



NEW WORK ITEM PROPOSAL	
Date of presentation 2009-08-13	Reference number (to be given by the Secretariat)
Proposer BSI	ISO/TC 147 / SC 2 N 1069 ISO/TC 147/SC 2/WG 52 N 0012
Secretariat DIN	

A proposal for a new work item within the scope of an existing committee shall be submitted to the secretariat of that committee with a copy to the Central Secretariat and, in the case of a subcommittee, a copy to the secretariat of the parent technical committee. Proposals not within the scope of an existing committee shall be submitted to the secretariat of the ISO Technical Management Board.

The proposer of a new work item may be a member body of ISO, the secretariat itself, another technical committee or subcommittee, or organization in liaison, the Technical Management Board or one of the advisory groups, or the Secretary-General.

The proposal will be circulated to the P-members of the technical committee or subcommittee for voting, and to the O-members for information.

See overleaf for guidance on when to use this form.

IMPORTANT NOTE: Proposals without adequate justification risk rejection or referral to originator.

Guidelines for proposing and justifying a new work item are given overleaf.

Proposal (to be completed by the proposer)

Title of proposal (in the case of an amendment, revision or a new part of an existing document, show the reference number and current title)	
English title	ISO 17378-2 "Water quality — Determination of arsenic — Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)"
French title (if available)	ISO 17378-2 "Qualité de l'eau — Dosage de l'arsenic — Partie 2: Méthode par spectrométrie d'absorption atomique à production d'hydrure (PA-SAA))"
Scope of proposed project This part of ISO 17378 specifies a method for the determination of arsenic. The method is applicable to drinking water, surface water, ground water and rain water. The linear application range of this standard is from 0,5 µg/l to 20 µg/l. Samples containing arsenic at higher concentrations than the application range can be analysed following appropriate dilution [1]. The method is unlikely to detect organo arsenic compounds. The sensitivity of this method is dependent on the selected operating conditions. It is important to use high purity reagents in all cases with minimum levels of arsenic. The concentration of the blank solution shall be less than the lower level of interest..	
Concerns known patented items (see ISO/IEC Directives Part 1 for important guidance) <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No If "Yes", provide full information as annex	
Envisaged publication type (indicate one of the following, if possible) <input checked="" type="checkbox"/> International Standard <input type="checkbox"/> Technical Specification <input type="checkbox"/> Publicly Available Specification <input type="checkbox"/> Technical Report	
Purpose and justification (attach a separate page as annex, if necessary) To determine trace levels of arsenic in water as required to meet requirements of new European legislation and in accordance with The European Union Water Framework Directive.	
Target date for availability (date by which publication is considered to be necessary)	
Proposed development track <input type="checkbox"/> 1 (24 months) <input type="checkbox"/> 2 (36 months - default) <input checked="" type="checkbox"/> 3 (48 months)	
Relevant documents to be considered	
Relationship of project to activities of other international bodies	
Liaison organizations	Need for coordination with: <input type="checkbox"/> IEC <input type="checkbox"/> CEN <input type="checkbox"/> Other (please specify)

Preparatory work (at a minimum an outline should be included with the proposal) <input checked="" type="checkbox"/> A draft is attached <input type="checkbox"/> An outline is attached. It is possible to supply a draft by The proposer or the proposer's organization is prepared to undertake the preparatory work required <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No		
Proposed Project Leader (name and address) Prof Peter B Stockwell P S Analytical Ltd Arthur House, Crayfields Industrial Park Main Road, St Pauls Cray Orpington, Kent BR5 3HP, UK E-Mail: pbs@psanalytical.com	Name and signature of the Proposer (include contact information) Prof Peter B Stockwell	
Comments of the TC or SC Secretariat Supplementary information relating to the proposal <input type="checkbox"/> This proposal relates to a new ISO document; <input type="checkbox"/> This proposal relates to the amendment/revision of an existing ISO document; <input type="checkbox"/> This proposal relates to the adoption as an active project of an item currently registered as a Preliminary Work Item; <input checked="" type="checkbox"/> This proposal relates to the re-establishment of a cancelled project as an active project. Other:		
Voting information The ballot associated with this proposal comprises a vote on: <input type="checkbox"/> Adoption of the proposal as a new project <input checked="" type="checkbox"/> Adoption of the associated draft as a committee draft (CD) (see ISO Form 5, question 2.3.1) <input type="checkbox"/> Adoption of the associated draft for submission for the enquiry vote (DIS or equivalent) (see ISO Form 5, question 2.3.2) Other:		
Annex(es) are included with this proposal (give details) <input checked="" type="checkbox"/> ISO/WD 17378-2 and Revised Secretariat Observations on former ISO/DIS		
Date of circulation 2009-08-28	Closing date for voting 2009-12-01	Signature of the TC or SC Secretary G. Barz

Use this form to propose:

- a) a new ISO document (including a new part to an existing document), or the amendment/revision of an existing ISO document;
 b) the establishment as an active project of a preliminary work item, or the re-establishment of a cancelled project;
 c) the change in the type of an existing document, e.g. conversion of a Technical Specification into an International Standard.

This form is not intended for use to propose an action following a systematic review - use ISO Form 21 for that purpose.

Proposals for correction (i.e. proposals for a Technical Corrigendum) should be submitted in writing directly to the secretariat concerned.

Guidelines on the completion of a proposal for a new work item

(see also the ISO/IEC Directives Part 1)

- a) **Title:** Indicate the subject of the proposed new work item.
 b) **Scope:** Give a clear indication of the coverage of the proposed new work item. Indicate, for example, if this is a proposal for a new document, or a proposed change (amendment/revision). It is often helpful to indicate what is not covered (exclusions).
 c) **Envisaged publication type:** Details of the types of ISO deliverable available are given in the ISO/IEC Directives, Part 1 and/or the associated ISO Supplement.
 d) **Purpose and justification:** Give details based on a critical study of the following elements wherever practicable. *Wherever possible reference should be made to information contained in the related TC Business Plan.*
- 1) The specific aims and reason for the standardization activity, with particular emphasis on the aspects of standardization to be covered, the problems it is expected to solve or the difficulties it is intended to overcome.
 - 2) The main interests that might benefit from or be affected by the activity, such as industry, consumers, trade, governments, distributors.
 - 3) Feasibility of the activity: Are there factors that could hinder the successful establishment or global application of the standard?
 - 4) Timeliness of the standard to be produced: Is the technology reasonably stabilized? If not, how much time is likely to be available before advances in technology may render the proposed standard outdated? Is the proposed standard required as a basis for the future development of the technology in question?

5) Urgency of the activity, considering the needs of other fields or organizations. Indicate target date and, when a series of standards is proposed, suggest priorities.

6) The benefits to be gained by the implementation of the proposed standard; alternatively, the loss or disadvantage(s) if no standard is established within a reasonable time. Data such as product volume or value of trade should be included and quantified.

7) If the standardization activity is, or is likely to be, the subject of regulations or to require the harmonization of existing regulations, this should be indicated.

If a series of new work items is proposed having a common purpose and justification, a common proposal may be drafted including all elements to be clarified and enumerating the titles and scopes of each individual item.

e) Relevant documents and their effects on global relevancy : List any known relevant documents (such as standards and regulations), regardless of their source. When the proposer considers that an existing well-established document may be acceptable as a standard (with or without amendment), indicate this with appropriate justification and attach a copy to the proposal.

f) Cooperation and liaison: List relevant organizations or bodies with which cooperation and liaison should exist.

