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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**

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*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.

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.

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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.

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.

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## 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);
- $\alpha, \alpha, \alpha$ -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

<i>m</i> -xylene	108-38-3
<i>p</i> -xylene	106-42-3
styrene	100-42-5
naphthalene	91-20-3

#### 4.4 Volatile halogenated hydrocarbons.

Compound	CAS No.
dichloromethane	75-09-2
trichloromethane	67-66-3
tetrachloromethane	56-23-5
1,1-dichloroethane	75-34-3
1,2-dichloroethane	107-06-2
1,1,1-trichloroethane	79-01-6
1,1,2-trichloroethane	79-00-5
1,2-dichloropropane	78-87-5
1,2,3-trichloropropane	98-18-4
<i>cis</i> -1,3-dichloropropene	10061-01-5
<i>trans</i> -1,3-dichloropropene	10061-02-6
<i>cis</i> -1,2-dichloroethene	156-59-2
<i>trans</i> -1,2-dichloroethene	156-60-5
3-chloropropene	107-05-1
trichloroethene	79-01-6
tetrachloroethene	127-18-4
monochlorobenzene	108-90-7
1,2-dichlorobenzene	95-50-1

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#### 4.5 Methanol (CAS No. 67-56-1)

The methanol used shall not contain more than 100 µg/l of the individual volatile aromatic compounds and not more than 10 µg/l of the volatile halogenated hydrocarbons that are to be analysed.

**4.6 Adsorbing agent**, polymer of 2,6-diphenyl-*p*-phenoxide<sup>1)</sup> (40 – 60 mesh) of a grade suitable for thermal desorption.

Other adsorbing agents may be used provided that their suitability has been tested.

#### 4.7 Cooling water for purge-and-trap.

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1) Tenax TA is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

The temperature of the cooling water depends on the dimensions of the purge-and-trap equipment (6.1). A temperature of about 10 °C is recommended. A cryostat shall be used if the temperature of the cooling water is too high.

4.8 **Inert carrier gas** for the gas chromatograph.

4.9 **Nitrogen** as inert gas for the purge equipment.

## 5 Preparation of standard solutions

5.1 **Primary standard solutions** for volatile aromatic and halogenated compounds in methanol, 4 g/l.

Weigh about 100 mg of the individual standard compounds (4.3 and 4.4) to an accuracy of 0,1 mg into a closed septum flask containing 25 ml methanol. Transfer the standard compounds into the flask using a syringe.

5.2 **Internal standard solutions** in methanol, 4 g/l.

Weigh about 100 mg of the individual internal standard compounds (4.2.1 and 4.2.2) to an accuracy of 0,1 mg into a closed septum flask containing 25 ml of methanol. Using a syringe, transfer the standard compounds into the flask.

5.3 **Calibration solutions** containing 0 mg/l to 200 mg/l of each primary standard (5.1) and the selected internal standard(s) (5.2), 200 mg/l.

Prepare the calibration solutions in methanol. Dilute the amounts indicated in Table 1 of the solutions obtained according to 5.1 and 5.2 with methanol (4.5) to 100 ml.

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Table 1 — Preparation of calibration solutions

Calibration solution	Internal standard solution (5.2) ml	Primary standard solution (5.2) ml	Concentration in the calibration solution mg/l	Quantity in µg/5 µl calibration solution in 100 ml (sample) water
5.3.1	5	0	0	0
5.3.2	5	1	40	0,2
5.3.3	5	2	80	0,4
5.3.4	5	3	120	0,6
5.3.5	5	4	160	0,8
5.3.6	5	5	200	1,0

## 6 Apparatus

Use the usual laboratory glassware, free of interfering compounds. All glassware shall be cleaned according to the usual procedures for this type of analysis.

