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Workplace air — Determination of particulate arsenic and arsenic compounds and arsenic trioxide vapour — Method by hydride generation and atomic absorption spectrometry

Air des lieux de travail — Dosage de l'arsenic particulaire, des composés particulaires de l'arsenic et des vapeurs de trioxyde d'arsenic — Méthode par production d'hydrures et spectrométrie d'absorption atomique

Reference number
ISO 11041:1996(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11041 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

Annex A of this International Standard is for information only.

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Workplace air — Determination of particulate arsenic and arsenic compounds and arsenic trioxide vapour — Method by hydride generation and atomic absorption spectrometry

WARNING — Arsenic and arsenic compounds are toxic and are recognized as human carcinogens (see reference [1] in annex A). Avoid any exposure by inhalation. Personal protection (e.g. an effective respirator) must be used in all cases where exposure to arsenic or arsenic compounds is possible.

1 Scope

This International Standard specifies a method for the determination of the mass concentration of particulate arsenic and arsenic compounds and arsenic trioxide vapour in workplace air, using either continuous-flow hydride generation or flow-injection-analysis hydride generation and atomic absorption spectrometry. The method is not suitable for determination of arsenic in the form of metal arsenides which decompose in the presence of water or acid (see 10.1).

The method is applicable to the determination of masses of approximately 100 ng to 125 µg of arsenic per sample, for analysis of test solutions prepared using sample solution aliquots in the recommended range (see 9.3.2). The concentration range for arsenic in air, for which this procedure is applicable, is determined in part by the sampling procedure selected by the user.

The method is applicable to personal and fixed-location sampling.

A number of transition metals may interfere with the determination of arsenic by hydride generation and atomic absorption spectrometry (see 10.3)

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3585:1991, *Borosilicate glass 3.3 — Properties.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 6955:1982, *Analytical spectroscopic methods — Flame emission, atomic absorption and atomic fluorescence — Vocabulary.*

ISO 7708:1995, *Air quality — Particle size fraction definitions for health-related sampling.*

ISO 8655-1:—[1], *Piston and/or plunger operated volumetric apparatus (POVA) — Part 1: Definitions.*

ISO 8655-2:—[1], *Piston and/or plunger operated volumetric apparatus (POVA) — Part 2: Operating considerations.*

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[1] To be published.

ISO 8655-3:—[1], *Piston and/or plunger operated volumetric apparatus (POVA) — Part 3: Methods of test.*

ISO 8655-4:—[1], *Piston and/or plunger operated volumetric apparatus (POVA) — Part 4: Specifications.*

ISO 8756:1994, *Air quality — Handling of temperature, pressure and humidity data.*

EN 482:1994, *Workplace atmospheres — General requirements for the performance of procedures for the measurement of chemical agents.* EN 482, CEN, Brussels, Belgium (1994).

EN 1232:—[1], *Workplace atmospheres — Pumps for personal sampling of chemical agents — Requirements and test methods.*

3 Principle

3.1 Particulate arsenic and arsenic compounds and arsenic trioxide vapour are collected by drawing a measured volume of air through a cellulose ester membrane filter and a back-up paper pad impregnated with sodium carbonate and mounted in a sampler designed to collect the inhalable fraction of airborne particles.

3.2 The cellulose ester membrane filter, back-up paper pad and collected sample are wet-ashed using nitric acid, sulfuric acid and hydrogen peroxide. The nitric acid and hydrogen peroxide are removed by boiling on a hotplate until dense, white fumes of sulfur trioxide are evolved, and the sample solution is then allowed to cool and diluted to a given volume with water.

NOTE 1 The wet-ashing procedure specified in 8.2.2 is based upon a NIOSH procedure (see reference [2] in annex A) which has been modified to avoid taking the sample solution to dryness.

3.3 A test solution is prepared by transferring an aliquot of the sample solution to a volumetric flask, together with appropriate volumes of dilute sulfuric acid, concentrated hydrochloric acid and potassium iodide solution, and diluting to volume with water.

3.4 The test solution is reacted with sodium tetrahydroborate solution in a continuous-flow hydride generation system or flow-injection-analysis hydride generation system to liberate arsine and hydrogen. These gaseous products are separated from the reaction liquid in a gas/liquid separator and carried by an inert purge gas into a silica or quartz absorption cell. This absorption cell is mounted in the optical path of an atomic absorption spectrometer equipped with an arsenic hollow cathode lamp or electrodeless discharge lamp, and it is heated either electrically or by an oxidizing air/acetylene flame.

