

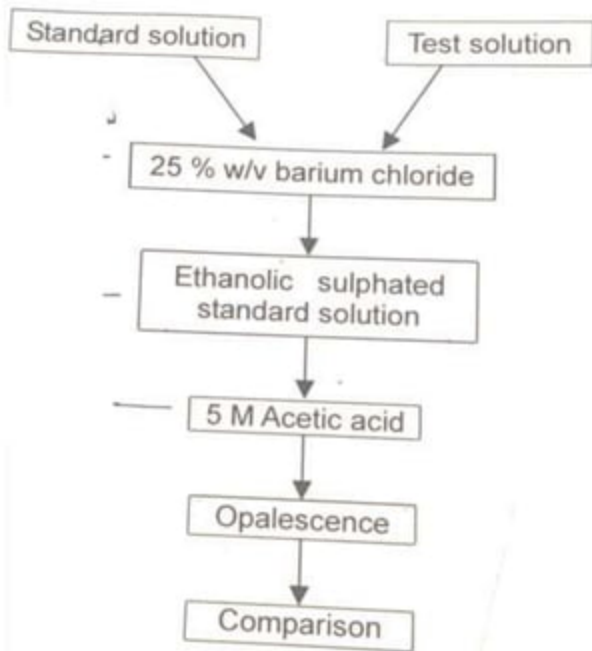
# LIMIT TEST SULPHATE

## Principle:

Limit test of sulphate is based on the reaction of **soluble sulphate** with **barium chloride** in presence of **dilute hydrochloric acid** to form **barium sulphate** which appears as solid particles (turbidity) in the solution.



Potassium sulphate has been added to increase the sensitivity of the test.



### Observation:

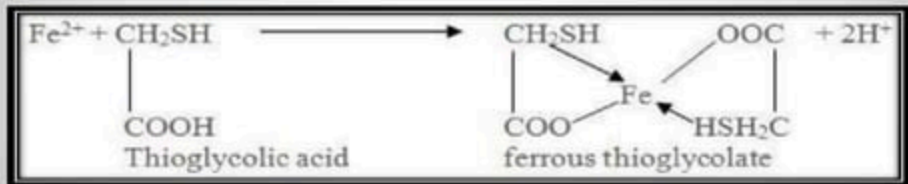
The turbidity produce in sample solution should not be greater than standard solution. If turbidity produces in sample solution is less than the standard solution, the sample will pass the limit test of sulphate and vice versa.

### Reasons:

- Hydrochloric acid helps to make solution acidic.
- Potassium sulphate is used to increase the sensitivity of the test by giving ionic concentration in the reagent.
- Alcohol helps to prevent super saturation and so produces a more uniform opalescence.

## Limit test for IRON:

- ❖ Limit test of Iron is based on the reaction of iron in ammonical solution with thioglycolic acid in presence of citric acid to form iron thioglycolate (Ferrous thioglycolate complex) which produces pale pink to deep reddish purple color in alkaline media.
- ❖ Thioglycolic acid is used as reducing agent.



- ❖ The color of the Ferrous thioglycolate complex fades in the presence of air due to oxidation.
- ❖ Also, the color is destroyed in presence of oxidizing agents and strong alkalis.
- ❖ The **purple color is developed only in alkaline media. So ammonia solution is used.**
- ❖ But ammonia reacts with iron, forms precipitate of **ferrous hydroxide**.
- ❖ Thus citric acid is used which prevents the precipitate of iron with Ammonia by forming a complex with iron as iron citrate.

# Procedure:

Test sample	Standard compound
Sample is dissolved in specific amount of water and then volume is made up to 40 ml	2 ml of standard solution of iron diluted with water upto 40 ml
Add 2 ml of 20 % w/v of citric acid (iron free)	Add 2 ml of 20 % w/v of citric acid (iron free)
Add 2 drops of thioglycollic acid	Add 2 drops of thioglycollic acid
Add ammonia to make the solution alkaline and adjust the volume to 50 ml	Add ammonia to make the solution alkaline and adjust the volume to 50 ml
Keep aside for 5 min	Keep aside for 5 min
Color developed is viewed vertically and compared with standard solution	Color developed is viewed vertically and compared 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.

