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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)

PROOF/ÉPREUVE

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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).

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

For an explanation on 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 the following URL: 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.

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 reference

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 ±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):

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

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 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).

Calibrate the XRF analyser (5.1) following the manufacturer operation manual with NIST standard reference materials (See Clause 7).

Dry a sufficient amount of sample (around 10 g) in an oven (5.3) at 105 °C for at least 2 h (preferably 4 h) and transfer it to a desiccator (5.4 and 6.3) at room temperature for at least 2 h or until it reaches a constant mass. Care shall be taken when transferring hot pyrotechnic compositions, using necessary personal protective equipment such as a shield. Check visually whether this sample is homogeneous enough before going to the next step.

Prepare the sample disc with pyrotechnic composition taken from this homogenous sample following the manufacturer operation manual. Maintain the surface as smooth as possible so that the probe will have good contact with the surface.

Analyse the sample disc for a minimum measuring time of 60 s.

Repeat the measurement at another two different locations of the disc.

Report the lead (Pb) content of the sample calculated from the XRF analyser with the assistance of the calibration procedure if necessary, using units of mg/kg, rounded to the nearest 1 mg/kg. Significant digits can be up to 3 figures depending on the range of analysis.

Calculate the dried mass by subtracting the moisture content and report the result based on the dried sample mass.

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 the future. For small measured values, it is commonly acceptable to use 25 % relative standard deviation for such newly modified technology.

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 22863-4:—;

— 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.