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Experiment 5: Distillation and Gas Chromatography

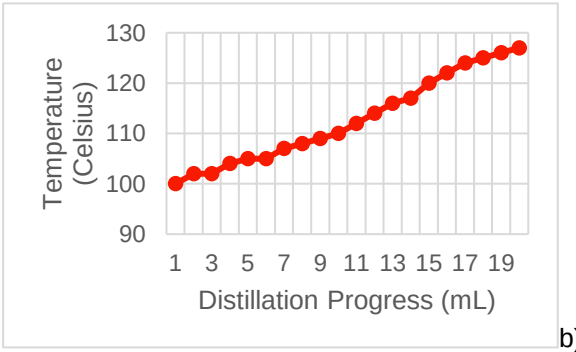
Introduction

The objective of this experiment was to separate 2 unknown alcohols using simple and fractional distillation methods. The resulting distillations were then analyzed via IR spectroscopy, gas chromatography, and boiling point analysis to determine the identity of the unknowns.

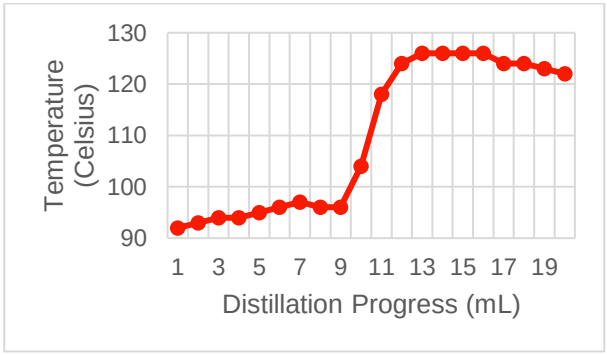
Distillation is a very common method of separating compounds based off of their boiling points. Distillation is used in the production of liquors, where pure alcohol is extracted from the rough product, so that the final product contains more alcohol than it would otherwise be able to.

Results

a)

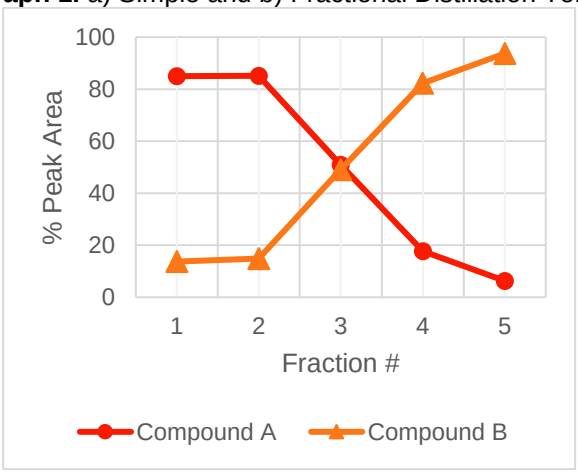


b)

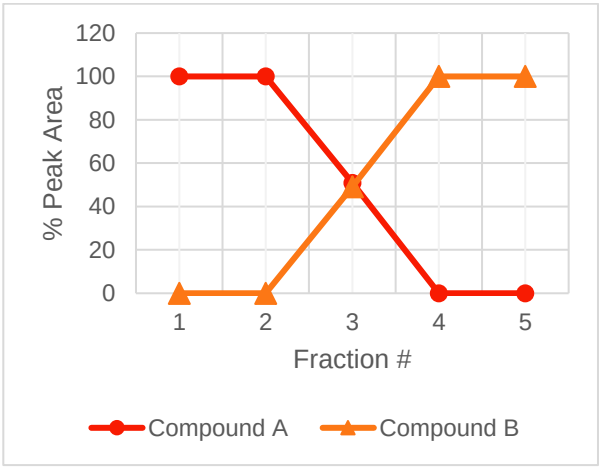


Graph 1. a) Simple and b) Fractional Distillation Temperature Curves.

a)



b)

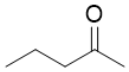


Graph 2. Composition of Fractions Analyzed by GC in a) Simple and b) Fractional Distillation.

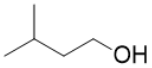
Scientific Principles

1. Fractional distillation drastically increases the surface area the gas must interact with while travelling up the column, and this large heatsink will cool and condense vapors as they try to move up the column, so that it will be far more difficult for the less volatile compound to escape the column before the more volatile compound. Fractional distillation has the effect of performing many distillations, which separates the compounds more completely.
2. 2-Pentanol has the lower GC retention time, as the mobile phase used in gas chromatography is an inert gas, meaning that less polar molecules will travel with the inert gas faster. Since 2-Pentanol is less polar than 3-Methyl-1-butanol, it travels through the GC faster, meaning it has the lower retention time. 3-

Methyl-1-butanol interacts more with the polar stationary phase due to its O-H bond, which makes it travel slower, increasing its retention time.



2-Pentanone



3-Methyl-1-butanol

3. Recrystallization is a useful purification method for compounds that are solid at room temperature, and are soluble in a (usually aqueous) solution, and it is for isolating compounds based off of polarity. Sublimation is very good at isolating a single compound due to the specificity of the triple point of a compound, but its use is limited by the feasibility of recreating the specific conditions needed for sublimation. Distillation is a purification method for compounds that are liquid at room temperature, and it separates compounds based on their volatility (aka boiling point).

Data Analysis

Yes, the objectives of this experiment were met, as the 2 compounds were able to be separated into pure fractions using fractional distillation, and they were able to be identified using IR and boiling point data as 2-Pentanol and 3-Methyl-1-butanol. Note: the unknown mixture was labeled **Unknown #2**.

The fractions obtained by the fractional distillation contained pure compounds, and this is reflected in the GC data obtained for these fractions. These GC graphs each had only one peak, as opposed to 2 peaks, meaning that there was only one compound to be measured in those fractions. The simple distillation method yielded GC data with 2 peaks of varying strength for all fractions, indicating that none of the fractions obtained in the simple distillation were pure. This is reflected in graphs 2a and 2b, where, except for the middle fraction, all data points in 2b, the fractional distillation data, were at either 100% or 0%. Clearly, fractional distillation is far more effective at separating the compounds than simple distillation.
For the 2 peaks, the shorter retention time was 0.999 min, and the longer retention time was 1.792 min. For the fractional distillation, the shorter retention time was the only peak present in the lower boiling point fractions, and the longer retention time was the only peak present in the higher boiling point fractions.

For the IR spectra, the more volatile compound had region 1 medium peaks at 2963 cm^{-1} and 2877 cm^{-1}, which were indicative of C-H bonds, and a strong, sharp region 3 peak at 1713 cm^{-1}, which was indicative of a C=O double bond. Given the estimated boiling point of 96 degrees C of the compound from fractional distillation, 2-Pentanone was the closest match.
The less volatile compound had an IR spectra with a broad medium peak in region 1 at 3324 cm^{-1}, which is indicative of an O-H bond, and strong, sharp region 1 peaks at 2930 cm^{-1} and 2861 cm^{-1}, which are indicative of C-H bonds. Given that the estimated boiling point was 126 degrees C in the fractional distillation, the best match was 3-Methyl-1-butanol.

Conclusion

The fractional distillation fractions on either end of the distillation all contained only a single peak in their GC data, indicating that they were all pure samples. Alternately, all simple distillation fractions had 2 peaks of varying strengths, indicating only partial separation, and demonstrating how fractional is a more effective means of distillation. Based on the boiling point estimates from the fractional distillation data as well as the IR spectra of the fractional distillation fractions, the unknown compounds were identified as 2-Pentanone and 3-Methyl-1-butanol.