BS ISO 14507:2003

Soil quality — Pretreatment of samples for determination of organic contaminants

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British Standards

National foreword

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The UK participation in its preparation was entrusted by Technical Committee EH/4, Soil quality, to Subcommittee EH/4/3, Chemical methods and soil characteristics, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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Qualité du sol — Prétraitement des échantillons pour la détermination des contaminants organiques

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Foreword

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ISO 14507 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Introduction

The properties of organic micropollutants can differ greatly according to chemical species:

- they can range from non-volatile to very volatile compounds (low to high vapour pressure);
- they can be labile or reactive at ambient or elevated temperatures;
- they can be biodegradable or UV-degradable;
- they can have considerably different solubilities in water;
- they require different analytical procedures.

Because of these differences, a general pretreatment procedure cannot be proposed. The goal of a pretreatment procedure is to prepare a test sample in which the concentration of the contaminant is equal to the concentration in the original soil, provided, however, that this procedure does not alter the chemical species to be analysed. For instance, if the sample contains only small particles and the contaminant is homogeneously distributed, it is not necessary to grind the sample. In this International Standard, the size 2 mm is used to distinguish between small and large soil particles.

Consistency among the following aspects is important:

- soil diversity;
- the aim of the analysis (including its accuracy);
- the nature of the chemical species to be analysed.

The particle size distribution of the sample in relation to the mass of sample taken for analysis is also important to pretreatment. For the analysis of organic contaminants, the sample mass taken in most cases is about 20 g.

With such a sample mass, and provided that the contaminant is homogeneously distributed and the particles in the sample are smaller than about 2 mm, further grinding of the sample is not necessary. If the sample contains large particles or if the contaminant is heterogeneously distributed (as occurs for instance with tar particles), it is not possible to take a representative test sample of about 20 g without grinding the sample. To improve the homogeneity, samples are ground to a size smaller than 1 mm. Prior to analysis, very often no information about the distribution of the contaminant in the soil is known.

Some analytical procedures start with a field-moist sample. Drying of the sample gives lower extraction results. If the sample is not dried, grinding is not possible.

In a situation in which accurate results are needed, the best available pretreatment procedure should be used. If it is necessary to establish whether the concentration is above a certain limit and it is already known that the soil is heavily polluted, the simplest pretreatment procedure may meet the need. In this case however, the result may not be presented as a representative value for the whole sample.

The choice depends above all on the volatility of the organic compounds under analysis. It also depends on the soil particle size distribution, the heterogeneity of the sample and the analytical procedure.

Soil quality — Pretreatment of samples for determination of organic contaminants

1 Scope

This International Standard specifies three methods for the pretreatment of soil samples in the laboratory prior to the determination of organic contaminants:

- if volatile organic compounds are to be measured;
- if moderately volatile to non-volatile organic compounds are to be measured, if the result of the subsequent analysis must be accurate and reproducible, and if the sample contains particles larger than 2 mm and/or the contaminant is heterogeneously distributed;
- if non-volatile organic compounds are to be measured and the extraction procedure prescribes a field-moist sample, or if the largest particles of the sample are smaller than 2 mm and the contaminant is homogeneously distributed. This procedure is also applicable if reduced accuracy and repeatability are acceptable.

The pretreatment described in this International Standard is used in combination with an extraction procedure in which the contaminant is available for the extraction liquid.

NOTE For the pretreatment of soil samples for the purposes of determining non-volatile inorganic compounds and physico-chemical soil characteristics, see ISO 11464.

2 Normative references

The following referenced documents are indispensable for the application 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 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 11074-2, *Soil quality — Vocabulary — Part 2: Terms and definitions relating to sampling*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 11074-2 and the following apply:

3.1
volatile compound
organic compound having a boiling point below 300 °C (at a pressure of 101 kPa)

NOTE 1 This includes volatile aromatic and volatile halogenated hydrocarbons as determined in accordance with ISO 15009. Some mono- and dichlorophenols, for instance, and naphthalene also belong to this group.

