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## BioNorm II

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**SPECIFIC TARGETED RESEARCH OR INNOVATION PROJECT**

**PRIORITY [6-1] – Sustainable energy systems**

### Deliverable DII.1.9

**Best Practice Guidelines for determination of visual recognizable impurities with an NTS > 2 mm (Part A) and Best Practice Guidelines for wet-determination of physically attached impurities in solid biofuels with an NTS < 2 mm (Part B)**

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## Revisions

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## **Abstract**

The suitability of the methods for the determination of the impurity content of solid biofuels was proven in the Bionorm-II-project. The method and equipment was developed during the project and the developed test guideline for the Bionorm-partner's tests can therefore serve as a basis for a new European standard.

In the here presented Best Practise Guideline all required properties and features of a suitable measuring method and equipment in order to ensure reproducible and repeatable test results is described. In order to be able to easily transform the guideline into a European standard the format and structure of a CEN technical specification was here chosen. The details of the proposed standard test procedure are described on the following pages.

The Bess Practice Guideline consists of two parts; Part A concerning impurities with a nominal top size below 2 mm and Part B concerning impurities with a nominal top size above 2 mm.

### **List of Abbreviations**


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## Part A

### 1 Introduction

Deliveries of solid biofuels may have been contaminated with impurities as a result of the performed harvesting, storage and transportation of the fuel or the raw material for the production of the fuel. Impurities in this context are particles of non-biofuel material as e.g. stones, metal pieces, plastic fragments and soil/sand being incidentally mixed up with the biofuel material.

This best practice guideline describes a method for the determination of the content of coarse impurities, larger than 2 mm, i.e. impurities as soil, sand and similar dirt materials, which are not included unless they are present as larger and robust lumps. Coarse impurities in solid biofuels may cause operational problems for the utilization of the fuel, in relation to the handling and feeding of the fuel. Further an excess of foreign particles as e.g. metal pieces and plastic fragments may be an indication of a non virgin origin of the biofuel. The method described in this best practice guideline may however be used as a first step in a complete determination of impurities, as a content of fine impurities subsequently may be determined on the obtained less than 2 mm fraction of the sample.

Determination of impurities with a nominal top size above 2 mm in materials within other fields is carried out according to the same principle as described in this guideline concerning solid biofuels, i.e. separation of the sample into different fractions by sieving and manually sorting out the impurities in the obtained fractions. The fractions chosen in this guideline however differ from the fractions used in existing methods concerning e.g. compost. According to [1] sieves with apertures 2 mm, 5 mm and 20 mm here are recommended for the fractionation.

The present guideline concerns the identification of impurities with a nominal top size above 2 mm; for impurities with a nominal top size below 2 mm the Best Practice Guideline *Wet-determination of physically attached impurities in solid biofuels with an NTS < 2 mm* (Part B) shall be consulted.

Sieving operations described in this best practice guideline shall generally follow the recommendations given in CEN/TS 15149-1 “Solid biofuels – Methods for the determination of particle size distribution – Part 1: Oscillating screen method using sieve apertures of 3.15 mm and above”, January 2006 [3].

All sampling and sample preparation including sample reduction shall follow the recommendations given in CEN/TC 335 14778 Solid biofuels – Sampling [7] and CEN/TC 335 Solid biofuels – Sample preparation [8]

### 2 Scope

This guideline describes a method for the determination of visible recognisable impurities > 2 mm by manual or mechanical sieving. The method described is meant for particulate biofuels only, namely materials that either have been reduced in size, such as most wood fuels, or are physically in a particulate form.

