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BETTER VACUUM BY REMOVAL OF DIFFUSION-PUMP-OIL CONTAMINANTS

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16. Abstract

The complex problem of why large space simulation chambers do not realize true ultimate vacuum was investigated. Some contaminating factors affecting diffusion pump performance were identified and explained, and some advances in vacuum distillation-fractionation technology were achieved which resulted in a two-decade-or-more lower ultimate pressure. Data are presented to show the overall or individual contaminating effects of commonly used phthalate ester plasticizers of 390 to 530 molecular weight on diffusion pump performance. Methods for removing contaminants from diffusion pump silicone oil during operation and for reclaiming contaminated oil by high-vacuum molecular distillation are described. Conceptual self-cleansing designs and operating procedures are proposed for modifying large diffusion pumps into high-efficiency distillation devices. The potential exists for application of these technological advancements to other disciplines, such as medicine, biomedical materials, metallurgy, refining, and chemical (diffusion-enrichment) processing.

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SUMMARY

Contamination problems associated with space simulation test programs, their effect on diffusion pump performance, and the rapid deterioration of silicone diffusion pump fluids were studied. The performance of identically contaminated oils in diffusion pumps with or without self-purification capabilities is compared.

A series of experimental scale-model glass diffusion pump vacuum systems were evolved to investigate certain physical properties of evaporative surface behavior and its effect on ultimate realizable vacuum. Innovations in the design of boiler fluid separation-purification devices were made which successfully removed the heavy-end fractions from the evaporative surface by skimming-bleeding methods and the light-end fractions by vacuum distilling - bleeding action at temperatures of approximately 410 K (280° F) and pressures of approximately 3×10^{-3} to 5×10^{-3} torr in the foreline. These diffusion pump innovations lowered the ultimate achievable pressure by more than 2 decades.

Methods are specified for modifying all vapor-operated diffusion pumps into self-cleansing devices such that both heavy- and light-end impurities absorbed into the motive fluid can be removed during pump operation without changing pump oil.

Documentation reveals the progressive improvement in molecular release rate corresponding with the increase in diffusion pump performance and resultant lower ultimate pressure. Other significant results revealed were as follows:

1. Contamination in the pump fluid limited the efficiency of diffusion pumps by 100 to 1000 times in terms of ultimate vacuum obtained.

2. Contaminants that are commonly found in large vacuum chambers and that adversely affect the performance of silicone oil in diffusion pumps were identified as phthalate ester plasticizers. These plasticizers are used in cable insulation jackets, alkyd paints, electronic components, and so forth.

3. Contaminated silicone diffusion pump fluid such as DC-705 can be successfully reprocessed by a triple-cut (two light end and one heavy end), high-vacuum, centrifugal molecular distillation method.

4. The effects of specific phthalate plasticizer contaminants or mixtures of various contaminants on diffusion pump performance were measured.

5. Self-cleansing modifications to diffusion pumps consisted of innovations to the foreline, the boiler, and the barrel (or housing).

6. Potential interdisciplinary applications of this technology exist for refining processes, metallurgy, biomedical devices, diffusion-enrichment, chemical processing and reprocessing distillations, and incorporation into qualitative and quantitative instrumentation devices.

INTRODUCTION

Large space simulation chambers are not presently capable of realizing their true ultimate vacuum because contamination limits the efficiency of their diffusion pumps. Present diffusion pumps are not totally suitable for self-purification, that is, they are not capable of removing or preventing contamination of the pump fluid. A rapid degradation in pump performance occurs when the pump motive fluid is exposed to the sources of outgassing contaminants that are associated with space simulation test programs. Therefore, a study of the fundamental causes limiting performance was initiated, with the following objectives:

(1) To identify contaminating factors that are commonly found in large vacuum chambers and that adversely affect silicone oil diffusion pump performance

(2) To measure the effect of various contaminants on the performance of diffusion pumps

(3) To develop means of removing contaminants from oil by economically reprocessing contaminated diffusion pump oil

(4) To devise means of removing contaminants from the oil or other motive fluid during diffusion pump operation by making the pumps totally self-cleansing

(5) To determine, define, and explain the limiting parameters related to decreasing pump performance

The motion-picture supplement C-280 is a comprehensive, dynamic documentary of this research chronology and the associated vacuum-distillation technology and is available on loan. See the request card at the end of this report.

In this study, valuable assistance was given by W. R. Carman and L. Underhill of NASA Kennedy Space Center; F. Gross and J. Colony of NASA Goddard Space Flight Center; V. R. Lalli of NASA Lewis Research Center; R. K. Lohwater of Bendix Corporation; K. Hickman of Rochester Institute of Technology; and A. R. Huntress of Dow Corning Corporation.

This investigation was conducted in the U. S. customary system of units. Conversion to the International System of Units (SI) was done for reporting purposes only.

BACKGROUND THEORY AND DEVELOPMENT

Overall Problem Background

During initial Brayton power system testing in 1969 (ref. 1) in the 22 700-cubic-meter (800 000-cu-ft) Space Power Facility (SPF) test chamber, accumulations of condensable volatiles were observed on the 30. 5-meter- (100-ft-) diameter floor (fig. 1(a)). The NASA Kennedy Space Center (KSC) Analytical Laboratory analyzed these condensables by a combination of column and gas chromatography and infrared, emission, and mass spectroscopy. Samples were found to consist primarily of phthalate esters, an aromatic amine, and smaller amounts of a hydrocarbon oil and silicone oil.

In 1970 and 1971 during Skylab payload shroud jettison tests (ref. 2) at a simulated altitude of 91.5 km (300 000 ft, or 3.3×10^{-4} torr), the DC-705 diffusion pump oil became contaminated because of the large outgassing load (fig. 1(b)). A complete oil change, costing $30 000, was implemented. The magnitude of the diffusion-pump-oil contamination problem was revealed by the 1.5-decade loss in performance of new DC-705 after 25 operational hours of Skylab shroud testing.

In order to thoroughly understand the outgassing contamination problem associated with large space simulation chambers, a materials inventory search was undertaken to establish which compounds of those presently being used were the most volatile. Thirty-nine major materials, in quantities ranging from 1580 to 4.54 kg (3500 to 10 lbm), were used in conjunction with the Brayton and Skylab test programs. Those materials that are formulated with the plasticizers found in the analyses of eight DC-705 oil samples are listed and described in the section RESULTS AND DISCUSSION.

Operation of Diffusion Pumps

A diffusion pump works on the principle that a liquid having relatively heavy molecules is vaporized in the pump boiler by raising its temperature in an environment of reduced pressure. The vapor rises up through the jet assembly to be emitted from nozzles or jets in a stream away from the region to be evacuated toward the high-pressure side of the pump in the direction of a mechanical forepump. The accelerated molecules of the vapor stream collide with the gas molecules entering through the pump intake port, forcing and compressing them toward the higher pressure side and the mechanical forepump. In this way a differential in molecular density, or a pressure difference, is created between the system side to be evacuated and the mechanical forepump. The vapors are recondensed on the cool barrel wall of the diffusion pump, where the liquid condensate is permitted to return to the bottom of the pump to be reheated and vaporized. Typical working fluids employed in diffusion pumps are mercury; organic fluids (2-ethylhexyl phthalate and 2-ethylhexyl sebacate), available as Octoil and Octoil-S from Consolidated Vacuum Corporation (CVC); pentaphenyl ether, available as Convalex-10 from CVC (ref. 3) or as Santovac from Monsanto; hydrocarbon oils such as Convoil-10, Convoil-20, and Apiezon C; and silicone oils such as DC-705 (pentaphenyltrimethyltrisiloxane) and DC-704 (tetramethyltetraphenyltrisiloxane) made by Dow Corning Corporation, whose performance was initially established in reference 4.

The present state of the art of typical large diffusion pumps is shown in figure 2. The pump inlet is at the top of the barrel (or housing) (ref. 5), and the discharge side is provided with a pipe, one end of which is eventually connected to a mechanical forepump. The interior of the pump includes a suitable nozzle assembly (normally called the "Christmas tree" jet assembly). The level of oil in the bottom of the diffusion pump boiler is indicated by a dashed line. The SPF has thirty-two 122-cm- (48-in.-) diameter diffusion pumps such as these, made by the Consolidated Vacuum Corporation, each having a rated pumping speed of 98 cu m/sec (98 000 liters/sec).

In the diffusion pump boiler (fig. 3) the motive fluid is normally heated by a heating element in an annular continuous metallic channel formed integrally with the boiler bottom (ref. 5). The heated channels, varying in number with manufacturer, with or without heat distribution fins, are concentrically arranged on the boiler bottom and have their axes in coincidence with the vertical axes of the boiler dome and the pump housing. The fluid level in the boiler is established and maintained through a pipe with one opening per channel.

Neither the foregoing diffusion pump design (ref. 5) nor other designs (e.g., refs. 6 to 17) have an effective means for purifying, separating, and removing contaminants in the pump fluid. These contaminants limit the overall vaporization rate and reduce pumping efficiencies. In 1935, Hickman established the state of the art with his small, glass, diffusion pump designs (ref. 18). Some of his small, slow-speed (0.025 cu m/sec; 25 liters/sec), glass, tandem laboratory diffusion pumps were successful in isolating some impurities within internal compartments. The impurity constituents were identified as light-end components (A to B) and a heavy-end component (Z). Current large metal diffusion pumps with speeds of 0.5 to 100 cu m/sec (500 to 100 000 liters/sec) do not employ any of Hickman's internal contaminant-collection innovations or any other successful impurity-separation devices. Therefore, the purity of the motive fluid, its vaporization rate, and the overall efficiency of these pumps are decreased 100 to 1000 times as the motive fluid absorbs impurities.

Background Information Relative to Contamination of Vapor Pumps

Diffusion pumps, commonly called inverted condensation pumps or vapor pumps, are dynamic equilibrium systems. These pumps depend on the respective vapor pressure - molecular weight - temperature interrelations associated with a mixture of working fluid constituents. As such, diffusion pumps are simply stills.

For years, it has been recognized that clean pump fluids generate the best ultimate vacuums (refs. 19 to 23). The previously mentioned common motive fluids when used in diffusion pumps to evacuate thermal-vacuum-radiation chambers tend to be subject, in varying degrees, to existing contamination from within the evacuated chambers. Commonly used auxiliary refrigerated traps are not entirely effective in keeping these contaminants from being absorbed and combined into different fractions within the diffusion pump motive fluid. Huntress (refs. 21 and 22) does state that less than 1 part per million of certain types of volatile contaminants was shown to alter seriously the effective vapor pressure of the working fluid. Removing contaminants has not been easy. Cryotrapping them on baffles is not a completely corrective measure since saturation eventually occurs, causing vacuum system pressure spikes and contamination of the test article. Also upon baffle warmup, condensed contaminants may drip back into the pump fluid.

One factor contributing to contamination is the quiet evaporative surface behavior designed into current large diffusion pumps. Since diffusion pump boiler eruptions, sometimes called burps, have been considered undesirable from a backstreaming standpoint, it has been the practice to minimize or eliminate vigorous boiling evaporative surfaces by design (ref. 23). Also diffusion pump manufacturers recognized the need for stability in vaporization rates and pump speed and for the elimination of pressure fluctuations caused by eruptive boiling (refs. 5 and 6). They therefore have designed pump boilers that create a quiet evaporative surface behavior.

Other factors related to contamination are large outgassing test hardware in large space simulation chambers which are difficult and expensive to clean, diffusion pump oil too costly to be periodically replaced or repeatedly reprocessed when quickly contaminated, and elaborate baffle systems too expensive to purchase and/or operate. A logical course of action was to investigate methods to increase the self-cleansing, self-purification - separation capabilities of our present diffusion pumps (stills).

Background Information Relative to Distillation

The art of distillation was practiced before the Christian era by the Egyptian temple priets. However, molecular distillation is relatively new. In the early 1950's, Hickman, while working with pot stills, found that when liquids are permitted to evaporate rapidly without restriction into a high-vacuum environment their surfaces often separate into two zones of widely different appearance and properties (refs. 19, 20, and 24). One zone, which is in rapid self-motion, is called "WORKING"; and the other, which is quiescent and photographically highly reflective, is called "TORPID." Figures 4(a) and (b) illustrate these two evaporative surface behavior patterns in a glass scale-model diffusion pump. An evaporative surface exhibiting a dual working-torpid behavior pattern or patterns is called "SCHIZOID" and is illustrated by figures 4(c) and (d) and photograph 15 (fig. 16(a)). A height difference of approximately 1 mm exists between the upper torpid area (location of little vaporization or molecular release) and the lower working area (location of immense vaporization or molecular release). Torpidity, a floatation phenomenon, is believed to be caused by traces of impurities that block the surface and prevent rapid evaporation.

Development Approach

The self-cleansing, one-millionth-scale, glass model of the SPF vacuum system was designed to meet the following objectives:

(1) To understand the fundamental phenomenon of torpidity and its effect on achieving ultimate vacuum or maximum pump speed

(2) To determine methods and operating parameters for preventing torpidity or for removing a torpid evaporative surface condition in order to generate a 100-percent-working surface

(3) To determine the percentage of contaminants that induces torpidity and/or its related limiting effects on ultimate vacuum

(4) To determine methods and operating parameters for removing light-end contaminants in the foreline

(5) To determine methods and operating parameters for removing heavy-end contaminants from the boiler fluid

This vacuum system consisted of a three-jet glass diffusion pump with a boiler approximately the same size as that used in all comparison tests of oil performance.

Supporting Keys to Self-Cleansing Design

The self-cleansing design arrived at in this study evolved from the concepts of torpid, schizoid, and working evaporative surfaces. Torpidity as a surface phenomenon is subject to removal by surface skimming, resulting in a schizoid evaporative surface.

Multiple groups of contaminants were shown to exist by spectroscopy analyses. Heavy-end contaminants (grouping at approximately 700 MW) and light ends (peaking at approximately 300 MW) established the necessity for a triple-cut molecular distillation process. The quantity of light-end contaminants is nearly two to three times the quantity of heavy-end contaminants.

The final key to the self-cleansing design was found within normal vacuum fractionation-distillation ranges (refs. 25 and 26). Hickman's early condensation pump designs all use the alembic concept of liquid-vapor equilibrium in the vertical stage. Acute observation revealed that, when the alembics fill up, the heavier volatile constituents overflow back into the pump boiler and are never fully discharged.

APPARATUS AND PROCEDURE

Analytical Apparatus

The following equipment was used to study the material outgassing contamination problem:

(1) Computerized Digilab FTS-20 infrared Fourier transform spectrophotometer and/or Perkins-Elmer model 621 infrared spectrophotometer (at Kennedy Space Center (KSC))
(2) Hitachi model RMU 6L mass spectrometer and Perkins-Elmer model 900 gas chromatograph linked with a Varian model 620-i data system (at Goddard Space Flight Center (GSFC))
(3) A Q-250 residual gas analyzer (at Lewis Research Center)
(4) Individual Water Associates model 502 liquid chromatographs (at KSC and GSFC)
(5) Gas chromatography mass spectroscopy units (at KSC, GSFC, and Lewis)
(6) AGA Corporation model 661 thermovision (at Lewis)
(7) Quartz crystal microbalance (at GSFC and Lewis)
(8) Environment control-instrumentation systems (at Lewis)
(9) One-millionth-scale glass models of the SPF vacuum systems (at Lewis): G-4, modified G-4, first- and second-generation self-cleansing pump designs (fig. 5)

Standard Diffusion Pump Oil Performance Test Setup

A single-stage glass diffusion pump (designated a G-4) having a rated pump speed of 0.004 cu m/sec (4 liters/sec) was used for all oil performance comparison tests (fig. 6). The G-4 vapor pump has long been used by the pump fluid and vacuum system manufacturers for comparing performance of various pump fluids. The prime reasons for using glass were the control over material contaminating factors and the benefit of visually observing the operational conditions within the vapor pump.

Molecular Distillation-Reclamation Process and Equipment

Conventional methods for reclaiming contaminated pump oil are relatively ineffective and/or expensive. A new method was developed for reclaiming contaminated vacuum diffusion pump oil by high-vacuum molecular distillation processes (ref. 27). A high-vacuum centrifugal molecular still is used to centrifugally separate volatile contaminant and component compounds from the oil at their individual respective evaporation temperatures. Preheated distilland feedstock is metered onto a heated rotating evaporator disk at a rate of 4.35 g/sec (35 lbm/hr). The first cut is performed at 423 K (302° F) and the second cut is performed at 453 K (356° F) to achieve 10 and 7 percent distillation separations, respectively, at approximately 35×10^{-3}-torr pressure. The light-end contaminant vapors are condensed on the cool inner surface of the bell jar and recovered as a distillate to be discarded. The pure oil and heavy-end compounds are not vaporized and are spun off the rotating disk by centrifugal force, caught in a water-cooled gutter, and collected as a residue. Final-cut distillation is performed in a similar manner except that the pure oil is vaporized at 485 to 496 K (413° to 433° F), condensed, and recovered as a distillate in a 90 percent cut, with about 10 percent of the heavy-end contaminants being separated as a residue. See figure 7 for reprocessing setup and figure 8 for the profile of the associated contamination removal process.

The acceptance criterion of molecular distilled - reprocessed DC-705 is attainment of a 5×10^{-6}-torr ultimate vacuum within 4 hours using the G-4 pump-oil-performance setup. Failure to meet the acceptance criterion indicates the residual existence of additional light-end contaminants, which necessitates another light-end cut distillation but at a slightly higher temperature of 463 to 470 K (374° to 386° F), see figure 8. A high-vacuum, molecular, wiped-film type of still may also be used successfully. For additional theory relative to molecular distillation, see Burrows (ref. 28) and Watt (ref. 29).

The closer the molecular weights of the contaminated mixture, the less efficient will be the separation of various fractions (or purification). This results in the need for successive re-runs of the distillate to meet the acceptance criterion, such as the two successive light-end distillation cuts mentioned in the preceding paragraph.

Also experience reveals that there is no predictable temperature at which distillation will occur; vaporization and condensation begin whenever a sufficient temperature differential exists between the evaporator and the condenser of the molecular still. Therefore, an infinite number of possible sets of operating condition exist for every contaminated oil or feed material. These parameters vary as a function of the contaminating environment or with variations in the specific feed material. To illustrate these complex variables further, refer to figure 9, which illustrates the relation between molecular weight and distillation temperature at a constant slow (5678 cu cm/hr, or 1.5 gal/hr) feed rate for all materials being distilled (ref. 30).

Furthermore, it is possible to anticipate the degree of separation of a mixture that will be achieved by high-vacuum distillation by using the relation of $P\sqrt{M}$ of the constituents, where P is the partial pressure and M is the molecular weight (refs. 28 to 30).

More extensive information on the reprocessing setup, including equipment description, molecular still operating philosophy, distillation processing, and G-4 solvent cleaning preparations, plus operating procedures may be obtained from the Technology Utilization Office at NASA Lewis Research Center (refer to ref. 27).

Self-Cleansing, One-Millionth-Scale, Glass Model of SPF Vacuum System

A three-jet glass diffusion pump, having approximately the same size boiler and resultant evaporative surface as the G-4 single-stage diffusion pump, was constructed to study the effects of inserting phthalate plasticizers and other contaminants directly into the pump fluid or into the chamber during pump operation (fig. 10(a)). The removal and identification of these volatile contaminating constituents are required in order to prevent the rapid deterioration of the pump fluid and the resultant reduced pump efficiency. See figure 10(b) for self-cleansing (purification-separation) design details.

The self-cleansing, one-millionth-scale, model apparatus generates ultrapure pump fluid with resultant lower ultimate chamber pressures. Follow-on foreline vacuum distillation designs have been fabricated which should further improve the separation capability of contaminants in pump fluid (fig. 10(c)).

The design solution unfolded as a merger of three interrelated concepts: (1) surface skimming of heavy-end contaminants; (2) periodic draining of different light-end fractions until the temperature of the foreline when it is heated externally stabilizes at a maximum of about 410 K (280° F), and (3) obtaining a 100-percent-working evaporative surface as indicated by a 5 to 7 K (9° to 13° F) boiler vapor-liquid temperature difference. Optimum performance was achieved with a boiler fluid depth of 5 mm ± 1 mm. Any one of the three concepts by itself or paired with another will not allow achievement of the optimum pump efficiency possible with a continuous 100-percent-working evaporative surface. Experience showed that the pump boiler fluid improved to a purity of 99.8 percent or better when the evaporative surface exhibited a continuous 100-percent-working condition. The critical point in the relation of the ratio of contaminant to pure liquid and the lowest realizable ultimate pressure is still uncertain. This uncertainty is primarily due to the existing noise levels in the spectroscopy analysis system.

The overall solution resolves into controlling the multicontamination problem while stabilizing diffusion pump performance deterioration by recognizing the following interrelated concepts:

(1) Both light- and heavy-end contaminants exist in all pump fluid simultaneously.

(2) Torpidity affects the molecular release rate from an evaporative surface by acting as a surface-tension grid network, similar to a screen with progressively smaller openings, and choking off holes in the evaporative surface which emit vapor (i. e., allow molecular release).

(3) Contaminants are not being sufficiently removed by present diffusion pumps. Thus, motive fluids contain sufficient impurities to prevent significant improvements in pump performance.

The conceptual glass model self-cleansing diffusion pump referred to in figure 10, incorporates, in a thermally vacuum-controlled manner, two molecular stills functioning as independent fractionation-purification-separation devices. One still (located in the foreline) performs light-end multimixture distillations, while the other (located in the diffusion pump boiler) provides heavy-end multimixture distillations occurring at the evaporative surface of the motive fluid. These innovations generate more efficient (sharper) separation of contaminant constituent impurities, by a factor of 50 times, than that which was performed by the triple-cut high-vacuum centrifugal distillation process previously described.

Foreline purification. - In the forelines of self-cleansing pumps (fig. 10), a series of progressive distillation processes occurs at a temperature between 310 and 410 K (100° and 280° F) or greater and at a pressure of 3×10^{-3} to 5×10^{-3} torr. The multimixture of soluble liquids and vapors is carried along by the pump throughflow to the foreline molecular distillation device. This device consists of two or more alembics with condensate collection plates that are either empty or filled with wire-wound coil packing made of stainless steel or other material to increase the liquid-vapor interface surface area. Separation of different molecular weight constituents occurs by evaporation and condensation at a constant distillation equilibrium, whereby the condensed part collected in the plates becomes richer in the most volatile constituents, commonly called light ends. This condensate, high in percentage of contaminant, is periodically drained through tubes into a vessel of slightly lower pressure while the diffusion pump is operating. The high-separation-purification foreline configuration of figure 10(c) contains baffles positioned over each alembic to further increase the vapor-condensation surface area for sharper molecular distillation efficiency. The baffle provides additional light-end enrichment, thereby increasing the percentage of contaminant that is removed as a condensate and isolated in the lower-pressure vessel.

If this highly volatile condensate is not removed and thus flows back into the diffusion pump boiler, chemically termed reflux, the volatile constituents will be revaporized in the diffusion pump boiler, along with the pure fluid. All constituent vapors then move up the Christmas-tree jet assembly and are discharged out the respective jet nozzles to be condensed on the cooler wall of the diffusion pump barrel. Both pure and light-end-contaminated fluid molecules follow similar paths. The primary distinction between the two types of vapor or condensate is the time each dwells on the surface of the diffusion pump barrel wall. For a specific temperature and pressure, more-volatile molecules tend to break the polar and dispersion bond forces and backstream up into the chamber. This process is also known chemically as reverse fractionation. Other less-volatile constituents and pure motive fluid condensed on the walls of the diffusion pump barrel are pulled down the walls by gravity as a reflux into the foreline inlet area of the diffusion pump, where the molecular throughflow or throughput carries or pushes these volatile constituents back into the foreline molecular still.

The purification-separation process becomes more clearly understood by considering that each volatile condensate mixture captured on each respective foreline still plate has a different molecular weight and/or relative percentage. As these contaminating constituents are progressively removed through tubes and isolated in the lower-pressure vessel, the efficiency of the pump increases, creating a stronger molecular throughflow and slightly increasing the temperature in the foreline still. This slight increase in temperature allows the next group of less-volatile contaminants of slightly greater molecular weight to be separated by evaporization and condensation under a new set of distillation equilibrium conditions at a specific pressure. As foreline pressure is reduced slightly because of the increased pump efficiency and the general reduction in existing volatile contaminants, another set of distillation parameters-conditions exists for the removal of the heavier light-end (less volatile) contaminants. These contaminating constituents have molecular weights between 300 and 500 (fig. 11). Thus, the removal of heavier light-end impurities becomes a progressive achievement based on the removal of the next lower group of impurities.

