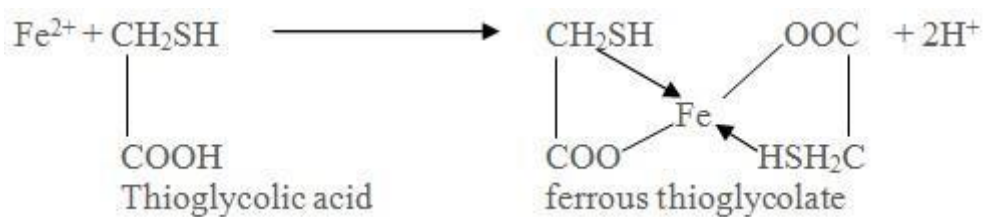


**Chemical Reaction:****Note: -**

1. Earlier ammonium thiocyanate reagent was used for the limit test of iron. Since thioglycolic acid is more sensitive reagent, it has replaced ammonium thiocyanate in the test.
2. **Standard Iron Solution:** Weigh accurately 0.1726 g of ferric ammonium sulphate and dissolve in 10 mL of 0.1 N sulphuric acid and sufficient distilled water to produce 1 Litre. Each mL of this solution contains 0.02 mg of Fe.
3. The colour developed in the sample and standard solution is compared by keeping the Nessler's cylinder against dark background and observing side by side.

### Practical - 3

Date: .../.../.....

**Aim:** To observe limits for iron impurities in the given sample as per Indian Pharmacopoeia.

**Reference:**.....  
.....

**Requirements:**

**Apparatus/Equipment required:**.....

**Chemical required:**.....  
.....

#### Principle

It depends on the reaction of iron in an ammonical solution with thioglycollic acid in the presence of citric acid when a pale pink to deep reddish-purple colour is produced. The colour is due to the formation co-ordination compound, ferrous thioglycollate which is stable in the absence of air but fades in air due to oxidation. Therefore, the colour should be compared immediately after the time allowed for full development of colour is over.

#### Procedure:

Take two 50 mL Nessler Cylinders. Label one as "Test" and the other as "Standard".

<b>Test Solution</b>	<b>Standard Solution</b>
Sample is dissolved in specific amount of distilled water and then volume is made up to 40 ml	2 ml of standard solution of iron diluted with water upto 40 ml
Add 2 mL of 20 % w/v of citric acid (iron free)	Add 2 mL of 20 % w/v of citric acid (iron free)
Add 2 drops of thioglycollic acid	Add 2 drops of thioglycollic acid
Add ammonia to make the solution alkaline and adjust the volume to 50 ml with distilled water	Add ammonia to make the solution alkaline and adjust the volume to 50 ml with distilled water
Keep aside for 5 min	Keep aside for 5 min
Observe the Turbidity	Observe the Turbidity

\*Compare the opalescence/turbidity produced by the test solution with a standard solution.

#### Observation:

The purple colour produce in sample solution should not be greater than standard solution. If purple colour produces in sample solution is less than the standard solution, the sample will pass the limit test of iron and vice versa.

#### Reasons:

- Ferrous thioglycollate is colourless in neutral or acid solutions.
- The colour develops only in the presence of alkali.

- The original state of iron is immaterial, as thioglycollic acid reduces ferric ( $\text{Fe}^{3+}$ ) to ferrous ( $\text{Fe}^{2+}$ ) form.
- Citric acid forms a soluble complex with iron and prevents its precipitation by ammonia as ferrous hydroxide.
- Interference of other metal cations is eliminated by making use of citric acid, which forms complex with other metal cations.

**Result:**.....  
.....

### Questions Bank

1. Write the chemical formula of thioglycollic acid.
2. Write the chemical formula of citric acid.
3. What is the meaning of precipitate?
4. Write the one example of ferric and ferrous salt.
5. What is the co-ordination compound?
6. What is the air oxidation?
7. Ferric salt converted in to ferrous salt, it is an oxidation or reduction reaction:
8. Write the chemical structure of Ferrous thioglycollate.
9. Why ammonium thiocyanate reagent is used in iron limit test?
10. Write the preparation method name of Ammonia.