EXPERIMENT 24

Quantitative Titration

Relative amounts of reactants and products of a reaction are commonly investigated in two ways in the laboratory: gravimetrically (by mass)—as in Experiments 7, 8, 10, 12, and 13—and volumetrically (by volume and concentration)—as in this experiment. **Titration** is the name given to the process for determining the volume of a solution needed to react with a given mass, or volume, of a sample. You will use this process to study quantitatively the reaction between an acid and a base. A common reaction in water solution is that of the hydrogen ion of an acid with the hydroxide ion of a base to form water. Phenolphthalein will be used as the indicator in this experiment since its color change occurs when the same number of moles of acid and base have been added. This point in the reaction is called the **endpoint**.

PROCEDURE

Using hydrochloric acid of known concentration, you will first standardize a sodium hydroxide solution—that is, determine its concentration expressed as moles per litre. Using this standard base, you will then titrate a known mass of an unknown solid acid and calculate the number of grams of this acid that will react with one mole of the base. After this experimental value has been determined, your teacher will tell you the formula of the acid. You will then write the equation for the reaction and calculate the value for the number of grams of acid that will react with one mole of the base and compare it with your experimental value.

PART I STANDARDIZATION OF THE SOLUTION OF A BASE

a. Obtain two burets. See the section on the care of burets at the end of this experiment. Clean the burets and rinse one with 10 ml of the standard hydrochloric acid. Rinse the other with 10 ml of the sodium hydroxide solution you are *instructed to use.* If there are not enough burets, you will use pipets to measure the volume of acid (see Experiment 12 for an explanation of their use). After rinsing the burets, fill the first with the standard acid and the second with the base. See Figure 24-2.

b. Record the liquid level in each buret by reading the bottom of each meniscus to the nearest 0.1 ml. Let about 10 ml of hydrochloric acid flow into a clean 250-ml Erlenmeyer flask. Add about 15 ml of distilled water and 3 drops of phenolphthalein.

c. Hold the neck of the Erlenmeyer flask with one hand and manipulate the buret with the other. As you add the sodium hydroxide, gently swirl the flask so the solutions will become mixed. Continue adding sodium hydroxide until the first faint pink color develops. If the color disappears upon mixing the solution, add more sodium hydroxide, drop by drop, until a persistent pink color is obtained. If you go beyond this endpoint, you may add a few drops of acid, and then complete the titration with a few more drops of sodium hydroxide. (Take care not to go beyond the last calibration marks on the buret.) Record the liquid level at the bottom of the meniscus of each buret. Rinse the Erlenmeyer flask thoroughly before repeating the titration.

d. Refill the burets with the proper solutions and perform at least one more titration. Repeat until you obtain ratios of volume of acid to volume of base which agree to 1% or 2%.

PART II TITRATION OF AN UNKNOWN ACID

a. Obtain a solid unknown acid from your teacher. Find the mass of the vial or test tube containing the sample to the nearest ±0.01 g. Remove a suitable amount (about 1 g, or as directed by your teacher) of the solid acid into a clean flask as shown in Figure 24-1. Find the mass of the vial and contents again. Dissolve the sample in 50 ml of distilled water and add 3 drops of phenolphthalein. If all the acid does not dissolve at this point, it will dissolve later during the titration when the acid will be converted to the more soluble sodium salt.

b. Refill the proper buret with some of the solution of base used previously and record the initial reading. Add the base to the acid solution until the first persistent, faint pink color appears. Be careful not to overrun the endpoint. If you pass the endpoint, add a little more of the solid acid and reweigh the vial. Be sure to include the mass of any solid acid added to the mass of your sample. Retitrate to the endpoint and record the final buret reading.

c. Repeat the titration with a similar sample. Use the knowledge you gained in the first titration. That is, assuming you used 40 ml of base to titrate a certain mass of acid, and that you have almost the same mass of acid for the second trial, you can run 35 ml of base into the flask rapidly and complete the last part of the titration cautiously.

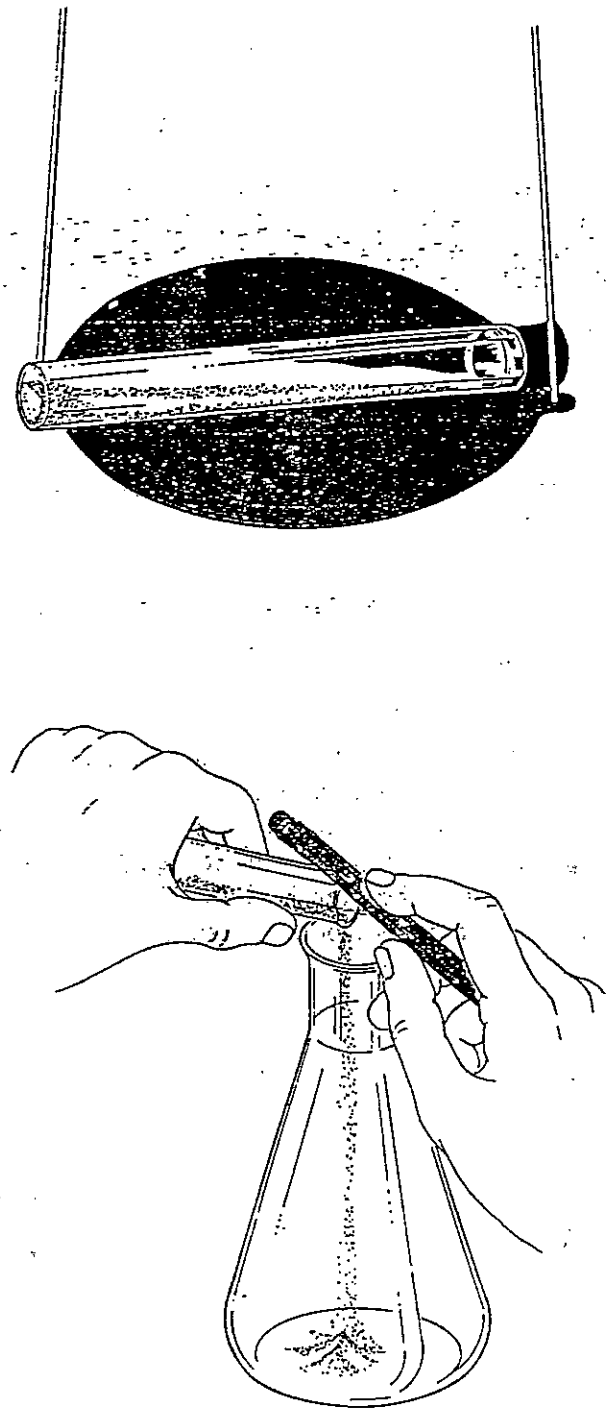

Fig. 24-1 Finding the mass of a sample and transferring it to a flask.

PART III OPTIONAL TITRATION

If time permits, you may bring from home commercially available household acidic or basic substances to titrate with either your standard acid or base. Examples of items readily available are lemon juice, vinegar, household ammonia, and washing powders. Determinations which are possible are the percent acetic acid, CH_3COOH, in vinegar; the percent citric acid, $C_6H_8O_7$, in lemon juice; the percent ammonia, NH_3, in household ammonia. If you need help in deciding on sample size or the proper indicator, ask your teacher.