NOTE 2 This International Standard describes the use of two types of hydride generation system. Continuous flow systems function by pumping a continuous stream of test solution to the mixing piece, and such systems generate a constant atomic absorption signal. Flow injection analysis systems inject a discrete volume of test solution, and produce a transient atomic absorption signal.

3.5 Absorbance measurements are made at 197,2 nm or 193,7 nm, and results are obtained by the analytical-curve technique (see ISO 6955:1982, subclause 6.1.1), or the analyte addition technique (see ISO 6955:1982, subclause 6.1.3).

4 Reactions

4.1 In most workplace situations where exposure to arsenic can occur (e.g. in the refining of base metals, welding and other hot metal processes) a significant proportion of the arsenic is present in the form of arsenic trioxide vapour (see reference [3] in annex A). This vapour is collected by reaction with sodium carbonate on an impregnated back-up paper pad.

$$As_2O_3 + Na_2CO_3 \rightarrow 2NaAsO_2 + CO_2$$

4.2 The majority of arsenic compounds which are commonly found in samples of workplace air are converted to soluble arsenate ions (AsO_4^{3-}) by the wet-ashing procedure specified in 8.2.2. However, if there is any doubt about the effectiveness of this procedure for dissolution of particulate arsenic compounds which could be present in the test atmosphere, investigate before proceeding with the method (see 10.2).

4.3 Prior to hydride generation (see 4.4) arsenate ions (AsO_4^{3-}) are reduced to arsenite ions (AsO_2^-) by reaction with potassium iodide (see 8.2.4).

$$AsO_4^{3-} + 2I^- + 4H^+ \rightarrow AsO_2^- + I_2 + 2H_2O$$

This reduction is necessary since pentavalent arsenic gives a lower analytical response than trivalent arsenic because it is less rapidly converted to arsine.

4.4 Hydride generation occurs as a result of the reaction between trivalent arsenic and nascent hydrogen produced by the action of hydrochloric acid on sodium tetrahydroborate solution.

$$BH_4^- + H^+ + 3H_2O \rightarrow H_3BO_3 + 8H \qquad \ldots (1)$$

$$AsO_2^- + H^+ + 6H \rightarrow AsH_3 + 2H_2O \qquad \ldots (2)$$

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[1] To be published.

4.5 Arsenic atoms are produced from arsine by the action of heat in a silica or quartz absorption cell, heated either by a lean air/acetylene flame or electrically.

5 Reagents

During the analysis, use only reagents of analytical grade, and only water as specified in 5.1.

5.1 Water, complying with the requirements for ISO 3696 grade 2 water (electrical conductivity less than 0,1 mS/m and resistivity greater than 0,01 MΩ·m at 25 °C).

5.2 Sodium carbonate, 1 mol/l solution in 5 % (*V/V*) glycerol solution.

Weigh 10,6 g of sodium carbonate (Na_2CO_3) into a 250 ml beaker (6.2.1.1). Add 5 ml of glycerol and 50 ml of water (5.1) and swirl to dissolve. Quantitatively transfer the solution to a 100 ml one-mark volumetric flask (6.2.1.5), dilute to the mark with water, stopper and mix thoroughly.

5.3 Hydrochloric acid (HCl), concentrated $\rho \approx$ 1,18 g/ml, 35 % (*m/m*) to 36 % (*m/m*).

The concentration of arsenic shall be less than 0,01 µg/ml.

WARNING — Concentrated hydrochloric acid is corrosive, and hydrochloric acid vapour is irritant. Avoid exposure by contact with the skin or eyes, or by inhalation of fumes. Personal protective equipment (e.g. gloves, face shield or safety spectacles, etc.) must be used when working with the concentrated or diluted hydrochloric acid, and concentrated hydrochloric acid must be used in a fume hood. The vapour pressure of hydrochloric is high, therefore beware of pressure build-up in stoppered flasks when preparing acid/water mixtures.

