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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Textiles-Determination of benzalkonium chloride

纺织品 苯扎氯铵的测定

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1 Scope

This document describes the test methods for the determination of benzalkonium chloride in textiles using high performance liquid chromatography-diode array detector (HPLC-DAD) or high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS).

This document applies to all types of textile products.

2 Normative references

GB/T 6682

3 Terms and definitions

For the purpose of this document, there are no terms and definitions that need to be defined.

4 Principle

Use acetonitrile to ultrasonically extract the benzalkonium chloride in the sample, after the extract is filtered through a filter membrane, it is determined by HPLC-DAD or HPLC-MS/MS and quantified by the external standard method.

5 Reagents and materials

Unless otherwise specified, all reagents are at least analytical grade.

5.1 Water. grade 2 water as specified in GB/T 6682.

5.2 Acetonitrile. CAS No. 75-05-8, chromatographic grade.

5.3 Methanol. CAS No. 67-56-1, chromatographic grade.

5.4 Formic acid. CAS No. 64-18-6.

5.5 Ammonium acetate. CAS No. 631-61-8.

5.6 Glacial acetic acid. CAS No. 64-19-7.

5.7 Three kinds of benzalkonium chloride standard substances. comply with the

provisions of Annex A, purity >= 97.0 %.

5.8 Mixed standard stock solution. Use an analytical balance (6.3) to weigh a certain amount of each benzalkonium chloride standard substance (5.7), dissolve and make up to the mark with acetonitrile (5.2), to prepare a mixed standard stock solution with a mass concentration of 1000 mg/L.

NOTE. The mixed standard stock solution is stored in a refrigerator at 0 °C ~ 8 °C and has a shelf life of 6 months.

5.9 Mixed standard working solution. Accurately pipette a certain volume of the mixed

standard stock solution (5.8) and dilute with acetonitrile (5.2) to obtain series mixed standard working solutions of at least 5 different concentrations. When using HPLC- DAD for analysis, prepare mixed standard working solutions of a series of concentrations, such as 5.0 mg/L, 10.0 mg/L, 20.0 mg/L, 50.0 mg/L, and 100.0 mg/L;

when using HPLC-MS/MS for analysis, prepare mixed standard working solutions of a series of concentrations, such as 0.05 mg/L, 0.10 mg/L, 0.20 mg/L, 0.50 mg/L, and 1.00 mg/L. This solution is prepared and used immediately.

5.10 Ammonium acetate solution. Weigh 7.70 g of ammonium acetate (5.5) and

place

in a beaker, add water (5.1) to dissolve and dilute to 1000 mL, adjust the pH to 5.0 with glacial acetic acid (5.6), and filter with an aqueous filter membrane (5.13).

5.11 0.1 % formic acid solution. Pipette 1 mL of formic acid (5.4) into a 1000 mL volumetric flask and make up to the mark with water (5.1).

5.12 Organic phase filter membrane. pore size 0.22 μm, made of

polytetrafluoroethylene (PTFE).

5.13 Aqueous phase filter membrane. pore size 0.22 μm, made of mixed cellulose ester (MCE).

6.1 High performance liquid chromatograph (HPLC). equipped with a diode array

detector (DAD).

6.2 High performance liquid chromatography-tandem mass spectrometer (HPLC-

MS/MS). equipped with an electrospray ionization source (ESI).

6.3 Analytical balance. the graduation values are 0.01 g and 0 0001 g.

6.4 Ultrasonic water bath generator. the working frequency is (40 ± 5) kHz, the water bath temperature can be controlled at (60 ± 3) °C.

6.5 pH meter. the graduation value is 0.01.

6.6 Glass container. 40 mL ~ 60 mL, made of hard glass, with a screw-on sealing cover.

6.7 Volumetric flask. 1000 mL.

7.1 Sample preparation

Take a representative sample, cut it into pieces less than 5 mm x 5 mm, and mix it thoroughly.

7.2 Sample extraction

Weigh (1 ± 0.1) g of sample, accurate to 0.01 g, place in a glass container (6.6), add 20 mL of acetonitrile (5.2), tighten the sealing cover, place in a (60 ± 3) °C ultrasonic water bath generator (6.4) for ultrasonic extraction for (30 ± 1) min, take out the glass container (6.6) and cool to room temperature, take part of the extract and filter through the organic phase filter membrane (5.12), and the filtrate is used as the test liquid for analysis on the instrument.

7.3 HPLC-DAD analysis

Under the same test conditions, take equal volumes of the test solution obtained in 7.2 and the mixed standard working solution (5.9) and alternately inject them for determination. The HPLC-DAD analysis conditions are shown in Annex B. Qualitative analysis is performed by comparing the retention time of the chromatographic peak at a specific detection wavelength and the ultraviolet absorption spectrum. If the relative deviation between the retention time of the target analyte in the test solution and the retention time corresponding to the standard substance is within the range of ±2.5 %, the ultraviolet absorption spectrum is consistent with that of the standard substance, and the maximum absorption wavelength deviation is within the range of ±2 nm, it can be determined that the corresponding target analyte exists in the sample.

Under the HPLC-DAD analysis conditions given in Annex B, the chromatogram and spectrum of benzalkonium chloride obtained are shown in Annex C.

According to the content of the target analyte in the test solution, select a mixed standard working solution (5.9) with a similar response value for analysis. With the response peak area of the target analyte as the vertical axis and the concentration of the target analyte as the horizontal axis, plot a standard working curve, and perform quantitative analysis according to the external standard method. The response values of the target analyte in the mixed standard working solution (5.9) and the test solution obtained in 7.2 shall be within the linear response range of the instrument. If the content exceeds the range of the standard curve, dilute with acetonitrile to an appropriate concentration before determination.

7.4 HPLC-MS/MS analysis

Under the same test conditions, take equal volumes of the test solution obtained in 7.2 and the mixed standard working solution (5.9) and alternately inject them for determination. The HPLC-MS/MS analysis conditions are shown in Annex D. Compare the retention times of the mass chromatographic peaks of the test solution and the mixed standard working solution (5.9). If the relative deviation between the retention time of the target analyte in the test solution and the retention time corresponding to the standard substance is within the range of ±2.5 %, and the deviation between the relative ion abundance of the qualitative ion pair and the relative ion abundance of the standard working solution with equivalent concentration do not exceed the range specified in Table 1, it can be determined that the corresponding target analyte exists in the sample.

Under the HPLC-MS/MS analysis conditions given in Annex D, the multiple reaction monitoring selected ion current chromatogram of each benzalkonium chloride is shown in Annex E.

According to the content of the target analyte in the test solution, select a mixed standard