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Characterization of waste — Preparation of test portions from the laboratory sample

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National foreword

This British Standard is the UK implementation of EN 15002:2015. It supersedes BS EN 15002:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/508/3, Characterization of waste.

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

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Characterization of waste - Preparation of test portions from the laboratory sample

Caractérisation des déchets - Préparation de prises d'essai à partir de l'échantillon pour laboratoire

Charakterisierung von Abfällen - Herstellung von Prüfmengen aus der Laborprobe

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Page

Foreword

This document (EN 15002:2015) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by October 2015, and conflicting national standards shall be withdrawn at the latest by October 2015.

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 supersedes EN 15002:2006.

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Introduction

In laboratory practice, very often different analytical procedures are bound to be applied to the laboratory sample that has been taken according to the sampling plan. For this purpose sub-sampling is bound to be applied in a way, that the different test portions are representative for the original laboratory sample with respect to the compounds of interest and the specific analytical procedures. The representativity of the laboratory sample and of the test portions is of major importance to guarantee the quality and accuracy of analytical results. The representativity of the laboratory sample is specified by the sampling plan. This European Standard specifies the correct sequence of operations to ensure the representativity of the test portions.

Safety remarks:

Anyone dealing with waste and sludge analysis is bound to be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples may contain hazardous (e.g. toxic, reactive, flammable and infectious) substances, which can be liable to biological and/or chemical reaction. Consequently it is recommended that these samples should be handled with special care. The gases that may be produced by microbiological or chemical activity are potentially flammable and will pressurize sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations should be followed with respect to all hazards associated with this method.

1 Scope

This European Standard is applicable for the preparation of representative test portions from the laboratory sample that has been taken according to the sampling plan (EN 14899), prior to physical and/or chemical analysis (e.g. preparation of eluates, extractions, digestion and/or analytical determinations) of solid (including monolithic material) and liquid samples and sludge. It is also applicable for the preparation of test portions from digests and eluates for the subsequent analyses.

This European Standard is intended to find the correct sequence of operations and treatments to be applied to the laboratory sample in order to obtain suitable test portions in compliance with the specific requirements defined in the corresponding analytical procedures.

2 Terms and definitions

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

2.1
drying
process of removing water from a sample

Note 1 to entry: For the purpose of test portion preparation, it may be useful to remove just the amount of water that could interfere with other processes involved (e.g. during crushing or milling). In order to minimize the alteration of the sample during test portion preparation, removing the total amount of water present in the sample is not necessarily needed.

2.2
fraction
sample obtained by procedures from the laboratory sample where the properties of interest may be unequally distributed

Note 1 to entry: A fraction may consist of metal pieces, stones, etc.

2.3
granular waste
waste that is neither monolithic, liquid, gas nor sludge

[SOURCE: EN 12457-1:2002, 3.10]

2.4
homogenisation
process of combining of components, particles, layers or phases into a more homogeneous state of the original sample or pre-treated fractions of the sample in order to ensure equal distribution of substances in and properties of the sample

2.5
laboratory sample
sample or sub-samples sent to or received by the laboratory

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 for 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. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

2.6
moderately volatile compounds

sum of semi-volatile organic compounds and moderately volatile inorganic compounds that can be lost during sample preparation

Note 1 to entry: Volatile inorganic compounds of e.g. mercury, arsenic cadmium, thallium can be lost during sample preparation, e.g. heating.

2.7
moderately volatile organic compound; semi volatile organic compound

organic compound having a boiling point above 180 °C (at a pressure of 101 kPa)

Note 1 to entry: This definition includes:

a) mineral oil;

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

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

d) organochlorine pesticides (see ISO 10382).

2.8
monolithic waste

waste which has certain minimum dimensions and physical and mechanical properties that ensure its integrity over a certain period of time in the considered scenario

2.9
particle size reduction

mechanical friction of the sample by milling, grinding, crushing or cutting

2.10
phase separation; fraction separation

process of dividing components, particles or phases if homogenization of the sample is practically not applicable and/or the analysis of different fractions or phases are appropriate

2.11
sample

portion of material selected from a larger quantity of material

2.12
sub-sample

sample obtained after sample size reduction of a larger sample

Note 1 to entry: A sub-sample may be:

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

b) the final sample of multistage sample-preparation;

c) in case of monolithic sample, the sample obtained after cutting or coring.

2.13
sub-sampling
process of selecting one or more sub-samples from a sample or, in case of monolithic waste, the process of cutting or coring to obtain a required regular shape

2.14
test portion; analytical portion
quantity of material of proper size, for measurement of the concentration or other properties of interest, removed from the test sample

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

2.15
test portion of monolithic waste of regular shape
test portion of monolithic waste, obtained either by cutting or coring and for which the surface area can be calculated on the basis of simple geometric formulas

2.16
test sample; analytical sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

2.17
volatile organic compound
compound which is liquid at room temperature (20 °C) and which generally has a boiling point below 180 °C

Note 1 to entry: This includes single-ring aromatic hydrocarbons and other low boiling halogenated hydrocarbons, which are used as solvents or fuels, and some degradation products.

[SOURCE: ISO 10381-7:2005, 3.23, modified — The original term abbreviation and example have been retrieved.]

3 Equipment

For the purpose of preparation of test portions from the laboratory samples appropriate equipment shall be chosen depending on the procedures selected according to Annex A.

In the selection of the type of treatment techniques, one should keep in mind that each of them has some potential impact on analytical results. It can generate loss of the analytes of interest, introduce contamination or alter the physical-chemical properties of the sample.

All glassware and devices that come in contact with the sample shall be made out of a suitable material, chemically compatible with the sample, selected in order to minimize contamination of samples and adsorption or absorption of the analytes (e.g. plastic materials for inorganic elemental analysis, quartz or glass for volatile and organic analytes). Care shall be taken to ensure a good cleaning, in order to avoid cross-contamination of samples.

An informative list of appropriate equipment for the sample treatment procedures is given in Annex C.

4 Interferences and sources of error

The (sub)-sample shall be re-homogenized after any particle size reduction operation that may have resulted in segregation of different sized particles. Care should be taken to avoid loss of material and contamination of the sample via the air, by dust, by the use of the apparatus (e.g. from the ambient laboratory atmosphere or between samples stored or processed close to one another).

Three types of contamination could occur from the apparatus:

a) abrasion;

b) cross-contamination;

c) chemical release.

It is recommended to perform treatment of waste material in a separate room used only for this purpose, especially crushing or sieving.

If the sample has a dust-like consistency or contains (semi)-volatile compounds, part of it may be lost and this may alter its physical-chemical properties.

If the sample is monolithic, changes of surfaces due to cutting or coring, may lead to changes in leaching properties. Due to possible heterogeneity of the samples they may not be fully representative. An option is to use more pieces constituting a test portion.

5 Procedure

5.1 Key concepts

Preparation of the test portion can be a complex process, because of a number of factors: sample type and its physical state, amount of laboratory sample, type and number of determinations to be carried out, etc.

The prepared test portions shall satisfy the following requirements at the same time:

— each test portion shall be as representative as possible of the laboratory sample;

— the amount and the physical state (e.g. particle size) of each test portion shall comply with the requirements of the respective analytical technique;

— for each test portion, no losses of and no contamination with respective analytes of interest should occur.

The preparation of the test portions from the laboratory sample, that has been taken according to the sampling plan, is related to the requested analytical determinations. This means that, if needed, contact shall be established among all involved parties such as the sampler, the customer and the analytical laboratory to achieve the requirements of the standards to be used for the requested determinations.

The preparation of test portions in the laboratory will frequently involve a sequence of operations such as homogenization, phase separation, drying, reducing particle size and sub sampling. Specific forms of these operations are described in A.2 to A.6. A number of decisions on the specific order of these operations for a particular laboratory sample shall be made. In some cases, the sequence of operations to be applied is rather straightforward, but in more complicated cases (e.g. when several determinations with different requirements shall be performed) it can be critical to choose the right sequence of such operations.

NOTE 1 For soil samples more specific procedures are described in ISO 11464 for inorganic parameters or in ISO 14507 for organic parameters.

In order to define the operations to be applied to a laboratory sample to produce one or more representative test portions, three main steps shall be considered:

a) Definition of analytical requirements:

First, the requirements of analytical procedures of interest shall be defined:

1) what methods shall be used;

2) how many test portions are necessary;

3) quantity and properties of the test portions necessary for each analytical procedure;

4) preservation requirements (e.g. time frame, temperature, addition of reagents).

NOTE 2 Indicative amounts of test portions and specific requirements of the analytical methods involved are given in Annex D.

It is recommended to prepare at least five times the amounts needed as test portions for the tests.

b) Definition of sequence of operations

Then, the sequence of operations shall be defined according to the flow sheet (Figure 1), based on the properties of the laboratory sample and the requirements of the analytical procedures: each single operation of this sequence shall be considered like an independent module; available modules are:

1) phase/fraction separation;

2) drying;

3) particle size reduction;

4) homogenization;

5) (sub)-sampling.

For practical reasons it is recommended to group the parameters in a way that test samples with similar requirements can be prepared for several parameters. The same test sample may be used for different parameters if it fulfils the necessary requirements.

Frequently, different determinations shall be performed on the laboratory samples. In those cases, modules shall be combined and/or repeated to obtain sub-samples, finally resulting in different test portions. In order to define the actual sequence of operations to be applied to a given sample, the flow sheet (Figure 1) shall be used.

c) Choice of appropriate procedures

According to the requirements of the respective analytical techniques and the properties of the sample the appropriate sample treatment technique shall be chosen within each module by following the instructions of Annex A. Instructions are given in this annex in which case a particular operation is appropriate to use.

