

Analytical Chemistry Laboratory Notebook Guidelines

I. Preliminaries

- A. You will be doing your lab work in pairs (or groups of three) all semester. On the cover of your notebooks, write your name and the name of your lab partner(s).
- B. You must use a bound laboratory notebook for recording all data and observations. Unless I explicitly tell you otherwise, you will present all analysis and discussion in your notebook as well.
- C. Always write in ink. Make any corrections by drawing a single line through the incorrect information. You should never white out or otherwise obliterate anything you write in your notebook.
- D. Leave the first page blank for a **Table of Contents** which lists each of the experiments and the pages the documentation for each occupies. Keep this Table of Contents updated!
- E. If your notebook does not already have page numbers, you need to add them to the upper right corner of each page. You should also write the date (including the year) you start writing on any page. If you write on a given page over more than one day, be sure to date those additional comments.
- F. It is best to write on only one side of each page. The paper in your notebooks is usually too thin to keep ink from bleeding through.

Note that you will lose points from your notebook score if any of the above mechanics are neglected.

II. Specifics

A. Before you get to lab:

- (1) At the top of a blank page, write the experiment's **Title** and a brief statement of its **Objective**.
- (2) Each person should draw a flowchart of the **Experimental Procedure** in his or her own notebook.
- (3) Your flowchart should be detailed enough that you could do the experiment without referring to the lab handout. As an example, look at the flowchart I outlined for a Macalester general chemistry experiment (next two pages).
- (4) Note that (unlike my example), you should leave space next to your flowchart to make notes during lab.

Brinton and I will check that each person has a flowchart at the start of lab. I will deduct 3 points (out of 25 total) if you have not already outlined your procedure.

The original procedure for a synthesis experiment. (Taken from Slowinski, E. J.; Wolsey, W. C.; Masterton, W. L. *Chemical Principles in the Laboratory*, 7th ed.; Harcourt: Fort Worth, 2001.)

A.1 Preparation of $\text{Co}_x(\text{NH}_3)_y\text{Cl}_z$ (Procedure I)

Using a piece of weighing paper and a top-loading balance, weigh out 10 ± 0.2 g of ammonium chloride, NH_4Cl . Pour the NH_4Cl into a 250-mL beaker and add 40 mL distilled water. To this add 8 ± 0.2 g of cobalt(II) chloride hexahydrate, $\text{CoCl}_2 \cdot 6 \text{H}_2\text{O}$ and 0.8 g activated charcoal (Norite). Heat this mixture to the boiling point, stirring to dissolve the soluble components.

Cool the beaker in water, then in an ice bath. Slowly, in a hood, add 40 mL 15 M NH_3 , ammonia.

CAUTION: This is a concentrated reagent, with a strong NH_3 odor. After stirring, add 50 mL 10% H_2O_2 , hydrogen peroxide, slowly, a few mL at a time, while continuing to stir. **CAUTION:** Be careful not to spill this reagent on your skin. Use gloves.

When the bubbling has stopped, place the 250-mL beaker in a 600-mL beaker containing 100 mL of water at about 60°C . Leave the beaker in the water bath for 30 to 40 minutes, holding the temperature of the bath at about 60°C as long as the liquid has a pink color. Stir occasionally.

While the mixture is reacting, prepare a fritted glass filter crucible. Fit the crucible on a filter flask; clean the crucible by pulling, under suction, about 15 mL 1 M HNO_3 through the disk, followed by 50 to 75 mL of distilled water. Put the crucible in an oven at 150°C for about an hour to dry. Prepare a second crucible the same way, and put it in the oven.

