



SLOVENSKI STANDARD
oSIST prEN ISO 16703:2024
01-november-2024

Trdni matriksi v okolju - Določanje ogljikovodikov v območju C10 do C40 s plinsko kromatografijo (ISO/DIS 16703:2024)

Environmental Solid Matrices - Determination of hydrocarbon content in the range of C10 to C40 by gas chromatography (ISO/DIS 16703:2024)

Bodenqualität- Bestimmung des Gehalts an Kohlenwasserstoffen im Bereich C10 bis C40 durch Gaschromatographie (ISO/DIS 16703:2024)

Matrices solides environnementales - Dosage des hydrocarbures de C10 à C40 par chromatographie en phase gazeuse (ISO/DIS 16703:2024)

Ta slovenski standard je istoveten z: prEN ISO 16703

[oSIST prEN ISO 16703:2024](https://standards.metat/catalog/standards/sist/94f0eb76-4b06-4772-8133-31c070c5941e/osist-pr-en-iso-16703-2024)

ICS:

13.080.10 Kemijske značilnosti tal Chemical characteristics of soils

oSIST prEN ISO 16703:2024

en,fr,de



DRAFT International Standard

ISO/DIS 16703

Environmental Solid Matrices — Determination of hydrocarbon content in the range of C₁₀ to C₄₀ by gas chromatography

ICS: ISO ics

ISO/TC 190/SC 3

Secretariat: DIN

Voting begins on:
2024-09-03

Voting terminates on:
2024-11-26

iTeh Standards
(<https://standards.iteh.ai>)
Document Preview

[oSIST prEN ISO 16703:2024](https://standards.iteh.ai/catalog/standards/sist/9afbcfb6-db80-4772-8f33-e1c076e59a1e/osist-pren-iso-16703-2024)

<https://standards.iteh.ai/catalog/standards/sist/9afbcfb6-db80-4772-8f33-e1c076e59a1e/osist-pren-iso-16703-2024>

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 16703:2024(en)

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENTS AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

© ISO 2024

ISO/DIS 16703:2024(en)

iTeh Standards (<https://standards.iteh.ai>) Document Preview

[oSIST prEN ISO 16703:2024](https://standards.iteh.ai/catalog/standards/sist/9afbcbf6-db80-4772-8f33-e1c076e59a1e/osist-pren-iso-16703-2024)

<https://standards.iteh.ai/catalog/standards/sist/9afbcbf6-db80-4772-8f33-e1c076e59a1e/osist-pren-iso-16703-2024>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2024

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

ISO/DIS 16703:2024(en)

Contents

	Page
Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Principle	2
5 Reagents	2
6 Equipment	4
7 Sample conservation and pretreatment	4
8 Procedure	5
8.1 Blank.....	5
8.2 Extraction and clean-up.....	5
9 Determination by gas chromatography	6
9.1 Test of the performance of the gas chromatographic system.....	6
9.2 Test of repeatability.....	6
9.3 Initial calibration.....	6
9.4 Working calibration.....	6
9.5 Validity check of the calibration function.....	6
9.6 Measurement.....	6
9.7 Integration.....	6
10 Calculation	7
11 Quality control	8
11.1 Suitability check of the clean-up procedure.....	8
11.2 Recovery of the hydrocarbon standard solution.....	8
11.3 Quality control sample.....	9
12 Performance characteristics	9
13 Test report	9
Annex A (informative) Examples of gas chromatograms of hydrocarbon standard and selected samples	10
Annex B (informative) Determination of the boiling range of mineral oil hydrocarbons from the gas chromatogram	14
Annex C (informative) Validation data	16
Annex D (informative) Determination of product type	18
Annex E (informative) Aqueous liquid waste	22
Bibliography	23

ISO/DIS 16703:2024(en)

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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO [had/had not] received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

oSIST prEN ISO 16703:2024

<https://standards.iteh.ai/catalog/standards/sist/9afbcbf6-db80-4772-8f33-e1c076e59a1e/osist-pren-iso-16703-2024>

Environmental Solid Matrices — Determination of hydrocarbon content in the range of C₁₀ to C₄₀ by gas chromatography

1 Scope

This International Standard specifies a method for the quantitative determination of the mineral oil (hydrocarbon) (C₁₀ to C₄₀) content in soil and waste samples by gas chromatography.

The method is applicable to mineral oil contents (mass fraction) between 100 mg/kg and 10 000 mg/kg soil, expressed as dry matter, and can be adapted to lower limits of detection.

Using this standard all hydrocarbons with a boiling range of approximately 175 °C to 525 °C, e.g. *n*-alkanes from C₁₀H₂₂ to C₄₀H₈₂, isoalkanes, cycloalkanes, alkyl benzenes, alkyl naphthalenes and polycyclic aromatic compounds are determined as hydrocarbons, provided they do not adsorb on the Florisil column during clean-up.