5.4 Hydrochloric acid, diluted 1 + 1.

Pour approximately 900 ml of water (5.1) into a 2 000 ml one-mark volumetric flask (6.2.1.5). Carefully add 1 000 ml of concentrated hydrochloric acid (5.3) to the flask and swirl to mix. Allow to cool, dilute to the mark with water, stopper and mix thoroughly.

NOTE 3 This is used as the solvent blank, as defined in ISO 6955:1982, subclause 5.4.2, but in this International Standard the solvent blank is referred to as the acid blank.

5.5 Hydrochloric acid, diluted 1 + 4.

Pour approximately 700 ml of water (5.1) into a 1 000 ml one-mark volumetric flask (6.2.1.5). Carefully add 200 ml of concentrated hydrochloric acid (5.3) to the flask and swirl to mix. Allow to cool, dilute to the mark with water, stopper and mix thoroughly.

5.6 Nitric acid (HNO_3), concentrated, $\rho \approx$ 1,42 g/ml, 69 % (*m/m*) to 71 % (*m/m*).

The concentration of arsenic shall be less than 0,01 µg/ml.

WARNING — Concentrated nitric acid is corrosive and oxidizing, and nitric acid fumes are irritant. Avoid exposure by contact with the skin or eyes, or by inhalation of fumes. Personal protective equipment (e.g. gloves, face shield or safety spectacles, etc.) must be used when working with the concentrated or diluted nitric acid, and concentrated nitric acid must be used in a fume hood.

5.7 Sulfuric acid (H_2SO_4), concentrated, $\rho \approx$ 1,84 g/ml, about 98 % (*m/m*).

The concentration of arsenic shall be less than 0,05 µg/ml.

WARNING — Concentrated sulfuric acid is corrosive and causes burns. Avoid exposure by contact with the skin or eyes. Personal protective equipment (e.g. gloves, face shield or safety spectacles, etc.) must be used when working with the concentrated or diluted sulfuric acid. Fumes produced by heating concentrated sulfuric acid are irritant, and this operation must therefore be carried out in a fume hood. Caution must be exercised if adding water to sulfuric acid, since this reacts violently with water (acid/water mixtures must be prepared by adding acid to water).

5.8 Hydrogen peroxide (H_2O_2), approximately 30 % (*m/m*) solution.

The concentration of arsenic shall be less than 0,01 µg/ml.

WARNING — Hydrogen peroxide is corrosive and oxidizing. Avoid exposure be contact with the skin or eyes. Personal protective equipment (e.g. gloves, face shield or safety spectacles, etc.) must be used when working with protective equipment hydrogen peroxide.

5.9 Potassium iodide, 100 g/l solution.

Weigh 10,0 g of potassium iodide (KI) into a 250 ml beaker (6.2.1.1). Add 50 ml of water (5.1) and swirl to dissolve. Quantitatively transfer the solution to a 100 ml one-mark volumetric flask (6.2.1.5), dilute to the mark with water, stopper and mix thoroughly.

Prepare a fresh solution each month.

5.10 Sulfuric acid, diluted 1 + 9.

Carefully add 25 ml of concentrated sulfuric acid (5.7) to 200 ml of water (5.1) in a 1 litre beaker. Swirl to mix, allow to cool and quantitatively transfer to a 250 ml one-mark volumetric flask (6.2.1.5). Dilute to the mark with water, stopper and mix thoroughly.

5.11 Arsenic stock standard solution, corresponding to 1 000 mg of As per litre.

5.11.1 Use a commercially available arsenic standard solution at a concentration of 1 000 mg/l. Observe the manufacturer's expiry date or recommended shelf-life.

Alternatively, prepare an arsenic standard solution according to the procedure specified in 5.11.2.

5.11.2 Accurately weigh 1,320 g ± 0,001 g of arsenic trioxide (As_2O_3) into a 50 ml beaker (6.2.1.1), add 10 ml of concentrated hydrochloric acid (5.3), cover with a watch glass (6.2.1.2) and heat to approximately 150 °C on the hotplate (6.2.5) in a fume hood until dissolution is complete. Remove the beaker from the hotplate, allow to cool, quantitatively transfer the solution to a 1 000 ml one-mark volumetric flask (6.2.1.5), dilute to the mark with hydrochloric acid diluted 1 + 1 (5.4), stopper and mix thoroughly.

This solution may be stored in a polypropylene bottle (6.2.2) for up to one year.

