

Chemistry 238

Experiment 55: A Separation Scheme

Experiments are to be done *individually*.

• Pre-lab:

Read: Experiment 55 (pp. 496 – 498)

Review: Techniques 11 and 12 (pp. 647 – 693)

Prepare for class on Monday, March 5: Before coming to lab, you must work out a detailed procedure (3 points) that you can follow. You may not be able to specify all the volumes of reagents to add ahead of time, but it should be as detailed as possible. It is also possible that you will not be able to specify which solvents you will use to crystallize your compounds. You should work out this procedure on your own and **the procedure must be written completely in your own words**. This should be **word-processed** and you must bring **an extra copy to hand in at the beginning of the first experiment 55 lab period**, which will be **Monday, March 5**. **You cannot begin doing any lab work until you have handed in a procedure.**

Before writing out your procedure, you should look at the list of available reagents under the “Advance Preparation” section on page 497. You should also have the composition of the mixture you will be separating (which I handed out in class). Finally, in addition to the procedure written out in paragraph form, please draw a “**separation scheme**” (2 points), like the one shown on page 561.

In your lab book: The *purpose* is easy: “To separate a mixture of three compounds using crystallization and extraction, and to purify and characterize the two major components.”

The *materials and methods* are easy: This should be your procedure and separation scheme (you may tape them into your notebook).

Notes on the lab: Before trying out your procedure, you will probably want to try to find appropriate solvents for crystallizing the two compounds you are going to isolate. Suggested solvents are: **water, methyl alcohol, 95% ethyl alcohol, isopropyl alcohol, 1-butanol, hexane, or toluene**. Other solvents will be available if you want to use a solvent not on this list. Samples of pure compounds will be provided for trying different solvents. You should use a procedure similar to the one on pages 649 and 650. Do not follow this blindly, but rather use some common sense in making decisions about appropriate solvent. You may want to consult the Merck Index for clues about possible solvents to use. Then you should try out your procedure. If everything works out well, it is possible that you will finish most of the work during the first lab period. If some part of your procedure does not work, you can revise it and you will be given a second sample to work on. If you revise the procedure before the 2nd lab period, you must **bring a copy of the new procedure** to hand in. If your first procedure worked, but you want to try it again to see if you can improve the

yield or purity, you may do so. **You must let me know at the end of the first lab period if you will need a second sample.**

You must turn in the final products in labeled vials. The labels should include your name, the name of the compound, melting point, and weight.

• **During lab:**

The *methods* section: Take clear notes about what parts of the procedure worked and what didn't. Make observations (either here or in the data section) about changes you notice in the appearance of your mixture/solutions.

The *data* section: Given that you will need to calculate percent recovery, it would be a good idea to record masses. Given that you will need to characterize the purity of your components, it would be a good idea to record melting points. Given that crystal form may indicate something about the purity of the material, it would be a good idea to record final crystal shape and color.

For extra credit, if you wish, you may try to characterize your compounds using IR or NMR spectroscopy. To earn the extra credit points, not only will you need to provide the spectrum, but you will also need to give a full interpretation and conclusion, consistent or inconsistent with the identity of the compound.

• **Post-lab:**

The *results* section should be easy: Give the identities, masses, percent recovery and melting point ranges of the two major component compounds. You do not need to worry about the 0.05 g component.

Comment on the purity of both isolated substances and compare your melting points to the literature values.

• **Lab Result Report: (Due Monday, March 12, at the beginning of lab)**

Begin the report by writing up the **final, revised procedure** (9 points) as *actually* performed on your mixture. This should be written as a procedure for someone else to perform, not as a report of what you did. The procedure should be written up in sufficient detail so that another student in Chemistry 238 could follow it to separate and purify your mixture. It *must* be written in paragraph form and it must be typed or word-processed (no handwriting for this one).

Within the procedure, *clearly* indicate the **composition** of your mixture in the procedure. You should also give the **structures** of the compounds in your mixture. All **amounts of reagents** (when possible) should be given. All steps should be described, except for crystallizations. In this case, you may refer to the appropriate section in the Technique chapter (for instance, "...performed in the manner described in Technique 11.3"). In describing extractions, you should indicate which layer (top or bottom) to remove the first time an extraction step is performed. You might want to consult some experiments in the textbook to see how various techniques are described in a procedure.

Your procedure must be written *completely in your own words*. Two points are based on spelling and grammar. The other seven points will be based on how well the procedure is written. **Each incomplete or incorrect instruction will be marked off one point. If a significant step is missing, this will be marked off one point.**

Then, make a photocopy of the **method and data pages** (3 points) of your lab notebook. These pages should show **tables** of masses of chemicals, and have a record of **observations** made of any changes in appearance of the mixture during the separation/purification procedure.

Attach a photocopy of the **results** section (or a separate word-processed document with the results).

Results grading:

Product #1 (identity, mass and % recovery): 4 points

Product #2 (identity, mass and % recovery): 4 points

Comment about purity based on melting point ranges: 2 points

I will also assess your ability to work independently (3 points).

No abstract is needed.