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Sample pre-treatment for soil, sludge and bio-waste

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Foreword

This document is a working document.

This document *TF WI* ### has been prepared by CEN/BT/Task Force 151 – Horizontal standards in the field of soil, sludge and bio-waste, the secretariat of which is held by Danish Standards.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For relationship with EU Directive(s), see informative Annex A, B, C or D, which is an integral part of this document.

This standard is applicable and validated for several types of matrices. The table below indicates which ones.

Material	Validated for (type of sample, e.g. municipal sludge, compost)	Document
soil	validated	ISO/FDIS 11464, ISO/FDIS 10381-8
sludge	validated	ISO 5667-13, ISO 5667-15, CEN/TC 308/WG 1/TG 4 N0058
bio-waste, soil improvers and growing media	Not yet validated	EN 12579, EN 13040
Sediment	validated	ISO 5667-13, ISO 5667-15, ISO/FDIS 11464
suspended solids	Not yet validated	
(soil-related) waste	validated	EN 13657

Contents

Introduction

This document is developed in the project 'Horizontal'. It is the result of a desk study "DS 33-1: Sample pre-treatment for soil, sludge and bio-waste" and aims at evaluation of the latest developments in sample pre-treatment of sludge, soil, treated bio-waste and neighbouring fields for subsequent physico-chemical and chemical analysis. After discussion with all parties concerned in CEN and selection of a number of pre-treatment methods described in this study the standard has been developed further as a modular horizontal method and has been validated within in the project 'Horizontal'.

A horizontal modular approach is being investigated and developed in the project 'Horizontal'. 'Horizontal' means that the methods can be used for a wide range of materials and products with certain properties. 'Modular' means that the pre-treatments developed in this approach concerns a specific step in a test procedure and not the whole test procedure (from sampling to analyses).

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the test procedure.

The other horizontal modules that will be available in due time are to be found in the informative annex [xxx] which contains a brief overview of the modules that will be worked out in the project 'Horizontal.'

The texts of the chapters 1 to 11 are normative; annexes are normative or informative, as stated in the top lines of the annexes.

1 Scope

This European standard specifies the pre-treatments required for soils (including sediments), sludges, bio-wastes and (soil-related) wastes that are to be subjected to chemical and physico-chemical analysis of stable and non-volatile parameters. The pre-treatment of samples aims at preparing a (small) test sample which is representative for the original sample.

Pre-treatment in this European standard is defined as operations and treatment steps done on a fresh sample until the production of a homogeneous test portion used for the subsequent analytical measurements. This excludes explicitly the issue of extraction and digestion of analytes.

This European standard describes the pre-treatments which are required after sampling (e. g. storage and preservation), pre-treatments which could be performed under field conditions if necessary and the sample pre-treatment which is only allowed under laboratory conditions.

The pre-treatment of samples for the purposes of determining organic compounds is the subject of a further desk study (see Bibliography). Nevertheless the pre-treatment of soil samples for the purposes of determining organic as well as inorganic compounds is considered if necessary.

The pre-treatment procedures described in this European standard are not applicable if they affect the results of the determinations to be made.

2 Normative references

ISO 565	Test sieves – Metal wire cloth, perforated metal plate and electroformed sheet – Nominal sizes of openings.
ISO/FDIS 10381-8	Soil quality – Sampling — Part 8: Guidance on sampling of stockpiles
ISO 11465	Soil quality – Determination of dry matter and water content on a mass basis – Gravimetric method
EN 12880	Characterization of sludges – Determination of dry residue and water content
ISO 16720	Soil quality – Pretreatment of samples by freeze-drying for subsequent analysis

3 Terms and Definitions

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

3.1
field sample
the quantity (mass or volume) of material obtained through sampling without any sub-sampling [EN 14899]

3.2
composite sample
two or more increments/sub-samples mixed together in appropriate proportions, either discretely or continuously (blended composite sample), from which the average value of a desired characteristic may be obtained [ISO 11074-2]

3.3
increment
sampling unit collected by a single operation of a sampling device and being used in a composite sample [ISO 11074-2]

NOTE 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 per definition a sample.

3.4
laboratory sample
sample or sub-sample(s) sent to or received by the laboratory (IUPAC definition)

NOTE 1 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 2 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.

3.5
maximum particle size (D_{95})
particle size that concurs with the mesh width of a sieve on which a maximum of 5 % (m/m) of the material remains

3.6
particle size reduction
procedure to reduce the particle size of the whole (sub-)sample through grinding or crushing without reducing the sample size (mass)

3.7
sample
portion of material selected from a larger quantity of material [ISO 11074-2]

NOTE 1 The manner of selection of the sample should be described in the sampling plan.

NOTE 2 The material can be sludge, soil, bio-waste or related material

3.8
sample division
procedure through which sub-samples of smaller size than the original sample are obtained without reducing the particle size of the individual particles

3.9
sampling plan
all information pertinent to a particular sampling activity

NOTE The sampling plan provides the sampler with a predetermined procedure for the selection, withdrawal, on-site pre-treatment, preservation and transportation of the portions to be removed from a stockpile (population) as a sample.

3.10
sample pre-treatment
collective noun for all procedures used for conditioning a sample to a defined state which allows subsequent examination or analysis or long-term storage

NOTE Depending on the requirements of the analytical method sample pre-treatment includes e.g. mixing, splitting, drying, crushing, and stabilisation.

3.11
sludge
mixture of liquid and solids separated from various types of liquids as a result of natural or artificial processes

3.12
sub-sample
sample obtained by procedures in which the items of interest are randomly distributed in parts of equal or unequal size [ISO 11074-2: 1998]

NOTE 1 A sub-sample may be:

a) 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 The term 'sub-sample' 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.

NOTE 3 Tools and other devices to obtain samples are sometimes also designated as 'samplers'. In this case use 'sampling devices' or 'sampling equipment'.

3.13
test portion; analytical portion
amount or volume of the test sample taken for analysis, usually of known weight or volume (IUPAC definition)

NOTE The test portion can be taken 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. A test portion is removed from the test sample for the performance of the test or for analysis.

3.14
test sample; analytical sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis (IUPAC definition)

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, and appropriate safety precautions should be taken.

In general, when sludge is sampled the safety advice given in parts of ISO 5667 is relevant on many occasions, for example see ISO 5667-3 and -10.

5 Principle

5.1 General

Pre-treatment in this European standard is the process of sub-sampling and considers the treatment of a sample from soon after sampling until the production of a homogeneous test sample under laboratory conditions. The operations and treatment steps are divided into pre-treatment procedures suitable in the field and pre-treatment procedures that are restricted to be performed in the laboratory.

NOTE For sampling refer to the parts of the desk study on sampling (see Bibliography).

Beside the requirements given in this European standard the analytical standards shall be regarded for particular regulations (e. g. different mesh sizes for sample sieving in the case of a compliance test for leaching of granular waste materials and sludges according EN 12457-1 to -4)

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

The type of sample pre-treatment 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 – has to be avoided since that process requires good defined conditions which can not be achieved in the field. Particle size reduction is restricted to being a laboratory operation.

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

NOTE 2 It should be noted that every type of pre-treatment will have an influence on several material properties.

One should realise that the quality of sample division (sub-sampling) 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 as a preferable option. The measures for sample division described in clause 7.5 are suitable for pre-treatment in the field as well as in the laboratory.

If several parameters have to be investigated, the sample pre-treatment shall be designed in such manner that the parameters of major importance are determining the pre-treatment. If this is not possible, e. g. the required precision for each parameter can not be achieved, separate pre-treatment shall be set up for each group of parameters.

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

The pre-treatments under laboratory conditions as described in clause 8 are appropriated for soil, sludge, (soil related) waste and for sediments. Soil improvers should be treated in accordance with EN 12579 and EN 13040.

NOTE 1 When preparing composite samples regard should be paid to analytical requirements. For example, composite samples are not appropriate if volatile compounds are to be determined.

NOTE 2 For the pre-treatment of soil, sludge and bio-waste samples for the purposes of determining organic compounds, volatile as well as non-volatile, refer to desk study on “Pre-treatment for organic parameters”.

NOTE 3 The determination of some parameters requires sample pre-treatment soon after sampling as specified in the respective methods.

