SNS COLLEGE OF PHARMACY AND HEALTH SCIENCES

Affiliated To The Tamil Nadu Dr. MGR Medical University, Chennai
Approved by Pharmacy Council of India, New Delhi.
Coimbatore -641035

COURSE NAME:PHARMACEUTICAL CHEMISTRY

I YEAR D PHARM

TOPIC 4: LIMIT TEST FOR CHLORIDE AND SULPHATE

Mind Map

3 TYPES

Test In Which There Is No Visible Reaction

- Testing as prescribed, there is no color, opalescence or precipitate→ Negative test indicate the absences of Large impurity

Comparison Methods

- Compare the amount of impurity in the substance with a standard of known concentration and determine whether impurity is within or the excess of the limit prescribed`

Quantitative Determination

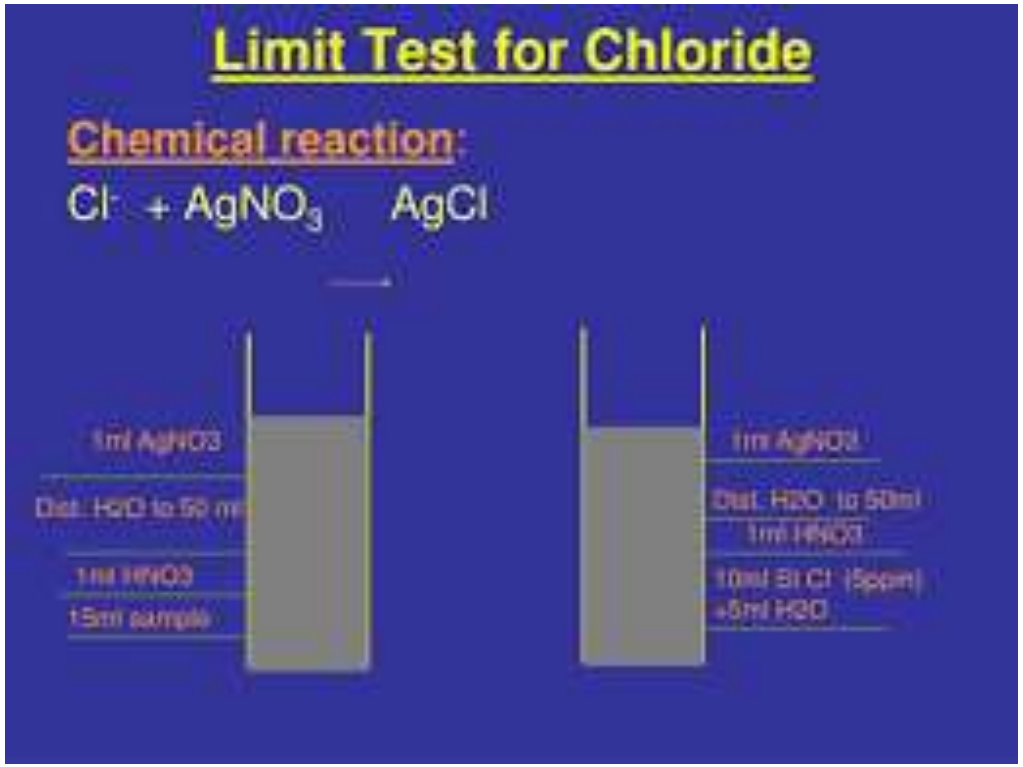
- Amount of impurity present in actually determined and compared with the numerical limit given in Pharmacopoeia

