GA no 282826

Production of Solid Sustainable Energy Carriers from Biomass by Means of Torrefaction

Deliverable No. D8.4

Round robin report 2 – Validation of new test methods

Dissemination Level		
PU	Public	X
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	

Nature		
R	Report	X
O	Other	

Deliverable Details	
Due date:	31.12.2014
Submission date:	19.02.2015
Authors:	Christoph Göbl (OFI) Klaus Jörg (OFI) Ute Wolfesberger-Schwabl (OFI)
Involved participants:	See Table 3
WP no. and title:	WP 8 Fuel specification, lab analysis and quality assurance
WP leader:	OFI
Task no. and title:	8.1 Fuel properties & investigation of “non-standard” parameters
Task leader:	OFI
Draft/Final:	FINAL
Keywords:	Round robin, new methods, water absorption, grinding energy

Table of Contents

Symbols and DECIMAL NOTATION

CV% coefficient of variation in percent
n_i number of repeat measurements made by an individual lab
n' number of repeat measurements necessary to ensure a sufficiently low s_r in comparison with $s*$
m general mean of the test property (used in the additional check of the test method accuracy)
$p*$ number of reporting laboratories (including all outliers)
p number of laboratories in the additional check for the test method accuracy (outliers eliminated)
r repeatability limit
R reproducibility limit
s estimate of a standard deviation
s_i within laboratory standard deviation
s_x standard deviation in the inspected set of -values x_i (in the computation of the Grubbs' statistics only)
$s*$ robust standard deviation
$\hat{\sigma}$ standard deviation for proficiency assessment; for the purpose of this PTS: $\hat{\sigma} = s*$
u_x standard uncertainty of the assigned value X
x_i test result (individual test result reported by laboratory i ;
depending on the test specification, x_i may be a result of a single measurement or a mean obtained by repeating measurements)
x_i within laboratory mean (this symbol is written as x_i , i.e. not over-lined, in all result tables due to settings in the software used in the statistical evaluation)
x_x arithmetic mean of the inspected set of x_i -values (in the computation of the Grubbs' statistics only)
$x*$ robust average
X assigned value for proficiency assessment, for the purpose of this PTS: $X = x*$
z score used for proficiency assessment

Symbols used as subscripts:

i identifier for a particular lab
k identifier for an individual test result in a laboratory i obtained under repeatability conditions
L between-laboratory (interlaboratory)
rel relative value (e.g. r_{rel} and R_{rel} ; in percent of the general mean m)
r repeatability
R reproducibility
x referring to x_i , e.g., x_x is the arithmetic mean of all x_i - values in the given set of data

The bulk of the symbols used in this report correspond with the symbols used in ISO 13528:2005, EN ISO/IEC 17043:2010 and ISO 5725-2:2002. In some cases, different symbols must be introduced to eliminate any confusion possibly caused by using symbols having different meanings in different documents.

Decimal notation:

In this deliverable, comma is used as decimal separator (as it is common in Middle Europe).

1 Introduction

After Round Robin I – Validation of "standard" test methods, which was organized at project start and focused on existing test methods listed in EN 14961 including physical and chemical parameters, the goal of SECTOR Round Robin II – Torrefied pellets, was to evaluate and validate new developed test methods. These methods are the "Water absorption" (OFI) and the "Grinding energy" (DBFZ).

The "Water absorption" is split into two parts. First part is an immersion test, to check how much water torrefied pellets can absorb. Second part is the determination of the loss of mechanical durability to see what happens with the mechanical quality of the pellets after contact with water. Torrefied pellets are much more water resistant (hydrophobic) than conventional wood pellets. So it is very meaningful and an important quality parameter to check up what happens to them in case of water contact. The other new method, "Grinding energy", should be a possibility, to determine the hardness of compressed materials and the comminution properties. Thereby a specified amount of pellets will be grinded in a lab mill and the energy consumption will be measured and so the specific "Grinding energy" can be determined.

Also some other methods were offered in this round robin test, to check and extend the data from SECTOR Round Robin I.

2 Organization of Round Robin test

2.1 Sample

In SECTOR Round Robin II a sample from an industrial plant was taken to represent typical conditions. A torrefied wood pellets sample (8 mm) was provided by Topell Nederland. The sample was produced from forest residues wood chips. In SECTOR project the sample is known as "Topell wood residues-torrefied pellets Dec13" with the SECTOR sample ID number 4027. The sample was delivered in three big bags. One big bag was used for the round robin test.

Figure 1: big bag with torrefied pellets

The whole big bag was emptied and evenly distributed on a big plane. To ensure homogeneity of the samples, the pellets were carefully mixed per hand for more than an hour. After this procedure the pellets were packaged in three different sizes:

- Sample A: Torrefied pellets – for carbon content, gross calorific value, ash melting behaviour and diameter and length
- Sample B: Torrefied pellets – for “Water absorption”
- Sample C: Torrefied pellets – for “Grinding energy”

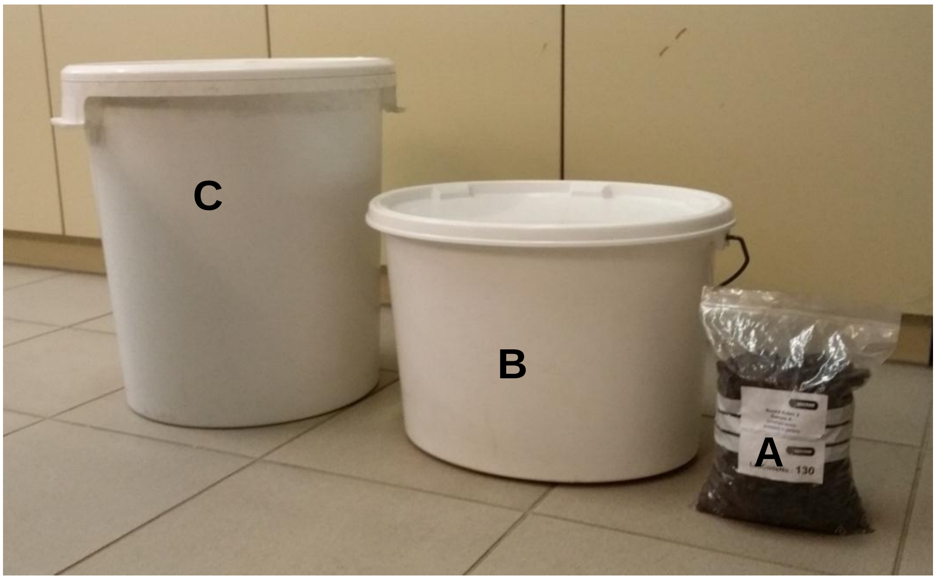


Figure 2: samples for round robin test

Within this round robin total about 450 kg (0,6 m³) of pellets were packaged and shipped.

2.2 Methods

A total of 6 test methods run in SECTOR Round Robin-II. The methods “Grinding energy” and “Water absorption” were tested for the first time in an interlaboratory comparison test. The method descriptions are attached in the appendix. The method “Grinding energy” was developed by DBFZ. It explains how much energy is necessary to grind the torrefied biomass pellets in the milling process. Therefore a certain amount of pellets is grinded in a cutting mill while the power consumption is recorded. The result of the method is the calculated specific “Grinding energy”. The immersion test for the “Water absorption” was developed by OFI by the reason of the specific behaviour of torrefied materials. Most biomass materials easily absorb moisture when they directly penetrated by water. This behaviour is especially problematical for storage and transport. Through the “Water absorption” the net calorific value decline and the mass of the material increase. Also the mechanical durability changes, this affects particular compressed biomass (e.g. pellets). The method explains how to measure the absolute “Water absorption” of torrefied pellets. Therefore a certain amount of pellets is immersed in water and the weight increase of the pellets is measured. Furthermore the mechanical durability is determined as well. With the calculation of the loss of mechanical durability, a rating of the mechanical quality of the pellets by contact with water is possible.

The other four methods were chosen by the reason of their undetermined results in Round Robin I. The following table shows the provided tests, the allowed standards/methods and the number of participants.

Table 1: provided tests and the number of participants per test

Test Series		Number of participants	
		registered	evaluated
Grinding energy	New method description	12	11
Water absorption	New method description	25	23/21
Carbon content	EN 15104	25	24
Gross calorific value	EN 14918	29	27
Ash melting behaviour	CEN/TS 15370	15	10
Diameter and length	ISO/DIS 17829 or EN 16127	26	20/24

2.3 Participants

A total of 31 testing laboratories from 15 countries participated in this interlaboratory comparison test series. The participation was open for everybody who was able to perform the selected test according to the given standards and method descriptions. The number of individual tests selected by a particular participant was not limited. The number of participants in different countries is given below.

Table 2: participants per country

Country	Number of participants
Austria	6
Germany	4
Finland	3
Croatia	2
Denmark	2
Netherlands	2
Poland	2
Sweden	2
Switzerland	2
Belgium	1
Greece	1
Scotland	1
Spain	1
UK	1
USA	1

In Table 3 all participating laboratories are listed. Table 4 shows the lab code number and for which methods they are registered. To guarantee the anonymity of the participating laboratories these lists are separately presented. The reason for the difference between the number of participating laboratories and the number of lab codes is that some companies participate with more laboratories.

Table 3: participating laboratories

Participant	Country
BEA-Institut für Bioenergie GmbH	Austria
Belab AB	Sweden
Bioenergy2020+ GmbH	Austria
Biomass Energy Lab	USA
BLT Wieselburg	Austria
Bränslelaboratoriet Umea AB	Sweden
CENER - National Renewable Energy Centre	Spain
CERTH/CPERI	Greek
CRA-W	Belgique
Danish Technological Institute	Denmark
DBZF Deutsches Biomasseforschungszentrum gemeinnützige GmbH	Germany
Doosan Babcock - Fuels & Chemistry	Scotland
E.ON Technologies (Ratcliffe) Limited	UK
Energy research Centre of the Netherlands	Netherlands
Faculty of Forestry, Forest Biomass Laboratory	Croatia
FORCE Technology	Denmark
Frauenhofer Institut UMSICHT	Deutschland
HAWK Hochschule Hildesheim/Holzminden/Göttingen	Germany
HEIG-VD	Switzerland
HEP-Proizvodnja d.o.o. CKTL	Croatia
Institute of Power Engineering, Fuel Analysis Research Laboratory	Poland
Intertek AG, Branch Schlieren	Switzerland
Labtium Oy, Jyväskylä	Finland
Österreichisches Forschungsinstitut/Energietechnik Labor	Austria
Technologie- und Förderzentrum im Kompetenzzentrum für Nachwachsende Rohstoffe (TFZ)	Deutschland
TLR International Laboratories	Netherlands
VTT Technical Research Centre of Finland	Finland
Wood Technology Institute	Poland

Table 4: registered tests

Lab. Code No.	grinding energy	water absorption	carbon content	g. calorific value	ash melting behaviour	diameter and length
101			X	X	X	X
102	X	X	X	X		X
103		X	X	X	X	X
104			X	X	X	X
105	X	X	X	X		X
106		X		X		X
107	X	X	X	X	X	X
108	X	X		X		X
109		X	X	X	X	X
110		X	X	X	X	X
111		X	X	X	X	X
112		X	X	X		X
113		X	X	X		X
114		X	X	X		
115	X	X				X
116			X	X		
117	X	X		X		X
118		X	X	X	X	X
119	X					
120			X	X		
121	X	X	X	X	X	X
122			X	X	X	X
123	X	X	X	X	X	X
124	X	X	X	X		X
125		X	X	X		
126	X	X	X	X		X
127	X	X		X	X	X
128		X	X	X	X	X
129	X	X	X	X		X
130	X	X	X	X	X	X
131	X	X	X	X	X	X

3 Evaluation

3.1 GENERAL INFORMATION ON THE EVALUATION

The testing according to a completely different 'in-house-method' did not occur but deviations from the required test procedure were often applied. All deviations, as far as reported, are denoted on the page in front of the concerned test methods.

