THE EFFECT OF THE VARIATION OF FEED AND EXTRACT REFLUX INPUT POSITIONS ON THE OPERATING CHARACTERISTICS OF A LIQUID-LIQUID EXTRACTOR

by

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TABLE OF CONTENTS

INDEX OF DRAWINGS

INDEX OF FIGURES

INDEX OF TABLES

1. Introduction

The unit operations, distillation, extraction and gas absorption constitute the analogous group of diffusion processes in chemical engineering. For many years the analogy among these operations has been recognized, and within the past thirty years, the object of a great deal of research has been to show the existing correlation among these operations. The collective result of this research effort has been the demonstration that the mechanisms of all diffusion processes are similar, if not identical, except as to the method by which they are carried out.

Of these diffusion processes, liquid-liquid extraction offers perhaps the greatest potentialities for application to separation problems. Largely due to lack of understanding in the past, utilization of the extraction operation has been almost wholly confined to separation problems to which other methods are not applicable, and obvious advantages of simplicity in operation and greater thermodynamic efficiency have been overlooked in favor of better understood methods. Also, largely due to this same lack of information, until recently, methods of calculation and scientifically designed equipment have not been generally available. Now, however, largely through a more intensive study of mass transfer rates, simplified methods of

calculation have been developed, while correlation of existing experimental data with theoretical considerations has resulted in the development of specialized equipment, scientifically designed to perform a given separation under optimum controlled conditions. This logical sequence has had several effects. Certain physical concepts of differences between washing, leaching and extraction are now clearly defined and generally understood in the chemical engineering field. The correlation of theoretical considerations with experimental data has resulted in a wide understanding of natural laws governing extraction operations. Finally, suitable nomenclature and classifications of processes have been developed. The present tendency is toward the investigation of new systems with the idea of extending the application of extraction to other separation problems in the chemical industry.

The purpose of this investigation was to determine the effect of the variation of extract reflux and feed input positions on the operating characteristics of a spiral-channel liquid-liquid extractor. The system investigated was methylcyclohexane - aniline - n-heptane, employing aniline as the selective solvent at 20°C and atmospheric pressure.

II. LITERATURE REVIEW

A. General Considerations

Definitions. For clarity, at this point it is desirable that certain terminology peculiar to the unit operation involved be defined. The definitions presented here are those generally accepted and prevalent in the present literature [24,37].

Solvent Extraction. Solvent extraction as defined by Sherwood [37], "refers to the separation of the components of a liquid with a immiscible solvent in which one or more of the components of the solution are soluble." To this definition, it might be well to add the qualifying statement that the solvent employed need not be entirely immiscible with any of the components of the system. In actual practice, complete immiscibility is never encountered in a liquid-liquid system. However, since the feasibility of the extraction operation relies largely on the law of partition, it is desirable that the relative solubility of one of the components of the liquid system be considerably greater than that of any of the other components. Elgin [24] has offered the following definition: "Liquid-liquid extraction consists in separating the

components of a liquid solution, or mixture, by treatment with an immiscible solvent which dissolves one or more of the components of the solution in preference to the others."

Solvent. The solvent is that liquid which is added to the feed and in which one or more of the components of the mixture is more soluble than the others. The solvent is said to be selective for that component of the feed which is found in greater ratio to the other in the extract product than in either the raffinate product or the original feed.

Solute. The solute is that portion of the feed for which the solvent is selective.

Carrier Solvent. The carrier solvent is that portion of the feed which is relatively immiscible with the solvent and is found in greater ratio in the raffinate layer than in either the original feed or in the extract product.

Extract. The extract is that residual material remaining from the solvent layer following the removal of the solvent. It consists mainly of the solute together with a small portion of the carrier solvent.

Raffinate. The raffinate is the residual material following the removal of the solute and consists largely of the carrier solvent together with small amounts of solute and solvent.

Principles of Extraction. (10,23) Liquid-liquid extraction has its basis in the fact that under certain conditions of concentration of the liquid components and under varying temperature conditions, certain liquids are soluble in varying degrees in other liquids. If one of the components is present in an amount greater than its solubility in a mixture of the other components present, under the existing temperature conditions, there will be a separation of the liquid system into two phases such that the components of each coexisting phase will be in equilibrium within itself. The components present will be distributed between these coexisting liquid phases, according to the amount and the relative solubility of each component in the phases present. Consequently, by performing the following operations in order, a separation of the components may be effected. These operations are:

1. Intimate mixing or contacting of the two immiscible phases to provide for the maximum rate of mass transfer.
2. Settling and separation of the two phases.
3. Removing the solvent from the separated phases.

Importance of Extraction. In demonstrating the industrial importance of the extraction operation, it may be well to mention a few of the instances in which extraction as a separation operation is superior to other better understood methods. Sherwood (37) mentions the fact that in many cases distillation is more effective and cheaper than extraction in separating liquid mixtures, however, under certain conditions, extraction may be far superior to other methods of separation. Elgin (13) cites the following instances where extraction is superior as a method of separation:

1. When the boiling points of the components of the liquid mixture are within a very few degrees of each other.
2. When one or more of the components of the liquid mixture is sensitive to heat.
3. When a comparatively small amount of solute is present and the boiling point of the solvent is lower than that of the solute, a considerable heat saving is realized by employing an extraction operation.
4. When the organic solvent used has a lower latent heat of vaporization than the carrier solvent, extraction may effect a considerable heat economy.

Uses of Solvent Extraction. In recent years, solvent extraction has come into rather wide use in the chemical industry. Although earlier use of this method of separation was almost wholly limited to the petroleum industry, recent developments have resulted in a rather general acceptance of solvent extraction as a separation method in various fields throughout the chemical industry. Among the more important industrial applications of solvent extraction are those in the wood chemical industry, the perfume and flavoring industries, the petroleum industry, and in the manufacture of edible and drying oils. Othmer [21] cites a number of patents on industrial extraction apparatus employed in these industries.

New Developments. Elgin [11] in a review of new applications of solvent extraction mentions the solvent dewaxing and deasphalting of lubricating oils by means of countercurrent contacting of the oils with liquid propane. Additional processes are the Solexal process for purifying edible fats and oils by means of liquid propane and the Esterol process for separation of fatty acids. Recovery of oils from vegetable seeds and beans continues to be studied, improved and developed commercially.

The latest development is the improved Allis-Chalmers process for soybean oil involving steaming and flaking the seeds and countercurrently extracting with hexane. Color bodies

distributed between the oil and meal are removed by additional processing of the extract oil with a resulting better color than first oil and an additional yield of 45 pounds per ton of seed over the old cold pressing method.

Other new applications of the extraction operation are in the separation of azeotropic mixtures of aromatic and aliphatic hydrocarbons employing sulfolenes or sulfonyl ethers as solvents. Polyolefin glycols of high molecular weight are employed in the separation of olefinic and aromatic hydrocarbons from paraffinic. Liquid-liquid extraction has also been found useful in the recovery of sulfanile halides in the preparation of surface active agents. Selective solvents such as nitromethane, methyl acetic acid and liquid sulfur dioxide are employed followed by treatment with an equal volume of an immiscible non-polar solvent such as hexane, propane or butane.

Use of Liquid Propane. Nixon and Nixon [17] have done extensive research on the use of liquid propane near the critical temperature as a selective solvent for fatty acids in systems involving mixtures of fatty acids and resin acids. Specifically these investigators have been interested in a study of the properties of liquid propane as a solvent and also in the separation of the components of tall oil by extractive means.

Double Selective Solvent Systems. Representative of the many recent patents granted on extraction processes for use in petroleum refining is a Dutch patent granted to N. V. De Bataafsche for the separation of naphthenic acids from mineral oil by extraction (9). In this process, a double selective solvent system is employed countercurrent to the liquid mixture to be separated. The selective solvents, 92 per cent ethyl alcohol and benzine at from 60 to 80 degrees centigrade, are introduced at opposite ends of the extraction column. Employing a one to one mixture of the mineral oil and naphthenic acids by weight as feed, 98 per cent recovery of each component is obtainable.

A United States patent granted to Stephen E. Freeman (14) describes the use of a two phase solvent system such as iso-octane and furfural useful in removing the free fatty acids occuring in natural edible and drying oils such as palm oil, coconut oil and tung oil. Other examples are given.

Recovery Considerations. Shrader (39) reports that a saving of 50 per cent in production cost due to decreased handling alone is obtained by employing solvent extraction for the recovery of oil from castor beans. Either benzene or petroleum naphtha may be employed as the solvent in this process. This same investigator reports that an additional advantage of the extraction process lies in the better oil recovery obtainable over the older expression methods.

Alcohol and Acetone Recovery. Othmer and Trueger [22] give data and details of operation for recovery of acetone and ethanol from aqueous solution by means of countercurrent extraction with a solvent immiscible with water. A suitable solvent suggested is isopropyl ether.

Shreve [40] mentions the application of liquid-liquid extraction for the recovery of essential oils vital to the manufacture of perfumes and flavorings.

In the past ten years, numerous patents have been granted for specific applications of liquid-liquid extraction to separate problems throughout the chemical industry.

B. Methods of Extraction

Liquid-liquid extraction may be classified into five operational types which are discussed in the following paragraphs. Schematic flow diagrams for typical operating methods will be found in Figure 1, page 12.

Single Contact. [25] The single contact method of extraction, Figure 1a, page 12, is regarded as analogous to simple flash distillation. In this system of operation the liquid mixture to be separated is contacted with a single charge of the solvent, the phases separated and solvent and extract recovered without further treatment. This method of operation is the

least effective and is rarely feasible on an industrial scale. Generally, in single contact extraction, equilibrium is closely approached. Thus the percentage recovery of the solute is governed by the proportion of solvent to feed and by the equilibrium conditions existing between the phases of the system employed. However, in any case, the recovery of extract is small compared to the large expenditure of solvent required, and concentration of the extract layer is invariably low; moreover, the degree of separation of the original components is poor.

Simple Multiple Contact. (25) Simple multiple contact, or concurrent multiple contact procedure, schematically represented in Figure 1b, page 12, differs from the single contact method only in that the solvent employed is divided into several portions with the feed being treated separately with each of these portions in successive steps or stages. Depending on the number of stages and the amount of solvent employed, varying degrees of separation of the extract may be obtained, but with a specified amount of solvent, a finite limit to the removal of the extract is approached as the number of successive stages becomes infinite and the solvent portions become infinitesimal. As in the case of the single contact method, the degree of separation obtainable is low as compared with the

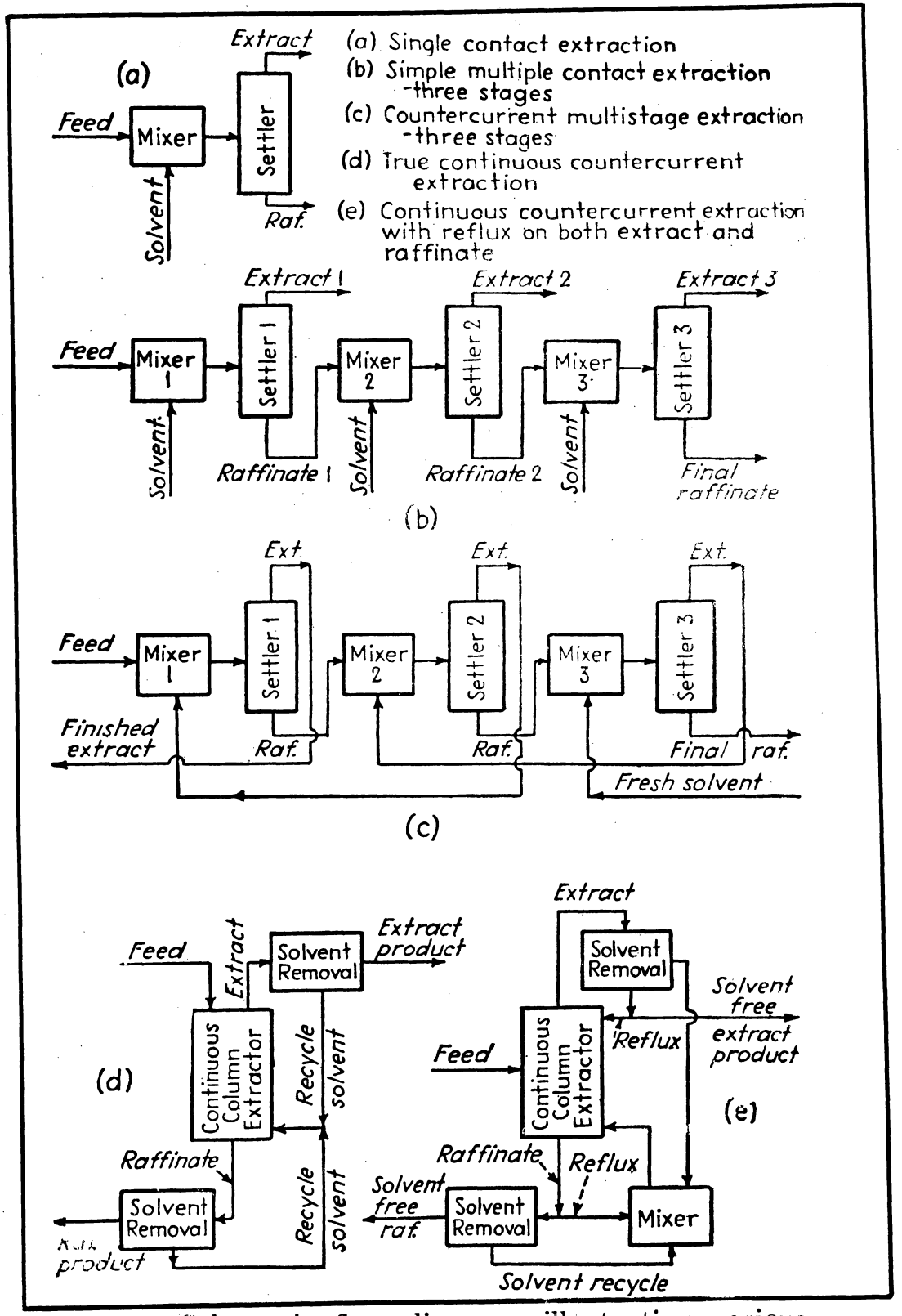


FIG. 1.—Schematic flow diagrams illustrating various methods of operating extraction processes.

proportionate amount of solvent required for the separation. Consequently, extract concentration is low and the process is industrially feasible only where solvent costs are negligible and where the solvent is highly selective for the extracted portion of the feed.

Countercurrent Multiple Contact. (26) In the countercurrent multiple contact method of extraction, outlined in Figure 1c, page 12, fresh solvent and feed are introduced at opposite terminals of an extraction battery, such that the final raffinate product leaves the battery after contact with the fresh solvent. The solvent and extract product are removed at the opposite end of the battery following contact with the incoming feed. Although any number of separate cells or stages may be employed, in practice the usual number is from three to six, depending on the system employed. The principle of operation is based on the fact that the weaker extract layer in any given stage, which has been contacted and brought to equilibrium with a more dilute raffinate phase , is still capable of removing extract from a more concentrated raffinate phase in the following stages. This scheme of operation, analogous to simple plate distillation, holds two distinct advantages over the two methods previously discussed in that (1) for any degree of extraction desired, it is more economical

of solvent, and (2) for a fixed number of stages and a given amount of solvent, a higher degree of separation of the extract and raffinate components is obtainable. At present, countercurrent multiple contact operation is the method most frequently employed industrially.

Continuous Countercurrent Liquid-Liquid Extraction. (27) Continuous countercurrent extraction, represented in Figure 1d, page 12, is accomplished by causing continuous countercurrent flow between the feed (liquid mixture to be separated) and the solvent. In practice, this is accomplished by dispersing one of the liquid phases in the other, or by causing one of the phases to distribute itself over the surface of an inert packing within the column and utilizing the difference in densities of the two liquid phases to cause countercurrent flow. Theoretically, either phase may be dispersed or caused to be discontinuous in the other, but this choice often depends on the system employed and the relative ratio of solvent to feed necessary to obtain the required degree of separation. However, regardless of which phase is dispersed, feed to the column is arranged such that the denser liquid enters at the top while the other is introduced at the base of the column. In cases where the solvent is only moderately selective and a high degree of separation is required, the solvent of necessity becomes the continuous phase. Considered from a theoretical standpoint, the continuous counter-

current system offers the maximum possible extraction efficiency, however actual results are governed to a great degree by the specific extraction rate and the excellence of contact obtainable in available equipment. Not only the relative size of equipment units, but also first cost and operating costs must be considered in evaluating the effectiveness of this method and the performance of continuous countercurrent equipment.

Continuous Countercurrent Liquid-Liquid Extraction with Reflux. (27) Continuous countercurrent extraction with reflux, schematically represented in Figure 1c, page 12, utilizes the principle of reflux in a manner analogous to that in fractional distillation. In practice, equipment operation is the same as that of the continuous countercurrent type with the exception that reflux is introduced at either one or both terminals of the column in order to enrich the existing raffinate or extract products. Extract reflux is supplied by returning a portion of the extract layer from which the solvent has been wholly or partially removed, usually just to the point where the extract is saturated with the solvent. Similarly, for raffinate reflux, a part of the raffinate layer is mixed with the incoming solvent, also just to the point of saturation. The reflux thus introduced is subject to certain limitations not met in distillation since any reflux return must not result in a completely miscible

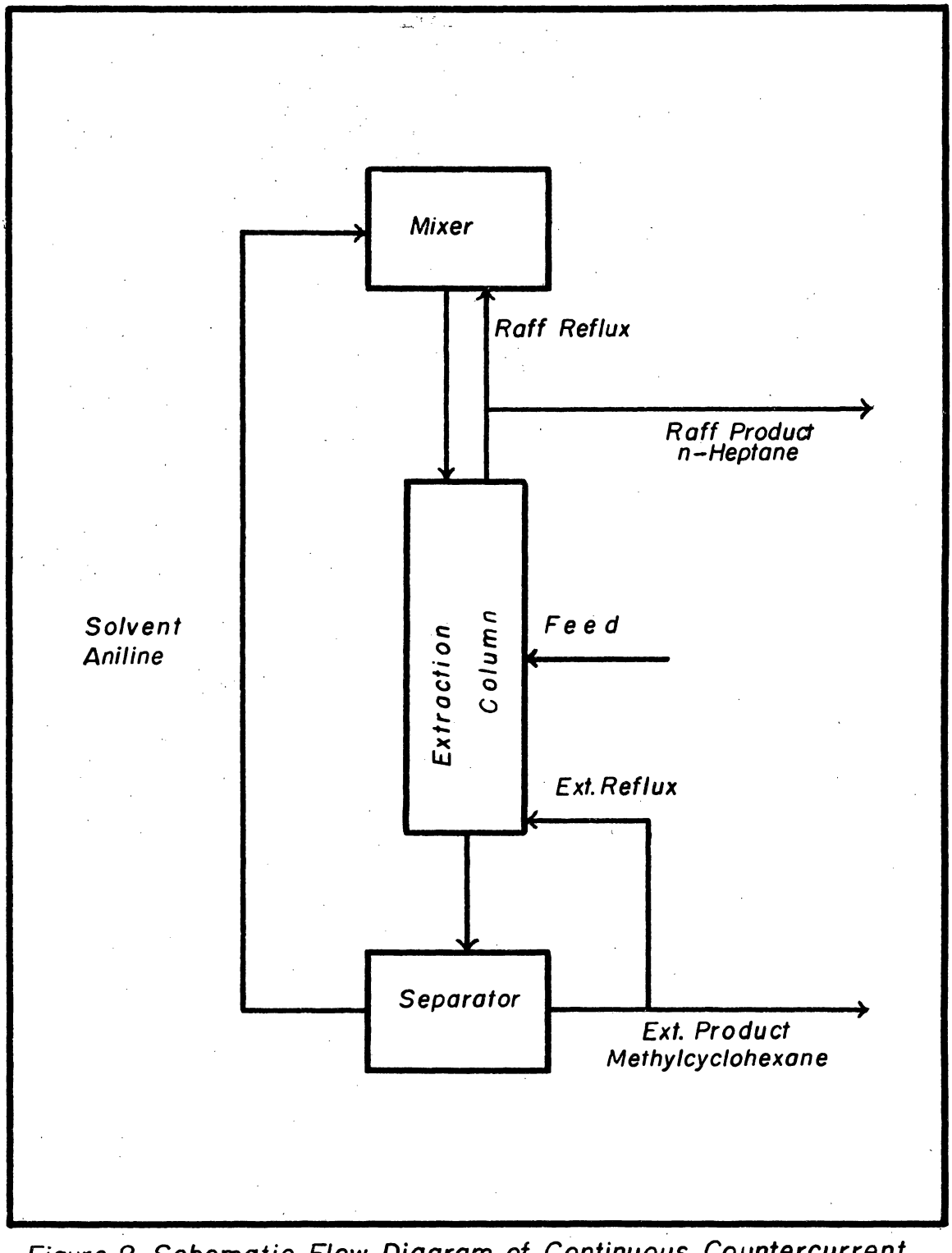


Figure 2 Schematic Flow Diagram of Continuous Countercurrent Extraction with Reflux Using the System Methylcyclohexane-n-Heptane-Aniline.

system at either end of the column. Thus, the proportions of reflux are restricted to those which give a system whose composition remains in the two layer region. In addition, the density of the reflux must be different from that of the extract, or correspondingly, of the raffinate layer. Use of reflux to obtain sharper separation of the feed into extract and raffinate products entails the use of larger quantities of solvent, and an equivalently larger heat consumption in effecting separation of the extract and solvent. The advantages to the application of reflux rest in the fact that it is possible to obtain, with moderately or only poorly selective solvents with reflux, the same degree of separation as can be obtained with a highly selective solvent in a process without reflux. Possibilities in the use of reflux must be determined in relation to the specific extraction system and its phase equilibria. The application of continuous countercurrent liquid-liquid extraction with reflux as applied to the system methylcyclohexane - aniline - n-heptane is schematically represented in Figure 2, page 16.

C. Equipment

Choice of Equipment. The type of equipment or apparatus employed in effecting separation of the components of a liquid mixture by extractive means varies rather widely, depending on the physical characteristics of the liquid mixture to be separated and the degree of separation desired. Physical size of the equipment is largely controlled by a number of economic factors such as the amortization of first cost, the unit value of the final product, comparative costs of major equipment, useful life of the equipment, and the size of the product market. To a lesser extent, the size and type of equipment selected depends on available plant space and the cost or availability of certain materials of construction. There are seven major types of extraction equipment which will be discussed in the following paragraphs.

Bubble Cap Columns. The bubble cap column applied in liquid-liquid extraction is structurally the same as that employed in fractional distillation. In normal operation, the continuous phase is introduced at the top of the column , flows downward across each successive plate and is withdrawn at the base of the column. The discontinuous phase, lighter in density, is introduced at the base of the column and rises past the down flowing continuous phase through bubble caps on each succeeding plate

to the top of the column. In passing through succeeding plates, this phase is further subdivided and the area of contact between the phases is effectively increased. The lighter discontinuous phase is then removed at the top of the column. The most serious limitation on this type of equipment lies in the fact that throughput is restricted by the small free cross-sectional area at the bubble plates.

Rogers and Thiele [34] studied the operation of a small, three plate bubble-cap column of glass construction, in which the interior of the column could be observed during operation. The liquids to be separated consisted of a mixture of light and heavy hydrocarbons (the latter constituting the motor oil fraction of the petroleum crude) with dichloroethyl ether employed as the solvent. The low plate efficiencies of 33 per cent, were attributed to the lack of agitation and resulting poor contact between the phases in the type of column employed. Row, Koffolt and Withrow [35] report that bubble cap and perforated plate columns differ markedly in operation, both from the standpoint of the fluctuation in the rates of bubble formation and in obtainable column efficiencies. These investigators also report that perforated plate columns are superior to bubble cap columns from the standpoint of liquid throughput.

Perforated Plate Columns. [32] The perforated plate column differs from the bubble-cap column in that a series of plates with small perforations replace the bubble plates. Operation of the column is similar. However, since the perforations may be spaced closer together and more uniformly across the plate, there is better dispersion of the discontinuous phase in the continuous phase. This results in an increase of interfacial area and consequently better extraction plate efficiency, although throughput is still limited by the free cross-sectional area at the plates. Treybal and Dunoulin [41] report that the design of plates and plate spacing have a profound effect on the permissible solvent throughput. However, with a plate spacing of two column diameters or greater this effect is less noticeable. Allerton, Strom and Treybal [1], investigating the characteristics of perforated plate and packed towers, found perforated plate column to be superior in throughput, and also found the overall extraction rates for the systems kerosene - benzoic acid - toluene and kerosene - benzoic acid - water to be superior in the perforated plate column. Moulton and Walkey [19] report that the ratio of solvent rate to feed rate is an important variable in efficiency considerations for perforated plate columns.

Packed Columns. [31] In the packed column, packing material such as berl saddles, metal cloth, Raschig rings, quartz or coke,

depending on the type used, is either stacked or dumped at random within the column. The principle of operation lies in the fact that the packing is preferentially wetted by one of the liquid phases employed. With the resultant spreading of this liquid over the surface of the packing, a large increase in transfer area is effected. With the lighter liquid introduced at the base of the column and the heavier liquid at the top, countercurrent flow between the two phases is established. Separation of the phases takes place simultaneously at the top or the bottom of the column depending on whether the lighter or heavier liquid constitutes the dispersed phase.

Row, Koffolt and Withrow (35) have employed the system toluene - benzoic acid - water in a study of the effect of various types of packing. These investigators report that the type of packing has a marked effect on the column throughput. Vartaressian and Fenske (43) report that throughput does not appreciably effect the extraction efficiency, however, shape or size of the packing and state of saturation of the feeds have considerable effect on performance. It is suggested that the composition of the feeds or the ratio between the feeds may also have some effect. Appel and Elgin (3) report that the column capacity is greatly dependent on the rate of feed of the discontinuous phase, and only slightly dependent on the feed

rate of the continuous phase. Drop size was also studied and found to be relatively unimportant in this connection. In comparing the operation of spray and packed columns, it was found that the spray column is either more or less effective in extraction than the packed column depending on the drop size produced in the former, and the nature and size of packing in the latter. Sherwood, Evans and Longcor (38) corroborate the findings of Appel and Elgin and also report that flooding rates for spray columns are much higher than for equivalent packed columns.

Spray Columns. (31) Perhaps the most widely used type of equipment for liquid-liquid extraction is the spray column. In this type of apparatus, large contact area between the continuous and discontinuous phases is attained by atomizing the discontinuous phase through a suitable dispersion nozzle into the continuous liquid mass within the column. In operation, liquid feeds are introduced at opposite ends of the column, the denser liquid entering the top of the column. Either liquid may constitute the continuous phase, the other being dispersed into it as the discontinuous phase. As the result of a difference in densities of the two liquids, a counterflow is established, with the rate of flow being determined by the magnitude of this difference and the physical dimensions of the apparatus employed. The limitation in throughput of most extraction apparatus is obviated in the

spray column, since there is no need for packing or baffling which seriously restricts flow within the column. The results of most investigations indicate that the spray column has the best operating characteristics of the equipment types generally available, notwithstanding serious difficulties encountered in obtaining good dispersion with certain liquid systems. (5)(7)

Johnson and Bliss [18], studying the operating characteristics of a spray column, reported that the magnitude of the mass transfer coefficient (Ka) is largely dependent on the direction of extraction. These investigators report that when extraction is from the continuous to the dispersed phase, the mass transfer coefficient is greater than when diffusion is in the opposite direction. Also, drop coalescence, which tends to reduce contact area, is less. Elgin and Browning [11], employing the system acetic acid - water - isopropyl ether with isopropyl ether as the selective solvent, report that the specific contact area (a) is variable and a function of drop diameter, velocity and the rates of flow of the phases. These investigators [12], studying the effect of flow rates on the same system, found that the H.T.U. and the H.E.T.P. decreased with increased ether flow where the ether constitutes the dispersed phase. However, where the ether phase is continuous, the H.T.U. and H.E.T.P. were found to be directly proportional to the rate of ether flow. The effect of flow rates of the aqueous phase was also studied and similar

results were obtained except that the effect was found to be less marked in the latter case. It was further determined that H.T.U. varies directly with the ratio of the dispersed phase to the continuous phase.