NOTE 2 The selection of the categories for volatile and moderately volatile compounds can be related in principle to the vapour pressure. However, as the vapour pressure of only a small number of compounds is known, and in view of the relationship between vapour pressure and boiling point, the boiling point has been chosen as the criterion for distinction. See Annex A.

3.2
moderately volatile compound
organic compound having a boiling point above 300 °C (at a pressure of 101 kPa)

NOTE 1 This definition includes:

a) mineral oil (see ISO/TR 11046);

b) most polycyclic aromatic hydrocarbons (PAH) (see ISO 13877);

c) polychlorobiphenyls (PCB) (see ISO 10382);

d) organochlorine pesticides (see ISO 10382).

NOTE 2 In ISO/TR 11046, mineral oil is defined as the group of hydrocarbons which, on chromatography, have retention times lying between that of n-decane ($C_{10}H_{22}$) and n-tetracontane ($C_{40}H_{82}$). As in this International Standard the limit for volatile compounds lies at a boiling point of 300 °C [approximately hexadecane (C_{16})], this means that mineral oil should be considered as a volatile compound, as part of the compounds to be determined fall within the boiling point range of volatile compounds. However for practical reasons, it has been decided that the pretreatment for moderately volatile compounds should be specified for the determination of mineral oil. As a result of cryogenic crushing, an improvement in the extraction yield occurs for compounds with a boiling point above 300 °C. The possible losses for the lower-boiling hydrocarbons (C_{10} to C_{16}) are assumed to be low, due to the retaining effect of the higher-boiling hydrocarbons present in mineral oil and to be compensated by the higher extraction yield of the other hydrocarbons present. As the total yield is used to determine the mineral oil as a group parameter, it is assumed that pretreatment using the method for moderately volatile compounds gives the best results at present.

4 Principle

4.1 Sampling

Sampling shall be carried out in accordance with ISO 10381-1. The samples taken shall be kept cool and processed as soon as possible. The method of pretreatment depends on the volatility of the organic compound(s) or group(s) of organic compounds to be determined.

When taking a subsample, the quantity of soil should be removed from the sample container in such a way that this quantity:

a) is a subsample over the full depth of the sample, and

b) can be taken quickly.

4.2 Volatile compounds

For determination of volatile organic compounds, core test samples are taken from the sample and extracted according to the relevant specific analytical procedure. If composite samples are required, extracts of individual samples are mixed. It is not possible to obtain composite samples without severe losses of volatiles. The procedure is described in 8.2.

4.3 Moderately volatile compounds

Samples are chemically dried at a low temperature (– 196 °C, liquid nitrogen). The dried samples are cooled with liquid nitrogen and ground with a cross-beater mill with a sieve of 1 mm mesh size (cryogenic crushing). After grinding, suitable test portions are processed according to the relevant specific analytical procedures. Composite samples can be prepared by mixing the ground samples. This procedure is described in 8.3.

If the extraction procedure requires a field-moist sample, drying and grinding are not possible.

If the original sample only contains a small fraction of particles greater than 2 mm and the distribution of contaminants is likely to be homogeneous, grinding may be omitted. In these two cases, suitable test portions are taken directly after mixing the sample. This procedure is described in 8.4.

NOTE 1 To distinguish the volatile organic compounds from the moderately volatile organic compounds, boiling points are used instead of the vapour pressure at ambient temperature. This is explained in Annex A, which also gives boiling points and vapour pressures of compounds regularly determined in soil investigations.

NOTE 2 For some specific components in the group of moderately volatile organic compounds, freeze-drying may give good results. In this International Standard freeze-drying is not described.

5 Reagents

Use only reagents of recognized analytical quality. Check samples of each batch of the reagents for the presence of contaminating compounds.

5.1 Sodium sulfate, anhydrous.

Heat the sodium sulfate before use for at least 6 h at about 550 °C to remove crystalline water and organic materials. After heating, allow to cool in a desiccator and store in a closed container.

5.2 Magnesium silicate (talcum powder).

5.3 Sand or gravel.

Before use, wash the sand or gravel at least twice with an equivalent mass of demineralized water and then heat for 6 h at about 550 °C to remove organic materials.

