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Solid biofuels — Sampling

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National foreword

This British Standard is the UK implementation of EN 14778:2011. It supersedes DD CEN/TS 14778-1:2005 and DD CEN/TS 14778-2:2005 and DD CEN/TS 14779:2005 which are withdrawn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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Compliance with a British Standard cannot confer immunity from legal obligations.

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English Version

Solid biofuels - Sampling

Biocombustibles - Echantillonnage

Feste Biobrennstoffe - Probenahme

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

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Foreword

This document (EN 14778:2011) 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of an identical text or by endorsement, at the latest by December 2011, and conflicting national standards shall be withdrawn at the latest by December 2011.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 14778-1:2005, CEN/TS 14778-2:2005 and CEN/TS 14779:2005.

This document differs from CEN/TS 14778-1:2005, CEN/TS 14778-2:2005 and CEN/TS 14779:2005 mainly as follows:

a) CEN/TS 14778-1:2005, CEN/TS 14778-2:2005 and CEN/TS 14779:2005 are merged into one document and upgraded to EN 14778:2011;

b) results of interlaboratory tests are supplemented as informative annexes;

c) the whole document is restructured and editorially revised;

d) decision schemes are updated;

e) 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

Solid biofuels are a major source of renewable energy. European Standards are needed for production, trade and use of solid biofuels.

This European Standard can be used with regard to production, trading, controlling and analysis of solid biofuels in general. It is also useful for buyers of solid biofuels, regulators, controllers and laboratories.

This standard creates new working methods and practices for a broad fuel source, while for coal there are many years of experience for a single fuel source. This standard is based on the coal sampling methods, however due to the limited experience of biomass sampling, it is recognized that this standard will change in future versions when more experience is gained. What today is utilized as solid biofuels may change in the future.

1 Scope

This European Standard describes methods for preparing sampling plans and certificates and taking samples of solid biofuels, for example, from the place where the raw materials grow, from production plant, from deliveries e.g. lorry loads, or from stock. It includes both manual and mechanical methods, and is applicable to solid biofuels that are either:

— fine (particle size up to about 10 mm) and regularly-shaped particulate materials that can be sampled using a scoop or pipe, for example: sawdust, olive stones and wood pellets;

— coarse or irregularly-shaped particulate materials, particle sizes up to about 200 mm that can be sampled using a fork or shovel, for example: wood chips and nut shells, forest residue chips, and straw;

— baled materials for example: baled straw or grass;

— large pieces (particles sizes above 200 mm) which are either picked manually or automatically;

— vegetable waste, fibrous waste from virgin pulp production and from production of paper from pulp that has been dewatered;

— round wood.

It may be possible to use this standard on other solid biofuels. The methods described in this European Standard may be used, for example, when the samples are to be tested for moisture content, ash content, calorific value, bulk density, durability, particle size distribution, ash melting behaviour and chemical composition. The methods are not intended for obtaining 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 14780, *Solid biofuels — Sample preparation*

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
bias
systematic error that leads to the average value of a series of results being persistently higher or persistently lower than those that are obtained using a reference sampling method

3.2
combined sample
sample consisting of all the increments taken from a lot or sub-lot

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

3.3
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

3.4
increment
portion of fuel extracted in a single operation of the sampling device

3.5
laboratory sample
combined sample or a sub-sample of a combined sample for use in a laboratory

3.6
large stockpile
a stockpile with a capacity > 40 tonnes

3.7
lot
defined quantity of fuel for which the quality is to be determined

NOTE See also sub-lot.

3.8
mass-reduction
reduction of the mass of a sample or sub-sample

3.9
nominal top size
aperture size of the sieve used in the EN 15149 method for determining the particle size distribution of solid biofuels through which at least 95 % by mass of the material passes

3.10
overall precision
closeness of agreement between independent test results obtained under stipulated conditions; including sample preparation and sample analysis

NOTE A determination might be made with great precision and the standard deviation of a number of determinations on the same sub-lot might, therefore, be low; but such results are accurate only if they are free from bias.

3.11
particle size-reduction
reduction of the nominal top size of a sample or sub-sample

3.12
sample
quantity of material, representative of a larger quantity for which the quality is to be determined

3.13
small stockpile
stockpile with a capacity ≤ 40 tonnes

3.14
sub-lot
part of a lot for which a test result is required

3.15
sub-sample
portion of a sample

3.16
test portion
sub-sample of a laboratory sample consisting of the quantity of material required for a single execution of a test method

3.17
test-sample
laboratory sample after an appropriate preparation made by the laboratory

4 Symbols and abbreviations

d_{95} is nominal top size biofuel, in mm

d_i is the difference between individual pair members

m_{lot} is mass of the lot or sub-lot, tonnes

n is number of increments per (sub)-lot

n_{min} is minimum number of increments per (sub)-lot

n_P is the number of pairs (for estimating V_{PT})

n_{mp} is the maximum practicable number of increments per sub-lot

N_L, N_{SL} is the number of lots/sub-lots

P_L is the overall precision for the sampling, sample preparation and testing for the whole biofuel lot at 95 % confidence level

P_{SL} is similar to P_L but then for the sub-lot

s is the sample estimate of the population standard deviation

V_{SPT} is the total variance of the results for replicate samples

$Vol_{increment}$ is volume of an increment, litre

Vol_{min} is minimum volume of increment, litre

V_I is the primary increment variance

V_{PT} is the preparation and testing variance

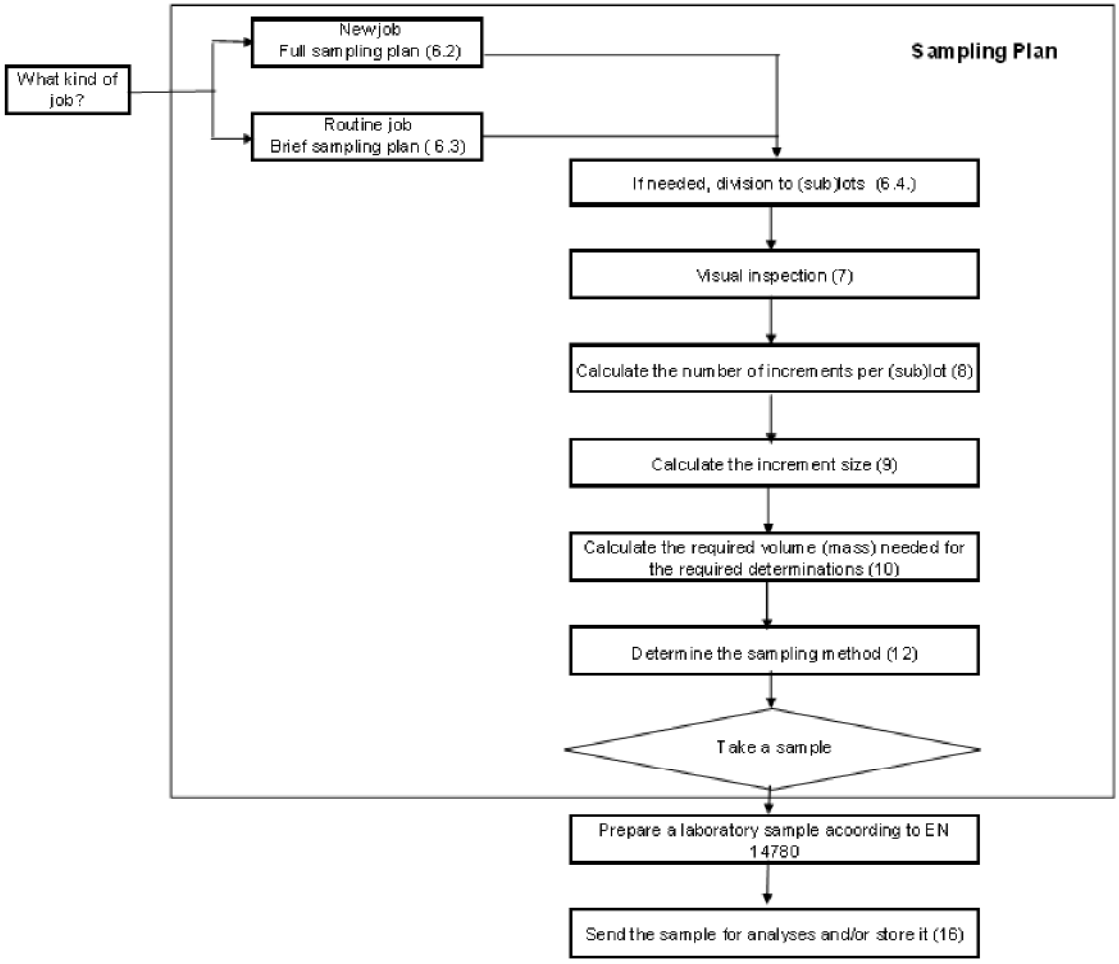
W is width of a sampling tool, mm

x_i is the value of the analysed parameter

5 Principle

The main principle of correct sampling is to obtain a representative sample (samples) from the whole lot concerned. Every particle in the lot or sub-lot to be represented by the sample should have an equal probability of being included in the sample. In order to do so a sampling plan is needed. Figure 1 shows the actions needed for the development of a sampling plan. When sampling is to be carried out according to the same plan repeatedly or continuously (e.g. daily), a full sampling plan shall be prepared according to 6.2 (it is necessary to do this only once). A brief sampling plan shall be prepared for routine use according to 6.3

(same type of sampling object or situation occasionally). In the case of a new material or supplier, the existing plan shall be checked and updated or a new full sampling plan shall be developed.



NOTE The numbers in Figure 1 refer to the clauses in this document.

Figure 1 — Procedure for sampling

6 Establishing a sampling scheme (sampling plan)

6.1 Principle

The sampler shall prepare a full sampling plan either by copying the forms presented in Annex A or by preparing his own forms or documents containing the appropriate items selected from those shown in Annex A. Each sampling plan shall be given a unique reference number or a code/name.

6.2 Full sampling plan

A Model Sampling Plan is presented in Annex A as forms that are to be completed by the sampler. Once completed these forms become sampling certificates.

6.3 Brief sampling plan

The sampling plan shall include the key elements:

— a reference to the full sampling plan (Annex A);

— the unique identification number of the sample;

— the date and time of sampling;

— the identity of the biofuel supplier;

— the identification number of the lot or the sub-lot.

Also consider including the following items:

— the name of the sampler;

— the mass or volume of the sub-lot or the lot;

— the identity of the carrier (transport company);

— storage information of the lot (like weather conditions, storage inside or outside)

— sampling technique, e.g. shovelling, cross stream cutter, hammer sampler, probe, stopped belt, etc.

— any other details that change from sample to sample.

6.4 Division of lots

The lot may be sampled as a whole, resulting in one sample, or divided into a number of sub-lots resulting in a possible sample from each. In case of manual sampling a lot may be sampled as a whole only when it has a maximum of 2 500 tonnes or as a series of sub-lots each to a maximum of 2 500 tonnes e.g. fuel dispatched or delivered over a period of time, a ship load, a train load, a wagon load, or fuel produced during a certain period, e.g. a shift. Such division into a number of sub-lots can be necessary to:

a) achieve the required precision (calculated by the procedure in 8.2),

b) maintain the integrity of the sample, e.g. avoiding bias that can result from the loss of moisture due to standing or changing of calorific value caused by biological activity,

c) create convenience when sampling lots over a long period, e.g. on a shift basis,

d) keep sample masses manageable, taking into account the maximum lifting capacity,

e) distinguish different components of a mixture of fuels, e.g. different biofuel types within one lot.

EXAMPLE Consider a power station that receives 140 lorry-loads of wood chips a month totalling 3 500 tonnes. In this example 4 sub-lots can be created where a sub-lot could be the quantity of fuel delivered in a week (about 35 lorry-loads).

NOTE In case of mechanical sampling e.g. from large shipments, the recommended maximum (sub) lot size should be decided by the parties involved.

7 Visual inspection

Visual inspection shall be used for the choice or verification of the classification of the solid biofuels. Based on the sampling plan, verification or selection of the sampling equipment and the sampling method shall also be made by visual inspection. If the biofuel consists of a mixture of substantially different materials, or if it contains impurities (such as soil or pieces of metal) this shall be reported in the sampling certificate. If the biofuel type or the quality of it is diverging strongly from the one expected, the sampler shall report without any delay to the appropriate party for further instructions.

8 Number of increments

8.1 General

In all methods of sampling, sampling preparation and analysis, errors are incurred and the experimental results obtained from such methods for any given parameter deviate from the true value of that parameter. As the true value cannot be known exactly, it is not possible to assess the accuracy of the experimental results, i.e. the closeness with which they agree with the true value. However, it is possible to make an estimate of the precision of the experimental results, i.e. the closeness with which the results of a series of experiments made on the same fuel agree among themselves.

It is possible to design a sampling scheme that, in principle, can achieve a desired level of precision with a material determined lower limit.

Precision is the closeness of agreement between the results obtained by applying the experimental procedure several times under prescribed conditions, and is a characteristic of the sampling scheme used and the variability of the biofuel being sampled. The smaller the random errors of the scheme, the more precise the scheme is. A commonly accepted index of precision is two times the sample estimate of the population standard deviation, and this index of precision is used throughout this European Standard.

If a large number of replicate samples are taken from a sub-lot of biofuel, prepared and analysed separately, the precision of a single observation, P, is given by Equation (1):

$$P = 2s = 2\sqrt{V_{SPT}} \tag{1}$$

where

s is the sample estimate of the population standard deviation;

V_{SPT} is the total variance of the results for replicate samples.

Here V_{SPT} is given by Equation (2):

$$V_{SPT} = \frac{V_I}{N_{SL} \cdot n} + \frac{V_{PT}}{N_{SL}} \tag{2}$$

Therefore the final overall precision, P_L, for the total quantity of biofuel:

$$P_L = 2\sqrt{\frac{V_I}{N_{SL} n} + \frac{V_{PT}}{N_{SL}}} \tag{3}$$

where

P_L is the overall precision for the sampling, sample preparation and testing for the whole biofuel lot at 95 % confidence level

V_I is the primary increment variance;

n is the number of increments per (sub)-lot;

N_{SL} is the number of sub-lots in the lot;

V_{PT} is the sample preparation and testing variance.

