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

Reference number
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Contents

Page

Foreword

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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)*

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.

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.

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.

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.

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

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 place the extraction vial in a horizontal mechanical shaker (6.2.2) and shake for 16 h.

After shaking, allow the soil particles to settle in the vial for 30 min. 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 syringe 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.

7.2.4 Extraction using Soxhlet apparatus

The extraction is carried out isothermically (extraction sample in the thimble always at boiling temperature) in a Soxhlet apparatus (6.2.3). To ensure isothermic working conditions while using a classical Soxhlet, it shall be covered by the steam chamber of the solvent. When using an extractor such as Soxtec®[1)] or Büchi-extractors[2)], the solvent distillate in the thimble shall always be heated to its boiling point.

Take approximately 50 g of the field-moist and homogenized sample, weigh it into an extraction thimble 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. Insert the thimble into the Soxhlet extractor, and add methanol (5.2.4) to the boiling vessel. The liquid level in the receiver (boiling vessel) should not drop below the upper rim of the heating mantle during extraction in order to prevent the formation of deposits on the inner wall of the vessel, because it may cause a loss of certain analytes.

Prior to analysis, check the absorption potential of extraction thimble material.

NOTE Experience has shown that the use of fibre-glass filters decreases the yield of TNB. Cellulose extraction thimbles seem to be most suitable.

The extraction is carried out for at least 4 h. When using a Soxhlet extractor, a cycle time of 6 min to 8 min should be reached. In every cycle the extractive shall be completely immersed in hot solvent distillate.

When the extraction is completed, let the extract cool down to room temperature before removing the reflux condenser.

The volume of the extract shall be determined or brought to a defined volume with the extraction solvent.

7.2.5 Pressurized liquid extraction (PLE)

Take approximately 20 g of the field-moist and homogenized sample, weigh it into a beaker 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 and mix with a suitable amount of diatomaceous earth or sea sand. Transfer the whole content of the beaker into the extraction cell, refill the dead volume with diatomaceous earth or sea sand and close the cell.

Prepare the apparatus according to the manufacturer's instructions.

Fill the solvent container of the device with methanol and place the prepared cell(s) and the vial(s) collecting the extract inside the apparatus. Select the appropriate adjustment of parameters (see Table 2).

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1) Soxtec® is the trade name of a product supplied by Foss. 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) Büchi-extractor is the trade name of a product supplied by BÜCHI Labortechnik AG. 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.

When the extraction is completed, the extract is held at a temperature of 20 °C. Since the extraction cells contain frits, filtration of the extracts is not necessary.

The volume of the extract shall be determined or brought to a defined volume using the extraction solvent.

Table 2 — Example of parameters and requirements for the PLE apparatus

Parameter	Requirement
Solvent	100 % methanol
Dimension of the cell, in ml	33
Preheat, in min	0
Heat, in min	5
Static, in min	15
Flush, in % of cell volume	60
Purge, in s	200
Cycles	1
Pressure, in hPa (psi)	890 (2 000)
Temperature, in °C	100

7.3 Storage of extract

If the acetonitrilic or methanolic extract cannot be analysed immediately, it shall be stored in a refrigerator at (4 ± 2) °C in the dark. In case of precipitation, ensure that the precipitate is re-dissolved before analysis, e.g. through ultrasonication.

8 High-performance liquid chromatographic analysis

8.1 General

The separation of the analytes is performed by means of a reversed-phase high-performance liquid chromatograph with a suitable column and detected using UV detection applying a diode array detector (DAD).

A defined volume of the acetonitrilic or methanolic extract, prepared according to Clause 7, shall be injected into the HPLC system. The injected volume shall be the same for the extract and the standards. The device parameters are optimized and adjusted according to the manufacturer's instructions.

8.2 High-performance liquid chromatographic system

8.2.1 High-performance liquid chromatograph (HPLC)

8.2.1.1 General

Optimize the high-performance liquid chromatograph to achieve a separation of the co-eluting analytes with a separation factor of at least 1,3 (see Annex B).

8.2.1.2 Stationary phase

To separate the compounds listed in Table 1, use temperature-controlled columns packed with reversed-phase material.

NOTE For verification purposes, where applicable, repeat the chromatographic separation using a column of different selectivity; CN reversed-phase column or phenyl-hexyl reversed-phase column are recommended.

8.2.1.3 Mobile phase

In order to ensure stable retention times and optimal separation of the analytes, it is recommended to use a buffered eluent as the mobile phase (e.g. aqueous KH_2PO_4 in combination with an organic modifier, under gradient conditions).

For the analysis of hexyl, an acidic mobile phase (pH 3 to pH 4) should be used (see 6.1.7).

8.2.2 Detector

Use a diode array detector (DAD) with a wavelength of 200 nm to 400 nm. The baseline drift and the noise of the detector shall be observed periodically. The following four wavelengths are recommended for detecting the explosive compounds listed in Table 1: 210 nm, 230 nm, 254 nm, 360 nm.

8.3 Calibration

The calibration is carried out by the external standard method.

The calibration solutions are prepared as described in 5.3.2. Take care to inject the same volume for calibration as for sample measurement.

The initial calibration serves to establish the linear working range of the calibration curve. This calibration is performed when the method is used for the first time and after maintenance and/or repair of the equipment.

The validity of the calibration shall be checked at the start and at the end of every sample series by a standard solution (5.3.2), consisting of compounds representing the retention range and substance polarities of all target analytes. The validity should additionally be proved after a maximum of 20 samples per series.

The recalibration has to cover the desired working range and is to be performed using a standard solution, whenever the validity checks show a deviation > 20 % from the expected concentration.

One feature of the resulting working range is the linear correlation between concentration and measured signal; this correlation shall be ensured for each analyte through at least five concentration levels. Working ranges exceeding the concentration decade are allowed as long as they are linear.

