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Sludge, treated biowaste and soil — Guidance for sample pretreatment

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Sludge, treated biowaste and soil - Guidance for sample pretreatment

Boues, bio-déchets traités et sols - Lignes directrices pour le prétraitement des échantillons

Schlamm, behandelter Bioabfall und Boden - Anleitung zur Probenvorbehandlung

This European Standard was approved by CEN on 23 June 2012.

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Foreword

This document (EN 16179:2012) has been prepared by Technical Committee CEN/TC 400 “Project Committee - Horizontal standards in the fields of sludge, biowaste and soil”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This document has been developed upon existing International Standards for the pretreatment of soils of specified particle fractions.

Historically this has been the case for analysing organic compounds after pretreatment according to e.g. ISO 14507. Standards describing pretreatment for chemical and physico-chemical parameters, e.g. ISO 11464, historically have divided the samples into fractions < 2 mm and > 2 mm where the fraction < 2 mm was taken for testing. By this the concentrations reported for organic compounds could be related to another part of the sample than those of the chemical and physico-chemical parameters. This European Standard stems on the assumption that the same part of the original sample is used for all parameters to be analysed. For environmental investigation it is assumed that generally the whole sample is of interest and will be pre-treated. Only extraneous materials may need to be removed under specific circumstances (and usually then will be reported accordingly).

Depending on legislative or other demands only specific fractions of the sample may be analysed (e.g. the fraction < 2 mm). In that case, the sample will be sieved prior to the pretreatment and the laboratory will report that only the fraction < 2 mm was analysed.

The pretreatment procedures described in this European Standard are not applicable if they affect the results of the determinations to be made. For example, the properties of the parameters to be analysed may differ greatly depending on 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 UV-degradable;

— they may have considerable different solubilities in water.

Some properties of chemical species require different analytical procedures.

Because of these differences, it is not possible to specify one general pretreatment procedure to fit all materials and goals of investigation. The aim of a pretreatment procedure is to prepare a test sample of which the content of a substance or a characteristic is equal to the original material, provided that the applied pretreatment procedure does not considerably alter the characteristic or the chemical nature of the substance to be analysed. It should be noted that every type of pretreatment will have an influence on several material properties.

Important for both sampling and pretreatment are the particle size distribution and form and the degree of chemical heterogeneity of the sample in relation to the minimum required mass of the sample. In general it can be stated that the smaller the particle size and form, and the less the chemical heterogeneity of the original material, the less sample mass is required for a reliable test or – the other way around – the coarser the particle size or the greater the range of particle size and forms, and the greater the chemical heterogeneity might be, the bigger the (sub)sample mass needs to be in order to perform a reliable test. Clause 5 and 8.3 deal with this subject.

WARNING — Persons using this European Standard should be familiar with usual laboratory practice. This European Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this European Standard be carried out by suitably trained staff.

1 Scope

This European Standard specifies the pretreatment required for sludge, treated biowaste and soil (including soil-like materials), that are subject to the analysis of organic as well as inorganic chemical and physico-chemical parameters.

The pretreatment of samples aims at preparing a (small) test sample which is representative for the original sample.

This European Standard describes the pretreatment which could be performed under field conditions if necessary (see Clause 8) and the sample pretreatment under laboratory conditions (Clause 10).

For determining inorganic chemical and physico-chemical parameters this European Standard describes procedures (see 10.2) to prepare:

— test samples for tests under field moist conditions;

— test samples for testing after drying, crushing, grinding, sieving etc.;

— test samples of liquid sludge.

For determination of organic compounds three pretreatment methods are specified:

— a pretreatment method if volatile organic compounds are to be measured (see 10.3.2);

— a pretreatment method if moderately volatile to non-volatile organic compounds are to be measured and the result of the following analysis will be accurate and reproducible (see 10.3.3);

— a pretreatment method if moderately volatile to non-volatile organic compounds are to be measured and the extraction procedure prescribes a field moist sample or if only indicative results are required (see 10.3.4).

The choice of the method depends above all on the volatility of the analyte. It also depends on the particle size distribution of the material (see Clause 5 and 8.3), the heterogeneity of the sample and the following analytical procedure.

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.

EN 15933, *Sludge, treated biowaste and soil — Determination of pH*

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN ISO 5667-13, *Water quality — Sampling — Part 13: Guidance on sampling of sludges (ISO 5667-13)*

EN ISO 5667-15, *Water quality — Sampling — Part 15: Guidance on the preservation and handling of sludge and sediment samples (ISO 5667-15)*

EN ISO 16720, *Soil quality — Pretreatment of samples by freeze drying for subsequent analysis (ISO 16720)*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 10381-8, *Soil quality — Sampling — Part 8: Guidance on sampling of stockpiles*

ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
composite sample
average sample
aggregated sample
two or more increments/subsamples mixed together in appropriate proportions - either discretely or continuously (blended composite sample) - from which the average value of a desired characteristic may be obtained

[SOURCE: ISO 11074:2005, 4.3.3]

3.2
extraneous material
materials not belonging to the matrix or particle fraction to be analysed

Note 1 to entry: Due to the variety of the matrices covered by this European Standard the definition of extraneous material is very broad. The decision to remove certain parts of the material depends on the analytical task.

3.3
field sample
quantity (mass or volume) of material obtained through sampling without any subsampling

[SOURCE: EN 14899:2005, 3.3]

3.4
increment
sampling unit collected by a single operation of a sampling device and being used in a composite sample

Note 1 to entry: When an individual portion of material is collected in a single operation of a sampling device and this portion is analysed as an individual unit, it is by definition a sample.

[SOURCE: ISO 11074:2005, 4.1.8]

3.5
laboratory sample
sample intended for laboratory inspection or testing

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, mixing, grinding or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or analysis.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

Note 3 to entry: Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes.

[SOURCE: ISO 11074:2005, 4.3.6]

3.6
maximum particle size
D_{95}
particle size that concurs with the mesh width of a sieve on which a maximum of 5 % (mass fraction) of the material remains

3.7
particle size reduction
grinding or crushing the sample in order to reduce the particle size of the whole (sub)sample without reducing the sample size (mass)

[SOURCE: ISO 11074:2005, 4.6.4]

3.8
sample
portion of material selected from a larger quantity of material

Note 1 to entry: The manner of selection of the sample should be described in the sampling plan.

[SOURCE: ISO 11074:2005, 4.1.16]

3.9
sample pretreatment
collective term for all procedures used for conditioning a sample to a defined state which allows subsequent examination or analysis or long-term storage

Note 1 to entry: Sample pretreatment includes, e.g. grinding, mixing, splitting, drying, crushing, stabilization.

3.10
sampling plan
predetermined procedure for the selection, withdrawal, on-site pretreatment, preservation, transportation and preparation of the portions to be removed from a population as a sample

[SOURCE: ISO 11074:2005, 4.1.22]

3.11
subsample
sample taken from a sample of a population

Note 1 to entry: A subsample may be:

a) portion of the sample obtained by selection or division;

b) an individual unit of the lot taken as part of the sample;

c) the final unit of multistage sampling.

Note 2 to entry: The term ’subsample’ is used either in the sense of a ’sample of a sample’ or as synonym for ’unit’. In practice, the meaning is usually apparent from the context or is defined.

[SOURCE: ISO 11074:2005, 4.1.30]

3.12
subsampling
sample division
process of selecting one or more subsamples from a sample of a population

[SOURCE: ISO 11074:2005, 4.6.9]

3.13
test sample
analytical sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

3.14
test portion
analytical portion
quantity of material, of proper size, for measurement of the concentration or other property of interest, removed from the test sample

Note 1 to entry: The test portion may be taken from the field sample or from the laboratory sample directly if no preparation of the sample is required (e.g. with liquids), but usually it is taken from the prepared test sample.

Note 2 to entry: A unit or increment of proper homogeneity, size, and fineness, needing no further preparation, may be a test portion.

[SOURCE: ISO 11074:2005, 4.3.13]

4 Safety remarks

Special precautions should usually be taken for samples from contaminated material. It is important to avoid any contact with the skin and special measures should be taken when drying such samples (ventilation, air removal, etc.). Samples may be hazardous because of the presence of chemical contaminants, fungal spores, or pathogens such as *leptospirosis*.

Appropriate national safety precautions shall be followed.

5 Principle

5.1 General

Pretreatment in this European Standard is the process of preparing the test portion from the field sample. The operations and treatment steps are divided into pretreatment procedures suitable in the field (Clause 8) and pretreatment procedures that are restricted to be performed in the laboratory (Clause 10).

It is important to note that the methods and instruments described are meant as examples for suitable routine procedures. A laboratory may decide to use other procedures and/or instruments as long as the requirements of the analytical task are met (see Clause 10).

Another important item concerns the part of the sample delivered to the laboratory which shall be taken for the determinations and the part of the sample which is removed before starting the tests. Depending on legislative or other demands only specific fractions of the sample may be analysed (e.g. the fraction < 2 mm). Extraneous materials shall be separated and recorded.

In case parameters are analysed in the fraction < 2 mm usually the results of the tests refer to the fraction < 2 mm. Using the mass fraction > 2 mm and the extraneous material removed from the sample the results also can be calculated referring to the mass of the whole sample. The report shall clearly state to which fraction the results refer.

Beside the requirements given in this European Standard, the subsequently applied analytical standards shall be regarded for particular requirements to be observed. The determination of some parameters requires sample pretreatment soon after sampling as specified in the respective analytical methods.

If several parameters are being investigated, the sample pretreatment shall be designed so that the parameters of major importance are determining the pretreatment. If this is not possible, e.g. the required precision for each parameter can not be achieved, separate pretreatment shall be set up for each group of parameters.

Whenever volatile compounds are to be determined, the process of sample pretreatment can result in a substantial loss of these compounds. Sample pretreatment shall be omitted in these cases by taking specific samples for the determination of volatile components. These samples shall be pretreated in accordance with the appropriate analytical standard and analysed as soon as possible after sampling.

When preparing composite samples the analytical requirements shall be considered. For example, composite samples are not appropriate if volatile compounds are to be determined.

Figure 1, Figure 2 and Figure 3 show flow charts for:

— pretreatment for inorganic and physico-chemical parameters in solid sludge, treated biowaste and soil (Figure 1),

— pretreatment for organic parameters in solid sludge, treated biowaste and soil (Figure 2),

— pretreatment for inorganic, physico-chemical and organic parameters in liquid sludge (Figure 3).

