

BS EN 14780:2011



BSI Standards Publication

# Solid biofuels — Sample preparation

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**National foreword**

This British Standard is the UK implementation of EN 14780:2011. It supersedes DD CEN/TS 14780:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/17, Solid biofuels.

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

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Supersedes CEN/TS 14780:2005

English Version

**Solid biofuels - Sample preparation**

Biocombustibles solides - Préparation des échantillons

Feste Biobrennstoffe - Probenherstellung

This European Standard was approved by CEN on 5 May 2011.

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## Foreword

This document (EN 14780:2011) has been prepared by Technical Committee CEN/TC 335 "Solid biofuels", the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2011, and conflicting national standards shall be withdrawn at the latest by December 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 14780:2005.

This document differs from CEN/TS 14780:2005 mainly as follows:

- a) results of interlaboratory tests are supplemented as informative annexes;
- b) new definitions for sampling are introduced;
- c) a method for determining the minimum number of increments for sampling is included;
- d) the whole document is restructured and editorially revised;
- e) decision schemes are updated;
- f) updated normative references are included.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

## Introduction

Biofuels are a major source of renewable energy. European Standards are needed for production, trade and use of solid biofuels. For sampling and sample preparation of biofuels the following European Standards can be used:

EN 14778, *Solid biofuels – Sampling*

EN 14780, *Solid biofuels – Sample preparation*

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

## 1 Scope

This European Standard describes 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 described in this European Standard may 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. 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 referenced documents are indispensable for the application 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.

EN 14588:2010, *Solid biofuels — Terminology, definitions and descriptions*

EN 14774-1, *Solid biofuels — Determination of moisture content — Oven dry method — Part 1: Total moisture — Reference method*

EN 14774-2, *Solid biofuels — Determination of moisture content — Oven dry method — Part 2: Total moisture — Simplified procedure*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 14588:2010 and the following apply.

### 3.1

#### **combined sample**

sample consisting of all the increments taken from a sub-lot

NOTE The increments may be reduced by division before being added to the combined sample.

### 3.2

#### **general analysis sample**

sub-sample of a laboratory sample having a nominal top size of 1 mm or less and used for a number of chemical and physical analyses

### 3.3

#### **increment**

portion of fuel extracted in a single operation of the sampling device

### 3.4

#### **laboratory sample**

combined sample or a sub-sample of a combined sample for use in a laboratory

### 3.5

#### **lot**

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

NOTE See also sub-lot.

### 3.6

#### **moisture analysis sample**

sample taken specifically for the purpose of determining total moisture according to EN 14774-1 and EN 14774-2

**3.7 nominal top size**  
aperture size of the sieve used in CEN, method for determining the particle size distribution of solid fuels, through which at least 95 % by mass of the material passes

**3.8 particle size-reduction**  
reduction of the nominal top size of a sample or sub-sample

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

**3.10 sample division**  
division of a sample or sub-sample to a appropriate size. This usually always leads to a mass reduction of a sample or sub-sample

**3.11 size analysis sample**  
sample taken specifically for the purpose of determining particle size distribution

**3.12 sub-lot**  
part of a lot for which a test result is required

**3.13 sub-sample**  
portion of a sample

**3.14 test portion**  
*sub-sample* either of a *laboratory sample* or a *test sample*

**3.15 test-sample**  
laboratory sample after an appropriate preparation made by the laboratory

## 4 Symbols and abbreviations

$M_p$  is the moisture loss, in percentage  
 $m_{\text{sample},1}$  is the initial mass of the sample, g  
 $m_{\text{sample},2}$  is the mass of the sample after pre-drying, g  
W is the width and is at least 2,5 times the nominal top size of the material

## 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 requirements of EN 14774-1 or EN 14774-2.

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

## 6 Apparatus

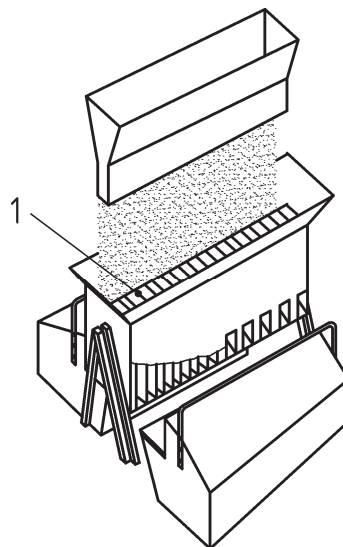
### 6.1 Apparatus for sample division

#### 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.

#### 6.1.2 Riffle boxes

A riffle box shall have an equal number of slots and at least 6 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).



