
**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)*

ISO 22863-5:2021

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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.

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).

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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 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 $\pm 0,1$ 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 $\text{Pb}(\text{NO}_3)_2$ (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:

- Prepare ICP calibration standard solutions of (0,00, 0,25, 0,50, 1,00, 5,00 and 10,0) $\mu\text{g}/\text{ml}$ by dilution of a 1 000 $\mu\text{g}/\text{ml}$ stock standard solution of lead (Pb) (6.1).
- Prepare an internal standard solution of 2 $\mu\text{g}/\text{ml}$ yttrium in 2 % nitric acid using a 1 000 $\mu\text{g}/\text{ml}$ 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 $\mu\text{g}/\text{ml}$ yttrium should be used.