

THE EFFECT OF CONDENSER TEMPERATURE AND LOCATION ON THE MOLECULAR
DISTILLATION CHARACTERISTICS OF STEARIC ACID

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I. INTRODUCTION

In general, distillation can be defined as that operation used for the separation of constituents in a liquid mixture by partial vaporization of the mixture and separate recovery of the vapor and residue. The field of distillation is comprised of four major divisions; they are commonly referred to as fractional, destructive, azeotropic, and molecular distillation.

A need for separating heat sensitive substances with high molecular weights and/or high boiling points resulted in the first design of the molecular still as early as 1910. The modern molecular still differs somewhat from its predecessor by the adoption of auxiliary equipment that is capable of producing higher vacuums. At present, molecular distillation offers the only means for distilling special organic compounds such as vitamins, plasticizers, etc., at 50 to 250 degrees centigrade lower than conventional means of distillation.

Numerous findings have indicated that the distance between the evaporator and condenser of the molecular still is not as critical, as far as mean-free path design

criteria are concerned, as formerly believed. It had been recommended that studies be made on the present apparatus at Virginia Polytechnic Institute to determine exactly how critical this factor is.

The purpose of this investigation was to study the effect of condenser temperature and condenser location on the molecular distillation characteristics of stearic acid, and from this study, determine this relationship of condenser temperature and location to be used in centrifugal molecular still operation.

II. LITERATURE REVIEW

A review of the available literature was made in order to gain an insight into the theory and development of molecular distillation. The history and development, theory, factors effecting molecular distillation, and method of evaluation are considered in the following sections.

History and Development

In the past, molecular distillation has been utilized almost solely as a laboratory technique for research purposes. It was not until recently that its application was extended to the processing of industrial chemicals at rates approximating a million gallons annually⁽²¹⁾.

Historical. During the period from 1910 to 1925, when substances of higher molecular weights and boiling points were coming under study, it became apparent to chemist that there was need for a better type of still for recovering organic chemicals⁽¹⁰⁾. The first molecular still appeared, to have been designed in 1922 by Bronsted and Havesy⁽¹⁾ to separate the isotopes of mercury. The first application of molecular distillation

to organic chemicals, and the first comprehensive statement of the potentialities of the operation were made by Burch (5,6) in England. Both Waterman (31), who applied molecular distillation to labile substances in Holland, and Washburn (30), who employed the technique in the United States, were responsible for important early developments in molecular distillation.

The lack of development of high-vacuum condensation and fractionating pumps had hindered the progress of molecular distillation. These developments were brought about in 1930 by the Eastman Kodak Company, after which the first industrial use of molecular distillation was employed to separate vitamin A and E from marine and vegetable oils.

Prototype Molecular Stills. The development of the molecular still has progressed through three prominent stages (18). The first still was 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. The second still was the falling-film still where the distilland is metered to the vacuum chamber, degassed, and allowed to pass in a film down the walls of the evaporator. The third still was the centrifugal still

where the distilland is metered to pass over a rotor generally housed in a bell jar and supported on a shaft to a motor drive.

Molecular Distillation Studies at Virginia Polytechnic Institute. Considerable work has been done in the field of molecular distillation at Virginia Polytechnic Institute. A summarization of these investigations are presented in the following sections.

Work of Coli. The first centrifugal molecular still at the Virginia Polytechnic Institute was constructed by Coli in 1949. The entire still was constructed of glass with the exception of the base plate, rotor, and gutter, which were made of brass. Such variables as film thickness of distilland on the rotor surface, initial feed temperature, and angle of inclination of the rotor surface were tested.

Work of Lombardi and Tyler. Using the all-glass still constructed by Coli, Lombardi ⁽²³⁾ and Tyler ⁽²⁹⁾ in 1950 and 1951, respectively, made the first efforts to molecularly distill fatty acids at the Virginia Polytechnic Institute. Lombardi determined the elimination maxima of lauric, myristic, palmitic, and stearic acids, while Tyler applied molecular

distillation to the separation of a mixture of palmitic acid and stearic acid.

Work of Bull and Demakis. Bull (2) and Demakis (8) constructed the first all-metal still at Virginia Polytechnic Institute. Using basically the same ideas set forth by Coli, they reconstructed the unit using copper tubing, tanks, and flow lines in place of the glass ones incorporated in the previous still. An investigation was made of such operating variables as temperature, pressure and time of distillation, rotor speed and rate of distillation, the shape of the elimination curve, and the position of the elimination maximum. In addition to the operating variables, composite elimination curves for binary and tertiary mixtures of anthraquinone dyes were determined to study the effect of the pressure of more than one material on the elimination maximum of individual constituents.

Work of Demakis and Mote. The first all-metal still constructed by Bull and Demakis was modified in 1955 by Demakis (9) and Mote (24) to prevent leakage. The seal around the rotor shaft was replaced by a magnetic drive. Demakis investigated the effects of various molecular structures of fatty

acids on their distillation characteristics. Mote determined the effect of molecular weight and degree of unsaturation on the elimination maximum of several fatty acids.

Theory of Molecular Distillation

Some of the more important aspects of molecular distillation theory are considered in the following sections: the rate of distillation, the film thickness, and the mean free path criteria.

Rate of Distillation. In molecular distillation, the rate of distillation is defined by Embree (14) to be proportional to the product of its concentration and its distillability. When expressed algebraically this becomes,

$$V = kND$$

where:

V = rate of distillation, mols per second

k = proportionality constant

N = concentration, mol fraction

D = distillability, probability that any molecule will distill.

If the distilland, or the substance being distilled, is considered to be an ideal solution, then the rate of distillation can be determined from Langmuir's equation,

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

where;

n = rate of distillation, moles 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.

Bronsted and Von Hevesy⁽¹⁾ found that the rate of distillation of the components of a mixture was inversely proportional to the square root of the molecular weights of these components as indicated by the Langmuir equation. Washburn et al⁽³⁰⁾ observed that the equation was valid for solutions if no recondensation occurred on the evaporating surface; for the actual rate of distillation will always be less than the theoretical rate because of the return of some molecules to the evaporating surface.

Film Thickness. An important factor in molecular distillation is the surface of the distilling liquid⁽²²⁾. The only molecules involved in the distillation are those on the surface. The remaining molecules serve only to replenish the surface layer. For this consideration, the reasoning behind a desire for a thin film thickness is evident.

A formula⁽²⁰⁾ for the relationship 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, experimentally determined

L = film thickness, milliliters.

According to Fawcett⁽¹⁵⁾, 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 and the vapor pressure decreases, and (2) the fractionating power will decrease since excessive quantities of the less volatile will appear in the distillate and the more volatile will appear in the residue.

Mean Free Path Criteria. Glasstone⁽¹⁷⁾ defines the mean free path of a molecule as the average distance a molecule travels between two successive paths or successive collisions between molecules. Maxwell's⁽¹⁷⁾ expression for the calculation of the mean free path is:

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

where:

l = mean free path, centimeters

N = Avagadro's number, 6.06×10^{23}

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

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

π = a constant.

Various formulae for the mean free path of like molecules and molecules in the presence of unlike molecules have been developed. The calculation for

large nonspherical molecules using Maxwell's equation and other formulae gives mean free paths which are likely to be in error. There is some indication that oil molecules for example have "undeflected mean free paths" several times longer than those calculated by classical theory⁽¹⁸⁾.

Factors Affecting Molecular Distillation

In molecular distillation, the rate of distillation of any compound 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⁽¹⁶⁾. Considered in the sections that follow are such variables as operating pressure, temperature of distilland, film thickness, splashing, rotor speed, angle of inclination of rotor surface, temperature of condensing surface, and distance between evaporator and condenser.

Operating Pressure. Once the residual gas pressure in the still equals the vapor pressure of the distilland at the temperature of operation, further reduction in pressure affects no further increase in the rate of distillation. Thus in true molecular distillation, the rate of evaporation is independent of pressure.

Bull (4) reports that an appreciable effect was noted in the shifting of the elimination maximum when the operating pressure was varied on the distillation of 1,4-diethyldiaminoanthraquinone. For absolute pressures between 2 and 100 microns of mercury, elimination maxima between 144 and 177 °C were obtained.

Temperature of Distilland. An increase in temperature of the distilland causes an increase in the vapor pressure and hence an increase in the rate of distillation. However, it should be remembered 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 increased vapor pressure.

Film Thickness. The exact effect of film thickness on molecular distillation is unknown. In molecular still design attempts are generally made to reduce the film thickness to insure fast diffusion rates such that the surface or distilling layer is representative of the fluid beneath.

Splashing. The degree of separation obtainable in molecular distillation can be affected by splashing because of the entrainment of the less volatile substance in the distillate. Improper degassing and too rapid evaporation have been mentioned ⁽²⁰⁾ to be causes of splashing.

Rotor Speed. Although the amount of distillate collected on the condenser varies with rotor speed, the shape of the elimination curve or the position of the elimination maximum is not affected by the rotor speed; for Hickman ⁽²¹⁾ reported that when the rotor speed was increased from 100 to 3000 revolutions per minute the amount of distillate collected on the condenser was quadrupled, while Bull ⁽³⁾ found that rotor speeds from 1000 to 3000 revolutions per minute had no effect on either the shape of the elimination curve or the position of the elimination maximum.

Angle of Inclination of Rotor Surface. From an investigation made by Coll ⁽⁷⁾ angles of inclination of the rotor surface between $52\frac{1}{2}$ and 75 degrees had no effect on the separation.

Temperature of Condensing Surface. The primary consideration of the condensing surface is that it be

at a temperature sufficiently below that of the distilling surface to prevent any revaporization of the distillate on the condenser⁽¹⁷⁾. Any further reduction in the condenser temperature will not improve either the rate of distillation or condensation.

Distance Between Evaporator and Condenser. Variations in the distance between the evaporator and the condenser do not produce any very noticeable variations in still performance provided the distance does not exceed the mean free path. Extremely small distances, however, will decrease the distillation rate, for the closeness of the condenser surface to the evaporating surface tends to permit the return of some of the vaporized molecules to the distilling surface which would not have returned otherwise.

Method of Evaluation

When molecular distillation is performed under standardized conditions, and the resulting yields of the product are plotted against the temperature at which the fraction had been distilled, a distillation curve is provided. Hickman⁽¹⁹⁾ has called this curve the "elimination curve." Much can be learned about

molecular distillation by studying the characteristics of the elimination curve. This type of curve has the general form shown in Figure 1 page 16.

The amount of substances distilling in each fraction increases as the temperature increases until the yields of the distillate finally reach a maximum and then decrease to zero, because the supply of the substance in the distilland becomes nearly exhausted⁽¹³⁾. The elimination curve has an easily recognized shape and a maximum which can be located to within a few degrees.

The shape of the elimination curve may be used to reveal many of the properties of a substance and the kind of treatment that is received during distillation. The effect of various operational variables on the elimination curve are considered in the following sections.

Distillation Time. The position of the elimination curve is greatly affected by the length of time during which the substance distills at each temperature in the series⁽²⁴⁾. It can be seen from the curve in Figure 2, page 17, that for distillation times of 1, 2, and 4 minutes at each temperature that as the time of distillation increases, the maximum of the curve shifts to a lower temperature value.

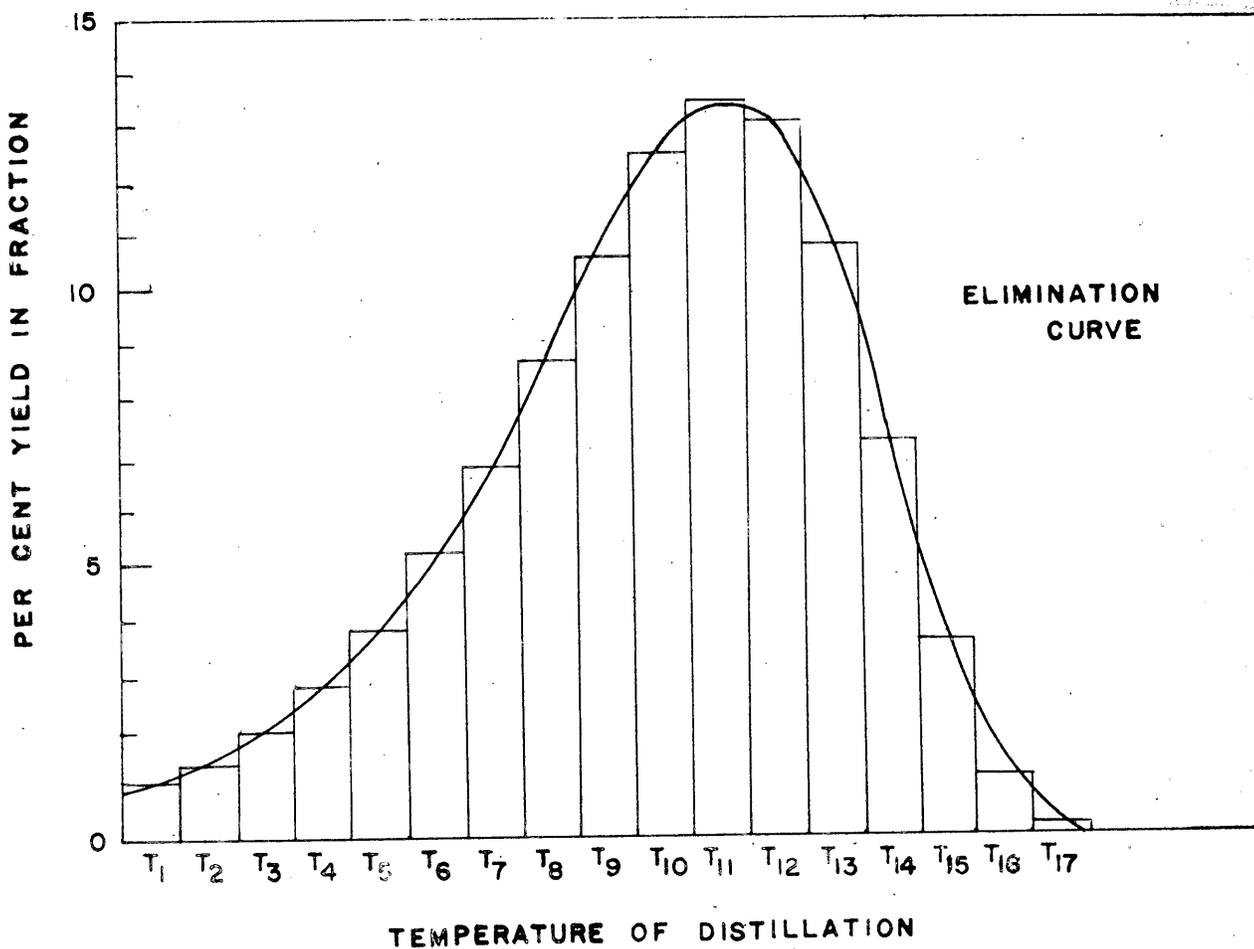


FIGURE 1. YIELD VS. TEMPERATURE CHART AND ELIMINATION CURVE

EMBREE, N.D.: THEORY OF ELIMINATION CURVE, IND. ENG. CHEM. 29, 975 (1937).

