BSI Standards Publication

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

bsi.

...making excellence a habit.™

National foreword

This British Standard is the UK implementation of EN 15443:2011. It supersedes DD CEN/TS 15443:2006 which is withdrawn.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.

© BSI 2011

ISBN 978 0 580 67496 9

ICS 75.160.10

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

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2011.

Amendments issued since publication

Date	Text affected

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15443

March 2011

ICS 75.160.10

English Version

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

Combustibles solides de récupération - Méthodes de
préparation des échantillons de laboratoire

Feste Sekundärbrennstoffe - Verfahren zur Herstellung von
Laboratoriumsproben

This European Standard was approved by CEN on 22 January 2011.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of 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 United Kingdom.

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 15443:2011: E

Contents

Page

Foreword

This document (EN 15443:2011) 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 September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

This document supersedes CEN/TS 15443:2006.

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 has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This European Standard is one of series of European Standards dealing with sampling solid recovered fuel.

EN 15442, *Solid recovered fuels — Methods for sampling.*

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

This document differs from CEN/TS 15443:2006 mainly as follows:

a)   results of interlaboratory tests supplemented as an informative Annex D;

b)   whole document editorially revised.

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 United Kingdom.

Introduction

Solid recovered fuels (SRF's) are a major source of renewable energy. European Standards are needed for production, trade and use of solid recovered fuels. For sampling and sample preparation of solid recovered fuels the following European Standards can be used:

EN 15442, *Solid recovered fuels — Methods for sampling;*

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

These European Standards can be used by production and trading of solid recovered fuels. They are also useful for buyers of solid recovered fuels, regulators, controllers and laboratories.

Figure 1 shows the links between the essential elements of a testing program.

The sample preparation technique adopted depends on a combination of different characteristics of the material and circumstances encountered at the sampling location. The determining factors are:

— the type of solid recovered fuel;

— the physical behaviour of the specific solid recovered fuel;

— the (expected) degree of heterogeneity (e.g. monostreams, mixed fuels, blended fuels).

For the sample preparation of solid biofuels prEN 14780 is available [1]. For the characterization of waste EN 15002 is available [2].
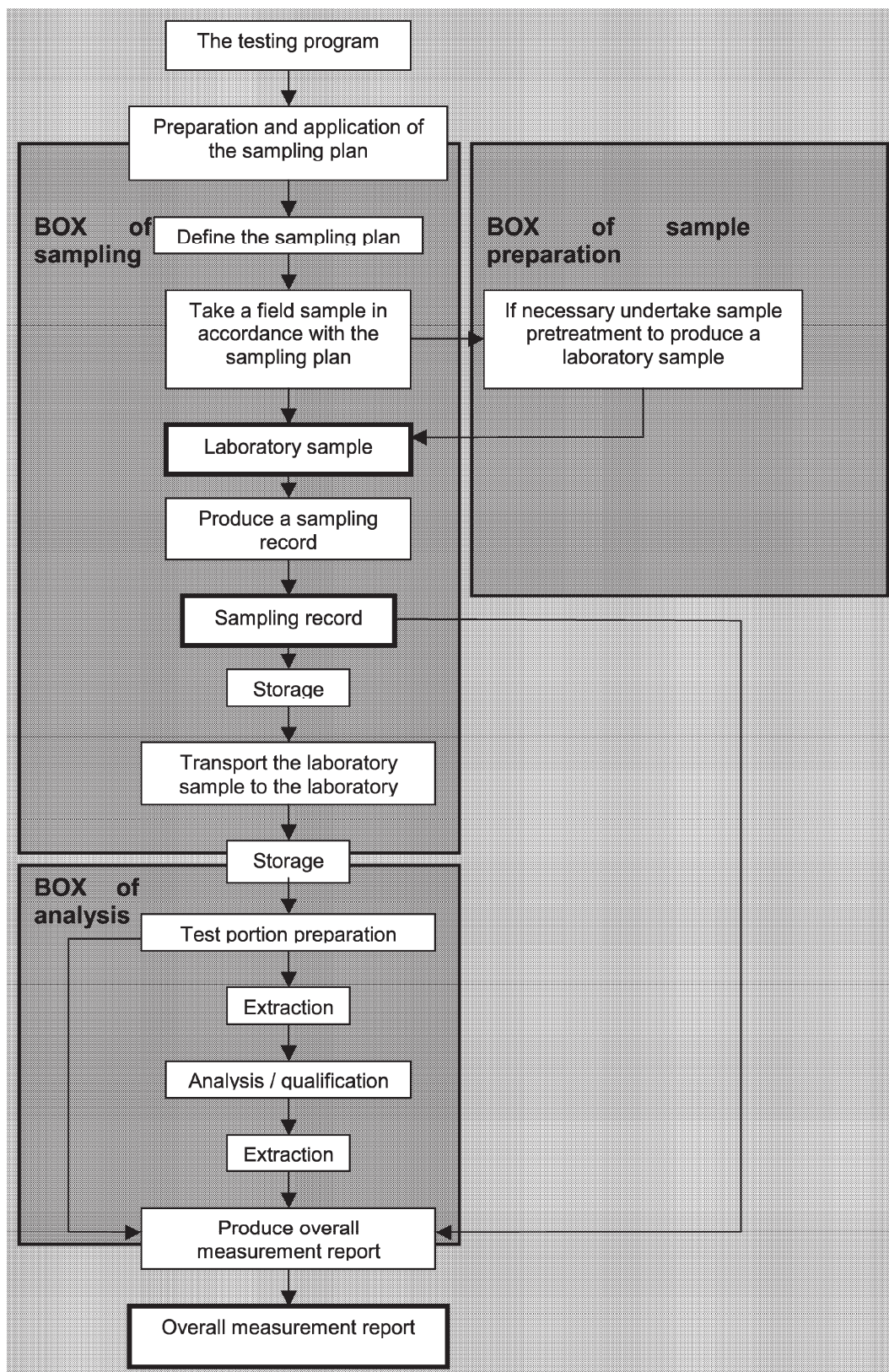
Figure 1 — Links between the essential elements of a testing program

1 Scope

This European Standard specifies methods for reducing combined samples to laboratory samples and laboratory samples to sub-samples and general analysis samples.

The methods described in this European Standard may be used for sample preparation, for example, when the samples are to be tested for bulk density, biomass determination, durability, particle size distribution, moisture content, ash content, ash melting behaviour, calorific value, chemical composition, and impurities. The methods are not intended to be applied to the very large samples required for the testing of bridging properties.

2 Normative references

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

EN 15297, *Solid biofuels — Determination of minor elements — As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V and Zn*

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

CEN/TS 15414-1:2010, *Solid recovered fuels —Determination of moisture content using the oven dry method — Part 1: Determination of total moisture by a reference method*

CEN/TS 15414-2:2010, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 2: Determination of total moisture by a simplified method*

EN 15414-3, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

EN 15415-1[1], *Solid recovered fuels — Determination of particle size and particle size distribution — Part 1: Screen method for small dimension particles*

EN 15442, *Solid recovered fuels — Methods for sampling*

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

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

3.2
general analysis sample
sub-sample of a laboratory sample having a nominal top size of 1 mm or less and used for a number of chemical and physical analyses

3.3
increment
portion of solid recovered fuel extracted in a single operation of the sampling device

---

[1] To be published.

3.4
laboratory sample
sample sent to or received by the laboratory

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 may 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.

3.5
lot
defined quantity of fuel for which the quality is to be determined

3.6
moisture analysis sample
sample taken specifically for the purpose of determining total moisture

3.7
nominal top size
d_{95}
aperture size of the sieve used in EN 15415-1 through which at least 95 % by mass of the material passes

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

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

3.10
sample division
reduction of the mass of a sample or sub-sample

3.11
sub-sample
portion of a sample

3.12
test portion
sub-sample of a laboratory sample consisting of the quantity of material required for a single execution of a test method

4   Symbols and abbreviations

For the purposes of this document, the following symbols and abbreviated terms apply.

