# **E12A - STANDARDIZATION OF SODIUM HYDROXIDE**

 Standard solutions for titrations are especially mixtures with precisely known concentrations for use in calculating stoichiometric relationships with an unknown. **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 crystalline oxalic acid dihydrate, H2C2O4 ⋅ 2H2O. 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) + H2C2O4⋅2H2O(aq) 🡪 Na2C2O4(aq) + 4 H2O(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 to expel dissolved CO2 gas and allow it to cool. Repeat with a second 300 mL sample of deionized water. Use this water to make your NaOH solution.

 Clean a 500 mL Florence flask, rinse it twice with 10 to 20 mL of your boiled DI water, and fit it with a good rubber stopper. Obtain enough 6.0 M NaOH(aq) to produce an approximate 0.1M NaOH solution. Remember: M1V1=M2V2. (Calculate this volume of more concentrated NaOH in advance!) Fill the flask about two-thirds full with boiled DI water, add the 6M NaOH and mix well, **with swirling**. Then fill with boiled DI 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 a 4 mL portion of your NaOH solution each time. Fill the buret with NaOH to just below the 0.00 mL mark. Keep your original NaOH solution stoppered and **SAVE IT** throughout the duration of Experiments 12A and 12B.

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

 To weigh out an exact sample of oxalic acid dihydrate, first pre-weigh a weighing boat on the centigram balance, then carefully tap out the approximate amount sample of oxalic acid, estimated in the pre-laboratory assignment, into the weighing boat. Record your approximate weight of oxalic acid in your notebook. Prepare a 125 mL Erlenmeyer flask (which must be clean but need not be dry on the inside). Next weigh the Erlenmeyer flask on the analytical balance and record its weigh.

Transfer the sample of oxalic acid out of the weighing boat and into the flask. Weigh the flask again on the analytical balance. The difference between the two weights is the mass of oxalic acid you will actually titrate. (You may prepare several samples at once but they should each differ slightly in the mass of sample taken. Also, you must titrate them in the same laboratory period. Be sure to label them and to record the mass data for each sample; do not mix them up.)

 Add about 25 mL of deionized water and 2-3 drops of phenolphthalein to the oxalic acid. Swirl the mixture to dissolve the oxalic acid. Read the initial buret volume 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 after gentle swirling. Try to carry out the titration so that the last half-drop of NaOH causes the change in color. Record the final buret volume. 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 then three portions of deionized water, and return it to the stockroom.

**KEEP THE REMAINING NaOH SOLUTION FOR EXPERIMENT, 12B.**

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REPORT SHEET

Section\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Name\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

 For each titration, you will report the following in your lab notebook. Repeat the data table and calculations for each titration. Indicate in your notebook any inaccurate trials and any that fall outside your confidence limit. On this report sheet only give **your three best** sample trial’s data.

Finally, tabulate the accurate 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:**

| **Measurement** | **Trial 1** | **Trial 2** | **Trial 3** |
| --- | --- | --- | --- |
| Mass of weighing boat/paper |  |  |  |
| Mass of weighing boat/paper & acid |  |  |  |
| Mass of flask & oxalic acid dihydrate |  |  |  |
| Mass of flask |  |  |  |
| Mass of sample  |  |  |  |
| Initial buret reading |  |  |  |
| Final buret reading |  |  |  |
| Volume of NaOH |  |  |  |
| Moles oxalic acid dihydrate used |  |  |  |
| Moles NaOH used |  |  |  |
| Volume of NaOH (L) |  |  |  |
| Molarity of NaOH |  |  |  |

Include an example of your calculations with your report.

# **E12A - STANDARDIZATION OF SODIUM HYDROXIDE**

POST LAB QUESTIONS

Section\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Name\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

1. Calculate the mass of acetic acid (HC2H3O2) that would be neutralized by 28.67 mL of **your** NaOH solution. Write the equation for the reaction, and show your method of calculation.

1. Potassium hydrogen oxalate can also be used as a primary standard. Its formula is KHC2O4 ⋅ H2O. 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.

# **E12A - STANDARDIZATION OF SODIUM HYDROXIDE**

PRE-LABORATORY ASSIGNMENT

Section\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Name\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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.

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