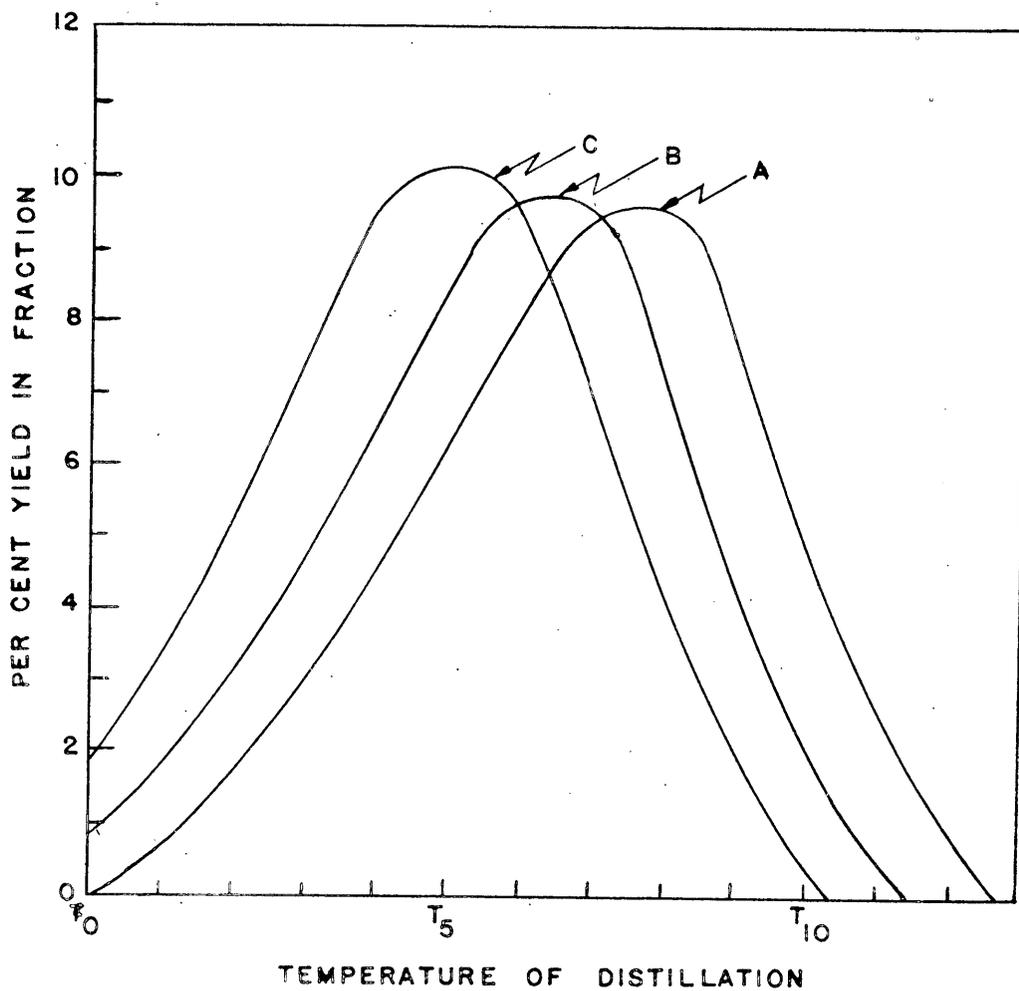


FIGURE 2. EFFECT OF TIME OF DISTILLATION ON ELIMINATION CURVES

- A. DISTILLATION FOR 1 MINUTE AT EACH TEMPERATURE.
- B. DISTILLATION FOR 2 MINUTES AT EACH TEMPERATURE.
- C. DISTILLATION FOR 4 MINUTES AT EACH TEMPERATURE.

EMBREE, N. D.: THEORY OF ELIMINATION CURVE, IND. ENG. CHEM. 29, 978 (1937).

Initial Temperature. The temperature at which distillation is started has no effect on the temperature at which the maximum occurs, but raises the relative yields at the various temperatures as shown in Figure 3 , page 19.

Heat of Vaporization. Because distillation is almost proportional to the vapor pressure, substances with a high heat of vaporization will have a distillation curve that differs from substances with a low heat of vaporization⁽²⁴⁾ . Substances with a high heat of vaporization have distillation curves that are narrow and steep, as compared with substances with a low heat of vaporization which have broader and flatter elimination curves. Examples are shown in Figure 4 , page 20 .

Solvent Distilling Over with Potent Material. In many distillations, it is desirable to have a small amount of the solvent distilling over with each fraction to aid in drainage or washing of the distillate from the condenser surface⁽²⁴⁾ . Figure 5 , page 21, shows the typical difference in the elimination curves when two per cent of the total solvent distilled over with the potent material in each fraction.

Effect of the Use of An Arithmetic Series of Temperatures. If the distillation were carried out using an arithmetic series of temperatures instead of a series chosen so that the distillability increases by a constant factor at each temperature, the elimination curve would have a somewhat different shape⁽¹⁹⁾ as shown in Figure 5, page 21.

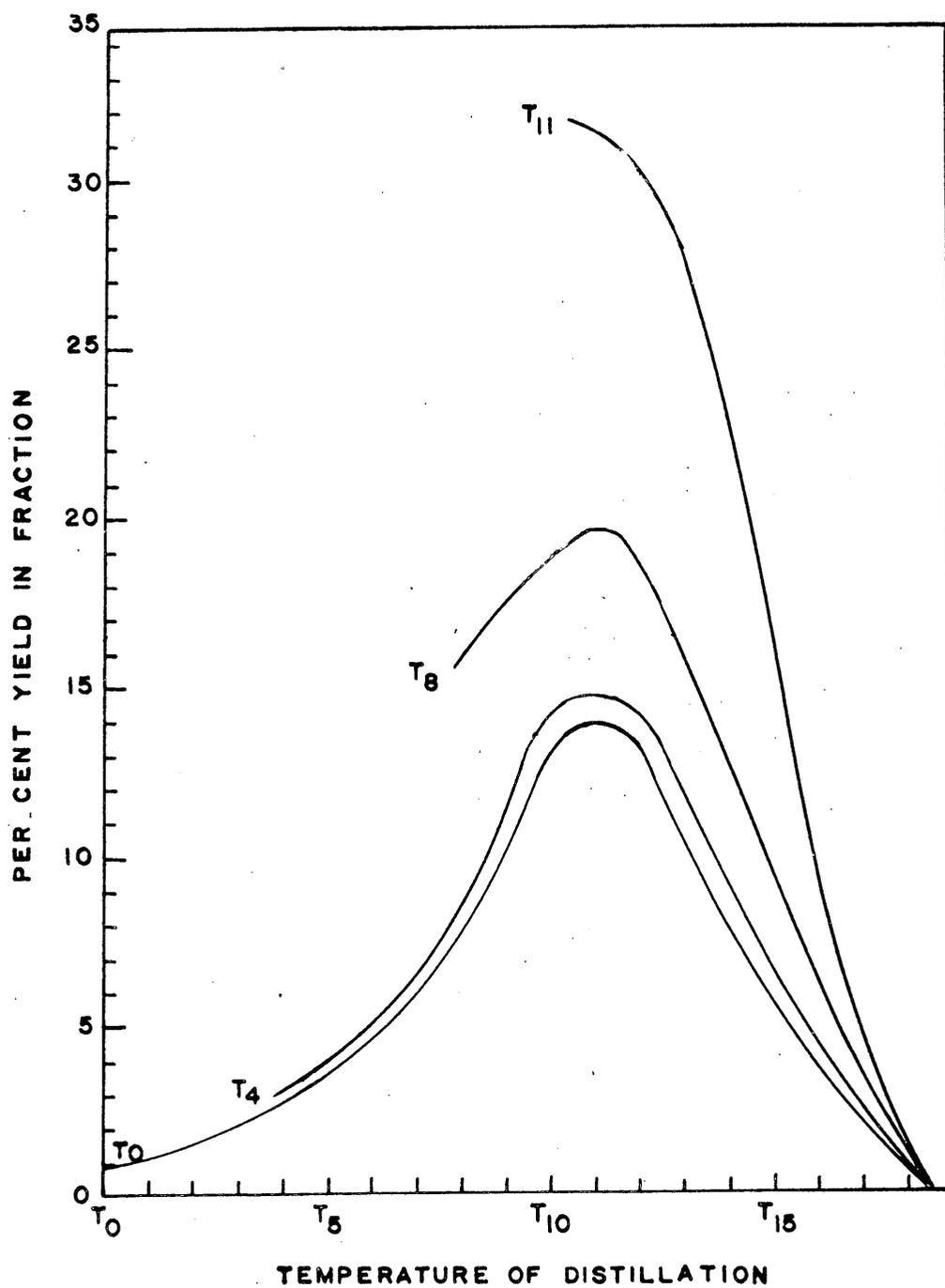


FIGURE 3. EFFECT OF INITIAL TEMPERATURE ON ELIMINATION CURVES

EMBREE, N.D.: THEORY OF ELIMINATION CURVE, IND. ENG. CHEM. 29, 978 (1937).

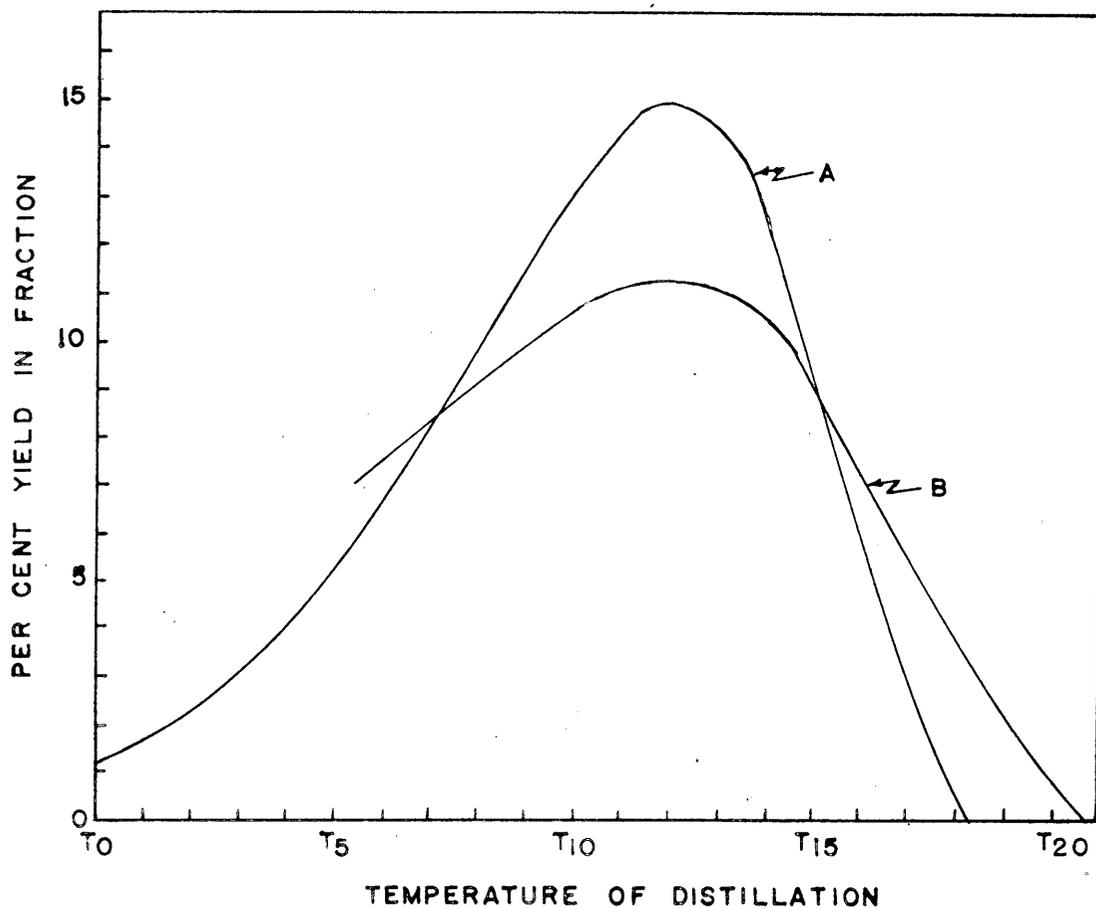


FIGURE 4. EFFECT OF HEAT OF VAPORIZATION ON ELIMINATION CURVES

- A. SUBSTANCE WITH HIGH HEAT OF VAPORIZATION.
- B. SUBSTANCE WITH LOW HEAT OF VAPORIZATION.

EMBREE, N.D.: THEORY OF ELIMINATION CURVE, IND. ENG. CHEM. 29, 978 (1937).

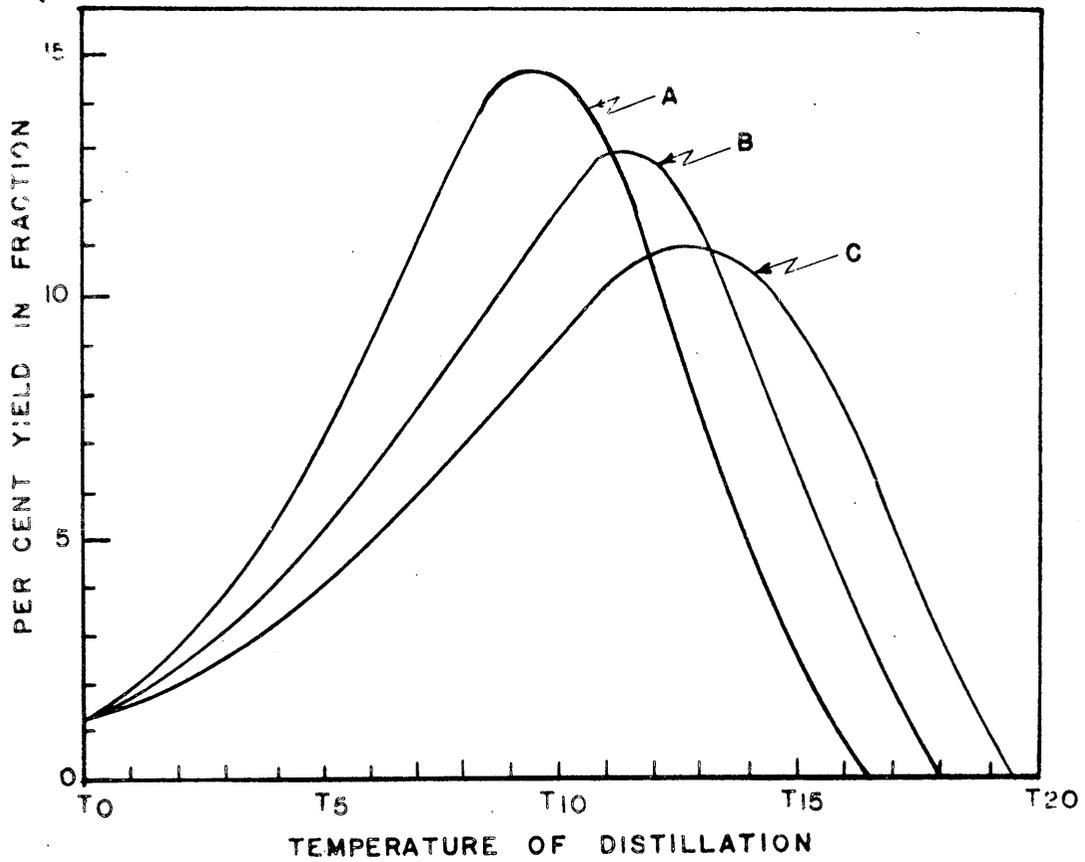


FIGURE 5. ELIMINATION CURVES FOR VARIOUS CONDITIONS

- A. TWO PER CENT OF SOLVENT DISTILLING IN EACH FRACTION.
- B. NO SOLVENT DISTILLING WITH POTENT MATERIALS.
- C. DISTILLATION WITH AN ARITHMETIC SERIES OF TEMPERATURES.

EMBREE, N.D.: THEORY OF ELIMINATION CURVE, IND. ENG.
CHEM. 29, 979 (1937).

III. EXPERIMENTAL

The purpose of this investigation, the plan of experimentation, the materials and apparatus used, the methods of constructional and operational procedure followed, and the data and results obtained in the investigation are presented.

Purpose of Investigation

The purpose of this investigation was to study the effect of condenser temperature and location on the molecular distillation characteristics of stearic acid, and from this study determine this relationship of condenser temperature and location to be used in centrifugal molecular still operation.

