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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).

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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.

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 materials, including 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:

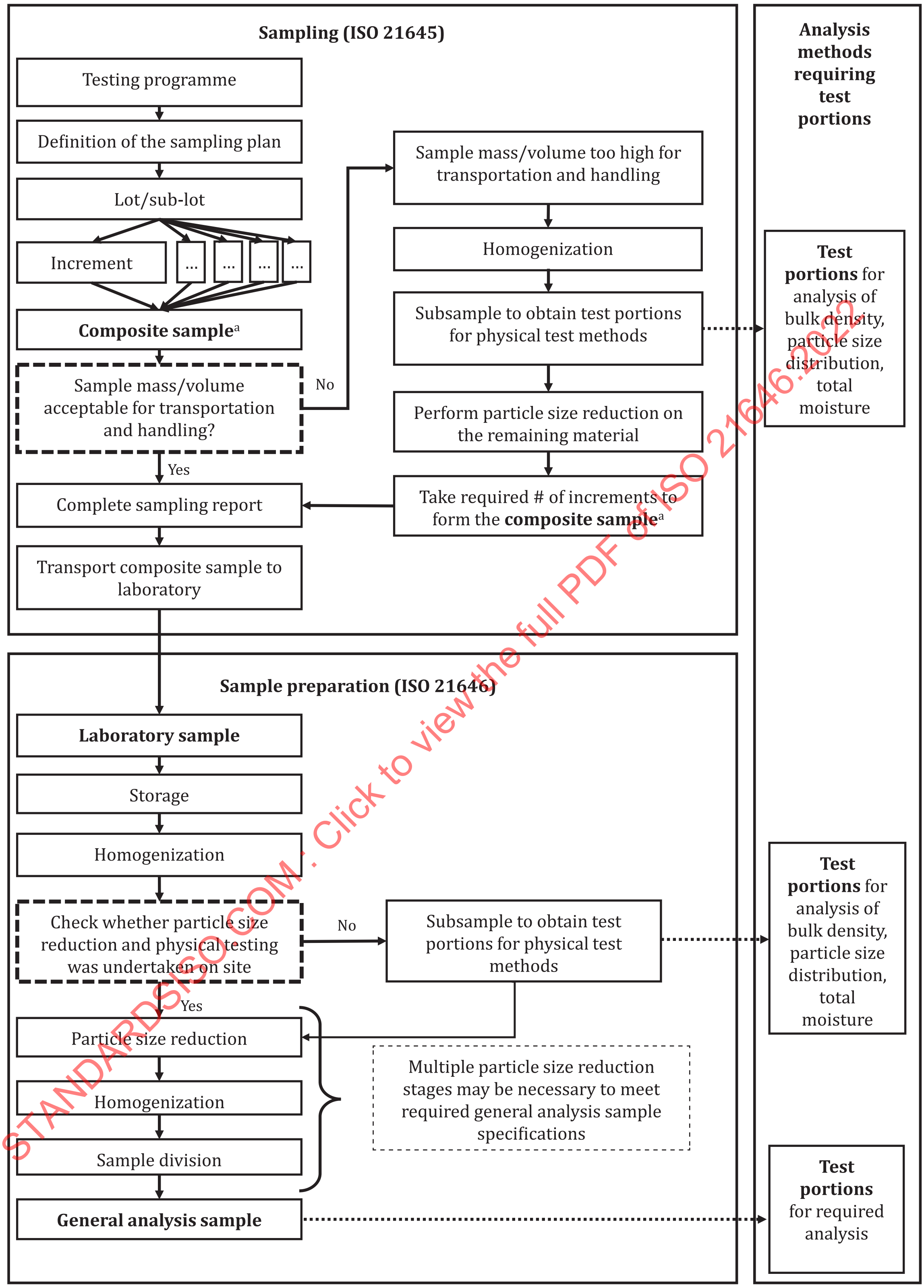
- the type of solid recovered fuel;
- the physical behaviour of the specific solid recovered fuel;
- the (expected) degree of heterogeneity (e.g. monostreams, mixed fuels, blended fuels).

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] ISO 21645:2021, B.2, steps 5) and 6).

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

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 specified 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.

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]

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 mm^3/mm^3
M	moisture, in per cent by mass
m	mass of a sample, in g

5 Safety remarks

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:

a) The apparatus for grinding, cutting, milling and homogenization shall be operated by skilled persons strictly according to the manufacturer's instructions.

b) 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:

$$m > \alpha \times d_{95}^{3} \tag{1}$$

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/mm^3.

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 α = 10 000 g/(50 mm)^3 = 0,08 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.

m g	***α*** g/mm^3	***d*$_{95}$** mm
10 000	0,08	50
5	0,08	3,97

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.

All materials that come into contact with the sample shall be:

a) made from suitable materials;

b) chemically compatible with the sample;

c) selected in order to minimize contamination.

Good cleaning shall be ensured to avoid cross-contamination of samples.

The equipment to be used for the sample preparation procedures shall be in accordance with Annex F.

8.2 Apparatus for sample division

8.2.1 Scoops and shovels (sampling tools)

8.2.1.1 Scoops

A scoop being used for manual sampling shall have:

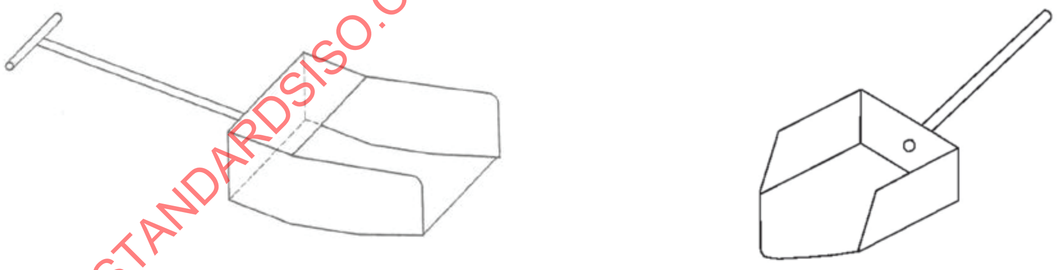
a) dimensions and an opening at least three times greater than the nominal top size of the material to be processed;

b) a flat bottom with edges raised high enough to prevent losing particles.

Figure 2 shows examples of scoops.

EXAMPLE 1 For a granular solid recovered fuel with a d_{95} of 20 mm, the minimum dimensions (l × b × h) of the sampling scoop are 60 mm × 60 mm × 60 mm. A sampling scoop like the one illustrated in Figure 2a can be used for this type of solid recovered fuel.

EXAMPLE 2 For a solid recovered fuel, which consists almost entirely of flat parts, the dimensions of the sampling scoop are at least equal to three times $d_{95,l}$, where $d_{95,l}$ is the maximum length of a fluff particle (a mass fraction of 95 % of the particles are smaller than $d_{95,l}$). Therefore, for fluff with a $d_{95,l}$ of 200 mm, the dimensions of the sampling scoop (l × b × h) are at least 600 mm × 600 mm × 600 mm. For sampling fluff-type solid recovered fuels, it is advisable to use a sampling scoop that has a sharp point underneath and upright walls, as illustrated in Figure 2b.



a) Example of scoop for granular solid recovered fuel[a]

b) Example of scoop for solid recovered fuel almost entirely consisting of flat parts[b]

[a] See Example 1.

[b] See Example 2.

Figure 2 — Example of scoops

8.2.1.2 Shovels

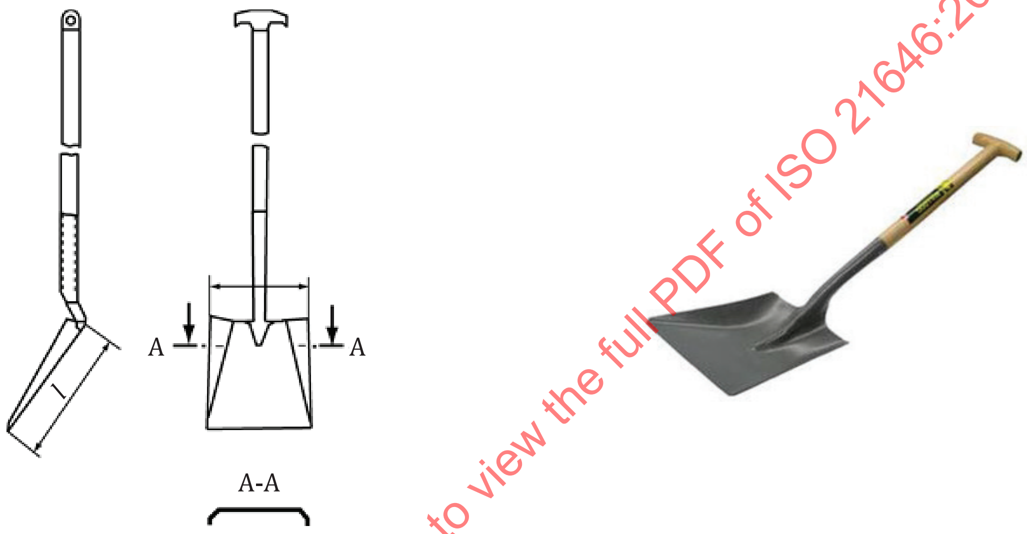
A shovel being used for manual sampling shall have:

a) dimensions and a blade at least three times greater than the nominal top size of the material to be processed;

b) a flat blade with edges raised high enough to prevent losing particles.

Figure 3 shows an example of a shovel.

NOTE As the high sides of a scoop can prevent it being inserted into the material, resulting in larger particles being pushed away rather than entering the scoop and so becoming part of the increment selected, the use of a shovel is preferred.



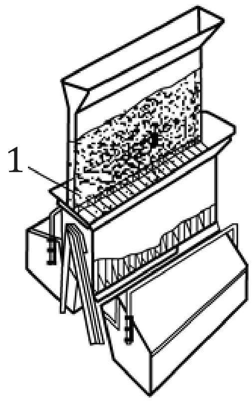
Key

l	length of shovel
A-A	sectional view

Figure 3 — Example of a shovel

8.2.2 Riffle boxes

A riffle box shall have at least 16 slots and an even number of slots, with adjacent slots directing material into different sub-samples. The width of the slots shall be at least three times the nominal top size of the material to be riffled. Figure 4 shows an example of a riffle box.



