CHAPTER-02

Experiment: 02

PERFORM AND REPORT THE LIMIT TEST FOR SULPHATE IN THE GIVEN SAMPLE OF SODIUM BICARBONATE AS PER INDIAN PHARMACOPOEIA

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Aim:

To perform and report the limit test for sulphate in the given sample of sodium bicarbonate as per I.P.

Requirements:

A. Glassware & Instruments:

2. Measuring cylinder
3. Pipette
4. Beaker
5. Nessler's cylinder
6. Glass rod

B. Chemicals & Reagents:

1. Na_2CO_3
2. Potassium sulphate
3. $BaSO_4$
4. HCl (2M)
5. Distilled water (DW)
6. EtOH
7. $BaCl_2$
8. CH_3COOH (5M)

Theory:

Limit tests are quantitative or semi-quantitative tests meant to identify and regulate the small amount of impurities possibly present in a substance. This test involves the interaction of $BaCl_2$ with soluble sulphate to form the insoluble in dilute hydrochloric acid $BaSO_4$ precipitate. The precipitate of barium sulphate is white in colour.

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Principle:

The sulphate limit test is dependent on the reaction between soluble sulphate and $BaCl_2$ in the presence of alcohol and potassium sulphate. Deposition of solid sulphate particles, such as $BaSO_4$ (For this purpose, $BaCl_2$, alcohol, and a small amount of potassium sulphate are utilised. The alcohol prevents supersaturation, while the potassium sulphate increases the sensitivity of the test by providing an ionic concentration in the reagent that just exceeds the solubility product of $BaSO_4$.). The opalescence/turbidity produced by the sample under test is compared to the opalescence/turbidity produced by the reference sample against the dark backdrop.

Reaction:

$$\underset{\text{Soluble}}{SO_4^-} + \underset{\text{Soluble}}{BaCl_2} \xrightarrow{CH_3COOH} \underset{\text{White precipitate}}{BaSO_4\downarrow} + \underset{\text{Soluble}}{2Cl^-}$$

Test Sample

$$\underset{\text{Soluble}}{Na_2SO_4} + \underset{\text{Soluble}}{BaCl_2} \xrightarrow{CH_3COOH} \underset{\text{White precipitate}}{BaSO_4\downarrow} + \underset{\text{Soluble}}{NaCl}$$

Refernce Sample

Procedure:

Procedure for Test Sample	Procedure for Reference Sample
➢ Take 1 ml of 25% w/v solution of $BaCl_2$ in the labeled Nesslers cylinder (T). ➢ Add 1.5 ml of ethanolic sulphate standard solution (10 ppm SO_4), mix and allow to stand for 1 minute. ➢ Weigh accurately 1.0 g of Na_2CO_3 and add to a labeled Nesslers cylinder. ➢ Add 10 ml DW, neutralise with HCl	➢ Take 1 ml of 25% w/v solution of $BaCl_2$ in the Nesslers cylinder (R). ➢ Add 1.5 ml of ethanolic sulphate standard solution (10 ppm SO_4), mix and allow to stand for 1 minute. ➢ Add 0.15 ml of 5M CH_3COOH (5M). ➢ Add sufficient DW to produce 50 ml, stir immediately with glass rod.

and dilute to 15 ml with distilled water. ➢ Add 0.15 ml of CH_3COOH (5M). ➢ Add sufficient DW to produce 50 ml, stir immediately with glass rod and allow to stand for 5 minutes. ➢ View transversely against a black background. ➢ Compare opalescence with that of standard solution.	➢ Allow to stand for 5 minutes.

Observation:

The opalescence produced by the Na_2CO_3 test solution is……. (less/equal/greater) than that of the standard solution.

Results:

The given sample of Na_2CO_3..... (complies/does not comply) the limit test for sulphate as per the I.P.

Viva Questions:

- Define the term impurities as it pertains to pharmaceuticals.
- What is supersaturation?
- Explain the definition of opalescence.
- Provide the technique for preparing standard solution for sulphate impurity testing.
- Provide the technique for preparing a test solution of purified water for sulphate impurity analysis.
- Describe the technique for preparing sodium bicarbonate test solutions for detecting sulphate contaminants.
- Provide the technique for preparing a test solution of calcium gluconate for sulphate impurity analysis.
- Describe the use of barium chloride and alcohol in sulphate limit tests.

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