**Acid-Base Titration: Determination of Unknown Sodium Carbonate**

Solutions of the strong acids and bases, which are used as standard solutions in titrimetric analysis, must be prepared *approximately* and the exact concentration is determined by titration with a primary standard material. Some equations which will be helpful in the completion of this lab can be found in the Proper Use of Lab Equipment handout.

**Prepare 0.1 M Hydrochloric Acid Solution**

Calculate the correct amount of concentrated (about 12 M) HCl needed to make 1 liter 0.1 M HCl. Place about 500 mL of distilled water in a 1 L volumetric flask and carefully add the acid. Fully dissolve the acid in the water and dilute the solution to the 1 L mark on the volumetric flask. Transfer the solution to a glass stoppered 1 L storage bottle. The HCl does not need to be measured precisely. *Students should avoid skin contact of conc. HCl and avoid inhaling* *its vapors.* ***Transfer conc. HCl in the hood.*** *Do not pipet by mouth. Always add acid to water.*

**Prepare 0.1 M Sodium Hydroxide Solution**

The NaOH solution from the first lab may be used in this lab. If enough NaOH solution is remaining from the previous lab this step may be skipped. Otherwise proceed to make a new NaOH solution as follows.

Calculate the correct amount of solid NaOH (NaOH = 40 g/mole) needed to make 1 L of 0.1 M NaOH. Add 500 mL of distilled water to a 1 L volumetric flask and add the NaOH to the water. Completely dissolve the NaOH, and then dilute the solution to the 1 L mark. This solution should be transferred to the storage bottle with the rubber stopper. The NaOH does not need to be weighed precisely.

**Standardize Sodium Hydroxide against Potassium Hydrogen Phthalate (KHP)**

If you are satisfied with the standardization of your NaOH from the previous lab, this step may be skipped. Otherwise re-standardize the base solution as follows.

Potassium hydrogen phthalate is a white, crystalline solid, which is suitable for use directly as ***a PRIMARY STANDARD***. It is best to dry KHP for a few hours in an oven at 100oC to remove traces of moisture before using it (your TA will have already placed the unknown samples in the oven for you). It is then best stored in a desiccator.

**KHC8H4O4 + NaOH 🡺 KNaC8H4O4 + H2O**

KHC8H4O4 is KHP or the first potassium salt of phthalic acid (benzene-1,2-dicarboxylic acid).

Obtain a quantity of dried primary KHP in a weighing bottle and keep it stored in your desiccator. If you see the blue drierite has turned pink, it’s time to change the drierite in your desiccator.

Weigh separate 0.7-0.8 g samples (to the nearest 0.1mg i.e., 0.0001 g) into a 250 mL Erlenmeyer flask, and dissolve in 50-75 mL distilled water. It is best if the samples of KHP are weighed by difference, but not necessary. If a weight has (0.1 mg) behind it, weigh it on the analytical balance. Otherwise use the regular balance.

Add 2-3 drops phenolphthalein indicator and titrate with base to its endpoint (the first hint of pink). Calculate the concentration of the NaOH solution. Repeat the titration at least two more times and calculate the average, standard deviation, and ppt. **Your results should be within 5 ppt**. The ppt (parts per thousand) equation is found at the end of proper uses of lab equipment and equations.

**Determine Acid Concentration**

Deliver 25 ml of your acid using a 25 ml volumetric pipet or burette into a clean 250 mL Erlenmeyer flask. Be sure to record the volumeof acid added to the flask. Add 2 or 3 drops of phenolphthalein indicator to the solution and begin to titrate base until afaint pink tint remains for at least 30 seconds. *(A good indication of pink is that it is faint close by the flask, but seems clear on the other side of the room.)* Record the volume of base used in the titration. Repeat the titration at least two more times and calculate the average, standard deviation, and ppt. Your results should be within 5 ppt.

**Determine Percent Sodium Carbonate in an Impure Sample**

The percent sodium carbonate (also known as “soda ash”) can be determined by virtue of the very slight ionization of carbonic acid, which causes the displacement reaction below to go completely to the right.

**Na2CO3 + 2 HCl 🡺 H2CO3 + 2NaCl**

where carbonic acid H2CO3 🡪 H2O + CO2(g)

Prepare two solutions to use as color “standards” for the starting point and the endpoint. To 50 – 75 mL of distilled water in an Erlenmeyer flask, add 6-7 drops of methyl orange indicator. This solution will serve as a comparison for the starting point of the titration. For the second solution weigh out 0.2 gram (0.1 mg) of sodium carbonate and dissolve it in 50-75 mL of distilled water in an Erlenmeyer flask. Add 6-7 drops of methyl orange. Calculate how much of your standardized HCl solution is needed to bring the titration to its endpoint and carefully add this volume, using a burette, to the flask while swirling. This solution will serve as the reference for the end point of the titration.

To determine the percent composition of the unknown, calculate the appropriate amount of unknown needed to titrate using about 40 mL of your standardized HCl solution. Weigh out the sample to the nearest 0.1 mg and place it into a 250 mL Erlenmeyer flask. Dissolve the unknown in 50 – 75 mL of distilled water. To the unknown add 6-7 drops of methyl orange indicator and carefully titrate with your standard HCl until the solution begins to JUST turn from yellow to faint orange. Use the color standards for comparison. NOTE: This titration will most likely NOT use 40 mL of acid to titrate to the endpoint.

As the previous titration most likely did not require 40 mL, calculate the new mass needed to titrate the unknown with about 40 mL of your standardized HCl solution. Repeat the titration at least two more times using the new calculated mass of unknown. Calculate the average percent composition, standard deviation, and ppt of the unknown. The unknown percentages are in the range 20-50%.