Aside from the parameter to be determined, the procedure to be applied also depends on the required minimum mass of the sample to be used in relation to the maximum particle size D_{95} of the sample. For this the relationship described in ISO 10381-8 is used (see Table 2 in 8.3).

Grinding of the sample depends on the mass of the subsample being taken. Table 1 gives the requirements for a wide group of parameters.

For some parameters the requirements stated in 8.3 need not to be followed exactly as the homogeneity of the samples is enough to obtain adequate results for those parameters.

EXAMPLES dry matter, total organic compounds (TOC), pH, EC.

Table 1 — Required maximum particle size (D_{95}) for the determination of several parameters.*(1 of 2)*

Parameter	Matrix[a]	Test portion on dry matter basis	D_{95} required, remarks	Procedure[b]
Ammonium- and nitrate-nitrogen	Solid	> 15 g	No D_{95} requirements, sample as received, no heating and no freeze drying allowed	Fig. 1, Route A
	Sludge		No D_{95} requirements, direct measurement in the liquid phase	Fig. 3
Anions (Cl^- , F^- , Br^- , PO_4^{3-} , SO_4^{2-})	Solid	> 15 g	No D_{95} requirements, sample as receive	Fig. 1, Route A
	Sludge		No D_{95} requirements, direct measurement in the liquid phase	Fig. 3
Adsorbable organic halogens (AOX)	All	5 mg to 100 mg	≤ 250 µm	Fig. 3
Cyanide	Solid	10 g to 40 g	No D_{95} requirements, sample as received, no heating allowed	Fig. 1, Route A
	Sludge		No D_{95} requirements, direct measurement in the liquid phase	Fig. 3
Dry matter fraction	Solid	30 g to 50 g	No D_{95} requirements	Fig. 1, Route A
	Solid, air dried	10 g to 15 g	No D_{95} requirements	
	Sludge	> 0,5 g	No D_{95} requirements	Fig. 3
Electrical conductivity	Solid	20 g	No D_{95} requirements, drying (optional)	Fig. 1, Route C
	Sludge		No grinding, direct measurement in liquid phase	
Loss on ignition (LOI) at 550 °C	All	< 0,2 g	≤ 250 µm	Fig. 1, Route A
		0,2 g to 2 g	≤ 500 µm	
		> 2 g	No D_{95} requirements, dried sample for determining dry matter is taken	
Metals: see trace elements				
Organic matter: see LOI				
Particle size distribution			No pretreatment allowed	
pH	Solid	5 ml	No D_{95} requirements, drying (optional)	Fig. 1, Route A
	Sludge		No D_{95} requirements, direct measurement in liquid phase	Fig. 3

Table 1 *(2 of 2)*

Parameter	Matrix[a]	Test portion on dry matter basis	D_{95} required, remarks	Procedure[b]
Phosphorus (total): see trace elements				
Total organic carbon (TOC)	All	< 0,2 g	≤ 250 µm	Fig. 1, Route C
		0,2 g to 2 g	≤ 500 µm	
		> 2 g	≤ 2 mm	
Total Kjeldahl nitrogen	Solid	< 0,2 g	≤ 250 µm	Fig. 1, Route C
		0,2 g to 2 g	≤ 500 µm	
		> 2 g	≤ 2 mm	
	Sludge	0,1 g to 1 g	No grinding, sample as received	Fig. 3
Total nitrogen (Dumas),	All	< 0,2 g	≤ 250 µm	Fig. 1, Route C
		0,2 g to 2 g	≤ 500 µm	
		> 2 g	≤ 2 mm	
Trace elements Extraction with *aqua regia* or nitric acid	Solid	< 0,2 g	≤ 250 µm	Fig. 1, Route C
		0,2 g to 2 g	≤ 500 µm	
		> 2 g	≤ 2 mm	
	Sludge		No D_{95} requirements, homogenisation with high speed mixer or sonification, then direct digestion	Fig. 3
Organic compounds, volatile (e. g. volatile aromatic and halogenated compounds)	All	See analytical standard	No D_{95} requirements and no homogenisation allowed	Fig. 2 Fig. 3
Organic compounds, moderately volatile	Solid	< 2 g	≤ 500 µm	Fig. 2
		2 g to 10 g	≤ 1 mm	
		> 10 g excluding drying agents	≤ 2 mm	
	Sludge	See analytical standard	No D_{95} requirements, sample as received	Fig. 3

[a] Matrix: “Solid” = solid sludge, treated biowaste, soil. "Sludge" = liquid sludge

[b] Procedure: figure number and option in that figure

5.2 Preservation and storage of samples

The samples shall be kept cool and processed as soon as possible; see ISO 18512 for preservation and storage of soil samples, EN ISO 5667-13 for guidance on sampling of sludge samples and EN ISO 5667-15 for preservation and storage of sludge samples. Treat biowaste samples the same as soil samples regarding storage.

5.3 Pretreatment in the field

When possible, the sample pretreatment should take place in the laboratory, as sample integrity can be best guaranteed under laboratory conditions. Subsampling by the methods given in 8.4.2 can be performed in the field as well as in the laboratory.

In the field, sample pretreatment is restricted to the process of subsampling by sample division. Pretreatment is necessary if

— field samples are too large to be transported to the laboratory, or

— if the amount of material sampled is larger than the amount of material necessary for the test or analysis.

When subsampling is necessary, the relation between the minimum mass of the subsamples and the maximum size of the particles (D_{95}) in the original field sample shall be taken into account (see 8.3).

Samples are divided into subsamples either mechanically or manually. Subsampling methods are described in 8.4.2.

In some cases the soil is strongly aggregated. Such macro aggregates can be reduced by hand (see 8.4.1) otherwise they should be seen as individual "particles".

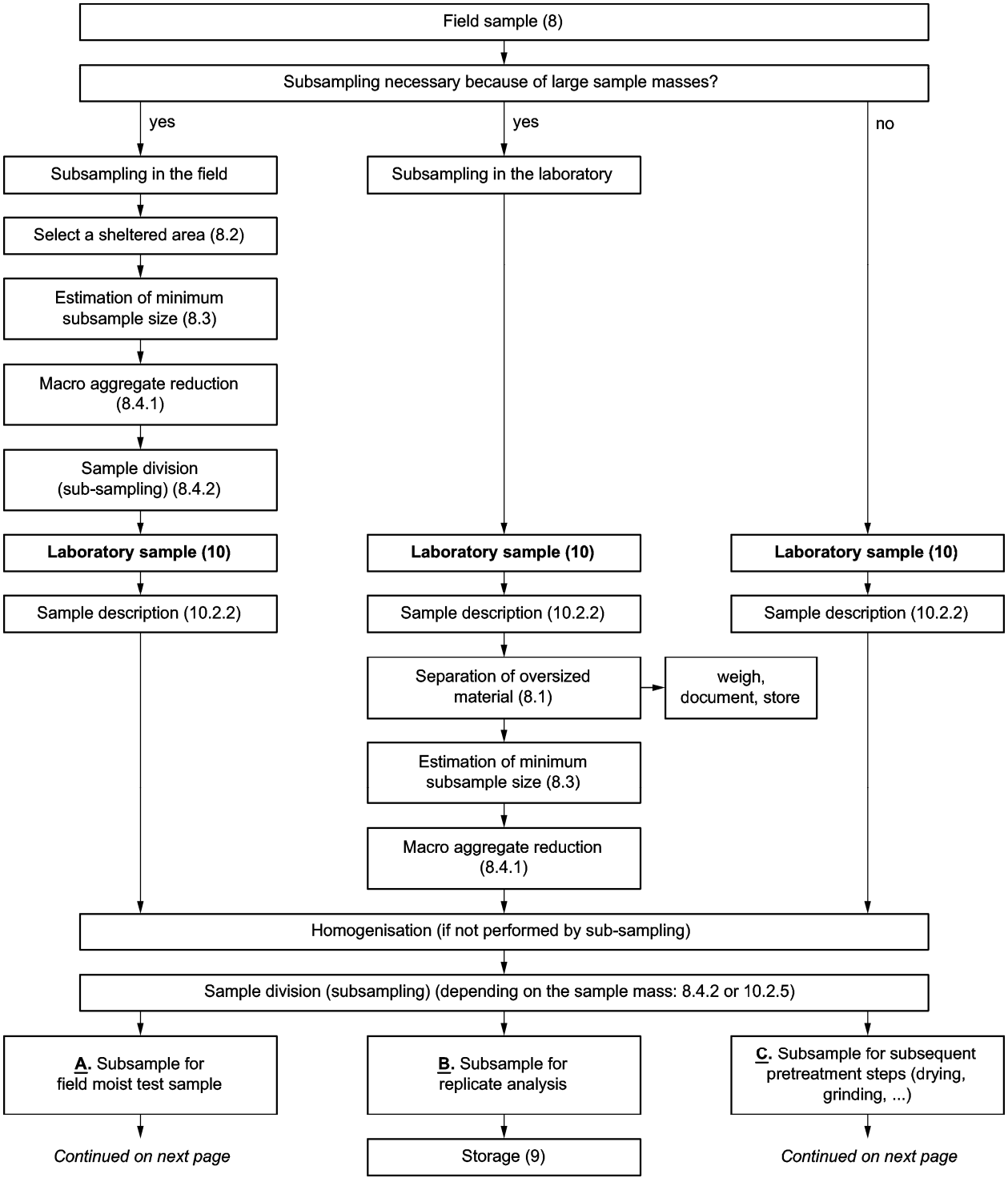

Figure 1 — Flow chart for the pretreatment of solid sludge, treated biowaste and soil for the determination of inorganic and physico-chemical parameters *(1 of 2)*

