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Solid biofuels — Sample preparation

Biocombustibles solides — Préparation des échantillons

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Foreword

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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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This document was prepared by Technical Committee ISO/TC 238, *Solid biofuels*.

Introduction

Biofuels are a major source of renewable energy. International standards are needed for production, trade and use of solid biofuels. For sampling of solid biofuels, see ISO 18135.

This document can be used in regard to production, controlling and analysis of solid biofuels in general.

This document was developed with significant content from EN 14780:2011.

Solid biofuels — Sample preparation

1 Scope

This document defines methods for reducing combined samples (or increments) to laboratory samples and laboratory samples to sub-samples and general analysis samples and is applicable to solid biofuels.

The methods defined in this document can be used for sample preparation, for example, when the samples are to be tested for calorific value, moisture content, ash content, bulk density, durability, particle size distribution, ash melting behaviour, chemical composition, and impurities.

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 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18134-1, *Solid biofuels — Determination of moisture content — Oven dry method — Part 1: Total moisture — Reference method*

ISO 18134-2, *Solid biofuels — Determination of moisture content — Oven dry method — Part 2: Total moisture — Simplified procedure*

ISO 18135, *Solid Biofuels — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

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

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

3.1
nominal top size
aperture size of the sieve through which at least 95 % by mass of the material passes

Note 1 to entry: For pellets (and other long materials), the diameter is used to determine the nominal top size.

Note 2 to entry: Includes additional information not found in ISO 16559.

4 Symbols

M_p	moisture loss, in percentage
$m_{\mathrm{sample},1}$	initial mass of the sample, g
$m_{\mathrm{sample},2}$	mass of the sample after pre-drying, g
W	width and is at least 2,5 times the nominal top size of the material and should be wide enough for normal oversized material particles to enter the sampling device

5 Principles of correct sample reduction

The main purpose of sample preparation is that a sample is reduced to one or more test portions that are in general smaller than the original sample. The main principle for sample reduction is that the composition of the sample as taken on site shall not be changed during each stage of the sample preparation. Each sub-sample shall be representative of the original sample. To reach this goal, every particle in the sample before sample division shall have an equal probability of being included in the sub-sample following sample division. Two basic methods are used during the sample preparation. These methods are:

- sample division;
- particle size-reduction of the sample.

CAUTION — Avoid loss of moisture and fine particles during milling and other operations.

Because of the risk of changes in the moisture content (loss of moisture), a sub-sample (moisture analysis sample) shall be separated at the earliest possible stage of the sample preparation procedure. As an alternative, a separate moisture analysis sample may be taken. The sample reduction shall be carried out by a procedure that does not conflict with the requirements of ISO 18134-1 or ISO 18134-2.

For materials that have to be examined for moisture content, care should be taken to avoid any significant heat build-up and risk of drying.

6 Apparatus

6.1 Apparatus for sample division

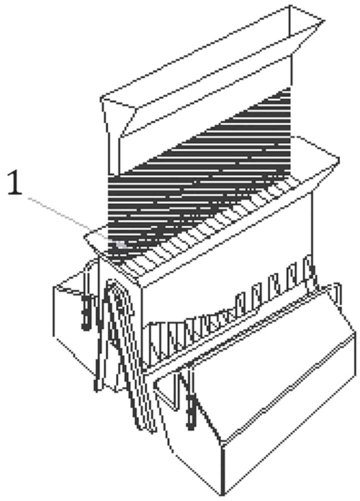
6.1.1 General

Sample division is the process of reducing the mass of the sample without reducing the size of the particles. This subclause gives some suitable apparatus for this purpose. To determine the correct use of each apparatus for different purposes, refer to Clause 8.

If the nominal top size of the sample is not known, a value of this parameter has to be assumed. After the sample reduction, the assumed value should be compared to the actual value to assure that the requirements of the apparatus for sample division and sample size used are met.

6.1.2 Riffle boxes

A riffle box should have an equal number of slots and at least six at each side (preferably more if possible), with adjacent slots directing material into different sub-samples, and the width of the slots shall be at least 2,5 times the nominal top size of the material to be riffled (see Figure 1).



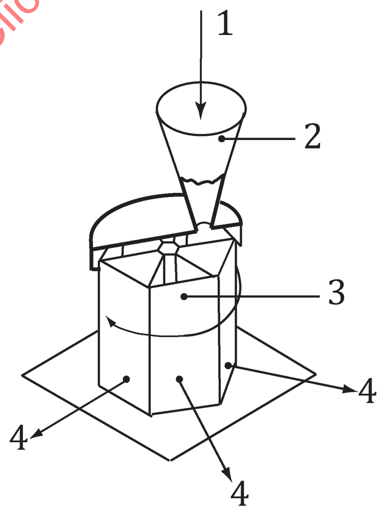
Key

1 width

Figure 1 — Example of a riffle box

6.1.3 Rotary sample dividers

The inner dimensions of the equipment where the sample is fed shall be at least 2,5 times as wide as the nominal top size of the material to be processed and should be large enough to handle normal oversized material particles. The rotary sample divider shall have a feeder device adjusted, so that the number of compartments multiplied by the number of rotations shall not be less than 120 while the sample is being divided. See Figure 2 for an example of a rotating divider.