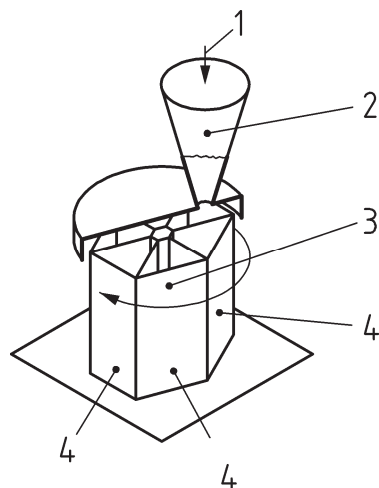
#### Key

1 W - slot width is at least 2,5 times the nominal top size of the material

**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. 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.



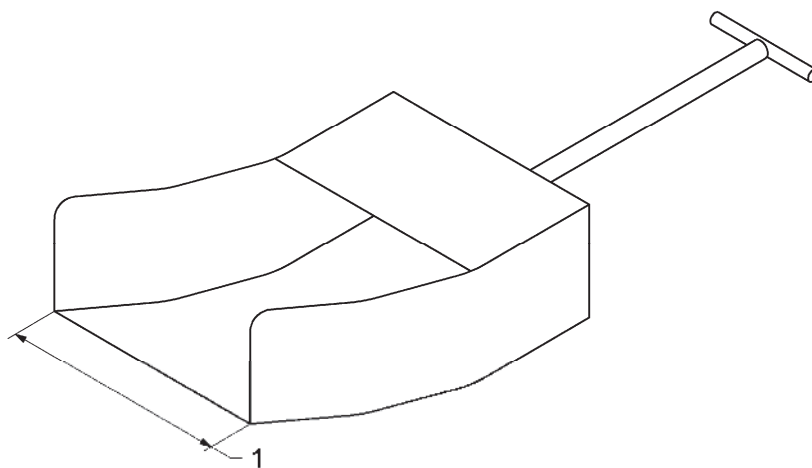
**Key**

- 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 rolling off, and be at least 2,5 times as wide as the nominal top size of the material to be processed. See Figures 3 and 4 for examples of a scoop and a shovel respectively.



