

**Vattenundersökningar – Bestämning av några utvalda explosiva föreningar med biprodukter – Vätskekromatografisk metod (HPLC) med UV-detektion (ISO 22478:2006)**

**Water quality – Determination of certain explosives and related compounds – Method using high performance liquid chromatography (HPLC) with UV detection (ISO 22478:2006)**

Europastandarden EN ISO 22478:2006 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 22478:2006.

The European Standard EN ISO 22478:2006 has the status of a Swedish Standard. This document contains the official English version of EN ISO 22478:2006.

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**EN ISO 22478**

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English Version

**Water quality - Determination of certain explosives and related compounds - Method using high-performance liquid chromatography (HPLC) with UV detection (ISO 22478:2006)**

Qualité de l'eau - Dosage de certains explosifs et de composés apparentés - Méthode utilisant la chromatographie en phase liquide à haute performance (CLHP) avec détection UV (ISO 22478:2006)

Wasserbeschaffenheit - Bestimmung ausgewählter Explosivstoffe und verwandter Verbindungen - Verfahren mittels Hochleistungs-Flüssigkeitschromatographie (HPLC) mit UV-Detektion (ISO 22478:2006)

This European Standard was approved by CEN on 6 February 2006.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## Foreword

This document (EN ISO 22478:2006) has been prepared by Technical Committee ISO/TC 147 "Water quality" in collaboration with Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2006, and conflicting national standards shall be withdrawn at the latest by August 2006.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

## Endorsement notice

The text of ISO 22478:2006 has been approved by CEN as EN ISO 22478:2006 without any modifications.

**EN ISO 22478:2006 (E)****Introduction**

Explosives and related compounds are frequently encountered in groundwater areas near to soil sites contaminated by armaments waste and may also be found in drinking water taken from nearby catchment areas. The range of pollutants will depend on the waste concerned, but will not, as a rule, include all the compounds listed in Table 1. Instead, samples of groundwater containing such pollutants may contain numerous other substances, such as nitro- and dinitrobenzoic acid, nitrophenols and aromatic amines. The compounds listed in Table 1 are frequently used for exploratory examinations of armaments waste.

When using this International Standard, it may be necessary in some cases to determine whether and to what extent particular problems will require the specification of additional conditions.

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

**WARNING** — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

**IMPORTANT** — It is absolutely essential that tests conducted in accordance with this International Standard be carried out by suitably trained staff.

## 1 Scope

This International Standard specifies a method for determination of certain explosives, in particular nitrotoluenes, nitroamines and nitrate esters, and related compounds (by-products and degradation products), such as those listed in Table 1, in drinking water, groundwater and surface water.

Depending on the type of sample and the compound to be analysed, the lower limit of the working range for nitroaromatics and nitramines can be assumed to be between 0,1 µg/l and 0,5 µg/l (in some cases, the lower limit may be extended down to 0,05 µg/l). The lower limit of the working range for nitrate esters may be assumed to be higher (0,5 µg/l or more).

Similar compounds, in particular other nitroaromatics, may also be determined by this method, but its applicability will have to be checked in each individual case.

## 2 Normative references

The following referenced documents are indispensable for the application 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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 5667-2, *Water quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

## 3 Principle

The substances in the water sample are concentrated by solid-phase extraction using a polystyrene/divinylbenzene-based adsorbent. After elution with a solvent mixture, the eluate is concentrated and the substances are separated by high-performance liquid chromatography (HPLC) and analysed using a UV photodiode array (UV-PDA) detector.

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**Table 1 — Explosives and related compounds determined by this method** (the compounds listed here are particularly likely to be encountered in water samples containing armaments waste)

Name	Abbreviation <sup>a</sup>	Other name	CAS <sup>b</sup> No.	Molar mass g/mol	Reference No./ letter in example chromatograms in Clause			
					A.1	A.2	A.3	B.1
2,4,6-Trinitrophenol	PA	Picric acid	88-89-1	229,1	1	2	1	a
1,3,5,7-Tetranitro-octahydro-1,3,5,7-tetrazocine	HMX	Octogen	2691-41-0	296,2	2	1	2	b
1,3,5-Trinitro-hexahydro-1,3,5-triazine	RDX	Hexogen	121-82-4	222,1	3	3	4	d
2,2',4,4',6,6'-Hexanitrodiphenylamine	—	Hexyl	131-73-7	439,2	4	19	3	c
Ethylene glycol dinitrate	EGDN	—	628-96-6	152,1	5	4	5	e
Diethylene glycol dinitrate	DEGN	—	693-21-0	196,1	6	5	6	f
1,3,5-Trinitrobenzene	1,3,5-TNB	—	99-35-4	213,1	7	6	7	g
1,3-Dinitrobenzene	1,3-DNB	—	99-65-0	168,1	8	7	9	i
<i>N</i> -Methyl- <i>N</i> -2,4,6-tetranitroaniline	CE	Tetryl	479-45-8	287,2	9	8	8	h
Glycerol trinitrate	NG	Nitroglycerine	55-63-0	227,1	10	9	10	j
2,4,6-Trinitrotoluene	2,4,6-TNT	TNT	118-96-7	227,1	11	10	11	k
4-Amino-2,6-dinitrotoluene	4-A-2,6-DNT	—	19406-51-6	197,1	12	11	12	l
2-Amino-4,6-dinitrotoluene	2-A-4,6-DNT	—	35572-78-2	197,1	13	12	13	m
2,6-Dinitrotoluene	2,6-DNT	—	606-20-2	182,1	14	13	14	n
2,4-Dinitrotoluene	2,4-DNT	—	121-14-2	182,1	15	14	15	o
2-Nitrotoluene	2-NT	—	88-72-2	137,1	16	15	17	q
Pentaerythritol tetranitrate	PETN	Nitropenta	78-11-5	316,2	17	18	16	p
4-Nitrotoluene	4-NT	—	99-99-0	137,1	18	16	18	r
3-Nitrotoluene	3-NT	—	99-08-1	137,1	19	17	19	s
Diphenylamine	DPA	—	122-39-4	169,24	20	—	—	—

