

Limit test for arsenic

- The original Gutzeit test was quantitative one and the liberated arsine gas is detected by covering the mouth of the flask with filter paper, on which small amount of silver nitrate crystals were placed. The liberated arsine turns silver nitrate complex black.

- In the modified test, filter paper dipped in mercuric chloride solution is used, which yields a yellow colored complex. It produces a stain in the filter paper.

Principle

- The arsenic impurity present in the sample is first converted into arsenious acid by the action of reducing agents like potassium iodide, Zinc, HCl and stannous chloride.

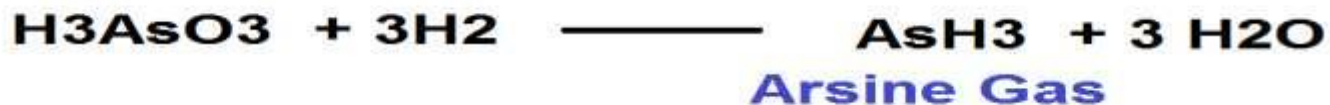


**Arsenic acid in
sample**

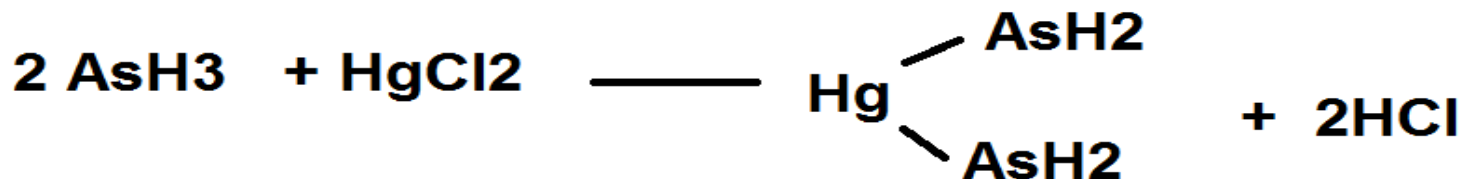


Arsenious acid

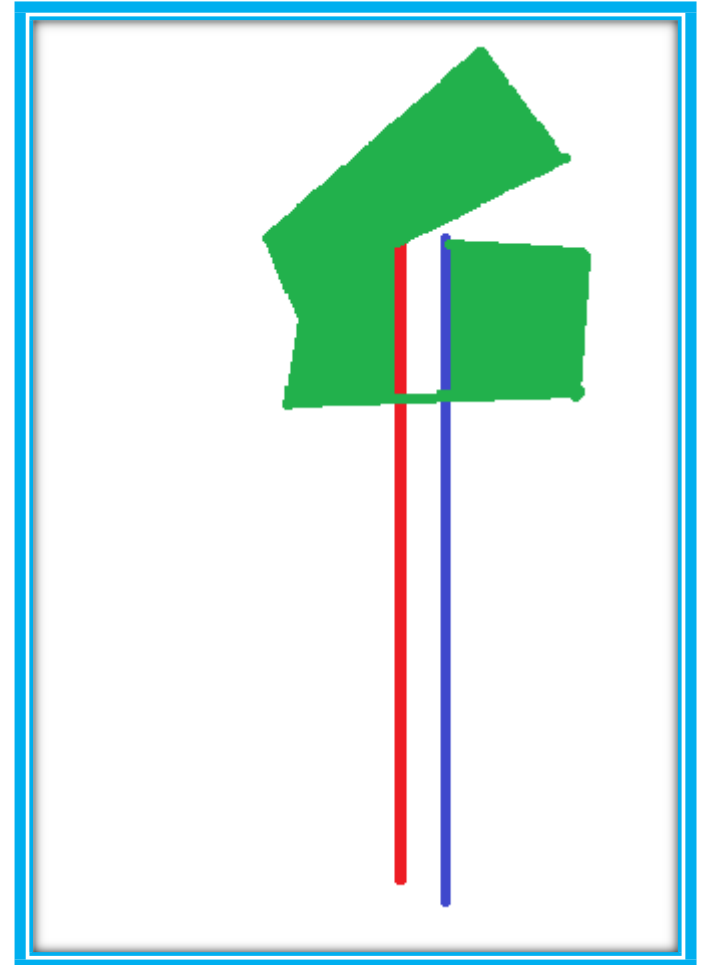
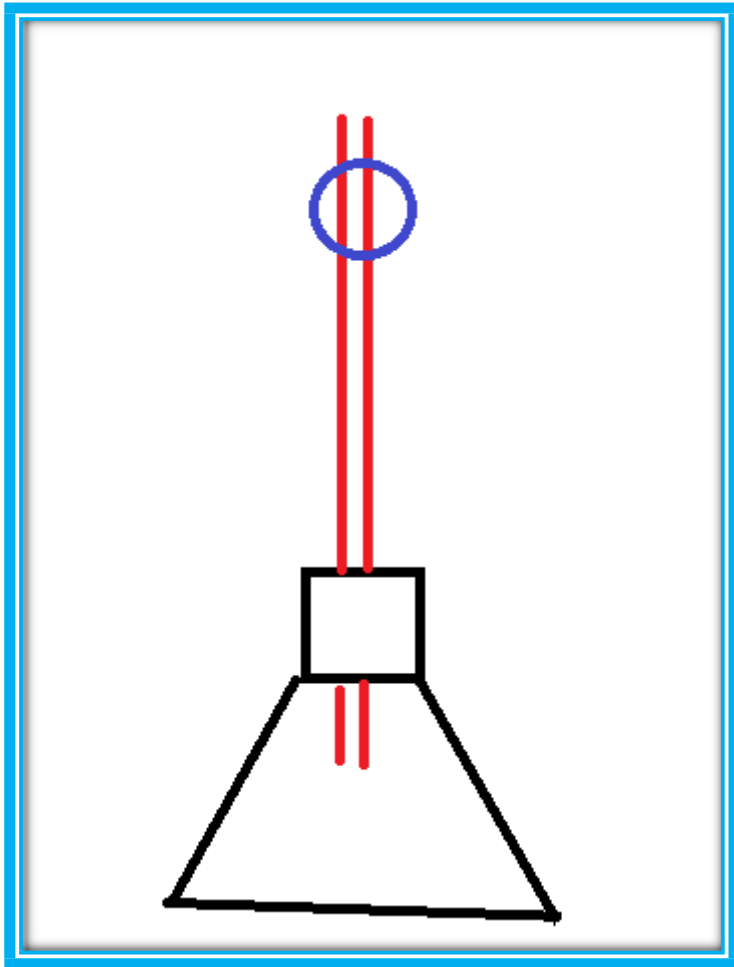
- The arsenious acid is then reduced to arsine by the action of nascent hydrogen which is produced by the reaction between Zinc and HCl.



- The liberated arsine gas reacts with mercuric chloride paper and forms yellow stain



Gutzeit apparatus



Procedure

- The required amount of sample solution is placed in the wide mouthed bottle.
- To this add 1 g of potassium iodide and 10 g of granulated zinc AST.
- Immediately close the bottle with stopper assembly.
- The acid present in the test solution and zinc reacts, produces nascent hydrogen 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.

- 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 in the inside of the apparatus.
- The rate of reaction is increased by placing the total assembly on a warm surface.

Standard of standard stain

- Standard stain is prepared by using dilute arsenic solution [0.132 g of arsenic in 100 ml]
- 1 ml is diluted to 100 ml .
- The comparison of the stain is done after the completion of the test immediately, so the test and standard should be carried out simultaneously.

Precaution

- If the stain present in the filter paper becomes dark, the test should be repeated by using pure reagents.
- The most suitable temperature for carry out this test is 40C.
- 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 the succeeding tests the tube must be washed with HCl AsT rinsed with water and dried.
- All the reagents used for this test should be free from arsenic and mentioned as AsT.