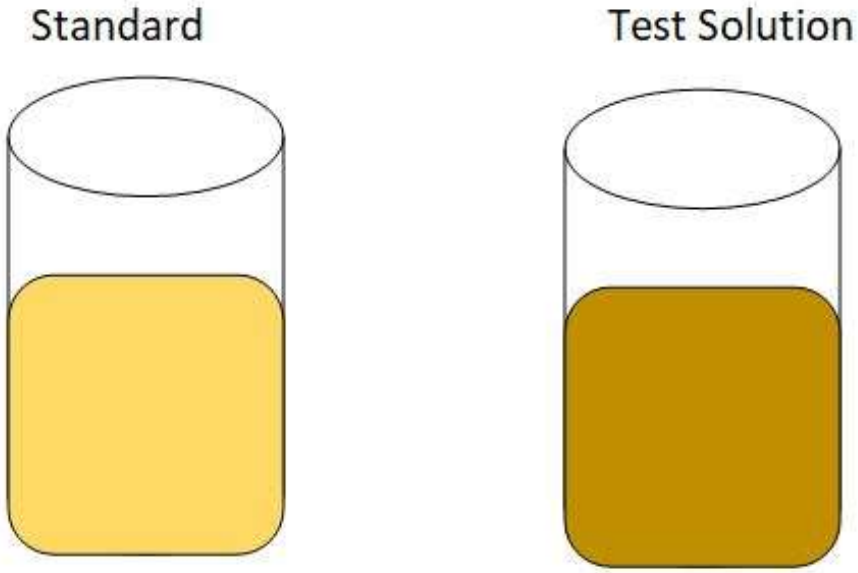
Limit Test For Chlorides

- **Principle**:

Limit test of chloride is based on the reaction of soluble chloride with silver nitrate in presence of dilute nitric acid to form silver chloride, which appears as solid particles (Opalescence) in the solution.

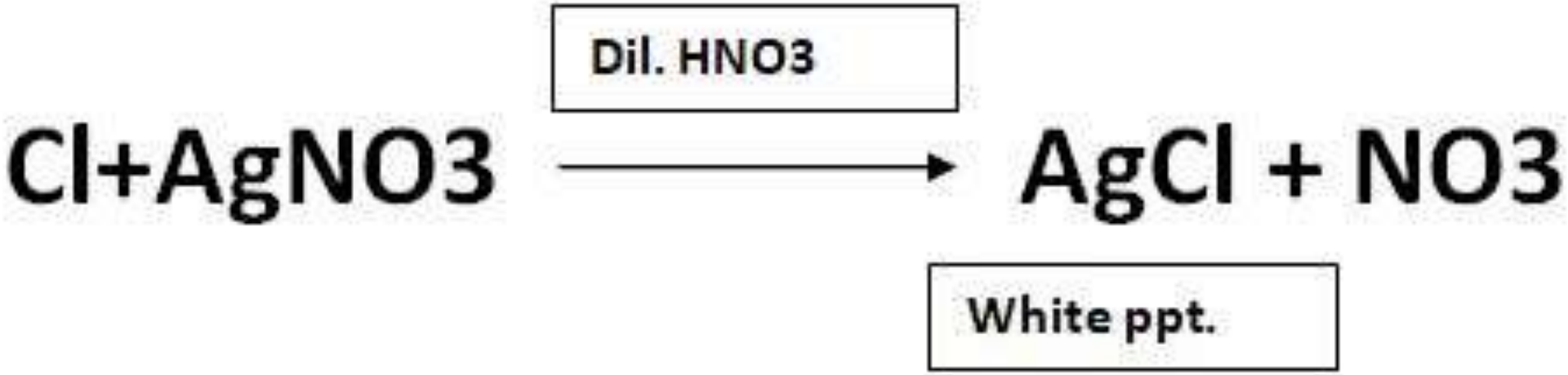
$$NaCl + AgNO_3 \xrightarrow{HNO_3} AgCl + NaNO_3$$

