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Textile trim materials in automotive interior applications - Determination of the emissions of volatile organic compounds - Chamber method

汽车内饰用纺织材料 挥发性有机物的测定 箱体法

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Textile trim materials in automotive interior applications - Determination of the emissions of volatile organic compounds - Chamber method

1 Scope

This document describes a test method for the collection and determination of the emissions of volatile organic compounds from textile trim materials in automotive interior applications using the chamber method.

This document applies to all types of textile trim materials in automotive interior applications, including woven fabrics, knitted fabrics, non-woven fabrics and coated fabrics, as well as composite fabrics.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

HJ/T 400-2007, Determination of volatile organic compounds and carbonyl compounds in cabin of vehicles

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

total volatile organic compounds; TVOC

Volatile organic compounds with retention time between n-hexane and n-hexadecane when using a Tenax tube for sampling, and a non-polar chromatographic column (polarity index less than 10) for analysis.

3.2

total hydrocarbon mass concentration

Glass or stainless-steel adsorption tube, which is filled with solid adsorption material with a particle size of 250 μm ~ 180 μm (60 mesh ~ 80 mesh).

Note: In most cases, Tenax-TA tubes are used, and adsorption tubes with other fillers can also be used.

3.10

DNPH tube

A sampling tube that is filled with 2,4-dinitrophenylhydrazine (DNPH) silica gel.

4 Principle

Put the sample into the test chamber; heat it under the specified temperature, humidity and air change rate; monitor the sampling environment in real time through the online hydrogen flame ionization detector (FID); use the sampling tube to collect the air in the chamber after a certain period of time; use gas chromatography/mass spectrometry (GC-MS) and high performance liquid chromatography (HPLC) to qualitatively and quantitatively determine the collected benzene series and aldehydes and ketones (see Appendix A).

5 Instruments and materials

5.1 Test chamber

5.1.1 General

5.1.1.1 The chamber capacity of the test chamber shall be 0.05 m^3 ~ 4.0 m^3, and a test chamber with a capacity of (1.00±0.05) m^3 is recommended. The interior of the chamber shall be composed of a gas mixing device, a temperature regulating device, a humidity regulating device, a bracket for placing test samples, air intake and exhaust pipes, etc.

5.1.1.2 Chamber material: The inner wall of the chamber and the bracket for placing test samples shall be made of electropolished stainless steel. If the necessary parts or facilities inside the chamber cannot be made of electropolished stainless steel due to technical reasons, they shall be made of materials that have a weak adsorption of organic matter and do not emit organic matter, and the surface area of the parts or facilities using these materials shall not exceed 5% of the total internal surface area of the chamber.

5.1.1.3 Tightness: In order to avoid unnecessary air exchange, the chamber shall have good airtightness.

5.1.1.4 Gas mixing: A gas mixing device shall be equipped, so that the airflow speed in the center of the chamber in the empty chamber state is greater than 0.1 m/s during the test process.

5.1.1.5 Chamber cleaning: By adopting appropriate cleaning methods, such as high-temperature thermal desorption, etc., ensure that the chamber is thoroughly cleaned after each test is completed. Before the start of the next test, all surfaces in the test chamber that are in contact with volatile organic compounds shall also be cleaned to ensure that the background value of the chamber will not affect the test results.

5.1.2 Temperature regulating device

The temperature regulating device shall ensure that the temperature difference at each point in the space of the test chamber does not exceed ±1 °C.

5.1.3 Humidity regulating device

The humidity regulating device shall ensure that the relative humidity of the gas entering the test chamber is 45% ± 5% at 23 °C and 5% ± 0.5% at 65 °C, and no steam or spray can be generated during the humidity regulating process.

5.1.4 Test chamber tightness

In order to avoid uncontrollable gas exchange, it shall be ensured that under the condition of positive pressure of 1 000 Pa, the gas leakage per minute is less than 0.5% of the volume of the test chamber. In order to prevent outside gas from entering the test chamber, especially during gas sampling, it shall be ensured that the pressure of the test chamber is slightly higher than atmospheric pressure, that is, it is operated under the condition of slight positive pressure.