Any of the procedures described in clause 7 and 8 and modifications should be mentioned in the pre-treatment report if applied during pre-treatment.

For main procedures see the flow diagram in figure 1 and figure 2.

5.2 Pre-treatment in the field

In the field sample pre-treatment is restricted to the process of sub-sampling by sample division. It will be necessary

– if field samples are too large to take to the laboratory

or

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

NOTE When possible, the sample pre-treatment will take place in the laboratory, as sample integrity can be best guaranteed under laboratory conditions. Sub-sampling by methods given in clause 7.5 can be performed in the field as well in the laboratory.

When sub-sampling is necessary the relation between the minimum size of the sub-samples and the maximum size of the particles (D_{95}) in the original field sample has to be taken into account (see 7.4).

Samples are divided into sub-samples either mechanically or manually. Sub-sampling methods are described in clause 7.5.

In some cases the soil is strongly aggregated. Such macro aggregates can be reduced by hand (see 7.4.1) otherwise they should be seen as individual "particles".

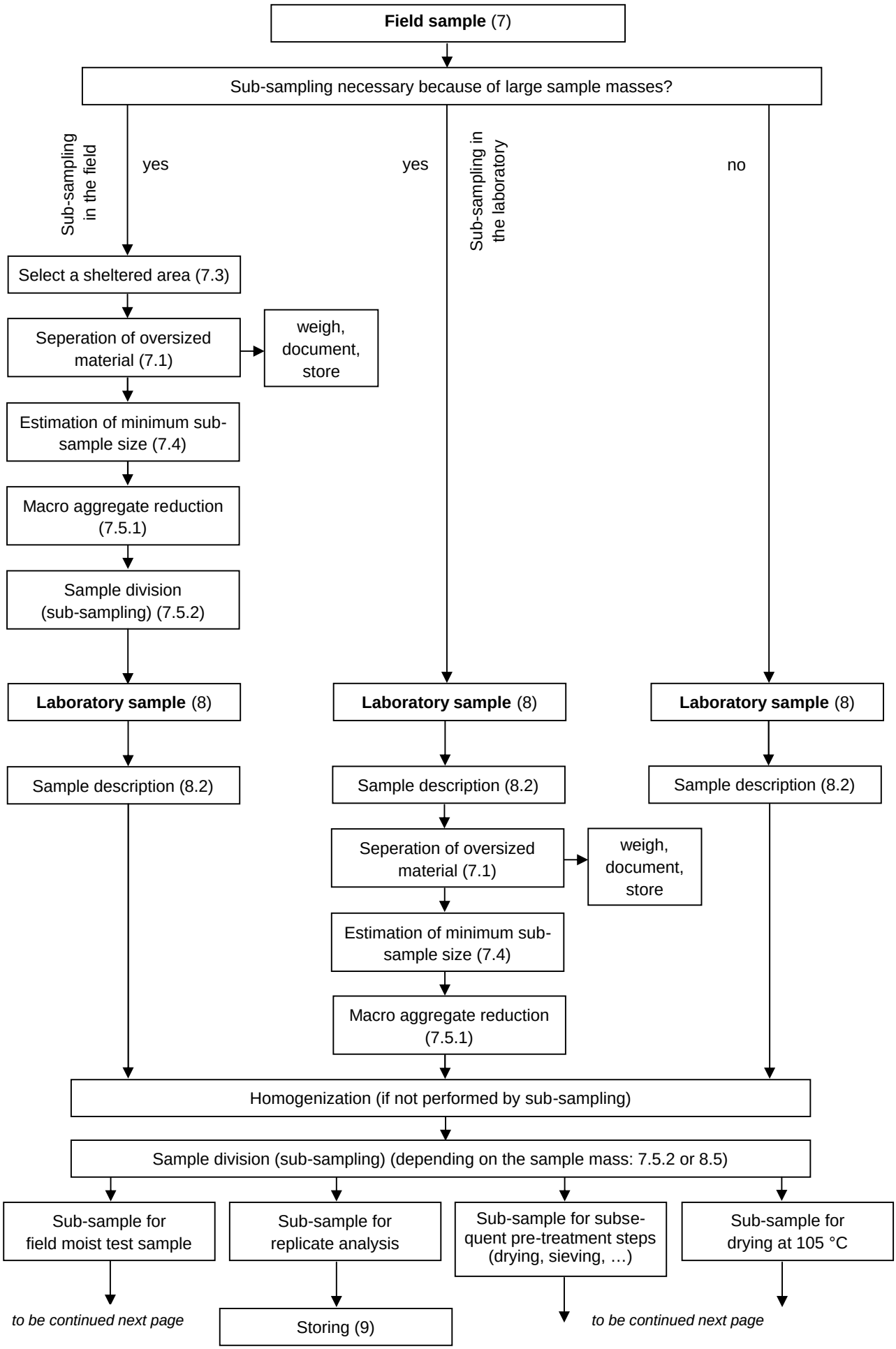

Figure 1 – Diagram for pre-treatment of soil, sludge (solid), sediment and (soil-related) waste samples

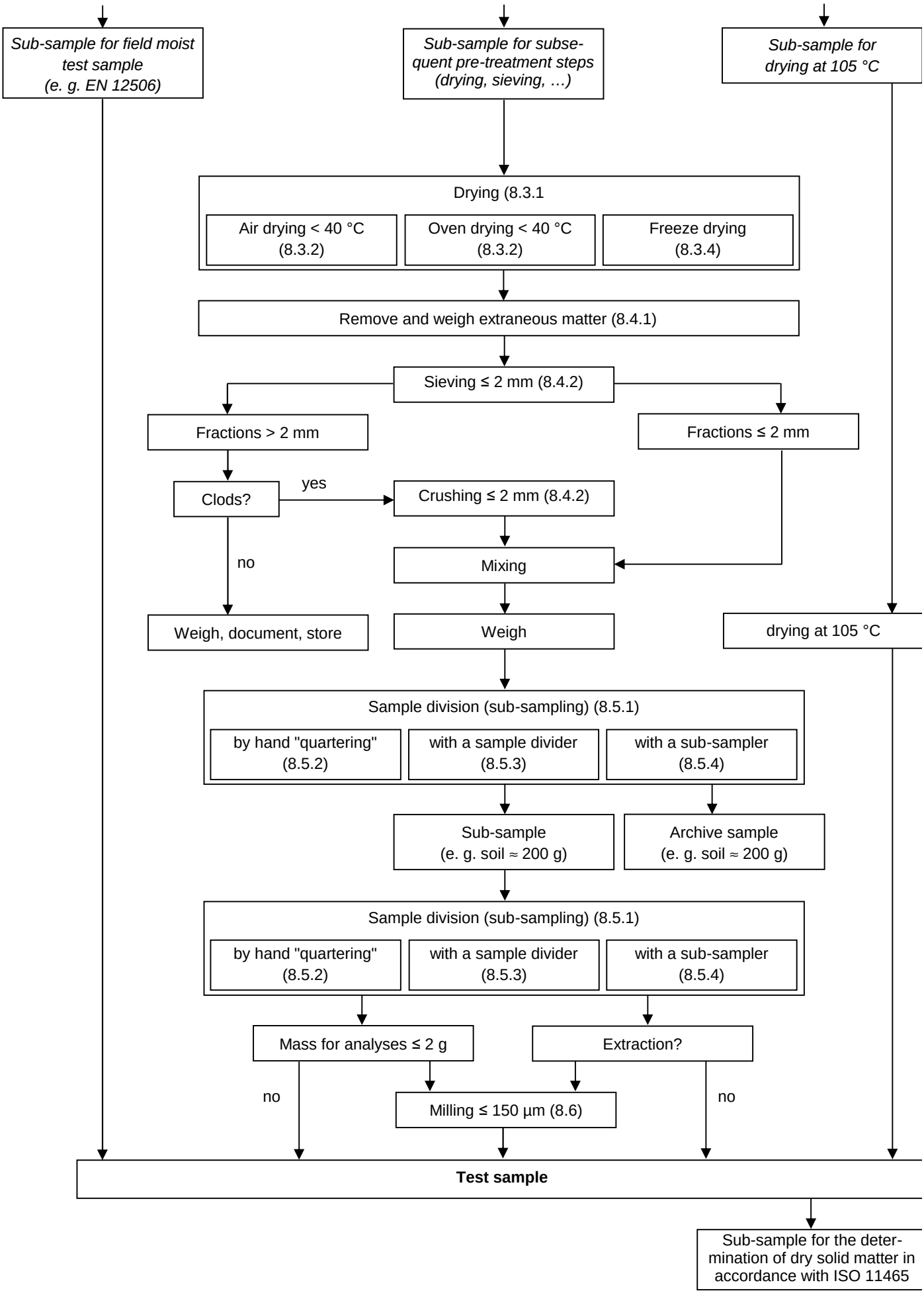

Figure 1 – Diagram for pre-treatment of soil, sludge (solid), sediment and (soil-related) waste samples(continued)