5.4 Liquid nitrogen.

For safety precautions, see warnings in 8.3.2.

6 Apparatus

Usual laboratory glassware and equipment, and in particular:

6.1 Glass containers with a volume of 750 ml to 1 000 ml, with a wide neck and screw cap with a polytetrafluoroethylene (PTFE) inlay.

6.2 Polyethylene containers with a volume of 750 ml to 1 000 ml, with a wide neck and screw cap. To prevent a significant headspace being formed, do not use larger containers.

6.3 Refrigerator, capable of maintaining a temperature below 10 °C.

6.4 Dewar vessel(s), capable of holding at least one polyethylene container of about 750 ml.

6.5 Cross-beater mill or mill with comparable qualities, with a sieve of mesh size 1 mm and accessories. A cross-beater mill as used in most soil laboratories is suitable for milling soil samples cooled with liquid nitrogen.

6.6 Gloves, suitable for working at low temperatures.

6.7 Oven, suitable for heating to about 550 °C.

6.8 Core cutter or similar instrument, for example apple corer. Depending on the type of soil (sand, clay), different instruments can be used.

7 Preservation and storage

Do not preserve the samples, but analyse them as soon as possible. Between sampling in the field and analysis store the samples below 10 °C, if possible in a refrigerator (6.3). The maximum storage time for a sample kept in the dark below 10 °C depends on the parameter(s) to be determined, but can also depend on the material used for the container. Details can be obtained from the relevant specific analytical standards.

The storage time also depends on possibilities of volatization and biological degradation. If these processes are likely to occur, then the storage time should not exceed four days. In cases with no volatization, poorly biodegradable compounds present and biologically less active soil, longer storage times are possible.

If rapid microbial decomposition of the compounds to be determined can be expected, the sample should be pretreated immediately after receipt at the laboratory. For samples containing volatile organic compounds in particular, the storage time should preferably be as short as possible and analysis carried out as soon as possible, e.g. within 1 day to 2 days.

NOTE Chemically dried and ground samples are generally stable for longer periods if stored in a cool, dark location. Freezing samples could extend the useful storage time.

8 Procedure

8.1 General

The pretreatment method depends on the volatility of the substance(s) or group(s) of substances to be determined. Two categories are distinguished here:

a) Volatile compounds: boiling point $<$ 300 °C.

b) Moderately volatile organic compounds: boiling point $>$ 300 °C:

   1) grinding necessary;

   2) grinding not possible or not necessary.

The method for pretreating samples containing volatile organic compounds is described in 8.2.

The method for pretreating samples containing moderately volatile organic compounds using grinding is described in 8.3. If grinding is not necessary or the required result is only qualitative, the method described in 8.4 is used.

If the pretreatment methods differ for various parameters to be determined, divide the sample before pretreatment into subsamples which are as large as possible.

If it is known in advance that both volatile organic compounds and other parameters are to be determined in a sample, it is essential that a separate sample be taken in the field in accordance with the appropriate International Standard.

For the purposes of calculation of the content of volatile and moderately volatile organic compounds on the basis of dry matter, the content of dry matter shall be determined in accordance with ISO 11465 on a subsample of the original (moist) sample.

8.2 Volatile compounds (boiling point < 300 °C)

8.2.1 General

For samples in which volatile compounds are to be determined, no sample pretreatment is carried out. Take test samples from the sample as soon as possible after sampling, to avoid losses.

Test samples can be taken and extracted in the field. Precautions should be taken to prevent contamination of the extraction liquid. This should be verified using field blanks, which are subject to the same procedures as the samples. Otherwise the sample should be covered with the extraction solution, the container tightly closed and transported to the laboratory under cool conditions, to perform the extraction.

8.2.2 Individual samples

Store the samples in the refrigerator (6.3) until further processing. Using a corer (6.8), take one or more soil cores from the container such that the combined mass of the cores corresponds to the required mass of the test sample (see Clause 2 and Bibliography for the respective International Standards).