### 3 Normative References

- [1] ISO 3310-1. Test sieves – Technical requirements and testing – Part 1: Test sieves of metal wire cloth.
- [2] ISO 3310-2. Test sieves – Technical requirements and testing – Part 2: Test sieves of perforated metal plate.
- [3] CEN/TS 15149-1 “Solid biofuels – Methods for the determination of particle size distribution – Part 1: Oscillating screen method using sieve apertures of 3.15 mm and above”, January 2006.
- [4] Impurities and stone content. C. Blok and G. Wever. NL –PPO-Glasshouse Horticulture. Project Horizontal. Revised version 24/01/06.
- [5] CEN/TS 14774-1. Solid biofuels – Determination of moisture content – Oven dry method – Part 1: Total moisture – Reference procedure.
- [6] CEN/TS 14774-2. Solid biofuels – Determination of moisture content – Oven dry method – Part 2: Total moisture – Simplified procedure.
- [7] CEN/TC 335 14778 Solid biofuels – Sampling.
- [8] CEN/TC 335 Solid biofuels – Sample preparation.

### 4 Terms

#### 4.1 Increment

portion of a material extracted in a single operation with a sampling device.

#### 4.2 Lot

defined quantity of a material of which one or more properties is to be determined.

### 5 Principle

A sample is sieved on sieves with aperture sizes of 45 mm, 16 mm, 8 mm and 2 mm respectively, separating the sample into the four fractions > 45 mm, 16 – 45 mm, 8 – 16 mm and 2 – 8 mm and a bottom fraction < 2 mm. Non-biofuel particles in the four fractions are manually sorted out and weighed.

## 6 Apparatus

### 6.1 Sieves

For the test the below specified 4 sieves are required. The effective sieve area of the sieves should be at least 1200 cm<sup>2</sup>. The geometry of the apertures, the thickness of the sieves, the hole distances and the opening of the holes should be in accordance with ISO 3310-1 [1] and ISO 3310-2 [2].

Required sieves:

- round hole sieves with hole diameters of 45 mm, 16 mm and 8 mm respectively;
- metal wire cloth sieve of 2 mm opening

### 6.2 Collecting Pan

Pan for collecting of the material passing through the sieves.

### 6.3 Mechanical oscillating equipment

If a mechanical device is used the shaking operation shall be horizontal oscillating (one or two dimensional) or vibrating (three dimensional), using an appropriate stroke frequency depending of the type of the material. A low stroke frequency may lead to incomplete particle separation.

### 6.4 Brush

Appropriate brush for cleaning the sieves.

### 6.5 Balance

A balance capable of measuring the mass of the sample to the nearest 0.1 g.

### 6.6 Balance

A balance capable of measuring the mass of the sorted out fractions to the nearest 0.01 g.

### 6.7 Drying Cabinet

Drying cabinet capable of being controlled at 60°C ± 5°C and 105°C ± 2°C.

### 6.8 Drying Containers

Containers made of non-corrodible and heat resistant material as e.g. metal trays.

## 6.9 Riffle Divider

Riffle divider for dividing the samples.

## 6.10 Tweezers

Tweezers for collecting the impurities.

# 7 Sample Size and Preparation

## 7.1 Sample Size

The minimum size of the test sample for the determination is 8 litres and preferably larger. As the distribution of impurities in a delivery of solid biofuel may be very heterogeneous, it is very important that the sample taken as far as possible represents the mean content of the impurities. By that the sample shall consist of a necessary number of increments from the lot, the increments being evenly distributed over the lot.

**NOTE** The stated test portion of 8 litres is valid for the sieving process. As the moisture content of the material to be sieved should be determined (7.2.1), the total size of the sample should be accordingly larger.

For fine grade biofuels a smaller sample size can be used:

- if 95 % of the sample material will pass the 16 mm sieve, a sample size of minimum 4 litres can be used;
- if 95 % of the sample material will pass the 8 mm sieve, a sample size of minimum 1 litre can be used. When the sample material for the sieving further is prepared according to 7.2.2, the sample size of 1 litre includes sample material for the moisture determination.

Depending of the size (area) of the sieves the test sample may have to be divided into several sub samples, which are processed in sequential sieving operations.

## 7.2 Sample Preparation

### 7.2.1 Particulate Biofuels

The moisture content of the test sample should be less than 15 weight% in order to prevent the particles from sticking together or to lose moisture during the determination. Unless the moisture content of the received sample with absolute certainty is less than the 15 weight% the sample material therefore is to be dried and subsequently brought into equilibrium with ambient temperature and humidity.

