



Al-Rasheed University College
Department of Medical Laboratory
Technique

Medical Chemistry

Lab 5-7

by
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EXPERIMENT 5 Determining the Acid Content of Vinegars

Discussion

The total acid content of a vinegar readily determined by titration with a standard base. It is customary to report the acid content of vinegar in terms of acetic acid, the principal acidic constituent, even though other acids are present. Most vinegars contain about 5% acid (w/v) expressed as acetic acid.

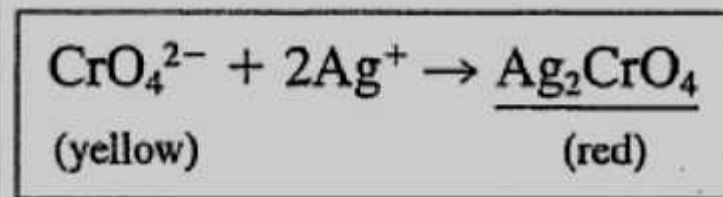
PROCEDURE

1. Pipet 2.50 mL of a vinegar into a 100-mL volumetric flask and add about 25 mL of water and 2 drops of phenolphthalein.
2. Mix thoroughly, and titrate with standard 0.1 M NaOH to the first permanent (30 s) pink color.
3. Report the acidity of the vinegar as percent (w/v) CH₃COOH (60.053 g/mol).

EXPERIMENT 6 DETERMINATION OF CHLORIDE IN A SOLUBLE CHLORIDE: MOHR' METHOD

Principle

The chloride content of a soluble sample is determined by titrated with standard AgNO_3 solution. A soluble chromate salt is added as the indicator. This produces a yellow solution. When the precipitation of the chloride is complete, the first excess of Ag^+ reacts with the indicator (CrO_4^{2-} ion) to precipitate red silver chromate (Ag_2CrO_4), which signals the end point.



Equations



Solutions and Chemicals Required

1. *Provided.* 5% Potassium chromate indicator solution.
2. *To prepare.* Standard 0.1 M AgNO₃. Obtain from the instructor about 4.5 g primary-standard-grade silver nitrate in a clean, dry weighing bottle; dry in the oven at 110 C for 1 to 2h, but no longer. This material will be used to prepare a standard solution by the direct method. Cool for 30 to 40min in a desiccator.

Using a weighing dish, weigh accurately (nearest 0.1mg) about 4.3 g AgNO₃. Transfer to a 250-mL beaker and dissolve in about 100mL distilled water. Carefully pour this solution into a 250-mL volumetric flask and rinse the beaker several times with distilled water, adding the rinsings to the flask. Dilute to the mark, shake well, and pour into a clean, dry 500-mL glass-stoppered amber bottle. Shake again to ensure a homogeneous solution. Keep away from strong light as much as possible. Store away from light. Calculate the molarity of the solution.

Things to Do before the Experiment

1. Prepare the 0.1 M AgNO₃ solution.
2. Obtain a sample of unknown chloride from your instructor and dry it in the oven at 120 C for 1 h or longer. It is okay to dry it overnight. Cool in a desiccator at least 30 to 40 min before weighing.

PROCEDURE

1. Dry NaCl at 110°C for at least 1 hr. Cool in a desiccator.
2. Weigh 0.25 g NaCl (to the nearest 0.1 mg) into 250-mL conical flasks, and dissolve in about 25 mL of distilled water.
3. Add small quantities of NaHCO₃ until effervescence ceases.
4. Introduce about 2 mL of K₂CrO₄ solution, and titrate to the first permanent appearance of red Ag₂CrO₄.
5. Report the percentage of Cl in the unknown.
6. Dispose of AgCl and reagents as directed by the instructor.



Figure 1 Before the addition of any silver nitrate the chromate indicator gives the clear solution a lemon-yellow color.

The endpoint of the titration is identified as the first appearance of a red-brown colour of silver chromate (figure 2).