Plan of Experimentation

The plan of investigation for the study of the effect of condenser temperature and location on the molecular distillation characteristics of stearic acid includes a review of the literature, equipment modifications, preliminary tests, experimental tests, and analysis of results.

Literature Review. A survey of past literature pertaining to molecular distillation was made to gain an insight into the work of previous investigators. Particular attention was given to experiments performed at the Virginia Polytechnic Institute.

Equipment Modification. The centrifugal molecular still built by Mote and Demakis (1955) was modified to improve the control of temperature and flow variations. Changes in the equipment included the re-location of feed valve and feed tank.

Preliminary Tests. A number of tests were made to familiarize the operators with the technique to be used in the operational tests.

Experimental Tests. A blend of stearic acid and constant yield-oil developed by Mote and Demakis⁽¹⁰⁾ was used along with a standardized technique of still operation. Experimental tests were made with the condenser plate

placed in three locations, $3/4$, $2-1/2$, and $5-3/8$ inches from the residue gutter. At each of these locations three temperature levels were studied, 25, 35, and 45 degrees centigrade.

Analysis of Results. The effect of condenser temperature and location was determined by studying the characteristics of the elimination curve obtained from each condition investigated and analyzing these results by statistical means. Recommendations were made for future studies and modifications of the centrifugal molecular still.

Materials

The source and description of the materials used throughout the investigation are presented in the following sections.

Acid, Hydrochloric. C. P., code 1090, meets A. C. S. specifications, 37-38 per cent, lot No. E111005. Obtained from General Chemical Division, Allied Chemical and Dye Corporation, New York, N. Y. Used to standardize potassium hydroxide solution.

Acid, Stearic. C. P., meets A. C. S. specifications. Obtained from Fisher Scientific Company, Silver Spring, Md. Used as the investigative media in the distillation studies.

Alcohol, Ethyl. Technical grade, 95 per cent, U. S. P., meets A. C. S. specifications. Obtained from U. S. Chemicals, Inc., Baltimore, Md. Used in titration of the distillate samples obtained from the molecular still.

Benzene. Purified, code 1444, lot No. G-100-J. Obtained from General Chemical Division, Allied Chemical and Dye Corporation, New York, N. Y. Used to clean equipment and glassware during the investigation.

Grease. High vacuum, celvacene heavy. Manufactured by Consolidated Vacuum Corporation, Rochester, N. Y. Used in testing for leaks around fittings and bell jar on molecular still set-up.

Oil. Cenco, hyvac, Nos. 93050C and 93055C. Obtained from Central Scientific Company, Chicago, Illinois. Used as lubricant and sealing fluid in hyvac and megavac pumps.

Oil. Mineral, colorless, light and heavy grades; light oil boiling range at 10 mm Hg, 330 to 360 °C; heavy oil boiling range at 10 mm Hg, 360 to 390 °C. Manufacturer unknown. Obtained from Tech Drug Store, Blacksburg, Va. Used as component of feed mixture as carrier oil for acid.

Oil. Voltesso No. 36. Obtained through courtesy of Esso Standard Oil Company, Linden, New Jersey. Used as component of feed mixture as carrier oil for acid.

Phenolphthalein. C. P., meets A. C. S. specifications. Obtained from Palo-Myers, Inc., New York, N. Y. Used as indicator in the titration of the samples obtained from still.

Potassium Hydroxide. C. P., pellets, lot No. E-355, meets A. C. S. specifications. Obtained from General Chemical Division, Allied Chemical and Dye Corporation, New York, N. Y. Used as neutralizing agent in the titration of the acid in the samples obtained from the still.

Sodium Carbonate . C. P. anhydrous, catalog No. S-263. Obtained from Fisher Scientific Company, Silver Spring, Md. Used to standardize hydrochloric acid.

Water, Tap. Obtained from V. P. I. water main. Used as cooling water in condenser of still.

Apparatus

The following section contains the description and application of the apparatus used in this investigation.

Balance. Analytical, with chainweights and magnetic damping attachments, capacity 200 grams each pan, sensitivity 1/20 milligram. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to weigh acid samples in preparation of feed mixture.

Cell. Dry, No. 6, 1-1/2 v, two used, connected in series. Manufactured by National Carbon Company, Cleveland, Ohio. Used in conjunction with potentiometer for temperature measurements.

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.

Gage. McLeod, vacuum type MG-07, tripole range, table model, pump operated, range 0 to 5000 microns. Manufactured by Consolidated Vacuum Corporation, Rochester, N. Y. Used to measure vacuum in the molecular still.

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.

Glassware. Assorted pyrex glassware such as beakers of various sizes, erlenmeyer flasks, graduated cylinders, funnels, and burets. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used in feed preparation and sample titrations.

Heater. Immersion coil type, flexible copper sheath, 115 v, 100 w. Obtained from American Instrument Company, Silver Spring, Maryland. Used as the heating element in the constant-temperature bath.

Hot Plate. Fisher autemp, catalog No. 11-467-1, model A, 115 v, ac. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to preheat feed mixture prior to addition to still, and to heat samples during titrations.

Molecular Still and Accessories. The component parts of the molecular still, and necessary operational accessories and their uses, are described in detail by Note ⁽²⁴⁾ in an earlier work.

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. Electric, type ADS, style 957656-B serial KU, 1/4 hp, variable speed. Manufactured by Westinghouse Electric Corporation, Pittsburg, 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 1.7 v. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to measure voltage across thermocouples during operation of molecular still.

Tachometer. Hand tachometer, type 25-A, serial No. 679, 200 to 10,000 rpm, triple scale. Obtained from the Metron Instrument Company, Denver, Colorado. Used to measure the speed of the rotor.

Tank. Open end, stainless steel, 2-gallon capacity. Manufacturer unknown. Used as the constant-temperature bath for condenser cooling water.

Thermometer. Certified, catalog No. 15-040, range -10 to 400° C in 1/10° C increments. Obtained from Eimer and Amend, New York, N. Y. Used in calibration of thermocouples and to measure temperature of water in constant-temperature bath.

Thermoregulator. Liquid-filled, expansion type thermostat, type R-1, No. 4980281G16, range 65 to 135° F, 110/220 v, 25 amp. Manufactured by General Electric Company, Schenectady, N. Y. Used to control the constant-temperature bath heating element.

Pump. Centrifugal, All-Rubber Bontam, No. AR-4360, complete with enclosed drive. Obtained from Schaar and Company, Minneapolis, Minnesota. Used to circulate cooling water to the still condenser coils.

Method of Procedure

The method of procedure utilized in this investigation is discussed in the following sections (1) modification of centrifugal molecular still, (2) preliminary tests, (3) operational procedure (4) distillation of stearic acid at three condenser plate temperature levels with various condenser plate locations, and (5) analytical procedure.

Modification of Centrifugal Molecular Still. The centrifugal molecular still built by Mote and Demakis (1955) was modified to improve temperature and flow variation control. Two changes were made in the equipment to bring about this improvement. First, the feed tank was elevated twenty-two inches above its original position, and secondly, the feed valve was installed in the feed line approximately fifteen inches before the preheater. By placing the feed valve above the preheater, as opposed to the original location of below the preheater, it was reasoned that the flow of feed would be subjected to less variation.

Preliminary Tests. A number of preliminary tests were performed using a successful operational technique developed by Mote and Demakis ⁽¹¹⁾ to familiarize the operators with the operating characteristics of the still and to make it possible to obtain data that would be on a comparable basis with data of previous experiments.

Operational Procedure. Before the actual operational test procedure could be started, the feed had to be prepared

and charged to the feed tank, the vacuum system had to be sealed and placed into operation, and the feed had to be heated and degassed. The distillate fractions could then be collected at the proper temperatures and feed rates. After the completion of the test, the system had to be shut down and prepared for subsequent tests. In describing the various procedures, reference is made to the schematic flow diagram, Drawing 1, page 32.

Preparation of Feed. A constant-yield oil was prepared for each test by blending 225 milliliters of No. 36 Voltesso, 175 milliliters of light mineral oil, and 425 milliliters of heavy mineral oil. This provided an initial feed volume of 825 milliliters, of which 35 milliliters were considered as feed holdup in the flow lines.

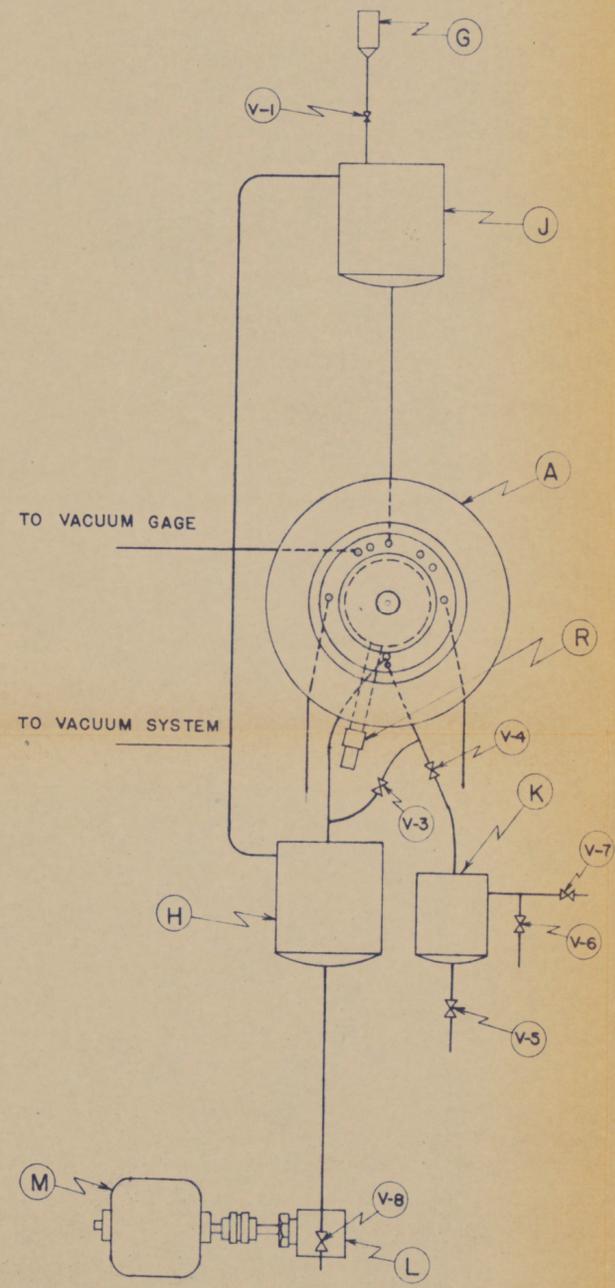
To this blend was added approximately 0.5 gram of stearic acid. The acid was accurately weighed to ± 0.0002 grams with an analytical balance, since the weight of acid used was important in the calculation of yields for each temperature fraction. After the addition of the acid, the feed mixture of acid and oils was heated to approximately 60 degrees centigrade to completely dissolve the acid before the oil solution was charged to the still.

Preparation of McLeod Gage. The hyvac pump used to operate the McLeod gage was turned "on" as the first step in readying the vacuum system for operation.

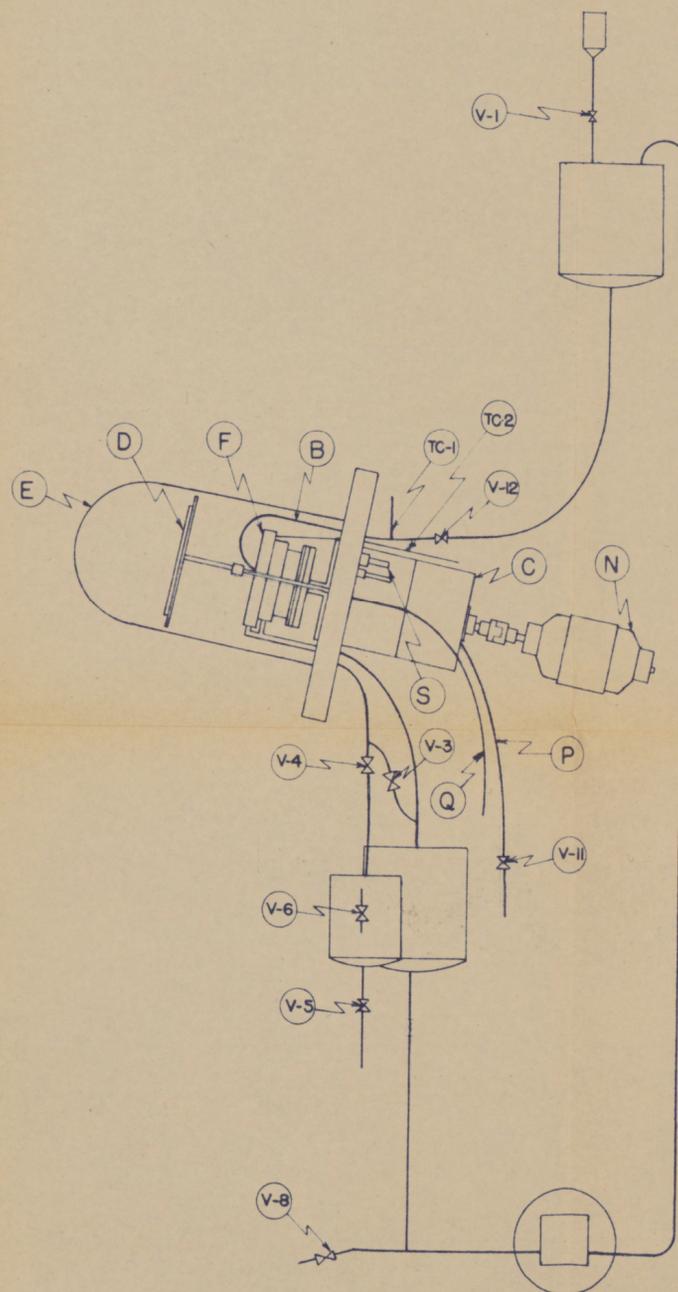
The gage was opened to the pump to allow the mercury in the gage reservoir to be relieved of any oil vapors from a previous test and to prevent surging of mercury in the gage when the still was evacuated. This "degassing" of the mercury required at least 15 minutes before the gage functioned properly.

Charging of Feed to Still. The feed mixture was charged by means of loading tank G and valve V-1. Valve V-12 was closed before charging the feed to the still to prevent feed from spilling onto the rotor. Valve V-1 was closed after the entire feed volume had drained into the feed tank J.

Start-up Procedure. In readying the still for operation, the following valves, V-3, V-6, and V-11, were opened. The silicone gasket was placed into position in the groove of the base plate, A, Drawing 1, page 32, and the bell jar, E, Drawing 1, page 32, was then centered on the gasket. The bell jar was pressed firmly against the gasket and the four megavac pumps were turned "on." The vacuum in the system served to hold the bell jar in place once the system was evacuated. The McLeod gage was observed at all times during the start-up, and the backing pressure to the gage (hyvac pump pressure) was balanced against the system pressure to prevent mercury from being drawn from the gage into the flow lines of the still.



FRONT ELEVATION



RIGHT SIDE ELEVATION

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 VACCUM LINE
- S - SPARK PLUGS
- TC-1 - FEED THERMOCOUPLE
- TC-2 - RESIDUE THERMOCOUPLE
- V-1 TO V-12 - VALVES

NOTE: ANGLE IRON SUPPORT FOR SYSTEM NOT SHOWN

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

CENTRIFUGAL MOLECULAR STILL
WITH GRAVITY FEED

SCALE: 1/8" = 1"	DATE	
DRAWN BY: J.F.M.	3/1/55	CASE NO. 55
CHECKED BY: J.F.M.	3/1/55	FILE NO. 799
APPROVED BY: J.F.M.	3/3/55	DRWG NO. 1

While the system was being evacuated, the tap water for the cold junction of the thermocouples was turned "on" and the potentiometer was balanced against the standard cell. The heating element for the constant-temperature bath and the condenser water pump were also turned "on."

