

CHAPTER-03



Experiment: 03

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

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Aim:

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

Requirements:

A. Glassware & Instruments:

- a. Measuring cylinder
- b. Pipette
- c. Beaker
- d. Nessler's cylinder
- e. Glass rod
- f. Analytical balance

B. Chemicals & Reagents:

1. Standard Fe Solution
2. NaCl
3. 20% w/v Fe free citric acid
4. Distilled water (DW)
5. Thioglycollic acid
6. ZnSO₄
7. Fe free ammonia

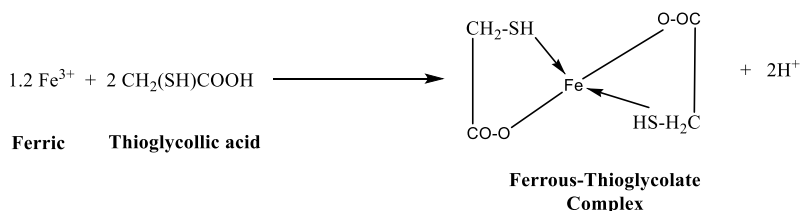
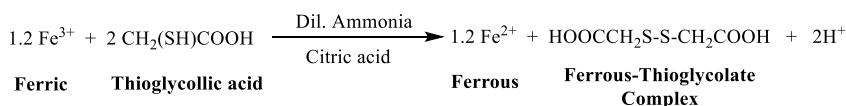
Theory:

Limit tests are quantitative or semi-quantitative tests meant to discover and control small quantities of contaminants that may be present in a substance. The iron limit test is determined by the interaction between iron and thioglycolic acid in the presence of citric acid in an ammonia solution. Iron precipitation with ammonia is prevented by citric acid. A deep, purplish-red hue is produced. In an acidic environment, ferrous thioglycolate is colourless, but in an alkaline environment, it turns purple.

Principle:

Test limit for iron It creates ferrous thioglycolate complex based on the chemical reaction of iron in ammonical solution in the presence of iron-free citric acid with thioglycolic acid. As a result of the synthesis of ferrous thioglycolate complex, a purple colour is produced. The initial condition of iron is irrelevant since thioglycolic acid converts ferric iron to ferrous iron. Using 20% citric acid, which forms complexes with other metal ions and reduces their interference, this test is extremely sensitive (Purple colour can only be produced in an alkaline media, thus ammonia solution is employed. However, ammonia solution produces a precipitate with iron, so citric acid is added to make an ammonium citrate buffer, which stabilises the ferrous thioglycolate complex.). The colour of the sample under examination is compared to the colour of the reference solution.

Reaction:



Procedure:

Procedure for Test Sample	Procedure for Reference Sample
<ul style="list-style-type: none"> ➤ Weigh accurately 2g of NaCl and dissolve in 20 ml of water in a labeled Nessler's cylinder (T). ➤ Add 2 ml of a 20% w/v solution of iron-free citric acid. ➤ And add 0.1 ml of thioglycolic acid, mix well and make alkaline with 	<ul style="list-style-type: none"> ➤ Take 2.0 ml of iron standard solution (20 ppm Fe) in labeled Nessler's cylinder (R). ➤ Add 2 ml of a 20% w/v solution of iron-free citric acid. ➤ And add 0.1 ml of thioglycolic acid, mix well, make alkaline

iron-free ammonia solution. ➤ Dilute to 50 ml with water and allow to stand for 5 min. ➤ View the colour intensity against white background and compare with that of standard.	with iron free ammonia solution. ➤ Dilute to 50 ml with water and allow to stand for 5 minutes.
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Observation:

The colour intensity produced by the NaCl test solution is..... (less/equal/greater) than that of the standard solution.

Results:

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

Viva Questions:

- Why is the iron limit test performed?
- What is the principle behind the iron limit test?
- Why are ammonia and citric acid solutions used in the iron limit test?
- Why is the iron limit test stained with a rich crimson-purple hue?
- Write the chemical reaction involved in the 'iron limit test'
- Write out the technique for preparing standard iron solution.
- Write out the method for preparing zinc sulphate test solution.
- Provide the preparation technique for sodium chloride test solution.
- Write the preparation protocol for magnesium sulphate test solution.
- How can the colour intensity be observed?
- Describe the function of thioglycolic acid in the iron limit test.