SLOVENSKI STANDARD
SIST ISO/TR 11046:1997

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Kakovost tal - Določanje mineralnih olj - Metoda infrardeče spektrometrije in plinske kromatografije

Soil quality -- Determination of mineral oil content -- Method by infrared spectrometry and gas chromatographic method

Qualité du sol -- Dosage des huiles minérales -- Méthode par spectrométrie à l'infrarouge et méthode par chromatographie en phase gazeuse

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13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

SIST ISO/TR 11046:1997 **en**

TECHNICAL REPORT

ISO TR 11046

First edition
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Soil quality — Determination of mineral oil content — Method by infrared spectrometry and gas chromatographic method

Qualité du sol — Dosage des huiles minérales — Méthode par spectrométrie à l'infrarouge et méthode par chromatographie en phase gazeuse

Reference number
ISO/TR 11046:1994(E)

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 main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

— type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;

— type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;

— type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 11046, which is a Technical Report of type 2, was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

The following reasons led to the decision to publish this document in the form of a Technical Report of type 2.

Due to the severe impact of chlorofluoro hydrocarbons on the environment, these compounds should not be used for a test method specified in an International Standard. However, at present, no alternative for the extraction agent used in the procedures specified here is available.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland

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Therefore, it was decided to retain the method using chlorofluoro hydrocarbon until alternatives which are applicable in the routine analysis are found, and to publish this document as a Technical Report.

Everybody working in the field of hydrocarbon analysis is encouraged to seek alternative solvents or methods.

Annexes A, B and C of this Technical Report are for information only.

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Soil quality — Determination of mineral oil content — Method by infrared spectrometry and gas chromatographic method

1 Scope

This Technical Report specifies two methods for the quantitative determination of mineral oil content in soil by infrared spectrometry (Method A) and gas chromatography (Method B).

Method A is applicable to mineral oil contents above 20 mg/kg on a dry matter basis. Method B is applicable to mineral oil contents above 100 mg/kg on a dry matter basis.

NOTES

1 It is emphasized that the methods described do not determine the origin of the substances that are considered to be "mineral oil" according to clause 4.

2 The infrared spectrometric method in particular is sensitive to false positive results caused by polar compounds.

3 With the infrared spectrometric method, the boiling range of compounds determined as mineral oil is not defined. With the gas chromatographic method, compounds with a boiling range of 175 °C to 525 °C are determined (*n*-alkanes $C_{10}H_{22}$ to $C_{40}H_{82}$). Petrol cannot be determined quantitatively with these methods, due to loss of volatile compounds during sample pretreatment.

4 Weak polar compounds of recent biogenic origin may be determined as mineral oil.

5 Relatively high contents of polar compounds give interferences in the determination. This applies especially to the infrared spectrometric method.

6 Halogenated hydrocarbons may interfere.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Technical Report. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Technical Report are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3924:1977, *Petroleum products — Determination of boiling range distribution — Gas chromatography method.*

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

3 Definition

For the purposes of this Technical Report, the following definition applies.

3.1 mineral oil: Compounds that are extractable from soil by use of 1,1,2-trichloro-1,2,2-trifluoroethane under the following conditions:

— they do not adsorb on magnesium silicate or aluminium oxide;

— they absorb radiation with a wavenumber of 2 925 cm^{-1}, and/or 2 958 cm^{-1}, and/or 3 030 cm^{-1} (Method A);

— they can be chromatographed with retention times between those of *n*-decane ($C_{10}H_{22}$) and *n*-tetracontane ($C_{40}H_{82}$) (Method B).

NOTE 7 The substances defined are mainly non-polar compounds containing aliphatic and/or C-H groups.

4 Principle

Moist soil taken from the field is chemically dried with a hygroscopic salt, crushed and then extracted with 1,1,2-trichloro-1,2,2-trifluoroethane. Polar compounds are removed either by adding magnesium silicate and shaking or in a closed circuit system containing aluminium oxide.

