TECHNICAL REPORT

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Textiles — Determination of certain flame retardants —

Part 3: Chlorinated paraffin flame retardants

Textiles — Détermination de certains retardateurs de flamme —
Partie 3: Retardateurs de flamme à base de paraffines chlorées

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Foreword

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This document was prepared by Technical Committee ISO/TC 38, *Textiles*.

A list of all parts in the ISO 17881 series can be found on the ISO website.

Textiles — Determination of certain flame retardants —

Part 3:
Chlorinated paraffin flame retardants

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1 Scope

This document describes a method for determining short-chain chlorinated paraffins (C_{10}-C_{13}) (SCCPs) in textiles using carbon skeleton reaction gas chromatography with a flame ionization detector (GC-FID).

It is applicable to all kinds of textile products.

2 Normative references

There are no normative references in this document.

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:

— IEC Electropedia: available at https://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Principle

SCCPs are extracted from textile specimens by an ultrasonic generator with *n*-hexane/acetone. SCCPs are pretested by gas chromatography with an electron capture detector (GC-ECD) and then quantified by GC-FID using an external standard method. SCCPs are dechlorinated by reacting with hydrogen to generate straight-chain alkane (C_{10}-C_{13}) under the action of a $PdCl_2$ catalyst. GC-FID is used to determine the quantity of straight-chain alkane following an external standard method, and then the quantity of SCCPs is calculated.

5 Reagents

Unless otherwise specified, only reagents of recognized analytical grade are used.

5.1 Short-chain chlorinated paraffins (SCCPs) (C_{10}-C_{13}, 51,5 % Cl), 100 µg/ml in cyclohexane.

5.2 Short chain chlorinated paraffins (SCCPs) (C_{10}-C_{13}, 55,5 % Cl), 100 µg/ml in cyclohexane.

5.3 Short-chain chlorinated paraffins (SCCPs) (C_{10}-C_{13}, 63 % Cl), 100 µg/ml in cyclohexane.

5.4 Straight-chain alkane (C_{10}-C_{13}), purity > 99,5 %.

5.5 *n*-hexane/acetone, 1:1 (volume fraction).

5.6 Diethyl ether.

5.7 Acetic acid solution, 5 % (volume fraction).

5.8 Ammonia, 25 % (mass fraction).

5.9 Mixture of *n*-hexane/diethyl ether, 90/10 (volume fraction).

5.10 Cyclopentane.

5.11 Cyclohexane.

5.12 Calcium carbonate ($CaCO_3$).

5.13 Palladium chloride ($PdCl_2$) catalyst.

After dissolving 0,08 g of $PdCl_2$ in 10 ml of 5 % acetic acid (5.7), the solution is transferred to the watch glass filled with 19 g of glass beads (6.11). The watch glass is placed into a boiling water bath, and the solution is constantly stirred to evaporate until dried. Distilled water is added on the watch glass, ammonia (5.8) is used to adjust the pH to 9, and the solution is evaporated until dried again. The glass beads are transferred to a glass filter crucible to be washed with 50 ml of cyclopentane (5.10) and then dried in the air.

6 Apparatus

6.1 Gas chromatograph with an electron capture detector (GC-ECD), used for pretesting SCCPs.

6.2 Gas chromatograph with a flame ionization detector (GC-FID), used for quantifying SCCPs.

The $PdCl_2$ catalyst (5.13) and reaction injection liners (6.9) are prepared and fitted into the GC-FID.

6.3 Ultrasonic generator, with a frequency of 35 kHz to 45 kHz.

6.4 Water bath and rotary evaporator.

6.5 Glass vials, 40 ml with tight closure.

6.6 K-D concentrator tube, 30 ml.

6.7 Round-bottom flask, 100 ml.

6.8 Florisil®[1] solid phase extraction (SPE) column, 1 g, 6 ml.

6.9 Reaction injection liners, successively filled with 0,5 cm rock wool, 0,2 cm $CaCO_3$, 2,0 cm $PdCl_2$ catalyst and 0,5 cm rock wool.

1) Florisil® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.