INTERNATIONAL STANDARD

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First edition

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)

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

Partie 6: Zirconium de granulométrie inférieure à 40 µm par spectrométrie d'émission optique à plasma à couplage inductif (ICP-OES)

PROOF/ÉPREUVE

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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.

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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.

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 6:
Zirconium with a particle size of less than 40 µm by Inductively Coupled Plasma Optical Emission Spectrometry(ICP-OES)

1 Scope

This document specifies the qualitative and quantitative analysis methods for the determination of the presence and content of zirconium with a particle size of less than 40 µm in pyrotechnic compositions by specific colour-rendering reactions and Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), with a minimum detection limit of 100 mg/kg.

The determination of the presence of zirconium with a particle size of less than 40 µm is required for application of ISO 25947-5. Quantitative analysis may bring complementary information if necessary.

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

Qualitative analysis of zirconium: decomposition of the sample with hydrochloric acid, nitric acid, hydrofluoric acid and perchloric acid, removal of fluorine ions by evaporation of perchloric acid, determination of the presence of zirconium by the specific coloration reaction with arsenazo III in strong acidic solution.

Hydrofluoric acid may not be used if the composition of the tested sample does not contain titanium.

Quantitative analysis of zirconium: decomposition of the sample with hydrochloric acid, nitric acid, hydrofluoric acid and perchloric acid and then use of Inductively Coupled Plasma Optical Emission Spectrometry to determine the zirconium content.

5 Reagents

Unless otherwise stated, only confirmed as analytical reagent, distilled water or deionized water or equivalent purity water shall be used.

5.1 Hydrochloric acid (ρ = 1,19 g/ml)

5.2 Diluted hydrochloric acid (1 part acid + 1 part water)

5.3 Nitric acid (ρ = 1,42 g/ml)

5.4 Hydrofluoric acid (ρ = 1,15 g/ml)

5.5 Perchloric acid (ρ = 1,67 g/ml)

5.6 Diluted Nitric acid (1 part acid + 1 part water)

5.7 Diluted Nitric acid (1 part acid + 19 parts water)

5.8 Tartaric acid solution (20 g of tartaric acid dissolved in 100 ml of water)

5.9 Urea solution (5 g of urea dissolved in 100 ml of water)

5.10 Arsenazo III solution (0,1 g of arsenazo III dissolved in 100 ml of water)

5.11 Standard solution of zirconium (1 000 µg/ml)

5.12 Standard diluted solution of zirconium (100 µg/ml):

Pour 10,00 ml of zirconium standard solution (5.11) into a 100 ml flask, add diluted nitric acid (5.7) up to the 100 ml graduation, then mix.

6 Apparatus

6.1 Inductively Coupled Plasma Optical Emission Spectrophotometer:

Operating conditions shall follow the manufacturer's instructions.

6.2 Analytical Balance: accuracy 0,1 mg.

6.3 Electric hot plate: capable of reaching 500 °C

6.4 Quartz beakers

6.5 Volumetric flasks: capable of 100 ml

6.6 Test tubes

7 Preparations

Before weighing the sample, the composition extracted from the fireworks shall be sieved to less than 40 µm according to ISO 22863-1 5.3.2.2.

8 Analysis

8.1 General

The analysis procedure may start from step 8.2. If the test result in qualitative analysis is negative (-), then conclude the absence of zirconium in the sample. Otherwise, continue to step 8.3 of the quantitative analysis procedure, to determine the content of zirconium. Where appropriate, step 8.2 of the qualitative analysis can also be omitted and the quantitative analysis directly started from step 8.3.

8.2 Qualitative analysis

8.2.1 Sample size

Take one 0,2 g sample, using the analytical balance (6.2).

8.2.2 Digestion process

Place the sample (8.2.1) in a quartz beaker (6.4), wet it with a small amount of distilled or deionized water, add diluted hydrochloric acid (5.2) dropwise until the violent reaction is completed, then add 20 ml~25 ml of hydrochloric acid (5.1), 5 ml of nitric acid (5.3), 1 ml~2 ml of hydrofluoric acid (5.4), mix it, heat at low temperature (e.g. 100-110 °C) on the electric hot plate (6.3) for about 15 minutes.

Add 10 ml of perchloric acid (5.4), add 15 ml of nitric acid (5.3), heat until it starts to evaporate (emission of vapours). When the solution is clear, cool, rinse the inside wall of the beaker above the surface of the solution with a small amount of water, and then continue to heat until it boils (take care not to cover the surface of the beaker) and reaches an almost dry salt state on an electric hot plate (6.3).

After cooling, add 15 ml of tartaric acid solution (5.8) to the salt.

Heat the solution to dissolve the salts and cool it again.

8.2.3 Testing

Place 0,5 ml of the solution (8.2.2) in a test tube (6.6), add 2 ml of diluted nitric acid (5.6), 5-6 drops of urea solution (5.9), mix. After 5 min, add 5 drops of arsenazo III solution (5.10), mix.

Observed the colour of the solution after 15 minutes ~20 minutes. If the solution is not changed to blue, the absence of zirconium can be concluded, and the result is negative (-); otherwise, the result is positive (+) and the content of zirconium in the sample may be determined according to step 8.3.

8.3 Quantitative analysis

8.3.1 Sample size

Take one 0,2 g sample, using the analytical balance (6.2)

Duplicate the sample.

8.3.2 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 a zirconium-free blank solution.