcting for leaks that may occur is not usually difficult and welds that have once been made tight are quite free from subsequent defects.

Joints in all main vacuum lines of the plant, including the installation of valves, traps, and fittings were made by arc welding, except for the few flange and gasket joints provided for the removal of parts requiring servicing.

The use of backing rings in making pipe joints is recommended, as well as the use of threadolettes in attaching small size pipe to large equipment.

For joints between nonferrous and small steel parts, brazing or soldering may be used. Soft solder should be used only in cases requiring little mechanical strength or those in which temperature considerations preclude the use of hard solder. Joints of these types, like welded joints, can be very good if skillfully made.

Brazing or soldering is frequently used to supplement and ensure tightness of threaded joints, as in the installation of many small valves and fittings. Joints in water-cooling lines internal to the vacuum system are hard-soldered. Oil-equalizer lines on the diffusion pumps were a source of trouble until they were welded or soldered in place.

(b) Breakable Joints. Gasketed flange joints are generally used for joining major parts of the vacuum system and for providing breakable joints in the large-size vacuum lines. Matching flanges, with a suitable gasket between, are drawn together tightly by bolts passing through them. Thus a strong, rigid, vacuum-tight joint is formed. Obviously the tightness of such joints depends, in a large measure, on the gaskets used. Since gaskets of the same type used in flange joints are also used in making airtight closures, as at the faceplates and cold traps, these will be considered in some detail.

A strip-type gasket of rubber or synthetic rubber and of square cross section is held in a suitably dimensioned endless groove machined in one of the two matching flange or closure surfaces. This is the holding gasket. To provide a means of testing its tightness, a second gasket held in a similar groove is placed outside the first and a test opening from the space between the gaskets to the outside is provided. A V-shaped groove between the two gasket grooves ensures against stoppage of the intergasket test space. Leakage past the holding gasket into the evacuated space may be tested for by:

1. Observing a soap film covering the test opening.
2. Observing the pressure drop in the system when the intergasket space is evacuated by an auxiliary pump.
3. Introducing a suitable detector gas into this space and observing the response of ionization gauge, Pirani gauge, or leak detector.

Figure 1.24a shows a typical arrangement of double gaskets and test opening, together with the groove dimensions adopted for gaskets of the sizes used in the plant.

With reasonable care in assembling, gaskets of this type are remarkably free of leaks, and undisturbed joints remain tight. Of almost five hundred large-gasketed joints between tank ducts and valve chambers in the Alpha I plants, none have become defective in two to two and one-half years of service. When such joints are frequently broken, however, as in the case of faceplate and cold-trap seals, gaskets must be renewed frequently because of their deformation under pressure. The present estimated useful life of these gaskets is seven or more operating cycles. It should be pointed out that this is certainly less than the true life since the practice is to replace gaskets when it appears that defects are likely to develop with further use. Thus gaskets are rarely retained to the limit of their useful life.

The extrusion of the gasket beyond the groove chamber, even to the extent of producing longitudinal rupture, is a serious defect of this type of gasket. It appears that by increasing the groove depth and chamfer cut and by using a gasket material of relatively high hardness and low permanent set, it should be possible to obtain tight sealing at

metal-to-metal contact without extrusion of the gasket beyond the groove limits. Such an arrangement would undoubtedly result in a substantial increase in gasket life.

GASKET SIZE	WIDTH	DEPTH	CHAMFER
$\frac{3}{8} \times \frac{3}{8}$	0.360	0.200	$\frac{1}{16} \times 45^\circ$
$\frac{1}{2} \times \frac{1}{2}$	0.370	0.210	$\frac{1}{16} \times 45^\circ$
$\frac{3}{4} \times \frac{3}{4}$	0.235	0.145	$\frac{1}{32} \times 45^\circ$
$\frac{1}{4} \times \frac{1}{4}$	0.245	0.155	$\frac{1}{32} \times 45^\circ$
$\frac{3}{16} \times \frac{3}{16}$	0.175	0.105	$\frac{1}{32} \times 45^\circ$
$\frac{1}{8} \times \frac{1}{8}$	0.180	0.110	$\frac{1}{32} \times 45^\circ$
$\frac{1}{8} \times \frac{1}{8}$	0.115	0.085	$\frac{1}{64} \times 45^\circ$
$\frac{1}{8} \times \frac{1}{8}$	0.120	0.090	$\frac{1}{64} \times 45^\circ$

Fig. 1.24—(a) Double gasket grooves and pump-out. (b) Planer-cut groove corners and inserts.

The machining of gasket grooves adds considerably to the cost of manufacturing joint and closures for this type of seal. This is especially true in the case of noncircular openings. Grooves in circular flanges or plates can usually be lathe cut and present little difficulty. Around rectangular openings, however, the grooves must be milled or planer cut. Milling is slow, but milled grooves can have unbroken, rounded corners and one-piece endless gaskets can be fitted into them. Planer-cut grooves are cut the full length of the work, however, and

(a)

(b)

(c)

(d)

Fig. 1.26—Insulating water-line couplings (a,b,c). (d) Ionization-gauge fitting.

Fig. 1.27—Hand-tightening instrument fittings.

The problem of attaching ionization gauges to large multiple-unit vacuum systems assumes major proportions when it is realized that such attachments must be free from leakage and outgassing, and must permit quick and easy replacement of defective tubes. Original plans contemplated the use of hand-tightened fittings of the above type for making these connections. A tube mounting sleeve, with its nut in place, was to be wax-sealed to the gauge tubulation, and this could be attached directly to the Kerotest outlet. For a number of reasons, this plan was never fully carried out. For one reason, service facilities for making up and pretesting the waxed seals could not be set up rapidly enough to meet the heavy demands resulting from short tube life in early stages of operation. Another reason was that the fittings provided limited the bulb tubulation size to $\frac{7}{16}$ in. O.D. or less, and a larger tubulation was desired in order to facilitate outgassing of the tube.

The ionization-gauge fitting shown in Fig. 1.26d was developed largely to overcome these difficulties. This is an adapter fitting designed to mount directly on the existing Kerotest fitting and to provide a rubber compression seal for the enlarged gauge tubulation. Although these fittings have served the purpose for which they were designed, their use has not been trouble free. Breakage of tubulations while installing or removing tubes or when tightening the compression nut has been a common source of trouble. The use of vacuum grease in making the assembly reduces breakage but introduces objectionable vapors into the gauge. The hand-tightened connection of the adapter body to the Kerotest fitting is a possible source of leakage, and also requires that the adapter be held to prevent turning when the compression nut is tightened or loosened. Rigid mounting of the seal body and substitution of a Wilson-type seal for the compression seal would be desirable in future installations.

8.2 Vacuum Seals. Vacuum seals, as the term is here used, fall naturally into two classes: (1) seals through which mechanical motion is transmitted to within the evacuated space and (2) seals through which electrically insulated lines pass into the evacuated space. Simple installations, such as the passage of an uninsulated water line through the wall of a vacuum system, are not considered since they give rise to no particular problems. Such a line may be welded, brazed, soldered, or threaded into place, and presents the same problems as other vacuum joints of these types.

(a) Mechanical Seals. Seals permitting the transmission of mechanical motion into the vacuum system are found at valve stems and shafts, at source- and receiver-adjustment controls, at removable

K-locks, and as parts of slit-cleaner and viewing-window shutter mechanisms. In the original designs of equipment, Wilson-type seals were used at all these points, and, although subsequent adoptions of seals of other types have occurred, this type of seal is still most commonly used for the transmission of mechanical motion.

The Wilson² seal depends for its sealing action on the high pressure along the line of contact between a properly dimensioned and deformed rubber washer and the surface of the shaft passing through it. High pressure contact is ensured by the elasticity of the washer, atmospheric pressure, and small area of contact between washer and shaft. At the same time the shaft, if properly lubricated, is relatively free to move in the seal. Motion may be rotational, reciprocating, or a combination of the two. Also, by proper design of the seal housing a limited rocking or nodding motion may be transmitted.

Figure 1.28 shows a standard Wilson-seal assembly typical of those used throughout the plant. A second seal and test space outside the holding seal provides for ease in testing. Table 1.9 lists dimensions and tolerances for the component parts of assemblies in a series of standard sizes.

Wilson seals used in the plant range in size from $\frac{1}{4}$ to $1\frac{3}{4}$ in. and allow for a variety of shaft motions: rotating, reciprocating, rocking, and various combinations of these. In service the various sizes appear to be about equally effective, and the average life and service rendered does not depend in any marked degree on the type of motion transmitted through the seal. It was originally thought that better sealing would be obtained at a rotating than at a reciprocating shaft and that a screw motion of the shaft would result in better performance than either straight rotational or reciprocating motion. This has not been confirmed in plant operation, and any difference in seal effectiveness that may exist for different shaft motions is so slight as to be a secondary consideration in the design of sealed mechanisms.

In service, Wilson seals have been the source of considerable vacuum trouble. Much of the trouble encountered in the early days of plant operation can be attributed to causes such as (1) improper assembly of seals in servicing, (2) defective washer material, (3) excessive tightening of the clamp nut, and (4) too-close fits between assembled parts.

Servicing has improved greatly with experience, with the availability of special tools, and with the adoption of wider tolerances in the fit of component parts. Valve washers of the same material used for closure gaskets (TEC Specifications DD-1000) give good service. Molded washers appear superior to cut washers, and there is some

Fig. 1.28—Wilson-seal assembly and details.

indication that performance might be further improved by molding washers to a shape differing from the flat washers now used. These improvements have resulted in a marked decrease in seal failures,

Table 1.9—Wilson Seals—Standard Dimensions in Inches

Shaft size*	Body			Gasket	
	D	E	Thread	I.D.†	O.D.‡
1/4	1	1/2	1 5/16-14	5/32	3/4
3/8			1-14	1/4	7/8
1/2			1 1/4-12	3/8	1 1/8
5/8			1 1/4-12	1/2	1 3/8
3/4			1 3/8-12	5/8	1 1/4
1			1 7/8-12	3/4	1 1/2
1 1/4	1 1/4	3/4	2-12	1	1 3/4
1 1/2			2 1/4-12	1 1/8	2 1/8
1 3/4			2 1/2-12	1 5/8	2 1/4
2			2 3/4-12	1 7/8	2 5/8

*All sizes given have tolerances of +0.000, -0.002.

†All diameters given have tolerances of +0.003, -0.003.

‡All diameters given have tolerances of +0.010, -0.000.

Body:

A = Shaft size + 0.001 to 0.005

B = Shaft size + 1/4

C = Shaft size + 1/2 ± 0.002

Pump-out ring:

O.D. = Shaft size + 1/2 - 0.002 to 0.006

I.D. = Shaft size + 0.008 to 0.012

D = Shaft size + 1/4

Pressure ring:

O.D. = Shaft size + 1/2 - 0.002 to 0.006

I.D. = Shaft size + 1/4

Clamp nut:

I.D. = Shaft size - 0.008 to 0.012

C.D. = Shaft size + 1/4

A = 3/16 for 1/4 to 1 1/4 size

= 1/4 for 1 1/2 to 2 size

Thread: Same as for body

and current failures are probably not more frequent than should be expected from any seal of this type.

A statistical estimate of the average life of seals indicates a year or more as the useful life of valve-stem and shutter seals. Slit-

cleaner, K-lock, and certain adjustment seals have shorter useful life, largely because of their exposure to relatively high temperatures. Present practice is to replace seals as a routine servicing procedure before they become defective.

Periodical lubrication, especially in the case of reciprocating-shaft seals, is essential if wear and leakage are to be held to a minimum. This and occasional tightening are the principal services required. Add to this the fact that seal replacement is a simple operation and it appears that in spite of occasional failures these seals provide a standard of performance that probably cannot be significantly improved.

The value of the double washer and test space as a means of testing seals was seriously questioned at one time partly because of the insensitivity of test methods and partly because of the frequent plugging of the test opening with seal lubricant or soap solution residue. At this time many seals in Alpha I were modified by plugging the test opening in the seal housing, removing the pump-out ring, and replacing it by a stack of rubber washers of equivalent thickness. The resulting multiple-washer seal gave good sealing but less freedom of shaft motion. With the introduction of helium leak detectors, higher sensitivity provided by the introduction of helium to the test space made possible positive testing of seals. Under these conditions, the test-space feature of the standard seals is well worth retaining.

The use of diaphragm and bellows seals at valve stems has already been mentioned. Bellows have also been used in sealing some of the source and receiver adjustments in the present Beta units. Advantages of this type of seal are positive closure and ability to withstand high temperature. Also, useful life should be greater for a bellows than for a Wilson seal provided the bellows is protected from torsional strains and over-extension or compression. To be weighed against these advantages are limited motion available and greater difficulty of replacement.

(b) Insulating Seals. With a few minor exceptions, insulating vacuum seals occur at the unit faceplates and are associated with lines supplying electric power to the source and with metering circuits on the source and receiver. Details of design and a summary of changes made in the seals associated with the source and receiver are given in Division I, Volume 7, National Nuclear Energy Series. Therefore no attempt will be made at this point to present the complex seal assemblies actually used on operating units, but rather the aim will be to discuss the various types of seals used with special reference to their vacuum characteristics.

Insulating seals of the following distinct types have been extensively used at one time or another: (1) Kovar seals, (2) spark plug-type seals, (3) ceramic-to-metal seals, and (4) rubber compression seals.

Kovar seals were used wherever possible in the original design and construction of Alpha and Beta units. Figure 1.29 shows three of these seals typical of the different models used in various installations.

Fig. 1.29—Kovar-type seals. (a) Single lead, 125 mil. (b) Double lead, 50 mil. (c) Four lead, 50 mil. (d) Four-lead compression seal.

As received from the manufacturer, seals of this type have been found uniformly good. However, two major difficulties arise in connection with their use. First, leaks occur all too frequently where the seal is welded or soldered to the assembly or where leads through the seal are soldered in. This is particularly likely to occur when the parts to be soldered are relatively inaccessible. This would be inconvenient but not too serious if it were possible to test these joints individually as they are made or repaired. Unfortunately, this seldom

is the case, however, and to test a single soldered joint it is necessary to subject the completely assembled unit to pressure or vacuum test.

The frequent cracking of glass seal parts, owing largely to the high temperatures incident to soldering, but sometimes occurring as a result of mechanical shock or strain, is a second major difficulty experienced with these seals. Here again inability to test seals individually aggravates the problem of detecting defective seals.

Largely because of these leakage troubles there has been a distinct trend away from the use of Kovar seals on production units. Their use has been completely eliminated on all currently operating Beta units, and a similar change to seals of more satisfactory types was well under way in both Alpha I and Alpha II at the time of closing down these buildings.

In dismissing Kovar seals as unsatisfactory for the service required in production units, it should be pointed out that this failure results from difficulties of installing, servicing, and testing that are characteristic of production units, and not from defects inherent in the Kovar seals. Conditions might well arise in which Kovar seals, with their positive sealing, freedom from outgassing, and ability to withstand high temperatures, would provide the ideal type of insulating seal.

Spark plug-type fittings of laminated mica construction have been used for the transmission of both electrical leads and water lines into the vacuum system. Usually these fittings are threaded into the unit faceplate. While additional solder sealing at the threaded closure may be necessary, leak-free seals may usually be attained by the use of soft copper gaskets and by observing the precautions normally applicable to threaded vacuum joints. B-G type plugs have been found uniformly good from a vacuum stand point but considerable leakage trouble has been experienced with similar plugs from other manufacturers.

Figure 1.30 shows representative spark plug-type fittings of both standard and special design.

The limited use that has been made of these plugs is due in part to their cost and in part to a feature of operating-unit design which requires that numerous leads and their associated seals be installed in a very limited space. Also their use is precluded in some cases because of inaccessibility of the installed plug for making the necessary internal connections.

The most satisfactory solution so far found to the insulated seal problem is the use of rubber compression seals. Simple in principle,

Fig. 1.30—Spark plug-type fittings.

Fig. 1.31 — Bushing assembly and seals.

compact in design, and easily installed and serviced, these seals give better vacuum service than the more complex devices they replace. Outgassing of the seal rubber is not serious in existing installations, nor is the operations temperature limit imposed by the use of rubber a serious handicap.

Figure 1.29d shows a small four-wire seal of this type used to replace the Kovar seal shown in Fig. 1.29c for bringing out bare thermocouple leads.

Figure 1.30 shows a typical compression-seal assembly in which a rubber disk about $\frac{1}{2}$ in. thick and $1\frac{1}{4}$ in. in diameter provides the seals for a water line, two water-cooled and three plain electric leads, and also seals the assembly at the inner wall of the tube that serves as the seal housing. A second washer and pump-out opening provides for ready testing of the assembled seal. Other seals of this type provide for as many as fourteen lines through a 4-in. disk.

As a final type of insulating seals mention should be made of those seals required in connection with the large ceramic bushings used for supporting and insulating certain elements of the operating units. The problems of vacuum seals at these bushings is only one of numerous problems, both mechanical and electrical, that arose in connection with their use. Since these bushings and their related problems are discussed fully in Division I, Volume 8, a single typical example will be considered at this point.

The bushing shown in Fig. 1.31 is held in the faceplate by an external clamp ring. The gaskets associated with the bushing flange are of cork composition and are not expected to provide a high-vacuum seal. The bushing-to-faceplate seal is made by soft-soldering a metal spinning to the faceplate and to a matching spinning that has been soft-soldered to the platinized bushing surface. The bushing cap, which is integral with the brass tube running the length of the bushing, is held in place by the ring nut on the outer end of the bushing tube. Neither of the bushing end gaskets are expected to form vacuum seals. The cap is sealed to the vacuum end of the bushing by soft-soldering to the cap a specially shaped spinning that has been previously soldered to the platinized bushing end. Water line and electrical leads passing through the bushing tube are sealed at its outer end by the multiple-lead rubber compression seal mentioned in the preceding paragraph. Three pump-out tubes provide for pretesting the three seals of the assembly.

In the Alpha II units, and on an experimental basis in present Beta units, the soldered seals, which are a source of trouble in assembling and servicing, have been eliminated by the use of a rubber sealing

gasket between bushing flange and faceplate and a rubber compression gasket between bushing and bushing tube. This compression gasket is placed at the cool outer end of the bushing. This arrangement, together with the rubber compression lead seal provides the most satisfactory method of insulated sealing that has been found.

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Chapter 2

MATERIALS USED IN VACUUM SYSTEMS

By C. E. Normand

Consideration is given here to certain problems that have arisen in plant operation regarding the relative merits of various materials used in the construction and operation of the plant vacuum systems. Typical of these problems is the choice of a diffusion-pump oil or a material for gaskets. Then too the introduction of any untried material into the evacuated space raises questions as to the outgassing characteristics of the material and possible effects on the material of long exposure to vacuum.

Problems relative to materials and their use in vacuum systems are selected for treatment on the basis of their apparent general importance.

1. MECHANICAL-PUMP OILS

The functions of the oil used in a mechanical vacuum pump of the Kinney or Beach-Russ type are threefold.

1. It provides an oil seal between moving parts.
2. It provides necessary lubrication of the moving parts.
3. It provides the valve lifting and ejecting mechanism during low-pressure pumping.

In addition to meeting these requirements the oil chosen should have as characteristics (1) a vapor pressure at operating temperatures which is lower than the pressure to be maintained by the pump and (2) resistance of the highest possible order to the adverse effects of any contaminants that may be present.

These functions are adequately met by almost any medium-weight turbine-type oil. SAE 20 oil is commonly used. However, in cold weather, or with unworn pumps, SAE 10 gives good sealing. Oil as heavy as SAE 40 has been successfully used.

Table 2.1 lists a representative number of oils whose use in the plant or in experimental tests has been sufficient to establish their satisfactory pumping qualities.

In addition to providing adequate lubrication and sealing, the vapor pressures of these oils at normal operating temperature are low enough to permit the attainment of blanked-off pressures of 10 μ Hg or better. If pressures lower than this are required, say 4 or 5 μ Hg,

Table 2.1 — Characteristics of Mechanical Vacuum-pump Oils

Brand	Type	Price, cents/gal	Viscosity, Saybolt units		Type of hydro- carbon	Resistance to Cl and HCl	Resistance to moisture clouding
			At 100°F	At 210°F			
Caloil Deturbo medium	Turbine						
Gulf Crest B*	Turbine	63	209	48	Par	Good	Poor
Gulf Crest C*	Vacuum	65	311	55	Par	Good	Very poor
Gulf Mechanism E	Machine	48	205	45	Naph	Good	
Gulf Harmony E	Turbine	46	603	64	Par	Fair	
MacMillan Vacuum Pump	Vacuum	68	350	50	Mixed	Poor	
Shell Turbo 41	Turbine						
Socony Vacuum Pump*	Vacuum	71			Par		
Socony D.T.E. light	Turbine	71	155	43	Par	Poor	
Texaco Regal B	Turbine	43	196	45	Par		
Texaco Regal C	Turbine	45	325	49	Par		
Texaco URSA P-10* (Navy symbol 2110)	Turbine	32	165	45	Par	Fair	Good

*These oils have had extensive use in the plant at one time or another.

the use of the special vacuum-pump oils may be justified. These oils are produced by taking a very narrow distillation cut, and some are specially dried. As a result their vapor pressures are even lower than those of the standard turbine oils. Common motor oils are usually of higher vapor pressure and are incapable of yielding the low pressures that can be attained with the turbine-type oils.

The principal pump-oil contaminants encountered in plant operation are water, free chlorine, and hydrochloric acid. Considerable work has been devoted to the choice and development of oils that will best withstand the effects of these contaminants. In a blanked-off pump all the oils previously discussed function adequately and there is little basis for choice between them. In their resistance to chlorination and to wetting, however, they are found to vary widely. Chlorination and wetting appears to be a particularly bad combination in that oils

which have high resistance to chlorination often behave poorly in the presence of water, and vice versa.

Since the deleterious effect of water on pump oil arises primarily from the formation of an emulsion that has essentially the vapor pressure of water when it is returned to the pump as a sealant, demulsibility tests may be applied as a first criterion in choosing an oil for use in the presence of water. Determinations of A.S.T.M. steam-emulsion numbers (D 157-36, for example) serve as a basis for eliminating from consideration oils of high emulsion numbers, but fail to differentiate between oils having low numbers and yet differing considerably in the completeness of their demulsification. All the oils in Table 2.1 have low emulsion numbers, but on separating from an emulsion some oils are quite clear whereas others remain clouded in varying degree. This visible cloudiness represents a water residue that is harmful in a vacuum-pump oil, and may be observed qualitatively as an indication of completeness of demulsification.

For similar oils of different viscosities, demulsification is more complete in the oil of lower viscosity. For this reason, it is advantageous to use oil of the lightest weight that will furnish adequate sealing.

The effects of severe chlorination of oil may be summarized as:

1. Appearance: The oil becomes an opaque black. Rapidity and extent of discoloration varies from one oil to another.

2. Sludge: On standing, a black sludge settles out, leaving a clear oil layer above. The extent of clearing may vary from 0 to 50 per cent for different highly chlorinated oils. Also, the extent of sludging is varied by the presence of metals during the chlorinating process.

3. Percentage absorption: The percentage of chlorine absorbed during an arbitrary exposure varies for different oils. In five of six oils tested, the variation was from 14.3 to 28.2 per cent; for the sixth sample, the percentage of chlorine was 75.8.

4. Viscosity: Viscosity increases markedly with increasing chlorination. For severe and equal exposures the viscosities of different oil samples increased by factors varying from 5.6 to 146.

5. Surface tension: Surface tension increases with chlorination. For the six samples mentioned above the increase varied from 7.5 to 13.7 per cent.

6. Specific gravity: Specific gravity increases with chlorination. Increases of 20 to 85 per cent have been observed to result from equal exposures of different samples.

done, it is likely that the cost of handling and prefiltering the used oil would exceed the cost of the new crude required for outright replacements.

3. GASKET MATERIALS

The problem of choosing gasket materials has been largely that of securing maximum useful gasket life. The standard gasket design (see Chap. 1, Sec. 8.1b) was such that initially tight-gasketed closures were obtainable with almost any type of rubber or rubberlike gasket material, but the replacement requirements varied widely from one material to another.

Extensive studies by UCRL, Tennessee Eastman Corporation, and various vendors, together with the cumulative experience gained in plant operation, resulted in the adoption of the following specifications for plant gasket material:

TEC Specifications DD-1000

Chemical composition

Hycar (OR-15) base; suitable compounding ingredients and curing to meet physical specifications

Physical properties

1. Mechanical

Tensile strength	1800 lb/sq in. minimum
Elongation	300% minimum
Shore hardness	60 $\pm$ 5
Compression set	15% maximum

2. Thermal

Maximum service temperature	150 to 170°F
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3. Chemical

Oil resistance	$\pm 6\%$ weight change maximum (70 hr at 212°F)
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Forms available

Sheets
Molded
Extruded

A typical compounding that has been shown to meet these specifications is given in the accompanying table. The compound is cured for 45 min at 310°F.

Previous to the adoption of Hycar compounds, Neoprene had been widely used because of its good oil-resistant characteristics.

Material	Amount, lb
Hycar Or-15	100
Zinc oxide	5
Tetramethylthiuram disulfide	3
Benzothiazyl disulfide	3
Gastex (carbon black)	85
Plasticizer S.C.	15
P-25 Cumar	5
Dibutyl phthalate	10
Stearic acid	0.5
Total	226.5

Limited use has been made of Du Pont's Teflon (D-29) as a gasket material. Its outstanding merits are inertness, low volatility, and high service temperature (200 to 300°C).

4. SEAL WASHERS AND BUSHINGS

The same rubber used for gaskets (TEC Specifications DD-1000) has been found highly serviceable for valve-disk inserts and Wilson seal washers and is now specified for these uses. For compression-type seals, however, a Hycar rubber of slightly different characteristics is now specified as TEC 1001. This rubber differs from that specified for gaskets in that minimum tensile strength is reduced to 1200 lb/sq in., Shore hardness is reduced to 40 ± 5 , and compression set may be as high as 50 per cent.

5. VACUUM GREASES

Vacuum greases are generally used to ensure the sealing of gasketed closures, to facilitate the assembly of compression seals, and to lubricate the moving parts of Wilson seal assemblies. Such greases should have low vapor pressure at service temperature both to prevent drying of the grease and to avoid the introduction of vapors into the vacuum system. In this last respect, occluded air may be a more serious source of trouble than vapor pressure.

Of the various known vacuum greases, Lubriseal, a product of the Arthur H. Thomas Company of Philadelphia, has been most widely used in the plant. Limited experience with other standard preparations, including Celvacine from Distillation Products and Vacuseal

from Central Scientific Company, indicates no marked superiority on the part of any one of these greases. Dow Corning's silicone-type stopcock grease has, however, proved superior to any of these in applications subject to moderately high temperature.

The approximate characteristics of various vacuum greases are given in Table 2.3.

Table 2.3—Comparison of Vacuum Greases

Trade name	Supplier	Melting point, °C	Vapor pressure at 20°C, mm Hg
Lubriseal	Arthur H. Thomas Co.	40	$<10^{-5}$
Vacuseal	Central Scientific Co.	50	10^{-5}
light			
Vacuseal	Central Scientific Co.	60	10^{-5}
heavy			
Celvacene	Distillation	90	10^{-6}
light	Products, Inc.		
Celvacene	Distillation	120	$<10^{-6}$
medium	Products, Inc.		
Celvacene	Distillation	130	$<10^{-6}$
heavy	Products, Inc.		
Stopcock grease	Dow Corning Corp.	215	$<10^{-5}$
Apiezon L	James G. Biddle Co.	Probably < 50	10^{-11} (10^{-3} at 300°C)
Apiezon M	James G. Biddle Co.	Probably < 50	6×10^{-7} (10^{-3} at 200°C)

6. VACUUM WAXES

The uses of waxes in making vacuum connections has been avoided as far as possible by the substitution of solder seals, Kovar seals, and gasket and compression seals. About the only exception to this is the early use of waxed seals for joining ionization gauges to the systems. For these installations ordinary red sealing wax was usually used for waxing the gauge tubulation into a loosely fitting metal sleeve. The simple technique of making such seals is readily developed to a point at which leaks rarely occur.