WARNING — Arsenic trioxide is toxic and is recognized as a human carcinogen (see reference [1] in annex A). See the general warning about arsenic or arsenic compounds, just after the title of this International Standard.

5.12 Arsenic working standard solution A, corresponding to 10 mg of As per litre.

Using a pipette (6.2.1.3), accurately add 1,00 ml of stock arsenic solution (5.11) to a 100 ml one-mark volumetric flask (6.2.1.5), dilute to the mark with hydrochloric acid diluted 1 + 1 (5.4), stopper and mix thoroughly.

This solution may be stored in a polypropylene bottle (6.2.2) for up to one month.

5.13 Arsenic working standard solution B, corresponding to 1 mg of As per litre.

Using a pipette (6.2.1.3), accurately add 10 ml of working arsenic solution A (5.12) to a 100 ml one-mark volumetric flask (6.2.1.5), dilute to the mark with hydrochloric acid diluted 1 + 1 (5.4), stopper and mix thoroughly.

This solution may be stored in a polypropylene bottle (6.2.2) for up to one month.

5.14 Sodium tetrahydroborate, solution corresponding to between 2 g and 20 g of sodium tetrahydroborate per litre in 0,1 mol/l sodium hydroxide solution.

Prepare a sodium tetrahydroborate solution at the concentration recommended by the manufacturer of the hydride generation system (6.2.8). Weigh between 2 g and 20 g of sodium tetrahydroborate ($NaBH_4$) pellets or powder and 4 g of sodium hydroxide (NaOH) pellets into a 1 litre beaker (6.2.1.1). Add 200 ml of water (5.1) and swirl to mix. Quantitatively transfer the solution to a 1 000 ml one-mark volumetric flask (6.2.1.5), filtering through a membrane filter using a suction filtration apparatus (6.2.6). Dilute to the mark with water (5.1), stopper and mix thoroughly.

Prepare a fresh solution daily.

NOTES

4 Filtration of the solution is necessary to remove undissolved particulate material which might otherwise cause clogging of the tubing or mixing piece of the hydride generation system (6.2.8). The addition of alkali minimizes hydrolysis of the sodium tetrahydroborate solution.

5 A few drops of anti-foaming agent may be added to the solution to reduce foaming in the gas/liquid separator of the hydride generation system (6.2.8) which may result in a noisy baseline signal.

6 The solution should be stored in a polypropylene bottle (6.2.2) if it is not transferred to the reductant reservoir of the continuous hydride generation system (6.2.8) immediately after preparation (see 8.4.2.2). The top of the bottle should not be fully tightened or pressure will build up due to the slow release of hydrogen.

5.15 Sodium hydroxide, 5 g/l solution.

Weigh 5,0 g of sodium hydroxide (NaOH) pellets into a 1 litre beaker (6.2.1.1). Add 250 ml of water (5.1) and swirl to dissolve. Quantitatively transfer the solution to a 1 000 ml volumetric flask (6.2.1.5), dilute to the mark with water, stopper and mix thoroughly.

5.16 Laboratory detergent solution, suitable for cleaning samplers and laboratory apparatus, diluted with water (5.1) according to the manufacturer's instructions.

5.17 Inert purge gas, for example argon or nitrogen, supplied in a cylinder or as a cryogenic fluid.

5.18 Air, compressed and filtered.

NOTE 7 This gas is not required if the silica or quartz absorption cell (6.2.9) used is electrically heated.

5.19 Acetylene, in a cylinder.

NOTE 8 This gas is not required if the silica or quartz absorption cell (6.2.9) used is electrically heated.

6 Apparatus

6.1 Sampling equipment

6.1.1 Samplers, for collection of the inhalable fraction of airborne particles (see 7.1.1) as defined in ISO 7708, suitable for use with the cellulose ester membrane filters and back-up paper pads (6.1.2) and compatible with the sampling pumps (6.1.3) used.

NOTES

9 A number of different terms are used to describe samplers designed for collection of the inhalable fraction of airborne particles, for example, sampling heads, filter holders, filter cassettes and air monitoring cassettes.

