# INTERNATIONAL STANDARD

# ISO 11709

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# Soil quality — Determination of selected coal-tar-derived phenolic compounds using high performance liquid chromatography (HPLC)

*Qualité du sol — Dosage d'une sélection de composés phénoliques dérivés du goudron de houille en utilisant la chromatographie liquide à haute performance (CLHP)*

Reference number
ISO 11709:2011(E)

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 11709 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

# Soil quality — Determination of selected coal-tar-derived phenolic compounds using high performance liquid chromatography (HPLC)

## 1 Scope

This International Standard specifies a method for the quantitative determination of selected coal-tar-derived phenols, namely phenol, methylphenols such as (*ortho*-, *meta*-, *para*-)cresols, and dihydroxybenzenes such as catechol, resorcinol and hydroquinone (see Table 1) in soil by using high performance liquid chromatography with ultraviolet/diode array (HPLC/UV/DAD) or fluorescence (HPLC/FLD) or electrochemical detection (HPLC/ELCD). It is applicable to all types of soil with contamination levels of individual phenols in the range of approximately 0,08 mg/kg to 10 mg/kg of soil.

NOTE Also with this method, other higher methylated phenols, for example, dimethylphenols such as (2,3-, 2,4-, 2,5-, 2,6-, 3,4- and 3,5-)xylenoles, 2-isopropylphenol, 2,3,5-trimethylphenol and 1-naphthol can be analysed, provided the suitability and the validity of the method are proven.

## 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 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

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

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

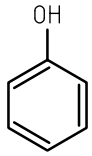
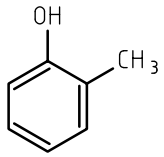
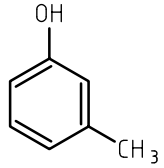
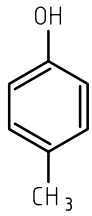
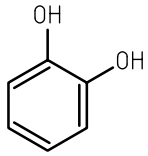
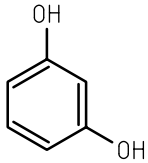
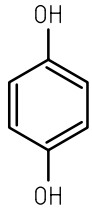
## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

**3.1**
**coal-tar-derived phenols**
monohydroxybenzene, monomethylphenols and dihydroxybenzenes

NOTE See Table 1 for details.

**Table 1 — Target phenolic compounds**

| Compounds | Molecular formula | Molecular mass g/mol | CAS-RN[a] | Chemical structure |
|---|---|---|---|---|
| Phenol monohydroxybenzene | $C_6H_6O$ | 94,11 | 108-95-2 |  |
| *o*-Cresol 2-methylphenol | $C_7H_8O$ | 108,14 | 95-48-7 |  |
| *m*-Cresol 3-methylphenol | $C_7H_8O$ | 108,14 | 108-39-4 |  |
| *p*-Cresol 4-methylphenol | $C_7H_8O$ | 108,14 | 106-44-5 |  |
| *o*-Dihydroxybenzene 1,2-Dihydroxybenzene (catechol) | $C_6H_6O_2$ | 110,10 | 120-80-9 |  |
| *m*-Dihydroxybenzene 1,3-Dihydroxybenzene (resorcinol) | $C_6H_6O_2$ | 110,10 | 123-31-9 |  |
| *p*-Dihydroxybenzene 1,4-Dihydroxybenzene (hydroquinone) | $C_6H_6O_2$ | 110,10 | 108-46-3 |  |

[a] CAS-RN Chemical Abstracts Services-Registry Number.

# 4 Principle

A sample of “as-received” or field-moist soil is extracted with methanol at low pH. Reversed-phase high performance liquid chromatography (HPLC) with direct injection of a definite extract volume and an appropriate detection technique, such as UV/DAD or fluorescence or electrochemical detection, is used to determine the phenolic compound content.

# 5 Interferences

## 5.1 Interference with sampling and extraction

Standard laboratory glassware, appropriately cleaned and free of interfering compounds.

Do not use any kind of plastics containers, since the phenols may be adsorbed onto these. Plastics materials may also contribute their impurities to the sample material.

## 5.2 Interferences with the HPLC

Substances which have the same or nearly the same retention time of the target analytes, and which give UV, fluorescence or electrochemical (amperometric) signals, may interfere with the determination.

# 6 Reagents

## 6.1 General

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

Solvents for HPLC shall be of the purity recommended by the HPLC manufacturer.

Only commercially available certified standards of high purity shall be used.

**WARNING — Phenols are both toxic and corrosive and should be handled with care. Methanol and acetonitrile are toxic and acetic acid is corrosive. Latex or nitrile gloves and eye protection should be worn at all times. Spills should immediately be wiped up with adsorbent tissue and placed in sealed containers used for the disposal of toxic chemicals. Samples should be treated as toxic and harmful.**

## 6.2 Mobile phase for HPLC

**6.2.1 Water,** ultra-pure water is required (HPLC-purity grade).

**6.2.2 Acetonitrile,** $C_2H_3N$, HPLC-purity grade.

**6.2.3 Acetic acid,** $CH_3COOH$, analytical grade.

**6.2.4 Helium,** He**,** of suitable purity for degasification of solvents.

## 6.3 Extraction

**6.3.1 Methanol**, $CH_3OH$, analytical grade.

**6.3.2 Acetic acid,** $CH_3COOH$, analytical grade.

**6.3.3 Acidic methanol,** 0,1 % volume fraction of acetic acid (6.3.2) in methanol (6.3.1), for the preparation of standard solutions and the extraction.

## 6.4 Standards

### 6.4.1 Reference substances (Table 1)

Certified solutions of phenols and single-solid phenol substances with certified purity are available from a limited number of suppliers.

### 6.4.2 Quality-control standard

For example, 4-fluorophenol (CAS 371-41-5).

This quality-control standard shall be used to verify the stability of the detection system, especially for ELCD measurements and also for the recovery check of the extraction. A recovery control check shall be made by addition of a suitable phenolic compound (not mentioned in the scope) to the sample before extraction, which is not interfering with the target analytes, e.g. 4-fluorophenol.