Key

1 feeder

2 funnel

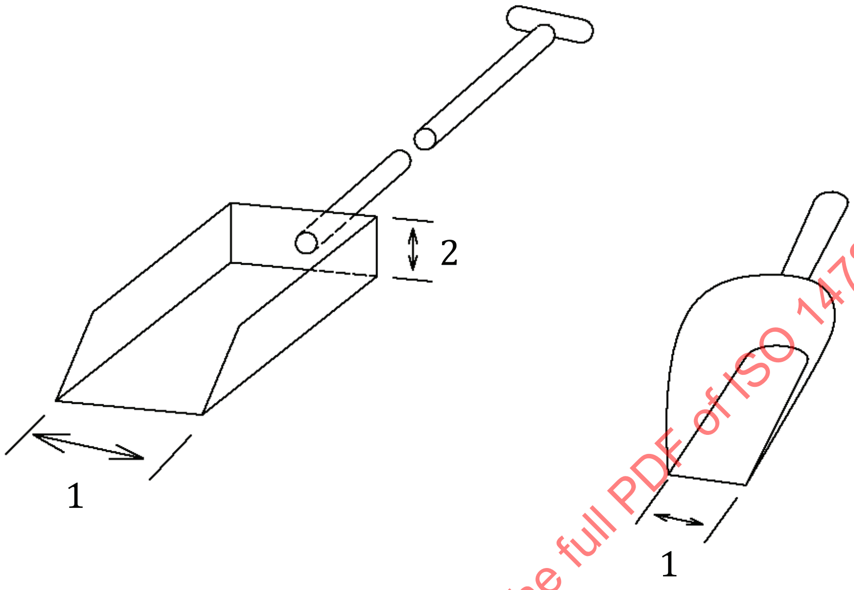
3 rotating receivers

4 divided sample

Figure 2 — Example of a rotary sample divider

6.1.4 Shovels and scoops

A shovel or scoop are tools used for manual sample division. They shall have a flat bottom, with edges raised high enough to prevent particles from rolling off, shall be at least 2,5 times as wide as the nominal top size of the material to be processed, and should be large enough to handle normal oversized material particles. See Figures 3 and 4 for examples of scoops and a shovel, respectively.



Key

1 width

2 height

Figure 3 — Example of scoops

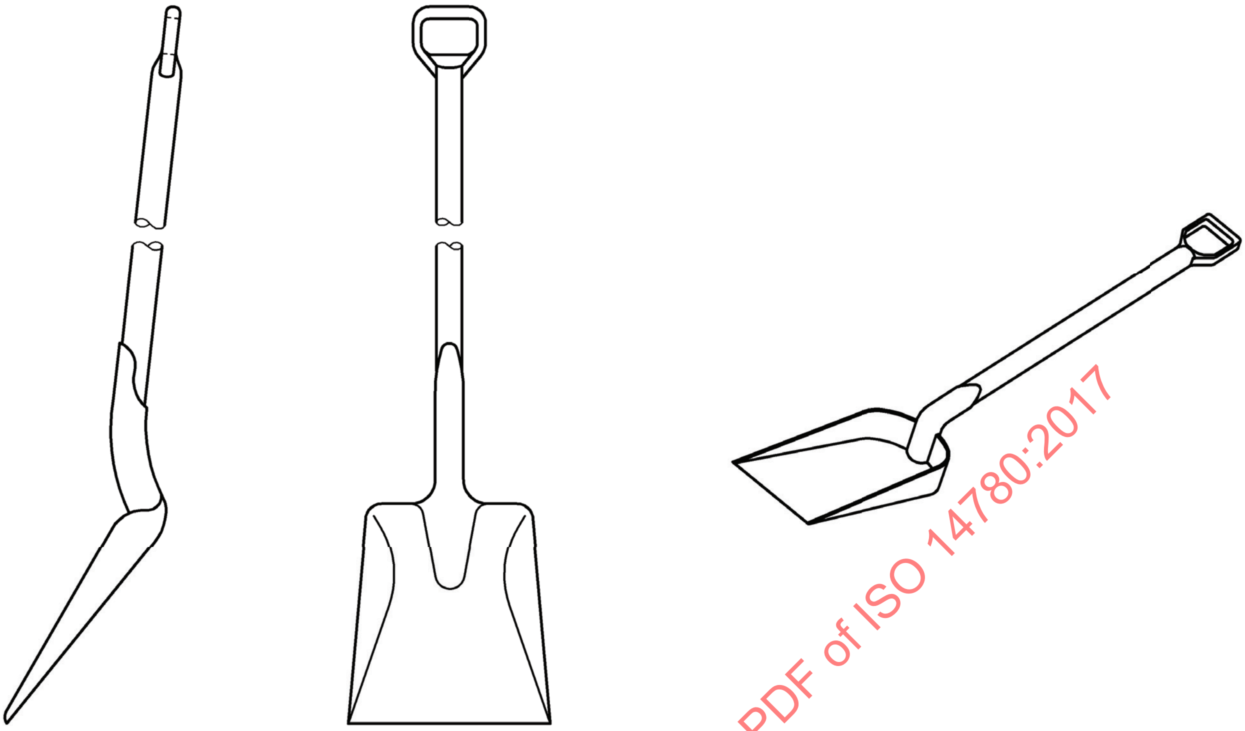


Figure 4 — Example of a shovel

6.2 Apparatus for particle size-reduction

CAUTION — When analysing metals (major or minor elements), take care that there is no contamination from the sample preparation or reduction equipment.