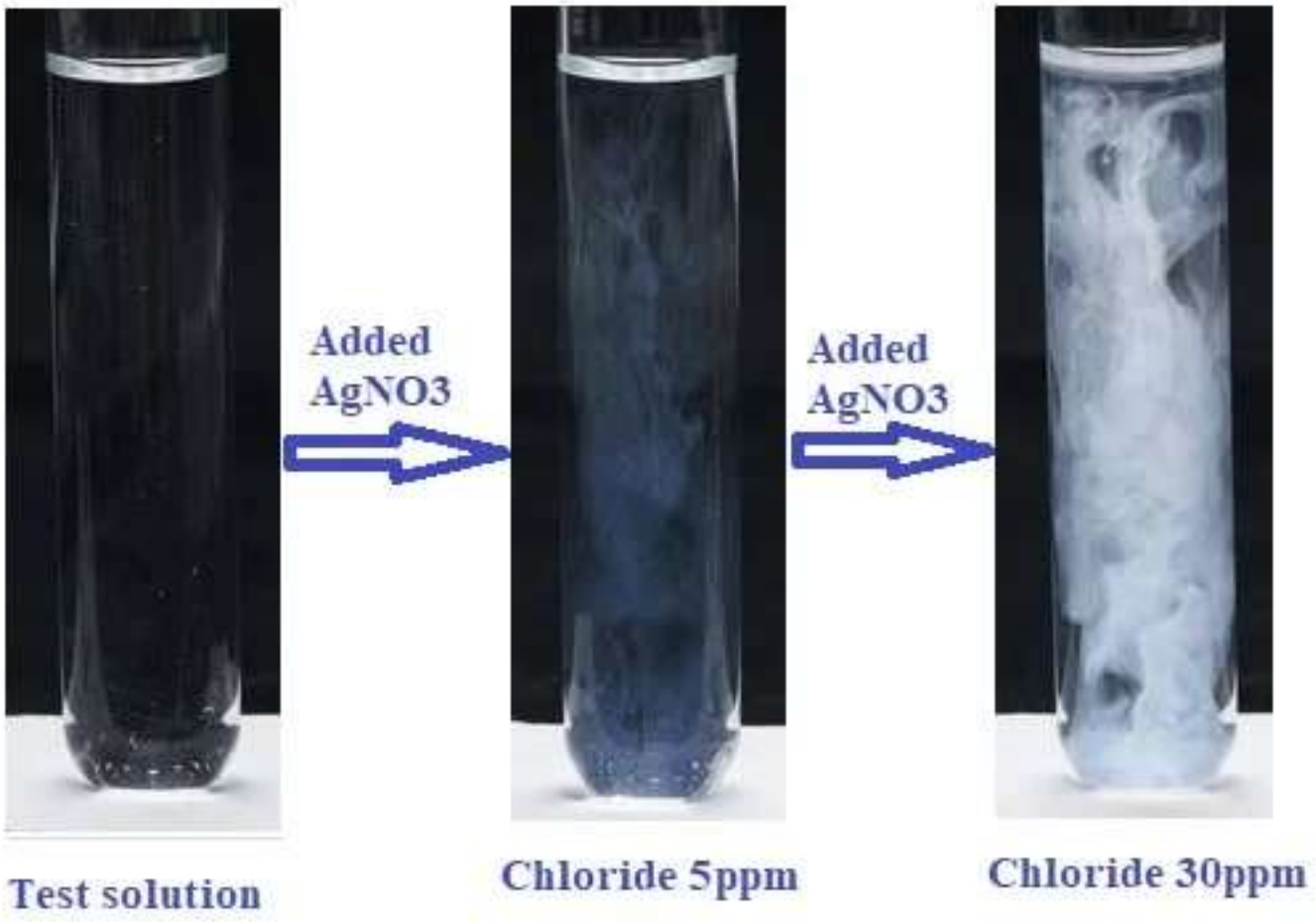

Limit Test for Chloride
Chemical reaction:
Cl⁻ + AgNO3 AgCl
1ml AgNO3
Dist. H2O to 50 ml
1ml HNO3
15ml sample
1ml AgNO3
Dist. H2O to 50ml
1ml HNO3
10ml Sl Cl (5ppm)
+5ml H2O



Limit Test failed if Turbidity of test solution is less as compare to standard

Sl. No.	Test sample	Standard sample
1.	Specific weight of compound is dissolved in water or solution is prepared as directed in the pharmacopoeia and transferred in Nessler cylinder	Take 1ml of 0.05845 % W/V solution of sodium chloride in Nessler cylinder
2.	Add 1ml of nitric acid	Add 1ml of nitric acid
3.	Dilute to 50ml in Nessler cylinder	Dilute to 50ml in Nessler cylinder
4.	Add 1ml of $AgNO_3$ solution	Add 1ml of $AgNO_3$ solution
5.	Keep aside for 5 min	Keep aside for 5 min
6.	Observe the Opalescence/Turbidity	Observe the Opalescence/Turbidity


Dil. HNO3
Cl+AgNO3
AgCl + NO3
White ppt.


