



PRACTICAL LAB MANUAL

LAB MANUAL PHARMACEUTICAL INORGANIC AND ANALYTICAL CHEMISTRY

(Course code: BP111P)

B. PHARM Ist YEAR

Contents

Exp. No.	Title of Experiment	Page No.
1.	Limit test and modified limit test for chloride as per Indian Pharmacopoeia.	
2.	Limit test and modified limit test for sulphate as per Indian Pharmacopoeia.	
3.	Limit test for iron.	
4.	Limit test for lead.	
5.	Limit test for arsenic.	
6.	Preparation of aluminium hydroxide.	
7.	Preparation of potash alum.	
8.	Preparation of ferrous sulphate.	
9.	Preparation of magnesium sulphate from magnesium oxide or magnesium carbonate.	
10.	Assessment of swelling power of bentonite as per Indian Pharmacopoeia.	
11.	Evaluation of acid neutralizing capacity of aluminium hydroxide gel.	
12.	Determination of potassium iodate and iodine in potassium iodide.	
13.	Assay of ammonium chloride by acid-base titration.	
14.	Assay of ferrous sulphate by cerimetry.	
15.	Assay of copper sulphate by iodometry.	
16.	Assay of calcium gluconate by complexometric.	
17.	Assay of hydrogen peroxide by permanganometry.	
18.	Assay of sodium benzoate by non-aqueous titration.	
19.	Assay of sodium chloride by precipitation titration (Modified Volhard's Method).	

Experiment No. 1

Aim: To perform Limit test and Modified limit test for Chloride in a given sample as per Indian Pharmacopoeia.

Practical Significance: It is a vital quantitative or semi-quantitative procedure designed to identify and control small amounts of inorganic impurities likely to be present in pharmaceutical substances. They ensure drug safety, determine raw material purity, and monitor stability. They prevent potentially harmful or avoidable contaminants from exceeding safe physiological thresholds in drug formulations. Fluctuations in impurity levels over time indicate chemical degradation, batch variations, or compromised packaging. They provide a rapid, cost-effective alternative to highly advanced analytical techniques (like chromatography) for routine batch release testing.

Relevant Theoretical Background: The limit test relies on the precipitation reaction between soluble chloride ions and silver nitrate in the presence of dilute nitric acid. This produces an insoluble silver chloride precipitate, which appears as opalescence or turbidity. The intensity of the turbidity depends directly on the amount of chloride present in the sample, and it is visually compared with a standard solution of known chloride concentration.

The general limit test fails if the pharmaceutical sample is deeply colored (e.g., Potassium Permanganate) because the color masks the white silver chloride opalescence. The modified limit test involves a pre-treatment step to decolorize the sample before executing the standard chloride procedure. The colored substance is reduced and decolorized using a reducing agent like ethanol before testing.

Reactions:

- $\text{Cl}^- + \text{AgNO}_3 \rightarrow \text{AgCl} \downarrow + \text{NO}_3^-$
- $\text{NaCl} + \text{AgNO}_3 \rightarrow \text{AgCl} \downarrow + \text{NaNO}_3$

Requirements:

- **(a) Apparatus:** Volumetric flask, Nessler cylinder, Pipette, Beaker, Stirrer, Water bath, Funnel etc.
- **(b) Chemicals:** Sodium chloride, Nitric acid, Silver nitrate, Potassium permanganate, Ethanol, Chloride-free distilled water.

Procedure:

1. **Preparation of Standard Solution:** Weigh 0.05845 g of sodium chloride and dissolve it in sufficient distilled water to make 1000 mL. 1 mL of this solution contains 0.01 mg of chloride ions.
2. **Preparation of 0.1 N AgNO₃:** Dissolve 16.987 g Silver nitrate in water and make up the volume up to 1000 mL.

(A) For General Limit Test of Chloride:

1. Wash the pair of Nessler cylinders with distilled water and dry them.
2. Label them as "Sample" and "Standard".
3. Treat the sample and standard as per following table:

Sample Solution	Standard Solution
Place the specified quantity of the sample in a Nessler cylinder and dissolve it in 15 mL of distilled water (or as specified). Add 10 mL of dilute nitric acid and dilute to 50 mL with distilled water.	In a separate Nessler cylinder, place 1 mL of standard sodium chloride solution, add 10 mL of dilute nitric acid, and dilute to 50 mL with distilled water.

4. Add 1 mL of 0.1 M silver nitrate solution to both cylinders. Stir immediately with a glass rod and allow to stand for 5 minutes protected from direct sunlight.
5. Compare the opalescence (turbidity) of the test solution against the standard solution, viewed against a black background.

(B) For Modified Limit Test of Chloride:

1. **Pre-treatment / Decolorization:** Weigh a specific quantity of the colored substance (1.5 g potassium permanganate) in a flask and dissolve it in water. Heat it on a water bath and slowly add ethanol until the intense purple color disappears and a colorless solution or suspension forms.
2. **Filtration:** Cool the reduced mixture, dilute it with water, and filter to remove any insoluble manganese dioxide. Use the colorless filtrate as your test sample.
3. Treat the sample in a similar way from steps 3 to 5.

Observations:

1. **For General Limit Test of Chloride:** The opalescence (turbidity) produced in the given sample of Sodium chloride is less than / more than that of standard solution.
2. **For Modified Limit Test of Chloride:** The opalescence (turbidity) produced in the given sample of Potassium permanganate is less than / more than that of standard solution.

Results:

1. **For General Limit Test of Chloride:** The opalescence (turbidity) produced in the given sample of Sodium chloride was found to be less than / more than that of standard solution, hence passes / fails in the General limit test of Chloride.
2. **For Modified Limit Test of Chloride:** The opalescence (turbidity) produced in the given sample of Potassium permanganate was found to be less than / more than that of standard solution, hence passes / fails in the Modified limit test of Chloride.

Reference: Indian Pharmacopoeia, 2024.

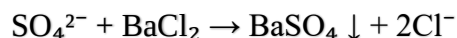
Experiment No. 2

Aim: To perform Limit test and Modified limit test for sulphate in the given sample as per Indian Pharmacopoeia.

Practical Significance: The practical significance of limit tests and modified limit tests for sulphate is to safely and semi-quantitatively control trace inorganic impurities in drug formulations. It ensures pharmaceutical safety by verifying that the sulphate content stays well below strict, permissible limits. It identifies and caps potentially harmful or reactive sulphate impurities (often residues from manufacturing processes like acid-based synthesis) that could cause adverse reactions or degrade the active pharmaceutical ingredient (API). It provides a rapid, visual, and cost-effective comparison between a sample and a standardized reference without needing advanced instrumental techniques (like HPLC). It proves whether a pharmaceutical substance meets official pharmacopoeial standards required for commercial release and patient consumption.

Relevant Theoretical Background: The Indian Pharmacopoeia outlines limit tests to control the presence of specific impurities in pharmaceutical substances. The standard and modified limit tests for sulphate are both precipitation reactions that yield turbidity, but they differ in how they handle samples that interfere with the reaction. This test relies on the reaction between soluble sulphate impurities and barium chloride in the presence of dilute hydrochloric acid. This forms an insoluble precipitate of barium sulphate that creates visible turbidity. Standard limit tests may fail if the active pharmaceutical ingredient (API) is colored, if it reacts with the barium reagent, or if it inhibits the formation of barium sulphate precipitate. A modified limit test is used for such substances.

Reaction:



Requirements:

- **(a) Apparatus:** Volumetric flask, Nessler cylinder, Pipette, Beaker, Stirrer, Water bath, Funnel etc.
- **(b) Chemicals:** Sample, Anhydrous sodium sulphate, HCl, Potassium permanganate, Ethanol, Sulphate-free distilled water.

Procedure:

1. **Preparation of Standard Solution:** Dissolve 0.147 g anhydrous sodium sulphate in 100 mL water.

(A) For General limit test of Sulphate:

1. Wash the pair of Nessler cylinders with distilled water and dry them.
2. Label them as "Sample" and "Standard".
3. Treat the sample and standard as per the following table:

Sample Solution	Standard Solution
Place the specified quantity of the sample in a Nessler cylinder, add 2 mL HCl, dilute to 45 mL of distilled water, add 1 mL of 5% barium chloride solution.	Place 1 mL of anhydrous sodium sulphate solution in a Nessler cylinder, add 2 mL HCl, dilute to 45 mL of distilled water, add 1 mL of 5% barium chloride solution.

4. Stir both the solutions immediately using a glass rod.
5. Allow both the cylinders to stand for 5 mins.
6. Compare the opalescence (turbidity) of the test solution against the standard solution, viewed against a black background.

(B) For Modified Limit Test of Sulphate:

1. If the sample contains inherent color (e.g., Potassium Permanganate) which interferes with the visibility of the turbidity, it must first be decolorized. Dissolve the sample in water, heat slowly on a water bath, and slowly add 95% ethanol until the color disappears.
2. Remove any resulting precipitate by filtration. Use the resulting colorless filtrate as your test solution.
3. Treat the sample in a similar way from steps 3 to 5.

Observations:

1. **For General Limit Test of Sulphate:** The opalescence (turbidity) produced in the given sample solution is less than / more than that of standard solution.
2. **For Modified Limit Test of Sulphate:** The opalescence (turbidity) produced in the given sample of Potassium permanganate is less than / more than that of standard solution.

Results:

1. **For General Limit Test of Sulphate:** The opalescence (turbidity) produced in the given sample was found to be less than / more than that of standard solution, hence passes / fails in the General limit test of Sulphate.
2. **For Modified Limit Test of Sulphate:** The opalescence (turbidity) produced in the given sample of Potassium permanganate was found to be less than / more than that of standard solution, hence passes / fails in the Modified limit test of Sulphate.

Reference: Indian Pharmacopoeia, 2024.

Experiment No. 3

Aim: To perform limit test for Iron in the given sample as per Indian Pharmacopoeia.

Practical Significance: The limit test for iron as per the Indian Pharmacopoeia (IP) is a semi-quantitative analytical procedure designed to identify and control trace amounts of iron impurities in pharmaceutical substances. Its primary practical significance is ensuring drug safety, stability, and adherence to global regulatory quality standards. The trace iron impurities can act as catalysts for the oxidative degradation of active pharmaceutical ingredients (APIs), reducing a drug's shelf life and causing toxicity. It verifies that raw materials and finished products fall below the maximum permissible limit of iron contamination (often expressed in ppm). Unwanted iron can react with excipients, altering color, odor, and therapeutic efficacy.

