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Soil quality — Simultaneous determination of multi-class pesticide residues in soil using gas chromatography-tandem mass spectrometry (GC-MS/MS) and liquid chromatography-tandem mass spectrometry (LC-MS/MS) analysis

Qualité du sol — Détermination simultanée de résidus de pesticides multiclasses dans le sol par chromatographie en phase gazeuse couplée à la spectrométrie de masse en tandem (CG-SM/SM) et par chromatographie en phase liquide couplée à la spectrométrie de masse en tandem (CL-SM/SM)

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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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This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization*.

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.

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Introduction

Pesticides are primarily used to protect plants and enhance crop yields. The global average consumption of pesticides sprayed on cultivated land is approximately 2,69 kg/10 000 m^2 This value reflects an increase compared to the past few decades. This upward trend in pesticide consumption contributes to continuous exposure to pesticides in environmental matrices such as soil, water and air. The presence of pesticide residues in soils can degrade soil quality and have adverse effects on soil biodiversity, impacting non-target biota, as well as other ecosystem through the off-site transport of contaminated soil particles and soil pore water into air or water. Pesticide residues in soil can potentially move downward to groundwater through leaching process or run off to surface water resulting in water contamination.

Information at the national level regarding the presence of pesticides residue in soil has been limited due to low number of soil samples and monitoring projects and it primarily focuses on the highly toxic pesticides used in the past. However, some recent studies have reported the detection of various types of current fungicides, herbicides and insecticides in arable soil, playground, urban park areas, ground water and surface water.

With respect to toxicologically characterized pesticide residues, environmental agencies throughout the world have established specific values that are used in risk assessment. Developing a standard analytical method targeting currently used pesticides in soil is crucial for conducting risk assessment at pesticide contaminated sites. A method available in analysing multi-class pesticide residues in soil simultaneously would be very effective due to the variety of pesticide used. In this context, the QuEChERS (quick easy cheap effective rugged safe) method can be an alternative method to analyse a number of pesticide residues simultaneously in soil with relatively simple pretreatment applicable to both GC-MS/MS and LC-MS/MS.

The QuEChERS methods has already been incorporated into EN 15662.

This document can support the creation of an analytical framework for various pesticide residues in soil. It can be the starting point for future work including additional pesticide residues that are not target analytes in this document.

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Soil quality — Simultaneous determination of multi-class pesticide residues in soil using gas chromatography-tandem mass spectrometry (GC-MS/MS) and liquid chromatography-tandem mass spectrometry (LC-MS/MS) analysis

1 Scope

This document provides a method for the simultaneous analysis of multi-class pesticide residues in soil using gas chromatography-tandem mass spectrometry (GC-MS/MS) and liquid chromatography-tandem mass spectrometry (LC-MS/MS) analysis following extraction with acetonitrile and buffering salts mixture, and cleanup via dispersive solid-phase extraction (d-SPE). This document is applicable to the 30 target analytes listed in Table 1. This document can be applicable to other pesticide residues if their validity is confirmed.

Table 1 — Target analytes of this document

Type of pesticide	Pesticide residues	Instrumental analysis	CAS-RN[a]
Fungicide	Azoxystrobin	LC-MS/MS	131860-33-8
	Carbendazim	LC-MS/MS	10605-21-7
	Difenoconazole (2 isomer)	GC-MS/MS	119446-68-3
	Fludioxonil	LC-MS/MS	131341-86-1
	Fluopicolide	LC-MS/MS	239110-15-7
	Fluopyram	GC-MS/MS	658066-35-4
	Flutolanil	LC-MS/MS	66332-96-5
	Isopyrazam	GC-MS/MS	881685-58-1
	Metconazole	LC-MS/MS	125116-23-6
	Metrafenone	LC-MS/MS	220899-03-6
	Paclobutrazol	GC-MS/MS	76738-62-0
	Propiconazole (2 isomer)	GC-MS/MS	60207-90-1
	Pyraclostrobin	LC-MS/MS	175013-18-0
	Tebuconazole	GC-MS/MS	107534-96-3
	Tetraconazole	GC-MS/MS	67915-31-5
	Triadimefon	GC-MS/MS	43121-43-3
Herbicide	Alachlor	GC-MS/MS	15972-60-8
	Linuron	LC-MS/MS	330-55-2
	Metolachlor	GC-MS/MS	51218-45-2
	Pendimethalin	GC-MS/MS	40487-42-1
	Simazine	LC-MS/MS	122-34-9
	Trifluralin	GC-MS/MS	1582-09-8

NOTE Under the specified conditions in this document, a limit of quantitation (LOQ) less than 0,1 mg/kg (expressed as dry matter basis) can be achieved for the target analytes (refer to Annex C).

[a] Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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Table 1 *(continued)*

Type of pesticide	Pesticide residues	Instrumental analysis	CAS-RN[a]
Insecticide	Chlorantraniliprole	LC-MS/MS	500008-45-7
	Chlorpyrifos	GC-MS/MS	2921-88-2
	Fenpropathrin	GC-MS/MS	39515-41-8
	Fenpyroximate	LC-MS/MS	111812-58-9
	Flubendiamide	LC-MS/MS	272451-65-7
	Imidacloprid	LC-MS/MS	138261-41-3
	Methoxyfenozide	LC-MS/MS	161050-58-4
	Pyridalyl	GC-MS/MS	179101-81-6

NOTE Under the specified conditions in this document, a limit of quantitation (LOQ) less than 0,1 mg/kg (expressed as dry matter basis) can be achieved for the target analytes (refer to Annex C).

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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 11464, *Soil quality — Pretreatment of samples for physico-chemical analysis*

ISO 11465, *Sludge and solid environmental matrices — Determination of dry residue or water content and calculation of the dry matter fraction on a mass basis*

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

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
matrix matched calibration
methodology for calibration that involves matching the components of the calibration standard solution with those of the test solution

Note 1 to entry: This aims to enhance quantification accuracy by minimizing potential interferences in chromatographic responses caused by the matrix effect.

3.2
matrix matched solution
solution reflecting the characteristics of the matrix

Note 1 to entry: It is used as a dilution solvent for preparing *matrix matched calibration* (3.1) standard solution.

4 Principle

Through a single step of extraction with simple clean-up and matrix-matched calibration, target pesticide residues in a soil sample are analysed using two complementary techniques: liquid chromatography-tandem

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