NOTE In the case that a total quantity of biofuel is divided into sub-lots, all sub-lots must be sampled. The number of sub-lots can be 1.

8.2 Primary increment variance (V_I)

The primary increment variance, V_I, depends upon the type and nominal top size of the fuel, the degree of pre-treatment and mixing, the absolute value of the parameter to be determined and the mass of increment taken. In general the increment variance (V_I) is different for the different parameters (in the same material) in practice. The calculation of the minimum number of increments should be based on different numbers on V_I, V_{PT} and P_L for each of the required parameters and the highest minimum number of increments should be selected (see also 8.5 for calculation of minimum number of increments).

The value of the primary increment variance, V_I, required for the minimum number of increments using Equation (6) or precision using Equation (3) can be obtained by either:

a) Determining it directly on the biofuel to be sampled by taking at least 30 increments spread over an entire lot of the same type of fuel and analysing each increment separately on the required parameters, preferably ash (dry basis) and total moisture.

$$V_I = \frac{1}{n-1}\left[\sum x_i^2 - \frac{\left(\sum x_i\right)^2}{n}\right] - V_{PT} \qquad (4)$$

where

x_i is the value of the analysed parameter;

See E.3 for an example for the determination of V_I.

b) Assuming values of V_I from similar materials or from previous characterization experience with similar fuel handling and sample preparation. The assumptions could preferably be verified afterwards if possible.

c) Assuming values of V_I listed in Annex D for the same type of materials. The assumptions could preferably be verified afterwards if possible.

8.3 Preparation and testing variance (V_{PT})

The value of the sample preparation and testing variance, V_{PT}, required for the calculation of the minimum number of increments using Equation (6) or precision using Equation (3) can be obtained by either:

a) Determining it directly on the fuel to be sampled by constituting at least 20 sub-samples spread over the entire lot of the same type of fuel. Each sub-sample is divided into two parts (constituting a pair) and prepared so that split portions of each sub-sample are taken at the first division stage. Each portion shall be prepared and tested for the parameters of interest, preferably ash (dry basis) and total moisture. The same analytical methods are applied as are used in routine operations. The difference between the two results shall be calculated for each pair and the preparation and testing variance, V_{PT}, can be calculated as follows:

$$V_{PT} = \frac{\sum d_i^{\,2}}{2n_P} \tag{5}$$

where

d_i is the difference between individual pair members

n_p is the number of pairs

See E.3 for an example for the determination of V_{PT}.

b) Assuming values of V_{PT} from similar materials or from previous characterization experience with similar fuel handling and sample preparation. The assumptions could preferably be verified afterwards if possible.

c) Assuming values of V_{PT} listed in Annex D for the same type of materials. The assumptions could preferably be verified afterwards if possible.

8.4 Overall precision (P_L)

The required overall precision for each relevant parameter on a lot should be agreed upon between parties concerned. In the absence of such an agreement, the values given in Tables D.1 to D.10 may be assumed. By keeping track of the results of the analyses, changes in the composition over time can be identified, which could be an indication to re-evaluate V_I and V_{PT}. This can be done using 8.2 and 8.3.

8.5 Calculation of number of increments per (sub)-lot

Determine the number of sub-lots required for practical reasons and then estimate the number of increments for a desired overall precision by transposing Equation (6) (rounded up):

$$n_{\min} = \frac{4V_I}{N_{SL}P_L^{\,2} - 4V_{PT}} \tag{6}$$

where

N_{SL} is the number of sub-lots in the lot, when the lot is not divided N_{SL}=1

n_{min} is the (minimum) number of increments per sub-lot, or per lot if the lot is not divided into sub-lots (N=1); if calculated if $n_{\min}$ is less than 10, it shall be set to $n_{\min}$=10 unless agreed upon otherwise

V_I is the primary increment variance

P_L is the overall precision for the sampling, sample preparation and testing for the whole biofuel lot at 95 % confidence level

V_{PT} is the preparation and testing variance

NOTE 1 Equation (3) is rewritten to yield Equation (6)

NOTE 2 Parties can agree on a different minimum number of increments, this can also be below 10. Parties should be aware of the possibility that extracting increments of extreme content will influence the final measured value.

Examples utilizing this equation are given in E.3.

A calculated value of $n_{\min}$ of infinity or a negative number indicates that the errors of preparation and testing are such that the required precision cannot be achieved with this number of sub-lots. In such cases, or if $n_{\min}$

is impracticably large, reduce the errors of sample preparation and testing, agree on a higher overall precision,or increase the number of sub-lots by one of the following means.

a) Choose a new number of sub-lots corresponding to a convenient sub-lot mass, recalculate n_{min} from Equation (6) and repeat this process until n_{min} is a practicable number.

b) Decide on the maximum practicable number of increments per sub-lot, n_{mp}, and calculate N_{SL} according to Equation (7):

$$N_{SL} = \frac{4(V_I + n_{mp}V_{PT})}{n_{mp}P_L^{\,2}} \quad (7)$$

Adjust N_{SL} upwards if necessary to a convenient number and recalculate n_{min}. A calculation example is found in E.3.

As described in 8.1 to 8.3, the tables in Annex D show reference or default values for V_I and V_{PT} when no other information is available. Tables D.1 to D.10 show empirical values for V_I and V_{PT} when no other information is available. It is recommended to measure the V_I and V_{pt} per type, group and/or supplier of biofuel.

The required overall precision on a lot should be agreed between the parties concerned. In the absence of such agreement, the values given in Tables D.1 to D.10 may be assumed. By keeping track of the results of the analyses, changes in the composition over time can be identified, which could be an indication to re-evaluate the measured V_I and V_{PT}.

9 Calculation of the size of increment

The minimum volume of the increment shall be:

$Vol_{incr} = 0{,}5$ for $d_{95} < 10$ (8)

$Vol_{incr} = 0{,}05 \cdot d_{95}$ for $d_{95} \geq 10$ (9)

where

Vol_{incr} is the minimum volume of the increment, litre;

d_{95} is the nominal top size, mm.

The sampler shall estimate and record the appropriate sampling tool. Take care, that samples are large enough for analyses.

10 Combined sample – Calculation of the volume of the combined sample

The sampler shall refer to 8.5 for the minimum number of increments n_{min} and the minimum volume of the individual increments Vol_{Incr} according to Clause 9 for the circumstances covered by the sampling plan.

The sampler shall consider what tests are to be done and calculate the required volume (mass) needed for the required determinations (Vol_{req}). In particular, the calculation shall take into account the need in some test methods for duplicate test portions, and for extra material to be available in case dubious results are obtained.

The calculated volume of the combined sample shall be of such a size that sufficient material is provided for all the tests to be performed, that is $Vol_{Combined\ Sample} > Vol_{req}$. Therefore the minimum sample volume should be estimated from the sampling plan. If the calculated volume is too small, the size or the number of increments

shall be increased. When the increments are reduced in volume before they are added to the combined sample, the volume Vol_{Incr} used in this calculation shall be the volume obtained after the reduction. The minimum increment volumes of Clause 9 should be used.

The sampler shall calculate the volume $Vol_{Combined\ Sample}$ required for the combined sample:

$$Vol_{CombinedSample} = n_{min} \times Vol_{incr} \quad (10)$$

where

$Vol_{Combined\ Sample}$ is the volume required for the combined sample, litre;

n_{min} is the minimum number of increments;

Vol_{incr} is the minimum volume of the individual increments, litre.

Table A.1 in Annex A can be used to record the results of the calculation. Annex C gives typical bulk densities of biofuels.

11 Sampling equipment

11.1 General

The equipment shall enable the sampler to take unbiased increments to provide a representative sample.

The opening of the sampling device shall be at least 2,5 times the nominal top size. The volume of the sampling device shall comply with the minimum required increment volume, Vol_{incr}, as described in Clause 9.

Sampling tools shall be robust, and be able to withstand physical force, wear and prolonged use without compromising functionality.

All moving parts should be accessible to inspection and maintenance.

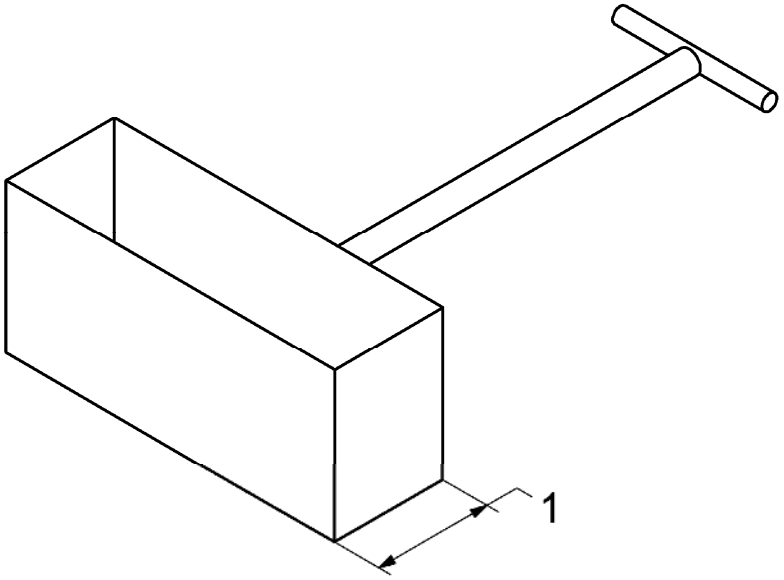
It is recommended that mechanical sampling equipment and manual sampling procedures shall be tested for bias after implementation, and this shall be repeated with a frequency that reflects the consequences of a possible bias. Bias testing of mechanical sampling equipment can be done according to ISO 13909-8, and manual sampling procedures according to the same principles.

The choice of sampling tool shall enable the sampler to extract the biofuel safely.

11.2 Equipment for manual sampling

11.2.1 Sampling box for falling-stream

The sampling box shall have a square or rectangular opening at the top. The opening *W* of the top of the sampling box shall be at least 2,5 times the nominal top size. The dimensions of the top opening of the sampling box shall be large enough so that the box cuts the whole of the stream to be sampled. The height of the sampling box shall be large enough to ensure that the box does not become full during sampling of the increment. The sampling box shall be provided with a handle or some other means of support (for instance mounted on rails) that enables the sampler to pass the box safely through the whole cross section of the falling stream of the biofuel to be sampled. Figure 2 shows an example of a sampling box.



Key

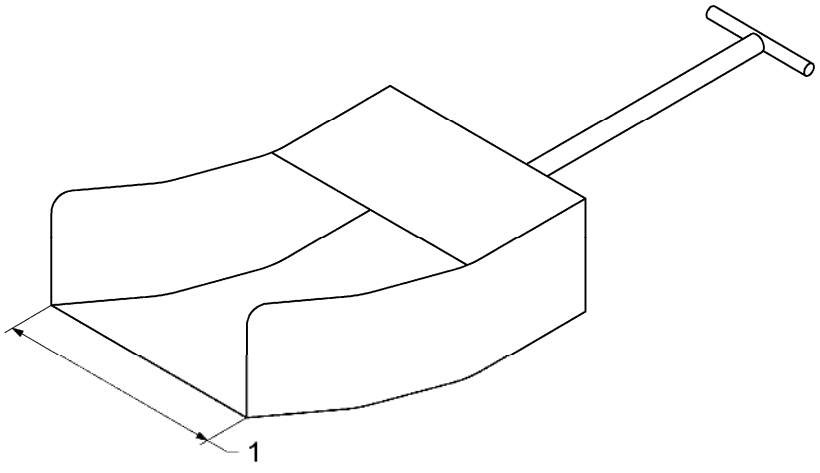
1 W is the width of the sampling box

NOTE For biofuel with large particle size, or high material flows, sampling boxes might become too big and heavy for manual sampling and mechanical sampling is recommended.

Figure 2 — Example of a sampling box

11.2.2 Scoops

A scoop can be designed as illustrated in Figure 3, complying with the general requirements for equipment design.



Key

1 W width and height of the scoop should be > 2,5 times nominal top size

Figure 3 — Example of a scoop

11.2.3 Shovels

A shovel can be designed as illustrated in Figure 4, complying with the general requirements for equipment design.

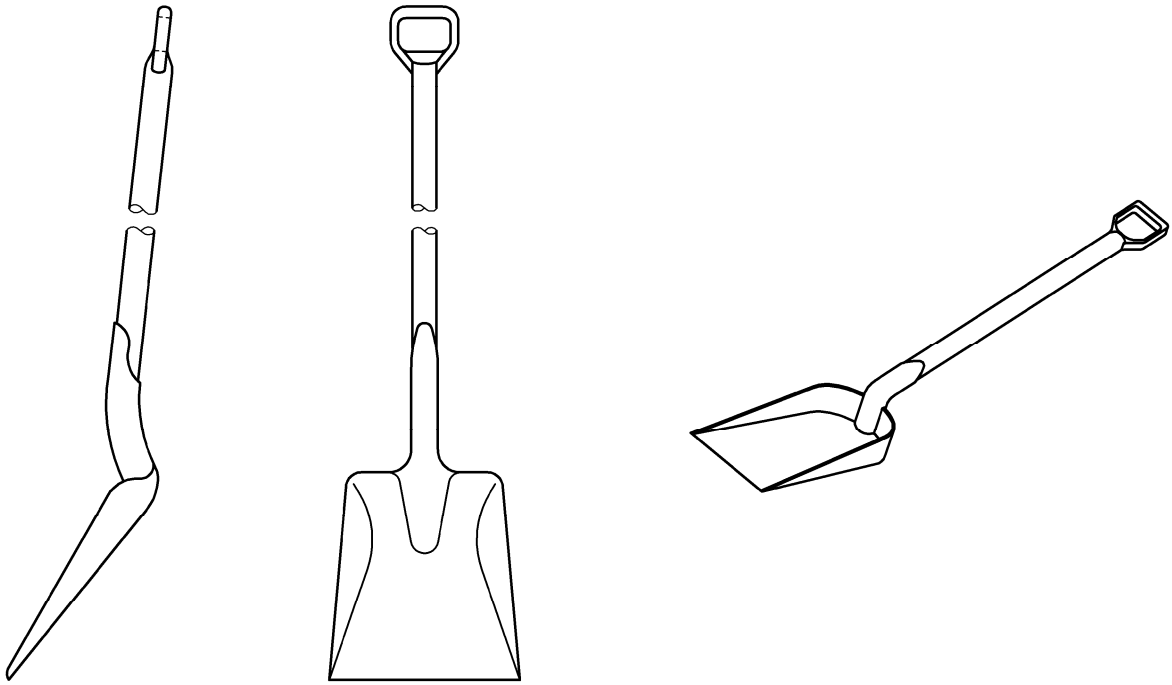


Figure 4 — Example of a shovel

11.2.4 Forks

When using a fork, see Figure 5, the smaller particles of the material being sampled will fall between the tines of the fork. The sampler shall check that the fork to be used for sampling a material has tines sufficiently close together to minimize the amount of particles falling between them. Any material losses will affect the quality of the sample and may lead to a biased result.

