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 1068** **ISO/TC 147/SC 2/WG 52** **N 0011**
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 17378-1 "Water quality - Determination of arsenic - Part 1: Method using hydride generation atomic fluorescence spectrometry (HG-AFS)"**

French title (if available): **ISO 17378-1 "Qualité de l'eau — Dosage de l'arsenic — Partie 1: Méthode par spectrométrie de fluorescence atomique à production d'hydrure (PA-SFA)**

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 0,02 µg/l to 100 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 should be less than the lower level of interest.

Concerns known patented items (see ISO/IEC Directives Part 1 for important guidance)

☐ Yes ☒ No If "Yes", provide full information as annex

Envisaged publication type (indicate one of the following, if possible)

☒ International Standard ☐ Technical Specification ☐ Publicly Available Specification ☐ 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 ☐ 1 (24 months) ☐ 2 (36 months - default) ☒ 3 (48 months)

Relevant documents to be considered

Relationship of project to activities of other international bodies

Liaison organizations	**Need for coordination with:**
	☐ IEC ☐ CEN ☐ Other (please specify)

Preparatory work (at a minimum an outline should be included with the proposal)

☒ A draft is attached ☐ 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 ☒ Yes ☐ No

Proposed Project Leader (name and address)

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Name and signature of the Proposer
(include contact information)

Prof. Dr. Peter B Stockwell

Comments of the TC or SC Secretariat

Supplementary information relating to the proposal

☐ This proposal relates to a new ISO document;

☐ This proposal relates to the amendment/revision of an existing ISO document;

☐ This proposal relates to the adoption as an active project of an item currently registered as a Preliminary Work Item;

☒ 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:

☐ Adoption of the proposal as a new project

☒ Adoption of the associated draft as a committee draft (CD) (see ISO Form 5, question 2.3.1)

☐ 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)

☒ **ISO/WD 17378-1 and Revised Secretariat Observations on former ISO/CD**

Date of circulation	Closing date for voting	Signature of the TC or SC Secretary
2009-08-28	**2009-12-01**	**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 1068

Date: 2009-08-27

ISO/WD 17378-1

ISO/TC 147/SC 2/WG 52 N 0011

Secretariat: DIN

Water quality — Determination of arsenic — Part 1: Method using hydride generation atomic fluorescence spectrometry (HG-AFS)

Qualité de l'eau — Dosage de l'arsenic — Partie 1: Méthode par spectrométrie de fluorescence atomique à production d'hydrure (PA-SFA)

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

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Contents

Page

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 17378-1 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

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 should 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 1: Method using hydride generation atomic fluorescence spectrometry (HG-AFS)

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 0,02 µg/l to 100 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 should 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 15587-1, *Water quality — Digestion for the determination of elements in water — Part 1: Aqua regia digestion*

3 Principle

An aliquot of sample is digested with hydrochloric acid (5.3). Potassium iodide-ascorbic acid reagent (5.9) 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 hydride (AsH_3). The hydride and excess hydrogen are swept out of the generation vessel using a stream of argon into a chemically generated hydrogen diffusion flame. The hydride is atomised and the resulting atoms excited by an intense arsenic light source, the resulting fluorescence is detected by atomic fluorescence spectrometry after isolation by an interference filter that transmits the arsenic resonance line at 193,76 nm. The procedure is automated by means of auto-sampler and control software.

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 should not be significant. The user should 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 should be assessed by performing spike recoveries;. However, the atomic fluorescence technique has a high linear dynamic range and a very low detection limit. In most cases any interferences can be removed by a simple dilution step.

The reaction conditions set out in this standard have been chosen so that any interferences are reduced to a minimum.

It is important that the light source does not contain any significant amount of other hydride forming elements (e.g. antimony) that will emit fluorescent radiation over the band pass of the interference filter used in the detector, if these elements are present in the sample.

Measurements carried out using the procedures in this standard will not suffer from interferences due to quenching.

Interference studies on a number of elements have been conducted and are shown in Table 1. It can be seen that easily reducible elements such as gold cause a significant negative bias. However, this element is unlikely to be present at the tested levels in the vast majority of water samples.

Interferences can be indicated by the irregularity of the signal peak shape. Normally the interference can be removed by diluting the samples.

