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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: Taux d'azote dans la nitrocellulose par titration de l'ion sulfate de fer (II)

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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 of 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 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 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:

$$4\ FeSO_4 + 2\ HNO_3 + 2\ H_2SO_4 \rightarrow N_2O_3 + 2\ Fe_2(SO_4)3 + 3\ H_2O$$

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.

5.1 Sulphuric acid (H_2SO_4) Chemical purity 94-97 %

5.2 Potassium Nitrate (Pure)

5.3 Iron(II) sulphate ($FeSO_4 \cdot 7H_2O$) Crystals (pure)

5.4 Distilled Water

5.5 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