
**Soil quality — Gas chromatographic
quantitative determination of volatile
aromatic and halogenated hydrocarbons
and selected ethers — Static headspace
method**

iTeh STANDARD PREVIEW
*Qualité du sol — Dosage des hydrocarbures aromatiques et halogénés
volatils et de certains éthers par chromatographie en phase gazeuse —
Méthode par espace de tête statique*
(standards.iteh.ai)

[ISO 22155:2005](https://standards.iteh.ai/catalog/standards/sist/be672e46-924f-44bf-a6e9-70a3265f2abc/iso-22155-2005)

<https://standards.iteh.ai/catalog/standards/sist/be672e46-924f-44bf-a6e9-70a3265f2abc/iso-22155-2005>



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 22155:2005](https://standards.iteh.ai/catalog/standards/sist/be672e46-924f-44bf-a6e9-70a3265f2abc/iso-22155-2005)

<https://standards.iteh.ai/catalog/standards/sist/be672e46-924f-44bf-a6e9-70a3265f2abc/iso-22155-2005>

© ISO 2005

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Principle	2
4 Reagents	2
5 Apparatus	5
6 Sampling, preservation and sample pre-treatment.....	6
7 Procedure	6
8 Calculation.....	10
9 Expression of results	10
10 Precision.....	10
11 Report	11
Annex A (informative) Relative retention times with respect to ethylbenzene-D10 of volatile aromatic hydrocarbons and volatile halogenated hydrocarbons on following columns: CP-Sil 5 CB and CP-Sil 13 CB	12
Annex B (informative) Validation	13
Bibliography	17

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 2.

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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22155 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO 22155:2005](https://standards.iteh.ai/catalog/standards/sist/be672e46-924f-44bf-a6e9-70a3265f2abc/iso-22155-2005)

<https://standards.iteh.ai/catalog/standards/sist/be672e46-924f-44bf-a6e9-70a3265f2abc/iso-22155-2005>

Soil quality — Gas chromatographic quantitative determination of volatile aromatic and halogenated hydrocarbons and selected ethers — Static headspace method

1 Scope

This International Standard specifies a static headspace method for quantitative gas chromatographic determination of volatile aromatic and halogenated hydrocarbons and selected aliphatic ethers in soil.

This International Standard is applicable to all types of soil.

The limit of determination is dependent on the detection system 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 the basis of dry matter):

- a) when using gas chromatography with a flame ionization detector (GC-FID), the typical limit of determination is 0,2 mg/kg for volatile aromatic hydrocarbons and 0,5 mg/kg for aliphatic ethers as methyl *tert*-butyl ether (MTBE) and *tert*-amyl methyl ether (TAME);
- b) when using gas chromatography with an electron capture detector (GC-ECD), the typical limit of determination is 0,01 mg/kg to 0,2 mg/kg for volatile halogenated hydrocarbons.

NOTE Lower limits of determination 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 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

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

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 22892¹⁾, *Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry*

1) To be published.

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 and methanol is added immediately.

The test sample is extracted with methanol. An aliquot of the methanol extract is transferred into a headspace vial with a defined amount of water and sealed. The temperature of the vials is stabilized in a thermostatic system to a temperature within the range 50 °C to 80 °C to achieve specified equilibrium conditions. Gas chromatographic analysis of the volatile compounds in gaseous phase in equilibrium with the water in the vials is carried out by using headspace injection and an appropriate capillary column. Volatile organic compounds are detected with appropriate detectors such as, a mass spectrometric detector (MS), a flame ionization detector (FID), an electron capture detector (ECD), a photo-ionization detector (PID) or an electrolytic conductivity detector (ELCD).

Identification and quantification is made by comparison of retention times and peak heights (or peak areas) comparing to the internal standard added.

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 Other injection technique such as purge and trap followed by thermal desorption or solid phase micro-extraction (SPME) can be used, provided that their applicability is proven.

iTeh STANDARD PREVIEW (standards.iteh.ai)

4 Reagents

All reagents shall be of recognised analytical grade. Verify whether the reagents are applicable for this specific purpose and are free of interfering compounds. [ISO 22155:2005](https://standards.iteh.ai/catalog/standards/sist/be672e46-924f-44bf-a6e9-70a52692a001-22155-2005)

4.1 Water, free of volatile organic compounds, showing negligible interferences in comparison with the smallest concentration to be determined.

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, which do not interfere with the compounds present in the methanol extract.

For the determination of volatile aromatic hydrocarbons and volatile halogenated hydrocarbons, preferably two internal standards shall be selected.

4.2.1 Examples of suitable internal standards are:

- Toluene-D8 (CAS No. 2037-26-5)
- Ethylbenzene-D10 (CAS No. 25837-05-2)
- 2-Bromofluorobenzene (CAS No. 1072-85-1)

4.2.2 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
<i>o</i> -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

4.5 Aliphatic ethers

Compound	CAS No.
methyl <i>tert</i> -butyl ether (MTBE)	1634-04-4
<i>tert</i> -amyl methyl ether (TAME)	994-05-8

NOTE This method can also be used for volatile organic compounds not included in this International Standard, provided it has been validated for each new compound.