Figure 5 — Example of a fork

11.2.5 Grabs

Both an open type grab and a closed type grab may be used. Figure 6 contains drawings of examples of a grab.

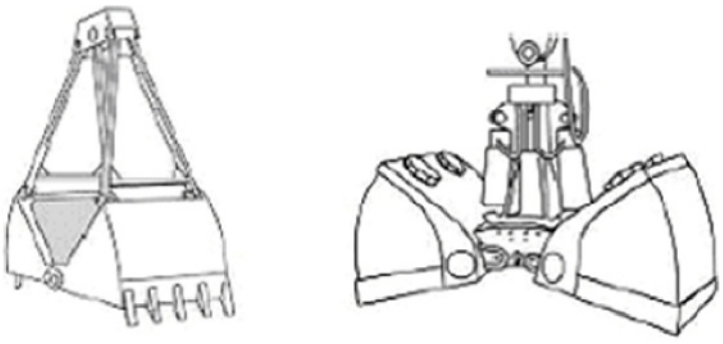
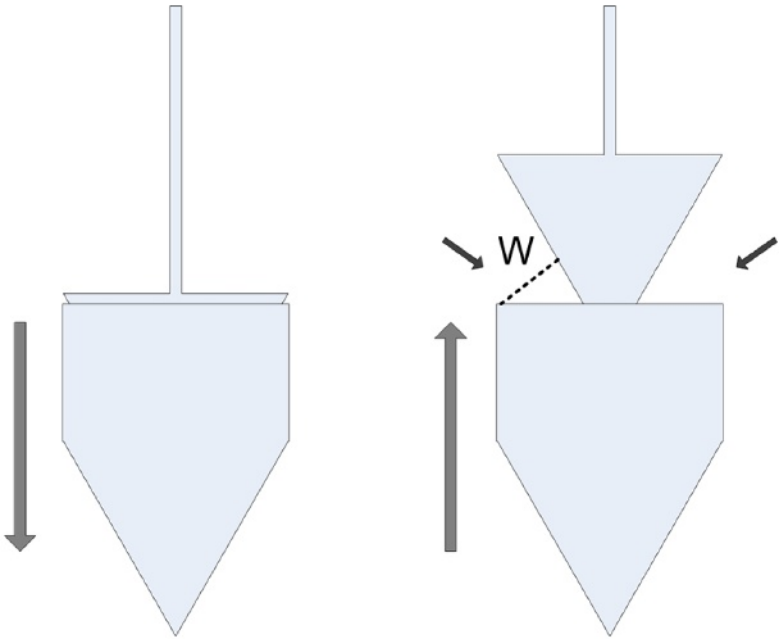


Figure 6 — Examples of grabs (open and closed type)

11.2.6 Probes (thieves)

Probes, see Figure 7, are most suitable for materials with a nominal top size less than 25 mm, but they can be designed and used also for larger particle sizes. The internal diameter of the probe shall be at least 2,5 times the nominal top size of the material to be sampled. The probe shall be designed so that it can be opened at an arbitrary depth inside the material to be sampled, and afterwards extracted without loss or gain of material.



Key

1 W aperture of the probe shall be > 2,5 times nominal top size

Figure 7 — Example of a probe

11.2.7 Pipes (spears)

The holes in the sampling pipe should be positioned as illustrated in Figure 8, and the pipe shall be constructed so that the holes open one after the other starting with the hole closest to the tip of the pipe. A sampling pipe is suitable only for sampling free flowing granular and uniform materials. The length of the pipe shall be sufficient to reach all the way into the container or heap. The opening of the holes in the pipe shall be at least 2,5 times the nominal top size of the material to be sampled.

Figure 8 — Example of a pipe (spear)

11.2.8 Frames

A sampling frame shall be used if increments are taken manually from a temporarily stopped conveyor. The sampling frame shall consist of two parallel metal plates with a distance between the two side plates of at least 2,5 times the nominal top size of the material to be sampled. The shape of the plates shall fit into the profile of the conveyor belt from which the sample is to be removed. The supports between the plates shall ensure a stable construction. A suitable tool shall be used to extract the material between the plates. Figure 9 is a schematic drawing of a sampling frame placed on a stopped conveyor belt.

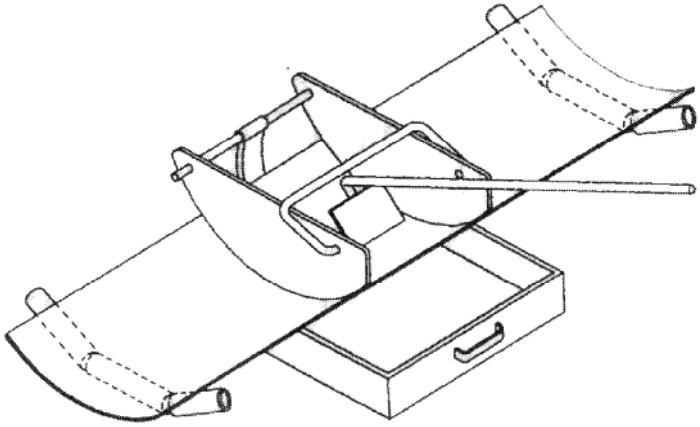


Figure 9 — Frame

11.2.9 Hooks

For sampling baled straw like material without taking apart the entire bale, a hook can be used, see Figure 10. The hook shall be constructed with a barb, so that it can be pushed into the bale and extract some straw when pulled back.

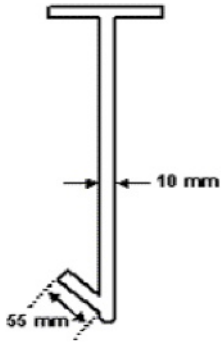


Figure 10 – Hook

11.2.10 Drills (augers)

A drill, see Figure 11, can be manually or mechanically driven. The centre should be encapsulated to prevent gaining or losing material that does not belong to the increment.

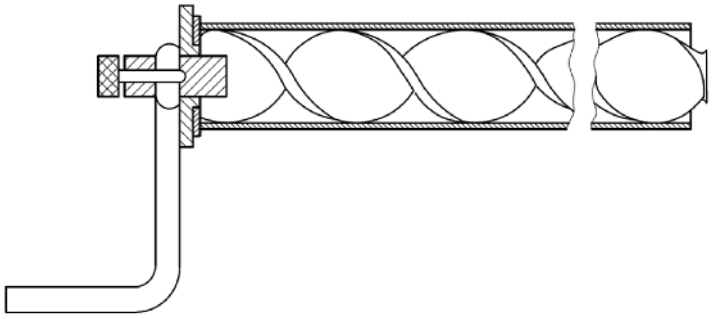


Figure 11 – A drill

NOTE The radius of the drill is the opening W of this sampling device.

11.3 Equipment for mechanical sampling

11.3.1 Falling-stream sampler

A falling stream sampler (cross stream cutter), see Figure 12, can be used for sampling materials that are free falling, for instance at the end of a conveyor belt. The device generally consists of a mechanically driven box, that moves at constant speed across (through) the falling material, with the opening (aperture) of the box at an angle as close to normal to the direction of the falling material as possible. The following design parameters shall be respected:

a) The cutter shall extract a complete cross section of the stream.

b) The cutter shall have parallel edges, ensuring even width of the cut across the stream.

c) The cutter shall move through the stream with constant velocity, avoiding slowing down as the cutter fills up.

d) The opening (aperture) of the cutter shall be minimum 2,5 times the nominal top particle size, to minimize the risk of blocking the flow into the cutter.

e) The cutter shall not be filled more than two thirds at maximum conveyor load.

f) The cutter edges shall be robust and able to withstand the force of the falling material during prolonged use.

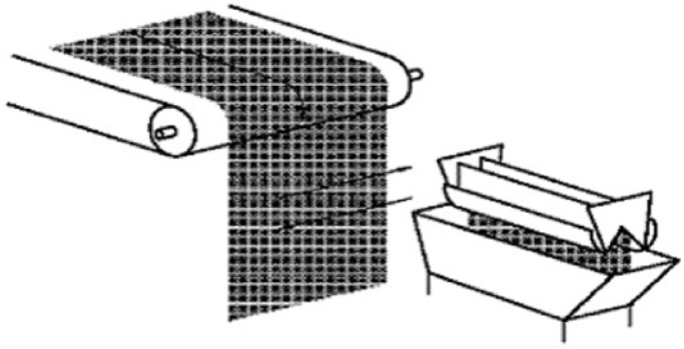


Figure 12 — Falling stream sampler

11.3.2 Cross-belt sampler

A cross-belt sampler (cutter) can be used for sampling materials from a moving conveyor belt. The equipment shall be designed so that it extracts a full cross cut of the material in the conveyor, it shall not only traverse over the full width of the belt, but it is important that the equipment extracts material all the way to the bottom of the belt. The sides (edges) of the cutter shall be parallel to ensure an even representation of all fractions of the flow.

The equipment shall be strong and durable, as it will have to retain the remaining flow of material while passing through the stream. For the same reason, normally no limitations are put on the speed of the cutter. However, it shall not be too high either, as too much material will be pushed off the belt from the leading edges of the cutter. The following design parameters shall be respected:

a) Cutter edges (sides) shall be parallel.

b) The cutter shall take a complete cross-section of the stream. The cut shall have an equal width across the belt (a “slice” with equal thickness across the belt width).

c) The velocity of the cutter through the material shall be uniform – avoid slowing down the cutter when it passes through the material.

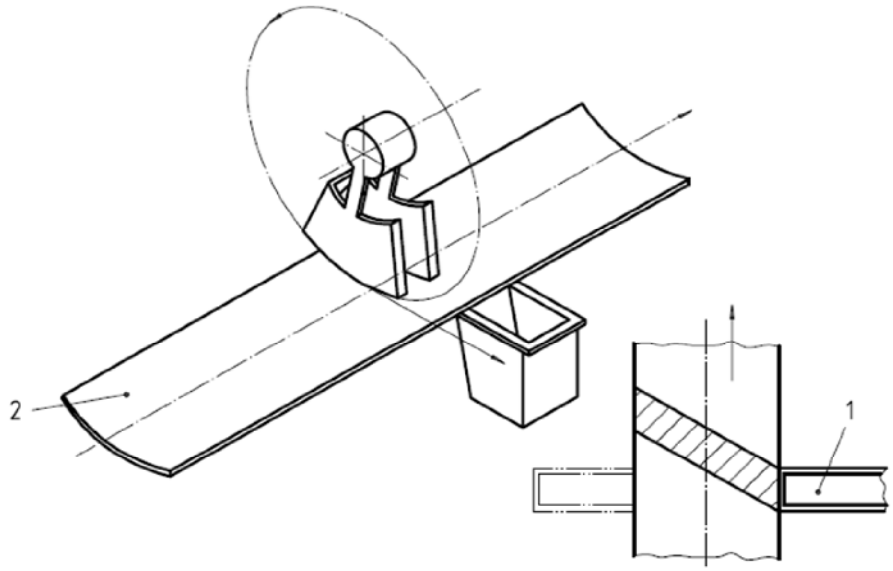
d) The aperture of the cutter shall be at least 2,5 times the nominal top size, to minimize the risk of blocking the flow into the cutter.

e) The capacity of the cutter shall be sufficient to hold all material from passing the stream at maximum conveyor load.

f) The bottom of the cutter can be fitted with blades, brushes or skirts to avoid damaging the belt and ensure extraction of particles close to the bottom of the belt. These shall be inspected regularly and replaced when close contact to the belt can no longer be ensured.

g) The cross-belt sample should yield with a single motion an increment size equal or larger than the minimum increment volume. This is dependent on the belt speed and the amount of material on the belt.

An example of a cross-belt cutter is shown in Figure 13. The right hand part of the figure illustrates the ideal extracted cut across the belt, with parallel sides.



Key

1 cutter

2 belt supported to maintain curvature

Figure 13 — Example of cross-belt sampler

NOTE Often cross-belt samplers have difficulties extracting ideal increments, as especially fines are left at the bottom of the belt and material at the leading edges of the cutter, that should ideally be in the increment, is not extracted. In general it is recommended to use falling stream cutters instead.

11.3.3 Mechanical probes

The principle of a mechanical probe is similar to a manual probe, see 11.2.6 (manual probes), but driven by pneumatics or a motor. Often mechanical probes are preferred, as it is difficult to manually drive a probe into a compact material.

11.3.4 Mechanical drills

The principle of a mechanical drill is similar to a manual drill, see 11.2.10 (manual drills).

12 Sampling in practice

12.1 General

It is difficult to take samples in a way that satisfies the principle of correct sampling, stating that all individual parts of the lot shall have an equal probability of becoming part of the final sample. The chance that this can be achieved when the material is stationary (for example, in a silo or stockpile, or in a lorry or ship) is low. It is easier when the material is moving (for example, on a conveyor belt, or being loaded into or unloaded from transport equipment). Hence sampling from moving materials is to be preferred wherever possible.

It is important to regularly ensure that the equipment in use is properly cleaned and maintained. If the equipment show signs of not functioning in accordance with the intended use, action shall be taken to test and repair or replace it.

The integrity of the sampled material shall be ensured, e.g. avoiding loss or gain of moisture, fines etc.