Wetted-Wall Columns. The wetted-wall column has been directly adapted for use in extraction operations from equipment rather widely used in gas absorption studies. In the operation of wetted-wall columns for gas absorption purposes, the absorbent liquid is caused to flow in a thin film down the inside wall of the column. The gas is then passed countercurrently to the liquid in substantially viscous flow with a resultant mass transfer from the gas to the liquid phase by reason of a concentration differential of the absorbed component in the two phases. The application of this equipment to extraction merely involves exchanging the gas phase for a second liquid phase with resultant mass transfer between the contacted liquid phases. Wetted-wall columns are useful almost solely as laboratory devices for determination of mass transfer rates. The serious limitation of the wetted-wall column which prevents its use industrially is the limited contact area obtainable between the phases, and the resulting low extraction rate per cubic foot of column volume.

Treybal and Work (42) employing the system acetic acid - water - benzene studied the effect of the flow rates of both

phases on the individual extraction coefficients of the film and core. These investigators report that neither phase may be said to control, and consequently it is not possible to break down the overall extraction coefficient into individual transfer coefficients. Cowings and Briggs (8) report that wetted-wall columns less than one-inch in diameter have transfer coefficients double those of packed columns at flow rates ten times as great. Consequently a one-inch wetted-wall column was found to have the same capacity as a three-inch packed column, but for equivalent extraction, the required height for the wetted-wall column was found to be five times as great as that for the packed column. Brinsmade and Ellis (6) employing the system acetic acid - methylisobutyl ketone - water in a wetted-wall column found the distribution coefficient to be approximately unity. They also report that either individual coefficient but not both may be affected by both flow rates.

Baffle Plate Columns (30) "A baffle plate column, reported to be an effective countercurrent extraction device, is receiving increasing attention in industrial use. It is provided at suitable intervals with solid baffle plates filling only partially the column cross-section. These are provided with a short lip to form a tray and so arranged that the space left free is on opposite sides of the column for alternate plates. The light liquid flows up underneath, around the baffles, and

up through the free area to the tray above. The heavier liquid flows down to the baffle tray at the edge near the wall, across the tray and overflows the lip to the tray below. The equipment depends upon the length of travel rather than subdivision to provide sufficient contact area."

Spinner Columns. (20,36) The spinner column is an internally agitated column with a comparatively large height to diameter ratio. Liquid flow in the column may be countercurrent but is usually cocurrent downward in the annular space between the spinner, a rotating central shaft, and the inner walls of the column. The denser liquid, a rotary motion imparted to it by the spinner, flows helically downward exiting at the base of the column. Rey and Lochte (20) attribute the introduction of the spinner column to Tiedcke. Schutze, Quebendeaux and Lochte (36) report a ten foot by ten millimeter diameter spinner glass column with a five millimeter diameter spinner to be effective in difficult laboratory extractions involving the separation of petroleum acids.

Other Liquid-Liquid Extraction Equipment. Numerous types of extraction equipment exist other than those already mentioned, however their usefulness in most cases has been limited to experimental study. Bergelin, Lockhart and Brown (4) employed a horizontal tube extractor and varying mass velocity rates of flow in an effort to determine overall transfer coefficients. These

investigators report that transfer coefficients depend on the relative rates of flow of the contacted phases with minimum transfer occurring at equal mass velocity rates of the solvent and extract.

Elgin (30) mentions the use of turbosgitators in the nitrobenzene process for the refining of lubricating oils, and in the Duosol process the use of specially designed nozzles for mixing the liquid layers as they enter each compartment of the extractor.

In 1912 Friedricks (15) reported on the introduction of a glass spiral liquid-liquid extractor for laboratory use. The condenser and inner extracting funnel-tube consisted of glass helices within another glass tube. In both cases, the jacketed spiral provided a long channel of contact.

D. The System: Methylcyclohexane - Aniline - Normal Heptane

Vartaressian and Fenske (44) compared thermodynamically the separation of mixtures of methylcyclohexane and normal heptane by fractional distillation with separation by liquid-liquid extraction employing aniline as a selective solvent at a temperature of 25°C and atmospheric pressure. As a definite problem, the separation of a 50 per cent by weight mixture of normal heptane and methylcyclohexane was considered. Physical

constants for the components were as shown in TABLE I, page 29.

Saturation and Equilibrium Data. Experimental values of solvent and hydrocarbon phase data at 25°C and atmospheric pressure were obtained by Varteressian and Fenske [44] using aniline as the solvent. They plotted on triangular coordinates, the saturation and equilibrium data as reproduced in Figure 3, page 30. In place of the eleven tie lines shown by these investigators, a conjugate tie line has been added to their plot.

Conjugate Tie Line. [28] To find the composition of a phase B in equilibrium with a phase A of known composition, a line is drawn from the composition of phase A parallel to the base of the diagram to the point of intersection with the conjugate line. From thence, a second line is constructed parallel to the leg of the triangle opposite phase A to the intersection with the equilibrium line of phase B. The point of intersection thus determined is the composition of phase B in equilibrium with the original composition of phase A.

Figure 4, page 31, is a graphical representation of their equilibrium data with percentages of methylcyclohexane in the solvent layer plotted against percentages of methylcyclohexane in the hydrocarbon layer. This equilibrium curve is used by Varteressian and Fenske [44] for the calculation of the number of theoretical plates or stages required for given separations.

TABLE I

PHYSICAL CONSTANTS OF THE COMPONENTS OF THE SYSTEM

METHYLCYCLOHEXANE - ANILINE - n-HEPTANE*

Component	Boiling Point °C	Density (gm/cc)	Refractive Index N_D^{20}
Methylcyclo-hexane	100.65	0.7691	1.4233
n-Heptane	98.4	0.6835	1.3878
Aniline	---	1.0215	1.5858

*Vartazarian, K. A. and Fenske, M. R. The System Methylcyclohexane - Aniline - n-Heptane. Ind. Eng. Chem. 29, 270-277 (1937).

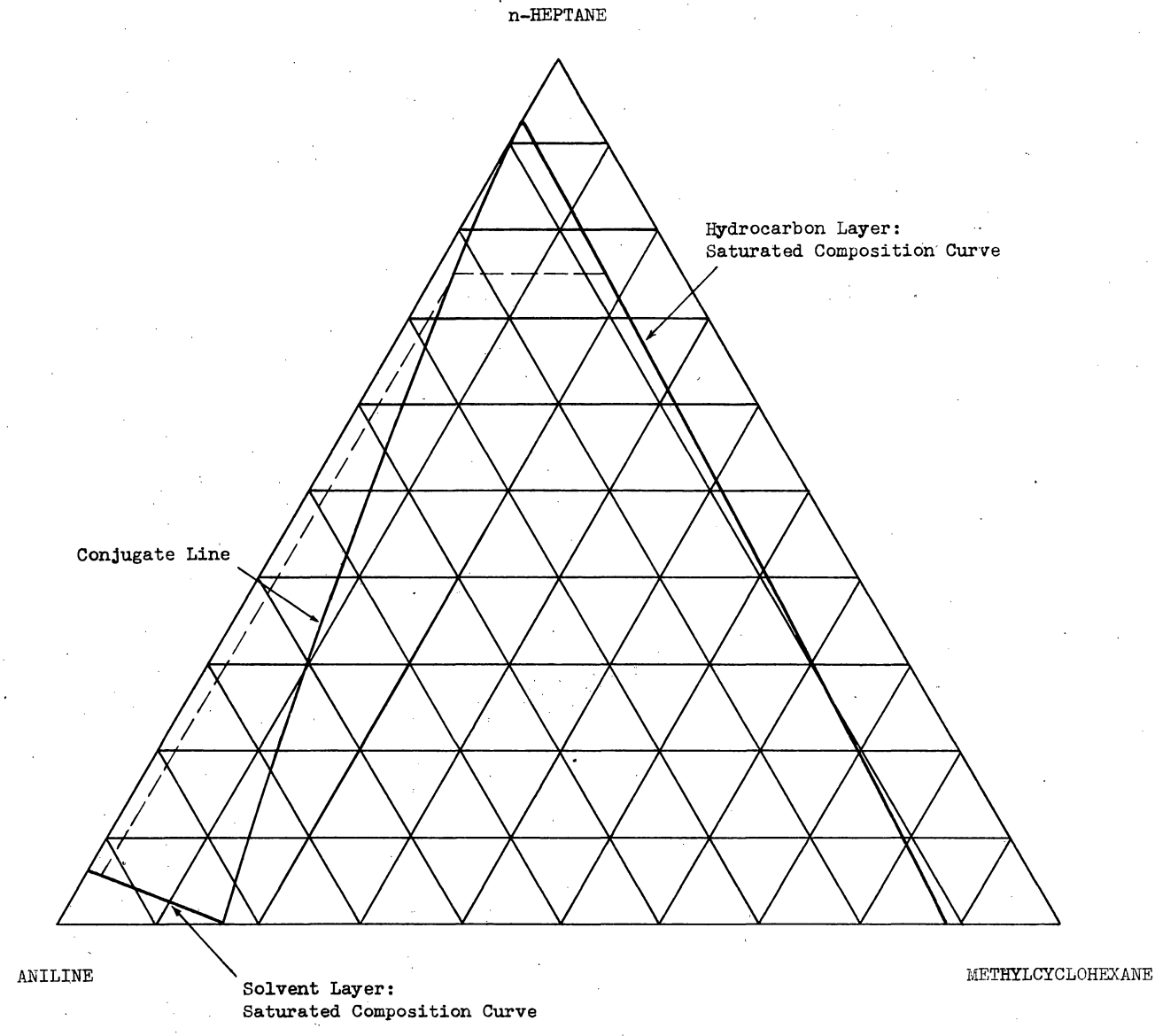


Figure 3. Saturation and Equilibrium Diagram in Weight Per Cent for the System Methylcyclohexane - Aniline - n-Heptane at 25°C and 1 Atmosphere

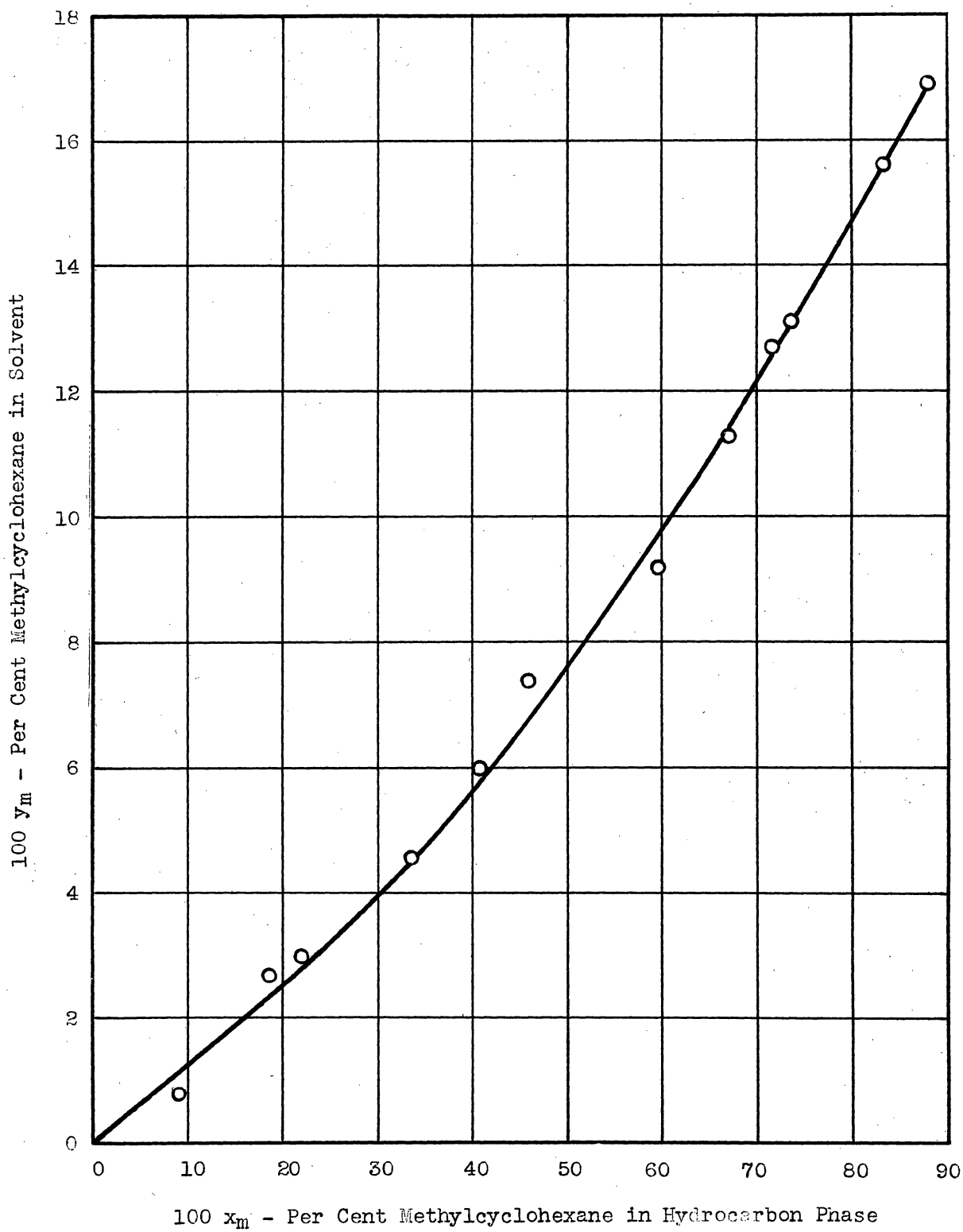


Figure 4. Equilibrium Compositions for Methylcyclohexane in the System: Methylcyclohexane - Aniline - n-Heptane at 25°C and 1 Atmosphere (42)

Figure 5, page 33, is a reproduction of their graph of refractive indices versus per cent methylcyclohexane by weight in both the solvent and raffinate layers as obtained by Varteressian and Fenske (44).

Saturation and Methods of Obtaining Equilibrium Data. The method employed by Varteressian and Fenske for obtaining saturation data is as follows: (44)

"Aniline was added to the point of turbidity, at 25°C., to mixtures of n-heptane and methylcyclohexane of known compositions and weights. From the weights of aniline added, the composition of the saturated hydrocarbon layers were determined. The refractive indices of these mixtures were obtained at the same temperature. Next, mixtures of n-heptane and methylcyclohexane of known compositions were added to known weights of aniline, again just to the point of separation of the second liquid phase. From the weights of n-heptane and methylcyclohexane added, the compositions of the saturated solvent layers were determined. The refractive indices of these mixtures were also obtained."

In determining equilibrium compositions, mixtures of the three components were prepared in such proportions as to separate into approximately equal amounts of the two phases. These mixtures were kept in a constant temperature bath at 25°C for 24 hours with hourly agitation. From the refractive indices of the separated phases, the compositions were determined.

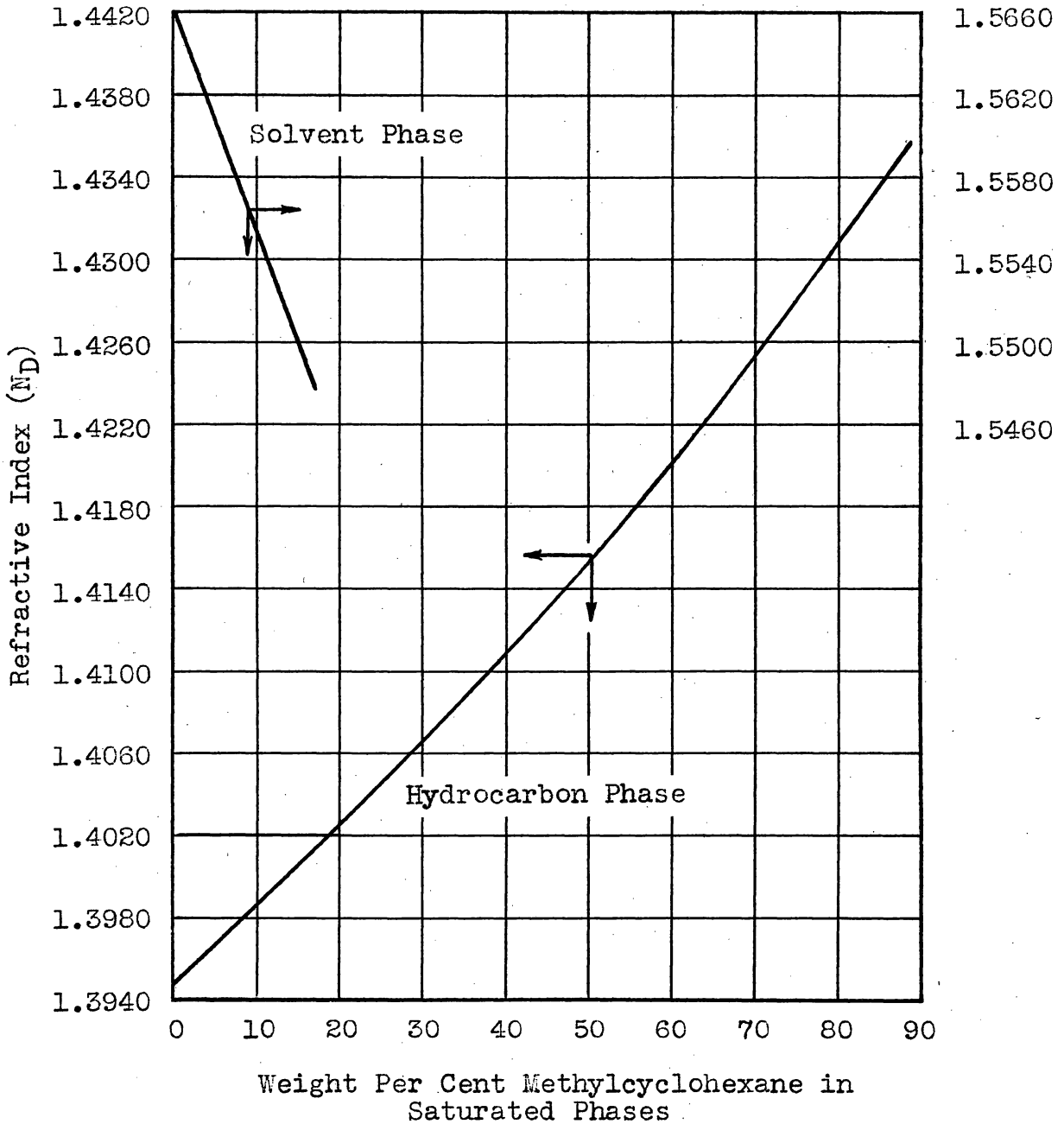


Figure 5. Weight Per Cent Methylcyclohexane in Satruated Phases Versus Refractive Indices (N_D) for the System: Methylcyclohexane - Aniline - n-Heptane at 25°C and 1 Atmosphere

E. Method of Extraction Calculations

General Considerations. As the result of increased application of the extraction operation to problems of separation, both in the laboratory and on an industrial scale, calculation methods have been devised which, when applied to operational data, yield quantitative relationships useful in evaluating certain operating characteristics. According to Elgin [11], the available methods allow calculation (1) of limiting conditions, (2) of the results to be expected, (3) of a comparison of the results to be anticipated from changes in operating variables, and (4) of different methods of conducting the extraction operation. The more important considerations involved in calculations for extraction operations and their application will be considered in the following paragraphs.

Nomenclature. The following nomenclature introduced by Varteressian and Fenske [44] will be employed throughout:
(See Figure 6, page 42)

X_n = Weight fraction of heptane in hydrocarbon phase.

X_m = Weight fraction of methylcyclohexane in hydrocarbon phase.

Y_h = Weight fraction of heptane in solvent phase.

X_m = Weight fraction of methylcyclohexane in solvent phase.

= Distribution coefficient

$V_f - 1$ = Weight of overflow (solvent) to feed section per unit time.

O_f = Weight of overflow (hydrocarbon phase) to feed section per unit time.

P_e = Weight of extract product saturated with solvent per unit time.

F = Composition of feed, on solvent basis, weight per cent.

f = Composition of feed, weight per cent.

f_e = Composition of solvent phase in equilibrium with feed.

V_s' = Weight of solvent phase to separator per unit time.

s' = Composition of solvent phase to separator, weight per cent.

O_e = Weight of hydrocarbon phase reflux per unit time.

E = Composition of extract product on solvent-free basis, weight per cent

e = Composition of hydrocarbon phase reflux, weight per cent.

S_o = Weight of solvent fed to mixer per unit time.

S = Solvent removed from separator per unit time.

a' = Operating point for enriching section.

b' = Operating point for exhausting section.

R = Composition of raffinate product on solvent-free basis, weight per cent.

r = Composition of hydrocarbon phase to mixer, weight per cent.

O_r = Weight of terminal hydrocarbon per unit time.

V_r' = Weight of solvent phase reflux per unit time.

P_r = Weight of raffinate product saturated with solvent per unit time.

f' = Composition of solvent phase differing from the feed only in solvent content.

Nernst's Distribution Law. (16) The addition of a solute to a system composed of two immiscible solvents at a constant temperature results in the distribution of that solute between the two coexisting phases in a constant ratio according to its solubility in each, and the relative amounts of each solvent present. This same relationship may be expressed algebraically as follows:

$$\frac{X_a}{X_b} = \beta \qquad (1)$$

where:

X_a = Weight fraction of the solute in phase a
X_b = Weight fraction of the solute in phase b

The corollary to this relationship is stated in the following paragraph:

If two components, miscible in all proportions, be added to a third component with which each of the other two components is only partially miscible, in sufficient quantity such that two phases are produced, the distribution of the two components in the third will be according to the solubility of each and the amount of each present. Algebraically, this relationship may be expressed as follows:

$$\frac{Y_m}{Y_n} = \frac{X_m}{X_n}\beta \qquad (2)$$

In the case of the system methylcyclohexane - aniline - n-heptane, the average value of the distribution coefficient (β) is 1.9 at 25°C and the selectivity of aniline for methylcyclohexane over n-heptane at that temperature is expressed as a ratio, 1.9 : 1. Varteressian and Fenske [44], employing the distribution coefficient and equation (2) as a check on

experimentally determined equilibrium data, found the calculated and observed data to be in very good agreement.

Graphical Solution Method - Countercurrent Extraction with Reflux. (29) Enriching by reflux involves the return to the extract terminal of a portion of the extract layer from which the solvent has been wholly or partially removed, usually to the point of saturation of hydrocarbon with solvent. This tends to cause the outgoing extract and solvent layer to approach equilibrium with a more concentrated material, the extract. Exhausting by reflux involves a similar treatment of the raffinate. Figure 6, page 42, illustrates the application of graphical calculation to the problem of countercurrent extraction with reflux employing the system methylcyclohexane - aniline - n-heptane.

Graphical Constructions. (29) Figure 6, page 42, is constructed in the following manner:

The three vertices of an equilateral triangle represent the three components of the system. Geometrical properties of the triangle are such that any point within the triangular area represents in a definite amount, with the sum of the component fractions equalling unity, or 100 per cent of the total mixture. These same properties then allow the plotting of the equilibrium data within the area of the triangle. Following the construction of the equilibrium lines, the conjugate line (R) determined by

the equilibrium data is plotted in such a manner that equilibrium compositions between phases may be determined graphically. Solvent-free compositions of the feed and the extract and raffinate products are then determined, and the composition points located on the diagram at F, E and R. The lines FS, ES and RS are then constructed determining points f, e and r at the saturation compositions in the hydrocarbon phase, and f', e' and r' at the corresponding saturation compositions of the solvent phase. Having ascertained the range of compositions of the contacted phases, it is necessary to obtain the operating line points and the reflux ratio.

In order to obtain the reflux ratio, the properties of the triangular plot may be utilized as follows: From a material balance around the separator there results:

$$V_e' = S + P_e + O_e \qquad (3)$$

and the composition S and P_e combined is given by operating point a'. Therefore, from the triangular plot it may be shown that:

$$\frac{S + P_e}{O_e} = (ee')/(a'e')^* \qquad (4)$$

$$(a'\delta') + (ee') = (a'e) \qquad (5)$$

* Quantities in parentheses indicate measureable distances between points in the triangular plot of Figure 6, page 42.

Substituting these values in equation (3) above, rearranging and solving for O_e/V_e' gives:

$$\frac{O_e}{V_e'} = \frac{(a'e')}{(a'e)} \qquad (6)$$

But since it is customary to define the reflux ratio as O_e/P_e rather than O_e/V_e', it is desirable to obtain an expression of reflux in terms of the former. This may easily be done when it is considered that $S/P_e = (ee')/(a'S)$. Rearranging and combining this expression with equation (4) above, the following relationship is obtained:

$$\frac{O_e}{P_e} = \frac{(a'e')}{(ee')} + \frac{(eS)}{(a'S)} \qquad (7)$$

Then with (ee') and (eS) defined by the geometrical properties of the chart, and (a'e') + (a'S) also defined as (Se'), the position of the operating point for the required minimum extract reflux ratio may be determined. To do this, line fa'b' is constructed to its intersection with line RS extended at point b', the operating point for the exhausting section under conditions of minimum reflux. The location of operating points for conditions other than minimum reflux may be calculated by means of equation (7).

Theoretical Stage Calculations. In calculating the number of theoretical stages to which the given separation is equivalent, the following graphical constructions are made:

A line is constructed through point r parallel to the base of the triangle to its point of intersection with the conjugate line. A second line is constructed from the point of intersection parallel to side AS of the triangle to its intersection with the solvent layer saturation line, determining the solvent composition in equilibrium with the exit raffinate. This latter point of intersection is then connected to point b' and the resulting straight line extended to its intersection with the hydrocarbon saturation curve determining the first theoretical extraction stage. This operation is repeated until the feed point is reached and is continued beyond the feed point by changing the operating points from b' to a' corresponding to the reflux ratio in the enriching section of the column. Constructions are continued until point e on the hydrocarbon saturation curve is reached, corresponding to the composition of the exit extract product. The total number of such constructions performed then becomes the number of theoretical extraction stages to which the column, as operated, is equivalent.

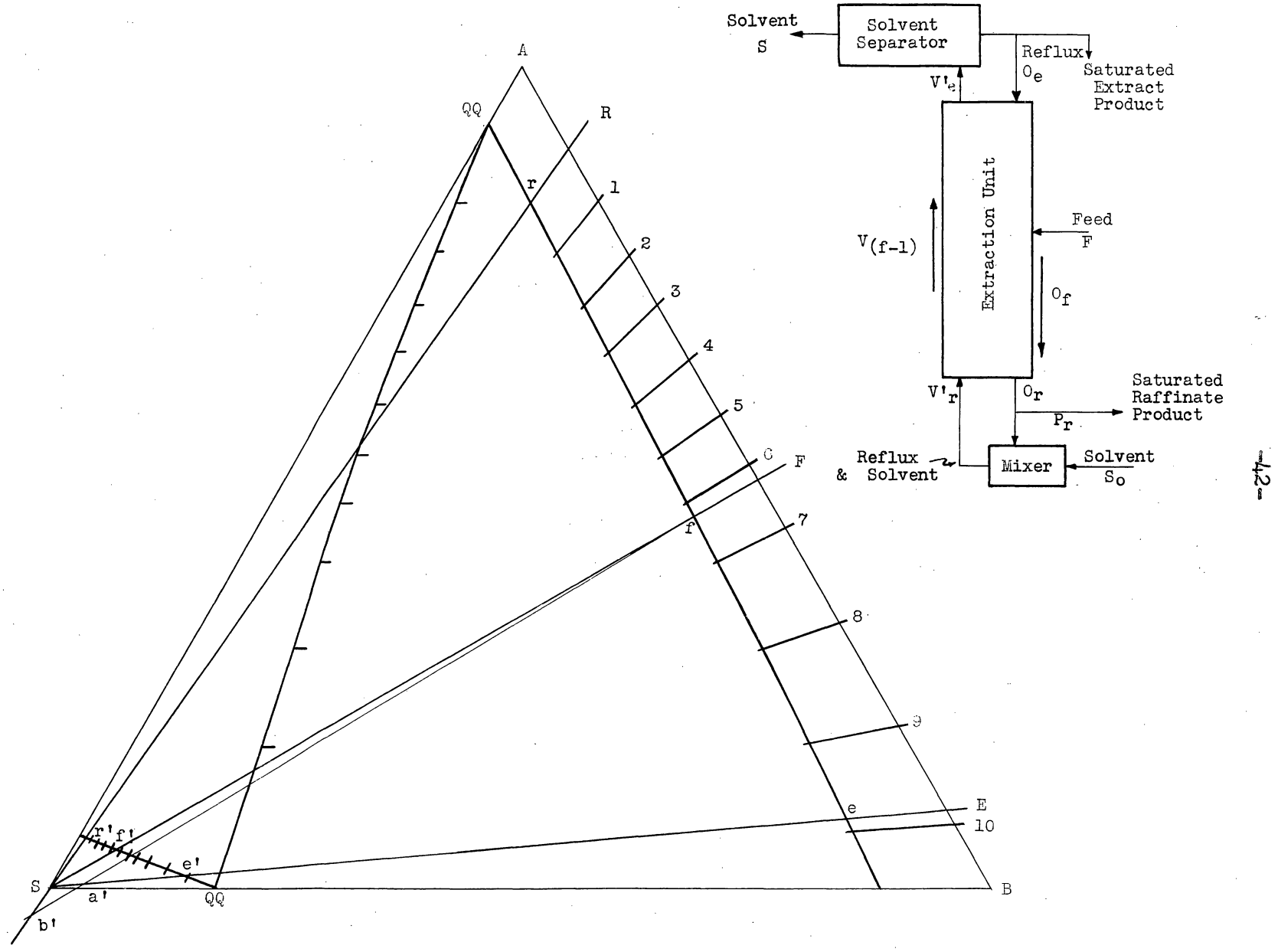


Figure 6. Elgin Method of Graphical Solution Applied to the System: Methylcyclohexane - Aniline - n-Heptane at 25°C and 1 Atmosphere

III. EXPERIMENTAL

A. Purpose of the Investigation

The purpose of this investigation was to determine the effect of the variation of feed and extract reflux introduction points on the product compositions obtained from a spiral-packed liquid-liquid extractor under constant flow rate and temperature conditions. A feed composed of 46.2 per cent methylcyclohexane, 46.2 per cent n-heptane and 7.6 per cent aniline by weight was employed. Operations were carried out at a temperature of 20°C and atmospheric pressure.