In case of monolithic waste the sequence of operations may be limited to only sub-sampling.

5.2 Sequence of treatment techniques

The flow sheet in Figure 1 describes the procedure to enable decisions on the specific order of treatment operations for a particular laboratory sample in order to yield in representative test portions. It shall be applied on the starting laboratory sample and repeated on all sample fractions or (sub)-samples subsequently obtained during the preparation, in an iterative cycle until all analytical requirements are fulfilled.

If volatile compounds or moderately volatile compounds are parameters of interest this should be considered in the sampling plan and may result in separate samples. In case of a single laboratory sample special care shall be taken in order to avoid losses of the volatile compounds during homogenization and/or reduction of particle size. A preliminary (sub)-sampling without any homogenization step may be necessary (see A.2.4, A.6.4) if the representativity of the remaining sample is not substantially altered.

NOTE In special cases (sub)-sampling without a drying step will not lead to representative (sub)-samples.

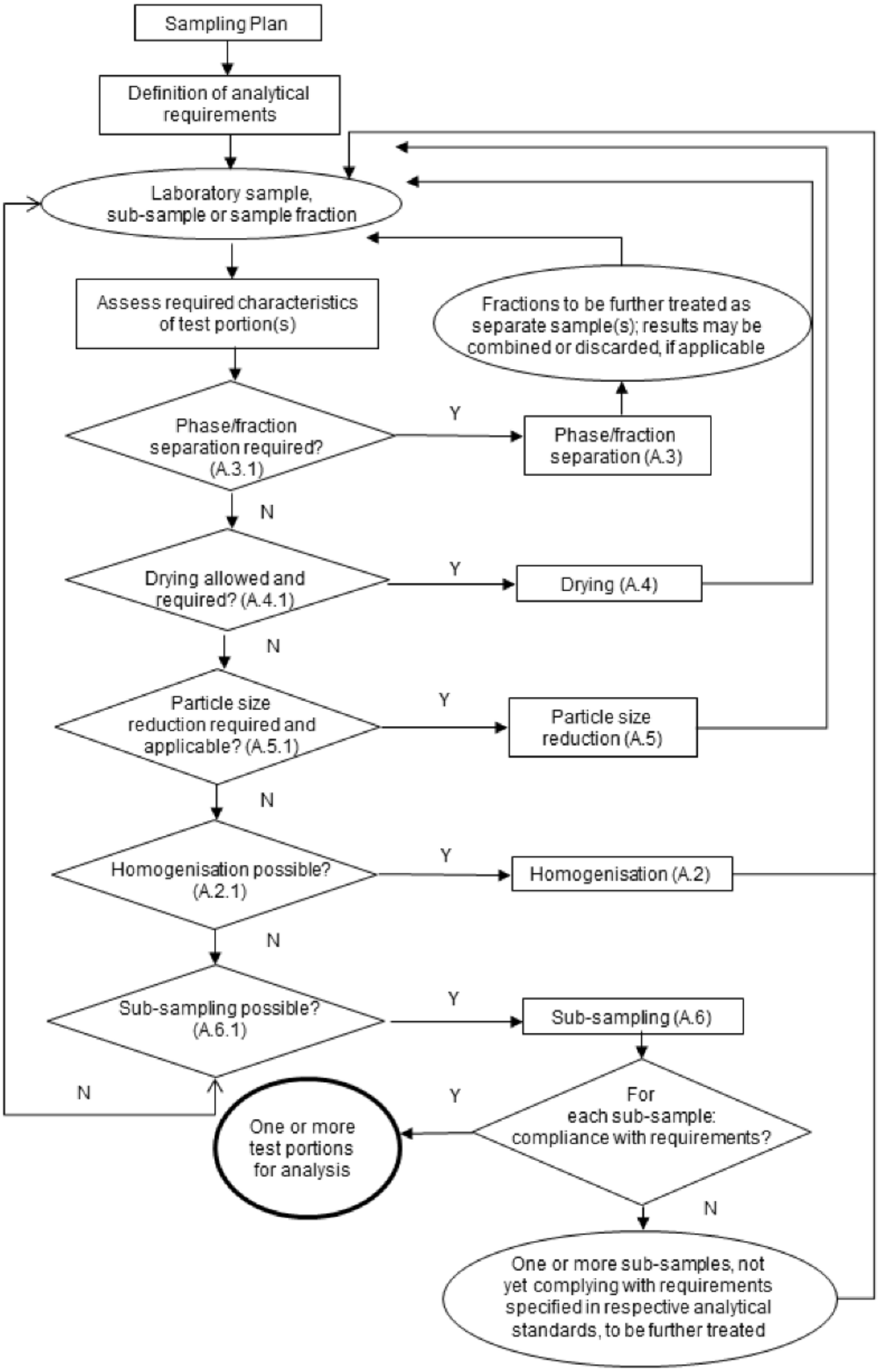


Figure 1 — Flow sheet – sequence of operations

6 Report

The work carried out by the testing laboratory shall be covered by a report which accurately, clearly and unambiguously presents all relevant information.

Each report shall include at least the following information:

a) name, address and location of any laboratory involved in the preparation of the test portions;

b) unique identification of report (such as serial number) and of each page and total number of pages of the report;

c) description and identification of the laboratory sample, (e.g. liquid, solid, granular, monolithic);

d) date of receipt of laboratory sample;

e) reference to this European Standard;

f) reference to the sampling report; if a sampling report is not available, precise reference shall be made to the company or persons responsible for the sampling;

g) whole sequence and operating conditions (procedures and apparatuses) actually applied to the laboratory sample for preparation of test portions;

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

For the subsequent performance of the analyses the dates of the preparation of the test portions shall be available.

Annex A
(normative)

Guideline for choosing sample treatment techniques

A.1 General

The preparation of test samples from a laboratory sample will frequently involve a sequence of operations such as homogenization, phase separation, drying, particle size reduction and sub-sampling. Specific forms of these operations are described in this annex.

The sample treatment techniques prescribed in the analytical standards shall be fulfilled in any case.

In case of monolithic waste, the laboratory sample may consist of one or several monoliths. This implies that some of the sample treatment techniques common for granular waste may not be necessary depending on the requirements of the relevant analytical method.

A.2 Homogenization

A.2.1 General information

Before each operation that implies (sub)-sampling (with the exception of monolithic test portions), a homogenization step is required, in order to guarantee that all (sub)-samples or sample fractions have the same properties and composition. The homogenization technique to be used is chosen depending on the properties of the sample.

In many cases before homogenization particle size reduction may be necessary. Incorrectly executed homogenization can increase the heterogeneity of the sample by segregating particles of different density or grain size. Particle size reduction prior homogenization reduces the risks of segregation in the homogenization process.

If homogenization of a sample is too difficult or even practically impossible (e.g. if the sample contains pieces of plastic or metal), its phases shall be separated and treated as if they were different samples (see A.3).

A.2.2 Solid samples

A.2.2.1 Manual homogenization

When to use it:

- generally usable;
- in cases when mechanical homogenization could lead to loss of volatile compounds of interest.

When not to use it:

- for samples that segregate because of the presence of particles of different size or density;
- for samples with particles of such a large size that homogenization cannot be reached by manual mixing.

Procedure:

Mix the sample with appropriate tool (e.g. shovel, scoop, pestle or mortar). If there is a risk of losses of volatile substances the manual homogenization shall be done very carefully.

A.2.2.2 Mechanical homogenization

When to use it:

- generally usable especially in cases when manual homogenization is not suitable;
- in cases of large sample sizes;
- for solid samples containing particles of nearly the same density (e.g. for materials that do not form “layers” after shaking).

When not to use it:

- for samples that form layers because of the presence of particles of different density; in this case, if homogenization is not possible separate and treat each layer as a different sample (see A.3);
- when the apparatus may heat the sample and loss of volatile analytes can occur during this process; in this case, a manual homogenization shall be performed.

Procedure:

Operate according to the manufactures instructions.

For small sample sizes it may be also possible to use a ball-mill without balls for homogenization.

NOTE Passing and merging the sample repeatedly 3 to 4 times through a mechanical sample splitter prior subsampling is a technique that may provide sufficient homogenization.

A.2.3 Liquid samples

A.2.3.1 Manual homogenization

When to use it:

- For liquid single-phase and visually homogeneous samples (with no visible particles).

When not to use it:

- For liquid samples where particles or emulsions can be visually identified, including sludge and slurries.

Procedure:

Stir with appropriate tool or shake the closed bottle.

A.2.3.2 Mechanical homogenization

When to use it:

- for liquid samples, possibly containing small particles like sludge or slurry, that do not settle or form layers within the time frame of subsampling;
- for liquid samples, containing more than one layer when emulsions may be formed.

When not to use it:

- for liquid samples containing particles that are too large to be homogenized and/or settle within the time frame of subsampling after homogenization, forming layers. In this case, homogenization is not possible, separate and treat each phase as a different sample (see A.3);
- for liquid samples, containing more than one layer when phase separation occurs instantaneously.

Procedure:

Different types of mechanical mixers (impeller, magnetic stirrer, propeller, ultrasonic, emulsifier, etc.) can be used, depending on the properties of the sample. The (sub)-sample shall be taken out as quickly as possible just after the homogenization, in order to avoid settling or phase separation.

A.2.4 Homogenization in case of volatile compounds

If volatile compounds of interest are expected in the sample homogenization should be avoided or shall be done very carefully and quickly to avoid losses. If losses cannot be avoided during homogenization process it may be a solution to take several non-homogenized (sub)-samples for analyses of volatile compounds and calculate the statistical mean as an estimate of the total content. This step should not lead to major alterations of the composition and the representativity of the remaining sample.