### Reasons:

- Citric acid forms complex with metal cation and helps precipitation of iron by ammonia by forming a complex with it.
- Thioglycolic acid helps to oxidize iron (II) to iron (III).
- Ammonia is added to make solution alkaline. The pale pink color is visible only in the alkaline media. The color is not visible in acidic media as ferrous thioglycolate complex decomposes in high acidic media.

## Limit test for Arsenic:

- ❖ Arsenic is a well known undesirable and harmful impurity which is present in medicinal substances.
- ❖ Pharmacopoeial method is based on the **Gutzeit test**.
- ❖ All the special reagents used in the limit test for Arsenic are marked and distinguished by letter 'As T', which means that they all should be Arsenic free and should themselves conform to the test for Arsenic.

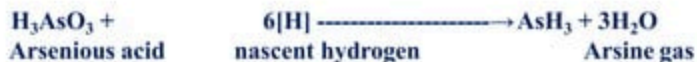
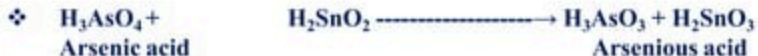
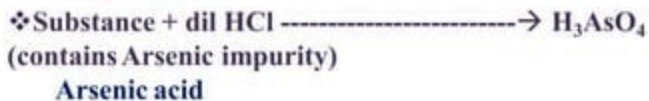


## Principle:

Limit test of Arsenic is based on the reaction of arsenic gas with hydrogen ion to form **yellow stain** on mercuric chloride paper in presence of reducing agents like potassium iodide.

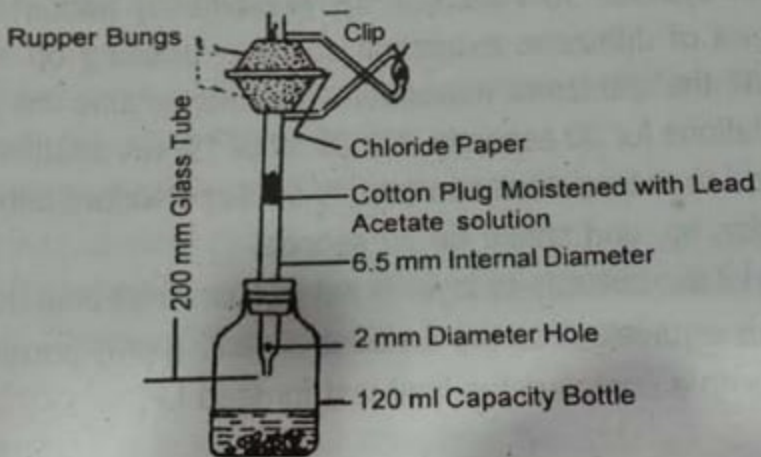
It is also called as **Gutzeit test** and requires special apparatus.

- ❖ Arsenic, present as arsenic acid ( $\text{H}_3\text{AsO}_4$ ) in the sample is reduced to arsenious acid ( $\text{H}_3\text{AsO}_3$ ) by reducing agents like potassium iodide, stannous acid, zinc, hydrochloric acid, etc. Arsenious acid is further reduced to arsine (gas) ( $\text{AsH}_3$ ) by hydrogen and reacts with mercuric chloride paper to give a yellow stain.



**The depth of yellow stain on mercuric chloride paper will depend upon the quantity of arsenic present in the sample.**

- ❖ When the sample is dissolved in **acid**, the Arsenic present in the sample gets converted to **Arsenic acid**.
- ❖ By action of reducing agents like Potassium iodide, stannous acid etc., Arsenic acid gets reduced to **arsenious acid**.
- ❖ The **nascent hydrogen** formed during the reaction, further reduces **Arsenious acid** to **Arsine gas**, which reacts with mercuric chloride paper, giving a yellow stain.



**Figure : Apparatus used for arsenic limit test**

## Reasons:

- Stannous chloride is used for complete evolution of arsine.
- Zinc, potassium iodide and stannous chloride is used as a reducing agent.
- Hydrochloride acid is used to make the solution acidic
- Lead acetate paper** are used to trap any hydrogen sulphide which may be evolved along with arsine.

