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GB/T 42705-2023

Textiles - Determination of benzene residues

纺织品 苯残留量的测定

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1 Scope

GB/T 42705-2023 measures residual benzene in textiles. Benzene is a confirmed human carcinogen with no established safe exposure level, and it is not deliberately applied to fabric - it arrives as a solvent residue, in adhesives, coatings and printing inks, or as an impurity in other solvents used in finishing. Because it is volatile, it continues to leave the material after manufacture, which means the exposure is by inhalation from the garment or the furnishing rather than by skin contact, and it also means the measurement has to capture what is escaping rather than what can be extracted. This document describes a method for determining benzene residues in textile products using headspace gas chromatography-mass spectrometry, in which the sample is heated in a sealed vial and the vapour above it is analysed - the technique matched to a volatile analyte at trace level in a solid matrix. It applies to all types of textile products. Under ICS 59.080.01 and CCS W04, it is written for textile testing laboratories, for brands operating restricted substance lists, and for printers, coaters and laminators whose process solvents are the source.

This document describes a method for the determination of benzene residues in textile products by using a headspace-gas chromatograph-mass spectrometer (HS-GC-MS). This document applies to all types of textile products.

2 Normative references

This document does not have normative references.

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3 Terms and definitions

This document does not have terms and definitions that need to be defined.

4 Principles

The sample is placed in an airtight container, and the benzene in the sample volatilizes into the container after equilibrating at the specified temperature and time. The air in the upper layer of the container is extracted and injected into a gas chromatograph-mass spectrometer for measurement, and the standard curve of the internal standard method is used for quantification.

5 Reagents

5.1 Methanol (CAS No. 67-56-1): chromatographically pure.

5.2 Benzene (CAS No. 71-43-2) standard solution: 1000 mg/L, or a standard solution of other appropriate concentrations.

5.3 Standard solution of deuterated toluene (toluene-d8, CAS No. 2037-26-5): 1000 mg/L, or a standard solution of other suitable concentrations.

5.4 Benzene standard stock solution (10 mg/L, 20 mg/L, 50 mg/L, 100 mg/L, 200 mg/L): Pipette 100 µL, 200 µL, and 400 µL of 1000 mg/L benzene standard solution (5.2) into three 2 mL volumetric flasks respectively, dilute to the mark with methanol (5.1), and mix well to obtain standard stock solutions of 50 mg/L, 100 mg/L, and 200 mg/L; take the benzene standard stock solution of 200 mg/L prepared above, and pipette 100 µL and 200 µL into two 2 mL volumetric flasks respectively, dilute to the mark with methanol (5.1), and mix well to obtain 10 mg/L and 20 mg/L standard stock solutions.

5.5 Internal standard working solution (100 mg/L): Accurately pipette 200 µL of deuterated toluene standard solution (5.3) into a 2 mL volumetric flask, dilute to the mark with methanol (5.1), and mix well. NOTE 1: Store the above standard solution in a refrigerator at (-18±4) °C. The benzene standard solution (5.2) and deuterated toluene (5.3) standard solution are valid for 3 months after opening or use. Benzene standard storage solution (5.4) and internal standard working solution (5.5) are valid for 1 week. NOTE 2: When preparing the solution, other volumetric flasks of appropriate volume can also be used.

6 Instruments and equipment

6.1 Headspace-gas chromatograph-mass spectrometer (HS-GC-MS): It shall be equipped with an electron bombardment ion source (EI).

6.2 Headspace vial: The capacity is 20 mL; it has a PTFE-coated sealing cap.

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6.3 Pipettor or microinjector: 100 μL, 1000 μL, 5000 μL, etc.

6.4 Brown volumetric flask: 2 mL.

6.5 Analytical balance: The graduation values are 0.0001 g and

0.01 g.