If several cores of soil are taken and/or if several test samples are taken from the same container, take the cores from different points in the container, if possible.

8.2.3 Composite samples

It is not possible to form composite samples for the analysis of volatile organic compounds. If the analysis method involves a liquid extract, a composite extract can be prepared by mixing equivalent volumes of the extracts from the different samples.

8.3 Moderately volatile organic compounds (boiling point > 300 °C) — Grinding necessary

8.3.1 General

Remove parts that are not representative of the soil, e.g. all visible metallic content.

A note shall be made to this effect in the analytical report.

8.3.2 Individual samples

8.3.2.1 Chemical drying

For each sample to be analysed, add approximately 200 g of sodium sulfate (5.1) and approximately 50 g of magnesium silicate (5.2) to a glass container (6.1). Determine the total mass of these substances to an accuracy of 0,1 g. After sealing the container, mix the two substances by shaking and cool to a temperature below 10 °C.

For each sample, add approximately 250 g of soil (weighed to 1 decimal point) to the glass container with the sodium sulfate and magnesium silicate.

Close the container and mix the soil and the additives by shaking. Place the container in a refrigerator (6.3) to stand cold for 12 h to 16 h. Shake the container vigorously every hour for the first 4 h to avoid clod formation. Before the end of the drying time, again shake the samples vigorously.

If large clods, e.g. larger than 3 cm, are formed in the initial phase, these can be crushed manually by cutting with a spatula in the container. This may particularly be necessary with heavy clay soil.

If the moisture content is greater than 60 %, add extra sodium sulfate instead of reducing the amount of sample. If a sample has a low moisture content and no clods are formed, it is possible to dry in less than 12 h. An adequate drying time should be ensured.

The sample should be kept in a cool environment as long as possible, not only before, but also after weighing.

NOTE 1 This procedure is less suitable for the determination of moderately volatile organic compounds in sludges or sediments with a high water content. Chemical drying of such a sample before crushing can cause problems due to insufficient drying and clod formation.

NOTE 2 If the soil is not properly mixed with the additives in the initial phase, large clods can form which do not dry further.

NOTE 3 If a sample is not completely dry before the cryogenic crushing, considerable contamination of the cross-beater mill can occur. In particular, clods which may not be fully dry on the inside (after too short a drying time) have a relatively long retention in the jaws of the crusher. This results in heating of the clod, and the moist matter is spread over the inside of the crusher. This contamination is very difficult to remove and can lead to serious contamination of subsequent samples.

8.3.2.2 Grinding

Fill the Dewar vessels (6.4) to be used with sufficient liquid nitrogen for the polyethylene containers (6.2) to be fully covered by the liquid nitrogen when placed in the Dewar vessels. Quickly transfer the content of each glass container with soil and additives (8.3.2.1) into a polyethylene container. Seal the polyethylene container and immerse completely in the liquid nitrogen. Allow the container to stand until the liquid nitrogen no longer boils vigorously. Cool for approximately 10 min. After complete cooling, retrieve the container from the liquid nitrogen and transfer the contents to the cross-beater mill (6.5).

Place the cross beater mill in a well-ventilated area. After handling each sample, the disintegrator should be cleaned to avoid contamination of subsequent samples. This can be done efficiently by crushing a quantity of clean (uncontaminated) gravel (5.4) and then cleaning the cross-beater mill with a vacuum cleaner.

After crushing, carefully remove the collection tray beneath the cross-beater mill and take the necessary test samples from the crushed soil. When removing the collection tray, take into account the release of fine dust. For this reason, do not remove the tray until a few minutes after the motor has stopped. However, do not wait too long so as to avoid heating the sample. The test sample should be carefully taken from the collection tray. Samples should be taken both in depth and over the (entire) surface so as to ensure as representative as possible a test sample. While removing the collection tray and taking the test sample, the sample should not be shaken as this can cause (further) separation on the basis of particle size and mass.

Weigh the samples and start the required extraction procedure immediately after weighing.

WARNING — Use suitable gloves and face protection when handling liquid nitrogen and soil cooled to – 196 °C.

