# INTERNATIONAL STANDARD

ISO 17830

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# Solid biofuels — Particle size distribution of disintegrated pellets

Biocombustibles solides — Détermination de la distribution granulométrique des granulés désintégrés





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Cor	ntents	Page				
Fore	word	iv				
Intro	oduction	v				
1	Scope	1				
2	Normative references	1				
3	Terms and definitions	1				
4	Principle	1				
5	Reagents					
6	Apparatus					
7	Sample preparation					
8	Procedure  8.1 Disintegration  8.2 Drying  8.3 Moisture conditioning  8.4 Sieving	3 3 3				
9	Calculation	4				
10	Performance characteristics	6				
11	Test report					
Anno	ex A (informative) Characteristics on determination of particle size distribution of material within pellets in accordance with the 2007 inter-comparison study	7				
Bibli	jography	8				

# **Foreword**

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 238, *Solid biofuels*.

# Introduction

In power plants with powder fuel burners for energy production, the operators need information about the particle size distribution of the fuel for optimising particle burnout during combustion. Fuel preparation equipment, such as pulverizers, are used for crushing pellets into the original particle sizes before the material was pressed into pellets. The method described in this International Standard is intended to characterize particle size distribution of the material contained within fuel pellets and also allows for a relative comparison of pellets of different manufacturing.

This method is based on experience with pellets made from sawdust, wood shavings and milled wood, as well as straw. The method may also be applicable for pellets produced from other solid biofuel materials provided that they can be dissolved into its constituents in water.

Pellets that are engineered to resist water, e.g. pellets from materials which have undergone some thermal treatments, cannot be characterised by this method.

# Solid biofuels — Particle size distribution of disintegrated pellets

## 1 Scope

This International Standard aims to define the requirements and method used to determine particle size distribution of disintegrated pellets. It is applicable for pellets that fully disintegrate in hot water.

#### 2 Normative references

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

ISO 16559, Solid biofuels — Terminology, definitions and descriptions

ISO 17827-2<sup>1)</sup>, Solid biofuels — Determination of particle size distribution for uncompressed fuels — Part 2: Vibrating screen using sieves for classification of samples with apertures of 3,15 mm and below

ISO 18134-1, Solid biofuels — Determination of moisture content — Oven dry method — Part 1: Total moisture — Reference method

EN 14778, Solid biofuels — Sampling

EN 14780, Solid biofuels — Sample preparation

#### 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

#### 3.1

#### sieve fraction

material collected on a sieve

# 4 Principle

The particle size distribution is determined after the sample pellets have been disintegrated in hot deionised water and dried in a drying cabinet or oven. The determination is performed by sieving the dried material in accordance with ISO 17827-2.

### 5 Reagents

Deionised water.

<sup>1)</sup> To be published.

# 6 Apparatus

**6.1 Disintegration container**, water proof container made of material such as stainless steel capable of withstanding a temperature of 100 °C. The container shall be able to hold at least 2 000 ml of the deionised water and the entire test portion of pellets without spilling over during stirring.

A lid or a cover, e.g. aluminium foil, shall be used to cover the container during the dissolving of the pellets in water.

Volume of container should be about 5 l.

- **6.2 Electric kettle or other suitable equipment for water heating**, capable of heating at least 2 000 ml of water.
- **6.3 Drying cabinet or oven**, shall be capable of maintaining a temperature of  $(60 \pm 5)$  °C with at least three air exchanges per hour. The air velocity shall be such that the test sample particles are not dislodged from the drying container(s).

NOTE Higher air exchange rates will shorten the drying time.

- **6.4 Drying containers**, shall consist of non-corrodible heat-resistant material such as metal, glass or porcelain and be able to hold sufficient volume to accommodate the slurry from the disintegration container.
- **6.5 Balance**, shall be capable of reading to the nearest 0,01 g.
- **6.6 Sieves**, set of sieves described in ISO 17827-2 and listed in <u>Table 1</u> shall be considered the default sieve set. However, other sieve sets can be used based on the specific requirements as agreed upon by the interested parties such as listed in <u>Table 2</u>.
- **6.7 Weighing containers**, an adequate number of weighing containers are required.