**NOTE** If the moisture content of the received sample is considered to be less than the 15 weight% it is recommended to equilibrate the sample with ambient temperature and humidity by placing the sample material on a tray for 24 hours in the laboratory.

The drying is carried out at maximum 65°C. If the drying is carried out at temperatures above ambient in a drying cabinet, the sample shall be cooled until moisture equilibrium with the ambient temperature and humidity is achieved. The equilibrium period will depend on the drying temperature used and the type of material.

The moisture content of the material to be sieved should be determined, i.e. that a representative sub sample of the received sample (if the moisture content of the sample is less than 15 weight%) or a representative sub sample of the dried and equilibrated sample for a determination of the moisture content should be withdrawn.

### **7.3 Moisture Determination**

The moisture content of the material to be sieved according to clause 8 is determined by drying the sub-sample specified in 7.2.1 at 105°C ± 2°C according to [5] or [6].

## **8 Procedure**

### **8.1 Manual Sieving**

8.1.1 Place the 45 mm sieve on a collecting pan.

8.1.2 Weigh the sample (or sub sample) to the nearest 0.1 g and spread it on an even layer on the sieve.

8.1.3 Start sieving and terminate the sieving when no further notable passing can be observed.

8.1.4 Spread the material retained on the 45 mm sieve (> 45 mm fraction) on a flat surface. Collect all particles of non-biofuel material as e.g. stones, metal pieces and plastic fragments, using the tweezers. Weigh the amount of separated out non-biofuel particles to the nearest 0.01 g. Also make an evaluation/note of the predominant type of these particles.

8.1.5 Place the 16 mm sieve on a collecting pan and spread the material which passed the 45 mm sieve on an even layer of the sieve.

8.1.6 Start sieving and terminate the sieving when no further notable passing can be observed.

8.1.7 Spread the material retained on the 16 mm sieve (16 - 45 mm fraction) on a flat surface. Collect all particles of non-biofuel material as e.g. stones, metal pieces and plastic fragments, using the tweezers. Weigh the amount of separated out non-biofuel particles to the nearest 0.01 g. Also make an evaluation/note of the predominant type of these particles.

8.1.8 Place the 8 mm sieve on a collecting pan and spread the material which passed the 16 mm sieve on an even layer of the sieve.

8.1.9 Start sieving and terminate the sieving when no further notable passing can be observed.

8.1.10 Spread the material retained on the 8 mm sieve (8 - 16 mm fraction) on a flat surface. Collect all particles of non-biofuel material as e.g. stones, metal pieces and plastic fragments, using the tweezers. Weigh the amount of separated out non-biofuel particles to the nearest 0.01 g. Also make an evaluation/note of the predominant type of these particles.

8.1.11 Place the 2 mm sieve on a collecting pan and spread the material which passed the 8 mm sieve on an even layer of the sieve.

8.1.12 Start sieving and terminate the sieving when no further notable passing can be observed.

8.1.13 Spread the material retained on the 2 mm sieve (2 - 8 mm fraction) on a flat surface. Collect all particles of non-biofuel material as e.g. stones, metal pieces and plastic fragments, using the tweezers. Weigh the amount of separated out non-biofuel particles to the nearest 0.01 g. Also make an evaluation/note of the predominant type of these particles.

8.1.14 If content of fine impurities is to be determined, provided that such a method has been developed, weigh the amount of the material in the collecting pan (the < 2 mm fraction) to the nearest 0.1 g and transfer the material to an air-tight packing for the subsequent analysis.

**NOTE** The manual sieving procedure could be performed in one sequence where all sieves are handled simultaneous, in the same manner as for mechanical sieving (8.2).

## 8.2 Mechanical Sieving

8.2.1 Assemble the mechanical sieving device with the sieves arranged with decreasing aperture size and ending with a collecting pan (45 mm sieve – 16 mm sieve – 8 mm sieve – 2 mm sieve – collecting pan).

8.2.2 Weigh the sample (or sub sample) to the nearest 0.1 g and spread it on an even layer on the top sieve (45 mm sieve).