4.6 Methanol (CAS-No. 67-56-1), as a solvent for the extraction of soil samples and for the preparation of standard solutions.

NOTE Other solvents which are readily soluble in water and do not interfere with the analytical process can be used as well, for example dimethylformamide (DMF) and dimethylsulfoxide (DMSO).

4.7 Carrier gases for gas chromatography, nitrogen or argon-methane mixture ultrapure.

Other gases for gas chromatography shall be used in accordance with the instrument manufacturer's instructions.

4.8 Standard solutions

4.8.1 Standard stock solutions for the volatile compounds in methanol

Prepare the stock solutions by adding defined amounts (e.g. 100 µl) of each standard compound (4.3 and 4.4) with a microlitre syringe. Immerse the tip of the needle in the solvent methanol and weigh with an accuracy of 0,1 mg.

NOTE 1 A convenient concentration (4 mg/ml) of the standard stock solution is obtained by weighing 100 mg of the standard substance and dissolving it in 25 ml of the solvent. The solution is stable for about 6 months when stored at -18 °C.

NOTE 2 For practical reasons, mixed standard stock solutions can also be prepared.

4.8.2 Internal standard stock solutions in methanol

Prepare the internal standard stock solutions with the individual internal standard compounds (4.2.1 and 4.2.2) in the same manner as in 4.8.1.

The containers containing the solutions shall be marked or weighed so that any evaporation losses of the solvent may be recognised. The solutions shall be stored in volumetric flasks with ground glass stoppers at a temperature of 4 °C ± 2° C in the dark. Prior to use, they shall be brought to ambient temperature.

4.8.3 Intermediate mixed standard solutions

Prepare intermediate mixed standard solutions by mixing a defined volume of each individual standard stock solution or a mixed standard stock solution and dilute with methanol.

NOTE A typical concentration is 40 µg/ml.

Store the intermediate mixed standard solutions at 4 °C ± 2° C not longer than 3 months.

4.8.4 Working standard solutions

Prepare at least five different concentrations (e.g. from 0,2 µg/ml to 3,2 µg/ml) by suitable dilutions of the intermediate mixed standard solutions. Add 50 µl to 500 µl of these concentrations to 10 ml of methanol using a microlitre syringe.

4.8.5 Working internal standard solutions

Prepare the working internal standard solutions of defined concentration (e.g. 0,4 µg/ml) as described in 4.8.3 and 4.8.4.

4.8.6 Aqueous calibration standard solutions

Prepare the calibration solutions (see Table 1) by adding a defined amount (e.g. 50 µl) of working standard solutions and internal standard solutions to a defined volume (e.g. 10 ml) of water in an appropriate headspace vial. Use a syringe and immerse the top of the needle in the water. Seal the vial tightly with a crimp cap fitted with polytetrafluoroethylene (PTFE) coated septum. The total volume of the methanol used for

calibration shall be the same as for extraction of the soil sample. Make sure that the content of the organic solvent in the final aqueous calibration standard solution does not exceed 2 % (volume fraction).

Table 1 — Example for the preparation of calibration solutions

Calibration solution	Working standard solutions (4.8.4) µl	Working internal standard solutions (4.8.5) µl	Concentration in working standard solution µg/ml	Quantity in calibration solution of 10 ml (sample) water ng	Concentration in aqueous calibration solution µg/l
1	50	50 (methanol)	0	0	0
2	50	50	0,2	10	1
3	50	50	0,4	20	2
4	50	50	0,8	40	4
5	50	50	1,6	80	8
6	50	50	3,2	160	16

5 Apparatus

Usual laboratory glassware, free of interfering compounds.

All glassware shall be cleaned according to the usual procedures for this type of analysis.

5.1 Glass vials, with suitable septum, fitted with a PTFE-coated septum, capable of being hermetically sealed in the field as well as at elevated temperatures.

- Glass vials (50 ml to 100 ml) with screw caps, for field moist soil samples taken in the field.
- Glass vials (10 ml for 5 ml water and 22 ml for 10 ml water) with crimped metallic caps, compatible with the headspace system, connected to an appropriate gas chromatographic system.

5.2 Crimping pliers

5.3 Headspace system

This method was developed for using a totally automated equilibrium headspace analyser available from several commercial sources. The system used shall meet the following specifications.

The system shall be capable of

- keeping the vials at a constant temperature (between 50 °C and 80 °C), and
- transferring accurately a representative portion of the headspace into a gas chromatograph fitted with capillary columns.

5.4 Shaking machine

A shaking machine with horizontal movement (200 movements to 300 movements per min).

5.5 Capillary columns

Fused silica capillary columns with a non-polar or semi-polar stationary phase allowing sufficient separation of the compounds of interest. A thick film of stationary phase increases the efficiency of the separation of more volatile compounds.

Examples are given in 7.4.