The first self-cleansing foreline distillation purification cut, performed with a typically contaminated motive fluid, removes primarily those impurities that are less than 300 molecular weight. This separation is comparable to the first light-end cut performed when reprocessing contaminated oil by high-vacuum centrifugal molecular distillation. However, some percentage of impurities greater than 300 molecular weight would also be removed, just as some percentage of impurities less than 300 molecular weight would remain. The first cut removes a wide range of impurities, because all three drain tubes are opened simultaneously, draining all collected condensate (10 cu m, note that 1 cu cm = 1 milliliter) in operating the second-generation diffusion pump scale model (fig. 10(b)). The second self-cleansing distillation cut removed condensate from the upper alembic, concentrating on impurities in the 250- to 400-molecular-weight range. Thus, overlapping sequentially numbered distillation cuts gradually depletes the total quantity of light-end contaminants, as illustrated by the family of curves in figure 11.

Each progressive foreline distillation cut separates sequentially heavier impurities. A corresponding increase in molecular release rate, density, consistency, and pump efficiency is achieved.

Surface skimming. - Surface skimming allows further improvement in diffusion pump performance to be achieved through the removal of concentrated heavy-end impurities from the boiler evaporative surface (fig. 10(b)). Convective heating circulation patterns within the motive fluid constantly force these heavier impurities to the top of the evaporative surface, similar to a scum layer. These heavy-end contaminants concentrate primarily between 600 to 800 molecular weight, as illustrated by figure 11. These impurities are removed by surface skimming. A drain tube and isolation valve in the boiler's side, located at the motive-fluid evaporative surface, enable efficient and quick removal of surface contaminants by sucking them into a vessel of lower pressure. The success of the skimming operation depends on the difference between the diffusion pump boiler pressure, usually at 0.1 to 1 torr, and the pressure of this lower-pressure vessel, usually at 1×10^{-3} to 3×10^{-3} torr.

The nature, type, and extent of contamination plus the physical characteristics of the evaporative surface determine the actual efficiency of any skimming operation and/or dictate the need for repeating such an operation. One skimming operation, or two at the most, is all that is necessary for a one-time purification, provided the sucking differential pressure is maintained at 100 to 1000 times. These evaporative surface skimming operations are usually less than 1 second in duration in order to prevent the unnecessary depletion of motive fluid. Insufficient boiler motive fluid, less than 50 percent normal quantity, necessitates replenishment to a 75 percent quantity. Because any makeup fluid contains more impurities than the self-cleansed motive fluid within the operating diffusion pump, an additional 0.1-second evaporative surface skimming operation and an upper foreline alembic condensate bleed draining may be required within an hour of any such replenishment.

Evacuating chambers which contain high outgassing materials will necessitate repeating these purification procedures about once per day for removal of light-end contaminants and every other day for removal of heavy-end contaminants. The type and quantity of outgassing load plus the nature of the vacuum environment being simulated affect the frequency of self-cleansing operations.

The proposed conceptual design and operating procedure for incorporating this purification-distillation technology into large diffusion pumps is described in a separate section following RESULTS AND DISCUSSION.

RESULTS AND DISCUSSION

Identifying Contaminating Factors

During the initial test programs at the SPF, accumulations of condensable volatiles were observed on the chamber floor. These samples were found to consist primarily of phthalate esters, a common plasticizer used by industry. Materials that are highly plasticized are used in cable insulation jackets, electronic components, alkyd paints, shock cords, power cables, instrumentation cables, termination boxes, and so forth. Common polyvinylchloride (PVC) cable formulations with 25 percent typical phthalate plasticizers represent the majority of the worst outgassing offenders. They continue to outgas (release volatile constituents) at nearly a constant rate even after being exposed to vacuum for over 3000 hours. The magnitude of this contamination source cannot be neglected, because clean thermal-vacuum conditions are required when environmentally testing manned or unmanned spacecraft.

Plastics in a vacuum environment are no real problem when the molecular weight of the polymer material is reasonably high and the polymer is free of lightweight components. However, polymers can degrade in vacuum by the breaking down of long-chain polymers into smaller more-volatile fragments (ref. 31). Plastics degrade in the form of chain rupture or scission at random points along the chain or by depropagation or an unzippering type of reaction which is the reverse of chain polymerization (ref. 32). Volatile contaminants then are formed which can outgas and contaminate the diffusion pump oil.

The overall plasticizer contamination problem is not unique to aerospace applications but is common to other disciplines as well. Phthalate esters, a common group of plasticizers used in some automotive and biomedical material polymer formulations, have also been found to leach out of basic plastic material into the surrounding air or fluid. In a typical automotive example, the vinyl interior exposed to a sunny, thermal, and confined environment will emit noticeable odor. These trapped volatile outgassing contaminants tend to condense on any cool surfaces, such as the glass windows, in the form of a foggy film. This film becomes visible to the naked eye when the condensed accumulation approaches 200 to 300 monolayers. However, in direct sunlight or headlight beams, significantly less contamination buildup can produce a mirror-like reflective surface on a windshield, thereby generating a driving safety hazard. In the typical biomedical example, phthalate esters have a poor hydrocarbon or fat resistance, thus being susceptible to leachout. This partially explains the presence of di-2-ethylhexyl phthalate (DEHP) in blood after it has been stored in PVC blood bags (ref. 33). For a comprehensive summary of recommended low outgassing polymeric materials, see reference 34. Possible medical utilization of the foregoing technology is available in an unpublished paper by this author, titled "Effect of Plasticizer in Body Fluid," which proposes potential methods for screening, determining, and studying material biocompatibility.

Table I lists some materials associated with the overall space simulation contamination problem. Both light- and heavy-end contaminants were found to exist within the basic pump fluid, whether new or used. The existence of multiple groups of contaminants was determined by spectroscopic analysis and molecular distillation reclamation of approximately 450 kg (1000 lbm) of 3000-hour-old DC-705 silicone oil normally having a molecular weight of 546.

Contaminating Compounds Affecting DC-705

Two basic methods were used in determining the degree to which materials are thermal-vacuum compatible.

(1) NASA Goddard Space Flight Center (GSFC) standardized outgassing tests were performed to determine the percentage of weight loss and the percentage of volatile condensable materials (VCM). A material sample was exposed to a 1×10^{-6}-torr pressure

at 398 K (257° F) for 24 hours or more (refs. 34 to 36 and the NASA Johnson Space Center IBM printout on the outgassing properties of Apollo nonmetallic materials (biannually updated)). Noncondensable (true gases) and hot vaporous condensable materials (VCM) which are deposited on a 296 K (73.4° F) surface make up the weight loss. Materials that qualify for long-term space missions must show a minimum of degradation (maximum stability) and reveal less than 1.0 percent total outgassing weight loss and less than 0.1 percent volatile condensables.

(2) Differential thermal analysis (DTA) was performed at NASA Kennedy Space Center (KSC). A similar sample was exposed to progressively higher temperatures in a 10^{-4}-torr environment while the decreasing weight was measured.

An inventory search of 99 percent of the SPF materials used for the Brayton and Skylab space simulation test programs revealed 39 different components. Only those materials that were found to be volatile compounds (extensive outgassing materials) are included with their respective analyses in table I(a). Comparing outgassing and DTA test results for typical volatile cable insulations (samples 5a and 6a and samples 5b and 6b) revealed a close correlation between the two methods. The DTA results show that volatile materials that were subjected to 3000 hours in a 10^{-5}- to 10^{-4}-torr vacuum had nearly the same weight loss (within 90 to 97 percent) as did those samples that were never exposed to vacuum.

For alkyd paint (samples 2a, 2c, 3a, and 4b), outgassing weight loss or VCM data revealed a greater difference in outgassing rates, 55 to 150 percent, for nearly 3000 hours difference in vacuum exposure time. Nearly the same outgassing rate (within 88 percent) existed for the relatively short difference in vacuum exposure of 600 hours.

The foregoing results confirm that polymer materials that are suitable for high-temperature service are also best for withstanding a high vacuum (ref. 31).

Identification of Worst Outgassing Materials and Compounds

Outgassing and DTA tests were conducted on Belden 8404 two-conductor and four-conductor shielded PVC-insulated instrumentation cable that contained 25 percent diisodecyl phthalate (DIDP) with a molecular weight of 446. This cable after being exposed to vacuum for 3000 hours in the SPF revealed a 13.9 percent total weight loss and 9.5 percent VCM (table I) with the standardized outgassing test. Another PVC formulation with 60 percent phthalate plasticizer (not shown), Belden wire per Mil-W-16878B, that was never exposed to vacuum exhibited a 15.4 percent weight loss and 10.42 percent VCM. (DTA tests of PVC revealed that hydrogen chloride is given off first (ref. 32).) Other gross offenders were other formulations of PVC or neoprene and alkyd oil paints. Independent KSC/GSFC chemical analyses and references 37 to 39 reveal the existence of phthalate esters, carbonaceous materials, aromatic amines, antioxidants or antiozonants, organic salts, and (to a lesser degree) nonphthalate-type ester resin in the material samples listed in table I(a).

The foregoing contaminants were also detected in analyses of various DC-705 oil samples listed in tables I(b) and (c).

Effect of Various Contaminants on Performance of Diffusion Pumps

Various aged-oil performance comparisons. - The G-4 single-stage glass diffusion pump system illustrated in figure 6 was primarily used as an oil performance-calibration device. The rapid deterioration of silicone pump fluid in an environment that is filled with high outgassing materials is illustrated by figure 12. New DC-705 in a typical G-4, 4-hour test consistently achieved an ultimate vacuum of 1×10^{-6} torr. New DC-705 after being exposed to a high outgassing environment for 25 hours during Skylab payload shroud jettison test 3 achieved a vacuum of only 4.8×10^{-5} torr, for a resultant performance loss of nearly 50 times.

Figure 1(c) shows the Brayton space radiator system; which produced the other contaminated oil samples, in the SPF chamber. Oil exposed to a high outgassing environment for 339 hours produced a 2×10^{-5}-torr vacuum. After 943.5 hours of operation, another oil sample was drawn from a 122-cm- (48-in. -) diameter pump and its performance checked. This 943.5-hour-old oil, for all practical purposes, was identical to 3000-hour-old oil in performance (off two decades from new oil, representing a 100 times loss in pumping efficiency). However, its color was slightly lighter. As described in the foregoing theory discussion, the G-4 now was operating at a higher equilibrium pressure, limited by the reverse fractionation rate of the contaminated pump oil, which in effect depends on the total number of contaminants, their respective quantities, and their respective vapor temperatures and pressures.

Interest in the 0.1-decade pressure fluctuation at the 4-hour point of the G-4 performance test of standard 339-hour-old oil led to confirming the repeatability and/or the extent of such fluctuations in pressure. Since any changes in ultimate pressure are indications of changes in pump efficiency, which is directly correlatable to pump speed, the approach of improving the self-cleansing capability of the diffusion pump as a still was embarked upon.

The results of the oil performance tests given in figure 12, performed with the apparatus illustrated in figure 6, show that the actual age of the diffusion pump oil and its color are not important. The ability of a specific diffusion pump oil sample to create a vacuum is governed by the outgassing environment and the length of time to which it is exposed.

Contaminant effect on oil performance. - Individual plasticizers, their effect when mixed into new DC-705 at various percentages, and the resultant effect on ultimate vacuum were studied. Some results are revealed in figure 13. One and 3 cu cm of diisooctyl phthalate (DIOP) mixed into 60 cu cm of new DC-705, for a total of 1.4 and 4.78 percent contaminants, respectively, achieved an ultimate vacuum of 4.2×10^{-6} and 2.2×10^{-5} torr, respectively. However, 5 cu cm of DIOP mixed into 60 cu cm of DC-705, for a total of 7.7 percent contaminants, achieved only a 2.3×10^{-4}-torr vacuum, representing a 100 times loss in efficiency.

The other phthalate plasticizer contaminant runs with new DC-705 followed the general trend of increasing pressure as revealed by the corresponding increase in percentages of DIOP contaminant runs. Refer to diisodecyl phthalate (DIDP) contaminant mixture data. However, in this report only the data associated with 7.7 percent of the following contaminant mixtures were included for dioctyl phthalate/di-2-ethylhexyl phthalate (DOP/DEHP) and ditridecyl phthalate (DTDP). Surprising was the slightly higher ultimate pressure obtained with DTDP (530 MW), contrary to the expectation that higher-molecular-weight contaminant would affect the DC-705 mixture the least. Evidently, additional study is required in this area.

These tests also revealed a 0.1-decade fluctuation after 4 hours (see 7.7 percent DIOP test data in fig. 13). This fluctuation was observed to be associated with the filling of the top alembics in the vertical portion of the G-4 glass diffusion pump. The following reduction in pressure corresponded to the spillover of concentrated contaminant fluid from these top alembics. Thus, the logical solution was to remove the fluid from the top alembics periodically, which was accomplished in a G-4 pump modified for this purpose.

Reclaiming contaminated vacuum diffusion pump oil. - DC-705 oil is chemically a pentaphenyltrimethytrisiloxane having a molecular weight of 546. As the oil becomes contaminated by adsorbing impurities, a portion of the fluid mixture changes partially by polymerization into a hexamer, heptamer, octamer, and so forth, and/or cracks by depropagation into a monomer, trimer, tetramer, and so forth. NASA Lewis and KSC spectroscopic analyses of various molecular distillation reprocessing cuts revealed (table I(b) and fig. 8) a contamination profile which confirms this theory. Volatile constituents were separated from the pure DC-705 oil by two light-end (10 and 7 percent) distillation cuts that removed contaminants in the 100- to 500-molecular-weight range as a distillate. In this manner, 17 percent of the light contaminants, peaking at approximately 300 MW and weighing about 78 kg (172 lbm) were removed. Of sample 41 (table I(b)), 49 kg (108 lbm) was separated by the first light-end distillation cut. Twenty-nine kg (64 lbm) was removed later by a second light-end cut. Heavy-end contaminants of 600 to 800 molecular weight or more (sample 42, table I(b)) were removed as a residue during another distillation cut. Nine percent of these heavy contaminants, concentrated at approximately 700 MW and weighing 42 kg (92 lbm), were removed.

Thus, by the triple-cut reclamation distillation process, in either a high-vacuum centrifugal molecular still or a high-vacuum molecular wiped-film type of still, 3000-

hour-old contaminated DC-705 oil was successfully reprocessed. Oil that was once considered worthless (453±15 kg, or 1000±30 lbm) was reclaimed, yielding approximately 74 percent new oil. For only two reclamations a $34 700 annual savings could be realized. Prior to reclamation, this oil achieved 2×10^{-4} torr in a 4-hour G-4 acceptance performance test (fig. 12); however, after triple-cut reprocessing, 1.65×10^{-6} torr was achieved, for a performance gain of over 100 times.

Figure 14 provides pressure and temperature characteristics of some other common pump fluids. Reprocessing most of these other pump fluids by molecular distillation would involve approximately the same operating philosophy, process, and procedures as for DC-705. However, specific distillation parameters would be slightly reduced because of the lower molecular weight of these fluids.

Using high-vacuum centrifugal molecular distillation as an occasional method of reclaiming a high-priced fluid such as DC-705 or DC-704 is certainly economical. A Consolidated Vacuum Corporation CMS-15A singular molecular still reprocessed the contaminated SPF oil in about 1 week at a continuous rate of approximately 4.35 to 4.53 g/sec (34.53 to 36 lbm/hr) per each distillation cut. However, if the diffusion pump oil deteriorates as rapidly as experienced at SPF (fig. 12), the necessity of repeated oil changes and reprocessing becomes impractical because of the hinderance to the test program. A solution to this problem would be a total self-cleansing diffusion pump.

Operation and Performance of Self-Cleansing One-Millionth-Scale Glass Model of SPF Vacuum System

Figures 15(a) to (c) are the product of one continuous test, lasting 40 days, of the one-millionth-scale, self-cleansing model of the SPF vacuum system. The data shown in figure 15(d) were obtained with the same apparatus. The same 6.35-cm- (2.5-in.-) diameter by 8-mm-thick, specially polished single crystal of sodium chloride (NaCl) was used for all comparison tests shown in figure 15. Because the critical interrelated parameters are time correlated and affected by specific events, letters and/or numerals are used to designate significant data. Specific photograph numbers are shown. For corresponding typical photographs and thermovision images of related evaporative surface behavior, see figure 16.

Performance of new DC-705 oil - phase I, increased purification nets 1.5-decade decrease in pressure. - The second-generation vacuum system incorporating contamination removal technology was initially charged with 55 cu cm of new DC-705 oil (figs. 4(a) and (b)). After 3 days of operation the upper ion gage in the one-millionth-scale-model chamber was indicating a consistent 5×10^{-5}-torr pressure. On the fourth day, point A (fig. 15(a)) reveals the effect of putting on all ion gages and slowly bringing the foreline heater up to full rated capacity (35 W). A 100-percent-working surface as previously defined existed 10 percent of the time. The effect of removing approximately 6 cu cm of light-end fractions from the foreline and 2 cu cm from each drain line was an almost instant 33 K (60° F) increase in foreline temperature, along with a correspondingly 0.6-decade-lower chamber pressure. Also an increasing trend in boiler vapor and liquid temperature was observed. After a night in this configuration the variation in boiler vapor and liquid temperatures was diminishing. The periods of 100-percent-working surface were now increased in duration and in frequency to 25 percent of the time.

On the fifth day the initial effect of removing approximately 7 cu cm of heavy-end (HE) fractions from the boiler evaporative surface was an increase in the 100-percent-working-surface periods of 50 percent of the time and stabilization of the boiler vapor and liquid temperatures. Only a slight reduction in chamber pressure resulted.

On the sixth day, 3 additional cu cm of light-end fractions were removed from the top alembic of the foreline. As on the fourth day, foreline temperature increased to 408 K (275° F), boiler vapor temperature rose to 497 K (435° F), while boiler liquid temperature reached 508 K (455° F). The jet molecular release rate was visually observed to intensify, resulting in a decreasing trend in ultimate chamber pressure. The 400-watt heater input to the boiler was reduced at point B.

Points C, D, and E in figure 15 are associated with establishing the optimum boiler heater input for the best diffusion pump performance corresponding to the lowest chamber pressure. Chamber pressure now ranged from 7×10^{-7} to 2×10^{-6} torr from the 10th day to the 12th day while the evaporative surface was working 75 percent of the time.

On the 12th day the boiler evaporative surface was 100 percent working for 75 percent of the time, the foreline temperature was stable at about 394 K (250° F), the boiler vapor temperature was stable within 480 to 490 K (405° to 420° F), and the boiler liquid temperature varied from 495 to 500 K (430° to 440° F). The only limitation remaining was the achievement of a continuous 100-percent-working evaporative surface. Thus, an additional 4 cu cm of light-end fractions were removed from the top alembic in the foreline in two equal steps within an hour. Boiler fluid depth was approximately 4 mm when this alembic again became full within 20 to 22 minutes. This removal of minute quantities of unknown light-end fractions resulted in a continuous 100-percent-working surface - for the first time. Further removal of 2 cu cm of light-end fractions from the top alembic resulted in a decrease in ultimate chamber vacuum. Foreline temperature was reduced by 14 to 140 K (25° to 250° F); however, fluctuations in boiler vapor and liquid temperature were, for all practical purposes, nonexistent. Both parameters were near their all-time weighted average maximum points, 491 and 501 K (425° and 442° F), respectively. The boiler fluid depth was now about 3 mm for 75 percent of the evaporative surface area; but because of vapor recoil action, 20 to 30 percent of the boiler bottom was bare, except for what appeared to be a minute molecular film layer. This layer was surmised to be left by the ever-moving vigorous-boiling evaporative surface. See photograph 21 (fig. 16) for visual illustration.

At point F (fig. 15(d)) 10 cu cm of new DC-705 was added directly to the diffusion pump boiler at a slow insertion rate of 1 cu cm/min, causing a small rise in chamber pressure. The foregoing addition of new DC-705 to the boiler brought the fluid level back up to approximately 7 mm. However, since the chamber pressure rose approximately 0.5 decade, to 5×10^{-6} torr, and the boiler evaporative surface was again 100 percent working for 75 percent of the time, the remaining light- and heavy-end contaminants in new DC-705 were suspect. Upon removing 2 cu cm of light-end fractions and approximately 0.5 cu cm of heavy-end fractions, chamber pressure decreased to between 1×10^{-6} and 2.5×10^{-6} torr.

Since it is very difficult to measure a highly dynamic liquid depth and/or to determine the exact amount being removed through relatively long glass tubes, the accuracy of all fluid depth and volume measurements is ±1 mm and ±1 cu cm, respectively.

Performance of new DC-705 oil - phase II, effect of plasticizer contaminants added to ultrapure DC-705 and their removal. - With the detailed self-cleansing operating philosophy established, the data of figure 15(b) are described more comprehensively.

Event I: Four cu cm of a phthalate ester mixture consisting of 2 cu cm each of DIOP, DIDP, and DOP/DEHP was inserted directly into the highly purified DC-705 diffusion pump boiler after phase I testing. A chamber pressure increase of 1000 to 10 000 times resulted. Foreline pressure also increased two to four times.

Event II: Chamber pressure was stabilized at 7×10^{-4} torr. Removing 4 cu cm of light-end contaminants from the top alembic of the foreline yielded the expected improvement in chamber pressure. Chamber pressure was 1.2 to 1.5 decades lower (2.5×10^{-5} torr) within 1 hour.

Event III: The remaining 2 cu cm of phthalate ester contaminant mixture was inserted slowly (at less than 1 cu cm/min) directly into the boiler. A duplication of the previous pressure fluctuation resulted. Ten cu cm of DC-705 was added so as not to deplete the boiler supply during the pending cleansing period. When 2 cu cm of light-end fractions was removed twice within a 30-minute period, the chamber pressure was reduced 3 decades within 1 hour, from 3×10^{-3} to 2.5×10^{-6} torr. Sample 8, 25 cu cm of light-end fractions, and sample 9, 5 cu cm of heavy-end fractions, were removed for analysis (table I(c) and fig. 15(b)). The following morning, 15 hours later, 8.5×10^{-7}-torr vacuum was achieved.

No improvement in chamber pressure occurred until the 17th day, when the one-millionth-scale glass model of the SPF chamber was baked out 383 K (230° F). A 0.25-decade improvement was achieved, to 7×10^{-7} torr. The boiler evaporative surface was

100 percent for 99 percent of the time. This behavior pattern was maintained for more than 3 days.

Event IV: The baffle temperature of the glass scale-model SPF chamber was lowered to 250 K (-10° F) in order to study its effect on ultimate chamber pressure. The results were inconclusive. Six days later (26th day of operation) the baffle temperature was reduced to 225 K (-55° F) without the expected corresponding change in chamber pressure. Chamber pressure now was 2×10^{-6} to 5×10^{-6} torr, up 0.5 decade.

Event V: Because the existence of light-end fractions was surmised, 1 cu cm was removed from the top foreline alembic. An immediate increase in pump efficiency was indicated by a 0.5-decade decrease in chamber pressure, to the range 7×10^{-7} to 1×10^{-6} torr. After a 16-hour period of chamber bakeout at 355 K (180° F), the lowest ultimate chamber pressure (5×10^{-7} torr) was achieved.

Performance of new DC-705 oil - phase III, effect of plasticizer contaminants added into chamber and their removal with cold and warm baffles. - The data of figure 15(c) are discussed.

Event VI: Four cu cm of a phthalate ester mixture consisting of 2 cu cm each of DIOP, DIDP, and DOP/DEHP was inserted slowly into the ultraclean test chamber. The expected chamber pressure increases of 1000 to 10 000 times resulted. Chamber baffle temperature was 225 K (-55° F).

Event VII: Sixteen hours later the chamber pressure was 1.5×10^{-6} to 2×10^{-6} torr. Plasticizer contaminant had condensed on the bottom of the test chamber. A chamber bakeout at 380 to 410 K (220° to 280° F) was initiated in an attempt to vaporize the newly added plasticizers. An immediate 1.2-decade rise in chamber pressure occurred. With the chamber heated to its highest bakeout temperature (380 to 410 K; 220° to 280° F), an additional 2 cu cm of the phthalate mixture was inserted into the top of the chamber. Chamber pressure increased from the 1×10^{-5}-torr range to the 3×10^{-3}-torr range. The chamber baffle was allowed to warm up to 265 K (20° F) in order to render it ineffective in protecting the highly purified DC-705 in the diffusion pumps. The outgassing load created by the vaporizing plasticizers on the heated chamber walls started to affect the pump efficiency until chamber pressure stabilized at 1.5×10^{-5} to 3×10^{-5} torr. This was a crude simulation of the outgassing from heated (heavily plasticized) cables.