Set the chamber temperature to 65 °C, and test the airtightness of the test chamber by applying a positive pressure of 1 000 Pa on its surface and recording the pressure change of the test chamber within 2 h. The pressure sensor used shall be able to detect the pressure below 100 Pa and the deviation shall not exceed ±5%. During the 2 h test period, the average leakage rate shall be calculated according to Formula (1):

$$n_L = \frac{1\ 000}{t} \times \left(\frac{P_1}{P_2} - 1\right) \qquad \cdots\cdots(1)$$

Where:

n_L – the average leakage rate of the volume of the test space, in permillage per minute (‰/min);

P_1 – the absolute pressure of the chamber at the beginning of the tightness test, in Pascals (Pa);

Before each sampling, use the gas flow calibrator (5.5) to calibrate the sampling flow of the sampling system under the sampling load conditions.

5.5 Gas flow calibrator

Used to calibrate the sampling speed of the sampling pump. The maximum allowable error of the flowmeter does not exceed ±1%. When calibrating the flow rate, the error of flow rates before and after shall be less than 5%.

6 Sample preparation and sample handling

6.1 Sample preparation

6.1.1 The to-be-tested samples shall be sealed and packaged in the pollution-free polyethylene film or aluminum foil within the specified time (negotiated and recorded by both the supplier and the buyer) after production is off-line, and put into the packing box. Since volatile organic compounds are greatly affected by the environment, it is advisable to make a complete record of the history of the samples before packaging (such as storage environment temperature and humidity conditions, storage time).

6.1.2 During the transportation of samples, it is advisable to avoid direct exposure to strong light or high temperature environment to avoid damage to the packaging. After receiving the samples in the laboratory, they shall be stored in an environment with a temperature not exceeding 23 °C in a well-packaged state.

6.2 Sample handling

6.2.1 Before the test starts, the sample needs to be pre-treated for 24 hours under the condition of 23 °C ± 2 °C. During the pre-treatment process, it is required by the relevant party whether to open the package. If there is no requirement, the default is that the sample package is intact. The samples shall be placed on the shelf or grid; the samples shall neither contact with each other nor other objects outside the grid. Requirements for the pollution concentration value of the environment before sample pretreatment: toluene mass concentration ≤20 μg/m^3, formaldehyde mass concentration ≤ 20 μg/m^3.

6.2.2 The test sample shall be representative; the sample size shall be according to the procedure specified in the product standard or the agreement between the two parties; an appropriate product loading factor shall be provided. The requirements for test material shall be negotiated between the supplier and the buyer. If there is no special requirement, the recommended product loading factor is 2 m^2/m^3, that is, in the standard test chamber, the exposed area of the sample is 2 m^2, and multiple samples can be combined and the samples cannot be stacked. Weigh the sample mass and record, accurate to 1 g.

7 Preparation before the test

7.1 Test chamber cleaning

7.1.1 Chamber cleaning.

First, mechanical cleaning methods (such as industrial vacuum cleaners) shall be used to remove residual particles and impurities from the previous test, and then high-temperature cleaning shall be used for aging of the chamber or alkaline detergent shall be used to clean the chamber. When using heated purge air to clean the test chamber at high temperature, it shall be ensured that the temperature of all inner surfaces in contact with the atmosphere in the test chamber exceeds 200 °C, and the air shall be changed and cleaned repeatedly for 10 times after reaching the heating temperature. If alkaline detergent is used, use deionized water to rinse the entire chamber twice, followed by a hot wash with clean air at the test temperature. Monitor the background concentration of the chamber according to the requirements of 7.2. If the background concentration does not meet the requirements, continue to clean the chamber until the background concentration of the chamber reaches the requirements of 7.2.

7.1.2 Cleaning of other accessories

7.1.2.1 For the components that come into contact with the gas in the test chamber but cannot be directly cleaned during the cleaning process in 7.1.1, technically equivalent cleaning measures shall be taken separately.

7.1.2.2 For other components (such as sealing rings) whose temperature resistance is lower than 180 °C in the chamber, generally use deionized water to clean, and dry at 120 °C for 2 h ~ 3 h to achieve the cleaning effect.