<sup>a</sup> Standard abbreviations, some of Anglo-Saxon origin.  
<sup>b</sup> Chemical Abstracts Service.

The structural formulae of selected compounds are given in Figure 1.

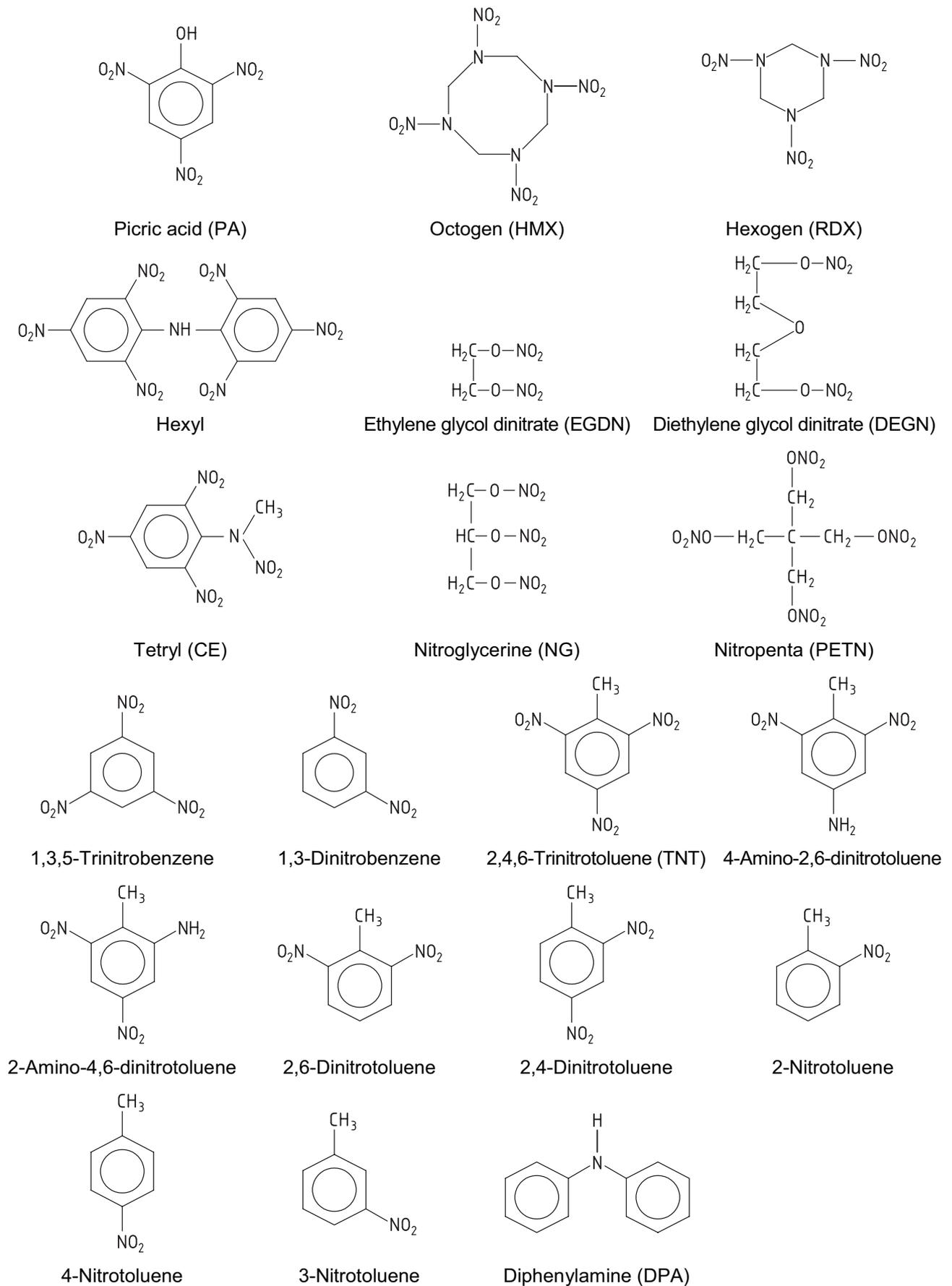


Figure 1 — Structural formulae of selected explosives

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### 4 Interference

#### 4.1 Sampling

To avoid interference, collect samples as specified in Clause 7, observing the instructions given in ISO 5667-1, ISO 5667-2 and ISO 5667-3.

