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Soil quality — Screening soils for isopropanol-extractable organic compounds by determining emulsification index by light attenuation

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National foreword

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**Soil quality — Screening soils for
isopropanol-extractable organic
compounds by determining
emulsification index by light
attenuation**

*Qualité du sol — Analyse rapide des sols pour les composés
organiques extractibles à l'isopropanol en déterminant l'indice
d'émulsification par l'atténuation de la lumière*





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Foreword

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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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The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Introduction

Light attenuation due to light scattering/absorption by emulsions can be used to screen for a variety of isopropanol-extractable organic substances such as total petroleum hydrocarbons (TPH). This is one of the main organic contaminants of many contaminated land sites.

Petroleum hydrocarbons do not typically have many functional groups or contain any hetero-atoms that are easily detected in common analytical procedures. When the non-polar nature of organic compounds is targeted, as in this proposed method, turbidity/emulsification is produced by a number of organic compounds including BTEX (benzene, toluene, ethylbenzene and xylenes); PNAs (poly-nuclear aromatics); fuels; oils and greases that frequently are found at contaminated land sites.

This method can be used for screening soil samples to estimate the total amount of recoverable petroleum hydrocarbon contamination in soil including a wide range of fuels, oils, and greases. The emulsification approach in this method is incorporated to rapidly screen soil samples using a system calibrated with the organic substance present on the investigated site. If the contaminant is unknown, commercially available diesel oil can be used for calibration. This makes turbidity/emulsification analysis a very versatile analytical method that can be used on most hydrocarbon/non-polar organic substance spills without prior knowledge of the exact BTEX or PNA content or composition of the contaminant. This method is more sensitive to non-polar heavier organic compounds from jet fuel to oils and greases, but less sensitive to more volatile and lighter hydrocarbon fuels. A turbidity analysis assists the user in recognizing the contamination by non-polar organic compounds at a given site.

This International Standard describes the procedure for on-site soil screening of organic compounds using emulsification and light attenuation due to light scattering/absorption analysis.

Soil quality — Screening soils for isopropanol-extractable organic compounds by determining emulsification index by light attenuation

1 Scope

This International Standard specifies the procedure to screen highly contaminated soils to detect organic compounds extractable with isopropanol, including a wide range of fuels, oils, and greases. The method is useful for finding hot spots. It is applicable both in laboratories and for site screening in the field. The working range is approximately 0,01 to 0,3 in absorbance units, corresponding to approximately 500 mg/kg to 10 000 mg/kg of isopropanol-extractable organic compounds in soil.

The light attenuation due to light scattering/absorption approach in this method is designed to quickly screen soil samples using calibration with the most appropriate substance(s) likely to be present on a given site to indicate the concentration levels.

This screening technique is applicable for a broad spectrum of organic compounds, mainly hydrocarbons. Organic compounds are a very broadly defined mixture of compounds, which show their own specific emulsification indices (see [Annex A](#)) and a gross emulsification index in a mixture sample, defined primarily by their insolubility in water. The more insoluble the compounds (e.g. non-polar compounds), the higher the response. Hydrocarbons are generally less-reactive and have little polarity. Determination of emulsification indexes uses their non-polar nature to detect organic compounds including a wide range of hydrocarbons from about C₈ to about C₃₆.

NOTE This method can also be applied to biological substances such as vegetable oils.

This method is not applicable for determination of specific organic compounds or groups of compounds that may be part of a larger organic compound mixture. As with other screening techniques, it is advisable to confirm a certain percentage of both positive and negative test results, especially when near or above a regulatory action limit or when the presence of background or when interfering organic compounds such as surface active substances are suspected to be present.

This method does not address the evaporation of any volatile organic compound mixtures (i.e., gasoline) during sampling, preparation and detection. Although the screening method can be used for the quantitative detection of volatile hydrocarbons, it is not intended that the method be used for the quantitative determination of volatile petroleum hydrocarbons unless evaporation during sample handling is addressed; the response factor be appropriately corrected, or the method performance be demonstrated on real samples.

If emulsifiers or surface active substances (e.g. detergents) are present, significantly negatively-biased or false negative results can be obtained. If there is any evidence for the presence of surfactances in the soil, this method cannot be applied.

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 11074, *Soil quality — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11074 and the following apply.

