

THE LABORATORY NOTEBOOK

Introduction

A laboratory notebook serves several purposes. The first is for your own reference. To avoid forgetting any important information, the details of each experiment will be recorded in the notebook. The second purpose is so that someone else can review your work and repeat it exactly. This facet of experimental work is necessary in research, and it is also necessary in a student laboratory. If a particular experiment does not work well for you, the instructor would want to know why. Your detailed written procedure and observations can give clues to what happened if an experiment fails.

Below, you will find one format for keeping a laboratory notebook. Your instructor may have a different format for you to follow. Make sure you clearly understand the instructions before you begin writing in your notebook.

The Correct Notebook

The correct notebook for the laboratory is a bound book containing lined pages. These are available at bookstores and stationary supply stores. A loose-leaf or spiral notebook is not satisfactory because the pages are easily lost.

Keeping the Notebook

If the pages in the notebook are not numbered, number them before using the book. Write your name and laboratory section number on the cover of your notebook.

Enter experiments consecutively in ink; use permanent blue or black ink because your book will become splashed and stained with use.

Your notebook can become cluttered and illegible if an organized format is not observed. For this reason, certain conventions have been developed.

At the top of the page, write the date on which the experiment was performed. As you go along, leave plenty of space for notes that you might want to insert later. If you will be running a distillation or determining more data for a particular experiment, be sure to leave blank pages as necessary before writing the procedure for the next experiment.

Write clearly so that your instructor will be able to read and grade your notebook. If you make errors, DO NOT RIP OUT THE PAGE OR USE WHITEOUT. Instead, ~~line-out~~ errors (or draw an "X" over the entire page) and go on.

Entering Experiments

You will be doing two types of experiments in the organic lab: *investigative experiments* and *preparative experiments*. Investigative experiments involve making observations and learning techniques that are common to laboratory work in organic chemistry but do not entail conversion of one compound into another. Some examples are thin-layer chromatography and recrystallization. Preparative experiments, in contrast, involve interconversion of different compounds.

The format of the laboratory notebook is usually different for these two types of experiments.

Notebook Format for Investigative Experiments

1. *Aim.* Give a brief aim of the experiment in which you clearly state the purpose(s) of the procedure. This should require no more than one-fourth of a page.
2. *Apparatus Required.* List all the apparatus you might require to carry out your experiment.
3. *Theory or Principle.* State the fundamental idea behind doing an experiment. For example, if your goal is to separate a mixture, then state the principle behind the separation—whether the separation technique is based on difference in solubility, or difference in boiling point, etc.
4. *Table of Reagents.* Whenever you use chemicals, include a table of reagents that lists the name, structure, molecular formula, molecular weight, melting/boiling points, density and quantity used (in g or mL as well as in moles) for each chemical you will use. For investigative experiments, you do not need to put in the amount of chemicals used, as it may be something like “1 drop”.
5. *Procedure.* List the steps involved in carrying out the experiment briefly, in points or by using a flow chart. Do not copy the experimental procedure from the book but provide a synopsis of it.
6. *Observations.* Enter a one- or two-line statement for each part of an experiment. Reserve sufficient room to record results as they are obtained.
7. *Discussion of Results:* Summarize your results and present your inferences based on your results and observations.
8. *Conclusions.* Record the conclusions that can be reached based on the results you have obtained in the experiment. If the procedure has involved identification of an unknown compound, summarize your findings in this section.

A sample write-up of an investigative experiment is given below.

Al Kane
Feb. 1, 2010

Separation of Green Leaf Pigments by TLC

Reference: *Organic Experiments* by Fieser and Williamson, Section 9.2.

AIM: The pigments in green leaves are to be extracted into an organic solvent, and the extract is to be analyzed by thin-layer chromatography (TLC).

APPARATUS REQUIRED: TLC Plates, TLC capillaries, TLC chamber, Ruler

THEORY: Thin layer chromatography is a method of separation of a mixture based on difference in polarities of the individual components making up the mixture. The presence of multiple spots on the developed TLC plate will indicate that more than a

single pigment is contained in the leaves. The R_f value is calculated as the ratio of distance moved by the solute to the distance moved by the developing solvent, and is

indicative of the relative polarities of the individual components. Lower the R_f value, higher the polarity and vice versa.

