
**Fireworks — Test methods for
determination of specific chemical
substances —**

**Part 4:
Analysis of lead and lead compounds
by X-ray fluorescence spectrometry
(XRF)**

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

*Partie 4: Analyse du plomb et de ses composés par spectrométrie de
fluorescence des rayons X (XRF)*



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Foreword

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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 4:

Analysis of lead and lead compounds by X-ray fluorescence spectrometry (XRF)

1 Scope

This document specifies the method for the determination of the content of lead and lead compounds in pyrotechnic compositions of fireworks by X-ray fluorescence spectrometry (XRF).

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

In XRF analysis, a source of X-rays irradiates a sample. The source can be an X-ray tube or a sealed radioisotope. When a sample is irradiated with X-rays, the source X-rays may undergo either scattering or absorption by the sample atoms. When an atom absorbs the source X-rays, the incident radiation can dislodge electrons from the innermost shells of the atom, creating vacancies. Electrons from outer shells will fill the inner shell vacancy and emit X-ray photons. The energy of the emitted X-ray depends on the difference in energy of the shell with the initial vacancy and the energy of the electron that fills the vacancy. Each atom has specific energy levels, so the emitted radiation is characteristic of that atom. By measuring the energy of the radiation emitted it is possible to identify which elements are present in a sample. By measuring the intensity of the emitted energies, it is possible to quantify how much of a particular element is present in a sample.

The test method uses energy dispersive X-ray fluorescence (EDXRF) spectrometry for the detection and quantification of lead (Pb) in homogenous pyrotechnic compositions. The method is applicable for pyrotechnic compositions containing lead mass fractions in the range of (100 to 50 000) mg/kg. For mass fractions that may be smaller than 100 mg/kg (100 ppm), other test methods shall be applied.

The geometry of the tube-sample-detector assembly needs to be kept constant. For that reason, the sample is normally prepared as a flat disc of compressed composition and then is placed at a small

distance from the tube window, with tight tolerances for this placement and for the flatness of the surface of the sample disk to maintain a repeatable X-ray flux.

5 Equipment

5.1 Niton XL3t 900S portable XRF spectrometer or equivalent XRF analyser.

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 Common laboratory wares which are clean for the purpose.

6 Standards and chemicals

6.1 National Institute of Standards and Technology (NIST) Standard Reference Material, NIST 2780#180-625 (Pb-0,577 %).

6.2 National Institute of Standards and Technology (NIST) Standard Reference Material, RCRA#180-436 (Pb, As in range of 400-600 mg/kg).

6.3 Silica, RCRA#180-472, SiO₂ >95 % (Pb, As, Hg, Cu < LOD).

6.4 General laboratory reagents, AR Grade.

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 some flat disks of standard reference Pb (6.1 and 6.2) at different contents of lead. Using the XRF spectrometer (5.1), measure the X-ray intensities of the standard disc to obtain a calibration curve. Obtain a relation between the contents of the standard reference Pb 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):

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

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 equation, $a = 0$).

When the calibration uses an outside source, verify the calibration by analysing more than one reference material.

8 Procedure

Turn on and warm up the XRF analyser for the recommended time, usually at least 15 min (consult the manufacturer's manual before operating any XRF).