GENERAL 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.

Clean a 500 mL Florence flask, rinse it twice with 10 to 20 mL of DI water, and fit it with a good rubber stopper. Fill the flask about two-thirds full with boiled water and mix well, **with swirling**. Then fill with enough DI water to make 400 mL and mix again. Label the flask. It now contains about 400 mL of approximately 2 M NaOH.

Check out a burette 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 burette with the approximately 2 M NaOH.

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

Prepare a 125- or 250-mL Erlenmeyer flask (which must be clean but need not be dry on the inside). Tap out a sample of about 3 g of oxalic acid into the flask. Add about 50 - 75 mL of deionized water and 3 drops of phenolphthalein to the oxalic acid. Swirl the mixture to dissolve the oxalic acid. Read the burette 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.

KEEP THE REMAINING NaOH SOLUTION SINCE IT IS NOW STANDARDIZED AND READY TO USE FOR ANY EXPERIMENT.