

Reminder: These notes are meant to supplement, not replace, the textbook and laboratory manual.

Fractional Distillation

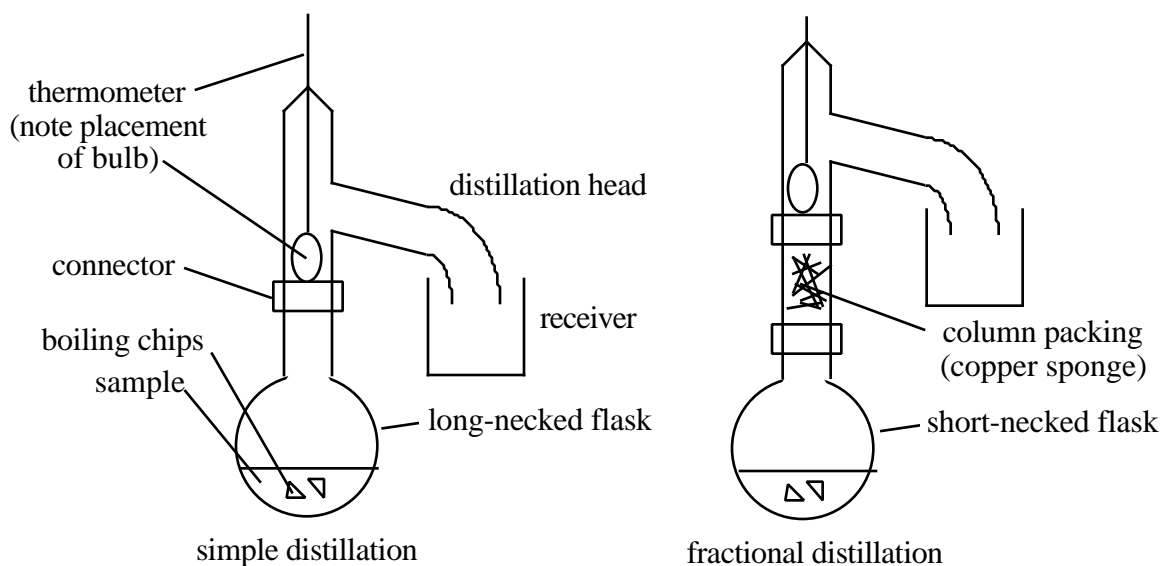
1. First, study the notes for the Boiling Point and Refractive Index experiment. You are responsible for that material as well as that given below.
2. In practice, simple distillation is carried out without any column packing, while fractional distillation does use column packing such as the copper sponge in this experiment. In theory, simple distillation involves as few as one theoretical plate (see below), while the fractional distillation apparatus gives many of them, for a more effective separation.
3. Here is some terminology related to simple and fractional distillation.

In reflux, liquids boil in the lower part of an apparatus, condense in the upper part, and drip down to the lower portions again.

A theoretical plate is a cycle of evaporation and condensation in a distillation. The number of theoretical plates is a measure of the ability of a distillation to separate substances. Fractional distillations have many theoretical plates; true simple distillations have only one.

Azeotrope: A mixture of liquids that distills at a constant temperature without changing composition. The best-known azeotrope is 95.5% ethanol and 4.5% water.

4. Here are rough drawings, not to scale, of the setups used for simple and fractional distillation. The simple distillation apparatus is shown for comparison purposes.



If you place the thermometer bulb too high, the vapors won't reach it before they go into the sidearm to be collected, and your observed boiling point will be lower than it should be. If you place the thermometer bulb too low, vapors of impurities may reach it, giving a high reading for the boiling point range.

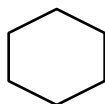
Superheating most often happens when you leave out the boiling stones. Boiling of a superheated sample can be irregular and even explosive.

Many organic compounds react with oxygen in the air over time to give low concentrations of peroxides. Peroxides are unstable; distillation of such samples to dryness may

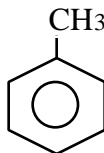
cause them to explode.

If you try to distil in a closed system (this most often happens when you use a connector to attach your receiver), the pressure of the heated gases inside the apparatus will cause a rupture. Most often you'll launch your thermometer.

5. The structures of the compounds used in this experiment are:



cyclohexane



toluene

6. Safety considerations for this experiment include:

A sand bath, especially the top portion, can get very hot during the experiment, and you won't be able to tell by looking. Handle the sand bath with care. Also, a sand bath is an electrical apparatus; notify your instructor if its wires are frayed or exposed, and don't splash water on it.

You'll be using a mercury thermometer. These are fragile--handle them carefully, especially when you're installing them in the top of the distillation apparatus. If you break a thermometer and spill mercury, notify your instructor, who has a special kit for cleaning up a mercury spill.

Toluene and cyclohexane are both highly flammable and slightly toxic. Our lab equipment and safety regulations are selected to avoid flames. Also, avoid direct contact with these compounds.

Notes for topics that may be included in quizzes given after the experiment:

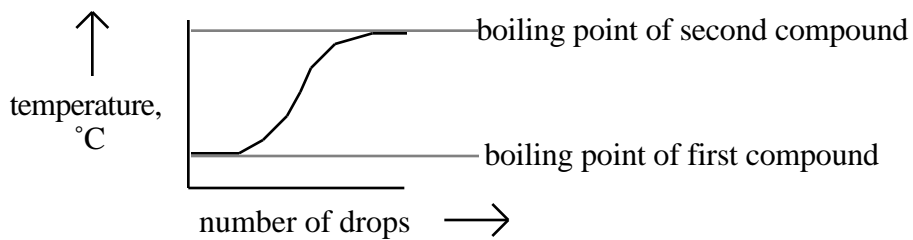
8. Here are factors that chemists consider when deciding what type of distillation to use:

Simple distillation is primarily used to separate compounds that have very large differences in boiling point, usually more than 100° C. Example: acetone (b. p. 57° C) can be separated from dodecane (C₁₂H₂₆, b.p. 216° C) using simple distillation.

Fractional distillation is used to separate compounds that have boiling points that are closer to each other. The compounds separated in this experiment are an example.

Vacuum distillation is used to separate and purify compounds that have very high boiling points, as well as those that decompose or react with air at temperatures near their boiling points.

9. Here is a sample distillation curve for a sample containing two compounds. The method for determining the boiling point is shown on the graph.



The curve for a fractional distillation is better defined than that for a simple distillation, as shown in the next graph.

