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**Textiles and textile products —  
Determination of organotin  
compounds —**

**Part 1:  
Derivatisation method using gas  
chromatography**

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## Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 248, *Textiles and textile products*, in collaboration with ISO Technical Committee ISO/TC 38, *Textiles*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

# Textiles and textile products — Determination of organotin compounds —

## Part 1: Derivatisation method using gas chromatography

**WARNING** — The use of this document involves hazardous materials. It does not purport to address all of the safety or environmental problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel and the environment prior to application of the document.

### 1 Scope

This document specifies a test method for the qualification and quantification of organotin compounds. This test method is applicable to all types of materials of textile products.

NOTE CEN/TR 16741 defines which materials are applicable to this determination.

### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

### 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

### 4 Principle

The organotin substances are extracted from the material of a textile product with a methanol-ethanol mixture using tropolone as a complexing agent.

The polar and high-boiling organotin is then converted to the corresponding volatile alkyl derivative, by reaction with sodium tetraethylborate, NaB(Et)<sub>4</sub>. Finally, it is detected and quantified by using a gas chromatograph fitted with a mass selective detector (GC-MS).

[Table 1](#) indicates the list of target compounds which can be analysed with this document.

This document is also applicable for further organotin substances provided that the method is validated with the additional compounds.

Table 1 — List of target compounds that can be analysed and internal standards

Type of compound	Compound	CAS <sup>a</sup> number
Monosubstituted	Internal standard: n-Heptyltin trichloride	59344-47-7
	Methyltin trichloride	993-16-8
	n-Butyltin trichloride	1118-46-3
	n-Octyltin trichloride	3091-25-6
	Phenyltin trichloride	1124-19-2
Disubstituted	Internal standard: Di-n-heptyltin dichloride	74340-12-8
	Dimethyltin dichloride	753-73-1
	Di-n-propyltin dichloride	867-36-7
	Di-n-butyltin dichloride	683-18-1
	Di-n-octyltin dichloride	3542-36-7
	Diphenyltin dichloride	1135-99-5
Trisubstituted	Internal standard: Tri-n-pentyltin chloride	3342-67-4
	Trimethyltin chloride	1066-45-1
	Tri-n-propyltin chloride	2279-76-7
	Tri-n-butyltin chloride <sup>b</sup>	1461-22-9
	Tri-n-octyltin chloride	2587-76-0
	Triphenyltin chloride (or fentin chloride)	639-58-7
	Tricyclohexyltin chloride	3091-32-5
Tetrasubstituted	Internal standard: Tetra-n-propyltin	2176-98-9
	Tetra-n-ethyltin	597-64-8
	Tetra-n-butyltin	1461-25-2
<sup>a</sup>	Chemical Abstract Service.	
<sup>b</sup>	If bis(tri-n-butyltin)oxide (TBTO), CAS number 56-35-9, is present, it is detected as tri-n-butyltin.	

## 5 Reagents

Unless otherwise specified, use only reagents of recognized analytical grade.

- 5.1 **Water**, grade 3 according to ISO 3696.
- 5.2 **Ethanol**, technical grade or industrial methylated spirit (IMS), CAS number: 64-17-5.
- 5.3 **Glacial acetic acid**, CAS number: 64-19-7.
- 5.4 **Sodium tetraethylborate**, CAS number: 15523-24-7.
- 5.5 **Tetrahydrofuran (THF)**, stabilized, CAS number: 109-99-9.
- 5.6 **n-Heptyltin trichloride**, CAS number: 59344-47-7 (internal standard).
- 5.7 **Di-n-heptyltin dichloride**, CAS number: 74340-12-8 (internal standard).
- 5.8 **Tri-n-pentyltin chloride**, CAS number: 3342-67-4 (internal standard).

**5.9 Tetra-n-propyltin**, CAS number: 2176-98-9 (internal standard).

If the recovery rate of the internal standards (5.6, 5.7, 5.8 and 5.9) is low, alternative internal standards may be used (for example deuterated compounds) (see also 9.1).

**5.10 Hexane**, CAS number: 110-54-3.

**5.11 Inert gas**, e.g. nitrogen or argon.

**5.12 Tropolone** (2-hydroxy-2,4,6-cycloheptatrien-1-one), CAS number: 533-75-5.

**5.13 Methanol**, of analytical grade, CAS number: 67-56-1.

**5.14 Sodium acetate**, CAS number: 127-09-3.

**5.15 Organotin compounds**, listed in [Table 1](#).

## 6 Apparatus and materials

The usual equipment and laboratory glassware, according to ISO 4787, shall be used, in addition to the following.

**6.1 Gas chromatograph** with a mass selective detector (GC-MS).

**6.2 Analytical balance**, with a resolution of 0,1 mg.

**6.3 Glass vessel**, with screw tops and a volume of, for example, 50 ml.

**6.4 Micropipettes**, 10 µl to 500 µl range, with disposable tips.

**6.5 Pipettes**, 1 ml to 10 ml capacity.

**6.6 Calibrated pH-meter**, with a glass combination electrode and range of 0 to 14.

**6.7 Ultrasonic bath**, with adjustable temperature suitable for operation at about 60 °C.

**6.8 Centrifuge**.

**6.9 Horizontal mechanical shaker**.

NOTE Horizontal shaker with minimum frequency of 5 s<sup>-1</sup>, path length 2 cm to 5 cm has been found suitable.

**6.10 Volumetric flasks**, 10 ml to 500 ml as required.

## 7 Preparation of the test piece

The test piece consists of a single material taken from the textile product, such as textile, coated material, polymer or other. The preparation of the sample should involve the removal of the individual materials from the textile product and the preparation of a test piece, which results in particles with a maximum edge length of 4 mm.

