GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 97.140

CCS Y 80

GB/T 40908-2021

Method for the determination of prohibited substances in furniture products and related materials - Flame retardants

家具产品及其材料中禁限用物质测定方法

阻燃剂

Issued on: October 11, 2021 **Implemented on: May 1, 2022**

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

Table of Contents

Method for the determination of prohibited substances in furniture products and related materials - Flame retardants

Warning -- The personnel using this document shall have practical experience in formal laboratory work. This document does not indicate all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by relevant national laws and regulations.

1 Scope

This document describes the gas chromatography-mass spectrometry method for the determination of polybrominated biphenyls, polybrominated diphenyl ethers, and 6 organophosphate flame retardants in furniture products and their raw materials.

This document is applicable to the determination of polybrominated biphenyls, polybrominated diphenyl ethers, and 6 kinds of organophosphorus flame retardants in furniture products and their raw materials.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

The following terms and definitions apply to this document.

3.1 Polybromobiphenyls; PBBs

A class of compounds formed by one or more bromine atoms replacing hydrogen atoms on biphenyls.

Note 1: The specific structural formula is shown in Figure 1.

polybrominated biphenyls and polybrominated diphenyl ethers from the sample; purify the extract by the solid-phase silica gel and make the extract concentrate to a constant volume; then, use a gas chromatograph-mass spectrometer to carry out a quantitative analysis.

4.2 Determination of organophosphate flame retardants

Use an acetone solvent to ultrasonically extract organophosphate flame retardants from the sample; make the extract concentrate to a constant volume; then, use a gas chromatograph-mass spectrometer to carry out a quantitative analysis.

5 Reagents or materials

Unless otherwise specified, only reagents confirmed to be analytical pure are used in the analysis.

5.1 N-hexane.

5.2 Dichloromethane.

5.3 Toluene.

5.4 Acetone: chromatographically pure.

5.5 Methanol: chromatographically pure.

5.6 Organic phase filtration membrane: the pore size shall be 0.45 μm.

5.7 Glass sample bottle: it shall be about 50mL, with a sealable cap.

5.8 Silica gel solid-phase extraction cartridge: the specification shall be 500 mg/6 mL or equivalent; before using, rinse it with 5 mL of n-hexane and dichloromethane mixed solvent (5.9) to keep it wet.

5.9 Mixed solvent of n-hexane (5.1) and dichloromethane (5.2): measure 2 volumes of n-hexane (5.1) and 3 volumes of dichloromethane (5.2), and mix them well.

5.10 PBB reference materials: see Appendix A for the names and molecular formulas of typical polybrominated biphenyls.

5.11 PBB standard stock solution: Weigh an appropriate amount of polybrominated biphenyls (5.10), then dilute it to 500 mg/L with toluene (5.3), and mix well. Store it in an airtight place at 0 °C~4 °C, and protect it from light. The certified standard solution can also be used directly.

5.12 PBDE reference materials: see Appendix A for the names and molecular formulas

series of mixed standard working solutions shall have at least 5 calibration points. Among them, the mass concentrations of TCEP, TDCPP, and TOCP shall be 0.1 mg/L~10 mg/L, and the mass concentrations of TEPA, DDBPP, and TDBPP shall be 5 mg/L~200 mg/L. The mixed standard working solutions shall be stored at 0 °C~4 °C in the dark and airtight.

6 Instruments and equipment

6.1 Gas chromatograph-mass spectrometer (GC-MS): it shall be equipped with an EI source.

6.2 Ultrasonic generator: it shall be equipped with a temperature controller; when the basket does not be placed in it, the power per unit water bath area shall reach at least 0.28 W/cm^2.

6.3 Rotary evaporator: its temperature can be controlled to 75 °C, and the vacuum degree can be up to 3 kPa.

6.4 Pulverizer or similar equipment: pulverizers, scissors, stainless steel blades, etc.

6.5 Analytical balance: it shall be accurate to 0.1 mg; its measuring range shall not be less than 100 g.

6.6 Nitrogen concentrator.

7 Samples

7.1 Sampling

Cut leather (including natural leather, regenerated leather, artificial leather), fiber fabric, foamed plastic, plastic, and wood materials from the furniture products and their materials; if there are other attachments on the sample, remove them by physical methods; each material needs to be tested separately.

The weight of each sample shall not be less than 10 g, and those less than 10 g shall not be tested.

7.2 Samples

The cut leather, fiber fabric, and foamed plastic are respectively made into small samples less than 5 mm × 5 mm.

Use a pulverizer or similar equipment (6.4) to pulverize the cut plastic and wood materials, and use a standard sieve with a bore diameter of less than 3 mm to screen the sample.

8 Test steps

8.1 Polybrominated biphenyls and polybrominated diphenyl ethers

8.1.1 Extraction

Use an analytical balance (6.5) to weigh 0.5 g of the sample, and the weight shall be accurate to 0.1 mg; put it into a glass sample bottle (5.7), add an appropriate amount of a mixed solvent of n-hexane and dichloromethane (5.9), and make the solvent completely submerge the sample; tighten the bottle cap, place it in an ultrasonic generator (6.2), and then perform ultrasonic extraction at room temperature for 30 min.

Rinse a silica gel solid-phase extraction cartridge (5.8) with 5 mL of a mixed solvent of n-hexane and dichloromethane (5.9). Transfer the sonicated solution to the silica gel solid-phase extraction cartridge (5.8) for purification; wash the sample twice with 20 mL of a mixed solvent of n-hexane and dichloromethane (5.9), and the cleaning solution shall be all passed through the silica gel solid-phase extraction cartridge (5.8) for purification. Combine the purified extract in a 100 mL round-bottom flask, and concentrate it to be nearly dry with a rotary evaporator (6.3); then, dilute it with a mixed solvent of n-hexane and dichloromethane (5.9) to a constant volume of 2.0 mL, and use a 0.45 μm membrane (5.6) to filter it. After filtration, analyze it with a gas chromatography-mass spectrometer (6.1).