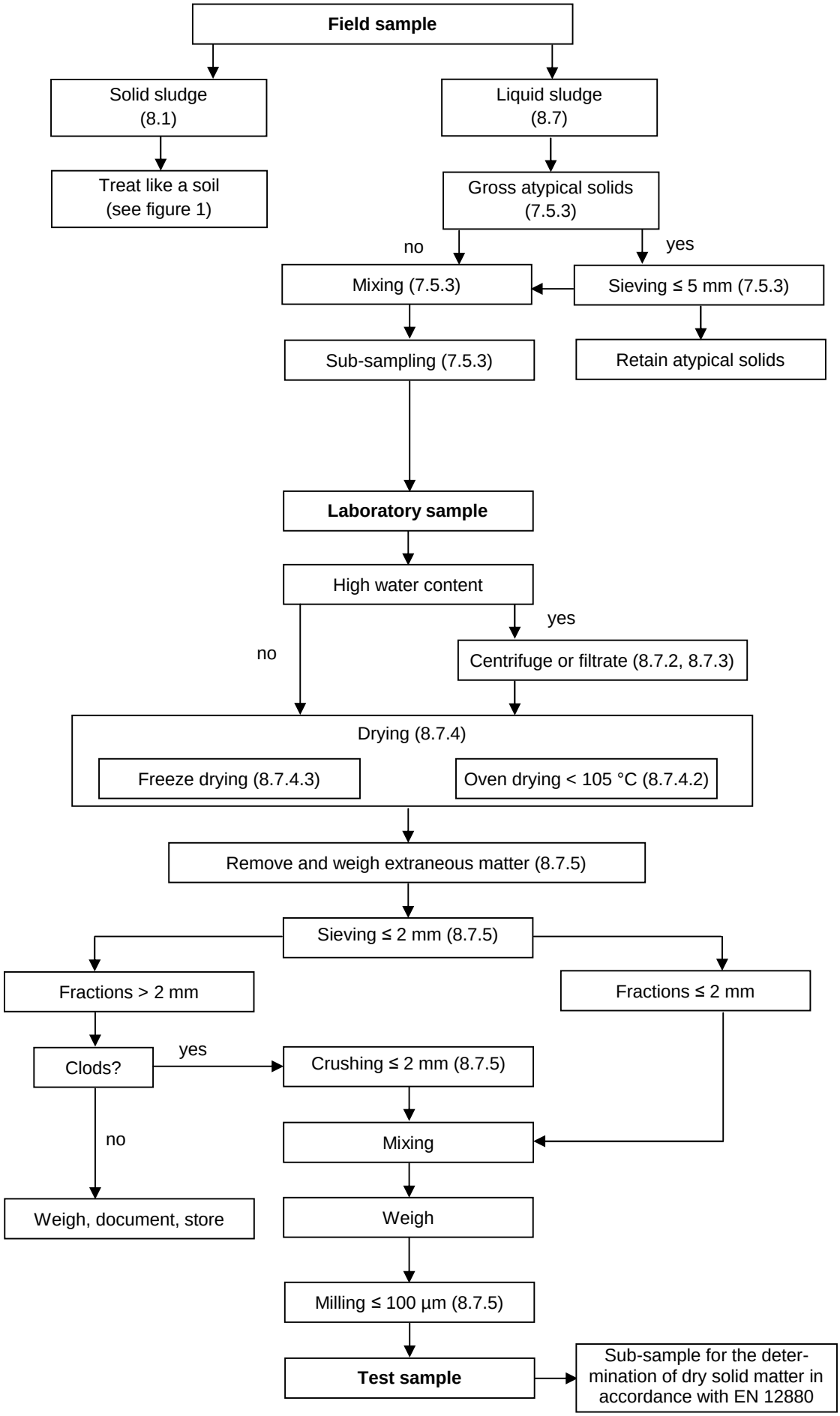

Figure 2 – Diagram for pre-treatment of liquid sludge

In the field sample division shall be carried out only if a sheltered area is available (see 7.3).

Soon after sampling until pre-treatment in the laboratory samples should be stored so that the characteristics of the sample are protected (e. g. cool and light protected, see 9).

5.3 Pre-treatment in the laboratory

Large sample masses which are not sub-sampled in the field are divided prior further treatment (see 8.1).

The laboratory samples are dried in air, or in an oven at temperature not exceeding 40 °C, or freeze-dried (see 8.3). If necessary, the soil sample is crushed while still damp and friable and again after drying (see 8.4). The sample is sieved (see 8.4) and the fraction smaller than 2 mm is divided into portions mechanically or by hand, to enable representative sub-sampling for analysis (see 8.5). The size of the particles of the fraction smaller than 2 mm is further decreased (see 8.6), e. g. if small sub-samples (< 2 g) are required for analysis or trace elements soluble in *aqua regia* should be extracted. For liquid sludge see clause 8.7.

When replicate analyses are required, it shall be clarified in the overall investigation plan at which stage of sub-sampling replicates must 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 changes due to microbial activity.

NOTE 2 The sieve aperture size of 2 mm is generally used. However, before the pre-treatment is started, it should be checked if any of the analytical methods to be applied later require other sieve sizes.

NOTE 3 Storing soil 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 soil parameters, especially solubility of both inorganic and organic fractions.

NOTE 4 Keeping an archive sample (see figure 1) is optional and should be clearly stated in the overall description of the investigation programme.

6 Apparatus

6.1 General

It is essential that the apparatus and tools used for the pre-treatment does not add or remove any of the substances under investigation (e. g. heavy metals). If the use of certain equipment and/or materials is not permitted in pre-treatment of samples required for particular physico-chemical analysis, this shall be mentioned in the relevant horizontal modules on analysis.

NOTE The apparatus to be used are not specified in detail. However, drawings of some items of equipment suitable for soil pre-treatment in the laboratory are provided in Annex C, figures C.1 to C.4. Most comparable national and European standards contain detailed equipment specifications and these may be used, provided they meet the basic performance requirements indicated in this horizontal module.

6.2 Analytical balance, readable and accurate to 1 g.

6.3 Analytical balances, readable and accurate to 0,1 g.

6.4 Balance, readable and accurate to 0,0001 g.

6.5 Centrifuge, optional.

6.6 Crusher(s), e. g. jaw crusher or cutting device.

NOTE Due to crushing, contamination of the sample can occur to an extent which affects the leaching of some constituents of concern e.g. cobalt and tungsten from tungsten carbide equipment or chromium, nickel and molybdenum from stainless steel equipment.

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

6.8 **Drying oven**, thermostatically controlled and capable of maintaining a temperature not exceeding 105 °C, optional.

6.9 **Freeze-drier**, optional.

6.10 **Grinding mill**, capable of grinding dried soils, sludges and sediments, to a size less than 150 µm without contamination by the elements to be determined in accordance with ISO 11466.

NOTE Both agate and zirconium oxide mills have been found suitable for the extraction according ISO 11466

6.11 **Large heavy-duty plastic sheeting**, optional.

6.12 **Mechanical mixer(s)**, optional.

6.13 **Mechanical shovel**, optional.

6.14 **Mechanical sieve shaker**, optional.

6.15 **Mechanised turntable/Rotating dividers**, optional.

6.16 **Mesh sieves**, complying with ISO 565, with apertures of 150 µm, 100 µm or of the size specified in the relevant test method.

