
**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*.

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

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.

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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.

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4 Principle

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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 **Tetrabutyltin**, TTBT, $(\text{C}_4\text{H}_9)_4\text{Sn}$.
- 6.17 **Monooctyltin trichloride**, MOTCl, $\text{C}_8\text{H}_{17}\text{SnCl}_3$.
- 6.18 **Diocetyl tin 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.

6.25.2 Solution of the internal standards in methanol, Stock solution B.

Weigh, to the nearest 0,1 mg, into a 100 ml volumetric flask, about:

- 120 mg of diheptyltin dichloride,
- 150 mg of monoheptyltin trichloride,
- 115 mg of tripropyltin chloride, and
- 100 mg of tetrapropyltin

and dissolve in a small amount of methanol (6.10). Make up to volume with methanol and mix well.

For the preparation of exactly 1 mg/ml of OC as specified in Table 2, weigh exactly 122,4 mg of diheptyltin dichloride, 148,8 mg of monoheptyltin trichloride, 114,3 mg of tripropyltin chloride and 100 mg of tetrapropyltin into the 100 ml volumetric flask and prepare as described above.

The solution is stable for three months when stored in the dark at 4 °C.

Table 2 — Amounts of organotin compounds and weighing factors for recalculation to organotin cations (for 100 % purity of the substances)

Substance	Weighing factor ^a	Mass ^b mg	Solution ^c
Monobutyltin trichloride	0,623	160,5	A
Dibutyltin dichloride	0,767	130,4	A
Tributyltin chloride	0,891	112,2	A
Tetrabutyltin	1,000	100,0	A
Monooctyltin trichloride	0,686	145,8	A
Diocetyl tin dichloride	0,830	120,5	A
Triphenyltin chloride	0,908	110,1	A
Tricyclohexyltin chloride	0,912	109,6	A
Monoheptyltin trichloride	0,672	148,8	B
Diheptyltin dichloride	0,817	122,4	B
Tripropyltin chloride	0,875	114,3	B
Tetrapropyltin	1,000	100,0	B

^a Weighing factor = molar mass (OC)/molar mass (OTC).
^b If the mass weighed of the compound is different from that given in this table, use the weighing factor to calculate the actual concentration of the OTC.
^c A for multicomponent standard solution in methanol.
 B for the solution of the internal standards in methanol.

6.25.3 Multicomponent spiking solutions for reference solutions.

Prepare the spiking solutions as specified in Table 3 using pipettes. Pipette the respective starting volume specified in Table 3 of the (stock) solution into a 100 ml volumetric flask. Make up to volume with methanol and mix well. The final mass concentration of the resulting spiking solution shall be between 10 ng/ml and 1 000 ng/ml of organotin cations in methanol. Dilutions steps greater than 1:100 are not allowed.

Table 3 — Example for the dilution series for preparation of the spiked solutions

Starting solution	Concentration of OC in starting solution ng/ml	Volume of starting solution ml	Final volume ml	Final concentration of OC ng/ml	Designation of final solution
A	1 000 000	10	100	100 000	A1
A1	100 000	10	100	10 000	A2
A1	100 000	15	100	15 000	A3
A2	10 000	20	50	4 000	A4
A2	10 000	5	100	500	A5
A2 + A3	10 000/15 000	1 + 6	100	1 000	AH6
A2 + A3	10 000/15 000	1 + 5	100	850	AH5
A2 + A3	10 000/15 000	1 + 4	100	700	AH4
A2 + A3	10 000/15 000	1 + 3	100	550	AH3
A2 + A3	10 000/15 000	1 + 2	100	400	AH2
A2 + A3	10 000/15 000	1 + 1	100	250	AH1
A4	4 000	6	100	240	AM6
A4	4 000	5	100	200	AM5
A4	4 000	4	100	160	AM4
A4	4 000	3	100	120	AM3
A4	4 000	2	100	80	AM2
A4	4 000	1	100	40	AM1
A5	500	7	100	35	AL6
A5	500	6	100	30	AL5
A5	500	5	100	25	AL4
A5	500	4	100	20	AL3
A5	500	3	100	15	AL2
A5	500	2	100	10	AL1

Solutions AH1 to AH6 are used for calibration AH (higher working range).

