THE REDESIGN AND RECONSTRUCTION OF A FIVE-INCH MAGNETICALLY-DRIVEN CENTRIFUGAL MOLECULAR STILL

by

Julian F. Mote III

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Approved:

_____
In Charge of Investigation

_____
Head of Major Department

Virginia Polytechnic Institute
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I. INTRODUCTION

Molecular distillation is one form of high vacuum, short-path distillation. The three basic requirements for unobstructed, short-path distillation are: an evaporating surface over which a thin film of distilland is distributed, a condensing surface, and a suitable system for evacuating the space between the two surfaces. When the distance between the evaporating and condensing surfaces is comparable to the mean free path of the vapor molecules in the residual gas, the process is known as molecular distillation.

This relatively new process is now an industrial reality and can be economically applied to the purification and separation of heat-sensitive, high-boiling materials which cannot be separated in conventional stills. The use of the centrifugal molecular still permits the distillation of organic substances which suffer thermal degradation even under the conditions of the conventional high vacuum still. Included among these materials are the so-called "undistillables," which include the natural fats and waxes, sugar derivatives, petroleum residues, and dyes.

The development of the molecular still has passed through three prominent stages; from the crude pot still developed in 1922, to the falling-film type still developed in 1935, and then to the much improved but still very inflexible centrifugal molecular still developed in 1944 by Hickman. Advancement and development to the present day commercial stills has largely come about since that date. Despite the many improvements, however, the centrifugal stills have continued to remain poor in separatory powers and are thermally inefficient. In addition, they are too compact in construction and too inflexible in design to permit proper study of the fundamental factors affecting their performance. One of the major problems in the construction of the centrifugal stills, yet to be solved satisfactorily, is the prevention of leaks through the bearings and packing of the rotor shaft.

The purpose of this investigation was to redesign and reconstruct a five-inch magnetically-driven centrifugal still, with the following objects in view: (1) to eliminate leaks around the rotor shaft by constructing a magnetically driven rotor, completely sealed within the still head and having no direct connections between the rotor shaft and the drive shaft of the motor; and (2) to gain flexibility

in the operation of the still by the use of an enlarged bell jar and the addition of a water-cooled condenser within the bell jar.

II. LITERATURE REVIEW

A review of the literature on molecular distillation is presented in this section under the headings of terminology, theory and application, and molecular still operation.

Terminology

Since molecular distillation is considerably different from ordinary distillation, explanation and definition of terminology is desirable for the sake of clarity.

Distillation. In general, distillation is the term applied to vaporization processes in which the vapor evolved is recovered, usually by condensation. For emphasis of differentiation between the various applications of the term, the following distillation methods are considered.

Conventional Distillation. According to Carey[24], "Distillation is the separation of the constitutents of a liquid mixture by partial vaporization of the mixture and separate recovery of vapor and residue. The more volatile constitutents of the original

mixture are obtained in increased concentration in the vapor; the less volatile in greater concentration in the liquid residue." Distillation is a term properly applied only to those operations where vaporization of a liquid mixture yields a vapor phase containing more than one constitutent, and it is desired to recover one or more of these constitutents in a nearly pure state.

Unobstructed-Path Distillation. Hickman[42] has made a differentiation between two types of distillation which may occur in a high vacuum, but does not quantitatively define high vacuum. The process of unobstructed-path distillation is described as the process of free transfer of molecules under high vacuum from evaporator to condenser.

Molecular Distillation. The second type of free transfer of molecules under high vacuum from evaporator to condenser is a limitation of the process just described. When the distance of transfer from evaporator to condenser is comparable to the mean free path of the vapor molecules in the residual gas the process is known as molecular distillation[42].

Molecular or non-equilibrant distillation received its name from the fact that vaporization is carried

> out in a vacuum so nearly perfect that "almost" every molecule travels toward the condensing surface unhindered by the presence of other molecules with a minimum of return to the main body of liquid. In high vacuum distillation, only the molecules at the evaporating surface enter into the process and at any given instant, all other molecules in the bulk of the distilland layer need not be considered as part of the distilling process[1].

Basic Differences in Distillation Procedures[24]. In conventional distillation processes, distillation begins at a well defined temperature, there is a definite boiling point of the material being distilled, and the distillatin process is accompanied by ebullition of the distilland. A dynamic equilibrium exists between the liquid and the vapor phases thus causing a large proportion of the molecules evaporating from the liquid surface to return to the same liquid surface.

In contrast, in high-vacuum distillation processes, there is no well defined temperature at which distillation begins. Distillation occurs at any temperature as long as a thermal gradient exists between the condenser and the evaporator. Due to the high vacuum maintained during the process, there is effectively no superincumbent air pressure

on the material being distilled and consequently there is no well defined boiling point or attending ebullition of distilland. The distilling vapor molecules pass directly from the vaporizing surface to the condensing surface without having to pass through a barrier of air molecules, thus the number of distilling molecules returning to the liquid is therefore considered insignificant. As a consequence, a dynamic equilibrium between vapor and liquid phases does not exist during high-vacuum distillation. Ideal distillation conditions are attained when the number of molecules being condensed is equal to the number of molecules of the distilland evaporating from the distilland surface.

High Vacuum. Anderson[1] has suggested that the term "high vacuum" need be given a more restrictive meaning than in ordinary usage. The term "high vacuum" should be considered to exist only when the pressure is sufficiently low so that the mean free path of the vapor molecules in the residual gas is of the same order of magnitude as the size of the container.

The units of pressure adopted in high vacuum work are millimeters of mecury and fractions thereof and the micron. Molecular distillations are generally conducted[35] at pressures of 1 to 7 microns, with about 3 microns providing the economic level in industrial applications.

Distilland. In distillation processes, and in molecular distillation in particular, the feed mixture to be distilled is referred to as the "distilland."

Residue. The undistilled portion of the distilland is referred to as the "residue."

Distillate. That portion of the distilland removed as vapor and condensed is referred to as the "distillate" product.

Theory and Application

Although molecular distillation was, until only recently, a laboratory technique used exclusively for research purposes, it has in the past decade moved into the chemical industry on the tonnage production basis. Numerous bibliographical references[26,27,42,51] to the theory, development, and application of molecular distillation are available and have been reviewed for a better understanding of one of the newest of unit operations.

Theory of Molecular Distillation. Some of the theory underlying the basic principles of molecular distillation is presented in the following paragraphs.

Mean Free Path. Glasstone[34] defines the mean free path of a molecule as the average distance the molecule travels between two successive collisions.

A mathematical expression for the determination of the mean free path of a molecule was proposed on the basis of Glasstone's definition. Maxwell modified the expression to take into account the relationship between relative velocity and actual velocity when a number of molecules all in motion are present. The expression in its present form is:

$$l = V/\pi\sqrt{2}N\sigma^2$$

where:

l = mean free path of molecule, centimeters

N = Avagadro's number, 6.06×10^{23}

V = volume of one gram mole of the gas at the existing pressure, cubic centimeters

σ = average diameter of molecule, 2×10^{-8} centimeters

π = a constant.

It is of interest to note that the mean free path of a gas molecule is independent of its molecular weight since it does not appear in the expression. However, the accuracy of the expression is limited by the assumption of an average diameter for all gas molecules.

Distillability. For distillation under any conditions, the rate of distillation of a substance is roughly proportional to its concentration and to its vapor pressure. For molecular distillation, the rate of distillation[30] is proportional to its number of molecules and the probability that any molecule will distill. This probability or tendency to distill is called the "distillability". It is also defined[37] as the ratio of the number of molecules of a given species leaving the distilling surface in any small interval compared with the number of similar molecules remaining undistilled in the surface layer during the same interval.

Rate of Distillation[31]. For distillation under any conditions, the rate of distillation of a substance is roughly proportional to its concentration and to its vapor pressure. In molecular distillation, heat is supplied to the distilland under almost equilibrium conditions and the majority of the molecules leaving the evaporator are collected almost immediately by the condenser. Thus, the rate of heat input (at constant temperature) and the shape of the still are not significant variables as in the case of ebullient distillation. In molecular distillation, the rate of distillation is proportional to the product of the

number of molecules and the probability that the molecule will distill. This probability or tendency to distill, as has been stated before, is called the "distillability." According to Embree[32], more exact but less illuminating definitions are: "The distillability of a substance is proportional to its rate of distillation divided by its concentration" or "the rate of distillation of a substance is proportional to the product of its concentration and its distillability." Algebraically expressed,

$$V = kND$$

where:

V= rate of distillation, mols per second

k= proportionally constant

N= concentration, mol fraction

D= distillability.

If the distilland be considered an ideal solution, the partial pressure of the substance considered may be taken as N times p, i. e., the mol fraction times the vapor pressure of the pure substance. The rate of distillation, then, by Langmuir's equation is:

$$n = NPA\sqrt{\frac{1}{2\,MRT}}$$

where:

n = rate of distillation, mols per second

N = concentration, mol fraction

P = vapor pressure, dynes per square centimeter

A = area of distilling surface, square centimeters

M = molecular weight

R = ideal gas constant

T = temperature, degrees Kelvin.

Langmuir's equation also indicates that the rate of distillation is proportional to the surface area. In other words, molecular distillation is a surface phenomenon and subject to the limitations imposed thereby. As the molecular weight and viscosity of the distilling liquid is increased, replenishment of the

desired component in the surface layer becomes of major concern. Hickman[42] has indicated that it is imperative to keep the layer of distilling liquid, or distilland, as thin as possible for this reason and also to reduce the risk of thermal decomposition.

Thermal Hazard. One of the greatest virtues of high vacuum distillation is the ability to distill successfully substances of high molecular weight which are thermally unstable and cannot be distilled by the conventional distillation processes. The thermal exposure becomes increasingly important as the size of the molecule becomes larger. The quantity of distilland being heated and the time of heating contribute to the thermal exposure of the substance. According to the hazard index concept[31], the decomposition hazard, D, is the product of time, t, in seconds of exposure and P, pressure in microns, expressed as:

$$D = t \times P$$

It is noted by Hickman and Embree[29] that the thermal hazard encountered by the centrifugal molecular still is far less than with any other type of still.

