INTERNATIONAL STANDARD

ISO 22863-5

First edition
2021-03

Fireworks — Test methods for determination of specific chemical substances —

Part 5:
Analysis of lead and lead compounds by inductively coupled plasma spectrometry (ICP)

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

Partie 5: Analyse du plomb et de ses composés par spectrométrie à plasma à couplage inductif (ICP)

Reference number
ISO 22863-5:2021(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 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 5:
Analysis of lead and lead compounds by inductively coupled plasma spectrometry (ICP)

1 Scope

This document specifies the method for the determination of the content of lead and lead compounds in pyrotechnic compositions of fireworks by Inductive coupled plasma 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 ICP-optical emission spectrometry (ICP-OES) is a spectrometric technique used to determine trace metal elements in aqueous solutions. The sample solution is aspirated continuously into an inductively coupled, argon-plasma discharge, where analytes of interest are converted to excited-state, gas-phase atoms or ions. As the excited-state atoms or ions return to their ground state, they emit energy in the form of light at wavelengths that are characteristic of each specific element. The intensity of the energy emitted at the chosen wavelength is proportional to the amount (concentration) of that element in the sample. Thus, by determining which wavelengths are emitted by the sample and their respective intensities, the elemental composition of the sample relative to a reference standard may be quantified. For complex solid samples, an appropriate acid digestion prior to analysis is necessary to ensure the ICP-OES analysis bears less bias.

A representing sample is digested with nitric acid, filtered and made up to a definite volume in acidic medium. Determination of the lead content of the sample solution is carried out using ICP-OES. The solution can be diluted to an appropriate volume so the concentration of lead content fits in with the calibration curve of the equipment.

5 Equipment

5.1 Inductively coupled, argon-plasma, optical emission spectrometer, with the operational accessories recommended by its manufacturer.

5.2 Analytical balance, capable of weighing to ±0,1 mg.

5.3 Oven, capable of keeping temperature at (105 ± 5) °C.

5.4 Crucible, of size 25 ml.

5.5 Volumetric flasks, 1 l and 100,0 ml.

5.6 Common laboratory wares which are clean for the purpose.

6 Standards and reagents

6.1 Standard lead (Pb) stock solution, commercially available in different concentrations, or prepared by dissolving exactly 1,598 5 g of lead nitrate $Pb(NO_3)_2$ (99,7 % pure, dried and conditioned at 105 °C before use) in a minimum amount of HNO_3 (50 %) (6.2). Add 10 ml HNO_3 (50 %) (6.2) and dilute to volume in a 1 l volumetric flask (5.5) with reagent water (6.3).

Lead salts are toxic if inhaled or swallowed. Wash hands thoroughly after handling.

6.2 Nitric acid, HNO_3 (50 %) AR Grade.

6.3 Reagent water, distilled or ionized water, which shall be free of interference.

7 Calibration

If the manufacturer’s calibration procedure is not available, the preparation of a calibration curve shall be adopted and carried out as follows:

a) Prepare ICP calibration standard solutions of (0,00, 0,25, 0,50, 1,00, 5,00 and 10,0) µg/ml by dilution of a 1 000 µg/ml stock standard solution of lead (Pb) (6.1).

b) Prepare an internal standard solution of 2 µg/ml yttrium in 2 % nitric acid using a 1 000 µg/ml stock standard (option).

Internal standard (Y) is recommended to be used if the analyte is at trace amount ppm level, otherwise comparison with an external standard (Pb) is sufficient.

Set the equipment (5.1) using the following conditions:

- Plasma flow: 15,0 l/min.
- Nebulizer flow: 0,75 l/min.
- Pump speed: 20 r/min.
- Auxiliary flow: 1,5 l/min.
- Wavelengths: Lead (Pb) 220,353 nm., Yttrium (Y) 324,228 nm (option)[1].
- Power: 1,30 kW.

---

[1] An internal standard such as 2 µg/ml yttrium should be used.

Obtain a relation between the contents of lead in the calibration standard solutions and the X-ray intensities with a quadratic regression formula or a linear regression formula in accordance with the least squares method, as given by Formula (1):

$$W_{Pb} = aI^2_{Pb} + bI_{Pb} + c \quad (1)$$

where

W_{Pb} is the content of lead, Pb (mg/kg);

I_{Pb} is the X-ray intensity of lead, Pb;

$a, b, c,$ are coefficients (in the case of the linear function formula, $a = 0$).

8 Procedure

Dry the weighed sample, m_0 mg in the 105 °C oven (5.3) until it reaches a constant mass, m_1 mg.

Prepare the analyte aliquot: Weigh *m* mg (e.g. 1 g) from the dried sample in a tared crucible (5.4) with the analytical balance (5.2) and digest it with nitric acid (6.2) three times separately.

Due to the rapid advances in microwave technology, microwave digestion is possible to obtain analyte aliquots, consult the manufacturer's recommended instructions for guidance on their microwave digestion system. (Option).

Filter the aliquots and combine all three aliquots together in the volumetric flask (100,0 ml) (5.5), make up to the mark with distilled water for determination.

Turn on the ICP instrument (5.1) and wait until the instrument become thermally stable before use.

Perform the wavelength calibration or torch alignments following the manufacturer operation manual.

Ensure the following element and wavelength are selected in analytical mode: Pb 220,353 nm. The other Pb line 217,00 µm can be used to ensure spectral interferences are not occurring during the analysis.

Perform a calibration using the calibration blank solution and at least 3 standard calibration solutions. Calibrations should be performed at a minimum of once a day when used for analysis, or each time the instrument is set up. Results for each standard should be within 5 % of the true value. If the values do not fall within this range, a recalibration is necessary.

Analyse the sample solution immediately after the calibration. The analysed value of Pb, c_1, should be within ±10 % of the expected value. If the Pb value is outside the ±10 % limit, a recalibration is required either by change of the sample amount or by change of the calibration concentration range.

At least one certified reference material (CRM) should be analysed with each batch of samples. The CRM should have a known amount of Pb and be of a material similar to the test specimen. Analysis recoveries should be within ±15 % of the expected values. If recoveries are outside this limit, the source of the problem should be identified and resolved before continuing analyses.

Dilute any samples that have Pb concentration values exceeding 1,5 times that of the highest calibration standard solution and reanalyse, determine Pb content.

Prepare a method blank test with the calibration standard solution of 0,00 µg/ml throughout the entire sample preparation and the instrument determination process, c_0.

Results of total Pb concentration, $c_{\mathrm{Pb,tot}}$, are calculated and reported in mg/kg, as given by Formula (2):

$$c_{\mathrm{Pb,tot}} = 0{,}1\ (c_1 - c_0)\ V_\mathrm{d}/m\ [1 - (m_0 - m_1)/m_0] \tag{2}$$

where

c is the concentration of Pb detected (µg/ml) (c_1 for the sample solution and c_0 for the blank solution);

d is the dilution volume (ml);

m is the mass of the dried sample digested (mg); the concentration can be calculated taking into account the present moisture.

9 Accuracy and precision

The accuracy and precision of this test method can be developed on the basis of interlaboratory study of controlled reference samples in future. Based on other applicable experimental data, a 10 % relative standard deviation of the result and a 90 % recovery are acceptable.

A potential interference to determine Pb arise from aluminium (Al) if it exists at more than 1 % by weight. Acceptable data is to be confirmed as soon as there is an inter-laboratory correlation.

10 Test report

The test report shall include but not be limited to the following information:

- name and address of the testing laboratory;
- date of issuing the test report;
- a reference to this document, i.e. ISO 22863-5:2021;
- 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.