α is a constant in third power law

d_{95} is the nominal top size in mm

m is the mass of a sample in gram

M is moisture in percent by weight

f is the shape factor

5   Principles of correct sample preparation

The main purpose of sample preparation is that a sample is reduced to one or more test portions that are in general smaller than the original sample. The main principle for sample preparation is that the composition of the sample as taken on site shall not be changed during each step of the sample preparation and that possible requirements of the analysis methods to be performed are obeyed. Each sub-sample shall be representative for the original sample. To reach this goal every particle in the sample before sample preparation shall have an equal probability of being included in the sub-sample retained after sample preparation. Also the loss of moisture and other volatile components shall be minimised if these components are analysed or influence the properties to be analysed.

Two basic methods are used during the sample preparation. These methods are:

— sample division;

— particle size reduction of the sample.

For granular materials generally the principle of the third-power law is accepted and shall be respected at each sample division step. The equation for this third power law is shown in Equation (1):

$$m > \alpha \times d_{95}^{3} \qquad\qquad (1)$$

where

m is the mass retained after each sample division step in g;

d_{95} is the nominal top size in mm;

α is a constant over the whole sample preparation procedure for a particular material in g/mm^3.

The value and unit of constant α is fixed by the nominal particle size, d_{95}, and the sample size, m, of the sample before sample preparation.

EXAMPLE

A sample of 10 kg of SRF fluff has d_{95} of 50 mm. For the analysis is a test portion of 5 g required.

The third power law results in α = 10 000 g divided by 50 mm to the third power. The value of α is now 0,08 g/mm^3. Using this value in Equation (1) for a reduced sample size results in a nominal top size for the particles in the test portion of 3,97 mm (cube root of 5,0 g divided by 0,08 g/mm^3). Below in the table are shown the figures.

m in g	α In g/mm^3	d_{95} in mm
10 000	0,08	50
5	0,08	3,97

Table 1 shows the resulting reduction factors for the minimum (sub-)sample size, if a certain reduction of the nominal top size is chosen and the third-power law is respected. The reduction factor of the nominal top size can be calculated by dividing the current nominal top size by the proposed nominal top size after size reduction.

Table 2 shows the resulting reduction factors for the minimum nominal top size, if a certain reduction of the (sub-)sample size is chosen and the third-power law is respected. The reduction factor of the minimum (sub-)sample size can be calculated by dividing the current minimum (sub-)sample size by the proposed minimum (sub-)sample top size after size reduction.

Equation (1) can be used to calculate the exact values for each specific situation.

Table 1 — Common values for desired reduction factor minimum (sub-)sample size

Chosen reduction factor of the nominal top size	Resulting reduction factor for the minimum (sub-)sample size
1,5	3,4
2	8
3	27
4	64
5	125
6	216
7	343
8	512
9	729
10	1 000
20	8 000
30	27 000

Table 2 — Common values for desired reduction factor nominal top size

Desired reduction factor for the minimum (sub-)sample size	Necessary reduction factor of the nominal top size
2	1,3
3	1,4
4	1,6
5	1,7
10	2,2
20	2,7
50	3,7
80	4,3
100	4,6
200	5,8
500	7,9
1 000	10,0

For SRF, however, many materials turn out to be far from granular. For example in fluff the particles turn out to be predominantly flat. Therefore, for solid recovered fuels, a correction can made for non-granular materials.

Care is needed to avoid loss of fine particles and volatile components such as moisture and mercury during milling and other operations.

If a sub-sample is required for the determination of moisture content, then the sample preparation shall be carried out by a procedure that does not conflict with the requirements of CEN/TS 15414-1, CEN/TS 15414-2 or EN 15414-3. It is recommended that, if moisture content of the material (as sampled) is to be determined, a separate moisture analysis sample is taken (as there is a risk of reducing the moisture content by sample preparation operations).

If a sub-sample is required for the determination of mercury content, then the sample preparation shall be carried out by a procedure that does not conflict with the requirements of EN 15297. It is recommended that, if mercury content of the material (as sampled) is to be determined, a separate mercury analysis sample is taken (as there is a risk of reducing the mercury content by sample preparation operations).

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

6   Apparatus

6.1 Apparatus for sample division

6.1.1   Riffle boxes

A riffle box shall have at least 16 slots and an even number of slots, with adjacent slots directing material into different sub-samples, and the width of the slots shall be at least 3 times the nominal top size of the material to be riffled (see Figure 2).
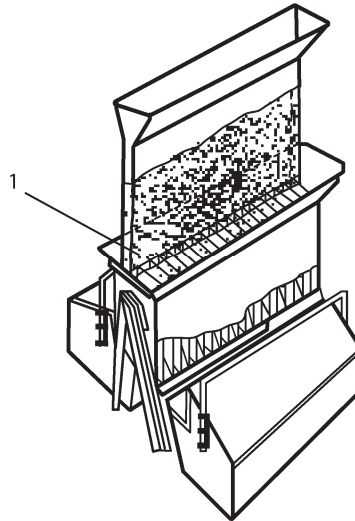
Key

1   slot, width is at least 3 times the nominal top size of the material

Figure 2 — Example of a riffle box

6.1.2   Rotary sample dividers

A rotary sample divider shall have a feeder device adjusted so that the divider rotates at least 20 times while the sample is being divided. See Figure 3 for an example of a rotating divider.

The manufacturer's instruction manual shall always be followed. The inner dimensions of the equipment where the sample is feed shall be at least 3 times as wide as the nominal top size of the material to be processed.
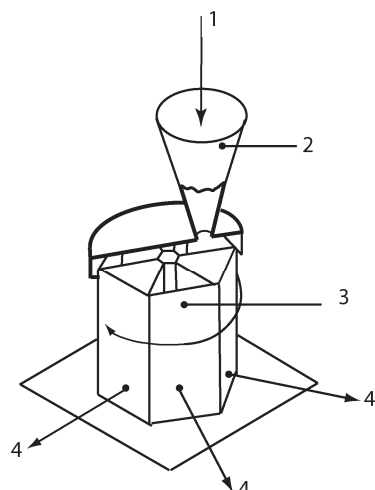
Key

1   feeder

2   funnel

3   rotating receiver

4   divided sample

Figure 3 — Example of a rotary sample divider

6.1.3   Shovels and scoops

A shovel or scoop used for manual sample division shall have a flat bottom, edges raised high enough to prevent particles rolling off, and shall be at least 3 times as wide as the nominal top size of the material to be processed. See Figures 4 and 5 for examples of a scoop and a shovel respectively.
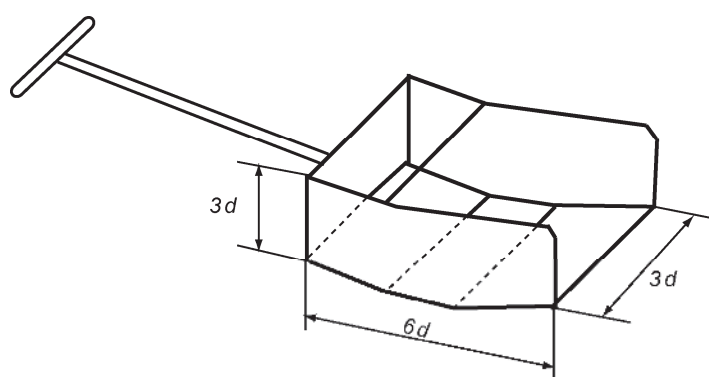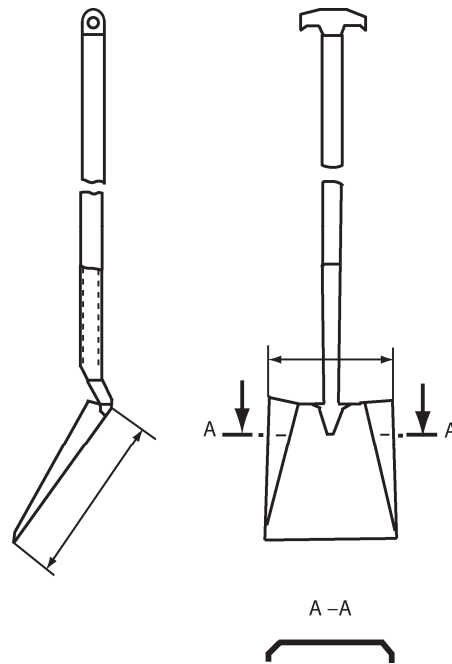

Key

d is the nominal top size

Figure 4 — Example of a scoop

Key

l is the length of the shovel

A - A   sectional view

Figure 5 — Example of a shovel

6.2 Apparatus for particle size reduction

6.2.1 Coarse cutting mill or wood crusher

Coarse cutting mills are used for cutting materials into lengths of about 10 mm to 30 mm (depending on the solid recovered fuel and the analyses to be performed). The equipment shall have a minimum of drying effect either by heating the materials or blowing air through them. The equipment shall be designed so that it does not lose dust or contaminate the material with pieces of metal, and shall be easy to clean. A cutting mill with no screens may be suitable for small quantities.