3.1 emulsification index

ratio of the sample measured absorbance value under the conditions of this test compared with that of the single calibration standard

4 Principle

4.1 General

This emulsification method for detection of organic contaminants in soil consists of three basic steps:

- extraction: extraction of organic compounds from soil samples with isopropanol;
- salting-out: decrease the water solubility of the extracted organic compounds in the water diluted isopropanol by increasing the ionic strength of this solution adding sodium chloride solution (salting-out effect);
- detection: measuring the light absorbance at 585 nm of the resulting salting-out solution in a 10 mm path length cell.

4.2 Interferences or other surfactants

Surface-active agents such as soap and common surfactants adversely affect emulsification or reduce the degree thereof.

NOTE Shaking a mixture of 1 g of soil sample and 10 ml of water to look for formation of pronounced foam may be useful to confirm the probability that significant amounts of surface-active agents are present in soil samples.

5 Reagents

5.1 Isopropanol.

5.2 Sodium chloride solution 30 % (m/V).

Mix 300 g of water-free NaCl with approximately 500 ml of water and dilute to 1 l.

6 Apparatus

6.1 Spectrometer, handheld, portable, or laboratory spectrophotometer working at a wavelength range of (585 ± 10) nm.

6.2 Spoon, metal or plastic (10 ml to 20 ml).

6.3 Sample tube, suitable glass tube (typically about 15 ml) with a tight-fitting cap for extraction by shaking.

6.4 Glass cell, 10 mm path length glass cell for assessing the degree of emulsification via light absorption at 585 nm. Disposable plastic 10 mm path length cells may also be used.

6.5 Membrane filter, pore size 0,45 µm, to remove soil particles from the salting-out extraction solution.

Filters made of isopropanol-resistant materials, such as PTFE and cellulose acetate, shall be used. The filter shall be checked for isopropanol resistance at calibration when it is not confirmed.

6.6 Balance, portable, with precision of 0,1 g to weigh a soil sample.

7 Procedure

7.1 Calibration

Prepare a calibration curve using a blank and at least three concentrations of the most likely present organic compound in isopropanol. Take 10 ml of the blank and each isopropanol solutions, fill up to 100 ml with water. Then add 1 ml of the sodium chloride solution (5.2) for salting-out. Further follow the procedure given in 7.3 and plot a curve (concentration vs. absorbance). Examples are shown in Annex A.

If the contaminant is unknown, commercially available diesel oil should be used for calibration.

7.2 Sample preparation

Ensure that samples are collected from below any vegetative layer of soil. No plants or other organic parts should remain in the sample matrix.

Ensure complete and proper homogenization by hand prior to sample division, if necessary.

Free water contained in soil samples will affect the resulting turbidity/emulsification. If the water content exceeds 20 % m/V, it is recommended that the sample is air-dried (<35 °C) before analysis. If the sample is air dried, the method is not applicable for volatile organic compounds.

7.3 Measurement

Weigh (1 ± 0,1) g of soil into a sample tube (6.3) and mix with (10 ± 0,5) ml of isopropanol. Shake the mixture for (5 ± 0,5) min. Allow to stand for (5 ± 0,25) min and filter using a 0,45 µm membrane filter (6.5). Dilute the filtrate with water up to 100 ml. Add 1 ml of the sodium chloride solution (5.2) for salting-out organic substances extracted with isopropanol. Shake the resulting solution for 1 min, and leave for at least 3 min.

If the volume of the filtrate is less than about 8 ml, take an aliquot and dilute 1:9 with water. Use the sodium chloride solution (5.2) in the same ratio as described above.

NOTE 1 It can be difficult to filter the total isopropanol volume when soil samples are clayish or silty.

Measure the absorbance of the solution with the spectrometer (6.1) at around 585 nm using a glass cell having a 10 mm optical path (6.4). The absorbance shall be between 0 and 0,3. If the absorbance observed is outside that range, use more isopropanol for extraction and repeat the procedure. In this case, take a 10 ml fraction of the filtrate to follow the subsequent procedure to dilute the filtrate with water and thereafter. The following procedure is also applicable:

- dilute the extract with the blank salt solution [the dilution factor is hereby designated as D_f in Formula (1) in 7.4] and repeat the measurement;
- report the result as exceeding the working range concentration.

NOTE 2 For example, AOS > 10 000 mg/kg.

