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Solid recovered fuels — Sample preparation

Combustibles solides de récupération — Préparation des échantillons

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html (standards.iteh.ai)

This document was prepared by Technical Committee ISO/TC 300, *Solid recovered materials, including solid recovered fuels*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 343, *Solid recovered fuels*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Solid recovered fuels are a major source of renewable energy. International Standards facilitate the production, trade and use of solid recovered fuels. For sampling and sample preparation of solid recovered fuels, ISO 21645 and this document, respectively, can be used (in conjunction) by different types of organizations, including but not limited to:

- solid recovered fuel production and trading companies;
- energy companies;
- regulatory bodies;
- conformity assessment bodies;
- laboratories.

The sample preparation technique adopted depends on a combination of different characteristics of the material and circumstances encountered at the sampling location. The determining factors are:

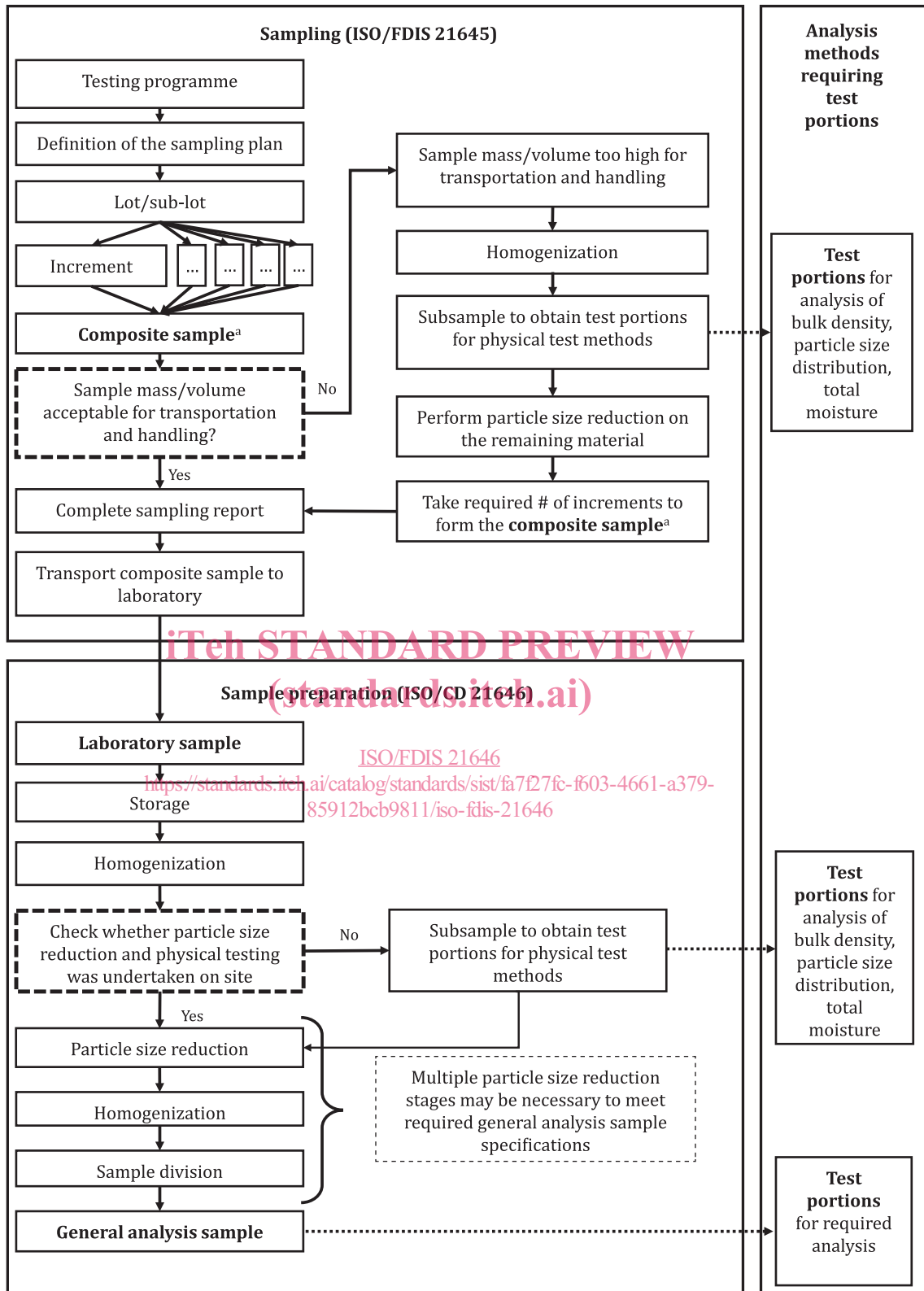
- the type of solid recovered fuel;
- the physical behaviour of the specific solid recovered fuel;
- the (expected) degree of heterogeneity (e.g. monostreams, mixed fuels, blended fuels).