7.2 Chamber background concentration monitoring

Under the condition of 65 °C, collect the background gas samples, and respectively determine the content of the target pollutants in the background gas samples. The sum of the mass concentration of total volatile organic compounds shall be less than 50 $\mu g/m^3$, and the mass concentration of each volatile organic compound shall be less than 5 $\mu g/m^3$. The total hydrocarbon mass concentration monitored by online FID shall be less than 1 600 $\mu g/m^3$.

7.3 Handling of connected gas-guide tubes

Treat at (100±5) °C for about 12 hours.

7.4 Handling of Tenax tubes

Before use, the Tenax tube needs to undergo high-temperature aging in an aging furnace. The aging conditions are as follows: the temperature is 300 °C, the nitrogen flow rate is 100 mL/min, the aging time of the new Tenax tube is 18 h, and the aging time of the

the recommended sampling flow rate is 100 mL/min, and the sampling time is 30 min; for the DNPH tube, the sampling flow rate is 400 mL/min, and the sampling time is 30 min.

Note: For Tenax tubes, it is generally recommended that the maximum sampling volume be ≤5 L.

Connect the gas-guide tube connected with the sampling tube to the outlet of the test chamber or other sampling ports of the discharge test chamber; pay attention to and record the time and temperature of connecting the sampling tube; record the atmospheric pressure if necessary. At the end of sampling, disconnect the gas-guide tube from the sampling port of the test chamber; note and record the time of disconnection; re-determine the sampling flow; turn off the pump. Disconnect the connection between the sampling tube and the gas-guide tube; use a screw cap fitting with a PTFE collar to seal both ends; write the number on it; wrap it in aluminum foil; send it to the instrument for analysis as soon as possible; analyze the samples in the Tenax tube and DNPH tube according to Appendix B and Appendix C in HJ/T 400-2007 respectively. If immediate analysis is not available, store it in a refrigerator at (4±2) °C, for a storage period up to 7 days.

8.6 During the entire test process, the temperature and humidity of the test chamber and the total hydrocarbon mass concentration ρ_x monitored by the online FID shall be recorded.

8.7 After the test, the test chamber shall be cleaned immediately according to 7.1.

9 Analytical test

9.1 Determination of benzene series

Follow the requirements of Appendix B in HJ/T 400-2007, where the lower limit of detection is 5 μg/m^3.

9.2 Determination of aldehydes and ketones

Follow the requirements of Appendix C in HJ/T 400-2007.

9.3 Determination of total hydrocarbon mass concentration

Read directly from the online hydrogen flame ionization detector, in micrograms per cubic meter (μg/m^3).

9.4 Calculation of results

9.4.1 Calculation of the mass concentration of a single volatile organic compound

Based on the area with the minimum area rejection value of 0.01 μg toluene, analyze and integrate all compounds with retention times between n-hexane and n-hexadecane, to obtain the total peak area; then, calculate using the linear equation of toluene (TIC). The mass concentration of total volatile organic compounds in the sample is shown in Formula (4):

$$\rho_T = \frac{m_T - m_B}{V \times 0.882} \times 1\,000 \qquad \cdots\cdots(4)$$

Where:

ρ_T – the mass concentration of total volatile organic compounds in the sample, in micrograms per cubic meter ($\mu g/m^3$);

m_T – the mass of total volatile organic compounds collected by the sampling tube, in micrograms (μg);

m_B – the mass of total volatile organic compounds in the blank tube, in micrograms (μg);

V – sampling volume, in liters (L);

0.882 – conversion coefficient of sampling volume at 65 °C to volume at 25 °C.

10 Test report

The test report shall include the following contents:

a) a reference to this document;

b) sample history;

c) arrival time record;

d) sample name and serial number;

e) sample size;

f) state of the sample during pre-treatment (unpackaged or intact package);

g) aging conditions, if Tenax tubes are aged according to the conditions provided by the manufacturer;

h) test conditions (chamber capacity, sampling flow rate, sampling time);

i) test results;

j) FID online monitoring curve report;

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