TECHNICAL SPECIFICATION

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Soil quality — Risk-based petroleum hydrocarbons —

Part 2: Determination of aliphatic and aromatic fractions of semi-volatile petroleum hydrocarbons using gas chromatography with flame ionization detection (GC/FID)

Qualité du sol — Hydrocarbures de pétrole à risque —

Partie 2: Détermination des fractions aliphatiques et aromatiques des hydrocarbures de pétrole semi-volatiles par chromatographie en phase gazeuse avec détection à ionisation de la flamme (CPG-FID)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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

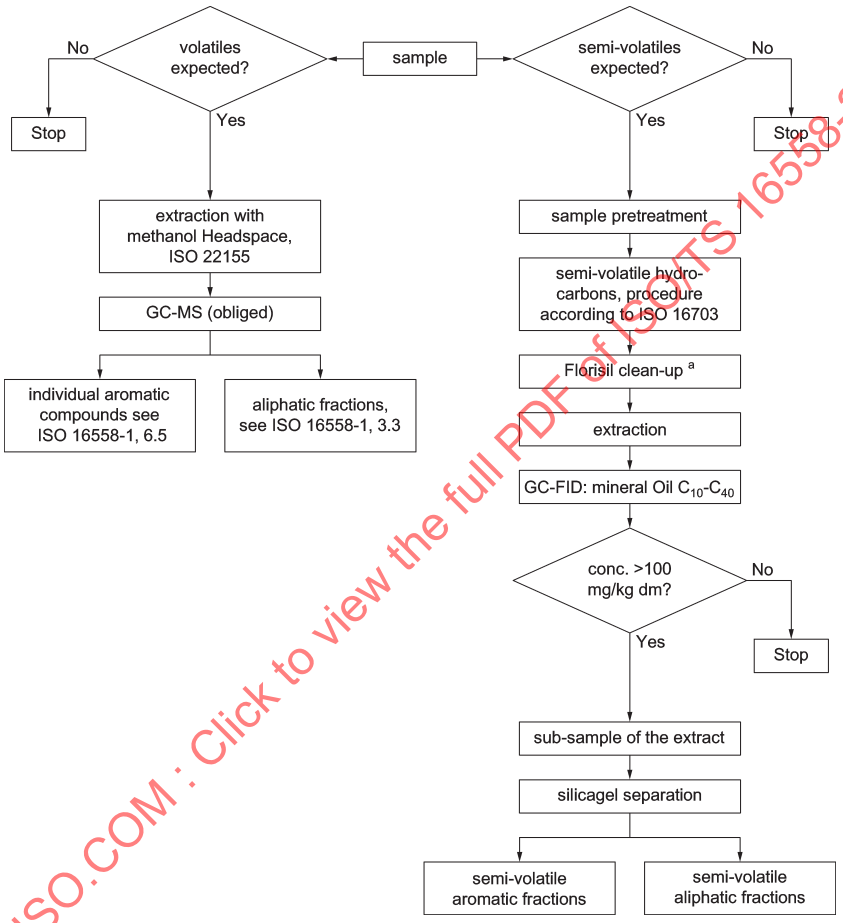
ISO 16558 consists of the following parts, under the general title *Soil quality — Risk-based petroleum hydrocarbons*:

— *Part 1: Determination of aliphatic and aromatic fractions of volatile petroleum hydrocarbons using gas chromatography (static headspace method)*

— *Part 2: Determination of aliphatic and aromatic fractions of semi-volatile petroleum hydrocarbons using gas chromatography with flame ionization detection (GC/FID)* [Technical Specification]

Introduction

ISO 11504 establishes a basis for the choice of fractions and individual compounds when carrying out analysis for petroleum hydrocarbons in soils and soil-like materials including sediments. It provides guidance for the appropriate use of the analytical results in risks assessment. This part of ISO 16558 specifies methods for the quantitative determination of the appropriate fractions of aliphatic and aromatic compounds. The methods described in this part of ISO 16558 are based on existing standards [mineral oil (ISO 16703) and volatile hydrocarbons (ISO 22155)]. The general use and relation between the two different parts of ISO 16558 are given in Figure 1.



Key

a Florisil®[b] clean-up: Only to be applied in case the test according to ISO 16703 is carried out. If the aliphatic and aromatic fractions have to be analysed, Florisil clean-up is not to be carried out. Florisil® is a trade name for a prepared diatomaceous substance, mainly consisting of anhydrous magnesium silicate.

b Florisil® is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 16558 and does not constitute an endorsement by ISO of this product.

Figure 1 — Use of different analytical International Standards during risk assessment of petroleum hydrocarbons

Soil quality — Risk-based petroleum hydrocarbons —

Part 2: Determination of aliphatic and aromatic fractions of semi-volatile petroleum hydrocarbons using gas chromatography with flame ionization detection (GC/FID)

1 Scope

This part of ISO 16558 specifies a method for the quantitative determination of the total extractable semi-volatile, aliphatic, and aromatic fractions of petroleum hydrocarbon content in field moist soil samples by gas chromatography.

