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First edition

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

PROOF/ÉPREUVE

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Contents

Page

Foreword

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This document was prepared by Technical Committee ISO/TC 264, *Fireworks*, WG 4.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The ISO 22863 series consists of the following parts, under the general title “Test methods for determination of specific chemical substances”:

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

— ISO 22863-2, Fireworks – Test methods for determination of specific chemical substances – Part 2: Hexachlorobenzene by gas chromatography

— ISO 22863-3, Fireworks – Test methods for determination of specific chemical substances – Part 3: Lead and lead compounds by atomic absorption

— ISO 22863-4, Fireworks – Test methods for determination of specific chemical substances – Part 4: Lead and lead compounds by X-ray fluorescence spectrometry (XRF)

— ISO 22863-5, Fireworks – Test methods for determination of specific chemical substances – Part 5: Lead and lead compounds by inductive coupled argon plasma optical emission spectrometry (ICAP-OES)

— ISO 22863-6, Fireworks—Test methods for determination of specific chemical substances – Part 6: Zirconium with a particle size of less than 40 µm by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)

— ISO 22863-7, Fireworks—Test methods for determination of specific chemical substances – Part 7: Chlorates content by Chemical Titration Analysis

— ISO 22863-8, Fireworks—Test methods for determination of specific chemical substances – Part 8: Arsenic content by hydride generation-atomic fluorescence spectrometry

— ISO 22863-9, Fireworks—Test methods for determination of specific chemical substances – Part 9: Mercury content by hydride generation-atomic fluorescence spectrometry

— ISO 22863-10, Fireworks — Test methods for determination of specific chemical substances - Part 10: Nitrogen content in nitrocellulose by Iron(II) sulphate titration

— ISO 22863-11, Fireworks — Test methods for determination of specific chemical substances – Part 11: Phosphorus content by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)

— ISO 22863-12, Fireworks — Test methods for determination of specific chemical substances – Part 12: Picrates and picric acid by high performance liquid chromatography

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:

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

where