The pairs of varieties $y_{i\mathrm{ej}}$ and $c_{i\mathrm{ej}}$ obtained from the j calibrating solutions for the reference substances i are displayed graphically and visually tested for linear dependence. If this is fulfilled, the best-fit line is calculated through linear regression using Formula (1).

$$y_{i\mathrm{e}} = a_i \times c_{i\mathrm{e}} + b_i \tag{1}$$

where

$y_{i\mathrm{e}}$ is the measuring value (signal area or height, respectively) of the substance i depending on $c_{i\mathrm{e}}$ (unit depends on evaluation);

$c_{i\mathrm{e}}$ is the mass concentration of the substance i in acetonitrilic or methanolic solution, respectively (calibrating solution), in µg/ml;

a_i is the gradient of the reference line for substance i, in ml/µg;

b_i is the intercept on the ordinate of the reference line, same unit as measuring value — b_i can have positive and negative values.

Signals (peak area or peak height) are quantified by means of the calibrating function, see Formula (1).

8.4 Identification and quantification

The compounds are identified by comparison of the retention times of the respective measuring signals with current reference retention times. Deviations in retention times as large as ± 2 % (but no more than 10 s) are acceptable.

A substance may be regarded as identified if the retention time meets the defined criteria and the achieved UV-spectra is congruent with the reference spectra of the accordant analyte.

Quantification shall be performed using the detector channel of highest response depending on the absorption maximum of the accordant analyte, e.g. 230 nm for tetryl. All other detector channels with responses resulting in signal-to-noise ratios > 10 may be used for confirmation purposes.

Only measuring signals within the working range may be quantified. If signals exceed the uppermost calibration mark, the respective extract is to be measured again after dilution of the sample. In this case, it is recommended not to exceed a higher dilution ratio than 1:100 per dilution step. Signals below the lowest calibration mark are considered not quantifiable.

9 Calculation of results

After HPLC analysis (Clause 8), calculate the concentration of the explosive compound in the soil sample using Formula (2).

The mass concentration c_{is} of the analyte i in the solid sample is

$$c_{is} = c_i \times V_T \times \frac{100}{m_f \times w_{dm}} \times f_{dil} \quad (2)$$

where

c_{is} is the mass concentration of the substance i in the solid sample, in milligrams per kilogram of dry matter (mg/kg dm);

c_i is the mass concentration of the substance i in the extract (calibrating standard or sample extract), in micrograms per millilitre (µg/ml = mg/l);

m_f is the mass of the soil used for extraction, in grams (g);

V_T is the total volume of the extract (see 7.2.2, 7.2.3, 7.2.4, 7.2.5), in millilitres (ml);

w_{dm} is the dry matter (dm) content of the soil sample, in percent of mass (%);

f_{dil} is the dilution factor of the extract.

10 Quality assurance

To demonstrate the validity of the procedure, the use of a suitable method-checking standard (e.g. 2,5-DNT or other suitable compounds not found in the sample) is recommended (Annex A). If it is not possible to choose a suitable standard, the efficiency of the extraction has to be checked frequently with the certified reference material.

The recovery rate of each single compound shall be determined, e.g. by spiking samples with other similar matrices parallel to the analysis of real samples or by performing the method of standard addition.

The determined extraction recoveries are not included in the calculation of the final result. Therefore, the constant control of the method recovery utilizing the entire analytical process, including all manual steps, has to be performed periodically. Reference materials are an adequate tool to carry out such experiments. The overall recovery rate shall be between 80 % and 110 % for each compound.

A mathematical correction of the measured values of the samples in the case of lower recoveries is not permitted.

To prove the absence of contaminants during the procedure, include at least one blank analysis per series.

Monitor the accessibility of the lower limit of the working range by an adequate standard solution with every sample series. The signal-to-noise ratio of the resulting signals for each compound shall be ≥ 10, otherwise maintenance at the HPLC-system (e.g. replacing or flushing of the column) should be performed.

The peak symmetry ($T = b/a$; measured at 10 % of peak height) should not exceed the value of 2 at any time for all analytes in order to allow proper integration and quantification.

11 Expression of results

Due to the measurement uncertainty, no more than two digits of the analytical result are significant. If, however, analytical results are included in further calculations, results are rounded to three digits (max.). Contents < 1 mg/kg dry matter shall be indicated with a precision of 0,01 mg/kg dry matter.

12 Test report

This test report shall contain at least the following information:

a) the test method used, together with a reference to this part of ISO 11916 (ISO 11916-1);

b) all information necessary for complete identification of the sample;

c) the results of the determination according to Clause 11;

d) any details not specified in this part of ISO 11916 or that are optional, as well as any other factors that may have affected the result.

Annex A
(informative)

Method-checking standard recovery limit

It is necessary to compare the method-checking standard recovery with the method-checking standard recovery limit in order to confirm the performance of the extraction and analytical system. The method-checking standard recovery limit is determined using the upper and lower control limits. The procedures for the calculation of the method-checking standard recovery limit are as follows: first, calculate the average percent recovery (p) and the standard deviation (s) for each of the method-checking standards after the analysis of 15 to 20 field samples. Calculate the upper and lower control limits for each of the method-checking standard according to the following formulae.

Upper control limit = $p + 3s$

Lower control limit = $p - 3s$

Thus, the range of method-checking standard recovery limits is from the upper control limit to the lower control limit.

The method-checking standard recovery should be in the range of the method-checking standard recovery limit.

Annex B
(informative)

HPLC-UV-DAD conditions

B.1 Example of the chromatographic conditions for the examination of 16 explosives and related compounds

HPLC-System	Gradient pump GP-50 with integrated degasser Autosampler GINA-50 Column thermostat STH 585 Detector UVD 340S
Separating column:	UltraSep ES EX 424/2, 250 mm × 3 mm internal diameter (ID), film thickness 5 µm
Eluent:	A – phosphoric buffer, pH = 4,5 B – methanol
Detection wavelength:	channel 1: 210 nm channel 2: 235 nm channel 3: 254 nm
Injection volume:	50 µl
Flow rate:	0,4 ml/min
Gradient:	35 % to 66 % methanol for 50 min
Column temperature	23 °C

