40 ACID–BASE TITRATIONS
EXPERIMENT

Text Reference
Section 21.1

PURPOSE

To measure the normality of ethanoic acid in vinegar using a standardized solution of sodium hydroxide.

BACKGROUND

Every day scientists in many fields conduct experiments designed to answer one question: How much acid or base does this solution contain? The chemical reactions used to answer this question are, for the main part, neutralization reactions, and *titration* is the method generally used.

You can neutralize an acid with a base very precisely by using the technique of titration. During a titration, a solution of known acidity (a standard solution) is gradually added to a solution of unknown basicity. At the point of neutralization, the number of equivalents of acid must be equal to the number of equivalents of base. Thus, titration tells you the equivalents of base in your unknown solution. The neutralization, or equivalence point, of the reaction is estimated by the color change of an acid–base indicator or by a neutral reading (pH 7.0) on a pH meter. You can also reverse the titration procedure so a standard base solution is used to titrate an unknown acidic solution.

In this experiment, you will prepare a standard solution of an acidic compound, potassium hydrogen sulfate ($KHSO_4$). You will then use this solution to make a standard solution of sodium hydroxide by titration. Finally, you will use your standardized sodium hydroxide solution to titrate vinegar, a dilute solution of ethanoic acid, CH_3COOH.

MATERIALS (PER PAIR)

safety goggles
1 10-mL graduated cylinder
1 100-mL graduated cylinder
2 50-mL burets
1 10-mL pipet
1 25-mL pipet
1 pipet suction bulb
1 100-mL beaker
1 100-mL volumetric flask
1 250-mL volumetric flask
2 250-mL Erlenmeyer flasks
3 250-mL plastic bottles
1 ring stand
1 double buret clamp
1 spatula
centigram balance
1 filter funnel
2 rubber stoppers, for volumetric flasks
1 weighing bottle
1 plastic wash bottle
labels
2 sheets white paper, 25 cm × 25 cm
potassium hydrogen sulfate, $KHSO_4$ $\boxed{T}$
6*M* sodium hydroxide, NaOH $\boxed{C}$ $\boxed{T}$
phenolphthalein solution
vinegar, dilute ethanoic acid, CH_3COOH
distilled water

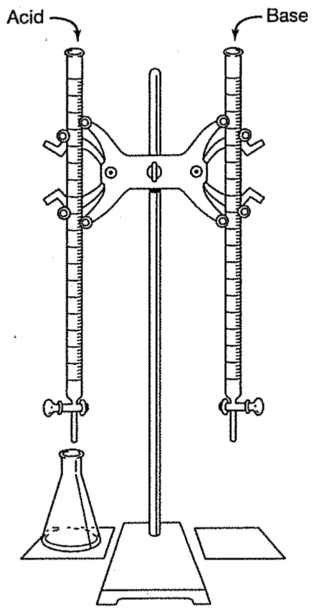

Figure 40.1

SAFETY FIRST!

In this lab, observe all precautions, especially the ones listed below. If you see a safety icon beside a step in the procedure, refer to the list below for its meaning.

Caution: Wear your safety goggles. (All steps.)

Caution: Sodium hydroxide is corrosive and can cause severe burns. (Steps 4–16.)

Caution: Potassium hydrogen sulfate is a toxic substance. (Steps 1–13.)

Note: Return or dispose of all materials according to the instructions of your teacher. (Steps 6, 12, and 16.)

PROCEDURE

As you perform the experiment, record your data and observations in Data Tables 1, 2, and 3.

Part A. Preparation of a Standard Solution of Potassium Hydrogen Sulfate

1. Determine the mass of a sample of potassium hydrogen sulfate (the *acid*) in a weighing bottle using the most accurate balance available to you. Using a funnel and a spatula, transfer 3–4 g of the acid to a clean 250-mL volumetric flask. Remeasure the mass of the weighing bottle and remaining sample. The difference between the two mass values is the mass of the acid in the flask.
2. Add about 100 mL of distilled water to the flask, washing into the flask any acid crystals clinging to the funnel. Remove the funnel and gently swirl the flask to dissolve the acid. When the acid is completely dissolved, fill the flask to the 250-mL mark with distilled water.
3. Stopper and mix the contents thoroughly by inverting the flask and swirling the mixture. Transfer the solution to a clean, dry 250-mL plastic bottle labeled "approximately 0.1N $KHSO_4$" and mark the label with your initials.

