A TECHNIQUE EXPERIMENT

In a technique experiment, you get to practice a certain operation *before* you have to do it in the course of a synthesis. Distilling a mixture of two liquids to separate them is a typical technique experiment.

Read the following handwritten notebook pages with some care and attention to the *typeset* notes in the margin. A thousand words are worth a picture or so (Figs. 2.1–2.3).

Notebook Notes

1. Use a descriptive title for your experiment. *Distillation.* This implies you've done *all* there is in the *entire* field of distillation. You haven't? Perhaps all you've done is *The Separation of a Liquid Mixture by Distillation.* Hmmmmmm.
2. Writing that first sentence can be difficult. Try stating the obvious.
3. There are no large blank areas in your notebook. Draw sloping lines through them. Going back to enter observations after the experiment is over is *not professional.* Initial and date pages anytime you write anything in your notebook.
4. Note the appropriate changes in verb tense. Before you do the work, you might use the present or future tense when you write about something that *hasn't happened yet.* During the lab, since you are supposed to write what you've actually done just after you've actually done it, a simple past tense is sufficient.

Numbered Page 6

This is a few days before lab. 01/20/03

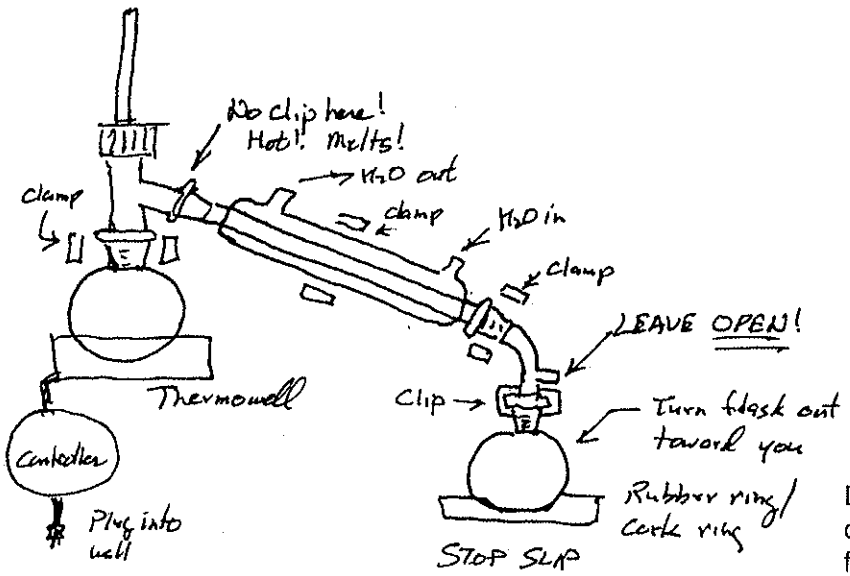
Explanatory Title

The Separation of a Liquid Mixture by Distillation

Distillation is one of the methods of separation and purification of liquids. We will be given an unknown liquid mixture and will have to separate it by distillation

It's often hard to start. Hint: State the obvious.

After we get the unknown, we shall ~~dry it with~~ immediately dry the liquid over anhydrous magnesium sulfate The setup is as detailed in the laboratory manual with some changes:



Local procedure change, probably from handout

We will be using Thermowell heaters + controllers and not Variacs. Vacuum adapter clamped at angle, rotated toward me in order to make it easy to change flasks.

01/20/03 JWZ

FIGURE 2.1 Notebook entry for a technique experiment (1).

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Separation of a Liquid Mixture (cont'd)

Obtained liquid unknown #20 from instructor and dried it over a slight XS of anhydrous magnesium sulfate. Set up distillation apparatus as described (p.6). Started with the smallest flask to collect fore-run as suggested by instructor. Filtered unknown into distilling flask with long stem funnel. Set heat controller to 40.

Instant modification

Mixture finally beginning to boil!

Liquid condensed on thermometer & temperature reading shot up to 79°C and stabilized at 81°C in a few seconds. Collected ≈ 2 mL as fore-run. Will discard this later. Dropped Thermowell to remove heat to stop distillation and change receiving flasks. Started heating again.

Do a bit of work and write a bit of text.

Collected liquid boiling from 81 to 83°C. Changed receiver as above. When new material came over, thermometer read 82°C (!) for a few minutes (mL) the distillation stopped. Temperature began dropping (!) Turned heat up (60) and mixture started boiling again – liquid came over @ 123°C. Collected a little of this and changed receivers again. Added fresh boiling stone each time heating stopped. Hard to label flasks. So many of them.

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FIGURE 2.2 Notebook entry for a technique experiment (2).

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Separation of a Liquid Mixture (con't).

Flask #	Contents
1	fore-run
2	81°C–83°C fraction
3	82°C–123°C change-over
4	120°C–123°C fraction
5	>123°C residue

Small amount of liquid left-over in the boiling flask can't get over. Dangerous to heat to dryness. Stopped distillation after collecting fraction from 120–123°C (Flask #4)

Cooled distilling flask and poured contents into a 50 ml Erlenmeyer (Flask #5)

Cleaned cork stoppers for security & have permission to store flasks, properly labelled, in hood until next lab.

JWZ 01/23/03

FIGURE 2.3 Notebook entry for a technique experiment (3).