All results which gave rise to a z-score > 10 were discarded to prevent an unnecessary distortion of the PT evaluation. Robust statistics applied in the evaluation is in fact insensitive to outliers but, for all that, extremely biased results would cause a slight shift in the calculated assigned value.

Additionally, the results of the performance assessment of all other participants would be too optimistic if definitely erroneous data would not be rejected. Wherever the rejection of submitted results was necessary, this was noted in the respective subsection of this report. In this case, the rejected data was marked blue and crossed through but left legible in the respective table and the colour of the corresponding bar in the z-score chart was converted to orange. All other z-score-values (dark-blue bars) were calculated after the extreme outliers (original z > 10) were rejected.

In all proficiency tests conducted in accordance with EN ISO/IEC 17043:2010, the **laboratory performance** is expressed by **'laboratory bias'**, i.e., by the deviation of the laboratory result ($\bar{x}_i$ or x_i) from an assigned value (accepted reference value) X . In the SECTOR RR-II, the **assigned value** X was determined in accordance with ISO 13528:2005, Clause 5.6, as '**consensus value from participants**', namely as a **robust average** $x*$.

Computation of a '**z-score**' relating to the participating labs is a common way how interlaboratory comparisons for proficiency assessment are evaluated. The z-score is a measure of the distance of an individual result from the mean; the scale unit is the standard deviation. The co called **standard deviation for proficiency assessment** $\hat{\sigma}$ is needed for the computation of the z-score. Like X , $\hat{\sigma}$ was determined in accordance with ISO 13528:2005, Clause 6.6, **from data obtained** as **robust standard deviation** ($\hat{\sigma} = s*$).

As the robust estimates $x*$ and $s*$ are insensitive to **outliers**, extreme results need not be eliminated before the assigned value X is determined. The only exception in the SECTOR RR-II was the rejection of evidently erroneous data.

Nevertheless, the identification of stragglers and outliers according to the Cochran's and Grubbs' tests using methods described e.g. in ISO 5725-2:1994 was kept up in the SECTOR RR-II for the comparison with previous PT-schemes provided by OFI. Outliers according to the **Grubbs'** test are extreme results with respect to the **deviation from the arithmetic mean of all results** ($\bar{x}_x$). Outliers according to **Cochran's** test are extreme results with respect to the **within-laboratory dispersion of the data** (Σs_i^2). In many other proficiency tests which do not utilize the data for the check of the test method accuracy, Grubbs' test is the only way how outliers are identified and later on excluded from the evaluation.

3.2 Contents of tables and charts

3.2.1 COMMENTS

On the first page of each test method related sub-section, **participants' remarks** were collected, i.e. additional information stated in the reports (particularly concerning the **test conditions** and **testing equipment**) and remarks concerning results, **properties of samples** and **comments** to any other corresponding matter of general interest. **Provider's comments** concerning the respective method were also collected here if it was necessary from the SECTOR RR-II - team point of view.

3.2.2 TABLES

The individual results reported by all participants are listed in the **first table** headed **'Results submitted by …'**. A statistical evaluation of all reported results (means $\bar{x}_i$ and standard deviations s_i, robust statistics for x^* and s^*, as well as the consistency analysis, i.e. analysis for outliers, for the sufficient repeatability and sufficiently low standard uncertainty u_x) is included in this table. One or two **asterisks** in the columns headed with "Cochran" and "Grubbs" mark the corresponding **stragglers** and **statistical outliers**, respectively.

A check of the **test method accuracy** is reported in the **third table** headed **'Additional check of the test method accuracy'**. In contrast to the proficiency testing based on the robust statistics, **general mean** m used here must be calculated from data freed from outliers. The second important condition is that the data should be **normally distributed** (see below). In the strict sense, the calculation of parameter which characterizes the test method accuracy is only correct when the input data comes from a normal distribution.

Outliers according to the definition "z>2" are marked by an '**X**' in the corresponding column in the **first table** in the sub-section 'Outliers'.

A **repeating of the tests** is necessary to obtain a sound base for the assessment of the **test method accuracy** and to get a s_r-value which is utilized in the section **'determination of laboratory performance'**. This repeatability standard deviation s_r shall not be too large in comparison with s^* and the repeating of the tests can decrease s_r. A corresponding remark ("NOT OK") concerning the number of the tests repetitions appears in the table 'Test results' if the ratio s_r / s^* is too high.

The **Anderson-Darling** test was applied on the data sets freed from outliers in the check for normal distribution of the data. This test was described by Stephens, M. A. in "EDF Statistics for Goodness of Fit and Some Comparisons", Journal of the American Statistical Association, Vol. 69, (1974), pp. 730-737. In the SECTOR RR-II, outliers (outlier laboratories) were eliminated on the basis of Cochran and Grubbs outlier tests. The test for the normal distribution of the data was conducted with all remaining individual results (x_i -values), not only with the respective averages $\bar{x}_i$.

If the test for normal distribution of the data is performed with a rather limited number of the data, the result is frequently positive (this gives "YES" in the corresponding table concerning the accuracy of the examined test method). As the number of data increases, the test response to the outlying data becomes more selective and the non-conformity of the data distribution with the assumption (data normally distributed) is indicated with an increased sensibility. Therefore, the significance of the Anderson-Darling test for the normal distribution of the data shall not be overestimated particularly if the number of data is rather low.

Taking the statistical nature of the data into account, it can be supposed that the **laboratory performance** has been **assessed properly** without any curtailment if the amount of data was high enough for a reliable and sufficiently sharp statistical evaluation. For this reason, **an internal limit number of participants (7) was set**. Really no doubts about the assessment reliability and correctness exist if additional collateral conditions were also met, i.e.:

- ✓ if the number of repeat measurements was high enough (to get a reasonable value of s_r)
- ✓ if the standard uncertainty of the assigned value $u_x \leq 0{,}3 \cdot s^*$, i.e. if the number of participants $p^* \geq 16$ (remark "NOT OK" concerning the standard uncertainty u_x appears in the table 'Test results' if $p^* < 16$); this requirement represents a considerable raising of the above mentioned limit
- ✓ if the data came from the normal distribution

On the other hand, the result of the **proficiency assessment** is **not very reliable** if the threshold number of participants (7) was not reached, and it is **slightly diminished**

- if the number of measurement repetitions was too low and the resulting ratio s_r / s^* too high or
- if the number of participants $p^* < 16$ or
- if the data did not come from a normal distribution

In such cases, laboratories could receive (false) warning signals (higher values of z-score) because of inaccuracy in the determination of the assigned value, not due to procedural flaws within the laboratories.

3.2.3 CHARTS AND PLOTS

In the **first diagram** all **mean values** $\bar{x}_i$ and the respective **standard deviations** s_i are plotted against the LabCodeNo. The **robust average** x^* , i.e., the assigned value X , is displayed by an orange horizontal line and the band width of ±**1 robust standard deviation** s^* is marked by two blue dotted lines in this chart. Additionally, general mean m obtained in the additional check of the test method accuracy (outliers eliminated) is displayed by a brown line for comparison with X .

In the **second diagram**, the ***z*-scores** obtained are plotted against the LabCodeNo. Additionally, the $z = 2$-level which helps to identify the outliers is displayed with an orange line in the z-score chart. The more common z-score was used in all tests in the SECTOR RR-II regardless of the ratio $u_x / s*$ resulting in the particular test. The problem of the too high ratio $u_x / s*$ caused by the too low number of participants could be solved if the lab proficiency would be assessed by means of z'-score instead of z-score. Drawbacks of the additional complexity seem to outweigh the advantage of the z'-score correctness. The only benefit of using z'-scores would be a slightly better performance, i.e., less number of 'warning signals' (z' > 2) and 'action signals' (z' > 3), in a few cases. In such situations ($p* << 16$) the participants are well-advised to look if the difference between their own result and the assigned value X is acceptable from the practical point of view, not (only) from the statistical point of view.

3.3 Terminology

Short basics concerning the corresponding calculation are presented in this chapter. The terms specified below were used in the statistical evaluation of the interlaboratory comparison. They are generally known or are defined, among other sources, in ISO 13528:2005 or ISO 5725-2:1994 including Technical Corrigendum 1:2002, as follows. See the list of symbols at the beginning of this Report for the symbol explanation if the explanation included here does not seem to be sufficiently clear.

Arithmetic mean, average ($\bar{x}$):

Quotient of the sum of independently identified individual values (in this test x_i) and their number n:

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

Note 1:

In the present proficiency test, the robust average $x*$ (i.e. assigned value for the proficiency test) is not an average value; it is derived from median of all $\bar{x}_i$ and calculated using the algorithm described in ISO 5725-5 and in ISO 13528:2005, Annex C.

Note 2:

In contrast to $x*$, the general mean m used in the additional check of the test method accuracy is an average value.

Note 3:

In the computing of the Grubbs' statistics, over-all average value $\bar{x}_x$ is used:

$$\bar{x}_x = \frac{1}{p^*}\sum_{i=1}^{p^*} \bar{x}_i$$

Variance (s^2):

Quotient of the sum of squares of deviations of the individual values from the arithmetic mean and ($n-1$), i.e. number of degrees of freedom:

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n} (x_i - \bar{x})^2$$

Standard deviation (s):

Positive value of the root of the variance of a series of measured values:

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2}$$

Note 1:

The robust standard deviation s^* is not calculated by the above simple formula but using the algorithm described in ISO 5725-5 and in ISO 13528:2005, Annex C.

Note 2:

In the computing of the Grubbs' statistics, standard deviation of the original results s_x is used:

$$s_x = \sqrt{\frac{1}{p^*-1}\sum_{i=1}^{p^*}(\bar{x}_i - \bar{x}_x)^2}$$

Coefficient of variation ($CV\%$):

Dispersion of individual results expressed as quotient of the standard deviation and arithmetic mean in percent.

