

# EXPERIMENT 5

## ACID-BASE TITRATION

### INTRODUCTION

Much of chemistry and biology is concerned with the behavior of acids and bases. Acids and bases are participants in many reactions in nature, and many reactions require a particular level of acidity or basicity. For example, the concentration of acid in the stomach must be quite high, in comparison to the rest of the body, to break down proteins during digestion.

This experiment involves the determination of the amount of acid or base in an aqueous solution, using an acid-base titration. A *titration* is the addition of a carefully measured volume of a solution of one substance to a solution of another substance until a reaction between the two substances is just complete. The titration in this experiment is based on a fundamental chemical property of acids and bases: their neutralization reaction producing water and a salt:



for which the net ionic equation is



If the amount of acid in one solution is known, then the amount of base in a second solution can be determined by measuring how much of it is required to neutralize the acid. Similarly, if the amount of base in one solution is known, the amount of acid in another solution can be determined by a titration.

EQUATION 5-1 can be used as a general equation to represent acid-base neutralization reactions. However, the generalized formulas in it do not accurately represent all acids and bases, for some acids contain more than one reactive hydrogen atom per formula unit and some bases contain (or produce) more than one hydroxide ion per formula unit; examples are  $\text{H}_2\text{SO}_4$ ,  $\text{H}_3\text{PO}_4$ ,  $\text{Mg}(\text{OH})_2$ , and  $\text{Fe}(\text{OH})_3$ . EQUATION 5-2 is a more general equation; it shows that a neutralization reaction involves equimolar amounts of hydrogen ions (in aqueous solution as hydronium ions) from the acid and hydroxide ions from the base:

The term *equivalent mass* emphasizes the essential chemical behavior of acids and bases. One equivalent mass of an acid is the mass of the acid that contains one mole of ionizable hydrogen atoms. One equivalent mass of a base is the mass of the base that produces one mole of hydroxide ions. For some compounds, equivalent mass equals molar mass; examples are  $\text{HCl}$ ,  $\text{HC}_2\text{H}_3\text{O}_2$ ,  $\text{NaOH}$ , and  $\text{NH}_3$ . For other compounds, equivalent mass is a particular fraction of the molar mass. For example, one mole of  $\text{Ca}(\text{OH})_2$  contains two moles of hydroxide ions and so one equivalent = 1/2 mole = 37 g. For  $\text{H}_3\text{PO}_4$ , one equivalent = 1/3 mole = 33g. In each case:

$$\text{equivalent mass} = \frac{\text{molar mass}}{\text{number of H}^+ \text{ or OH}^- \text{ per formula unit}} \quad (5-3)$$

In an acid-base titration, the *equivalence point* is reached when equimolar amounts of hydrogen ion from acid and hydroxide ion from base have been combined in solution, EQUATION 5-2. We can also say that at the equivalence point, equal numbers of equivalent masses (or equivalents) of acid and base have been combined. If the identity of one reactant is known, the number of equivalents of that reactant can be calculated. The titration requires the same number of equivalents of the other reactant, and so the number of equivalents of it is known also. Since you will be titrating an unknown acid with a solution of sodium hydroxide,

$$\text{at the equivalence point, equivalents of H}_3\text{O}^+ = \text{moles of OH}^- \quad (5-4)$$

## TECHNIQUE

You will carry out acid-base titrations using the acid-base indicator phenolphthalein. It is colorless in acidic solutions and pink in basic solutions; the color ranges from pale pink to dark purplish pink, depending on the concentrations of indicator and base. If you titrate to the appearance of a light pink color, the amount of excess base will be numerically insignificant; hence, this lightest possible pink color indicates the end point. By choosing an indicator properly, endpoint = equivalence point.

