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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 33273-2016

Textiles - Determination of triclosan residues

纺织品 三氯生残留量的测定

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4 Reagents and materials

Unless otherwise stated, the reagents used in the analysis are of analytical grade.

4.1 Water. The pretreatment water is the grade 3 water specified in GB/T 6682. The water for analytical instrument is the grade 1 water specified in GB/T 6682.

4.2 Methanol. Chromatographically pure.

4.3 N-hexane. Chromatographically pure.

4.4 Acetic anhydride. Purity>=98.5%.

4.5 Sodium tetraborate solution,

0.1 mol/L. WEIGH

38.1 g of sodium tetraborate; USE water to dissolve and dilute to 1000 mL.

4.6 Triclosan standard. CAS No. 3380-34-

5.Purity >99.5%.

4.7 Standard stock solution. Accurately weigh an appropriate amount of triclosan standard (4.6) to the nearest

0.1 mg; USE methanol (4.2) to prepare a triclosan standard stock solution with a concentration of 100 mg/L.

4.8 Standard working solution. Accurately pipette an appropriate volume of triclosan standard stock solution (4.7); USE methanol (4.2) to dilute to the standard solution with required concentration.

Note. The standard stock solution is stored in the refrigerator at 0 °C~4 °C; the expiry date is 6 months. The standard working solution is stored in the refrigerator at 0 °C~4 °C; the expiry date is 3 months.

4.9 Organic-phase filter membrane. 0.45 μm filter membrane.

5 Apparatus

5.1 Analytical instruments. According to the selected method, select the following instruments.

5.1.1 High-performance liquid chromatograph. Equipped with diode array detector or ultraviolet detector.

5.1.2 Liquid chromatograph-tandem mass spectrometer. Equipped with electrospray ionization source.

5.1.3 Gas chromatograph. Equipped with electron capture detector. times. The upper organic phase is filtered through a filter membrane (4.9); and then analyzed by gas chromatography (5.1.3), gas chromatography-mass spectrometry (5.1.4), or gas chromatography-tandem mass spectrometry (5.1.5). If necessary, perform instrument analysis after dilution.

7.1 High-performance liquid chromatography

7.1.1 Analysis conditions of high-performance liquid chromatography Since the test results depend on the instrument used, it is impossible to give general parameters for liquid chromatography analysis. The set parameters shall ensure that, during chromatographic determination, the measured component and other components can be effectively separated. The parameters given below are proved to be feasible.

a) Chromatographic column. C18 reversed-phase chromatographic column; 250 mm (column length)x

4.6 mm (inner diameter)x5 μm (particle size); or equivalent;

b) Mobile phase. Methanol + water (90+10, volume ratio);

d) Column temperature. 40 °C;

e) Detection wavelength. 282 nm;

f) Injection volume. 20.0 μL.

7.1.2 Standard working curve According to 7.1.1, the standard working solution (4.8) is injected for determination. USE the triclosan concentration as the abscissa and the peak area as the ordinate, to draw the standard working curve. Under the above liquid chromatography analysis conditions, the liquid chromatogram of the triclosan standard solution is shown in Figure A.1

a) in Appendix A.

7.1.3 Qualitative and quantitative analysis According to 7.1.1, the sample solution (6.2.1) is injected for determination. According to the retention time of chromatographic peak, perform qualitative analysis. When necessary, confirm in conjunction with ultraviolet absorption spectrogram. Based on the peak area of the chromatographic peak, use the external standard method for quantification.

f) Injection volume. 1.0 μL;

g) Injection method. Splitless injection.

7.3.2 Standard working curve After the standard working solution (4.8) is derivatization pretreated according to the method of 6.2.2, according to 7.3.1, it is injected for determination. USE the triclosan concentration as the abscissa and the peak area as the ordinate, to draw the standard working curve. Under the above conditions, the gas chromatogram of the standard solution of triclosan derivative is shown in Figure A.2

a) of Appendix A.

7.4 Gas chromatography-mass spectrometry

7.4.1 Analysis conditions of gas chromatography-mass spectrometry Since the test results depend on the instrument used, it is impossible to give general parameters for gas chromatography-mass spectrometry analysis. The set parameters shall ensure that, during chromatographic determination, the measured component and other components can be effectively separated. The parameters given below are proved to be feasible.

7.4.1.1 Analysis conditions of gas chromatography.

a) Chromatographic column. DB-5MS quartz capillary column; 30 m (column length)x

0.25 mm (inner diameter)x0.25 μm (film thickness); or equivalent;

b) Chromatographic column temperature. 150 °C 280 °C (

c) Inlet temperature. 280 °C;

d) Chromatograph-mass spectrometer interface temperature. 280 °C;

e) Carrier gas. Helium, purity>=99.999%. Flow rate is

f) Injection volume. 1.0 μL;

g) Injection method. Splitless injection. Open the valve after

1.0 min. to draw the standard working curve. Under the above conditions, the gas chromatography-tandem mass spectrometry chromatogram of the standard solution of triclosan derivative is shown in Figure A.2

c) of Appendix A.

7.5.3 Qualitative and quantitative analysis According to 7.5.1, the sample solution (6.2.2) is injected for determination. According to the retention time of chromatographic peak, perform qualitative analysis. Confirm in conjunction with the mass spectrogram. Based on the peak area of the chromatographic peak, use the external standard method for quantification.

8 Blank tests

Except that no sample is added, the determination steps of 6.1~

7.5 above are followed.