

Soil quality - Gas chromatographic determination of the content of volatile aromatic hydrocarbons, naphthalene and volatile halogenated hydrocarbons - Purge-and-trap method with thermal desorption (ISO 15009:2012)

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN ISO 15009:2013 sisaldab Euroopa standardi EN ISO 15009:2013 ingliskeelset teksti.	This Estonian standard EVS-EN ISO 15009:2013 consists of the English text of the European standard EN ISO 15009:2013.
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English Version

Soil quality - Gas chromatographic determination of the content
of volatile aromatic hydrocarbons, naphthalene and volatile
halogenated hydrocarbons - Purge-and-trap method with
thermal desorption (ISO 15009:2012)

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 (ISO 15009:2012)

Bodenbeschaffenheit - Gaschromatographische
Bestimmung des Anteils an flüchtigen aromatischen
Kohlenwasserstoffen, Naphthalin und flüchtigen
Halogenkohlenwasserstoffen - Purge-und-Trap-
Anreicherung mit thermischer Desorption (ISO 15009:2012)

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Foreword

The text of ISO 15009:2012 has been prepared by Technical Committee ISO/TC 190 "Soil quality" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 15009:2013 by 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 15009:2012 has been approved by CEN as EN ISO 15009:2013 without any modification.

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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 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 determination 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 determinations apply (expressed on a basis of dry matter):

- a) Typical limit of determination when using gas chromatography/flame ionization detection (GC/FID):
 - volatile aromatic hydrocarbons: 0,1 mg/kg.
- b) Typical limit of determination when using gas chromatography/electron capture detector (GC/ECD):
 - volatile halogenated hydrocarbons: 0,01 mg/kg

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

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 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 11465:1993/Cor 1:1994, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method — Technical Corrigendum 1*

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, 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 an 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 FIDs and ECDs, 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 1 This International Standard follows the description of an off-line purge-and-trap method. The use of commercial 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 manufacturer's manual, especially regarding the items composing the apparatus which are listed in 5.1.1 to 5.1.9.

NOTE 2 Other injection techniques, such as static headspace followed by thermal desorption (ISO 22155) or solid-phase micro-extraction (SPME), can be used, provided that their applicability is proven.

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 standard compounds

4.2.1 For the determination of volatile aromatic hydrocarbons, preferably two internal standard compounds shall be selected that do not interfere with compounds present in the sample extract.

Examples of suitable internal standards are

- toluene-D8 (CAS RN 2037-26-5),
- ethylbenzene-D10 (CAS RN 25837-05-2), and
- 2-bromofluorobenzene (CAS RN 1072-85-1).