Table 1 — Interference study for arsenic

Interfering substance		Concentration of interfering substance	As recovery	
		mg/l	2 $\mu g l^{-1}$ As	10 $\mu g l^{-1}$ As
Thallium nitrate	Tl(III)	20	94,8 ± 0,9	89,9 ± 2,8
Strontium nitrate	Sr(II)	20	107,3 ± 5,9	100,0 ± 2,5
Zinc nitrate	Zn(II)	1	101,7 ± 5,5	91,0 ± 3,1
Di-ammonium Silicon hexafluoride	Si(IV)	1	94,4 ± 3,7	102,4 ± 1,7
Aluminium nitrate	Al(III)	1	104,6 ± 0,8	98,9 ± 0,8
Calcium chloride	Ca(II)	200	101,8 ± 1,3	103,3 ± 1,3
Sodium chloride	Na(I)	200	104,2 ± 1,6	100,6 ± 1,8
Potassium bromide	K(I)	200	96,3 ± 0,7	97,6 ± 0,7
Indium nitrate	In(III)	1	99,4 ± 1,4	99,1 ± 1,5
Barium nitrate	Ba(II)	1	95,2 ± 3,1	105,9 ± 1,4
Magnesium oxide	Mg(II)	1	99,7 ± 3,5	97,2 ± 1,5
Cadmium nitrate	Cd(II)	1	100,4 ± 0,9	97,2 ± 0,2
Ammonium di-hydrogen phosphate	P(V)	1	100,3 ± 1,3	100,5 ± 2,1
Sodium fluoride	F(I)	1	113,3 ± 2,6	109,4 ± 1,0
Gold chloride	Au(III)	0,1	97,8 ± 6,4	103,2 ± 1,4
Gold chloride	Au(III)	1	80,9 ± 1,9	93,8 ± 1,5
Ortho-boric acid	B(III)	1	99,5 ± 3,0	99,7 ± 3,4
Iron(II) nitrate	Fe(II)	1	99,0 ± 0,9	100,2 ± 0,7
Lead(II) nitrate	Pb(II)	1	87,0 ± 4,1	95,3 ± 0,7
Bismuth nitrate	Bi(III)	1	121,4 ± 0,9	107,0 ± 0,2
Tin nitrate	Sn(IV)	1	95,1 ± 1,9	104,8 ± 1,8
Germanium chloride	Ge(IV)	1	104,4 ± 3,0	102,1 ± 1,1
Mercury	Hg(II)	1	100,7 ± 0,7	98,1 ± 0,4
Chromium(III) nitrate	Cr(III)	1	101,0 ± 1,2	98,4 ± 0,6
Cobalt nitrate	Co(II)	1	103,1 ± 0,7	99,9 ± 2,0
Silver nitrate	Ag(I)	1	97,9 ± 1,8	95,9 ± 2,3
Nickel(II) nitrate	Ni(II)	1	100,2 ± 0,4	98,8 ± 1,2
Telluric acid	Te(IV)	0,01	90,7 ± 2,9	99,3 ± 0,7
Telluric acid	Te(IV)	0,1	100,1 ± 1,2	98,0 ± 0,9
Telluric acid	Te(IV)	1	101,5 ± 0,6	100,3 ± 1,0
Antimony oxide	Sb(III)	0,01	101,0 ± 0,8	102,1 ± 1,1
Antimony oxide	Sb(III)	0,05	107,3 ± 1,8	97,5 ± 1,7
Antimony oxide	Sb(III)	0,1	118,3 ± 1,0	96,7 ± 2,7
Copper(II) sulfate	Cu(II)	0,1	101,8 ± 1,9	102,7 ± 2,4
Copper(II) sulfate	Cu(II)	0,2	100,5 ± 2,8	101,4 ± 0,1
Copper(II) sulfate	Cu(II)	0,5	99,8 ± 1,1	99,0 ± 1,0
Copper(II) sulfate	Cu(II)	1	94,0 ± 4,7	100,7 ± 1,9
Copper(II) sulfate	Cu(II)	2	98,8 ± 1,1	99,0 ± 1,0
Iron(II) nitrate	Fe(III)	200	114,3 ± 0,7	105,0 ± 0,6

5 Reagents and standards

5.1 General requirements

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

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

5.3 Hydrochloric acid, HCl ρ(HCl) = 1,16 g/ml.

5.4 Hydrochloric acid, ρ(HCl) = 1 mol/l.

5.5 Sodium tetrahydroborate, $NaBH_4$.

Available as pellets – shelf life of maximum of six months.

5.6 Sodium hydroxide, NaOH.

5.7 Sodium tetrahydroborate solution, $\rho(NaBH_4)$ = 13 g/l.

Dissolve (13,0 ± 0,1) g of sodium tetrahydroborate in 500 ml water (5.2) and add (4,0 ± 0,1) g of sodium hydroxide (5.6). Dilute to 1 000 ml with water (5.2). Filter the solution through a 0,45 µm 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 6 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 See Clause 8.

NOTE 3 Alternatively smaller volumes can be prepared on a pro rata basis.

5.8 Nitric acid, $\rho(HNO_3)$ = 1,40 g/ml.

NOTE Nitric acid is available both as $\rho(HNO_3)$ = 1,40 g/ml ($\rho(HNO_3)$ = 650 g/kg) and $\rho(HNO_3)$ = 1,42 g/ml ($\rho(HNO_3)$ = 690 g/kg).