Relevant Theoretical Background: The ferric and ferrous ions in the sample react with thioglycollic acid. Thioglycollic acid acts as a reducing agent, converting all iron present in the sample into the ferrous state (Fe^{2+}). The ferrous ions then form a pale pink to deep reddish-purple ferrous thioglycollate complex. The color is distinctly visible only in an alkaline environment, necessitating the addition of ammonia. The intensity of the purple color produced by the test sample is compared visually against a standard iron solution. If the sample color is less intense than the standard, it passes the test.

Reactions:

1. Reduction of Ferric Iron (Fe^{3+} to Fe^{2+}): Thioglycollic acid reduces ferric ions to ferrous ions:
$$2\text{Fe}^{3+} + 2\text{HSCH}_2\text{COOH} \rightarrow 2\text{Fe}^{2+} + \text{HOOCCH}_2\text{S} - \text{SCH}_2\text{COOH} + 2\text{H}^+$$

(Thioglycollic acid gets oxidized to dithioglycollic acid.)
2. Formation of Ferrous Thioglycollate Complex: In ammoniacal (alkaline) medium
$$\text{Fe}^{2+} + 2\text{HSCH}_2\text{COO}^- \rightarrow \text{Fe}(\text{SCH}_2\text{COO})_2$$

This forms a purple/violet colored ferrous thioglycollate complex.

Requirements:

- **(a) Apparatus:** Volumetric flask, Nessler cylinder, Pipette, Beaker, Stirrer, Measuring cylinder etc.
- **(b) Chemicals:** Sample, Ferric ammonium sulphate, Thioglycollic acid, Citric acid, Ammonia solution, Ethanol, Distilled water.

Procedure:

1. **Preparation of Standard Iron Solution:** Add 0.1726 g ferric ammonium sulphate to 1.5 mL of HCl, add sufficient distilled water to produce 1000 mL.
2. Wash the pair of Nessler cylinders with distilled water and dry them.
3. Label them as "Sample" and "Standard".
4. Treat the sample and standard as per the following table:

Sample Solution	Standard Solution
Dissolve specific quantity of sample in 20 mL distilled water.	Place 2 mL standard solution and dilute to 20 mL with distilled water.
Add 2 mL of 20% w/v citric acid solution (iron-free) to prevent precipitation of iron by ammonia.	Add 2 mL of 20% w/v citric acid solution (iron-free) to prevent precipitation of iron by ammonia.
Add 0.1 mL of thioglycollic acid and mix.	Add 0.1 mL of thioglycollic acid and mix.
Make the solution alkaline by adding an iron-free ammonia solution.	Make the solution alkaline by adding an iron-free ammonia solution.
Dilute the final volume to 50 mL with distilled water. Allow the solutions to stand for 5 min.	Dilute the final volume to 50 mL with distilled water. Allow the solutions to stand for 5 min.

5. Compare the color intensity of the test solution against the standard solution.

Observation: The color intensity produced in the given sample is less than / more than that of standard solution.

Result: The color intensity produced in the given sample was found to be less than / more than that of standard solution, hence passes / fails in the general limit test of Iron.

Reference:

1. Indian Pharmacopoeia, 2024.

Experiment No. 4

Aim: To perform the limit test for Lead in the given sample as per pharmacopoeial standards.
Practical

Significance: The limit test for lead is a crucial qualitative and semi-quantitative analytical tool used to control heavy metal contamination in pharmaceutical substances. Lead is a cumulative poison that poses severe health hazards even in trace amounts. Conducting this test ensures that the sample meets the strict official standards for purity and safety before therapeutic use. Deviations or failures in the limit test indicate heavy metal contamination, which may arise from manufacturing equipment, raw materials, or improper storage conditions.

Relevant Theoretical Background: The limit test for Lead is based on the reaction between lead impurities and diphenylthiocarbazone (dithizone) in an alkaline medium. This extraction process yields a red-colored lead-dithizone complex. While the free dithizone reagent retains a distinct green color in chloroform, the formation of the complex shifts the organic layer toward a violet/red hue. The intensity of this color is directly proportional to the amount of lead present in the sample.

To ensure analytical accuracy, potential interferences from other heavy metal ions are completely eliminated by utilizing a specific cocktail of masking agents at an optimized alkaline pH:

- **Ammonium citrate:** Prevents the precipitation of iron and other metal hydroxides in alkaline media.
- **Potassium cyanide:** Forms stable, water-soluble complexes with metals like copper and zinc, stopping them from reacting with dithizone.
- **Hydroxylamine hydrochloride:** Acts as a reducing agent to prevent the oxidation of dithizone.

Reaction:

$\text{Pb}^{2+} + 2 \text{S}=\text{C}(\text{NH}-\text{NH}-\text{C}_6\text{H}_5)(\text{N}=\text{N}-\text{C}_6\text{H}_5) \xrightarrow{\text{[Alkaline media]}} \text{Pb-Dithizone Complex (Red)}$

Requirements:

- **(a) Apparatus:** Nessler's cylinders, Separating funnels, Pipettes, Beakers, Measuring cylinders, Glass rods.
- **(b) Chemicals:** Test sample, Standard lead solution, Diphenylthiocarbazone (Dithizone) extraction solution, Ammonium citrate solution, Potassium cyanide solution, Hydroxylamine hydrochloride, Phenol red indicator, Strong ammonia solution, Chloroform.

Procedure:

- **Test Sample (Prepared from Sample):**
 1. Take a known quantity of the test sample dissolved in water, or a solution prepared as directed in the individual monograph, and place it into a separating funnel.
 2. Add 6 mL of ammonium citrate solution.
 3. Add 2 mL of potassium cyanide solution and 1 mL of hydroxylamine

hydrochloride solution.

4. Add 2 drops of phenol red indicator and make the solution alkaline by adding strong ammonia solution until a distinct red color develops.
5. Extract the mixture by shaking vigorously with small portions (approx. 5 mL) of dithizone extraction solution until the chloroform layer remains green.
6. Combine the chloroform extracts, shake with a dilute acid/buffer if required by the monograph, and transfer the final organic layers to matching Nessler's cylinders for visual comparison.

- **Standard Lead Solution (Reference):**

1. Pipette out a standard volume of Standard Lead Solution (e.g., corresponding to the target limit) into a separate separating funnel.
2. Add 6 mL of ammonium citrate solution.
3. Add 2 mL of potassium cyanide solution and 1 mL of hydroxylamine hydrochloride solution.
4. Add 2 drops of phenol red indicator and make the solution alkaline by adding strong ammonia solution until a distinct red color develops.
5. Extract the mixture by shaking vigorously with small portions (approx. 5 mL) of dithizone extraction solution until the chloroform layer remains green.
6. Combine the chloroform extracts, shake with a dilute acid/buffer if required by the monograph, and transfer the final organic layers to matching Nessler's cylinders for visual comparison.

Precaution: Potassium cyanide is highly toxic. Handle it with extreme care using gloves, and never pipette cyanide solutions by mouth. Ensure all extractions are performed in a well-ventilated fume hood.

Observations:

- **(a) Reagent Color (Free Dithizone in Chloroform):** Green.
- **(b) Complex Color (Lead-Dithizone in Chloroform):** Violet / Pinkish-Red.
- **(c) Color Intensity of Standard Solution:** High / Moderate.
- **(d) Color Intensity of Test Solution:** _____

Observation Table:

Cylinder	Intensity of Red/Violet Color in Chloroform Layer	Inference
Standard Solution	Reference standard color intensity	Compares with standard
Test Solution		

Result: The intensity of the color in the chloroform layer of the test solution was found to be (more than / less than) that of the standard solution. Therefore, the percent or trace concentration of Lead in the given sample is (within / above) the prescribed official pharmacopoeial limit.

Conclusion: The given sample of _____(passes / fails) the limit tests for Lead as per the pharmacopoeial standards.

Reference:

1. Indian Pharmacopoeia, 2024.

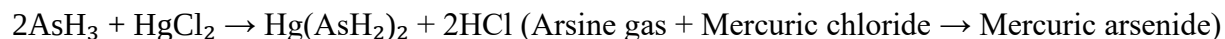
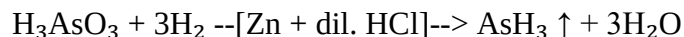
Experiment No. 5

Aim: To perform the limit test for Arsenic in the given sample of Ammonium Chloride.

Practical Significance: The limit test for Arsenic is a highly critical quality control check used to detect trace levels of arsenic contamination in pharmaceutical ingredients. Arsenic is a notorious cumulative toxin, making its strict monitoring essential to guarantee patient safety and product purity. Conducting this test confirms whether the batch complies with official pharmacopoeial purity guidelines before it can be cleared for medical formulation. Any deviation or failure in this test points to heavy metal impurities, which generally slip into the product via raw manufacturing components, chemical reagents, or processing machinery.

Relevant Theoretical Background: The limit test for Arsenic is entirely based on the Gutzeit test, where all arsenic impurities present in the sample are chemically reduced into volatile arsine gas (AsH_3). The sample is treated with an acid to transform arsenic impurities into arsenic acid. Powerful reducing agents like potassium iodide, stannous chloride, and zinc metal progressively reduce this into arsenious acid. Nascent hydrogen, generated continuously through the reaction between zinc and hydrochloric acid, then reduces the arsenious acid into gaseous arsine.

As the arsine gas rises up the tube of the Gutzeit apparatus, it encounters a mercuric chloride test paper, reacting with it to produce a distinct yellow-to-brown stain. The intensity and physical depth of this stain correlate directly with the amount of arsenic present. To eliminate potential chemical interference from sulfides, a lead acetate cotton plug is placed in the tube to scrub out any hydrogen sulfide (H_2S) gas by trapping it as lead sulfide.

Reaction Sequence:**Requirements:**

- **(a) Apparatus:** Gutzeit apparatus complete with bottle and specialized glass tubes, measuring cylinders, beakers, pipettes, and a water bath or warm hot plate.
- **(b) Chemicals:** Ammonium chloride test sample, Zinc metal AsT, Potassium iodide AsT, Stannous chloride AsT, Hydrochloric acid AsT, Lead acetate cotton wool, Mercuric chloride paper, and Arsenic trioxide.