## 6.5 Preparation of standard solutions

### 6.5.1 Preparation of calibration solution of phenols

#### 6.5.1.1 Single-substance stock solutions

Prepare solutions of the single substances (see Table 1) and also the quality-control standard in acidic methanol (6.3.3) with a mass concentration of, for example, 10 mg/ml. These solutions are used for confirmation and identification of single phenols in the chromatogram.

Weigh, for example, 10 mg of single-solid phenol substances into a 10 ml volumetric flask and fill up to the mark with acidic methanol (6.3.3).

#### 6.5.1.2 Multiple-substance stock solution

Prepare the multiple-substance stock solution by mixing appropriate volumes of single-substance stock solutions (6.5.1.1) in acidic methanol (6.3.3).

The solutions 6.5.1.1 to 6.5.1.2 are stable for at least 6 weeks when stored in the dark at 4 °C to 7 °C and protected from evaporation.

#### 6.5.1.3 Calibration solutions

Prepare at least five calibration solutions by appropriate dilution of the multiple-substance stock solution (6.5.1.2), using acidic methanol (6.3.3).

Transfer, for example, 100 µl of the stock solution and a constant volume of quality-control standard solution (6.5.2) into a graduated 10 ml flask and fill up to the mark.

1 µl of the calibration solutions contain 0,5 ng to 5 ng of the respective individual substances.

Check the stability of the calibration solutions regularly.

NOTE Checking the mass concentration of the phenols in the stock solution is only possible by comparison with an independent, preferably certified, standard solution.

#### 6.5.2 Preparation of quality-control standard solution

Weigh 25 mg of 4-fluorophenol in 50 ml of acidic methanol (6.3.3) in a volumetric flask.

The same volume of quality-control standard solution shall be used for calibration and for the samples.

#### 6.5.3 Preparation of mobile phase

Transfer, for example, 1 ml of acetic acid (6.2.3) into a 1 l volumetric flask and fill up to the mark with water (6.2.1).

## 7 Apparatus

Usual laboratory apparatus and in particular the following.

### 7.1 Extraction procedures

Customary laboratory glassware.

All glassware and material that comes into contact with the sample or extract shall be thoroughly cleaned.

**7.1.1 Glass sample bottles**, equipped with a glass stopper or screw cap and a polytetrafluorethene (PTFE) seal. Size in agreement with the amount of sample taken, e.g. 20 ml vials with a screw cap.

**7.1.2 Shaking device**, shaking machine with horizontal movement (200 to 300 strokes/min).

**7.1.3 Pipettes**, of 1 ml and 10 ml (ISO 648, class A), and Pasteur pipettes.

**7.1.4 Volumetric flasks**, of different volumes, e.g. 10 ml, 100 ml or 1 000 ml (ISO 1042, class A).

**7.1.5 Syringes**, 50 µl, 100 µl and 500 µl.

**7.1.6 Luer lock syringe with micro-membrane filter**, e.g. PTFE-membrane, pore size 0,2 µm to 0,45 µm, for filtration of the HPLC extracts.

### 7.2 High performance liquid chromatograph (HPLC)

#### 7.2.1 HPLC system

An HPLC system equipped according to requirements with either an ultraviolet or a fluorimetric or an electrochemical detection system and a data evaluation system, including

— a degassing assembly, e.g. for degassing with vacuum or helium,

— analytical pumps, isocratic or gradient elution,

— a sample injection system,

— a column thermostat, capable of keeping the temperature constant to within ±0,5 °C,

— a fluorescence detector, including damping/amplification, preferably equipped with monochromator(s), or

— a UV detector (with variable wavelength) or diode array, or

— an electrochemical detector for the DC amperometry mode with, for example, an appropriate electrode system.

#### 7.2.2 Analytical separation column

Use a reversed-phase HPLC column meeting the separation requirements given in the experimental conditions (see Annex D for example).

The analytical column can be protected using a guard column. When using guard columns, it is recommended to use column connectors with a low dead volume.

## 8 Sampling and preservation of samples

Take samples in accordance with ISO 10381-1.

Place the samples as received in glass bottles with PTFE caps. It is recommended to fill the bottles completely.

As phenols in soil are subject to biodegradation, the sample containers should be filled on site to preserve the phenols. Bottles containing reagent should be pre-weighed in the laboratory before being filled with sample material and re-weighed on receipt in the laboratory.

Store the samples in the dark in the laboratory at either −18 °C or 4 °C.

Phenols can be subject to microbial conversion under certain conditions. It is recommended that samples be frozen if they are stored for more than 2 days. Samples can be stored for one week at a temperature between 0 °C and <10 °C, and for a year at temperatures below −18 °C.

## 9 Procedure

### 9.1 Blank test

Perform a blank determination using the same amount of reagents that are used for the pre-treatment, extraction and analysis of a soil sample.

### 9.2 Extraction

Weigh 5 g to 20 g (with 2 decimals) of the sample and place it in an extraction bottle (7.1.1), e.g. a screw-cap flask (20 ml to 80 ml) with PTFE seal.

Add a defined volume of the quality-control standard solution (6.5.2), e.g. 100 µl.

Calculate the concentration with the volume of the quality-control standard in the final extract.

Add 20 ml to 80 ml of acidic methanol (6.3.3), close the screw cap and place the flask in a horizontal position on the shaking device (7.1.2).

Shake for at least 30 min with 250 strokes/min. Take an aliquot of the extract and clean up by filtration over a syringe filter (7.1.6) into a vial for analysis. Use PTFE septa for the caps.

### 9.3 Liquid chromatography

#### 9.3.1 General

Adjust the HPLC system according to the manufacturer's instructions.

Regularly check the HPLC with the different modules against the specifications by the manufacturer. If the results of these tests do not meet the specified values, determine and eliminate the reasons.

### 9.3.2 Chromatographic separation

Use a column and chromatographic conditions which allow efficient separation of the phenols stated in the scope.

