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Fireworks — Test methods for determination of specific chemical substances —

Part 3:
Lead and lead compounds by atomic absorption

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

Partie 3: Plomb et composés du plomb par absorption atomique

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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 3:
Lead and lead compounds by atomic absorption

1 Scope

This document specifies the test method for determination of the lead content in pyrotechnic compositions of fireworks by flame atomic absorption spectrophotometry, with a minimum detection limit of 10 mg/kg.

It is generally recommended to perform a simple preliminary qualitative test to check whether a quantitative analysis is required.

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 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

The test method consists to digest the samples with hydrochloric acid, nitric acid, hydrofluoric acid and perchloric acid and then to use atomic absorption spectrophotometer to determine the lead 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 **Perchloric acid** (ρ = 1,67 g/ml).

5.3 **Nitric acid** (ρ = 1,42 g/ml).

5.4 Hydrofluoric acid (ρ = 1,15 g/ml).

5.5 Diluted hydrochloric acid (1 part acid + 11 parts water).

5.6 Diluted hydrochloric acid (1 part acid + 1 part water).

5.7 Mixed acid (4 parts nitric acid+ 1 part perchloric acid).

5.8 Standard solution of lead (1 000 µg/ml).

5.9 Standard diluted solution of lead (100 µg/ml): Pour 10,00 ml of lead standard solution (5.8) into a 100 ml flask, add diluted hydrochloric acid (5.5) up to the 100 ml graduation, then mix.

6 Apparatus

6.1 Microwave digestion oven, equipped with PTFE or other suitable digestion vessel. Operating conditions of the apparatus and the corresponding digestion times shall follow the manufacturer's instructions.

NOTE Other equipment can be used provided it complies with the same purpose.

6.2 Flame atomic absorption spectrophotometer, equipped with lead element hollow cathode lamp. Operating conditions shall follow the manufacturer's instructions.

6.3 Analytical balance, accuracy 0,1 mg.

6.4 Electric hot plate, capable of reaching 500 °C.

7 Preparations

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

8 Analysis steps

8.1 Sample size

Take 1,0 g sample, using the analytical balance (6.3).

Duplicate the sample.

8.2 General requirement

The analysis of the two samples shall be carried out simultaneously.

For error correction, a blank test shall be carried out in parallel with a lead-free blank solution.

8.3 Test procedure

8.3.1 Digestion process

8.3.1.1 Samples not containing titanium

Wet the sample parts (8.1) with a small amount of water, add diluted hydrochloric acid (5.6) dropwise until the violent reaction is completed, then add 8 ml of mixed acid (5.7), stir, close tightly the digestion vessel, put it into the microwave digestion oven (6.1) and complete the digestion process during 30 minutes at 210 °C (See Annex A).

Upon completion of the digestion process, the digestion vessel shall be cooled down to room temperature and then the solution shall be cleared of any solid residue by filtering it and transferred to a PTFE beaker.

Heat the cleared solution until it boils and reaches an almost dry salt state on an electric hot plate (6.4).

After cooling, 10 ml of diluted hydrochloric acid (5.6) shall be added 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 and mixed with water up to the 100 ml graduation.

8.3.1.2 Samples containing titanium

Place each sample (8.1) into a microwave digestion vessel, wet it with a small amount of water, add diluted hydrochloric acid (5.6) dropwise until the violent reaction is completed.

Then add 3 ml of hydrofluoric acid (5.4) and 5 ml of mixed acid (5.7), shake, close tightly the digestion vessel, put it into the microwave digestion oven (6.1) and complete digestion according to the settled operation procedures.

Upon completion of the digestion procedure, the digestion vessel shall be cooled down to room temperature and then the solution shall be transferred into a polytetrafluoroethylene beaker and heated until it reaches an almost dry salt state on an electric hot plate (6.4).

After cooling, add 10 ml of diluted hydrochloric acid (5.6) and dissolve the salts with heating, and then cool it again.

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

8.3.2 Calibration curve

Prepare a set of calibration samples by dilution of the standard diluted solution of lead (5.9) to concentrations of 0, 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 up to the 100 ml graduation. Carry out the calibration process with the spectrophotometer (6.2) according to its manufacturer's instructions.

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

8.3.3 Measurement

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

Perform the test according to (8.2) and record the measurement.

If the content of lead in the sample solutions is beyond the calibration curve, the solution (8.3.1) shall be diluted to an appropriate ratio and then measured again.

9 Calculations

Calculate the lead content of the two samples using the following formula:

$$X = \frac{(C - C_0).V.f}{m}$$

where

X is the lead content of the sample (mg/kg);

C is the measured concentration of lead in the sample solution (μg/ml);

C_0 is the measured concentration of lead in the lead-free blank solution (μg/ml);

V is the volume of the sample solution (ml);

f is the dilution factor of the sample solution;

m is the mass of the sample (g).

Calculation results shall be expressed with two significant digits.

Standard addition method may be used instead of the methods specified in Clause 7 and Clause 8 (see Annex C).

10 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.

11 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 22863-3:2020;
- necessary description of the sample and how it was obtained according to ISO 22863-1;
- results of the analysis;
- any anomaly that occurred while performing the tests.

Annex A
(informative)

Reference working conditions of microwave digestion oven

Microwave digestion oven is used to digest samples at high temperature and high pressure. For the sake of safety, strictly comply with the use instructions and specifications provided by the manufacturer of the equipment and the highest temperature and pressure shall not exceed the specified range. The microwave digestion procedure can refer to Table A.1. Since different manufacturers provide different designs of microwave digestion ovens, give priority to the digestion procedure provided by manufacturer.

Table A.1 — Reference working conditions of microwave digestion oven

Power (W)	**Rising time** (minutes)	**Pressure** (hPa)	**Temperature** (°C)	**Holding time** (minutes)
1 000	30	55	210	30
NOTE Different models of microwave digestion ovens from different manufacturers can have different digestion procedures (See supplied user's instructions).				

Annex B
(informative)

Reference working conditions of atomic spectrometer

Table B.1 — Reference working conditions of atomic spectrometer

Wavelength (nm)	Slid width (nm)	Hollow cathode lamp (mA)	Air flow rate (l/min)	Acetylene flow (l/min)
283,3	0,5	4	12,5	2,0

Annex C
(informative)

Standard addition method

C.1 General

This second method eliminates the “matrix” effects that result from the digestion process where other ions corresponding to other compounds than lead 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.

C.2 Sample size

Take one 0,5 g sample, using the analytical balance (6.3).

Duplicate the sample.

C.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 lead-free blank solution.

C.4 Test procedure

C.4.1 Digestion process

The same digestion process as described in (8.3.1) shall be carried out to obtain the digested sample solution that is to be diluted and tested according to the following clauses, (C.4.2) and (C.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 (C.2).

C.4.2 Dilution of the digested sample solution

Prepare 100 ml of a diluted solution of the standard diluted solution of lead (5.9) 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.