The results of the test carried out can be used for risk assessment studies related to contaminations with petroleum hydrocarbons.

This part of ISO 16558 provides a method applicable to petroleum hydrocarbon contents from about 100 mg/kg soil expressed as dry matter for the whole aliphatic fraction C_{10} to C_{40}, as well as the aromatic fraction C_{10} to C_{40}. For sub-fractions, lower limits of determination can be reached.

If lower detection limits are required, large volume injection can be used or concentration of the final extract can be carried out.

NOTE 1 Low concentrations of aliphatic and aromatic compounds can be found in natural uncontaminated organic rich soils like peat soils.

With this method, all hydrocarbons with a boiling range of 174 °C to 525 °C, *n*-alkanes between $C_{10}H_{22}$ to $C_{40}H_{82}$, isoalkanes, cycloalkanes, alkyl benzenes, and alkyl naphthalenes and polycyclic aromatic compounds are determined as total extractable semi-volatile petroleum hydrocarbons C_{10} to C_{40}; besides that, semi-volatile aliphatic and aromatic fractions are specified.

For the determination of volatile aliphatic and aromatic fractions of petroleum hydrocarbons in soil samples, see ISO 16558-1.

NOTE 2 The sub-fractions proposed in this part of ISO 16558 have shown to be suitable for risk assessment studies. However, other sub-fractions between $C_{10}H_{22}$ to $C_{40}H_{82}$ can also be determined in conformity with this part of ISO 16558.

On the basis of the peak pattern of the gas chromatogram and of the boiling points of the individual *n*-alkanes listed in Annex B, the approximate boiling range of the mineral oil and some qualitative information on the composition of the contamination can be achieved.

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

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 14507, *Soil quality — Pretreatment of samples for determination of organic contaminants*

ISO 16703, *Soil quality — Determination of content of hydrocarbon in the range C10 to C40 by gas chromatography*

ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

3 Terms and definitions

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

3.1
total extractable semi-volatile petroleum hydrocarbon content by gas chromatography
sum of compounds extractable with acetone/*n*-heptane (2+1) that can be detected with a flame ionization detector and chromatographed on a non-polar capillary column with retention times between those of *n*-decane ($C_{10}H_{22}$) and *n*-tetracontane ($C_{40}H_{82}$)

Note 1 to entry: Substances that comply with that definition are mainly long chain or branched aliphatic, alicyclic, lower polycyclic, or alkyl substituted aromatic hydrocarbons.

3.2
semi-volatile aliphatic fraction of petroleum hydrocarbons
fraction of the total semi-volatile petroleum hydrocarbons which are eluted with pentane, hexane, or heptane after adsorption on silicagel

Note 1 to entry: This is a method defined in this part of ISO 16558. It is unknown and unpredictable if the same compounds will elute from the silicagel with other solvents.

3.3
semi-volatile aromatic fraction of petroleum hydrocarbons
fraction of the total semi-volatile petroleum hydrocarbons which are eluted with dichloromethane or with a 1:1 mixture of dichloromethane and *n*-heptane after adsorption on silicagel

Note 1 to entry: This is a method defined in this part of ISO 16558. It is unknown and unpredictable if the same compounds will elute from the silicagel with other solvents.

4 Interferences

Compounds not related to petroleum hydrocarbon contaminations with a boiling point between C_{10} and C_{40} (e.g. halogenated hydrocarbons) might interfere with the determination.

5 Principle

A known amount of the homogenized soil sample is extracted by mechanical shaking or sonication with acetone/*n*-heptane. The organic layer is separated and washed twice with water. An aliquot of the extract is analysed by capillary gas chromatography with flame ionization detection. The total peak area between the range defining standards *n*-decane and *n*-tetracontane is measured as the amount of total extractable semi-volatile petroleum. The extract is split into two fractions containing, respectively, the aliphatic and aromatic hydrocarbons using a column containing silicagel. The fractions are also analysed by gas chromatography.

Instead of *n*-heptane, another single hydrocarbon solvent or technical mixture of hydrocarbons, boiling range 36 °C to 99 °C, can be used.

6 Reagents

All reagents shall be of recognized analytical grade. Verify whether the reagents are applicable for this specific purpose and free of interfering compounds.

6.1 Acetone, $(CH_3)_2CO$ (CAS-RN[1)] 67-64-1).

6.2 *n*-Heptane, C_7H_{16} (CAS-RN 142-82-5).

Instead of *n*-heptane, another single hydrocarbon solvent or technical mixture of hydrocarbons, boiling range 36 °C to 99 °C, may be used.

6.3 Dichloromethane, CH_2Cl_2 (CAS-RN 75-09-2).

6.4 Silicagel, particle size 63 µm to 200 µm (70 mesh to 230 mesh) heated for at least 16 h at 140 °C and stored in a desiccator over a molecular sieve.

6.5 Anhydrous sodium sulfate, Na_2SO_4, heated for at least 2 h at 550 °C.

6.6 Retention time window (RTW) standard solution, is the range defining standard solution containing *n*-tetracontane and *n*-decane.