NOTE Possible procedures for volatile organic compounds in soil are given in ISO 14507:2003, 8.2 and in EN ISO 22155:2013, Clause 6.

The sampling plan should consider the presence of volatile and moderately volatile compounds.

A.3 Phase/fraction separation

A.3.1 General information

For heterogeneous and multi-phase samples, depending on their nature and on the determinations of interest, one or more techniques of phase separation can be applied to obtain two or more different (sub)-samples that are to be analysed separately. For samples consisting of different fractions, separation of some fractions may be necessary (e.g. nuts, bolts, stones). For this reason, the mass of each separated phase (sub)-sample) shall be directly or indirectly measured after the separation, in order to allow a final weighed combination of different phases analysis results.

The test report shall clearly state the technique(s) used for phase separation, the mass and analytical results related to all (sub)-samples obtained from phase separation, as well as the “weighted” results.

A.3.2 Solid – liquid separation

A.3.2.1 General

Depending on the nature of the sample and the analytical techniques to be used, one or more of the following separation techniques can be applied.

A.3.2.2 Settling

When to use it:

- In every case, provided that complete separation of the two phases can be reached in appropriate time without alterations of analytes of interest.

Settling with appropriate cooling may be the method of choice, if volatile compounds are to be analysed in the sample.

When not to use it:

- when the properties of the sample do not allow a good separation of the phases in a reasonable time;
- if it is necessary to recover the maximum amount possible of liquid phase, or when it is necessary to obtain a solid phase that is as dry as possible, because normally a certain amount of water is still present in the settled phase;
- when biological or chemical alterations are expected during settling time.

NOTE A reasonable time could be 2 h.

Procedure:

Let the bottles stand undisturbed until the phases can be separated with an appropriate technique.

A.3.2.3 Filtration

When to use it:

- when other solid-liquid separation techniques fail or are practically not applicable;
- when the separation should be performed in short time;
- when the liquid phase shall be separated as quantitatively as possible;
- when prescribed by the analytical method (e.g. DOC, AOX).

When not to use it:

- when the properties of the sample do not allow a good filtration in a reasonable time;
- when it can alter the properties of interest of the sample;
- when loss of volatile compounds is to be considered
- in case of adsorption to the filter of analytes of interest.

NOTE A reasonable time could be 1 h.

Do not use vacuum techniques when volatile or moderately volatile compounds of interest are supposed to be in the sample.

Procedure:

Filter the sample with appropriate filtration technique and equipment.

Depending on the type of sample and the analytes of interest the appropriate technique (e.g. pressure filtration, atmospheric pressure filtration, vacuum filtration), the appropriate filter material (e.g. glass-fibre, paper filter, membrane filter) and porosity of the filter shall be chosen. Information on the appropriate technique and filter material shall be taken from the corresponding analytical standards.

A.3.2.4 Centrifugation

When to use it:

- When other solid-liquid separation techniques fail or are practically not applicable: for example, when settling takes too long and filtration could lead to chemical alterations or adsorption.

When not to use it:

- when the properties of the sample do not allow a good separation in a reasonable time;
- when loss of volatile compounds is to be considered.

NOTE 1 A reasonable time could be 30 min.

NOTE 2 Appropriate cooling and the use of centrifuges tubes with small headspace may diminish the loss of volatile compounds.

Procedure:

Apply proper centrifugation conditions. If the first centrifugation does not lead to a good phase separation, centrifugation can be applied for longer time and/or higher acceleration; if centrifugation appears to be not enough effective, filtration can be applied on the whole sample or just on the liquid suspension that is still present after centrifugation.

A.3.3 Liquid – liquid separation

A.3.3.1 Separation with separating funnel

When to use it:

- In most cases, provided that complete separation of the two phases can be reached in appropriate time without alterations of analytes of interest.

When not to use it:

- when losses e.g. by adsorption on the surface of the separating funnel may occur;
- when phases do not separate in reasonable time.

NOTE A reasonable time could be 2 h.

Procedure:

Fill into a separating funnel and let separate the phases.

In some cases addition of salts or acids may accelerate the separation process, provided that no interference with any analytes of interest will occur.

A.3.3.2 Centrifugation

When to use it:

- When other liquid-liquid separation techniques fail or are practically not applicable.

When not to use it:

- when the properties of the sample do not allow a good separation;
- when loss of volatile compounds is to be considered.

NOTE Appropriate cooling may diminish the loss of volatile compounds.

Procedure:

Apply proper centrifugation conditions. If the first centrifugation does not lead to a good phase separation, centrifugation can be applied for longer time and/or higher acceleration.

A.3.4 Solid – solid separation; separation into different fractions

A.3.4.1 General

In cases when the sample is constituted by different macroscopic phases, a separation of such phases can be performed, especially if this can make subsequent particle size reduction, homogenization and (sub)-sampling easier. In cases of visible heterogeneity of separable fractions (e.g. metal pieces, stones) the separation of different fractions may be necessary.

A.3.4.2 Manual separation

When to use it:

- When several fractions, e.g. bolts, nuts, stones, can be distinguished.

When not to use it:

- When contamination or losses of analytes of interest may occur.

Procedure:

Manually select macroscopic pieces of different nature and store them in separate containers, either by hand (with protective gloves) or by using appropriate tools (e.g. tweezers, magnet).

A.3.4.3 Sieving

When to use it:

- when separation of fractions of different particle size is necessary.

When not to use it:

- when contamination or losses of analytes of interest may occur.

Procedure:

Sieve the sample by shaking either by hand or apparatus through sieves with appropriate mesh size and material.

A.4 Drying

A.4.1 General information

Depending on the nature of the sample and the specific requirements of the test portion, a drying step might be needed during sample treatment for test sample preparation. For the purpose of this European Standard, drying is just used to remove the amount of water that could interfere with test sample preparation (e.g. during crushing or milling). For the determination of water content a separate sub-sample may be necessary.

Drying is very likely to introduce analytical errors for volatile or moderately volatile compounds, and should be avoided when not strictly required. If a (sub-)sample for the determination of volatile or moderately volatile compounds is to be dried, the actual drying technique shall be selected in order to minimize losses of volatile compounds. The test report shall clearly state the technique(s) used for drying, along with the mass of (sub)-sample(s) before and after each drying step.

It is likely that a certain drying technique is not applicable for all requested determinations. In such cases, different (sub)-samples shall be dried in different ways, choosing the appropriate sequence of techniques for each one.

The drying time will depend on the technique chosen, the thickness of the layer of the sample, the nature of the sample, moisture content of the sample and of the air and the rate of ventilation.

The “grade of dryness” that shall be reached with the drying step depends on the subsequent treatments to be applied to the sample. Typically, it is not necessary to wait until constant weight: e.g. the sample shall be just dried enough to make crushing, grinding, etc. possible.

A.4.2 Procedures

A.4.2.1 Air drying at room temperature

When to use it:

- in every case where drying can be reached in appropriate time without alterations of analytes of interest;
- if loss of volatile compounds of interest does not occur at room temperature.

When not to use it:

- when time is critical, and the properties of the sample do not allow a good drying in a reasonable time at room temperature, and a higher temperature drying step can be safely applied;
- when degradation may occur, e.g. due to light, biological activity;
- when contamination or reactions with air may occur.

NOTE A reasonable time could be a maximum time of one week.

Procedure:

Spread the sample on the trays in a thin layer and allow it to get dry enough. Care shall be taken in order to minimize possible contamination e.g. by dust and exposition to direct sunlight or light sources. The use of a desiccator may accelerate the drying process for small amounts of (sub-)samples.

A.4.2.2 Oven drying at 40 °C

When to use it:

- if loss of volatile compounds of interest does not occur at 40 °C;
- when time is critical, and the properties of the sample do not allow a good drying in a reasonable time at room temperature.

When not to use it:

- when the properties of the sample do not allow a good drying in a reasonable time at this temperature, and a higher temperature drying step can be safely applied;
- when losses of volatile compounds of interest is suspected even at this temperature.

NOTE 1 A reasonable time could be a maximum time of one week.

Procedure:

Spread the sample on the trays in a thin layer and allow it to get dry in the oven at 40 °C. Air renewal may accelerate the drying process, provided that it does not cause loss of dust-like particles.

NOTE 2 Oven drying at other temperatures may be used if they fit with analytical procedures to be applied.

A.4.2.3 Oven drying at 105 °C

When to use it:

- in cases when loss of volatile analytes is not likely to occur at 105 °C;
- on (sub)-sample for which volatile compounds are not of interest.

When not to use it:

- when losses of volatile compounds of interest is suspected at this temperature;
- in cases auto-ignition can be expected.

NOTE 1 A reasonable time could be a maximum time of one day.

NOTE 2 Losses of most organic compounds and even some inorganic constituents may occur.

Procedure:

Spread the sample on the trays in a thin layer and allow it to get dry in the oven at 105 °C. Air renewal may accelerate the drying process, provided that it does not cause loss of dust-like particles.

A.4.2.4 Freeze-drying

When to use it:

- in cases when loss of volatile analytes is likely to occur with other procedures;
- to facilitate crushing and homogenization of some types of waste (forming cohesive clods e.g. clay).

When not to use it:

- — when another simpler drying technique can be safely applied;
- — when vacuum can lead to loss of analytes of interest.

Procedure:

Operate according to the freeze-drier manufacturer's instructions.

A.4.2.5 Chemical drying

Chemical drying is a process where water free inorganic salts (e.g. sodium sulfate and magnesium silicate) are added to the sample in order to bind its water.

When to use it:

- — in cases when loss of volatile analytes is likely to occur.

