

CEN/TC ...

Date: 2006-03

prEN XXX:2006

CEN/TC ...

Secretariat: ...

Soil quality — Guidance for sample pretreatment for determination of organic contamination in soil, sludges and treated biowaste

Einführendes Element — Haupt-Element

Élément introductif — Élément central

ICS:

Descriptors:

Contents

Page

Foreword.....	3
Introduction	4
1 Scope.....	5
2 Normative references.....	5
3 Terms and definitions	6
4 Principle.....	6
4.1 Sampling.....	6
4.2 Volatile compounds	6
4.3 Moderately volatile compounds	6
5 Reagents.....	9
5.1 General	9
5.2 Sodium sulfate anhydrous.....	9
5.3 Magnesium silicate (Talcum powder).....	9
5.4 Sand or gravel	9
5.5 Liquid nitrogen	10
6 Apparatus	10
7 Procedure	10
7.1 General	10
7.2 Preservation	11
7.3 Drying	11
7.4 Homogenisation (grinding, milling and sieving).....	11
7.5 Volatile compounds (boiling point < 300 °C).....	11
7.5.1 General	11
7.5.2 Individual samples	12
7.5.3 Composite samples	12
7.6 Moderately volatile organic compounds (boiling point > 300 °C) - Grinding necessary.....	12
7.6.1 General	12
7.6.2 Individual samples	12
7.6.3 Composite samples	14
7.7 Moderately volatile organic compounds (boiling point > 300 °C) - Grinding not possible or not necessary.....	14
7.7.1 General	14
7.7.2 Individual samples	14
7.7.3 Composite samples	14
7.8 Blank measurements	14
8 Test report	15
Annex A (informative) Information concerning vapour pressure, boiling and melting points of volatile organic compounds	16

Foreword

This document (prEN XXX:2006) has been prepared by CEN/BT TF 151 "Horizontal", the secretariat of which is held by DS.

This document is a working document.

Personal acknowledgement of the author:

This draft standard is based on the preliminary work of Mrs. Marion Gferer from Austria who died in a tragically holiday accident shortly after presenting the report of her desk study on pre-treatment for organic parameters within the consortial project HORIZONTAL. Since this report did not include a draft proposal for a concise European standard covering the matrices soil, sludge and treated biowaste, the author volunteered to prepare such a draft to finalize the work done by Marion Gferer. It was clear from her report that the red thread was stemming on descriptions in ISO 14507. Therefore, this ISO 14507 forms the framework for this draft proposal, and was added by other suitable information from the report and other sources.

It was clear for Mrs. Gferer and also can be seen by this draft proposal that there will be no unique method of sample pre-treatment – neither for organics nor inorganics. Since the material to be investigated may vary greatly in terms of kind and amount of contaminants, size fractions and a lot of other guiding physico-chemical parameters, any pre-treatment principally should follow basic requirements but needs to be adopted to the case.

Together with the report and draft proposal prepared by Frank Brauer of Umweltbundesamt, Dessau, Germany, for pre-treatment of samples for inorganic contaminant analysis, this draft may form the starting document for a merged approach, a pre-treatment description covering all matrices and all contaminants included under project HORIZONTAL.

Andreas Paetz (2006-04-12)

Annexes A is for information only.

Introduction

The properties of organic micro-pollutants may differ greatly according to chemical species:

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

Because of these differences a general pretreatment procedure can not be proposed. The goal of a pretreatment procedure is to prepare a test sample of which the concentration of the contaminant is equal to the concentration in the original solid material, provided, however, that this procedure does not alter the chemical species to be analysed. If the sample contains only small particles and the contaminant is homogeneously distributed it is for instance not necessary to grind the sample. In this European Standard the size 2 mm is used to distinguish between small and large particles. Care should be taken to ensure consistency between the following aspects:

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

Important to pretreatment is the particle size distribution of the sample in relation to the mass of sample taken for analysis. For the analysis of organic contaminants, the 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 (for instance 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 grinded 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 will give lower extraction results. Because 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 be meet the needs. In that case however the result may not be presented as a representative value for the whole sample.

1 Scope

This European Standard specifies three methods for the pretreatment of solid material samples in the laboratory prior to the determination of organic contaminants.

- A method for pretreatment if volatile organic compounds are to be measured.
- A method for pretreatment if moderately volatile to non-volatile organic compounds are to be measured and the result of the following analysis must be accurate and reproducible. The sample contains particles larger than 2 mm and/or the contaminant is heterogeneously distributed.
- A method for pretreatment 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 may also be used if reduced accuracy and repeatability are acceptable.

