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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

This British Standard is the UK implementation of EN 15413:2011. It supersedes DD CEN/TS 15413:2006 which is withdrawn.

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

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

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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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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

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of an identical text or by endorsement, at the latest by March 2012, and conflicting national standards shall be withdrawn at the latest by March 2012.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

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.

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.

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.

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.

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.

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.

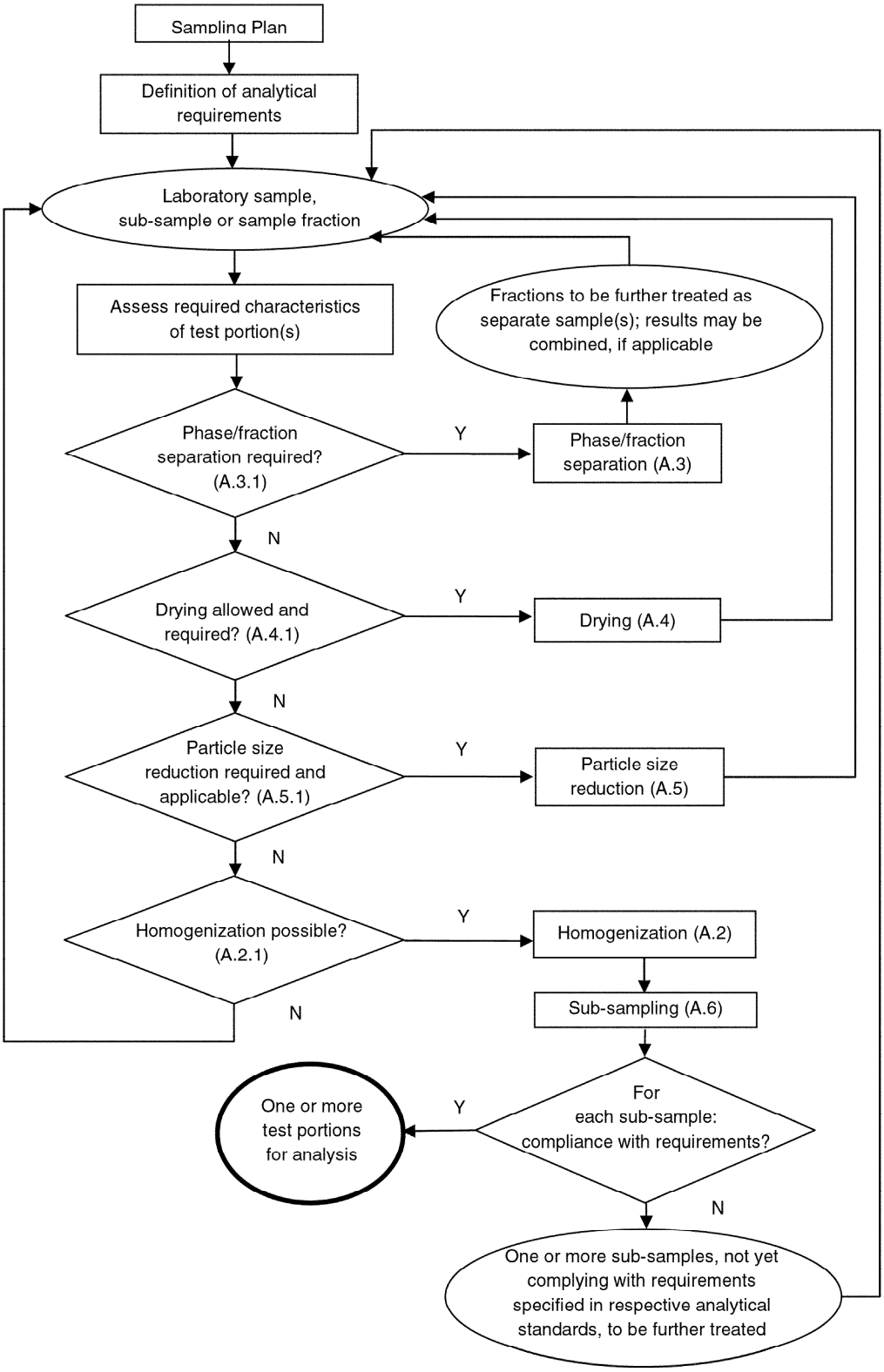


Figure 1 — Flow sheet - sequence of operations

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.

Annex A
(normative)

Guideline for choosing sample treatment techniques

A.1 General

The preparation of test samples from a laboratory sample will frequently involve a sequence of operations such as homogenisation, phase separation, drying, particle size reduction and sub-sampling. Specific forms of these operations are described in this annex.

The sample treatment techniques prescribed in the analytical standards have to be fulfilled in any case.

A.2 Homogenisation

A.2.1 General information

Before each operation that implies sub-sampling, a homogenisation step is required, in order to guarantee that all sub-samples or sample fractions have the same properties and composition. The homogenisation technique to be used is chosen depending on the properties of the sample.

In many cases before homogenisation, particle size reduction may be necessary.

A.2.2 Homogenisation techniques

A.2.2.1 Manual homogenisation

When to use it

— Generally usable;

— in cases when mechanical homogenisation could lead to loss of volatile compounds of interest (mercury).

When not to use it

— For samples that form layers because of the presence of particles of different density;

— for samples with particles of such a large size that homogenisation by manual mixing cannot be reached;

— for samples that form layers because of the presence of particles with large differences in particle size.

Procedure

Mix the sample with an appropriate tool (e.g. shovel, pestle and mortar). If there is a risk of losses of volatile substances the manual homogenisation has to be done very carefully.

A.2.2.2 Mechanical homogenisation

When to use it

— Generally usable especially in cases when manual homogenisation is not suitable;

— in cases of sample of large particle size;

— for solid samples containing particles of nearly the same density (e.g. for materials that do not form "layers" after shaking).