When not to use it:

- — when another simpler drying technique can be safely applied;
- — when the addition of salts can lead to alterations of analytes, properties of interest or interfere with subsequent testing;
- — for samples with high water content (e.g. sludges).

Procedure:

Mix thoroughly the weighed (sub-)sample with the weighed chosen salt mixture preferably in a cool environment and closed bottle. An adequate drying time should be ensured (e.g. 12 h to 16 h). The amount of salts added shall be taken into account in the calculations of concentrations.

NOTE A procedure for drying samples containing volatile organic compounds is given in ISO 14507:2003, 8.3.

A.5 Particle size reduction

A.5.1 General information

In order to reduce the intrinsic heterogeneity of the representative test portion, one or more particle-size reduction steps might be needed. Guidance on estimation of the influence of the particle size on the intrinsic heterogeneity of the test portion is given in Annex B.

Typically, particle-size reduction is a multi-step operation that implies the use of a sequence of different techniques; in some cases, it might be necessary to repeat a step until the sample reaches the requested particle size.

Particle-size reduction is a critical step in sample preparation because of potential loss of volatile compounds due to heating, because of loss of dust-like material and because of contamination coming from the equipment itself or from other samples. Care shall be taken in selecting the appropriate equipment and keeping it clean.

The test report shall clearly state the technique(s) and operating conditions used for particle-size reduction. Non-crushable fractions shall be separated (according to A.3.4), weighed and, if needed, analysed as separate sub-samples.

A.5.2 Procedures

A.5.2.1 Crushing

When to use it:

- when representative sub-samples cannot be taken because of large particle size;
- when the particle size of the sample is larger than the allowed inlet particle size for the milling or grinding equipment;
- when the analytical requirements demand a particle size in the mm-order of magnitude.

When not to use it:

- when not applicable because of the nature of the sample (e.g. soft materials, paste-like materials);
- when contamination or losses of compounds of interest may occur by the equipment.

Procedure:

Break large pieces of the sample with a hammer and/or crush the sample with appropriate apparatus (e.g. shredder, jaw-crusher) according to the manufacturer's instructions to the desired particle size. Pieces that are not crushable can be cut with other kinds of manual or mechanical devices and recombined afterwards.

A.5.2.2 Freeze crushing

When to use it:

- when moderately volatile organic compounds are the compounds of interest and particle size reduction is necessary;
- when the sample has a plastic or paste-like consistency;
- when representative (sub)-samples cannot be taken because of large particle size;
- when the particle size of the sample is larger than the allowed inlet particle size for the milling or grinding equipment;
- when the analytical requirements demand a particle size in the mm-order of magnitude.

When not to use it:

- When contamination or losses of compounds of interest may occur by the equipment.

Procedure:

Wrap the sample in a polyethylene container. Fill a Dewar vessel with sufficient liquid nitrogen and immerse the wrapped sample in the liquid nitrogen. Allow the container to stand until the liquid nitrogen no longer boils vigorously. Cool for approximately 10 min. After complete cooling, retrieve the container from the liquid nitrogen and break large pieces of the sample with a hammer and/or crush the sample with appropriate apparatus (e.g. shredder, jaw-crusher) according to the manufacturer's instructions to the desired particle size.

A.5.2.3 Milling/grinding

When to use it:

- when representative (sub)-samples cannot be taken because of large particle size;
- when the requested particle size is less than 1 mm.

When not to use it:

- when the initial particle size is too coarse: a crushing step is required in these cases;
- when losses of volatile compounds or contamination with compounds of interest may occur by the equipment;
- when not applicable, because of the nature of the sample (e.g. soft materials, paste-like materials).

Procedure:

Mill the sample according to the mill's manufacturers instructions until it reaches the desired particle size; in order to prevent losses of volatile analytes, care shall be taken to avoid excessive heating of the sample during milling: a sequence of short and low-speed millings is to be preferred to a long and/or high-speed treatment; it is necessary to let the equipment cool down between each milling operation and the subsequent one. The use of a freeze-head mill can minimize the loss of volatile compounds, if this is suspected.

NOTE If the sample has a plastic or paste-like consistency, freezing it down to low temperatures (e.g. –20 °C to -30 °C) can make it easier to mill.

A.5.2.4 Cutting

When to use it:

- when representative sub-samples cannot be taken because of large particle size;
- when the material of the sample is not hard enough to allow crushing or milling, e.g. plastic, textile.

When not to use it:

- When losses of volatile compounds or contamination with compounds of interest may occur by the equipment.

Procedure:

Cut the sample according to the cutting mill's manufacturers instructions to the desired particle size; in order to prevent losses of volatile analytes care shall be taken to avoid excessive heating of the sample during cutting; it is necessary to let the equipment cool down between each cutting operation and the subsequent one. If loss of volatile compounds is suspected a cooled cutting mill should be used or manual cutting by scissors may minimize the loss.

A.5.2.5 Freeze cutting

When to use it:

- when moderately volatile organic compounds are the compounds of interest and particle size reduction is necessary;
- when the sample has a plastic, paste-like or fibred consistency;

- when representative sub-samples cannot be taken because of large particle size;
- when the analytical requirements demand a particle size in the mm order of magnitude.

When not to use it:

- When losses of compounds of interest or contamination by the equipment may occur.

Procedure:

If necessary wrap the sample in a polyethylene container. Fill a Dewar vessel with sufficient liquid nitrogen and immerse the (wrapped) sample in the liquid nitrogen. Allow the container to stand until the liquid nitrogen no longer boils vigorously. Cool for approximately 10 min. After complete cooling, retrieve the sample from the liquid nitrogen, cut it with a preferably cooled cutting mill according to the manufacturer’s instructions to the desired particle size.

A.6 Sub-sampling

A.6.1 General information

The laboratory sample shall usually be divided into different test portions for the analyses. Most analytical techniques allow only small quantities of test portions to be analysed. Laboratory sample, conversely, can be in some cases very large, so only a small, representative portion of it shall be taken for analyses.

The sub-sampling shall be performed in a way that the obtained sub-samples are as representative as possible, taking into account the quantity of test portion to be analysed, the quantity of laboratory sample, particle size and homogeneity. If the sample is not homogeneous enough, or if particle size is too coarse, a representative portion cannot be taken: one or more particle size reduction steps followed by homogenization steps are needed. For determination of the minimum amount of sample in relation to the particle size and the heterogeneity of the sample the information in Annex B could be taken into account.

Because of practical limitations it might happen in some cases that representative sub-samples cannot be obtained. In this case replicates should be performed.

The test report shall clearly state the way of determination of minimum amount of sub-samples and the technique(s) used for sub-sampling, along with the weight of each analysed fraction.

In the case of monolithic waste, monolithic test portions of simple geometrical forms may be required (cube, cylinder, rectangular). These sub-samples may directly be obtained from the laboratory sample for example by cutting or coring.

A.6.2 Manual division of solid samples by coning and quartering

When to use it:

- in case of a large amount of laboratory sample;
- when the sample is dry enough and clods can be divided manually.

When not to use it:

- when the sample contains clods that cannot be divided;
- when losses of volatile compounds may occur at room temperature;
- for sludge and liquids.

Procedure:

Spread the sample on a flat surface coated by an inert sheet in a circular shape; mix it with a shovel or a scoop and make a cone.

- Divide the sample in four slices, e.g. with a sheet metal cross, of the same shape and size, see Figure A.1:

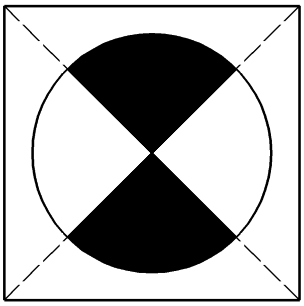


Figure A.1 — Sample division

- Discard two opposite slices, and mix thoroughly the remaining part of the sample.

This sequence shall be repeated until the requested quantity of sub-sample is reached (see Annex B).

A.6.3 Dry cutting

When to use it:

- When representative a monolithic sub-samples cannot be taken because of too large size of the laboratory sample.

When not to use it:

- When losses of volatile compounds or contamination with compounds of interest may occur by the equipment.

Procedure:

Cut the monolithic sample according to the cutting device or core drilling device's manufacturer's instructions to the desired dimensions meeting the requirements of the standard; in order to prevent losses of volatile analytes care shall be taken to avoid excessive heating of the sample during cutting; it is necessary to let the equipment cool down between each cutting operation and the subsequent one.

In case wet cutting is the only option available, then the contact time should be as short as possible.

A.6.4 Mechanical division of solid samples

When to use it:

- when the sample is dry enough and there are no clods in it;

- when the particle size is small enough for the used apparatus.

When not to use it:

- when the sample contains clods that cannot be divided;
- when losses of volatile compounds may occur;
- for sludge and liquids.

Procedure:

Mechanical sample splitters can achieve similar or better results compared to manual division. Divide the sample by using the sample splitter, e.g. riffle box, tyler divider, rotary divider, according to the manufacturer's instructions.

A.6.5 Sub-sampling for volatile compounds

When to use it:

- when losses of volatile compounds may occur by other techniques;
- when no separate sample for volatile compounds is available.

When not to use it:

- when another simpler technique can be safely applied;
- when not applicable, because of the nature of the sample.

Procedure:

If volatile compounds are expected in the sample sub-sampling for the determination of these volatile compounds should be the first step preferably before any other treatment of the sample. Take one or several core test samples by using a core cutter.

NOTE A possible method for coring is described in ISO 14507:2003, 8.2.

A.6.6 Sub-sampling for moderately volatile organic compounds

When to use it:

- when losses of moderately volatile compounds may occur by other techniques.