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

The purpose of the pretreatment described in this European Standard is to prepare a test sample in which the concentration of the contaminant equals the degree of contamination of the original solid material as close as possible or as close as necessary for the investigation. The pretreatment procedure shall be used in combination with an extraction procedure in which the contaminant is available for the extraction liquid.

This European Standard is applicable for samples of which the following organic compounds may need to be analysed:

- Polycyclic aromatic hydrocarbons (PAH);
- Polychlorinated biphenyles (PCB);
- Organochlorine pesticides (OCP);
- Phenols and chlorophenols;
- Phthalates;
- Nonylphenol-Polyethoxylates (NPnE);
- Linear alkylbenzenesulfonates (LAS);
- Polychlorinated dibenzo-*p*-dioxins/furans (PCDD/F);
- Polybrominated diphenylethers.

The applicability of this European Standard with regard to the analysis of pharmaceuticals has not been tested.

NOTE For the pretreatment of samples of soils, sludges and treated biowastes for the purposes of determining non-volatile inorganic compounds and physico-chemical soil characteristics, refer to EN XXXXXX.

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.

prEN XXXXX Pretreatment of samples from soils, sludges and treated biowastes for physico-chemical analysis (in preparation)

3 Terms and definitions

For the purposes of this document, the following apply.

3.1

volatile compounds

Group of organic compounds having a boiling point below 300 °C (at a pressure of 101 kPa)

NOTE 1 These include volatile aromatic and volatile halogenated hydrocarbons. Some mono- and dichlorophenols, for instance, and naphthalene also belong to this group.

3.2

moderately volatile compounds

Group of organic compounds having a boiling point above 300 °C (at a pressure of 101 kPa)

NOTE 1 This group includes:

- a) mineral oil (see prEN XXXXX);
- b) most polycyclic aromatic hydrocarbons (PAH) (see prEN XXXXX);
- c) polychlorobiphenyls (PCB) (see prEN XXXXX);
- d) organochlorine pesticides (see prEN XXXXX).

4 Principle

4.1 Sampling

Sampling shall be carried out according to **ENXXXXX** or other suitable document. After being taken, the samples 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.

NOTE 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.**

4.2 Volatile compounds

For determination of volatile organic compounds, core test samples are taken from the sample and extracted according to the 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 one mm (cryogenic crushing). After grinding suitable test portions are processed according to the specific analytical procedures. Composite samples can be prepared by mixing of the ground samples. This procedure is described in 8.3.

If the extraction procedure prescribes a field moist sample, drying and grinding is not possible.

If the original samples 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 directly taken after mixing of 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. Annex A 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 European Standard freeze-drying is not described.

NOTE 3 For practical reasons the pretreatment for moderately volatile compounds should be prescribed 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 (C₁₀ to C₁₆) 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.

If in accordance with specific EN for determination of organic micropollutants, subsampling, crushing and grinding shall be carried out according to prEN XXXXX (Pretreatment inorganics).

Figure 1 gives a flowchart of steps of pre-treatment from sampling prior to analysis.