ISO/TC 147/SC 2 N **1069**

Date: 2009-08-27

ISO/WD 17378-2

ISO/TC 147/SC 2/WG 52 N **0012**

Secretariat: DIN

Water quality — Determination of arsenic — Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)

Qualité de l'eau — Dosage de l'arsenic — Partie 2: Méthode par spectrométrie d'absorption atomique à production d'hydruure (PA-SAA)

Warning

This document is not an ISO International Standard. It is distributed for review and comment. It is subject to change without notice and may not be referred to as an International Standard.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

Document type: International Standard

Document subtype:

Document stage: (20) Preparatory

Document language: E

X:\TA2\TG2-2\NA119\Normen\Normen_FB_01_bja_dom_foi_got_ptz\ISO-Normen_Normen_ISO_TC_147\ISO_17378_P2_Arsenic_AAS_SC2_WG52\NWIP_ISO_WD_17378-2_(E).doc STD Version 2.2

Copyright notice

This ISO document is a working draft or committee draft and is copyright-protected by ISO. While the reproduction of working drafts or committee drafts in any form for use by participants in the ISO standards development process is permitted without prior permission from ISO, neither this document nor any extract from it may be reproduced, stored or transmitted in any form for any other purpose without prior written permission from ISO.

Requests for permission to reproduce this document for the purpose of selling it should be addressed as shown below or to ISO's member body in the country of the requester:

[Indicate the full address, telephone number, fax number, telex number, and electronic mail address, as appropriate, of the Copyright Manager of the ISO member body responsible for the secretariat of the TC or SC within the framework of which the working document has been prepared.]

Reproduction for sales purposes may be subject to royalty payments or a licensing agreement.

Violators may be prosecuted.

Contents

Page

Foreword	iv
Introduction.....	v
1 Scope	1
2 Normative references	1
3 Principle	2
4 Interferences	2
5 Reagents and standards.....	3
6 Apparatus	5
7 Sampling and sample preparation.....	6
8 Instrumental set up	7
9 Procedure	8
10 Calibration and data analysis.....	9
11 Expression of results	10
12 Test report.....	10
Annex A (informative) Additional information	11
Annex B (informative) Schematic flow diagram and signal response	12
Annex C (informative) Example of enrichment technique	14
Annex D (informative) Precision data	16
Bibliography.....	18

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17378-2 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This edition cancels and replaces the edition (ISO 11969:1996), which has been technically revised.

ISO 17378 consists of the following parts, under the general title *Water quality — Determination of arsenic*:

- *Part 1: Method using hydride generation atomic fluorescence spectrometry (HG-AFS)*
- *Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)*

Introduction

This part of ISO 17378 shall be used by analysts experienced with the handling of trace elements at very low concentrations.

In natural water sources, arsenic compounds generally occur in very small quantities, typically less than 1 µg/l. Higher concentrations may be found, for example, in industrial waste water. Arsenic occurs naturally in organic and inorganic compounds and may have valency states –3, 0, 3 and 5.

In order to fully decompose all of the arsenic compounds, a digestion procedure is necessary. Digestion can only be omitted if it is certain that the arsenic in the sample can form a covalent hydride without the necessity of a pre-oxidation step.

The user should be aware that particular problems could require the specification of additional marginal conditions.

Water quality — Determination of arsenic — Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)

WARNING — Persons using this part of ISO 17378 should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this part of ISO 17378 be carried out by suitably trained staff.

1 Scope

This part of ISO 17378 specifies a method for the determination of arsenic. The method is applicable to drinking water, surface water, ground water and rain water. The linear application range of this standard is from 0,5 µg/l to 20 µg/l. Samples containing arsenic at higher concentrations than the application range can be analysed following appropriate dilution [1]. The method is unlikely to detect organo-arsenic compounds.

The sensitivity of this method is dependent on the selected operating conditions.

It is important to use high purity reagents in all cases with minimum levels of arsenic. The concentration of the blank solution shall be less than the lower level of interest.

2 Normative references

The following reference documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 5667-2, *Water quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

ISO 8466 1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second order calibration function*

ISO 15587-1, *Water quality — Digestion for the determination of elements in water — Part 1: Aqua regia digestion*

3 Principle

The sample solutions are treated with sodium tetrahydroborate to generate the covalent gaseous arsenic trihydride (arsine, AsH_3). The hydride and excess hydrogen are swept out of the generation vessel into a heated silica glass cell. After atomisation, the absorbance of arsenic is determined at a wavelength $\lambda = 193,76 \text{ nm}$. The procedure is automated by means of auto-sampler and control software.

Other measurement techniques may be applicable providing the performance criteria can be adequately demonstrated or exceeded by the user laboratory. (See Annex C).

4 Interferences

The hydride generation technique is prone to interferences by transition and easily reducible metals. For the majority of natural water samples, this type of interference shall not be significant. The user shall carry out recovery tests on typical waters and also determine the maximum concentrations of potentially interfering elements, using appropriate methods. If such interferences are indicated, the level of interferences shall be assessed by performing spike recoveries.

The reaction conditions in this standard have been chosen to minimise these interferences.

Further information concerning these interferences and the technique are available from the Bibliography.

Elements such as Sb, Se, Te, Hg and Sn are also volatilised by this procedure and may cause interferences because of decrease of absorption. These elements will not cause interferences provided the concentrations specified in Table 1 are not exceeded.

Table 1 — Maximum concentration by mass of hydride-forming elements causing no interferences

		Element				
	Unit	Sb	Se	Sn	Te	Hg
Concentration by mass of interfering elements in test solution	[mg/l]	1	1	0,1	1	0,1

If these concentrations are exceeded, it may be necessary to use the standard addition method (9.3). Assuming that the arsenic content is high enough, an appropriate dilution of the water sample is preferred.

Metals which are readily reduced by sodium tetrahydroborate may also cause interferences. In particular, these include chromium, iron, copper, nickel and lead. If the concentrations of these elements specified in Table 2 are exceeded, a significant decrease of absorption may occur.

Table 2 — Maximum concentration by mass in test solution of interfering heavy metals (valid for flow systems)

		Interfering element				
	Unit	Cr	Fe	Cu	Ni	Pb
Concentration by mass in test solution	[mg/l]	500	500	500	250	100

NOTE If batch systems are used, concentrations which are appreciably lower by mass than those specified in Table 1 and Table 2 may cause interferences.

Since most organic solvents (e.g. alcohols, benzene, ketones) cause severe interferences (signal suppression), they shall be removed by digestion prior to analysis.

5 Reagents and standards

5.1 General requirements

Reagents may contain arsenic as an impurity. All reagents shall have arsenic concentrations below that which would result in an arsenic blank value for the method being above the lowest level of interest.

Reagents shall be prepared to manufacturer's recommendations using the following series as an example.

5.2 Water, complying with grade 1 water as defined in ISO 3696 for all sample preparation and dilutions.

5.3 Hydrochloric acid, $\rho(\text{HCl}) = 1,16 \text{ g/ml}$.

5.4 Hydrochloric acid, $c(\text{HCl}) = 1 \text{ mol/l}$.

5.5 Sodium hydroxide, NaOH.

5.6 Sodium tetrahydroborate solution, $\rho(\text{NaBH}_4) = 13 \text{ g/l}$.

Dissolve $13,0 \text{ g} \pm 0,1 \text{ g}$ of sodium tetrahydroborate in 500 ml water (5.2) and add $4,0 \text{ g} \pm 0,1 \text{ g}$ of sodium hydroxide (5.5). Dilute to 1 000 ml with water (5.2). Filter the solution through a $0,45 \mu\text{m}$ membrane filter before use.

