

PHARMACEUTICAL INORGANIC CHEMISTRY- BP104T

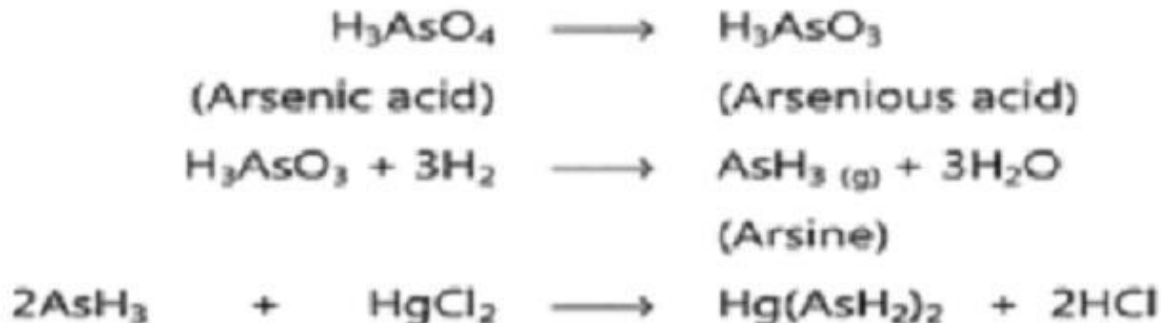
UNIT: 1 (IMPURITIES IN PHARMACEUTICAL SUBSTANCES)

CLASS: 6

TOPIC: LIMIT TEST FOR ARSENIC**Principle:**

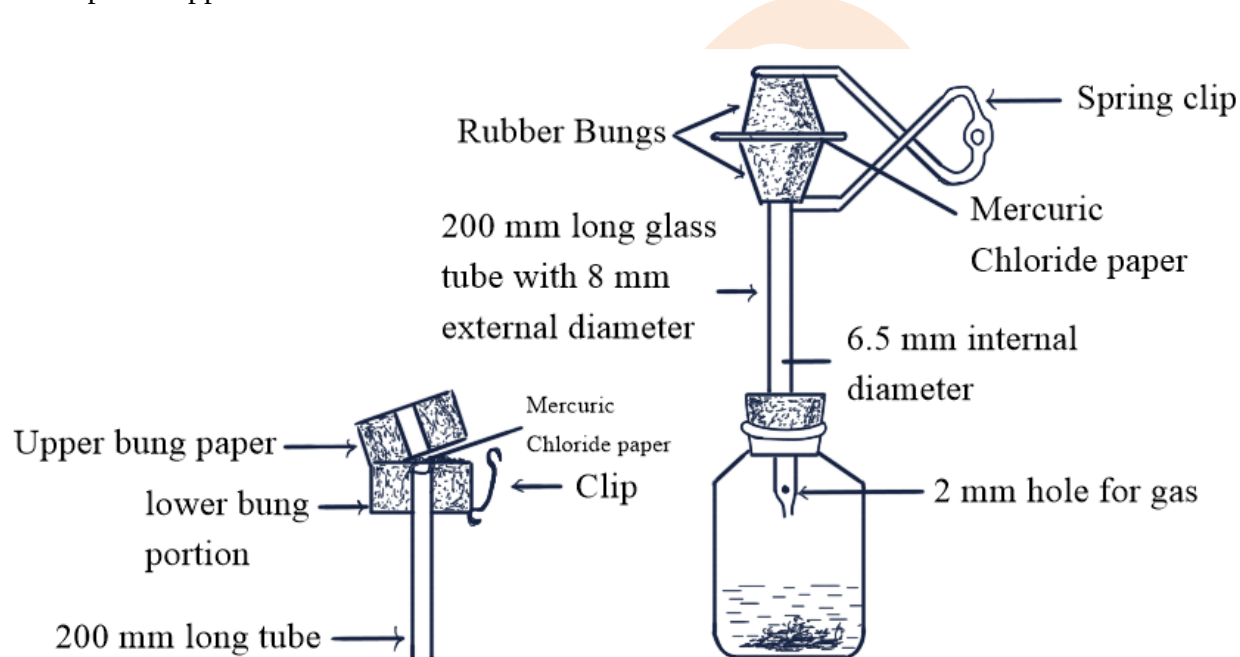
Limit test of Arsenic is based on the reaction of arsenic gas with hydrogen ion to form yellow stain on mercuric chloride paper in presence of reducing agents like potassium iodide.