6.2.1 Coarse cutting mill or wood crusher

Coarse cutting mills can be used for cutting materials into lengths of about 10 mm to 30 mm (depending on the biofuel and the analyses to be performed). Drying of the material during coarse cutting shall be avoided by limiting heat production and air flow through the material. The equipment shall be designed so that it does not lose dust or contaminate the material with pieces of metal, and shall be easy to clean.

NOTE To prevent moisture losses during particle size reduction, a cutting mill or crushing mill with as low a cutting or crushing speed as possible is preferred.

6.2.2 Cutting mill

Cutting mills can be used for reducing the nominal top size of materials used as biofuels from about 10 mm to 30 mm down to about 1 mm or less (depending on the biofuel 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 they are designed so that they do not get blocked with the material that is being processed. Avoid the use of cutting mills whose cutting faces contain significant quantities of an element that is to be determined in the analysis.

NOTE 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.

6.2.3 Axe

An axe is used for cutting wood logs or coarse material down to a maximum 30 mm thickness or suitable size to be processed in a cutting mill provided with a 30 mm sieve.

6.2.4 Hand saw

A hand saw is used to saw off wood logs or coarse material down to a maximum 30 mm thickness or suitable size to be processed in a cutting mill provided with a 30 mm sieve.

A chain saw may contaminate the sample by chain oil and should therefore not be used. A saw machine should not be used for size reduction to avoid the risk of losing moisture in the sample as a result of heat caused by friction.

6.2.5 Sieves

A wire-mesh sieve with an aperture size of 1 mm, in accordance with ISO 3310-1, is recommended to check the nominal top size of general analysis samples. A wire-mesh sieve with an aperture size of 0,25 mm will be recommended if sub-samples with this nominal top size are required.

6.2.6 Balance

A balance is required 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.

7 Sample reduction — General principles

For every sample division stage to be carried out, it is important that a sufficient mass of material is retained, otherwise the sub-samples produced or the test portions to be taken may not be representative of the original sample. Due to various shape and size of different solid biofuels, different methods for determination of minimum sample masses have to be applied. Table 2 gives a guideline for minimum masses to be retained after each sample division stage, depending on the nominal top size of the material.

Regarding pellets, the pellets diameter shall be considered the nominal top size and the opening of the equipment shall be large enough for the longest pellets to pass through.

In addition to the minimum masses stated in Table 2, the mass after sample division shall be sufficiently large for the actual test or tests to be performed. Table 1 gives a guideline for the selection of the method to be applied to determinate the minimum sample mass to be retained after each sample division stage. Supplementary requirements concerning the masses of the test portions are given in international standards for test methods of solid biofuels.

Table 1 — Guideline to choose a method to determine the minimum masses to be retained during sample division stages