Procedure:

- **Part A: Preparation of Specialized Reagents**
 - **Stannous chloride solution AsT:** Dissolve 330 g of stannous chloride in 100 mL of hydrochloric acid and add sufficient water to make 1000 mL. Boil an equal volume of this solution with hydrochloric acid AsT to reduce it back to its original volume, then filter.
 - **Stannated hydrochloric acid AsT:** Thoroughly mix 1 mL of stannous chloride solution AsT with 100 mL of hydrochloric acid AsT.
 - **Potassium iodide solution:** Dissolve 16.6 g of potassium iodide in a sufficient volume of water to yield a final volume of 100 mL.
 - **Standard arsenic solution (10 ppm As):** Dissolve 0.330 g of arsenic trioxide in 5 mL of 2M sodium hydroxide, and dilute to 250.0 mL with water. Dilute 1 volume of this stock solution to 100 volumes with water immediately before use.
- **Part B: Assembly and Limit Test Run**
 - **Standard System Setup:** To a clean Gutzeit bottle, add 1 g of potassium iodide AsT and 10 g of zinc dust AsT. Add 10 mL of stannated HCl AsT and exactly 1 mL of dilute standard arsenic solution. Add 50 mL of distilled water to dilute the mixture. Pack the lower section of the apparatus glass tube firmly with lead acetate cotton wool. Sandwich a fresh sheet of mercuric chloride test paper securely between the upper glass flanges. Attach the glass tube assembly immediately to the bottle bung and place the setup on a warm hot plate/surface for 40 to 45 minutes.
 - **Test Sample Setup (Ammonium Chloride):** To a clean Gutzeit bottle, add 1 g of potassium iodide AsT and 10 g of zinc dust AsT. Add 10 mL of stannated HCl AsT and 1 mL of your prepared test solution (2.5 g of ammonium chloride dissolved in 50 mL of water). Ensure the total liquid volume is comparable by verifying the sample dilution. Pack the lower section of the apparatus glass tube firmly with lead acetate cotton wool. Sandwich a fresh sheet of mercuric chloride test paper securely between the upper glass flanges. Attach the glass tube assembly immediately to the bottle bung and place the setup on a warm hot plate/surface for 40 to 45 minutes.

Observations:

- **Test Reaction Duration:** 40–45 minutes.
- **Color Spectrum of the Stain:** Yellow to Brownish-Yellow.

- **Character of Standard Stain:** A distinct yellow stain of reference intensity is produced on the reference mercuric chloride paper.
- **Character of Test Stain:** _____

Observation Table:

Setup Container	Visual Profile of Stain on Mercuric Chloride Paper	Inference
Standard Stain		Reference standard color intensity. Establishes reference threshold limit (max. 4 ppm).
Test Sample Stain		Shows level of arsenic impurities in the sample.

Calculations and Evaluation:

- Official Permissible Pharmacopoeial Limit for Arsenic in Ammonium Chloride: Not more than 4 ppm.
- Visual Analysis: Compare the test paper side-by-side with the standard paper in bright natural daylight.

Result: The yellow stain produced on the mercuric chloride paper by the test solution is (more intense / less intense) than the stain produced by the standard arsenic solution. Consequently, the concentration of arsenic impurity in the given Ammonium Chloride sample is (within / exceeding) the official limit of 4 ppm.

Conclusion: The given sample of Ammonium Chloride (passes / fails) the limit test for Arsenic as per official pharmacopoeial guidelines.

Reference:

1. Indian Pharmacopoeia (IP), 2022.

Experiment No. 6

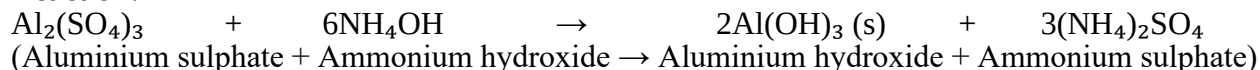
Aim: To prepare and submit a sample of Aluminium Hydroxide $[\text{Al}(\text{OH})_3]$.

Practical Significance: The preparation of aluminium hydroxide is an essential process for creating compounds used in pharmaceutical and industrial applications. It serves as a primary ingredient in antacids to treat heartburn and indigestion. In industrial settings, it is used as a mordant in dyeing fabrics and as a flocculant in water purification to trap impurities.

Understanding the precipitation process helps ensure the correct quality and safety of pharmaceutical preparations. Proper washing and drying techniques are critical to avoid contamination from unreacted reagents like sulphate ions.

Relevant Theoretical Background: Aluminium hydroxide is prepared by the reaction of a soluble aluminium salt (like aluminium sulphate) with an aqueous base (ammonia solution). Unlike other salts that require crystallization, aluminium hydroxide forms a gelatinous white precipitate immediately upon mixing. The reaction is a double displacement/neutralization process where the hydroxide ions replace the sulphate ions to form the insoluble base.

Reaction:



Requirements:

- (a) Apparatus: Beakers (100 mL and 250 mL), Glass rod, Funnel, Filter paper, Measuring cylinder, Wash bottle.
- (b) Chemicals: Aluminium sulphate $[\text{Al}_2(\text{SO}_4)_3]$, Ammonia solution (NH_4OH) (Liquor ammonia), and Distilled water.

Procedure:

1. **Preparation of Salt Solution:** Weigh accurately about 5 g of aluminium sulphate and dissolve it in approximately 50 mL of distilled water in a beaker. Stir well with a glass rod until clear.
2. **Precipitation:** Slowly add ammonia solution (NH_4OH) to the aluminium sulphate solution with constant stirring. A white, jelly-like precipitate will form. Continue adding ammonia until the smell of ammonia persists, indicating the reaction is complete.
3. **Filtration and Washing:** Filter the precipitate using a funnel and filter paper. Wash the precipitate on the filter paper multiple times with hot distilled water to remove sulphate ions or unreacted ammonia.
4. **Drying:** Dry the precipitate by pressing it gently between folds of filter paper or by placing it in an oven at a low temperature.

Observations:

Parameter	Preparation of $\text{Al}(\text{OH})_3$
Initial Appearance	Clear solution
Precipitating Agent	Ammonia solution
Colour of Precipitate	White
Consistency	Gelatinous/Jelly-like

Observation Table:

Sr. No.	Weight of Empty Filter Paper (g)	Weight of Paper + Dry Product (g)	Practical Yield (g)
1.			

Calculations:

- Theoretical Yield:** 342.15 g of $\text{Al}_2(\text{SO}_4)_3$ produces 155.96 g of $\text{Al}(\text{OH})_3$. Therefore, 5 g of $\text{Al}_2(\text{SO}_4)_3$ produces _____ g of $\text{Al}(\text{OH})_3$.
- Percentage Yield:** % Yield = (Practical yield / Theoretical yield) $\times$ 100 = %

Result: Aluminium hydroxide was prepared and submitted.

- Description:** White gelatinous powder.
- Percentage Yield:** The percentage yield was found to be _____%.

Conclusion: The preparation of aluminium hydroxide was successfully performed, and the product obtained meets the descriptive standards of a gelatinous white precipitate.

Reference:

1. Indian Pharmacopoeia, 2022.

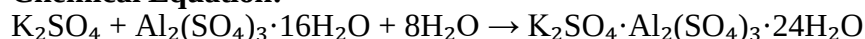
Experiment No. 7

Aim: To prepare and submit potash alum $[K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O]$.

Practical Significance: Potash alum is a significant pharmaceutical and industrial compound used for its astringent and antiseptic properties. It is a double salt containing univalent and trivalent metal salts crystallized with 24 moles of water. Understanding the crystallization process is vital for ensuring the purity and quality of pharmaceutical ingredients. Accurate preparation and yield calculations help in assessing the efficiency of the synthesis in a laboratory or industrial setting.

Relevant Theoretical Background: Alum is a double salt consisting of a univalent metal salt and a trivalent metal salt with 24 moles of water per mole. It is prepared by reacting Aluminium sulphate and Potassium sulphate in stoichiometric proportions followed by crystallization. Unlike single salts, double salts exist only in the solid state and dissociate into their constituent ions when dissolved in water.

Chemical Equation:



(Potassium sulphate + Aluminium sulphate + Water $\rightarrow$ Potassium aluminium sulphate tetracosahydrate)

Requirements:

- **(a) Apparatus:** Beaker, Measuring cylinder, Glass rod, China dish, Funnel, Filter paper.
- **(b) Chemicals:** Aluminium sulphate, Potassium sulphate, Distilled water.

Procedure:

1. Weigh accurately 1.25 g of potassium sulphate and 4.25 g of aluminium sulphate.
2. Dissolve them in a minimum quantity of distilled water in two separate beakers.
3. Heat both solutions simultaneously.
4. Add the hot potassium sulphate solution to the hot aluminium sulphate solution with constant stirring.
5. Evaporate the resulting solution in a china dish until the volume is reduced to 3/4th of its original volume.
6. Set the solution aside undisturbed for crystallization.

7. Filter the crystals, dry them, and record the yield.

Calculations:

1. Theoretical Yield: 2.5 g of K_2SO_4 produces 13.57 g of Potash Alum. Therefore, 5g of $Al_2(SO_4)_3$ produces _____ g of Potash Alum.
2. Percentage Yield: % Yield = (Practical yield / Theoretical yield) $\times$ 100 = %

Result: Potash alum was prepared and submitted.

- **Description:** Colorless, transparent octahedral crystals.
- **Percentage Yield:** The percentage yield of potash alum was found to be _____%.

Conclusion: Colorless, transparent, and octahedral crystals of potash alum were successfully prepared from an equimolar mixture of potassium sulphate and aluminium sulphate.

- **Reference:**

1. Indian Pharmacopoeia, 2022.

Experiment No. 8

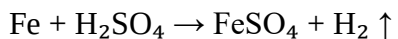
Aim: To prepare and submit Ferrous Sulphate [$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$].

Practical Significance:

- Ferrous sulphate, also known as green vitriol, is a vital medicinal compound used as a hematinic.
- It is essential for the treatment of iron deficiency anemia.
- Due to its efflorescent nature and tendency to oxidize in air to form brown ferric salts, understanding its preparation and storage is crucial for pharmaceutical safety.
- Laboratory preparation through the reaction of iron with acid demonstrates fundamental chemical synthesis and purification.