Heating and Degassing of Feed. The rotor motor, N, was turned "on" and the controlling powerstat adjusted for a rotor speed of approximately 1000 revolutions per minute. The feed was allowed to flow to the rotor by opening valve V-12. The feed rate was adjusted to approximately 60 milliliters per minute. The exact feed rate was unimportant at this point since this was primarily a preheating and degassing period. The feed recycle pump, L, was turned "on" to return the feed from the residue tank, H, to the feed tank, J. Initially, the voltage input to the rotor heater coil was about 30 volts. The voltage to both the feed tank heater and the residue and distillate tank heater was adjusted and remained at 50 volts.

The preheating and degassing operation required at least two hours. When the absolute pressure in the system leveled off at 20 microns or below, the degassing operation was considered complete.

Collection of Distillate Fractions. When the degassing of the feed was complete, the feed rate and feed and residue temperatures were adjusted in preparation

for collection of the first distillate fraction. The feed rate was adjusted by partially opening or closing valve V-12. This adjustment could only be based on the visual observation and approximation of the feed rate by the operator. The temperature of the feed and residue were adjusted and maintained at the proper temperatures (62 and 86 degrees centigrade, respectively for the first distillation fraction) by powerstat control of the heater voltage. The feed was permitted to recycle to the rotor, to the residue tank, and back to the feed tank. Any distillate which collected in the bell jar during the preheating, degassing, and temperature adjustment period was continuously returned to the feed mixture through bypass valve V-3.

When the proper feed and residue temperatures were reached, the collection of the first (86-degree) distillate fraction was started by closing valve V-3. The feed pump was immediately turned "off" and the electric timer was started. The total feed volume (with the exception of the feed holdup in the flow lines) was allowed to pass over the rotor. The total elapsed time for the "pass" was determined by noting when the last of the feed had left the nozzle. By dividing the feed volume by the elapsed time, the feed rate was determined.

Distillate fractions at feed rates between 60 and 65 milliliters per minute were considered "acceptable." If the feed rate was not within this range, the distillate fraction was "not acceptable." The two possibilities require different procedures.

Acceptable Feed Rate Procedure. If the feed rate were satisfactory, the distillate collected at the lowest point of the bell jar was drained into the distillate tank, K, by opening valve V-4. When drainage was complete, valve V-4 was closed, valve V-3 was opened, the feed pump was turned "on," to again recycle the feed. The distillate was removed from tank K by turning "off" the distillate-tank megavac, venting the tank to the atmosphere by opening valve V-7, and draining the distillate by opening valve V-5. After complete drainage of the fraction, valve V-5 and V-7 were closed and the megavac pump again turned "on" to reevacuate the distillate tank for collection of the next fraction.

The distillate fraction was collected in a graduated cylinder for measurement of the oil volume which distilled over with the fatty acid. This was done in order to determine the new volume of feed in the still for feed rate calculations for the subsequent fraction.

When a fraction had been collected, feed and residue temperatures were increased in preparation for taking the next higher fraction. Minor adjustments of the feed rate were made, again on the judgment of the operator. It was important that at least 10 minutes be allowed between the end of one fraction collection and the beginning of the next to insure that all feed mixture was returned from the residue tank to the feed tank.

Fractions were collected at residue temperatures of 86, 92, 98, 104, 110, 116, 122, 128, 134, 140, and 146 degrees centigrade with the feed temperature in each case being 24 degrees below the residue temperature. In other words, heating of the feed on the rotor raised its temperature by 24 degrees.

Unacceptable Feed Rate Procedure. If the feed rate were unsatisfactory (that is, below 60 or above 65 milliliters per minute) the distillate was returned to the feed tank in preparation for recollection of the fraction. This was done by opening valve V-3 to allow the distillate to drain from the bell jar to the residue tank. The feed pump was again turned "on" to recycle the feed. The feed rate was adjusted by partially opening or closing valve

V-12 ; after at least 10 minutes of pumping and recycling the feed, the collection of an acceptable fraction could again be attempted.

Shut-down Procedure. As soon as the final distillate fraction was collected, the power to the rotor heater, the feed preheater, the tank heaters, the feed pump, the rotor motor, the condenser water heater, and the vacuum pumps were turned "off". The tap water for the thermocouple cold junction was also turned "off".

Preparation of the Still for Subsequent Tests.

The bell jar was removed, and the residue remaining in tank H was drained by opening valve V-8. As soon as drainage of the residue was complete, valve V-8 was closed, and approximately 250 milliliters of light grade mineral oil (preheated to 100 degrees centigrade) was added to the feed tank through the loading tank, G, by opening valve V-1. The feed pump and rotor were briefly turned "on" and the "wash" oil was circulated through the flow lines and tanks and over the revolving rotor for several minutes. The "wash" oil was then drained by opening valves V-8 and V-5. The system was then considered "clean" and ready for the next test.

Distillation of Stearic Acid at Three Condenser Plate Temperature Levels with Various Condenser Plate Locations.

Stearic acid was distilled using the experimental procedure described above. Experimental tests were made with the condenser plate placed in three locations, $3/4$, $2-1/2$, and $5-3/8$ inches from the residue gutter of the rotor evaporator. At each of these locations three temperature levels were studied, 25, 35, and 45 degrees centigrade. The operating conditions for each test were as follows: (1) feed rate of 60 to 65 milliliters per minute, (2) operating pressure of 20 ± 2 microns of mercury, absolute, or below, after the removal of the first and second fractions, (3) a 24-degree centigrade temperature differential between the feed and the residue, (4) one pass of the feed over the rotor per fraction, and (5) a rotor speed of 1000 ± 50 revolutions per minute.

Analytical Procedure. The stearic acid fractions were analyzed by titration with standardized potassium hydroxide solution using a modification of the method presented by Snall and Biffen ⁽²⁸⁾. The entire fraction of stearic acid and oil solution was poured from the graduated cylinder, used for collection, into a 250-milliliter erlenmeyer flask. Fifty milliliters

of 95 per cent ethyl alcohol that had been neutralized to the phenolphthalein end point with 0.0169 normal alkali solution were added to extract the stearic acid from the oil. The immisible liquids were heated to boiling and the flask swirled to dissolve the free stearic acid as completely as possible in the alcohol layer. The mixture was titrated with 0.0169 normal potassium hydroxide solution (with continued swirling) until the pink end point was reached and persisted for at least 30 seconds. The weight of the acid in each fraction was calculated directly from the volume of potassium hydroxide solution in the titration.

Data and Results

The data and results of this investigation are presented in tabular and graphic form.

Thermocouple Calibration. The calibration data for the feed and residue thermocouples are given in Table I, page 40, and the calibration curve is given in Figure 6, page 41.

Constant-yield Oil. A summary of the data obtained for the distillation of constant-yield oil during the operational tests is given in Table II, page 42.

Summary of Individual Elimination Maxima. A list of the individual elimination maxima of stearic acid for three condenser temperatures and three condenser locations is presented in Table III, page 43.

Molecular Distillation of Stearic Acid for Three Condenser Temperatures and Three Condenser Locations. The data obtained from the molecular distillation of stearic acid for three condenser temperatures and three condenser locations is presented in Table IV, page 43; and the elimination curves for these conditions are presented in Figures 7 to 19, pages 45 to 57.

TABLE I

Calibration Data for Feed and Residue Thermocouples

Temperature °C	Potentiometer Readings	
	Feed Thermocouple mV	Residue Thermocouple mV
0	-0.60	-0.60
50	1.63	1.60
60	1.91	2.00
70	2.55	2.61
80	2.86	2.90
90	3.28	3.20
98	3.63	3.66
100	3.74	3.71
110	4.22	4.08
120	4.55	4.50
130	4.95	5.10
140	5.47	5.65
150	6.00	5.95
160	6.45	6.43
170	7.00	6.90

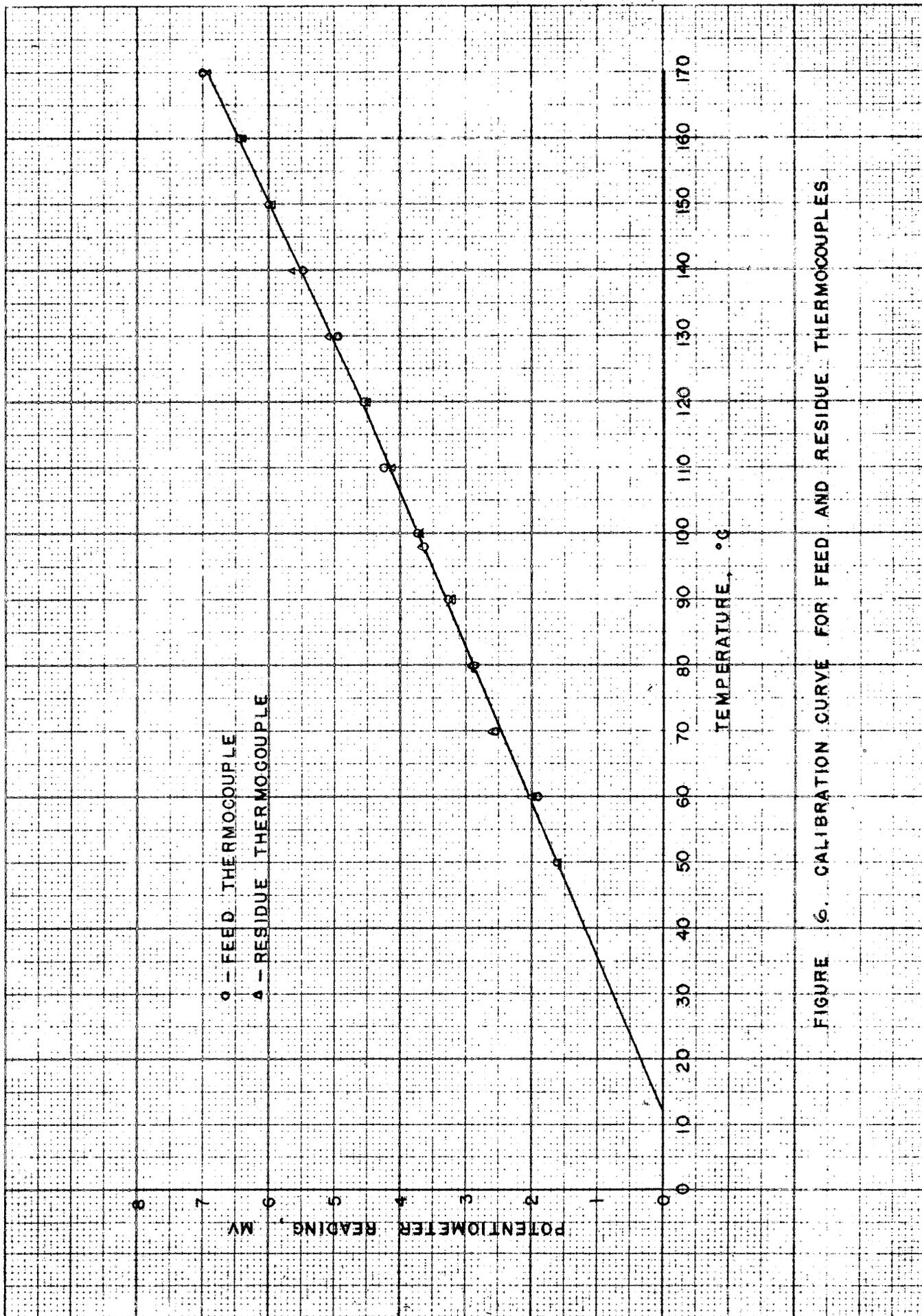


FIGURE 6. CALIBRATION CURVE FOR FEED AND RESIDUE THERMOCOUPLES

TABLE II

Molecular Distillation of Constant-Yield Oil

Temperature of Fraction	Volume of Fraction		Average Volume ml	Yield of Original Blend
	Smallest ml	Largest ml		
86	8	40	23.4	2.84
92	25	65	38.6	4.68
98	32	55	41.0	4.96
104	33	49	38.2	4.64
110	27	46	38.0	4.61
116	33	46	39.8	4.82
122	35	56	45.5	5.51
128	40	55	46.2	5.60
134	38	58	48.0	5.82
140	39	55	45.5	5.51
Residue	--	--	420.8	51.10
Total	--	--	825.0	100.00

Data obtained from 9 operational tests on stearic acid.

TABLE III

Summary of Elimination Maxima of Stearic Acid
for Three Condenser Plate Locations
at Three Temperature Levels

Condenser Location*	Condenser Water Temperature	Elimination Maximum
in.	°C	°C
3/4	25	116
3/4	25	120
3/4	35	114
3/4	45	113
2-1/2	25	116
2-1/2	25	122
2-1/2	35	117
2-1/2	35	122
2-1/2	45	110
2-1/2	45	118
5-3/8	25	110
5-3/8	35	110
5-3/8	45	110

* Distance between condenser plate and evaporator gutter

TABLE IV

Data and Results for the Molecular Distillation (a)