Limitation of Process. In true molecular distillation, equilibrium does not exist between the vapor and liquid, i. e., molecules do not reenter the distilland. The quantity of a given material distilling at a given temperature is proportional to $P/\sqrt{M}$ as indicated by the Langmuir equation[48] and the relative quantities of two or more constituents are:

$$P_1/\sqrt{M_1},\quad P_2/\sqrt{M_2} \ldots\ldots P_n/\sqrt{M_n}.$$

This property of molecular distillation makes it impossible to separate, by a single distillation, substances having $P_1/\sqrt{M_1} = P_2/\sqrt{M_2}$. In many molecular distillation operations, where the vapor pressure and/or molecular weight of the constituents do not differ radically, the molecular still simply increases the concentration of one constituent in the distillate rather than giving a sharp separation. However, the molecular still is valuable because distillation is accompolished at very low temperatures, not because it gives good separations[43].

Azeotropic mixtures, which cannot be readily separated in the ordinary still, may be separated by molecular distillation provided their molecular weights are different.

Types of Molecular Stills. The development of the molecular still has progressed through three prominent stages[35]: (1) the pot still with a "boiling flask" similar to that employed in conventional distillation but operating under high vacuum and with a condenser relatively close to the "boiling flask", (2) the falling-film still where the distilland is metered to the vacuum chamber, degassed, and allowed to pass in a thin film down the walls of the evaporator, and (3) the centrifugal still where the distilland is metered to and passes over a rotor generally housed in a bell jar and supported on a shaft to the motor drive. Table I, page 22, shows the transition from the earliest pot still to the centrifugal stills giving the approximate dates when each type of still was developed and the film thickness and times of exposure encountered in the still.

Early Stills. The molecular still of Bronsted and von Hevesy[43] was relatively simple in construction, consisting of two concentric flasks. The outer vessel served as a boiler and the inner, cooled with liquid air, served as a condenser. The space between the two vessels was evacuated to 10^{-3} mm Hg and the mecury was slowly distilled at this pressure. Burch[21] later described an apparatus, adapted from that of

TABLE I

Transition from Pot Still to Centrifugal Still

Approx Date	Type of Still	Approx Distilland Thickness(a)	Molecular Thickness(b)	Approx Time of Exposure
1922	Laboratory pot still	1-5 cm	5×10^7	1-5 hr
1928	Laboratory tray still	0.1-1 cm	5×10^6	5-60 min
1930	Laboratory falling-film still	0.1-0.3 m	5×10^4	10-50 sec
1935	Industrial falling-film still	1-3 mm	5×10^5	2-10 min
1936	Laboratory centrifugal still	0.01-0.02 mm	3×10^3	0.04-0.08 sec
1940	Industrial centrifugal still	0.03-0.06 mm	1×10^4	0.1-1 sec
1942	High-speed centrifugal rim still	0.001-0.005 mm	4×10^2	0.001-0.005 sec

(a) Assuming similar throughput for same unit area of all stills.
(b) Assuming that the molecule of glyceride fat has an effective diameter of 15 Å.

Hickman, K. C. D.: "Science in Progress," p. 217. Yale University Press, New Haven, Conn., 1945. 4th Series

Bronsted and von Hevesy, in which petroleum products were subjected to molecular distillation. The apparatus consisted of a horizontal, electrically-heated tray of copper, having a surface area of 40 square centimeters and a capacity of 30 cubic centimeters of distilland. Surrounding the tray was a water-cooled condenser, having a surface area of 200 square centimeters, placed at a 'slight' angle to the horizontal. A spout at the lower end of the condenser was evacuated by a mecury condensation pump without the use of a cold trap. Temperatures were measured by a thermocouple soldered to the evaporating tray and pressures were estimated by means of a high tension discharge. It is of interest to note that Burch incorporated in the above apparatus two of the prerequisities of a good still; the condenser and evaporator should be coembrasive, without intervening obstruction, and the pump and connecting tube should be large[42].

Falling-Film Type. The next significant step in the evolution of the molecular still was the falling-film type which was described in minute detail by Hickman[36]. This type of still construction was an attempt to overcome some of the factors contributing to the rather poor performance of earlier stills.

One inherent difficulty encountered in molecular distillation is that ebullition accompanying ordinary boiling does not occur and the distilland remains unstirred during distillation. The distilling surface may thus become impoverished of its more volatile constituents and cease to be representative of the main body of the liquid. As a result, the distillate will contain less of the light and more of the heavy fractions than it should. This difficulty can be largely overcome by distributing the distilland in a very thin layer over the distilling surface.

In addition, the whole bulk of the distilland was maintained at or above the temperature of distillation in some stills, such as Burch's still described previously, although only the surface at any moment is envolved in distillation. Thus, a process whose main attribute is avoidance of thermal decomposition may actually result in prolonged exposure to heat. This drawback was eliminated by making the still in two parts, one part to store the distilland at low temperature and the other to carry out distillation at a higher temperature. The mass of distilland was kept small compared with that in the storage reservoir and the total heat exposure was reduced accordingly.

In the cyclic-column still of Hickman, the evaporator was a polished metal tube 15 centimeters in length and 3.7 centimeters in diameter. The column was heated internally by a helical electrical heater immersed in oil. Two collars of wire gauze, one having 60 meshes per inch and the other 120 meshes per inch, were fastened around the top of the column to aid in distributing the distilland evenly around the periphery of the column. The evaporator was surrounded by a glass, air-cooled condenser tube at the bottom of which was an annular trough for collecting distillate. Below and integral with the column were two storage reservoirs and a magnetic circulating pump made of nickel and glass and operated by an external electromagnet which received periodic impulses from a pendulum. By means of a series of ball valves incorporated in the still the flow of liquids could be directed as desired. In the concentration of vitamin A, found in halibut liver oil, this still showed an 80 per cent recovery as compared to an efficiency of 50 per cent achieved with the conventional pot still.

Centrifugal Type. While the falling-film type still offered some advantages over earlier types of stills it was by no means perfect. Its chief defect was the tendency of the distilland to gather into

streams or rivulets despite the various distributors that were employed[37,2,47] at the top of the columns. A rapidly rotating centrifugal still which utilized centrifugal force many times that of gravity to hold the film of distilland against the evaporator was built by Hickman[42]. This still had a slightly conical rotor, with a deeper cone at the center, mounted on a shaft which in turn was placed in bearings housed ina vertical back plate. The various connections necessary for still operation were brought through the plate and the whole assembly was covered with a bell jar which served as the condenser. Distilland was fed to the center of the cone, which was made of spun aluminum, and passed across the surface of the cone. Since the cone was rotating at 3000 to 6000 revolutions per minute, the distilland was discharged from its edge as a fine spray into a water-cooled gutter. The distilland was maintained at the distillation temperature for a fraction of a second in a layer which was considerably thinner than had been secured with a falling-film still, as an inspection of Table L will indicate.

Two stills of this type were first offered for sale commercially by Distillation Products, Incorpo-

rated, in 1947[46]. The smaller unit of the two stills has an evaporator which is 14 inches in diameter. The evaporator consists of a cone with a 15° slope which thus embraces an angle of 105°. This still has a batch capacity of 16 liters and a feed rate of 3 to 12 liters per hour.

The larger unit was designed to handle 50 to 250 gallons per hour of oils and heavy chemicals[45]. This still utilizes a rotating evaporator five feet in diameter and shaped like a flower pot. It is constructed of cast aluminum and turned on a precision lathe so that the sides of the evaporator slope upward at an angle of 10 to 25° to the vertical. The distilland is allowed to enter at the base of the cone through a nozzle pointing in the direction of rotation. The feed rate and nozzle diameter are such that the feed stream is projected onto the cone at the peripheral speed of the rotor. The distilland flows up the hot sides of the evaporator and passes over the top into a collecting gutter; the undistilled residue passes to a pump which removes it from the still. The degree of separation is generally between 0.8 and 0.95 theoretical molecular plate, which is often two or three times as good as that available from a non-fractionating pipe or pot still. The improvement in separation is

attributed to the thinness and turbulence of the evaporating film on the centrifugal evaporator. Enhanced separation is routinely obtained with this still by such alternatives as partial redistillation with a single still, partial redistillation with a feedback in multiple still assemblies, or redistillation through fractionating barriers, such as a wire mesh screen hung between the evaporator and condenser.

The latest centrifugal molecular still to be offered for sale commercially by Distillation Products, Incorporated, is a laboratory scale batch still with a heated five-inch rotor. It is claimed by the manufacturer that distillation takes place from a thin film exactly as it does under the condition present in larger commercial models.

Applications of Process. Molecular distillation has rendered distillable a whole category of substances, natural and synthetic, which could not be distilled otherwise primarily because of thermal decomposition at the high temperatures required. The range of usefulness of the molecular still is for organic chemicals of molecular weights between 250 and 1200 with a lower range if much oxygen, sulfur, or halogens are present in the molecules (44).

It would be impractical to attempt to give a complete list of all the specific applications of molecular distillation which have been reported. However, a list of materials to which the technique has been applied might be calssified under the following headings[27]:

Vegetable, animal and marine oils for edible and drying oil purposes

Solid fats

Petroleum oils and jellies

Long-chain hydrocarbons

Aromatic hydrocarbons and derivatives

Vegetable and fruit waxes

Carbohydrates

Vitamins

Sterols and sterol esters

Hormones

Saponins

Amino acids, polypeptides and derivatives

Condensation polymers

Phythaloyl esters

Dyes

Drugs

Miscellaneous substances such as digitonin, cholanic acid, querbrachol, fungoid growths, and others.

Further information on the application of molecular distillation can be found in the abstracts of Detwiler and Markley[27,28], and Todd[50], and the bibliographies of Hickman[42], Blasco[4], Burch[21], Fawcett[33], Burrows[23], and Waterman and van Vlodrop[52].

Molecular Still Operation

In molecular distillation, the rate of distillation of any component and the degree of separation of the components of a mixture depend on a number of interdependent variables in the design and operation of the still. These variables include temperature of distilland, film thickness, splashing, and distance between evaporator and condenser.