Key

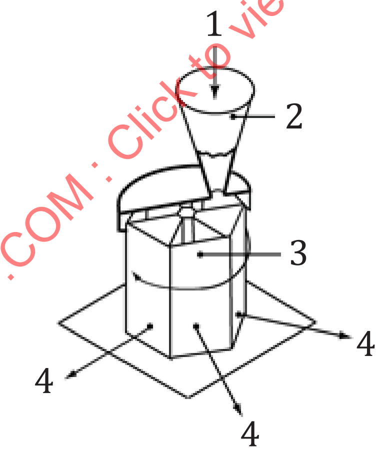
1 slot, width is at least three times the nominal top size of the material

Figure 4 — Example of a riffle box

8.2.3 Rotary sample dividers

A rotary sample divider shall have a feeder device adjusted so that the divider rotates at least 20 times (or cuts the falling sample stream at least 20 times) while the sample is being divided (e.g. 120 cuts during the time the sample is being fed to the divider). Figure 5 shows an example of a rotating divider.

The manufacturer's instruction manual shall be followed. The inner dimensions of the equipment shall be at least three times as wide as the nominal top size of the material to be processed.



Key

1 feeder

2 funnel

3 rotating receiver

4 divided sample

Figure 5 — Example of a rotary sample divider

8.3 Apparatus for particle size reduction

8.3.1 Shredder

A shredder is an apparatus with a rotor equipped with hammers that shred the material which is fed to the shredder. Shredders are used for reducing the particle size down to 30 mm. In the case of hard and strong materials, it can be necessary to perform the particle size reduction in more than one step. The use of shredders for particle size reduction causes a risk of losing moisture and fine fractions. The use of shredders shall be avoided when possible for this reason.

NOTE Many types of solid recovered fuel contain plastics and metals, making the use of a shredder necessary.

8.3.2 Coarse cutting mill

Coarse cutting mills are used for cutting materials into lengths of about 10 mm to 30 mm (depending on the solid recovered fuel and the analyses to be performed). The equipment shall:

a) have a minimum of drying effect either by heating the materials or blowing air through them;

b) be designed so that it does not lose dust or contaminate the material with pieces of metal;

c) be easy to clean.

8.3.3 Cutting mill

Cutting mills are used for particle size reduction of materials used as solid recovered fuels from about 10 mm to 30 mm down to about 1 mm or less (depending on the solid recovered fuel and the analyses to be performed). The mill shall be provided with screens of various aperture sizes covering this range, including an appropriate sieve to control the nominal top size of the material produced. Other apparatus may be used provided that it is designed so that it does not get blocked with the material that is being processed. The use of cutting mills, whose cutting faces contain significant quantities of an element that is to be determined in the analysis, shall be avoided. A cutting mill with no screens can be suitable for small quantities.

NOTE 1 Cross-beater mills can be used without any excessive dusting, when fitted with dust filters (like a filter sock) between the mill and the receiving container. They are suitable for final grinding of hard, wood-type materials after the pre-grinding with cutting-type mills.

NOTE 2 Annex D provides additional guidelines.

8.4 Sieves

A wire-mesh sieve with an aperture size of 1,00 mm shall be used to check the nominal top size of general analysis samples. If analysis sub-samples with a nominal top size of 0,250 mm are required, a sieve with apertures relating to that particle size shall be used.

8.5 Balance

A balance shall be used that is capable of determining the mass of samples to an accuracy of 0,1 % of the sample mass, and the mass of sub-samples to an accuracy of 0,1 % of the sub-sample mass.

9 Sample preparation procedure

9.1 General

This clause describes the general procedure that shall be followed to undertake sample preparation, based on the following steps:

1) collecting the relevant information of the material for sample preparation (see 9.2);

2) making a sample preparation plan (see 9.3);

3) performing the sample preparation plan (see 9.4).

9.2 Step 1: Collecting the relevant information of the material for sample preparation

The following information relating to the material for sample preparation is available:

a) the minimum sample mass;

b) the actual mass of the sample, m_0;

c) the nominal top size of the sample;

d) the shape factor of the sample;

e) the requirements in terms of particle size reduction for the analysis to be performed;

f) the required amounts for each of the size fractions and their restrictions to the sample preparation methods.

NOTE Information regarding the items a) to d) is available from the sampling plan.

Sample preparation provides a sample for a number of tests to be performed on the sample. Some of these tests require no particle size reduction or drying of the sample. Other tests require very tiny homogenized sub-samples with small particle sizes. A sample preparation plan shall meet all these requirements.

9.3 Step 2: Making a sample preparation plan

9.3.1 General

Step 2 specifies the making of a sample preparation plan. The actual making of the sample preparation plan is the most crucial phase during sample preparation. Sample preparation is a combination of sample division (see 9.3.2) and particle size reduction (see 9.3.3). To what level a sample of solid recovered fuel is to be prepared onsite depends on available equipment onsite, the requirements of the laboratory and the preferences of the client of the sampling activities.

9.3.2 Sample division

The aim of sample division of a sub-sample is to reduce the sub-sample mass remaining or to make several duplicate sub-samples out of one laboratory sample. During the performance of sample division, the minimum sub-sample mass shall be retained to ensure the representativeness of the sub-sample of the laboratory sample (see 9.3.4). Clause 10 describes the available methods for sample division.

9.3.3 Particle size reduction of a sample

The aim of particle size reduction is to reduce the nominal top size of the particles to reduce the minimum sub-sample mass without losing representativeness. During the performance of particle size reduction, all materials should be included. Leaving out ash contributors and hard metals (to prevent damage to equipment) in particular can cause significant errors in the measured values of these and possibly accompanying metals.

The sample preparation plan shall contain at least the information shown in Table 3. It specifies all activities that shall be performed during the whole process of sample preparation. The actual structure of Table 3 shall be adjusted to the properties of the investigated solid recovered fuel and the equipment selected in the laboratory (e.g. steps can be skipped or added if the nominal top size is already smaller than 30 mm or the coarse shredder results in a nominal top size different from 30 mm). Some information needed to complete Table 3, such as techniques, shape factor, nominal top size and particle size reduction steps, shall be chosen or determined.

Annex A describes the general determination of the shape factor that shall be undertaken at commencement or the initial sampling stage. The determination of how the shape factor can change through the processes of sample preparation shall be in accordance with Annex B. The determination of mass and particle size reduction techniques and apparatus are specified in Clause 8. Clause 11 describes the methods of sample division before particle size reduction.

A sample preparation plan can be used multiple times for a specific product as long as the physical composition of the solid recovered fuel does not change.

Table 3 can be used as a sample preparation plan. Annex C shows an example of how a sample preparation plan is made.

Annex D provides guidelines for choosing the most suitable sample preparation procedures.

Table 3 — Sample preparation plan

Step	Description	Method of reduction	Used technique and apparatus	Mass before reduction	Mass after reduction	Nominal top size before reduction	Nominal top size after reduction	Shape factor before reduction	Shape factor after reduction	Mass to be withheld for analysis	Purpose of product of this reduction step
Step 1	Sample division of the composite sample into a sub-sample for further sample preparation and a sub-sample of untreated material	Sample division									Determination of bulk density, durability of pellets, particle size distribution
Step 2	Particle size reduction in order to make further sample division possible	Particle size reduction to < 30 mm					30 mm				
Step 3	Sample division in order to reduce the remaining mass or obtain sub-samples as general analysis samples	Sample division				30 mm	30 mm				Sub-samples for moisture content
Step 4	Particle size reduction of the remaining sub-sample in order to produce a general analysis sample	Particle size reduction to < 1,0 mm				30 mm	1,0 mm		1,0		
Step 5	Sample division of the remaining sample material into the required general analysis sample(s)	Sample division				1,0 mm	1,0 mm	1,0	1,0		Sub-samples for determination of ash, calorific value, chemical analysis
Step 6	Particle size reduction in order to make further sample division possible	Particle size reduction to < 0,25 mm				1,0 mm	0,25 mm	1,0	1,0		
Step 7	Sample division of the remaining sample material into the required test portions	Sample division				0,25 mm	0,25 mm	1,0	1,0		Sub-samples for analysis where < 0,25 mm is required

NOTE Mass can only be withheld during a sample division step and not during a size reduction step.

9.3.4 Retaining the minimum (sub-)sample mass

During every sample division step, every particle in the sample before sample division shall have an equal probability of being included in the sub-sample retained after sample division. For non-granular materials with a shape factor significantly smaller than 1,0, a correction shall be made for the changing shape factor in accordance with Annex A.

Each step of a sample preparation procedure shall conform to Formula (2):

$$\frac{m_1}{m_2} \geq \frac{f_1}{f_2} \times \left(\frac{d_1}{d_2}\right)^3 \tag{2}$$

where

m_1 is the sample mass of sample before particle size reduction;

m_2 is the sample mass of sample after particle size reduction;

f_1 is the shape factor of sample before particle size reduction;

f_2 is the shape factor of sample after particle size reduction;

d_1 is the nominal top size of sample before particle size reduction;

d_2 is the nominal top size of sample after particle size reduction.

For materials that are more or less granular, the shape factors f_1 and f_2 are close to 1. In this case, it may be assumed that these two shape factors are 1, as this is easier and cheaper. For materials that are far from granular, the shape factor f_1 shall be determined in accordance with Annex A. The shape factor changes during the particle size reduction. Therefore, the shape factor approaches 1,00 during particle size reduction. An estimation for the increase of the shape factor after particle size reduction shall be specified in accordance with Annex B. For each particle size reduction step a new shape factor and nominal top size shall be determined to establish whether the proposed sample preparation step conforms to Formula (2). The shape factor may be assumed to be 1,00. This requires additional particle size reduction but simplifies the procedure and does not influence the representativeness.

The effect on sample mass requirements during sample subdivision as a result of the change in shape factor created by the process of particle size reduction during sample preparation is illustrated in the following two cases:

1) Laboratory sample

The laboratory sample of a fluff-type solid recovered fuel has a particle density of 1 000 kg/m^3 and a bulk density of 150 kg/m^3. A mass fraction of approximately 95 % of the fluff has a nominal top size of 190 mm. A value of 0,1 can be used for the coefficient of variation C_V and the factor p. In this example the particle size distribution of the fluff is assumed to be quite large, so a value of 0,25 can be assumed for g. As the fluff has an extremely variable particle shape, a value of 0,05 can be used for the shape factor. This leads to a minimum sample mass with a weight of $\pi/(6 \times 10^9) \times 190^3 \times 0{,}05 \times 1\,000 \times 0{,}25 \times (1-0{,}1)/(0{,}1^2 \times 0{,}1) = 40{,}40$ kg, and a volume of $40{,}40/150 = 0{,}269\,3$ m^3 = 269 l.