Picein and de Khotinsky cements have also been satisfactorily used for making ionization-gauge attachments and in various experimental assemblies. All these are low-vapor-pressure waxes as compared with normal operating pressures. Variation in melting point is likely to be the basis of choice of a suitable wax.

7. SURFACE-COATING MATERIALS

The vacuum characteristics of various surface-coating materials are considered as a phase of the tank outgassing problem, which is discussed in Chap. 6.

Experimental studies in both the laboratory and the plant indicate that a number of materials, including red and clear glyptal and silicone varnish, have outgassing rates within the limits allowable in plant systems even when they are applied extensively. This is true only after an initial period of 12 to 24 hr under vacuum, during which outgassing or vapor liberation may be quite severe. Also, such coatings must be protected from excessive temperature and from direct bombardment by stray ion beams. Painting the inside of process tanks was precluded by these conditions; the inner surfaces of a number of vacuum docks were painted, however, as a protection against corrosion.

8. DETERMINATION OF OUTGASSING BY WEIGHT LOSS

A method developed by N. S. Herod for comparing the outgassing characteristics of surface coating materials is worthy of notice because of its promise of yielding useful results in this and in related problems.

This method gives a comparison of the percentage of weight loss of different materials as a function of time under vacuum. To determine the curve of weight loss vs. time for a material, a number of samples are prepared by coating vanes of thin sheet copper with the material under test. These vanes are weighed before coating and after the coatings have dried to determine the weight of each sample. The samples are placed in individual compartments attached by Kerotest valves to a small vacuum chamber. The complete chamber assembly may be placed in an electric oven for making tests under controlled high-temperature conditions.

Evacuation of all samples begins simultaneously, and the samples are removed one by one at regular time intervals and weighed to determine their loss in weight. These weight losses, reduced to a percentage basis, are then plotted as a function of time under vacuum to represent the total outgassing characteristics of the material.

The apparatus is of such a scale that all weighing can be made on a sensitive balance to 0.1 mg.

Typical outgassing curves for three materials are shown in Fig. 2.1.

An even more sensitive and versatile method of determining weight-loss vs. time curves for various materials is provided by the vacuum balance devised by G. W. Monk and previously unreported.

The equipment used for these outgassing measurements is illustrated in Fig. 2.2. It consists essentially of an electrically operated analytical balance mounted in vacuum. The sample is suspended by a platinum wire within a radiation furnace below the commodity pan.

Fig. 2.1—Percentage weight loss of materials in vacuum. Curve A, clear glyptal; curve B, silicone varnish; curve C, red glyptal.

The wire passes through a hole $\frac{1}{8}$ in. in diameter and about 2 in. long separating the system containing the oven from that containing the balance and motors. The sensitivity of the balance is 0.05 mg and the maximum load is 200 g. The external electrical controls permit operation of the beam-clamping mechanism, positioning of the rider, and adjustment of the chain so that a change in mass of 1,100 mg or less can be measured while the system is under vacuum.

In operation a sample 1 by 3 in. or less is suspended inside the furnace by means of the wire and the system is evacuated to 0.03μ Hg or less. Observation can then be made of the change in mass as a function of the time, temperature (up to 700°C), and pressure.

Preliminary results show that there are three different phenomena associated with measurements on outgassing. Small quantities of material with an appreciable vapor pressure are eventually removed so that the outgassing rate decreases almost exponentially and the weight

Fig. 2.2 — Sketch of equipment used for outgassing measurements.

of the sample drops asymptotically to some value depending upon its temperature. In other cases in which there are large quantities, such as the sample itself, of material having an appreciable vapor pressure, the outgassing rate approaches some fixed value from which

the vapor pressure at that temperature can be calculated. Using this equipment, vapor pressures as low as 10^{-6} dynes/sq cm at room temperature for a molecular weight of 50 can be measured in a few hours. The third type of weight change that is observed is due to

Fig. 2.3 — Typical outgassing curves of a carbon cylinder for different temperatures.

condensation onto the sample of vapors already in the system. Since the sample is foreign to the system and its surface must come into equilibrium with the vapors present, the sample may gain weight at certain temperatures.

Preliminary experiments have been made on samples of carbon, aluminum, tantalum, and Teflon and are given in part only to illustrate the nature of the results to be expected from this equipment.

Figure 2.3 shows the outgassing curves for a typical carbon cylinder as a function of time for three different temperatures. The last point on each curve is the first point on the next curve since time measurements were started again at each increase in temperature. An outgassing curve for aluminum alloy is shown in Fig. 2.4, in which case the alloy apparently has an appreciable vapor pressure at 500°C.

Fig. 2.4 —Outgassing curve for an aluminum cylinder at two different temperatures.

In addition to its ability to measure mass changes under vacuum the equipment can be used as an absolute gauge of the Knudsen type, as a flowmeter for gases at low pressure, and for other interesting applications.

By methods such as the two just described it is possible to determine the relative outgassing characteristics of various material under various conditions of pressure and temperature. Much work in this field remains to be done, partly because such determinations are time-consuming and partly because the outgassing of many materials may be dismissed as negligible when they are used in high-speed systems such as those here involved. Since ample pumping speed is

provided for producing operating pressure in the presence of the considerable outgassing that occurs from tank walls and extensive areas of operating units, any additional outgassing due to items such as rubber bushing, carbon or lavite plates, plastic, ceramic, or mica insulation, etc., can hardly be significant unless the outgassing characteristics of these materials are very bad indeed.

Changes produced in certain physical characteristics of materials by exposure to vacuum are often unknown but may usually be determined directly. The hardening of rubber and the drying out of greases may be mentioned as typical. Also, the change in thermal conductivity of heat-insulating materials and in the conductivity between surfaces in contact are problems that have arisen.

By way of summary it should be emphasized that the passage from small low-speed vacuum systems to large systems provided with high pumping speed has required a complete reevaluation of the significant vacuum characteristics of materials. Slight differences that were significant in the materials incorporated in small systems become unimportant in the case of high-speed systems, and materials that were previously unusable are now widely used. Criteria of usefulness based on experience with old type systems are no longer valid, and new criteria are only now being established. Only with increasing experience in the use of large vacuum systems will full utilization of these additional materials be possible, but the varied and intricate design of vacuum equipment that will be made possible is already clearly indicated.

Chapter 3

COLD TRAPS AND REFRIGERANTS

By C. E. Normand and Frank A. Knox

1. FUNCTIONS OF COLD TRAPS

Inclusion of cold traps as essential elements in all plant vacuum systems has been mentioned in Chap. 1. No detailed consideration has been given, however, to the functioning of these traps in plant operation or to the problems arising in connection with their use.

The functions of cold traps in the plant vacuum systems are (1) to act as pumps in so far as the removal of condensable vapors from the system is concerned and (2) to serve to protect pump fluids from contamination by vapors originating in the system.

A third, and sometimes important, function of cold traps—the prevention of upstream diffusion of pump-fluid vapors—is not significant in these systems because of the use of low-pressure pump fluids, and because of the great distances separating mechanical pumps from diffusion pumps and diffusion pumps from process tanks.

1.1 Pumping Action of Traps. The pumping action of a cold trap toward condensable vapors arises from the condensation of vapors coming in contact with the cold-trap surface. If it is assumed that all vapors making contact are condensed, the trap surface acts as an aperture opening into a perfect vacuum in so far as these vapors are concerned, and it is permissible to apply aperture-flow formulas in arriving at what may be called the “pumping speed of the trap.” Thus under conditions of molecular flow the speed of a trap may be given as

$$S = \frac{dv}{dt} = A \sqrt{13.2 \frac{T}{M}} \quad (1)$$

where S is the speed of trap measured at the partial pressure of the vapor in the region of the trap in liters per second; A is the effective

trap area in square centimeters; T is the temperature of the vapor in degrees Kelvin; M is the molecular weight of the vapor.

Since the trapping of water vapor at or near room temperature (20°C) is of particular importance in plant systems, Eq. 1 may be reduced for this case to

$$S_w = 14.65A \quad (2)$$

and if A' is the effective area in square inches,

$$S_w = 94.5A' \quad (3)$$

The relation of effective trap area to geometrical surface area is usually determinable only by estimate. It is clear, however, that effective area refers only to that part of the trap surface that is cold and depends, therefore, on the amount of refrigerant present. Also, the effective area will be reduced to the extent that the trap is not freely accessible from all directions. A trap placed near the side walls of a chamber has smaller effective area than a similar trap at the center of the chamber. In practice effective trap area is usually arrived at by reducing the refrigerated area by an estimated factor based on the trap location.

Obviously the above relations for cold-trap speed apply only to conditions of molecular flow. When flow becomes laminar the aperture formula no longer applies, and, moreover, directional effects come into play resulting in unequal condensation rates on various surface areas. Trap speed is then a function of trap geometry and it cannot even be said with certainty whether the speed of a given trap at laminar flow is greater or less than at molecular flow. Fortunately a knowledge of trap pumping speed under laminar flow conditions is much less important than at the low pressures associated with molecular flow.

So far no account has been taken of the fact that any material condensed on a cold trap reevaporates into the system at a rate proportional to the vapor pressure of the condensate at cold-trap temperatures. As long as this vapor pressure is negligibly small compared with the partial pressure of the vapor remaining in the system this reevaporation may be neglected and the net speed of the trap is given as above. As the partial pressure of the remaining vapor is reduced to a value approaching the vapor pressure of the condensate, however, the net rate of condensation approaches zero. Thus for any combination of vapor and refrigerant there is a base pressure, equal to the

vapor pressure of the condensate at refrigerant temperature, below which the partial pressure due to vapor in the system cannot be reduced by trapping. It follows that the choice of a refrigerant is dictated by the particular vapors present and the pressure desired in the trapped system.

In plant systems water is by far the major source of vapor. It has been estimated that about 1 qt of water is liberated by the outgassing of an apparently dry Alpha I tank and unit. Other vapors and gases that are condensable at trap temperatures occur in much smaller quantities and will be dealt with in a later section.

Table 3.1 lists the minimum trap temperatures and corresponding vapor pressure of water for several possible refrigerants.

Table 3.1—Refrigerant Temperatures and Resulting Vapor Pressure of Water

Refrigerant	Temperature, °C	Vapor pressure of water, μ Hg
Ice	0	4569
Mechanical refrigeration*	-50	30
Solid CO ₂	-78.5	0.6
Liquid N ₂	-195.8	$10^{-10}\dagger$

*This represents the lowest temperature obtained in experimental tests made with a small single-stage York compressor.

†From F. H. Newman, "Production and Measurement of Low Pressures," Ernest Benn, Ltd., London, 1925.

An examination of the above table shows that, although CO₂ provides adequate trap refrigeration in the roughing and fore-vacuum systems in which pressures approaching 10 μ Hg are the lowest expected, this refrigerant is quite inadequate in the process tank where pressures must approach 0.04 μ Hg. For this reason CO₂ traps are used in roughing and finishing lines throughout the plant, and liquid-nitrogen traps are provided in connection with all process tanks.

1.2 Protective Action of Traps. The protective action of cold traps depends on their removing from the system those vapors that affect adversely other elements such as pumps and gauges. The necessity for this protection depends on the vapors present and the seriousness of their effects. The effectiveness of the protection depends on the completeness with which these vapors are removed. These aspects of plant trapping will be treated in connection with subsequent discussions of the two types of traps used.

2. DRY-ICE (SOLID CO₂) TRAPS

2.1 Trap Design. A uniform type of trap designed to use dry ice as a refrigerant was installed adjacent to and on the intake side of each mechanical pump in the plant. These traps (see Fig. 3.1) were designed to meet the requirements of the largest systems involved and had considerable surplus capacity when used in small systems.

2.2 Trap Installations. In all production installations traps were placed with inlet and outlet as shown in Fig. 3.1. Direction of flow through the trap appears to be immaterial, however, and might have been reversed to meet varied piping requirements. This was done in the case of one vacuum docking system installed after completion of the production plant.

In the first tracks built traps were placed some 6 ft above the floor in direct line from pumps to overhead headers. The difficulties encountered in servicing these elevated traps were so great, however, that in later installations they were placed near floor level, even though this required an increase in the length of connecting lines.

2.3 Thermal Efficiency. Though no exact figures are available, it is certain that the thermal efficiency of these traps is quite low. This is indicated by (1) heavy icing of the top flange caused by heat conduction along the cold wall; (2) condensation of water on the outer side wall caused by convection and radiation across the inter-trap space; (3) frosting of the bottom of the outer shell of the trap caused by an involved mode of heat transfer described in a later section.

Although these losses might be considerably reduced, no very effective steps have actually been taken in this direction. Considerable reduction in the icing of the top flange results from operating the trap only three-fourths filled with refrigerant, but this reduces trap efficiency, especially since only about half the surface facing the inlet opening is then cold and this is by far the most effective condensing area. Insulation of the top beyond that provided by a removable wood cover interferes more or less seriously with trap servicing. External side-wall insulation would not only improve efficiency but would also eliminate the unsightly conditions caused by the dripping of water from the trap. So serious was this dripping, especially in installations in which traps were directly above the mechanical pumps, that special drip pans were provided to cope with this nuisance. Insulation, however, was applied only on a few experimental traps. The use of radiation shielding in this connection has also been suggested but has not been put into actual practice. Frosting of the trap bottoms occurs seldom and is not considered a major cause of thermal inefficiency.

Fig. 3.1—Cold traps.

2.4 Speed and Conductance. No reliable estimate can be made as to the vapor-pumping speed of the dry-ice traps since they operate at pressures above the molecular flow range. With their cold-surface area of about 800 sq in., however, it is safe to assume for them a speed well in excess of that of the associated mechanical pumps or even of the connecting lines. Conductance of the traps to the passage of gas can be approximately evaluated and is certainly not less, and may be much greater, than that of an equal length of 6-in. pipe.

High trap conductivity is important if the full advantage of high mechanical-pumping speed is to be realized at all times. High trapping speed assumes greater importance as the ratio of condensable vapors to noncondensable gases increases. Considerable water is trapped during the roughing-down of a system, and the roughing period would be appreciably prolonged if this additional load were thrown on the mechanical pump. During the bake-out period the maximum rate of evacuation is limited by the total fore-pumping speed; since water vapor is the principal product being removed, high trapping speed is particularly desirable during this prolonged period. After steady operation is established the total quantity of gases and vapors to be removed is reduced to such a point that the mechanical pumps alone would provide adequate backing for the diffusion pumps for the remainder of the run. Since there is no provision for isolating the traps from the system, however, refrigeration must be continued to prevent the release of the material previously condensed.

2.5 Protective Action of Traps. The demands made of a dry-ice trap in protecting the associated mechanical pump depend on the nature of the vapors present and the effects of these vapors on the pump. The effectiveness of the trap in providing protection depends on the completeness with which these vapors are removed by the trap.

Since water is the dominant vapor present in production systems and since contamination of the pump oil by water reduces the efficiency and elevates the base pressure of the pump, the first demand of the dry-ice traps is that they protect the mechanical pumps against water vapor. Although considerable ice collects on traps during a normal run (probably 3 or 4 lb), it is readily apparent that much vapor passes the traps to be condensed by compression and remain in the pump oil. As a result, pump oil must be removed at intervals depending on the total quantity of vapor removed from the system. Roughing pumps, especially if their use is extended through the bake-out period, are subjected to the greatest exposure and require most frequent oil renewal. Finishing pumps normally operate for a week or more without oil change. A minute water leak occurring in an

operating tank may necessitate changing of the pump oil at intervals of a few hours, however.

There is no doubt that removal of water vapor could be made more complete by installing a system of baffles designed to bring about more intimate and prolonged contact of the gas with the cold surface. But such baffling would seriously reduce the conductance past the trap and thus may well be more objectionable than the required oil renewals.

Besides water vapor, there are many other vapors that occur occasionally or in tolerable quantities but normally present no trapping problem. High-vapor-pressure oils and greases are excluded from the system. The use of cleaning solvents other than water is restricted to highly volatile materials (carbon tetrachloride, xylene, trichloroethylene) whose residual vapors are quickly removed. Evaporation of plasticizer from gaskets, insulating materials, etc., becomes negligible after moderate exposure to vacuum. There remains, however, one material that requires special consideration, i.e., hydrogen chloride.

Chlorine is liberated from the charge material throughout the operating process and combines readily with various materials present in the system to form compounds such as ferric chloride, copper chloride, and hydrogen chloride. Formation of the first two compounds results in destructive corrosion of metal parts of the system, but also results in these metal parts acting as a pump for removing chlorine gas from the system. Hydrogen chloride, if allowed to enter the mechanical pump, accelerates corrosion especially of valve parts, and may contribute to the formation of waxes and sludge in the pump oil. It is, therefore, desirable that these vapors be trapped as completely as possible.

At the suggestion of W. E. Bush, UCRL, a rather detailed study of the trapping of hydrogen chloride¹ was made in connection with the previously mentioned frosting of the bottom of the outer trap shell. In this connection, it was pointed out that while the freezing point of a hydrogen chloride-water solution is, in general, above dry-ice temperature (-79°C), solutions having a concentration ranging from 23.7 to 25.2 per cent hydrogen chloride have freezing points lower than this, and will not be retained by the trap. If during the operating cycle any considerable portion of the trapped material reaches a concentration within this rather narrow range, it will melt, drop from the cold trap, and reevaporate from the trap bottom, thus setting up a refrigerating cycle. This would account for the observed frosting of the trap. The fact that analyses of drippings collected in the bottom of

the trap showed a hydrogen chloride concentration of 24 ± 1.4 per cent is in good agreement with the proposed explanation.

Since the above phenomenon occurred infrequently and had no serious effect on plant operation, no steps were taken to prevent its occurrence. The obvious solution would be to use a lower temperature refrigerant. A temperature of -90°C would be adequate, but no such refrigerant is known. Recycling of the eutectic mixture could be prevented by the introduction of a chemical absorbent such as NaOH to react with the HCl drippings. However, the seriousness of the problem did not appear to justify the added servicing involved.

2.6 Trap Servicing. Normal servicing of the dry-ice traps involves (1) scheduled replenishment of the dry-ice supply in each trap, (2) replenishing the heat transfer fluid as required by depletion, and (3) scheduled cleaning to free the trap surfaces of accumulated ice.

Dry ice in the form of briquets is added to each operating trap on a 1- to 2-hr schedule. This schedule was determined by operating experience and provided a considerable margin of safety. Operators performing this work were required to use heavy gloves and goggles.

The use of a heat-transfer fluid in conjunction with dry ice to assure efficient heat transfer from the trap is general practice. A number of the fluids commonly used for this purpose, however, are objectionable in large-scale operations because of the fire or health hazard arising from their use. Both ether and acetone are objectionable for one or both these reasons. Trichloroethylene was first used in the plant, but it had several undesirable characteristics: it evaporated rapidly; it was mildly toxic; and, being very fluid even at dry-ice temperature, it splashed badly. Its use was later abandoned in favor of Butyl Cellosolve. This fluid has a low rate of evaporation, is relatively nontoxic, and is quite viscous at dry-ice temperature.

The rate of depletion of this heat-transfer fluid is quite moderate and arises mostly from losses associated with trap cleaning.

The inner compartment of each trap is removed periodically and cleaned of its accumulation of condensed material. Since this operation requires that the trap and its associated pump be taken out of service, cleaning cannot be rigorously scheduled but must be fitted in with the plant operating schedule. Roughing traps, because of the large amount of water collected, require cleaning at 1- or 2-day intervals in Alpha systems but less frequently in Beta systems. Finishing traps normally require cleaning about once a week.

The weight of the charged trap is such as to require two men for its removal unless special handling equipment is provided. This is the chief disadvantage resulting from the large size of the traps used.

The heat-transfer fluid and any dry ice remaining in the trap are transferred to a separate vessel and retained for future use. In this connection the use of open-top steel-mesh baskets fitted inside the refrigerant vessel has proved very convenient. By pulling this basket, all dry ice is removed, and the remaining fluid may be poured from the trap without excessive splashing. After emptying, the trap is cleaned with hot water and steam, and its surface is wiped dry. Prior to reassembly, the inside of the trap shell is also wiped clean and, as need appears, closure gaskets are renewed.

2.7 Storage and Handling of Dry Ice. In addition to the problems associated directly with trap servicing, other problems arose as a result of the enormous quantities of dry ice received, stored, and distributed to the various plant buildings.

At the time of full plant operation, receipts of dry ice averaged approximately two million pounds per month, or more than 30 tons per day. Table 3.2 shows plant consumption figures based on normal operation. Start-up periods and periods of curtailed use of refrigerants have not been considered.

Dry ice was shipped to the plant area by freight and was stored in an insulated warehouse. As received, the dry ice was individually wrapped in cubes about 12 in. on a side and weighing approximately 60 lb each. In the warehouse these cubes were sawed into blocks of approximately 4 by 6 by 12-in. dimensions, and these blocks were stored in heavily insulated portable bins of about 1 ton capacity. Trucks delivered these filled bins to the various buildings and returned empty bins for refilling.

Within each building smaller insulated boxes, mounted on casters and having a capacity of 250 to 300 lb, were used for distribution to individual traps.

The over-all efficiency of this method of distribution has not been well established, but losses were probably considerable. Losses in shipment to the plant averaged 7.5 to 10 per cent, depending on season and time in transit, and subsequent losses in storage, handling, and carry-over were probably greater than this. It is doubtful if as much as 75 per cent of the dry ice loaded at the factory ever reached the traps in the plant.

2.8 The Elimination of Dry-ice Traps. Because of the considerable cost involved, \$660,000 per year for dry ice plus handling and servicing costs, consideration was given throughout the course of plant operation to the possibility of eliminating or at least curtailing the use of dry-ice traps. All experience indicated that steps in this direction could hardly improve and might seriously impair operation,

however, and for this reason the matter was not pushed prior to the end of the war.

The possibility of substituting small single-stage refrigerating units for dry-ice refrigeration of individual traps was investigated, but the project was abandoned when the lowest trap-temperature attainable was found to be -50°C . Although it was known that adequately low temperature could be attained by the use of large multiple-stage refrigerating units, lack of flexibility and the cost and production outage involved in converting existing equipment to this type of refrigeration precluded its adoption.

Table 3.2—Consumption of Dry Ice

Building	Track No.	Average monthly consumption, tons	Average daily consumption, lb	Traps served	Lb per trap per day
Alpha I	1, 2	89 ± 30	5,940	48	123
	3, 4	85 ± 27	5,660	48	118
	5	58 ± 11	3,870	24	141
Alpha II	1, 2	240 ± 46	16,000	120	133
	3, 4	236 ± 30	15,700	120	130
Beta	1, 2	71 ± 15	4,730	24	197
	3, 4	71 ± 12	4,730	24	197
	5, 6	144 ± 32	9,470	84	113
	7, 8	110 ± 25	7,300	84	87

Total consumption: 1,104 tons per month; 73,400 lb per day.

Cost at \$50 per ton: \$55,200 per month; \$662,400 per year.

With the ending of the war, maximum production at any cost was no longer the first rule of plant operation, and in many phases of the production process, including the cold-trap situation, attention was turned to economy of operation.

Slightly prior to this time, a series of tests had been undertaken by Frank A. Knox, CEW-TEC, to determine the extent to which mechanical-pump oil might be kept dry and serviceable by admitting a continuous stream of dry air at the pump discharge and allowing it to pass out through the oil-separator tank. It was known that this scheme had been used successfully for the moderate drying of the oil in pumps handling large quantities of water, as in dehydration processes; but the effectiveness of this treatment in systems requiring a much higher degree of drying was not known. Actually, the plan proved very effective. Pumps that had previously required daily changes of oil now operated for weeks without a change, and the action of the dry-ice

traps in protecting mechanical pumps from water vapor no longer appeared necessary.*

In the Alpha I plant, where air drying of the pump oil was first practiced, mechanical pumping capacity was in all cases adequate for handling the additional load imposed by discontinuation of the use of dry-ice traps. Also, any resulting small loss of product material through the pumping system could be ignored because of the moderate value of this material. There was, therefore, no serious argument against the installation of air drying in all mechanical pumps of the Alpha I systems and discontinuance of the use of the dry-ice traps. Even in Alpha II, where fore-pumping capacity was already a limiting factor, no serious difficulty arose with the elimination of dry ice since large-capacity roughing pumps were available for backing purposes throughout the bake-out period. Because of the closing down of all Alpha buildings, however, the conversion of Alpha I and Alpha II to air drying was cut short in an advanced experimental stage.

With the closing down of the Alpha plants, attention turned to the air drying of the pump oil and the elimination of the use of dry ice in the Beta plants. In this connection possible material loss became an important consideration owing to the value of Beta charge material. Preliminary experiments showed that this material could be satisfactorily recovered by the installation of suitable exhaust scrubbers, and by March 1946 air-drying systems were in operation on all Beta-plant pumps and the use of dry ice was almost completely eliminated.

Subsequent plant-wide experience with this revised method of operation may be summarized as follows:

1. Adequate air drying of the mechanical-pump oil gives better protection against vapors than was ever provided by the dry-ice traps.
2. Material recovery by the use of scrubbers can more than compensate for any loss of material previously recovered from the trap surfaces. In fact, some recent tests have indicated that the amount of uranium-bearing material collected on the trap is no greater if the trap is refrigerated than if it remains in the system at room temperature.
3. With the eventual elimination of both dry ice and liquid nitrogen, the additional load placed on the mechanical pumps, owing to the loss of pumping action of the cold traps for water vapor, has proved excessive in those systems relying on small mechanical pumps for backing the diffusion pumps during bake-out. This difficulty has been

* The air drying of mechanical-vacuum-pump oil as applied in the plant is covered by U. S. Patent 2,551,541, May 1, 1951, "Vacuum Pumping Method with Mechanical Oil-sealed Pumps," Frank A. Knox.

overcome by utilizing the large-capacity roughing pumps for backing purposes throughout the bake-out stage. In some systems, this involved no more than a change in operating procedure; in others, it was necessary to install cross-tie lines between existing roughing and finishing lines.

4. Present operation of the four Beta buildings shows no significant production loss attributable to the elimination of dry ice and an estimated saving of some \$33,000 per month in operating cost.

Methods other than air drying might have been employed to protect the pump oil from water. These are:

1. Continuous oil flow with a full supply of oil passing from pump to oil purifier (Hilco) to storage tank and back to the pump would undoubtedly be effective, but would require very great purifier capacity. As a compromise measure, a number of pumps were operated satisfactorily with fresh oil flowing into the oil-separator tanks at less than half the rate required for pump operation. Used oil was drained from the tanks at the same rate to be purified and returned to the fresh oil supply. The maintenance of rates of flow to meet pump requirements and purifier capacities and the expense of operating purifiers at all times to meet maximum requirements that occur at short periods only are objectionable features of this method of treatment.

2. Drying by the application of heat, as provided by heating coils installed in the separator tanks of some commercial pumps, is effective if the quantity of water to be removed is great and if drying need be only moderately thorough. If the total quantity of water involved is small, however, and a high degree of drying is required, this method becomes very inefficient.

3. Removal of water by centrifuging is subject to all the objections to the previously mentioned continuous flow method if a single centrifuge is used to serve a number of pumps. The use of individual centrifuges would be expensive both in initial cost and in servicing.

On the whole, air drying of the pump oil is adequate for plant operation and appears to be more practical than other proposed methods.

3. LIQUID-NITROGEN TRAPS

3.1 Function and Requirements of Liquid-nitrogen Traps. Each process tank in the production plant is equipped with a trap cooled by liquid nitrogen and serving primarily to remove condensable vapors from the evacuated system. The fact that these traps may also serve, in some measure, to protect diffusion- and mechanical-pump fluids from these vapors and the fact that a limited amount of stray charge material may be recovered from their surfaces is of distinctly secondary importance.

An incidental but useful function of the liquid-nitrogen traps is their action as indicators of water leaks. A water leak occurring in any tank will cause an increase in the rate of liberation of nitrogen from the trap, which is readily detected if the leak is moderately large.

Since water is the only vapor normally present in sufficient quantity to prevent the attainment of operating pressure by the diffusion pumps alone, the pumping action of the traps for water vapor is of particular interest. In this connection, it may be assumed that each trap has a pumping speed of approximately 100 liters/sec per square inch of effective surface area and a base pressure equal to the very low vapor pressure of ice at liquid-nitrogen temperature.

