1. EXPERIMENT # 2   GRACE KIM
    7/9/2007   MIKE HALL
   FRACTIONAL AND SIMPLE DISTILLATION

2. PURPOSE AND INTRODUCTION

        A mixture of cyclohexane and toluene (mixture X) was separated via fractional and simple distillation.

        The effect of fractionation on increasing the efficiency of separation was assessed by comparing the stillhead temperature in fractional and simple distillation as a function of distillate produced. The three fractions collected during fractional distillation were also analyzed using gas liquid chromatography (GC) to determine the extent of fractionation.
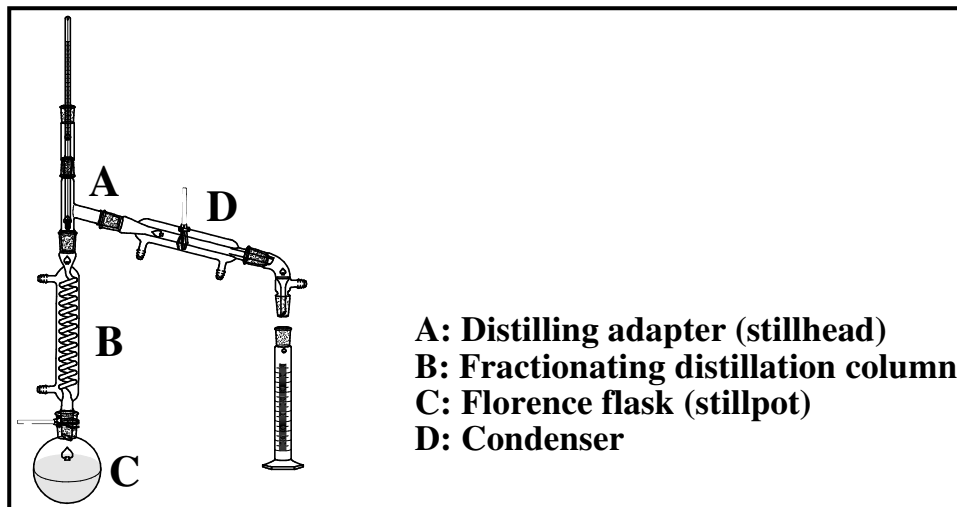
        The peak areas in the GC printout were also calculated using manual methods (i.e. triangulation and cut and weigh) to compare their accuracy against the computer's integration of the peak area.

3. EXPERIMENTAL PROCEDURES:

Table of Reagents

Compound	Molecular Weight	Physical Properties		
Cyclohexane, C_6H_{12}	84.1608 g/ mol	flammable	b.p. = 80.7^0C	flash pt. = -18^0C
Toluene, $C_6H_5CH_3$	92.1402 g/ mol	flammable	b.p. = 110.6^0C	flash pt. = 4^0C

Fractional Distillation:

        0.5-mL of the mixture X was reserved for GC analysis and 26.0-mL of the mixture was placed in the florence flask of the fractional distillation setup shown in Figure 1.

    A: Distilling adapter (stillhead)
    B: Fractionating distillation column
    C: Florence flask (stillpot)
    D: Condenser

    The fractionating distillation column was packed loosely with steel sponge, and the distillation column and the florence flask neck were insulated with aluminum foil to maintain the proper temperature gradient. The heat source, a Thermowell heater, was adjusted to obtain a distillation rate of about 1 drop/ second.

Figure 1. Fractional distillation setup.

        The stillhead temperature and volume were recorded for every 2-mL of distillate produced. Three fractions were collected. The first fraction, containing mostly cyclohexane, was collected at 79-83^0C. The second fraction, containing a mixture of cyclohexane and toluene, was collected as the

temperature rose sharply to ~104^{0}C. The last fraction, containing mostly toluene, was collected at ~104^{0}C. Samples of each fraction were analyzed by GC to determine the extent of fractionation and the relative amounts of cyclohexane and toluene present in the original mixture.

Simple Distillation:

The distillate from the fractional distillation and the pot residue were recombined and distilled using a simple distillation setup (i.e. the same as the fractional distillation setup, but without the fractionating distillation column). The stillhead temperature and volume were recorded for every 2-mL of distillate produced.