2) Laboratory sample following particle size reduction during sample preparation

During the sample preparation procedure the laboratory sample of the fluff-type solid recovered fuel detailed in case 1) was shredded to reduce the particle size. This will allow subdivision of the sample by taking smaller sub-samples due to the reduced particle size. For the calculation at this stage of the sample preparation process, consider the sample to have a particle density of 1 000 kg/m^3 and a bulk density of 200 kg/m^3. A mass fraction of approximately 95 % of the fluff has a nominal top size of 30 mm. A value of 0,1 can be used for the coefficient of variation C_V and the factor p. At this stage of sample preparation, the particle size distribution of the fluff has been reduced and is more homogeneous, therefore a value of 1,0 can be assumed for g. As the particle shape is now more uniform, a value of 1,0 can be used for the shape factor. This leads to a minimum sample mass with a weight of $\pi/(6 \times 10^9) \times 30^3 \times 1{,}0 \times 1\,000 \times 1{,}0 \times (1-0{,}1)/(0{,}1^2 \times 0{,}1) = 12{,}72$ kg, and a volume of $12{,}72/200 = 0{,}063\,6$ m^3 = 64 l.

9.4 Step 3: Performing the sample preparation plan

In the third step, sample preparation shall be performed as specified in the sample preparation plan.

During the actual sample preparation, the following procedures shall be performed:

a) Homogenize the sub-sample material at every step thoroughly.

b) Ensure that no material of the sub-sample gets lost.

c) If possible, do not leave out any components.

When it is necessary to remove hard substances like metal (steel) to avoid damage to the mill, this shall be reported. The report shall include the mass percentage and type of substance which were removed from the sample. The material removed, weighed and reported contributes to the total ash content of the solid recovered fuel. In the determination of ash content, the removed ash contributors shall be included in the analysis report.

d) Produce enough milled sample to perform all the required analyses.

10 Methods for homogenization and sample division

10.1 General

This clause describes the methods that shall be used to produce a smaller but representative sample. It is essential, to achieve representative subdivision for each method described in this clause, that the sample is thoroughly mixed prior to the commencement of the subdivision process. Annex D provides additional guidelines for these methods.

10.2 Homogenization

The sample shall be formed into a heap on a dry, contamination-free surface. The sample shall be moved using a shovel to form into a new pile by placing shovelfuls on top of the preceding one such that it is evenly distributed and different particle sizes become well mixed. This process shall be repeated three times, forming a new pile each time. On completion of this process, the sample can then be subdivided using one of the methods listed in 10.3.

10.3 Sample division methods

10.3.1 General

The remaining sample mass shall not be less than the minimum sample mass. In the case of a sampling scoop, the sampling tools and sample division equipment used shall have minimum length, depth and height dimensions of three times d_{95}. In the case of a shovel that has open, flat surfaces and slightly raised edges to assist in retaining increments when inserted into the material pile or flow, three times d_{95} shall relate to the breadth of the shovel blade.

The sample division methods are described in 10.3.2 to 10.3.8.

10.3.2 Riffling

Riffling may be used for materials that can be passed through the riffle without bridging. It is not suitable for fluff or other materials containing elongated particles, or for wet materials. Brittle materials should be handled with care to avoid fine materials being produced. When using riffling, the following procedure shall be followed:

a) put the whole of the thoroughly homogenized, composite sample into one of the containers of the riffle so that it is evenly distributed throughout the container;

b) place the other two containers in position under the riffle;

c) pour the contents of the first container down the centre line of the riffle;

d) pour the solid recovered fuel sufficiently slowly that bridging does not occur;

e) do not move the container from side to side (this would cause the end slots to receive less solid recovered fuel);

f) discard the solid recovered fuel that falls into one of the containers;

g) repeat the riffling process [steps a) to f)] until a sub-sample of the required mass is obtained.

Figure 4 shows an example of a riffle box.

10.3.3 Strip division

Strip division may be used for all materials. This is a convenient method when a composite sample is to be divided into a small number of laboratory samples. When using strip division, the following procedure shall be followed:

a) place the whole composite sample on a clean, hard surface and homogenize it by mixing with a shovel;

b) position a vertical plate at each end of where the strip will be formed;

c) use a shovel to distribute the material along the length of the strip as evenly as possible, working from end to end and from both sides, in which the length-to-width ratio of the strip shall not be less than 10:1;

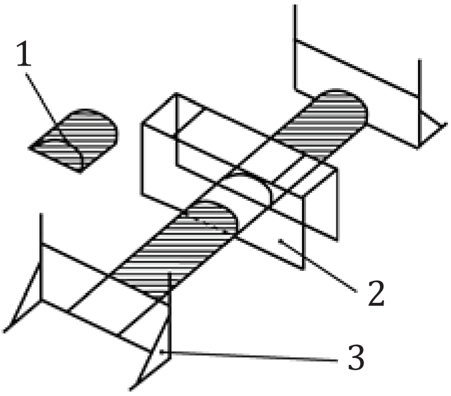
   NOTE The more passes from end to end, while building the strip, improves the quality of the division.

d) obtain a laboratory sample by taking at least 20 increments from locations evenly spaced down the length of the strip.

e) take each increment by inserting two plates vertically into the strip and removing all the material from between the plates, taking into account that:

   - the two plates should be inserted the same distance apart each time so that each increment contains the same quantity of material;
   - the distance between the plates should be chosen so that the method yields a laboratory sample of the required mass.

Figure 6 illustrates the principle of strip division.



Key

1 increment

2 sampling frame (>3 d_{95})

3 end plates

Figure 6 — Strip division

10.3.4 Long strip

Long strip division may be used for all materials. This is a convenient method when a composite sample is to be divided into several laboratory samples. In this method, the whole of the composite sample is divided into a number of laboratory samples. When using long strip, the following procedure shall be followed:

a) start by forming a strip in accordance with the strip division method (see 10.3.3);

b) choose the number of laboratory samples to be produced so that they will have the required mass;

c) arrange this number of containers conveniently near the strip;

d) use a sampling tool or shovel of a size such that each laboratory sample will receive at least 20 increments, taking into account that if the laboratory samples are each to be of mass $m_{\text{laboratory sample}}$ kg, the sampling tool should have a capacity of no more than $m_{\text{laboratory sample}}/20$ kg;

e) always take increments from the same end of the strip without leaving fine particles behind and place each increment in each container in turn until all the material has been used.

10.3.5 Manual increment division

Manual increment division is suitable for pellets and other solid recovered fuels with a small particle size that can be worked with a scoop. When using manual increment division, the following procedure shall be followed:

a) place the whole composite sample on a clean, hard surface and homogenize it by mixing with a scoop;

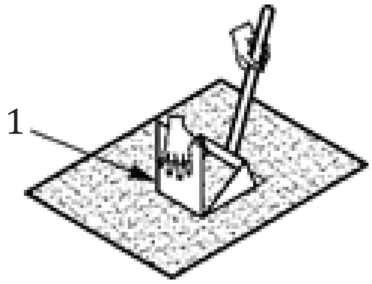
b) use the scoop to spread the composite sample into a rectangle with a thickness of not more than three times the nominal top size [see Figure 7 a)];

c) lightly mark the surface of the rectangle with the scoop to divide it into at least 20 parts [see Figure 7 b)];

d) use the scoop and a bump plate to take an increment from each of the 20 parts by inserting the scoop to the bottom of the heap each time [see Figure 7 c)] and combine the increments to form the required sub-sample.

a) Crushed sample spread into a rectangle with a maximum thickness three times the nominal top size

b) Arrangement of sample into 20 equal parts (e.g. into five equal parts length-wise and four equal parts breadth-wise)



c) Detail of taking an increment in one of the 20 parts by using the bump plate shown in b)

Key

1 bump plate

Figure 7 — Manual increment division

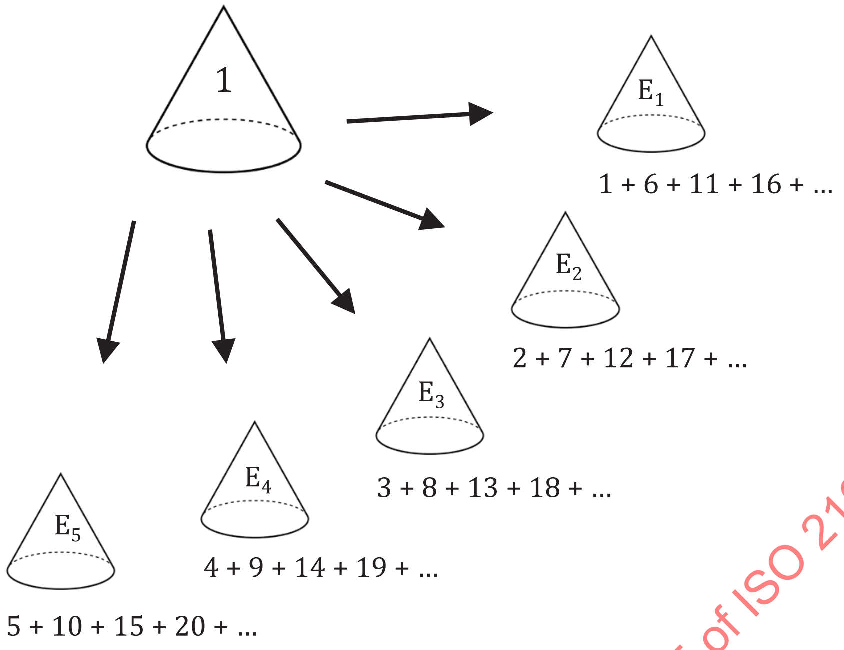
10.3.6 Rotary sample divider

A rotary sample divider can be used as a mechanical method to reduce the mass of a sample. The rotary sample divider shall have a feeder device adjusted so that the divider rotates at least 20 times while the sample is being divided. Figure 5 shows an example of a rotary sample divider.

10.3.7 Fractional shovelling

Fractional shovelling is used by splitting the sample to be divided into N twin fractions. Shovelfuls shall be taken from the (sub-)sample and shall be placed one by one, in order, onto the heaps, heap 1, heap 2, ... heap 5, and again heap 1, heap 2, ... heap 5, and so on, until the (sub-)sample is exhausted. Figure 8 illustrates the fractional shovelling method.

Fractional shovelling allows for the drawing of one sample with a mass $1/N$, several samples each with a mass $1/N$ and also combinations of several samples of $1/N$.



Key

1 lot *L*

NOTE Five potential samples: rate 1/5.

SOURCE Reference [11].

