
**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*



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

This document is a preview generated by EVS



COPYRIGHT PROTECTED DOCUMENT

© ISO 2009

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	2
3 Terms and definitions	2
4 Principle	2
5 Reagents	4
5.1 General	4
5.2 Chemicals	4
5.3 Standards	5
5.4 Preparation of reagents and solutions	6
5.5 Clean-up	7
6 Apparatus	7
6.1 Requirements for glassware	7
6.2 Sampling apparatus	8
6.3 Additional apparatus	8
7 Procedure	8
7.1 Sampling and sample pretreatment	8
7.2 Sample extraction	9
7.3 Clean-up of the extract	10
7.4 Determination of dry mass	11
7.5 Measurement	11
8 Calibration	12
9 Recovery rates of the internal standard compounds	12
10 Quantification	13
11 Expression of results	14
12 Precision	14
13 Test report	14
Annex A (informative) Information about the procedure	15
Annex B (informative) Additional clean-up procedures	17
Annex C (informative) Information about typical instrumental conditions	20
Annex D (informative) Information about GC/MS identification	31
Annex E (informative) Validation data	33
Bibliography	37

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 23161 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Introduction

It is absolutely essential that tests conducted in accordance with this International Standard 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.

This document is a preview generated by EVS

Soil 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.

1 Scope

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

Table 1 — Organotin compound, which can be determined in accordance with this International Standard

$R_n\text{Sn}^{(4-n)+}$	R	<i>n</i>	Name	Acronym
Organotin cations^a				
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
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
Peralkylated organotin				
Bu_4Sn	Butyl	4	Tetrabutyltin	TTBT
^a Organotin compounds are measured after derivatization.				

NOTE When applying this method to the determination of other organotin compounds not specified in the scope, its suitability is 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

$R_n\text{Sn}^{(4-n)+}$	R	<i>n</i>	Name	Acronym
MeSn^{3+}	Methyl	1	Monomethyltin cation	MMT
$\text{Me}_2\text{Sn}^{2+}$	Methyl	2	Dimethyltin cation	DMT
Me_3Sn^+	Methyl	3	Trimethyltin cation	TMT

Organotin cations can only be determined in accordance with this International Standard 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 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 International Standard 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 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, *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 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

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

3.1

organotin compound

substance containing 1 to 4 Sn-C bonds

NOTE 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)

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 homogeneity of the sample to be achieved. 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).