When not to use it:

- when another simpler technique can be safely applied;
- when not applicable, because of the nature of the sample.

Procedure:

Take carefully sub samples manually after using pre-treatment techniques as chemical drying and grinding after cooling with liquid nitrogen, if applicable.

NOTE Possible methods for manual subsampling are described in ISO 14507:2003, 8.3 and 8.4.

A.6.7 Sub-sampling of sludge and liquid

When to use it:

- for homogeneous sludge and liquids.

When not to use it:

- when the sample contains clods that cannot be divided;
- when precipitate or layers are present in the sample;
- when losses of volatile compounds may occur.

Procedure:

After manual or mechanical homogenization take the appropriate amount of sub-samples. In case of the presence of volatile compounds, the sub-samples should be taken preferably after cooling.

A.6.8 Sub-sampling of monolithic sample

When to use it:

- when the monolithic sample is not in the required geometrical form.

When not to use it:

- this procedure may not be applicable to materials reacting with the leachant, leading for example to excessive gas emission or an excessive heat release

Procedure:

The test sample is prepared by cutting, sawing, coring or by combinations of these operations and shall result in a simple geometrical form of which the length, width, height and/or diameter can easily be obtained with an uncertainty of ± 1 mm.

In case wet cutting is the only option available, then contact time should be as short as possible.

Annex B
(informative)

Relationship between minimum amount of (sub-)sample and particle size

B.1 Formula for the estimation of the minimum amount of (sub-)sample:

The minimum amount of (sub-)sample can be estimated by the following formula (CEN/TR 15310-1):

$$M_{\text{sam}} = \frac{1}{6}\pi \cdot \left(D_{95}\right)^3 \cdot \rho \cdot g \cdot \frac{\left(1-p\right)}{\text{CV}^2 \cdot p} \qquad \text{(B.1)}$$

where (see [61])

M_{sam} is the mass of the sample, in g

D_{95} is the ‘maximum’ particle size (defined as the 95-percentile), in cm

ρ is the average density of the particles in the material, in g/cm^3

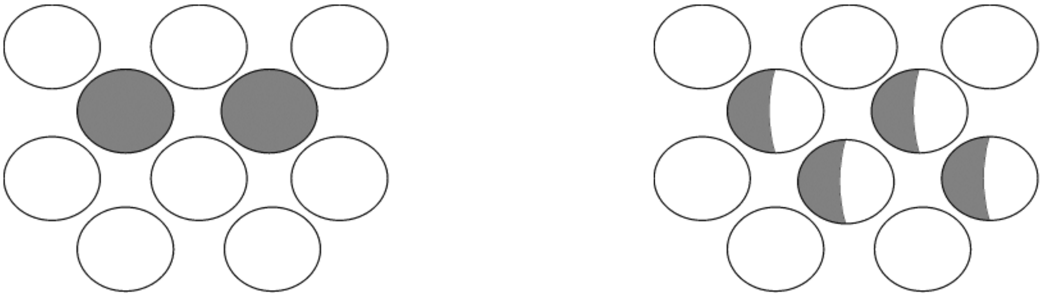
NOTE 1 This parameter does not represent the bulk density of the sample, but the average density of all particles. For sand or soil particles the density amounts 2,6 g/cm^3.

g is the correction factor for the particle size distribution of the material

NOTE 2 This value depends on the ratio between D_{05}/D_{95}, and gives an indication for the particle size distribution. A g value close to 1 reflects a narrow particle size distribution (most particles of equal size), a small g value (<0,01) reflects a broad particle size distribution. For materials as natural soils a typical value is 0,25. This value can be used as default value if the size distribution of a material is not known.

p is the fraction of the particles with the property of interest, in m/m

NOTE 3 If every single particle of the sample would carry the same amount of the parameter of interest, $p = 1$. For parameters being present at a major level in the sample (as e.g. dry matter or loss of ignition) a value $p = 0,1$ can be used. For parameters being present at a minor level (as e.g. trace metals) a value $p = 0,001$ can be used. In Figure B.1 the difference between p-values is illustrated.



a) The sample is made of 10 particles, and two of them carry the analyte of interest.
$p = 0,2$

b) The sample is made of 10 particles, and four of them carry the analyte of interest (the total amount of the analyte is the same as example 1).
$p = 0,4$

Figure B.1 — Particles with property of interest

CV is the desired coefficient of variation caused by the fundamental error

NOTE 4 A typical value for CV is 0,1. Depending of the nature of the waste and the aim of the analysis it may vary from 0,1 to 0,3.

EXAMPLE
Assumption
CV = 0,1
$g = 0{,}25$
$\rho_p = 2{,}6$ g/cm^3

Minimum amount of sample see Table B.1, M_{sam} in g.

Table B.1 — Minimum amount of sample, in gram

	D_{95} = 4 cm	D_{95} = 1 cm	D_{95} = 0,4 cm	D_{95} = 0,2 cm	D_{95} = 0,1 cm	D_{95} = 0,02 cm
very heterogeneous p = 0,001	2 180 000	34 000	2 180	272	34	0,27
heterogeneous p = 0,02	107 000	1 670	110	13	1,7	0,01
rather homogeneous p = 0,1	19 600	306	20	2,5	0,3	0,002

B.2 Empirical rule

An empirical rule that may be used for estimation of the minimum amount of rather homogeneous (sub-)samples with particles of high density (e.g. sand) is given by:

mass (kg) = 0,06 × maximum particle size (mm)

For rather homogeneous waste types and small amounts of (sub-)samples this rule proved to be applicable too especially for samples with a grain size smaller than 1 cm.

Minimum amount of sample see Table B.2, M_{sam} in g.

Table B.2 — Empiric minimum amount of sample, in gram

D_{95} = 4 cm	D_{95} = 1 cm	D_{95} = 0,4 cm	D_{95} = 0,2 cm	D_{95} = 0,1 cm	D_{95} = 0,02 cm
2 400	600	240	120	60	12

Annex C
(informative)

Sample treatment equipment

The preparation of test samples from a laboratory sample will frequently involve a sequence of operations such as homogenization, phase/fraction separation, drying, particle size reduction and sub-sampling. Equipment to be used for these operations see Table C.1.

Table C.1 — Sample treatment equipment

Equipment	A.2 homogenization	A.3 phase/fraction separation	A.4 drying	A.5 particle size reduction	A.6 sub-sampling
mill	X			X	
shredder	X			X	
cutting device				X	X
core drilling device					X
mechanical mixer	X				
magnetic stirrer	X				
ultrasonic	X				
emulsifier	X				
hammer				X	
jaw-crusher				X	
pestle and mortar	X			X	
shovel or scoop	X				X
sheet cross					X
tweezer		X			
magnet		X			
riffle box					X
tyler divider					X
rotary divider					X
centrifuge		X			
separating funnel		X			
filtration equipment		X			
sieves		X		X	X
drying oven			X		
freeze drier			X		
desiccator			X		
balance		X	X	X	X

Annex D
(informative)

Examples for analytical methods

Examples for requirements for test portions from liquid samples of different analytical methods see Table D.1.

Table D.1 — Requirements for test portions from aqueous laboratory samples

Analysis or test	Standard	Indicative amount of test portion[a]	Specific requirements/conservation[b]
pH	EN ISO 10523	*50* ml	– measure as quickly as possible but not later than 6 h after sampling
conductivity	EN 27888	*50* ml	– measure as soon as practicable, store in the dark at 4 °C (however analyse after equilibrium at 25 °C)
dry matter	EN 14346	– drying at 105 °C: residue obtained at least 0,5 g – azeotropic distillation: 100 g	– store biologically active samples at least –18 °C
chloride, bromide, orthophosphate, and nitrate	EN ISO 10304-1	*2* ml	– filter through a membrane filter (of pore size 0,45 µm) – analyse as soon as possible, stabilize by cooling between 4 °C and 6 °C or by deep-freezing until the analysis is performed – add eluent concentrate and filter the sample again prior to analysis
	EN ISO 10304-1	*2* ml	– filter through a membrane filter (of pore size 0,45 µm) – analyse as soon as possible, stabilize by cooling between 4 °C and 6 °C or by deep-freezing (–16 °C to –20 °C) until the analysis is performed – add eluent concentrate and filter the sample again prior to analysis
sulfate	EN ISO 10304-1	*2* ml	– filter through a membrane filter (of pore size 0,45 µm) – analyse as soon as possible, stabilize by cooling between 4 °C and

Analysis or test	Standard	Indicative amount of test portion[a]	Specific requirements/conservation[b]
			6 °C or by deep-freezing until the analysis is performed – add eluent concentrate and filter the sample again prior to analysis
	EN ISO 10304-1	*2* ml	– in the presence of sulfide add zink acetate – filter through a membrane filter (of pore size 0,45 µm) – analyse as soon as possible, stabilize by cooling between 4 °C and 6 °C or by deep-freezing (–16 °C to –20 °C) until the analysis is performed – add eluent concentrate and filter the sample again prior to analysis
Fluoride	EN ISO 10304-1	*2* ml	– filter through a membrane filter (of pore size 0,45 µm) – analyse as soon as possible, stabilize by cooling between 4 °C and 6 °C or by deep-freezing until the analysis is performed – add eluent concentrate and filter the sample again prior to analysis
	ISO 10359-1	*50* ml	– analyse as soon as possible, preferably within 3 days
Nitrite	EN ISO 10304-1	*2* ml	– fill the sample vessel completely – filter through a membrane filter (of pore size 0,45 µm) – analyse as soon as possible, stabilize by cooling between 4 °C and 6 °C or by deep-freezing until the analysis is performed – add eluent concentrate and filter the sample again prior to analysis
	EN 26777	40 ml	– analyse as soon as possible, within 24 h, – stabilize by cooling in darkness between 2 °C and 5 °C
	EN ISO 10304-1	*2* ml	– fill the sample vessel completely and seal with airtight stopper – filter through a membrane filter (of pore size 0,45 µm) – analyse as soon as possible, stabilize by cooling between 4 °C and 6 °C or by deep-freezing (–16 °C to –20 °C) until the analysis is performed – add eluent concentrate and filter the sample again prior to analysis
Nitrite and nitrate nitrogen and the sum of both	EN ISO 13395	*10* ml	– store at 2 °C to 5 °C and analyse within 24 h – filtration is necessary if it contains particulate matter