of Stearic Acid for Three Condenser

Plate Locations at Three

Temperature Levels

Test Conditions	Temperature of Fraction		Absolute Pressure in Still microns	Feed Rate ml/min	Weight of Acid in Fraction gm	Yield of Acid in Fraction	
	Feed °C	Residue °C				Fraction wt%	Cumulative wt%
Test 1, 0.5044 gm Stearic Acid Condenser Water Temp: 25°C Condenser Location: 3/4 in ^(b)	62	86	13.0	62.0	0.0204	4.04	4.04
	68	92	10.0	65.0	0.0169	3.36	7.40
	74	98	12.0	64.0	0.0310	6.15	13.55
	80	104	11.0	63.0	0.0431	8.55	22.10
	86	110	11.0	61.0	0.0635	12.60	34.70
	92	116	12.0	65.0	0.0707	14.00	48.70
	98	122	12.0	61.0	0.0771	15.30	64.00
	104	128	12.0	60.0	0.0630	12.50	76.50
	116	140	13.0	62.0	0.0412	8.18	84.68
			63.0	0.0223	4.42	89.10	
Test 2, 0.5008 gm Stearic Acid Condenser Water Temp: 25°C Condenser Location: 3/4 in	62	86	11.0	60.0	0.00728	1.45	1.45
	68	92	12.0	65.0	0.0097	1.94	3.39
	74	98	13.0	62.0	0.0160	3.20	6.59
	80	104	14.0	61.5	0.0856	17.10	23.69
	86	110	15.0	64.5	0.0882	17.60	41.29
	92	116	16.0	62.0	0.1080	21.60	62.89
	98	122	15.0	63.0	0.0786	15.60	78.49
	104	128	15.0	59.0	0.0540	10.80	89.29
	116	140	15.0	65.0	0.0252	5.05	94.34
		18.0	66.0	0.0179	3.58	97.92	
Test 3, 0.5014 gm Stearic Acid Condenser Water Temp: 35°C Condenser Location: 3/4 in	62	86	14.0	62.0	0.0155	3.10	3.10
	68	92	12.0	63.0	0.0204	4.06	7.16
	74	98	13.0	63.0	0.0388	7.75	14.91
	80	104	16.0	63.0	0.0601	12.00	26.91
	86	110	13.0	62.0	0.0728	14.50	41.41
	92	116	15.0	67.0	0.0699	13.90	55.31
	98	122	15.0	63.0	0.0626	12.50	67.81
	104	128	15.0	63.0	0.0461	9.20	77.01
Test 4, 0.5350 gm Stearic Acid Condenser Water Temp: 45°C Condenser Location: 3/4 in	62	86	23.0	65.0	0.0029	0.55	0.55
	68	92	17.0	60.0	0.0136	3.94	4.49
	74	98	14.0	60.0	0.0548	10.20	14.74
	80	104	13.0	63.0	0.0660	12.30	27.04
	86	110	14.0	62.0	0.0960	18.00	45.04
	92	116	14.0	63.0	0.0985	18.40	63.44
	98	122	14.0	65.0	0.0660	12.30	75.74
	104	128	14.0	65.0	0.0544	10.10	85.84
	116	134	14.0	60.0	0.0475	8.90	94.74
Test 5, 0.5066 gm Stearic Acid Condenser Water Temp: 25°C Condenser Location: 2-1/2 in	62	86	25.0	60.3	0.0139	2.74	2.74
	68	92	22.0	64.2	0.0206	4.07	6.81
	74	98	22.0	58.6	0.0486	9.61	16.42
	80	104	23.0	63.5	0.0351	6.94	23.36
	86	110	25.0	62.6	0.0625	12.32	35.68
	92	116	27.0	62.5	0.0649	12.80	48.48
	98	122	24.0	61.6	0.0802	15.85	64.33
	104	128	24.0	61.66	0.0714	14.09	78.42
	116	140	27.0	65.0	0.0379	7.49	85.91
122	146	24.0	65.5	0.0322	6.35	92.26	
			60.0	0.0206	4.07	96.33	
Test 6, 0.4823 gm Stearic Acid Condenser Water Temp: 25°C Condenser Location: 2-1/2 in	62	86	19.0	60.0	0.0083	1.70	1.70
	68	92	14.0	63.5	0.0097	2.00	3.70
	74	98	12.0	65.0	0.0209	4.30	8.00
	80	104	12.0	65.0	0.0301	6.20	14.20
	86	110	13.0	63.0	0.0524	10.80	25.00
	92	116	18.0*	61.0	0.1040	21.50	46.50
	98	122	13.0	60.0	0.0862	17.80	64.30
Test 7, 0.5104 gm Stearic Acid Condenser Water Temp: 35°C Condenser Location: 2-1/2 in	62	86	15.0	61.5	0.0029	0.63	0.63
	68	92	14.0	66.0	0.0111	2.18	2.81
	74	98	14.0	64.5	0.0228	4.46	7.27
	80	104	14.0	64.0	0.0266	5.23	12.50
	86	110	16.0	63.5	0.0525	10.25	22.75
	92	116	15.0	65.0	0.0680	13.30	36.05
	98	122	15.0	61.0	0.1000	19.60	55.65
	104	128	15.0	61.0	0.0785	15.40	71.05
	116	134	15.0	60.5	0.0596	11.70	82.75
Test 8, 0.4933 gm Stearic Acid Condenser Water Temp: 35°C Condenser Location: 2-1/2 in	62	86	24.0	65.6	0.0043	0.88	0.88
	68	92	24.0	65.0	0.0202	4.11	4.99
	74	98	23.0	58.0	0.0308	6.26	11.25
	80	104	23.0	66.0	0.0422	8.56	19.81
	86	110	23.0	64.0	0.0408	8.30	28.11
	92	116	23.0	60.0	0.0899	18.20	46.31
	98	122	23.0	58.0	0.1075	21.80	68.11
	104	128	25.0	62.5	0.0664	13.40	81.51
	116	140	24.0	60.5	0.0514	10.40	91.91
116	140	24.0	60.5	0.0308	6.26	98.17	
Test 9, 0.5023 gm Stearic Acid Condenser Water Temp: 45°C Condenser Location: 2-1/2 in	62	86	13.0	62.0	0.0117	2.33	2.33
	68	92	7.0	58.0	0.0694	13.85	16.18
	74	98	7.0	58.0	0.0567	11.30	27.48
	80	104	7.0	68.0	0.0488	9.74	37.22
	86	110	7.0	65.0	0.0682	13.60	50.82
	92	116	9.0	66.0	0.0871	17.37	68.19
	98	122	19.0	54.0	0.0808	16.10	84.29
	104	128	10.0	54.0	0.0482	9.61	93.90
	116	134	12.0	59.0	0.0256	5.21	99.11
116	140	13.0	49.0	0.0155	3.09	102.20	
Test 10, 0.5007 gm Stearic Acid Condenser Water Temp: 45°C Condenser Location: 2-1/2 in	62	86	17.0	61.5	0.0095	1.89	1.89
	68	92	15.0	61.5	0.0212	4.23	6.11
	74	98	20.0	70.0	0.0234	4.67	10.78
	80	104	17.0	56.0	0.0682	13.60	24.38
	86	110	21.0	61.5	0.1090	21.75	46.13
	92	116	18.0	64.0	0.0852	16.98	63.11
	98	122	16.0	62.0	0.0694	13.82	76.93
Test 11, 0.5088 gm Stearic Acid Condenser Temp: 25°C Condenser Location: 5-3/8 in	62	86	13.0	64.8	0.0211	4.15	4.15
	68	92	12.0	62.0	0.0442	8.70	12.85
	74	98	10.0	68.0	0.0585	11.50	24.35
	80	104	10.0	59.5	0.1025	20.20	44.55
	86	110	10.0	64.5	0.0960	18.90	63.45
	92	116	10.0	63.0	0.0894	17.60	81.05
	98	122	10.0	61.5	0.0620	12.20	93.25
	104	128	10.0	62.4	0.0370	7.26	100.51
	116	134	10.0	58.0	0.0250	4.91	105.42
Test 12, 0.4731 gm Stearic Acid Condenser Water Temp: 35°C Condenser Location: 5-3/8 in	62	86	14.0	66.0	0.0096	2.03	2.03
	68	92	14.0	64.0	0.0379	8.01	10.04
	74	98	14.0	61.6	0.0634	13.40	23.44
	80	104	13.0	67.0	0.0580	12.30	35.74
	86	110	13.0	64.0	0.0870	18.40	54.14
	92	116	13.0	64.0	0.0786	16.60	70.74
	98	122	14.0	60.3	0.0850	17.95	88.69
	104	128	14.0	60.5	0.0417	8.91	97.60
	116	140	14.0	65.0	0.0269	5.68	103.28
116	140	14.0	59.0	0.0144	3.04	106.32	
Test 13, 0.4989 gm Stearic Acid Condenser Water Temp: 45°C Condenser Location: 5-3/8 in	62	86	13.0	65.0	0.0063	1.26	1.26
	68	92	13.0	62.5	0.0194	3.89	5.15
	74	98	15.0	64.3	0.0417	8.35	13.50
	80	104	16.0	60.0	0.0626	12.55	26.05
	86	110	17.0	60.0	0.0766	15.40	41.45
	92	116	17.0	63.5	0.0714	14.30	55.75
	98	122	18.0	60.0	0.0810	16.20	71.95
	104	128	16.0	61.5	0.0534	10.70	82.65
	116	134	17.0	64.5	0.0349	7.00	89.65
116	140	17.0	59.0	0.0199	4.00	93.65	

(a) Constant Still Conditions:
Rotor Speed: 1000 \pm 50 RPM
Feed Passes Across Rotor Per Fraction: One
Constant Yield Oil Composition: 225 ml Volt Esso No. 36
175 ml Light Mineral Oil
425 ml Heavy Mineral Oil

(b) Refers to distance between condenser plate and evaporator gutter.

ROTOR SPEED: 1000 ± 50 RPM
FEED RATE: 60-65 ML/MIN
FEED-RESIDUE ΔT : 24°C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 12 ± 1 MICRONS
CONDENSER TEMPERATURE: $25 \pm 1^\circ\text{C}$
CONDENSER DISTANCE: $3/4$ IN

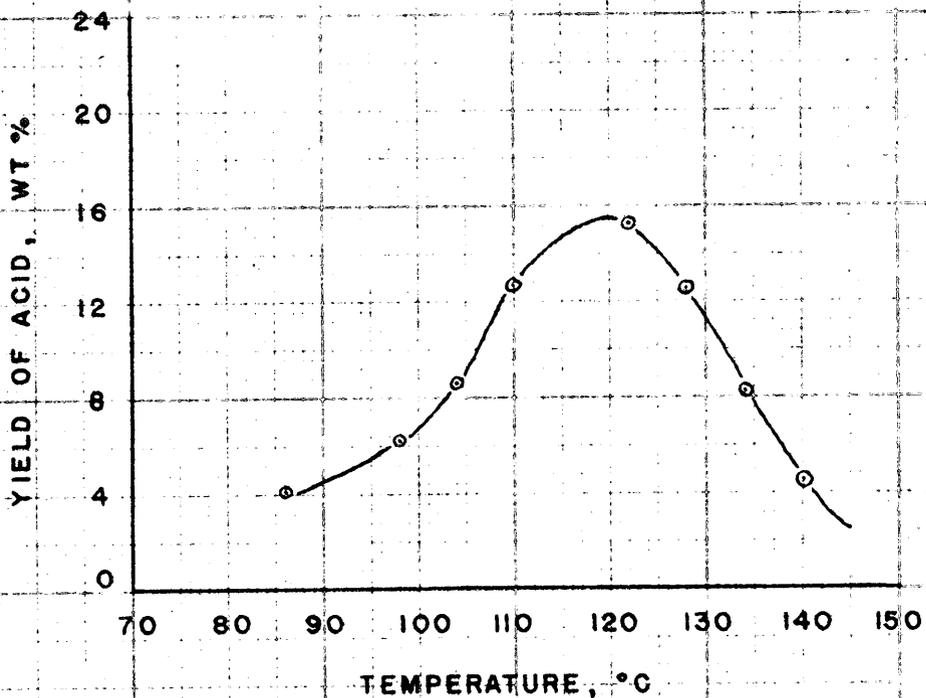


FIGURE 7. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID

ROTOR SPEED: 1000 ± 50 RPM
FEED RATE: 60-65 ML/MIN
FEED RESIDUE AT 24 °C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 15 ± 1 MICRONS
CONDENSER TEMPERATURE: 25 ± 1 °C
CONDENSER DISTANCE: 3/4 IN

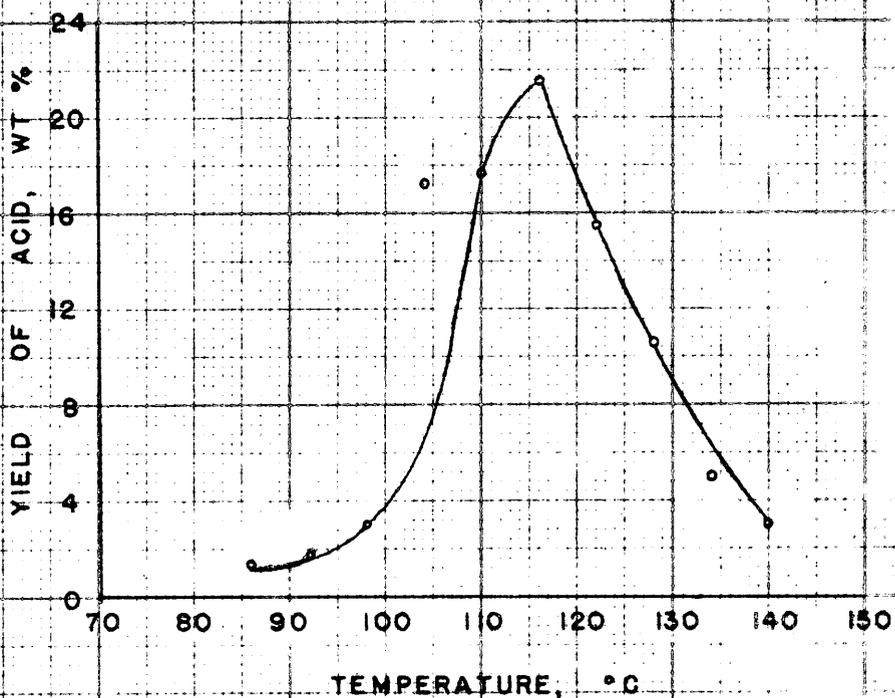


FIGURE 8. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID.

ROTOR SPEED: 1000 ± 50 RPM
FEED RATE: 60-65 ML/MIN
FEED-RESIDUE ΔT : 24°C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 14 ± 2 MICRONS
CONDENSER TEMPERATURE: $35 \pm 1^\circ\text{C}$
CONDENSER DISTANCE: $3/4$ IN

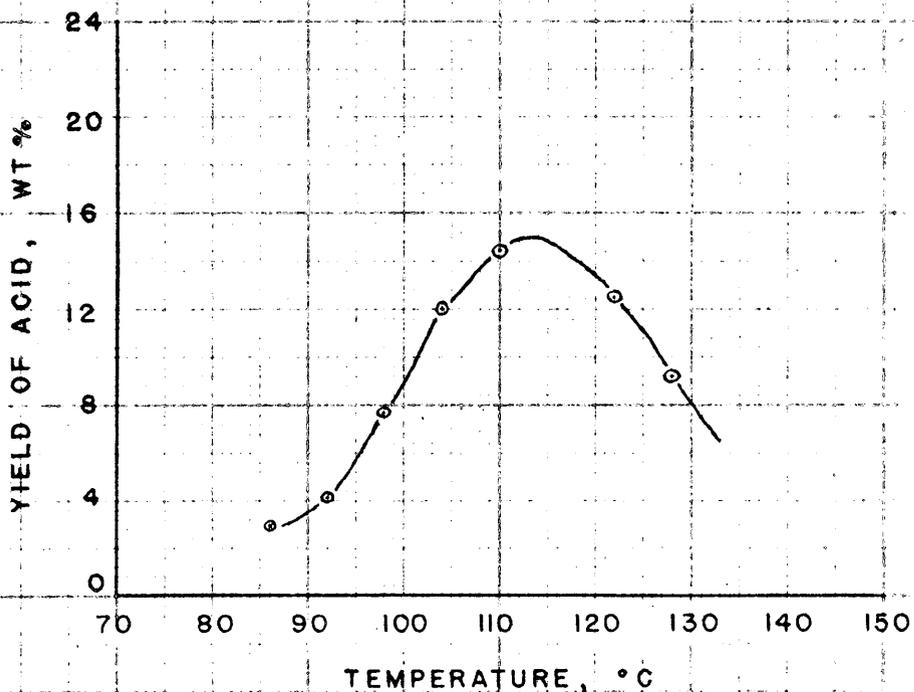


FIGURE 9. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID

ROTOR SPEED : 1000 ± 50 RPM
FEED RATE 60-65 ML / MIN
FEED - RESIDUE Δ T : 24 °C
NUMBER OF PASSES : ONE
OPERATING PRESSURE : 14 ± 1 MICRONS
CONDENSER TEMPERATURE : 45 ± 1 °C
CONDENSER DISTANCE : 3/4 IN