6.17 **Pestle and mortar**, made of porcelain or sintered corundum.

6.18 **Porcelain dish**, diameter 30 cm, or bigger.

6.19 **Riffle box**, optional.

6.20 **Sample splitter** or utensils for cone and quartering for sub-sampling of test samples (optional).

6.21 **Screens**, having a mesh size of 2 mm for air-dried samples and of 5 mm to 8 mm for freshly collected samples and which do not cause contamination by elements to be determined.

NOTE Due to sieving, contamination of the sample can occur to an extent which affects the leaching of some constituents of concern e. g. cobalt and tungsten from tungsten carbide equipment or chromium, nickel and molybdenum from stainless steel equipment.

6.22 **Sledge hammer**, optional.

6.23 **Spade**, optional.

6.24 **Spoon, metal and porcelain**, optional.

6.25 **Tyler divider**, optional.

6.26 **Wooden or other soft-faced hammer**, optional.

7 Pre-treatment procedures in the field (from field sample to laboratory sample)

7.1 General

Sample pre-treatment (in the field or laboratory) is the process of sub-sampling, necessary to obtain a representative sub-sample for further measures which have to be carried out under laboratory conditions. A selection of pre-treatment techniques suitable for sample division in the field as well as in the laboratory is given in clause 7.5.

Care has to be taken to avoid contamination of the sample via the air or by dust (e. g. between samples stored or processed close to one another).

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

If the sampling involves the separation of oversized material (i. e. mineral grains, sand, pebble and all other materials) due to very coarse grained or heterogeneous conditions, the material removed shall be weighed or estimated and recorded and described to enable the analytical results to be given with reference to the composition of the original sample.

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 sub-sampling. Therefore the requirements for e. g. pre-treatment, preservation and transportation have to 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 1 In most guidelines on sampling for agricultural or similar investigations it is recommended that composite samples are collected by taking a number of increments (according to ISO 10381-4 at least 25 increments should be obtained) and combining them to form a composite sample.

NOTE 2 When preparing composite samples regard should be paid to analytical requirements. For example, composite samples should never be used if volatile compounds are to be determined.

7.2 Sample quantity

This European standard does not specify the sample quantities used for determination of physical and inorganic parameters – details are given in the respective methods. Nevertheless the relationship between particle size and minimum sampling size has to be regarded (see 7.4). For estimation purposes of quantities required some examples are given.

At least 500 g to 1 000 g of fresh soil as sampled should be obtained for physico-chemical and chemical analysis. This figure applies both to single samples and composite samples, in the latter case after sufficient homogenization.

Soil samples obtained to serve as reference material or to be stored in a soil specimen bank should be of larger size, usually larger than 2 000 g.

The determination of the particle size distribution may need a very large mass of soil material. The actual mass required will depend on the largest grain size to be determined (see ISO 11277).

Little guidance can be given as to the size of sludge samples. This is because this criterion is dependent on the variability of the sampled material and the type of analysis to be carried out. For instance for the characterisation of waste and sludges by leaching according EN 12457-4 obtain a laboratory sample of at least 2 000 g of the material. The analyst should always be consulted as to the quantities of sludge required.

NOTE Without reference to the material the size of the laboratory sample depends on the parameters to be investigated, the particle size or the size of components, the number of parallel analysis as well as on the size of the archive sample, if required.

7.3 Workplace

The division of the field sample into a number of representative sub-samples shall be carried out only when the integrity of the sample and sub-samples 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 Nevertheless, also sample division can 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.

7.4 Estimation of minimum sub-sample size

The minimum size of the sub-sample is determined by the maximum size of the particles that are present in the sample. When the sample contains macro aggregates, the maximum size of the macro aggregates determines the minimum size of the sub-samples whenever the macro aggregates behave like individual particles during sample pre-treatment (that is when macro aggregates will not be cut in pieces by the (sub-) sampling equipment used). See also 7.5.1 for macro aggregate size reduction.

The relation between the minimum size of the sub-sample and the maximum size of the particles (D_{95}) in the original sample is given in table 1. The relation is based on the formula for the minimum sample size as given in the ISO 10831-8.

NOTE 1 Some soils (partly) contain (very) large boulders. When these boulders should be considered as part of the sample, this would result in extremely large samples, both in field as well as for the material to be transferred to the laboratory. However, often only the smaller soil fraction is of interest and therefore these boulders can be neglected both during sampling and sample pre-treatment. Whenever such a situation is encountered, the sampling plan should clearly define the material that is to be sampled/sub-sampled.

NOTE 2 For small particle sizes the minimum size of the sub-sample can be very small which is relevant for sub-sampling in the laboratory in order to obtain the analytical sample. For sub-sampling in the field a minimum amount of approximately 200 grams should at least be transferred as sample to the laboratory. Further sub-sampling will then take place in the laboratory.

Table 1 – Minimum size of sub-samples as a function of the maximum size of macro aggregates or particles present in the sample (from: ISO/FDIS 10381-8 Soil quality – sampling)

Maximum size of macro aggregates or particles in the sample [mm]	Minimum size of sub-sample(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

It shall be noted that the minimum size of the sub-sample(s) as given in table 1 does not necessarily mean that this is the actual size to be used. Larger sizes of sub-samples might be needed for analysis, and therefore the size of the sub-sample(s) shall be checked with the laboratory.

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

For practical reasons, the maximum size of the samples to be sent to the laboratory should be not larger than approximately 20 kg to 30 kg. When larger sub-samples 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 sub-sample of an acceptable size to the laboratory. When grinding or crushing for particle size reduction 'in the field' is truly necessary, it is only allowed under laboratory conditions, see 5.2. For these situations either a mobile laboratory or on site laboratory is needed.

7.5 Pre-treatment methods

7.5.1 Procedure for macro aggregate reduction by hand

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

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 size of the sub-sample as a starting point as given in table 1. When the desired size of the sub-sample is smaller than a given minimum size of the sub-sample, 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 ease of access around the whole sample when spread evenly on the surface.

- Place a clean protective floor covering, preferable 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 sledge hammer gently reduce the size of the macro aggregates until all oversized material is less than or equal to the required particle size.

7.5.2 Sub-sampling methods

A sample can be divided into sub-samples either mechanically or manually. Potentially it is preferable to use a mechanical system for sub-sampling, since this results in more representative sub-samples. Especially for soil this is however only true 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 sub-sampling in the field directly after sampling (see 5.2).

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. And even when the mechanical division is still possible, mechanical sub-sampling devices will probably function incorrectly, and therefore will result in biased sub-samples. As a consequence, the manual sub-sampling methods are often to be preferred for sub-sampling in the field.

In Annex A the following sub-sampling 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 The sub-sampling methods in Annex A are also suitable for sample division in the laboratory.

To obtain a representative sample of sludge cake, the mass accumulated will always be too large for laboratory manipulation at the bench. Sample size reduction is, therefore, best carried out in the field by coning and quartering as described in Annex A.2 for soil sub-sampling.

Sludges that have a gelatinous appearance and behave more like a jelly than a mineral solid like gravel, are unlikely to be suitably homogenized by coning and quartering. Mixing, such as that employed for the hand or mechanical preparation of cement mortar, may be more appropriate. Division into sub-samples can still be achieved by the combination of diametrically opposed quarters.

7.5.3 Sludge handling

For some types of liquid sludge, particularly raw sewage sludge, gross atypical solids, 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.

Atypical solids 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.

Large volumes of liquid sludges accrued by the combination of representative samples will need to be homogenized before sub-sampling. The mixing process should preferably be tested to ensure efficiency of mixing. In the event of there being any risk of demixing, the sub-sample shall be taken during the mixing process. The homogenization can be achieved in a container such as a plastics dustbin using a suitable paddle to prevent settlement.

8 Pre-treatment procedures in the laboratory (from laboratory sample to test sample)

8.1 General

In case of large sample masses (e. g. of soil or waste) sub-sampling methods prior further treatment according to clause 7.5 should be achieved in the laboratory to reduce the initial sample size. The relation between the minimum size of the sub-sample and the maximum size of the particles that are present in the sample have to be taken into account (see 7.4).

Archive and replicate samples should be taken at this stage.

