A **research notebook** is perhaps one of the most valuable pieces of equipment you can own. With it you can duplicate your work, find out what happened at leisure, and even figure out where you blew it. General guidelines for a notebook are:

1. The notebook must be bound permanently. No loose leaf or even spiral-bound notebooks will do. It should have a sewn binding so that the only way pages can come out is to cut them out. (8 ½ x 11 in. is preferred). **Duplicate carbon notebooks** are available that let you make a carbon copy that is removable and that you can hand in. And if the pages aren't already numbered, you might want to do it yourself.
2. *Use waterproof ink! Never pencil!* Pencil will disappear with time, and so will your grade. Cheap ink will wash away and carry your grades down the drain. Never erase! Just draw *one* line through ~~yuor errers~~ your errors so that they can still be seen. And never, never, never cut any pages out of the notebook!
3. Leave a few pages at the front for a table of contents.
4. Your notebook is your friend, your confidant. Tell it:
   a. What you have done. Not what it says to do in the lab book. What you, yourself, have done.
   b. Any and *all* observations: color changes, temperature rises, explosions..., anything that occurs. Any *reasonable* explanation *why* whatever happened, happened.
5. Skipping pages is *extremely* poor taste. It is **NOT** done!
6. List the IMPORTANT chemicals you'll use during each reaction. You should include USEFUL **physical properties:** the name of the compound, molecular formula, molecular weight, melting point, boiling point, density, and so on. You might have entries for the number of moles and notes on handling precautions. Useful information, remember. The *CRC Handbook of Chemistry and Physics*, originally published by the Chemical Rubber Company and better known as the *CRC Handbook*, is one place to get this stuff (see Chapter 3, "Interpreting a Handbook").

Note the qualifier "USEFUL," if you can't use any of the information given, do without it! You look things up *before* the lab so you can tell what's staring back out of the flask at you during the course of the reaction.

Your laboratory experiments can be classified as either of two major types: a technique experiment or a synthesis experiment. Each requires different handling.

## 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. 2-4).

### 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 the time you've actually done it, a simple past tense is sufficient.

## A SYNTHESIS EXPERIMENT

In a synthesis experiment, the point of the exercise is to prepare a clean sample of the product you want. All the operations in the lab (e.g., distillation, recrystallization) are just means to this end. The preparation of 1-bromobutane is a classic synthesis and is the basis of the next series of handwritten notebook pages.

From: Zubrick "The Organic Chem Lab Survival Manual"

Explanatory title

Numbered page

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9/13/86

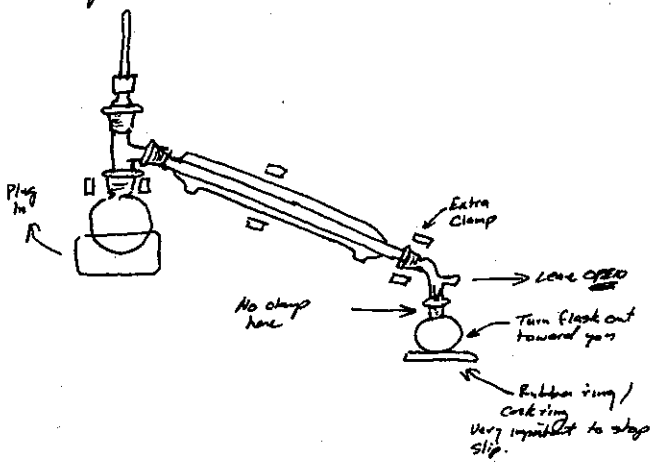
## The Separation of a Liquid Mixture by Distillation

This is the Saturday before lab.

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, should ~~do~~ ~~not~~ ~~wash~~ ~~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 and will not need Variacs. Vacuum adapter clamped at angle, rotated toward me in order to make it easy to change flasks.

9/13/86 Jay

*Fig. 2* Notebook entry for a technique experiment (1).

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## Separation of a Liquid Mixture (cont'd)

Instant modification.

Obtained liquid unknown #20 from instructor & 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 50. Mixture beginning to boil.

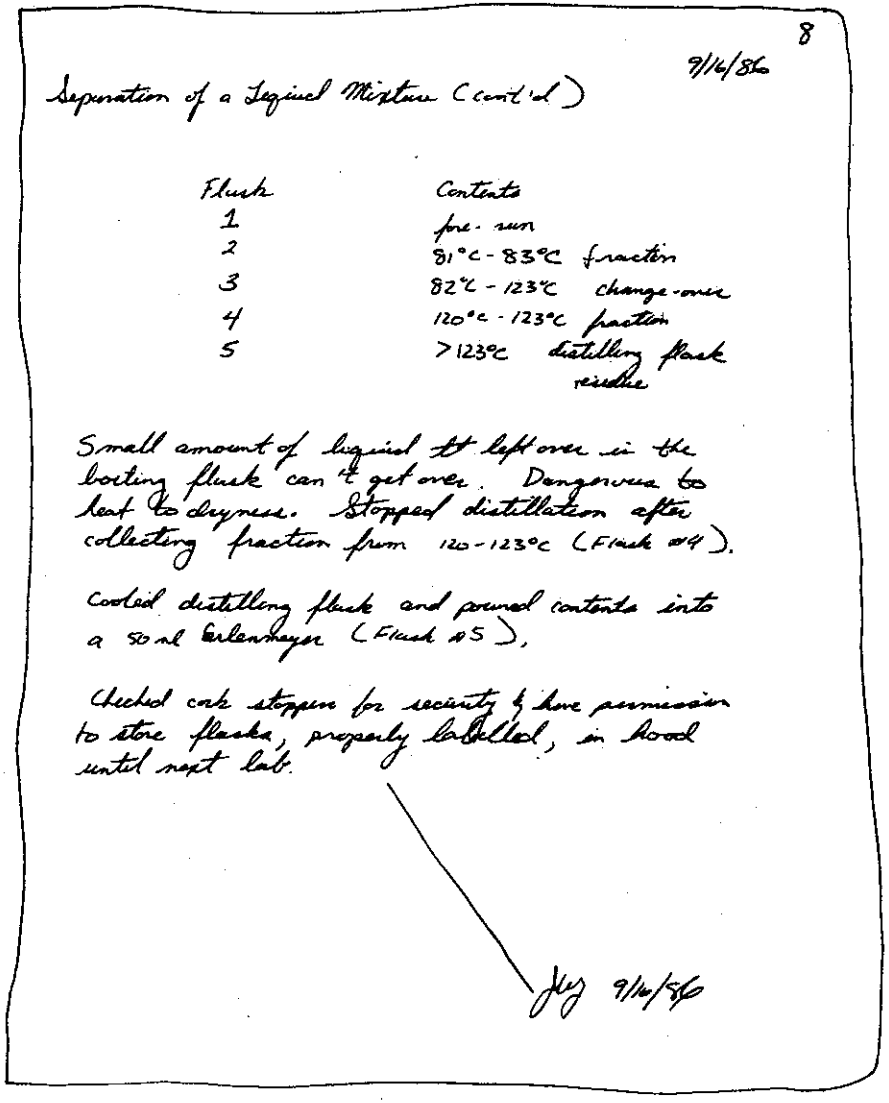
Do a bit of work and write a bit of text.

