Experiment 5
The Separation and Identification of an Unknown Mixture

In this lab, you will be given an unknown mixture of compounds. Your goal will be to separate and identify the components using the techniques you have learned this semester. The unknown mixture you will consist of a combination of organic/inorganic acidic, basic and neutral solids. A complete listing of possible compounds is given below. For identification purposes melting point, boiling point, color and infrared spectroscopy may be used routinely. In difficult cases, NMR Spectroscopy may be used. Please work with a partner on this lab. The following pages describe the methods commonly used for measuring the IR of solids.

Potential Compounds

trans- stilbene
4-acetamidophenol
acetanilide
benzil
benzoic acid
naphthalene
p-chloroaniline
p-nitroaniline
salicylic acid
sand
sodium chloride
tetraphenylcyclopentadienone
1-naphthol
2-naphthol

In preparation for this lab you should construct a general flow chart outlining how you would separate such a mixture. Please use the flowchart format given in this lab manual. In working out your procedure, please reread experiment no. 3 in this lab manual and consider the following facts. It is considered quite normal to require some guidance on this one.

1. All the organic compounds listed below are soluble in dichloromethane. The inorganic compound is not.
2. It is safe to assume that you will have a maximum of three compounds from four categories: acid, base, neutral, or inorganic.
3. It is a really good idea to look up info on the potential compounds before coming to lab, i.e., structure, color and m.p.
4. Two grams of solid unknown is a reasonable amount of material to start with.
5. Acidic organics are frequently extracted with 3 M NaOH solution. Basic organics are frequently extracted with 3 M HCl. How can you figure out how much to use?
6. Use the solubility flow chart from experiment 4 to figure out which of the compounds are acidic and which are basic. If you are stuck, please get some help.
7. When neutralizing protonated or deprotonated organic compounds, 6 M HCl, or NaOH are frequently used.
Lab Report – Please turn in one lab report per group.

I. Flow Chart - if you revised what you were doing during lab you can turn in your latest flow chart.

II. Introduction: Keep it brief - one paragraph only. A sentence or two about the lab and a sentence or two about the big picture. Then give a procedural references and any modifications to that procedure.

III. Experiments and Results

This should have all your observations, raw data and processed data. Raw data in this lab would consist of your weights, melting points, IR spectra, and colors of the compounds. Processed Data includes the identities of each component, the calculation of percent recovery for each component, the calculation of percent composition and the interpretation of any spectral data.

If you choose to present your IR data in tabular form with interpretation, you could also put it here.

IV. Discussion

Identity

You should talk about all the evidence that points toward the identity of your compounds. Realize that physical state, color, and the way the compound behaved during the separation all support identity. It would be good to go over the chemistry showing why your compound behaved the way it did. You also need to compare the melting points and the IR spectra to the literature. If you did not interpret the IR above, you should do it here. In the fingerprint region the IR should look just like the literature spectra. Realize that the high frequency end of pellets tend to be very weak looking and that nujol mulls will have extra peaks from the nujol (three to be exact and they are quite prominent).

Yield - please see practice discussion in lab book for ideas – it is at the end of experiment 3.

Purity - this section may be redundant and unnecessary depending on what you did with the identity section. If not you should talk about the melting point depression broadening and any evidence you have of contamination from the IR. You should try to name compounds that are contaminants.

There will be no postlab questions associated with this lab, but it is hoped that you will use what you have learned about writing lab reports to turn in a strong report. Your instructor will (MNS) will grade this report.
Running the IR of a Solid Sample Making

a KBr Pellet

1. Weigh out 2 mg (how many grams is this?) of sample and grind it in agate mortar and pestle. Use the balances in the general chemistry lab. Once you get a sense of how much 2 mg is, you can estimate the quantity.

2. Add 100 mg of KBr powder. This can be done with the organic balances. Do not grind the two together. Mix them gently, but thoroughly.

3. Place one bolt in the barrel and rotate it five full turns.

4. Deposit 50 - 100 mg of the sample-matrix mixture on the polished surface of the bolt inside the barrel.

5. Insert the second bolt and rotate it until it is finger tight.

6. Using the bench vise and wrench, gradually increase pressure on each bolt. Maximum pressure should be applied for about a minute.

7. A good pellet should be more or less translucent.

8. Run the IR as per usual.

9. When finished punch the pellet out with a pointed instrument. Wipe out inside of barrel with kimwipe.

10. Be sure to put bolts, barrels and KBr back in dessicator.

Making a Nujol Mull (faster, but often gives inferior spectra)

1. Thoroughly grind 2-5 mg of the solid sample.

2. Add 1-2 drops of Nujol.

3. Grind the mixture until it has a uniform consistency.

4. Run the spectrum as you would a liquid, i.e., press the liquid between to NaCl plates.