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**Soil quality — Determination
of organochlorine pesticides by
gas chromatography with mass
selective detection (GC-MS) and
gas chromatography with electron-
capture detection (GC-ECD)**

iTeh STANDARD PREVIEW

*Qualité du sol — Détermination des pesticides organochlorés par
chromatographie en phase gazeuse avec détection sélective de masse
(CG-SM) et chromatographie en phase gazeuse avec détection par
capture d'électrons (CG-DCE)*

ISO/FDIS 23646

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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.

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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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization*. [ISO/FDIS 23646](https://standards.iteh.ai/catalog/standards/sist/44e3e472-fa34-4cbb-8790-2af1132301d6-2116)

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Introduction

Organochlorine pesticides (OCPs) are organic synthetic substances which are globally used. The vast majority of OCPs have been released directly in the environment as agricultural insecticides but they have been also used as by-products for different applications, e.g. as a wood preserver. OCPs are persistent, bioaccumulating and prone to long-range atmospheric transport and deposition. They are ubiquitous in the environment (water, soil, sediment and waste) and their presence is regularly monitored and controlled.

This document describes the determination of OCPs in soil and sediments. At present, determination of OCPs is carried out in these matrices in most of the routine laboratories following the preceding steps for sampling, pretreatment, extraction and clean-up by measurement of a specific OCP by means of gas chromatography in combination with mass spectrometric detection (GC-MS) or gas chromatography with electron capture detector (GC-ECD). GC-MS/MS is also applicable (see [Annex C](#) for an example of GC-MS/MS measurement conditions for OCPs). The described analytical steps are also applicable for the determination of polychlorinated biphenyls (PCBs). However, for the determination of PCBs, a specific European Standard, EN 17322, is available. Both standards are very similar; differences exist especially in a broader variety of clean-up steps for PCBs.

Considering the different matrices and possible interfering compounds, this document does not contain one single possible way of working. Several choices are possible, in particular relating to clean-up. Detection with both mass spectrometry and electron capture is possible. Three different extraction procedures and four clean-up procedures are described. The use of internal and injection standards is described in order to have an internal check on the choice of the extraction and clean-up procedure.

This document is applicable and validated for several types of matrices as indicated in [Table 1](#) (see also [Annex A](#) for the results of the validation).

Table 1 — Matrices for which this document is applicable and validated

Matrix	Materials used for validation
Soil	Sandy soil, contaminated with OCPs Soil from the vicinity of Berlin
Humic rich soil	Humic rich soil Mix of soil from the vicinity of Berlin, Germany and PCB-free German reference soil
Sediment	Validation results from ISO 10382 (WC 102 and WC 106)

Soil quality — Determination of organochlorine pesticides by gas chromatography with mass selective detection (GC-MS) and gas chromatography with electron-capture detection (GC-ECD)

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document are carried out by suitably trained staff.

1 Scope

This document specifies a method for quantitative determination of organochlorine pesticides (OCPs) and semi-volatile chlorobenzenes in soil and sediment, using GC-MS and GC-ECD (see [Table 2](#)).

