

PHARMACEUTICAL INORGANIC CHEMISTRY- BP104T

UNIT: 1 (IMPURITIES IN PHARMACEUTICAL SUBSTANCES)

CLASS: 10

TOPIC: MODIFIED LIMIT TEST FOR SULPHATES

Principle:

Modified limit test is performed; if the limit tests for a sample (Colored compound) cannot be done by normal method. For example, potassium permanganate is de-colored by boiling with ethanol, filtered to remove precipitated manganese dioxide and the filtrate is subjected to the test. 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 sulphate. The barium chloride has been replaced by barium sulphate reagent which is having barium chloride, sulphate-free alcohol, and a solution of potassium sulphate. Potassium sulphate has been added to increase the sensitivity of the test. The ionic concentrations in the reagent has been so adjusted that the solubility product of barium sulphate gets exceeded, and the very small amount of barium sulphate present in the reagent acts as a seeding agent for precipitation of barium sulphate, if sulphate be present in the substance under test. Alcohol helps to prevent super-saturation and thus produces a more uniform opalescence/turbidity. Acetic acid helps to make solution acidic and barium sulphate precipitate formed is insoluble which gives turbidity/opalescence.

Procedure:

Preparation of Test Solution (KMnO₄): Dissolve 1.5 g in 50 mL of distilled water, heat on a water-bath and add gradually 6 mL of ethanol (95%), cool, dilute to 60 mL with distilled water and filter. Take two 50 ml. Nessler's Cylinders. Label one as "Test" and the other as 'Standard'

Test	Standard
Take 10ml of the above solution into a Nessler's cylinder	Mix 15ml of sulphate standard solution and 15ml of distilled water in a nessler's cylinder.

Add 0.15ml of 5M acetic acid solution	Add 0.15ml of 5M acetic acid solution
Dilute to 50ml with distilled water	Dilute to 50ml with distilled water
Add 5ml of barium sulphate reagent	Add 5ml of barium sulphate reagent
Make up to 50ml with distilled water	Make up to 50ml with distilled water
Stir with a glass rod and allow it to stand for 5 minutes protected from light.	Stir with a glass rod and allow it to stand for 5 minutes protected from light.

2 MARKS QUESTIONS

1. Write the principle involved in modified limit test for sulphates

5 MARKS QUESTIONS

1. Explain the principle and procedure involved in modified limit test for sulphates

10 MARKS QUESTIONS

1. Write the principle, procedure, reaction involved in modified limit test for sulphates