INTERNATIONAL STANDARD

ISO 22863-11

First edition
2022-07

Fireworks — Test methods for determination of specific chemical substances —

Part 11: Phosphorus content by inductively coupled plasma optical emission spectrometry (ICP-OES)

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Reference number
ISO 22863-11:2022(E)

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Published in Switzerland

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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 of 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 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 11: Phosphorus content by inductively coupled plasma optical emission spectrometry (ICP-OES)

1 Scope

This document specifies the test method for the determination of phosphorus content in firework compositions by inductively coupled plasma optical emission spectrometry (ICP-AES).

Such test is applied to fireworks excluding Christmas crackers, party poppers, or snaps according to ISO 25947-5. The use of white phosphorus can lead to fireworks of hazardous chemical stability that can be detected by possible self-ignition during the thermal conditioning test according to ISO 25947-4:2017, 6.16 (75,0 ± 2,5 °C for 48 h).

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 22863-1:2020, *Fireworks — Test methods for determination of specific chemical substances — Part 1: General*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The sample of pyrotechnic composition is digested and decomposed by nitric acid and hydrogen peroxide in a microwave nitrification oven, then diluted to a certain volume and sprayed into the plasma. The sample solution is used as a light source. Its spectral intensity is measured at the wavelength of phosphorus element by use of an inductively coupled plasma optical emission spectrometer. the content of elements is calculated by comparison to a calibration curve obtained from spectrometric measurements of standard solutions with different concentrations.

5 Reagents

Except as otherwise specified, the reagents of high purity shall be used. Water shall be preferably deeply demineralized by reverse osmosis process (“deionized water”).

5.1 Nitric acid, with density of 1,42 g/ml.

5.2 Hydrogen peroxide, of analytical purity.

5.3 Nitric acid, consisting of 1 part nitric acid (5.1) +19 parts water.

5.4 Phosphorus standard solution (100 µg/ml)

Weigh 0,439 g of potassium dihydrogen phosphate (KH_2PO_4, purity ≥ 99 %) using the analytical balance (6.2) and dissolve it in less than 1 000 ml of deionized water. Transfer the solution into a 1 000 ml capacity bottle (6.4), dilute it to the 1 000 ml graduation or purchase a standard solution with the same concentration.

5.5 Phosphorus standard diluted solutions for working curve preparation

Prepare a set of calibration samples consisting of diluted solutions of the phosphorus standard solution (5.4) to concentrations of 0,0, 4,0, 8,0, 16,0 and 20,0 µg/ml, as follows.

Pour 0,0 ml, 4,0 ml, 8,0 ml, 16,0 ml, 20,0 ml of phosphorus standard solution (5.4) respectively in five 100 ml flasks (6.6), dilute with nitric acid (5.3) to the 100 ml graduation, mix and transfer each solution into clean polyethylene bottles (6.5) awaiting further use in the analytical process.

Laboratory operations should comply with appropriate safety requirements for flammable and explosive materials and samples as well as strong acids and toxic materials. Operators should wear appropriate protection equipment and follow appropriate safety rules. Special measures should be taken for contingencies or uncontrollable reactions.

6 Apparatus

6.1 Inductively coupled plasma optical emission spectrometer

The purity of argon should be more than 99,99 % to provide a stable and clear plasma flame. The determination shall be carried out under the selected working conditions of the instrument. The method can be applied to the determination of trace elements in sample solutions if it meets the following requirements:

— Stability: Short-range stability C_V < 2,0 %, long-term stability C_V < 4,0 %.

— Detection limit: the detection limit of phosphorus is 0,25 g/kg.

— Calibration (working) curve: the linear correlation coefficient r of regression curve is higher than 0,999.

— Analytical line wavelength: 213,618 nm or 178,287 nm.

6.2 Analytical balance, with an accuracy 0,1 mg.

6.3 Microwave nitrification oven, capable of heating energies higher or equal to 600 W.

6.4 1 000 ml flasks.

6.5 Clean polyethylene bottles, with a capacity ≥ 100 ml.

6.6 100 ml volumetric flasks.

