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**Kakovost vode - Določevanje arzena (III) in arzena (V) - Metoda tekočinske kromatografije visoke ločljivosti (HPLC) z masno spektrometrijo z induktivno sklopljeno plazmo (ICP/MS) ali atomsko fluorescenčno spektrometrijo s hidridno tehniko (HG/AFS)**

Water quality - Determination of arsenic(III) and arsenic(V) species - Method using high performance liquid chromatography (HPLC) with detection by inductively coupled plasma mass spectrometry (ICP-MS) or hydride generation atomic fluorescence spectrometry (HG-AFS)

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Qualité de l'eau - Détermination des formes chimiques (III) et (V) d'arsenic - Méthode par chromatographie en phase liquide à haute performance (HPLC) avec détection par spectrométrie de masse par torche à plasma (ICP-MS) ou génération d'hydrure fluorescence atomique (HG-AFS)

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# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>2</b>
<b>4 Principle</b> .....	<b>3</b>
<b>5 Interferences</b> .....	<b>3</b>
<b>6 Apparatus</b> .....	<b>3</b>
<b>7 Reagents and standards</b> .....	<b>4</b>
<b>8 Sampling, preservation and storage of samples</b> .....	<b>6</b>
<b>9 Procedure</b> .....	<b>6</b>
9.1 HPLC instrument optimization.....	6
9.2 Calibration.....	7
9.3 Sample measurement.....	7
<b>10 Expression of results</b> .....	<b>7</b>
<b>11 Test report</b> .....	<b>8</b>
<b>Annex A (normative) Detection using inductively coupled plasma mass spectrometry (ICP-MS)</b> .....	<b>9</b>
<b>Annex B (normative) Detection using hydride generation atomic fluorescence (HG-AFS)</b> .....	<b>20</b>
<b>Annex C (informative) Determination of organic arsenic species</b> .....	<b>27</b>
<b>Annex D (informative) Storage and preservation of arsenic species in water samples</b> .....	<b>29</b>
<b>Bibliography</b> .....	<b>40</b>

## ISO/TS 19620:2018(E)

## 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](http://www.iso.org/directives)).

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## Introduction

In the environment, metals and metalloids are found in the form of various chemical species. Chemical speciation makes it possible to identify and quantify these different species. For the same metal or metalloid, given that the toxicity of each compound may vary significantly, it can be useful to quantify each of the species present in a given sample. For arsenic, the toxicity of the various species varies considerably; inorganic species are recognized as being more toxic than organic species and, for example, the toxicity of As(III) is greater than that of As(V). This method is primarily applicable to the determination of arsenite (As(III)) and arsenate (As(V)) as these are the main species of interest and are the predominant species found in potable water samples from the underlying geological strata in many parts of the world. Arsenic speciation measurements are important to establish and select the best water treatment technology for arsenic removal from raw waters containing significant levels of arsenic.

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# Water quality — Determination of arsenic(III) and arsenic(V) species — Method using high performance liquid chromatography (HPLC) with detection by inductively coupled plasma mass spectrometry (ICP-MS) or hydride generation atomic fluorescence spectrometry (HG-AFS)

**WARNING** — Persons using this document should be familiar with normal laboratory practice. This document is not intended to cover any safety problems associated with its use, if applicable. It is the responsibility of the user to establish appropriate safety and health practices.

**IMPORTANT** — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

## 1 Scope

This document specifies a method primarily developed for the determination of inorganic arsenic species (arsenite (As(III)) and arsenate (As(V)) dissolved in a sample after a preservation process in waters with a low total organic carbon content such as potable water, tap water, surface waters, ground waters and rain waters. Information is provided on the determination of potentially relevant organo-arsenic species such as methylarsonic acid (MMA) and dimethylarsinic acid (DMA) which may be encountered at very low levels in natural surface waters.

The linear working dynamic range depends on the operating conditions and the method of detection used; under standard conditions, it typically ranges from 0,5 µg/l to 50 µg/l for each species. Samples containing arsenic at concentrations higher than the linear dynamic range can be analysed after suitable dilution.

This method is based on high performance liquid chromatography separation of arsenic species with either inductively coupled mass spectrometry (ICP-MS) or hydride generation atomic fluorescence spectrometry (HG-AFS) as a method of detection.