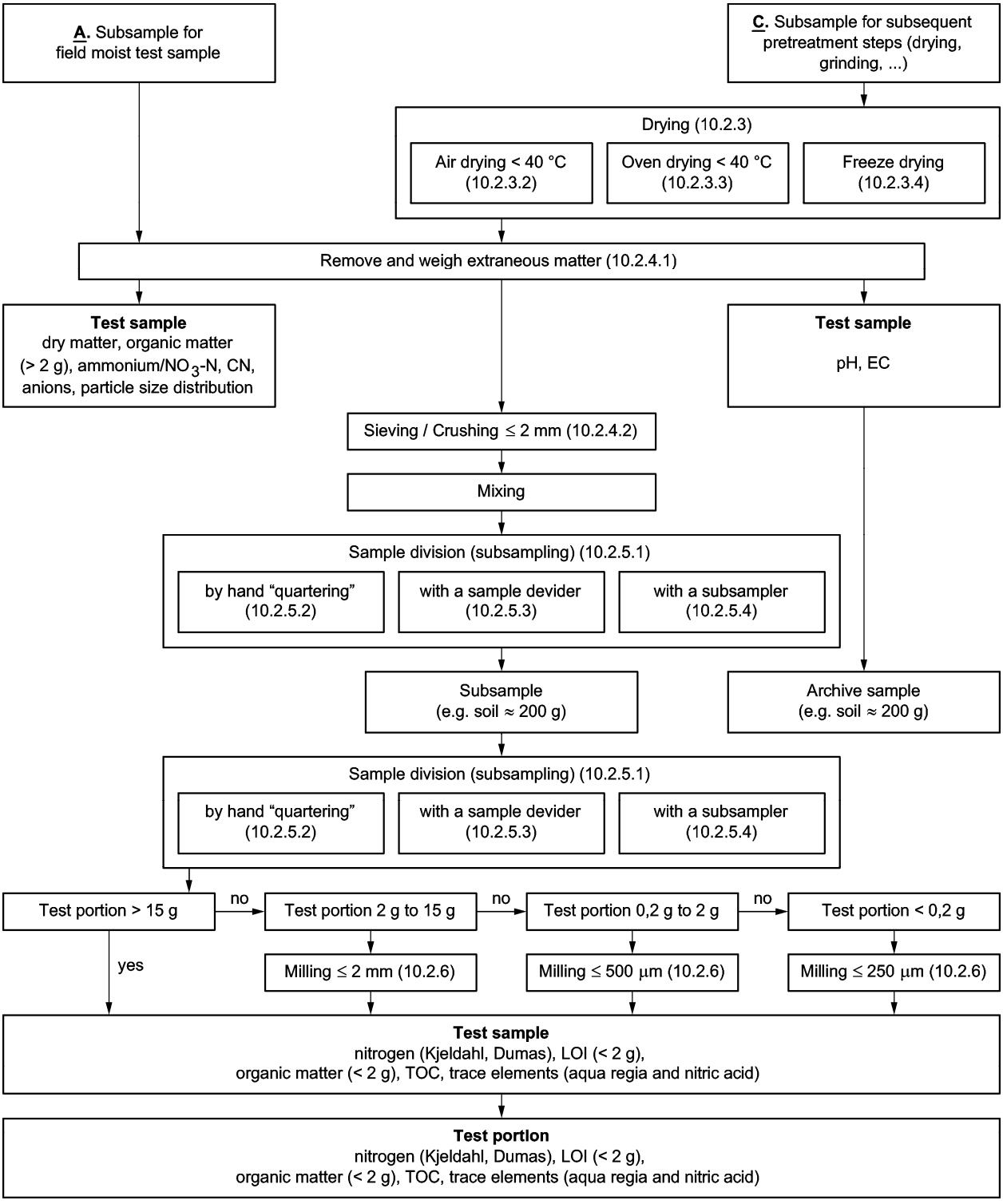

Figure 1 — Flow chart for the pretreatment of solid sludge, treated biowaste and soil for the determination of inorganic and physico-chemical parameters *(2 of 2)*

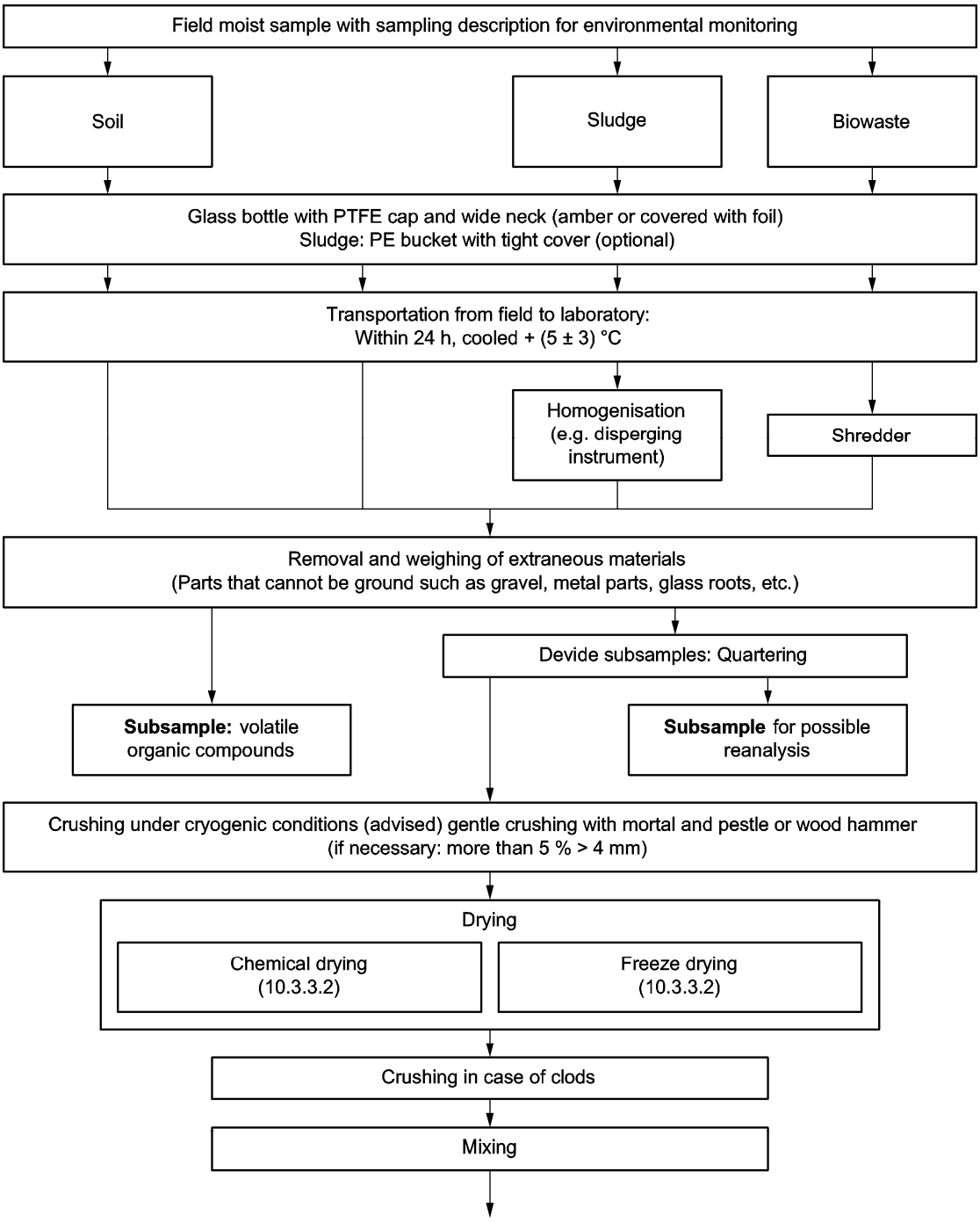

Figure 2 — Flow chart for the pretreatment of solid sludge, treated biowaste and soil for the determination of organic parameters *(1 of 2)*

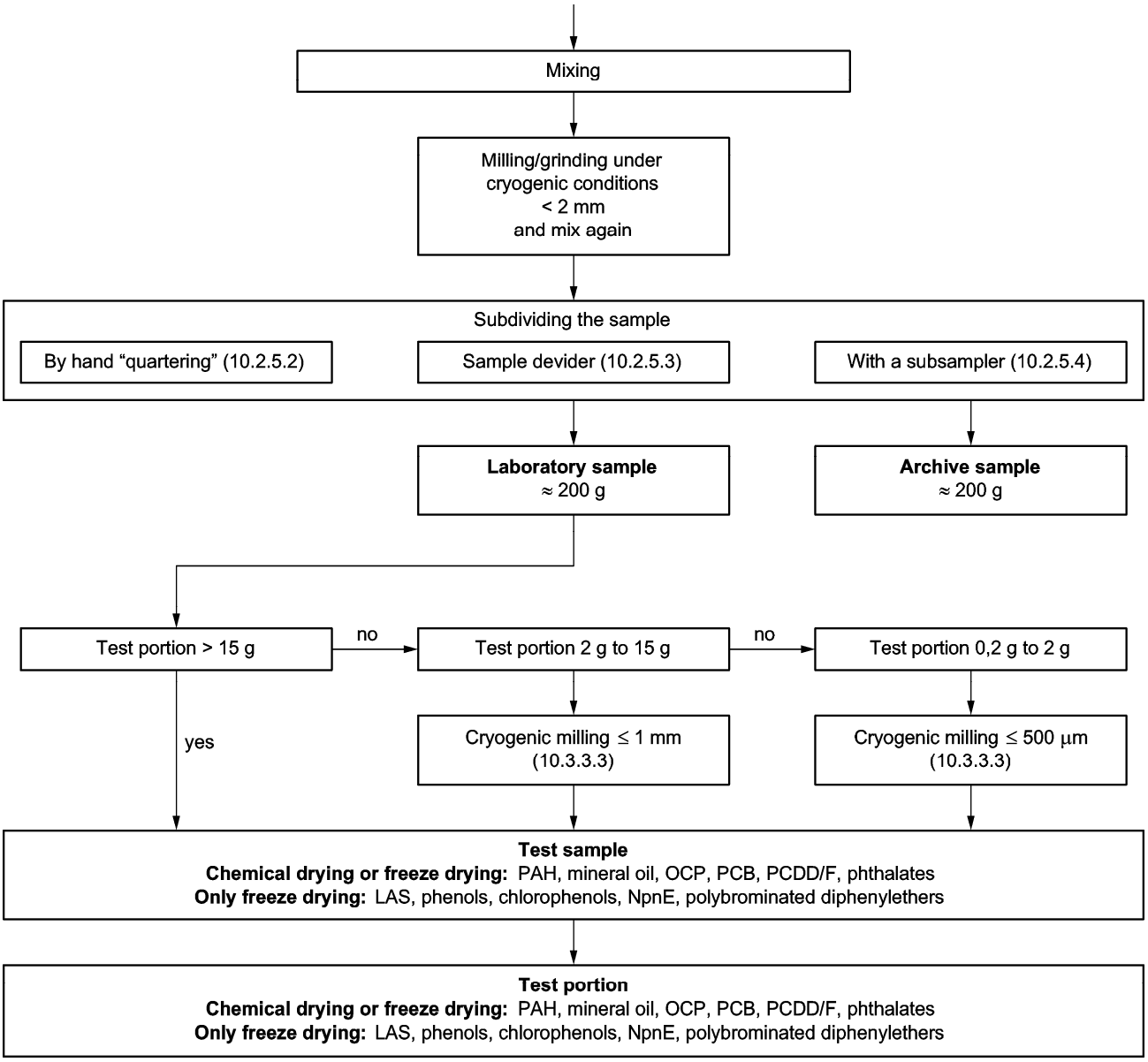

Figure 2 — Flow chart for the pretreatment of solid sludge, treated biowaste and soil for the determination of organic parameters *(2 of 2)*