Your first activity will be to *standardize* a solution of NaOH; this means that you determine its concentration as accurately and precisely as possible. The NaOH solution supplied is labeled as having a concentration of 0.1M; you will determine the actual concentration to at least three significant figures. You do this by using the NaOH to neutralize an accurately weighed sample of  $\text{KHC}_8\text{H}_4\text{O}_4$  (potassium hydrogen phthalate, KHP), a monoprotic acid that has a molar mass of 204.22 g. You can calculate the number of moles of acid used in the titration:

$$\text{Moles KHP} = \text{g KHP/molar mass KHP} \quad (5-5)$$

Since KHP is monoprotic, this is the same as the number of moles of hydrogen ion provided by the acid. It is also the number of moles of hydroxide ions from the base that the acid neutralizes. The NaOH solution is dispensed from a buret and its volume is carefully measured; you use the numbers of moles of hydroxide ions and volume of NaOH solution to calculate a precise molarity (moles/liter) of the NaOH solution.

You will use your *standardized* NaOH solution to determine the equivalent mass of an unknown solid acid. Since you do not know whether your unknown acid is mono- di- or triprotic, you will generate enough data to determine the acid's equivalent mass, but not its molar mass. You do this by using your standardized NaOH solution to neutralize a carefully weighed sample of the acid. Using the molarity and volume of the NaOH solution, you can calculate moles of hydroxide ions required to neutralize the acid. At the equivalence point of the titration, this equals equivalents of hydrogen ions provided by the acid, EQUATION 5-4. The equivalent mass of the acid is the mass of the acid that contains one mole of ionizable hydrogen. Therefore, you can calculate the equivalent mass from your data:

$$\text{equivalent mass unknown acid} = \text{g unknown acid/equivalents of H}_3\text{O}^+ \quad (5-6)$$

## EQUIPMENT NEEDED

analytical balance  
beakers  
buret  
buret clamp  
Erlenmeyer flasks  
graduated cylinders

## CHEMICALS NEEDED

$\text{KHC}_8\text{H}_4\text{O}_4$ ; potassium hydrogen phthalate (KHP)  
0.1M NaOH; sodium hydroxide  
phenolphthalein indicator solution  
unknown solid acid

## PROCEDURE

### A. Standardization of NaOH Solution

1. Obtain ~100 mL of 0.1 M NaOH solution in a beaker. Clean a buret, rinse the buret with a small amount of the solution, and then fill it with the NaOH solution. Be sure to drain some of the solution so that the solution completely fills the tip of the buret.
2. On the analytical balance, weigh out 0.2–0.3 g of  $\text{KHC}_8\text{H}_4\text{O}_4$  (be sure to record the mass to as many decimal places as the balance displays) and put it into an Erlenmeyer flask. To do this, first place a small beaker or weighing boat on the analytical balance, and press the bar or button on the balance to zero the balance. Then add 0.2–0.3 g of the KHP to this container. Go back to your laboratory desk and pour the acid into the flask; use a stream of water from a wash bottle to complete the transfer. Dissolve the acid in about 20 mL of distilled water. Add two drops of phenolphthalein indicator solution to this.
3. Record the initial buret reading to the nearest 0.01 mL. If there is a drop of solution on the tip of the buret, remove it by touching the tip of the buret to the inside wall of a waste beaker. Place the flask (containing the acid) under the buret and place a piece of white paper under the flask so that you can easily see any color in the solution. Position the buret so that the tip of it is just slightly below the rim of the flask.
4. Begin the titration by adding several mL of base solution in a rapid stream; constantly swirl the flask. At the site of addition, there will be a pink color that disappears quickly as the acid and base solutions mix. When this color seems to linger, turn the stopcock to shut off the flow of solution from the buret. At this point, you can introduce the NaOH solution into the flask by turning the stopcock 180 degrees. The speed with which you do this will determine how much solution is allowed into the flask—a leisurely turn will introduce a few tenths of a milliliter, while a quick flick will introduce a single drop. Decrease the rate of addition of base as the pink color persists longer before it disappears (remember to swirl after each addition of solution). Occasionally wash solution off the inside wall of the flask with a stream of water from a wash bottle. Add base until the lightest possible pink color