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Fireworks — Test methods for determination of specific chemical substances —

Part 8: Arsenic content by hydride generation atomic fluorescence spectrometry

Artifices de divertissement — Méthodes d'essai pour la détermination de substances chimiques spécifiques —

Partie 8: Teneur en arsenic par spectrométrie de fluorescence atomique par génération d'hydrures

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Foreword

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This document was prepared by Technical Committee ISO/TC 264, *Fireworks*.

A list of all the parts in the ISO 22863 series can be found on the ISO website.

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Fireworks — Test methods for determination of specific chemical substances —

Part 8:
Arsenic content by hydride generation atomic fluorescence spectrometry

1 Scope

This document specifies the test method for the determination of the arsenic content in pyrotechnic compositions by hydride generation -atomic fluorescence spectrometry

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 22863-1, *Fireworks — Test methods for determination of specific chemical substances — Part 1: General*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 22863-1 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/

4 Principle of the method

After the sample is dissolved by acidic heating, the pentavalent arsenic is first reduced to trivalent arsenic by thiourea in an acidic medium and then potassium borohydride is reacted to form a volatile hydride (AsH_3), which is then loaded by a carrier gas (argon). The hydride is decomposed by the atomizer into atomic arsenic. Under the illumination of the arsenic hollow cathode lamp, the fluorescence of the characteristic wavelength is emitted, and the fluorescence intensity is proportional to the arsenic concentration in the liquid to be measured and is quantitatively compared with the standard series.

5 Reagents

5.1 Perchloric acid (GR)

5.2 Nitric acid (GR)

5.3 Thiourea (AR)

5.4 Ascorbic acid (AR)

5.5 Potassium borohydride (AR)

5.6 Sodium hydroxide (AR)

5.7 Nitric acid solution (volume fraction 5 %):

Take 50 ml of nitric acid (5.2) with a pipette and dilute it to 1 000 ml with water.

5.8 Thiourea (5 % by mass) - ascorbic acid (5 % by mass) mixed solution:

Weigh 10,0 g of thiourea (5.3), dissolve it in water, and weigh 10,0 g of ascorbic acid (5.4), add it into the water solution of thiourea, dilute the resulting solution with water to 200 ml; dilution shall be made just before use.

5.9 Sodium hydroxide solution (mass fraction 0,2 %):

Weigh 1,0 g of sodium hydroxide (5.6) and dissolve it in 500 ml of water.

5.10 Potassium borohydride solution (mass fraction 2 %):

Weigh 1,0 g of potassium borohydride (5.5) and dissolve it in 500 ml of sodium hydroxide solution (5.9).

5.11 Arsenic standard solution (1 000 mg/l)

5.12 Arsenic standard solution (1 µg/ml):

Take 100 µl of arsenic standard solution (5.11) and add nitric acid solution (5.7) to 100 ml.

5.13 Preparation of Arsenic standard “work curve” solutions:

Place 0,0 ml, 0,1 ml, 0,2 ml, 0,5 ml, 0,7 ml, 1,0 ml of arsenic standard solution (5.12) in 100 ml volume bottles, add 20 ml of thiourea (mass fraction 5 %) - ascorbic acid (mass fraction 5 %) mixed solution (5.8), add nitric acid solution (5.7) to 100 ml and mix, so as to make the arsenic standard “work curve” solutions The resulting concentrations are 0 µg/l, 1 µg/l, 2 µg/l , 5 µg/l , 7 µg/l and 10 µg/l respectively.

6 Apparatus

6.1 Agate mortar

6.2 80 mesh standard sample sieve

6.3 Heating plate

6.4 Atomic fluorescence photometer: equipped with a arsenic hollow cathode lamp

6.5 Teflon capped beaker

6.6 Volumetric flasks (100 and 50 ml)

6.7 Analytical balance, accurate to 0,0001 g

7 Test procedure

7.1 Sample pre-treatment, digestion and preparation of the solution to be tested

Firstly, the agent is ground in an agate mortar (6.1), sieved with a standard sample sieve (6.2), and then the sieved sample powder is weighed to 0,2 g using the analytical balance (6.7) and placed in a 50 ml Teflon capped beaker (6.5).

Add 2 ml of water, shake to mix, then add 5 ml of nitric acid (5.2) and 15 ml of perchloric acid (5.1).

After shaking for a while, the beaker is capped and heated with its content on a heating plate (6.3) at 210 °C until the sample is completely dissolved. When it takes a transparent light-yellow colour, remove the cap and heat the solution to evaporate to 1-2 ml, cool to room temperature.