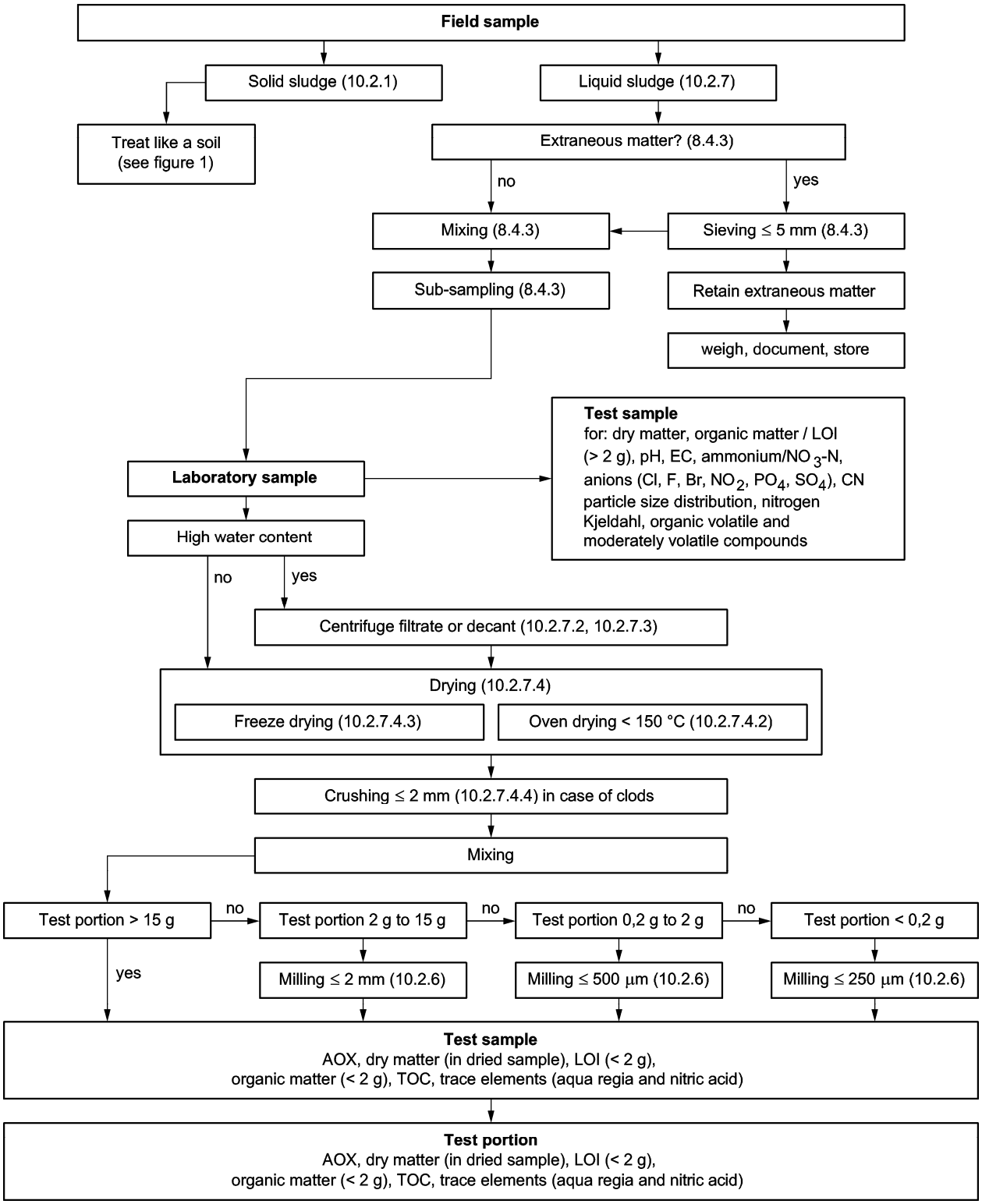

Figure 3 — Flowchart for the pretreatment of liquid sludge samples for the determination of inorganic, organic and physico-chemical parameters

Sample division in the field shall be carried out only if a sheltered area is available (see 8.2).

Soon after sampling, and up until pretreatment in the laboratory, the samples should be stored in such a way that the characteristics of the samples are preserved (e.g. cool and light protected, see Clause 9).

5.4 Pretreatment in the laboratory for determination of inorganic and physico-chemical parameters

Large sample masses which are not subsampled in the field are divided prior to further treatment (see 10.2).

After removal of extraneous materials some of the parameters are analysed directly in the field-moist sample (Route A in Figure 1).

For other parameters (e.g. trace elements) the laboratory samples are dried in the air, or in an oven at temperatures not exceeding 40 °C, or freeze-dried (Route C in Figure 1; see 10.2.3). If necessary, the soil sample is crushed while still damp and friable and again after drying (see 10.2.4). Depending on the required amount of test portion, grinding part of the laboratory sample is required. For liquid sludge see 10.2.7.

When replicate analyses are required, it shall be clarified in the overall investigation plan at which stage of subsampling replicates shall be separated. The most representative stage would be a very early one, e.g. the laboratory sample.

NOTE 1 A drying temperature of 40 °C in an oven is preferable to air drying at room temperature because the increased speed of the drying limits variability caused by to microbial activity.

NOTE 2 Storing samples, including samples that are as received, air dried, refrigerated or stored in the absence of light, for a long time may have an influence on a number of parameters, especially solubility of both inorganic and organic fractions.

NOTE 3 Keeping an archive sample (see Figure 1) is optional and should be clearly stated in the overall description of the investigation programme.

5.5 Pretreatment in the laboratory for determination of organic volatile compounds

The method of pretreatment depends on the volatility of the compound(s) or group(s) of 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 considering the relationship of vapour pressure and boiling point, the boiling point has been chosen as the criterion for distinction. See Annex B.

For determination of volatile organic compounds, test portions are taken from the field 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 10.3.2.

5.6 Pretreatment in the laboratory for determination of moderately volatile organic compounds

Samples are either chemically dried and ground at a low temperature, e.g. with liquid nitrogen or dry ice (6.4), or freeze dried. The dried samples are cooled and cryogenically ground. Direct cryogenical grinding of the sample is also possible, see 10.3.3. Grinding results in suitable test portions. Composite samples are prepared by mixing the ground samples. This procedure is described in 10.3.3.

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

If the original samples only contain 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 10.3.4. If hand mixing is used this shall be stated clearly in the report; a remark shall be made that the results are indicative.

NOTE 1 If a laboratory wants to claim the results to be more than indicative, it should support this in the report by clear reasoning that the sample itself was already homogeneous.

NOTE 2 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 B. Annex B also provides boiling points and vapour pressures of compounds regularly determined in soil investigations.

NOTE 3 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. For freeze drying see EN ISO 16720.

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 of 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. The loss is also 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 presently provides the best results. It is important to realise that some mineral oils (gasoline, petroleum) have high fractions of compounds with boiling points below C10. Losses of these fractions will be severe when using the method described in this European Standard.

6 Reagents

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

6.1 Sodium sulfate, Na_2SO_4, 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.

Heating of the Sodium sulfate at 550 °C may be necessary in order to ensure that it contains no organic compounds. Heating at a lower temperature, e.g. 150 °C during at least 16 h may be also enough to dry the sodium sulfate. It shall be shown that the sodium sulfate is clean enough.

6.2 Magnesium silicate, $Mg_3Si_4O_{10}(OH)_2$, (talcum powder)

6.3 Sand or gravel

Before use, wash the sand or gravel at least twice with an equivalent quantity (same mass) of demineralised water. If the washed sand or gravel proofs to be contaminated, it may be required to heat the material before use; heating for 6 h at about 550 °C to remove organic materials may be necessary. See also the NOTE in 6.1.

6.4 Cooling aid, liquid nitrogen, N_2, dry ice, CO_2

WARNING — When handling liquid nitrogen for the cooling of samples, temperatures of –196 °C can be reached. Suitable gloves and face protection shall always be used in this case as sincere "burning" can occur. Polyethene containers are fragile at very low temperatures.

7 Apparatus

7.1 General

It is essential that the apparatus and tools used for pretreatment do not add or remove any of the substances under investigation. Refer to the related standards for the individual tests for particular requirements.

Due to particle size reduction, contamination of the sample can occur to an extent that affects the leaching of some of the compounds of interest e.g. cobalt and tungsten (from tungsten carbide equipment) or chromium,

nickel and molybdenum (from stainless steel equipment). The laboratory shall show that the equipment used does not significantly increase the concentrations of the compounds to be determined.

NOTE The apparatus to be used is not specified in detail. Most comparable European and National Standards contain detailed equipment specifications which could be used, provided they meet the basic performance requirements indicated in this document.

7.2 Balance, readable and accurate to 1 g.

7.3 Precision Balance, readable and accurate to 0,1 g.

7.4 Analytical balance, readable and accurate to 0,000 1 g.

7.5 Centrifuge, optional.

7.6 Crusher

7.7 Shredder, cutting device

7.8 Drying oven (optional), thermostatically controlled, with forced ventilation and capable of maintaining a temperature not exceeding 40 °C.

7.9 Drying oven (optional), thermostatically controlled and capable of maintaining a temperature of (105 ± 5) °C.

7.10 Freeze-drier, optional.

7.11 Grinding mill, capable of grinding dried materials to a required particle size without contaminating the samples with compounds to be determined.

