BRITISH STANDARD

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Implementation of
ISO 11041:1996

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

ICS 13.040.30

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee EH/2, Air quality, to Subcommittee EH/2/2, Workplace atmospheres, upon which the following bodies were represented:

Asbestos Information Centre Limited
Asbestosis Research Council
BCIRA
British Ceramic Research Limited
British Gas plc
British Occupational Hygiene Society
Chemical Industries Association
Department of Trade and Industry (Laboratory of the Government Chemist)
Employers Association of Catering Equipment Engineers Limited
Engineering Equipment and Materials Users Association
Fibre Cement Manufacturers Association Limited
Health and Safety Executive
Institute of Energy
Institution of Occupational Hygienists
Institute of Occupational Medicine
London Transport
Royal Society of Chemistry

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Contents

National foreword

This British Standard reproduces verbatim ISO 11041:1996 and implements it as the UK national standard.

This British Standard is published under the direction of the Health and Environment Sector Board whose Technical Committee EH/2 has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international committee any enquiries on interpretation, or proposals for change, and keep UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

NOTE International and European Standards, as well as overseas standards, are available from Customer Services, BSI, 389 Chiswick High Road, London W4 4AL.

Textual errors

When adopting the text of the international standard, the textual errors listed below were discovered. They have been marked in the text and have been reported to ISO in a proposal to amend the text of the international standard.

1. **8.2.2** "cover with a watch" should read "cover with a watch glass".

2. **8.2.3** The word "NOTE" should precede "34 The solutions".

3. **8.4.1** The word "NOTE" should precede "38 The 193,7 nm arsenic line", and the text should read "but the use of the latter" and not "but its use", for clarity.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the ISO title page, pages ii to iv, page 1 to 18, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

INTERNATIONAL STANDARD

ISO 11041

First edition
1996-04-15

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)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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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.*

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.

[1)] To be published

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.

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

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

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 (Kl) 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.

NOTE 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.

NOTE 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.

NOTE 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.

NOTE 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.

NOTE 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.

NOTE 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.

NOTE 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.

NOTE 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.

NOTE 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.

NOTE 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.

NOTE 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.

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

NOTE 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.

NOTE 26 In order to estimate a sampling period of appropriate duration it is necessary to consider the flow rate used (see **7.1.1**) and the anticipated concentration of arsenic in the test atmosphere. When low arsenic-in-air concentrations are anticipated, the lower limit of the working range of the method (see **9.3.2**) should be taken into consideration. For example, to determine arsenic in air at a concentration of 0,1 $\mu g/m^3$, the minimum sampling time at a flow rate of 2 l/min is approximately 2 h. When high arsenic-in-air concentrations are anticipated, the sampling time should not be long enough to risk overloading the filter with particulate matter.

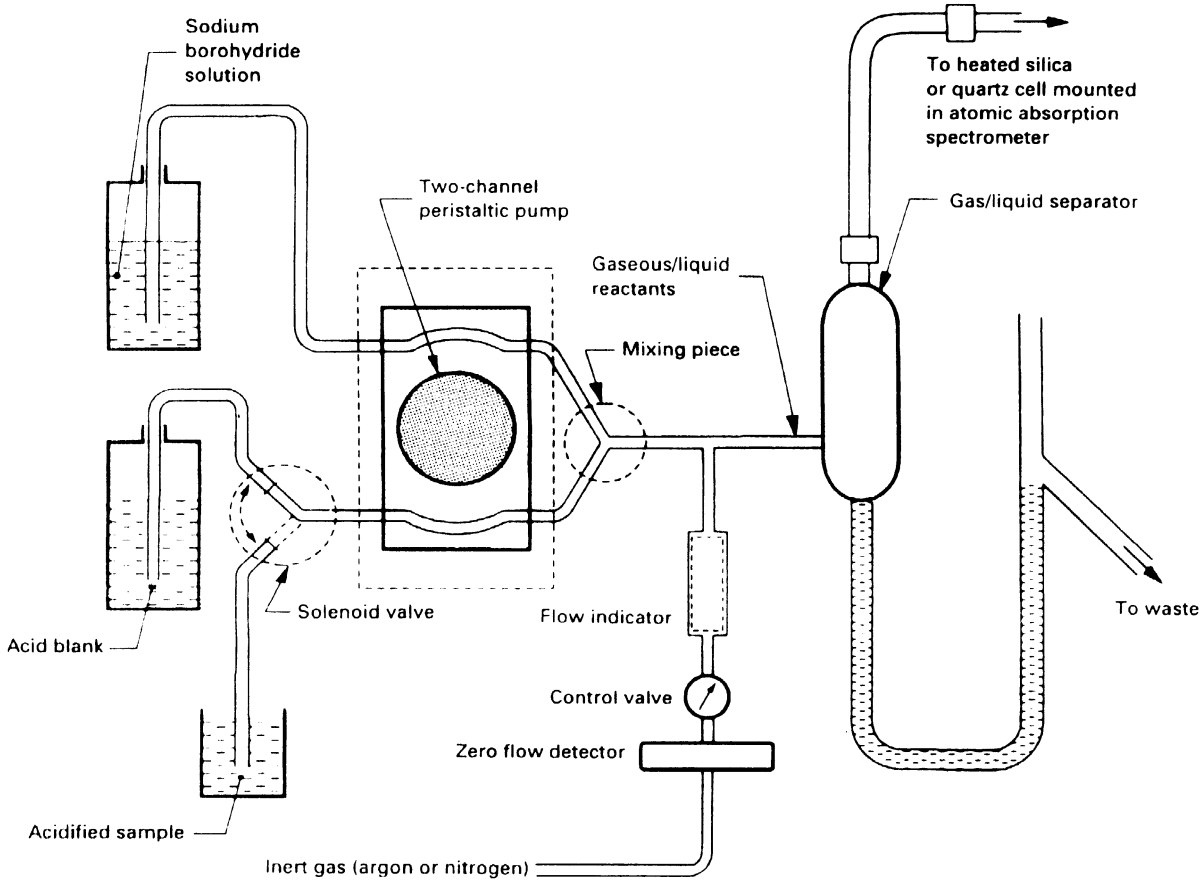
7.1.3 *Temperature and pressure effects*

