

Limit test

Date: / / Page no: _____

Definition:

A limit test is quantitative or semi-quantitative test that identify and controls small amount of impurities, in a substance.

Impurity: The unwanted substance present in Active pharmaceutical substance.

⇒ Mechanism of limit test

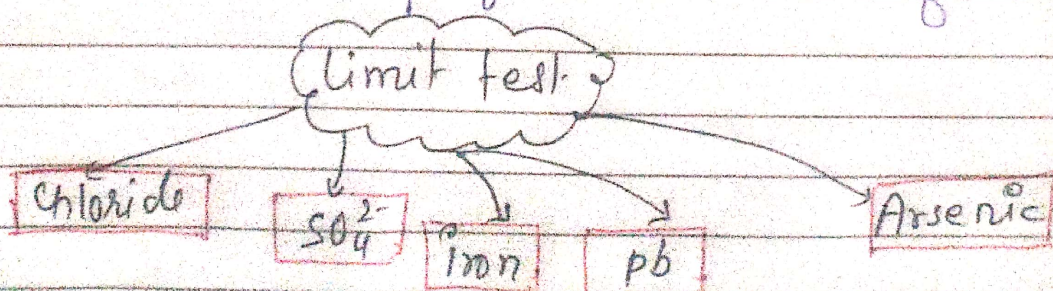
→ It is a comparison of unknown sample to a standard one that contain the impurity at product limit.

→ It is a colour reaction in c intensity of colour (Opalescence) shows the conⁿ of impurities.

Use of limit test

→ They are used to ensure that toxic substance or heavy metals are below the acceptable limits.

* Limit test are performed for the following



Limit test for chloride

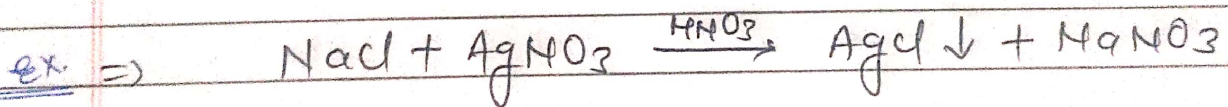
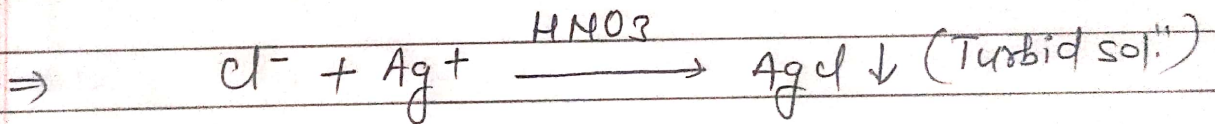
Date: / / Page no: _____

→ Limit test for chloride is a test that determines the amount of chloride in a substance by observing the reaction of chloride \equiv AgNO_3 in presence of HNO_3 .

Principle:

→ It is based on the reaction when soluble chloride (Cl^-) react \equiv silver nitrate (AgNO_3) in presence of dil HNO_3 to form silver chloride (AgCl)

→ This silver chloride appears as solid particles (opalescence) in solution.



Procedure

For test sample

→ ∇ Specific weight of compound is dissolved in distilled H_2O or solution is prepared according to pharmacopiea.

→ This solution is transferred to Nessler's cylinder.

- 3) Add 1 ml of HNO_3 (dil)
- 4) Dilute it with distilled H_2O and make up the volume upto 50 ml. in Nessler cylinder.
- 5) Add 1 ml of AgNO_3 solution.
- 6) stir solution immediately and kept for 5 minutes aside.

For standard solution

- Take 1 ml of 0.05845% w/v solution of HCl in Nessler cylinder.
- Add 1 ml HNO_3
- dilute the solution upto 50 ml.
- Add 1 ml. AgNO_3 & stir it & kept for 5 min.

OBSERVATION

- The opalescence produced in sample solution should not be greater than standard one.
- If opalescence is less than standard → Sample pass
- If opalescence is greater than standard → Sample fail

⇒ Role of HNO_3

- HNO_3 help to make solution acidic.
- It dissolve other impurities other than chloride
- Prevent the precipitate of Agel to disperse.
- Help to maintain cloudiness of Agel at the end.

Limit test for Sulphate

Date: / / Page no:

Limit test is a procedure to know the impurity present in solution.

Principle:

It is based on the reaction b/w soluble SO_4^{2-} & BaCl_2 in presence of dil HCl or Acetic acid (CH_3COOH).



