BS EN 15413:2011

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Solid recovered fuels — Methods for the preparation of the test sample from the laboratory sample

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National foreword

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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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English Version

Solid recovered fuels - Methods for the preparation of the test sample from the laboratory sample

Combustibles solides de récupération - Méthodes pour la préparation d'échantillons pour essai à partir d'échantillons pour laboratoire

Feste Sekundärbrennstoffe - Verfahren zur Herstellung der Versuchprobe aus der Laboratoriumsprobe

This European Standard was approved by CEN on 15 July 2011.

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Foreword

This document (EN 15413:2011) has been prepared by Technical Committee CEN/TC 343 “Solid Recovered Fuels”, the secretariat of which is held by SFS.

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of an identical text or by endorsement, at the latest by March 2012, and conflicting national standards shall be withdrawn at the latest by March 2012.

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 15413:2006.

This document differs from CEN/TS 15413:2006 as follows:

a) only the dissolution methods that have passed the validity test have been considered.

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.

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Introduction

In laboratory praxis, different analytical procedures often need to be applied to the laboratory sample that has been taken according to the sampling plan. For this purpose, sub-sampling is applied in a way that the different test portions are representative for the original laboratory sample with respect to the compounds of interest and the specific analytical procedures. The representativity of the laboratory sample and of the test portions is of major importance to guarantee the quality and accuracy of analytical results. The representativity of the laboratory sample is specified by the sampling plan.

This European Standard is largely based on the work already done by CEN/TC 292 "Characterization of waste", and in particular on latest drafts of just published EN 15002; in fact, some experts who developed EN 15002 also actively participated in the preparation of this European Standard.

EN 15002 was developed for the majority of waste samples, and most of its concepts and specifications are indeed also applicable to SRF samples, but there would be a number of major problems:

— several points of Annex A (normative) of EN 15002:2006 ("Guideline for choosing sample treatment techniques") are simply not applicable to SRF samples due to the very particular nature of these samples and in some cases this could be misleading;

— the main peculiarity that makes SRF samples significantly different from other kinds of waste is that very often SRFs are solid, but neither "granular" nor monolithic; it often happens that SRF samples are fibrous-like materials, so the statistical formula for sampling (Annex B (normative) of EN 15002:2006, that links the minimum amount of sample depending on the particle size and other parameters), that is one of the foundations of EN 15002, is not applicable "as it is": one more term in the statistical equation is needed, namely the "shape factor" (*f*);

— all examples contained in Annex E of EN 15002:2006 are just not applicable for SRF samples, which may lead users who need to analyze SRF samples to misunderstandings.

Because of these reasons, a significant revision of the recently published EN 15002 would have been necessary in order to fulfil all requirements for SRF samples, which presumably would be better carried out jointly by CEN/TC 292 and CEN/TC 343. Moreover, other CEN/TC 292 standards and ENs on sampling of waste would have become inconsistent and would have had to be revised in order to include the "shape factor" in the statistical formula. However, all of this work would probably have caused unacceptable delays for both ENs. Therefore, CEN/TC 343 decided to proceed with the development of a new Standard.

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1 Scope

This European Standard specifies the correct sequence of operations to ensure the representativity of the test portions that have been taken according to the sampling plan, prior to physical and/or chemical analysis (e.g. extractions, digestion and/or analytical determinations) of solid samples.

This European Standard specifies the correct sequence of operations and treatments to be applied to the laboratory sample in order to obtain suitable test portions in compliance with the specific requirements defined in the corresponding analytical procedures.

2 Normative references

The following referenced documents are indispensable for the application of this European Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15357:2011, *Solid recovered fuels — Terminology, definitions and descriptions*

EN 15443, *Solid recovered fuels — Methods for the preparation of the laboratory sample*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15357:2011 and the following apply.

3.1
drying
process of removing water from a sample

NOTE For the purpose of test portion preparation, it may be useful to remove just the amount of water that could interfere with other processes involved (e.g. during crushing or milling). In order to minimise the alteration of the sample during test portion preparation, removing the total amount of water present in the sample is not necessarily needed.