For a choice of columns and the corresponding settings, see Annex A.

### 9.3.3 Detection

**9.3.3.1** Ultraviolet detectors, fluorescence detectors, electrochemical detectors or a combination of the detectors with ELCD as the last module in the series can be used.

#### 9.3.3.2 Ultraviolet detector/diode array detector (UV/DAD)

Use an appropriate wavelength for the optimal UV/DAD measurements, for example, at 275 nm due to a better specificity.

The absorbance maxima of the target phenols are given in Annex A.

#### 9.3.3.3 Fluorescence detector (FLD)

For an optimal detection, 275 nm for excitation and 312 nm for emission measurements are recommended. See the emission spectra in Annex A.

Dissolved oxygen in the eluent can reduce the fluorescence signal; hence, variations in the oxygen concentrations affect the reproducibility. The oxygen content of the eluent should be kept as low and constant as possible by degassing the eluent using, for example, helium or vacuum.

#### 9.3.3.4 Electrochemical detector (ELCD)

Due to different types and design of the commercially available ELCD, manufacturer's manuals for application, maintenance and especially the regular (cleaning intervals) cleaning of the electrodes are to be strictly followed to have an optimal stage of detection. See the example of the experimental conditions for ELCD in Annex A.

NOTE The optimal detection stage of the electrodes can be checked by the use of the quality-control standard.

### 9.3.4 Identification of individual compounds

An individual compound is assumed to be present if the retention time of the substance in the chromatogram of the sample agrees with the retention time in the chromatogram obtained from a reference substance in a reference solution, measured under the same conditions (tolerance ±1 %, max. 10 s).

### 9.3.5 Calibration

#### 9.3.5.1 General

A distinction is made between initial calibration, working calibration and checking of the validity of the calibration curve. Initial calibration determines the working range and the linearity of the calibration function in accordance with ISO 8466-1. Perform this calibration when the apparatus is used for the first time. In the next step, establish the final working range and perform the routine calibration. Repeat this calibration after maintenance (e.g. replacement of the column), after repair of the HPLC system, and if the system has not been in use for a longer period of time, or if the validity criteria cannot be met. Check the validity of the initial calibration with each series of samples to be analysed.

#### 9.3.5.2 Initial calibration

Establish the preliminary working range by analysing at least five dilutions of the calibration standard mixture (6.5.1.3). Test for linearity in accordance with ISO 8466-1.

#### 9.3.5.3 Routine calibration

After examining the final working range, analyse a minimum of five dilutions of the standard calibration mixture (6.5.1.3). Calculate a calibration function by linear regression analysis of the corrected peak areas. The actual sensitivity of the method may be estimated from the calculated regression function.

#### 9.3.5.4 Check of the validity of the calibration function

Check the validity of the calibration function from the routine calibration with each batch of samples by analysis of one standard solution after every ten samples. The concentration of this standard solution shall be between 40 % and 80 % of the working range. Make sure that the individual results do not deviate by more than 10 % of the working calibration line. If this criterion is met, assume that the calibration is valid. If not, recalibrate in accordance with 9.3.5.3.

#### 9.3.5.5 Measurement of samples

Equilibrate the measuring system before measurement of samples.

NOTE Reproducible retention times are usually achieved after 2 or 3 injections of a reference solution (6.5.1.3).

Measure the sample, the calibration solutions and the blank in the liquid chromatograph.

Ensure that the peaks of each sample are being integrated correctly, and correct if necessary.

If the calculated mass concentration of a substance in the sample exceeds the calibration range, dilute the measuring sample and repeat the measurement.

The measured value of the quality-control standard, which is intended here for recovery check as well as for the checking of optimal detection, should lie in the range between 80 % and 110 %.

## 10 Calculation and expression of results

Assuming that the expected peak area or peak height lies within the linear measuring range, the quantified result of an identified substance, following Equation (1) is:

$$w_n = \frac{A_\mathrm{i} \cdot f_\mathrm{i} \cdot \left[ V_\mathrm{a} + \left( 1 - \frac{w_\mathrm{dm}}{100} \right) \cdot m \right]}{m \cdot \frac{w_\mathrm{dm}}{100}} \qquad (1)$$

where

$w_n$ is the mass fraction of the substance i of a sample, in milligrams per kilogram (mg/kg) (dry soil);

$A_\mathrm{i}$ is the peak area or peak height of substance i in the chromatogram;

$f_\mathrm{i}$ is the response factor of substance i, in counts per microgram per millilitre (µg/ml) (slope of the recalibration curve);

$V_\mathrm{a}$ is the volume of acetic methanol added, in millilitres (ml);

$m$ is the mass content of soil sample (wet soil), in grams (g);

$w_\mathrm{dm}$ is the dry-matter content of the soil sample, determined in accordance with ISO 11465, in percent (%).

The result shall be expressed in milligrams per kilogram of dry soil. For mass fractions less than 1 mg/kg, the results shall be expressed with two figures after the decimal point, and for mass fractions above that, it shall be reported with these significant figures. The dry mass content of soil shall be determined on a separate subsample in accordance with ISO 11465.

EXAMPLE

The mass fraction of individual phenols, in milligrams per kilogram, on the basis of dry matter, rounded off in such a way that no more than two significant values are obtained (e.g. 12 mg/kg; 5,5 mg/kg; 0,36 mg/kg; 0,082 mg/kg; 0,006 9 mg/kg dry matter).

## 11 Test report

The test report shall contain at least the following data:

a) a reference to this International Standard;

b) the information required to identify the sample;

c) the extraction and detection method;

d) the result of the determination in accordance with Clause 10;

e) any details not specified in this International Standard or which are optional, as well as any factor which may have affected the results.