Analysis or test	Standard	Indicative amount of test portion[a]	Specific requirements/conservation[b]
Ammonium	EN ISO 11732	*10* ml	– analyse the sample immediately after collection or add sulfuric acid (pH = 2), store at 2 °C to 5 °C and analyse within 24 h – In exceptional cases, if sample is membrane filtered after acidification store up to 2 weeks
	ISO 7150-1	maximum 40 ml	– analyse the sample as quickly as possible or add sulfuric acid (pH < 2), store at 2 °C to 5 °C – if sample is coloured or saline a test portion shall be prepared by distillation (ISO 5664)
easily liberatable cyanides	ISO 6703-2	100 ml	– analyse the sample as soon as possible, keep it cool and in the dark – add immediately after sampling sodium hydroxide
	EN ISO 14403 (all parts)	*10* ml	– immediately after sampling and filtration, bring to pH 12 – analyse as soon as possible but at the latest within three days – store the sample in the dark
Cyanides	EN ISO 14403 (all parts)	*10* ml	– immediately after sampling and filtration, bring to pH 12 – analyse as soon as possible but at the latest within three days – store the sample in the dark
Metals (As, Ba, Cd, Co, Cr, Cu, Mo, Ni, Pb, V, Zn) and total S	EN ISO 11885	*10* ml	– dissolved elements: filter the sample (0,45 µm membrane filter) as soon as possible, acidify the filtrate with 0,5 ml of nitric acid per 100 ml sample (pH < 2) – particulate elements: filter the sample (0,45 µm membrane filter) as soon as possible. transfer the filter to a container – total elements: acidify the sample with 0,5 ml of nitric acid per 100 ml sample (pH < 2)
Metals (Co, Ni, Cu, Zn, Cd, Pb)	ISO 8288	*50* ml	– dissolved elements: filter the sample (0,45 µm membrane filter) as soon as possible, acidify the filtrate with nitric acid (pH 1 to 2) – total elements: acidify the sample with nitric acid (pH 1 to 2)
As	EN ISO 11969	*10* ml	– add 20 ml HCl per litre sample or more until pH < 2
Chromium VI	ISO 11083	50 ml	– analyse as soon as possible – if no reducing or oxidizing substances are present in the sample: adjust

Analysis or test	Standard	Indicative amount of test portion[a]	Specific requirements/conservation[b]
			pH between 7,0 to 7,2, allow to precipitate for at least 2 h and filtrate – if reducing or oxidizing substances are present: carry out additional preservation.
Mercury	EN ISO 12846	100 ml (maximum 1 000 ml)	– add 10 ml $K_2Cr_2O_7$ (5 g/l) – adjust pH to 1
AOX	EN ISO 9562	*100* ml	– analyse as soon as possible, store at 4 °C or deep freezing – add 2 ml of nitric acid per litre sample (pH < 2) and fill completely the bottle – add sodium sulfite if oxidizing agents are present
phenol index	ISO 6439	*500* ml	– analyse within 4 h, or – acidify with sulfuric acid (pH = 4) – add 1 g of copper sulfate per litre of sample – store at 5 °C to 10 °C, analyse within 24 h
	EN ISO 14402	5 ml	– analyse immediately, alternatively add sulfuric acid or hydrochloric acid (pH = 2) – store in the dark at 2 °C to 5 °C, analyse within 24 h
TOC/DOC	EN 1484	*5* ml	– fill the bottle completely – store at 2 °C to 5 °C, analyse within 7 days or keep at –15 °C to –20 °C for several weeks – if biological activity is expected, acidify to pH with phosphoric acid – if volatile organic compounds are suspected, do not acidify and analysis within 8 h – for DOC determination, filtrate through a 0,45 µm membrane filter before analysis

[a] All text and information are taken from the respective standards. Where not explicitly cited in the standard an amount of test portion is suggested in *italics*.

[b] All text and information are taken from the respective standards.

Most of the parameters above listed in the above table were validated in eluates (EN 16192).

Examples for requirements for test portions from solid samples of different analytical methods see Table D.2.

Table D.2 — Requirements for test portions from the solid laboratory sample

Analysis or test	Standard	Indicative amount of test portion[a]	Specific requirements[b]
Hydrocarbons	EN 14039	20 g	– keep sealed in darkness at 4 °C and extract within 1 week or store at –18°C or lower – un-dried
	EN 14345	20 g	– keep sealed in darkness at 4 °C and extract within 1 week or store at –18°C or lower – un-dried
Halogens (chloride, bromide, fluoride, iodide), sulfur	EN 14582	– Calorimetric bomb: 0,1 g to 1 g – Schöniger flask: 10 mg to 50 mg	– store at 4 °C and analyse within 1 week or store at –18 °C or lower – analyse untried sample – particle size less than 200 µm
Metals	EN 13656	– mass above 200 mg – closed vessels: 200 mg to 500 mg – semi-open vessels: 200 mg to 1000 mg	– particle size of 250 µm – amount of organic carbon < 100 mg in the case of closed digestion vessel
	EN 13657	– mass above 200 mg – closed vessels: 200 to 500 mg – semi-open vessels: 200 to 1000 mg – reflux systems: 1 g to 10 g	– particle size of 250 µm – amount of organic carbon < 100 mg in the case of closed digestion vessel
	EN 15309	*10 g dried sample*	
Dry matter	EN 14346	– drying at 105 °C: residue obtained at least 0,5 g – azeotropic distillation: 100 g	– store biologically active samples at least –18 °C
PCB	EN 15308	*20* g	– grain size preferably below 1 mm
Cr VI	EN 15192	*2,5* g	– un-dried sample
Loss on ignition	EN 15169	0,5 g to 5 g	
TOC	EN 13137	*1* g	– store at 4 °C and analyse within 1 week or store at –18°C or

Analysis or test	Standard	Indicative amount of test portion[a]	Specific requirements[b]
			lower – particle size less than 200 µm – loss of volatiles shall be prevented (if possible, sample should be analysed without drying)
Leaching test	EN 12457–1	– test portion: 175 g dry mass	– grain size of at least 95 % (mass) less than 4 mm
Leaching test	EN 12457–2	– test portion: 90 g dry mass	– grain size of at least 95 % (mass) less than 4 mm
Leaching test	EN 12457–3	– test portion: 175 g dry mass	– grain size of at least 95 % (mass) less than 4 mm
Leaching test	EN 12457–4	– test portion: 90 g dry mass	– grain size of at least 95 % (mass) less than 10 mm
Leaching test	EN 14997	– test portions of 15 g, 30 g or 60 g as dry matter	– grain size of 95 % less than 1 mm – drying temperature shall not exceed 40 °C
Leaching test	CEN/TS 14405	– 5 cm diameter column: volume after compaction 0,8 l – 10 cm diameter column: volume after compaction 3,2 l	– depending on particle size different column type is used – drying temperature shall not exceed 40 °C
Leaching test	EN 15863	No requirement	Minimum 4 cm diameter
Leaching test	CEN/TS 15862	No requirement	Minimum 4 cm diameter
Leaching test	CEN/TS 15864	No requirement	Minimum 4 cm diameter

[a] All text and information are taken from the respective standards. Where not explicitly cited in the standard an amount of test portion is suggested in *italics.*

[b] All text and information are taken from the respective standards.

Annex E
(informative)

Examples for preparation of test samples

E.1 Example 1

This example shows the application of this procedure for the preparation of two test portions, each requiring different conditions for preparation.

a) Sample: 10 kg of fine-grain (D_{95} = 0,8 mm) fly ash sample, with high content of dry matter

b) Required tests:

   1) dry matter - moisture content (EN 14346)

   2) compliance leaching test (EN 12457-2) and subsequent analysis of eluate for pH (EN ISO 10523), and Cr, Cu, Mo, Zn (EN ISO 11885)

Preparation of test samples corresponding to five times the amount of test portions (see Annex D) is recommended. According to the requirements of the analytical procedures to be applied, the properties of the test samples to be prepared are given in Table E.1.

Table E.1 — Requirements of the test samples

Test sample ID	Test to be applied	Amount of test sample (5 × test portion)	Physical characteristics of test sample	Constraints for preparation
TS 1	EN 14346 (dry matter – moisture content)	No fixed value; for practical reasons, an amount of 25 g or more, up to 500 g, is appropriate	No special needs	Loss of water need to be prevented during test portion preparation
TS 2	EN 12457–2 (compliance leaching test)	Corresponding to 425 g to 475 g of dry matter	95 % < 4 mm	Drying is allowed (at 40 °C max.) only if strictly necessary for eventual crushing

Actually, the preparation of the two test samples need to be done separately, because it is necessary first to prepare test sample for ISO 11465 and analyse it for dry matter; afterwards, it will be possible to calculate the actual amount of test sample “as is”, needed for EN 12457-2 because for the preparation of eluates drying at maximum 40 °C is allowed.