On the 31st day, upon removing 3 cu cm of light-end contaminants from the top alembic in the foreline, the expected typical improvement started immediately. However, because of a power outage in the SPF vacuum technology laboratory sometime that night, a discontinuity in the data exists.

Event VIII: As the DC-705 pump fluid became repurified, the chamber pressure returned to its prior levels of 6×10^{-7} torr. The corresponding foreline temperature of 403 K (265° F) indicated a high molecular throughput. The chamber baffle temperature was 240 K (-25° F). Warming up the chamber baffle to ambient after the diffusion pump was ultrapure did result in a slight chamber pressure rise of approximately 0.5 decade. Evidently, the vast majority of deteriorating contaminants were removed by the self-cleansing process of this diffusion pump vacuum system. Sample 4, 6 cu cm of residual boiler fluid, and sample 5, 6 cu cm of light-end fractions, were removed for analysis at the end of the test (table I(c) and fig. 15(c)).

Effect of boiler surface behavior on pump performance. - Even though the typical schizoid (torpid/working) surface behavior patterns have been repeatedly duplicated with various DC-705 contamination modes, discussion of the photographic, thermovision, and motion-picture documentation has been purposely deferred to this section.

The test sample was DC-705 previously exposed to an average gamma dose rate of 25 milliroentgen per hour for 5 years in the vacuum diffusion pump system of the Michigan State University Cyclotron. The oil was discolored by use to a dark brown. This oil achieved a 2.5×10^{-5}-torr ultimate vacuum in the standard G-4 oil performance test (fig. 12). This oil was always handled with the best of vacuum practices; that is, backfilling the vacuum system with dry gaseous nitrogen upon diving the cyclotron to atmospheric pressure, never thermally shocking it to atmospheric environmental contaminants, and so forth.

Figure 15(d) shows that after a day of operation the ultimate test chamber vacuum obtained was 5×10^{-5} torr. A corresponding chamber baffle temperature of 233 K (-40° F) did not appear to limit the deteriorating effect or reverse fractionation of light-end contaminants. For a comparison of boiler evaporative surface behavior patterns at various generated ultimate chamber pressures, see figure 16.

Figures 4(b) and (c), and photograph 15 of figure 16(a), taken at a chamber pressure of 2×10^{-4} torr, reveal the different evaporative surface behavior phenomena existing for this level of pump performance: a completely torpid surface, a schizoid surface of many isolated and/or interconnected working holes, and a two-phase schizoid (torpid/working) surface, respectively. All behaviors are typical of the silicone pump fluids. A broken light source similar to Hickman's, consisting of 16 high-intensity lamps arranged into a square with the distance between lamps equal to the width of the bulb, was used to highlight the evaporative-surface behavior patterns (refs. 19 and 24).

The motion-picture film supplement C-280 contains high-speed and slow-speed film clips of the numerous evaporative surface behavior patterns existing during pump operations of various efficiencies, along with the thermovision record. The thermovision camera essentially thermally maps what it views by utilizing isotherm markers (ref. 40). The NaCl lens afixed to the boiler allows 90 percent transmittance between wavelengths of 0.2 to 13 μm. Typical thermovision images in figure 16(a) show a schizoid (torpid/working) evaporative surface with numerous holes and a single working hole in a torpid surface. The evaporative surface horizon in the diffusion pump boiler is easily identified. Significant is the localized eruption of molecules being released from the working hole well into the pump boiler vapor cavity. This thermovision photograph confirms

Hickman's description (refs. 19 and 20) of working valleys of different depth. The deepest part of the working valley emits the most vapor. Molecular release is greatest from these areas since the evaporative surface is always changing. The size and shape of the working holes are also changing as well as their existence, location, growth, movement, and so forth.

Motion-picture supplement C-280 reveals the speed at which the evaporative surface behavior quickly changes from torpid behavior to groups of isolated working holes in a torpid surface.

Another typical thermovision record (fig. 16(b)) was taken with a Corning Glass 9754 germinate (germanium dioxide) composition lens, 90 percent transmittance in the 0.2- to 6-μm range only, and new DC-705 oil under operating conditions and parameters identical to those for the 5-year-old oil. These sequential enlarged thermovision 16-mm motion-picture frames show the numerous vapor streams being released from a schizoid evaporative surface.

Two other photographs (figs. 16(c) and (d)) were taken within a few seconds of each other. One, (fig. 16(c)) taken at a rising chamber pressure of 5×10^{-5} to 10×10^{-5} torr, portrays primarily a torpid surface generating a 0.5-decade-higher chamber pressure. The other (fig. 16(d)) portrays a 100-percent-working evaporative surface yielding an improved vacuum or lower chamber pressure of 5×10^{-5} torr. The evidence of increased flow is everywhere in this photograph - inside the jet assembly, at the rim of the individual jets, in molecular buildup on the barrel surface, in the foreline, and so forth.

A marked increase in pump performance was apparent as soon as light-end and heavy-end contaminants began to be removed from the pump fluid. Within 8 hours from the start of cleansing-separation-purification processing, a 50 times increase in pump performance was achieved, as indicated by a 1.5-decade-lower chamber pressure - varying between 1×10^{-6} and 5×10^{-6} torr. An increase in diffusion pump performance beyond 50 times, or 1.5 decades, becomes tied to consistent maintainence of a 100-percent-vigorously-boiling evaporative surface. This surface behavior exists only 70 percent of the time at this particular motive-fluid purity (or impurity) level. After the second day of total operation, the first full day of contaminant pump fluid cleansing activity, an ultimate chamber pressure varying between 8×10^{-7} and 1×10^{-6} torr was obtained. The corresponding boiler evaporative surface behavior associated with this pump efficiency was 100 percent working for approximately 90 percent of the time. The fluid color was still dark brown.

After the first 7 days of system operation, the ultimate chamber vacuum fluctuated between 5×10^{-7} and 1×10^{-6} torr, with a corresponding improved evaporative surface now being 100 percent working for approximately 99 percent of the time. As indicated by figure 15(d), a total of five individual purification-separation steps were performed thus far, removing about 17 cu cm of light-end contaminants and 8 cu cm of heavy-end contaminants. Performance gains of 100 times and a 2-decade decrease in pressure were achieved with what had been old discarded pump oil. These gains are directly related to the maintainence of an almost continuous 100-percent-working (vigorously boiling) evaporative surface. Impurity levels are reduced to the parts-per-million to parts-per-billion range, with separations of nearly identical molecular-weight contaminant constituents possible when these performance gains are realized. This comment is based on analyzes performed by A. R. Huntress of Dow Corning Corporation on various concentrated contaminant fractions isolated and separated by this self-cleansing pump and reference 22. All other critical operating parameters were similar to those of the previous tests. Points A and B (fig. 15(d)) confirmed the interdependence of all critical parameters with minute changes in chamber baffle temperature. This is evident even at nominal baffle temperatures of 233 to 246 K (-40° to -15° F). The initial 269 K (25° F) warmup of the baffle released some capture volatiles which affected the overall chamber pressure in two ways:

(1) Their existence in the chamber and pump motive fluid created an overall higher dynamic equilibrium system pressure.

(2) They tended to polymerize and collect on the boiler evaporative surface, thereby increasing its surface tension. This gradually chokes the 100-percent-working vaporization surface for an instant and causes the molecular release rate to be significantly reduced.

The occasional higher isolated pressure-rise spikes are caused by the restartup of the total 100-percent-working surface with its increased vaporization rate, as noted by points B_1 (pressure spike) and B_2 (normal operating level) in figure 15(d).

Point C (fig. 15(d)) shows again that further increases in pump efficiency (decreases in chamber pressure) can be expected as long as there are some light-end contaminants available for removal, regardless of the chamber fluctuations in baffle temperature.

Point D (fig. 15(d)) indicates achievement of a continuous 100-percent-working evaporative surface. Point E shows the effect of changing the flow and distribution of the cooling air in the SPF vacuum technology laboratory and/or the effect of minute increases in diffusion pump heater voltage.

Point F (fig. 15(d)) is noteworthy in that the color of the boiler fluid changed from dark brown to light yellow overnight after the heater voltage was increased approximately 1 volt. The expected effect of turning on an additional ion gage is also shown (fig. 15(c)). Figure 16 shows further evidence of an increase in molecular flow throughout the entire pump when 100 percent of the evaporative surface is continuously working.

An investigation into the effectiveness of the chamber baffle at 5×10^{-7}-torr-vacuum range was undertaken. The effect of the first major baffle temperature warmup cycle to 278 K (40° F) on all critical parameters is indicated by event I (fig. 15(d)). Respective baffle temperature warmup cycles are denoted by events II, III, IV, and VI. As Huntress (ref. 22) implied and the foregoing results confirm, if fluid vapor pressure is maintained at its lowest absolute value (10^{-10} torr for DC-705), corresponding to an ultra-high-purity condition, many hours of vapor-free testing are provided with no more than a water-cooled baffle. Significant reductions in the cost of operating large space simulation facilities result.

Figure 16(f) reveals the violent dynamics of a continuous 100-percent-working evaporative surface. Note the partially bare boiler bottom, the vapor recoil action, the gobs of liquid actually carried along with the high molecular release rate from the boiler fluid, and the heavy buildup of molecules and liquid formations throughout all portions of the diffusion pump assembly.

Events VII and VIII reveal the criticality of the proper amount of boiler heater input in successfully maintaining a continuous 100-percent-working evaporative surface with its associated increase in optimum diffusion pump efficiency. Sample 1, 6 cu cm of light-end fractions, and sample 2, 4 cu cm of heavy-end fractions, were removed for analysis at the end of the test (table I(c) and fig. 15(d)).

CONCEPTUAL DESIGN MODIFICATIONS AND PROPOSED OPERATION OF A SELF-CLEANSING 122-CENTIMETER- (48-IN. -) DIAMETER DIFFUSION PUMP

With a basic understanding of the self-cleansing technology established, the implementation of these innovations as modifications to presently installed diffusion pumps regardless of the pump fluid being used is the principal object of this last section. It is believed that incorporating these self-cleansing innovations into any or all existing large vapor-operated diffusion pumps will provide performance gains similar to those achieved by the scale-model pump.

How these self-cleansing concepts can be incorporated into a typical large 122-cm- (48-in. -) diameter diffusion pump, for example, is illustrated by figure 17. This figure is a cutaway view illustrating all modifications incorporated into the typical large diffusion pump previously shown in figures 2 and 3. Three distinct innovations are proposed in this conceptual design (fig. 17): First, the original foreline discharge pipe of figure 2 is to be modified into the molecular distillation device (1900). Second, the original boiler is to be modified into a heavy-end-impurity device (3600). Third, the original diffusion pump barrel (or housing) is to be modified into an ultrahigh molecular-purification-separation device (1000).

A detailed description of each of these devices is given in the following paragraphs.

Foreline Distillation Modification Design

The foreline discharge pipe was modified to act as a molecular still (fig. 17, 1900). Eight to 16 light-end contaminant fractions could be removed by eight condensate collection plates (1910, 1920, etc.) located in the foreline discharge pipe. The first three plates in the foreline elbow act as dams. They provide liquid collection points for the lighter light-end contaminants during the initial startup phase of the diffusion pump. Each dam contains two condensate drain tubes. In the case of the lowest plate the first tube (1911) drains the bottom, and its isolation valve (1912) removes the A fraction impurity. The next tube (1913) skims the condensate surface, and its isolation valve (1914) removes the B fraction impurity. Removal of these light light-end impurity fractions by skimming and draining them into a vessel of lower pressure is identical to the self-cleansing process described previously in connection with figure 10. It is apparent that with the larger number of foreline molecular distillation plates, purification-separation efficiency is increased. The solenoid-operated vent valve (1915), and the vacuum isolation valve (1916), control the pressure difference between the lower-pressure vessel (1901), and the foreline still (1900). Samples for analysis are provided by the drain valve (1917) on the lower-pressure vessel (1901).

Figure 18 illustrates the effect of removing impurity fractions A, B, C, D, E-1, and E-2 collected by the plates (1910, 1920, 1930) in the foreline elbow. The slightly heavier light-end impurities identified as C, D, E-1, and E-2 are captured by progressive evaporation and condensation at plates located at higher elevations within the foreline molecular still and are removed sequentially after those previously identified at A and B have been removed.

Similarly, the heavy light-end fractions that collect on the upper-foreline-still plates (1940, 1950, 1960, 1970, and 1980) are removed sequentially as F, G, H, I, and J fractions on a timed quantity drain cycle, based on a predetermined schedule, into another vessel of lower pressure. The draining of each contaminant fraction occurs a specific time from the startup of the diffusion pump. The extra-heavy light-end fractions K-1, K-2, K-3, L-1, and L-2 are removed later by skimming operations that discharge the contaminants into a yet another lower-pressure vessel (1903), in a similar sequential manner. However, the K and L fractions may be successfully removed only after the heavy-end contaminants have been skimmed from the boiler evaporative surface. The foreline still is wrapped with external heaters (1990) and insulated conventionally (1991) in order to generate and maintain the desired temperature conditions for distillation.

Diffusion Pump Boiler Modification Design

The diffusion pump boiler was modified to act as a heavy-end-impurity separation device (fig. 17, 3600) to remove the contaminants floating on the motive-fluid evaporative surface. Removal is always achieved regardless of the fluid depth by eight pairs of skimming tubes, four pairs of tubes of different heights located at the center area of the boiler and four pairs of tubes of equivalent height at the boiler's peripheral. The four pairs of peripheral boiler tubes, each pair diametrically opposed, remove the heavier heavy-end contaminants by skimming and sucking them into a vessel of lower pressure (1904). A liquid level indicator (142) electrically closes all boiler skimming bleed valves that are below the motive-fluid evaporative surface.

The scheduled point in the self-cleansing operation at which the heavy-end fractions are removed depends on the amount and type of contaminants that exist in the motive fluid. However, for the average case, at any time immediately following the removal of the J light-end fraction, the heavy-end impurities can be removed by opening one pair of diametrically opposed peripheral skimming valves for approximately 1 second. It may be necessary to repeat this skimming operation only once more 1/2 hour later, if the heat distribution fins (fig. 3) are removed from between the concentrically arranged annular heating channels (fig. 19). Heating distribution fins in the motive fluid tend to impede the removal of the surface contaminants. Therefore, numerous peripheral skimming operations may be necessary to remove the heavy heavy-end fractions if these fins remain in the diffusion pump boiler.

An hour after the heavier heavy-end impurity constituents have been successfully removed, the light heavy-end fractions can be removed by using a central pair of diametrically opposed skimming tubes. The efficiency of this skimming operation is subject to the impedance of any heat distribution fins. All subsequent skimmings of heavy-end contaminants tend to remove the more difficult lighter heavy-end contaminants and discharge them into a vessel (1905) of 100- to 1000-times lower pressure.

Table II outlines a typical, proposed, self-cleansing, timed-sequence operating schedule with critical parameters. The motive fluid used in this example is DC-705. However, using another pump fluid would only tend to alter slightly the self-cleansing timed-sequence schedule of critical temperature parameters.

Throughout the foregoing discussion illustrating the proposed self-cleansing procedural operations and design philosophy, several assumptions were made:

(1) The motive fluid was surmised to consist primarily of fractions M and N, however, another diffusion pump fluid could have its primary components as J and K, I and J, O and P, or Q and R, and so forth.

(2) An absolutely pure pump fluid does not exist; however, some fluids reject contaminants easier than others.

(3) The innovations shown in figures 17 and 19 will allow any pump to separate contaminants that exist in and/or are absorbed by its motive fluid. The light heavy-end fractions are more easily removed from the evaporative surface closer to the boiler dome, while the heavier heavy-end fractions are removed at the outer portion of the boiler. Several levels of evaporative surface skimming are required because fluid quantity decreases as impurities and contaminant constituents are removed. The light-end fractions are removed by the foreline still through sequential opening of each valve from A to K-3 for periods varying from 1 to several seconds.

(4) The degree to which impurities are absorbed by different diffusion pump fluids alters the typical (fig. 18) three-peak constituent-mixture distribution profile in shape, location, total quantity, and so forth. Thus, if the foreline isolation valve A (fig. 17, 1912) is opened, draining the condensate collected at the first plate (1910), for simplicity called the A fraction, of a given contaminated fluid mixture, the molecular weight of this fraction will vary with the different pump motive fluids being used. Therefore, the molecular weight of fraction A from one pump fluid may coincide with the molecular weight of the B or C fraction from another pump fluid. Likewise, the molecular weight of fraction B may be similar to that of fraction C or D of another fluid.

Figure 18 illustrates the individual sequential distillation cuts made according to the scheduled operating procedure outlined in table II. Each fraction removed is designated by a letter and associated with its approximate molecular weight profile. It becomes apparent that the boiler and foreline modifications contain some limitations, because some residual (minute) contaminants probably do exist as the heaviest of light-end impurities, identified as L-3 and L-4 fractions, plus the lightest of heavy-end impurities, identified as O-1, O-2, P-1, and P-2 fractions.

Figures 11, 14, and 18 reveal why it is impossible to decrease the impurity level further by just incorporating a foreline molecular still and boiler evaporative surface skimming. The primary difficulty was briefly stated in conjunction with figure 8 and the problems associated with reprocessing by using a high-vacuum centrifugal molecular still. The closer the impurity molecular weights are to the molecular weight of the basic fluid mixture, the less efficient will be the desired separation for a specific temperature and pressure, thereby necessitating re-runs of the distillate. Because the modifications will be applied to existing diffusion pumps of all types and designs, the probability of increasing area foreline or boiler external temperatures much beyond what has been proposed by using external heaters and insulation is not too likely. The only other option is to reduce the pressure in the foreline further into the 10^{-4}- to 10^{-5}-torr range. This will provide a significant separation of impurities which are close to the molecular weight of the basic fluid mixture and will thereby increase molecular distillation efficiency.

Extensively modifying large vacuum facilities to further reduce their foreline pressures by increasing roughing pump efficiency and capabilities is quite costly. Therefore, it is more likely that diffusion pump barrel modifications would be incorporated selectively into one or two pumps. These pumps would then act as ultrahigh purification-separation-distillation slaves to all the other pumps, which would have just the previously discussed boiler and foreline modifications. This selective modification would work because pressures within an operational diffusion pump range from 10^{-7} torr at the inlet to 10^{-4} torr near the discharge outlet. Modifications to the diffusion pump barrel are discussed in the next section. For further details see patent application (ref. 41).

Diffusion Pump Barrel Distillation Design

The barrel (or housing) of the diffusion pump (fig. 17, 1000) was modified to act as an ultrahigh molecular-purification-separation device. Five to 10 contaminant fractions with molecular weight nearly equal to that of the basic motive-fluid mixture can be removed by distillation plates (fig. 17, 1100 to 1500). These distillation plates act as condensate collection gutters along the internal peripheral surface of the diffusion pump barrel (or housing). They are located primarily below the main vapor stream being discharged from each respective jet nozzle on the Christmas tree assembly. The two upper distillation plates (1100 and 1200) provide soluble liquid collection points for the heaviest of light-end contaminants, identified as L-3 and L-4, after the foreline molecular still has removed the other light-end fractions. Otherwise, all light-end impurity fractions would be also present, as previously stated in connection with figure 10. Each plate contains two condensate drain tubes. One tube (1101) and its isolation valve (1102) bleed and drain the L-3 fraction from the upper surface area into a lower-pressure vessel (1103). This captured condensate is formed by what little reverse fractionation occurs from the barrel walls. Solenoid-operated vent valves (1104 and 1204) plus vacuum isolation valves (1105, 1106, 1206) control the pressure difference between the lower-pressure vessel (1103), and the diffuser barrel (1000). Samples for analysis are provided by the drain valve (1107) on vessel 1103.

The condensates, identified as M and N, which is the basic motive-fluid mixture which has crept up the diffusion pump barrel wall, are periodically drained into the next lower distillation plate (1200) by another tube (1108) and its isolation valve (1109). This fluid mixture is then allowed to overflow that plate in its return trip to the pump boiler. If chamber pressure should rise slightly upon this mixture's return to the boiler, indicating residual impurities, the next condensate accumulation on the upper two plates (1100 and 1200) will have a larger percentage permanently separated into vessels 1103 and 1104 as L-3 and L-4 heavy light-end fractions.

The proposed removal of light heavy-end impurities O-1, O-2, and P-2 follows a similar waterfall separation process. Because of a dynamic molecular distillation evaporation-condensation equilibrium existing at the four lower distillation plates (1200 to 1500), with constant replenishment from the Christmas tree jet assembly, the individual enrichment of each fraction may take several minutes before that fraction exists in sufficient quantity to warrant isolation into its respective storage vessel (1203, 1303, 1403, 1503, or 1603).

The diffusion pump shown in figures 17 and 19, modified by all three innovations, now is an ultra-high-efficiency molecular purification-separation device, in addition to being a diffusion pump, with capabilities of increased performance greater than 100 to 1000 times.

It was previously stated that this device may be best used as a slave to remove impurities that other, only partially modified pumps, could not. In this use, the boiler drain line (143) shown in figures 17 and 19 and the isolation slave feedline would be interconnected to 15 other diffusion pumps. Figure 15 and supporting research data using the pump configuration in figure 10 show that the boiler fluid level may be reduced by 50 percent without affecting the pump's performance or sustained reliability. The 15 other partially modified diffusion pumps, those having only foreline and boiler modifications, would sequentially drain a portion of their motive fluid through their respective drain lines (143) and isolation valves (144) to the slave pump. The fluid could be transferred by a small magnetic-coupled pump. Or because the normal boiler pressure is approximately 100 times greater than the internal pressure of the inlet port tube (1601), sufficient differential pressure may exist to force the fluid into the slave pump. Fluid feed rate would be regulated by an isolation solenoid valve (1608). The open-close ratio of this valve may be set initially at 1 second open and 10 to 20 seconds closed if highly volatile outgassing conditions exist in the test chamber. For additional details, see reference 41.

The advances in space simulation vacuum-distillation technologies described herein represent a more-thorough treatment than that which was presented in reference 42.

SUMMARY OF RESULTS

In investigating the complex problem of why large space simulation chambers do not realize true ultimate vacuum the following results were obtained:

1. Torpidity, a menace to high-vacuum pot stills but not generally considered a serious factor in vapor pumps, is a definite factor in reducing pump efficiency by 100 times. Data taken with the self-cleansing, one-millionth-scale, glass model of the SPF diffusion pump showed that 100-percent-working evaporative surfaces produce ultimate pressures 100 to 1000 times lower than those of torpid evaporative surfaces. It is surmised that torpidity is the result of increased surface tension caused by impurities

collecting on the evaporative surface, which acts as a barrier limiting the molecular release rate.

2. No specific chemical which would confer torpidity when added to a nontorpid liquid had previously been known. The SPF scale-model test data showed that phthalate esters, namely diisooctyl phthalate (DIOP) and/or diisodecyl phthalate (DIDP), added directly into the boiler fluid or vacuum system chamber caused a nontorpid surface to become partially schizoid (100 percent working for 50 percent of the time), with a corresponding increase in pressure of 3 decades. Analyses of light- and heavy-end fractions separated from contaminated (torpid or schizoid) DC-705 silicone oil detected phthalate ester plasticizers.

3. Although further increases in specific pumping speed have not been considered likely, the self-cleansing glass scale-model diffusion pump, incorporating contamination-removal-technology innovations, consistently produced lower ultimate pressures than previously achieved. For new DC-705 oil, the chamber pressure was 1.5 decades lower; for 5-year-old DC-705, over 2 decades lower; and for new DC-705 oil intentionally contaminated with phthalate esters, nearly 3 decades lower. Diffusion pump innovations to allow removal of contaminants from pump oil during operation were successfully tested. Vacuum distillation removal of light-end-fraction contaminants and boiler bleed removal of heavy-ends fractions from the evaporative surface resulted in a 2-decade-or-more lower ultimate pressure.

4. When self-purification of the intentionally contaminated diffusion pump oil was not allowed, no significant improvement in ultimate vacuum was achieved after continuous operation in excess of 1 month, thereby indicating the necessity of removing these phthalate esters.