Part B. Standardization of a Solution of Sodium Hydroxide

4. **CAUTION:** *Sodium hydroxide is corrosive.* Using a 10-mL graduated cylinder, measure out 5 mL of 6M sodium hydroxide. Transfer this solution to a clean dry 250-mL plastic bottle labeled "approximately 0.1N NaOH" and mark the label with your initials. Add 250 mL of distilled water to the bottle, cap it, and shake it to mix the contents.
5. Clean and mount two 50-mL burets, as shown in Figure 40.1. Place a white sheet of paper or a white plastic base beneath each buret. Label the left buret "acid" and the right buret "base."

6. Rinse the "acid" buret with three 5-mL portions of the standard solution of potassium hydrogen sulfate. Let each portion drain out of the buret before adding the next rinse. Discard these rinses. Fill the buret with the potassium hydrogen sulfate solution. Before beginning the titration, remove any bubbles trapped in the tip of the buret and the stopcock.

7. Using the sodium hydroxide solution, rinse and fill the "base" buret. Use a wash bottle of distilled water to rinse off the tip of each buret; catch the runoff in a 100-mL beaker. Record the initial volume in each buret to the nearest 0.01 mL.

8. Add 10–12 mL of the acid solution to a clean 250-mL Erlenmeyer flask. Use the wash bottle to rinse the last drop of acid from the tip of the buret into the flask. Add 50 mL of distilled water and 1–2 drops of phenolphthalein to the flask.

9. Now, slowly add sodium hydroxide solution from the "base" buret to the flask. As you add the base, gently swirl the solution in the flask. A pink color will appear and quickly disappear as the solutions are mixed. As more and more base is added, the pink color will persist for a longer time before disappearing. This is a sign that you are nearing the equivalence point, also called the end point. Wash down the sides of the flask and the tip of the buret with distilled water from the wash bottle. Continue to add sodium hydroxide more slowly, until a single drop of base turns the solution a pale pink color that persists for 15–30 seconds.

10. If you overshoot the end point—that is, if you add too much base so the solution turns bright pink—simply add a few drops of acid from the acid buret to turn the solution colorless again. Approach the end point again, adding base drop by drop, until one drop causes the color change to pale pink.

11. When you are sure that you have achieved the end point, record the final volume reading of each buret. Note: Do not allow the level of the solution in either buret to go below the 50-mL mark. If you do, you will have to discard your sample and begin again.

12. Discard the solution in the Erlenmeyer flask as directed by your teacher, and rinse the flask well with distilled water. Refill both burets, if necessary. Read the initial volume in each buret and do another titration, as described in Steps 8–11.

13. Before proceeding, calculate the normality of the sodium hydroxide solution for each titration, as described in Analyses and Conclusions, problems 1–6. If the normalities obtained from the two titrations do not agree within 1%, perform a third titration.

Part C. Determination of the Normality of Ethanoic Acid in Vinegar

14. Using a clean, dry 10-mL pipet and suction bulb, transfer 10 mL of commercial vinegar into a clean 100-mL volumetric flask. Fill the flask to the 100-mL mark with distilled water. Stopper the flask and mix the solution by inverting the flask 20–30 times. Transfer this solution to a clean, dry 250-mL plastic bottle. Label the bottle "10% vinegar" and mark the label with your initials.
15. Using a clean, dry 25-mL pipet, transfer a 20-mL sample of the diluted vinegar to a clean 250-mL Erlenmeyer flask. Add 50 mL of distilled water and 1–2 drops of phenolphthalein to the flask. Titrate the vinegar with the sodium hydroxide solution that you standardized in Part B. Note: If you overshoot the end point in these titrations, you will have to discard the sample and begin again. Do at least two titrations that agree to within 1%.
16. Follow your teacher's instructions for proper disposal of the materials.