6.2.2 Cutting mill

Cutting mills are used for particle size reduction of materials used as solid recovered fuels from about 10 mm to 30 mm down to about 1 mm or less (depending on the solid recovered fuel and the analyses to be performed). The mill shall be provided with screens of various aperture sizes covering this range, including an appropriate sieve to control the nominal top size of the material produced. Other apparatus may be used provided that they are designed so that they do not get blocked with the material that is being processed. Avoid the use of cutting mills whose cutting faces contain significant quantities of an element that is to be determined in the analysis.

NOTE   Cross beater mills can be used without any excessive dusting, when fitted with dust filters (like a filter sock) between the mill and the receiving container. They are suitable for final grinding of hard, wood type materials after the pre-grinding with cutting type mills.

6.2.3 Shredder

A shredder is an apparatus with a rotor equipped with hammers that shred the material which is fed to the shredder. Shredders are used for reducing the particle size down to 30 mm. In case of hardy and strong materials it can be necessary to perform the particle size reduction in more than one step. The use of

shredders for particle size reduction causes a risk of losing moisture and fine fractions. Therefore the use of shredders shall be avoided when possible. Unfortunately many types of solid recovered fuel contain plastics and metals and make therefore the use of a shredder necessary.

6.3 Sieves

A wire-mesh sieve with an aperture size of 1,00 mm is required to check the nominal top size of general analysis samples. A wire-mesh sieve with an aperture size of 0,250 mm will be required if sub-samples with this as the nominal top size are required.

6.4 Balance

A balance is required that is capable of determining the mass of samples to an accuracy of 0,1 % of the sample mass, and the mass of sub-samples to an accuracy of 0,1 % of the sub-sample mass.

7 Sample preparation procedure

7.1 General structure

Figure 6 outlines the general procedure that shall be followed in order to perform the sample preparation according to this European Standard.

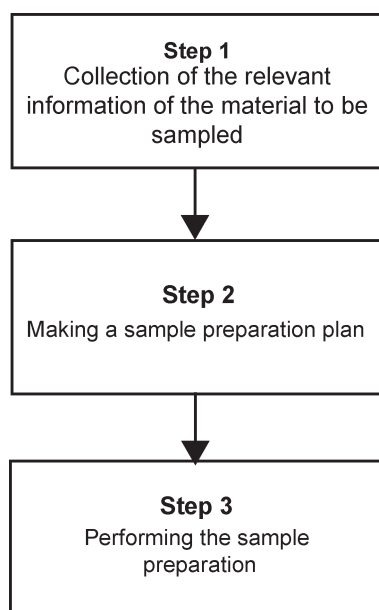**Figure 6 — General sample preparation procedure**

7.2 Step 1: Collection of the relevant information of the material to be sampled

In the first step of sample preparation information shall be collected about the material to be sampled:

a) the minimum sample size out of the sampling plan;

b) the actual size of the sample, m_0;

c) the nominal top size of the sample;

d) the shape factor of the sample;

e) the requirements in terms of size reduction for the analysis that need to be performed;

f) the required amounts for each of the size fractions and their restrictions to the sample preparation methods. Sample preparation prepares a sample for a number of tests which will be performed on the sample. Some of these tests require no particle size reduction or drying of the material in the sample. Other tests require very tiny homogenized sub-samples with small particle sizes. A sample preparation plan shall have to meet all these requirements.

7.3 Step 2: Making a sample preparation plan

7.3.1 General

This subclause specifies the making of a sample preparation plan. The actual making of the sample preparation plan is the most crucial phase during sample preparation. Sample preparation is a combination of sample division and particle size reduction. Until what level a sample of solid recovered fuel shall be prepared on site depends on available equipment on site, the requirements of the laboratory and the preferences of the client of the sampling activities. These two essential activities are specified below.

Sample division

The aim of sample division of a sub-sample is to reduce the mass remaining sub-sample or to make several duplicate sub-samples out of one original sub-sample available. During the performance of sample division it is of eminent importance that the minimum sub-sample size shall be retained in order to sustain the representatively of the sub-sample for the original combined sample. Clause 7 describes the available methods for sample division.

Particle size reduction of a sample

The aim of particle-size reduction is to reduce the nominal top size of the particles in order to reduce the minimum sub-sample size without losing representatively. During the performance of particle size reduction it is important that all materials are included. Leaving out metals causes significant errors in the measured values of these and possibly accompanying metals.

In order to make a valid sample preparation plan the plan shall contain at least the information as shown in Table 3. Table 3 can be used as a sample preparation plan. It specifies all activities that shall be performed during the whole process of sample preparation. The actual structure of Table 3 shall be adjusted to the properties of the investigated SRF and the equipment selected in the laboratory takes place. This means that e.g. steps can be skipped or added if the nominal top size is already smaller than e.g. 30 mm or the coarse shredder results in a nominal top size different from 30 mm. Some of the information used to complete Table 3 such as techniques, shape factors, nominal top sizes and particle size reduction steps shall be chosen or determined. The determination of the changing shape factor is specified in 7.3.2. The determination of mass and particle size reduction techniques and apparatus are specified in Clause 8. Annex C shows an example how a sample preparation plan is made. Clause 8 describes the methods of sample division before particle size reduction. Clause 9 describes how the sampling preparation plan of Table 3 shall be implemented.

A sample preparation plan can be used multiple times for a specific product as long as the physical composition of the solid recovered fuel does not change.

Table 3 — Sample preparation plan

	Description	Method of reduction	Used technique and apparatus	Mass before reduction	Mass after reduction	Nominal top size before reduction	Nominal top size after reduction	Shape factor before reduction	Shape factor after reduction	Mass to be withheld for analysis	Purpose of product of this reduction step
Step 1	sample division of the combined sample in a sub-sample for further sample preparation and a sub-sample of untreated material	sample division									determination of bulk density, durability of pellets, particle size distribution etc.
Step 2	particle size reduction in order to make further sample division possible	particle size reduction to < 30 mm					30 mm				
Step 3	sample division in order to reduce the remaining mass or obtain sub-samples as general analyses sample	sample division				30 mm	30 mm				sub-samples for moisture content, etc.
Step 4	particle size reduction the remaining sub-sample in a sub-sample for further sample preparation sub-samples as general analyses sample	particle size reduction to <1,0 mm				30 mm	1,0 mm		1,0		
Step 5	sample division of the remaining sample material into the required general analysis sample(s)	sample division				1,0 mm	1,0 mm	1,0	1,0		sub-samples for determination of ash, calorific value, chemical analysis etc.
Step 6	particle size reduction in order to make further sample division possible	particle size reduction to <0,25 mm				1,0 mm	0,25 mm	1,0	1,0		
Step 7	sample division of the remaining sample material into the required test portions	sample division				0,25 mm	0,25 mm	1,0	1,0		sub-samples for analysis where < 0,25 mm is required

NOTE   Mass can only be withheld during a sample division step and not during a size reduction step.

7.3.2 Retaining the minimum (sub-)sample size

During every sample division step, every particle in the sample before sample division shall have an equal probability of being included in the sub-sample retained after sample division. For non-granular materials with a shape factor significantly smaller than 1,0 a correction shall be made for the changing shape factor. How this correction shall be calculated is specified in Annex A.

Each step of a sample preparation procedure shall comply with Equation (2):

$$\frac{m_1}{m_2} \geq \frac{f_1}{f_2} \times \left(\frac{d_1}{d_2}\right)^3 \tag{2}$$

where

m_1 is the sample size of sample before particle size reduction;

m_2 is the sample size of sample after particle size reduction;

f_1 is the shape factor of sample before particle size reduction;

f_2 is the shape factor of sample after particle reduction;

d_1 is the nominal top size of sample before particle size reduction;

d_2 is the nominal top size of sample after particle size reduction.

