

LIMIT TEST FOR IRON

Aim :

- To perform the limit test for iron on a given sample as per Indian pharmacopoeia and report on its standard.

Apparatus required:

Nessler's cylinder



Glass rod-2



Measuring cylinder



Pipette



Dropper



Rubber stopper



Chemicals required :

Iron free citric acid

20 % w/v solution of Citric acid

20 g of citric acid in 100 ml of water

Ammonia

- 10 % w/v of ammonia in water.

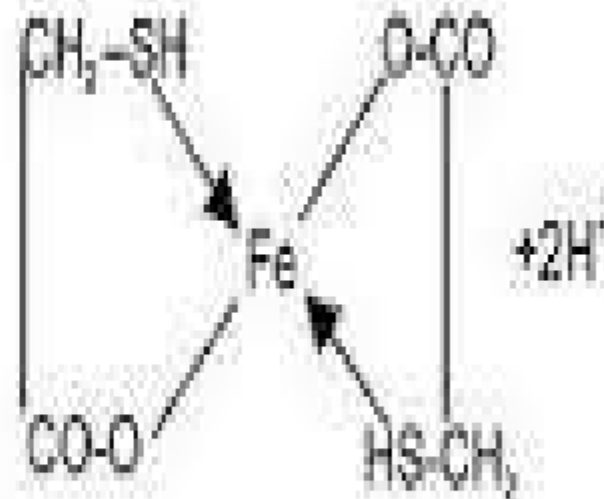
Standard iron solution

- weigh accurately 0.1728 g of ferric ammonium sulphate and dissolve it in 10 ml of 0.1N sulfuric acid and make up to 1000 ml with water.
- 1ml = 0.02 mg of Fe.

Principle:



Ferric Thioglycolic acid Ferrous



Ferrous

Thioglycolic acid

(Ferrous thioglycolate complex)

- The limit test for iron is based on the reaction of iron in ammonical solution in the presence of citric acid with thioglycolic acid. When a pale pink to deep reddish purple colour is formed due to the ferrous thioglycolate complex. The color produced from a specific amount of substance from test is compared with the color produced in standard solution by viewing vertically .

- Ferric ammonium sulphate is used as standard.
- The test is very sensitive , interference of other metal cations is eliminated by using of 20 % w/v citric acid .
- The iron free citric acid is used to complex metal cations other than iron if any.

- Ferrous thioglycollate complex is colorless in acidic or neutral solutions.
- Only in the presence of alkali pale pink color will be produced.
- citric acid forms ammonium citrate buffer when ammonia is added to make alkaline which in turn stabilizes the complex formed.

Standard	Test
Pipette out 2 ml of standard Iron solution into the Nessler's cylinder marked as standard	Dissolve the specified quantity of given sample in distilled water in Nessler's cylinder marked as test
Dilute it to 40 ml with distilled water	Dilute it to 40 ml with distilled water
Add 2 ml of 20 % w/v solution of iron free citric acid	Add 2 ml of 20 % w/v solution of iron free citric acid
Add 0.1ml of thioglycolic acid.	Add 0.1ml of thioglycolic acid.
Mix and make alkaline with iron free ammonia solution and dilute to 50ml with water	Mix and make alkaline with iron free ammonia solution and dilute to 50ml with water
Immediately stir with a glass rod and allow it to stand for five minutes	Immediately stir with a glass rod and allow it to stand for five minutes

- Compare the color produced in test solution with standard solution.
- **Precaution:**
- Same glass rod should not be used because it will affect your observation.

Sample I [Pass Sample] :

- **Observation:**
- The Colour produced in the test solution is lesser than standard solution.
- **Inference:**
- The given substance passes the limit test for Iron as per Indian pharmacopoeia when compared with that of a standard substance.

Sample II [Fail Sample] :

Observation:

The Colour produced in the test solution is more than standard solution.

Inference:

The given substance fails the limit test for Iron as per Indian pharmacopoeia when compared with that of a standard substance.

Mechanism of litmus paper

- Red litmus contains a weak diprotic acid. When it is exposed to a basic compound, the hydrogen ions react with the added base. The conjugated base, formed from the litmus acid, has a blue color, so the wet red litmus paper turns blue in alkaline solution