The need for this trap pumping action occurs primarily during the bake-out period of the operating cycle. Much water vapor must be removed during roughing but, at the high average pressures existing during this stage, removal by mechanical pumping is quite rapid. Also, the accumulation during this stage of a heavy coating of condensate having high vapor-pressure constituents impair the action of the trap during later stages. For these reasons, liquid-nitrogen traps are not filled until the beginning of diffusion pumping.

With the completion of bake-out, the total rate of gas and vapor evolution in the systems is reduced to such a value that the diffusion pumps alone are able to maintain operating pressures. For this reason, the action of the traps is not essential during the remaining period of production, and refrigeration must be continued merely to prevent the reevaporation of materials previously collected. During the bake-out period, however, water vapor is the major product of outgassing, and the added pumping action provided by the traps not only reduces the pressure at a given rate of outgassing, but also makes possible a higher rate of outgassing without a break in diffusion pumping. Thus, bake-out can be accelerated without overloading of the pumps and operating pressure is attained at an earlier stage of bake-out.

To realize the full effect of this action during the bake-out period, the trap should have a pumping speed for vapor at least equal to, and probably considerably greater than, the speed of the diffusion pumps. Tests indicate, however, that under existing conditions little advantage is gained from trap speeds greater than 8,000 to 10,000 liters/sec. Since such speeds are obtained from single traps² of moderate size, the use of very large traps, or of two or more traps per tank, does not appear to be justified.

3.2 Design and Location of Traps. Traps of three types are used in Alpha I, Alpha II, and Beta systems, each system requiring a dif-

ferent type. These are shown in Fig. 3.1, and their essential dimensions are given in Table 3.3. All are welded stainless-steel construction, and all are supported from circular steel plates that serve as cover plates at the ports through which the traps are inserted and

Table 3.3—Characteristics of Liquid-nitrogen Traps

Characteristic	Alpha I	Alpha II	Beta
Bulb length $\times$ diameter, in.	13.5 $\times$ 8	13.5 $\times$ 8	30 $\times$ 6
Filling-tube I.D., in.	$\frac{7}{8}$	$\frac{7}{8}$	1 $\frac{3}{8}$
Capacity, gal	2.4	2.4	3.4
Surface area, sq in.	345	345	565
Effective speed, ^a liters/sec	23,000	23,000	37,700
Bin-to-trap conductance, ^b liters/sec	30,400	50,700	61,300
Bin-to-trap speed, ^c liters/sec	13,100	18,200	23,300
Bin-to-pump speed, ^d liters/sec	6,300	12,000	6,000
Obstruction ratio, ^e %	6.6	Negligible	34
Consumption, ^f gal/day	8	21	31

^a Effective speed = $100 \times \frac{2}{3}$ trap area

^b Bin-to-trap conductance = $\frac{100 \times \text{duct area}}{1 + \frac{3}{4} L/D}$

^c Bin-to-trap speed: $1/S = 1/S_T + 1/C_D$; S_T = trap speed; C_D = duct conductance

^d Bin-to-pump speed: average measured speed

^e Obstruction ratio = $\frac{\text{sectional area of trap}}{\text{sectional area of duct}} \times 100$

^f Consumption: estimate based on measurements

removed from the systems. Supporting members, including the filling tube, are designed to reduce conductive heat transfer to a minimum. The surface areas and resulting pumping speeds of all traps are considerably greater than necessary, but the resulting large capacity is a distinct advantage in prolonging the refill period.

The location of traps in the systems (Chap. 1, Figs. 1.1, 1.2, and 1.3) involves conflicting considerations. Placing the traps in the tanks, thus assuring maximum conductance,^a results in their being directly exposed to radiant heat and ion bombardment with resulting high rate of consumption of refrigerant. Placing them well back in the tank duct avoids this exposure but introduces unwanted tank-to-trap impedance. In any case, the traps must be accessible for filling and servicing.

The location of the Alpha I trap at the bend in the tank-to-pump duct results in a considerable loss in speed of trap pumping at the tank, but protects the trap well from ion bombardment and from radiant heat. Also, it places the trap within the relatively cool plenum chamber. Consequently, the rate of consumption of liquid nitrogen is less in the Alpha I system than in the other systems.

The location of the Alpha II trap is more favorable in so far as conductance is concerned, and it probably has a higher pumping speed than the Alpha I trap in spite of its location in a relatively dead end space above the pump-head valve. Because of its direct exposure to heated parts of the system, however, its consumption rate is high.

The Beta trap also is exposed to direct radiation from within the tank and has a correspondingly high consumption rate. Since recovery of charge material is important in the Beta process, this trap is designed to act as a cold baffle, and its sectional area is 34 per cent of that of the duct in which it is mounted. Conductance from tank to pump is, therefore, materially reduced by the presence of the trap.

3.3 Servicing of Liquid-nitrogen Traps. The servicing of the liquid-nitrogen traps involves (1) maintaining an adequate supply of refrigerant in the traps throughout the bake-out and production phases of a run and (2) removing, cleaning, and reinstalling of traps at the termination of every run.

Trap refilling is carried out on a routine schedule varying from 2 hr in Alpha I to 20 or 30 min in Beta. These schedules were adopted to prevent wide fluctuations of refrigerant level since any great reduction in level results in the release of condensable material to the system. Inability on the part of those responsible to maintain fairly constant refrigerant level in the traps has been a common cause of pressure variation and consequent operating trouble.

Methods of refilling traps are discussed in a later section dealing with the problems of handling and distribution of liquid nitrogen.

At the termination of each run, the trap is removed and cleaned. Alpha traps are simply washed with hot water and wiped dry. In the case of Beta traps, where collected charge material has recovery value, the traps are steamed and wiped dry. Drippings and wiping rags are sent to recovery.

3.4 Handling of Liquid Nitrogen. The storage and distribution of liquid nitrogen in the plant presented serious problems because of the great quantity of material involved and the high rate of loss in handling and in storage.

Table 3.4 shows the average consumption of liquid nitrogen by buildings over a one-year period of normal operation. Figures are

based on receipts at the plant and include all plant storage and handling losses. On this basis, the average rate of consumption of liquid nitrogen is well over a million gallons per month.

According to initial plans, liquid nitrogen was shipped to the plant area in insulated tank cars of 5,000 to 8,600 gal capacity and on receipt was transferred to suitably insulated storage tanks. Two of

Table 3.4—Consumption of Liquid Nitrogen

Building	Track No.	Average monthly consumption, thousand gal	Average daily consumption, gal	Traps serviced	Gal per trap per day
Alpha I	1, 2	96 $\pm$ 15.25	3,202	192	16.7
	3, 4	103 $\pm$ 10.7	3,453	192	18.0
	5	77 $\pm$ 16.0	2,568	96	26.8
Alpha II	1, 2	141 $\pm$ 17.6	4,700	192	24.5
	3, 4	135 $\pm$ 19.8	4,503	192	23.5
Beta	1, 2	130.5 $\pm$ 32.6	4,661	72	64.7
	3, 4	132.0 $\pm$ 32.7	4,715	72	65.5
	5, 6	117.8 $\pm$ 18.9	4,208	72	58.4
	7, 8	121.8 $\pm$ 14.4	4,351	72	60.4
Total consumption:		36,361 gal/day			
		1,090,830 gal/month			
		13,271,765 gal/year			
Cost at \$0.28/gal:		\$10,181.08/day			
		\$305,432.40/month			
		\$3,716,094.20/year			

these storage tanks, of 13,000 gal capacity each, were provided. From the storage tanks, the liquid nitrogen was transferred to portable containers for distribution to the various operating buildings. Two types of transfer containers were used. Standard 7- and 14-gal metal Dewar flasks were used for supplying laboratories, test systems, etc., and tanks or dollies of 60 gal capacity were used for supplying all production systems. These dispensing tanks, Fig. 3.2, were specially designed for use in the routine refilling of traps in the production systems.

Insulation of tank cars, storage tanks, and dispensing tanks was provided by double-wall construction in which the inter-wall was filled with a finely divided insulating material and evacuated to a pressure of 150 μ Hg or less.

High rate of consumption of liquid nitrogen and an increase in plant size beyond that originally planned combined to render initial storage

Fig. 3.2— Liquid-nitrogen dolly.

and distributing facilities inadequate. Plans made to relieve this condition also included the reduction of handling losses and the assuring of a continuous supply of liquid nitrogen to each production building.

Storage and distributing systems were, therefore, proposed and eventually provided for each building. Each of these systems consists of a storage tank of the type and size previously used and insulated pipe lines for conducting liquid nitrogen from this tank into the various production areas. The storage tank is located on the roof of the building, and flow through the distributing lines is by gravity. These lines are so constructed that when no liquid nitrogen is being used the gas formed in the lines forces the liquid from the pipe into the storage tank, thereby reducing line losses.

The distribution lines are of 2½-in. copper tubing surrounded by a 14-in. welded-steel jacket. The interspace is filled with Santocel, and maintained under vacuum by the continuous pumping of a Kinney pump. The choice of Santocel over other types of insulation, such as rock-wool, cork, copper foil, and aluminum foil-expanded paper lamination, was based on experimental studies showing the superiority of this type of insulation at pressures easily attainable with mechanical pumps.

Within each production area the liquid nitrogen is transferred to the dispensing tanks originally provided for distribution to the various traps.

The installation of these systems in the nine production buildings, and the addition in the meantime of one area-storage tank, increased the total storage capacity for the area to $12 \times 13,000 = 156,000$ gal or about 4½ days' supply. This supply is adequate for meeting normal delivery fluctuations, and excessive reserve is to be avoided because of high storage losses. Interruptions of supply to the operating areas are also eliminated by these systems. Such interruptions were not uncommon when delivery to the buildings was in small (60-gal) batches.

So far the distribution of liquid nitrogen through pipes has been carried only to individual operating areas. Proposals to extend distribution to the individual traps have been frequently made, and such proposals offer obvious advantages. However, difficulties are also involved, especially in applying such a system to a plant that was not originally designed for such servicing.

The chief advantages of such a system would appear to be (1) a reduction in handling losses,¹ (2) a reduction in trap-servicing personnel, and (3) the possibility of developing full automatic trap-filling that would assure constant refrigerant level.

With this last end in view, a float-type control was designed, tested, and carried through several stages of improvement by C. B. Graham and J. W. Withers, CEW-TEC. But work in this direction was discontinued as progress was made in the elimination of the use of liquid nitrogen in the production systems.

3.5 Liquid-nitrogen Losses. Frequent mention has been made of the losses involved in the storage and handling of liquid nitrogen. A breakdown³ of these losses is given in Table 3.5. The seriousness of these losses, viewed in the light of total cost involved, is easily apparent. Of all the liquid nitrogen received at the plant approximately one-third (34.2 per cent) is lost in storage and handling. If the conservative \$10,200 per day cost figure of Table 3.4 is accepted, this

Table 3.5—Distribution of Liquid-nitrogen Losses

Type of loss	Loss, gallons per day per building				Total
	Alpha I bldgs. 1, 2	Alpha I bldg. 3	Alpha II bldgs. 1, 2	Beta bldgs. 1 to 4	
Car to storage	28	34	86	26	366
Tank storage	28	28	28	28	252
Tank to track	32	22	83	21	336
Transfer to dolly	209	217	920	466	4,339
Dolly storage	46	23	92	46	483
Dolly to trap	374	401	1,705	727	7,467
Trap consumption	1,290	1,400	5,950	2,400	25,480
Total to track	2,007	2,125	8,864	3,714	38,723
Vacuum dock and ion gauge	630	720	840	850	7,060
Total consumption	2,637	2,845	9,704	4,564	45,783

represents a daily loss of \$3,400. An average shipping loss of 14 per cent between factory and plant does not appear directly, but is absorbed in the unit price of 28 cents per gallon.

If the various losses are examined individually with a view to effecting substantial reductions, these points are apparent:

1. Any reduction in shipping loss is unlikely as long as the liquid nitrogen is supplied by freight shipment from a remote source.
2. Since transfers of liquid nitrogen to the dispensing dolly and from dolly to trap constitute the major sources of loss, it is in these operations that any real hope of loss reduction lies. An analysis of losses during transfer to and from the dispensing dolly gives little basis for hope that these can be substantially reduced by methods short of complete elimination of use of the dolly. For this reason, proposals to pipe liquid nitrogen to individual traps, thereby eliminating the dolly, were considered with favor. Dolly-storage losses would also be eliminated by pipe distribution, and although a substantial increase in pipe-line loss would undoubtedly result a considerable net saving seems assured.

3. Of the remaining sources of loss—loss in transfer to storage, loss in storage, and loss in pipe line—none can be entirely eliminated, nor would the saving be great if it could. In fact, it is doubtful if any of these losses could be very greatly reduced.

3.6 Reduction in Trap Consumption of Liquid Nitrogen. In addition to losses occurring in storage and handling of liquid nitrogen, it is evident that losses of considerable proportion occur within the trap. Trap efficiency, calculated from the ratio of heat liberated by all vapor condensed on the trap during a run to the heat absorbed by the liquid nitrogen used, is less than 0.1 per cent. Thus, the possibility exists of effecting relatively large savings of liquid nitrogen in the trap itself. All proposals for realizing such savings are based on one or both of two considerations:

1. High rate of consumption results from a high rate of heat-transfer to the trap from surrounding bodies and may be reduced by limiting this heat transfer.

2. Total consumption per operating cycle depends on the length of time for which the trap is refrigerated and may be reduced by limiting the use of the trap to the bake-out period of the cycle.

Limiting of heat-transfer to the trap has been approached in various ways. In all cases it is easily shown that heat transfer by gaseous convection or conduction is negligible at existing pressures. Likewise, thermal conduction through trap supports can account for only a very small part of the total heat transfer. It follows that radiant heating is primarily responsible for low thermal efficiency of the trap, and all attempts to improve efficiency have been based on this premise.

The following four methods of reducing trap consumption of liquid nitrogen have been tested experimentally.

(a) **Reduction of Effective Trap Area.** Reduction of the cold-trap area has been effected by substituting a trap of small diameter for the standard Beta trap, by operating with the Beta trap only partially filled, and by operating with the Beta trap partially withdrawn from the duct into a well-insulated side chamber. In all cases, adequate pumping action was provided by the smaller trapping area. In the first two cases, however, losses arising from more frequent trap-refilling tend to neutralize any savings resulting from reduction in trap area. In the last case, that part of the trap withdrawn from the duct becomes, in effect, a reservoir for supplying the exposed part of the trap. If the side chamber is effectively insulated this is an efficient reservoir, and trap filling with its associated losses is required less frequently than at present. Thus a twofold economy is realized.

(b) Radiation Shielding of Trap. Numerous tests involving the shielding of traps from radiation by the interposing of one or more sheets of metal between trap and radiation source have been made. All tests show that such shielding can be highly effective in reducing heat transfer. The principal problem is that of designing shields to give the maximum reduction in radiation while interfering least with trapping speed, duct conductance, trap servicing, etc.

The following shielding installations are described as typical of two extreme types. Both types were tested in Alpha I systems.

In the first installation, the shield consisted of an open-end cylinder of stainless steel surrounding the trap and supported from the trap support braces. Later, a larger coaxial cylinder was added and still later the space between the coaxial cylinders was filled with several layers of aluminum foil separated by expanded paper. Since the shield was removed with the trap for cleaning, its over-all size, and therefore its distance from the trap, was limited. The resulting loss in trapping speed was within allowable limits, and an appreciable reduction in consumption of liquid nitrogen was effected. Interference of the shield with trap cleaning was an objectionable feature.

In the second installation, shielding consisted of ten layers of aluminum foil separated by alternate layers of expanded paper and held between two stainless-steel sheets. All duct walls in the vicinity of the trap were lined with this material, and stainless-steel baffles slightly larger than the trap section were so placed as to shade the trap from the high-temperature radiations from tank and diffusion pumps. With this arrangement savings of liquid nitrogen up to 35 per cent were realized; the loss in trapping speed was less than 5 per cent.

While shielding of the types described is applicable to the Alpha I traps, and possibly to those of Alpha II, space limitations preclude their use in the Beta system.

Other measures proposed, but not adequately tested, as a means of reducing radiant heat transfer may be mentioned:

1. Relocation of the trap to avoid exposure to direct radiation from high-temperature parts of the operating system.
2. Shields or baffles of limited size placed to shade the trap from localized regions of high temperatures.
3. Design of the trap so that the smallest possible area is presented to the regions of highest temperature.
4. Cooling of the duct walls by external refrigerating coils.

Two plans have been considered for reducing the time during which the trap is used. Any such plan must provide adequate trapping during

a period corresponding roughly to bake-out and must prevent reevaporation of the condensed material in the system after trapping is no longer required.

(c) Retractable Liquid-nitrogen Trap. Late in 1943, the construction and performance of a retractable liquid-nitrogen trap developed at UCRL was reported.^{2,4} This trap is designed so that it can be completely isolated from the vacuum system at any stage of operation by withdrawing it into a side chamber. A Wilson seal at the trap neck permits this retractile motion, and at full retraction a disk attached to the trap forms a closure between the side chamber and the system that is vacuum tight at small pressure differences. While in its retracted position, the space surrounding the trap is maintained under rough vacuum, and vapors that are liberated as the refrigerant is exhausted are removed by the roughing pump. If the trap is refilled it may be reinserted in the system should occasion demand. Since this is not usually necessary, no liquid nitrogen is required during the major part of the production cycle. Thus during an eight-day operating cycle liquid nitrogen may be required for only one day, giving a net saving of some 88 per cent.

(d) Clamshell-type Trap Insulation. Figure 3.3 shows a typical trap of the clamshell type⁵ as designed and tested under the direction of O. E. Miller, CEW-TEC. The semicylindrical shells are of stainless steel inside and out, and the space between inner and outer shells carries 12 to 15 layers of aluminum foil separated by alternate layers of expanded paper. When the trap is in use the shell is open as shown; its speed, though reduced, is still adequate under these conditions. When the trap is no longer needed, the halves of the shell are closed by means of an external control operating through a sylphon seal. To prevent evaporation of the material collected on the trap, the supply of liquid nitrogen must be maintained throughout the remainder of the run. Because of the highly efficient shielding provided, the rate of consumption in the closed-off trap is only 4 to 9 per cent of that normally experienced. Thus it is estimated⁵ that in the Alpha plants a net saving of 79 per cent would be realized.

As in the case of radiation shielding, space limitations preclude the use of the clamshell-type of trap in present Beta installations.

That none of these methods of reducing the consumption of liquid nitrogen in the traps progressed beyond the experimental stage prior to the end of the war is directly attributable to the wartime policy requiring maximum plant production. With the close of the war, however, attention was turned to economy of production, and the whole liquid-nitrogen conservation problem came up for consideration. By

this time, or very shortly thereafter, all Alpha units were shut down, and attention was concentrated on the reduction of liquid nitrogen in the Beta plant.

A survey of storage and handling losses and a review of the applicable methods of reducing trap consumption indicated that any great reduction in consumption, through the realization of small savings at a number of points, would be expensive, difficult, and time-consuming.

Fig. 3.3—Clamshell-type trap and insulation.

It was at this point that a decision was made to examine thoroughly the possibility of operating the Beta system without liquid nitrogen.

3.7 Elimination of Liquid Nitrogen. Up to the end of 1945, no serious effort had been made to operate production systems without liquid nitrogen. All evidence indicated that, although possible, such operation would result in some reduction in production. How serious this reduction would be, however, or to what extent it might be compensated for by altered procedures, was not known. Frank A. Knox and Alan J. Samuel were assigned the problem of answering these questions.

Several production tanks were designated for experimental use, and a series of tests were planned to provide answers to the following fundamental questions:

1. Is operation of the Beta system without liquid nitrogen possible?
2. What are the resulting losses in production, and at what points do they occur?
3. What changes in equipment or procedure can compensate for these losses, and how complete will this compensation be?
4. What secondary problems such as material recovery and total pump load will arise, and how may they be met?
5. Will elimination of liquid nitrogen reduce or increase the unit cost of the plant product, and by how much?

The first question was answered affirmatively when experimental tanks having the traps removed and the trap port blanked off were consistently carried through essentially normal operating cycles.

The resulting losses in production could be determined only by a statistical comparison of the operating data from experimental and normal tanks. First tests showed only moderate production loss; moreover, this loss was due in a large part to increased pump-down time.

Changes in operating procedures were adopted to compensate for a major part of the increase in pump-down time, and it was shown that operation without liquid nitrogen could be accomplished with an increase of 20 per cent or less in pump-down time and an over-all loss in production of less than 1 per cent.

With the removal of the liquid-nitrogen trap from the system, the small amount of charge material previously recovered from the trap surface was dissipated in the vacuum system, thus reducing total immediate recovery. Baffles of various types have been designed to compensate for this loss. No final adoption has been made of a baffle providing maximum recovery and minimum impedance, but it has been shown that baffles providing lower impedance and higher recovery than that provided by the traps are possible.

Removal of the trap increases the conductance of the tank duct thereby offsetting, in part, the loss of vapor pumping action previously provided by the trap. However, the fact that all gas and vapor must now be removed by the pumps has resulted in an overloading, especially during the bake-out period, of the fore pumps in those systems using small-size mechanical pumps for backing purposes. This condition has been made more acute by the simultaneous elimination of dry-ice traps in the fore-vacuum system and is being met by the use of large-capacity roughing pumps for backing purposes during this period.

The cost of liquid nitrogen, at the time these tests were under way, was estimated as about $3\frac{1}{2}$ per cent of the total plant-operating cost. Since elimination of this refrigerant resulted in a loss of only 1 per cent in production, it was apparent that complete elimination was economically feasible.

Elimination of liquid nitrogen throughout the plant progressed through a series of stages to allow time to accumulate more reliable statistical data and to familiarize operating personnel with the altered operation and procedures. From a few test tanks, the program was expanded to include a whole track. Six test tanks in each of the plant buildings were then added to the program. With results continuing favorable, plant-wide elimination of liquid nitrogen was put into effect early in June 1946 without serious disturbance of production, and it is on this basis that operation is now being carried on.

It is unsafe to assume that other systems, such as those of the Alpha plants, could have also been operated without liquid nitrogen without incurring serious production loss. Outgassing and bake-out were always less serious problems in Beta than in Alpha, and this might make the difference between success and failure. It is true, however, that liquid-nitrogen trapping is unnecessary, even in Alpha systems, throughout a major part of the operating cycle, and great savings could be realized by designing future systems to take advantage of this fact.

Additional experience in both Alpha- and Beta-type systems confirmed the feasibility and economy of operating such systems without liquid-nitrogen traps.

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Chapter 4

VACUUM-SYSTEM INSTRUMENTATION

By W. R. Perret and G. W. Monk

1. INTRODUCTION

The instrumentation required for practical operation of vacuum systems in a manufacturing process depends upon the complexity of the system and the processes that take place within the system. Fundamentally, such instruments are responsive to residual gases within the system, and may be required (1) to indicate pressure, (2) to signal changes between certain critical pressure ranges, (3) to operate process controls within critical pressure ranges, or (4) to serve in the detection and location of leaks in the system.

This chapter deals with the nature and performance of the instruments used in the Alpha and Beta plants and with the development work done in connection with both plant and laboratory instrumentation of vacuum systems. Brief discussions of the use and performance of certain commercial instruments that were tested in the laboratory are also included. Only the indicating, signalling, and controlling functions of the instruments are discussed in this chapter; the following chapter deals with leak-detecting instruments and techniques.

It is the function of a vacuum gauge to register the quantity of residual gas in an evacuated system at any time and at some particular point within the system. To accomplish this, instruments are designed to be responsive to one or another of the various physical characteristics of gases. For rough measurement of relatively high pressures, usually above a few millimeters of mercury, purely mechanical means of balancing against atmospheric pressure are used in the open- or closed-tube manometer or the Bourdon gauge. The McLeod gauge uses the pressure-volume relation of Boyle's gas law to achieve reasonable precision over a wide range of pressures in the high-vacuum region, for example, below 1,000 μ Hg and above 1 μ Hg.

Thermal phenomena such as heat-transfer and thermal-mechanical energy conversion provide the means for low-pressure indication by the Pirani, thermocouple, and Knudsen gauges. Electrical properties of gases and gaseous ions are used by the thermionic and Philips gauges to measure pressures below 1μ Hg in the extreme high-vacuum range. Most of these gauges give continuous indications of pressure, but the McLeod gauge requires external actuation and, therefore, indicates intermittently.

The location of a gauge in a vacuum system has a very important influence upon its function, beyond the obvious fact of its connection into either the fore-vacuum or the high-vacuum portions. Nearly all gauges are attached to a vacuum system by means of a length of pipe or tubing that includes a valve. The outstanding exception to this is the Philips gauge, which in this plant has been located within the main high-vacuum chamber. The critical factor involved in the gauge location is the impedance offered to gas flows by various parts of the system, particularly by such things as valves and baffles. Such impedances will produce pressure gradients within the system and lagging response in side branches such as gauge connections. Another highly important factor controlling the effectiveness of a gauge is the combination of line impedance and leakage rate of the connection between the gauge and system. The usefulness of gauges in some plant installations has been limited by these factors.

The customary system of mensuration employed in vacuum gauging is based on absolute or standard barometric pressure, which is defined as the pressure that will support a column of mercury 760 mm high at sea level at 0°C within a sealed glass barometric tube. Vacuum gauges are calibrated in terms of the length of mercury column that would be supported by the residual gas pressure acting on the gauge. Except in the case of Bourdon gauges, on which the calibration is usually given in inches, the customary units are millimeters or microns. The micron is the more convenient unit for very-high-vacuum measurements since it is 10^{-6} meters or 10^{-3} millimeters. In certain applications, particularly in those concerning flow of gases at very low pressures, it is more convenient to deal with molecular mean-free-path length rather than gas pressures. Some consideration has been given to the use of such a parameter, but the system of units usually used is that of the millimeter and micron of mercury column.

Numerous types of vacuum gauges had been developed and were commercially available prior to construction of the electromagnetic plant. Four general types of gauges were installed in the original plant units, Bourdon, McLeod, and Pirani gauges in the fore-vacuum

systems and thermionic gauges in the high-vacuum systems. Development of a hot-wire vacuum switch at UCRL led to the installation of these instruments in later plant units as a safety control, and development work on the Philips gauge both at CEW-TEC and at UCRL led to its widespread adoption in the process tanks throughout most of the plant. The major part of developmental work on the thermionic-gauge power supply was done at UCRL prior to construction of this plant, and only a few changes have been made in that circuit here. As problems concerned directly with getting the plant vacuum systems into satisfactory operation became less pressing, new approaches to vacuum-instrumentation problems were undertaken here. This volume is concerned with those phases of the work that were directed toward

Table 4.1—Ranges of Pressure-measuring Instruments

Gauge	Useful pressure range, μ Hg	
	Upper limit	Lower limit
McLeod (Stokes)	700	1
McLeod (Westinghouse)	700	1
Allis-Chalmers Pirani	200	1
DPI Pirani	750	1
35T Thermionic	2	0.001
Alpha II a-c Philips	0.8	0.02
Alpha II d-c Philips	1.2	0.001
Beta d-c Philips	0.5	0.001
Bourdon	760 (mm Hg)	20 (mm Hg)*

*Approximate.

improving instruments and methods for operation of the plant vacuum systems. Another phase of the work concerned with measuring and controlling vapor pressures in the source units has been described in National Nuclear Energy Series, Division I, Volume 8, which deals with vapor production and control.

The useful ranges of the various instruments used in the plant for measurement of pressures cover a wide spread as indicated by Table 4.1.

Plant vacuum instruments can best be discussed as (1) fore- or rough-vacuum instruments and (2) high- or fine-vacuum instruments.

Gauges of both classes may be required to indicate pressure, to signal pressure changes, or to control operating processes in case of excessive pressure.

2. PLANT FORE-VACUUM INSTRUMENTS

2.1 Bourdon Gauge. The Bourdon gauge is a widely used instrument for the measurement of fluid pressures. It is usually comprised of a tube of elliptical cross section formed in a circular arc and rigidly fixed at one end. A system of levers and gears attached to the free end operates a pointer, which moves over a calibrated scale. Differences of pressure between the gas within the tube and that surrounding it alter its curvature, with a consequent movement of the pointer.

