



Effect of composition on recovery for several azeotropic systems
by Harry C Carpenter

A THESIS Submitted to the Graduate Committee in partial fulfillment of the requirements for the degree of Master of Science in Chemical Engineering at Montana State College
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Abstract:

The purpose of this paper is to determine whether the per cent recovery of the less volatile component in any binary system is independent of the composition of the original charge when separated by means of azeotropic distillation.

Several different systems were investigated using liquids representing the various hydrogen bond classes of compounds. The liquids used were hydrocarbons, alcohols, ketones, acids, chlorinated hydrocarbons, and diethers.

Azeotropic distillations were made upon each of the systems investigated and non-azeotropic distillations were made wherever possible for control runs.

The investigation showed that the recovery of both the less volatile and the more volatile components was increased by azeotropic distillation. The recovery of the less volatile component was independent of the charge composition while the recovery of the more volatile component varied with the charge composition. The purity of the less volatile component was not improved by azeotropic distillation, but the purity of the more volatile component was improved when the separation was difficult.

EFFECT OF COMPOSITION ON RECOVERY FOR
SEVERAL AZEOTROPIC SYSTEMS

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HARRY C. CARPENTER

A THESIS

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ABSTRACT

The purpose of this paper is to determine whether the per cent recovery of the less volatile component in any binary system is independent of the composition of the original charge when separated by means of azeotropic distillation.

Several different systems were investigated using liquids representing the various hydrogen bond classes of compounds. The liquids used were hydrocarbons, alcohols, ketones, acids, chlorinated hydrocarbons, and diethers.

Azeotropic distillations were made upon each of the systems investigated and non-azeotropic distillations were made wherever possible for control runs.

The investigation showed that the recovery of both the less volatile and the more volatile components was increased by azeotropic distillation. The recovery of the less volatile component was independent of the charge composition, while the recovery of the more volatile component varied with the charge composition. The purity of the less volatile component was not improved by azeotropic distillation, but the purity of the more volatile component was improved when the separation was difficult.

I INTRODUCTION

Azeotropic distillation is the term applied to distillation or rectification which involves the formation of constant boiling mixtures. An azeotropic mixture is one which boils or distills without change in composition and it has a boiling point higher or lower than that of any of its pure constituents. (6)

Azeotropic distillation finds one of its principle applications in the separation of mixtures whose components boil too closely together for economical use of simple fractional distillation. The method is particularly applicable when the components to be separated differ in chemical structure so that their volatility is changed in differing degrees by the addition of a third substance. It frequently happens that substances of dissimilar chemical nature which boil close together will form azeotropes between themselves which are entirely incapable of separation by simple distillation, and in these instances, azeotropic distillation is absolutely essential if they are to be separated by any type of distillation process. (1)

In azeotropic distillation a solvent or entrainer not present in the mixture to be separated is added to increase the difference in volatility between the key components. This entrainer forms a constant boiling mixture with one or more of the key components and some is necessarily removed

with the distillate. (1) A method for selecting the entrainer has been described in the literature by Ewell, Harrison, and Berg. (2) By this method liquids may be divided into five classes according to their hydrogen bond-forming capabilities. This system of classification makes it possible to predict the extent of the deviation from ideality. A system that shows a positive deviation from ideality will form a minimum boiling azeotrope if any azeotrope is formed. A maximum boiling azeotrope may be formed if the deviation is negative. The five classes are listed below: (2, 5)

Class I. Liquids capable of forming 3 dimensional networks of strong hydrogen bonds.

Class II. Other liquids composed of molecules containing both active hydrogen atoms and donor atoms (oxygen, nitrogen, and fluorine).

Class III. Liquids composed of molecules containing donor atoms but no active hydrogen atoms.

Class IV. Liquids composed of molecules containing active hydrogen atoms but no donor atoms.

Class V. All other liquids, i.e., liquids having no hydrogen bond-forming capabilities.

In the study made by Daly on the binary system methylcyclohexane - toluene, involving various reflux ratios and charge compositions, it was found that the weight per cent recovery of toluene was independent of the weight per cent of toluene in the charge. 1-Propanol was used as the entrainer. (3) This study was undertaken to determine whether or not this system represented an isolated case or a general trend.