The sensitivity of this method depends on the method of detection and the instrumental operating conditions selected. The limit of quantification (LOQ) of the method also depends on the operating conditions of the analytical system used and the extent of the calibration range used. LOQ examples for As(III) and As(V) are provided; LOQs are generally less than 1 µg/l.

This document does not apply to arsenobetaine and other organic arsenic species which are not present in natural water samples.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 and sampling techniques*

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

## ISO/TS 19620:2018(E)

ISO 17294-1, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines*

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

#### 3.1 analyte

substance to be determined

[SOURCE: ISO/TS 28581:2012, 3.1]

#### 3.2 blank calibration solution

solution prepared in the same way as the calibration solution but leaving out the analyte

[SOURCE: ISO 17294-1:2004, 3.3]

#### 3.3 calibration solution

solution used to calibrate the instrument, prepared from (a) stock solution(s) or from a certified standard

[SOURCE: ISO 17294-1:2004, 3.4]

#### 3.4 stock solution

solution with accurately known analyte concentration(s), prepared from suitably pure chemicals

[SOURCE: ISO 17294-1:2004, 3.30]

#### 3.5 determination

entire process from preparing the test sample solution up to and including measurement and calculation of the final result

[SOURCE: ISO 17294-1:2004, 3.6]

#### 3.6 limit of quantification

##### LOQ

lowest concentration of an analyte that can be determined with a specified degree of accuracy and precision

#### 3.7 limit of detection

##### LOD

lowest concentration of an analyte that can be detected with a specified degree of accuracy and precision

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## 4 Principle

The different arsenic species are separated using a specific column in a high performance liquid chromatograph (HPLC). The separation of arsenic species in natural water is typically achieved using strong anion exchange ion chromatography. The species can be separated using isocratic conditions however faster more efficient separations can be achieved using gradient elution.

This method permits the use of ICP-MS (see [Annex A](#)) or HG-AFS (see [Annex B](#)) for individual detection of the various arsenic species.

[Annex C](#) provides information on the determination of organic arsenic species.

[Annex D](#) provides information on the stability of arsenic species using different storage and preservations.

## 5 Interferences

Retention time shift may occur for some water samples, especially when they are enriched with significant levels of various water matrix ions. These ions compete with the active sites on the column. Sample spikes using each of the arsenic species should therefore be used to confirm species identity if a retention time shift is observed. The sample may also be diluted to overcome this effect or a smaller injection volume may be used with an inferior LOQ. The co-elution of organic arsenic species with arsenite (As(III)) and arsenate (As(V)) may cause a positive interference; therefore, the separation conditions should be well proven and established.

To detect ICP-MS interferences, follow the procedure specified in [Annex A](#).

To detect HG-AFS interferences, follow the procedure specified in [Annex B](#).

## 6 Apparatus <https://standards.iteh.ai/catalog/standards/sist/0c65eca1-ba68-4654-833d-f688faa784d5/sist-ts-iso-ts-19620-2018>

Due to significant differences between the various instrument models and brands available, it is not possible to give detailed instructions on their operation. The operator shall thus refer to the instructions provided by the manufacturer of each instrument.

Usual laboratory glassware and equipment and, in particular, the following:

**6.1 High performance liquid chromatograph (HPLC)**, including a column for analyte separation and optionally a chromatographic guard column.

The HPLC system may be equipped with an autosampler, in-line degassing system and auto injection system for introducing the sample. In most cases, an isocratic pump can be used; although, the use of gradient pumps to provide optimal separation times is permitted.

**NOTE** Various column/eluent pairings can be used for separating arsenic species. A strong anion exchange column is typically used. Some examples are provided in [Annexes A](#) and [B](#).

**6.2 Vacuum filtration system**, for filtering the eluent and reagents prepared.

**6.3 pH meter**, for adjusting pH of eluent to one decimal place between 1,0 and 14,0.

**6.4 Inductively coupled plasma mass spectrometer (ICP-MS).**

See [Annex A](#).

**6.5 Hydride generation atomic fluorescence spectrometer (HG-AFS).**

See [Annex B](#).

## ISO/TS 19620:2018(E)

## 7 Reagents and standards

### 7.1 General requirements

All reagents shall be of known analytical grade. The concentration of the analyte or interfering substances in the reagents and water should be negligible compared to the lowest concentration to be determined.

NOTE Standard stock solutions are commercially available or can be prepared using chemicals of known analytical purity.