Added
AgNO3
Added
AgNO3
Test solution
Chloride 5ppm
Chloride 30ppm

Apparatus required:

Nessler's cylinder

2. Limit Test for Sulphate

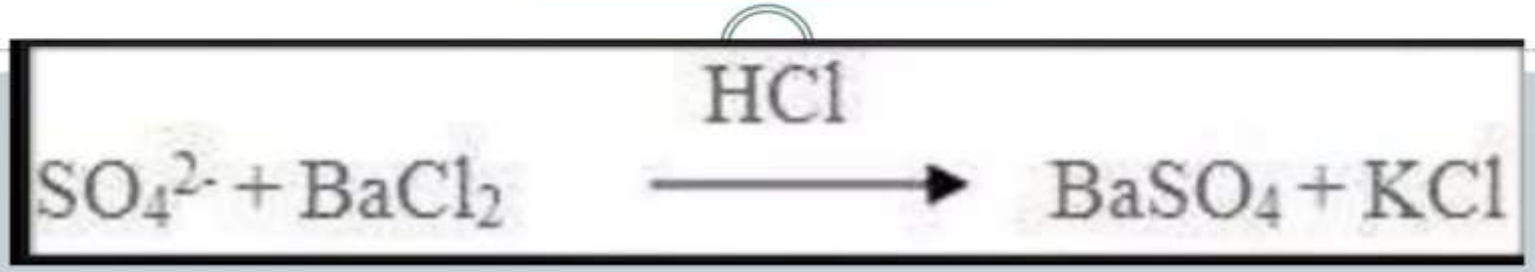
❖PRINCIPLE

- This is based on the reaction between **barium chloride** and soluble **sulphates** in presence of dilute **hydrochloric acid.**
- An opalescence is produced by the precipitation of barium sulphate and compared with the opalescence produced in a standard containing a known quantity of sulphate and similarly treated.

$$SO_4^- + BaCl_2 \rightarrow BaSO_4 + 2Cl^-$$

- Test substance passes the limit test, if the opalescence in it is less intense than that in the standard. If opalescence is found to be more, then it fails the test.