The first binary system studied was that of Daly, methylcyclohexane - toluene. (3) The entrainer used was 2-butanone. The hydrocarbons are Class V compounds, 1-propanol is a Class II compound, and 2-butanone is a Class III compound. (2)

The second system investigated comprised benzene-cyclohexane with acetone, a Class III compound, as entrainer. The hydrocarbons chosen, benzene and cyclohexane, themselves form an azeotrope and cannot be separated by ordinary distillation. (8)

The third system chosen also involved hydrocarbons, ethylbenzene and n-octane, but the entrainer was acetic acid, a Class II compound.

In order to increase the generality of the investigation, the final system chosen involved no hydrocarbons. The binary system to be separated was dioxane and ethylene dichloride, Class III and Class IV compounds, respectively. The entrainer was 1-propanol.

In each of the different systems investigated it was desirable that only the entrainer be soluble in water in order that it might be extracted and the raffinate analyzed after drying by means of a refractometer.

Non-azeotropic runs were made for all systems except the methylcyclohexane - toluene system which was reported by Daly and the benzene - cyclohexane system which cannot be separated by straight rectification. (3, 8)

The purpose of this paper is to determine whether the per cent recovery of the less volatile component in any binary system is independent of the composition of the original charge when separated by means of azeotropic distillation.

II EQUIPMENT, METHODS AND COMPOUNDS

A. Equipment

The following equipment was used in this investigation: a precision rectification column, a Corad constant reflux ratio condenser, a graduated water-cooled receiver, a Harvard type triple-beam balance, a mercury filled "U" type manometer, round bottom glass distilling flasks with side arms, a Valentine refractometer, a ceramic heater, Powerstats, glass stem mercury thermometers, and several 250 ml. separatory funnels.

The column was constructed of three lengths of glass tubing arranged concentrically and held in this position by strips of asbestos tape and glue. The inner tube, 33 mm. inside diameter, was packed with 1/8 inch, stainless steel, Fenske packing. A thermometer was fastened to the outside of this tube near the center. The middle tube was wound with Nichrome wire to provide heat for the column. The amount of heat supplied was controlled by means of a Powerstat. The outside tube provided additional insulation and also protection for the Nichrome winding. The overall height of the column was 48 inches. The height of the packing was 46-1/2 inches.

The Corad head was attached to the top of the column by a 29/42 standard tapered ground glass joint. It was adjusted to give a reflux ratio of 20:1. A second thermometer was inserted in the head to measure vapor temperature. The

thermometer was protected from the descending stream of cold reflux by a small glass shield built into the head.

The distilling flasks used were one and two liter round bottom flasks with 35/25 ground glass ball joints on the necks to fit the bottom of the column. Each flask also had a side arm ending in an 18/9 ball joint to fit the manometer.

The manometer was constructed of glass tubing bent in a "U" shape. One end of the tube was left open to the atmosphere and the other connected to the side arm on the distilling flask. The manometer was approximately 12 inches high and was about half filled with mercury.

The heater used consisted of Nichrome coils mounted on a ceramic base. The rate of heating was controlled by means of a second Powerstat.

The Valentine refractometer was of the glass prism type. The refractive indices were read at $20 \pm 0.1^{\circ}\text{C}.$, with the exception of the readings taken on mixtures of acetic acid and n-octane which were read at $30 \pm 0.1^{\circ}\text{C}.$

The Powerstats were small autotransformers manufactured by Superior Electric Company. The maximum input was 7-1/2 amperes at 110 volts. The output ranged from 0 to 135 volts.

B. Methods

1. Determination of the Azeotropic Compositions:

The composition of each azeotrope was obtained from the literature. (8) A charge of this composition was prepared and distilled. Samples of the distillate were taken until the refractive index became constant. The composition of the azeotrope was obtained from a plot of refractive index versus composition. This plot was made by determining the refractive index of several samples of known composition. In order to check the experimental value, a new charge was prepared using the experimental value of the azeotropic composition. This charge was distilled and samples of the distillate were taken until the refractive index became constant. In all cases this composition agreed with the original experimental value of the azeotropic composition. In order to use this method on the acetic acid - n-octane azeotrope, it was necessary to determine the refractive index at 30°C. At 20°C. the samples were two-phase and could not be analyzed by refractive index. However, at 30°C. the samples were all single-phase. Azeotropic data are given in Table I.

2. Preparation of Charges for the Azeotropic Runs:

In each case the charge consisted of 300 grams of the two components to be separated, excluding the entrainer. The amount of the more volatile component was either 20, 40 or 60 per cent of the 300 grams with the less volatile component

making up the remainder. (See sample calculations) Sufficient entrainer, as determined from the azeotropic composition, was added to remove all the more volatile component and in all cases two grams excess were added to insure complete removal.

