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Simple Distillation (rev. 6/05/13)

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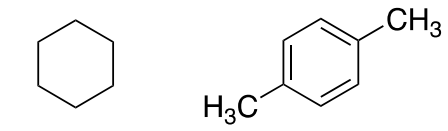
The most popular method for separating miscible compounds with different boiling points is by simple distillation. The impure liquid is placed in a round-bottom flask and fitted with a distillation head, which is in turn attached to a condenser. The end of the condenser is placed over a collection vessel in which the distillate is collected. The flask is heated, and the liquids will begin to vaporize (the lowest boiling first). The vapor rises through the distillation head and passes into the condenser. In the condenser the vapor is cooled by the running water causing it to condense into the liquid phase and drip into the collection flask. Distillations can be monitored by observing the temperature at the entrance to the condenser; as the apparatus heats up, the vapor from the first component (with the lowest boiling point) will reach the thermometer and enter the condenser while the higher boiling point components condense lower in the apparatus. The temperature will remain constant until all of the first liquid has been removed at which point the temperature will increase and plateau again until the second liquid is distilled away; this pattern will repeat until all liquids have been distilled.

Things to note:

- Work in the fume hood! This is a volatile and toxic mixture – plus, it smells awful.
- Never boil the flask to dryness**.**
- **NEVER PLUG THE HEATING MANTLE DIRECTLY INTO THE OUTLET. USE A VARIAC.**



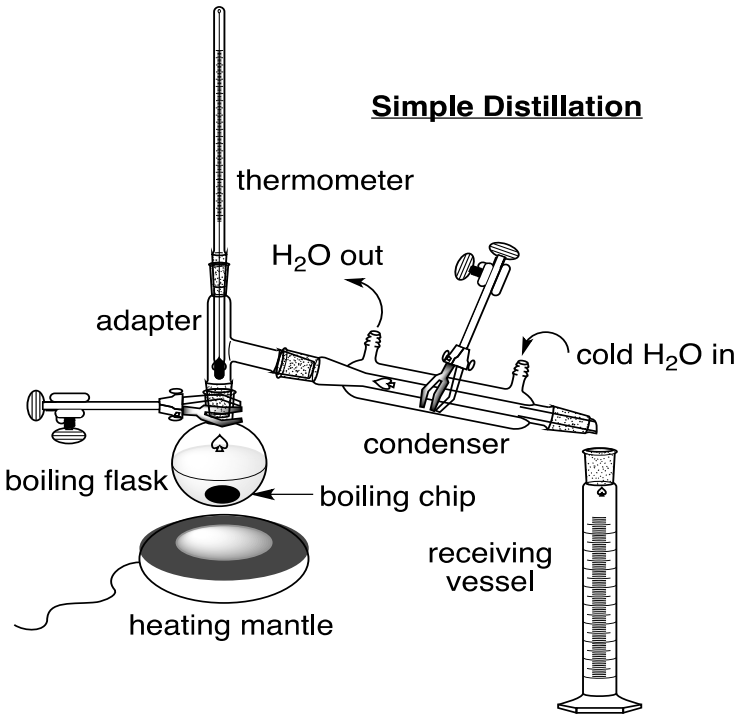
cyclohexane *p*-xylene

In this lab, you will distill a mixture of cyclohexane and *p*-xylene. The success of a distillation can be evaluated by collecting temperature and spectroscopic data as the fractions are being separated. A good distillation will produce a temperature vs. fraction graph with a plateau for each compound collected and a steep rise in temperature between fractions and spectroscopic data will show little or no contamination from neighboring fractions.

PROCEDURE:

1. Place 24 mL of the prepared 50/50 mixture of cyclohexane and *p*-xylene in a 50 mL round bottom flask; add a few boiling chips.

2. Assemble the apparatus as shown. To ensure accurate temperature readings, make sure that the thermometer bulb is placed even with the joint in the adapter (as shown below). Wrap the top of the flask and the distillation head in aluminum foil to prevent heat loss. Use a 25 mL graduated cylinder as the initial receiving flask.



3. Turn on the heating mantle. Use a low voltage setting at first (~40), then increase as needed about every 5 minutes. Heating mantles respond slowly, so be patient. Adjust the heat until the distillate is collected at a rate of about one drop every 2-3 seconds.

4. Record the temperature after each 1 mL collected. Continue recording the temperature until a total of 20 mL have been collected. Collect the 5th and 20th mL in a test tube and acquire ^{1}H-NMR using the picoSpin 45 spectrometer.

5. Plot a graph of temperature vs. mL collected. Use this graph to determine the boiling points of the two liquids.

6. Using the integration values from your NMR spectra, determine the relative concentrations of cyclohexane and p-xylene in the 5th and 20th mL fractions.