In laboratory practice, different analytical procedures often need to be applied to the laboratory sample that has been taken according to the sampling plan. For this purpose, sub-sampling is applied in a way that the different test portions are representative of the laboratory sample with respect to the compounds of interest and the specific analytical procedures. The representativeness of the laboratory sample and of the test portions is of major importance to guarantee the quality and accuracy of analytical results. The representativeness of the laboratory sample is specified by the sampling plan.

This document is largely based on the work done by CEN/TC 343, *Solid recovered fuels*, and CEN/TC 292, *Characterization of waste* (now integrated in CEN/TC 444, *Environmental characterization of solid matrices*), and in particular EN 15002, which was developed for the majority of waste samples. Most of its concepts and specifications are also applicable to solid recovered fuel samples. However, the foundations of EN 15002 are not completely applicable to solid recovered fuel, as the nature of this material is substantially different and can lead to misrepresentation of the fuel quality.

The main characteristic that makes solid recovered fuel samples significantly different from other kinds of waste is that very often solid recovered fuels are solid, but neither 'granular' nor monolithic. It often happens that solid recovered fuel samples are fibrous-like materials, so that the statistical formula for sampling as defined in EN 15002 is not applicable. One additional term in the statistical formula is needed, namely the 'shape factor' (f).

This document is part of the testing programme for solid recovered fuels. This programme consists of various steps leading to the analysis sample for fuel quality testing as outlined in [Figure 1](#).



^a Step B.2.5 and B.2.6 in ISO 21645:2021, Annex B.

Figure 1 — Links between the essential elements of a testing programme

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Solid recovered fuels — Sample preparation

1 Scope

This document specifies methods for sample preparation to ensure representativeness of the samples throughout the preparation procedures to produce general analysis samples. Suitable test portions can be taken from the laboratory or general analysis samples and used for analysis according to the specific requirements defined in the corresponding analytical procedures.

This document specifies the correct sample preparation sequence to be applied to:

- a) the composite sample, in order to produce a laboratory sample (taking into account large pieces of solid recovered fuel);
- b) each sub-sampling step throughout the testing programme;
- c) the laboratory sample, in order to obtain suitable test portions;
- d) ensure the representativeness of the test portions that have been taken according to the sample preparation plan, prior to physical analysis, chemical analysis or both (e.g. extractions, digestion, analytical determinations).

The methods described in this document can be used for sample preparation, for example, when the samples are to be tested for bulk density, biomass content determination, mechanical durability, particle size distribution, moisture content, ash content, ash melting behaviour, calorific value, chemical composition, impurities and self-heating properties. The methods are not intended to be applied to the very large samples required for the testing of bridging properties.

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2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

ISO 21637:2020, *Solid recovered fuels — Vocabulary*

ISO 21660-3, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

CEN/TS 15414-1, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 1: Determination of total moisture by a reference method*

CEN/TS 15414-2, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 2: Determination of total moisture by a simplified method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 21637 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

composite sample

sample (3.12) consisting of all the *increments* (3.6) taken from a *lot* (3.8) or a sub-lot

Note 1 to entry: The increments can be reduced by division before being added to the composite sample.

Note 2 to entry: The minimum sample mass shall be retained during the collection of increments to form the composite sample.