For more or less granular materials the shape factors f_1 and f_2 will be close to 1. In this case it is easier and cheaper to assume these two shape factors to be 1. For materials which are far from granular the shape factor f_1 needs to be determined. The determination of the shape factor shall be as specified in EN 15442. The shape factor will change during the particle size reduction. Therefore the shape factor will approach 1,00 during particle size reduction. In Annex A is specified how a prognosis shall be made for the increase of the shape factor after particle size reduction. For each particle size reduction step a new shape factor and nominal top size shall be determined in order to establish whether the proposed sample preparation step complies with Equation (2). The shape factor can always be assumed to be 1,00. This will require additional particle size reduction, but will simplify the procedure and not influence the representativeness.

7.4 Step 3: Performing the sample preparation plan

In the third step the sample preparation shall be performed as specified in the sample preparation plan.

During the procedures of the actual sample preparation the following procedures shall be performed at any times:

— homogenize the sub-sample material at every step thoroughly;

— make sure no material of the sub-sample gets lost;

— if possible do not leave out any components. When it is necessary to remove hard substances like metal (steel) to avoid damage to the mill this is allowed but shall be reported. The report shall include the weight percentage and type of substance which has been removed from the sample;

— at all times enough sample material shall be withheld in order to perform all analyses.

8   Methods for sample division

The following methods shall be used to produce a smaller but representative sample.

The remaining sample size shall not be less than the minimum sample size. The sampling implements and sample division equipment used here shall have apertures with a minimum size of 3 times d_{95}.

a)   **Riffling.** This may be used for materials that can be passed through the riffle without bridging. It is not suitable for fluff or other materials containing elongated particles, or for wet materials. Brittle materials should be handled with care to avoid fine materials to be produced. Put the whole of the combined sample into one of the containers of the riffle so that it is evenly distributed throughout the container. Place the other two containers in position under the riffle. Pour the contents of the first container down the centre line of the riffle. Pour the solid recovered fuel sufficiently slowly that bridging does not occur. Do not move the container from side to side (this would cause the end slots to receive less solid recovered fuel). Discard the solid recovered fuel that falls into one of the containers. Repeat the riffling process until a sub-sample of the required size is obtained. See Figure 2 for an example of a riffle box.

b)   **Strip mixing.** This may be used for all materials, and will be a convenient method when a combined sample is to be divided into a small number of laboratory samples. Place the whole combined sample on a clean, hard surface and homogenise it by mixing with a shovel. Position a vertical plate at each end of the strip. Use a shovel to distribute the material along the length of the strip as evenly as possible, working from end to end and from both sides. The more passes from end to end, while building the strip, will improve the quality of the division. The length to width ratio of the strip shall not be less than 10:1. Obtain a laboratory sample by taking at least 20 increments from locations evenly spaced down the length of the strip. Take each increment by inserting two plates vertically into the strip and removing all the material from between the plates. The two plates should be inserted the same distance apart each time so that each increment contains the same quantity of material. The distance between the plates should be chosen so that the method yields a laboratory sample of the required size. See Figure 8 for the principle of strip mixing.

c)   **Long pile.** This may be used for all materials, and will be a convenient method when a combined sample is to be divided into several laboratory samples. In this method, the whole of the combined sample is divided into a number of laboratory samples. Start by forming a strip as in the strip mixing method. Choose the number of laboratory samples to be produced so that they will have the required mass. Arrange this many containers conveniently near the strip. Use a shovel of a size such that each laboratory sample will receive at least 20 shovelfuls. (If the laboratory samples are each to be of mass $m_{\text{laboratory sample}}$ kg, then the shovel should have a capacity of no more than $m_{\text{laboratory sample}}$ / 20 kg). Take shovelfuls of material always from the same end of the pile, being careful not to leave fine particles behind, and place a shovelful in each container in turn until all the material has been used.

d)   **Manual increment division.** This is suitable for pellets and other solid recovered fuels with a small particle size that can be worked with a scoop. Place the whole combined sample on a clean, hard surface and homogenise it by mixing with a scoop. Use the scoop to spread the combined sample into a rectangle with a thickness of not more than three times the nominal top size, and lightly mark the surface of the rectangle with the scoop to divide it into at least 20 parts. Use the scoop and a bump plate to take an increment from each of the 20 parts, inserting the scoop to the bottom of the heap each time, and combine the increments to form the required sub-sample. See Figure 9.

e)   **Rotary divider.** A mechanical method to reduce the mass of a sample is using a rotary sample divider. The rotary sample divider shall have a feeder device adjusted so that the divider rotates at least 20 times while the sample is being divided. See Figure 3 for an example of a rotary divider.
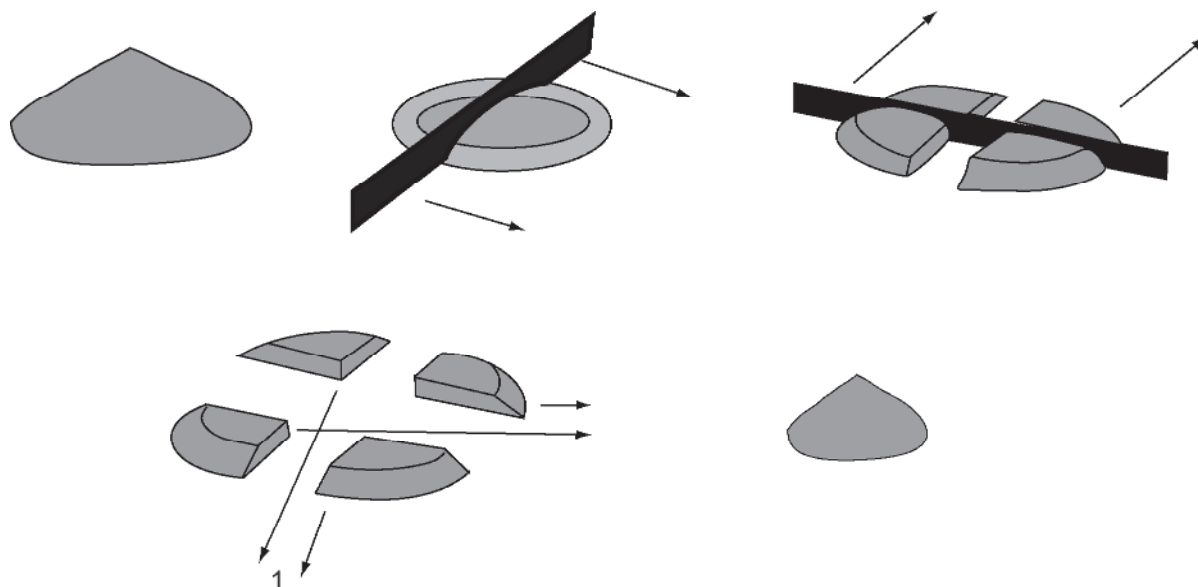
Key

1   discard

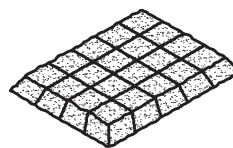**Figure 7 — Quartering[2]**
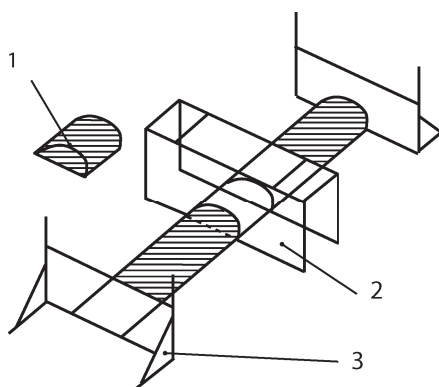
Key

1   increment

2   sampling frame (> 3 d_{95})