REACTION

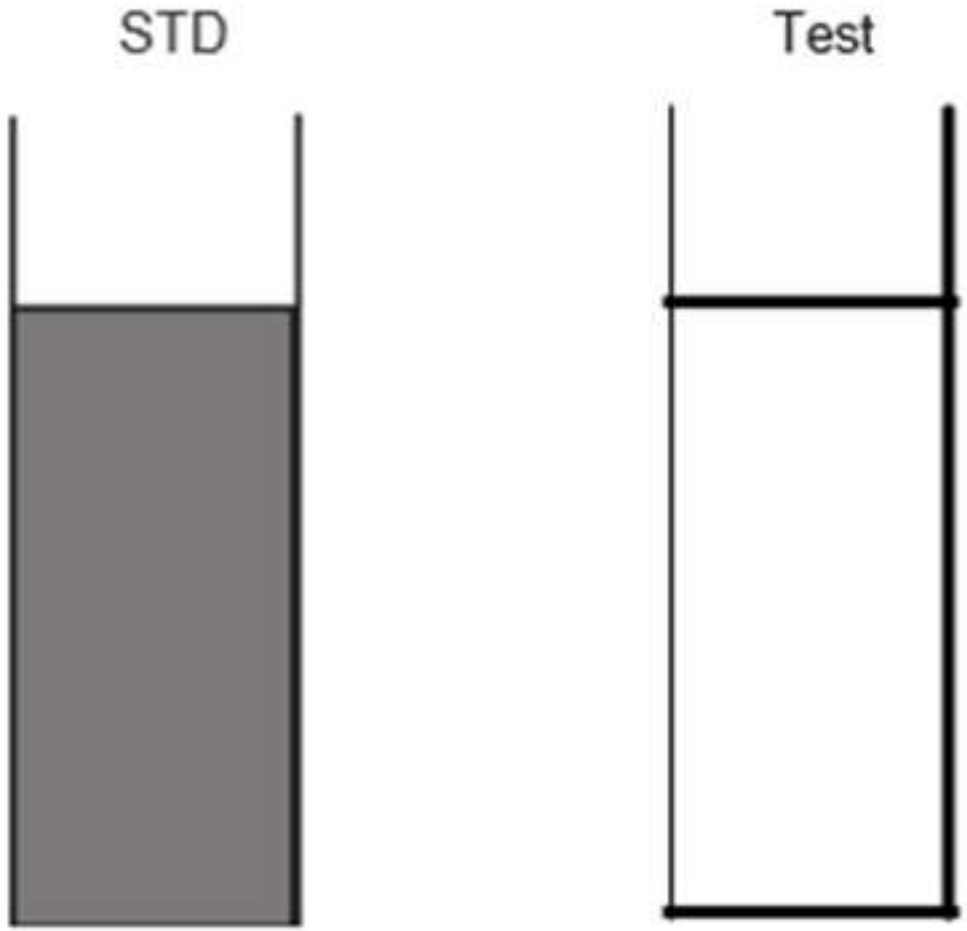


$$Na_2SO_4 + BaCl_2 \xrightarrow{HCl} BaSO_4 + 2NaCl$$

Soluble sulphates → Precipitate

Note :

- **HCl is used to prevent precipitation of other acid radicals.**
- **In the presence of HCl only sulphates precipitate.**


STD
Test

Sl. No.	Test sample	Standard sample
1.	The specified amount of compound is dissolved in water or prepared solution as per pharmacopoeia guidelines, then transferred into a Nessler cylinder.	Take 1ml of 0.1089 % W/V solution of potassium sulphate in Nessler cylinder
2.	Add 2ml of dilute hydrochloric acid	Add 2ml of dilute hydrochloric acid
3.	Dilute to 45 ml in Nessler cylinder	Dilute to 45 ml in Nessler cylinder
4.	Add 5ml of barium sulphate reagent	Add 5ml of barium sulphate reagent
5.	Keep aside for 5 min	Keep aside for 5 min
6.	Observe the Turbidity	Observe the Turbidity

$$SO_4^{2-} + BaCl_2 \xrightarrow{\text{dil } H_2SO_4} BaSO_4\downarrow + 2Cl^-$$