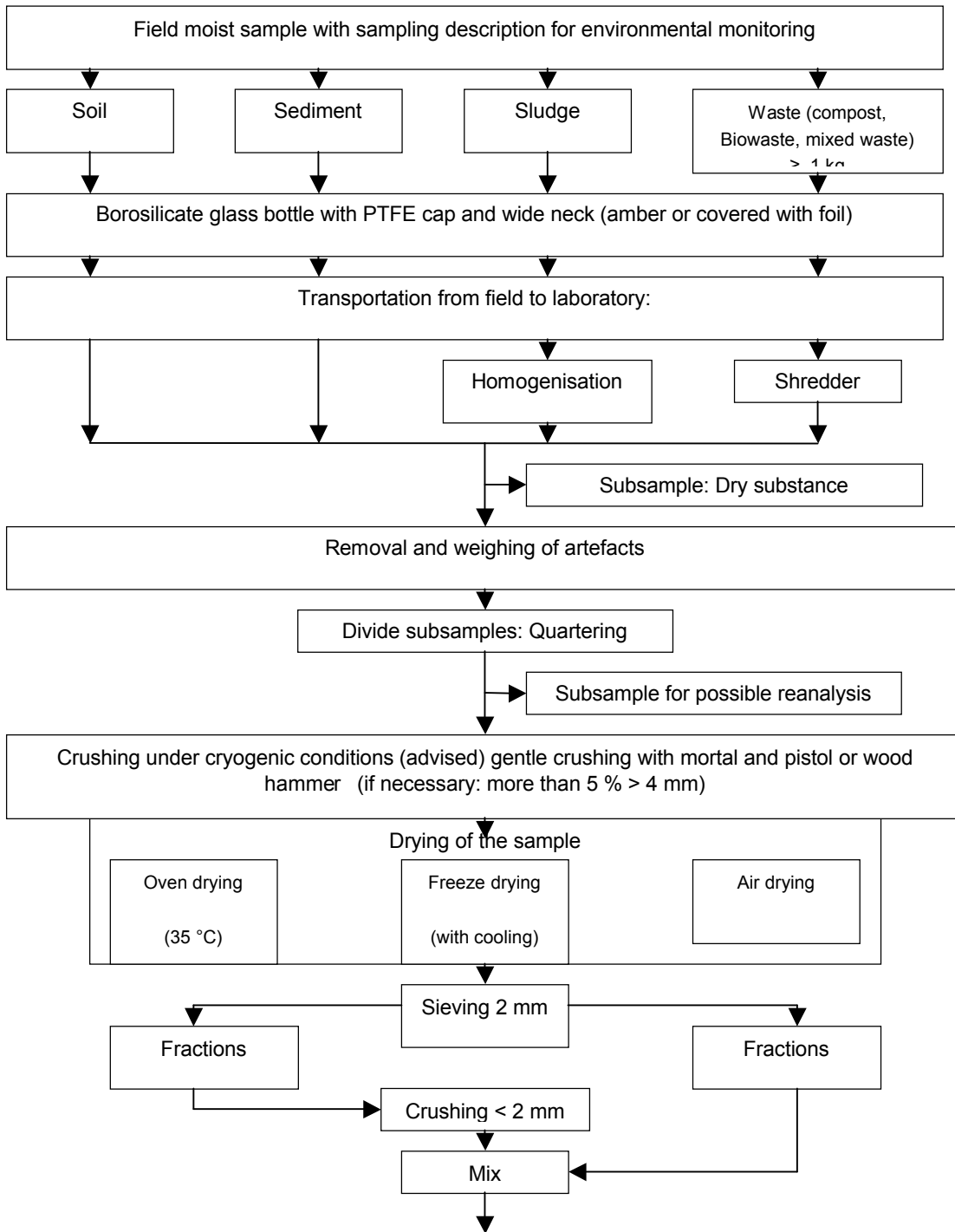


Figure 1 – Diagram for sample pre-treatment (to be continued)