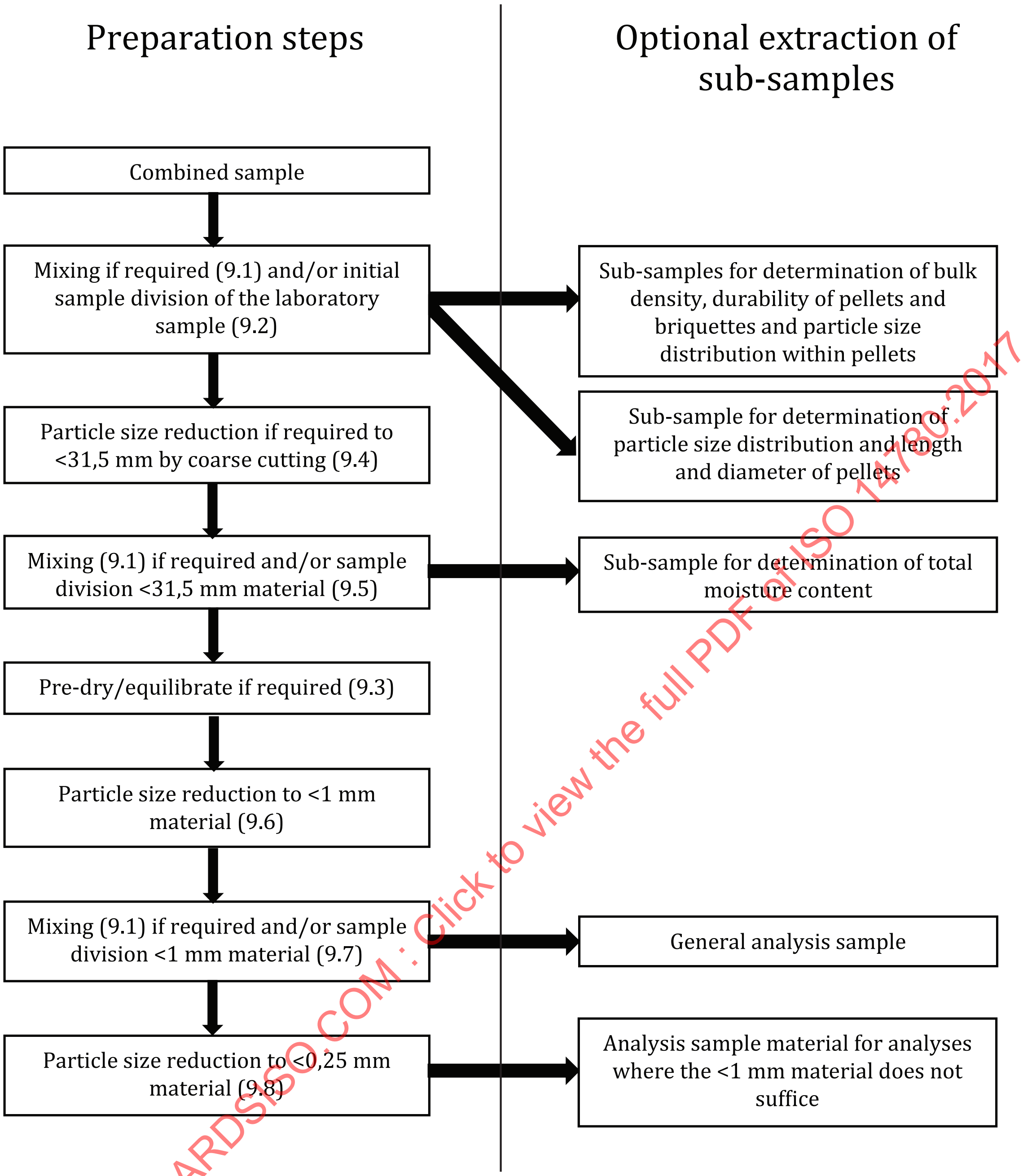
	1	2	3	4
Material	Bulk material of less than or equal to 100 mm nominal top size as defined in ISO 16559	Large pieces >100 mm material, e.g. wood logs or briquettes	Straw-like material with a low bulk density (≤BD 200 kg/m^3) and lengths >31,5 mm	Irregular/mixed materials, e.g. hogfuels, logging residues, bark
Method for determination of minimum sample mass	Calculate the minimum sample mass according to Table 2	Number of pieces should be stated, e.g. 10 pieces randomly collected	Minimum sample mass should be defined, e.g. 500 g	To be determined according to fuel type

Table 2 — Guideline for minimum masses to be retained during sample division stages, applicable for material according to column 1 in Table 1

Nominal top size mm	**Minimum mass** g
100	15 000
63	4 000
45	1 500
31,5	500
16	350
8	250
3,15	100
1	30
0,25	10

According to particle size distribution, shape and density of particles, the calculation of minimum sample mass should be adapted to the methods given here at the best in practical terms.

Figure 5 summarizes the range of steps that can be taken during sample reduction.



NOTE List of optional sub-sample extractions can not be exhaustive.

Figure 5 — Flow sheet for sample preparation and optional extraction of sub-samples

8 Methods for sample division

8.1 General

Combined samples may be divided into two or more laboratory samples and laboratory samples are in general further divided in sub-samples (test portions). This clause describes the methods and procedures for sample division. For every division step, the mass of retained material given in Table 2 shall be taken into account.

8.2 Riffling

This may be used for materials that can be passed through the riffle without bridging. It is not suitable for straw, bark, or other materials containing elongated particles, or for wet and/or sticky materials. Brittle materials should be handled with care to avoid the production of fine materials. Place the whole of the combined sample into one of the containers of the riffle so that it is evenly distributed throughout the container. Place the other two containers in position under the riffle. Pour the contents of the first container down the centre line of the riffle. Pour the biofuel sufficiently slowly to avoid bridging occurring. Do not move the container from side to side (this would cause the end slots to receive less biofuel). Discard the biofuel that falls into one of the containers, chosen at random. Repeat the riffling process until a sub-sample of the required mass is obtained. See Figure 1 for an example of a riffle box.

8.3 Strip mixing

This may be used for all materials and will be a convenient method when a combined sample is to be divided into a small number of laboratory samples. Place the whole combined sample on a clean, hard surface and homogenize it by mixing with a shovel, and form into a strip at least 20 times as long as it is wide. Distribute the sample along the length of the strip as evenly as possible, working randomly from end to end. Building up the strip with several layers will increase the quality of the division.