All sampling equipment shall be handled according to the described use, and it is important to ensure uniform extractions in repeated use.

The sampler shall always ensure that all extracted material is transferred from the sampling device to a sample container, without loss or gain.

If an increment or combined sample mass (volume) is too large to be handled or transported, the mass shall be reduced according to the methods described in EN 14780.

All personnel performing sampling shall be properly instructed or trained in the specific use of the device or method, and preferably understand the consequences of improper use of it and to avoid human influence on sample quality. All rules and legislation with regard to health and safety shall be respected at all times.

12.2 Methods for sampling stationary material

12.2.1 Sampling from small packages (< 50 kg)

When sampling a lot consisting of individual packages, a primary increment consists of an entire or partial package. Packages shall be chosen at random from the entire lot, making sure all packages have an equal probability of being selected. The number of selected packages (increments) shall be calculated according to 8.5 and Equation (6).

If the packages are transported on a conveyor, a lot can be defined as a certain time frame, a certain number of packages or similar. Increments shall then be chosen either systematically, randomly from defined strata, or completely at random, from the entire lot.

If the packages are stored, it is important to ensure that packages are chosen at random from the entire lot. If the packages are bundled and wrapped on pallets it may be necessary to minimize the number of opened pallets, but then the possible consequences of not respecting the principle of correct sampling shall be stated in the sampling report. Likewise, when access to all pallets is difficult, or impossible, this shall also be clearly stated in the sampling report.

12.2.2 Sampling from containers, lorries and wagons

An individual container, lorry or wagon load, may be regarded as the entire lot/sub-lot, or a part of the lot, see 6.4 (division of lots). If the lot consists of a single container, the increments shall be extracted from different

parts of the container, chosen at random. If the lot consists of more than one container, increments shall be extracted from either all, or a fraction of, the number of containers, dependent on the required number of increments. The fraction of the number of containers sampled shall be stated in the sampling report. It is not recommended to extract all required increments from the same container.

When sampling containers special care shall be taken to encompass the possible segregation of the material in the container, e.g. extract increments that cover the entire direction of segregation (a “drill-core” or selecting increments at different depths).

EXAMPLE 15 big bags of wood pellets (500 kg each) from the same supplier are considered a lot, when delivered to a small heating plant. The required number of increments is 10, but for practical reasons 12 increments is chosen. It is considered very likely that fines are found at the bottom part of the bags. The 12 increments are distributed as follows: 4 bags are selected at random, and 3 increments are extracted with a probe from top, middle and bottom in the bags, and the increments combined to form the final sample

When using a sampling pipe, see Figure 8 (11.2.7), insert the pipe into the material at an angle between 30º and 90º Insert the pipe completely before opening the sampling holes. Shaking the pipe can help to fill it. Take care when removing the increment from the pipe to collect all the fine particles. When using pipes with holes twisted around the perimeter of the pipe, it shall be used only at 90º.

Alternatively samples can be extracted from the freshly exposed surface during discharge, using a probe, auger or shovel. Care has to be taken to overcome the possible rolling segregation on sloped surfaces, especially for materials with wide particle size distributions or differences in physical characteristics. It is recommended to take as many (possibly smaller) increments as possible spread on the entire surface.

NOTE 1 It is always recommended if possible to sample when the biofuel is in motion, e.g. during loading or unloading.

NOTE 2 During the unloading of lorries it is recommended to check for foreign objects.

NOTE 3 Probes and pipes are only to be used for free flowing materials, e.g. pellets, dry olive kernels, etc.

NOTE 4 It shall always be stated in the sampling report when a sampling device can not reach the bottom of the container, with the risk of under representing a certain particle size fraction etc.

12.2.3 Sampling from stockpiles

12.2.3.1 General

Stockpiles shall preferentially be sampled during build-up or reclaiming as this ensures accessibility to all parts of the lot which in turn minimizes the effect of segregating materials. Only relatively small stockpiles (< 40 tonnes) may be sampled while stationary. The best practice for sampling large stationary stockpiles (> 40 tonnes) is described in Annex B.

A scoop, shovel, fork, auger, grab, probe or pipe shall be used to extract increments.

12.2.3.2 Sampling from stockpiles during build up or reclaiming

Increments shall be extracted either from the working face of the stockpile, or from the bucket of a front-end loader or grab or from a single, discrete load delivered to the stockpile before being pushed into the main stockpile. If a conveyor is used in stacking or reclaiming, or elsewhere in the material handling process, this is the optimal sampling point, and the methods for sampling moving material shall be used (see 12.3).

When sampling the working face of the pile, consider the possible (rolling) segregation on the surface. Ensure that a manual probe/auger or scoop is inserted at right angles to the surface, and that insertion of the probe/auger, is spread evenly over the entire surface of the pile. No portion of the increment should be lost during extraction of the scoop from the surface. Owing to the difficulty of insertion, a probe/auger shall be used only for fuels on which a full column of fuel can be extracted so that a representative increment is taken.

If sampling selected front-end loader buckets, grabs or individual discrete deliveries to the pile, these shall be discharged onto a hard, clean, and dry surface and then the fuel shall be sampled by either full-depth sampling or dividing of the load. Full depth sampling from the unloaded fuel can be done using a probe, pipe, auger or similar. Load division can be done by sequentially shovelling the material into smaller piles, randomly selecting a smaller pile for repeated division, until the required increment volume/mass is achieved. All smaller piles shall consist of a minimum of 10 shovelfuls. Division is laborious, but when the number of shovelfuls used to build every sub-pile is large (>30), chosen at random, and all material is divided, this method works very well and ensures against bias. If possible, a large riffle divider, rotating divider or similar is preferred, see EN 14780.

NOTE If the time between initiating sampling and analysis results in a bias (e.g. due to loss of moisture) the use of large equipment like front-end loaders, bulldozers can be used to create small piles.

12.2.3.3 Sampling from stationary stockpiles

To decide the height at which the increments are taken, the sampler shall visually divide the heap into three horizontal layers, and take a number of increments from each layer in proportion to the volume contained in each layer. The positions around the circumference of the heap from which the increments are taken shall be equally-spaced. A bucket loader may be used to dig into the heap to reach the sampling points. Care shall be taken when extracting increments at the lowest part of the heap, to avoid impurities, segregation etc. Figure 14 shows a possible arrangement of the sampling points on a heap.

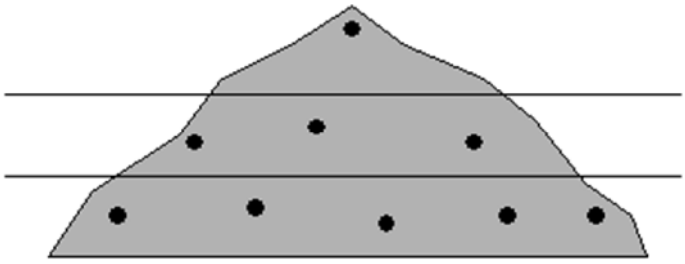


Figure 14 — Side view of an example of sampling points on a small stockpile

If there is any reason to suspect that the material in the stockpile is segregated then it is required that the material is moved (e.g. into a new stock pile) and that the increments are taken during the reclaiming or build up as described in 12.2.3.2.

12.2.4 Sampling from ships and barges

Sampling shall always be performed from a point where the biofuel is in motion, when possible. If it is necessary to sample from the hold of the ship, increments shall be extracted from a number of points distributed over various layers of the biofuel in the hold, which are exposed from time to time as the ship is loaded or unloaded.

A probe, auger, scoop, shovel or similar shall be used to extract increments. The aperture of the device shall comply with the description in 11.2.

Increments shall be spaced as evenly as possible over the surface. It is important to note that segregation during handling often results in the accumulation of lumps, e.g. near one or more walls of the hold depending on the handling system. This shall be considered when selecting increment extraction points.

When extracting increments, the probe, auger, scoop or similar shall be inserted at right angles to the surface of the fuel. No portion of the increment should be lost during extraction of the scoop from the surface. Owing to the difficulty of insertion, a probe/auger shall be used only for fuels on which a full column of fuel can be extracted so that a representative increment is taken.

12.2.5 Sampling from bales

An individual bale, may be regarded as the entire lot/sub-lot, or a part of the lot, see 6.4 (division of lots). If the lot consists of a single bale, the increments shall be extracted from different parts of the bale, chosen at random. If the lot consists of more than one bale, increments shall be extracted from either all, or a fraction of the number of bales, dependent on the required number of increments. The fraction of the number of bales sampled shall be stated in the sampling report. It is not recommended to extract all required increments from the same bale.

When sampling bales, special care shall be taken to encompass the possibility of uneven distribution of especially moisture and fines.

A minimum of two increments (e.g. drill cores) shall be taken from different sides (preferably opposite sides) of the bale and to such a depth that the increments taken will represent in a correct ratio the moisture/fines distribution in the bale. If the bales seem to be uneven by quality, the number of increments shall be increased and they shall be spread representatively around the bale.

An increment consisting of a drill core traversing the entire bale across the most likely direction of moisture/fines distribution is preferred. Alternatively a hook can be used to pull straw from as many different parts of the bale that is practically possible to form an increment, attempting to represent any uneven distribution of e.g. moisture.

NOTE Fines are difficult to sample correctly using a hook.

12.3 Methods for sampling moving material

12.3.1 General

The lot or sub-lot shall be defined as all the material in the container (ship hold, wagon, etc.) that the sample is to represent, or in the case of continuous production or conveying, all the material passing the sampling point during a specified time interval. An interval can also be defined in terms of mass or volume.

Increments shall be distributed over the entire lot according to one of two scenarios:

— Systematic increment extraction: Increments are taken at fixed time, mass or volume intervals evenly spread over the entire lot.

— Stratified random increment extraction: The lot is divided into equal strata (time, weight or volume) and an increment is taken from each at random. This approach is preferred when periodicities or cycles are expected in the process, to avoid taking increments at a frequency (or multiple thereof) coinciding with the frequency of the cycle.

12.3.2 Sampling from falling streams

12.3.2.1 Mechanical sampling

Falling stream samplers (see Figure 12) are often installed at the end of a conveyor belt or similar. When the material is falling into the cutter, it is important to avoid bouncing and filling the cutter completely. When the cutter has travelled through the stream, it shall be emptied mechanically, and no material should be lost during this operation. Often the bottom of the cutter opens, and the material simply falls into a temporary holding compartment. When the compartment is full, the material can be divided mechanically, using for instance a rotating plate divider, rotating disc divider, rotating nozzle divider or similar. Care shall be taken to avoid damaging the material during the division process. While the cutter is in the park position, no material (e.g. dust) shall fall inside it, creating a biased next increment. It is important to inspect the cutter regularly during operation, to avoid problems with clogging etc. Preferentially it shall be possible to change the frequency of the cutter, to allow different materials, qualities etc. to be sampled at the same plant. To avoid biased results care shall be taken about the complete emptying of the sampling device between different biofuels and also between increments of the same biofuel.

12.3.2.2 Manual sampling

Usually manual sampling is only suited for low mass flows.

Sampling shall be carried out using a sampling box (see Figure 2) or other suitable equipment that is passed through the stream of falling material so that it cuts the whole cross section of the falling stream.

Sampling from falling streams can also be done by taking the increments from randomly selected points of the stream. In these cases careful attention shall be put on possible segregation of fuel flow. If it is not possible to take the increment covering the entire stream, it is recommended to take an increased number of increments.

12.3.3 Sampling from conveyor belts

12.3.3.1 Mechanical sampling

Cross stream cutters (see Figure 13) are often installed on a conveyor belt. When the material is flowing into the cutter, it is important to avoid bouncing and filling the cutter completely. When the cutter has travelled through the stream, it shall be emptied mechanically, and no material should be lost during this operation. The material from the cutter often falls into a temporary holding compartment. When the compartment is full, the material can be divided mechanically, using for instance a rotating plate divider, rotating disc divider, rotating nozzle divider or similar. Care shall be taken to avoid damaging the material during the division process. While the cutter is in the park position, no material (e.g. dust) shall gather inside it, creating a biased next increment. It is important to inspect the cutter regularly during operation, to avoid problems with clogging etc. Preferentially it shall be possible to change the frequency of the cutter, to allow different materials, qualities etc. to be sampled at the same plant. To avoid biased results, care shall be taken about the complete emptying of the sampling device between different biofuels and also between increments of the same biofuel.

12.3.3.2 Manual sampling

A sampling frame (see Figure 9) shall be used to separate the material to be taken as an increment. The frame shall be placed on top of the material on the stopped conveyor belt and forced to the bottom. All the material contained within the sampling frame shall be taken as the increment. If material is trapped under the edges of the frame, the material trapped under one edge shall be included in the increment and the material trapped under the other edge shall be excluded from the increment.

12.3.4 Sampling from bucket conveyors, drag conveyors, bucket loaders or grabs

A number of bucketfuls, grabfuls or compartments of the drag conveyor shall be selected for sampling during the discharge of the lot or sub-lot.

Either take all of a selected bucketful, grabful or a compartment of the drag conveyor as an increment, or take a smaller representative increment by:

a) Emptying the entire contents onto a clean, hard surface, and take an increment from the tipped material according to the method described in section 12.2.3.2 (stockpiles, during build up/ reclaiming).

b) Taking an increment in the bucket, drag conveyor etc. by digging into the material as many times as feasible, and at different depths, to form a combined increment. This is the case if the material cannot be emptied from the bucket etc.

12.4 Sampling of roundwood

12.4.1 General method

Select the appropriate number of logs according to the required number of increments (see 8.5). Cut one slice (disc), with an individual thickness of approximately 3 cm to 5 cm from the centre of each log. Each slice has to be considered as an increment. If the moisture content has to be measured, cut at least three slices (discs),

spread evenly along the length of each log, avoiding the end parts (0,20 m). Alternatively for moisture content samples the method described in 12.4.2 can be applied. A best effort shall be made to ensure that the entire lot is represented when selecting where to cut for the extraction of slices/discs.

NOTE Moisture can systematically change along the length of round wood logs due to drying from the end surface.

It is recommended to use a power saw to avoid or minimize loss of moisture and contamination with lubricants etc. If using a chainsaw, it should preferably be operated without the use of chain-oil. Thus extreme care shall be taken to avoid heating of the chain, and the possible personal hazard from such.