Temperature of Distilland. The measurement of the temperature of the actively distilling surface of the distilland presents a problem which has not been satisfactorily solved. There exists a temperature gradient of about five degrees through the distilland in a falling-film still[33]. The temperature gradient is probably less in the centrifugal still because of the decrease in film thickness. There is also a temperature gradient from the center of the rotor to the outer rim which varies with the feed temperature and the heat applied to the rotor. The customary practice has been to control the average temperature of the distilland be use of a source of heat at a constant rotor temperature and a constant temperature difference between the evaporator and the condenser. The average temperature of the distilland of a centrifugal still may be estimated by inserting a thermocouple in the collecting gutter.

Increasing the temperature of distillation increases the vapor pressure of the distilland and hence the rate of distillation. However, it should be recalled that the increase in distilling temperature causes a secondary effect of reducing the mean free path which would ordinarily be expected to reduce distillation. In general, this slight reduction in the rate of distillation will be much more than compensated by the increase resulting from the increased vapor pressure. According to Fawcett[33] the highest gross distillation will occur at the highest possible temperature short of decomposition , even though the distillation may shift from the molecular to the non-molecular type.

Film Thickness of Distilland. The exact effect of the film thickness on molecular distillation is unknown. Recent still design has attempted to reduce the film thickness in order to insure that diffusion will take place sufficiently rapid to make the surface or distilling layer representative of the fluid beneath.

A most important factor in molecular distillation is the surface of the distilling liquid, or distilland[44]. It must be remembered that only this surface is involved in the distillation. Momentarily, none of the other molecules in the still need be there. The only reason they are in the still is to replenish the surface layer, While only

the molecules that are at the surface are needed for distillation, all the molecules waiting their turn in the still are at the same temperature and thus, exposed to thermal decomposition. The reasoning behind a desire for a thin layer of distilland is evident.

A formula for the relationship[42] between the rotor speed and film thickness for centrifugal stills may be expressed as:

$$\log u = K \frac{1}{\log L}$$

where:

u = velocity, revolutions per minute

K = proportionality constant

L = film thickness, millimeters.

This formula was determined through a study of the spiral streams caused by feeding both colored and colorless oils slightly off rotor center and at the rotor center, respectively. Since the colored oil was applied eccentrically, it formed a small separate stream within the main colorless stream and the composite pattern of alternate colored and colorless spirals proceeded outward from the center of the rotor. The thickness at any position was determined to be directly proportional to the distance

between the spirals and inversely proportional to the area of the particular portion of the rotor considered. The persistance of the spiral pattern across the rotor is interpreted as indicating that the distilland is present in such a thin film that the evaporating surface at any moment represents the composition of the film beneath. The diffusion is so rapid that all of the molecules will come to the surface at some time during the travel across the rotor. According to Fawcett[33], a concentration gradient will be set up through the film if the rate of evaporation is greater than the rate of diffusion to the outer layer. A concentration gradient will be of importance in decreasing the efficiency of the process because (1) the gross rate of evaporation will decrease as the more volatile constituents are stripped out of the distilland and the vapor pressure decreased, and (2) the fractionating power will decrease since excessive quantities of the less volatile component will appear in the distillate and the more volatile will appear in the residue.

Splashing. In the preceeding section one reason for keeping the distilland film very thin was advanced; namely, to have a film so thin that diffusion takes place rapidly enough to make surface composition of the distilling film representative of the liquid beneath. The second reason is to prevent splashing, or bubbling of the distilland.

Splashing will affect the degree of separation obtainable in molecular distillation since substances of lower volatility will be carried into the distillate. The maintainance of a thin film on the rotor will not by itself insure the absence of splashing, but it does aid greatly in degassing the distilland. Improper degassing and too rapid evaporation have been mentioned[42] as the major causes of splashing. Too rapid evaporation would likely occur if the heat imput to the distilland and the subsequent temperature of distillation were higher than necessary for proper distillation.

Distance Between Evaporator and Condenser. The mean free path considerations have been discussed in a preceeding section. In general, variations of the gap between the evaporator and condenser do not produce any very noticeable variations in still performance provided that the gap does not become larger than the mean free path of the molecules. It is also possible that the gap may be reduced to such an extent that distillation rate will be decreased[33]. Distillation tests made using linseed oil with gaps varying from 1 to 6 centimeters showed no significant change. A gap of 0.5 centimeters resulted in decreased distillation. The closeness of the condenser to the evaporating surface tended to permit the return of some of the vaporized molecules to the distilling surface which would not have returned otherwise.

III. EXPERIMENTAL

Presented in this section are the purpose and plan of the investigation, a list of materials and apparatus used in the construction, and the methods of procedure used in the design and construction of the molecular still and accessories.

Purpose of the Investigation

The purpose of this investigation was to redesign and reconstruct a five-inch magnetically-driven centrifugal molecular still, with the following objects in view: (1) to eliminate leaks around the rotor shaft by constructing a magnetically-driven rotor, completely sealed within the still head and having no direct connections between the rotor shaft and the drive shaft of the motor; and (2) to gain flexibility in the operation of the still by use of an enlarged bell jar and the addition of a water-cooled condenser within the bell jar.

Plan of Investigation

The investigation was carried out according to the following plan:

Review of the Literature. All available literature pertaining to molecular distillation was reviewed for a thorough understanding of the subject, with particular attention being devoted to design features and recommendations by Bull[19].

Design of Magnetic Drive. The still head and magnetic drive were designed to function as an integrated unit, with the necessary design changes being made to the base plate to simplify operation of the entire still. Sketches were then made of the proposed assembly. Brass was used as the material of construction for the base plate, still head, and component parts of the magnetic drive.

Construction of Feed Lines and Accessory Equipment. In order to centralize and facilitate the operation of the still, a panel board was constructed from which all operational changes to the still could be made. The flow lines and necessary tanks were constructed of aluminum, while the vacuum lines were constructed of copper tubing. The tanks were tested for leaks before being installed into the unit proper. The flow lines and necessory

additional equipment were then installed and secured into place in the system. The electrical circuits necessary to complete the unit were then made and connected to the panel board.

Vacuum Tests. Vacuum tests were made to determine the effectiveness of the design changes which had been incorporated in the still. The tests served also to indicate any leaks that would hinder the future operation of the still.

Materials

The following materials were used in the course of this investigation:

Flux. Meets specification U. S. N. 51F4A, Army Air Corps No 11316A, and Army Ordance Department No AXS - 500. Used as flux for silver soldering of copper tubing-fitting connections.

Grease. High-vacuum, celvacene heavy, lot No 665. Manufactured by Consolidated Vacuum Corporation (formerly Distillation Products Industries), Rochester, N. Y. Used in testing for leaks around fittings and tanks in assembled set-up.

Oil. Amoil-S, stock No 8815. Obtained from Consolidated Vacuum Corporation (formerly Distillation Products Industries), Rochester, N. Y. Used as working fluid in diffusion pumps.

Oil. Cenco, hyvac, No 93050C. Obtained from Central Scientific Company, Chicago, Illinois. Used as lubricant and sealing fluid in Cenco hyvac and megavac forepumps.

Pilot Dye. 1,4-dimethyldiaminoanthraquinone, chemical formula, $C_{16}H_{14}O_2N_2$. Obtained through courtesy Tennessee Eastman Company, Kingsport, Tennessee. Used as pilot dye in test run.

Solder. Sil-fos, 15% silver content alloy, starts to melt at 1185 °F and is completely liquid at 1300 °F. Distributed by Southern Oxygen Company, Roanoke, Virginia. Used in welding numerous connections in the construction of the still.

Apparatus

The following equipment and apparatus were required to perform this investigation:

Cell. Dry, No 6, 1-1/2 v. Manufactured by the National Carbon Company, Cleveland, Ohio. Used in conjunction with potentiometer for thermocouple calibration.

Cell. Standard, Eppley, student, cadmium, unsaturated, catalog No 11-506-38. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used for zero balancing of potentiometer.

Feed Pump. Nitralloy Rotary Gear, size 4327-01-85557, serial No 3084, rotation either direction; maximum capacity 20 to 25 cubic inches per minute at 1750 rpm. Pump equipped with standard mounting brackets; specially fitted with oil cup No 1003 and with 1/4-inch IPT for No 1600 cast iron lantern ring. Obtained from Northern Ordance Pump Company, Minneapolis, Minnesota. Used to circulate feed mixture to and from still.

Feed and Product System. Drawing 2, page 55, shows the feed and product system used in this investigation. The component parts of the system are:

Heater Element. Thermalink, model No 42T2C2, 0.281-inch outside diameter, flexible copper sheath, 230 v, ac, 1750 w. Obtained from Electro-Therm Incorporated, Silver Spring, Maryland. Used as auxiliary heater to preheat the feed.

Fittings. Swagelok, brass and aluminum, assorted tees, adapters, valves for 1/4-inch copper tubing and 1/4 and 5/16-inch aluminum tubing. Obtained from Shelby Jones Company, Havertown, Pennsylvania. Used in connecting copper and aluminum flow lines in the system.

Tank, Loading. See item G, Drawing 2, page 55, copper, cylindrical, 1-1/2 inches outside diameter by 2-1/4 inches high, open at one end, concave bottom with 1/4-inch tubing welded to center for drain. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used as feed loading tank.

Tanks, Feed and Residue. Aluminum, cylindrical, 6-1/2 inches outside diameter by 7 inches high, closed at both ends, top flat, bottom concaved for drainage, all Swagelok connections with 1/8-inch male adapters.

See items J and H, Drawing 2, page 55, Constructed in the Industrial Engineering Shops Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Tank J used as primary feed tank, and tank H used in conjunction with calibration of feed pump.

Tank, Distillate. Aluminum, cylindrical, 4-1/2 inches lutside diameter by 5 inches high, closed both ends, necessary connections for venting, evacuation, entry and drainage of product. See item K, Drawing 2, page 55, Constructed in the Industrial Engineering Shops Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used as collecting tank for product.

Tape, Heating. Electrothermal, 130 v, ac, 3.8 amps maximum. Made in England. Obtained from E. Machlett and Son, New York, N. Y. Used for preliminary heating of feed and distillate tanks, and distillate-product draw-off line.