1. The K_{sp} of AgCl is larger than the K_{sp} of K_2CrO_4 , yet K_2CrO_4 is more soluble than AgCl . Explain the fallaciousness of the above statement.
2. Calculate the molarity of the chloride ion in your unknown. Calculate the grams of chloride ion per liter.

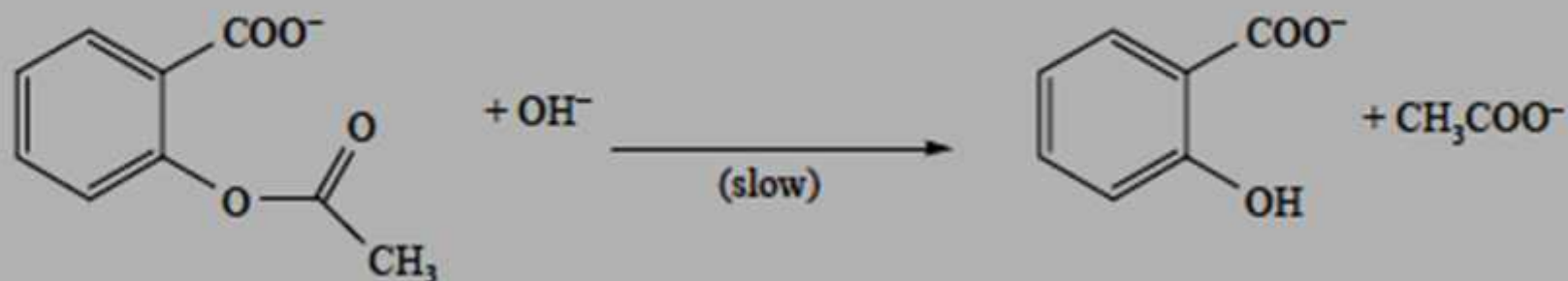
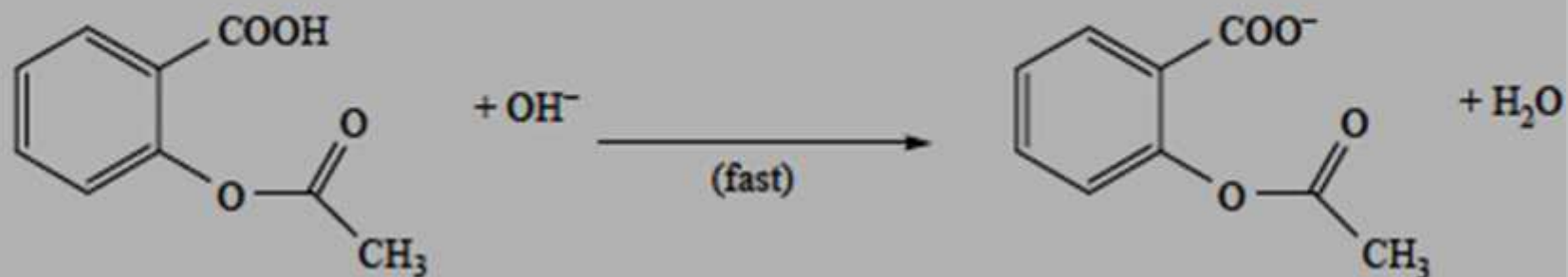
EXPERIMENT 7 DETERMINATION OF ASPIRIN USING BACK TITRATION

This experiment is designed to illustrate techniques used in a typical indirect or back titration. Standardized NaOH will be used to back titrate an aspirin solution and determine the concentration of aspirin in a typical analgesic tablet.

Principle

Many reactions are slow or present unfavorable equilibria for direct titration. Aspirin is a weak acid that also undergoes slow hydrolysis (i.e., each aspirin molecule reacts with two hydroxide ions) in a two-step reaction. The first step of the reaction is fast; the second step is very slow. To overcome the slow hydrolysis, a known excess amount of 0.1M NaOH is added and allowed to react with the aspirin. The sample is heated to speed the hydrolysis. Then, the excess NaOH is back-titrated with 0.1M HCl. Standardized 0.1M NaOH solution is supplied. A diluted, standardized solution of 0.1M HCl must be prepared.