5.8.1 Nitric acid cleaning mixture

Dilute nitric acid (5.8) with an equal volume of water (5.2) by carefully adding the acid to the water.

5.9 Potassium iodide-ascorbic acid solution

Dissolve (250 ± 0,1) g of potassium iodide and (50 ± 0,1) g of ascorbic acid in approximately 400 ml water (5.2) and dilute to 500 ml. Prepare freshly each day (see Note 3 in 5.7).

5.10 Reagent blank

For each 1 000 ml, prepare a solution containing 300 ml ± 3 ml of hydrochloric acid (5.3) and 20 ml ± 0,5 ml of potassium iodide-ascorbic acid solution (5.9). 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 levels of detectable amounts of arsenic it is important that the same reagents are used for both sample and standard preparation as well as for 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.

5.11 Arsenic standard solutions

5.11.1 Arsenic stock solution A, ρ(As) = 1 000 mg/l.

Use a quantitative stock solution with a traceable arsenic(III) content of (1 000 ± 2) mg/l. This solution is considered to be stable for at least one year.

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

Place 1,7343 g ± 0,002 g of sodium metaarsenite $NaAsO_2$ in a 1 000 ml volumetric flask.

Add 50 ml ± 0,5 ml of hydrochloric acid (5.3) and dissolve the sodium metaarsenite completely by stirring.

Dilute to 1 l with water (5.2).

5.11.2 Arsenic standard solution B, ρ(As(III)) = 10 mg/l.

Pipette 1 ml ± 0,01 ml of arsenic stock solution A (5.11.1) into a 100 ml volumetric flask, add 30 ml ± 0,5 ml of hydrochloric acid (5.3) and 2 ml ± 0,1 ml of potassium iodide-ascorbic acid solution (5.9) and fill up to the mark with water (5.2). This solution should be prepared weekly.

5.11.3 Arsenic standard solution C, ρ(As(III)) = 100 µg/l.

Pipette 1 ml ± 0,01 ml of arsenic standard solution B (5.11.2) into a 100 ml volumetric flask, add 30 ml ± 0,5 ml of hydrochloric acid (5.3) and 2 ml ± 0,1 ml of potassium iodide-ascorbic acid solution (5.9) and fill up to the mark with water (5.2). This solution should be prepared on the day of use.

5.11.4 Arsenic standard solution D, ρ(As(III)) = 10 µg/l.

Pipette 10 ml ± 0,1 ml of arsenic standard solution C (5.11.3) into a 100 ml borosilicate volumetric flask. Fill up to the mark with reagent blank solution (5.10). This solution should be prepared freshly on the day of use.

5.11.5 Arsenic standard solution E, ρ(As(V)) = 1 000 mg/l.

Dissolve 1,000 g ± 0,002 g of pure arsenic powder in 10 ml ± 0,1 ml of concentrated nitric acid (5.8).

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

Cool and then take up the hydrated arsenic(V) oxide in 50 ml ± 0,5 ml of cold hydrochloric acid (5.3).

Transfer the solution quantitatively to a 1 000 ml volumetric flask and fill up to the mark with water (5.2).

This standard should be used to prepare a suitable arsenic(V) standard to check quantitative recovery of arsenic(V).

The solution is stable for at least six months.

5.11.6 Arsenic calibration solutions

A minimum of five independent calibration solutions shall be used. The calibration is described in ISO 8466-1. The calibration solutions are prepared by suitable dilution of the arsenic standard C (5.11.3) or D (5.11.4).

Each calibration solution shall contain 30 ml ± 0,5 ml of hydrochloric acid (5.3) and 2 ml ± 0,01 ml of potassium iodide-ascorbic acid solution (5.9) per 100 ml in borosilicate volumetric flasks. Prepare on day of use.

For example, for the concentration range from 0,1 µg/l to 1 µg/l, proceed as follows:

Pipette into a series of five 100 ml volumetric flasks 2 ml ± 0,02 ml, 4 ml ± 0,04 ml, 6 ml ± 0,06 ml, 8 ml ± 0,08 ml and 10 ml ± 0,1 ml respectively of arsenic standard solution D (5.11.4).

Fill up to the mark with reagent blank solution (5.10) 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 0,2 µg/l, 0,4 µg/l, 0,6 µg/l, 0,8 µg/l and 1 µg/l arsenic respectively. They should 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

6.1 General

Atomic fluorescence systems should be set up to the manufacturer's recommendations. The following example shows a typical example of a system specific for these measurements.