10 In general, the collection characteristics of inhalable samplers are such that particulate material collected on the filter is the inhalable fraction of airborne particles, and any deposited on the internal surfaces of the sampler is not of interest. However, some samplers are designed such that airborne particles which pass through the entry orifice(s) constitute the inhalable fraction; in which case any particulate material deposited on the internal surfaces of the sampler is part of the sample. Certain samplers of this type incorporate an internal filter cassette or cartridge which may be removed from the sampler to enable this material to be easily recovered.

11 Samplers which are assembled by means of screw-threaded fittings may be unsuitable for use with a cellulose ester membrane filter and a back-up paper pad. The high restriction of a cellulose ester membrane filter, compared with that of a paper pad, means that there is a tendency for air to take the path of least resistance and to be drawn along screw threads and in through the edges of the paper pad, rather than through the cellulose ester membrane filter. Leakage can sometimes be eliminated by tightening screw-threaded fittings as much as possible to compress and seal the edges of the paper pads, but this is not fully effective for certain types of sampler. Samplers with push-fit components can, in general, be used more reliably.

12 Samplers manufactured in non-conducting material have electrostatic properties which may influence representative sampling. Electrostatic influences should be reduced, where possible, by using samplers manufactured from conducting material.

6.1.2 Cellulose ester membrane filters and back-up paper pads, of a diameter suitable for use in the selected sampler (6.1.1).

The mass of arsenic of a cellulose ester membrane filter and back-up paper pad shall be less than 0,01 µg.

6.1.2.1 The cellulose ester membrane filters shall have a retentivity not less than 99 % for particles of median aerodynamic diameter 0,3 µm (see ISO 7708:1995, subclause 2.2).

6.1.2.2 The back-up paper pads shall be impregnated with sodium carbonate in an area where arsenic contamination is known to be low, using the following procedure:

Place the paper pads on a clean polytetrafluoroethylene (PTFE) sheet or similar, inert, flat surface (6.2.4). Establish the volume of sodium carbonate solution (5.2) required to just wet the entire paper pad after the solution has been allowed to spread for a few minutes. Dispense this volume of sodium carbonate solution onto each paper pad and allow to dry for several hours at room temperature. Store the paper pads impregnated with sodium carbonate in an airtight container and use within one week of preparation.

NOTES

13 The volume of sodium carbonate solution required to impregnate the back-up paper pads is typically 175 µl for a 25 mm diameter paper pad or 400 µl for a 37 mm diameter paper pad.

14 The drying time for paper pads impregnated with sodium carbonate may be reduced by placing them in an oven at 40 °C for 45 min.

15 Glass-fibre or quartz-fibre filters impregnated with sodium carbonate have also been shown to be efficient for collecting arsenic trioxide vapour (see reference [4] in annex A) and may be used as an alternative to cellulose ester membrane filters and back-up paper pads impregnated with sodium carbonate. Neither glass-fibre nor quartz-fibre filters are dissolved by the wet-ashing procedure specified in 8.2.2, but this may be modified to permit their use (see note 33).

6.1.3 Sampling pumps, complying with the requirements of EN 1232, with an adjustable flow rate, incorporating a flowmeter or a flow-fault indicator, and capable of maintaining the appropriate flow rate (see 7.1.1) to within ± 5 % of the nominal value throughout the sampling period (see 7.1.2). For personal sampling, the pumps shall be capable of being worn by a person without impeding normal work activity. The pumps shall give a pulsation-free flow (if necessary, a pulsation damper shall be incorporated between the sampler and the pump, as near to the pump as possible).

NOTE 16 Flow-stabilized sampling pumps may be required to maintain the flow rate within the limits specified in 6.1.3.

6.1.4 Portable flowmeter, capable of measuring the appropriate flow rate (see 7.1.1) to within ± 5 %, and calibrated against a primary standard, i.e. a flowmeter of which the accuracy is traceable to national standards.

NOTES

17 The flowmeter incorporated in the sampling pump may be used provided that it has adequate sensitivity, that it has been calibrated against a primary standard with a loaded sampler in line, and that it is read whilst in a vertical orientation if it is of the supported float type. However, it is important to ensure that there are no leaks in the sampling train between the sampler and the flowmeter, since in this event a flowmeter in the sampling pump or elsewhere in line will give an erroneous flow rate.

18 A soap bubble flowmeter may be used as a primary standard, provided its accuracy is traceable to national standards.

19 If appropriate (see 7.1.3.2), the atmospheric temperature and pressure at which the flowmeter was calibrated should be recorded.