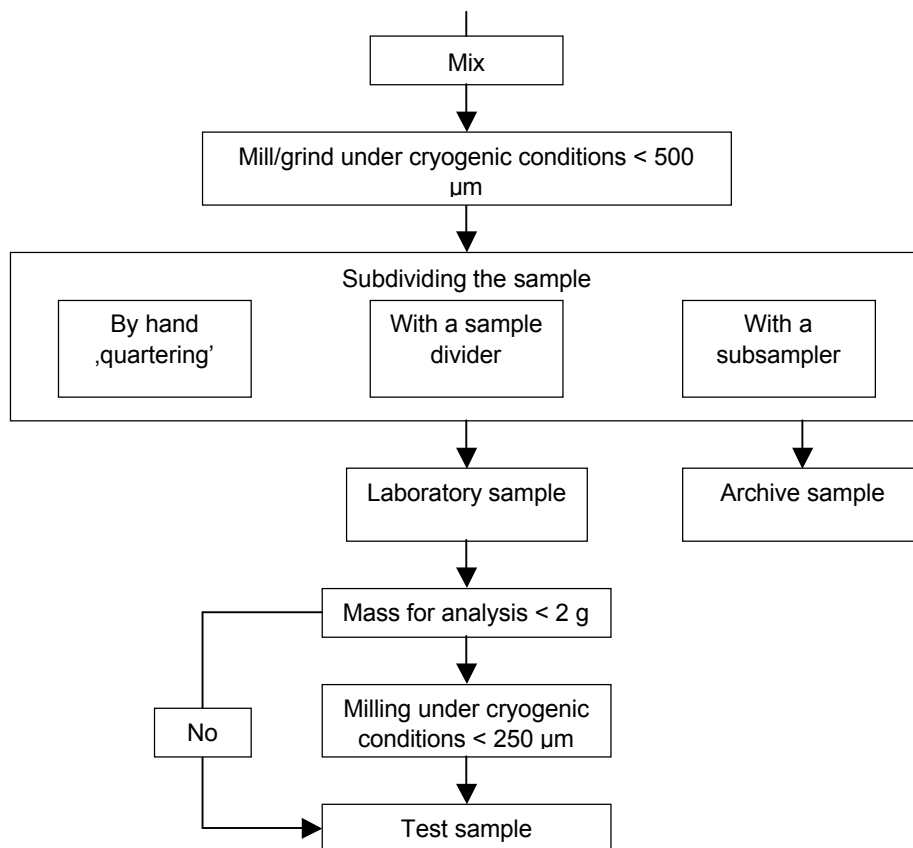


Figure 1 – Diagram for sample pre-treatment.

5 Reagents

5.1 General

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

5.2 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.

5.3 Magnesium silicate (Talcum powder)

5.4 Sand or gravel

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

5.5 Liquid nitrogen

WARNING —

- 1) When handling liquid nitrogen and samples cooled to $-196\text{ }^{\circ}\text{C}$, suitable gloves and face protection shall be used.
- 2) It shall be remembered that polyethylene containers are fragile at a temperature of $-196\text{ }^{\circ}\text{C}$.

6 Apparatus

Usual laboratory glassware and equipment and in particular:

- 6.1 Glass containers with a volume of 750 ml to 1000 ml, with a wide neck and screw cap with a polytetrafluoroethylene (PTFE) inlay.
- 6.2 Polyethylene containers with a volume of 750 ml to 1000 ml, with a wide neck and screw cap. Do not use larger containers to prevent a significant headspace being formed.
- 6.3 Refrigerator, capable to maintain a temperature below $10\text{ }^{\circ}\text{C}$.
- 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 of soil samples cooled with liquid nitrogen.

The cross beater mill shall be placed in a well-ventilated area. At all times, a dust mask shall be used in the case of the release of dust and inhalable quartz. Also, contaminated matter can escape in the form of dust; the personal protection should be designed for this.

- 6.6 Gloves, suitable for working at low temperatures.
- 6.7 Oven, suitable for heating to about $550\text{ }^{\circ}\text{C}$.
- 6.8 Core cutter or similar instrument, for example apple corer.

NOTE When taking a subsample, the quantity of soil should be removed from the 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.

Depending on the type of soil (sand, clay), different instruments can be used.

7 Procedure

7.1 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 $< 300\text{ }^{\circ}\text{C}$.
- b) Moderately volatile organic compounds: boiling point $> 300\text{ }^{\circ}\text{C}$.

- 1) grinding necessary
- 2) grinding not possible or not necessary.

The method for volatile organic compounds is described in 8.2.

The method for moderately volatile organic compounds using grinding is described in 8.3. If grinding is not necessary or the result is only indicative the method is described in 8.4.

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 is taken in the field in accordance with the appropriate 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 ENYYYYY in a subsample of the original (moist) sample.

7.2 Preservation

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 of a sample kept in the dark below 10 °C depends on the parameter(s) to be determined but may also depend on the material used for the container. Details may be obtained from the specific analytical standards.

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

NOTE 1 If rapid microbial decomposition of the compounds to be determined can be expected, the sample should be pretreated immediately after receipt at the laboratory.

NOTE 3 For the group of volatile organic compounds in particular, the storage time should preferably be as short as possible and the analysis carried out as soon as possible e.g. within 1 day to 2 days.

7.3 Drying

Drying procedures for volatile and semi-volatile organic as well as inorganic trace components are critical. Only freeze-drying seems to be advisable for such components, even though, it is recommended that the sample be cooled during lyophilisation to < 10 °C. For non-volatile trace components oven-drying at lower temperatures could be applied.

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

7.4 Homogenisation (grinding, milling and sieving)

Containers and tools for homogenisation can lead to contaminations and losses as in the other analytical steps.

7.5 Volatile compounds (boiling point < 300 °C)

7.5.1 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 to avoid losses.

NOTE Test samples may 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.

7.5.2 Individual samples

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

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

7.5.3 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.

7.6 Moderately volatile organic compounds (boiling point > 300 °C) - Grinding necessary

7.6.1 General

Remove parts that are not representative for the soil e.g. all visible metal parts.

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

7.6.2 Individual samples

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 with an accuracy of 0,1 g. After sealing the container mix the two substances by shaking and cool to a temperature of 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 sulfide.

Close the container and mix the soil and the additives by shaking. Place the container in a refrigerator (6.3). Shake the container vigorously every hour for the first 4 h to avoid clod formation. Leave the containers to stand cold for 12 h to 16 h.