The weighing of the sieved particle fractions can be performed either by weighing the remaining material directly on the tarred weighed sieves or by collecting and weighing the material in weighing containers.

- **6.8 Spoon**, shall be made of non-corrodible material for stirring the disintegration slurry.
- **6.9 Mechanical sieving equipment**, sieving equipment in accordance with ISO 17827-2 shall be used for determination of the particle size distribution of the disintegrated pellets and to break down agglomerates of particles formed during the drying of the slurry.

Some sieving machines have adjustable parameters. The results of the sieving might differ depending on how adjustable parameters are controlled. It is therefore important, for comparative purposes, to report how the adjustable parameters have set in terms of frequency, amplitude, duration, etc. If machines have adjustable dimensionless settings, an estimate of the adjustable degree shall be recorded to the best of the ability of the operator.

**6.10 Flat surfaced tool**, or flat brush, shall be used for stirring the dried material and for separating agglomerated particles after drying and sieving.

# 7 Sample preparation

The laboratory sample used for the determination of particle size of disintegrated pellets shall be obtained in accordance with EN 14778 and a test sample shall be extract using volume reduction methods in accordance with EN 14780. The recommended size of the test sample is  $(300 \pm 10)$  g.

NOTE If a larger test sample is used, the amount of water, container sizes, etc. needs to be adjusted accordingly.

#### 8 Procedure

#### 8.1 Disintegration

The test sample of pellets shall be transferred into the disintegration container.

Approximately 2 000 ml of deionised water at the temperature just below the boiling point shall be poured over the pellets. In order to avoid chemical dissolving components of the material, the water temperature shall not be maintained when the pellets are disintegrating. The amount of water used shall be sufficient to assure that the material fully absorbs its maximum capacity of water. This is indicated by the presence of free water in the disintegration container after about 30 min.

For pellets with high swelling ratio, such as straw pellets, the test sample can be reduced and/or the water volume be increased.

Using a spoon, the slurry shall be carefully stirred from the bottom and up until particles are segregated from each other.

The spoon shall be rinsed with deionised water in the container ensuring that all particles remain in the slurry.

The container shall be covered with a lid to protect from contamination and to prevent evaporation of water and left for at least 30 min or as long as is required to disintegrate the pellets. Some pellets may require longer time for full disintegration.

NOTE Some pellets can take 16 h to 24 h or more to disintegrate.

#### 8.2 Drying

The disintegrated slurry shall be mixed well and transferred to an adequate number of drying containers. The disintegration container shall be rinsed carefully with deionised water and emptied into the drying containers.

Dry at a temperature not exceeding  $60\,^{\circ}\text{C}$  in a drying cabinet or oven to reach a moisture content of between  $5\,\text{w}\text{-}\%$  and  $15\,\text{w}\text{-}\%$ .

NOTE The moisture content can be checked by periodic weighing provided that the exact weight of the empty drying container(s) and the exact weight and moisture content of the test sample are known.

#### 8.3 Moisture conditioning

After drying is completed, stir the dried slurry with a flat surfaced tool to break up any agglomerates of particles or crust. The drying container(s) with the dried slurry are then placed in room atmosphere for at least 2 h in order for the material to reach moisture equilibrium with the room atmosphere.

The equilibrated test sample of the disintegrated pellets shall be divided into two test portions of approximately 150 g each in accordance with EN 14780 and marked with test portions "A" and "B".

Use test portion "A" to verify that the moisture content of the equilibrated test sample prepared for determination of the particle size distribution is between 5 % and 15 % by conducting a moisture test in accordance with ISO 18134-1.

#### 8.4 Sieving

Weigh the test portion B to the nearest 0,01 g.

Divide test portion "B" into two sub portions called B1 and B2 of approximately 75 g each.

Regarding straw and other materials with low density, the sub portions shall be further divided into two equally sized sub portions (B1-1, B1-2 and B2-1, B2-2) before sieving to secure that none of the sieves become overloaded due to the size of the sub portions.

