BS ISO 11916-1:2013

BSI Standards Publication

Soil quality — Determination of selected explosives and related compounds

Part 1: Method using high-performance liquid chromatography (HPLC) with ultraviolet detection

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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INTERNATIONAL STANDARD

ISO 11916-1

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Soil quality — Determination of selected explosives and related compounds —

Part 1: Method using high-performance liquid chromatography (HPLC) with ultraviolet detection

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

Partie 1: Méthode utilisant la chromatographie liquide à haute performance (CLHP) avec détection ultraviolet

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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. www.iso.org/directives

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The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

ISO 11916 consists of the following parts, under the general title *Soil quality — Determination of selected explosives and related compounds*:

— *Part 1: Method using high-performance liquid chromatography (HPLC) with ultraviolet detection*

— *Part 2: Method using gas chromatography (GC) with electron capture detection (ECD) or mass spectrometric detection (MS)*

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INTERNATIONAL STANDARD ISO 11916-1:2013(E)

Soil quality — Determination of selected explosives and related compounds —

Part 1: Method using high-performance liquid chromatography (HPLC) with ultraviolet detection

1 Scope

This part of ISO 11916 specifies the measurement of explosive and related nitrocompounds (as given in Table 1) in soils and soil materials. This part of ISO 11916 is intended for the trace analysis of explosives and related compounds by high-performance liquid chromatography (HPLC) using an ultraviolet (UV) detector.

Under the conditions specified in this part of ISO 11916, concentrations as low as 0,1 mg/kg to 1 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 may be analysed using this method. However, the applicability should be checked on a case-by-case basis.

Table 1 — Explosive and related nitrocompounds for analysis

Compound	Abbreviation	CAS-RN[a]
Nitrobenzene	NB	98-95-3
1,3-Dinitrobenzene	1,3-DNB	99-65-0
1,3,5-Trinitrobenzene[b]	1,3,5-TNB	99-35-4
2-Nitrotoluene	2-NT	88-72-2
3-Nitrotoluene	3-NT	99-08-1
4-Nitrotoluene	4-NT	99-99-0
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[b]	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-Tetranitrooctahydro-1,3,5,7-tetrazocine	HMX	2691-41-0
Pentaerythrityltetranitrate[b]	PETN	78-11-5

[a] CAS-RN: Chemical Abstract Service-Registry Number

[b] PETN, 1,3,5-TNB and tetryl gave poor interlaboratory trial results and their analysis could be problematic

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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

ISO 22478, *Water quality — Determination of certain explosives and related compounds — Method using high-performance liquid chromatography (HPLC) with UV detection*

3 Principle

Explosive materials in soils are extracted with acetonitrile or methanol, using one of the following techniques:

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

— horizontal mechanical shaker at room temperature (MSE);

— Soxhlet apparatus that works isothermically at boiling temperature (SOX);

— pressurized liquid extraction (PLE).

The extract containing the analytes is either injected directly, or if necessary diluted prior to injection, into a reversed-phase high-performance liquid chromatography system (HPLC) and the analytes are detected by means of a diode array detector (DAD).

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.

4 Interferences

Solvents, reagents, glassware, and other hardware used for sample processing may yield artefacts 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.

Tetryl may decompose in methanol or water at (or above) room temperature. Degradation products of tetryl appear as a shoulder of 2,4,6-TNT when a C_{18} column is used. In this case, evaluation of 2,4,6-TNT should be based on peak height rather than peak area.

Samples containing 2,4,6-trinitrobenzoic acid should not be extracted with acetonitrile as it may result in the overestimation of 1,3,5-TNB, due to decarboxylation.

5 Reagents

5.1 General

All reagents shall be blank-free and of recognized analytical grade. They shall not contain any measurable quantities of UV-absorbing substances that may interfere with the determination.

5.2 Chemicals

5.2.1 Water.

5.2.2 Acetone, C_3H_6O, for the cleaning of containers and devices.

5.2.3 Acetonitrile, CH_3CN, HPLC grade or equivalent.

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5.2.4 Methanol, CH_3OH, HPLC grade or equivalent.

5.2.5 Diatomaceous earth or sea sand, pelletized and calcinated (for PLE).

5.3 Standard substances and solutions

5.3.1 Standard substances

5.3.1.1 Reference substances

Compounds listed in Table 1.

5.3.1.2 Method-checking standard

Suitable compound(s) not found in the sample, e.g. 2,5-DNT.

5.3.2 Standard solutions

5.3.2.1 General

All standard solutions used in this method shall be prepared as described below.

NOTE If commercially available certified standard stock solutions (Table 1) are used, calibration solutions are prepared in volumetric flasks by diluting the stock solutions with acetonitrile (5.2.3) or methanol (5.2.4), respectively.

All dilution steps shall not exceed the factor 100.

5.3.2.2 Single-substance stock solutions

For the preparation, weigh 50 mg ± 0,1 mg of the reference substances into 50 ml measuring flasks (scale: mg/ml), fill up to the mark with acetonitrile (5.2.3) or methanol (5.2.4), respectively and let them dissolve completely.

Transfer the stock solutions to amber-glass flasks and seal with PTFE-coated screw caps.

The stock solutions can be kept in the refrigerator at 2 °C to 6 °C in the dark for up to 1 year.

5.3.2.3 Multi-component stock solutions

Prepare multi-component stock solutions of different concentrations from the various single-substance stock solutions (5.3.2.2) by mixing and diluting with acetonitrile (5.2.3) or methanol (5.2.4), respectively.

At concentrations below 1 mg/ml, solutions should be checked after one week as reference substances may decompose.

For calibration standards, a minimum of 5 concentration levels is needed.