TABLE OF REAGENTS:

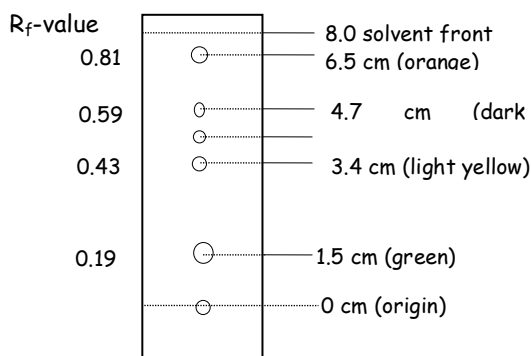
Name	Mol. Formula, struc.	Mol. weight	m.p/b.p °C	Density g/mL	Amount used	moles

Fill in all the reagents you will be using and include the appropriate properties along with the amounts you will be using.

PROCEDURE: Grind 5 spinach leaves in mortar and pestle with 5 mL of 2:1 pet. ether and EtOH. Swirl soln. with 3 x 2 mL portions of H₂O in sep. funnel; dry org. soln. for few min over anhyd. Na₂SO₄ in Erlenmeyer. Decant and concentrate soln. if not dark colored. Spot 10 cm x 2 cm TLC plate about 1.5 cm from end with dried extract; spot should be less than 2 mm diam. Develop plate with CHCl₃.

OBSERVATIONS AND RESULTS:

Procedure followed exactly as described in reference. Org. soln. was dark green in color; aq. extracts were yellowish. Half of org. layer lost. TLC plate had five spots having colors and R_f -values shown in the drawing below.



DISCUSSION OF RESULTS: Based on the developed TLC plate, the spinach extract is comprised of five individual components of which, orange colored component is the least polar and the green colored component is the most polar. There is an unidentified spot in between 0.43 and 0.59.

CONCLUSIONS

Based on TLC analysis, the procedure used allows the extraction of at least five different pigments from the spinach leaves. Judging from colors, one of these is a carotene, three are xanthophylls, and the last is chlorophyll *b*.

Notebook Format for Preparative Experiments

1. *Aim.* Same as for investigative experiments.
2. *Apparatus.* Sketch the apparatus set-up for elaborate experimental set-up.
3. *Main Reaction(s) and Mechanism(s).* Write balanced equations giving the main reaction(s) for conversion of starting material(s) to product(s). The reason for balancing the equations is given in Part 4 (below). If required by your instructor, include detailed mechanisms for the reactions that you have written.
4. *Table of Reactants and Products.* Set up a Table of Reactants and Products as an aid in summarizing the amounts and properties of reagents and catalysts being used and the product(s) being formed. Reagents, catalysts, and products that appear in the main reaction(s) should be listed in the table; along with other reagents that may be used in the work-up and purification of the reaction mixture.

The following items should appear in the table:

- (a) The name and/or structure of each reactant, catalyst, and product. (Rule of thumb: If it's organic, include the structure.)
 - (b) The molecular weight of each compound.
 - (c) The weight used, in grams, of each reactant and the volume of any liquid reactant.
 - (d) The molar amount of each reactant used. (Calculated from parts *b* and *c*.)
 - (e) The theoretical mole ratio expressed in whole numbers, for the reactants and products.
 - (f) Physical properties of the reactants and products; this might include bp, mp, density, solubility, color, and odor. What you include will depend on the particular experiment.
4. *Yield Data.* Compute the maximum possible amount of product that can be formed, called the **theoretical yield**. You may need to use your knowledge about "limiting reactant", if necessary. Once the isolation of the desired product(s) has been completed, you should also calculate the **percent yield**. Generally, the calculated value of percent yield is rounded to the nearest whole number. As a point of reference, most organic chemists consider yields of 90% or greater as being "excellent," and those below 20% as "poor."
 5. *Procedure and Notes.* Provide an outline of the experimental procedure in the left-hand column that contains enough detail so that you do not have to refer to the textbook repeatedly while performing the experiment. Note any variations and observations in the right-hand column that you make while carrying out the formation and isolation of the product(s).
 6. *Observed Properties of Product.* Record the physical properties of the product you have isolated in the experiment. This might include: bp, mp, odor, color, and crystalline form, if the product is a solid. Compare your observations to those available on the compound in various reference books (e.g., the *CRC Handbook of Chemistry and Physics* or the *Merck Index*).
 7. *Side Reactions.* List possible side reactions; those reactions leading to undesired products, which are likely to occur in the experiment. It is important to consider such processes because the process used to purify the desired product must remove the by-products that are

formed. You may need to consult your lecture notes and textbook in order to predict what side reaction might be occurring.