Obtain a laboratory sample by taking at least 20 increments from locations evenly spaced along the length of the strip. Take each increment by inserting two plates vertically into the strip and removing all the material from between the plates. 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 at least 2,5 times the nominal top size and at least the length of normal oversized material particles. See Figure 6 for the principle of strip mixing.

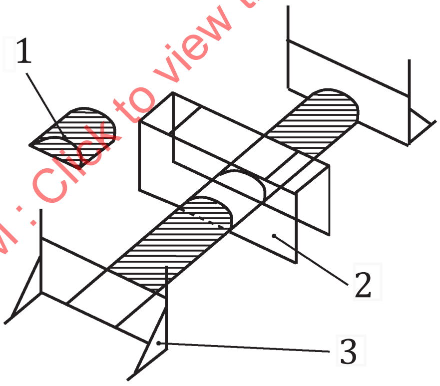


Figure 6 — Strip mixing

Key

1	increment
2	sampling frame
3	end plates

8.4 Long pile-alternate shovel method

This sub-sampling method is recommended for the reduction of samples in excess of approximately 50 kg. Place the entire combined sample on a clean and even floor and mix it thoroughly by manually moving the material to form a long pile. Carry out this process at least three times to ensure thorough mixing. When forming new piles, deposit each shovelful on the end of the new pile. Divide the last pile into two equal piles by using a shovel. Place alternate shovel loads to either side and form into two piles with at least 10 shovel loads (increments) in each pile. One pile is randomly selected and the process is repeated, using appropriately smaller increments as the piles are getting smaller. For straw-like material, this method may require excessive manual work.

For samples in excess of 500 kg, a mechanical shovel should be considered and used if appropriate.

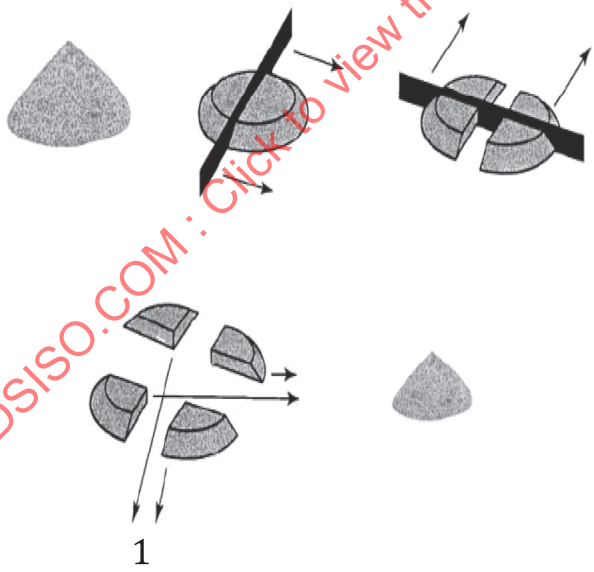
8.5 Rotary divider

A rotary sample divider is a mechanical method that can be used to reduce the mass of a sample. This method is recommended for dry free flowing material. The rotary sample divider shall have a feeder device adjusted, so that the number of compartments multiplied by the number of rotations shall not be less than 120 while the sample is being divided. See Figure 2 for an example of a rotary divider.

8.6 Coning and quartering

This method may be used for materials such as sawdust and woodchips that can be manually worked with a shovel. It is suitable for producing sub-samples of these materials down to approximately 1 kg. Place the whole combined sample on a clean, hard surface. Shovel the sample into a conical pile, placing each shovelful on top of the preceding one in such a way that the biofuel runs down all sides of the cone and is evenly distributed and different particle sizes become well mixed. Repeat this process three times, forming a new conical pile each time. Flatten the third cone by inserting the shovel repeatedly and vertically into the peak of the cone to form a flat heap that has a uniform thickness and diameter and is no higher than the blade of the shovel. Quarter the flat heap along two diagonals at right angles by inserting the shovel vertically into the heap (see Figure 7) (A sheet-metal cross may be used for this operation if available). Discard one pair of opposite quarters. Repeat the coning and quartering process until a sub-sample of the required size is obtained.

For materials and particles that tend to roll off the pile and cause segregation, this method should only be used when methods described in 8.2 to 8.5 cannot be applied.



Key

1 discard

Figure 7 — Coning and quartering

8.7 Mass reducing straw-like material (handful sampling)

This method is suitable for small sample sizes of straw-like material. Place the whole sample in an air-tight bag and homogenize it by inverting and kneading the bag several times. Take several handfuls and put them alternately into two piles. Homogenize the material remaining in the bag again, then remove several more handfuls and add them alternately to the two piles. Continue like this until the material in the bag is all used. At least 20 handfuls shall be placed into each of the two sub-samples.

9 Method for reducing laboratory samples to sub-samples and general analysis samples

9.1 Mixing

Mixing is recommended before each step of sample division. It should be noted that the sample division does not ensure that samples are adequately mixed. Additional mixing can be achieved by pouring it through a riffle (see 8.2) or a container-type divider (see Figure 2) three times, reuniting the parts after each pass.

