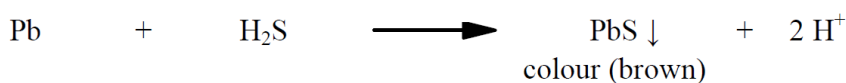


## LIMIT TEST FOR HEAVY METALS: LEAD

### PRINCIPLE:

In this experiment, the test color obtained by the reaction of heavy metal impurities with a saturated solution of hydrogen sulphide is compared with the standard color obtained by the reaction of a known quantity of lead with a saturated solution of hydrogen sulphide.

The precipitate of heavy metal sulphide formed gives colour. Dilute acetic acid and ammonia are used to maintain pH between 3.0 and 4.0 so that the precipitate formed is colloidal and uniform. The saturated solution of hydrogen sulphide has to be prepared freshly for the experiment. Here hydrogen sulphide gas is generated using a specially devised apparatus called as Kipp's apparatus in which ferrous sulphide sticks are made to react with equal volumes of concentrated hydrochloric acid and water. Indian pharmacopeia 1996, provides four methods depending on the resulting solution of substance (i.e., based on solubility, colour etc). Method I uses hydrogen sulphide solution, method II uses hydrogen sulphide solution after igniting the substance and method III uses sodium sulphide solution after treating the substance with sodium hydroxide solution.



**Lead      Hydrogen sulphide      Lead sulphide**

### PROCEDURE:

#### **Method I:**

It is used for the substance which gives a clear, colorless solution under specified conditions.

| Test sample  | Standard compound  |
|--|--|
| Solution is prepared as per the monograph and 25 ml is transferred in Nessler's cylinder       | Take 2 ml of standard lead solution and dilute to 25 ml with water                             |
| Adjust the pH between 3 to 4 by adding dilute acetic acid 'Sp' or dilute ammonia solution 'Sp' | Adjust the pH between 3 to 4 by adding dilute acetic acid 'Sp' or dilute ammonia solution 'Sp' |

|  |  |
|--|--|
| Dilute with water to 35 ml                               | Dilute with water to 35 ml                               |
| Add freshly prepared 10 ml of hydrogen sulphide solution | Add freshly prepared 10 ml of hydrogen sulphide solution |
| Dilute with water to 50 ml                               | Dilute with water to 50 ml                               |
| Allow to stand for five minutes                          | Allow to stand for five minutes                          |
| View downwards over a white surface                      | View downwards over a white surface                      |

### Method II:

It is used for the substance which does not give a clear, colorless solution.

| Test sample   | Standard compound  |
|---|--|
| Weigh specific quantity of test substance, moisten with sulphuric acid and ignite on a low flame till completely charred<br>Add few drops of nitric acid and heat to 500 °C<br>Allow to cool and add 4 ml of hydrochloric acid and evaporate to dryness<br>Moisten the residue with 10 ml of hydrochloric acid and digest for two minutes<br>Neutralize with ammonia solution and make just acid with acetic acid | Take 2 ml of standard lead solution and dilute to 25 ml with water                             |
| Adjust the pH between 3 to 4 and filter if necessary  | Adjust the pH between 3 to 4 by adding dilute acetic acid 'Sp' or dilute ammonia solution 'Sp' |

|  |  |
|--|--|
| Dilute with water to 35 ml                               | Dilute with water to 35 ml                               |
| Add freshly prepared 10 ml of hydrogen sulphide solution | Add freshly prepared 10 ml of hydrogen sulphide solution |
| Dilute with water to 50 ml                               | Dilute with water to 50 ml                               |
| Allow to stand for five minutes                          | Allow to stand for five minutes                          |
| View downwards over a white surface                      | View downwards over a white surface                      |

### **Method III:**

It is used for the substance which gives a clear, colorless solution in sodium hydroxide medium.

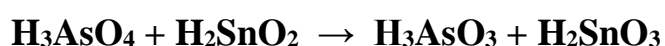
| <b>Test sample</b>   | <b>Standard compound</b>  |
|--|---|
| Solution is prepared as per the monograph and 25 ml is transferred in Nessler's cylinder or weigh specific amount of substance and dissolve in 20 ml of water and add 5 ml of dilute sodium hydroxide solution | Take 2 ml of standard lead solution   |
| Make up the volume to 50 ml with water   | Add 5 ml of dilute sodium hydroxide solution and make up the volume to 50 ml with water |
| Add 5 drops of sodium sulphide solution  | Add 5 drops of sodium sulphide solution   |
| Mix and set aside for 5 min  | Mix and set aside for 5 min   |
| View downwards over a white surface  | View downwards over a white surface   |

## **LIMIT TEST FOR ARSENIC**

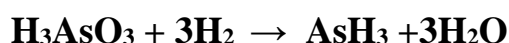
### **PRINCIPLE:**

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.

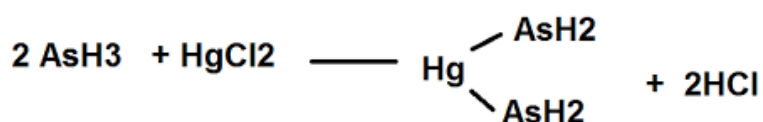
Arsenic, present as arsenic acid in the sample is reduced to arsenious acid by reducing agents like potassium iodide, stannous acid, zinc, hydrochloric acid, etc. Arsenious acid is further reduced to arsine (gas) by hydrogen and reacts with mercuric chloride paper to give a yellow stain.



**Arsenic acid**                      **Arsenious acid**



**Arsenious acid**                      **Arsine**



**Mercuric chloride**                      **Mercuric arsine**

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

**PROCEDURE:**

| <b>Standard solution</b>                                      | <b>Test solution</b>   |
|---|--|
| Add 1 ml standard solution of Arsenic (10 ppm) in 50 ml water | Dissolved sample in 50 ml water                              |
| Add 10 ml of Stannous Hcl acid                                | Add 10 ml of Stannous Hcl acid                               |
| Add further 1 gm of Potassium iodide + 10 gm granulated zinc  | Add further 1 gm of Potassium iodide + 10 gm granulated zinc |
| Place in Gutzeit Apparatus for 40 minutes                     | Place in Gutzeit Apparatus for 40 minutes                    |

Keep the solution aside for 40 min and stain obtained on mercuric chloride paper is compared with standard solution.

**Reasons:**

Stannous chloride is used for the complete evolution of arsine.

Zinc, potassium iodide and stannous hydrochloric acid are used as reducing agent.

Arsenic is reduced to the arsine by the combined action of these reagents.

Lead acetate-soaked cotton plug is used to trap any hydrogen sulfide which may be evolved along with arsenic.