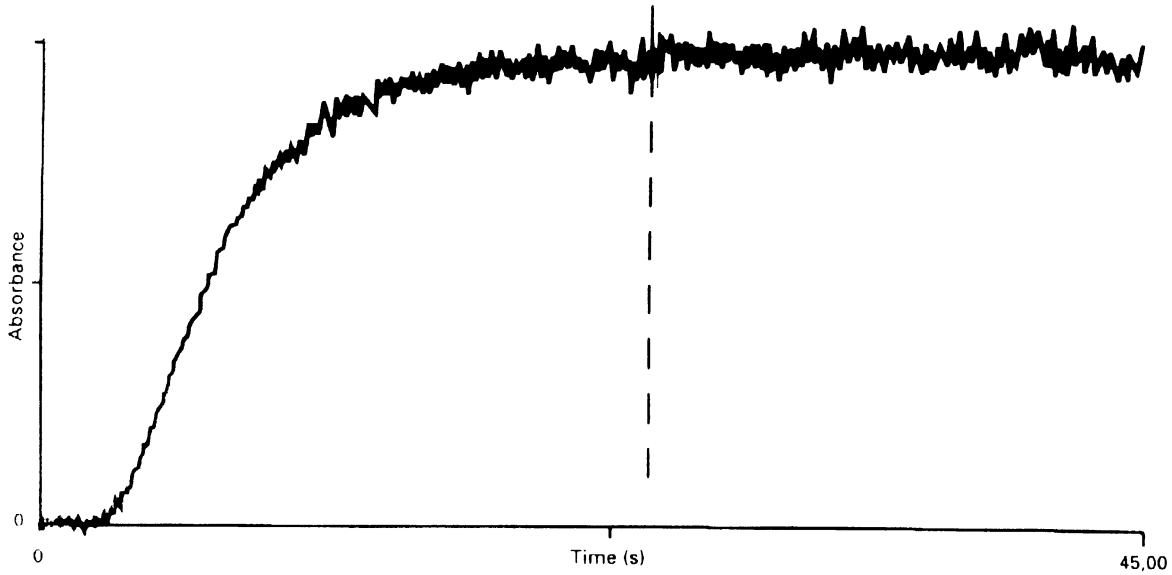
7.1.3.1 Consider whether it is necessary to recalculate the mass concentration of arsenic in the air to reference conditions of temperature and pressure in order to comply with national standards and regulations (see ISO 8756). If appropriate, measure and record the atmospheric temperature and pressure throughout the sampling period (see **7.3.2**, **7.3.3** and **7.3.5**) and use the equation given in **9.1.3** to apply the necessary correction.

NOTE 27 Arsenic-in-air concentrations are generally stated for the actual environmental conditions (temperature, pressure) at the workplace.

7.1.3.2 The indicated flow rate of certain types of flowmeter is dependent upon temperature and pressure. Therefore, refer to the manufacturer's directions for the particular flowmeter used, and consider whether it is necessary to make a correction to take into account any difference between the atmospheric temperature and pressure at the time of calibration of the flowmeter and at the time of sampling. Make such a correction if it is considered possible that an error of greater than ± 5 % will be introduced by not doing so. If a correction is to be made, measure and record the atmospheric temperature and pressure at which the flowmeter (**6.1.4**) was calibrated.

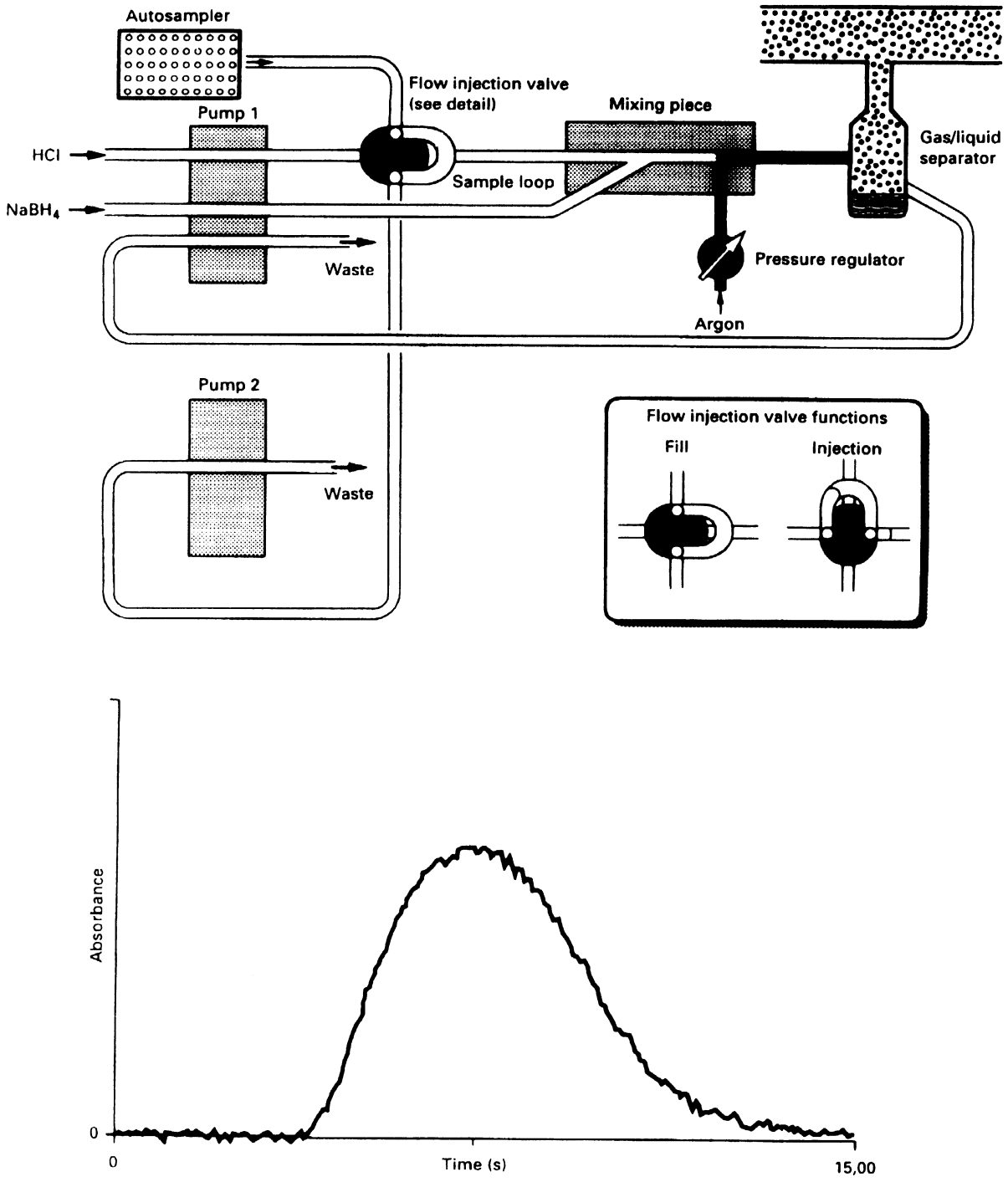
NOTE 28 An example of temperature and pressure correction for the indicated flow rate is given in **9.1.2**, for a flowmeter of variable area with constant pressure drop.





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



Atomic absorption output when sample valve is operated, injecting a fixed volume of acidified sample solution into the acid blank flow

Figure 2 — Schematic example of a configuration of a flow-injection-analysis hydride generation system

7.2 Preparation of sampling equipment

Perform the following in an area where arsenic contamination is known to be low.

7.2.1 Clean the samplers (**6.1.1**) before use. Disassemble the samplers, soak in laboratory detergent solution (**5.16**), rinse thoroughly with water (**5.1**), wipe with absorptive tissue and allow to dry before reassembly.