Tubing. Aluminum, 1/4 and 5/16-inch. Obtained from Shelby Jones Company, Havertown, Pennsylvania. Used for all flow, vacuum, and vent lines in feed and product system.

Galvanometer. Pointer type, DC, catalog No 11-506-27, model B with scale of 60 divisions of 1 millimeter each, sensitivity, 0.20 microampere per millimeter. Obtained

from Fisher Scientific Company, Silver Spring, Maryland. Used in conjunction with potentiometer for temperature readings of feed and residue thermocouples during calibration.

Gage. McLeod, vacuum, type MG-07, triple range, table model, pump operated, range 0 to 5000 microns. Manufactured by and obtained from Consolidated Vacuum Corporation (formerly Distillation Products Incorporated), Rochester, N. Y. Used to measure vacuum in the molecular still.

Hot Plate. Fisher autemp, catalog No 11-467-1, model A for 115 v, ac. Obtained from Fisher Scientific Company, Silver Spring, Maryland, Used to heat oil bath in thermocouple calibration.

Instrument Panel. The following items are mounted on the instrument panel for the still.

Autotransformers. Eight used, powerstat, type 116, 115 v, 60 cy, ac, 1 KVA maximum. Manufactured by Superior Electric Company, Bristol, Connecticut. One powerstat each used to control temperature of rotor heater, feed preheater, rotor speed, residue, product, and feed tank heaters, and two used to control temperatures of the two diffusion pumps.

Pilot Lights. One-hundred ten volts alternating current. Obtained from General Electric Supply,

Roanoke, Virginia. Used to control flow in various circuits

Switches. Single-pole, single-throw, toggle. Obtained from General Electric Supply, Roanoke, Virginia. Used in various circuits on the instrument panel.

Magnets. Fifty used, permanent type, No 4Y21P4, style rectangular U sintered shape, ground and magnetized. Obtained from Permag Company, Brooklyn, New York. Used as component part of magnetic drive.

Motor. Electric, companion type SPH, single phase, 115 v, 60 cy, ac, 1/4 hp, 1750 rpm. Obtained from Sears, Roebuck and Company, Roanoke, Virginia. Used to drive feed pump.

Motor. Type ADS, style 957656-B, serial KU, 1/4 hp, 50 ^{o}C temperature rise, 115 v, 60 cy, ac, 10,000rpm, variable speed. Manufactured by Westinghouse Electric Corporation, Pittsburgh, Pennsylvania. Used as drive for rotor.

Potentiometer. Fisher, type S, catalog No 11-506-1, two ranges, 0 to 0.017 v, and 0 to 1.70 v. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to measure voltage across thermocouples for calibration purposes.

Still Head Assembly. Drawing 1, page 53, shows an assembly drawing of the still head. The component parts of the assembly are described as follows:

Base Plate. Shown as item A, brass, 15 inches in diameter, 1-1/2 inches thich; drilled to accommodate feed inlet, vacuum, distillate, and residue outlets, rotor heater leads (spark plugs), vacuum gage and residue thermocouple outlets, and water connections for the condenser; circular groove 8-5/16 inches I. D. by 9-5/8 inches O. D. by 1/16-inch deep cut into the face of plate to accommodate silicone gasket for seal between bell jar and base plate. Original Plate obtained from Smith-Courtney Company, Richmond, Virginia. Modifications made in the Industrial Engineering Shops Department, Virginia Polytechnic Institute, Blacksburg, Virginia.

Bearing Housing. Shown as item B, brass, 3-1/4 inches long by 6 inches O. D., end section one inch long by 4 inches O. D., drilled to accommodate bearing 2-5/8 inches long by 4 inches O. D. Bearing drilled to accommodate 1/2-inch shaft set in two parts of 1/2-inch roller bearings. Shaft threaded on one end for rotor connection, and drilled on opposite end to make connection with machined flange 7/8-inch wide

by 2-7/8 inches in diameter. Twenty-five permanent Alnico magnets, 7/8-inch long by 1/4-inch wide by 1/2-inch high, bolted to outside of flange with 25 1/8-inch machine bolts. Machined from 6-inch diameter by 12-inch long brass rod. Rod obtained from Noland Company, Roanoke, Virginia. Modifications and machining done by the Industrial Engineering Shops Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used to contain bearings for magnetic drive and to serve as the inner section of the drive.

Bell Jar. Pyrex, high form, 14-1/2 inches long by 8-7/16 inches inside diameter. Obtained from Fisher Scientific Company, New York, N. Y. Used to enclose rotor and to form vacuum chamber for distillation.

Collecting Gutter. See item F, Drawing 2, page 55. Rolled bronze; gutter constructed to fit around the periphery of rotor with a clearance of 1/32-inch; outside diameter of gutter is 5-3/4 inches; inside diameter is 4-1/4 inches; thickness is 3/4-inch. Soldered to the back of the gutter is a 3/4-inch copper sleeve, 4-3/4 inches in diameter with a 1/4-inch lip to catch any dripping or overflow from the gutter. There is a 5/16-inch copper tubing outlet on the outer edge of the gutter and a corresponding outlet

on the sleeve extension. Both outlets enter a common header that serves as the residue draw-off line. Fabricated by the Comas Cigarette Machine Company, Salem, Virginia. Will be used to collect the residue of the feed mixture as it is thrown from the edge of the rotor in a fine spray during tests.

Gasket. Silastic, No 7-170, silicone rubber, 8-3/8 inches I. D. by 9-1/2 inches O. D. by 1/8-inch thick. Obtained from Dow-Corning Corporation, Midland, Michigan. Used for seal between bell jar and base plate of the still.

Heater. Chromalox ring element, catalog No A-20, type A, 115 v, 60 cy, ac, 300 w, chrome steel sheath, single heat, for temperatures up to 1200 °F maximum. Obtained from Edwin L. Wiegand Company, Pittsburgh, Pennsylvania. Used as heater for rotor.

Outside Housing. Shown as item F, Drawing 1. Brass, 3-5/8 inches long by 6 inches O. D. Machined to accommodate flange 5-3/16 inches O. D. by 7/8-inch deep, and 1/2-inch drive shaft set in two sets of 1/2-inch roller bearings press fitted into the housing. One end of shaft bolted to flange, other end fitted with flexible coupling for a connection to motor. Used to accommodate outer section of magnetic drive.

Rotor. Shown as item F, Drawing 2, aluminum; outside diameter 4.11 inches, thickness at outer edge 2.075 inches, angle of inclination of rotor surface 52-1/2 degrees, slant height of rotor surface 2.59 inches. Fabricated by Comas Cigarette Machine Company, Salem, Virginia. Used as the evaporating surface of the still.

Spark Plugs. AC, No 45; side contacts removed, 1/2-inch length of a No 10 bolt was welded to the center terminals to attach heater leads. Base plate drilled and tapped to accommodate plugs; vacuum-tight seal obtained by tightening plugs down on lead gaskets. Manufactured by General Motors Corporation, Detroit, Michigan. Used to connect heater to electric power source through base plate.

Thermocouples. Copper-constantan. Constructed from 3/32-inch copper tubing (serving as copper lead) and insulated Leeds and Northrup No 24 constantan wire. Wire inserted into tube and beaded at one end forming junction; junction secured and made vacuum tight by fusing the copper and constantan together at the junction. Thermocouples inserted into the feed and vacuum lines through the run of an aluminum-tubing tee and secured into place by compression fittings around a 1/4-inch collar soldered to the thermocouple tube. Constantan wire obtained from Leeds and

Northrup Company, Philadelphia, Pennsylvania. Used to secure temperatures of residue and feed during operation of the still.

Thermometer. Certified, catalog No 15-040, range -10 to 300 °C in 1/10 °C increments. Obtained from Eimer and Amend, New York, N. Y. Used in calibration of the thermocouples.

Vacuum Pumping System. The pumps used in the evacuation of various parts of the still are described as follows:

Hyvac. Cenco, 10 liters per minute capacity, 340 rpm, equipped with a 1/4-hp, 115 v, 60 cy, ac, 1750 rpm motor. Obtained from Central Scientific Company, Chicago, Illinois. One used in conjunction with the operation of the McLeod gage, and one used to evacuate distillate-product tank after each fraction was removed from still.

Megavac. Cenco, 31 liters per minute capacity, 325 rpm, equipped with a 1/4-hp, 115 v, 60 cy, ac, 1750 rpm motor. Obtained from Central Scientific Company, Chicago, Illinois. Two megavacs used in parallel as forepumps for diffusion pumps.

Diffusion. Metal, vertical, 500-milliliter oil capacity, 115 v, 60 cy, ac heater, water cooled. Other specifications unknown. Obtained from war surplus.

Manufactured by Consolidated Vacuum Corporation (formerly Distillation Products Industries), Rochester, New York. Two pumps used in parallel to obtain ultimate vacuum in system.

Methods of Procedure

The redesign and reconstruction of a five-inch magnetically-driven centrifugal molecular still was undertaken to overcome difficulties encountered in a previous design[5]. The still head assembly was completely redesigned to eliminate leaks around the drive shaft, and the still in general was made more flexible for experimental investigations.