4. RESULTS AND OBSERVATIONS

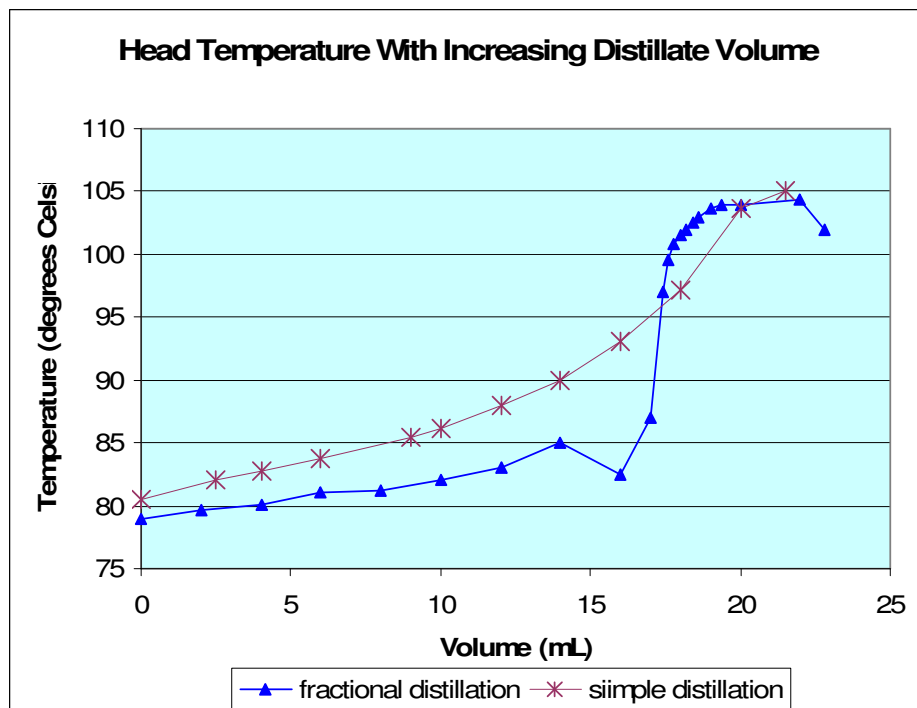## Fractional Distillation and Simple Distillation

Table 1. Fractional Distillation, Head Temperature With Increasing Distillate Volume

	Vol (mL)	Temp (deg C)		Vol (mL)	Temp (deg C)		Vol (mL)	Temp (deg C)
A	0.0	79.0	**B**	17.0	87.0	**C**	20.0	104.0
	2.0	79.7		17.4	97.0		22.0	104.3
	4.0	80.1		17.6	99.5		22.8	102.0
	6.0	81.0		17.8	100.8			
	8.0	81.2		18.0	101.5			
	10.0	82.0		18.2	102.0			
	12.0	83.0		18.4	102.5			
	14.0	85.0		18.6	103.0			
	16.0	82.5		19.0	103.7			
				19.4	104.0			

Table 2. Simple Distillation Head Temperature With Increasing Distillate Volume

Vol (mL)	Temp (deg C)
0.0	80.5
2.5	82.0
4.0	82.8
6.0	83.8
9.0	85.5
10.0	86.1
12.0	88.0
14.0	90.0
16.0	93.0
18.0	97.1
20.0	103.7
21.5	105.0

Graph 1. Fractional Distillation versus Simple Distillation

Head Temperature With Increasing Distillate Volume

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Gas Liquid Chromatography Analysis of Mixture X and Fractions

Table 3. Gas liquid chromatography analysis of fractions from fractional distillation: Percentages and volumes of cyclohexane and toluene in each fraction and in the total distillate