Weigh (30 ± 1) mg of *n*-tetracontane into a 1 l volumetric flask, dissolve completely in an appropriate volume of *n*-heptane (6.2), add 30 µl of *n*-decane (about 21 mg), mix well, fill up to volume with *n*-heptane and homogenize. This solution shall be used for all dilution steps of the hydrocarbon standard (6.7) and be stored at room temperature.

NOTE *n*-tetracontane is only moderately soluble in *n*-heptane. Slight warming and/or sonication accelerates the dissolution process.

6.7 Hydrocarbon standard solution for calibration.

Mix approximately equal masses of two different types of mineral oil. Weigh accurately this mixture and dissolve in the RTW standard solution (6.6) to give a hydrocarbon content of about 8 g/l.

Preparation of the calibration solutions can be done by diluting an aliquot of this standard solution (6.7) with the internal standard solution (6.6).

The first oil type should show discrete peaks (e.g. a diesel fuel) in the gas chromatogram, as can be seen in Figure A.1 (left part of the chromatogram). The second type should have a boiling range higher than the first one and should show a hump in the gas chromatogram, as can be seen in Figure A.1 (right part of the chromatogram). A suitable oil of this type is, for example, a lubricating oil without any additives.

NOTE General purpose hydrocarbon standards for calibration can be obtained from many commercial organizations. Calibration standards specific to this part of ISO 16558 can be purchased e.g. from Bundesanstalt für Materialforschung und -prüfung, Fachgruppe I.2, Richard-Willstätter-Strasse 11 D-12489 Berlin, Germany, or VSL BV, Thijsseweg 11, 2600 Delft, Netherlands (product RIVM-NMi-001). This information is given for the convenience of users of this part of ISO 16558 and does not constitute an endorsement by ISO of this product.

6.8 Control solution.

Prepare an independent control solution according to 6.7 with a hydrocarbon concentration of about in the middle of the working range System performance standard solution.

1) CAS-RN: Chemical Abstracts Service Registry Number.

6.9 Retention time standard solution.

Prepare a mixture of equal amounts, on a mass basis, of the *n*-alkanes with carbon numbers from C_{10} to C_{40}, dissolved in *n*-heptane (6.2), to give concentrations of about 50 mg/l of each *n*-alkane. Store at room temperature.

NOTE 1 This solution is also used to verify the suitability of the gas chromatographic system for the resolution of *n*-alkanes, as well as for the detector response.

NOTE 2 This solution is used to give information of the retention times of the *n*-alkanes to characterize the hydrocarbons in the samples.

6.10 Preparation of the silicagel column.

Push a plug of pre-washed glass wool or a PTFE frit down into the column (7.10). Add successively 3 g of silicagel (6.4) and 2 g sodium sulfate (6.5). Prepare the column immediately before use.

NOTE Commercially available cartridges are also applicable.

6.11 Control solution silicagel column efficiency.

6.11.1 Control solution aliphatic split.

Prepare a mixture of equal amounts, on a mass basis, of the *n*-alkanes with carbon numbers from C_{10} to C_{40}, dissolved in *n*-heptane (6.2), to give concentrations of about 50 mg/l of each *n*-alkane. Store at room temperature.

6.11.2 Control solution aromatic split.

Prepare a mixture of equal amounts, on a mass basis, polycyclic aromatic hydrocarbons containing 16 PAH according to EPA in *n*-heptane (6.2), to give mass concentrations of about 50 mg/l of each compound. Store at room temperature.

7 Apparatus

7.1 Usual laboratory glassware, which shall be heated 2 h in an oven at 200 °C to 300 °C and after cooling, rinsed with acetone (6.1) and dried before use.

The cleaning procedure of the glassware can be replaced by any method if it shows (e.g. by blank samples) that the glassware does not give a positive contribution to the concentration of the compounds of interest in this part of ISO 16558.

7.2 Devices for extraction.

Mechanical shaker, horizontal movement, at least 120 shaking movements per minute. Alternatively, an ultrasonic bath can be used.

7.3 Laboratory centrifuge, capable of producing an acceleration of at least 1 500 *g*.

7.4 Gas chromatograph, equipped with a non-discriminating injection system (preferably on-column or programmable temperature vaporization injection-PTV), a capillary column, and a flame ionization detector (FID).

NOTE The use of a large volume injection system can improve the limit of detection considerably.

7.5 Capillary column, a fused silica capillary column with one of the following stationary phases and dimensions:

— stationary phase: non-polar, e.g. immobilized 100 % dimethyl polysiloxane, 95 %-dimethyl-5 %-diphenyl polysiloxane, modified siloxane polymer;

— length: 10 m to 25 m;

— internal diameter: 0,1 mm to 0,32 mm;

— film thickness: 0,1 µm to 1,0 µm.

The column shall give a baseline separation for the *n*-alkanes in the system performance standard solution (6.7).

Thermally stable low bleed columns are preferred.

The use of a pre-column, e.g. wide-bore (0,53 mm internal diameter) deactivated fused silica of at least 2 m of length that suits to the analytical column and connected to it using zero-volume connector, is recommended.

