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Soil quality — Determination of selected explosives and related compounds —

Part 3: Method using liquid chromatography-tandem mass spectrometry (LC-MS/MS)

Qualité du sol — Dosage d'une sélection d'explosifs et de composés apparentés —

Partie 3: Méthode utilisant la chromatographie en phase liquide couplée à la spectrométrie de masse en tandem (CL-SM/SM)

Reference number
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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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For an explanation of 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 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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 444, *Environmental characterization of solid matrices,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

A list of all parts in the ISO 11916 series can be found on the ISO website.

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

Currently two ISO standards exist for the analysis of explosives and related compounds in soil: ISO 11916-1 (HPLC with UV detection method), ISO 11916-2 (GC-ECD or MS). According to the results of inter-laboratory trial with ISO 11916-1, it showed some problematic aspects to analyze PETN, 1,3,5-TNB and tetryl. In case of ISO 11916-2, it also gave poor inter-laboratory trial results for 1,3,5-TNB. Therefore, it is necessary to develop new method effectively applicable to the determination of PETN, 1,3,5-TNB and tetryl. In addition to this, lower risk-based PRGs (Preliminary Remediation Goal), new regulatory concerns, and change of land use have created the atmosphere to apply more sensitive and selective instruments to determine explosive and related compounds. From the view of these aspects, liquid chromatography–tandem mass spectrometry (LC-MS/MS) is one of alternative methods for these purposes. LC-MS/MS method provides 10-20 times or more lower detection limit than that of HPLC/UV method. In this document, LC-MS/MS method is intended for the trace analysis of explosives and related compounds and applicable to 12 compounds (1,3-DNB, 1,3,5-TNB, 2,4-DNT, 2,6-DNT, 2,4,6-TNT, 4-A- 2,6-DNT, 2-A-4,6-DNT, Tetryl, Hexyl, RDX, HMX, PETN) listed in ISO 11916-1 (soil, HPLC with UV detection method) except for nitrobenzene, 2-nitrotoluene, 3-nitrotoluene and 4-nitrotoluene (see Annex E). In case of nitrobenzene and nitrotoluenes, they have the low sensitivity in LC-MS/MS measurement than using HPLC with UV detection method. In particular LC-MS/MS measurement is effective for the analysis of PETN, 1,3,5-TNB and tetryl when comparing with the method using HPLC with UV detection method. Also LC-MS/MS method is getting more familiar in ISO standard development (e.g. ISO 22104 Water quality-Microcystins, ISO/NP 21677 Water quality-HBCD, ISO 21675 Water quality-PFAS).

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Soil quality — Determination of selected explosives and related compounds —

Part 3: Method using liquid chromatography-tandem mass spectrometry (LC-MS/MS)

1 Scope

This document specifies the measurement of explosives and related nitrocompounds (as given in Table 1) in soil and soil materials. This document is intended for the trace analysis of explosives and related compounds by liquid chromatography–tandem mass spectrometry (LC-MS/MS). Generally, LC-MS/MS measurement shows the lower LOQ (limit of quantification) for each compound in Table 1 than using high-performance liquid chromatography (HPLC) with UV-detection (see Annex B and Annex C).

Under the conditions specified in this document, concentrations as low as 0,005 mg/kg to 0,014 mg/kg-dry matter can be determined, depending on the substance. Similar compounds, in particular various nitroaromatics, by-products and degradation products of explosive compounds can be analysed using this method provided that the applicability is checked on a case-by-case basis.

Table 1 — Explosive and related nitrocompounds for analysis

Compound	Abbreviation	CAS-RN®[1]
1,3-Dinitrobenzene	1,3-DNB	99-65-0
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4
2,4-Dinitrotoluene	2,4-DNT	121-14-2
2,6-Dinitrotoluene	2,6-DNT	606-20-2
2,4,6-Trinitrotoluene	2,4,6-TNT	118-96-7
4-Amino-2,6-dinitrotoluene	4-A-2,6-DNT	19406-51-6
2-Amino-4,6-dinitrotoluene	2-A-4,6-DNT	35572-78-2
N-Methyl-*N*-2,4,6-tetranitroaniline	Tetryl	479-45-8
2,4,6-Trinitro-*N*-(2,4,6-trinitrophenyl)aniline	Hexyl	131-73-7
1,3,5-Trinitrohexahydro-1,3,5-triazine	RDX	121-82-4
1,3,5,7-Tetranitro-octahydro-1,3,5,7-tetrazocine	HMX	2691-41-0
Pentaerythrityl tetranitrate	PETN	78-11-5

[1] CAS Registry Number® (CAS RN®) is a trademark of CAS corporation. 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.

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 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

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3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Principle

Explosive materials in soils are extracted with acetonitrile by using one of the following techniques:

— ultrasonic bath with ultrasonic waves as medium (USE);

— horizontal mechanical shaker at room temperature (MSE).

There are two further extraction procedures such as pressurized liquid extraction (PLE) and soxhlet apparatus that works isothermically at boiling temperature (SOX). However, they might not be suitable for PETN, tetryl and 1,3,5-TNB.

The extract containing the analytes is either injected directly, or if necessary diluted prior to injection, into a reversed-phase high-performance liquid chromatograph–tandem mass spectrometer (LC-MS/MS).

WARNING — Take care when transporting, storing or treating explosive materials. High temperature, high pressure and static electricity shall be prevented when storing explosive materials. Small amounts of explosive materials should be kept moist in a cool, dark place. Soil samples containing explosives with a mass fraction of less than 1 % do not have a risk of explosion.

5 Interferences

Solvents, reagents, glassware, and other hardware used for sample processing can yield artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials shall therefore be demonstrated to be free of contaminants and interferences through the analysis of method blanks.

Samples containing 2,4,6-trinitrobenzoic acid should not be extracted with acetonitrile as it can result in the overestimation of 1,3,5-TNB due to decarboxylation. To avoid this interference, methanol extraction can be an alternative method for 1,3,5-TNB.

When comparing with acetonitrile, methanol has showed serious problem in the recovery test for HMX, RDX and tetryl with LC-MS/MS detection (see Annex D). Also, it should be avoided to extract tetryl at temperatures above room temperature.

6 Reagents

6.1 General

All reagents shall be blank-free and of recognized analytical grade.

6.2 Chemicals

6.2.1 Water, with an electrical conductivity of ≤ 0,01 mS/m (25 °C).

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