

The distillation of an unknown compound



The Distillation of an Unknown Compound INTRODUCTION Distillation is the process heating a liquid to form vapor which is then collected when cooled and separated from the original liquid. The purpose of this experiment was to determine the identity of an unknown substance using simple and fractional distillation. Gas chromatography analysis the components of a mixture by separation of the materials by their different boiling points. EXPERIMENTAL The simple distillation apparatus was assembled using a 10-mL round bottom flask, distillation head, thermometer, adapter, thermometer, and condenser as shown in the Figure 1 (below). A boiling chip was added into the round bottom flask and 7.0 mL of the unknown mixture #2 was added. Figure 1 The mixture was heated to a boil. The first drop was recorded at 67° C. After the first 0.5 mL of distillate, the vapor temperature at every .5-mL interval was recorded and a graduated cylinder was used to measure the volume. Table 1 shows that the final sample temperature was at 80.5°C. After we collected 1.0 mL of distillate, this liquid was transferred into a vial and labeled the vial with the group name. After 3.5 mL of distillate was collected in was placed inside of a dry conical vial. Afterward, the remainder of the distillate was collected. This is the final sample. The fractional distillation was setup and this process was repeated using a distillation column. The first drop according to Table 2 was recorded at 70° C. The final sample temperature for the fractional distillation was 79° C. One microliter of sample was injected into the gas chromatograph. " Typical response factors for the possible components are as follows: hexane (1.50), cyclohexane (1.80), heptane (1.63), toluene (1.41), ethyl benzene (1.00). " DISCUSSION Table 1 Simple Distillation Boiling Points | 1st Drop | 67° C | .5 mL | 69° C | 1.0 mL | 71° C | 3.5 mL | 80.5° C | Table 2 Fractional Distillation Boiling Points | 1st

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Drop | 70° C | . 5 mL | 72° C | 1. 0 mL | 82° C | 3. 5 mL | 79° C | Table 3
Parameters on Gas Chromatographer | Column Type | 20% | He Flow Type |
50 L/m | Injector Temperature | 200° C | Detector Temperature | 195° C |
Column Temperature | 120° C | Graph 1 The data shows that the substances
in unknown compound #2 had similar boiling points. Between the initial
samples of the simple and fractional distillation differed three degrees
Celsius. Between the final samples of the simple and fractional distillation
differed 1. 5 degrees Celsius. Analyzing the natural boiling points of the
given known compounds, we saw that the compound hexane was similar to
unknown compound #2. Hexane has a boiling point of 69° C and Table 1
shows that the initial sample had a boiling point range of 69° C. Graph 1
shows the results of both simple and fractional distillation trials. Table 3
shows the parameters of the gas chromatographer. Analysis of the Chrom
Perfect Chromatogram Report, the simple initial has three peaks, the highest
reading at . 37 millivolts. This response is indicative of hexane. Analysis of
the simple final chart shows that three peaks were made. The two of the
highest of the three points were between . 39 to . 50 millivolts. The fractional
initial chromatogram report shows three peaks, however the middle peak
was the highest, reaching . 39 millivolts. This once again indicates a hexane
presence in the unknown. The fractional final, however, only had one peak,
and this peak was at . 49 millivolts. According to the text, this means that
heptane is present in the compound as well. This data presents that there is
hexane and heptane in Unknown Compound #2. CONCLUSION It was
concluded that Unknown Compound #2 was composed of both hexane and
heptane. This was concluded by the analysis of the known boiling points and
the data collected from both trials. The analysis of the chromatogram reports
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also confirmed the results. REFERENCES Anne B. Padãas, Making the Connections: A How-To Guide for Organic Chemistry Lab Techniques. 2nd edition. (Distillation (pp. 141 — 154) Chromatography, general (pp. 162 — 163) Gas Chromatography (pp. 179 — 187)) Answers to Questions 1. Simple distillation is used when the boiling points of two different substances are very different. Fractional distillation is the separation of a mixture of multiple components into its various fractions according to the difference in their boiling temperatures. a.) Hexyl acetate and butyl acetate- Simple distillation should be used for these two compounds because their boiling points are very different. b.) Hexanol and cyclopentanol- Fractional distillation should be used because there is a small difference of boiling points between these two compounds. 2. 3. (a) In a vacuum distillation system, boiling occurs when the vapor pressure of a liquid surpasses the ambient pressure. Once the pressure drops, the boiling point decreases, and the liquid is quickly sucked from the distillation flask through the condenser into the flask, and then into the pump. (b) (1 atm = 760 torr) Vapor pressure is higher than atmospheric pressure, so the boiling point would be higher.