Figure 8 — Fractional shovelling (into *N* = 5 twin fractions)

10.3.8 Quartering

Quartering may be used for materials such as pellets, chips and fluff that can be worked with a shovel. It is suitable for producing sub-samples of these materials down to approximately 1 kg. When using mixing and quartering, the following procedure shall be followed:

a) place the whole mixed, composite sample on a clean, hard surface;

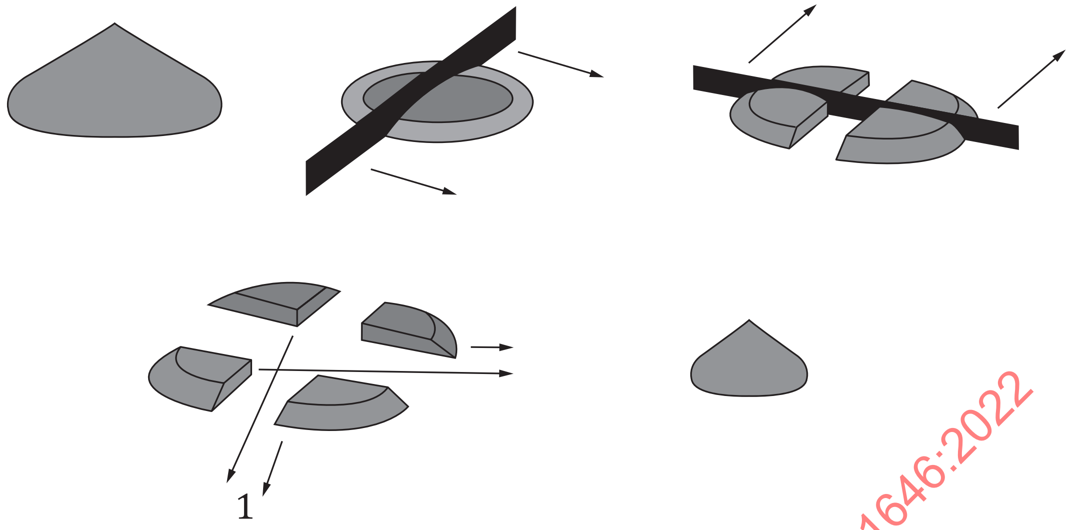
b) flatten the pile by inserting the shovel into it and spread the pile to form a flat heap that has a uniform thickness no greater than the blade of the shovel;

c) quarter the flat heap along two diagonals at right angles by inserting the shovel vertically into the heap, for which a sheet-metal cross may be used, if available;

d) discard one pair of opposite quarters;

e) repeat the mixing and quartering process [steps a) to d)] until a sub-sample of the required mass is obtained.

Figure 9 illustrates the quartering process [steps b) and c)].



Key

1 discard

Figure 9 — Quartering

Quartering is only used if the methods described in 10.3.2 to 10.3.7 are not suitable.

11 Methods for mass and particle size reduction of laboratory samples and general analysis samples

11.1 General

This clause describes the sample preparation procedures required to maintain the conformity and representativeness of a sample.

11.2 Initial sample division

If the initial mass of the laboratory sample exceeds the minimum sample mass or the adjusted minimum sample mass, the laboratory sample may be divided using one of the methods described in Clause 10. If this sample mass is larger than the maximum quantity requested by the laboratory, the particle size shall be reduced to enable an extra step of sample division to give a smaller sample mass. The remaining sample mass shall be kept above the minimum sample mass.

11.3 Initial mass determination

Before the laboratory sample is subject to any handling or treatment that can allow loss of moisture, mercury or dust, the mass of the laboratory sample shall be determined by weighing on a balance accurate to 0,1 % (or better) of the laboratory sample mass. This mass shall be recorded as $m_{\text{sample,1}}$, expressed in g.

11.4 Pre-drying

Pre-drying of wet samples is carried out to minimize moisture loss in the subsequent sample-division processes, to facilitate the sample preparation processes and to minimize biological activity. If it is necessary to dry a sample by heating, it shall be dried in an oven at a temperature not exceeding 40 °C. If it can be demonstrated that a higher pre-drying step does not have a systematic negative influence on mercury determination, a higher temperature for pre-drying of up to 60 °C may be used.

Total moisture determination shall be performed for a separate (sub-)sample taken specifically for this purpose.

NOTE 1 If the moisture content of the laboratory sample is unimportant, for example if only particle size distribution is to be determined or if a separate "total moisture analysis sample" is sampled as well, the calculation of the moisture loss can be omitted. In this case it is not necessary to obtain complete equilibrium with the temperature and the humidity in the laboratory as described in 11.7.

All samples, including those that have been dried by heating, shall be spread out in a tray to a depth of 50 mm to 100 mm and left for at least 24 h in the laboratory.

For coarse materials, 24-h retention time under laboratory conditions is possibly not long enough to become sufficiently dry to continue sample preparation. The moisture content shall be monitored by placing the sample or a sub-sample on an electronic balance during the retention time in the laboratory. The sample shall be placed in a coarse cutting mill fitted with a 30-mm sieve. Sample division may be done by alternative methods described in Clause 10.

NOTE 2 To prevent losses of moisture during the grinding process, a low-speed grinder is preferred.

If information about the moisture losses during the pre-drying is required, the mass of the sample after the pre-drying process has been completed shall be determined according to CEN/TS 15414-1, CEN/TS 15414-2 or ISO 21660-3, using the same balance for the weighing as used in 11.3. This mass shall be recorded as $m_{\text{sample},2}$, expressed in g.

If required, the moisture loss during pre-drying shall be calculated as a percentage of the initial mass of the sample using Formula (3):

$$M_{\text{p}} = 100 \times \frac{m_{\text{sample},1} - m_{\text{sample},2}}{m_{\text{sample},1}} \quad (3)$$

where

M_{p} is the moisture loss, in per cent;

$m_{\text{sample},1}$ is the initial mass of the sample;

$m_{\text{sample},2}$ is the mass of the sample after pre-drying.

The result to the nearest 0,1 % shall be recorded.

11.5 Particle size reduction to < 30 mm

If the material contains particles that will be retained on a 30,0 mm sieve, a 30,0 mm sieve shall be used to separate the sample into a coarse fraction (retained on the 30,0 mm sieve) and a fine fraction (passing through the 30,0 mm sieve).

Materials in the sample (e.g. hard alloy steel and stones) shall be removed from the sample, if necessary, before coarse cutting to protect the grinder from damage. Magnetic materials can be removed by using a magnet. The removed materials shall be weighed and reported in the sample preparation report (see Clause 14).

The coarse fraction shall be processed using a coarse cutting mill so that it passes through the 30,0 mm sieve.

NOTE 1 Depending on the material, mills with a cutting or grinding action as well as the coarse cutting action can be used.

For some types of solid recovered fuels (e.g. for rubber and plastics when these material types are difficult to grind at room temperature), pre-treatment by freezing can be necessary before grinding to ensure that the correct particle size of the analysis sample is achieved. If this is necessary, the sample shall be:

a) placed in a freeze-resistant bucket;

b) treated with carbon dioxide ice or liquid nitrogen;

c) removed immediately from the bucket to the inlet of the moving cutting mill (grinder).

CAUTION — Be aware of the hazards associated with using carbon dioxide ice or liquid nitrogen.

The processed coarse fraction and the fine fraction shall be recombined. The sample shall be homogenized.

If required, this procedure may be used to achieve particle size reduction to a size below 30 mm by the use of a suitable coarse cutting mill and sieve. More instructions on the different (cryogenic) crushing and cutting methods and when to use them are given in D.5.

NOTE 2 Grinding of the whole sample through the sieve fitted in the mill while producing the particle size needed can also assist in homogenizing the sample at the same time.

11.6 Sample division of < 30 mm material

One of the manual methods described in Clause 10 shall be used for sample division. The masses of retained sub-samples shall respect the values determined using Formula (1).

A balance accurate to 0,1 % of the sub-sample masses shall be used for the weighing required by this subclause. The masses of the containers, into which the sub-samples are to be put, shall be determined before starting the sample division.

The container shall be sealed immediately if a sub-sample is required for moisture content determination or another test for which it is important not to lose moisture.

The mass of each sub-sample shall be determined. This mass shall be recorded as $m_{\text{subsample},1}$, expressed in g.

When a sub-sample is to be used as a test portion, the sub-sample shall have a mass of not less than the minimum test-portion mass specified in the appropriate test method.

NOTE The minimum amount of sample for each step of sample preparation and sub-sampling can be directly estimated by Formula E.1.

11.7 Particle size reduction of < 30 mm material to < 1 mm

When a sub-sample of material is required with a nominal top size of about 1 mm, a cutting mill shall be used to reduce the whole of a sub-sample to this particle size. If necessary, the process shall be carried out in several steps using a finer sieve in the mill at each step, finishing with a sieve having an appropriate aperture.

If subpopulations with a different physical behaviour are expected (e.g. sand, metal particles), sample dividers should be used to avoid segregation.

Grinding or cutting to a nominal top size of 1 mm can cause problems, because some materials are difficult to grind and subsequently generate large amounts of heat when reduced in particle size. This heat affects the composition of the sample and can cause a loss of content (e.g. moisture or mercury). Therefore, it can be necessary to grind or cut the sample under cryogenic conditions or with a slow rotating grinder. If the temperature in the sample increases to more than 60 °C, the particle size reduction shall be performed under cryogenic conditions. As it is difficult to measure the temperature during milling, any temperature increase shall be monitored. In the case of temperature increase, the process shall be paused to allow it to cool before continuing.

In the case of cryogenic cutting or grinding, the mills or cutters shall be suitable for grinding or cutting at very low temperatures. Cooling shall be carried out with either solid carbon dioxide (−79 °C) or liquid nitrogen (−196 °C). The sample shall be mixed or immersed in the cooling medium. In some cases, it is necessary to cool the mill or cutter as well with the cooling medium to keep the temperature low enough. An increasing temperature can also be avoided by decreasing the throughput or rotation frequency of the mill or the cutter. This may also be applied as long as the temperature does not rise above 60 °C. In addition, cryogenic cutting or milling shall be performed if determination of the chloride content is required in samples that can possibly contain PVC.

NOTE Depending on the material, other mills than the cutting mill can be used.

EXAMPLE If the sample is to be reduced first to pass a 5-mm screen, and then to pass a 1-mm screen, the following procedure applies:

a) a 5,00-mm sieve is used to separate the sample into a coarse fraction (retained on the 5,00-mm sieve) and a fine fraction (passing the 5,00-mm sieve);

b) the coarse fraction is processed using a cutting mill containing a 5-mm screen;

c) the processed coarse fraction is recombined with the fine fraction and homogenized;

d) a 1,00-mm sieve is used to separate the sample into a coarse fraction (retained on the 1,00-mm sieve) and a fine fraction (passing the 1,00-mm sieve);

e) the coarse fraction is processed using a cutting mill containing a 1-mm screen;

f) the processed coarse fraction is recombined with the fine fraction and the sample is homogenized.