BaSO_4 is formed & appears as solid particle or ppt (white flakes)

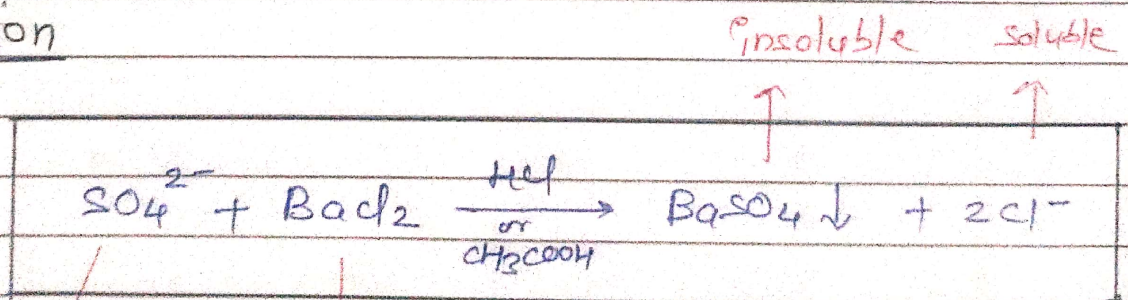


BaSO_4 produce turbidity or opalescence in solution



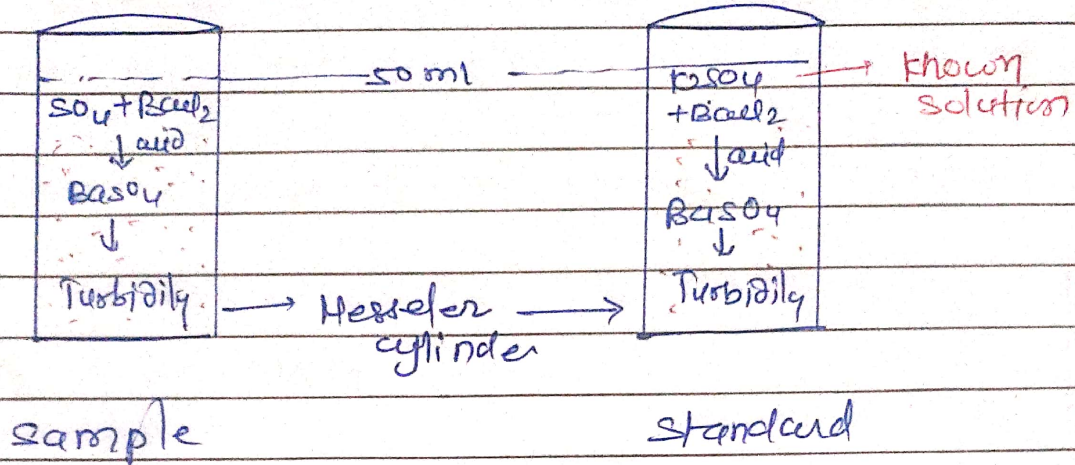
Turbidity of sample & standard will be compared.

Reaction



soluble

Soluble
 SO_4^{2-}



OBSERVATION

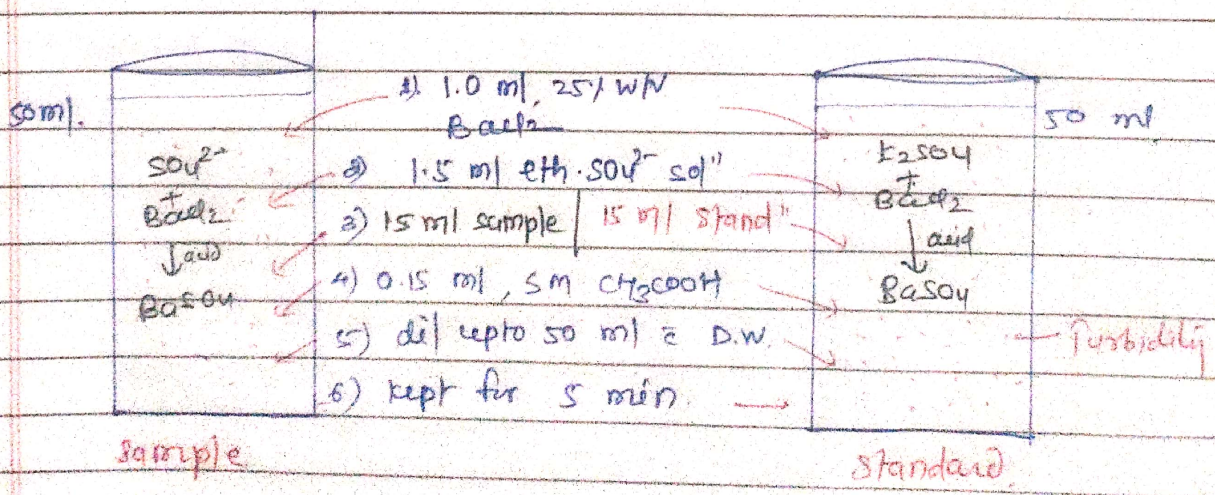
1) If sample solution is less turbid than standard = sample pass

2) If sample solution is more turbid than standard = sample fail

Procedure (As per I.P. 2018)

S.N.	sample	Standard
1.	Take 1.0 ml of 25% w/v solution of BaCl ₂ in M.C.	Take 1.0 ml of 25% w/v sol ⁿ of BaCl ₂ in M.C.
2.	Add 1.5 ml of ethanolic SO ₄ ²⁻ standard sol ⁿ (10 ppm), mix & allow to stand for 1 min.	Add 1.5 ml of ethanolic sulphate standard sol ⁿ (10 ppm) mix & allow to stand for 1 min.