3.2
fraction separation
process of dividing components, particles or layers if homogenisation of the sample is practically not applicable and/or the analyses of different fractions or phases are appropriate

3.3
homogenisation
process of combining of components, particles or layers into a more homogeneous state of the original samples (in the case of composite samples) or pre-treated fractions of samples in order to ensure equal distribution of substances in and properties of the sample

3.4
sub-sampling
process of selecting one or more sub-samples from a sample

3.5
test portion; analytical portion
quantity of material of proper size, for measurement of the concentration or other properties of interest, removed from the test sample

NOTE The test portion may be taken from the laboratory sample directly if no preparation of sample is required (e.g. with liquids or samples of proper homogeneity, size and fineness), but usually it is taken from the prepared test sample.

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3.6
test sample; analytical sample
sample, prepared from the laboratory sample, from which test portions are removed for testing or analysis

NOTE 1 When the laboratory sample is further prepared (reduced) by subdividing, mixing, grinding, or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test or for analysis.

NOTE 2 The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

NOTE 3 Several laboratory samples can be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

4 Safety remarks

The safety in handling of potentially hazardous materials is dealt with relevant national and European regulations, which every laboratory should refer to.

In addition the following information is given:

— the apparatus for grinding, cutting, milling, and homogenisation may result harmful for the users. They have to be operated by skilled personnel strictly according to the manufacturer's instructions;

— all procedures have to be performed in a hood or in closed force-ventilated equipment, due to the possibility of generation of fine powders.

5 Principle

The laboratory sample is reduced in particle size and mass using different apparatus and procedures depending on the type of sample and the type of analysis to which the sample will be submitted.

6 Apparatus

For the purpose of preparation of test portions from the laboratory samples, appropriate equipment has to be chosen depending on the procedures selected according to Annex A.

In the selection of the type of treatment techniques, one should keep in mind that each of them has some potential impact on analytical results, because it can introduce contamination or alter the physical-chemical properties of the sample.

All glassware and devices that come in contact with the sample shall be made out of a suitable material, chemically compatible with the sample, selected in order to minimize contamination of samples. Care shall be taken to ensure good cleaning, in order to avoid cross-contamination of samples.

An informative list of appropriate equipment for the sample treatment procedures is given in Annex C.

7 Interferences and sources of error

The (sub)-sample shall be re-homogenised after any operation that may have resulted in segregation of different sized particles.

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Care should be taken to avoid loss of material and contamination of the sample via the air, by dust, by the use of the apparatus (e.g. from the ambient laboratory atmosphere or between samples stored or processed close to one another).

Three types of contamination could occur from the apparatus:

— abrasion;

— cross-contamination;

— chemical release.

Chemical reaction due to generated heat can be as well a source of error and material alteration.

It is recommended to perform treatment of waste material in a separate room used only for this purpose, especially crushing or sieving.

If the sample has a dust-like consistency or contains (semi)-volatile compounds, part of it may be lost and this may alter its physical-chemical properties.

8 Procedure

8.1 Sample conservation and pre-treatment

The laboratory samples shall be stored according to guidelines defined in Annex D.

Furthermore any possible source of contamination during the laboratory sample preparation according to EN 15443 (e.g. grinding with metallic apparatus, mainly aluminium or aluminium alloy) shall be avoided or reduced as much as possible.

The laboratory sample should be stored and delivered in sealed high-density plastic containers.

8.2 Key concepts

Preparation of the test portion can be a complex process, because of a number of factors: sample type and its physical state, amount of laboratory sample, type and number of determinations to be carried out, etc. The prepared test portions shall satisfy the following requirements at the same time:

— each test portion shall be a representative of the laboratory sample;

— the amount and the physical state (e.g. particle size) of each test portion have to comply with the requirements of the respective analytical technique;

— for each test portion, no losses of and no contamination with respective analytes of interest should occur.