B. Plan of Investigation

The plan of this investigation was as follows:

1. Review of Literature. A review of the literature was made to obtain information reported by previous investigators pertinent to the uses, methods of operation and types of equipment employed in the liquid-liquid extraction operation. Further information was obtained on the system: methylcyclohexane - aniline - n-heptane and methods of extraction calculations.

2. Construction of Equipment. Equipment, including a continuous separation still, methylcyclohexane condenser, solvent

cooler, necessary pump and motor assemblies and tankage, was designed, constructed and installed for use in this investigation.

3. Experimental Determinations. Apparatus and instruments were calibrated and saturation and equilibrium compositions as well as densities and refractive indices of such mixtures for the system: methylcyclohexane - aniline - n-heptane were obtained at 20°C ± 0.1°C. Tests runs were made employing different feed and extract reflux input positions as follows:

a. A series of thirty tests employing the various feed and extract reflux input positions was conducted.

b. Samples of extract and raffinate products were obtained at ten minute intervals for the first equilibrium test and at five minute intervals for all subsequent tests until the compositions of such products were constant as determined by refractive index measurements.

c. Refractive indices of products obtained were interpreted as product compositions from refractive index - composition relationships previously obtained.

4. Evaluation of Results. The results obtained in the experimental tests were evaluated by means of theoretical plate calculations. The mathematical correlation between theoretical plates and theoretical flights for the stripping section was obtained by graphical analysis. Theoretical plate - theoretical flight - actual flight relationships were determined statistically.

C. Materials

The following materials were employed in this investigation:

Methylcyclohexane (Technical). Refractive index (n_D^{20}): 1.4239. Specific gravity (d_4^{20}): 0.770. Boiling point at 716 mm pressure: 98.1°C. Dry point at 716 mm pressure: 99°C. Composition:(2) 95 mol per cent methylcyclohexane (minimum). Probably impurities: toluene and ethylcyclopentane (no complete analysis available). Purchased from the Phillips Petroleum Company, Chemical Products Department, Bartlesville, Oklahoma.

Normal Heptane (Pure). Refractive index (n_D^{20}): 1.3873. Specific gravity (d_4^{20}): 0.682. Boiling point at 716 mm pressure: 95.4°C. Dry point at 716 mm pressure: 95.6°C. Composition: (2) 99 mol per cent normal heptane (minimum). Probable impurity: 3-methylhexane (no complete analysis available). Purchased from the Phillips Petroleum Company, Chemical Products Department, Bartlesville, Oklahoma.

Aniline (Technical). Refractive index (N_D^{20}): 1.5858. Specific gravity (d_4^{20}): 1.023. Boiling point at 716 mm pressure: 180.0°C. Dry point at 716 mm pressure: 181°C. Composition: 95 mol per cent aniline (minimum). Probably impurities: oxidation and polymerization products of aniline (aniline black). No analysis available. Purchased from the Dow Chemical Company, Midland, Michigan.

D. Apparatus

The following equipment and apparatus were employed in this investigation:

Constant Temperature Equipment:

Immersion Heaters. (Two). Calrod units. Two hundred forty watts at 220 volts, sixty watts at 110 volts. Used for heating water in constant temperature bath. Manufactured by the General Electric Company, Schenectady, New York.

Pump, Centrifugal. (One). Model A-1. Type 2720DA. Seven gallon per minute capacity. Used for circulation of cooling water to refractometer. Manufactured by Eastern Engineering Company, New Haven, Connecticut.

Stirrer, Laboratory. (Two). 115 volts, 60 cycle, A. C. Used for circulation of water in constant temperature bath. Manufactured by the Fisher Scientific Company, Pittsburgh, Pennsylvania.

Tank, Glass. (One). Dimensions: Thirty-six inches by fifteen inches by fifteen inches deep. Insulation: One-quarter inch corrugated board. Used as a constant temperature bath.

Temperature Regulator, Precision. (One). Model B. 115 volts, 10 amperes. Used for temperature regulation of constant temperature bath. Manufactured by the Eastern Engineering Company, New Haven, Connecticut.

Extraction Equipment:

Condenser and Receiver, Methylcyclohexane. (One). Single pass. Seven, one-half inch tubes, four feet long. Surface area: 6.15 square feet. Constructed of black iron pipe. Used to condense methylcyclohexane vapors for recycle to extract reflux storage. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia.

Cooler, Solvent. (One). Single pass. Seven, one-half inch tubes, four feet long. Area, surface, 6.15 square feet. Constructed of black iron pipe. Used to cool solvent recycled from continuous stripping still before entering solvent reservoir. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia.

Extractor, Spiral Channel. [33] (One). Consisting of a five inch heavy duty pipe, five feet long, in which is inserted a one-quarter inch thick mild steel spiral wrapped around a star-

dard two-inch pipe to a pitch of one and three-quarters inches and machined to a light driving fit inside the five-inch pipe. Sealed at both ends by a five-inch cap and packing gland assembly. Used for contacting liquids in countercurrent extraction. Constructed in the Norfolk and Western Railway Car Shops, Roanoke, Virginia.

Nozzles, Extract Reflux. (Six). Seven-sixteenths inch diameter, one and one-half inches long. Capacity: 0.15 gallon per minute at ten pounds per square inch pressure with 120° conal dispersion. Constructed of brass. Used to disperse extract reflux into the continuous aniline phase at the base of the extractor. Constructed in the Machine Shop of the Industrial Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia.

Nozzles, Feed. (Five). Nine-sixteenths inch diameter, one and one-half inches long. Capacity: 0.3 gallon per minute at ten pounds per square inch pressure. Constructed of brass. Used to disperse feed into the continuous aniline phase at the center of the extraction column. Constructed in the Machine Shop of the Industrial Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia.

Pump, Centrifugal and Motor. (One). Type: AE 3A1A. Size: 89-9. Motor: 115 volts, 60 cycles A. C. 267 watts, 2.6 amperes. Five gallon per minute pump capacity at 1550 RPM. Constructed of 24ST aluminum. Used to pump

extract product from the base of the extractor into the continuous stripping still. Manufactured by the American Instrument Company, Silver Springs, Maryland.

Pump, Centrifugal. (One). Type B-1. Motor: 110 volts, 60 cycles. Seven gallon per minute pump capacity. Constructed of bronze. Used to pump raffinate product to overhead feed tank. Purchased from Eimer and Amend, New York, New York.

Pumps, Rotary. (Two). Type: ½ GI. Five gallon per minute pump capacity at 1750 RPM. Constructed of cast iron with hardened steel gears. Used for removing recovered solvent from continuous stripping still and for removing extract product from the methylcyclohexane condenser. Manufactured by the Worthington Pump Company, Harrison, New Jersey.

Pump, Rotary. (One). Type 1. Three gallon per minute capacity at 1750 RPM. Constructed of cast iron with hardened steel gears. Used for circulation of solvent. Manufactured by the Brown and Sharpe Company, Providence, Rhode Island.

Pump, Vacuum. (One). Oil immersion type. Ten cubic feet displacement per minute. Size: 1460. Speed: 350 RPM. Horsepower: 3/4. Lot Number: 98455. Serial Number: A6630. Constructed of cast iron. Used to maintain a vacuum in the continuous stripping still. Manufactured by the F.J. Stokes Machine Company, Philadelphia, Pennsylvania.

Reservoir, Solvent. (One). Consisting of a thirty-gallon steel tank. Used for the storage of recovered

solvent, for saturating the solvent with raffinate reflux and for temperature control in conjunction with a tempering coil consisting of twenty feet of three-quarter inch copper tubing installed in the reservoir. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia.

Still, Stripping and Flash Chamber. (One). Consisting of nineteen, three-eighths inch copper tubes, three feet long, enclosed in a three-foot length of four inch pipe. Pressure Rating: ninety pounds per square inch gage steam pressure. Safety factor of 8 when operated at sixty pounds per square inch steam pressure and twenty inches of vacuum. Used for separating extract product from the solvent for recycle to the system. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia.

Tanks. (Three). Fifteen-gallon steel drums. Used for the storage of feed, extract reflux, and raffinate reflux. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia.

Tank. (One). Five-gallon steel drum. Used for storage and distribution of extract reflux and product. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia.

Beakers, Burettes, Flasks, Sample Bottles and Miscellaneous Glassware. Used in the determination of saturation and equilibrium data and for taking samples during and experimental tests.

Pressure Gage. (One). Zero to sixty pounds per square inch range in two pounds per square inch increments. Used to indicate steam pressure in the continuous stripping still. Manufactured by the United States Gauge Company, New York, New York.

Refractometer. (One). Spencer Abbe' Refractometer. Model Number 547. Refractive index range: 1.300 to 1.710 in increments of: 0.001. Accuracy: ± 0.0002. Used to obtain refractive indices of saturation and equilibrium mixture of product obtained during experimental tests. Manufactured by the Spencer Lens Company, Buffalo, New York.

Rotameters. (Two). Type 1. Serial Numbers 84644 and 84645. Range 0.0 gallons per minute to 1.2 gallons per minute in 0.02 gallon per minute increments with liquids having a specific gravity of 1.023. Used to measure flow rates of aniline phase liquids through the extractor. Manufactured by the Brooks Rotameter Company, Lansdale, Pennsylvania.

Rotameters. (Three). Type 1. Serial Numbers 94635, 94636 and 124774. Range 0.0 to 0.12 gallon per minute in 0.002 gallon per minute increments with liquids having a

specific gravity of 0.764. Used to measure flow rates of feed and extract reflux to the extractor, and methylcyclohexane feed make-up. Manufactured by the Brooks Rotameter Company, Lansdale, Pennsylvania.

Rotameter. (One). Type 1. Serial Number 104629. Range 0.0 to 0.13 gallon per minute in 0.002 gallon per minute increments with liquids having a specific gravity of 0.684. Used to measure flow rates of the raffinate product from the extractor. Manufactured by the Brooks Rotameter Company, Lansdale, Pennsylvania.

Rotameter. (One). Type 1. Serial Number 126775. Range 0.0 to 0.12 gallon per minute in 0.002 gallon per minute increments with liquids having a specific gravity of 0.68. Used to measure flow rates of raffinate feed make-up. Manufactured by the Brooks Rotameter Company, Lansdale, Pennsylvania.

Thermometers. (Two). Type 2219. Range - 10° to 110°C in 1° increments. Used to measure liquid temperatures entering and leaving the extractor. Manufactured by the Weston Electrical Instrument Company, Newark, New Jersey.

Timer. (One). Trade name: "Time-It". 110 volts, 60 cycle A. C. Increments of 0.1 second. Used for timing of sampling operations during experimental tests. Manufactured by the Precision Scientific Instrument Company, Chicago, Illinois.

Vacuum Gage. (One). Bourdon type. Range zero to thirty inches of vacuum in one inch increments. Used to indicate the vacuum in the continuous stripping still flash chamber. Manufactured by the Schutte-Koerting Company, Philadelphia, Pennsylvania.

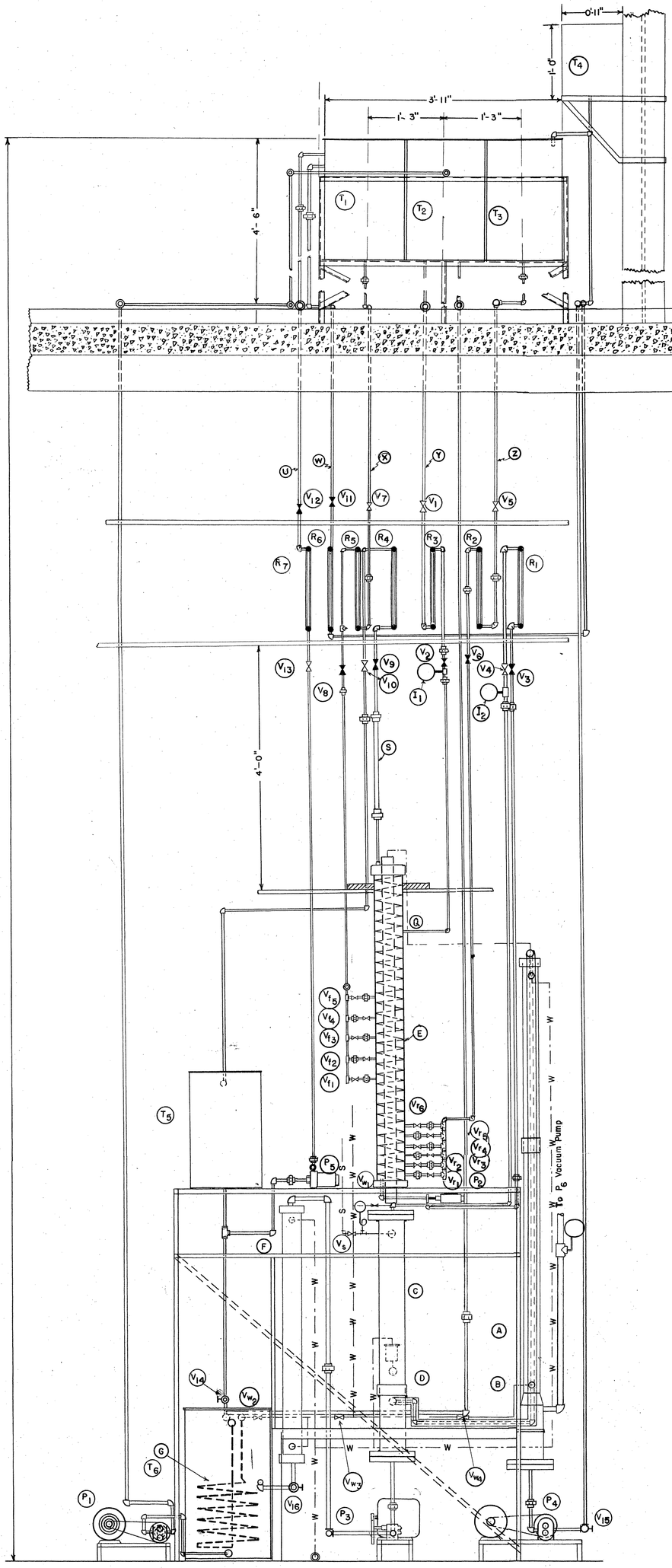
B. Design and Construction

General. At the outset, it was found necessary to make a number of major alterations and refinements in the existing liquid-liquid extraction equipment in order to enable operation of that equipment on a truly continuous basis. These changes and refinements were made in order that:

1. Handling of materials may be minimized.
2. Total amounts of materials handled may be reduced.
3. Actual operation may be maintained over extended intervals of time.
4. Definite comparative results may be btained by providing sufficient operating time for the attainment of equilibrium in the system.

The changes and refinements required to convert the equipment to continuous operation are enumerated and discussed in the following paragraphs.

Extract Reflux Dispersion Nozzles. Drawing Number 2, page 54, shows the details of construction of the extract reflux



A. Methylcyclohexane Condenser
B. Methylcyclohexane Receiver
C. Separation Still Heat Exchanger
D. Separation Still
E. Spiral-Channel Extractor
F. Solvent Cooler
G. Tempering Coil
R_1 Extract Product Rotameter
R_2 Extract Reflux Rotameter
R_3 Solvent Rotameter
R_4 Raffinate Product Rotameter
R_5 Feed Rotameter
R_6 Methylcyclohexane Feed Make-up Rotameter
R_7 n-Heptane Feed Make-up Rotameter
S Sightglass
T_1 Feed Tank
T_2 Solvent Tank
T_3 Extract Reflux Tank
T_4 Methylcyclohexane Recovery Tank
T_5 Raffinate Product Tank
T_6 Solvent Storage Tank
V_1 Solvent Control Valve
V_2 Solvent Cut-off Valve
V_3 Extract Product Cut-off Valve
V_4 Extract Product Control Valve
V_5 Extract Reflux Control Valve
V_6 Extract Reflux Cut-off Valve
V_7 Feed Control Valve
V_8 Feed Cut-off Valve
V_9 Raffinate Product Cut-off Valve
V_{10} Raffinate Product Control Valve
V_{11} Methylcyclohexane Feed Make-up Control Valve
V_{12} n-Heptane Feed Make-upCut-off Valve
V_{13} n-Heptane Feed Make-up Control Valve
V_{14} n-Heptane Reflux Control Valve
V_{15} Recovered Methylcyclohexane Cut-off Valve
V_{16} Recovered Solvent Cut-off Valve
V_{f1-5} Feed Introduction Cut-off Valves
V_{f1-6} Extract Reflux Introduction Cut-off Valves
V_s Steam Control Valve
V_w Extractor Cooling Water Control Valve
V_{w2} Tempering Coil Cooling Water Control Valve
V_{w3} Solvent Cooler Water Control Valve
V_{w4} Methylcyclohexane Condenser Water Control Valve

P_1 Solvent Circulating Pump
P_2 Extract Product Pump
P_3 Recovered Solvent Pump
P_4 Recovered Methylcyclohexane Pump
P_5 n-Heptane Feed Make-up Pump
P_6 Vacuum Pump
I_1 Thermometer – Solvent
I_2 Thermometer – Extract Product

—— S —— Steam
—— W —— Water
- - W - - Hot Water

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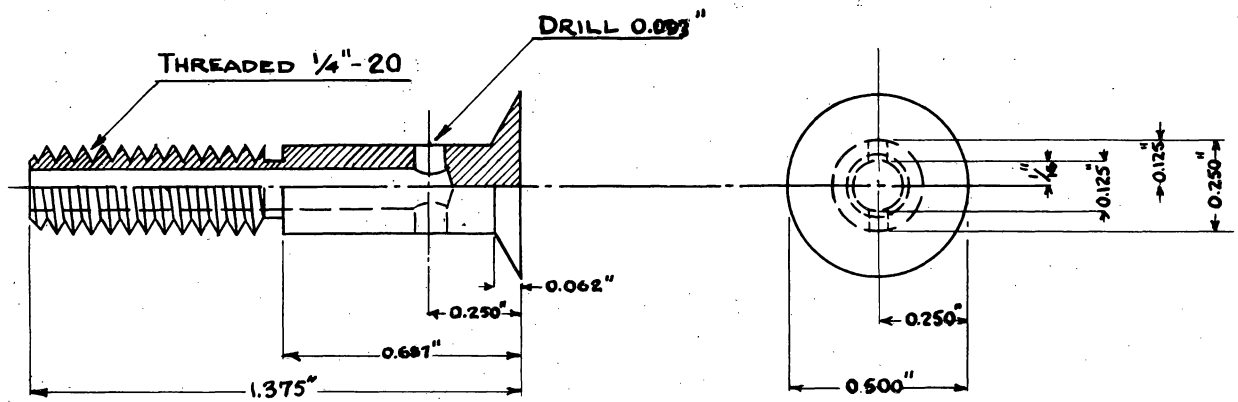
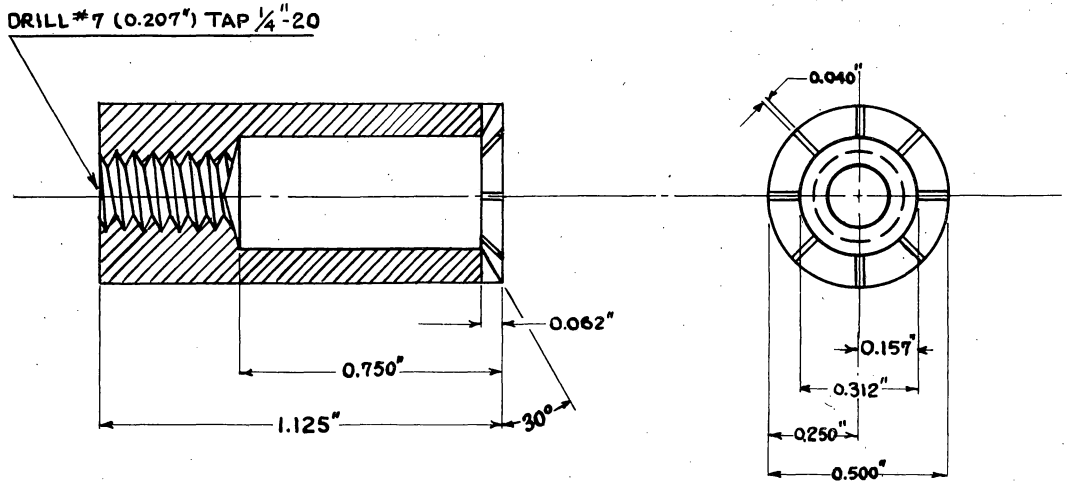
ELEVATION
EXTRACTION ASSEMBLY

Scale 0'-1"=1'-0"
Drawn by [illegible] Date 5/25/48
Traced by [illegible] Date 5/27/48
Checked by [illegible] Date 5/27/48
Approved by [illegible] Date 5/28/48

Case No. 48
File No. 439
Dwg. No. 1



NOZZLE INSERT



NOZZLE SHANK

MATERIAL: Common Brass

FINISH: F.A.O.

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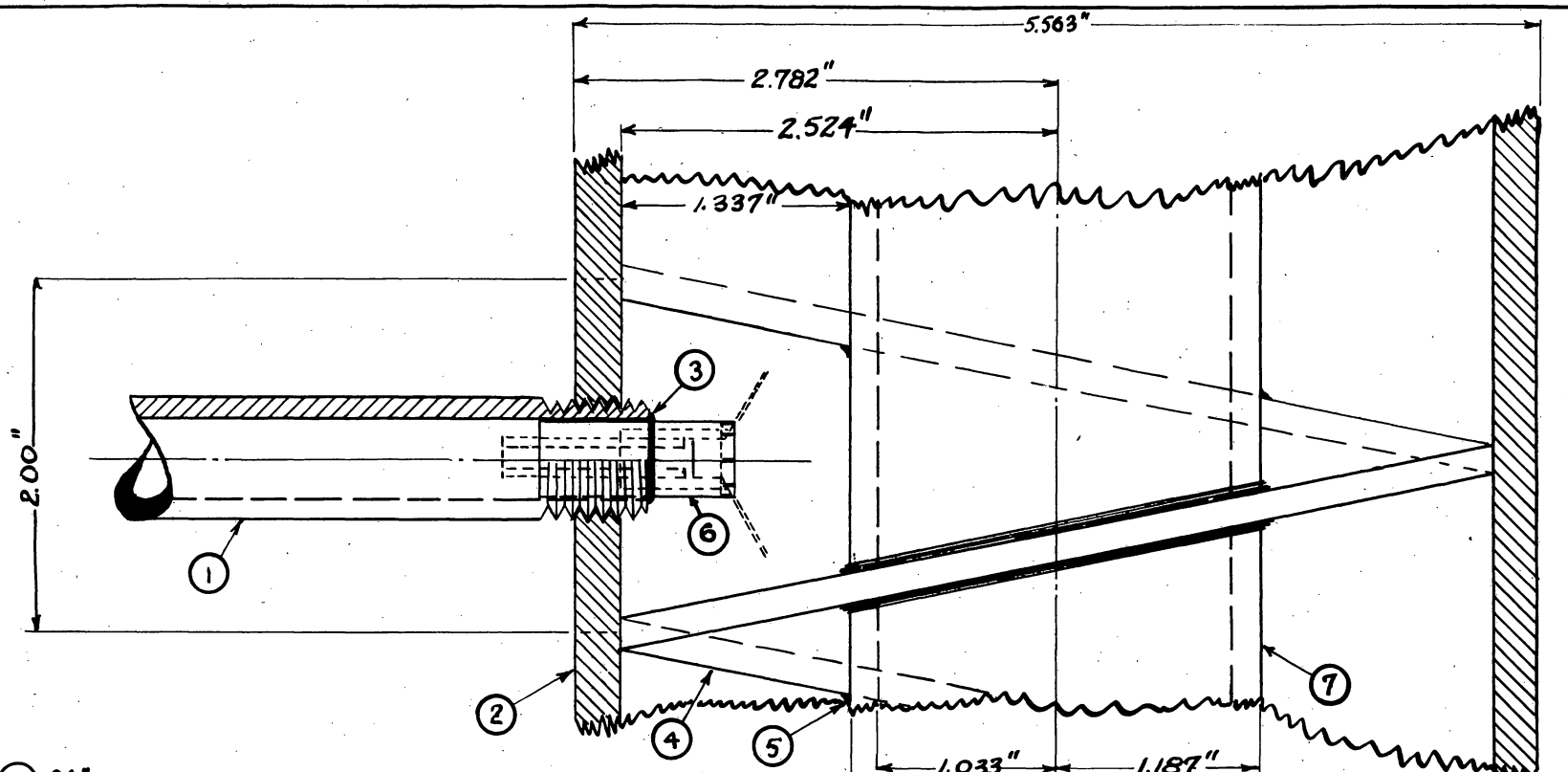
DETAIL OF
REFLUX DISPERSION NOZZLE

SCALE: 2" = 1"			CASE NO: 47
DRAWN BY: g.w.l.	DATE 5/17/47		FILE NO: 439
TRACED BY: g.w.l.	DATE 5/17/47		DR'W'G NO: 2
CHECKED BY: [illegible]	DATE [illegible]		
APPROVED BY: [illegible]	DATE 5/18/47		

dispersion nozzles employed in the column. The nozzles, constructed of brass, were designed for a maximum throughput of 0.15 gallons per minute under a head equivalent to 20 feet of water. Dispersion is accomplished through eight apertures spaced 45° apart around the periphery of the nozzle head, each aperture having an effective diameter of 0.018 inches. The conal dispersion angle of 120° is determined by the taper of the head end of the nozzle insert.

Six extract reflux dispersion nozzles were installed, one on each of the first six flights beginning at the base of the column. Drawing Number 3, page 56, illustrates the method of installation of a single nozzle in the column. The six nozzles are controlled independently by separate valves and connected to the extract reflux supply line through a header, the connection being made in such a manner that extract reflux may be introduced independently at any one of the six lowest flights in the column. Extract reflux is supplied through extract reflux rotameter (R_2) and regulating valve from extract reflux tank (T_3) (Drawing Number 1, page 53a) in order that a constant flow rate may be set and maintained at all times during operation of the equipment.