8.2.3 Start the sieving operation. The sieving operation shall be continued for 15 minutes.

8.2.4 Disassemble the sieves. If content of fine impurities is to be determined, provided that such a method has been developed, weigh the amount of the material in the collecting pan (the < 2 mm fraction) to the nearest 0.1 g and transfer the material to an air-tight packing for the subsequent analysis.

8.2.5 Spread the material retained on the 45 mm sieve (> 45 mm fraction) on a flat surface. Collect all particles of non-biofuel material as e.g. stones, metal pieces and plastic fragments, using the tweezers. Weigh the amount of separated out non-biofuel particles to the nearest 0.01 g. Also make an evaluation/note of the predominant type of these particles.

8.2.6 Spread the material retained on the 16 mm sieve (16 - 45 mm fraction) on a flat surface. Collect all particles of non-biofuel material as e.g. stones, metal pieces and plastic fragments, using the tweezers. Weigh the amount of separated out non-biofuel particles to the nearest 0.01 g. Also make an evaluation/note of the predominant type of these particles.

8.2.7 Spread the material retained on the 8 mm sieve (8 - 16 mm fraction) on a flat surface. Collect all particles of non-biofuel material as e.g. stones, metal pieces and plastic fragments, using the tweezers. Weigh the amount of separated out non-biofuel particles to the nearest 0.01 g. Also make an evaluation/note of the predominant type of these particles.

8.2.8 Spread the material retained on the 2 mm sieve (2 - 8 mm fraction) on a flat surface. Collect all particles of non-biofuel material as e.g. stones, metal pieces and plastic fragments, using the tweezers. Weigh the amount of separated out non-biofuel particles to the nearest 0.01 g. Also make an evaluation/note of the predominant type of these particles.

## 9 Calculations

The contents of visual recognisable impurities > 2 mm is expressed as a weight percentage of the total weight of the sample sieved for the four different size fractions:

$$\text{Impurities } > 45 \text{ mm, } I_{>45 \text{ mm}} : I_{>45 \text{ mm}} = \frac{W_{>45 \text{ mm}}}{m} \cdot 100 \%$$

$$\text{Impurities 16 - 45 mm, } I_{16-45 \text{ mm}} : \quad l_{16-45\text{mm}} = \frac{W_{16-45\text{mm}}}{m} \cdot 100 \%$$

$$\text{Impurities 8 - 16 mm, } I_{8-16 \text{ mm}} : \quad l_{8-16\text{mm}} = \frac{W_{8-16\text{mm}}}{m} \cdot 100 \%$$

$$\text{Impurities 2 - 8 mm, } I_{2-8 \text{ mm}} : \quad l_{2-8\text{mm}} = \frac{W_{2-8\text{mm}}}{m} \cdot 100 \%$$

where

$m$  is the total weight of the sample sieved (8.1.2 or 8.2.2), in g

$W_{>45 \text{ mm}}$  is the total weight of the sorted out non-biofuel material with a size > 45 mm (8.1.4 or 8.2.5), in g

$W_{16-45 \text{ mm}}$  is the total weight of the sorted out non-biofuel material with a size of 16 – 45 mm (8.1.7 or 8.2.6), in g

$W_{8-16 \text{ mm}}$  is the total weight of the sorted out non-biofuel material with a size of 8 – 16 mm (8.1.10 or 8.2.7), in g

$W_{2-8 \text{ mm}}$  is the total weight of the sorted out non-biofuel material with a size of 2 – 8 mm (8.1.13 or 8.2.8), in g

The reporting of the calculated contents of the impurities should be supplied with a description of the predominant types of impurities in the four size fraction. Also the moisture content of the sieved material should be reported.

If no impurities were observed for one or more of the four fractions, non-detectable (n.d.) content is to be stated.