3   end plates

Figure 8 — Strip mixing

---

[2]   Source: Journal of chemometrics 2002;16 321-328 R. W. Gerlach, D. E. Dobb, G. Q. Raab and J. M. Nocerino
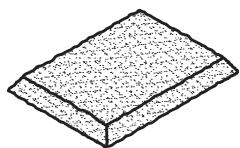
a) spread the crushed sample into a rectangle with a maximum thickness three times the nominal top size

b) arrange into 20 equal parts, e.g. into five equal parts length-wise and four equal parts breadth-wise
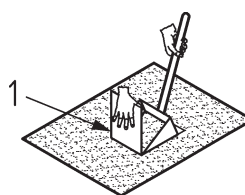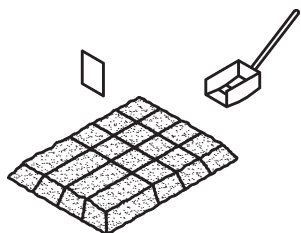
1

Key

bump plate

c) take a scoopful of samples at random from each of the 20 parts by inserting the scoop to the bottom of the sample layer. Combine the 20 scoopfuls into a divided sample

d) detail of taking an increment in one of the 20 parts by using the bump plate shown in c)

Figure 9 — Manual increment division

f)   **Coning-and-quartering.** This may be used for materials such as pellets, chips and fluff that can be worked with a shovel. It is suitable for producing sub-samples of these materials down to approximately 1 kg. Place the whole combined sample on a clean, hard surface. Shovel the sample into a conical pile, placing each shovelful on top of the preceding one in such a way that the solid recovered fuel runs down all sides of the cone and is evenly distributed and different particle sizes become well mixed. Repeat this process three times, forming a new conical pile each time. Flatten the third cone by inserting the shovel repeatedly and vertically into the peak of the cone to form a flat heap that has a uniform thickness and diameter and is no higher than the blade of the shovel. Quarter the flat heap along two diagonals at right angles by inserting the shovel vertically into the heap. See Figure 7 (A sheet-metal cross may be used for this operation if available.) Discard one pair of opposite quarters. Repeat the coning and quartering process until a sub-sample of the required size is obtained.

NOTE   This method is only used in case the other methods (a) to e)) are not applicable.

9   Methods for reducing laboratory samples to sub-samples and general analysis samples

9.1 General

This clause describes the sample preparation performance.

9.2 Initial sample division

If the initial mass of the laboratory sample exceeds the minimum mass sample size or the adjusted minimum sample size, the laboratory sample may be divided using one of the methods described in Clause 8. If this sample mass is larger than the maximum quantity requested by the laboratory, the particle size shall be reduced to enable an extra step of sample division to a smaller sample size. The remaining sample size shall be kept above the minimum sample size.

9.3 Initial mass determination

Before the laboratory sample is subject to any handling or treatment that might allow loss of moisture, mercury or dust, determine the mass of the laboratory sample by weighing on a balance accurate to 0,1 % (or better) of the laboratory sample mass.

Record this mass as $m_{sample,1}$ g.

9.4 Pre-drying

Pre-drying of wet samples is carried out to minimise moisture loss in the subsequent sample-division processes, to facilitate the sample preparation processes, and to minimise biological activity. If it is necessary to dry a sample by heating, it shall be dried in an oven at a temperature not exceeding 40 °C.

NOTE 1   If the moisture content of the original sample is unimportant, e.g. if only particle size distribution is to be determined or if a separate "moisture analysis sample" is sampled as well, the calculation of the moisture loss can be omitted. Also in this case it is not necessary to obtain completely equilibrium with the temperature and the humidity in the laboratory.

All samples (including those that have been dried by heating) shall be spread out in a tray no more than a few particles deep, and left for at least 24 h in the laboratory, until constant weight is achieved.

For among others coarse materials 24 h retention time under laboratory conditions is not enough to reach a constant weight. Monitor the moisture content by placing the sample or a sub sample on an electronic balance during the retention time in the laboratory.

Put the sample in a coarse cutting mill provided with a 30 mm sieve. Sample division may be done by alternative methods described in Clause 8.

NOTE 2   To prevent losses of moisture during the grinding process a grinder that is possible to drive at low speed is preferred.

If the information about the moisture losses during the predrying is required, determine the moisture content from an undried and a dried sub-sample according to CEN/TS 15414-1, CEN/TS 15414-2 or EN 15414-3 and calculate the difference. Using the same balance as used for the weighing in 9.2, determine the mass of the sample after the pre-drying process has been completed, and record this mass as $m_{sample,2}$ g.

Calculate the moisture loss during pre-drying as a percentage of the initial mass of the sample:

$$M_{p} = 100 \times \frac{m_{sample,1} - m_{sample,2}}{m_{sample,1}} \tag{3}$$

where

M_{p} is the moisture loss, in percentage;

$m_{sample,1}$ is the initial mass of the sample;

$m_{sample,2}$ is the mass of the sample after pre-drying.

Record the result to the nearest 0,1 %.

9.5   Coarse cutting (particle size reduction to < 30 mm)

If the material contains particles that will be retained on a 30,0 mm sieve:

—— use a 30,0 mm sieve to separate the sample into a coarse fraction (retained on the 30,0 mm sieve) and a fine fraction (passing the 30,0 mm sieve);

Materials in the sample e.g. hard alloy steel and stones shall (if necessary) be removed from the sample before coarse cutting to protect the grinder from damage. Magnetic materials can be removed by using a magnet. These materials shall be weighed and reported in the test report (see Clause 11).

—— process the coarse fraction using a coarse cutting mill so that it passes the 30,0 mm sieve;

For some types of SRF pre-treatment by freezing may be necessary before grinding. Place the sample in a freeze resistant bucket and treat the sample with carbon dioxide ice or liquid nitrogen. Remove the sample immediately from the bucket to the inlet of the moving cutting mill (grinder).

CAUTION — Be aware of the risks using carbon dioxide ice and liquid nitrogen.

NOTE 1   Depending on the material other mills than the cutting mill can be used.

—— re-combine the processed coarse fraction and the fine fraction and homogenise the sample.

If required, the above procedure may be used to achieve particle size reduction to a size below 30 mm by the use of a suitable coarse cutting mill and sieve.

NOTE 2   Grinding of the whole sample through the sieve equipped in the mill will produce the particle size needed and it also homogenizes the sample at the same time.

9.6   Sample division of <30 mm material

One of the manual methods described below shall be used for sample division. The masses of retained sub-samples shall respect the values stated in Table 1.

Use a balance accurate to 0,1 % of the sub-sample masses for the weighing required by this clause.

—— Determine the masses of the containers into which the sub-samples are to be put before starting the sample division.

A method for sample division shall be selected in Clause 8.

—— Seal the container immediately if a sub-sample is required for moisture content determination, or another test for which it is important not to lose moisture.

—— Determine the mass of each sub-sample and record this mass as $m_{subsample,1}$.

When a sub-sample is to be used as a test portion, the sub-sample shall have a mass not less than the minimum test-portion mass specified in the appropriate test method.

9.7   Particle size reduction of < 30 mm material to < 1 mm

When a sub-sample of material is required with a nominal top size of about 1 mm, use a cutting mill to reduce the whole of a sub-sample to this particle size. Carry out the process in several steps, if necessary, using a finer sieve in the mill at each step, finishing with an appropriate sieve.

If subpopulations with a different physical behaviour are expected then care shall be taken especially for the segregation of particles, so the use of sample dividers is recommended. Examples of these subpopulations are for example sand and/or metal particles.

Grinding or cutting to a nominal top size of 1 mm can cause problems, because some materials are difficult to grind and subsequently generate large amounts of heat when reduced in particle size. This heat affects the composition of the sample and may cause a loss of e.g. moisture and mercury. Therefore, it may be necessary to grind or cut the sample under cryogenic conditions or with a slow rotating grinder. If the temperature in the sample increases to more than 70 °C the particle size reduction shall be performed under cryogenic conditions.

In case of cryogenic cutting or grinding, the mills or cutters shall be suitable for grinding or cutting at very low temperatures. Cooling shall be carried out with either solid carbon dioxide (-79 °C) or liquid nitrogen (-196 °C). The sample shall be mixed or immersed in the cooling medium. In some cases it is necessary to cool the mill or cutter as well with the cooling medium in order to keep the temperatures low enough.

Another strategy of avoiding an increasing temperature is to decrease to the throughput or rotation frequency of the mill or the cutter. This may also be applied as long as the temperatures do not rise above 70 °C.

NOTE 1   Depending on the material other mills than the cutting mill can be used.