The standard can also be applied to determine any fraction in-between the range *n*-C₁₀ to *n*-C₄₀.

Volatile hydrocarbons cannot be quantitatively determined using this standard. This will affect the determination of some common hydrocarbon fuels, e.g. petrol.

NOTE 1 On the basis of the peak pattern of the gas chromatogram (see [Annex D](#)) and of the boiling points of the individual *n*-alkanes listed in [Annex B](#) the approximate boiling range of the hydrocarbons and some qualitative information on the nature of the hydrocarbons can be obtained.

NOTE 2 At the moment there is no sufficient information on how to handle organic liquid wastes (e.g. by-product of a manufacturing process, oils and greases) for the analysis of hydrocarbons.

NOTE 3 Aqueous liquid waste samples can be analyzed in accordance with EN ISO 9377-2 or the procedure specified in [Annex E](#).

NOTE 4 Plastic containing samples must be treated by a hexane-like extraction solvent without usage of acetone in order to avoid overfindings (false-high content).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 14507, *Soil quality — Pretreatment of samples for determination of organic contaminants*

ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods — Part 1: Linear calibration function*

ISO/DIS 16703:2024(en)

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

hydrocarbon content by gas chromatography

sum of compounds that are extractable with acetone/hexane-like solvent (2+1) from environmental solid matrices, that do not adsorb on a specified single Florisil column cleanup and can be chromatographed on a non-polar capillary column with retention times between those of *n*-decane (C₁₀H₂₂) and *n*-tetracontane (C₄₀H₈₂)

Note 1 to entry: Substances that comply with this definition are mainly non-polar long chain or branched aliphatic, alicyclic, alkyl substituted aromatic or polycyclic aromatic compounds.

Note 2 to entry: This definition differs from that given by utilizing a gravimetric approach (e.g. EN 14345).

Note 3 to entry: Non-polar and weakly polar compounds (e.g. halogenated hydrocarbons) and high contents of more polar compounds, e.g. animal and vegetable fats and oils, may exceed the adsorption capacity of the Florisil used.

4 Principle

A known amount of the homogenized sample is extracted by mechanical shaking or sonication with acetone/hexane-like solvent. The organic layer is separated and washed twice with water. Polar compounds are removed by chromatography on Florisil. An aliquot of the purified extract is analyzed by capillary gas chromatography with flame ionization detection.

The total peak area between the retention time window standards *n*-decane and *n*-tetracontane is measured and the amount of hydrocarbons in the sample is quantified against an external standard consisting of equal amounts of two different types of mineral oil.

NOTE 1 hexane-like solvent can be understood as *n*-heptane, or other non-polar solvent (e.g. petroleum ether, cyclohexane).

NOTE 2 If lower detection limits are required, large-volume injection or concentration of the final extract can be applied.

5 Reagents

In general, all reagents shall be reagent grade and suitable for their specific purposes.

5.1 Acetone, (CH₃)₂CO.

5.2 hexane-like solvent as ***n*-Heptane**, C₇H₁₆; alternatively, other hexane-like solvents after proving suitability (range of boiling point 60 °C-100 °C).

5.3 **Florisil for preparation of clean-up column**, particle size 150 µm to 250 µm (60 mesh to 100 mesh), heated for at least 16 h at 140 °C and stored in a desiccator over a molecular sieve.

NOTE Commercially available cartridges containing Florisil and sodium sulfate are also applicable.

5.4 **Anhydrous sodium sulfate** (Na₂SO₄), heated for at least 2 h at 550 °C. Afterwards the sodium sulfate should be stored under dry conditions (e.g. a desiccator).

ISO/DIS 16703:2024(en)

5.5 Test solution of stearyl stearate (C₃₆H₇₂O₂); *n*-octadecanoic acid octadecyl ester.

Dissolve e.g. (100 ± 1) mg of *n*-octadecanoic acid octadecyl ester in 100 ml hexane-like solvent [5.2] to yield a concentration of 1 mg/ml. The solution is stable for 6 months when stored in a refrigerator between 1 °C and 5 °C.

5.6 Retention-time window (RTW) standard solution, containing *n* tetracontane and *n*-decane.

Retention-time window (RTW) standard solution is the range-defining standard solution. Weigh (30 ± 1) mg of *n*-tetracontane into a 1 l volumetric flask, dissolve completely in an appropriate volume of *n*-heptane (6.2), add 30 µl of *n*-decane (about 21 mg), mix well, fill up to volume with hexane-like solvent and homogenize. This solution shall be used for all dilution steps of the hydrocarbon standard (6.7).

