

საქართველოს სტანდარტი

სსკ: 13.080.10

ნიადაგის ხარისხი — ქლორორგანული პესტიციდების
განსაზღვრა გაზის ქრომატოგრაფიით მასის სელექციური
გამოვლენა (GC-MS) და გაზის ქრომატოგრაფიით ელექტრონის
დაჭერის გამოვლენა (GC-ECD)

სსტ ისო 23646:2022/2025

საინფორმაციო მონაცემები

1 მიღებულია და დაშვებულია გამოქმედდეს: სსიპ-საქართველოს სტანდარტებისა და მეტროლოგიის ეროვნული სააგენტოს გენერალური დირექტორის 13/02/2025 წლის № 9 განკარგულებით

2 მიღებულია „თავფურცლის“ თარგმნის მეთოდით: სტანდარტიზაციის საერთაშორისო ორგანიზაციის (ისო) სტანდარტი ისო 23646:2022 „ნიადაგის ხარისხი — ქლორორგანული პესტიციდების განსაზღვრა გაზის ქრომატოგრაფიით მასის სელექციური გამოვლენა (GC-MS) და გაზის ქრომატოგრაფიით ელექტრონის დაჭერის გამოვლენა (GC-ECD)“

3 პირველად

4 რეგისტრირებულია: სსიპ-საქართველოს სტანდარტებისა და მეტროლოგიის ეროვნული სააგენტოს რეესტრში: 13/02/2025 წლის №268-1.3-041703

წინამდებარე სტანდარტის ნებისმიერი ფორმით გავრცელება სააგენტოს ნებართვის გარეშე აკრძალულია

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

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





COPYRIGHT PROTECTED DOCUMENT

© ISO 2022

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	2
3 Terms and definitions	2
4 Principle	3
5 Interferences	4
5.1 Interference with sampling and extraction.....	4
5.2 Interference with GC.....	4
6 Safety remarks	4
7 Reagents	4
7.1 General.....	4
7.2 Reagents for extraction.....	4
7.3 Reagents for clean-up.....	5
7.3.1 Clean-up A using aluminium oxide.....	5
7.3.2 Clean-up B using silica gel 60 for column chromatography.....	5
7.3.3 Clean-up C using gel permeation chromatography (GPC).....	5
7.3.4 Clean-up D using Florisil®.....	6
7.4 Reagents for gas chromatographic analysis.....	6
7.5 Standards.....	6
7.5.1 General.....	6
7.5.2 Calibration standards.....	6
7.5.3 Internal, extraction and injection standards.....	7
7.6 Preparation of standard solutions.....	9
7.6.1 Preparation of calibration standard solutions of OCPs.....	9
7.6.2 Preparation of internal standard solution.....	9
7.6.3 Preparation of injection standard solution.....	10
7.6.4 Preparation of solution for liner checking.....	10
8 Apparatus	10
8.1 Extraction and clean-up procedure.....	10
8.2 Gas chromatograph.....	11
8.2.1 General.....	11
8.2.2 Capillary columns.....	11
9 Sample storage and pre-treatment	11
9.1 Sample storage.....	11
9.2 Sample pretreatment.....	11
10 Procedure	12
10.1 Blank test.....	12
10.2 Extraction.....	12
10.2.1 General.....	12
10.2.2 Extraction procedure 1 — Agitation or sonication.....	13
10.2.3 Extraction procedure 2 — Pressurized liquid extraction (PLE).....	13
10.2.4 Extraction procedure 3 — Soxhlet.....	14
10.3 Concentration.....	14
10.4 Clean-up of the extract.....	14
10.4.1 General.....	14
10.4.2 Clean-up A — Aluminium oxide.....	15
10.4.3 Clean-up B — Silica gel.....	15
10.4.4 Clean-up C — Gel permeation chromatography.....	15
10.4.5 Clean-up D — Florisil® ²⁾	16

10.5	Addition of the injection standard.....	16
10.6	Gas chromatographic analysis (GC).....	16
	10.6.1 General.....	16
	10.6.2 Setting the gas chromatograph.....	16
10.7	Mass spectrometry (MS).....	16
	10.7.1 Mass spectrometric conditions.....	16
	10.7.2 Calibration of the method using an internal standard.....	17
	10.7.3 Measurement.....	19
	10.7.4 Identification.....	19
	10.7.5 Check on method performance.....	19
	10.7.6 Calculation.....	20
10.8	Electron capture detection (ECD).....	21
	10.8.1 General.....	21
	10.8.2 ECD conditions.....	21
	10.8.3 Calibration of the method using an internal standard.....	21
	10.8.4 Measurement.....	21
	10.8.5 Identification.....	21
	10.8.6 Check on ECD method performance.....	21
	10.8.7 Calculation.....	22
11	Performance characteristics.....	22
12	Precision.....	22
13	Test reports.....	23
	Annex A (informative) Repeatability and reproducibility data.....	24
	Annex B (informative) Calibration strategy.....	27
	Annex C (informative) Example of GC-MS/MS measurement conditions for OCPs.....	28
	Bibliography.....	30

საინფორმაციო ნაწილი. სრული ტექსტის სანახავად შეიძინეთ სტანდარტი.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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*.

This first edition cancels and replaces ISO 10382:2002, which has been technically revised.

The main changes are as follows:

- polychlorinated biphenyls have been deleted from the Scope;
- modern extraction techniques and commonly used methods with optimized extraction time, proven clean-up methods and state of the art quantification methods have been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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)