New Chapter - **LIMIT TEST**

Q. What is Limit test?

⇒ It is defined as Quantitative test design to identify and control small amount of impurities which is likely to be present in the substance.

Limit test

→ Chloride
→ Sulphate
→ Iron
→ Lead
→ Arsenic

Limit test of chloride

It is based upon chemical reaction between silver nitrate and soluble chloride ion in presence of dilute nitric acid to give opalescence, precipitate (ppt) of silver chloride.

Opalescence produce is compared with the standard solution.
If the opalescence of sample is less than the standard, it passes the test.

If it is more than or equal to the standard it means that it fail the test.

$$AgNO_3 + Cl^- \xrightarrow{HNO_3} AgCl \downarrow \text{(opalescence)}$$

Reaction:-

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

Silver Nitrate — ppt

Procedure:-

Test Sample:

(i) Specific weight of compound/Test sample is dissolved in water.
(ii) Add 10ml of dil. nitric acid.
(iii) Dilute to 50ml in Nessler's cylinder.
(iv) Add 1ml of 0.1M $AgNO_3$ solution, stir properly and keep aside for 5 min.
(v) Observe the opalescence/turbidity.

Standard Compound.

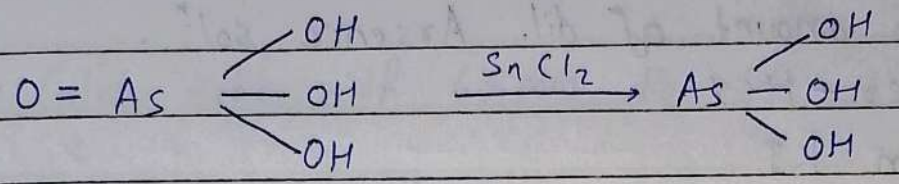
(i) Take 1ml of 0.0584% w/v solution of sodium chloride in Nessler's cylinder.

(ii) Add 10ml of dil. Nitric acid.
(iii) Dilute to 50ml in Nessler's cylinder.
(iv) Add 1ml of 0.1M $AgNO_3$ solution, stir properly. and keep aside for 5 min.

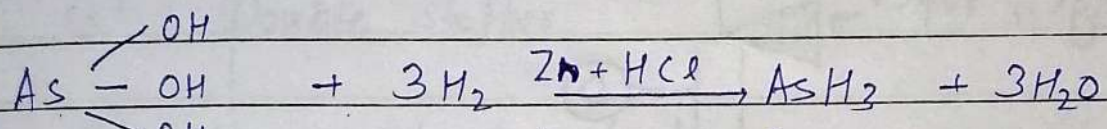
(v) Observe the opalescence/turbidity.

Limit Test for Arsenic - Gutzeit Apparatus.

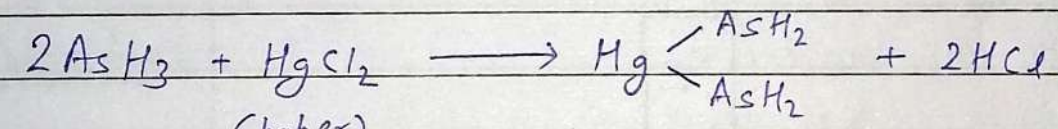
Rxn →



Arsenic acid (Pentavalent) — Arsenious Acid.



Arsenious acid — Nesscent Hydrogen — Arsine Gas (Arsenic hydride).



Yellow Stain

① Test Sample

(i) Sample
(ii) HCl (Arsenic Free)
(iii) 1gm KI
(iv) 5ml of stannous chloride ($SnCl_2$) acid.
(v) 10gm of Zn.

② Preparation of standard soln.

(i) Known amount of dil. Arsenic soln.
(ii) Water + HCl
(iii) 1 gm KI
(iv) 5 ml of stannous chloride ($SnCl_2$) acid.
(v) 10 gm of zn.