Feed Dispersion Nozzles. Details of the feed dispersion nozzles constructed of brass with steel inserts, are illustrated



1. 3/8" BLACK IRON PIPE - DRILLED 0.500" AT THREADED END TO RECEIVE NOZZLE
2. 5" STANDARD BLACK IRON PIPE
3. NOZZLE INSERTED & BRAZED TO 3/8" PIPE
4. HELICAL FLIGHTS 3/16" SAE 1020 STEEL
5. FLIGHTS WELDED TO CENTRAL 2" PIPE
6. NOZZLE ASSEMBLY - SEE DETAIL DRAWING 2
7. 2" STANDARD BLACK IRON PIPE

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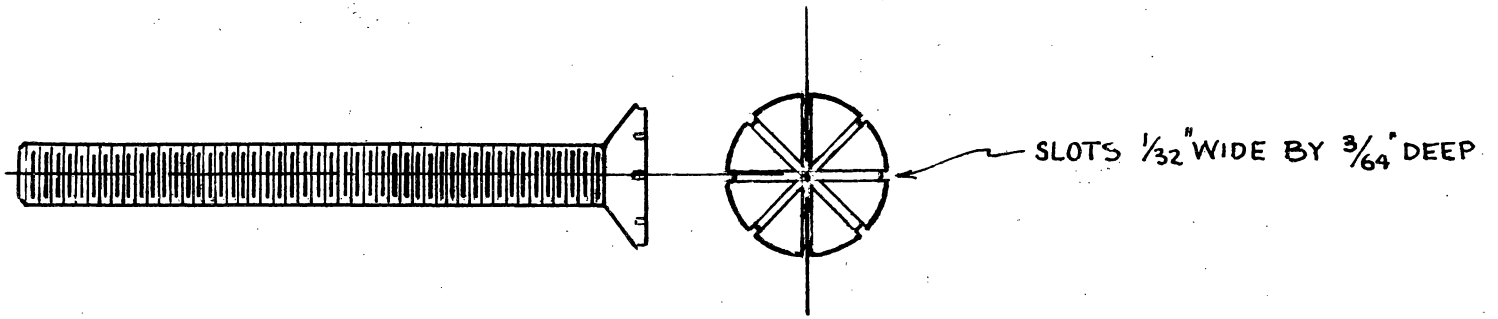
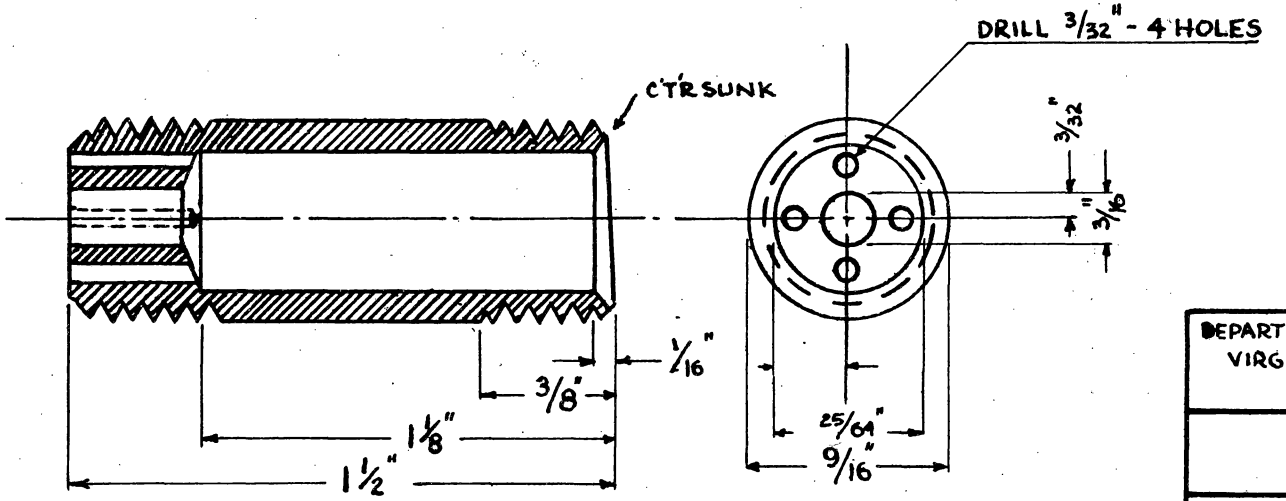
INSTALLATION OF NOZZLE ASSEMBLED.

SCALE: FULL SIZE	CASE NO.: 47
DRAWN BY: JWK DATE: 5/21/47	FILE NO.: 439
TRACED BY: JWK DATE: 5/21/47	DR'W'G NO.: 3
CHECKED BY: GHO DATE: 5/22/47	
APPROVED BY: [illegible] DATE: 5-22-47	



STANDARD 10-32 MACHINE SCREW - 1 1/2 INCHES LONG



NOZZLE SHANK

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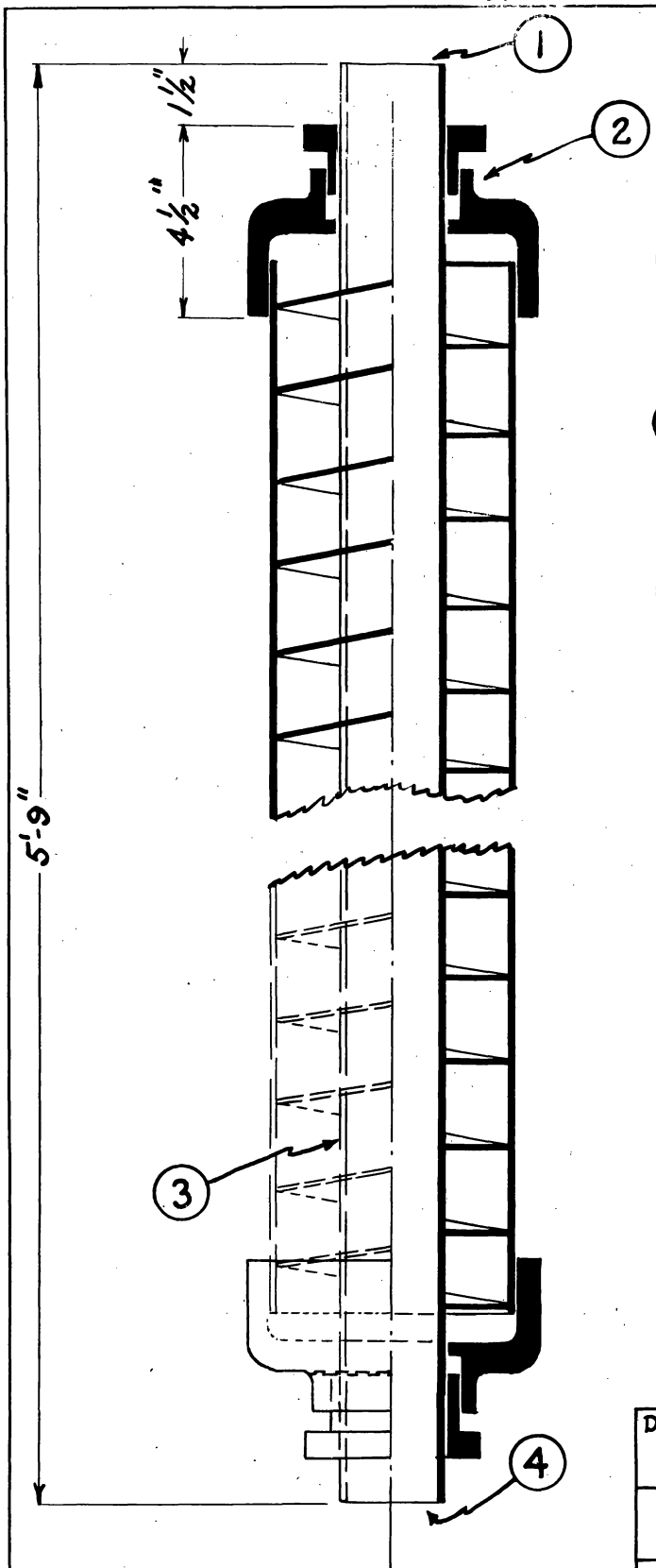
FEED NOZZLES
DETAILS

SCALE: 0'-2" = 0'-1"	CASE NO.: 539
DRAWN BY: J.W.L. DATE: 9/18/47	FILE NO.: 48
TRACED BY: J.W.L. DATE: 9/18/47	DRAWING NO.: 4
CHECKED BY: J.W.L. DATE: 9/18/47	
APPROVED: JWB DATE: 9/20/47	

in Drawing Number 4, page 57. The feed dispersion nozzles were designed with eight apertures having an effective diameter of 0.032 inches, with the angle of dispersion perpendicular to the direction of flow in the column; i.e. transverse to the helicoid flight. Installation of the feed dispersion nozzles is the same as that for the extract reflux dispersion nozzles except that the five feed nozzles are spaced two flights apart with one nozzle mounted at the center flight in the column, two nozzles spaced two flight apart above the center flight and two below it. Each feed nozzle is controlled by a separate valve which is connected through a header to the feed supply line, the connection being made in such a manner that feed may be introduced independently to any one of the five feed nozzles. Feed is supplied at the header from a constant head feed tank (T_1), (Drawing Number 1, page 53a) through the feed rotameter (R_2) and regulating valve.

Water Jacket - Temperature Control. It was realised early during this investigation that a means of temperature control must be supplied in order that definite equilibrium conditions might be attained, and in order that variations in the temperature of the system might be minimised. Accurate control of temperature is essential in that interpretations of refractive index readings are significant only at the temperature for which the equilibrium conditions are determined. Variation of temperature



(1) COOLING WATER EXIT, ST'D 2" B.I. PIPE FORMING CORE FOR HELICOID ACTS ALSO AS COOLING LINE.

(2) PACKING GLAND, TO PREVENT FLUID LEAKAGE ARDUND CENTRAL PIPE.

(3) CENTRAL PIPE, ST'D 2" B.I. PIPE COOLING WATER INSIDE ANILINE OUTSIDE.

(4) COOLING WATER IN

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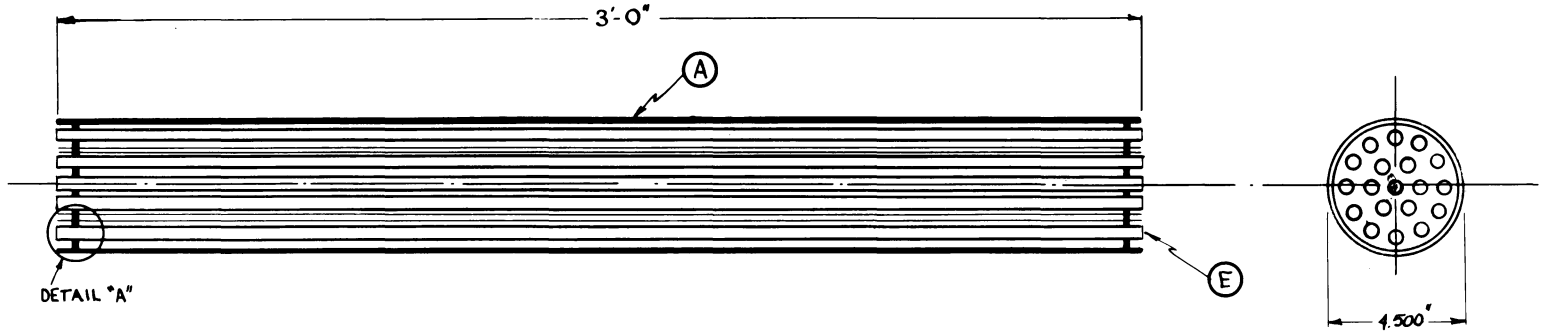
EXTRACTOR
COOLING SYSTEM

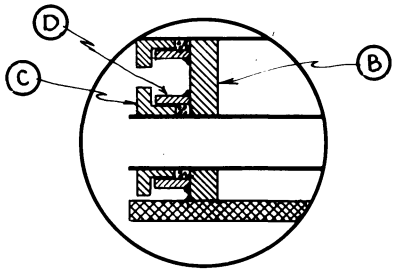
SCALE: 0'-3" = 1'-0"	CASE NO.: 539
DRAWN BY: JWR DATE: 12/17/47	FILE NO.: 48
TRACED BY: JWR DATE: 12/17/47	DRAWING NO.: 5
CHECKED BY: DATE:	
APPROVED BY: DATE: 5/28/48	

results in a change of equilibrium concentrations and introduces errors which may not easily be compensated for. The central 2-inch pipe, about which the helicoid is wrapped, provided an excellent means for temperature control. With cooling water available at a relatively constant temperature of $11^{o}C \pm 2^{o}C$ and a prevailing room temperature of between $25^{o}C$ and $30^{o}C$ sufficient heat exchange area is provided by the central pipe to maintain a temperature of $20^{o}C$ throughout the system.

The actual construction details of the water jacket and cooling system are illustrated in Drawing Number 5, page 59. To each end of the central 2-inch pipe, a 2-inch by 1 1/4-inch reducer, and a 1 1/4-inch by 3/4-inch reducing elbow were attached, and the 3/4-inch opening bushed to 1/2-inch. The 1/2-inch opening at the base of the column was then connected to the cold water supply line through a union and a standard 1/2-inch gate valve. The top or cooling water discharge end of the water jacketing system was likewise bushed to 1/2-inch and a cooling water discharge line was installed to provide overflow to the open floor drain on the ground level.

Continuous Stripping Still. In order to operate the extractor on a continuous basis with the limited amounts of materials available, it was found necessary to provide a means for continuous separation of solvent and extract, with a portion of the extract being employed as extract reflux and the





DETAIL "A" - TUBE PACKING JOINTS
SCALE: 0'-8" = 1'-0"

• BILL OF MATERIALS •

No.	QUAN.	DESCRIPTION
A	1	3' SECTION ST'D 4" B.I. PIPE
B	2	4" DIAM. X ¼" TUBE SHEETS
C	38	⅜" X ⅛" BUSHINGS
D	38	⅜" B.I. COUPLINGS
E	19	⅜" DIAM. 20 BWG Cu TUBES 3' LONG

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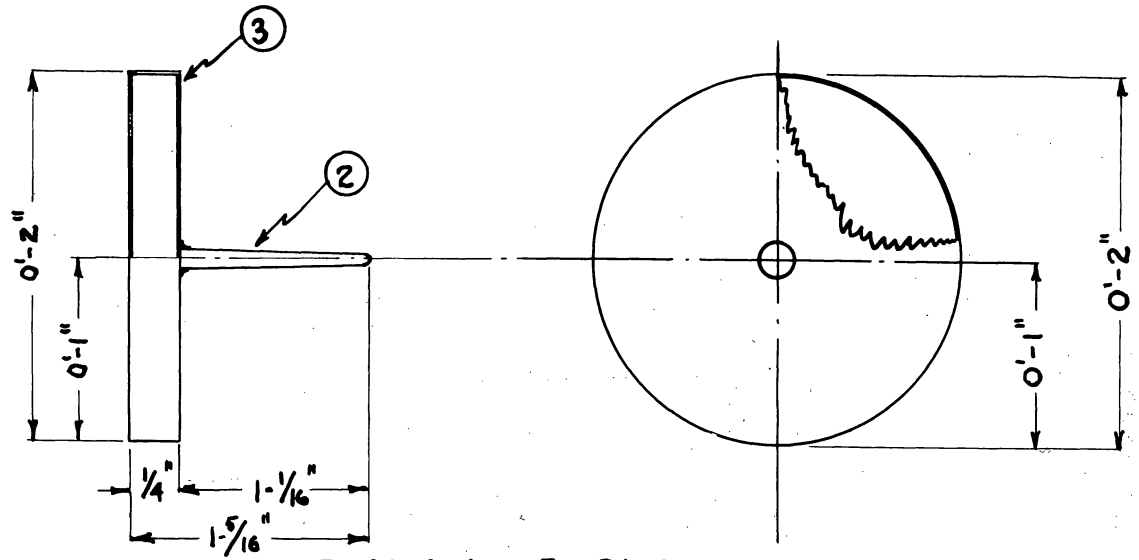
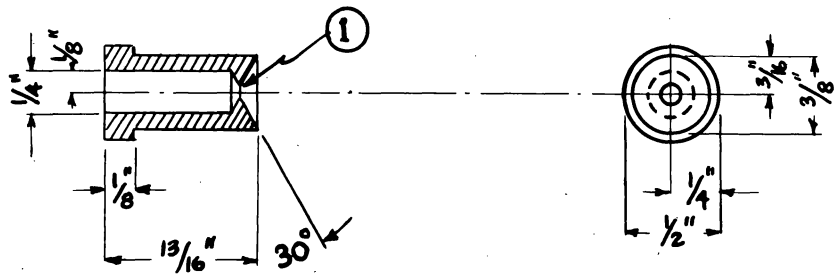
CONTINUOUS STRIPPING STILL
TUBE BUNDLE ASSEMBLY

SCALE: 0'-2" = 1'-0"
DRAWN BY: J.W.L. DATE: 10/20/47
TRACED BY: J.W.L. DATE: 10/26/47
CHECKED BY: DATE:
APPROVED BY: DATE: 10/30/47

CASE NO.: 529-39
FILE NO.: 48
DRAWING NO.: 6



FLOAT ASSEMBLY



VALVE BODY

NOTES

1. VALVE OPENING MACHINED TO 3/32" DIAM. – VALVE BODY MACHINED FROM 1/2" DIAM. BRASS ROD
2. TAPER PIN 1 1/16" LONG SOLDERED TO FLOAT 1/8" DIAM AT BASE 1/16" DIAM AT TIP.
3. HOLLOW BRASS CYLINDER, SOLDERED CONSTRUCTION; MAT'L 0.008" BRASS.

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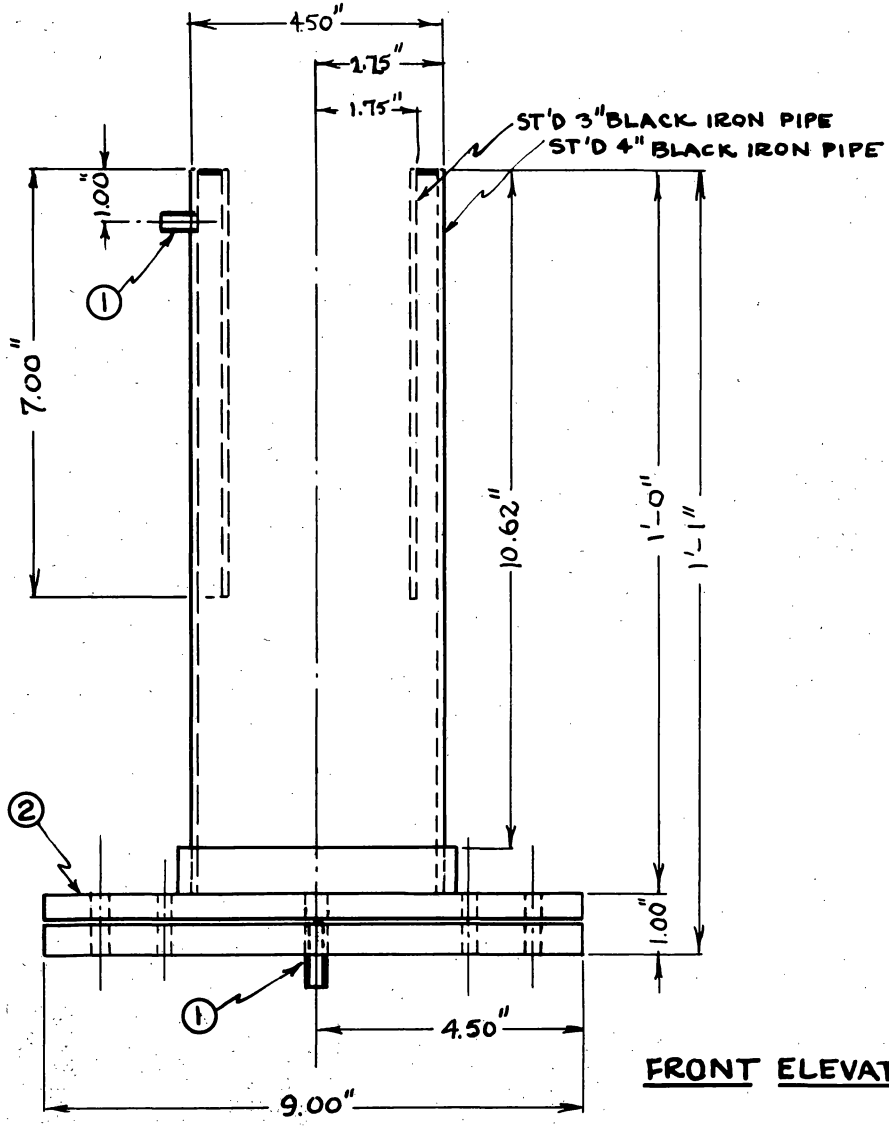
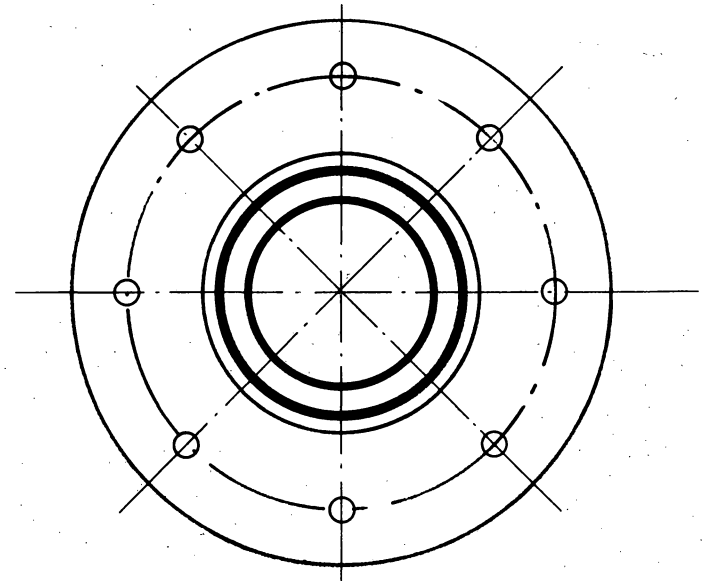
FLOAT CHECK VALVE FOR STRIPPING STILL

SCALE: FULL SIZE	CASE NO.: 539
DRAWN BY: J.W.R. DATE: 12/16/47	FILE NO.: 48
TRACED BY: J.W.R. DATE: 12/16/47	DRAWING NO.: 7
CHECKED BY: DATE:	
APPROVED BY: J.W.B. DATE: 1/16/48	



FRONT ELEVATION



TOP VIEW

① ST'D ½" COUPLING WELDED TO 4" BLACK IRON PIPE - VACUUM CONNECTION

② ST'D 4" CAST IRON FLANGES, SCREWED FIT COMPANION FLANGE & BLIND FLANGE

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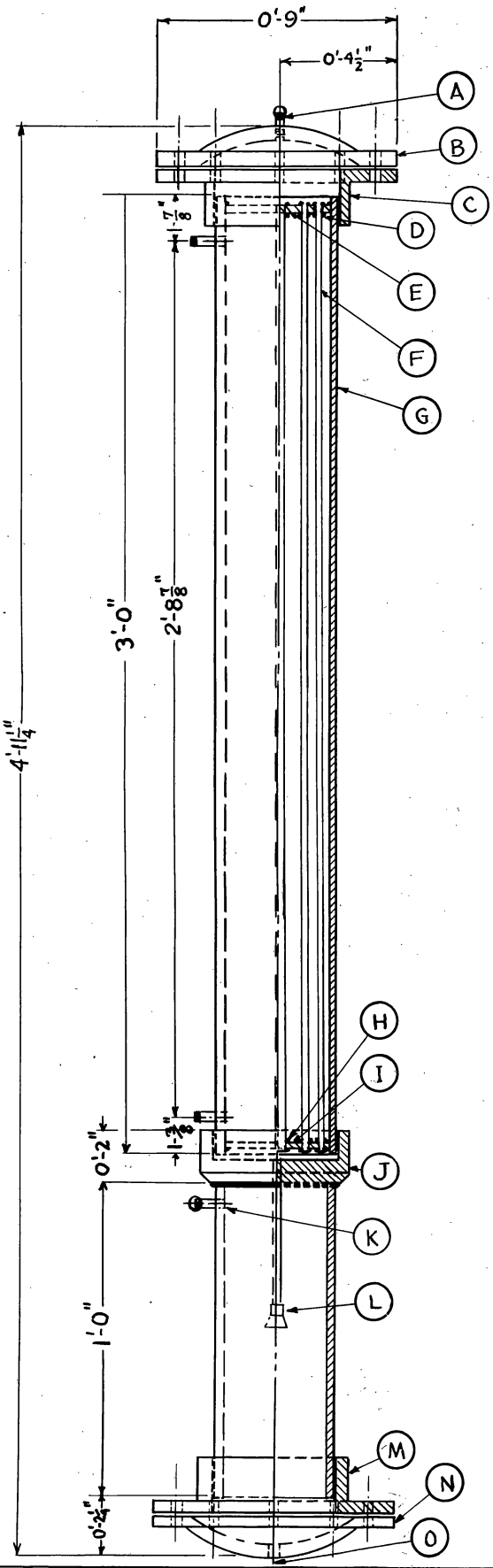
CONTINUOUS STRIPPING STILL
FLASH CHAMBER

SCALE: 0'-4" = 1'-0"
DRAWN BY: JWR DATE: 11/15/47
TRACED BY: JWR. DATE: 11/15/47
CHECKED BY: DATE:
APPROVED BY: [signature] DATE: 11/[illegible]/47

CASE NO.: 529-39
FILE NO.: 48
DRAWING NO.: 8



• BILL OF MATERIALS •

No.	Quan.	DESCRIPTION
A	1	½" STREET ELBOW – LIQUID FEED INLET.
B	1	4" STANDARD, FLANGED CAP – HEADER
C	1	4" STANDARD PIPE FLANGE
D	19	¼" CAP & ¼" X ⅛" BUSHING – PACKING GLAND
E	1	UPPER TUBE SHEET – ¼" X 4" DIAM.
F	19	⅜"-20 BWG COPPER PIPES, 3' LONG
G	1	3' LENGTH – 4" STANDARD IRON PIPE
H	19	¼" CAP & ¼" X ⅛" BUSHING – PACKING GLAND
I	1	LOWER TUBE SHEET – ¼" D 4" DIAM.
J	1	4" THREADED PIPE CAP
K	1	½" STREET ELBOW – VACUUM LINE
L	1	LIQUID DISPERSION HEAD.
M	1	4" PIPE FLANGE – FLASH CHAMBER
N	1	4" FLANGED PIPE CAP – FLASH CHAMBER
O	1	½" STREET ELBOW – LIQUID REMOVAL (NOT SHOWN)

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CONTINUOUS SEPARATION STILL

SCALE: 0'-2" = 1'-0"
DRAWN BY: J.W.L. DATE: 9/30/41
TRACED BY: J.W.L. DATE: 9/30/41
CHECKED BY: DATE:
APPROVED BY: DATE: 9/30/41

CASE NO: 539
FILE NO: 48
DRAWING NO: 9

remaining extract product being recycled, along with the raffinate product as feed to the extractor. Drawing Numbers 6, 7 and 8, pages 61, 62, 63, illustrate the specific details of construction of the continuous stripping still (C); while Drawing Number 9, page 64, shows the still assembled, preparatory to operation.

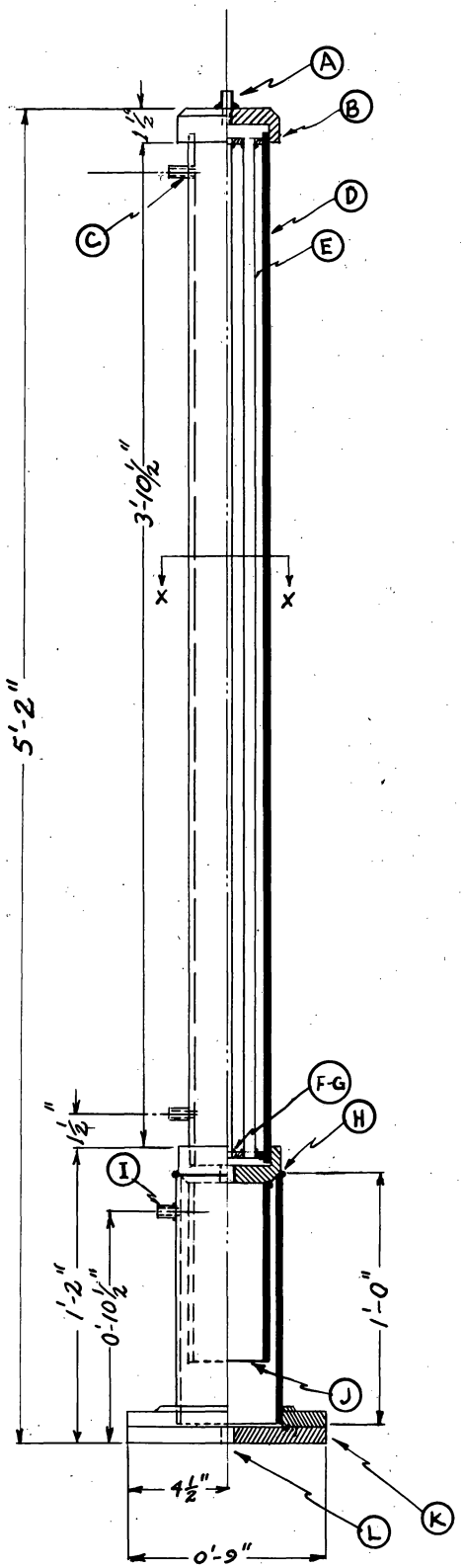
The steam chest, or shell of the still, consists of a 3-foot length of standard 4-inch black iron pipe, flanged at one end. Within this outer shell is inserted a tube bundle (Drawing Number 6, page 61) consisting of nineteen 3-foot lengths of 3/8-inch 20 BWG standard copper pipe with a wall thickness of 0.035 inches, and having a useful heat transfer area of 5.7 square feet, based on the inside diameter of the 3/8-inch copper pipe.