Beware that mixing may result in loss or gain of moisture.

If the material is too wet to handle, pre-drying should be carried out first.

NOTE 1 In theory, thorough mixing of a sample prior to its division reduces errors occurring in sample preparation. In practice, this is not easy to achieve and some methods of hand mixing, e.g. forming and reforming into a conical pile, can have the opposite effect, leading to increased segregation.

NOTE 2 If mechanical sample dividers are used in the course of preparation, an additional mixing step is normally not necessary to meet the required precision.

NOTE 3 Mechanical mixing can be useful at the final stage of preparation of test samples.

9.2 Initial sample division

If the initial mass of the laboratory sample exceeds the minimum mass given in Table 2, the laboratory sample may be divided using one of the methods described in Clause 8.

If the material is too wet to handle, pre-drying should be carried out first.

9.3 Pre-drying

Pre-drying of wet samples is in general 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. All samples (including those that have been dried by heating) shall be spread out on a tray no more than a few particles deep and left for at least 24 h in the laboratory until they reach equilibrium with the temperature and humidity in the laboratory.

Before the laboratory sample or the divided laboratory sample according to 9.2 is subject to pre-drying, determine the mass of the sample by weighing on a balance accurate to at least 0,1 % of the sample mass. Record this mass as $m_{\text{sample,1}}$. After pre-drying and equilibrium with the surroundings, record the mass as $m_{\text{sample,2}}$.

Calculate the moisture loss during pre-drying as a percentage of the initial mass of the sample as shown in Formula (1):

$$M_{\text{p}} = 100 \times \frac{m_{\text{sample,1}} - m_{\text{sample,2}}}{m_{\text{sample,1}}} \tag{1}$$

where

M_{p} is the moisture loss in percentage;

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

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

Record the result to the nearest 0,1 %.

NOTE If the moisture content of the original sample is unimportant, e.g. only properties of the dry matter or mechanical properties are to be determined or if a separate "moisture analysis sample" is separated or sampled as well, the calculation of the moisture loss can be omitted. Also in this case, it is not necessary to obtain complete equilibrium with the temperature and humidity in the laboratory.

For coarse materials, for example, 24 h under laboratory conditions is not enough to reach equilibrium moisture content in all particles. Constancy in mass should be monitored by re-weighing at intervals at e.g. 4 h. The sample material should be turned around from time to time during the equilibrium process.

9.4 Coarse cutting (particle size reduction to <31,5 mm)

If the material contains particles that will be retained on a 31,5 mm sieve:

- use a 31,5 mm sieve to separate the sample into a coarse fraction (retained on the 31,5 mm sieve) and a fine fraction (passing the 31,5 mm sieve);
- process the coarse fraction using a coarse cutting mill so that it passes the 31,5 mm sieve;

NOTE 1 Depending on the material, other mills than the cutting mill can be used.

- re-combine the processed coarse fraction and the fine fraction and homogenize the sample.

If required, the above procedure may be used to achieve particle size reduction to a size below 31,5 mm by the use of a suitable coarse cutting mill and sieve.

NOTE 2 Grinding of the whole sample through the sieve fitted in the mill will produce the particle size needed.

9.5 Sample division of <31,5 mm material

One of the methods described in Clause 8 shall be used for sample division.

Determine the masses of the containers into which the sub-samples are to be put before starting the division.

Weigh the sub-sample masses using a balance accurate to 0,1 %. The masses of retained sub-samples shall comply with the values stated in Table 2.

Seal the container immediately if a sub-sample is required for moisture content determination or another test for which it is important not to lose moisture.

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

9.6 Particle size reduction of <31,5 mm material to <1 mm

Use a cutting mill for the preparation of the general analysis sample material <1 mm from the <31,5 mm material. Carry out the process in several steps, if necessary, using a finer sieve in the mill at each step, finishing with an appropriate sieve.

NOTE 1 Depending on the material, other mills than the cutting mill can be used.

NOTE 2 Grinding of the whole sample through the sieve fitted in the mill will produce the particle size needed and it also homogenizes the sample at the same time.

EXAMPLE If the sample is to be reduced first to pass a 5 mm screen and then to pass a 1 mm screen:

- use a 5 mm sieve to separate the sample into a coarse fraction (retained on the 5 mm sieve) and a fine fraction (passing the 5 mm sieve);
- process the coarse fraction using a cutting mill containing a 5 mm screen;

- re-combine the processed coarse fraction and the fine fraction and homogenize;
- use a 1 mm sieve to separate the sample into a coarse fraction (retained on the 1 mm sieve) and a fine fraction (passing the 1 mm sieve);
- process the coarse fraction using a cutting mill containing a 1 mm screen;
- re-combine the processed coarse fraction and the fine fraction and homogenize the sample.

If the material contains seeds or grains, there is a risk that they will rotate in the mill or get stuck in the screen. Likewise, if the material contains straw, some straw may come to rest on the screen and not pass through. Examine the mill after the milling is completed. If any such particles are found, grind them manually until they pass the sieve and add this material to the sub-sample.