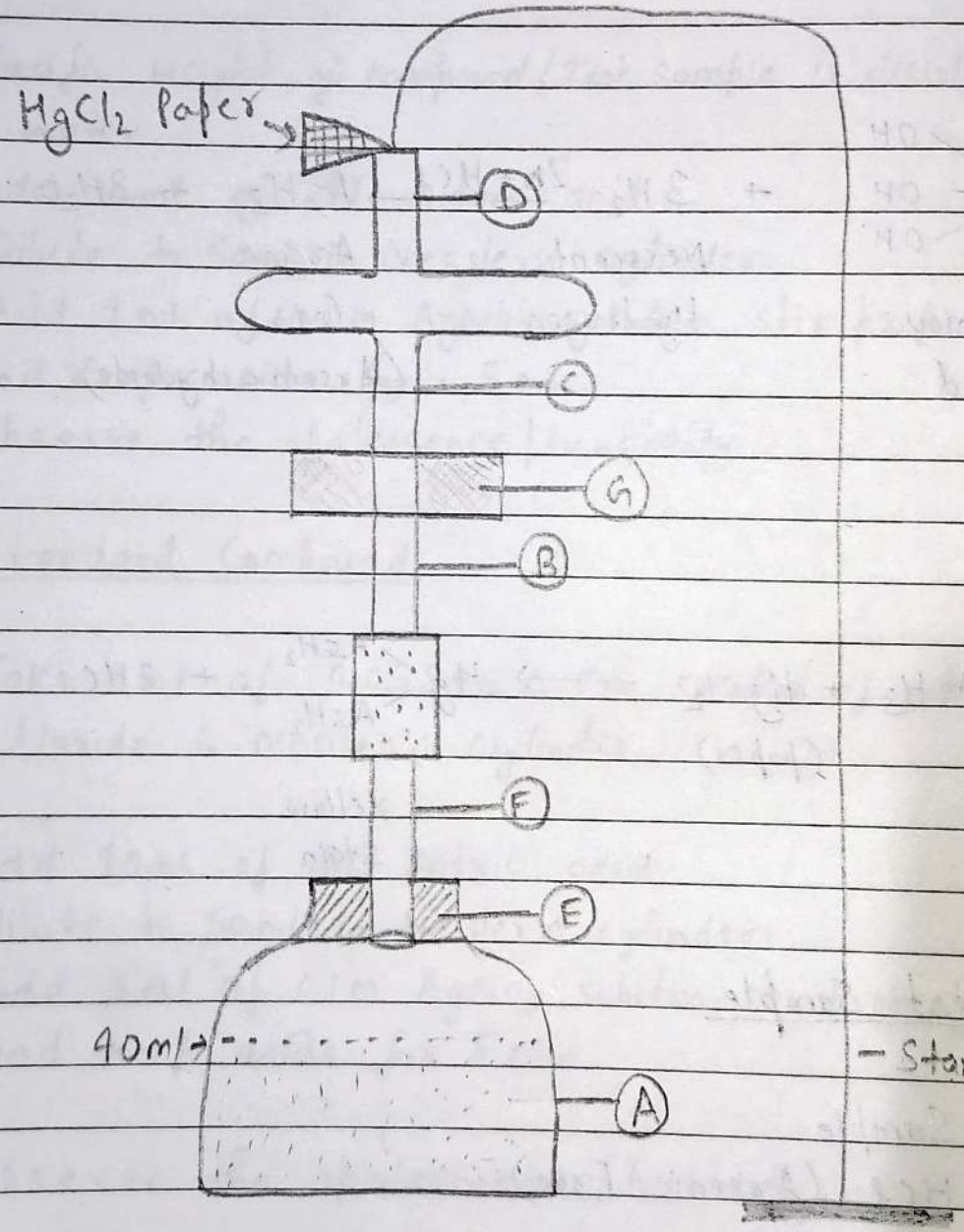


Fig. Gutzeit Apparatus

(A) → Generator bottle (capacity 60 ml)
40 ml indicating line.

(B) → Glass tube with 6.5 mm diameter.

(C) and (D) → Ground joint glass tube
→ Outer diameter 18 mm
→ Inner diameter 6.5 mm

(E) → Double stopper

(F) → Narrow parts of glass

(G) → Rubber Board,
lead acetate cotton plug.

Principle:

Limit test for Arsenic is based on the reaction of Arsine gas with hydrogen ion to form yellow stain on mercuric chloride paper in presence of reducing agent like potassium iodide. It is also called Gutzeit test. Arsenic present as Arsenic acid in the sample is reduced to Arsenious acid by reducing agent like potassium iodide, stannous acid, zinc, HCl.