7.2.2 Load the back-up paper pads impregnated with sodium carbonate (**6.1.2.2**), followed by the cellulose ester membrane filters (**6.1.2.1**) into clean, dry samplers (**7.2.1**) so that the filter is upstream in relation to the back-up paper pad when air is drawn through the sampler. Handle the filters only with clean, flat-tipped forceps (**6.1.5**). Connect each loaded sampler to a sampling pump (**6.1.3**) using plastics tubing (**6.1.5**), ensuring that no leaks can occur. Switch on the sampling pump, attach the calibrated flowmeter (**6.1.4**) to the sampler so that it measures the flow through the sampler inlet orifice(s), and set the appropriate flow rate (see **7.1.1**) with an accuracy of ± 5 %. Switch off the sampling pump and seal the sampler with its protective cover or plug to prevent contamination with arsenic during transport to the sampling position.

NOTE 29 It might be necessary to warm up certain types of sampling pump (it is recommended to refer to the manufacturer's instructions).

7.3 Collection of samples

7.3.1 For personal sampling, fix the sampler to the lapel of the worker, in the breathing zone and as close to the mouth and nose as is reasonably practicable. Then, either place the sampling pump in a convenient pocket or attach it to the worker in a manner that causes minimum inconvenience, for example, to a belt (**6.1.5**) around the waist. For fixed-location sampling, position the sampler at the sampling site.

NOTE 30 The breathing zone has been defined in EN 1540 (reference [6] in annex A) as the space around the worker's face from where he takes his breath. For technical purposes, a more precise definition can be provided, as follows: hemisphere (generally accepted to be 0,3 m in radius) extending in front of the human face, centred on the midpoint of a line joining the ears; the base of the hemisphere is a plane through this line, the top of the head and the larynx.

7.3.2 When ready to begin sampling, remove the protective cover or plug from the sampler and switch on the sampling pump. Record the time at the start of the sampling period and, if the sampling pump has an elapsed time indicator, set this to zero. If appropriate (see **7.1.3.1**), measure the atmospheric temperature and pressure at the start of the sampling period using the thermometer (**6.1.6**) and barometer (**6.1.7**) and record the measured values.

7.3.3 Since it is possible for a filter to become clogged, monitor the performance of the sampler frequently, a minimum of once per hour. Measure the flow rate with an accuracy of ± 5 % using the calibrated flowmeter (**6.1.4**) and, if appropriate (see **7.1.3.1**), measure the atmospheric temperature using the thermometer (**6.1.6**) and the atmospheric pressure using the barometer (**6.1.7**). Record the measured values.

NOTE 31 Regular observation of the flow-fault indicator is an acceptable means of ensuring that the flow rate of a flow-stabilized sampling pump is maintained satisfactorily, provided that the flow-fault indicator indicates malfunction when the flow rate is outside ± 5 % of the nominal value.

7.3.4 Terminate sampling and consider the sample to be invalid if the flow rate is not maintained to within ± 5 % of the nominal value throughout the sampling period.

7.3.5 At the end of the sampling period (see **7.1.2**), measure the flow rate with an accuracy of ± 5 % using the calibrated flowmeter (**6.1.4**), switch off the sampling pump and record the flow rate and the time. Also observe the reading on the elapsed time indicator, if fitted, and consider the sample to be invalid if the reading on the elapsed time indicator and the timed interval between switching the sampling pump on and off do not agree to within ± 5 %, since this may suggest that the sampling pump has not been operating throughout the sampling period. Reseal the sampler with its protective cover or plug and disconnect it from the sampling pump. If appropriate (see **7.1.3.1**), measure the atmospheric temperature and pressure at the end of the sampling period using the thermometer (**6.1.6**) and barometer (**6.1.7**) and record the measured values.

7.3.6 Carefully record the sample identity and all relevant sampling data (see clause **11**). Calculate the mean flow rate by averaging the flow-rate measurements taken throughout the sampling period, and, if appropriate (see **7.1.3.1**), calculate the mean atmospheric temperature and pressure. Calculate the volume of air sampled, in litres, at atmospheric temperature and pressure, by multiplying the mean flow rate, in litres per minute, by the sampling time, in minutes.

7.3.7 With each batch of 10 samples, submit for analysis two unused cellulose ester membrane filters (**6.1.2.1**) and back-up paper pads impregnated with sodium carbonate (**6.1.2.2**) from the same lots as those used for sample collection. Subject these blank filters to exactly the same handling procedure as the samples, but do not draw air through them.

7.4 Transportation

Perform the following in an area where arsenic contamination is known to be low.

7.4.1 For samplers which collect the inhalable fraction of airborne particles on the filter (see note 10), remove the cellulose ester membrane filter and back-up paper pad from each sampler using clean flat-tipped forceps (**6.1.5**), place in a labelled filter-transport cassette (**6.1.5**) and close with a lid.

7.4.2 For samplers which have an internal filter cassette (see note 10), remove the filter cassette from each sampler, fasten with the transport clip supplied by the manufacturer, and label appropriately.

7.4.3 For samplers designed such that airborne particles which pass through the entry orifice(s) constitute the inhalable fraction, but which do not have an internal filter cassette (see note 10), and for samplers of the disposable cassette type, transport samples to the laboratory in the samplers in which they were collected.

7.4.4 Transport the filter-transport cassettes (see **7.4.1**), sampler filter cassettes (see **7.4.2**) or samplers (see **7.4.3**) in a container which has been designed to prevent damage to the samples in transit and which has been labelled to assure proper handling.

8 Procedure for analysis

8.1 Cleaning of glassware and polypropylene bottles

8.1.1 Before use, clean all glassware to remove any residual grease or chemicals, by soaking in laboratory detergent solution (**5.16**) and then rinsing thoroughly with water (**5.1**).

8.1.2 After initial cleaning (see **8.1.1**), clean all beakers used in the wet-ashing procedure specified in **8.2.2** with hot nitric acid. Fill to one-third capacity with concentrated nitric acid (**5.6**), cover with a watch glass (**6.2.1.2**), heat to approximately 150 °C on the hotplate (**6.2.5**) in a fume hood for 1 h, allow to cool, and then rinse thoroughly with water (**5.1**).

8.1.3 After initial cleaning (see **8.1.1**), clean all glassware other than beakers used in the wet-ashing procedure specified in **8.2.2**, by soaking in hydrochloric acid diluted 1 + 4 (**5.5**) for at least 24 h, and then rinsing thoroughly with water (**5.1**).

8.1.4 Thoroughly rinse glassware which has been previously subjected to the entire cleaning procedure specified in **8.1.1**, **8.1.2** and **8.1.3**, and which has been reserved for analysis of arsenic by this method, first with hydrochloric acid diluted 1 + 4 (**5.5**) and then with water (**5.1**).

8.1.5 Before use, clean the polypropylene bottles (**6.2.2**) by soaking in hydrochloric acid diluted 1 + 4 (**5.5**) for at least 24 h and then rinsing thoroughly with water (**5.1**).

