

LIMIT TEST FOR ARSENIC



By...

Prof. Sonali R. Pawar

Assistant Professor, Pharmaceutical Chemistry Department,

JSPM's Charak College of Pharmacy and Research, Wagholi , Pune 412207

To perform limit test for **Arsenic** for given unknown sample

S.N.	Chemical	Quantity	Apparatus	Quantity
1	Lead acetate solution (10% w/v) $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$	Q.S.	Arsenic apparatus (Gutzeit Apparatus)	02
2	Potassium iodide (KI) (AsT)	2 gm	Beaker (100 ml)	02
3	Zinc (Zn) (AsT)	20 gm	Glass Rod	01
4	HgCl_2 Paper	Q.S.	Stand	01

REAGENT PREPARATION

- 1. Preparation of the test solution:** The solution of water soluble substance is prepared with water and stannated HCl AsT. The solution of substance such as metallic carbonates, which effervesces with acids, is obtained with brominated HCl AsT. The substances, which are insoluble, e.g.: BaSO_4 , bentonite or kaolin are diffused in water.
- 2. Stannated Chloride solution AsT:** It is prepared by adding Stannous Chloride solution to an equal volume of HCl AsT, reducing the original volume by boiling and filtering through a fine-grain filter paper.
- 3. Stannated Hydrochloric acid AsT:** It is prepared by adding 1 ml of stannous chloride solution AsT to 100 ml of HCl AsT.
- 4. Preparation of standard arsenic solution (10 ppm As):** Dissolved 0.330 g of arsenic trioxide in 5ml of 2 M sodium hydroxide and dilute to 250.0 ml with water. Dilute 1 volume of this solution to 100 volumes with water.
- 5. Zinc AsT:** It is the granulated zinc which complies with the following additional test:
 - To 10 gm of the granulated zinc adds 15 ml of the stannous chloride solution AsT and 5 ml of 0.1 M potassium iodide.
 - Apply the general test but allow the reaction to continue for one hour.
 - NO visible stain should be produced on the mercuric chloride paper.
 - Repeat the test by adding 0.1 ml of standard arsenic solution (10 ppm As); a faint but distinct yellow stain is produced.

PRINCIPLE

Arsenic is harmful due to its toxic nature

Pharmacopoeia method is based on 'Gutzeit Method'.

Concentration of arsenic beyond 0.01 mg/L in pollutant by the World Health Organization (WHO).

All arsenic present converted into arsenic gas (AsH_3) by reduction with zinc and Hydrochloric acid.

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.

British Pharmacopoeia suggest the use of mercuric chloride paper instead of mercuric bromide paper.

The standard stain prepared from a definite quantity of arsenic is used for comparison and provide the limit,

• REACTION:

1

2

- The sample dissolved in acid where by the arsenic present as impurity in sample

3

- Converted into arsenic acid (Arsenic, present as arsenic acid in the sample)

4

- Is reduced to Arsenious acid by reducing agents like potassium iodide, stannous acid, zinc, hydrochloric acid, etc.

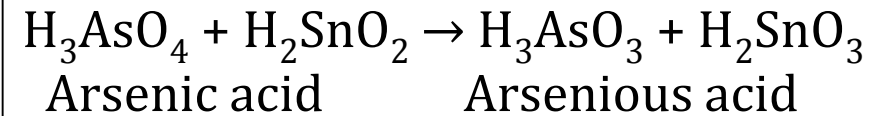
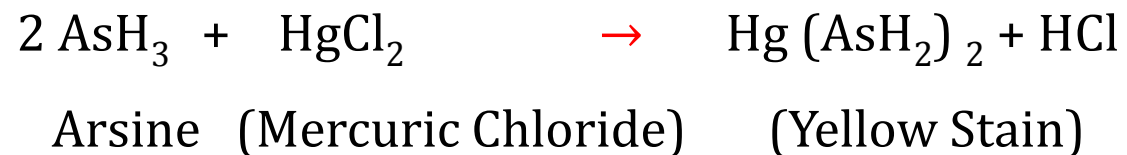
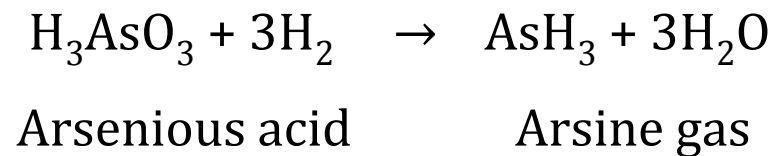
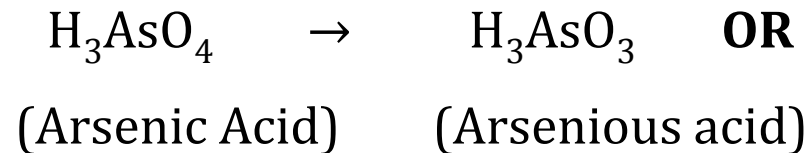
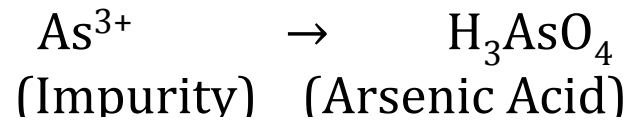
5

