Chemistry 101 **12-STANDARDIZATION OF SODIUM HYDROXIDE**

Standard solutions for titrations are especially pure mixtures with exactly known concentrations. **Primary standards** are very pure solids. They have the advantage that they can be weighed (the analytical balance is normally the most accurate instrument in the laboratory) and they are stable under laboratory conditions. In this experiment, the primary standard is oxalic acid dihydrate, $H_2C_2O_4 \cdot 2H_2O$. It will be used to standardize a solution of sodium hydroxide.

Sodium hydroxide solutions pick up carbon dioxide from the air. This contamination can affect the strength of the base solution and can spoil the sharpness of the end point in the titration. The procedure below is designed to prepare and standardize carbonate-free NaOH.

Equation

$$2\ NaOH(aq) + H_2C_2O_4 \cdot 2H_2O(s) \rightarrow Na_2C_2O_4(aq) + 4\ H_2O(l)$$

PROCEDURE

Wear your **safety glasses** while doing this experiment.

Place 300 mL of deionized water in a large beaker and bring it to the boiling point. Boil it vigorously for 5 minutes and allow it to cool. Repeat with a second 300 mL sample of deionized water.

Clean a 500 mL Florence flask, rinse it twice with 10 to 20 mL of your boiled water, and fit it with a good rubber stopper. Take the flask to your instructor, who will give you about 2.5 mL of 50% NaOH(aq). Fill the flask about two-thirds full with boiled water and mix well, **with swirling**. Then fill with boiled water to just below the neck and mix again. Label the flask. It now contains about 500 mL of approximately 0.1 M NaOH.

Check out a buret from the stockroom. Rinse it well with tap water, then distilled water. Finally, rinse it three times with about 4 mL of your NaOH solution each time. Fill the buret with NaOH and cover the top of the buret with plastic wrap until you are ready to use it.

Make a data table in your notebook. See the **Report Sheet** for a list of data entries and calculated quantities.

Obtain a sample of oxalic acid dihydrate in a clean, dry shell vial. **DO NOT HANDLE** the shell vial with your fingers. Use tongs, or a paper strip to carry the vial. Weigh the vial with oxalic acid on the analytical balance.

Prepare a 125 mL Erlenmeyer flask (which must be clean but need not be dry on the inside). Tap out a sample of about 0.20 to 0.25 g of oxalic acid into the flask. Weigh the vial again on the analytical balance. The difference between the two weights is the mass of oxalic acid you will titrate. (You may prepare several samples at once but you must titrate them in the same laboratory period. Be sure to label them and to record the mass data for each sample.)

Add about 25 mL of deionized water and 3 drops of phenolphthalein to the oxalic acid. Swirl the mixture to dissolve the oxalic acid. Read the buret to the nearest 0.01 mL, and titrate the oxalic acid with NaOH. The end point has been reached when the pale pink color of the phenolphthalein persists for 30 seconds. Try to carry out the titration so that the last half-drop of NaOH causes the change in color. Calculate the molarity of the NaOH solution.

Repeat the titration and calculations until you have three determinations that agree within 5 parts per thousand (0.5%). You may use the Q test to reject "bad" values.

A "shortcut" to check agreement of values during the experiment is to calculate the ratio of volume of base for a trial divided by the mass of oxalic acid used in that trial. If this ratio varies only in the last significant figure for three trials, the calculated molarities will also have little variation. This calculation can also be used to predict the base volume required to titrate any sample of oxalic acid, once one accurate trial has been completed. Ask your instructor to explain if you cannot reason out this method.

When all titrations are completed, drain the buret, rinse it with three portions of tap water and three portions of deionized water, and return it to the stockroom.

KEEP THE REMAINING NaOH SOLUTION FOR THE NEXT EXPERIMENT.

Chemistry 101 **12-STANDARDIZATION OF SODIUM HYDROXIDE**

Section_____ Name_____

Report Sheet

For each titration, you will report the following. Repeat the data table and calculations for each titration.

Finally, tabulate the values for the molarity; calculate the average value and the average deviation. See the "Measurement" experiment for this procedure and for the Q test.

Data:

Mass of vial and oxalic acid dihydrate _____
Mass of vial less sample _____

Mass of sample _____

Initial buret reading _____
Final buret reading _____

Volume of NaOH _____

Calculations:

Moles oxalic acid dihydrate used _____

Moles NaOH used _____

Volume of NaOH (liters) _____

Molarity of NaOH _____

Include an example of your calculations with your report.

Chemistry 101 **12-STANDARDIZATION OF SODIUM HYDROXIDE**

Questions

1. Calculate the mass of acetic acid ($HC_2H_3O_2$) that would be neutralized by 28.67 mL of **your** NaOH solution. Write the equation for the reaction, and show your method of calculation.

2. Potassium hydrogen oxalate can also be used as a primary standard. Its formula is $KHC_2O_4 \cdot H_2O$. When this compound is used to react with 0.1 M NaOH, we would not use 0.2 g samples, as we did with our oxalic acid dihydrate. Would the correct sample size of potassium hydrogen oxalate monohydrate be greater or less than 0.2 g? Explain.

Chemistry 101 **12-STANDARDIZATION OF SODIUM HYDROXIDE**

Section_____ Name_____

Pre-Laboratory Assignment

1. The density of 50% NaOH solution is about 1.5 g/mL. Calculate the volume of 50% NaOH solution that contains 0.050 mole of NaOH.

2. Calculate the molarity of a NaOH solution if 32.02 mL of the solution neutralizes 0.2262 g of oxalic acid dihydrate.

3. Will the calculated molarity of the NaOH solution be too high or too low if a student "overshoots" the end point of the titration? Explain.