The preparation of the test portions from the laboratory sample, which has been taken according to the sampling plan, is related to the requested analytical determinations. This means that, if needed, contact has to be established among all involved parties such as the sampler, the customer and the analytical laboratory to achieve the requirements of the standards to be used for the requested determinations.

The preparation of test portions in the laboratory will frequently involve a sequence of operations such as homogenisation, fraction separation, drying, reducing particle size and sub sampling. Specific forms of these operations are described in A.1 to A.5, respectively. A number of decisions on the specific order of these operations for a particular laboratory sample have to be made. In some cases, the sequence of operations to be applied is rather straightforward, but in more complicated cases (e.g. when several determinations with different requirements have to be performed) it can be critical to choose the right sequence of such operations.

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In order to define the operations to be applied to a laboratory sample to produce one or more representative test portions, three main steps have to be considered:

a) Definition of analytical requirements

First, the requirements of analytical procedures of interest shall be defined:

1) methods to be used;

2) amount of test portions necessary;

3) the quantity and properties of the test portions necessary for each analytical procedure;

4) preservation requirements (e.g. time frame, temperature, addition of reagents).

NOTE 1 It is recommended to prepare at least five times the amounts needed as test portions for the test sample.

b) Definition of sequence of operations

Then, the sequence of operations shall be defined according to the flow sheet (Figure 1), based on the properties of the laboratory sample and the requirements of the analytical procedures: each single operation of this sequence has to be considered like an independent module; available modules are:

1) fraction separation;

2) drying;

3) particle size reduction;

4) homogenisation;

5) sub-sampling.

NOTE 2 For practical reasons it is recommended to group the parameters in a way that test samples with similar requirements can be prepared for several parameters. The same test sample may be used for different parameters if it fulfils the necessary requirements.

Frequently, different determinations have to be performed on the laboratory samples. In those cases, modules have to be combined and/or repeated to obtain sub-samples, finally resulting in different test portions. In order to define the actual sequence of operations to be applied to a given sample, the flow sheet (Figure 1) shall be used.

c) Choice of appropriate procedures

According to the requirements of the respective analytical techniques and the properties of the sample the appropriate sample treatment technique has to be chosen within each module by following the instructions of Annex A. Instructions are given in this annex in which case a particular operation is appropriate to use.

8.3 Sequence of treatment techniques

The flow sheet in Figure 1 describes the procedure to enable decisions on the specific order of treatment operations for a particular laboratory sample in order to yield in representative test portions. It shall be applied on the starting laboratory sample and repeated on all sample fractions or sub-samples subsequently obtained during the preparation, in an iterative cycle until all analytical requirements are fulfilled.

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In the case of mercury determination special care shall be taken in order to avoid losses of these volatile compounds during homogenisation and/or reduction of the particle size.