If the material contains granular particles, there is a chance that they will rotate in the mill or get stuck in the screen. Likewise, if the material contains very elongated particles, some of these particles can come to rest on the screen and not pass through. The mill shall be examined after the milling is completed. If any such particles are found, they shall be ground manually until they pass through the sieve. This material shall then be added to the sub-sample.

The sub-sample shall be spread out in a tray no more than a few millimetres deep. It shall be left for at least 4 h in the laboratory, until it reaches equilibrium with the temperature and humidity in the laboratory.

A sub-sample prepared by the method described in this subclause may be used as a general analysis sample, in which case its mass shall be at least the mass calculated in Table 3.

Material that has been subjected to the particle size reduction operations described in this subclause shall not be used for the determination of the total moisture content of the fuel.

11.8 Sample division of < 1 mm material

The material in the sample container shall be homogenized by stirring with a spatula. The spatula shall be used to take out the quantity required.

If subpopulations with a different physical behaviour are expected (e.g. sand, metal particles), sample dividers should be used to avoid segregation. The sub-sample mass taken for further grinding shall be large enough to ensure representativeness.

11.9 Particle size reduction of < 1 mm material to < 0,25 mm

When a sub-sample is required with a nominal top size of 0,25 mm, a cutting mill shall be used to reduce the whole of the sub-sample to this particle size. The cutting mill shall be fed with small portions of material from the general analysis sample. The whole of each portion shall be allowed to pass the 0,25-mm screen to prevent excess heat generation.

NOTE Depending on the material, other mills than the cutting mill can be used.

12 Handling considerations of the general analysis sample and the test portion

12.1 Key concepts

Preparation of the general analysis sample is a complex process because of a number of factors, such as:

- sample type and its physical state;
- initial amount of the laboratory sample;
- type and number of determinations to be carried out.

The prepared samples shall satisfy the following requirements at the same time:

a) each general analysis sample is representative of the laboratory sample;

b) the amount and the physical state (e.g. particle size) of each general analysis sample conform to the requirements of the respective analytical technique;

c) for each general analysis sample, losses of and contamination with respective analytes of interest is prevented.

The preparation of the general analysis sample from the laboratory sample, which is taken according to the sampling plan, is related to the requested analytical determinations (see Annex G for minimum analysis sample masses). This means that, if needed, all involved parties, such as the sampler, the customer and the analytical laboratory, shall agree on the requirements in the standards to be used for the requested determinations.

The preparation of general analysis samples involves the sequence of operations, such as homogenization, fraction separation, drying, reducing particle size and sub-sampling as specified in Clause 11. Specific forms of these operations are described in D.1 to D.6, respectively. A number of decisions on the specific order of these operations for a particular laboratory sample are to be made. In some cases, the sequence of operations to be applied is rather straightforward; in more complicated cases (e.g. when several determinations with different requirements will need to be performed), it can be critical to choose the right sequence of such operations.

To define the operations to be applied to a laboratory sample to produce one or more representative general analysis samples, the following three main steps shall be considered:

a) Define the requirements of analytical procedures of interest, including:

  - methods to be used;
  - number of test portions necessary;
  - quantity (mass) and properties of the test portions necessary for each analytical procedure in accordance with Annex G;
  - preservation requirements (e.g. time frame, temperature, addition of reagents).

  It is recommended that at least five times the amount of each test portion requirement is prepared as the general analysis sample.

b) Define the sequence of operations according to Figure D.1, based on the properties of the general analysis sample and the requirements of the analytical procedures, in which each single operation of this sequence shall be considered an independent process, namely:

  - fraction separation;
  - drying;
  - particle size reduction;

- homogenization;
- sub-sampling.

For practical reasons, it is recommended that the parameters are grouped in a way that general analysis samples with similar requirements can be prepared for several analytical parameters. The same general analysis sample can be used for different parameters if it fulfils the necessary requirements.

c) Choose, according to the requirements of the respective analytical techniques and the properties of the sample, the appropriate sample treatment technique within each module in accordance with Annex D.

12.2 Sequence of preparation procedures

Figure D.1 describes the procedure to enable decisions on the specific order of sample preparation procedures for a particular laboratory sample to produce representative test portions from the general analysis sample. This procedure shall be applied on the starting laboratory sample and repeated on all sample fractions or sub-samples subsequently obtained during the preparation process.

Losses of the volatile compounds (e.g. mercury) during homogenization and/or particle size reduction shall be avoided.

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

13 Storage, preservation and labelling of samples

The sampling plan shall identify the procedure(s) selected for packaging, preservation, storage and transport of the laboratory sample.

All composite samples, laboratory samples, sub-samples and general analysis samples shall be clearly identified and uniquely labelled with detail of the sample from which it was obtained. Samples shall be delivered and stored in tightly closed, high-density plastic containers.

If the period of storage of the samples is likely to be long-term or they are undried, the samples should be stored in tightly closed high-density plastic containers at or below 4 °C.

If biological parameters are to be taken into account, the retained sample(s) shall be frozen irrespective of the nature of the sample (e.g. solid or sludge types).

All possible sources of contamination during sample preparation of the laboratory sample according to this document (e.g. grinding with metallic apparatus, mainly aluminium or aluminium alloy) shall be avoided or reduced as much as possible.

14 Sample preparation report

The sample preparation report shall include at least the following information:

a) reference to this document, i.e. ISO 21646:2022;

b) name, address and location of all laboratories involved in the preparation of the sample;

c) date of receipt of laboratory sample and dates of the sample preparation procedures (beginning and end);

d) description and identification of the sample;

e) unique report identification with page number and total number of pages in the report recorded;

f) the sample preparation plan;

g) the mass of the laboratory sample received and any materials excluded from the sample preparation process (e.g. stones or metals);

h) any deviation from the sample preparation procedures and the reason for this deviation.

The impurities referred to in g) shall be taken into account by instruction included in each of the analysis standards when calculating any effect on specific analytical parameters.

15 Precision

The test results for a measurement on solid recovered fuels are usually applied for the enforcement of legal or other obligatory requirements (e.g. contracts). In such situations, it is vital to know the associated uncertainties for the tests.

Data regarding the uncertainty of sample preparation according to this document have been acquired through the validation investigation QUOVADIS. The results of QUOVADIS are not available as normative data on the precision for the following reasons:

— the trueness of a measurement regarding solid recovered fuel is by definition not available, because knowledge on the true composition of solid recovered fuel is by definition unknown;

— validation data needs to be available for each specific matrix and therefore for each type of solid recovered fuel.

Data on the precision of the methods for the preparation of laboratory samples of solid recovered fuels are shown in Annex H. Data regarding ruggedness for the preparation of the test sample from the laboratory sample are shown in Annex I.

Annex A
(normative)

Determination of the shape factor

A.1 General

This annex specifies how the shape factor shall be determined.

The shape factor of the particles has a large influence on the minimum (sub-)sample mass for materials where this factor is much smaller than 1,0. This correction is only necessary for solid recovered fuels like fluff, in particular fluff with a larger particle size.

A.2 Procedure

This annex specifies how the shape factor shall be calculated after particle size reduction. If the solid recovered fuels are of the fluff type, the shape factor *f* corrects the minimum sample mass, as such materials are not granular, but often composed of flat pieces.

For fluff-type materials, Formula (A.1) shall be used to determine the shape factor:

$$f = \frac{V_{95}}{{d_{95,l}}^3} \tag{A.1}$$

where

f is the shape factor, in mm^3/mm^3;

V_{95} is the maximum volume of a fluff particle (a mass fraction of 95 % of the particles are smaller than V_{95}), in mm^3 (where $V = l \times b \times h$);

d_{95} is the maximum length of a fluff particle (a mass fraction of 95 % of the particles are smaller than $d_{95,l}$), in mm.

The shape factor *f* is not constant but depends on the type of fluff-type material.

For pellet-type solid recovered fuels, which are roughly granular, the shape factor is not corrective (i.e. for pellet-type solid recovered fuels the value of *f* is considered to be 1,0).

Annex B
(normative)

Determination of the changing shape factor

B.1 General

This annex specifies how the shape factor changes during the process of particle size reduction.

The changing shape factor of the particles has a large influence on the minimum (sub-)sample mass for materials where this factor is much smaller than 1,0. This correction is only necessary for solid recovered fuels like fluff, in particular fluff with a larger particle size.

B.2 Procedure

This annex specifies how a prognosis shall be made of the shape factor after particle size reduction. Table B.1 gives a prognosis of the shape factor after particle size reduction based on the shape factor before particle size reduction, the global shape of the particles and how many times the nominal top size is decreased in the particle size reduction process. Instead of using Table B.1, a real analysis of the shape factor as specified in Annex A may be made.

The following aspects shall be taken into account:

a) Table B.1 does not provide every single possible result. So, in some cases an intermediate value shall be estimated by means of rounding up.

b) The values in Table B.1 were established for particles with specific dimensions that serve as a model for a specific type of particle (flat or elongated). Not only the particle types, but also the actual dimensions of the particles, are important. The values for the mixed or average shape of particles in Table B.1 were calculated by averaging the values for the extreme shapes.

EXAMPLE For flat particles, the length and breadth are always assumed to be equal, though in practice this is probably not the case. This assumption is generally not a problem, as the composition of solid recovered fuels is often so heterogeneous that particle dimensions can only be estimated.

c) For shape factor f = 0,25, no mixed or average shape type is included in Table B.1. For shape factor f_0 = 0,50 to f_0 = 1, no distinction whatsoever is made between shape types. The reason for this is that the effect of the shape type decreases as the shape factor increases, and so it is irrelevant in the long run.