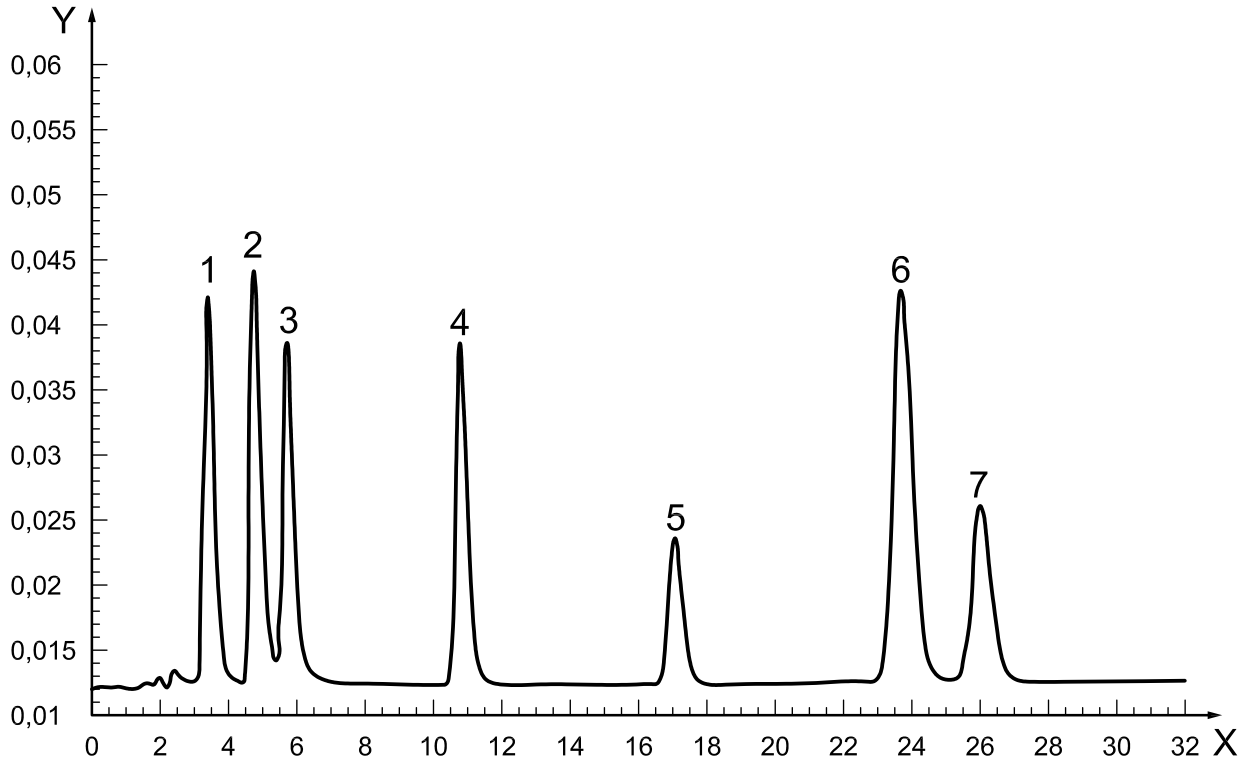
# Annex A
(informative)

# Example of chromatographic conditions and chromatogram

**HPLC conditions for Figures A.1 to A.3.**

| | |
|---|---|
| HPLC system: | Dionex M480-Gradient pump system[1)] |
| Auto-sampler: | Dionex ASI 100[1)] |
| Column: | Acclaim PA, C16, 150 × 2,1 mm, 3 µm (Dionex) |
| Mobile phase: | A: water (6.2.1), B: acetonitrile (6.2.2) |
| Isocratic: | A:B = 90:10 |
| Flow: | 0,30 ml/min |
| Column temperature: | 25 °C |
| Injection volume: | 5 µl calibration solution with 3 µg/ml |
| Detection: | ELCD, FLD and UV/DAD |

1) Dionex is an example of a suitable product. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