NOTE 1 If the moisture content is greater than 60 %, extra sodium sulfate is added instead of reducing the amount of sample.

NOTE 2 This section of this European Standard 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 3 The sample should be kept in a cool environment as long as possible, not only before, but also after weighing.

NOTE 4 If the soil is not properly mixed with the additives in the initial phase, large clods can form which do not dry further.

NOTE 5 If large clods are formed in the initial phase e.g. larger than 3 cm, these can be crushed manually by cutting with a spatula in the container. This may particularly be necessary with heavy clay soil.

NOTE 6 If a sample has a low moisture content and no clods are formed, it is possible to dry for less than 12 h. An adequate drying time should be ensured.

NOTE 7 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 the 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.

Before the end of the drying time, the samples are again shaken vigorously.

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 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 content to the cross beater mill (6.5).

After crushing, carefully remove the collection tray beneath the cross beater mill and take the necessary test samples from the crushed soil. Start the prescribed extraction procedure immediately after weighing.

NOTE 8 Usually, it takes about half an hour for the sample to be fully cooled in the liquid nitrogen. This cooling period can be lengthened slightly to guarantee complete cooling in the container.

NOTE 9 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.

NOTE 10 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.

NOTE 11 After each sample, the disintegrator should be cleaned to avoid contamination of the following 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.

NOTE 12 As a result of the better accessibility of the soil due to cryogenic grinding, the analysis result after cryogenic grinding may be found higher than in the untreated sample.

After analysis correct the calculation of contents for the dry weight and the additives. Carry out the latter correction by multiplying the measured content by the additive factor f_t :

$$\boxed{} \quad (1)$$

where:

Q_m is the content present in the sample;

Q is the content measured in the test sample;

f_t is the additive factor.

with:

$$f_t = \frac{[\text{mass (sample+sodiumsulfate+magnesium silicate)}]}{\text{mass sample}} \quad (2)$$

As the water present in the sample is not removed then correct the content in the sample calculated in this way for the dry weight as specified in **ENYYYYY**.

7.6.3 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 equivalently into a composite extract or by mixing cryogenically crushed test samples before extraction. Here too, the content shall be corrected for the dry weight and additives. The latter correction can be carried out by multiplying the measured content by the additive factor f_i (see 8.3.2).

With composite samples f_i is the average of the additive factors of the individual samples:

$$f_i = \frac{\sum_{i=1}^n [\text{mass}_i(\text{sample+sodiumsulfate+magenesium silicate})]}{\sum_{i=1}^n \text{mass}_i \text{ sample}} \quad (3)$$

where n is the number of samples combined.

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

7.7.1 General

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

7.7.2 Individual samples

Samples shall be kept cool 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 for the solid material, e.g. all visible metal parts. If possible reduce the particle size by moderate grinding by hand (for instance mortar and pestle). This is possible if the sample 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 larger test samples are taken (further information may be obtained from the specific analytical procedure).

NOTE In contradiction to the test sample prepared according to 8.3 the sample from this method contains free water. This may have an effect on the analytical procedure used after the pretreatment and should be mentioned in the test report.

7.7.3 Composite samples

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

7.8 Blank measurements

To determine whether the pretreatment process causes contamination in the samples a blank measurement shall be carried out. This blank measurement shall be carried out each time a new compound (or compounds group) is to be determined following a pretreatment method specified in this European Standard, and then 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.

8 Test report

The test report shall at least contain the following information:

- a) a reference to this European Standard;
- b) complete identification of the sample;
- c) the storage time and storage conditions of the sample (time between taking the 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 European 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 A gives an overview of the volatile organic compounds with associated vapour pressure, boiling and melting points.

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

Compounds, which are regularly determined in soil investigations, have been listed. The vapour pressure at 20 °C is an approximation. The Handbook of Chemistry and Physics gives the associated temperatures for a number of substances for fixed vapour pressures (1 mm, 10 mm, 40 mm, 100 mm, 400 mm and 760 mm Hg). In the case where the vapour pressures are given for temperatures above and below 20 °C, linear interpolation is used to determine the vapour pressure in kPa 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. In view of the fact that 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 mm Hg (= 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 (see [1] in Bibliography). Hexadecane is one of the last compounds to be clearly indicated in gas chromatographic analysis of volatile hydrocarbons.

This results in the limit in boiling point between compounds in the volatile group and the moderately volatile group lying around 300 °C. Thus 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	97
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