Table B.1 — The shape factor after particle size reduction

Shape factor before reduction f_0	Global shape of the particles	Number of times d_{95} is reduced n													
		2	3	4	5	6	7	8	9	10	20	30	40	50	100
0,01	Flat	0,02	0,03	0,04	0,05	0,06	0,07	0,08	0,09	0,10	0,20	0,30	0,40	0,50	1,00
	Mixed/average	0,03	0,06	0,10	0,15	0,21	0,28	0,36	0,45	0,55	0,60	0,65	0,70	0,75	1,00
	Elongated/needle-shaped	0,04	0,09	0,16	0,25	0,36	0,49	0,64	0,81	1,00	1,00	1,00	1,00	1,00	1,00
0,02	Flat	0,04	0,06	0,08	0,10	0,12	0,14	0,16	0,18	0,20	0,40	0,60	0,80	1,00	1,00
	Mixed/average	0,06	0,12	0,20	0,30	0,42	0,56	0,58	0,59	0,60	0,70	0,80	0,90	1,00	1,00
	Elongated/needle-shaped	0,08	0,18	0,32	0,50	0,72	0,98	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,04	Flat	0,08	0,12	0,16	0,20	0,24	0,28	0,32	0,36	0,40	0,80	1,00	1,00	1,00	1,00
	Mixed/average	0,12	0,24	0,40	0,60	0,62	0,64	0,66	0,68	0,70	0,90	1,00	1,00	1,00	1,00
	Elongated/needle-shaped	0,16	0,36	0,64	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,05	Flat	0,10	0,15	0,20	0,25	0,30	0,35	0,40	0,45	0,50	1,00	1,00	1,00	1,00	1,00
	Mixed/average	0,15	0,30	0,50	0,63	0,65	0,68	0,70	0,73	0,75	1,00	1,00	1,00	1,00	1,00
	Elongated/needle-shaped	0,20	0,45	0,80	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,10	Flat	0,20	0,30	0,40	0,50	0,60	0,70	0,80	0,90	1,00	1,00	1,00	1,00	1,00	1,00
	Mixed/average	0,30	0,60	0,70	0,75	0,80	0,85	0,90	0,95	1,00	1,00	1,00	1,00	1,00	1,00
	Elongated/needle-shaped	0,40	0,90	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,15	Flat	0,30	0,45	0,60	0,75	0,90	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	Mixed/average	0,45	0,72	0,80	0,87	0,95	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	Elongated/needle-shaped	0,60	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,20	Flat	0,40	0,60	0,80	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	Mixed/average	0,60	0,80	0,90	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	Elongated/needle-shaped	0,80	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,25	Flat	0,50	0,75	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	Elongated/needle-shaped	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,50	All shapes	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,75	All shapes	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
1	All shapes	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

Annex C
(informative)

Examples of sample preparation procedures

C.1 General

This annex provides two examples of how this document can be used.

C.2 Example 1 – Pellets

A recycling plant that combines several types of waste residues from the industry produces pellets with a maximum particle size of 20 mm (assuming this is the diameter as with solid biofuels). Since the components in the products are already processed in a particle size reduction process, most particles inside the pellets can be assumed to be more or less granular. This results in a shape factor of approximately 1,0 (see Annexes A and B). The bulk density is 300 kg/m^3 and the particle density is 0,9 g/cm^3. The distribution factor is 1,00, because the particles show a wide range of evenly distributed particle sizes. C_V and p can be assumed to be 0,1. These figures have resulted in a minimum sample mass of 3,4 kg or 11,3 l, which are determined using Formula (E.1). For the analysis of chlorine, the sample will undergo sample preparation procedures resulting in a maximum particle size of 1,0 mm.

C.3 Example 2 – Fluff

A recycling plant separates municipal solid waste and produces a solid recovered fuel from the separation of the paper or plastic fraction by wind sifting. The particle size of this fraction is rather coarse (>500 mm) and the solid recovered fuel is therefore ground using a shredder to a final product with a nominal top size of 300 mm. Since the components in the products are mostly very flat, the average shape factor (see Annexes A and B) measures 0,05 with average dimensions. The bulk density is 90 kg/m^3 and the particle density is 0,9 g/cm^3. The distribution factor is 0,25, because the particles show a wide range of evenly distributed particle sizes. C_V and p can be assumed to be 0,1. These figures have resulted in a minimum sample mass of 143 kg or 1 590 l, which are determined using Formula (E.1). For the determination of the bulk density at least 40 kg of sample is needed that has not been ground. The solid recovered fuel is shredded to a particle size of 30 mm. For the analysis of mercury and other analytical parameters, a maximum particle size of 1,0 mm is required.

Table C.1 provides a worked-out sample preparation plan, including the activities to be performed on site.

Table C.1 — Example of sample preparation plan for fluff

Step	Description	Method of reduction	Used technique and apparatus	Mass before reduction	Mass after reduction	Nominal top size before reduction	Nominal top size after reduction	Shape factor before reduction	Shape factor after reduction	Mass to be withheld for analysis	Purpose of product of this reduction step
Step 1	Sample division of the composite sample into a sub-sample for further sample preparation and a sub-sample of untreated material	Sample division	Long pile reduction	183 kg	143 kg	300 mm	300 mm	0,05	0,05	40 kg	Determination of bulk density, durability of pellets, particle size distribution
Step 2	Particle size reduction in order to make further sample division possible	Particle size reduction to < 30 mm	Shredder	143 kg	143 kg	300 mm	30 mm	0,05	0,5		
Step 3	Sample division in order to reduce the remaining mass or obtain sub-samples as general analysis samples	Sample division	Long pile reduction	143 kg	1,43 kg	30 mm	30 mm	0,5	0,5		Sub-samples for determination of moisture content
Step 4	Particle size reduction of the remaining sub-sample in order to produce a general analysis sample	Particle size reduction to < 1,0 mm	Cutting mill	1,43 kg	1,43 kg	30 mm	1,0 mm	0,5	1,0		
Step 5	Sample division of the remaining sample material into the required general analysis sample(s)	Sample division	Incremental method	1,43 kg	1,0 g	1,0 mm	1,0 mm	1,0	1,0	1,0 g	Sub-samples for determination of ash, calorific value, chemical analysis
Step 6	Particle size reduction in order to make further sample division possible	Particle size reduction to < 0,25 mm									
Step 7	Sample division of the remaining sample material into the required test portions	Sample division									Sub-samples for analysis where < 0,25 mm is required

NOTE Mass can only be withheld during a sample division step and not during a size reduction step.

C.4 Large pieces of solid recovered fuels – Particle size-reduction and sub-population separation of laboratory samples

This subclause applies to samples of large pieces of solid recovered fuels taken for the purposes of characterization, especially by chemical analysis, requiring a particle size reduction down to 0,5 mm. Typical examples are shredded tyres and demolition wood. In both cases, the solid recovered fuel consists of large pieces (e.g. for shredded tyres in the range of 20 mm to 500 mm) to maximize energy production, since particle size reduction consumes significant amounts of energy. Consequently, the particle size reduction of the laboratory sample needs a special procedure to deal with large pieces and a large mass (e.g. for shredded tyres in the range of 20 kg to 30 kg) and the presence of several subpopulations which are mixed in the pieces (e.g. for shredded tyres rubber, metal, textile fibres).

The process described in this subclause is more complex than the usual particle size-reduction processes as a result of the large pieces aspects and the sub-populations separation. The fractions obtained at all the different stages are therefore to be weighed.

The following main steps are to be applied for particle size reduction of large pieces of solid recovered fuels and the separation of sub-populations:

a) obtain a representative laboratory sample and weigh it (e.g. for shredded tyres typically in the range of 20 kg to 30 kg), taking into account that large pieces can contain metal cable for which a saw can be used to extract the cable from the rubber.

b) reduce the particle size in a shredder using successively at least three different grids in order to reach the particle size of 10 mm.

For shredded tyres, the shredder has a typical capacity of 200 kg/h. A typical selection of grids is consecutively 40 mm, 20 mm and 10 mm. The shredder should be equipped with a ventilation system, including a filter to collect the light fraction (released fibres). The ventilation can also induce an air draught through the system resulting in a cooling effect. Cable passing through the grid is reintroduced in the shredder. This results in the “10 mm sample” to be weighed, as well as the collected fibres. Subsequently, the following steps are to be applied:

a) quarter the “10 mm sample” to obtain a sample of about 2,5 kg and weigh this sample;

NOTE 1 The other fractions are weighed and stored.

b) reduce the particle size in the same shredder using at least two different grids, where the last grid does not allow particles to go through it;

NOTE 2 For shredded tyres, a typical selection of grid is a 6 mm grid and a “solid” grid (i.e. without holes or with holes below 0,5 mm allowing air or nitrogen cooling). The shredder is fitted with a ventilation system, including a filter in order to collect the released fibres.

c) quarter this sample so as to obtain a sample of about 0,7 kg and weigh this sample;

NOTE 3 For shredded tyres, a sample of about 0,7 kg corresponds to 0,5 kg of rubber. The other fractions are weighed and stored.

d) separate the metal sub-population using a magnet;

NOTE 4 This is easily done by spreading the sample on a flat surface and slowly moving a magnet above this surface. For shredded tyres, this results in a “rubber fraction” consisting mainly of rubber and a “metallic fraction” consisting mainly of metals. Both fractions are weighed.

e) clean the light fraction collected at the different steps by sieving this light fraction;

NOTE 5 For shredded tyres, this results in a rubber powder considered as part of the “rubber fraction” and in a “fibres fraction” consisting mainly in fibres.

f) dip the powder sample into liquid nitrogen and thereafter micronize the powder sample down to 0,5 mm.

NOTE 6 The powder can then be divided with a rotary sample divider in the powder amounts needed for the requested determinations. The obtained samples are weighed.

g) store and send the obtained samples for the requested determinations;

h) produce a report on this treatment, including the mass determined at the different steps.

Annex D
(normative)

Guidelines for choosing sample preparation procedures

D.1 General

The preparation of a general analysis sample from a laboratory sample involves a sequence of operations such as homogenization, fractional separation, drying, particle size reduction and sub-sampling. This annex describes specific forms of these operations. Figure D.1 shows a flow chart of the steps detailed in this annex.

