

Limit test for sulphate

Aim

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

Apparatus required:

Nessler's cylinder



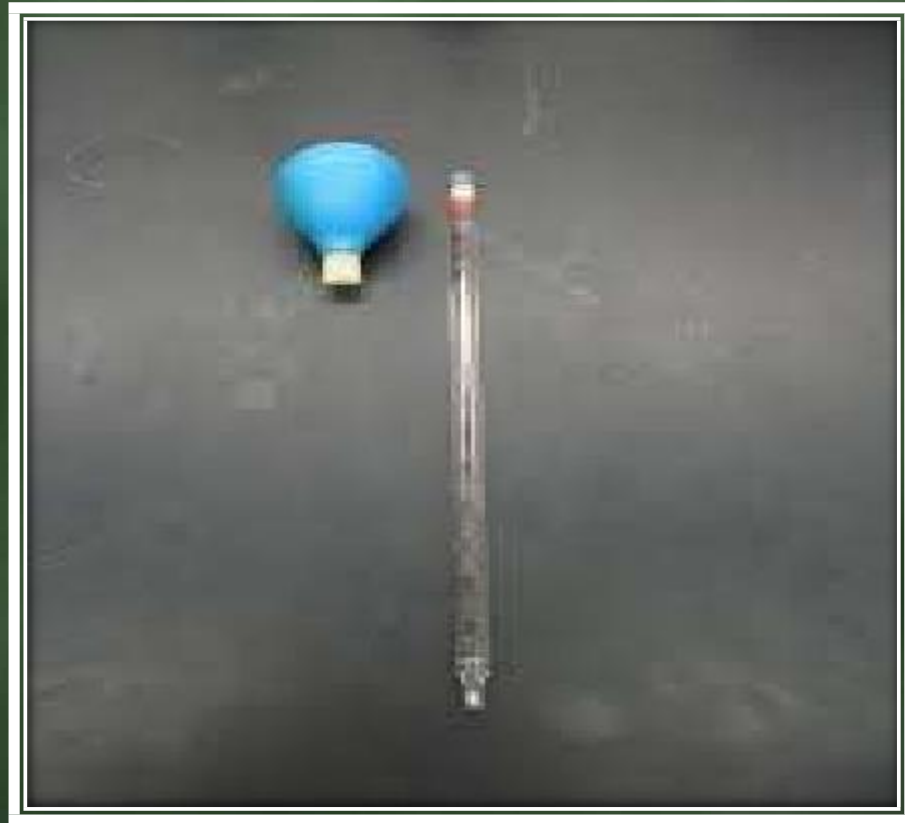
Glass rod-2



Measuring cylinder



Pipette



Dropper



Rubber stopper



Chemicals required :

- Barium sulphate reagent:
- 0.05 M Barium chloride solution is prepared by dissolving 12 g of barium chloride in 1000ml of water, to the 15 ml of the above solution add 55 ml of water, 20 ml of alcohol and 5ml of 0.0181% w/v solution of potassium sulphate and the final volume was made up to 100 ml.

Preparation of ethanolic sulphate standard solution (10 PPM SO_4^{2-}):

- Dilute 1 volume of a 0.1089% w/v solution of potassium sulphate in ethanol (30%) to 100 volumes with ethanol (30%).

Preparation of sulphate standard solution (10 PPM SO_4^{2-}):

- Dilute 1 volume of a 0.1089 % w/v solution of potassium sulphate in distilled water to 100 volumes with distilled water

Principle :

- The limit test for sulphate is based on the reaction between barium chloride and soluble sulphates in presence of dilute hydrochloric acid. Then the turbidity produced is compared with the standard turbidity .



Barium sulphate reagent contains

- Barium chloride,
- Sulphate Free Alcohol
- Potassium Sulphate in Water.

Potassium sulphate is used to increase the sensitivity of the test.

It reacts with the barium chloride and forms barium sulphate in small quantity which will act as a seeding agent.

The ionic concentration has been so adjusted, such that the solubility product of barium sulphate is exceeded and a very small amount of barium sulphate acts as a seeding agent for the precipitation of barium sulphate.

- Alcohol is to prevent super saturation and thus produces uniform turbidity.
- Hydrochloric acid helps to make solution acidic and the barium sulphate precipitate formed is insoluble which gives turbidity.

Procedure

Standard	Test
Pipette out 1 ml of standard Sulphate 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
Add 2 ml of dilute nitric acid	Add 2 ml of dilute nitric acid
Dilute it to 45 ml with distilled water	Dilute it to 45 ml with distilled water
Add 5 ml of Barium sulphate reagent	Add 5 ml of Barium sulphate reagent
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
Observe it under black background	Observe it under black background

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

Sample I [Pass Sample] :

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

Sample II [Fail Sample] :

- **Observation:**
- The turbidity produced in the test solution is more than standard solution.
- **Inference:**
- The given substance fails the limit test for sulphate as per Indian pharmacopoeia when compared with that of a standard substance.