For spectrometry (Method A), an infrared spectrum of the extract is recorded from 3 125 cm^{-1} to 2 800 cm^{-1}. The CH_2-absorption band at about 2 925 cm^{-1}, the CH_3-absorption band at about 2 958 cm^{-1} and the aromatic CH-absorption band at about 3 030 cm^{-1} are a measure of the mineral oil content. The mineral oil content of the sample is calculated from the determined absorbances using empirically determined absorption coefficients.

For quantitative determination of mineral oil contents (Method B), part of the purified extract is added to hexane and analysed by gas chromatography. For the separation, a column with a non-polar immobile phase is used. For detection, a flame ionization detector (FID) is used. The total area under the peaks from decane ($C_{10}H_{22}$) to *n*-tetracontane ($C_{40}H_{82}$) is a measure of the amount of mineral oil. The mineral oil content of the sample is calculated using an external standard prepared from a standardized oil.

5 Reagents

All reagents shall be of recognized analytical grade and suitable for their specific purpose.

5.1 Reagents used for methods A and B

5.1.1 1,1,2-Trichloro-1,2,2-trifluoroethane, ($C_2Cl_3F_3$)

The suitability of this reagent, for use in infrared spectroscopy shall be verified by recording an infrared spectrum from 3 125 cm^{-1} to 2 800 cm^{-1} in a cell with an optical path length of 4,00 cm, with an identical empty cell as a reference. The solvent is suitable when the transmittance in the range of 3 000 cm^{-1} to 2 900 cm^{-1} is greater than about 30 %.

NOTE 8 This solvent will be referred to in this Technical Report as "CFE".

5.1.2 Magnesium silicate, of particle size 150 µm to 250 µm (mesh: 60 to 100), heated for 16 h at 140 °C and stored in a desiccator.

NOTES

9 Magnesium silicate under the trade name of "Florisil"[1] has been found to be suitable. It is made from diatomae and mainly composed of anhydrous magnesium silicate.

10 The thickness of the layer of the magnesium silicate during the heating shall not be greater than 0,5 cm.

11 The suitability of magnesium silicate is checked by adding 1,0 g to 40 ml of lauric acid solution (5.1.3) followed by shaking for 30 min on a shaking machine (6.1.3). After decanting and measuring, the transmittance in the range from 3 030 cm^{-1} to 2 925 cm^{-1} shall be 35 % to 45 % using cells with and optical path length of 1,00 cm^{-1}.

5.1.3 Lauric acid solution

Dissolve 2,00 g of *n*-dodecanoic acid ($C_{12}H_{24}O_2$) in "CFE" (5.1.1).

5.1.4 Internal standard stock solution

Dissolve exactly 200 mg of *n*-tetracontane in 1 litre of "CFE" (5.1.1). Dilute the solution 10 times to give a concentration of 20,0 mg/l.

5.1.5 Aluminium oxide (Al_2O_3), basic or neutral activity I, particle size 63 µm to 200 µm (mesh 70 to 230).

NOTE 12 The suitability of the aluminium oxide is tested by passing 40 ml of lauric acid solution through the aluminium oxide column. The transmittance of the eluate in the range from 3 030 cm^{-1} to 2 925 cm^{-1} shall be 35 % to 45 % using cells with an optical path length of 1,00 cm.

5.1.6 Anhydrous sodium sulfate, heated for at least 2 h at 550 °C.

5.2 Reagents used for method A

5.2.1 *n*-Hexadecane ($C_{16}H_{34}$)

Dissolve 180 mg of *n*-hexadecane in 1 000 ml of "CFE" (5.1.1).

5.3 Reagents used for method B

5.3.1 *n*-Hexane.

5.3.2 *n*-Alcane standard.

Either

a) a certified mixture of equal amounts, on a mass basis, of the *n*–alcanes with even carbon numbers

1) Florisil 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.

of C_{10} to C_{40}, dissolved in *n*-hexane (5.3.1), with a content of 50 mg/l of each *n*-alkane; or

b) an *n*-alkane standard in accordance with ISO 3924.

NOTE 13 This standard is used to verify the suitability of the gas chromatographic system for the separation as well as for the response.

5.3.3 Mineral oil standard.

A mixture of equal amounts, on a mass basis, of two different mineral oil types, dissolved in *n*-hexane, with a mineral oil content of 8,00 g/l and a C_{40} content of exactly 20,0 mg/l.

