SLOVENSKI STANDARD
oSIST prEN ISO 23161:2017

01-december-2017

Kakovost tal - Določevanje izbranih organokositrovih spojin - Metoda plinske kromatografije (ISO/DIS 23161:2017)

Soil quality - Determination of selected organotin compounds - Gas-chromatographic method (ISO/DIS 23161:2017)

Bodenbeschaffenheit - Bestimmung ausgewählter Organozinnverbindungen - Gaschromatographisches Verfahren (ISO/DIS 23161:2017)

Qualité du sol - Dosage d'une sélection de composés organostanniques - Méthode par chromatographie en phase gazeuse (ISO/DIS 23161:2017)

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ICS:

13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

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DRAFT INTERNATIONAL STANDARD

ISO/DIS 23161

ISO/TC **190**/SC **3** Secretariat: **DIN**

Voting begins on: **2017-10-20**

Voting terminates on: **2018-01-12**

Soil quality — Determination of selected organotin compounds — Gas-chromatographic method

Qualité du sol — Dosage d'une sélection de composés organostanniques — Méthode par chromatographie en phase gazeuse

ICS: 13.080.10

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Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
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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).

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The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics.*

This second edition cancels and replaces the first edition (ISO 23161:2009), of which it constitutes a full revision.

The changes compared to the previous edition are as follows:

— Note to Clause 1 and Table 2 have been moved to Clause 4;

— Former Note 4 to Clause 4 has been changed to normal text and moved above Note 1;

— Former second sentence in 5.5.5 has been changed to Note;

— In 7.2.2 and 7.2.3, a Note has been added;

— Presentation of tables in Annex E has been improved;

— Bibliography has been updated.

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Introduction

It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

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

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Soil quality — Determination of selected organotin compounds — Gas-chromatographic method

WARNING — Persons using this document should be familiar with usual laboratory practice. This document 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 in accordance with this document, be carried out by suitably qualified staff.

1 Scope

This document specifies a gas-chromatographic method for the identification and quantification of organotin compounds (OTCs) in soils as specified in Table 1.

This document is also applicable to samples from sediments, sludges and wastes (soil-like materials).

The working range depends on the detection technique used and the amount of sample taken for analysis.

The limit of quantification for each compound is about 10 µg/kg.

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Table 1 — Organotin compounds, which can be determined in accordance with this document

$R_nSn^{(4-n)+}$	R	*n*	Name	Acronym
Organotin cations[a]				
$BuSn^{3+}$	Butyl	1	Monobutyltin cation	MBT
Bu_2Sn^{2+}	Butyl	2	Dibutyltin cation	DBT
Bu_3Sn^{+}	Butyl	3	Tributyltin cation	TBT
$OcSn^{3+}$	Octyl	1	Monooctyltin cation	MOT
Oc_2Sn^{2+}	Octyl	2	Dioctyltin cation	DOT
Ph_3Sn^{+}	Phenyl	3	Triphenyltin cation	TPhT
Cy_3Sn^{+}	Cyclohexyl	3	Tricyclohexyltin cation	TCyT
Peralkylated organotin				
Bu_4Sn	Butyl	4	Tetrabutyltin	TTBT

[a] Organotin compounds are measured after derivatization.

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Organotin cations can only be determined in accordance with this document after derivatization. The anionic part bound to the organotin cation is mainly dependent on the chemical environment and is not determined using this method. The peralkylated organotin compounds behave in a completely different

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way from their parent compounds. Tetraalkylated organotin compounds which are already peralkylated, such as tetrabutyltin, are determined directly without derivatization.

The properties such as particle size distribution, water content and organic matter content of the solids to be analysed using this document vary widely. Sample pretreatment is designed adequately with respect to both the properties of the organotin compounds and the matrix to be analysed.

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 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

ISO 14507, *Soil quality — Pretreatment of samples for determination of organic contaminants*

ISO 16720, *Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis*

ISO 22892, *Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry*

3 Terms and definitions

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

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

— IEC Electropedia: available at http://www.electropedia.org/

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— ISO Online browsing platform: available at http://www.iso.org/obp

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3.1
organotin compound
substance containing 1 to 4 Sn-C bonds

Note 1 to entry: The number of Sn-C bonds is a measure for the degree of substitution.