Prepare on day of use and do not keep in a closed container because of pressure build-up due to hydrogen evolution.

NOTE 1 Suitably stored sodium tetrahydroborate pellets have a shelf life of six months. The concentration of NaBH_4 will be dependent on the hydride generator manifold and flow-rate conditions. See recommendations of the manufacturer.

NOTE 2 Alternatively smaller volumes can be prepared on a pro rata basis.

5.7 Nitric acid, $\rho(\text{HNO}_3) = 1,40 \text{ g/ml}$.

NOTE Nitric acid is available both as $\rho(\text{HNO}_3) = 1,40 \text{ g/ml}$ ($w(\text{HNO}_3) = 650 \text{ g/kg}$) and $\rho(\text{HNO}_3) = 1,42 \text{ g/ml}$ ($w(\text{HNO}_3) = 690 \text{ g/kg}$).

5.7.1 Nitric acid cleaning mixture

Dilute nitric acid (5.7) with an equal volume of water (5.2) by carefully adding the acid to the water.

5.8 Potassium iodide-ascorbic acid solution

Dissolve $(250 \pm 0,1) \text{ g}$ of potassium iodide and $(50 \pm 0,1) \text{ g}$ of ascorbic acid in approximately 400 ml water and dilute to 500 ml. Prepare fresh on day of use. (See Note 2 in (5.6)).

5.9 Reagent blank

For each 1 000 ml, prepare a solution containing $300 \text{ ml} \pm 3 \text{ ml}$ of hydrochloric acid (5.3) and $20 \text{ ml} \pm 0,5 \text{ ml}$ of potassium iodide-ascorbic acid solution (5.8). Dilute to volume with water (5.2).

NOTE On the continuous flow system, the reagent blank solution is run as background. Since the blank solution may contain trace level detectable amounts of arsenic it is important that the same reagents are used for sample and standard preparation as well as the preparation of the reagent blank.

The analyte signal will be superimposed on the top of this signal once the sample is introduced into the measurement cycle.

5.10 Arsenic standard solutions

5.10.1 Arsenic stock solution A, $\rho(\text{As(III)}) = 1\,000\text{ mg/l}$.

Use a quantitative stock solution with an arsenic content of $1\,000\text{ mg/l} \pm 2\text{ mg/l}$. This solution is considered to be stable for at least one year.

NOTE Other stock solutions may be available and can be used providing the uncertainty of the measurement is not compromised.

Alternatively, use a stock solution prepared from high purity grade chemicals:

Place $1,7343\text{ g} \pm 0,0020\text{ g}$ of sodium metaarsenite NaAsO_2 in a $1\,000\text{ ml}$ volumetric flask.

Add $50\text{ ml} \pm 0,5\text{ ml}$ of hydrochloric acid (5.3) and dissolve the sodium metaarsenite completely by stirring.

Dilute to 1 l with water (5.2).

5.10.2 Arsenic standard solution B, $\rho(\text{As(III)}) = 10\text{ mg/l}$.

Pipette $1\text{ ml} \pm 0,01\text{ ml}$ of arsenic stock solution A (5.10.1) into a 100 ml volumetric flask, add $30\text{ ml} \pm 0,5\text{ ml}$ of hydrochloric acid (5.3) and $2\text{ ml} \pm 0,1\text{ ml}$ of potassium iodide-ascorbic acid solution (5.8) and fill up to the mark with water (5.2). This solution is stable for one week.

5.10.3 Arsenic standard solution C, $\rho(\text{As(III)}) = 100\text{ }\mu\text{g/l}$.

Pipette $1\text{ ml} \pm 0,01\text{ ml}$ of arsenic standard solution B (5.10.2) into a 100 ml volumetric flask, add $30\text{ ml} \pm 0,5\text{ ml}$ of hydrochloric acid (5.3) and $2\text{ ml} \pm 0,1\text{ ml}$ of potassium iodide-ascorbic acid solution (5.8) and fill up to the mark with water (5.2). This solution is stable for one week.

5.10.4 Arsenic standard solution E, $\rho(\text{As(V)}) = 1\,000\text{ mg/l}$.

Dissolve $1,000\text{ g} \pm 0,002\text{ g}$ of pure arsenic powder in $10\text{ ml} \pm 0,1\text{ ml}$ of concentrated nitric acid (5.7).

Heat the solution to boiling and evaporate off the excess nitric acid.

Cool and then take up the hydrated arsenic(V) oxide in $50\text{ ml} \pm 0,5\text{ ml}$ of cold hydrochloric acid (5.3).

Transfer the solution quantitatively to a $1\,000\text{ ml}$ volumetric flask and fill up to the mark with water (5.2).

This standard shall be used to prepare a suitable arsenic(V) standard to check quantitative recovery of arsenic (V). Should the presence of As(V) in the samples be suspected then this standard must be used to check recovery of this analyte. The solution is stable for at least six months.

NOTE Other stock solutions may be available and can be used providing the uncertainty of the measurement is not compromised.

5.10.5 Arsenic calibration solutions

A minimum of five independent calibration solutions shall be used. The calibration is described in ISO 8466-1.

Prepare a minimum of five arsenic calibration solutions from the arsenic standard solution C (5.10.3) covering the working range of expected arsenic concentrations.

For the operating range from $1\text{ }\mu\text{g/l}$ to $5\text{ }\mu\text{g/l}$, for example, proceed e.g. as follows:

Pipette into a series of five 100 ml volumetric flasks $1\text{ ml} \pm 0,01\text{ ml}$, $2\text{ ml} \pm 0,02\text{ ml}$, $3\text{ ml} \pm 0,03\text{ ml}$, $4\text{ ml} \pm 0,04\text{ ml}$ and $5\text{ ml} \pm 0,05\text{ ml}$ respectively of arsenic standard solution C (5.10.3).

Add 30 ml of hydrochloric acid (5.3) and 2 ml of potassium iodide-ascorbic acid solution (5.8).

Dilute to 100 ml with water (5.2) and mix thoroughly.

Allow to stand for at least 2 h before using the solution. This will ensure quantitative reduction of arsenic(V) to arsenic(III).

These calibration solutions contain 1 µg/l, 2 µg/l, 3 µg/l, 4 µg/l and 5 µg/l arsenic respectively. They shall be prepared on the day of use.

The use of piston pipettes is permitted and enables the preparation of lower volumes of calibration solutions. The application of dilutors is also allowed.

Once a well established calibration pattern has been established the number of standards used routinely may be reduced. Any such change shall not alter the result obtained from tests or the ranking with other samples.

6 Apparatus

The following are set out as guidelines. In general, the manufacturer's instructions shall be followed.

6.1 Atomic absorption spectrometer

An atomic absorption spectrometer equipped with a hydride generation system and a heated quartz tube atomiser or a graphite furnace atomiser (preferably equipped with a background correction system) is recommended. Automated flow systems (flow injection analysis (FIA) or continuous flow analysis (CFA)) for hydride generation are suitable and more usual but batch systems are also adequate. The description below follows a continuous flow regime.

6.1.1 Radiation source for the determination of arsenic

Use arsenic hollow cathode lamp or electrodeless discharge lamp.

6.2 Gas supply

Use argon with a grade specified by the manufacturer.