The head of the still consists of a standard 4-inch blind flange drilled and tapped to receive a standard 90° 1/2-inch street elbow through which the liquid mixture to be separated is introduced into the still. At the base of the still, the heated liquid mixture is passed through a spray nozzle (Drawing Number 7, page 62) into the flash chamber (D) which consists of a 12-inch length of 4-inch standard black iron pipe, flanged and closed at the base by a standard 4-inch blind flange. Provision for liquid removal from the flash chamber was made by drilling and tapping and blind flange to receive a standard 1/2-inch pipe. One inch below the welded connection of the standard 4-inch pipe, constituting the flash chamber, and the 4-inch cap, a second hole

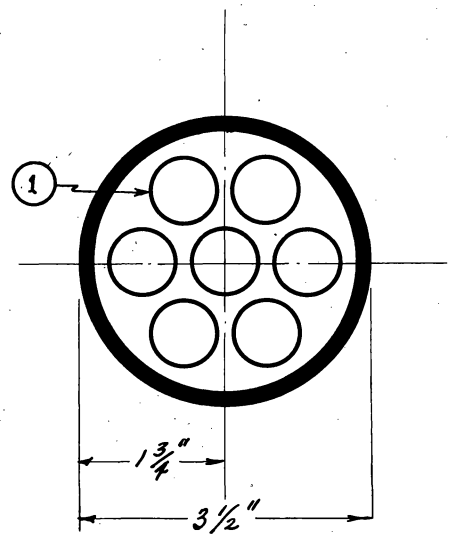
was drilled and tapped to receive a 1/2-inch pipe which was connected to the Stokes vacuum pump (P_6) through the methylcyclohexane condenser (A) and receiver (B). Details of the flash chamber assembly are shown in Drawing Number 8, page 63.

Methylcyclohexane Condenser. The methylcyclohexane condenser (A) consists of a 4-foot length of standard 3-inch galvanized iron pipe into which seven 4-foot lengths of 1/2-inch standard black iron pipes are enclosed as shown in Drawing Number 10, page 67. The total area thus available for heat transfer is 6.15 square feet based on the outside diameter of the standard 1/2-inch black iron pipe. The capacity of the exchanger is then sufficient to cause condensation of the methylcyclohexane vapors and subsequent cooling to 20°C with cooling water available at 13°C, flow of cooling water in the outer jacket of the condenser being countercurrent to the flow of vapor and condensate.

Since temperature conditions under which the condenser is to be operated are essentially constant, and since there is no differential coefficient of expansion between the condenser tubes and the outer shell, the tubes were welded to the tube sheets, and the entire assembly was welded into the outer condenser jacket. The receiver (B), consisting of a 12-inch length of 4-inch standard black iron pipe, was connected to the lower end of the condenser through a 3-inch screwed cap brazed to the



FRONT ELEVATION



SECTION VIEW X-X
SCALE ½" = 1"

(1) ALL TUBES SPACED 15/16" BETWEEN CENTERS.

BILL of MATERIALS ~ NOTES

No.	Quan	Description:
A	1	½" COUPLING WELDED TO CAP, VAPOR INLET
B	2	ST'D 3" PIPE CAPS
C	2	½" COUPLING WELDED TO PIPE, COOLING WATER
D	1	4' LENGTH STD 3" B.I. PIPE, COND. SHELL
E	7	4' LENGTHS - ST'D ½" B.I. PIPE, COND. TUBES
F	—	½" CONDENSER TUBES WELDED TO TUBE SHEETS
G	2	3" DIAM x ¼" STEEL TUBE SHEETS
H	—	WELDED CONSTRUCTION
I	1	½" COUPLING WELDED TO RECEIVER — VACUUM
J	1	9" LENGTH ST'D 3" PIPE, VAPOR BAFFLE
K	1	ST'D 4" BLIND & COMPANION FLANGE ASSEM.
L	1	DRILLED & TAPPED ½"-14 NPT, PRODUCT OUTLET

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METHYLCYCLOHEXANE CONDENSER

SCALE: 0'- ⅛" = 0'-1"
DRAWN BY: g.w.f. DATE: 12/16/47
TRACED BY: g.w.f. DATE: 12/15/47
CHECKED BY: DATE:
APPROVED BY: J.W.B. DATE: 5/28/48

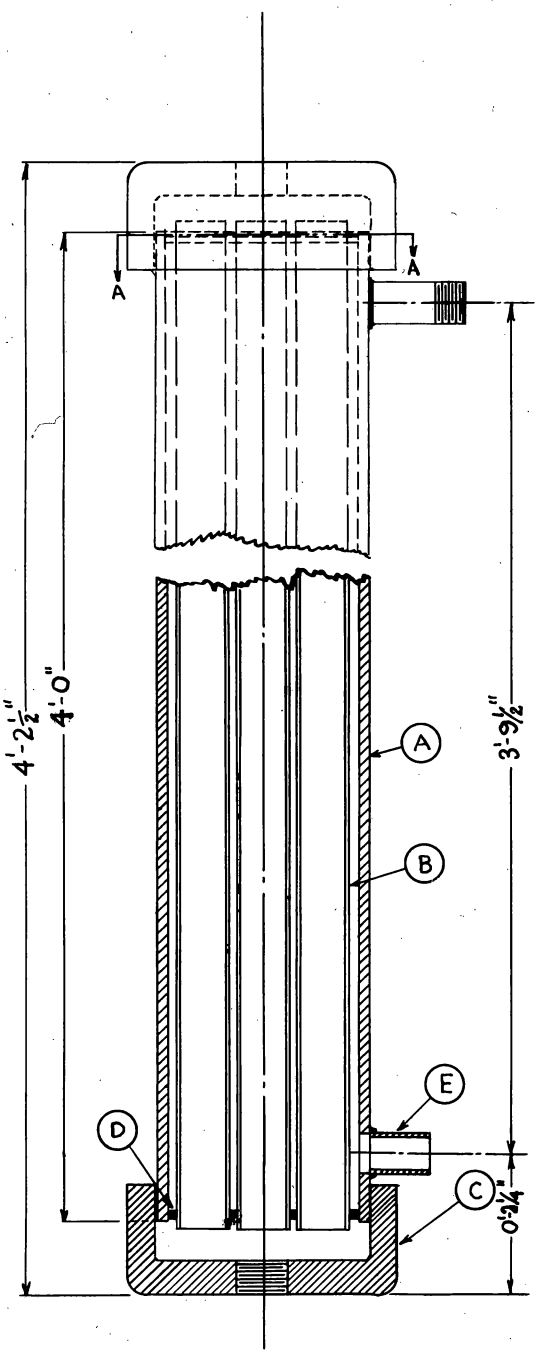
CASE NO.: 539
FILE NO.: 48
DRAWING NO.: 10

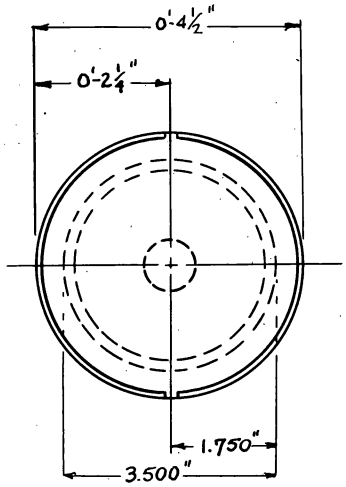
4-inch section, with a 3/4-inch pipe fitted to the cap to provide for condensate discharge and to act as a liquid entrainment separator. The lower end of the receiver (B) was fitted with a 4-inch pipe flange and sealed with a standard 4-inch blind flange.

Through the center of the blind flange, a hole was drilled and tapped to take a standard 1/2-inch street elbow to provide for methylcyclohexane removal and recycling to the reflux and feed tanks. One inch below the coupling to the receiver (12-inch length of 4-inch standard black iron pipe) a hole was drilled and tapped to take a standard 1/2-inch pipe which was connected through a 1/2-inch gate valve to the Stokes vacuum pump (P_6).

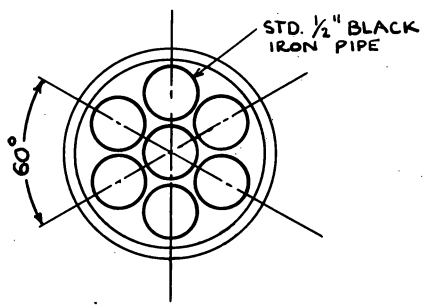
Solvent Cooler. Solvent aniline leaves the continuous separation still at a temperature of between 150°F and 160°F and must be cooled to 68°F (20°C) before recycling to the system. For this purpose, a solvent cooler (F) (Drawing Number 11, page 69) was constructed and installed between the continuous stripping still (C) and the solvent reservoir (T_6). Structural features for the solvent cooler are identical with those for the methylcyclohexane condenser.

The cooler shell consists of a 4-foot length of 3-inch standard galvanized iron pipe into which is inserted a tube bundle consisting of seven 4-foot lengths of 1/2-inch standard





TOP VIEW



SECTION A-A

•BILL OF MATERIALS•

No.	Quan.	Description
A	1	4' LENGTH STD. 3" BLACK IRON PIPE
B	7	4' LENGTH STD. ½" BLACK IRON PIPE
C	2	STD. 3" THREADED PIPE CAP.
D	2	¼" THICK BY 3" DIAM. STEEL PLATE
E	2	½" PIPE WELDED TO SHELL

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SOLVENT COOLER

DRAWN BY: J.W.L. DATE: 10/1/47
TRACED BY: J.W.L. DATE: 10/1/47
CHECKED BY: DATE:
APPROVED BY: J.W.B DATE: 10-7-47
SCALE: 0'-4" = 1'-0"

CASE NO. 539
FILE NO. 48
DRAWING NO. 11

black iron pipe. The tube bundle is of brazed construction and is in turn brazed to the outer shell. Both ends of the cooler are fitted with standard 3-inch black iron caps with a central hole drilled and tapped in each to receive standard 1/2-inch street elbows.

Solvent Tempering Coil. In addition to the solvent cooler (F) it was found necessary to include a solvent tempering coil (G) in order that exact temperature conditions might be maintained in the solvent reservoir to provide for saturation of the solvent with raffinate reflux. For the tempering coil a 20-foot length of 3/4-inch copper pipe was installed in the solvent reservoir (T_6), and was connected to the cold water supply line through a standard 1/2-inch galvanized iron pipe and a 1/2-inch needle valve to provide for adjustment of cooling water flow. The cooling water discharge line consists of a standard 1/2-inch galvanized iron pipe and exit cooling water discharges directly to the open floor drain.

Raffinate Reflux and Product Recovery System. To provide for continuous cycling of the raffinate product and reflux, a weir was installed in the raffinate product and distribution tank (T_5) (Drawing Number 1, page 53a) and an outlet from the weir pipe was provided for the continuous withdrawl of the raffinate product for recycling to the feed tank (T_2) by the raffinate feed make-up pump (P_5). The remaining raffinate

reflux then flows by gravity into the solvent reservoir (T_6) through the raffinate reflux regulating valve (V_{14}) and return line.

Extract Reflux and Product Recovery System. Extract product and reflux from the methylcyclohexane condenser is pumped to the methylcyclohexane distribution tank (T_4) (Drawing Number 1, page 53a), and the extract product is withdrawn from an outlet in the base of the distributing tank through methylcyclohexane feed make-up rotameter (A_7) and regulating valve and is discharged into the feed tank. The extract reflux flows through an overflow weir installed in the bottom of the distribution tank into the extract reflux tank (T_3) immediately below it.

F. Methods of Procedure

The following methods of procedure were employed in making all determinations for this investigation.

Determination of Saturation and Equilibrium Data. The method of Vertzeanian and Fenske [44], used in obtaining saturation and equilibrium data for the system: methylcyclohexane - aniline - n-heptane, is outlined as follows:

1. Various mixtures of methylcyclohexane and n-heptane of known weight composition were titrated at 20°C

in a water bath into measured quantities of aniline just to the point of turbidity. The compositions of the resulting solutions were calculated, determining the saturation compositions of the hydrocarbon mixtures in the aniline phase. Refractive indices and densities of these solutions were obtained at 20°C.

2. Aniline was then titrated into mixtures of methylcyclohexane and n-heptane of known composition in a water bath at 20°C, again just to the point of turbidity. The compositions of the resulting solutions were calculated, determining the saturation compositions of aniline in the hydrocarbon phase. Refractive indices and densities of these solutions were obtained at 20°C.

3. In obtaining equilibrium data for the system, various mixtures of methylcyclohexane and n-heptane of known composition were added to approximately equal weights of aniline. The resulting mixtures were agitated and placed in a water bath maintained at a constant temperature of 20°C ± 0.1°C.

4. The liquid mixtures in the constant temperature bath were agitated hourly, for 24 hours, at the end of which time the refractive indices of the separated

aniline and hydrocarbon phases were obtained.

5. The compositions of these equilibrium layers were then obtained directly from a plot of refractive index versus saturated composition, Figure 7, page 83 which had previously been determined from the saturation data.

The results of these determinations are included in TABLES III and IV, pages 81 and 82 and are graphically represented in Figures 7, 8 and 9, pages 83, 84 and 85.

Rotameter Calibration. In order to determine accurately the rates of flow of liquids in the extractor, the rotameters were calibrated with liquids of known specific gravity. The method of calibration is as follows:

1. The rotameters used to measure the flow rates of the hydrocarbon phase liquids were calibrated at flow rates of 0.01, 0.02, 0.04, 0.06, 0.08 and 0.10 gallons per minute with a liquid mixture made up of the components of the system used. Liquid specific gravities of 0.72, 0.74 and 0.76 were used for purposes of calibration. Calibration was accomplished by allowing the liquid mixture to flow through the rotameter at a constant rate adjusted at the rotameter, and collecting and weighing the liquid passing through the rotameter during a period of one minute.

2. Rotameters used to measure the flow rates of the solvent phase liquids were calibrated at flow rates of 0.1, 0.2, 0.4, 0.6, 0.8 and 1.0 gallons per minute. Each rotameter was calibrated with a liquid mixture made up of the components of the system to a specific gravity of 0.90. Calibration was accomplished in the same manner as for the rotameters used to measure hydrocarbon flow rates.

The results of the calibrations are included in TABLES V, VI and VII, pages 86, 88 and 90 and are represented graphically in Figures 10, 11 and 12, pages 87, 89 and 91.

Operating Procedure - Apparatus. Referring to Drawing Number 1, page 53a, the following procedure was observed in the operation of the equipment during experimental tests:

1. Fifty pounds of a feed composed of 46.2 per cent methylcyclohexane, 46.2 per cent n-heptane and 7.6 per cent aniline by weight was charged to the feed tank (T_1).
2. Sixty pounds of a solution composed of 89.6 per cent methylcyclohexane and 10.4 per cent aniline by weight was charged to the extract reflux tank (T_3) to serve as extract reflux until flow rates in the system were established.

3. Three hundred pounds of a solution composed of 94.6 per cent aniline and 5.4 per cent n-heptane was charged to the solvent reservoir (T_1) and circulated to the solvent supply tank (T_2) by pump (P_1).

4. Tempering coil valve (V_{w2}) was opened, and cooling water was circulated in the solvent tempering coil (G) until the solvent temperature had been reduced to 20°C.

5. Solvent was then introduced into the extractor (E) through solvent rotameter (R_3) by opening solvent and solvent control valves (V_1) and (V_2).

6. After the extractor had been filled with solvent, as indicated by the liquid level in sight glass (S), cooling water was allowed to flow through the methylcyclohexane condenser (A) the solvent cooler (F) and the extractor (E) by opening valves (V_{w1}) (V_{w3}) and (V_{w4}). Steam at 35 pounds pressure was admitted to the heat exchanger section of the continuous stripping still (C) by adjusting valve (V_s).

7. Recovered solvent pump (P_3), recovered methylcyclohexane pump (P_4), and vacuum pump (P_6) were then turned on and recovered solvent valve (V_{16}) and recovered methylcyclohexane valve (V_{15}) were opened to provide

for removal of recovered liquids from the continuous stripping still flash chamber (C) to the solvent reservoir (T_6) through solvent cooler (F) and from the methylcyclohexane receiver (E) to the methylcyclohexane distribution tank (T_4).

8. Extract product pump (P_2) was then started and extract product and control valves (V_3) and (V_4) were opened to allow the extract product to flow through the extract product rotameter (R_1) and into the continuous stripping still (C).

9. Extract reflux was next allowed to flow into the extractor through the extract reflux rotameter (R_2) and reflux inlet valve (V_{r1}) by opening extract reflux and control valves (V_5) (V_6).

10. Feed was admitted to the extractor through the feed rotameter (R_3) and feed inlet valve (V_{f1}) by opening feed and control valves (V_7) and (V_8).

11. Raffinate product was then removed from the column to the raffinate product and distribution tank (T_5) through sightglass (G), and raffinate rotameter (R_4) by opening raffinate and control valves (V_9) and (V_{10}).

12. Flow rates through the extractor were then adjusted to the amounts previously determined by graphical calculation and material balance. Reflux to the extractor

was taken as 2.5 times the minimum based on the extract product. Refractometer scale readings as determined from Figures 10, 11 and 12, pages 87 89 and 91 were as follows in TABLE II on page 79.

13. During operation, samples were taken of the extract and raffinate products at 10 minute intervals after the start of the equilibrium run, until the compositions remained unchanged as determined by measurement of the refractive index at 20°C.

14. Sampling for all subsequent equilibrium determinations was made at five minute intervals.

15. The extract reflux input position was varied for each series of determinations with the same feed input position.

16. The procedure of step 15 was repeated for a total of five feed input positions.

Rotameter Scale Readings for Operation of the Countercurrent Liquid-Liquid Extractor Employing the System Methylcyclohexane - Aniline - n-Heptane at 25°C and 1 Atmosphere.

TABLE II

Rotameter	Scale Reading gal/min	Throughput lbs/min
Extract Product (R_1)	0.333	2.359
Extract Reflux (R_2)	0.030	0.233
Solvent (R_3)	0.330	2.1675
Raffinate Product (R_4)	0.027	0.214
Feed (R_5)	0.020	0.166
Methylcyclohexane Feed Makeup (R_6)	0.008	0.0846
Raffinate Feed Makeup (R_7)	0.009	0.0821

C. Data and Results

The following is a listing of tables and figures appearing in this section:

1. TABLE III and TABLE IV, pages 81 and 82, show the saturation and equilibrium data and specific gravities of saturated mixtures for the system: methylcyclohexane - aniline - n-heptane at 20°C. Graphical representations of these data appear in Figures 7, 8 and 9, pages 83, 84 and 85.
2. TABLES V, VI and VII, pages 86, 88 and 90, show the results of calibrating the rotameters employed for the measurement of flow rates during the operation of the spiral-channel liquid-liquid extractor. These data are plotted in Figures 10, 11 and 12, pages 87, 89 and 91.
3. TABLES VIII, IX, X, XI and XII, pages 92, 93, 94, 95 and 96, show the product compositions obtained during the operation of the extractor at the feed and extract reflux input positions indicated.
4. TABLE XIII, page 97 and Figure 13, page 98, show the relationship between the extract product composition obtained and the number of flights in the rectifying section of the extractor.

5. TABLE XIV, page 99, and Figure 14, page 100, show the relationship between the raffinate product composition and the number of theoretical flights in the stripping section of the extractor.
6. Figure 15, page 101 and Figure 16, page 102 show the results of TABLE XIII and TABLE XIV plotted against the total number of flights in the rectifying and stripping sections of the extractor.
7. TABLE XV, page 103, shows the number of calculated theoretical plates for the entire extractor and for the stripping section for each condition of feed and extract reflux input positions employed.
8. TABLE XVI, page 104, and Figure 17, page 105 show the results of a frequency distribution analysis for the theoretical plate equivalent of one theoretical flight in the stripping section of the extractor.
9. TABLE XVII, page 106 and Figure 18, page 107, show the results of a frequency distribution analysis for the theoretical plate equivalent of one actual flight in the rectifying section of the extractor.

TABLE III

SATURATION DATA FOR THE SYSTEM: METHYLCYCLOHEXANE - ANILINE - n-HEPTANE AT 20°C AND 1 ATMOSPHERE

Hydrocarbon Layer			
Me-cyclohexane Weight %	n-Heptane Weight %	Refractive Index n_D^{20}	Density d_4^{20}
0.00	93.94	1.3957	0.698
10.46	83.36	1.3974	0.706
20.56	72.88	1.4035	0.715
30.38	62.62	1.4071	0.724
39.80	52.93	1.4110	0.732
49.09	43.41	1.4150	0.742
57.06	33.95	1.4190	0.753
66.09	25.02	1.4230	0.762
74.26	16.42	1.4272	0.771
82.24	8.10	1.4313	0.782
89.64	0.00	1.4350	0.790
Aniline Layer			
Me-cyclohexane Weight %	n-Heptane Weight %	Refractive Index n_D^{20}	Density d_4^{20}
0.00	5.36	1.5719	0.991
1.41	4.71	1.5707	0.991
2.79	4.34	1.5690	0.990
4.15	3.99	1.5670	0.987
5.49	3.40	1.5656	0.985
6.80	2.95	1.5644	0.983
8.08	2.05	1.5635	0.982
10.50	1.44	1.5611	0.979
11.79	1.16	1.5606	0.975
13.00	0.63	1.5585	0.973
14.20	0.00	1.5570	0.971

TABLE IV

EQUILIBRIUM DATA FOR THE SYSTEM: METHYLCYCLOHEXANE - ANILINE - n-HEPTANE AT 20°C AND 1 ATMOSPHERE

Hydrocarbon Layer			Aniline Layer		
Methylcyclohexane Weight %	n-Heptane Weight %	Refractive Index n_D^{20}	Methylcyclohexane Weight %	n-Heptane Weight %	Refractive Index n_D^{20}
0.0	93.94	1.3957	0.0	5.36	1.5719
7.8	85.8	1.3984	1.9	4.8	1.5699
18.6	75.0	1.4026	3.4	4.2	1.5681
27.2	65.9	1.4060	5.1	3.6	1.5663
37.1	55.8	1.4100	6.1	3.2	1.5652
47.0	45.2	1.4141	7.9	2.5	1.5635
54.9	37.0	1.4176	9.0	2.1	1.5623
63.3	28.1	1.4215	10.1	1.8	1.5612
72.7	18.4	1.4261	12.3	0.9	1.5591
81.5	9.4	1.4307	13.7	0.3	1.5578
89.64	0.0	1.4350	14.28	0.0	1.5570

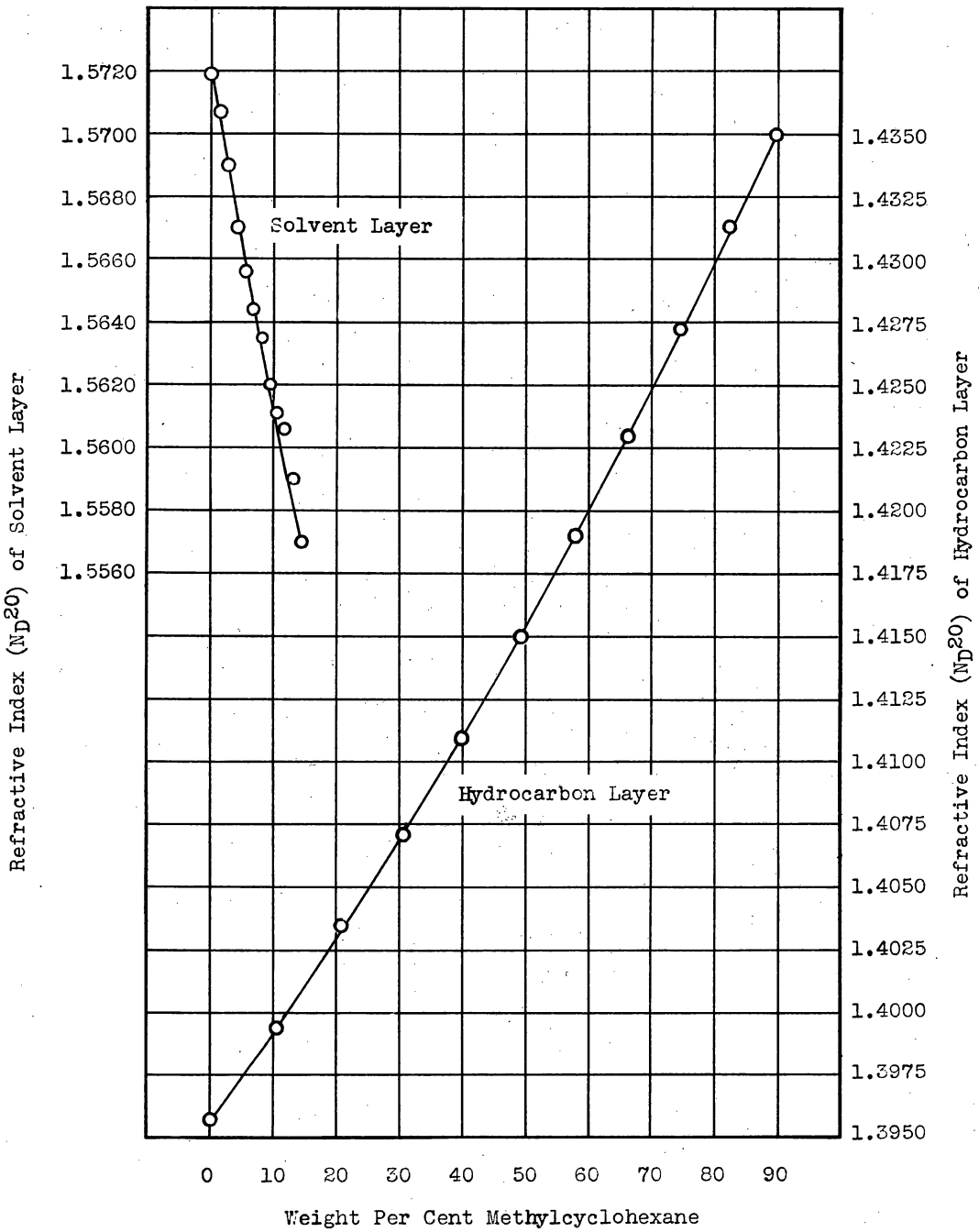


Figure 7. Weight Per Cent Methylcyclohexane in Saturated Phases Versus Refractive Indices (N_D) for the System: Methylcyclohexane - Aniline - n-Heptane at 20°C and 1 Atmosphere

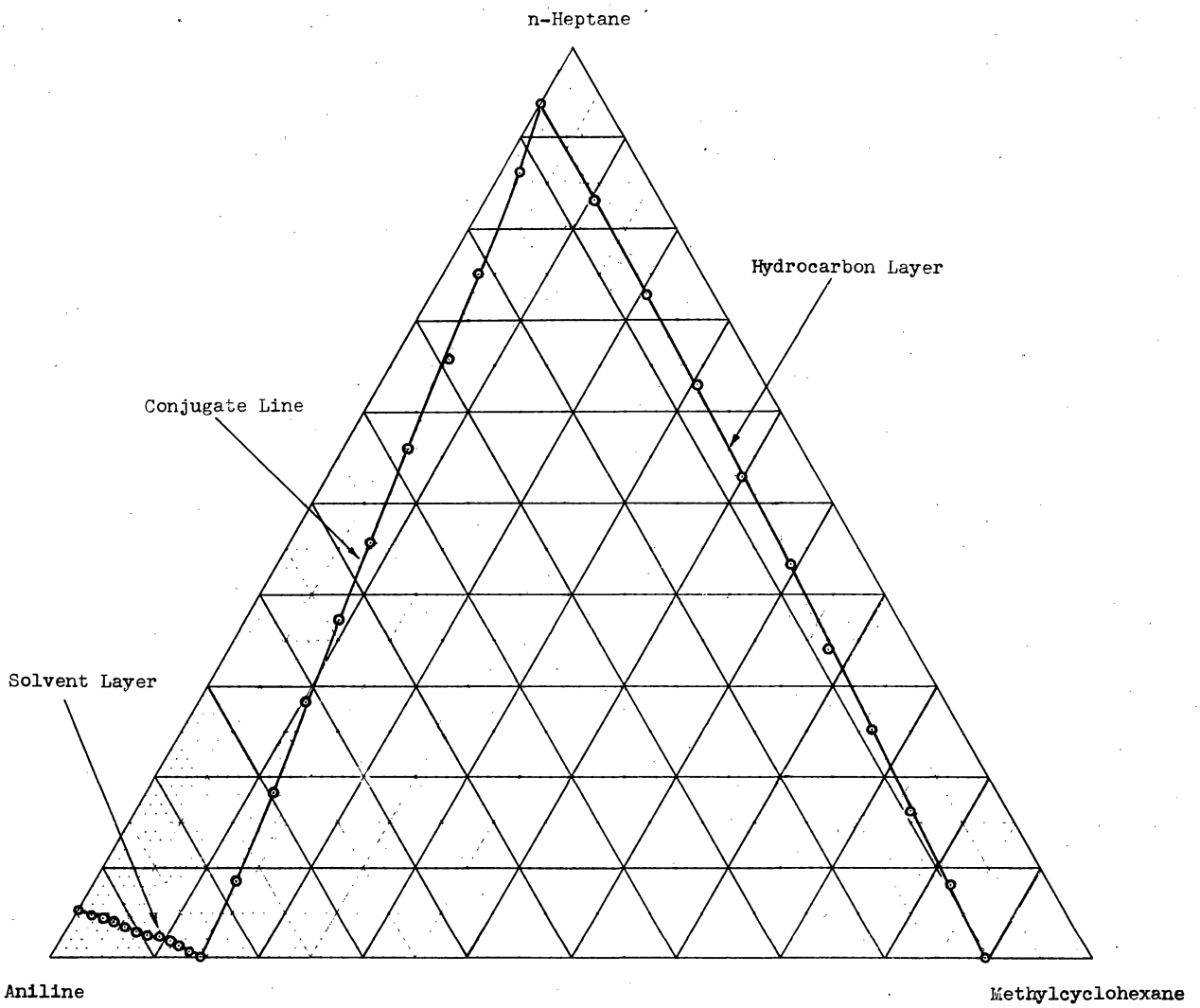


Figure 8. The System: Methylcyclohexane - Aniline - n-Heptane at 20°C and 1 Atmosphere.