Bourdon gauges were initially installed on the fore-vacuum systems of all dry docks and on both roughing- and finishing-header systems in all process buildings with the exception of the finishing headers in the last two Alpha II tracks. Most of these gauges were of the vacuum type calibrated from 0 to 30 in., the zero end of the scale corresponding to atmospheric pressure. The exceptions to this case were the compound gauges, used on most dry docks, which included a pressure range up to 30 lb/sq in. for use in pressure-testing units for leaks while in the dry docks.

These gauges were used very little on the vacuum systems and in some cases were either removed from the systems entirely or isolated by valves to eliminate them as possible sources of leakage. Their principal function has been for checking quickly and roughly the condition of the header system during the early stages of pumping out a unit or following a major vacuum accident.

2.2 McLeod Gauges. McLeod gauges are the most widely known of the high-vacuum gauges. They operate by trapping and compressing a known volume of gas at the pressure of the evacuated system in such a manner that application of Boyle's law to the measured final volume and pressure permits determination of the unknown initial pressure as a function of the initial volume. Because of the compression, which is a part of its operation, the McLeod gauge measures only that part of the total pressure which is represented by the permanent gases and the uncondensed portion of the condensable vapors. Consequently, it does not give a true measure of the total pressure within a vacuum system and must under some conditions be used in conjunction with another gauge that measures total pressure, such as a Pirani.

Two types of McLeod gauges were in use in this plant, the Stokes tilting gauge, Fig. 4.1, and the Westinghouse cam-operated gauge, Fig. 4.2. The Stokes gauge was used throughout the initial plant units on all header systems and dry docks. The Westinghouse gauge, which was introduced later, has been installed in several of the newer plant

Fig. 4.1—Stokes McLeod gauge shown (above) in the position for reading and (below) for evacuating.

Fig. 4.2—Principle of the Westinghouse McLeod gauge. The scale is illuminated from behind.

units. Because of their different modes of operation and certain difficulties inherent in their use, it has become an established practice to use only one type of gauge throughout a complete track or building.

The Stokes gauge consists of a glass unit in which mercury is contained in a reservoir when the gauge is in a horizontal position. When the gauge is rotated to a vertical position, the mercury flows out to trap and compress the gas contained in a bulb and closed capillary. A sheet-metal case provided with a swivel mount holds the glass unit, which is connected to the vacuum system by a short length of flexible rubber tubing. Figure 4.1 illustrates the operation of this gauge by showing it in the initial and final positions. This gauge, like all other McLeod gauges, indicates the pressure within the bulb at the time of trapping only and must be rotated to the horizontal position and allowed to remain thus for a short time before another observation can be made.

The performance of these gauges has been reasonably good. Their accuracy is adequate for use on fore-vacuum headers, subject only to the inherent disadvantage of all McLeod gauges that no response is possible to partial pressures due to condensable vapors, with the consequence that erroneously low pressures are frequently recorded, particularly in the early stages of pump-down. Considerable difficulty has been experienced with breakage of the glass portions of these gauges. Mercury hammer, resulting in some cases from rapid rotation of the gauge but in most cases from jarring the gauge in order to free droplets of dirty mercury from the capillary, caused a high mortality among Stokes McLeods, particularly during the earlier stages of the project.

The Westinghouse McLeod gauges are made almost entirely of metal, the only glass parts being a closed capillary and a reference capillary with a relatively large closed-off space at its top. Mercury, contained in a metal bellows reservoir, is forced upward, to trap and compress the gas contained in the gauge, into the closed capillary by means of a cam, operated by a handle, shown in Fig. 4.2. This gauge is connected to the vacuum system through a metal pipe with a rubber-gasketed flange connection.

The performance of these gauges has been somewhat better than that of the Stokes McLeods, principally because of their less fragile construction. However, the mercury column is extremely sensitive to very slight rotation of the cam, particularly at the final part of the movement. This makes the gauge somewhat difficult to adjust for proper reading without considerable practice. Some failures, mostly cracked bellows and leaks in the gaskets which seal the capillaries,

have occurred, but the number of such failures is much less than those suffered by the Stokes gauges.

During the first year of plant operation, only the Stokes type of McLeod gauge was used and a very large number were shattered, many beyond repair. For a period an acute shortage of usable gauges existed. This situation accelerated the establishment of a planned gauge-service area. A shop for servicing and maintaining an adequate supply of clean McLeod gauges was set up. This shop cleans and, when possible, has repairs made on all gauges delivered to it, cleans the mercury from the gauges, and maintains a supply of clean gauges and mercury for the entire plant. A procedure has been established which requires that all dirty gauges having mercury stuck in the capillary must be returned to the shop for servicing. These servicing facilities and this procedure have resulted in a considerable saving to plant operation, since it was a general occurrence that a broken McLeod gauge would cause the fore vacuum to be lost, with consequent loss of operation and considerable damage to several process units.

During the peak operation of the Alpha and Beta plants, the McLeod servicing shop received and sent back to the plant an average of 20 gauges per week. Currently, with only the Beta plant operating and with about half that plant equipped with Westinghouse gauges, the average servicing figure is eight gauges per week including those being given routine checks.

2.3 Pirani Gauges. The Pirani type of high-vacuum gauge depends upon the variation with pressure of thermal conductivity of gases to cool two electrically heated filaments in a bridge circuit. Within a pressure range of from 50 mm Hg or more to 1μ Hg, the unbalance of the bridge circuit can be indicated by a meter suitably calibrated in terms of the gas pressure. This type of gauge gives continuous indication of pressure and can be readily adapted to give continuous records, to signal, or to control other circuits within specific pressure ranges. Since the operation of a Pirani gauge depends upon both the thermal emissivity of the filament and the thermal conductivity of the gas, it follows that its calibration will be adversely affected by contamination of the filament surface and similarly its calibration will be different for gases of different molecular masses and specific heats.

Two types of Pirani gauge are used in the plant fore-vacuum systems. One of these, the Allis-Chalmers gauge, Fig. 4.3, contains four filaments, each in a glass envelope, two of which are sealed off at atmospheric pressure and two of which are connected to a suitable opening in the vacuum system. The four filaments are connected into

a Wheatstone-bridge circuit to which a constant, regulated direct current is supplied and the unbalance of the bridge circuit is indicated by a meter. A contacting device with which the meter is equipped divides the meter range into two or three segments and may be used to operate relays, which give an alarm, or to interlock process circuits when conditions cause the pressure to enter the critical range.

Fig. 4.3—Allis-Chalmers Pirani gauge circuit.

A portable Pirani gauge, Fig. 4.4, produced by Distillation Products, consists of two filaments. One of these is contained in a sealed, evacuated glass envelope, the other is contained in a glass envelope which may be connected to the vacuum system. A special bridge circuit into which the two filaments are connected as two arms is supplied from a direct current source manually adjusted to give a constant input voltage, the bridge unbalance being indicated by a suitable meter.

The distribution of Pirani gauges throughout the plant is shown in Table 4.2. This table shows the functions of the gauges and the final use made of them.

The D.P.I. portable Pirani gauges are used in the plant for testing purposes on the main vacuum systems and have been installed on dry docks in the Beta plant for use in conjunction with the helium leak detectors. Their function is pressure indication only.

Fig. 4.4—Distillation Products, Inc., Pirani gauge circuit.

Initially, Pirani gauges were serviced in the plant by the electrical division, but more recently most of the service work on these gauges has been conducted in the vacuum-electronic shop. Records of this shop show that the Beta plant requires servicing of one or two Allis-Chalmers Pirani gauges and about three or four D.P.I. Pirani gauges per week. Some calibration of both types of gauge is performed in the plant by the vacuum staff.

2.4 Hot-wire Vacuum Switch or Wieske Gauge. Early in the experimental program that led to the design and construction of the production plant it was recognized that some means was needed for the removal of power from the diffusion pumps and process supplies if the pressure in the tank increased above safe levels. When the preliminary designs for the plant were made, it was decided that a

number of tanks would be serviced by common finishing and roughing headers. This situation made even more important the development of an instrument which would signal and interlock with the high-voltage cubicle if excessive pressures occurred in the vacuum system. Several means of providing a fore-vacuum interlock were tried, including a mercury switch and Geissler discharge, but none of these

Table 4.2—Distribution and Use of Pirani Gauges

Building	Tracks	Roughing header	Finishing header	Dry docks
Alpha I	1, 2	Indicating	Indicating and signal	Indicating
	3, 4	Indicating	Indicating and signal	Indicating
	5	Indicating	Indicating and signal	Indicating
Alpha II	1, 2	None	None	Indicating
	3, 4	None	None	Indicating
Beta	1, 2	Indicating and signal	Indicating, signal, and control	Removed
	3, 4	Indicating and signal	Indicating, signal, and control	Not used
	5, 6	Indicating	None	Not used
	7, 8	Indicating	None	None

was particularly successful because of the high pressure required for actuation or the instability of the cutout point. The first successful instrument was a modification of the Allis-Chalmers Pirani gauge with its contact-making meter.

It was apparent, however, that the long contact interval (1 min) and the excessive cost of this instrument made it advisable to develop one that would operate practically instantaneously and would be somewhat cheaper. On Nov. 18, 1943, UCRL reported a hot-wire vacuum switch,¹ which is shown in Fig. 4.5.

In this instrument a microswitch is mounted on a rigid bracket to which are also attached two insulated filament mounts. A 0.003 in. tungsten filament attached to these mounts is stretched over a quartz operating rod, which is guided by a bushing and bears against the plunger of the microswitch. Through this filament wire is passed a constant current, which heats it to a temperature determined by the rate of heat dissipation from the wire. The whole assembly is mounted inside a vacuum-tight box connected to the vacuum system. As the pressure in the system rises, the wire cools because of the increasing portion of the heat carried away by gaseous conduction, and the wire shrinks. If the pressure drops the reverse is true. This expansion or contraction of the wire causes the quartz rod, which is guided by the bracket, to open or close the microswitch contacts. Coarse adjustment of the cutout point is provided internally by an

Fig. 4.5—Early hot-wire vacuum interlock switch with a detail of the microswitch and element at the right.

adjusting nut, which moves the filament mounts up and down against a leaf spring. Further adjustment is obtained by varying the current through the filament by means of an external resistor.

The entire assembly is mounted on a heavy mounting plate and supported inside the chamber by springs to reduce the effect of vibration on the action of the switch. Electrical leads for the filament and microswitch are brought out through four Kovar seals. Several models of this gauge were sent to CEW-TEC early in 1944, but little was done about having them installed in the plant until late in 1944 when 72 were ordered for Beta tracks 5 and 6.

Use of these gauges indicated several difficulties. First, the switches did not hold their calibration well, which necessitated frequent checking and resetting. Furthermore, resetting or adjusting of the tension on the hot wire required breaking the vacuum in the gauge and the parts were so delicate that filament breakage was frequent. These difficulties were quite severe and for a time it appeared that the interlock might be abandoned.

In the meantime, considerable work had been undertaken at UCRL to improve the performance of the hot-wire switch.² Reports on this work described changes in the electrical-supply circuit which brought closer together the cutout and cut-on points, improvements in the electrical calibrating devices, and a change of the microswitch from type BZR-8 to type BZR. All these changes tended to improve the performance of the instrument and to add to its usefulness but did not overcome the objection that recalibration required breaking the vacuum and opening the instrument. In addition, the change of the microswitch did not completely remove calibration drift from the troubles present in the gauge. It was established that creep, slippage, and fatigue of the leaf spring supplying the tension in the tungsten wire was the major factor in causing drift. However, CEW-TEC had approved final designs for both Buildings 9204-3 and 9204-4 prior to the UCRL improvements with the consequence that the plant installations do not make use of the improvements.

Independently of UCRL, R. R. Cyr and W. R. Felts, of CEW-TEC, were engaged in talks with Allis-Chalmers, who manufactured the first hot-wire switches for Building 9204-3, relative to improvements of the design incorporating external adjustment through a packing gland and elimination of the leaf spring. Allis-Chalmers submitted a design, shown in Fig. 4.6, which incorporated both these features, and it was adopted for use in Building 9204-4.

In this model, the tension on the tungsten filament is provided by the adjusting screw bearing on the bottom of the pivoted microswitch.

This more positive means of positioning the microswitch with respect to the operating rod and tungsten filament has for all practical purposes eliminated drift. Further, the bellows-spring-mass system

Fig. 4.6—Improved hot-wire vacuum interlock switch showing external adjustment mechanism.

on which the active element is mounted provides adequate isolation from vibration.

This new model has given satisfactory service in Building 9204-4 for approximately six months with most gauges maintaining their original calibration within $\pm 10 \mu$ Hg.

3. PLANT HIGH-VACUUM INSTRUMENTS

Instruments suitable for measurement of pressure in the range from 1 to 0.01μ Hg or less are very different in both principle and construction from those used for fore-vacuum systems. Of the instruments developed for measuring subatmospheric pressures, only the triode ionization gauge and the Philips gauge are readily adaptable to plant use for the measurement in the high-vacuum range. Another, the Knudsen gauge, measures absolute pressure below 0.001μ Hg, but certain features of its construction and operation preclude it from consideration for any but laboratory use. A fourth gauge that might be included in this category is a specially constructed McLeod in which a low-vapor-pressure, low-density vacuum oil is substituted for the customary mercury. Such gauges are, however, difficult to maintain in good operating condition and are not suitable for plant use. The thermionic gauge and the Philips gauge, which are usable within the high-vacuum range, depend upon measurement of the ionization of residual gas. Consequently, because of differences in ionizing characteristics of various gases, the calibrations of gauges will vary depending upon the gas or mixture of gases within the system. The complexity of the factors entering into the ionization process causes computed calibration for gauges in this class to be too unreliable for practical purposes, and calibration must be made by comparison with an absolute gauge, such as a McLeod or a Knudsen, or with a standard gauge established initially by comparison with an absolute gauge.

The thermionic gauge consists of a triode, similar to an ordinary amplifier tube, which is connected by a tubulation to the vacuum system, a suitable power supply to provide a constant, regulated electron-emission current to the triode, and an amplifier to provide the necessary amplification of the ion current so that it may be conveniently measured. The power supply and amplifier are usually located at some remote point to permit convenient use of the gauge indications.

The Philips gauge usually consists of an anode formed of a wire ring and a double cathode formed of two parallel-plane metal plates connected together and parallel to the plane of the anode ring, which is midway between them. The electrodes are placed within a magnetic field of 1,000 gauss or more normal to the cathode and are supplied with either an alternating or a direct potential of about 1,000 to 3,000 volts. The current passed by the gauge and indicated by a d-c meter is proportional to the gas pressure in the gauge within its usable range.

3.1 Thermionic Gauges. The ion-gauge tube adopted for general plant use was a modification of the Eimac 35T produced by Eitel and McCullough. This tube is a standard amplifier triode having a thoriated tungsten filament cathode and a plate connection made through a seal in the glass envelope opposite the cathode and grid press. A 0.5-in. tubulation 2 in. long has been added at the side of the bulb. The choice of these tubes as thermionic gauges for the plant appears to have been one of the many dictated by expediency, since they are not wholly satisfactory although usable. At the time this plant was being planned, the 35T was the only tube of its type which could be made available in the quantities required.

Undoubtedly, many of the difficulties found with this tube during the early days of plant operation were inherent in the general personnel problems of staffing a plant of this type, but during the entire period of operation of Alpha I, the tube life was short and replacement costs were high. Initially, a large proportion of such replacements were probably due to breakage that occurred while the tube was being connected into the system. Such breakage was somewhat less when the tubes were waxed into fittings than with the more recent rubber-gasketed compression fittings, but the latter method was more convenient and introduced fewer leakage problems.

Several factors besides personnel errors, such as breakage and operation under adverse conditions, contributed to the unsatisfactory performance of these tubes. The calibration of the 35T, although generally satisfactory for plant use in the early part of the tube life, became unreliable toward the latter part of its life. The thoriation of the filaments of the 35T has been consistently light so that it has not proved practical to reactivate them and, as a result of occasional operation at excessive pressures, the useful tube life has often been short.

The average life of a 35T in the Alpha I plant, as of May 1945, has been estimated at 400 hr with an average monthly replacement cost of \$1,000 per month per track. Currently, only two tracks of the Beta plant are using the 35T thermionic gauge for general purposes, the remainder of the plant being equipped with Philips gauges and using 35T's for stand-by purposes. However, the average 35T life for these two tracks is 1,200 hr, with a monthly replacement cost of about \$100 per track. It must be noted in connection with this cost figure that the Beta tracks are equipped with less than half as many process tanks as Alpha tracks and that the book value of the 35T tubes has been reduced by 25 per cent. This improvement in life can be credited to the fact that all gauges are now operated with a liquid-nitrogen trap placed between the gauge and the vacuum system, thus preventing

considerable ion bombardment of the filament, and to the fact that the operating personnel are now more experienced. They now have less tendency than in the earlier period to condemn a usable tube or to operate the ion gauge under poor vacuum conditions.

The thermionic gauge operates on the principle that at very low pressures the ratio of ions formed per electron emitted in a tube such as the 35T is proportional to the gas pressure in the tube. It follows, then, that if either the emission current or the ion current is held constant the other will be a measure of the pressure. The customary procedure is to use a constant emission current and interpret the ion current as the measure of pressure. The design of a power supply for such a gauge must consequently take into consideration the maintenance of a constant electron emission from the filament or cathode of the ion-gauge tube and the measurement of the small ion currents derived at low pressures.

The power supplies used with the 35T gauges in the plant were manufactured by the General Electric Company, who furnished them mounted in the process-operating cubicles and in portable metal boxes. As a result of experience with the initial circuit in the Alpha I plant and the first Beta-plant unit, revisions were made in the circuit furnished for the Alpha II plant and for the portable supplies used in the more recent Beta plant additions. Some minor revisions have also been made in the plant as a result of experience with the supplies in operation.

The circuits include, in addition to the necessary sources of filament, grid, and plate voltages, an emission-current regulator, a vacuum-tube voltmeter in the ion-current circuit, and a protective relay circuit. In the original circuit, Fig. 4.7, the emission current was regulated from a 6SJ7 in the 35T grid circuit operating through a saturable reactor in the 35T filament supply to reduce the filament current when the grid current increased. The grid potential was maintained 150 volts positive with respect to ground and the filament was operated at 30 volts positive with respect to ground. The plate of the ion tube was grounded through one of a group of resistors, which were connected through the range-selector switch. The voltage produced by the ion current through the resistor was fed to one grid of a 6SN7, which formed part of the amplifier circuit, to measure the ion current. The grid of a 6SH7 was connected to the circuit that ran from the 6SN7 grid to the 35T plate; this caused the tube to operate a relay contact in the 35T filament circuit when the ion current became too high, thus protecting the 35T against overload due to excessively high pressures. This protective circuit operated when the range selector switch was on any of the four measuring positions but was in-

Fig. 4.7—Ionization gauge supply for the Eimac 35T tube. This supply is used in Alpha I and Beta plants.

operative when the switch was in either the "check" or "zero" position. The power supply was also equipped with means for resetting the relay circuit, for checking the grid-to-plate leakage, the emission current, and the amplifier zero. Several changes have been made in the power supplies having this circuit. These changes include use of a more precise resistor in the highest sensitivity scale range, elimination of the least-sensitive range, giving a maximum range of 2μ Hg instead of 10μ Hg, and incorporation of additional resistors and capacitors in the grid circuits of the 6SN7 and the 6SH7 to reduce oscillation in the circuit. These changes have not removed all faults from the power supplies but have resulted in more satisfactory operation.

The revised circuit which was furnished for the Alpha II plant is shown in Fig. 4.8. In this circuit, emission regulation is controlled by a 6AG7 in the 35T grid circuit which operates a saturable reactor in the primary circuit of a separate 35T filament transformer. Adjustment of the emission current is also provided by a potentiometer that forms part of a voltage-divider circuit from which the grid voltage is derived. In this circuit, only half of the 6SN7 serves the 35T plate circuit as an amplifier for the vacuum-tube voltmeter; the other half serves as the relay-control tube in the 35T filament circuit. A switch and resistor are included in the emission-regulator circuit, which permits operation of the 35T in a stand-by condition with the filament heated but at a sufficiently low temperature to reduce materially the chance of damaging it at relatively high pressures. A few changes of a minor nature have also been made in this circuit at the plant. The principal one of these was the inclusion of a 2,000-ohm resistor in the cathode circuit of the 6AG7 to provide more bias voltage.

Operational faults of these power-supply circuits which have not been corrected include the lack of protection for the 35T when the range-selector switch is in either the "check" or the "zero" position and the failure to provide means for outgassing the parts of the tube by electron bombardment. During periods of high humidity, there has been a tendency for moisture condensation within the power supply to produce very appreciable electrical leakage in the circuit. Both these circuits tend to sustain oscillations in the ion tube at pressures in the upper ranges to such an extent as to render them useless for pressure measurement at times. These oscillations appear to be partly due to the 35T and partly to the power-supply circuit.

The servicing of the ion-gauge power supplies has been performed in a centralized electronics shop during the entire operation of the plant. This shop required most of the time of about 18 employees for servicing these gauges. During the period of peak production of both

Fig. 4.8—Improved ionization-gauge power supply used in the Alpha II plant.

the Alpha and Beta plants, an average of 52 power supplies for ion gauges were serviced each week. Currently, with only two Beta tracks depending entirely on 35T gauges and the other six tracks using them only as stand-by gauges with portable power supplies, an average of eight supplies per week are being serviced in the electronics shop. Reports from the servicing shop indicate that the repairs required for the 35T power supplies are of a minor nature. The portable supplies require more attention than the cubicle units, principally because of pulled-off connector plugs on the leads and because of broken meters and range-selector switches. The latter troubles are fairly common to the cubicle supplies also. Tube replacements, which were initially scheduled on a three-month cycle, are now made as needed.

3.2 Philips Gauges. The Philips gauge is a high-vacuum instrument, which because of its simplicity, ruggedness, and dependence upon a strong magnetic field was ideal for the measurement of pressures within the process apparatus in the electromagnetic plant of this project. The principle of this gauge was discovered by F. M. Penning, of the Philips Lamp Works, Eindhoven, Holland.^{3,4} Very little use, if any, appears to have been made of the Philips gauge in this country prior to its introduction to this project.

Operation of the Philips gauge depends upon the fact that certain geometrical forms of electrodes, subject to both electric and magnetic fields, will provide regions within which electrons may oscillate without draining rapidly to the anode. As a result of these oscillations, the effective length of path of the electrons is increased enough to cause a great increase in the ionization of gas molecules within the confines of the gauge at low pressures. The electrode geometrical form standardized for plant use was a cathode made up of two plane metal plates 1.5 in. square mounted with planes parallel at 0.5 in. separation and connected electrically, and a wire-ring anode 1 in. in diameter mounted with its plane parallel to and midway between the cathode plates, from which it was insulated. The electrodes are mounted with their planes normal to the direction of a strong magnetic field.

Three different types of plant installations were made, principally as a consequence of the simultaneous development and installation of plant facilities which characterized the urgencies of wartime. Initially, Philips gauges powered by a simple 60-cycle a-c source were planned, and the Alpha II plant was originally equipped with such gauges. Further investigation of the a-c gauges indicated that they would not be suitable for Beta plant operation owing to extinction at about 0.003μ Hg pressure. A d-c power supply was developed and

found highly satisfactory for Beta use and much more suitable than with d-c Philips gauges, and Alpha II tracks were equipped with an improved d-c gauge prior to the stand-by order that closed the Alpha process plant.

(a) Alpha II A-c Philips Gauges. The first installation of the Philips gauge designed for plant operation was the a-c powered gauge for Alpha II shown in Fig. 4.9. At the time that specifications were submitted, very little was known about that type of gauge so that the fact that a usable, though not ideal, gauge was obtained can be credited rather to the simplicity and uncritical nature of Philips gauges than to good design. A 3-in. hole in the top of each process tank midway between the sides and 2 ft back of the center line accommodated the gauge, which was mounted on a metal flange equipped with double Hycar gaskets to give a vacuum seal over the hole. The power supply was mounted near the gauge on the magnet coil and the meter and switch were mounted in the heater-control cubicle.

A spark plug-type insulator served both as a vacuum-tight high-voltage bushing and as a support for the anode ring. A ceramic sputter-shield protected most of the spark-plug insulation within the gauge from direct contamination. The cathode was formed as a box with expanded skirts extending to the mounting flange on all sides except for two openings. The cathode was grounded to the flange and was intended to keep the high-voltage insulator shielded both electrically and physically from the ion beam. Cylindrical shields, closed at one end and mounted flush with the flange but slotted on the anti-beam side with five slanting saw cuts, were found necessary after several months of operation. These shields enclosed the gauge units and protected them from the erosion and contamination to which location in the top of the tank made them extremely vulnerable. Figure 4.9 shows a shielded Alpha II gauge that had been operated for over a month without servicing.

The power supply for these gauges, Fig. 4.10, consisted essentially of a transformer, a current-limiting resistor, a d-c microammeter with range shunts, and necessary switches and protective capacitors and resistors.

Prior to the installation of the a-c gauges in the Alpha II plant, it had been found that the d-c gauges were more satisfactory and specifications for conversion to d-c gauges for Alpha II had been issued. However, a delay of several months in the arrival of the new gauges resulted in operation of the Alpha II plant with a-c Philips gauges during the interim.

The a-c gauges were calibrated against 35T thermionic gauges on several occasions in the Alpha plant. The results of these tests

Fig. 4.9—Alpha II a-c Philips gauge shown after a run. The anode ring and the ceramic shield for the spark plug are shown below after the cathode box has been removed.

Fig. 4.10—Circuit diagram for Alpha $\square$ a-c Philips gauge.

showed that, at a field strength of about 3,400 gauss, the gauges with 2,050 volts supplied through a 750,000-ohm current-limiting resistor had an essentially constant sensitivity of $950 \mu\text{a}$ per micron between 0.8 and $0.02 \mu\text{Hg}$. Above $0.80 \mu\text{Hg}$ the sensitivity decreased continuously; below $0.02 \mu\text{Hg}$ the gauge would not fire every "ring-positive" half-cycle, making operation erratic.

For purposes of process operation, a calibration factor of $1,000 \mu\text{a}$ per micron was adopted for convenience and was found adequate.

The a-c Philips gauges were not wholly satisfactory for plant operation because the extinction pressure, $0.02 \mu\text{Hg}$, was above the minimum pressure and also rendered their use for vacuum checking somewhat difficult. Electrical leakage and distortion of the normal gauge discharge which resulted from fouling of the gauge were found to be the causes of frequent extinction at pressures well up in the operating range. The introduction of the slitted shields improved gauge operation to such an extent that the cleaning became essential only after from four to six process runs instead of after each run.

It was also found that at low pressures during normal process operation a small current of inverse polarity was present in as many as 10 per cent of the operating units. This current was believed to occur during the ring-negative half-cycle of the gauge as a result of field emission from sharp-pointed deposits on the anode rings of a dirty gauge. Adequate shielding and cleaning schedules reduced the occurrence of such currents. Inverse currents of this type, which were observed to be as high as $40 \mu\text{a}$, obviously produced false indications of low pressure.

Although the a-c Philips gauges were definitely inferior to d-c gauges, they were used on the last two tracks on the Alpha II plant throughout their operation and were found to be superior to the thermionic gauges that they replaced for all but emergency purposes.

(b) Alpha II D-c Philips Gauges. The d-c gauges for the Alpha II plant were designed to be used in the same location as the a-c gauges. The electrodes, consisting of a wire-ring anode and a cathode formed of a U-shaped piece of sheet metal, were supported on brackets fastened to two spark-plug insulators, which served as bushings through the mounting flange (see Fig. 4.11).

A cylindrical shield closed at one end and standing off from the mounting plate enclosed the gauge. Ceramic sputter-shields protected most of the gauge end of each spark plug from direct cathode sputtering by the gauge.

The Alpha II d-c power supply, Fig. 4.12, which was developed after the Beta d-c supply, included a half-cycle rectifier and filter capaci-

Fig. 4.11 — Alpha II d-c Philips gauge. The upper portion of the figure shows the complete unit; the lower portion shows the shield removed. Both the cathode and the anode are insulated from ground to eliminate the effect of leakage.

Fig. 4.12—Circuit diagram for the Alpha II d-c Philips gauge.

tor in addition to the transformer and current-limiting resistor of the a-c supply.