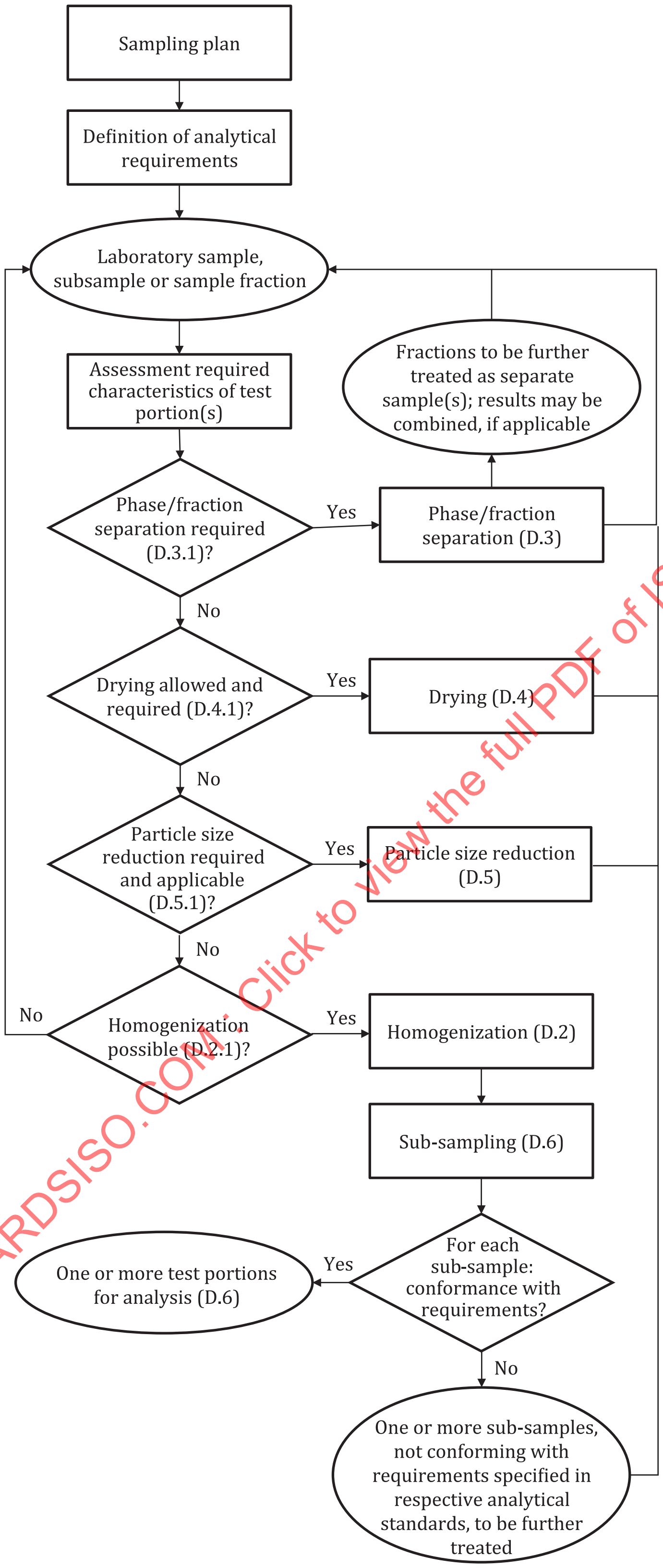


Figure D.1 — Flow chart of sequence of operations

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

D.2 Homogenization

D.2.1 General information

Before each operation that implies sub-sampling, a homogenization step is required to ensure 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, particle size reduction will be necessary before homogenization.

D.2.2 Homogenization techniques

D.2.2.1 Manual homogenization

Manual homogenization can be generally used, including in cases when mechanical homogenization can lead to loss of volatile compounds of interest (mercury).

Manual homogenization shall not be used for samples:

- that form layers due to the presence of particles of different density;
- with particles of such a large size that homogenization by manual mixing cannot be reached;
- that form layers due to the presence of particles with large differences in particle size.

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

D.2.2.2 Mechanical homogenization

Mechanical homogenization can be generally used, especially:

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

Mechanical homogenization shall not be used:

- for samples that form layers due to the presence of particles of different density; in this case, if homogenization is not possible, each layer shall be separated and treated as a different sample;
- when the apparatus can heat the sample and loss of volatile analytes can occur during this process; in this case, a manual homogenization shall be performed.

When using mechanical homogenization, this shall be performed according to the manufacturer's instructions.

NOTE For samples of small particle size, it is also possible to use a ball-mill without balls for homogenization.

D.2.2.3 Homogenization in the case of volatile compounds (mercury)

If mercury is to be analysed, sample homogenization shall be done very carefully and quickly to avoid losses. If losses cannot be avoided during the homogenization process, it can be necessary to take several non-homogenized sub-samples for analyses 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 representativeness of the remaining sample.

NOTE The presence of volatile compounds is included in the sampling plan.

D.3 Fraction separation

D.3.1 General information

Depending on the nature of heterogeneous samples and the determinations of interest, one or more techniques of fraction separation may 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 can be necessary. For this reason, the mass of each separated sub-sample shall be directly or indirectly measured after the separation to allow a final weighed combination of different fractions' analysis results.

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

D.3.2 Separation into different fractions

D.3.2.1 General

In cases of visible heterogeneity of separable fractions, the separation of different fractions can be necessary, especially if this can make subsequent particle size reduction, homogenization and sub-sampling easier.

D.3.2.2 Manual separation

Manual separation can be used when:

- several fractions can be distinguished;
- non-crushable fractions (e.g. copper wire) exist.

Manual separation shall not be used when contamination or losses of analytes of interest can occur.

When using manual separation, macroscopic pieces of a different nature shall be manually selected, either by hand (with protective gloves) or by using appropriate tools (e.g. tweezers, magnet). The selected pieces shall be stored in separate containers.

D.3.2.3 Sieving

Sieving can be used:

- when separation of fractions of different particle size is necessary;
- for checking the particle size of the sample or the particle size distribution.

Sieving shall not be used when contamination or losses of analytes of interest can occur.

When sieving, the sample shall be sieved by shaking, either by hand or apparatus, through sieves with appropriate mesh size and material.

D.4 Drying

D.4.1 General information

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

Drying is very likely to introduce analytical errors for volatile compounds and should be avoided when not strictly required. If a sub-sample or test portion for volatile compounds determination is to be dried, the actual drying technique shall be selected 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” to 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 mass (e.g. the sample can be just dried enough to make crushing or grinding possible).

D.4.2 Procedures

D.4.2.1 Air drying at room temperature

Air drying at room temperature can be used:

- in every case where drying can be reached in appropriate time without alterations of analytes of interest;
- in the case of mercury determination.

Air drying at room temperature shall not be used when time is critical, 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 using air drying at room temperature, the sample shall be spread on the trays in a thin layer to dry it to an appropriate moisture content. Possible contamination (e.g. by dust) shall be minimized. The use of a desiccator can accelerate the drying process for small amounts of (sub-)samples.

D.4.2.2 Oven drying at 40 °C

Oven drying at 40 °C can be used:

- when time is critical, and the properties of the sample do not allow for good drying in a reasonable time at room temperature;
- in the case of mercury determination.

Oven drying at 40 °C shall not be used when the properties of the sample do not allow for good drying in a reasonable time at this temperature, and a higher temperature drying step can be safely applied.

When using oven drying at 40 °C, the sample shall be spread on the trays in a thin layer and shall be dried in the oven at 40 °C. Air renewal can accelerate the drying process and may be applied, provided that it does not cause loss of dust-like particles.

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

D.4.2.3 Oven drying at 60 °C

Oven drying at 60 °C can be used:

- when time is critical, and the properties of the sample do not allow for good drying in a reasonable time at room temperature;
- in the case of mercury determination.

Oven drying at 60 °C shall not be used when the properties of the sample do not allow for thorough drying in a reasonable time at this temperature, and a higher temperature drying step can be safely applied.

When using oven drying at 60 °C, the sample shall be spread on the trays in a thin layer and shall be dried in the oven at 60 °C. Air renewal can accelerate the drying process and may be applied, provided that it does not cause loss of dust-like particles.

D.4.2.4 Oven drying at 105 °C

Oven drying at 105 °C can be used:

- on a sub-sample for which mercury is not determined;
- in the case of a single-stage total moisture content determination.

Oven drying at 105 °C shall not be used:

- in the case of mercury determination;
- in cases where auto-ignition can be expected.

When using oven drying at 105 °C, the sample shall be spread on the trays in a thin layer and shall be dried in the oven at 105 °C. Air renewal can accelerate the drying process and may be applied, provided that it does not cause loss of dust-like particles.

D.5 Particle size reduction

D.5.1 General information

To achieve a homogeneous and representative test portion, one or more particle-size reduction steps can be needed. The choice of the technique to be used depends strongly on the nature of the sample and the particle size needed.

Typically, particle-size reduction is a multi-step operation that implies the use of a sequence of different techniques. In some cases, it can 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 mercury due to heating, loss of dust-like material, and contamination coming from the equipment itself or from other samples. These aspects shall be taken into account when selecting the appropriate equipment. The equipment used shall be kept clean.

The test report shall clearly state the technique(s) and operating conditions used for particle-size reduction. Non-crushable fractions (e.g. copper wire) shall be separated in accordance with D.3.2, weighed and, if needed, analysed as separate sub-samples.

D.5.2 Procedures

D.5.2.1 Crushing or grinding

Crushing or grinding can be used:

- 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;
- to produce an analytical sample of the required particle size.

Crushing or grinding shall not be used:

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

When crushing or grinding, large pieces of the sample shall be broken and/or the sample shall be crushed with appropriate apparatus, according to the manufacturer's instructions, to the required particle size.

D.5.2.2 Milling

Milling can be used:

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

Milling shall not be used:

- when the initial particle size is too coarse; in this case, a crushing step is required;
- when not applicable because of the nature of the sample.

When milling, the sample shall be milled according to the mill manufacturer's instructions until it reaches the required particle size. Excessive heating of the sample during milling shall be avoided to prevent losses of mercury. A sequence of short and low-speed millings is to be preferred to a long and/or high-speed treatment. The equipment shall be cooled between each milling operation. The use of a freeze-head mill can minimize the loss of mercury.

NOTE If the sample has a plastic consistency, freezing it down to low temperatures (e.g. −20 °C to −30 °C) can make milling easier.

D.5.2.3 Freeze crushing

Freeze crushing can be used:

- when the sample contains large amounts of plastics;
- 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.

Freeze crushing shall not be used when contamination or losses of compounds of interest can be caused by the equipment.

When freeze crushing, the sample shall be wrapped in a polyethylene container. The wrapped sample shall be immersed in a Dewar vessel filled with sufficient liquid nitrogen. The container shall stand until the liquid nitrogen no longer boils vigorously. The container shall be cooled for approximately 10 min. After complete cooling, the container shall be retrieved from the liquid nitrogen. Large pieces of the sample shall be broken with a hammer and/or the sample shall be crushed with appropriate apparatus, according to the manufacturer's instructions, to the required particle size.

Alternatively, commercial apparatus for freeze crushing is available.

D.5.2.4 Cutting

Cutting can be used:

- 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).

Cutting shall not be used when contamination with compounds of interest can be caused by the equipment.

When cutting, the sample shall be cut according to the cutting mill manufacturer's instructions to the required particle size. Excessive heating of the sample during cutting shall be avoided to prevent losses of mercury. The equipment shall be cooled between each cutting operation. A cooled cutting mill should be used to minimize loss of mercury. Manual cutting with scissors can minimize the loss of mercury.

D.5.2.5 Freeze cutting

Freeze cutting can be used:

- when the sample has a plastic 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.