Limit test for Sulphate

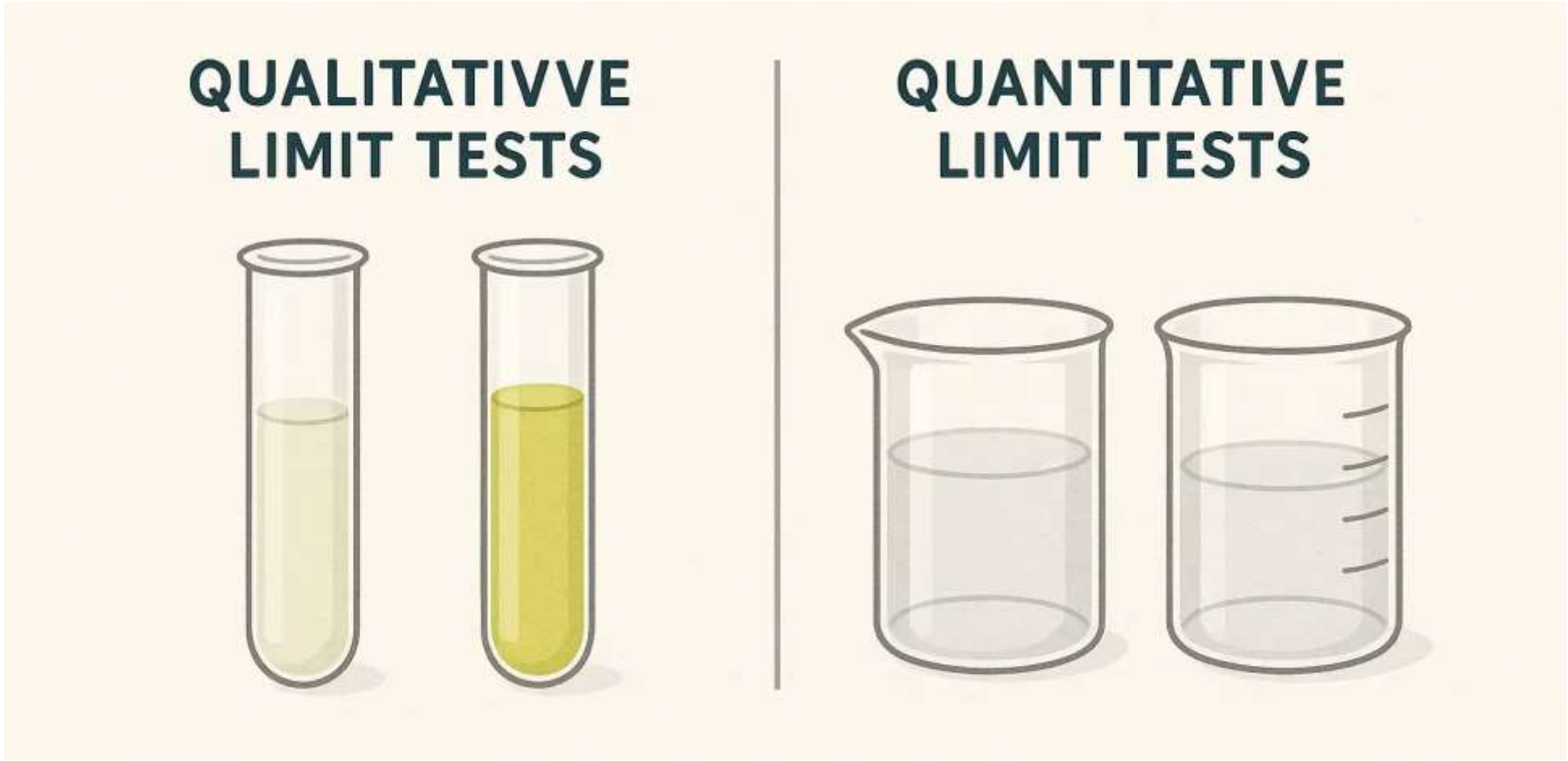
IMPORTANCE OF LIMIT TESTS

1ST ELEMENT

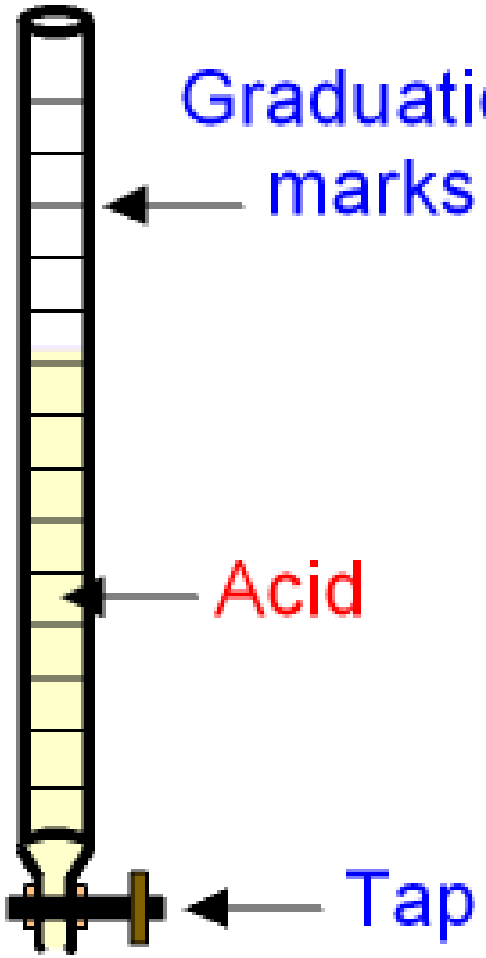
TO FIND OUT THE HARMFUL AMOUNT OF IMPURITIES