7.6 Data system, capable of integrating the total area of the chromatogram, compensating for column bleed, and reintegrating after defining a new baseline.

7.7 Glass extraction vessel, with a volume of at least 100 ml, with screw caps provided with an inlay of polytetrafluoroethylene (PTFE).

7.8 Glass tube, 25 ml with a ground-glass stopper or screw caps provided with an inlay of polytetrafluoroethylene (PTFE).

7.9 Separatory funnel, at least 500 ml, with a ground glass stopper.

7.10 Chromatography column for split into aliphatic and aromatic fractions, glass columns of about 10 mm internal diameter shall be used.

The upper part of the column should be widened to use as solvent reservoir and the lower part to be narrowed to form a tip.

8 Sampling, sample conservation, and pretreatment

Sampling shall be carried out according to ISO 10381-1 and in coordination with the analytical laboratory.

For preservation and holding times of the samples, see ISO 18512.

If this is not possible, samples shall be stored at −18 °C or lower. Before analysis, the samples shall be pretreated according to ISO 14507 or other suitable pretreatment methods.

9 Procedure

9.1 Blank

With each series of samples, a blank determination shall be carried out according to 9.2 using all reagents in identical amounts but without a sample. If blank values are unusually high (more than 10 % of the lowest value of interest), every step in the procedure shall be checked to find the reason for these high blanks.

9.2 Extraction

9.2.1 Total petroleum hydrocarbons

Weigh exactly about 20 g of the pretreated soil sample according to ISO 14507 into a glass extraction vessel (7.7) and add (40 ± 1) ml of acetone (6.1). After short shaking by hand, add (20 ± 0,1) ml of the RTW standard solution (6.6). Close the vessel and extract the sample for minimal 1 h by mechanical shaking or sonication (7.2). After settling of the solid material, transfer as much as possible of the supernatant into a separatory funnel (7.9). To remove the acetone, wash the organic phase twice by shaking thoroughly (5 min) with 100 ml of water. Collect the organic layer in a glass tube (7.8). Add sufficient amount of sodium sulfate so that no lumps are formed anymore. Transfer an aliquot of the purified extract to a GC-vial and analyse by gas chromatography.

The result of this analysis is the content of total petroleum hydrocarbons.

If appropriate, test portions of 5 g to 30 g can be used (e.g. smaller test portion should be used if samples adsorb the major portion of the extraction solvent added, sample intake should be increased if high sensitivity is required).

9.2.2 Split into aliphatic and aromatic fractions

9.2.2.1 General

In case of a positive result (depending on the requirements of the project) of the total extractable semi-volatile petroleum hydrocarbons, the (non-purified) extract has to be split into a fraction containing the aliphatic and a fraction containing the aromatic compounds (see 9.2.2.2 and 9.2.2.3).

NOTE As the fractions of interest for this standard comprise unknown individual compounds and different adsorption materials and different solvents used for the elution might lead to (unpredictable) different recoveries of the fractions, the procedures described in 9.2.2.2 and 9.2.2.3 are method defined.

9.2.2.2 Semi-volatile aliphatic fractions

Pre-rinse the silicagel column (6.10) successively with 10 ml dichloromethane and 10 ml *n*-heptane. Transfer 1,0 ml of the sample extract on to the column and elute the column. Elute the column successively two times with 1 ml of *n*-heptane (or used extraction solvent). Elute the column with three portions of 4 ml *n*-heptane (or used extraction solvent) and collect the entire eluate containing the aliphatic fraction.

It is important to elute the column using the mentioned portions. Fewer portions with larger volumes can lead to insufficient elution of the aliphatics and contaminate the aromatic fraction with the aliphatics.

Concentrate the extracts, if necessary, under a gentle stream of nitrogen.

9.2.2.3 Semi-volatile aromatic fractions

Elute the column with three portions of 4 ml dichloromethane or three portions of 4 ml of a mixture of dichloromethane:*n*-heptane (1:1) and collect the entire eluate containing the aromatic fraction. Do not allow the solvents to fall below the top of the column packing during all elution steps.

NOTE Experiments have shown that dichloromethane and a 1:1 mixture of dichloromethane:*n*-heptane give comparable results (see Annex C). The use of *n*-heptane might be helpful as a keeper in circumstances where the lab temperature is too high to use pure dichloromethane. The use of only dichloromethane, however, is recommended as the use of a mixture of *n*-heptane:dichloromethane can lead to gaschromatographic problems (shift in retention times). This problem does not arise if the *n*-heptane:dichloromethane is concentrated by evaporation, in which case, the dichloromethane will be removed largely.

Concentrate the extracts, if necessary, under a gentle stream of nitrogen.

9.3 Determination by gas chromatography

9.3.1 Test of the performance of the gas chromatographic system

Use a capillary column with one of the specified stationary phases (7.5) for gas chromatographic analysis. Adjust the gas chromatograph (7.4) to provide an optimal separation. The *n*-alkanes in the retention time standard solution (6.9) shall be baseline separated. The relative response of the *n*-tetracontane (C_{40}) shall be at least 0,8, with respect to *n*-eicosane (C_{20}). Use the retention times of the *n*-alkanes to define the window of the different fraction according to 9.3.6.