Limit test of Sulphate

Rxn :

$$SO_4^{2-} + BaCl_2 \xrightarrow{\text{dil. HCl}} BaSO_4 + KCl$$

↓	↓		ppt.
Soluble Sulphate	Barium chloride		↓ Barium sulphate (it shows turbidity)

Principle:

Limit test for sulphate is based on the reaction between barium chloride and soluble sulphate in presence of dil. HCl. The turbidity form by a given amount of sample is compared with a reference on standard turbidity obtained from an authentic amount of sulphate under the same experimental condition.

Procedure :-

For test solution :-

(i) Specific amount of compound is dissolved in water.

(ii) Transfer into Nessler's cylinder and add 2 ml of dil. HCl.

(iii) Add 5 ml of $BaCl_2$ sol^n.

(iv) Diluted upto 50 ml with water. (H_2O).

(v) Keep aside for 5 min then observe the turbidity.

For standard solution :-

(i) Take 1 ml of 0.1089 % W/v of potassium sulphate in Nessler cylinder.

(ii) Add 2ml of dilute HCl.

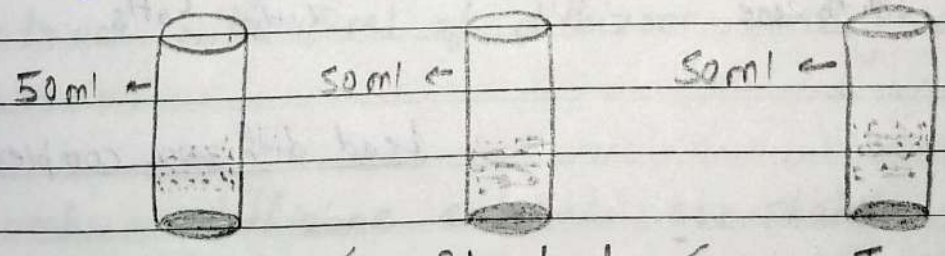
(iii) Add 5 ml of $BaCl_2$ sol^n

(iv) Dilute upto 50ml with water (H_2O).

(v) Keep aside for 5 min, then observe the turbidity.

Observation

Compare the turbidity against a black background into nessler's cylinder.



Less turbidity pass

More Turbidity fail

Note :- HCl helps to make solution acidic and Barium sulphate precipitate ($BaSO_4$) formed in insoluble which gives turbidity.

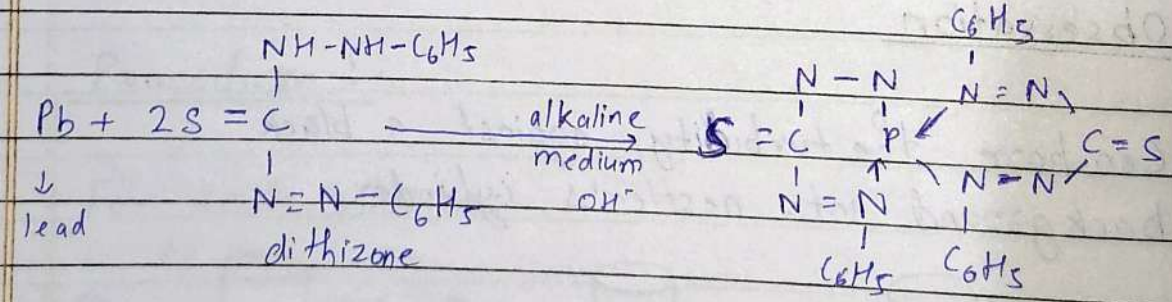
Limit test of lead (Pb)

Principle:

Limit test of lead is based on the reaction of lead and diphenyl thiocarbonate (dithizone) in alkaline solution to form lead dithizone complex which is ~~read~~ red in colour.