6.2 Atomic fluorescence system

A schematic block diagram of an example of an automated arsenic analysis system is shown in Annex B. This consists of

6.2.1 Auto-sampler, where operated in an automatic regime.

6.2.2 Appropriate interference filter

6.2.3 Continuous flow vapour generator

6.2.4 Gas liquid separator, a moisture removal system.

6.2.5 Atomic fluorescence spectrometer, with a control computer.

6.2.6 Appropriate calculation and reporting software

A typical signal response is also shown in Annex B.

The background level is the summation of the instrumental blank, the reagent blank and the flame blank. Additional background levels may be contributed if a mixture of argon and hydrogen is used. See Annex B which illustrates the typical signal response from a continuous flow vapour generator atomic fluorescence system.

6.3 Gas supply

Use argon with a grade specified by the manufacturer.

The gas supply should 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 result in a significantly reduced sensitivity.

Compressed air from a cylinder or oil free compressor can be used as the dryer gas.

6.4 Moisture removal

Moisture removal is provided using a Nafion hygroscopic membrane which continuously removes moisture present. Details of a suitable unit are provided in Annex B. Air, argon or nitrogen can be used as the dryer gas.

6.5 Laboratory ware

6.5.1 General requirements

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 (5.8.1) for at least 24 h and rinsed five times with water (5.2).

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

6.5.2 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.

6.5.3 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 absorbs or desorbs the analyte under test.

6.5.4 Auto-sampler vials

Use vials constructed of materials specified in (6.5.2).

6.6 Sample processing equipment

6.6.1 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.

6.6.2 Balances

Analytical balance, capable of accurately weighing (standards) to ± 0,1 mg; and a top-pan balance, preparation of solutions, accurate to ± 0,1 g.

7 Sampling and sample preparation

7.1 Sampling techniques

Carry out the sampling as specified in ISO 5667-1, ISO 5667-2 and ISO 5667-3, using sampling vessels as specified in (6.5.2).

For the determination of arsenic in aqueous samples, acidify at time of sampling to pH < 2. Hydrochloric acid (5.3) 3 ml ± 0,5 ml per litre is sufficient for most samples. Ensure that the pH is less than 2; otherwise, add more hydrochloric acid as required.

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

A continuous flow procedure is used for this standard accordingly an appropriate blank must be prepared and analysed as required. All samples, blanks and standards must be prepared in the same matrix: i.e. matrix matched.

NOTE Sample preservation using nitric acid (5.8) may be suitable providing it is shown that the arsenic determinations are unaffected using this reagent.

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

Pre-treat water samples, 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 container.

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

Add 2 ml ± 0,1 ml of potassium iodide-ascorbic acid solution (5.9), 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.

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 (5.8) and hydrochloric acid (5.4). Blanks and standard solutions must also be matrix matched.

8 Instrumental set up

Configure the instrumentation as described in the instrument manufacturer's manual. An example of the configuration is given at Annex B.

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.

Fill the reagent reservoirs with reagent blank solution (5.10) and sodium tetrahydroborate solution (5.7) respectively.

Set up the continuous flow vapour generator system according to the manufacturer's recommendations. Ensure that reagent flows are within the accepted tolerances and that the hydrogen flame has ignited. Once stable conditions are established analysis can proceed.

Where the manufacturer's instrument uses a hydrogen flame the reagent (5.7) has a two fold function a) to reduce the arsenic to its hydride and b) to generate hydrogen for the atomisation source. The later may require that the concentration of $NaBH_4$ (5.7) will need to be optimised to suit the pumping and gas flow rates used on the instrumentation and to obtain noise levels consistent with the detection levels required by this standard.

Both standards and samples shall be quantified using the same flow characteristics.

Turn on the argon (6.3) to provide carrier gas. A suitable dryer (moisture removal) system shall be used (6.4). Turn on the dryer gas (6.3). Flow rates shall be set according to the instrument manufacturer's recommendations.

Select the required amplification for the atomic fluorescence detector. Ensure that the selected detector range is appropriate to the sample concentration being determined.

For samples which are above the calibration for a given range setting, either reanalyse at a lower sensitivity or dilute the sample into the calibration range. If the sample is diluted, then the diluent shall be the reagent blank solution (5.10), i.e. matrix matched.

Samples which are digested shall be matrix matched against standards and blanks using the same acid concentrations to provide reliable data.

9 Procedure

Follow the manufacturer's instructions to set up instrumental conditions and software procedures to establish quantitative analysis.

With the reagent blank (5.10) and the sodium tetrahydroborate solution (5.7) flowing to the gas liquid separator, ensure that the system is equilibrated by monitoring for a stable fluorescence detector background. If sufficient warm up time is not allowed the detector baseline can change during an analytical cycle.

Analyse calibration solutions (5.11.6), samples (see Clause 7) and blanks (5.10) sequentially in the manner required or else run automatically in the following manner:

Load the auto-sampler with the calibration solutions (5.11.6), samples (see Clause 7) and blanks (5.10) and start the auto-sampler programme. Analysis of a field blank within a sample run will establish whether contamination has occurred. Should significant level of contamination be established, the analytical results will be brought into question.