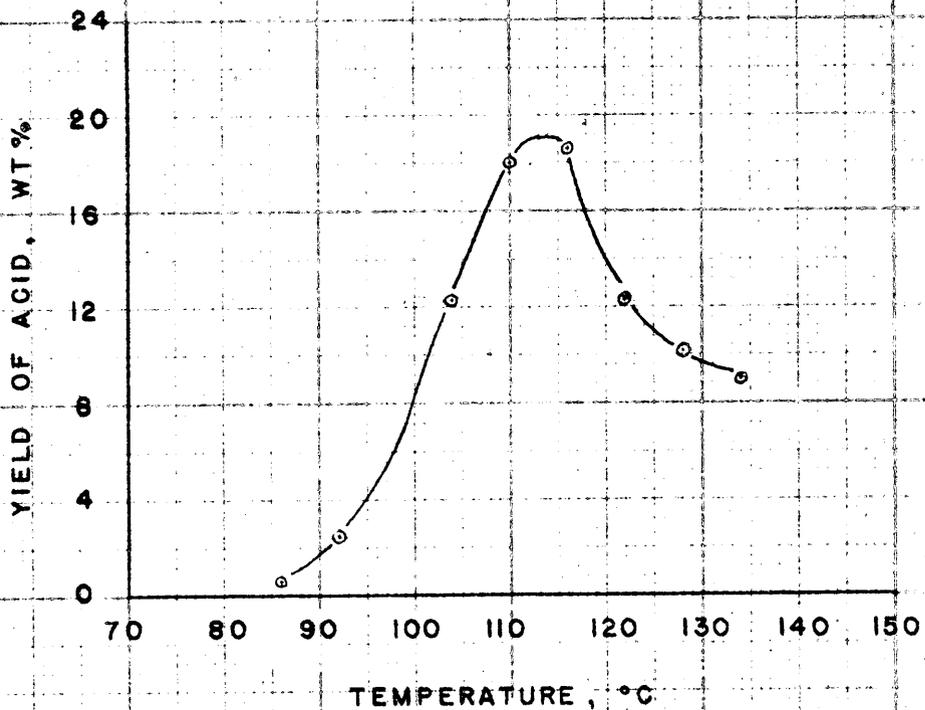


FIGURE 10. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID

ROTOR SPEED: 1000 ± 50 RPM
FEED RATE: 60 - 65 ML / MIN
FEED - RESIDUE ΔT : 24°C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 25 ± 2 MICRONS
CONDENSER TEMPERATURE: $25 \pm 1^\circ\text{C}$
CONDENSER DISTANCE: 2-1/2 IN

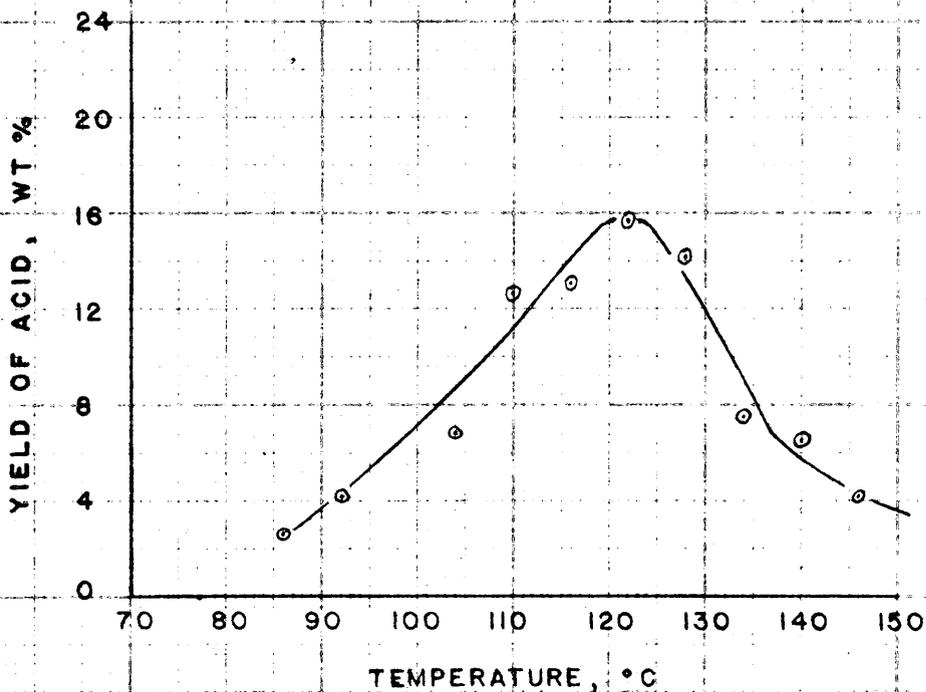


FIGURE 11. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID

ROTOR SPEED: 1000 ± 50 RPM
FEED RATE: 60 - 65 NL/MIN
FEED RESIDUE AT: 24 °C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 12 ± 1 MICRONS
CONDENSER TEMPERATURE: 25 ± 1 °C
CONDENSER DISTANCE: 2 - 1/2 IN

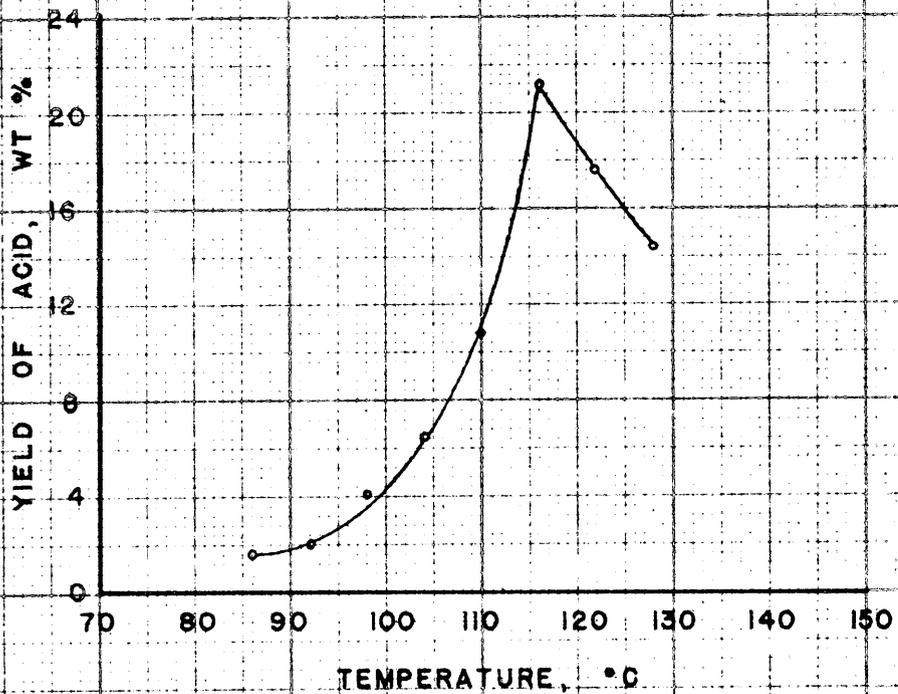


FIGURE 12. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID

ROTOR SPEED: 1000 ± 50 RPM
FEED RATE: 60-65 ML/MIN
FEED-RESIDUE ΔT : 24°C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 24 ± 1 MICRONS
CONDENSER TEMPERATURE: $35 \pm 1^\circ\text{C}$
CONDENSER DISTANCE: 2-1/2 IN

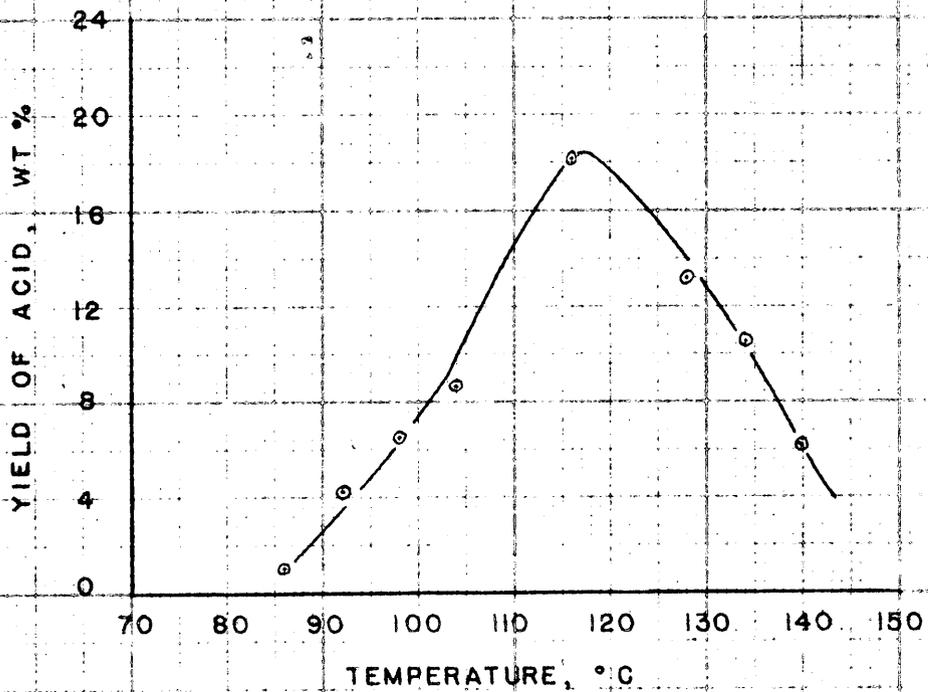


FIGURE 13. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID

ROTOR SPEED: 1000 ± 50 RPM
FEED RATE: 60-65 ML/MIN
FEED RESIDUE ΔT: 24°C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 15 ± 1 MICRONS
CONDENSER TEMPERATURE: 35 ± 1°C
CONDENSER DISTANCE: 2-1/2 IN.

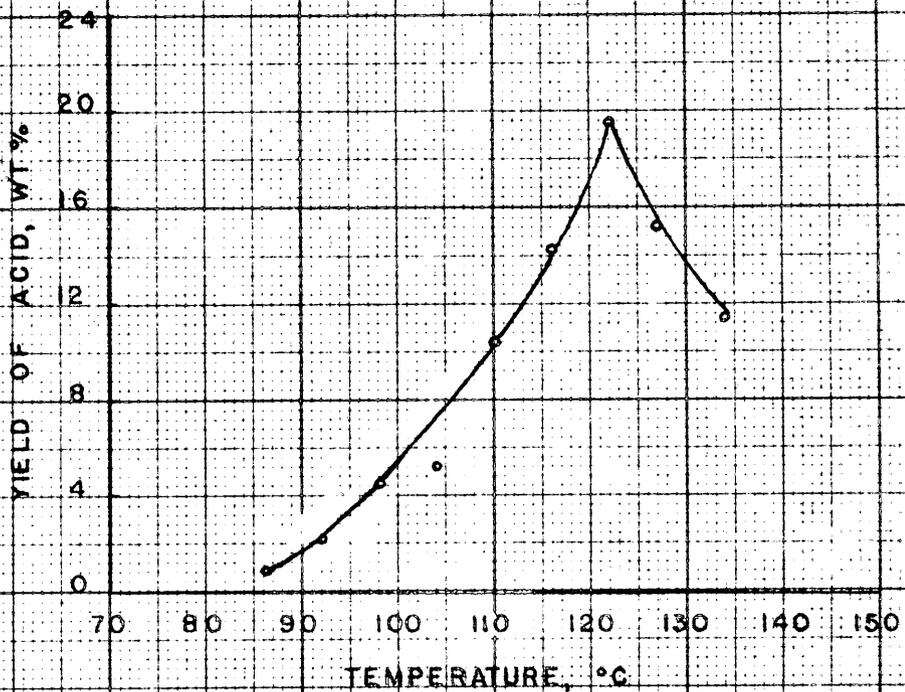


FIGURE 14. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID

ROTOR SPEED: 1000±50 RPM
FEED RATE: 60-65 ML/MIN
FEED-RESIDUE ΔT: 24°C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 7±2 MICRONS
CONDENSER TEMPERATURE: 45 ±1°C
CONDENSER DISTANCE: 2-1/2 IN

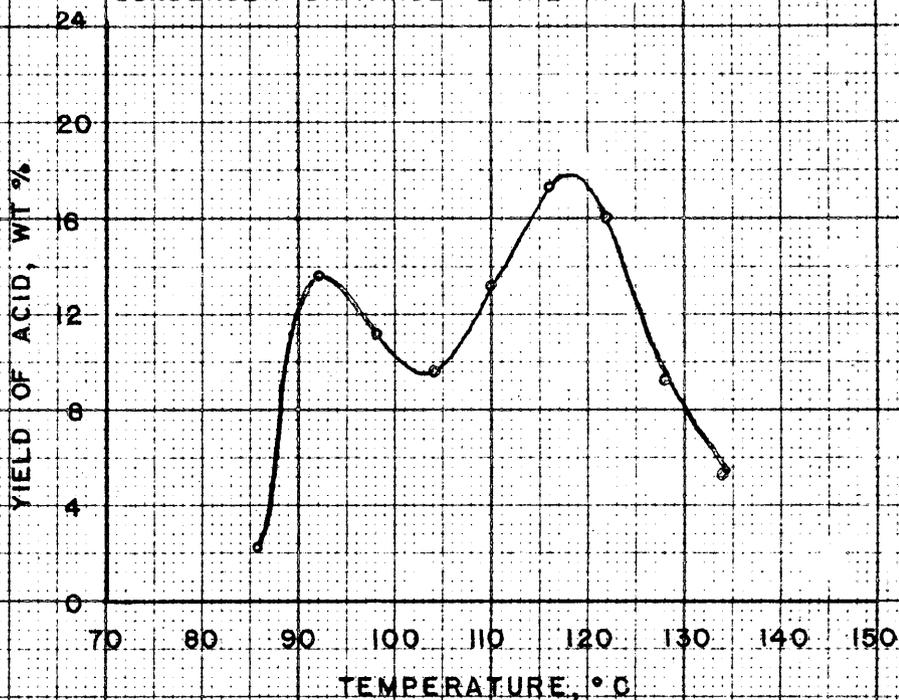


FIGURE 15. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID.

ROTOR SPEED: 1000 ± 50 RPM
FEED-RESIDUE AT: 24°C
FEED RATE: 60-65 ML/MIN
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 17 ± 2 MICRONS
CONDENSER TEMPERATURE: 45 ± 1°C
CONDENSER DISTANCE: 2-1/2 IN.

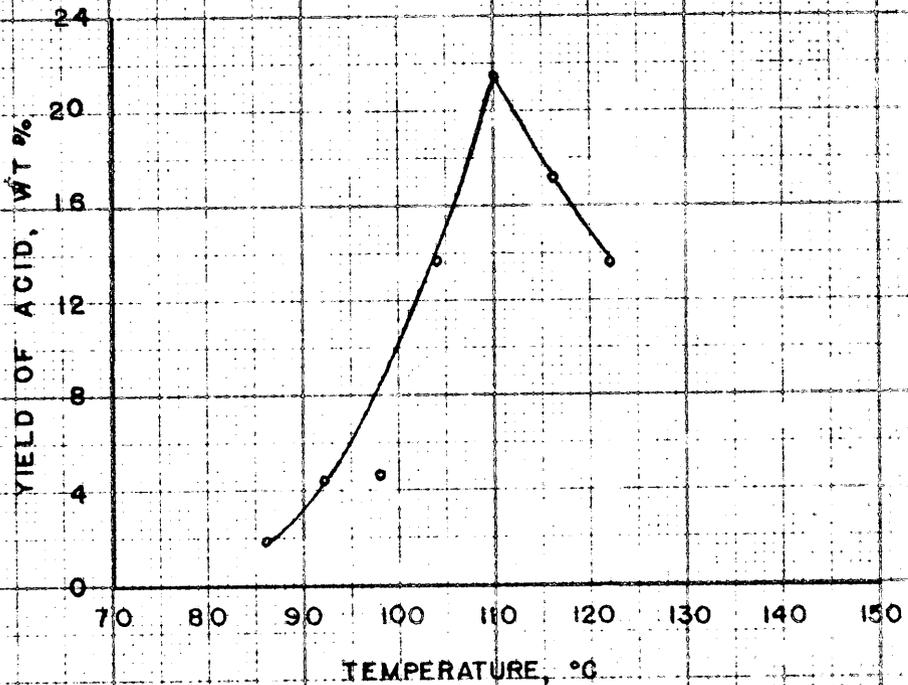


FIGURE 16 ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID

ROTOR SPEED: 1000±50 RPM
FEED RATE: 60-65 ML/MIN
FEED RESIDUE ΔT: 24 °C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 10±1 MICRONS
CONDENSER TEMPERATURE: 25 ±1 °C
CONDENSER DISTANCE: 5-3/8 IN

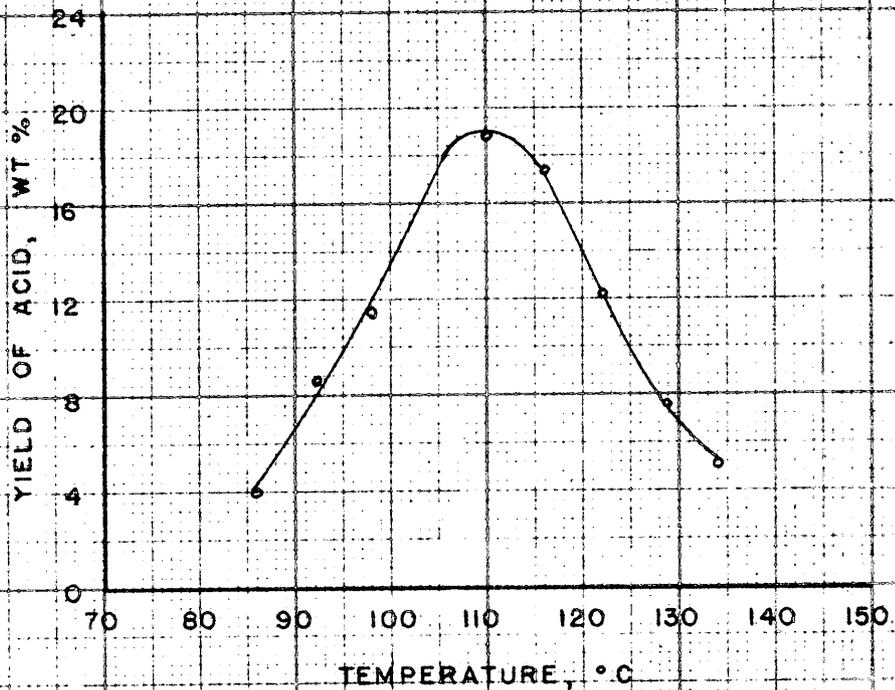


FIGURE 17. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID.