**Key**

- 1 W width of the scoop

**Figure 3 — Example of a scoop**

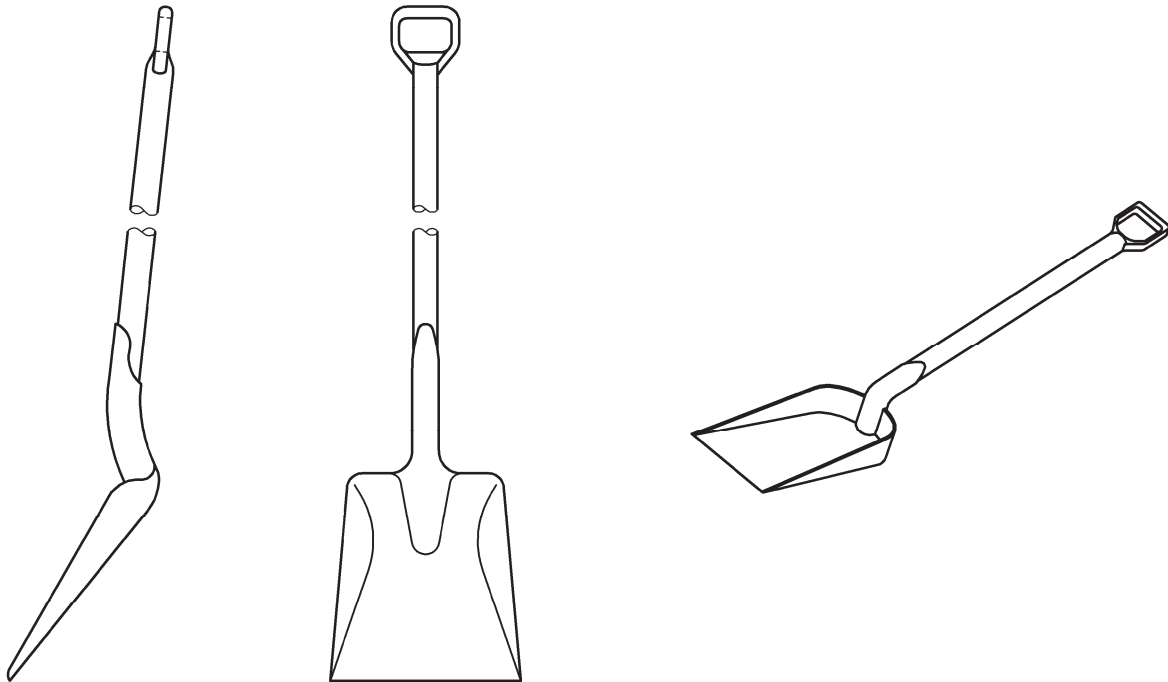


Figure 4 — Example of a shovel

NOTE For very large sample sizes, it is more practical to use a push dozer or bulldozer or any other heavy machinery that has a large "scoop" with raised edges.

## 6.2 Apparatus for particle size-reduction

**CAUTION —** When analysing metals, 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 are 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 should 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 losses of moisture during particle size reduction a grinder with as low a grinding speed as possible is preferred.

### 6.2.2 Cutting mill

Cutting mills are 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 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 maximum 30 mm thickness or suitable size to be processed in a cutting mill provided with a 30 mm sieve.

NOTE 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.

## 6.3 Sieves

A wire-mesh sieve with an aperture size of 1,00 mm is required to check the nominal top size of general analysis samples. A wire-mesh sieve with an aperture size of 0,25 mm will be required if sub-samples with this nominal top size are required.