Each test portion shall be sieved separately in accordance with ISO 17827-2. As described in 6.9, some sieving machines have adjustable parameters and is important to report how the adjustable parameters have been set during sieving.

If agglomerated particles are observed on a sieve, separate the agglomerated particles by gently using the flat surfaced tool or fingers. Continue the sieving if agglomerates are still found on the sieves.

If separation of all particles is not possible, then the minimum requirement of this method has not been achieved and the test report shall state that the pellets could not be fully disintegrated.

NOTE It can be possible to break up agglomerated particles by controlling the frequency, amplitude, duration functions of the sieving equipment (see ISO 17827-2).

#### 9 Calculation

The following tables provide examples of recording the particle size distribution obtained from the method assuming that the sub portions B1 and B2 are not further divided. <u>Table 1</u> and <u>Table 2</u> illustrate a sieve set configuration used by industry for determination of particle size distribution. <u>Table 2</u> illustrates the specific sieve set configuration specifically used for quality control of particle size distribution in industrial pellets delivered under ISO 17225-2 (see also ISO 17827-2).

NOTE Other sieve sizes may purposely be selected as required and the reporting table adjusted accordingly.

Table 1 and Table 2 illustrate how to calculate the cumulative oversize mass percentages. The cumulative undersize mass percentages may be calculated as 100 % minus the cumulative oversize mass percentages or alternatively, by summing up the mass fractions percentage in column 5 in the table from the bottom and up, i.e. from the collecting pan being the total percentage below 0,25 mm and up.

The obtained mass for each sub portion, B1 and B2, shall be summed up vertically in column 1 and 2, respectively.

The total mass of each sieve fraction shall be summed up horizontally in column 3 and recorded in g to the nearest 0.01 g and expressed in column 4 as percent of the sum of all mass fractions. The cumulative w-% passing through is summed up in column 5.

The difference between the mass of sub portion B and the total mass of all fractions in column 3 shall be recorded in the lower section of <u>Table 1</u> or <u>Table 2</u> and expressed in percent of the mass of the sub portion B.

The moisture content of test portion A shall be recorded in the lower section of <u>Table 1</u> or <u>Table 2</u> and expressed as w-%.

The difference between the mass of test portion B and the total mass of all the fractions in column 3 shall be recorded in the lower section of Table 1 or 2 and expressed in percent of the mass of test portion B. Larger differences can occur due to lost or retained particles or due to changes in moisture content. In these cases, the causes for the deviation should be investigated and the measurement repeated. If this is not practical or the result still deviates by more than 2 %, then it shall be noted in the test report.

If the test sample is divided in to additional sub portions, the tables shall be expanded with additional columns.

Table 1 — Results of the size distribution analysis using sieve set configuration often used by the industry for determination of particle size distribution

		(1)	(2)	(3)	(4)	(5)
Sieve	Fraction in mm	Mass fraction of test portion B1, in g	Mass fraction of test portion B2, in g	Total mass fraction, columns (1) + (2), in g	Percentage mass fraction, based on the total mass of all fractions in column 3	Cumulative w-% passing through (summing up the mass fraction percentages in column 4)
1. sieve (3,15 mm)	above 3,15					
2. sieve (2,8 mm)	2,8 to 3,15					
3. sieve (2,0 mm)	2,0 to 2,8					
4- sieve (1,4 mm)	1,4 to 2,0					
5. sieve (1,0 mm)	1,0 to 1,4					
6. sieve (0,5 mm)	0,5 to 1,0					
7. sieve (0,25 mm)	0,25 to 0,5					
Collecting pan	below 0,25					
Total mass of all fractions	All				100 %	

# Other recordings:

Mass of test portion B, in g	
Difference between the mass of test portion B and the total mass of all fractions (column 3), expressed in percent of the mass of test portion B	
Moisture content of test portion A, in w-%	

Table 2 — Results of the size distribution analysis using a specific sieve set configuration specifically used for quality control of industrial pellets delivered under ISO 17225-2

