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Fireworks — Test methods for determination of specific chemical substances —

Part 9: Mercury content by hydride generation atomic fluorescence spectrometry

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

Partie 9: Teneur en mercure par spectrométrie de fluorescence atomique par génération d'hydrures

PROOF/ÉPREUVE

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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 9: Mercury content by hydride generation atomic fluorescence spectrometry

1 Scope

This document specifies the test method for the determination of the mercury 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

The sample is heated and digested in a boiling water bath using a nitric acid - hydrochloric acid mixed reagent. In the acidic medium, the mercury in the sample is reduced to atomic mercury by potassium borohydride, and then loaded into the atomic fluorescence photometer by a carrier gas (argon). Under the irradiation of a mercury hollow cathode lamp, the mercury atoms will emit fluorescence with a characteristic wavelength when they transit from high energy state to ground state. The fluorescence intensity is proportional to the mercury concentration in the liquid to be measured and is quantitatively compared with mercury standard solutions.

5 Reagents

5.1 Hydrochloric acid (GR)

5.2 Nitric acid (GR)

5.3 Potassium dichromate (GR)

5.4 Sodium hydroxide (GR)

5.5 Potassium borohydride (AR)

5.6 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.

5.7 Nitric diluted solution of potassium dichromate (0,5 g/l):

Weigh and dissolve 0,5 g of potassium dichromate (5.3) in 1 000 ml of nitric acid solution (5.6).

5.8 Hydrochloric acid-nitric acid mixed reagent [(1 + 1) aqua regia]:

Mix 150 ml of hydrochloric acid (5.1) with 50 ml of nitric acid (5.2) and then dilute it with water to double.

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

Weigh and dissolve 1,0 g of sodium hydroxide (5.4) in 500 ml of water.

5.10 Potassium borohydride solution (mass fraction 2 %):

Weigh and dissolve 10,0 g of potassium borohydride (5.5) in 500 ml of sodium hydroxide solution (5.9).

5.11 Mercury standard solution (1 000 mg/l)

5.12 Mercury standard intermediate solution (1 µg/ml):

Take 100 µl of mercury standard solution (5.11) with a pipette, dilute it to 100 ml by adding nitric diluted potassium dichromate solution (5.7), shake well.

5.13 Mercury standard use solution (20 µg/l):

Take 1 ml of mercury standard intermediate solution (5.12) with a pipette and dilute it to 50 ml by adding nitric diluted potassium dichromate solution (5.7), shake well.

5.14 Preparation of mercury standard working curve solutions:

Separately place 0,0 ml, 0,5 ml, 1,0 ml, 2,0 ml, 3,0 ml, 5,0 ml mercury standard use solution (5.13) in a 50 ml volumetric flask (6.5), dilute with the nitric diluted potassium dichromate solution (5.7) to 50 ml so that the concentrations of the mercury standard working curve solutions are 0,0 µg/l, 0,2 µg/l, 0,4 µg/l, 0,8 µg/l, 1,2 µg/l and 2,0 µg/l. Shake well.

6 Apparatus

6.1 Agate mortar

6.2 80 mesh standard sample sieve

6.3 Water bath

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

6.5 Volumetric flasks (50 ml)

6.6 Capped test tubes: volume 100 ml

6.7 Filter paper

6.8 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 sample is crushed in the agate mortar (6.1) and then sieved with a 80-mesh standard sample sieve (6.2). The sieved sample powder is weighed to 0,2 g using the analytical balance (6.8) and placed in the 100 ml capped test tube (6.6), add 2 ml of water, shake to mix.

Then 15 ml of the hydrochloric acid - nitric acid mixed reagent (5.8) is added. Shake well and place it for 2 hours in the boiling water bath (6.3), and then take it out from the water bath and let it cool for a short while. Add nitric diluted solution of potassium dichromate (5.7) to 100 ml and shake well.

Filter the solution through a filter paper (6.7) and then place it on the atomic fluorescence photometer (6.4). Perform the test.

At the same time, a blank test shall be carried out. Prepare a blank test solution by mixing 2 ml of water with 15 ml of the hydrochloric acid - nitric acid mixed reagent (5.8). Shake well and place it for 2 hours in the boiling water bath (6.3), and then take it out from the water bath and let it cool for a short while. Add nitric diluted solution of potassium dichromate (5.7) to 100 ml and shake well. 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: 30 mA; furnace height: 10 mm; carrier gas flow: 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, fluorescence intensity range of not more than 5.

8 Calculations

Calculate the mercury concentration by using Formula (1):

$$W(Hg)=\frac{(p-p_0)\cdot V}{1000m} \quad (1)$$

where

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

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

p_0 is the concentration of the reagent blank measured on the atomic fluorescence photometer, µg/l