ROTOR SPEED: 1000 ± 50 RPM
FEED RATE: 60-65 ML/MIN
FEED RESIDUE ΔT: 24 °C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 13 ± 1 MICRONS
CONDENSER TEMPERATURE: 35 ± 1 °C
CONDENSER DISTANCE: 5-3/8 IN

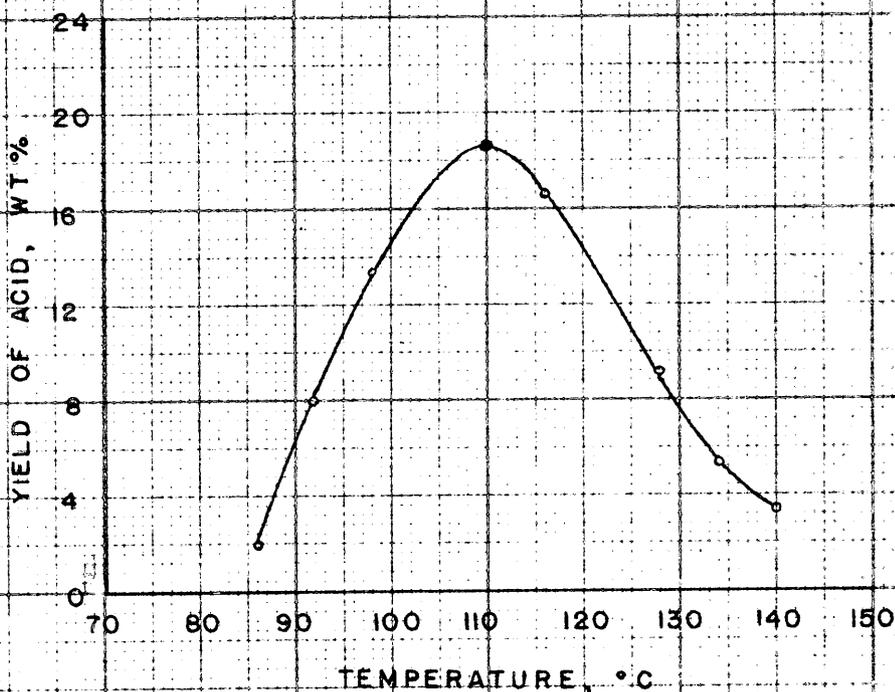


FIGURE 18. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID.

ROTOR SPEED: 1000 ± 50 RPM
FEED RATE: 60 - 65 ML/MIN
FEED-RESIDUE ΔT: 24°C
NUMBER OF PASSES: ONE
OPERATING PRESSURE: 17 ± 2 MICRONS
CONDENSER TEMPERATURE: 45 ± 1°C
CONDENSER DISTANCE: 5 - 3/8 IN

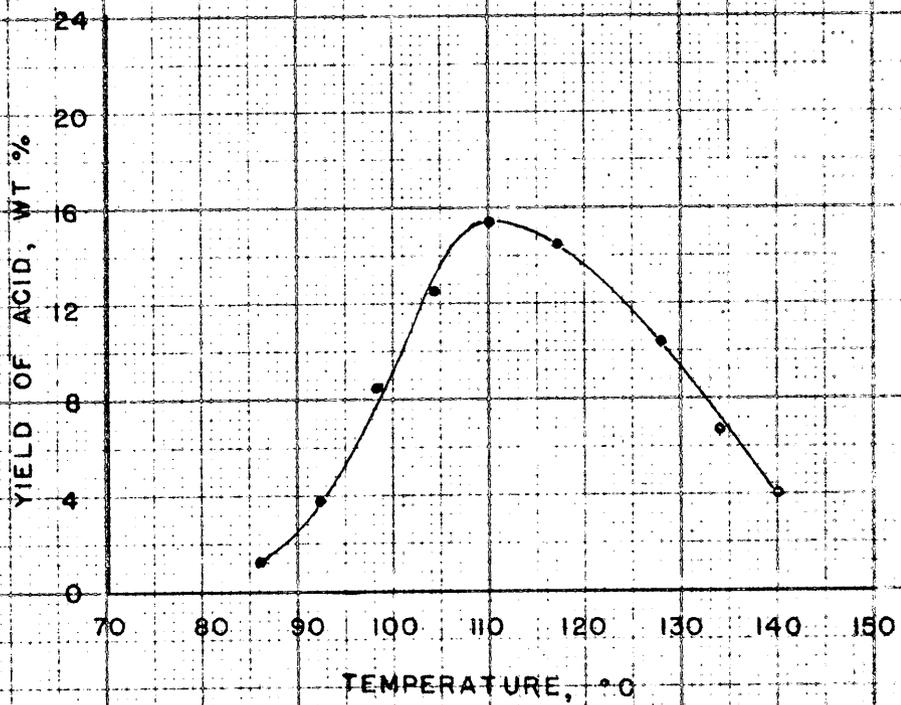


FIGURE 19. ELIMINATION CURVE FOR THE MOLECULAR DISTILLATION OF STEARIC ACID.

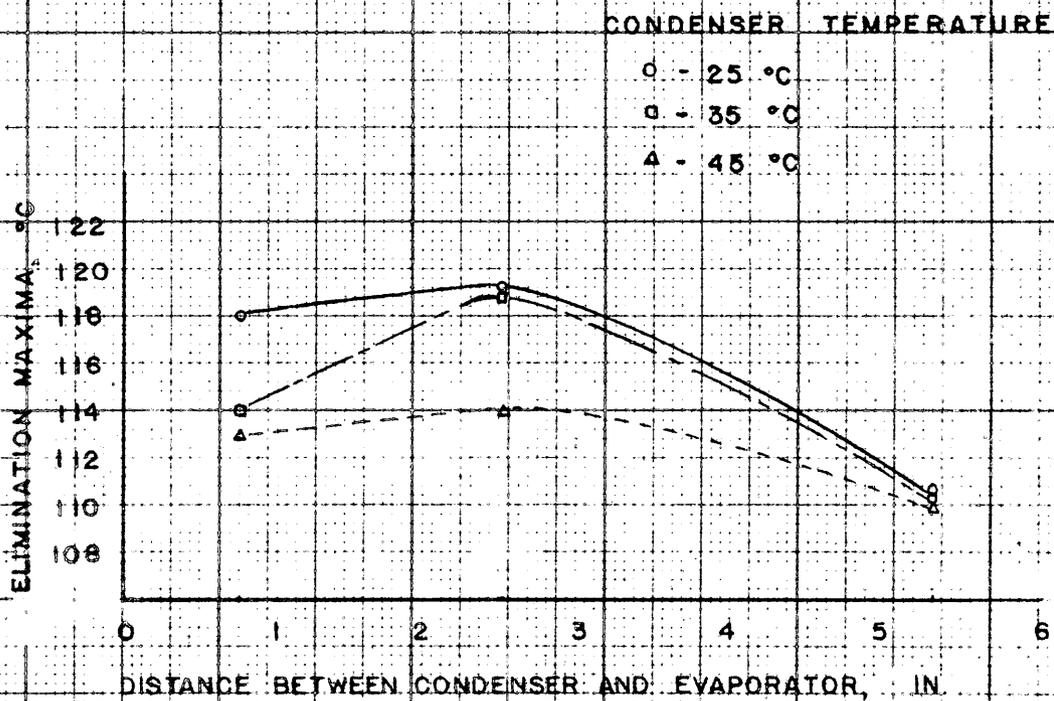


FIGURE 20. EFFECT OF CONDENSER TEMPERATURE AND LOCATION ON THE ELIMINATION MAXIMA OF STEARIC ACID.

IV. DISCUSSION

The purpose of this investigation was to study the effect of condenser temperature and condenser location on the molecular distillation characteristics of stearic acid. The following sections present a discussion of the results obtained in this investigation, the limitations imposed on the experimental conditions employed in this study, and recommendations for future molecular distillation studies.

Discussion of Results

A discussion of the results obtained from this investigation are reviewed in the following sections entitled preliminary tests, modification of the still, experimental conditions and procedure, combined effect of condenser temperature and location on the elimination maximum, errors, and statistical analysis.

Preliminary Tests. During the initial phase of this investigation several preliminary tests were made to acquaint the operators with the equipment and to familiarize them with the procedure and techniques employed during the investigation. It was reasoned at that time that the results of the preliminary tests would be even more

beneficial if these tests were conducted under a set of conditions identical to those conditions utilized by previous investigators⁽¹¹⁾. By comparing the results of the preliminary tests with those cited in the literature, obvious errors could be detected and necessary corrections could be made prior to performing the tests in the experimental part of this investigation. The experimental conditions employed in an investigation conducted by Mote and Demakis^(27,12) were selected for the preliminary tests, because the elimination maximum for stearic acid (the test medium employed in this investigation) had been determined by them using the same molecular still apparatus selected for this study. These experimental conditions are presented in the limitations section of this report.

The results of the preliminary tests using stearic acid compared favorably with results found in the literature, yielding an average elimination maximum of 114 degrees centigrade as compared with 112 degrees centigrade, respectively. The average elimination maximum in both cases was obtained by averaging the results of two tests. The difference of two degrees centigrade between the two elimination maxima was attributed to experimental error believed to be brought about by the difference in technique employed by the operators of the two investigations.

The preliminary tests not only served to indicate whether or not the operators of this investigation could obtain acceptable results from the procedure prescribed by Mote and Demakis but also served as a means for comparing the results of this investigation with those of the literature.

Modification of the Still. During the conduct of the preliminary tests, difficulty was frequently encountered while trying to maintain a constant feed flow rate and feed temperature. This difficulty was partially attributed to the original location of the feed valve in the feed line. Reference is made to Drawing 1, page 32. The feed rate had to be adjusted by manipulating the feed valve according to the judgment of the operator which in turn was based on visual observation of the feed as it issued from the feed spout and passed across the rotor. The feed valve was located by Mote and Demakis in a position on the feed line near the rotor in order to facilitate the ease of adjusting feed flow rates while observing the rotor simultaneously.

When the feed valve was located in this position in the feed line, each change in the temperature of the feed, resulted in a fluctuation in the flow rate of the feed. These fluctuations over a period of nine to twelve minutes (average time required for the feed of each fraction to completely pass over the rotor) seriously affected the flow

rate of the feed to such an extent that fractions had to be repeated as often as six to eight times before a flow rate of 60 to 65 milliliters per minute could be obtained.

By relocating the feed valve in a position on the feed line prior to the feed preheating coils, and elevating the feed tank approximately eighteen inches for additional head, it was reasoned that a more constant feed rate could be obtained because (1) changes in the temperature of the preheater coil would no longer affect the flow rate of the feed, and (2) the feed would enter the feed valve at a constant temperature from the feed tank which was maintained at a relatively constant temperature.

After the valve was relocated to a new position in the feed line, an improvement was noted with respect to maintaining a constant feed flow rate. Despite the improvement derived from this change, however, flow rate difficulties were never completely eliminated.

Experimental Conditions and Procedure. In a previous work of Mote and Demakie^(27,12) considerable effort was devoted to determining a set of experimental conditions and a procedure suitable for studying the effect of molecular structures of various fatty acids. The experimental conditions and procedure selected for use throughout the conduct of this investigation were identical to those

employed by these two investigators because of several reasons, (1) the conditions and procedure were developed specifically for the same still that was used in this investigation, (2) the conditions and procedure were developed specifically for distilling various fatty acids including stearic acid, the test medium selected for this investigation, and (3) these conditions and procedure proved to be acceptable. The procedure and conditions are discussed more fully in the procedure and limitations section of this report.

Variables Studied. By studying the molecular distillation characteristics of stearic acid when varying the temperature and location of the condenser it was possible to determine a relationship between the condenser temperature and location that could be used in future operations of centrifugal molecular stills. The reasons for selecting these two variables, the methods used in accomplishing this study, and the results obtained from this investigation are discussed in the following sections entitled condenser location and condenser temperature.

Condenser Location. Numerous findings⁽¹⁵⁾ have indicated that the distance between the evaporator and the condenser is not as critical as formerly believed, as far as mean free-path design criteria are concerned. It was recommended by Mote⁽²⁵⁾ that

studies be made on the present molecular still at Virginia Polytechnic Institute to determine exactly how critical this stipulation is. To accomplish such a study, Mote further recommended that a series of distillation tests be conducted with the condenser located at six different positions inside of the bell jar of the molecular still, such that the distance between the evaporator and the condenser would be at some multiple of the mean free-path distance, namely $1/2$, 1, $1-1/2$, 2, $2-1/2$, and 3. For such a study, stearic acid was suggested to be an excellent compound for the test medium, since its elimination maximum occurs approximately mid-range of the constant-yield oil used in this investigation.

Because of the limitations of time imposed on the investigation, three rather than six condenser locations were chosen for this study. Distances between the evaporator and the condenser of $3/4$, $2-1/2$, and $5-3/8$ inches were selected for this investigation because: the $3/4$ inch position was the nearest possible position that the condenser could be located from the evaporator, the $5-3/8$ inch position was the most extreme possible location of the condenser from the evaporator when a

sixteen inch bell jar was used, and the 2-1/2 inch position was used in previous molecular distillation studies by other investigators, thus by selecting this position the results of this investigation could be compared with the results of previous investigations. The mean free-path distance calculated for the molecular still in this investigation was approximately 2.2 centimeters. The distances of 3/4, 2-1/2, and 5-3/8 inches correspond to 0.8, 2.9, and 6.2 multiples of the mean free-path, respectively. The selection of these condenser locations was reasoned to be sufficient for determining the effect of mean free-path distance on the molecular distillation characteristics of stearic acid.

The copper condenser was constructed from an 1/8-inch copper plate, 8 inches in diameter. To the back (opposite side from the rotor) of the condenser plate, six concentric spirals of 1/4 inch copper tubing were soft-soldered. The inlet and outlet water lines which passed through the condenser plate to the base plate of the still served as the support for the condenser. By adjusting the length of the inlet and outlet cooling water lines the position of the condenser could be varied. This adjustment was accomplished by inserting the

appropriate length of $1/4$ inch galvanized pipe between the base plate and the copper cooling water lines of the condenser. This method for supporting the condenser proved to be acceptable even for the extreme condenser position studied in this investigation.