6.1.5 Ancillary equipment, including flexible plastics tubing of a diameter suitable for making a leakproof connection from the samplers (6.1.1) to the sampling pumps (6.1.3); belts or harnesses to which the sampling pumps can conveniently be fixed, unless they are small enough to fit in workers' pockets; flat-tipped forceps for loading and unloading cellulose ester membrane filters and paper pads into samplers; and filter-transport cassettes or similar, if required (see 7.4.1), to transport samples to the laboratory.

6.1.6 Thermometer, 0 °C to 50 °C, graduated in divisions of 1 °C or better, for measurement of atmospheric temperature (see 7.1.3).

6.1.7 Barometer, for measurement of atmospheric pressure (see 7.1.3).

6.2 Analytical or laboratory apparatus

Ordinary laboratory apparatus, and

6.2.1 Glassware, made of borosilicate glass 3.3 complying with the requirements of ISO 3585.

NOTE 20 It is preferable to reserve a set of glassware for analysis of arsenic by this method. Heavily contaminated glassware in general usage may not be satisfactorily cleaned by the cleaning procedure specified in 8.1.4.

6.2.1.1 Beakers, of capacity 50 ml for wet-ashing of cellulose ester membrane filters and back-up paper pads of the diameter used in the sampler (see 8.2.2), and for preparation of the arsenic stock standard solution (5.11.2); of capacity 250 ml for preparation of the sodium carbonate solution (5.2) and the potassium iodide solution (5.9); and of capacity 1 litre for preparation of sodium tetrahydroborate solution (5.14) and sodium hydroxide solution (5.15).

6.2.1.2 Watch glasses, to fit the 50 ml beakers (6.2.1.1).

6.2.1.3 One-mark pipettes, complying with the requirements of ISO 648, as an alternative to piston-operated volumetric apparatus (6.2.3).

6.2.1.4 Measuring cylinders, of capacities between 10 ml and 1 litre.

6.2.1.5 One-mark volumetric flasks, of capacities between 10 ml and 2 000 ml, complying with the requirements of ISO 1042.

6.2.2 Polypropylene bottles, of capacity 1 litre.

NOTE 21 Bottles made of alternative plastics may be used, provided that they are suitable for the intended use (see 5.11.2, 5.12, 5.13 and 5.14).

6.2.3 Piston-operated volumetric apparatus, complying with the requirements of ISO 8655-1 to ISO 8655-4. Automatic pipettes, as an alternative to one-mark pipettes (6.2.1.3) for preparation of the working standard solutions (5.12 and 5.13), calibration solutions (see 8.3) and sample solutions (see 8.2.4), and dispensers for dispensing acids and potassium iodide solution (see 8.2 and 8.3).

6.2.4 PTFE sheet, or other similar inert flat surface suitable for treatment of filters and paper pads with sodium carbonate solution.

6.2.5 Hotplate, thermostatically controlled, capable of maintaining surface temperatures of approximately 150 °C (see 8.1.2), 175 °C and 200 °C (see 8.2.2).

NOTE 22 The efficiency of thermostatting of hotplates is sometimes deficient, and the surface temperature can also vary considerably with position on a hotplate with a large surface area. It may therefore be useful to characterize the performance of the hotplate before use.

6.2.6 Suction filtration apparatus

6.2.6.1 Filter funnel with support assembly, for filtration through a 47 mm diameter filter, made of borosilicate glass 3.3 complying with the requirements of ISO 3585.

6.2.6.2 Conical flask, of capacity 1 litre, either standard or Buchner type according to the design of the filter funnel with support assembly (6.2.6.1), which may incorporate the vacuum connection, and made of borosilicate glass 3.3 complying with the requirements of ISO 3585.

6.2.6.3 Filter pump, water-operated or **vacuum pump,** connected to the filter funnel with support assembly (6.2.6.1) or the conical flask (6.2.6.2) with plastics tubing (6.1.5).

6.2.6.4 Membrane filters, of diameter 47 mm and pore size 0,8 µm, made of cellulose ester, PVC or other material not degraded by sodium tetrahydroborate solution (5.14).

