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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](http://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](http://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](#)

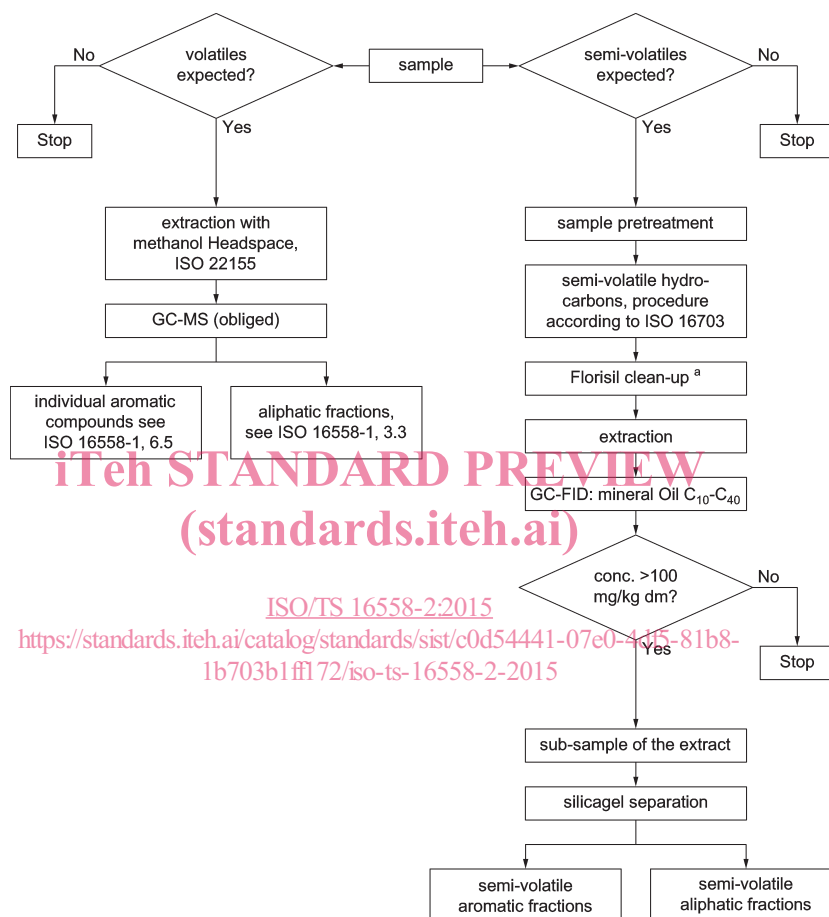
The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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

- Florisol<sup>®</sup> 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, Florisol clean-up is not to be carried out. Florisol<sup>®</sup> is a trade name for a prepared diatomaceous substance, mainly consisting of anhydrous magnesium silicate.
- Florisol<sup>®</sup> 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**

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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)

## 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<sub>10</sub> to C<sub>40</sub>, as well as the aromatic fraction C<sub>10</sub> to C<sub>40</sub>. 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<sub>10</sub>H<sub>22</sub> to C<sub>40</sub>H<sub>82</sub>, isoalkanes, cycloalkanes, alkyl benzenes, and alkyl naphthalenes and polycyclic aromatic compounds are determined as total extractable semi-volatile petroleum hydrocarbons C<sub>10</sub> to C<sub>40</sub>; 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<sub>10</sub>H<sub>22</sub> to C<sub>40</sub>H<sub>82</sub> 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 C<sub>10</sub> to C<sub>40</sub> 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<sub>10</sub>H<sub>22</sub>) and *n*-tetracontane (C<sub>40</sub>H<sub>82</sub>)

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<sub>10</sub> and C<sub>40</sub> (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**,  $(\text{CH}_3)_2\text{CO}$  (CAS-RN<sup>1)</sup> 67-64-1).

**6.2 *n*-Heptane**,  $\text{C}_7\text{H}_{16}$  (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**,  $\text{CH}_2\text{Cl}_2$  (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**,  $\text{Na}_2\text{SO}_4$ , 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  $(30 \pm 1)$  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.

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**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<sub>10</sub> to C<sub>40</sub>, 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<sub>10</sub> to C<sub>40</sub>, 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, of 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  $\mu\text{m}$  to 1,0  $\mu\text{m}$ .

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  $-18\text{ }^{\circ}\text{C}$  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.