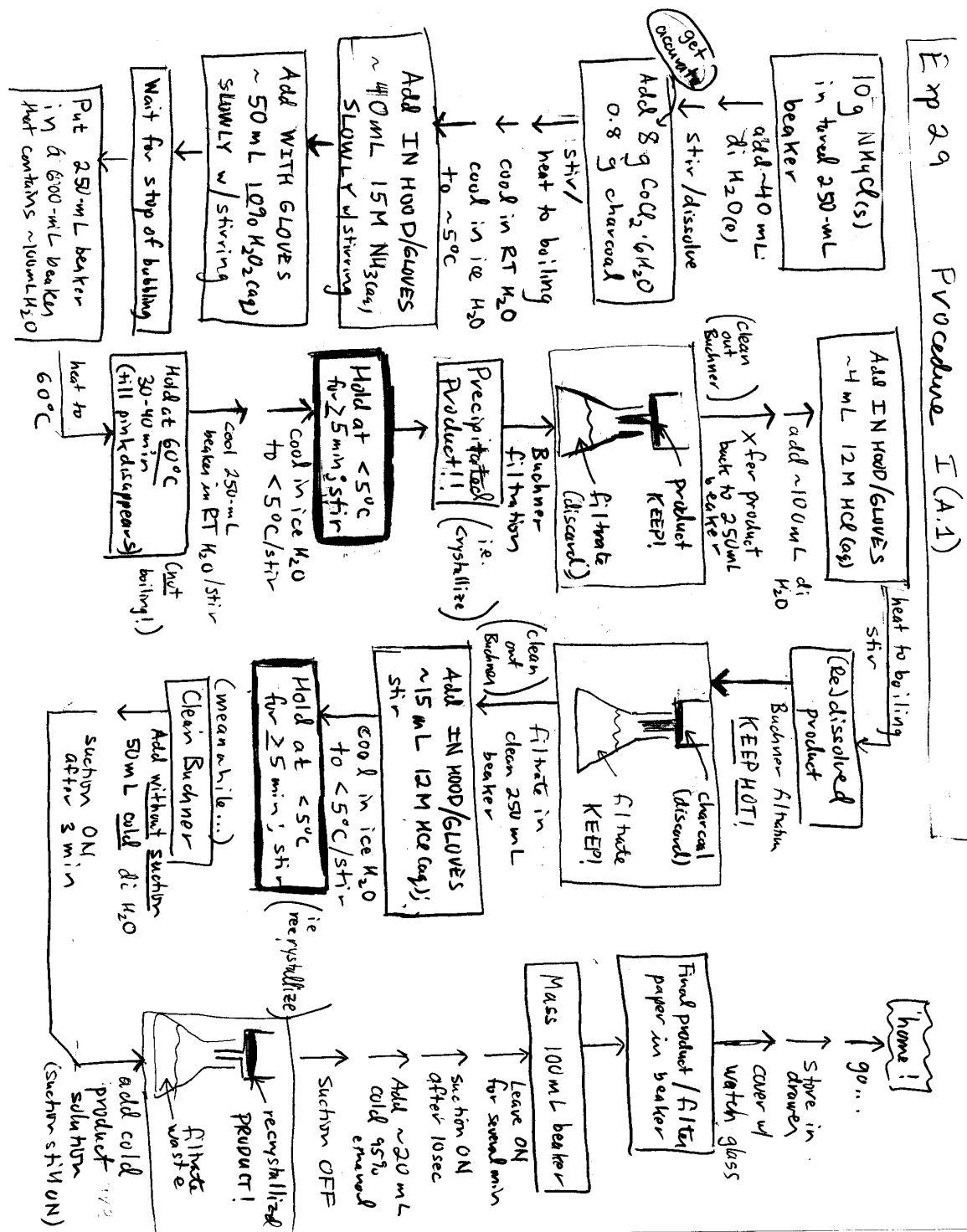
When the 40 minutes are up, remove the 250-mL beaker from the water bath and cool it, first in a water bath and then in an ice bath, until the liquid in the beaker has a temperature below 5°C . Hold the mixture at this temperature for at least 5 minutes, stirring occasionally to promote crystallization of the crude product. Set up a Buchner funnel and, with suction, filter the mixture through the filter paper in the funnel. Discard the filtrate in the waste crock.

Scrape the solid from the filter paper into the 250-mL beaker. Add 100 mL distilled water and, in a hood, 4 mL 12 M HCl . **CAUTION:** This reagent has a choking odor. Heat the mixture to boiling, with stirring, to dissolve the crystals. Rinse out the suction flask with distilled water, and reassemble the Buchner funnel. Holding the beaker with a pair of tongs or a folded paper towel, filter the hot mixture through the paper in the funnel, under suction. In this operation, the charcoal is removed and should be on the filter paper. The filtrate should be golden yellow and contains the product we seek.