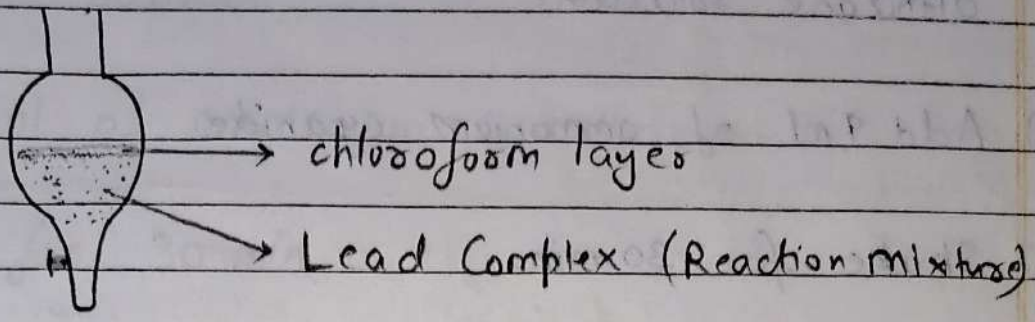
Dithizone is green in colour in chloroform and lead-dithizone complex violet in colour, so the resulting colour at the end of the process is red.

Rx^



Lead dithizone complex

Test Sample



Separating Funnel.

Test Solution

(1) A known quantity of sample solution is transferred in a separating funnel.

(2) Add 6ml of ammonium citrate.

(3) Add 2ml of potassium cyanide and 2ml of hydroxylamine hydrochloride. ($NH_2OH_2.HCl$)

(4) Add 4 drops of Phenol Red.

(5) Make solution alkaline by adding ammonia soln.

(6) Extract with 5ml of dithizone until it becomes green.

(7) Combine dithizone extracts are shaken for 30 mins with 30 ml of nitric acid and the chloroform layer is discarded.

(8) To the acidic solution add 5 ml of standard dithizone solution.

(9) Add 4 ml of ammonium cyanide.

(10) Shake for 30 min.

(11) Observe the colour.

Standard Solution

(1) A standard lead solution is prepared equivalent to the amount of lead permitted in the sample under examination.

(2) Add 6 ml of ammonium citrate.

(3) Add 2 ml of potassium cyanide and 2 ml of hydroxylamine hydrochloride ($NH_2OH.HCl$).

(4) Add 2 drops of phenol Red.

(5) Make solution alkaline by adding ammonia solution

(6) Extract with 5 ml of dithizone until it becomes green.

(7) Combine dithizone extracts are shaken for 30 mins with 30 ml of Nitric acid and the chloroform layer is discarded.

(8) To the acidic solution add 5 ml of standard dithizone solution.

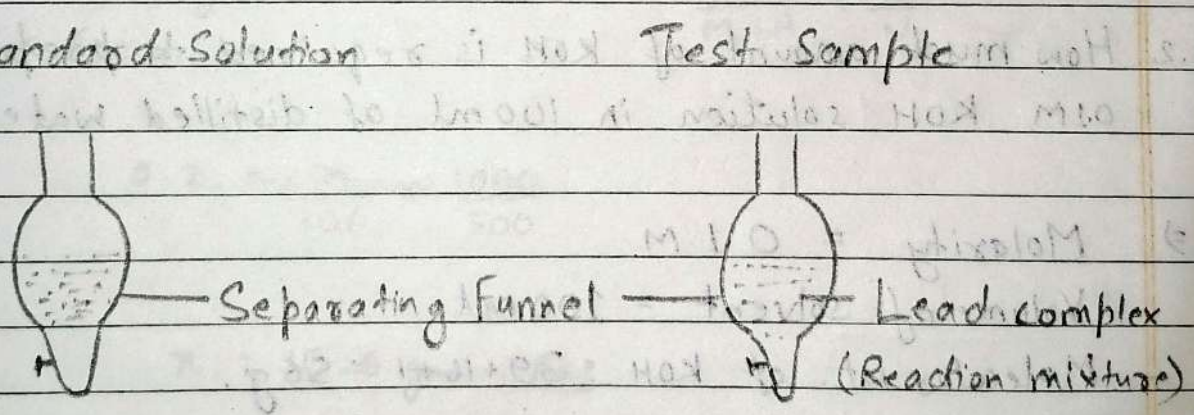
(9) Add 4 ml of ammonium cyanide.

(10) Shake for 30 min.

(11) Observe the colour.

Observation.

The intensity of the ~~complex~~ colour of complex, is depend on the amount of lead in the solution the colour produced in the sample solution should not be greater than standard solution. If colour produced in the sample solution is less than the standard solution. The sample will pass the limit test of lead.



Colour intensity of Standard > Colour intensity of test sample

∴

Limit test pass.

