

LIMIT TESTS – 01

Limit Test for Arsenic

Principle, reactions, the Gutzeit apparatus, procedure and the viva questions examiners ask most.

01 – PRINCIPLE

How the test works

Arsenic in the sample is first reduced to the trivalent form (arsenious acid) by **potassium iodide** and **stannous chloride**. **Zinc and hydrochloric acid** produce nascent hydrogen, which reduces arsenious acid to **arsine gas** (AsH_3). The arsine rises through the apparatus and reacts with **mercuric chloride paper** to give a **yellow stain**.

IN PLAIN WORDS

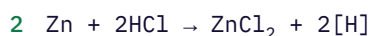
It is a comparison test. The stain from the sample is compared with the stain from a known amount of arsenic, run under the same conditions.

02 – REACTIONS

Four reactions to remember



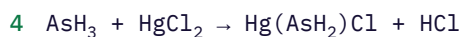
Arsenic acid is reduced to arsenious acid



Zinc and acid produce nascent hydrogen



Arsenious acid becomes arsine gas



Arsine forms the yellow stain on the paper

03 – APPARATUS

Gutzeit apparatus

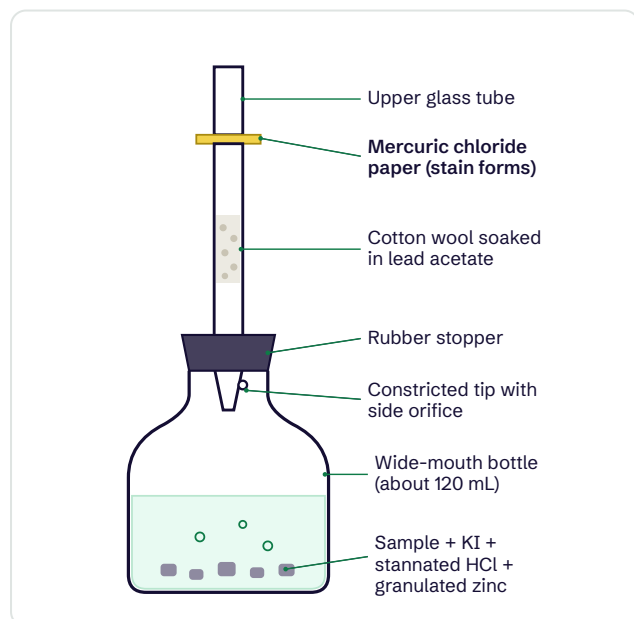


FIG. 1 – GUTZEIT APPARATUS, LABELLED

04 – REQUIREMENTS

What you need

Apparatus: two Gutzeit apparatus (test and standard), glass rod, analytical balance, water bath, mercuric chloride paper.

Reagents: potassium iodide AsT, stannated hydrochloric acid AsT, granulated zinc AsT, lead acetate solution, standard arsenic solution.

AsT = "arsenic test" grade, free from arsenic.

Reagent	Why it is there
KI + SnCl_2	Reduce pentavalent arsenic to trivalent
Zn + HCl	Produce nascent hydrogen to form arsine
Lead acetate cotton	Traps H_2S , which would give a black stain
HgCl_2 paper	Reacts with arsine to give the yellow stain

Run the test and the standard side by side

TEST

Dissolve the specified quantity of sample in water in the first bottle.

STANDARD

Take the specified volume of standard arsenic solution in the second bottle.

STEP 1

Add reagents

To each bottle, add KI AsT, stannated HCl AsT and granulated zinc AsT.

STEP 2

Assemble at once

Fit lead acetate cotton and mercuric chloride paper; close immediately so no arsine escapes.

STEP 3

Warm 40 min

Keep both in a water bath at about 40 °C for 40 minutes for steady gas evolution.

STEP 4

Compare stains

Remove both papers and compare the depth of the yellow stains side by side.

06 – RESULT

Pass or fail

PASSES



Test stain is **not deeper** than the standard stain.

FAILS



Test stain is **deeper** than the standard stain.

07 – PRECAUTIONS

Six things that protect the result

- Use arsenic-free (AsT) reagents and water throughout.
- Run the test and standard at the same time, under identical conditions.
- Keep mercuric chloride paper dry and away from sunlight.
- Assemble the apparatus immediately after adding zinc.
- If the test stain is darker, repeat with fresh reagents before concluding.
- Clean the apparatus with arsenic-free HCl, rinse with water and dry.

08 – KNOWLEDGE CHECK

Viva questions

Q1 Which apparatus is used for the limit test for arsenic?

The Gutzeit apparatus.

Q2 What gas is formed in the test?

Arsine (AsH_3), from nascent hydrogen reducing arsenious acid.

Q3 Why is cotton soaked in lead acetate used?

To trap hydrogen sulphide, which would give a black stain on the paper.

Q4 Why are KI and stannous chloride added?

To reduce pentavalent arsenic to the trivalent form, which forms arsine readily.

Q5 What colour stain indicates arsenic?

Yellow, on the mercuric chloride paper.

Q6 Why is the test kept at about 40 °C?

For steady, controlled gas evolution, so test and standard are comparable.

Q7 Why is a standard run alongside the test?

The limit test is comparative: the result depends on comparing stain depth with a known amount of arsenic.

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Revision notes for students. For official procedures, always follow the current pharmacopoeia monograph.