8.2 Preparation of sample solutions and blank test solutions

8.2.1 Open the filter-transport cassettes (see **7.4.1**), sample filter cassettes (see **7.4.2**) or samplers (see **7.4.3**) and transfer each cellulose ester membrane filter and back-up paper pad into an individual clean, labelled 50 ml beaker (**6.2.1.1**) using clean, flat-tipped forceps (**6.1.5**). If the sampler used was of a type in which airborne particles deposited on the internal surfaces of the sampler filter cassette or sampler form part of the sample (see note 10), wash any particulate material adhering to the internal surfaces into the beaker using a minimum volume of water (**5.1**). Follow the same procedure for the blank filters (see **7.3.7**).

8.2.2 Add 5 ml of concentrated nitric acid (**5.6**) and 1 ml of concentrated sulfuric acid (**5.7**) to each beaker, [2]cover with a watch (**6.2.1.2**), and heat to approximately 175 °C (see note 32) on the hotplate (**6.2.5**) in a fume hood. When the initial vigorous reaction has subsided, slide back the watch glasses so that the beakers are only partially covered. Continue to heat each beaker until the solution volume has been reduced to approximately 1 ml and then remove from the hotplate. Allow the solutions to cool and then carefully add 2 ml of hydrogen peroxide solution (**5.8**) to each beaker. Replace the beakers on the hotplate, covering again with the watch glasses, and, when the initial vigorous reaction has subsided, slide back the watch glasses so that beakers are only partially covered. Continue to heat until dense white fumes of sulfur trioxide are evolved (raise the temperature of the hotplate to 200 °C, if necessary). If the solution becomes coloured due to charring of residual organic material, carefully add hydrogen peroxide solution, drop by drop, until a clear solution is obtained, and then evaporate again until dense white fumes appear. Remove the beakers from the hotplate and allow the solutions to cool.

NOTE 32 The exact temperature of the hotplate is not critical. A temperature of 175 °C is suggested because it is not high enough to evaporate the nitric acid and hydrogen peroxide at a rate at which there is insufficient time available for oxidation of organic matter, but it is high enough to evaporate all nitric acid and hydrogen peroxide to leave a fuming sulfuric acid solution.

NOTE 33 If glass-fibre or quartz-fibre filters impregnated with sodium carbonate are used (see note 15), it is necessary to use polytetrafluoroethylene (PTFE) laboratory apparatus and to add hydrofluoric acid to dissolve the filters before addition of concentrated nitric and sulfuric acids.

[2] See national foreword for details of textual errors.

DANGER — Concentrated hydrofluoric acid and hydrogen fluoride vapour are extremely toxic and intensely corrosive, and diluted hydrofluoric acid can also cause serious and extremely painful burns which may not be felt until up to 24 h after contact. Avoid exposure by contact with the skin or eyes, or by inhalation of the vapour. Use of personal protection (e.g. gloves, face shield or safety spectacles, etc.) is essential when working with concentrated or diluted hydrofluoric acid, and concentrated hydrofluoric acid must be used in a fume hood. It is essential that hydrofluoric acid antidote gel containing calcium gluconate is available to workers, both during and for 24 h after the use of hydrofluoric acid.

8.2.3 Carefully rinse the watch glass and the sides of each beaker with a small volume of water (**5.1**) and transfer each solution quantitatively to a 10 ml one-mark volumetric flask (**6.2.1.5**).

If necessary, remove any undissolved particulate material by filtering through a cellulose (paper) filter which has been prewashed with sulfuric acid diluted 1 + 9 (**5.10**) and then with water (**5.1**)

Finally, dilute to the mark with water (**5.1**), stopper and mix thoroughly.

NOTE 34 The solutions prepared in **8.2.3** can be stored for up to[3] one week before the sample solutions are prepared (see **8.2.4**) and analysed (see **8.5**).

8.2.4 Prepare blank test solutions and sample solutions for analysis. Transfer an aliquot, V_a ml (see note 35), of the solution (see **8.2.3**) and $(5 - V_a)$ ml of sulfuric acid diluted 1 + 9 (**5.10**) to a 25 ml one-mark volumetric flask (**6.2.1.5**). Add 12,5 ml of concentrated hydrochloric acid (**5.3**) and 2,5 ml of potassium iodide solution (**5.9**), dilute to the mark with water (**5.1**), stopper and mix thoroughly. Record the dilution factor, $25/V_a$, used for blank test solutions, F_0, and sample solutions, F_1. Allow to stand for 1 h for reduction of pentavalent arsenic to take place before analysis (see **4.3**).

NOTE 35 National occupational exposure limits for arsenic vary considerably, and therefore the volume of the aliquot of sample solution used may be varied up to a maximum of 5 ml, according to the detection limit required. For the lowest detection limit, an aliquot volume of 5 ml should be used; an aliquot volume of 1 ml is suggested when arsenic-in-air concentrations are to be compared with a limit value in the region of 0,01 mg of As per cubic metre; and, for limit values in the region of 0,1 mg of As per cubic metre, an aliquot volume of 0,1 ml is suggested.

NOTE 36 The test solution matrix is hydrochloric acid diluted 1 + 1, sulfuric acid diluted 1 + 49 and 10 g of Kl per litre. It is important that test and calibration solutions be matrix-matched, since hydride generation and atomic absorption spectrometric measurements of arsenic are affected by variations in sulfuric acid concentration.

8.3 Preparation of calibration solutions

Prepare at least six calibration solutions, including a zero-member calibration solution (see ISO 6955:1982, subclause **5.4.7**), to cover the range 0 ng of As per millilitre to 50 ng of As per millilitre when the 197,2 nm arsenic line is used or 0 ng of As per millilitre to 25 ng of As per millilitre when the 193,7 nm arsenic line is used (see **8.4.1**). Add approximately 50 ml of hydrochloric acid diluted 1 + 1 (**5.4**) to separate, labelled 100 ml one-mark volumetric flasks (**6.2.1.5**), add 2 ml of concentrated sulfuric acid (**5.7**) and then wash down the necks of the flasks with a little more hydrochloric acid diluted 1 + 1. Add 10 ml of concentrated hydrochloric acid (**5.3**), 10 ml of the potassium iodide solution (**5.9**), swirl to mix and allow to cool. Using a pipette (**6.2.1.3**), accurately add the appropriate volume of the arsenic working standard solution B (**5.13**) to each flask, dilute to the mark with hydrochloric acid diluted 1 + 1, stopper and mix thoroughly.

Prepare fresh calibration solutions daily.

NOTE 37 The range of the set of calibration solutions is given as a guide. The upper limit of the working range depends upon the performance characteristics of the hydride generation system (**6.2.8**) used and other instrumental factors which affect sensitivity and the linearity of the calibration. Accordingly, the range of the set of calibration solutions may be varied, but when making any changes it should be ensured that the response of the spectrometer over the alternative range of concentrations selected is such that it complies with the limitations on curvature indicated in note 45.

8.4 Calibration

8.4.1 *Selection of analytical line*

Select an analytical line for making absorbance measurements. The 197,2 nm arsenic line shall be used unless the highest sensitivity is required.

[4]NOTE 38 The 193,7 nm arsenic line is approximately twice as sensitive as the 197,2 nm arsenic line, but its use is preferable since the calibration obtained at this wavelength has a greater linear range.