If necessary the slices (discs) are subsequently carefully divided into smaller pieces with a hand axe/chisel and hammer. If mass reduction is necessary, the reduced part of the disc should represent heartwood, sapwood and eventually bark in the same proportions as the whole disc. This can be achieved by reducing a round disc into circular sectors.

Further sample preparation (cutting, crushing and dividing) at the laboratory shall be in accordance with EN 14780. Care shall be taken to avoid loss of moisture at all stages.

12.4.2 Method for fast moisture-content determination

Sampling of roundwood is to be done immediately before or after the determination of the weight (as received) of the delivery.

The number of cuts (increments) has to be calculated according to Equation (6).

The log sampling has to be done with the help of a chainsaw or a cutter with sawdust collector, taking into account that the circular saw is sharpened or the chainsaw has a correct tension. If a cutter is used, the shape of the blade should allow cuts to be made in the form of a circular sector.

Sampling with a chainsaw is done by cutting the logs in semi section until the core of the log or by cutting the whole log through in two pieces and collecting the sawdust. If the average diameter of the logs in the lot is bigger than 30 cm, sampling can be undertaken by cutting circular sectors with a chain saw.

Cutting with a cutter is done by piercing with the top of the cutter until it reaches the core of the log.

For long logs (≥ 2m) the cutting has to be done with a distance of at least 50 cm from the ends of the log and in the case of short logs (< 2m) at least 15 cm from the ends.

If the collector is filled with sawdust (particularly with logs of large dimensions) the collector shall be emptied or replaced by a second collector. All the collected sawdust from one delivery lot has to be mixed and homogenised before taking the analysis sample.

The whole quantity of sawdust produced by sampling one delivery lot has to be preserved in airproofed containers protected against outside influences. The samples have to be labelled and the sampling information has to be documented.

NOTE 1 For safety reasons all necessary safety precautions for the work with chainsaws, like security clothing and chainsaws with safety features shall be implemented.

NOTE 2 Large snow loads, ice or dirt have to be removed before sampling.

NOTE 3 Moisture content is not uniform in the log. For example Heartwood usually contains less moisture than sapwood. In the case of fresh coniferous wood the moisture content of the heartwood is about 35 % and of sapwood around 55 %. In the case of broad-leaf wood the difference is significantly smaller. It is also possible that the Heartwood has a higher moisture content than the sapwood, for example in the case of the poplar.

13 Sample generation

13.1 Combined samples and laboratory samples

One of the following options shall be used:

a) All the increments are placed directly into one container to form a combined sample, which is sent to the laboratory. In this case the combined sample is also the laboratory sample.

b) The increments are mixed together to form a combined sample, which is then divided and prepared as described in EN 14780.

c) Each increment is placed in a separate container, and sent to the laboratory. The laboratory combines the increments to form the laboratory sample.

NOTE It is recommended that the samples are mixed prior to division preferably on a dry and dust free sampling site.

14 Performance characteristics

The overall precision of sampling of the lot (characterizes the precision for the lot, either as a specification which has to be reached or for the result which has been obtained) can be calculated using the calculation as described in Clause 8, for each sampling scheme individually. The formula below is used:

$$P_L = 2\sqrt{\frac{V_I}{N_{SL}n} + \frac{V_{PT}}{N_{SL}}} \tag{11}$$

Empirical values for specific materials for V_i and V_{PT} can be found in Annex D. These data are based on validation research partly done within the BioNorm research project.

15 Handling and storage of samples

15.1 Packaging, storing and transport of samples

Samples shall be placed in airtight plastic containers or bags. Depending on the parameter to be determined, extra care should be taken for:

a) In every case the sample can be placed in air-tight packages such as plastic buckets (with lids) or plastic bags (to be closed). If moisture is to be determined, the weight of the packaging after removing of the sample shall be determined before and after drying (as moisture may be absorbed on the inside of the packaging).

b) As an alternative for some types of biofuels which can re-absorb condensed moisture (e.g. sawdust), it is permissible that the bag or container together with the sample it contains is shaken so that the condensed moisture is fully re-absorbed into the sample.

c) When only particle size distribution is to be determined, the sample can be placed in a box or other convenient packaging.

d) If transparent packaging is used, the sample shall be kept away from direct sunlight.

e) When it is necessary to guard against aging of the sample, the sample container shall be sealed.

f) When it is necessary to minimise biological activity, the sample shall be submitted for testing within 24 h or the sample can be stored in a refrigerator or cold room at 4 °C or below and analysed as soon as

possible, in most cases after no longer than one week. Check the sample at periodic intervals for the presence of funghi and other symptoms of increased biological activities. In this case the sample has to be treated immediately. Alternatively, the sample shall be air-dried as described in EN 14780 or deep-frozen (< -18 °C). If the moisture content is to be determined, the weight loss caused by air-drying shall be recorded and submitted together with the air-dry sample.

g) If little or no biological activity occurs the sample should be stored in a dry cool area for no longer than 6 months.

h) Integrity of the sample should always be safeguarded during storage.

15.2 Identification / labelling

The container shall carry a label showing:

— the unique identification number of the sample;

— the date and time of sampling;

— the identification number or code of the lot or sub-lot number.

And when necessary:

— the type of biofuel;

— the reference number of the sampling plan;

— the name of the sampler.

16 Sampling certificates

A sampling certificate shall:

— either contain all the information required by the full sampling plan,

— or contain all the information required by the brief sampling plan.

When a unique delivery ticket is used, also as a sampling plan, a brief sampling plan should be either included or added to it by the supplier.

Annex A
(informative)

Model Sampling Plan and Sampling Certificate

Table A.1 — Example of a full sampling plan

Sampling plan reference number	
Unique sample identification number	Date & time
Name of sampler	Mobile
E-mail	Telephone
Address	Fax
Name of client	Mobile
E-mail	Telephone
Address	Fax
Lot or sub-lot identification number	Packaging of the laboratory sample Airtight plastic container Other:
Product	Visual inspection remarks:
Trade name	
Biofuel supplier	
Agreed overall precision on the lot	
Mass or volume of sub-lot ton or m³	
Approximate nominal top size mm	
Mass of laboratory sample and container kg	
Weather conditions (e.g. cloudy, strong wind, approx. 18 °C)	

Lot type (silo, container, pile, lorry, barge, etc.)	Indoor ☐ Outdoor ☐ Uncovered ☐ Covered ☐
Method of sampling:(stationary/moving, cross-belt, scoop, etc.)	
Name and address of supplier	
Name and address of carrier	
Name and address of laboratory	

Date	Sampling equipment used
Aim of sampling	

Property	EN standard	Mass required [kg]	
Moisture Particle size distribution			
Bulk density			Description of sampling point: (pictures are recommended)
Particle density			
Mechanical durability			
Ash			
Calorific value			
Sulphur and Chlorine			Procedure for selecting sub-lots from lots for sampling
CHN			

Others			

Total mass required kg	Requirements according to EN 14778
Bulk density estimated kg/litre	Minimum number of increments (n_{min})
Total volume required for tests (Vreq) litre	Minimum volume, one increment (Vol_{incr}) litre
If total volume required (Vol_{req}) exceeds the calculated volume of combined sample (Vol_{com}), then increase the number of increments or the volume of the increments:	Volume of combined sample (Vol_{com}) litre
Actual number of increments(n_{act}), larger than Vol_{req}/ Vol_{incr}	Method of preparing the laboratory sample from the combined sample:
Actual volume of the combined sample (n_{act} x Vol_{inc}) litre	Volume of the laboratory sample (Vol_{lab}) litre

Annex B
(informative)

Sampling from large stockpiles

B.1 Initial assessment of the stockpile

The sampler shall inspect the stockpile visually. If the stockpile appears to contain significantly deviating areas, sub-lots of each area have to be made. The sampler shall sample each sub-lot and make a note of the estimated proportions of each area on the sampling certificate.

The sampler shall establish how the stockpile was formed as this can cause the stockpile to be heterogeneous. For example, if material is allowed to fall from the end of a conveyor to form a stockpile in the form of a conical heap, coarser particles tend to collect at the outside and at the base of the stockpile, and finer particles collect in the interior of the stockpile. However, if such a stockpile is exposed to the wind, then finer particles will be carried by the wind to the downwind side of the stockpile. If a stockpile is built over a lengthy time period, the inaccessible material on the inside may be totally different from the accessible material on the outside or at the different end of a long stockpile.

B.2 Taking samples

Take the increments manually using a scoop, shovel or fork or pipe. If segregation is expected to occur during sampling, drive a board or metal plate horizontally into the heap and withdraw the increment immediately underneath. The increments shall be taken mass proportionally if possible.

Holes or ditches should be dug using e.g. a front end bucket loader. If possible, these holes or ditches should be dug from the top of the stockpile and down to 4/5 of the height of the stockpile. If this is not possible, the holes or ditches should be dug from the sides of the stockpile (evenly distributed). From the excavated material, increments can be taken using a scoop, shovel or fork.

As alternative mechanical probes and augers can be used for sampling in the same way as for railway wagons and ships, see 12.2.2 and 12.2.4.

B.3 Marking, packaging and dispatch of samples

See Clause 15.

B.4 Certificate of sampling

The sampler shall prepare a sampling certificate according to Clause 16. He shall report that the sampling was undertaken on a large stationary stockpile and any other reasons why the sample may not be representative of the stockpile on the sampling certificate.

Annex C
(informative)

Bulk densities of biofuels

Typical bulk densities for solid biofuels are given in Table C.1. These values can be used in case no other information on bulk density is available.

Table C.1 — Typical bulk densities of biofuels

Biofuel	Bulk density kg/m^3
Pellets	550 – 700
Briquettes	500 – 650
Fuel powder	150 – 250
Dry fuel powder	100 – 150
Bark	250 – 400
Sawdust	250 – 380
Shavings	80 – 170
Wood chips	250 – 400
Straw bales	130 – 180
Chopped straw	80 – 120
Reed canary grass, round bales	~165
Reed canary grass, square bales	~125
Reed canary grass, chopped	30 – 80
Miscanthus chopped	100 – 120

Annex D
(informative)

Empirical values for P_L, V_I and V_{PT}

D.1 Introduction

In Clause 8 the determination of P_L, V_L, V_{PT} and n_{min} are described. If values cannot be determined in this procedure the values given in this annex can be assumed initially. The assumptions could preferably be verified afterwards if possible. The required overall precision on a lot should be agreed between the parties concerned. In the absence of such agreement, the values given in Tables D.1 to D.10 may be assumed. The number of increments per sub-lot can be calculated depending on the number of sub-lots and the values of V_I and V_{PT}. See Tables E.1 to E.10 for the numbers of increments per sub-lot calculated with these empirical values.

D.2 Large shipment of wood pellets from different sources

An investigation has been performed into the increment variance and the preparation and test variance of a large shipment of heterogeneous wood pellets. An empirical value for V_I and V_{PT} has been calculated with these values (see F.4). Tables D.1 to D.10 shows empirical values for V_I and V_{PT} when no other information is available.

Table D.1 — Mixed wood pellets 6-8 mm from different sources

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Total moisture	0,20 w-%	0,34 w-%^{2}	0,002 w-%^{2}
Ash (db)	0,20 w-%	0,53 w-%^{2}	0,015 w-%^{2}
Gross Calorific value (db)	0,100 MJ/kg	0,038 (MJ/kg)^2	0,0061 (MJ/kg)^2

The number of increments per sub-lot can be calculated depending on the number of sub-lots with the above values. See Table E.1.

Table D.2 — Wood pellets (6 mm) produced from one production site with a constant quality of raw materials

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Total moisture	0,20 w-%	0,025 w-%^{2}	0,014 w-%^{2}
Ash (db)	0,20 w-%	0,0008 w-%^{2}	0,0071 w-%^{2}
Mechanical durability	0,20 w-%	0,005 w-%^{2}	0,0016 w-%^{2}

Table D.3.1 — Wood pellets (8 mm) from stemwood from one production site

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Total moisture	0,20 w-%	1,35 w-%^{2}	0,002 w-%^{2}
Ash (db)	0,20 w-%	0,0004 w-%^{2}	0,0003 w-%^{2}
Particle size distribution	0,1 w-%	0,045 w-%^{2}	0,001 w-%^{2}

Table D.3.2 — Mixed woodpellets (8 mm) from one production site with changing raw material quality

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Total moisture	0,20 w-%	0,958 w-%^{2}	0,003 w-%^{2}
Ash (db)	0,20 w-%	0,0054 w-%^{2}	0,0003 w-%^{2}
Mechanical durability	0,20 w-%	0,208 w-%^{2}	0,0061 w-%^{2}

Table D.4 — Woodchips, including bark with a nominal top size of 16 mm

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Total moisture	1,00 w-%	12,5 w-%^{2}	0,059 w-%^{2}
As (db)	0,10 w-%	0,05 w-%^{2}	0,0004 w-%^{2}
Particle size distribution	2 w-%	25,4 w-%^{2}	0,86 w-%^{2}

Table D.5 — Sawdust from conifer

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Total moisture	1,00 w-%	6,0 w-%^{2}	0,06 w-%^{2}
Ash (db)	0,10 w-%	0,003 w-%^{2}	0,0006 w-%^{2}
Particle size distribution	2 w-%	14 w-%^{2}	1,6 w-%^{2}

Table D.6 — Bark from Scots pine with a nominal top size of 100 mm

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Total moisture	1,00 w-%	8,00 w-%^{2}	0,68 w-%^{2}
Ash (db)	0,15 w-%	0,019 w-%^{2}	0,015 w-%^{2}
Gross Calorific value (db)	0,100 MJ/kg	0,081 (MJ/kg)^2	0,0042 (MJ/kg)^2

Table D.7 — Logging residue from conifer, from conifer with nominal top size of 64 mm

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Total moisture	1,50 w-%	10 w-%^{2}	0,73 w-%^{2}
Ash (db)	1 w-%	1,15 w-%^{2}	0,37 w-%^{2}
Particle size distribution	5 w-%	54 w-%^{2}	25,6 w-%^{2}