The gas supply shall be with a two stage regulator and the argon supplied at a pressure recommended by the manufacturer.

The use of a gas purifier consisting of activated carbon is recommended.

Nitrogen gas may also be used but will have a reduced sensitivity.

6.3 Laboratory ware

6.3.1 General requirements

For the determination of arsenic at very low concentrations, contamination and loss are of critical consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment. A clean laboratory work area, designated for trace element sample handling shall be used.

All re-usable laboratory ware in contact with the sample shall be cleaned prior to use.

Laboratory ware shall be soaked in the nitric acid cleaning mixture (5.7.1) for at least 24 h and rinsed five times with water (5.2).

Following this, refill laboratory ware with hydrochloric acid, $c(\text{HCl}) = 1 \text{ mol/l}$ (5.4) and leave for 24 h.

Disposable (single-use) plastic laboratory ware does not require special cleaning provided that negligible arsenic contamination in that material is demonstrated.

6.3.2 Storage and sample processing bottles

Use sampling vessels constructed of quartz, borosilicate glass, plastic materials (e.g. polytetrafluoroethene (PTFE), perfluoro (ethylene-propylene) (FEP)) or other material that neither adsorbs nor desorbs the analyte under test.

6.3.3 Instrument reagent reservoir

The reagents are delivered via a peristaltic pump from glass reagent bottles through PTFE transfer lines. All pump tubing shall be compatible with reagents in use and neither adsorbs nor desorbs the analyte under test.

6.3.4 Auto-sampler vials

Use vials constructed of materials as specified in 6.3.2.

6.4 Sample processing equipment

6.4.1 Air displacement pipette

Micro-pipette system capable of delivering volumes from 10 μl to 1 000 μl with an assortment of metal-free disposable pipette tips.

6.4.2 Balances

Analytical balance, capable of accurately weighing (standards) to $\pm 0,1 \text{ mg}$; and a top-pan balance for the preparation of solutions, accurate to $\pm 0,1 \text{ g}$.

7 Sampling and sample preparation

7.1 Sampling technique

Carry out the sampling as specified in ISO 5667-1, ISO 5667-2 and ISO 5667-3, using sampling vessels as specified in 6.3.2.

For the determination of arsenic in aqueous samples, acidify at time of sampling to $\text{pH} < 2$. Hydrochloric acid (5.3) 3 ml \pm 0,1 ml per litre is sufficient for most samples. Ensure that the pH is less than 2; otherwise, add more hydrochloric acid as required.

On receipt in the laboratory, all samples shall be logged and processed in a timeframe that will not affect the integrity of the analyte.

For all types of samples, prepare an appropriate field blank and analyse as required. Use the same type of vessel and quantity of acid as used in the sample.

7.2 Pre-reduction

Since only arsenic(III) reacts quickly and quantitatively under the conditions used in the hydride technique, arsenic(V) has to be reduced to arsenic(III) prior to the step of hydride generation.

7.2.1 Standard procedure for water samples with no solid or organically bound arsenic

Pre-treat water samples, free from solid materials and organically bound arsenic field blanks and blank solutions in the following way:

Accurately transfer an aliquot of the sample (40 ml to 50 ml) to a 100 ml tared volumetric flask.

Add 30 ml \pm 0,5 ml of hydrochloric acid (5.3).

Add 2 ml \pm 0,1 ml of potassium iodide-ascorbic acid solution (5.8), mix and allow to stand for at least 2 h. This will ensure quantitative reduction of arsenic(V) to arsenic(III).

Transfer to a volumetric flask and dilute to 100 ml with water (5.2).

If other sample volumes are applied, use reagents and equipment adequate for the chosen volumes.

NOTE For greater accuracy the sample may also be dispensed by weight. In this case calculate the volume from the density and the weight and record the volume.

7.2.2 Samples requiring additional digestion

Samples that contain significant amounts of solid material and/or organically bound arsenic will require an additional digestion step as specified in ISO 15587-1. This is outside the scope of this standard but samples may be analysed using a similar procedure providing correctly matrix-matched reagents shall be prepared using the correct proportion of nitric acid (5.7) and hydrochloric acid (5.3). Blanks and standard solutions must also be matrix-matched.

8 Instrumental set up

Configure the instrumentation as described in the instrument manufacturer's manual. The following relates to continuous flow systems, for details on flow injection instruments or batch analysers refer to manufacturer's instruction manual.

Check tubing for wear and pumping reliability each day that the system is used and replace if necessary. All tube distances between the auto-sampler, vapour generator and detector shall be kept to a minimum length.

Fill the reagent reservoirs with reagent blank solution (5.9) and sodium tetrahydroborate solution (5.6) respectively.

Set up the continuous flow vapour generator according to the manufacturer's instructions. Ensure that the reagent flows are within the accepted balances. Once stable conditions are established analysis can proceed. Both standards and samples shall be quantified using the same flow characteristics.

Select the required amplification range for the atomic absorption spectrometer to ensure that it is appropriate for the sample concentration being determined.

For samples which are above the calibration at a given range setting, reanalyse after dilution of the sample into the calibration range. If the sample is diluted, then the diluent shall be the reagent blank (5.9) i.e. matrix-matched. Samples which are digested shall be matrix-matched against standards and blanks to provide reliable data.

9 Procedure

9.1 General requirements

Before beginning the measurement procedure, set the instrument parameters according to the manufacturer's instructions (wavelength $\lambda = 193,76$ nm).

Align the quartz tube atomiser or in case of using the graphite furnace atomisation condition an iridium-coated graphite tube according to the manufacturer's instructions.

Set the argon or nitrogen flow until a constant signal has been reached.

If a flow system is used, hydrochloric acid (5.4) shall be used as carrier solution. See recommendations of the manufacturer.

The sodium tetrahydroborate solution (5.6) and all pre-reduced test solutions (reagent blank solution (5.9), calibration solutions (5.10.5) and samples (7.2) are introduced to the flow system according to the manufacturer's instructions and measurements are carried out.

Measure the absorbance of each solution at least twice.

9.2 Analysis using the method of standard calibration

A standard calibration shall only be applied if matrix effects can be excluded. Otherwise samples could be diluted, assuming that the arsenic content is high enough. If not, use the standard addition method (9.3).

Plot a calibration graph for the analysis using the following procedure:

Prepare arsenic calibration solutions as described in 5.10.5.

Determine the absorbance references of the arsenic calibration solutions (5.10.5) and of the undigested, but pre-reduced reagent blank solution (5.9).

Establish a calibration graph using the series of results thus obtained.

Measure the absorbance references of the test solutions and of the digested, pre-reduced reagent blank solution (5.9). If the arsenic content of the test solution is not within the range of the calibration graph, dilute the solution with reagent blank solution (5.9).

After each series of samples, and in any case after about 10 to 20 measurements, use the reagent blank solution (5.9) and a calibration solution of average concentration to check whether the measured values still agrees with the calibration graph.

Measure the absorbance of each solution at least twice.

Calculate the arsenic content of the measured solutions according to 10.2.

9.3 Analysis using the standard addition method of calibration

The use of the method of standard addition can compensate for errors due to matrix effects, provided that no additive errors occur and that the absorbance references of the spiked measurement solutions are within the working range of the calibration function.

Prepare and measure at least two test solutions containing each standard addition. An example for the preparation of 10 ml test portions (10 ml volumetric flasks) is shown in Table 3.

