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First edition

Fireworks — Test methods for determination of specific chemical substances —

Part 10: Nitrogen content in nitrocellulose by Iron(II) sulfate titration

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

Partie 10: Teneur en azote dans la nitrocellulose par titrage du sulfate de fer (II)

PROOF/ÉPREUVE

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Contents

Page

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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 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.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

This document was prepared by Technical Committee ISO/TC 264, *Fireworks, WG4*

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.

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 10: Nitrogen content in nitrocellulose by Iron(II) sulfate titration

1 Scope

This document specifies the method for determination of the nitrogen content in nitrocellulose within pyrotechnic compositions of fireworks by Iron(II) sulphate titration.

2 Normative reference

The following referenced documents are indispensable for the application of this document. For dated reference documents, only dated editions are applicable to be used. For undated references, the latest edition of the referenced document (including any subsequent amendments) applies.

ISO 22863-1, *Fireworks — Test methods for determination of specific chemical substances — Part 1: General*

3 Terms and definitions

No terms and definitions are listed in this document.

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

In a preliminary step, samples of pyrotechnic compositions containing nitrocellulose are submitted to special treatment to make them suitable to the determination, without error, of the nitrogen content of nitrocellulose. Thereby, the other molecules that possibly contain nitrogen atoms such as nitrates - except nitrocellulose (cellulose nitrate) - shall be removed from the sample before that determination.

The remaining cellulose nitrate in the sample is released by concentrated sulphuric acid, forming nitric acid which is then titrated with Iron(II) sulphate ($FeSO_4$) according to the following reaction:

$$4\ FeSO4 + 2\ HNO3 + 2\ H2S04 \rightarrow N2O3 + 2\ Fe2(SO4)3 + 3\ H2O$$

The reaction is followed by potentiometry. The titration curve obtained exhibits an inflexion point corresponding to the quantitative neutralization of the nitrogen radicals of the sample.

5 Reagents and materials

All reagents shall be of recognized analytical grade. Verify whether the reagents are applicable for this specific purpose and free of interfering compounds.

4.1 Sulphuric acid (H_2SO_4) Chemical Purity 94-97 %

4.2 Potassium Nitrate (Pure)

4.3 Iron(II) sulphate ($FeSO_4 \cdot 7H_2O$) Crystals (Pure)

4.4 Distilled Water

4.5 Acetone (Pure and anhydrous)

6 Apparatus

6.1 Any (manual or automatic) pH meter capable of measuring pH with a precision better than 0,1.

6.2 Platinum Electrode for pH measurement

6.3 Glass, graphite or tungsten Electrode for pH measurement (reference electrode).

6.4 Magnetic Stirrer base with stirring bar

6.5 Blender

6.6 Cooling bath (e.g. ice bath) and/or other mechanical cooling system

6.7 Opaque vessel

6.8 Agate mortar

6.9 150 ml and 200 ml beakers

6.10 Petri dishes

6.11 250 ml Erlenmeyer

6.12 Antistatic plastic bags

6.13 Balance, accurate to 0,0001 gram or 1/10000

6.14 Desiccator with drying agent (with colour indicator).

6.15 Laboratory reflux apparatus

6.16 100 °C drying oven.

6.17 40 °C drying oven.

6.18 50 °C drying oven.

6.19 Timer (seconds).

6.20 Tissue paper or filtration paper

7 Preparation of the sample

7.1 Preliminary precaution

Before preparing the sample, all information on the chemical content of the pyrotechnic composition to be tested (at least chemical name and proportions) shall be given by the manufacturer.

The determination of the nitrogen content of the nitrocellulose requires that all other molecules that contain nitrogen atoms are extracted from the sample before the titration process. In most cases, such molecules are nitrates that can be extracted by dissolution with hot distilled water: depending on the solubility of nitrates in water and their percentage in the composition, such operation may need an iterative process as described in 7.3.

When other nitrogen-containing molecules are present and are not soluble in water, a specific solvent shall be used provided it does not dissolve nitrocellulose.

The extraction of nitrocellulose from the sample is carried out by dissolution in acetone. Such operation is applied to the sample after elimination of all nitrates and nitrogen-containing molecules if any. It shall then be checked whether acetone is also a solvent for the other remaining ingredients of the pyrotechnic composition to be tested. In such case, acetone shall be replaced by another solvent which, among the ingredients of the pyrotechnic composition, only dissolves nitrocellulose.

At the end of Step Three (7.4), the last solid residue should then only be nitrocellulose.

7.2 Step One

Take 10 grams of the composition containing nitrocellulose, accurately weighed to 1/10000 (6.13). Split it in three equal quantities and place each one in three antistatic plastic bags (6.12). Close the bags tightly.

Place the quantity of one first bag in a Petri dish (6.10) and then place the whole in an oven at 40 °C (6.17) for 12 hours.

Grind the dried composition in an agate mortar (6.8) to obtain a fine powder of grain size less than 500 µm. Warning: compositions that are known to be sensitive to impact and friction shall be grinded cautiously with appropriate personal protective equipment or by use a specific grinding apparatus.

Keep the grinded dry composition in a desiccator for less than 3 h. If the sample is out of the oven for more than 3 hours, re-dry it in an oven at 100 °C (6.16) for 1 hour.

Weigh 3 grams of the dry grinded composition to 0,0001 gram (6.13).

If the pyrotechnic composition does not contain nitrates, except nitrocellulose, go to Step Three.

7.3 Step Two

Place the weighed 3 grams in a 250 ml Erlenmeyer (6.11), add 100 ml of distilled water (5.4) and heat the whole in a laboratory reflux apparatus (6.15) at 100 °C for 3 h. Take care that water does not evaporate quickly and totally during the reflux process.

Separate the solid residue by filtration on an appropriate tissue paper or filtration paper (6.20).

Rinse carefully with 30 ml of warm (> 80 °C) distilled water. Repeat the operation 3 times.

Check for the remaining presence of nitrates by means of infrared spectrometry: the characteristic peaks of the nitrates contained initially in the pyrotechnic composition (except those that characterize nitrocellulose) shall not appear in the infrared spectrum of the solid residue.

If such peaks are still observed, add 100 ml of distilled water (5.4) to the solid residue and heat the whole in a laboratory reflux apparatus (6.15) at 100 °C for 3 hours. Separate the solid residue by filtration on an appropriate tissue paper or filtration paper (6.20).