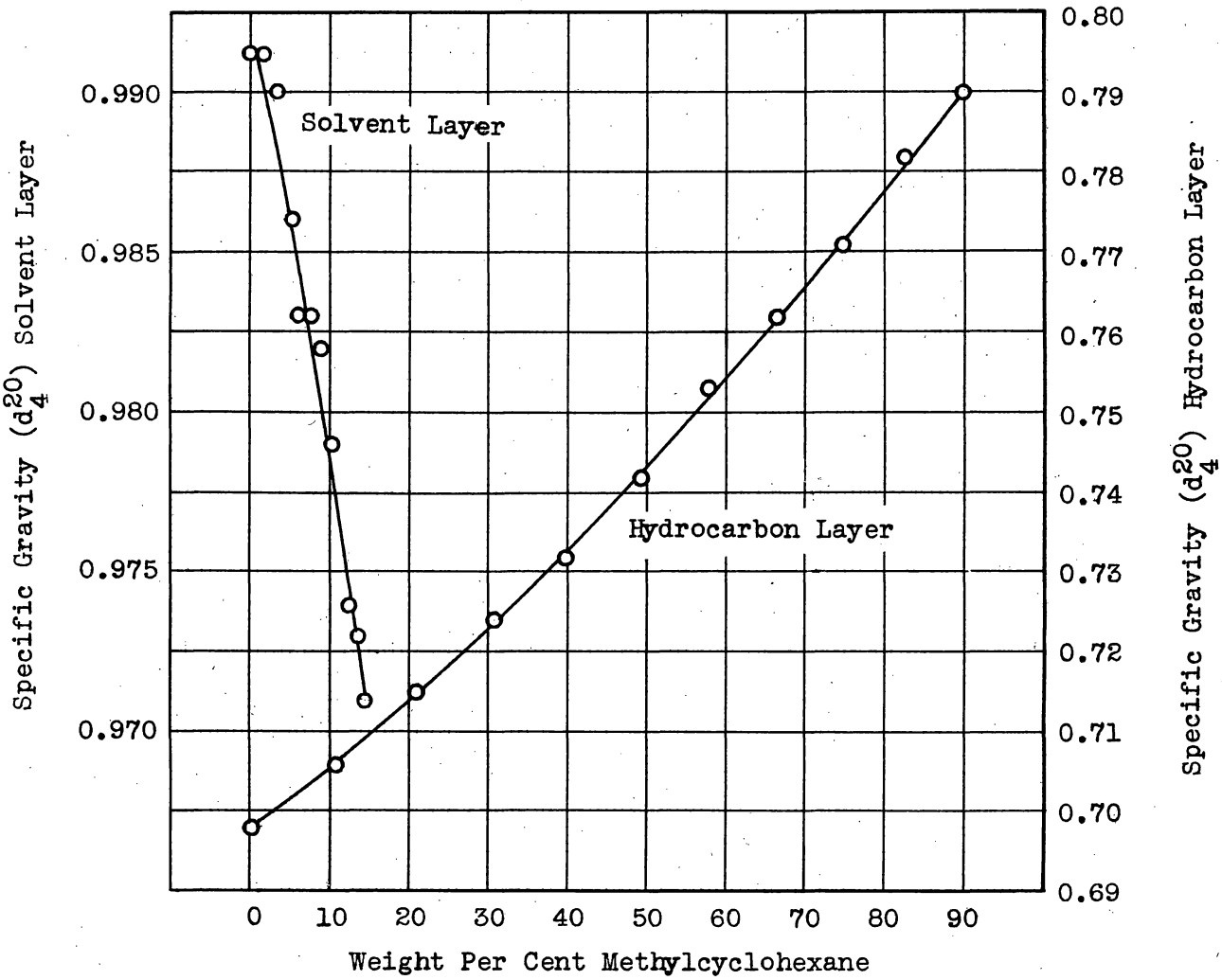


Figure 9. Weight Per Cent Methylcyclohexane in Saturated Phases Versus Specific Gravity (d_4^{20}) for the System: Methylcyclohexane - Aniline - n-Heptane at 20°C and 1 Atmosphere

TABLE V*

CALIBRATION DATA FOR FEED, EXTRACT REFLUX
AND METHYLCYCLOHEXANE MAKE-UP ROTAMETERS

Rotameter Reading (gal/min)	Volume per Minute (cc)			Specific Gravity d_4^{20}	Flow Rate (cm/min)			Flow Rate (lbm/min)		
	(1)	(2)	(3)		(1)	(2)	(3)	(1)	(2)	(3)
0.00	0.0	0.0	0.0	-	0.0	0.0	0.0	0.000	0.000	0.000
0.02	105.0	105.0	100.0	0.76	79.8	79.8	76.0	0.176	0.176	0.168
0.04	190.0	185.0	185.0	0.76	144.4	140.6	140.6	0.318	0.311	0.311
0.06	282.0	275.0	267.0	0.76	214.3	209.0	202.8	0.473	0.461	0.447
0.08	350.0	355.0	367.0	0.76	266.1	269.8	279.0	0.587	0.596	0.615
0.10	447.0	440.0	430.0	0.76	340.2	334.4	326.7	0.750	0.739	0.721
0.12	505.0	510.0	520.0	0.76	383.5	387.6	395.2	0.845	0.856	0.871

(1) Feed Rotameter
(2) Extract Reflux Rotameter
(3) Methylcyclohexane Make-Up Rotameter

* Specific gravity data for determinations at 0.72 and 0.74 Specific Gravities not shown.

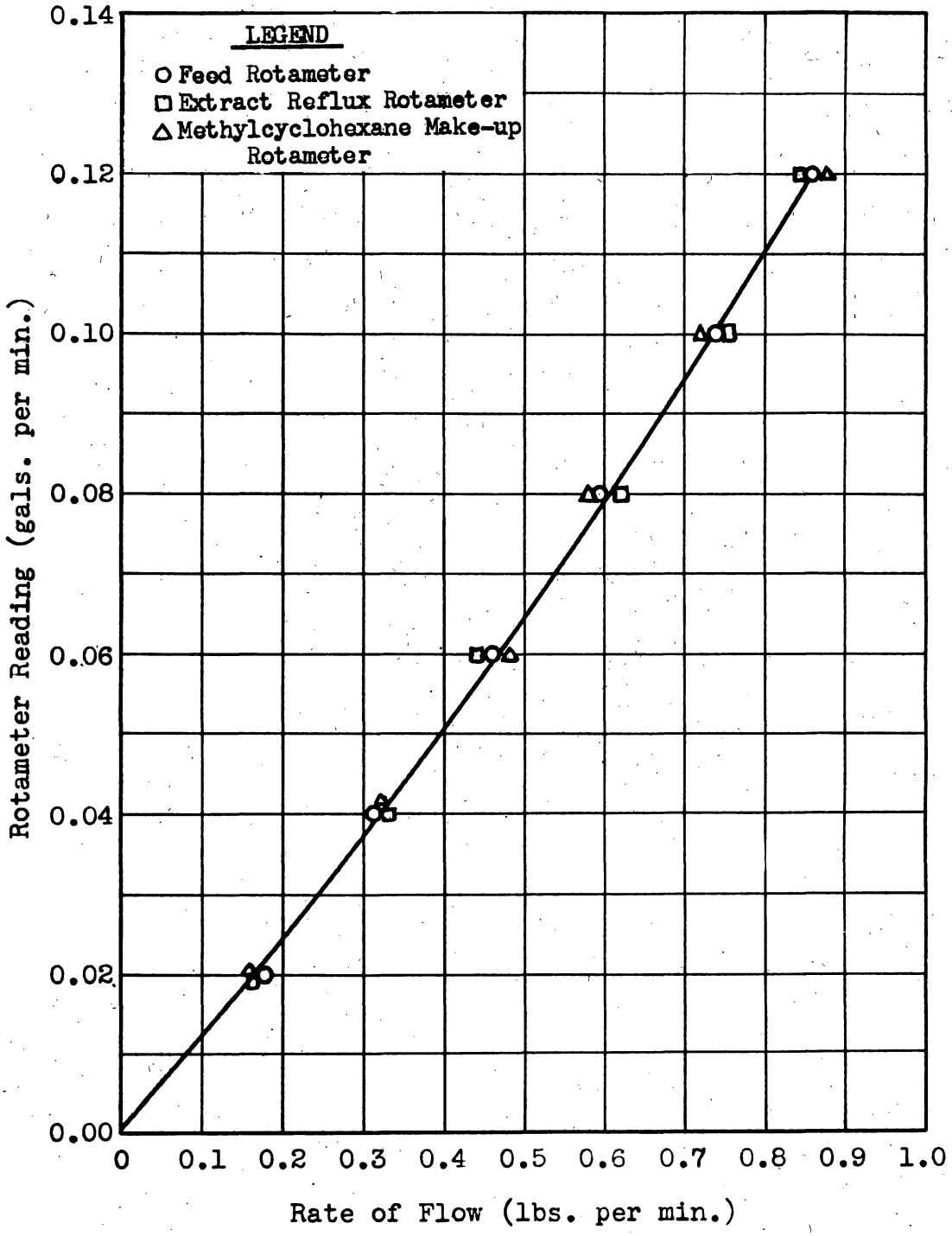


Figure 10. Calibration Curve for Feed, Extract Reflux and Methylcyclohexane Make-up Rotameters.

TABLE VI

CALIBRATION DATA FOR RAFFINATE PRODUCT AND RAFFINATE FEED MAKE-UP ROTAMETERS

Rotameter Reading (gal/min)	Volume per Min. (cc)			Specific Gravity d_4^{20}			Flow Rate (gms/min)			Flow Rate (lbs/min)		
	(1)	(2)	(3)	(1)	(2)	(3)	(1)	(2)	(3)	(1)	(2)	(3)
0.00	0.0	0.0	0.0	-	-	-	0.0	0.0	0.0	0.000	0.000	0.000
0.01	60.0	65.0	67.0	0.76	0.72	0.72	45.6	46.8	48.2	0.101	0.103	0.106
0.02	100.0	115.0	120.0	0.76	0.72	0.72	76.0	82.8	86.4	0.176	0.182	0.190
0.04	185.0	212.0	210.0	0.76	0.72	0.72	140.6	152.7	151.0	0.310	0.336	0.334
0.06	267.0	292.0	295.0	0.76	0.72	0.72	203.0	210.3	212.3	0.448	0.465	0.468
0.08	350.0	370.0	367.0	0.76	0.72	0.72	266.0	263.4	264.2	0.586	0.581	0.584
0.10	430.0	460.0	447.0	0.76	0.72	0.72	326.8	331.2	322.0	0.721	0.730	0.711
0.12	505.0	545.0	550.0	0.76	0.72	0.72	383.8	392.4	396.0	0.847	0.865	0.873

(1) Raffinate Product Rotameter with liquid specific gravity of 0.76
(2) Raffinate Feed Make-up Rotameter with liquid specific gravity of 0.72
(3) Raffinate Product Rotameter with liquid specific gravity of 0.72

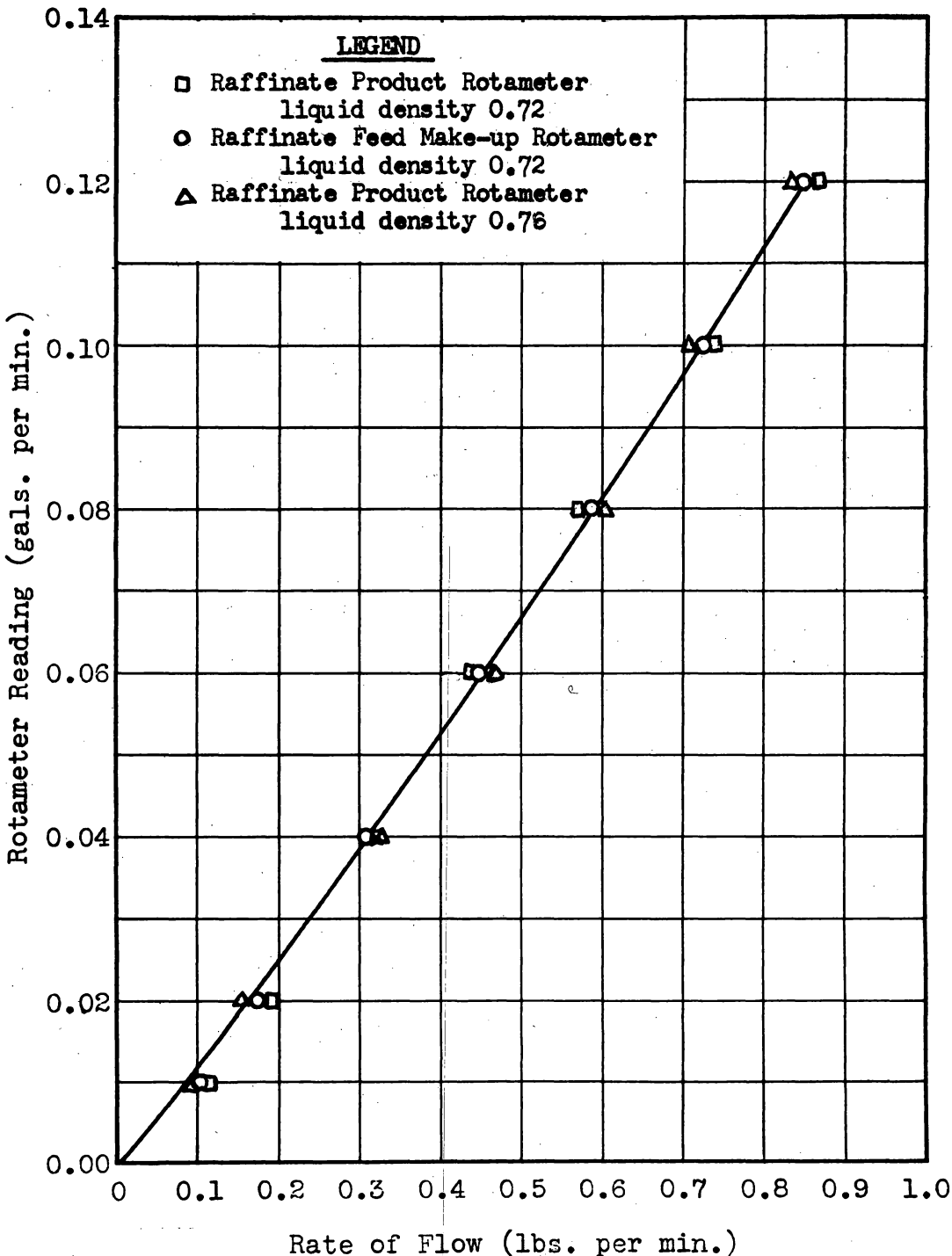


Figure 11. Calibration Curve for Raffinate and n-Heptane Make-up Rotameters.

TABLE VII

CALIBRATION DATA FOR EXTRACT PRODUCT AND SOLVENT ROTAMETERS

Rotameter Reading (gal/min)	Volume per Min. (cc)		Sp. Gr. d_4^{20}	Flow Rate (gms per min.)		Flow Rate (lbs. per min.)	
	(1)	(2)		(1)	(2)	(1)	(2)
0.0	0.0	0.0	-	0.0	0.0	0.000	0.000
0.1	225.0	230.0	0.99	223.0	228.0	0.491	0.503
0.2	520.0	530.0	0.99	515.0	525.0	1.136	1.158
0.3	955.0	950.0	0.99	945.0	941.0	2.083	2.076
0.4	1320.0	1308.0	0.99	1307.0	1297.0	2.882	2.860
0.6	2215.0	2200.0	0.97	2153.0	2179.0	4.830	4.805
0.75	2616.0	2630.0	0.99	2590.0	2605	5.709	5.740

(1) Solvent Rotameter
(2) Extract Product Rotameter

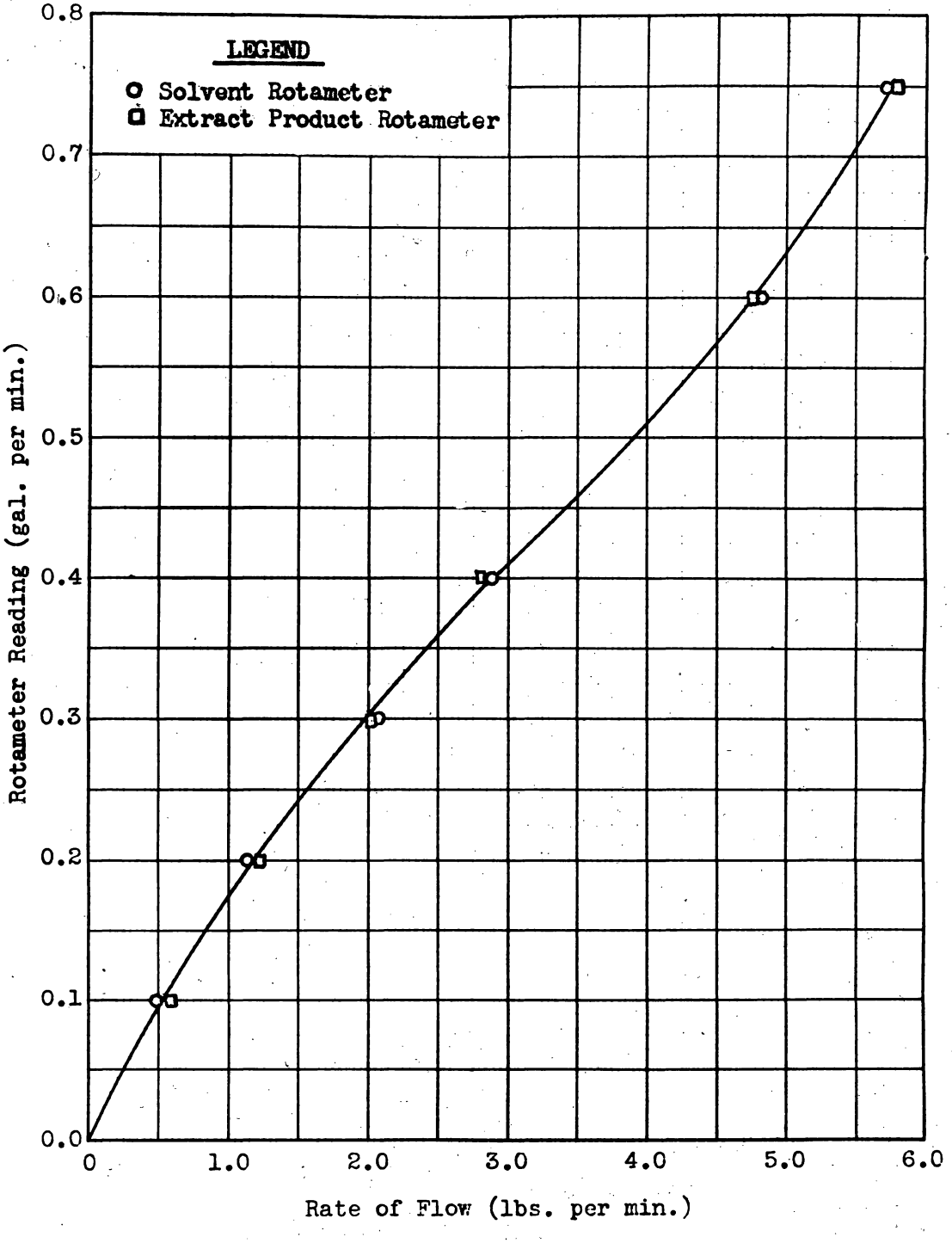


Figure 12. Calibration Curves for Solvent and Extract Product Rotameters

TABLE VIII

PERCENTAGE COMPOSITIONS AND REFRACTIVE INDICES OF EXTRACT AND RAFFINATE PRODUCTS OBTAINED FOR EQUILIBRIUM OPERATION EMPLOYING FEED INPUT NUMBER I*

Reflux Input Flight Number	Time min.	EXTRACT		RAFFINATE	
		Refractive Index N_D^{20}	Per Cent Methylcyclohexane (Solvent-free)	Refractive Index N_D^{20}	Per Cent Methylcyclohexane (Sat'd basis)
30	0	1.5682	44.1	1.4234	66.9
	10	1.5669	55.2	1.4198	59.5
	20	1.5654	62.6	1.4162	51.6
	30	1.5647	67.5	1.4130	44.5
	40	1.5630	80.5	1.4090	35.1
	50	1.5622	84.9	1.4056	26.6
	60	1.5620	86.1	1.4051	25.5
	70	1.5620	86.1	1.4051	25.5
29	0	1.5620	86.1	1.4051	25.5
	5	1.5621	85.5	1.4052	25.6
	10	1.5622	84.9	1.4050	25.0
	15	1.5622	84.9	1.4053	25.8
28	0	1.5622	84.9	1.4052	25.6
	5	1.5625	83.5	1.4050	25.0
	10	1.5626	83.0	1.4053	25.8
27	0	1.5627	81.2	1.4054	26.1
	5	1.5630	80.5	1.4052	25.6
	10	1.5630	80.5	1.4053	25.8
26	0	1.5630	80.5	1.4053	25.8
	5	1.5633	78.6	1.4056	26.6
	10	1.5634	78.2	1.4057	26.8
25	0	1.5634	78.2	1.4056	26.6
	5	1.5636	77.2	1.4058	27.1
	10	1.5638	76.3	1.4059	27.4

*Feed input located 19 flights from the top of the column and 11 flights from the bottom

TABLE IX

PERCENTAGE COMPOSITIONS AND REFRACTIVE INDICES OF EXTRACT AND RAFFINATE PRODUCTS OBTAINED FOR EQUILIBRIUM OPERATION EMPLOYING FEED INPUT NUMBER II*

Reflux Input Flight Number	Time min.	EXTRACT		RAFFINATE	
		Refractive Index N_D^{20}	Per Cent Methylcyclohexane (Solvent-free)	Refractive Index N_D^{20}	Per Cent Methylcyclohexane (Sat'd basis)
30	0	1.5617	86.6	1.4072	30.6
	5	1.5618	86.4	1.4071	30.4
	10	1.5613	88.0	1.4072	30.6
	15	1.5612	88.3	1.4070	30.1
29	0	1.5621	85.9	1.4073	30.8
	5	1.5618	86.4	1.4071	30.4
	10	1.5616	86.8	1.4071	30.4
28	0	1.5623	85.7	1.4075	31.5
	5	1.5622	85.9	1.4075	31.5
	10	1.5621	86.5	1.4073	30.8
27	0	1.5628	84.8	1.4076	31.8
	5	1.5629	84.8	1.4078	32.0
	10	1.5625	85.8	1.4076	31.8
	15	1.5623	86.7	1.4075	31.5
26	0	1.5630	84.5	1.4079	32.3
	5	1.5628	84.8	1.4076	31.8
	10	1.5628	84.8	1.4076	31.8
25	0	1.5638	76.3	1.4059	27.4
	5	1.5637	76.8	1.4066	29.0
	10	1.5635	77.7	1.4062	28.1
	15	1.5636	77.2	1.4066	29.0
	20	1.5633	78.6	1.4069	29.7
	25	1.5631	79.9	1.4075	31.5
	30	1.5630	80.5	1.4078	32.0
	40	1.5630	80.5	1.4079	32.3

*Feed input located 17 flights from the top of the column and 13 flights from the bottom

TABLE X

PERCENTAGE COMPOSITIONS AND REFRACTIVE INDICES OF EXTRACT AND RAFFINATE PRODUCTS OBTAINED FOR EQUILIBRIUM OPERATION EMPLOYING FEED INPUT NUMBER III*

Reflux Input Flight Number	Time min.	EXTRACT		RAFFINATE	
		Refractive Index	Per Cent Methylcyclohexane (Solvent-free)	Refractive Index	Per Cent Methylcyclohexane (Sat'd basis)
30	0	1.5612	88.3	1.4070	30.1
	5	1.5614	87.6	1.4078	32.2
	10	1.5609	90.0	1.4083	33.4
	15	1.5610	89.3	1.4080	32.6
	20	1.5608	90.3	1.4084	33.7
	25	1.5608	90.3	1.4085	33.9
29	0	1.5608	90.3	1.4085	33.9
	55	1.5611	88.6	1.4088	34.6
	10	1.5609	90.0	1.4086	34.1
	15	1.5610	89.3	1.4089	34.8
28	0	1.5610	89.3	1.4089	34.8
	5	1.5612	88.3	1.4088	34.6
	10	1.5612	88.3	1.4090	35.1
27	0	1.5612	88.3	1.4090	35.1
	5	1.5616	86.8	1.4089	34.8
	10	1.5615	87.2	1.4091	35.4
26	0	1.5615	87.2	1.4091	35.4
	5	1.5618	86.4	1.4093	36.0
	10	1.5619	86.4	1.4092	35.8
25	0	1.5619	86.2	1.4092	35.8
	5	1.5621	85.5	1.4094	36.2
	10	1.5622	84.9	1.4094	36.2

*Feed input located 15 flights from the top of the column and 15 flights from the bottom

TABLE XI

PERCENTAGE COMPOSITIONS AND REFRACTIVE INDICES OF EXTRACT AND RAFFINATE PRODUCTS OBTAINED FOR EQUILIBRIUM OPERATION EMPLOYING FEED INPUT NUMBER IV*

Reflux Input Flight Number	Time min.	EXTRACT		RAFFINATE	
		Refractive Index N_D^{20}	Per Cent Methylcyclohexane (Solvent-free)	Refractive Index N_D^{20}	Per Cent Methylcyclohexane (Sat'd basis)
30	0	1.5607	90.6	1.4108	39.1
	5	1.5624	91.7	1.4106	38.7
	10	1.5609	92.0	1.4106	38.7
29	0	1.5612	88.3	1.4109	39.4
	5	1.5610	89.3	1.4110	40.0
	10	1.5606	90.3	1.4108	39.1
	15	1.5608	90.3	1.4108	39.1
28	0	1.5617	86.6	1.4111	40.2
	5	1.5613	88.0	1.4110	40.0
	10	1.5612	88.3	1.4109	39.4
27	0	1.5615	87.2	1.4113	40.6
	5	1.5617	86.6	1.4109	39.7
	10	1.5617	86.6	1.4110	40.0
26	0	1.5618	86.4	1.4115	41.0
	5	1.5617	86.6	1.4113	40.6
	10	1.5615	87.2	1.4113	40.3
	15	1.5615	87.2	1.4112	40.3
25	0	1.5622	84.9	1.4093	36.0
	5	1.5624	83.8	1.4102	38.0
	10	1.5620	86.1	1.4106	38.7
	15	1.5616	86.8	1.4109	39.4
	20	1.5617	86.6	1.4116	41.2
	25	1.5617	86.6	1.4114	40.8