When not to use it

— For samples that form layers because of the presence of particles of different density; in this case, if homogenisation is not possible, separate and treat each layer as a different sample;

— when the apparatus may heat the sample and loss of volatile analytes can occur during this process; in this case, a manual homogenisation shall be performed.

Procedure

Operate according to the manufactures instructions.

NOTE For sample of small particle size, it may be also possible to use a ball-mill without balls for homogenisation.

A.2.3 Homogenisation in case of volatile compounds (mercury)

If mercury should be analysed, the sample homogenisation has to be done very carefully and quickly to avoid losses. If losses cannot be avoided during homogenisation process it may be a solution to take several non-homogenised sub-samples for analyses and calculate the statistical mean as an estimate of the total content. This step should not lead to major alterations of the composition and the representativity of the remaining sample.

NOTE The sampling plan should consider the presence of volatile compounds.

A.3 Fraction separation

A.3.1 General information

For heterogeneous samples, depending on their nature and on the determinations of interest, one or more techniques of fraction separation can be applied to obtain two or more different sub-samples that are to be analysed separately. For samples consisting of different fractions, separation of some fractions may be necessary. For this reason, the weight of each separated sub-sample shall be directly or indirectly measured after the separation, in order to allow a final weighed combination of different fractions' analysis results.

The test report shall clearly state the technique(s) used for fraction separation, the weight and analytical results related to all sub-samples obtained from fraction separation, as well as the “weighted” results.

A.3.2 Separation into different fractions

A.3.2.1 General

In cases of visible heterogeneity of separable fractions, the separation of different fractions may be necessary, especially if this can make subsequent particle size reduction, homogenisation and sub-sampling easier.

A.3.2.2 Manual separation

When to use it

— When several fractions can be distinguished;

— when non-crushable fractions (e.g. copper wire) are existing.

When not to use it

— When contamination or losses of analytes of interest may occur.

Procedure

Manually select macroscopic pieces of different nature and store them in separate containers, either by hand (with protective gloves) or by using appropriate tools (e.g. tweezers, magnet).

A.3.2.3 Sieving

When to use it

— When separation of fractions of different particle size is necessary;

— for checking the particle size of the sample or the particle size distribution.

When not to use it

— When contamination or losses of analytes of interest may occur.

Procedure

Sieve the sample by shaking either by hand or apparatus through sieves with appropriate mesh size and material.

A.4 Drying

A.4.1 General information

Depending on the nature of the sample and the specific requirements of the test portion, a drying step might be needed during sample treatment for test portion preparation. For the purpose of this European Standard, drying is only used to remove the amount of water that could interfere with test portion preparation (e.g. during crushing or milling). For the determination of water content a separate sub-sample may be necessary.

Drying is very likely to introduce analytical errors for volatile compounds, and should be avoided when not strictly required. If a sub-sample or test portion for volatile compounds determination is to be dried, the actual drying technique shall be selected in order to minimise losses of volatile compounds. The test report shall clearly state the technique(s) used for drying, along with the weight of sub-sample(s) before and after each drying step.

It is likely that a certain drying technique is not applicable for all requested determinations. In such cases, different sub-samples shall be dried in different ways, choosing the appropriate sequence of techniques for each one.

The drying time will depend on the technique chosen, the thickness of the layer of the sample, the nature of the sample, moisture content of the sample and of the air and the rate of ventilation.

The “grade of dryness” that shall be reached with the drying step depends on the subsequent treatments to be applied to the sample. Typically, it is not necessary to wait until constant weight: e.g. the sample shall be just dried enough to make crushing, grinding etc. possible.

A.4.2 Procedures

A.4.2.1 Air drying at room temperature

When to use it

— In every case where drying can be reached in appropriate time without alterations of analytes of interest;

— in case of mercury determination.

When not to use it

— When time is critical, and the properties of the sample do not allow a good drying in a reasonable time at room temperature, and a higher temperature drying step can be safely applied.

Procedure

Spread the sample on the trays in a thin layer and allow it to get dry enough. Care shall be taken in order to minimise possible contamination e.g. by dust. The use of a desiccator may accelerate the drying process for small amounts of (sub-)samples.

A.4.2.2 Oven drying at 40°C

When to use it

— When time is critical, and the properties of the sample do not allow for good drying in a reasonable time at room temperature;

— in case of mercury determination.

When not to use it

— When the properties of the sample do not allow for good drying in a reasonable time at this temperature, and a higher temperature drying step can be safely applied.

Procedure

Spread the sample on the trays in a thin layer and allow it to get dry in the oven at 40°C. Air renewal may accelerate the drying process, provided that it does not cause loss of dust-like particles.

Oven drying at other temperatures may be used if they fit with analytical procedures to be applied.

A.4.2.3 Oven drying at 105°C

When to use it

— On sub-sample for which mercury is not determined.

When not to use it

— In case of mercury determination;

— in cases where auto-ignition can be expected.

Procedure

Spread the sample on the trays in a thin layer and allow it to get dry in the oven at 105°C. Air renewal may accelerate the drying process, provided that it does not cause loss of dust-like particles.

A.5 Particle size reduction

A.5.1 General information

In order to achieve a homogeneous and representative test portion, one or more particle-size reduction steps might be needed. The choice of the technique to be used depends strongly on the nature of the sample and on the particle size needed.

Typically, particle-size reduction is a multi-step operation that implies the use of a sequence of different techniques; in some cases, it might be necessary to repeat a step until the sample reaches the requested particle size.

Particle-size reduction is a critical step in sample preparation because of potential loss of mercury due to heating, because of loss of dust-like material and because of contamination coming from the equipment itself or from other samples. Care shall be taken in selecting the appropriate equipment and keeping it clean.

The test report shall clearly state the technique(s) and operating conditions used for particle-size reduction. Non-crushable fractions (e.g. copper wire) shall be separated (according to A.3.2), weighed and, if needed, analysed as separate sub-samples.