Spread out the sub-sample in a tray no more than a few millimetres deep and leave it for at least 4 h in the laboratory until it reaches equilibrium with the temperature and humidity in the laboratory.

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

NOTE 3 Sample division can be carried out between subsequent particle reduction steps as long as the minimum mass in Table 1 is respected.

9.7 Sample division of <1 mm material

If required, e.g. for back-up samples, divide the <1 mm material into two or more sub-samples using one of the methods described in Clause 8. It is strongly recommended to use division instead of simple extraction by scoop/spatula whenever possible.

If impurities (like sand, etc.) are expected, then care should be taken especially for the segregation of particles, so the use of sample dividers is recommended. The sub-samples for further grinding shall keep the minimum masses stated in Table 2.

9.8 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, use a cutting mill to reduce one of the <1 mm sub-samples to this size. Feed the cutting mill with small portions of material from the <1 mm sub-sample and let each portion pass through the 0,25 mm screen to prevent excess heat generation.

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

10 Storage and labelling

Samples shall be placed in air-tight plastic containers or bags. Each sample shall be labelled with a unique identification number of the sample, date and time of sampling and identification number or code of the lot or sub-lot number.

11 Performance characteristics

Data regarding the uncertainty of this document have been acquired through the validation investigation, these among others being the BioNorm projects. These results give information on the uncertainty of the sample preparation and analysis. Results are presented in Annex A. For the validated fuel types, these values can be used in combination with their own laboratory performance characteristics and a desired coverage factor to get the overall uncertainty that is demanded.

The precision of the overall sampling shall be in accordance with ISO 18135.

Additionally, Annex B and Annex C are included to schematically communicate sample preparation for samples from single deliveries and from continuous delivery applications.

Annex A

(informative)

Precision in relation to division method

The referred results below were obtained in the research study of the EU projects BioNorm and BioNorm2. The study was performed as repeatability experiments where the sample types in Table A.1 were divided into 16 sub-samples by the reduction methods coning and quartering, long pile, riffle box and handful sampling with and without coarse cut, respectively. Each sub-sample was analysed for the parameters in Tables A.2 to A.13 as single analyses. The investigation of sample types 1, 2, 6, 8, 9 and 10 was performed by a Swedish laboratory, sample types 3, 4 and 5 by an Italian laboratory and sample type 7 by a Danish laboratory. The performance of the reduction methods was evaluated by comparison of the coefficient of variance ($C_{V,r}$) of the repeatability. Outliers were detected using the Dixon outlier test. The riffle box used in Sweden had a total number of eight slots with a width of 45 mm each, while the riffle box used in Italy had a total number of 16 slots and a slot width of 25 mm.

Table A.1 — Description of solid biofuel materials

Nr.	Sample type	Sub-sample size kg dry matter	Sample origin
1	Bark[a]	1,5 ± 0,05	Bark from Scots pine with a nominal top size of 100 mm
2	Wood chips[a]	0,5 ± 0,03	Wood chips from stem wood including bark with a nominal top size of 16 mm. Conifer: Deciduous 95:5
3	Olive residue[a]	0,5 ± 0,02	Olive residues (typical Mediterranean materials) with a nominal top size of 3 mm
4	Grape residue[a]	0,5 ± 0,03	Grape residues (typical Mediterranean materials) with a nominal top size of 16 mm
5	Pellets (6 mm)[a]	0,9 ± 0,05	6 mm pellets produced from a whole deciduous tree
6	Stem wood pellets (8 mm)[a]	0,5 ± 0,03	8 mm pellets produced from stem wood including bark (see wood chips above)
7	Wheat straw[b]	0,25 ± 0,01	Wheat straw in square bales
8	Sawdust[b]	0,25 ± 0,02	Sawdust from conifer with a nominal top size of 5,6 mm
9	Logging residue[b]	1,0 ± 0,07	Logging residue from conifer including bark with a nominal top size of 64 mm
10	Pellets (8 mm)[b]	0,5 ± 0,05	8 mm pellets from sawdust of conifer
11	Palm kernel shells[c]	0,029 ± 0,005	Palm kernel shell in 5 mm to 25 mm size available from the palm oil milling process

[a] Materials investigated in BioNorm2.

[b] Materials investigated in BioNorm.

[c] Palm Oil Engineering Bulletin No. 70.