The procedures for drying, fraction separation and size reduction are set out in 8.3 and 8.5. At several stages in the procedure, the analyst 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 rehomogenized after any separation, sieving, crushing or milling operation (that may have resulted in segregation of different sized particles) has been carried out.

The field moist laboratory sample is used if e. g. cyanides are determined. Some soils have to be extracted and homogenized in the freshly collected state as described in Annex B. For example soil samples from organic horizons (having more than 30 % by mass of organic matter – such as bog soil or topsoil) and samples from reductomorphic horizons (horizons affected by backwater or ground water – such as coastal marsh soil or gley soil).

NOTE 1 Care should be taken to avoid contamination of the sample via the air or by dust (e. g. from the ambient laboratory atmosphere or between samples stored or processed close to one another).

NOTE 2 It is recommended that pre-treatments always be performed in a room used only for this purpose and remote from locations where analytical measurements are made.

NOTE 3 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, or the tests to be carried out, will in some cases affect the possibilities or methods of sub-sampling. Therefore the requirements for the pre-treatment (e. g. drying and reduction of particle size) have to be in accordance with the analytical method(s).

As long as a sludge sample is not solid it is treated like a liquid sludge sample (see 8.7) otherwise like a soil sample (see 8.3 to 8.6) because in the analysis of sludges, various and high water contents of the sludge may cause difficulties.

NOTE Depending on the parameter liquid sludge samples are analysed with or without drying. Drying is not required if $N\text{-}NH_3$, $o\text{-}PO_4$, SO_4, Cl^-, NO_3 or NO_2 are determined.

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

8.2 Laboratory sample description

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

8.3 Drying of soil and waste

8.3.1 General

The laboratory samples are dried in air, or in an oven at 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 set out in 8.3.2, 8.3.3 or 8.3.4. Dry until the loss in mass of the soil sample is not greater than 5 % (mass fraction) per 24 h. After the drying process has been completed, determine and record the total mass of the dried sample.

To accelerate the drying process, break down the size of larger aggregates (larger than 15 mm) during the process. When samples are dried in air, crush them lightly by hand using a wooden hammer or a mortar and pestle, taking great care to avoid contamination. When samples are dried in an oven, remove them temporarily from the oven and treat them in the same way. This procedure also makes it easier to separate the particles larger than 2 mm.

Freeze-drying has the advantage that the sample to be dried rarely dries into clods; it usually breaks up into parts.

For the determination of the pH of soil samples according ISO 10390, for the extraction of trace elements soluble in *aqua regia* according ISO 11466 and the determination of trace elements according ISO 14870 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.

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.

NOTE 3 Ammonia nitrogen is lost during freeze-drying of sludges. Therefore determination of ammonia nitrogen content should be avoided after freeze-drying, at least for liquid sludges.

8.3.2 Air drying

Spread all the material, in a layer not thicker than 5 cm, on a tray which does not absorb any moisture from the soil and which 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.

8.3.3 Oven drying

Spread all the material, in a layer not thicker than 5 cm, on a tray made of material which does not absorb any moisture from the soil and which does not cause contamination. Put the tray in the drying oven (6.7) and dry at a temperature that is not higher than 40 °C.

8.3.4 Freeze-drying

Freeze-drying shall be performed according to ISO 16720.

8.4 Crushing and removal of coarse materials

8.4.1 Separation of stones, etc.

Before crushing the sample, which will be necessary if samples, especially soil, have dried into large aggregates, extraneous matters should be removed from the dried sample. This are particles that cannot be ground, such as metal, screws etc. and in the case of soils fragments of glass, rubbish and roots. Oversized material like stones is removed, too. This process may be facilitated by the use of a 2 mm sieve (6.21) and by hand picking (see NOTE 2). Care should be taken to minimise the amount of fine material adhering to the extraneous matter removed.

The mass and the nature of the extraneous matters and oversized material shall be weighed and documented and the removed material – especially the oversized material – has to be kept for any further research that may be performed.

NOTE 1 It depends on the matrix what is understood by “extraneous matters”. Which matters should be removed is described in the sampling plan.

NOTE 2 If the material under examination is a contaminated soil or waste, the analyst may wish to crush the complete sample, including for example, pieces of slag, to pass the 2 mm sieve.

8.4.2 Crushing

If a 2 mm sieve has been used to facilitate removal of extraneous and oversized (> 2 mm) matter, any large dried particles remaining on the 2 mm sieve should be crushed (using suitable apparatus, see 6.6) to less than 2 mm. The apparatus used should be adjusted so that any crushing of the large particles is minimised to enable crushed particles to pass through the 2 mm sieve.

If a 2 mm sieve has not been used to facilitate removal of extraneous matter, then the dried sample should be sieved through a 2 mm sieve. Any large dried particles remaining on the 2 mm sieve should be crushed (using suitable apparatus) to less than 2 mm. The apparatus used should be adjusted so that any crushing of the large particles is minimised to enable crushed particles to pass through the 2 mm sieve.

The sample material ≤ 2 mm as well as the fraction > 2 mm shall be weighed and documented. The dry matter and water content are determined in accordance with ISO 11465. The sample passing through the 2 mm sieve has to be well mixed and pre-treated in order to produce the test-sample.

Over-sized and extraneous matter is kept if separate analysis of this material is required.

NOTE 1 If the fraction of aggregates greater than 2 mm is low it may be more efficient to sieve out particles smaller than 2 mm prior to crushing.

NOTE 2 In special cases the entire sample may be crushed.

NOTE 3 Fractions may be recombined after crushing using a mechanical mixer (6.12).

8.5 Sub-sampling

8.5.1 General

Sub-sampling is necessary when the sample cannot be stored (laboratory sample and archive sample) or used (test sample) completely, because of its size. Divide the dried, crushed and sieved laboratory sample (now < 2 mm) into representative portions of 200 g to 300 g according to 8.5.2 or 8.5.3. For the prepa-

ration of a test sample, split up the laboratory sample into representative portions until the required sizes of samples are obtained. Avoid the production of dust as much as possible.

NOTE 1 It could be of advantage to divide large laboratory samples according 7.5 prior further sub-sampling.

NOTE 2 It may be necessary to mill the material (8.6) between sub-sampling stages, to ensure homogeneity as the mass of the sub-sample is decreased. The procedures described in 8.5.2 and 8.5.3 may be used to produce sub-samples/test portions of the materials less than 2 mm and not less than 2 g in mass.

NOTE 3 Milling of the dried sample to a particle size < 2 mm prior to further treatments is also required if the total element content according ISO/FDIS 14869-1, -2 has to be investigated and prior to *aqua regia* digestion (e. g. ISO 11466, ISO 16772).

Select the method of sub-sampling (8.5.2, 8.5.3 or 8.5.4) according to the nature of the sample, the requirements of the subsequent determinations and the equipment available.

8.5.2 Sub-sampling by hand (quartering)

Mix the soil sample thoroughly using a suitable mechanical mixer (6.12) and spread it into a thin layer on a tray of a type which will not influence the composition of the sample. Separate the soil into four equal portions (quadrants). Combine two of the four portions diagonally, rejecting the other two. Repeat this procedure until the desired amount of soil is obtained.

8.5.3 Use of the sample divider

A suitable example of a sample divider of the multiple-slot type (riffle box) is shown in Annex C, figure C.2. This splits the sample into two equal parts.

NOTE The dimensions of the equipment should be chosen in such a way as to suit the amount and particle size of the materials to be divided (see Annex C, figure C.2 and table C.1).

8.5.4 Mechanical sub-sampling

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

An example of mechanical sub-sampling equipment is illustrated in Annex C, figure C.3. This operates according to the following procedure.

Pour the soil sample into the funnel of the sub-sampler (Annex C, figure C.3) and screw the sample bottles into place. Start the sub-sampler. After sub-sampling, 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 rehomogenized between each stage of sub-sampling. The contents of more than one container may be thoroughly mixed and used for subsequent phases of the sub-sampling routine.

8.6 Milling

If a test sample of less than 2 g is to be taken for the analysis, it is preferable that the whole sub-sample (see 8.5) be milled to a particle size < 150 µm prior to further sub-sampling for analysis.