Transfer the solution to a 50 ml volumetric flask (6.6), add 10 ml of thiourea (5 % by mass) - ascorbic acid (5 % by mass) mixed solution (5.8), and dilute to 50 ml with a nitric acid solution (5.7). Mix, filter the solution through a filter paper and then place it on the atomic fluorescence photometer (6.4). Perform the test.

Prepare a blank test solution by mixing 2 ml of water with 5 ml of nitric acid (5.2) and 15 ml of perchloric acid (5.1). Heat the solution to evaporate to 1-2 ml (same volume as for the sample solution).

Transfer the blank test solution to a 50 ml volumetric flask (6.6), add 10 ml of thiourea (5 % by mass) - ascorbic acid (5 % by mass) mixed solution (5.8), and dilute to 50 ml with a nitric acid solution (5.7). Mix, filter the solution through a filter paper and then place it on the atomic fluorescence photometer (6.4). Perform the blank test.

7.2 Test conditions

The operative conditions of the atomic fluorescence spectrometer (6.4) shall be set to the appropriate settings to obtain the best performance.

For instance, the following requirements shall apply to the atomic fluorescence photometer where appropriate:

Negative high pressure/voltage: 270 V; lamp current: 60 mA; furnace height: 8 mm; carrier gas flow rate: 500 ml/min; shielding flow: 1 000 ml/min; reading mode: peak area; measurement method: standard curve method.

Instrument precision requirements: the blank test solution is measured several times and the fluorescence intensity is not more than 5.

7.3 Calculations

Calculate the arsenic concentration in the samples by using Formula 1:

$$W(As) = \frac{50(p - p_0)}{1000\ m} \quad (1)$$

where

$W(As)$ is the content of arsenic in the sample, mg/kg or µg/g.

p is the concentration of the sample test solution as measured by the atomic fluorescence photometer, µg/l.

p_0 is the concentration of the blank test solution as measured by the atomic fluorescence photometer, µg/l

m is the mass of the sample, g

The calculation results shall be given with two significant digits.

8 Accuracy

The absolute difference between two independent determinations obtained under repeatability conditions shall not exceed 20 % of the arithmetic mean.

Accuracy improvement can be obtained using the standard addition method (See Annex A)

9 Other

When the sample is 0,2 g and the volume is 50 ml, the limit of detection (LOD) is 0,0,1 mg/kg and the limit of quantification (LOQ) is 0,3 mg/kg.

10 Test report

The test report shall include at least the following information:

— name and address of the testing laboratory;

— date of issue;

— reference to this document, i.e. ISO 22863-8:2021;

— necessary description of the sample and how it was obtained according to ISO 22863-1;

— the identification of qualitative analysis and quantitative analysis;

— results of the analysis;

— any anomaly that occurred while performing the tests.

Annex A
(normative)

Standard addition method

A.1 General

This second method eliminates the “matrix” effects that result from the digestion process where other ions corresponding to other compounds than arsenic ones may have been formed and remain in the digested sample solution to be tested. Such ions are likely to have an impact on the spectrometric records.

It may be used to improve the accuracy of measurements.

A.2 Sample size

Take one 0,5 g sample, using the analytical balance (6.7)

Duplicate the sample.

A.3 General requirement

The analysis of the two samples shall be carried out immediately one after the other.

For error correction, a blank test shall be carried out in parallel with an arsenic-free blank solution.

A.4 Test procedure

A.4.1 Digestion process

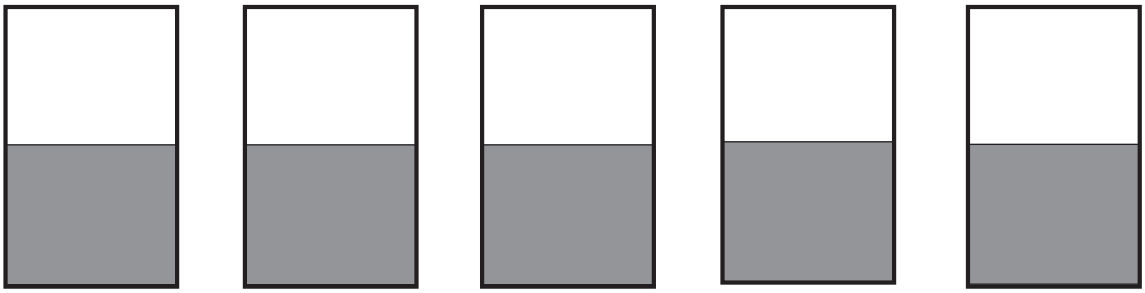
The same digestion process as described in (7.1) shall be carried out to obtain the digested sample solution that is to be diluted and tested according to A.4.2 to A.4.4.