Table 2 — Target analytes of this document

Target analyte	CAS-RN	Formula
Aldrin	309-00-2	C ₁₂ H ₈ Cl ₆
Dieldrin	60-57-1	C ₁₂ H ₈ Cl ₆ O
Endrin	72-20-8	C ₁₂ H ₈ Cl ₆ O
Isodrin	465-73-6	C ₁₂ H ₈ Cl ₆
Telodrin	297-78-9	C ₉ H ₄ Cl ₈ O
Heptachlor	76-44-8	C ₁₀ H ₅ Cl ₇
Heptachloro epoxide (exo-, cis-isomer)	1024-57-3	C ₁₀ H ₅ Cl ₇ O
Heptachloro epoxide (endo-, trans-isomer)	28044-83-9	C ₁₀ H ₅ Cl ₇ O
α-Endosulfan	959-98-8	C ₉ H ₆ Cl ₆ O ₃ S
β-Endosulfan	33213-65-9	C ₉ H ₆ Cl ₆ O ₃ S
Endosulfan sulfate	1031-07-8	C ₉ H ₆ Cl ₆ O ₃ S
p,p'-DDE (1,1-bis-(4-chlorophenyl)-2,2-dichloroethen)	72-55-9	C ₁₄ H ₈ Cl ₄
o,p'-DDD (1-(2-Chlorophenyl)-1-(4-chlorophenyl)-2,2-dichloroethan)	53-19-0	C ₁₄ H ₁₀ Cl ₄
o,p'-DDT (1,1,1-Trichloro-2-(2-chlorophenyl)-2-(4-chlorophenyl)ethan)	789-02-6	C ₁₄ H ₉ Cl ₄
p,p'-DDD (1,1-Dichloro-2,2-bis(4-chlorophenyl)ethan)	72-54-8	C ₁₄ H ₁₀ Cl ₄
o,p'-DDE (2-(2-Chlorophenyl)-2-(4-chlorophenyl)-1,1-dichloroethen)	3424-82-6	C ₁₄ H ₈ Cl ₄
p,p'-DDT (1,1,1-Trichlor-2,2-bis-(4-chlorophenyl)ethan)	50-29-3	C ₁₄ H ₉ Cl ₄
Methoxychlor	72-43-5	C ₁₆ H ₁₅ Cl ₃ O ₂
HCB Hexachlorobenzene	118-74-1	C ₆ Cl ₆
α-HCH (α-Hexachlorocyclohexane)	319-84-6	C ₆ H ₆ Cl ₆
β-HCH (β-Hexachlorocyclohexane)	319-85-7	C ₆ H ₆ Cl ₆
γ-HCH (γ-Hexachlorocyclohexane)	58-89-9	C ₆ H ₆ Cl ₆
δ-HCH (δ-Hexachlorocyclohexane)	319-86-8	C ₆ H ₆ Cl ₆
Hexachloro-1,3-butadiene	87-68-3	C ₄ Cl ₆
α-Chlordane	5103-71-9	C ₁₀ H ₆ Cl ₈
γ-Chlordane	5103-74-2	C ₁₀ H ₆ Cl ₈
1,2,4-Trichlorobenzene	120-82-1	C ₆ H ₃ Cl ₃
1,2,3-Trichlorobenzene	87-61-6	C ₆ H ₃ Cl ₃
1,3,5-Trichlorobenzene	108-70-3	C ₆ H ₃ Cl ₃

Table 2 (continued)

Target analyte	CAS-RN	Formula
1,2,3,4-Tetrachlorobenzene	634-66-2	C ₆ H ₂ Cl ₄
1,2,3,5-Tetrachlorobenzene	634-90-2	C ₆ H ₂ Cl ₄
1,2,4,5-Tetrachlorobenzene	95-94-3	C ₆ H ₂ Cl ₄
Pentachlorobenzene	608-93-5	C ₆ HCl ₅

The limit of detection and the limit of application depends on the determinants, the sample intake, the equipment used, the quality of chemicals used for the extraction of the sample and the clean-up of the extract.

Under the conditions specified in this document, lower limits of application from 1 µg/kg (expressed as dry matter) for soils to 10 µg/kg (expressed as dry matter) for sediments can be achieved. The necessity to achieve these lower limits of application depends on the analyses order and the current limit values.

Soils and sediments can differ in properties as well as in the expected contamination levels of OCPs and the presence of interfering substances. These differences make it impossible to describe one general procedure. Based on the properties of the samples, this document contains decision tables regarding drying-, extraction- and clean-up procedures. This method is performance based. The method can be modified if all performance criteria given in this method are met.

The method can be applied to the analysis of other chlorinated compounds not specified in the scope in cases where suitability has been proven by proper in-house validation experiments.

NOTE The validation data are shown in Annex A. This document is validated only for α-HCH, β-HCH, γ-HCH, δ-HCH, o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD, o,p'-DDT and p,p'-DDT. For sediments, data are displayed measured using an ECD detection. The comparability of ECD and MS data in terms of the approach of this document was demonstrated on additional matrices.