A.5.2 Procedures

A.5.2.1 Crushing/grinding

When to use it

— When representative sub-samples cannot be taken because of large particle size;

— when the particle size of the sample is larger than the allowed inlet particle size for the milling or grinding equipment;

— when the analytical requirements demand a particle size in the mm order of magnitude.

When not to use it

— When not applicable because of the nature of the sample (e.g. soft materials);

— when contamination or losses of compounds of interest may occur by the equipment.

Procedure

Break large pieces of the sample and/or crush the sample with appropriate apparatus according to the manufacturer’s instructions to the desired particle size.

A.5.2.2 Freeze crushing

When to use it

— When the sample contains large amount of plastics;

— when representative sub-samples cannot be taken because of large particle size;

— when the particle size of the sample is larger than the allowed inlet particle size for the milling or grinding equipment;

— when the analytical requirements demand a particle size in the mm order of magnitude.

When not to use it

— When contamination or losses of compounds of interest may occur by the equipment.

Procedure

Wrap the sample in a polyethylene container. Fill a Dewar vessel with sufficient liquid nitrogen and immerse the wrapped sample in the liquid nitrogen. Allow the container to stand until the liquid nitrogen no longer boils vigorously. Cool for approximately 10 min. After complete cooling, retrieve the container from the liquid nitrogen and break large pieces of the sample with a hammer and/or crush the sample with appropriate apparatus according to the manufacturer's instructions to the desired particle size.

Alternatively commercial apparatus for freeze crushing are available.

A.5.2.3 Milling

When to use it

— When representative sub-samples cannot be taken because of large particle size;

— when the requested particle size is less than 1 mm.

When not to use it

— When the initial particle size is too coarse: a crushing step is required in these cases;

— when not applicable, because of the nature of the sample.

Procedure

Mill the sample according to the mill's manufacturers instructions until it reaches the desired particle size; in order to prevent losses of mercury, care shall be taken to avoid excessive heating of the sample during milling: a sequence of short and low-speed millings is to be preferred to a long and/or high-speed treatment; it is necessary to let the equipment cool down between each milling operation and the subsequent one. The use of a freeze-head mill can minimise the loss of mercury.

NOTE If the sample has a plastic consistency, freezing it down to low temperatures (e.g. –20°C to –30°C) can make it easier to mill.

A.5.2.4 Cutting

When to use it

— When representative sub-samples cannot be taken because of large particle size;

— when the material of the sample is not hard enough to allow crushing or milling, e.g. plastic, textile.

When not to use it

— When contamination with compounds of interest may occur by the equipment.

Procedure

Cut the sample according to the cutting mill's manufacturers instructions to the desired particle size; in order to prevent losses of mercury care shall be taken to avoid excessive heating of the sample during cutting; it is necessary to let the equipment cool down between each cutting operation and the subsequent one. To minimise loss of mercury a cooled cutting mill should be used or manual cutting by scissors may minimize the loss.

A.5.2.5 Freeze cutting

When to use it

— When the sample has a plastic or fibred consistency;

— when representative sub-samples cannot be taken because of large particle size;

— when the analytical requirements demand a particle size in the mm order of magnitude.

When not to use it

— When contamination from the equipment may occur.

Procedure

If necessary, wrap the sample in a polyethylene container. Fill a Dewar vessel with sufficient liquid nitrogen and immerse the (wrapped) sample in the liquid nitrogen. Allow the container to stand until the liquid nitrogen no longer boils vigorously. Cool for approximately 10 min. After complete cooling, retrieve the sample from the liquid nitrogen, cut it with a preferably cooled cutting mill according to the manufacturer's instructions to the desired particle size.

A.6 Sub-sampling

A.6.1 General information

The laboratory sample usually has to be divided into different test portions for the analyses. Most analytical techniques allow only small quantities of test portions to be analysed. Laboratory samples, conversely, can be in some cases very large, so only a small, representative portion of them shall be taken for analysis.

The sub-sampling shall be performed in a way that the obtained sub-samples are as representative as possible, taking into account the quantity of test portion to be analysed, the quantity of laboratory sample, particle size and homogeneity. If the sample is not homogeneous enough, or if particle size is too coarse, a representative portion cannot be taken: one or more particle size reduction steps followed by homogenisation steps are needed.

For determination of the minimum amount of sample in relation to the particle size and the heterogeneity of the sample the information in Annex B should be taken into account.

NOTE Because of practical limitations it might happen in some cases that representative sub-samples cannot be obtained. In this case replicates should be performed.

The test report shall clearly state the way of determination of minimum amount of sub-samples and the technique(s) used for sub-sampling, along with the weight of each analysed fraction.

A.6.2 Manual division of solid samples by quartering

When to use it

— In case of a large amount of laboratory sample;

— when the sample is dry enough and there are no clods in it;

— if there is no evidence of heterogeneous distribution of chemical constituents.

When not to use it

— When the sample contains clods that cannot be divided.

Procedure

Spread the sample on a flat surface coated by an inert sheet in a circular shape; mix it with a shovel and make a cone.

— Divide the sample in four slices, e.g. with a sheet metal cross, of the same shape and size, as shown in this figure:

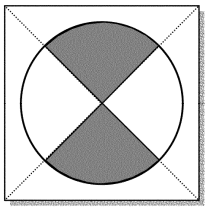


Figure A.1 — Division by quartering

— discard two opposite slices, and mix thoroughly the remaining part of the sample.

This sequence shall be repeated until the requested quantity of sub-sample is reached, provided that the obtained sub-sample is still representative of the original sample.

A.6.3 Mechanical division of solid samples

When to use it

— When the sample is dry enough and there are no clods in it;

— when the particle size is small enough for the used apparatus;

— if there is no evidence of heterogeneous distribution of chemical constituents.

When not to use it

— When the sample contains clods that cannot be divided.

Procedure

Mechanical sample splitters can achieve similar or better results compared to manual division. Divide the sample by using the sample splitter, e.g. riffle box, tyler divider, according to the manufacturer's instructions.