7.12 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.

If other equipment is used it should be proven that D_{95} is < 1 mm of the ground material. Disc mills have also proven to be suitable for reaching a particle size of < 1 mm or < 500 µm.

7.13 Large heavy-duty plastic sheeting, optional.

7.14 Mechanical mixer(s), optional.

7.15 Mechanical shovel, optional.

7.16 Mechanical sieve shaker, optional.

7.17 Mechanised turntable/rotating dividers, optional.

7.18 Test sieves, complying with ISO 565, with apertures of 0,25 mm, 0,5 mm, 1 mm, 2 mm, and 5 mm to 8 mm for freshly collected samples. Test sieves shall be free of contaminants.

7.19 Pestle and mortar, made of porcelain or sintered corundum.

7.20 Porcelain dish, minimum diameter 30 cm, or bigger.

7.21 Riffle box, optional.

7.22 Sample splitter or utensils for cone and quartering for subsampling of test samples, optional.

7.23 Hammer, optional.

7.24 Spade, optional.

7.25 Spoon, metal and porcelain, optional.

7.26 Tyler divider, optional.

7.27 Wooden or other soft-faced hammer, optional.

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

7.29 Polyethene containers, volume 750 ml to 1 000 ml; wide neck and screw cap.

Do not use larger containers to prevent the formation of significant headspace.

7.30 Refrigerator, capable to maintain a temperature of 1 °C to 5 °C.

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

7.32 Gloves, suitable for working at low temperatures.

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

7.34 Core cutter or similar instrument, for example an apple corer.

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

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

— can be taken quickly.

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

8 Sampling and pretreatment procedures in the field (from field sample to laboratory sample)

8.1 General

Sample pretreatment (in the field or laboratory) is the process of subsampling, necessary to obtain a representative subsample for further measures which shall be carried out under laboratory conditions. A selection of pretreatment techniques suitable for sample division in the field as well as in the laboratory is given in 8.4.

Regarding sample pretreatment in the field the following remarks can be made:

— The requirements for sample pretreatment in the field are the same as for sample pretreatment in the laboratory. In general, the sample pretreatment shall not affect subsequent examinations, i.e. contamination of the sample and/or involuntary loss of material or components shall be avoided.

— The type of sample pretreatment that is allowed in the field is limited to sample division, as the circumstances are in most situations not at all comparable to laboratory conditions. Particle size

reduction – for example by grinding or crushing – shall be avoided since that process requires good defined conditions which can not be achieved in the field. Particle size reduction is restricted to be a laboratory operation.

— One should realise that the quality of sample division (subsampling) in the field is less than the quality of sample division in the laboratory, due to both the (environmental) circumstances for sample division as to the inability to use the best possible division method. When transfer of the sample(s) to the laboratory is possible, this should be considered the preferable option. The methods for sample division described in 8.4 are suitable for pretreatment in the field as well as in the laboratory.

NOTE 1 Only when laboratory conditions are available on site (there is a sample pretreatment laboratory/facility present) the full range of sample pretreatment activities – thus also including particle size reduction – can be carried out directly after sampling.

NOTE 2 If the sample has a dust-like consistency, part of it may be lost and this may alter its physico-chemical properties.

The compounds to be analysed in the sample(s), or the test to be carried out, will in some cases affect the possibilities or methods of subsampling. Therefore the requirements for e.g. pretreatment, preservation and transportation shall be described in the sampling plan and/or communicated by the laboratory.

The methods applied (e.g. sample size reduction) shall be documented and recorded in the test report.

NOTE 3 In most guidelines on sampling for agricultural or similar investigations it is recommended that composite samples are collected by taking a number of increments (e.g. according to ISO 10381-4 at least 25 increments should be obtained) and combining them to form a composite sample.

When preparing composite samples, the analytical requirements shall be considered. For example, composite samples may not be used if volatile compounds are to be determined.

8.2 Selection of workplace

The division of the field sample as received into a number of representative subsamples shall be carried out only when the integrity of the sample and subsamples can be assured. To assure this effectively, a sheltered area is necessary in most situations. Without adequate shelter, weather conditions like wind and rain can pose a serious threat to the quality of the samples. The area should be preferably flat and large enough to allow ease of access around the whole sample when spread evenly on the surface.

It is recommended to protect the sample from contamination by the surface by a clean protective floor covering, preferable heavy-duty plastic sheeting.

NOTE Sample division may also result in significant changes in the composition of the material when no or inadequate precautions are taken. Examples include loss of moisture or volatile components due to evaporation and loss of fine particles due to air entrainment.

8.3 Estimation of minimum subsample mass

The minimum mass of the subsample is determined by the maximum size of the particles (D_{95}) that are present in the sample. When the sample contains macro aggregates, the maximum size of the macro aggregates determines the minimum mass of the subsamples whenever the macro aggregates behave like individual particles during sample pretreatment (that is when macro aggregates will not be cut in pieces by the (sub-) sampling equipment used). See also 8.4.1 for macro aggregate size reduction.

The relationship between the minimum mass of the subsample and the maximum size of the particles (D_{95}) in the original sample is given in Table 2. The relationship is based on the equation for the minimum sample mass as given in ISO 10831-8.

Table 2 indicates that for small particle sizes the minimum mass of the subsample can be very small. For subsampling in the field a minimum amount of about 500 g shall be obtained. Further subsampling will then take place in the laboratory.

NOTE 1 The relationship between particle size and sample mass given in ISO 10831-8 was developed for sampling in the field. In this European Standard this relationship is also used for subsampling in the laboratory.

NOTE 2 Some soils (partly) contain (very) large rocks. When these rocks should be considered as part of the sample, this would result in extremely large samples, both in the field as well as for the material to be transported to the laboratory. Whenever such a situation is encountered, the sampling plan should clearly define the material that is to be sampled/subsampled.

Table 2 — Minimum mass of subsamples as a function of the maximum size (D_{95}) of macro aggregates or particles present in the sample (according to ISO 10381-8:2006)

Maximum particle size (D_{95}) of macro aggregates or particles in the sample mm	**Minimum mass of subsample(s)** g
0,2	0,01
0,4	0,1
0,6	0,4
0,8	0,8
1	2
2	15
4	110
6	360
8	850
10	1 600
12	2 900
14	4 600
16	6 800
18	9 700
20	13 000
22	18 000
24	23 000
26	29 000

The minimum mass of the subsample(s) as given in Table 2 does not necessarily mean that this is the actual mass to be used. Larger masses of subsamples might be needed for analysis, and therefore the mass of the subsample(s) shall be checked with the laboratory.

In order to avoid large sample masses, the mass of the laboratory sample required should be specified in the sampling plan and/or communicated by the laboratory.

For practical reasons, the maximum mass of the samples to be send to the laboratory should be not larger than approximately 20 kg to 30 kg. When larger subsamples are needed because of the large particle size, the particle size should be reduced adjacent to sampling in order to be able to send a representative subsample of an acceptable mass to the laboratory. When grinding or crushing for particle size reduction 'in the field' is necessary, it is only allowed under laboratory conditions, see Clause 10. For these situations either a mobile laboratory or on site laboratory is needed.

8.4 Pretreatment methods

8.4.1 Procedure for macro aggregate reduction by hand

In some cases a sample – especially soil samples – can be strongly aggregated. Macro aggregates should be seen as individual "particles" when the method of sampling and sample pretreatment is not able to sample part of a macro aggregate. For sample pretreatment this happens for instance when a riffle box is used for dividing clay-like soil. As the particle size determines the minimum mass of the subsample(s), it is preferable that the size of macro aggregates can be reduced during or prior to subsampling.

As reduction of macro aggregates by hand will result in a relative long and intense contact of the sample with the air, this method may only be applied when sample integrity is not influenced during this period.

— Identify the maximum size of the macro aggregates, using the minimum mass of the subsample as a starting point as given in Table 2. When the desired mass of the subsample is smaller than a given minimum mass of the subsample, further reduction of the macro aggregate size is necessary.

— Identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow easy access around the whole sample when spread evenly on the surface.

— Place a clean protective floor covering, preferably heavy-duty plastic sheeting, to protect the sample from contamination by the surface.

— Place the sample on the covering/plastic sheeting and spread evenly to identify all macro aggregates within the sample.

— Using the base of a spade or the head of a hammer gently reduce the size of the macro aggregates until all oversized material is less than or equal to the required particle size.

8.4.2 Subsampling methods

A sample may be divided into subsamples either mechanically or manually. If possible, a mechanical system should be used for subsampling, since this results in more representative subsamples. For soil samples this is only applicable when the material is dry and particles can move through a stream of particles on an individual basis.

NOTE 1 This situation can be realised in the laboratory, but is not possible for subsampling in the field directly after sampling (see Clause 10).

NOTE 2 If the particles in the sample behave cohesively, mechanical division is often impossible due to cohesion of soil in the system and subsequent blockage of the divider. Even when the mechanical division is still possible, mechanical subsampling devices will probably function incorrectly, and therefore result in biased subsamples. As a consequence, the manual subsampling methods are often to be preferred for subsampling in the field.

In Annex A the following subsampling methods are described:

— long pile and alternate shovel method, see A.1;

— coning and quartering, see A.2;

— riffling, see A.3;

— application of Tyler divider, see A.4;

— application of mechanised turntable (rotating divider), see A.5.

NOTE 3 The subsampling methods described in Annex A are also suitable for sample division in the laboratory.

The accumulated sample mass is usually too difficult to handle at the laboratory bench. Hence, in order to obtain a representative sample of sludge cake, sample size reduction should be carried out in the field by coning and quartering as described in A.2.

8.4.3 Liquid sludge handling

For some types of liquid sludge, particularly raw sewage sludge, extraneous material, such as rags, may be removed by passing the sample through a stainless steel or plastics screen of aperture size not less than 5 mm.

NOTE It should be remembered that stainless steel contains chromium and nickel. Neither would be expected to be a significant problem in terms of release to the sample, but awareness of the presence of these metals would be prudent when extremes of pH are encountered. With plastics screens, the plasticizer used in manufacture may interfere with biocide analysis.

Extraneous materials may be needed for further examination and should be retained. Some samples may change significantly because of biological activity and it is therefore important that such samples be analysed as soon as possible after collection, see EN ISO 5667-15.

