Name: _____ Section: _____

2. Use the reagent pump to add 7.0 mL of 12 M (conc) HCl to the solution and cover with a small watch glass.

3. Place the beaker inside a 250 mL beaker which is about one-third full of DI water. Heat this water bath to a GENTLE boil for about 5 minutes or until a solid material forms in the smaller beaker. Keep the small watch glass on the inside beaker while boiling.

4. Cool the solution by placing the smaller beaker in a cold water bath.

5. Weigh and record the mass of a piece of filter paper. Pour your newly synthesized compound Y mixture through this filter paper so that the liquid drains into a 250 mL Erlenmeyer flask. Wash any remaining solid into the filter paper by rinsing the beaker with small amounts of cold DI water from a wash bottle. Allow the crystals to drain. Wash the precipitate 3 or 4 times with 5 mL portions of cold distilled water.

6. Transfer the crystals and filter paper to a previously weighed large watch glass. Dry the crystals by using one of the following methods: (1) place the watch glass over an appropriate sized beaker about one-third full of boiling water; or (2) place in a drying oven.

7. Once the sample is dry, weigh and record the mass of the watch glass, filter paper, and crystals. Label the crystals as compound Y and keep them in a stoppered test tube in your locker for further analysis. Do not leave the crystals in an open container in your locker, as this may damage the locking mechanism.

   Mass of Compound Y: _____

PART II

1. In the fume hood, take a piece of thick copper wire and heat it in a Bunsen burner flame until its glowing. Note the flame color. Let the wire cool for 20 seconds, and dip the wire into a container with Parlon. Cover the tip in the powder, place the compound in the flame and burn it. Observe the color. Parlon contains chlorine, which burns with a distinctive color in the presence of copper.

2. Follow the same procedure using another wire to test a sample of Chemical X. This should not have any unusual color, only burning with the orange color of a typical hydrocarbon fuelled fire.

3. Follow the same procedure using another wire to test a sample of Chemical Y. Does it contain chlorine?

   Parlon Flame Color: _____ Chemical X Flame Color: _____

   Chemical Y Flame Color: _____

Name: _____ Section: _____

PART III

Use the mel-temp apparatus for melting point determination. To determine the melting point, place a small amount of crystals into a thin-walled capillary tube (about 0.7–1.0 cm). Place the tube into the mel-temp apparatus, turn it on, and record the temperature range at which the sample melts. You can heat quickly to start, but when you get near the melting point (10 – 20 ^{o}C), lower the rate of heating so you can get a more accurate reading. If you don't know the melting point, you can do one fast run to get an approximate reading, then do a more careful and gradual second run. Please note that you cannot rerun a sample once it has melted.

The melting point of compound Y is close to 300 ^{o}C. Do NOT attempt to take its melting point as the electronic thermometers will fail at such a high temperature.

While you are testing compound X, also test 4-aminobenzoic acid and phenacetin to calibrate your results. The Mel-temp will run three samples at the same time. Perform two separate trials for each substance. These two values should be within 3^{o} of each other.

	Trial 1	Trial 2	CRC Handbook
4-Aminobenzoic Acid	_____	_____	_____
Phenacetin	_____	_____	_____
Compound X	_____	_____	_____
Compound Y	*(not experimentally measured)*		~300 °C

PART IV

Dissolve about 0.2 gram of compounds X and Y separately in a 100 mL beaker containing 40 mL DI water. Use these solutions for all tests in this section.

Compare the solubility of X and Y in water.

X: Y:

Use pH paper to determine the pH of both solutions: X: _____ and Y: _____

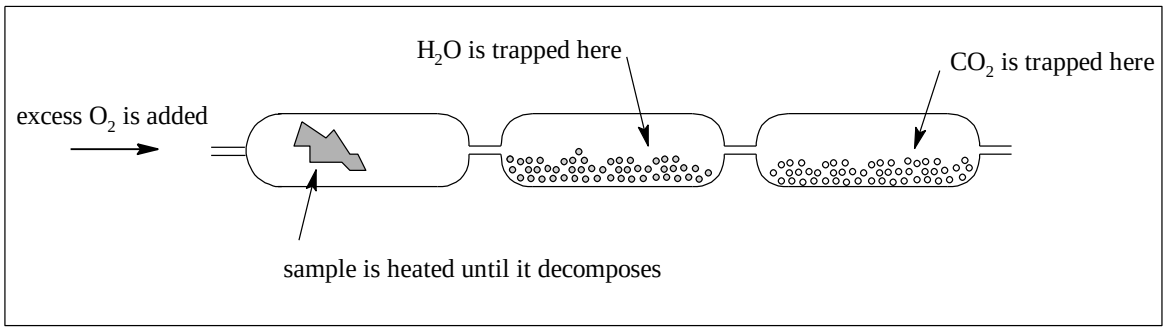
To each solution add a small quantity of Na_2CO_3. What happened? What class of compounds causes sodium carbonate to react this way?

X: Y:

Name: _____ Section: _____

PART V

The empirical formula of an organic compound can be determined via combustion analysis. A typical combustion apparatus is shown below:



A sample of the chemical is burned in oxygen in a tube packed with copper oxide to ensure complete combustion. The carbon in the compound is converted to CO_2, which is absorbed in a tube packed with ascarite. The hydrogen is converted to water vapor and is absorbed in a tube packed with drierite. The increase in weight of the absorber tubes corresponds to the CO_2 and H_2O produced during combustion. Compounds X and Y do NOT contain any nitrogen, sulfur, or halogen atoms; both compounds do contain oxygen.