NOTE 2   Grinding of the whole sample through the sieve equipped in the mill will produce the particle size needed.

EXAMPLE   If the sample is to be reduced first to pass a 5 mm screen, and then to pass a 1 mm screen:

— a 5,00 mm sieve is used to separate the sample into a coarse fraction (retained on the 5,00 mm sieve) and a fine fraction (passing the 5,00 mm sieve);

— the coarse fraction is processed using a cutting mill containing a 5 mm screen;

— the processed coarse fraction is re-combined and the fine fraction and homogenised;

— a 1,00 mm sieve is used to separate the sample into a coarse fraction (retained on the 1,00 mm sieve) and a fine fraction (passing the 1,00 mm sieve);

— the coarse fraction is processed using a cutting mill containing a 1 mm screen;

— the processed coarse fraction is re-combined and the fine fraction and the sample is homogenised.

If the material contains granular particles, there is a risk that they will rotate in the mill or get stuck in the screen. Likewise if the material contains very elongated particles, some of these particles may come to rest on the screen and not pass through. Examine the mill after the milling is completed. If any such particles are found, grind them manually until they pass the sieve and add this material to the sub-sample.

Spread out the sub-sample in a tray no more than a few mm deep, and leave it for at least 4 h in the laboratory, until it reaches equilibrium with the temperature and humidity in the laboratory.

A sub-sample prepared by the method described in this clause may be used as a general analysis sample, in which case its mass shall be at least the mass calculated in Table 3.

Material that has been subjected to the particle size reduction operations described in this clause shall not be used for the determination of the total moisture content of the fuel.

9.8   Sample division of < 1 mm material

Homogenise the material in the sample container by stirring with a spatula, and use the spatula to take out the quantity required.

If subpopulations with a different physical behaviour are expected then care shall be taken especially for the segregation of particles, so the use of sample dividers is recommended. Examples of these subpopulations are for example sand and/or metal particles. The sub-sample size taken to further grinding shall be large enough to ensure representativeness.

9.9 Particle size reduction of < 1 mm material to < 0,25 mm

When a sub-sample is required with a nominal top size of 0,25 mm, use a cutting mill to reduce the whole of the sub-sample to this size. Feed the cutting mill with small portions of material from the general analysis sample and let each portion pass the 0,25 mm screen to prevent excess heat generation.

NOTE   Depending on the material other mills than the cutting mill can be used.

10 Storage and labelling of sub-samples

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

11 Test report

In order to conform to this European Standard include the following information in the test report:

a)   reference to this European Standard, i.e. EN 15443;

b)   date of receipt of laboratory sample and dates of the test (beginning and end);

c)   a complete identification of the laboratory sample;

d)   the sample preparation plan;

e)   identification of the test equipment and instruments used;

f)   the weight of the laboratory sample and the materials excluded from the sample preparation process e.g. stones or metals, shall be weighed and reported;

g)   any deviation from the test method and the reason of this deviation together with all circumstances that have influenced the results.

12 Precision

The test results for a measurement on solid recovered fuels are usually applied for the enforcement of regulation or for contract execution. In such legal situations, it is vital that the associated uncertainties for the tests are known.

Data regarding the uncertainty of EN 15443 has been acquired through the validation investigation QUOVADIS. The results of QUOVADIS give information on the uncertainty of the sample preparation [4, 5].

Two reasons cause that these results of QUOVADIS cannot be available as normative data on the precision.

1)   The trueness of a measurement regarding solid recovered fuel is by definition not available, because knowledge on the true composition of solid recovered fuel is by definition unknown.

2)   Validation data shall be available for each specific matrix and therefore for each type of solid recovered fuel.

Therefore, data on the precision of the methods for the preparation of the laboratory sample is only shown in Annex D. Annex D shows data on the precision of methods for the preparation of the laboratory sample of solid recovered fuels.

Annex A
(normative)

Determination of the changing shape factor

A.1 Introduction

This annex specifies how the shape factor will change during the process of particle size reduction.

The changing shape factor of the particles has a large influence on the minimum (sub-)sample size for materials where this factor is much smaller than 1,0. This correction will only be necessary for solid recovered fuels like fluff and fluff with a larger particle size in particular.

A.2 Procedure

This annex specifies how a prognosis shall be made of the shape factor after particle size reduction. Table A.1 gives a prognosis of the shape factor after particle size reduction based on the shape factor before particle size reduction, the global shape of the particles and how many times the nominal top size is decreased in the particle size reduction process. Instead of using this table it is always possible to make a real analysis of the shape factor as specified in Annex B.

Here, the following points are important:

a) not every single possible result is provided in Table A.1, and so in some cases an intermediate value shall be estimated by means of rounding up;

b) the values in the tables have been established for particles with specific dimensions that serve as a model for a specific type of particle (flat or elongated). In fact, it is not just the particle types that are important. The actual dimensions of the particles are too. For flat particles, the length and breadth, for example, are always assumed to be equal, though in practice this is probably not the case. This assumption is generally not a problem, as the composition of solid recovered fuels is often so heterogeneous that particle dimensions can only be estimated. The values for the mixed/average shape of particles in Table A.1 have been calculated by averaging the values for the extreme shapes;

c) for shape factor f = 0,25, no mixed/average shape type has been included, and for shape factor f_0 = 0,50 to 1, no distinction whatsoever has been made between shape types. The reason for this is that the effect of the shape type decreases as the shape factor increases, and so it is irrelevant in the long run.

Table A.1 — The shape factor after particle size reduction

Shape factor before reduction (f_0)	Global shape of the particles	Number of times d_{95} is reduced (n)													
		2	3	4	5	6	7	8	9	10	20	30	40	50	100
0,01	flat	0,02	0,03	0,04	0,05	0,06	0,07	0,08	0,09	0,10	0,20	0,30	0,40	0,50	1,00
	mixed/average	0,03	0,06	0,10	0,15	0,21	0,28	0,36	0,45	0,55	0,60	0,65	0,70	0,75	1,00
	elongated/needle-shaped	0,04	0,09	0,16	0,25	0,36	0,49	0,64	0,81	1,00	1,00	1,00	1,00	1,00	1,00
0,02	flat	0,04	0,06	0,08	0,10	0,12	0,14	0,16	0,18	0,20	0,40	0,60	0,80	1,00	1,00
	mixed/average	0,06	0,12	0,20	0,30	0,42	0,56	0,58	0,59	0,60	0,70	0,80	0,90	1,00	1,00
	elongated/needle-shaped	0,08	0,18	0,32	0,50	0,72	0,98	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,04	flat	0,08	0,12	0,16	0,20	0,24	0,28	0,32	0,36	0,40	0,80	1,00	1,00	1,00	1,00
	mixed/average	0,12	0,24	0,40	0,60	0,62	0,64	0,66	0,68	0,70	0,90	1,00	1,00	1,00	1,00
	elongated/needle-shaped	0,16	0,36	0,64	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,05	flat	0,10	0,15	0,20	0,25	0,30	0,35	0,40	0,45	0,50	1,00	1,00	1,00	1,00	1,00
	mixed/average	0,15	0,30	0,50	0,63	0,65	0,68	0,70	0,73	0,75	1,00	1,00	1,00	1,00	1,00
	elongated/needle-shaped	0,20	0,45	0,80	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,10	flat	0,20	0,30	0,40	0,50	0,60	0,70	0,80	0,90	1,00	1,00	1,00	1,00	1,00	1,00
	mixed/average	0,30	0,60	0,70	0,75	0,80	0,85	0,90	0,95	1,00	1,00	1,00	1,00	1,00	1,00
	elongated/needle-shaped	0,40	0,90	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,15	flat	0,30	0,45	0,60	0,75	0,90	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	mixed/average	0,45	0,72	0,80	0,87	0,95	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	elongated/needle-shaped	0,60	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,20	flat	0,40	0,60	0,80	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	mixed/average	0,60	0,80	0,90	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	elongated/needle-shaped	0,80	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,25	flat	0,50	0,75	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
	elongated/needle-shaped	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,50	all shapes	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
0,75	all shapes	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00
1	all shapes	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

Annex B
(normative)

Determination of the shape factor

B.1 Introduction

This annex specifies how the shape factor will be determined.

