Chemical Reaction:

$$K_{2}SO_{4} + BaCl_{2} \xrightarrow{Acetic Acid} BaSO_{4} \downarrow + 2KCl$$
$$SO_{4}^{2} + BaCl_{2} \xrightarrow{Acetic Acid} BaSO_{4} \downarrow + Cl^{-}$$

Note: -

1. Barium Sulphate Reagent: Mix 10 mL of 25% w/v solution of barium chloride and 15 mL of ethanolic sulphate standard solution (10 ppm SO₄) and allow standing for 1 minute. It should always be prepared fresh.

2. Sulphate standard solution (10 ppm SO₄): Dilute 1.0 mL of a 0.181% w/v solution of potassium sulphate in distilled water to 100 mL with the same solvent.

3. Ethanolic sulphate standard solution (10 ppm SO₄): Dilute 1.0 mL of a 0.181% w/v solution of potassium sulphate in ethanol (30%) to 100 mL with the same solvent.

4. 5.0 M Acetic acid: Dilute 28.5 mL of glacial acetic acid in sufficient distilled water to produce 100 mL.

5. This test is not used for water-immiscible liquids.

6. The opalescence in the sample and standard solution is compared by keeping the Nessler cylinders against dark background and observing side by side.



Aim: To observe limit test for Sulphate impurities in the given sample as per Indian Pha	Date://armacopoeia.
Reference:	
Requirements:	
Apparatus/Equipment required:	
Chemical required:	

Principle

The limit test for sulphate is carried out on the basis of the reaction between barium chloride and soluble sulphates in the presence of Acetic acid. Then, the comparison of the turbidity produced by a given amount of the substance is done with a standard turbidity obtained from a known amount of sulphates.

Procedure:

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

Test Solution	Standard Solution
Specific weight of compound is dissolved in water or	Take 1ml of 0.1089 % W/V solution of potassium
solution is prepared as directed in the pharmacopoeia	sulphate in Nessler cylinder
and transferred in Nessler cylinder	
Add 2 ml of dilute hydrochloric acid	Add 2 ml of dilute hydrochloric acid
Dilute to 45 ml in Nessler cylinder	Dilute to 45 ml in Nessler cylinder
Add 5ml of barium sulphate reagent	Add 5ml of barium sulphate reagent
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 turbidity produce in sample solution should not be greater than standard solution. If turbidity produces in sample solution is less than the standard solution, the sample will pass the limit test of sulphate and vice versa.

Reasons:

- Hydrochloric acid helps to make solution acidic.
- Potassium sulphate is used to increase the sensitivity of the test by giving ionic concentration in the reagent alcohol helps to prevent super saturation.

Result:

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Questions Bank

- 1. Write the chemical formula of acetic acid.
- 2. What is the meaning of Turbidity?
- 3. What is the meaning of ppm?
- 4. What is ethanolic sulphate standard solution?
- 5. Why we do the limit test of sulphate?
- 6. What is the glacial acetic acid?
- 7. What is the distilled water?
- 8. What is the meaning of 5.0 M Acetic acid?
- 9. Write the formula of Normality calculation.
- 10. What is the difference between test solution and standard solution?