PRIVATE CIRCULATION
AW/275_14_0285
For comment/vote - Action Due Date: 2015/01/31

BSI, 389 Chiswick High Road London W4 4AL

Tel: +44 (0)20 8996 9000
Fax: +44 (0)20 8996 7400
www.bsigroup.com

Our Ref: AW/275

Date: 14 October 2014

Direct details:
Telephone: 0208 996 7009
Fax: 0208 996 7198
E-mail: csc@bsigroup.com

Dear Member

CEN ENQUIRY PROCEDURE

DEFAULT UK VOTE: ABSTAIN
REPLY TO CSC@BSIGROUP.COM BEFORE 31ST JANUARY 2015

Please find attached:

30267877 DC prEN 16802 Foodstuffs - Determination of elements and their chemical species - Determination of inorganic arsenic in foodstuffs of marine and plant origin by anion-exchange HPLC-ICP-MS following water bath extraction

This CEN draft is circulated under the Enquiry Procedure for comment. Acceptance of a draft CEN Standard means agreement to its being published as a British Standard without further change if it becomes an EN.

Please let us have your views on whether to vote for possible acceptance as a European Standard. Members are asked to also notify CSC of any National legislative/administrative deviation or National technical/economic deviation that you may wish us to raise for consideration by the CEN Secretariat.

Any UK comments that you wish to be considered for submission to CEN should be sent to CSC by the above date, together with your opinion on the vote to be returned.

When submitting comments please ensure that they are entered into the CEN comments template. If you have any queries in how to use the template then please do not hesitate to contact the Committee Service Centre.

If we do not hear from you by the above date, CSC will submit a vote to "abstain" on behalf of the UK.

Yours sincerely

Committee Service Centre

DPC: 14 / 30267877 DC

BSI Group Headquarters

389 Chiswick High Road London W4 4AL

Tel: +44 (0)20 8996 9000
Fax: +44 (0)20 8996 7400
www.bsigroup.com

Date: 14 October 2014
Origin: European

Latest date for receipt of comments: 31 January 2015

Project No. 2012/02128

Responsible committee: AW/275 Food analysis - Horizontal methods

Interested committees:

Title: Draft BS EN 16802 Foodstuffs - Determination of elements and their chemical species - Determination of inorganic arsenic in foodstuffs of marine and plant origin by anion-exchange HPLC-ICP-MS following waterbath extraction

Please notify the secretary if you are aware of any keywords that might assist in classifying or identifying the standard or if the content of this standard

i) has any issues related to 3rd party IPR, patent or copyright
ii) affects other national standard(s)
iii) requires additional national guidance or information

WARNING: THIS IS A DRAFT AND MUST NOT BE REGARDED OR USED AS A BRITISH STANDARD.
THIS DRAFT IS NOT CURRENT BEYOND 31 January 2015

This draft is issued to allow comments from interested parties; all comments will be given consideration prior to publication. No acknowledgement will normally be sent. **See overleaf for information on the submission of comments.**

Further copies of this draft may be purchased from BSI Shop **http://shop.bsigroup.com**
or from BSI Customer Services, Tel: +44(0) 20 8996 9001 or email cservices@bsigroup.com.
British, International and foreign standards are also available from BSI Customer Services.

Information on the co-operating organizations represented on the committees referenced above may be obtained from **http://standardsdevelopment.bsigroup.com**

Responsible Committee Secretary: **Committee Service Centre (BSI)**
Direct tel: **020 8996 7009**
E-mail: **csc@bsigroup.com**

Introduction

This draft standard is based on European discussions in which the UK has taken an active part. Your comments on this draft are welcome and will assist in the preparation of the consequent British Standard. Comment is particularly welcome on national, legislative or similar deviations that may be necessary.

Even if this draft standard is not approved by the UK, if it receives the necessary support in Europe, the UK will be obliged to publish the official English Language text unchanged as a British Standard and to withdraw any conflicting standard.

UK Vote

Please indicate whether you consider the UK should submit a negative (with reasons) or positive vote on this draft.

