

Aim: Limit Test for Chlorides and Sulphates

References:

1. Indian Pharmacopoeia (IP) 2022, Vol I & II, Limit Tests.
2. British Pharmacopoeia (BP) 2023, Appendix VIII, Limit Tests.
3. United States Pharmacopoeia (USP) 43-NF 38, General Chapter <231> Heavy Metals.

Objective:

To determine the presence of chlorides and sulphates as impurities in a given pharmaceutical substance and to ensure they are within permissible limits as per the standards provided in pharmacopoeias (such as IP, BP, USP).

Materials and Reagents:

1. For Chlorides:

- Nitric acid (HNO_3)
- Silver nitrate solution (AgNO_3)
- Standard sodium chloride solution (NaCl)
- Purified water
- Nessler cylinder

2. For Sulphates:

- Hydrochloric acid (HCl)
- Barium chloride solution (BaCl_2)
- Standard potassium sulphate solution (K_2SO_4)
- Purified water
- Nessler cylinder

Apparatus:

- Nessler cylinders (50 mL)
- Measuring cylinder

- Glass rod
- Beakers (100 mL)
- Pipettes
- Analytical balance

Procedure

1. Limit Test for Chlorides:

Preparation of Standard Solution:

- **Standard Sodium Chloride Solution (0.05845% w/v):** Dissolve 58.45 mg of sodium chloride (NaCl) in sufficient purified water to make 100 mL of solution.

Test Solution Preparation:

- **Sample Preparation:** Dissolve the specified quantity of the test substance in 15 mL of water. If insoluble, use a specified solvent to dissolve the sample.
- Add 1 mL of nitric acid to the test solution.
- Make up the volume to 50 mL with purified water.

Blank Preparation:

- Prepare a blank using the same quantity of reagents without the sample.

Procedure:

1. Transfer 10 mL of the test solution into a Nessler cylinder.
2. Add 1 mL of dilute nitric acid and 1 mL of silver nitrate solution.
3. Make up the volume to 50 mL with purified water and stir well.
4. Repeat the above steps with the standard sodium chloride solution.
5. Allow the solutions to stand for 5 minutes and observe the opalescence or turbidity formed in the solutions against a dark background.

Observation:

- The opalescence or turbidity produced in the test solution should not be more than that produced in the standard solution.

2. Limit Test for Sulphates:

Preparation of Standard Solution:

- Standard Potassium Sulphate Solution (0.1089% w/v): Dissolve 108.9 mg of potassium sulphate (K_2SO_4) in sufficient purified water to make 100 mL of solution.

Test Solution Preparation:

- **Sample Preparation:** Dissolve the specified quantity of the test substance in 15 mL of water or as directed in the pharmacopeia.

- Add 2 mL of dilute hydrochloric acid to the test solution.

- Make up the volume to 50 mL with purified water.

Blank Preparation:

- Prepare a blank using the same quantity of reagents without the sample.

Procedure:

1. Transfer 10 mL of the test solution into a Nessler cylinder.
2. Add 2 mL of hydrochloric acid and 1 mL of barium chloride solution.
3. Make up the volume to 50 mL with purified water and stir well.
4. Repeat the above steps with the standard potassium sulphate solution.
5. Allow the solutions to stand for 5 minutes and observe the opalescence or turbidity formed in the solutions against a dark background.

Observation:

- The opalescence or turbidity produced in the test solution should not be more than that produced in the standard solution.

Conclusion

The test ensures that the chloride and sulphate impurities in the sample are within the permissible limits as specified in the pharmacopeias. This is an essential quality control measure to guarantee the safety and efficacy of pharmaceutical substances.

Result: The sample passes the limit test if the opalescence or turbidity of the test solution is not greater than that of the standard solution.

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