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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Textiles - Determination of perfluorinated and polyfluorinated compounds - Part 2: Gas chromatography-mass spectrometry method

纺织品 全氟及多氟化合物的测定 第2部分：气相色谱-质谱法

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1 Scope

Warning: those who use this document should have practical experience of regular laboratory work. This document does not point out all the possible safety problems. The user has the responsibility to take suitable safety and health measures and to ensure compliance with the conditions laid down in the relevant national regulations.

This document describes a method for determining ten perfluorinated and polyfluorinated compounds (see Annex A) in textiles by gas chromatography-mass spectrometry (GC-MS).

This document applies to textiles of every kind.

4 Principle

The perfluorinated and polyfluorinated compounds in the test specimen are extracted with tert-butyl methyl ether under ultrasound; the extract is concentrated and filtered through a microporous membrane filter, and is then determined by gas chromatography-mass spectrometry (GC-MS), qualitative analysis being made on the instrument and

quantification by the external standard method.

5 Reagents

Unless otherwise specified, all the reagents are of analytical grade.

5.1 tert-butyl methyl ether: chromatographic grade. 5.2 perfluorobutyl ethanol (4:2 FTOH): CAS number 2043-47-2, purity not less than 97.0 %. 5.3 perfluorohexyl ethanol (6:2 FTOH): CAS number 647-42-7, purity not less than 97.0 %. 5.4 perfluorooctyl ethanol (8:2 FTOH): CAS number 678-39-7, purity not less than 97.0 %. 5.5 perfluorodecyl ethanol (10:2 FTOH): CAS number 865-86-1, purity not less than 96.0 %. 5.6 perfluorohexylethyl acrylate (6:2 FTA): CAS number 17527-29-6, purity not less than 97.0 %. 5.7 perfluorooctylethyl acrylate (8:2 FTA): CAS number 27905-45-9, purity not less than 97.0 %. 5.8 perfluorodecylethyl acrylate (10:2 FTA): CAS number 17741-60-5, purity not less than 95.0 %. 5.9 methyl perfluorooctanoate (Me-PFOA): CAS number 376-27-2, purity not less than 95.0 %. 5.10 ethyl perfluorooctanoate (Et-PFOA): CAS number 3108-24-5, purity not less than 95.0 %. 5.11 2-(perfluorooctyl)ethyl methacrylate (8:2 FTMA): CAS number 1996-88-9, purity not less than 97.0 %.

5.12 Standard solutions, prepared as in a) to c). a) Standard stock solutions: the reference materials listed in 5.2 to 5.11 are each weighed accurately, dissolved in tert-butyl methyl ether (5.1) and made up to volume, to give single-component standard stock solutions of the target compounds at a mass concentration of 500 mg/L. b) Mixed intermediate standard solution: 1 mL is pipetted accurately from each of the standard stock solutions [5.12 a)] into the same volumetric flask and made up to 100 mL with tert-butyl methyl ether (5.1), to give a mixed intermediate standard solution at a mass concentration of 5 mg/L. c) Standard working solutions: prepared as needed. For example, a suitable volume of the mixed intermediate standard solution [5.12 b)] is taken and diluted with tert-butyl methyl ether (5.1) to give standard working solutions at mass concentrations of 50 μg/L, 100 μg/L, 200 μg/L, 400 μg/L and 800 μg/L. Note: kept sealed in the dark at 0 °C to 4 °C, the standard stock solutions have a life of six months, the mixed intermediate standard solution a life of three months and the standard working solutions a life of one month.

