

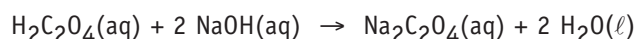
# ACID-BASE TITRATION

## Form A

### The Molar Mass of an Unknown, Diprotic Acid

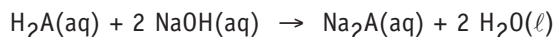
**T**itration is the process for ascertaining the exact volume of one solution that is chemically equivalent to a given amount of another substance, either another solution or a given amount of solid material dissolved in a solvent. The apparatus usually used in titrations is a buret. If a solution of an acid is titrated with a solution of a base, the **equivalence point**, the point at which chemically equivalent quantities of acid and base have been mixed, can be found by means of an indicator.

In this experiment, you will **standardize** (determine precisely the concentration for) a solution of the base sodium hydroxide, NaOH, using oxalic acid dihydrate,  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ , as a primary standard acid. A primary standard acid is a solid acid whose mass is an accurate measure of the number of moles of  $\text{H}^+$  ions it will furnish. The balanced equation for the acid-base reaction involved in the standardization procedure is



This equation specifies that there are two moles of  $\text{H}^+$  supplied by each mole of oxalic acid in this reaction (since 1 mole of  $\text{H}^+$  is consumed per mole of NaOH). From the mass of oxalic acid dihydrate used in the reaction (which you know by weighing the sample), you can calculate the moles of acid used. This is related to the amount of NaOH by a stoichiometric factor of (2 moles NaOH/1 mole acid). With the moles of NaOH known, and the volume of solution in which these are contained, you can calculate the concentration of the NaOH (moles of NaOH/liters of solution). That is, you have now standardized the solution of NaOH and can use it to determine the molar mass of an unknown, diprotic acid.

In this experiment you will be given a sample of an unknown acid. You only know that it reacts with NaOH according to the general equation



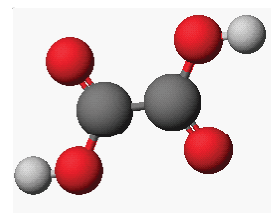
That is, the acid is known to supply two moles of  $\text{H}^+$  per mole of acid. When you titrate a weighed sample of the unknown acid with standardized NaOH, you will determine the volume of NaOH required. Therefore, you know the quantity of NaOH (mol) used from the relation

$$\text{Amount of NaOH (mol)} =$$

$$\text{Concentration of NaOH (mol/L)} \cdot \text{Volume of NaOH consumed (L)}$$

The moles of NaOH consumed is related, through the stoichiometric factor (of 1/2, based on the balanced equation) to the moles of acid that were contained in the sample.

$$\text{Amount of unknown acid (mol)} = \text{Amount of NaOH used (mol)} \cdot \frac{1 \text{ mol acid}}{2 \text{ mol NaOH}}$$



**Oxalic acid,  $\text{H}_2\text{C}_2\text{O}_4$ .** This simple, organic acid has two ionizable H atoms, so two moles of NaOH are required to consume one mole of the acid.

The technique of titration is well illustrated on

<http://www.dartmouth.edu/~chemlab/techniques/titration.html>

Finally, the molar mass of the acid can be found from the relation

$$\text{Molar mass of acid (g / mol)} = \frac{\text{Mass of acid sample (g)}}{\text{Mol of acid in sample}}$$

