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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)

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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.

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.

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 22863-1:2020, 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.

8.3.3 Test procedure

8.3.3.1 Digestion process

Place the sample (8.3.1) in a quartz beaker (6.4), wet it with a small amount of 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 10 ml of diluted nitric acid (5.6) to the salt.

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

Lastly, the solution shall be transferred into a 100 ml volumetric flask (6.5) and mixed with water up to the 100 ml graduation.

8.3.3.2 Calibration curve

Prepare a set of calibration samples consisting of diluted solutions of the zirconium standard diluted solution of zirconium (5.12) to concentrations of 0, 0,2, 0,5, 1,0, 2,0, 5,0 and 10,0 µg/ml. These solutions shall be placed in a group of 100,0 ml volumetric flasks (6.5) up to the 100 ml graduation. Zirconium test wavelength is set to 343,823 nm: carry out the calibration process with the Inductively Coupled Plasma Optical Emission Spectrophotometer (6.1).

The calibration curve shall be plotted in a system of coordinates based on the concentration of zirconium in abscissas (horizontal axis) and absorbances in ordinates (vertical axis).

8.3.3.3 Measurement

Set up the spectrophotometer (6.1) to the optimized working conditions according to its manufacturer's instructions.

Perform the test according to manufacturer’s instructions and record the measurement.

If the content of zirconium in the sample solutions is beyond the calibration curve, the solution (8.3.3.1) shall be diluted to an appropriate ratio and then measured again.

A blank test shall be carried out using the solution with a concentration of 0 µg/ml of zirconium (See 8.3.4)

8.3.4 Calculations

Calculate the zirconium content of the two samples using Formula (1):

$$X = \frac{(C - C_0) \cdot V \cdot f}{m} \tag{1}$$

where

X is the zirconium content of the sample, mg/kg

C is the concentration of zirconium in sample solution, µg/ml

C_0 is the concentration of zirconium in blank solution, µg/ml

V is the volume of the sample solution, ml

f is the dilution multiple of sample solution

m is the mass of the sample, g

8.3.5 Accuracy

The absolute difference between the two measured results shall not exceed 10 % of their arithmetic mean.

If the results exceed the above accuracy, the test shall be resumed, maybe using samples of increased mass.

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

9 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 22863-6:2021;

— 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 zirconium 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.

A.2 Sample size

Take one 0,5 g sample, using the analytical balance (6.2)

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 a zirconium-free blank solution.

A.4 Test procedure

A.4.1 Digestion process

The same digestion process as described in (8.3.3.1) shall be carried out to obtain the digested sample solution that is to be diluted and tested according to (A.4.2) and (A.4.3).

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 (8.3.1).

A.4.2 Dilution of the digested sample solution

Prepare 100 ml of a diluted solution of the standard diluted solution of zirconium (5.12) 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.