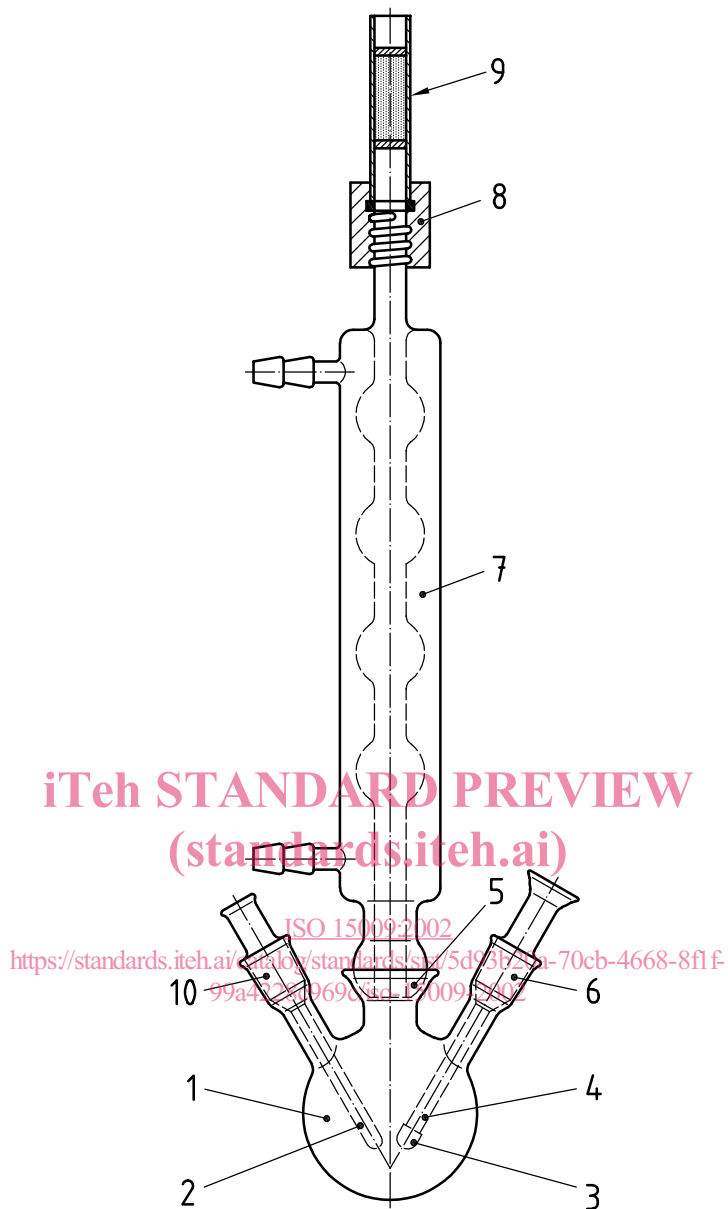
### 6.1 Purge-and-trap apparatus.

An example of an off-line purge-and-trap system is given in Figure 1.

NOTE 1 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 manufacturer's instruction manual especially regarding items 6.1.1 to 6.1.9

NOTE 2 This method is only validated for the off-line procedure.



**Key**

- 1 Round-bottom flask, 100 ml (6.1.1)
- 2 Inlet tube for thermocouple (6.1.5)
- 3 Tip of sintered glass
- 4 Gas inlet tube (6.1.2)
- 5 Spherical joint, in accordance with ISO 641, with PTFE ring (6.1.3)
- 6 Conical joint 14/23 in accordance with ISO 383
- 7 Condenser (6.1.6)
- 8 Screwcap (6.1.7)
- 9 Adsorption tube (6.1.8)
- 10 Conical joint 14/23 in accordance with ISO 383

**Figure 1 — Example of off-line purge-and-trap apparatus**

## ISO 15009:2002(E)

**6.1.1 Round-bottom flask** with three angled side necks, volume 100 ml.

**6.1.2 Gas inlet tube** with a tip of sintered glass.

**6.1.3 Spherical joint**, with PTFE.

**6.1.4 Flow adjustment mechanism.**

The nitrogen flowrate shall be  $(40 \pm 2)$  ml/min.