Molarity

$$\text{Molarity} = \frac{\text{Weight of solute (g)} \times 1000\,ml}{\text{Molecular weight of solute (g/mol)} \times \text{Volume of Solvent taken (ml)}}$$

Q1. Calculate the molarity / molar concentration of solution when 5g of NaOH in ~~500ml~~ 50ml of water.

⇒ Weight of NaOH = 5g
Volume of Solvent = 50ml
Molecular Wt. of NaOH = 23 + 16 + 1 = 40.

$$\text{Molarity} = \frac{Wt \times 1000\,ml}{M.Wt \times V\,(ml)} = \frac{5 \times 1000}{40 \times 50} = \frac{5}{2} = 2.5M$$ Ans

Q.2. How much amount of KOH is required to prepare 0.1M KOH solution in 100ml of distilled water.

⇒ Molarity = 0.1 M
Volume of solvent = 100ml
Molecular Wt. of KOH = 39 + 16 + 1 = 56g.

$$M = \frac{Wt}{M.W} \times \frac{1000\,ml}{V(ml)}$$

$$\Rightarrow 0.1M = \frac{x}{56} \times \frac{1000}{100}$$

$$\Rightarrow x = \frac{56}{100} = .56\,gm$$ Ans

Q.3 To prepare 0.2 M Na_2CO_3 in 250 ml of distilled water. How much gram of Na_2CO_3 required.

⇒ M = 0.2 M

V. of water = 250 ml

M.W of Na_2CO_3 = 46 + 12 + 48 = 106.

$$M = \frac{x}{M.W} \times \frac{1000\ ml}{V.\ of\ water}$$

$$0.2 = \frac{x}{106} \times \frac{1000}{250}$$

$$x = \frac{106 \times 2}{4 \times 10} = \frac{53}{10} = 5.3\ g \quad \text{Ans.}$$

Q.4. To prepare 0.2 M Na_2CO_3 in 500 ml of distilled water. How much gm of Na_2CO_3 required.

⇒ M = 0.2 M

M.W = 106 g

V = 500 ml

$$M = \frac{Wt}{M.Wt} \times \frac{1000}{V}$$

$$0.2 = \frac{x}{106} \times \frac{1000}{500}$$

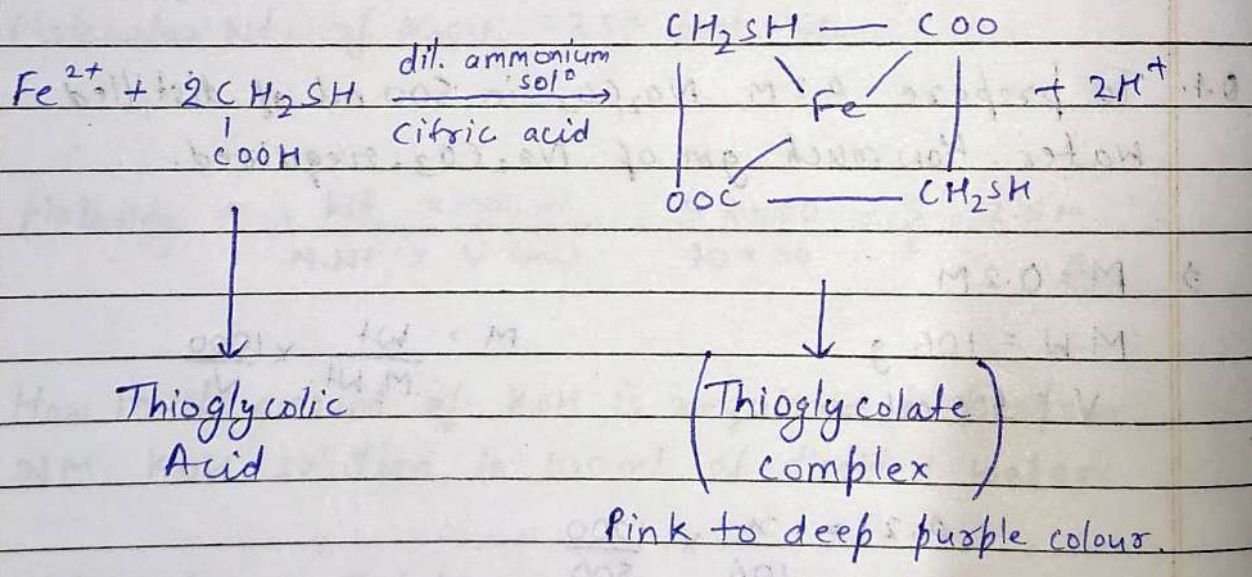
$$x = 10.6\ gm \quad \text{Ans.}$$

Limit test of ~~Chloride~~ Iron (Fe)

Principle / Theory :

The limits test of iron depends upon the reaction of iron in ammonical solution in presence of citric acid with thioglycolic acid to obtain a pink to deep purple colour. Citric acid helps precipitation of iron by ammonia by forming a complex with it. The colour is obtain due to formation of ferrous salt i.e. ferrous thioglucolate which disappear in air due to oxidation.