5. The color of DC-705 is not absolutely indicative of its purity or its capability to obtain the maximum molecular release rate from an evaporative surface. Similar ultimate vacuum levels and conditions were realized with a dark brown oil as with a lighter color oil.

6. A water baffle at ambient temperature may be all that is required for a high-efficiency diffusion pump, provided the boiler fluid is maintained in an ultra-high-purity condition (fluid vapor pressure kept at its lowest absolute value, 10^{-10} torr for DC-705) by vacuum systems incorporating contaminant-removal-technology innovations.

7. Contaminants that are commonly found in large vacuum chambers and that adversely affected silicone oil diffusion pump performance were identified as phthalate ester plasticizers. These plasticizers are used in cable insulation jackets, alkyd paints, electronic components, and so forth. Those having the most deteriorating effect on DC-705 are DIOP (390 MW), ditridecyl phthalate (DTDP, 530 MW), DIDP (446 MW), dioctyl phthalate/di-2-ethylhexyl phthalate (DOP/DEHP, 390 MW), respectively.

8. Contaminated DC-705 diffusion pump oil can be successfully reprocessed by high-vacuum molecular distillation with approximately a 75 percent yield. The reprocessed oil is capable of achieving an ultimate vacuum close to that of new DC-705 oil.

9. Degradation mechanisms of polymeric materials commonly used in space simulation involve random chain rupture or scission, producing small more-volatile fragments, and depropagation or an unzippering-type of reaction, which is the reverse of chain polymerization.

10. Self-cleansing modifications were conceptually proposed, designed, developed, and/or tested for making diffusion pumps into ultrahigh purification-separation devices

   a. Which eliminate contaminants that are accumulated or adsorbed in various diffusion pump fluids, whether new or used, thereby increasing their ability to vaporize and increasing pump performance 50 to 1000 times
   b. Which provide a novel method for enrichment of constituents having nearly identical molecular weight
   c. Which create a novel diffusion pump of increased performance
   d. Which produce technology that may have a wider utilization than that of just providing a cleaner and better space simulation environment (potential interdisciplinary applications exist for metallurgy and refining processes; screening, developing, or conditioning biomedical materials; supplemental diffusion-enrichment of isotopes; chemical processing-reprocessing distillations; and incorporation into qualitative and quantitative instrumentation devices.

11. Further study is warranted to improve the purification-separation concepts described, the methods of measuring changes in molecular release rates, and the spectroscopy fingerprints identifying contaminating constituents and related physical properties on evaporative surfaces.

Lewis Research Center,
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Cleveland, Ohio, October 23, 1974,
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TABLE I. - ANALYSIS OF MATERIALS ASSOCIATED WITH SPACE POWER

(a) Volatile (outgassing) materials

Material type	Description	Manufacturer	Total quantity exposed		Length of time exposed to vacuum, hr	Outgassing test results[a]	
						Weight loss, percent	Amount of hot vaporous condensable material, VCM, percent
Cable	Two- or four-conductor shielded cable:	Belden Manufacturing Co.					
	Sample 3 or 6a		5485 m	18 000 ft	0	----	---
	Sample 5a		5485 m	18 000 ft	3000	13. 9	9. 5
	Shielded coaxial cable:						
	Sample 8 or 6b	Belden Manufacturing Co.	305 m	1 000 ft	0	----	---
	Sample 5b	Consolidated Wire and Associated Corp.	305 m	1 000 ft	3000	2. 9	1. 2
	Neoprene^{TR}	E. I. duPont de Nemours & Co.	------	--------	0	3. 01 - 7. 95	1. 5 - 4. 14
	Three-conductor neoprene cable, American Wire Gage (AWG) 16 (sample 14)	Carol Cable Co.	915 m	3 000 ft	40	----	---
	Neoprene O-ring seal (1.9 cm, or 3/4 in.)	Irving B. Moore	137 m	450 ft	3000	----	---
	Neoprene cable (350 thousand circular mil (MCM), 5000 V); outer jacket, butyl rubber	Paranite Co., Div. of Essex Wire & Cable Co.	76 m	250 ft	3065	----	---
	Single-conductor or shielded cable:						
	Sample 17 (10 AWG)	Carol Cable Co.	760 m	2 500 ft	40	----	---
	Sample 18 (12 AWG)	Apex Wire & Cable Co.	610 m	2 000 ft	3065	----	---
	Sample 21 (18 AWG)	Belden Manufacturing Co. or Raychem Corp.	3050 m	10 000 ft	40	----	---
Paint	Gray used on polar crane	Unknown	46 m^2	500 ft^2	----		
	Green (sample 2a), used on auxiliary hoist	Paulding & Harnischfeger	1. 9 m^2	20 ft^2	65	[c]6. 9	[c]2. 0
	Red primer under gray (sample 2b), from main hoist gearbox	Rustoleum primer; Sherwin-Williams Kem Lustral top coat	9 m^2	100 ft^2	65	[d]3. 8	[d]0. 4
	Gray (sample 2c), from hoist motor	General Electric Co.	0. 9 m^2	10 ft^2	65	[e]6. 7	[e]2. 0
	Green (sample 3a), used on traction drive motor	Paulding & Harnischfeger	0. 37 m^2	4 ft^2	665	[c]6. 1	[c]1. 5
	Light gray (sample 4a), used on control cabinet door	General Electric Co. paint; Sherwin-Williams Kem Lustral top coat	5. 6 m^2	60 ft^2	3065	[d]3. 6	[d]0. 6
	Dark gray (sample 4b), used on another control cabinet door	General Electric Co.	5. 6 m^2	60 ft^2	3065	[e]4. 9	[e]1. 1
	Black (sample 4c), used on mercury vapor light cabinet	Unknown	0. 9 m^2	10 ft^2	3065	1. 7	0. 4
	Sample 36, used on catch-net tower structure	Unknown	279 m^2	3000 ft^2	65	----	----

[a] Obtained with Hitachi model RMU 6L mass spectrometer and Perkins-Elmer model 900 gas chromatograph linked with a Varian model 620-i

[b] Obtained with Perkins-Elmer model 621 spectrophotometer.

[c] Identical source.

[d] Identical source.

[e] Identical source.

Differential thermal analysis (DTA) test results (298 to 613 to 653 K), percent	General information provided by manufacturer or from analysis at Kennedy Space Center
63.6 58	Approximately 50 percent polyvinylchloride (PVC) and 25 percent diisodecyl phthalate (DIDP) plasticizer (Belden information). KSC analysis: gray outer jacket of PVC containing a phthalate and a phosphate ester; fibrous wrap (cellulose) conductor coatings of colored polyethylene; per DTA, first weight loss (plasticizer) was 59 percent, second weight loss at higher temperature (polymer) was 20 percent, inorganic fillers comprised 10 percent.
31 30	Nitrile-modified PVC (no phthalate ester plasticizers) in Geon 8720 (B. F. Goodrich nitrile rubber product), Belden and Goodrich information.
33.6 (298 to 573 K)	Outgassing test data from NASA Johnson Space Center computer printout of outgassing properties of Apollo nonmetallic materials.
------------	Outer jacket of neoprene-type rubber; inner jacket conductor insulation of styrene/butadiene-type rubber.
------------	No data given.
31.2 neoprene 31.6 butyl	Outer layer of neoprene-type rubber (7 percent inorganic fillers) with high level of aromatic amines and antioxidants (or antiozonants); inner layer of butyl rubber (polyisobutylene) with 51.6 percent inorganic fillers. Per DTA, first weight loss (plasticizer) was 22.6 percent, second weight loss at higher temperature was 8.6 percent.
------------	PVC resin with phthalate ester plasticizer and carbonate and silica filler - samples 17 and 18 and outer jacket of sample 21. Sample 21 - Mylar tape and terephthalate ester wrap with polyethylene conductor insulation. Per DTA, sample 17 white insulation had first weight loss of 61 percent and second of 30 percent with 10 percent inorganic fillers; sample 17 green insulation had first weight loss of 59 percent and second of 18 percent with 23 percent inorganic fillers; sample 18 purple insulation had first weight loss of 59 percent and second of 26 percent with 17 percent inorganic fillers; sample 21 (outer jacket) insulation had first weight loss of 62 percent and second of 13 percent.
------------	Gray alkyd oil paint (typical)[b]
------------	Phthalic anhydride (molecular weight, 148.1; content, approximately 24 percent); alkyd binder; titanium oxide; and traces of DC-705 oil.
------------	Phthalate type (butyl phthalate; molecular weight, 278); alkyd binder; talc; silica.
------------	Nonphthalate ester alkyd; carbonate; sulfate; titanium oxide; traces of DC-705 oil.
------------	Ratio of 665-hour (sample 3a) to 65-hour (sample 2a) outgassing rate is 88 percent; ratio of 665-hour to 65-hour VCM rate is 75 percent.
------------	Ratio 3065-hour (sample 4a) to 65-hour (sample 2b) outgassing rates is 95 percent; ratio of 3065-hour to 65-hour VCM rate is surmised to be over 150 percent because of absorption of silicone oil with time.
------------	Ratio of 3065-hour (sample 4b) to 65-hour (sample 2c) outgassing rates is 75 percent; ratio of 3065-hour to 65-hour VCM rate is 55 percent.
------------	No data given.
------------	Phthalate-type alkyd binder; organic salt (rosin salt); probably zinc oxide; and traces of DC-705 oil.

data system.

TABLE I. - Concluded. ANALYSIS OF MATERIALS ASSOCIATED WITH SPACE POWER FACILITY OIL CONTAMINATION PROBLEM

(b) Contaminated and reprocessed DC-705 oil

Sample	Description	Color	Analysis by infrared spectrophotometric techniques at Kennedy Space Center
41	3040 Hours old; 10 percent distillation of light-end contaminants	Clear white	Phthalate-type ester or alkyd
42	3040 Hours old; 90 percent final-cut distillation of heavy-end contaminants	Dark brown	Phthalate-type ester; polyurethane resin
44	3040-Hour-old oil used in Space Power Facility	Brown	Some phthalate ester; carbonaceous material showing carbonyl and silicone character, probably degradation of a reaction product

(c) Contaminated oil from self-cleansing, one-millionth-scale, glass model of Space Power Facility diffusion pump and from Michigan State University Cyclotron

Date	Procedure	Sample	Color	Description
3-7-73 (at approx. event II, fig. 15(b))	Samples of contaminated DC-705 oil taken after direct insertion into boiler fluid of 6 cu cm of phthalate plasticizers (2 cu cm each of DIDP, DIOP, and DOP/DEHP)	8 (27 cu cm)	Clear white	Light-end fractions from foreline; 18.4 percent phthalate ester content
		9 (5 cu cm)	Clear white	Heavy-end fractions skimmed from boiler surface; 1.7 percent phthalate ester content
3-29-73 (at approx. event VIII, fig. 15(c))	Samples of contaminated DC-705 taken after direct insertion into top of chamber of 6 cu cm of phthalate plasticizers (2 cu cm each of DIDP, DIOP, and DIOP/DEHP)	4 (6 cu cm)	Clear white	Post-test residual boiler fluid; 0.7 percent phthalate ester content[f,g]
		5 (6 cu cm)	Clear white	Post-test light-end fractions from foreline; 6 percent phthalate ester content[g]
4-19-73 (at approx. event VIII, fig. 15(d))	Five-year-old samples of DC-705 oil taken from Michigan State Cyclotron; color, dark brown	1 (6 cu cm out of 19 cu cm analyzed)	Clear white	Light-end fractions from foreline; 1.2 percent phthalate ester content[g]
		2 (4 cu cm out of 8 cu cm analyzed)	Deep yellow	Heavy-end fractions skimmed from evaporative surface; 0.7 percent phthalate ester content[g]

[f] One percent is the smallest quantity detectable with any certainty because of instrument system noise and/or no assurance that the curve goes to zero.

[g] A FTS-20 infrared Fourier transform spectrometer was used to determine the phthalate ester content of the DC-705 silicone oil samples.

TABLE II. - TYPICAL OPERATING PROCEDURE FOR SELF-CLEANSING PUMP

[Motive fluid, DC-705 oil.]

Time, hr	Chamber	Foreline	Vapor	Liquid	Foreline vapor temperature, K	Impurity fraction		Removal time, sec
	Pressure, torr		Boiler temperature, K			Letter	Location	
0	50×10^{-3}	50×10^{-3}	293	293	293	-----	---------------	---
1.0	1×10^{-3}	40×10^{-3}	488	503	373	A	Lower foreline	1
1.1	-------	-------	---	---	395	B	↓	↓
1.2	-------	-------	---	---	397	C	↓	↓
1.3	-------	-------	---	---	401	D	↓	↓
1.4	-------	-------	---	---	403	E-1	Midforeline	↓
1.5	5×10^{-4}	30×10^{-3}	490	503	403	E-2	Midforeline	↓
1.6	-------	-------	---	---	404	F	Lower foreline molecular still	↓
1.7	-------	-------	---	---	405	G	Lower foreline molecular still	↓
1.8	-------	-------	---	---	406	H	Midforeline still molecular	↓
1.9	-------	-------	---	---	407	I	Upper foreline molecular still	↓
2.0	2×10^{-4}	20×10^{-3}	491	505	408	J	Upper foreline molecular still	↓
2.1	1×10^{-4}	-------	---	505	409	S to Z	Boiler peripheral	↓
2.6	5×10^{-5}	15×10^{-3}	---	506	410	S to Z	Boiler peripheral	↓
3.6	2×10^{-5}	-------	---	507	411	P to R	Central boiler	↓
4.0	5×10^{-6}	10×10^{-3}	---	508	412	P to R	Central boiler	↓
4.1	4×10^{-6}	9×10^{-3}	493	509	413	K-1	Lower foreline molecular still	↓
4.6	3×10^{-6}	8×10^{-3}	---	510	↓	K-2	Lower foreline molecular still	↓
5.1	2×10^{-6}	7×10^{-3}	---	511	↓	K-3	Midforeline molecular still	↓
5.6	1×10^{-6}	6×10^{-3}	---	512	↓	L-1	Upper foreline molecular still	↓
6.1	9×10^{-7}	5×10^{-3}	496	513	↓	L-2	Upper foreline molecular still	↓
6.6	8×10^{-7}	5×10^{-3}	↓	↓	↓	P	Central boiler	↓
6.6	8×10^{-7}	5×10^{-3}	↓	↓	↓	L-2	Upper foreline molecular still	↓
24.0	5×10^{-7} - 8×10^{-7}	4×10^{-3}	↓	↓	↓	L-2	Upper foreline molecular still	.5
48.0	3×10^{-7} - 6×10^{-7}	4×10^{-3}	↓	↓	↓	L-2	Upper foreline molecular still	.5

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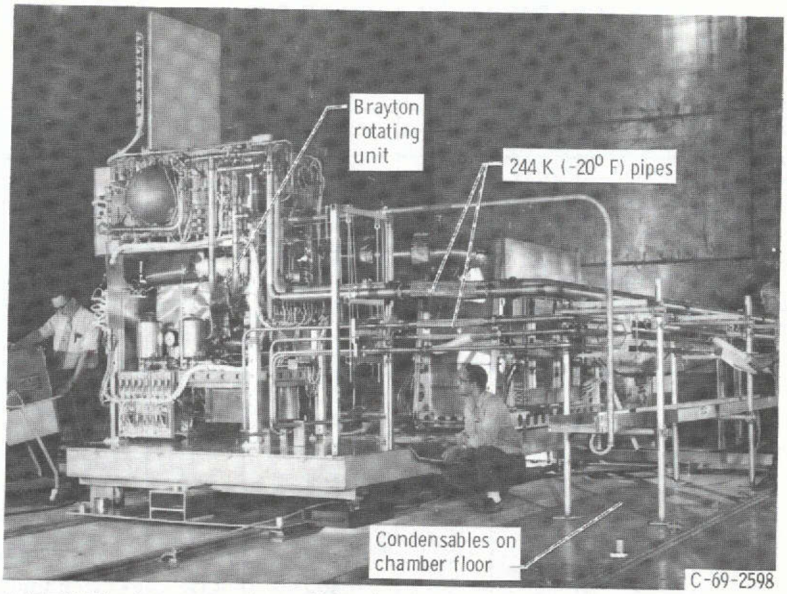
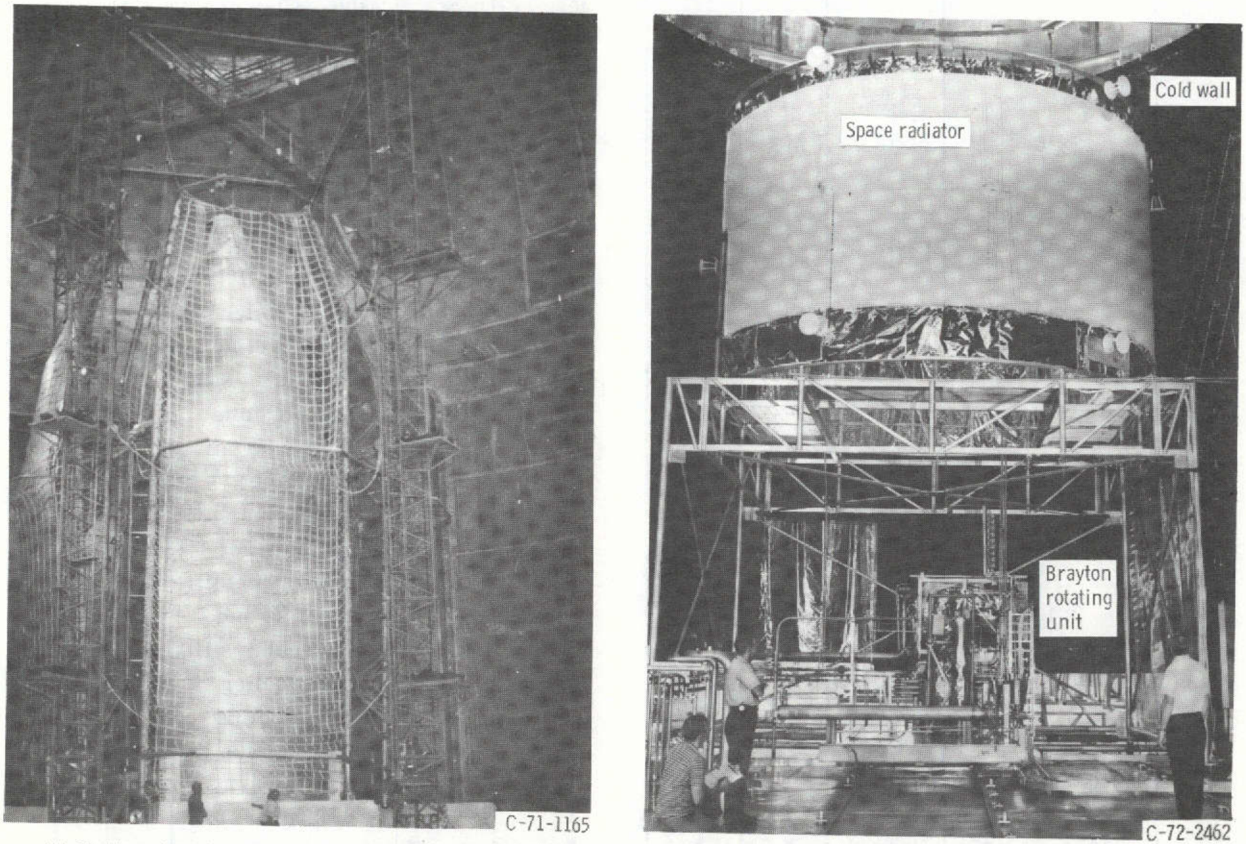


(a) Initial Brayton power system, which primarily created the 3000-hour-old oil, in SPF chamber.



(b) Skylab payload shroud jettison test setup, which created the 25-hour-old oil, in SPF chamber. The 3000-hour-old oil had been used for 64 hours of Skylab testing.

(c) Brayton space radiator system, which primarily created the 339-hour-old and 943.5-hour-old oil samples, in SPF chamber.

Figure 1. - Typical Space Power Facility (SPF) testing programs.

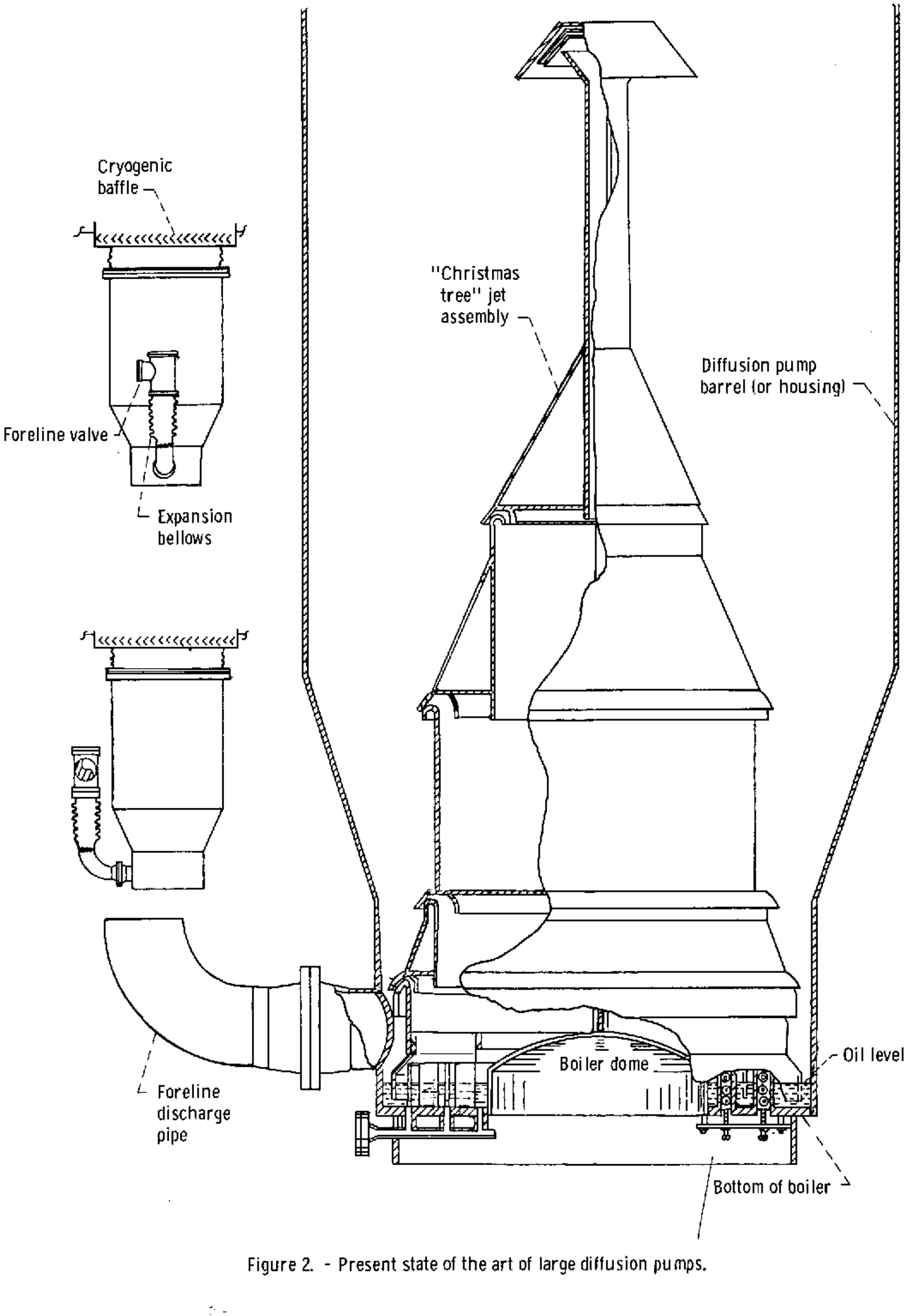


Figure 2. - Present state of the art of large diffusion pumps.