Repeatability conditions:

Independent test results obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

Reproducibility conditions:

Test results obtained with the same method on identical test items in different laboratories with different operators using different equipment.

Repeatability variance (s_r^2):

Arithmetic mean of s_i^2 taken over all those labs taking part in the accuracy experiment which remained after outliers have been eliminated

$$s_r^2 = \frac{\sum_{i=1}^{p}(n_i - 1)s_i^2}{\sum_{i=1}^{p}(n_i - 1)}$$

Between-laboratory variance (s_L^2):

Term including between-operator and between-equipment variabilities, relating to experiments with single level and equal or unequal number of measurements in all labs (cf. ISO 5725-2:2002; Clause 7.4.5.2)

$$s_L^2 = \left\{\frac{1}{p-1}\left[\sum_{i=1}^{p} n_i(\bar{x}_i - m)^2\right] - s_r^2\right\} \quad / \quad \left\{\frac{1}{p-1}\left[\sum_{i=1}^{p} n_i - \left(\sum_{i=1}^{p} n_i^2 / \sum_{i=1}^{p} n_i\right)\right]\right\}$$

Where, owing to random effects, a negative value for s_L^2 was obtained from the calculations for a particular data set, the value was assumed to be zero (cf. ISO 5725-2:2002, Clause 7.4.5.4). In these cases, $r = R$ and $s_r = s_R$ results from the corresponding calculation, although this is generally not true (usually $R>r$).

Reproducibility standard deviation (s_R):

The standard deviation of test results obtained under reproducibility conditions:

$$s_R = \sqrt{(s_r^2 + s_L^2)}$$

Repeatability limit (r):

A value less than or equal to what the absolute difference between two test results obtained under repeatability conditions may be expected to be with a probability of 95%:

$$r = 2{,}8 \cdot s_r$$

Note:

Two test results obtained under repeatability conditions shall be judged not equivalent if they differ by more than the "r". Vice versa, two test results obtained under repeatability conditions shall be judged to be equivalent if they differ by less than the "r". Any such judgment would have an approx. 95 % probability of being correct. This may be an important perception particularly in accredited laboratories which are obliged to know the measurement uncertainty of applied testing methods (cf. ISO / IEC 17025) and in the assessment of obtained test results for compliance with specified limit values.

Reproducibility limit (R):

A value less than or equal to what the absolute difference between two test results obtained under reproducibility conditions may be expected to be with a probability of 95%:

$$R = 2{,}8 \cdot s_R$$

Note:

Two test results obtained under reproducibility conditions shall be judged not equivalent if they differ by more than the "R". Vice versa, two test results obtained under reproducibility conditions shall be judged to be equivalent if they differ by less than the "R". This may be an important perception particularly if results obtained in two or more labs are compared. Any such judgment would have an approx. 95 % probability of being correct.

Outlier according to Grubbs' test:

With this test, the extreme values of $\bar{x}_i$ ($x_{extr} = \bar{x}_{\max}$ or $\bar{x}_{\min}$) are tested to be an outlier ("outlier regarding the mean value")

$$Grubbs \quad criterion \quad G = \frac{\left| \bar{x}_x - \bar{x}_{extr} \right|}{s_x}$$

where

$\bar{x}_x$ = arithmetic mean of the inspected set of data $\bar{x}_i$
$\bar{x}_{extr}$ = extreme value of $\bar{x}_i$
s_x = standard deviation of the inspected set of data $\bar{x}_i$
and G-values for statistical outliers (probability 99%) and possible outliers, i.e. stragglers, (probability 95%) are listed in the corresponding literature.

Outlier according to Cochran's test:

With this test, the within-laboratory variances are tested for homogeneity ("outliers regarding standard deviations"):

$$Cochran\ \ criterion\ \ C = \frac{s_{max}^2}{\sum_{i=1}^{n} s_i^2}$$

where

s_{max} = highest value of s_i

and C-values for statistical outliers (probability 99%) and possible outliers, i.e. stragglers, (probability 95%) are listed in the corresponding literature.

Standard uncertainty of the assigned value (u_x):

When the robust average $x*$ is the assigned value, the standard uncertainty of the assigned value is estimated as:

$$u_x = \frac{1{,}25 \cdot s*}{\sqrt{p*}}$$

***z*-score:**

In the calculation of performance statistics the z-score is a commonly used variability measure.

$$z = \frac{(\bar{x}_i - X)}{\hat{\sigma}}$$

This score is used in different variants depending on the selection of $\hat{\sigma}$ and X values. The robust standard deviation $s*$ was set for $\hat{\sigma}$ and the robust average $x*$ was set for X in this PTS.

A z-score >2 denotes that the result of the respective laboratory deviates by more than $\pm 2\hat{\sigma}$ from the accepted reference value for the proficiency assessment X (= 'warning signal'). Approximately 95% of all results may lie in the interval $X \pm 2\hat{\sigma}$ if data is normally distributed. A z-score >3 shall be considered to give an "action signal", i.e., the respective laboratory shall start up to look for reasons of its extreme bias immediately.

The resulting data is assessed as follows:

$|z| \leq$ 1..........the performance of the laboratory is **very good**

1 < $|z| \leq$ 2..........the performance of the laboratory is **satisfactory**

2 < $|z| \leq$ 3..........the performance of the laboratory is **questionable**

$|z| >$ 3..........the performance of the laboratory is **unsatisfactory**

4 Results

RESULTS

SECTOR Round Robin II -

Torrefied Pellets

GRINDING ENERGY

according to method description

Remarks

Lab. No.:	Remarks
102	As we were not able to make test according the official protocol, we used our protocol. It consists on measuring continually the energy (integrated signal provided by the LEM device, step of 0.5 s) of the cutting mill in charge, less the energy used by the cutting mill without load (for the same period) and to divide the result with the weight of the dust collected in the collecting vessel. The dosing rate was around 2.7 g/s and the quantity tested for each test was around 1 kg.
108	Maximal possible mass flow was from 8.4 to 8.8 g/s. Power of cutting mill increased very slowly during milling. As a consequence, maximal total active power was reached only for about 50% of the milling process. For calculation, mean total active power of the whole milling process was used.
115	Feeding rate of pellets to the mill varied between 4.6 - 4.9 g/s in the experiments, which was much lower than the value given in the method description. Higher feeding rates caused flooding and accumulation of material to the mill. To get the same material feeding rate from the start to the end, the level of pellets in the feeding silo was kept constant and material was fed to the mill for 525 seconds in all the experiments. It is hard to determine the duration of milling, because the power curve is not so ideal as in the example figure. The pellet feeding time was used as duration of milling in calculations. It is not precisely said in the method description what is the mass that should be used in calculations. The mass of milled material was used in calculations of these grinding energies.
119	1) Due to the volume of mill container the sample size was reduced to approximately 1,5 kg per determination. 1) Screening of the pellets did not result with a substantial amount of fines, but due to abrasion it can have an impact to the sample. 2) The milling process is not completed when no more material is fed to the mill. It continues for some time until the whole sample is grinded. Therefore it is challenging (based on the method description) to retrieve the point when the grinding is finished. We determined the point of grinding finish based on the plotted power vs. time (number of samples per s = 10) chart. Maybe it would be better to analyse only the data in power peak values and calculate the mass of the grinded sample based on the flow rate and time.
123	Initial flowrate: 19,3 g/s. It is unclear what tG (duration of milling) is. We take tG as the time from starting the metering unit until it is turned off (no more pellets is falling down into the mill), as these are the times to be recorded according to the Method Description. It could also be the times from pellets entering the mill until the milling process is completed (mill empty).
126	The requested 15-20 g/s feeding speed was not possible with our machine. After consultation with OFI it was decided to feed 2,5 kg pellets manually and log the energy consumed in grinding the total batch. Addition of the grinding energy minus the idle energy per second has led to the required grinding energy.
127	The average feed rate was only 3,7 g/s
130	Mass flow = 2 g/s
131	Mass flow = 2 g/s

Additional information

Lab. No.:	Metering unit	Cutting mill
102	by gravity	Retsch SM100 with a grid of 1mm
108	Retsch DR of Retsch ZM 100	Retsch SM 2000
115	Silo for pellets + vibrating feeder	Universal cutting mill Fritsch Pulverisette 19, trapezoidal 1 mm sieve
119	Retsch DR 100	Retsch SM 300 (3000 m-1; 6-disc rotor; 1.0 mm trapezoid holes sieve)
121	-	Fritsch Pulverisette 19
123	Retsch ZR1	Retsch SM300
126	manual feeding	Retsch SM2000
127	Watt	Pulverisette 19, 1 mm trapezoidal perforation
129	Metering screw	Fritsch Pulverisette 19
130	None	Fritsch Pulverisette 19
131	None	Fritsch Pulverisette 19

Grinding Energy E_m Torrefield pellets

Method description grindability

Results submitted by participants

i.e., individual results x_{ik}
+ number of the test repetitions made by each lab (n_i)
+ within laboratory means (x_i) and standard deviations (s_i)
+ results of tests for outliers

Number of reporting laboratories p*: 11
Number of reported test results Σn_i: 27

Lab Code No.	Test results in Wh/kg – Test replication No. (k) 1	2	3	4	5	6	Statistical evaluation of the submitted test results x_{ik}: n_i	x_i	s_i	Outliers: Cochran	Grubbs	z > 2
115	4,9	5,4					2	5,15	0,3536			
102	9,7	9,3	9,4				3	9,47	0,2082			
129	9,4	10,6					2	10,02	0,8344			
127	13,7	13,9					2	13,80	0,1414			
131	14,5	14,9	13,6				3	14,33	0,6658			
130	15,3	15,6					2	15,45	0,2121			
121	16,3	16,7	15,9				3	16,30	0,4000			
108	18,3	18,1	17,5				3	17,98	0,4230			
126	24,2	22,6					2	23,40	1,1314			
119	25,5	26,9					2	26,20	0,9899			
123	29,2	30,7	28,7				3	29,53	1,0408			

