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Textiles - Determination of chlorinated organic solvents

纺织品 含氯有机溶剂的测定

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1 Scope

Warning. Persons using this document should have practical experience of proper laboratory work. This document does not point out every possible safety problem. It is the responsibility of the user to take suitable safety and health measures and to make sure that the conditions meet the relevant national regulations.

This document describes a method for determining 18 chlorinated organic solvents in textiles by gas chromatography with mass spectrometry (GC-MS).

This document applies to textile products of every kind.

3 Terms and definitions

The following term and definition apply to this document.

3.1 chlorinated organic solvents. Alkanes or alkenes in which carbon is combined with one or more chlorine atoms. Note: the chlorinated organic solvents referred to in this document are those commonly used as industrial solvents.

4 Principle

The specimen is extracted by ultrasound into n-tetradecane, then determined by GC-MS and quantified by the internal standard method.

5 Reagents

Unless otherwise stated, all reagents are of analytical grade.

5.1 n-Tetradecane, CAS number 629-59-4, chromatographic grade.

5.2 Standard samples or reference materials of the 18 chlorinated organic solvents, meeting Annex A, of purity not less than 99 %.

5.3 Fluorobenzene, internal standard 1, CAS number 462-06-6, purity not less than 99 %.

5.4 1-Chloro-2-bromopropane, internal standard 2, CAS number 3017-95-6, purity not less than 99 %.

5.5 4-Bromofluorobenzene, internal standard 3, CAS number 460-00-4, purity not less than 99 %.

5.6 Standard stock solution, 10 000 mg/L. Weigh 100 mg of each standard sample or reference material (5.2) accurately into a 10 mL volumetric flask, dissolve in n-tetradecane (5.1) and make up to the mark. Note: the standard stock solution is kept at 0 °C to 4 °C and has a shelf life of six months.

5.7 Mixed intermediate standard solution, 500 mg/L. Pipette 0.5 mL of each standard stock solution (5.6) into a 10 mL volumetric flask and make up with n-tetradecane (5.1). Note: the mixed intermediate standard solution is kept at 0 °C to 4 °C and has a shelf life of six months.

5.8 Internal standard stock solution, 1 000 mg/L. Weigh 10 mg of each internal standard substance (5.3 to 5.5) accurately into a 10 mL volumetric flask, dissolve in n-tetradecane (5.1) and make up to the mark. Note: the internal standard stock solution is kept at 0 °C to 4 °C and has a shelf life of six months.

5.9 Standard working solutions. Using n-tetradecane (5.1) as the reagent, pipette suitable amounts of the mixed intermediate standard solution (5.7) and of the internal standard stock solution (5.8) to prepare at least five standard working solutions of suitable concentration, for example 0.05 mg/L, 0.1 mg/L, 0.5 mg/L, 2.0 mg/L and 10.0 mg/L, in each of which the mass concentration of the three internal standards (5.3 to 5.5) is 5 mg/L. Prepare fresh before use.

5.10 Extraction solvent. Pipette 5 mL of the internal standard stock solution (5.8) into a 1 000 mL volumetric flask and make up to the mark with n-tetradecane (5.1) to give a solution of n-tetradecane containing 5 mg/L of the internal standards. Note: the extraction solvent is kept at 0 °C to 4 °C and has a shelf life of one month.

6 Apparatus

6.1 Gas chromatograph with mass spectrometer (GC-MS), fitted with an electron impact ionization source.

6.2 Ultrasonic generator with water bath, working frequency 40 plus or minus 5 kHz, able to hold 40 plus or minus 2 °C.

6.3 Extraction bottle, about 25 mL, of hard glass, with a stopper.

6.4 Analytical balance, with scale divisions of 0.1 mg and of 0.01 g.

6.5 Organic phase syringe filter, of polytetrafluoroethylene, pore size 0.45 micrometres.

7 Test procedure

7.1 Preparation of the specimen. Take enough representative sample, cut it into pieces of no more than 5 mm in the largest dimension, mix well, and weigh out 1.0 g of it accurately on the analytical balance (6.4), to the nearest 0.01 g, as the specimen.

7.2 Pretreatment. Place the specimen prepared under 7.1 in a 50 mL extraction bottle (6.3), add 10 mL of extraction solvent (5.10) accurately and seal. Extract by ultrasound in the water bath ultrasonic generator (6.2) at 40 plus or minus 2 °C for 20 plus or minus 5 min, cool to room temperature, and filter part of the supernatant through the organic phase syringe filter (6.5) into a sample vial for GC-MS determination. For a bulky or strongly absorbent specimen the volume of extraction solvent may be increased until the specimen is completely wetted and submerged, and the volume added recorded.

7.3 GC-MS conditions. Annex B gives examples of GC-MS test parameters.

7.4 Qualitative and quantitative analysis. Under the same test conditions, inject equal volumes of the sample solution (7.2) and of the standard working solution (5.9) alternately and analyse them under the conditions of Annex B. Where the analyte in the specimen has the same retention time as the standard sample or reference material, within a tolerance of plus or minus 0.1 min, and the relative abundances of the qualifier ions of the specimen agree with those of the standard sample or reference material, the maximum permitted relative deviation of each ion abundance being within the range given in Table 1, the analyte can be judged to be present in the specimen. The GC-MS total ion current chromatogram of the standard samples or reference materials of the chlorinated organic solvents is given in Annex C.

Table 1 sets the maximum permitted relative deviation for confirmation against the relative ion abundance: for a relative ion abundance above 50 %, plus or minus 10 %; above 20 % and up to 50 %, plus or minus 15 %; above 10 % and up to 20 %, plus or minus 20 %; and 10 % or less, plus or minus 50 %.

A standard working curve is drawn with the ratio of the peak area of the target compound in the quantifier ion channel to the peak area of the corresponding internal standard on the vertical axis and the ratio of the concentration of the target compound to that of the corresponding internal standard on the horizontal axis, and the content of the target compound in the specimen is calculated from the chromatographic peak areas of the target compound and the internal standard in the quantifier ion channel of the sample solution. The response of the target compound in the sample solution shall lie within the linear range of the instrument; where it goes beyond the range of the standard working curve, the sample solution is diluted with extraction solvent (5.10) to a suitable concentration and analysed again.

7.5 Blank test. The steps of 7.2 to 7.4 are carried out with no specimen present.

8 Treatment of test data

The content of each chlorinated organic solvent in the specimen is calculated from Formula (1) and the result reported separately for each solvent. The result is rounded as required by GB/T 8170 and expressed to one decimal place.

In Formula (1) the content of each chlorinated organic solvent in the specimen, in milligrams per kilogram, is obtained from the mass concentration of that solvent read from the standard working curve, in milligrams per litre, less the mass concentration read from the standard working curve for the blank solution, multiplied by the volume of extraction solvent in millilitres and by the dilution factor, and divided by the mass of the specimen in grams.

9 Limit of quantification and precision

9.1 Limit of quantification. The limit of quantification of each of the 18 chlorinated organic solvents is 1.0 mg/kg.

9.2 Precision. In the same laboratory, by the same operator using the same equipment and the same test method, and within a short interval on the same test object, the absolute difference between two independent test results shall not be greater than 10 % of the arithmetic mean of the two values, on the basis that cases exceeding 10 % of the arithmetic mean of the two values do not amount to more than 5 %.

10 Test report

The test report shall give at least the following: a) the number of this document; b) the source and description of the sample; c) the analytical instrument used; d) the test result; e) details of any departure from this document; f) the date of the test.