Annex B
(informative)

Relationship between minimum amount of sample and particle size - Equation for the estimation of the minimum amount of sample

The minimum amount of sample can be estimated by the following equation:

$$M_{\text{sam}} = \frac{1}{6}\pi \times (D_{95})^3 \times f \times \rho \times g \times \frac{(1-p)}{CV^2 \times p}$$

where

M_{sam} is the mass of the sample in g;

D_{95} is the 'maximum' particle size (defined as the 95-percentile), in cm;

f is the shape factor (for materials which are granular-like or smaller than 50 mm, a value of 1 can be assumed);

This value shall be determined by the following equation:

$$f = \frac{V_{95}}{d_{95l}{}^3}$$

where

f is the shape factor, in cm^3/cm^3;

V_{95} is the "maximum" volume of a particle (a mass fraction of 95% of the particles are smaller than V_{95}), in cm^3 (where V = l x b x h);

d_{95l} is the "maximum" length of a particle (a mass fraction of 95% of the particles are smaller than d_{95l}), in cm;

The shape factor is not constant but depends on the type of material. The shape factor generally increases if a material is comminuted.

ρ is the average density of the particles in the material, in g/cm^3;

NOTE 1 This parameter does not represent the bulk density of the sample, but the average density of all particles.

g is the correction factor for the particle size distribution of the material;

This value depends on the ratio between D_{95}/D_{05} and gives an indication for the particle size distribution. A D_{95}/D_{05} value close to 1 reflects a narrow particle size distribution (most particles of equal size), a high D_{95}/D_{05} value (> 4) reflects a broad particle size distribution.

D95 is the “maximum” particle size (a mass fraction of 95% of the particles are smaller than D95), and D05 is the “minimum” particle size (a mass fraction of 5% of the particles are smaller than D05).

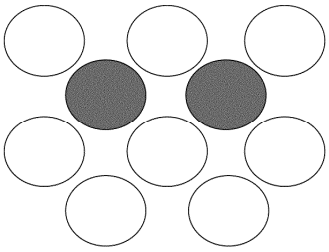
Table B.1 shall be used for choosing the correct “g” value:

Table B.1 — Choice of g value

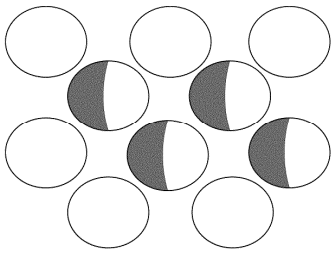
Ratio D_{95}/D_{05}	***g***
$D_{95}/D_{05} > 4$	*0,25*
$4 \geq D_{95}/D_{05} \geq 2$	*0,50*
$2 > D_{95}/D_{05} > 1$	*0,75*
$D_{95}/D_{05} = 1$	*1,00*

p is the fraction of the particles with the property of interest (m/m);

NOTE 2 If every single particle of the sample would carry the same amount of the parameter of interest, $p = 1$.



Example 1: the sample is made of 10 particles, and two of them carry the analyte of interest. p = 0,2.



Example 2: the sample is made of 10 particles, and four of them carry the analyte of interest (the total amount of the analyte is the same as example 1). p = 0,4.

Figure B.1 — Distribution of parameters in the sample

CV is the desired coefficient of variation caused by the fundamental error.

NOTE 3 A typical value for CV is 0,1. Depending of the nature of the sample and the aim of the analysis it may vary from 0,1 to 0,3.

EXAMPLE

Assumptions:

CV = 0,1

g = 0,25

f = 1 (see Note 5)

ρ_p = 1 g/cm³

Table B.2 — Minimum test portion/sample size, M_{sam} in g

p		D_{95} (cm)						
		10	4	1	0,4	0,2	0,1	0,02
very heterogeneous (traces)	0,001	13 070 250	836 496	13 070	836	104,6	13,07	0,105
heterogeneous (minor constituents)	0,02	641 083	41 029	641	41	5,1	0,64	0,005
rather homogeneous (major constituents)	0,1	117 750	7 536	118	8	0,9	0,12	0,001

NOTE 4 The rate of heterogeneity is dependent on the parameters of interest and may vary for one sample – (e.g. for major constituents p ~ 0,1 and for trace constituents p ~ 0,001). If the same sample or aliquot is to be used for determination of different parameters, the most conservative (lowest) value shall be used.

NOTE 5 In the above table the shape factor *f* has been assumed to be fixed to the most conservative value (*f* = 1); for cases with coarse particle size, it might have sense to evaluate the actual shape factor, possibly resulting to smaller amounts of sample needed, especially for fiber- or fluff- like materials.

Annex C
(informative)

Sample treatment equipment

The preparation of test samples from a laboratory sample will frequently involve a sequence of operations such as homogenisation, phase/fraction separation, drying, particle size reduction and sub-sampling. Equipment to be used for these operations is listed below.

Equipment	A.2 homogenisation	A.3 phase/fraction separation	A.4 drying	A.5 particle size reduction	A.6 sub-sampling
mill	X			X	
freeze-head mill	X			X	
shredder	X			X	
cutting device				X	
mechanical mixer	X				
hammer				X	
jaw-crusher				X	
pestle and mortar	X			X	
shovel	X				X
sheet cross					X
tweezer		X			
magnet		X			
riffle box					X
tyler divider					X
sieves		X		X	X
drying oven			X		
freeze drier			X		
desiccator			X		
balance		X	X	X	X

Annex D
(normative)

Guidelines - Characteristics of the laboratory sample for chemical analysis of SRF

The following requirements apply when preparing the laboratory sample for the chemical characterisation of SRF samples according to this European Standard.

NOTE Equivalent requirements apply in all chemical test method specifications for SRF, i.e. EN 15407, EN 15408, EN 15410, EN 15411, CEN/TS 15412 and EN 15413.

A maximum amount of laboratory sample of 10 kg and maximum particle size of 1 cm is established on the basis of number and type of parameters to be determined, sample representativity and practical reasons for handling samples. In the following table the requirements are summarised both for single or grouped chemical parameters.

