EXPERIMENT

27 Acid/Base Titrations

Choice I. Analysis of an Unknown Acid Sample

Objective

An unknown acid, either a sample of vinegar or an acid salt, will be analyzed by the process of titration, using a standard sodium hydroxide solution. The sodium hydroxide solution to be used for the analysis will be prepared approximately and will then be standardized against a weighed sample of a known acidic salt.

Introduction

The technique of titration finds many applications, but is especially useful in the analysis of acidic and basic substances. Titration involves measuring the exact volume of a solution of known concentration that is required to react with a measured volume of a solution of unknown concentration, or with a weighed sample of unknown solid. A solution of accurately known concentration is called a **standard solution.** Typically, to be considered a standard solution, the concentration of the solute in the solution must be known to four significant figures.

In many cases (especially with solid solutes) it is possible to prepare a standard solution by accurate weighing of the solute, followed by precise dilution to an exactly known volume in a volumetric flask. Such a standard is said to have been prepared determinately. One of the most common standard solutions used in analyses, however, cannot be prepared in this manner.

Solutions of sodium hydroxide are commonly used in titration analyses of samples containing an acidic solute. Although sodium hydroxide is a solid, it is not possible to prepare standard sodium hydroxide solutions by weight. Solid sodium hydroxide is usually of questionable purity. Sodium hydroxide reacts with carbon dioxide from the atmosphere and is also capable of reacting with the glass of the container in which it is provided. For these reasons, sodium hydroxide solutions are generally prepared to be approximately a given concentration. They are then standardized by titration of a weighed sample of a primary standard acidic substance. By measuring how many milliliters of the approximately prepared sodium hydroxide are necessary to react completely with a weighed sample of a known primary standard acidic substance, the concentration of the sodium hydroxide solution can be calculated. Once prepared, however, the concentration of a sodium hydroxide solution will change with time (for the same reasons outlined earlier). As a consequence, sodium hydroxide solutions must be used relatively quickly.

In titration analyses, there must be some means of knowing when enough titrant has been added to react exactly and completely with the sample being titrated. In an acid/base titration analysis, there should be an abrupt change in pH when the reaction is complete. For example, if the sample being titrated is an acid, then the titrant to be used will be basic (probably sodium hydroxide). When one excess drop of titrant is added (beyond that needed to react with the acidic sample), the solution being titrated will suddenly become basic. There are various natural and synthetic dyes, called indicators, that exist in different colored forms at different pH values. A suitable indicator can be chosen that will change color at a pH value consistent with the point at which the titration reaction is complete. The indicator to be used in this experiment is phenolphthalein, which is colorless in acidic solutions, but changes to a pink form at basic pH.

Safety Precautions

- Wear safety glasses at all times while in the laboratory.
- The primary standard acidic substance potassium hydrogen phthalate (KHP) will be kept stored in an oven to keep moisture from adhering to the crystals. Use tongs or a towel to remove the KHP from the oven.
- Sodium hydroxide is extremely caustic, and sodium hydroxide dust is very irritating to the respiratory system. Do not handle the pellets with the fingers. Wash hands after weighing the pellets. Work in a ventilated area and avoid breathing NaOH dust.
- Use a rubber safety bulb when pipetting. *Never pipet by mouth.*
- The unknowns to be used are acidic and may be irritating/damaging to the skin. Avoid contact, and wash after using them.

Apparatus/Reagents Required

two burets and clamp, buret brush, 5-mL pipet and safety bulb, soap, 1-L glass or plastic bottle with stopper, sodium hydroxide pellets, primary standard grade potassium hydrogen phthalate (KHP), phenolphthalein indicator solution, unknown vinegar or acid salt sample

Procedure

Record all data and observations directly in your notebook in ink.

A. Preparation of the Burets and Pipet

For precise quantitative work, volumetric glassware must be scrupulously clean. Water should run down the inside of burets and pipets in sheets and should not bead up anywhere on the interior of the glassware. Rinse the burets and the pipet with distilled water to see if they are clean.

If not, partially fill with a few milliliters of soap solution, and rotate the buret/pipet so that all surfaces come in contact with the soap.

