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**Exp. 9 A Volumetric Analysis**

**Purpose:**

* To prepare and standardize a sodium hydroxide solution.
* To determine the molar concentration of a strong acid.

**Materials and Reactions (if any):**

* Pipets
* Burets
* Volumetric flasks
* Titrant (Sodium hydroxide)
* Phenolphthalein indicator
* 25.00 mL of the acid solution
* three clean 125-mL or 250-mL Erlenmeyer flasks
* 1L deionized water
* dry potassium hydrogen phthalate (KHC8H4O4)

**Procedure:**

* **Safety precautions**
  + Don’t stand on a lab stool to read the meniscus

1. The Standardization of a Sodium Hydroxide Solution
   1. Dry the primary standard acid. Dry 2–3 g of KHC8H4O4 at 110°C for several hours in a constant-temperature drying oven. Cool the sample in a desiccator.
   2. Prepare an approximate 0.15 M NaOH solution. Calculate and measure on a balance (±0.01g) the mass of NaOH required to prepare 500 mL of a 0.15 M NaOH solution. Fill a 500 mL polyethylene bottle about 1/3 full with previously boiled, deionized water cooled to room temperature. Add the solid NaOH in the 500 mL polyethylene bottle. Swirl the solution (do not shake!). Finally, dilute the volume to ~500 mL with the previously boiled, deionized water. Cap the polyethylene bottle to prevent the absorption of CO2 and label the bottle.
   3. Prepare the primary standard acid.
      1. Calculate the mass of KHC8H4O4 that will require about 15–20 mL of your diluted NaOH solution to reach the stoichiometric point.
      2. Measure this mass (±0.001 g) of KHC8H4O4 on a tared piece of weighing paper (Figure 9.3) and transfer it to a clean, labeled Erlenmeyer flask. Complete the preparation of all three samples while you are occupying the balance. Dissolve the KHC8H4O4 in about 50 mL of previously boiled, deionized water and add 2 drops of phenolphthalein.
   4. Prepare a clean buret. Wash a 50-mL buret and funnel thoroughly with soap and water using a long buret brush. Flush the buret with tap water and rinse several times with deionized water. Rinse the buret with three 5-mL portions of the diluted NaOH solution, making certain that the solution wets the entire inner surface. Drain each rinse through the buret tip. Discard each rinse in the Waste Bases container.
   5. Fill the buret. Using a clean funnel, fill the buret with the NaOH solution. After 10–15 seconds, read the volume by viewing the bottom of the meniscus with the aid of a black line drawn on a white card. Record this initial volume according to the guideline in Technique 16A.2, using all certain digits (from the labeled calibration marks on the glassware) plus one uncertain digit (the last digit which is the best estimate between the calibration marks). Place a sheet of white paper beneath the Erlenmeyer flask.
   6. Titrate the primary standard acid. Slowly add the NaOH titrant to the first acid sample prepared in Part A.3. Swirl the flask after each addition. Initially, add the NaOH solution in 1- to 2-mL increments. As the stoichiometric point nears, the color fade of the indicator occurs more slowly. Occasionally rinse the wall of the flask with (previously boiled, deionized) water from your wash bottle. Continue addition of the NaOH titrant until the endpoint is reached. The endpoint in the titration should be within one-half drop of a slight pink color. The color should persist for 30 seconds. After 10–15 seconds, read and record the final volume of NaOH in the buret.
   7. Repeat the analysis with the remaining standard acid samples. Refill the buret and repeat the titration with the remaining two samples prepared in Part A.3.
   8. Do the calculations. Calculate the molar concentration of the diluted NaOH solution. The molar concentrations of the NaOH solution from the three analyses should be within ±1%. Place a corresponding label on the 500-mL polyethylene bottle
2. Molar Concentration of an Acid Solution
   1. Prepare the acid samples of unknown concentration. In an Erlenmeyer flask, pipet 25.00 mL of the acid solution. Add 2 drops of phenolphthalein.
   2. Fill the buret and titrate. Refill the buret with the standardized NaOH solution and, after 10–15 seconds, read and record the initial volume. Refer to Parts A.5 and A.6. Titrate the acid sample to the phenolphthalein endpoint. Read and record the final volume of titrant.
   3. Repeat. Similarly titrate the remaining samples of the acid solution.
   4. Calculations. Calculate the average molar concentration of your acid unknown.

**Calculations (if any):**