

Introduction to Limit Test

Limit test is defined as quantitative or semi-quantitative test that are designed to identify and control the small quantities of impurities which are likely to be present in drugs or pharmaceutical substances.

The most common acid radical in drugs and pharmaceuticals are chlorides and sulphides which generally arise from the use of tap water. If we are using tap water the other impurities may be interfere in the limit test. The impurities are foreign undesirable substances which are present in very trace amount, but their presence may affect pharmaceutical preparation.

Limit tests are performed in a special glassware called Nessler cylinders.

The impurities are classified into two types:

Soluble impurities: These include chlorides, sulphates, calcium and magnesium.

Insoluble impurities: These include metallic impurities such as iron, zinc, arsenic and heavy metals.

EXPERIMENT

1

Limit Test for Chlorides

Aim

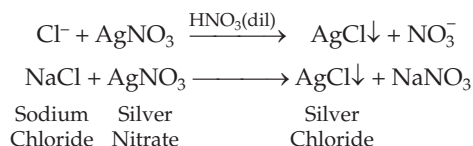
To perform the limit test for chlorides.

Requirements

Apparatus: Nessler cylinder, measuring cylinder, glass rod, pipette.

Chemicals: 0.5845% w/v NaCl solution (standard solution), dilute HNO₃, AgNO₃ solution, distilled water.

Principle: Soluble chlorides react with soluble silver nitrate in the presence of dilute HNO₃ to give an insoluble precipitate of silver chloride. The precipitate is observed in the form of opalescence in the test solution. The turbidity of the test solution is compared with the standard solution. If it is less than or equal to the standard, the test is passed; if it is more, the test is failed.



Procedure

Preparation of standard solution: Pipette out 1 mL of 0.5845% w/v NaCl solution into a Nessler cylinder. Then add 1 mL of dilute HNO₃ and 1 mL of AgNO₃ solution. Make up the volume to 50 mL with distilled water. Stir the solution with a glass rod and keep it aside for 5 minutes.

Preparation of test solution: Weigh accurately 1 g of the test sample and dissolve it in 10 mL of distilled water. Pipette out 1 mL of the test solution into a Nessler cylinder. Add 1 mL of dilute HNO₃ and 1 mL of AgNO₃ solution. Make up the volume to 50 mL with distilled water. Stir the solution using a glass rod and keep it aside for 5 minutes.

Observation

The opalescence present in the test sample was less than the standard sample.

Inference

The given sample passes the limit test for chlorides.

Limit Test for Sulphates

Aim

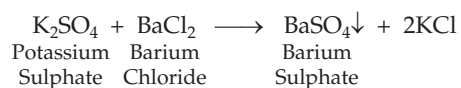
To perform the limit test for sulphates.

Requirements

Apparatus: Nessler cylinders, beaker, glass rod, measuring cylinder, pipette.

Chemicals: Standard K_2SO_4 solution, dilute HCl, barium sulphate reagent, distilled water.

Principle: The limit test for sulphates is designed to identify the presence of sulphate as an impurity. Soluble sulphates react with soluble barium chloride in the presence of dilute HCl to form insoluble barium sulphate precipitate. The precipitate is observed in the form of opalescence. The turbidity of the test solution is compared with the standard solution. If it is less than or equal to the standard, the test is passed; if it is more, then the test is failed.



Procedure

Preparation of standard solution: Pipette out 1 mL of standard K_2SO_4 solution into a Nessler cylinder. Then add 2 mL of dilute HCl and 5 mL of barium sulphate reagent. Make up the volume to 50 mL with distilled water, stir the solution with a glass rod and keep it aside for 5 minutes.

Preparation of test solution: Weigh 1 g of the test sample and dissolve it in 20 mL of distilled water. Pipette out 1 mL of this solution into a Nessler cylinder. Then add 2 mL of dilute HCl and 5 mL of barium sulphate reagent. Make up the volume to 50 mL with distilled water, stir the solution with a glass rod and keep it aside for 5 minutes.

Observation

The opalescence present in the test sample was less than the standard sample.

Inference

The given sample passes the limit test for sulphates.