3. Azeotropic Runs:

After drying the column by passing compressed air through it, the charge was placed in the distilling flask and the flask attached to the column. The manometer was attached to the side arm of the distilling flask. The heater and the Nichrome winding on the column were attached to the Powerstats and the power turned on. The column was allowed to flood to insure complete wetting of the packing. After flooding, the Powerstat connected to the heater was adjusted until the pressure drop across the packing was 15 ± 1 mm. Hg. This pressure drop was equal to approximately 80 per cent of the pressure drop at the flooding point and was so maintained for all of the runs. The Powerstat connected to the column winding was adjusted until the thermometer on the column showed a temperature approximately 10°C . above that of the overhead vapor. The column was then allowed to run at total reflux until the vapor temperature became constant, usually about one hour.

The initial vapor temperature was noted and the distillate was allowed to pass into the cold receiver. The

temperature was recorded every 5 ml. until the boiling point of the azeotropic mixture at this pressure was reached; at this time the first sample was taken. Comparatively large samples were taken until all of the azeotropic mixture was removed. During the transition from azeotrope to less volatile component, the samples were reduced in order that the distillation curve might be defined more accurately. Larger samples were again taken after the transition until the charge was nearly exhausted from the distilling flask.

Each sample was weighed and the refractive index determined. Next, the samples were placed in separatory funnels and washed with distilled water until all of the entrainer was removed. They were then placed over calcium chloride and allowed to dry. After drying, the refractive index of each sample was determined again.

The column was allowed to drain back into the distilling flask and, after cooling, the flask was removed. The bottoms were weighed and the refractive index of the bottoms determined.

4. Presentation of Azeotropic Data:

Figures 1-4 are examples of the plots prepared from the data for each azeotropic run. These figures show only the runs containing 40 per cent of the most volatile component. Two curves were drawn on each plot. The first is vapor temperature versus weight per cent distilled and the second is

refractive index versus weight per cent distilled. In all cases the refractive index is reported at 20°C. as the acetic acid was removed by water washing the n-octane. In Figure 4 the refractive index curve is plotted without water washing. The weight per cent recovery was determined from these plots. (See sample calculations)

5. Non-azeotropic Runs:

Charges for the non-azeotropic runs were made in exactly the same manner as for azeotropic runs with the exception that the entrainer was omitted. The same column was used under the same conditions. In each case the total charge was 300 grams.

6. Presentation of Non-azeotropic Data:

The same type of plots were made for the non-azeotropic runs as for the azeotropic runs. The recovery was determined from these plots. Examples of the curves are shown in Figures 5-7.

7. Presentation of Azeotropic and Non-Azeotropic Recovery Data:

In Figure 8, the weight per cent recovery of the less volatile component is plotted versus the weight per cent of the less volatile component in the charge for azeotropic distillations. Figure 9 shows the same plot for non-azeotropic distillations. No curve is shown for benzene on this plot because there is no recovery of benzene. Figure 10 is a plot of the weight per cent recovery of the more volatile

component in the charge by azeotropic distillation versus its fraction of the charge. Figure 11 is the same plot for non-azeotropic distillations. No curve is shown for cyclohexane because there is no recovery due to the azeotrope formed by benzene and cyclohexane.

Figures 12-14 show a comparison between azeotropic and non-azeotropic distillation.

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C. Compounds

All of the reagents used in this investigation were purified by fractionation. Only the fraction having a boiling range of $\pm 0.2^{\circ}\text{C}$. was used.

Compound	Grade	Refractive Index		Source
		Obsvd.	Lit.	
Methylcyclohexane	Tech.	1.4235	1.4230(4)	Dow Chemical Co.
Toluene	C.P.	1.4965	1.4968(4)	J.T.Baker Chemical Co.
2-Butanone	Tech.	1.3787	1.3807*(7)	Shell Chemical Corp.
Benzene	Tech.	1.5010	1.5012(4)	General Chemical Co.
Cyclohexane	Tech.	1.4262	1.4262(4)	Shell Chemical Corp.
Acetone	99.5 per cent	1.3588	1.3588**(7)	B.R.Elk & Co., Inc.
Ethylbenzene	Tech.	1.4958	1.4958(4)	Dow Chemical Co.
n-Octane	Tech.	1.3979	1.3976(4)	Conn. Hard Rubber Co.
Acetic Acid	99.5 per cent	1.3683	1.3718(7)	Merck & Co., Inc.
Dioxane	Tech.	1.4224	1.4232(7)	Carbide and Carbon Chemicals Corp.
Ethylene Dichloride	Tech.	1.4449	1.4443(7)	Dow Chemical Co.
1-Propanol	C.P.	1.3859	1.3854(7)	Eimer and Amend

* Observed at 15.9°C .

