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Fundamentals of Practical Pharmaceutical Analysis

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Mr. Shaik Nabi Rasool is currently working as an Assistant Professor in the Department of Pharmaceutical Analysis at Acharya & BM Reddy College of Pharmacy, Bangalore. He completed his Master of Pharmacy (Pharmaceutical Analysis) from JNTUK, Kakinada, prioritizing analytical method development and validation, quality control of pharmaceutical formulations, and regulatory aspects of pharmaceutical analysis. He has 2.5 years of academic experience in teaching and research. Mr. Rasool is committed to strengthening practical learning in instrumental analysis and contributes actively to departmental and academic initiatives, staying current with advancements in pharmaceutical sciences. He is passionate about innovation in pharmaceutical analysis, with a growing interest in applying AI to analytical sciences

Mr. Anjan D is an Assistant Professor in the Department of Pharmaceutical Chemistry at Acharya & BM Reddy College of Pharmacy, Bangalore. He holds M.Pharm degree in Pharmaceutical Chemistry and is dedicated to both teaching and research. His key areas of interest include medicinal chemistry, drug design, synthesis of novel therapeutic agents, and impurity profiling. He has presented his research at various national and international conferences and published in reputed journals. Mr. Anjan D actively mentors students and organizes academic events, with current work focused on green chemistry and computational drug discovery.

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Mr. Ashwin Ravichandran is an Assistant Professor at Acharya & BM Reddy College of Pharmacy, Bangalore. He holds a Master's in Pharmaceutics from JSS College of Pharmacy, Ooty, and an Advanced Diploma in Drug Regulatory Affairs from Jamia Hamdard University, New Delhi. With academic and industry experience, he focuses on formulation development, regulatory documentation, and product analysis, emphasizing GLP, SOP compliance, and QbD. He is proficient in analytical instruments, manufacturing equipment, and software tools. Ashwin has received fellowships, grants, and awards, including national hackathon wins, and holds national and international patents and research publications in pharmaceutical innovation.

Preface

This book, *Fundamentals of Practical Pharmaceutical Analysis*, is designed to provide pharmacy students with a clear, concise understanding of essential analytical techniques used in pharmaceutical laboratories. It bridges theoretical concepts with practical application, focusing on accuracy, reproducibility, and regulatory relevance. The content includes important definitions, limit tests for common impurities, preparation and standardization of reagents, and the assay of pharmaceutical compounds using various volumetric techniques. Modern electro-analytical methods such as conductometric and potentiometric titrations are also covered to introduce students to instrumental analysis.

Each experiment is presented with its principle, reaction involved, procedure, and standardization steps, making it easy for learners to follow and perform confidently. This book aims to serve as a valuable guide for pharmacy undergraduates and instructors alike, fostering strong foundational skills in pharmaceutical analysis and promoting good laboratory practices essential for academic and professional success.

Mr. Shaik Nabi Rasool
Mr. Anjan D
Ms. S. Keerthana Madhuri
Dr. R. Aishwarya Reddy
Mr. Ashwin Ravichandran

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Important Terms

Limit Test: A limit test is a quantitative or semi-quantitative procedure used to find trace amounts of impurities that are probably present in a material.

- Basically, limit test for inorganic compound is carried out to determine whether the amount of inorganic impurity present in the pharmaceutical substance is in the limit or exceed its prescribed limit by pharmacopoeias.
- In the limit test the impurity is identified and the presence of impurity is compared with the standard taking the specified amount of impurity.
- The standard substances of limit test contain the maximum amount of impurity which can be allowed in pharmaceutical substance.
- The comparison of sample with standard involves the physical changes like colour, Turbidity or opalescence etc.

Volumetric Analysis: The process whereby the concentration of a reagent is determined by reacting with a known quantity of a second reagent.

Titrant: The substance of known concentration which is used to determine the unknown concentration of substance.

Titrand: The substance whose concentration is not known.

Titration: Titration is the process of adding measured amounts of a standard solution that reacts with the unknown to find the concentration of the unknown ingredient in solution. The stereochemistry of the reaction and the quantity of standard solution required to achieve the so-called end point can then be used to determine the concentration of the unknown.

Indicator: An indicator is an external material added to the titration that, through a visible change, shows that the reaction is complete.

End point: The point at which the reaction between titrant and titrand is completed and the indicator change its colour to indicate the completion of the reaction is called as end point.

Equivalent point: The exact point at which the reaction between titrant and titrand is completed before the colour change of indicator.

Titration error: Little difference between endpoint and equivalent point is called as titration error.

Standard solution: Solution whose strength is known.

Standardisation: Standardisation is a process to find out the exact strength of some solution using standard solution.

Primary standard: A primary standard is a chemical which has certain properties such as

- It should be available with maximum purity.
- It should not absorb moisture (hygroscopic) or CO2 while weighing.
- It should be stable and remain its chemical composition during use.
- It should be nontoxic
- It should have high molecular weight to avoid weighing error.
- It should dissolve with solvent freely

E.g.: Sodium carbonate, oxalic acid, potassium dichromate, potassium hydrogen phthalate etc.

Secondary standard: Reagents that do not meet these requirements are considered secondary standards. A secondary standard's concentration has to be calculated in relation to a primary standard.

Eg: Sodium hydroxide, sodium thiosulphate, disodium edetate, ceric ammonium sulphate etc.

Types of Titrations

I. Based upon the Principle of Reaction

- **1. Acid-Base Neutralisation Titration**
    - Acidimetry
    - Alkalimetry
    - Non-aqueous Titration
- **2. Complexometric Titration**
- **3. Precipitation Titration**

- Mohr's Method
- Volhard Method
- Fajan's Method

- **4. Redox Titration**
  - Permanganometry
  - Dichrometry
  - Cerimetry
  - Titanometry
  - Titration with Iodine
    - Iodometry
    - Iodimetry
  - Titration with KBrO3
  - Titration with 2,4-Dichlorophenol Indophenols

II. Based upon the Method

1. Direct Titration
2. Back Titration
3. Modified / Indirect Titration
4. Replacement Titration

Methods for expressing concentration:

Concentration of solution is the amount of solute dissolved in a known amount of the solvent or solution.

1. Molarity (M): Molarity provides a measure of how concentrated a solution is by indicating the number of moles of a substance that have been dissolved to create one liter of the final solution. This is a very common unit in chemistry as it directly relates to the number of molecules available for a chemical reaction.

2. Normality (N): Normality is a concentration unit that focuses on the reactive potential of a substance within a solution. It is determined by the number of gram equivalents of the solute present in one liter of the solution. This measurement is particularly useful in

the context of acid-base and redox reactions where the number of reactive species is important.

3. Molality (m): Molality defines the concentration based on the mass of the solvent rather than the volume of the solution. It is calculated as the number of moles of a solute dissolved in every 1000 grams (1 kilogram) of the solvent. A key advantage of molality is that it remains constant even if the temperature or pressure of the solution changes, as it is based on mass.

4. Parts Per Million (ppm): When dealing with extremely small amounts of a substance in a solution, parts per million is a convenient way to express concentration. It signifies the ratio of one part of a solute for every one million parts of the total solution. This is often used to describe the presence of trace elements or contaminants.

Expression of Content:

5. Percent (w/w): Also known as weight-by-weight percentage, this method defines concentration by comparing the mass of the substance to the total mass of the mixture. It specifically indicates the mass in grams of a substance contained within 100 grams of the final product.

6. Percent (v/v): Referred to as volume-by-volume percentage, this measurement is used to express the concentration of a substance based on its volume. It represents the number of milliliters of a substance present for every 100 milliliters of the entire solution, and is commonly used when mixing liquids.

Expression of Concentration:

7. Percent (w/v): Weight-by-volume percentage is a common way to express the concentration of a solid dissolved in a liquid. It is defined as the mass of the substance in grams for every 100 milliliters of the final product.

8. Percent (v/w): This less frequent measurement, known as volume-by-weight percentage, specifies the volume of a substance relative to the mass of the final product. It indicates how many milliliters of a substance are present in 100 grams of the mixture.

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Chapter 1: Limit Test for Chloride

1 Introduction

A qualitative method for identifying and managing the amount of chloride impurities in pharmaceutical products is the limit test for chlorides. This test guarantees compliance to pharmacopeial standards since excess chloride ions can compromise the stability and safety of medications. In this experiment, the sample is treated in Nessler's cylinders with a 5% silver nitrate solution and diluted nitric acid, which causes a white turbidity to appear if chlorides are present. A reference chloride solution (0.05845% w/v) produced in the same way is used to visually compare the degree of turbidity. For exact measurements and mixing, tools like pipettes and glass rods are utilised. The sample satisfies the chloride limit and is considered to be of acceptable purity if its turbidity is less than or equal to the standard.

2. Principle

In the presence of diluted nitric acid, soluble chloride and silver nitrate reagent combine to form insoluble silver chloride, which is the basis for the limit test for chloride. The solution is turbid due to the silver chloride that is produced during the reaction. The quantity of chlorides (an impurity) in the sample determines how much opalescence is created. The opalescence of the sample is compared to that of a reference solution that is prepared similarly and has the required quantity of chlorides.

If the sample's opalescence is less than the standard, it passes the limit test and is considered pure or standard. The sample fails the test and the material is deemed impure

or substandard if the amount of opalescence in the sample exceeds the standard.

$$\textbf{Test:}\quad Cl^- + AgNO_3 \xrightarrow{\text{dil. } HNO_3} AgCl + NO_3$$

$$\textbf{Standard:}\quad NaCl + AgNO_3 \xrightarrow{\text{dil. } HNO_3} AgCl + NaNO_3$$

2.1 Discussion

1. Nitric acid is used because silver chloride precipitate formed is insoluble in inpresence of it.
2. Other impurities like Carbonates, Bicarbonates, Phosphate, and Hydroxide etc alsoreact with silver nitrate to give their respective precipitates. These precipitates dissolves in water in presence of dilute nitric acid. Thereby nitric acid prevents interference of other impurities.
3. The dilute nitric acid also increases the sensitivity of the reaction by common ion effect. The nitrate ion of nitric acid and silver nitrate is common so the formation ofprecipitates of silver chloride from silver nitrate will produce fast.
4. The reagent, which is used in experiment, should be free from chlorides except standard NaCl solution.
5. Chloride standard solution is prepared by diluting 5 ml of 0.0824 % w/v solution of sodium chloride solution to 100 ml with distilled water.
6. Limit test for chlorides IP 1985 differs from that of IP 1996 only in the preparation of chloride standard solution. The conc. of standard chloride solution is 0.05845 % w/v. Add 1 ml for the preparation of standard opalescence.

3 Procedure

Assign the labels "test" and "standard" to two 50 ml Nessler's cylinders.

Test opalescence	Standard opalescence
1. Transfer a predetermined amount of the material to a Nessler's cylinder after dissolving it in water. 2. In 10 millilitres of distilled water, dissolve. 3. Include 10 millilitres of diluted nitric acid solution. 4. Use distilled water to get the volume up to 50 ml. 5. Pour in 1 millilitre of 0.1 M $AgNO_3$ solution. 6. Using a glass rod, thoroughly mix and set away for five minutes.	1. Fill a standard Nessler's cylinder with 1 millilitre of standard chloride solution (25 parts per million of Cl). 2. Pour in 10 millilitres of purified water. 3. Include 10 millilitres of diluted nitric acid solution. 4. Use distilled water to get the volume up to 50 ml. 5. Include 1 millilitre of 0.1 M $AgNO_3$ solution. 6. Using a glass rod, thoroughly mix and set away for five minutes.