** ... statistical outlier (99%) * ... straggler (95%)
X ... z > 2

Results of robust statistics

Convergence assumed at iteration number: 9

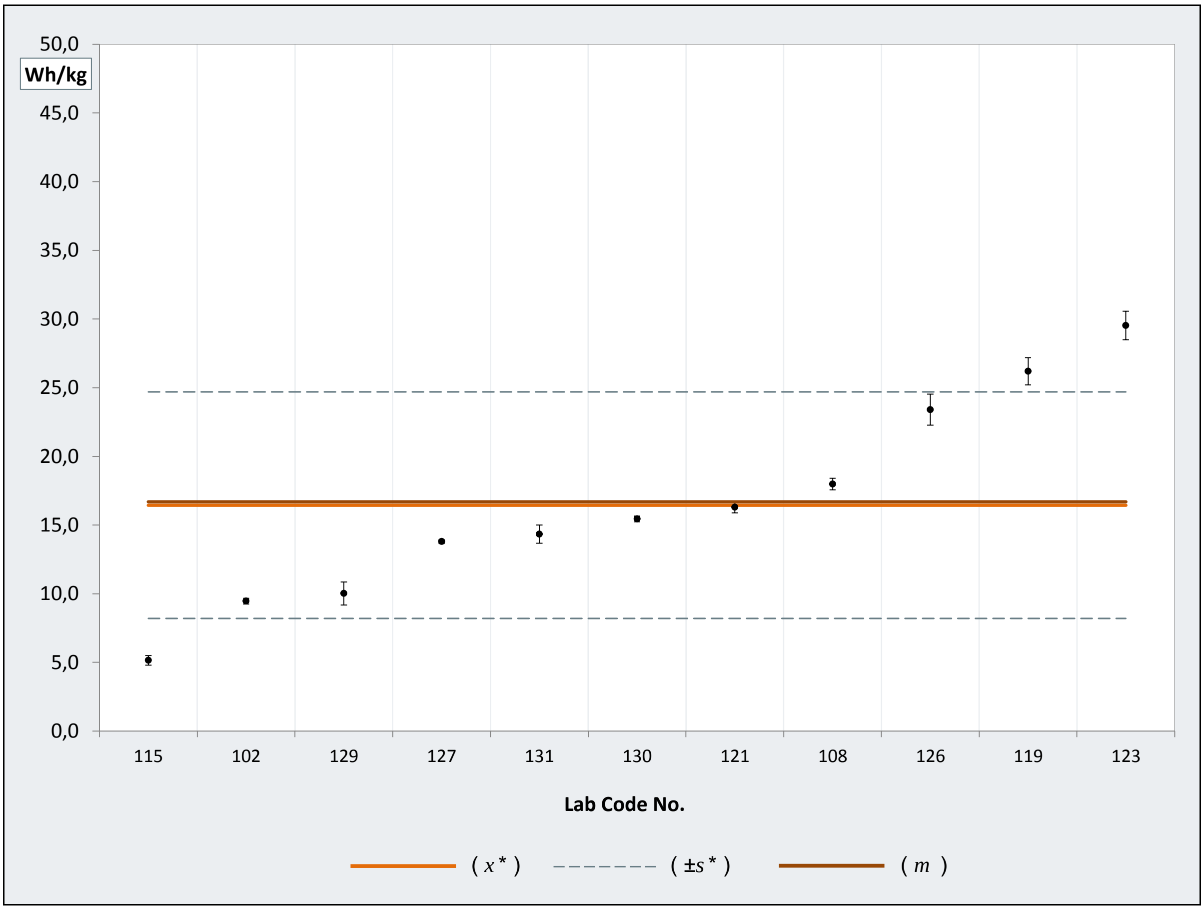
Robust average: x^* =	**16,4**	assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: s^* =	**8,25**		
Number of repeate measurements necessary due to s_r/s^*-ratio: n' =	1	**OK**	see page 4 for the meaning of **NOT OK**
Standard uncertainty of the assigned value: u_x =	3,10859	**NOT OK**	

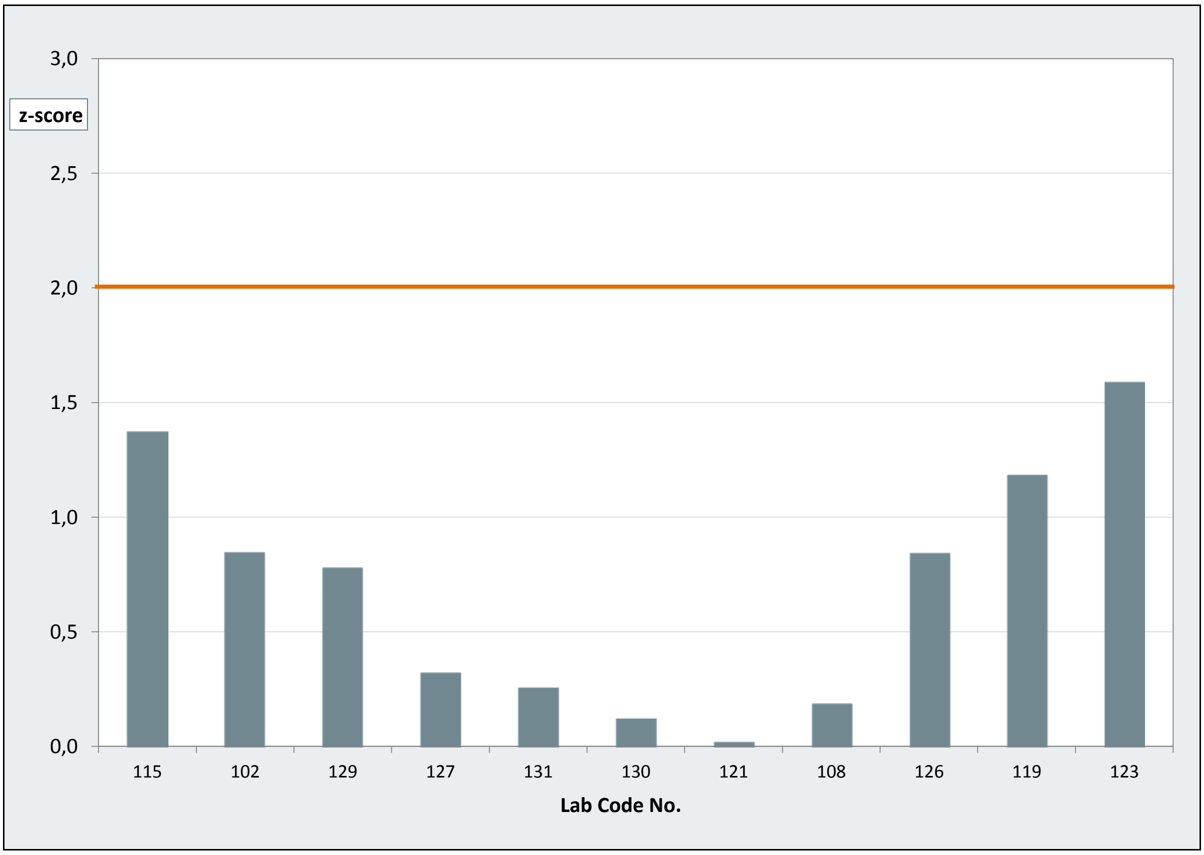
Additional check of the test method accuracy

Do the input data come from a normal distribution ? (The results listed below shall be considered as really justified only if the input data come from a normal distribution)			**YES**
General mean $\Sigma n_i x_{ik} / \Sigma n_i$	m	16,7	Wh/kg
Repeatability variance	s_r^2	0,4352500	
Repeatability standard deviation	s_r	0,65973	Wh/kg
Repeatability coefficient of variation	$CV\%_r$	3,951	%
Between-laboratory variance	s_L^2	53,8023062	
Between-laboratory standard deviation	s_L	7,33501	Wh/kg
Between-laboratory coefficient of variation	$CV\%_L$	43,924	%
Reproducibility variance s_R^2	$s_r^2 + s_L^2$	54,2375562	
Reproducibility standard deviation	s_R	7,3646	Wh/kg
Reproducibility coefficient of variation	$CV\%_R$	44,101	%
Repeatability limit	r	**1,8**	Wh/kg
Relative repeatability limit	r_{rel}	**11,06**	%
Reproducibility limit	R	**20,6**	Wh/kg
Relative reproducibility limit	R_{rel}	**123,48**	%
Number of participants included in the accuracy evaluation	p	11	
Number of tests included in the accuracy evaluation	Σn	27	

Performance of individual laboratories in the specific test or measurment

Grinding Energy - E_m - Torrefield pellets





RESULTS

SECTOR Round Robin II -

Torrefied Pellets

WATER ABSORPTION

according to method description

Remarks

Lab. No.:	Remarks
103	Plastic bag from sample B had two holes.
105	There appears to be an issue with trial 2 in which WA and the stabilized moisture content are in the same general range, but the durability loss is much less. Perhaps this data point can be excluded.
110	Replicate 4 had more untorrefied wood peices left then the others. Water temperature 19-21 degrees celsius.
112	WA - Mean value: 39.22%; Standard Deviation: 0.59% Du loss - Mean value: 17.38%; Standard Deviation: 1.83%
113	in Versuch 3 wurde zum Trennen von Wasser und Pellets eins Sieb mit der Porenweite 315 µm benutzt. Bei allen anderen Versuchen wurde dafür ein groberes Sieb verwendet
115	Moisture content according to EN 14774-2 should be reported 0.1% accuracy Mechanical durability of pellets according to EN 15210-1 should be reported 0.1% accuracy
118	During tests we noticed that it is very important to define sieve which is appropriate for separation wet pellets from water (30 minutes). We used 3,15 mm round hole sieve, and we had significant sample loss through these holes. It is also important to define how to calculate water content after stabilization. We weren't sure what weights we should use to determine water content after stabilization.
123	The "sieve appropriate for liquid/solid separation" should be specified! The problem is, that all particles should be retained in this sieve - still allowing the water to drip off. We did not quite know what to use - so we used two pairs of tights (!)
125	Test done in triplicate as this is in line with EN 15210-1 for the durability test. Note that we are unclear as to why the moisture content of the as received pellet is included in the calculation of WA as this will include a contribution from the inherent moisture content which shouldn´t be counted as "absorbed". Also, the immersed pellet were substantially more degraded than indicated by the loss in durability, with very few of the pellets remaining intact and a substantial portion of the material in the 3,15 – 10 mm range.
127	WC% is made on the pellets after mechanical durability and is only one determination instead of the two determinations according to the standard. The pellets stood to stabilize for two days after they had been in water and dried.
128	Information about "appropriate sieve" is missing. Depending on mesh size or round hole size or size of openings the disintegrated parts of pellets can maybe go trough the sieve and are missing on the weight of pellets. Also the separation of pellets and liquid is depending on the choose sieve, so the sieve should be exactly defined by method. It is not described how to proceed with pellet parts in the soaking pan after 1h leaching procedure. How this small particles should be removed from the soaking pan (rinse?, mechanically removed?). The temperature of water for leaching is not given by method. This information is very important, because the disintegration of pellets is depending on water temperature (see EN16126).
129	- not stirred, but dip in water vertical - a cover plate was installed over the soaking pan to avoid floating of the pellets

Additional information

Lab. No.:	Moisture in analysis sample (w-%)	Original mechanical durability (%)
102	8,95	98,65
103	8,8	
105	8,87	98,5
106	8,93	98,58
107	9,21	
108	1,19	98,2
109	9,18	98,38
110	9,3	98,3
111	9,35 (1st and 2nd test) and 8,6 (3rd and 4th test)	98,14
112	9,15	98,33
113	7,1 (16.07.14 Analyse 1+2), 8,8 (23.07.14 Analyse 3+4), 8,8 (19.08.14 Analyse 5)	98,5
114	8,77	98,42
115	9,3	98,4
117	5,9	98,49
118	9,01	98,5
121	8,72	98,20
123	9,15 (moisture of the original sample, WCorg)	98,32 and 98,24, i.e. mean 98,28 %
124	9,33	98,55
125	7,60	98,47
126	9,26	98,36
127	8,9	98,2
128	8,86	98,5
129	7,66	98,5
130	8,29	98,24
131	5,33	98,11