- **At all times, use personal protection including a dust mask in the case of the release of dust, inhalable quartz, or contaminated matter in the form of dust.**
- **Note that polyethylene containers are fragile at a temperature of – 196 °C.**

NOTE 1 Usually, it takes about half an hour for the soil to be fully cooled in the liquid nitrogen. This cooling period can be lengthened slightly to guarantee complete cooling in the container.

NOTE 2 Due to the better accessibility of the soil to reagents, the result from analysis after cryogenic grinding may be found higher than in the untreated sample.

After analysis, correct the calculation of content for the dry mass and the additives. Carry out the latter correction by multiplying the measured mass by the additive factor f_{t}:

$$m_{\mathrm{s+a}} = m \cdot f_{\mathrm{t}} \tag{1}$$

where

$m_{\text{s+a}}$ is the mass of the sample plus additives;

m is the original mass of the test sample;

f_{t} is the additive factor,

where

$$f_{\text{t}} = \frac{(m + m_{\text{Na}_2\text{SO}_4\text{+talc}})}{m} \tag{2}$$

where $m_{\text{Na2SO4+talc}}$ is the mass of additives.

As the water present in the sample is not removed, then correct the mass of the sample calculated in this way for the dry mass as specified in ISO 11465.

8.3.3 Composite samples

It is not possible to make composite samples from unpretreated samples. For composite samples, follow the method in 8.3.2. After extraction, the extracts of the samples to be mixed are combined in equal amounts into a composite extract or by mixing cryogenically crushed test samples before extraction. Here too, the content shall be corrected for the dry mass and additives. The latter correction can be carried out by multiplying the measured content by the additive factor f_{t} (see 8.3.2).

With composite samples, f_{t} is the average of the additive factors of the individual samples:

$$f_{\text{t}} = \frac{\sum_{i=1}^{n} \left(m + m_{\text{Na}_2\text{SO}_4\text{+talc}}\right)_i}{\sum_{i=1}^{n} m_i} \tag{3}$$

where n is the number of samples that have been combined.

8.4 Moderately volatile organic compounds (boiling point > 300 °C) — Grinding not possible or not necessary

8.4.1 General

For this method, mixing by hand is the only pretreatment procedure. This method may also be used as a qualitative measurement for moderately volatile organic compounds if procedure 8.3 is necessary for accurate results.

8.4.2 Individual samples

Keep samples cool at temperatures below 10 °C as long as possible. The time between removing the samples from the refrigerator and taking a test sample shall be as short as possible. Mix the sample in the container or in a separate vessel. Remove the parts that are not representative of the soil, e.g. all visible metallic components. If possible reduce the particle size by moderate grinding by hand (for instance mortar and pestle). This is possible if the soil contains aggregates composed of more or less weakly cohesive materials and plant residues. Take a representative test sample with a spoon or corer. The accuracy and reproducibility will be better if large test samples are taken (further information can be obtained from the relevant analytical procedure).

In contrast to the test sample prepared according to 8.3, the sample prepared via this method contains free water. This can have an effect on the analytical procedure used after the pretreatment, and should be mentioned in the test report.

8.4.3 Composite samples

If this method is to be used as a qualitative method, preparing composite samples will further reduce the value of the results. Composite samples are preferably not prepared by mixing the soil samples but by equivalent mixing of the extracts from the different soil samples.

8.5 Blank measurements

To determine whether the pretreatment process contaminates the soil samples, carry out a blank measurement. This blank measurement shall be carried out each time a new compound (or compounds group) is to be determined in accordance using a pretreatment method specified in this International Standard, and thereafter as often as required for the purposes of quality control of the analytical results of the laboratory.

To carry out the blank measurement for procedure 8.3, take equal quantities of washed and calcined sand or gravel (5.4) instead of the soil sample, and follow the procedure described.