		(1)	(2)	(3)	(4)	(5)
Sieve	Fraction, in mm	Mass fraction of test por- tion B1, in g	Mass fraction of test portion B2, in g	Total mass fraction, column (1) + (2), in g	Percentage mass fraction, based on the total mass of all fractions in column 3	Cumulative weight percent passing through (summing up the mass fraction percentages in column 4)
1. sieve (3,15 mm)	above 3,15					
3. sieve (2,0 mm)	2,0 to 3,15					
5. sieve (1,0 mm)	1,0 to 2,0					
Collecting pan	below 1,0					
Total mass of all fractions	All				100 %	

#### ISO 17830:2016(E)

#### Other recordings:

Mass of test portion B, in g	
Difference between the mass of test portion B and the total mass of all fractions (column 3), expressed in percent of the mass of test portion B	
Moisture content of test portion A, in w-%	

#### 10 Performance characteristics

The achievable performance of the method is given in <u>Annex A</u> showing the results obtained by an inter-comparison study carried out for test samples of wood and straw.

# 11 Test report

The test report shall include at least the following information:

- a) identification of the laboratory performing the test and the date of the test;
- b) identification of product (or sample) tested;
- c) a reference to this International Standard, i.e. ISO 17830;
- d) results of the test as indicated in <u>Clause 9</u>, <u>Table 1</u> or <u>Table 2</u>;
- e) sieve set configuration;
- f) recording of all adjustable sieving machine settings (reference to ISO 17827-2);
- g) if the 2 w-% difference between the mass of the sub portion B and the total mass of all fractions in percent of the mass of the sub portion B has been exceed it shall be clearly stated;
- h) any unusual features noted during the determination, which can affect the result;
- i) any deviation from this International Standard, or operations regarded as optional.

# Annex A

(informative)

# Characteristics on determination of particle size distribution of material within pellets in accordance with the 2007 intercomparison study

An inter-comparison study was carried out in 2007 by five European laboratories (in Denmark, Finland and Germany). The purpose of the examination was to test the method described in the present draft of a method for determining the particle size of material within pellets. The method was elaborated on the basis of the experiences obtained from Reference [4].

For the obtained results in the studies, the cumulative percentages of the material retained on each sieve were calculated for each set of results. For these obtained cumulative distributions, the 25 %, 50 % and the 75 % quantiles were used for a statistical evaluation of the performance of the method. The results of this evaluation appear in  $\underline{\text{Table A.1}}$ .

Table A.1 — Performance characteristics for the determination of the particle size distribution of disintegrated pellets in accordance with the 2007 inter-comparison study

Sample	Quantiles	N	X mm	s <sub>r</sub> mm	s <sub>R</sub> mm	<i>S</i> <sub>r</sub> %	<i>S</i> <sub>R</sub> %
	25 %	5	0,46	0,018	0,039	3,94	8,53
Wood pellets, coniferous	50 %	5	0,83	0,016	0,049	1,94	5,93
	75 %	5	1,28	0,018	0,063	1,40	4,92
TAT 1 11 .	25 %	5	0,33	0,0082	0,055	2,51	16,80
Wood pellets, broad-leaf trees	50 %	5	0,72	0,016	0,089	2,22	12,36
broad lear trees	75 %	5	1,33	0,028	0,087	2,11	6,54
	25 %	3	0,46	0,025	0,056	5,46	12,23
Straw pellets	50 %	3	0,91	0,039	0,067	4,30	7,37
	75 %	3	1,44	0,057	0,092	3,96	6,31

*N* is the number of values.

*X* is the mean value.

 $s_r$  is the repeatability standard deviation.

 $S_{\rm r}$  is the relative repeatability standard deviation.

 $s_{R}$  is the reproducibility standard deviation.

 $S_{\rm R}$  is the relative reproducibility standard deviation.

# **Bibliography**

- [1] ISO 18134-2, Solid biofuels Determination of moisture content Oven dry method Part 2: Total moisture Simplified method
- [2] ISO 17225-2, Solid biofuels Fuel specifications and classes Part 2: Graded wood pellets
- [3] Characterization of Solid Biofuels 2004 Development of Methods. PSO project no. 5297. Summary Report and Guidelines. October 2008. Prepared by Jonas Dahl, Danish Technological Institute



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