7 Test steps

7.1 Sample preparation Take a representative sample, cut it into fragments smaller than 5 mmx5 mm, and mix them; weigh

0.5 g of sample from the fragments, accurate to

0.01 g, and place it in a 20 mL headspace vial (6.2).

7.2 Adding internal standard Pipette 50 μL of the internal standard working solution (5.5), put it into the headspace vial containing the sample, and immediately seal it tightly with a lid. Test it later.

7.3 Drawing of working curve Take six 20 mL headspace vials (6.2), and add 50 μL internal standard working solution (5.5) to each headspace vial. Add 50 μL of 10 mg/L, 20 mg/L, 50 mg/L, 100 mg/L, and 200 mg/L benzene standard stock solution (5.4) to 5 headspace vials that have been added with the internal standard working solution, and immediately seal them with lids. At this time, the working solutions with the total toluene-d8 mass of 5 μg and the total benzene mass of 0 μg, 0.5 μg, 1 μg, 2.5 μg, 5 μg, and 10 μg respectively are obtained. Carry out the measurement according to the provisions of 7.4; take the total amount of benzene in the series of working solutions as the abscissa (in micrograms), take the peak area ratio of benzene to toluene-d8 as the ordinate, and draw the working curve. The correlation coefficient R2 of the obtained working curve shall be greater than 0.99. NOTE: In order to reduce the volatilization of benzene as much as possible, when adding the standard solution, pour it into the bottom of the bottle gently to avoid it from contaminating the bottle wall. After adding the standard solution at each concentration point, immediately cover and seal the bottle; the working curve solution shall be prepared newly when it will be used; after the standard stock solution is taken out of the refrigerator, let it stand in a closed state and return to room temperature, and then carry out the preparation work.

7.4 Headspace-gas chromatography-mass spectrometry analysis The equilibration time of the automatic headspace sampler is 50 min, and the oven temperature is 120 °C. Place the sample (7.2) to which the internal standard has been added into the headspace sampler, and perform the determination referring to the instrument conditions listed in Appendix A. If the selected monitoring ions of the sample (7.2) to which the internal standard has been added and that of the working curve (7.3) appear at the same retention time (±0.5%), and the allowable deviation between the relative ion abundance of the qualitative ion and the

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relative ion abundance of the working curve does not exceed the range specified in Table 1, then it is qualitatively confirmed that the corresponding target analyte exists in the sample. Table 1 -- The allowable deviation range of relative ion abundance in the qualitative confirmation Relative ion abundance / % >50 >20~50 >10~20 <=

10 Allowable deviation range / % ±10 ±15 ±20 ±

50 The internal standard curve method is used for quantification, and the total amount of benzene in the sample is read from the working curve according to the peak area ratio of benzene to toluene-d8 in the sample.

Appendix A

(Informative) Headspace-GC-MS reference conditions A.1 Overview Since the test results depend on the instrument used, it is impossible to give general parameters for chromatographic analysis. The gas chromatography and mass spectrometry conditions given in this appendix have been proven to be suitable. A.2 Measurement conditions and parameters A.2.1 The conditions of the automatic headspace sampler are as follows:

a) Quantitative loop temperature: 125 °C;

b) Quantitative loop volume: 1 mL;

c) Transfer line temperature: 220 °C;

d) Pressurization time:

0.2 min;

e) Injection time: 1 min. A.2.2 Gas chromatography conditions are as follows:

a) Chromatographic column: a 6% cyanopropylphenyl-94% methylpolysiloxane quartz capillary column, with a column length of 30 m, a film thickness of 1.8 μm, an inner diameter of

0.32 mm; or other chromatographic columns with equivalent performance;

b) Headspace injector temperature: 250 °C;

c) Injection mode: split injection, with a split ratio of 10:1;

d) Temperature program: The initial temperature is 40 °C, and keep for 3 minutes; heat up to 230 °C at 10 °C/min, and keep for 5 minutes;

e) Carrier gas: helium (the purity is >=99.999%), and the flow rate is

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