Submission of Comments

- The guidance given below is intended to ensure that all comments receive efficient and appropriate attention by the responsible BSI committee. **Annotated drafts are not acceptable and will be rejected.**

- All comments must be submitted, preferably electronically, to the Responsible Committee Secretary at the address given on the front cover. Comments should be compatible with version 6.0 or version 97 of Microsoft Word for Windows, if possible; otherwise comments in ASCII text format are acceptable. **Any comments not submitted electronically should still adhere to these format requirements.**

- All comments submitted should be presented as given in the example below. Further information on submitting comments and how to obtain a blank electronic version of a comment form are available from the BSI website at: **http://drafts.bsigroup.com/**

Template for comments and secretariat observations

Date: xx/xx/20xx	Document: **ISO/DIS xxxx**

1	2	(3)	4	5	(6)	(7)
MB	**Clause No./ Subclause No./Annex** (e.g. 3.1)	**Paragraph/ Figure/ Table/Note**	**Type of com-ment**	**Commend (justification for change) by the MB**	**Proposed change by the MB**	**Secretariat observations on each comment submitted**
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 16802

October 2014

ICS 67.050; 67.060; 67.120.30

English Version

Foodstuffs - Determination of elements and their chemical species - Determination of inorganic arsenic in foodstuffs of marine and plant origin by anion-exchange HPLC-ICP-MS following waterbath extraction

Produits alimentaires - Détermination des éléments et de leurs espèces chimiques - Détermination de la teneur en arsenic inorganique dans les produits alimentaires d'origines marine et végétale, par CLHP avec échange d'anions et spectrométrie de masse à plasma induit par haute fréquence (ICP-SM), après extraction par bain d'eau

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Bestimmung von anorganischem Arsen in Lebensmitteln marinen Ursprungs und pflanzlichen Lebensmitteln mit Anionenaustausch-HPLC-ICP-MS nach Wasserbadextraktion

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 275.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

This draft European Standard was established by CEN in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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.

Warning : This document is not a European Standard. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a European Standard.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Foreword

This document (prEN 16802:2014) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

1 Scope

This draft European Standard describes a procedure for the determination of inorganic arsenic in foodstuffs of marine and plant origin by anion-exchange HPLC-ICP-MS following waterbath extraction.

This method has been validated in an interlaboratory test on white rice, wholemeal rice, leek, blue mussels, fish muscle and seaweed with an inorganic arsenic mass fraction in the range 0,073 mg/kg to 10,3 mg/kg.

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.

EN 13804, *Foodstuffs — Determination of trace elements — Performance criteria, general considerations and sample preparation*

EN ISO 3696, *Water for analytical laboratory use – Specification and test methods (ISO 3696)*

3 Principle

Inorganic arsenic consists of arsenite, As(III) and arsenate, As(V). This standard describes a method for the determination of inorganic arsenic (=sum of As(III) and As(V)). A representative test portion of the sample is treated with a diluted nitric acid and hydrogen peroxide solution in a heated waterbath. Hereby the analytes of interest are extracted into solution and As(III) is oxidised to As(V). The inorganic arsenic is selectively separated from other arsenic compounds using anion exchange HPLC (High Performance Liquid Chromatography) coupled on-line to the element-specific detector ICP-MS (Inductively Coupled Plasma Mass Spectrometry) for the determination of the mass fraction of inorganic arsenic. External calibration with solvent matrix-matched standards is used for quantification of the amount of inorganic arsenic.

WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

4 Reagents

4.1 General

The concentration of the trace elements in the reagents and water used shall be low enough to not affect the results of the determination. When using a multi-elemental method of high sensitivity like ICP-MS, the control of the blank levels of water, acid and other reagents is very important. Generally ultra-pure water complying with ISO 3696 grade 1 (i. e. electrical conductivity below 0,1 µS/cm at 25 °C) and acid of high purity, e. g. cleaned by sub-boiling distillation, are recommended. Reagents should be of minimum p.a. quality where possible. Special facilities should be used in order to avoid contamination during the steps of preparation and measurement (e. g. uses of laminar flow benches or comparable clean room facilities).

4.2 Nitric acid (HNO_3), concentrated, ≥ 65% (mass fraction), of approximately ρ (HNO_3) 1,4 g/ml

High purity is essential to avoid potential contamination. Therefore only use nitric acid available with high purity or perform a clean-up by a sub-boiling distillation.

