

Aim :-

~~28/02/22~~ To perform limit test for Iron.

Reference:

A practical book of Pharmaceutical chemistry - I,  
Dr. K.S. Jain, J.N. Kadam, Fourth Edition, 2020,  
Nisali Publication, Page No- 1.8 to 1.10.

Requirement:

- Apparatus: → Pair of Nesster's cylinder,
  - Glass rods
  - Stand
  - Measuring cylinder.
  - Spatula
  - Pipette
- Chemicals :
  - Citric Acid
  - Thioglycolic acid
  - Iron Standard Solution
  - Ammonia Solution

Theory:

→ Preparation: Iron Standard Solution (20 ppm):

Dilute 1 volume of 0.172% w/v solution of

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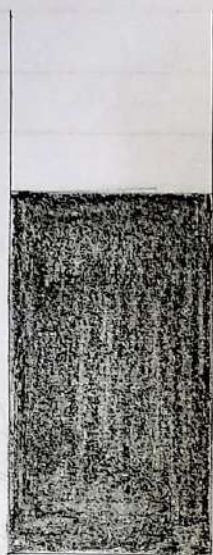


Ferric ammonium sulphate in 0.05 M sulphuric acid to 10 volumes with water. Contains iron in ferric state.

### Principle:

The test involves the chemical reaction between ferrous iron and thioglycolic acid in the presence of citric acid and ammoniacal alkaline medium. When a pale pink to deep reddish purple colour is obtained. Ferric iron is reduced to ferrous iron by the thioglycolic acid and ferrous thioglycollate is produced. Citric acid forms a soluble complex with iron and prevents its precipitation by ammonia as ferrous hydroxide. Ferrous thioglycollate is colourless in neutral or acid solutions. The colour due to ferrous thioglycollate compound gets destroyed by oxidizing agents. To avoid the interference of other ions, 20% iron free citric acid is used. Citric acid forms a complex with other metal cations. The colour produced from test substance is compared by viewing vertically with standard solution (Ferric ammonic sulphate). If the colour of test solution is less dark than standard solution, then test sample passes the test.



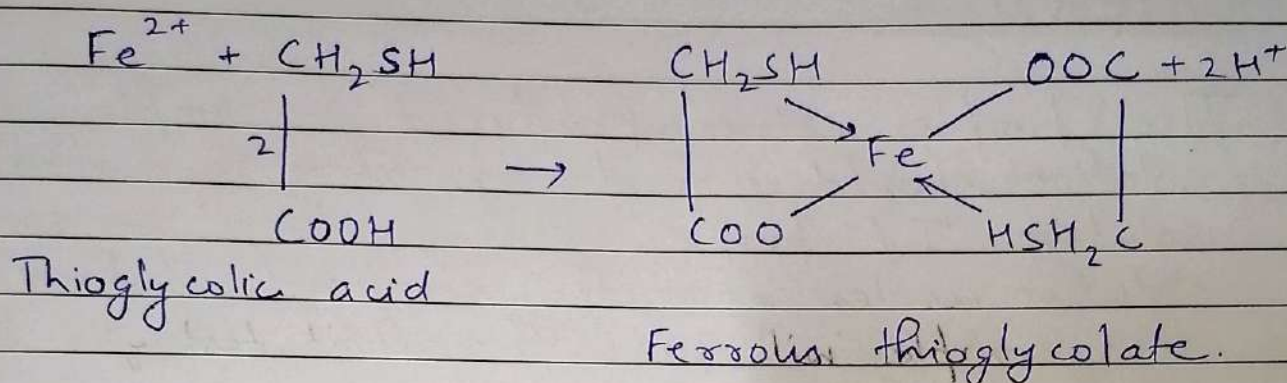


Standard



Test

Fig:- The intensity of the colour in STD is seen more than that of the Test. Thus, the sample passes the limit test for iron.



### Procedure:

Take two 50 ml Nessler's cylinders. Label one as 'Test' and the other as 'Standard'.

#### (A) Test Solution:

1. Dissolve the specified quantity of substance under examination in distilled water or prepare a solution as directed in individual monograph and transfer to a Nessler's cylinder.
2. Add 2 ml of 20% w/v solution of iron free citric acid and 0.1 ml of Thioglycolic acid. Mix, make alkaline with iron free ammonia solution.
3. Dilute to 50 ml with distilled water and allow to stand for 5 minutes.

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## Obseovation :

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

(B) Standard Solution

1. Take 2 ml of Iron. Standard solution (20 ppm) in 20 ml distilled water and transfer to a Nessler's cylinder.
2. Add 2 ml of 20% w/v solution of iron free Citric acid and 0.1 ml of Thioglycollic acid. Mix, make alkaline with iron free ammonia solution.
3. Dilute to 50 ml with distilled water and allow to stand for 5 minutes.

<u>Test Solution</u>	<u>Standard Solution</u>
→ Dissolve the specified quantity of substance under examination in distilled water and transfer in a Nessler's cylinder.	→ Take 2 ml of Iron Standard Solution (20 ppm) in 20 ml water and transfer to a Nessler's cylinder.
→ Add 2 ml of 20% w/v solution of iron free Citric acid and 0.1 ml of Thioglycollic acid.	→ Add 2 ml of 20% w/v solution of iron free Citric acid and 0.1 ml of Thioglycollic acid.

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→ Make alkaline with iron free ammonia solution.

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→ Observe the intensity of the purple, color developed by viewing vertically and compare with ~~of~~ of the standard.

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### Note:

All the reagents used in the limit test for iron should themselves be iron-free. Hence, they themselves should conform to the limit.

### Result:

Given sample passes the limit test for iron.