6.7 Nitrification beaker, with a capacity 500 ml.

6.8 Filter Nr 41 or equivalent.

7 Preparations

Preparation of the sample shall be performed according to ISO 22863-1:2020, 5.2 and 5.3.

8 Procedure

8.1 Sample size

Take one 0,1 g sample, using the analytical balance (6.2).

8.2 Nitrification process

Place the sample in a nitrification beaker (6.7), add 2 ml of deionized water and shake to wet the full sample, then add 5 ml of nitric acid (5.1). After complete reaction, add 3 ml of nitric acid (5.1) and 3 ml of hydrogen peroxide (5.2).

Place the nitrification beaker containing the solution in the microwave nitrification oven (6.3) and start the microwave nitrification process: first step for 1 min at 240 W, second step for 3 min at 360 W, third step for 5 min at 600 W.

After nitrification and cooling to ambient temperature, transfer the solution into a 100 ml volumetric flask (6.6), dilute with nitric acid (5.3) to the 100 ml graduation, mix and then filter (6.8). Transfer the solution into clean polyethylene bottle (6.5) awaiting further use in the analytical process.

8.3 Blank test

A blank spectrometric test shall be carried out using the solution with a concentration of 0,0 µg/ml of phosphorus (see 5.5).

8.4 Calibration curve

Set the phosphorus test wavelength to 213,618 nm or 178,287 nm; carry out the calibration process with the inductively coupled plasma optical emission spectrophotometer (6.1).

Determine the spectral intensity of the five phosphorus standard diluted solutions (5.5) in a sequence. Plot the measurement results in a system of coordinates based on the concentration of phosphorus in abscissas (horizontal axis, µg/ml) and spectral intensity values in ordinates (vertical axis).

Then draw the calibration curve from the five plotted points by linear regression and calculate the interception point with the horizontal axis *a*, the slope of the line *b*, and the linear correlation coefficient *r*. That coefficient should comply with the requirements of (6.1).

NOTE According to the test requirements and the use instructions given by the manufacturer of the spectrometer, the optimum analytical conditions are set up and the optimum working state of the instrument is adjusted before the test.

8.5 Sample determination

Set up the spectrometer (6.1) to the optimized working conditions according to its manufacturer's instructions and the requirements of (6.1).

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

Calculate the concentration of phosphorus in the sample solution from the calibration curve (or the equation of the regression line).

If the content of phosphorus in the sample solution is beyond the calibration curve, the sample solution (8.2) shall be diluted to an appropriate ratio and then measured again.

8.6 Parallel test

A second sample shall be processed according to (8.2) and measured according to (8.5).

9 Results calculation

The content of phosphorus shall be calculated according to Formula (1):

$$W = \frac{(c - c_0)V}{m} \quad (1)$$

where

W is the content of phosphorus in the sample in milligrams per kilogram (mg/kg) or micrograms per gram (μg/g);

c is the concentration of phosphorus in the sample solution obtained from the calibration curve in micrograms per millilitre (μg/ml).

c_0 is the concentration of phosphorus in the blank solution obtained from the calibration curve in micrograms per millilitre (μg/ml).

V is the volume of sample solution after the digestion process, in millilitres (ml);

m is the sample mass in grams (g).

The calculation results retain two significant digits.

10 Precision

When the content of phosphorus in the sample is more than 1 mg/kg (1 μg/g), the absolute difference between the two independent results obtained under repetitive conditions should not exceed 10 % of the arithmetic average value; when the content of phosphorus is less than or equal to 1 mg/kg (1 μg/g) and greater than 0,1 mg/kg (0,1 μg/g), the absolute difference between the two independent results obtained under repetitive conditions should not exceed 15 % of the arithmetic average value. When it is equal to 0,1 mg/kg (0,1 μg/g), the absolute difference between the two independent measurements obtained under repeated conditions shall not exceed 20 % of the arithmetic average.

11 Detection limit

The detection limit is 0,01 mg/kg (0,1 μg/g) and the quantitative limit is 0,03 mg/kg (0,03 μg/g) when the weighed sample quantity is 0,1 g and the volume of the dilutes solution in the digestion process is 100 ml.

12 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;

— 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.