Liquid sludges shall be homogenised before subsampling. The mixing process should preferably be tested to ensure efficiency of mixing. If there is a risk of demixing (e.g. by settling, floating), the subsample shall be taken during the mixing process. The homogenisation can be achieved in a large plastic container using a suitable paddle to prevent settlement.

9 Storing and preservation

9.1 General

Storage begins when the sample is taken. Samples are liable to change in the basic characteristics as a result of various causes. Requirements for the storage and preservation shall be in accordance with the analytical method(s) and shall be described in the sampling plan and/or communicated by the laboratory.

To ensure the integrity and identity of the sample methods, materials and requirements are described for:

— storing the sample(s) prior to transport;

— preserving the sample(s).

Storage of the sample as described in this European Standard only deals with short-term sample storage between sampling and, if applied, sample pretreatment in the field, during transport to the laboratory for further treatment (analysis) and storage in the laboratory.

In most cases sample preservation is achieved by storing them in a dark and cool environment.

In this European Standard general requirements on storage are given to ensure the integrity of the sample when transported to the laboratory.

Guidance for packing, preservation, storage, transportation and delivery of samples are given in ISO 10381-8, ISO 18512 and EN ISO 5667-15.

The methods applied shall be documented and recorded in the test report.

9.2 Appropriate sample container

The purpose of the sample container is to protect the sample, e.g. during transport and storage, until it is further treated or analysed. The type and size of the container shall prevent changes in the sample. See EN ISO 5667-13, EN ISO 5667-15 and ISO 18512 for further details.

Suitable sample containers shall be selected before the beginning of the sampling. Requirements shall be incorporated in the sampling plan.

9.3 Preservation

Storage time between sampling and analysis shall be kept to a minimum to avoid sample alteration. For detailed requirements see EN ISO 5667-13, EN ISO 5667-15 and ISO 18512. Treat biowaste samples like soil samples (ISO 18512).

The method of preservation influences the acceptable storage time between sampling and analysis. It depends on the components to be determined and the storage time prior to analysis.

10 Pretreatment procedures in the laboratory (from laboratory sample to test sample)

10.1 General

This clause contains two sections: sample pretreatment in the laboratory for inorganic chemical and physico-chemical parameters (10.2) and for organic compounds (10.3). Basic requirements for the various tests, and flow charts for the pretreatment processes are given in Clause 5, see especially Table 1 and Figure 1 through Figure 3.

For organic parameters the procedure starts directly with the analysis (volatile compounds) or chemical/freeze drying and grinding (moderately volatile compounds). For chemical and physico-chemical parameters the procedure starts directly with the analysis (e.g. dry matter, organic matter, particle size distribution) or drying and grinding (e.g. determination of elements). Instruments and processes described in this standard shall be seen as examples. A laboratory may decide to use instruments not mentioned in this standard (e.g. milling equipment) and to develop its own pretreatment procedure as long as the following requirements are met:

— the procedure shall lead, in case of heterogeneous samples, to significant improvement of the homogeneity of the sample processed;

— the instruments used may not cause a change in concentration of the parameter to be analysed (i.e. contamination by the materials of the instrument);

EXAMPLE Material of the discs of a disc mill, material of the balls of a ball mill may not cause a contamination.

— the procedure may not cause the loss of the parameter to be analysed, the requirements regarding recovery in the analytical standards shall be met;

— the procedure may not cause the loss of sample material, at least 90 % of the material shall be retained during each sieving, sub-dividing and grinding step;

— the relationship between particle size and sample mass as described in 5.1 and 8.3 and the requirements in Table 1 shall be met;

— if the analytical standard describes specific requirements regarding the pretreatment this may not be changed.

10.2 Pretreatment for determination of chemical and physico-chemical parameters

10.2.1 General

In case of large sample masses subsampling methods prior to further treatment according to 8.4 should be applied in the laboratory in order to reduce the initial sample mass. The relationship between the minimum

mass of the subsample and the maximum size of the particles that are present in the sample shall be taken into account (see Table 2).

Archive and replicate samples should be taken at this stage.

The procedures for drying, fraction separation and size reduction are described in 10.2.3 to 10.2.6. At several stages in the procedure, the laboratory will be required to make decisions, referring in particular to whether size fractions are to be combined or treated separately: this will depend on the nature of the material and the objectives of the analytical programme.

The sample shall be re-homogenised after each separation, sieving, crushing or milling procedure that may have resulted in segregation of different sized particles.

Care should be taken to avoid contamination of the sample through air or dust (e.g. laboratory atmosphere, close approximation to other samples).

Pretreatment should always be performed in a separate room used only for this purpose and remote from locations where analytical measurements are made.

If the sample has a dust-like consistency, part of it may be lost and this may alter its physico-chemical properties.

As long as a sludge sample is not solid it is treated like a liquid sludge sample (see 10.2.7) otherwise like a soil sample (see 10.2.3 through 10.2.6) because high water contents may cause difficulties during the analysis of sludges.

10.2.2 Laboratory sample description

Examine the sample as received and record its appearance, including details of extraneous matter, remains of vegetation, and other noticeable or relevant features.

10.2.3 Drying of solid samples

10.2.3.1 General

The laboratory samples are dried in air, or in an oven at a temperature not exceeding 40 °C. Dry the complete laboratory sample in air or in a ventilated drying oven from which the moist air is removed or in a freeze dryer. Depending on the chosen method of drying, follow the procedure described in 10.2.3.2, 10.2.3.3 or 10.2.3.4. The aim of drying is to produce a material that can be ground with the equipment used. Drying to complete dryness is not necessary for all equipment. After the drying process has been completed, determine and record the total mass of the dried sample.

To accelerate the drying process, break down larger aggregates (larger than 15 mm) during the process. When samples are dried in air, crush them slightly by hand using a wooden hammer or a mortar and pestle, taking great care to avoid contamination. When samples are dried in an oven, temporarily remove them from the oven and treat them in the same way. This procedure also makes it easier to separate the extraneous materials.

Freeze drying rarely dries samples into clods; samples usually disintegrate.

For pH determination according to EN 15933, the sample should be fresh or air dry. For the extraction of trace elements soluble in *aqua regia* according to EN 16174 or soluble in nitric acid according to EN 16173 air-drying or drying at a temperature not higher than 40 °C is recommended.

NOTE 1 Drying can influence the pH of the soil. In some soil samples, particularly those containing sulfides, drying can lower the pH substantially (see EN 15933).

NOTE 2 The drying time depends on the type of material, the thickness of the layer, the initial moisture content of the material and of the air, and on the rate of ventilation. In a drying oven, the drying time for sandy soils is usually not more than 24 h and for clay soils more than 48 h. For soils containing a large proportion of fresh organic matter (e.g. plant roots etc.), 72 h to 96 h may be required.

10.2.3.2 Air drying

Spread all the material, in a layer not thicker than 2 cm to 3 cm, on a tray that does not absorb any moisture from the sample and that does not cause contamination.

It is essential that direct sunlight is avoided and the temperature does not exceed 40 °C.

NOTE Direct sunlight could create large temperature differences in the sample, especially between the partly or completely dried top layer and the lower layers.

10.2.3.3 Oven drying

Spread all the material, in a layer not thicker than 2 cm to 3 cm, on a tray made of material that does not absorb any moisture from the sample and that does not cause contamination. Put the tray in the drying oven (7.7) and dry at a temperature not higher than 40 °C.

10.2.3.4 Freeze drying

Perform freeze drying according to EN ISO 16720.

10.2.4 Removal of extraneous materials and crushing

10.2.4.1 Removal of extraneous materials

Before crushing the sample, which will be necessary if samples, especially soil, have dried into large aggregates, extraneous material shall be removed from the dried field sample. Care should be taken to minimise the amount of fine material adhering to the extraneous matter removed.

The mass of the extraneous material shall be weighed and documented and the removed material shall be kept for any further research that may be performed.

10.2.4.2 Crushing

After separation of extraneous material the sample shall be crushed (7.6) if it contains large dried particles.

10.2.5 Subsampling

10.2.5.1 General

Subsampling is necessary when the sample cannot be stored (laboratory sample and archive sample) or used (test sample) completely, because of its mass. Divide the dried and crushed laboratory sample into representative subsamples according to 10.2.5.2 or 10.2.5.3. For the preparation of a test sample, split up the laboratory sample into representative portions until the required masses of samples are obtained. Avoid the formation of dust as much as possible.

NOTE 1 It could be of advantage to divide large laboratory samples according to 8.4 prior to further subsampling.

NOTE 2 It may be necessary to mill the material between subsampling stages, to ensure homogeneity as the mass of the subsample decreased.

Select the method of subsampling (10.2.5.2, 10.2.5.3 or 10.2.5.4) according to the nature of the sample, the requirements of the subsequent determinations and the equipment available. See Figure 1, Figure 2 and Figure 3 for further guidance and the requirements concerning grinding in Table 1.

10.2.5.2 Subsampling by hand (quartering)

Mix the soil sample thoroughly using a suitable mechanical mixer (7.14) and spread it in a thin layer on a tray made of material that does not influence the composition of the sample. Separate the sample into four equal portions (quadrants). Combine two of the four portions diagonally, rejecting the other two. Repeat this procedure until the desired amount of sample is obtained.

10.2.5.3 Use of the sample divider

A suitable example of a sample divider of the multiple-slot type (riffle box) is described in Annex A. This splits the sample into two equal parts.

NOTE The dimensions of the equipment should be chosen to suit the amount and particle size of the materials to be divided (see Annex A).

10.2.5.4 Mechanical subsampling

A variety of appropriate equipment for subsampling is available, often manufactured according to national standards; it may be used for subsampling in accordance with the appropriate national standard and the manufacturer's instructions.

An example for mechanical subsampling equipment is given in Annex A. This operates according to the following procedure.

Pour the sample into the funnel of the subsampler and screw the sample bottles into place. Start the subsampler. After subsampling, pour the contents of the bottles into other sample containers. Repeat this procedure, if necessary, with the contents of one of the containers until the desired amount of soil is obtained. The material should be rehomogenised between each stage of subsampling. The contents of more than one container may be thoroughly mixed and used for subsequent phases of the subsampling routine.