NOTE 14 This mineral oil standard should consist of two different types of oil. The first type should show discrete peaks in the gas chromatogram as can be seen, for example, in annex A, figure A.1 a) (left part of chromatogram). The second type should have a boiling range higher than the first type and should show a "hump" in the gas chromatogram, as can be seen, for example, in figure A.1 a) (right part of chromatogram). An oil of this type is, for example, a lubricating oil without any additives.

6 Apparatus

6.1 General

Usual laboratory glassware, which shall be washed and rinsed with "CFE" (5.1.1) and then dried before use.

6.1.1 Glass sample containers, of capacity at least 0,5 litre, with screw caps provided with an inlay of polytetrafluoroethylene (PTFE).

6.1.2 Grinding apparatus.

6.1.3 Shaking machine, with a horizontal movement with up to 200 moves per minute.

6.1.4 Glass-fibre filters, with a diameter of 60 mm, heated for 3 h at 500 °C.

6.1.5 Soxhlet extractor, of capacity 150 ml.

6.1.6 Chromatography column, with a closed circuit complying with figure 1.

Dimensions in millimetres

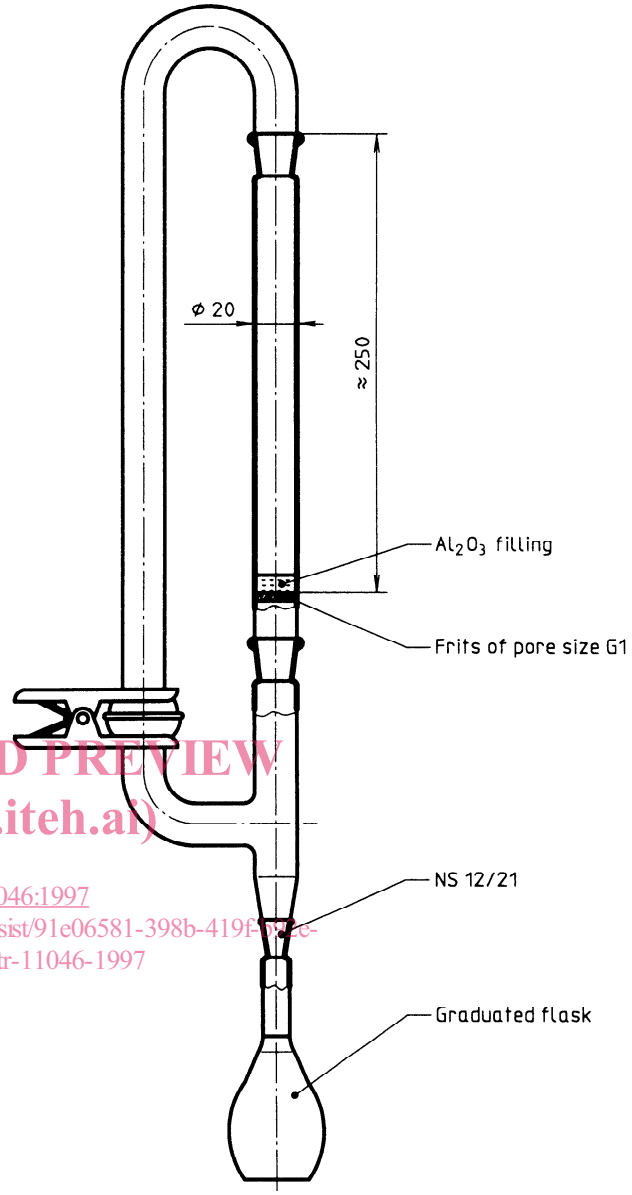

Figure 1 — Closed circuit chromatography column

6.2 Apparatus used for method A

6.2.1 Quartz optical cells, that can be closed and are suitable for infrared measurements, with optical path lengths of 1,00 cm and 4,00 cm (optional 0,2 cm).

6.2.2 Infrared spectrometer, suitable for application in the range of at least 3 200 cm^{-1} to 2 800 cm^{-1}.

NOTE 15 Throughout this Technical Report, the procedure of taking infrared spectra is described for conventional dispersive double beam spectrometers. However, the