3.1 Modified limit test for chloride:

Test opalescence	Standard opalescence
1. Fill a Nessler's cylinder with 40 millilitres of the sample solution. 2. Include 10 millilitres of diluted nitric acid solution. 3. Use distilled water to get the volume up to 50 ml. 4. Pour in one millilitre of 0.1 M $AgNO_3$ solution. 5. Use a glass rod to thoroughly mix, then set aside for five minutes.	1. Fill a standard Nessler's cylinder with 1 millilitre of standard chloride solution (25 parts per million of Cl). 2. Pour in 10 millilitres of purified water. 3. Include 10 millilitres of diluted nitric acid solution. 4. Use distilled water to get the volume up to 50 ml. 5. Include one millilitre of 0.1 M $AgNO_3$ solution. 6. Using a glass rod, thoroughly mix and set away for five minutes.

4 Observation

The sample exhibits a degree of opalescence that is either greater or lesser than the established control.

5 Result

The provided sample either passes or fails the chloride limit test.

Reference

1. Indian Pharmacopoeia Commission. Indian Pharmacopoeia 2018. Vol. I. Ghaziabad: IPC; 2018.
2. Beckett AH, Stenlake JB. *Practical Pharmaceutical Chemistry*. Vol. 1. 4th ed. New Delhi: CBS Publishers; 2005.

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Chapter 2: Limit test for Sulphate

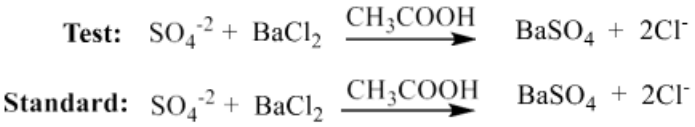
1 Introduction

A qualitative method for determining whether sulphate ions are present in pharmaceutical substances and confirming that their concentration is within permissible pharmacopeial limits is the limit test for sulphates. The safety and quality of drugs can be impacted by high sulphate impurity levels. If sulphates are present, a white precipitate of barium sulphate will develop as a result of treating the test solution with diluted hydrochloric acid (HCl) and barium chloride solution. To visually compare the sample's turbidity with that of a reference solution with a known concentration of potassium sulphate, the test is conducted in Nessler's cylinders. For accurate handling and mixing, glass rods, pipettes, distilled water, and barium sulphate reagent are utilised. If the turbidity of the sample does not exceed the standard, the test verifies conformity.

2 Principle

The interaction between soluble sulphate ions and barium chloride ($BaCl_2$) in the presence of acid (acetic acid or diluted HCl) provides the basis for the sulphate limit test. The reaction produces barium sulphate, which is insoluble in acidic media. The test solution becomes murky due to the formation of barium sulphate. The quantity of sulphate impurity in the sample determines how turbid it is. A standard turbidity/opalescence caused by a known quantity of sulphate ions is used for comparison. The sample will pass the test if the test solution's turbidity or opalescence is below the acceptable level, or the opposite is true.

Both test and standard Nessler's cylinders should be viewed transversely against a black background.



Composition of barium sulphate reagent:

Combine 20 millilitres of sulphate-free alcohol, 55 millilitres of water, and 15 millilitres of 0.5 M barium chloride. Add 5 millilitres of a potassium sulphate solution that is 0.0181 percent w/v. Mix with water to dilute to 100 ml. The reagent barium sulphate needs to be made fresh.

In later iterations of the Indian Pharmacopoeia, the limit test for sulphate has been altered. Each component of the barium sulphate reagent is employed independently in place of the reagent itself. The following reagents were used: 0.15 ml of 5M acetic acid, 1.5 ml of ethanolic sulphate standard solution (10 ppm), and 1 ml of 25% barium chloride solution. 15 millilitres of sulphate standard solution (10 ppm) are used as the standard. When barium sulphate precipitates, potassium sulphate serves as a seeding agent. Alcohol helps to create a more uniform turbidity and prevents over saturation.

3 Procedure

Assign the labels "test" and "standard" to two 50 ml Nessler's cylinders.

Test	Standard
1. Fill a Nessler cylinder with 1 millilitre of a 25% w/v barium chloride solution. 2. Stir in 1.5 ml of the standard solution for ethanolic sulphate, then let it stand for a minute. 3. Use 15 millilitres of distilled water to dissolve the stated amount of material. then move to the Nessler cylinder. 4. Pour in 0.15 millilitres of 5M acetic acid. 5. Use distilled water to get the volume up to 50 ml. 6. Use a glass rod to stir right away, then let it stand for five minutes.	1. Fill a Nessler cylinder with 1 millilitre of a 25% w/v barium chloride solution. 2. Stir in 1.5 ml of the standard solution for ethanolic sulphate, then let it stand for a minute. 3. Fill the Nessler cylinder with 15 millilitres of the sulphate standard solution. (10 ppm SO4) 4. Pour in 0.15 millilitres of 5M acetic acid. 5. Use distilled water to get the volume up to 50 ml. 6. Use a glass rod to stir right away, then let it stand for five minutes.

3.1 Modified limit test for sulphate:

Test	Standard
1. Place the sample solution inside the Nessler cylinder. 2. Fill a Nessler cylinder with 1 millilitre of a 25% w/v barium chloride solution. 3. Mix in 1.5 ml of the standard solution of ethanolic sulphate (10 ppm SO4), then let it stand for a minute. 4. Pour in 0.15 millilitres of 5M acetic acid. 5. Use distilled water to get the volume up to 50 ml. 6. Use a glass rod to stir right away, then let it stand for five minutes.	1. Fill a Nessler cylinder with 1 millilitre of a 25% w/v barium chloride solution. 2. Pour in 1.5 ml of the standard solution of ethanolic sulphate (10 ppm SO4), stir, and let stand for 1 minute. 3. Fill the Nessler cylinder with 15 millilitres of the sulphate standard solution. 4. Pour in 0.15 millilitres of 5M acetic acid. 5. Use distilled water to get the volume up to 50 ml. 6. Use a glass rod to stir right away, then let it stand for five minutes.

4 Observation

The sample exhibits a degree of opalescence that is either greater or lesser than the established control.

5 Result

The provided sample either passes or fails the sulphate limit test.

Reference

1. Indian Pharmacopoeia Commission. Indian Pharmacopoeia 2018. Vol. I. Ghaziabad: IPC; 2018.
2. Beckett AH, Stenlake JB. Practical Pharmaceutical Chemistry. Vol. 1. 4th ed. New Delhi: CBS Publishers; 2005.

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Chapter 3: Limit test for Iron

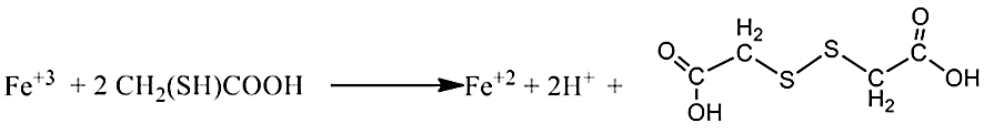
1 Introduction

A qualitative method for identifying and managing trace levels of iron in pharmaceutical materials is the limit test for iron. The stability of products can be impacted by excess iron, which can catalyse breakdown events. In order to decrease ferric ions to ferrous form and produce a purple colouration in an alkaline media, the sample is treated with iron-free citric acid (20% w/v) and thioglycolic acid in this test. Red litmus paper is used to confirm that the solution has turned alkaline after ammonia solution has been added. A solution of ferric ammonium sulphate at 0.173 g/1000 mL concentration serves as a reference to evaluate the color intensity of the test solution through Nessler's cylinders. The sample meets the required iron threshold when its color does not exceed the standard intensity level.

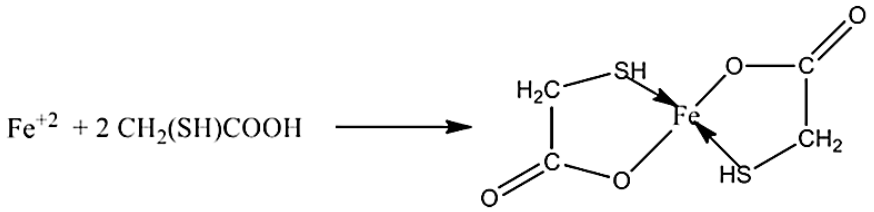
2 Principle

This limit test is based on the reaction of iron (in ferrous state) with thioglycolic acid resulting in the formation of ferrous thioglycolate complex. Ferrous thioglycolate is coloured complex that ranges from pale pink to deep reddish purple depending on the concentration of complex and in turn concentration of iron. Because thioglycolic acid is a potent reducing agent that converts ferric ions to ferrous ions, the oxidation state of iron is irrelevant throughout the process.

Since reactions only occur in alkaline media, ammonia is added to the solution to make it alkaline. In alkaline media, ferrous thioglycolate gives pink colour but remain colour less in acidic or neutral media. But in alkaline pH, iron reacts with water and forms iron oxide precipitate leading to false results. Therefore, citric acid is used to prevent the precipitation of iron in the form of hydroxides.



Carboxymethyldisulfanyl-acetic acid



Ferrous thioglycolate complex

Here the colour intensity of the test and standard will be compared by viewing vertically against a white background. Ferrous thioglycolate complex is unstable in air. The pink colour fades in air due to oxidation.

3 Procedure

Assign the labels "test" and "standard" to two 50 ml Nessler's cylinders.

Test	Standard
1. Use distilled water to dissolve a certain amount of material. 2. Include 2 millilitres of a 20% w/v iron-free citric acid solution. 3. Include one millilitre of thioglycolic acid. 4. Red litmus turns blue when ammonia solution is added to make the solution alkaline. 5. Use distilled water to make up to 50 ml. 6. Use a glass rod to thoroughly mix the solution, then let it standardise for five minutes.	1. Measure two millilitres of standard iron solution (20 ppm Fe). 2. Include 2 millilitres of a 20% w/v iron-free citric acid solution. 3. Include one millilitre of thioglycolic acid. 4. Red litmus turns blue when ammonia solution is added to make the solution alkaline. 5. Use distilled water to make up to 50 ml. 6. Use a glass rod to thoroughly mix the solution, then let it stand for five minutes.

4 Observation

The sample exhibits a degree of opalescence that is either greater or lesser than the established control.

5 Result

The provided sample either passes or fails the iron limit test.

Reference

1. Indian Pharmacopoeia Commission. Indian Pharmacopoeia 2018. Vol. I. Ghaziabad: IPC; 2018.
2. Beckett AH, Stenlake JB. Practical Pharmaceutical Chemistry. Vol. 1. 4th ed. New Delhi: CBS Publishers; 2005.