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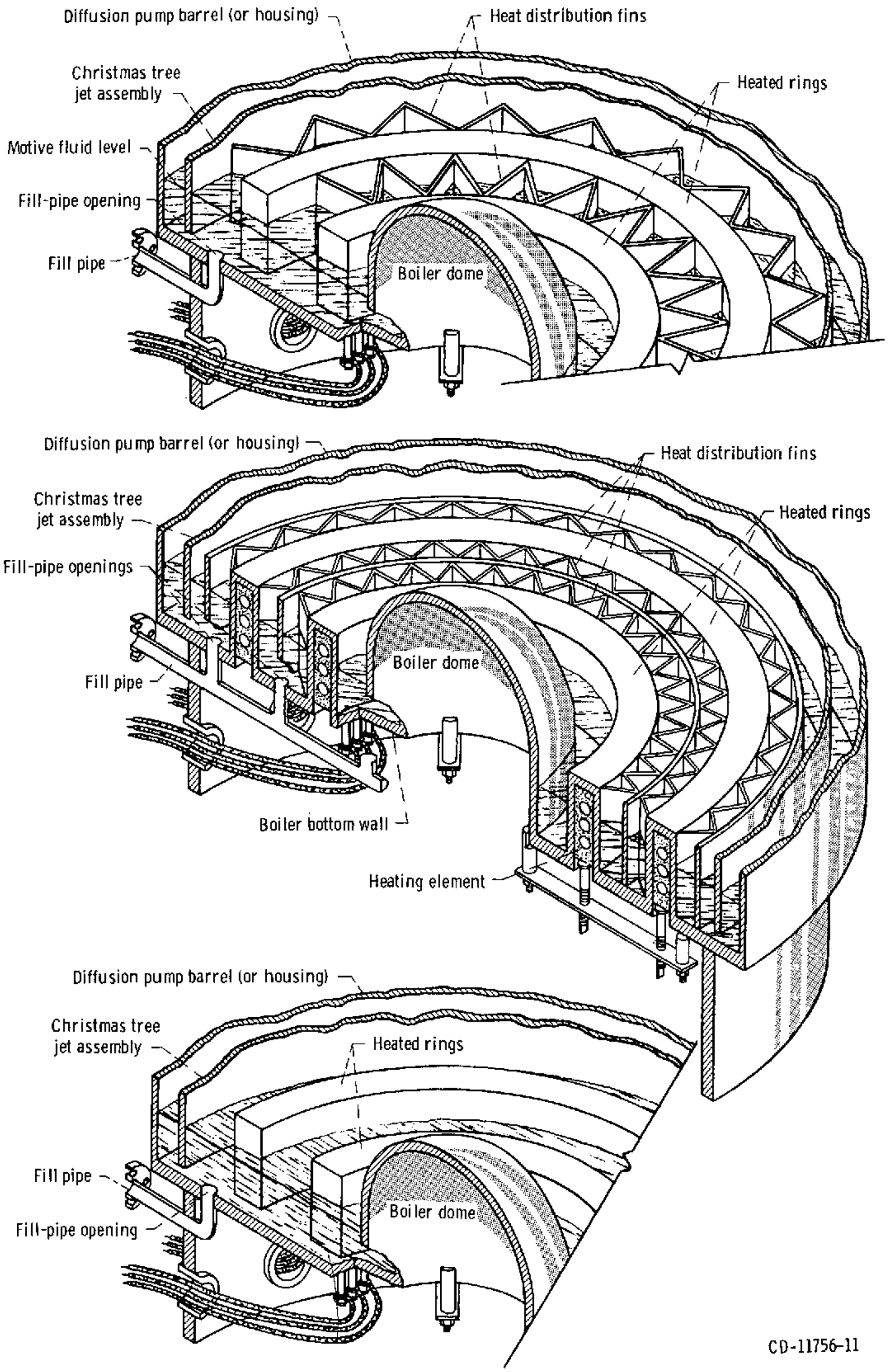
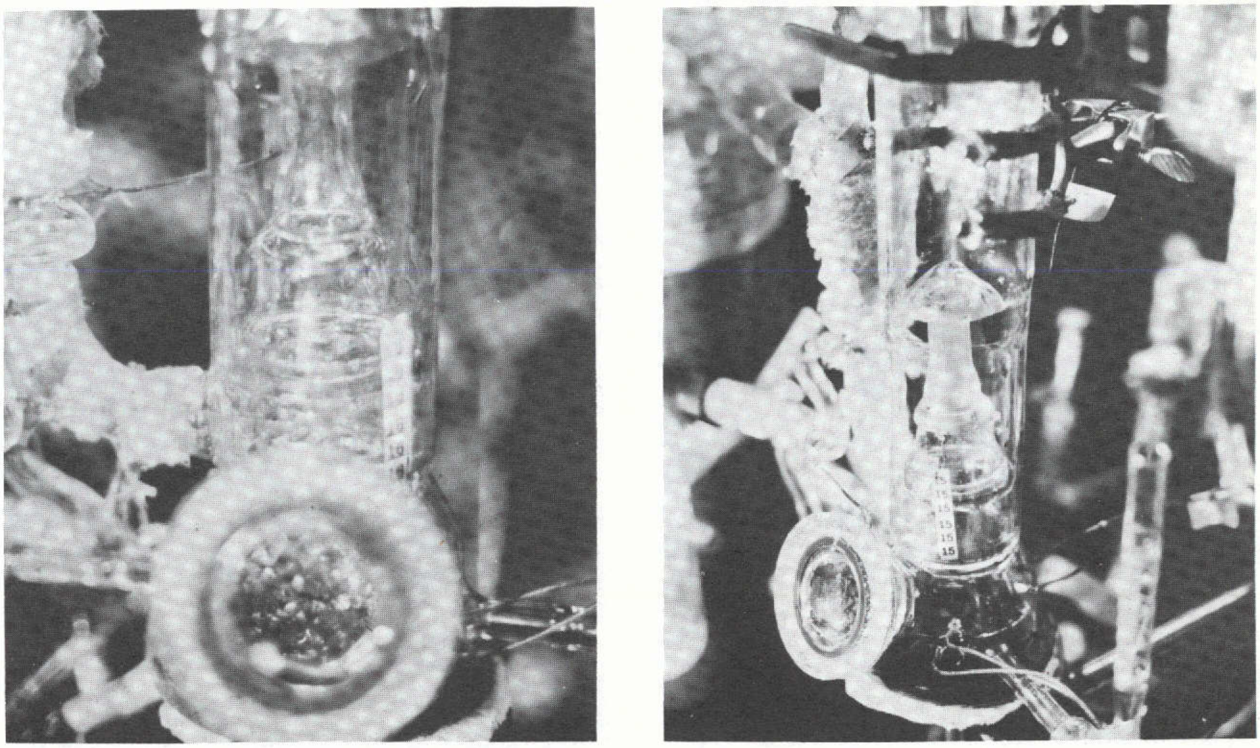


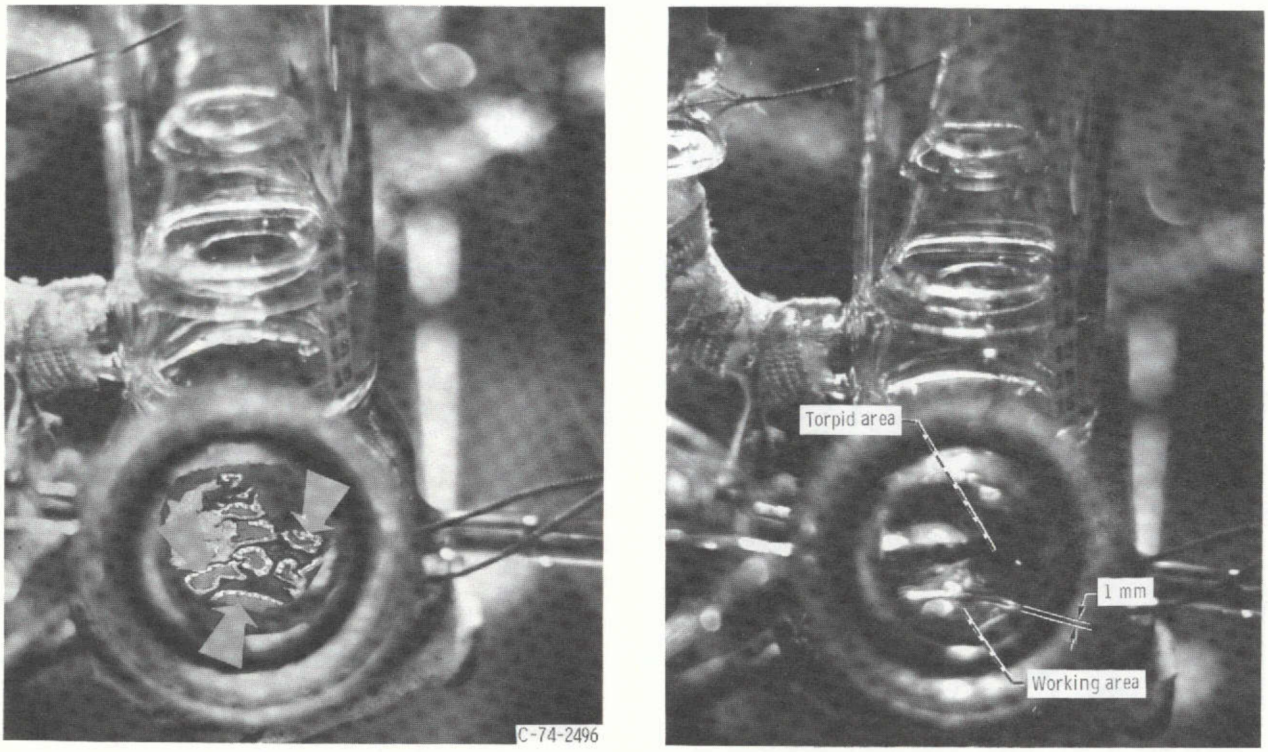
Figure 3. - Present state of the art of large diffusion pump boilers. (The number of heating elements, rings, and conductive fins (or plates) and the boiler geometry may vary with the manufacturer.)



(a) Working.

(b) Torpid.

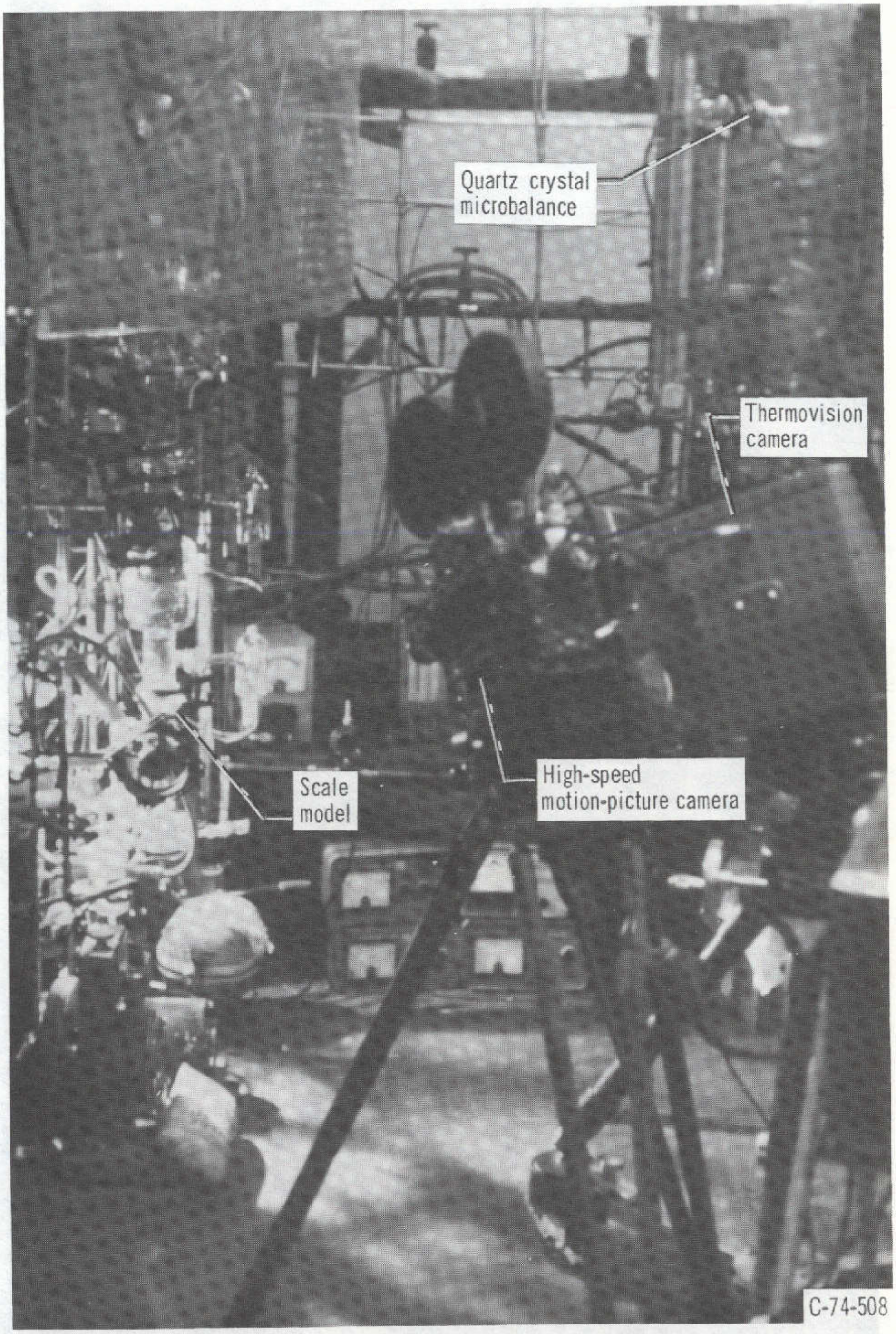
Figure 4. - Typical evaporative surfaces.



(c) Schizoid, illustrating isolated working holes in a torpid fluid field.

(d) Schizoid, illustrating two separate working and torpid areas.

Figure 4. - Concluded.



(a) Setup for tests with self-cleansing, one-millionth-scale, glass model of SPF diffusion pump.

Figure 5. - Typical test setups in Space Power Facility vacuum technology laboratory.

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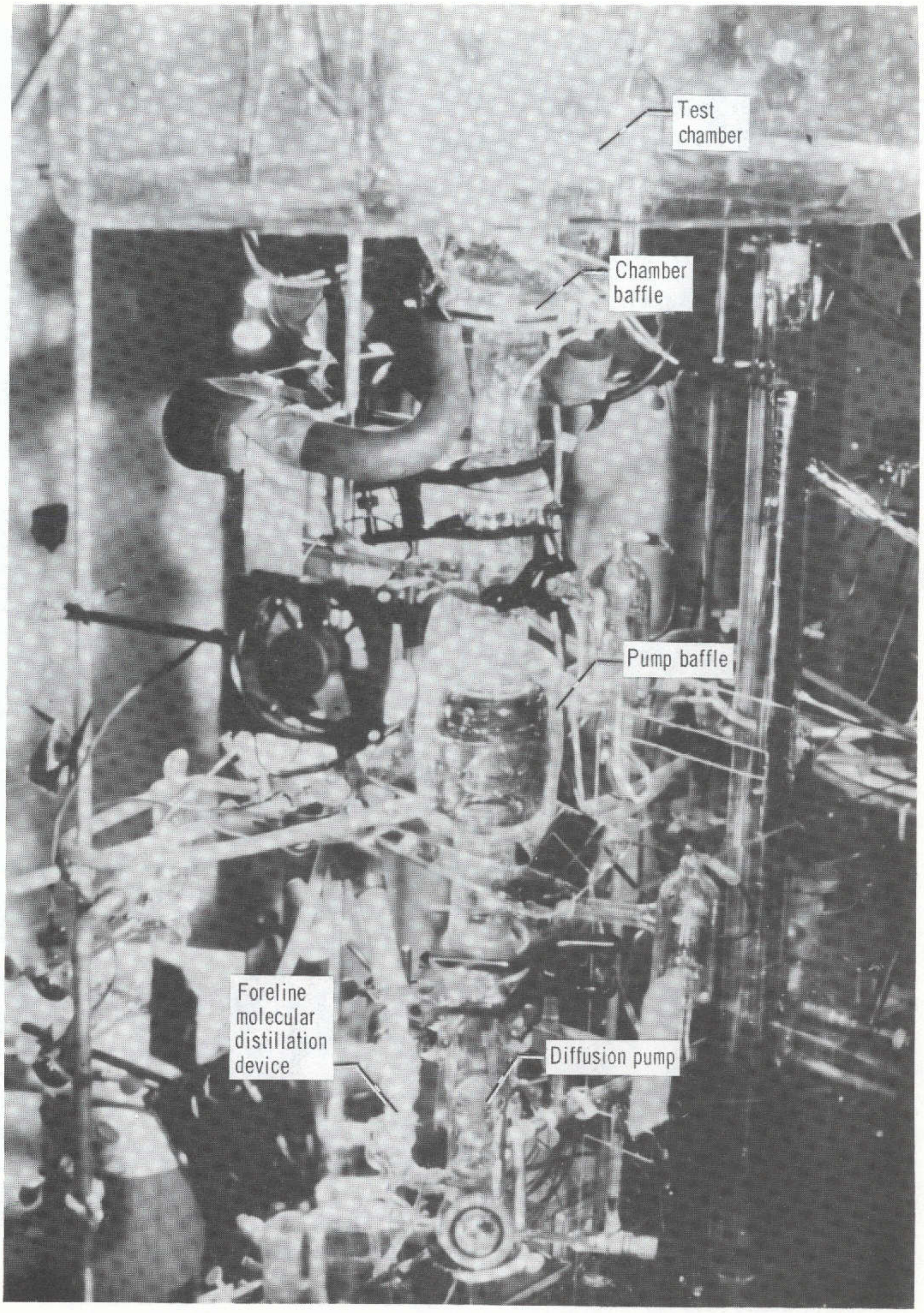
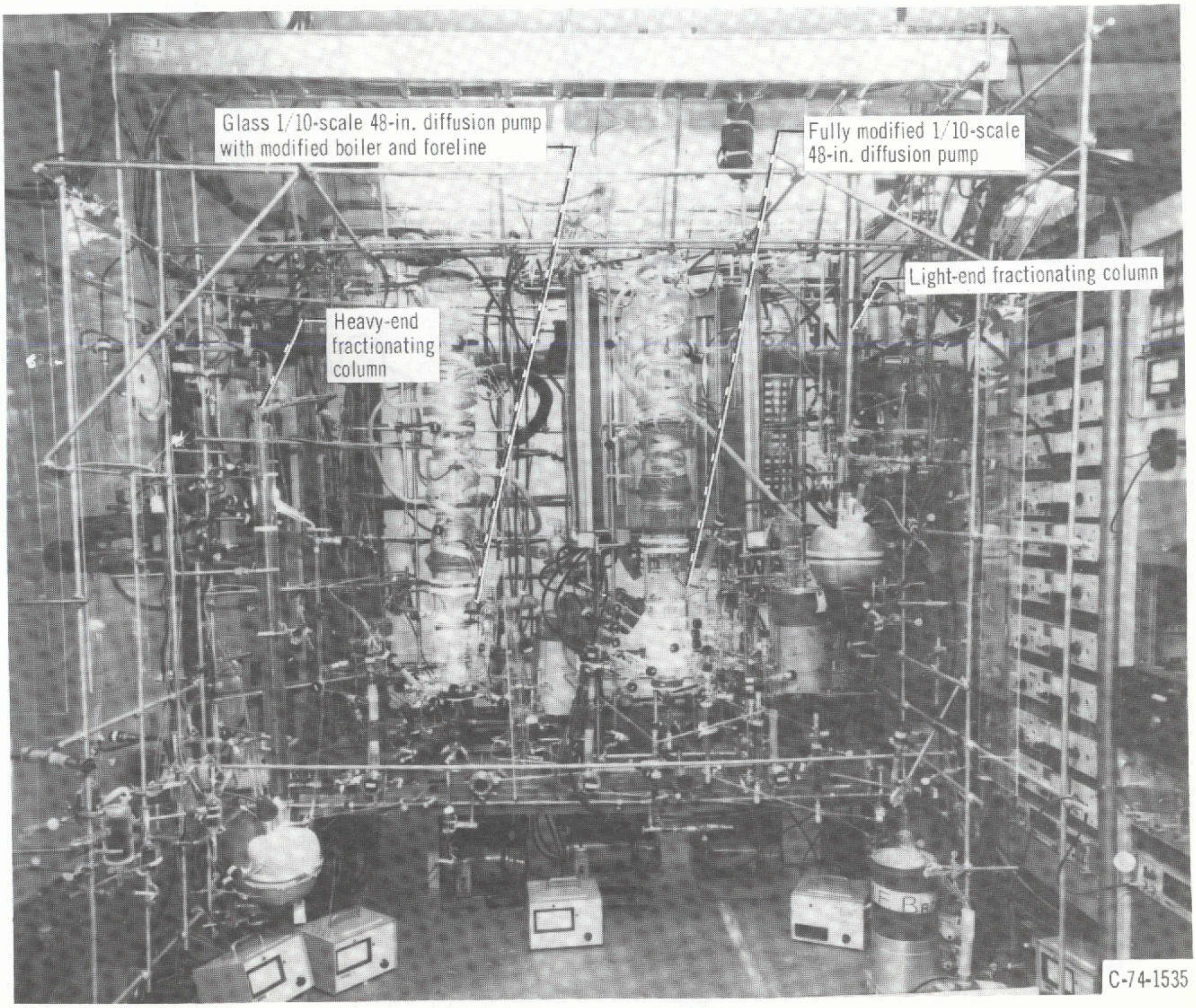


(b) Closeup of self-cleansing scale model.

Figure 5. - Continued.



(c) Final configuration of vacuum technology laboratory.

Figure 5. - Concluded.

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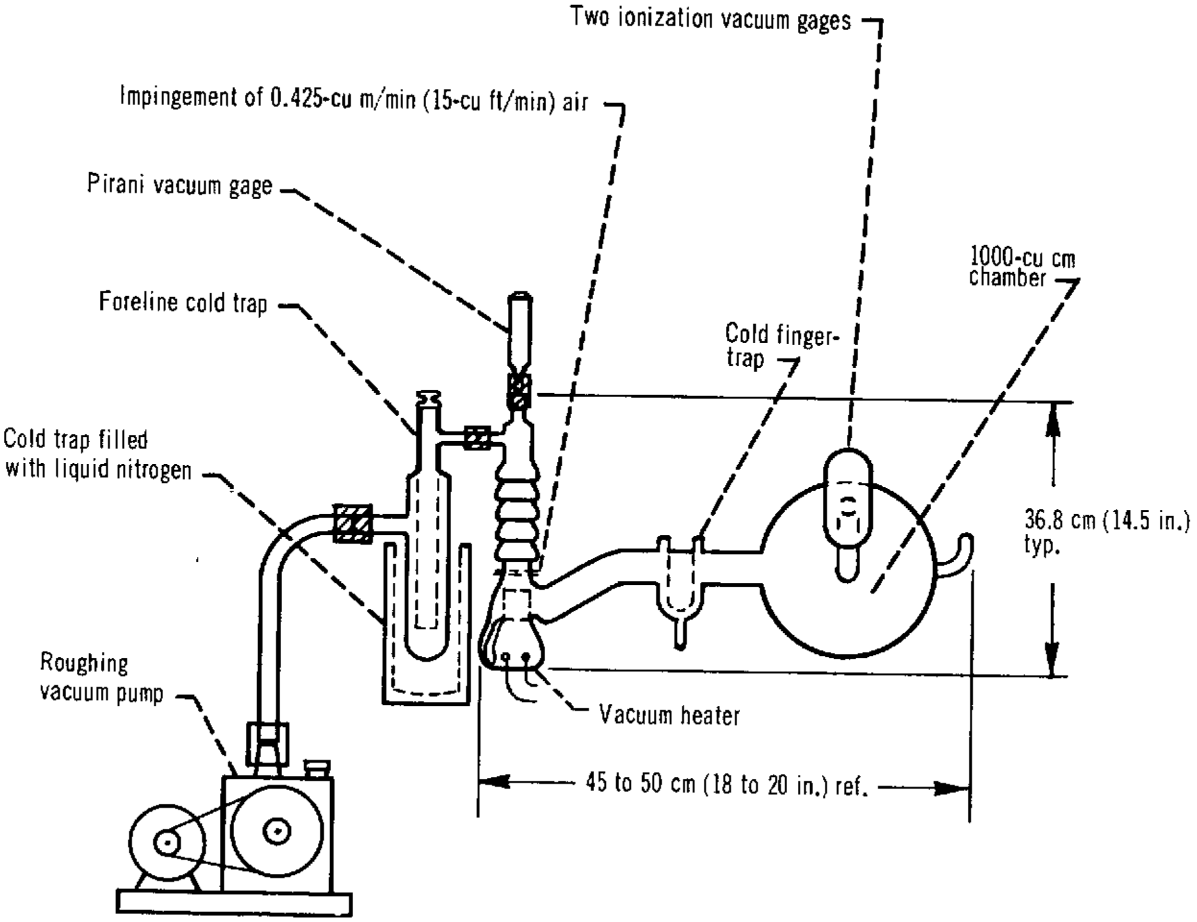

Figure 6. - Typical setup used for evaluation of DC-705 oil performance - G-4, single-stage, glass, diffusion pump vacuum system.