6 Apparatus

6.1 Gas chromatograph-mass spectrometer: fitted with an electron impact ionization source (EI).

6.2 Analytical balance: with scale divisions of 0.01 g and of 0.1 mg respectively.

6.3 Extractor: with a tight stopper, 30 mL to 50 mL, made of hard glass.

6.4 Ultrasonic generator: temperature (50 +/- 2) °C, working frequency (40 +/- 5) kHz.

6.5 Centrifuge: speed not lower than 4 000 r/min, with centrifuge tubes.

6.6 Graduated centrifuge tube (with screw cap): about 15 mL.

6.7 Nitrogen evaporator.

6.8 Syringe filter: organic nylon membrane, pore size 0.22 μm.

7 Test procedure

7.1 Preparation and extraction of the test specimen. A representative sample is taken, cut into pieces of about 5 mm by 5 mm and mixed evenly. (1.00 +/- 0.05) g of the mixed specimen, weighed to the nearest 0.01 g on the analytical balance (6.2), is placed in the extractor (6.3), 20.0 mL of tert-butyl methyl ether (5.1) is added accurately, and after sealing, the extractor (6.3) is placed in the ultrasonic generator (6.4) at (50 +/- 2) °C and extracted under ultrasound for (40 +/- 2) min. Once the extract has cooled to room temperature it is transferred to a centrifuge tube and centrifuged for 3 min in the centrifuge (6.5); 5.0 mL of the supernatant is taken into the graduated centrifuge tube (6.6), concentrated to 0.5 mL to 1.0 mL on the nitrogen evaporator (6.7) and made up to 1.0 mL with tert-butyl methyl ether (5.1); the solution is filtered through the syringe filter (6.8) into a sample vial for determination on the gas chromatograph-mass spectrometer (6.1).

7.2.1 Qualitative analysis. The analysis conditions are given in Annex B. Under the same test conditions, if the analyte in the sample has the same retention time as the reference material determined at the same time (a deviation of +/- 0.1 min being allowed), and if the relative abundances of the qualifier ions of each component in the chromatogram of the sample, compared with the relative abundances of the corresponding qualifier ions in the chromatogram of a standard solution of similar concentration, do not deviate by more than the range specified in Table 1, then the corresponding analyte may be judged to be present in the sample. The total ion chromatograms of the ten target compounds are given in Annex C. Table 1 gives the maximum permitted deviation of the relative abundance of the qualifier ions: for a relative ion abundance K greater than 50 %, +/- 10 %; for K greater than 20 % and not greater than 50 %, +/- 15 %; for K greater than 10 % and not greater than 20 %, +/- 20 %; and for K not greater than 10 %, +/- 50 %.

7.2.2 Quantitative analysis. A standard working curve is drawn with the peak area of the target compound as the ordinate and the concentration of the target compound as the abscissa, and quantification is carried out by the external standard method. The response values of the analyte in the standard working solutions and in the sample solution shall both lie within the linear response range of the instrument; if the content exceeds the range of the standard curve, the solution shall be diluted with tert-butyl methyl ether (5.1) to a suitable concentration before analysis.

8 Blank test

The determination is carried out following the steps of 7.1 to 7.2 but without adding the test

specimen.

9 Calculation and expression of the results

9.1 Calculation of the result. The content of the target compound is calculated by formula (1). The legend of formula (1) is as follows: Xi, the content of the component determined in the test specimen, in milligrams per kilogram (mg/kg); the mass concentration of the component determined in the sample solution obtained from the standard working curve, in micrograms per litre; the mass concentration of the component determined in the blank solution obtained from the standard working curve, in micrograms per litre; V, the volume of the extracting solution, in millilitres; f, the dilution factor; and m, the mass of the test specimen, in grams. The equation itself has come through the digitization corrupted and is not reproduced here.

9.2 Expression of the result. The test results are expressed separately for each of the perfluorinated and polyfluorinated compounds, the mean of two parallel determinations being taken and rounded to two decimal places in accordance with GB/T 8170.

10 Limit of quantification

The limit of quantification of this method is 0.20 mg/kg.

11 Precision

In the same laboratory, for two independent test results obtained by the same operator using the same equipment, by the same test method and within a short interval on the same test object, the absolute difference shall be not greater than 20 % of the arithmetic mean of the two determined values, on the condition that the case in which it is greater than 20 % of the arithmetic mean of the two determined values does not occur in more than 5 % of instances.

12 Test report

The test report shall include at least the following: a) the number of this document; b) the instrument used; c) the description of the test specimen; d) the content of the component determined in the test specimen, in milligrams per kilogram (mg/kg); e) the details of any departure from this document; f) the date of the test.

Annex A The ten perfluorinated and polyfluorinated compounds and their characteristic ions (informative)

The ten perfluorinated and polyfluorinated compounds and their characteristic ions are given in Table A.1, which lists for each compound the CAS number, the retention time in