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CHEM 3321-181

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Experiment 8 Prelab: Distillation: Separating a Mixture of Two Liquids

Introduction: The purpose of this experiment to purify a compound by separating the volatile part from the less-volatile part using distillation. Another purpose is to separate two miscible liquids using their different boiling points.



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| Name | MW (g/mol) | MP(oC) | BP(oC) | Density (g/mL) | Solubility | Hazards |
| Acetone | 58.08 | -95.35 | 56.2 | 0.79 | s water | Hazardous is case of skin or eye contact, ingestion, flammable |
| Ethyl Acetate | 88.11 | -83 | 77 | 0.902 | s water, acetone | Hazardous in case of ingestion or inhalation, slightly hazardous in case of eye or skin contact, flammable |

Procedure:

1. Obtain a 1:1 25 mL (12.5 mL of each) mixture of acetone-ethyl acetate, add a couple of boiling chips.
2. If using the fractional method, obtain a fractionating column.
3. Set up the distillation apparatus as illustrated in 8-3, grease all the glass-on-glass joints using a grease syringe and Keck clips.
4. When setting up the apparatus, the lab jack should be raised up a few centimeters from its lowest position to allow for easy cooling of the round-bottom flask.
5. Set the stirring hotplate to a heating power of 5, when the mixture begins boiling, adjust the setting as necessary so that the distillate collects at a rate of 1-2 drops/sec.
6. As the distillation proceeds, collect the liquid in a 10 mL graduated cylinder, take a temperature reading about every 1.0 mL. When 5 mL have been collected, remove the grad cylinder and substitute it with a sample vial, collect 10 drops. Save this sample for GC analysis.
7. Replace the graduated cylinder back under the vacuum adaptor and continue collecting.
8. During this time, run the first sample on the GC.
9. Continue recording the temperature of the vapors every 1.0 mL. After 10 mL have been collected, empty the graduated cylinder into a beaker, after 20 mL have been collected, replace the graduated cylinder once again with a sample vial and collect 20 drops.
10. Discontinue the distillation by lowering the stirring hotplate and lab jack, turn off the heat and allow the apparatus to cool and collect GC data.
11. Run a Gas Chromatography for each of the samples collected.
12. Disassemble the apparatus one it is cool, remove the grease by pouring some hexanes or acetone onto a Kimwipe, and then wiping out the glass joints.