Relevant Theoretical Background: Ferrous sulphate is prepared by treating iron filings or powder with dilute sulphuric acid. The reaction is an oxidation-reduction process where hydrogen gas is evolved (effervescence). The crystals are bluish-green, crystalline, and efflorescent in air.

Chemical Equation:



(Iron + Sulphuric acid $\rightarrow$ Ferrous sulphate + Hydrogen gas)

Requirements:

- **(a) Apparatus:** Beaker, Measuring cylinder, Glass rod, China dish, Funnel, Water bath.
- **(b) Chemicals:** Iron filings, Dilute sulphuric acid, Distilled water.

Procedure:

1. Measure 25 mL of dilute sulphuric acid in a beaker and bring it to a boil.
2. Add iron filings in small portions with gradual heating.
3. Continue addition and stirring until the effervescence of H_2 gas ceases, indicating the reaction is complete.
4. Filter the hot solution to remove any unreacted iron.
5. Allow the filtrate to cool slowly to obtain crystals.
6. Separate the crystals, dry them, and recrystallize if necessary using distilled water.

Result: Ferrous sulphate was prepared and submitted.

- **Description:** Bluish-green crystalline powder.
- **Percentage Yield:** The percentage yield of ferrous sulphate was found to be _____%.

Reference:

1. Indian Pharmacopoeia, 2022.

Experiment No. 9

Aim: To prepare and submit Magnesium Sulphate from Magnesium Oxide.

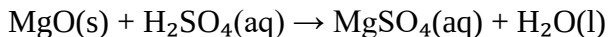
Practical Significance: The preparation and crystallization of magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) serves as a core analytical methodology for synthesizing essential pharmaceutical inorganic compounds. Commonly known as Epsom salt, magnesium sulphate is a versatile therapeutic agent widely utilized as an osmotic laxative, anticonvulsant, and electrolyte replenisher. Synthesizing this compound from its basic oxide provides fundamental insights into salt formation via neutralization reactions, solubility limits, and isolation techniques. Mastering this preparation is vital for ensuring quality control, determining precise percentage yields, and evaluating the structural integrity of crystalline hydrates intended for compounding and official medicinal formulations.

Relevant Theoretical Background: The preparation of magnesium sulphate is classified as a salt-generation synthesis based on a classic acid-base neutralization reaction. Magnesium oxide (a basic metal oxide) is reacted with dilute sulfuric acid to form a soluble salt, magnesium

sulphate, and water. Because magnesium oxide is added in slight excess, any unreacted solid oxide is separated out via filtration while hot, leaving behind a clear filtrate. Slow evaporation of this concentrated solution yields crystals wrapped in a specific geometry containing seven molecules of water of crystallization, yielding magnesium sulphate heptahydrate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$), famously known as Epsom salt.

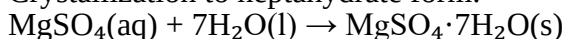
Reaction Equations:

1. Neutralization reaction:



(Magnesium oxide + Sulphuric acid $\rightarrow$ Magnesium sulphate + Water)

2. Crystallization to heptahydrate form:



(Magnesium sulphate + Water $\rightarrow$ Magnesium sulphate heptahydrate)

Requirements:

- (a) Apparatus: Beaker (100 mL), Measuring cylinder, Glass rod, China dish (Evaporating dish), Funnel, Filter paper, Tripod stand, Wire gauze, Water bath, Bunsen burner.
- (b) Chemicals: Magnesium oxide (MgO), Dilute Sulfuric acid (1 M H_2SO_4), Distilled water.

Procedure:

1. **Measurement of Reactants:** Weigh out approximately 1.5 g of magnesium oxide powder using a digital balance. Using a clean measuring cylinder, measure exactly 25 mL of 1 M sulfuric acid and transfer it into a beaker.
2. **Thermal Dissolution:** Warm the sulfuric acid gently on a wire gauze to about 60°C . While stirring continuously with a glass rod, add the magnesium oxide powder a little at a time. Ensure that each small portion dissolves completely before adding the next.
3. **Saturation Observation:** Continue adding the oxide until about 1.0 g to 1.2 g has been introduced. When no more solid dissolves and a cloudy suspension persists in the beaker, the acid is completely neutralized.
4. **Filtration:** Filter the hot mixture through a filter paper nestled in a glass funnel into an evaporating china dish to remove the excess unreacted magnesium oxide.
5. **Concentration and Crystallization:** Place the clear filtrate over a hot water bath regulated at approximately 60°C . Evaporate the solvent slowly until the crystallization point is reached (indicated by crystal crust formation on a glass rod dipped into the solution).
6. **Isolation:** Allow the concentrated solution to cool down naturally and undisturbed. Colorless, needle-like crystals of magnesium sulphate heptahydrate will separate. Filter the crystals, dry them gently between folds of filter paper, and submit the sample.

Precaution: Avoid heating the dry crystals directly over an open flame, as overheating will drive off the water of crystallization, converting the heptahydrate into an amorphous, anhydrous white powder.

Medicinal Utility: Primarily administered as an osmotic Laxative (purgative) to treat acute constipation, and topically as an anti-inflammatory soaking agent.

Observations:

- (a) Color of Prepared Crystals: Colorless / Crystalline white.
- (b) Shape of Prepared Crystals: Orthorhombic / Needle-shaped structures.
- (c) Weight of Empty Evaporating Dish (W_1): _____ g.

(d) Weight of Dish + Dried Crystals (W_2): _____ g.

(e) Practical Yield of Product Obtained ($W_2 - W_1$): $m =$ _____ g

Stoichiometric Estimation Table:

Compound Name	Molecular Formula	Molar Mass (g/mol)	Mass Taken / Expected
Magnesium oxide (Reactant)	MgO	40.30 g/mol	1.5 g (Added in excess)
Magnesium sulphate heptahydrate	MgSO ₄ ·7H ₂ O	246.47 g/mol	Theoretical Yield (Yt)

Calculations:

1. Determination of Theoretical Yield (Yt):

The theoretical calculation is based on the limiting reactant. Here, 25 mL of 1 M H₂SO₄ acts as the limiting reactant because magnesium oxide is added in excess.

(a) Number of moles of H₂SO₄ = Molarity × Volume in litres = 1 M × 0.025 L = 0.025 moles. (b) Based on the chemical equation, 1 mole of H₂SO₄ yields 1 mole of MgSO₄·7H₂O.

(c) Therefore, 0.025 moles of H₂SO₄ will produce 0.025 moles of MgSO₄·7H₂O. Theoretical yield (Yt) = Moles × Molar mass of product Yt = 0.025 mol × 246.47 g/mol = 6.162 g

3. Calculation of Percentage Yield:

Let the practical yield obtained in lab be m grams.

Percentage yield = (Practical yield (m) / Theoretical yield (Yt)) × 100

Percentage yield = (m / 6.162) × 100 = % w/w

Result: Magnesium sulphate was successfully prepared and submitted.

- Practical yield obtained = _____ g
- Theoretical yield expected = 6.162 g
- Percentage yield = _____ % w/w

Conclusion: The synthesis confirms that the action of a mineral acid (H₂SO₄) on a basic metal oxide (MgO) yields its corresponding hydrated double-cation matrix. The prepared sample displays physical characteristics identical to official Epsom salt configurations.

References:

1. Indian Pharmacopoeia, 2022.
2. Vogel's Textbook of Practical Organic and Inorganic Chemistry.

Experiment No. 10

Aim: To determine the swelling power of Bentonite.

Practical Significance: Determining the swelling power of Bentonite is an important quality control test in pharmaceutical and industrial applications. Bentonite is widely used as a stabilizing, suspending, and gelling agent. Its ability to swell significantly in water dictates its viscosity-building properties and its efficacy in formulations like suspensions, ointments, and pastes. This test ensures the sample complies with pharmacopoeial standards and maintains batch-to-batch consistency.

Relevant Theoretical Background: Bentonite is a naturally occurring colloidal hydrated aluminum silicate. It presents as a very fine, odorless, pale buff or cream-colored, hygroscopic powder with a slightly earthy taste. It is completely insoluble in organic solvents and does not swell within them. However, when added to water, it exhibits a distinct swelling behavior, expanding to approximately 12 times its original volume. An aqueous suspension of Bentonite typically exhibits an alkaline pH between 9.0 and 10.0, and its average particle size is targeted around 44 microns.

Requirements:

- **(a) Apparatus:** Graduated measuring cylinder (100 mL), weighing balance, stopwatch.
- **(b) Chemicals:** Bentonite, Sodium lauryl sulphate, Distilled water.

Procedure:

1. Prepare 100 mL of a 1% w/v sodium lauryl sulphate solution and place it into a 100 mL graduated measuring cylinder.
2. Weigh exactly 2 g of the Bentonite sample and divide it into 20 equal portions (approx. 0.1 g each).
3. Add one portion of the Bentonite powder to the cylinder. Allow the portion to settle completely before adding the next.
4. Repeat this addition process at strict 2-minute intervals for all 20 portions.
5. Once the final portion is added, allow the cylinder to stand undisturbed for 2 hours.
6. Observe and record the apparent volume of the sediment gathered at the bottom of the cylinder.

Precaution: Ensure each portion settles before adding the next to avoid clump formation, which artificially alters the true sedimentation value.

Pharmacopoeial Limit: The apparent volume of the sediment at the bottom of the cylinder should not be less than 24 mL.

Observations:

- **(a) Medium Used:** 100 mL of 1% sodium lauryl sulphate solution.
- **(b) Total Weight of Sample:** 2.0 grams.
- **(c) Interval Time:** 2 minutes.
- **(d) Settling Duration:** 2 hours.

Observation Table:

Trial No.	Weight of Bentonite (g)	Volume of Sediment Observed (mL)
1	2.0 g	
2	2.0 g	
3	2.0 g	
Mean		mL

Calculations: Swelling power = Mean volume of sediment observed (mL).

Result: The swelling power of Bentonite was found to be _____mL.

Conclusion: The given sample of Bentonite passes / fails the swelling power test as per the official pharmacopoeial limits.

Reference:

1. Indian Pharmacopoeia, 2022.

Experiment No. 11

Aim: To determine the acid neutralizing capacity of Aluminum Hydroxide Gel.