For an example of gas chromatographic conditions, see Annex A.

9.3.2 Repeatability test

Record a gas chromatogram of the column bleed by injection of an appropriate volume of *n*-heptane. Then inject the same volume of a suitable concentration of the control solution (6.8) three times and record the chromatogram for each injection. Integrate the chromatograms according to 9.3.6 and calculate the mean of the measured peak areas and the corresponding standard deviation. The relative standard deviation shall not be greater than 5 %.

9.3.3 Calibration

When the method is used for the first time and/or when the apparatus or operator is changed, a basic calibration according to ISO 8466-1 including the determination of the limit of detection and limit of determination shall be carried out.

An external calibration is performed by analysing a minimum of five dilutions of hydrocarbon standard solution (6.9) which should cover the working range. Calculate a calibration function by linear regression analysis of the corrected peak areas. A chromatogram of *n*-heptane is used to correct the peak area of the chromatograms of the hydrocarbon standard solutions for the column bleed. From the calculated regression line, the actual sensitivity of the method is determined.

9.3.4 Validity check of the calibration function

The validity of the calibration function shall be checked within each batch of samples by analysis of one independent control solution (6.8). The validity check identifies problems of calibration before real samples are run. Check whether the result is within ±10 % of the reference value of the control solution. If this is the case, the actual calibration function is assumed to be valid. If not, a new calibration according to 9.3.3 shall be performed.

NOTE It is good analytical practice to perform both a calibration check and analytical quality control using an independent solution randomly placed during the analysis of the batch of samples. This independent solution can perform both functions.

9.3.5 Measurement

Analyse the blank (9.1) and the sample extracts (9.2), calibration standards (6.7) and control solutions (6.8), retention time standard solution (6.9) under identical gas chromatographic conditions.

n-heptane shall be analysed in each sample batch. The resulting chromatogram is used to correct chromatograms of blanks (9.1), sample extracts (9.2), calibration standards (6.7), and control solutions (6.8) for column bleed prior to integration.

9.3.6 Integration

Integrate the total area between the *n*-decane (C_{10}) and *n*-tetracontane (C_{40}) peaks of the chromatogram. Start the integration at the retention time just after the end of the *n*-decane-peak at the signal level in front of the solvent peak. End the integration of the total area at the retention time just before the

beginning of the *n*-tetracontane peak at the same signal level (see Annex A). Integrate *n*-tetracontane (C_{40}) as a separate peak for the recovery check.

Integrate the area of the following fractions: > C_{10} to C_{12}, > C_{12} to C_{16}, > C_{16} to C_{21}, > C_{21} to C_{35}, > C_{35} to < C_{40}.

The presence of peaks on the tail of the solvent peak with retention times less than that of *n*-decane indicates that the sample contains low boiling volatile hydrocarbons. This should be mentioned in the test report and ISO 16558-1 has to be applied.

A non-horizontal baseline at the end of the chromatogram (retention time greater than that of *n*-tetracontane) with a signal level greater than the bleed indicates that the sample contains high-boiling hydrocarbons with more than 40 carbon atoms. This should be mentioned in the test report. It should be ensured that these compounds elute completely from the column. Otherwise, they can cause interferences with the subsequent sample analysis.

All chromatograms should be checked visually for correct integration. The start and stop times of the integration should be visible on the chromatogram.

The range of the carbon numbers of *n*-alkanes present in the sample is determined by comparing the gas chromatogram of the sample extract with that of the integration time standard solution (6.9). The corresponding boiling range can be derived from Annex B.

NOTE Peak shape and signal intensity of *n*-tetracontane are sensitive to changes in the surface properties of the injector and/or the pre-column due to contamination by sample constituents. Therefore, they can be used as a good indication for replacing pre-column and/or liner.

9.3.7 Calculation of the total petroleum hydrocarbons

Calculate the total extractable semi-volatile petroleum hydrocarbon content of the soil sample using Formula 1:

$$c = c_s \cdot \frac{V_h}{M} \cdot f \cdot \frac{100}{d_m} \cdot \frac{1}{p} \quad (1)$$

with

$$c_s = \frac{A_s - b}{a} \quad (2)$$

where

c is the hydrocarbon content of the sample, in milligrams per kilogram dry matter (mg/kg dm);

c_s is the hydrocarbon content of the extract calculated from the calibration function, in milligrams per litre (mg/l);

V_h is the volume of the *n*-heptane extract, in millilitres (ml);

f is the dilution factor (when the extract is diluted);

p is the fraction of the soil extract used for analysis;

M is the mass of the sample taken for analysis, in grams (g);

d_m is the dry matter content, expressed as percentage, %, by weight, according to ISO 11465;

A_s is the integrated peak area of the sample extract, expressed in instrument dependent units;

b is the intercept of the ordinate, expressed in instrument dependent units;

a is the slope of the calibration function, expressed in litres per milligram (l/mg).

Round off the result to two significant figures.