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

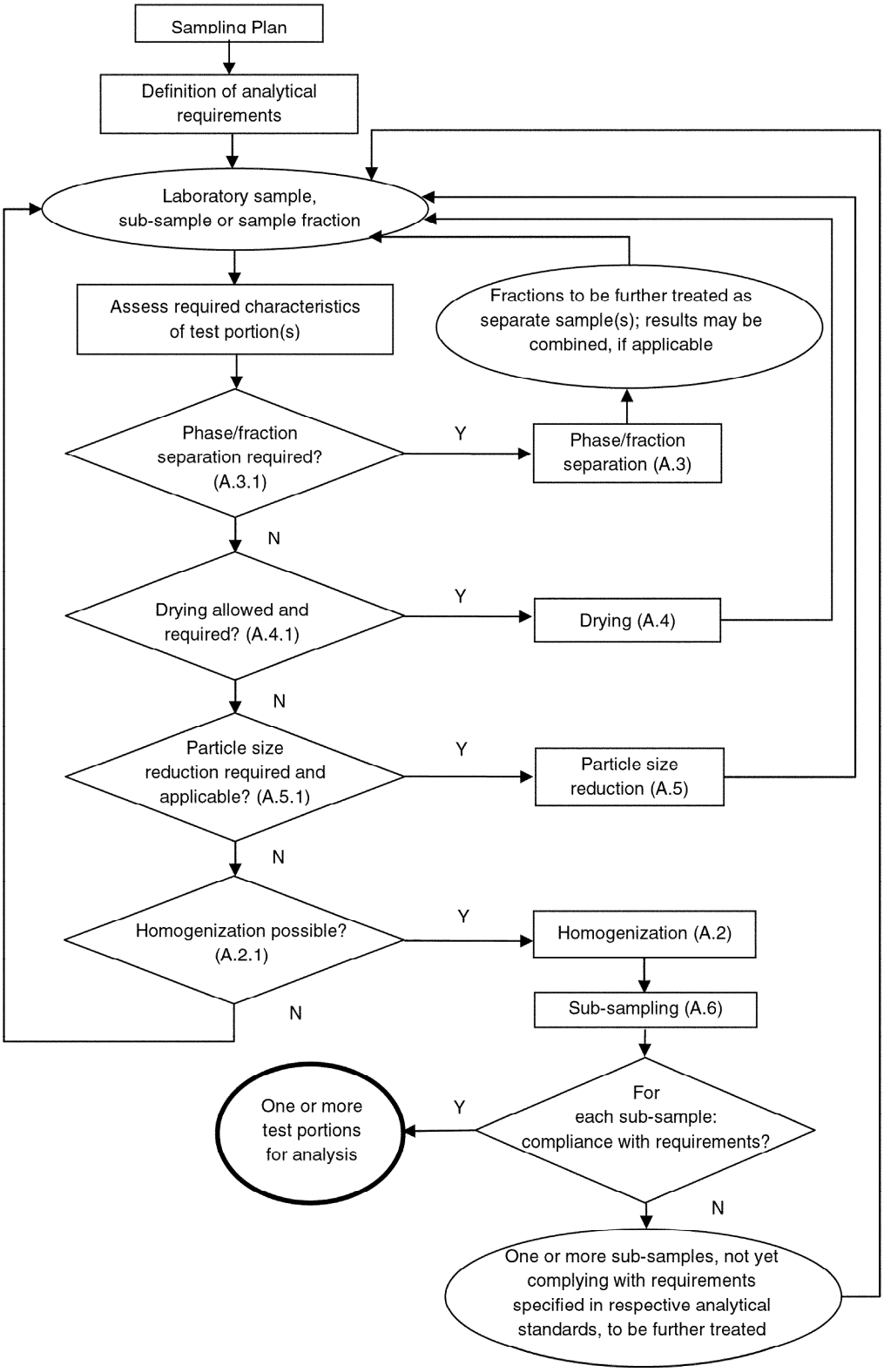

Figure 1 — Flow sheet - sequence of operations

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9 Quality control

Tests shall be carried out to detect possible contaminations from used apparatus.

10 Performance characteristics

Data about performances of the methods will be available after validation in QUOVADIS project (Quality Management Organisation, Validation of Standards, Developments and Inquiries for SRF).

11 Test report

The test report shall contain at least the following information:

a) name, address and location of any laboratory involved in the preparation of the test portions;

b) description and identification of the laboratory sample;

c) date of receipt of laboratory sample and date(s) of performance of test;

d) reference to this European Standard, i.e. EN 15413;

e) reference to the analytical standards used for the determination for each element;

f) the analytical results, referring to the relevant clause in the standards specified in e);

g) a reference to the sampling report;

h) the whole sequence and operating conditions (procedures and apparatuses) actually applied to the laboratory sample for preparation of test portions;

i) any details not specified in this European Standard or which are optional, and any other factors which may have affected the results;

j) unique identification of report (such as serial number) and of each page and total number of pages of the report.

The laboratory should keep a trace of any analytical steps and intermediate results (chromatograms, raw data and calculation details) that should be kept available in case of specific requirements.

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