Table D.1 — Requirements for the laboratory sample for the analysis of SRF

Parameter (single or group)	Minimum laboratory sample amount (g) [a]	Short term storage conditions before delivery to the lab	Long term storage condition before delivery to the lab	Container material
C, H, N	100	In the same condition of SRF production	refrigeration 4°C	plastic bottle or bag
Cl, S, Br, F	100	In the same condition of SRF production	refrigeration 4°C	non-PVC plastic bottle or bag
Metallic Al	200	In the same condition of SRF production	refrigeration 4°C	plastic bottle or bag
Major elements	400	In the same condition of SRF production	refrigeration 4°C	plastic bottle or bag
Trace elements excluding Hg	200	In the same condition of SRF production	refrigeration 4°C	plastic bottle or bag
Hg	100	In the same condition of SRF production	refrigeration 4°C	Glass or PFA bottle
C, H, N, Cl, S, Br, F	150	In the same condition of SRF production	refrigeration 4°C	non-PVC plastic bottle or bag
Major elements + Trace elements excluding Hg	500	In the same condition of SRF production	refrigeration 4°C	plastic bottle or bag
Major elements + Trace elements + Hg	600	In the same condition of SRF production	refrigeration 4°C	Glass bottle (100 g) + plastic bottle or bag

Table D.1 (*concluded*)

Major elements + Trace elements + Hg + metallic Al	700	In the same condition of SRF production	refrigeration 4°C	Glass bottle (100 g) + plastic bottle or bag
Complete analysis	800	In the same condition of SRF production	refrigeration 4°C	Glass bottle (100 g) + non-PVC plastic bottle or bag
a The maximum particle size (mm) is related to the laboratory sample amount (g) in order to guarantee sample homogeneity. It is established following the rules reported in previous sections of this European Standard.				

Annex E
(informative)

Results of ruggedness testing

E.1 Abstract

This annex describes the evaluation of the robustness of the “Methods for the preparation of the test sample from the laboratory sample”.

Robustness testing is carried out by applying the procedure to be validated with some controlled variations of key parameters in repeatability conditions, in order to evaluate the influence of those parameters on the quality of the final results. The key parameters chosen are the composition, tested on two different kinds of materials and the influence of particle size reduction system.

Robust methods are more likely to return repeatable and reproducible results.

The statistical formula of Annex B has been proven to be a useful tool for the user in finding the best compromise between the mass of (sub) sample to be analyzed, its main physical characteristics and the expected fundamental error. Anyway, the choice of factor *p* cannot be limited to the relative abundance of the analytes of interest, because there might be some dramatic exceptions of “heterogeneous” major components. Therefore, a good knowledge of the sample and its nature is necessary.

The applicability of the method has been successfully tested on two materials, with different levels of heterogeneity.

Cutting mill performs better than rotor beater mill with respect to recovery rate, recovery of volatile compounds and resulting homogeneity.

E.2 Preparation of samples and application of statistical formula

The samples chosen for the study were:

- **QR-D:** Fluff-like SRF from Municipal Solid Waste (MSW) containing small pieces of tyre residues, passing through a 10 cm grid.

- **QR-B:** SRF produced from demolition wood.

For the application of the statistical formula one kg of material QR-D has been processed, obtaining 16 sub-samples of about 50 g each by manual quartering. Five of these sub-samples (labelled A, D, G, L, O) have been used for the verification of the statistical formula, by a two step particle size reduction by cutting mill to 2 mm and by centrifugal mill to 0,5 mm. Table E.1 shows the expected CVs as a function of p-factor for test samples (50 g) and test portions (1 g) of the material after particle size reduction.

Table E.1 — Application of statistical formula on the sample QR-D

Test samples: D_{95} = 0,95 cm, *f* = 0,0328, M = 50 g				
p	0,1	0,01	0,005	0,001
CV	0,0365	0,12	0,17	0,38
Test portions: D_{95} = 0,05 cm, *f* = 0,46, M = 1 g				
p	0,1	0,01	0,005	0,001
CV	0,02	0,07	0,09	0,21

Analyses (moisture, ash, elements and carbon) have been performed on six replicates for each test sample A, D, G, L, O; averages have been then used to calculate the average and RSD% (Relative Standard Deviation) among test samples (grand mean and RSD%). Results are reported in Table E.2. Colours have been used in order to better mark some RSD that exceed the expected values according to the statistical formula.

Table E.2 — Results for application and verification of the statistical formula (concentrations in mg/kg where not specified differently)

