

Water quality — Determination of selected organotin compounds — Gas chromatographic method

The European Standard EN ISO 17353:2004 has the status of a British Standard

ICS 13.060.50

Confirmed July 2008

National foreword

This British Standard is the official English language version of EN ISO 17353:2005. It is identical with ISO 17353:2004.

The UK participation in its preparation was entrusted by Technical Committee EH/3, Water quality, to Subcommittee EH/3/2, Physical, chemical and biochemical methods, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Additional information

The responsible BSI committee would like to draw attention to the following. This international standard is applicable to waters containing a maximum of 2 g/l of suspended material. However, the application of this standard to samples containing amounts smaller than 2 g/l of suspended material can affect recovery during the alkylation and extraction stages. This is due to the adsorption of the suspended material onto the solid material. Once the performance of this standard has been verified, samples containing suspended material can be analysed.

Cross-references

The British Standards which implement international publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, the EN ISO foreword page, the ISO title page, pages ii to v, a blank page, pages 1 to 31 and a back cover.

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English Version

Water quality - Determination of selected organotin compounds -
Gas chromatographic method (ISO 17353:2004)

Qualité de l'eau - Dosage de composés organostanniques
sélectionnés - Méthode par chromatographie en phase
gazeuse (ISO 17353:2004)

Wasserbeschaffenheit - Bestimmung von ausgewählten
Organozinnverbindungen - Verfahren mittels
Gaschromatographie (ISO 17353:2004)

This European Standard was approved by CEN on 18 July 2005.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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Foreword

The text of ISO 17353:2004 has been prepared by Technical Committee ISO/TC 147 "Water quality" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 17353:2005 by Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2006, and conflicting national standards shall be withdrawn at the latest by February 2006.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 17353:2004 has been approved by CEN as EN ISO 17353:2005 without any modifications.

INTERNATIONAL STANDARD

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Water quality — Determination of selected organotin compounds — Gas chromatographic method

*Qualité de l'eau — Dosage de composés organostanniques
sélectionnés — Méthode par chromatographie en phase gazeuse*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 17353 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Introduction

It should be noted whether and to what extent particular problems will require the specification of additional boundary conditions.

This International Standard describes a gas-chromatographic/organotin specific determination of organotin compounds after derivatization with sodium tetraethyl borate and liquid/liquid extraction.

The user should be aware that particular problems could require the specification of additional marginal conditions.

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WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method for the identification and quantification of organotin compounds and/ or cations as mentioned in Table 1 in drinking water, surface water and wastewater containing not more than 2 g/l of suspended material. The working range is 10 ng/l to 1 000 ng/l. The respective anions are not determined.

This method can also be applicable to other compounds such as R = methyl, $n = 1$ to 2 and R = phenyl, $n = 1$ to 2. This International Standard is also applicable to marine water.

Table 1 — Organotin compounds and cations determined using this International Standard

$R_n\text{Sn}^{(4-n)+}$	R	n	Name	Acronym
BuSn^{3+}	Butyl	1	Monobutyltin cation	MBT
$\text{Bu}_2\text{Sn}^{2+}$	Butyl	2	Dibutyltin cation	DBT
Bu_3Sn^+	Butyl	3	Tributyltin cation	TBT
Bu_4Sn	Butyl	4	Tetrabutyltin	TTBT
OcSn^{3+}	Octyl	1	Monooctyltin cation	MOT
$\text{Oc}_2\text{Sn}^{2+}$	Octyl	2	Diocetyl tin cation	DOT
Ph_3Sn^+	Phenyl	3	Triphenyltin cation	TPhT
Cy_3Sn^+	Cyclohexyl	3	Tricyclohexyltin cation	TCyT

2 Normative references

The following referenced documents are indispensable for the application 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design on sampling programmes*

ISO 5667-2, *Water quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1

organotin compound

OTC

substance with at least one Sn-C bond

NOTE The number of Sn-C bonds is a measure of the degree of substitution.

3.2

organotin cation

OC

part of the organotin compound that contains all Sn-C bonds (and which is formally loaded)

NOTE In this International Standard, the abbreviation OC is also used for the non-dissociated tetrasubstituted organotin. OC therefore comprises the cations MBT, DBT, TBT, TTBt, MOT, DOT, TCyT, and TPhT.

