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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 37633-2019

Textiles - Determination of 1,2-dichloroethane, ethylene chlorohydrin and chloroacetic acid

纺织品 1,2-二氯乙烷、氯乙醇和氯乙酸的测定

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1 Scope

China's national method for determining 1,2-dichloroethane, ethylene chlorohydrin and chloroacetic acid in textiles. It specifies the principle, the reagents, the instruments and equipment, the determination procedure, and the calculation and expression of results. The three are related by a common origin: they are residues and by-products of ethylene oxide chemistry, which is used in textile processing to make the ethoxylated surfactants that dominate scouring, dyeing and finishing formulations. 1,2-dichloroethane is a carcinogen and a restricted solvent; ethylene chlorohydrin is acutely toxic and forms wherever ethylene oxide meets chloride; chloroacetic acid is corrosive and is used in making carboxymethyl cellulose sizing agents. None is deliberately present in a finished fabric, which is exactly why they need a method - a residue nobody intended is one nobody is looking for unless a standard says to look.

This Standard stipulates the gas chromatography-mass spectrometry (GC-MS) method for the determination of 1,2-dichloroethane, ethylene chlorohydrin and chloroacetic acid in textiles. This Standard is applicable to various types of textile products.

2 Normative References

The following documents are indispensable to the application of this document. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

3 Principle

Use methanol to conduct ultrasonic extraction of 1,2-dichloroethane, ethylene chlorohydrin and chloroacetic acid in textile products. Use filter membrane to filtrate and purify the extract. Then, adopt gas chromatograph-mass spectrometer to determine 1,2-dichloroethane, ethylene chlorohydrin and chloroacetic acid. After methyl ester derivatization, tert-butyl methyl ether extraction and filtration and purification with filter membrane, adopt gas chromatograph-mass spectrometer to determine chloroacetic acid in the extract. Use external standard method to quantify it.

4 Reagents

Unless it is otherwise stipulated, all the adopted reagents shall be analytically pure. Water shall be Grade-3 water, which is stipulated in GB/T 6682.

5.7 Vortex mixer.

5.8 Centrifuge: rotating speed shall be not lower than 3,500 r/min.

5.9 Muffle furnace.

5.10 Gas chromatograph-mass spectrometer: equipped with electron impact (EI) source.

6 Procedures of Determination

6.1 Extraction Take a representative sample; cut it into pieces under the size of 5 mm 5 mm; mix them up. Weigh-take (1.00 ± 0.01) g of sample; place it in the extractor (5.2). Add 10 mL of methanol (4.1), then, seal it up. In the ultrasonic generator (5.3), conduct constant-temperature extraction for 30 min. Thus, obtain the extract.

6.2 Preparation of Sample Solution

6.2.1 Preparation of 1,2-dichloroethane and ethylene chlorohydrin sample solution Use the filter membrane (5.4) to filter the extract (6.1). It shall be used for gas chromatography-mass spectrometry, and the determination of 1,2-dichloroethane and ethylene chlorohydrin.

6.2.2 Preparation of chloroacetic acid derivatized sample solution Accurately transfer-take

5.0 mL of the extract (6.1), then, place it into the centrifuge tube with a plug (5.5). Add

0.9 mL of concentrated sulfuric acid (4.3); accurately add 5 mL of tert-butyl methyl ether (4.2). After mixing it up, place it still in water bath at (50 ± 2) °C and conduct esterification reaction for 60 min. After cooling it down in ice bath, add 2 mL of sodium sulfate solution (4.6). After vortex mixing and centrifugation, remove the lower layer of solution. Then, add 2 mL of statured sodium carbonate solution (4.7). After vortex mixing, conduct centrifugation, remove the lower layer of solution. Add around 2 g of anhydrous sodium sulfate (4.4) to

dehydrate it. After filtering it through the filter membrane (5.4), reserve it for gas chromatography-mass spectrometry, and the determination of chloroacetic acid. NOTE 1: when adding concentrated sulfuric acid, slowly add it drop-by-drop along the tube wall, so as to prevent dramatic temperature rise and bumping of the solution. NOTE 2: when adding saturated sodium carbonate solution, slowly add it, so as to prevent bumping of the solution. NOTE 3: during the experiment, safety goggles and protective gloves should be used, so In accordance with the conditions stipulated in 6.4.1, respectively conduct iso-volumetric inserted sample injection into the standard working solution and the sample solution. The response value of the substance being tested in the standard working solution and the sample solution shall be within the linear range of instrument detection. If the content exceeds the range of the standard curve, dilute the sample solution into an appropriate concentration, then, conduct the analysis. If in the selective ion chromatogram of the sample solution and the standard working solution, chromatographic peak emerges in the same retention time, then, take the type and the abundance ratio of selective ions in Appendix A as a reference for qualitative determination; use external standard method to quantify it. Under the above- mentioned analytical conditions, the selective ion chromatogram and mass spectrum of gas chromatography-mass spectrometry of 1,2-dichloroethane, ethylene chlorohydrin and methyl chloroacetate shall take Appendix B as a reference.

6.5 Blank Test Except from the step of adding sample, comply with the remaining steps in 6.1 ~ 6.4.

7 Result Calculation and Expression

7.1 Calculation The content of 1,2-dichloroethane, ethylene chlorohydrin and chloroacetic acid in the sample shall be calculated in accordance with Formula (1): Where, Xi

---content of test component in the sample, expressed in (mg/kg); ci

---concentration of test component solution obtained from the standard working curve, expressed in (mg/L); c0

---concentration of blank test solution obtained from the standard working curve, expressed in (mg/L); V

---volume of extracting solution, expressed in (mL); f

---dilution factor; m

---sample's mass, expressed in (g).