Practical Significance: Aluminium hydroxide gel is a well-known antacid used to treat symptoms of hyperacidity, peptic ulcers, and acid indigestion. Evaluating its acid neutralizing capacity (ANC) ensures that the formulation can effectively counteract gastric acid in patients. Testing this property in a laboratory framework confirms the therapeutic strength, stability, and dose effectiveness of the batch before it is cleared for medicinal administration.

Relevant Theoretical Background: Aluminium hydroxide gel is an aqueous, white, viscous suspension consisting of hydrated aluminium oxide along with varying fractions of basic aluminium carbonate. Its primary therapeutic action relies on its acid-consuming capability. Because the direct titration of a weak base suspension can yield a slow equilibrium endpoint, a back-titration method is utilized. An excess volume of standard hydrochloric acid (HCl) is mixed with the gel to completely dissolve and neutralize it. The remaining unreacted residual acid is subsequently back-titrated against a standard sodium hydroxide (NaOH) volumetric solution.

Requirements:

- **(a) Apparatus:** Conical flask, Burette, Burette stand, Volumetric measuring cylinder, Water bath (37°C).
- **(b) Chemicals:** Aluminium hydroxide gel, 0.1 N Hydrochloric acid (HCl), 0.1 N Sodium hydroxide (NaOH), Bromophenol blue indicator.

Procedure:

1. Pipette or transfer exactly 1.5 mL of the aluminium hydroxide gel sample into a clean conical flask.
2. Accurately add 50 mL of 0.1 N hydrochloric acid into the flask and mix thoroughly.
3. Place the flask into a temperature-controlled water bath held at (37°C) and shake continuously or periodically for exactly 1 hour.
4. Cool the mixture to room temperature and add a few drops of bromophenol blue indicator solution.
5. Titrate the excess, unreacted acid with 0.1 N sodium hydroxide solution until a stable color transition occurs.

Note: 1 g of the gel should consume not less than 12.5 mL and not more than 25.0 mL of 0.1 N HCl.

Observations:

- (a) **Volume of Gel taken:** 1.5 mL
- (b) **Volume of 0.1 N HCl added:** 50.0 mL
- (c) **Titrant:** 0.1 N sodium hydroxide (NaOH)
- (d) **Indicator:** Bromophenol blue
- (e) **End point:** Yellow to Blue-Violet

Observation Table:

Sr. No.	Volume of Gel (mL)	Volume of 0.1 N HCl Added (mL)	Burette Reading of 0.1 N NaOH (mL)	Mean Burette Reading (V_2 mL)
1.	1.5	50.0		

Calculations:

Let the mean burette reading of 0.1 N NaOH = V_2 mL

Volume of 0.1 N HCl consumed by the gel (V_1) is calculated as:

$$V_1 = 50.0 \text{ mL} - V_2 \text{ mL}$$

Result: The amount of acid consumed by the aluminium hydroxide gel sample was found to be _____ mL.

Conclusion: The acid neutralizing capacity of the given aluminium hydroxide gel sample passes / fails the specified official requirements.

References:

1. Indian Pharmacopoeia, 2022.
2. Vogel's Textbook of Quantitative Chemical Analysis.

Experiment No. 12

Aim: To perform a test for purity of potassium iodide by examining the presence of iodate in the sample.

Practical Significance: Potassium Iodide (KI) is extensively utilized in pharmaceutical formulations, nutritional supplements, and as an analytical reagent. Iodate (IO_3^-) is a prominent synthesis and degradation impurity that can compromise the purity of the compound and alter its physiological actions. Conducting this limit/purity test confirms that the trace limits of dangerous oxidizing contaminants remain within the safety criteria mandated by standard Pharmacopoeias.

Relevant Theoretical Background: Iodate ions present as impurities in Potassium Iodide samples can be selectively monitored under acidic conditions. In the presence of dilute sulfuric acid (H_2SO_4), any trace iodate (IO_3^-) reacts with the iodide (I^-) ions to undergo a symproportionation reaction, liberating free elemental iodine (I_2). The liberated iodine instantly forms an intense blue complex with starch. By running a concurrent standard solution matching the 4 ppm threshold, a side-by-side color comparison reveals if the test sample contains an unallowable level of iodate impurity.

Requirements:

- **(a) Apparatus:** Test tubes, Measuring jar, Color comparison tubes (Nessler cylinders), Volumetric pipette.
- **(b) Chemicals:** Potassium iodide (KI), Dilute sulfuric acid (H_2SO_4), 1 N H_2SO_4 , Iodide-free starch solution, Concentrated ammonia, Standard iodate solution.

Procedure:

- Test-1 (Qualitative Screening): Dissolve 0.5 g of the potassium iodide sample in 10 mL of carbon dioxide-free water. Add 0.15 mL of dilute H_2SO_4 followed by 1 drop of iodide-free starch solution. Observe for 2 minutes. If no blue color develops, it signifies the basic absence of immediate gross iodate contaminations.
- Test-2 (Quantitative Limit Match): Dissolve 1.1 g of the potassium iodide sample in a mixture of sufficient ammonia and carbon dioxide-free water to make exactly 10 mL. Transfer this solution to a clear color comparison tube. Add 1 mL of starch solution and 0.25 mL of 1 N H_2SO_4 . Mix the components thoroughly.
- Preparation of Standard Solution: In a separate matching color comparison tube, combine 100 mg of pure potassium iodide, 1 mL of the standard iodate solution, 1 mL of starch solution, and 0.25 mL of 1 N H_2SO_4 . Dilute to match the test volume if necessary. Compare the blue color intensity generated in Test-2 directly against this standard setup.

Precaution: The water used must be strictly carbon dioxide-free to prevent spontaneous, air-catalyzed oxidation of iodide to iodine which yields false-positive blue results.

Pharmacopoeial Limit: The blue color intensity produced in the test solution must not exceed the color produced in the standard solution (4 ppm).

Observations:

- **(a) Test-1 Result (After 2 minutes):** Colorless / Blue color observed.
- **(b) Test-2 (Sample Tube) Color Profile:** _____
- **(c) Standard Tube Color Profile:** Intense light blue (4 ppm benchmark).
- **(d) Visual Comparison:** Test tube color is less intense / more intense than the standard tube.

Observation Table:

Sr. No.	System Cylinder	Components Added	Color Intensity
1	Test Solution (Sample)	1.1 g KI + Ammonia + Water + Starch + 1N H_2SO_4	
2	Standard Solution	100 mg KI + 1 mL Standard iodate + Starch + 1N H_2SO_4	

Sr. No.	System Cylinder	Components Added	Color Intensity

Calculations: Visual estimation of compliance. Color intensity of test solution vs Color intensity of standard solution (4 ppm).

Result: The test for purity for the given sample of potassium iodide (KI) was performed, and the presence of iodate ions was found to be (passed / failed) as the color intensity (did not exceed / exceeded) the standard solution.

Conclusion: The given sample of potassium iodide complies / does not comply with the purity test requirements for iodate impurities as per the standard lab guidelines.

References:

1. Indian Pharmacopoeia, 2022.
2. Vogel's Textbook of Quantitative Chemical Analysis.

Experiment No. 13

Aim: To perform an assay of Ammonium Chloride by acid-base titration.

Practical Significance: An assay of ammonium chloride by acid-base titration is a significant analytical tool to determine its purity, strength, and quality for pharmaceutical, industrial, and laboratory use. Ammonium chloride is used in expectorants, diuretics, and acidifying agents. Accurate assay ensures the correct dose and safety of pharmaceutical preparations. The assay measures the actual amount of ammonium chloride present in the sample and checks whether it meets official standards or not. Deviations in assay values may indicate contamination, moisture absorption, or decomposition during storage and handling. Acid-base titration helps to confirm the amount of ammonium ion released during the reaction, ensuring analytical accuracy. The official standards given in pharmacopoeias require assay testing in analytical laboratories before a batch is approved for use or sale.

Relevant Theoretical Background: The assay of ammonium chloride is carried out by aqueous acid-base titration which is based on the neutralization reaction. It is a direct type of titration. Ammonium chloride reacts with previously neutralized formaldehyde solution (formaldehyde reacts with ammonium) to form hexamethylenetetramine, liberating hydrochloric acid. The liberated acid is titrated with standard sodium hydroxide solution using phenolphthalein indicator.

Reactions:

1. Formaldehyde reacts with ammonium ions:
$$4\text{NH}_4\text{Cl} + 6\text{HCHO} \rightarrow (\text{CH}_2)_6\text{N}_4 + 4\text{HCl} + 6\text{H}_2\text{O}$$

(Ammonium chloride + Formaldehyde → Hexamethylenetetramine + Hydrochloric acid + Water)
2. The liberated HCl is titrated with NaOH:
$$\text{HCl} + \text{NaOH} \rightarrow \text{NaCl} + \text{H}_2\text{O}$$

Requirements:

- **(a) Apparatus:** Volumetric flask, Burette, Pipette, Conical flask, Burette stand, Water bath, Beaker, Funnel etc.
- **(b) Chemicals:** Sodium hydroxide, Potassium hydrogen phthalate (KHP), Phenolphthalein, Ammonium chloride, Formaldehyde etc.

Procedure:

1. **Preparation of standard 0.1 M NaOH solution:** Dissolve 4 g of sodium hydroxide in sufficient carbon dioxide-free water to produce 1000 mL 0.1 M NaOH solution.
2. **Standardization of prepared NaOH:** Accurately weigh about 0.5 g of the dried potassium hydrogen phthalate (previously powdered and dried at 120°C for 2 hours).

Dissolve the potassium hydrogen phthalate in 75 mL of carbon dioxide-free distilled water. Add 0.1 mL of phenolphthalein indicator and titrate with NaOH until a permanent pink colour is obtained. The endpoint is reached when a slight pink colour persists for at least 30 seconds. Factor: 0.2042 g of KHP is equivalent to 1 mL of 1 M NaOH.

3. **Assay of Ammonium Chloride:** Weigh accurately about 0.1 g of ammonium chloride. Dissolve in 20 mL of water. Add a mixture of 5 mL of formaldehyde solution and 20 mL of water. Add few drops of phenolphthalein indicator. After few minutes, titrate slowly with 0.1 M sodium hydroxide until faint pink colour appears. Factor: 0.05349 g of NH_4Cl is equivalent to 1 mL of 1 M NaOH.

Precaution: Formaldehyde solution should be neutralized before use because it may contain formic acid, which can affect the titration result.