Rinse with tap water, followed by several portions of distilled water. If the burets are still not clean, they should be scrubbed with a buret brush. If the pipet cannot be cleaned, it should be exchanged.

In the subsequent procedure, it is important that water from rinsing a pipet/buret does not contaminate the solutions to be used in the glassware. This rinse water would change the concentration of the glassware's contents. Before using a pipet/buret in the following procedures, rinse the pipet/buret with several small portions of the solution that is to be used in the pipet/buret. Discard the rinsings.

B. Preparation of the Sodium Hydroxide Solution

Clean and rinse the 1-L bottle and stopper. Label the bottle "Approx. 0.1 *M* NaOH." Put about 500 mL of distilled water into the bottle.

Weigh out approximately 4 g (0.1 mol) of sodium hydroxide pellets (*Caution*!) and transfer to the 1-L bottle. Stopper and shake the bottle to dissolve the sodium hydroxide.

When the sodium hydroxide pellets have dissolved, add additional distilled water to the bottle until the water level is approximately 1 inch from the top. Stopper and shake thoroughly to mix.

This sodium hydroxide solution is the titrant for the analyses to follow. Keep the bottle tightly stoppered when not actually in use (to avoid exposure of the NaOH to the air).

Set up one of the burets in the buret clamp. See Figure 27-1 on the following page. Rinse and fill the buret with the sodium hydroxide solution just prepared.

C. Standardization of the Sodium Hydroxide Solution

Clean and dry a small beaker. Take the beaker to the oven that contains the primary standard grade potassium hydrogen phthalate (KHP).

Using tongs or a towel to protect your hands, remove the bottle of KHP from the oven, and pour a few grams into the beaker. If you pour too much, do *not* return the KHP to the bottle. Return the bottle of KHP to the oven, and take the beaker containing KHP back to your lab bench.

Allow the KHP to cool to room temperature. While the KHP is cooling, clean three 250-mL Erlenmeyer flasks with soap and water. Rinse the Erlenmeyer flasks with 5–10 mL portions of distilled water. Label the Erlenmeyer flasks as 1, 2, and 3.

When the KHP is completely cool, weigh three samples of KHP between 0.6 and 0.8 g, one for each of the Erlenmeyer flasks. Record the exact weight of each KHP sample at least to the nearest milligram, preferably to the nearest 0.1 mg (if an analytical balance is available). Be certain not to confuse the samples.

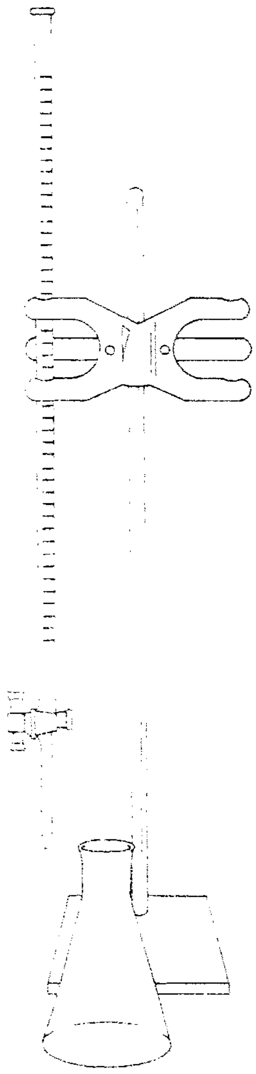

Figure 27-1. Set-up for titration.

At your lab bench, add 100 mL of water to KHP sample 1. Add 2–3 drops of phenolphthalein indicator solution. Swirl to dissolve the KHP sample completely.

Record the initial reading of the NaOH solution in the buret to the nearest 0.02 mL, remembering to read across the bottom of the curved solution surface (meniscus).

Begin adding NaOH solution from the buret to the sample in the Erlenmeyer flask, swirling the flask constantly during the addition. (See Figure 27-2.) If your solution was prepared correctly, and if your KHP samples are of the correct size, the titration should require at least 20 mL of NaOH solution. As the NaOH solution enters the solution in the Erlenmeyer flask, streaks of red or pink will be visible. They will fade as the flask is swirled.

Eventually the red streaks will persist for a longer and longer period of time. This indicates the approach of the endpoint of the titration.