The particle size of the original sample fits with the needs of both analytical standards, so particle size reduction should not be necessary provided that required amount of test samples are still representative for the original sample. Criteria from Annex B need to be taken into account to assess the minimum amount of representative (sub-)sample that can be handled, as described below.

Data to be used for the application of the statistical model for the determination of the minimum (sub-)sample size:

— D_{95} sieve opening that lets pass 95 % by mass of the sample (D_{95}) = 0,08 cm;

- — ρ average particle density (the actual value can be measured, in this example it has the same value as sand) = 2,6 g/cm^3;
- — g correction factor for the particle size distribution (depending on D_{05}/D_{95} – the actual value can be measured, in this example it has a similar or even broader distribution compared to a typical soil) = 0,25;
- — p the fraction of the particles with the property of interest; it is difficult to determine the actual value to be used for each case, so here only an estimated value will be used. This sample is rather homogeneous by nature, so for determination of dry matter a value of 0,1 can be safely applied; for leaching test and subsequent analysis for the listed elements (that are supposed to be present at minor or trace levels), a conservative smaller value like 0,001 need to be used;
- — CV a coefficient of variation of 0,1 (10 %) is commonly used.

Application of the statistical model of Annex B leads to these values:

- — Minimum mass of Test Sample 1: m_1 = 0,16 g
- — Minimum mass of Test Sample 2: m_2 = 17,41 g

Since the input values for “p” were not actually measured but just estimated, the results of the above calculations needs to be considered as “estimations” as well, and may be rounded to m_1 = ~0,2 g and m_2 = ~20 g for simplicity.

As the amount of test sample for both analytical standards is larger than the estimated minimum amount of test samples, it is considered to be representative: sub-sampling can be applied directly without any need for particle size reduction. The combination of flow sheet and Annex A is given in Table E.2.

Table E.2 — Application of flow sheet and Annex A for preparation of Test Sample TS 1

	(Sub)-sample	What to do (flow sheet, Figure 1)	How to do it (Annex A)
1		Clearly identify the nature of the sample; contact customer and sampling team if needed; consult standards regarding requested analytical determinations. Done: see above	
2		Assess number and characteristics of test portion(s) to be prepared from this sample fraction, by consulting relevant analytical standard(s): One test sample, named TS 1 (25 g to 500 g), as described above	
3		Assess the eventual need of separating fractions or phases, if applicable (e.g. nuts, bolts, stones, layer): No separation of fractions needed, because the material is already finely grained	
4		Drying allowed and required? Not allowed	
5		Particle size reduction required and applicable? No, the sample is already finely grained	

	(Sub)-sample	What to do (flow sheet, Figure 1)	How to do it (Annex A)
6		Homogenization possible? Yes, because the sample appears easy to handle and it doesn't show the tendency to form aggregates	
7	10 kg, < 0,8 mm	**Perform homogenization**, see A.2	A.2 describes both manual and mechanical homogenization; the use of a mechanical mixer is a concern for possible loss of water by evaporation, and moisture content is the analyte of interest: so, in this case a manual homogenization is to be preferred.
8		**Perform (sub)-sampling** (see A.6)	The minimum representative amount for this sub-sample is m_1 = ~0,2 g, as calculated above. The required amount of test sample is 25 g to 500 g. A.6 describes two possible procedures: manual and mechanical division; in this case, both are applicable. For example, manual division is applied by quartering starting from the 10 kg of sample; this way, sub-samples of different weight (5 kg; 2,5 kg; 1,25 kg; 0,62 kg; 0,31 kg; 0,15 kg) are obtained, the latter being the interesting one for this case (the 0,31 kg fraction could be used as well, but for practical reasons it is preferable to work with the fraction of 0,15 kg). Hint: it would be useful not to remix again the remaining sub-samples at this point, because there are chances that one of them could be used for preparation of Test Sample TS 2 later.
9	Sub-sample of 150 g, < 0,8 mm	Does the sub-sample comply with requirements of EN 14346? Yes, sub-sample now becomes the Test Sample TS 1, to be analysed for dry matter; preparation of single test portions from TS 1 will be done according to A.6.	

Now, determination of dry matter is performed on test portion TS 1. For example, suppose that the result of dry matter determination on TS 1 is 92 %. This means that the required amount of TS 2 is 462 g to 516,5 g "as is", corresponding to 425 g to 475 g of dry matter.

Now the test portion TS 2 needs to be prepared by following a similar sequence of operations. If sub-samples from the step 8 above were not remixed together, it is possible to save some work by starting from the fraction of 620 g and applying quartering for two times, obtaining fractions of approximately 310 g and 155 g respectively; recombine a fraction of 310 g with a fraction of 155 g to obtain an amount of 465 g that fits with TS 2 requirements.

The test portion TS 2 can now be used for the leaching test extractions according to EN 12457-2. For each extraction, a clean aqueous solution will be obtained and analysed for pH, Cr, Cu, Mo and Zn. Representative test samples of each aqueous extract need to be prepared for each analysis: again, refer to the flow sheet (Figure 1) and Annex A, as follows.

— sample: one litre of clean solution extracted according to EN 12457-2;

— required tests: pH (EN ISO 10523);

— Cr, Cu, Mo, Zn (EN ISO 11885).

Preparation of test samples corresponding to five times the amount of test portions is recommended. According to the requirements of the analytical procedures to be applied, the properties of the test samples to be prepared are given in Table E.3:

Table E.3 — Requirements of the test samples

Test sample ID	Test to be applied	Amount of test sample (5 × test portion)	Physical characteristics of test sample	Constraints for preparation
TS-extr1	pH (EN ISO 10523)	250 ml	No special needs	no pH-adjustment allowed; measure as quickly as possible but not later than 6 h after sampling
TS-extr2	Cr, Cu, Mo, Zn (EN ISO 11885)	50 ml		acidify the sample with 0,5 ml of nitric acid per 100 ml sample (pH < 2)

The extracted solutions cannot (yet) be acidified at this point because they shall be used for pH determination also. The combination of flow sheet and Annex A is given in Table E.4.

Table E.4 — Application of flow sheet (Figure 1) and Annex A for preparation of Test Sample TS-extr1

	(Sub)-sample	What to do (flow sheet, Figure 1)	How to do it (Annex A)
1		Clearly identify the nature of the sample; contact customer and sampling team if needed; consult standards regarding requested analytical determinations. *Done: see above*	
2		Assess number and characteristics of test portion(s) to be prepared from this sample fraction, by consulting relevant analytical standard(s): *Test Sample, named TS-extr1 (250 ml), as described above*	
3		Assess the eventual need of separating fractions or phases, if applicable (e.g. nuts, bolts, stones, layers…): *Clean, aqueous solution: No separation of fractions or phases needed*	
4		Drying allowed and required? *Not applicable*	
5		Particle size reduction required and applicable? *Not applicable*	
6		Homogenization possible? *Yes*	
7	1 l of clean solution	**Perform homogenization**, see A.2	Procedure described in A.2.3.1 (manual homogenization for liquid samples) can be applied, just by shaking the container.
8		**Perform sub-sampling** (see A.6)	A.6.6 (sub-sampling for sludge and liquid) by just taking the requested amount of solution after homogenization.
9	Sub-sample of 250 ml	Does sub-sample comply with requirements of pH EN ISO 10523? Yes, sub-sample now becomes Test Sample TS-extr1, to be analysed for pH.	

A similar procedure may be followed for preparation of Test Sample TS-extr2, that can be subsequently acidified and analysed for Cr, Cu, Mo, Zn according EN ISO 11885.

E.2 Example 2

This example shows the application of this procedure for the preparation of test portions, each requiring different conditions for preparation.

a) Laboratory sample: 40 kg of solid waste, hard and possible to crush, grain size 10 mm to 40 mm, with high content of dry matter;

b) Required tests:

   1) dry matter – moisture content;

2) TOC;
3) loss on ignition;
4) cadmium, mercury, lead;
5) PCB;
6) compliance leaching test (EN 12457-2) and subsequent analysis of eluate for pH, conductivity, cadmium, mercury, lead, ammonium, chloride, cyanide easily liberatable, fluoride, nitrate, nitrite, phosphate, sulfate, sulfide, TOC.

How to proceed:

1) choice of methods according to requirements, the necessary detection limits, the nature of the sample and the laboratory equipment;
2) amount and required characteristics of test portions for each parameter (according Annex D) requirements for conservation (when needed);
3) definition of number and amount of test samples;
4) define the sequence of operations.

Choice of methods and definition of amount and characteristics of test portions:

According to the requirements of the analytical procedures to be applied, also summarized in Annex D, the properties of the test portions to be prepared are given in Table E.5.

Table E.5 — Definition of amount and characteristics of the test portions

Test to be applied	Method	Amount of test portion	Requirements of test portion
dry matter	EN 14346	drying at 105 °C: residue obtained at least 0,5 g; azeotropic distillation: 100 g	– store biologically active samples at least –18 °C
loss on ignition	EN 15169	0,5 g to 5 g	– particle size less than 200 µm
TOC	EN 13137	1 g	– store at 4 °C and analyse within 1 week or store at –18 °C or lower – particle size less than 200 µm – loss of volatiles need to be prevented (if possible, sample should be analysed without drying)
digestion for analysis of cadmium, mercury, lead	EN 13657	– mass above 200 mg – closed vessels: 200 mg to 500 mg – semi-open vessels: 200 mg to 1 000 mg – reflux systems: 1 g to 10 g	– particle size less than 250 µm – amount of organic carbon < 100 mg in the case of closed digestion vessel
compliance leaching test	EN 12457–2	90 g dry mass	– grain size of at least 95 % (mass) < 4 mm
PCB	EN 15308	20 g	– particle size preferably < 1 mm

Definition of amount and characteristics of the different test samples:

The minimum mass of the test samples needs to be sufficient for repeated analysis (it is recommended to use at least five times of the required amount of test portions (see Annex D) cited in the standards). During this step it is necessary to group the parameters in order to prepare test samples with similar requirements together and find the correct sequence of operations. The requirements of the test samples are given in Table E.6.