Table D.8 — Straw from wheat in bales

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Total moisture	2,5 w-%	100 w-%^{2}	3,06 w-%^{2}
Ash (db)	0,5 w-%	1,17 w-%^{2}	0,06 w-%^{2}
Chlorine	0,02 w-%	0,01 w-%^{2}	0,00005 w-%^{2}

Table D.9 — Olive residue, typical Mediterranean materials with a nominal top size of 3 mm

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Moisture	0,4 w-%	0,23 w-%^{2}	0,029 w-%^{2}
Ash (db)	1w-%	1,490 w-%^{2}	0,527 w-%^{2}
Al	150 ppm	22900 ppm^2	15216 ppm^2
Ca	1500 ppm	1082992 ppm^2	1260426 ppm^2
Mg	500 ppm	30473 ppm^2	157282 ppm^2
Na	50 ppm	4029 ppm^2	1733 ppm^2
P	50 ppm	4010 ppm^2	1342 ppm^2
Si	2000 ppm	3562823 ppm^2	1675212 ppm^2
K	1000 ppm	622058 ppm^2	269210 ppm^2
N	0,1 w-%	0,01 w-%^{2}	0,007 w-%^{2}

Table D.10 — Grape residue, typical Mediterranean materials with a nominal top size of 16 mm

Parameter	Suggested precision (P_L)	Increment variance (V_I)	Preparation and test Variance (V_{PT})
Moisture	1,5 w-%	6,8 w-%^{2}	1,88 w-%^{2}
Ash (db)	1 w-%	0,720 w-%^{2}	0,202 w-%^{2}
Al	150 ppm	12023 ppm^2	5468 ppm^2
Ca	3500 ppm	11221543 ppm^2	5098208 ppm^2
Mg	200 ppm	22390 ppm^2	10347 ppm^2
Na	50 ppm	11982 ppm^2	551 ppm^2
P	200 ppm	71904 ppm^2	19991 ppm^2
Si	1000 ppm	164663 ppm^2	374276 ppm^2
K	1500 ppm	3383501 ppm^2	1241161 ppm^2
N	0,1 w-%	0,009 w-%^{2}	0,0045 w-%^{2}

The number of increments per sub-lot can be calculated depending on the number of sub-lots with the above values, see the tables of Annex E.

Annex E
(informative)

Guidelines for the number of increments to be taken

E.1 General

Clause 8 describes how the number of increments should be calculated. It is preferred that measured values for the different types of biofuel are used. When no values are available the data collected in either the BioNorm, Bionorm2, and investigation on large shipments of wood pellets can be used. The background to the experiments is described in Annex F.

E.2 Estimation of the number of increments from empirical values

Below are tables with estimated numbers of increments per sub-lots, depending on the number of sub-lots based on the initial and empirical values as described in Annex F (F.2 for: Tables E.2 to E.10 and F.3 for Table E.1). All values of 10 indicate that the calculated minimum increment number is 10 or less than 10 (see 8.5).

NOTE These tables sometimes show that for a small number of sub-lots a large amount of increments are needed which is impracticable. In some cells "too low P_L" is given, indicating that any amount of increments would not yield the required precision. More sub-lots would then be needed. Or alternatively the overall precision (P_L) can be changed if agreed upon by the involved parties.

Table E.1 — Number of increments per (sub)-lot for wood pellets 6-8 mm from different sources (see F.3)

	number of increments (n) per (sub)-lot		
number of sub-lots (N_{SL})	Total moisture P_L=0,2 w-%	Ash P_L=0,2 w-%	GCV (db) P_L=0,1 MJ/kg
1	43	Too low P_L	Too low P_L
2	19	106	Too low P_L
3	12	35	27
4	10	21	10
5	10	15	10
6	10	12	10
7	10	10	10
8	10	10	10
9	10	10	10
10	10	10	10
NOTE GCV: Gross Caloric Value (db): dry basis			

Table E.2 — Number of increments per (sub)-lot for wood pellets (6 mm) produced from one production site with a constant quality of raw materials

	Number of increments (n) per (sub)-lot		
Number of sub-lots (N_{SL})	Total moisture P_L=0,2 w-%	Ash P_L=0,2 w-%	Mechanical durability P_L=0,2 %
1	Too low P_L	10	10
2	10	10	10
3	10	10	10
4	10	10	10
5	10	10	10
NOTE Samples were taken directly after production line, and from one production unit with constant incoming raw materials and consisting of up to 3 months production.			

Table E.3 — Number of increments per (sub)-lot for wood pellets from stem wood (8 mm) from one production site with changing raw material quality

	Number of increments (n) per (sub)-lot			
number of sub-lots (N_{SL})	Total moisture P_L=0,2 w-%	Ash P_L=0,2 w-%	Mechanical durability P_L=0,2 %	Particle size distribution P_L=0,1 w-%
1	169	10	53	30
2	75	10	15	11
3	48	10	10	10
4	36	10	10	10
5	28	10	10	10
6	23	10	10	10
7	20	10	10	10
8	17	10	10	10
9	15	10	10	10
10	14	10	10	10
NOTE From one production unit with constant incoming raw materials and consisting of up to 3 months production.				

Table E.4 — Number of increments per (sub)-lot for woodchips, including bark with a nominal top size of 16 mm

	number of increments (n) per sub-(lot)		
number of sub-lots (N_{SL})	Total moisture P_L=1,0 w-%	Ash P_L=0,1w-%	Particle size distribution P_L=2,0 w-%
1	65	24	181
2	28	11	22
3	18	10	12
4	13	10	10
5	10	10	10
NOTE Sieve range used for particle size distribution: 16 mm, 8 mm, 5 mm, 3 mm and 2 mm.			

Table E.5 — Number of increments per (sub)-lot for sawdust from conifer

	number of increments (n) per (sub)-lot		
number of sub-lots (N_{SL})	Total moisture P_L=1,0 w-%	Ash P_L=0,1 w-%	Particle size distribution P_L=2,0 w-%
1	32	10	Too low P_L
2	14	10	35
3	10	10	10
4	10	10	10
5	10	10	10
NOTE Sieve range used for particle size distribution: 5,6 mm, 4,0 mm, 2,8 mm, 2,0 mm, 1,4 mm, 1,0 mm, 0,5 mm.			

Table E.6 — Number of increments per (sub)-lot for bark, Bark from Scots pine with a nominal top size of 100 mm.

	number of increments (n) per (sub)-lot		
number of sub-lots (N_{SL})	Total moisture P_L=1,0 w-%	Ash P_L=0,15 w-%	GCV (db) P_L=0,1 MJ/kg
1	Too low P_L	Too low P_L	Too low P_L
2	Too low P_L	Too low P_L	101
3	114	10	25
4	25	10	14
5	14	10	10
6	10	10	10

Table E.7 — Number of increments per (sub)-lot for logging residue, from conifer with nominal top size of 64 mm

	number of increments (n) per (sub)-lot		
number of sub-lots (N_{SL})	Total moisture P_L=1,5 w-%	Ash P_L=1,0 w-%	Particle size distribution P_L=5,0 w-%
1	Too low P_L	Too low P_L	Too low P_L
2	25	10	Too low P_L
3	10	10	Too low P_L
4	10	10	Too low P_L
5	10	10	10

Table E.8 — Number of increments per sub-lot from wheat straw in bales

	number of increments (n) per (sub)-lot		
number of sub-lots (N_{SL})	Total moisture P_L=2,5 w-%	Ash P_L=0,5 w-%	Chlorine P_L=0,02 w-%
1	Too low P_L	468	200
2	1538	18	67
3	61	10	40
4	31	10	29
5	21	10	22
6	16	10	18
7	13	10	15
8	11	10	13
9	10	10	12
10	10	10	11

Table E.9 — Number of increments per (sub)-lot for olive residue, typical Mediterranean materials with a nominal top size of 3 mm

	number of increments (n) per (sub)-lot[a)]									
number of sub-lots (N_{SL})	Moisture P_L=0,4	Ash P_L=1,0	Al P_L=150	Ca P_L=1500	Mg P_L=500	Na P_L=50	P P_L=50	Si P_L=2000	K P_L=1000	N P_L=0,10
1	21	Too low P_L	Too low P_L	Too low P_L	Too low P_L	Too low P_L	Too low P_L	Too low P_L	Too low P_L	Too low P_L
2	10	Too low P_L	Too low P_L	Too low P_L	Too low P_L	Too low P_L	Too low P_L	11	10	Too low P_L
3	10	10	14	10	10	28	10	10	10	20
4	10	10	10	10	10	10	10	10	10	10
5	10	10	10	10	10	10	10	10	10	10

[a)] The P_L given for the elements correspond to ppm, except for nitrogen, ash and moisture which are in weight percentages.

Table E.10 — Number of increments per (sub)-lot for grape residue, typical Mediterranean materials with a nominal top size of 16 mm

	number of increments (n) per (sub)-lot [a)]									
number of sub-lots (N_{SL})	Moisture P_L=1,5	Ash P_L=1,0	Al P_L=150	Ca P_L=3500	Mg P_L=200	Na P_L=50	P P_L=200	Si P_L=1000	K P_L=1500	N P_L=0,10
1	Too low P_L	15	77	Too low P_L	Too low P_L	162	Too low P_L	Too low P_L	Too low P_L	Too low P_L
2	Too low P_L	10	10	11	10	17	7989	10	Too low P_L	18
3	Too low P_L	10	10	10	10	10	10	10	10	10
4	18	10	10	10	10	10	10	10	10	10
5	10	10	10	10	10	10	10	10	10	10

[a)] The P_L given for the elements correspond to ppm, except for nitrogen, ash and moisture which are in weight percentages.

E.3 Examples for determining V_{PT}, V_I, N_{SL} and n_{min}

Clause 8 describes how to determine V_{PT} and V_I from a given lot. F.3 describes an example on determining and calculating these parameters.

EXAMPLE 1 Determining the minimum number of increments when sampling a seagoing vessel carrying wood pellets:

A seagoing vessel is loaded with wood pellets from a large stockpile stored inside a warehouse. The pellets are taken from the stockpile with a front loader, and put into a hopper feeding a conveyor belt running to the wharf. At the end of the conveyor belt the material is dumped into a ship using a chute. The total amount to be loaded is 6 000 tonnes. There is no mechanical sampler on the conveyor belt, which would have been the preferred method of sampling. The falling stream from the chute cannot safely be reached with a sampling tool. It was therefore decided that the normative method to be used in this case was sampling from a stockpile during reclaiming. Because of safety concerns it was first agreed with the shipper that the front loader would be parked at a safe distance when increments from the freshly exposed surface were taken. Because a sub-lot for manual sampling can only be 2 500 tonnes maximum (see 6.4), the 6000 ton lot was divided into 3 equal sub lots, each of approximately 2 000 tonnes, for sampling and analyses.

It was decided that the final overall precision should be 0,25 w-% for total moisture.

The minimum number of increments per sub-lot was calculated using the empirical values of Annex D: V_I = 0,34 w-%^{2} and V_{PT} = 0,002 w-%^{2}

and applying the equation below:

$$n_{\min} = \frac{4V_I}{N_{SL}P_L^{\,2} - 4V_{PT}} = \frac{4 \cdot 0{,}34}{3 \cdot 0{,}25^2 - 4 \cdot 0{,}002} = 8$$

n_{min} is less than the minimum number of increments per sub-lot (see 8.5), and therefore changed to n=10.

n_{min}=10 is the number of increments that should be taken from *each* of the three sub-lots. The increments from the individual sub-lots together form three (3) combined samples. On each combined sample a moisture measurement is performed and an average value is calculated.

If ash content and gross calorific value have to be measured, the numbers of increments for these parameters have to be calculated individually as well, and the highest number shall be used.

For ash, with an agreed end precision of P_L = 0,20 w-%, and suggested values of V_I=0,53 w-%^{2} and V_{PT}= 0,015 w-%^{2} (see Annex D) the above formula would give a minimum number of increments per sub-lot of n_{min}=35.

For gross calorific value (db) an agreed end precision of P_L= 0,1 MJ/kg and suggested values of V_I= 0,038 (MJ/kg)^2 and V_{PT}=0,0061 (MJ/kg)^2 (see Annex D) the above formula would give a minimum number of increments of per sub-lot of n_{min}=27.

This means that for the sample of each sub lot on which total moisture, ash and gross caloric value have to be analysed, a minimum of 35 increments for each of the 3 sub-lots shall be taken to comply with the minimum n of each parameter.

Improved precision

The setup is the same as above. If the overall precision for ash, P_L is changed to 0,15 w-% (instead of 0,20 w-%, above) and suggested values of V_I=0,53 w-%^{2} and V_{PT}= 0,015 w-%^{2} (see Annex D) Equation (6) would give a minimum number of increments of n_{min}= 283.

This is not a very practical number. It can be decided to increase the number of sub-lots from 3 to 6, for example, meaning extraction of one sample per 1 000 tonnes sub-lot. With N_{SL}=6 sub-lots the number of increments would then drop to n_{min}=28 per sub-lot (a total of 168 increments from the entire lot).

EXAMPLE 2 Durability of wood pellets delivered to a power plant by lorries.

During a week 7 lorries will deliver 8 mm wood pellets to a power plant. Sampling needs to be done to determine a calculated average durability of the pellets of each lorry during this week. The total amount of pellets will be considered as the lot.

First the calculations are done regarding the entire delivery as a single lot, with no division into sub-lots.

The durability measurement shall only be performed on the *total* combined sample (composited from all the increments) collected during the entire week. The minimum number of increments would then be as follows:

$$n = \frac{4V_I}{NP_L{}^2 - 4V_{PT}} = \frac{4 \cdot 0{,}208}{1 \cdot 0{,}20^2 - 4 \cdot 0{,}0061} = 53$$

n_{min}=53 is the number of increments that should be taken during the entire week over the entire lot. This means per lorry a minimum number of samples of 53/7=8 increments shall be taken. The durability test is performed on the combined sample (composited from the 53 increments collected during the week). No weighted average is necessary.

If 53 increments, for instance, is considered too many to handle as a single combined sample (it may be too heavy to carry or similar), a new number of required sub-lots, with a chosen number of increments making up each combined sample (one for each sub-lot), can be calculated with Equation (7).