	Test sample A		Test sample D		Test sample G		Test sample L		Test sample O	
	Mean	*RSD %*	Mean	*RSD %*	Mean	*RSD %*	Mean	*RSD %*	Mean	*RSD %*
% Moi	3,91	*4,5*	3,03	***12,9***	3,16	*8,3*	3,22	*7,7*	3,01	*8,2*
% Ash	13,8	*1,6*	14,8	*6,6*	12,9	*2,5*	13,7	*7,6*	14,0	*4,2*
% C	46,5	4,6	47,3	3,2	48,8	4,7	47,7	3,7	47,9	4,2
Al	12 844	***16,5***	16 069	***34,7***	15 266	***25,7***	12 250	***15,4***	15 617	***19,6***
Ba	138,8	***20,0***	169,9	***44,9***	223,5	***70,9***	167,9	***19,6***	479,6	***42,3***
Ca	29 044	*5,7*	25 664	***19,7***	26 480	*6,2*	26 653	*1,9*	25 568	*7,2*
Cr	131,1	***20,9***	218,7	***25,8***	162,7	***23,0***	312,6	*7,5*	296,5	***21,8***
Cu	1 848,5	***83,2***	647,0	***95,5***	1 014,9	***112,5***	4 335,1	***168,1***	192,4	***128,8***
Fe	3 645	***16,4***	3 111	***39,4***	2 336	*5,1*	2 505	*7,6*	5 395	***42,1***
K	4 631	*4,5*	3 851	***23,4***	4 494	*5,6*	5 001	*6,8*	5 509	*7,6*
Mg	2 516	*7,0*	2 416	***20,6***	2 330	*6,2*	2 421	*5,4*	2 189	*7,0*
Mn	88,9	*16,4*	81,2	***24,6***	85,7	***23,2***	85,6	*8,9*	88,1	*7,9*
Na	4 035	*3,0*	3 419	***21,3***	3 261	*6,2*	3 380	*4,7*	3 296	*6,5*
Ni	32,7	*13,2*	28,7	*31,3*	15,5	*17,4*	23,2	*29,5*	34,1	*21,0*
Si	15 887	*3,7*	19 189	***21,9***	15 589	*6,1*	16 762	*4,4*	17 307	*8,3*
Sr	38,6	*5,7*	35,7	*25,0*	36,2	*12,4*	35,2	*3,5*	40,0	*9,1*
Ti	2 357	*6,1*	4 208	***20,7***	1 824	*6,3*	1 787	*5,7*	3 911	***13,8***
W	22,9	*16,8*	18,4	*21,7*	19,7	*22,3*	37,0	*8,1*	21,4	*10,4*
Zn	133,5	***22,5***	231,8	***25,7***	182,8	***23,3***	347,0	*9,9*	324,3	***22,7***

Color Key	***White: Good/acceptable RSD***	***Light yellow: RSD slightly higher than expected***	***Light brown: RSD much higher than expected***	***Orange: RSD extremely high***

It is necessary to consider that overall variability of such results includes the uncertainties associated with (sub)sampling, preparation and analysis; generally, the RSD associated with preparation and analysis can be estimated to about 5% for most of components determined above, i.e. all CVs associated with sampling are less than those shown here. By looking at Table E.2, some considerations can be drawn:

- For several major components (i.e. ash, moisture, C, Ca, K, Mg, Na, Si), the RSD associated with (sub) sampling is about 10% or less, as expected from the statistical formula with factor ***p*** from 0,1 to 0,05.

- For other major elements like Al, and mostly Cu, Fe and Ti, RSD is above 25%, despite their high concentration. Even an application of factor ***p*** = 0,01 appear to be inadequate for these cases.

- For minor elements like Mn, Ni, Sr, and W the RSD is in the range 10% - 30% as expected from the application of statistical formula with ***p*** = 0,001; Cr, Zn and mostly Ba show higher RSD.

As expected, the statistical formula helps the user in finding the best compromise between the mass of (sub) sample to be analyzed, its main physical characteristics (i.e. particle size and shape), and the expected fundamental error associated with (sub) sampling. But, for its correct application, criteria for the choice of factor ***p*** cannot be limited to the relative abundance of the analytes of interest (the higher concentration → the higher ***p***), because there might be some dramatic exceptions of "heterogeneous" major components (like Cu, Al, Fe in this case). *Therefore, a good knowledge of the sample and its nature is necessary*.

E.3 Evaluation of influence of particle size reduction systems

E.3.1 General

The evaluation of the influence of particle size reduction system has been carried out by means of use of three different systems on the two different SRF samples.

The three different treatment sequences, named "low", "mid" and "high" stress processing are respectively:

- **Low stress** processing: particle size reduction to 2 mm by cutting mill SM 2000, followed by further particle size reduction to 0,5 mm, by centrifugal mill ZR-1.

- **Mid stress** processing: particle size reduction to 2 mm by rotor beater mill SR 300, followed by further particle size reduction to 0,5 mm, by centrifugal mill ZR-1.

- **High stress** processing: particle size reduction to 6 mm by rotor beater mill SR 300, followed by further particle size reduction to 0,5 mm by centrifugal mill ZR-1.

E.3.2 Tests on QR-D sample (municipal solid waste)

Nine of the sixteen test samples prepared as described in E.2 have been used for evaluation of robustness, by submitting them to the three different treatment sequences: B, C, E for low stress processing; F, I, M, for mid stress processing; P, Q, R for high stress processing.

After each step of treatment during "low", "high" and "mid" stress processing, the temperature inside the mills have been measured, and sub-samples have been weighed. The increment of temperature of the cutting devices were practically negligible in the case of cutting mill SM 2000, less than 10°C for rotor beater mill SR 300 and less than 20°C for centrifugal mill ZM-1. Moreover, milling duration has been checked in the range from 15' to 45': it did not induce any significant variation of temperature. For each procedure the mean mass recovery (R%) has been calculated and reported in Table E.3.

Table E.3 — Mass recovery on test samples QR-D

	1st step		2nd step	
	Device	Recovery %	Device	Recovery %
Low	SM 2000 2 mm	96	ZM-1 0,5 mm	96
Mid	SR 300 2 mm	82	ZM-1 0,5 mm	96
High	SR 300 6 mm	86	ZM-1 0,5 mm	95

Recovery rate is almost quantitative (about 95%) for cutting mill used in “low” stress processing, while 15% or more of the mass is lost (as dispersed dust) with the rotor beater mill used in “high” and “mid” stress processing). The ZM-1 centrifugal mill caused about 5% of mass loss, independently from the kind of processing previously applied to the sub-sample.

On each test sample, the following analyses have been performed in 15 independent replicates each: CHN, direct determination of Hg, moisture, ash and elements. The average concentrations and a qualitative evaluation of macro-homogeneity, i.e. the variability among the different 50 g test samples for each treatment procedure is shown in Table E.4.