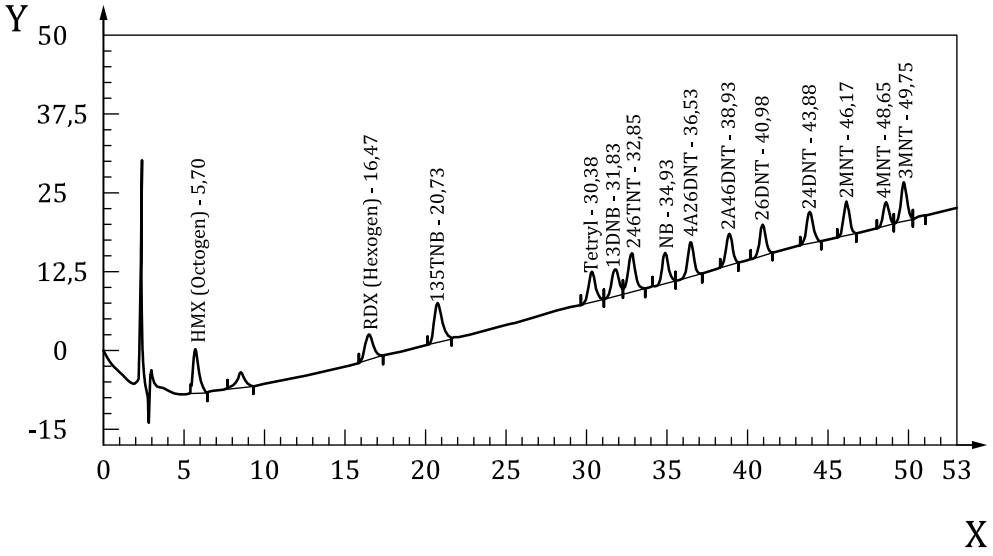
NOTE 1 Gradient pump GP-50, autosampler GINA-50, column thermostat STH 585, and detector UVD 340S are the trade names of products supplied by Dionex Corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the products named. Equivalent products may be used if they can be shown to lead to the same results.

NOTE 2 UltraSep ES EX 424/2 is the trade name of a product supplied by Separation Service Berlin. 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.

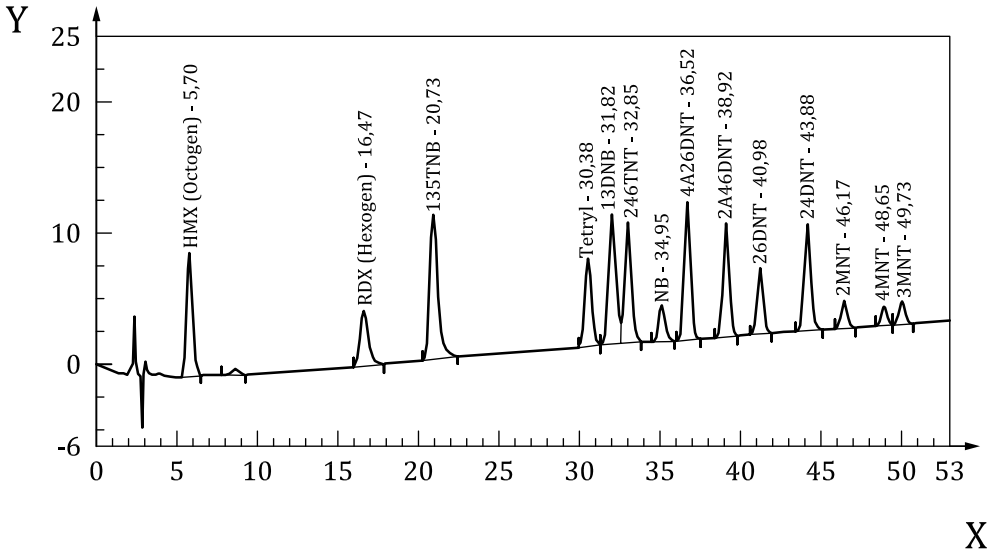
B.2 Examples of chromatograms for the effect of detection with different wavelength

See Figure B.1 and Figure B.2.