If content of fine impurities is to be determined the mass fraction of the < 2 mm material,  $\omega_{<2 \text{ mm}}$  is calculated as:

$$\omega_{<2\text{mm}} = \frac{W_{<2\text{mm}}}{m} \cdot 100 \%$$

where

$w_{<2 \text{ mm}}$  is the total weight of the < 2 mm fraction in g

A content of fine impurities in the < 2 mm fraction, expressed as a weight percent of the < 2 mm fraction,  $I_{\text{fine}, <2 \text{ mm}} \%$  then can be calculated into a content of fine impurities in the original, received sample,  $I_{\text{fine}} \%$  by the following equation:

$$I_{\text{fine}} = I_{\text{fine}, <2\text{mm}} \cdot \frac{\omega_{<2\text{mm}}}{100} \%$$

## 10 Performance Characteristics

Based on a round robin with five laboratories and three replicate measurements of each biofuel at each laboratory the following figures for repeatability and reproducibility is calculated.

Biofuel	Repeatability ( $S_r$ )	Reproducibility ( $S_R$ )
Forest chips	0.348	0.579
Logging residues	0.322	0.335
Norway spruce	0.871	0.933

## 11 Test Reporting

The test report shall include at least the following information:

- Identification of the laboratory and the testing date
- The results from the calculation in Clause 9.
- Any deviation from this guideline

## Part B

### 12 Introduction

Deliveries of solid biofuels may have been contaminated with impurities as a result of the performed harvesting, storage and transportation of the fuel or the raw material for the production of the fuel. Impurities in this context are particles of non-biofuel material as e.g. stones, metal pieces, plastic fragments and soil/sand being incidentally mixed up with the biofuel material.

This best practice guideline describes a method for the partial determination of the content of fine impurities, smaller than 2 mm, i.e. impurities as soil, sand and similar dirt materials. The method described in this guideline may however be used as a first step in a complete determination of impurities. Subsequently the content of fine impurities may be determined in terms of ash content or in a pneumatic sorter.

Fine mineral impurities in solid biofuels may cause operational problems for the utilization of the fuel, primarily in relation to the ash melting behavior.

The present guideline concerns the identification of impurities with a nominal top size below 2 mm. For impurities with a nominal top size above 2 mm the *Best Practice Guideline Determination of visual recognizable impurities with an NTS > 2 mm* (Part A) shall be consulted.

Ash determination shall be performed as described in CENTS 14775 Solid biofuels – Determination of ash content [1]

All sampling and sample preparation including sample reduction shall follow the recommendations given in CEN/TC 335 14778 Solid biofuels – Sampling [2] and CEN/TC 335 Solid biofuels – Sample preparation [3].

### 13 Scope

This guideline describes a method for determination of fine impurities < 2 mm by washing in water. The method described is meant for particulate biofuels only, namely materials that either have been reduced in size, such as most wood fuels, or are physically in a particulate form.

### 14 Normative references

[1] CENTS 14775 Solid biofuels – Determination of ash content

[2] CEN/TC 335 14778 Solid biofuels – Sampling.

[3] CEN/TC 335 Solid biofuels – Sample preparation.

### 15 Terms

NTS nominal top size

## 16 Principle

A test portion of fuel chips is washed in deionised water. The detached impurities and fine particles with a NTS < 2 mm is separated through a 2 mm mesh wire sieve.

## 17 Reagents

Deionised water.

## 18 Apparatus

Containers for flushing of the impurities from the fuel chips a set of two containers are required. The inner container (sample container) consists of a metal bucket with Ø 200 mm and height 272 mm. The container is equipped with a mesh wire bottom with an aperture size of 2 mm. The outer container (flushing container) consists of a metal bucket with Ø 250 mm and height 340 mm.



**Figure 1:** Inner (sample) and outer (flushing) container for detaching of mineral impurities by washing in deionised water.

### 18.1 Vacuum Pump

Vacuum pump for evacuating the water from the impurities.

### 18.2 Büchner Funnel, Ø 240 mm

### 18.3 Rubber Sleeve

Rubber sleeve, conical, to connect the büchner funnel and the büchner flask.

#### 18.4 Büchner Flask, 15000 ml

#### 18.5 Filter Paper

Ashless (Munktell 005) Ø 240 mm (The filter papers shall be placed in a desiccator with silica gel 2 days previous to the test and store them there during the test (see Cl. 7.2.1)).