The negative side of the rectifier was connected through a 250,000-ohm current-limiting resistor to the cathode and the positive side was grounded so that the gauge operated with the cathode at high negative potential and the anode grounded through the indicating meter. This type of supply eliminated the possible difficulties inherent in high-potential positive insulators in a magnetic field and permitted use of bare-wire high-voltage connections between the gauge and the liner-door spark plugs.

As a result of the introduction of full-recovery liners in the first two tracks of Alpha II, it was found desirable that the gauge be more responsive to pressure changes near the ion source. Consequently, the gauges were moved from the top of the tank to a position within the cavity formed by the liner and the liner door. This position was chosen as the result of a series of tests by L. G. Glasser¹⁸ which indicated that response of the gauge to gas bursts and other pressure variations that were critical to operation during the bake-out period was much better near the ion source than at more remote locations because of the pumping impedance of the liner.

Numerous calibrations of these gauges were made in the plant. Sensitivity factors ranged from 4,800 to 5,200 μa per micron in a 3,400-gauss field with approximately 3,000 volts d-c applied across the 250,000-ohm current-limiting resistor and the gauge. A factor of 5,000 μa per micron was used for plant operation.

No low-pressure extinction was observed for any of the Alpha II d-c gauges. The response to pressure was found to be essentially linear up to about 1.2 $\mu\text{ Hg}$ with the 5,000 μa per micron sensitivity. Above 1.2 $\mu\text{ Hg}$, the sensitivity decreased continuously owing to voltage drop in the series current-limiting resistor.

These gauges were cleaned following each process run since they were mounted on the liner and came under the procedure which required cleaning at that time. No servicing problems worthy of note were encountered with the Alpha II d-c gauges and they were considered by the operating and servicing personnel to be superior to both the thermionic and the a-c Philips gauges.

(c) Beta D-c Philips Gauges. The study at UCRL of Philips gauges at Beta field intensity led to the recommendation of direct current in preference to alternating current. Tests at this site bore out the findings at Berkeley that the extinction pressure of about 0.03 $\mu\text{ Hg}$ was too high for proper process operation. Consequently, a design for specifications of d-c gauge and power supply was immediately undertaken. Since design time was very limited, the adequacy of the Beta

d-c Philips gauges must be credited, as was that of the Alpha II a-c gauge, to the inherent characteristics of Philips gauges rather than to rigorous design. A circuit diagram for the Beta d-c Philips gauge is shown in Fig. 4.13.

The problems of electrical leakage between the high-potential electrode and ground are different in the two types of gauges. Obviously, in the a-c gauge the leakage is alternating current and in the d-c gauge it is direct current. In either case, the solution is to feed this current through only the power supply and not through the d-c meter, which measures the gauge discharge current that is responsive to pressure changes.

In the a-c gauge, the circuit for the high-potential electrode includes the d-c meter, the power supply, and a large capacitor in parallel with the meter to by-pass the a-c component due to leakage. The low-potential electrode is grounded.

In the d-c gauge, the circuit for the high-potential electrode includes the power supply through which both the gauge discharge and leakage currents pass. The d-c meter is placed between the low-potential electrode and ground and measures only the gauge-discharge current. In the d-c gauge, both electrodes must be insulated from ground.

Consequently, the Beta d-c gauge was designed as a modification of the Alpha a-c Philips gauge in which the cathode was insulated from the mounting plate by ceramic bushings (see Fig. 4.14). The anode ring was mounted on a central spark plug as in the a-c gauges. Since the location chosen for the Beta gauges was within the liner, it was not necessary that the parts of the gauge be mounted with vacuum sealed connections so that the ceramic bushings which insulated the cathode were adequate. The gauge projected into the liner proper through a hole in the central front portion, which forms a cavity for water line connections that enter there through the liner door. Two spark-plug insulators serve as bushings in the liner door by means of which electrical connections are made to the gauge unit.

The sensitivity of Beta Philips gauges has been determined on several occasions by comparison with 35T thermionic gauges using both static and dynamic calibration methods. Results of these tests indicate that the calibration factor is $2,000 \mu\text{a}$ per micron over the pressure range below $0.5 \mu\text{Hg}$ and $667 \mu\text{a}$ per micron in the pressure range between 2.5 and $0.5 \mu\text{Hg}$. The transition at $0.5 \mu\text{Hg}$ is not sharp and there is reason to believe that there is a continuously decreasing sensitivity above this pressure. Recent tests of Philips gauges in the plant indicate that the continuous decrease occurs and that it is caused by circuit parameters such as the large current-

Fig. 4.13—Circuit diagram of the d-c Philips gauge for the Beta plant.

Fig. 4.14 — Beta d-c Philips gauge.

limiting resistor and the loading of the circuit by the small bleeder resistor. However, the use of the factor $2,000 \mu\text{a}$ per micron has been found satisfactory for plant operation in four Beta tracks for over a year.

Since these gauges were mounted within the liner, it was necessary that they be disassembled and cleaned in the chemical-recovery area after each run. In the course of the cleaning process and in the mechanical-servicing area where the gauges were reassembled, checked, and installed in liners preparatory for a new process run, considerable damage to some of the parts and rehabilitation or replacement of faulty parts became relatively costly. For four Beta tracks these costs were estimated at \$30,000 per year for parts and \$16,000 per year for salaries, or \$46,000 per year for servicing exclusive of electrical service and parts for the power supply.

Electrical troubles involving the Beta Philips gauges in use in four tracks were surveyed over a period of four months. The most serious electrical trouble, amounting to about one-third of all the Philips-gauge electrical troubles reported for the survey period, was the failure by either grounding, short-circuiting, or breaking of the jumper cable and plugs that connect the liner-door junction box to the junction box on the front of the magnet coil. These failures are the more serious in that they generally require from 15 min to 1 hr of process outage for correction. As a consequence of a survey of the Philips gauge problems of the Beta plant, a new simplified gauge unit was designed and has been tested with very satisfactory results in the plant. A description and discussion of the new gauge is contained in a later section of this chapter. At the time of writing, the new gauge was being favorably considered for plant-wide installation to replace the present gauge units.

4. LABORATORY INSTRUMENTATION

In addition to the instruments used throughout the plant, there were many that found limited application in laboratories and shops only. In general they were commercially available and no development work was done on them here.

4.1 Cenco McLeod with Alterations. The McLeod gauges built by Central Scientific Company and modifications of them allowing pneumatic operation were used for some rough calibration work. They suffered from the usual difficulties of conventional McLeod gauges and were used only when necessary.

4.2 Thermocouple Gauges. Thermocouple-type gauges were used only to a limited extent since Pirani gauges were more readily available.

4.3 Thermionic-gauge Power Supplies. The most satisfactory power supply for triode ionization gauges in the laboratory has been found to be the unit designed by UCRL and shown schematically in Fig. 4.15. While several improvements—such as a built-in calibrating voltage—would be desirable, the unit functions well when used in conjunction with any one of several common triode ionization gauges. It is usual practice to adjust the emission current so that the sensitivity of the gauge is 10 μ a per micron in air.

4.4 Triode Ionization Gauge Tubes. For purposes of reference, the nominal operating values for several commercially available ion gauges are given in Table 4.3.

Table 4.3—Nominal Operating Values for Ionization Gauges

Type	Emission current, ma	Filament voltage, volts	Filament current, amp	Ion-collector voltage, volts	Electron-collector voltage, volts	Sensitivity, ^a μ a per μ Hg
35T ^b	3	6	4	-25	125	10
D-79510 ^c	1.17	4.5	1.6	-25	125	10
N.U.-R1038 ^d	20	3.0	1.8	-13	200	100
Televac 502 ^e	10	12.0	4.45	-35	310	280
VG-1A ^f	5	3.0-7.5	3.5-5	-25	150	120
VG-2 ^f	10	4-8	2.8-4.1	-25	150	32
WL-408 ^g	10	2.5-8.0	2-4.0	-20	130	62
NR-502 ^h	5			-20	150	50
NR-507 ^h	5	5	5	-20	150	100

^aSensitivity is given at the emission stated for air at 20°C.

^cWestern Electric Co.

^eGeorge E. Fredericks Co.

^gWestinghouse Electric & Manufacturing Co.

^bEitel-McCullough, Inc.

^dNational Union Radio Corp.

^fDistillation Products, Inc.

^hNational Research Corp.

4.5 Televac Recorder. The Televac Recorder, manufactured by the George E. Fredericks Company and sold by the Precision Scientific Company, automatically records pressure as a function of time from 0.004 μ Hg to atmospheric pressure on two manually selected scales. The output of a large triode ionization gauge operates a Micromax recorder directly for the range 0 to 0.4 μ Hg. A thermocouple gauge consisting of two reference junctions and two pressure-responsive junctions connected in series operates the same Micromax for the remainder of the pressure range. Protection from high pressure is provided for the ionization gauge, but the recorder must be manually switched from the high range to the low. The instrument has been used to record pump-down cycles and long rates of rise and to observe high-pressure breakdown and oscillation in vacuum-pumping systems.

Fig. 4.15—Ionization-gauge power supply used for experiments.

4.6 Alphatron. This is an ionization gauge with a radium source to cause ionization of the gas and is manufactured by National Research Corporation. The gauge has been found to be very rugged and reliable to about $\pm 2 \mu$ Hg. The three scales are 0 to 0.1 mm with 0.001 mm per scale division, 0 to 1.0 mm with 0.01 mm per scale division, and 0 to 10 mm with 0.1 mm per scale division.

4.7 Cold Cathode Gauge. The a-c Philips gauge, manufactured by Universal X-ray Corporation, is a glass-enclosed unit with an externally attached permanent magnet. The operating range is from 7×10^{-5} to 1.5×10^{-3} mm Hg. The gauge will apparently become covered with a semiopaque insulating film of unknown origin which appears in many electrical devices that run rather cool under a vacuum.

5. RESEARCH AND DEVELOPMENT

5.1 Philips Gauge. The Philips gauge was first described by Penning,⁴⁻⁵ while he was working at the Philips Lamp Works in Eindhoven, Holland. It was apparently introduced into this country late in 1943 and given much attention by UCRL⁸⁻¹³ during 1945, and also at CEW-TEC. W. R. Perret, CEW-TEC, did most of the calibration work and initial investigation here and the greater part of this section will be taken from his report,¹⁴ although some of the material contained in it was reported earlier by Nelson.¹⁵ Some additional work in the plant was done later on special applications of the gauge.¹⁶⁻¹⁸ Development work on measuring vapor pressure in the source is described in National Nuclear Energy Series, Division I, Volume 8.

The first Philips gauge on the area was built from information supplied by UCRL early in 1944, and is shown in Fig. 4.16. It was run experimentally in one of the XAX tanks, mounted in a rear-viewing window. The reliable range of the gauge was found to lie above 0.02μ Hg, a value below which unsteadiness and even extinction occurred. When used with an a-c supply, gauges of this type act as half-wave rectifiers so that a condenser filter across the meter is necessary as shown in the circuit diagram in Fig. 4.16.

Plans of a proposed gauge for the Alpha II plant were the basis of a new gauge shown in Fig. 4.17 to be run in XAX. The copper cathode plates, $2\frac{1}{2}$ in. square, were each $\frac{1}{4}$ in. from and parallel to the plane of an anode ring which had a 1-in. outside diameter and was formed of 0.050-in. copper wire. It must be emphasized that no fundamental work has been done on the Philips gauge relating the various parameters to characteristics of operation, and gauges were designed empirically since the exigencies of war demanded the quick adoption of a workable design. The data on this gauge gave widely scattered

Fig. 4.16—Earliest Philips gauge at CEW-TEC.

Fig. 4.17—Early experimental Philips gauge.

calibration points with an estimated sensitivity of about $500 \mu\text{a}$ per micron. A limited amount of experimental data in the plant obtained at about the same time showed general agreement with the XAX experience.

Laboratory experimental work on the Philips gauge was done on essentially the two models shown in Figs. 4.18 and 4.19. The glass-enclosed model was used in conjunction with a small electromagnet for work simulating Alpha plant conditions (3,400 gauss), whereas the thinner, metal unit was used if fields up to 8,400 gauss were needed. Both gauges used standard dimensions, i.e., $\frac{1}{2}$ -in. cathode spacing and a 1-in.-diameter anode ring of wire, since the usefulness of the existing design for plant use was to be tested. Calibration data were taken as a function of the various parameters involved for both a-c and d-c operation. The results will only be summarized here.

(a) A-c Operation. In general, low pressure at which extinction of a-c gauges occurs was shown to be somewhat too high for satisfactory plant application. Different combinations of molybdenum and copper for electrode materials indicated that no appreciable change in operation is apparent with different materials, but, of course, the sputtering rate of the cathode will depend upon the material of which it is constructed. Results of running the glass-enclosed unit at different magnetic fields indicated that the ion current at low pressures was proportional to the field strength, and that the over-all sensitivity was inversely proportional to the field in the range 600 to 6,700 gauss. Tests made with a 0.75-megohm resistor and a variable power supply showed that a 10 per cent increase in voltage results in a 15 per cent increase in sensitivity at least over the range of voltages tested (1,000 to 2,360 volts). When the voltage is low (1,000 to 1,500 volts), the extinction pressure is definitely increased over the values at higher voltages. The magnitude of the series resistor controls the range of linear response and ballasts the circuit to prevent runaway currents at higher pressures. During the conducting half-cycle of a Philips gauge, the current has the form of high-frequency oscillation superimposed on half the 60-cycle sine wave. The high-frequency variation in current will be discussed in more detail in connection with the d-c gauge.¹⁵ Electron emission from the anode ring during the nonconducting half-cycle apparently accounts for 20 to $30 \mu\text{a}$ of current that may occur during this period, causing the gauge to read low by that amount.

(b) D-c Philips Gauge Studies. Figures 4.20 and 4.21 show the important operating characteristics of the experimental unit of Fig. 4.18 when run with simple half-wave condenser-filtered d-c power supply.

The extinction pressure was less than $0.003 \mu \text{ Hg}$, well below the pressure reached in the operating tanks.

The oscillatory nature of the current to the gauge was investigated by means of an oscilloscope connected across a 1,000-ohm resistor in series with the power supply between the meter and ground. The wave form was roughly saw-toothed with an additional sharp current peak at the end just before the value dropped to near zero. The frequency of the oscillation was independent of the magnetic field over a

Table 4.4 — Pressure-Frequency Characteristics
of the Philips Gauge

Pressure, $\mu \text{ Hg}$	Gauge current, d-c $\mu \text{ a}$	Frequency, kilocycle/sec
0.002	2	2.2
0.0025	2	2.5
0.003	3	3.1
0.005	5	5.0
0.006	6	6.0
0.02	38	17.0
0.002	42	15.0
0.03	54	22.5
0.08	200	100.0

6 to 1 range but some phase shift resulted at certain field strengths. The oscillation seemed independent of the external-circuit parameters but varied with the pressure as shown in Table 4.4 which gives the relation between pressure, current, and frequency measured by means of the oscilloscope sweep frequency.

No comprehensive study of the Philips gauge discharge was attempted, the work reported here having been done to test models of plant gauges, but certain facts observed during the course of these tests may help to indicate the nature of the discharge. The existence of a plasma within the gauge volume and the sawtooth-plus-pulse current pattern have been tentatively explained as attributable to a type of pumping action¹⁹ in which gradual build-up of the volume of the plasma increases the potential gradients from it to the electrodes with consequent increased drain to them. These gradients eventually become so steep when the plasma virtually fills the whole volume that it is drained suddenly, and the discharge is momentarily extinguished. The frequency with which this cycle can be performed depends, at least in part, upon the molecular density and the nature of the gas; therefore, the frequency increases with pressure. This explanation is admittedly

Fig. 4.18—Experimental gauge for use at Alpha field strengths.

Fig. 4.19—Experimental gauge for use at Beta field strengths.

Fig. 4.20—Effect of series resistor (R_s) on sensitivity of Philips gauge shown in Fig. 4.18. $H = 3,400$ gauss. Supply voltage = 2,500 volts direct current. Pressure measured on a 35T triode ionization gauge.

very crude, but for the present appears to fit most of the facts established concerning the nature of Philips gauge operation.

Figure 4.22 is an exploded view of the preliminary design for the unit proposed to replace the Beta d-c gauge. It should be noted that no vacuum seals are involved since the entire assembly is mounted on the liner inside the tank, and jumpers and spark plugs through the

door provide the electrical connections. The electrodes serve either as the anode or cathode, depending upon their polarity. They are mounted on an inexpensive ceramic plate and all the shields, baffles, and bolts are simple and economical to replace. The baffling is so effective that chemical recovery from the electrodes and ceramic plate is unnecessary, thus extending the life of the gauge.

5.2 Race and Drain Gauges.²⁰ Both these gauges are special arrangements of electrodes providing the conditions necessary for a

Fig. 4.21—Effect of d-c supply voltage (V_s) on the sensitivity of the Philips gauge shown in Fig. 4.18. $H = 3,400$ gauss. Series resistor = 0.25 megohms. Pressure measured on a 35T triode ionization gauge.

Fig. 4.22—Exploded view of a proposed unpolarized Philips gauge for the plant. The entire unit shown is to be mounted inside the tank of the liner.

discharge between the cold electrodes in a magnetic field. They have been used in studies of such discharge and to measure pressure in experimental equipment.

Fig. 4.23—Flow diagram of the vacuum divider. The valves can be used to adjust the intake and exhaust conductances.

5.3 Vacuum Divider Gauge.²¹ The vacuum divider is an arrangement that allows pressure in the ionization-gauge range to be read on more rugged instruments such as Pirani, thermocouple, or McLeod gauges. Figure 4.23 illustrates the essential features. Both the intake

and exhaust of a small diffusion pump (about 5 liters/sec) are connected to the vacuum system to be checked, and a gauge is provided to read the fore pressure of the pump. Assuming molecular flow to exist, the fore pressure p_f is

$$p_f = p \frac{S_i}{S_e} \mu \text{ Hg} \quad (1)$$

where p is the tank pressure, S_i is the intake pumping speed and S_e is the exhaust speed from the gauge back into the main system. By adjusting the ratio of S_i to S_e the instrument can be used to amplify the tank pressure for easy reading. Factors limiting the amplification are (1) the speed of response is determined by the ratio of the volume of the fore-vacuum region to the exhaust speed and (2) a very small opening for the exhaust will change in size with time because of oil vapor and other contaminants. With a 5 liters/sec pump, the amplification can easily be made 40, and a value of 75 has been obtained with more difficulty. Larger pumps will make higher amplification practical, of course. Figure 4.24 shows typical calibrations using a Stokes McLeod and a thermocouple gauge. In the case of the McLeod, the pressure amplification is about 50 at $0.1 \mu \text{ Hg}$, and the range extends well up into the pressure covered by the McLeod directly. Figure 4.25 is a calibration at low pressure using ionization gauges in an attempt to verify the point (0,0) as being on the curve. It is interesting to note that the calibration curve would be linear even though the pressure to be measured is below the base pressure of the vacuum-divider diffusion pump when operated conventionally. This would tend to verify the theory that the base pressure of a pump is the pressure at which its actual pumping speed equals its intrinsic leakage rate, so that at pressures below this base it still causes gas to flow from the high-vacuum into the low-vacuum region.

Of course, any vacuum-pumping system with diffusion pumps can be considered as a nonlinear vacuum divider since the fore pressure can be shown to be a function of the high-vacuum pressure.

5.4 Modified Knudsen Gauge.²² Experimental work was done by G. W. Monk in 1945 on a Knudsen type gauge, illustrated in Fig. 4.26. The work was done only to establish the soundness of the principle involved, and no time was available to carry the development work to completion. The cold vanes of 0.001-in. aluminum were carried on the ends of a beam fixed to the coil of a meter movement with jewel bearings. The hot plates were grounded copper shields over nichrome heaters, mounted so as to cause the movable vanes to rotate about

the coil axis. A small glass mirror mounted rigidly on the beam indicates a change in the position of the beam by changing the amount of light striking two photocells. The difference in output of the two cells is amplified and applied to the meter movement in such a direction as to restore the beam to its original position. The current required through the coil to overcome the repulsive force of the hot and

Fig. 4.24—Typical calibration curves of a vacuum divider for a Stokes McLeod and a Televac recording thermocouple-gauge indicator. The fore-vacuum conductance was different in the two cases.

cold plates is thus proportional to this force and hence proportional to the pressure, according to theory developed by Knudsen.²³

The limited amount of work done with this gauge established its practicability, and while the poorly adjusted time constants in the arrangement tended to result in oscillations, changes in pressure of $0.005 \mu \text{ Hg}$ were easily observed. The temperature of the hot plates was read by means of a permanently attached thermocouple. The temperature of the cold vanes was estimated from measurements made under static conditions with a temporarily attached thermocouple.

5.5 Mean-free-path Gauge.²⁴ The elementary theory of the triode ionization gauge gives the relation between the molecular density, N , the electron current, i_e , and the ion current, i_i , as

$$i_i = k i_e N \quad (2)$$

where k is a constant depending upon the nature of the gas and the geometrical form. In the ordinary application, i_e is held constant by

Fig. 4.25—Vacuum divider calibration using ionization gauge (D-79510) to measure both pressures.

regulating the filament current so that i_i is proportional to the molecular density and, hence, to the pressure at a given temperature. At any temperature, however, the molecular density is inversely proportional to the mean free path, L , of the gas so that

$$i_e = K' i_i L \quad (3)$$

In the mean-free-path gauge, i_i is held constant by regulating the filament current so that i_e is proportional to the mean free path. No amplifier is required to read i_e so that zero drift and calibration

Fig. 4.26—Modified Knudsen gauge illustrating the degenerative arrangement used to prevent vane motion.

Fig. 4.27—Circuit diagram of the mean-free-path gauge.

problems are not encountered. The circuit shown in Fig. 4.27 is a modification of the conventional circuit of Fig. 4.16, which allows the mean free path of a gas to be read directly from 0.5 to 5,000 cm. The circuit contains a d-c amplifier to allow the ion current to be observed continuously, but this was added only for experimental reasons. An apparent advantage of the mean-free-path gauge is that at high pressure the filament temperature is automatically lowered, but this advantage does not increase tube life in practice, partly because at low pressures the filament temperature is automatically increased. The circuit shown in Fig. 4.27 proved very satisfactory in practice although there was some unsteadiness above about 2,000 cm.

5.6 Viscosity Gauge and Magnetic-ionization Gauge.²⁵ The need for a gauge to measure charge-vapor pressure (about 3 μ Hg) in the process units provoked preliminary work on a vibrating quartz-fiber viscosity gauge and a special low-voltage ionization gauge to be immersed in the magnetic field. Work on the ionization gauge was abandoned because of the rapid formation of insulating film on gauge parts owing to nearby discharges and the critical alignment required.

The development of the viscosity gauge was not carried to completion, but it apparently could be made a useful instrument in spite of the fact that nonvisual determination of the damping was difficult owing to harmonic vibrations of the fiber.

5.7 Automatic Pump-down Equipment.²⁶ In the early days of plant operations the complexity of the vacuum system coupled with the inexperience of the operators resulted in many mistakes in the manipulation of the various valves, which resulted in loss of production on one or more of the process tanks interconnected by the roughing and finishing headers. At one time this one factor alone caused as high as three header failures per day in the Alpha I buildings.

It became obvious that a system of motor-operated valves provided with the necessary interlocks would be desirable if economically justifiable. Accordingly, Limitorque operators for the 6-in. Chapman valves and main gate valves were purchased from the Philadelphia Gear Works for an experimental installation in XBX. While designing the circuit to operate these valves, it was decided to provide, in addition to interlocks, a complete sequencing circuit for the roughing-down operation. The completed circuit used signals obtained from the Pirani gauge and limit switches on the Limitorque operators to drive relays, which initiated the various operations in the sequence, as outlined in Fig. 4.28.

A circuit incorporating the above design was built and installed in XBX and was found to operate satisfactorily. Owing to the wartime scarcity of the materials used, it was necessary to use substitute

components, which in some cases were not suited for the particular application. However, sufficient data were obtained to indicate that with proper adjustment of correct equipment the circuit would function in

Fig. 4.28—Automatic pump-down flow diagram and sequence layout.

exactly the manner predicted and, further, that failures of mechanical and electrical components did not have any disastrous results either to the circuit or to the vacuum-pumping system.

No further design or development work was done on the equipment since the level of training of vacuum operators in the plant had risen to a point where header failures due to mistakes was an insignificant cause of outage.

5.8 Automatic Rate-of-rise Indicators.²⁶ Automatic rate-of-rise indicators were developed in an effort to eliminate the possibility of errors by the operators when calculating a rate of rise. Two different circuits were used experimentally, one of which was given a limited plant trial.

Both these circuits depend upon the fact that if a voltage whose rate of change is proportional to the rate of rise of pressure is applied to a series combination of a condenser and a resistor, the current that results will also be proportional to the rate of rise after a time that is long compared to the resistance-capacity product of the series circuit. In practice, the voltage developed across the plate resistor of an ionization gauge is applied to a series combination of a resistor and a condenser so that the voltage across this resistor is proportional to the rate of change of pressure after a second or less.

Unfortunately, the current delivered by the ion gauge is so small that amplification is necessary. The circuit in Fig. 4.29 is a modification of a standard ionization-gauge supply, which allows the signal on the left side of the vacuum-tube voltmeter circuit to be removed and the grid grounded through a 1-megohm resistor. The right-hand grid at the same time is removed from ground and connected across the 100,000-ohm resistor, which is in series with the 4- μ f condenser across the 6SJ7. The signal taken from the ion-tube plate is fed to the grid of the 6SJ7 relay driver as before. Then, when the pressure in the ion tube, and thus the ion current, begins to increase, an increasing positive signal is placed on the grid of the 6SJ7, which causes a negative swing of the plate.

If the voltage change across the resistance-capacity circuit is steady, in a very short time a steady negative voltage appears across the 100,000-ohm resistor and the right-hand grid of the 6SN7. A negative signal on this grid then produces a positive or up-scale reading on the meter M. In the modified circuit described above, the 100,000-ohm resistance was adjusted to give 0.01 μ Hg per second at full scale on the $\times 0.1$ scale. The time constant of the system is a maximum of 0.4 sec so that essentially a constant reading is reached in 1 sec.

With this simple modification of the circuit, it was merely necessary for the operator to close the pump-head valve, flip the switch from pressure to rate of rise, wait for a second or two, and read directly the rate of rise in the vacuum system. Several of these circuits were built and used in the first Beta building, where they gave

study of the constancy of pressure in the vacuum system under various conditions and the variation in the rate of pressure change during rate-of-rise measurements.

Fig. 4.30—Improved rate-of-rise indicator circuit.

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Chapter 5

VACUUM TESTING AND LEAK DETECTION

By G. W. Monk and C. E. Normand

1. INTRODUCTION

This chapter describes methods of testing equipment for the existence of leaks and techniques and instruments used to locate any leaks.

Since producing and maintaining a uniform magnetic field of 3,000 to 7,000 gauss over a large area is complicated and expensive, it is essential that as little time as possible be spent in reducing the pressure in the tanks to operating values. In the Beta plant, where the operating pressure is about 0.015μ Hg and the pumping speed is assumed to be about 7,000 liters/sec, the total leakage rate during operation is roughly 100μ -liters/sec or about 0.13 cc/sec at atmospheric pressure. This is the virtual leakage rate during operation with the high voltage and heaters on and includes large quantities of gases released owing to the high temperature and ion bombardment of the equipment. One estimate would place the actual leakage rate at about 0.013 cc/sec or one-tenth of the virtual rate.

The problem is to detect and locate any leaks larger than about 0.01 cc/sec or smaller leaks totaling greater than 0.01 cc/sec on surfaces of 179, 461, and 652 sq ft in Beta, Alpha I, and Alpha II. (These figures assume the source and receiver units to be flat plates and so do not adequately convey the extreme degree of complexity of the surfaces to be tested.) In practice most of the leak hunting is limited to the equipment that is removed and replaced at intervals, since the tank, manifold, and pumps remain tight for long periods, barring accidents.

In order to ensure tight systems and low operating pressures in a reasonable pump-down time, both vacuum-testing and leak-detecting methods were used and developed. The former included methods of checking the leakage rate of a system after evacuation had started, while the latter included all methods necessary to determine the existence of

a leak and its exact location. Of course, many techniques and instruments used in the plant are standard in any high-vacuum work and, consequently, receive only limited treatment here except in so far as special problems or variations were found necessary. Other instruments and techniques were developed wholly or in a large part in the plant and associated laboratories to fill special needs of the operations. They are described in more detail.