8. *Method of Purification.* Develop a flow chart that summarizes the sequence of operations that will be used to purify the desired product. The chart will show at what stages of the work-up procedure unchanged starting materials and unwanted by-products are removed. Considering the chemical and physical properties of both the desired and undesired substances does this.

9. Results and Discussion: Summarize your results and discuss any deviation from the norm and possible scientific explanations for the deviations.

A detailed example is provided below.

Al Kane
April 1, 2010

Dehydration of Cyclohexanol

Reference: *Organic Experiments* by Fieser and Williamson, Section 11.1.

INTRODUCTION

Cyclohexene is to be prepared by the acid-catalyzed dehydration of cyclohexanol.

MAIN REACTION(S) AND MECHANISM(S) (mechanism not provided here, but you should include it in your notebook)

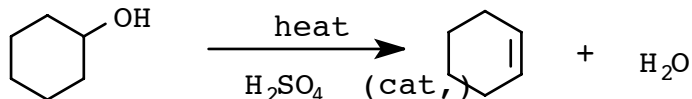


TABLE OF REACTANTS AND PRODUCTS

Fill in as said earlier.

Limiting reagent: cyclohexanol

YIELD DATA

Theoretical yield of cyclohexene

$$\begin{aligned} &= \text{moles of limiting reagent (cyclohexanol)} \times (1 \text{ mole} \\ \text{Cyclohexene formed/} & 1 \text{ mole cyclohexanol reacted}) \times \text{M.W. of cyclohexene} \\ &= 0.05 \text{ mol} \times (1/1) \times 82.2 \text{ g/mol} \\ &= 4.1 \text{ g} \end{aligned}$$

Actual yield = 2.7 g

Percent yield = $[\text{Actual yield(g)}/\text{theoretical yield(g)}] \times 100 = [2.7/4.1] \times 100 = 66\%$

PROCEDURE

Put alcohol in 25-mL rb flask and add H₂SO₄.

Swirl to mix and add 1 or 2 boiling chips.

Attach fractional dist. apparatus.

Heat with thermowell; heating rate such that temp. stays less than 90°C.

Stop heating when approx. 2.5 mL remain in reaction flask.

Put distillate in 25 mL Erlenmeyer and add 1-2 g K₂CO₃. Swirl mix for 15 min.

Transfer liquid to 10 mL rb with a pipette.

Add boiling chip and do simple dist (receiver must be near condenser). Collect product from 80-85°C.

NOTES

Done

Done

Done

Done, distillate cloudy, two layers in receiver

Done, liquid in still pot darkened as reaction proceeded

Done, K₂CO₃ caused evolution of a few bubbles (CO₂?). Had to add 0.5 g more to get rid of cloudiness. Left for one week.

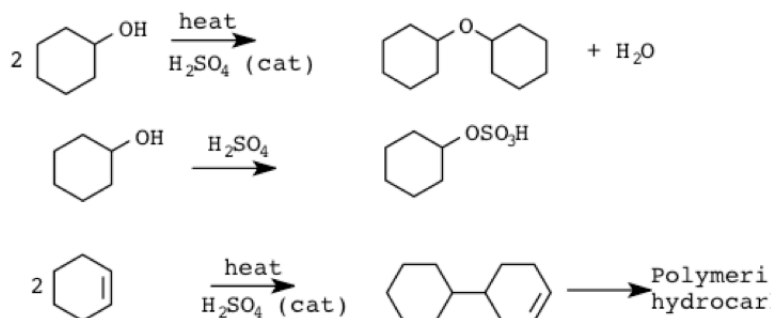
Done

Collected cyclohexene in ice-cooled 10 mL rb flask attached to vacuum adapter protected with CaCl₂ tube. Stopped distillation when about 1 mL of yellowish liquid remained in stillpot.

OBSERVED PROPERTIES OF PRODUCT

bp 80-84°C (760 torr); colorless liquid; insoluble in water; decolorizes Br₂/CCl₄ solution and produces brown precipitate upon treatment with KMnO₄/H₂O.

SIDE REACTIONS



FLOW CHART FOR PURIFICATION