Water absorbtion WA Torrefield pellets

Method description water absorbtion

Results submitted by participants

i.e., individual results x_{ik}
+ number of the test repetitions made by each lab (n_i)
+ within laboratory means (x_i) and standard deviations (s_i)
+ results of tests for outliers

Number of reporting laboratories p *: 25
Number of reported test results Σn_i: 110

Lab Code No.	Test results in % — Test replication No. (k) 1	2	3	4	5	6	Statistical evaluation of the submitted test results x_{ik} — n_i	x_i	s_i	Outliers — Cochran	Grubbs	z > 2
108	23,08	23,17	25,82	27,14			4	24,803	2,0109	*		X
106	29,08	28,92	29,92	30,34			4	29,565	0,6777			
114	29,93	30,03	29,54	29,22			4	29,680	0,3725			
102	32,46	31,85	32,11	32,23	32,62		5	32,254	0,3004			
131	32,33	32,16	32,33	33,16	33,49	32,49	6	32,660	0,5358			
126	33,01	33,21	33,11	32,56			4	32,973	0,2869			
~~117~~	~~35,30~~	~~32,00~~	~~39,20~~	~~34,80~~			4	~~35,325~~	~~2,9635~~	**		
103	35,44	35,31	35,42	35,19			4	35,340	0,1152			
123	35,78	35,95	35,82	35,88			4	35,858	0,0741			
109	36,13	35,90	35,84	36,25			4	36,030	0,1927			
110	37,83	35,72	36,03	37,34	36,07		5	36,598	0,9275			
125	36,40	36,60	37,20				3	36,733	0,4163			
124	37,05	37,77	36,93	37,00	36,68		5	37,086	0,4080			
113	36,50	37,20	40,10	38,20	37,00		5	37,800	1,4265			
130	37,82	37,61	39,26	38,42			4	38,278	0,7395			
112	39,68	39,91	39,11	38,92	38,27	39,45	6	39,223	0,5908			
107	39,18	39,06	39,56	39,38			4	39,295	0,2205			
129	41,12	39,00	38,49	39,63			4	39,560	1,1397			
115	39,50	40,40	40,20	39,20			4	39,825	0,5679			
128	39,03	40,78	40,25				3	40,020	0,8974			
118	38,90	40,82	39,22	41,37	44,03	42,25	6	41,098	1,9201			
127	42,34	40,54	41,17	41,05	40,79	41,01	6	41,150	0,6241			
121	43,12	44,26	44,16	44,02			4	43,890	0,5227			
105	45,87	44,74	42,34	43,88			4	44,208	1,4880			
~~111~~	~~48,33~~	~~48,53~~	~~40,17~~	~~41,10~~			4	~~44,533~~	~~4,5172~~	**		

** ... statistical outlier (99%) * ... straggler (95%)
X ... z > 2

Results of robust statistics

Convergence assumed at iteration number: 16

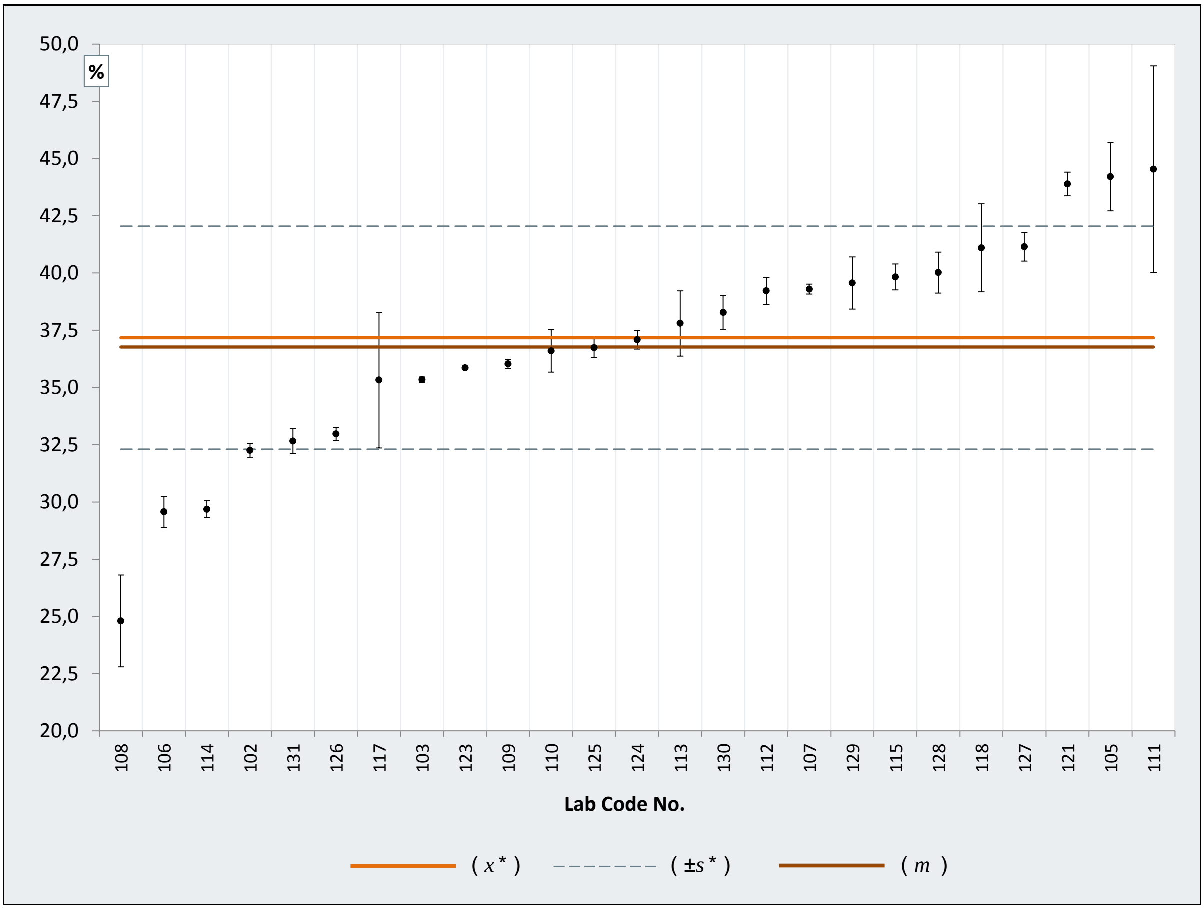
Robust average: x^* =	**37,17**	assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: s^* =	**4,875**		
Number of repeate measurements necessary due to s_r/s^*-ratio: n' =	1	OK	see page 4 for the meaning of **NOT OK**
Standard uncertainty of the assigned value: u_x =	1,21863	OK	

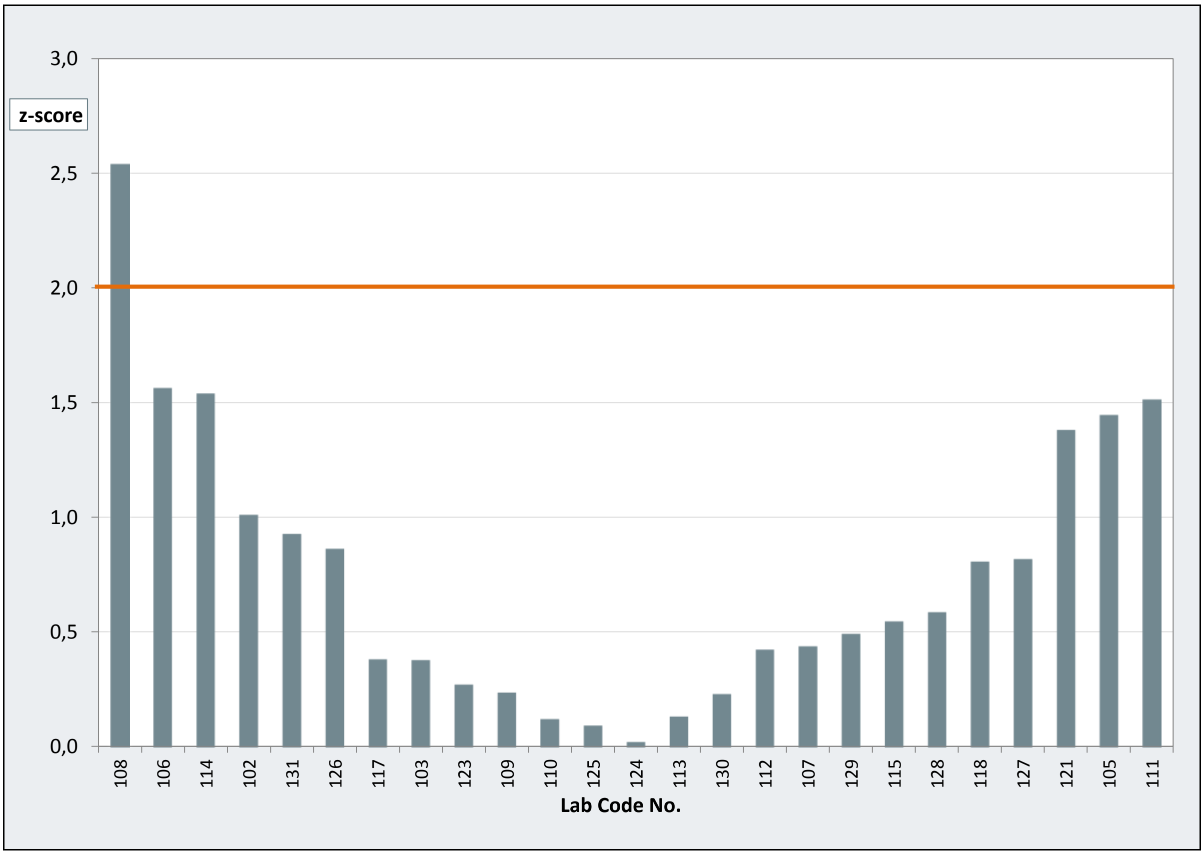
Additional check of the test method accuracy