4.3 Hydrogen peroxide, not less than 30 % (mass fraction)

High purity is essential to avoid potential contamination. Commercially available hydrogen peroxide for analysis should be tested for contamination of arsenic.

4.4 Extractant solution 1, 0,1 mol/l HNO_3 in 3 %(v/v) H_2O_2

Pour 800 ml of H_2O and then 6,5 ml of HNO_3 (4.2) and thereafter 100 ml of H_2O_2 (4.3) into a 1 000 ml volumetric flask. Fill it up to the mark at 1 000 ml with H_2O. This solution should be prepared on the same day of use.

It is recommended that the total volume needed for the analysis is estimated and only this amount is produced.

4.5 Extractant solution 2, 0,2 mol/l HNO_3 in 6 % H_2O_2

Pour 70 ml of H_2O, 1,3 ml of HNO_3 (4.2) and 20 ml of H_2O_2 (4.3) into a 100 ml volumetric flask. Fill it up to the mark at 100 ml with H_2O. This solution should be prepared on the same day of use.

It is recommended that the total volume needed for the analysis is estimated and only this amount is produced.

4.6 Ammonium carbonate, mass fraction w ≥ 99,999 %, for production of mobile phase solution

4.7 Aqueous ammonia, w ≥ 25%, for adjustment of pH in the mobile phase

4.8 Methanol (CH_3OH), HPLC grade, for production of mobile phase solution

4.9 Mobile phase, e.g. 50 mmol/l ammonium carbonate in 3 % methanol at pH 10,3

Dissolve e.g. 4,80 g of ammonium carbonate (4.6) in approximately 800 ml of water and add 30 ml of methanol (4.8). Adjust the pH to 10,3 with aqueous ammonia (4.7) and fill up to 1 000 ml with water. Filter the mobile phase solution through a 0,45 µm filter prior to use.

NOTE 1 The optimal concentration of ammonium carbonate in the mobile phase depends on the analytical column used (e.g. brand, particle size and dimensions). The optimal concentration of ammonia carbonate is in the discretion of the analyst and should fulfil the criteria for sufficient resolution of the arsenate peak as stated in 5.10.

NOTE 2 Methanol is added to the mobile phase in order to enhance the signal intensity for arsenic (carbon enhancement effect [1]). The optimal concentration of methanol to get optimal signal enhancement effect depends on the instrument used and should be decided by the analyst.

4.10 Diarsenic trioxide (As_2O_3), $w(As_2O_3)$ ≥ 99,5 %

4.11 Potassium hydroxide solution, ρ (NaOH)= 20 g/100 ml

4.12 Sulfuric acid solutions, $w(H_2SO_4)$ = 20% and $w(H_2SO_4)$ = 1%

4.13 Phenolphthalein

4.14 Standard solutions, with an arsenic mass concentration of 1 000 mg/l

The use of commercial standards of arsenic, arsenic III and/or V, with a mass concentration of 1 000 mg/l is recommended.

Otherwise proceed as follows: Dissolve e.g. 1,320 g of diarsenic trioxide (4.10) in 25 ml of potassium hydroxide solution (4.11), neutralize with 20 % sulfuric acid solution (4.12) with phenolphthalein (4.13) as indicator and dilute to 1 000 ml in a volumetric flask with 1 % sulfuric acid solution (4.12).

NOTE By preparing the standard in the extractant solution 1 (4.4) all arsenite will be completely oxidized to arsenate.

4.15 Calibration solutions

Prepare a range of standards including a blank calibration solution that covers the linear range of the analyte to be determined by diluting the analyte stock solution with extractant solution (4.4). Appropriate matrix matching of the calibration solutions shall be performed by using the extractant solution (4.4) for the final dilution step, which furthermore will prevent reduction of arsenate to arsenite. Transfer an aliquot of the calibration solutions to HPLC vials prior to analysis (6.3.2).

The quantitative oxidation of arsenite to arsenate in the standard solutions should be verified (e.g. visual inspection of chromatogram).

5 Apparatus and equipment

5.1 General

To minimise the contamination, all apparatus and equipment that come into direct contact with the sample and the solutions shall be carefully pre-treated. It is recommended to avoid the use of glassware, since this may cause contamination with arsenate, see [2].