*Feed input located 13 flights from the top of the column and 17 flights from the bottom

TABLE XII

PERCENTAGE COMPOSITIONS AND REFRACTIVE INDICES OF EX
EXTRACT AND RAFFINATE PRODUCTS OBTAINED FOR EQUILIBRIUM OPERATION EMPLOYING FEED INPUT NUMBER V*

Reflux Input Flight Number	Time min.	EXTRACT		RAFFINATE	
		Refractive Index N_D^{20}	Per Cent Methylcyclohexane (Solvent-free)	Refractive Index N_D^{20}	Per Cent Methylcyclohexane (Sat'd basis)
30	0	1.5603	92.0	1.4106	38.7
	5	1.5604	91.7	1.4118	41.6
	10	1.5600	92.7	1.4119	42.0
	15	1.5599	93.0	1.4120	42.2
	20	1.5600	92.7	1.4120	42.2
29	0	1.5601	92.5	1.4120	42.2
	5	1.5602	92.3	1.4123	42.9
	10	1.5602	92.3	1.4124	43.2
28	0	1.5602	92.3	1.4125	43.3
	5	1.5605	91.9	1.4129	44.2
	10	1.5606	91.8	1.4130	44.5
27	0	1.5607	91.4	1.4130	44.5
	5	1.5606	91.8	1.4128	44.0
	10	1.5606	91.8	1.4130	44.5
26	0	1.5606	91.8	1.4131	44.7
	5	1.5607	91.4	1.4133	45.2
	10	1.5608	90.3	1.4134	45.5
25	0	1.5608	90.3	1.4133	45.2
	5	1.5607	91.4	1.4137	46.0
	10	1.5609	90.0	1.4136	45.8
	15	1.5609	90.0	1.4138	46.2

*Feed input located 11 flights from the top of the column and 19 flights from the bottom

TABLE XIII

EXTRACT PRODUCT COMPOSITION IN PER CENT METHYLCYCLOHEXANE RELATED TO FEED INPUT POSITION AND NUMBER OF FLIGHTS IN RECTIFYING AND STRIPPING SECTIONS

Feed Input Flight Number (Flights from top)	Number of Flights in Rectifying and Stripping Sections					
	19	20	21	22	23	24
19	76.3	78.2	80.5	83.0	84.9	86.1
17	80.5	81.8	83.7	85.5	86.8	88.3
15	84.9	86.4	87.2	88.3	89.3	90.3
13	86.6	86.4	87.2	88.3	90.3	92.0
11	90.0	90.3	91.8	91.8	92.3	92.7
	Per Cent Methylcyclohexane in Extract (Solvent-free Basis)					

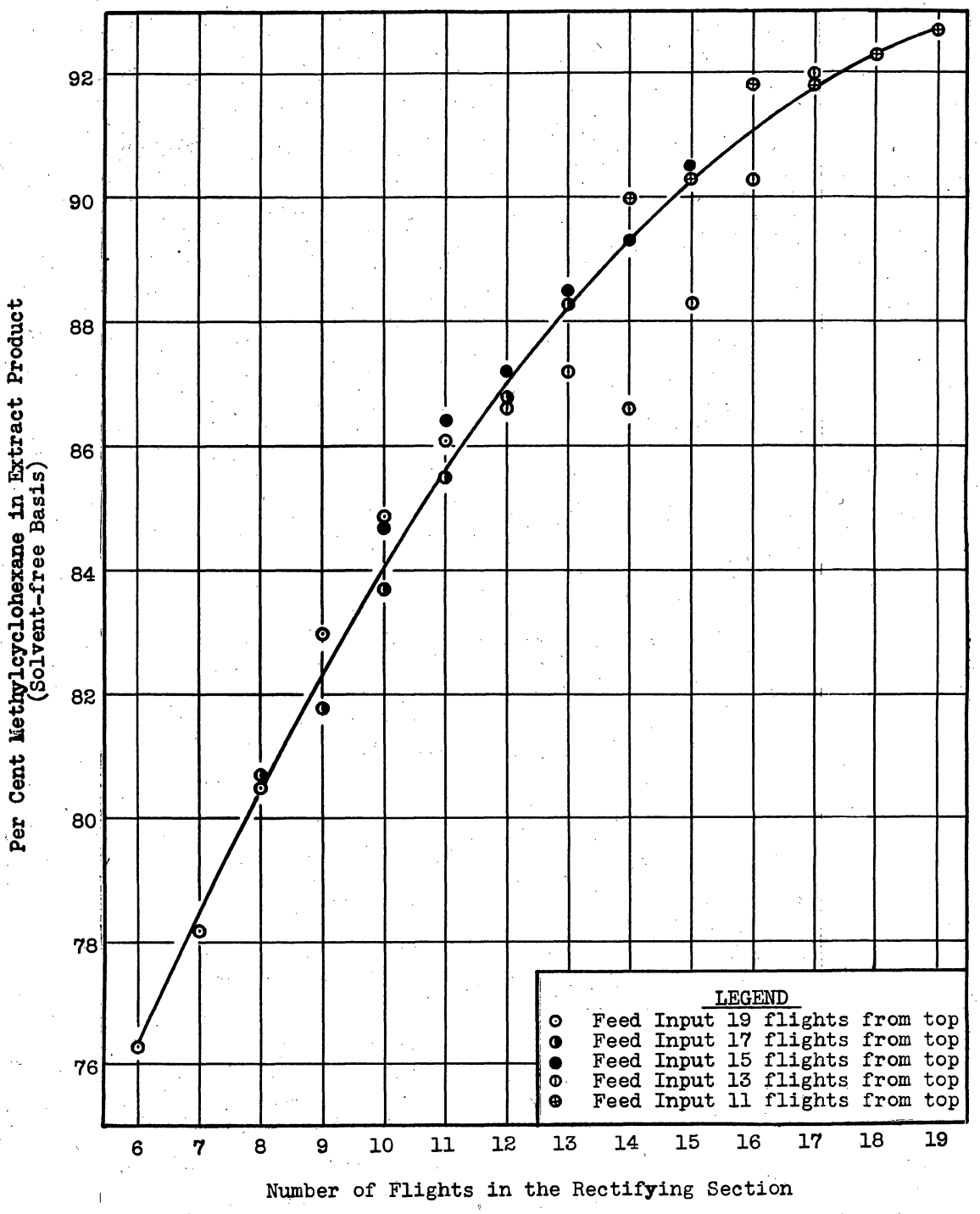


Figure 13. Extract Product Composition as Per Cent Methylcyclohexane Versus Number of Flights between the Feed and Extract Reflux Inputs

TABLE XIV

PER CENT METHYLCYCLOHEXANE IN RAFFINATE PRODUCT AND CALCULATED NUMBER OF THEORETICAL PLATES IN THE STRIPPING SECTION

Feed Position	Feed Input Flight No.	Reflux Input Flight No.	a	b	N	Per Cent Methylcyclohexane in Raffinate Product
V	11	25	5	14	0.00	46.2
		26	5	15	0.16	45.5
		27	5	16	0.32	44.5
		28	5	17	0.48	44.5
		29	5	18	0.64	43.2
		30	5	19	0.80	42.2
IV	13	25	7	12	1.68	40.8
		26	7	13	1.84	40.3
		27	7	14	2.00	40.0
		28	7	15	2.16	39.4
		29	7	16	2.32	39.1
		30	7	17	2.48	38.7
III	15	25	9	10	3.36	36.2
		26	9	11	3.52	35.8
		27	9	12	3.68	35.4
		28	9	13	3.84	35.1
		29	9	14	4.00	34.8
		30	9	15	4.16	33.9
II	17	25	11	8	5.04	32.3
		26	11	9	5.20	31.8
		27	11	10	5.36	31.5
		28	11	11	5.52	30.8
		29	11	12	5.68	30.4
		30	11	13	5.84	30.1
I	19	25	13	6	6.72	27.4
		26	13	7	6.88	26.8
		27	13	8	7.04	25.8
		28	13	9	7.20	25.8
		29	13	10	7.36	25.8
		30	13	11	7.52	25.5

a = Number of actual flights in stripping section
b = Number of actual flights in rectifying section
N = Number of theoretical flights in stripping section as defined on page 111. N= a + 0.16b - 7.24

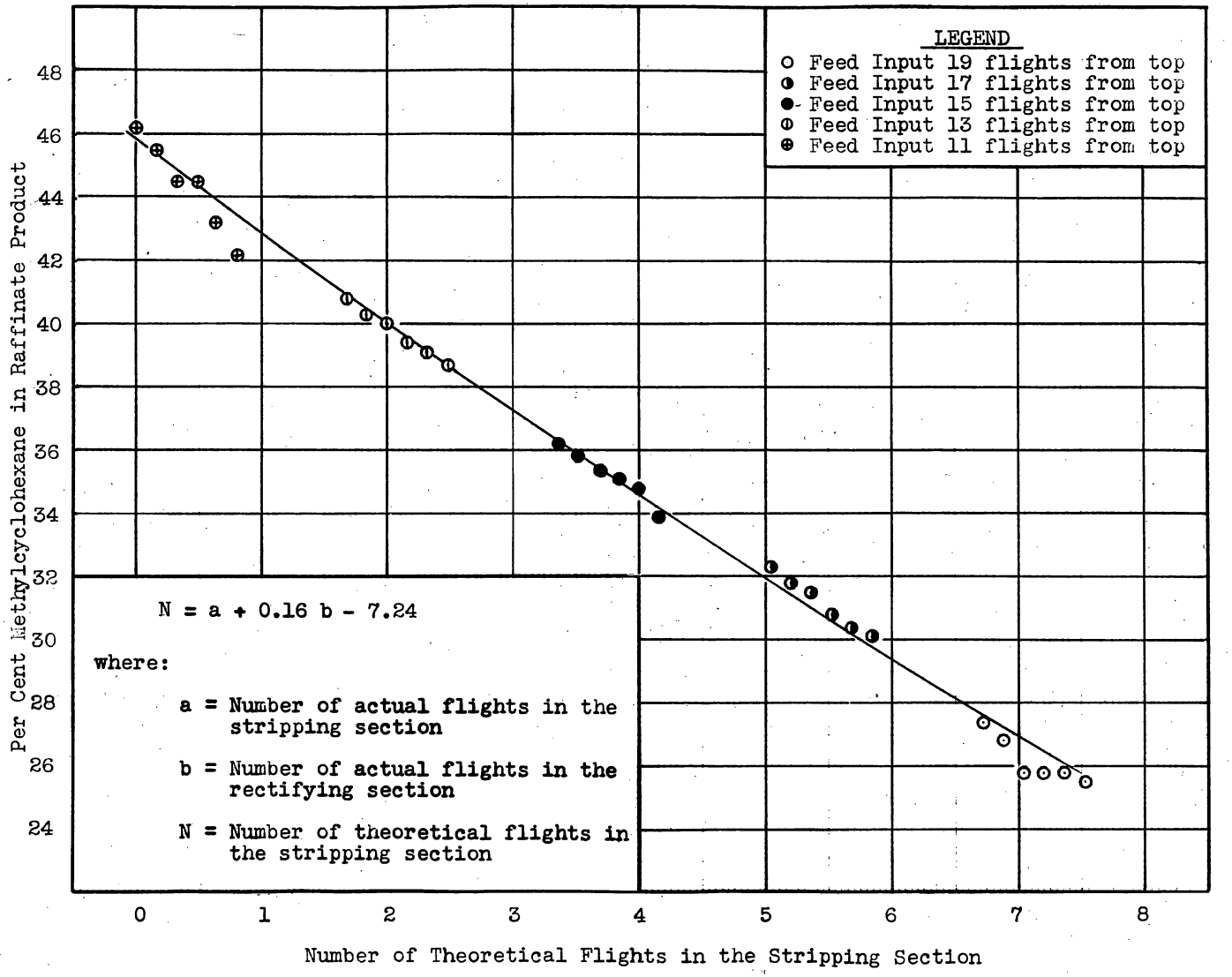
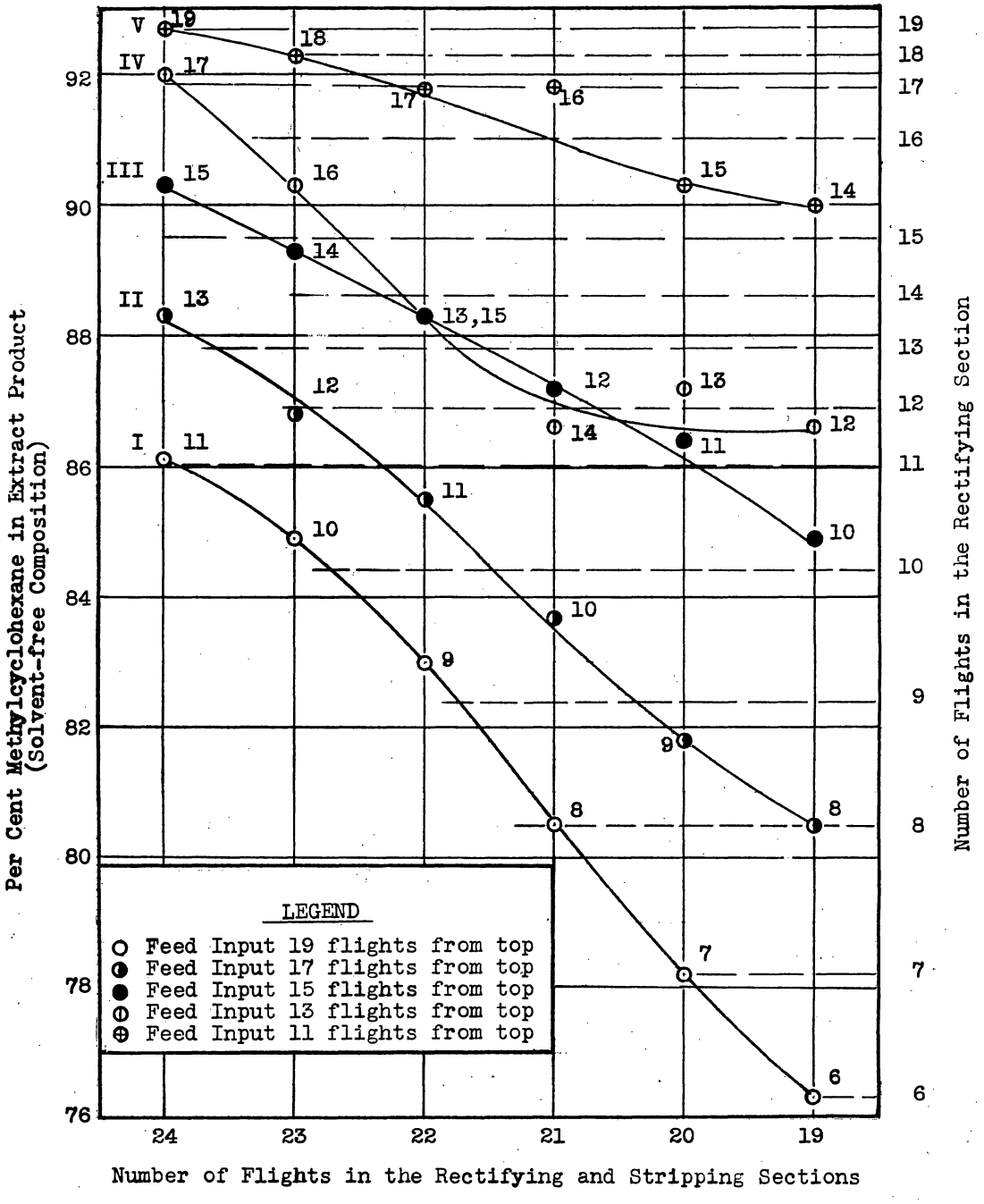


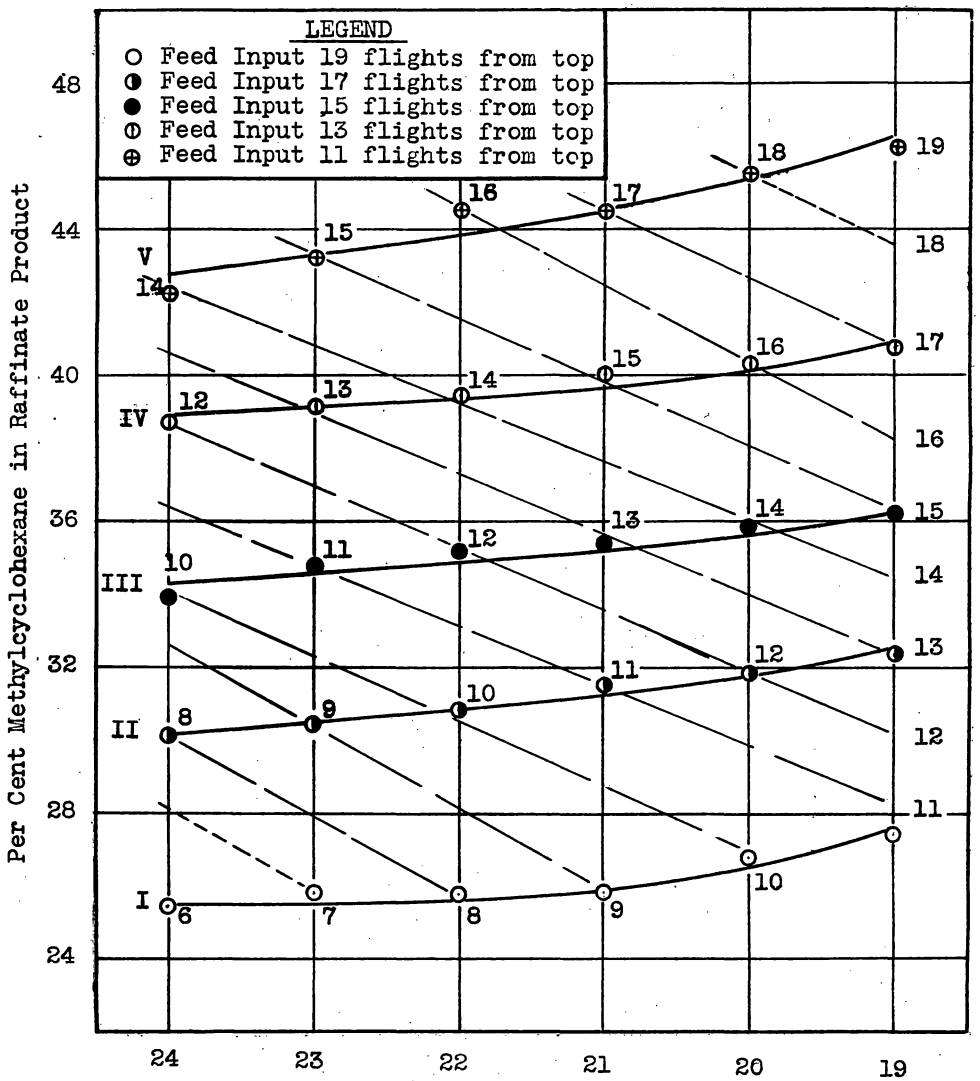
Figure 14. Per Cent Methylcyclohexane in the Raffinate Product Versus Number of Theoretical Flights in the Stripping Section



Arabic numerals indicate the number of flights in the rectifying section.

Roman numerals indicate feed input position on the extraction column.

Figure 15. Relationship between Extract Product Composition and the Number of Flights between Solvent, Extract Reflux and Feed Inputs



Arabic numerals indicate the number of flights in the rectifying section.

Roman numerals indicate feed input positions on the extraction column.

Figure 16. Relationship between Raffinate Product Composition and the Number of Flights between Solvent, Extract Reflux and Feed Inputs

TABLE XV

NUMBER OF THEORETICAL PLATES EQUIVALENT TO SEPARATIONS OBTAINED AT EXTRACT REFLUX AND FEED INPUTS EMPLOYED, CALCULATED BY THE METHOD OF FENSKE (44)

Feed Input Position (Plates from top)	Extract Reflux Input Plate Number (Plates from top)					
	30	29	28	27	26	25
19	3.6 1.6*	3.5 1.6*	3.4 1.6*	3.2 1.6*	2.9 1.5*	2.7 1.5*
17	3.7 1.3*	3.5 1.3*	3.4 1.2*	3.1 1.2*	2.9 1.2*	2.8 1.1*
15	3.6 1.0*	3.5 1.0*	3.4 0.9*	3.1 0.9*	2.9 0.9*	2.7 0.8*
13	3.6 0.6*	3.4 0.5*	3.3 0.5*	2.8 0.5*	2.8 0.5*	2.8 0.5*
11	3.5 0.2*	3.4 0.2*	3.2 0.1*	3.2 0.1*	2.8 0.1*	2.7 0.0*

Values marked (*) are the number of calculated theoretical plates in the stripping section. Other values are the calculated theoretical plates for the entire extractor.

44. Vartoressian K. A. and Fenske, M. R. The System: Methylcyclohexane - Aniline - n-Heptane. Ind. Eng. Chem. 29, 270-277 (1937).

TABLE XVI

NUMBER OF THEORETICAL PLATES AND EQUIVALENT NUMBER

OF CALCULATED THEORETICAL FLIGHTS

IN THE STRIPPING SECTION

Feed Input Flight No.	Reflux Input Flight No.	Theor. Plates Strip. Sect. From Table XV	Theor. Flights Strip. Sect. From Table XIV	Plate Equiv. of One Flight
19	30	1.6	7.52	0.21
	29	1.6	7.36	0.22
	28	1.6	7.20	0.22
	27	1.6	7.04	0.23
	26	1.5	6.88	0.22
	25	1.5	6.72	0.22
17	30	1.3	5.84	0.22
	29	1.3	5.68	0.23
	28	1.2	5.52	0.22
	27	1.2	5.36	0.22
	26	1.2	5.20	0.23
	25	1.1	5.04	0.22
15	30	1.0	4.16	0.24
	29	1.0	4.00	0.25
	28	0.9	3.84	0.23
	27	0.9	3.68	0.24
	26	0.9	3.52	0.26
	25	0.8	3.36	0.27
13	30	0.6	2.48	0.24
	29	0.5	2.32	0.22
	28	0.5	2.16	0.23
	27	0.5	2.00	0.25
	26	0.5	1.84	0.27
	25	0.5	1.68	0.30
11	30	0.2	0.80	0.25
	29	0.2	0.64	0.32
	28	0.1	0.48	0.21
	27	0.1	0.32	0.31
	26	0.1	0.16	0.62
	25	0.0	0.00	0.00

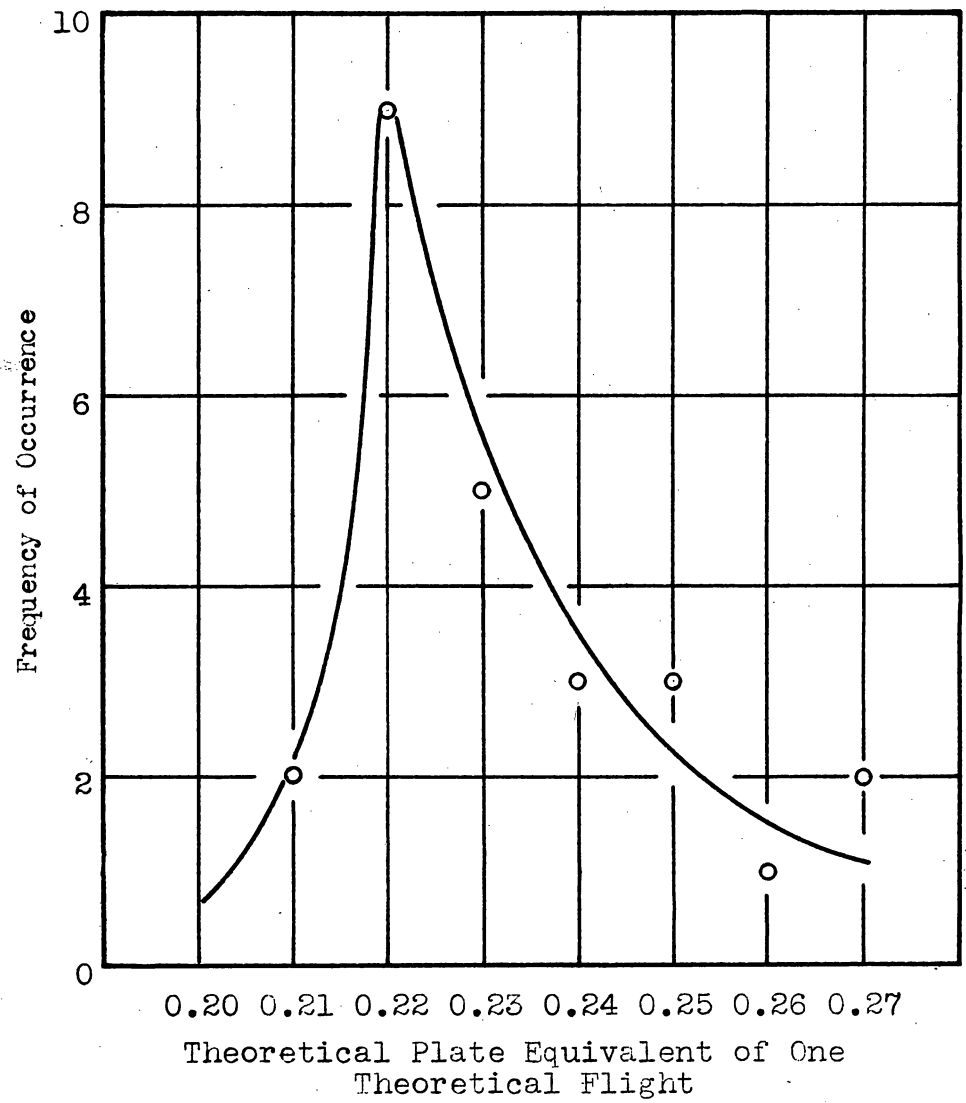


Figure 17. Frequency of Occurrence of Theoretical Plate Equivalent of One Theoretical Flight in the Stripping Section

TABLE XVII

NUMBER OF ACTUAL FLIGHTS AND EQUIVALENT NUMBER OF CALCULATED THEORETICAL PLATES IN THE RECTIFYING SECTION

Feed Input Flight No.	Reflux Input Flight No.	Actual Flights Rect. Section	Theoretical Plates Rect. Section	Plate Equiv. of one Flight
19	30	11	2.0	0.18
	29	10	1.9	0.19
	28	9	1.8	0.20
	27	8	1.6	0.20
	26	7	1.4	0.20
	25	6	1.2	0.20
17	30	13	2.4	0.19
	29	12	2.2	0.18
	28	11	2.2	0.20
	27	10	1.8	0.18
	26	9	1.7	0.19
	25	8	1.7	0.21
15	30	15	2.6	0.17
	29	14	2.5	0.18
	28	13	2.4	0.18
	27	12	2.3	0.19
	26	11	2.0	0.18
	25	10	1.8	0.18
13	30	17	3.0	0.18
	29	16	2.9	0.18
	28	15	2.8	0.19
	27	14	2.3	0.16
	26	13	2.3	0.18
	25	12	2.2	0.18
11	30	19	3.3	0.17
	29	18	3.2	0.18
	28	17	3.1	0.18
	27	16	3.1	0.19
	26	15	2.7	0.18
	25	14	2.7	0.19

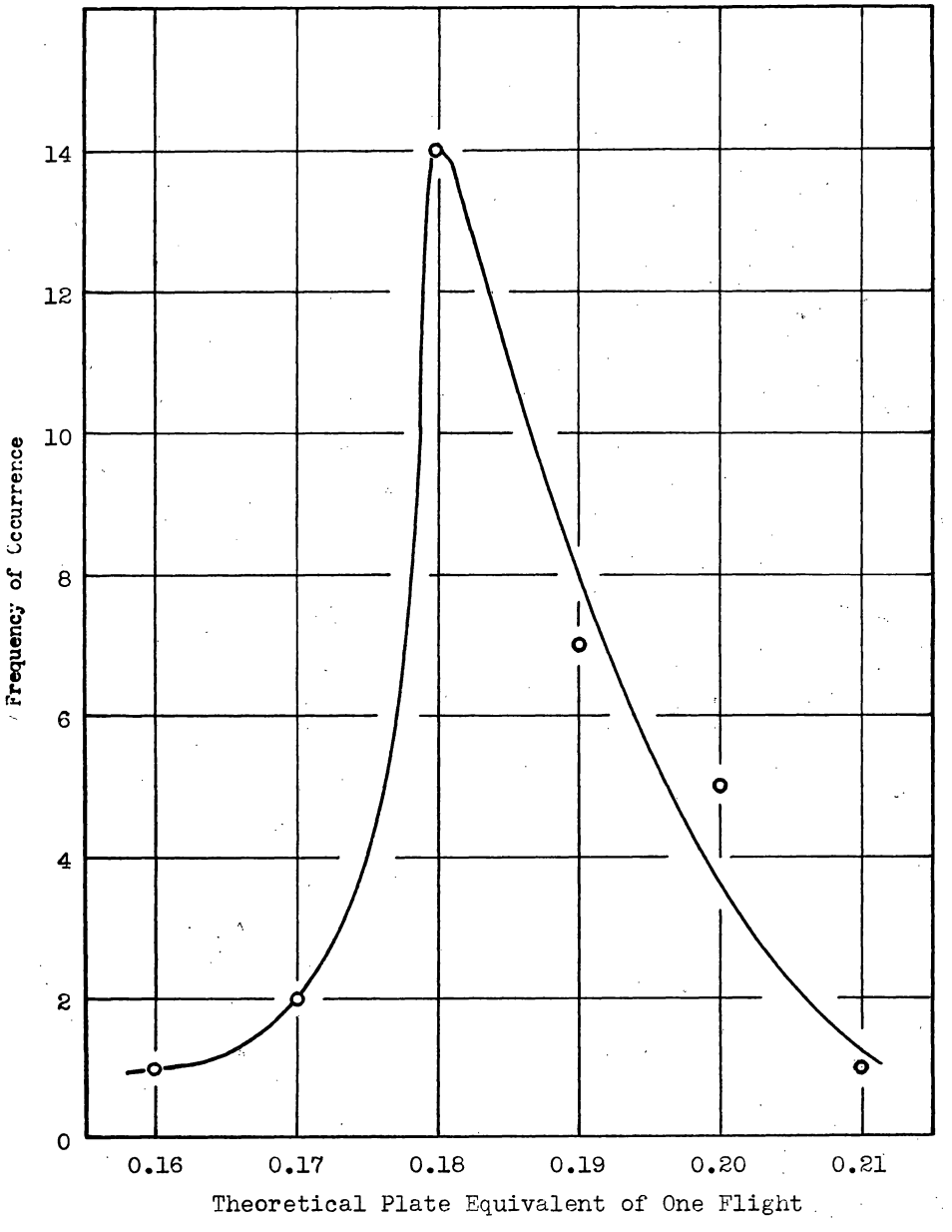


Figure 18. Frequency of Occurrence of Theoretical Plate Equivalent of One Actual Flight in the Rectifying Section

IV. DISCUSSION

A. Discussion of Results

In the operation of a countercurrent liquid-liquid extractor employing the principle of reflux, the essential considerations involved are (1) the mutual solubilities of the materials comprising the system concerned at the temperature of operation, (2) the positions of liquid inputs and outlets to and from the column for a given feed composition and product compositions desired, (3) the relative flow rates of the materials entering and leaving the extractor and the total throughput per unit transverse channel area, (4) the ratio of the amount of reflux to the total amount of product obtained and (5) the size and type of contacting equipment employed. These five considerations cover the four essential variables involved in mass transfer equipment operation, namely, the time of contact between the phases, temperature of operation, relative concentration of the contacted materials and the available interfacial contact area. The variable, contact area, although dependent on the nature of the contacted materials and flow rate conditions, is largely affected by the method of dispersion of the discontinuous phase and the type and size of the apparatus employed.