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Chapter 4: Limit Test for Arsenic

1 Introduction

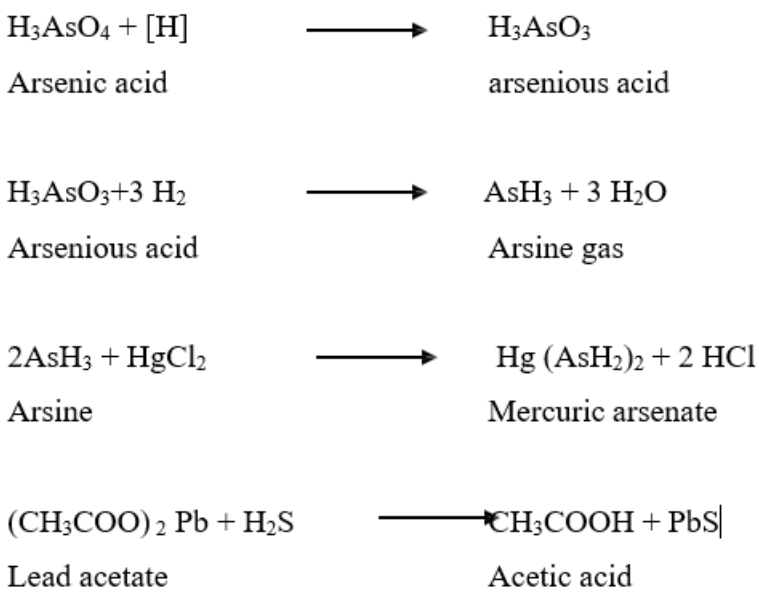
Since even minute concentrations of arsenic are extremely hazardous and poisonous, the limit test for arsenic is a qualitative method used to find trace amounts of arsenic in medicinal compounds. The basis of the test is the transformation of arsenic compounds into arsine gas (AsH_3), which produces a yellow to brown stain when it interacts with mercuric chloride paper. To produce arsine gas in the event that arsenic is present, the sample is treated with stannated hydrochloric acid (HCl), potassium iodide, and zinc granules using a Gutzeit device. An arsenic trioxide-based standard is used to compare the stain intensity of the test paper. The sample satisfies the arsenic limit criteria if the stain from the test solution is not more intense than the standard.

2 Principle

Limit test for arsenic is based on the conversion of arsenic to arsine gas. Arsenic that is present in arsenious state is reduced to arsine gas. When this gas is passed over mercuric chloride paper it produces a stain which ranges in colour from yellow to brown.

The amount of arsenic in the sample will determine how deeply the yellow stain appears on mercuric chloride paper. The standard and the colour generated by the test are contrasted. The test or sample fails the limit test if the colour intensity exceeds the standard, and vice versa.

Arsenic will exhibit in two forms one is trivalent (arsenite) and pentavalent (arsenate) form. Reduction of arsenic to arsine will be achieved by zinc, acid, stannous chloride, KI as these three reagents acts as reducing agents.



Arsenic acid is reduced to arsenious acid and then reduced to arsine gas. Arsine gas that is formed in the apparatus rises through the tube and reacts with mercuric chloride/mercuric bromide to give mercuric arsenate which is in yellow colour. Yellow colour intensity depends on the concentration of arsenic present in given sample and is compared with the standard.

During the procedure sulphur impurity can interfere with the limit test. The impurity sulphur reacts with hydrogen to form hydrogen sulphide and this gas also travels to mercuric chloride paper and leaves yellow stain. This yellow stain interferes with yellow stain of mercuric arsenate resulting in false positive. To prevent this, lead acetate is kept in the tube just below mercuric chloride paper. Hydrogen sulphide formed during the reaction reacts with lead acetate to form lead sulphide and thereby it does not reach mercuric chloride paper.

Limit test is carried out at 40 °C so that evolution of hydrogen gas is optimum. If the temperature is increased to 60 °C, evolution of hydrogen gas will be faster and it carries arsine gas to mercuric chloride paper very fast and the spot will be like brown point that makes comparison difficult. If the test is done at room temperature, hydrogen release will be very slow and the spot formed on the mercuric chloride paper will diffuse and makes comparison difficult. So, 40 °C is considered as optimum for limit test for arsenic.

Standard stain:

It is produced by 1 ml dilute arsenic trioxide solution having 0.00001 g concentration.

Description of Apparatus:

The device is made out of a 100 ml wide-mouth glass container. The Stopper assembly is prepared by choosing a rubber stopper with a central hole through which a glass tube of about central hole about 8 mm external diameter, and exactly 6.5 mm internal diameter. The tube is of 200 mm length, passing through a stopper. The upper end is fire polished, flat and flush with the top of a rubber bunk. The lower end is slightly drawn to a narrow opening of about 1 mm diameter and has also a side hole of about 2 mm diameter, the drawn end together with the hole should be below the lower edge of the stopper but above the final liquid level of about 70 ml. The drawn end helps in allowing condensed vapours to drop into the bottle without blocking the side hole through which vapours can enter and rise in the rube. Inside this tube, about 25 mm below the top end, cotton wool dipped in lead acetate solution is inserted lightly. The fairly dry cotton wool becomes sufficiently moist due to rise in moisture vapour during the experiment. All the vapours must pass through this cotton wool so that any hydrogen sulphide liberated during latter reaction would be trapped and reacts with the lead acetate, forming brown to black lead sulphide. On the top of the rubber bung a small strip of filter paper dipped in mercuric chloride solution and dried is placed and secured in position by placing another inverted rubber bunk, the upper inverted rubber bunk also has a hole of 6.5 mm diameter. Exactly corresponding in position with the glass tube fitted in the lower bung. The two rubber bungs are held in position by means of spring clips. The purpose of this assembly is to guide the rising vapor from the wide mouth bottle through the tube, through the cotton wool and through the mercuric chloride paper in that order.

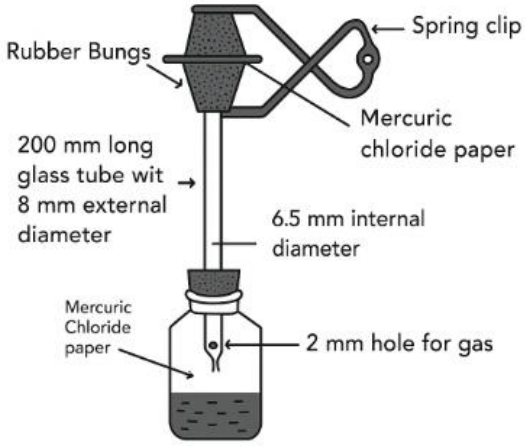


Fig. 1 Gutzeit apparatus

3 Procedure

Take two Gutzeit devices and mark one as standard and the other as test.

Test	Standard
1. Dissolve a specified quantity of 2. sample in 50 ml distilled water. 3. Add 10 ml of stannated HCl 4. Add 1 g of potassium iodide and10 g of zinc granules 5. Place the apparatus immediately in position. 6. Allow the reaction to stand for 45 minutes.	1. To 1 ml of std. arsenic solution add 2. 50 ml distilled water. 3. Add 10 ml of stannated HCl 4. Add 1 g of potassium iodide and 10 g of zinc granules 5. Place the apparatus immediately inposition. 6. Allow the reaction to stand for 45 minutes.

4 Observation

The sample exhibits a degree of opalescence that is either greater or lesser than the established control.

5 Result

The provided sample either passes or fails the arsenic limit test.

Reference

1. Indian Pharmacopoeia Commission. Indian Pharmacopoeia 2018. Vol. I. Ghaziabad: IPC; 2018.
2. Beckett AH, Stenlake JB. Practical Pharmaceutical Chemistry. Vol. 1. 4th ed. New Delhi: CBS Publishers; 2005.

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Chapter 5: Preparation and standardization of 0.1N Sodium hydroxide

1 Introduction

It is not appropriate for direct standardisation by weighing because sodium hydroxide (NaOH) is a strong base that absorbs moisture and carbon dioxide from the air. As a result, it is roughly prepared before being standardised using a main standard, such oxalic acid. This experiment uses phenolphthalein as an indicator, which becomes pink at the endpoint, to titrate a 0.1N NaOH solution against a known quantity of oxalic acid. A burette, pipette, conical flask, and beaker are used in the process. For precise future analytical applications, the amount of NaOH needed for neutralisation helps in determining its exact normality.

2 Principle

Sodium hydroxide (NaOH) is a strong base but not suitable as a primary standard because it is hygroscopic and absorbs carbon dioxide from the air. Therefore, it is standardized against a primary standard such as oxalic acid, which is stable, pure, and accurately weighed. During titration, phenolphthalein is used as the indicator. It is colourless in acidic medium and turns pink in basic medium. The appearance of a pale pink colour marks the endpoint, indicating complete neutralization of oxalic acid by sodium hydroxide, confirming the accurate concentration of the NaOH solution.

$$(COOH)_2 + 2\ NaOH \longrightarrow (COONa)_2 + 2H_2O$$

3 Procedure

Preparation of 0.1N sodium hydroxide:

Dissolve about 4 g of sodium hydroxide in specified amount of distilled water in a 1000 ml volumetric flask and make up the volume to the mark using distilled water.

Preparation of 0.1N oxalic acid:

In a 100 ml volumetric flask, precisely weigh 0.63 g of oxalic acid, then dissolve it in water. Use distilled water to adjust the volume to the proper level.

Standardization of 0.1N sodium hydroxide:

Pipette and add a few drops of phenolphthalein as an indicator to 10 millilitres of 0.1 N oxalic acid solution in a conical flask, then titrate against a standard 0.1 N NaOH solution. The appearance of a pale, permanent pink colour is the end result.

4 Observation

Conical flask: 10ml of Oxalic acid

Burette: NaOH

Indicator: Phenolphthalein

End point: Possessing of pale pink colour

Sl. No	Burette reading in ml		Volume of titrant
	Initial	Final	
1			
2			
3			

4.1 Calculation:

Normality of sodium hydroxide

$$N_1V_1 = N_2 V_2$$

V_1= Volume of oxalic acid

N_1= Normality of oxalic acid

V_2= Volume of sodium hydroxide

N_2= Normality of sodium hydroxide

$$N_2 = V_1 \times N_1 / V_2$$

5 Result

The normality of sodium hydroxide is found to be ……. N

Reference

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Chapter 6: Preparation and standardization of 0.1N Sulphuric acid

1 Introduction

Due to its highly concentrated, sulphuric acid (H_2SO_4), a powerful diprotic acid that is frequently employed in titrations, needs to be diluted and standardised before use. Since dilution alone cannot guarantee precise concentration, a main standard such as sodium carbonate (Na_2CO_3) is used to standardise it. About 0.1N sulphuric acid is titrated in this experiment against a known amount of sodium carbonate solution. A burette, pipette, conical flask, and beaker are used for the titration. The volume of acid needed aids in determining the endpoint's precise normalcy for analytical usage, and it is usually determined using an appropriate indicator.

2 Principle

The reaction between sodium carbonate and sulphuric acid is an example of an acid–base neutralization reaction. Sodium carbonate, a basic salt, reacts with sulphuric acid, a strong acid, to form sodium sulphate, water, and carbon dioxide gas. The balanced chemical equation is:

$$Na_2CO_3 + H_2SO_4 \rightarrow Na_2SO_4 + H_2O + CO_2\uparrow$$

This reaction is used in standardization procedures because sodium carbonate is a primary standard – it is stable, pure, and non-hygroscopic. The evolution of carbon dioxide confirms the reaction's progress, and the endpoint is usually detected using methyl orange as the indicator, changing from yellow to orange-red.