Inorganic arsenic occurs in two oxidation states; As(V) and As(III). It is essential to convert all arsenic species to the As(III) state prior to generating the hydrides. Arsenic(V) gives a significantly lower response than arsenic(III).

NOTE See Clause 4.

Prepare As(V) standards (5.11.5) at known concentrations and analyse after pre-reduction to validate the pre-reduction stage of this procedure.

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, ρ[As] in the samples using Equation (1):

$$\rho[As] = \frac{(F_s - F_b) \cdot V_M}{b \cdot V_P} \quad (1)$$

where

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

F_s is the fluorescence response of the water sample;

F_b is the fluorescence response of the blank solution;

b is the slope of the calibration curve and a measure of the sensitivity in litres per micrograms, l/µg;

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.

11 Expression of results

Report the results in µg/l and round them to the nearest 0, 01 µg/l. Do not use more than two significant places.

EXAMPLE

Arsenic(As) 0,04 µg/l

Arsenic(As) 14 µg/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 International Standard (ISO/WD 17378-1);

b) complete identification of the laboratory;

c) experience of staff using analysers;

d) complete identification of the sample;

e) expression of results as indicated in Clause 11;

f) sample pre-treatment;

g) any deviations from this method and details of all circumstances which could have affected the result.

Annex A
(informative)

Additional information

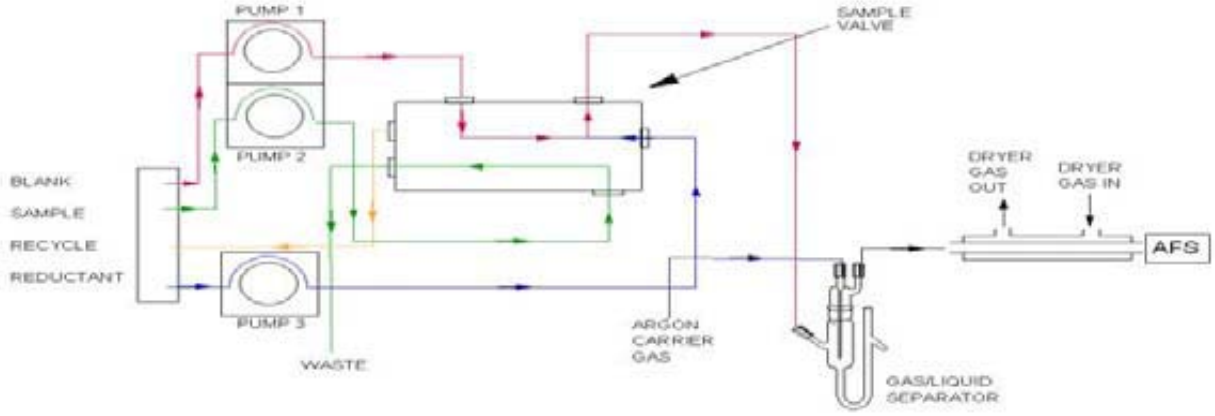
A.1 The method and any variation from it should be rigorously checked for performance using statistical data and analytical quality control sample materials, including certified reference materials.

A.2 Whilst any inert gas may be used to purge the arsenic from the gas/liquid separator, the optimum signal, response will be provided using argon. Nitrogen can be used but will quench the fluorescence signal reducing sensitivity. Air should not be used because of explosion risk.

A.3 Water vapour may also be removed using a desiccant tube. Care shall be taken using this approach to avoid trapping arsenic in the trap due to excess moisture retention.

Annex B
(informative)