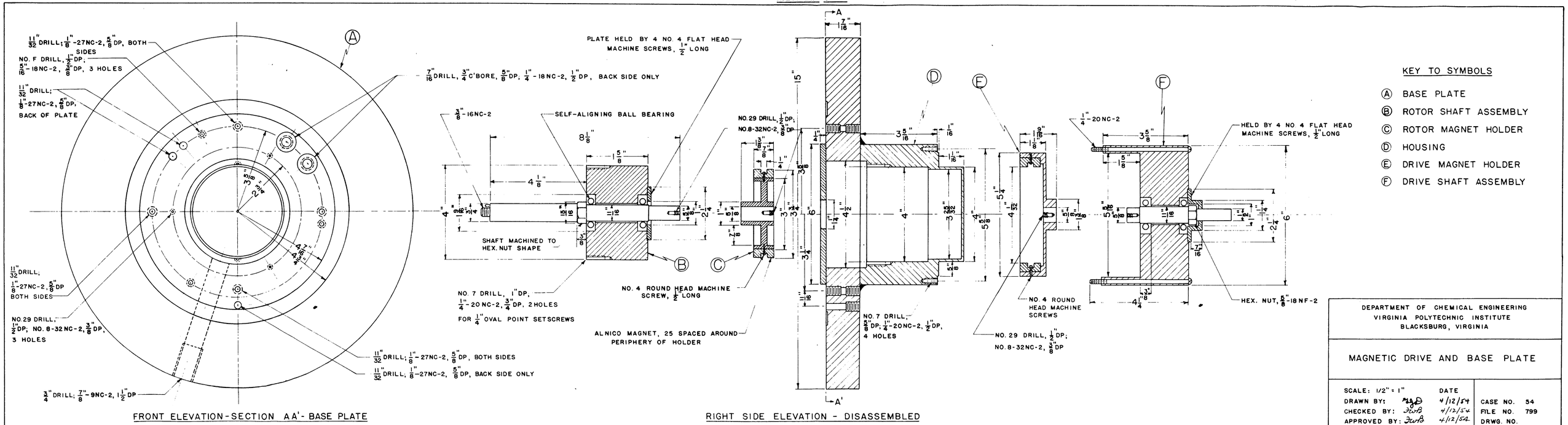
Frame. The supporting frame for the still was constructed of 1-1/4 by 1-1/4 by 1/8-inch angle iron, 36 inches high, 21 inches wide, and 16 inches long. The construction was such that the space between the supporting members was kept at a maximum to accommodate the necessary tanks, tank supports, flow lines, and pumps. The still head assembly was mounted on top of the frame and supported by two 2 by 2 by 3/16-inch pieces of angle iron, 15-1/2 inches long, welded to a 16 by 6-1/4 by 1/2-inch iron plate, which in turn was welded to the frame. The mount for the motor was constructed from an iron plate, 7 by 5-1/4 by 3/8 inches, bolted on one end to an 1/8-inch supporting iron brace, and welded on the other end to a 3/8 by 16 by 1-inch piece of strip iron. The strip iron and support were in turn bolted to the top of the frame.

The entire frame was bolted to the floor for additional support with 1/4-inch diameter, 1-1/2-inch long wall anchors.

Still Head Assembly. The still head assembly, shown in Drawing 1, page 53 , was redesigned and reconstructed to include the magnetic drive, a brass base plate, a condenser, and a "high-form" bell jar. The material of construction for the assembly, with exception of the bell jar, was brass, so as to prevent interference with the magnetic field set up by the magnets. The component parts of the assembly are explained as follows:

Magnetic Drive. Due to the difficulties encountered by Bull[15] in trying to prevent leaks through the packing around the rotor shaft, it was decided to eliminate any direct connection between the rotor shaft and the drive shaft of the motor. This was accompolished by machining a 6-inch diameter, 3-1/2-inch long brass housing, item D, to accommodate a 3-inch brass disk fitted with 25 permanent Alnico magnets; the disk was attached to one end of the 7/8-inch diameter inner rotor shaft, shown in item B. The outer magnets were bolted to a 5-1/4-inch diameter machined countersunk disk, item F, and so fitted that when assembled, the outer magnets would engage the inner magnets, setting up the necessary magnetic field

 Drawing 1. Details of Magnetic Drive and Base Plate for Centrifugal Molecular Still



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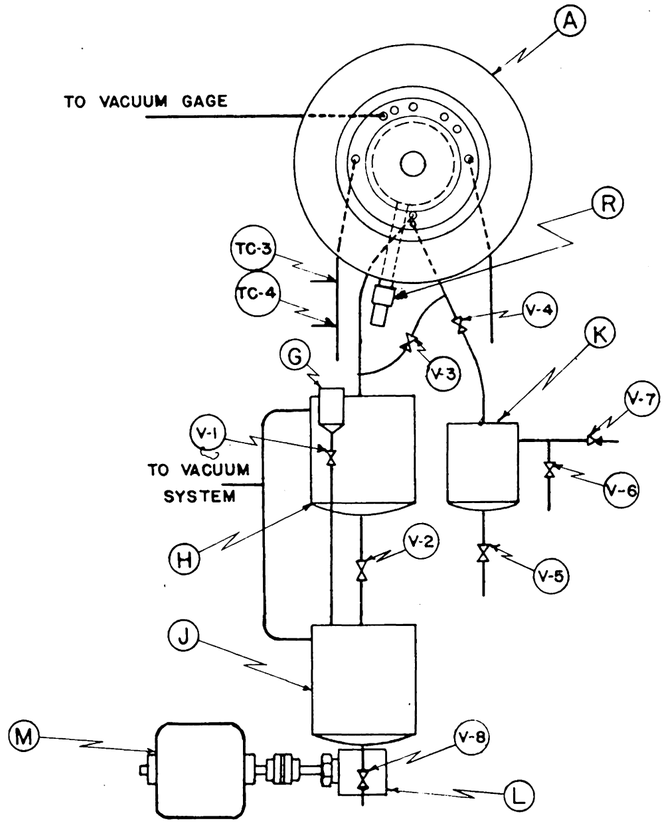
MAGNETIC DRIVE AND BASE PLATE

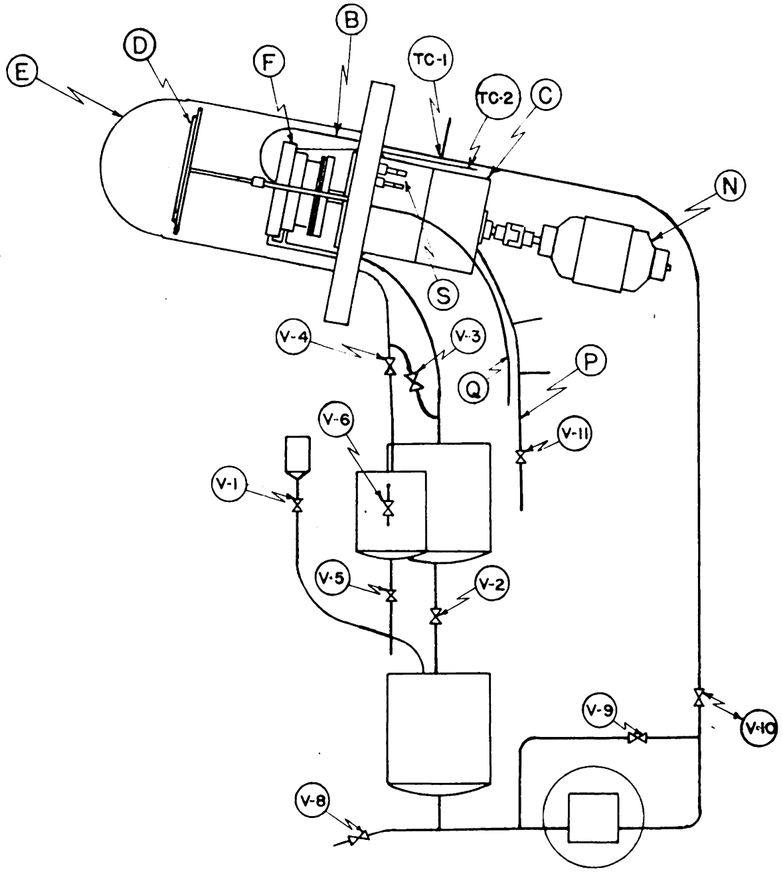
SCALE: 1/2" = 1"	DATE		
DRAWN BY: [illegible]	4/12/54	CASE NO.	54
CHECKED BY: JWB	4/12/54	FILE NO.	799
APPROVED BY: JWB	4/12/54	DRWG. NO.	

required for the drive. The parts of the assembly were so machined that a minimum of air gap and metal thickness separated the magnets; the housing was 1/8 of an inch thick, with approximately 1/32 of an inch air gap on either side. The magnets were arranged in alternating matching groups -- six, six, six, and seven; alternating N-S, S-N, N-S, and S-N, respectively.

For alignment purposes, the rotor shaft was machined to fit through two 1/2-inch roller bearings, which were press-fitted and locked into a 4-inch diameter, 2-5/8-inch long machined sleeve. The rotor shaft projected through the base plate, shown in item D, far enough to accommodate the face plate, rotor heater, and the rotor; all shown in Drawing 2, page 55. The outer flange was aligned so that it would maintain proper clearance between the housing and the magnets by connecting it to one end of the 7/8-inch diameter outer drive shaft, shown in item F. The drive shaft was held in place by two sets of 1/2-inch roller bearings and connected by means of a flexible coupling to the motor, item N, Drawing 1. In this manner, the inner magnets, flange, and rotor shaft were kept as a completely sealed unit, with no direct connection between the rotor shaft or the drive shaft of the motor.



FRONT ELEVATION



RIGHT SIDE ELEVATION

NOTE: ANGLE IRON SUPPORT FOR SYSTEM NOT SHOWN.

KEY TO SYMBOLS

A	- BASE PLATE
B	- FEED NOZZLE
C	- MAGNETIC DRIVE
D	- CONDENSER
E	- BELL JAR
F	- GUTTER AND ROTOR HEATER ASSEMBLY
G	- LOADING TANK
H	- RESIDUE TANK
J	- FEED TANK
K	- DISTILLATE TANK
L	- FEED PUMP
M	- FEED PUMP MOTOR
N	- ROTOR MOTOR
P	- INLET WATER LINE
Q	- OUTLET WATER LINE
R	- MAIN VACUUM LINE
S	- SPARK PLUGS
TC-1	- FEED THERMOCOUPLE
TC-2	- RESIDUE THERMOCOUPLE
TC-3	- FEED TC COLD JUNCTION
TC-4	- RESIDUE TC COLD JUNCTION
V-1 TO V-11	- VALVES

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CENTRIFUGAL MOLECULAR STILL

WITH MAGNETIC DRIVE

SCALE: 1/8" = 1"	DATE	CASE NO. 54
DRAWN BY: [illegible]	4/7/54	FILE NO. 799
CHECKED BY: [illegible]	4/12/54	DRWG NO. 2.
APPROVED BY: [illegible]	4/12/54	

Base Plate. To accommodate the changes necessitated by the magnetic drive, a new brass base plate, shown in item D, had to be constructed; it was based on the design used by Bull[14] in his studies. The plate was machined from a brass disk 15 inches in diameter and 1-1/2 inches long, to accommodate taps for spark plugs for heater connections, vacuum outlet, feed inlet, distillate and residue outlets, vacuum gage connections, residue thermocouple inlet, and water connections for the condenser. A circular groove 8-5/16 inches I. D. by 9-5/8 inches O. D. by 1/16-inch deep was cut into the face to accommodate the silicone rubber gasket, which acted as a seal between the bell jar and the base plate.