3.2

drying

process of removing water from a *sample* (3.12)

Note 1 to entry: For the purpose of *test portion* (3.16) preparation, it can be useful to remove just the amount of water that can possibly 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.

[SOURCE: ISO 21637:2020, 3.19, modified — “solid fuel” was replaced with “sample” in Note 1 to entry.]

3.3

fraction separation

process of dividing components, particles or layers if *homogenization* (3.5) of the *sample* (3.12) is practically not applicable and/or the analyses of different fractions or phases are appropriate

[SOURCE: ISO 21637:2020, 3.32]

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3.4

general analysis sample

sub-sample (3.14) of a *laboratory sample* (3.7) having a *nominal top size* (3.10) of 1 mm or less and used for a number of chemical and physical analyses

3.5

homogenization

process of combining of *increments* (3.16) making up a combined sample, components, particles or layers into a more homogeneous state than in the samples (in the case of composite samples) or pre-treated fractions of samples in order to ensure equal distribution of substances in and properties of the *sample* (3.12)

3.6

increment

portion of solid recovered fuel extracted from a *lot* (3.8) or sub-lot in a single operation of the sampling device

[SOURCE: ISO 21637:2020, 3.39]

3.7

laboratory sample

composite sample (3.1) received by the laboratory on which *sample* (3.12) preparation procedures are undertaken

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, mixing, grinding or by combinations of these operations, leading to a *nominal top size* (3.10) ≤ 1 mm, the result is the *general analysis sample* (3.4). A *test portion* (3.16) is removed from the general analysis sample for the performance of the test or for analysis. When no preparation of the laboratory sample is required, the laboratory sample may become the test portion.

Note 2 to entry: The composite sample becomes the laboratory sample when it is delivered into the laboratory for commencement of the sample preparation procedures.

3.8

lot

defined quantity of fuel for which the quality is to be determined

[SOURCE: ISO 21637:2020, 3.40]

3.9

minimum sample mass

minimum amount or dimension of the *sample* (3.12) required during sampling and sample preparation from the point of view of preserving its representativeness

Note 1 to entry: The minimum sample mass is at least equal to the increment mass multiplied by the number of *increments* (3.6) and is linked directly to the *nominal top size* (3.10).

[SOURCE: ISO 21645:2021, 3.14]

3.10

nominal top size

smallest aperture size of the sieve used for determining the particle size distribution of solid recovered fuels through which at least 95 % by mass of the total material passes through the sieve

[SOURCE: ISO 21637:2020, 3.48]

3.11

particle size reduction

reduction of the *nominal top size* (3.10) of a *sample* (3.12) or *sub-sample* (3.14)

3.12

sample

quantity of material, representative of a larger quantity for which the quality is to be determined

3.13

sample division

reduction of the mass of a *sample* (3.12) or *sub-sample* (3.14)

[SOURCE: ISO 21637:2020, 3.64, modified — “by mass” was deleted from the term.]

3.14

sub-sample

portion of a *sample* (3.12)

Note 1 to entry: A sub-sample is obtained by procedures in which the particles are randomly distributed in parts of equal or unequal size.

Note 2 to entry: A sub-sample may be either a portion of the sample obtained by selection, or division of the sample itself, or the final sample of a multistage sample preparation procedure.

[SOURCE: ISO 21637:2020, 3.82, modified — “items of interest” was replaced by “particles” in Note 1 to entry and Note 2 to entry was added.]

3.15

sub-sampling

process of selecting one or more *sub-samples* (3.14) from a *sample* (3.12)

3.16

test portion

sub-sample (3.14) of a *laboratory sample* (3.7) or *general analysis sample* (3.4) consisting of the quantity of material required for a single execution of a test method

Note 1 to entry: The test portion may be taken from the laboratory sample directly if no preparation of *sample* (3.12) is required (e.g. for bulk density determination or particle size distribution).

3.17

total moisture sample

sample (3.12) taken specifically for the purpose of determining the total moisture content

4 Symbols

For the purposes of this document, the following symbols apply.