The shape factor of the particles has a large influence on the minimum (sub-)sample size for materials where this factor is much smaller than 1,0. This correction will only be necessary for solid recovered fuels like fluff and fluff with a larger particle size in particular.

B.2 Procedure

This annex specifies how the shape factor shall be calculated of the shape factor after particle size reduction. If the solid recovered fuels are of the fluff type, the shape factor s corrects the minimum sample size, as such materials are not granular, but are often composed of flat pieces. For pellet-type solid recovered fuels, which are roughly granular, this factor is not corrective (for pellet-type solid recovered fuels the value of f is (virtually) 1,0).

For fluff-type materials, the following equation is used to determine the shape factor f:

$$f = \frac{V_{95}}{d_{95,l}{}^3}$$ (B.1)

where

f is the shape factor, in mm³/mm³;

V_{95} is the maximum volume of a fluff particle (a mass fraction of 95 % of the particles are smaller than V_{95}), in mm³ (where $V = l \times b \times h$);

$d_{95,l}$ is the maximum length of a fluff particle (a mass fraction of 95 % of the particles are smaller than $d_{95,l}$), in mm.

The shape factor f is not constant, but depends on the type of fluff-type material.

Annex C
(informative)

Examples of sample preparation

C.1 Introduction

In this annex two situations have been worked out as an example on how this European Standard is used.

C.2 Example 1 pellets

A recycling plant that combines several types of waste residues from the industry produces pellets with a maximum particle size of 20 mm. Since the components in the products are already processed in a particle size reduction process most particles inside the pellets can be assumed to be more or less granular. This results in a shape factor of approximately 1,0. The bulk density is 300 kg/m^3 and the particle density is 0,9 g/cm^3. The distribution factor is 1,00, because the particles show a wide range of evenly distributed particle sizes. VC and p can be assumed to be 0,1. These figures have resulted in a minimum sample size of 3,4 kg or 11,3 l. The material requires for the analysis of chlorine a maximum particle size of 1,0 mm.

How shall the sample preparation plan be made and what activities have to be performed on site?

The case as described in the text above has been worked out in Table C.1.

C.3 Example 2 fluff

A recycling plant that separate municipal solid waste and produces solid recovered fuel which is separated from paper plastic fraction by wind shifting. The particle size of this fraction is rather coarse (> 500 mm) and the SRF is therefore comminuted with a shredder to a final product with a nominal top size of 300 mm. Since the components in the products are mostly very flat the average shape factor measures 0,05 with average dimensions. The bulk density is 90 kg/m^3 and the particle density is 0,9 g/cm^3. The distribution factor is 0,25, because the particles show a wide range of evenly distributed particle sizes. VC and p can be assumed to be 0,1. These figures have resulted in a minimum sample size of 143 kg or 1 590 l. For the determination of the bulk density at least 40 kg of sample is needed that has not been comminuted. The shredder of the producer of the SRF is able to reduce the particle size down to 30 mm. The material requires for the analysis of mercury a maximum particle size of 1,0 mm.

How shall the sample preparation plan be made and what activities have to be performed on site?

The case as described in the text above has been worked out in Table C.2.

Table C.1 — Sample preparation plan

	Description	Method of reduction	Used technique and apparatus	Mass before reduction	Mass after reduction	Nominal top size before reduction	Nominal top size after reduction	Shape factor before reduction	Shape factor after reduction	Mass to be withheld for analysis	Purpose of product of this reduction step
Step 1	sample division of the combined sample in a sub-sample for further sample preparation and a sub-sample of untreated material	sample division	long pile reduction	4,4 kg	3,4 kg	20 mm	20 mm	1,0	1,0	1 kg	determination of bulk density, durability of pellets, particle size distribution etc.
Step 2	particle size reduction in order to make further sample division possible	particle size reduction to < 30 mm									
Step 3	sample division in order to reduce the remaining mass or obtain sub-samples as general analysis sample	sample division									sub-samples for determination of particle size distribution, abrasion resistance, moisture content etc.
Step 4	particle size reduction in order to make further sample division possible	particle size reduction to < 1,0 mm	cutting mill	4,4 kg	4,4 kg	20 mm	1,0 mm	1,0	1,0		
Step 5	sample division of the remaining sample material into the required general analysis sample	sample division	manual increment division	4,4 kg	1,0 g	1,0 mm	1,0 mm	1,0	1,0	1,0 g	Sub-samples for determination of ash, calorific value, chemical analysis etc.
Step 6	particle size reduction in order to make further sample division possible	particle size reduction to < 0,25 mm									
Step 7	sample division of the remaining sample material into the required test portions	sample division									Sub-samples for analysis where < 0,25 mm is required
NOTE Mass can only be withheld during a sample division step and not during a size reduction step.											

Table C.2 — Sample preparation plan

	Description	Method of reduction	Used technique and apparatus	Mass before reduction	Mass after reduction	Nominal top size before reduction	Nominal top size after reduction	Shape factor before reduction	Shape factor after reduction	Mass to be withheld for analysis	Purpose of product of this reduction step
Step 1	sample division of the combined sample in a sub-sample for further sample preparation and a sub-sample of untreated material	sample division	long pile reduction	183 kg	143 kg	300 mm	300 mm	0,05	0,05	40 kg	determination of bulk density, durability of pellets, particle size distribution etc.
Step 2	particle size reduction in order to make further sample division possible	particle size reduction to < 30 mm	shredder	143 kg	143 kg	300 mm	30 mm	0,05	0,5		
Step 3	sample division in order to reduce the remaining mass or obtain sub-samples as general analyses sample	sample division	long pile reduction	143 kg	1,43 kg	30 mm	30 mm	0,5	0,5		sub-samples for determination of moisture content etc.
Step 4	particle size reduction the remaining sub-sample in a sub-sample for further sample preparation sub-samples as general analyses sample	particle size reduction to < 1,0 mm	cutting mill	1,43 kg	1,43 kg	30 mm	1,0 mm	0,5	1,0		
Step 5	sample division of the remaining sample material into the required general analysis sample(s)	sample division	incremental method	1,43 kg	1,0 g	1,0 mm	1,0 mm	1,0	1,0	1,0 g	sub-samples for determination of ash, calorific value, chemical analysis etc.
Step 6	particle size reduction in order to make further sample division possible	particle size reduction to < 0,25 mm									
Step 7	sample division of the remaining sample material into the required test portions	sample division									sub-samples for analysis where < 0,25 mm is required
NOTE Mass can only be withheld during a mass reduction sample division step and not during a size reduction step.											

C.4 Large pieces SRF – Size-reduction and sub-population separation of field samples

This annex applies to samples of large pieces SRF taken in view of characterisation, especially by chemical analysis, requiring a size reduction down to 0,5 mm. Typical examples are shredded tires and demolition wood. In both cases the SRF is constituted of large pieces (e.g. for shredded tires 20 mm to 500 mm) in order to maximise energy production, since size reduction costs significant amounts of energy. Consequently the size reduction of the field sample needs a special procedure to deal with large pieces in the field sample, a large mass for field samples (e.g. for shredded tires 20 kg to 30 kg) and the presence of several subpopulations which are mixed in the pieces (e.g. for shredded tires rubber, metal, textile fibres).

NOTE 1   The process described in this annex is more complex than the usual size reduction processes as a result of the large pieces aspects and the sub-populations separation. Therefore it is vital to weigh the fractions obtained at all the different stages.

The main steps to be applied for particle size reduction of large pieces SRF and the separation of sub-population are as follows:

1.   Obtain a representative field sample and weigh it. Typically for shredded tires 20 kg to 30 kg. Large pieces can contain metallic rod bunches. Use a saw to extract the rod bunch from the rubber.

2.   Reduce the particle size in a shedder using successively at least three different grids in order to reach the particle size of 10 mm.

For shredded tires, the shredder has a typical capacity of 200 kg/h. A typical selection of grids is consecutively 40 mm, 20 mm and 10 mm. The shredder shall be equipped with a ventilation system including a filter in order to collect the light fraction (released fibres). It is to be noted that the ventilation can also induce an air draught through the system resulting in a cooling effect. Metallic rods passing through the grid are re-introduced in the shredder. This results in the "10 mm sample" to be weighed, as well as the collected fibres.

