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

**Background**

Solutions of strong acids and bases are used as standard solutions in titrimetric analysis. These solutions must be prepared *approximately* and the exact concentration then determined by titration with a primary standard. The process of approximate preparation of a solution and then determination of its exact concentration is called the *standardization* of a solution. A *primary standard* is a highly pure substance that also must be stable (@ room or elevated temperatures) and not hygroscopic in nature. Some helpful equations used in this experiment are located in the Proper Use of Lab Equipment handout. *Quantitative transfer* is defined as the complete transfer of *all* of a reagent from one vessel into another vessel. Primary standards are always to be quantitatively transferred to ensure the weight recorded on an analytical balance [measured out] is exactly what ends up reacting in the experiment.

**Prepare 0.1 M Hydrochloric Acid Solution (HCl)**

Calculate the volume of concentrated HCl (12.0 M) needed to prepare 500.0 mL of ~0.1 M HCl. Put about 200 mL of Nanopure H2O in a 500.0 mL volumetric flask and then carefully add the acid [***do this in a fume hood***]. Swirl then dilute the solution to the mark on the volumetric flask using Nanopure H2O. Cap flask and mix; then transfer the solution from the flask into the glass stoppered 1L storage bottle. *Students should avoid skin contact of conc. HCl and avoid inhaling* *its vapors. Always add acid to water, not water to acid!*

**Prepare 0.1 M Sodium Hydroxide Solution**

Use the standardized NaOH solution from lab #1 (KHP Lab). It is wise to re-standardize this solution (1-2 titrations) to check its concentration. If after one “check standardization titration” the combined molarity data is still < 5 ppt [or close to an approved ppt] your precision indicates moving on to unknown analysis.

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

Potassium Hydrogen Phthalate (KHP) is a white, crystalline solid, suitable for use as a ***PRIMARY STANDARD***. Before use, it is best to dry KHP for a few hours in an oven at 100oC to remove traces of surface moisture (the TA will have already dried the pure KHP in the oven). The molar mass of KHP is 204.2212 grams/mole.

**KHC8H4O4 + NaOH 🡺 KNaC8H4O4 + H2O**

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

Obtain a quantity of dried, pure KHP in a glass weighing bottle/top and keep it stored in a desiccator.

Weigh a 0.7-0.8 g sample (to the nearest 0.1mg i.e., 0.0001 g) of KHP. Use an analytical balance and record all significant digits reported. It is best if the samples of KHP are weighed by difference, but not necessary. The use of weigh boats or weighing paper is allowed as long as KHP aliquot is quantitatively transferred into a 250 mL Erlenmeyer flask. Once the KHP aliquot is transferred to the 250 mL Erlenmeyer flask, dissolve it in 50-75 mL of Nanopure H2O measured using a 100.0 mL graduated cylinder.

Add 2-3 drops of phenolphthalein indicator to the KHP solution in the 250 mL Erlenmeyer flask. Titrate with base until a very *faint pink* color remains for at least 30 seconds (the endpoint). Record the volume of base used in the titration to two significant digits past the decimal (xx.xx mL). Calculate the molarity of the NaOH. Repeat the process at least two more times. Calculate the average molarity, standard deviation, and ppt.

**Determine Acid (HCl) Concentration**:

Fill one clean buret with the standardized NaOH solution. (*Remember to remove all air in the buret tip and label burets with the solution they contain to avoid neutralization of your solutions.)* Record initial buret reading.Deliver 25.00 mL of the acid solution just prepared **using a 25.00 mL volumetric pipet** into a clean 250 mL Erlenmeyer flask. Record this volume in your notebook for reference. 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 final buret reading. Repeat the titration at least two more times and calculate mean, standard deviation, and ppt. **Your results (ratios) should be within 5 ppt**. The ppt (parts per thousand) equation is found at the end of the Proper Use of Lab Equipment handout. If results are not precise, perform additional titrations until satisfactory results are achieved.

**Determine Percent Sodium Carbonate (Na2CO3) in an Impure Sample:**

Percent sodium carbonate (also known as “soda ash”) can be determined by virtue of the very slight ionization of carbonic acid (H2CO3), which causes the displacement reaction to go completely to the right.

The molar mass of Na2CO3 is 105.988 grams/mole.

**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. People see colors/ranges of color differently so it is important each student individually assess their own reaction mixtures. That is the best advice to keep consistent in determining endpoints:

Solution A: Add 50 – 75 mL of Nanopure H2O to an Erlenmeyer flask, and then add 6-7 drops of methyl orange indicator. Swirl & Parafilm. This solution will serve as a comparison for the *starting point* of the titration. This is a *must* to prepare.

Solution B: Weigh out 0.2 gram (0.1 mg) of sodium carbonate and dissolve it in 50-75 mL of Nanopure H2O in an Erlenmeyer flask. Add 6-7 drops of methyl orange. Calculate the required volume of your standardized HCl solution needed for “Solution B” to reach its endpoint. Carefully add this volume of standardized HCl, using a buret, to the flask while swirling. Parafilm flask. This solution will serve as the reference for the *endpoint* of the titration. This is *optional* to prepare; pros/cons to Solution B prep will be discussed.

To determine the percent composition of the unknown, calculate the appropriate amount of unknown needed to titrate using at least 30.00 mL of your standardized HCl solution. Weigh out an aliquot of unknown [appropriate amount] and place it into a 250 mL Erlenmeyer flask. Dissolve the unknown in 50 – 75 mL of Nanopure H2O. Add 6-7 drops of methyl orange indicator. Carefully titrate with your standard HCl until the solution begins to *JUST turn from yellow to a faint orange*. NOTE: This titration will most likely NOT use ~30.00 mL of acid to reach the endpoint. This titration is still taken into account in the calculations.

As the previous titration most likely did not require ~30.00 mL, calculate the new mass (adjusted amount) of unknown needed to titrate the unknown with 30.00 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. Unknown carbonate percentage ranges from 20.00 - 50.00 %.