## 6.4 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

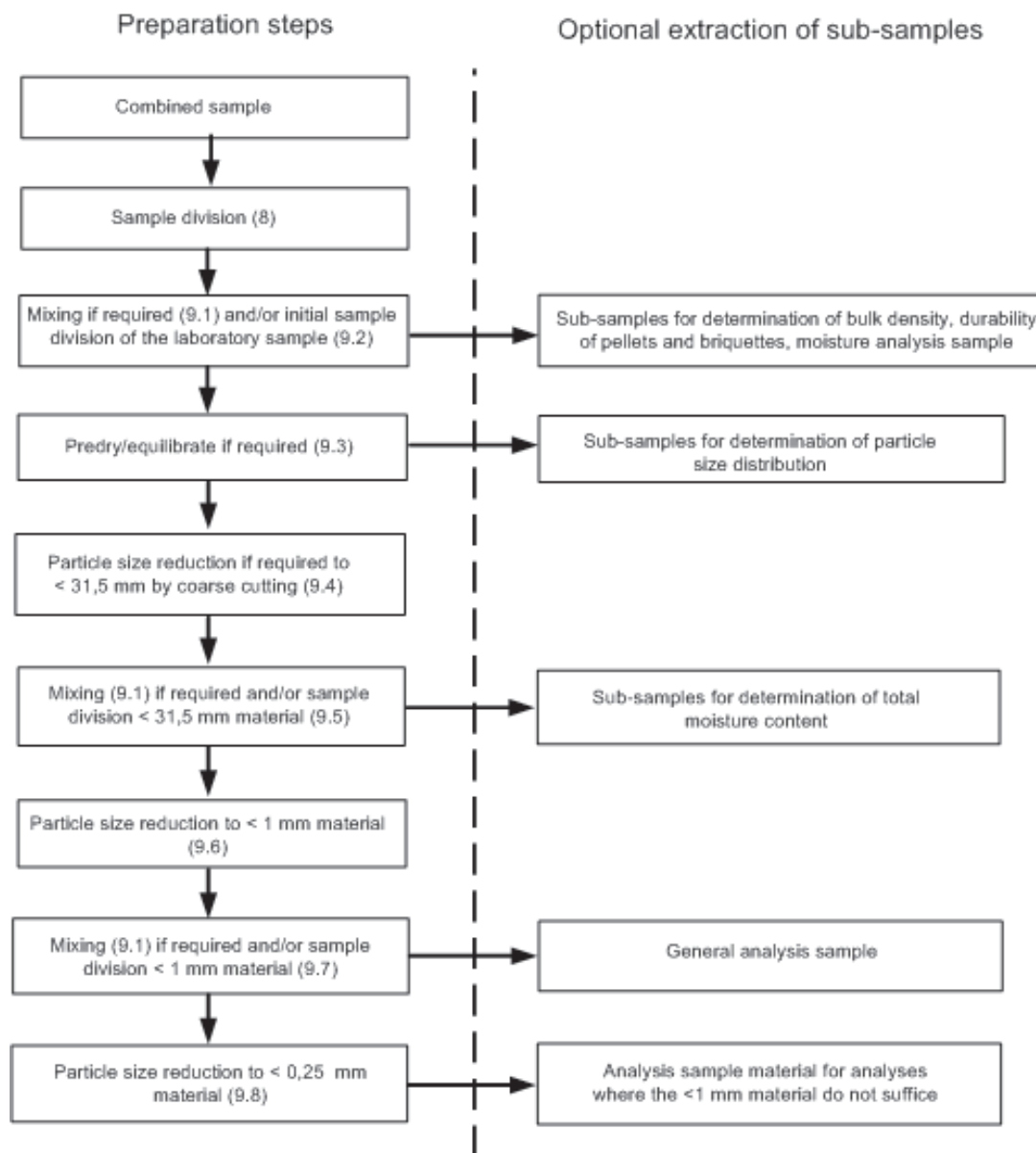
For every sample division stage to be carried out it is important that a sufficient mass of material is retained, otherwise the sub-samples produced or the test portions to be taken may not be representative of the original sample. Table 1 gives a guideline for minimum masses to be retained after each sample division stage, depending on the nominal top size of the material. In addition to the minimum masses stated in Table 1, it shall be ensured that the mass after a sample division stage is sufficiently large for the actual test or tests to be performed. Supplementary requirements concerning the masses of the test portions are given in European Standards for test methods of solid biofuels.

**Table 1 — Guideline for minimum masses to be retained during sample division stages**

<b>Nominal top size (mm)</b>	<b>Minimum mass (g)</b>
200	120000
150	50000
100	15000
63	4000
50	2000
45	1500
40	1000
30	500
10	150
5	100
1	50
0,25	10

NOTE This guideline does not take in to account: particle size distribution, shape of a particle, density of a particle, etc.

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



NOTE List of optional sub-sample extractions may 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 1 shall be taken into account.

### 8.2 Riffing

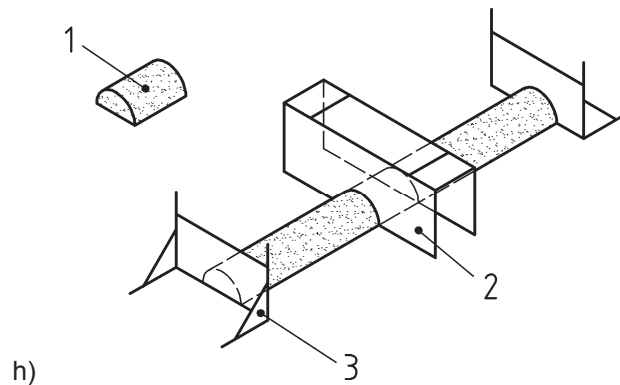
g) This may be used for materials that can be passed through the riffle without bridging. It is not suitable for straw, bark, or other materials containing elongated particles, or for wet and/or sticky materials. Brittle materials should be handled with care to avoid the production of fine materials. 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 homogenise 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. See Figure 6 for the principle of strip mixing.



