

A Separation and Purification Scheme

Reading Experiment 5 in Pavia. Also see all of the Required Reading for this experiment and review Experiment 4. This includes Techniques 11 and 12. Also, review the acid/base chemistry of carboxylic acids and amines in Pavia, Technique 10.2B and also in Klein (3rd ed.), Sections 3.4, 20.3 (carboxylic acids), and 22.2 (amines). See also the hanout, **Separation Schemes – Tips and Notes**

Overview

The purpose of this experiment is to use both extraction and crystallization as a means to separate a complex mixture of organic compounds – in this case a carboxylic acid (R-CO₂H) or an organic base (R-NH₂) and two neutral compounds (R-H). You will prepare and utilize your own procedure to separate an assigned mixture. Your Separation Scheme will detail how to separate and purify *two* of three compounds in the mixture. You will use the same techniques as in Experiments 3 and 4, especially how to choose the crystallization solvent to purify the two compounds. Read the sections *Advanced Preparation* and *Purification* (pages 45 and 46) in Pavia thoroughly for lots of hints on how to be successful in this experiment.

Prelab Exercise (10 points)

Devise a **Procedure** and **Separation Scheme** to separate your assigned mixture. Through careful reading of the sections on pages 45 and 46 of Pavia, and noting what solvents and solutions will be available, you will be able to develop a procedure and the separation scheme. This assignment must be submitted for approval prior to the laboratory period. When it has been approved then put the procedure in your notebook and use it to guide your separation of the mixture. In your Separation Scheme, you must show exactly which compounds are being separated – you will be assigned one of mixtures in the list found on the **The Mixtures** handout.

Prelab

Include your **Name**, the **Date**, a **Title**, the **Purpose**, and your approved procedure. Additionally, you will need to show your **Separation Scheme**. Your separation scheme should clearly show where each compound is at each point of the scheme.

Procedure

Follow the procedure and the separation scheme that you came up with in the Prelab assignment. If you make a change to your procedure during the separation, then be sure to clearly note it in your notebook. The mixtures of compounds will be available on a separate list. You will need to isolate and purify the two most abundant substances in the mixture.

To Complete the Experiment – Partial Report (no templates)

Determine the mp of your isolated purified compounds and check their identity using the mixed melting-point technique (pure samples will be available). For your conclusion, answer the Report topics on p. 46. This includes the calculations of percent recovery for both isolated compounds (e.g., mass of neutral compound obtained)/(mass of original mixture) x 100 (use the amount you actually weighed out, which should be ~ 0.5 g). Use the format found in the **Laboratory Report Format and Check-list** for partial reports.