Any reagents used in the preparation of the mobile phases of the HPLC procedure are column and instrument specific and are not included here.

### 7.2 Water, grade 1, as defined in ISO 3696.

### 7.3 HPLC grade water.

HPLC grade water is used to prepare the mobile phase and the calibration solutions and to produce the sample dilutions. It can be prepared by suitably purifying water (7.2).

### 7.4 Sodium hydroxide.

### 7.5 Sodium hydroxide solution, 1 mol/l.

Weigh 4 g of sodium hydroxide pellets (7.4) and add them to a 100 ml beaker. Then add approximately 50 ml de-ionized water and stir until the pellets have dissolved. Transfer to a 100 ml volumetric flask and make up to the mark with de-ionized water (7.2).

### 7.6 Hydrochloric acid, mass fraction 35 % to 37 %.

### 7.7 Hydrochloric acid preservation solution for HG-AFS, approximately 6 mol/l.

Carefully add 500 ml of hydrochloric acid (7.6) to 250 ml of water (7.2). Make up to a final volume of 1 000 ml with water (7.2).

### 7.8 Nitric acid, mass fraction, 68 % to 72 %.

### 7.9 Nitric acid preservation solution for ICP-MS, approximately 6 mol/l.

Carefully add 365 ml of nitric acid (7.8) to 250 ml of water (7.2). Make up to a final volume of 1 000 ml with water (7.2).

### 7.10 Standard substances

As(III): Arsenious oxide  $\text{As}_2\text{O}_3$  (CAS No. 1327-53-3);

As(V): Di-sodium hydrogen arsenate  $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$  (CAS No. 10048-95-0);

DMA: Dimethylarsinic acid, refer to [Annex C](#);

MMA: Methylarsonic acid acid, refer to [Annex C](#).

### 7.11 Stock solutions 1 000 mg/l

For each of the species, As(III) and As(V), prepare a stock solution with a concentration of 1 000 mg/l expressed as As.

These solutions are prepared from the standard substances (7.10).

In 100 ml volumetric flasks, dissolve the appropriate mass of each substance (7.10) as shown in Table 1. Commercially available As(III) and As(V) stock solutions of the required concentration can also be used.

**Table 1 — Preparation guidelines for 1 000 mg/l arsenic standards**

Species	Mass g	Final volume ml
As(III)	0,132	4 ml of 1 mol/l NaOH (7.5) made up to 100 ml with water (7.2)
As(V)	0,416	100 ml in water (7.2)

These solutions, when stored protected from light and at 4 °C, are considered to be stable for one year.

### 7.12 Single component stock solutions 10 mg/l

Pipette 1 ml of 1 000 mg/l stock solution (7.11) and dilute to 100 ml in a volumetric flask.

The 10 mg/l single-component working solutions are also stable for one year if they are stored protected from light and at 4 °C and stabilized in 0,04 mol/l NaOH (7.5) for As(III).

### 7.13 Calibration solutions

The calibration solutions are prepared from the 1 000 mg/l stock solutions (7.12). Tables 2 and 3 are given as examples. The method is primarily for As(III) and As(V). It may be necessary to include DMA and MMA standards to prove that the chromatographic separation is acceptable. Additional guidance is provided in Annex C. Working standards should be prepared daily.

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**Table 2 — Preparation guidelines for working arsenic standards**

Concentration µg/l	Volume of each stock solution µl	Solution for dilution	HPLC grade water (7.3)
1 000	100 µl of each stock solution	1 000 mg/l (7.11) As(III), As(V)	100 ml
5	500 µl	1 000 µg/l	100 ml
10	1 000 µl	1 000 µg/l	100 ml
25	2 500 µl	1 000 µg/l	100 ml
50	5 000 µl	1 000 µg/l	100 ml

To prepare standard solutions below 5 µg/l use the 10 mg/l intermediate stock to produce a mixed standard of 100 µg/l. Table 3 is given as an example.

**Table 3 — Preparation guidelines for working arsenic standards**

Concentration µg/l	Volume of each working solution µl	Solution for dilution	HPLC grade water (7.3)
100	1 000 µl of each solution	10 mg/l (7.12) As(III), As(V)	100 ml
0,2	200 µl	100 µg/l	100 ml
0,5	500 µl	100 µg/l	100 ml
1,0	1 000 µl	100 µg/l	100 ml
2,0	2 000 µl	100 µg/l	100 ml