
**Soil quality — Gas chromatographic
determination of the content of
volatile aromatic hydrocarbons,
naphthalene and volatile halogenated
hydrocarbons — Purge-and-trap
method with thermal desorption**

Qualité du sol — Détermination par chromatographie en phase gazeuse des teneurs en hydrocarbures aromatiques volatils, en naphthalène et en hydrocarbures halogénés volatils — Méthode par purge et piégeage avec désorption thermique

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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*.

This third edition cancels and replaces the second edition (ISO 15009:2012), which has been technically revised.

Soil quality — Gas chromatographic determination of the content of volatile aromatic hydrocarbons, naphthalene and volatile halogenated hydrocarbons — Purge-and-trap method with thermal desorption

1 Scope

This International Standard specifies a method for quantitative gas-chromatographic determination of volatile aromatic hydrocarbons, naphthalene and volatile halogenated hydrocarbons in soil.

This International Standard is applicable to all types of soil.

NOTE In the case of unsaturated peaty soils, absorption of the extraction solution may occur.

The lower limit of quantification is dependent on the equipment used and the quality of the methanol grade used for the extraction of the soil sample.

Under the conditions specified in this International Standard the following limits of quantification apply (expressed on basis of dry matter):

Typical limit of quantification when using GC-FID:

- Volatile aromatic hydrocarbons: 0,1 mg/kg

Typical limit of quantification when using GC-ECD:

- Volatile halogenated hydrocarbons: 0,01 mg/kg

Lower limits of quantification for some compounds can be achieved by using mass spectrometry (MS) with selected ion detection.

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 4799, *Laboratory glassware — Condensers*

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

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

ISO 10381-5, *Soil quality — Sampling — Part 5: Guidance on the procedure for the investigation of urban and industrial sites with regard to soil contamination*

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

ISO 15680, *Water quality — Gas-chromatographic determination of a number of monocyclic aromatic hydrocarbons, naphthalene and several chlorinated compounds using purge-and-trap and thermal desorption*

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

ISO 22892, *Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry*

3 Principle

Test samples are taken from an untreated field moist soil sample. To prevent losses of the volatiles, samples are taken as undisturbed as possible in the field with a tube corer or by adding methanol immediately in the field.

The test sample is extracted with methanol. After centrifugation, if necessary, part of the methanol extract is brought into a purge vessel filled with water. The volatile compounds are purged with nitrogen or helium and adsorbed on a suitable adsorbing agent. The adsorbed compounds are desorbed thermally and by means of a carrier gas flow, whether or not via a cold trap, brought into a gas chromatograph. The various compounds are separated by using a capillary column with an immobile phase of low polarity. Volatile organic compounds are detected with appropriate detectors such as: mass spectrometric detector (MS), flame ionization detector (FID), electron capture detector (ECD), photo ionization detector (PID) or electrolytic conductivity detector (ELCD). Identification and quantification takes place by comparison of retention times and peak heights (or peak areas) towards internal standard added with the corresponding variables of an external standard solution. The efficiency of the procedure depends on the composition of the soil that is investigated. The described procedure does not take into account incomplete extraction caused by structure and composition of the soil sample.

When using non-specific detectors such as FID and ECD, the confirmation of the identity of the detected compounds and their concentrations should be done by repeating the gas chromatographic analysis using a column of different polarity. When using gas chromatography-mass spectrometry (GC-MS), the identity confirmation and the quantification can be done in a single run.

NOTE This International Standard follows the description of an 'off-line purge and trap' method. The use of commercially available online instruments is allowed provided that equivalent results are obtained during validation of this equipment. With such an instrument purge and trap occurs on line with gas chromatography and detection. Follow the manual of the manufacturer especially regarding the items [5.2.1](#) to [5.2.9](#).

4 Reagents

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

4.1 Water, free of volatile aromatic and volatile halogenated hydrocarbons

Usually boiler water with a temperature of at least 80 °C and 1 day old can be applied. Purging with an inert gas, e.g. a flow of 10 ml/min of nitrogen for 30 min, is another means of removing interfering compounds from water. A sufficient amount of water from the same batch should be available to complete each batch of analyses, including all preparations.

4.2 Internal standards

4.2.1 For the determination of volatile aromatic hydrocarbons preferably two internal standards shall be selected from [Table 1](#). They shall not interfere with compounds present in the sample extract.

Table 1 — Examples of suitable internal standards for the determination of volatile aromatic hydrocarbons

Compound name	CAS-RN ^a	EC-N ^b
Toluene-D8	2037-26-5	218-009-5
Ethylbenzene-D10	25837-05-2	247-292-8
2-Bromofluorobenzene	1072-85-1	214-018-3
^a Chemical Abstracts Service Registry Number		
^b EC-Number		

4.2.2 For the determination of volatile halogenated hydrocarbons preferably two internal standards shall be selected from [Table 2](#). They shall not interfere with compounds present in the sample extract.

Table 2 — Examples of suitable internal standards for the determination of volatile halogenated hydrocarbons

Compound name	CAS-RN ^a	EC-N ^b
1,4-Dichlorobutane	110-56-5	203-778-1
α,α,α -Trifluorotoluene	98-08-8	202-635-0
2-Bromofluorobenzene	1072-85-1	214-018-3
^a Chemical Abstracts Service Registry Number		
^b EC-Number		

4.3 Standard compounds

4.3.1 Volatile aromatic hydrocarbons

[Table 3](#) contains volatile aromatic hydrocarbons for use as standard compounds.

Table 3 — Examples of volatile aromatic hydrocarbons suitable as standard compounds

Compound name	CAS-RN ^a	EC-N ^b
Benzene	71-43-2	202-753-7
Toluene	108-88-3	203-625-9
Ethylbenzene	100-41-4	202-849-4
<i>o</i> -Xylene	95-47-6	202-422-2
<i>m</i> -Xylene	108-38-3	203-576-3
<i>p</i> -Xylene	106-42-3	203-396-5
Styrene	100-42-5	202-851-5
Naphthalene	91-20-3	202-049-5
^a Chemical Abstracts Service Registry Number		
^b EC-Number		

4.3.2 Volatile halogenated hydrocarbons

[Table 4](#) contains volatile halogenated hydrocarbons for use as standard compounds.