3 Procedure

Caution: remember the safety guideline while diluting acid solutions: always add acid to water, never pour water into acid. A substance's equivalent weight per litre is present in normal (N) solutions. The molecular weight is divided by the quantity of hydrogen ions to determine the equivalent weight of acids. Consequently, the molecular weight divided by two equals the equivalent weight of sulphuric acid.

- In a 1L solution, 1 N sulphuric acid contains 98/2 = 49-gram acid.
- One litre of 0.1 N sulphuric acid solution contains 49/10 = 4.9 gram of acid.

Convert grams of acid to millilitres by using the specific gravity (grams divided by the specific gravity = millilitres). The specific gravity of the conc. sulphuric acid is 1.84 therefore 4.9/1.84 = 2.66 mL sulphuric acid per litre solution gives 0.1 N concentrations.

Preparation of 0.1N Sulphuric acid:

2.66 mL of concentrated sulphuric acid (H_2SO_4, sp. Gr-1.84) should be measured into a graduated cylinder and gradually added to 400 mL of water in a 600 mL beaker to create a 0.1 N solution. Pour water into the beaker to rinse the cylinder. After mixing the acid-water combination and letting it cool, pour it into a 1-liter volumetric flask. Mix well, dilute with water to the appropriate level, and store in a glass container that is firmly sealed.

Preparation of 0.1N sodium carbonate:

Transfer 0.22 g of the dry Na_2CO_3 to a 500 mL conical flask after precisely weighing it. After adding 50 millilitres of water and swirling to dissolve the Na_2CO_3, add two drops of a 0.1% methyl red in alcohol solution. Titrate the H_2SO_4 solution until a crimson tint appears, then gently boil the mixture to prevent colour loss until the colour is released. After cooling to room temperature, proceed with the titration by alternating the addition of H_2SO_4 solution, boiling, and cooling until a slight red colour appears, which does not disappear with additional heating.

Standardization of 0.1N Sulphuric acid:

Pipette and add few drops of methyl red in alcohol are added to 10 millilitres of 0.1 N Na_2CO_3 solution in a conical flask, and the mixture is titrated against a standard 0.1 N sulphuric acid solution. The look of a faint, persistent pink colour is the ultimate result.

4 Observation

Conical flask: 10ml of 0.1 N Na_2CO_3

Burette: H_2SO_4

Indicator: Methyl Red

End point: Possessing of pale pink colour

Sl. No	Burette reading in ml		Volume of titrant
	Initial	Final	
1			
2			
3			

4.1 Calculation:

Calculate the normality of the sulphuric acid solution as follows

$$\mathbf{N_1V_1 = N_2\ V_2}$$

V_1= Volume of Sodium carbonate

N_1= Normality of Sodium carbonate

V_2= Volume of Sulphuric acid

N_2= Normality of Sulphuric acid

$$\mathbf{N_2 = V_1\ x\ N_1\ /\ V_2}$$

5 Result

The normality of sulphuric acid is found to be ……. N

Reference

1. Harris DC. *Quantitative Chemical Analysis*. 10th ed. New York: W. H. Freeman and Company; 2022.
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Chapter 7: Preparation and standardization of 0.1N Sodium thiosulphate

1 Introduction

Although sodium thiosulphate is frequently used in iodometric titrations, it must be newly produced and standardised before to use since it is unstable over extended periods of time. Potassium dichromate is used as the major standard in this experiment to standardise a sodium thiosulphate solution of around 0.1N. Potassium dichromate oxidises potassium iodide in an acidic media, releasing iodine, which is subsequently titrated using the thiosulphate solution. Usually employed as an indication, starch and iodine combine to generate a blue complex that vanishes at the terminus. The procedure employs distilled water and sodium carbonate to stabilise the solution, along with common titration tools such a burette, pipette, conical flask, and beaker.

2 Principle

This titration is a redox process where potassium dichromate reacts with potassium iodide and concentrated hydrochloric acid to liberate iodine. The reaction is:

$$Cr_2O_7^{2-} + 14H^+ + 6I^- \rightarrow 2Cr^{3+} + 3I_2 + 7H_2O$$

The liberated iodine (I_2) is titrated with sodium thiosulphate:

$$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$$

Starch is used as an indicator, forming a blue complex with iodine. As sodium thiosulphate reduces iodine to iodide, the blue color disappears. The endpoint is observed when the blue color changes to colorless, indicating complete reaction and accurate titration.

3 Procedure

Preparation of 0.1N sodium thiosulphate:

Dissolve about 25 g of sodium thiosulphate in 100 ml of water and add 0.2 gm of sodium carbonate in distilled water and dilute to 1000 ml with water.

Preparation of 0.1N potassium dichromate:

Accurately weigh out 4.9 grammes of potassium dichromate, then dissolve it in enough water to make 1000 millilitres.

Standardization of 0.1N sodium thiosulphate:

Pipette 10 ml of potassium dichromate solution in an iodine flask. Add 0.5 gm of potassium iodide and 1 ml of concentrated hydrochloric acid and stand for 10 minutes in a dark place. After this titrate the liberated iodine against the standard solution of sodium thiosulphate until a pale-yellow colour is obtained. Add starch as indicator and continue the titration until blue colour disappears and light green colour appears.

Each ml of 0.1 M sodium thiosulphate is equivalent to 0.049 gm of $K_2Cr_2O_7$

4 Observation

Conical flask: 10 ml of $K_2Cr_2O_7$ + 0.5 gm of KI + 1 ml Conc. HCl

Burette: $Na_2S_2O_3$

Indicator: Starch Solution

End point: Possessing of light green colour

Sl. No	Burette reading in ml		Volume of titrant
	Initial	Final	
1			
2			
3			

4.1 Calculation:

Normality of sodium thiosulphate = Weight of $K_2Cr_2O_7$ X Exact Normality/Volume of $Na_2S_2O_3$ X IP factor of $K_2Cr_2O_7$

5 Result

The normality of Sodium thiosulphate is found to be ……. N

Reference

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Chapter 8: Preparation and standardization of 0.1N Potassium permanganate

1 Introduction

In redox titrations, potassium permanganate ($KMnO_4$), a strong oxidising agent, is frequently utilised. However, because it is unstable and has to be standardised before usage, it is not a primary standard. Oxalic acid is used in this experiment to standardise around 0.1N $KMnO_4$ in the presence of diluted sulphuric acid. The oxidation is started and finished by heating the reaction since it moves slowly at ambient temperature. Burette, pipette, volumetric flask, funnel, beaker, and conical flask are used in the standardisation process. The endpoint is indicated by the emergence of a persistent pink colour, which makes it possible to precisely determine the $KMnO_4$ solution's normalcy.

2 Principle

This is a redox titration in which potassium permanganate ($KMnO_4$), a strong oxidizing agent, reacts with dilute sulphuric acid to liberate nascent oxygen:

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

The nascent oxygen oxidizes oxalic acid ($C_2H_2O_4$) to carbon dioxide and water:

$$2KMnO_4 + 5C_2H_2O_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2$$

The reaction occurs in warm acidic medium (around 70°C). Oxalic acid, a primary standard, is used to standardize potassium permanganate. $KMnO_4$ acts as a self-indicator, changing from purple to colourless at the endpoint.

3 Procedure

Preparation of 0.1N potassium permanganate:

In a 100 ml volumetric flask, dissolve approximately 0.32 gramme of KMnO4 in a small amount of distilled water, then top up the capacity with distilled water. After an hour of heating in a water bath, let it stand for two days. Use glass wool as a filter.

Standardization of 0.1N potassium permanganate:

Weigh accurately about 0.63 gm of oxalic acid and dissolved in 1000 ml of water and make up the volume up to the mark in a 100 ml volumetric flask. Pipette 10 ml of 0.1 N oxalic acid solution into a conical flask. Add 5ml of Conc. H_2SO_4 and worm the solution up to 60-70° C. In worm condition titrate with standard solution of $KMnO_4$ (0.1N). The final result is a persistently light pink. To obtain the concordant results, repeat the titration.

4 Observation

Conical flask: 10 ml Oxalic acid + 5 ml Sulphuric acid

Burette: Potassium permanganate

Indicator: Self-indicator ($KMnO_4$)

End point: Possessing of pale pink colour

Sl. No	Burette reading in ml		Volume of titrant
	Initial	Final	
1			
2			
3			

4.1 Calculation:

Normality of potassium permanganate

$$N_1V_1 = N_2 V_2$$

V_1= Volume of oxalic acid

N_1= Normality of oxalic acid

V_2= Volume of potassium permanganate

N_2= Normality of potassium permanganate

$$N_2 = V_1 \times N_1 / V_2$$

5 Result

The normality of potassium permanganate is found to be ……. N

Reference

1. Harris DC. *Quantitative Chemical Analysis*. 10th ed. New York: W. H. Freeman and Company; 2022.
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Chapter 9: Preparation and standardization of 0.1N Ceric ammonium sulphate

1 Introduction

A strong oxidising agent used in redox titrations, especially cerimetry, is ceric ammonium sulphate. Because of its sensitivity to air and light, it needs to be standardised before use. In this experiment, ferrous ammonium sulphate is used as the primary standard in the presence of diluted sulphuric acid to standardise a solution of around 0.1N ceric ammonium sulphate. 1,10-phenanthroline, an indicator that produces a red complex with Fe2+, is used to track the redox process. The end point is identified by the red colour absence. Standard titration tools such as a burette, pipette, conical flask, beaker, and distilled water are used in the process.

2 Principle

This is a redox titration involving ceric ammonium sulphate (Ce^{4+}), a strong oxidizing agent, and ferrous ammonium sulphate (Fe^{2+}), a reducing agent. In acidic medium, Ce^{4+} oxidizes Fe^{2+} to Fe^{3+}, while itself getting reduced to Ce^{3+}:

$$\mathbf{Ce^{4+} + Fe^{2+} \rightarrow Ce^{3+} + Fe^{3+}}$$

The indicator used is 1,10-phenanthroline, which forms an orange-red complex with Fe^{2+}. During titration, as Fe^{2+} is oxidized, the color fades. The endpoint is indicated by the disappearance of the orange-red color, signaling the complete oxidation of Fe^{2+} to Fe^{3+}. Ceric ammonium sulphate is commonly used to assay iron-containing compounds in this titration.

3 Procedure

Preparation of 0.1N ceric ammonium sulphate:

Dissolve about 65 gm of ceric ammonium sulphate with aid of gentle heat in a mixture of 30 ml of sulphuric acid and 500 ml of water. Cool, filter the solution if turbid and dilute with water to 1000 ml.

Preparation of ferrous ammonium sulphate:

Dissolve about 40 gm of ferrous ammonium sulphate in a previously cooled mixture of 40 ml of sulphuric acid and 200 ml of water, dilute with sufficiently freshly boiled and cooled water to produce 1000 ml.

