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Diploma in Pharmacy 1st Year
Pharmaceutical Chemistry
Experiment
To perform limit test for sulphate in given compound.

Aim:

To perform limit test for sulphate in given compound.

Reference :

‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 9 - 11

Requirements:

Chemicals: Standard sulphate solution (10 ppm), ethanolic standard sulphate solution (10 ppm), 25% w/v barium chloride solution, 5 M acetic acid, distilled water.

Apparatus: Nessler's cylinder, glass rod, beaker, dropper, volumetric flasks (100 ml) pipette (1 and 10 ml), etc.

Theory:

- Limit test for sulphate is based on reaction of barium chloride and soluble sulphate in acidic solution.
- The opalescence formed in test solution is compared with the standard opalescence obtained by the fixed quantity of the sulphate under the same experimental conditions.
- Test solution may contain small amount of potassium sulphate, which increases the sensitivity of test. Any sulphate ion impurity in test substance will
- produce barium Sulphate in excess of already dissolved amount, causing turbidity.
- To prevent supersaturation of barium sulphate, which may occur, small amount of ethanol is added.

- The opalescence produced by test substance should be less intense than that of standard substance to pass the limit test for chloride.

$BaCl_2 + Na_2SO_4 - BaSO_4 \downarrow + 2NaCl$

White ppt.

Procedure :

- **Preparation of ethanolic sulphate standard solution:** Dilute 1 volume of 0.181% w/w of potassium sulphate solution in 30% ethanol to 100 volumes with 30% ethanol. It gives ethanolic standard sulphate solution of 10 Ppm.
- **Preparation of sulphate standard solution:** Dilute 1 volume of 0.181% w/v of potassium sulphate solution in distilled water to 100 volumes with distilled water It gives standard sulphate solution of 10 ppm.
- **Preparation of test opalescence:** To 1.0 ml of a 25.0 % w/v solution of barium chloride in a Nessler's cylinder, add 1.5 ml of ethanolic sulphate standard solution (10 ppm SO_4), mix and allow to stand for 1 minute. Add 15 ml of the solution prepared as directed in the monograph or a solution of the specified quantity of the Substance under examination in 15 ml of water and 0.15 ml of 5 M acetic acid. Add suficient water to produce 50 ml solution. Stir immediately with a glass rod and allow to stand for 5 minutes.
- **Preparation of standard opalescence:** To 1.0 ml of a 25.0 % w/v solution of Barium chloride in a Nessler's cylinder, add 1.5 ml of ethanolic sulphate standard Solution (10 ppm SO_4). Mix and allow to stand for 1 minute. Add 15 ml of sulphate standard solution (10 ppm SO_4) in 15 ml of water and 0.15 ml of 5 M acetic acid. Add sufficient water to produce 50 ml solution. Stir immediately with a glass rod and allow to stand for 5 minutes.

Observation: After 5 minutes, view both standard and test cylinder transversely, against a black background.

Result: The opalescence of both solutions is compared. The given compound Passed pass the limit test for sulphate.

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