6.2.7 Atomic absorption spectrometer, equipped with an arsenic hollow cathode lamp or electrodeless discharge lamp. If the absorption cell (6.2.9) is heated by an air/acetylene flame, the atomic absorption spectrometer shall be fitted with an air/acetylene burner assembly, suitable for mounting the absorption cell, and supplied with compressed air (5.18) and acetylene (5.19).

6.2.8 Hydride generation system, of one of the types described in 6.2.8.1 and 6.2.8.2.

6.2.8.1 Continuous-flow hydride generation system, set up and operated according to the manufacturers' instructions; incorporating

a) reservoirs for sodium tetrahydroborate solution and acid blank;

b) an autosampler for presentation of the sample solution (optional);

c) an inert switching valve(s), either solenoid or pneumatically actuated, to facilitate switching between sample and acid blank streams (optional);

d) peristaltic pumps or a multi-channel peristaltic pump, fitted with appropriate acid-resistant pump tubing;

e) a chemically inert mixing piece(s) to facilitate mixing of acid blank or test solution, sodium tetrahydroborate solution and inert gas streams;

f) a reaction coil (optional);

g) a gas/liquid separator, with appropriate inlets for the reaction liquid stream and inert purge gas, and outlets for waste liquid and the purge gas plus gaseous products.

A schematic diagram of a typical system is given in figure 1.

NOTE 23 Continuous-flow hydride generation systems all work on the same principle, but the plumbing of the various systems is different. In particular, the configuration of some continuous flow hydride generation systems is such that there is (are) no switching valve(s), and both acid and test solutions are continuously pumped to an additional mixing piece situated upstream of the mixing piece where the sodium tetrahydroborate solution is introduced.

6.2.8.2 Flow-injection-analysis hydride generation system, set up and operated according to the manufacturer's instructions; incorporating

a) reservoirs for sodium tetrahydroborate solution and acid blank;

b) multi-channel peristaltic pumps, fitted with appropriate acid-resistant pump tubing;

c) an autosampler for presentation of the sample solution;

d) an inert injection valve, either solenoid or pneumatically actuated, to inject a reproducible volume of sample solution into the acid blank stream;

e) a chemically inert mixing piece(s) to facilitate mixing of acid blank or test solution, sodium tetrahydroborate solution and inert purge gas streams;

f) a reaction coil (optional);

g) a gas/liquid separator, with an inlet for the reaction liquid stream and outlets for waste liquid and the purge gas plus gaseous products.

A schematic diagram of a typical system is given in figure 2.

WARNING — Arsine (AsH_3) is generated when solutions containing arsenic are reacted with sodium tetrahydroborate. This gas is very toxic, but it will normally be produced only in very small quantities. However, in order to eliminate the possibility of exposure to arsine, it is essential that the liquid waste container used be equipped with efficient local exhaust ventilation to prevent any gases emanating from the liquid waste from entering the general laboratory environment.

6.2.9 Absorption cell, made of silica or quartz, heated either electrically or by an air/acetylene flame, and mounted in the optical path of the atomic absorption spectrometer (6.2.7).

NOTE 24 Spray from the gas/liquid separator may be carried into the absorption cell by the argon stream in some hydride generation systems. This is detrimental to the stability of response of the system and damaging to quartz cells. It is recommended that a membrane filter made of PTFE be inserted into the tubing connecting the gas/liquid separator to the absorption cell.

WARNING — Arsine (AsH_3) is passed into the absorption cell. This gas is very toxic, but it will normally be decomposed in the cell. However, in order to eliminate the possibility of exposure to arsine, it is essential that efficient local exhaust ventilation be installed to prevent waste gases from entering the general laboratory environment.

6.2.10 Analytical balance, capable of weighing to the nearest 0,1 mg.

6.2.11 Disposable gloves, impermeable, to avoid the possibility of contamination from the hands and to protect them from contact with toxic and corrosive substances. Poly(vinyl chloride) (PVC) gloves are suitable.