Standardization of 0.1 N ceric ammonium sulphate:

Measure accurately about 10 ml of the solution into a flask, add 10 ml of dil. H2SO4 and 2 drops of 1,10 phenanthroline as indicator and titrate with standard solution of ceric ammonium sulphate (0.1N) until red colour is changed to greenish yellow. Each ml of 0.1 N ceric ammonium sulphate is equivalent to 0.03921 gm of Fe $(NH_4)_2(SO_4)_2.6H_2O$.

4 Observation

Conical flask: 10 ml Ferrous ammonium sulphate + 10 ml dil. H_2SO_4

Burette: Ceric ammonium sulphate

Indicator: 1,10 phenanthroline

End point: Colour changes from red to green

Sl. No	Burette reading in ml		Volume of titrant
	Initial	Final	
1			
2			
3			

4.1 Calculation:

Normality = Weight of ferrous ammonium sulphate x Exact Normality / Volume of ceric ammonium sulphate x IP factor of FAS

5 Result

The normality of ceric ammonium sulphate is found to be ……. N

Reference

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Chapter 10: Assay of Ammonium chloride

1 Introduction

Ammonium chloride is often used in industry and pharmaceuticals, and its purity needs to be ascertained to guarantee quality and standard compliance. By releasing ammonia and titrating it with a standard oxalic acid solution, this experiment indirectly estimates the % purity of ammonium chloride in a given sample. Phenolphthalein, an indicator that becomes pink at the endpoint, and sodium hydroxide, which releases ammonia from the salt, are used in the titration process. A burette, pipette, conical flask, and beaker are among the standard titration tools used. The amount of oxalic acid used aids in determining the sample's purity percentage.

2 Principle

Acid-base indirect titration is the concept used in the ammonium chloride assay Here, a diluted formaldehyde solution is used to treat ammonium chloride. Formaldehyde converts ammonium chloride to hexamine (hexamethylene tetra amine) and liberated equivalent amount of hydrochloric acid. The liberated hydrochloric acid is titrated with standard sodium hydroxide solution using phenolphthalein as indicator.

$$4NH_4Cl + 6HCHO \rightarrow (CH_2)_6N_4 + 4HCl + 6H_2O$$

$$HCl + NaOH \rightarrow NaCl + H_2O$$

Sodium hydroxide is a secondary standard and is standardized by using a primary standard oxalic acid.

NOTE: The formaldehyde solution should be previously neutralized with 0.1N NaOH by adding phenolphthalein indicator because on auto oxidation formaldehyde liberates formic acid which will interfere the titration and gives the error in burette reading.

3 Procedure

Preparation of 0.1N sodium hydroxide:

Dissolve about 4 g of sodium hydroxide in specified amount of distilled water in a 1000 ml volumetric flask and make up the volume to the mark using distilled water.

Preparation of 0.1N oxalic acid:

In a 100 ml volumetric flask, precisely weigh 0.63 g of oxalic acid, then dissolve it in water. Use distilled water to adjust the volume to the proper level.

Standardization of 0.1N sodium hydroxide:

Pipette and add a few drops of phenolphthalein as an indicator to 10 millilitres of 0.1 N oxalic acid solution in a conical flask, then titrate against a standard 0.1 N NaOH solution. The appearance of a pale, permanent pink colour is the end result.

Assay of ammonium chloride

Weigh precisely 0.1 gram of NH_4Cl, then transfer it to a conical flask and mix it with 20 millilitres of water and 5 millilitres of formaldehyde (which has been neutralised with phenolphthalein). Let it stand for ten minutes. Next, use phenolphthalein as an indicator to titrate with a standard solution of NaOH (0.1N). The look of a light pink hue is the ultimate result.

Each ml of 0.1N NaOH is equivalent to 0.005349 gm of NH_4Cl.

4 Observation for Standardization

Conical flask: 10ml of Oxalic acid

Burette: NaOH

Indicator: Phenolphthalein

End point: Possessing of pale pink colour

Sl. No	Burette reading in ml		Volume of titrant
	Initial	Final	
1			
2			
3			

4.1 Calculation:

Normality of sodium hydroxide

$$N_1V_1 = N_2 V_2$$

V_1= Volume of oxalic acid

N_1= Normality of oxalic acid

V_2= Volume of sodium hydroxide

N_2= Normality of sodium hydroxide

$$\text{Normality } (N_2) = V_1 \times N_1/V_2$$

5 Observation for Assay

Conical flask: 0.1 gm of NH_4Cl + 20 ml of water + 5.0 ml of formaldehyde

Burette: 0.1N NaOH

Indicator: Phenolphthalein

End point: Appearance of pale pink colour

Sl. No	Burette reading in ml		Volume of titrant
	Initial	Final	
1			
2			

5.1 Calculation:

$$\% \text{ Purity} = \frac{\text{Titre value} \times \text{IP factor of } NH_4Cl \times \text{Normality of NaOH}}{\text{Weight of sample} \times \text{exact Normality} \times 100}$$

6 Result

The percentage purity of NH4Cl is found to be ……. %

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Chapter 11: Assay of Ferrous sulphate

1 Introduction

The purity of ferrous sulphate is crucial for quality control since it is used extensively in industry and medicines. Using ceric ammonium sulphate as the oxidising agent, redox titration is used in this experiment to assess the % purity of a ferrous sulphate sample. To keep the medium acidic, the titration is performed with diluted sulphuric acid present. When ferroin indicator is applied, the endpoint exhibits a noticeable colour shift. Standard titration tools including a burette, pipette, conical flask, and beaker are used in the process. The amount of ceric ammonium sulphate taken may be used to calculate the percentage purity of ferrous sulphate.

2 Principle

Ferrous sulphate is assayed by cerimetry, a redox titration where ferrous sulphate ($FeSO_4$) acts as a reducing agent and ceric ammonium sulphate [$(NH_4)_4Ce(SO_4)_4$] as an oxidizing agent. In the presence of dilute sulphuric acid, Fe^{2+} is oxidized to Fe^{3+} and Ce^{4+} is reduced to Ce^{3+}:

$$Ce^{4+} + Fe^{2+} \rightarrow Ce^{3+} + Fe^{3+}$$

Ferroin, an internal indicator, forms a red complex with Fe^{2+}. During titration, the red colour fades as Fe^{2+} is oxidized. When ferrous sulphate completely oxidises to ferric sulphate, colour changes from red to pale blue or white, signifying the endpoint.

$$2\ Ce(SO_4)_2(NH_4)_2SO_4 + 2\ FeSO_4 \longrightarrow Fe_2(SO_4)_3 + 2\ (NH_4)_2\ SO_4 + Ce_2(SO_4)_3$$

3 Procedure

Preparation of Ferrous ammonium sulphate:

Dissolve about 40 g of ferrous ammonium sulphate in a previously cooled mixture of 40 ml of sulphuric acid and 200 ml of water, dilute with sufficiently freshly boiled and cooled water to produce 1000 ml.

Standardization of 0.1 N ceric ammonium sulphate:

Measure accurately about 25 ml of above solution into a flask, add 2 drops of ferroin indicator and titrate with standard solution 0.1 N ceric ammonium sulphate until red colour is changed to blue.

Each ml of 0.1 N ceric ammonium sulphate is equivalent to 0.03921 g of Fe $(NH_4)_2(SO_4)_2.6H_2O$.

Assay Procedure:

About 3 grams of the sample should be precisely weighed in a 100 millilitre volumetric flask. Add water to get the volume up to the required level. Pipette 10 ml of solution, add 15 ml of dil.H_2SO_4, and then use ferroin as an indicator to titrate it with the standard solution of 0.1 N ceric ammonium sulphate until the red hue turns green. 0.1 N ceric ammonium sulphate is equal to 0.0278 g of $FeSO_4$ per millilitre.

4 Observation for Standardization

Conical flask: 10 ml Ferrous ammonium sulphate + 10 ml dil. H_2SO_4

Burette: Ceric ammonium sulphate

Indicator: 1,10 phenanthroline

End point: Colour changes from red to green

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	

4.1 Calculation:

$$\text{Normality} = \frac{\text{Weight of ferrous ammonium sulphate x Approximate Normality}}{\text{Volume of ceric ammonium sulphate x IP factor of FAS}}$$

5 Observation for Assay

Conical flask: 10 ml Ferrous sulphate + 15 ml dil. H_2SO_4

Burette: Ceric ammonium sulphate

Indicator: 1,10 phenanthroline (Ferroin)

End point: Colour changes from red to green

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	
1			
2			

5.1 Calculation:

$$\% \text{ Purity} = \frac{\text{Titre value} \times \text{IP factor of } FeSO_4 \times \text{Normality of CAS}}{\text{Weight of sample} \times \text{exact Normality} \times 100}$$

6 Result

The percentage purity of ferrous sulphate is found to be ……. %

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Chapter 12: Assay of Copper sulphate

1 Introduction

Determining the amount of copper sulphate in a sample is crucial for quality control since it is often utilised in a variety of chemical and pharmaceutical procedures. Iodometric titration, in which copper (II) ions react with potassium iodide to release iodine, is used in this experiment to measure the quantity of copper sulphate. A typical sodium thiosulphate solution is then used to titrate the released iodine using starch as an indicator, which reacts with iodine to form a blue complex and becomes colourless at the endpoint. The reaction is conducted in an iodine flask with strong hydrochloric acid and glacial acetic acid present. Accurately determining the copper sulphate concentration is aided by the amount of thiosulphate utilised.

2 Principle

The iodometric (redox) technique is used to determine copper sulphate. Here, a typical sodium thiosulphate solution is used to titrate the released iodine. When potassium iodide and glacial acetic acid are added to copper sulphate, cupric iodide and K2SO4 are produced. The formed cupric iodide decomposes into cuprous iodide with the liberation of iodine. The liberated iodine is titrated with standard solution of sodium thiosulphate using starch mucilage as the indicator.

The conversion of cupric iodide to cuprous iodide and iodine is reversible, so it is necessary to make the reaction irreversible by adding potassium thiocyanate. Otherwise, the liberated iodine again reacts with cuprous iodide and form cupric iodide. The potassium thiocyanate added, react with cuprous iodide and produce cuprous thiocyanate.

$$CuSO_4 + 2KI \longrightarrow CuI_2 + K_2SO_4$$

$$2CuI_2 \longrightarrow Cu_2I_2 \text{ (or } 2CuI) + I_2$$

$$2\ KSCN + Cu_2I_2 \longrightarrow 2\ KI + 2\ CuSCN$$

$$I_2 + 2\ Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$$

3 Procedure

Preparation of 0.1 N sodium thiosulphate:

Dissolve about 25 g of sodium thiosulphate in 100 ml of water and add 0.2 g of sodium carbonate in distilled water and dilute to 1000 ml with water.

Standardization of 0.1 N sodium thiosulphate:

Accurately weigh out 4.9 grams of potassium dichromate, then dissolve it in enough water to make 1000 millilitres. Ten millilitres of potassium dichromate solution should be pipetted into an iodine flask. After adding 1 millilitre of strong hydrochloric acid and 0.5 grams of potassium iodide, leave the mixture in a dark area for ten minutes. Following this, titrate the released iodine against the sodium thiosulphate standard solution until a pale-yellow hue is achieved. Continue the titration after adding starch as an indicator until the blue colour fades and a light green tint emerges.