Solutions AM1 to AM6 are used for calibration AM (medium working range).

Solutions AL1 to AL6 are used for calibration AL (lower working range).

Solutions AH1 to AH6 are prepared from two Stock solutions A2 and A3.

6.25.4 Spiking solutions containing internal standards.

Dilution steps greater than 1:100 are not allowed.

Pipette 1 ml of Stock solution B (see Table 4) into a 100 ml volumetric flask. Make up to volume with methanol (6.10) and mix well (Solution B1).

Pipette 1 ml of Solution B1 into a 100 ml volumetric flask. Make up to volume with methanol and mix well (Solution B2).

Use Solution B2 for all samples. The mass concentration of Solution B2 is about 100 ng/ml of organotin cation in methanol, depending on the original mass weighed according to 6.25.1 (Table 2).

Table 4 — Example of a dilution series for the preparation of the spiking solutions of the internal standards

Starting solution	Concentration of OC in starting solution ng/ml	Volume of starting solution ml	Final volume ml	Final concentration of OC ng/ml	Designation of final solution
B	1 000 000	1	100	10 000	B1
B1	10 000	1	100	100	B2

6.25.5 Blank solution, consisting of 1 l of water (6.1) put into a 1 000 ml sampling bottle.

6.25.6 Reference solutions (aqueous multicomponent reference solution).

For each working range, prepare at least six reference solutions, distributed equidistantly over the chosen working range. The working range should not exceed one order of magnitude.

Add 1 000 ml of water (6.1) to each of six 1 000 ml sampling bottles. Under vigorous stirring, dip the tip of the pipette below the surface of the water and add 1 ml of the respective spiking solution (AH1 to AH6, AM1 to AM6, or AL1 to AL6, see Table 3) ensuring that the spiking solution is distributed evenly in the water. Stir for an additional 20 min.

Depending on the working range, the reference solutions shall contain the following mass concentration of OC in water:

- higher range: 1 000 ng/l, 850 ng/l, 700 ng/l, 550 ng/l, 400 ng/l and 250 ng/l;
- medium range: 240 ng/l, 200 ng/l, 160 ng/l, 120 ng/l, 80 ng/l and 40 ng/l; or
- lower range: 35 ng/l, 30 ng/l, 25 ng/l, 20 ng/l, 15 ng/l, and 10 ng/l.

6.25.7 Acetate buffer solution.

Dissolve about 1 mol of sodium acetate (equal to 82 g of anhydrous sodium acetate) (6.5) in 500 ml of water (6.1) into a 1 000 ml volumetric flask. Add sufficient glacial acetic acid (6.3) to reach a pH of 4,5. Make up to volume with water (6.1) and mix well.

6.25.8 Derivatization agent A (2 % mass concentration in water).

Weigh about 200 mg of sodium tetraethylborate (6.12) into a 10 ml volumetric flask and make up to volume with water (6.1).

This solution is not stable, and should be used immediately.

6.25.9 Derivatization agent B (20 % mass concentration in tetrahydrofuran).

Weigh about 2 g of sodium tetraethylborate (6.12) into a 10 ml volumetric flask and make up to volume with tetrahydrofuran (6.8).

This solution is stable for about three months if stored under an inert gas blanket. Its use is recommended for large series of samples.

6.25.10 Drying agent.

Place about 250 g to 300 g of powdered sodium sulfate (6.6) onto a quartz plate and dry for at least 4 h at a temperature of 180 °C. Add the dried sodium sulfate to a wide-necked bottle and allow to cool to room temperature in a desiccator. Remove the bottle from the desiccator and close tightly.