If it is decided to try to obtain a maximum of 20 increments per sub-lot, n_{MP}, N_{SL} from (n_{MP} = 20) gives:

$$N_{SL} = \frac{4(V_I + n_{MP}V_{PT})}{n_{MP}P_L{}^2} = \frac{4(0{,}208 + 20 \cdot 0{,}0061)}{(20 \cdot 0{,}20^2)} = 1{,}7$$

Now this value is rounded up to 2 sub-lots, which is used in Equation (1), which now yields:

$$n_{\min} = \frac{4V_I}{N_{SL}P_L{}^2 - 4V_{PT}} = \frac{4 \cdot 0{,}208}{2 \cdot 0{,}20^2 - 4 \cdot 0{,}0061} = 15$$

Using this approach, 15 increments have to be extracted from each of the two sub-lots (a total of 2 x 15 = 30 increments over the entire lot).

Lastly, the lorries can each be considered as a sub-lot (N_{SL}=7 sub-lots).

In this case each lorry will be sampled, the increments from each are composited and analysed, yielding 7 analysis results. The lot is the total of the 7 lorries and the final overall precision is based on the entire lot of 7 sub-lots.

It is decided that the final overall precision should be 0,20 w-%, (the recommended precision from Table D.3). The minimum number of increments is calculated using the reference figures of Annex D: V_I=0,208 w-%^{2} and V_{PT}=0,0061 w-%^{2}.

$$n_{\min} = \frac{4V_I}{N_{SL}P_L{}^2 - 4V_{PT}} = \frac{4 \cdot 0{,}208}{7 \cdot 0{,}20^2 - 4 \cdot 0{,}0061} = 3$$

Since 3 is less than the minimum number of increments, $n_{\min}$ should be set to 10 (see 8.5)

$n_{\min}$=10 is the number of increments that should be taken from each sub-lot, in this case each lorry. On the combined sample of each sub-lot the durability measurement is performed. At the end of the week a weighted average is calculated based on the weight of each load. The average value is thus based on 7 x 10 = 70 increments in total over the entire lot.

EXAMPLE 3 Biofuel production facility

One supplier of logging residue delivers 2 lorry loads (40 tonnes each) to a power station per day. The supplier would like to know the moisture content of the material that is transported from the production facility, and want to design a sampling scheme. The increment variance, V_I, is not known and should now be estimated. Until such an estimate can be achieved, the empirical values in Annex D are adopted to calculate the minimal number of increments, $n_{\min}$.

P_L = 1,50 w-%, V_I = 10 w-%^{2} and V_{PT} = 0,73 w-%^{2}

Initially the calculation is done without division into sub-lots (N_{SL}=1). In accordance with Equation (1), the minimum number of increments is calculated:

$$n_{\min} = \frac{4V_I}{N_{SL}P_L{}^2 - 4V_{PT}} = \frac{4 \cdot 10}{1 \cdot 1{,}50^2 - 4 \cdot 0{,}73} = -60$$

This number is negative, meaning that the final overall precision cannot be achieved without division into sub-lots.

Instead the final overall precision is changed to P_L = 2,5 w-%. Now the calculation yields:

$$n_{\min} = \frac{4V_I}{N_{SL}P_L{}^2 - 4V_{PT}} = \frac{4 \cdot 10}{1 \cdot 2{,}50^2 - 4 \cdot 0{,}73} = 12$$

12 increments is possible to use in practice.

EXAMPLE 4 Parties agree to a different minimal increment - from a production process

As described in this standard parties may decide upon a precision and even a lower minimum number of increments. Care should be taken that all parties agree and understand the consequences.

For example a pellets factory production is audited according to existing certification program. In the program it is clearly described between all parties involved the following:

— It will suffice to have a low precision (higher value for P_L) to get an indication of the quality management system.

NOTE Inspection is described in certification program.

— The sample material needed is withdrawn from the product stream in the form of at least 10 separate increments, each with a mass of at least 1 kg.

— The samples shall be taken at the last possible place in the production stream.

— The separate samples shall be taken at sufficiently long intervals to allow a multiple of (at least five times) the mass of each single increment to pass along the conveyor between one sampling time and the next. All increments are put together to form a combined sample for analysis.

Annex F
(informative)

Quality parameters for various solid biofuels in BioNorm projects and large shipments of wood pellets

F.1 General

In this annex a description is given of the BioNorm experiments including the analytical results of these experiments. In addition the experimental results on large shipments of mixed wood pellets from different sources are included in this annex.

F.2 Products investigated as part of the BioNorm projects

The variances referred to in this standard have been extracted from sampling experiments performed in the EU-projects BioNorm and BioNorm2. In both projects the experimental design used for the estimation of the variances was a nested (hierarchical) design. Ten different biofuel materials were studied in the sampling experiments, namely sawdust, logging residue, straw and 8 mm pellets from sawdust in BioNorm and bark, wood chips, olive residue, grape residue, 6 mm pellets and 8 mm pellets from stem wood including bark in BioNorm2. A description of the materials is shown in Table F.1.

Table F.1 — Description of biofuel materials

Sample	Sample origin
Sawdust	Sawdust from conifer with a nominal topsize of 5,6 mm (without bark).
Logging residue	Logging residue from conifer with a nominal topsize of 64 mm.
Bark	Bark from Scots pine with a nominal top size of 100 mm.
Wood chips	Wood chips from stem wood including bark with a nominal top size of 16 mm.
Stem wood pellets (8mm)	8 mm pellets from stem wood including bark (see wood chips above) from one production site with changing raw material quality over 3 months production.
Pellets from sawdust (8mm)	8 mm pellets produced from sawdust (see sawdust above).
Deciduous pellets (6mm)	6 mm pellets produced from whole tree of deciduous trees.
Olive residue	Olive residues (typical Mediterranean materials) with a nominal top size of 3 mm.
Grape residue	Grape residues (typical Mediterranean materials) with a nominal top size of 16 mm.
Straw	Straw from wheat in bales

Sub-lots of bark, sawdust and logging residue were sampled from a heap tipped on a hard, surface and from a stopped conveyor, respectively. From the sub-lots, four increments of three different increment masses were taken by these two different sampling methods.

Sub-lots of wood chips, 8mm pellets from wood chips, olive residue and grape residue were sampled both from a falling stream at the end of a moving conveyor and from a heap. From each sub-lot, four increments of three different increment masses were sampled by these two different sampling methods.

The 8 mm pellets from sawdust and 6 mm deciduous pellets were both sampled from randomly chosen 16 kg bags and from heaps. From each sub-lot, four increments of three different masses were collected and sampled by these two different sampling methods.

For all materials two sub-samples of each increment were tested for moisture content and ash content. In addition, particle size distribution was tested in logging residue and wood chips, gross calorific value in bark, mechanical durability in the 8 mm pellets from stem wood including bark and 6 mm pellets, chloride in straw and 8 major elements in olive and grape residue.

The sampling experiments with bark, sawdust, 8 mm sawdust pellets, wood chips, 8 mm stem wood pellets and logging residue were carried out in Sweden. The straw was grown and sampled in Denmark. Olive residue, grape residue and 6 mm deciduous pellets were sampled in Italy.

Laboratory samples were prepared and analysed in three different labs namely in Italy, Denmark and Sweden. No round robin was conducted.

F.3 Summary of results from BioNorm projects

Results from BioNorm, WP I

In BioNorm I different scenarios were investigated namely:

— for sawdust, logging residue and wood pellets small stockpile (tipped lorry load) and cross stream cut from a stopped conveyor were compared,

— for straw bales the core drilling and hook sampling methods were compared,

— for sawdust 3 different increment sizes 0,2 l; 1,0 l and 5,0 l were compared,

— for pellets 3 different increment sizes 0,25 kg ; 1,0 kg and 4,0 kg were compared,

— for logging residue 3 different increment sizes 1 l; 4 l and 20 l were compared.

The results represented here are the combined results of these scenarios since there were no significant differences in the end results.

Table F.2 — Analytical results from sawdust conifer

Parameter	Average	Stddev	CV	n
Moisture (w-%)	51,9	1,92	3,7	5
Ash (w-% db)	0,30	0,04	14,0	5
Particle size > 5,6 mm (w-%)	2,90	1,33	45,8	5
Particle size < 5,6 > 4,0 mm (w-%)	5,21	0,67	12,9	5
Particle size < 4,0 > 2,8 mm (w-%)	7,96	0,70	8,8	5
Particle size < 2,8 >2,0 mm (w-%)	12,0	0,62	5,2	5
Particle size < 2,0 > 1,4 mm (w-%)	21,3	3,09	14,5	5
Particle size < 1,4 > 1,0 mm (w-%)	17,8	0,51	2,9	5
Particle size < 1,0 > 0,5 mm (w-%)	23,2	1,76	7,6	5
Particle size < 0,5 mm (w-%)	9,66	0,48	5,0	5

Table F.3 — Analytical results from pellets of sawdust (8 mm)

Parameter	Average	Stddev	CV	n
Moisture (w-%)	7,56	1,32	17,5	5
Ash (w-% db)	0,29	0,006	2,2	5

Table F.4 — Analytical results from wheat straw in bales

Parameter	Average	Stddev	CV	n
Moisture (w-%)	20,3	4,78	23,5	5
Ash (w-% db)	5,54	1,03	18,5	5
Chloride (w-%)	0,34	0,075	21,9	5

Table F.5 — Analytical results from logging residue from conifer with nominal top size of 64 mm

Parameter	Average	Stddev	CV	n
Moisture (w-%)	43,4	2,08	4,8	5
Ash (w-% db)	3,12	0,62	19,7	5
Particle size > 64 mm (w-%)	2,51	1,05	42,0	5
Particle size < 64 > 32 mm (w-%)	10,9	2,29	21,0	5
Particle size < 32 > 16 mm (w-%)	26,1	0,61	2,3	5
Particle size < 16 > 8 mm (w-%)	26,5	1,34	5,0	5
Particle size < 8 > 4 mm (w-%)	14,8	1,54	10,4	5
Particle size < 4 > 2 mm (w-%)	8,73	0,75	8,6	5
Particle size < 2 mm (w-%)	10,4	2,34	22,5	5

NOTE 1 The data in the tables have been calculated from an investigation within the BioNorm, WP I (2002-2004) project where a number of sub-lots (n) have been analysed for various analytical parameters, In this study a limited number of sub-lots during a short period of time (about one week) were examined in order to estimate individual variance values (sub-lot, increment and test variances) for the various materials.

Results from BioNorm 2, WP I

In BioNorm2 different scenarios were investigated namely:

— for bark small stockpile (tipped lorry load) and cross stream cut from a stopped conveyor were compared

— for wood chips, pellets, grape residue and olive residue small stockpile and cross stream cut from a falling stream were compared

— for bark 3 different increment sizes 5,0 l; 10,0 l and 20,0 l were compared

— for pellets 3 different increment sizes 2,5 l; 4,0 l and 8,0 l were compared

— for wood chips 3 different increment sizes 2 l; 4 l and 10 l were compared

— for grape residue and olive residue 3 different increment sizes 2 l; 5 l and 10 l were compared

The results represented here are the combined results of these scenarios since there were no significant differences in the end results.

Table F.6 — Analytical results from bark from pine

Parameter	Average	Stddev	CV	n	Replicate std dev of test method
Moisture (w-%)	57,33	2,57	4,49	24	-
Ash (w-% db)	2,13	0,50	23,6	24	0,0057
GCV (MJ/kg)	20,66	0,21	1,03	24	0,017

Table F.7 — Analytical results from wood chips

Parameter	Average	Stddev	CV	n	Replicate std dev of test method
Moisture (w-%)	45,11	3,72	8,24	21	-
Ash (w-% db)	0,62	0,19	30,7	21	0,012
Particle size < 16 mm > 8mm (w-%)	14,5	3,3	22,8	21	-
Particle size < 8 mm > 5 mm (w-%)	41,5	3,2	7,7	21	-
Particle size < 5 mm >3 mm (w-%)	31,0	3,2	10,4	21	-
Particle size < 3 mm > 2 mm (w-%)	4,9	1,1	23,1	21	-
Particle size < 2 mm (w-%)	7,4	2,1	28,8	21	-

Table F.8 — Analytical results from wood pellets (8 mm) from stem wood including bark from one production site with changing raw material quality

Parameter	Average	Stddev	CV	n	Replicate std dev of test method
Moisture (w-%)	8,44	0,62	7,30	25	-
Ash (w-% db)	0,62	0,048	7,78	25	0,013
Mechanical durability (%)	97,63	0,15	0,15	25	-

Table F.9 — Analytical results from wood pellets (6 mm) from one production site with a constant quality of raw materials (long time variation)

Parameter	Average	Stddev	CV	n
Moisture (w-%)	8,05	0,59	7,31	40
Ash (w-% db)	1,37	0,29	21,2	40
Mechanical durability (%)	97,2	1,01	1,04	40

Table F.10 — Analytical results from wood pellets (6 mm) from one production site with a constant quality of raw materials

Parameter	Average	Stddev	CV	n	Replicate std dev of test method
Moisture (w-%)	8,09	0,074	0,92	5	-
Ash (w-% db)	0,80	0,036	4,47	5	0,10
Mechanical durability (%)	98,21	0,059	0,060	5	-

Table F.11 — Analytical results from grape residue, typical Mediterranean materials with a nominal top size of 16 mm

Parameter	Average	Stddev	CV	n	Replicate std dev of test method
Moisture (w-%)	62,7	3,7	5,9	23	-
Ash (w-% db)	6,70	0,52	7,76	23	0,22
Aluminium (mg/kg)	201	41,3	20,6	23	59,1
Calcium (mg/kg)	6933	955	13,8	23	108
Magnesium (mg/kg)	1171	169	14,4	23	44,1
Sodium (mg/kg)	195	64,8	33,2	23	10,8
Phosphorous (mg/kg)	2359	231	9,8	23	59,1
Silicon (mg/kg)	1357	499	36,8	23	149
Potassium (mg/kg)	21812	1875	8,60	23	528
Nitrogen (w-%)	2,18	0,091	4,18	23	0,033