Name ______________________ Class __________ Date __________

OBSERVATIONS

DATA TABLE 1: NORMALITY OF POTASSIUM HYDROGEN SULFATE	
initial mass of weighing bottle and $KHSO_4$	
final mass of weighing bottle and $KHSO_4$	
mass of $KHSO_4$ used	
gram equivalent mass of $KHSO_4$	
equivalents of $KHSO_4$ used in 250 mL	
normality of $KHSO_4$	

DATA TABLE 2: NORMALITY OF SODIUM HYDROXIDE

	Trial 1		Trial 2		Trial 3	
	Acid	Base	Acid	Base	Acid	Base
final volume						
initial volume						
volume used						
normality of NaOH						
average normality of NaOH						

DATA TABLE 3: NORMALITY OF VINEGAR

	Trial 1		Trial 2		Trial 3	
	Vinegar	Base	Vinegar	Base	Vinegar	Base
final volume						
initial volume						
volume used						
normality of vinegar						
average normality of 10% vinegar						

Name ________________________ Class ____________ Date ____________

ANALYSES AND CONCLUSIONS

As you do the following calculations, enter the results in Data Table 1, Data Table 2, or Data Table 3.

1. Determine the mass of potassium hydrogen sulfate used.
2. Find the gram equivalent mass of potassium hydrogen sulfate.

$$\text{gram equivalent mass} = \frac{\text{gram formula mass}}{\text{number of equivalents per mole}}$$

3. Calculate the number of equivalents of potassium hydrogen sulfate used.

$$\text{number of equivalents} = \frac{\text{mass}}{\text{gram equivalent mass}}$$

4. Determine the normality of your standard solution of potassium hydrogen sulfate. Be sure to use units properly in your calculation.

$$\text{normality} = \frac{\text{number of equivalents}}{\text{volume}}$$

5. Determine the volumes of acid and base used in each titration performed in Part B.
6. The normality of the sodium hydroxide solution is calculated with this equation:

$$\text{normality}_{\text{base}} = \text{normality}_{\text{acid}} \times \frac{\text{volume}_{\text{acid}}}{\text{volume}_{\text{base}}}$$

Calculate the normality of your sodium hydroxide solution for each titration. Record the value obtained for each trial, as well as the average value for all trials.

7. Determine the volume of 10% vinegar and sodium hydroxide solutions used in each titration.

Name ______________________ Class ______________ Date ______________

8. Using the average normality of sodium hydroxide that was calculated in problem 6, determine the normality of your 10% vinegar solution (ethanoic acid).

$$\text{normality}_{acid} = \text{normality}_{base} \times \frac{\text{volume}_{base}}{\text{volume}_{acid}}$$

Record the value for each trial and the average value.

9. Examine the class results for the normality of vinegar. Account for any difference among these values.

10. Explain why the plastic bottles into which you transferred your potassium hydrogen sulfate and vinegar solutions had to be dry, but you could add distilled water to the titration flask at any time and not affect your results.

Name ______________________ Class ____________ Date ____________

11. Why are the burets rinsed with the acid and base solutions before filling?

12. Use your data from this experiment to calculate the mass/volume percent of ethanoic acid present in commercial, undiluted vinegar.

13. Explain why you cannot prepare a standard sodium hydroxide solution by weighing the solid and dissolving it in a measured amount of water, as you did with the potassium hydrogen sulfate.

GOING FURTHER

Develop a Hypothesis

Based on the results of this lab, develop a hypothesis about how the pH of the 10% solution of vinegar changes during the titration with NaOH. In what ways would the pH curves for the titration of ethanoic acid (vinegar), ascorbic acid (vitamin C), and citric acid (lemon juice) be the same? Different?

Design an Experiment

Propose an experiment to test your hypothesis. If resources are available and you have your teacher's permission, perform the experiment.