Table 3 — Example of standard addition method (volumes adapted for 10 ml volumetric flasks)

Test solution	Test solution ml	KI-Ascorbic-acid-solution (5.8) ml	Arsenic standard solution C (5.10.3) ml	Concentration of arsenic in solution added µg/l
Test solution according to 7.2	5	0,5	0	0
Reference solution 1	5	0,5	0,1	2
Reference solution 2	5	0,5	0,2	4
Reference solution 3	5	0,5	0,3	6

Fill up all volumetric flasks to the mark with water (5.2).

NOTE The filling of the volumetric flasks to 10 ml is not to be considered as a dilution step in the subsequent calculation.

Treat and measure the reagent blank solution (5.9) in the same way.

Measure the absorbance of each solution at least twice.

Calculate the arsenic content of the measured solutions according to (10.3).

If volumetric flasks of other nominal volumes are used, adapt volumes of test, reagent and standard solution accordingly and maintain the quantitative ratios.

10 Calibration and data analysis

10.1 General requirements

The dilution factor of each sample shall be applied. If additional dilutions were made to any samples, the appropriate factor shall be applied to the calculated sample concentrations. Concentrations of samples where additional reagents were added to preserve the sample shall be corrected with the corresponding blank subtraction. Care shall be exercised to correctly matrix match these solutions.

10.2 Calculation using the calibration curve

Determine the calibration curve from the data measured for the calibration solutions e.g. by using the method of linear regression.

Calculate the concentration of arsenic, $\rho[As]$ in the samples using Equation (1):

$$\rho[As] = \frac{(A_s - A_{s0}) \cdot V_M}{b \cdot V_p} \quad (1)$$

where

$\rho[As]$ is the concentration of arsenic in the sample in micrograms per litre, µg/l;

A_s is the absorbance response of the water sample;

A_{s0} is the absorbance of the reagent blank solution;

b is the slope of the calibration curve and a measure of the sensitivity in litres per microgram, l/ μ g;

V_M is the volume of measurement solution in millilitres, ml;

V_P is the volume of sample used to prepare the measurement solution in millilitres, ml.

If dilutions deviating from the above mentioned steps are used, the calculation shall be modified accordingly.

A second order calibration function is allowed. In this case, the calculations shall be carried out according to ISO 8466-2.

The use of appropriate instrument software for the calculations is also allowed.

10.3 Calculation using the standard addition method

Prepare a calibration line with concentration as abscissa and absorbance as ordinate. Use the measured absorbance references of the unspiked and spiked measurement solutions step-wise with increasing arsenic concentrations.

Obtain the concentration of arsenic in the sample by extrapolation of the calibration line to absorbance $A = 0$. Similarly, determine the arsenic concentration of the reagent blank solution and subtract from the result obtained for the sample.

Alternatively, carry out the evaluation by an automated method of linear regression.

11 Expression of results

Report the results in μ g/l and round them to the nearest 0,1 μ g/l. Do not use more than two significant figures.

EXAMPLE

Arsenic (As) 2,3 μ g/l

Arsenic (As) 13 μ g/l

12 Test report

This clause specifies which information is to be included in the test report. The clause shall require information to be given on at least the following aspects of the test:

- a) a reference to this part of ISO 17378;
- b) complete identification of the sample;
- c) expression of results as indicated in Clause 11;
- d) sample pre-treatment;
- e) any deviations from this method and details of all circumstances which could have affected the result;
- f) identification of laboratory and experience of these measurements.

Annex A

(informative)

Additional information

A.1 The method requires proper attention to detail to attain the low levels of measurement. Other stabilisation strategies have been proven particularly the addition of high purity nitric acid.

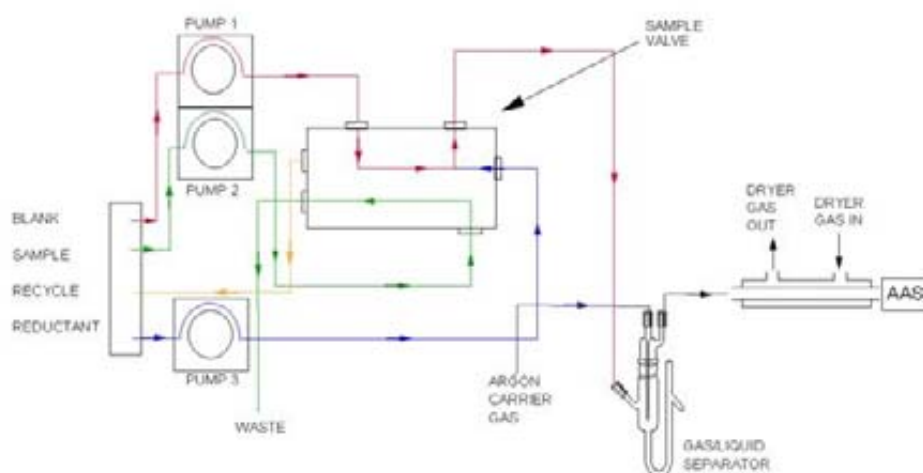
A.2 The method and any variation from it shall be rigorously checked for performance using statistical data and analytical quality control sample materials, including certified reference materials.

A.3 Air shall not be used because of explosion risk.

A.4 Although water vapour is not normally a problem with atomic absorption methods in automated instruments moisture could condense in the transfer lines. A Nafion dryer system can alleviate this potential problem; these are commercially available.

Annex B (informative)

Schematic flow diagram and signal response



Key

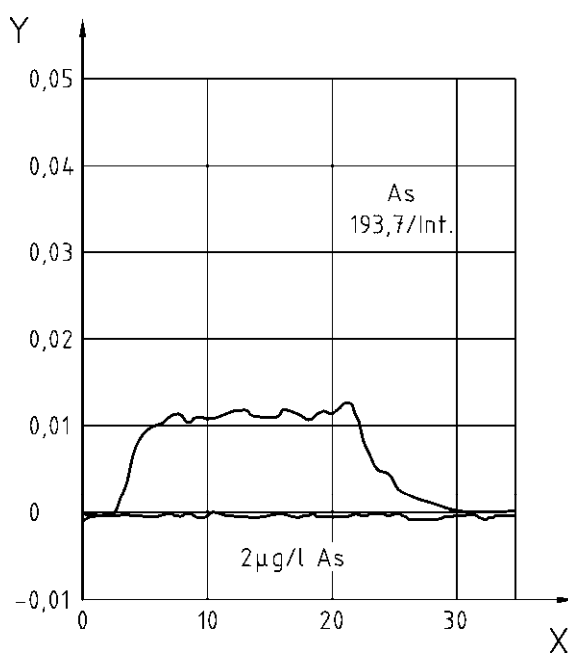
1	pump 1	8	sample valve
2	pump 2	9	waste
3	pump 3	10	argon carrier gas
4	blank	11	gas liquid separator
5	sample	12	dryer gas out from Nafion dryer system (Annex A.4)
6	recycle	13	dryer gas in to Nafion dryer system (Annex A.4)
7	reductant	14	atomic absorption spectrometer

Argon or nitrogen can be used.

NOTE This continuous flow vapour generator consists of a constant speed peristaltic pump to deliver sodium tetrahydroborate (5.6), reagent blank and sample. A switching valve alternates between the reagent blank and sample or standard solutions. The vapour generator switches between reagent and sample solution on a prescribed sequence so that the measured signal is directly related to the background levels of arsenic in the sample. The typical signal response is shown below in Figure B.2.

