

ISO 23161:2018 (E)

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

Contents

	Foreword
1	Scope
2	Normative references
3	Terms and definitions
4	Principle
5	Reagents
5.1	General
5.2	Chemicals
5.3	Standards
5.4	Preparation of reagents and solutions
5.4.1	General requirements
5.4.2	Blank solution
5.4.3	Aqueous calibration solutions (multicomponent solution of organotin compounds in water)
5.4.4	Methanolic potassium hydroxide solution
5.4.5	Acetate buffer solution
5.4.6	Solvent mixture
5.4.7	Derivatization agent
5.5	Clean-up
5.5.1	General requirements
5.5.2	Silica gel for the clean-up column
5.5.3	Aluminium oxide for the clean-up column
5.5.4	Clean-up column
5.5.5	Eluent for extract cleaning with silica gel
5.5.6	Eluent for extract cleaning with aluminium oxide
6	Apparatus
7	Procedure
7.1	Sampling and sample pretreatment
7.2	Sample extraction
7.2.1	General
7.2.2	Acidic extraction and derivatization of an aliquot
7.2.3	Alkaline treatment and in situ derivatization
7.2.4	Separate determination of TTBT in the field-moist sample
7.3	Clean-up of the extract
7.3.1	General
7.3.2	Silica and aluminium oxide clean-up
7.4	Determination of dry mass
7.5	Measurement
7.5.1	Gas chromatographic separation
7.5.2	Detection and identification
8	Calibration
9	Recovery rates of the internal standard compounds
10	Quantification

11 Expression of results

12 Validation

13 Test report

Annex A (informative) Information about the procedure

- A.1 Derivatization agent
- A.2 Stability of stock solutions (A, B, C) and spiking solutions
- A.3 Internal standards
- A.4 Volume reduction of extracts

Annex B (informative) Additional clean-up procedures

- B.1 Clean-up with silica/silver nitrate
 - B.1.1 Supplementary reagents
 - B.1.2 Silica/silver nitrate adsorbent for the clean-up column
 - B.1.3 Silica/silver nitrate clean-up
- B.2 Clean-up with aluminium oxide/silver nitrate
 - B.2.1 Supplementary reagents
 - B.2.2 Aluminium oxide/silver nitrate adsorbent for the clean-up column
 - B.2.3 Aluminium oxide/silver nitrate clean-up
- B.3 Pyrogenic copper
 - B.3.1 Supplementary reagents
 - B.3.2 Pyrogenic copper for clean-up column
 - B.3.3 Pyrogenic copper clean-up column

Annex C (informative) Information about typical instrumental conditions
1 The instruments mentioned in this annex are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

- C.1 Example of common gas chromatographic conditions
- C.2 Examples of common detection conditions
 - C.2.1 Atomic absorption spectrometric detection
 - C.2.2.1 Continuous flame
 - C.2.2.2 Pulsed flame
 - C.2.2.3 Pulsed flame photometric detection
 - C.2.2 Flame photometric detection
 - C.2.3 Atomic emission spectrometric detection
 - C.2.4 ICP/MS detection
 - C.2.5 Mass spectrometric detection
 - C.2.6 GC-MSⁿ detection
- C.3 Example of a chromatogram
- C.4 Examples of mass spectra (for MS conditions, see C.2.5)
 - C.4.1 Butyltriethyltin (see Figure C.2)
 - C.4.2 Dibutyldiethyltin (see Figure C.3)
 - C.4.3 Tributylethyltin (see Figure C.4)
 - C.4.4 Tetraethyltin (see Figure C.5)
 - C.4.5 Octyltriethyltin (see Figure C.6)
 - C.4.6 Dioctyldiethyltin (see Figure C.7)
 - C.4.7 Triphenylethyltin (see Figure C.8)
 - C.4.8 Tricyclohexylethyltin (see Figure C.9)
 - C.4.9 Tripropylethyltin (see Figure C.10)
 - C.4.10 Tetrapropyltin (see Figure C.11)
 - C.4.11 Heptyltriethyltin (see Figure C.12)
 - C.4.12 Diheptyldiethyltin (see Figure C.13)
 - C.4.13 Tetrapentyltin (see Figure C.14)

Annex D (informative) Information about GC-MS identification

- D.1 Characteristic masses for mass spectrometric detection

Annex E (informative) Performance data

- E.1 Design of the study and description of the sample materials
- E.2 Remarks on validation results and improvement of performance