10.2.6 Milling

Depending on the mass of the subsample to be taken the sample shall be ground, see Table 2 for the maximum particle size related to the mass of the subsample. The mill to be used depends on the particle size to be reached. Ball mills e.g. are suitable for very small particle sizes (< 250 µm), cross beater mills for particle sizes < 1 mm and disc mills for < 500 µm. See 7.11 and 7.12.

NOTE 1 Grinding is designed to give a more homogenous sample from which a subsample is taken and to increase the efficiency of acid extraction by increasing the surface area of the particles for example when analysing trace elements.

NOTE 2 For some soils, experience has shown that there is little difference between the results before and after such grinding. However, it is difficult to predict, with certainty, which soils will behave in this way. Therefore, the user should verify that the use of ground or unground soil yields results suitable for the purpose of the investigation. Whether ground or unground soil has been used should be stated in the test report.

Mill a representative subsample (see 10.2.5) of the dried, crushed and sieved sample.

On a regular basis the laboratory shall prove that the required maximum particle size (D_{95}) is reached, i.e. that > 95 % of the ground material passes the corresponding sieve.

If more than one analysis is to be made, sufficient amount of material shall be ground to the smallest particle size specified, to enable all the analysis to be made on this one test sample.

10.2.7 Liquid sludge samples

10.2.7.1 General

For subsampling of liquid sludge see 8.4.3.

Depending on the parameter liquid sludge samples are analysed with or without drying.

Liquid sludge samples are dried in an oven at a temperature not exceeding 105 °C or freeze dried (see 10.2.7.4.2, 10.2.7.4.3). After drying the sludge sample can be sieved.

NOTE If $N\text{-}NH_3$, PO_4^{3-}, SO_4^{2-}, Cl^-, NO_3^- or NO_2^- are determined the original material is used.

Depending on the kind of subsequent pretreatment, use spoons (7.25) for homogenisation. If heavy metals shall be analysed use a porcelain spoon.

If the sludge sample shall be dried by freeze drying or in the oven and the particles are sedimented remove the supernatant.

10.2.7.2 Centrifugation

Sludge can be centrifuged (7.5) according to the manufacturer's instructions to achieve a lower water content. After centrifugation the remaining water may be discarded unless it is required in order to determine the total contaminant content including water soluble contaminants.

NOTE For some sewage sludges with a high water content, centrifugation may not be suitable because even after 12 h centrifugation particles may still float on the surface, or are still in suspension.

10.2.7.3 Filtration

To obtain a sample with a lower water content the samples can also be filtered through a suction filter. The filter cake can be dried in the oven or by freeze drying afterwards. Some of the inorganic parameters can be analysed directly out of the remaining water.

Depending of the parameter to be analysed, glass fibre filters can be applied. If organic parameters shall be analysed, use glass frits.

10.2.7.4 Drying

10.2.7.4.1 General

Dry the sample in a drying oven or in a freeze drier. Depending on the method of drying, follow the procedures described in 10.2.7.4.2 or 10.2.7.4.3.

10.2.7.4.2 Drying oven

Spread the sludge sample in a porcelain dish (7.20). Put the porcelain dish in the drying oven (7.8) and dry at a temperature of (105 ± 5) °C until the mass is constant.

10.2.7.4.3 Freeze Drying

Perform freeze drying according to EN ISO 16720.

If trace elements shall be analysed, fill the sample into porcelain dishes and carry out the freeze drying.

Freeze drying shall be performed in such a way that evaporation losses of the substances are avoided. In particular, it shall be ensured that the sample is unable to thaw during the freeze drying process. Sewage

sludge with high water content should be partially dewatered by centrifugation prior to freeze drying. The separated centrifugate shall not contain particles.

10.2.7.4.4 Removal of extraneous materials, crushing and milling

Remove extraneous materials.

Mill a representative subsample (7.11, 7.12) to the required particle size. For the requirements see Table 1.

10.3 Pretreatment for determination of organic compounds

10.3.1 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, see 10.3.2.

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

   1) grinding necessary, see 10.3.3;

   2) grinding not possible or not necessary, see 10.3.4.

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 EN 15934 in a subsample of the original (moist) sample.

10.3.2 Volatile compounds

10.3.2.1 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 to avoid losses after removal of extraneous materials.

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.

Some compounds, e.g. naphthalene, concerning to this definition belong to the volatile compounds. Naphthalene however is in practice often analysed together with the group of polycyclic hydrocarbons belonging to the group of semi-volatile compounds. The suitability of the pretreatment method used shall be proven, i.e. the requirements concerning recovery shall be met.

10.3.2.2 Individual samples

Using a corer (7.34) take at least three cores from different points in the container such that the combined mass of the cores corresponds to the required mass of the test sample (see Clause 5 and 8.3 for details).

10.3.2.3 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.

10.3.3 Moderately volatile organic compounds – Grinding necessary

10.3.3.1 General

Remove extraneous materials from the sample. A note shall be made to this effect in the test report.

10.3.3.2 Drying

Either dry the sample by freeze drying or by chemical drying. Perform freeze drying according to EN ISO 16720. Chemical drying is described below.

Chemical drying can be carried out for solid samples like solid sludge, treated biowaste and soil. Liquid sludges cannot be chemically dried as they contain too much water. The use of chemical drying has been proven to be suitable for the analysis of non-polar compounds. Examples are PAH, mineral oil, OCP, PCB.

Naphthalene, because of its boiling point, strictly belongs to the group of volatile organic compounds and can be analysed e.g. together with the volatile aromatic compounds. However naphthalene is also often analysed together with the PAH; the chemical drying process has been proven to be suitable also for Naphthalene.

NOTE 1 For polar compounds the suitability of chemical drying has not been shown yet and should be proven by the laboratory if to be applied.

For each sample to be analysed, add approximately 200 g of sodium sulfate (6.1) and approximately 50 g of magnesium silicate (6.2) to a glass container (7.28) or polyethene container (7.29). 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 (7.30). 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 2 If the moisture content is greater than 60 %, extra sodium sulfate is added instead of reducing the amount of sample.

NOTE 3 This section of this European Standard is less suitable for the determination of moderately volatile organic compounds in sludges or sediments with high water content. Chemical drying of such samples before crushing can cause problems due to insufficient drying and clod formation (only relevant for soil and treated biowaste).

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.

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 6 If a sample is not completely dry before the cryogenic crushing, considerable contamination of the grinding instrument can occur. In particular, clods (which may not be fully dry on the inside after too short a drying time) may have a relatively long retention in the grinding instrument. This results in the heating of the clod and the moist material 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 (7.31) to be used with a sufficient amount of liquid nitrogen for the polyethene containers (7.29) to be fully covered by the liquid nitrogen when placed in the Dewar vessels. Quickly transfer the content of each glass container with sample and additives into a polyethene container. Seal the polyethene container and immerse completely in the liquid nitrogen. Allow the container to stand until the liquid nitrogen no longer boils vigorously. After complete cooling, retrieve the container from the liquid nitrogen and transfer the content to the mill (7.11).

10.3.3.3 Grinding

Grind the sample to the required maximum particle size (see Clause 5 and 8.4) and take the necessary test portions from the ground sample. The test portions shall be carefully taken from the collection tray. Samples shall be taken both in depth and over the (entire) surface to ensure the test portions are as representative as possible. While taking the test portions, the ground sample should not be shaken as this can cause (further) separation on the basis of particle size and mass.

Start the prescribed extraction procedure from the analytical method as soon as possible after grinding. See EN ISO 5667-13, EN ISO 5667-15 and ISO 18512 for maximal storage periods. Chemically dried and ground samples are generally stable for longer periods if stored cool and in the dark. Freezing samples can also extend the storage time. Dried samples are assumed to be stable for at least 4 weeks.

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

When removing the collection tray from the mill, take into account the release of fine dust. Therefore, do not remove the tray immediately after the motor has stopped but wait a few minutes to allow fine particles to settle. However, do not wait too long to avoid warming of the sample.

After each sample, the grinding equipment shall be cleaned to avoid contamination of the following samples. This can be done efficiently by grinding a quantity of clean (uncontaminated) gravel (6.3) and then cleaning the cross beater mill with a vacuum cleaner.

NOTE As a result of increased accessibility of the sample 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 analyte concentrations for the dry matter and the additives. Carry out the latter correction by multiplying the measured content by the additive factor f_t according to Formula (1):

$$Q_m = Q \times f_t \quad (1)$$

where

Q_m is the analyte concentration present in the sample;

Q is the analyte concentration measured in the test sample;

f_t is the additive factor

with

$$f_t = \frac{m_{tot}}{m_s} \quad (2)$$

where

m_{tot} is the mass of the sample with additives (e.g. sodium sulfate (6.1) and/or magnesium silicate (6.2));

m_s is the mass of the sample.

As the water present in the sample is not removed, correct the content in the sample calculated in this way for the dry matter as specified in EN 15934.

10.3.3.4 Composite samples

It is not possible to prepare composite samples from untreated samples as part of the compounds may be lost. If a composite sample shall be prepared either mix the cryogenically ground samples before extraction or combine the extracts of the samples to be mixed equivalently into a composite extract. Here too, the content shall be corrected for dry matter and additives. The latter correction can be carried out by multiplying the measured content by the additive factor f_t according to Formula (3) (see 10.3.3.3).

For composite samples the average of the additive factors of the individual samples is calculated with Formula (3):

$$f_t = \frac{\sum_{i=1}^{n} m_{i,tot}}{\sum_{i=1}^{n} m_{i,s}} \quad (3)$$

where

f_t is the additive factor;

n is the number of samples combined;

$m_{i,tot}$ is total mass of the composite sample in kilograms (kg);

$m_{i,s}$ is the mass of the subsample i in kilograms (kg).

If a composite sample is prepared prior to pretreatment, it shall be stated in the test report that the results are indicative because semi-volatile compounds may (partially) have been lost during the mixing process.

10.3.4 Moderately volatile organic compounds – Grinding not possible or not necessary

10.3.4.1 General

For this method mixing by hand is the only pretreatment procedure. If mixing by hand is applied this shall be stated in the test report.

10.3.4.2 Individual samples

Mix the sample in the container or in a separate vessel. Remove extraneous materials from the sample. If required, e.g. if the sample contains aggregates composed of more or less weakly cohesive materials and plant residues, reduce the particle size by moderate grinding by hand (e.g. with mortar and pestle (7.19)). Take a representative test sample with a spoon or corer. The accuracy and reproducibility will be increased 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 10.3.3 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.

