

Part 1:
Limit Tests for Impurities

CHAPTER-01



Experiment: 01

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

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Chapter Id: ASU/NSP/EB/APHPC/2022/Ch-01

doi: <https://doi.org/10.52458/9789391842611.nsp2022.eb.asu.ch01>

Aim:

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

Requirements:

A. Glassware & Instruments:

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

B. Chemicals & Reagents:

1. Na_2CO_3
2. AgNO_3 solution (0.1 M)
3. Dilute HNO_3
4. Standard solution of NaCl
5. Distilled water (DW)

Theory:

A limit test is a quantitative or semi-quantitative test that is developed to identify and regulate a small amount of impurities that are likely to be present in the substance. In this experiment, AgNO_3 is reacted with soluble chloride to produce precipitates of AgCl , which is insoluble in diluted HNO_3 . The amount of silver chloride that is generated, or the total number of chloride ions that are present in the substance, is directly proportional to the degree to which precipitation occurs. Under the same experimental conditions, the opalescence that is produced in the test solution is compared to the opalescence that is produced in a reference or standard solution.

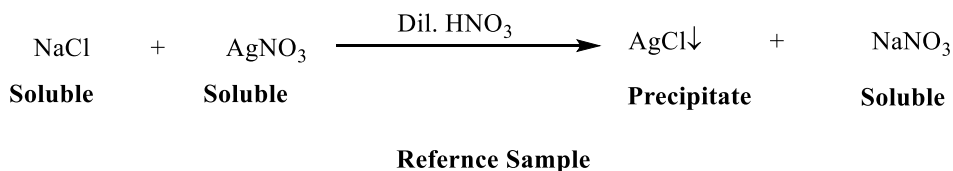
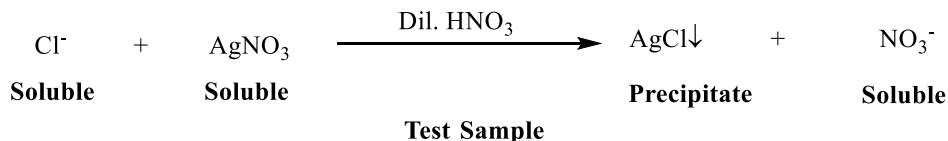
Principle:

The chloride limit test depends on the reaction between chlorides and AgNO_3 in the presence of diluted HNO_3 . Solid chloride particle deposition as AgCl . As an impurity, chloride is present in very minute concentrations. The precipitation of silver

To perform and report the limit test for chloride in the given sample of sodium bicarbonate as per

chloride during the chemical reaction shows as opalescence. To derive conclusions, it is compared to normal opalescence in Nessler cylinders under homogeneous lighting circumstances.

Reaction:



Procedure:

Procedure for Test Sample	Procedure for Reference Sample
<ul style="list-style-type: none"> ➤ Weigh accurately 1.25 g of Na₂CO₃ ➤ Dissolve it in 15 ml of DW in a labeled Nessler's cylinder (T). ➤ Add 2 ml of dil. HNO₃ and mix well. ➤ Dilute to 50 ml with distilled water. ➤ Add 1 ml of AgNO₃ (0.1M) solution. ➤ Stir immediately with glass rod and allow to stand for 5 minutes, protect from light. ➤ View transversely, against a black background. ➤ Compare the opalescence produced with that of standard solution. 	<ul style="list-style-type: none"> ➤ Take 10 ml of chloride standard solution. (25 ppm Cl) in labeled Nessler's cylinder (R). ➤ Add 5 ml of DW. ➤ Add 10 ml of dilute HNO₃ and mix well. ➤ Dilute to 50 ml with DW. ➤ Add 1 ml of dil. AgNO₃ (0.1M) solution. ➤ Stir immediately with glass rod and allow to stand for 5 minutes, protect from light.

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 chlorides as per the I.P.

Viva Questions:

- Explain the definition of opalescence.
- Write the limit test principle for chlorides.
- Provide the chemical equation for the chloride limit test.
- Provide the rationale for opalescence production.
- Describe the method for preparing the standard solution and the sodium bicarbonate test solution for the chloride limit test I.P.
- Explain why silver nitrate is used in the chloride limit test.
- Explain why nitric acid is used in the chloride limit test.