### **Use of stannated Hydrochloric acid:**

If pure zinc and HCl are used, the steady evolution of gas does not occur. This produces improper stain (e.g slow evolution produces short but intense stain while rapid evolution of gas produces long but diffused stain.)

So, to get steady evolution of gas, stannated hydrochloric acid is used.

### **Use of Lead Acetate solution:**

H<sub>2</sub>S gas may be formed during the experiment as zinc contains sulphides as impurities. It gives black stain to HgCl<sub>2</sub> paper and so will interfere the test.

Hence, gases evolved are passed through cotton wool plug moistened with lead acetate, where H<sub>2</sub>S gas is trapped as PbS.

### **Use of Potassium iodide:**

KI is converted to HI which brings about reduction of unreacted pentavalent arsenic to trivalent Arsenic. Thus, reproducible results can be obtained. If it is not used then some pentavalent Arsenic may remain unreacted.

## Limit test for heavy metals

- ❑ The limit test for heavy metals is designed to determine the content of **metallic impurities** that are coloured by **hydrogen sulphide** or sodium sulphide under the condition of the test should not exceed the heavy metal limits given under the individual monograph.
- ❑ The heavy metals (metallic impurities) may be iron, copper, lead, nickel, cobalt, bismuth, antimony etc.. **The limit for heavy metals is indicated in the individual monograph in term of ppm of lead i.e. the parts of lead per million parts of the substance being examined**
- ❑ In substances the proportion of any such impurity (Heavy metals) has been expressed as the quantity of lead required to produce a color of equal depth as in a standard comparison solution having a definite quantity of lead nitrate.
- ❑ The quantity is stated as the heavy metal limit and is expressed as parts of lead (by weight) per million parts of the test substance.
- ❑ **The limit test for heavy metals has been based upon the reaction of the metal ion with hydrogen sulphide, under the prescribed conditions of the test causing the formation of metal sulphides.**
- ❑ These remain distributed in colloidal state, and give rise to a brownish coloration.

- ❖ I.P limit for heavy metals in 20 ppm.
- ❖ The test solution is compared with a standard prepared using a lead solution (as the heavy metal).The metallic impurities in substance are expressed as parts of lead per million parts of substance.
- ❖ IP has adopted 3 methods for this:
  - ❖ **Method I:** The method is applicable for the samples which give clear colourless solutions under specified conditions of test.
  - ❖ **Method II:** The method is applicable for the samples which DO NOT give clear colourless solutions under specified conditions of test.
  - ❖ **Method III:** Used for substances which give clear colourless solutions in sodium hydroxide medium.

## **Limit test for lead:**

Lead is a most undesirable impurity in medical compounds and comes through use of sulphuric acid, lead lined apparatus and glass bottles use for storage of chemicals.

### **Principle:**

Limit test of lead is based on the reaction of lead and diphenyl thiocarbazon (dithizone) in alkaline solution to form lead dithizone complex which is red in color.



- ❖ Dithizone in chloroform, is able to extract lead from alkaline aqueous solutions as a lead dithizone complex (Red in colour)
- ❖ The original dithizone is having a green colour in chloroform while the lead-dithizone is having a violet color. So, resulting color at the end of the process is read.
- ❖ **The intensity of the color of complex is dependant upon the amount of lead in the solution.**
- ❖ The color of the lead-dithizone complex in chloroform has been compared with a standard volume of lead solution, treated in the same manner.
- ❖ In this method, the lead present as an impurity in the substances, gets separated by extracting an alkaline solution with a dithizone extraction solution.
- ❖ **The interference and influence of the other metal ions has been eliminated by adjusting the optimum pH for the extraction by employing Ammonium citrate/ potassium cyanide.**

- **Observation:**

The intensity of the color of complex, is depends on the amount of lead in the solution. The color produced in sample solution should not be greater than standard solution. If color produces in sample solution is less than the standard solution, the sample will pass the limit test of lead and vice versa.

**Reasons:**

- **Ammonium citrate, potassium cyanide, hydroxylamine hydrochloride** is used to make pH optimum so interference and influence of other impurities have been eliminated.
- Phenol red is used as indicator to develop the color at the end of process Lead present as an impurities in the substance, gets separated by extracting an alkaline solution with a dithizone extraction solution.