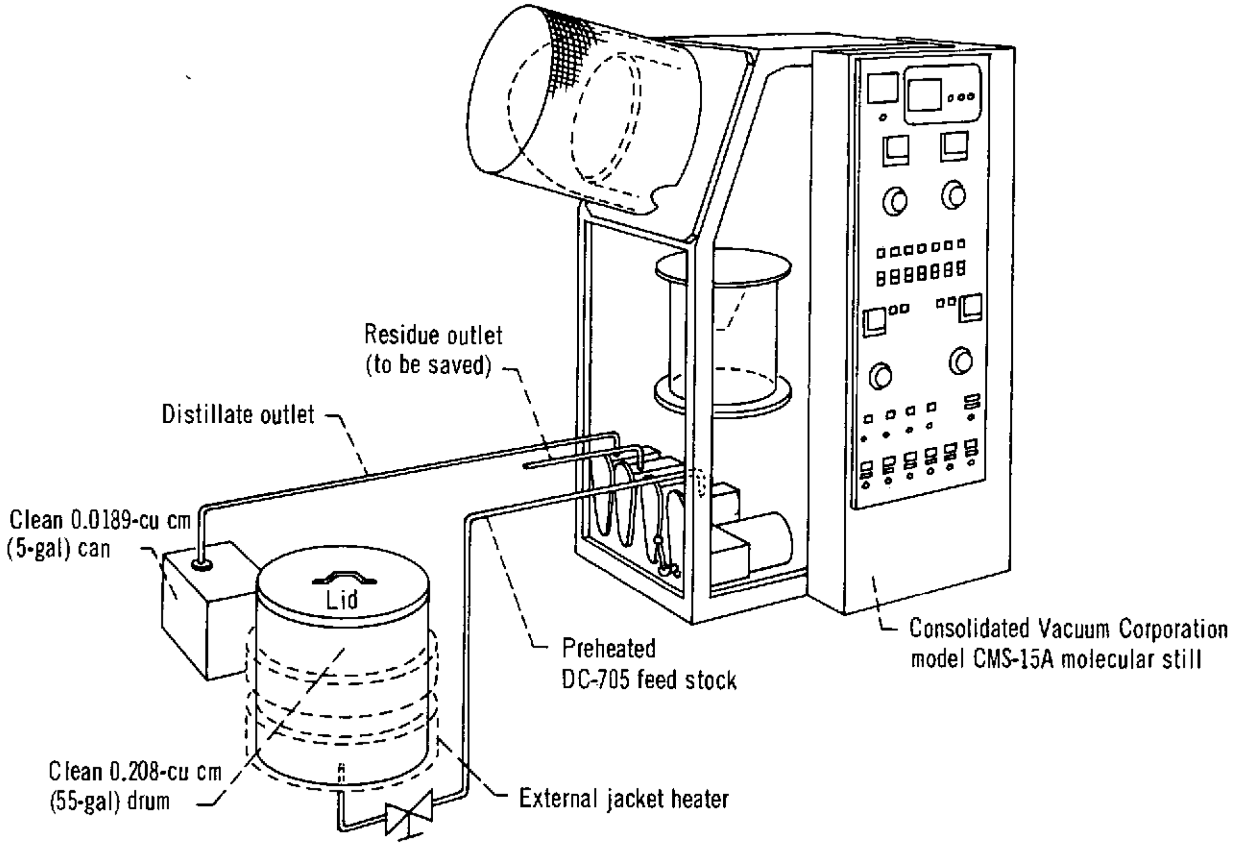

Figure 7. - Typical setup used for reprocessing DC-705 oil.

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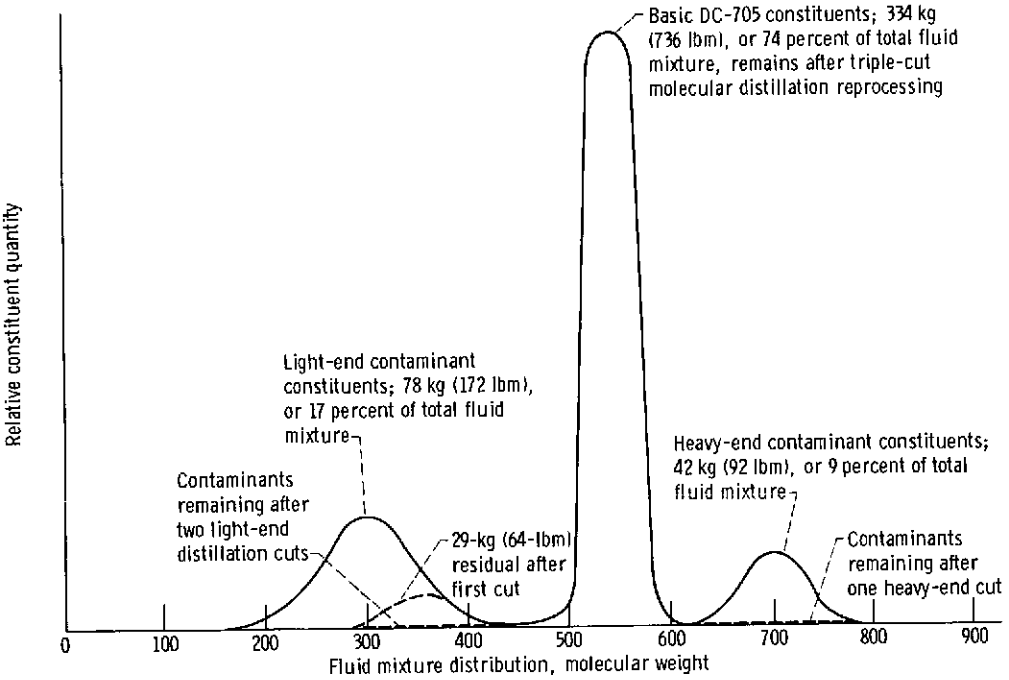


Figure 8. - Contamination removal profile of DC-705 oil.

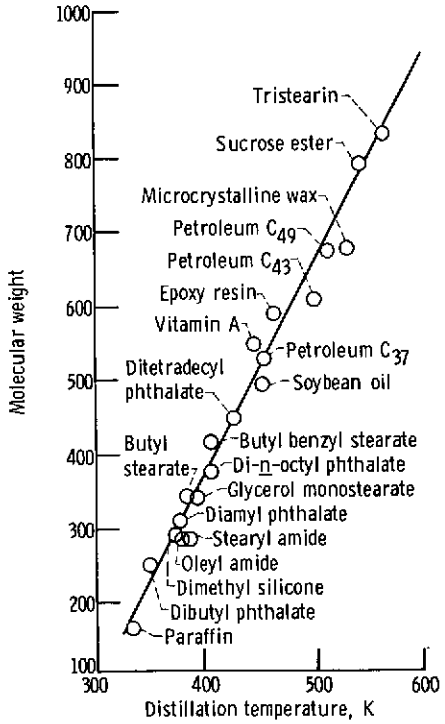
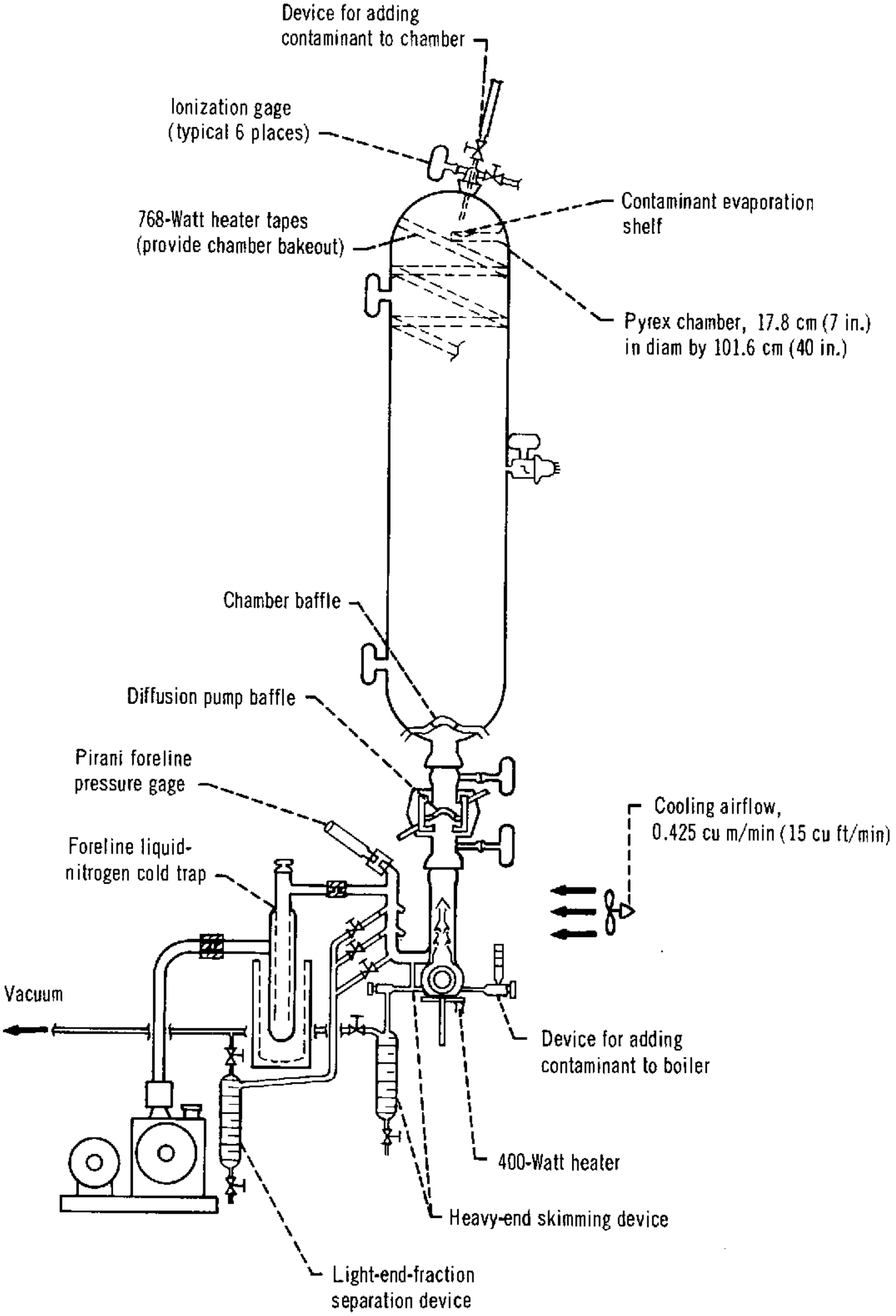
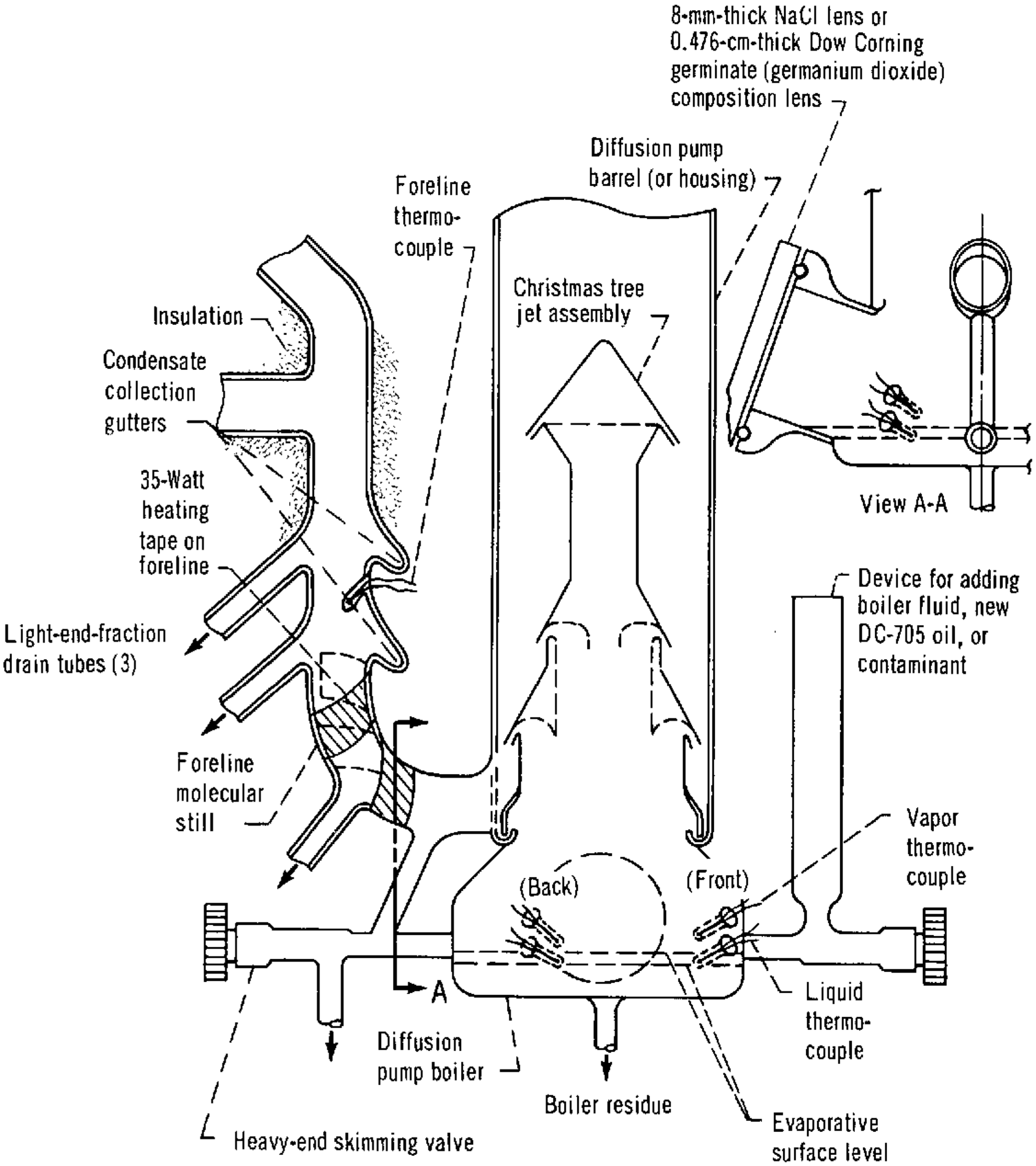


Figure 9. - Relation between molecular weight and distillation temperature for numerous fluids. Pressure, 10^{-3} torr.



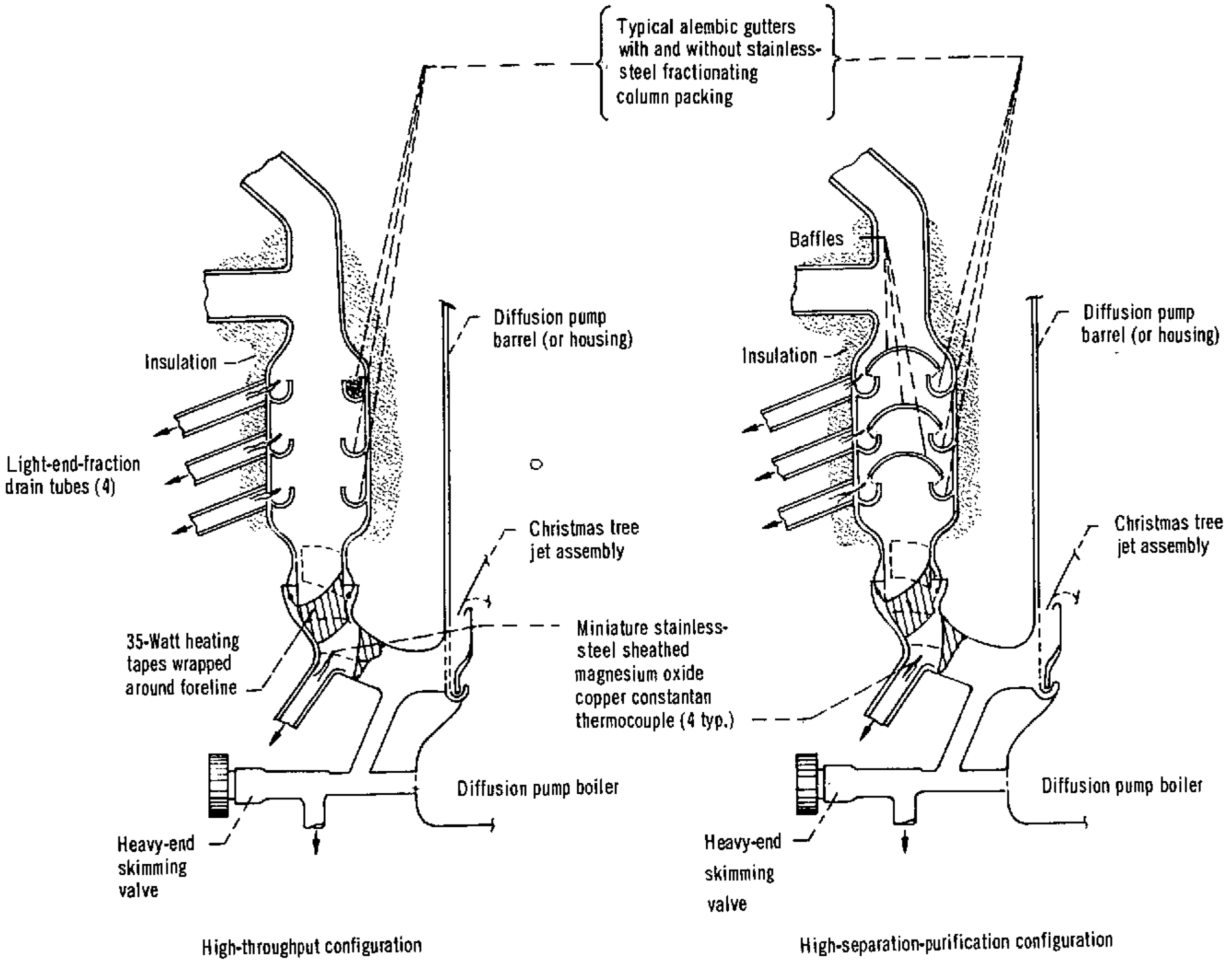
(a) Overall system view.

Figure 10. - Cutaway views of conceptual glass scale-model of a self-cleansing diffusion pump.



(b) Detailed view of pump.

Figure 10. - Continued.



(c) Two advanced foreline molecular still configurations.

Figure 10. - Concluded.

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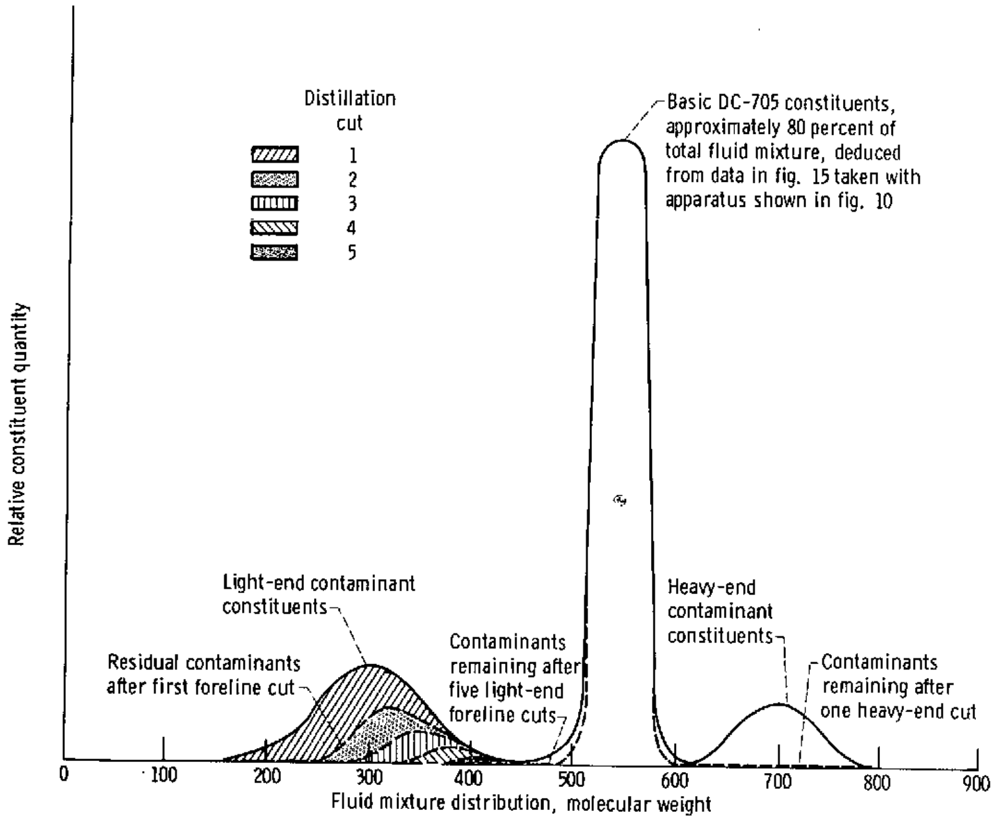


Figure 11. - Contamination removal profile resulting from typical operation of self-cleansing pump.

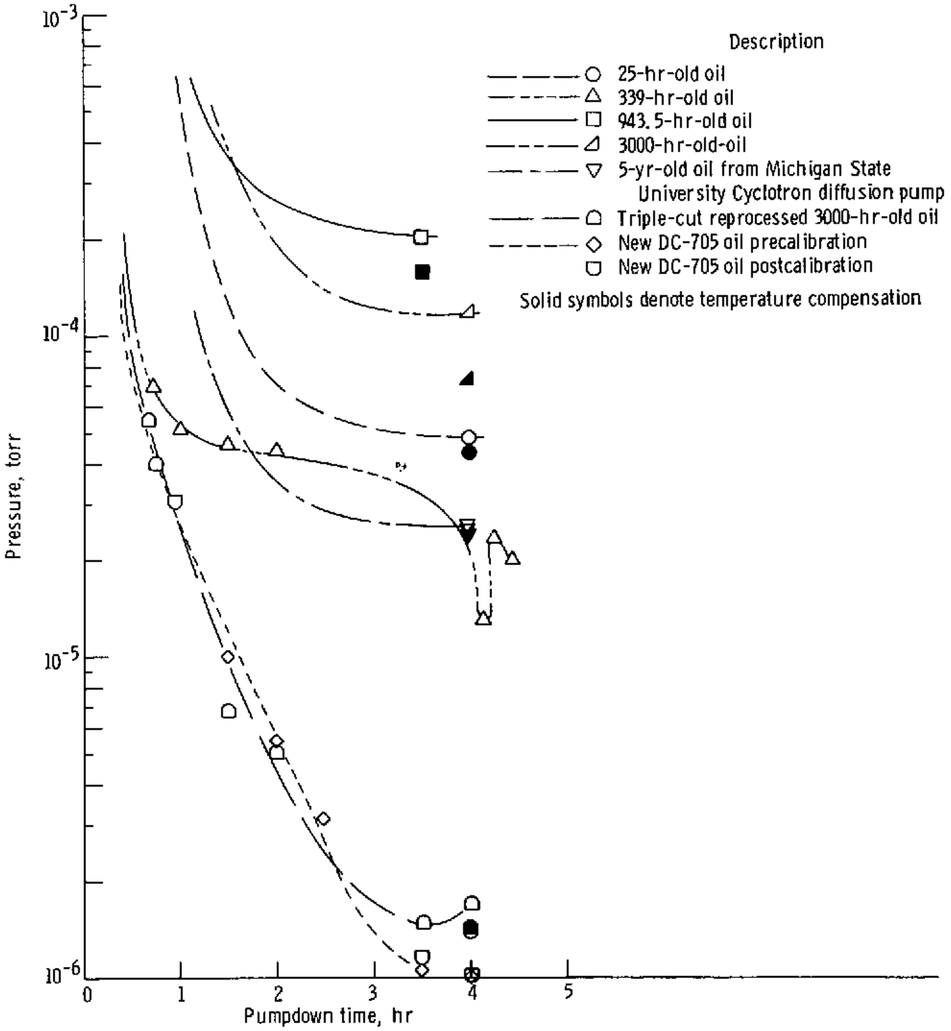


Figure 12. - Performance of used DC-705 oil compared with performance of new and reprocessed oil, in G-4 single-stage glass diffusion pump.