Reaction.



Procedure.

Test Sample.

(i) Sample is dissolve in specific amount of water and then volume is made upto 40 ml.

(ii) Add 2 ml of 20% W/v of citric acid (iron free).

(iii) Add 2 drops of thioglycolic acid.

(iv) Add ammonia to make the solution alkaline and adjust the volume upto 50 ml.

(v) Keep aside for 5 min.

(vi) Colour developed is viewed vertically and compared with standard solution.

Standard Solution.

(i) Add 2 ml of standard solution of iron diluted with water upto 40 ml.

(ii) Add 2 ml of 20% W/v of citric acid (iron free).

(iii) Add 2 drops of thioglycolic acid.

(iv) Add ammonia to make the solution alkaline and adjust the volume upto 50ml.

(v) Keep aside for 5 min.

(vi) Colour developed is viewed vertically and compared with standard solution.

Observation.

The purple colour produced in the sample solution should not be greater than standard solution.

Q.1. Calculate the molarity / molar concentration of solution when 10 gm of NaOH in 50ml of distilled water.

⇒ Wt = 10 gm
M.W = 40 gm
V = 50 ml

$$M = \frac{Wt \times 1000\,ml}{M.Wt \times V}$$

⇒ $$M = \frac{10}{40} \times \frac{1000}{50}$$

M = 5 M Ans.

Q.2. How much amount of KOH is required to prepare 0.2 M KOH solution in 200 ml distilled water.

⇒ M = 0.2 M
V = 200 ml
M.W = 56 gm

$$M = \frac{Wt.}{M.Wt} \times \frac{1000}{V}$$

⇒ $$0.2\,M = \frac{x}{56} \times \frac{1000}{200}$$ ~~= 56~~ ⇒ $$x = \frac{56}{25}$$

x = 2.24 gm Ans

Q.3 To prepare 0.5 M Na_2CO_3 in 500 ml of distilled water. How much quantity of Na_2CO_3 is required.

» M = 0.5 M

V = 500 ml

M.W = 106 gm

$$M = \frac{Wt}{M.Wt} \times \frac{1000}{V}$$

$$0.5\ M = \frac{x}{106} \times \frac{1000}{500}$$

$$x = \frac{106}{2} \times \frac{5}{10}$$

$$x = 26.5\ gm$$ Ans.

Limit test of Heavy Metals.

This limit test is for detecting and limiting the impurities of Heavy metals likely to be present in many drugs.

The Heavy metals are precipitated as their sulphides by the addition of either hydrogen sulphide or sodium sulphide solution under specific conditions.

A dark brown or light brown colour is produced depending upon the amount of heavy metals present.

Procedure:

(i) Preparation of test sample/solution:

- Solution is prepared as per monograph and 25 ml is transferred into Nessler's cylinder.
- Adjust the pH b/w 3-4 by adding dilute ~~acid~~ acetic acid or dilute ammonium solution.
- Add freshly prepared 10 ml of hydrogen sulphide solution.
- Then dilute with water upto 50 ml.
- Allow to stand for 5 mins.
- Then view downwards over a white surface.

(ii) Preparation of standard solution:

- Take 2 ml of standard heavy metal solution and dilute upto 25 ml with distilled water.
- Adjust the pH b/w 3-4 by adding dilute acetic acid or dilute ammonium solution.
- Add freshly prepared 10 ml of hydrogen sulphide solution.
- Then dilute with water upto 50 ml.
- Allow to stand for 5 mins.
- Then view downwards over a white surface.

Observations:

The colour produced in sample solution should not be greater than standard solution. If the colour produced in sample solution is less than the standard solution, the sample will pass the limit test of heavy metal and vice-versa.