9 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard;

b) complete identification of the soil sample;

c) the storage time and storage conditions of the soil sample (time between taking the soil sample and pretreatment or, if the time of taking the sample is not known, the time between receipt of the sample by the laboratory and the pretreatment);

d) the method used; that for volatile organic compounds according to 8.2, the method for moderately volatile organic compounds using grinding according to 8.3 or the method for moderate volatile organic compounds if grinding is not possible or not necessary according to 8.4. If 8.4 is used in cases when grinding should be applied, it should be mentioned in the report that the results are only indicative;

e) 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)

Information concerning vapour pressure, boiling and melting points of volatile organic compounds

This Annex gives an overview of volatile organic compounds which are regularly determined in soil investigations, with their associated vapour pressures, boiling and melting points.

The compounds are arranged in Tables A.1 and A.2 by increasing boiling point.

The vapour pressure at 20 °C is an approximation. The *Handbook of Chemistry and Physics* [8] gives the associated temperatures for a number of substances for fixed vapour pressures (1 mmHg, 10 mmHg, 40 mmHg, 100 mmHg, 400 mmHg and 760 mmHg). Where the vapour pressures are given for temperatures above and below 20 °C, linear interpolation is used to determine the vapour pressure, in kilopascals, at 20 °C, which is given in Table A.1 with the boiling and melting points of the compounds concerned. When carrying out the interpolation, a linear relationship has been assumed between the temperature and the vapour pressure over the period around 20 °C. Since only the trend in vapour pressure in relation to the boiling and melting points is of interest, the error in this approximation is not important. If the lowest vapour pressure given (1 mmHg = 0,13 kPa) lies above 20 °C, the temperature at which this vapour pressure occurs and the boiling and melting point of the compound concerned are given in Table A.2, as interpolation is not possible in these cases.

Tables A.1 and A.2 show a clear relation between vapour pressure and boiling point. In contrast, there is no relation between vapour pressure and melting point. As the vapour pressure decreases, the boiling point increases. As the vapour pressure is known only for a limited number of compounds, classification on the basis of volatility is, for practical reasons, better related to the boiling point than to the vapour pressure.

For cryogenic crushing, losses are observed for substances with boiling points below or close to that of hexadecane [7]. Hexadecane is one of the most recent compounds to be clearly indicated in gas chromatographic analysis of volatile hydrocarbons. Thus the boiling point cut-off between compounds in the volatile group and the moderately volatile group lies around 300 °C. In this International Standard, distinction is made between the following two categories:

a) volatile organic compounds: boiling point < 300 °C

b) moderately volatile organic compounds: boiling point > 300 °C

Table A.1 — Volatile compounds with associated vapour pressure at 20 °C, boiling and melting points

Compound	Vapour pressure at 20 °C kPa	Boiling point °C	Melting point °C
Pentane	57,3	36	– 130
Dichloromethane	47,8	40	– 95
1,1-Dichloroethane	29,0	57	– 97
Trichloromethane	24,9	61	– 64
Hexane	18,2	69	– 95
1,1,1-Trichloroethane	13,3	74	– 31
Tetrachloromethane	12,0	77	– 23
Benzene	10,6	80	6
1,2-Dichloroethane	9,4	84	– 35
2-Methylhexane	7,4	90	– 118
3-Methylhexane	6,8	92	– 119
Heptane	4,9	98	– 91
Toluene	3,5	111	– 95
1,1,2-Trichloroethane	3,1	113	– 37
3-Methylheptane	2,4	115	– 121
2-Methylheptane	2,5	118	– 110
Octane	1,5	125	– 57
Chlorobenzene	1,2	132	– 45
Ethylbenzene	1,13	136	– 95
p-Xylene	1,08	138	13
m-Xylene	1,05	139	– 48
o-Xylene	0,93	144	– 25
Nonane	0,75	151	– 54
1,3,5-Trimethylbenzene	0,47	165	– 45
1,2,4-Trimethylbenzene	0,36	170	– 44
Decane	0,24	174	– 30
2-Chlorophenol	0,40	175	7
1,2,3-Trimethylbenzene	0,56	176	– 26

Table A.2 — Volatile compounds with the temperature associated with 0,13 kPa vapour pressure, and boiling and melting points

