NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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Textiles - Determination of organotin compounds - Part 1: Derivatisation method using GC-MS

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1 Scope

This document specifies the test method of determining organotin compounds (see Table 1) in textile products through the derivatisation method using GC-MS.

This document is applicable to the various types of textile products.

2 Normative References

Through the normative references in this text, the contents of the following documents constitute indispensable clauses 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 (GB/T 6682-2008, ISO 3696:1987, MOD)

3 Terms and Definitions

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

4 Principle

Take tropolone solution as the complexing agent and use methanol - ethanol mixed solvent to extract organotin compounds in the textiles.

Through the reaction with sodium tetraethyl-boride, convert the polar and high-boiling organotin into the corresponding volatile alkyl derivatives. Use gas chromatograph - mass spectrometer (GC-MS) for determination and adopt the internal standard method for quantitative determination.

5 Reagents

Unless it is otherwise specified, all reagents are analytically pure.

5.9 Tetra-n-propyltin, CAS No.: 2176-98-9 (internal standard substance).

NOTE: if the recovery rate of the internal standard substances (5.6, 5.7, 5.8 and 5.9) is relatively low, other internal standard substances (for example, deuterated compounds) may be selected (see also 9.1).

6.6 pH meter, equipped with a glass composite electrode, with a measuring range of 0 ~ 14 and

the measurement accuracy accurate to 0.1.

6.7 Ultrasonic generator, with a working frequency of (40 ? 5) kHz and the temperature

controllable.

6.9 Horizontal mechanical vibrator.

NOTE: the minimum frequency of the horizontal vibrator is 5 s?1, and the amplitude is 2 cm ~ 5 cm.

6.10 Volumetric flask, with a volume of 10 mL ~ 500 mL.

NOTE: before use, all glassware shall be soaked in nitric acid (volume fraction: 5%) for 24 h, and rinsed with water.

7 Sample

The specimen is taken from a single texture of the textile product, for example, fabric, coating material, polymer or other textures. The specimen preparation process includes separating a single texture from the product and preparing it into small pieces with a

maximum dimension not exceeding 4 mm.

NOTE: in consideration of the detection limit and quantification limit, for samples of the same texture, a maximum of three samples can be mixed in testing, and the three samples shall be of equal mass.

8.1 General

SAFETY WARNING: since sodium tetraethyl-borate is air-sensitive and will spontaneously ignite in air, sodium tetraethyl-borate solution shall be prepared in a large-volume fume hood. Organotin is toxic and endocrine disruptor, so special care shall be taken.

NOTE: before use, all reagents stored below room temperature should be taken out and placed to reach room temperature.

8.2 Preparation of Sodium Tetraethyl-borate Solution

In an inert environment, weigh-take 2 g of sodium tetraethyl-borate (5.4); add 10 mL of tetrahydrofuran (5.5) to dissolve it.

Store this solution in an inert gas (5.11) environment. The validity period is 3 months.

NOTE: sodium tetraethyl-borate or its solution can be purchased in fixed weights. An example of preparation: transfer-take 10 mL of tetrahydrofuran into a 40 mL sample bottle with a screw cap; open the 2 g package of sodium tetraethyl-borate; add it to the sample bottle and dissolve it. If the sodium tetraethyl-borate is in 5 g package, accordingly increase the volume of the added tetrahydrofuran to ensure a concentration of approximately 0.2 g/mL.

8.3.1 Conversion of organotin cation

The standard substance of organotin chloride is available on the market, but the mass concentration of the standard curve and the expression of results are all expressed in terms of organotin cation, and the unit is mg/kg.

Example 1: the corresponding cation of Bu2SnCl2 is Bu2Sn2+.

Table 2 provides the amount of organotin chloride and the weighting factor for re-calculation

of cation (assuming that the chloride purity is 100%).

The target compound solution can be directly purchased with a certificated standard solution, or prepared by the user.

Each substance is separately prepared: use the analytical balance (6.2) to weigh-take an appropriate amount of the target compound (see Table 1); select a volumetric flask with an appropriate volume; use methanol (5.13) to dissolve it and reach a constant volume to the scale.

The concentration of the organotin cation is 1,000 mg/L.

Store the standard solution in the refrigerator to reduce the volatilization of the solvent, and the storage period shall not exceed one year.

8.3.5 Target compound - working solution (the mass concentration of organotin cation is

10 mg/L)

Use the pipette (6.5) to transfer-take an appropriate volume of the target compound - stock solution (8.3.4) to a volumetric flask of an appropriate volume. Use methanol (5.13) to reach a constant volume to the scale. Thus, the target compound - working solution with organotin cation concentration of 10 mg/L is obtained.

Store the target compound - working solution in the refrigerator to reduce the volatilization of the solvent, and the storage period shall not exceed one month.

8.4 Preparation of Tropolone Solution

Use the analytical balance (6.2) to weigh-take 0.500 g of tropolone (5.12) into a glass beaker;

use about 20 mL of methanol (5.13) to dissolve it, then, transfer it to a 100 mL volumetric flask.

Use methanol to dilute to the scale. Thus, tropolone solution with a concentration of 5 g/L is obtained.

Store this solution in the refrigerator to reduce the volatilization of the solvent, and the storage period is one month.

8.5 Preparation of Buffer Solution

Prepare sodium acetate solution with a concentration of 0.2 mol/L. For example, weigh-take

16.4 g of anhydrous sodium acetate (5.14) into 1 L of water (5.1); use glacial acetic acid (5.3)

to adjust the pH value to 4.5.

8.6.1 Optionally prepare standard solutions with a concentration of 100 ?g/L, 200 ?g/L, 300

?g/L, 400 ?g/L and 500 ?g/L.

8.6.2 Use a micropipette (6.4) to draw 20 ?L, 40 ?L, 60 ?L, 80 ?L and 100 ?L of the target