Figures



Key

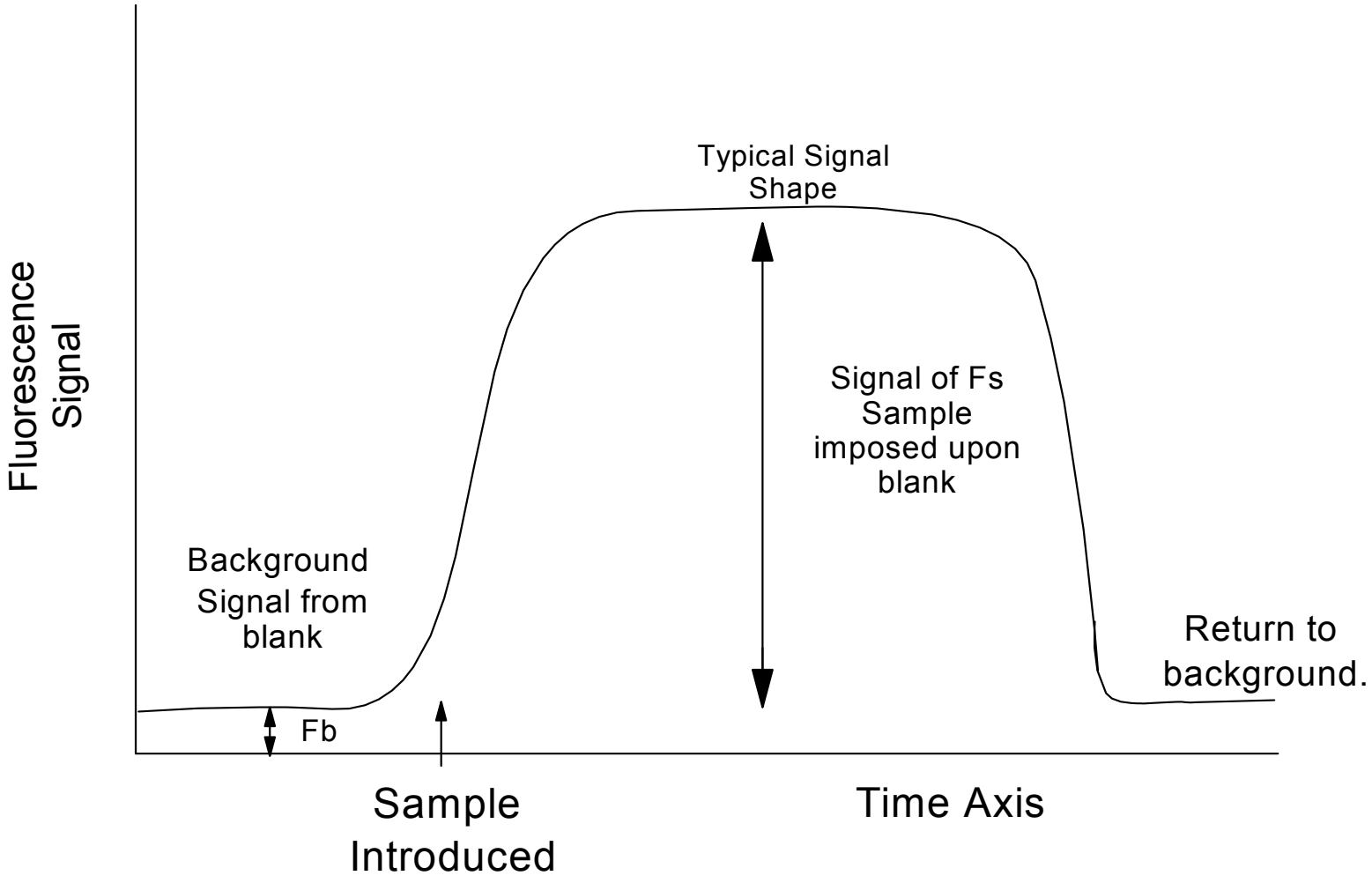
1	pump 1	8	sample valve
2	pump 2	9	waste
3	pump 3	10	argon carrier gas
4	blank	11	gas liquid separator
5	sample	12	dryer gas out from Nafion dryer system (6.3)
6	recycle	13	dryer gas in to Nafion dryer system (6.3)
7	reductant	14	atomic fluorescence spectrometer

NOTE 1 This continuous flow vapour generator consists of a constant speed peristaltic pump to deliver sodium tetrahydroborate(5.7), reagent blank and sample. A switching valve alternates between the reagent blank and sample or standard solutions. The vapour generator switches between reagent blank (5.10) 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 signal response is shown in Figure B.2.

NOTE 2 Nafion hygroscopic membranes for moisture removal are commercially available from Perma Pure Inc, 8 Executive Drive, PO Box 2105, Toms River, New Jersey 08754, USA.

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.



Peak rises to maximum as sample introduced and returns to baseline.
Matrix of sample/standards and blank **must** be matrix matched

Key

X = Time in seconds
Y = Fluorescence signal

Figure B.2 — Typical signal response from arsenic in water sample by hydride generation atomic fluorescence spectrometry

Explanation: Signal rises to a plateau as the sample is introduced and returns back to the baseline once the sample is replaced by the reagent blank (5.10).

The matrix of the samples; standards and blanks shall be matrix matched.

Annex C
(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 C.1 and the performance data are summarised in Table C.2.

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

Table C.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% to 60%) spike	As(III), Sb(III), Se(IV)
4	Real sample 2[a] (hard water) + high-range spike (70% to 90%)	As(V), Sb(V), Se(VI)
5	Real sample 3[a] (intermediate hardness water) + low-range (10% to 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 C.2.