The nature of gaseous flow through small holes across which a large pressure exists may be investigated quantitatively.¹ Loeb² gives Knudsen's equation for the volume (measured at unit pressure) rate flow, per unit pressure difference, of gas in a cylindrical tube across which there exists a small pressure difference.

$$Q' = \frac{\pi}{8} \frac{v}{\eta} \frac{R^4}{L} p + \frac{4}{3} \sqrt{\frac{2\pi}{\rho_1}} \frac{R^3}{L} \left[\frac{1 + 2.00 (\sqrt{\rho_1}/\eta) R p}{1 + 2.47 (\sqrt{\rho_1}/\eta) R p} \right] \frac{\text{cc bar}}{\text{sec bar}} \quad (1)$$

where 1 bar is 1 dyne/sq cm; η is the viscosity in poises; R is the radius of tube in centimeters; L is the length of tube in centimeters; p is the average pressure in dynes/sq cm (bars); ρ_1 stands for M/R_0T ; M is the molecular weight of the gas; T is the absolute temperature; and R_0 is the gas constant per mole.

Of course, Q' in Eq. 1 (multiplied by the pressure difference) is a mass rate of flow in cc bar/sec for a particular gas and, since it is inversely proportional to the length, Q will hereafter be understood to be the mass rate of flow in cc bar/sec for a leak 1 cm long. Thus, after making obvious substitutions, the mass-flow rate for a difference in pressure, dp , is

$$dQ = ap \, dp + b \left(\frac{1 + C_1 p}{1 + C_2 p} \right) dp \quad (2)$$

This is a general equation which is applicable to any cylindrical tube providing it is integrated over the total pressure difference and the impedance of the end can be neglected. Integrating Eq. 2 between limits p_1 and p_2 to obtain the flow for a difference in pressure of $(p_1 - p_2)$ gives

$$Q = \frac{a}{2} (p_1^2 - p_2^2) + b \frac{C_1}{C_2} (p_1 - p_2) + \frac{b}{C_2} \left(1 - \frac{C_1}{C_2} \right) \ln \left(\frac{1 + C_2 p_1}{1 + C_2 p_2} \right) \quad (3)$$

The first term is Poiseuille's law and depends only upon the viscosity of the gas, while the other two terms result from the integration of Knudsen's law for molecular flow. The second term is proportional to

the square root of the molecular weight, while the third involves both the viscosity and molecular weight.

To apply the equation to leaks into vacuum systems, p_1 must be taken at atmospheric pressure and p_2 as the exhaust pressure on the vacuum side. If it is assumed that the gas cannot enter the vacuum system at greater than sonic velocities, the pressure at the exhaust end of the hole cannot be less than

$$p_2 = \frac{Q}{\pi R^2 v} \text{ dynes/sq cm} \quad (4)$$

where v is the velocity of sound in the gas. If the pressure in the system is lower, the additional drop must take place after the stream has left the leak. If higher, p_2 must be taken as the system pressure. It will be shown later that the minimum pressure as given by Eq. 4 is negligible compared to atmospheric for small leaks. For larger leaks Poiseuille's law can be solved for Q directly.

Consequently, since $C_2 \ll 1$, for $R < 10^{-2}$, Eq. 3 becomes

$$Q = \frac{a}{2} p_1^2 + b \frac{C_1}{C_2} p_1 + \frac{b}{C_2} \left(1 - \frac{C_1}{C_2} \right) \ln (1 + C_2 p_1) \quad (5)$$

Equation 5 holds where the exhaust pressure is negligible compared to the high pressure and, if it is not, the flow is predominantly viscous so that the second and third terms in Eq. 3 can be dropped and the value of p_2 from Eq. 4 can be substituted in the first term, giving

$$Q = \frac{a}{2} \left(p_1^2 - \frac{Q^2}{\pi^2 R^4 v^2} \right) \quad (6)$$

assuming, of course, that the system pressure is lower than the limiting pressure as given in Eq. 4. Equation 6 yields

$$Q = \frac{\pi^2 R^4 v^2}{a} \left(\sqrt{1 + \frac{a^2 p_1^2}{\pi^2 R^4 v^2}} - 1 \right) \quad (7)$$

and substituting the value for a ,

$$Q = \pi v^2 8 \eta \left(\sqrt{1 + \frac{R^4 p_1^2}{8 \eta^2 v^2}} - 1 \right) \quad (8)$$

If the square root of the fraction under the radical is large compared to unity, as it would be for $R > 5 \times 10^{-2}$, then

$$Q = \pi R^2 v p \quad (9)$$

For purposes of numerical calculations the following values of the constants are given for air, hydrogen, and helium at 23°C when the pressure is given in bars and Q in cc bar/sec.

Table 5.1 — Constants for Air, Hydrogen, and Helium

Constant	Air	Hydrogen	Helium
M	29	2	4
η	182×10^{-6}	88.2×10^{-6}	196.2×10^{-6}
a/R^4	2.2×10^3	4.4×10^3	2.0×10^3
b/R^3	1.0×10^5	3.7×10^6	2.6×10^5
C_2/R	0.45	0.25	0.16
C_1/C_2	0.81	0.81	0.81
v	3.4×10^4	13×10^4	8.3×10^4

Table 5.2 — Leakage Rates

Capillary radius, cm	Leak rate, micron-liters per sec			Limiting pressure for air
	Air	H ₂	He	
10^{-2}	5.0×10^3	13×10^3	7.2×10^3	47 cm Hg
10^{-3}	0.87	3.9	0.91	7.1 mm Hg
10^{-4}	0.14×10^{-3}	2.5×10^{-3}	0.24×10^{-3}	130 μ Hg
10^{-5}	0.075×10^{-6}	2.5×10^{-6}	0.22×10^{-6}	7.0 μ Hg

An exact calculation of Q for the general case would require solution of the transcendental equation resulting when p_2 from Eq. 4 is substituted in Eq. 3. Equations 5 and 8 are approximations to the solution and are applicable, respectively, when p_2 is and is not negligible. In calculating, p_2 is first assumed negligible and Q is calculated from Eq. 5; if p_2 turns out consequently to be appreciable, then Q must be recalculated from Eq. 8. As a numerical example of the magnitude of leakage rates, Table 5.2 has been calculated for leaks 1 cm long, and a pressure of 10^6 bars. Values for $R > 10^{-3}$ cm were calculated from Eq. 8, the others from Eq. 5.

2. VACUUM TESTING METHODS

After evacuation of a system has started, there are two ways to check the leakage rate without using auxiliary equipment: (1) observation of the rate of decrease of pressure due to a known rate of pumping, and (2) measurement of the rate of rise of pressure in the system when isolated from the pumps. Both methods require a knowledge of the pumping speed, volume of the system, and pressure.

2.1 Rate of Pump-down. If it is assumed that the system has a volume of V liters and a total constant leakage rate (virtual plus actual) of L μ -liters/sec, and is being evacuated at a constant volumetric speed of S liters/sec, the differential equation governing the pressure p in the system at a time t is

$$V dp = (L - Sp) dt \quad (10)$$

This equation fails at both very high and very low pressures, where the assumptions made above fail to represent the actual case. When pumping down a system, the equation must be applied twice with two different sets of values for L and S , first for the roughing operation and again when the diffusion pumps are opened. From Eq. 10 the instantaneous rate of change of pressure is

$$\frac{dp}{dt} = \frac{(L - Sp)}{V} \quad (11)$$

For this rate to vary appreciably with the size of the leak, L must not be insignificant compared to Sp . Since the plant uses fast-pumping systems with S about 8,000 liters/sec, either the leak must be very large or the system must be pumped an inconveniently long time in order to detect the leak by observation of the instantaneous rate of change of pressure. For example, to detect by this method a total leakage rate of 0.1 cc/sec, at atmospheric pressure, the system must be pumped down to a pressure of 0.092 μ Hg before L becomes 0.1 of Sp (using the diffusion pumps).

The solution of Eq. 1, when p_0 is the pressure at $t = 0$ and p is the pressure at time t , is

$$t = -\frac{V}{S} \ln \left(\frac{Sp - L}{Sp_0 - L} \right) \quad (12)$$

In all practical cases, L is small with respect to Sp_0 and can be neglected, giving

$$t = -\frac{V}{S} \ln \left(\frac{Sp - L}{Sp_0} \right) \quad (13)$$

The equation shows that for the time required to reach a certain pressure to be a significant function of the leakage rate, this leakage rate must not be negligible compared to Sp . Since S is very large, either p must be very small or L very large.

Equations 2 and 4 show that observation of neither the instantaneous rate of change of pressure nor the time required to reach a given pressure will provide evidence of the leakage rate unless the base pressure is approached. Measurement of the base pressure itself will allow the leakage rate to be determined if the pumping speed is known, since for t in Eq. 13 to be infinite Sp must equal L , and L is then proportional to the base pressure.

While the base pressure is the final criterion of the necessary tightness of the system, units or lines are seldom pumped down to their base pressures purely for test purposes. Early dry-docking procedure, however, included this technique. Observation of the rate of pump-down plays no significant part in the testing methods used in the plant, even though the time constant, V/S , is less than 0.5 sec in some cases, making the evacuation time very small.

2.2 Rate of Rise. If the pumps are closed off from a system after it has been evacuated to some pressure p , the rate of increase of pressure is given by setting S equal to zero in Eq. 10, giving

$$\frac{dp}{dt} = \frac{L}{V} \mu \text{ Hg/sec} \quad (14)$$

The leakage rate is given then by the product of the measured rate of rise and the known volume if a negligible fraction of L is in the pumps. This value of L will depend upon the time the system has been pumped and the temperature of the system, since the amount of outgassing is a function of the time and temperature. Another factor affecting the value of L is that any cold trap left in the system will continue to act as a pump for vapors condensable at the temperature of the trap. In the Beta plant a liquid-nitrogen trap of 565 sq in. probably provides a pumping speed of the order of magnitude of 20,000 liters/sec for many vapors; in the Alpha plant, the corresponding figure is about 10,000 liters/sec.

In the plant, a maximum allowable rate of rise is determined mostly by experience, and bins must be tight enough to pass this test before

operation begins. Before 1945, when the use of helium leak detectors became standard practice on all dry docks, units were given a rate-of-rise test in the dock after being pumped down. After the pretested units are placed in the bins, the entire system must pass another rate-of-rise test before the bin is turned over to the operating crews, although recent successful operation shows that the last rate of rise is unnecessary. The maximum allowable rate of rise in the Alpha plant is $0.003 \mu \text{ Hg/sec}$, while in the Beta plant the figure is $0.004 \mu \text{ Hg/sec}$. Standard practice is to take the rate of rise on an ionization (triode) gauge or Philips gauge by means of a stop watch according to certain specified rules, but several instruments have been developed to measure the rate of change of pressure automatically or semiautomatically and have been described in Chap. 4, Sec. 5.8.

3. COARSE METHODS OF LEAK DETECTION

The leak-detecting methods used in the plant can be conveniently classified into types according to the minimum size of the leak that can be detected. The mass spectrograph used with helium is the most sensitive and is the only satisfactory instrument for locating minute leaks. Other methods that can be used for large leaks will be the subject of this section.

3.1 Pressure Testings. This method of testing consists of filling the system with a gas (usually air) under as high pressure as is safely and easily attainable, and detecting the leakage by immersing the whole system in water or covering the suspected parts with a soap solution.

Neglecting for the moment the method of detecting the escape of air, the gauge pressure can be calculated which will give the same leakage rate as existed when the system was evacuated. When Poiseuille's law is solved for the gauge pressure that will give a leakage rate of $Q \text{ cc-bar/sec}$, with p_1 in bars, the result is

$$p_3 = 14.7 \left(\sqrt{1 + 2Q/ap_1^2} - 1 \right) \text{ lb/sq in.} \quad (15)$$

Using the values of Q for air from Table 5.2, Table 5.3 has been calculated giving the required gauge pressure for capillaries of different radii and for different gases.

Table 5.3 shows that there is a great practical advantage in testing with hydrogen under high pressure when possible. Safety considerations eliminate hydrogen for many testing purposes and also limit the magnitude of the pressure that may be used.

In considering the sensitivity of leak hunting by this method, factors to be considered are (1) the maximum pressure and type of gas to be used, (2) the type of liquid and its surface tension since it will tend to plug small holes by capillary action, and (3) the minimum bubble rate that can be readily detected.

Table 5.3 — Gauge Pressure Required for Leakage Rates

Radius, cm	Leakage rate under vacuum for air, μ -liters/sec	Gauge pressure required for same leakage rate under pressure, lb/sq in.		
		Air	H ₂	He
10^{-2}	5.0×10^3	3.2	2.0	3.5
10^{-3}	0.87	6.5	3.4	6.7
10^{-4}	0.14×10^{-3}	9.8	5.3	10
10^{-5}	0.075×10^{-6}	30	20	34

For the third factor, it will be arbitrarily assumed that 1 bubble per min of 1 mm diameter is the minimum bubble rate that can be detected. This corresponds to a leakage rate of about $6.62 \times 10^{-3} \mu$ -liters/sec since the gas in the bubble is essentially at atmospheric pressure. It will also be assumed that 6.00 lb/sq in. of air is to be used in the system. This is approximately the safe limit for the tanks, but small systems can be put under as much as 100 to 200 lb/sq in. of hydrogen. The minimum detectable leakage rate assumed above will occur through a leak of about 3×10^{-4} cm radius.

A bubble will start forming at the end of the capillary when it is covered with liquid if

$$p > \frac{2T}{R} \quad (16)$$

where p is the air pressure in the capillary in dynes/sq cm (bars); T is the surface tension of the liquid in dynes/cm; and R is the radius of the capillary in centimeters. Table 5.4 has been calculated for water at 23°C. Soap solutions would have a surface tension about one-third that of water, and the corresponding critical pressure for soap solution would be one-third that given for water. For the minimum detectable leak estimated above, the critical pressure is about 7.1 lb/sq in. Since the tank pressure is only 6 lb/sq in., bubble formation would not take place unless soap or some other wetting agents were added. There are no experimental data on the minimum detectable leak by this method

since the results depend mostly on the technique and skill of the operator. An estimate would place the order of magnitude at about 0.05μ -liters/sec for routine work on the bins. For small systems, which can be put under large pressures of hydrogen and immersed, the figure is much smaller.

Table 5.4—Pressure Equivalent of Capillary Action

Capillary radius, cm	10^{-2}	10^{-3}	10^{-4}	10^{-5}
Pressure, lb/sq in.	0.21	2.1	21	210

The foregoing discussion concerning bubble formation applies when the apparatus is submerged in a liquid. If a soap solution is applied in a thin layer with a brush, twice the pressure given by Eq. 16 will be necessary since the bubble will be of the two-surface type. In this case the advantage of low surface tension, resulting from the application of soap, is partly negated by the presence of a double surface bubble.

The calculations serve to illustrate the application of the equations; however, each special problem must be analyzed individually.

3.2 Pump-outs and Sight Glasses. All gasketed joints and Wilson seals in the original equipment installed in the plant had double seals with so-called "pump-outs" connecting the space between the two gaskets to the atmosphere. This pump-out provided a means of testing the inner gasket for leaks by application of pressure or vacuum to the space and observation of any change in pressure in the system. If the inner gasket leaked, it was possible to plug the pump-out or continually evacuate it. Another method of testing the gasket consisted of covering the pump-out with a thin film of soap and observing if the film is pulled in.

On gasketed joints that were seldom opened sight glasses were originally provided. That is, the intergasket space was filled with low-vapor-pressure oil communicating with a column of the oil visible in a small sight glass. Any leakage by the inner gasket would now show as a decrease in the column height, and only low-vapor-pressure oil would enter the system. All sight glasses have now been discarded after being found impractical for several reasons. It is almost impossible to fill the intergasket space without trapping pockets of air, which expand and contract with variations in the room temperature and give false indications of leakage. More important, however, is the fact that once a gasket joint is made tight it very seldom begins to leak at some later date.

Plant experience seems to indicate that double gaskets and pump-outs are easily stopped up with soap, and application of vacuum or pressure causes little change in pressure. However, a single gasketed

joint is easily made reliable with only moderate care if proper materials and surfaces are available.

The main vacuum-system welds were all made double by the manufacturer to facilitate pretesting. Joints were welded from the inside and outside so that two separate beads existed, and the space between could be put under high gas pressure and the welds could be soap-tested.

3.3 Pressure and Vacuum in Water Lines. After the tank or dry dock had been evacuated, the water lines could be tested by varying the pressure in the lines and observing any changes of pressure within the tank. The method relied upon the fact that the leakage rate is dependent upon the pressure difference across the leak. The results of this test must be interpreted with caution since it was very difficult to dry the lines both inside and out. Any water remaining at the leak could cause it to freeze shut (owing to cooling by evaporation under vacuum), block the line so that application of pressure or vacuum would not appreciably affect the rate of evaporation of water into the system, or plug the leak by capillary action.

3.4 Leak Hunting at Reduced Pressure inside the Vacuum System.³ This method is applicable only to large tanks, and it found only limited use in the plant. The operator works inside the tank with necessary lights and soap solution while the pressure is held to about 6 in. Hg below atmospheric. Standard soap-bubble technique is used to locate leakage from the outside to the interior. It is desirable to use a very fast pump so that a relatively large leak can be admitted for the comfort of the operator. Practically no discomfort is felt unless the pressure is changed rapidly.

3.5 Use of Gases with Pressure Gauges.¹ The response of thermionic, Pirani, and Philips gauges depends upon the characteristics of the gas as well as the absolute pressure and temperature. If the vicinity of a leak is flooded with a foreign gas, then part of the leakage will be due to this new gas and, if its characteristics are different from the gas already present, a change will occur in the pressure-gauge reading. This replacement method is most effective on a single leak so that a large fraction of the gas in the system will eventually be replaced by the new gas.

The sensitivity of this method is determined by the difference in response of the gauge to the new and the old gases and the length of time the leak is flooded. If the gauge indicates $k_1 p_1$ in air and $k_2 p_1$ in the foreign gas at the same pressure, and all the air in the system is replaced by the foreign gas, the reading will change from $k_1 p_1$ to $k_2 p_2$. In any practical gauge there is a minimum deflection change, Δd , which

can be relied upon as definite because of variations that are due to other causes than changes in the nature of the gas. Assuming a linear gauge, the minimum mixture ratio in which the foreign gas can be detected by setting the change in deflection equal to this minimum is

$$\Delta d = k_1 (p_a + p_g) - (k_1 p_a + k_2 p_g) \quad (17)$$

if p_a and p_g are the partial pressures of the air and the gas, respectively, after equilibrium has been reached. From Eq. 17 the ratio of air to helium in the leanest mixture in which the foreign gas can be detected is

$$M = \frac{p_a}{p_g} = \frac{(k_1 - k_2)p_a}{\Delta d} \quad (18)$$

which shows that the sensitivity increases with the difference in the calibration constants for air and the gas, the operating pressure and stability of the gauge. Equation 18 shows only the maximum mixture ratio that can be detected in a closed system, and does not entirely specify the sensitivity of this leak-hunting method, since several other factors need be considered.

If the rate of diffusion through a leak or the pumping speed of the system is different for air and the foreign gas, the partial pressure due to the gas will be different from the partial pressure originally due to the air leaking through the hole. Let

$$L_r = \frac{L_g}{L_a} \quad (19)$$

and

$$S_r = \frac{S_g}{S_a} \quad (20)$$

where S_g and L_g , S_a and L_a are the pumping speeds and leakage rates of the gas and air, respectively. Obviously, then, any partial pressure originally due to air flowing through a leak will be increased in the ratio L_r/S_r when the leak is flooded with the foreign gas. Modifying Eq. 17 correspondingly, an expression is found for a more accurate leakage ratio that can be detected after equilibrium.

$$M = \left(k_1 - k_2 \frac{L_r}{S_r} \frac{p_a}{\Delta d} \right) \quad (21)$$

or

$$M = \left(1 - \frac{k_2 L_r}{k_1 S_r}\right) \frac{k_1 p_a}{\Delta d} \quad (22)$$

In addition to the things shown by Eq. 18, Eq. 21 shows that a difference in calibration constants can be at least partially negated by unfavorable leakage rate or pumping speed. The pumping speed is the effective speed considering all cold traps and location of the leak and gauge. The leakage will depend upon the molecular weight, the viscosity, and the size of the opening. All these factors, except the variation in pumping, have been confirmed experimentally. At time of writing no reliable data were available concerning the speed of diffusion pumps for different gases.

According to Eq. 21, the sensitivity is proportional to the operating pressure, but an upper limit is set by certain practical considerations. First, the pressure can be raised by one of two methods, letting in an additional leak or throttling the pump. The first method increases the mixture ratio in proportion to the pressure so that nothing is gained, while throttling the pump may increase the response time since the interdiffusion rate is inversely proportional to the pressure at high pressures. The time constant is V/S if V is the volume of the tank and S the speed of the pump, and this ratio, as well as the interdiffusion rate, determines the response and cleanup time when leak hunting. If the pressure increases so as to require a higher scale on the gauge, a greater change in pressure is needed for detection. Experience has shown that it is practical to throttle the pump until the pressure is near the top of the lowest possible scale.

When the pumps are throttled and the pressure is sufficiently low for molecular flow, S_r in Eq. 20 can be approximated by calculating the relative diffusion rate of the gas through an orifice or pipe. That is,

$$S_r = \sqrt{M_a/M_g} \quad (23)$$

where M_a and M_g are the molecular weights of air and the gas, respectively.

Several considerations determine the selection of the gas used for leak hunting:⁴

1. Toxic or anesthetic properties.
2. Odor.
3. Inflammability.
4. Expense.
5. Calibration constant relative to that of air for the gauge to be used.

No reliable data are available on the calibration constant of different gauges for different gases.

6. Molecular weight (diffusion properties).
7. Condensability at the temperature of any cold traps in the system.
8. Leakage rate through small holes between large pressure differences. See introduction to this chapter.

In practice helium is the most desirable with thermionic or Philips ionization gauges, whereas propane gives much better response with an untrapped Pirani gauge. Propane, however, has an obnoxious odor and is inflammable.

Using Eq. 23, the speed of the throttled pumps is seen to be about 2.5 times faster for helium than for air, and it has been observed that helium may flow through a small leak about 1.2 times as fast as air. Since the calibration constant for helium is less than for air, Eq. 21 shows that the low molecular weight is a decided advantage. Assuming the helium calibration constant to be 0.5 of that for air, it is estimated that a sensitivity of about 1 part in 100 is the upper limit using standard power supplies. The maximum sensitivity with an ordinary Pirani gauge is of the same magnitude. Experiments⁵ seem to show that under given conditions either propane or helium will give about twice the change in deflection on a Philips gauge that it will on a 35T ionization gauge.

The early plant practice of leak hunting with a foreign gas and a pressure gauge has now been superseded generally by the more sensitive methods using mass-spectrometer helium detectors.

3.6 Freon-gas Detection. In this method water lines on other small systems are filled with Freon gas under pressure, and leakage of the gas is determined by sampling the air in the vicinity of suspected spots. The sample is picked up by a rubber hose and fed into an acetylene or gas flame. Even minute quantities of Freon color the flame distinctively. This method is very sensitive and widely used for pretesting water lines. Recently, however, the helium leak detectors have been replacing Freon testing equipment.

3.7 Ammonia and Hydrochloric Acid. If a swab saturated with hydrochloric acid is brought into contact with ammonia gas, a visible white vapor of ammonium chloride is formed so that gas leaking from a tank of ammonia under pressure can be detected. In practice, the tank is filled with the maximum safe pressure of ammonia gas, and the operator moves the saturated swab around the vicinity of suspected parts. The method is capable of as high sensitivity as the Freon test (and is more economical for large systems), and is one of the methods sometimes used when more conventional methods fail for leak hunting on the tank. The low viscosity of ammonia enhances the leak. One great

disadvantage is the possible saponification of the roughing-pump oil when the tank is pumped down, and another is the danger to personnel from the ammonia gas. Measurements have not been made to determine the minimum leak that can be detected by this method.

A variation of this method is to smell for the leakage of ammonia or other aromatic gases. Precise localization of the leak is difficult in either case.

3.8 Penetrating Liquids. If one side of a suspected leak is covered with white powder or chalk and penetrating oil or kerosene is painted on the other side, any oil finding its way through leaks by capillary action will discolor the powder and thus give the position of the leak. Penetration may take several hours or a day, but the method is practical in many cases and is sometimes used in the plant. As with the previous method, no experimental data have been obtained on the minimum leak detectable.

3.9 High-pressure Water-line Test. This method, which has found limited use in the plant, is a practical method of testing for large leaks or weak spots in water lines. Water is pumped into the lines by hand up to a pressure of 200 to 300 lb/sq in. Leaks are shown up by formation of water drops on the line, and weak spots in tubing or joints are burst open. This method will not locate very small leaks, and it requires a skilled operator.

For example, Poiseuille's law gives a flow rate of only 0.492 cc/min through a leak 10^{-3} cm in radius and 0.1 cm long when the water pressure is 300 lb/sq in. Such a hole would have a leakage rate of 8.7 μ -liters/sec of air after the system was evacuated. Of course, for small leaks the surface tension of the liquid will stop the flow. Taking the expression for the radius in terms of the pressure necessary to start the flow from Eq. 16 and substituting in Poiseuille's expression for the volume rate of water flow through a hole 1 cm long,

$$\frac{V}{t} = \frac{2\pi}{\eta} \frac{T^4}{p^3} \text{ cc of water/sec} \quad (24)$$

is the expression for the volume rate below which the leak is effectively stopped because of surface tension. For a water pressure of 300 lb/sq in. the limiting rate from Eq. 24 is 2.2×10^{-12} cc/sec, much smaller than anything detectable by observation, so the practical limitation to this method is not due to surface tension.

3.10 Detection of Water Leaks by Observation of Boiling Rate of Liquid Nitrogen. If a water leak suddenly occurs so that the partial pressure of water vapor in a vacuum system increases, the vapor will

condense on the liquid-nitrogen trap causing the nitrogen to vaporize more rapidly. With many identical systems side by side, as on a track, it is easy to detect by comparison a trap that is boiling excessively. Thus, it is sometimes possible to determine definitely that a water leak is the cause of a pressure rise.

To estimate the minimum leakage that can be detected by observation of the trap boiling rate, let it be assumed that a 10 per cent increase in boiling rate can be observed. In Alpha I the liquid-nitrogen consumption rate is about 0.289 gal/hr per trap and, since each gallon requires 580 Btu for vaporization, a 10 per cent increase in rate of vaporization would represent an increase in heat absorption of 4.7×10^{-3} Btu/sec. If the pumping speed of the pumps is neglected and it is assumed that after equilibrium water vapor is condensed by the trap at the same rate at which it is entering the system, the heat-absorption rate given above if balanced against the heat liberated in reducing the temperature of the water to the temperature of liquid nitrogen (1320 Btu/lb) means that 1.6×10^{-3} cc of water is leaking into the system per second. Then the flow rate of water vapor would be about 170 μ -liters/sec, which would increase the pressure near the trap only 0.017 μ Hg if the pumping speed of the trap is assumed to be 10,000 liters/sec. However, such a large leakage rate near the units, where many constrictions exist, would probably cause a dangerous increase in local pressure.

In conclusion, it seems that if an increase in boiling rate of 10 per cent is noticeable, a water leak of the order of 170 μ -liters/sec can be detected by this method. A flow indicator may be required to detect an increase of 10 per cent, however, and direct observation may require five to ten times this increase.

4. MASS-SPECTROGRAPHIC HELIUM DETECTORS

The mass-spectrographic detectors can detect quantitatively the presence of helium in mixtures as lean as 1 part of helium in 100,000 to 600,000 parts of air. Such high sensitivity characterizes leak hunting with this type of detector and places the method in a category of its own, since it makes possible detection of leaks a thousand times smaller than any of the previously mentioned methods. The introduction and use of helium-detector leak hunting has done more to decrease vacuum outages in the plant than the use of any other technique or instrument.