3.2
organotin cation
part of the organotin compound (3.1) that contains all Sn-C bonds and is formally charged

3.3
organotin cation derivatives
non-dissociated tetrasubstituted organotin compounds which are produced by derivatization

3.4
solid
soil, sediment, sludge and waste (soil-like material)

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4 Principle

For the ionic and the non-ionic organotin compounds (see Table 1), a different sample pretreatment and sample preparation are necessary. For the determination of organotin cations, laboratory samples are pretreated by freeze drying and grinding. This procedure enables to achieve homogeneity of the sample. The determination of non-ionic TTBT cannot be carried out with freeze-dried materials due to evaporation losses; thus, it shall be determined in the field-moist sample. Organotin cations can only be determined after derivatization, whereas TTBT is already peralkylated and can be determined without derivatization (see the flowchart in Figure 1).

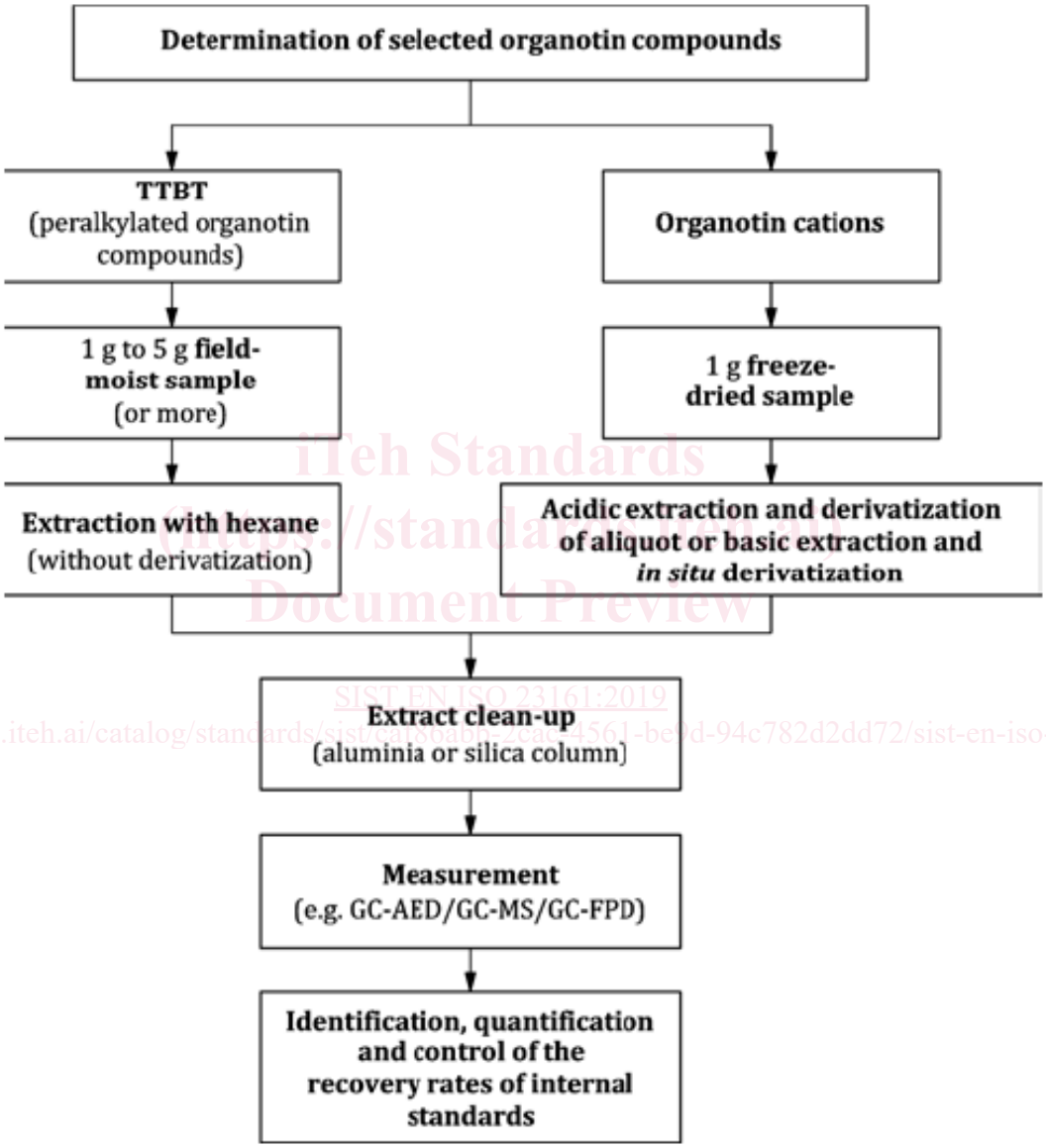

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Figure 1 — Flowchart for the pretreatment and analysis of selected organotin compounds

Beside freeze drying other pretreatment procedures can be carried out, if the suitability has been proven.

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For the determination of organotin compounds, two alternative extraction methods are given, both followed by in situ derivatization with a tetraethylborate compound and simultaneous extraction with hexane:

a) treatment with acetic acid;

b) treatment with methanolic potassium hydroxide.

Treatment with potassium hydroxide provides some degree of digestion and is recommended especially when the solid contains high amounts of organic and biological materials.

NOTE 1 If it is necessary to take a large amount of sample, extraction and derivatization can be done in two steps. An aliquot of the extract can be taken for derivatization. This also applies for samples with high levels of contamination by organotin compounds.

NOTE 2 During in situ derivatization, the solid phase is still present. This supports the extraction by continuous changing of the polar organotin cations to the non-polar organotin cation derivates. In situ methods can improve the extraction efficiency, particularly for monoalkylated organotin compounds.

