Limit Test

Limit: a value or amount that is likely to be present in a substance.
Test: to examine or to investigate.
Impurities: a foreign matter present in a compound.

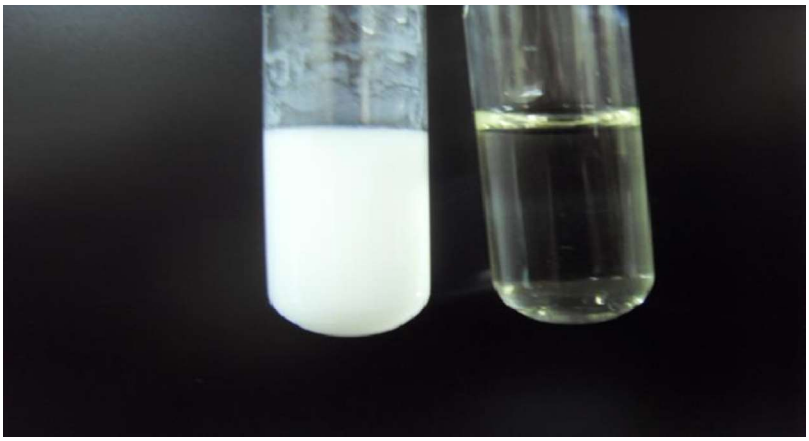
"Limit test is defined as quantitative or semi-quantitative test designed to identify and control small quantities of impurities which is likely to be present in the compound".

Limit test is generally carried out to determine the inorganic impurities present in compound.

Types:

- Tests in which there is no visible reaction
- Comparison methods
- Quantitative determination

Turbidity: is a measure of the degree to which the water loses its transparency due to the presence of suspended particulates.



Nessler cylinders

Limit test of Chlorides

Principle:

Limit test of chloride is based on the reaction of between silver nitrate and soluble chlorides to obtain silver chloride which is insoluble in dilute nitric acid.

$$\underset{\text{(Soluble)}}{\text{Chlorides}} + \underset{\text{(Soluble)}}{AgNO_3} \xrightarrow{HNO_3} \underset{\text{(Precipitate)}}{AgCl} + \underset{\text{(Soluble)}}{NO_3^-}$$

$$NaCl + AgNO_3 \xrightarrow{HNO_3} AgCl + NaNO_3$$

The silver chloride produced in the presence of dil. nitric acid makes the test solution turbid, the extent of turbidity depending upon the amount of chloride present in the substance is compared with the standard turbidity produced by addition of silver nitrate to the standard solution having the known amount of chloride.

- 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. It also prevents the precipitation of other acid radicals such as phosphate, sulphate etc.

Apparatus and Chemicals:

Apparatus Required

a) Nessler cylinders
b) Glass rod
c) Stand

Chemicals Required

a) Dilute Nitric acid (10%)
b) Silver nitrate (5%)
c) Sodium chloride

Procedure:

Method of limit test for Chlorides I.P. 1985		
S.No.	Test	Standard
1.	Dissolve specified weight (1 g) in about 10 ml of distilled water and transfer it to a Nessler cylinder labelled as 'Test'.	Take 1 ml of 0.05845 % w/v solution of sodium chloride in Nessler cylinder labelled as 'Standard'.
2.	Add 10 ml of dil. Nitric acid	
3.	Dilute to 50 ml mark with distilled water.	
4.	Add 1 ml of 0.1 M $AgNO_3$ solution.	
5.	Stir properly with glass rod and keep aside for 5 min	

- Compare the turbidity transversely against a dark (preferably Black) background.

Observation:

If the opalescence/ turbidity produced in the 'Test' (sample) is more as compared to in the 'Standard' then it means that sample contains more quantity of chloride (impurities) than the prescribed limit.

Modified Chloride Limit Test

- With reference to International Pharmacopoeia 6th Edition, 2016 the limit test has been modified in the context of standard solution preparation. Earlier the standard solution of chloride was prepared by dissolving NaCl (Known Cl^- impurity) but now it has been modified by using HCl instead of NaCl.

Limit test of Sulphates

Principle:

Limit test of Sulphates depends upon the interaction of sulphates with barium chloride in the presence of hydrochloric acid. This results in the precipitation of sulphates as barium sulphate.

$$\underset{\text{(Soluble)}}{BaCl_2} + \underset{\text{(Soluble)}}{\text{Sulphates}} \xrightarrow{\text{dil. HCl}} \underset{\text{(precipitate)}}{BaSO_4} + \underset{\text{(Soluble)}}{\text{Chlorides}}$$

$$BaCl_2 + Na_2SO_4 \xrightarrow{\text{dil. HCl}} BaSO_4 + 2NaCl$$

When only very small quantity of sulphate ions are present, Barium sulphate appears as turbidity which is compared under uniform conditions of illumination with standard turbidity in Nessler cylinder.