4 Principle

Organotin compounds in water are alkylated with sodium tetraethylborate and extracted with hexane. The extract can be cleaned with silica. After concentration, the tetrasubstituted OTC are separated by capillary gas chromatography and detected with a suitable system such as MS (mass spectrometry), FPD (flame photometric detection), AED (atomic emission detection). The concentration is determined by calibration for the total procedure using an internal standard mixture.

5 Interferences

The reagents sometimes contain impurities of organotin compounds. It is absolutely essential to verify the blanks (see A.4.6).

6 Reagents

Use reagents of highest purity.

6.1 Water, free of substances causing interference with this method and complying with ISO 3696:1987, Grade 1.

6.2 Nitric acid, $\rho(\text{HNO}_3) = 1,4 \text{ g/ml}$.

6.3 Acetic acid, CH_3COOH , glacial.

6.4 Sodium hydroxide solution, $c(\text{NaOH}) = 1 \text{ mol/l}$.

6.5 Sodium acetate, CH_3COONa , anhydrous.

- 6.6 Sodium sulfate**, Na_2SO_4 , anhydrous.
- 6.7 Silica**, grain size 0,2 mm to 0,063 mm (200 mesh to 63 mesh).
- 6.8 Tetrahydrofurane**, $\text{C}_4\text{H}_8\text{O}$, free of peroxides and water.
- 6.9 Acetone**, (propanone) $(\text{CH}_3)_2\text{CO}$.
- 6.10 Methanol**, CH_3OH .
- 6.11 Hexane**, C_6H_{14} .
- 6.12 Sodium tetraethylborate**, $\text{NaB}(\text{C}_2\text{H}_5)_4$.
- 6.13 Monobutyltin trichloride**, MBTCl, $\text{C}_4\text{H}_9\text{SnCl}_3$.
- 6.14 Dibutyltin dichloride**, DBTCl, $(\text{C}_4\text{H}_9)_2\text{SnCl}_2$.
- 6.15 Tributyltin chloride**, TBTCl, $(\text{C}_4\text{H}_9)_3\text{SnCl}$.
- 6.16 Tetraethyltin**, TTBT, $(\text{C}_4\text{H}_9)_4\text{Sn}$.
- 6.17 Mono-octyltin trichloride**, MOTCl, $\text{C}_8\text{H}_{17}\text{SnCl}_3$.
- 6.18 Dioctyltin dichloride**, DOTCl, $(\text{C}_8\text{H}_{17})_2\text{SnCl}_2$.
- 6.19 Triphenyltin chloride**, TPhTCl, $(\text{C}_6\text{H}_5)_3\text{SnCl}$.
- 6.20 Tricyclohexyltin chloride**, TCyTCl, $(\text{C}_6\text{H}_{11})_3\text{SnCl}$.
- 6.21 Monoheptyltin trichloride**, MHTCl, $\text{C}_7\text{H}_{15}\text{SnCl}_3$; (internal standard).
- 6.22 Diheptyltin dichloride**, DHTCl, $(\text{C}_7\text{H}_{15})_2\text{SnCl}_2$; (internal standard).
- 6.23 Tripropyltin chloride**, TPTCl, $(\text{C}_3\text{H}_7)_3\text{SnCl}$; (internal standard).
- 6.24 Tetrapropyltin**, TTPT, $(\text{C}_3\text{H}_7)_4\text{Sn}$; (internal standard).
- 6.25 Multicomponent solutions and prepared reagents.**

Since stability of multicomponent standard solutions is a matter of concern, it is recommended to prepare several solutions containing solely organotin compounds with the same degree of alkylation/arylation (e.g. four solutions respectively for mono-, di-, tri-, and tetrasubstituted compounds). Stability can be assessed by the absence of degradation products.

6.25.1 Multicomponent-standard solution in methanol, Stock solution A.

For the preparation of 1 mg/ml of organotin cation stock solution, weigh, to the nearest 0,1 mg, the amounts of organotin compounds specified in Table 2 into a 100 ml volumetric flask. Dissolve these compounds in a small amount of methanol (6.10). Then make up to volume with methanol and mix well.

If stored at 4 °C in the dark, the solution is stable for up to one year.