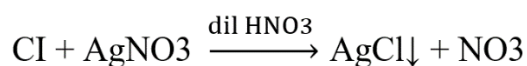


LIMIT TEST

Limit tests are quantitative or semi-quantitative tests designed to identify and control small quantities of impurity, which are likely to be present in the substance.

LIMIT TEST FOR CHLORIDES**PRINCIPLE:**

The limit test for chlorides is based on simple reaction between silver nitrate and soluble chlorides to obtain silver chloride which is insoluble in dilute nitric acid, which extracts a common ion effect by furnishing nitrate ions and thereby suppression of dissociation of silver chloride. The opalescence produced in the test solution is compared with the opalescence produced in the standard (sodium chloride) solution treated similarly 0.05845% w/v solution of sodium chloride is taken as standard.

**PROCEDURE:**

Test sample	Standard compound
Take 1g of given sample in Nessler cylinder. and add 5ml of distilled water.	Take 1ml of 0.05845 % W/V solution of sodium chloride in Nessler cylinder
Add 10ml of dil. nitric acid	Add 10ml of dil. nitric acid
Add 1ml of 0.1M AgNO ₃ solution.	Add 1ml of 0.1M AgNO ₃ solution.
Dilute to 50ml with water.	Dilute to 50ml with water.
Keep aside for 5 mins.	Keep aside for 5 mins.
Observe the Opalescence/ Turbidity.	Observe the Opalescence/ Turbidity.

Reasons:

Nitric acid is added in the limit test of chloride to make solution acidic and helps silver

chloride precipitate to make solution turbid at the end of process.

LIMIT TEST FOR SULPHATES**PRINCIPLE:**

The limit test for sulphate is based on the reaction between barium chloride and soluble sulphates in presence of dilute hydrochloric acid. Then the turbidity produced is compared with the standard turbidity obtained from known amount of barium sulphate reagent.

Barium sulphate reagent contains barium chloride, sulphate free alcohol and potassium sulphate in water.

Potassium sulphate is used to increase the sensitivity of the test. The ionic concentration has been so adjusted, such that the solubility product barium sulphate get exceeded and very small amount of barium sulphate acts as seeding agent for precipitation of barium sulphate alcohol used to prevent super saturation and thus produces uniform turbidity.

**PROCEDURE:**

Test sample	Standard compound
Dissolve the given 1g sample with sufficient quantity of water in Nessler's cylinder.	Take 1ml of 0.1089 % W/V solution of potassium sulphate in Nessler cylinder
Add 2ml of dilute hydrochloric acid	Add 2ml of dilute hydrochloric acid
Dilute to 45 ml with water.	Dilute to 45 ml with water.
Add 5ml of barium sulphate reagent	Add 5ml of barium sulphate reagent
Keep aside for 5 min	Keep aside for 5 min
Observe the Turbidity	Observe the Turbidity

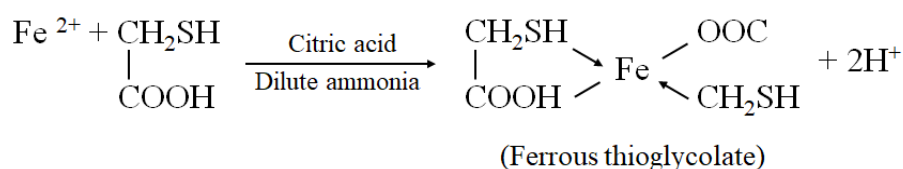
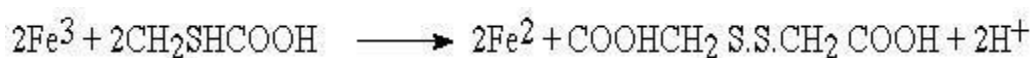
Barium sulphate reagent contains barium chloride, sulphate free alcohol and small amount of potassium sulphate.

Reasons:

Hydrochloric acid helps to make solution acidic. Potassium sulphate is used to increase the sensitivity of the test by giving ionic concentration in the reagent. Alcohol helps to prevent super saturation.

LIMIT TEST FOR IRON**PRINCIPLE:**

The limit test for iron is based on the reaction of iron in ammoniacal solution in the presence of citric acid with thioglycolic acid. When a pale pink to deep reddish purple colour is formed due to the ferrous complex. The color produced from a specific amount of substance from test is compared with the color produced in standard solution by viewing vertically. Ferric ammonium sulphate is used as standard. Thioglycolic acid is reducing agent (reducing ferric to ferrous) and also forms complex with ferrous ion. Citric acid prevents the precipitation of iron by ammonia. Ferrous thioglycolate complex is colorless in acidic or neutral solutions. Only in the presence of alkali pale pink color produced.

**PROCEDURE:**

Test sample	Standard compound
Take 2g of sample and dissolve it in specific amount of water and then volume is made up to 40 ml	2 ml of standard solution of iron diluted with water upto 40ml
Add 2 ml of 20 % w/v of citric acid (iron free)	Add 2 ml of 20 % w/v of citric acid (iron free)

Add 2 drops of thioglycollic acid	Add 2 drops of thioglycollic acid
Add ammonia to make the solution alkaline and adjust the volume to 50 ml	Add ammonia to make the solution alkaline and adjust the volume to 50 ml
Keep aside for 5 min	Keep aside for 5 min

Compare the color produced in test solution with standard solution.

Reasons:

Citric acid helps precipitation of iron by ammonia by forming a complex with it.

Thioglycolic acid helps to oxidize iron (II) to iron (III).

Ammonia to make solution alkaline.