**6.1.5 Inlet tube** for the thermocouple.

**6.1.6 Allin- or Graham-type condenser**, in accordance with ISO 4799.

**6.1.7 Screwcap with cut-off ring** made of silicone rubber with PTFE inlay.

**6.1.8 Adsorption tubes**, made of glass or stainless steel, filled with at least 240 mg of adsorbing agent (4.6).

The adsorbent is kept in place using inert material, e.g. silanized glass fibre. The tubes shall be suitable for direct use in connection with the apparatus for thermal desorption. The tubes shall be marked on one side. The tubes shall be provided with caps of inert material, e.g. polyethylene or metal, with screwcaps and PTFE ring, allowing tight closing after purging.

Before use, the adsorbent shall be activated and purified by slowly heating the tubes to 250 °C and keeping them at that temperature for 3 h while a nitrogen flowrate of 10 ml/min is maintained. The adsorbent shall be cooled under nitrogen and the tubes analysed. The result of a blank determination shall not exceed the equivalent of 1 ng of a compound to be analysed. When the result is higher than this, the adsorbent shall be desorbed once more.

The use of commercially available tubes is recommended.

Tubes that are used should not be used again unless the blank determination meets the above-mentioned requirements.

Care should be taken to avoid cross-contamination. A heavily loaded tube can contaminate a lightly loaded tube in the sample change platform.

**6.1.9 Heating block** with thermocouple, suitable for heating 100 ml flasks.

**6.2 Centrifuge**, suitable for centrifuging tubes of 200 ml with such a rotation frequency that the radial acceleration is 2000 *g* to 3000 *g*.

**6.3 Centrifuge tubes** with a volume of 200 ml.

**6.4 Capillary columns**, of fused silica, with a non-polar or semipolar stationary phase allowing sufficient separation of the compounds of interest.

A thick film of stationary phase increases the efficiency toward the more volatile compounds.

Examples are given in 8.4 and annex A.

**6.5 Gas chromatograph** provided with a flame ionization detector (FID), an electron capture detector (ECD) based on Ni63, and a capillary column with effluent splitter 1:10 and 1:100.

Selective detectors such as photo-ionization detectors (PID) or mass selective detectors (MSD, ITD) are permitted.

NOTE PID, MSD and ITD may increase the selectivity for a certain compound with respect to interfering compounds.

**6.6 Apparatus for thermal desorption**, having the following characteristics:

- a primary desorption oven with adjustable desorption temperature up to 250 °C and adjustable desorption time;
- a cold trap/secondary desorption oven;
- a connecting tube to the gas chromatograph, with adjustable heating up to 150 °C;
- carrier gas flowrate adjustable up to 40 ml/min.

NOTE Instruments for thermal desorption are commercially available.

**6.7 Electronic integrator** or automatic recorder.**6.8 Syringes**, volume 5 µl, readable to 0,1 µl and volume 50 µl, readable to 1 µl.**6.9 Shaking machine.****7 Sampling, preservation and sample pretreatment**

Carry out sampling in accordance with ISO 10381-1 using equipment in accordance with ISO 10381-2 after coordination with the analytical laboratory.

Avoid exposure of samples to air, even during sampling, as far as possible. Avoid the use of plastics, other than PTFE.

Analyse samples as soon as possible. Keep the samples in the dark at a temperature of 3 °C ± 2 °C no longer than 4 days. Samples are not pretreated.

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**8 Procedure****8.1 Blank determination**

For each series of samples, carry out a blank determination by treating 50 ml water (4.1), to which 5 µl to 50 µl of methanol (4.5) has been added, as a sample. Laboratories shall ensure that no contamination occurs from the laboratory atmosphere.

**8.2 Dry matter content**

Determine the dry matter content of the field-moist sample in accordance with ISO 11465.

**8.3 Extraction**

Prepare a test sample by combining various small soil cores that are taken from the untreated field-moist soil using a core cutter.

Weigh 50 g of test sample into a centrifuge tube (6.3).

Add 50 ml of methanol (4.5) and close the tube. Place the tube on the shaking machine (6.9) and shake for 30 min.

Take the tube out of the shaking machine and place in the centrifuge. Centrifuge for 10 min at a rotational frequency that results in a radial acceleration of 2000 g.