- Arsenious acid is further reduced to arsine (gas)

6

- 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,
which is compared with that of standard stain
produced from
known amount of arsenic.*



- Stannous chloride is used for complete evolution of arsine.



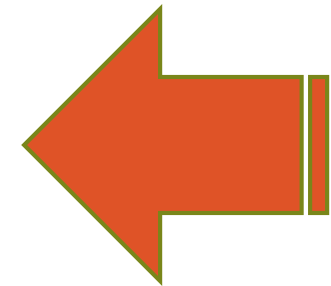
- Zinc, potassium iodide and stannous chloride is used as a reducing agent.



- Hydrochloride acid is used to make the solution acidic.



- Lead acetate pledger or papers are used to trap any hydrogen sulphide, which may be evolved along with arsine.



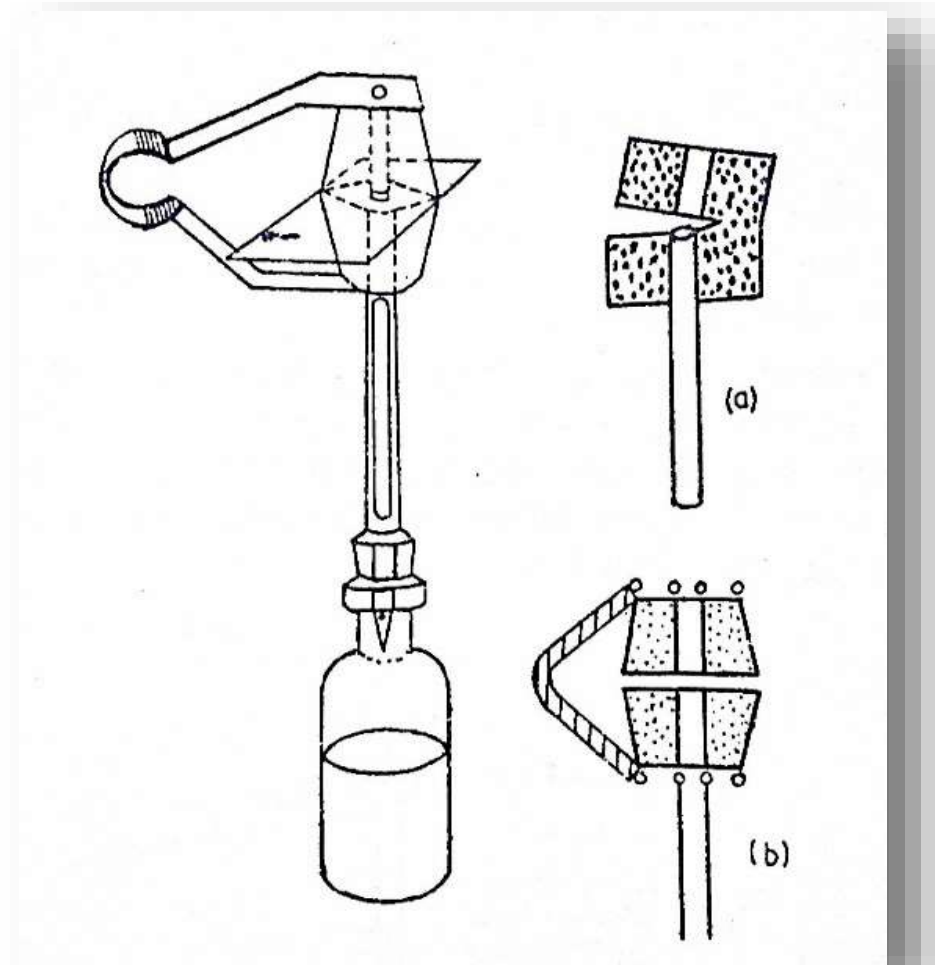
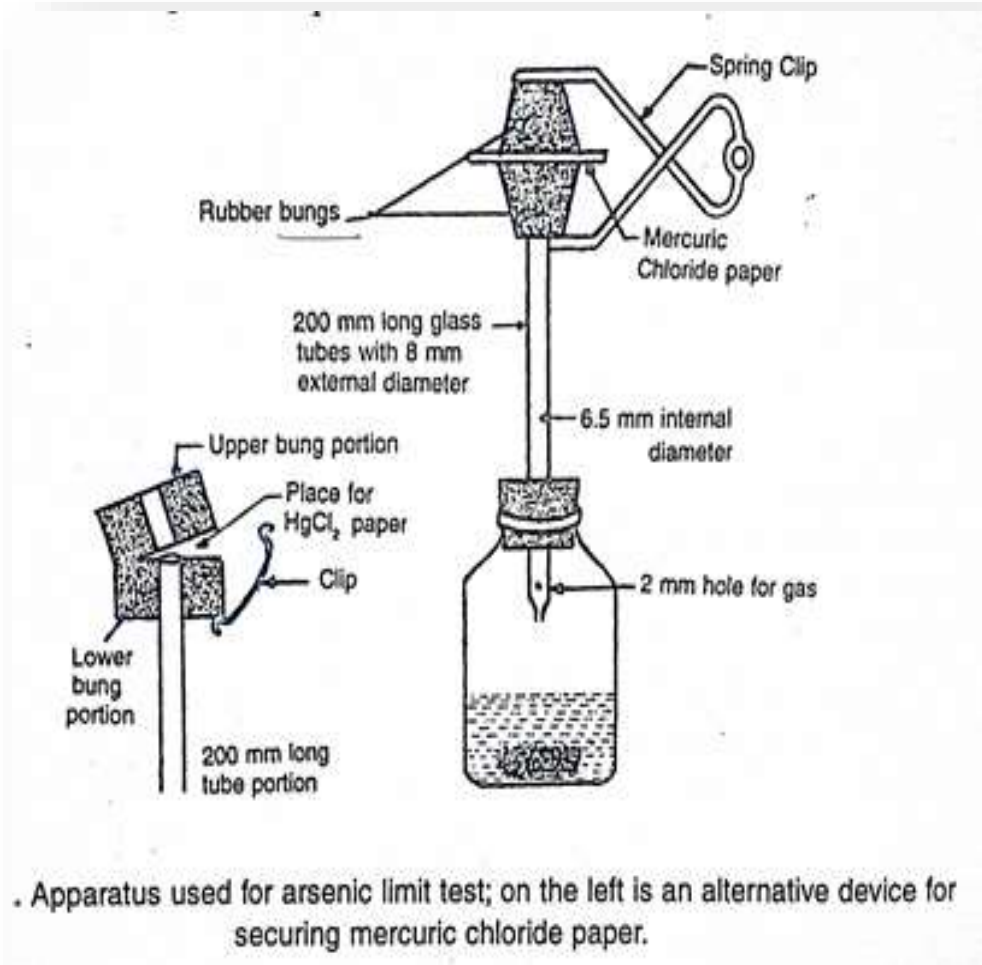
Reasons

PROCEDURE

Take 250 ml of the arsenic LT apparatus bottles. Labelled one is 'Test' and other is 'standard'.

SN	Standard	Test
1	A know amount of dilute arsenic solution is kept in the wide mouthed bottle of the apparatus.	The test solution is prepared by dissolving specific amount in water and stannated HCl (arsenic free) and kept in a wide mouthed bottle.
2	To this solution 1 gm of KI, 5 ml of stannous chloride acid solution and 10 gm of zinc is added (all this reagents must be arsenic free)	To this solution 1 gm of KI, 5 ml of stannous chloride acid solution and 10 gm of zinc is added (all this reagents must be arsenic free)
3	Keep the solution aside for 40 min	Keep the solution aside for 40 min
4	Compare the stain obtained on mercuric chloride paper with standard solution.	Compare the stain obtained on mercuric chloride paper with standard solution.

Diagram



Regular arsenic apparatus with alternate device (a) and (b) for fixing mercuric chloride paper.

OBSERVATION

If the stain produced by test is no deeper than standard stain, then sample complies limit test for arsenic.

CONCLUSION

After 40 minutes, if the intensity of the yellow stain produced in the standard is more that in the test, the sample complies with the limit test of lead.

RESULT

Limit test of arsenic passes the test.

Thank you!

