

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

ICS 13.080.10

National foreword

This British Standard reproduces verbatim ISO 15009:2002 and implements it as the UK national standard.

The UK participation in its preparation was entrusted by Technical Committee EH/4, Soil quality, to Subcommittee EH/4/3, Chemical methods and soil characteristics, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

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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

*Qualité du sol — Détermination par chromatographie en phase gazeuse
des teneurs en hydrocarbures aromatiques volatils, en naphtalène 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15009 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Annexes A and B of this International Standard are for information only.

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.

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

- volatile hydrocarbons and naphthalene: 0,1 mg/kg;
- volatile halogenated hydrocarbons: 0,01 mg/kg.

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.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method — Technical corrigendum 1*

3 Principle

From an untreated field-moist soil sample, test samples are taken.

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, optionally via a cold trap, brought into a gas chromatograph. The various compounds are separated using a capillary column

with an immobile phase of low polarity. Volatile aromatic hydrocarbons are detected with a flame ionization detector (FID). Volatile halogenated hydrocarbons are detected with an electron capture detector (ECD). Volatile aromatic hydrocarbons and volatile halogenated hydrocarbons can be determined in one gas chromatographic run. Part of the effluent is brought to the FID and part to the ECD, e.g. via an effluent splitter. Identification and quantification takes place by comparison of retention times and peak heights (or peak areas) of 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.

Extraction with methanol is preferred to the use of acetone because acetone extracts are not suitable for “purge-and-trap”.

For confirmation of the identity of detected compounds and the concentrations found, further investigation is recommended. Confirmation can be done by repeating the gas chromatographic analysis using a column of different polarity and/or by using gas chromatography-mass spectrometry (GC-MS).

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 halogenated hydrocarbons.

Usually boiler water with a temperature of at least 80 °C and 1 day old can be used. Purging with an inert gas, e.g. nitrogen at a flowrate of 10 ml/min for 30 min, is another way to remove interfering compounds from water. Sufficient 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 should be selected that do not interfere with compounds present in the sample extract.

Examples of suitable internal standards are:

- Toluene-D8 (CAS No. 2037-26-5);
- Ethylbenzene-D10 (CAS No. 25837-05-2).

4.2.2 For the determination of volatile halogenated hydrocarbons, preferably two internal standards should be selected that do not interfere with compounds present in the sample extract.

Examples of suitable internal standards are:

- 1,4-dichlorobutane (CAS No. 110-56-5);
- α, α, α -trifluorotoluene (CAS No. 98-08-8);
- 2-bromofluorobenzene (CAS No. 1072-85-1).

4.3 Volatile aromatic hydrocarbons.

Compound	CAS No.
benzene	71-43-2
toluene	108-88-3
ethylbenzene	100-41-4
o-xylene	95-47-6