4.1 General Characteristics. In general, the helium leak detector consists of a small mass spectrometer set to detect helium only, a vacuum system, and an adjustable bleeder valve at the inlet to the de-

tector for maintaining the optimum operating pressure of the detector. When connected to an evacuated system that is being examined for leaks with helium, samples of gas from the system pass through the bleeder valve, and the spectrometer gives an output current proportional to the partial pressure of helium in the detector. In actual practice, however, the bleeder valve is adjusted for the optimum total pressure, and the helium-ion output of the spectrometer is proportional to the fraction of this optimum total pressure that is due to helium. Because of this the sensitivity of the detector is given in terms of the minimum mixture ratio in which helium can be detected. For example, if a detector has a sensitivity of 1 part in 100,000, the output meter will give an appreciably greater indication than that due to random variations when a mixture of 1 part of helium and 100,000 parts of air is leaked into the detector at a rate sufficient to raise the pressure to the operating value.

The maximum pressure at which the unit can be operated is determined by two main considerations; as the pressure increases (1) the filament life decreases and (2) the background due to scattering increases. In practice, the optimum pressure is such that the mean free path is about equal to the ion path in the spectrometer. For instance, in the Westinghouse leak detector the operating pressure is $0.2 \mu \text{ Hg}$, which means that in a 1 to 100,000 mixture the helium is at a pressure of $2 \times 10^{-6} \mu \text{ Hg}$.

The helium leak detector can be used in a variety of ways, several of which are illustrated schematically in Fig. 5.1. In any application, the gas sample to be tested is bled into the leak detector through a valve so as to raise its pressure to the operating value. In Fig. 5.1a is illustrated the general method of sampling gas at high pressure, while Fig. 5.1c illustrates a practical application of the method in testing a vacuum system. The method is especially applicable to water-line testing. The volume being tested is put under helium pressure, and a small needle valve connected to the leak detector "sniffs" the air in the vicinity of suspected leaks. A small fraction of the helium escaping from the leak will find its way into the detector through the "sniffer." The sniffer valve is opened until the detector reaches its operating pressure before leak hunting starts. A satisfactory sniffer valve, designed by A. J. Samuel,⁶ is illustrated in Fig. 5.2. The valve is connected to the detector by means of outgassed rubber hose or metallic sylphon hose. Note the very small volume trapped at high pressure, and the short needle and seat. This is necessary since the cleanup or response time is determined by the ratio of this volume to the speed of the leak and, since the latter must be small, the former must also be kept small. In this method, the mixture ratio entering the

Fig. 5.1 — Helium leak-detector applications.

Fig. 5.2—“Sniffer valve.” This valve is used in conjunction with a helium leak-detector to sample gas at atmospheric pressure.

detector is determined by the location of the sniffer and not by the system under test. The method has been found very practical in the plant even though it suffers from two disadvantages. First, the operator must work skillfully because of the rather long response time; second, the volume is saturated with helium and will be troublesome to pump clean if other techniques requiring evacuation are tried later. The latter difficulty is not real, however, when the method is used almost exclusively on water-line systems, as is common practice in the plant.

The method of using leak-detector vacuum pumps that also evacuate the system under test, is illustrated by Fig. 5.1b. If the leakage rate and volume are small, the throttling valve can be left open so that the system is at an equilibrium pressure not much higher than that in the detector. If this is not possible, the valve can be closed until the operating pressure is reached. In the former case, the pressure can be any value less than the operating value without affecting the sensitivity of the method. If there is only one leak, the flow of gas into the spectrometer will be all helium when the leak is flooded. If there is more than one leak, the mixture ratio will be determined by the ratio of the flooded leak to the total leak. On the other hand, if the valve is partially closed these considerations will be true only after equilibrium has been reached, which may take many minutes in a large volume with small leaks. The practical sensitivity, however, will be reduced since the mixture ratio increases slowly with time after the leak is flooded, and the helium tube may have been moved beyond the leak by the time an indication occurs. This method is often used for small volumes and some water lines, although it requires a fairly long pump-down time even if the system is roughed by a mechanical pump. As in every case where a water line or other damp surface is evacuated, there is great danger that leaks may freeze shut from the cooling effect of rapid evaporation of the water at low pressures.

The three remaining methods illustrated in Figs. 5.1d, e, and f differ from the previous methods in that the mixture ratio is determined by the system being tested and the sample is bled from a portion of the system that is maintained at low pressure by an auxiliary pumping system. After a leak is flooded with helium, the helium content within the system increases until, at equilibrium, the mixture ratio is determined by the size of the leak being flooded and the total leakage rate. If there are no leaks in the pumps, the mixture ratio will be the same in all parts of the system (neglecting the difference in conductance and pumping speed for helium) and the sample may be taken off at any part of the system. The total leakage rate is arrived at by assuming it to be equal to the speed of the pumps at the particular pres-

Fig. 5.3—Throttling valve for use with leak detectors.

sure at which they are working, so that if the mixture ratio is measured by the detector the size of the leak being flooded can be calculated. While the mixture ratio after equilibrium is independent of the point at which the sample is taken, several practical considerations determine the type of test to be used. The interdiffusion rate is about inversely proportional to the pressure, but the minimum pressure is limited to the value that will cause enough gas flow to the spectrometer (through practical connections) to maintain its operating pressure. A good operating range is 10 to 200 μ Hg but the method can be used up to 1 mm Hg without great difficulty. Since these pressures are easily reached with mechanical pumps, the test illustrated by Fig. 5.1d is widely used.

The throttling valve⁶ used in these tests is usually a $\frac{3}{8}$ -in. Kerotest valve with a 20 to 1 worm-gear drive for fine adjustments. This arrangement is shown in Fig. 5.3.

The testing of units is usually done in dry docks, which are small tanks designed to receive the individual units. The dock is evacuated by means of a fast mechanical pump. After the leak detector is throttled into the header, individual water lines and pump-outs are put under helium pressure, and the indication of the detector is noted. If no leaks are indicated, a hood is placed over the exterior of the unit and flooded with helium so that any leaks in the unit will show up. If the water lines leak, they are given a soap test or the unit is reversed in the dock so that the lines are exposed and evacuated. Leak hunting then proceeds on the lines. If, however, a leak is indicated in the unit proper, the hood is removed and individual parts are tested. Localization of the leaks is quite rapid.

The sensitivity of the leak detector is measured by admitting a known mixture of air and helium and calculating from the deflection the mixture ratio that would give the minimum reliable deflection. The known mixtures are obtained in three ways:

1. Known air and helium leaks are admitted into a vacuum system from which the sample is taken.
2. A known helium leak is admitted into a system with an accurately known pumping speed. The air leak is assumed to be that which would maintain the system at the pressure held by the pump.
3. A mixture is made by any of a variety of ways and stored in pressure tanks until ready for a test.

The last method is least reliable but all three find application in the plant. The three methods usually agree within 20 to 30 per cent on successive measurements on the same detector.

It is interesting to note that leaks are easily found in the plant which are so small as not to affect operations, but the high sensitivity of the

detectors is very desirable in leak hunting where quick response is necessary.

The reason for the choice of helium as a leak-hunting gas to use with the mass spectrometer deserves some discussion. Aside from the obvious advantages mentioned in Sec. 2.6, helium has some special advantages peculiar to its detection with a mass spectrometer:

1. The molecular weight is low so that no stringent requirements are placed on the resolution of the spectrometer.
2. Helium is not ordinarily found in the air in appreciable quantities.
3. Helium is monoisotopic so that all the ion current due to helium can be collected at one mass position.
4. Most important, however, is the fact that the mass 4 position is the only position in the spectrum of the lighter elements that does not suffer from excessive background. Since high sensitivity is desired, the gain of the receiver amplifier is increased until the background is almost objectionable. With this high sensitivity multiple ionization and metastable ions formed from the ever present O_2 , N , H_2O , C , CO , CO_2 , H_2 , and many others, including hydrocarbons and organic compounds, cause objectionable background except at mass 4.

For these reasons, there seems to be little likelihood that other gases will ever find extensive application in leak hunting with the mass spectrometer.

4.2 Types of Helium Leak Detectors. Only three fundamentally different types of helium leak detectors have been used extensively in the plant. These are the General Electric (Nier type), Westinghouse (180-deg deflection), and the cold-cathode type (180-deg deflection and P.I.G. source) as well as variations on the first two. Since the General Electric and Westinghouse types are commercially available and the third type is described elsewhere⁷ by R. Loevinger, who perfected it, this section will be limited to operational characteristics evident in the plant, experimental work done on the detectors, and a superficial description.

(a) **General Electric Leak Detector.** This detector was developed from original work done by Andrew Hustrulid and Alfred O. C. Nier at the University of Minnesota under contract No. OEMar-149.⁸ Figures 5.4 and 5.5 are views of a recent-model detector showing pertinent features. The spectrometer is contained in the Y tube. The source consists of a filament providing an electron beam, which bombards the gas, the ions being drawn out and collimated by means of slits. The ion beam is deflected approximately 60 deg by a small permanent magnet located inside the evacuated Y tube at the point where the pumping connection joins the tube. The collector end consists of a defining slot, a positive low-energy ion repeller, a collector plate, and a 954

acorn tube used as an electrometer tube mounted in vacuum with its 10^{11} -ohm input resistor.

The vacuum-pumping system consists of a MC-275 (D.P.I.) diffusion pump and a Welch mechanical pump. The system is double trapped, that is, the sample bleeds through a tube immersed in the liquid-nitrogen trap that serves the vacuum system. The electrical equipment is supplied from the 110-volt a-c line except for a 6-volt storage battery and B batteries supplying the d-c amplifier. Cooling-water connections for the diffusion pump limit the location of the detector to a slight extent.

An early model by G.E., very similar to the one described in the report mentioned, was tried in XAX during the latter part of 1943, but was rejected for plant application as too complex and delicate for use without an extensive training program. A more rugged model with an oil diffusion pump and metal spectrometer tube was introduced in the fall of 1944 and found usable. By May 1945 their use was general throughout the plant, and by May 1946 about 100 G.E. detectors were on the area with only 60 machines in the operating cycle, of which 40 were in use at any one time. Servicing costs are \$400 per month for each of the 60 machines, while the initial cost was \$3,000 each. Their average sensitivity is 200,000 to 250,000 and may run as high as 600,000; however, no completely satisfactory method of accurately checking sensitivity is available.

(b) Westinghouse Leak Detector.⁹ This detector, shown in Figs. 5.6 and 5.7, was developed at the Westinghouse Research Laboratory for their own use but was made available to CEW-TEC during the spring of 1945. Deliveries during 1945 have brought the total number as of May 1946 to about 40 machines, with only 14 in the operating cycle owing to lack of repair parts. The initial cost was \$5,000 each, while the upkeep is somewhat less than for the General Electric machine.

Figure 5.7 shows the major features of the unit, while Fig. 5.8 is a detail of the spectrometer tube with one of the magnetic cover plates removed and the magnet swung out of the way. The Philips ion gauge is shown with the near iron cathode removed, allowing the ring and the opposite cathode with its pump-out holes to be visible.

The ion source consists of a gas box in which the gas is ionized by an electron beam accelerated from the tungsten filament and collimated by the magnetic field. The helium ions are deflected through 180 deg in a $1\frac{3}{8}$ -in. radius; they pass through an energy filter and strike the collector plate. The collector plate is connected through a Kovar seal to a 2×10^{11} -ohm input resistor contained in the roughly evacuated electrometer chamber with the 507 electrometer tube. The vacuum system is all metal with a liquid-nitrogen trap, a Westinghouse air-cooled diffusion pump (20 liters/sec), and a Welch mechanical pump.

Fig. 5.4—General Electric leak-detector unit. The upper panel contains the controls and meters for the spectrometer. The lower panel is the ionization-gauge supply. The small case on top is the thermocouple-gauge power supply.

Fig. 5.5 —Rear view of the General Electric detector showing many of the essential parts. The small Alnico deflecting magnet is contained in the Y tube at its junction with the vacuum system.

Fig. 5.6 — Westinghouse leak-detector unit, showing the control panel on the front, indicating unit on top, and vacuum valves on the right side. The sample is admitted through the connection shown.

Fig. 5.7—Rear view of the Westinghouse leak detector, showing the location of the major parts. The 714 trap is kept filled with liquid nitrogen.

Fig. 5.8 — Interior view of the Westinghouse mass spectrometer tube with the cover plate and magnet removed. The baffles limit the angular spread of the beam. The near iron cathode of the Philips ionization gauge has been removed.

(c) Cold-cathode Leak Detector. The cold-cathode leak detector as eventually used in the plant was a 180-deg deflection type with a radius of 2 in. The ion source was a P.I.G. with a semicylindrical anode. The ions were drawn out of a slot placed lengthwise in the flat side of the anode. As with the other types described, a positive-grid energy filter was used before the collector to decrease background, due in this case to the energy distribution of the ions emerging from the anode. The vacuum system had no cold trap and used a Distillation Products diffusion pump (10 liters/sec) backed by a Cenco Hyvac. In contrast with the other two types, this detector uses an a-c component on the d-c accelerating voltage so that the beam swept across the collector, the helium peak causing an alternating input to the a-c amplifier. The output of the amplifier passed through a synchronous filter, which rejected most of the unwanted high-frequency components due to noise, etc.

In the spring of 1945 R. Loevinger,¹⁰ working in the CEW-TEC Vacuum Research Laboratory, completed alterations on three leak detectors sent from UCRL. These revised units proved satisfactory in the plant, requiring little attention and skill on the part of the operator, but orders pending on the Westinghouse and General Electric detectors made it inadvisable to procure additional cold-cathode units of this type.

4.3 Evaluation. Experience on this project with helium leak detectors, while demonstrating the value of mass-spectrometer leak detecting, has indicated the desirability of further development to combine the advantages of the various types. The cold-cathode detector is by far the simplest to operate, requires least maintenance, is the most rugged, and, advantageously, requires no cold trap. While all three types have adequate sensitivity for plant use, the Westinghouse machine is apparently the most sensitive and the cold-cathode detector least sensitive. The cold-cathode detector shows great promise for the future if sufficient development work is undertaken.

5. DRY DOCKS AND DRY-DOCKING PROCEDURES

When the plant was originally designed, it was assumed that every unit would have to be tested for tightness under vacuum before it was transferred to the bin so that non-innage time due to leaks would be a minimum. Consequently, buildings were provided with numerous auxiliary vacuum systems, or dry docks, in which different units would be tested. Before mass-spectrometer helium detectors found general use, systems were pumped down and were required to pass a rate-of-rise and base-pressure test. This was too slow to provide tested units for all the process bins, and many units were sent directly to the tank without pretests. Opinions on the adequacy or necessity of dry-docking

varied considerably until the advent of helium leak hunting in the dry docks, when it was definitely shown that only a few dry docks with mechanical pumps were required when used in conjunction with leak detectors since testing and localization of the leaks were so rapid. Section 3.1 describes the technique used with the dry dock and helium detector.

In addition to test docks for M's, E's, and liners, special docks were often provided for testing Alpha I K's, Alpha II K tools, ionization gauges, and spark plugs.

M and E docks were sometimes fitted with vacuum-pressure hoods, which enclosed the external part of the unit. This arrangement allowed the external pressure to be changed over a considerable range so that changes in pressure in the dry dock would indicate changes in leakage rate and hence the presence of a leak. The hoods never found widespread use and at time of writing light loose-fitting hoods were used to cover the unit when it was flooded with helium.

6. RESEARCH AND DEVELOPMENT

6.1 Vacuum Analyzer. During the summer of 1944, two vacuum analyzers built at UCRL¹¹ were run experimentally at XAX. Extensive tests indicated that no gain in production would result from a plant installation of analyzers. Since delivery of even a few experimental units could not be made for six months, installation plans were dropped, and adaptation of the units as leak detectors was attempted.

6.2 Cold-cathode Leak Detectors. Since the vacuum analyzer was a sturdy form of mass spectrometer, attempts were made here, at UCRL, and at Westinghouse Research Laboratories to develop it into a helium detector suitable for use as a leak detector. All the work indicated that a small, compact unit was possible, but the maximum sensitivity was about 1 part in 1,000. In January 1945, Westinghouse delivered two experimental models of a cold-cathode leak detector without a self-contained vacuum system, patterned after the earliest models used at UCRL. Even after various improvements were made here, the sensitivity was too low for revised plant requirements and Westinghouse engineers turned their attention to delivery of a number of more conventional, filament-type helium detectors. By fall of 1945, all concentrated leak-detector development was halted.¹²

6.3 Philips Gauge Frequency Response to Helium. The characteristic high-frequency oscillation in the current to a d-c Philips gauge is found to vary in frequency with the pressure and nature of the gas.¹³ A limited amount of experimental work was done which seemed to show that if the quiescent oscillations could be stabilized, a very sensitive

leak detector could be constructed using a heterodyne method of detecting changes in frequency due to the presence of helium.

6.4 Helium Detection with Optical Spectroscopy. A. J. Samuel and L. Foote¹⁴ did experimental work on detecting, by means of a spectro-scope, the presence of helium in a discharge connected to the vacuum system. Their preliminary work involved visual observation of the discharge spectrum with a small-grating spectroscope. Maximum sensitivity obtained was about 1 part in 1,000.

6.5 Standard Leaks.⁶ Satisfactory operation of helium leak detectors requires the use of small, known leaks with which to check the sensitivity of the detector. No satisfactory fixed leak has been found, but a small adjustable and filter-equipped needle valve has proved very satisfactory.

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Chapter 6

PUMP-DOWN AND OUTGASSING

By C. E. Normand and Alan J. Samuel

1. THE INNAGE-TO-OUTAGE RATIO

In an operation such as the electromagnetic separation of uranium, maximum plant utilization demands that the ratio of average innage time to outage time be as large as possible. To realize the maximum average innage it is necessary that in each operating cycle reception of the resolved ion beam be established as early as possible and continue without interruption to charge exhaustion. Also, charge utilization, i.e., the ratio of charge-consumption rate to rate of reception, must be kept at the lowest attainable value. To assure minimum average outage per cycle, not only must interruption of established reception be avoided, but also the time consumed in necessary inter-run operations must be reduced to a minimum.

Interruptions of reception and premature terminations may result from numerous operational errors and equipment failures. Many of these errors and failures have been substantially reduced in the course of plant operation, but their complete elimination can hardly be expected.

The magnitude of these reductions in outage is illustrated in Table 6.1, which gives a breakdown of the average operating cycle in an Alpha I building at two widely separated dates.

Note that during this 14-month period the length of cycle has increased 15.3 per cent; innage has increased from 62 to 92 per cent of the total cycle; start-up time has decreased by 37.5 per cent; and outage during the production run has decreased from 43 per cent of innage to an almost negligible 1.67 per cent.

Vacuum failures were a serious cause of outage during runs in early plant operation. This situation has been greatly improved, both by a reduction in the frequency of such failures and by improvements

in the techniques of detecting and correcting failures that do occur. Production stoppages due to errors in vacuum operation have decreased with greater experience on the part of operating personnel and with improved operating procedures. Stoppages due to failures of vacuum equipment have been reduced by improved equipment, improved servicing, and improved operation. All these factors have contributed to the increase in innage per cycle.

Table 6.1—Typical Operating Cycles in Alpha I, Tracks 3 and 4

Date	Time in operation, months	Av. length* of cycle, hr	Av. innage per cycle, hr	Av. start-up time, hr	Av. outage during run, hr
6/44	3	282	175	32	75
8/45	17	325	300	20	5

*Only those runs attaining innage are considered.

2. THE INTERRUN PERIOD

The time elapsing between the termination of one run and the beginning of reception in the succeeding run has likewise been reduced in the course of plant operation, but this has always been, and at the time of writing still remained, the major cause of outage. Operations such as the removal and installation of units are now carried out efficiently by trained handling crews. Better scheduling of terminations allows more efficient utilization of start-up equipment and personnel, and the scheduled overlapping of operations wherever possible has effected added economies of time. There are three operations, however, that have presented serious outage problems. These are (1) the cooling of units prior to their removal from the process tank; (2) the heating of units to operating temperature; (3) the evacuation of the system to operating pressure.

2.1 Cooling Down. To prevent destructive oxidation of equipment, product operating units must be allowed to cool to moderate temperatures (250°C) following the termination of a run and prior to breaking the system to air. In the Alpha plants 1.5 hr or more has been allowed for cooling down. The corresponding time in the Beta system is 1 to 1.25 hr. Realizing the unfavorable cooling conditions existing in an evacuated space, the practice of introducing some inert gas (helium) to a pressure of about 2 mm Hg immediately following ter-

mination has been adopted. The resulting acceleration in rate of cooling has reduced the cool-off period for Beta units to about 15 to 20 min.

2.2 Heating Up. Heating of the units to operating temperature at the beginning of the run is carried out concurrently with the pumping down of the system and does not contribute directly to interrune outage. Since heating up, striking the arc, and establishing the ion beam affect the rate of outgassing of the system being evacuated, however, these operations become significant parts of the general pump-down problem.

2.3 Pumping-down. The pump-down phase of the operating cycle embraces the period of evacuation from atmospheric pressure to a pressure permitting acceptable reception of the resolved ion beam. Concurrent operations such as those mentioned above may affect the pump-down time but do not, of themselves, limit the beginning of innage. The pump-down problem may then be properly thought of as a vacuum problem complicated by the influence of such related operations as heat-up and arc and beam initiation.

During the early days of plant operation, pump-down contributed significantly but not predominantly to total outage. As operating routine was established, however, it became the major outage factor and accounted for some 75 per cent of the average total outage per cycle. It is not surprising, therefore, that much thought and effort has been directed toward the reduction of pump-down time in the various plant systems, or at least to the maintenance of a favorable ratio of pump-down to innage time.

Major factors influencing directly the time required for pumping down a system are (1) leakage detection and correction, (2) removal of free air from the system, and (3) reduction of outgassing to a tolerable rate.

Most vacuum troubles arise during the pump-down period, and the amount of time devoted to the detection and correction of these troubles has varied widely from one pump-down to the next and from one period of operation to another. Records of operation of the first four Alpha I tracks show that for the last week of June and the first week of July 1944 almost 28 per cent of all outage was charged to "time spent determining suspected vacuum trouble." A year later, however, this figure had dropped to a little less than 7 per cent. This improvement may be attributed in part to experience, in part to improved operating procedures and servicing techniques, but mainly, perhaps, to the initiation in the interim of a program of pretesting of all units in vacuum docks prior to their installation.

In the Alpha II and Beta systems vacuum-docking facilities for pretesting units were available from the first. Some experience had also been gained in the operation of the Alpha I systems before these later plants were put into production. Consequently, vacuum outage was less serious in these than in the earlier Alpha I systems.

3. THE CURRENT OPERATING CYCLE

Still further reductions in vacuum outage have resulted from the more sensitive pretesting made possible by the use of helium leak detectors. Data recorded in early 1946 on the operation of eight Beta tracks showed the average total outage for all runs attaining innage to be only 6 per cent of the cycle, and of the total outage only 3.6 per cent was due to vacuum failures. Thus outage due to vacuum trouble accounted for 0.22 per cent of the average operating cycle and was no longer a serious problem either as a factor prolonging pump-down or as a cause of interruption of established operation.

Likewise, 1946 data on Beta operations shows that of 348 runs initiated, 329 runs (94 per cent) attained innage and only 19 runs (6 per cent) terminated without innage. The 19 runs terminating without innage continued for less than an hour on the average and added only 1 per cent to the total outage in all successful runs. Of the 329 runs attaining innage, 17 runs (5.2 per cent) were terminated short of charge exhaustion because of various vacuum failures. The average innage for these 17 runs was 38.2 hr as contrasted with a 71.8-hr plant average. Therefore, but for these premature terminations the average innage time per cycle would have been greater by some 1.9 hr (2.6 per cent).

Comparable data covering established operation in Alpha I and Alpha II are not available. It is believed, however, that at peak operating efficiency the percentage values of various outages due to vacuum failures in these systems were not substantially lower than in the Beta system.

4. THEORETICAL PUMP-DOWN TIME

The most obvious and also the most definite requirement of the pump-down operation is that of removing free gas from the system being evacuated. Unfortunately, however, the time required for this operation proves to be a very poor criterion for predicting actual pump-down time. This is due in part to the unpredictable delays introduced by leaks but even more to the outgassing of various parts of the evacuated system.

If it is assumed that there are no pump-down delays due to vacuum failures and that outgassing of the evacuated system is negligible, it is then possible to calculate with some precision the time required to reduce the pressure in a given system from atmospheric pressure to any required value.

In making such calculations it is necessary to consider pump-down as occurring in three stages:

1. Roughing, in which a mechanical pump evacuates the system from atmospheric pressure to an arbitrary pressure at which the diffusion pumps will function effectively.

2. Prefinishing, in which the pressure is further reduced by the diffusion pumps operating at their maximum fore pressure but at less than their maximum speed.

3. Finishing, in which the diffusion pumps, having attained their full speed, further reduce the pressure to the required operating value.

Throughout the roughing stage the volumetric speed of evacuation is constant except for the decreasing efficiency of the mechanical pump and the decreasing conductance of the roughing line as the pressure is reduced. Since these variations are not large, an average speed based on average volumetric efficiency of the pump may be used without introducing serious error.

The pressure then decreases exponentially as expressed by the equation

$$(P_2 - P) = (P_1 - P)e^{-St_1/V} \quad (1)$$

Solving Eq. 1 for t_1 ,

$$t_1 = \frac{V}{S} \ln \frac{P_1 - P}{P_2 - P} \quad (2)$$

where P is the base pressure of pump in microns of mercury, P_1 is the initial pressure in system in microns of mercury, P_2 is the final roughing pressure in microns of mercury, S is the average volumetric speed of roughing pump in liters per second, V is the volume of system in liters, and t_1 is the time in seconds to reduce pressure from P_1 to P_2 .

In the prefinishing stage, on the other hand, evacuation occurs at an essentially constant mass rate limited by the limiting diffusion-pump fore pressure and the speed of the backing pump at this intake pressure.

$$Q = P' \times S'' \quad (3)$$

where P' is the limiting diffusion-pump fore pressure in microns of mercury, S'' is the volumetric speed of backing pump in liters per second, and Q is the rate of evacuation in micron-liters per second, and where the pressure in the system is reduced at a constant rate. But,

$$P_2 - P_3 = \frac{Q}{V} t_2 \quad (4)$$

Solving Eq. 4 for t_2 ,

$$t_2 = \frac{V}{Q} (P_2 - P_3) \quad (5)$$

where P_2 is the initial prefinishing pressure in microns of mercury, P_3 is the final prefinishing pressure in microns of mercury, and t_2 is the time in seconds to reduce the pressure from P_2 to P_3 .

The final prefinishing pressure, P_3 , is not arbitrary but is determined by the attainment of full diffusion-pumping speed. Thus,

$$P_3 \times S' = P'S'' \quad (6)$$

where S' is the full speed of diffusion pumps.

Once full-speed diffusion pumping has been attained, evacuation continues at this constant volumetric rate until the required operating pressure P_4 is reached, and the time required for this finishing stage is, according to Eq. 2,

$$t_3 = \frac{V}{S'} \ln \frac{P_3 - P_0}{P_4 - P_0} \quad (7)$$

The results, after the indicated calculations for typical plant systems have been made, together with essential operating data are shown in Table 6.2.

It may be noted that in all systems finishing time is quite negligible compared with roughing time. This is also true for prefinishing time except in the case of Alpha II systems, in which large volume, small finishing-pump capacity, and low-limiting fore pressure combine to require a relatively long prefinishing period.

When these theoretical pump-down times are compared with the times actually required in plant operation, however, two pertinent facts are revealed:

1. Actual roughing time, under optimum conditions, is not significantly greater than the calculated time. Average roughing time is greater because optimum conditions are seldom realized.