Pharmacopoeial Limit: Ammonium chloride should contain 99.0% to 100.5% of NH_4Cl on a dried basis.

Observation Tables:

1. For Standardization of NaOH:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

2. For Assay of Ammonium Chloride:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

Calculations:**1. For Standardization of NaOH:**

Factor: 204.23 g of KHP is equivalent to 1000 mL of 1 M NaOH.

0.2042 g of KHP is equivalent to 1 mL of 1 M NaOH.

0.02042 g of KHP is equivalent to 1 mL of 0.1 M NaOH.

Therefore, 0.5 g of KHP is equivalent to mL of x M NaOH

$$x \text{ M NaOH} = (0.5 \times 1 \times 0.1) / (0.02042 \times \dots)$$

$$x \text{ M NaOH} = \dots \text{ M}$$

2. For Assay of Ammonium Chloride:

Factor: 53.49 g of ammonium chloride is equivalent to 1000 mL of 1 M NaOH.

0.05349 g of ammonium chloride is equivalent to 1 mL of 1 M NaOH.

0.005349 g of ammonium chloride is equivalent to 1 mL of 0.1 M NaOH.

Therefore, x g of ammonium chloride is equivalent to mL of x M NaOH.

$$x \text{ g} = (0.005349 \times \dots \times \dots) / (1 \times 0.1)$$

$$x \text{ g} = \dots \text{ g}$$

Percent Purity:

$$0.1 \text{ g ammonium chloride} = 100\%$$

$$x \text{ g ammonium chloride} = x \%$$

$$x \% = (x \text{ g} \times 100) / 0.1$$

$$x \% = \dots \dots \dots \% \text{ w/w}$$

Result: The percent purity of ammonium chloride by acid-base titration as per Indian Pharmacopoeia was found to be ____% w/w.

Conclusion: The assay of ammonium chloride by acid-base titration passes / fails as per Indian Pharmacopoeia.

Reference:

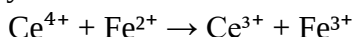
1. Indian Pharmacopoeia, 2018.

Experiment No. 14

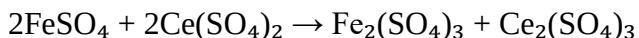
Aim: To perform an assay of Ferrous Sulphate by Cerimetry.

Practical Significance: The assay of ferrous sulphate by cerimetry is practically significant because it ensures the correct potency, purity, stability, and safety of ferrous sulphate in pharmaceutical and industrial applications through an accurate oxidation-reduction titration method. Official pharmacopeias include cerimetry for assay of iron preparations because of its reliability and precision and it helps to determine the purity and strength of ferrous sulphate as a raw material. This assay detects oxidation of Fe^{2+} to Fe^{3+} during storage because it is widely used as an iron supplement for treating anaemia. The assay ensures tablets, capsules, or syrups contain the correct iron content required for therapeutic effectiveness. This assay also helps to monitor degradation and shelf life of pharmaceutical preparation.

Relevant Theoretical Background: It is based on redox titration where ferrous sulphate is a reducing agent (Fe^{2+}) and is oxidized by a powerful oxidizing agent, ceric ammonium sulphate (Ce^{4+}), in the presence of dilute sulphuric acid. The titration uses ferroin (ferrous-1,10-phenanthroline) as an indicator. At the equivalence point, the slight excess of Ce^{4+} oxidizes the ferroin indicator, causing a color change from red to light green. In cerimetry, Fe^{2+} is oxidized to Fe^{3+} by Ce^{4+} in acidic medium where ceric ion act as a strong oxidizing agent.



(Ferrous ion (Fe^{2+}) is oxidized to ferric ion (Fe^{3+}). Ceric ion (Ce^{4+}) is reduced to cerous ion (Ce^{3+}).)



The reaction is carried out in acidic medium, usually with sulphuric acid.

Requirements:

- **(a) Apparatus:** Volumetric flask, Burette, Pipette, Conical flask, Burette stand, Water bath, Beaker, Funnel etc.
- **(b) Chemicals:** Arsenic trioxide, Sodium hydroxide, Osmic acid, Sodium hydrogen carbonate, Ferrous sulphate, Sulphuric acid, Ceric ammonium sulphate, Ferroin.

Procedure:

1. **Preparation of standard 0.1 M Ceric ammonium sulphate solution:** Dissolve 63.26 g of ceric ammonium sulphate in a mixture of 30 mL sulfuric acid and 500 mL water, using gentle heat. Cool the solution, filter if turbid, and dilute to 1000 mL with water.
2. **Standardization of prepared Ceric ammonium sulphate:** Prepare standard solution of sodium arsenite (0.025 M) by dissolving 1.2365 g of arsenic trioxide in a mixture of NaOH (5 M, 5 mL) and 5 mL water in a graduated flask and dilute to 200 mL with water. Add 2M HCl until the solution becomes neutral. Dissolve 0.5 g sodium hydrogen carbonate in the solution and dilute with water to 250 mL. Titrate the ceric ammonium

sulphate solution (approx. 0.1 M, 25 mL) with 0.025 M sodium arsenite in the presence of osmic acid solution (0.15 mL) and ferroin sulphate solution as an indicator. *Factor: 0.004946 g of arsenic trioxide is equivalent to 1 mL of 0.1 M ceric ammonium sulphate.*

3. **Assay of Ferrous sulphate:** Accurately weigh about 0.5 g of ferrous sulphate (dried) and place it into a conical flask. Dissolve the sample in a mixture of 20 mL of 1 M sulphuric acid and 30 mL of freshly boiled and cooled water. Add 0.1 mL of Ferroin solution as an indicator. Titrate immediately with 0.1 M ceric ammonium sulphate until the red color changes to a green color. *Precaution: Ferrous sulphate is readily oxidized to ferric sulphate by air. Prepare the sample just before titration and handle quickly. Factor: 0.03291 g of ferrous sulphate is equivalent to 1 mL of 0.1 M ceric ammonium sulphate.*

Pharmacopoeial Limit: It contains not less than 98.0% and not more than 105.0% of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.

Observation Tables:

1. For Standardization of Ceric ammonium sulphate:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

1. For Assay of Ferrous sulphate:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

Calculations:

1. For Standardization of Ceric ammonium sulphate:

Factor: 0.004946 g of arsenic trioxide is equivalent to 1 mL of 0.1 M ceric ammonium sulphate.

1.2365 g of arsenic trioxide is equivalent to mL of x M ceric ammonium sulphate.

$$x \text{ M ceric ammonium sulphate} = (1.2365 \times 1 \times 0.1) / (0.004946 \times \text{.....})$$

$$x \text{ M ceric ammonium sulphate} = \text{..... M.}$$

2. For Assay of Ferrous sulphate: Factor:

0.03291 g of ferrous sulphate is equivalent to 1 mL of 0.1 M ceric ammonium sulphate.

Therefore x g of ferrous sulphate is equivalent to mL of x M ceric ammonium sulphate.

$$x \text{ g} = (0.03291 \times \text{.....} \times \text{.....}) / (1 \times 0.1)$$

$$x \text{ g} = \text{..... g}$$

3. Calculation of Percent Purity:

$$0.5 \text{ g ferrous sulphate} = 100\%$$

$$x \text{ g ferrous sulphate} = x \%$$

$$x \% = (x \text{ g} \times 100) / 0.5$$

$$x \% = \text{.....} \% \text{ w/w}$$

Result: The percent purity of ferrous sulphate by cerimetric titration as per Indian Pharmacopoeia was found to be ___% w/w.

Conclusion: The assay of ferrous sulphate by cerimetric titration passes / fails as per Indian Pharmacopoeia.

References:

1. Indian Pharmacopoeia, 2018.
2. Practical Pharmaceutical Chemistry, Part I (Beckett & Stenlake).

Experiment No. 15

Aim: To perform an assay of Copper Sulphate by Iodometry.

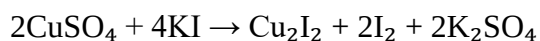
Practical Significance: The assay of Copper sulphate by iodometry is a vital quantitative redox titration used to determine the exact purity and concentration of copper in a given sample. It serves as a key quality control standard for validating pharmaceutical, agricultural, and industrial raw materials. The assay helps determine the percentage purity of copper sulphate pentahydrate in pharmaceutical and analytical samples. This ensures the substance complies with official standards. Iodometry is highly accurate because liberated iodine can be measured precisely with sodium thiosulphate solution.

Relevant Theoretical Background: The assay of copper sulphate by iodometry is a precise, indirect redox titration. Copper sulphate reacts with potassium iodide to form cupric iodide, which is highly unstable and rapidly decomposes into cuprous iodide (a white/yellow precipitate) and free iodine. The liberated free iodine, which is chemically equivalent to the amount of copper present, is titrated against standard sodium thiosulphate. Near the titration endpoint, starch solution is added to form a deep blue complex. Titration continues dropwise until the blue color completely disappears.

Reactions:

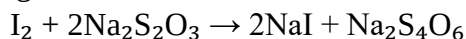
1. Liberation of Iodine:

Copper sulphate reacts with potassium iodide in an acidic medium (acetic acid) to liberate iodine:



2. Titration of Liberated Iodine:

The liberated iodine is titrated with sodium thiosulphate. The iodine acts as an oxidizing agent and converts to iodide ions, while thiosulphate is oxidized to tetrathionate:



Requirements:

(a) Apparatus: Volumetric flask, Burette, Pipette, Conical flask, Burette stand, Water bath, Beaker, Funnel etc.

(b) Chemicals: Copper sulphate, Sodium thiosulphate, Potassium dichromate, Sodium carbonate, Potassium iodide, Hydrochloric acid, Starch etc.

