NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 45367-2025

Textiles - Determination of camphor derivates

纺织品樟脑衍生物的测定

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1 Scope

GB/T 45367-2025 specifies how to measure camphor derivatives in textiles. The two compounds it targets, 3-benzylidene camphor and 4-methylbenzylidene camphor, are ultraviolet filters: they were developed for sunscreen and they are applied to fabric for the same reason, to absorb UV and to protect either the wearer or the dyes in the cloth from fading. Both are under scrutiny as endocrine disruptors and are restricted in cosmetics in several jurisdictions, and a substance restricted on skin contact in a cream raises the same question in a garment worn against the skin all day. This document describes a test method for determining 3-benzylidene camphor and 4-methylbenzylidene camphor in textiles by liquid chromatography-tandem mass spectrometry, a technique chosen for the selectivity needed to identify trace residues in a dyed and finished textile matrix without interference. It applies to all types of textile products. It is written for textile testing laboratories, for brands and retailers maintaining restricted substance lists, and for finishers and garment manufacturers who need a defined method behind a compliance declaration.

This document describes a test method for the determination of camphor derivatives (3-benzylidene camphor and 4-methylbenzylidene camphor) in textiles using liquid chromatography-tandem mass spectrometry (LC-MS/MS). This document applies to all types of textile products.

2 Normative references

The following referenced documents are indispensable for the application of this document.

For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682, Water for analytical laboratory use -- Specification and test methods

GB/T 8170, Rules of rounding off for numerical values and expression and judgement of limiting values

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 Principle

The specimen is extracted with methanol by ultrasonic method. The extract is filtered through a filter membrane and then determined by LC-MS/MS. The external standard method is used for quantification.

5 Reagents and materials

Unless otherwise stated, all reagents used are analytically pure.

5.1 Water. Meet the requirements of Grade one water in GB/T 6682.

5.2 Methanol. CAS No. 67-56-1; chromatographically pure.

5.3 Ammonium formate. CAS No. 540-69-2; purity is not less than 99.0% (mass fraction).

5.4 Ammonium formate solution.

0.01 mol/L. Weigh

0.64 g of ammonium formate (5.3). Dissolve in water (5.1) and dilute to 1000 mL. Shake well. 5.5 3-Benzalkonium camphor standard substance/standard sample. CAS No. 15087-24- 8; purity is not less than 98.1% (mass fraction). 5.6 4-Methylbenzalkonium camphor standard substance/standard sample. CAS No. 36861-47-9; purity is not less than 98.1% (mass fraction).

5.7 Standard stock solution. The mass concentration is not less than 200 mg/L. Accurately weigh 3-benzylidene camphor standard substance/standard sample (5.5) and 4-methylbenzylidene camphor standard substance/standard sample (5.6). Prepare with methanol (5.2). NOTE. This solution should be stored in a refrigerator at 0°C~4°C away from light. The validity period is 6 months.

5.8 Standard working solution. The mass concentration is

0.02 mg/L~

0.5 mg/L. Accurately pipette an appropriate amount of standard stock solution (5.7). Dilute

with methanol (5.2) to prepare. NOTE. This solution should be stored in a refrigerator at 0°C~4°C away from light. The validity period is 3 months.

6 Instruments and equipment

6.1 Liquid chromatography-tandem mass spectrometer (LC-MS/MS). equipped with electrospray ionization source (ESI).

6.2 Analytical balance. graduation values are

0.01 g and 0.0001 g respectively.

6.3 Extractor. sealed with stopper; about 50 mL; made of hard glass.

6.4 Ultrasonic generator. working frequency is (40±5) kHz; temperature can be controlled to (50±2)°C.

6.5 Organic phase needle filter. polytetrafluoroethylene; pore size is 0.22 μm.

7 Analysis steps

7.1 Specimen preparation Select representative specimens. Cut into pieces of approximately 5 mm x 5 mm. Mix well.

7.2 Extraction Use an analytical balance (6.2) to weigh about

1.0 g of specimen (accurate to

0.01

g) and place in an extractor (6.3). Add

50.0 mL of methanol (5.2) accurately. Seal the extractor. Extract for (30±2) min in an ultrasonic generator (6.4) at (50±2)°C. Cool to room temperature within 2 min. Filter the specimen solution through an organic phase needle filter (6.5). Use LC-MS/MS (6.1) to analyze.

7.3 LC-MS/MS determination

7.3.1 Qualitative analysis Under the same test conditions, take equal volumes of the specimen solution (7.2) and the standard working solution (5.8). Inject them for determination. Test and analyze according to the conditions of 7.3.Qualitative analysis is performed by comparing the retention time and relative abundance of characteristic ions of the target in the specimen solution and the standard working solution. The deviation between the retention time of the target in the specimen and the retention time of the target in the standard working solution is within the range of ±2.5%. The relative abundance of the characteristic ions of the target in the sample is consistent with the relative abundance of

the target in the standard working solution. If the relative abundance allowable deviation does not exceed the range specified in Table 1, it can be judged that the corresponding analyte exists in the sample. Annex A gives examples of LC-MS/MS analysis conditions.