#### 18.6 Crucibles

Quadratic and flat to determine the ash content of the biofuel sample.

#### 18.7 Crucibles

Round to determine the ash content of the filter paper and impurities.

#### 18.8 Tubes

For connection of the vacuum pump and the büchner flask.

#### 18.9 Spoon

For stirring the wood chips sample in the water.

#### 18.10 Wash Bottle

#### 18.11 Beaker

For measuring the water.

#### 18.12 Drying Cabinet

Capable of operating at  $105 \pm 2^\circ\text{C}$ .

#### 18.13 Drying Containers

For drying the samples after flushing.

#### 18.14 Plastic Bags

For storage of dried solid biofuel.

#### 18.15 Cutting Mill

For size reduction of the biofuel for ash determination.

### 18.16 Spoon

Small for collecting sample for ash determination.

### 18.17 Desiccator

### 18.18 Balance

Having a sufficient capacity to enable the dish containing the sample to be weighed to the nearest 0.1 mg.

### 18.19 Furnace

Capable of giving a zone of uniform temperature at the levels required by the procedure and reaching these levels in the specified times (Solid biofuels – Method for the determination of ash content).

## 19 Sample Size

The minimum size of the test sample for the determination is 1 litre. As the distribution of impurities in a delivery of solid biofuel may be very heterogeneous, it is very important that the sample taken as far as possible represent the mean content of the impurities. By that the sample shall consist of a necessary number of increments from the lot, the increments being evenly distributed over the lot.

## 20 Procedure

### 20.1 Flushing Part

20.1.1 Place the inner container in the outer container and place the sample in the inner container.

**Note:** Be careful to remove all fine particles from the plastic bag the sample is delivered in.

20.1.2 Add 7 l of deionised water at ambient temperature to the flushing container. Stir the slurry thorough and leave the particles in the water for 5 minutes before detaching the impurities.

20.1.3 Detach the impurities by stirring/pressing the particles through the water column with the spoon for 60 seconds and accordingly lift the sample container the full way up and down through the water column 5 times to rinse down the impurities to the outer container

Repeat the procedure 3 times (60 seconds stirring and 5 times lifting).

20.1.4 Lift out the inner container and let the water run from the inner container to the outer for 120 seconds. Remove the fuel sample from the inner container and place it in a drying container (use hands). Flush the particles from the inner side of the inner container into the 'fuel sample' part.

Gently tip the inner container and rinse any particles of the outer side and the bottom of the sample container into the flushing (impurity part) container (Figure 1).

**Note:** Small, and in principle negligible, amounts of small particles may unintended remain in the inner container after the fuel sample is removed and the small particles are flushed from the container. However, to secure that no contamination is obtained between subsequent tests the inner container shall be rinsed carefully before conducting a new test.



**Figure 2:** Gently tip the inner container and rinse any particles of the bottom of the inner container into the outer container.

20.1.4 Place the drying container with the wet sample in a drying oven for 24 hours at  $105 \pm 2^\circ\text{C}$ . After drying the sample is stored in plastic bags for ash determination (Continue with cf. 8.3 B).

20.1.5 Clean the inner and outer bucket completely before the next analysis is conducted.

## 20.2 Water Evacuation

20.2.1 Place the filter papers in a desiccator with silica gel 2 days previous to the test and store them there during the test.

20.2.2 Connect the vacuum pump with the tube to the büchner flask, place the rubber fitting and the büchner funnel on the büchner flask (Figure 2).



**Figure 3:** The vacuum pump is connected with the büchner flask and the rubber fitting; the büchner funnel is placed on the büchner flask.

20.2.3 Determine the mass of the filter paper to the nearest 0.001 g (note the mass in a4 in the excel sheet) and place the filter paper in the büchner funnel. Moisten the filter paper and switch on the vacuum pump.

**Note:** By moisten the filter paper the risk of fine particles and impurities percolate to the 1st cm of the underside of the filter paper is reduced.