A 1.0542 g sample of X was burned in a combustion apparatus. The results were:

Weight of ascarite tube before combustion	75.2382 g
Weight of ascarite tube after combustion	76.8377 g
Weight of drierite tube before combustion	81.4128 g
Weight of drierite tube after combustion	81.7418 g

Given this data, calculate the empirical formula of compound X: _____

A 1.4745 g sample of Y was burned in a combustion apparatus. The results were:

Weight of ascarite tube before combustion	80.7821 g
Weight of ascarite tube after combustion	83.0196 g
Weight of drierite tube before combustion	78.2988 g
Weight of drierite tube after combustion	78.7560 g

Given this data, calculate the empirical formula of compound Y: _____

Name: _____ Section: _____

PART VI

Before you can titrate your unknowns, you need to first determine the exact molarity of the NaOH solution you are going to use, a process known as standardization. Sodium hydroxide solutions often change concentration if they are exposed to air for long periods, and they are usually checked when first made and after a few days of use.

1. Pour about 150 mL of the approximate 0.2 M NaOH solution into a clean, dry 250 mL beaker which is labeled NaOH. Fill a clean buret with this solution.

2. Into a clean 125 mL Erlenmeyer, place about 0.5 g of KHP acid (KHP = Potassium Hydrogen Phthalate = $KHC_8H_4O_4$; MW of KHP = 204.2 g/mol). Record the exact amount of KHP in the flask.

3. Dissolve the KHP acid in your flask in about 30 mL of DI water. Add about 3 drops of phenolphthalein indicator to the flask.

4. Take the initial buret reading of the level of NaOH, and slowly add NaOH to the flask from the buret. Swirl flask continually. The solution will change from clear to light pink in exactly one drop, so watch closely. If the color disappears with swirling, then you have not reached the endpoint. A dark pink color indicates you have passed the endpoint. When you think you are close to the endpoint, begin to add the NaOH one drop at a time. Record the exact volume of NaOH solution added.

5. Repeat the titration until the molarity is consistent within 0.004 M.

<u>Data for Part VI Standardization:</u>

Recall that at the endpoint, moles OH^- = moles H^+. Because each mole of KHP contains one acid group, the moles of H^+ = moles KHP.

Mass KHP acid	_____	_____	_____
Moles KHP acid	_____	_____	_____
Volume of NaOH added	_____	_____	_____
Molarity NaOH	_____	_____	_____

Average Molarity (Use values within limit) _____

Name: _____ Section: _____

Now knowing that both compounds X and Y are acidic, you can titrate each using your previously standardized NaOH solution. Moreover, if you could determine the molecular weight using another technique (Part VII), you can ultimately determine how much mass of the compound is "attached" to each acidic proton. This is known as the "gram equivalent weight".

1. To an empty 250 mL Erlenmeyer flask, add about 0.20 grams of X to the flask (record its exact mass).

2. To the flask, add about 30 mL of DI water and 3 drops of phenolphthalein indicator. Titrate the sample with standardized NaOH solution. The endpoint will be a faint pink color that persists for more than 30 seconds.

3. Repeat the titration until you get two consistent results (within 2%), and compare the mass X / mole H^+ values.

Data for Part VI Titration of Compound X

Mass X	_____	_____	_____
Volume of NaOH added	_____	_____	_____
Moles OH^-	_____	_____	_____
Moles H^+	_____	_____	_____
Grams X per mole H^+	_____	_____	_____
Average grams X per mole H^+ (Use values within limit)		_____	
Mole H^+ per mole X (Must know molecular weight from Part VII)		_____	

Name: _____ Section: _____

Repeat the titration using compound Y. Compound Y titrates slowly, and it may take longer for the pink color to persist as Y will still be dissolving during the titration. You may wish to start the titration before Y has completely dissolved. It should dissolve before you are halfway through adding the NaOH solution.

Data for Part VI Titration of Compound Y

Mass Y	_____	_____	_____
Volume of NaOH added	_____	_____	_____
Moles OH^-	_____	_____	_____
Moles H^+	_____	_____	_____
Grams Y per mole H^+	_____	_____	_____

Average grams Y per mole H^+
(Use values within limit) _____

Mole H^+ per mole Y
(Must know molecular weight from Part VII) _____

Name: _____ Section: _____

Data for Part VII

Mass of compound X _____

Mass of H_2O _____

	Trial 1	Trial 2
Freezing temperature of pure water	_____ =	_____
Freezing temperature of solution	_____	_____
ΔT_f	_____	_____
Molecular weight of compound X	_____	_____

Average Molecular Weight (values must be within 10%): _____

PART VIII

Chemists generally turn to instrumentation first in most research and industrial settings. In this section, you are going to use two techniques to investigate your compounds.

Mass Spectrometry (MS) is a powerful technique that gives you the molecular weight of most compounds quickly and accurately. It is easy enough to employ that it is used for multiple purposes, for example, to scan luggage for explosive residues in airports. Analysis of the small fragments and exact patterns of the mass spectra is difficult, but finding the molecular weight of the compound is usually trivial. A mass spectrum features various mass-to-charge ratios of charged particles, where unknown compounds are identified by their fragmentation patterns. For this lab, you are looking for the *molecular* (or *parent*) *ion* which corresponds to the unknown compound's molecular mass. It is important to note (as you will see) that the molecular ion can be weak with a small height on the actual spectrum; don't assume that the largest peak present in your spectrum necessarily corresponds to the molecular ion peak. Consider the following: does your average molecular weight value from Part VII correlate with any peaks in your mass spectra in Part VIII?

Infrared Spectroscopy (IR), which is more difficult to interpret, gives you information about which type of functional groups are present in a molecule. By comparing peaks to known values, you can determine if your molecule has those types of functional groups.

On the next page are the MS and IR for both compounds. Your instructor will give you guidance about the level of interpretation that is expected. You can find databases of spectra from the SDBS (spectral data base system from Japan's AIST) online to compare these spectra to.