#### Key

- 1 increment
- 2 sampling frame
- 3 end plates

Figure 6 — Strip mixing

### 8.4 Long pile-alternate shovel method

This sub-sampling method is recommended for the reduction of samples in excess of approximately 50 kg. When the entire combined sample is on the floor, mix it thoroughly by turning it over. Carry out this operation at least three times to ensure thorough mixing. When forming the new piles, deposit each shovelful on the end of the new pile.

i) Lay the combined sample out in a long pile, and separate 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.

j) For straw like material this method may require excessive manual work.

NOTE 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 is only to be recommended when 8.2 to 8.5 cannot be applied. This can be used for materials such as sawdust and woodchips that can be worked with a shovel or in case of straw using a fork. 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.

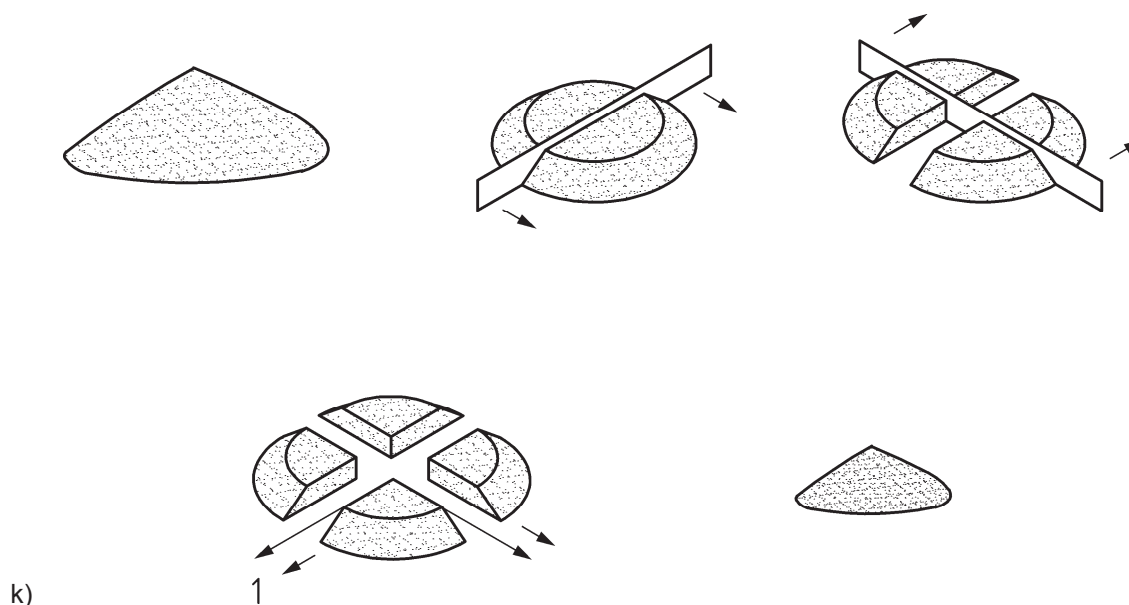


Figure 7 — Coning and quartering

## 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 homogenise it by inverting and kneading the bag several times. Take several handfuls and put them alternately into two piles. Homogenise 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. Often sample division includes an appropriate mixing. Additional mixing can be achieved by pouring it through a riffle (8.2) or a container-type divider (see Figure 2) three times, reuniting the parts after each pass.

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. Mixing can therefore result in loss of moisture.

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

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

NOTE 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 1, the laboratory sample shall 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 minimise moisture loss in the subsequent sample-division processes, to facilitate the sample preparation processes, and to minimise 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 clause 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:

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

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 1 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.

NOTE 2 For example coarse materials 24 h under laboratory conditions is not enough to reach equilibrium moisture content in all particles. Monitor whether constancy in mass is obtained 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 homogenise 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 and it also homogenises the sample at the same time.

#### 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 1.

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 homogenises 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 homogenise;
- 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 homogenise 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 mm 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 clause shall not be used for the determination of the total moisture content of the fuel.

### **9.7 Sample division of < 1 mm material**

If required, e.g. for back-up samples, mix the material (see 9.1) and 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 shall be taken especially for the segregation of particles, so the use of sample dividers is recommended. The sub-samples taken for further grinding shall be large enough to ensure they are representative.