Figure B.1 — Schematic flow diagram of hydride generation system

This is only an example; any other suitable system may be used subject to satisfactory performance data.



Key

X time in seconds
Y absorbance signal

NOTE Background signal is made up of instrumental blank and reagent blank. When a sample is analysed the signal from arsenic in the sample is super imposed on the background level.

Peak rises to its maximum as sample is introduced and returns to baseline.

Matrix of sample/standards and blank **shall** be matrix-matched.

**Figure B.2 — Typical signal response from arsenic in water sample
by atomic absorption spectrometry**

Annex C

(informative)

Example of enrichment technique

C.1 Introduction

If arsenic is enriched in an iridium-coated graphite tube prior to atomisation and measurement by AAS the lower limit of the working range is 0,02 µg/l within the scope of this standard. The concentration of the arsenic in the blank solution shall not exceed 10 ng/l. If the concentration of the blank is controlled then the levels of detection achieved with the enrichment techniques will exceed the requirement set out in the scope of this method.

C.2 Principles involved in this technique

An aliquot of the sample is digested with concentrated hydrochloric acid. Potassium iodide-ascorbic acid reagent is added to ensure quantified reduction of the arsenic(V) to arsenic(III). The subsequent sample solutions are then treated with sodium tetrahydroborate to generate the covalent gaseous arsenic trihydride (AsH₃). The hydride and excess hydrogen are swept out of the generation vessel and transferred and sequestered in an iridium-coated graphite tube. After atomisation, the absorbance of arsenic is determined at a wavelength $\lambda = 193,76$ nm. The procedure is automated by means of an auto-sampler and control software.

C.3 Additional reagents and standards

C.3.1 Use a commercially available quantitative stock solution with an iridium content of 1 000 mg/l. This solution is considered to be stable for at least one year, but in reference to guaranteed stability, see the recommendations of the manufacturer.

C.4 Additional apparatus and instrumentation

C.4.1 Graphite furnace atomic absorption spectrometer

An atomic absorption spectrometer equipped with a hydride system and a graphite furnace atomiser (preferably equipped with a background correction system) is recommended. Automated flow systems (FIA or CFA) for hydride generation are suitable and more usual but batch systems are also adequate.

In case of analyte trapping on the graphite tube, the generated hydride is guided with the help of the furnace auto-sampler into the graphite tube. The tube has to be heated to about 300 °C to decompose the hydrides. The analyte arsenic is trapped on the activated graphite surface. The auto-sampler capillary has to be equipped with a quartz tip. The auto-sampler graphite furnace and hydride generator have to be synchronised normally by computer control.

C.5 Instrumental set up

Configure the instrumentation as described in the instrument manufacturer's manual.

Check tubing for wear and pumping reliability each day the system is used and replace if necessary. All tube distances between the auto-sampler, vapour generator and detector shall be kept to a minimum length.

Prior to trapping the analyte in a graphite tube, the electrothermal atomiser shall first be activated. Introduce the recommended volume of Iridium chloride solution (C.3.1) into the graphite tube. Dry, pyrolyze and heat out according to the manufacturers' recommendations. The process of tube activation is repeated twice and will require about 10 min. The activated tubes can be used for at least 300 measurement cycles before they have to be reactivated.

The programmes for furnace and hydride generator are set according to the recommendations of the manufacturers.

Fill the reagent reservoirs with reagent blank solution (5.9) and sodium tetrahydroborate solution (5.6) respectively.

C.6 Procedure

C.6.1 General requirements

Before beginning the measurement procedure, set the instrument parameters according to the manufacturer's instructions (wavelength $\lambda = 193,76$ nm).

In case of analyte trapping in the graphite tube the process of hydride generation is similar to the quartz atomiser procedure. The generated hydride however is preconcentrated in the graphite tube. The sensitivity and the detection limits are therefore a function of the preconcentration time. A preconcentration time of between 10 s to 30 s is usually required to obtain detection limits required by this standard.

Follow the manufacturer's recommendations for the hydride generator programme. The trapped arsenic is atomised using a short graphite furnace programme consisting of a step to remove excess hydrogen from the graphite tube (approximately 10 s at 300 °C), an atomisation step (approximately 5 s at 2,300 °C) and a cleaning step (approximately 5 s at 2,500 °C). For more detailed information follow the manufacturer's recommendations.

While passing argon or nitrogen, adjust the zero point after a steady baseline signal has been reached.

If a flow system is used, hydrochloric acid (5.4) shall be used as carrier solution. See recommendations of the manufacturer.

The sodium tetrahydroborate solution (5.6) and all pre-reduced test solutions (reagent blank solution (5.9), calibration solutions (5.10.5) and samples (7.2) are introduced to the flow system according to the manufacturer's instructions and measurements are carried out.

Measure the absorbance of each solution at least twice. The user shall confirm that the operation of the above procedures provides data in the range of detection and repeatability required. The results shall be traceable to international standards.

Annex D (informative)

Precision data

An international laboratory trial was organised by Professor Peter B Stockwell, Convenor of WG52 with the assistance of Professor Clive Thompson and performed in October 2006 by P S Analytical Ltd, Orpington, UK and Alcontrol Ltd, Rotherham, UK. 17 laboratories from 7 countries took part (UK: 5, France: 5, Germany: 3, Italy: 1, The Netherlands: 1, Slovakia: 1 and USA: 1).

A set of 12 samples containing drinking water, surface water and waste water were analysed in accordance with the standard method. The sample matrix is shown in Table D.1 and the performance data is summarised in Table D.2.

The data summarised in Table D.2 proved the validity of this standard method and the draft will therefore proceed to the next stage of implementation.

Table D.E.1 — Interlaboratory trial samples for antimony, arsenic and selenium

Sample No	Description	Speciation of added analyte
1	Low standard 10% of calibration range	As(III), Sb(III), Se(IV)
2	High standard 90% of calibration range	As(V), Sb(V), Se(VI)
3	Real sample 1 ^a (soft water) + mid-range (40 - 60%) spike	As(III), Sb(III), Se(IV)
4	Real sample 2 ^a (hard water) + high-range spike (70 – 90%)	As(V), Sb(V), Se(VI)
5	Real sample 3 ^a (intermediate hardness water) + low-range (10 – 20%) spike	As(III), Sb(III), Se(IV)
^a All three real samples will be analysed by two prestigious laboratories prior to spiking to demonstrate that the background level of the three elements is negligible.		