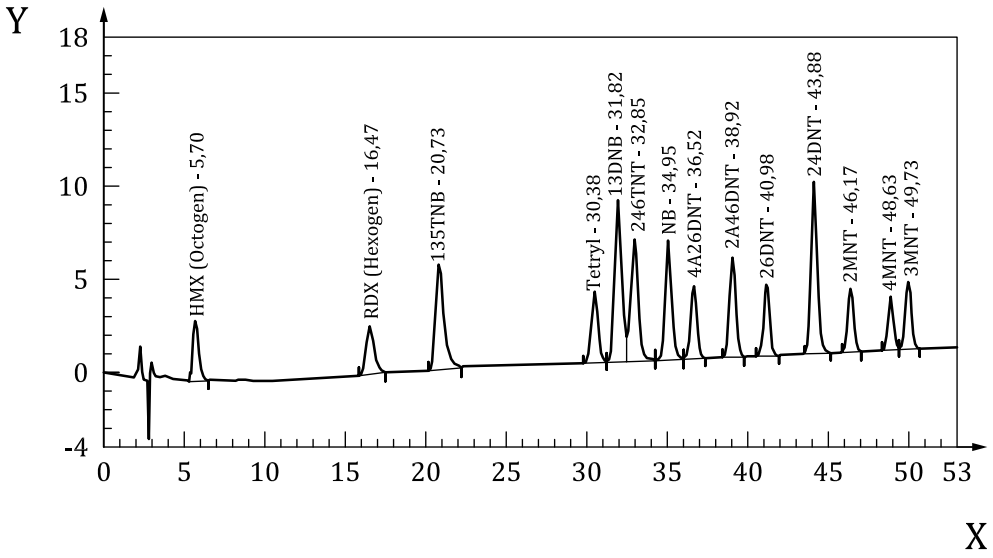
Wavelength: 210 nm



Wavelength: 235 nm



Wavelength: 254 nm



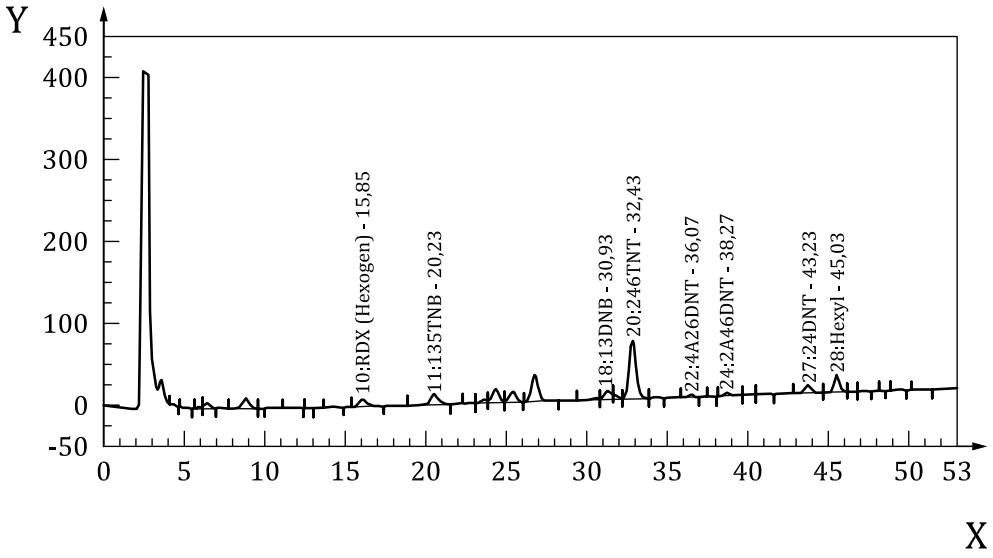
Key

X time

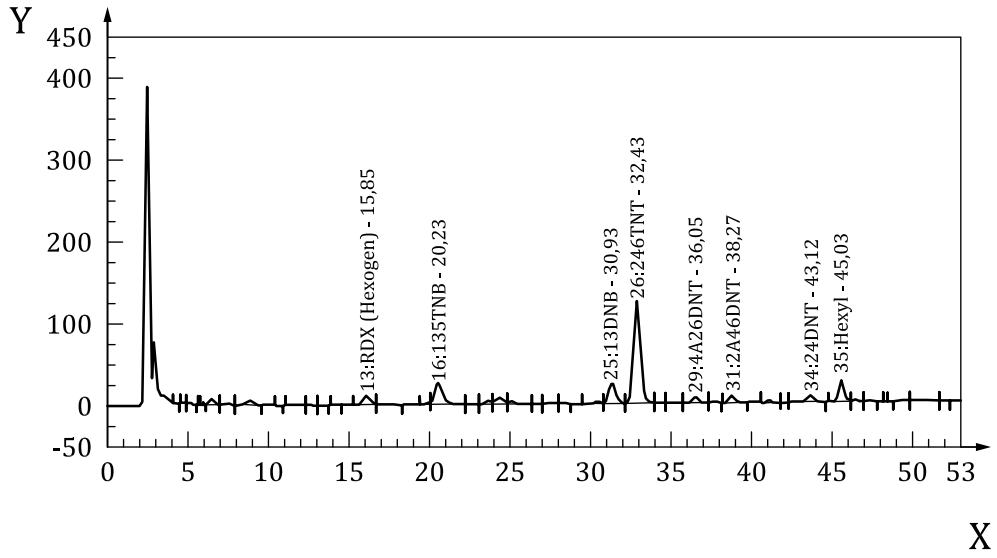
Y response, mV

Figure B.1 — Multi-component standard solution

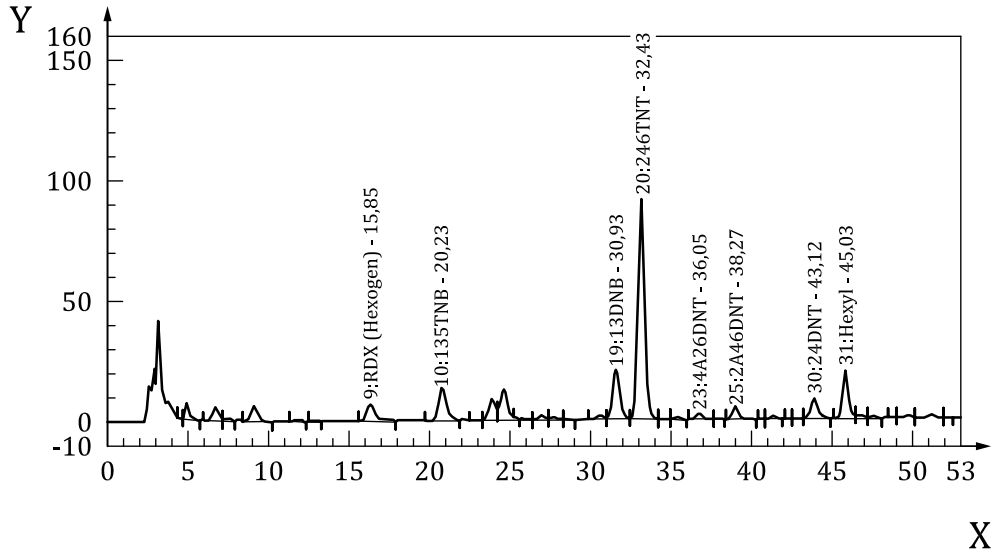
Wavelength: 210 nm



Wavelength: 235 nm



Wavelength: 254 nm



Key

X time,

Y response, mV

Figure B.2 — Extract of a contaminated soil

Annex C
(informative)

Precision data

C.1 Study concept and description of sample material

In 2011 the Fraunhofer Institute for Molecular Biology and Applied Ecology (IME) organized an interlaboratory validation study of a draft International Standard for the determination of selected explosives in soils on behalf of the German Environmental Agency (for details refer to UBA Final Report FKZ 3710 74 209).

Field samples of different sites contaminated by explosives were air-dried and sieved < 2 mm. Three individual mixtures were made from the sample materials in order to cover as many as possible of the explosive compounds listed in this part of ISO 11916. One of the three materials was spiked additionally with 1,3,5,7-tetranitro-octahydro-1,3,5,7-tetrazocine (HMX) and *N*-methyl-*N*-2,4,6-tetranitroanilin (tetryl), because these compounds were not present in the sampled soils. The mixtures were homogenized and subsamples were filled into brown glass bottles. The participants of the interlaboratory study received one sample (about 250 g dry weight) of each material. The draft version of this part of ISO 11916 served as a working instruction.