8.4.2 *Setting up of the instrument*

8.4.2.1 If the hydride generation system (**6.2.8**) has not been used for some time, clean the gas/liquid separator and the silica or quartz absorption cell (**6.2.9**) before use, following the procedure specified in **8.7.3**.

[3] See national foreword for details of textual errors.

[4] See national foreword for details of textual errors.

8.4.2.2 Prepare the hydride generation system (**6.2.8**) for operation following the manufacturer's instructions. Fill the reservoir for reductant with sodium tetrahydroborate solution (**5.14**) and the reservoir for acid blank with hydrochloric acid diluted 1 + 1 (**5.4**).

NOTE 39 Optimum concentrations of reagents, liquid flow rates, purge-gas flow rate, etc., may vary somewhat according to the exact configuration of the system. This may also influence the magnitude of interference effects (see **10.3**).

NOTE 40 Hydride generation systems are sensitive to changes in temperature. Reagents and test solutions should therefore be allowed to equilibrate to room temperature before commencing analysis.

NOTE 41 Sodium tetrahydroborate solution gradually becomes saturated with hydrogen which may then degas when the solution is pumped. If bubbles of hydrogen reach the mixing piece, there will be a transient change in signal which will affect the analytical result, and this may not necessarily be evident for flow-injection systems. Bubble formation becomes more likely after the sodium tetrahydroborate solution has been standing for several hours, but it may be alleviated by degassing in an ultrasonic bath, or by continuously stirring the reservoir. It may also be helpful to insert a bubble trap in the tubing leading to the mixing piece. If bubble formation is still a problem, consider replicate analysis of the test solutions.

8.4.2.3 Install the silica or quartz absorption cell (**6.2.9**) in the optical path of the spectrometer following the manufacturer's instructions.

NOTE 42 The length of tubing connecting the absorption cell to the outlet of the gas/liquid separator of the hydride generation system (**6.2.8**) should be kept to a minimum.

8.4.2.4 Set-up the atomic absorption spectrometer (**6.2.7**) to make absorbance measurements at either the 197,2 nm or 193,7 nm arsenic line (see **8.4.1**), following the manufacturer's recommendations for specific parameters for the operation of instruments. If an air/acetylene flame is used to heat the silica or quartz absorption cell (**6.2.9**), use an oxidizing flame.

8.4.3 ***Presentation of solutions***

8.4.3.1 Inject the calibration solution, sample solution or blank test solution into the hydride generation system (**6.2.8**) by placing the sample uptake capillary in the solution concerned. This may be carried out manually or using an autosampler.

8.4.3.2 For continuous-flow hydride generation systems (**6.2.8.1**), the configuration is such that initially the acid blank solution is pumped to the mixing piece. When ready to analyse the sample solution, the configuration is changed so that the test solution is pumped to the mixing piece.

8.4.3.3 For flow-injection-analysis hydride generation systems (**6.2.8.2**), the acid blank solution is continuously pumped to the mixing piece and a precise volume of the test solution is injected into the acid blank stream for analysis.

8.4.4 ***Conditioning the hydride generation system***

Condition the system before use in order to ensure that a stable signal is obtained before proceeding to carry out a calibration.

8.4.4.1 Place the reductant, acid blank and sample-uptake capillaries in a container of water (**5.1**), and allow the pump(s) to operate for 5 min for the flow rates to stabilize. Fill a 10 ml measuring cylinder (**6.2.1.4**) to a convenient mark with water (**5.1**), and determine each flow rate in turn by placing the appropriate uptake capillary in the measuring cylinder of water and observing the volume of water pumped out in 1 min. Verify that the flow rates are within the nominal specification recommended by the manufacturer of the hydride generation system (**6.2.8**) and adjust the pressure exerted on the peristaltic pump tubing by the pump head, and/or install new pump tubing as necessary. Replace the uptake capillaries for the reductant and acid blank in the appropriate reservoirs.

8.4.4.2 Depending on the type of hydride generation system used, proceed according to **8.4.4.2.1** or **8.4.4.2.2**.

8.4.4.2.1 For continuous-flow hydride generation systems (**6.2.8.1**), alternately pump acid blank (**5.4**) and the most concentrated calibration solution (see **8.3**) to the mixing piece of the continuous-flow hydride generation system, and repeat absorbance measurements with a suitably short integration period. Continue this sequence until a repeatable analytical response is obtained, and record the parameters necessary for operation of the continuous-flow hydride generation system used. In particular, record the stabilization delay time, which is the time taken for the analytical response to reach a stable value when a solution is presented to the system, and the baseline delay time, which is the time taken for the signal response to return to the baseline when the acid blank is pumped again. Record both of these delay times. If the continuous-flow hydride generation system is interfaced to the atomic absorption spectrometer for automatic operation, set the necessary parameter(s) to the appropriate value(s).

8.4.4.2.2 For flow-injection-analysis hydride generation systems (**6.2.8.2**), fill the sample loop by pumping the most concentrated calibration solution (see **8.3**) through it, and then inject this solution into the acid blank stream. Note the delay time before an atomic absorbance peak is obtained. Optimize integration or peak height measurement parameters, and then repeat injections until a repeatable analytical response is obtained.

NOTE 43 If a repeatable analytical response is not obtained, this is likely to be due to contamination of the system. In this case, further operations should be suspended, and the gas/liquid separator and the silica or quartz absorption cell (**6.2.9**) should be cleaned following the procedure specified in **8.7.3**.

8.4.5 *Determination of reagent blank*

8.4.5.1 Place the reductant, acid blank and sample-uptake capillaries in a container of water (**5.1**) and, after allowing sufficient time for flushing out the system, adjust the spectrometer zero.

8.4.5.2 Replace the uptake capillaries for reductant and acid blank in the appropriate reservoirs. Pump acid blank (**5.4**) and reductant to the mixing piece and measure the absorbance after allowing sufficient time for the water to be replaced.

NOTE 44 If the reagent blank is higher than normal, the analytical performance of the system will be degraded, and in particular the detection limit will be poorer. A high blank may be due to contamination of one or more of the reagents or to contamination of the system. If it is considered that contamination of the reagents is the likely cause of the high blank, new sodium tetrahydroborate solution and hydrochloric acid diluted 1 + 1 (**5.4**) should be prepared. If it is considered that contamination of the system is the likely cause of the high blank, the gas/liquid separator and the silica or quartz absorption cell (**6.2.9**) should be cleaned following the procedure specified in **8.7.3**. The reagent blank should then be redetermined by repeating the procedure specified in **8.4.5.1** and **8.4.5.2**.

8.4.6 *Spectrometric measurements*

8.4.6.1 Adjust the spectrometer zero while pumping acid blank (**5.4**) to the mixing piece of the hydride generation system (**6.2.8**).

8.4.6.2 Depending on the type of hydride generation system used, proceed according to **8.4.6.2.1** or **8.4.6.2.2**.

8.4.6.2.1 For continuous-flow hydride generation systems (**6.2.8.1**), pump each calibration solution (see **8.3**) in turn through the sample-uptake tubing to the mixing piece, and measure its absorbance after the determined stabilization delay time (see **8.4.4.2.1**). Pump acid blank (**5.4**) to the mixing piece, in between each calibration solution, and wait for the determined baseline delay time (see **8.4.4.2.1**) before proceeding to measure the next calibration solution.