An interlaboratory trial carried out in 2006 yielded the results given in Table D.2

Table D.2 — Performance data

Sample	Matrix	l	n	n_{OP} %	\bar{X} $\mu\text{g/l}$	x_{ass} $\mu\text{g/l}$	η %	s_R $\mu\text{g/l}$	CV_R %	s_r $\mu\text{g/l}$	CV_r %
1	Nutwell hard water 90% spike	8	22	0,0	18,4	18,0	102,1	1,38	7,5	0,95	5,2
2	Rotherham interm. water 15%	8	22	0,0	2,81	3,0	93,7	0,389	13,8	0,174	6,2
3	Bradfort soft water 60% spike	8	23	0,0	11,8	12,0	98,4	0,83	7,1	0,59	5,0
4	Low standard 20%	8	22	0,0	3,82	4,0	95,4	0,479	12,6	0,181	4,7
5	Blank	-	-	-	-	-	-	-	-	-	-
6	High standard 80%	8	23	0,0	15,6	16,0	97,3	1,09	7,0	0,90	5,8
7	Blank	-	-	-	-	-	-	-	-	-	-
8	Rotherham interm. water 15%	8	22	0,0	3,07	3,0	102,3	0,263	8,6	0,145	4,7
9	Low standard 20%	8	22	0,0	3,98	4,0	99,5	0,301	7,6	0,217	5,5
10	High standard 80%	8	22	0,0	15,0	16,0	94,0	1,27	8,4	0,99	6,6
11	Nutwell hard water 90% spike	8	22	0,0	18,4	18,0	102,2	1,30	7,1	0,78	4,2
12	Bradfort soft water 60% spike	8	22	0,0	11,8	12,0	98,4	0,96	8,1	0,36	3,1
13	Standard 30 $\mu\text{g/l}$	7	19	13,6	28,4	30,0	94,6	1,96	6,9	0,87	3,1
l is the number of laboratories after outlier rejection; n is the number of analytical results after outlier rejection; n_{OP} is the number of outliers; \bar{X} is the total mean of results (without outliers); x_{ass} is the assigned value; η is the recovery; s_R is the reproducibility standard deviation; CV_R is the reproducibility variation coefficient; s_r is the repeatability standard deviation; CV_r is the repeatability variation coefficient.											

Bibliography

- [1] DEDINA, J. and TSALAV, D.L., Hydride Generation Atomic Absorption Spectrometry, Wiley, 1995
- [2] WELZ, B. and SPERLING, M.: Atomic Absorption Spectrometry, 4th ed., WILEY-VCH, Weinheim (Germany); 1997
- [3] SCHLEMMER, G.; RADZIUK, B.: Analytical Graphite Furnace Atomic Absorption Spectrometry, A Laboratory Guide, Birkhäuser publisher, Basel (Switzerland); 1999

Template for comments and secretariat observations					Date:2006-09-21	Document: ISO/DIS 17378-2
REVISED 2009-08-13						
1	2	(3)	4	5	(6)	(7)
MB ¹	Clause No./ Subclause No./ Annex (e.g. 3.1)	Paragraph/ Figure/ Table/ Note (e.g. Table 1)	Type of comment ²	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
SK 001	Content	10	ge	Data analysis and calibration	We suggest changing order Calibration and data analysis.	?
** 002	D	Table D.1	ed	To be consistent with ISO 17353:2004, please use "o" as the symbol for the percentage of outliers instead of "n _{AP} ".		Accept.
FI 003	Foreword		ge	Does this new standard cancel and replace ISO 11969:1996 Water quality – Determination of arsenic – Atomic absorption spectrometric method (hydride technique)? If yes, it should be mentioned in foreword.		Yes.
SK 004	1	1 st para	te	The method is applicable to drinking water, surface water, ground water and rain water.	We suggest adding waste water I also agree with this proposal, we use it every day for waste water	No, not agreed But we have a note in .7.2.2
FI 005	3		ed	...sodium tetrahydroborate... Please check the whole text regarding this reagent.	...sodium borohydride...	No, tetrahydroborate.
SK 006	3	1st para	te	(arsine, AsH ₃)	According to the IUPAC terminology –arsane AsH ₃	Arsine OK
SK 007	4		te		The concentration of arsenic which is not influenced by these interferants listed in Table 1 and Table 2 is not given in the text. Please give this concentration. Does the test solution referred to in Table 1 and Table 2 contain individual elements or is a mixture of the elements listed in these tables? Please specify.	Check with Ulrich. Must be done???
CZ 008	5.4		ed	Instead of „p(HCl) = 1 mol/l “ should be „c(HCl) = 1 mol/l”.		OK
FI 009	5.5		ed	Is it necessary to mention sodium tetrahydroborate (sodium borohydride) in an own clause? E.g. in clause 5.9. potassium iodide and ascorbic acid are mentioned only in this clause. The matter "Available as pellets, too." could be transferred into clause 5.7 as a note.		See As method.

Template for comments and secretariat observations					Date:2006-09-21	Document: ISO/DIS 17378-2
REVISED 2009-08-13						
1	2	(3)	4	5	(6)	(7)
MB ¹	Clause No./ Subclause No./ Annex (e.g. 3.1)	Paragraph/ Figure/ Table/ Note (e.g. Table 1)	Type of comment ²	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
SK 010	5.7		te		Please give reference to sodium tetrahydroborate: (5.5)	OK
CZ 011	5.7		ed	Instead of „C(HNO ₃) = 650 kg/g and C(HNO ₃) = 690 kg/g“ should be „w(HNO ₃) = 650 kg/g and w(HNO ₃) = 690 kg/g“.		Accept.
CZ 012	5.9		ed	Reference to Note 3 in (5.7) is introduced but the note is missing.		Not in my copy.
FI 013	5.9		ed	Please add the chemical formulas of potassium iodide and ascorbic acid.		Not necessary it is not in AFS method
FI 014	5.9		ed	...Prepare freshly each day...	...Prepare on the day of use...	OK accept.
FI 015	5.9		ed	"(See Note 3 in (5.7))." In 5.7 there is not Note 3.		There is in mine. Not in mine version
SK 016	5.9		te	Potassium iodide–ascorbic acid solution	Please give potassium iodide and ascorbic acid as individual chemicals/reagents (to keep the same format as with sodium tetrahydrogenborate and sodium hydroxide)	Accept.
SK 017	5.9	1 st para	ed	Prepare freshly each day.	Prepare fresh on the day of use.	See above.
SK 018	5.11		te		Please specify oxidation state of As in the heading and first line	Accept. Where is 5.11 ???
SK 019	5.11		te		We highly recommend amending this part. We suggest that As(V) be used for the preparation of As standard and calibration solutions. The recovery of As(III) from As(V) due to pre-reduction can be check by a suitable certified reference material.	? Where is 5.11 ???
CZ 020	5.11.1	Paragraph 3	ed	Instead of “1,7343 g ± 0,002 g” would be better “1,7343 g ± 0,0020 g”.		Accept.
CZ 021	5.11.1	Paragraph 4	ed	Please delete “potassium”.		Accept.

Template for comments and secretariat observations					Date:2006-09-21	Document: ISO/DIS 17378-2
REVISED 2009-08-13						
1	2	(3)	4	5	(6)	(7)
MB ¹	Clause No./ Subclause No./ Annex (e.g. 3.1)	Paragraph/ Figure/ Table/ Note (e.g. Table 1)	Type of comment ²	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
IR 022	5.11.1	title	Ed	The sentence should be completed as:	P(AS) = 1 000 mg/l changes to P(As(111))=...	done
IT 023	5.11.1		te	Arsenic stock solution of 1000 +/- 2 mg/l is reported. Other solution are available on the market with different uncertainty values (may be 3 or 5 mg/l).	In our opinion it should be possible to use these solutions taking into consideration the uncertainty values for the calculation of the overall method uncertainty.	See Se standard.
SK 024	5.11.1	4 th para	te	potassium sodium metaarsinate	Please delete potassium in potassium sodium metaarsinate in last but one paragraph	Accept.
SK 025	5.11.2	1 st para	te	This solution shall be prepared weekly.	This solution is stable for a week.	Accept.
FI 026	5.11.2 and 5.11.3		ed	...This solution shall be prepared weekly...	...This solution is stable for one week...	Accept.
FI 027	5.11.4		te	There is no reference to this reagent in the procedure of the standard. If it is not obligatory to check quantitative recovery of arsenic(V) in connection with every analytical cycle, please transfer clause 5.11.4 and instructions for its use into an informative annex.		It is necessary to check the prereduction if one suspects As(V)present in samples. Prereduction As(V)→As(III)
FI 028	6		ed	Apparatus and instrumentation (See ISO 78-2, clause 5.)	Apparatus	Accept.
SK 029	6.1.1	1 st para	ed	hollow cathode lamp or electrodeless discharge lamps	hollow cathode lamp or electrodeless discharge lamp	Drop the "s".
SK 030	6.2	4 th para	te	Nitrogen gas may also be used but will have a reduced sensitivity.	Nitrogen gas may also be used but will result in reduced sensitivity.	Not true only for AFS.
CZ 031	6.3.2		ed	Instead of "ethylene-propylene" would be better "ethene-propene".		no
SK 032	6.3.2	1 st para	ed	plastics	plastic	OK