Independent of the sample mass milling of the sample to a particle size < 150 µm is also required if the total element content according ISO 14869-1 and ISO 14869-2 has to be investigated and prior to *aqua regia* digestion (e. g. ISO 11466, ISO 16772).

NOTE Such grinding is designed to
- give a more homogenous sample from which a sub-sample (test-samle) is taken;
- increase the efficiency of acid attack by increasing the surface area of the particles.

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 shall be stated in the test report.

Mill a representative sub-sample (see 8.5) of the dried, crushed and sieved soil. Milling shall be continued until the complete sub-sample just passes through a sieve of 150 µm or a size otherwise specified in the test method (see 6.10).

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

NOTE 1 For the determination of some parameters based on chemical extractions, milling is not permitted because it increases the surface area of the sample and thus the reactivity of the sample.

NOTE 2 If required, the fraction > 2 mm can be milled and mixed with the fraction ≤ 2 mm before chemical analysis is performed.

8.7 Liquid sludge samples

8.7.1 General

For sub-sampling of liquid sludge see clause 7.5.3.

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 8.7.6, 8.7.7). After drying the sludge sample can be sieved (see 8.7.8).

NOTE The original material is used if $N\text{-}NH_3$, $o\text{-}PO_4$, SO_4, Cl^-, NO_3 or NO_2 are determined.

Depending on the kind of subsequent pre-treatment, use spoons (6.26) for homogenisation. In the case that heavy metals shall be analysed use a porcelain spoon (6.26).

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

8.7.2 Centrifugation

Sludge can be centrifuged (6.5) according to the procedures recommended by the manufacturers of the centrifuge to achieve a lower water content. After centrifugation the remaining water can be removed. It has to be stored if a balance of the total contaminant content inclusive water soluble contaminants is required.

NOTE Depending on the sludge material, especially sewage sludge with a high water content, centrifugation doesn't make sense, because even after 12 hours centrifugation particles can still swim on the surface, or are still in sedimentation.

8.7.3 Filtration

To obtain a sample with a lower water content the samples can also be filtrated 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. In the case that organic parameters shall be analysed use glass filters.

NOTE Organic solvents may be components of the glass fibre filters, which may solve organic parameters from the particles. Therefore use glass filters if organic compounds shall be analysed.

8.7.4 Drying

8.7.4.1 General

Dry the sample in a drying oven or in a freeze drier. Depending on the chosen method of drying, follow the procedures set out in 8.7.6 or 8.7.7.

8.7.4.2 Drying oven

Spread the sludge sample in a porcelain dish (6.18). Put the porcelain dish in the drying oven (4.1) and dry at a temperature < 105°C until the mass constance is reached.

NOTE Losses of some parameter can't be avoided (example: PCB, Dioxins, Hg). Check the losses for the various parameters.

8.7.4.3 Freeze Drying

Freeze-drying shall be performed in accordance with ISO 16720.

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

Freeze-drying must be performed in such a way that evaporation losses of the substances to be analysed 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 centrifuge prior to freeze-drying. The separated centrifugate shall not contain particles.

NOTE 1 Freeze dried material is hygroscopic, therefore dry the freeze dried material before analysis with Na_2SO_4, if the storage time of the material is longer than 2 h.

NOTE 2 The drying time depends on the type of sludge and the water content. Drying time between 36 h to 96 h may be required.

8.7.5 Sieving / Crushing

The dried sludge is sieved to less than 2 mm. Remove extraneous materials (for example glass) remaining in the sieve. The sample material ≤ 2 mm as well as the fraction of oversized material > 2 mm and extraneous matter shall be weighed and documented.

Mill a representative sub-sample (see 6.10) to a particle size of < 100 µm. Measure the dry residue according to EN 12880.

9 Storing and preservation

9.1 General

Storage begins when the sample is taken. Soil samples are liable to change in the basic characteristics as a result of various causes. 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 standard deals only with the short-term storage of the sample between sampling and, when relevant, sample pre-treatment in the field and during transport to the laboratory were it is delivered for further treatment (analysis).

In most cases the suitable method of preservation will only be storing the sample in a dark and cool environment.

In this European standard on sample pre-treatment general requirements on storing are given which are necessary to guarantee the integrity of the sample when transporting to the laboratory. Further precautions recommended to minimise chemical/physical and biological changes while the sample is stored within a container from the point of sampling to the time of analysis are given in the desk study on sampling desk study on sampling D 2.2 – part C (see Bibliography).

Suitable packing, preservation, storage, transportation and delivery of soil samples are also given in ISO/DIS 10381-8 and ISO 5667-15.

Storage and preservation can affect the integrity of the sample. Therefore the requirements for the storage and preservation have to be in accordance with the analytical method(s) and have to be described in the sampling plan and/or communicated by the laboratory.

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 during transport and storage until it is further treated or analysed. The type and size of the container shall prevent changes in the sample.

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

Suitable sampling containers for example for volatiles as well as for inorganic analyses are coloured plastic bottles with plastic caps and Polytetrafluorethylene (PTFE) seals. For further instructions see the desk study on sampling D 2.2 – part C (see Bibliography).

Suitable materials for sludge and sediments are in many cases polyethylene, polypropylene or glass containers. Polyethylene containers may not be suitable for collecting samples to be subjected to some trace metal analysis (for example mercury); these containers should only be used if preliminary tests indicate acceptable levels of interference. For further instructions see ISO 5667-13 and -15.

When subsequent freezing is used for preservation, the necessary additional space in the sampling containers shall be left.

NOTE A change of preservation method on arrival at the laboratory would often result in repackaging the sample, which could do more harm than good to the sample.

9.3 Preservation

Time between sampling and analysis has to be kept to a minimum to avoid sample alteration – ideally, soil samples should be analysed immediately after collection.

The method of preservation will influence the acceptable time between sampling and analysis. It depends on the components to be determined and the length of time for which the sample must be kept prior to analysis.

NOTE 1 The chosen method of preservation will often be the same from sampling over the whole period before analysis.

NOTE 2 An interruption is acceptable if pre-treatment in the field is necessary.

A number of preservation methods are available for soil, soil related, sludge and sediment samples:

– Air tight storage;

– Dark storage;

– Cooled storage at (4 ± 2) °C

Refrigeration at (4 ± 2) °C is the recommended basic preservation method for samples.

The storage durations depends on the sample material and the storage conditions:

– soil and soil related samples can be stored unlimited without cooling, if they are dried prior to storage

NOTE If the sample is not dried but cooled at (4 ± 2) °C the storage duration depends on the parameter of interest.

– reducing soils (when reducing characteristics are to be maintained): air tight storage, maximum period of storage < 4 d

– sludge and sediments: normally the storage condition is (4 ± 2) °C; however, container and particularly storage duration depend on the parameters measured.

NOTE 1 The maximum storage time for a sample kept in the dark at low temperatures depends on the parameters to be determined. For further information see desk study on sampling, D 2.2 – Part C (see Bibliography).

NOTE 2 Other preservation methods are also available, like drying or freezing, but can only be applied under laboratory conditions.

NOTE 3 The addition of chemical preservatives or stabilizing agents is not a common practice for soil sampling. This is because a single soil sample is usually used for a large number of different determinations, and moreover has to undergo preparation (drying, milling etc.) during which unwanted and unquantifiable reactions of the preservatives may occur.

NOTE 4 The use of liquid nitrogen for immediate deep freezing of soil sample in vapour phase is effective, the use of containers made of stainless steel (not chromium or nickel plated) is recommended.

Freezing of sludge or addition of chemicals to sludge samples is recommended for determining organic constituents. All means of preservation, if practical, should be carried out in the field prior to transportation.

If final preservation methods are not possible in the field, the sludge sample should be transported in coolers filled with ice to retain the integrity of the collected material. To avoid loss of volatile species, samples should be collected in a completely filled container, overfilling it before capping or sealing. Temperature is the most important factor affecting the samples, from the time of sample collection through handling to the final analyses. Samples that are to be frozen may simply be placed in a cooler with dry ice. Any deviation should be recorded in a sampling protocol.

More detailed guidance on specific sample preservation methods for sludges and sediments are given in ISO 5667-13 and -15.

