

## LIMIT TEST

Limit test are quantitative or semi-quantitative test designed to identify and control small quantities of impurity that are present in the substance.

Limit = Certain or Fix value

Test = To examine / To investigate

- It basically involves small comparison of opalescence, turbidity or colour with fixed standards.
- Generally it carried out in nessler's cylinder.

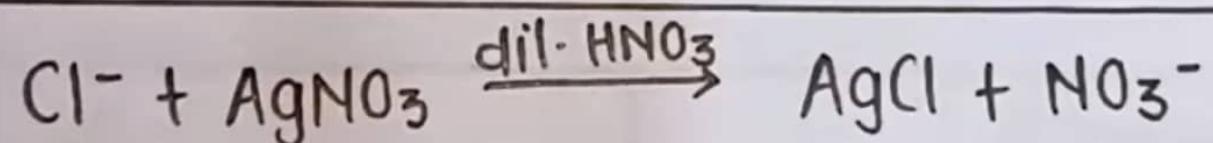
## Limit test in our syllabus

- Limit test of chloride
- Limit test of sulphate
- Limit test of Iron
- Limit test of Arsenic
- Limit test of Lead
- Limit test of Heavy Metals

## Limit Test For Chloride

### Principle :

The principle of limit test of chloride is based on the reaction of soluble chlorides with silver nitrate in the presence of dilute nitric acid to form silver chloride which appears as turbidity / opalescence .



- Nitric acid is added in the solution to make solution acidic
- Dissolve other impurities
- Provide common ion effect and help silver chloride precipitate to make solution turbid at the end of process

### Apparatus Required

- Nessler Cylinder
- Glass Rod
- Stand

### Chemical Required

Dilute Nitric Acid (10%)

[106 ml conc.  $\text{HNO}_3$  in 1000 ml water]

Silver Nitrate (5%)

[5g  $\text{AgNO}_3$  in 100 ml water]

Sodium Chloride

[0.05845 g  $\text{NaCl}$  in 100 ml water]

## Procedure

TEST	STANDARD
<ul style="list-style-type: none"><li>• Specific amount of subst. dissolved in nessler cylinder as directed in pharmacopoeia</li></ul>	<ul style="list-style-type: none"><li>• Take 1 ml of 0.0584% w/v solution of NaCl in a Nessler Cylinder</li></ul>
<ul style="list-style-type: none"><li>• Add 10 ml dil. <math>\text{HNO}_3</math></li></ul>	<ul style="list-style-type: none"><li>• Add 10 ml dilute <math>\text{HNO}_3</math></li></ul>
<ul style="list-style-type: none"><li>• Dilute the solution to 50 ml with water</li></ul>	<ul style="list-style-type: none"><li>• Dilute the solution to 50 ml with water</li></ul>

- Add 1 ml silver Nitrate solution

- Observe the opalescence/turbidity

- Add 1 ml silver Nitrate solution.

- Observe the opalescence/turbidity

### Observation

- If turbidity of test solution is less than turbidity of standard solution then sample will pass the limit test.
- If turbidity of test solution is greater than turbidity of standard solution limit test fails

## Limit Test For Sulphate

### Principle :

The principle of limit test of sulphate is based on the reaction of soluble sulphate with barium chloride to form barium sulphate in the presence of dilute hydrochloric acid which appears as turbidity / opalescence

### Apparatus Required

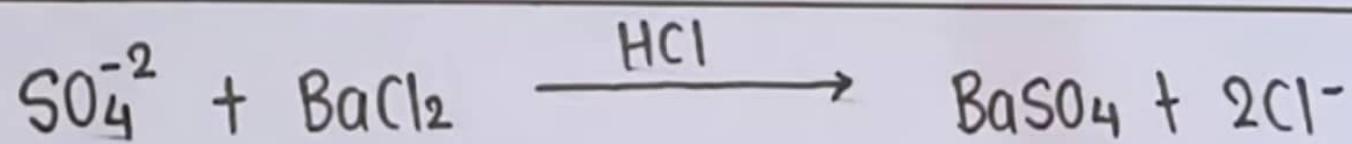
- Nessler Cylinder
- Glass Rod
- Stand

### Chemical Required

- Dilute Hydrochloric Acid
- Standard potassium sulphate sol.
- Barium sulphate reagent

[ Prepared by mixing 15 ml of 0.5 m  $\text{BaCl}_2$  + 55 ml of water  
+ 20 ml alcohol + 5 ml 0.0181% w/v  $\text{K}_2\text{SO}_4$  then diluted to  
100 ml ]

## Chemical Reaction



## TEST

- Dissolve specific amount of substance in nessler cylinder as directed in pharmacopoeia
- Add 2 ml dilute HCl
- Dilute the solution to 45 ml with water
- Add 5 ml barium sulphate reagent

## STANDARD

- 1 ml of 0.1089% w/v solution of  $K_2SO_4$  in Nessler Cylinder
- Add 2 ml dilute HCl
- Dilute the solution to 45 ml with water
- Add 5 ml barium sulphate reagent

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| <ul style="list-style-type: none"><li>• Add 5 ml barium sulphate reagent</li><li>• Observe the opalescence</li></ul> | <ul style="list-style-type: none"><li>• Add 5 ml barium sulphate reagent</li><li>• Observe the opalescence</li></ul> |
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## Observation

- If turbidity of test solution is less than turbidity of standard solution then sample will pass the limit test
- If turbidity of test solution > turbidity of standard solution limit test fails.