Each ml of 0.1 N sodium thiosulphate is equivalent to 0.0049 g of $K_2Cr_2O_7$.

Assay Procedure (Estimation of Copper Sulphate):

Weigh accurately about 1 g of copper sulphate in a 100 ml volumetric flask and makeup the volume to the mark with distilled water. Mix well and pipette 10 ml of the solution into iodine flask. Add 3 ml of 10 % potassium iodide and 3 ml of glacial acetic acid stopper the flask and allow the reaction to take place for about 5 minutes. Titrate against the standard solution of sodium thiosulphate (0.1 N) till a pale-yellow colour is obtained. Now add the starch solution and 1 g of potassium thiocyanate and continue the titration till the blue colour disappears.

Each ml of 0.1 N $Na_2S_2O_3$ is equivalent to 0.02497 g of copper sulphate

4 Observation for Standardization

Conical flask: 10 ml of 0.1N $K_2Cr_2O_7$ + 0.5 gm of KI + 1 ml Conc. HCl

Burette: $Na_2S_2O_3$

Indicator: Starch Solution

End point: Appearance of light green colour

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	

4.1 Calculation:

$$\text{Normality of Sodium thiosulphate} = \frac{\text{Wt. of } K_2Cr_2O_7 \text{ in 10 ml} \times 0.1}{\text{Volume of } Na_2S_2O_3 \times \text{ IP factor of } K_2Cr_2O_7}$$

5 Observation for Assay

Conical flask: 10 ml of copper sulphate solution + 3 ml of potassium iodide + 3 ml of glacial acetic acid + 1 gm of potassium thiocyanate

Burette: Sodium thiosulphate

Indicator: Starch solution

End point: Disappearance of blur colour

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	
1			
2			

5.1 Calculation:

$$\text{Percentage purity} = \frac{\text{Titre value} \times \text{I.P factor} \times \text{Calculated normality of } Na_2S_2O_3 \times 100}{\text{Weight of the sample} \times \text{Approximate normality}}$$

6 Result

The percentage purity of copper sulphate is found to be ……. %

Reference

1. Sharma BK. *Instrumental Methods of Chemical Analysis*. 25th ed. Meerut: Goel Publishing House; 2019.
2. Vogel AI. Vogel's *Quantitative Chemical Analysis.* 7th ed. Pearson Education; 2014.
3. Sethi PD. *Quantitative Analysis of Drugs in Pharmaceutical Formulations*. 3rd ed. New Delhi: CBS Publishers; 2008.
4. Christian GD. *Analytical Chemistry.* 6th ed. John Wiley & Sons; 2004.

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Chapter 13: Assay of Calcium gluconate

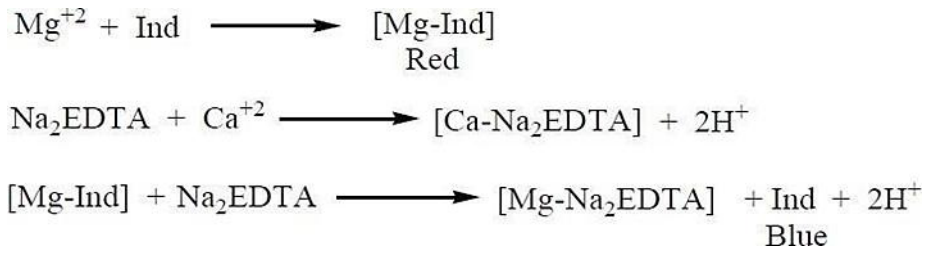
1 Introduction

Evaluating the purity of calcium gluconate, a calcium supplement used in pharmaceutical formulations, is crucial for quality control. Using disodium EDTA as the titrant, complexometric titration is used in this experiment to determine the calcium gluconate's percentage purity. A stable complex is created when the EDTA and calcium ions in the sample interact. The pH is maintained by adding mordant black II indicator, which changes colour at the endpoint, and a strong ammonia-ammonium chloride buffer. Standard tools including a burette, pipette, conical flask, and beaker are used for the titration. Calculating the calcium concentration and purity of the sample is made possible by the amount of EDTA employed.

2 Principle

The complexometric replacement titration technique is used to measure calcium gluconate. Ammonia-ammonium chloride buffer and a specified amount of magnesium sulphate are added in order to estimate the calcium ions. Calcium does not react with indicator satisfactory and will not give proper end point. So $MgSO_4$ is used which reacts with indicator and free calcium ions easily react with standard Na_2EDTA (Titrant) to produce Ca-EDTA complex. When all the Ca^{++} ions gets reacted, extra amount of Na_2EDTA react with $MgSO_4$. And at last, when all the Mg^{++} form Mg-EDTA complex, free indicator shows different colour.

The same amounts of reagent are used in a second titration without a sample. This gives the amount of Na_2EDTA used by magnesium sulphate. The quantity of disodium edetate that the calcium gluconate consumed is determined by the difference between two titrations.



3 Procedure

Preparation of 0.05 M Zinc chloride:

Weigh 6.15g of zinc chloride and transfer into 1000 ml volumetric flask. Dissolve in minimum amount of water and make up the volume to 1000ml with water.

Standardization of 0.05 M Disodium EDTA:

Pipette out 10ml of this solution into a clean conical flask. Add 10 ml of ammonia-ammonium chloride buffer (pH 10) and 2 drops Mordant black II indicator. Titrate against 0.05 M disodium edetate till the colour changes to blue.

Assay Procedure:

Fill a 250 ml conical flask with precisely 0.5 g of calcium gluconate, then dissolve it with 50 ml of water. Using a mordant black mixture as an indicator, titrate the mixture with the standard solution of disodium edetate (0.05 M) after adding 5 ml of magnesium sulphate (0.05 M) and 10 ml of strong ammonia-ammonium chloride solution until the colour becomes blue. Perform the blank titration using same reagents except the sample and note the reading of edetate solution. The difference gives the amount of disodium edetate required by the sample.

Each ml of 0.05 M disodium edetate is equivalent to 0.02242 g of calcium gluconate

4 Observation for Standardization

Conical flask: 10 ml Zinc chloride solution + 10 ml Ammonia – Ammonium chloride buffer

Burette: Disodium EDTA

Indicator: Mordant black II indicator

End point: Colour change to blue

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	

4.1 Calculation:

Molarity of disodium edetate

$$V_1 M_1 = V_2 M_2$$

V_1= Volume of zinc chloride

M_1= Molarity of zinc chloride

V_2= Volume of disodium edetate

M_2= Molarity of disodium edetate

5 Observation for Assay

Conical flask: calcium gluconate + magnesium sulphate + ammonia-ammonium chloride solution

Burette: Disodium EDTA

Indicator: Mordant black II indicator

End point: Colour change to blue

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	
1			
2			

5.1 Calculation:

Percentage purity = $\frac{\text{Titre value x I.P factor} \times \text{calculated Molarity of disodium edetate}}{\text{Weight of the sample} \times \text{Exact Molarity} \times 100}$

6 Result

The percentage purity of calcium gluconate is found to be ……. %

Reference

1. Vyas SP, Kohli DV. *Pharmaceutical Drug Analysis.* 2nd ed. New Delhi: CBS Publishers; 2020.
2. Chatwal GR, Anand SK. *Instrumental Methods of Chemical Analysis*. 5th ed. Mumbai: Himalaya Publishing House; 2014.
3. Harvey D. *Modern Analytical Chemistry*. 1st ed. New York: McGraw-Hill; 2000.
4. Basset J, Denney RC, Jeffery GH, Mendham J. Vogel's *Textbook of Quantitative Chemical Analysis***.** 6th ed. Harlow: Pearson Education; 2000.

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Chapter 14: Assay of Hydrogen peroxide

1 Introduction

Strong oxidising agents like hydrogen peroxide are frequently employed as bleaching and disinfecting agents. To guarantee appropriate use and safety, its strength needs to be precisely established. This experiment uses 0.1N potassium permanganate as the titrant in an acidic medium supplied by diluted sulphuric acid to determine the quantity of hydrogen peroxide in a given sample using redox titration. When hydrogen peroxide is oxidised to oxygen by potassium permanganate, a faint, persistent pink colour appears as an endpoint. Standard equipment including a burette, pipette, conical flask, and beaker are used for the titration.

2 Principle

The principle involved in the assay of H_2O_2 is oxidation-reduction reaction. It serves as an illustration of permanganometric titration. One weak oxidising agent is hydrogen peroxide. It functions as a reducing agent when a potent oxidising agent, such as potassium permanganate, is present. Potassium permanganate in presence of sulphuric acid reduces hydrogen peroxide to water and oxygen. There is no indicator used because the KMnO4 itself indicate the end point of the titration (shelf indicator) by imparting its pink/purple colour to solution.

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$H_2O_2 + [O] \longrightarrow H_2O + O_2 \quad \text{——— X 5}$$

$$2KMnO_4 + 3\,H_2SO_4 + 5\,H_2O_2 \longrightarrow K_2SO_4 + 2\,MnSO_4 + 8\,H_2O + 5\,O_2$$

Principle involved in standardization:

Primary standards such as oxalic acid in the presence of diluted sulphuric acid at 70 °C are used to standardise potassium permanganate. Standardization of $KMnO_4$ is a

permanganometric type of titration, where $KMnO_4$ is a powerful oxidizing agent that reacts with H_2SO_4 and liberates nascent oxygen. The liberated oxygen oxidizes oxalic acid to carbon dioxide and water.

$$\mathbf{2\ KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2}$$

Note:

1. Because $KMnO_4$ is poorly soluble in water, water must be added repeatedly, bit by portion.
2. The $KMnO_4$ is heat sensitive so we can't apply heat to increase solubility.
3. The reactivity of oxalic acid with $KMnO_4$ is very slow. to make the reaction faster we should the carry out the reaction at 70°C

3 Procedure

Preparation of 0.1N potassium permanganate:

In 100 millilitres of volumetric flask, dissolve approximately 0.32 grams of $KMnO_4$ in a small amount of distilled water, then add more distilled water to make up the remaining content. Let it stand for two days after heating it in a water bath for an hour. Pass through glass wool as a filter.

Standardization of 0.1N potassium permanganate:

Accurately weigh 0.63 grams of oxalic acid, dissolve it in 1000 millilitres of water, and then fill a 100 millilitre volumetric flask to the appropriate level. Ten millilitres of 0.1 N oxalic acid solution should be pipetted into a conical flask. Before worming the solution up to 60–70° C, add 5 ml of concentrated H_2SO_4. In worm condition titrate with standard solution of $KMnO_4$ (0.1N). The final result is a persistently light pink look. To obtain the concordant results, repeat the titration.

Assay Procedure:

Pipette and titrate with the standard potassium permanganate (0.1 N) solution, which serves as a self-indicator, after adding 5 ml of diluted sulphuric acid to 10 ml of H_2O_2 solution in a conical flask. The appearance of pink is the finale.

Each ml of 0.1 N $KMnO_4$ is equivalent to 0.001701 g of H_2O_2.