### **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 stored in tightly-closed containers. Each sample shall be labelled with a unique identification containing the identification of the sample from which it was obtained.

## **11 Performance characteristics**

Data regarding the uncertainty of EN 14780 has 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 informative 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.

For the precision of the overall sampling see EN 14778.

## 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 were evaluated by comparison of the Coefficient of Variance ( $CV_r$ ) of the repeatability. Outliers were detected using the Dixon outlier test. The riffle box used in Sweden had a total number of 8 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 biofuel materials**

Nr.	Sample type	Sub-sample size (kg dry matter)	Sample origin
1	Bark <sup>a)</sup>	1,5 ± 0,05	Bark from Scots pine with a nominal top size of 100 mm
2	Wood chips <sup>a)</sup>	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 <sup>a)</sup>	0,5 ± 0,02	Olive residues (typical Mediterranean materials) with a nominal top size of 3 mm.
4	Grape residue <sup>a)</sup>	0,5 ± 0,03	Grape residues (typical Mediterranean materials) with a nominal top size of 16 mm.
5	Pellets (6mm) <sup>a)</sup>	0,9 ± 0,05	6 mm pellets produced from a whole deciduous tree
6	Stem wood pellets (8mm) <sup>a)</sup>	0,5 ± 0,03	8 mm pellets produced from stem wood including bark (see wood chips above)
7	Wheat straw <sup>b)</sup>	0,25 ± 0,01	Wheat straw in square bales
8	Sawdust <sup>b)</sup>	0,25 ± 0,02	Sawdust from conifer with a nominal top size of 5,6 mm
9	Logging residue <sup>b)</sup>	1,0 ± 0,07	Logging residue from conifer including bark with a nominal top size of 64 mm
10	Pellets (8mm) <sup>b)</sup>	0,5 ± 0,05	8 mm pellets from sawdust of conifer
a) Materials investigated in BioNorm2			
b) Materials investigated in BioNorm			

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

Sample type	Coning and quartering				Long pile				Riffle box				Handful sampling without coarse cut				Handful sampling with coarse cut			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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 <sup>a)</sup>	0,48 <sup>a)</sup>	15	6	5,49	0,77	16	0	5,45	0,64								

a) For sample type 10 a rotary divider was used instead of coning and quartering

where

*l* is the number of outlier-free individual analytical values per level

*o* is the percentage of outlying values from the replicate determinations

$x_m$  is the overall mean in %

$CV_r$  is the coefficient of the variation of the replicates

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

Sample type	Coning and quartering				Long pile				Riffle box				Handful sampling without coarse cut				Handful sampling with coarse cut			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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 <sup>a)</sup>								
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 <sup>b)</sup>	0,023 <sup>b)</sup>	16	0	0,34	6,6	16	0	0,34	2,4								

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)

Sample type	Coning and quartering				Long pile			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
1	16	0	20,00	0,45	16	0	19,77	0,37

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

Sample type	Coning and quartering				Long pile				Riffle box			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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)

Sample type	Coning and quartering				Long pile				Riffle box			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
3	16	0	5254	17	16	0	7973	16	16	0	6573	28
4	16	0	15727	17	16	0	23414	8	16	0	20251	15

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

Sample type	Coning and quartering				Long pile				Riffle box			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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 < 16 mm > 8 mm (%)

Sample type	Coning and quartering				Long pile				Riffle box			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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 < 8 mm > 5 mm (%)

Sample type	Coning and quartering				Long pile				Riffle box			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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 < 5 mm > 3 mm (%)

Sample type	Coning and quartering				Long pile				Riffle box			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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 < 3 mm > 2 mm (%)

Sample type	Coning and quartering				Long pile				Riffle box			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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 > (%)

Sample type	Coning and quartering				Long pile				Riffle box			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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 (%)

Sample type	Coning and quartering				Long pile				Handful sampling without coarse cut				Handful sampling with coarse cut			
	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$	<i>l</i>	<i>o</i>	$x_m$	$CV_r$
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

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