The test materials were prepared from sieved, but not ground, soil material originating from real contaminated sites. Due to well-known properties of soils contaminated with explosives, the homogeneity of the materials was partially not satisfactory. This was, in particular, true for the 2,4,6-trinitrotoluene (2,4,6-TNT) with variation coefficients (C_V) of up to 30 % in materials 1 and 3. Apart from that, variation coefficients were predominantly < 10 %, except for 1,3,5-trinitro-hexahydro-1,3,5-triazine (RDX) which had a C_V of up to 14 % in materials 1 and 2. The homogeneity obtained in the test materials reflects the reality of samples originating from sites contaminated with explosives. Thus, the result of the interlaboratory study as presented below allows a realistic assessment of data determined according to this part of ISO 11916.

For quality assurance two standard solutions containing several explosives dissolved in methanol were sent to each participating laboratory. The concentration of each explosive substance in the standard solution was about 2,5 mg per litre which was communicated to the participants.

Tables C.1 to C.5 summarize the results of the interlaboratory study. Data evaluation was performed according to ISO 5725-2. Data listed are given as milligrams per kilogram of dry soil.

Table C.1 — HPLC-UV data of extracts from mechanical shaking extraction (MSE)

Material 1	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	6	12		0,0	0,14	0,06	43,97	0,04	24,82
2,4-DNT	11	25	2	7,4	5,96	1,17	19,58	0,61	10,30
2,4,6-TNT	11	25	2	7,4	13,51	5,60	41,48	5,60	41,48
4-A-2,6-DNT	4	8	2	20,0	0,13	0,09	70,00	0,02	11,54
2-A-4,6-DNT	5	9		0,0	0,13	0,08	63,36	0,02	11,45
RDX	13	30		0,0	19,54	3,05	15,62	2,26	11,54
PETN	11	25		0,0	61,26	13,50	22,04	2,50	4,07
2,6-DNT	13	29		0,0	2,42	0,76	31,39	0,31	12,85

Table C.1 *(continued)*

Material 1	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
Material 2	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	10	24		0,0	0,69	0,24	34,45	0,09	13,28
2-NT	10	22	3	12,0	9,88	1,60	16,14	0,42	4,26
3-NT	11	22		0,0	1,20	0,71	58,88	0,39	32,61
4-NT	12	27		0,0	4,23	0,69	16,26	0,40	9,40
2,4-DNT	12	27		0,0	5,68	0,84	14,82	0,64	11,27
2,4,6-TNT	12	29		0,0	440,0	169,7	38,57	153,9	34,97
4-A-2,6-DNT	13	31		0,0	17,71	4,57	25,78	1,87	10,56
2-A-4,6-DNT	13	27		0,0	8,76	2,75	31,34	0,47	5,35
Hexyl	9	19	5	20,8	40,08	9,68	24,16	5,15	12,85
RDX	12	27	5	15,6	3,07	1,93	63,04	0,29	9,52
PETN	5	8		0,0	4,17	1,32	31,76	0,13	3,10
2,6-DNT	12	26		0,0	2,49	0,49	19,78	0,48	19,33
Material 3	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	9	20	1	4,8	0,50	0,08	16,30	0,06	11,27
2,4-DNT	5	12		0,0	0,49	0,21	43,12	0,16	31,78
2,4,6-TNT	13	29		0,0	130,5	85,73	65,68	61,36	47,00
4-A-2,6-DNT	12	24	3	11,1	6,80	1,27	18,74	0,41	6,05
2-A-4,6-DNT	13	27		0,0	3,43	1,11	32,53	0,37	10,66
Tetryl	12	28		0,0	15,76	7,34	46,56	1,85	11,76
Hexyl	8	16	7	30,4	22,89	8,32	36,34	2,75	12,03
RDX	13	30		0,0	9,30	4,59	49,39	0,88	9,43
HMX	13	30		0,0	70,19	54,48	77,62	9,03	12,87
PETN	11	25		0,0	7,13	2,45	34,29	0,77	10,83

Explanation of symbols

l	Number of laboratories after elimination of outliers
n	Number of single-analysis data without outliers
n_A	Number of outliers
n_{AP}	Number of outliers in %
x	Mean value in mg/kg dry matter
s_R	Standard deviation of reproducibility in mg/kg dry matter
$C_{V,R}$	Variation coefficient of reproducibility in %
s_r	Standard deviation of repeatability in mg/kg dry matter
$C_{V,r}$	Variation coefficient of repeatability in %

Table C.2 — HPLC-UV data of extracts from ultrasonification (USE)