The Column. The extraction column employed in this investigation was designed for a previous investigation by Quirathon [33]. The design principle involved was that of compressing a 29.5 foot

channel length into a single compact five-foot column. To do this, a column was constructed with a helical channel formed in the annular space between two-inch and five-inch standard black iron pipes. The helix itself consists of a steel ribbon, one-quarter inch in thickness, wound around and welded to the central two-inch pipe to a pitch of one and three-quarters inches. This helical assembly was machined to a light driving fit inside the five-inch pipe. The extractor contains a helix of thirty-one turns and thirty flights, the space between consecutive turns of the helix being defined as a flight. Flights in the column are numbered consecutively from flight number one at the top of the column to flight number thirty at the base of the column. Feed input positions on flight numbers 11, 13, 15, 17 and 19 were employed in this investigation. Extract reflux was introduced on each flight from flight number 25 to flight number 30 inclusive. Solvent and raffinate reflux combined was introduced on the 6th flight for all experimental tests made.

Previous Tests. Previous tests of the extractor were conducted by Quirnbach [45] employing the system methylcyclohexane - aniline - petroleum naphta at 25°C. The results obtained during this investigation indicated that the extractor was equivalent to five theoretical stages and the separation obtained closely approached that reported by Vartoressian and Fenske [44] who employed a 28-foot packed column to obtain a 90 per cent separation

of methylcyclohexane and n-heptane using aniline as the selective solvent. The equilibrium characteristics of the two systems were similar, however, no definite comparison of the two columns could be made in that comparative flow rate conditions were not known.

No effort was made during this investigation to compare the present spiral-channel extractor with a standard spray column having the same channel length (29.5 feet) in that comparative operating characteristics of such a column were not available. The purpose of this investigation was to determine the effect of varying the feed and extract reflux input positions on the operating characteristics of a spiral channel liquid-liquid extractor.

Equipment Modifications. The following modifications recommended by Quinebush (46) were made in order to provide for continuous operation of the extraction equipment:

1. A continuous separation still, a solvent cooler and methylcyclohexane condenser were installed to provide for the continuous separation of the solvent and extract product, for the condensation of the extract product vapors formed and for cooling the solvent for recycle to the system.
2. Feed input dispersion nozzles were installed at five points on the 11th, 13th 15th, 17th and 19th flights. These feed input positions were utilized in determining the effect of varying feed input positions on the

operating characteristics of the extractor. The purpose of these installations was to provide a means of varying the effective channel length in the rectifying and stripping sections.

3. Extract reflux dispersion nozzles were installed on all flights from flight number 25 to flight number 30 inclusive. The installations were made to provide a means for varying the effective channel length in the constant composition zone below the rectifying section to prevent hydrocarbon entrainment in the solvent. In addition, it was desired to provide a means for varying the effective length of the rectifying section independently of the stripping section.

4. Provision was made to utilize the central pipe of the extractor for temperature control.

B. Effect of Varying Feed and Extract Reflux Input Positions

The results of experimental tests are included in Tables XIII and XIV, pages 97 and 99, and are graphically represented in Figures 13 and 14, pages 98 and 100. Figures 15 and 16, pages 101 and 102 are graphical representations of the same results plotted in different form. From these results, it may be seen that the variation of the extract reflux input position had a

marked effect on the extract product compositions obtained at the different feed input positions employed. For all experimental tests, the feed composition was maintained at 50 per cent methylcyclohexane and 50 per cent n-heptane by weight on the solvent-free basis and flow rates were maintained constant. Extract product compositions are expressed in weight per cent on the solvent-free basis.

Feed Input Position 1. With feed introduced at feed input position number 1 on the 19th flight, it was found that the extract product composition increased from 76.3 per cent to 86.1 per cent methylcyclohexane by weight as the extract reflux input position was moved from the 25th flight to the 30th flight inclusive. Moving the extract reflux input position from the 25th to the 30th flight effectively increased from 6 to 11 the number of flights in the rectifying section, and resulted in an overall increase of 9.8 per cent in the methylcyclohexane content of the extract product. This effective increase in the length of the rectifying section of five flights corresponds to an increase in the effective channel length of approximately five feet.

These results would indicate that the effect of hydrocarbon entrainment in the rectifying section of the column is of minor importance as compared to the effect of contact channel length. Furthermore, it is doubtful whether entrainment of the

hydrocarbon phase in the solvent saturated with extract leaving the rectifying section is at all undesirable. Since the hydrocarbon phase, having the same solvent-free composition as the solvent saturated with extract product, is in equilibrium with a solvent phase having a higher concentration of methylcyclohexane, it is indicated that entrainment at this point might even be beneficial rather than detrimental to obtaining a higher degree of separation in the column.

Feed Input Number II. With the feed composition held constant, moving the feed input position from the 19th to the 17th flight effected an overall increase in the length of the rectifying section of two flights. This corresponded to an overall increase of approximately two feet in the effective contact channel length. As a result of this change in the number of flights in the rectifying section, the extract product composition was found to increase in methylcyclohexane content from 80.5 per cent to 88.3 per cent by moving the extract reflux input position from the 25th to the 30th flight. In this case, moving the extract reflux input from the 25th to the 30th flight effectively increased from 8 to 13 the number of flights in the rectifying section. It was noted that a total increase in methylcyclohexane content of the extract product of only 7.8 per cent was obtained by moving the extract reflux input from the 25th to the 30th flight with feed introduced on the 17th flight.

The same variation of extract reflux input positions with the feed input on the 19th flight resulted in a 9.8 per cent increase in the methylcyclohexane content of the extract. This apparent decrease in the separation efficiency may be explained by the fact that the amount of separation obtained on successive flights decreases as the total number of flights in the rectifying section increases or as the extract product composition approaches 100 per cent methylcyclohexane on the solvent-free basis. On observation of the results obtained at feed input positions on the 15th, 13th and 11th flights this trend of a decrease in separation per flight becomes definite as shown in Figure 13, page 98. The curve is exponential.

Feed Input Numbers III, IV and V. Results obtained with the feed input on the 15th, 13th and 11th flights show a consistent increase in the methylcyclohexane content of the extract. The extract product composition ranges obtained for these three feed input positions were from 84.9 per cent to 90.0 per cent, from 86.6 per cent to 92.0 per cent and from 90.0 per cent to 92.7 per cent respectively. Extract reflux input positions were varied between the 25th and 30th flights, as before, for each feed input position.

The number of effective flights in the rectifying section with the feed input position on the 15th flight varied between 10 and 15 depending on the extract reflux input position employed.

The number of effective flights for feed input positions on the 13th and 11th flights ranged from 12 to 17 and from 14 to 19 respectively. If it is remembered that a single flight corresponds to a mean channel length of approximately one foot, it may be seen that the total effective channel length in the rectifying section was varied from six to nineteen feet over the range of feed and extract reflux input positions employed. As in the case with feed input on the 17th flight, results of tests with feed inputs on the 15th, 13th and 11th flights showed marked decreases in the amount of separation obtained per flight. With feed input on the 15th flight, the total change in methylcyclohexane content of the extract was 5.4 per cent; with feed on the 13th flight, 5.4 per cent and with feed on the 11th flight, 2.7 per cent.

Separation Per Flight. As pointed out previously, the separation per flight decreased as the extract product composition approached 100 per cent methylcyclohexane. This decrease in separation per flight may be explained by the fact that as the product composition approached 100 per cent, the diffusional driving force, in this case the concentration difference between the contacted phases, decreased and the rate of mass transfer between phases decreased also. Consequently, as the concentration difference between the contacted phases decreased, the amount of

contact time required to transfer a quantity of material from one phase to the other increased, and the total amount of material transferred per flight decreased.

Variation in Raffinate Product Composition. With feed introduced on the 19th flight, a total of thirteen effective flights was available in the stripping section, between the feed input on the 19th flight and the solvent and raffinate reflux input on the 6th flight. Under these conditions, with the extract reflux input varied between the 25th and 30th flights inclusive, raffinate product compositions containing from 27.5 per cent to 25.5 per cent methylcyclohexane respectively were obtained. This change in raffinate composition with a variation in the effective length of the rectifying section of the extractor indicates that the product composition obtained from the stripping section is dependent not only on the length of the contacting channel in that section, but also on the channel length and the degree of separation obtained in the rectifying section.

Variations in the raffinate product composition between 32.3 per cent and 30.1 per cent; 36.2 per cent and 31.9 per cent; 40.3 per cent and 38.7 per cent and between 46.2 per cent and 42.2 per cent were obtained employing feed input positions on the 17th, 15th, 13th and 11th flights respectively. The extract reflux input was varied from the 25th to the 30th flight inclusive

for each feed position employed. In every case, the highest percentage composition of methylcyclohexane in the raffinate product corresponding to the poorest separation, was obtained for each feed input employed with extract reflux introduced on the 25th flight.

This would indicate that the degree of separation obtainable in the stripping section of the column is limited by the separation obtained in the rectifying section. The very fact that the length of the rectifying section had a distinct effect on the operating characteristics of the stripping section made possible a determination of the theoretical flight relationships for that section. It was also noted that methylcyclohexane content of the raffinate increased from 27.4 per cent to 46.2 per cent with extract reflux introduced on the 25th flight while varying the feed input position from the 19th to the 11th flight. This represents an overall increase in the methylcyclohexane content of the raffinate of 18.7 per cent with an effective decrease of eight flights or a decrease of approximately eight feet in the channel length of the stripping section. With extract reflux introduced on the 30th flight, an increase in the methylcyclohexane content of the raffinate from 25.5 per cent to 42.2 per cent or 16.7 per cent was obtained for the same change of feed input positions as before.

c. Actual Flight - Theoretical Flight - Theoretical Plate Relationships

Theoretical Flight Relationships. A plot of the results of TABLE XIV, page 99, is shown in Figure 14, page 100, and is an attempt to arrive at the relationship between the number of theoretical plates and equivalent number of theoretical flights in the stripping section of the extractor. A theoretical flight may be defined as a single 360° helical contacting device in which the contacted liquid phases present are in equilibrium at all points on the helix. The theoretical flight relationships derived here are useful only in connection with the apparatus employed in this investigation, since one flight, defined as a single 360° helix, must have the same mean channel length and transverse area as the actual flight with which it is being compared. The actual flights in the present spiral-channel liquid-liquid extractor are formed in the annular space between a five-inch and a central two-inch pipe. One flight has a transverse channel area of 2.05 square inches and a mean channel length of 0.983 feet.

A theoretical plate in a separation device is a plate upon which the liquid phases present undergo a change in composition equivalent to a single calculated plate under equilibrium conditions. The theoretical plate relationships for any given

separation may be determined by graphical construction by the method Fenske [44], or by graphical integration of the Colburn equation [7]. The theoretical plate is also referred to in the literature as a theoretical stage or a theoretical transfer unit. [7,9,10]

The actual raffinate product compositions obtained during operation of the extractor were first plotted against the corresponding number of effective flights in the stripping section and the variation in the raffinate product composition, caused by varying the extract reflux input position, was noted. Mathematical expression for the number of theoretical flights was next derived graphically by determining the coefficient of (b) in the equation:

$$N = a + xb + c$$

where:

N = the number of theoretical flights in the stripping section

a = the number of actual flights in the stripping section

b = the number of actual flights in the rectifying section

c = proportionality constant

x = constant determined by plot of experimental data

The actual number of flights in both sections are known, being determined from the feed, extract reflux and solvent input position employed for each determination. The value (x) in the foregoing equation was determined graphically by finding the constant which would cause the experimental points to fit best a characteristic logarithmic curve similar to that obtained by directly plotting the extract products obtained from the rectifying section against the number of flights in that section (Figure 13, page 98). After several trial plots, the value of 0.16 for (x) was obtained as shown in the plot of raffinate product composition versus the number of theoretical flights in the stripping section (Figure 14, page 100). Raffinate product composition was, in one case, the same as that of the feed, and an arbitrary value of zero was assigned as the number of theoretical flights, since the theoretical plate equivalent of the stripping section under those conditions was zero. This gave a value for (c) of 7.24. In any case where the raffinate and feed compositions are not the same, the value of (c) may be easily obtained by extrapolating the data.

With the number of calculated theoretical plates available (TABLE XV, page 103), the theoretical plate - theoretical flight relationships were calculated and appear in TABLE XVI, page 104. To arrive at the actual values for the relationship between theoretical plates and theoretical flights in the stripping section,

the values obtained for theoretical flight equivalents in terms of theoretical plates were plotted in Figure 17, page 105, a frequency distribution plot. This distribution curve shows a distinctly sharp maximum at a value of 0.22 which was accepted as the relationship between one calculated theoretical plate and one theoretical flight. One theoretical flight was thus determined to be equivalent to 0.22 calculated theoretical plates as a separation device.

Actual Flight Relationships. The calculated number of theoretical plates in the stripping section and the total number of calculated theoretical plates appear in TABLE XV, page 103. The total number of theoretical plates in the rectifying section was then determined by the difference in these two values. In TABLE XVII, page 106, the relationship between the number of actual flights in the rectifying section and the total number of calculated theoretical plates is shown. Values obtained for this relationship vary between 0.16 and 0.21 theoretical plates per flight. The average theoretical plate equivalent for one actual flight in the rectifying section was determined by dividing the number of calculated theoretical plates by the total number of effective flights. A frequency distribution analysis of the theoretical plate equivalent of one actual flight in the rectifying section is included as Figure 18, page 107. The results of this test of

the data reveal a sharp maximum at a value of 0.18 which was accepted as a relationship between one calculated theoretical plate and one actual flight. One actual flight in the rectifying section was found to be equivalent to 0.18 theoretical plates as a separation device.

Actual Flight, Theoretical Flight and Theoretical Plate Correlation. It was originally desired to show a definite correlation between actual flights, theoretical flights and theoretical plates could be made for both the rectifying and stripping sections of the extractor. However, sufficient data was not available for calculating the theoretical flight - theoretical plate relationships for the rectifying section. The effect of variation of the solvent and raffinate reflux input position on the operating characteristics of the extractor was not studied. As a result, there was no method whereby the effect of such a variation on the operating characteristics of the rectifying section could be determined. Consequently it was not possible to determine the operating efficiency for this section. A comparison of actual and theoretical flights for the stripping section shows an average flight efficiency of 7.0/13.0 or 54 per cent with the feed input on the 19th flight. The calculated efficiencies for the stripping section with other feed input positions range downward to a low value of 9 per cent

with the feed input on the 11th flight from the top of the extractor. This variation of efficiencies may be attributable to any or all of the following:

1. Insufficient reflux for the attainment of separation in the stripping section
2. Improper dispersion of the feed in the aniline phase resulting in poor contact and possible channeling
3. Depressed solvent level in the extractor, below the solvent input position

As an example, if it is arbitrarily assumed that the solvent level is depressed four flights below the solvent input position on the 6th flight, the flight efficiencies for the stripping section range from 45 per cent with the feed introduced on the 11th flight to 79 per cent with the feed input on the 15th, 17th or 19th flights. If, on the other hand, insufficient raffinate reflux is supplied to effect the desired separation, the total obtainable separation is reduced, and the resulting raffinate product is richer in the component of the extract than is desirable. Finally, improper dispersion of the feed results in reduced interfacial contact and affords a greater opportunity for channeling to take place.

Comparison with a Standard Spray Column. From the results obtained, no comparison of the spiral-channel extractor used in

this investigation with a standard spray column could be made. In order for a valid comparison to be made, operating characteristics for such a spray column having a channel length of 29.5 feet and a transverse channel area of 2.05 square inches must be obtained. A complete comparison would include operating characteristics such as data on obtainable separations at the same flow rate conditions that were employed in this investigation and with feed, extract reflux and raffinate reflux and solvent input positions at the same relative points in the column as in the present extractor. Data on flooding rates for the two columns would yield a comparison of the throughput capacities. In addition, some means would have to be devised to study the characteristics of phase dispersion in the two column, both from the standpoint of the relative rates of drop coalescence in the two column. It is possible that the present spiral-channel extractor may exhibit certain advantages over the standard spray column by reason of the type of channel employed, however, no conclusions can be drawn in this connection without comparative data.

D. Recommendations

Changes in Apparatus. On the basis of observations made during the operation of the liquid-liquid extraction apparatus, certain changes in the apparatus are suggested in order to improve its operating characteristics. Referring to Drawing Number 1, page 53a, the following changes in apparatus are recommended:

1. It is recommended that the solvent and raffinate reflux input be removed from its present location (Q) to the top of the extractor in order to increase effectively the useable channel length and provide for better separation of the components than is now obtainable.
2. Screen filters should be installed in the process pipelines at points (U), (W), (X), (Y) and (Z) between the liquid storage tanks and the rotameters. The purpose of these filters is to remove oxidation and polymerization products of aniline and small particles of rust which enter the rotameters and either block the metering rotor or adhere to it causing erroneous indications of flow rates.
3. Water jackets should be installed on recovered solvent and recovered methylcyclohexane pumps (P_3) and (P_4) in

order to prevent overheating and subsequent vapor locking under vacuum operation.

4. Vent pipes should be installed at the rotameters in order to bleed off air which pockets above the metering tube and causes fluctuations in the flow rates.

Operational Changes. It is recommended that additional tests be conducted employing reflux ratios of 2.5 times the minimum based on a raffinate product composition of 10 per cent methylcyclohexane and 90 per cent n-heptane on a solvent-free basis. Useful information of aid in determining theoretical flight relationships for the rectifying section and actual flight relationships for the stripping section might also be obtained by studying the effect of varying the solvent and raffinate reflux input position with the feed input position while maintaining the extract reflux input position on the same flight.

Future Investigations. On the basis of results obtained, it is suggested that further tests be conducted employing flow rate conditions of 1.5, 2.0 and 2.5 times those used in this investigation. The purpose of further tests would be to determine flow rates for optimum operating conditions. It would also be advisable to conduct tests employing a standard spray column with the same channel length and cross-sectional area and with feed

and extract reflux inputs at the same relative positions in the column as in the present spiral-channel extractor. In that manner, the operating characteristics of the present apparatus might easily be compared with those of a standard spray column. Comparison of the two columns would then be on the basis of maximum throughput, optimum flow rates and excellence as a mass transfer device. The standard column equivalent of the present extractor would have a height of 29.5 feet and a transverse area of 2.05 square inches. The required transverse area of 2.05 square inches is the same as that of a standard 1-1/2 inch pipe.

E. Limitations

This investigation was conducted under the following limiting conditions:

Temperature. The operating temperature in the extractor was maintained constant at 20°C ± 0.5°C by manual regulation of cooling water flow rates in the central pipe of the extraction column. Temperature conditions were based on both the inlet and exit temperatures of the solvent phase.

Flow Rates. Flow rates employed were chosen on the basis of the separation of ten pounds per hour of a feed composed of 46.2 per cent methylcyclohexane, 46.2 per cent n-heptane and 7.6 per cent aniline by weight, and were regulated to within

± 0.001 gallons per minute (± 0.006 pounds per minute) for the hydrocarbon phase liquids and to within ± 0.006 gallons per minute (± 0.04 pounds per minute) for the aniline phase liquids.

Extract Reflux. Extract reflux was introduced at a constant rate of flow of 0.239 pounds per minute corresponding to a practical reflux ratio of 2.86 to 1 equal to 2.5 times the minimum reflux required for a product containing 90 per cent methylcyclohexane on the solvent-free basis. The extract reflux flow rate was maintained constant without regard for changes in product composition.

Analysis. Saturated compositions of solvent phase and hydrocarbon phase products were analyzed by measuring the refractive indices of such mixtures. Maximum error in the hydrocarbon phase compositions, attributable to the limits of error in the measurement of the refractive index, was ± 0.2 per cent methylcyclohexane. The maximum error in aniline phase compositions was ± 0.5 per cent methylcyclohexane. Error in both cases is expressed as per cent methylcyclohexane on a solvent-free basis.

V. CONCLUSIONS

On the basis of the results obtained during the operation of the spiral-channel liquid-liquid extractor, having an effective channel length of 29.5 feet in 30 helical flights and a transverse channel area of 2.05 square inches; employing a reflux ratio (Oe/Fe) of 2.86 with the system: methylcyclohexane - aniline - n-heptane at 20°C ± 0.5°C and atmospheric pressure; using a feed composed of 46.2 per cent methylcyclohexane, 46.2 per cent n-heptane and 7.6 per cent aniline at the following flow rates

1.	Solvent and Extract Product	2.359 lbs/min
2.	Extract Reflux	0.833 lbs/min
3.	Solvent and Raffinate Reflux	2.167 lbs/min
4.	Raffinate Product	0.214 lbs/min
5.	Feed	0.166 lbs/min
6.	Methylcyclohexane Feed Make-up	0.005 lbs/min
7.	Raffinate Feed Make-up	0.002 lbs/min

the following conclusions may be drawn:

1. The calculated number of theoretical plates to which the extractor, as operated, was equivalent decreased on the average from 0.1 to 0.2 for each change of one flight above the base of the extractor in the extract reflux input position.

2. The methylcyclohexane content of the raffinate increased from 25.5 per cent to 46.4 per cent with a decrease from thirteen to five effective flights in the stripping section. The methylcyclohexane content of the extract increased from 76.3 per cent to 92.7 per cent with an increase in the number of flights in the rectifying section from six to nineteen flights.

3. The variation of feed input positions affected the extract and raffinate compositions only insofar as the distribution of the flights in the column between the rectifying and stripping sections was affected. With a feed input on the 11th flight and with extract reflux introduced on the 30th flight, an extract containing 86.1 per cent methylcyclohexane and a raffinate containing 25.5 per cent methylcyclohexane were obtained. At the same extract reflux input position with feed introduced on the 11th flight, an extract containing 92.7 per cent methylcyclohexane and a raffinate containing 42.2 per cent methylcyclohexane were obtained.

4. The variation of extract reflux and feed input positions had no appreciable effect on the overall flight efficiency in the rectifying section as evidenced by values of 0.18 $\pm$ 0.02 obtained as the theoretical plate equivalent of one actual flight in the rectifying

section.

5. An indicated flight efficiency of 82 per cent was obtained for the rectifying section.

6. One theoretical flight in the stripping section was found to be equivalent to 0.22 theoretical plates, as determined by a frequency distribution analysis.

7. One actual flight in the rectifying section of the extractor was equivalent to 0.18 theoretical plates as determined by a frequency distribution analysis.

VI. SUMMARY

The principle involved in the liquid-liquid extraction operation is that of substituting a liquid of greater or lesser volatility for the carrier solvent having the same volatility as the desired product. The resulting solution of the desired product (the extract) in the substituted solvent is then easily separable by fractional distillation or simple equilibrium distillation with resultant heat savings. The principle of reflux, used here as in fractional distillation operations, allows an efficient separation of the original mixture to be made in a single continuous operation, even when the selectivity ratio of the solvent for the desired product is low.

Auxiliary equipment necessary for continuous operation including the recycling of the extract product and solvent was designed and constructed for use in conjunction with the available spiral-channel liquid-liquid extractor which has a channel length of 29.5 feet in 30 flights and a transverse channel area of 2.05 square inches. The auxiliary equipment consisted of a continuous separation still for the separation of the extract product from the solvent, a condenser to recover the extract product vapors formed in the still, and a solvent cooler to reduce the temperature of the recovered solvent for recycle to the system. Minor equipment, such as tankage and pumps, was installed at

necessary points. Feed and extract reflux dispersion nozzles were designed, constructed and installed in the extractor. Feed dispersion nozzles were installed on the 11th, 13th, 15th, 17th and 19th flights and extract reflux dispersion nozzles were installed on all flights from the 25th to the 30th flights inclusive.

In operation, the solvent, saturated with raffinate reflux, was introduced into the top of the column and feed enters at one of the five positions midway in the column. Extract reflux, a portion of the extract product, was introduced into the column at one of the six inlet positions near the base of the extractor. Raffinate product saturated with solvent left the extractor at the top, while solvent saturated with extract was removed at the bottom of the extractor. Reflux ratios and flow rates for the liquid phases were determined by a material balance calculated by the method of Fenske (44).

Tests were conducted with the extractor employing a feed composed of 46.2 per cent methylcyclohexane, 46.2 per cent n-heptane and 7.6 per cent aniline, with aniline used as the selective solvent at a temperature of 20°C ± 0.5°C and at atmospheric pressure. A practical reflux ratio (Oe/Fe) of 2.8%, based on the extract product, was employed. Extract products varying between 76.3 per cent and 92.7 per cent methylcyclohexane on a solvent-free basis were obtained by employing the various extract

(44) _____. The System Methylcyclohexane - Aniline - n-Heptane. Ind. Eng. Chem. 29, 270-277 (1937)

reflux and feed input positions. Raffinate product compositions for the same extract reflux and feed input positions as above varied between 25.5 per cent and 46.2 per cent methylcyclohexane on the saturated basis.

The theoretical flight relationships for the stripping section were determined graphically by plotting the equation:

$$N = a + 0.10b - 7.24$$

where:

N = Number of theoretical flights in the stripping section

a = Number of actual flights in the stripping section

b = Number of actual flights in the rectifying section

The equivalent of one theoretical flight in the stripping section was found to be 0.22 theoretical plate, and the equivalent of one actual flight in the rectifying section was found to be 0.18 theoretical plate under the conditions of operation employed.

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Addenda

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