Table E.4 — Average and RSD% of test samples for evaluation of macro-homogeneity on QR-D

		Low stress		Mid stress		High stress	
		Mean	***RSD%***	**Mean**	***RSD%***	**Mean**	***RSD%***
Moi	%	2,25	***20,5***	1,8	*10,9*	2,04	*8,2*
Ash	%	14	*0,5*	13,86	*3,3*	14,21	*6,5*
C	%	50,6	*2,2*	51,6	*4,1*	50,6	*2,5*
H	%	5,9	*4,0*	5,5	*4,8*	5,4	*3,1*
N	%	1,0	*8,5*	1,06	*0,9*	0,92	*7,1*
Al	mg/Kg	20 457	*9,5*	19 258	***11,7***	17 932	*9,8*
Ba	mg/Kg	352,7	***51,8***	504,3	***110,7***	299,4	***89,2***
Ca	mg/Kg	26 954	***11,5***	25 495	*1,8*	23 065	***10,8***
Cr	mg/Kg	155,1	***31***	238,7	***24,6***	106,4	***35,4***
Cu	mg/Kg	1 472,4	***117***	878,9	***58,9***	509,4	***44,8***
Fe	mg/Kg	4 198	***13,4***	4 647	***28,5***	5 134	***35,1***
Hg	µg/Kg	476,8	*11,6*	474,5	*13,6*	488,3	*12,5*
K	mg/Kg	5 981	***18,4***	5 636	*9,7*	4 995	*7*
Mg	mg/Kg	2 815	***11,7***	2 522	*2,5*	2 368	*5*
Mn	mg/Kg	109,1	*12,8*	104,6	*12,6*	97,9	*13,3*
Na	mg/Kg	3 293	*6,2*	3 307	*5,5*	3 035	***11,8***
Si	mg/Kg	20 164	*1,5*	18 532	*5,1*	16 919	*7*
Sr	mg/Kg	47	*11,8*	53,3	***36,4***	37,5	*18,2*
Ti	mg/Kg	1 484	***14,6***	1 560	*2,9*	1 463	*5,1*
Zn	mg/Kg	162,2	***33,4***	252,9	***26,3***	107,6	***33,6***

Color Key	***RSD slightly higher than expected***	***RSD much higher than expected***	***Extremely high RSD***

For evaluation of variability, one should keep in mind that it includes the uncertainties associated with sampling, preparation and analysis. Determination of moisture, ash content and elements has been carried out using the whole amount of test sample, so sampling error in these cases is not an issue.

A qualitative comparison of the above values shows that “low”, “high” and “mid” stress procedures do not differ significantly in the resulting macro-homogeneity.

Almost all these analyses imply the use of a test portion in the order of magnitude of 1 g, that is suitable for a qualitative evaluation of micro-homogeneity: for each test sample the variability between the 15 test portions of about 1 g each is listed in Table E.5.

Table E.5 — RSD% between 15 replicates for evaluation of micro-homogeneity on QR-D

	Low stress			Mid stress			High stress		
	B	**C**	**E**	**F**	**I**	**M**	**P**	**Q**	**R**
Moi	15	8	6	9	9	8	9	8	10
Ash	3	3	3	3	3	3	5	3	3
C	3	3	3	4	5	5	5	6	4
H	5	3	5	3	4	5	3	3	3
N	7	7	10	14	11	8	7	14	9
Al	12	16	17	15	9	11	19	14	13
Ba	13	24	18	18	16	32	18	15	8
Ca	9	10	8	5	8	5	4	6	7
Cr	10	16	11	19	6	12	11	11	10
Cu	60	92	28	69	52	46	91	118	92
Fe	23	21	20	29	13	19	18	19	18
Hg	19	7	5	21	8	8	6	14	10
K	16	13	11	26	11	6	8	9	10
Mg	7	9	8	8	6	5	4	5	6
Mn	7	11	15	13	8	6	9	7	10
Na	10	10	9	7	7	5	4	7	8
Si	9	10	11	5	5	6	6	4	6
Sr	10	16	11	9	7	8	6	6	8
Ti	10	9	8	7	6	5	4	4	5
Zn	12	16	12	20	7	11	9	9	9

A qualitative comparison of the above values shows that “low”, “high” and “mid” stress procedures do not differ significantly in the resulting micro-homogeneity as well.

E.3.3 Tests on QR-B sample (demolition wood)

The test samples for QR-B have been prepared according to the same scheme as for QR-D, described in E.2. The nine samples used for evaluation of robustness, submitted to the three different treatment sequences, are: C, E, G for low stress processing; I, L, M, for mid stress processing; N, R, S for high stress processing.

For each treatment procedure, the temperatures and mass recovery have been measured; for temperatures, the same considerations already written for QR-D apply; for mass recovery there are some small differences, as one can see by looking to Table E.6.

Table E.6 — Mass recovery on test samples QR-B

	1st step		2nd step	
	Device	Recovery %	Device	Recovery %
Low	SM 2000 2 mm	95	ZM-1 0,5 mm	89
Mid	SR 300 2 mm	94	ZM-1 0,5 mm	86
High	SR 300 6 mm	87	ZM-1 0,5 mm	89

The differences in the recovery of second step can be due to the nature of samples QR-D and QR-B, the latter being more subject to develop fine dust-like particles.

On each test sample of QR-B determination of moisture, ash and elements have been performed in 6 independent replicates each; direct determination of Hg has been done in 12 replicates.

The statistical formula of Annex B has been applied with appropriate values for QR-B. Table E.7 shows the expected CV for macro and micro homogeneity, where macro-homogeneity represents the variability among the different 55 g test samples for each treatment procedure, and the micro-homogeneity represents the variability between the smaller test portions of about 1 g each.

Table E.7 — Application of statistical formula to the sample QR-B

Test samples: D_{95} = 0,95 cm, f = 0,000 8, $\rho = 0.7$, g = 0,25, M = 55 g				
p	0,1	0,01	0,005	0,001
CV	0,01	0,03	0,05	0,11
Test portions: D_{95} = 0,05 cm, f = 0,8, $\rho = 0.7$, g = 0,75, M = 1 g				
p	0,1	0,01	0,005	0,001
CV	0,02	0,05	0,07	0,17

A qualitative evaluation of macro-homogeneity can be done by looking at Table E.8.