Begin adding NaOH one drop at a time, with constant swirling, until one single drop of NaOH causes a permanent pale pink color that does not fade on swirling. Record the reading of the buret to the nearest 0.02 mL.

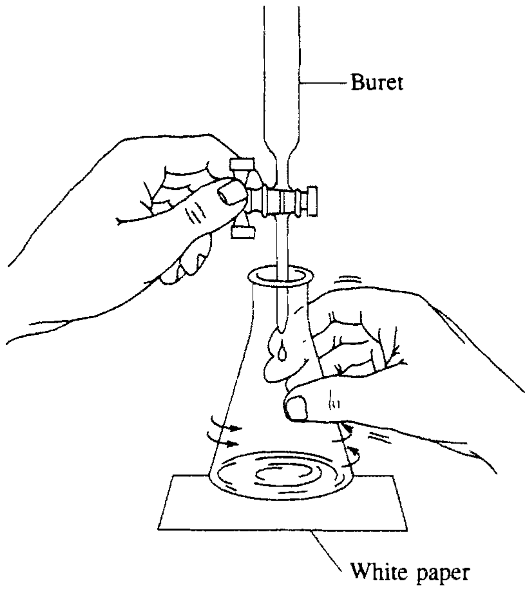

Figure 27-2. Titration technique. A right-handed person should titrate with the left hand, swirling the flask with the right hand.

Repeat the titration of the remaining two KHP samples. Record both initial and final readings of the buret to the nearest 0.02 mL.

Given that the molecular weight of potassium hydrogen phthalate is 204.2, calculate the number of moles of KHP in samples 1, 2, and 3.

From the number of moles of KHP present in each sample, and from the volume of NaOH solution used to titrate the sample, calculate the concentration of NaOH in the titrant solution in moles per liter. The reaction between NaOH and KHP is of 1:1 stoichiometry.

If your three values for the concentration differ by more than 1%, weigh out an additional sample of KHP and repeat the titration. Use the average concentration of the NaOH solution for subsequent calculations for the unknown.

D. Analysis of the Unknown Acid Sample

Two types of unknown acid samples may be provided. Your instructor may ask you to analyze either or both of these.

1. Analysis of a Vinegar Solution

Vinegar is a dilute solution of acetic acid and can be effectively titrated with NaOH using the phenolphthalein endpoint.

Clean and dry a small beaker, and obtain 25–30 mL of the unknown vinegar solution. Cover the vinegar solution with a watch glass to prevent evaporation. Record the code number of the sample.

Clean three Erlenmeyer flasks, and label as samples 1, 2, and 3. Rinse the flasks with small portions of distilled water.

Using the rubber safety bulb to provide suction, rinse the 5-mL pipet with small portions of the vinegar solution and discard the rinsings.

Using the rubber safety bulb, pipet a 5-mL sample of the vinegar solution into each of the Erlenmeyer flasks. Add approximately 100 mL of distilled water to each flask, as well as 2–3 drops of phenolphthalein indicator solution.

Refill the buret with the NaOH solution and record the initial reading of the buret to the nearest 0.02 mL. Titrate Sample 1 of vinegar in the same manner as in the standardization until one drop of NaOH causes the appearance of the pale pink color.

Record the final reading of the buret to the nearest 0.02 mL.

Repeat the titration for the other two vinegar samples.

Based on the volume of vinegar sample taken, and on the volume and average concentration of NaOH titrant used, calculate the concentration of the vinegar solution in moles per liter.

Given that the formula weight of acetic acid is 60.0, and that the density of the vinegar solution is 1.01 g/mL, calculate the percent by weight of acetic acid in the vinegar solution.

2. Analysis of a Solid Acid

As you saw with the KHP used in the standardization of NaOH, some solid substances are quite acidic. Your instructor will provide you with a solid acidic unknown substance and will tell you approximately what weight of the substance to use in your analysis. Record the code number of the sample.

Clean three Erlenmeyer flasks and label them as samples 1, 2, and 3.

Based on the instructor's directions, weigh out three samples of the solid unknown, one into each Erlenmeyer flask. Make the weight determination at least to the nearest milligram, or preferably, to the nearest 0.1 mg (if an analytical balance is available).

Dissolve the unknown samples in approximately 100 mL of distilled water, and add 2–4 drops of phenolphthalein indicator solution.