Table 6.2—Theoretical Pump-down of Typical Plant Vacuum Systems.*

	Symbol	Unit	Alpha I	Beta	Alpha II
Volume of system	V	Liters	8,840	2,450	13,100
Initial pressure	P ₁	μ Hg	750,000	750,000	750,000
Final roughing pres.	P ₂	μ Hg	50	50	50
Final prefinishing pres.	P ₃	μ Hg	1.94	2.67	0.254
Final finishing pres.	P ₄	μ Hg	0.04	0.04	0.04
Base pres. roughing pump	P	μ Hg	10	10	10
Base pres. diff. pump	P ₀	μ Hg	0.004	0.004	0.004
Limiting fore pres. diff. pump	P'	μ Hg	130	130	130
Capacity of roughing pump		Cfm	303	105	303
Av. speed, roughing pump	S	Liters/sec	114	40	114
Capacity of backing pump		Cfm	303	303	105
Speed backing pump at P ₃	S''	Liters/sec	93	93	30
Speed of diffusion pump at tank	S'	Liters/sec	6,300	4,500	14,000
Roughing time $t_1 = \frac{V}{S} \ln \frac{P_1 - P}{P_2 - P}$	t ₁	Min, sec	12 min, 42 sec	10 min, 3 sec	18 min, 50 sec
Prefinishing time $t_2 = \frac{V}{S''} \ln \frac{P_2 - P_3}{P_3 - P_0}$	t ₂	Min, sec	35 sec	10 sec	4 min, 1 sec
Finishing time $t_3 = \frac{V}{S'} \ln \frac{P_3 - P_0}{P_4 - P_0}$	t ₃	Min, sec	6 sec	2 sec	2 sec
Total pump-down time	t	Min, sec	13 min, 23 sec	10 min, 15 sec	22 min, 53 sec

*Dry air and no leakage of outgassing assumed.

2. Prefinishing and finishing times in plant operation are not of the same order of magnitude as corresponding calculated times, but are many times greater. As a result, actual pump-down times for the various systems are many times greater than the times calculated.

Table 6.3 compares calculated with observed average total pump-down time for each of three types of systems.

All evidence points to outgassing of the evacuated system as the principal cause of this wide discrepancy between observed and calculated pump-down time. Minimum observed pump-down times are never less than about half the average values given above, and these can occur only in the absence of leakage and when outgassing conditions are most favorable. It follows that any substantial reduction

in the pump-down time of existing systems can be realized only if the outgassing situation can be improved.

Table 6.3—Comparison of Pump-down Times

System	Calculated time, min	Av. observed time, hr	Obs./calc.
Alpha I	13.4	24	107
Beta	10.3	3.5	20
Alpha II	23.0	18	47

5. THE OUTGASSING PROBLEM

Outgassing may be defined as the releasing into an evacuated space of gases and vapors occluded in or adsorbed on the surfaces of bodies exposed to the vacuum. Thus the principal contributing factors are (1) vaporizations of liquids wetting exposed surfaces, (2) liberation of adsorbed gaseous films from these surfaces, and (3) liberation of gases and vapors adsorbed physically or chemically onto surface materials.

5.1 Vaporization of Liquids. Exposed liquids having high vapor pressures vaporize readily at moderately reduced pressure. At room temperature water boils when the pressure above it is reduced to 17.5 mm Hg, and at 4.57 mm Hg pressure boiling occurs even at 0°C. Thus water or any other volatile liquid occurring as an exposed liquid in the system is removed during the early roughing stage of evacuation and contributes little to the total pump-down time. Oils and other low-vapor-pressure liquids vaporize less rapidly and may continue to contribute to the total outgassing throughout the evacuation process. Reasonable care in cleaning exposed surfaces of oil, grease, flux, etc., effectively eliminates this factor.

5.2 Composition of Adsorbed Gases and Vapors. Adsorbed materials are much less readily released, however, and it is primarily these which are responsible for prolonged outgassing. Analysis¹ by elementary mass-spectrographic methods of the gases present in a process tank during the outgassing period shows the presence of air, water vapor, hydrogen, and carbon monoxide, but of these constituents water vapor is by far the dominant factor. This is shown graphically in Fig. 6.1. From the preponderance of water vapor in the outgassing system it is obvious that any effort to minimize outgassing may be devoted to the exclusion or accelerated release of water vapor from the system.

5.3 Rate of Outgassing as a Function of Pressure. Gases and vapors adsorbed on and beneath the exposed surfaces are liberated at a rate that increases as the pressure in the system is reduced. Considering the system to be at room temperature, contributions from these sources may be considered negligible during the roughing stage of pump-down (760 mm Hg to 100 μ Hg). Beyond this point, however, an increasing rate of outgassing and a decreasing pumping speed result in a pressure equilibrium at which the quantity of gas and vapors removed by pumps and trap per second is just equal to the quantity liberated to the system from all sources.

Fig. 6.1—Composition of tank gases during bake-out.

As outgassing continues the rate decreases owing to (1) a reduction in the residual quantity of adsorbed material and (2) an impoverishment of the more freely liberated constituents. Consequently, the equilibrium pressure is reduced and the desired operating pressure is eventually obtained.

5.4 Methods of Reducing Outgassing Time. If means are sought for reducing the prolonged interval during which the pressure in a system remains above the operating value because of excessive outgassing, three distinct approaches appear: (1) the speed of pumping from the system may be increased; (2) the initial quantity of occluded gas and vapor may be reduced; (3) the average rate of outgassing may be accelerated.

5.5 Effect of Pumping Speed on Outgassing Time. Any increase in the speed of pumping from a system, and this includes both dif-

fusion pumps and cold traps in so far as condensable vapors are concerned, will result in the attaining of operating pressure at a higher rate of outgassing than would otherwise be possible, but will have little if any effect on the rate of outgassing at any given pressure. Consequently, a considerable increase in pumping speed may result in a relatively small decrease in pump-down time. An increase of some 60 per cent in the speed of the pumps on the fifth Alpha I track resulted in a reduction of 15 per cent or less in pump-down time.

5.6 Reduction of Initial Amount of Adsorbed Material. Many schemes have been proposed for reducing the quantity of material, especially water, adsorbed by the tank and units between runs, or of reducing this quantity by some preevacuation treatment. To the extent that such measures can be effectively applied, the initial equilibrium pressure is reduced, thereby shortening the time required to attain operating pressure.

Various methods of surface cleaning, interrun bake-out, and coating of tank walls have proven rather ineffective in reducing outgassing. Likewise, the prebaking of units in vacuum docks has little effect unless the units are protected against exposure to the air while in transit from dock to tank.

Moisture exclusion as a means of reducing outgassing is highly effective provided exclusion can be reasonably complete. This is very difficult to realize, however, in systems designed without thought being given to such operation. The quick-change procedure in which a Beta or Alpha source or receiver is removed and replaced in the shortest time possible yields favorable results. However, complete exclusion would involve the use of unit-change locks in removing and inserting units, and the use of some really dry gas in bringing the tank to atmospheric pressure. Although the incorporation of change locks in the original design of equipment might be feasible, their addition to existing equipment presents considerably difficulty.

5.7 Acceleration of Outgassing. The most effective method found applicable to plant vacuum systems as a means of reducing the pump-down time is that of accelerated outgassing. To attain operating pressure under normal operating conditions, a minimum quantity of adsorbed material, largely water vapor, must be removed from the system. Assume, for the moment, that some means exists by which the rate of liberation of adsorbed gas and vapor may be completely controlled while this quantity of material is being removed. It would then no longer be necessary to wait for the pressure to drop slowly from the initial equilibrium value to operating value. By controlling the rate of outgassing, material could be liberated as rapidly as it could be removed by pumps and traps. Thus gas would be removed at

the maximum rate, and the required outgassing would be accomplished in a fraction of the original time. As soon as this had been accomplished, normal operating conditions could be restored, and with the resulting decrease in rate of outgassing the pressure would drop sharply to operating value.

Fortunately the assumed control of outgassing can be partially if not fully realized as a result of the dependence of the outgassing rate on temperature. While no quantitative statement of this dependence can be made for a complex case in which various gases and vapors are adsorbed on and in surfaces of various compositions, it can be stated, in general, that the rate of outgassing of a given surface increases with increasing temperature.

Fortunately, also, the heating of certain elements of the operating unit is a necessary part of the start-up procedure. Heating of the charge, striking of the arcs, and initiation of the ion beams are operations that may be so timed and executed as to accelerate the rate of outgassing. These operations heat only limited parts of the system, however, and they heat these parts only to normal operating temperature.

If the required total outgassing is to be accomplished in the shortest possible time, all parts of the system must be heated to temperatures well above their normal operating values. Moreover, this heating must be initiated early in the pump-down and must be maintained at such a level as to produce an outgassing rate equal to the full capacity of the pumping system. Finally, it must be possible, once the required outgassing has been accomplished, to reduce all temperatures quickly to their normal operating values.

5.8 Forced Bake-out Procedure. The above conditions are most nearly realized by the use of heating-cooling coils or pads attached to units, liners, and tank walls. The circulation of hot water through these coils and pads, augmenting operational heating, raises temperatures above normal operating values. Substitution of cold for hot water at the end of the bake-out period quickly reduces temperatures, outgassing rate, and pressure to operating values.

Figure 6.2 shows idealized pump-down curves under various conditions of outgassing. The total quantity of gas to be removed is represented by the areas under the curves and is assumed to be the same in all cases.

Complete application of the above procedure was not generally applicable to plant systems since no provision was made for heating the Alpha I or Beta tanks, nor were units originally designed for the circulation of both hot and cold water through their coils. By the early

and rapid application of operational heating, abetted by whatever auxiliary heating was available, a forced bake-out procedure² was found to reduce the pump-down time in Alpha II tanks by approximately 50 per cent.

All experience with forced bake-out procedures indicates that higher temperatures than are attainable with hot water would be advantageous. Although the use of internal electric heaters³ has been shown to be effective, the design of existing plant equipment precluded their efficient installation. The use of high-pressure steam has been considered but never applied, largely because of the hazards involved.

Fig. 6.2—Forced vs. slow bake-out.

Heated air has been proposed as a possible solution, but the proposal has not been carried out.

5.9 Elimination of Cold Traps. Since the closing down of the Alpha plants the problem of outgassing and pump-down have diminished in importance because of the very satisfactory conditions that have always obtained in the Beta plants. With the recent elimination of refrigerated traps from these systems, however, the water vapor liberated by forced outgassing has in some cases overtaxed the capacity of the pumps. This condition has occurred in the fore-vacuum system and has been met by an increase in the available backing-pump capacity during the bake-out period.

5.10 Quantity and Origin of Water Liberated by Outgassing. Estimates have been made of the quantity of water vapor released by the outgassing of typical Alpha tanks and units. Estimates based on anal-

yses of pump-down cycles and on the quantity of ice collected on the liquid-nitrogen trap are in essential agreement and indicate the liberation of about a quart of water during an average pump-down.

Considerable evidence appears to support the assumption that this water is adsorbed, in large measure, from the air during the time units are being changed. The fact that a thoroughly outgassed system can be reevacuated in a much shorter time after having been brought to atmospheric pressure by the introduction of dry nitrogen than after similar treatment with ordinary air, the fact that a seasonal variation of pump-down time with absolute atmospheric humidity can be detected, and the fact that the occasional washing of tank walls between runs has little effect on the subsequent pump-down time all indicate that under normal interrun exposure to the atmosphere moisture is adsorbed to a point approaching equilibrium.

5.11 Design of Systems for Small Outgassing Time. From the above considerations it follows that any effective plan for excluding moisture during change of units must provide complete exclusion, thus requiring elaborate change equipment. If exclusion is not practiced and forced bake-out is relied upon, provision must be made for the rapid release and removal of the large volume of vapor represented by the vaporization at low pressure of approximately a quart of water. If bake-out occurs at an average pressure of 2μ Hg this volume of water yields approximately 500,000 liters of vapor, or 575 times the capacity of an Alpha I tank.

In the design of future systems of the types here considered, serious thought should be given to the practicability of providing:

1. Materials of construction whose surfaces are initially, and will remain, less adsorbent of gases and vapors than the materials now employed.
2. Facilities for excluding air and moisture from the system to the highest possible degree.
3. Means of accelerating outgassing to and beyond the point required for the attainment of operating pressures.

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Appendix A

VACUUM FORMULAS

By C. E. Normand

In the following pages are listed the various formulas found useful in the design of vacuum equipment and systems and in the evaluation of their performance. Formulas are presented in their simplest form. Accuracy has been sacrificed for simplicity in some cases, but wherever this has been done the accuracy retained is adequate for the practical requirements of most large system problems.

For derivations and detailed treatments of these formulas, the reader is referred to the paper by Loevinger in National Nuclear Energy Series, Division I, Volume 1, Chap. 1. Also, reference is made to CEW-TEC work on diffusion pumps and diffusion pump tests,¹ and on gaseous flow.²

All formulas given in this appendix apply to the removal of air at normal temperature, unless otherwise stated, and the symbols and units used therein are as follows:

- A = cross-sectional area of pipe or duct; area of aperture in square inches.
- A' = effective area of cold trap in square inches.
- B = volumetric flow rate in liters per second.
- b = atmospheric pressure in millimeters of mercury.
- C, C₁, C₂ = conductance of pipe, duct, or aperture in liters per second.
- d = diameter of circular aperture or section in inches.
- e, w = cross-sectional dimensions of rectangular duct, $e \geq w$.
- G = mass flow rate in grams per second.
- L = length of pipe or duct in feet.
- O = cross-section perimeter in inches.
- P = pressure in microns of mercury.
- P₀ = base pressure in microns of mercury.

$\bar{P}$ = average pressure in microns of mercury.

P_1 = intake pressure or initial pressure in microns of mercury.

P_2 = discharge pressure or final pressure in microns of mercury.

Q = quantity flow rate in micron-liters per second.

R = rate of pressure rise.

ΔR = change in rate of pressure rise due to any arbitrary leak (pressure in any units).

ΔP = change in equilibrium pressure due to same leak as ΔR above (pressure in same unit employed above).

r = leak rate in cubic centimeters per second measured at atmospheric pressure.

S = speed of pumping at any point in liters per second.

S_p = speed of a pump in liters per second.

S_T = speed of a trap in liters per second.

t = time in seconds.

V = volume in liters.

ρ = density of air at 1 μ Hg pressure in grams per liter
(1.6×10^{-6} g/liter for air).

1. Pumping speed of a pump = volume of gas entering the pump per second, measured at intake pressure.

(a) By metered leak method,

$$S_p = \frac{r b}{P_1 - P_0} \quad (1)$$

(b) By rate-of-rise method,

$$S_p = V \frac{\Delta R}{\Delta P} \quad (2)$$

2. Conductance of a pipe, duct, or aperture.

(a) Conductance = quantity of gas ($P \times V$) flowing per second per unit pressure drop.

$$C = \frac{Q}{P_1 - P_2} \quad (3)$$

(b) If elements having conductances C_1 , C_2 , etc., are connected in series, their joint conductance, C , is such that

$$\frac{1}{C} = \frac{1}{C_1} + \frac{1}{C_2} + \dots \quad (4)$$

(c) If a tank is evacuated by a pump of speed S_p connected by a pipe of conductance C , the speed S of pumping from the tank is such that

$$\frac{1}{S} = \frac{1}{S_p} + \frac{1}{C} \quad (5)$$

3. Three modes of gaseous flow are recognized.

- (a) Turbulent flow occurs only at high pressure and is of negligible significance in vacuum systems.
- (b) Laminar flow is normal in roughing and fore-vacuum systems.
- (c) Molecular flow is normal in high-vacuum systems.
- (d) Between laminar and molecular flow a transition of flow occurs.

4. Conductance of long round pipe; laminar flow.

$$C = 0.246 \frac{d^4}{L} \bar{P} \quad (6)$$

Flow is laminar if $\bar{P} d > 215$; pipe is long if $L \geq 2d$. Otherwise, treat as a short pipe.

5. Conductance of short round pipe; laminar flow.

$$C = 0.246 \frac{d^4 \bar{P}}{L + d/4} \quad (7)$$

(This equation is approximate, based on experiment.)

6. Conductance of long noncircular duct; laminar flow. Treat as circular pipe of equivalent cross-sectional area, or treat as though flow were molecular, realizing that conductance is actually higher than indicated by this treatment.

7. Conductance of aperture; laminar flow.

$$C = 82A \quad (8)$$

if $P_2/P_1 \leq 0.7$; conductance less for higher ratios.

8. Conductance of long round pipe; molecular flow

$$C = 6.54 \frac{d^3}{L} \quad (9)$$

Flow is molecular if $\bar{P} d < 7$; pipe is long if $L \geq d$. Otherwise, treat as short pipe.

9. Conductance of short round pipe; molecular flow

$$C = \frac{75A}{1 + 9L/d} \quad (10)$$

That is,

$$\frac{1}{C \text{ short pipe}} = \frac{1}{C \text{ long pipe}} + \frac{1}{C \text{ entrance aperture}} \quad (11)$$

10. Conductance of long noncircular duct; molecular flow. Treat as round pipe in which

$$d^3 = \frac{16A^2}{\pi O} \quad (12)$$

11. Conductance of short noncircular duct; molecular flow. Treat as round pipe of equivalent diameter (Eq. 10). Note: for ducts of long, narrow rectangular section ($e \times w$) the conductance is greater than that given by Eqs. 10 and 11 by the following factors:

$$e/w = 3, k = 1.20; \quad e/w = 5, k = 1.30; \quad e/w = 8, k = 1.40$$

12. Conductance of aperture, molecular flow.

$$C = 75A \quad (13)$$

13. Pumping speed of a cold trap for water vapor.

$$S_T = 100 A' \quad (14)$$

14. Conductance of long round pipe; transition flow.

$$C = 0.246 \frac{d^4}{L} \bar{P} + 5.4 \frac{d^3}{L} \quad (15)$$

if $215 > d \bar{P} > 7$.

15. Effective length of elbow; all types of flow.

$$L_{\text{eff}} = L_{\text{axial}} + K$$

$$0 < K < d$$

16. Rate of evacuation of a system.

$$P_2 - P_0 = (P_1 - P_0) \exp \left(-\frac{S}{V} t \right) \quad (16)$$

$$t = \frac{V}{S} \ln \frac{P_1 - P_0}{P_2 - P_0} \quad (17)$$

If $P_2 = \frac{P_1}{2}$ and $P_2 \gg P_0$

$$\text{Time to reduce pressure to } \frac{P_1}{2} = 0.7 \frac{V}{S} \quad (18)$$

17. For an element of conductance C:

(a) The quantity flow rate, Q (μ -liters/sec), is

$$Q = C (P_2 - P_1) \quad (19)$$

(b) The volumetric flow rate B (liters/sec) at pressure P , is

$$B = C \frac{P_1 - P_2}{P} \quad (20)$$

(c) The mass flow rate, G (g/sec), is

$$G = C \rho (P_2 - P_1) \quad (21)$$

18. Conversion of units.

(a) $1 \mu \text{ Hg} = 10^{-3} \text{ mm Hg} = 1.33 \text{ dynes/sq cm} = 1.934 \times 10^{-5} \text{ lb/sq in.}$

(b) $1 \text{ cc-atm} = 760 \mu\text{-liters}$

(c) $1 \text{ cfm} = 0.47 \text{ liters/sec}$

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2. C. E. Normand, Gaseous Flow, CEW-TEC Report D-4.440.8, Aug. 27, 1946.

Appendix B

SUPPLIERS OF VACUUM EQUIPMENT AND MATERIALS

Supplier Index

1. Allis-Chalmers Mfg. Company (A-C),* Milwaukee, Wis.; indicating and signaling Pirani gauge, thermal vacuum switch.
2. American Brass Company (Am-Br), Waterbury, Conn.; flexible metal hose.
3. Anchor Packing Company (AP), 401 N. Broad St., Philadelphia, Pa.; rubber (vacuum-gasket material).
4. Armstrong Company (Arm.), Lancaster, Pa.; cork Neoprene (vacuum-gasket material).
5. Associated Rubber Products Company (ARP), Quakertown, Pa.; rubber (vacuum-gasket material).
6. Beach-Russ Company (B-R), 50 Church St., New York, N. Y.; mechanical vacuum pumps.
7. James G. Biddle Company (Biddle), 1211-1213 Arch St., Philadelphia, Pa.; Apiezon products (oils, greases, waxes).
8. Chapman Valve Company (Chapman), Indian Orchard, Mass.; vacuum valves, 2 in. to 30 in., custom built.
9. Chicago Metal Hose Company (Ch-Met. Hose), Maywood, Ill.; flexible metal hose, metal bellows.
10. Consolidated Engineering Corporation (Cons. Eng.), 620 N. Lake Ave., Pasadena, Calif.; mass spectrometer.
11. Continental Rubber Works (CRW), 1902 Liberty St., Erie, Pa.; rubber (vacuum-gasket material).
12. Distillation Products, Inc. (D.P.I.), 755 Ridge Road West, Rochester, N. Y.; oil diffusion pumps (glass), oil diffusion pumps (metal, 5 to 7,000 liters/sec), Pirani gauges, Philips gauges, ionization-gauge tubes and power supplies, diffusion-pump fluids, vacuum greases and waxes, vacuum-coating units, packaged vacuum systems.

*Abbreviations following firm names are used in Material Index.

13. Dow Corning Corporation (Dow Corning), Midland, Mich.; silicone diffusion-pump oils, silicone vacuum grease.
14. E. I. du Pont de Nemours Company (DPN), 626 Schuyler Ave., Arlington, N. J.; Teflon (vacuum-gasket material).
15. Eitel-McCullough, Inc. (EIMAC), San Bruno, Calif.; diffusion pumps, vacuum valves, diffusion-pump oil, ionization-gauge tubes, vacuum switches.
16. Fisher and Porter Company (F & P), Hatboro, Pa.; rotameters.
17. Fulton Sylphon Company (Fulton), Knoxville, Tenn.; vacuum valves (packless), sylphon bellows.
18. Garlock Packing Company (GP), 402 Main St., Palmyra, N. Y.; rubber (vacuum-gasket material).
19. General Electric Company (G.E.), Schenectady, N. Y.; ionization-gauge tubes and power supplies, thermocouple vacuum gauges, helium leak detectors.
20. Goodrich Rubber Company (Goodrich), 500 S. Main St., Akron, Ohio; rubber (vacuum-gasket material).
21. Goodyear Rubber Company (Goodyear), 2400 Third St., San Francisco, Calif.; (vacuum-gasket material).
22. Hilliard Corporation (Hilco), Elmira, N. Y.; Hilco oil purifiers.
23. Imperial Brass Manufacturing Company (Imp. Brass), 1200 W. Harrison St., Chicago, Ill.; needle valves, tubing fittings.
24. Johnson Rubber Company (JRC), 101 Vine St., Middlefield, Ohio; rubber (vacuum-gasket material).
25. Kerotest Manufacturing Company (Kerotest), Pittsburgh, Pa.; vacuum valves (diaphragm type), tubing fittings.
26. Kinney Manufacturing Company (Kinney), Washington St., Boston, Mass.; mechanical vacuum pumps, vacuum valves.
27. Linde Air Products Company (LAP), Kittaning, Pa.; Essington, Pa.; East Chicago, Ill.; liquid nitrogen (refrigerants).
28. Liquid Carbonic Company (LC), Memphis, Tenn.; Atlanta, Ga.; dry ice (refrigerants).
29. Litton Engineering Laboratories (Litton), Redwood City, Calif.; oil diffusion pumps (20 to 1000 liters/sec), diffusion-pump oil, packaged vacuum system.
30. Mathiesen Alkali Works (MA), Saltville, Va.; dry ice (refrigerants).
31. National Research Corporation (N.R.C.), 100 Brookline Ave., Boston, Mass.; oil diffusion pumps (5 to 1,250 liters/sec), refrigerated vapors traps, vacuum valves, vacuum seals, thermocouple gauges, ionization-gauge tubes, ionization-gauge power supplies, Alphasatron gauge, evaporation units, packaged vacuum systems, helium leak detectors (G.E.).

32. National Union Radio Corporation (N.U.), Newark, N. J.; ionization-gauge tubes.
33. Precision Scientific Company (Precision), 1736-54 Springfield Ave., Chicago, Ill.; Televac recording thermocouple and ionization gauge, vacuum leaks (fixed and variable).
34. Pure Carbonic Company (PC), Wyandotte, Mich.; dry ice (refrigerants).
35. Scientific Supply Companies (Sci. Sup. Cos.), miscellaneous supplies: Burrell Technical Supply Company, Pittsburgh, Pa.; Central Scientific Company, Chicago, Ill.; Eberbach and Son, Inc., Ann Arbor, Mich.; Fisher Scientific Company, New York, N. Y.; Welch Manufacturing Company, Chicago, Ill.; Will Corporation, Rochester, N. Y.
36. F. J. Stokes Machine Company (Stokes), Philadelphia, Pa.; McLeod gauges (tilting type), McLeod gauges (recording type), mechanical vacuum pumps, vacuum-processing equipment.
37. Arthur H. Thomas Co. (Thomas), Philadelphia, Pa.; Lubriseal stop-cock grease.
38. Universal X-Ray Products Inc. (Universal), 1800-1802 Francisco Ave., Chicago, Ill.; thermocouple gauges, Philips ionization gauges, mercury diffusion pumps, vacuum glassware.
39. Vacuum Specialties Laboratory (Vac. Spec. Lab.), 530 S. Greenwood Ave., Pasadena, Calif.; Knudsen-type vacuum gauges.
40. Western Electric Company (W.E.), ionization-gauge tubes.
41. Westinghouse Air Brake Company (W.A.B.C.), Wilmerding, Pa.; rubber (vacuum-gasket material).
42. Westinghouse Electric Manufacturing Company (W.E.M. Co.), East Pittsburgh, Pa.; oil diffusion pumps, McLeod gauges, vacuum valves, ionization-gauge tubes, thermocouple gauges, helium leak detectors.

Material Index*

1. Mechanical vacuum pumps: B-R, Kinney, Stokes, Sci. Sup. Cos.
2. Oil diffusion pumps: D.P.I., EIMAC, Litton, N.R.C., W.E.M. Co., Sci. Sup. Cos.
3. Mercury diffusion pumps: D.P.I., Litton, N.R.C., Sci. Sup. Cos.
4. Vacuum valves: Chapman, EIMAC, Fulton, Kerotest, Kinney, N.R.C., W.E.M. Co.
5. Needle valves and vacuum leaks: Crane Co., Imp. Brass, Precision, Sci. Sup. Cos.

*See Supplier Index for firm names.

6. Flexible hose and metal bellows: Am-Br, Ch-Met. Hose, Fulton.
7. Tubing fittings: Am-Br, Imp. Brass, Kerotest.
8. Vacuum seals: B-G Spark Plug Co., N.R.C.
9. Vapor traps: N.R.C., Sci. Sup. Cos.
10. Vacuum-processing equipment (driers, evaporators, extractors, stills): D.P.I., Litton, N.R.C., Stokes.
11. Packaged vacuum systems: D.P.I., Litton, N.R.C., Sci. Sup. Cos.
12. McLeod gauges: Stokes, W.E.M. Co., Sci. Sup. Cos.
13. Pirani gauges: A-C, D.P.I.
14. Thermocouple gauges: D.P.I., N.R.C., Precision, Universal, W.E.M. Co., Sci. Sup. Cos.
15. Philips gauges: D.P.I., Universal, W.E.M. Co.
16. Knudsen gauges: Vac. Spec. Lab.
17. Ionization-gauge tubes: D.P.I., EIMAC, N.R.C., N.U., Precision, W.E., W.E.M. Co., Sci. Sup. Cos.
18. Ionization-gauge power supplies: D.P.I., N.R.C., Precision, G.E.
19. Mass spectrometers and leak detectors: Cons. Eng., G.E., W.E.M. Co., N.R.C. (G.E.).
20. Rotameters: F & P.
21. Vacuum switches: A-C, EIMAC, Sci. Sup. Cos.
22. Oil purifiers: Hilco.
23. Mechanical-pump oil: Kinney, Stokes, Sci. Sup. Cos.
24. Diffusion-pump fluid: Biddle, D.P.I., Dow Corning, EIMAC, Litton, N.R.C.
25. Vacuum greases, waxes, etc.: Biddle, D.P.I., Dow Corning, N.R.C., Thomas, Sci. Sup. Cos. (Glyptal - G.E.).
26. Cork Neoprene (vacuum-gasket material): Arm.
27. Rubber (vacuum-gasket material): AP, ARP, CRW, GP, Goodrich, Goodyear, JRC, W.A.B.C.
28. Teflon (vacuum-gasket material): DPN.
29. Dry ice (refrigerants): MA, PC.
30. Liquid nitrogen (refrigerants): LAP.

Appendix C

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