5.6 Gas chromatograph

A gas chromatograph equipped with one or two appropriate detectors. Detectors like flame ionization detector (FID), electron capture detector (ECD), photo-ionization detector (PID) or electrolytic conductivity detector (ELCD) and mass spectrometer (MS) can be used, depending on the substances to be analysed and their target level of contamination. The mass spectrometer should be able of operating over the total mass range of interest and being equipped with a data system capable of quantifying ions using selected m/z values.

5.7 Electronic integrator or computer with chromatographic software.

5.8 Syringes, 5 μ l, 10 μ l, 50 μ l, 100 μ l, 250 μ l and 500 μ l volumes.

6 Sampling, preservation and sample pre-treatment

Sampling shall be carried out according to ISO 10381-1, using equipment specified in ISO 10381-2 after coordination with the analytical laboratory.

Exposure of samples to air, even during sampling, shall be avoided as far as possible.

Samples shall be analysed as soon as possible. Samples shall be stored in the dark at a temperature of $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ no longer than 4 days. Samples shall not be pretreated.

For the determination of volatile organic compounds, it is strongly recommended to take the test sample under controlled conditions in the field, to add methanol there as described in 7.2 and to properly seal the sample container. Make sure that the sample is completely covered with methanol. For practical reasons, it is convenient to transfer a defined volume of soil, using an appropriate device into a pre-weighed vial, which is filled with a defined volume of methanol. Prevent leakages by cleaning the top of the vessel before sealing it. The soil samples should be taken from undisturbed material using an appropriate sample cutter of known volume, e.g. a modified 20-ml disposable plastic syringe with the tip cut off. The soil sample should be collected immediately after exposing a fresh soil surface of the drilling core, e.g. an open window sampler or trial pit wall. The incorporation of material, like roots or stones, should be avoided as far as possible. At least one blank sample on every site shall be prepared in the field, by opening the prepared vial for the same time period as that necessary for the soil sample.

The sampling vials should be kept dark in a cooler (before and after sampling) throughout the entire transportation.

Determine the dry matter content of the field moist sample according to ISO 11465. For the determination of dry matter content, a separate sample container should be completely filled with field-moist soil and sealed immediately, and opened only to take the test sample for measurement.

NOTE The loss of volatile components from solid samples is difficult to avoid. As the number of manipulative operations used in the handling of the sample increases, the loss of volatile components will become more significant.

7 Procedure

7.1 Blank determination

For each series of samples, a solvent blank determination shall be carried out by adding 10 μ l to 100 μ l of methanol (4.6) to 5 ml to 10 ml of water (4.1), as it is done with a sample. Ensure that no contamination occurs from the laboratory atmosphere.

7.2 Extraction

Add a defined amount of test sample (25 g to 50 g), collected as described in sampling (Clause 6) with a sampling device into a pre-weighed vial (50 ml to 100 ml) (5.1) filled with a defined amount of methanol (25 ml

to 50 ml). Screw the cap fitted with PTFE-coated septum onto the vial. After the transportation of the sealed vials, preferably kept dark in coolers to the laboratory, weigh the vials, place them on the horizontal shaking machine (5.4) and shake for 30 min.

Other shaking devices, such as end-over-end shaking, may be applied. However, due to less intensive mixing the shaking time should be prolonged to 1 h.

7.3 Procedure

Take the vial out of the shaking machine and allow it to stand 10 min to 15 min to settle out the solid materials.

If there is no settling of solid materials on standing, centrifuge for 10 min at a rotational speed, giving a radial acceleration of 2 000 *g*.

Transfer a defined volume of water (5 ml to 10 ml) into a headspace vial. Inject 10 µl to 100 µl of the methanol extract, obtained according to 7.2, to the bottom of the vial and seal tightly with a crimp cap fitted with PTFE-coated septum. From this point on, after preparing the spiked water samples, proceed to the analysis in a very similar way to the water analysis. Prepare the calibration samples in the same way, with the same volume [10 µl to 100 µl of the calibration solutions (4.8.6)].

NOTE 1 A lower detection limit could be achieved by addition of sodium chloride, NaCl (e.g. 3 g per 10 ml).

Stabilize the sample temperature in the headspace system in the following manner:

Place the vials of water samples in the thermostatted tray of the headspace system at a fixed temperature in the range between 50 °C and 80 °C, for at least 30 min and for the same time for all vials.

NOTE 2 For specific equipment working at equilibrium, the time required to reach equilibrium can vary, depending on the volatile organic substance and the volume of the vials used. Experience has shown that at least 30 min are necessary.

ISO 22155:2005

7.4 Gas chromatographic analysis

7.4.1 General

Example of gas chromatographic conditions for this analysis:

Stationary phase:	low polarity, e.g. DB 5, DB 624, DB 1701 Restek volatiles
Film thickness:	1 µm to 3 µm
Column length:	50 m to 60 m
Internal diameter:	0,25 mm to 0,32 mm
Oven temperature:	40 °C during 4 min 4 °C/min up to 200 °C 200 °C during 10 min
Detector temperature:	300 °C
Carrier gas:	helium
Gas flow:	20 cm/s to 30 cm/s
Inlet:	200 °C
Split ratio:	1:20