10.3.4.3 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 samples.

11 Test report

The test report shall contain at least the following information:

a) a reference to this European Standard (EN 16179);

b) a complete identification and description of the sample;

c) the date and time of sampling (or, if the time of taking the sample is not known, the time of receipt of the sample by the laboratory);

d) the presence and mass of extraneous material that might have been removed from the sample;

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)

Subsampling methods

A.1 Long pile and alternate shovel method

This subsampling method is suitable for samples exceeding the mass of 100 kg.

Identify the maximum particle size of the sample and determine the minimum mass of the subsample(s) according to Table 1. When the minimum mass of the subsamples is larger than required and the maximum particle size is related to the size of macro aggregates, the macro aggregate size can be reduced according to 8.4.1. The subsampling process shall be stopped when the mass of the subsample is equal to or larger than the minimum mass of the subsample as derived from Table 1.

— Identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow easy access around the whole sample when spread on the surface.

— Place a clean protective floor covering, preferable heavy duty plastic sheeting, to protect the sample from contamination by the surface.

— Shovel the sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. For samples exceeding the mass of 500 kg, the use of a mechanical shovel (e.g. excavator) is recommended.

— When the entire sample is on the floor, circumvent the cone systematically depositing shovelfuls from the base to the apex of the cone so that the centre of the cone is not displaced. Repeat the process twice.

— Form the cone into a long pile as follows:

  — Take a shovelful from the base of the cone and spread the material into a ribbon having an initial width equal to that of a shovel and a length of 1,5 m to 3,0 m.

  — Take the next shovelful from a different point at the base of the cone and spread directly over the previous shovelful, but in the opposite direction.

  — Repeat the above step until one long pile is formed.

— Discard half the sample in the following manner:

  — Take a shovelful from the bottom of one end of the pile and set aside.

  — Take the next shovelful immediately adjacent to the first by advancing along the side of a pile a distance equal to the width of the shovel and discard.

  — Again, advancing in the same direction a distance of one shovel width, take the third shovelful and add to the first.

  — Continue along the pile following the above procedure, discarding alternate shovelfuls so that the pile is decreased gradually and uniformly.

— Repeat the above procedure (from forming the coning to halving the pile) until the retained amount of material is equal to the desired mass of the subsample (but no less than the minimum mass of the subsample in accordance with Table 1).

— Transfer the subsample to an appropriate sample container in accordance with Clause 10.

A.2 Coning and quartering

This procedure is suitable for all samples down to a mass of approximately 1 kg.

— Identify the maximum particle size of the sample and determine the minimum mass of the subsample(s) according to Table 1. When the minimum mass of the subsamples is larger than desired and the maximum particle size is related to the size of macro aggregates, the macro aggregate size can be reduced according to 8.4 or 10.2.4.2. The subsampling process shall be stopped when the mass of the subsample is equal to or larger than the minimum mass of the subsample as derived from Table 1.

— Identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow easy access around the whole sample when spread on the surface.

— Place a clean protective floor covering, preferable heavy-duty plastic sheeting, to protect the sample from contamination by the surface.

— Shovel the sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. For samples exceeding the mass of 500 kg, the use of a mechanical shovel (e.g. excavator) is recommended. Manual handling is preferred for samples smaller than 100 kg.

— When the entire sample is on the floor circumvent the cone systematically taking shovelfuls from the base and forming a second cone with all the material from the first cone transferred to the apex of the second cone. Repeat the process twice.

— Flatten the cone so that the height is less than or equal to the height of the shovel or spade used.

— Divide the pile into quarters along two lines intersecting at 90° to each other, using either Method 1 or Method 2, and then transfer the subsample to an appropriate sample container in accordance with Clause 9:

Method 1:

— Place the centre of a sheet metal cross, made with four blades joined together at the centre at 90° to each other, at the centre of the flattened cone and press the lower edges of the metal cross through the sample. The height and length of the blades forming the cross should be greater than that of the flattened cone.

— With the metal cross left in position discard opposite diagonal quarters and brush clean the space they occupied.

— Remove the metal cross and mix together the remaining two quarters.

— Cone and quarter again using the previous stages until the volume of remaining sample is equal to the desired mass of the subsample (but no less than the minimum mass of the subsample in accordance with Table 1).

— Transfer the subsample to an appropriate container in accordance with Clause 10.

Method 2:

— Quarter the flattened cone along two diagonals intersecting at right angles, using a shovel inserted vertically into the sample.

— Discard one pair of opposite quarters and shovel the remainder into a stockpile.

— Check if the mass of the discarded material is equal to half the mass of the (sub)sample before subdivision, allowing a variation of ± 10 % (mass fraction). When this condition is not met, the discarded material should be added and mixed again, where after the subdivision can continue.

— Repeat the process of mixing and quartering until the volume of remaining sample is equal to the desired mass of the subsample (but no less than the minimum mass of the subsample in accordance with Table 1).

— Transfer the subsample to an appropriate sample container in accordance with Clause 10.

NOTE Coning and quartering are known to be subject to bias. This bias is partly caused by the tendency of larger particles to roll down the side of the cone and to collect at the base. This results in segregation of particles from the top to the bottom of the cone. The same problem arises when taking subsamples when the areas to be subsampled are not previously separated (for instance by the metal cross as described in Method 1 of quartering).

A.3 Riffling

The use of a riffle box is possible when the sample is dry enough to allow free flow of the particles through the riffle box. Division of the sample with a riffle box is most often only practical for samples less than approximately 100 kg (but depending on the size of the riffle box).

Division of the sample with a riffle box will result in a reduction to one half or one quarter (depending on the riffle) at each operation.

— Identify the maximum particle size of the sample and determine the minimum mass of the subsample(s) according to Table 1. When the minimum mass of the subsamples is larger than desired and the maximum particle size is related to the size of macro aggregates, the macro aggregate size can be reduced according to 8.4.1. The subsampling process shall be stopped when the mass of the subsample is equal to or larger than the minimum mass of the subsample as derived from Table 1.

— Identify an area of hard surface sheltered from the effects of wind and rain, preferably flat and large enough to allow ease of access around the whole sample when spread on the surface.

— Place a clean protective floor covering, preferable heavy-duty plastic sheeting, to protect the sample from contamination by the surface.

— Shovel the sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. Manual handling is preferred for samples smaller than 100 kg.

— When the entire sample is on the floor circumvent the cone systematically taking shovelfuls from the base and forming a second cone with all the material from the first cone transferred to the apex of the second cone. Repeat the process twice.

— Check that the slot widths of the riffle box are at least three times larger than the maximum particle size of the sample to be subsampled.

— Using a shovel or container, pour the material into the riffle box. It is essential that the sample is poured evenly over the whole riffle in order to prohibit biased subsampling.

— Remove one subsample as the reduced sample, discarding the remaining material.

— Check if the mass of the discarded material is equal to half (or three quarters of) the mass of the (sub)sample before subdivision, allowing a variation of ± 10 % (mass fraction). When this condition is not met, the discarded material should be added and mixed again, where after the subdivision can continue.

— Repeat the process of riffling until the volume of remaining sample is equal to the desired mass of the subsample (but no less than the minimum mass of the subsample in accordance with Table 1).

— Transfer the subsample to an appropriate sample container in accordance with Clause 10.

A.4 Application of Tyler divider

The sloping plate of the Tyler divider provides a reduction ratio of 16:1. Material flows over the plate and is reduced successively in steps at each station down the plate by means of slots or holes placed in the plate. Each reduction is to one half the amount passing the station and a means for re-mixing after each stage is incorporated in the plate. An essential requirement in applying a Tyler divider is that the sample is dry enough to allow free flow of the particles.

The mechanical feed should be set at a constant rate suitable for the material being sampled and as identified in the sampling plan. This implies the requirement for the hopper width to be equal to that of the sloping plate and a gate of variable height.

— Identify the maximum particle size of the sample.

— Check that the slot width of the Tyler divider is at least three times larger than the maximum particle size.

— Determine the minimum mass of the subsample(s) according to Table 1 and calculate if the reduction ratio of the divider will result in a subsample that is equal to or larger than the minimum mass of the subsample. If not, this type of divider shall not be used.

— Start the division process by pouring the sample into the divider with a constant rate and catch the subsamples(s) in (an) appropriate sample container(s).

— When necessary repeat the process of subsampling be using one or more of the resulting subsamples until a subsample of the required mass is obtained (but is no less than the minimum mass of the subsample in accordance with Table 1).

— Transfer the subsample to an appropriate sample container in accordance with Clause 10.

A.5 Application of mechanised turntable (rotating divider)

The mechanised turntable comprises a number of prismatic containers, of equal size, mounted round the periphery of a circle which pass under the falling stream of the sample fed from a hopper mounted above the turntable, and off-set from the centre.

The turntable should operate at a constant speed of rotation that should not change (significantly) while sample material is coming into the turntable.

— Check that the slot width of the turntable is at least three times larger than the maximum particle size.

— Transfer the sample with a constant speed into the turntable. The speed should be relatively low in order to allow all particles to fall freely into the slot of the turntable and it will take a large number of rotations of the turntable before the full amount of sample is transferred into the slot.

— After completion of the division process, one or more of the subsamples is (are) collected.

— Check the mass of one of the subsamples. If the mass is not equal to the product of the total mass and the inverse number of subsamples in the rotating divider, allowing a variation of ± 10 % (m/m), all subsamples shall be added and the subsampling step shall be repeated.

— The subsamples obtained are (if necessary) divided again, until a subsample of the required mass is obtained, or until the minimum sample mass is achieved, see Table 1.

— Transfer the subsample to an appropriate sample container in accordance with Clause 10.

Annex B
(informative)

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

This Annex B gives an overview of the volatile organic compounds with associated vapour pressure, boiling and melting points.

The compounds are arranged in Tables B.1 and B.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 B.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 B.2 as interpolation is not possible in these cases.

Table B.1 and Table B.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. Hexadecane is one of the last compounds to be clearly indicated in gas chromatographic analysis of volatile hydrocarbons.

This results in a categorization of components. Therefore, this European Standard distinguishes the following two categories:

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

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

Table B.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
1,4-dimethylbenzene (p-xylene)	1,08	138	13
1,3-dimethylbenzene (m-xylene)	1,05	139	–48
1,2-dimethylbenzene (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 B.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

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