5.2 Laboratory grinder, capable of grinding to a particle size of less than 0,5 mm

5.3 Analytical balance, capable of weighing to an accuracy of 1 mg

5.4 Filtering device, for filtration of mobile phase solution

5.5 Waterbath, capable of programming of the temperature at 90 °C

5.6 Centrifuge, capable of a centrifuge speed of minimum 4 000 min^{-1} (2 010 g)

5.7 Single use syringe filters (0,45 µm) or HPLC vials with filters, compatible with acidic solutions for filtering of test solutions prior to analysis

5.8 One-mark plastic volumetric flasks, for preparation of mobile phase and calibration solutions

NOTE If calibration standards are prepared by weighing, plasticware without marks can be used.

5.9 High Pressure Liquid Chromatography equipment (HPLC)

5.10 Strong anion exchange column (SAX), suitable for selective separation of arsenate from other arsenic compounds present in the sample extracts.

NOTE 1 It is advisable to use a guard column to prolong the life-time of the analytical column.

NOTE 2 As a guideline the minimum acceptable retention time for the analyte is twice the retention time corresponding to the void volume of the column. Furthermore the nearest peak in the chromatogram should be separated from the analyte peak by at least one full peak width at 10 % of the analyte peak height. It is recommended to verify sufficient separation of the analyte peak using a solution of organic arsenic compounds (e.g. monomethylarsenous acid (MA), dimethylarsinic acid (DMA) and arsenobetaine (AB)) and arsenate. Examples of chromatographic SAX columns used in the collaborative trial can be found in Annex B.

5.11 Inductively coupled plasma mass spectrometer (ICP-MS)

5.12 Argon gas, purity ≥ 99,99 %

6 Procedure

6.1 General

Follow procedures for sample preparation as given in EN 13804.

6.2 Waterbath extraction

Weigh a test portion of approximately 0,2 g to 0,5 g sample corresponding to dry weight into a tube and fill up to 10,00 ml with extractant solution 1 (4.4). Include also a reagent blank sample. The tubes shall be securely closed with a tight lid. Shake the tubes thoroughly in order to get good contact between sample and solvent (e.g. by vortexing). The solutions are then placed in a heated waterbath at 90 °C +/- 2 °C and extracted for 60 min +/- 5 min.

If a fresh sample, containing 0,2 g to 0,5 g dry matter is extracted, the water content has to be taken into account. The concentration of extractant solution should be adjusted accordingly, keeping the matrix matching at the same level. Proceed e.g. as follows: weigh in the test sample and add water up to 5 ml and mix thoroughly then add double concentrated extractant solution 2 (4.5) to 10 ml and mix. Proceed as described above.

It is important that the sample is wetted sufficiently in the extractant solution 1 (4.4) prior to placing it in the waterbath in order to ensure a satisfactory extraction of the analyte. Some finely powdered samples may need extended wetting time (e.g. overnight) prior to the waterbath treatment.

Following the waterbath extraction step, let samples be cooled to room temperature and subsequently centrifuge the tubes (10 min, 4 000 min^{-1} (2 010 g)). Transfer the supernatant transferred to clean containers which and can usually be stored in a refrigerator (at approximately 4 °C) for a maximum of one week until analysis (6.3.3). All sample extracts should be filtered (5.7) and transferred to HPLC vials prior to analysis.

6.3 Determination of inorganic arsenic by HPLC-ICP-MS

6.3.1 Preparation of HPLC-ICP-MS for analysis

The HPLC and ICP-MS operating conditions shall be based on the general information provided by the manufacturer of the instruments taking into account the operating conditions of the analytical column.

NOTE An arsenic solution, e.g. 10 µg/l arsenate in 3 % methanol, may be used to optimize the test system according to the manufacturer's instructions. Arsenic is mono-isotopic and can be evaluated at a mass/charge ratio (m/z) of 75.

It is advisable to allow the HPLC system (incl. the analytical column) to equilibrate and ensure stable conditions by turning on the HPLC flow in advance prior to start of the analysis. Repeated injections of a sample extract may be necessary until stable chromatography is achieved and the analytical sequence can be started. Make sure that the HPLC run is long enough for chloride (m/z 35) to elute from the column prior to injection of the next sample. It should furthermore be ensured that the arsenate and chloride peaks do not co-elute in order to avoid interference from the polyatomic ion 40Ar35Cl+.