Do the input data come from a normal distribution ? (The results listed below shall be considered as really justified only if the input data come from a normal distribution)		**NO**	
General mean $\Sigma n_i x_{ik} / \Sigma n_i$	m	**36,77**	%
Repeatability variance	s_r^2	0,8427706	
Repeatability standard deviation	s_r	0,91803	%
Repeatability coefficient of variation	$CV\%_r$	2,496	%
Between-laboratory variance	s_L^2	21,2146921	
Between-laboratory standard deviation	s_L	4,60594	%
Between-laboratory coefficient of variation	$CV\%_L$	12,525	%
Reproducibility variance s_R^2	$s_r^2 + s_L^2$	22,0574627	
Reproducibility standard deviation	s_R	4,6965	%
Reproducibility coefficient of variation	$CV\%_R$	12,771	%
Repeatability limit	r	**2,57**	%
Relative repeatability limit	r_{rel}	**6,990**	%
Reproducibility limit	R	**13,15**	%
Relative reproducibility limit	R_{rel}	**35,760**	%
Number of participants included in the accuracy evaluation	p	23	
Number of tests included in the accuracy evaluation	Σn	102	

Performance of individual laboratories in the specific test or measurment

water absorbtion - WA - Torrefield pellets





Loss of mechanical durability $D_{U\ loss}$ **Torrefield pellets**

Method description water absorbtion

Results submitted by participants

i.e., individual results x_{ik}
+ number of the test repetitions made by each lab (n_i)
+ within laboratory means (x_i) and standard deviations (s_i)
+ results of tests for outliers

Number of reporting laboratories p*: 23
Number of reported test results Σn_i: 101

Lab Code No.	Test results in % — Test replication No. (k) 1	2	3	4	5	6	Statistical evaluation of the submitted test results x_{ik}: n_i	x_i	s_i	Outliers: Cochran	Grubbs	z>2
114	7,02	6,90	5,14	5,00			4	6,015	1,0938			X
131	8,31	9,77	10,77	11,89	12,03	11,14	6	10,652	1,4104			
126	10,45	10,96	11,68	10,69			4	10,945	0,5324			
108	11,08	10,59	11,91	11,85			4	11,358	0,6361			
124	9,54	13,10	12,63	12,23	11,59		5	11,818	1,3887			
113	11,50	12,30	13,30	12,00	-		4	12,275	0,7588			
106	11,65	12,87	13,15	11,92			4	12,398	0,7249			
123	12,33	12,51	13,23	12,58			4	12,663	0,3927			
~~105~~	~~15,03~~	~~7,21~~	~~14,31~~	~~14,53~~			~~4~~	~~12,770~~	~~3,7189~~	**		
130	12,08	12,75	13,28	13,34			4	12,863	0,5852			
128	12,40	13,25	13,01				3	12,887	0,4382			
129	13,48	12,57	13,14	12,75			4	12,985	0,4068			
110	13,87	12,25	12,30	13,86	12,78		5	13,012	0,8057			
121	12,93	13,20	13,20	13,62			4	13,238	0,2850			
111	14,00	15,33	11,86	12,52			4	13,428	1,5522			
109	13,01	13,78	13,09	14,31			4	13,548	0,6147			
117	11,40	12,50	16,90	13,80			4	13,650	2,3784			
115	14,50	13,60	13,60	13,80			4	13,875	0,4272			
127	16,04	16,35	16,10	16,95	15,41	16,11	6	16,160	0,4986			
112	18,24	18,37	17,13	16,51	14,38	19,66	6	17,382	1,8296			
118	18,27	19,59	17,46	19,40	20,06	18,88	6	18,943	0,9520			X
125	18,49	19,64	19,64				3	19,257	0,6640			X
~~102~~	~~33,40~~	~~28,22~~	~~32,28~~	~~32,51~~	~~28,57~~		~~5~~	~~30,996~~	~~2,4141~~	*	**	X
103			no results reported									X
107			no results reported									X

** ... statistical outlier (99%) * ... straggler (95%)
X ... z > 2

Results of robust statistics

Convergence assumed at iteration number: 32

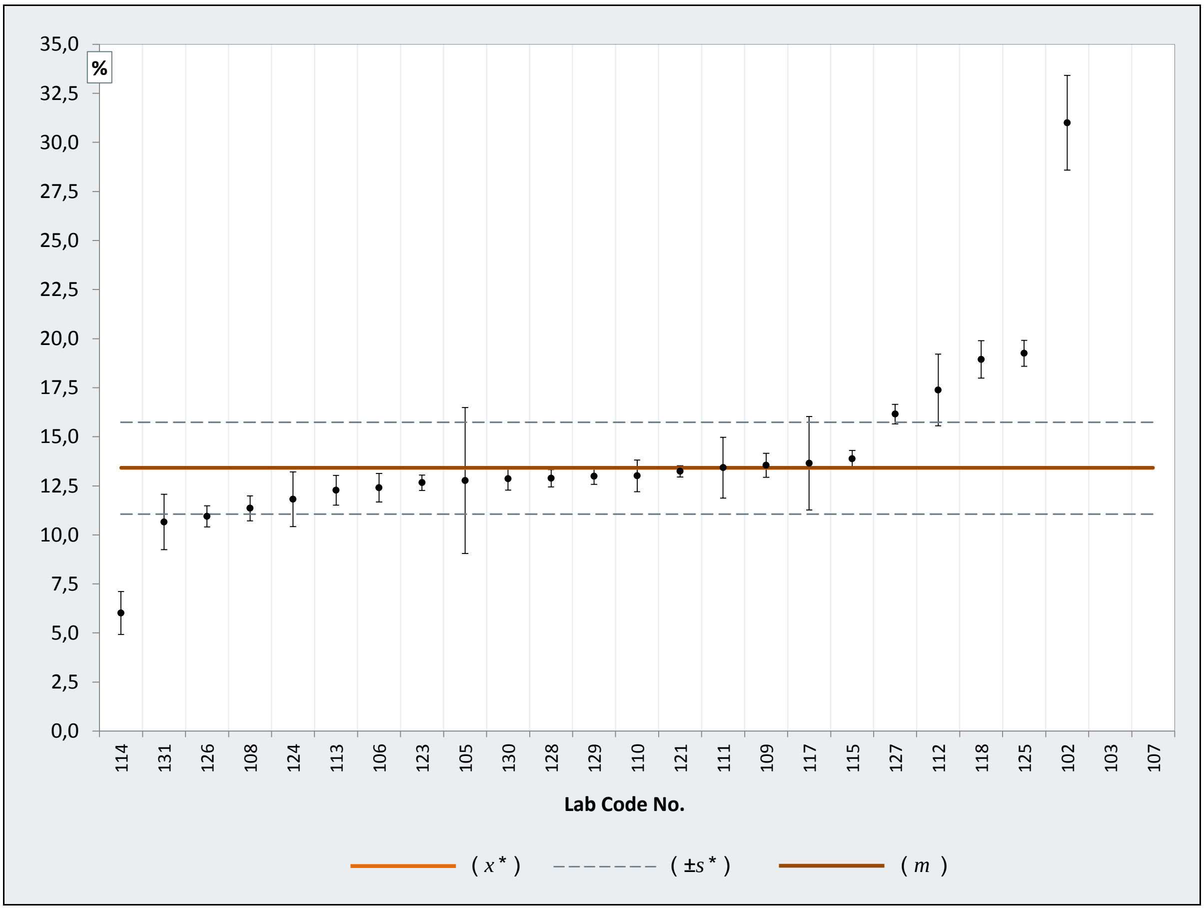
Robust average: x* =	13,39	assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: s* =	2,342		
Number of repeate measurements necessary due to s_r/s*-ratio: n' =	3	OK	see page 4 for the meaning of **NOT OK**
Standard uncertainty of the assigned value: u_x =	0,61038	OK	

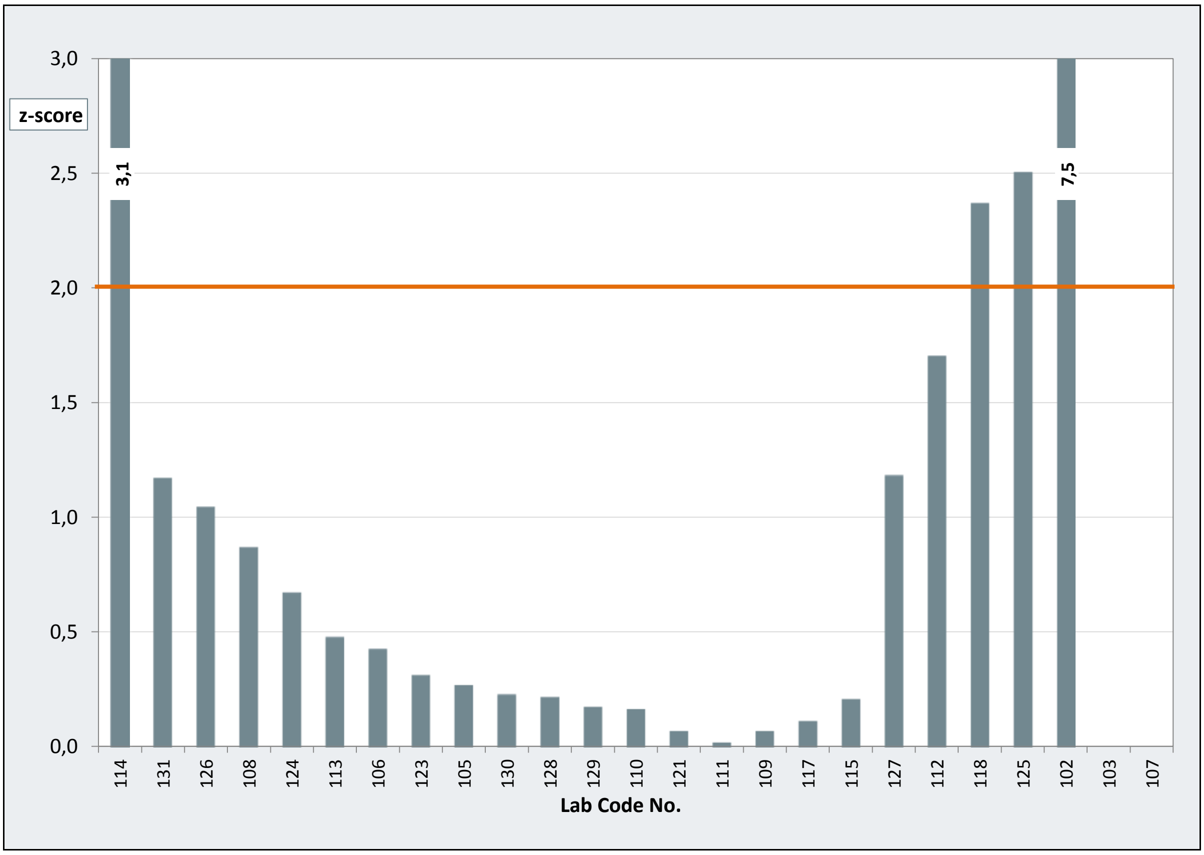
Additional check of the test method accuracy