The entire procedure and its conditions are schematically summarized in Annex B.

7.4 Calculation

Take the absorbance measured and use the calibration curve (see 7.1) to calculate the amount of organic compounds in the salting-out solution, *AOC* (in mg/l).

The amount of an organic compound in soil, *AOS* (in mg/kg), is calculated from the *AOC* by the Formula (1):

$$AOS = \frac{AOC \cdot 10 \cdot V_{\text{isopropanol}}}{M_{\text{sample}} \cdot D_f} \quad (1)$$

where

AOC is the amount of an organic compound in a salting-out solution, in mg/l;

*M*_{sample} is the mass of soil sample, in g;

*V*_{isopropanol} is the volume of isopropanol used for extraction, in ml;

*D*_f is the factor of the dilution with the blank salt solution.

NOTE 1 When 10 ml or 20 ml of isopropanol is used for extraction, for example, *V*_{isopropanol} is 10 or 20, respectively.

NOTE 2 When no dilution was made, *D*_f = 1.

7.5 Report

Report the results in milligrams of organic compound per kilogram soil.

AOS shall be rounded off and reported as follows:

- a) to the nearest 100 mg/kg up to 10 000 mg/kg;
- b) to the nearest 1 000 mg/kg over 10 000 mg/kg.

EXAMPLE

- (a) 1 520 mg/kg measured is reported as *AOS* 1 500 mg/kg;
- (b) 11 650 mg/kg measured is reported as *AOS* 12 000 mg/kg.

8 Quality assurance (QA) and quality control (QC)

A program of field QA/QC should include a minimum of periodic sand blanks, equipment blanks, soil spikes, and duplicates. Other procedures should be implemented depending on the specific requirements of each site.

To ensure accurate quantification and repeatable results, it is recommended that a re-calibration be carried out at a certain interval or at least daily.

The analytical results contain uncertainty arising from numerous sources, heterogeneity of the 1 g soil sample aliquot used, difficulty in using matrix matched reference samples, etc. The expanded uncertainty of measurement can be estimated from the uncertainty budget with standard uncertainties.

9 Test report

The test report shall contain the following information:

- a) a reference to this International Standard, i.e. ISO 17183;

b) complete identification of the sample;

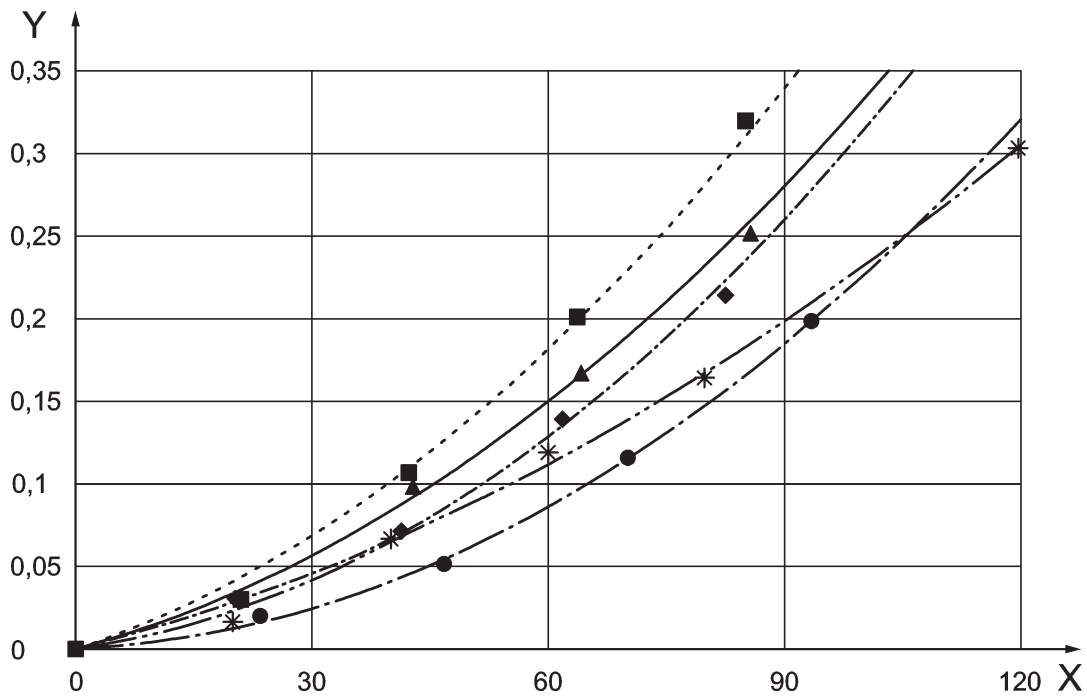
c) the results of the determination (see [7.5](#));

EXAMPLE AOS = 1 500 mg/kg (determined as diesel oil).

d) any details not specified in this International Standard or which are optional, as well as any factor which may have affected the results.