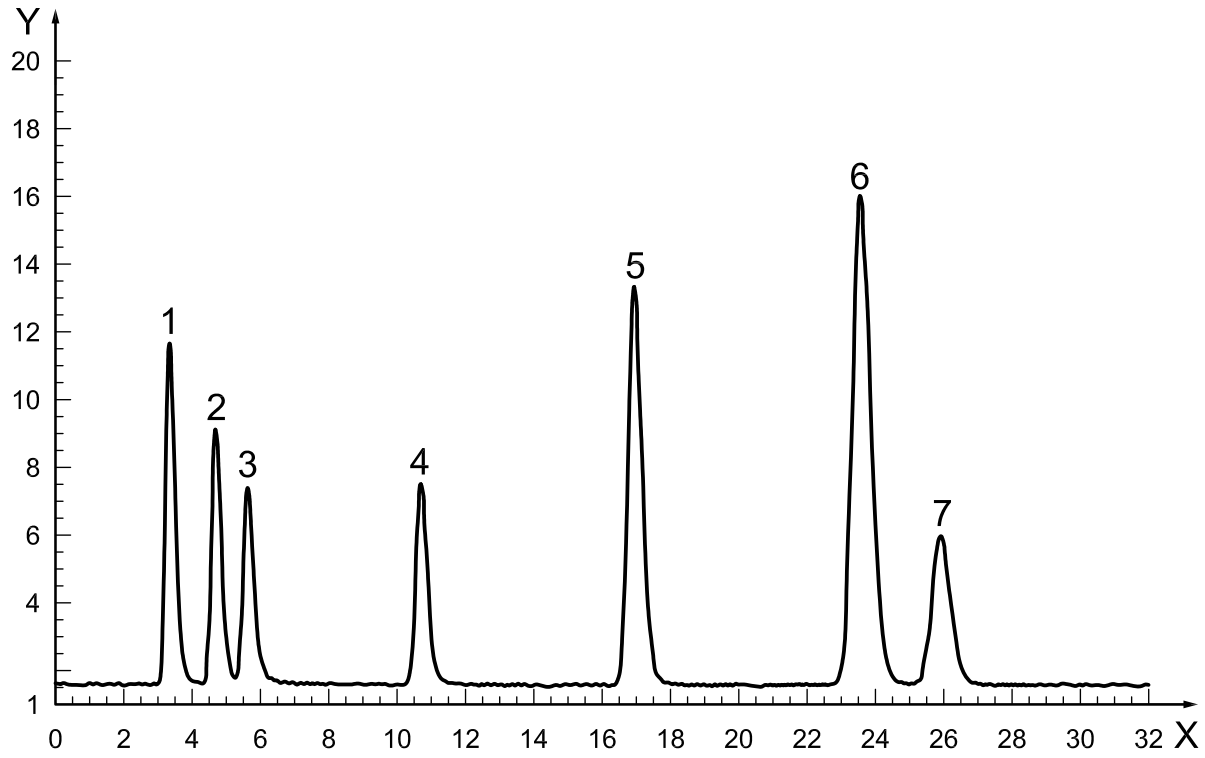



**Key**

X $t$, min
Y STD CAL level 4; DCAmp_1 µA
1 hydroquinone
2 resorcinol
3 catechol
4 phenol
5 4-fluorophenol
6 *m/p*-cresol
7 *o*-cresol

NOTE ELCD system from Dionex ED 50a; working electrode: glassy carbon; reference electrode: Ag/AgCl/3 mol/l KCl; voltage 1,25 V.

**Figure A.1 — Chromatogram with electrochemical detection**



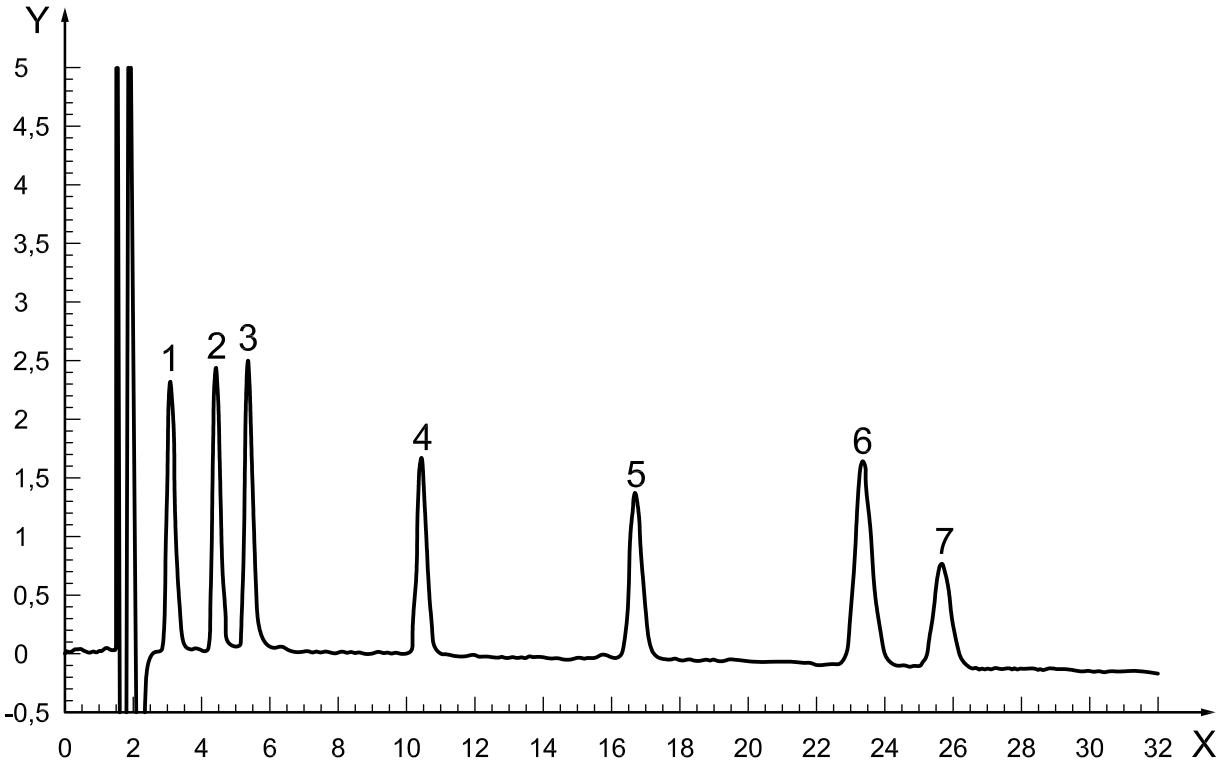

**Key**

X $t$, min
Y STD CAL level 4; emission mAU EM: 312 nm
1 hydroquinone
2 resorcinol
3 catechol
4 phenol
5 4-fluorophenol
6 *m/p*-cresol
7 *o*-cresol

NOTE FLD system from Shimadzu RF-551[2)], fixed band width 15 nm, extinction 275 nm and emission 312 nm.

**Figure A.2 — Chromatogram with fluorescence detection**

2) Shimadzu RF-551 is an example of a suitable product. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.




**Key**

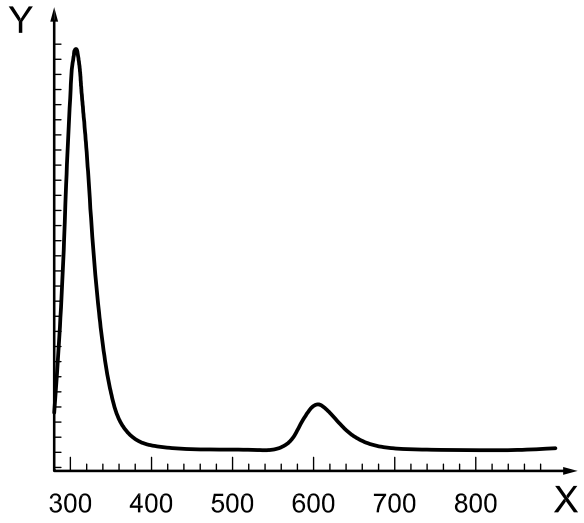
X $t$, min
Y STD CAL level 4; UV VIS 1 mAU; WVL: 275 nm
1 hydroquinone
2 resorcinol
3 catechol
4 phenol
5 4-fluorophenol
6 *m/p*-cresol
7 *o*-cresol

NOTE UV/DAD system from Dionex UVD 320S; 15 µl cell volume, 9 mm path length, 275 nm absorptions wavelength, 12 nm band width.