S.N.	Sample	Standard
3.	Take 15 ml of specific amount of test substance dissolve in dist. H ₂ O or 15 ml of test solution prepared as per I.P.	Take 15 ml of sulphate standard solution (10 ppm)
4.	Add 0.15 ml of 5M acetic acid	4.) Add 0.15 ml of 5M acetic acid
5.	Make up to 50 ml \pm distilled H ₂ O	5) Make up to 50 ml \pm dist H ₂ O.
6.	Stir \pm glass rod & allow for 5 min	— do —
7.	observe the turbidity & compare	— do —



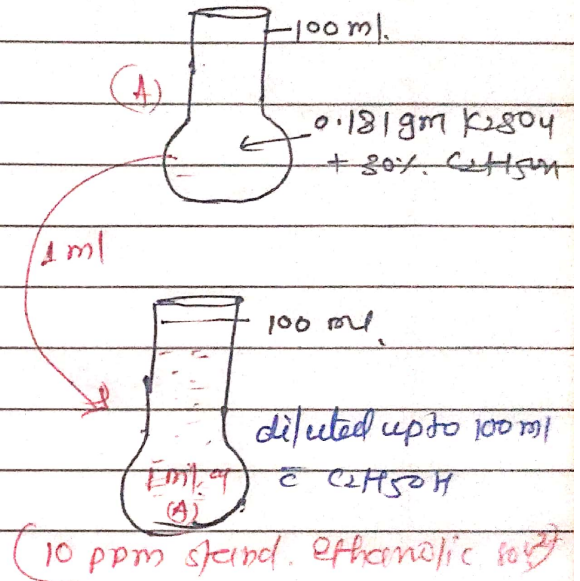
Preparation of chemical

(1) Preparation of ethanolic SO_4^{2-} standard solution

Dissolve 0.181 gm K_2SO_4 in 100 ml of 30% $\text{C}_2\text{H}_5\text{OH}$



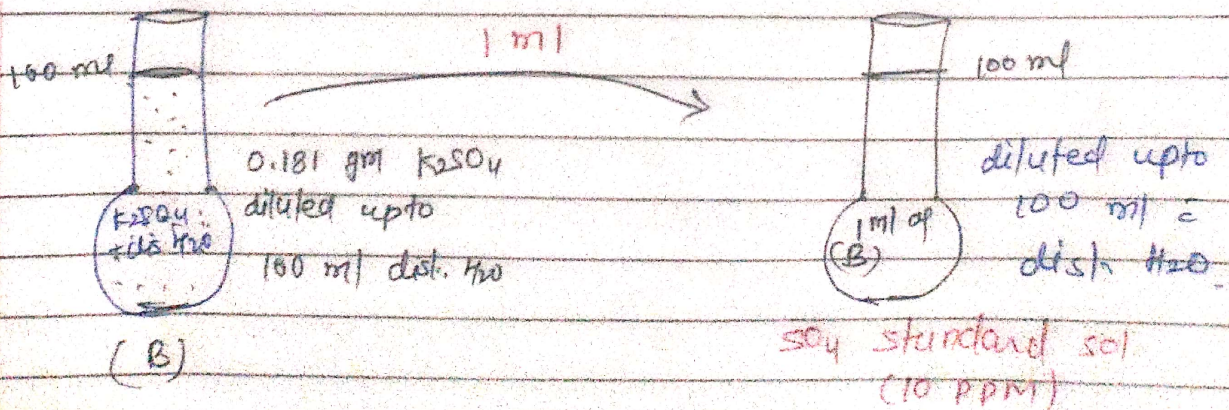
take 1 ml. of above solⁿ and dilute upto 100 ml $\text{C}_2\text{H}_5\text{OH}$.



Dilute 1 ml of 0.181% w/v solution of K_2SO_4 in $\text{C}_2\text{H}_5\text{OH}$ (30%) to 100 ml of $\text{C}_2\text{H}_5\text{OH}$ (30%)

(2) Preparation of sulphate standard solution (10 ppm)

Dilute 1 ml of 0.181% w/v solution of K_2SO_4 in distilled H_2O to 100 ml H_2O .



Role of CH_3COOH

- CH_3COOH help to make solⁿ acidic.
- It help Basou to precipitate & make solⁿ turbid at end.

Role of $\text{K}_2\text{S}_2\text{O}_8$

- ↑ sensitivity of test by giving ionic conⁿ of reagent.

Role of Alcohol

- It help to prevent supersaturation.