8.4.6.2.2 For flow-injection-analysis hydride generation systems (**6.2.8.2**), inject each calibration solution (see **8.3**) in turn into the blank acid stream and measure the peak height or peak area of the atomic absorption signal.

8.4.7 *Calibration function*

For instruments controlled by a microprocessor or personal computer, use a suitable algorithm to generate the calibration function. For instruments without this capability, prepare a calibration graph by plotting the absorbance of the calibration solutions versus the concentration of arsenic, in nanograms per millilitre, in the respective solutions.

NOTE 45 In general it is best to work in the linear range of an atomic absorption calibration, where absorbance is proportional to the concentration of arsenic in solution. However, a certain amount of curvature can be tolerated, but, ideally the slope of the top 20 % of the calibration curve should be not less than 70 % of the slope of the bottom 20 % calculated in the same manner. However, hydride generation atomic absorption calibrations are more curved than flame atomic absorption calibrations, and discretion should be exercised in assessing whether recalibration over a lower concentration range is necessary.

8.5 Determination

8.5.1 Depending on the type of hydride generation system used, proceed according to **8.5.1.1** or **8.5.1.2**.

8.5.1.1 For continuous-flow hydride generation systems (**6.2.8.1**), adjust the spectrometer zero whilst pumping the acid blank (**5.4**) to the mixing piece of the system. Pump each sample solution and blank test solution (see **8.2.4**) in turn through the sample-uptake tubing to the mixing piece, and make absorbance measurements after the determined stabilization delay time (see **8.4.4.2.1**). Pump acid blank (**5.4**) to the mixing piece, in between each sample solution, and wait for the determined baseline delay time (see **8.4.4.2.1**) before proceeding to measure the next sample solution.

8.5.1.2 For flow-injection-analysis hydride generation systems (**6.2.8.2**), pump each sample solution and blank test solution (see **8.2.4**) sequentially through the sample valve, inject into the acid blank stream, and measure the peak height or peak area of the atomic absorption signal.

8.5.2 If a significant baseline drift is observed whilst pumping acid blank, readjust the spectrometer zero.

8.5.3 For instruments controlled by a microprocessor or personal computer, use the calibration function (see **8.4.7**) to calculate the concentration of arsenic in the sample solution and blank test solution, and obtain a direct read-out of the results in concentration units. For instruments without this possibility, determine the concentration of arsenic in the sample solution and blank test solution (see **8.2.4**) from the calibration graph (see **8.4.7**).

8.5.4 Analyse a mid-range calibration solution after each five to ten sample solutions and make an absorbance measurement. If this indicates that the sensitivity has changed by more than ± 5 %, take one of the following corrective measures: either use the available software facilities of the microprocessor or personal computer to correct for the sensitivity change (reslope facility); or suspend analysis and recalibrate the spectrometer as specified in **8.4.6** and **8.4.7**. In both cases, reanalyse the sample solutions which were analysed during the period in which the sensitivity change occurred.

8.5.5 If high concentrations of arsenic are found, repeat the analysis on a new test solution (see **8.2.4**) prepared using a smaller aliquot of the sample solution, to bring the concentration within the range of the calibration. Alternatively, if using the 193,7 nm arsenic line, change to the less sensitive 197,2 nm arsenic line.

8.5.6 Calculate the mean arsenic concentration in the blank test solutions.

8.6 Estimation of the instrumental detection limit

8.6.1 Estimate the instrumental detection limit (see note 47) under the working analytical conditions, following the procedure specified in **8.6.2** and **8.6.3**, and repeat this exercise whenever these conditions are changed.

8.6.2 Prepare a reference solution at a concentration of 0,25 ng of As per millilitre by diluting arsenic working standard solution B (**5.13**). Do this by following a procedure similar to that used for preparation of calibration solutions (see **8.3**).

8.6.3 Make at least 20 absorbance measurements on the reference solution (see **8.6.2**) and calculate the instrumental detection limit using standard statistical methods.

NOTE 46 The limit of detection of an atomic absorption method is defined in ISO 6955:1982, subclause **6.2.3**, as the concentration of analyte for which the absorbance has a value equal to k times that of the standard deviation of a series of readings measured on a solution, the concentration of which is distinctly detectable above, but close to, that of the blank (k is generally taken as either 2 or 3). For the purposes of this International Standard, k should be taken as 3.

NOTE 47 The limit of detection calculated from results obtained using the procedure specified in **8.6.2** and **8.6.3** is an instrumental detection limit. This is of use in identifying changes in instrument performance, but it is not a detection limit of the method, and is likely to be unrealistically low because it only takes into account the variability between instrumental readings. Determinations made on one solution do not take into consideration variability from the matrix or sample variability. A more realistic detection limit for the analytical procedure specified in this International Standard may be obtained by making measurements on at least 10 blank test solutions, i.e. solutions of blanks (see **7.3.7**). The standard deviation of such measurements, made over a longer time interval than between successive calibrations, may be used to obtain an estimate of the detection limit of the method.

8.7 Close-down procedure

8.7.1 Switch off the flame or electricity supply to the heating coil of the silica or quartz absorption cell (**6.2.9**).

8.7.2 Place the reductant, acid blank and sample-uptake capillaries in a container of water (**5.1**), and allow the pump to operate for 5 min to flush out the system.

8.7.3 Disconnect the tube connecting the gas/liquid separator of the hydride generation system (**6.2.8**) and the silica or quartz absorption cell (**6.2.9**). Disconnect other inlets and outlets to the gas/liquid separator, and remove it and the absorption cell from their mountings. Soak the gas/liquid separator, the absorption cell and the tube which connects the two pieces of apparatus in the sodium hydroxide solution (**5.15**) for at least 30 min. Remove them from the solution, wash thoroughly with hydrochloric acid diluted 1 + 1 (**5.4**), and rinse them thoroughly with water (**5.1**). Reassemble the system.

8.7.4 Follow any other aspects of the close-down procedure recommended by the manufacturer.

9 Expression of results

9.1 Calculations

9.1.1 Calculate the mass concentration of arsenic in the air sample, ρ_{As}, in milligrams per cubic metre, at ambient conditions, using the equation

$$\rho_{As} = \frac{\left(\rho_{As,1} \cdot V_1 \cdot F_1\right) - \left(\rho_{As,0} \cdot V_0 \cdot F_0\right)}{1\,000\,V}$$

where

$\rho_{As,\,0}$ is the mean concentration of arsenic, in nanograms per millilitre, in the blank test solutions (see **8.5.6**);

$\rho_{As,\,1}$ is the concentration of arsenic, in nanograms per millilitre, in the sample solution (see **8.5.3**);

V is the volume, in litres, of the air sample (see **9.1.2**);

V_0 is the volume, in millilitres, of the blank test solution, i.e. 10 ml (see **8.2.3**);

V_1 is the volume, in millilitres, of the sample solution, i.e. 10 ml (see **8.2.3**);

F_0 is the dilution factor used in the preparation of the blank test solution (see **8.2.4**);

F_1 is the dilution factor used in the preparation of the sample solution (see **8.2.4**);

1 000 is the factor used to convert the result to milligrams per cubic metre.

