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Fireworks — Test methods for determination of specific chemical substances —

Part 13: Qualitative detection of elemental metals in firework compositions

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

Partie 13: Détection qualitative des métaux élémentaires dans les compositions pyrotechniques d'artifices

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Foreword

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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.

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Fireworks — Test methods for determination of specific chemical substances —

Part 13: Qualitative detection of elemental metals in firework compositions

1 Scope

This document specifies a method for the qualitative detection of reactive elemental metals (e.g. Mg, Al, Be, Mn, Zn, Fe, Co, Ni, Sn) used in compositions of fireworks.

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*

ISO 22863-16, *Fireworks—Test methods for determination of specific chemical substances— Part 16: Procedure for identification of report or burst charges*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 22863-1 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle of the method

Based on the property of the reactive metal to be oxidized with production of hydrogen under a certain pH value, the sample is reacted with a hydrochloric acid solution, nitric acid, hydrofluoric acid or sodium hydroxide solution in a closed vessel, and the presence of the reactive metal is determined by detecting the hydrogen that is generated.

5 Reagents

Except as otherwise specified, reagents of high purity shall be used. Water shall be demineralized by reverse osmosis process (“deionized water”).

5.1 Ethanol (AR)

5.2 Ethanol solution (50 %, v/v)**:**1 part ethanol (5.1)+ 1 part deionized water.

5.3 Hydrochloric acid (AR, ρ = 1,19 g/ml).

5.4 Diluted hydrochloric acid (1 part hydrochloric acid (5.3)+ 1 part deionized water).

5.5 Hydrofluoric acid (AR, 40 % v/v, to be kept in a plastic bottle).

WARNING — Specific personal protective equipment shall be worn when operating with hydrofluoric acid.

5.6 Sodium hydroxide (AR).

5.7 Diluted sodium hydroxide, weight 20 g Sodium hydroxide (5.6) and dissolve it in 100 ml of deionized water.

5.8 Nitric acid (AR, 65 % to 70 % v/v).

5.9 Mixed hydrochloric acid and nitric acid (3 parts hydrochloric acid (5.3) + 1 part nitric acid (5.8), v/v). This mixture shall be prepared less than 30 min before use.

6 Apparatus

6.1 Surface dish, Ø100 mm.

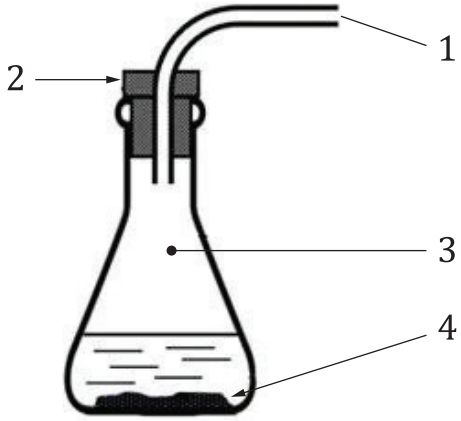
6.2 Hydrogen detector, 0 ppm to 1 000 ppm, resolution ≤ 10 ppm, accuracy ≤ 3 %.

6.3 Analytical Balance, accuracy 0,01 g.

6.4 Gas generator with glass Erlenmeyer flask, 200 ml (see Figure 1).

6.5 Gas generator with PTFE Erlenmeyer flask, 200 ml (see Figure 1).

6.6 Electric hot plate, capable of reaching 300 °C.



Key

1 connection to hydrogen detector
2 rubber stopper
3 erlenmeyer
4 sample

Figure 1 — Gas generator

7 Samples

Disassemble the firework sample in accordance with the method described in ISO 22863-1 or ISO 22863-16 for report effect units and burst charges.

8 Qualitative identification

8.1 Preliminary step

Take a sample of approximately 0,1 g, using the analytical balance (6.3). Place the sample in the centre of the surface dish (6.1), add 2 or 3 drops of ethanol solution (5.2) on it, wait for the liquid to spread within the sample.

Add a few drops of diluted hydrochloric acid (5.4) at the centre of the wetted sample, let it spread within the sample. Observe whether any bubbles are generated. If so, record the intensity of bubble generation.

8.2 Precise identification

8.2.1 For samples that significantly produce gas in 8.1.

Option 1: Take a 0,2 g to 0,5 g sample using the analytical balance (6.3). The amount of sample is determined according to the intensity of the bubble generation: the more intense the more intense the bubble generation is, the smaller sample shall be, and vice versa. Place the sample in a glass gas generator (6.4), add a small quantity of ethanol solution (5.2). Shake the Erlenmeyer flask. Close it tightly with the rubber stopper.

Option 2: Take a 0,5 g to 1 g sample using the analytical balance (6.3), Place the sample in a glass Erlenmeyer flask (6.4), add a few drops of ethanol solution(5.2). Shake the Erlenmeyer flask. Add in 10 ml of mixed hydrochloric acid and nitric acid (5.9). Close it tightly with the rubber stopper. Heat it until it boils using the electric hot plate (6.6).

Turn on the hydrogen detector (6.2) and follow the instrument operating instructions to prepare for the test. Add in 15 ml of diluted hydrochloric acid (5.4) and gently shake the Erlenmeyer. If hydrogen production

is detected, it is determined that an active metal is present in the sample and, if not, complete the operations described in 8.2.2 below.

8.2.2 For samples that produce very little gas in 8.1.

Take a 0,5 g to 1 g sample using the analytical balance (6.3). Place the sample in a PTFE gas generator (6.5), add a few drops of ethanol solution (5.2). Shake the Erlenmeyer flask. Add in 5 ml of hydrofluoric acid (5.5). Close it tightly with the rubber stopper.

Turn on the hydrogen detector (6.2) and follow the instrument operating instructions to prepare for the test. Add in 15 ml of diluted hydrochloric acid (5.4) and gently shake the Erlenmeyer. If hydrogen production is detected, it is determined that there is an active metal in the sample and, if not, determine that there is no reactive metal in the sample.

8.2.3 Possible presence of aluminium.

If no bubbles are observed in the preliminary step (8.1), the samples shall be tested for aluminium with a sodium hydroxide solution (5.7): add a few drops of sodium hydroxide at the centre of the wetted sample, let it spread within the sample. Observe whether any bubbles are generated. If so, record the intensity of bubble generation.

Turn on the hydrogen detector (6.2) and follow the instrument operating instructions to prepare for the test. If hydrogen production is detected, it is determined that there is an active metal in the sample and, if not, determine that there is no reactive metal in the sample.

9 Test report

The test report shall include at least the following information:

— name and address of the testing laboratory;

— date of issue or date of signature;

— reference to this standard, i.e. ISO 22863-13:2025;

— necessary description of the sample and how it was obtained according to ISO 22863-1 or ISO 22863-16;

— the identification of qualitative analysis (elemental metals present or elemental metals not present);

— results of the analyses;

— any anomaly that occurred while performing the tests;

— Name and position/title of person conducting the tests;

— Signature of person conducting the test.