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. The intensity and length of yellow stain on mercuric chloride paper will depend upon the amount of arsenic present in the sample.

**Apparatus:**

- It consists of a wide mouth glass bottle fitted with two rubber bungs through which glass tube (length 200mm, outer diameter 8.0mm and internal diameter 6.5mm) is passed.
- The tube has opened upper end and the lower end has a diameter of 1mm and a small hole in the side of lower end (2mm).
- Small diameter of lower end is meant for slow and smooth passage of arsine gas and a side hole provides an alternative passage for arsine if some water gets condensed at lower end of tube.

- Mercuric chloride paper is sandwiched between the rubber bungs with the help of spring clips.
- The upper rubber bung with the hole of 6.5mm diameter, exactly corresponding in position with the glass tube fitted into the lower bung.
- The glass tube is first slightly packed with cotton wool moistened with lead acetate to entrap any hydrogen sulphide gas otherwise it will provide some stain in mercuric chloride paper.
- Separate apparatus is used for test and for the standard solution.



Procedure:

Preparation of test stain:

1. The required amount of sample is placed in a wide mouthed bottle
2. To this add 1gm of potassium iodide and 10gm of granulated zinc
3. Immediately close the bottle with a stopper assembly
4. The acid present in the test solution and zinc reacts, produces nascent hydrogens which reacts with arsenious acid present in the sample and liberates arsine gas which passes through the tube and reacts with mercuric chloride paper and produces yellow stain.
5. The reaction is maintained for 40 minutes to ensure complete conversion of arsenious acid to arsine gas. Some heat, pressure are produced during the reaction due to the increased amount of gas inside the apparatus.

6. The rate of a reaction is increased by placing the total assembly on a warm surface.

Preparation of standard stain:

1. Standard stain is prepared by using dilute arsenic solution i.e., 0.132gm of arsenic in 100ml of distilled water.
2. 1ml of the above solution is diluted to 100ml. this solution is transferred into a wide mouthed bottle.
3. To this add 1gm of potassium iodide and 10gm of granulated zinc
4. Immediately close the bottle with a stopper assembly
5. The acid present in the test solution and zinc reacts, produces nascent hydrogens which reacts with arsenious acid present in the sample and liberates arsine gas which passes through the tube and reacts with mercuric chloride paper and produces yellow stain.
6. The reaction is maintained for 40 minutes to ensure complete conversion of arsenious acid to arsine gas. Some heat, pressure are produced during the reaction due to the increased amount of gas inside the apparatus.
7. The rate of a reaction is increased by placing the total assembly on a warm surface.

Observation:

- The stain produced in the filter paper fades quickly, so the comparison should be done quickly.
- If the intensity of the stain produced in the test is less than the standard, the test sample complies with the limit test for arsenic
- If the stain present in the filter paper becomes dark, the test should be repeated by using pure reagents

Note:

- The most suitable temperature for carry out this test is 40 °C.
- Cotton wool dipped in lead acetate solution is used to trap any hydrogen sulphide gas liberated with arsine gas.
- Care must be taken that the filter paper remains quite dry during the reaction.

- During succeeding tests the tube must be washed with hydrochloric acid, rinsed with water and dried.
- All the reagents used for this test should be free from arsenic.

2 MARKS QUESTIONS

1. Write the principle involved in limit test for arsenic
2. Draw a neat labeled diagram of Gutzeit apparatus

5 MARKS QUESTIONS

1. Explain the principle and procedure involved in limit test for arsenic
2. Describe the construction of Gutzeit apparatus

10 MARKS QUESTIONS

1. Explain the principle & procedure involved in limit test for arsenic with a neat labeled diagram of Gutzeit's apparatus