

ICS 13.080.10

English Version

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)
(ISO/TS 16558-2:2015)

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 d'ionisation de la flamme (ISO/TS 16558-
2:2015)

Bodenbeschaffenheit - Mineralölkohlenwasserstoffe
für die Risikobeurteilung - Teil 2: Teil 2: Bestimmung
aliphatischer und aromatischer Fraktionen
schwerflüchtiger Mineralölkohlenwasserstoffe mittels
Gaschromatographie mit Flammenionisationsdetektion
(GC/FID) (ISO/TS 16558-2:2015)

This Technical Specification (CEN/TS) was approved by CEN on 13 July 2015 for provisional application.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (CEN ISO/TS 16558-2:2015) has been prepared by Technical Committee ISO/TC 190 “Soil quality” in collaboration with Technical Committee CEN/TC 345 “Characterization of soils” the secretariat of which is held by NEN.

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Endorsement notice

The text of ISO/TS 16558-2:2015 has been approved by CEN as CEN ISO/TS 16558-2:2015 without any modification.

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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).

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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](#)

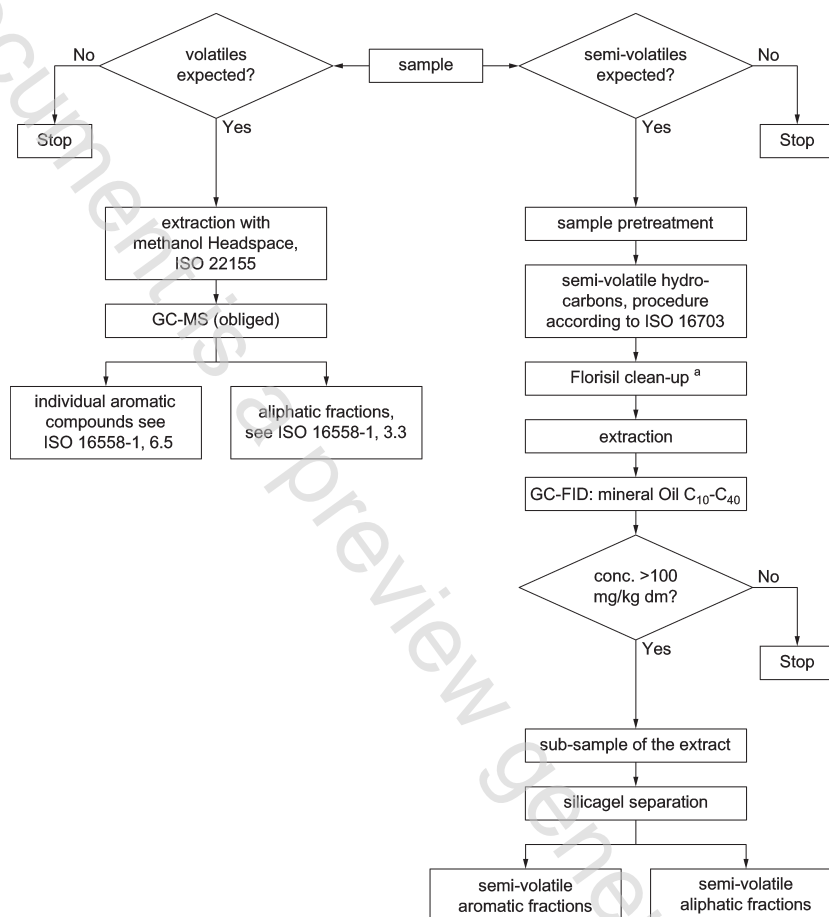
The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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

- Florisil[®] 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.
- 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 C₁₀ to C₄₀, as well as the aromatic fraction C₁₀ to C₄₀. 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 C₁₀H₂₂ to C₄₀H₈₂, isoalkanes, cycloalkanes, alkyl benzenes, and alkyl naphthalenes and polycyclic aromatic compounds are determined as total extractable semi-volatile petroleum hydrocarbons C₁₀ to C₄₀; 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 C₁₀H₂₂ to C₄₀H₈₂ 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 (C₁₀H₂₂) and *n*-tetracontane (C₄₀H₈₂)

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 C₁₀ and C₄₀ (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.