Store at room temperature.

NOTE *n*-Tetracontane is only moderately soluble in hexane-like solvent. Slight warm-up and/or sonicate to accelerate the dissolution process.

5.7 Hydrocarbon standard solution for calibration

Mix approximately equal masses of two different types of mineral oil. Weigh accurately this mixture and dissolve in the RTW standard solution (6.6) to give a hydrocarbon mass concentration of about 8 g/l.

The first oil type should show discrete peaks (e.g. a diesel fuel) in the gas chromatogram, as can be seen in Figure A.1 (left part of the chromatogram). The second type should have a boiling range higher than the first one, and should show a hump in the gas chromatogram, as can be seen in Figure A.1 (right part of the chromatogram). A suitable oil of this type is for example a lubricating oil without any additives.

The calibration solutions can be prepared by diluting an aliquot of this standard solution with different volumes of the RTW standard solution (6.6).

5.8 Calibration control solution

Prepare an independent control solution in accordance with (6.7) using a hydrocarbon concentration about in the middle of the working range.

5.9 System-performance standard solution

Prepare a mixture of equal amounts, on a mass basis, of the *n*-alkanes with even carbon numbers from C₁₀ to C₄₀, dissolved in hexan-like solvent [5.2], to give a concentration of about 50 mg/l of each *n*-alkane. Store at room temperature.

This solution is used to verify the suitability of the gas chromatographic system for the resolution of *n*-alkanes as well as for the detector response.

NOTE This solution is in addition used to give information of the retention times of the *n*-alkanes to characterize the hydrocarbons in the samples (c.f. Annex D).

5.10 Solution for definition of retention times

For all fraction based on even carbon numbers, the system performance standard solution (6.9) can be used for determination of retention times. If fractions are to be determined with odd carbon numbers (e.g. >C₁₀-C₂₅), a standard solution containing at least all relevant *n*-alkanes have to be applied.

5.11 Preparation of the clean-up column

A plug of pre-washed glass wool or a PTFE frit is pushed down into the column [6.9]. Then, successively 2 g Florisil [5.3] and 2 g sodium sulfate [6.4] are added. The column shall be prepared immediately before use or stored in a dry environment (e.g. desiccator). In addition, commercially available columns can be used, if suitability has been proven.

ISO/DIS 16703:2024(en)

After proving suitability, a miniaturization of the cleanup can be applied, and the ratio used extract to Florisil/sodium sulfate must remain the same (10:2:2; e.g. 5 mL with 1 g Florisil and 1 g sodium sulfate).

6 Equipment

6.1 Standard laboratory glassware, which shall be treated at high temperatures or rinsed with acetone (5.1) and dried before use.

6.2 Devices for extraction, mechanical shaker (at least 120 rpm) or ultrasonic bath.

6.3 Gas chromatograph, equipped with a non-discriminating injector, a flame ionization detector (FID) and a temperature programmable oven.

NOTE The use of a large-volume injection system can improve the limit of detection considerably.

6.4 Capillary column, fused silica column with suitable stationary phase and dimensions, e.g.

stationary phase: non-polar (e.g. immobilised 100 % dimethyl polysiloxane, 95 %-dimethyl-5 %-diphenyl polysiloxane, modified siloxane polymer, etc.);

length: 2 to 20 m;

internal diameter: 0,1 mm to 0,32 mm;

film thickness: 0,1 µm to 1,0 µm.

The column should give a base-line separation of the *n*-alkanes when the system performance standard solution [6.9] is run.

NOTE 1 Thermally stable low bleed columns should be preferred.

NOTE 2 The use of a pre-column, e.g. wide-bore (0,53 mm internal diameter) deactivated fused silica of at least 2 m of length is recommended.

6.5 Data system, capable of integrating the total area of the chromatogram, compensating for column bleed and reintegrating after defining a new baseline.

6.6 Glass extraction vessel of volume at least 100 ml, with screw caps provided with a septum coated with polytetrafluoroethylene (PTFE).

6.7 Glass tube, e.g. 25 ml, with ground glass stopper or screw caps incorporating a septum coated with polytetrafluoroethylene (PTFE).

6.8 Separation funnel, at least 500 ml, with a ground glass stopper.

6.9 Chromatography column for clean-up, glass columns of about 10 mm internal diameter. Preferably, the upper part of the column should be widened to use as solvent reservoir and the lower part to be narrowed to form a tip.

Plastic columns can be used if they meet the requirements given in chapter 8.2.

7 Sample conservation and pretreatment

Storage and pretreatment should be according to ISO 18512, if not otherwise specified.

Before analysis the samples shall be homogenized.