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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 5667-15, *Water quality — Sampling — Part 15: Guidance on the preservation and handling of sludge and sediment samples*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

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 18512, *Soil quality — Guidance on long and short term storage of soil samples*

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

EN 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

3 Terms and definitions

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

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

calibration standard

solution of organochlorine pesticides (OCPs) prepared from a secondary standard and/or stock solutions of native OCPs and used to calibrate the response of the instrument with respect to analyte concentration

3.2

internal standard

labelled organochlorine pesticide (OCP) or other OCP that is unlikely to be present in the sample, added to the sample prior to extraction and used for quantification of OCP content

3.3

extraction standard

chemical substance which is only used for checking the extraction efficiency and not used for quantification purposes

3.4

injection standard

labelled organochlorine pesticide (OCP) or other OCP that is unlikely to be present in the sample, added to the extract before injection into the gas chromatograph, and used to monitor variability of instrument response and the recovery of the *internal standards* (3.2)

3.5

performance standard

one calibration solution used for the determination of *performance criteria* (3.6), which contains the same amount of internal, extraction and injection standards (3.4) used in the samples

3.6

performance criterion

value for the recovery of standards which describes the capacity of the analytical method or parts of the analytical method

4 Principle

Due to the multi-matrix character of this document, different procedures for different steps (modules) are allowed. Which modules should be used depends on the sample. A recommendation is given in this document. Performance criteria are described and it is the responsibility of the laboratories applying this document to show that these criteria are met. Use of spiking standards (internal standards) allows an overall check on the efficiency of a specific combination of modules for a specific sample. But it does not necessarily give the information regarding the extensive extraction efficiency of the native OCPs bonded to the matrix.

After pre-treatment, the test sample is extracted with a suitable solvent or solvent-mixture.

The extract is concentrated by evaporation. If necessary, interfering compounds are removed by a clean-up method suitable for the specific matrix, before this concentration step.

The extract is analysed by gas chromatography. The compounds are separated using a capillary column with a stationary phase of low polarity. Detection takes place with mass spectrometry (MS) or with an electron capture detector (ECD). GC-MS/MS is also applicable if the described performance criteria (see 10.7.5) and performance characteristics (see Clause 11) are met.

OCPs are identified and quantified by comparison of relative retention times and relative peak heights (or peak areas) with respect to internal standards added. The efficiency of the procedure depends on the composition of the matrix that is investigated.

5 Interferences

5.1 Interference with sampling and extraction

Use sampling containers of materials (preferably of steel, aluminium or glass) that do not affect the sample during the contact time. Avoid plastics and organic materials during sampling, sample storage or extraction. Keep the samples away from direct sunlight and prolonged exposure to light.

During storage of the samples, losses of OCPs can occur due to adsorption on the walls of the containers. The extent of the losses depends on the storage time.

5.2 Interference with GC

Substances that coelute with the target OCPs can interfere the determination. These interferences can lead to incompletely resolved signals and can, depending on their magnitude, affect accuracy and precision of the analytical results. Peak overlap does not allow an interpretation of the result. Asymmetric peaks and peaks being broader than the corresponding peaks of the reference substance suggest interferences.

Depending on the utilized stationary phase, some isomers (e.g. 1,2,4,5- and 1,2,3,5-Tetrachlorobenzene) can coelute or be not fully separated. In this case, a positive result should be reported as the sum of both isomers or a different stationary phase should be applied to ensure a separation, which allows to give results for both single isomers.

6 Safety remarks

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Some OCPs are toxic and shall be handled with extreme care. Avoid contact with solid materials, solvent extracts and solutions of standard OCPs. It is strongly advised that standard solutions are prepared centrally in suitably equipped laboratories or are purchased from suppliers specialized in their preparation.

Solvent solutions and samples containing OCPs shall be disposed of in a manner approved for disposal of toxic wastes.

For the handling of hexane, precautions shall be taken because of its neurotoxic properties.

Precautions shall be taken with respect to all hazards associated with this method.

7 Reagents

7.1 General

All reagents shall be of recognized analytical grade. The purity of the reagents used shall be checked by running a blank determination as described in [10.1](#). The blank shall be less than 50 % of the lowest reporting limit.

7.2 Reagents for extraction

7.2.1 Acetone (2-propanone), (CH₃)₂CO.

7.2.2 n-heptane, C₇H₁₆.