## EXPERIMENTAL PROCEDURE

### 1. Standardization of a Solution of Sodium Hydroxide

- a) Clean and rinse your graduated cylinder and Erlenmeyer flasks. Clean a buret and rinse with distilled water until the water drains cleanly from the inverted buret.
- b) Prepare a dilute solution of sodium hydroxide by placing 50 mL of 3 M stock solution (provided at the end of the lab bench) in a 500 mL Erlenmeyer flask. Add 250 mL of distilled water, stopper the flask and shake well.
- c) In the *Pre-Lab Assignment* for this Experiment you calculated the mass of  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  required in the standardization. On a piece of weighing paper, and using the analytical balance, weigh some  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  to the nearest 0.001 g and be sure to record your balance readings.
- d) Place the sample in a clean 125 or 250 mL Erlenmeyer flask, dissolve it in about 25 mL of distilled water and add two drops of phenolphthalein indicator.
- e) Rinse your buret once more with distilled water and then several times with small (5-10 mL) portions of the NaOH solution you have prepared, draining off the solution through the buret tip. Fill the buret nearly to the top of the graduated portion with the NaOH solution and make sure that the buret tip is full of solution. With a piece of paper towel, remove any drop of NaOH hanging from the tip. See your instructor if your buret does not drain properly, if there are air bubbles trapped in the tip, or if it leaks.
- f) Make a preliminary titration to learn approximately how the titration proceeds. Place a sheet of white paper under the flask so that the color of the solution is easily observed. Read and record the position on the buret of the lowest point of the meniscus of the NaOH solution. Swirl the flask and add the NaOH solution. Occasionally rinse down the walls of the flask with distilled water from your wash bottle. Titrate until the last drop of NaOH solution leaves a permanent pink color in the solution.
- g) No drop should be left hanging on the buret tip. Read and record the level of the meniscus in the buret, and compute the volume of basic solution used in the titration.
- h) Now titrate at least one more sample of standard acid, being certain that the buret is refilled nearly to the top of the graduated portion with NaOH solution and that you use a clean flask. In this and subsequent runs you may add NaOH solution from the buret very rapidly up to about 2 mL of the volume you estimate on the basis of your first titration. Then carefully add the rest of the base drop by drop so that you can determine the end-point accurately.
- i) After performing two titrations you must *check your data for consistency*

The dilution procedure can be done approximately, since you will standardize the solution later.

What is the approximate concentration of the dilute NaOH?

The calculated mass of hydrated oxalic acid should be placed in an Erlenmeyer flask. ALWAYS USE ERLLENMEYER FLASKS FOR TITRATIONS. NEVER USE A BEAKER.

The exact amount of water added to the acid sample is not important. However, make sure your sample is completely dissolved by the time you reach the end-point of the titration.

The first appearance of a permanent pink color occurs when the NaOH that has been added is equivalent to the quantity of oxalic acid used.

using the following procedure:

$$\text{Consistency check} = \frac{\text{volume of NaOH used (ml)}}{\text{mass of sample (g)}} = \text{volume NaOH per gram}$$

This will give you the quantity of base (in mL) you would have used each time if the acid sample had weighed 1 gram. *These values should agree within  $\pm 0.3$  mL* If not, you will need to carry out more titrations until satisfactory agreement is obtained.

**The solution of NaOH you have just standardized will now be used to determine the molar mass of an unknown acid. Do not waste the solution or throw it away!**

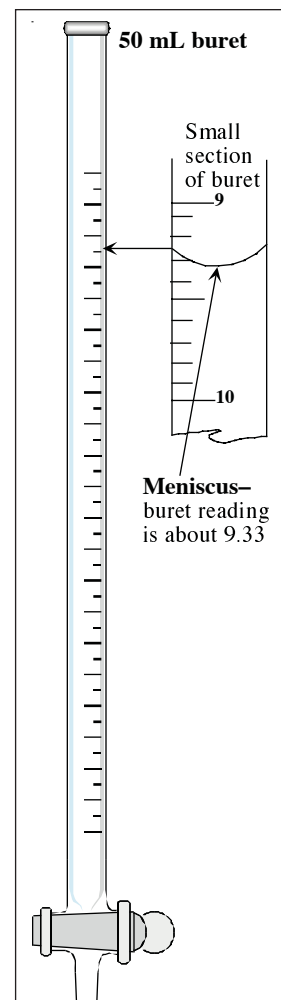
## 2. Determination of the Molar Mass of an Unknown, Diprotic Acid

Your instructor will give you a portion of an unknown solid acid in a stoppered test tube. Take this sample and two clean Erlenmeyer flasks to your analytical balance and weigh out two samples to the nearest 0.001 g; the samples should be about 1 gram each.