Water-Cooled Internal Condenser. To gain flexibility in the operation of the still, it was decided to employ a "high-form" bell jar, item E, Drawing 2, 14-1/2 inches high by 9-3/8 inches O. D., instead of the low-form used by Bull[19]. Since the bell jar was too long to serve as an effective condenser, a copper condenser, item D, Drawing 2, was built. It consisted of a 1/8-inch copper plate, 8 inches in diameter, to the back of which six concentric spirals of 3/16-inch copper tubing were soft soldered. Cooling water circulated through these

coils to provide the necessary cooling medium. The connections for the condenser are shown in Drawing 1.

Features of the Still Head Assembly Not Affected by the Design. In redesigning and reconstructing the still, some features of the still head assembly used by Bull were retained and incorporated into the present still. They were the five-inch rotor[10], the flanged collecting gutter[9], and the rotor heater[9]. These items are explained in detail in the above references.

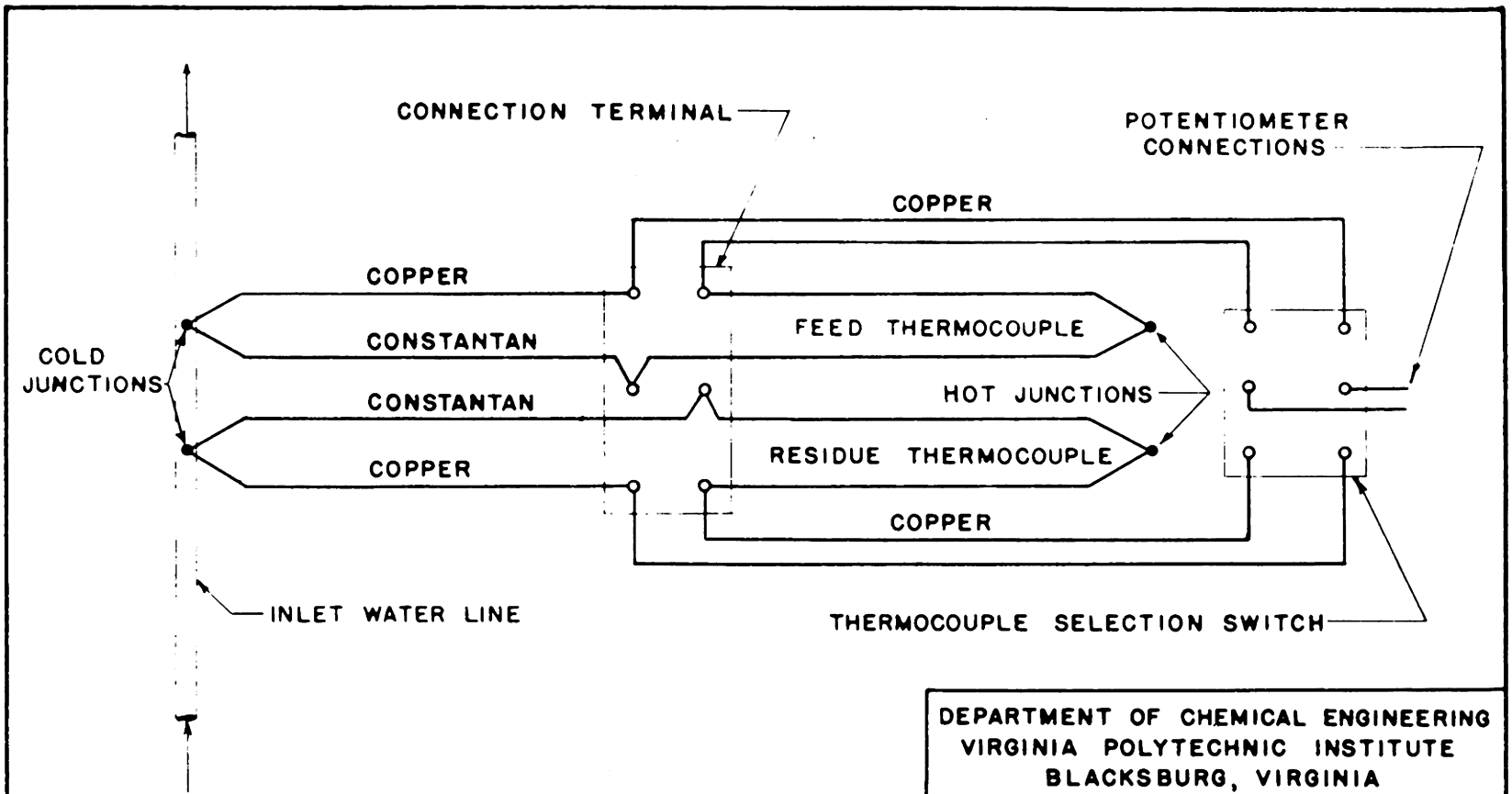
Still Accessories. In the all-metal still built by Bull, the flow lines, tanks, and accessory equipment were constructed of copper or copper tubing. To gain increased capacity and flexibility in the operation of the still, the following changes were made.

Distilland and Residue Tanks. The distilland and residue tanks were made larger[6] than those constructed by Bull in order to handle greater quantities of liquid. The tanks, items H and J, shown in Drawing 1, constructed of aluminum, were 6-1/2 inches O. D. by 7 inches high, closed at both ends with a flat top and a concave bottom for drainage. They were installed in the assembly with assorted aluminum Swagelok tees, adapters, and vacuum tight Hoke valves. All lines, with the exception of the

vacuum lines, were constructed of 1/4-inch aluminum tubing. The vacuum lines were constructed of 5/16-inch aluminum tubing.

Distillate Tank. The distillate tank, item K, was also constructed of aluminum. The tank was 4-1/2 inches O. D. by 5 inches high, and closed at both ends; the top was flat and the bottom concave, to allow for drainage. Necessary connections were provided to accommodate lines for venting, evacuation, entry and drainage of the product.

Thermocouples. The feed and residue thermocouples were constructed according to the procedure described by Bull[16]. The feed and residue thermocouples were inserted into their respective locations and secured by compression type fittings. To eliminate the use of an ice bath for the cold junction, the following change was made. Two more thermocouples were constructed, similar to the ones already described, and were installed into the water line through the run of tees; in this way the cooling water replaced the ice as the cold junction. The leads from the cold junction were brought to a terminal board, and to the feed and residue thermocouples and also to the potentiometer. A schematic wiring diagram for the set-up is shown in Drawing 3, page 59.



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VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

THERMOCOUPLE CONNECTIONS

SCALE: NONE DATE
DRAWN BY: J.F.M. 4/8/54
CHECKED BY: [illegible] 4/8/54
APPROVED BY: [illegible] 4/12/54

CASE NO. 54
FILE NO. 799
DRWG NO. -

Feed Pump and Heaters. On the recommendation of Bull[17], the magnetic feed pump was replaced by a type 4327-01-85557 positive displacement Nitralloy rotary gear pump; maximum capacity 20 to 25 cubic inches per minute at 1750 rpm. The pump was specially machined to accommodate an oil cup and a No 1600 cast iron lantern ring. When operating under vacuum the oil cup maintained a continuous film of oil between the shaft and the lantern ring, and by virtue of this oil seal the pump was made vacuum tight.

The heaters used in the system were the same ones employed by Bull[7] in his design; they consisted of a ring-type heater element for supplying heat to the rotor, a flexible sheath heater for preheating the feed as it passed from pump to rotor, and heating tape heaters for preheating the feed tanks, and for maintaining the proper temperature for the distillate-product tank.

Vacuum Producing System. The vacuum pumping system consisted of forepumps (megavacs), diffusion pumps, and a product tank evacuating pump.

Forepumps. Two megavacs, connected in parallel to two diffusion pumps, served as forepumps for the vacuum system. The pumps were connected to the

diffusion pumps through 1/2-inch copper tubing; all connections were welded.

Diffusion Pumps. Two vertical metal diffusion pumps were used in parallel in the system; the low pressure side of each of the pumps was connected to the megavacs through copper tubing. The high vacuum side of each pump was soldered into the run-ends of a 1-1/2-inch copper tee, the other end of which was soldered to a water-cooled cold trap. The cold trap in turn was connected to the vacuum outlet of the base plate by means of a 3/4-inch copper line. All joints were silver-soldered to minimize the possibility of leaks. With this arrangement, the system could be evacuated to 5 microns, 5×10^{-3} mm Hg, absolute pressure.

Product Tank Evacuating Pump. A product tank evacuating pump, similar to the one described by Bull[12], was connected directly to the tank to be used in evacuating it after the collection of the various distillate samples had been made.

Vacuum Measuring System. The vacuum in the system was measured by a standard triple scale, pump operated, McLeod gage; range 0 to 5,000 microns. The gage was connected to the base plate by means of 1/4-inch copper tubing, as

shown in Drawing 2, page . A hyvac vacuum pump was used to operate the gage.

Panel Board. To facilitate and better control the operation of the molecular still, a 30-1/2 by 46 by 3/8-inch pine panel board was constructed, and on it were installed the following items: eight autotransformers for the control of the rotor heater, the feed preheater, the rotor speed, the residue, product, feed tank, and the two diffusion pump heaters; two single-pole single-throw on-off switches for the two hyvacs; and a double-pole double-throw switch for the feed and residue thermocouples. An electric clock with a sweep second hand was also installed on the panel board. The board was mounted on 1/2 by 1/2 by 1/8-inch angle iron, and anchored to the wall by 1/8-inch wall anchors.