4 Observation for Standardization

Conical flask: 10 ml Oxalic acid + 5 ml Sulphuric acid

Burette: Potassium permanganate

Indicator: Self-indicator ($KMnO_4$)

End point: Appearance of pale pink colour

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	

Normality of potassium permanganate

$$N_1V_1 = N_2 V_2$$

V_1= Volume of oxalic acid

N_1= Normality of oxalic acid

V_2= Volume of potassium permanganate

N_2= Normality of potassium permanganate

Normality (N_2) = V_1 x N_1/V_2

5 Observation for Assay

Conical flask: 10 ml Hydrogen peroxide + 5 ml Sulphuric acid

Burette: Potassium permanganate

Indicator: Self-indicator ($KMnO_4$)

End point: Appearance of pale pink colour

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	
1			
2			

5.1 Calculation:

$$\text{Percentage Purity} = \frac{\text{Titre Value x I.P Factor x Normality of KMnO4 x 100}}{\text{Weight of the sample x Exact Normality}}$$

6 Result

The percentage purity of Hydrogen peroxide is found to be ……. %

Reference

1. Sharma BK. *Instrumental Methods of Chemical Analysis*. 25th ed. Meerut: Goel Publishing House; 2019.
2. Indian Pharmacopoeia Commission. Indian Pharmacopoeia 2018. Vol. II. Ghaziabad: IPC; 2018.
3. Skoog DA, West DM, Holler FJ, Crouch SR. *Fundamentals of Analytical Chemistry*. 9th ed. Cengage Learning; 2014.
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Chapter 15: Assay of Sodium benzoate

1 Introduction

Sodium benzoate is frequently used in food and pharmaceutical products as a preservative. To ensure adherence to quality requirements, the % purity must be verified. Perchloric acid is used as the titrant in a glacial acetic acid medium in this non-aqueous titration experiment to measure the purity of sodium benzoate. Crystal violet, an indicator that changes colour at the endpoint, is used to perform the titration. The perchloric acid solution is standardised using potassium hydrogen phthalate. For precise titration, the process uses common equipment such a burette, pipette, conical flask, and beaker.

2 Principle

The assay of sodium benzoate is carried out by using non-aqueous titration method. Sodium benzoate is weak base. It is dissolved in acetic acid which exerts the leveling effect on it and increase the basic strength. Acetous perchloric acid give rise to onium ion, which is a very strong acid. On titrating with perchloric acid, sodium benzoate yields benzoic acid and sodium per chlorate. Crystal violet is used as indicator.

$$HClO_4 + CH_3COOH \rightarrow CH_3COOH^{2+} + ClO4^-$$

$$C_6H_5COO^- Na^+ + CH_3COOH \rightarrow CH_3COO^- + C_6H_5COOH^+ Na^+$$

$$CH_3COOH^{2+} + CH_3COO^- \rightarrow 2CH_3COOH$$

$$\text{Final reaction: } C_6H_5COONa + HClO_4 \rightarrow C_6H_5COOH + NaClO_4$$

Perchloric acid is standardized by using the primary standard potassium hydrogen phthalate in non aqueous solution. Potassium hydrogen phthalate, the primary standard used here is also a weak base. The reaction is expressed in following reaction.

$$KHP\ (C_8H_5KO_4) + HClO_4 \rightarrow KClO_4 + C_8H_6O_4$$

Note: Acetic anhydride used in the preparation of perchloric acid reacts with water present in it and converts it to acetic acid rendering the mixture virtually anhydrous.

3 Procedure

Preparation of 0.1 N Perchloric acid

Mix 500 millilitres of anhydrous glacial acetic acid and 25 millilitres of acetic anhydride with about 8.5 millilitres of 70% perchloric acid. Anhydrous glacial acetic acid is added after cooling to get the volume up to 1000 millilitres.

Standardization of 0.1 N Perchloric acid

Weigh accurately about 0.35 g of potassium hydrogen phthalate and dissolved in 25 ml of glacial acetic acid, add 2-3 drops of crystal violet as indicator and titrate with standard solution of perchloric acid (0.1 N) until emerald green colour is obtained.

Perform a blank titration to find out the volume of perchloric acid required by 25 ml of glacial acetic acid.

Each ml of 0.1 N $HClO_4$ is equivalent to 0.02041 g of $C_8H_5KO_4$

Assay Procedure

Weigh accurately about 0.3 g of Sodium benzoate sample and add 25 ml of glacial acetic acid. Mix well and titrate with the standard solution of 0.1 N perchloric acid using crystal violet as the indicator. Perform a blank determination to make necessary correction.

Each ml of 0.1 N perchloric acid is equivalent to 0.01441 g of sodium benzoate.

4 Observation for Standardization

Conical flask: potassium hydrogen phthalate + glacial acetic acid

Burette: Perchloric acid

Indicator: Crystal violet

End point: Appearance of emerald green colour

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	

$$\text{Normality of perchloric acid} = \frac{\text{Weight of KHP} \times \text{Normality}}{\text{Titre value} \times \text{IP factor of KHP}}$$

5 Observation for Assay

Conical flask: Sodium benzoate + glacial acetic acid

Burette: Perchloric acid

Indicator: Crystal violet

End point: Appearance of emerald green colour

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	
1			
2			

5.1 Calculation:

$$\textbf{Percentage purity} = \frac{\text{Titre value} \times \text{I.P factor} \times \text{Normality of } HClO_4 \times 100}{\text{Weight of the sample} \times \text{Approximate normality}}$$

6 Result

The percentage purity of sodium benzoate was found to be ……%

Reference

1. Vyas SP, Kohli DV. Pharmaceutical Drug Analysis. 2nd ed. New Delhi: CBS Publishers; 2020.
2. Jain NK. Pharmaceutical Product Development. 1st ed. New Delhi: CBS Publishers; 2016.

3. Ahuja S. Modern Pharmaceutical Analysis. 1st ed. New Delhi: CBS Publishers; 2007.

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Chapter 16: Assay of Sodium chloride

1 Introduction

A common constituent in medicines, sodium chloride requires precise purity determination in order to ensure quality and standard compatibility. In this experiment, the Volhard's method of precipitation titration is used to measure the % purity of sodium chloride. This method develops a precipitate of silver chloride by reacting sodium chloride with an excess of conventional silver nitrate. After that, nitrobenzene is added to stop the coagulation of silver chloride, and the unreacted silver nitrate is back-titrated with standard ammonium thiocyanate using ferric ammonium sulphate as an indicator. Common glassware is utilised, such as conical flasks, pipettes, and burettes.

2 Principle

Sodium chloride is assayed by Volhard's method of precipitation reaction. In this method excess of silver nitrate is used. Sodium chloride reacts with silver nitrate forming silver chloride and sodium nitrate.

$$NaCl + AgNO_3 \xrightarrow{\text{dil. } HNO_3} AgCl + NaNO_3$$

Unreacted silver nitrate is titrated with standard solution of ammonium thiocyanate. silver nitrate will react with ammonium thiocynate to form silver thiocynate.

$$Ag\,NO_3 + NH_4SCN \longrightarrow AgSCN + NH_4\,NO_3$$

Then finally the indicator ferric ammonium sulphate will react with ammonium thiocynate to form ferric thiocynate, which gives the complex reddish yellow colour.

$$NH_4Fe(SO_4)_2 + 3\,NH_4SCN \longrightarrow Fe(SCN)_3 + 2\,(NH_4)_2SO_4$$

The formed AgCl may interfere with other reaction, so nitrobenzene is added to coat the precipitate of silver chloride, this coating of AgCl decreases its solubility and prevent it

from reaction with ammonium thiocyanate. In the absence of nitrobenzene more ammonium thiocynate will be required during titration. Nitric acid is given to stop ferrous salt from hydrolysing and to stop precipitating silver carbonate, phosphate, etc.

3 Procedure

Preparation of 0.1 N Ammonium thiocyanate:

Dissolve about 7.612 g of ammonium thiocyanate in sufficient water to produce 1000 ml.

Standardization of 0.1 N Ammonium thiocyanate:

Pipette and fill a 50 ml volumetric flask with 30 ml of 0.1 N silver nitrate, dilute it with 50 ml of water, add 2 ml of nitric acid and 2 ml of ferric ammonium sulphate solution, and titrate using the ammonium thiocyanate standard solution. The arrival of reddish-brown is the last stage.

Each ml of 0.1 N NH_4SCN is equivalent to 0.007612 g of silver nitrate.

Assay Procedure:

Weigh accurately about 0.1 gm of NaCl and dissolve in 50 ml of distilled water in a glass stoppered flask, add 50 ml of 0.1N $AgNO_3$solution and 3 ml of nitric acid. Shake well and add 5 ml of nitrobenzene and 2 ml of ferric ammonium sulphate (0.1N). The end point is the appearance of red colour.

Each ml of 0.1 N $AgNO_3$ is equivalent to 0.005845 gm of sodium chloride

4 Observation for Standardization

Conical flask: Silver nitrate

Burette: ammonium thiocyanate

Indicator: Ferric ammonium sulphate

End point: Appearance of Reddish-brown colour

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	

Normality of potassium permanganate

$$N_1V_1 = N_2 V_2$$

V_1= Volume of Silver nitrate

N_1= Normality of Silver nitrate

V_2= Volume of ammonium thiocyanate

N_2= Normality of ammonium thiocyanate

$$\text{Normality } (N_2) = V_1 \text{ x } N_1/V_2$$

5 Observation for Assay

Conical flask: Sodium chloride + Silver nitrate

Burette: Ammonium thiocyanate

Indicator: Ferric ammonium sulphate

End point: Appearance of Reddish-brown colour

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	
1			
2			

5.1 Calculation:

$$\% \text{ Purity} = \frac{\text{T.V X IP factor of } AgNO_3 \text{ X Normality}}{\text{Weight of the sample X exact Normality}} \text{ X } 100$$

6 Result

The percentage purity of sodium chloride was proved to be ……%

Reference

1. Harris DC. *Quantitative Chemical Analysis*. 10th ed. Freeman; 2022.
2. Patil RP, Chaudhari SR. *Pharmaceutical Drug Analysis*. 2nd ed. Pune: Career Publications; 2021.
3. Kulshreshtha V. *Analytical Chemistry: Theory and Practice*. 1st ed. Meerut: Pragati Prakashan; 2020.
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Chapter 17: Strong acid vs Strong base Conductometric titration

1 Introduction

An analytical method based on measuring changes in conductivity during a chemical reaction is called conductometric titration. By titrating the provided hydrochloric acid (HCl) solution with a standard sodium hydroxide (NaOH) solution, the experiment's normalcy is ascertained. A conductometer is used to measure the change in the solution's ionic concentration and conductivity when HCl is neutralised by NaOH. A conductivity vs. volume plot is used to graphically identify the endpoint. Throughout the titration, consistent mixing is ensured using a magnetic stirrer. Before titrating a NaOH solution, oxalic acid can be used as a reference or standard.

2 Principle

Conductometric titration of strong acid against strong base.

$$HCl + NaOH \rightarrow NaCl + H2O$$

At the beginning of titration, when HCl is neutralized by adding NaOH this conditions hydrogen and chloride ions since hydrogen ion possess the greater mobility of ion if it follows that the greater part of conductivity will be due to it.

