

Arsenic Test



Some host rocks, especially shales, may contain up to 10ppm of Arsenic. This can impart levels in excess of 50ppb (50µg/l) to the water, definitely too much by today's standards of less than 10ppb.

1.1-Test 46 Arsenic employs quality components and highly stable, user-friendly liquid reagent concentrates. The perfect way of obtaining more information on this hazard at every place where ground water is utilized.



**RIELE**

46 ARSENIC

5 - 200 ppb \ 50 Tests

Catalysed HCl-free As-bromomercurate colorimetry

Occurrence: Well-known minerals of arsenic are Arsenopyrite FeAsS and the arsenosulfides of copper from which arsenic is recovered as an unwanted by-product during the refining process. The quantities so obtained far exceed the industrial demand. Hazardous uses of former times included biocides for agriculture, wood preservatives, and the pigment "Scheele's Green" CuHAsO₃. Eruptive rocks contain an average of 1,5ppm. Among the sedimentary rocks lime- and sandstones average around 1ppm As, with shales and slates sometimes enriched up to values above 10ppm. Ground water from such horizons will contain measurable quantities of arsenic. The most prominent region is Bangla Desh, where wells were drilled to replace the often polluted surface waters in common use. In 1993 one began to realize that many of these sources, while now delivering bacteriologically acceptable water, carried high levels of arsenic, exceeding 0,05ppm (50ppb, 50µg/l) at 20% of the sampling points. This value, formerly accepted as a tolerance level in many countries, is considered decidedly too high today. The WHO recommends <10ppb, which the US-EPA has also adopted.

Arsine generation: The determination of arsenic in water is based on the conversion to arsine AsH₃, its volatile hydride. The reactant is nascent hydrogen, either liberated from a medium of zinc (Reagent As-3) in acid, aluminium in caustic alkali or by an agent such as lithium borohydride. The former option, being easily reproducible, is most commonly used in field analysis. Classical methods employed hydrochloric acid, necessarily concentrated to avoid excessive sample dilution. This was unpleasant to handle, with the additional problem of diffusing through the walls of PE-bottles used in kits, corroding the contents of the latter. A more recent alternative are non-volatile polyhydroxy carbonic acids as used in the food industry (Reagent As-1). These required additional metal catalysts in a dilute medium of mineral acid (Reagent As-2). As liquid concentrates, both are easier and cleaner in application than the powders used in comparable products. 15 drops of each reagent added to a 50ml sample result in a copious evolution of

micro-bubbles of gas evident in the arsine generator pictured at the bottom right. In the rare cases of sulfide presence, able to simulate small quantities of As, this has to be oxidized with persulfate. The tiny amounts of AsH₃ potentially evolved in water analysis are completely harmless. A tough, high quality glass bottle with a pressure seal adds to the certainty of collecting all the evolved arsine, an inherent problem with some of the commonly supplied plastic flip-top bottles.

Arsine determination: In semi-quantitative field tests, arsine is collected by a small filter paper disk impregnated with colourless mercuric bromide, HgBr₂. This acts as a membrane, here of 4.5mmØ, through which the evolved gas must pass. Within the layer of filter-paper, the (HgBr)⁺-cation is taken to consecutively replace the hydrogen atoms of incoming AsH₃ to give AsH₂(HgBr), yellow AsH(HgBr)₂ followed by brown As(HgBr)₃ and ultimately black Hg₃As₂. The colour tint of the reaction products represents the arsine delivered at a fixed time and possibly temperature. To secure reproducibility it is necessary to control the state of the HgBr₂ within the filter paper. Classical technology requires them to be freshly impregnated and dried, whereas test kits tend to supply these ready-for-use. Although reactive when fresh, there is a danger of activity loss with age and consequently stunted colour development. The kit therefore includes an alcoholic solution of HgBr₂ for impregnating the disks as required. Although all other reagents are highly stable, there is the danger of the solvent evaporating in the HgBr₂ dropping-bottle in time especially under warmer storage conditions. In such a case a small quantity (~1ml) of this in one form or another (methylated spirit) should be added with the tightly closed bottle shaken vigorously after the addition. The test can be used for determining Arsenic in other media that can be brought into aqueous solution. There is extensive literature on this reaction and its potential interferences since first described by *Gutzeit* in 1879. H₂S is recognized by a yellow spot differing from that of AsH₃ by its spread in the paper.

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Analysis procedure:

- Transfer filter paper disks required for the next 1 to 3 days to into the black screw cap with central stud, impregnate with HgBr₂ in this free-floating state and let dry. Retain these separately in the cap in the combined enclosure as shown, in a dark place.
- Place one dry impregnated disk into the cap of the glass bottle.
- Fill the bottle to the mark (50ml) with water sample.
- (Dissolve 1 spoon of persulfate and set aside for 5' in case of sulfide in water. Check: 3.2-Test 25).
- Add 15 drops each of **As-1** and **As-2** followed by 3 spoons of of **As-3**.
- Plug the bottle with the mushroom-shaped inset with 4.5mmØ orifice using the O-ring as a seal between both.
- Close with the cap containing the impregnated disk.
- Swirl gently, set aside for 30 minutes.
- Remove cap, compare the colour of the disk according to the scale inside the box.
- Wash out the bottle directly afterwards to prevent reacted zinc granules from forming a cake adhering to the bottom of the glass. A subsequent removal may require concentrated mineral acid.
- Avoid contact with HgBr₂. Use tweezers. Retain contaminated waste for qualified disposal.