7.2.3 Petroleum ether, boiling range 40 °C to 60 °C.

7.2.4 Hexane-like solvents, boiling range between 30 °C and 89 °C.

7.2.5 Anhydrous sodium sulfate, Na₂SO₄. The anhydrous sodium sulfate shall be kept carefully sealed.

7.2.6 Distilled water or water of equivalent quality, H₂O.

7.2.7 Sodium chloride, NaCl, anhydrous.

7.2.8 Keeper substance. Non-polar organic solvent with high boiling point, i.e. octane, nonane.

7.3 Reagents for clean-up

7.3.1 Clean-up A using aluminium oxide

7.3.1.1 Aluminium oxide, Al₂O₃.

Basic or neutral, specific surface 200 m²/g, activity Super I in accordance with Reference [6].

7.3.1.2 Deactivated aluminium oxide.

Deactivated with approximately 10 % water.

Add approximately 10 g of water (7.2.6) to 90 g of aluminium oxide (7.3.1.1). Shake until all lumps have disappeared. Allow the aluminium oxide to condition before use for about 16 h, sealed from the air. Use it for a maximum of two weeks.

NOTE The activity depends on the water content. It can be necessary to adjust the water content.

7.3.2 Clean-up B using silica gel 60 for column chromatography

7.3.2.1 Silica gel 60, particle size 63 µm to 200 µm.

7.3.2.2 Silica gel 60, water content: mass fraction w(H₂O) = 10 %.

Silica gel 60 (7.3.2.1), heated for at least 3 h at 450 °C, cooled down and stored in a desiccator containing magnesium perchlorate or a suitable drying agent. Before use, heat at least for 5 h at 130 °C in a drying oven. Then allow cooling in a desiccator and add 10 % water (mass fraction) in a flask. Shake for 5 min intensively by hand until all lumps have disappeared and then for 2 h in a shaking device. Store the deactivated silica gel in the absence of air. Use it for a maximum of two weeks.

7.3.3 Clean-up C using gel permeation chromatography (GPC)

7.3.3.1 Bio-Beads®¹⁾ S-X3.

7.3.3.2 Ethyl acetate, C₄H₈O₂.

1) Bio-Beads® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. Equivalent products can be used if they can be shown to lead to the same results.

7.3.3.3 Cyclohexane, C₆H₁₂.

Preparation of GPC, for example:

- put 50 g Bio-Beads®¹⁾ S-X3 (7.3.3.1) into a 500 ml Erlenmeyer flask and add 300 ml elution mixture made up of cyclohexane (7.3.3.3) and ethyl acetate (7.3.3.2) 1:1 (volume) in order to allow the beads to swell;
- after swirling for a short time until no lumps are left, maintain the flask closed for 24 h;
- drain the slurry into the chromatography tube for GPC;
- after approximately three days, push in the plungers of the column so that a filling level of approximately 35 cm is obtained;
- to further compress the gel, pump approximately 2 l of elution mixture through the column at a flow rate of 5 ml min⁻¹ and push in the plungers to obtain a filling level of approximately 33 cm.

7.3.4 Clean-up D using Florisil®²⁾

7.3.4.1 Florisil®²⁾, baked for 2 h at 600 °C. Particle size 150 µm to 750 µm.

7.3.4.2 Iso-octane, C₈H₁₈.

7.3.4.3 Toluene, C₇H₈.

7.3.4.4 Iso-octane/Toluene 95/5 (volume fraction).

7.3.4.5 Diethylether, C₄H₁₀O.

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7.4 Reagents for gas chromatographic analysis

Operating gases for gas chromatography ECD or MS, of high purity and in accordance with the manufacturer's specifications.

7.5 Standards

7.5.1 General

Solvents for preparing standard solutions shall be free of OCPs. Hexane, cyclo-hexane, iso-hexane or other hexane-like solvents can be used. Verify the stability of the standards regularly.

7.5.2 Calibration standards

The calibration standards should contain the target compounds selected from [Table 2](#).

2) Florisil® is a trade name for a prepared diatomaceous substance, mainly consisting of anhydrous magnesium silicate. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. Equivalent products can be used if they can be shown to lead to the same results.