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.

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) then distillation stopped. Temperature began dropping! Turned heat up (70). Mixture starting to boil again and liquid came over @ 123°C. Collected a little of this then changed receiver as above. Shook distilling flask a little & added boiling stone before heating. Had to label flasks. So many of them.

9/16/86 Jay

*Fig. 3* Notebook entry for a technique experiment (2).



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Separation of a Liquid Mixture (cont'd)

| Flask | Contents |
|---|---|
| 1 | pre-run |
| 2 | 81°C - 83°C fraction |
| 3 | 82°C - 123°C change-over |
| 4 | 120°C - 123°C fraction |
| 5 | >123°C distilling flask residue |

Small amount of liquid ~~it~~ 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).

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

JKJ 9/14/86

***Fig. 4*** **Notebook entry for a technique experiment (3).**

## Notebook Notes

1. Use a descriptive title for your experiment. *n-Butyl Bromide.* So what? Did you drink it? Set it on fire? What?! *The Synthesis of 1-Bromobutane from 1-Butanol*—now *that's* a title.
2. Do you see a section for unimportant side reactions? No. Then don't include any.
3. In this experiment, we use a 10% aqueous sodium hydroxide solution as a wash (see Chapter 15, "Extraction and Washing") and anhydrous calcium chloride as a drying agent (see Chapter 10, "Drying Agents"). These are not listed in the Table of Physical Constants. They are neither reactants nor products. Every year, however, somebody always lists the physical properties of *solid* sodium hydroxide, calcium chloride drying agent, and a bunch of other reagents that have nothing to do with the main synthetic reaction. I'm especially puzzled by the listing of solid sodium hydroxide in place of the 10% solution.
4. **Theoretical yield** (not yeild) calculations always seem to be beyond the ken of a lot of you, even though these are exercises right out of the freshman year chemistry course. Yes, we do expect you to remember some things from courses past, the least of which is where to look this up. I've put a sample calculation in the notebook (Fig. 6), that gets the mass (g) of the desired product (1-bromobutane) from the volume (ml) of one reactant (1-butanol). Why from the 1-butanol and not from the sulfuric acid or sodium bromide? It's the 1-butanol we are trying to convert to the bromide, and we use a **molar excess** (often abbreviated XS) of everything else. The 1-butanol is, then, the **limiting reagent:** the reagent present in the smallest molar ratio. Note the use of the density to get from volume to mass (ml to g), molecular weight to go from mass to number of moles (g to mol), the stoichiometric ratio (here 1:1) to get moles of product from moles of limiting reagent, and, finally, reapplication of molecular weight to get the mass (g) of the product.

Note that this mass is *calculated*. It is NOT anything we've actually produced. In theory, we get this much. That is theoretical yield.

5. I'm a firm believer in the use of units, factor-label method, dimensional analysis, whatever you call it. I know I've screwed up if my units are (g 1-butanol)$^2$/mole 1-butanol.
6. Remember the huge writeup on the *Separation of a Liquid Mixture by Distillation*, drawings of apparatus and all? Well, the line "the mixture was purified by distillation" (Fig. 9) is all you write for the distillation during this synthesis.
7. At the end of the synthesis, you calculate the **percent yield**. Just divide the amount you *actually prepared* by the amount you calculated you'd get, and multiply this fraction by 100. For this synthesis, I *calculated* a yield of 25.44 g of product. For this reaction on the bench, I *actually* obtained 16.2 g of product. So:

$$(16.2\text{ g}/\ 25.44\text{ g})(100) = \mathbf{63.6\%\ yield}$$

Note that's not 63.624715321%. **Significant figures**, please. The product is weighed to one part in ten ([+| 0.1) and calculated to one part in one hundred ([+| 0.01). If the product weight can vary by [+| 0.1 g, what's the use of all those figures?

## THE ACID TEST

After all this, you're still not sure what to write in your notebook? Try these simple tests at home.

1. Before lab. "Can I carry out this experiment without any lab manual?"
2. After lab. (I mean *immediately* after; none of this "I'll write my observations down later" garbage.) Ask yourself: "If this were someone else's notebook, could I duplicate the results exactly?

If you can truthfully answer yes to these two questions, you're doing very well indeed.