BS ISO 17378-2:2014

BSI Standards Publication

Water quality — Determination of arsenic and antimony

Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)

bsi.

...making excellence a habit.™

Document Sharing Hub

Full document details: BS ISO 17378-2:2014

National foreword

This British Standard is the UK implementation of ISO 17378-2:2014. It supersedes BS EN ISO 11969:1996 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/3/2, Physical chemical and biochemical methods.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2014. Published by BSI Standards Limited 2014

ISBN 978 0 580 70964 7

ICS 13.060.50

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 28 February 2014.

Amendments issued since publication

Date	Text affected

Documents
Document Sharing Hub

Full document details: BS ISO 17378-2:2014

INTERNATIONAL STANDARD

ISO 17378-2

First edition
2014-02-01

Water quality — Determination of arsenic and antimony —

Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)

Qualité de l'eau — Dosage de l'arsenic et de l'antimoine —

Partie 2: Méthode par spectrométrie d'absorption atomique à génération d'hydrures (HG-AAS)

Reference number
ISO 17378-2:2014(E)

Documents
Document Sharing Hub

© ISO 2014

Full document details: BS ISO 17378-2:2014

COPYRIGHT PROTECTED DOCUMENT

© ISO 2014

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

© ISO 2014 – All rights reserved

Documents
Document Sharing Hub

Full document details: BS ISO 17378-2:2014

Contents

Page

Foreword ... **iv**
Introduction ... **v**
1 Scope ... **1**
2 Normative references ... **1**
3 Principle ... **2**
3.1 Arsenic ... 2
3.2 Antimony ... 2
4 Interferences ... **2**
4.1 General ... 2
4.2 Arsenic ... 3
4.3 Antimony ... 3
5 Reagents ... **3**
5.1 General requirements ... 3
6 Apparatus ... **8**
7 Sampling and sample preparation ... **9**
7.1 Sampling technique ... 9
7.2 Pre-reduction ... 9
8 Instrumental set up ... **10**
9 Procedure ... **10**
9.1 General requirements ... 10
9.2 Analysis using the method of standard calibration ... 11
9.3 Analysis using the standard addition method of calibration ... 11
10 Calibration and data analysis ... **12**
10.1 General requirements ... 12
10.2 Calculation using the calibration curve ... 12
10.3 Calculation using the standard addition method ... 13
11 Expression of results ... **13**
12 Test report ... **13**
Annex A (informative) **Additional information** ... **14**
Annex B (informative) **Schematic flow diagram and signal response** ... **15**
Annex C (informative) **Example of enrichment technique** ... **17**
Annex D (informative) **Performance data** ... **19**
Bibliography ... **22**

© ISO 2014 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 17378-2:2014

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This first edition of ISO 17378-2 cancels and replaces ISO 11969:1996, which has been technically revised.

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

— *Part 1: Method using hydride generation atomic fluorescence spectrometry (HG–AFS)*

— *Part 2: Method using hydride generation atomic absorption spectrometry (HG–AAS)*

© ISO 2014 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 17378-2:2014

Introduction

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

Arsenic concentrations in natural waters are highly variable, from <10 µg/l to as high as several milligrams per litre in some parts of Asia, South America and the USA, notable in the Ganges delta where arsenic poisoning from contaminated tube wells is a serious problem. Antimony concentrations in natural waters are generally well below 10 µg/l. Arsenic and antimony occur naturally in organic and inorganic compounds and may have oxidation states –III, 0, III and V.

In order to fully decompose all of the arsenic or antimony compounds, a digestion procedure is necessary. Digestion can only be omitted if it is certain that the arsenic or antimony 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.

The method for determining arsenic or antimony is identical in all aspects except for the preparation of standard solutions to be tested. To avoid repetition or duplication the text refers to both arsenic and antimony where the text is equally applicable to both instances. The subclause dealing with preparation of standard solutions is divided into 5.11.1, which deals with solutions of arsenic, and 5.11.2, which deals with solutions of antimony.

© ISO 2014 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 17378-2:2014

Documents
Document Sharing Hub

Full document details: BS ISO 17378-2:2014

INTERNATIONAL STANDARD ISO 17378-2:2014(E)

Water quality — Determination of arsenic and antimony —

Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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 document be carried out by suitably trained and experienced staff.

