SLOVENSKI STANDARD
SIST EN 14778:2011

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SIST-TS CEN/TS 14778-1:2006
SIST-TS CEN/TS 14778-2:2005
SIST-TS CEN/TS 14779:2005

Trdna biogoriva - Vzorčenje

Solid biofuels - Sampling

Feste Biobrennstoffe - Probenahme

Biocombustibles solides - Echantillonnage

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75.160.10	Trda goriva	Solid fuels

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English Version

Solid biofuels - Sampling

Biocombustibles - Echantillonnage

Feste Biobrennstoffe - Probenahme

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

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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of an identical text or by endorsement, at the latest by December 2011, and conflicting national standards shall be withdrawn at the latest by December 2011.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

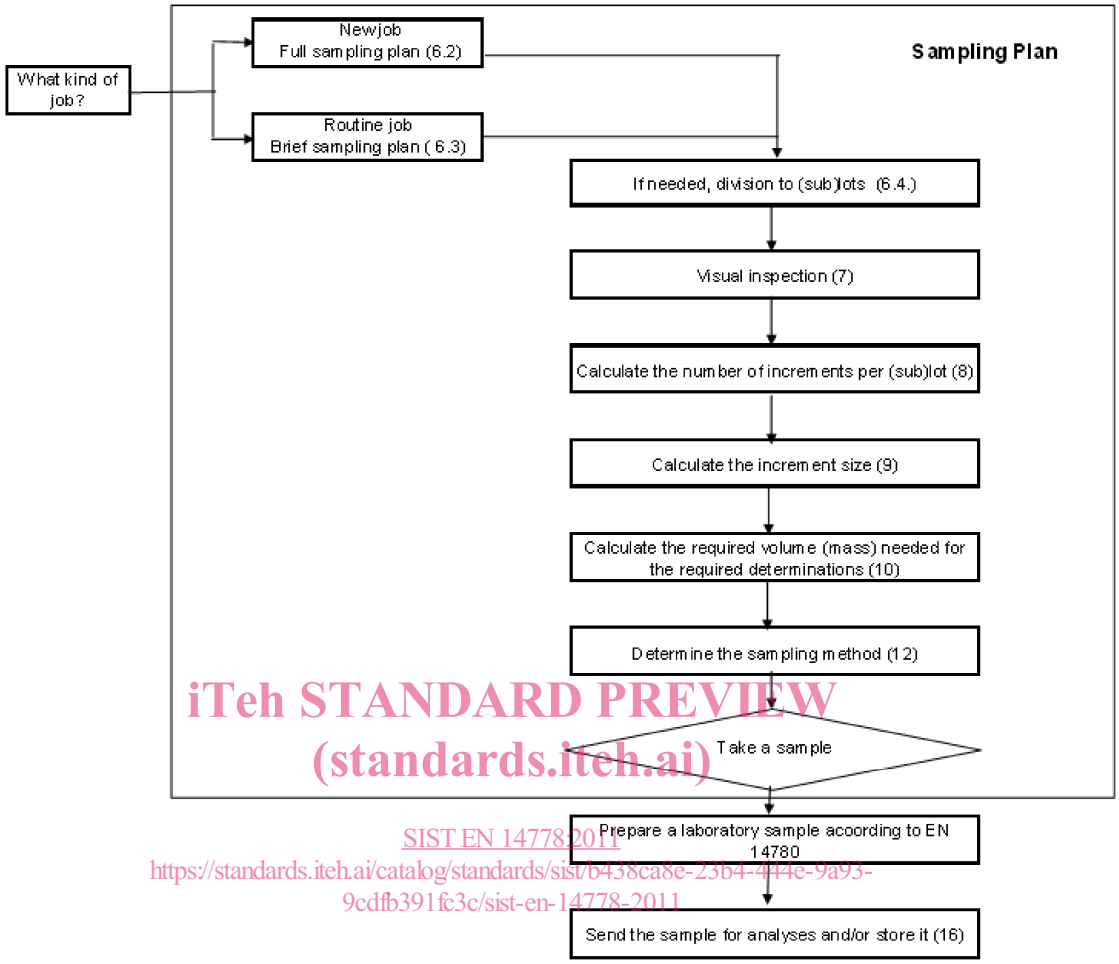
W is width of a sampling tool, mm

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 $n_{\min}$