Annex A (informative)

Examples of turbidity indexes for various kinds of organic compounds



Key

- X concentration, in mg/l
- Y absorbance
- ◆ diesel fuel
- heavy fuel oil
- ▲ lubricant
- * jet fuel
- corn oil

Figure A.1 — Examples of turbidity indexes

Annex B (normative)

Schematic procedure of the method and its analytical conditions

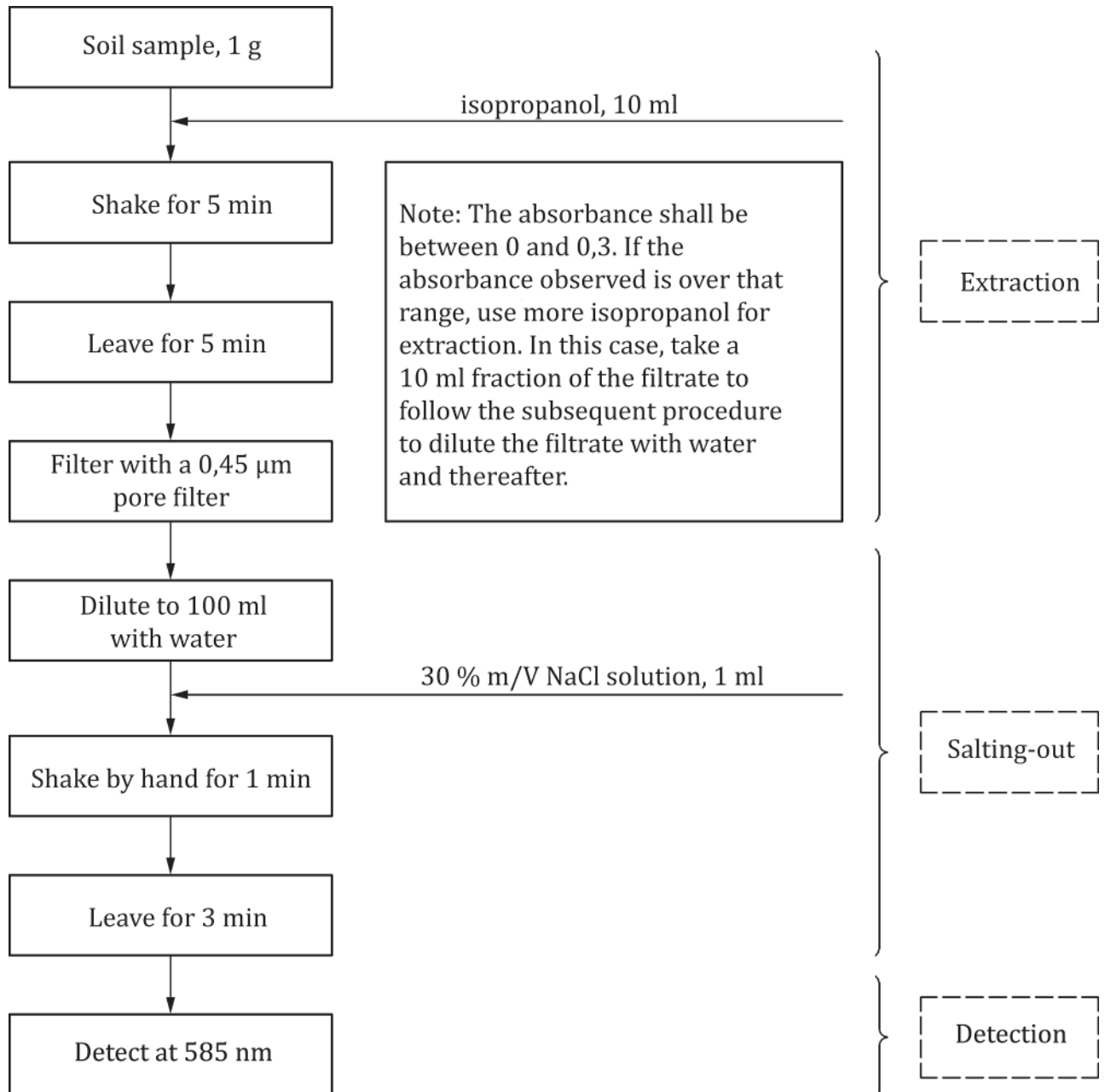


Figure B.1 — Screening procedure for detection of organic compounds extracted with isopropanol using turbidimetry

Annex C (informative)

Results on an interlaboratory test for determination of an emulsification index by light attenuation to screen soils for isopropanol-extractable organic compounds

C.1 General

An interlaboratory validation trial was organized by the Japanese National Committee for ISO/TC 190/SC 3/WG 10. Five organizations from two countries participated in the trial.

C.2 Samples

Seoul National University in Korea prepared plural samples using an agricultural soil as a matrix. The soil particles typically have a size of 15,7 μm , standard deviation of 14,2 μm and a specific surface area of 7,05 m^2/g . Lubricant oil is contained in the samples as an organic compound. The concentrations of the lubricant oil in the samples are (703 ± 58) mg/kg and $(1\ 060 \pm 107)$ mg/kg , which were determined by gas chromatography as stipulated in Reference [9]. All samples were prepared as reference materials by the National Instrumentation Center for Environmental Management, Seoul National University, Seoul, Korea.

C.3 Procedure

The concentrations of the lubricant oil contained in samples from Seoul National University were determined by applying three times the determination method specified in this International Standard. In case the concentration was lower than the detection limit, 2 g of the samples were applied to the determination.

C.4 Results

See [Table C.1](#). For summary of the results, see [Table C.2](#).