NOTE 3 Other extraction techniques can be applied if a comparable extraction efficiency is achieved.

When applying this method to the determination of other organotin compounds not specified in the scope, its suitability has to be proven by proper in-house validation experiments, e.g. methyltin compounds (see Table 2). Methyltin cations are unlikely to evaporate from aqueous solvents, but peralkylated methyltin compounds are volatile and subject to losses (see C.3). Therefore, additional precautions are established.

Table 2 — Methyltin compounds

$R_nSn^{(4-n)+}$	R	*n*	**Name**	**Acronym**
$MeSn^{3+}$	Methyl	1	Monomethyltin cation	MMT
Me_2Sn^{2+}	Methyl	2	Dimethyltin cation	DMT
Me_3Sn+	Methyl	3	Trimethyltin cation	TMT

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The internal standard mix comprises four compounds representing four alkylation states in order to mimic the behaviour of the target compounds. After alkylation, they cover a wide range of volatility. A recovery of at least 80 % for derivatization/extraction and again 80 % for each clean-up step of the internal standard compounds should be achieved. (For more information, see A.3.) Tetraalkylborate is very reactive and will also alkylate other compounds in the matrix. Those compounds (and also boroxines) may interfere with the target compounds during gas chromatographic determination and influence detection. In order to protect the column and to reduce the interference in chromatography, it will be necessary to apply a pre-cleaning step to most samples. Clean-up with silica or aluminium oxide is the minimum; further clean-up steps (e.g. aluminium oxide/silver nitrate, silica/silver nitrate, pyrogenic copper; see Annex B) may be applied if necessary.

The determination of the tetrasubstituted organotin compounds is carried out after clean-up and concentration steps by separation with capillary gas chromatography and detected with a suitable system [mass spectrometer (MS), (MS/MS), flame photometric detector (FPD), atomic absorption spectrometer (AAS), atomic emission detector (AED), inductively coupled plasma/mass spectrometer ICP/MS]. The concentrations are determined by calibration over the total procedure using aqueous multi-component calibration standard solutions in accordance with 5.4.3.

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