2ND ELEMENT

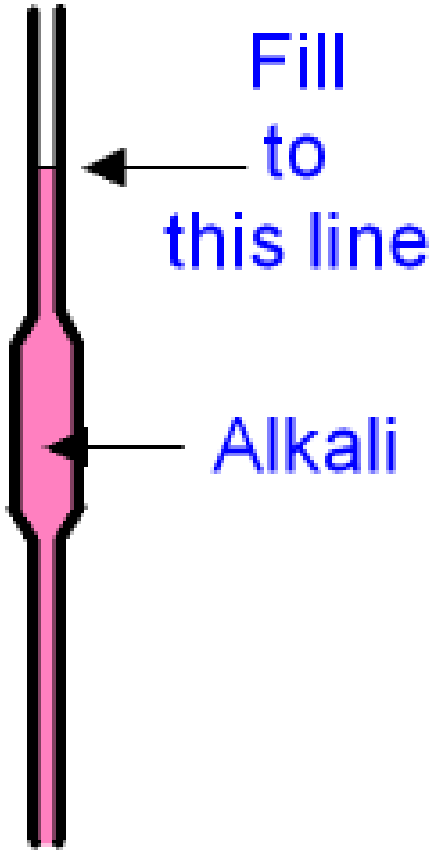
TO FIND OUT AVOIDABLE/ UNAVOIDABLE AMOUNT OF IMPURITIES


