Part 4:
Assay of Pharmaceuticals

CHAPTER-09

Experiment: 09

TO PERFORM ASSAY OF FERROUS SULPHATE (REDOX TITRATION) BY CERIMETRY AND STANDARDIZATION OF TITRANT

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Aim:

To perform assay of **ferrous sulphate** (Redox titration) by cerimetry and standardization of titrant.

Requirements:

A. Glassware & Instruments:

1. Pipette (25 cm^3)
2. Pipette filler
3. Burette
4. Filter funnel
5. Conical flask (250 cm^3)
6. White tile
7. Graduated cylinders (100 cm^3)
8. Retort stand
9. Clamp
10. Beakers (250 cm^3)

B. Chemicals & Reagents:

1. Ferrous sulphate
2. Sulphuric Acid=1M
3. Ceric Ammonium Sulphate = 0.1M
4. Ferroin salt as indicator
5. Arsenic Trioxide
6. Sodium Hydroxide 87%

Principle:

Ceric ammonium sulphate in sulphuric acid medium can act as a powerful oxidising agent and has a high oxidation propensity. Sufficient sulphuric acid is required to avoid hydrolysis and precipitation of basic salts.

Ceric ammonium sulphate, a salt with appropriate solubility for preparing the standard solution, has the approximate formula $Ce(SO_4)_2.2(NH_4)_2.2H_2O$, although the solution must be standardised against a solution of arsenic trioxide (As_2O_3). Due to the fact that ceric ammonium sulphate is a potent oxidizer. Simple titration can be used to identify numerous reducing chemicals. In the presence of a reducing agent, it is reduced to the cerous form.

Procedure:

Standardization of 0.1m $Ce(SO_4)_2$ Solution

- Weigh accurately about 0.2 g of As_2O_3, previously dried at 105 degree C for 1 hour and transfer it to 500ml conical flask.
- Wash down the inner walls of the flask with NaOH and then add 25ml of 8% w/v solution of sodium hydroxide
- Stir until dissolved, then add 100ml of water and combine.
- Add 30 ml of dilute Sulphuric acid, 0.15 ml of osmic acid solution.0.1ml of ferroin sulphate solution
- Then slowly titrate with $Ce(SO_4)_2$ Solution until the pink color is changed to very pale blue colour.

Assay:

- Weigh accurately about 0.5gm of ferric sulphate dissolve in a mixture of 30 ml water and 20 ml of 0.1N sulphuric acid.
- Titrate with 0.1M Ceric ammonium sulphate solution using ferroin sulphate solution as indicator.

IP Factor- Each ml of 0.1M $Ce(SO_4)_2$ is equivalent to 0.01519 of Fe_2SO_4

Reaction:

$2Ce(SO_4)_2 + 2(FeSO_4) \rightarrow Ce_2(SO_4)_3 + Fe_2(SO_4)_3$

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Calculation:

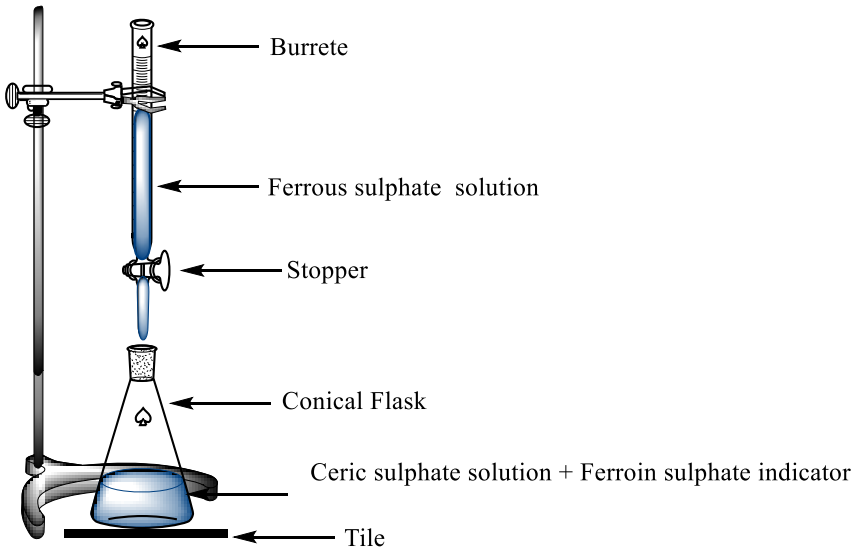
Observation table

S.no	Volume of H_2SO_4 solution	Initial Reading	Final Reading	Volume of $Ce(SO_4)_2$ rundown (ml)
1.				
2.				
3.				

M of $Ce(SO_4)_2$ = wt taken * expected M / Titration vol * IP Factor

% Purity of $FeSO_4$ = vol of $Ce(SO_4)_2$ * IP factor * 100 * M (actual)/ wt. of $FeSO_4$ * M (expected)

Diagram:



Application:

1. The medication ferrous sulphate is used to treat and prevent iron deficiency anaemia.
2. Iron aids in the production of healthy red blood cells, which transport oxygen throughout the body. Some factors, such as blood loss, pregnancy, or inadequate iron intake, might deplete your iron stores, resulting in anaemia.

Result:

The percent purity of ferrous sulphate is ……...

Viva questions:

- What is the chemical formula of ferrous sulphate?
- Describe the characteristics of potassium permanganate and its chemical reaction.
- In which indicator is this titration conducted?
- Explain the definition of oxidation-reduction titration.
- How does potassium permanganate function as an indicator?
- Define the terms oxidation and reduction.
- Write the reactions involved in the determination of ferrous sulphate.
- Which standard solution is employed to standardise potassium permanganate solution?
- Provide the method for preparing 0.1N oxalic acid solution.
- Write the assay principle for ferrous sulphate.
- Write the assay standard and factor for ferrous sulphate.

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