** Observed at 19.4°C .

III SAMPLE CALCULATIONS

The calculations in this section are all based upon Run #1. This run contained 40 weight per cent methylcyclohexane and 60 weight per cent toluene.

a. To determine the amount of entrainer to add to the charge:

The azeotropic composition was found to be 74.4 weight per cent 2-butanone and 25.6 weight per cent methylcyclohexane (See Table I). The total weight of hydrocarbon charge was 300 grams.

Weight methylcyclohexane:

$$(0.40)(300) = 120 \text{ grams.}$$

Weight toluene:

$$(0.60)(300) = 180 \text{ grams.}$$

Weight 2-butanone plus 2 grams excess:

$$\frac{(120)(74.4)}{(25.6)} + 2 = 351 \text{ grams.}$$

Total charge = 120 + 180 + 351 = 651 grams.

b. To determine weight per cent recovery of methylcyclohexane:

Figure 1 shows that beyond 69.3 weight per cent distilled the Refractive Index curve exceeds 1.4235.

Weight per cent methylcyclohexane recovery:

$$\frac{(0.693)(651)(0.256)}{(120)} = 96.4$$

c. To determine weight per cent recovery of toluene:
Figure 1 shows that at 74.5 weight per cent distilled the
Refractive Index curve has reached a value of 1.4963.

Weight per cent toluene recovery:

$$\frac{(1 - 0.745)(651)}{(180)} = 92.3$$

IV RESULTS

The first system studied, methylcyclohexane-toluene with 2-butanone as the entrainer, gave the same results as were reported by Daly. (3) When separated by azeotropic distillation in a batch column the weight per cent recovery of the toluene which is the less volatile component, is independent of the per cent of toluene in the charge. The recovery of methylcyclohexane, the more volatile component, increased as the per cent of methylcyclohexane in the charge was increased. Runs 1-3 shown in Table II give a comparison of the recoveries and the weight per cents of the components in the original charge. The purity of the recovered products exceeded 99.5 weight per cent. Daly (3) showed that when separated by non-azeotropic distillation, the weight per cent recovery of the toluene increased as the toluene in the charge was increased. The purity of the toluene recovered exceeded 99.5 weight per cent but the methylcyclohexane did not reach this purity. The recovery of methylcyclohexane of a given purity, 88.5 weight per cent, increased as the methylcyclohexane in the charge increased. A graphical comparison of the recoveries by both azeotropic and non-azeotropic distillation is shown in Fig.12

To expand further the generality of the study, the binary system benzene-cyclohexane with acetone as the entrainer was used. As benzene and cyclohexane together form an azeotrope, no results are shown in Table III for the non-

azeotropic separation of this system.* Runs 4-6 in Table II show that the purity of the recovered product exceeds 99.5 weight per cent and that the weight per cent recovery of benzene, the less volatile component, is independent of the per cent of benzene in the original charge, while the recovery of cyclohexane, the more volatile component is dependent on its proportion in the original charge.

Acetic acid was the entrainer employed in the separation of n-octane from ethylbenzene. Table II shows that the purity of the recovered products and the recoveries when separated by azeotropic distillation are in accord with the rest of the study. The results of the non-azeotropic separation are shown in Table III and correspond with the results shown for methylcyclohexane-toluene. A comparison of azeotropic and non-azeotropic separations of the binary system n-octane-ethylbenzene is shown in Figure 12.

A non-hydrocarbon system, dioxane-ethylene dichloride, with 1-propanol as the entrainer gave results that were similar to the other systems reported. In Table III it is shown that the purity of ethylene dichloride that was recovered exceeded a value of 99.5 weight per cent. The fact that the purity of ethylene dichloride obtained by non-azeotropic distillation was approximately the same as that obtained by azeotropic distillation, namely slightly higher than 99.5 per cent, indicates that the purity of the more

volatile component is not always increased by azeotropic distillation. Figure 14 shows a comparison between azeotropic and non-azeotropic distillation for the system dioxane-ethylene dichloride.