Procedure:

1. **Preparation of standard 0.1 M sodium thiosulphate solution:** Dissolve 26 g sodium thiosulphate and 0.20 g sodium carbonate in water and make up the volume up to 1000 mL with recently boiled and cooled purified water.
2. **Standardization of prepared sodium thiosulphate solution:** Weigh exactly 0.21 g of potassium dichromate and dissolve it in 100 mL of water in a glass stoppered conical flask. Add 2 g potassium iodide, and 5 mL hydrochloric acid quickly. Immediately stopper the flask, swirl gently, and let it stand in the dark for exactly 10 minutes. Titrate liberated

iodine with sodium thiosulphate until yellowish-green. Add 3 mL starch indicator (turns deep blue). Continue titrating drop-by-drop until blue color disappears. *Factor: Each mL of 0.1 M sodium thiosulphate is equivalent to 0.004 g of potassium dichromate.*

3. **Assay of copper sulphate:** Accurately weigh about 0.65 g of copper sulphate into an iodine flask. Dissolve in 50 mL distilled water. Add 4 mL of 6 M acetic acid and 3 g potassium iodide. Titrate with standardized 0.1 M sodium thiosulphate until brown color fades to pale yellow. Add 2 g potassium thiocyanate and 3 mL starch solution. Titrate until blue color completely disappears leaving a milky-white/faint-yellow precipitate. *Factor: 1 mL of 0.1 M sodium thiosulphate is equivalent to 0.02497 g of copper sulphate.*

Pharmacopoeial Limit: It should not contain less than 98.5% and not more than 101.0% of CuSO_4 .

Observation Tables:

1. **For Standardization of Sodium thiosulphate solution:**

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

1. **For Assay of Copper sulphate:**

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
2.				
3.				

Calculations:

1. For Standardization of Sodium thiosulphate:

Factor: 0.0049 g of potassium dichromate is equivalent to each mL of 0.1 M sodium thiosulphate.

Therefore, 0.21 g of potassium dichromate is equivalent to mL of x M sodium thiosulphate.

$$x \text{ M} = (0.21 \times 1 \times 0.1) / (0.0049 \times \text{.....})$$

$$x \text{ M} = \text{..... M}$$

2. For Assay of Copper sulphate: Factor:

0.02497 g of copper sulphate is equivalent to 1 mL of 0.1 M sodium thiosulphate.

x g of copper sulphate is equivalent to mL of x M sodium thiosulphate.

$$x \text{ g} = (0.02497 \times \text{.....} \times \text{.....}) / (1 \times 0.1)$$

$$x \text{ g} = \text{..... g}$$

3. Calculation of Percent Purity:

0.65 g copper sulphate = 100%

x g copper sulphate = x %

$x \% = (x \text{ g} \times 100) / 0.65$

x % = % w/w

Result: The percent purity of copper sulphate by iodometry as per Indian Pharmacopoeia was found to be ____% w/w.

Conclusion: The assay of copper sulphate by iodometry passes / fails as per Indian Pharmacopoeia.

Reference:

1. Indian Pharmacopoeia, 2018.

Experiment No. 16

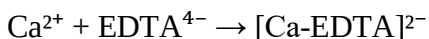
Aim: To perform an assay of Calcium Gluconate by Complexometry.

Practical Significance: Performing an assay of calcium gluconate by complexometry is important for verifying its pharmaceutical purity and ensuring precise dosing for clinical use because calcium gluconate is used to treat critical conditions like severe hypocalcemia and magnesium toxicity. Therefore, exact concentration quantification is a strict safety and regulatory requirement. Complexometric assay is widely used in pharmaceutical industries for routine quality testing of calcium gluconate raw material and formulations to ensure batch-to-batch consistency.

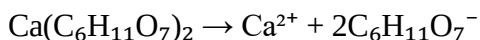
Relevant Theoretical Background: The assay of calcium gluconate is based on

complexometric titration using disodium EDTA (ethylene diaminetetraacetic acid) as titrant. Calcium ions present in calcium gluconate react quantitatively with EDTA in an alkaline medium to form a stable, soluble calcium-EDTA complex. The endpoint is detected using Mordant Black-II / Calcon indicator.

Reactions:

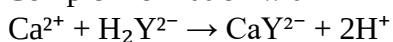


1. Dissociation of Calcium Gluconate:



(Calcium gluconate dissociates in solution to release calcium ions.)

2. Complex formation with EDTA:



where, $\text{H}_2\text{Y}^{2-} = \text{EDTA}$, $\text{CaY}^{2-} = \text{Calcium-EDTA complex}$

Requirements:

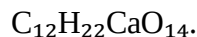
(a) Apparatus: Volumetric flask, Burette, Pipette, Conical flask, Burette stand, Water bath, Beaker, Funnel etc.

(b) Chemicals: Calcium gluconate, Calcon, Disodium edetate, Ammonium chloride, Ammonia, Granulated zinc, HCl, Bromine water, Sodium hydroxide, Mordant black-II, Sodium chloride.

Procedure:

1. **Preparation of 0.05 M disodium edetate solution:** Weigh accurately 18.61 g of disodium edetate and dissolve it in purified water to make up to 1000 mL.
2. **Preparation of Ammonia Buffer Solution (pH 10):** Dissolve 5.4 g Ammonium chloride in 70 mL 5 M ammonia and dilute with water to 100 mL.
3. **Standardization of disodium edetate:** Accurately weigh approximately 0.125 gm granulated zinc, dissolve in a minimum volume of 7 M hydrochloric acid, add 0.2 mL bromine water to ensure oxidation of trace iron impurity to iron (III), which forms a much less stable edetate complex than iron (II). Gently boil to remove excess bromine. Cool, add 2 M sodium hydroxide until almost neutral, dilute to 250 mL and add ammonia buffer pH 10 until the precipitate is just dissolved and then 5 mL in excess. Add a mixture of Mordant Black-II and sodium chloride (1:99, 50 mg) as indicator and titrate with 0.05 M disodium edetate solution until the solution becomes green. Factor: 0.003269 g Zn is equivalent to 1 mL of 0.05 M disodium edetate.
4. **Assay of Calcium Gluconate:** Accurately weigh approximately 0.1 g sample calcium gluconate, dissolve in 3 mL 2 M hydrochloric acid and 10 mL water. Boil for 2 minutes, cool and dilute to 50 mL with water. Titrate with 0.05 M disodium edetate almost to the expected end point. Add 4 mL 10 M NaOH and a mixture of calcon and anhydrous sodium sulphate (1:99, 0.1 g) as indicator and continue the titration until the colour becomes full blue. Factor: 0.02242 g of calcium gluconate is equivalent to 1 mL of 0.05 M disodium edetate.

Pharmacopoeial Limit: Contains not less than 98.0% and not more than 102.0% of



Observation Tables:

1. For Standardization of 0.05 M Disodium Edetate:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

1. For Assay of Calcium Gluconate:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

Calculations:

- For Standardization of 0.05 M Disodium edetate solution:
 Factor: 0.003269 g Zn is equivalent to 1 mL of 0.05 M disodium edetate.
 0.125 g Zn is equivalent to mL of x M disodium edetate.

$$x \text{ M} = (0.125 \times 1 \times 0.05) / (0.003269 \times \dots)$$

$$x \text{ M} = \dots \text{ M}$$
- For Assay of Calcium gluconate: Factor:
 0.02242 g of calcium gluconate is equivalent to 1 mL of 0.05 M disodium edetate.
 x g of calcium gluconate is equivalent to mL of x M disodium edetate.

$$x \text{ g} = (0.02242 \times \dots \times \dots) / (1 \times 0.05)$$

$$x \text{ g} = \dots \text{ g}$$
- Calculation of Percent purity:
 0.1 g calcium gluconate = 100%
 Therefore, x g calcium gluconate = x %

$$x \% = (x \text{ g} \times 100) / 0.1$$

$$x \% = \dots \% \text{ w/w}$$

Result: The percent purity of calcium gluconate by complexometry was found to be _____ %
 _____ w/w.

Conclusion: The assay of calcium gluconate by complexometry passes / fails as per Indian Pharmacopoeia.

References:

- Indian Pharmacopoeia, 2018.
- Practical Pharmaceutical Chemistry, Part I (Beckett & Stenlake).

Experiment No. 17

Aim: To perform an assay of Hydrogen Peroxide by Permanganometry.

Practical Significance: Hydrogen peroxide is used in oxidation and wastewater treatment processes. The assay confirms proper dosing and ensures antiseptic solutions contain the correct concentration. Over-concentrated peroxide can damage tissues; under-concentrated peroxide may be ineffective.

Relevant Theoretical Background: The assay is a redox titration in which hydrogen peroxide is oxidized by potassium permanganate in an acidic medium (H_2SO_4). KMnO_4 acts as a self-indicator. The endpoint is the appearance of a permanent faint pink color.

Reactions:

Reduction half-reaction of permanganate: $\text{MnO}_4^- + 8\text{H}^+ + 5\text{e}^- \rightarrow \text{Mn}^{2+} + 4\text{H}_2\text{O}$

Oxidation half-reaction of hydrogen peroxide: $\text{H}_2\text{O}_2 \rightarrow \text{O}_2 + 2\text{H}^+ + 2\text{e}^-$

Overall balanced reaction: $2\text{MnO}_4^- + 5\text{H}_2\text{O}_2 + 6\text{H}^+ \rightarrow 2\text{Mn}^{2+} + 5\text{O}_2 + 8\text{H}_2\text{O}$

Requirements:

- **(a) Apparatus:** Volumetric flask, Burette, Pipette, Conical flask, Burette stand, Water

bath, Beaker, Funnel etc.

- **(b) Chemicals:** Potassium permanganate, Sodium oxalate, Sulphuric acid, Hydrogen peroxide.

Procedure:

1. **Preparation of 0.02 M Potassium permanganate:** Dissolve 3.16 g potassium permanganate in water and make the volume up to 1000 mL with water.
2. **Standardization of prepared 0.02 M Potassium permanganate:** Weigh accurately 6.7 g sodium oxalate into a litre graduated flask, dissolve in water and make up to volume. Pipette out 20 mL (0.134 g) of this solution, add 5 mL concentrated sulphuric acid and warm up to 70°C. Add potassium permanganate solution from the burette. The first few drops result in pink colour persisting about 20 seconds. The end point is reached when a faint pink colour persists for 30 seconds upon shaking the flask. Wait until the colour disappears and then continue the titration. Formation of brown colour during the titration is caused by insufficient acid, by using too high temperature or by using dirty flask. Factor: 0.0067 g sodium oxalate is equivalent to 1 mL of 0.02 M KMnO_4 .
3. **Assay of Hydrogen peroxide:** Pipette 10 mL hydrogen peroxide into a 100 mL graduated flask and titrate 20 mL (0.0608 g) of this solution with 0.02 M KMnO_4 in a cold after adding 5 mL 50% H_2SO_4 . The end points reached when a faint pink colour persists for 30 seconds upon shaking the flask. Factor: 0.001701 g H_2O_2 is equivalent to 1 mL of 0.02 M KMnO_4 .