α	constant in third power law, in g/mm^3
d_{05}	nominal minimum particle size (a mass fraction of 5 % of the particles are smaller than d_{05}), in mm
d_{95}	nominal top size of a particle (a mass fraction of 95 % of the particles are smaller than d_{95}), in mm
f	shape factor, in m^3/m^3
M	moisture, in per cent by mass
m	mass of a sample, in g

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5 Safety remarks

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Safety issues relating to the handling of potentially hazardous materials are dealt with in relevant national and international regulations, to which every laboratory should refer.

In addition, the following applies:

- The apparatus for grinding, cutting, milling and homogenization shall be operated by skilled persons strictly according to the manufacturer's instructions.
- All operations shall be performed in a hood or in closed force-ventilated equipment, due to the possibility of generation of fine particles.

6 Principles of correct sample preparation

The main purpose of sample preparation is to reduce the mass and/or the particle size of a sample to obtain one or more test samples that are in general smaller than the laboratory sample. The principle of correct sample preparation is that the composition of the composite sample collected does not change during each step of the sample preparation procedures. When correct sample preparation is performed, any sub-sample or test portion is representative of the laboratory sample and every particle in that sample has then an equal probability of being included in any sub-sample retained. Also, the loss of moisture and other volatile components is minimized following the procedures described in this document. Equally, any contamination of the sample during the sample preparation processes is addressed and measures are taken to avoid contamination.

Three basic methods are used during the sample preparation:

- homogenization;
- sample division;

— particle size reduction of the sample.

For granular materials generally, the principle of the third-power law is accepted and respected at each sample division step. [Formula \(1\)](#) shows this third-power law:

$$m > \alpha \times d_{95}^3 \quad (1)$$

where

m is the mass retained after each sample division step in g;

d_{95} is the nominal top size in mm;

α is a constant over the whole sample preparation procedure for a particular material, in g/mm³.

The value and unit of constant α is fixed by the particle nominal top size, d_{95} , and the sample mass, m , of the sample collected following the sampling plan and before sample preparation.

The minimum amount of sample for each step of sample preparation and sub-sampling can be directly estimated by [Formula \(E.1\)](#) in [Annex E](#), including [Table E.2](#).

EXAMPLE A sample of 10 kg of solid recovered fuel has d_{95} of 50 mm. For the analysis, a test portion of 5 g is required. The third-power law results in $\alpha = 10\,000 \text{ g}/(50 \text{ mm})^3 = 0,08 \text{ g/mm}^3$. Using this value in [Formula \(1\)](#) for a reduced sample mass results in a nominal top size for the particles in the test portion of 3,97 mm [i.e. $\sqrt[3]{(5,0 \text{ g} / 0,08 \text{ g/mm}^3)}$]. The resultant figures are shown in the following table.

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m g	α g/mm ³	d_{95} mm
10 000	0,08	50
5	0,08	3,97

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[Table 1](#) shows the resulting reduction factors for the minimum (sub-)sample mass, if a certain reduction of the nominal top size is chosen and the third-power law is respected. The reduction factor of the nominal top size can be calculated by dividing the current nominal top size by the proposed nominal top size after mass reduction.

[Table 2](#) shows the desired reduction factors for the minimum nominal top size, if a certain reduction of the (sub-)sample mass is chosen and the third-power law is respected. The reduction factor of the minimum (sub-)sample mass can be calculated by dividing the current minimum (sub-)sample mass by the proposed minimum (sub-)sample top size after mass reduction.

[Formula \(1\)](#) can be used to calculate the exact values for each specific situation.

Table 1 — Common values for desired reduction factor minimum (sub-)sample mass

Chosen reduction factor of the nominal top size	Resulting reduction factor for the minimum (sub-)sample mass
1,5	3,4
2	8
3	27
4	64
5	125
6	216
7	343
8	512
9	729

Table 1 (continued)

Chosen reduction factor of the nominal top size	Resulting reduction factor for the minimum (sub-)sample mass
10	1 000
20	8 000
30	27 000

Table 2 — Common values for desired reduction factor nominal top size

Desired reduction factor for the minimum (sub-)sample mass	Necessary reduction factor of the nominal top size
2	1,3
3	1,4
4	1,6
5	1,7
10	2,2
20	2,7
50	3,7
80	4,3
100	4,6
200	5,8
500	7,9
1 000	10,0

For solid recovered fuels, many materials turn out to be far from granular (e.g. in fluff the particles turn out to be predominantly flat). Therefore, for solid recovered fuels, a correction can be made for non-granular materials.