Material 1	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	8	16		0,0	0,16	0,08	49,39	0,01	6,71
2,4-DNT	13	28		0,0	7,09	3,28	46,28	3,17	44,69
2,4,6-TNT	11	23	3	11,5	12,82	2,40	18,72	1,60	12,46
4-A-2,6-DNT	5	11	1	8,3	0,25	0,15	59,20	0,04	16,80
2-A-4,6-DNT	6	13	1	7,1	0,15	0,09	58,17	0,03	18,95
RDX	12	27	3	10,0	19,52	2,88	14,75	2,71	13,90
PETN	11	23	3	11,5	56,55	23,40	41,38	5,55	9,82
2,6-DNT	13	28		0,0	2,76	1,17	42,29	0,89	32,39
Material 2	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	10	22		0,0	0,73	0,31	41,95	0,21	28,20
2-NT	11	23	3	11,5	9,20	1,74	18,92	0,81	8,75
3-NT	8	14	5	26,3	1,07	0,60	56,53	0,05	5,07
4-NT	10	19	9	32,1	4,18	0,71	17,08	0,11	2,56
2,4-DNT	10	20	4	16,7	5,57	1,00	17,92	0,26	4,58
2,4,6-TNT	12	28		0,0	384,1	89,35	23,26	81,77	21,29
4-A-2,6-DNT	12	26	3	10,3	18,27	4,96	27,14	1,83	9,99
2-A-4,6-DNT	13	25	1	3,8	8,52	3,54	41,48	0,74	8,71
Hexyl	9	19	2	9,5	38,97	22,40	57,47	11,75	30,16
RDX	11	23	8	25,8	2,98	1,87	62,74	0,21	7,08
PETN	5	9	2	18,2	3,02	1,61	53,31	0,25	8,38
2,6-DNT	11	24	2	7,7	2,48	0,85	34,26	0,39	15,56
Material 3	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	10	21	2	8,7	0,57	0,18	31,10	0,11	18,73
2,4-DNT	6	14	2	12,5	0,54	0,22	40,11	0,15	27,36
2,4,6-TNT	13	28		0,0	122,1	65,93	54,01	50,73	41,56
4-A-2,6-DNT	13	28		0,0	8,79	3,03	34,43	1,42	16,09
2-A-4,6-DNT	11	21	5	19,2	3,01	0,77	25,57	0,37	12,26
Tetryl	12	27		0,0	14,49	4,17	28,79	1,40	9,67
Hexyl	10	20	2	9,1	21,74	11,69	53,75	4,19	19,25
RDX	13	29		0,0	10,48	4,93	47,05	1,24	11,81
HMX	12	26	3	10,3	68,74	54,80	79,71	2,21	3,21
PETN	11	24		0,0	7,93	2,90	36,50	1,18	14,84

Explanation of symbols:

See Table C.1.

Table C.3 — HPLC-UV data of extracts from Soxhlet extraction (SOX)

Material 1	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB[a]	*3*	*6*		*0,0*	*0,29*	*0,16*	*56,55*	*0,06*	*18,97*
2,4-DNT	7	16	2	11,1	5,38	1,08	20,14	0,62	11,48
2,4,6-TNT	6	14	2	12,5	12,22	3,84	31,41	3,80	31,11
4-A-2,6-DNT[a]	*2*	*5*		*0,0*	*0,26*	*0,25*	*97,67*	*0,04*	*17,05*
2-A-4,6-DNT[a]	*3*	*7*		*0,0*	*0,30*	*0,23*	*77,03*	*0,12*	*38,85*
RDX	7	16	2	11,1	16,80	5,11	30,44	1,73	10,32
PETN	6	13	2	13,3	38,73	14,16	36,56	3,83	9,89
2,6-DNT	7	16		0,0	2,31	1,34	58,26	0,19	8,20
Material 2	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	6	13		0,0	1,71	1,01	59,00	0,27	15,54
2-NT	6	13		0,0	10,72	6,58	61,41	0,84	7,86
3-NT	4	8		0,0	1,29	0,58	45,10	0,18	13,92
4-NT	7	15		0,0	4,56	2,44	53,43	0,41	8,94
2,4-DNT	5	11	2	15,4	5,14	1,16	22,60	0,35	6,71
2,4,6-TNT	6	13	2	13,3	397,9	136,4	34,29	128,9	32,40
4-A-2,6-DNT	7	15	2	11,8	19,46	4,81	24,71	1,26	6,47
2-A-4,6-DNT	8	17		0,0	12,00	5,86	48,80	0,35	2,93
Hexyl	4	8	4	33,3	43,43	13,20	30,39	2,09	4,81
RDX	5	10	2	16,7	4,07	1,79	44,02	0,22	5,50
PETN[a]	*2*	*3*		*0,0*	*3,18*	*0,30*	*9,44*	*0,16*	*4,91*
26DNT	6	13		0,0	2,96	2,01	68,02	0,25	8,41
Material 3	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	6	13		0,0	0,83	0,60	72,65	0,13	15,78
2,4-DNT[a]	*3*	*6*		*0,0*	*1,55*	*1,64*	*105,73*	*0,13*	*8,24*
2,4,6-TNT	6	14	2	12,5	108,4	73,80	68,06	66,63	61,45
4-A-2,6-DNT	8	18		0,0	10,57	4,33	41,00	0,72	6,81
2-A-4,6-DNT	7	16		0,0	4,99	2,54	50,97	0,35	6,97
Tetryl[a]	*3*	*7*	*2*	*22,2*	*5,64*	*5,90*	*104,5*	*0,13*	*2,23*
Hexyl	5	11	2	15,4	27,05	9,61	35,51	5,86	21,68
RDX	6	13		0,0	16,86	9,64	57,20	2,17	12,89
HMX	7	16	2	11,1	90,22	22,94	25,43	7,42	8,22
PETN	5	11	2	15,4	5,59	2,40	42,85	0,72	12,78

Explanation of symbols:

See Table C.1.

[a] Less than 8 single analysis data sets.

Table C.4 — HPLC-UV data of extracts from pressurized liquid extraction (PLE)