Preliminary Operation. The preliminary operation of the still consisted of thermocouple calibration, vacuum testing, and an actual test run to determine the effectiveness of the modifications incorporated in the still design.

Thermocouple Calibration. Two copper leads were connected from the cold junctions of the two copper-constantan thermocouples to a switch, which in turn was connected to a Fisher type S potentiometer. The thermocouples were then calibrated by placing the bead of each in an oil bath together with a standard

thermometer and an electric stirrer. The oil bath was agitated, and heat was supplied by means of an electric hot plate. The temperature of the bath was raised at approximately 20 °C intervals to a maximum temperature of 210 °C, and allowed to reach a constant value at each interval before any potentiometer readings were recorded. Readings were also taken while allowing the bath to cool, again allowing the temperature to reach a constant value at each interval. A plot was then made of the potentiometer readings versus temperature.

Vacuum Testing. To test the many connections, fittings, accessories, and other featured characteristics of the still, ten vacuum tests were conducted, with the final result that a vacuum of 5 microns Hg, absolute pressure, could be readily attained within 35 to 45 minutes of operation. In the construction of the still, provisions were made to test parts of the system separately or in combination with one another in such a manner as to readily isolate any leak that may develop.

The first portion of the system to be tested for vacuum tightness was the pumping system, consisting of the two megavacs and the two diffusion pumps. Thereafter as each additional component part of the

system was added, attempts were made to evacuate that particular part. If a vacuum of 30 microns absolute pressure or less could not be attained within 45 minutes with the forepumps alone, it was fairly certain that a leak would be found somewhere in the unit under test. In the case of welded connections, each one was given a thin coating of high vacuum grease, and if the leak was not isolated, then each fitting was tightened, one by one, until the leak was finally discovered. It was found that most of the leaks occurred in the fittings, and they were eliminated usually by merely tightening the compression fittings. No glyptal had to be used on the system at all, except to seal off the drain connections for the oil on the diffusion pumps.

Test Run. To test the general workability of the design, a test run was conducted on the system, using 0.0143 grams of 1-4 dimethyldiaminoanthraquinone dye dissolved in 650 milliliters of an equal mixture of light and heavy mineral oils. The start-up and operational procedure followed was the same one as outlined and recommended by Bull[13]. Data were taken for approximate qualitative evaluations, but no attempt was made to secure any quantitative relationships.

IV. DISCUSSION

The constructional features and design changes made in the molecular still, as recommended in part by Bull[19], are discussed in the following section; a listing of recommendations for future improvements is also given.

Design Changes in the Still

The design changes incorporated in the construction of the still are discussed in the following section.

Still Head Assembly. Complete redesign and reconstruction of the still head assembly included the magnetic drive, base plate, and water-cooled condenser; a "high form" bell jar was also made use of.

Magnetic Drive. Since Bull experienced considerable difficulty in maintaining a vacuum because of leaks around the rotor shaft, and later encountered difficulties with the "shorting out" of the drive motor when enclosed within the vacuum chamber, it was planned to design and build a more satisfactory and permanent still head arrangement. The first step in the construction was that of perfecting a magnetic drive, constructed so that

there would be no direct connections between the drive shaft of the motor and the rotor shaft; thus eliminating leaks around the rotor shaft, and leaving the drive motor outside the vacuum chamber where ample cooling was available. Brass was selected as the material of construction for all component parts of the still head assembly, both because of its non-magnetic properties and its high machinability.

The drive itself consisted of two concentric brass flanges, with 25 permanent Alnico magnets arranged around the periphery of each one, so that the magnetic forces were directed between the rows of magnets. Tolerances were such that the magnets were separated by a total distance of 3/16-inch air gap and metal thickness. The magnets were equally spaced around the flanges in three groups of six and one group of seven; that is, six were arranged with all the north poles in one direction, the next six with all the north poles in the opposite direction, and so on. The magnets on the outer flange were arranged exactly opposite to this, so that proper functioning would be attained. The outer flange was connected by a shaft to a 10,000 rpm type ADS variable speed motor. The inner flange was connected through a shaft to the rotor, contained

inside the bell jar. Both the rotor shaft and the drive shaft rotated on two sets of roller bearings.

In actual tests, it was found that the drive functioned very satisfactorily, provided certain precautions were followed when first starting up the speed of the rotor. Because of the small size and the fact that they were somewhat separated, the magnets exerted a very low starting torque, as compared to the inertia of the rotor and the rotor shaft. If the motor speed were increased too fast, the magnets would not stay "matched", and the rotor shaft would stop, even though the motor shaft were still turning. Consequently, it was necessary to increase the speed of the motor from rest very slowly until the powerstat reading was 40. After the rotor had reached the speed corresponding to this reading - approximately 1750 rpm - changes in the rotor speed could be made without fear of the magnets slipping past each other.

Base Plate. No radical changes were made in the base plate design; it was essentially the same type as the one employed by Bull[11] in his studies. Additional inlet and outlet connections were included in the base plate to accommodate the water condenser, but aside from these changes, the same flow-line

inlets and outlets were employed. They were drilled and tapped to accommodate 11/32-inch aluminum tubing adaptors. The plate itself was machined from a 1-1/2-inch thick brass plate, 15 inches in diameter. It was welded to the rotor housing to ensure a vacuum-tight seal.

The vacuum system was connected to the base plate by means of 3/4-inch copper tubing; all joints were silver-soldered, as this method proved to be the most effective in preventing leaks. An absolute pressure of 8 microns was obtained during the first vacuum test of the still head assembly, which indicated that the welds, connections, and fittings were all very satisfactory. The fact that no subsequent leaks developed in the still head during the remainder of the tests proved the above to be true.

Water Condenser and Bell Jar. In an effort to gain flexibility in the operation of the still, a "high-form" bell jar was secured for use with the still; it was 14-1/2 inches high as opposed to the low-form used by Bull which was only 4-1/2 inches high.

In keeping with the recommendation by Bull[20], a water-cooled copper condenser was constructed and installed within the bell jar; it will be used to

obtain additional information and a possible correlation between the elimination maxima and the distance between the cooling surface and the rotor surface. The condenser consisted of a smooth round copper plate, 1/8-inch thick and 8 inches in diameter. On the back of the condenser six turns of 3/16-inch copper tubing were soft soldered; the ends of the tubing were passed through holes at the edge of the copper plate and secured by means of tubing adapters to the water pipe and drain pipe connections. The pipe and tubing connections also served as the support for the condenser. As can be seen from this arrangement, the still is very adaptable for studies of condenser-rotor distances, as the condenser may be varied over a range of 0 to 9 inches from the rotor by merely reducing or lengthening the pipe connections as desired.

Still Accessories. A discussion of the changes made in the still accessories, including feed, residue, and product tanks, thermocouples, and feed system is presented in the following section.

Feed, Residue, and Product Tanks. To increase the capacity of the still, feed and residue tanks with 10,000 cubic centimeters capacity and a

product tank with 4,000 cubic centimeters capacity were installed in the system. The feed and residue tanks were constructed from a 6-inch diameter aluminum pipe, while the product tank was constructed from a 4-inch pipe. The tanks were interconnected with 1/4-inch aluminum tubing, and fitted with the necessary valves, fittings, and connections for the proper and most flexible operation.

The top and bottom of each tank was welded to the precut section of pipe, and after construction all tanks were leak tested under water at 80 pounds per square inch, gage, air pressure. As no leaks were detected, the tanks were assumed to be sufficiently tight for use in the vacuum system.

Thermocouple Calibration. Because of the satisfactory performance of the copper-constantan thermocouples used by Bull, it was decided to use the same type again in the system to measure the temperature of the feed and residue.

In an attempt to simplify operation, it was decided to use the cooling water as the cold junction for the thermocouples, since the temperature of the tap water used for cooling remains nearly constant, $50 \pm 1\ ^{o}F$, at all times. Accordingly, another

thermocouple was constructed similar to the feed and residue thermocouples, and installed in the cooling water line to the condenser. Considerable difficulty was experienced in trying to get the set-up to operate at first; later this was found to be due to the fact that the terminal board employed in the system did not provide the proper insulation between the two thermocouples. The only solution to this problem, other than a double-pole double-throw switch, was to install a second cold junction in the water line; this was done, but the thermo-couples still would not function properly. It was finally decided to insulate the cold junctions from each other and the rest of the system entirely by placing sections of rubber hose on either side of the tees that served as the connections for the thermocouples into the water lines. This last method proved to be the solution, for after the feed and residue thermocouples were thus insulated, they were found to function properly.

Feed System. Among the changes made in the feed system were (1) the construction of all flow lines and tanks of aluminum and (2) the use of a nitralloy rotary gear feed pump.

To allow the use of the still for systems other than oil soluble dyes, it was decided to construct all flow lines, tanks, and other pieces of equipment that were to come into contact with the hot feed, of aluminum. This metal was chosen because of its low catalytic decomposition effect on heat sensitive substances, such as fatty acids. Copper, on the other hand, has a very marked effect on these materials, as corrosion tests using stearic acid and samples of copper and aluminum showed.

Also, in keeping with Bull's recommendation[18], a new feed pump was sought which would give a steady continuous flow of feed to the rotor, thereby eliminating the pulsation and splashing of the feed caused by the magnetic pump. It was learned[49] that the Northern Ordance Pump Company produced a rotary gear pump which operated successfully under very high vacuum. Such a pump, specially fitted with an oil cup and lantern ring, was ordered, tested, and found to hold a vacuum of 6 microns Hg, absolute pressure. An interesting feature of the pump was that it held a vacuum of 6 microns pressure while it was operating, but when shut off the pressure in the still increased to 13 microns. This increase was

due to the fact that while running, the oil cup maintained a thin film of oil between the lantern ring and the shaft, which acted as an additional seal against leaks around the shaft.

Diffusion Pump Assembly. The diffusion pump assembly was modified slightly in the reconstruction from the set-up used by Bull. Instead of operating them in series, the pumps were placed in parallel, together with the forepumps, and connected to a cold trap, which in turn was connected to the vacuum outlet in the base plate. Tests on this modified set-up indicated that optimum operating capacity of the two pumps could be obtained if the pump boilers were filled with 500 milliliters of oil, and if they were operated on 90 volts. With such an arrangement, 5 microns absolute pressure could be obtained in the system.