Do the input data come from a normal distribution ? (The results listed below shall be considered as really justified only if the input data come from a normal distribution)		**NO**	
General mean $\Sigma n_i x_{ik} / \Sigma n_i$	m	13,44	%
Repeatability variance	s_r^2	1,1422707	
Repeatability standard deviation	s_r	1,06877	%
Repeatability coefficient of variation	$CV\%_r$	7,953	%
Between-laboratory variance	s_L^2	8,4393655	
Between-laboratory standard deviation	s_L	2,90506	%
Between-laboratory coefficient of variation	$CV\%_L$	21,617	%
Reproducibility variance s_R^2	$s_r^2 + s_L^2$	9,5816361	
Reproducibility standard deviation	s_R	3,0954	%
Reproducibility coefficient of variation	$CV\%_R$	23,034	%
Repeatability limit	r	**2,99**	%
Relative repeatability limit	r_{rel}	**22,268**	%
Reproducibility limit	R	**8,67**	%
Relative reproducibility limit	R_{rel}	**64,495**	%
Number of participants included in the accuracy evaluation	p	21	
Number of tests included in the accuracy evaluation	Σn	92	

Performance of individual laboratories in the specific test or measurment

loss of mechanical durability - $D_{U\ loss}$ - Torrefield pellets





RESULTS

SECTOR Round Robin II -

Torrefied Pellets

CARBON CONTENT

according to EN 15104

Remarks

Lab. No.:	Remarks
110	The determination was made on dry samples
112	std.dev. = 0,2467; % Rel. S.D. = 0,476; variance = 0,0609 Analysed fraction of samples below 0.2 mm. Multilevel calibration tested on Birch Leaf Std (producer - Elemental Microanalysis Ltd - CatNo.B2166-Certificate no.136621) Substance calibration - producer - Elemental Microanalysis Ltd: 1. BBOT OAS (CatNo.B2044&B2046,Certificate no.200648); 2. Sulphanilamide OAS (CatNo B2036&B2048, CerNo 183407); 3.wheat Flour Std OAS (CatNo B2156, CerNo 114857); 4. High Organic Sediment Std OAS (CatNo B2150, CerNo 175032); 5. Soy Bean Meal Std (CatNo B2271, CerNo 413C)
122	We have used a combustion tube – heated to 1350°C, gases evolved collected in absorption bottles. Gravimetric determination
125	BBOT is the acronym for (2,5-di(5-tert-butylbenzoxazol-2-yl)thiophene)

Additional information

Lab. No.:	Moisture in analysis sample (w-%)
101	9,14 / 9,23 / 8,74
102	8,5
104	1,86
105	8,04
107	6,2
109	7,48
110	<0,1
111	5,44
112	6,68
113	8,4
114	7,30
116	8,44
118	0,7
120	8,3
121	4,87
122	8,45
123	9,1
124	9,33
125	5,97
126	9,26
128	8,6
129	8,66
130	6,24
131	5,33

Carbon content *C* Torrefield pellets

EN 15104

Results submitted by participants

i.e., individual results x_{ik}

+ number of the test repetitions made by each lab (n_i)

+ within laboratory means (x_i) and standard deviations (s_i)

+ results of tests for outliers

Number of reporting laboratories p *: 24

Number of reported test results Σn_i: 102

Lab Code No.	Test results in w-% — Test replication No. (k) 1	2	3	4	5	6	Statistical evaluation of the submitted test results x_{ik}: n_i	x_i	s_i	Outliers: Cochran	Grubbs	z > 2
131	47,47	47,29	47,07	47,86	47,37		5	47,412	0,2906		*	X
113	48,00	48,20	48,00	48,30			4	48,125	0,1500			X
121	49,31	49,39	49,27	48,97	49,16		5	49,220	0,1625			
130	50,42	49,99	50,26	50,12	50,12		5	50,182	0,1638			
102	50,40	50,53	50,31	49,97	49,98		5	50,238	0,2525			
126	51,00	51,00	51,00	50,00	51,00		5	50,800	0,4472	*		
104	51,18	51,31	51,27				3	51,253	0,0666			
114	51,44	51,49	51,22	51,13			4	51,320	0,1726			
124	51,19	51,57	51,50	51,42	51,30		5	51,396	0,1527			
112	51,94	51,78	51,63	51,60	52,19		5	51,828	0,2435			
120	51,98	51,95	52,00	52,19	52,03		5	52,030	0,0941			
107	52,19	52,00	52,25				3	52,147	0,1305			
118	51,93	52,17	52,16	52,27	52,23		5	52,152	0,1320			
111	52,20	52,18	52,18	52,10			4	52,165	0,0443			
110	52,30	52,30	52,39	52,29			4	52,320	0,0469			
122	52,23	52,39	52,44	52,28	52,31		5	52,330	0,0846			
109	52,39	52,38	52,24	52,33			4	52,335	0,0686			
105	52,48	52,46	52,43	52,40	52,49		5	52,452	0,0370			
116	52,48	52,49	52,39				3	52,453	0,0551			
125	52,34	52,71	52,76	52,60			4	52,603	0,1873			
123	52,72	52,70	52,80				3	52,740	0,0529			
129	52,76	52,82	52,61	52,82	52,82		5	52,766	0,0910			
101	54,01	54,41	53,70				3	54,040	0,3559			
128	53,76	54,24	54,50				3	54,167	0,3754			

** ... statistical outlier (99%) * ... straggler (95%)

X ... z > 2

Results of robust statistics

Convergence assumed at iteration number: 21

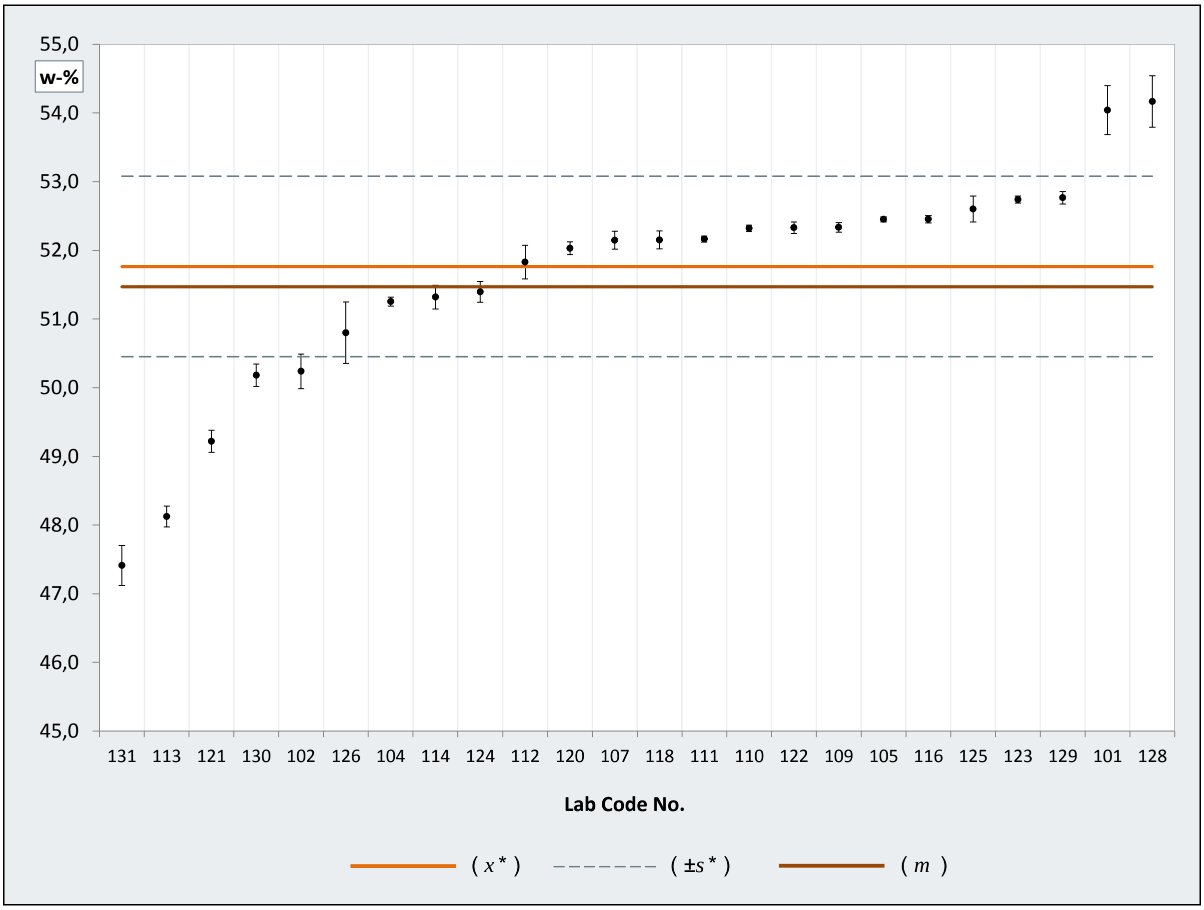
Robust average: x^* =	**51,77**	assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: s^* =	**1,313**		
Number of repeate measurements necessary due to s_r/s^*-ratio: n' =	1	**OK**	see page 4 for the meaning of **NOT OK**
Standard uncertainty of the assigned value: u_x =	0,33509	**OK**	

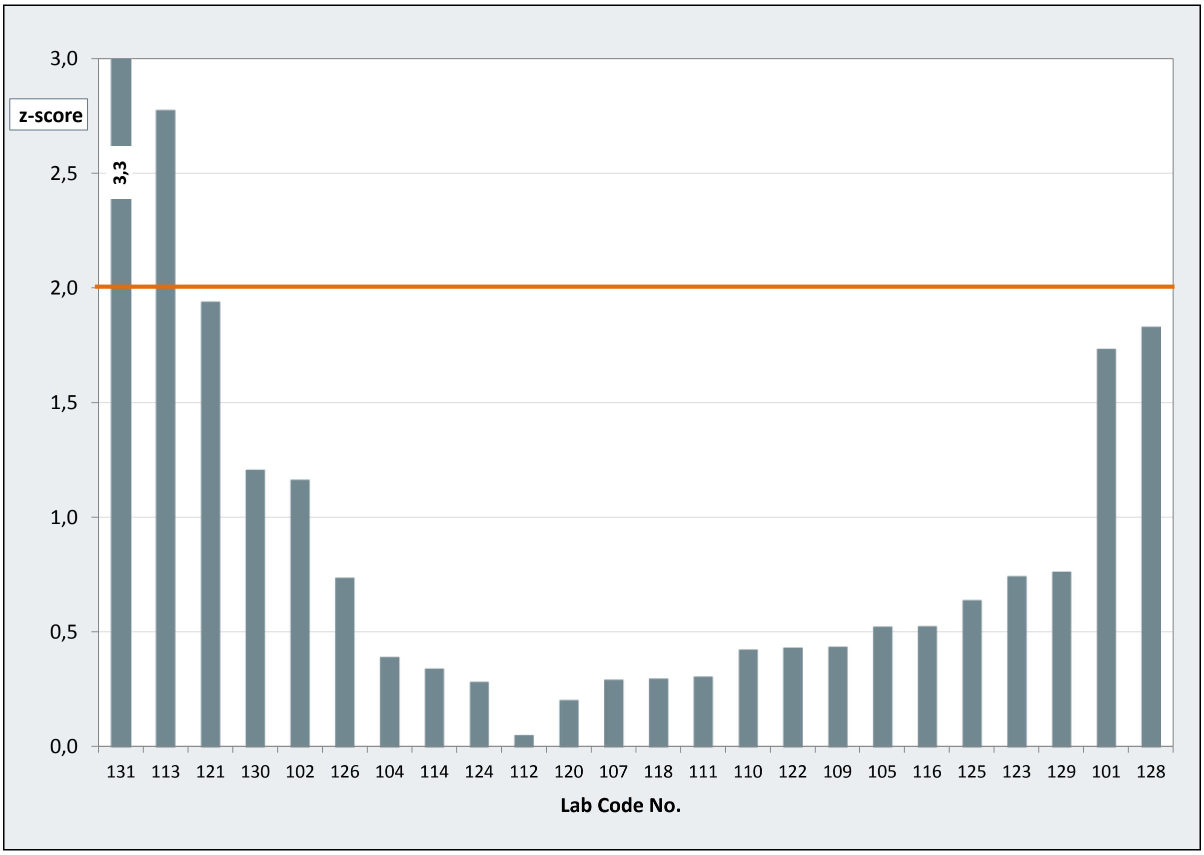
Additional check of the test method accuracy

