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Diploma in Pharmacy 1st Year Pharmaceutical Chemistry Experiment

To perform the limit test for iron.

Aim:

To perform the limit test for iron.

Reference:

⁶ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ² "Practical Manual of Pharmaceutical Chemistry" Published by Nirali Prakashan, Page no 30 - 32

Requirements:

Apparatus Required : Nessler cylinder, Glass rod, Test tube, Dropper, Pipette, Measuring Cylinder, Beaker, Rubber Stopper,

Chemicals Required : Ammonium citrate buffer, Mercaptoacetic acid, Citric acid, Thioglycolic acid, Ammonia,Distilled water.

Theory:

- The limit test for iron relies on the reaction in which iron reacts with mercaptoacetic acid (thioglycolic acid) in a solution with ammonium citrate buffer.
- It results in the formation of a purple colour solution due to the formation of ferrous mercaptoacetate (a coordination compound) and ferric iron being reduced to the ferrous state by the reagent.
- This purple colour is compared with the standard colour, containing a known amount of iron.



Test Solution Specified substance (1gm) + 10ml of water 1ml of HNO3

Standard Solution 1ml of 0.05845% w/v solution of sodium chloride

1ml of HNO₃

Diluted to 50ml in Nessler cylinder Diluted to 50ml in Nessler cylinder A + 1ml of AgNO₃ solution

Opalescence/turbidity

 $B + 1ml of AgNO_3$ solution

Opalescence/turbidity

Procedure:

In this limit test, a standard and test solution is prepared and the appearance of these two solutions is compared:

1) Test Solution : 4 oml of water is added to the sample and treated with 2ml of 20% w/v citric acid. Then 2 drops of thioglycolic acid are added, the solution is mixed, made alkaline with ammonia, and volume is made up to 50ml. Then, the solution is allowed to stand for 5 minutes so that a colour develops which is viewed vertically and compared with the standard solution.

2) Standard Solution : 40ml of water is added to 2ml of standard solution of iron. Then 2ml of 20% w/v citric acid and 2 drops of thioglycolic acid is added to the above solution. The solution is made alkaline with ammonia and volume is made up to 50ml. The resultant solution is allowed to stand for 5 minutes so that a colour develops which is viewed vertically and compared with the test solution.



When the colour of both the solutions is compared, the intensity of the colour of the test solution should be less than that of the standard solution.

Some essential points that should be kept in mind while performing the limit test of iron are as follows :

- Colour is developed and not turbidity.
- The solutions should be compared immediately within 5 minutes, else the colour fades away due to oxidation, making the test unreliable.
- Mercaptoacetic acid test is very sensitive.

Observation

• The intensity of purple colour produced in the sample solution should not exceed that of the standard solution. If the purple colour intensity of the sample solution is less than the standard solution, the sample will pass the limit test of iron and vice versa.

Result: The limit test of iron was performed.



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