Template for comments and secretariat observations					Date:2006-09-21	Document: ISO/DIS 17378-2
REVISED 2009-08-13						
1	2	(3)	4	5	(6)	(7)
MB ¹	Clause No./ Subclause No./ Annex (e.g. 3.1)	Paragraph/ Figure/ Table/ Note (e.g. Table 1)	Type of comm ent ²	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
SK 033	6.3.3	1 st para	te	via a peristaltic	Please add pump after via a peristaltic	OK
SK 034	6.3.3	1 st para	ed	or desorbs	nor desorbs	OK
SK 035	6.4.2	1 st para	ed	a top-pan balance	Please add for the after a top-pan balance	OK
FI 036	7.2.1	Paragraph 2	ed	"For greater accuracy ... In this case calculate..." These two sentences could be written as a note.		OK
FI 037	9.1 and 9.2		te	"Measure the absorbance of each solution at least twice." Is it really obligatory to measure every sample at least twice? In our opinion it is too much if not the software and sampler of the device makes that automatically.		Yes agreed in Japan.
IT 038	9.2		ed	calculation according to 10.2 and not to 10.1		OK
IT 039	9.3		ed	calculation according to 10.3 and not to 10.2		OK
** 040	10.2	Equation (1) and where clause under it	ed	In equations, single symbols with subscripts are used. (See Example 5 under 6.6.10.1 of the ISO/IEC Directives Part 2, 2004.) For the concentration of arsenic, this would give ρ_{As}		OK
FI 041	10.2		ed	ρ = [As]... Please write the symbols in equation (1) with italic letters.	$\rho[As]$ = ...	OK
IR 042	10.2		Ed	The sentence should be completed as:	P=[As] changes to P[As]=.....	OK
IT 043	10.2		te	in the formula the first term (between brackets) on the right should be moved to the left.		OK

Template for comments and secretariat observations					Date:2006-09-21	Document: ISO/DIS 17378-2
REVISED 2009-08-13						
1	2	(3)	4	5	(6)	(7)
MB ¹	Clause No./ Subclause No./ Annex (e.g. 3.1)	Paragraph/ Figure/ Table/ Note (e.g. Table 1)	Type of comment ²	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
SK 044	10.2 equation 1 and the explanations of symbols used	2 nd para	te		Please change the position of the symbol of equivalence (=) and replace symbol F with A (A for absorbance)	OK
FI 045	11		ed	... two significant places.	... two significant figures.	OK
** 046	A.4		ed	Please add a disclaimer footnote. (See 6.6.3 of the ISO/IEC Directives Part 2, 2004.)		Not necessary since only supplier.
CZ 047	C.2	line 3	te	Instead of "arsenic dihydride (AsH ₂)" should be "arsenic trihydride (AsH ₃)".		Agreed.
CZ 048	C.6.1	Paragraph 2, Line 4	ed	Instead of "0,05 µg/ml" should be "0,05 µg/l".		OK
CZ 049	D.1	Table D.1	te	The results are the same as in table D.1 of ISO/DIS 17379-2.		Same as Se – Wrong data!
SK 050	Annex A	A.1	te	high purity nitric acid	high purity hydrochloric acid	No
SK 051	Annex A	A.4	ge	A suitable system is available from Perma Pure Inc. 8 Executive Drive, PO Box 2105, Toms River, New Jersey 08754, USA.	Is it appropriate (not discriminative to other producers) to give a full reference of a particular company? We suggest that this paragraph be modified: Suitable dryer systems are commercially available which can alleviate this potential problem.	OK
FI 052	Annex B	Figure B.1	ed	"Key: 11 and 12 ... a Nafion dryer system..." Please delete the commercial name and give general requirements of a system. Please connect the separate sentence "Can use argon or nitrogen" into point 9 of key. Please transfer the sentence "This is only an example..." as a note above the figure designation and title.		No

Template for comments and secretariat observations					Date:2006-09-21	Document: ISO/DIS 17378-2
REVISED 2009-08-13						
1	2	(3)	4	5	(6)	(7)
MB ¹	Clause No./ Subclause No./ Annex (e.g. 3.1)	Paragraph/ Figure/ Table/ Note (e.g. Table 1)	Type of comment ²	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
SK 053	Annex B	Key	te		Please add pump 3	OK
SK 054	Annex B	2 nd para	ed	Can use argon or nitrogen	Argon or nitrogen can be used.	OK
SK 055	Annex B	Note, 2 nd para	ed	Peak rises to maximum as sample introduced....	Peak rises to its maximum as sample is introduced....	OK
IR 056	Annex C	C.2	Ed	The sentence should be completed as:	A5H2 changes to A5H2	Do not understand.
IR 057	Annex C	C.5	ed	The sentence is not completed and need more explanations.	It would be better to write more details about activation of graphite tube with iridium Chloride Solution, because usually there is not any manufacturers' recommendation in the manual of instruction.	Described already.
SK 058	Annex C	C.1	te		The expression of concentration units should be consistent (in accordance with p.10, 11Expression of results)- in µg/l As	No
SK 059	Annex C	C.2	te	arsenic dihydride (AsH ₂)	arsenic trihydride (AsH ₃)	No it's correct in my copy.
SK 060	Annex C	C.6.1, 2 nd para	te		Second paragraph, last line.... detection limits below 0,05 µg/ml (0,05 ppm =50 ppb) is in controversy with the statement on p.14, C.1 ..the lower limit of the working range is 0,02 µg/l (0,02 ppb = 20 ppt) within the scope of this standard. The concentration of selenium in the blank solution shall not exceed 10 ng/l should be 0,01 µg/l) The expression of concentration units should be consistent (in accordance with p.10, 11Expression of results)	Put phrase at beginning to say equal to or exceeds the value Must be done by you Peter. In my opinion do we need this annexe C ? How is carrying out this method nowadays ??
SK 061	Annex D	Table D.1	te		The data in Table D.1 are identical with the ones given in ISO/DIS 17379-2 (Determination of Se by HG-AAS, p. 16, Annex D, Table D.1)	Oh dear!!

Template for comments and secretariat observations	Date:2006-09-21	Document: ISO/DIS 17378-2
---	-----------------	----------------------------------

REVISED 2009-08-13

1	2	(3)	4	5	(6)	(7)
MB¹	Clause No./ Subclause No./ Annex (e.g. 3.1)	Paragraph/ Figure/ Table/ Note (e.g. Table 1)	Type of comm ent²	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
FI 062	Annex A, A.4		ed	Proprietary trade names should as far as possible be avoided. See ISO/IEC Directives, Part 2, 2004, clause 6.6.3.		Acceptable in this case.