NOTE Up to three test specimens (of equal mass) of the same material type can be tested together, taking into consideration the limits of detection and quantification.

## 8 Procedure

### 8.1 General

**SAFETY PRECAUTIONS** — As sodium tetraethylborate is air-sensitive and can spontaneously combust in the presence of air, the solution using it shall be prepared in a high-volume fume hood. Organotins are both toxic and known endocrine system disrupters; therefore, they should be treated with utmost care.

All the chemicals that are stored below room temperature should be allowed to reach room temperature before an aliquot is taken.

### 8.2 Preparation of the sodium tetraethylborate solution

Weigh about 2 g of sodium tetraethylborate (5.4) into a 10 ml volumetric flask (6.10) and make up to volume with tetrahydrofuran (5.5).

This solution is stable for about three months if stored under an inert gas blanket (5.11).

NOTE Pre-weighed tetraethylborate or commercial solutions are available on the market.

### 8.3 Preparation of standard solutions

#### 8.3.1 General

The organotin compounds are available on the market as their chloride forms, but the mass concentration for the calibration curve and the result are expressed in mg/kg of organotin cations.

EXAMPLE 1 With dibutyltin dichloride,  $\text{Bu}_2\text{SnCl}_2$  (dibutyltin dichloride) is the chloride form and  $\text{Bu}_2\text{Sn}^{2+}$  is the cation form.

Table 2 gives the amount of organotin chloride and the weighting factor for recalculation of organotin cations (for 100 % purity of the chloride form).

**Table 2 — Amount of organotin chloride and weighting factor for recalculation of organotin cations**

Compound	Weighting factor	Amount of organotin chloride required to have a solution of 1 000 mg/l of organotin cation (in a 100 ml flask) mg
<b>Target compounds</b>		
Methyltin trichloride	0,557	179,5
n-Butyltin trichloride	0,623	160,5
n-Octyltin trichloride	0,686	145,8
Phenyltin trichloride	0,648	154,3
Dimethyltin dichloride	0,677	147,7
Di-n-propyltin dichloride	0,742	134,8
Di-n-butyltin dichloride	0,767	130,4
Di-n-octyltin dichloride	0,830	120,5
Diphenyltin dichloride	0,793	126,1
Trimethyltin chloride	0,821	121,8
Tri-n-propyltin chloride	0,875	114,3
Tri-n-butyltin chloride	0,891	112,2

<sup>a</sup> These compounds have no chloride and therefore the weighting factor is 1,000.



Table 2 (continued)

Compound	Weighting factor	Amount of organotin chloride required to have a solution of 1 000 mg/l of organotin cation (in a 100 ml flask) mg
Tri-n-octyltin chloride	0,927	107,9
Triphenyltin chloride	0,908	110,1
Tricyclohexyltin chloride	0,912	109,6
Tetra-n-ethyltin <sup>a</sup>	1,000	100,0
Tetra-n-butyltin <sup>a</sup>	1,000	100,0
<b>Internal standards</b>		
n-Heptyltin trichloride	0,672	148,8
Di-n-heptyltin dichloride	0,817	122,4
Tri-n-pentyl chloride	0,906	110,4
Tetra-n-propyltin <sup>a</sup>	1,000	100,0

<sup>a</sup> These compounds have no chloride and therefore the weighting factor is 1,000.

The mass concentration of organotin cation is usually calculated using [Formula \(1\)](#):

$$\rho_{\text{Sn}} = \rho_{\text{Cl}} \times W_F \quad (1)$$

where

$\rho_{\text{Sn}}$  is the mass concentration of organotin cation (mg/l);

$\rho_{\text{Cl}}$  is the mass concentration of organotin chloride (mg/l);

$W_F$  is the weighting factor.

**EXAMPLE 2** If you weigh 160,5 mg of monobutyltin trichloride ( $\text{BuSnCl}_3$ ), into 100 ml solvent, you have a solution of 1 605 mg/l of monobutyltin trichloride, which corresponds to a mass concentration of:  $1\ 605 \times 0,623 = 1\ 000$  mg/l of monobutyltin cation ( $\text{BuSn}^{3+}$ ).

**EXAMPLE 3** If you weigh 110,4 mg of dioctyltin dichloride [ $(\text{C}_8\text{H}_{17})_2\text{SnCl}_2$ ], into 100 ml solvent, you have a solution of 1 104 mg/l of dioctyltin dichloride, which corresponds to a mass concentration of:  $1\ 104 \times 0,830 = 916$  mg/l of dioctyltin cation [ $(\text{C}_8\text{H}_{17})_2\text{Sn}^{2+}$ ].

### 8.3.2 Internal standards — Stock solution (100 mg/l of organotin cation)

Internal standards are available commercially as certified solutions, or a solution of internal standards can be made. Four internal standards shall be used as solutions in methanol ([5.13](#)).

To prepare an internal standard solution use the analytical balance ([6.2](#)) to weigh the appropriate amount of n-heptyltin trichloride ([5.6](#)), di-n-heptyltin dichloride ([5.7](#)), tri-n-pentyltin chloride ([5.8](#)) and tetra-n-propyltin ([5.9](#)). Dissolve them together in methanol ([5.13](#)) in a single volumetric flask ([6.10](#)) to obtain the mass concentration of 100 mg/l for each organotin cation.

Store the standard solution for a maximum of one year in a refrigerator at about 6 °C, when not in use, to minimize evaporation of the solvent.

### 8.3.3 Internal standards — Working solution (10 mg/l of organotin cation)

Use a pipette ([6.5](#)) to transfer an appropriate volume of the internal standard solution ([8.3.2](#)) into a volumetric flask ([6.10](#)) and make the solution up to volume with methanol ([5.13](#)) to create a 10 mg/l solution of each organotin cation.