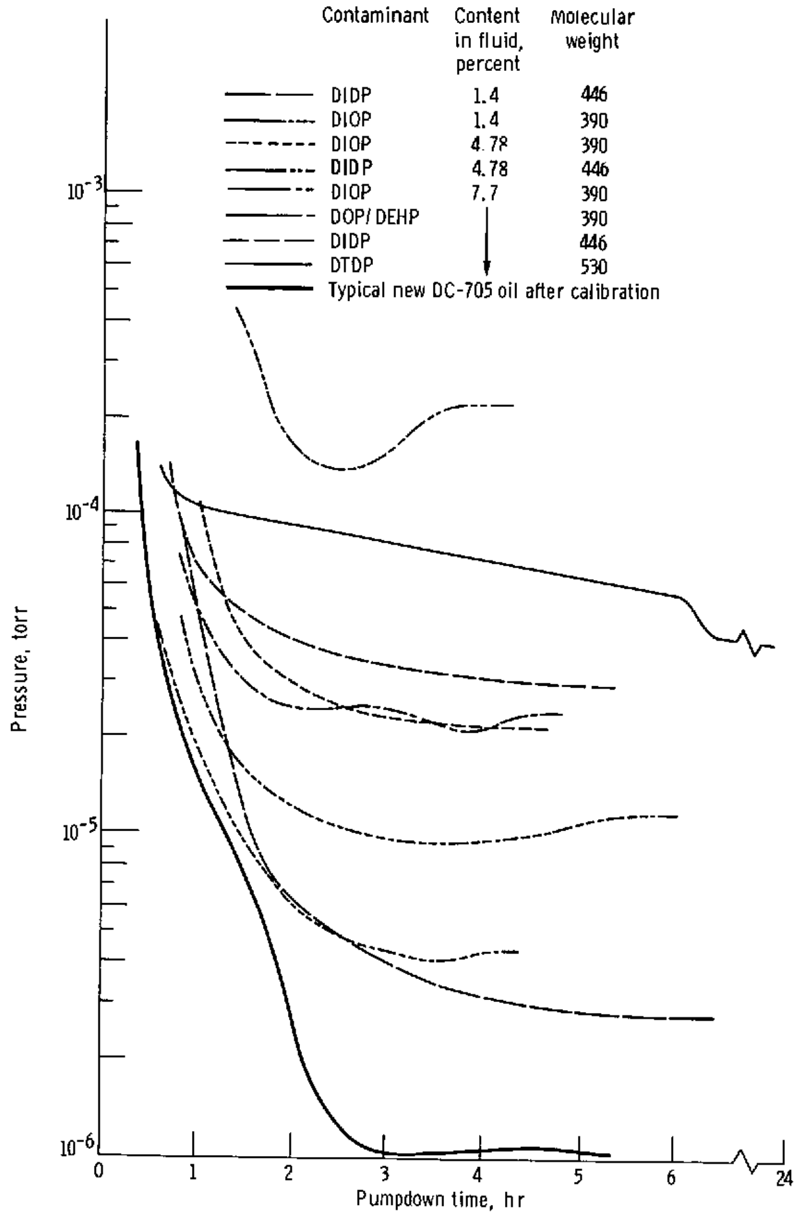


Figure 13. - Performance of DC-705 oil with various contaminants, in G-4 single-stage glass diffusion pump.

Brand name[a]	Chemical type	Molecular weight	Boiling point, K	
			At 0.5 torr	At 0.01 torr
Convoil-10	Hydrocarbon oil	250	418	348
Convoil-20	Hydrocarbon oil	400	463	399
Butyl phthalate	Phthalate ester	278.3	418	354
Octoil	2-Ethylhexyl phthalate (DOP/ DEHP)	390.5	456	399
Octoil-S	2-Ethylhexyl sebacate (EHS)	426.7	472	414
Convalex-10	Polyphenyl esters	454	548	---
DC-704	Tetramethyltetraphenyl-trisiloxane	484	463	418
DC-705	Pentaphenyltrimethyl-trisiloxane	546	518	---

[a] Data extracted from technical bulletins and literature published by Consolidated Vacuum Corporation (CVC) and for Dow Corning Corporation.

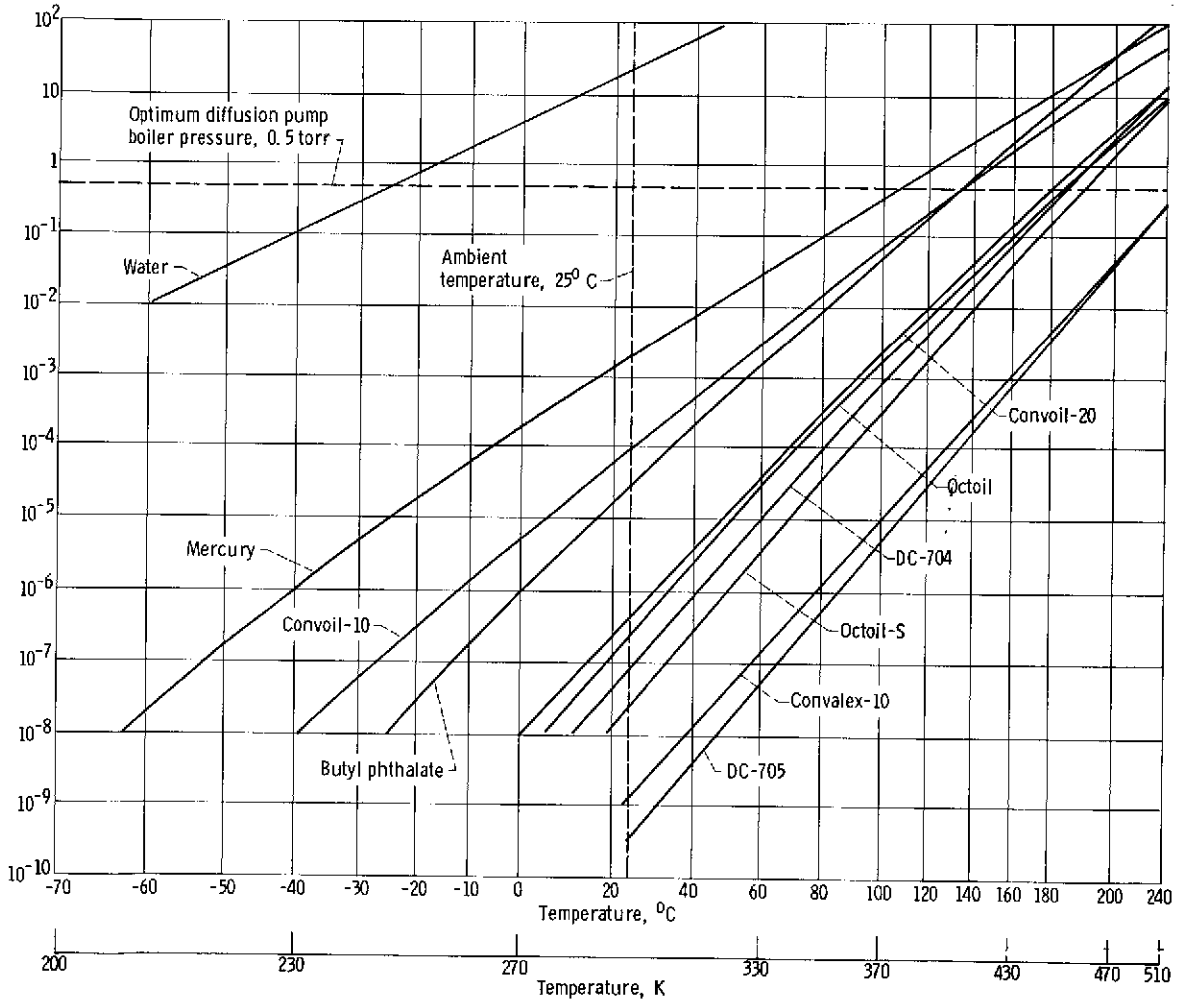
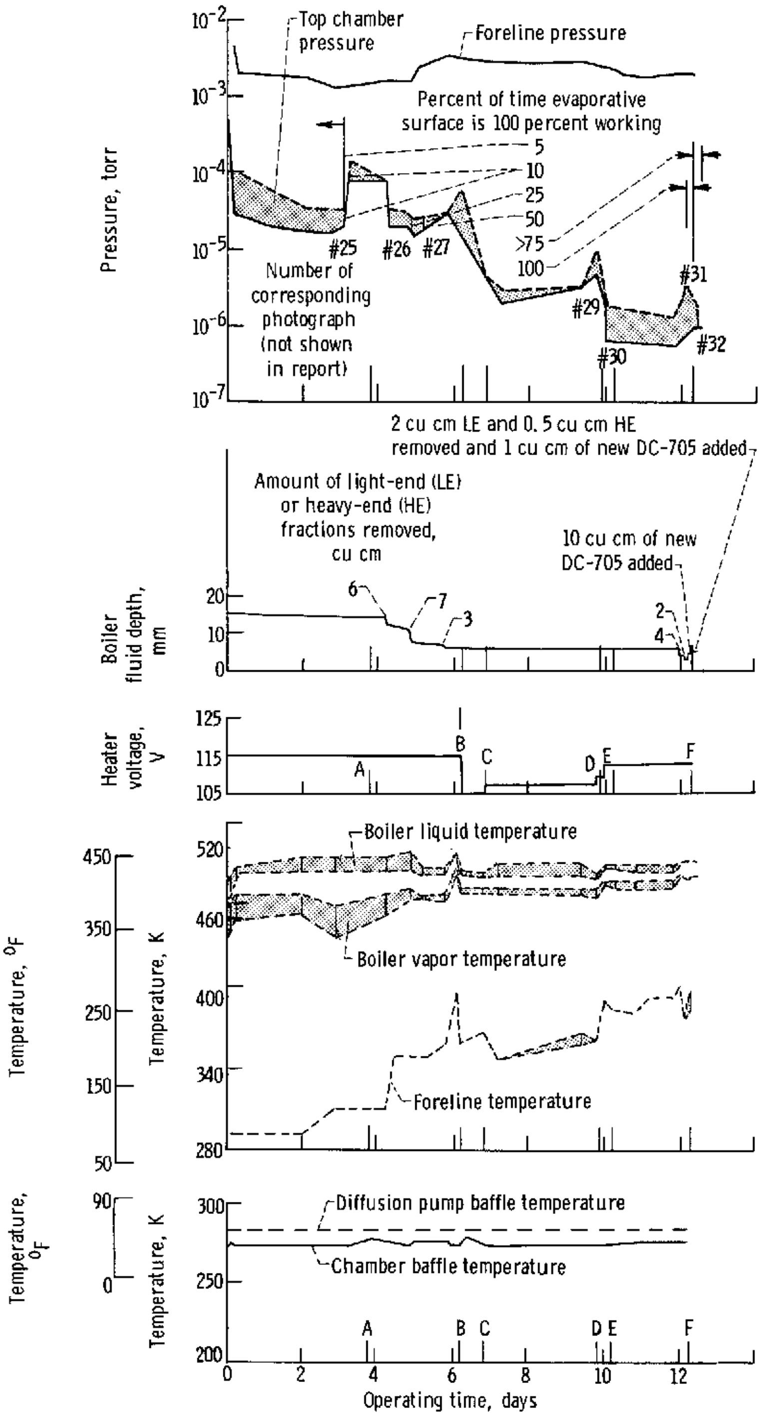
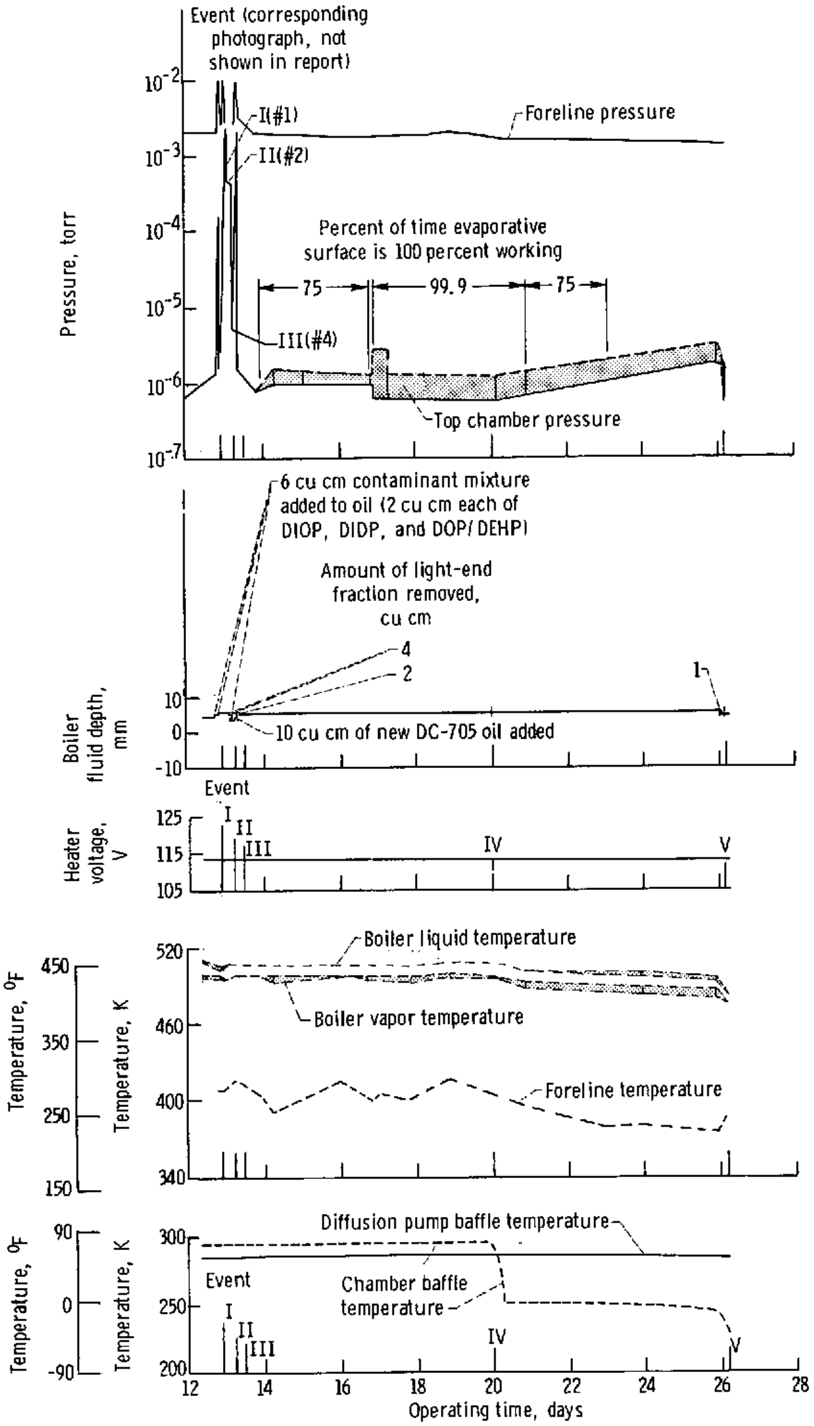


Figure 14. - Pressure-temperature characteristics of some pump fluids.



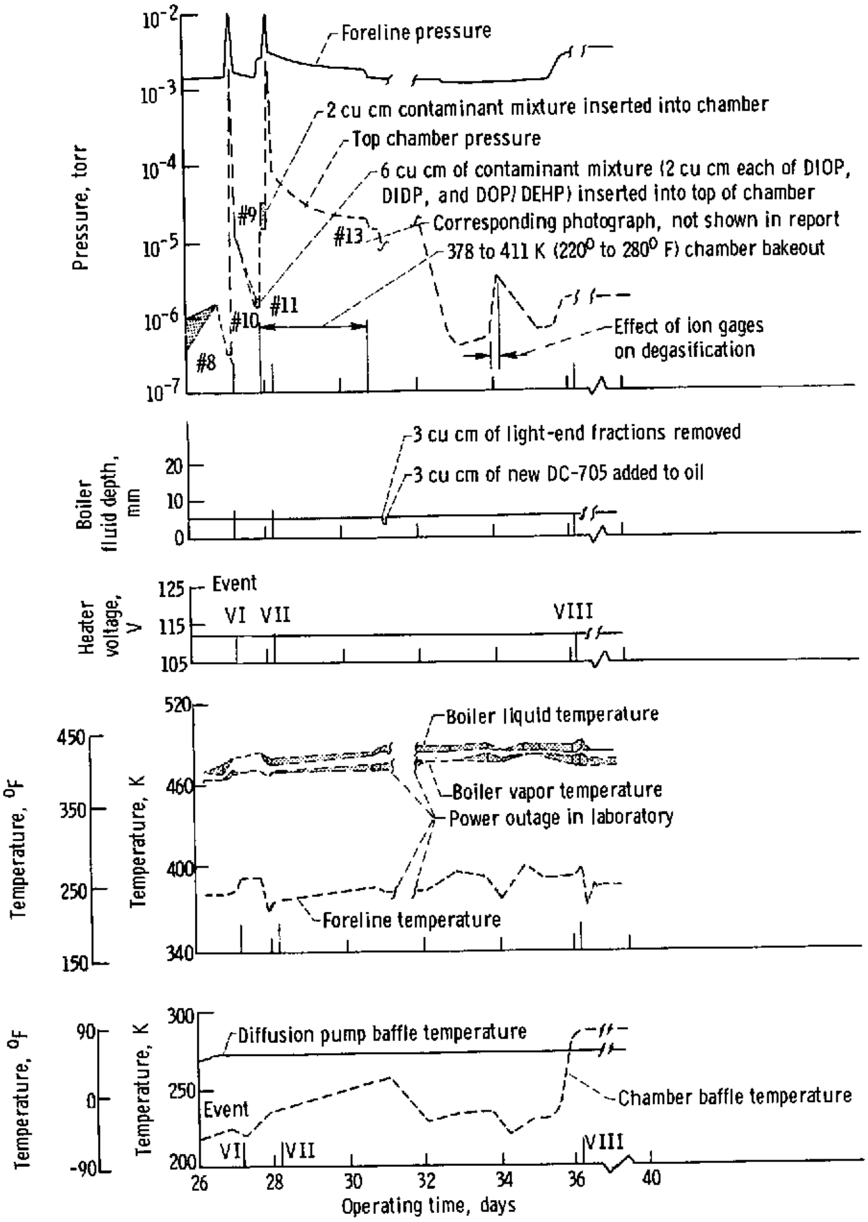
(a) Phase I - increased purification nets 1.5-decade decrease in pressure.

Figure 15. - Performance of new DC-705 oil in one-millionth-scale model of SPF vacuum system.



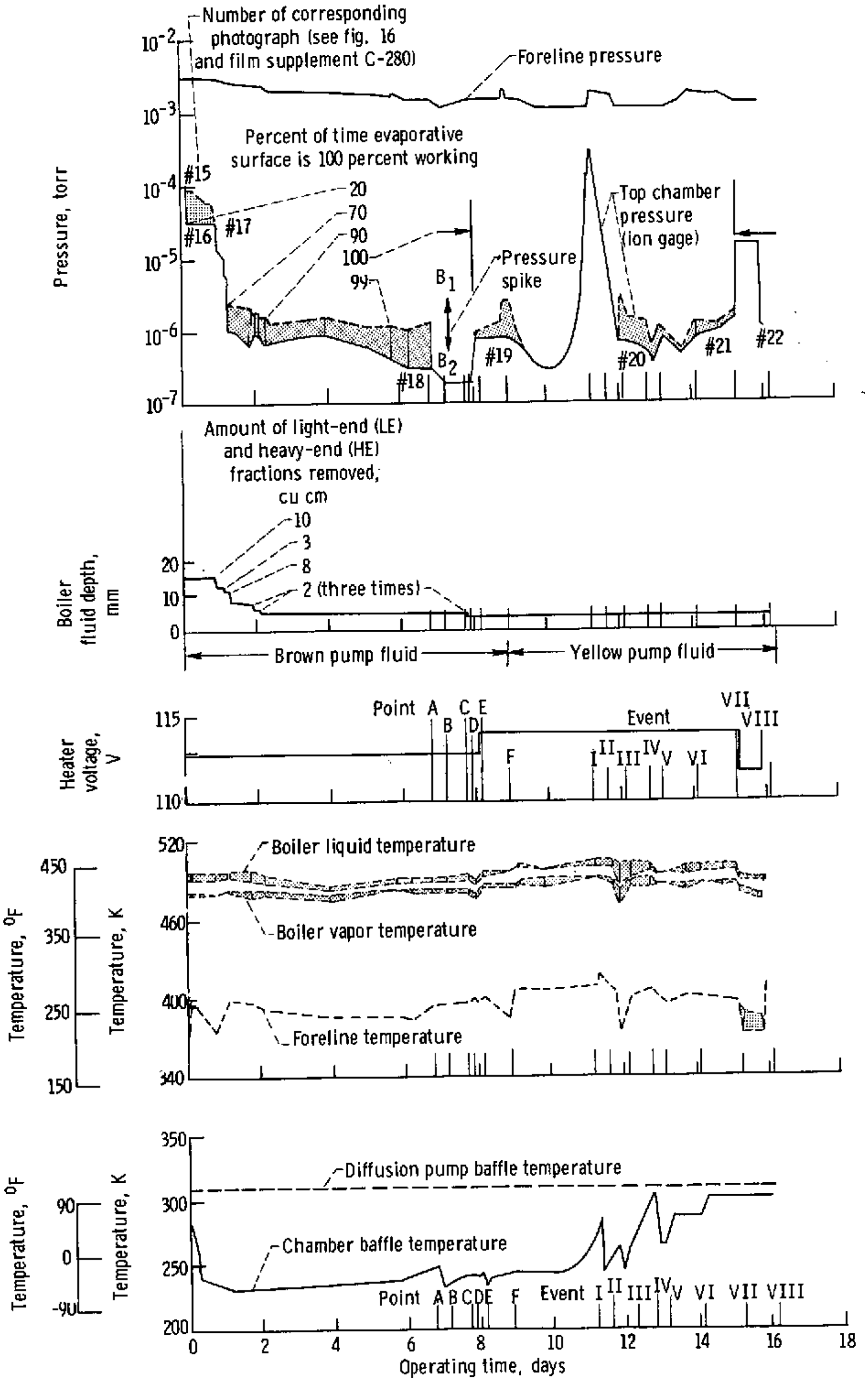
(b) Phase II - effect of plasticizer contaminants added to ultrapure DC-705 and their removal.

Figure 15. - Continued.



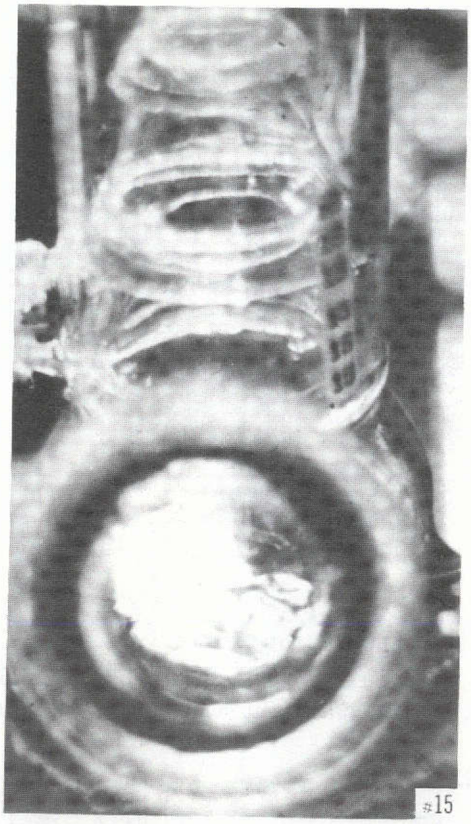
(c) Phase III - effect of plasticizer contaminants inserted into chamber and their removal by cold and warm baffles.