9.3.8 Calculation of the individual extractable fractions

Calculate, using the same procedure as described in 9.3.7, the concentrations of the next fractions: > C_{10} to C_{12}, > C_{12} to C_{16}, > C_{16} to C_{21}, > C_{21} to C_{35}, > C_{35} to < C_{40} of both the aliphatic and aromatic fractions. Use for the calculation of each fraction the integrated peak area of that fraction (A_s).

Note that C_{10} is not included in the first fraction, as this is included in the last fraction of the volatile aliphatic compounds (see ISO 16558-1). C_{40} is not included in the last fraction.

9.4 Quality control

9.4.1 Suitability check of the split procedure

The split efficiency of each batch of silica shall be checked (if commercial columns are used, their suitability for the clean-up procedure shall be checked in the same way) by the following procedure.

Bring 1,0 ml of the control solutions 6.11.1 and 6.11.2 on a separate silicagel column and proceed according to the procedure described in 9.2.2. Analyse the fractions together with the non treated control solutions. The aliphatic fraction shall contain at least 80 % *n*-alkanes. The aromatic fraction shall contain at least 80 % PAH.

10 Expression of results

Report results in milligrams of aromatic and aliphatic fraction per kilogram of dry soil and up to two significant figures.

11 Test report

The test report shall include at least the following information:

a) a reference to this part of ISO 16558, i.e. ISO/TS 16558-2;

b) a complete identification of the sample;

c) a reference to the method used for extraction (shaking or sonication or other) and clean-up;

d) the results of the determination according to Clause 10;

e) any details not specified in this part of ISO 16558 or which are optional, as well as any other factor which might have affected the results.

Annex A
(informative)

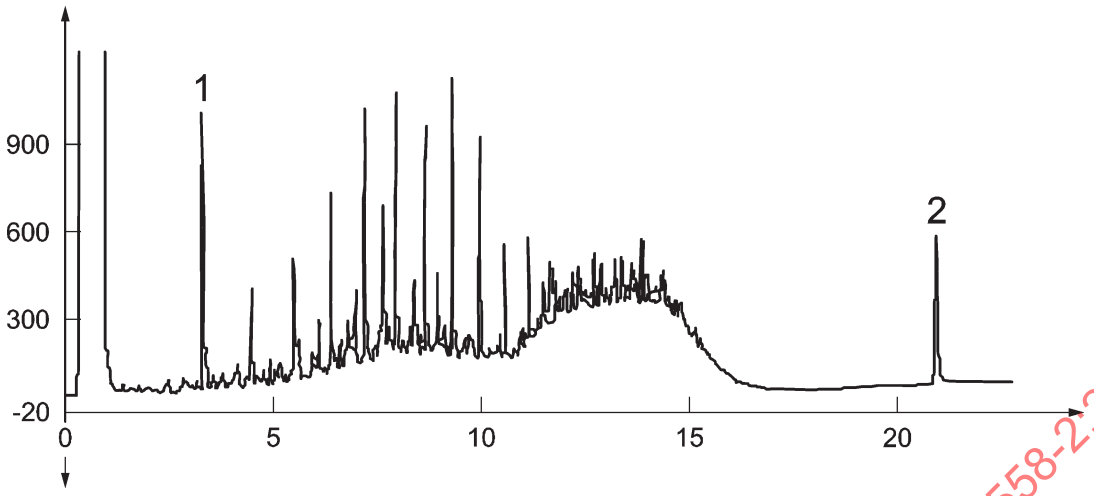
Examples of gas chromatograms of total extractable petroleum hydrocarbon and aliphatic and aromatic fractions in a standard solution and in soil samples

Figure A.1 shows the gas chromatogram of the calibration mixture of mineral oil consisting of equal parts of a diesel fuel and a lubricating oil. Figure A.2 shows the gas chromatogram of the "column bleed" after injection of *n*-heptane and Figure A.3 shows the integrated gas chromatogram of the calibration mixture of mineral oil corrected for the "column bleed". The total peak area between *n*-decane (C_{10}) and *n*-tetracontane (C_{40}) used for quantification is indicated as hatched area.

The gas chromatograms have been recorded under the following conditions:

Injection technique:	on-column
Injection volume:	1 µl to 3 µl
Column type:	WCOT fused silica
Column length:	12 m
Internal diameter:	0,32 mm
Liquid phase:	BPX-5
Film thickness:	1,0 µm
Precolumn:	deactivated fused silica capillary,
	2 m x 0,53 mm
Carrier gas:	Helium
Pressure:	100 kPa
Detector:	Flame ionization detector
Detector Temperature:	360 °C
Temperature program:	80 °C for 1 min;
	20 °C/min to 360 °C;
	360 °C for 15 min

NOTE "WCOT fused silica" and BPX5 are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 16558 and does not constitute an endorsement by ISO of this product.