**Figure A.3 — Chromatogram with diode array detection**

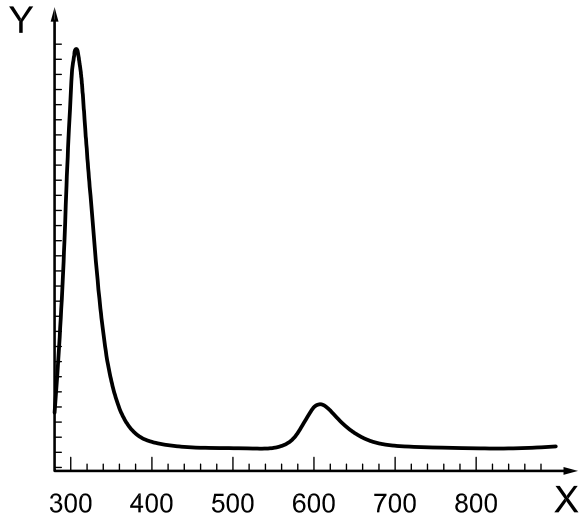
# Annex B
(informative)

## Emission spectrum (FLD, 280 nm to 900 nm) recorded with FLD Agilent 1100[3)]



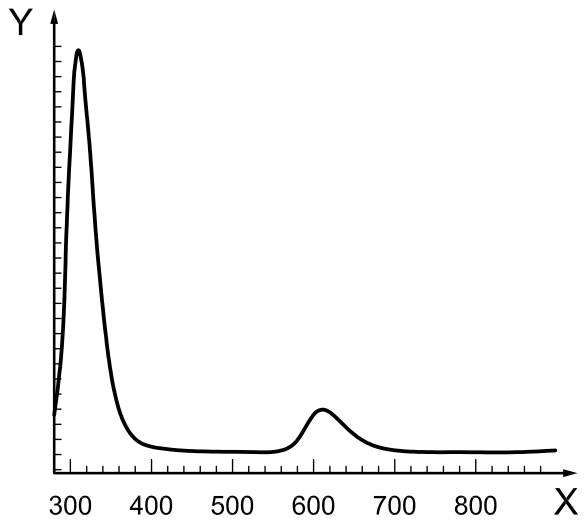

**Key**
X wavelength, in nanometres (nm)
Y response

**Figure B.1 — Phenol**



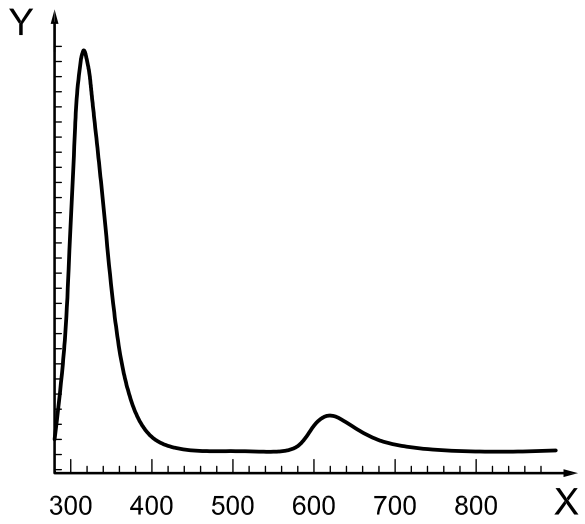

**Key**
X wavelength, in nanometres (nm)
Y response

**Figure B.2 — *o*-Cresol**




**Key**
X wavelength, in nanometres (nm)
Y response

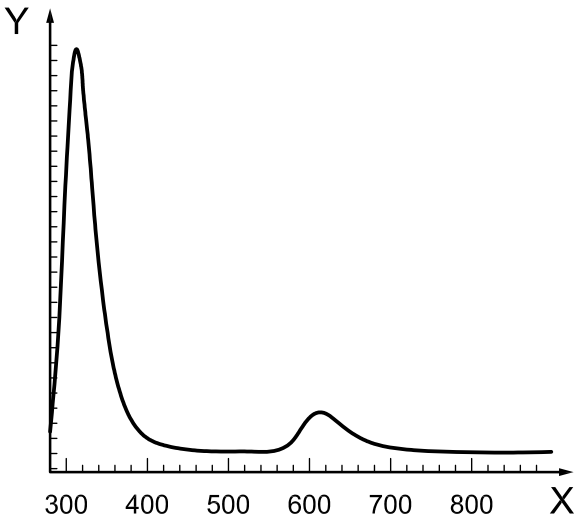
**Figure B.3 — *m/p*-Cresol**




**Key**
X wavelength, in nanometres (nm)
Y response

**Figure B.4 — Catechol**

3) Agilent 1100 is an example of a suitable product. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

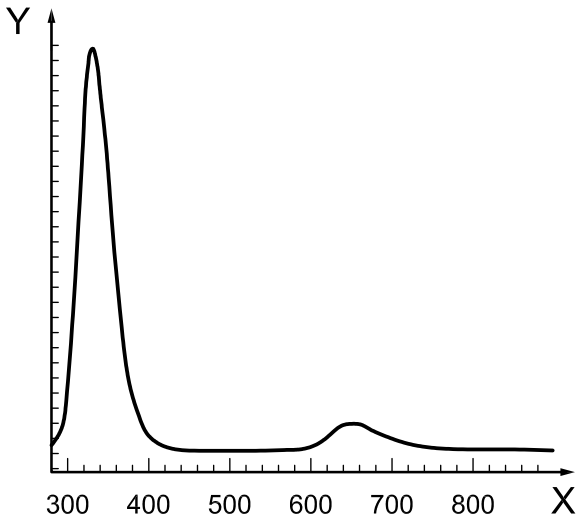



**Key**

X wavelength, in nanometres (nm)