Material 1	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB[a]	*3*	*7*		*0,0*	*0,19*	*0,06*	*29,53*	*0,02*	*9,33*
2,4-DNT	4	9		0,0	5,41	0,76	14,06	0,13	2,37
2,4,6-TNT	5	12		0,0	14,71	6,98	47,45	2,98	20,27
4-A-2,6-DNT[a]	*2*	*5*		*0,0*	*0,15*	*0,06*	*43,24*	*0,02*	*16,22*
2-A-4,6-DNT[a]	*2*	*3*		*0,0*	*0,10*	*0,04*	*34,95*	*0,01*	*13,59*
RDX	5	12		0,0	18,72	3,72	19,87	2,52	13,45
PETN	4	9		0,0	56,54	12,43	21,99	3,25	5,75
2,6-DNT	5	12		0,0	2,11	0,46	21,71	0,09	4,42
Material 2	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	4	10		0,0	0,91	0,06	6,27	0,02	1,98
2-NT	5	12		0,0	8,15	1,85	22,74	0,67	8,26
3-NT[a]	*3*	*7*	*2*	*22,2*	*0,81*	*0,24*	*30,09*	*0,06*	*7,15*
4-NT	5	12		0,0	3,66	0,96	26,16	0,31	8,50
2,4-DNT	4	10		0,0	4,92	0,68	13,87	0,22	4,56
2,4,6-TNT	5	12		0,0	389,3	129,6	33,29	64,27	16,51
4-A-2,6-DNT	5	12		0,0	20,21	4,93	24,37	1,13	5,58
2-A-4,6-DNT	5	12		0,0	11,08	4,57	41,27	1,08	9,77
Hexyl	3	8	2	20,0	42,41	9,23	21,77	3,07	7,23
RDX	5	12		0,0	5,29	1,17	22,18	0,50	9,43
PETN[a]	*1*	*2*		*0,0*	*11,35*		*0,00*		*0,00*
2,6-DNT	4	10		0,0	2,49	0,39	15,65	0,21	8,53
Material 3	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	4	10		0,0	0,59	0,05	8,59	0,04	7,41
2,4-DNT	1	3		0,0	0,35		0,00		0,00
2,4,6-TNT	5	12		0,0	115,1	63,48	55,18	33,03	28,71
4-A-2,6-DNT	5	12		0,0	7,89	2,17	27,54	0,46	5,88
2-A-4,6-DNT	5	12		0,0	3,97	1,56	39,34	0,29	7,31
Tetryl	5	12		0,0	8,48	5,75	67,89	1,88	22,16
Hexyl	4	10		0,0	47,34	51,13	108,0	1,28	2,70
RDX	5	12		0,0	11,55	3,66	31,69	0,55	4,78
HMX	5	12		0,0	81,74	37,37	45,72	3,17	3,88
PETN	4	10		0,0	5,89	1,30	22,08	0,69	11,64

Explanation of symbols:

See Table C.1.

[a] Less than 8 single analysis data sets.

Table C.5 — HPLC-UV analysis of the explosives standard solutions

Solution 1	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3-DNB	11	25	4	13,8	2,41	0,18	7,26	0,06	2,61
2,4,6-TNT	10	24	4	14,3	2,58	0,13	4,93	0,10	3,80
4-A-2,6-DNT	12	28	1	3,4	2,54	0,20	7,91	0,09	3,70
2-A-4,6-DNT	13	29		0,0	2,46	0,37	15,05	0,08	3,25
Hexyl	10	19	3	13,6	2,18	0,60	27,42	0,08	3,44
RDX	13	29		0,0	2,40	0,41	17,17	0,14	5,79
HMX	12	26	3	10,3	2,21	0,43	19,50	0,06	2,72
PETN	12	27		0,0	2,35	0,48	20,54	0,24	10,31
Solution 2	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
1,3,5-TNB	11	26	3	10,3	2,46	0,16	6,63	0,09	3,70
2-NT	11	27	1	3,6	2,41	0,26	10,71	0,10	4,32
3-NT	12	28	1	3,4	2,48	0,24	9,52	0,12	4,72
4-NT	11	26	3	10,3	2,46	0,31	12,53	0,05	2,07
2,4-DNT	12	28	1	3,4	2,45	0,22	8,76	0,08	3,06
2,4,6-TNT	11	26	3	10,3	2,80	0,35	12,44	0,06	2,18
4-A-2,6-DNT	12	28	1	3,4	2,57	0,21	7,99	0,11	4,13
2-A-4,6-DNT	12	28	1	3,4	2,52	0,22	8,90	0,11	4,37
Tetryl	10	23	2	8,0	2,87	1,48	51,51	0,19	6,68
2,6-DNT	12	28	1	3,4	2,47	0,15	6,24	0,08	3,37

Explanation of symbols:

x, s_R and s_r in mg/l, all other symbols see Table C.1.

C.2 Supplemental experiments

As indicated by the variation coefficient of reproducibility, the participating laboratories were able to analyse the explosives standard solutions with satisfying reproducibility. However, the data also showed high variability for substances Hexyl, HMX, PETN and tetryl.

At an expert meeting on March 13th 2012 in Frankfurt (Germany), organized by the Fh-IME, some concern was raised about the consistency of the quality of the commercial standard calibration solutions, and the test materials that may separate and settle due to particle size during storage. It was concluded that an additional test with one ground material would be conducted. For calibration of the analytical instruments a multi-standard solution (10 mg/l of each compound dissolved in methanol) was sent to each laboratory. To check the performance of the chemical analysis, a separate multi-standard test solution in methanol was sent to the laboratories for blind testing. The concentration of each compound was 2 mg/l remained unknown to the laboratories.

As the results shown in Table C.6 demonstrate, the provision of a common calibration standard improved the results of the chemical analysis significantly with variation coefficient of reproducibility below 10 % for each compound, except for PETN.

Table C.6 — HPLC-UV analysis of the explosives standard solutions with common calibration solution

Substance	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
Tetryl	11	32	3	8,6	1,79	0,16	8,91	0,04	2,42
2,4,6-TNT	10	30	2	6,3	2,08	0,12	5,53	0,05	2,43
4-A-2,6-DNT	11	32	0	0,0	2,00	0,12	5,94	0,06	3,01
2-A-4,6-DNT	10	29	3	9,4	2,02	0,12	5,99	0,04	2,02
Hexyl	9	28	5	15,2	1,97	0,19	9,78	0,19	9,78
RDX	11	32	3	8,6	2,07	0,11	5,06	0,10	4,62
1,3,5-TNB	10	30	2	6,3	2,06	0,05	2,48	0,03	1,31
2-NT	10	30	0	0,0	2,00	0,09	4,38	0,07	3,52
3-NT	10	30	2	6,3	2,02	0,12	5,69	0,08	3,97
4-NT	10	30	2	6,3	1,99	0,09	4,44	0,07	3,59
2,4-DNT	10	30	2	6,3	1,97	0,06	2,92	0,04	2,22
2,6-DNT	10	30	2	6,3	2,03	0,08	4,00	0,07	3,60
PETN	10	29	0	0,0	2,83	0,71	24,94	0,09	3,12
Explanation of symbols: x, s_R and s_r in mg/l, all other symbols see Table C.1.									