Key

1 internal standard *n*-decane (C_{10})

2 internal standard *n*-tetracontane (C_{40})

Figure A.1 — Example of a gas chromatogram of the calibration mixture consisting of equal parts of diesel fuel and lubricating oil

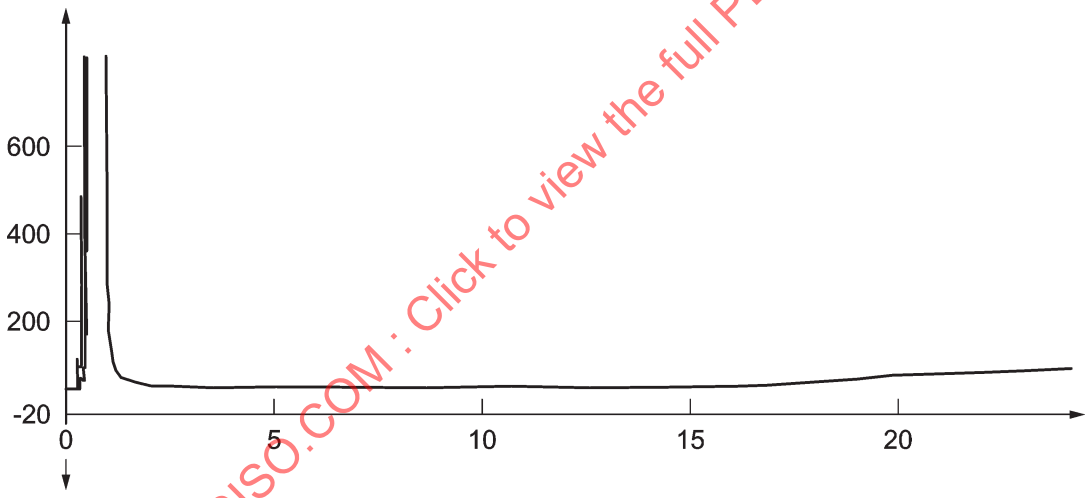
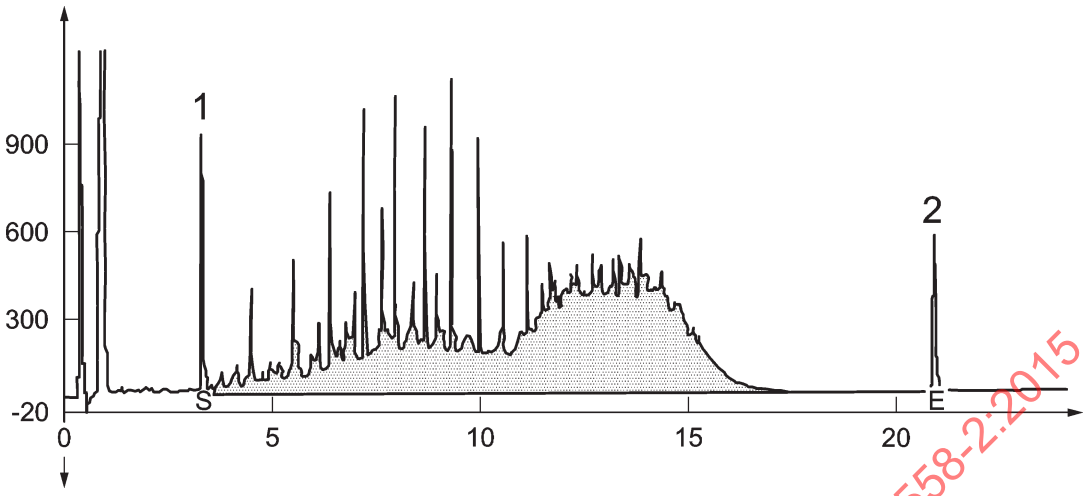


Figure A.2 — Example of a gas chromatogram of the "column bleed"



Key

1 internal standard *n*-decane (C_{10})

2 internal standard *n*-tetracontane (C_{40})

Figure A.3 — Example of an integrated gas chromatogram of the calibration mixture of mineral oil corrected for the "column bleed"

Annex B
(informative)

Determination of the boiling range of mineral oil hydrocarbons from the gas chromatogram

Using the data from Table B.1, the approximate boiling range of the hydrocarbons in the sample can be estimated by comparison of the peak pattern of the sample chromatogram and that of the *n*-alkane mixture.

Table B.1 — Boiling points of the *n*-alkanes with from 6 to 44 carbon atoms

Number of carbon atoms	Boiling point °C
6	69
7	98
8	126
9	151
10	174
11	196
12	216
13	235
14	253
15	271
16	287
17	302
18	317
19	331
20	344
21	356
22	369
23	380
24	391
25	402
26	412
27	422
28	432
29	441
30	450
31	459
32	468
33	476
34	483

Table B.1 *(continued)*

Number of carbon atoms	Boiling point °C
35	491
36	498
37	505
38	512
39	518
40	525
41	531
42	537
43	543
44	548

Annex C
(informative)

Information on split of aliphatic and aromatic fractions using silicagel

This annex provides some background information on the split of the aliphatic and aromatic fractions with the use of silicagel.