An approximate estimation of the feed rate could be obtained by the operator through visual observation of the feed as it passed over the rotor. When the condenser was located in a position $3/4$ inch from the evaporator, visual observation of the rotor was not possible, thus estimates of the feed rate could no longer be obtained by this method. To circumvent this obstacle, a $1/2$ inch hole was drilled through the center of the condenser plate and a two-inch length of $1/2$ inch copper tubing was inserted in the hole and inclined at an angle of approximately 80 degrees to a plane parallel to the condenser surface; thus the operator was allowed to view the rotor and thereby be permitted to estimate the feed rate. The two-inch copper tubing was inserted in the hole of the condenser plate and inclined at an angle to furnish additional surface area in order to condense as much as possible any vapors that tended

to pass through the hole in the condenser plate. It was reasoned that this modification of the condenser plate had a negligible effect on the final results obtained in this investigation; because the surface area of the condenser plate was reduced less than 1.5 per cent, and by inserting the two-inch copper tube in the hole of the condenser plate the reduction in the available area of the condenser was considered to be even less than this value.

The distance between the condenser and the evaporator was found to have very little effect on the elimination maximum temperature of stearic acid when the condenser temperature was maintained at 25, 35, and 45 degrees centigrade and the distance between the condenser and the evaporator did not exceed a mean free-path value by a multiple of three. The greatest effect of condenser location on the elimination maximum of stearic acid was noted to take place when the distance between the condenser and the evaporator exceeded a mean free-path multiple of three. It was observed, however slight as it may be, that for each condenser temperature studied that as the distance between the condenser and the evaporator increased the elimination maximum of stearic acid gradually shifted to a higher value and reached a

maximum when the condenser was located at a position with a mean free-path multiple of three. When the condenser was located past this position, the elimination maximum decreased sharply to a lower value. This phenomenon is believed to be caused by one or two reasons, namely: (1) as the distance between the condenser and the evaporator was increased, an increased amount of bell jar surface was exposed for the vapors to condense upon, and because there was a temperature difference between the bell jar surface and the copper condenser surface, varying amounts of distillate were condensed, and (2) when the distance between the condenser and the evaporator exceeded a mean free-path multiple of three, for large oil molecules, the effect of the distance between the condenser and the evaporator then has a greater effect on the elimination maximum.

Condenser Temperature. From a series of tests made by Mote²⁶) it was observed by him that at lower condenser temperatures a considerably larger volume of constant-yield oil was obtained in the lower fractions than was obtained with the higher condenser temperatures. From these results it was believed

that the temperature of the condensing surface had a decided effect on the elimination maximum. It was recommended that a series of distillation tests be made using a standardized set of operating conditions, and varying the condenser water temperature to determine what effect the variation of condenser water temperature had on the elimination maximum.

Based on the above recommendation, this investigation was made to determine the effect of condenser temperature on the elimination maximum using stearic acid as the test medium. The condenser water temperatures selected for this study were 25, 35, and 45 degrees centigrade. The 45-degree centigrade condenser water temperature level was selected because previous molecular distillation studies had been conducted at this temperature, thus by selecting this temperature the results of this investigation could be compared with the results of other investigations. The 25- and 35-degree centigrade condenser water temperature levels were arbitrarily chosen, but it was reasoned that if the condenser water temperature had any appreciable effect on the elimination maximum this effect could be detected if the condenser water temperature levels had a differential of ten degrees centigrade.

Little difficulty was encountered in maintaining the condenser water temperature constant (i.e. within ± 1 degree centigrade). By varying the powerstat on the control panel condenser water temperature levels of 35 and 45 degrees centigrade were easily obtained. In order to obtain a condenser water temperature of 25 degrees centigrade, however, a slight modification of the equipment was necessary. This was accomplished by inserting in the cooling water return line one twelve-inch long double-tube, single-pass, counter-current heat exchanger constructed of 1/4-inch and 1-inch copper tubing. By passing tap water at approximately 15 degrees centigrade through the annulus of the heat exchanger the condenser water temperature was easily maintained at 25 degrees centigrade.

The terms condenser temperature and condenser water temperature are used interchangeably from one literature source to another to mean the same thing. In the true sense of the word, this is not correct. Although the actual difference between condenser surface temperature and the condenser water temperature was not determined in this investigation, it is believed that a negligible temperature differential

exists between the two, because (1) the cooling water temperature did not increase more than one degree centigrade after completely passing through the condenser coils of the condenser plate, and (2) the temperature of the cooling water had sufficient control over the condenser surface temperature to cause condensation or solidification of certain fatty acids during distillation.

The elimination maximum of stearic acid was increased approximately five degrees centigrade when the temperature of the condenser was decreased from 45 to 25 degrees centigrade when the condenser was located at positions less than 2-1/2 inches from the evaporator, and condenser temperatures between 25 and 45 degrees centigrade had no effect on the elimination maximum when the distance between the condenser and evaporator was 5-3/8 inches.

For the three condenser locations studied in this investigation, it was found that the condenser temperature (between 25 and 45° C) had no effect on the amount of constant-yield oil collected per fraction.

Combined effect of Condenser Temperature and Location

on the Elimination Maximum. The elimination maximum of stearic acid was increased from 113 to 118 degrees centigrade

when the temperature of the condenser was decreased from 45 to 25 degrees centigrade when the condenser was located in a position $3/4$ inch from the evaporator. When the condenser was located in a position $2-1/2$ inches from the evaporator, the elimination maximum was increased from 114 to 119 degrees centigrade when the condenser temperature was decreased from 45 to 25 degrees centigrade. From this data it was learned that the elimination maximum of stearic acid was increased approximately 1 degree centigrade for every 4 degrees centigrade decrease in condenser temperature when the condenser was located in positions less than $2-1/2$ inches from the evaporator. In addition it was observed that when the condenser temperature was held constant (either the 25, 35, or 45-degree centigrade level) the elimination maximum was not affected when the condenser was moved from $3/4$ inch to $2-1/2$ inches from the evaporator. It was reasoned that the temperature of the condenser should have some effect on the elimination maximum; however, it was not understood why a decrease in condenser temperature increased rather than decreased the elimination maximum. When the condenser was located at a distance of $5-3/8$ inches from the evaporator, the elimination maxima was 110 degrees centigrade for all condenser temperatures studied. From this data, it was obvious that the temperature

of the condenser had no effect on the elimination maximum when the condenser was located in a position with a mean free-path multiple of approximately 6. From this investigation, it was concluded that the location of the condenser had no effect on the elimination maximum when the condenser location did not exceed a mean free-path multiple of 3. It is noted at this point that this conclusion substantiates previous findings and concurs with the mean free-path theory presented in the literature.

Errors. When stearic acid was distilled in one of the tests in this investigation with the condenser temperature at 45 degrees centigrade and the condenser located at a position of 2-1/2 inches from the evaporator, a distillation curve was obtained with two maxima. Reference is made to Figure 15, page 53. The lowest maximum was believed to be caused by impurities in the feed mixture used. It was never actually determined how the impurity got in the feed mixture, but precautions were taken to avoid any contamination of the feed mixture in the tests that followed, and as a result the distillation curves obtained had only one maximum value in the tests that followed.

Duplicate tests were made on four of the nine conditions studied in this investigation. It was found that the distillation curves of stearic acid could be reproduced within \pm three degrees centigrade.

In several of the tests, difficulty was encountered in an attempt to obtain a satisfactory feed flow rate (60 to 65 milliliters per minute). From an investigation to seek the cause of this trouble, it was learned that the feed line had been contaminated with some foreign material believed to be bits of packing from the feed pump. This condition was corrected by dismantling the feed line and then cleaning it with a light grade mineral oil. When the flow rate was less than 60 milliliters per minute for any fraction, a greater yield of acid was obtained; and conversely, when the flow rate was greater than 65 milliliters per minute, a lower yield of acid was obtained as seen in Figures 8, 11, 14, 16, pages 46, 49, 52, and 54. It was reasoned that if the feed flow rate was unsatisfactory for two or less fractions in any one test that the elimination maximum was not affected.

In some of the tests, the temperature of the feed, the temperature of the residue, the temperature differential between the feed and residue, or the feed flow rate for some fractions were not within limits. If one or more of these conditions were not within limits for any fraction, the data were rejected and not included when the elimination curve was drawn.

Statistical Analysis. In a statistical analysis of the data obtained in this investigation, it was found that the variable of condenser temperature was not significant and that the variable condenser distance was significant at a significance level of five per cent. This data was analyzed using a two-way classification with unequal replication.

Recommendations

On the basis of the knowledge gained during the conduct of this investigation, the following recommendations are offered for the improvement of the molecular still assembly and for the advancement of molecular distillation studies.

Molecular Still Modification. To eliminate some of the difficulties encountered during the operation of the molecular still and to improve the equipment for obtaining a better reproduction of the data, the following recommendations are considered.

Instrumentation. Throughout the conduct of this investigation considerable difficulty was encountered with respect to maintaining constant feed and residue temperatures. It was frequently necessary to repeat as often as four or five times some of the distillation fractions in order to obtain the desired temperatures that were required. This difficulty was believed to be caused by the lag of time that existed between the time that the powerstat was adjusted and the time that this adjustment caused a change in the temperature of the feed or residue. For example, if the temperature of the

residue, which was determined by reading the dial on the potentiometer, was two degrees low, the powerstat setting was increased approximately ten divisions; and approximately one to two minutes was required before the residue assumed the desired temperature level. In order to maintain a constant temperature, it was necessary to make frequent potentiometer readings to detect very slight changes in the temperature of the residue, thus making it possible to predict even larger temperature fluctuations that might follow. When these slight changes in residue temperature were detected, the powerstat was immediately adjusted to correct the anticipated larger temperature fluctuation that may have followed. This procedure was used to compensate for the lag time. Frequently this procedure failed, and as a result the distillation fraction had to be repeated.

It is recommended that an automatic temperature control instrument be installed for controlling the temperature of the feed and residue. This installation would not only improve the ease of operation but would also insure a better reproduction of the data obtained.

Filter. In several of the tests difficulty was met in an attempt to obtain a satisfactory feed flow rate (60 to 65 milliliters per minute). From an investigation to seek the cause of this trouble, it was learned that the feed line had been contaminated with some foreign material. It would be advantageous to have some means for correcting this condition. It is recommended that a small filter be placed in the feed return line between the feed pump and the feed tank.

Future Molecular Distillation Studies. To supplement the data and further verify the findings of this investigation, it is recommended that another investigation be made similar to this investigation. For such a study it would be beneficial to study the effect of condenser locations of 1-1/2, 3, and 4-1/2 inches from the evaporator, using margaric acid as the test medium.

Limitations

A centrifugal molecular still with a five-inch rotor and provided with a magnetic drive was used to study the effect of condenser temperature and location on the molecular distillation characteristics of stearic acid. The limitations imposed on this investigation include those of the acid used, operating pressure, feed rate, rotor speed, temperature differential between feed and residue, condenser water temperatures, condenser locations, number of passes of the feed over the rotor per fraction, mineral constant-yield oil, analysis of distillate samples, and experimental procedure.

Acid Used. Stearic acid was employed for all tests in this investigation. The weight of acid used for each test varied from 0.4500 to 0.5500 gram.

Operating Pressure. The tests were conducted at an operating pressure varying from 9 to 23 microns.

Feed Rate. A feed rate varying from 60 to 65 milliliters per minute was used for the distillation of all fractions. Feed rates were determined by timing a known volume to completely pass over the rotor.

Rotor Speed. A rotor speed of $1,000 \pm 50$ revolutions per minute was used in all the tests.

Feed-Residue Temperature Differential. A temperature differential of $24 \pm 1/2$ degrees centigrade between feed and residue was maintained for all fractions collected in the tests.

Condenser Water Temperature. The condenser cooling water temperatures studied in this investigation were 25 ± 1 , 35 ± 1 , and 45 ± 1 degrees centigrade. The flow rate of the water passing through the condenser was such that the temperature was not raised more than one degree.

Condenser Locations. The condenser was located at distances of $3/4$, $2-1/2$, and $5-3/8$ inches from the evaporator residue gutter in planes perpendicular to the normal of the evaporator.

Number of Feed Passes. For all distillate fractions collected in this test, the feed was allowed to pass over the rotor once. The time required for one pass of the feed over the rotor varied from 13.3 minutes for the 86-degree centigrade fraction to 7.6 minutes for the 140-degree centigrade fraction.

Constant-Yield Oil. The constant-yield oil employed in this investigation was composed of 225 milliliters

of No. 36 Voltesso, 175 milliliters of light-grade mineral oil, and 425 milliliters of heavy-grade mineral oil.

Analysis of Distillate Samples. The distillate fractions were analyzed by extracting the fatty acid with ethyl alcohol and titrating with a potassium hydroxide solution to the phenolphthalein end point.

Experimental Procedure. The experimental procedure employed throughout this investigation was a procedure developed by Mote and Demakis⁽¹¹⁾.

V. CONCLUSIONS

The effect of condenser temperature and location on the molecular distillation characteristics of stearic acid was determined using the following operating conditions with a centrifugal molecular still: operating pressure, 24 ± 2 microns of mercury, absolute; feed rate, 60 to 65 milliliters per minute; rotor speed, 1000 ± 50 revolutions per minute; feed-residue temperature differential, $24 \pm$ degrees centigrade; condenser water temperatures, 25-, 35-, 45-degree centigrade levels; condenser locations, $3/4$, $2-1/2$, and $5-3/8$ inches from the evaporator; and number of passes of the feed over the rotor per fraction, one. From the investigation the following conclusions may be drawn:

1. The location of the condenser has no effect on the elimination maximum of stearic acid when the condenser location does not exceed a mean free-path multiple of three, for condenser temperatures of 25, 35, and 45 degrees centigrade.

2. The elimination maximum of stearic acid is increased approximately one degree centigrade for every four degrees centigrade decrease in condenser temperature,

when the condenser is located in positions with a mean free-path multiple less than three.

3. The elimination maximum of stearic acid is not affected by condenser temperature when the condenser is located in a position with a mean free-path multiple of six.

4. The elimination maximum of stearic acid is decreased when the condenser is located in positions with a mean free-path multiple exceeding three.

VI. SUMMARY

Numerous findings have indicated that the distance between the evaporator and condenser of the molecular still is not as critical, as far as mean free-path design criteria are concerned, as formerly believed. It had been recommended that studies be made on the present apparatus at Virginia Polytechnic Institute to determine exactly how critical this factor is.

The purpose of this investigation was to study the effect of condenser temperature and location on the molecular distillation characteristics of stearic acid, and from this study, determine this relationship of condenser temperature and location to be used in centrifugal molecular still operation.

This investigation was accomplished using a magnetically-driven centrifugal molecular still with a five-inch rotor and employing the following operating conditions: operating pressure, 24 ± 2 microns of mercury, absolute; feed rate, 60 to 65 milliliters per minute; rotor speed, 1000 ± 50 revolutions per minute; feed-residue temperature differential, 24 ± 1 degrees centigrade; condenser water temperature, 25, 35, and 45 degrees centigrade; condenser locations,

3/4, 2-1/2, and 5-3/8 inches from the evaporator; and number of passes of the feed over the rotor per fraction, one.

It was concluded from this investigation that if the location of the condenser did not exceed a mean free-path multiple of three, that the location of the condenser had no effect on the elimination maximum of stearic acid, and the elimination maximum was increased approximately one degree centigrade for every four degrees centigrade decrease in condenser temperature. The elimination maximum was not affected by condenser temperature when the condenser was located in a position with a mean free-path multiple of six.

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