10 Sampling plan

The sampling plan has to incorporate instructions on pre-treatments in the field, preservation and storing between sampling and testing. Especially, instructions should be given if minor (small) changes and major changes during sampling and pre-treatment are necessary. For major changes which can have effects on the results of sampling (e. g. necessity of pre-treatment: pre-treatment was not planned but appears to be necessary due to some reasons) the consultation of the project manager or laboratory is obligatory.

Preservation techniques, storage time, sample containers, collection of duplicate samples and blanks for quality control and quality assurance should be established at the design phase of the sample programme, in consultation with the laboratory analyst.

11 Report

The report shall include the following information:

a) a reference to this European standard;

b) the date and time of sampling;

c) the sampling site;

d) information on any sample preservation technique used;

e) information on any specific sample storage requirements;

f) pre-treatments in the field;

g) major and minor (small) changes during sampling and pre-treatment;

h) which processes, procedures and apparatus were used, including the drying temperature;

i) a complete identification and description of the sample, including the presence (and if necessary relative masses) of stones, fragments of glass, detritus, etc., odour (if any) and colour;

j) any details not specified in this European standard or which are optional, and any other factors which may have affected the results.

Annex A

(informative)

Sub-sampling methods

A.1 Long pile and alternate shovel method

This sub-sampling method is suitable for samples in excess of approximately 100 kg.

Identify the maximum particle size of the sample and determine the minimum size of the sub-sample(s) according to table 1. When the minimum size of the sub-samples 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 7.5. The sub-sampling process shall be stopped when the size of the sub-sample is equal to or larger than the minimum size of the sub-sample 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 soil sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. For samples in excess of approximately 500 kg, the use of a mechanical shovel is to be preferred above the use of a (manually handled) spade.
- When the entire soil 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:
  - Taking a shovelful from the base of the cone 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 soil 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 size of the sub-sample (but no less than the minimum size of the sub-sample in accordance table 1).
- Transfer the sub-sample to an appropriate sample container in accordance to clause 9.

A.2 Coning and Quartering

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

- Identify the maximum particle size of the sample and determine the minimum size of the sub-sample(s) according to table 1. When the minimum size of the sub-samples 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 7.5 or 8.4.2. The sub-sampling process shall be stopped when the size of the sub-sample is equal to or larger than the minimum size of the sub-sample 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 soil sample into a conical pile on the protective floor covering, placing each shovelful on the top of the preceding one. For samples in excess of approximately 500 kg, the use of a mechanical shovel is to be preferred above the use of a (manually handled) spade. Manual handling is preferred for samples smaller than 100 kg.

- When the entire soil 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 one of the following methods:

  - 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 soil 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 soil is equal to the desired size of the sub-sample (but no less than the minimum size of the sub-sample in accordance with table 1).

  - Method 2:

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

    - 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 % (m/m). 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 soil is equal to the desired size of the sub-sample (but no less than the minimum size of the sub-sample in accordance with table 1).

– Transfer the sub-sample to an appropriate sample container in accordance to clause 9.

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 sub-samples when the areas to be sub-sampled are not previously separated (for instance by the metal cross as described in the first method of quartering).

A.3 Riffling

The use of a riffle box is possible when the soil is dry enough to allow free flow of the soil 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 size of the sub-sample(s) according to table 1. When the minimum size of the sub-samples 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 7.5. The sub-sampling process shall be stopped when the size of the sub-sample is equal to or larger than the minimum size of the sub-sample 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 soil 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 soil 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 soil to be sub-sampled.

– Using a shovel or container, pour the material into the riffle box. It is essential that the soil is poured evenly over the whole riffle in order to prohibit biased sub-sampling.

– Remove one sub-sample 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 % (m/m). 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 soil is equal to the desired size of the sub-sample (but no less than the minimum size of the sub-sample in accordance with table 1).

  – Transfer the sub-sample to an appropriate sample container in accordance to clause 9.

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 soil is dry enough to allow free flow of the soil 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 size of the sub-sample(s) according to table 1 and calculate if the reduction ratio of the divider will result in a sub-sample that is equal to or larger than the minimum size of the sub-sample. 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 sub-samples(s) in (an) appropriate sample container(s).
- When necessary repeat the process of sub-sampling be using one or more of the resulting sub-samples until a sub-sample of the required size is obtained (but is no less than the minimum size of the sub-sample in accordance with table 1).
- Transfer the sub-sample to an appropriate sample container in accordance to clause 9.

A.5 Application of mechanised turntable (rotating divider)

The mechanised turntable comprises a of 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 soil 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 soil is transferred into the slot.
- After completion of the division process, one or more of the sub-samples is (are) collected.
- Check the mass of one of the sub-samples. If the mass is not equal to the product of the total mass and the inverse number of sub-samples in the rotating divider, allowing a variation of ± 10 % (m/m), all sub-samples shall be added and the sub-sampling step shall be repeated.
- The sub-samples obtained are (if necessary) divided again, until a sub-sample of the required size is obtained, or until the minimum sample size is achieved, see Table 1.
- Transfer the sub-sample to an appropriate sample container in accordance to clause 9

Annex B

(informative)

Notes on homogenization, initial sample mass and extraction

(for extraction of trace elements in soils using ammonium nitrate solution)

Homogenization of freshly collected samples

Depending on their cohesion, freshly collected samples should be forced by hand through a 2 mm, 5 mm or 8 mm screen using gloves. For samples of mineral soils, particles exceeding about 2 mm in diameter can be picked out by hand. If homogenization is inadequate, larger sample masses may be extracted (e. g. 100 g of soil with 250 ml of ammonium nitrate solution), but the ratio of air-dried or freshly collected soil to solution shall be kept constant in order to obtain reproducible results.

Extraction of organic horizons

Samples from organic horizons shall be weighed out in the freshly collected state since dried samples are frequently hydrophobic or absorb only some of the ammonium nitrate solution. The mixing of the sample can be improved by increasing the amounts extracted (e. g. 40 g of soil with 100 l of ammonium nitrate solution) and adding glass beads (e.g. 20 g of beads with a diameter of 3 mm). The ratio of air-dried or freshly collected soil to solution shall be kept constant in order to obtain reproducible results.

Extraction of reductomorphic horizons

Samples from reductomorphic horizons shall be processed with oxygen excluded from the beginning of sampling to the extraction. Extraction should be carried out as soon as possible after sample preparation (in particular the reduction in size of large aggregates).

Annex C

(informative)

Examples of apparatus

Examples of apparatus given in clauses 6.1, 8.5.3 are illustrated in figures C.1 to C.4.

The design of a mechanical sample divider illustrated in figure C.2 has been found satisfactory but alternative designs may be employed, provided that the essential requirements are fulfilled.

