

PHARMACEUTICAL INORGANIC CHEMISTRY- BP104T

UNIT: 1 (IMPURITIES IN PHARMACEUTICAL SUBSTANCES)

CLASS: 4

TOPIC: LIMIT TEST FOR CHLORIDES & SULPHATES

Limit Tests

Limit = a value or amount that is likely to be present in a substance

Test = to examine or to investigate

Limit test is defined as quantitative or semi quantitative test designed to identify and control small quantities of impurity which is likely to be present in the substance. Limit test is generally carried out to determine the inorganic impurities present in compound.

In short, limit test is nothing but to identify the impurities present in the substance and compare it with standard.

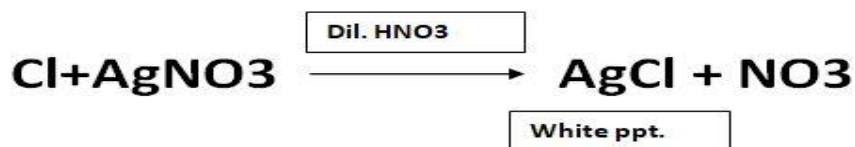
Importance of Limit tests:

- To find out the harmful amount of impurities
- To find out the avoidable/unavoidable amount of impurities

1. LIMIT TEST FOR CHLORIDES

Principle:

Limit test of chloride is based on the reaction of soluble chloride with silver nitrate in presence of dilute nitric acid to form silver chloride, which appears as solid particles (Opalescence) in the solution.



Procedure:

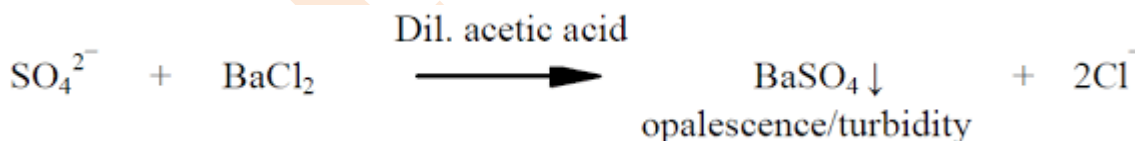
Test	Standard
Specific weight of compound is dissolved in water or solution is prepared as directed in the pharmacopoeia and transferred in Nessler cylinder	Take 1ml standard solution of chloride in Nessler cylinder
Add 10 ml of nitric acid	Add 10 ml of nitric acid
Dilute to 50ml in Nessler's cylinder	Dilute to 50ml in Nessler's cylinder
Add 1ml of AgNO ₃ solution	Add 1ml of AgNO ₃ solution
Keep aside for 5 min	Keep aside for 5 min
Observe the Opalescence/Turbidity	Observe the Opalescence/Turbidity

Dilute nitric acid is used to prevent the precipitation of other radicals like CO³⁻, HCO³, OH^{etc.}, may be the impurities other than chloride. Thus dilute nitric acid only helps to precipitate the silver chloride and also prevents the reversible reaction. AgNO₃ is used because all the metallic halides are soluble in water except AgX (X=F, Cl, Br, I)

Observation: The opalescence produce in sample solution should not be greater than standard solution. If opalescence produces in sample solution is less than the standard solution, the sample will pass the limit test of chloride and vice versa.

2. LIMIT TEST FOR SULPHATES**Principle:**

Limit test of sulphate is based on the reaction of soluble sulphate with barium chloride in presence of dilute hydrochloric acid to form barium sulphate which appears as solid particles (turbidity) in the solution.

**Procedure:**

Test	Standard
Take 1ml of 25%w/v BaCl ₂	Take 1ml of 25%w/v BaCl ₂
Add 1.5ml of ethanolic sulphate solution mix well and stand for 1minute	Add 1.5ml of ethanolic sulphate solution mix well and stand for 1minute
Add 1gm of sample and make up to 50ml with	Add 15ml of 0.1089%w/v of potassium

distilled water.	sulphate solution.
Add 0.15ml of 5M acetic acid	Add 0.15ml of 5M acetic acid
Make up to 50ml with distilled water	Make up to 50ml with distilled water
Keep aside for 5 min	Keep aside for 5 min
Observe the Opalescence/Turbidity	Observe the Opalescence/Turbidity

Barium sulphate reagent contains barium chloride, sulphate free alcohol and small amount of potassium sulphate.

Preparation of ethanolic sulphate standard solution (10 ppm SO_4^{2-}): Dilute 1 volume of a 0.181% 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 0.181% w/v solution of potassium sulphate in distilled water to 100 volumes with distilled water.

Reasons:

- Hydrochloric acid or acetic acid helps to make solution acidic and prevents turbidity due to other carbonate impurities.
- Potassium sulphate is used to increase the sensitivity of the test by giving ionic concentration in the reagent
- Alcohol helps to prevent super saturation and produces uniform turbidity/opalescence

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.

2 MARKS QUESTIONS

1. Write the principle involved in limit test for chlorides
2. Write the principle involved in limit test for sulphates

5 MARKS QUESTIONS

1. Write the principle & procedure involved in limit test for chlorides
2. Write the principle & procedure involved in limit test for sulphates