Table E.6 — Requirements of the test samples

Test sample ID	Test to be applied	Minimum amount of test sample according to analytical standards	Physical characteristics of test portion	Constraints for preparation
1	dry matter EN 14346	5 g or more, up to 100 g	no special needs	loss of water need to be prevented during test portion preparation
1	loss on ignition EN 15169	2,5 g to 25 g		
2	TOC EN 13137	5 g	< 200 µm	loss of volatile organic compounds need to be prevented
2	digestion for analyses of cadmium, mercury, lead EN 13657	1000 mg to 2500 mg	< 250 µm	no loss of volatile metal compounds no contamination with elements of interest
3	PCB	100 g	< 1 mm	without drying, if necessary, air drying
4	EN 12457–2	corresponding to 500 g of dry matter	95 % < 4 mm	drying is allowed (at 40 °C maximum) only if strictly necessary for eventual crushing

— **Test Sample 1: Dry matter and loss on ignition (can be determined from the same sample after dry matter determination):**

Amount: approximately 100 g

— **Test Sample 2: Digestion for analyses of cadmium, mercury, lead and TOC:**

If sample preparation without loss of volatile substances is possible, the sample preparation can be done together. Care shall be taken by choosing the material of crushing and milling equipment in order to avoid contamination. Milling to a grain size of < 200 µm is necessary for both samples.

Amount: approximately 10 g

— **Test Sample 3: PCB:**

For the preparation of organic parameters losses of moderately volatile compounds shall be avoided, so special care shall be taken by choosing the correct operations.

Amount: approximately 100 g

— **Test Sample 4: Compliance leaching test:**

For the preparation of eluates the procedures of sample preparation should be as minimal as possible according to the requirements of the respective standards. The amount of the test sample can be calculated after determination of dry matter in Test Sample 1.

Amount: approximately 500 g

There shall be 4 different test portions prepared with different requirements from the laboratory sample. It is necessary first to prepare the test portion(s) for ISO 11465 and analyse it for dry matter; afterwards, it will be possible to calculate the actual amount of test portion needed for EN 12457-2.

If necessary determination of dry matter of sub-samples can be considered for calculation purposes especially for hygroscopic material.

Define the sequence of operations:

The sequence of operations need to be defined according to the flow sheet (Figure 1), based on the properties of the laboratory sample and the requirements of the analytical procedures in combination with Annex A, see Table E.7.

Table E.7 — Application of flow sheet (Figure 1) and Annex A for preparation of the test samples

	(Sub)-sample	What to do (flow sheet, Figure 1)	How to do it (Annex A)
1		Clearly identify the nature of the sample; contact customer and sampling team if needed; consult standards regarding requested analytical determinations. Done: see above	
2	1	Define requirements: No loss of water, amount 5 g to 100 g	
3		Assess the eventual need of separating fractions or phases, if applicable (e.g. nuts, bolts, stones, layers): No separation of fractions needed	
4		Drying allowed and required? Not allowed	
5		Particle size reduction required and applicable? Not necessary as requirement of the test portion, but necessary because representative sub-sampling to a test portion of about 100 g is not possible with a grain size of 40 mm.	Due to the empirical rule that may be used for estimation of the minimum amount of a rather homogeneous sub-sample mass (kg) = 0,06 × maximum particle size (mm). The minimum amount of a test portion with a particle size of 40 mm is 2,4 kg. Depending on the heterogeneity of the sample this may even increase to about 20 kg or more by using the statistical formula. The asked amount of test sample is about 100 g.
6		Homogenization possible? Yes	Manual homogenization A.2.2
7		Sub-sampling of 2,4 kg of the laboratory sample Start again with flow chart	Sub-sampling (A.6) Requirement: No loss of water and moderately volatile organic compounds (PCB) Manual coning and quartering (A.6.2)
8	TS 1	Question 2 to 4 same answers, question 5: Yes, particle size reduction necessary for homogenization before further sub-sampling	Particle size reduction (A.5) to about 1 mm Requirement: No loss of water and moderately volatile organic compounds (PCB) A.4.2.1 crushing to about 1 mm (A.5.2.1) (gently enough to avoid losses of

	(Sub)-sample	**What to do (flow sheet, Figure 1)**	**How to do it (Annex A)**
			volatiles)
9		Homogenization possible? Yes	Homogenization (A.2): Requirement: No loss of water Manual homogenization
10	TS 1	**Perform sub-sampling for test portion(s)**	Sub-sampling (A.6): manual sub-sampling The minimum representative amount for this sub-sample is 60 g according to the empirical rule, calculated it would be 2,6 g for a rather homogeneous sample. The required amount of test sample is about 100 g. Take about 100 g of sub-sample for determination of water content and loss on ignition, either by coning and quartering or by mechanical apparatus provided that no losses of water or volatiles can occur.
11		Does sub-sample comply with requirements of EN 14346? Yes, sub-sample now becomes Test Sample TS 1, to be analysed for dry matter; preparation of single test portions from TS 1 will be done according to A.6.	Hint: it would be useful not to remix again the remaining sub-samples at this point, because they may be used for preparation of further test samples
12	Sub-sample < 1 mm	As for moderately volatile compounds it is recommended to take sub-samples too, without many steps of pretreatment and the requirements are fulfilled, sub-sampling for Test Sample 3 is the next step:	
13	TS 3	Define requirements for sub-sample 3: – loss of volatile or moderately volatile compounds shall be avoided – particle size < 1 mm – requirements and particle size fulfilled	
14	Sub-sample of 100 g, < 1 mm	Perform sub-sampling for test portion(s)	Sub-sampling (A.6): manual sub-sampling. The minimum representative amount for this sub-sample is 60 g according to the empirical rule it would be 34 g according to the statistical formula for a rather homogeneous sample. The required amount of Test Sample is about 100 g. Take about 100 g of sub-sample for determination of PCB (probably a suitable sub-sample is already available as a result of the step 9 above).
15	Sub-sample < 1 mm	Sub-sampling for Test Sample 2	As for trace element analysis the estimation of the necessary amount of sample is too rough, it is calculated[a] with the statistical model Minimum mass of Test Sample 34 g Take an aliquot of the sub-sample crushed to 1mm obtained from step 9, reduce it to about

	(Sub)-sample	What to do (flow sheet, Figure 1)	How to do it (Annex A)
			35 g to 50 g by coning and quartering or mechanical division for further treatment
16	TS 2	Define requirements for Test Sample 2: – Loss of moderately volatile organic compounds need to be prevented. – No loss of volatile metal compounds. – No contamination with elements of interest. – Particle size < 200 µm. – Not fulfilled. – Start with flow chart.	
17		Assess the eventual need of separating fractions or phases, if applicable (e.g. nuts, bolts, stones, layers): No separation of fractions needed	
18		Drying allowed and required? Not required, because milling possible without drying	
19		Particle size reduction required and applicable? Yes	Particle size reduction according to A.4 A.5.2.3 Milling (gently enough to avoid losses of volatile elements)
20		Homogenization possible? Yes	Homogenization (A.2):
21	Test portions for TOC, cadmium mercury lead	**Sub-sampling for test portions of test-sample 2**	Sub-sampling (A.6) of test sample into different test portions Requirement: Loss of moderately volatile compounds need to be prevented No contamination with elements of interest Manual or mechanical sub-sampling (appropriate material) possible

[a] Data to be used for the application of the statistical model for the determination of the minimum sample size of Test Samples 2 and 3:

D_{95} = 1 mm

ρ = 2,6 g/cm^3;

g = 0,25;

p the fraction of the particles with the property of interest; it is difficult to determine the actual value to be used for each case, so here only an estimated value will be used for digestion and subsequent analysis on trace elements a conservative smaller value like 0,001 need to be used;

CV a coefficient of variation of 0,1 (10 %) is commonly used.

Application of the statistical model of Annex B leads to these values:

— Minimum mass of Test Samples 2 and 3: = 34 g

After determination of dry matter is performed on Test Sample TS 1, the required amount of Test Sample 4 can be calculated and Test Sample 4 can be prepared according to Table E.8.

Table E.8 — Application of flow sheet (Figure 1) and Annex A for preparation of Test Sample TS4

	(Sub)-sample	What to do (flow sheet, Figure 1)	How to do it (Annex A)
21	TS 4	Define requirements: – Drying is allowed (at 40 °C maximum) only if strictly necessary for eventual crushing – Particle size < 4 mm – Not fulfilled – start with flow chart	The procedure starts again with the original laboratory sample or a representative sub-sample of 2,4, kg of the original laboratory sample with a grain size of 40 mm (obtained in step 7)
22		Assess the eventual need of separating fractions or phases, if applicable (e.g. nuts, bolts, stones, layers): No separation of fractions needed	
23		Drying allowed and required? Not allowed, only if strictly necessary for eventual crushing Not necessary	
24		Particle size reduction required and applicable? Yes	Particle size reduction according to A.5.2.1 To a particle size of < 4 mm, gently enough to avoid losses of volatile elements
25		Homogenization possible? Yes	Manual homogenization according to A.2.2
26	TS 4	**Perform sub-sampling for test portion(s)**	– Sub-sampling (A.6) – Requirement: Loss of volatile elements need to be prevented – No contamination with elements of interest – Manual or mechanical sub-sampling (appropriate material) possible

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