Figure C.1 shows the GC-FID chromatogram of a mixture of *n*-alkanes and PAH. Figures C.2 and C.3 show, respectively, the aliphatic and aromatic fractions after silicagel split, as described in this part of ISO 16558. Table C.1 shows the recovery (%) of the individual *n*-alkanes in the aliphatic and the individual PAH compounds in the aromatic fractions.

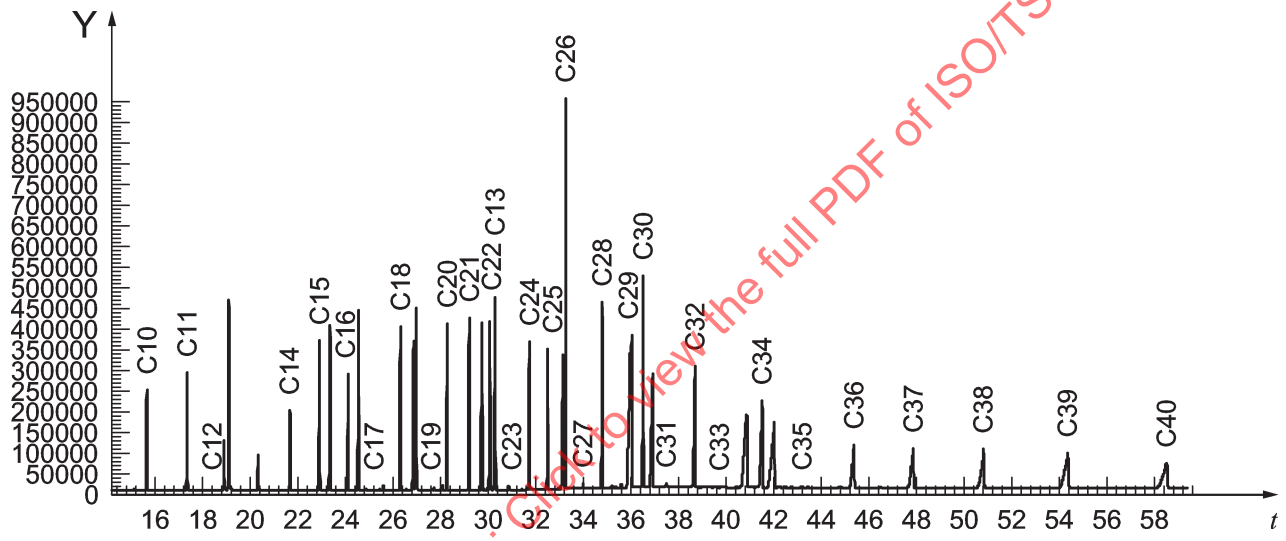


Figure C.1 — Example of a chromatogram of a mixture of n-alkanes/16 EPA PAH before silicagel cleanup

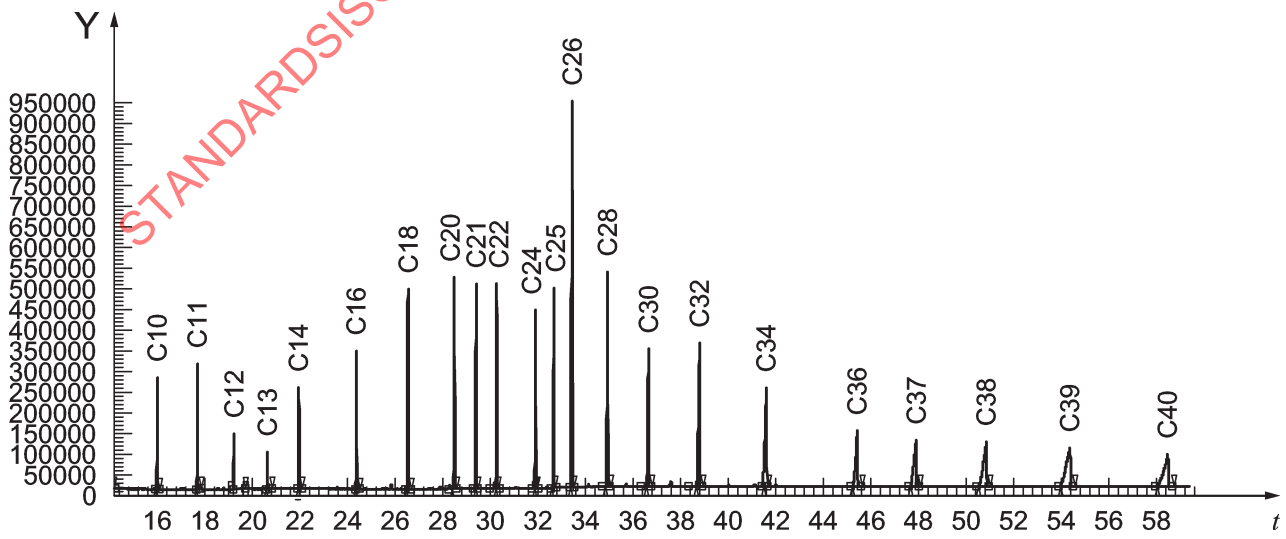


Figure C.2 — Example of a chromatogram of *n*-alkanes in aliphatic fraction after silicagel cleanup