1 Scope

This part of ISO 17378 specifies a method for the determination of arsenic and antimony. The method is applicable to drinking water, surface water, ground water, and rain water. The approximate linear application range of this part of ISO 17378 for both elements is from 0,5 µg/l to 20 µg/l. Samples containing higher concentrations than the application range can be analysed following appropriate dilution.

Generally sea water is outside the scope of this part of ISO 17378. Sea water samples can be analysed using a standard additions approach providing that this is validated for the samples under test. The method is unlikely to detect organo-arsenic and organo-antimony compounds.

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

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 and sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 5667-5, *Water quality — Sampling — Part 5: Guidance on sampling of drinking water from treatment works and piped distribution systems*

ISO 5667-6, *Water quality — Sampling — Part 6: Guidance on sampling of rivers and streams*

ISO 5667-8, *Water quality — Sampling — Part 8: Guidance on the sampling of wet deposition*

ISO 5667-11, *Water quality — Sampling — Part 11: Guidance on sampling of groundwaters*

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 functions*

© ISO 2014 – All rights reserved

AVA Documents Document Sharing Hub

Full document details: BS ISO 17378-2:2014

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

3 Principle

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

3.1 Arsenic

An aliquot of sample is acidified with hydrochloric acid (5.3). Potassium iodide–ascorbic acid reagent (5.9) is added to ensure quantified reduction of arsenic(V) to arsenic(III). The sample solutions are treated with sodium tetrahydroborate (5.5) to generate the covalent gaseous arsenic trihydride (arsine, AsH_3). The hydride and excess hydrogen are swept out of the generation vessel in case of batch mode and out of the gas/liquid separator in case of the continuous mode into a heated silica cell. After atomization, the absorbance of arsenic is determined at a wavelength λ = 193,7 nm. The procedure is automated by means of auto-sampler and control software.

3.2 Antimony

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 antimony(V) to antimony(III). The sample solutions are then treated with sodium tetrahydroborate (5.5) to generate the covalent gaseous antimony trihydride (stibane, SbH_3). The hydride and excess hydrogen are swept out of the generation vessel in case of batch mode and out of the gas/liquid separator in case of continuous mode. After atomization, the absorbance of antimony is determined at a wavelength λ = 217,6 nm. The procedure is automated by means of auto-sampler and control software.

4 Interferences

4.1 General

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 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.

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 1 are exceeded, a significant decrease of absorption may occur.

Table 1 — Maximum mass concentration in test solution of interfering heavy metals (valid for flow systems)

Interfering element				
Cr	Fe	Cu	Ni	Pb
mg/l				
500	500	500	250	100

The reaction conditions in this part of ISO 17378 have been chosen to minimize these interferences.

Further information on these interferences and the technique are given in References.[1][2]

NOTE If batch systems are used, mass concentrations which are appreciably lower than those specified in Table 1, Table 2 and Table 3 can cause interferences.

© ISO 2014 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 17378-2:2014

4.2 Arsenic

Elements such as antimony, selenium, tellurium, mercury, and tin are also volatilized by this procedure and may cause interferences. These elements do not cause interferences providing the concentrations specified in Table 2 are not exceeded.

Table 2 — Maximum mass concentration of hydride-forming or volatile elements causing no interferences

Element				
Sb	Se	Sn	Te	Hg
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.

4.3 Antimony

Elements such as arsenic, selenium, tellurium, mercury, and tin are also volatilized by this procedure and may cause interferences. These elements do not cause interferences providing the concentrations specified in Table 3 are not exceeded.

Table 3 — Maximum mass concentration of hydride-forming or volatile elements causing no interferences

Element				
As	Se	Sn	Te	Hg
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 antimony content is high enough, an appropriate dilution of the water sample is preferred.

5 Reagents

5.1 General requirements

It is important to use high purity reagents in all cases with minimum levels of arsenic or antimony.

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

Use only reagents of recognized analytical grade, unless otherwise specified.

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

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

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

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

© ISO 2014 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 17378-2:2014