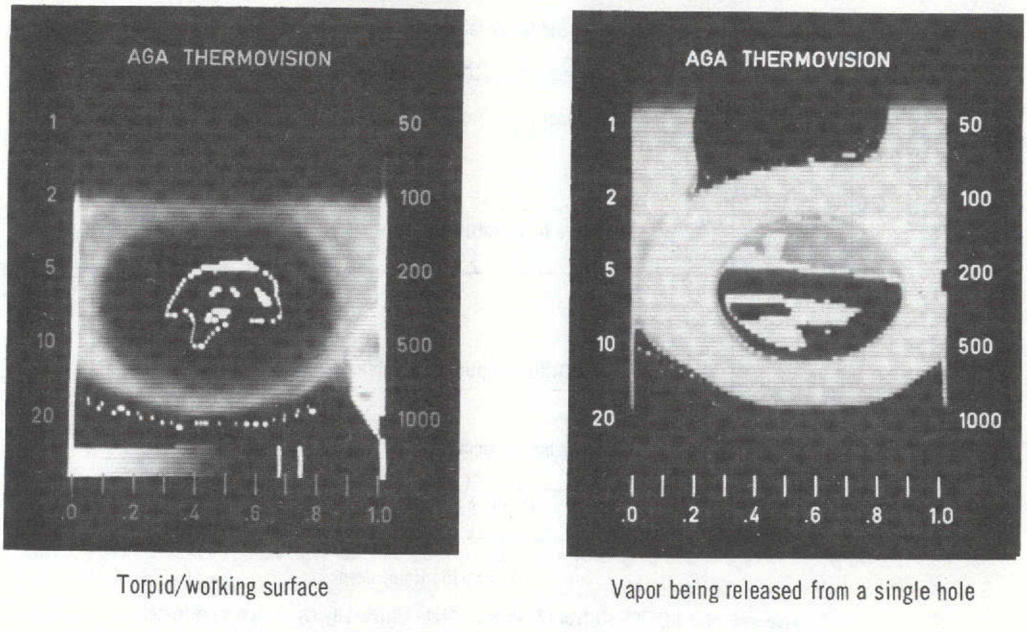
Figure 15. - Continued.



(d) Five-year-old DC-705 oil from Michigan State University Cyclotron run in self-cleansing, one-millionth-scale, glass model of SPF diffusion pump.

Figure 15. - Concluded.



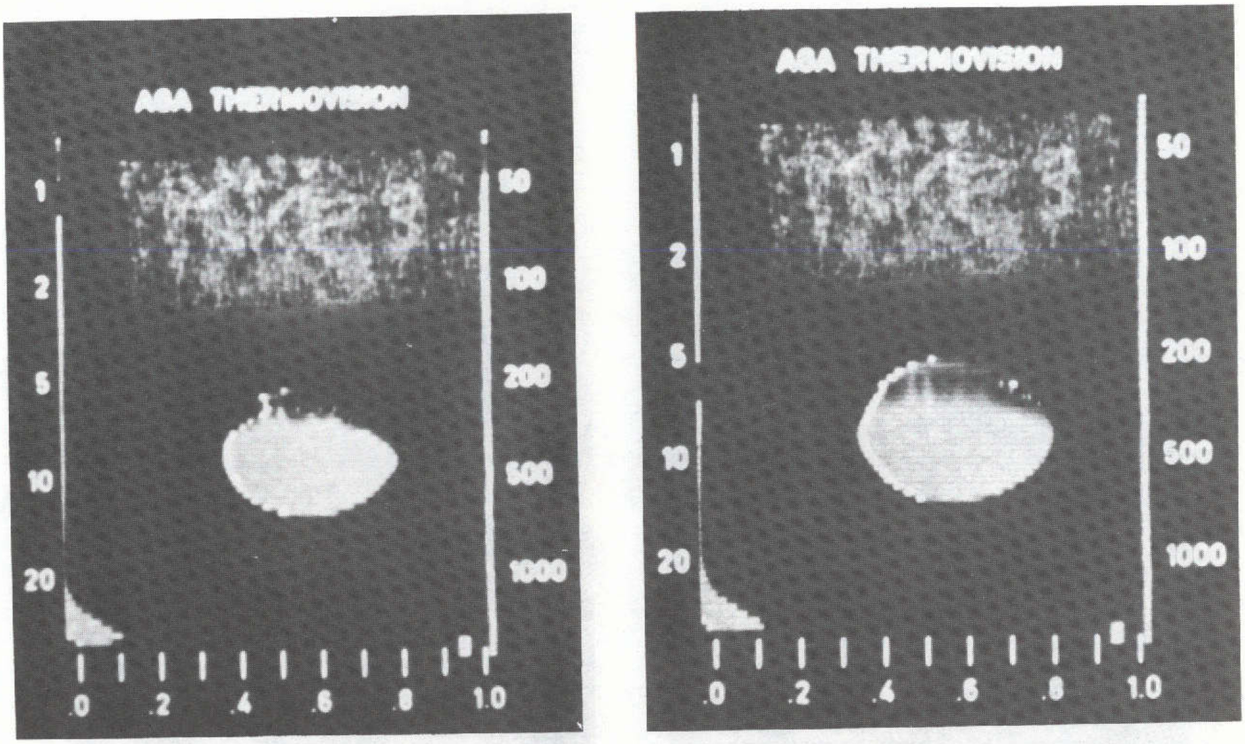


Torpid/working surface

Vapor being released from a single hole

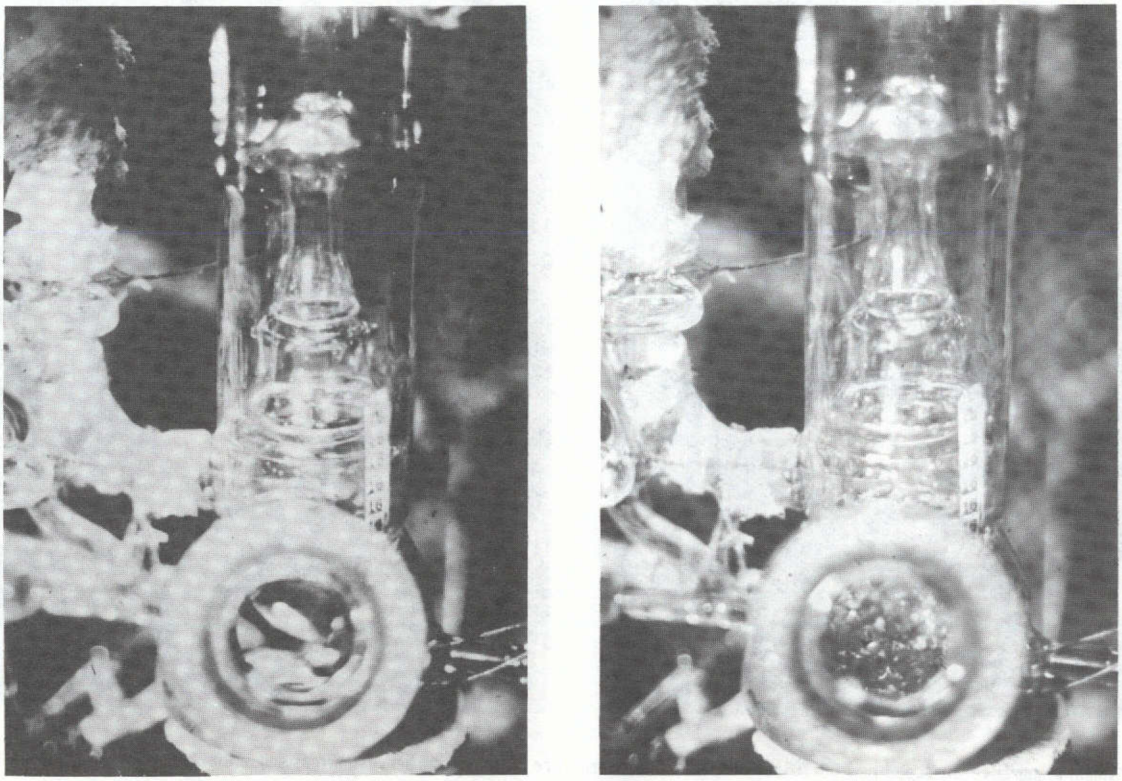
(a) Typical schizoid (torpid/working) evaporative surface of 5-year old DC-705 oil from Michigan State University Cyclotron at 2×10^{-4}-torr pressure at top of chamber - shown as still photograph and thermovision images.

Figure 16. - Typical schizoid (torpid/working) and 100-percent-working evaporative surfaces.



(b) Typical evaporative surface of new DC-705 oil with 2×10^{-4} -torr pressure at top of chamber - shown as sequential frames of 16-mm motion picture of thermovision monitor.

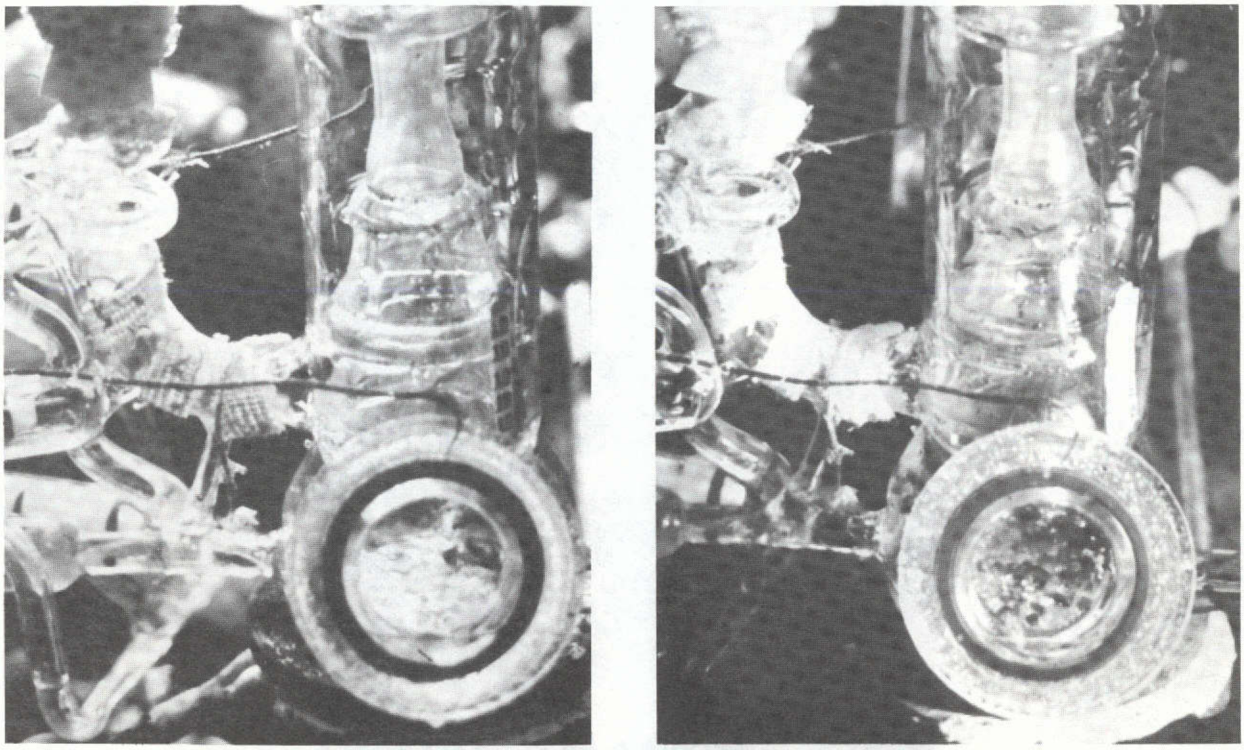
Figure 16. - Continued.



(c) Schizoid (torpid/working) evaporative surface with 5×10^{-5}- to 10×10^{-5}-torr vacuum at top of chamber. #16.

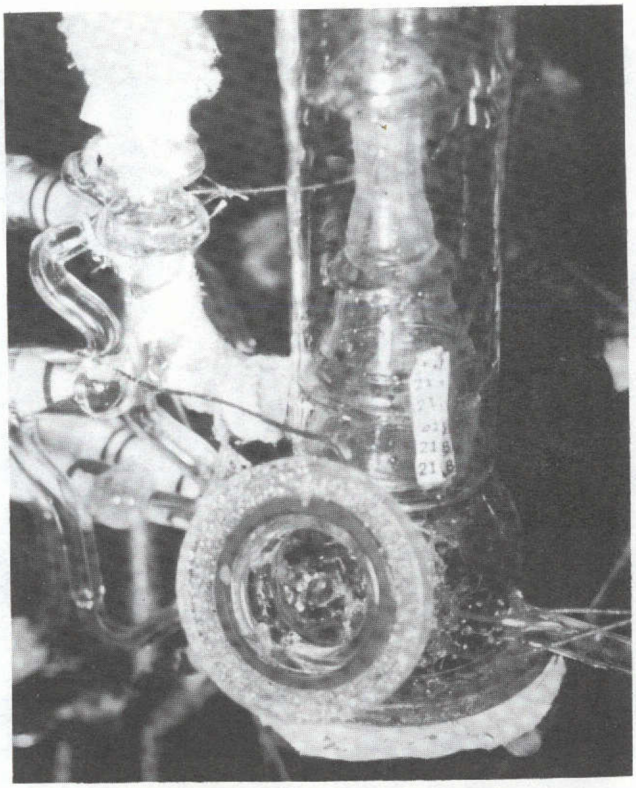
(d) 100-Percent-working evaporative surface (20 percent of the time) with 5×10^{-5}-torr vacuum at top of chamber. #16.

Figure 16. - Continued.



(e) Continuous 100-percent-working evaporative surfaces with 1.5×10^{-6}- to 4×10^{-6}-torr vacuum realized at top of chamber with baffle temperatures of 294 K (70° F) at pump and 242 K (-25° F) at bottom of chamber. #19.

Figure 16. - Continued.



(f) Continuous 100-percent-working evaporative surface with 1×10^{-6}-torr vacuum at top of chamber. Twenty to 30 percent of boiler bottom appears bare due to vapor recoil action. #21.

Figure 16. - Concluded.

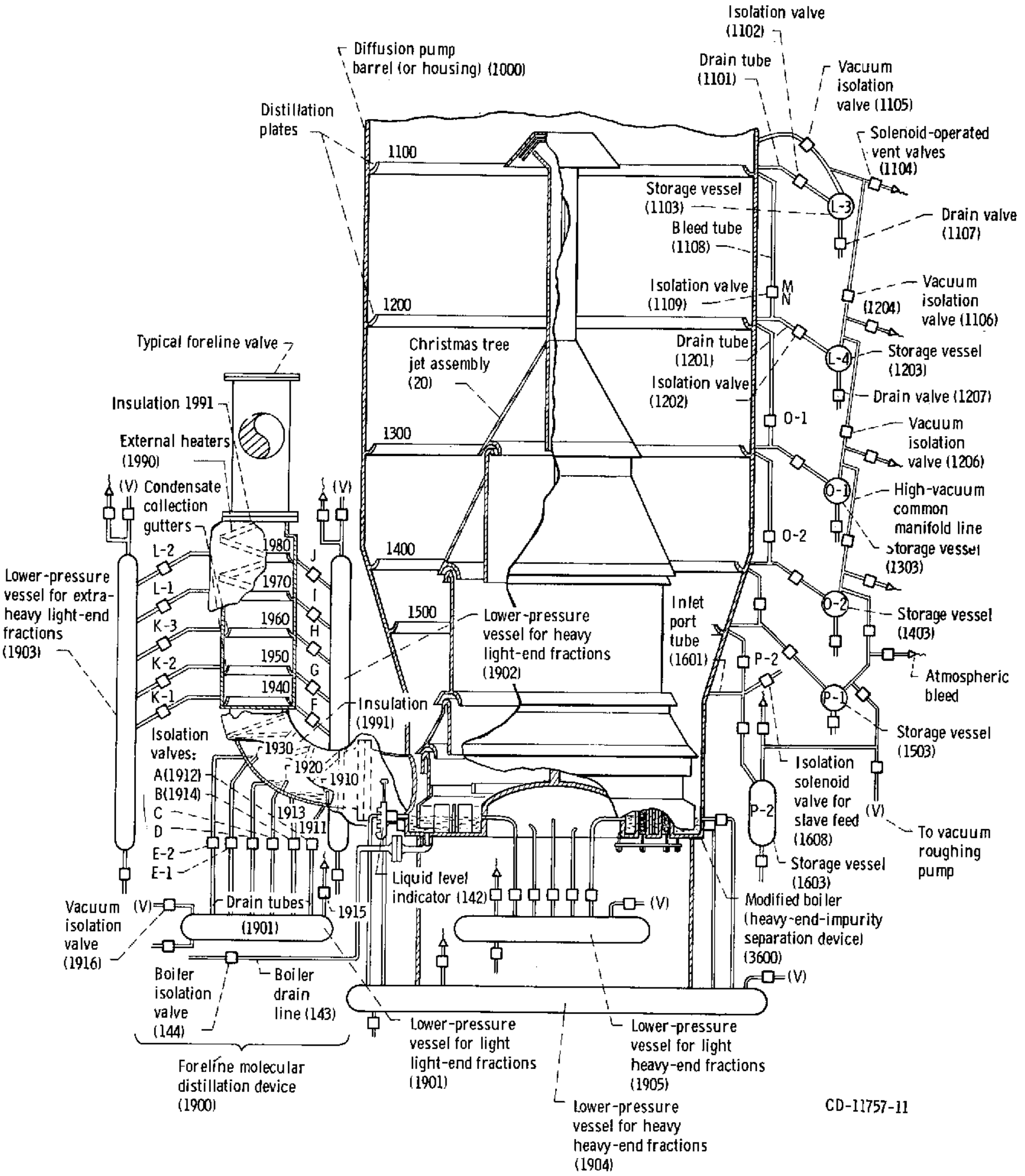


Figure 17. - Cutaway view of large self-cleansing diffusion pump with ultrapurification-separation innovations.

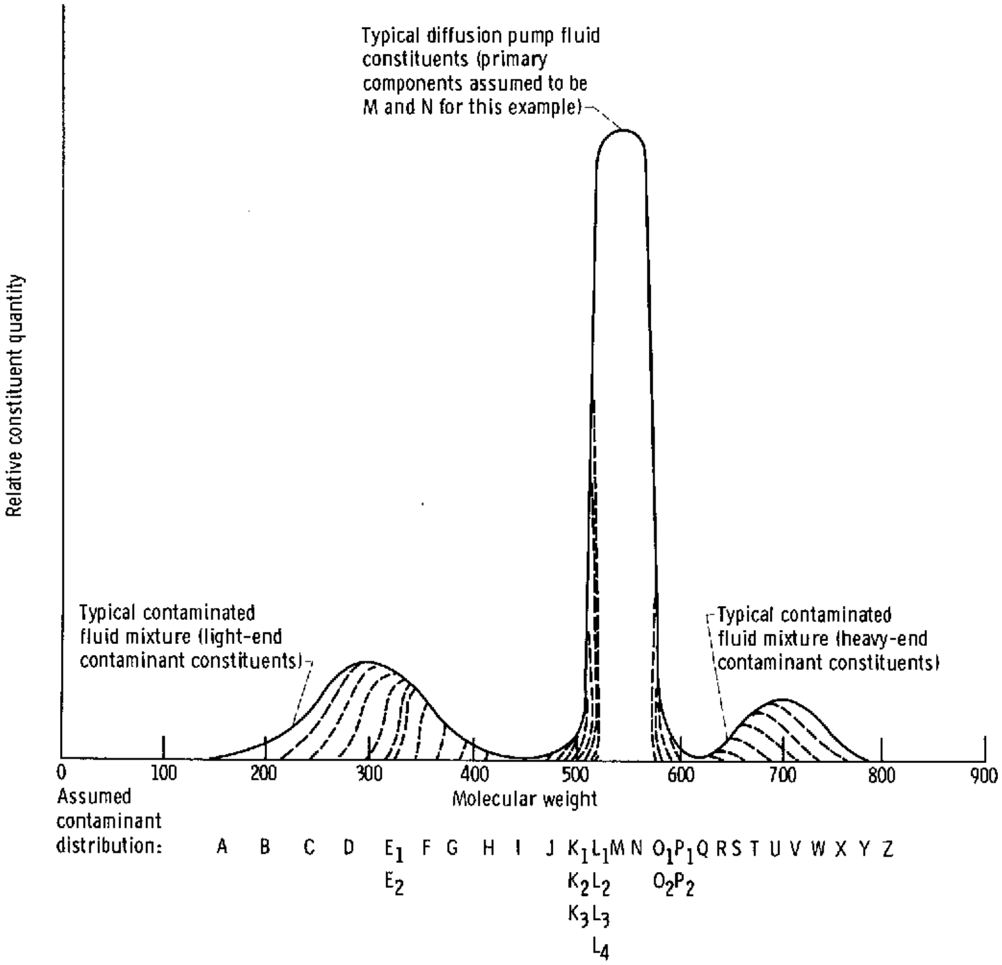


Figure 18. - Typical contamination-removal profile illustrating diffusion pump fluid purification process in self-cleansing pump.

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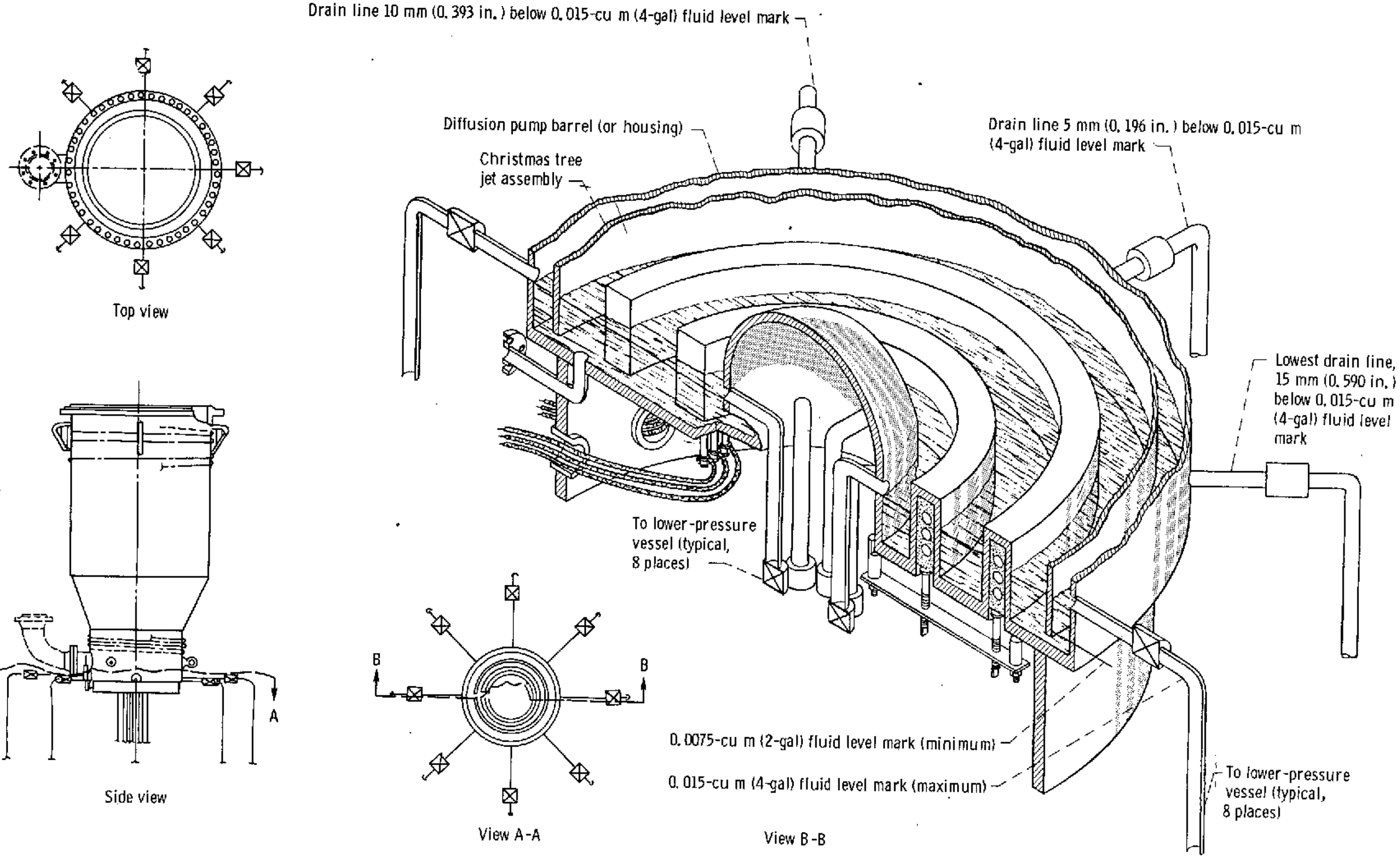

Figure 19. - Large self-cleansing diffusion pump boiler.

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