ICS 59.080.01
CCS W 04

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 20384-2024

Replacing GB/T 20384-2006

Textiles - Determination of chlorobenzenes and chlorinated toluenes

纺织品 氯化苯和氯化甲苯类化合物的测定

Issued on: March 15, 2024 **Implemented on: October 1, 2024**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

Table of Contents

2 Warning, normative references, terms and definitions, and principle

Warning: those who use this document should have practical experience of regular laboratory work. The document does not point out all the possible safety problems. It is the responsibility of the user to take suitable safety and health measures and to make sure that the conditions laid down by the relevant national regulations are met.

2 Normative references: the document has no normative references.

3 Terms and definitions: the document has no terms and definitions that need defining.

4 Principle: the test specimen is extracted with dichloromethane under ultrasound, determined by gas chromatography mass spectrometry in the selected ion monitoring mode, and quantified by the external standard method.

5 Reagents or materials

Unless otherwise stated, all the reagents used are of analytical grade.

5.1 Dichloromethane, chromatographic grade.

5.2 Reference materials of the chlorobenzenes and chlorinated toluenes, meeting Annex A, of purity not less than 98 % by mass.

5.3 Standard stock solutions at 1000 mg/L: a given quantity of each of the reference materials listed in Annex A is weighed out, dissolved in dichloromethane and made up to volume, giving single-component standard stock solutions at a mass concentration of 1000 mg/L. Note: the standard stock solution is kept at 0 °C to 4 °C in the dark and is valid for 12 months.

5.4 Intermediate standard solution A at 10 mg/L: 0.1 mL of the standard stock solution of each substance (5.3) is pipetted into the same 10 mL volumetric flask and made up to volume with dichloromethane. Note: intermediate standard solution A is kept at 0 °C to 4 °C in the dark and is valid for 3 months.

5.5 Intermediate standard solution B at 1 mg/L: 1 mL of intermediate standard solution A (5.4) is pipetted into a 10 mL volumetric flask and made up to volume with dichloromethane. Note: intermediate standard solution B is kept at 0 °C to 4 °C in the dark and is valid for 3 months.

5.6 Standard working solutions: a suitable volume of intermediate standard solution B (5.5) is pipetted and diluted with dichloromethane to give a series of five standard working solutions of different concentration, for example at mass concentrations of 0.01 mg/L, 0.05 mg/L, 0.1 mg/L, 0.2 mg/L and 0.5 mg/L. Note: all the standard solutions are kept at 0 °C to 4 °C in the dark and are valid for 1 month.

5.7 Polytetrafluoroethylene (PTFE) filter membrane of 0.45 μm pore size.

6 Apparatus

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

6.2 Ultrasonic generator with a working frequency of (40 +/- 5) kHz.

6.3 Extraction vessel, made of hard glass, tubular and with a tight-fitting stopper, for example a 50 mL glass tube with a screw cap.

6.4 Volumetric flasks of 10 mL, 50 mL and other capacities.

6.5 Analytical balances with scale divisions of 0.001 g and 0.0001 g.

7 Test procedure

7.1 Preparation of the test specimen. A representative sample is taken from the laboratory

sample, cut into pieces smaller than 5 mm by 5 mm and mixed.

7.2 Extraction of the test specimen. A specimen of 1 g +/- 0.05 g, weighed to the nearest 0.001 g, is placed in the extraction vessel (6.3) and 10 mL of dichloromethane (5.1) is added accurately, making sure that the solution covers the specimen completely. After shaking, the extraction vessel is placed in the ultrasonic generator (6.2) and extraction is carried out at room temperature for (30 +/- 1) min. The vessel is taken out and the extract filtered through the PTFE membrane (5.7) into a sample vial for analysis by the GC-MS (6.1). Note: where the structure of the specimen is bulky, the mass weighed out is reduced accordingly so that the specimen stays fully covered by the extractant.

7.3.1 Qualitative analysis. Under the analysis conditions of Annex B, qualitative analysis is made by comparing the retention time and the characteristic ions, see Table B.1, of the target compound in the specimen solution with those in the standard working solution. Where the relative deviation between the retention time of the chromatographic peak of the target compound in the specimen solution and the retention time of the reference material is within +/-0.5 %, and the relative abundance ratio of the qualifying ions does not depart from that of a standard working solution of comparable concentration by more than Table 1 allows, the target compound may be judged present in the sample. Table 1 gives the maximum permitted relative deviation of the qualifying ion against the relative abundance ratio of the qualifying ion to the quantifying ion: +/-10 % where that ratio is above 50 %, +/-15 % where it is above 20 % and up to 50 %, +/-20 % where it is above 10 % and up to 20 %, and +/-50 % where it is 10 % or less.