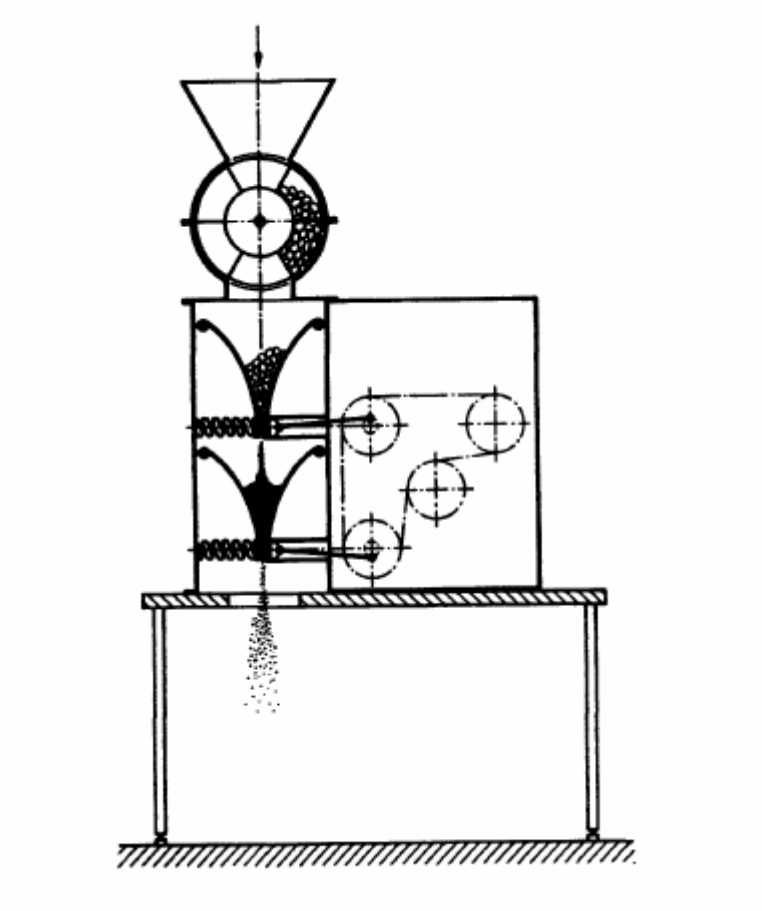

Figure C.1 — Example of a mechanical soil crusher

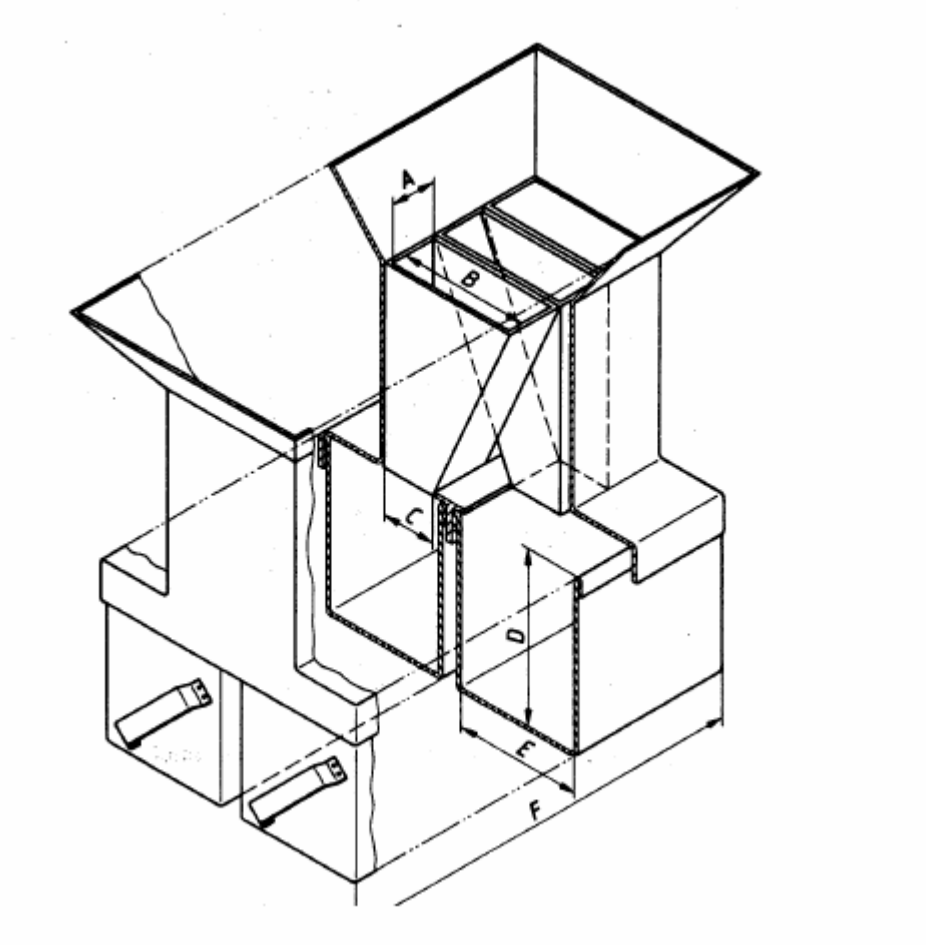

Figure C.2 — Example of a mechanical sample divider

Table C.1 — Dimensions of a mechanical sample divider.

All dimensions, except A, are approximate only.

Maximum size of sample mm	Number of slots	Internal dimensions			Internal dimensions of the boxes (three required)		
		A mm	B mm	C mm	A mm	B mm	C mm
40	8	50	150	70	230	150	400
20	10	30	130	40	150	100	300
10	12	15	80	30	120	90	200
5	12	7	20	15	50	50	90
2	12	5	20	15	50	50	90

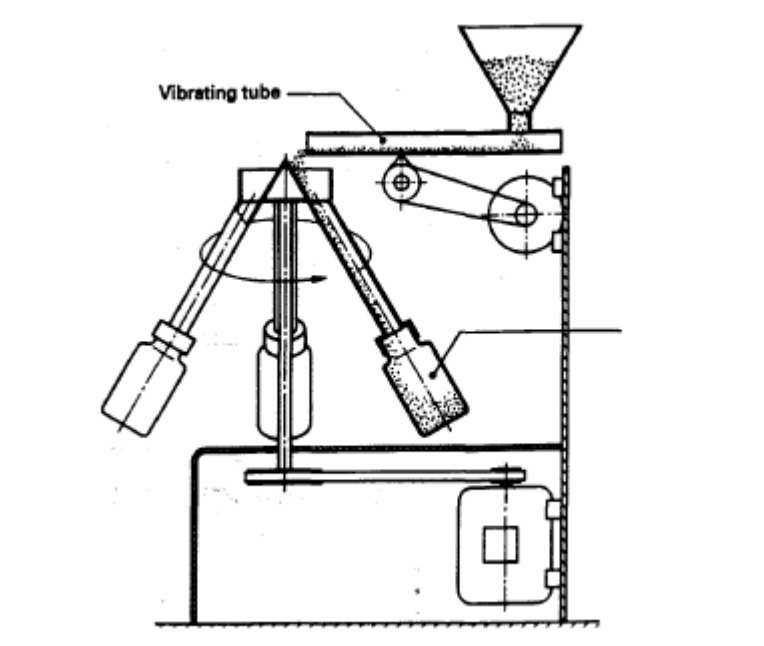

Figure C.3 — Example of a mechanical sub-sampler

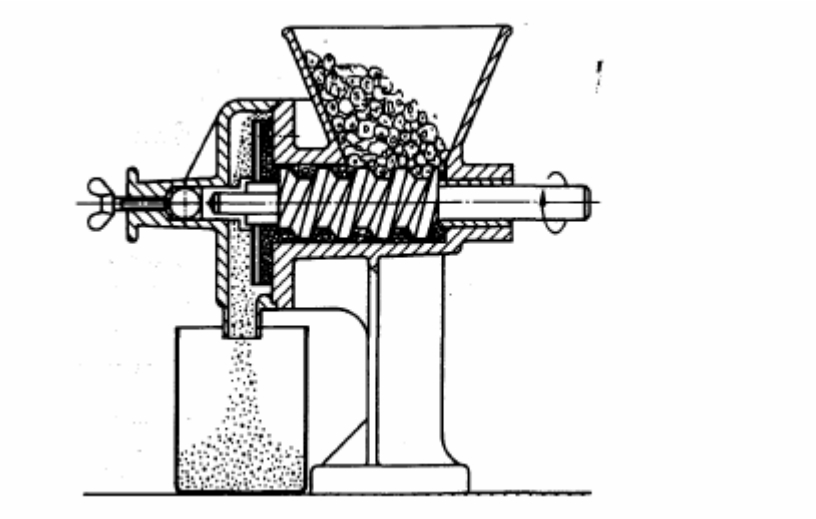

Figure C.4 — Example of a mechanical mill

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[3] ISO 5667-13 Water quality – Sampling – Part 13: Guidance on sampling of sludges from sewage and water-treatment works

[4] ISO 5667-15 Water quality – Sampling – Part 15: Guidance on preservation and handling of sludge and sediment samples

[5] ISO 10381-4 Soil quality – Sampling – Part 4: Guidance on the procedure for investigation of natural, near-natural and cultivated sites

[6] ISO 10390 Soil quality – Determination of pH

[7] ISO 11074-2 Soil quality – Vocabulary – Part 2: Terms and definitions relating to sampling

[8] ISO 11277 Soil quality – Determination of particle size distribution in mineral soil material – Method by sieving and sedimentation

[9] ISO 11466 Soil quality – Extraction of trace elements soluble in *aqua regia*

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