3.   Quarter the "10 mm sample" in order to obtain a sample of about 2,5 kg. This sample is weighed.

NOTE 2   The other fractions are weighed and stored.

4.   Reduce the particle size in the same shedder using at least two different grids. The last grid does not allow particles to go through it.

NOTE 3   For shredded tires, a typical selection of grid is a 6mm grid and a "solid" grid (i.e. without holes or with holes below 0,5mm allowing air or nitrogen cooling). The shredder is fitted with a ventilation system including a filter in order to collect the released fibres.

5.   Quartering this sample so as to obtain a sample of about 0,7 kg. This sample is weighed.

NOTE 4   For shredded tires a sample of about 0,7 kg corresponds to 0,5 kg of rubber. The other fractions are weighed and stored.

6.   Magnetic separation of the metal sub-population.

NOTE 5   This is easily done by spreading the sample on a flat surface and move slowly a magnet above this surface. For shredded tires this results in a "rubber fraction" consisting mainly of rubber and of a "metallic fraction" consisting mainly of metals. Both fractions are weighed.

7.   Clean the light fraction collected at the different steps by sieving this light fraction.

NOTE 6   For shredded tires this results in a rubber powder considered as part of the "rubber fraction" and in a "fibres fraction" consisting mainly in fibres.

8. Dip the powder sample into liquid nitrogen and thereafter micronise the powder sample down to 0,5 mm.

NOTE 7   Then the powder can be divided with a rotary divider in the powder amounts needed for the requested determinations. The obtained samples are weighed.

9. Store and send the obtained samples for the requested determinations.

10. Produce a report on the above treatment and including the weights determined at the different steps.

Annex D
(informative)

Data on the precision of sample preparation

D.1 Introduction

The precision can be subdivided into: the trueness, the repeatability, the reproducibility and the robustness of a method. In the European project QUOVADIS research has been performed in order to obtain data on the precision of technical specifications on solid recovered fuels. Deliverable 4.2, [3] and deliverable 4.3, [4] of QUOVADIS deal with methods of sampling and methods for the preparation of the laboratory sample. The data on the precision in this clause shows the combined results of the entire process of sample preparation, digestion and analysis. Within the work of QUOVADIS it was not feasible to determine the precision of the sample preparation step alone. The sample preparation (in the laboratory), digestion and analysis, however, have been performed by the same laboratory with identical procedures.

D.2 Scope

This annex contains informative data on the precision of sample preparation of solid recovered fuels.

D.3 Trueness

The trueness is the extent to which method gives the correct result. The determination of trueness requires information about the exact composition of the solid recovered fuel before sample preparation. The composition of waste is by definition unknown. Therefore, it is not possible to determine the trueness of a method, unless synthetic waste is produced. It is unfeasible to produce synthetic lots of solid recovered fuels with a common lot size of 300 tons to 1 500 tons.

D.4 Repeatability and reproducibility

The program of QUOVADIS did not specifically include testing on validation and robustness of the methods for the preparation of the laboratory sample. The QUOVADIS program on the EN 15442 on methods of sampling for solid recovered fuels included tests on the repeatability of methods for sample preparation. Reproducibility was not tested.

In QUOVADIS the repeatability of methods for the preparation of the laboratory sample of solid recovered fuels has been determined on 5 types of solid recovered fuel. Those 5 types of solid recovered fuel were sampled by 5 different samplers each from another EU country. The 5 types of SRF were:

1) soft pellets derived from municipal solid waste;

2) soft pellets derived from commercial solid waste;

3) hard pellets derived from commercial and bulky waste;

4) mining residues and industrial residues.

5) soft pellets derived from commercial solid waste (low ash content).

Table D.1 shows data on the repeatability of measurements on solid recovered fuels. These measurements include analysis, digestion and sample preparation. The values for thallium were not determined, because all analysis values were below the detection limit.

Table D.1 — Repeatability and reproducibility

Parameter	Statistical parameter	Average	Upper limit 90-% confidence interval
Dry matter	Relative repeatability	0,3 %	0,6 %
Net calorific value	Relative repeatability	4 %	8 %
Chlorine	Relative repeatability	20 %	50 %
Antimony	Relative repeatability	30 %	70 %
Arsenic	Relative repeatability	10 %	20 %
Lead	Relative repeatability	20 %	40 %
Cadmium	Relative repeatability	40 %	70 %
Chromium	Relative repeatability	20 %	60 %
Cobalt	Relative repeatability	20 %	40 %
Copper	Relative repeatability	100 %	200 %
Manganese	Relative repeatability	10 %	20 %
Nickel	Relative repeatability	40 %	100 %
Mercury	Relative repeatability	30 %	70 %
Thallium	Relative repeatability	Not determined	Not determined
Vanadium	Relative repeatability	20 %	30 %

For parameters with repeatabilities or reproducibilities larger than 25 % it is recommended to take and analyse duplicate samples for a single lot

D.5 Robustness

D.5.1 General

The robustness is the extent of the method to what it is subject to influencing parameters. For methods of laboratory sample preparation two important influencing parameters exist the type of solid recovered fuel and the level of particle size reduction.

D.5.2 Type of solid recovered fuel

The type of solid recovered fuel has a tremendous effect on the precision of the measurement. Solid recovered fuels with a very small particle size and a very homogenous bulk composition show good repeatabilities and reproducibilities. Solid recovered fuels with a larger nominal top size and a heterogeneous bulk composition show poor repeatabilities and reproducibilities for micro parameters such as copper, nickel and mercury. The poor robustness for the type of solid recovered fuel is mostly caused by imperfection of the sample preparation method to deal with extremely heterogeneous materials. Table D.1 shows large variation for the repeatabilities and reproducibilities for a single parameter. This large variation is mostly caused by the differences between the investigated types of solid recovered fuel.

D.5.3 Level of particle size reduction

The level of particle size reduction is a very important factor when it comes to proper sample preparation. Sample preparation is in principle taking a sample of a sample. Reducing the representative sample size is only possible if nominal top size of the particles is sufficiently reduced. The big question is what is sufficient.

This depends strongly on the factor p. The factor p can be described as the fraction of particles that influences the property which is to be analysed. This factor p is specific for each combination of the type of solid recovered fuel and the property to be investigated. If factor p is taken too large it will result in analyses results that have no normal distribution and frequently show spikes in the results. A common figure for p is 0,1. This is sufficient for bulk properties such as lower heating value, biomass content, carbon content, hydrogen and ash content. This value, however, is usually in case of solid recovered fuels insufficient for e.g. heavy metals such as mercury and cadmium. Assuming a smaller value of p during sample preparation will improve the results, but will increase the efforts for sample preparation significantly. It will also result in a smaller required nominal top size for the final test portion.

In this European Standard the factor p is not addressed, but it is included in the factor α present in Equation (1). The factor p can be determined with Annex D. If the sample plan is properly designed, the sample will be representative and by using Equation (1), the factor p is also respected.

Bibliography

[1]   prEN 14780, *Solid biofuels — Methods for sample preparation*

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

[3]   QUOVADIS Deliverable 4.2 – 2005 – Results of ruggedness testing of the sampling procedures. EU-Project number: EIE 2003 031 - Grant Agreement EIE/031/S07.38597, Tauw BV, Deventer, 2005.

[4]   QUOVADIS Deliverable 4.3 – 2005 – Report on the validation of the sampling procedures including recommendations to TC 343 for the eventual revision of the TS before its upgrade to a European standard (EN). EU-Project number EIE 2003 031 - Grant Agreement EIE/031/S07.38597, Tauw BV, Deventer 2005.

This page deliberately left blank

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards-based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email bsmusales@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Copyright

All the data, software and documentation set out in all British Standards and other BSI publications are the property of and copyrighted by BSI, or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI. Details and advice can be obtained from the Copyright & Licensing Department.

Useful Contacts:

Customer Services
Tel: +44 845 086 9001
Email (orders): orders@bsigroup.com
Email (enquiries): cservices@bsigroup.com

Subscriptions
Tel: +44 845 086 9001
Email: subscriptions@bsigroup.com

Knowledge Centre
Tel: +44 20 8996 7004
Email: knowledgecentre@bsigroup.com

Copyright & Licensing
Tel: +44 20 8996 7070
Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

bsi.

...making excellence a habit.™