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

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

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ISO/CEN PARALLEL PROCESSING

This final draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO-lead** mode of collaboration as defined in the Vienna Agreement. The final draft was established on the basis of comments received during a parallel enquiry on the draft.

This final draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel two-month approval vote in ISO and formal vote in CEN.

Positive votes shall not be accompanied by comments.

Negative votes shall be accompanied by the relevant technical reasons.

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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 22155:2011), which has been technically revised.

Soil quality — Gas chromatographic 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 quantification 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 quantifications apply (expressed on basis of dry matter).

Typical limit of quantification when using GC-FID:

- volatile aromatic hydrocarbons: 0,2 mg/kg;
- aliphatic ethers as methyl tert.-butyl ether (MTBE) and tert.-amyl methyl ether (TAME): 0,5 mg/kg.

Typical limit of quantification when using GC-ECD:

- volatile halogenated hydrocarbons: 0,01 mg/kg to 0,2 mg/kg.

Lower limits of quantification for some compounds can be achieved by using mass spectrometry (MS) with selected ion detection (see [Annex D](#)).

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 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*

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

When using non-specific detectors 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.

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 organic compounds

Water, free from volatile organic contaminants shall show 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 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

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

Compound name	CAS-RN ^a	EC-N ^b
1,4-dichlorobutane	110-56-5	203-778-1
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.

Table 4 — Examples of volatile halogenated hydrocarbons suitable as standard compounds

Compound name	CAS-RN ^a	EC-N ^b
Dichloromethane	75-09-2	200-838-9
Trichloromethane	67-66-3	200-663-8
Tetrachloromethane	56-23-5	200-262-8
1,1-dichloroethane	75-34-3	200-863-5
1,2-dichloroethane	107-06-2	203-458-1
1,1,1-trichloroethane	71-55-6	200-756-3
1,1,2-trichloroethane	79-00-5	201-166-9
1,2-dichloropropane	78-87-5	201-152-2
1,2,3-trichloropropane	98-18-4	202-486-1
<i>cis</i> -1,3-dichloropropene	10061-01-5	233-195-8
^a Chemical Abstracts Service Registry Number. ^b EC-Number.		

Table 4 (continued)

Compound name	CAS-RN ^a	EC-N ^b
<i>trans</i> -1,3-dichloropropene	10061-02-6	208-826-5
<i>cis</i> -1,2-dichloroethene	156-59-2	205-859-7
<i>trans</i> -1,2-dichloroethene	156-60-5	205-860-2
3-chloropropene	107-05-1	203-457-6
Trichloroethene	79-01-6	201-61-04
Tetrachloroethene	127-18-4	204-825-9
Monochlorobenzene	108-90-7	203-628-5
1,2-dichlorobenzene	95-50-1	202-425-9
^a Chemical Abstracts Service Registry Number. ^b EC-Number.		

4.4 Aliphatic ethers

[Table 5](#) contains aliphatic ethers for use as standard compounds.

Table 5 — Examples of aliphatic ethers suitable as standards compounds

Compound name	CAS-RN ^a	EC-N ^b
methyl tert.-butyl ether (MTBE)	1634-04-4	216-653-1
tert.-amyl methyl ether (TAME)	994-05-8	213-611-4
^a Chemical Abstracts Service Registry Number. ^b EC-Number.		

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.

4.5 Methanol

Methanol (see [Table 6](#)) is used as solvent for the extraction of soil samples and for the preparation of standard solutions.

Table 6 — Registration data

Compound name	CAS-RN ^a	EC-N ^b
Methanol	67-56-1	200-659-6
^a Chemical Abstracts Service Registry Number. ^b EC-Number.		

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).

4.6 Gases for gas chromatography

Helium, nitrogen or argon-methane mixture ultrapure. Other gases for gas chromatography shall be used in accordance with the instrument manufacturer's instructions.

4.7 Standard solutions

4.7.1 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, 4.4 and 4.5) with a microlitre syringe (5.8). 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 stock solution is stable for about 6 m when stored at -18 °C.

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

4.7.2 Internal standard stock solutions in methanol

The containers containing the solutions shall be weighed so that any evaporation losses of the solvent may be recognized. The solutions shall be stored at a temperature of (4 ± 2) °C in the dark. Prior to use, they shall be brought to ambient temperature.

Prepare the internal standard stock solutions with the individual internal standard compounds (4.2.1 and 4.2.2) in the same procedure as in 4.7.1.

4.7.3 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 ± 2) °C not longer than 3 m.

4.7.4 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 adding 50 µl to 500 µl of 4.7.3 to methanol (10 ml) using a microlitre syringe (5.8).

4.7.5 Working internal standard solutions

Prepare the internal standard solutions of defined concentration (e.g. 0,4 µg/ml) as described in 4.7.3.

4.7.6 Aqueous calibration standard solutions

Prepare the calibration solutions (see Table 7) 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 which will be taken for the methanol extract of the soil sample (see 7.3). Make sure that the content of the organic solvent in the final aqueous calibration standard solution does not exceed the volume fraction of 2 %.

Table 7 — Example for the preparation of calibration solutions

Calibration solution	Working standard solution (4.7.4)	Working internal standard standards (4.7.5)	Concentration in working standard solution	Quantity in calibration solution of 10 ml (sample) water	Concentration in aqueous calibration solution
	µl	µl	µg/ml	ng	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

4.8 Methanol containing internal standards

Prepare methanol containing a suitable concentration of the internal standards (e.g. 0,4 µg/ml). The concentration shall be such that the end concentration in the water extract in the headspace vial is of the same level as in the calibration standards.

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.

Glass vials (50 ml to 100 ml) and screw cap, fitted with a PTFE-coated septum 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 a PTFE-coated septum and crimped metallic cap, compatible with the headspace system connected to an appropriate gas chromatographic system. The vials shall be capable of being hermetically sealed in the field as well as at elevated temperatures.

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).

The system shall be capable of transferring accurately a representative portion of the headspace into a gas chromatograph fitted with capillary columns.

5.4 Shaking machine, with horizontal movement (200 to 300 movements per minute).

5.5 Capillary columns.

Fused silica capillary columns 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 of the separation of more volatile compounds.

Examples are given in [7.4.1](#).