Note: Hydrochloric acid is added to prevent precipitation of the other acid radicals by common ion effect.

Procedure:

Limit test for sulphate		
S.No.	**Test**	**Standard**
1.	Dissolve specified quantity of sample in about 10 ml distilled water and transfer it to a Nessler cylinder labelled as 'Test'.	Place 1 ml of 0.1089 w/v solution of potassium sulphate in Nessler cylinder labelled as 'Standard'. Add about 9ml of distilled water.
2.	Add 2 ml of dil. HCl	

3.	Dilute to 45 ml with distilled water.
4.	Add 5 ml of 'Barium sulphate reagent'
5.	Stir each solution with a glass rod and allow to stand for 5 minutes. Compare the turbidity transversely.

- According to I.P. 1985 'Barium sulphate reagent' is used instead of solution of barium chloride alone in the test.

To the 15 ml of 0.5 M Barium chloride solution add 55 ml of water, 20 ml of alcohol and 5 ml of 0.0181% w/v solution of potassium sulphate and the final volume was made upto 1000 ml.

- Barium sulphate reagent is used because:

i) presence of small portion of potassium sulphate increases sensitivity of this test.

ii) alcohol helps to prevent supersaturation and keep precipitated barium sulphate in the form of turbidity.

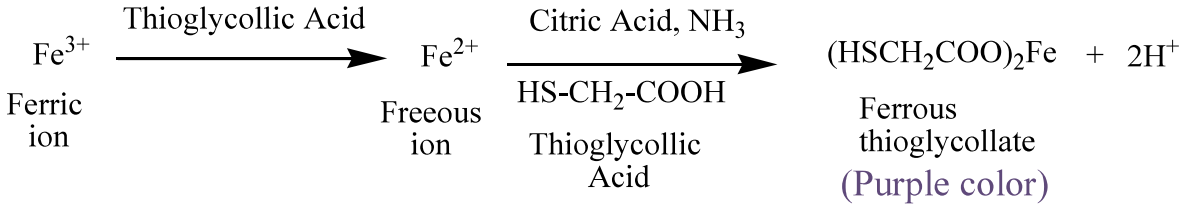
Observation:

If the opalescence/turbidity produced in the 'Test' (sample) is more as compared to in the 'Standard' then it means that sample contains more quantity of chloride (impurities) than the prescribed limit.

Limit test of Iron

Principle:

- Limit test of iron based on the interaction of iron (Fe^{2+}) with thioglycollic acid in the presence of citric acid and ammonia. This results in the formation of purple colored Ferrousthioglycollate.



- Thioglycollic acid performs the following two functions:

I) Iron impurities may be present in the trivalent ferric form (Fe^{3+}) or in the divalent ferrous form (Fe^{2+}). If it is present in ferric form then thioglycollic acid reduces it to ferrous form.

$$Fe^{3+} + 2HSCH_2COOH \longrightarrow Fe^{2+} + 2HSCH_2COO^- + 2H^+$$

II) Thioglycollic acid produces purple color with the ferrous iron in the ammonical alkaline medium and in presence of citric acid.

- Citric acid prevents precipitation of iron with ammonia as iron hydroxides. It keeps iron in the solution form even in the presence of ammonia by forming a complex.

$$2Fe + 10NH_3 \longrightarrow 2Fe(NH_2)_5 + 5H_2$$

$$2C_6H_8O_4 + Fe \longrightarrow Fe(C_6H_6O_4)_2 + 2H_2$$

Procedure:

Test sample	Standard compound
1. Place Sample dissolved in specific amount of waterand then volume is made up to 40 ml with distilled water in a Nessler cylinder labelled as 'Test'.	1. Place 2 ml of standard iron solution (20 ppm Fe) in a Nessler cylinder labelled as 'Standard' and diluted with distilled water up to 40ml.
2. Add 2 ml of 20 % w/v of citric acid (iron free)	
3. Add 0.1 ml of thioglycollic acid	
4. Add ammonia to make the solution alkaline andadjust the volume to 50 ml	
5. Keep aside for 5 min	
6. Color developed is viewed vertically andcompared with standard solution	

Observation:

The purple color produce in sample solution should not be greater than standard solution. If purple color produces in sample solution is less than the standard solution, the sample will pass the limit test of iron and vice versa. Means; the intensity of colour produced by sample is less than that of standard solution colour, it passes the test.