6.3.2 Calibration

Inject an appropriate volume of the arsenic calibration solutions (4.15) into an anion exchange HPLC-ICP-MS system and determine the peak area of each of the calibration points to construct a calibration curve.

6.3.3 Determination of samples and blank solution

Inject an appropriate volume of the reagent blank solution and the sample test solutions (6.2) into an anion exchange HPLC-ICP-MS and determine the peak areas under appropriate HPLC-ICP-MS settings, e. g. such as listed in Table 1. Test solutions, which give a response outside the linear calibration range, should be diluted appropriately with extractant solution 1 (4.4) to give a response within the linear calibration range. If a

significant blank value occurs, identify the source of this blank. The source should be eliminated and the analysis repeated.

6.3.4 HPLC sequence

Take measures to control the stability of the instrument sensitivity during the analytical run. Control the instrument sensitivity by e.g. analysing a calibration standard solution throughout the sequence (for example, after each five to ten samples) and, if necessary, use the results for re-calibration of the system. Another possibility is to introduce an internal standard (e.g. Germanium) post-column (by e.g. a T-split) and use the signal for correction of instrument drift (if any) during the analytical run.

6.3.5 Typical HPLC-ICP-MS settings

Table 1 — Example of typical settings of HPLC-ICP-MS instrumentation

ICPMS settings -	
ICP-MS	Agilent 7 500ce
RF power (W)	1 500
Carrier gas flow (l min^{-1})	1,2
Plasma gas flow (l min^{-1})	15
Auxiliary gas flow (l min^{-1})	1,0
Mass resolution (amu)	0,6 to 0,8
Integration time (ms)	1000
Isotopes monitored (m / z)	75 (As), 35 (Cl)
HPLC settings -	
HPLC	Agilent 1 100
Column	IonPac AS7 (250 mm × 4 mm)
Flow rate (ml min^{-1})	0,15
Operating pressure (bar)	50
Injection volume (µl)	5
Measurement time (min)	15

6.4 Quality control

As an analytical control, reference samples having reliable known inorganic arsenic contents shall be analysed in parallel with all the series of samples to estimate the trueness. The reference samples are to be subjected to all the steps in the method starting from waterbath extraction. This also applies to the preparation of blank solution.

If reference samples are not available spike experiments should be performed and the recovery used to estimate trueness of the analysis. It is advisable to check for memory effects, e.g. by analysis of blank solutions after reference materials.

The oxidation of arsenite to arsenate should be verified by performing recovery experiments from spiking of a known amount of arsenite to a test sample in the extraction step. If this oxidation is not complete the amount of sample has to be reduced and/or the hydrogen peroxide solution (4.3) has to be checked for degradation.

7 Calculation

7.1 Integration of peaks

The retention time of arsenate is identified from the analysis of the calibration solutions. The arsenate peak area in the standards, reagent blank and sample extract solutions is determined.

7.2 Inorganic arsenic in test solutions

Calculate the concentration of inorganic arsenic in the test solutions using the calibration function established by linear regression from the calibration curve.

7.3 Calculation of inorganic arsenic in the samples

Calculate the mass fraction w of inorganic arsenic in milligram per kilogram sample according to equation 1:

$$w = \frac{c_t \times V_s \times F}{m_t} \quad (1)$$

Where

c_t concentration of arsenic in the sample test solution, in microgram per liter];

V_s volume of extractant solution for waterbath extraction (usually 0,01 l), in liter;

m_t mass of test portion, in gram;

F dilution factor.

If the sample is dried prior to analysis, the result should be corrected for the moisture content.

The concentration of inorganic arsenic in the blank solution shall be as low as possible (see 6.3.3). If the inorganic arsenic concentration in the blank is constant and not avoidable, it should be subtracted from c_t.

8 Precision

8.1 General

Results from a collaborative test are summarised in Annex A. The values derived from this collaborative test may not be applicable to concentration ranges and matrices other than those given in Annex A. Further information can be found in a report on the conduction and results from the collaborative test [3].