Y response

**Figure B.5 — Resorcinol**




**Key**

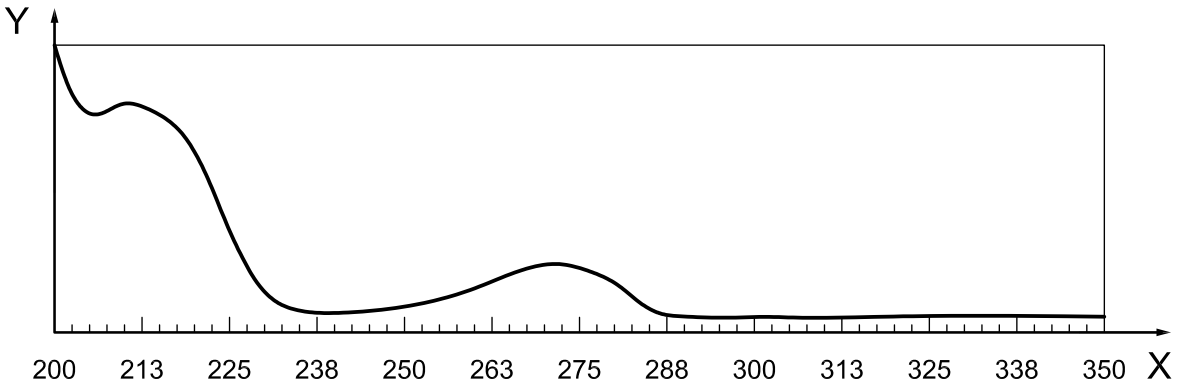
X wavelength, in nanometres (nm)

Y response

**Figure B.6 — Hydroquinone**

# Annex C
(informative)

## Ultraviolet spectrum (DAD, 200 nm to 350 nm), recorded with Dionex UVD 320S[4)]

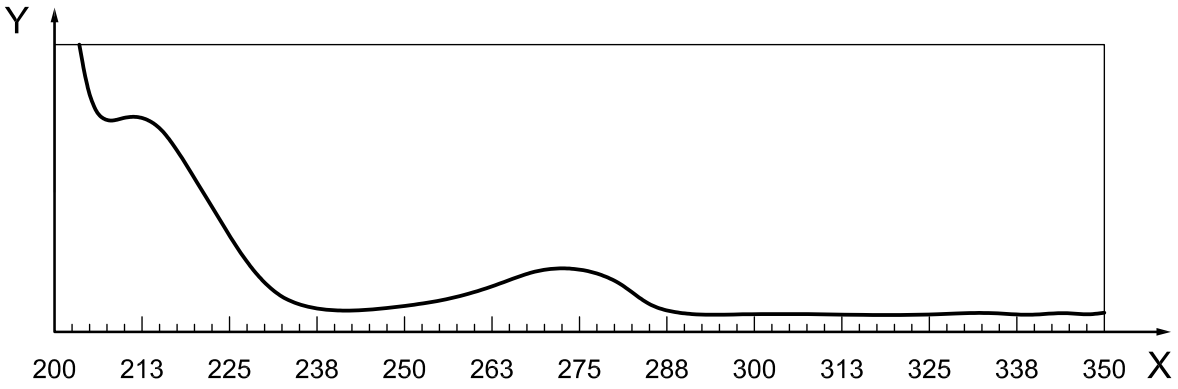



**Key**

X wavelength, in nanometres (nm)

Y response

**Figure C.1 — Phenol (measurement time 10,64 min)**



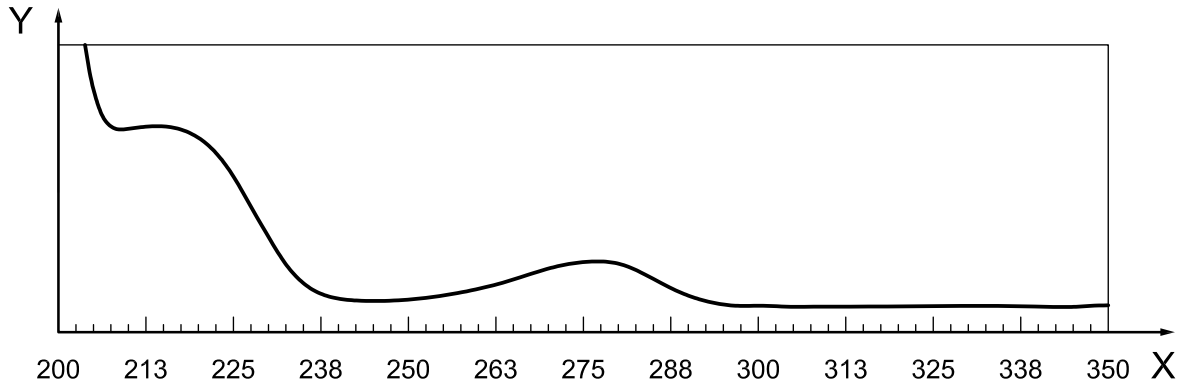

**Key**

X wavelength, in nanometres (nm)

Y response

**Figure C.2 — *o*-Cresol (measurement time 26,68 min)**

4) Dionex is an example of a suitable product. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

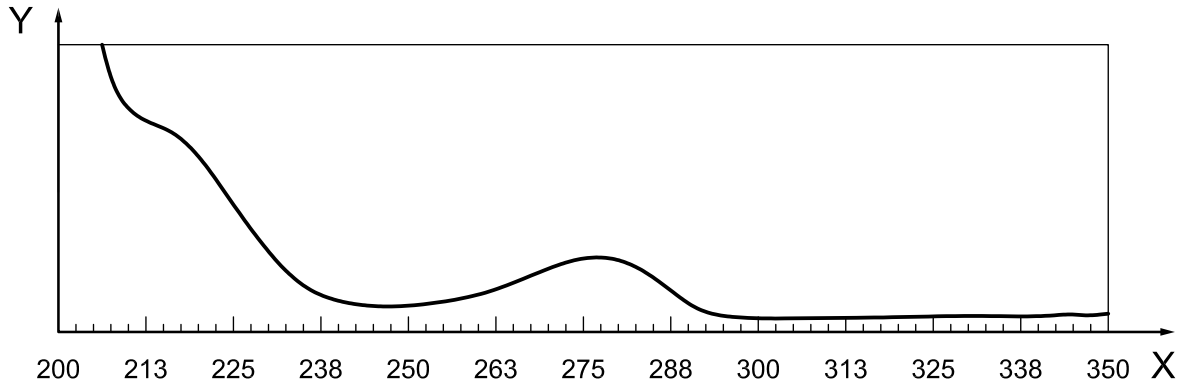



**Key**

X wavelength, in nanometres (nm)