If a sub-sample is required for the determination of total moisture content, the sub-sample is prepared by a procedure that does not conflict with the requirements of CEN/TS 15414-1 and CEN/TS 15414-2. If the total moisture content of the material (as sampled) is to be determined, it is recommended that a separate total moisture sample is taken as there is a risk of reducing the moisture content by sample preparation procedures.

If mercury content of the material (as sampled) is to be determined, it is recommended that a separate mercury analysis sample is taken, as there is a risk of reducing the mercury content by sample preparation operations if it is assumed that the material contains elemental mercury.

For materials that are examined for moisture and mercury content, significant heat build-up and risk of loss of moisture and mercury can occur.

If samples are to be tested for substances that volatilize (e.g. mercury), care shall be taken during the sample preparation procedures, in particular the stages of particle size reduction, to minimize any increase in temperature.

7 Quality control and sources of error

Quality control during sample preparation procedures shall be demonstrated by carrying out at least the following everyday laboratory practices:

- a) all balances used for weighing sample masses shall be calibrated by an external calibration body on a regular basis;

NOTE 1 This can be undertaken in accordance with the quality control procedures of the laboratory at 6-month or 12-month intervals, for example.

- b) a weight check on each balance shall be undertaken at a regular basis, preferably at the commencement of each working day and by using calibrated weights that fall within the typical weighing range of the balances in use, and the readings of this weight check shall be recorded;
- c) the operating temperature of drying oven(s) shall be checked, preferably at the commencement of each working day and in addition at a 6-month or 12-month frequency by using a calibrated temperature recorder, to ensure that samples are dried at the correct temperature (within the acceptable range permitted by the test method), and the reading(s) of the operating temperature shall be recorded;
- d) the milled product (i.e. the general analysis sample from which the test portion is taken) shall be checked for oversize (i.e. > 1 mm) on a regularly basis, preferably on a weekly basis, or following any mechanical changes (e.g. new screens, hammers) to ensure that the particle size is within specification, and the results are recorded;

NOTE 2 This check also confirms that there is no problem with the operation of the milling equipment.

- e) the sieves used in the laboratory shall be manufactured according to the specifications in ISO 3310-1 and ISO 3310-2 and their apertures shall be checked as described according to the manufacturer's instructions at an agreed periodicity;
- f) all balances, ovens, milling and sieving equipment shall have unique identification and shall exhibit the date of any calibrations undertaken as well as the 'next due' date.

The sub-sample shall be re-homogenized after any operation that can result in segregation of different sized particles.

Loss of material (e.g. fine particles) and volatile components, such as moisture and mercury during milling, and contamination of the sample via the air, by dust or by the use of the apparatus (e.g. from the ambient laboratory atmosphere or between samples stored or processed close to one another) shall be avoided.

The following types of contamination can occur from the sample preparation apparatus:

- abrasion;
- cross-contamination;
- chemical release;
- chemical reaction due to generated heat (which can be a source of error and can cause material alteration).

Treatment of samples should be performed in a separate room that is used only for this purpose, especially for crushing or sieving.

If the sample has a dust-like consistency or contains (semi-)volatile compounds, losses shall be minimized as these can alter its physical-chemical properties.

Tests shall be carried out to detect possible contamination from the apparatus used during sample preparation.

8 Apparatus

8.1 Selection of equipment

For the purpose of preparation of the general analysis sample, test portions from the laboratory sample, or both, appropriate equipment shall be chosen depending on the procedures selected according to [Annex D](#). In the selection of the type of treatment techniques, it should be taken into account that each of them has potential impact on analytical results, due to introducing contamination or altering the physical-chemical properties of the sample.