6 Apparatus

6.1 General

Usual laboratory apparatus and the following.

6.1.1 Amber glass containers with caps containing polytetrafluoroethene (PTFE) coated lining.

6.1.2 Amber glass vials with caps containing septa with polytetrafluorethene (PTFE) coated lining

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6.1.3 Amber glass conical bottles with ground-in stopper.

6.1.4 Perforated metal plate sieve, complying with ISO 565.

6.1.5 Analytical balance, with a precision of at least 0,1 mg.

6.1.6 Laboratory centrifuge, capable of producing an acceleration of at least 1 000*g*.

6.1.7 pH-meter, to adjust the pH of the mobile phase for HPLC.

6.1.8 Filter and suitable filter discs, 0,45 µm pore size.

Any adsorption of the target analytes shall be avoided. No interfering material shall be eluated. PTFE or polyamide material is recommended.

6.2 Equipment for extraction

6.2.1 Temperature-controlled ultrasonic bath, 35 Hz, effective HF-power of at least 140 W.

Water bath capable of maintaining the temperature at (30 ± 5) °C or at (50 ± 5) °C during ultrasonic extraction.

6.2.2 Horizontal mechanical shaker

The shaker shall maintain a frequency of 100 cycles/min and offer a shaking width of about 10 cm.

6.2.3 Soxhlet apparatus

Extractor, whose extraction chamber and syphon are placed inside the steam chamber and suitably covered, or extractor with additionally heated extraction chamber, complete with boiling vessel, suitable heating mantle and reflux condenser, suitable for the extraction of a 50 g sample of soil with a hot solvent distillate through complete flooding of the extractive.

6.2.4 Pressurized liquid extractor (PLE)

Pressurized liquid extraction device, equipped with extraction cells made of stainless steel or other material capable of withstanding the pressure levels (890 hPa/2 000 psi) necessary for this procedure; vials for collection of extracts, 40 ml or 60 ml, pre-cleaned, open-top screw-cap with polytetrafluoroethylene (PTFE)-lined septum; filter disc, cellulose or glass fibre; cell cap sealing disc.

6.3 High-performance liquid chromatography (HPLC) system with DAD

HPLC system, consisting of a pump which supports a pressure of at least 40 MPa (400 bar), an injection system with a loop capacity of 100 µl and a diode array detector (DAD) with wavelength range of 200 nm to 400 nm (or higher).

Other specifications of the HPLC system should be in accordance with ISO 22478.

7 Procedure

7.1 Sample pretreatment, sample storage and determination of water content

While taking a field-moist sample, remove coarse impurities, e.g. plant residues and stones. Put the sample in an amber glass flask and store immediately in a cool, dark transport container.

Soil samples shall be analysed as soon as possible.

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When sample treatment is proceeded within 1 week after sampling, store the sample in a dark place at (4 ± 2) °C. Samples that are stored for longer periods (i.e. > 1 week) prior to analysis, shall be stored at –20 °C.

Homogenize the sample by sieving through a sieve with an aperture of 2 mm (6.1.4).

For the determination of volatile nitroaromatics (2-NT, 3-NT, 4-NT, NB) a sample withdrawal is to be carried out, in order to minimize evaporative losses.

Samples that were primarily taken for the determination of volatile compounds may also be taken by filling them immediately (on-site) in an extraction vial containing methanol. Then, pretreat samples according to 7.2.2 or 7.2.3. These samples have to be considered and reported as non-homogenized unscreened random samples.

In order to calculate the dry matter based content of explosive compounds, determine the dry matter content of the field-moist soil in accordance with ISO 11465. Be aware of potential evaporation of volatile toxic contaminants.

7.2 Extraction

7.2.1 General

For extraction, the following four methods may be applied:

— extraction using ultrasonic waves (7.2.2);

— extraction using mechanical shaking (7.2.3);

— extraction using Soxhlet apparatus (7.2.4).

— pressurized liquid extraction (7.2.5).

The use of a method-checking standard is recommended. Method-checking standards have to be added prior to extraction. For the selection of suitable method-checking standards, refer to 5.3.1.2.

For the analysis of HMX, hexyl, and PETN, acetonitrile (5.2.3) should be used as a solvent because of their poor solubility in pure methanol.

7.2.2 Extraction using ultrasonic waves

Take approximately 20 g of the field-moist and homogenized sample and weigh it into the extraction vial (6.1.1) with a precision of ± 0,1 g and add the method-checking standard (5.3.1.2), if used, with a concentration range of 1 mg/l to 10 mg/l in the final extract. Add 40 ml ± 0,1 ml of acetonitrile (5.2.3) or methanol (5.2.4), respectively, and seal with a cap containing a PTFE coated lining. Shake the vial briefly by hand, then apply ultrasonic extraction in the bath (6.2.1) for 16 h at (30 ± 5) °C or 4 h at (50 ± 5) °C.

During extraction, the water level in the bath should be at least 1 cm above the level of the solvent inside the extraction flasks.

After applying ultrasonic extraction, allow the soil particles to settle for 30 min. Do not open the vial before it has cooled down to room temperature. If necessary, filter an aliquot of the supernatant using a 0,45 µm PTFE or polyamide filter or centrifuge at 1 000*g* for 20 min.

It is recommended to lightly moisten the filter with solvent prior to filtration.

The total volume of the extract corresponds to the volume of solvent used for extraction plus the water content of the soil sample.

7.2.3 Extraction using mechanical shaking

Take approximately 20 g of the field-moist and homogenized sample and weigh it into the extraction vial (6.1.1) with a precision of ± 0,1 g and add the method-checking standard (5.3.1.2), if used, with a

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