#### 4.2 Filtration

If the sample contains suspended matter, first filter it through a borosilicate glass fibre filter to prevent blockage of the solid phase concentrating cartridge and record this operation in the test report.

#### 4.3 Concentration by evaporation

Mononitrotoluenes may be lost when the eluates are evaporated as specified in 8.2.

#### 4.4 Chromatography

Asymmetrical peaks and peaks that are wider than normal may indicate incompletely resolved peaks or peak overlapping due to compounds with similar retention times and/or absorption at the same or at a similar wavelength to those of the compounds being determined. To detect peak overlaps, check the peak purity and compare the UV spectrum with that of a reference material.

Interference may also be caused by humic matter eluted in the same range as the analytes being determined.

#### 4.5 Exposure to light

Reference solutions, samples and sample extracts may decompose on exposure to light. This is particularly important in the case of tetryl (see Annex C). Therefore a detailed investigation of the recovery of tetryl should be carried out.

#### 4.6 Determination of hexogen

When determining small concentrations of hexogen, interference may be caused by high concentrations of nitroaromatics.

### 5 Reagents

The reagents shall not have blank values that would interfere with the HPLC analysis.

If available, use reagents of HPLC grade or residue grade. The solvents shall not contain any measurable UV-absorbing substances that interfere with the method.

**5.1 Water**, complying with grade 1 as defined in ISO 3696. The water shall not contain any measurable quantities of UV-absorbing substances that interfere with the determination.

**5.2 Nitrogen**, purity not less than 99,996 % by volume, for drying the adsorbent and, if necessary, for concentrating the eluates.

**5.3 Helium**, purity not less than 99,996 % by volume, for degassing the eluent if a vacuum degasser is not used.

**5.4 Microporous polystyrene/divinylbenzene adsorbent**, with a high specific surface area (e.g. greater than 900 m<sup>2</sup>/g). Other adsorbents may be used provided that the recovery rate is greater than 85 % for octogen (HMX) at a concentration of 1 µg/l in a 1 l sample.

**5.5 Methanol**, CH<sub>3</sub>OH.

**5.6 Acetonitrile**, CH<sub>3</sub>CN.

**5.7 Sodium chloride**, NaCl.

**5.8 Potassium dihydrogen phosphate solution**,  $c(\text{KH}_2\text{PO}_4) = 0,025 \text{ mol/l}$  (3,40 g/l).

**5.9 Orthophosphoric acid**,  $w(\text{H}_3\text{PO}_4) = 85 \%$ .

**5.10 Reference compounds**, as listed in Table 1, some of which are obtainable only as commercial solutions with a specified mass concentration.

**5.11 Solutions of reference compounds**, prepared as follows:

Add methanol (5.5) to 100 mg of the reference compound in a 100 ml volumetric flask, dissolve the reference compound and make up to volume with methanol.

Prepare solutions of hexyl and octogen using acetonitrile (5.6) because of their poor solubility in methanol.

Store the solutions at a maximum of 6 °C, protected from light; their shelf-life is limited (six months at most).

**5.12 Stock solutions**, prepared as follows:

Transfer 1 ml of each of the solutions of reference compounds (5.11) to a separate 100 ml volumetric flask and make up to volume with methanol (5.5).

Store the solutions at a maximum of 6 °C, protected from light; the solutions are stable for up to six months.

**5.13 Reference solutions for multipoint calibration**, prepared as follows:

Prior to each calibration, prepare at least five dilutions of each of the stock solutions prepared in 5.12, preferably using a methanol/water mixture (50 + 50 parts by volume) as solvent.

Store the reference solutions at a maximum of 6 °C, protected from light; check their concentrations regularly as their stability is limited.

**5.14 Buffer solution**, to improve gradient elution of hexyl and picric acid, prepared by adjusting the pH of a 0,025 mol/l solution of potassium dihydrogen phosphate (5.8) to 3,2 using orthophosphoric acid (5.9).

## 6 Apparatus

Those parts of the apparatus that come into contact with the sample or the extract shall be free of compounds that may produce blank values. The apparatus shall be made of materials that do not adsorb the compounds under investigation and that provide protection from light, e.g. brown glass, stainless steel or perfluorinated plastics.

**6.1 Brown-glass narrow-necked flat-bottomed flasks**, 1 000 ml.

**6.2 Vacuum or pressure equipment**, for sample enrichment.

**6.3 Glass fibre filter**, diameter of fibres 0,75 µm to 1,5 µm, of borosilicate glass containing an inorganic binder.

**6.4 Solvent-resistant micromembrane filter**, e.g. polyamide or cellulose membrane, pore size of 0,2 µm to 0,45 µm, for filtration of HPLC extracts.

**6.5 Glass or polypropene cartridges**, packed with a polystyrene/divinylbenzene-based adsorbent (5.4).