Panel Board and Wiring. The construction and installation of the panel board greatly centralized the operation of the still. All wiring from the still, including thermocouples, was brought to the back of the panel board through conduits, and connected to the proper switches and powerstats. While making a test, it was possible for the operator to tell at a glance all the conditions prevailing at any one time. It also increased

safety measures, for if anything happened to go wrong with the functional components of the still, all controls could readily be reached before any extensive damage could be done.

Preliminary Operation of the Still. Although it was the prime purpose of this investigation to redesign and reconstruct a five-inch centrifugal still, vacuum tests and a test run were conducted on the system to ascertain whether or not the changes made in the still design were satisfactory from an operational standpoint. These tests are discussed in the following section.

Vacuum Tests. As explained previously in the Methods of Procedure, pages 63 through 64 , the component parts of the still were separately vacuum-tested as they were installed in the system. Most of the leaks were found to be around the adapter end of the Swagelok fittings;these were remedied by merely tightening the fittings into the base plate, or tanks, whichever the case happened to be. The silver-soldered connections were carefully made in the vacuum line, and were found to contain no detectable leaks. In all, due primarily to the careful planning, use of vacuum fittings and valves wherever possible, and careful assembly, no major leaks were found in the system, and none developed over the period of the vacuum tests.

Actual Operation of the Still. When the still head assembly had been completed, the thermocouples calibrated, and the vacuum test completed, it was decided to perform an actual test run on the still. Accordingly, a feed mixture containing 0.0143 gram of 1-4, dimethyldiaminoanthraquinone dye[27] dissolved in 650 milliliters of equal parts of light and heavy mineral oils was used.

The system was evacuated to 11 microns absolute pressure, the feed added, rotor speed set constant at 2,000 rpm, and the feed pump started. Data were taken from time to time to serve as a check on the operational characteristics of the still, but no effort to deduce any quantitative results was made.

In general, the operation of the still was very successful, the only resulting trouble was encountered in the feed line and the manner in which the feed was being preheated. The operation of the feed pump was found to be successful between feed rates of 100 to 150 milliliters per minute; rates below this range resulted in a pulsing flow rate, and rates above this range were too large for the gutter to handle properly, and caused the residue to spill over into the distillate fraction.

As was mentioned above, the major fault with the present system was found to be in the manner in which the feed was preheated before being sent through the feed nozzle and discharged across the rotor. Due to the many contractions and expansions caused by the fittings, valves, and bypass, the feed had a tendency to superheat in the line fromthe feed pump to the rotor. This superheating of the feed, resulting in the formation of vapor in the feed line, caused considerable splashing of feed off the rotor, and into the distillate fraction. This splashing became very pronounced at temperatures above 130 °C. It was thought that this condition can be remedied by first pumping the feed cold (75 °F) to a constant head tank, heating the feed in the tank to the distillation temperature, and then allowing it to feed onto the rotor by gravity.

In testing the system, it was also found that the original aluminum feed line was unable to withstand the heating of the nicrome wire heater, and melted when approximately half the line voltage had been applied to the heater. The melting point of aluminum is 680 °C, and although the tubing was insulated from direct contact with the wire heater, the heater itself must have exceeded this temperature, even

though the temperature of the feed flowing through the tubing was only 130 °C. The aluminum tubing was replaced by a 1/4-inch copper line, which withstood the heater temperature successfully.

It was also noted during the test that the water condenser was very effective in condensing the distillate. At the higher temperatures, the distillate was observed to run off of the condenser in an almost steady flow of liquid.

Recommendations

In the recommendations for future studies, considerations of improvement to the molecular still assembly are necessary to advance the study of molecular distillation. A few of these improvements are listed in the following section.

Use of the Feed Pump and Head Tank for Feed System. In the preliminary test made on the molecular still, it was observed that the range of feed rates was very small. The minimum rate for continuous steady feed was 100 milliliters per minute; rates below this value were unsteady and had a tendency to pulse rather strongly. Because of the limited size of the residue gutter, the maximum feed rate was 160 milliliters per minute.

In order to obtain a wider range of operation and a more satisfactory method of controlling the feed rate below 100 milliliters per minute, it is recommended that the present feed tank be elevated at least two feet above the still head assembly, and the feed be introduced onto the rotor by means of gravity flow. The rates could then be controlled over a much wider range by one valve, and steady feed flow could be maintained at all times. The feed pump could then be used to pump the residue to

the overhead feed tank, instead of pumping the feed directly onto the rotor.

Method of Preheating the Feed. It was found in the present set-up;that is, preheating the feed in the line from the feed pump to the rotor, that considerable splashing resulted probably from the feed becoming superheated and forming vapor pockets in the feed line.

To remedy this situation, it is recommended that, in addition to elevating the feed tank, the feed be brought to within five degrees of the required distillation temperature in the overhead tank and then allowed to distill. In addition to better control, this method of heating would probably prevent or greatly reduce the tendency for the feed to superheat and vaporize in the line, and therefore eliminate the present undesirable splashing of the feed off the rotor.

In connection with heating the feed in the feed tank, it is also recommended that two water-cooled aluminum condensers, approximately 5 inches high and 2 inches in diameter, be constructed and installed with the necessary fittings and valves in the vacuum line leading from the feed tank to the main vacuum line. These condensers would function in the following manner. While the feed in the tank was being brought up to the desired distillation

temperature, the first condenser could be used to collect and condense any feed vapors that may have evaporated from the liquid surface in the feed tank and passed out the vacuum line. The contents of this condenser could be recycled back into the feed tank. Once the desired distillation temperature was reached, and a distillate fraction was started, the second condenser could be cut in. At the end of the distillation time, the contents of the second condenser could be drained and added to the volumn of the fraction collected in the distillate tank. As long as this method is followed every time a fraction is collected, any error that may be introduced will be "blanked", and the results will be comparable. It is believed that this procedure will reduce to a great extent the superheating and splashing of the feed off the rotor.

Residue Gutter. As was mentioned previously, the present gutter will not adequately handle feed rates above 160 milliliters per minute.

It is recommended that studies be made to determine how best to increase the capacity and the design of the present gutter so that it will add a greater range of operation and capacity to the molecular still.

Replacement of the Rotor Heater. It was noted in this investigation that full line voltage, 115 volts, was required by the 300 watt chromalox ring heater element

used to heat the rotor in order to obtain 150-degree Centigrade residue temperatures. It is recommended that the 300-watt heater be replaced by a 1000-watt, Type A-66 heater which will give a greater heater capacity to the still. Such a heater may be purchased from Edwin L. Wiegand Company, Pittsburgh, Pennsylvania.

V. CONCLUSIONS

From several tests conducted on the constructional features of the redesigned and recons tructed five-inch magnetically-driven centrifugal molecular still, the following conclusions were drawn:

1. The magnetic drive functioned very satisfactorily over a range of speed varying from 500 to 8,000 rpm, and was a major improvement in the operational characteristics of the molecular still. As the rotor was completely sealed within the vacuum chamber, there were no direct connections between the rotor shaft and the drive shaft of the motor, and therefore no possibility for leaks to develop around the shaft as all packing, bearings, etc., were eliminated.

2. The rotary feed pump performed the functions necessary for successful operation in a vacuum system very well. Vacuum tests showed that it held a vacuum of 10 microns Hg, absolute pressure, while not operating, and after operating continuously for 13 hours pumping feed at the rate of 125 milliliters per minute, it successfully held a vacuum of 6 microns of mecury, absolute pressure.

3. The use of a high-form bell jar did not in any way cause serious problems to develop in the operation of the molecular still. The increased capacity apparently had no effect on the efficiency of the vacuum pumps, as they were still capable of evacuating the entire system to 5 microns of mccury, absolute pressure, within 35 to 40 minutes time.

4. The water-cooled condenser installed within the bell jar was very adapt at providing the necessary condensing surface for the collection of the distillate molecules that were evaporated from the rotor surface. It was seen by actual observation that the condensation was very rapid and the liquid drained off the condenser in almost a steady stream.

VI. SUMMARY

Molecular or short-path distillation is one form of high vacuum distillation, and offers a means of separating heat-sensitive, high-boiling materials which cannot be separated by conventional methods. Many of the so-called "undistillables," which include the natural fats and waxes, sugar derivatives, petroleum residues, plasticizers, dyes, and a host of other substances now fall within the scope of molecular distillation. The use of the process is limited, however, due to the high costs involved and the poor separatory powers which are still characteristic of this method of distillation.

Although there have been many mechanical improvements in the constructional designs of the molecular stills since the early beginnings of the crude pot stills, they still remain too inflexible in design and too compact in construction to allow the proper study of the fundamental factors affecting their performance. One of the problems yet to be solved satisfactorily is the prevention of leaks through the bearings and packings of the rotor shaft. It was the purpose of this investigation to redesign and reconstruct a five-inch magnetically-driven centrifugal

molecular still, and by so doing to eliminate leaks around the rotor shaft by enclosing the rotor within the still head, and to gain flexibility by the use of a high-form bell jar, and a water-cooled condenser.

The still head assembly consisted of a number of integrated units, many of which were of the same design and construction as the units used in other stills by previous investigators. Some of the more important changes were; a magnetic drive, consisting of an inner flange of magnets connected to the rotor shaft, and an outer flange of magnets connected to the drive shaft of the motor, a nitralloy rotary gear feed pump, specially fitted and constructed as a vacuum tight unit, and a water-cooled copper condenser, so constructed that its distance from the rotor surface could be varied. The feed, residue, and distillate tanks, as well as all flow lines for the feed, were constructed of aluminum or aluminum tubing; the vacuum lines were constructed of copper tubing, and all joints on the vacuum lines were either bronzed or silver-soldered to minimize the possibilities of leaks. All other constructional features of the still, such as electrical connections, vacuum connections to the base plate, bell jar seal, and thermocouple construction, were the same as used by previous investigators.

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