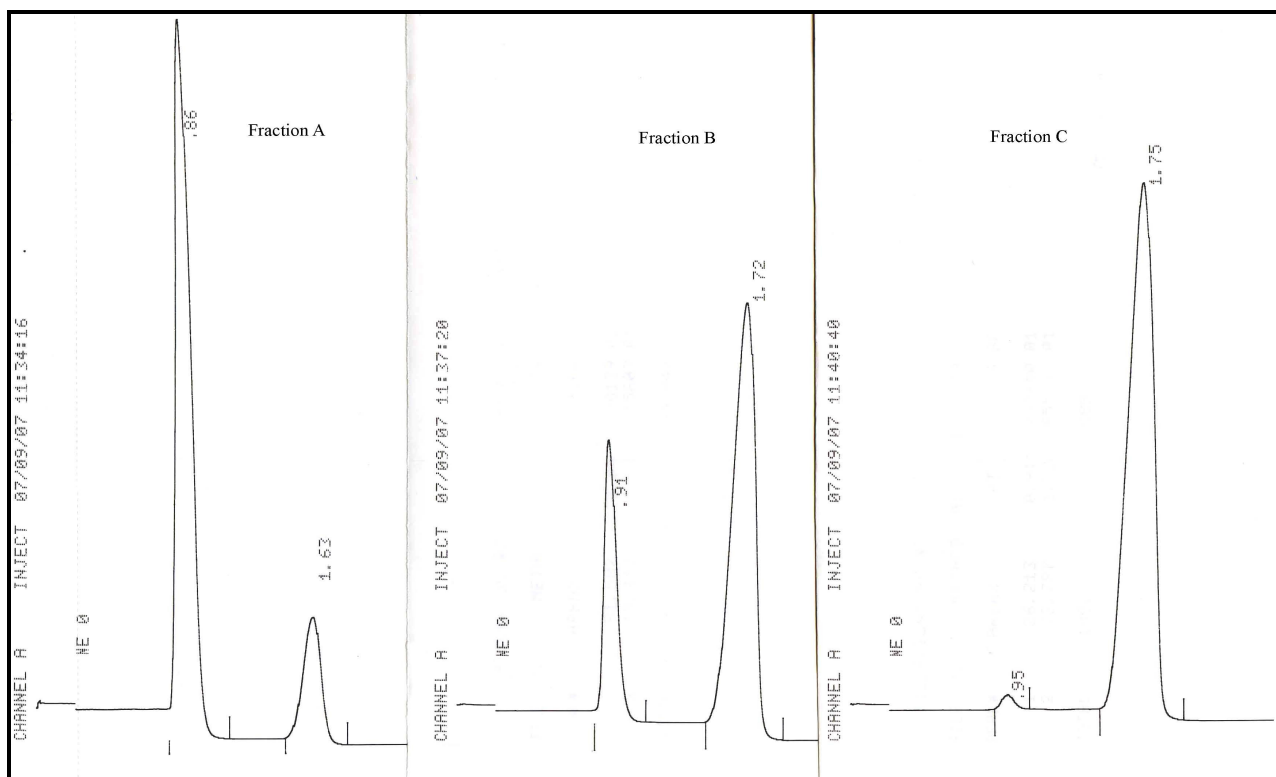|	% cyclohexane	% toluene	mL cyclohexane	mL toluene	total vol (mL)
Fraction A	81.582%	18.418%	13.05	2.95	16
Fraction B	26.213%	73.787%	0.89	2.51	3.4
Fraction C	1.457%	98.543%	0.05	3.35	3.4
Fraction A+B+C	61.404%	38.596%	14.0	8.8	22.8
Mixture X, expected	**56.472%**	**43.528%**	12.9	9.9	22.8

Figure 2. GC chart for fraction A, B, and C from the fractional distillation. The first peak for each fraction represents cyclohexane, the second peak represents toluene.