20.2.4 Transfer the water completely from the outer container to the büchner funnel (use water to rinse any particles from the outer container to the büchner funnel) and evacuate the water through the filter paper. Material left on the sides of the inner side of the büchner funnel shall be rinsed gently with the washing bottle to the filter paper.

**Note:** Be sure that the vacuum pump is running continuously during all filtering operations.

**Note:** The water may have to be transferred stepwise in more than one operation.

Material left on the bottom of the büchner funnel shall be transferred by hand (finger) to the filter paper (this is unavoidable and any loss is regarded as minor and reflects part of the uncertainty in the analysis. Be aware that it is not possible to flush any particles from the büchner funnel to the filter paper. This may provoke a loss of particles from the filter paper). Any particles sticking to the bottom side of the filter paper shall be left on the paper.

**Note:** Avoid complete emptying of the büchner funnel during the evacuation of the water from the büchner funnel.

20.2.5 When the separation is completed, lift the filter paper from the büchner funnel using the tweezers.

**Note:** Particles attached to the underside of the filter paper belongs to the sample.

20.2.6 Place the filter paper in the drying container and fold it according to the procedure shown in Figure 3 and dry it for 24 hours at  $105 \pm 2^\circ\text{C}$ .

20.2.7 Let the filter paper cool in a desiccator (with desiccant) and determine the mass of the filter paper incl. impurities to the nearest 0.1 mg and record the mass (the recorded figure shall be added to the mass of the empty, dry dish).



**Figure 3:** Procedure for folding the filter paper.

**IMPORTANT:** The preparation is now completed and the further quantification of the ash content shall be performed as a determination of total ash according to *CEN/TS 14775:2003, Solid biofuels - Method for the determination of ash*. The determination shall be performed for the filter paper, the retained impurities and the flushed, dried solid biofuel. Using this procedure the relative ash content of the retained impurities compared to the solid biofuel is determined.

## 20.3 Ash Determination

20.3.1 Sample preparation:

a) Filter paper with particles

- no preparation needed

### b) Biofuel

- Reduce the sample size to approximately 15 gram by using a rifflebox or by strip mixing (see below)  
**Note:** the exact size depends on the size of the biofuel sample, e.g. a 350 g sample will result in approximately 11 g sample, 250 g sample will give approximately 15 g sample
- Reduce the particle size to below 1 mm using a cutting mill
- Homogenize the subsample and collect **2.0 g** using a spoon (correspond to  $0.1 \text{ g/cm}^2$ )
- Dry the subsample for 24 hours at  $105 \pm 2^\circ\text{C}$

**Note:** The subsample is now ready for the ash determination, however if the ash determination is not performed immediately the subsample shall be stored in a desiccator (with desiccant) until the ash determination is performed.

**IMPORTANT:** Alternatively the full biofuel sample could be size reduced in a hammer mill and then the mass reduction could be performed in a rotating divider.

**Strip mixing.** This may be used for all materials and this 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 homogenize the sample by mixing it 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 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 (CEN/TS 14780 Solid biofuels – Methods for sample preparation).

**IMPORTANT:** Instead of performing the ash determination on a wet sample, the sample could be dried for 24 hours at  $105 \pm 2^\circ\text{C}$ . If the ash determination is performed on a dry sample the moisture determination is avoided and the figure for moisture ( $m_1$ ,  $m_2$  and  $m_3$ ) in the datasheet is noted as 0.

## 21 Calculation

The intensity of impurity contamination can be calculated either as:

- the difference between the ash content in the biofuel part and the impurity part (content on the ash free filter paper) or
- the relative difference between the ash content in the biofuel part and the impurity part (content on the ash free filter paper) and the corresponding figure calculated based on the difference between the ash content in the biofuel part and the impurity part (content on the ash free filter paper) using a relevant debarked wood sample (e.g. Norway spruce) as reference material.

## **22 Test Reporting**

The test report shall include at least the following information:

- Identification of the laboratory and the testing date
- The results from the calculation in Clause 21.
- Any deviation from this guideline