Figure 8 shows that the recovery of the less volatile component in a binary mixture is independent of the proportion of that component in the original charge when the mixture is separated by azeotropic distillation. Figure 9 shows that the recovery of the less volatile component is dependent on the charge composition when the mixture is separated by non-azeotropic distillation.

Figures 10 and 11 show that the recovery of the more volatile component is dependent on the proportion of that component in the original charge when the mixture is separated by either azeotropic or non-azeotropic distillation.

Comparison of Table II with Table III shows that the amount of each component of a given purity that was recovered was increased by azeotropic distillation. This comparison also shows that the purity of the less volatile component that was recovered was not increased by azeotropic distillation. The purity of the more volatile component that was recovered was increased by azeotropic distillation for all the system where the separation was somewhat difficult. In the case of dioxane-ethylene dichloride, however, the purity of the ethylene dichloride was not increased.

The results of this study are in accord with the results reported by Daly. (3) He reported the recovery for one binary system representing two hydrogen bond classes, II and V, while this study reports the recoveries for four binary systems representing four hydrogen bond classes, II, III, IV and V. Further studies might well include Class I (5) compounds which were not covered by either of these papers, as well as other types of liquids such as ethers and amines.

V CONCLUSIONS

From the results of this study the following conclusions may be made:

1. The recovery of the less volatile component in the charge is independent of its per cent in the charge when the charge is separated by azeotropic distillation.
2. The purity of the less volatile component in the charge is not increased by azeotropic distillation but the quantity at a given purity is increased.
3. The recovery of the more volatile component in the charge is dependent on its per cent in the charge for both azeotropic and non-azeotropic distillation.
4. The purity of the more volatile component in the charge is not always increased by azeotropic distillation but the quantity at a given purity recovered is increased.

VI ACKNOWLEDGMENT

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TABLE I

AZEOTROPIC DATA

Component	Entrainer	Azeotrope Boiling Pt. °C,		Press., mm.	Azeotrope Wt. % Entrainer	
		Ref. (8)	Obsvd.		Ref. (8)	Obsvd.
Methylcyclohexane	2-Butanone	78.0	72.4	639.8	70	74.4
Cyclohexane	Acetone	<54.0	48.3	639.4	<85	68.6
n-Octane	Acetic Acid	105.5	99.4	639.7	52.5	55.0
Ethylene Dichloride	1-Propanol	80.65	74.8	633.7	19.0	16.0

CHARGE COMPOSITIONS, ENTRAINERS AND RECOVERIES FOR
AZEOTROPIC RUNS

Run No.	Charge Composition Weight Per Cent		Entrainer	Weight Per Cent Recovery 99.5 Wt.% Purity	
	Toluene	Methylcy- clohexane		2-Butanone	Toluene
1	80	20		92.3	92.9
2	60	40		92.3	96.4
3	40	60		92.6	96.4
	Benzene	Cyclohexane	Acetone	Benzene	Cyclo- hexane
4	80	20		89.5	88.2
5	60	40		89.3	94.4
6	40	60		88.1	94.6
	Ethyl- benzene	n-Octane	Acetic Acid	Ethyl- benzene	n-Octane
7	80	20		89.5	85.7
8	60	40		89.6	89.4
9	40	60		89.2	91.4
	Dioxane	Ethylene Dichloride	1-Propanol	Dioxane	Ethylene Dichloride
10	80	20		88.1	48.2
11	60	40		88.3	76.5
12	40	60		88.1	79.3

TABLE III

CHARGE COMPOSITION AND RECOVERIES
FOR NON-AZEOTROPIC RUNS

Run No.	Charge Composition Weight Per Cent		Weight Per Cent Recovery		
			99.5 Wt. % Purity		88.5 Wt. %
	Toluene	Methylcyclo- hexane	Toluene	Methylcyclohexane	
1 (3)	80	20	77.5	0	0
2 (3)	60	40	73.4	0	56.4
3 (3)	40	60	66.3	0	79.2
	Benzene	Cyclohexane	Benzene Cyclohexane Separation Impossible, Azeo- trope forms between benzene and cyclohexane		
	Ethyl- benzene	n-Octane	Ethyl- benzene	n-Octane	
4	80	20	78.5	0	73.1
5	60	40	75.0	0	87.5
6	40	60	73.7	0	95.9
	Dioxane	Ethylene Dichloride	Dioxane	Ethylene Dichloride	Dichloride not determined
7	80	20	65.3	0	
8	60	40	64.1	50.0	"
9	40	60	63.6	60.8	"

