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Fireworks — Test methods for determination of specific chemical substances —

Part 7: Chlorates content by chemical titration analysis

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

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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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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 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.

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 (ClO_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 (ρ = 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 (ρ = 1,84 g/ml)

6.6 Phosphoric acid (ρ = 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 μm~4 μm

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 22863-1:2021, 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

the washing water into the suction filter bottle (7.6). Pour the content of the suction filter bottle in a volume bottle (7.7).

Wash the beaker and the suction filter bottle and pour the used washing water in the same volume bottle as before. Cool and add water to the 250 ml scale and then mix.

Take 50 ml of the solution from the previous step with a graduated pipette (7.8) place it in the erlenmeyer (7.9), add 50 ml of ammonium ferrous sulphate solution (6.7), add 40 ml of mixed acid (6.8), mix, wait for 10 minutes, add 2 ml of sodium di-aniline sulfonate (6.9) and then add potassium dichromate standard solution (6.10) until the solution turns purple as the end point of the titration process.

A blank test shall be carried out in parallel with a chlorate-free blank solution made of water coming from the same source as the water used for the above washing process with addition of ammonium ferrous sulphate solution (6.7), mixed acid (6.8) and sodium di-aniline sulfonate (6.9) in the same proportions as for the sample test solution.

9.3.4 Calculations

Calculate the chlorate content (in % ClO_3^{-1}) of the two samples W using Formula (1):

$$W = \frac{(V - V_0) \cdot c \cdot M}{6m} \tag{1}$$

where

W is the chlorate content of the sample, %

c is the accurate value of potassium dichromate standard solution, mol/l

V_0 is the volume of potassium dichromate standard solution in blank test, ml

V is the volume of potassium dichromate standard solution in sample test, ml

M is the molar mass of the root of chlorate (M = 83,45 g/mol)

m is the mass of the sample, g

The arithmetic mean is the test result and is given with two decimal digits.

9.3.5 Accuracy

The absolute difference of the two results shall be calculated and conform with Table 1.

Table 1 — Absolute difference of parallel results

Quality fraction of chlorate	Absolute difference of parallel results
< 20 %	≤ 0,20 %
20 %~50 %	≤ 0,30 %
> 50 %	≤ 0,50 %

10 Test report

The test report shall include at least the following information:

— name and address of the testing laboratory;

— date of issue;

— reference to this document, i.e ISO 22863-7:2021;

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

— the identification of qualitative analysis and quantitative analysis;

— results of the analysis;

— any anomaly that occurred while performing the tests.

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