Transfer the filtrate to the (rinsed-out) 250-mL beaker and, in the hood, add 15 mL 12 M HCl . Place the beaker in an ice bath and stir for 5 minutes or so to promote formation of the golden crystals of the product, $\text{Co}_x(\text{NH}_3)_y\text{Cl}_z$. Check to see that the temperature is below 5°C .

Pour about 50 mL of ice-cold distilled water on to a piece of filter paper in the Buchner funnel. After 2 or 3 minutes, turn on the suction and pull the water into the suction flask. Empty the flask. Then filter the cold mixture containing the product through the funnel, using suction. Turn the suction off, and pour about 20 mL 95% ethanol on to the crystals. Wait about 10 seconds, and then turn on the suction to pull through the ethanol, which should carry with it most of the water and HCl remaining on the crystals. Draw air through the crystals for several minutes. Weigh a 100-mL beaker to 0.1 g. Transfer the crystals from the filter paper to the beaker. Put the beaker in your locker. Take the fritted glass crucibles out of the oven, using tongs or a folded paper towel, and put the crucibles on a paper towel in your locker. Discard the liquid in the suction flask in the waste crock or as directed by your instructor.

The flowchart I constructed for the synthesis experiment. You should leave space along one side of your chart to write down observations, numerical data, and changes in procedure, .



B. Once in lab:

- (5) Record your qualitative and quantitative **Observations** next to your procedure flowchart. I expect you to write down observations like changes in colors and (qualitative) changes in temperature. Do not just write down numbers! Also note any deviations from the expected procedure. Do not write down data on scraps of paper, paper towels, or Kimwipes! Try to record data in tables whenever appropriate.
- (6) If you are running out of room next to a section of your flowchart, you are encouraged to make a reference to a later page in the notebook. Continue writing down your observations and data on that page. (I do this all the time with my research notebooks.)
- (7) Write down each number to as many significant figures as justified by the instrument you are using. Always note the uncertainty in that number as well as its units.
- (8) You should “think in writing” during the experiment. Write down any questions, insights, or suspicions in your notebook.
- (9) You and your lab partner should think about how to divide up the labor so that your time in lab can be spent as efficiently as possible. Each person should then write down what she did in her own notebook. Do not waste time copying into your notebook procedures, observations, numerical data, etc. your lab partner did or collected.

C. After lab:

You and your lab partner can choose to collaborate completely on your post-lab work. If you choose to do this, your Data Analysis and Discussion should appear in only of the notebooks. Both notebooks should say that you have chosen this option. Note that you are always free to turn in completely separate write-ups as well.

- (10) Document your **Data Analysis**. This will always include a determination of uncertainties. If you are performing the same set of calculations on a data set, you should write out one set of sample calculations (only).
- (11) If you are performing linear regressions, you must use Excel or another spreadsheet program. Print out a copy of both tables and graphs, and tape them into your notebook. (You should also e-mail me your Excel file.) Your spreadsheets and graphs should not just contain numbers! You should clearly label columns of data on spreadsheets, and provide a title and axis labels for all graphs. In your notebook, you should also provide a brief explanation of the calculations you did with the spreadsheet. (You will see examples of this when we cover calibration.)
- (12) Write a **Discussion** of your results. Indicate if the **Objective** has been achieved. If the **Objective** has not been achieved, all hope is not lost! You will need to discuss, however, what went wrong in the experiment and how you would improve the procedure if you were to repeat the experiment. You should always discuss the possible sources of both systematic and random error in the experiment, and judge what the dominant source may be. Identify the sign of each systematic error, and try to estimate its magnitude. You should also include a

comparison with the “accepted” value, if one is available. Other reporting requirements for each experiment will be detailed on your handouts.

When you are done with your write-up, remember to update your **Table of Contents!**

III. Grading

Up to **25 points** per experiment, earned in the following ways:

3 points: Flowchart done before lab

5 points: Record-keeping and other notebook mechanics

5 points: Accuracy and precision of your results

12 points: Quality of your data analysis and discussion

Note that you and your partner will always receive the same score for the accuracy and precision of your results. If you choose to do your data analysis and discussion together, you will share that score as well. Each notebook’s flowchart and record-keeping will be graded separately.

Just as with problem sets, there will be a 20% per day penalty if your notebook is late.