7 Sampling

7.1 Sampling procedure

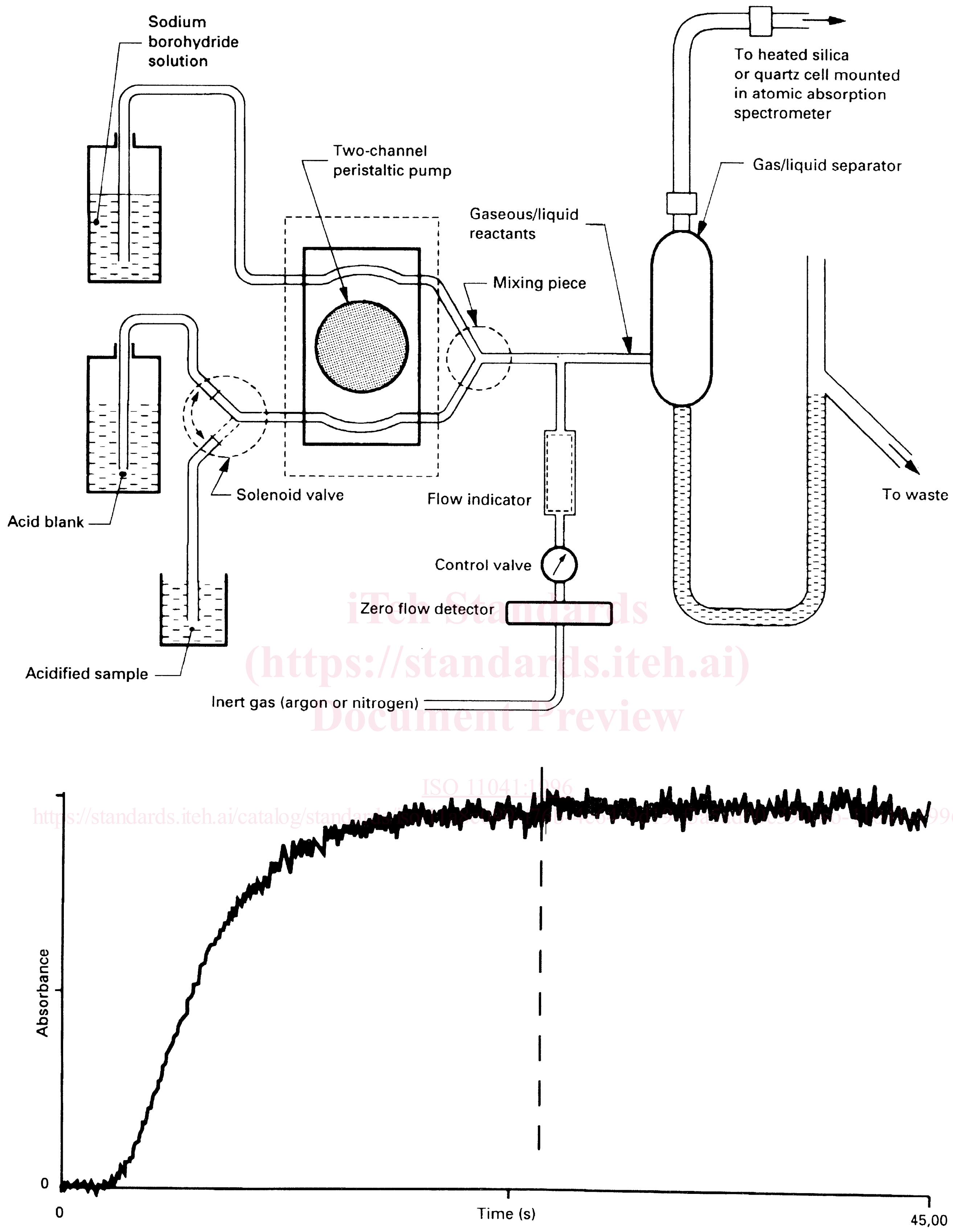
7.1.1 Collection characteristics and flow rate

Select a sampler (6.1.1) suitable for collection of the inhalable fraction of airborne particles, as defined in ISO 7708, and use at the flow rate at which the sampler exhibits the required collection characteristics.

NOTE 25 Inhalable samplers are typically used at a flow rate of around 2l/min (it is advisable to refer to the manufacturer's recommendations).

7.1.2 Sampling period

Select a sampling period of appropriate duration, using any available information about the work process and test atmosphere, so that the amount of arsenic collected is within the recommended working range of the method.



Atomic absorption output when valve is operated, changing the flow to the mixing piece from acid blank to acidified sample solution

Figure 1 — Schematic example of a configuration of a continuous-flow hydride generation system