Freeze cutting shall not be used when contamination from the equipment can occur.

When freeze cutting, the sample shall be wrapped in a polyethylene container, if necessary. The (wrapped) sample shall be immersed in a Dewar vessel filled with sufficient liquid nitrogen. The container shall stand until the liquid nitrogen no longer boils vigorously. The container shall be cooled for approximately 10 min. After complete cooling, the sample shall be retrieved from the liquid nitrogen. The sample shall be cut with a preferably cooled cutting mill, according to the manufacturer's instructions, to the requested particle size.

D.6 Sub-sampling

D.6.1 General information

The laboratory sample is usually divided into different test portions for the analyses. Most analytical techniques allow only small quantities of test portions to be analysed. A laboratory sample can be very large in some cases. Then only a small, representative portion of the laboratory sample shall be taken for analysis (see Table G.1).

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 and one or more particle size reduction steps followed by homogenization steps are needed.

Annex E should be taken into account when determining the minimum amount of sample in relation to the particle size and the heterogeneity of the sample.

If representative sub-samples cannot be obtained due to practical limitations, replicates should be performed.

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

D.6.2 Manual division of solid samples by mixing and quartering

Manual division of solid samples by mixing and quartering can be used:

- in the case of a large amount of laboratory sample;
- when the sample is dry enough and there are no clumps in it;
- if there is no evidence of heterogeneous distribution of chemical constituents.

Manual division of solid samples by mixing and quartering shall not be used when the sample contains clumps that cannot be divided.

When using manual division of solid samples by mixing and quartering, the sample shall be spread on a flat surface coated by an inert sheet in a circular shape. The sample shall be mixed with a shovel and a cone shall be made. The sample shall be divided into four slices of the same shape and size, as shown in Figure D.2 (e.g. with a sheet metal cross). Two opposite slices shall be thoroughly mixed together and the remaining part of the sample shall be discarded. This sequence shall be repeated until the requested quantity of sub-sample is reached, provided that the obtained sub-sample is still representative of the laboratory sample.

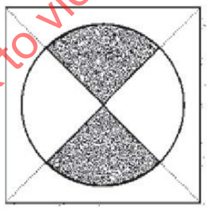


Figure D.2 — Division by quartering

D.6.3 Mechanical division of solid samples

Mechanical division of solid samples can be used:

- when the sample is dry enough and there are no clumps in it;
- when the particle size is small enough for the used apparatus;
- if there is no evidence of heterogeneous distribution of chemical constituents.

Mechanical division of solid samples shall not be used when the sample contains clumps that cannot be divided.

Mechanical sample splitters can achieve similar or better results than manual division. When using mechanical division of solid samples, the sample shall be divided by using the sample splitter (e.g. riffle box, rotary sample divider) according to the manufacturer’s instructions.

Annex E
(informative)

Relationship between minimum amount of sample and particle size – Formula for the estimation of the minimum amount of sample

The minimum amount of sample can be estimated by using Formula (E.1):

$$M_m = \frac{1}{6} \pi \times (D_{95})^3 \times f \times \rho \times g \times \frac{(1-p)}{{C_V}^2 \times p} \tag{E.1}$$

where

M_m is the mass of the sample, in g;

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

f is the shape factor for which the value is determined by using Formula (E.2):

$$f = \frac{V_{95}}{{d_{95l}}^3} \tag{E.2}$$

where

f is the shape factor, in cm^3/cm^3;

V_{95} is the "maximum" volume of a particle (a mass fraction of 95 % of the particles are smaller than V_{95}), in cm^3 (where $V = l \times b \times h$);

d_{95l} is the "maximum" length of a particle (a mass fraction of 95 % of the particles are smaller than d_{95l}), in cm;

NOTE 1 The shape factor is not constant but depends on the type of material. The shape factor generally increases when the sample undergoes comminution as the particles become more regular in form and, as a consequence, the shape factor will get closer to a value of 1. For materials which are granular-like or smaller than 50 mm, a shape facture value of 1 can be assumed.

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

NOTE 2 This parameter does not represent the bulk density of the sample, but the average density of all particles.

g is the correction factor for the particle size distribution of the material to be chosen from Table E.1.

NOTE 3 This value depends on the ratio between D_{95}/D_{05} and gives an indication for the particle size distribution. A D_{95}/D_{05} value close to 1 reflects a narrow particle size distribution (most particles of equal size); a high D_{95}/D_{05} value (>4) reflects a broad particle size distribution. D_{95} is the "maximum" particle size (a mass fraction of 95 % of the particles are smaller than D_{95}) and D_{05} is the "minimum" particle size (a mass fraction of 5 % of the particles are smaller than D_{05}).

p is the fraction of the particles with the property of interest, as a mass fraction (see Figure E.1 for examples);

NOTE 4 If every single particle of the sample carries the same amount of the parameter of interest, $p = 1$.

C_V is the desired coefficient of variation caused by the fundamental error.

NOTE 5 A typical value for C_V is 0,1. Depending of the nature of the sample and the aim of the analysis, it can vary from 0,1 to 0,3.

Table E.1 — Choice of particle size distribution correction factor

Ratio D_{95}/D_{05}	g
$D_{95}/D_{05} > 4$	0,25
$4 \geq D_{95}/D_{05} \geq 2$	0,50
$2 > D_{95}/D_{05} > 1$	0,75
$D_{95}/D_{05} = 1$	1,00

a) Example 1: the sample is made of 10 particles, and two of them carry the analyte of interest ≥ $p = 0,2$

b) Example 2: 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 E.1 — Examples of distribution of parameters in the sample

Table E.2 presents examples of minimum test portion/sample mass by using Formula (E.1) and applying the assumptions that $C_V = 0,1$, $g = 0,25$, $f = 1$ and $\rho_p = 1$ g/cm^3. The minimum mass of the sample required to provide adequate material for the respective analysis requirements should be in accordance with Annex G. If this is not the case, the mass should be increased to meet the requirements in D.6.1.

Table E.2 — Minimum test portion/sample mass

p		D_{95} cm						
		10	**4**	**1**	**0,4**	**0,2**	**0,1**	**0,02**
		M_m g						
Very heterogeneous (traces)	0,001	13 070 250	836 496	13 070	836	104,6	13,07	0,105

NOTE 1 The rate of heterogeneity is dependent on the parameters of interest and can vary for one sample (e.g. for major constituents $p \sim 0,1$ and for trace constituents $p \sim 0,001$). If the same sample or aliquot is to be used for determination of different parameters, the most conservative (lowest) value is to be used.

NOTE 2 For the shape factor f the most conservative value ($f = 1$) has been used. For cases with coarser particle sizes, it can be preferable to evaluate the actual shape factor, possibly resulting in smaller sample masses, especially for fibre- or fluff-like materials.

NOTE 3 The precision of the values shown are given for the user to enable the calculations to be checked if required.

Table E.2 *(continued)*

p		D_{95} cm						
		10	**4**	**1**	**0,4**	**0,2**	**0,1**	**0,02**
		M_m g						
Heterogeneous (minor constituents)	0,02	641 083	41 029	641	41	5,1	0,64	0,005
Rather homogeneous (major constituents)	0,1	117 750	7 536	118	8	0,9	0,12	0,001

NOTE 1 The rate of heterogeneity is dependent on the parameters of interest and can vary for one sample (e.g. for major constituents $p \sim 0{,}1$ and for trace constituents $p \sim 0{,}001$). If the same sample or aliquot is to be used for determination of different parameters, the most conservative (lowest) value is to be used.

NOTE 2 For the shape factor f the most conservative value ($f = 1$) has been used. For cases with coarser particle sizes, it can be preferable to evaluate the actual shape factor, possibly resulting in smaller sample masses, especially for fibre- or fluff-like materials.

NOTE 3 The precision of the values shown are given for the user to enable the calculations to be checked if required.

Annex F
(normative)

Sample preparation equipment

The preparation of general analysis samples from a laboratory sample frequently involves a sequence of operations such as homogenization, phase/fraction separation, drying, particle size reduction and sub-sampling. For these operations the equipment listed in Table F.1 shall be used.

Table F.1 — Sample preparation equipment

Equipment	Homogenization (see D.2)	Phase/fraction separation (see D.3)	Drying (see D.4)	Particle size reduction (see D.5)	Sub-sampling (see D.6)
Mill	X			X	
Freeze-head mill	X			X	
Shredder	X			X	
Cutting device				X	
Mechanical mixer	X				
Hammer				X	
Jaw-crusher				X	
Pestle and mortar	X			X	
Shovel	X				X
Sheet cross					X
Tweezer		X			
Magnet		X			
Riffle box					X
Tyler divider					X
Sieves		X		X	X
Drying oven			X		
Freeze drier			X		
Desiccator			X		
Balance		X	X	X	X

Annex G
(normative)

Characteristics of the laboratory sample for chemical analysis of solid recovered fuel

The requirements in Table G.1 apply when preparing the laboratory sample for the chemical characterization of solid recovered fuel samples according to this document. A maximum amount of laboratory sample of 10 kg and maximum particle size of 1 cm is established on the basis of number and type of parameters to be determined, sample representativeness and practical reasons for handling samples. Table G.1 summarizes the requirements for both single and grouped chemical parameters.

NOTE Equivalent requirements apply in all chemical test method publications for solid recovered fuel, i.e. ISO 21663, EN 15408, EN 15410, EN 15411, CEN/TS 15412 and EN 15413.

Table G.1 — Requirements for the laboratory sample for the analysis of solid recovered fuel

Parameter (single or group)	Minimum analysis sample mass[a] g	Short-term storage conditions before delivery to laboratory	Long-term storage condition before delivery to laboratory	Container material
C, H, N	100	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Plastic bottle or bag
Cl, S, Br, F	100	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Non-PVC plastic bottle or bag
Metallic Al	200	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Plastic bottle or bag
Major elements	400	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Plastic bottle or bag
Trace elements excluding Hg	200	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Plastic bottle or bag
Hg	100	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Glass or PFA bottle
C, H, N, Cl, S, Br, F	150	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Non-PVC plastic bottle or bag
Major elements + trace elements excluding Hg	500	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Plastic bottle or bag
Major elements + trace elements + Hg	600	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Glass bottle (100 g) + plastic bottle or bag
Major elements + trace elements + Hg + metallic Al	700	In the same condition of solid recovered fuel production	Refrigeration 4 °C	Glass bottle (100 g) + plastic bottle or bag

[a] The maximum particle size (mm) is related to the laboratory sample amount (g) in order to guarantee sample homogeneity. It is established following the rules reported in Annex E.