Y response

**Figure C.3 — *m*/*p*-Cresol (measurement time 24,27 min)**

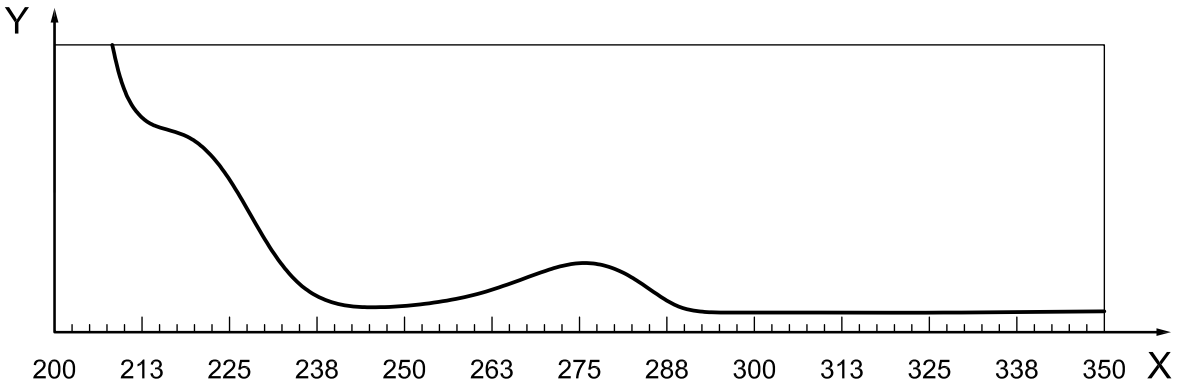



**Key**

X wavelength, in nanometres (nm)

Y response

**Figure C.4 — Catechol (measurement time 5,55 min)**

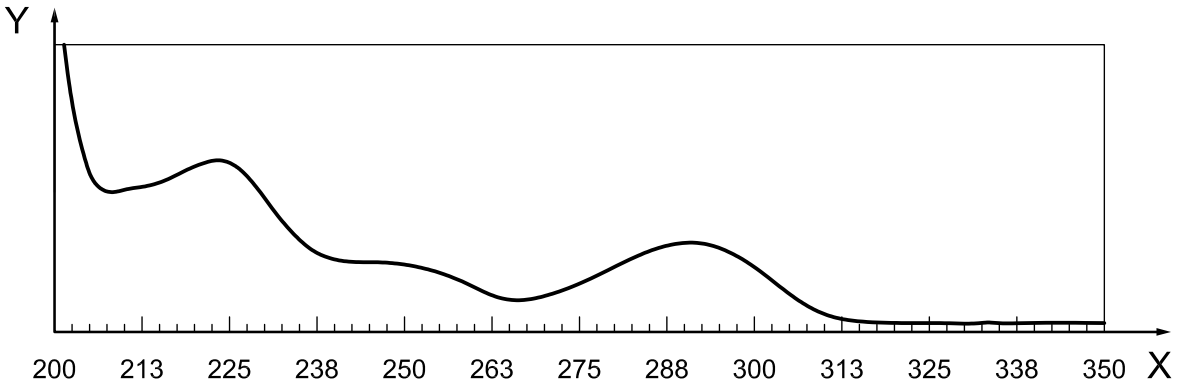



**Key**

X wavelength, in nanometres (nm)

Y response

**Figure C.5 — Resorcinol (measurement time 4,49 min)**




**Key**

X wavelength, in nanometres (nm)

Y response

**Figure C.6 — Hydroquinone (measurement time 3,18 min)**

# Annex D
(informative)

# Examples of limits of determinations of coal-tar-derived phenols for different HPLC detectors

Calculated from the calibration straight line in accordance with DIN 32645.

**Table D.1 — Limits of detections of different phenols in standard solutions (ng/ml) as measured in Figures A.1 to A.3**

| Detector | Phenol | *o*-Cresol | *m*/*p*-Cresol | Catechol | Resorcinol | Hydroquinone |
|---|---|---|---|---|---|---|
| ELCD | 20 | 20 | 30 | 15 | 25 | 25 |
| FLD | 45 | 50 | 50 | 40 | 45 | 40 |
| DAD | 60 | 35 | 50 | 40 | 50 | 50 |

The limit of determination of phenols in soil is dependent upon the intake amount of soil and the extract volume.

EXAMPLE Extraction of 5 g of soil with 20 ml of extraction solvent would give for phenol the following limits of detection:

ELCD: 80 µg/kg

FLD: 180 µg/kg

DAD: 240 µg/kg

# Bibliography

[1] ISO 648, *Laboratory glassware — Single-volume pipettes*

[2] ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

[3] ISO 10381-2, *Soil quality — Sampling — Part 2: Guidance on sampling techniques*

[4] ISO 14154, *Soil quality — Determination of some selected chlorophenols — Gas-chromatographic method with electron-capture detection*

[5] ISO 16072:2002, *Soil quality — Laboratory methods for determination of microbial soil respiration*

[6] DIN 32645, *Chemical analysis — Decision limit, detection limit and determination limit under repeatability conditions — Terms, methods, evaluation*

[7] VDLUFA method — *Determination of phenols in soils, sewage sludges, waste water, compost, vegetable material and water by gas chromatography-mass spectrometry*

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