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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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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).

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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 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

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*

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

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

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.

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.

6.7 Glass test tube: 10 ml.

6.8 Pipette.

7 Preparations

Preparation of sample shall be performed according to ISO 22863-1:2020, 5.2 and 5.3.

8 Procedure

8.1 Extraction

Weigh 1 g sample (accurate to 0,1 mg) into a 10 ml centrifuge tube (6.6), add 2 ml of the extraction solvent (5.3), mix 2 min on the fast vortex mixer, centrifuge 2 min at 3 000 rpm (6.3), and place the supernatant into a 10 ml graduated centrifuge tube (6.6). Repeat the procedure two times to extract the residue. Combine the supernatants, dry the combination at 40 °C on the sample processing instrument (6.5) to below 1 ml, add the extraction solvent up to 1 ml and then mix.

NOTE Ultrasonic extraction with a mix of the extraction solvent and acetone can also be used to prepare the sample.

8.2 Purification

Purify the sample by adding concentrated sulphuric acid (5.2) until impurities precipitates and the upper layer becomes colourless, centrifuge 2 min at 3 000 rpm, and then put the extraction solvent layer into a glass vial for gas chromatographic analysis.

8.3 Determination

Place 1 µl of the first standard working solution (5.6) in the gas chromatograph (6.1). Carry out the test according to the manufacturer's instructions and record the gas chromatogram of the sample of the first standard working solution. Repeat the test with the three other standard working solutions (5.6) and record the corresponding chromatograms. Calculate the peak areas of HCB in the four samples of the standard working solutions (see Annex A).

Place 1 µl of the purified sample solution (8.2) in the gas chromatograph (6.1). Carry out the test according to the manufacturer's instructions and record the gas chromatogram of the sample solution. Calculate the peak area A of HCB in the sample solution (see Annex A).

Determine which standard working solution exhibits a peak area A_S that is the closest to the peak area of the sample solution. Mix 1,0 ml of that standard working solution with 1,0 ml of the sample solution. Place 1 µl of the mixture in the gas chromatograph (6.1). Carry out the test according to the manufacturer's instructions and record the gas chromatogram of the sample solution. Calculate the peak area A_M of HCB in the mixture (see Annex A).

Under the conditions described in 6.1 for the gas chromatography, HCB reference retention time is about 8,2 min.

Figure A.1 shows a chromatogram of standard sample.

9 Calculation of results

Calculate the following quantity:

$$Q = 2 \,.\, A_M - A_S - A$$

The above quantity shall be close to zero.

If it is so, calculate the HCB content in the sample using the chromatographic data processor (PDP) or Formula (1):

$$X = \frac{A \times C_S \times V}{A_S \times m} \quad (1)$$

where

X is the HCB content in samples in mg/kg;

A is the peak area of HCB in sample solution;

C_S is the concentration of HCB in standard working solution in µg/ml;

A_S is the peak area of HCB in standard working solution;

V is the final volume of sample solution in ml;

m is the sample mass in g.

Calculation results shall be expressed with two significant digits.

If Q is not close to zero, no conclusion can be drawn from the measurements.

10 Minimum detection limit and recovery rate

10.1 Limit of detection

The limit of detection of this method is 1,0 mg/kg.

10.2 Recovery rate

At the end of a series of HCB determination tests, a second determination of the HCB content of the 5,0 µg/ml standard working solution shall be carried out. The result of such test shall be between 84,2 % and 101,6 % of the expected value of 5,0 µg/ml. Otherwise, the series of HCB determination tests shall be carried out again.

11 Test report

The test report shall include at least the following information:

a) name and address of the testing laboratory;

b) date of issue;

c) reference of this document, i.e. ISO 22863-2;

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

e) analysis results;

f) any anomaly that occurred while performing the tests.