Table C.1 — Results on the evaluation of petroleum concentration in soil samples by determining emulsification index by light attenuation

Sample	Matrix/ content	Reference concentration mg/kg	Determined or detected concentration mg/kg (n=3)						
			isopropanol extraction method (Sample, 2 g)						
			A	B	C	D	E	F	G
			Filtered with PTFE filters			Filtered with cellulose acetate filters			
TL	Agricultural soil, TPH free/ lubricant	703 ± 58	720		444	538	686	476	525
			864	-	417	574	584	555	566
			877		440	671	508	487	602
TH	Agricultural soil, TPH free/ lubricant	1 060 ± 107	1 530		884	979	1 090	866	817
			1 708	-	854	1 066	1 308	855	901
			1 816		833	952	1 147	972	874
TL	Agricultural soil, TPH free/ lubricant	703 ± 58		830	488		739	691	
			-	849	551	-	772	637	-
				858	519		706	642	
TH	Agricultural soil, TPH free/ lubricant	1 060 ± 107		1 294	1 172		998	1 072	
			-	1 194	1 123	-	1 021	1 046	-
				1 241	1 018		1 047	1 068	
			Filtered with PTFE filters						
TL	Agricultural soil, TPH free/ lubricant	703 ± 58	-	-	641		770	677	583
					577	-	805	571	554
					545		738	607	584
TH	Agricultural soil, TPH free/ lubricant	1 060 ± 107	-	-	957		1 119	923	1 098
					903	-	1 020	877	920
					1 017		1 046	901	831
			Filtered with filters mixed cellulose ester, (by F)			Filtered with cellulose acetate filters, (by F)			
TH	Agricultural soil, TPH free/ lubricant	1 060 ± 107	161			892			
			241			847			
			121			958			
			Filtered with PTFE filters, (by F)			Filtered with cellulose acetate filters, (by F)			
TH	Agricultural soil, TPH free/ lubricant	1 060 ± 107	1 042			1 071			
			1 070			1 094			
			1 068			1 069			

Table C.2 — Summary of the results

Sample	Round	Reference concentration in mg/kg	Average in mg/kg	Repeatability	Reproducibility	Relative repeatability in %	Relative reproducibility in %
TL	1	703 ± 58	585	57	133	10	23
TL	2		690	27	127	4	18
TH	1	1 060 ± 107	1 081	75	309	7	29
TH	2		1 108	42	96	4	9

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