Table 4. Peak Area Determination for Mixture X

Computer integration

% cyclohexane	56.472%
% toluene	43.528%

Cut & Weigh

	mass (g)	%	% error
cyclohexane	0.0695	57.5%	1.8%
toluene	0.0513	42.5%	2.4%

*weighed with the Mettler-Toledo balance in Rm510

Triangulation

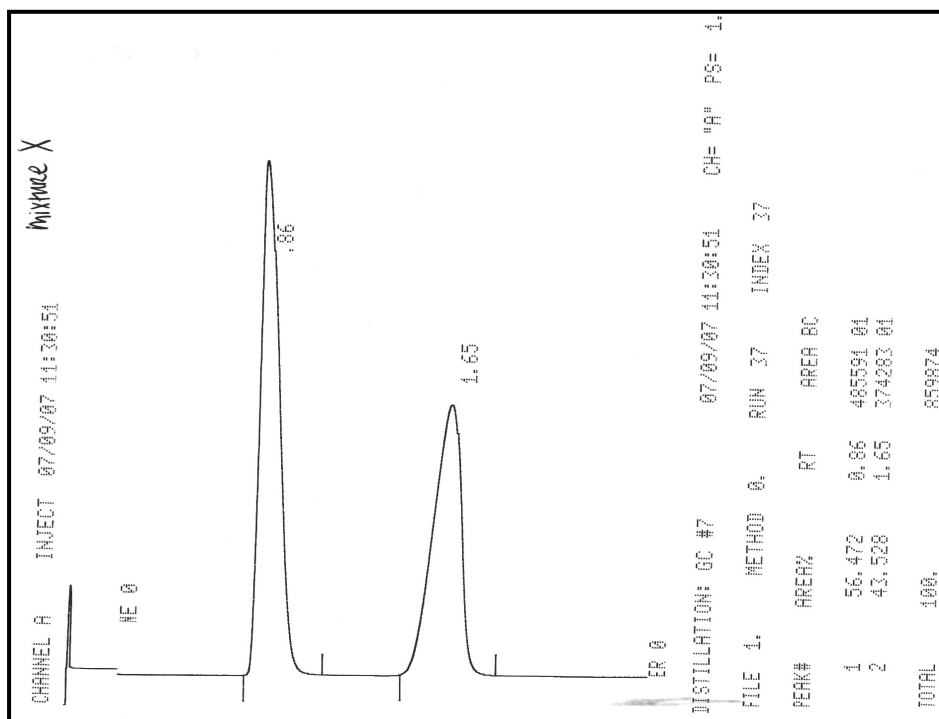|	height (mm)	width at half-height (mm)	area (mm squared)	%	% error
cyclohexane	131.5	3.2	420.8	54.5%	3.5%
toluene	69	5.1	351.9	45.5%	4.6%

Figure 3. GC chart for Mixture X. The first peak represents cyclohexane, and the second peak represents toluene.

5. CONCLUSIONS

   A comparison of the fractional distillation and simple distillation curve showed that fractional distillation was the more efficient technique for separating cyclohexane and toluene. The fractional distillation curve showed a sharper, steeper temperature change separating the two boiling point plateaus of cyclohexane and toluene. Since the slope (corresponding to fraction B) was steeper, this indicates that there was less distillate collected which was an even mixture of both cyclohexane and toluene. Also, since the plateaus (corresponding to fraction A and C) for the boiling points of cyclohexane and toluene were flatter, the distillate collected for each component was more highly enriched (i.e. purer) than what was obtained through just simple distillation.

   The extent of fractionation was shown by the high percentage of cyclohexane and low percentage of toluene collected in fraction A, and the high percentage of toluene and low percentage of cyclohexane collected in fraction C. The large percentage of toluene collected in fraction B and the low volume of distillate collected in fraction C suggest that the collection of fraction C's distillate should have occurred earlier than it did.

   A comparison of the manual methods for peak area determination for the GC and the computer-integration method showed that the manual methods, while prone to human error, are reliable and accurate.

6. ANSWERS TO ASSIGNED PROBLEMS

#11. At 50^0C, $\quad$ $n_{methanol} = 0.1$ mol $\quad$ $P_{vap,\ methanol}^{\ 0} = 406$ torr

$\qquad\qquad\qquad$ $n_{ethanol} = 0.2$ mol $\quad$ $P_{vap,\ ethanol}^{\ 0} = 222$ torr

$$P_{vap,\ methanol\ in\ soln} = \left(\frac{0.1mol}{0.1mol + 0.2mol}\right)406torr = 135torr$$

$$P_{vap,\ ethanol\ in\ soln} = \left(\frac{0.2mol}{0.1mol + 0.2mol}\right)222torr = 148torr$$

$P_{total\ soln} = P_{vap,\ methanol\ in\ soln} + P_{vap,\ ethanol\ in\ soln} = 135$ torr $+ 148$ torr $= 283$ torr

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