As NaOH is added the concentration of H^+ ion is decreased and although H^+ ion is replaced by Na^+ ion, the mobility of the latter is much less so that conductivity of solution decreases. The solution of neutralization at the end point of contact contains only sodium, chloride ions and will have minimum conductance. Now if little NaOH is added after neutralization the conductivity again increases owing to the presence of OH^- ions when compared to Na^+ ions, since it has greater mobility thus the titration is carried out at constant temperature, conductivity is plotted against quantity of NaOH added, a curve is obtained. The end point is the intersection of two lines.

3 Procedure

Take 20 ml of 1N HCl solution into a container and maintain at room temperature. Prepare 1N solution of NaOH and standardize against standard oxalic acid solution. Transfer the NaOH into clean burette and make up to the volume. Start the titration against 1 N HCl by slowly adding 0.1 ml of NaOH every time and measure the conductance value. The conductance value initially decreases, passes through minimum and then increases again.

Plot a graph of conductance value measured on y-axis against volume of titrant NaOH added. From the graph determine the endpoint. Carry out a titration of the same volume of HCl against 1 N NaOH using phenolphthalein as indicator and determine the end point by normal titration method. Co-relate the two-end point by two different methods find out the normality of the given HCl solution and report.

4 Observation for Standardization

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	

$$\text{Normality of NaOH} = \frac{\text{Volume of oxalic acid taken x Normality of oxalic acid}}{\text{Volume of NaOH consumed}}$$

5 Observation for Assay

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	
1			
2			

5.1 Calculation:

$$\text{Normality of HCl} = \frac{\text{Volume of NaOH consumed x Normality of NaOH}}{\text{Volume of HCl consumed}}$$

Reading of Conductometric titration

Volume of HCl	Volume of NaOH added	Measured conductance

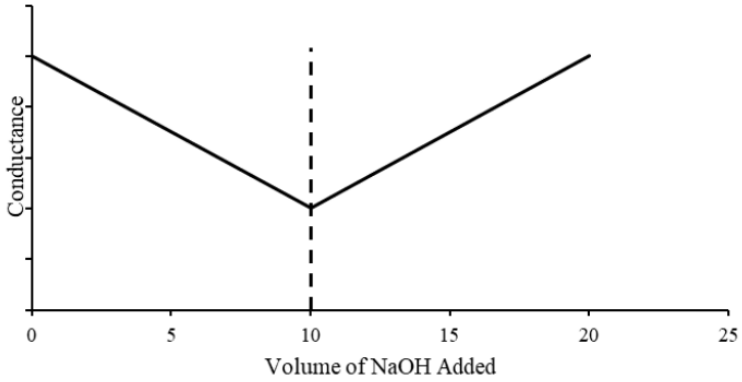

Fig. 2 Conductometric titration of strong acid against strong base

It should be possible to shake the conductivity cell used for this titration and add the reagent from the burette. It is best to prevent a significant volume increase during titration.

6 Result

The normality of the given HCl solution by Conductometric titration is ___N

Reference

1. Sharma BK. *Instrumental Methods of Chemical Analysis*. 25th ed. Meerut: Goel Publishing House; 2019.
2. Chatwal GR, Anand SK. *Instrumental Methods of Chemical Analysis*. 5th ed. Mumbai: Himalaya Publishing House; 2014.
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Chapter 18: Weak acid vs Strong base Conductometric titration

1 Introduction

By measuring variations in the solution's electrical conductivity, conductometric titration provides a practical method for identifying a reaction's endpoint. This experiment uses a standard sodium hydroxide (NaOH) solution to titrate acetic acid, a weak acid, in order to establish its normalcy. Because highly mobile acetate and hydroxide ions are formed when NaOH is added, the conductivity rises. Plotting conductivity vs NaOH volume yields the endpoint. Conical flasks, burettes, pipettes, beakers, conductometers, and conductivity cells are among the equipment utilised. The NaOH solution can be standardised with oxalic acid.

2 Principle

At the beginning of titration, when CH3COOH is taken in a beaker as titrate, the initial conductivity is low because weak acid does not dissociates into H+ ions. When NaOH is added as a titrant, formation of CH3COONa takes place there is a slight increase in the conductivity till the end point. After the end point, the addition of NaOH causes to increases rapidly. The point of interaction of two lines is the end point.

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$$

3 Procedure

Take 10 ml of 1N acetic acid solution into a container and maintain at room temperature. Prepare 1N solution of NaOH and standardize against standard oxalic acid solution. Transfer the NaOH into clean burette and make up to the volume. Start the titration against 1 N acetic acid slowly adding 0.1 ml of NaOH every time and measure the conductance value. The conductance value initially decreases, passes through minimum

and then increases again. Plot a graph of conductance value measured on y-axis against volume of titrant NaOH added. From the graph determine the endpoint. Carry out a titration of the same volume of acetic acid against 1 N NaOH using phenolphthalein as indicator and determine the end point by normal titration method. Co-relate the two-end point by two different methods find out the normality of the given acetic acid solution and report.

4 Observation for Standardization

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	

$$\text{Normality of NaOH} = \frac{\text{Volume of oxalic acid taken x Normality of oxalic acid}}{\text{Volume of NaOH consumed}}$$

5 Observation for Assay

Sl. No	Reading of the burette in millilitre		Volume of titrant
	Initial	Final	
1			
2			

$$\text{Normality of HCl} = \frac{\text{Volume of NaOH consumed x Normality of NaOH}}{\text{Volume of HCl consumed}}$$

Reading of Conductometric titration

Volume of Acetic acid	Volume of NaOH added	Measured conductance

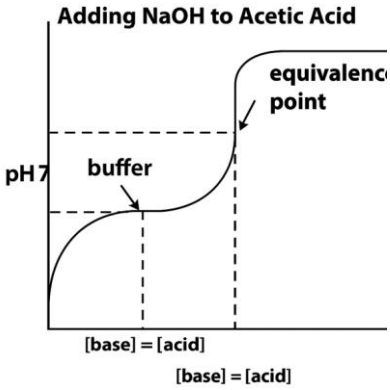

Fig. 3 Conductometric titration of weak acid against strong base

For this titration, the conductivity cell should allow for shaking agitation and the addition of reagent from the burette. Avoid a significant volume increase throughout the titration process.

6 Result

The normality of the given acetic acid solution by Conductometric titration is ___N

Reference

1. Harris DC. *Quantitative Chemical Analysis*. 10th ed. Freeman; 2022.
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5.

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Chapter 19: Strong acid vs Strong base Potentiometric titration

1 Introduction

To determine the endpoint of a reaction, an instrumental method known as potentiometric titration examines the change in pH or electrode potential. In this experiment, a potentiometer or pH meter is used to titrate a strong acid (0.1N HCl) against a strong base (0.1N NaOH). After each addition of NaOH, the pH is measured, and the titration curve's abrupt pH shift indicates the endpoint. Devices such a conical flask, pipette, burette, beaker, and magnetic stirrer are employed. To guarantee accuracy, the NaOH may be standardised with oxalic acid prior to the titration.

2 Principle

An electrical analytical technique called potentiometry measures the electrical potential, or emf, of an electrolyte solution (analyte) while the current is constant (zero current).

The potential of the provided sample is directly proportional to the concentration of its electroactive ions, or more precisely, the activity of the electroactive ions, or pH, which is the foundation of the principle. At the end (equivalence) point, the voltage across the appropriate reference and indicator electrodes submerged in the solution changes dramatically, which is necessary for potentiometric measurement of the end point. Although the potentiometric approach is more precise, this change is comparable to the colour shift caused by the indication in the visual method. When no appropriate colour indicator is available, potentiometric titrations might be helpful. Plotting the normal, first derivative, and second derivative curves allows for the precise determination of the equivalency point.

3 Procedure

3.1 Preparation of buffer solution buffer solution:

Dissolve the content of pH 4 capsule completely in small quantity of distilled water and make up the volume to 100 ml with distilled water.

pH 7 solution:

Dissolve completely the content of pH 7 capsule in distilled water and make up to 100 ml with distilled water.

pH 9 solution:

Dissolve completely the content of pH 9 capsule in distilled water and make up to 100 ml with distilled water.

3.2 Calibration of pH meter:

- Switch on the pH meter before 5 minutes to its use, immerse the electrode in distilled water for few minutes and wipe it dry with tissue paper. Place the electrode in pH 4 buffer solutions observe the reading and set the instrument to read pH 4. Then remove the electrode and wash it with distilled water, dry again with tissue paper.
- Place the electrodes in pH solution observe the reading calibrate to read pH 7 if required. Similarly calibrate with pH 9 solutions also.
- Standardization of 0.1 N NaOH solution: the standardization is done against the standard 0.1N Oxalic acid solution using 4 pH indicator and calculate the normality of NaOH.
- Visual method of titration of given HCl and titrate against NaOH solution: pipette out 10 ml of given HCl and titrate with standard NaOH solution.
- Potentiometric titration of given HCl: pipette out 50 ml of given HCl to beaker and place the electrode in solution and note the pH value. Titrate the HCl with standard NaOH adding the NaOH in suitable increment and note down pH value after each addition add the titrant in small increment of 0.1 ml in vicinity of endpoint.

- Plot a graph of pH Vs Volume of titrant added and locate the endpoint from graph. Determine the endpoint also by the 1st and 2nd derivative method and report.

4 Observation

Standardization of 0.1N NaOH by oxalic acid

W1 =

W2 =

W1 – W2 =

$$\text{Strength of oxalic acid} = \frac{0.1 \times \text{Weight taken}}{0.63}$$

Oxalic acid vs NaOH

Sl. No	Initial reading	Final reading	Difference

$$\text{Normility of NaOH:} \frac{\text{Normality of Oxalic acid} \times \text{volume taken}}{\text{Volume of NaOH consumed}}$$

0.1 N Vs 0.1N NaOH

Sl. No	Initial reading	Final reading	Difference

$$\text{Normality of HCl} = \frac{\text{Volume of NaOH consumed} \times \text{Normality of NaOH}}{\text{Volume of HCl consumed}}$$

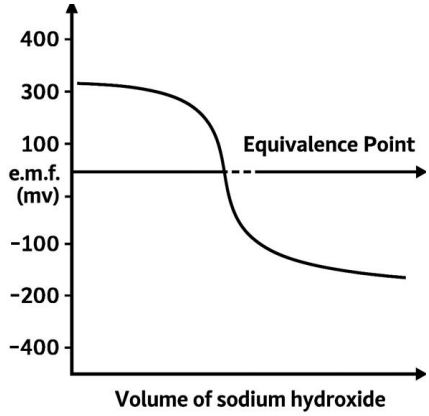

Fig. 4 Strong acid vs Strong base Potentiometric titration

6 Result

Using potentiometric titration, determine whether the provided acetic acid solution is ___N

Reference

1. Sharma BK. *Instrumental Methods of Chemical Analysis*. 25th ed. Meerut: Goel Publishing House; 2019.
2. Skoog DA, West DM, Holler FJ. *Fundamentals of Analytical Chemistry*. 9th ed. Boston: Cengage Learning; 2014.
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