Fill the buret with the NaOH titrant and record the initial volume to the nearest 0.02 mL. Titrate sample 1 to the pale pink endpoint as described in the standardization of NaOH above. Record the final volume to the nearest 0.02 mL. Repeat the titration for samples 2 and 3.

Based on the weight of unknown sample taken, and the volume and concentration of the NaOH used to titrate the sample, calculate the molecular weight of the solid unknown acid.

Name ______________________ Section ______________

Lab Instructor ______________________ Date ______________

EXPERIMENT 27 Acid/Base Titrations

PRE-LABORATORY QUESTIONS

CHOICE I. ANALYSIS OF AN UNKNOWN ACID SAMPLE

1. Using your textbook or a handbook, look up the formula and structure of potassium hydrogen phthalate (KHP) used to standardize the solution of NaOH in this experiment. Calculate the formula weight of KHP.

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2. Using your textbook or a chemical dictionary, write the definition of *indicator*.

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3. Suppose a sodium hydroxide solution were to be standardized against pure solid primary standard grade KHP. If 0.4538 g of KHP requires 44.12 mL of the sodium hydroxide to reach a phenolphthalein endpoint, what is the molarity of the NaOH solution?

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CHOICE II. ANALYSIS OF STOMACH ANTACID TABLETS

1. What is meant by a standard solution of acid or base?

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2. Some of the common bases used as the active ingredient in commercial antacid tablets are listed. Calculate the number of milliliters of 0.100 *M* HCl solution that could be neutralized by 1.00 g of each of the substances.

$CaCO_3$ ______________________________ $NaHCO_3$ ______________________________

$Mg(OH)_2$ ______________________________ $Al(OH)_3$ ______________________________

Name ______________________ Section ______________

Lab Instructor ______________________ Date ______________

EXPERIMENT 27 Acid/Base Titrations

CHOICE I. ANALYSIS OF AN UNKNOWN ACID SAMPLE

RESULTS/OBSERVATIONS

√ **A. Standardization of NaOH Titrant**

	Sample 1	Sample 2	Sample 3
Weight of KHP taken √			
Initial NaOH buret reading √			
Final NaOH buret reading √			
Volume NaOH used √			
★ Moles KHP present √			
★ Molarity of NaOH solution √			

★ Mean molarity of NaOH solution ______________________

Balance eq for NaOH + KHP   Why H_2O does not affect titration

B. Analysis of Vinegar Solution

Identification number of vinegar sample used ______________________

	Sample 1	Sample 2	Sample 3
Quantity of vinegar taken			
Initial NaOH buret reading			
Final NaOH buret reading			
Volume NaOH used			
Molarity of vinegar			

Mean molarity of vinegar ______________________

% by weight acetic acid present ______________________

C. Analysis of a Solid Acid

Identification number of solid acid used ______________________________

	Sample 1	Sample 2	Sample 3
Weight unknown taken	________	________	________
Initial NaOH buret reading	________	________	________
Final NaOH buret reading	________	________	________
Volume of NaOH used	________	________	________
Moles of NaOH used	________	________	________
Molecular weight unknown solid	________	________	________

Mean molecular weight ______________________________

QUESTIONS

1. Commercial vinegar is generally 5.0 $\pm$ 0.5% acetic acid by weight. Assuming this to be the true value for your unknown, by how much were you in error in your analysis?

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2. The solid acids chosen for the analysis were typically monoprotic acidic salts such as $NaHSO_4$, $KHSO_4$, etc. Explain why such salts behave as strong enough acids to be titratable with NaOH using phenolphthalein as indicator.

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Lab Report for Experiment 27 Acid/Base Titrations

1. Complete Pre-Laboratory Questions 1-3 Choice I Analysis of an Unknown Acid sample. (p387)

2. Recreate Choice I A. Standardization of NaOH Titrant Results/Observations. Using your 3 best titration results complete the table. Show calculation for the * data after the created data table.

3. Give a complete balanced equation for the reaction of sodium hydroxide with potassium phthalate.

4. Explain why water does not affect the titration results.

5. Recreate Choice I C. Analysis of a Solid Acid. Show calculations for the * data after the created data table.