QUALITATIVVE
LIMIT TESTS
QUANTITATIVE
LIMIT TESTS

Burette



Pipette



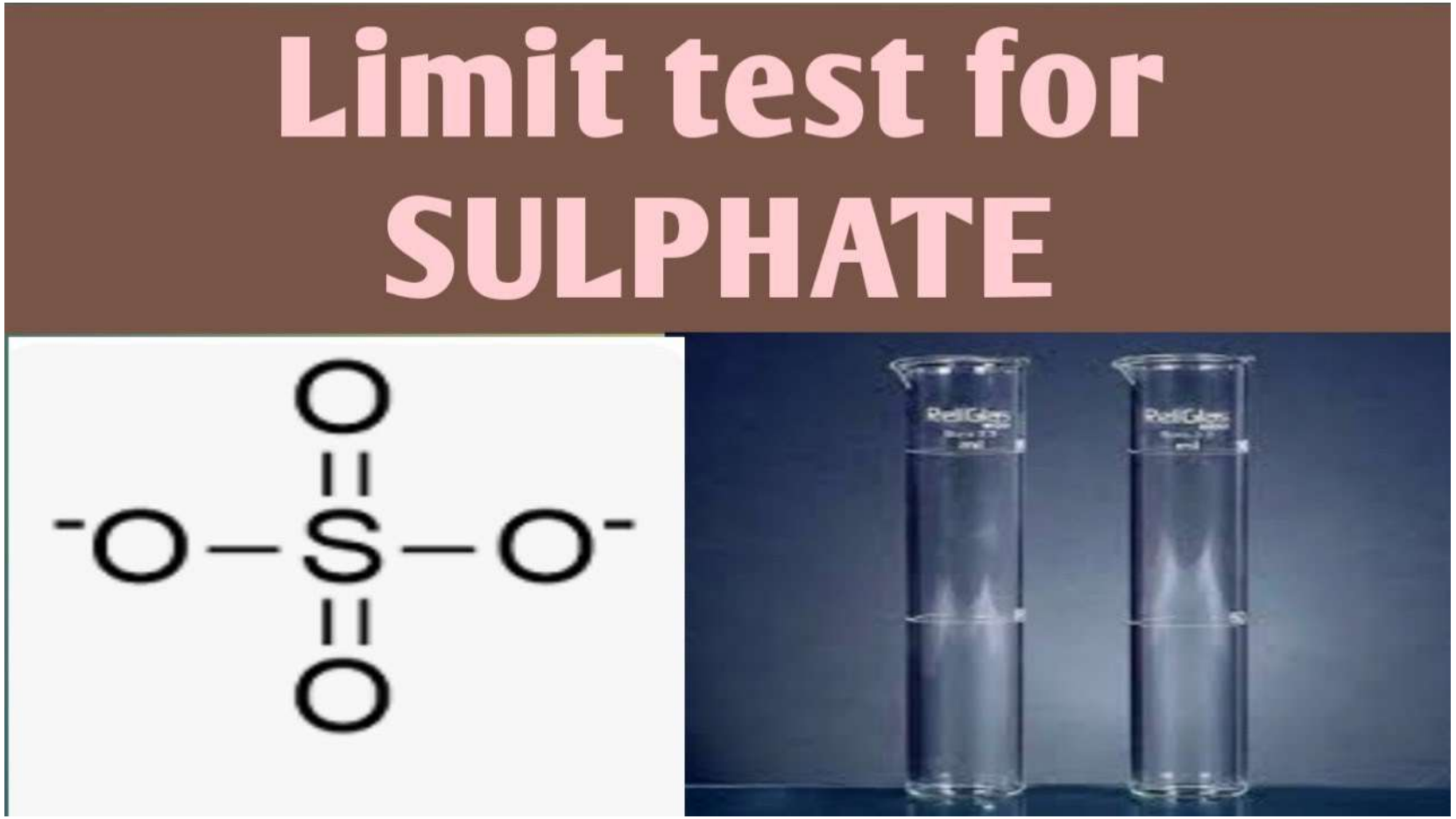
SUMMARY

❖ LIMIT TEST

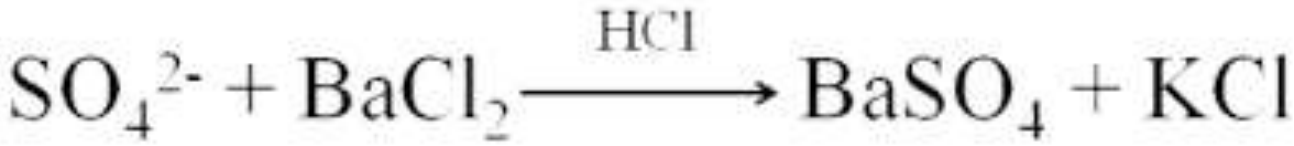
- ❑ Limit tests are **quantitative tests** or semi-quantitative tests which are designed to **detect and limit / control small quantities of impurities present in the substance.**
- ❑ All the limit tests that are prescribed in the pharmacopoeias are based on the **comparison of standard turbidity or colour** with that of the sample under test.
- ❑ Usually the limits are prescribed in **parts per million (PPM)**.
- ❑ For the preparation of standard turbidity or colour the pharmacopoeias **prescribe the limit of particular impurities for particular substances** and it varies for different compounds.
- ❑ The amount of test samples to be taken is mentioned in the individual monograph of the pharmacopoeias.

ASSESMENTS

1. Give a note on limit test for sulphate ?



2. Explain the reaction?



3. Name the below given apparatus?

REFERENCES

- **Thakur Publication** (various authors including Dr. Desh Deepak Pandey, Mr. Neeraj Kumar, Dr. Arpit Katiyar)

- **Udit Pharmacy** (author Udit Narayan Vishwakarma)

- ***Handbook of Inorganic Impurities in Pharmaceuticals*** (authored by Parjanya Kumar Shukla and Amita Verma)

- ***Practical Pharmaceutical In-organic Chemistry*** (author K. Subba Rao)


Thank You