7.3.2 Quantitative analysis. Under the best working conditions of the instrument, the standard working solutions (5.6) are determined and a standard working curve plotted with the peak area of the quantifying ion of the target compound as the ordinate and its concentration as the abscissa; the sample under test is quantified by the external standard method from that curve. The response of the target compound in the specimen solution shall fall within the range of the standard working curve; where the content exceeds that range, the specimen solution is diluted to a suitable concentration before analysis. Under those analysis conditions, the GC-MS total ion current chromatogram of the chlorobenzenes and chlorinated toluenes is shown in Annex C.

7.4 Blank test. The steps of 7.2 and 7.3 are followed at the same time as the specimen but with no specimen added.

8 Calculation and expression of the result

8.1 The content of each chlorobenzene or chlorinated toluene in the specimen is calculated by formula (1), in which the terms are the content of each compound i in the specimen in milligrams per kilogram; the mass concentration of that compound in the specimen solution in milligrams per litre; the mass concentration of that compound in the blank solution in milligrams per litre; the volume of the dichloromethane extract in millilitres; the mass of the

specimen in grams; and the dilution factor.

The result is expressed as the content of the individual chlorobenzene or chlorinated toluene compound or as a sum, and the calculated result is kept to one decimal place. Note: some of the isomers cannot be separated effectively under the chromatographic conditions of this document, see Annex B, and their results are expressed as a sum.

9 Limit of quantification, precision and test report

9.1 Limit of quantification. The limit of quantification for every target compound in this document is 0.1 mg/kg.

9.2 Precision. The absolute difference between two independent test results obtained in the same laboratory by the same operator using the same equipment, by the same test method and on the same test object within a short interval, shall be not greater than 15 % of the arithmetic mean of the two determined values, on the premise that the cases exceeding 15 % of the mean of the two determined values do not exceed 5 %.

10 Test report. The test report shall give at least the number of this document; the description of the sample; the test result; the details of any departure from this document; and the date of the test.

A Annex A (normative) Basic information on the chlorobenzenes and chlorinated toluenes

Table A.1 lists the basic information on the chlorobenzenes and chlorinated toluenes in four columns after the serial number: the compound name, the CAS number, the relative molecular mass and the molecular formula. The 29 entries are chlorobenzene; 1,2-dichlorobenzene; 1,3-dichlorobenzene; 1,4-dichlorobenzene; 1,2,3-trichlorobenzene; 1,2,4-trichlorobenzene; 1,3,5-trichlorobenzene; 1,2,3,4-tetrachlorobenzene; 1,2,3,5-tetrachlorobenzene; 1,2,4,5-tetrachlorobenzene; pentachlorobenzene; hexachlorobenzene; 2-chlorotoluene; 3-chlorotoluene; 4-chlorotoluene; alpha-chlorotoluene; 2,3-dichlorotoluene; 2,4-dichlorotoluene; 2,5-dichlorotoluene; 2,6-dichlorotoluene; 3,4-dichlorotoluene; 2,3,6-trichlorotoluene; 2,4,5-trichlorotoluene; alpha,alpha,alpha-trichlorotoluene; 2,3,4,5-tetrachlorotoluene; 2,3,4,6-tetrachlorotoluene; 2,3,5,6-tetrachlorotoluene; alpha,alpha,alpha,4-tetrachlorotoluene; and 2,3,4,5,6-pentachlorotoluene. Two of the entries, 2,3,4,5-tetrachlorotoluene and 2,3,5,6-tetrachlorotoluene, carry two CAS numbers each.

The relative molecular masses given in the table follow the degree of chlorination: 112.56 for chlorobenzene, 147.0 for the dichlorobenzenes, 181.45 for the trichlorobenzenes, 215.89 for the tetrachlorobenzenes, 250.34 for pentachlorobenzene and 284.78 for hexachlorobenzene; and 126.58 for the monochlorotoluenes, 161.03 for the dichlorotoluenes, 195.47 for the trichlorotoluenes, 229.92 for the tetrachlorotoluenes and