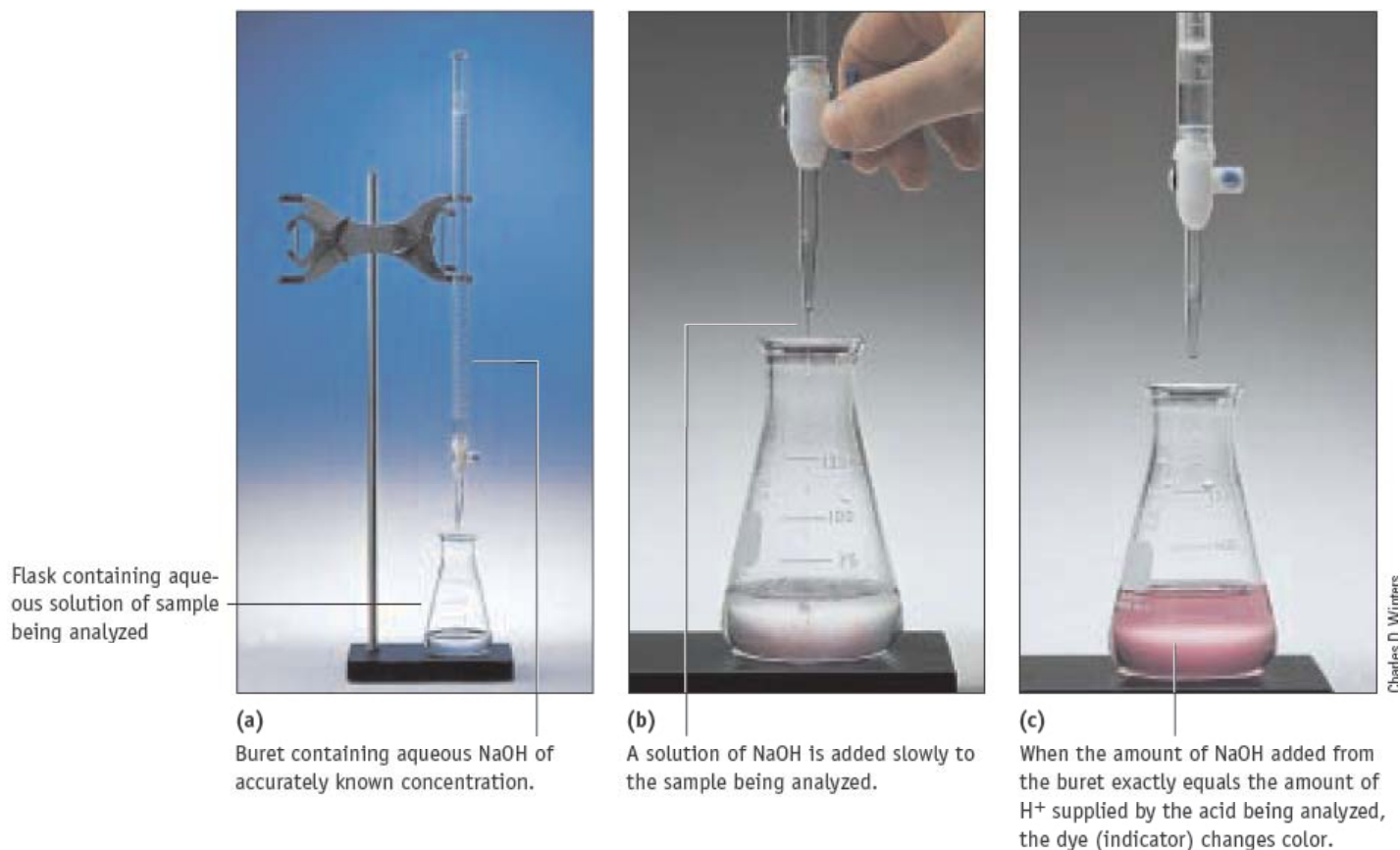
The unknown number is on a gummy label on the test tube. *Transfer this label to your Report Form immediately!* It is your responsibility to know this number. Your instructor does not keep a record of these numbers.

Add 50 mL of distilled water and two drops of phenolphthalein to each flask, and titrate with the standardized NaOH solution to a permanent faint pink endpoint. Your unknown sample may not all dissolve when you add distilled water, but the solution process should be completed as you add NaOH; make sure that it is dissolved at the endpoint.

Check your data for consistency as in the standardization section above. If the two runs on the unknown are not consistent with one another, perform another titration.



**Reading the buret.** The volume in the buret is read, to the nearest 0.01 mL, at the bottom of the meniscus (the concave surface of the aqueous solution). Here the reading would be approximately 9.33 mL.



**Active Figure 5.23** Titration of an acid in aqueous solution with a base. (a) A buret, a volumetric measuring device calibrated in divisions of 0.1 mL, is filled with an aqueous solution of a base of known concentration. (b) Base is added slowly from the buret to the solution containing the acid being analyzed and an indicator. (c) A change in the color of an indicator signals the equivalence point. (The indicator used here is phenolphthalein.)

**GENERAL Chemistry Now™** See the General ChemistryNow CD-ROM or website to explore an interactive version of this figure accompanied by an exercise.

See page 216 of *Chemistry & Chemical Reactivity*, 6th edition. See also Screen 5.19 of the *ChemistryNow CD-ROM*.

See also <http://www.dartmouth.edu/~chemlab/techniques/titration.html>