Table E.8 — Average and RSD% of test samples for evaluation of macro-homogeneity on QR-B

		Low stress		Mid stress		High stress	
		Mean	***RSD%***	**Mean**	***RSD%***	**Mean**	***RSD%***
Moi	%	7,27	2,6	6,28	4,7	6,23	**9,1**
Ash	%	1,80	3,9	2,09	**24,3**	1,71	**19,7**
Al	mg/kg	334	**18,2**	277	**24,4**	222	15,2
Ba	mg/kg	99	**20,1**	117	**39,0**	68	**39,9**
Ca	mg/kg	1 788	7,5	2 260	9,5	1 884	**12,7**
Cr	mg/kg	61	15,7	54	**44,0**	62	**46,9**
Cu	mg/kg	35	13,3	36	**63,2**	32	**59,0**
Fe	mg/kg	295	4,6	349	**28,0**	245	**32,7**
K	mg/kg	831	7,8	954	7,5	780	2,9
Mg	mg/kg	4 588	**16,8**	7 004	**32,9**	4 882	**26,3**
Mn	mg/kg	72	3,3	78	6,5	75	5,9
Na	mg/kg	242	4,5	342	**29,1**	246	**30,2**
P	mg/kg	78	**25,6**	86	12,8	66	2,7
Pb	mg/kg	46	**124,1**	15	13,3	14	**72,3**
Si	mg/kg	1 427	**19,4**	1 374	**24,4**	885	**23,7**
Ti	mg/kg	387	4,2	355	**25,3**	317	15,0
Zn	mg/kg	54	12,3	54	**43,0**	56	**48,1**
Hg	µg/kg	14,53	18,7	28,43	71,3	11,17	17,0

Color Key	***RSD slightly higher than expected***	***RSD much higher than expected***	***Extremely high RSD***

The mid- and high- stress processing could lead to some loss of volatiles: moisture values are significantly different between the low-stress processed samples (i.e. those treated with the cutting mill) and both other series of samples (treated with the rotor beater mill).

For most parameters, the variability is significantly affected by the processing; for the resulting macro-homogeneity, “low” stress procedure shows generally lower variability compared to “high” and “mid” stress procedures. A notable exception regards Pb, whose variability among low-stress replicates appear to be very high; by looking to individual results (not reported here), only a test sample has a content of Pb ten times higher than all others; this can be explained by the possible presence of a single “fragment” of highly contaminated wood that segregated only in that test sample: the variability of Pb appears not to be due to the processing but to the sample nature itself.

An evaluation of micro-homogeneity can be done by looking at RSD% reported in Table E.9.

Table E.9 — RSD% between replicates for evaluation of micro-homogeneity on QR-B

	Low stress			Mid stress			High stress		
	C	**E**	**G**	**N**	**R**	**S**	**I**	**L**	**M**
Moi	1	1	1	1	1	1	1	1	2
Ash	5	3	2	3	3	2	4	3	2
Al	12	4	14	4	15	6	30	10	29
Ba	8	5	5	5	11	7	11	11	7
Ca	8	6	7	4	10	3	11	8	6
Cr	9	9	9	18	8	7	44	31	10
Cu	10	4	16	26	17	114	19	34	12
Fe	8	8	7	12	34	23	36	21	15
K	12	3	7	9	16	10	16	15	10
Mg	10	3	8	5	14	3	11	9	5
Mn	8	5	3	6	14	7	12	8	10
Na	6	10	5	4	10	9	13	13	8
P	11	6	4	5	15	12	13	7	8
Pb	18	18	9	9	15	10	15	30	22
Si	11	4	10	4	14	6	12	9	6
Ti	8	7	4	6	14	9	12	9	14
Zn	9	7	7	14	11	7	38	22	8
Hg	30	14	20	14	13	10	20	12	7

A qualitative comparison of these values shows that for each parameter there are some significant differences due to the processing: “high stress” processing shows generally higher variability between test portions.

E.4 Conclusions

The statistical formula of Annex B has been proven to be a useful tool for the user in finding the best compromise between the mass of (sub) sample to be analyzed, its main physical characteristics and the expected fundamental error. Nevertheless, the choice of factor *p* cannot be limited to the relative abundance of the analytes of interest, because there might be some dramatic exceptions of “heterogeneous” major components. Therefore, a good knowledge of the sample and its nature is necessary.

Several particle size reduction techniques have been tried on sample QR-D, including milling, freeze-milling and cutting with different devices. Milling has never been applicable, both to the present sample and after freezing the sample in liquid nitrogen for several minutes. A single step particle size reduction to 0,5 mm has also been tried, resulting as not applicable due to the excessive heating that leads the sample to burning. Cutting has been the only applicable technique in this case for particle size reduction.

The applicability of the European Standard has been successfully tested on two materials.

QR-D (municipal solid waste) has a very high intrinsic heterogeneity, so that some effects of treatment have been hidden or not clearly shown; QR-B (demolition wood), on the other hand, is made up of a less complex matrix so some results are more clearly evident, especially those about loss of analytes and resulting homogeneity of the treated samples.

Low stress processing (cutting mill) performs better than mid- and high-stress processing (rotor beater mill) with respect to recovery rate, recovery of volatile compounds and resulting homogeneity.

Bibliography

[1] EN 15407, *Solid recovered fuels — Methods for the determination of carbon (C), hydrogen (H) and nitrogen (N) content*

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[3] EN 15410, *Solid recovered fuels — Methods for the determination of the content of major elements (Al, Ca, Fe, K, Mg, Na, P, Si, Ti)*

[4] EN 15411, *Solid recovered fuels — Methods for the determination of the content of trace elements (As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Tl, V and Zn)*

[5] CEN/TS 15412, *Solid recovered fuels — Methods for the determination of metallic aluminium*

[6] EN 15442, *Solid recovered fuels — Methods for sampling*

[7] EN 15002:2006, *Characterization of waste — Preparation of test portions from the laboratory sample*

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