Pharmacopoeial Limit: Contains 95.0% to 105.0% of H_2O_2 .

Observation Tables:

1. For Standardization of 0.02 M KMnO_4 :

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

1. For Assay of Hydrogen Peroxide:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

Calculations:

1. For Standardization of 0.02 M KMnO_4 solution:
Factor: 0.0067 g sodium oxalate is equivalent to 1 mL of 0.02 M KMnO_4 .
0.134 g sodium oxalate is equivalent to mL of x M KMnO_4
 $x \text{ M} = (0.134 \times 1 \times 0.02) / (0.0067 \times \dots)$
 $x \text{ M} = \dots \text{ M}$
2. For Assay of Hydrogen peroxide: Factor:
0.001701 g H_2O_2 is equivalent to 1 mL of 0.02 M KMnO_4 .
x g H_2O_2 is equivalent to mL of x M KMnO_4

$$x \text{ g} = (0.001701 \times \dots \times \dots) / (1 \times 0.02)$$

$$x \text{ g} = \dots \text{ g}$$

3. Calculation of percent purity:

$$0.0608 \text{ g hydrogen peroxide} = 100\%$$

$$\text{Therefore } x \text{ g hydrogen peroxide} = x \%$$

$$x \% = (x \text{ g} \times 100) / 0.0608$$

$$x \% = \dots \% \text{ w/w}$$

Result: The percent purity of hydrogen peroxide was found to be __% w/w.

Conclusion: The assay of hydrogen peroxide passes / fails as per Indian Pharmacopoeia.

References:

1. Indian Pharmacopoeia, 2018.
2. Practical Pharmaceutical Chemistry, Part I (Beckett & Stenlake).

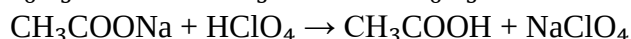
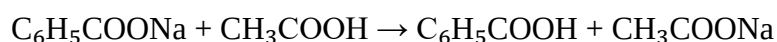
Experiment No. 18

Aim: To perform an assay of Sodium Benzoate by non-aqueous acid-base titration.

Practical Significance: Provides a highly accurate method to quantify weak, water-insoluble, or poorly ionizing compounds without the levelling effects of water. Sodium benzoate is used as a preservative in oral pharmaceutical formulations.

Relevant Theoretical Background: Glacial acetic acid acts as an acidic (protogenic) solvent to enhance the basic properties of sodium benzoate. Perchloric acid is used as titrant and crystal violet as indicator.

Reactions:



Requirements:

- **(a) Apparatus:** Volumetric flask, Burette, Pipette, Conical flask, Burette stand, Water bath, Beaker, Funnel etc.
- **(b) Chemicals:** Perchloric acid, Acetic anhydride, Glacial acetic acid, Potassium hydrogen phthalate, Crystal violet, Sodium benzoate.

Procedure:

1. Preparation of 0.1 M Perchloric acid: Mix 8.5 mL of 70%-72% HClO_4 with 500 mL of anhydrous glacial acetic acid and 25-30 mL of acetic anhydride. Cool, dilute to 1000 mL with acetic acid, and let it stand for 24 hours to ensure all water is consumed.
2. Standardization of 0.1 M Perchloric acid: Weigh accurately about 0.35 g of potassium hydrogen phthalate, previously powdered lightly and dried at 120°C for 2 hours and dissolve it in 50 mL of anhydrous glacial acetic acid. Add 0.1 mL of crystal violet solution and titrate with the perchloric acid solution until the violet colour changes to emerald-green. Perform a blank determination and make any necessary correction. Factor: Each mL of 0.1 M perchloric acid is equivalent to 0.02042 g of $\text{C}_8\text{H}_5\text{KO}_4$.
3. Assay of sodium benzoate: Weigh accurately about 0.25 g, dissolve in 20 mL of anhydrous glacial acetic acid, warming to 50°C if necessary, cool and titrate against 0.1 M perchloric acid using crystal violet as an indicator. Perform a blank determination and make any necessary correction. Factor: Each mL of 0.1 M perchloric acid is equivalent to 0.01441 g of $\text{C}_7\text{H}_5\text{NaO}_2$.

Pharmacopoeial Limit: It should not contain less than 99.0% and not more than 100.5% of $\text{C}_7\text{H}_5\text{NaO}_2$ calculated on a dried basis.

Observation Tables:

1. For Standardization of 0.1 M Perchloric acid solution:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

1. For Assay of Sodium Benzoate:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

Calculations:

1. For Standardization of 0.1 M Perchloric acid solution:

Factor: 0.02042 g of $C_8H_5KO_4$ is equivalent to each mL of 0.1 M perchloric acid.

0.35 g of $C_8H_5KO_4$ is equivalent to mL of x M perchloric acid.

$$x \text{ M} = (0.35 \times 1) / (0.02042 \times \dots)$$

$$x \text{ M} = \dots \text{ M}$$

2. For Assay of Sodium benzoate:

Factor: 0.01441 g of $C_7H_5NaO_2$ is equivalent to each mL of 0.1 M perchloric acid.

x g of $C_7H_5NaO_2$ equivalent to mL of x M perchloric acid.

$$x \text{ g} = (0.01441 \times \dots \times \dots) / 0.1$$

$$x \text{ g} = \dots \text{ g}$$

3. Calculation of Percent purity:

0.25 g sodium benzoate = 100%

Therefore, x g sodium benzoate = x %

$$x \% = (x \text{ g} \times 100) / 0.25$$

$$x \% = \dots \% \text{ w/w}$$

Result: The percent purity of sodium benzoate by non-aqueous acid-base titration was found to be % w/w.

Conclusion: Assay passes / fails as per Indian Pharmacopoeia.

References:

1. Indian Pharmacopoeia, 2018.
2. Practical Pharmaceutical Chemistry, Part I (Beckett & Stenlake).

Experiment No. 19

Aim: To perform an assay of Sodium Chloride by precipitation titration (Modified Volhard's Method).

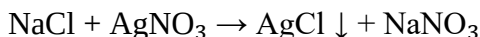
Practical Significance: Serves as a benchmark for quantitative, pharmacopoeial-grade assays of Sodium Chloride raw materials and finished products. Carried out in an acidic (nitric acid) environment to prevent precipitation of basic silver salts.

Relevant Theoretical Background: The assay of sodium chloride by the Modified Volhard's Method relies on an indirect (back) precipitation titration in an acidic medium. An excess of standard silver nitrate precipitates the chloride. The unreacted AgNO_3 is back-titrated with standard potassium or ammonium thiocyanate (SCN^-) using ferric salt indicator giving reddish brown precipitate.

Reactions:

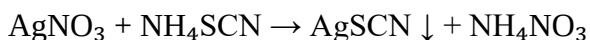
1. Precipitation of Chloride:

Sodium chloride reacts with a measured excess of silver nitrate to form a white precipitate of silver chloride:



2. Back-Titration of Excess Silver:

The unreacted silver nitrate is titrated with standardized ammonium thiocyanate:



3. End Point Indication:

Once all the excess silver ions are consumed, the first slight excess of thiocyanate reacts with ferric (Fe^{3+}) ions from the ferric ammonium sulphate indicator to produce a soluble, reddish-brown ferric thiocyanate complex:



Requirements:

- **(a) Apparatus:** Volumetric flask, Burette, Pipette, Conical flask, Burette stand, Water bath, Beaker, Funnel etc.
- **(b) Chemicals:** Ammonium thiocyanate, Silver nitrate, Nitric acid, Ferric ammonium sulphate, Nitrobenzene etc.

Procedure:

1. Preparation of 0.1 M Ammonium thiocyanate: Accurately weigh 7.612 g of Ammonium thiocyanate, transfer it to 1000 mL volumetric flask, and dissolve it in approximately 500 mL of purified water. Once fully dissolved, dilute to the 1000 mL mark with purified water and mix thoroughly.

- Standardization of prepared 0.1 M Ammonium thiocyanate: Pipette 30 mL of 0.1 M silver nitrate into a glass-stoppered conical flask and dilute with 50 mL of purified water. Add 2 mL of nitric acid and 2 mL of ferric ammonium sulphate solution as an indicator. Titrate dropwise with the prepared ammonium thiocyanate solution while shaking continuously. The endpoint is reached at the first appearance of a persistent red-brown (or reddish-yellow) color.
- Assay of Sodium chloride: Dissolve accurately weighed 0.2 g sodium chloride in 35 mL distilled water, add 15 mL dilute nitric acid, 5 mL nitrobenzene and 50 mL 0.1 M silver nitrate, shake vigorously for 1 min. Add 2 mL ferric ammonium sulphate indicator. Titrate the excess silver nitrate with 0.1 M ammonium thiocyanate until a permanent faint reddish-brown colour appears. Carry out a blank determination under the same conditions. Factor: 0.005844 g sodium chloride is equivalent to 1 mL of 0.1 M AgNO₃.

Pharmacopoeial Limit: 99.0% to 100.5% of NaCl.

Observation Tables:

1. For Standardization of 0.1 M Ammonium thiocyanate:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

1. For Assay of Sodium Chloride:

Sr. No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Mean Burette Reading (mL)
1.				
2.				
3.				

Calculations:

1. For Standardization of 0.1 M Ammonium thiocyanate solution:

$$M_1 V_1 = M_2 V_2$$

$$M_2 = (M_1 V_1) / V_2$$

$$M_2 = (0.1 \times 30) / \dots$$

$$M_2 = \dots \text{ M}$$

2. For Assay of Sodium chloride: Factor:

0.005844 g sodium chloride is equivalent to 1 mL of 0.1 M AgNO₃.

x g sodium chloride is equivalent to mL of M AgNO₃.

$$x \text{ g} = (0.005844 \times \dots \times \dots) / (1 \times 0.1)$$

$$x \text{ g} = \dots \text{ g}$$

3. Calculation of percent purity:

$$0.2 \text{ g sodium chloride} = 100\%$$

x g sodium chloride = x %

x % = (x g × 100) / 0.2

x % = % w/w

Result: Percent purity was found to be _____% w/w.

Conclusion: Assay passes / fails as per Indian Pharmacopoeia.

References:

1. Indian Pharmacopoeia, 2018.
2. Practical Pharmaceutical Chemistry, Part I (Beckett & Stenlake).