Compound	Temperature °C	Boiling point °C	Melting point °C
Benzaldehyde	26	178	– 56
Phenol	40	182	41
Butylbenzene	23	183	– 88
Undecane	33	195	– 26
2,4-Dichlorophenol	53	206	45
Naphthalene	53	211	80
3-Chlorophenol	44	213	33
Dodecane	48	216	– 10
2,6-Dichlorophenol	60	219	68
4-Chlorophenol	50	220	42
Tridecane	59	234	– 6
Tetradecane	75	253	6
Pentadecane	92	270	10
2-Chlorobiphenyl	89	274	34
Hexadecane	105	287	19
4-Chlorobiphenyl	96	291	76
Heptadecane	115	303	23
Octadecane	120	316	28
Nonadecane	133	330	32
Anthracene	145	340	218
Phenanthrene	118	340	100

Annex B
(informative)

Validation

Annex B gives results of a validation carried out in the Netherlands. In the protocol used, the following performance characteristics were determined for individual compounds or groups of compounds (see Tables B.1 to B.3):

- variation in content in the original unground sample;
- mean content in the original unground sample;
- variation in content after grinding the sample;
- mean content after grinding the sample.

Results cannot be generalized. Even for a given soil type there is a wide range of contaminated soil samples that are received on a laboratory (from homogeneous to completely heterogeneous). It is impossible to cover all eventualities.

Table B.1 — Measurement of some organochloropesticides and polychlorobiphenyls in a soil sample after different pretreatment procedures (n = 10)

Compound	Pretreated according to 8.2		Pretreated according to 8.3 (grinding)	
	Average content µg·kg^{-1} d.m.[a]	Coefficient of variation %	Average content g·kg^{-1} d.m.[a]	Coefficient of variation %
OCP				
αHCH	57	58	49	33
βHCH	342	64	301	29
γHCH	100	63	84	26
PCB				
PCB 101	117	71	115	26
PCB 153	82	83	62	35
PCB 138	62	69	53	27
PCB 180	76	71	64	31
	CV_{av}[b] = 68 % s_{CVav}[c] = 8 % (Abs.)		CV_{av}[b] = 30 % s_{CVav}[c] = 3,6 % (Abs.)	

[a] d.m. = dry matter

[b] CV_{av} = average coefficient of variation

[c] s_{CVav} = standard deviation in this coefficient of variation

Table B.2 — Measurement of mineral oil in some soil samples after different pretreatment procedures (n = 10)

Sample description	Pretreated according to 8.2		Pretreated according to 8.3 (grinding)	
	Average content mg·kg^{-1} d.m.[a]	Coefficient of variation %	Average content mg·kg^{-1} d.m.[a]	Coefficient of variation %
Clay with light oil	48	30	34	39
Clay with heavy oil	41	21	61	25
Sand with light oil	102	40	180	49
Sand with light oil	424	26	603	22
Clay with light oil	680	15	758	23
Clay with heavy oil	1 890	45	2 460	42
Sand with heavy oil	3 670	32	3 610	34
	CV_{av}[b] = 26 % s_{CVav}[c] = 14,6 % (Abs.)		CV_{av}[b] = 33 % s_{CVav}[c] = 10,5 % (Abs.)	

[a] d.m. = dry matter

[b] CV_{av} = average coefficient of variation

[c] s_{CVav} = standard deviation in this coefficient of variation

Table B.3 — Measurement of polycyclic aromatic hydrocarbons (PAHs) in soil samples after different pretreatment procedures

Sample description	Pretreated according to 8.2		Pretreated according to 8.3 (grinding)	
	Average content mg·kg^{-1} d.m.[a]	Coefficient of variation %	Average content mg·kg^{-1} d.m.[a]	Coefficient of variation %
Soil	33,1	23	33,5	12
Soil + straw[b]	45,9	4,5	38,5	16

NOTE For soil sample, n = 10; for soil sample mixed with straw, n = 5. PAHs are expressed as the 10 PAHs from the national list.

[a] d.m. = dry matter

[b] After grinding, separation of the sample occurs due to the differences in density of the soil and the straw

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