8.1.2 Gas chromatography and mass spectrometry conditions

Ensure the set parameters of the used instrument to be able to separate the tested component and other components effectively during the chromatographic determination. Since the test results depend on the used instrument, it is impossible to give the general parameters of the instrumental analysis. The following parameters have been proven to be suitable for the testing:

a) Chromatographic column: DB-5HT, 15 m×250 μm×0.10 μm, or other equivalent chromatographic columns;

b) Carrier gas: helium, purity ≥99.999%;

c) Carrier gas flow rate: 1.8 mL/min;

d) Sample injection method: pulse splitless injection, open the valve after 1.8 min;

e) Temperature of injection port: 280 °C;

f) Injection volume: 1.0 μL;

g) Solvent delay time: 4 min;

a low vacuum; then, placed it in a nitrogen concentrator (6.6), and dry it slowly with nitrogen at room temperature. Dissolve the residue with 10 mL of acetone (5.4), use a 0.45 μm filter membrane (5.6) to filter it, and analyze it with a gas chromatography-mass spectrometer (6.1). If necessary, dilute it appropriately before instrumental analysis.

8.2.2 Gas chromatography and mass spectrometry conditions

Ensure the set parameters of the used instrument to be able to separate the tested component and other components effectively during the chromatographic determination. Since the test results depend on the used instrument, it is impossible to give the general parameters of the instrumental analysis. The following parameters have been proven to be suitable for the testing:

a) Chromatographic column: DB-5MS quartz capillary column, 30 m×250 μm×0.25 μm, or other equivalent chromatographic columns;

b) Carrier gas: helium, purity ≥99.999%;

c) Carrier gas flow rate: 1.0 mL/min;

d) Sample injection method: splitless injection, open the valve after 1.0 min;

e) Temperature of injection port: 250 °C;

f) Injection volume: 1.0 μL;

g) Solvent delay time: 4 min;

h) Programmed temperature step: initial column temperature shall be 100 °C and held for 1 min; raise the temperature to 250 °C at a rate of 30 °C/min; then, raise the temperature to 300 °C at a rate of 10 °C/min and hold it for 3 min;

i) Chromatography-mass spectrometry interface temperature: 280 °C;

j) Ionization method: EI;

k) Ionization energy: 70 eV;

l) Mass scan range: 50 amu~800 amu;

m) Determination method: Selected ion monitoring method.

8.2.3 Standard working curve

The standard working solution is injected and determined according to the analysis conditions of 8.2.2; use the concentration of the six organophosphate flame retardants

ω_i --- The content of each polybrominated biphenyl and polybrominated diphenyl ether in the sample, in milligrams per kilogram (mg/kg);

A_i --- The chromatographic peak area of each polybrominated biphenyl and polybrominated diphenyl ether in the sample solution;

A_0 --- The chromatographic peak area of the blank sample;

A_s --- The chromatographic peak area of each polybrominated biphenyl and polybrominated diphenyl ether in the standard working solution;

ρ_s --- The mass concentration of each polybrominated biphenyl and polybrominated diphenyl ether in the standard working solution, in milligrams per liter (mg/L);

V --- The final volume of the sample solution, in milliliters (mL);

m --- The sample's weighed mass, in grams (g).

The calculation result is rounded to one decimal place.

9.1.2 Detection limit

The detection limit for each PBB and PBDE in this document is 5 mg/kg.

9.1.3 Recovery rate

The recovery rates above the detection limit for each PBB and PBDE in this document are 80%~120%.

9.2 Processing of test data of organophosphate flame retardants

9.2.1 Calculation of the result

Calculate the content of each organophosphate flame retardant in the sample according to formula (2):

$$w = \frac{(\rho_i - \rho_0) \times V}{m} \qquad \cdots\cdots(2)$$

Where:

w --- The content of each organophosphate flame retardant in the sample, in milligrams per kilogram (mg/kg);

ρ_i --- The mass concentration of each organophosphate flame retardant in the sample solution read from the standard working curve, in milligrams per liter (mg/L);

ρ_0 --- The mass concentration of each organophosphate flame retardant in the blank solution read from the standard working curve, in milligrams per liter (mg/L);

V --- The final volume of the sample solution, in milliliters (mL);

m --- The sample's weighed mass, in grams (g).

The calculation result is rounded to one decimal place.

9.2.2 Detection limit

The detection limits of the six organophosphate flame retardants are 35.0 mg/kg (TEPA), 1.0 mg/kg (TCEP), 1.0 mg/kg (TDCPP), 35.0 mg/kg (DDBPP), 1.0 mg/kg (TOCP), 35.0 mg/kg (TDBPP).

9.2.3 Recovery rate

The recovery rate above the detection limit of each organophosphate flame retardant in this document is 80%~120%.

10 Precision

10.1 Repeatability

In the same laboratory, the same operator uses the same equipment, according to the same test method, and tests the same test object within a short period. The absolute difference between the two independent test results obtained from the two tests carried out independently shall not be greater than 10% of the arithmetic mean of the two measured values; the situation that the absolute difference is more than 10% of the arithmetic mean of the two measured values shall not exceed 5%; it is the premise.

10.2 Reproducibility

In different laboratories, different operators use different equipment, according to the same test method, and test the same test object. The absolute difference between the two independent test results obtained from the two tests carried out independently shall not be greater than 20% of the arithmetic mean of the two measured values; the situation that the absolute difference is more than 20% of the arithmetic mean of the two measured values shall not exceed 5%; it is the premise.

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------