Table C.2 — Performance data

Sample	Matrix	l	n	n_{OP}	$\bar{\bar{X}}$	x_{ass}	η	s_R	CV_R	s_r	CV_r
				%	µg/l	µg/l	%	µg/l	%	µg/l	%
1	Nutwell hard water 90% spike	9	21	0,0	19,0	18,0	105,8	1,18	6,2	0,56	2,9
2	Rotherham interm. water 15%	9	21	0,0	2,90	3,0	96,7	0,333	11,5	0,130	4,5
3	Bradfort soft water 60% spike	9	21	0,0	11,9	12,0	98,8	0,68	5,7	0,34	2,9
4	Low standard 20%	9	21	0,0	3,95	4,0	98,9	0,405	10,2	0,163	4,1
5	Blank	-	-	-	-	-	-	-	-	-	-
6	High standard 80%	9	21	0,0	15,7	16,0	98,3	1,31	8,3	0,79	5,0
7	Blank	-	-	-	-	-	-	-	-	-	-
8	Rotherham interm. water 15%	9	21	0,0	2,94	3,0	97,9	0,375	12,8	0,165	5,6
9	Low standard 20%	9	21	0,0	3,88	4,0	97,1	0,389	10,0	0,147	3,8
10	High standard 80%	9	21	0,0	15,5	16,0	97,0	1,61	10,4	0,31	2,0
11	Nutwell hard water 90% spike	9	21	0,0	18,9	18,0	105,0	0,97	5,1	0,33	1,8
12	Bradfort soft water 60% spike	9	21	0,0	11,9	12,0	99,2	0,92	7,7	0,28	2,3
13	Standard 30 µg/l	7	17	15,0	29,4	30,0	98,0	2,04	6,9	0,72	2,5

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;
$\bar{\bar{X}}$	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.

Template for comments and secretariat observations

Date: 2005-03-17	Document: **ISO/CD 17378-1**

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 com-ment**[2]	**Comment (justification for change) by the MB**	**Proposed change by the MB**	**Secretariat observations** on each comment submitted
FI	Whole doc			We object to six separate standards for the determination of Sb, As and Se. These should be combined to two standards (HG-AFS and HG-AAS) because their content is equal.		Not accepted by WG52
FR	5.11.2	Arsenic standard solution B	te	Correct requirement of +/- 0,01 ml on 2 ml of potassium iodide-ascorbic acid solution (5.9)	According to 17378-2 we should have the same requirement of +/- of 0.1 ml on 2 ml of this reagent.	
FR	5.11.3	Arsenic standard solution C	te	Correct requirement of +/- 0,01 ml on 2 ml of potassium iodide-ascorbic acid solution (5.9)	According to 17378-2 we should have the same requirement of +/- of 0.1 ml on 2 ml of this reagent.	
FR	7.1	Sampling techniques	te	Add a note about sample preservation with nitric acid in this paragraph	According to ISO 15586 and ISO 17294-2 sample preservation can be carried out following the same procedure knowing that this element is determined by these standards.	Additional sentences added.
FR	7.1	Sampling techniques	te	The sentence: For all type of samples……used in the sample.	The meaning of these blanks should be explained. Is it field blank, acid blank or sampling vessel blank? It may be not necessary for all types of samples, it depends of the aim of the measurement and of the level of the relevant element.	Additional sentences added.
FR	8	Instrumental set up	ed	Turn on the dryer gas (6.5)	Correct (6.5) to 6.2).	OK
FR	10.2	Calculation using the calibration curve	te	Slope is expressed in nanogram and As concentration is expressed in microgram, there is a problem of copy and paste between documents.	According to 17378-2 there is a mistake in the unit expression, all concentration should be expressed in µg/l as the calibration solutions	Changed.
FR	11	Expression of results	te	Report the results in µg/l and round them to the nearest 0,001 µg/l	Regarding the linear application range defined in the scope (0,02 µg/l to 100 µg/l) the nearest 0,01 should be acceptable for this method.	Accept.

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2 **Type of comment:** **ge** = general **te** = technical **ed** = editorial

NOTE Columns 1, 2, 4, 5 are compulsory.

