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3. Purification of Liquids by Distillation

3.1. Competent Chemist Rating: “How Did the Peach Get in the Banana?”

Techniques Checklist:

- Setting up distillation glassware correctly
- Performing atmospheric pressure distillations
- Using Gas Chromatography (GC) to analyze samples

Pre-lab Discussion and Required Reading:

- Theory of distillation: Zubrick Ch. 36, LLP Ch. 11.3
- Distillation glassware and how to set it up: Zubrick Ch. 20
- Use of the GC: Zubrick Ch. 32

Equipment:

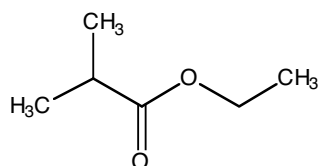
- Round-bottomed flasks (1x25-mL, 1x50-mL)
- Magnetic stir bar
- Distillation kit (distillation head)
- Ground glass thermometer
- Keck clips
- Glass wool and aluminum foil (optional)
- Heating mantle (w/ sand) and variac

Goal:

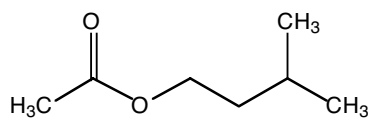
- To purify a mixture of two liquids using distillation.

Experiment Outline:

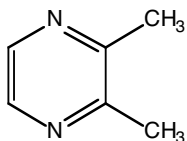
- You will receive a vial containing 11.20 g of a mixture of 2 compounds whose boiling points differ by about 40 °C (See possible compounds below.).
- Analyze the mixture using the GC - *see GC Sample Preparation and GC Operation Guides.*
- Perform atmospheric pressure distillation - *see Distillation Guide.*
- Prepare a GC sample of your purified low-boiling product.
- Obtain a mass and a gas chromatogram of your purified low-boiling compound.



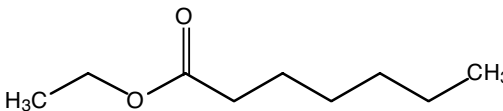
Ethyl Isobutyrate (Peach)
(BP 112–113 °C)



Isoamyl Acetate (Banana)
(BP 142–143 °C)



2,3-Dimethylpyrazine
(BP 156 °C)



Ethyl Heptanoate
(BP 188–189 °C)

Helpful Hints:

- Make sure all of your joints are lightly greased and sealed well. Otherwise, you will lose your product into the atmosphere.
- Insulate your distillation head with cotton and foil to speed things up.
- Do not heat your mixture too fast, or your entire sample will end up in your collection flask.
- Be aware that the temperature reading on the thermometer may not correlate accurately with the boiling point of the distilling liquid.

Results:

• To obtain your "CC Rating" in Purification of Liquids by Distillation, you must obtain at least 7.00 g of the low-boiling material that is 92% pure or better as determined using GC analysis. You must also correctly identify the two components of your mixture. Think boiling points and smell!