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Method for the determination of prohibited substances in furniture products and related materials - Polycyclic aromatic hydrocarbons

家具产品及其材料中禁限用物质测定方法

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Table of Contents

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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 a gas chromatography-mass spectrometry method for the determination of 18 polycyclic aromatic hydrocarbons in furniture products and their materials.

This document applies to the determination of 18 polycyclic aromatic hydrocarbons in furniture products and their 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 Polycyclic aromatic hydrocarbons; PAHs

A series of hydrocarbon compounds and their derivatives in which two or more benzene rings are fused together, and the rings may also have short alkyl or cycloalkyl substituents.

Note: 18 kinds of polycyclic aromatic hydrocarbons are shown in Table A.1 of Appendix A.

4 Principles

By ultrasonic wave, extract polycyclic aromatic hydrocarbons from the sample with

toluene; purify the extract by silica gel solid-phase extraction cartridge, and concentrate it to a constant volume; then, determine it by gas chromatography-mass spectrometry (GC-MS) method, and use selected ion monitoring mode and external standard method for quantification.

5 Reagents or materials

5.1 N-hexane: chromatographically pure.

5.2 Petroleum ether: chromatographically pure.

5.3 Toluene: chromatographically pure.

5.4 Nitrogen: purity ≥99.99%.

5.5 Helium: purity ≥99.999%.

5.6 18 kinds of polycyclic aromatic hydrocarbon reference materials: purity ≥98%; or 18 kinds of certified mixed standard solutions with known concentrations.

5.7 18 kinds of polycyclic aromatic hydrocarbon mixed standard working solutions: there shall be at least 5 calibration points, which can cover samples with concentrations ranging from 0.2 mg/kg~10 mg/kg. That is, the range of the standard solution is 10 ng/mL~500 ng/mL. The mixed standard working solution of polycyclic aromatic hydrocarbons was stored at 0 °C~4 °C in the dark and airtight.

5.8 Silica gel solid-phase extraction cartridge: 2 g/10mL or equivalent; before using, rinse it with n-hexane to keep it wet.

5.9 Centrifuge tubes.

5.10 Sample bottle: it shall be about 50 mL, with a sealable cap.

5.11 Filter membrane: organic phase syringe filter membrane with a pore size of 0.45 μm.

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.

Accurately weigh 0.5 g of the sample, and the weight shall be accurate to 0.1 mg; put it into the sample bottle (5.10), add 10 mL of the toluene (5.3) solution, and seal the bottle; after the sample is fully soaked, place the bottle in the frameless ultrasonic generator (6.2) and hang it at the temperature of (60±5) °C, then, carry out the ultrasonic extraction for 60 min. After the extraction is complete, take out the sample bottle, cool it to room temperature, and shake it well; then, transfer the extract to the centrifuge tube (5.9), and place the tube in the centrifuge (6.5) for centrifugation (if the solution after ultrasonic extraction can be stratification through standing, then the centrifugation is unnecessary). The supernatant is to be purified.

8.3 Purification

It can be selective to purify the samples with interfering substances. Before purification, rinse the silica gel solid-phase extraction cartridge (5.8) with 5 mL of n-hexane (5.1) to keep it wet; take 2 mL of the supernatant after extraction (8.2) and transfer it to the silica gel solid-phase extraction cartridge (5.8); elute with 5 mL of n-hexane (5.1), control the flow rate to be 0.5 drops/s, and discard the above passing-through-column liquid. Then, elute with 10 mL of petroleum ether (5.2), collect the petroleum ether eluent, and add 1 mL of toluene (5.3) to the collected petroleum ether eluent; slowly blow the liquid to about 1 mL with a nitrogen concentrator (6.6), and finally, make up to 2 mL with toluene (5.3).

8.4 Filtration

Filter the liquid after constant volume processing with an organic microporous membrane (5.11) and analyze it by a gas chromatography-mass spectrometer (6.1).

8.5 Determination

8.5.1 Gas chromatography and mass spectrometry reference 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-EUPAH or equivalent, 20 m (column length) × 0.18 mm (inner diameter) × 0.14 μm (film thickness);

b) Temperature of injection port: 290 °C;

c) Chromatographic column heating program: initial column temperature shall be 50 °C and held for 1 min; heat up to 200 °C at a rate of 20 °C/min and hold for 0.5 min; then, raise the temperature to 310 °C at a rate of 8 °C/min and hold for 5 min;

d) Temperature of transmitter interface: 280 °C;

e) The temperature of the ion source shall be 230 °C, the ionization method shall be EI, and the ionization energy shall be 70 eV;

f) Temperature of quadrupole rod: 150 °C;

g) Determination method: Selected ion monitoring (SIM);

h) Sample injection method: splitless injection;

i) The carrier gas shall be the helium, and the purity shall be ≥99.999%; the carrier gas flow rate shall be 1.0 mL/min;

j) Injection volume: 1 μL;

k) Solvent delay time: 4.0 min.

8.5.2 Qualitative and quantitative analysis by the GC-MS method

According to the analysis conditions of 8.5.1, determine 18 kinds of PAHs mixed standard working solution (5.7) and the filtered solution (8.4). Integrate the peak area of the quantitative ion (see Appendix A); take the peak area as the ordinate and the corresponding standard solution concentration as the abscissa, to draw a standard working curve, and the correlation coefficient shall be ≥0.995; use the external standard method for quantification. If the retention time of the chromatographic peak in the solution to be tested is similar to that of the standard working solution (5.7), and the type and abundance ratio of the selected ions (see Appendix A) of the target compound in the solution to be tested are consistent with those of the standard working solution, it can be determined that the target compound exists in the solution to be tested. Typical GC-MS selected ion chromatograms of 18 PAHs are shown in Appendix B.

8.5.3 Blank test

Carry out the blank test according to the above steps, except that no sample is added.

9 Processing of test data

9.1 Calculation of PAH content

Calculate the content of each PAH in the sample according to formula (1):

$$w_i = \frac{(\rho - \rho_0) \times V \times F}{m \times 1\,000} \qquad \cdots\cdots(1)$$

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