INTERNATIONAL STANDARD

ISO 22863-2

First edition
2020-02

Fireworks — Test methods for determination of specific chemical substances —

Part 2: Hexachlorobenzene by gas chromatography

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

Partie 2: Hexachlorobenzène par chromatographie en phase gazeuse

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ISO 22863-2:2020
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Reference number
ISO 22863-2:2020(E)

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Published in Switzerland

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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.

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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Fireworks — Test methods for determination of specific chemical substances —

Part 2: Hexachlorobenzene by gas chromatography

1 Scope

This document specifies the test method for the determination of hexachlorobenzene (HCB) in pyrotechnic compositions by gas chromatography.

The limit of detection depends on the substance to be determined, the equipment used, the quality of chemicals used for the extraction of the sample, and the clean-up of the extract.

Under the conditions specified in this document, a limit of detection of 1 mg/kg (expressed as dry matter) can be achieved.

2 Normative references

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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 22863-1:2020, *Fireworks — Test methods for determination of specific chemical substances — Part 1: General*

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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

Solid-liquid extraction of pyrotechnic composition is carried out by an extraction solvent, e.g. *n*-hexane or preferably ethanol or heptane. After increasing the concentration of components of low volatility and after purification with concentrated sulfuric acid, the sample is analysed by gas chromatography, using a hydrogen flame ionization detector (FID).

5 Reagents

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

Laboratory operations should comply with appropriate safety requirements for flammable and explosive materials and samples as well as strong acids and toxic materials. Operators should wear

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appropriate protection equipment and follow appropriate safety rules. Special measures should be taken for contingencies or uncontrollable reactions.

5.1 Standard HCB: Formula: C_6Cl_6, CAS:118-74-1, purity: 99,5 %.

5.2 Concentrated sulphuric acid (98 %).

5.3 Extraction solvent: chromatographically pure (e.g. *n*-hexane or preferably ethanol or heptane).

5.4 Standard solution: After accurately weighing the correct amount of standard HCB (5.1) (accurate to 0,1 mg), prepare a 100 µg/ml solution with the extraction solvent (5.3). Store it between 0 °C and 4 °C in the dark.

5.5 Standard intermediate solution: A typical value is 10 µg/ml. Transfer 1,0 ml standard solution (5.4) into a 10 ml volumetric flask, dilute it with the extraction solvent (5.3) to the 10 ml graduation and mix. Then store between 0 °C and 4 °C in the dark.

5.6 Standard working solutions: Prepare at least four solutions with different concentrations of HCB, 0,5 µg/ml, 1,0 µg/ml, 2,0 µg/ml and 5,0 µg/ml, by dilution of the standard intermediate solution (5.5).

6 Apparatus

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6.1 Gas chromatograph: equipped with hydrogen flame ionization detector (FID) and hydrogen generator, with the following conditions:

a) Chromatographic column: HP - 5 quartz capillary column [30 m × 0,25 mm (inner diameter) × 0,25 µm], or equivalent.

b) Temperature program: keep the initial temperature of 80 °C for 1 min, speed up to 230 °C at 20 °C/min, then keep it on for 10 min.

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c) Temperature of the injection port: 250 °C.

d) Temperature of the equipment: 300 °C.

e) Carrier gas: Nitrogen, greater than or equal to 99,99 % purity, flow rate of 1,0 ml/min.

f) Hydrogen: 40 ml/min.

g) Air: 400 ml/min.

h) Make-up gas: 30 ml/min.

i) Sample introduction: splitless injection.

j) Sample quantity: 1 µl.

6.2 Analytical balance: accuracy 0,1 mg.

6.3 Centrifuge: greater than or equal to 3 000 rpm.

6.4 Fast vortex mixer.

6.5 Multifunctional micro sample processing instrument or other equivalent instruments.

6.6 Graduated centrifuge tube: 5 ml, 10 ml.

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