CARBOXYLIC ACID UNKNOWN
Candidates: Table 70.1, p 764,765

A. Solubility Tests: Water, $NaHCO_3$, and NaOH

Test the solubility of your acid first in neutral water, in $NaOH/H_2O$, and in $NaHCO_3$. For each test, add 15 drops of aqueous solution to a small test tube, and then add 2 drops of sample if it is a liquid, or a little spatula quantity if you have a solid. Swirl/mix well.

- Water Test: Only acids with small numbers of carbons should be soluble
- $NaOH/H_2O$: Carboxylic acids (as well as phenols) are ionized by NaOH, and the resulting sodium carboxylates are soluble. (note: if it is so small that it dissolves in neutral water, then dissolving in $NaOH/H_2O$ tells nothing extra.)
- $NaHCO_3/H_2O$: An acid-base reaction should lead to solution, but the other unique thing is that acid-base protonation of bicarbonate leads to CO_2 bubbles. If the solubility is poor the bubbles are small and slow, but even with a solid you can often see little bubbles forming.

B. Melting Point/Boiling Point

If your carboxylic acid is a solid, take its melting point. If it is a liquid, take its micro-boiling point.

C. Titration/Neutralization Equivalence & Molecular Weight Determination

Weigh, as accurately as possible, around 200 mg of your acid into a 125 mL Erlenmeyer flask. You want 3-4 significant figures after the decimal for this, so the usual balances are unacceptable. Whether you have 200 mg or 220 or 180 doesn't matter, so long as you know exactly what your original mass is. If you have a liquid, add drops until you get to about the same mass. Dissolve your material in around 25 mL of ethanol. [Logic: It is vital that the solution be homogeneous, so you need ethanol to keep it dissolved. But the indicator needs water to work right.] Add 2 drops of phenolphthalein indicator solution. Titrate the solution with _____ M NaOH. (Copy the concentration down from the bottle!)

Summary of titration logic: Molecular weight (or "formula weight", FW), is the ratio of mass per mole. Having weighed your acid, you know the mass very precisely; but how do you know how many moles? By titrating against the precisely standardized base! From the precisely known volume of base and the molarity of the base, you can determine the # of moles of base used. Since the mole/mole stoichiometry is 1 mole of base per 1 mole of acid, the # of moles of base tells the # of moles of acid. Knowing mass of acid and moles of acid, the ratio gives you the formula weight.

[Note: To determine the formula weight (molecular weight) of specific unknown candidates, look in the Aldrich catalog on the lab desk.]

D. Anilide Derivative (See p 764, 765)

Place 10 drops of the acid chloride into a large test tube. Add a stir bar, and add 2 pipets of ether. To this solution add 20 drops of aniline, dropwise (may spatter if you add it all at once) and stir for 5 minutes. The primary precipitate that forms is the aniline hydrochloride salt. Then add 2 pipets of aqueous NaOH, and continue stirring for an additional five minutes. This will dissolve the aniline hydrochloride salt; if some precipitate remains it is the derivative itself. Use a long pipet to remove the aqueous layer from the bottom of the test tube. (Any unreacted acid chloride should be removed by the basic water.) Then add 2 pipets of aqueous HCl, and stir vigorously. Use a long pipet to remove the aqueous layer. (The aniline should be removed in the process.)

If you have a significant amount of precipitate at this point, it is the desired derivative. Filter directly over a Hirsch funnel. Rinse with some HCl/water and then some cold ethanol to get your crude derivative.

If following the acid wash you do not have a precipitate, transfer the ether to a 25-mL Erlenmeyer and concentrate it on the hot plate to remove ether. Remove the residue and let cool; should be crystalline derivative.

Recrystallize from ethanol. Ideal volumes will vary depending on your unknown, but normally a good starting guess will be about 4 mL of ethanol. The addition of some drops of water may help to produce the crystals.

E. NMR

-^{1}H may be useful. Solubility will probably be too low to get a worthwhile ^{13}C.

Carboxylic Acid Unknown Report Sheet

Unknown No. Name

Structure and Compound Name Date

---

1. Physical Examination of Starting Material

a) Physical State_____b) Color_____c) Odor_____

2. Solubility Tests on Starting Material

Solvent:	Water	If Insoluble in Water, Does it Float or Sink?	Aq NaOH	Aq NaHCO
Solubility:	_____	_____	_____	_____

3. Preliminary Candidates (my mp/bp is _____)

Possible Compounds	literature mp or bp	mw	literature mp of derivatives	What else can I do, or think about, that could shorten this list?

4. Probable Compound(s)

5. What is the approximate molecular weight (mw) of my sample, based on my titration?

Approximately _____ g/mol. (Attach a sheet that details your weights, calculation!)

6. Derivative Prepared: Anilide or p-Toluidide?

	observed mp	literature mp
Crude		
Recrystallized		

7. What structural information do my NMR's tell me?

^{1}H NMR:

^{13}C NMR:

8. What is My Actual Unknown? (Structure and Name)

9. Comments, difficulties, etc..