Table A.2 — Performance data for moisture content (%)

	Coning and quartering				Long pile				Riffle box				Handful sampling without coarse cut				Handful sampling with coarse cut			
Sample type	***l***	***o***	x_m	$C_{V,r}$	***l***	***o***	x_m	$C_{V,r}$	***l***	***o***	x_m	$C_{V,r}$	***l***	***o***	x_m	$C_{V,r}$	***l***	***o***	x_m	$C_{V,r}$
1	16	0	54,2	0,57	16	0	56,2	0,59												
2	16	0	48,40	0,30	16	0	49,5	0,34	15	6	50,38	0,23								
3	14	12	5,33	0,79	16	0	10,7	0,30	14	12	4,91	1,6								
4	16	0	62,4	2,3	16	0	56,9	1,2	16	0	56,6	1,1								
7	16	0	15,6	0,74	16	0	15,5	1,0					16	0	16,0	1,8	16	0	15,3	1,8
8	16	0	55,2	0,47	15	6	54,8	0,45	16	0	55,6	0,52								
9	16	0	51,2	0,64	16	0	51,8	0,95	16	0	51,8	0,54								
10	16	0	5,67[a]	0,48[a]	15	6	5,49	0,77	16	0	5,45	0,64								
11													5	0	12,0	1.1				

[a] For sample type 10, a rotary divider was used instead of coning and quartering.

l number of outlier-free individual analytical values per level

o percentage of outlying values from the replicate determinations

x_m overall mean in %

$C_{V,r}$ coefficient of the variation of the replicates

Table A.3 — Performance data for ash content (%)

	Coning and quartering				Long pile				Riffle box				Handful sampling without coarse cut				Handful sampling with coarse cut			
Sample type	*l*	*o*	x_m	$C_{V,r}$	*l*	*o*	x_m	$C_{V,r}$	*l*	*o*	x_m	$C_{V,r}$	*l*	*o*	x_m	$C_{V,r}$	*l*	*o*	x_m	$C_{V,r}$
1	16	0	6,53	6,3	16	0	7,60	4,8												
2	16	6	0,59	7,8	14	0	0,77	2,6	16	0	0,74	2,4								
3	16	0	9,5	34,1	16	0	10,2	37,7	16	0	10,5	22,7								
4	16	0	6,60	8,1	16	0	7,02	9,1	16	0	7,98	10,0[a]								
5	16	0	0,61	21,8	16	0	0,54	35,9	16	0	0,47	17,1								
6	16	0	0,36	5,2	16	0	0,34	6,8	16	0	0,34	4,4								
7	16	0	4,9	4,7	16	0	4,1	4,7					16	0	4,2	4,6	16	0	4,2	3,5
8	16	0	0,29	8,8	16	0	0,28	10,5	16	0	0,39	13,0								
9	16	0	2,9	13,8	16	0	3,1	10,3	16	0	3,3	9,7								
10	16	0	0,30[b]	0,023[b]	16	0	0,34	6,6	16	0	0,34	2,4								
11													5	0	3,0	1,3				

[a] Grape material is sticky. This negatively influences the performance of the riffle box.

[b] For sample type 10, a rotary divider was used instead of coning and quartering.

Table A.4 — Performance data for calorific value (MJ/kg)

	Coning and quartering				Long pile			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
1	16	0	20,00	0,45	16	0	19,77	0,37

Table A.5 — Performance data for mechanical durability (%)

	Coning and quartering				Long pile				Riffle box			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
5	16	0	96,4	0,30	16	0	96,4	0,16	14	12	96,6	0,12
6	15	6	96,7	0,13	15	6	96,8	0,10	16	0	96,9	0,13

Table A.6 — Performance data for potassium content (mg/kg)

	Coning and quartering				Long pile				Riffle box			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
3	16	0	5 254	17	16	0	7 973	16	16	0	6 573	28
4	16	0	15 727	17	16	0	23 414	8	16	0	20 251	15

Table A.7 — Performance data for nitrogen content (%)

	Coning and quartering				Long pile				Riffle box			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
3	16	0	1,28	39,7	16	0	1,55	38,3	16	0	0,83	40,4
4	16	0	2,36	13,3	15	6	2,70	5,4	14	12	2,56	13,8

Table A.8 — Performance data for particle size distribution >8 mm to <16 mm (%)

	Coning and quartering				Long pile				Riffle box			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
2	16	0	13,7	6,8	16	0	14,0	7,8	16	0	12,3	7,1

Table A.9 — Performance data for particle size distribution >5 mm to <8 mm (%)

	Coning and quartering				Long pile				Riffle box			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
2	16	0	35,6	2,2	16	0	36,1	1,6	15	6	34,3	1,1

Table A.10 — Performance data for particle size distribution >3 mm to <5 mm (%)

	Coning and quartering				Long pile				Riffle box			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
2	16	0	32,5	2,7	16	0	31,3	2,4	16	0	33,1	2,2

Table A.11 — Performance data for particle size distribution >2 mm to <3 mm (%)

	Coning and quartering				Long pile				Riffle box			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
2	16	0	8,0	5,6	16	0	7,8	5,9	16	0	8,4	3,2

Table A.12 — Performance data for particle size distribution <2 mm (%)

	Coning and quartering				Long pile				Riffle box			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
2	16	0	9,7	4,0	16	0	10,2	5,5	16	0	11,0	3,2

Table A.13 — Performance data for chloride content (%)

	Coning and quartering				Long pile				Handful sampling without coarse cut				Handful sampling with coarse cut			
Sample type	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$	l	o	x_m	$C_{V,r}$
7	16	0	0,397	1,5	16	0	0,404	2,0	16	0	0,391	2,7	16	0	0,389	1,5