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

Committees responsible for this Draft for Development

The preparation of this Draft for Development was entrusted by the Environment and Pollution Standards Policy Committee (EPC/-) to Technical Committee EPC/48, upon which the following bodies were represented:

- Association of Consulting Scientists
- Association of County Councils
- Association of Environmental Consultants
- Association of Public Analysts
- British Gas plc
- British Geological Survey
- British Society of Soil Science
- Chemical Industries' Association
- Confederation of British Industry
- Department of Agriculture Northern Ireland
- Department of the Environment
- Department of the Environment (Drinking Water Inspectorate)
- Department of Trade and Industry
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Institute of Groundsmanship
- Institute of Terrestrial Ecology
- Institution of Environmental Health Officers
- Institution of Water and Environmental Management
- Macaulay Land Use Research Institute
- Ministry of Agriculture, Fisheries and Food
- Soil Survey and Land Research Centre
- Water Research Centre
- Water Services Association of England and Wales

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

- British Ceramic Research Ltd.
- Institute of Occupational Medicine
- National Rivers Authority
- Royal Society of Chemistry
- Society of Chemical Industry

This British Standard, having been prepared under the direction of the Environment and Pollution Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 15 September 1994

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National foreword

This Draft for Development has been prepared under the direction of the Environment and Pollution Standards Policy Committee and is identical with ISO/TR 11046:1994 *Soil quality — Determination of mineral oil content — Method by infrared spectrometry and gas chromatographic method*, published by the International Organization for Standardization (ISO). ISO 11046 was produced as a result of international discussion in which the United Kingdom took an active part.

ISO/TR 11046 was prepared by Technical Committee ISO/TC 190, Soil quality, Subcommittee SC 3, Chemical methods and soil characteristics.

ISO/TR 11046 is a Technical Report type 2, issued while the subject is still under technical development. Technical Reports of type 2 are subject to review within 3 years of publication to decide whether they can be converted into International Standards.

The future of this Draft for Development is bound to that of ISO 11046 and the Draft for Development will not be reviewed or developed separately.

Cross-reference

International Standard	Corresponding British Standard
ISO 11465:1993	BS 7755 <i>Soil quality</i> Part 3 <i>Chemical methods</i> Section 3.1:1994 <i>Determination of dry matter and water content on a mass basis by a gravimetric method</i> (Identical)

The Technical Committee has reviewed the provisions of ISO 3924, to which normative reference is made in the text, and has decided that they are acceptable for use with this Draft for Development.

This publication is not to be regarded as a British Standard

It is being issued in the Draft for Development series of publications and is of a provisional nature for the following reasons. The method uses chlorofluorohydrocarbons which, because of their impact on the environment, should not be specified in an International Standard. However, at present, there are no satisfactory alternative extraction agents known and users of this Draft for Development are encouraged to seek alternative solvents or methods, so that information and experience of its practical application may be obtained and reported to the responsible ISO Technical Committee. Users of this Draft for Development are invited to comment on its technical content, ease of use and any ambiguities or anomalies. These comments will be taken into account when preparing the UK national response to ISO on the question of whether the Technical Report can be converted to an International Standard. Comments should be sent in writing to BSI, 2 Park Street, London W1A 2BS, quoting the number of this Draft for Development, the relevant clause and any proposed revision, within 2 years of the issue of this Draft for Development.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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.

NOTE 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.

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

NOTE 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 C₁₀H₂₂ to C₄₀H₈₂). Petrol cannot be determined quantitatively with these methods, due to loss of volatile compounds during sample pretreatment.

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

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

NOTE 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 absorb 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 (C₁₀H₂₂) and *n*-tetracontane (C₄₀H₈₂) (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 CH₂-absorption band at about 2 925 cm⁻¹, the CH₃-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 (C₁₀H₂₂) to *n*-tetracontane (C₄₀H₈₂) 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, (C₂Cl₃F₃)

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.

NOTE 9 Magnesium silicate under the trade name of “Florisol”¹⁾ has been found to be suitable. It is made from diatomae and mainly composed of anhydrous magnesium silicate.

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

NOTE 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 an optical path length of 1,00 cm⁻¹.

5.1.3 Lauric acid solution

Dissolve 2,00 g of *n*-dodecanoic acid (C₁₂H₂₄O₂) 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 (Al₂O₃), 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 (C₁₆H₃₄)

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*-alkanes with even carbon numbers of C₁₀ to C₄₀, 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 C₄₀ 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.

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 use of Fourier-Transform infrared spectrometers (FT-IR) is also possible. All procedures should be adopted to the single beam technique according to the instructions given in the operating manual.

¹⁾ Florisol 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.