9.1.2 In some instances, it is necessary to apply a temperature and pressure correction for the indicated sampling flow rate (see **7.1.3.2**). A typical example of when such a correction is necessary is when the sampling pump used incorporates a flowmeter of variable area with constant pressure drop, which was calibrated and used to measure the flow rate in **7.3.2**, **7.3.3** and **7.3.5**. In this instance, use the following equation to calculate the volume of air samples

$$V_{corr} = q_V \cdot t \sqrt{\frac{p_1 \cdot T_2}{p_2 \cdot T_1}}$$

where

V_{corr} is the corrected volume, in litres;

q_V is the mean flow rate, in litres per minute;

t is the sampling time, in minutes;

p_1 is the atmospheric pressure, in kilopascals, during calibration of the sampling pump flowmeter;

p_2 is the mean atmospheric pressure, in kilopascals, during the sampling period;

T_1 is the temperature, in kelvins, during calibration of the sampling pump flowmeter;

T_2 is the mean temperature, in kelvins, during the sampling period.

Any other flowmeter may also require correction for variation in temperature and pressure. Follow the manufacturer's instructions for such corrections.

9.1.3 If appropriate (see **7.1.3.1**), calculate the mass concentration of arsenic in the air sample at reference temperature and pressure (273 K and 101,3 kPa, respectively) $\rho_{As,\,corr}$, using the equation

$$\rho_{As,\,corr} = \rho_{As} \times \frac{101{,}3\,T_2}{p_2 \times 273}$$

where

ρ_{As} is the mass concentration of arsenic in the air sample, in milligrams per cubic metre, at ambient conditions, as calculated in **9.1.1**;

T_2 is the mean temperature, in kelvins, during the sampling period;

p_2 is the mean atmospheric pressure, in kilopascals, during the sampling period;

273 is the reference temperature, in kelvins;

101,3 is the standard atmospheric pressure, in kilopascals.

9.2 Performance of the method

9.2.1 Laboratory experiments indicate that the analytical method does not exhibit significant bias. The mean analytical recovery for dosed filters in the range 5 µg to 100 µg of arsenic was determined (see reference [5] in annex A) to be 100,7 % using continuous-flow hydride generation and atomic absorption spectrometry and 99,3 % using flow-injection-analysis hydride generation and atomic absorption spectrometry, for a sample solution aliquot of 0,1 ml (see **8.2.4**). The mean analytical recovery for dosed filters in the range 0,5 µg to 10 µg of arsenic was determined to be 98,8 % using continuous-flow hydride generation and atomic absorption spectrometry and 102,7 % using flow-injection-analysis hydride generation and atomic absorption spectrometry, for a sample solution aliquot of 1 ml (see **8.2.4**).

NOTE 48 If the wet-ashing procedure specified in **8.2.2** is ineffective for the dissolution of particulate arsenic compounds present in the test atmosphere, and an alternative more vigorous dissolution procedure has not been used (see **10.2**), results will be subject to a significant negative bias.

9.2.2 The component of the coefficient of variation of the method that arises from analytical variability, $CV_{analysis}$, is dependent upon a number of factors, including the volume of the test solution aliquot used in the preparation of the sample solution (see **8.2.4**) and whether continuous-flow or flow-injection-analysis hydride generation and atomic absorption spectrometry is used (see note 2). The coefficient of variation $CV_{analysis}$ is at a minimum when the concentration of arsenic in the sample solution is in the mid-range of the calibration and, in laboratory experiments, it has been estimated (see reference [5] in annex A) to be about 1 % using continuous-flow hydride generation and atomic absorption spectrometry and about 3 % using flow-injection-analysis hydride generation and atomic absorption spectrometry, for measurements made at 197,2 nm on sample solutions with an arsenic concentration in the range 10 ng/ml to 40 ng/ml. This gives a measure of the repeatability of the analytical method.

NOTE 49 The repeatability of an atomic absorption method at a given level is defined in ISO 6955:1982, subclause **6.2.7** as the closeness of agreement between successive results obtained using the same method on identical material submitted for the test under the same conditions (same operator, same equipment, same set of reagents, same laboratory).

9.2.3 The overall uncertainty of the method (see note 51), as defined in EN 482, has been determined (see reference [5] in annex A) to be within the specifications prescribed in EN 482 for the overall uncertainty of measurements for comparison with limit values (see note 52).

This assumes that the coefficient of variation of the method that arises from inter-specimen sampler variability, CV_{inter}, is negligible and that the coefficient of variation of the method that arises from flow-rate variability, CV_{flow}, is limited to 5 % (see **7.3.4**).

NOTE 50 Overall uncertainty (of a measuring procedure or of an instrument) has been defined in EN 482 as the quantity used to characterize as a whole the uncertainty of a result given by an apparatus or measuring procedure. It is expressed, on a relative basis, by a combination of bias and precision, usually according to the formula

$$\frac{|\bar{x} - x_{ref}| + 2s}{x_{ref}} \times 100$$

where

$\bar{x}$ is the mean value of results of a number n of repeated measurements;

x_{ref} is the true or accepted reference value of concentration;

s is the standard deviation of measurements.

NOTE 51 CEN (see EN 482) has described general performance requirements for the performance of procedures for the measurement of chemical agents in workplace atmospheres. Upper limits of acceptability for relative overall uncertainty have been specified for a number of measurement tasks, and these may be used as a guide for the purposes of this International Standard. CEN requirements are less stringent for screening measurements than for measurements for comparison with limit values; and they are less stringent for measurements for comparison with limit values when these are made in the range 0,1 to 0,5 times the exposure limit value (overall uncertainty less than 50 %) than when they are made in the range 0,5 to 2,0 times the exposure limit value (overall uncertainty less than 30 %).

9.2.4 The component of the coefficient of variation of the method that arises from analytical variability, $CV_{analysis}$, has also been determined (see reference [5] in annex A) in an interlaboratory exercise, in order to obtain a measure of the reproducibility of the analytical method. For measurements made at 197,2 nm using continuous-flow hydride generation and atomic absorption spectrometry, $CV_{analysis}$ was found to be 3,2 % for filters spiked with 5 µg of arsenic, using a sample solution aliquot of 1 ml (see **8.2.4**), and 3,5 % for filters spiked with 50 µg of arsenic, using a sample solution aliquot of 0,1 ml (see **8.2.4**).

NOTE 52 The reproducibility of an atomic absorption method at a given level is defined in ISO 6955:1982, subclause **6.2.8** as the closeness of agreement between individual results obtained using the same method on identical material submitted for the test but under different conditions (different operators, different equipment, different laboratories, different times).

9.2.5 The sampling efficiency of a similar method has been determined to be 1,00 for laboratory-generated arsenic aerosols and 0,98 for arsenic trioxide vapour (see reference [3] in annex A). Field trials indicate that the sampling procedure described in this International Standard has an equivalent performance.