Table F.12 — Analytical results from olive residue, typical Mediterranean materials with a nominal top size of 3 mm

Parameter	Average	Stddev	CV	n	Replicate std dev of test method
Moisture (w-%)	11,42	0,18	1,54	5	-
Ash (w-% db)	6,14	0,97	15,7	5	0,21
Aluminium (mg/kg)	593	99,5	16,8	5	80,1
Calcium (mg/kg)	3763	653	17,3	5	824
Magnesium (mg/kg)	527	92,2	17,5	5	40,1
Sodium (mg/kg)	562	59,8	10,6	5	12,2
Phosphorous (mg/kg)	310	41,2	13,3	5	28,5
Silicon (mg/kg)	3878	965	24,9	5	984
Potassium (mg/kg)	8330	716	8,59	5	229
Nitrogen (w-%)	1,00	0,066	6,57	5	0,042

Table F.13 — Analytical results from olive residue, typical Mediterranean materials with a nominal top size of 3 mm (long time variation)

Parameter	Average	Stddev	CV	n
Moisture (w-%)	16,48	0,29	1,76	10
Ash (w-% db)	4,97	0,56	11,3	10
Aluminium (mg/kg)	463	185	39,9	10
Calcium (mg/kg)	6805	1468	21,6	10
Magnesium (mg/kg)	746	121	16,2	10
Sodium (mg/kg)	438	40,0	9,15	10
Phosphorous (mg/kg)	817	158	19,3	10
Silicon (mg/kg)	2735	1203	44,0	10
Potassium (mg/kg)	12447	1068	8,58	10
Nitrogen (w-%)	1,25	0,088	7,09	10

NOTE 2 BioNorm2, the data in the tables have been calculated from two different investigations within the BioNorm 2, WP I project (2007-2009), where a number of sub-lots (n) have been analysed for various analytical parameters, In a first study five sub-lots during a short period of time (about one week) were examined in order to estimate individual variance values (sub-lot, increment and test variances) for the various materials. A second study was used to verify the data in the first experiments and a larger number of sub-lots (10-40) over a longer period of time (5-12 weeks) were investigated. If no significant differences in averages and precisions were found between the two investigations, pooled values have been reported. If significant differences between averages and/or precisions were obtained, separate tables for the two examinations are presented.

F.4 Large shipments

In order to verify the standard against larger shipment the tests were carried out on a sea going vessel with a bulk cargo of wood pellets being discharged in the Netherlands.

For the test a relative heterogeneous cargo wood pellets produced by various production units was selected for the test. The total cargo quantity was approximately 10 000 tonnes. The vessel was being discharged from the seagoing vessel directly into river barges by a floating crane, The river barges are used for transporting the wood pellets to power plants. The discharge speed is approximately 500 tonnes per hour per crane. Up to two cranes would be used for the discharge at the same time. The pellets were between white-brown to dark brown. The diameter was from 6 mm to 8 mm, with a length up to 30 mm.

Sampling was performed from the piles as they were being built up in the barge in accordance with the "stock pile during build up" procedure.

For the increment variance 50 increments were taken both for the quality and moisture sample. An increment would be drawn every 200 tonnes on different places. For the test and preparation variance 20 samples were drawn each consisting of 24 increments of approximately 0,6 kg. The test and preparation variance sample were drawn per lot of 500 tonnes.

— the increment samples used to calculate the increment variance were individually prepared and analysed.

— the samples for the preparation and test variance were carefully mixed with the use of a riffle divider and then split into two separate parts A and B. The part A and B were than separately prepared and analysed using the normal procedure,

— all analyses were performed in duplicate and the average was reported.

The sampling, preparation and tests were performed in accordance with the above named methods.

The results are shown in the following tables. The figures for ash and calorific value have been calculated to a dry basis with the use of the moisture in the analysis sample.

Table F.14 — Preparation and Test variance

Sample part A				Sample part B					Ash			Total moisture			GCV	
Sample	Total Moisture w-%	Ash w-%	GCV MJ/kg	Sample	Total Moisture w-%	Ash w-%	GCV MJ/kg		d_i	d_i^2		d_i	d_i^2		d_i	d_i^2
1	5,53	2,03	20,21	**1**	5,44	1,83	19,96		0,20	0,039296		0,09	0,0081		0,25	0,062752
2	5,53	0,89	20,23	**2**	5,51	0,82	20,15		0,07	0,005402		0,02	0,0004		0,08	0,006879
3	5,21	1,70	20,25	**3**	5,22	1,43	20,16		0,27	0,072906		-0,01	1E-04		0,09	0,007431
4	5,11	1,76	20,33	**4**	5,19	1,60	20,21		0,16	0,024331		-0,08	0,0064		0,12	0,013581
5	5,12	0,74	20,40	**5**	5,16	0,72	20,42		0,02	0,000512		-0,04	0,0016		-0,02	0,000274
6	5,37	2,30	20,13	**6**	5,33	2,31	20,18		-0,01	0,000118		0,04	0,0016		-0,05	0,002605
7	5,56	1,26	20,31	**7**	5,52	0,97	20,55		0,30	0,088197		0,04	0,0016		-0,24	0,058593
8	5,56	1,34	20,28	**8**	5,54	1,47	20,27		-0,13	0,017299		0,02	0,0004		0,01	6,55E-05
9	5,57	1,58	20,27	**9**	5,71	1,38	20,22		0,20	0,041793		-0,14	0,0196		0,05	0,002466
10	5,64	1,57	20,17	**10**	5,73	1,89	20,22		-0,32	0,102196		-0,09	0,0081		-0,05	0,002545
11	4,88	1,06	20,34	**11**	4,8	0,78	20,26		0,29	0,08187		0,08	0,0064		0,08	0,005757
12	4,48	1,34	20,15	**12**	4,49	1,18	20,10		0,15	0,022791		-0,01	1E-04		0,05	0,002098
13	5,46	0,69	20,36	**13**	5,44	0,72	20,32		-0,03	0,001011		0,02	0,0004		0,05	0,002167

Table F.14 *(continued)*

Sample part A				Sample part B					Ash			Total moisture			GCV	
Sample	Total Moisture w-%	Ash w-%	GCV MJ/kg	Sample	Total Moisture w-%	Ash w-%	GCV MJ/kg		d_i	d_i^2		d_i	d_i^2		d_i	d_i^2
14	4,5	0,27	20,45	**14**	4,5	0,45	20,29		-0,18	0,031718		0	0		0,16	0,025429
15	4,59	0,81	20,29	**15**	4,62	1,05	20,17		-0,24	0,059812		-0,03	0,0009		0,12	0,01358
16	4,2	0,39	20,30	**16**	4,21	0,36	20,25		0,03	0,001182		-0,01	1E-04		0,05	0,002922
17	4,66	0,47	20,29	**17**	4,57	0,48	20,33		-0,01	0,000138		0,09	0,0081		-0,04	0,001277
18	4,49	0,70	20,38	**18**	4,51	0,76	20,26		-0,06	0,003479		-0,02	0,0004		0,12	0,01491
19	5,23	0,68	20,40	**19**	5,2	0,59	20,28		0,09	0,007893		0,03	0,0009		0,12	0,013809
20	4,2	0,50	20,28	**20**	4,1	0,39	20,21		0,12	0,01386		0,1	0,01		0,07	0,005289
mean	5,04	1,10	20,29	**mean**	5,04	1,06	20,24	$\frac{\sum d_i^2}{2n_p}$		0,615802			0,0752			0,244432
									V_{PT}=	0,015		V_{PT}=	0,002		V_{PT}=	0,0061

Table F.15 — Increment variance

Sample part 50						Ash			Total moisture			GCV	
Sample	Total moisture (w-%)	Ash (w-%)	GVC (MJ/kg)			x_i	x_i^2		x_i	x_i^2		x_i	x_i^2
1	5,350	0,700	20,433			0,70	0,49		5,35	28,6225		20,433	417,5075
2	5,600	0,560	20,486			0,56	0,3136		5,6	31,36		20,486	419,6762
3	5,490	1,440	20,284			1,44	2,0736		5,49	30,1401		20,284	411,4407
4	5,070	2,160	19,999			2,16	4,6656		5,07	25,7049		19,999	399,96
5	5,290	1,450	20,117			1,45	2,1025		5,29	27,9841		20,117	404,6937
6	5,210	0,500	20,363			0,50	0,25		5,21	27,1441		20,363	414,6518
7	5,170	0,765	20,244			0,77	0,5856		5,17	26,7289		20,24421	409,8281
8	5,150	0,480	20,273			0,48	0,2304		5,15	26,5225		20,273	410,9945
9	4,890	3,230	19,744			3,23	10,432		4,89	23,9121		19,744	389,8255
10	4,680	0,590	20,209			0,59	0,3481		4,68	21,9024		20,209	408,4037
11	4,810	2,490	19,862			2,49	6,2001		4,81	23,1361		19,862	394,499
12	5,160	0,580	20,201			0,58	0,3364		5,16	26,6256		20,201	408,0804
13	5,420	2,910	20,104			2,91	8,4681		5,42	29,3764		20,104	404,1708
14	5,630	2,310	20,030			2,31	5,3361		5,63	31,6969		20,03	401,2009
15	5,440	1,290	20,417			1,29	1,6641		5,44	29,5936		20,417	416,8539
16	6,340	2,670	20,195			2,67	7,1289		6,34	40,1956		20,195	407,838
17	5,340	1,940	20,077			1,94	3,7636		5,34	28,5156		20,077	403,0859
18	5,210	1,390	20,136			1,39	1,9321		5,21	27,1441		20,136	405,4585
19	5,070	2,000	20,028			2,00	4		5,07	25,7049		20,028	401,1208
20	5,100	2,100	19,989			2,10	4,41		5,1	26,01		19,989	399,5601
21	4,920	0,760	20,258			0,76	0,5776		4,92	24,2064		20,258	410,3866
22	5,170	2,340	20,141			2,34	5,4756		5,17	26,7289		20,141	405,6599
23	5,020	1,480	20,127			1,48	2,1904		5,02	25,2004		20,127	405,0961
24	4,420	1,420	20,169			1,42	2,0164		4,42	19,5364		20,169	406,7886

Table F.15 *(continued)*

Sample part 50						Ash			Total moisture			GCV	
Sample	Total moisture (w-%)	Ash (w-%)	GVC (MJ/kg)			x_i	x_i^2		x_i	x_i^2		x_i	x_i^2
25	4,230	1,360	20,184			1,36	1,8496		4,23	17,8929		20,184	407,3939
26	4,510	0,920	20,088			0,92	0,8464		4,51	20,3401		20,088	403,5277
27	4,330	1,490	20,054			1,49	2,2201		4,33	18,7489		20,054	402,1629
28	5,260	0,850	20,193			0,85	0,7225		5,26	27,6676		20,193	407,7572
29	4,370	1,300	20,215			1,30	1,69		4,37	19,0969		20,215	408,6462
30	4,170	1,110	19,989			1,11	1,2321		4,17	17,3889		19,989	399,5601
31	4,080	0,460	20,215			0,46	0,2116		4,08	16,6464		20,215	408,6462
32	5,040	0,600	20,338			0,60	0,36		5,04	25,4016		20,338	413,6342
33	4,110	1,080	20,293			1,08	1,1664		4,11	16,8921		20,293	411,8058
34	4,820	0,670	20,424			0,67	0,4489		4,82	23,2324		20,424	417,1398
35	4,000	0,460	20,316			0,46	0,2116		4	16		20,316	412,7399
36	4,030	0,440	20,312			0,44	0,1936		4,03	16,2409		20,312	412,5773
37	4,020	0,560	20,400			0,56	0,3136		4,02	16,1604		20,4	416,16
38	4,040	0,530	20,298			0,53	0,2809		4,04	16,3216		20,298	412,0088
39	4,290	1,190	20,220			1,19	1,4161		4,29	18,4041		20,22	408,8484
40	3,800	1,160	20,212			1,16	1,3456		3,8	14,44		20,212	408,5249
41	4,200	0,390	20,276			0,39	0,1521		4,2	17,64		20,276	411,1162
42	4,810	0,800	20,329			0,80	0,64		4,81	23,1361		20,32907	413,2712
43	3,910	0,370	20,246			0,37	0,1369		3,91	15,2881		20,246	409,9005
44	4,870	0,560	21,131			0,56	0,3136		4,87	23,7169		21,13076	446,509
45	4,910	0,440	20,730			0,44	0,1936		4,91	24,1081		20,73	429,7329
46	4,470	1,050	20,152			1,05	1,1025		4,47	19,9809		20,152	406,1031
47	4,220	1,310	20,234			1,31	1,7161		4,22	17,8084		20,234	409,4148

Table F.15 *(continued)*

Sample part 50						Ash			Total moisture			GCV	
Sample	**Total moisture (w-%)**	**Ash (w-%)**	**GVC (MJ/kg)**			x_i	x_i^2		x_i	x_i^2		x_i	x_i^2
48	4,370	0,819	20,305			0,82	0,6715		4,37	19,0969		20,30496	412,2917
49	4,110	0,520	20,335			0,52	0,2704		4,11	16,8921		20,335	413,5122
50	3,560	0,410	20,361			0,41	0,1681		3,56	12,6736		20,361	414,5703
Mean =						1,17			4,75			20,23472	
Sum =						58,40	94,865		237,48	1144,908		1011,736	20474,34
sum^2 =						3411,1			56396			1023609	
$\frac{1}{n-1}\left[\sum x_i^2 - \frac{\left(\sum x_i\right)^2}{n}\right] - V_{PT}$					=	0,53			0,34			0,038	

Bibliography

[1] EN 14774 (all parts), Solid biofuels — Determination of moisture content — Oven dry method

[2] EN 15103, Solid Biofuels — Determination of bulk density

[3] EN 15149 (all parts), Solid biofuels — Determination of particle size distribution

[4] EN 15210-1, Solid Biofuels — Determination of mechanical durability of pellets and briquettes — Part 1: Pellets

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

[6] ISO 13909-7, Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing

[7] ISO 13909-8, Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias

[8] ISO 18283, Hard coal and coke — Manual sampling

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