

CHAPTER-02



Experiment: 02

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

Author (s)

Dr. KAPIL KUMAR

Dr. NARENDER YADAV

Chapter Id: ASU/NSP/EB/APHPC/2022/Ch-02

doi: <https://doi.org/10.52458/9789391842611.nsp2022.eb.asu.ch02>

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)

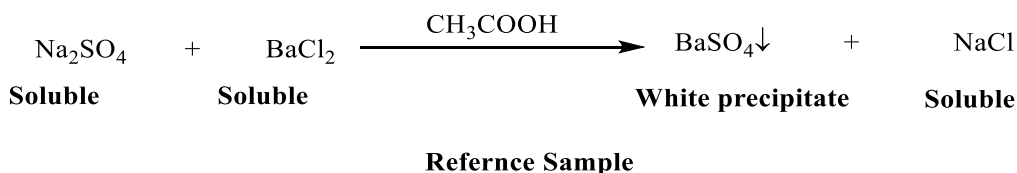
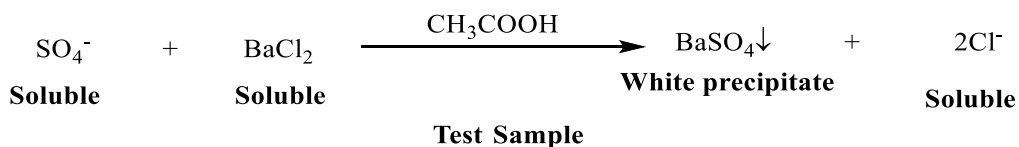
Theory:

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.

Principle:

The sulphate limit test is dependent on the reaction between soluble sulphate and BaCl₂ in the presence of alcohol and potassium sulphate. Deposition of solid sulphate particles, such as BaSO₄ (For this purpose, BaCl₂, 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 BaSO₄). 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:



Procedure:

Procedure for Test Sample	Procedure for Reference Sample
<ul style="list-style-type: none"> ➤ Take 1 ml of 25% w/v solution of BaCl₂ in the labeled Nessler's cylinder (T). ➤ Add 1.5 ml of ethanolic sulphate standard solution (10 ppm SO₄), mix and allow to stand for 1 minute. ➤ Weigh accurately 1.0 g of Na₂CO₃ and add to a labeled Nessler's cylinder. ➤ Add 10 ml DW, neutralise with HCl 	<ul style="list-style-type: none"> ➤ Take 1 ml of 25% w/v solution of BaCl₂ in the Nessler's cylinder (R). ➤ Add 1.5 ml of ethanolic sulphate standard solution (10 ppm SO₄), mix and allow to stand for 1 minute. ➤ Add 0.15 ml of 5M CH₃COOH (5M). ➤ Add sufficient DW to produce 50 ml, stir immediately with glass rod.

<p>and dilute to 15 ml with distilled water.</p> <ul style="list-style-type: none">➤ Add 0.15 ml of CH₃COOH (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.	<ul style="list-style-type: none">➤ Allow to stand for 5 minutes.
--	---

Observation:

The opalescence produced by the Na₂CO₃ test solution is.....
(less/equal/greater) than that of the standard solution.

Results:

The given sample of Na₂CO₃..... (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.