9.3 Detection limit and working range

9.3.1 Detection limits for the determination of arsenic are dependent upon the analytical line at which absorbance measurements are made (see **8.4.1**) and upon the hydride generation system (**6.2.8**) and atomic absorption spectrometer (**6.2.7**) used. However, the qualitative and quantitative instrumental detection limits for arsenic, defined as three times and ten times the standard deviation of absorbance measurements made as described in **8.6**, have been estimated (see reference [5] in annex A) to be approximately 0,3 ng/ml and 1 ng/ml for the 197,2 nm arsenic line. For an air sample volume of 960 litres and a sample solution aliquot of 5 ml (see **8.2.4**), this corresponds to arsenic-in-air concentrations of 0,015 µg/m^3 and 0,05 µg/m^3.

9.3.2 The working range of the method is approximately 100 ng to 125 µg of arsenic per sample for absorbance measurements made at the 197,2 nm arsenic line on sample solutions prepared using test solution aliquots in the range 5 ml to 0,1 ml (see **8.2.4**).

10 Special cases

10.1 Certain metal arsenides (e.g. alkali metal, aluminium, calcium and zinc arsenides) decompose in the presence of water or in acid solution to liberate arsine, at or before the wet-ashing stage (see **8.2.2**). This method will therefore not give accurate arsenic results for particulates containing such compounds.

10.2 If there is any doubt about the suitability of the wet-ashing procedure specified in **8.2.2** for the dissolution of particulate arsenic compounds which may be present in the test atmosphere, determine its effectiveness by analysing a bulk sample of known arsenic content, which is representative of the material in the test atmosphere. If the efficiency of recovery is less than 95 %, use an alternative, more vigorous dissolution procedure which is not specified in this International Standard. Do not use a correction factor to compensate for an apparently ineffective dissolution procedure, since this may also lead to erroneous results.

NOTE 53 In designing an experiment to determine the effectiveness of the wet-ashing procedure, it should be recognized that the particle size distribution of a bulk sample may have an important influence on the efficiency of its dissolution. Also, amounts in micrograms of relatively insoluble material are frequently much more readily soluble than bulk amounts.

10.3 A number of transition metals, mainly those of Groups VIII and IB, cause signal depression in the determination of arsenic by hydride-generation atomic absorption spectrometry. This interference has been shown to be associated with the reduction of metal ions to the free metal. The resulting finely dispersed metal precipitate captures and decomposes the hydride formed in a secondary reaction. The metal may be slow to redissolve, in which case low results may be obtained when subsequent sample solutions are analysed. If high levels of interfering elements are present, it is advisable to analyse calibration solutions between sample solutions, to check that signal depression is not occurring. It has been shown that this type of interference is minimized by using a high concentration of hydrochloric acid and a low concentration of sodium tetrahydroborate solution. The most severe interferences are caused by nickel, copper and cobalt, but, with the reagent concentrations used in this method, the signal depression caused by 10 µg/ml of these three metals is less than 10 %, for a solution containing 10 ng of As per millilitre. Flow-injection-analysis hydride generation systems (**6.2.8.2**) are much less affected by metal interferences than continuous-flow hydride generation systems (**6.2.8.1**).

10.4 The method specified in this International Standard may be applicable, with modifications, to the determination of other elements which form volatile hydrides, for example, antimony, selenium and tellurium. However, it is essential that the element is in the appropriate oxidation state, for example, Sb(III), Se(IV) and Te(IV), and an alternative to potassium iodide may be required to achieve the necessary reduction when sample solutions are prepared (see **8.2.4**). It is necessary for the user to determine how the method shall be modified, to confirm that the element of interest is not lost through volatilization in the wet-ashing procedure specified in **8.2.2**, and that matrix interferences are not a problem.

11 Test report

The test report shall include the following information:

a) all details necessary for a complete identification of the air sample, including the date of sampling, the place of sampling, the type of sample (personal or fixed location), and either the identity of the individual whose breathing zone was sampled (for a personal sample) or the location at which the general occupational environment was sampled (for a fixed-location sample);

b) a reference to this International Standard and to the atomic absorption spectrometric method used (continuous-flow hydride generation or flow-injection-analysis hydride generation);

c) the type and diameter of cellulose ester membrane filter and back-up paper pad used;

d) the type of sampler used;

e) the type of sampling pump used, and its identification;

f) the type of flowmeter used, the primary standard against which it was calibrated, the range of flow rates for which the flowmeter was calibrated, and the atmospheric temperature and pressure at which it was calibrated, if appropriate (see **7.1.3.2**);

g) the time at the start and end of the sampling period, and the duration of the sampling period, in minutes;

h) the flow rate at the start and end of the sampling period, and the mean flow rate, in litres per minute;

i) the mean atmospheric temperature and pressure during sampling, if appropriate (see **7.1.3.1**);

j) the volume of air sampled, in litres, at ambient conditions;

k) the name of the person who collected the sample;

l) the mass concentration of arsenic in the air sample, in milligrams per cubic metre, at ambient conditions and, if appropriate (see **7.1.3.1**), adjusted, as required for compliance with national standards and regulations, to reference conditions, e.g. 20 °C or 25 °C;

m) the analytical variables used in **9.1** to calculate the mass concentration of arsenic in the air sample, including the arsenic concentration in the sample solution and blank test solution, the volume of the sample solution and blank test solution, and the dilution factor;

n) any interferents known to be present;

o) the estimated detection limit under the working analytical conditions (see **8.6.3**);

p) any operation not specified in this International Standard, or regarded as optional;

q) the name of the analyst;

r) the date of the analysis.

Annex A (informative)
Bibliography

[1] WORLD HEALTH ORGANIZATION/INTERNATIONAL AGENCY FOR RESEARCH ON CANCER. *IARC Monographs on the Evaluation of the Carcenogenic Risk of Chemicals to Humans,* Volume 23 — Some Metals and Metallic Compounds, IARC, Lyon, France (1980).

[2] U.S. NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH. *NIOSH Manual of Analytical Methods,* 4th Edition: Method 7901 — Arsenic trioxide (as As). U.S. Department of Health and Human Services, (NIOSH) Publication 94-113 (1994).

[3] COSTELLO, R.J., ELLER, P.M., and DELON HULL, R., Measurement of multiple inorganic arsenic species, *Am. Ind. Hyg. Assoc. J.* **44** (1), pp. 21-28 (1983).

[4] DEMANGE, M., VIEN, I., HECHT, G., and HERY, M., Mise au point d'une méthode de prélèvement du trioxyde de diarsenic, *Cahiers de notes documentaires* no 146, 1st trimestre. Institut national de recherche et de sécurité, Vanduvre-lès-Nancy, France (1992).

[5] FOSTER, R.D., and HOWE, A.M., *A revised procedure for the determination of arsenic and inorganic compounds of arsenic in air (excluding arsine) using hydride generation and atomic absorption spectometry.* IR/L/IS/93/05, HSE, Sheffield, United Kingdom (1993).

[6] EN 1540:—, *Workplace atmospheres — Terminology*[5)].

[5)] To be Published.

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