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MB[1]	**Clause No./ Subclause No./ Annex** (e.g. 3.1)	**Paragraph/ Figure/Table/ Note** (e.g. Table 1)	**Type of com-ment**[2]	**Comment (justification for change) by the MB**	**Proposed change by the MB**	**Secretariat observations** on each comment submitted
FR	Annex C	Precision data	te	In Table C.1 waste water spiked sample is included but this type of water is not in the scope and in paragraph 7.2.2 it written that type of samples are outside the scope of the standard	This point should be clarified, otherwise this type of sample shall be removed from the table C.1	New set of samples for test. No test samples received
DE	Whole doc			We regret our negative vote. Whilst we appreciate the effort on the preparation of the single documents, we think it cannot be justified to set up 6 different standards which are so much alike. It might be possible to combine all determinations (hydride technique and fluorescence) within one single standard. We would, however, prefer the elaboration of two standards, one for fluorescence and one for hydride technique, each standard dealing with antimony, arsenic and selenium. The (already advanced) methods on antimony can be used as a basis and the information on arsenic and selenium my be added. It is regrettable that the single documents editorially differ so much from each other which makes them difficult to read. This means as well that the future method(s) need thorough checking in editorial respect. We are prepared to offer our help, if this is asked for. The offer includes as well the preparation of the absolutely essential interlab trial.		Not accepted by WG52
DE	General		te	The DIS cannot be accepted as long as interlab trial data are missing for validation of the method		Accepted
DE	3		ed	Correct the spelling of arsenic		OK
DE	4	Para 3	ed	Delete arsenic, insert antimony		OK
DE	5.10	NOTE	ed	Correct the spelling of arsenic		OK
DE	5.11.1	Para 3	ed	Correct the spelling of $NaAsO_2$		OK
DE	6.4.2	NOTE	ed	Correct the spelling of polytetrafluoroethene		OK
DE	6.4.3	1st line	ed	Insert “pump” after “peristaltic”		OK

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DE	6.5.2		ed	Delete the clause, it is obvious		OK
DE	7.2	1st line	ed	Correct the spelling of arsenic		OK
DE	7.2.2	Para 4	te	This para is not very helpful as it includes the need to look up 15587-1. It would be more helpful to give exact description in this place.		Outside scope anyway so what is problem?
DE	Table 1		ed	Correct the spelling – no capitals for the anions, sulfate instead of sulphate		OK
IT	6.4.4		Te	Not necessary		Don't agree.
IT	11		Te	Using the suggested calibration solutions it should be better to round the results to the nearest 0,01 µg/l and not to the 0,001 µg/l		Agreed.
IT	12		Te	Other information such as identification of the lab and so on must be added in the test report		OK
NO	Whole doc			Because the content of this standard is very similar to the ISO 23914-1 and ISO 17379-1, they should be combined to one standard.		Not accepted by WG52
RU	4	Table 1	ed	It is not clear whether concentration value (column "Concentration") corresponds with the ion form of an interfering substance or with the substance itself	Please specify to which form of the substance the concentration relates	OK
RU	4	Table 1	ed	Some of substances enlisted in the first column cannot exist in solutions, e.g. magnesium oxide, antimony oxide, mercury, tin nitrate. Form iron (II) nitrate is hardly ever obtainable because of redox reactions that might occur in this case	We suggest that the information should be presented as follows: "Copper (II) as Sulphate" etc	OK
RU	4	Table 1	ed	Some of ion form enlisted in the second column can exist in solutions on no occasion, e.g. Si^{4+}, Sn^{4+}, P^{3+}; existence of Sb^{3+}, Te^{4+} is a matter of discussion.	We suggest that in these cases symbols like Si(IV) etc appears more appropriate	OK

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RU	4	Table 1, line "Thallium nitrate"	ed	The ion form is "Tl^{3+}" not "Ti^{4+}"	Please correct	OK
RU	4	Table 1, line "Ammonium Di-hydrogen Phosphate"	ed	The ion form is "P(V)" not "P^{3+}"	Please correct	OK
RU	4	Table 1, lines "Gold Chloride"	ed	The ion form is "Au^{3+}" not "Au^{+}"	Please correct	OK
RU	4	Table 1, line "Iron (II) Nitrate"	ed	The ion form is "Fe^{2+}" not "Fe^{3+}"	Please correct	OK
RU	5.7	Note 2	ed	In our opinion this statement is not to the point here	Please transfer it into the clause 8 or 9 where appropriate	
RU	5.11.1	Paragraph 3	ed		Please replace "AS" by "As"	OK
RU	10.2	Explication to equation, Line "b"	ed	The slope of the calibration curve is to be expressed as litres per microgram	Please replace "... per nanogram, l/ng" by "... per microgram, l/μg"	OK
RU	Annex B	Figure B.2	ed	Concentration of arsenic is highly desirable	Please submit concentration of arsenic	??

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SE	Whole document		ge	Our opinion is that these two drafts, concerning the determination of arsenic, should be combined with ISO/CD 17379 "Determination of selenium" (part 1 & 2) and the NWIP concerning the determination of antimony (TC 147/SC 2 N 784 and N 785). The contents of these six proposed standards methods are so similar (large parts of the text are identical), that it would be most suitably if they could be merged together into only two standards with the following titles: *"Determination of antimony, arsenic and selenium using hydride generation atomic fluorescence spectrometry"* and *"Determination of antimony, arsenic and selenium using hydride generation atomic absorption spectrometry".*		Not accepted by the WG52
TR	7.2	1st line	ed	typo	"srsenic" should be "arsenic"	OK

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