An additional test material originating from an explosives contaminated site was ground to 250 µm particle size by the German Federal Institute for Materials Research and Testing. The homogeneity of the material was determined by Fh-IME. However, the grinding did not result in a significant improvement of the explosives distribution within the soil material. But due to the better homogeneity of the material no separation of particles was observed. Thus, subsamples were much more representative of the material. In combination with the more reliable chemical analysis of the common calibration solutions, the potential of the present ISO standard to obtain reproducible results is demonstrated in the following tables.

The data set obtained for PLE was not sufficient for a reliable statistical analysis. But in order to get an idea of the PLE performance with the new material the data are presented in Tables C.7 and C.8.

Table C.7 — HPLC-UV data of extracts from ground material

MSE	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
Tetryl	7	24	4	14,3	158,3	49,98	31,57	5,72	3,61
2,4,6-TNT	6	22	4	15,4	275,9	14,07	5,10	4,94	1,79
4-A-2,6-DNT	8	28	0	0,0	16,30	5,54	33,96	0,51	3,14
2-A-4,6-DNT	8	28	0	0,0	8,92	3,43	38,44	0,26	2,90
Hexyl	6	22	4	15,4	40,29	7,13	17,70	1,23	3,05
RDX	6	20	8	28,6	104,4	41,73	39,95	1,06	1,01
1,3,5-TNB	7	24	0	0,0	0,61	0,14	23,14	0,07	11,47
2-NT	6	22	4	15,4	6,76	0,69	10,20	0,25	3,76
3-NT	7	24	0	0,0	0,58	0,17	29,38	0,11	19,00
4-NT	6	20	8	28,6	3,15	0,70	22,22	0,16	5,05
2,4-DNT	7	24	0	0,0	4,73	0,50	10,52	0,24	5,10
2,6-DNT	6	20	4	16,7	2,74	0,69	25,26	0,18	6,56
PETN	3	10	8	44,4	2,28	1,31	57,48	0,03	1,42
USE	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
Tetryl	7	24	8	25,0	160,7	57,76	35,95	3,93	2,45
2,4,6-TNT	8	30	4	11,8	283,3	14,39	5,08	6,23	2,20
4-A-2,6-DNT	9	32	4	11,1	19,17	2,11	10,99	0,42	2,20
2-A-4,6-DNT	10	36	0	0,0	9,09	2,67	29,35	0,35	3,83
Hexyl	9	34	0	0,0	38,89	15,27	39,27	1,40	3,61
RDX	8	28	8	22,2	100,6	42,85	42,61	1,50	1,50
1,3,5-TNB	7	23	4	14,8	0,62	0,13	21,42	0,05	8,38
2-NT	9	34	0	0,0	7,01	0,69	9,87	0,28	4,03
3-NT	7	26	2	7,1	0,71	0,14	20,27	0,08	11,77
4-NT	9	32	4	11,1	3,37	0,43	12,64	0,19	5,71
2,4-DNT	7	24	8	25,0	5,01	0,53	10,63	0,09	1,78
2,6-DNT	9	32	0	0,0	2,54	0,46	17,95	0,18	6,96
PETN	5	18	4	18,2	2,71	1,13	41,76	0,13	4,74

Explanation of symbols:

See Table C.1.

Table C.8 — HPLC-UV data of extracts from ground material

SOX	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
Tetryl	4	13	0	0,0	98,85	52,47	53,08	8,18	8,27
2,4,6-TNT	4	16	0	0,0	215,7	45,85	21,25	14,82	6,87
4-A-2,6-DNT	4	14	4	22,2	13,59	7,11	52,29	1,08	7,98
2-A-4,6-DNT	4	14	4	22,2	8,35	5,18	62,11	0,55	6,53
Hexyl	3	12	4	25,0	31,35	14,99	47,84	3,04	9,71
RDX	5	18	0	0,0	91,05	38,16	41,91	13,04	14,32
1,3,5-TNB	3	10	4	28,6	0,55	0,23	42,18	0,03	6,03
2-NT	3	12	4	25,0	5,23	1,71	32,70	0,30	5,77
3-NT	4	14	0	0,0	0,51	0,27	52,31	0,08	16,37
4-NT	5	18	0	0,0	2,90	1,56	53,70	0,24	8,40
2,4-DNT	3	10	4	28,6	3,86	1,26	32,66	0,13	3,28
2,6-DNT	4	14	0	0,0	2,31	1,14	49,34	0,23	9,93
PETN[a]	*1*	*4*	*0*	*0,0*	*0,86*				
PLE	l	n	n_A	n_{AP} %	x	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
Tetryl[a]	*2*	*7*	*0*	*0,0*	*125,3*	*16,87*	*13,46*	*15,82*	*12,62*
2,4,6-TNT	2	8	4	33,3	175,2	52,91	30,20	9,77	5,58
4-A-2,6-DNT	2	8	4	33,3	18,06	9,49	52,57	0,99	5,47
2-A-4,6-DNT	2	8	4	33,3	10,85	8,07	74,35	0,44	4,05
Hexyl	2	8	4	33,3	49,10	15,43	31,42	0,74	1,51
RDX	2	8	4	33,3	115,9	38,79	33,44	5,13	4,42
1,3,5-TNB[a]	*2*	*7*	*0*	*0,0*	*1,24*	*0,09*	*7,36*	*0,07*	*5,73*
2-NT	2	8	4	33,3	5,81	3,88	66,76	0,34	5,89
3-NT[a]	*2*	*5*	*0*	*0,0*	*0,42*	*0,04*	*8,55*	*0,04*	*8,55*
4-NT	2	8	4	33,3	2,69	1,72	63,98	0,14	5,16
2,4-DNT[a]	*2*	*7*	*0*	*0,0*	*5,71*	*1,93*	*33,80*	*0,22*	*3,88*
2,6-DNT[a]	*2*	*7*	*0*	*0,0*	*3,16*	*0,83*	*26,18*	*0,09*	*2,84*
PETN[a]	*1*	*4*	*0*	*0,0*	*0,95*				

Explanation of symbols:

See Table C.1.

[a] Less than 8 single analysis data sets.

Bibliography

[1] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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