The mentioned quantities of each of the acids to be used shall be multiplied by 2,5 to take into account the larger sample size as given in (A.2).

A.4.2 Dilution of the digested sample solution

Prepare 100 ml of a diluted solution of the standard diluted solution of arsenic (5.13) to a concentration of 10,0 µg/l.

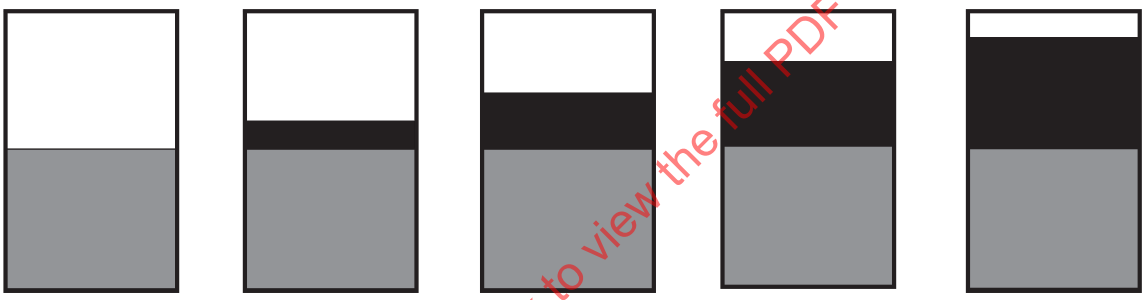
Pour 50 ml of the digested sample solution in each of a set of 100 ml flasks that shall be numbered from 1 to 5.



Key

1 Nr 1
2 Nr 2
3 Nr 3
4 Nr 4
5 Nr 5

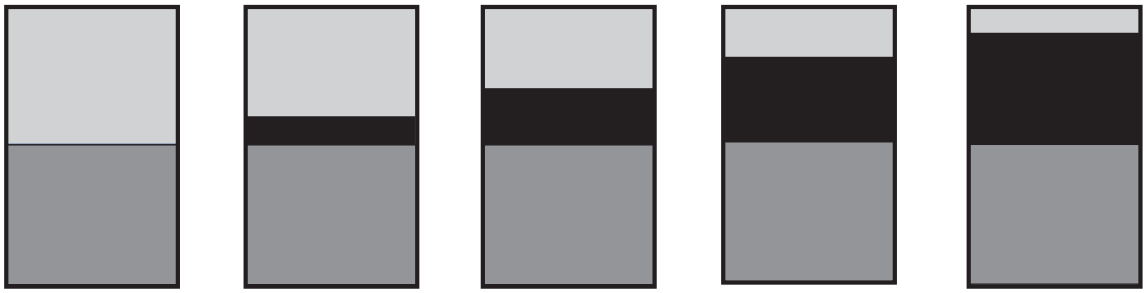
Add carefully 10 ml of the above 10,0 µg/l diluted solution of arsenic in flask Nr 2, 20 ml of the same solution in flask Nr 3, 30 ml in flask Nr 4 and 40 ml in flask Nr 5.



Key

1 Nr 1
2 Nr 2
3 Nr 3
4 Nr 4
5 Nr 5

Add water in all flasks 1 to 5 up to the 100 ml graduation and mix minutely.



Key

1 Nr 1
2 Nr 2
3 Nr 3
4 Nr 4
5 Nr 5

The dosing of the digested sample solution and of the 10,0 µg/l diluted solution of arsenic in each of the flasks shall be done as accurately as possible by using the best laboratory practices.

A.4.3 Measurement

Set up the atomic fluorescence photometer (6.4) to the optimized working conditions according to its manufacturer's instructions.

Perform the test according to manufacturer's instructions and record the absorbances for each of the solutions of flasks Nr 1 to 5.

A.4.4 Calculations

Calculate the concentration of added 10,0 µg/l diluted solution of arsenic for each flask as measured after dilution.

Plot recorded absorbances vs. corresponding calculated concentrations of added 10,0 µg/l diluted solution of arsenic. The plotted points should be approximatively aligned.

Determine the equation of the regression line which is the closest from all points. Most spreadsheet software that are currently available have such capability.

Using the equation of the regression line, calculate the value where the regression line intercepts the x-axis. That value represents the concentration of arsenic in flask Nr 1 and shall be multiplied by 2 to obtain the actual concentration C of the digested sample solution.