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

**Part 7:
Chlorates content by chemical
titration analysis**

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

Partie 7: Teneur en chlorates par analyse chimique par titrage

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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.

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This document was prepared by Technical Committee ISO/TC 264, *Fireworks*, WG 4.

A list of all the 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.

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Introduction

The ISO 22863 series consists of the following parts, under the general title “Test methods for determination of specific chemical substances”:

- ISO 22863-1, Fireworks – Test methods for determination of specific chemical substances – Part 1: General
- ISO 22863-2, Fireworks – Test methods for determination of specific chemical substances – Part 2: Hexachlorobenzene by gas chromatography
- ISO 22863-3, Fireworks – Test methods for determination of specific chemical substances – Part 3: Lead and lead compounds by atomic absorption
- ISO 22863-4, Fireworks – Test methods for determination of specific chemical substances – Part 4: Lead and lead compounds by X-ray fluorescence spectrometry (XRF)
- ISO 22863-5, Fireworks – Test methods for determination of specific chemical substances – Part 5: Lead and lead compounds by inductive coupled argon plasma optical emission spectrometry (ICAP-OES)
- ISO 22863-6, Fireworks—Test methods for determination of specific chemical substances – Part 6: Zirconium with a particle size of less than 40 µm by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
- ISO 22863-7, Fireworks—Test methods for determination of specific chemical substances – Part 7: Chlorates content by Chemical Titration Analysis
- ISO 22863-8, Fireworks—Test methods for determination of specific chemical substances – Part 8: Arsenic content by hydride generation-atomic fluorescence spectrometry
- ISO 22863-9, Fireworks—Test methods for determination of specific chemical substances – Part 9: Mercury content by hydride generation-atomic fluorescence spectrometry
- ISO 22863-10, Fireworks — Test methods for determination of specific chemical substances - Part 10: Nitrogen content in nitrocellulose by Iron(II) sulphate titration
- ISO 22863-11, Fireworks — Test methods for determination of specific chemical substances – Part 11: Phosphorus content by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
- ISO 22863-12, Fireworks — Test methods for determination of specific chemical substances – Part 12: Picrates and picric acid by high performance liquid chromatography

Fireworks — Test methods for determination of specific chemical substances —

Part 7: Chlorates content by chemical titration analysis

1 Scope

This document specifies the qualitative and quantitative analysis methods for the determination of the chlorates content in pyrotechnic compositions by chemical titration analysis, with the minimum detection limit (Cl O_3^{-1}) of 1 000 mg/kg.

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

Qualitative analysis: if any, the chlorates in the sample are dissolved in water, and then the presence of chlorates is detected by special coloration reaction of aniline hydrochloride under strong acidic conditions.

Quantitative analysis: After the sample is extracted by ethanol, the chlorate(s) is(are) dissolved in hot water and reacted totally with an excess of ammonium ferrous sulphate solution. The remaining content of ammonium ferrous sulphate in the sample test solution is then titrated by a potassium dichromate standard solution. The initial content of chlorate in the sample test solution is calculated from the difference between (1) the volume of consumed potassium dichromate standard solution in the sample solution titration and (2) the volume of consumed potassium dichromate in a blank titration of a solution that contains the same quantity of ammonium ferrous sulphate as added to the sample test solution.

5 Safety Requirements

Laboratory operations should comply with appropriate safety requirements: peculiarly, for flammable, explosive, highly toxic and other dangerous materials and samples as well as strong acids, strong alkali and other corrosive materials, operators should wear appropriate protection equipment and follow appropriate safety rules.

Special measures should be taken for contingencies or uncontrollable reactions.

6 Reagents

Unless otherwise stated, only confirmed as analytical reagent, distilled water or deionized water or equivalent purity water shall be used.

6.1 Hydrochloric acid ($\rho = 1,19$ g/ml)

6.2 Aniline Hydrochloride Solution: 5 g of aniline hydrochloride dissolved in 50 ml of hydrochloride acid (6.1)

6.3 Ethanol Solution (1 part Ethanol + 1 part water)

6.4 Barium chloride solution (15 g of barium chloride + 3 g of potassium hydroxide dissolved in 100 ml of water)

6.5 Sulfuric acid ($\rho = 1,84$ g/ml)

6.6 Phosphoric acid ($\rho = 1,87$ g/ml)

6.7 Ammonium Ferrous Sulphate Solution (39,2 g/l)

6.8 Mixed acid (20 ml) of sulfuric acid (6.5) and 5 ml of phosphoric acid (6.6) (slowly added in 50 ml of water, cooled before use)

6.9 Sodium di-aniline sulfonate solution 0,5 % (0,5 g of sodium di-aniline sulfonate dissolved in 100 ml of water, add 1 drop of sulfuric acid (6.5))

6.10 Potassium dichromate: pure (primary standard reagent quality)

6.11 Potassium dichromate standard titration solution: $c(1/6 K_2Cr_2O_7) = 0,1$ mol/l, prepared as follows:

Place 4,9035 g of potassium dichromate in a 250 ml beaker, add the right amount of water until complete dissolution, and then place all into the 1 000 ml volume bottle, add water to the scale, mix evenly.

7 Apparatus

7.1 Analytical Balance: accuracy 0,1 mg

7.2 Filter Paper

7.3 Beaker: 200 ml

7.4 Electric hot plate: capable of reaching 300 °C

7.5 Sand core crucible: filter: aperture $3 \mu m \sim 4 \mu m$

7.6 Suction filter bottle: 500 ml

7.7 Volume bottle: 250 ml

7.8 Graduated pipette: 50 ml

7.9 Erlenmeyer: 300 ml

7.10 Acid type burette: 50 ml

8 Preparations

Preparation of samples shall be performed according to ISO 22863-1, 5.3.2.2.

9 Analysis

9.1 General

The analysis procedure may start from step 9.2. If the test result in qualitative analysis is negative (-), then conclude the absence of chlorates in the sample. Otherwise, continue to step 9.3 of the quantitative analysis procedure to determine the content of chlorates. Where appropriate, step 9.2 of the qualitative analysis may also be omitted and the quantitative analysis directly started from step 9.3.

9.2 Qualitative analysis

9.2.1 Sample size

Take 0,1 g sample, using the analytical balance (7.1).

9.2.2 Digestion process

Place the sample (9.2.1) in the centre of the filter paper (7.2), add 1 drop of water on it, wait for the water to spread within the sample.

Add 1 drop of aniline hydrochloride solution (6.2) at the centre of the wetted sample, let it spread within the sample to the sides of the filter paper. If the filter paper doesn't appear blue with a purple ring, the absence of chlorates can be concluded and the result is negative (-); otherwise, the result is positive (+) and then the content of chlorates in the sample shall be determined according to step 9.3.

9.3 Quantitative analysis

9.3.1 Sample size

Take one 1,0 g sample, using the analytical balance (7.1)

Duplicate the sample.

9.3.2 General requirement

The analysis of the two samples shall be carried out immediately one after the other.

9.3.3 Test procedure

Place the sample (9.2.1) in a beaker (7.3), wet it with a small amount of ethanol solution (6.3), add 10 ml of barium chloride solution (6.4), add 20 ml of water, heat to boiling on the electric hot plate (7.4) for 2 minutes~3 minutes. Remove the solution from the electric hot plate and wait for 40 minutes.

Transfer the solution into the sand core crucible (7.5) and let it filter through it. Wash the residue with water 5~6 times successively to obtain a total of 100 ml of washing water in a beaker (7.1). Filtrate