Equations



Solutions and Chemical Required

- 1. Provided.** Aspirin tablets, Ethanol, Hydrochloric acid, 37% wt%, Phenolphthalein indicator solution (1% in ethanol), Standardized 0.1M NaOH.
- 2. To Prepare.** Standardized 0.1M HCl solution.

Procedure

1. Use a mortar and pestle to crush an appropriate amount of aspirin tablets to produce a 0.5g sample.
2. Transfer 0.5g sample into dry 100-mL Erlenmeyer flask and add 20 mL ethanol and three drops of phenolphthalein.
3. Titrate the first aspirin sample with NaOH to a permanent pink color. This pink endpoint marks the completion of the first fast step of the hydrolysis, which quickly consumes one mole of NaOH.
4. Add the known excess amount of NaOH (10 mL + volume of NaOH used to first endpoint).
5. Heat the sample for about 15 minute to quicken the slow hydrolysis step (do not boil the sample).
6. Cool the sample approximately 5 minute. The solution should be dark pink. (If it is colorless, add more phenolphthalein. If it remains colorless, add 10 mL NaOH and reheat.) The NaOH that remains in the flask will be excess NaOH that has not reacted with aspirin.
7. Titrate the excess NaOH with 0.1M HCl until the pink color disappears.

Calculation

Calculate the aspirin amount in mg. The total moles of NaOH minus moles of HCl gives the moles of NaOH that reacted with the aspirin. Since there is a 2:1 ratio of NaOH to aspirin used in the hydrolysis, half the moles of reacted NaOH will equal the moles of Aspirin in the sample. Change moles of Aspirin to grams then to milligrams.

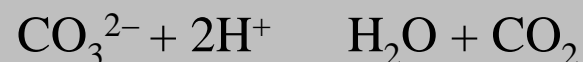
$$\text{mmol reagent reacted} = \text{mmol taken} - \text{mmol back-titrated}$$

$$\begin{aligned} \text{mg analyte} &= \text{mmol reagent reacted} \times \text{factor (mmol analyte/mmol reagent)} \\ &\quad \times \text{fw analyte (mg/mmol)} \end{aligned}$$

1. Aspirin is slow to react with NaOH and is therefore determined by back-titration. A 0.5-g sample containing aspirin plus inert matter is analyzed by adding 30 mL of 0.1 M NaOH, a slight excess. The sample is heated to speed the hydrolysis. Then, the excess NaOH is back-titrated with 0.1M HCl. If 12.7 mL HCl is required for the back-titration, how many milligrams of aspirin are contained in the sample?

Problems

1. Describe the preparation of 50 mL of 0.1 M NaOH from the solid reagent.
2. Describe the preparation of 50 mL of 0.5 M HCl from the commercial reagent that has a specific gravity of 1.18 and is 37% (w/w) HCl (36.5 g/mol).
3. An approximate 0.1 M hydrochloric acid solution is prepared from the commercial concentrated hydrochloric acid. It is standardized by titrating 0.1876 g of dried primary standard sodium carbonate:



The titration required 35.86 mL acid. Calculate the molar concentration of the hydrochloric acid.

4. Acetic acid in 2.5-mL vinegar sample is determined by titrated with 0.1 M NaOH solution, requiring 12.7 mL. Calculate the percent of CH₃COOH in the sample.
5. The aspirin in a 0.5-g sample is analyzed by adding 30 mL of 0.1 M NaOH, a slight excess. The sample is heated to speed the hydrolysis. The excess NaOH is back-titrated with 12.7 mL of 0.1 M HCl. Calculate the aspirin amount in the sample in mg.