8.2 Repeatability

The absolute difference between two independent single test results obtained with the same test method on identical test material in the same laboratory by the same operator using the same apparatus within a short time interval will exceed the repeatability limit r given in Table 2 in not more than 5 % of the cases.

8.3 Reproducibility

The absolute difference between two single test results obtained with the same test method on identical test material in different laboratories by different operators using different equipment will exceed the reproducibility R given in Table 2 not more than 5 % of the cases.

Table 2 — Mean values, repeatability and reproducibility limits for inorganic arsenic in foodstuffs included in the collaborative test.

Foodstuff	$\overline{x}$	*r*	*R*
	mg/kg	mg/kg	mg/kg
White rice	0,073	0,010	0,022
Wholemeal rice	0,47	0,03	0,12
Leek	0,086	0,015	0,033
Blue mussels	0,33	0,06	0,14
Fish muscle	0,27	0,05	0,11
Seaweed	10,3	1,2	3,4

9 Test report

The test report should fulfil the requirements in ISO/IEC/17025 and specify at least the following:

a) all information necessary for the complete identification of the sample;

b) test method used and the element to be determined, with reference to this document;

c) test results obtained and the units in which they are specified;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

g) all operating details not specified in this document, or regarded as optional, together with details of any incidents occurred when performing the method which may have influenced the test result(s).

Annex A
(informative)

Validation data

The precision and trueness of the method was established by the CEN TC 275 “Food analysis – Horizontal methods” Working Group 10 “Elements and their chemical species” in an interlaboratory test among thirteen laboratories performed in 2013 under the mandate given by the EU Commission. The participants analyzed six different foodstuff samples in duplicates. Further details on the study can be found in the report from the collaborative trial [3].

Lyophilised samples		White rice	Wholemeal rice	Leek	Blue mussels	Fish muscle	Seaweed
No of labs		13	13	13	13	13	13
No of valid labs		13	12	12	13	13	13
No of outliers		0	1	1	0	0	0
Overall mean $\bar{x}$	mg/kg	0,073	0,47	0,086	0,33	0,27	10,3
Repeatability, S_r	mg/kg	0,003 6	0,009 0	0,005 4	0,020	0,017	0,44
RSD_r	%	4,9	1,9	6,3	6,2	6,3	4,3
Repeatability limit, r_L	mg/kg	0,010	0,025	0,015	0,057	0,049	1,2
Reproducibility, S_R	mg/kg	0,008	0,043	0,012	0,049	0,038	1,2
RSD_R	%	11,0	9,1	13,8	14,9	13,8	11,8
Reproducibility limit, R_L	mg/kg	0,022	0,12	0,033	0,14	0,11	3,4
Horwitz value	%	22	17,8	22	18,8	19,3	11,2
HorRat	-	0,5	0,5	0,6	0,8	0,7	1,0

Annex B
(informative)

Supplementary information about chromatographic conditions

The following strong anion exchange (SAX) columns were used by the participants in the collaborative trial:

- IonPac AS7
- ICSep Ion120
- Hamilton PRP X-100

It is recommended to use a guard column to protect and prolong the life-time of the analytical column. The instructions from the column producers should be followed when selecting the chromatographic conditions for the column e.g. temperature, mobile phase flow, injection volume.

The participants in the collaborative trial used typically a mobile phase concentration in the range of 20 mmol/l to 50 mmol/l to achieve a satisfactory separation of the arsenate peak with the peaks of other arsenic compounds.

Bibliography

[1] Larsen, E. H. and Stürup, S., Carbon-enhanced Inductively Coupled Plasma Mass Spectrometric Detection of Arsenic and Selenium and Its Application to Arsenic Speciation, Journal of Analytical Atomic Spectrometry, 1994, 9, 1099-1105.

[2] Francesconi, K. A., Tanggaard, R., McKenzie C. J., and Goessler, S., Arsenic metabolites in human urine after ingestion of an arsenosugar, Clin. Chem., 2002, 48, 539.

[3] Sloth, J. J, Determination of inorganic arsenic in foodstuffs of marine and plant origin by HPLC-ICPMS – results from a collaborative trial, 2014, to be published later