Do the input data come from a normal distribution ? (The results listed below shall be considered as really justified only if the input data come from a normal distribution)		**NO**	
General mean $\Sigma n_i x_{ik} / \Sigma n_i$	m	**51,47**	w-%
Repeatability variance	s_r^2	0,0382944	
Repeatability standard deviation	s_r	0,19569	w-%
Repeatability coefficient of variation	$CV\%_r$	0,380	%
Between-laboratory variance	s_L^2	2,6135574	
Between-laboratory standard deviation	s_L	1,61665	w-%
Between-laboratory coefficient of variation	$CV\%_L$	3,141	%
Reproducibility variance s_R^2	$s_r^2 + s_L^2$	2,6518518	
Reproducibility standard deviation	s_R	1,6285	w-%
Reproducibility coefficient of variation	$CV\%_R$	3,164	%
Repeatability limit	r	**0,55**	w-%
Relative repeatability limit	r_{rel}	**1,065**	%
Reproducibility limit	R	**4,56**	w-%
Relative reproducibility limit	R_{rel}	**8,859**	%
Number of participants included in the accuracy evaluation	p	24	
Number of tests included in the accuracy evaluation	Σn	102	

Performance of individual laboratories in the specific test or measurment

Carbon content - *C* - Torrefield pellets





RESULTS

SECTOR Round Robin II -

Torrefied Pellets

GROSS CALORIFIC VALUE

according to EN 14918

Remarks

Lab. No.:	Remarks
104	Sulphur & nitrates utilising Parr 1108CL bomb and EN-15289
112	Mean value: 20663 J/g STD dev: 31.9 J/g; RSD: 0.154%; Correctness of method: 0.12%; Extended uncertainty: 130 J/g
116	Sample milling < 1mm and pressed pellet. Calibration Benzoic Acid AR 208C Alpha
123	For the calculation of the Gross calorific values a sulphur content of 0,05 w-% dry basis is used.
125	Sample was analysed 3 times on one day (first three) then a 4th time another day, the 4th result has a spate moisture result.

Additional information

Lab. No.:	Moisture in analysis sample (w-%)	Method		
		Adiabatic	Isoperibol	Other
101	9,14/9,23/8,74			Dynamic
102	8,9			Compensed static
103	8,8	X		
104	1,86		X	
105	8,04		X	
106	0,96	X		
107	-	-	-	-
108	7,69	X		
109	7,86		X	
110	3,72		X	
111	5,44		X	
112	6,31		X	
113	8,4		X	
114	7,30		X	
116	8,44	X		
117	9,1	X		
118	0,7		X	
120	8,3		X	
121	4,87	X		
122	8,45		X	
123	9,1		X	
124	0			Doppeltrocken
125	6,05 (first three) / 5,97 (last)		X	
126	9,26	X		
127	7,7	X		
128	8,6	X		
129	9,04	X		
130	6,24	X		
131	5,33	X		

Gross calorific value q_{gross} torrefied pellets

EN14918

Results submitted by participants

i.e., individual results x_{ik}
+ number of the test repetitions made by each lab (n_i)
+ within laboratory means (x_i) and standard deviations (s_i)
+ results of tests for outliers

Number of reporting laboratories p *: 29
Number of reported test results Σn_i: 116

Lab Code No.	Test results in J/g – Test replication No. (k) 1	2	3	4	5	6	Statistical evaluation of the submitted test results x_{ik} – n_i	x_i	s_i	Outliers – Cochran	Grubbs	$z > 2$
~~113~~	~~18931~~	~~19001~~	~~18935~~	~~18967~~			~~4~~	~~18958,5~~	~~32,593~~		**	X
121	20195	20163	20241				3	20199,7	39,209			X
~~124~~	~~20099~~	~~20559~~	~~20510~~	~~20115~~	~~20465~~		~~5~~	~~20349,6~~	~~224,015~~	**		
131	20396	20404	20384	20377	20451		5	20402,4	29,108			
126	20500	20400	20400	20400	20400		5	20420,0	44,721			
104	20363	20520	20453				3	20445,3	78,780			
118	20445	20460	20410	20481	20479		5	20455,0	29,163			
130	20471	20409	20500				3	20460,0	46,487			
102	20515	20658	20432	20506	20595		5	20541,2	87,159			
114	20615	20592	20495				3	20567,3	63,689			
106	20525	20595	20604				3	20574,7	43,247			
122	20690	20620	20510	20630	20620		5	20614,0	65,038			
112	20645	20639	20661	20709			4	20663,5	31,723			
108	20685	20670	20690	20682	20684		5	20682,2	7,430			
111	20708	20758	20722	20628	20635		5	20690,2	56,659			
128	20661	20688	20736				3	20695,0	37,987			
105	20707	20705	20713	20687			4	20703,0	11,195			
109	20731	20731	20744	20727			4	20733,3	7,411			
129	20713	20762	20785	20719	20691		5	20734,0	38,406			
107	20710	20730	20770				3	20736,7	30,551			
125	20724	20741	20733	20767			4	20741,3	18,518			
127	20703	20758	20765	20769	20771		5	20753,2	28,499			
110	20752	20792	20781	20710			4	20758,8	36,619			
120	20736	20790	20819	20699			4	20761,0	53,771			
116	20750	20759	20753	20774	20792		5	20765,6	17,416			
123	20783	20773	20746				3	20767,3	19,140			
117	20773	20795	20780				3	20782,7	11,240			
103	20810	20890	20870				3	20856,7	41,633			
101	20860	20930	20970				3	20920,0	55,678			

** ... statistical outlier (99%) * ... straggler (95%)
X ... $z > 2$

Results of robust statistics

Convergence assumed at iteration number: 17

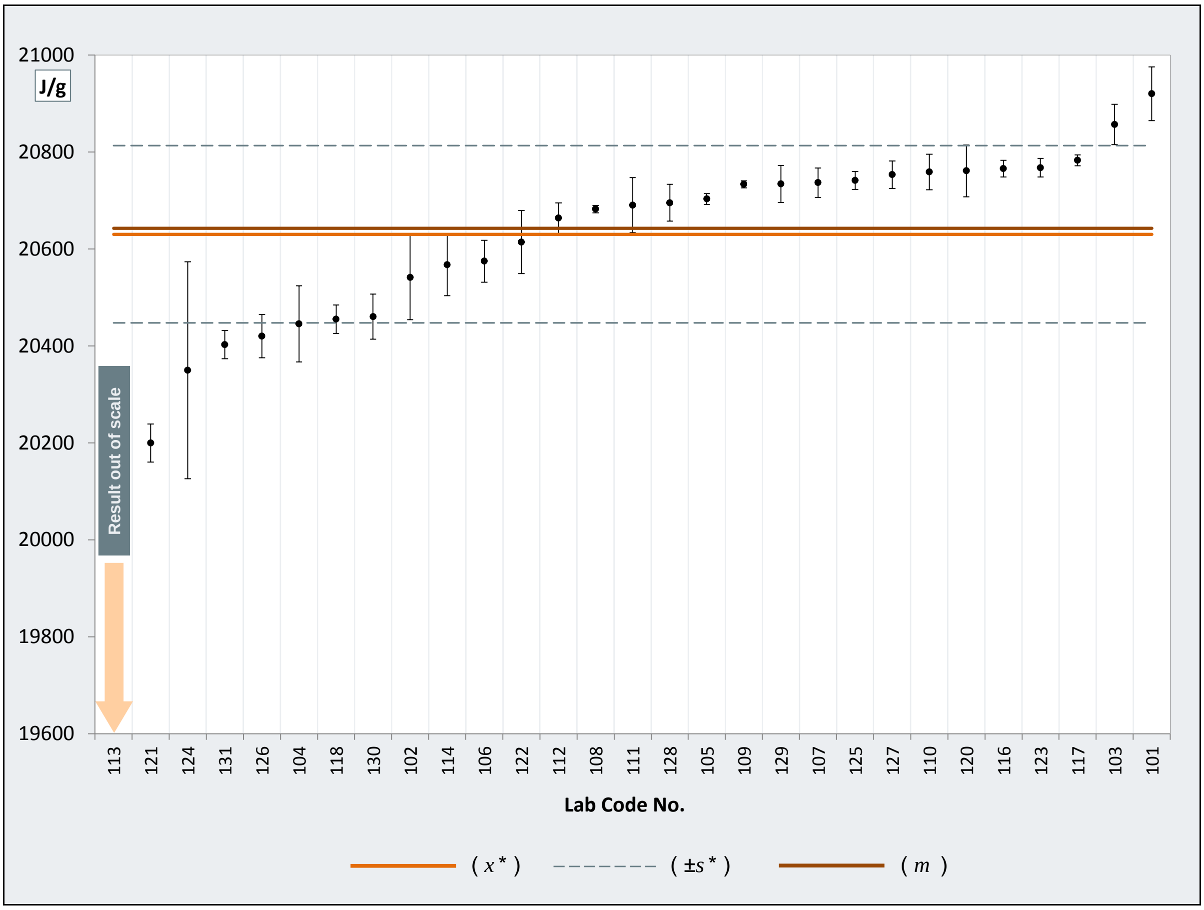
Robust average: x^* = **20630,2**	assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: s^* = **182,95**		
Number of repeate measurements necessary due to s_r/s^*-ratio: n' = 1	OK	see page 4 for the meaning of **NOT OK**
Standard uncertainty of the assigned value: u_x = 42,46524	OK	

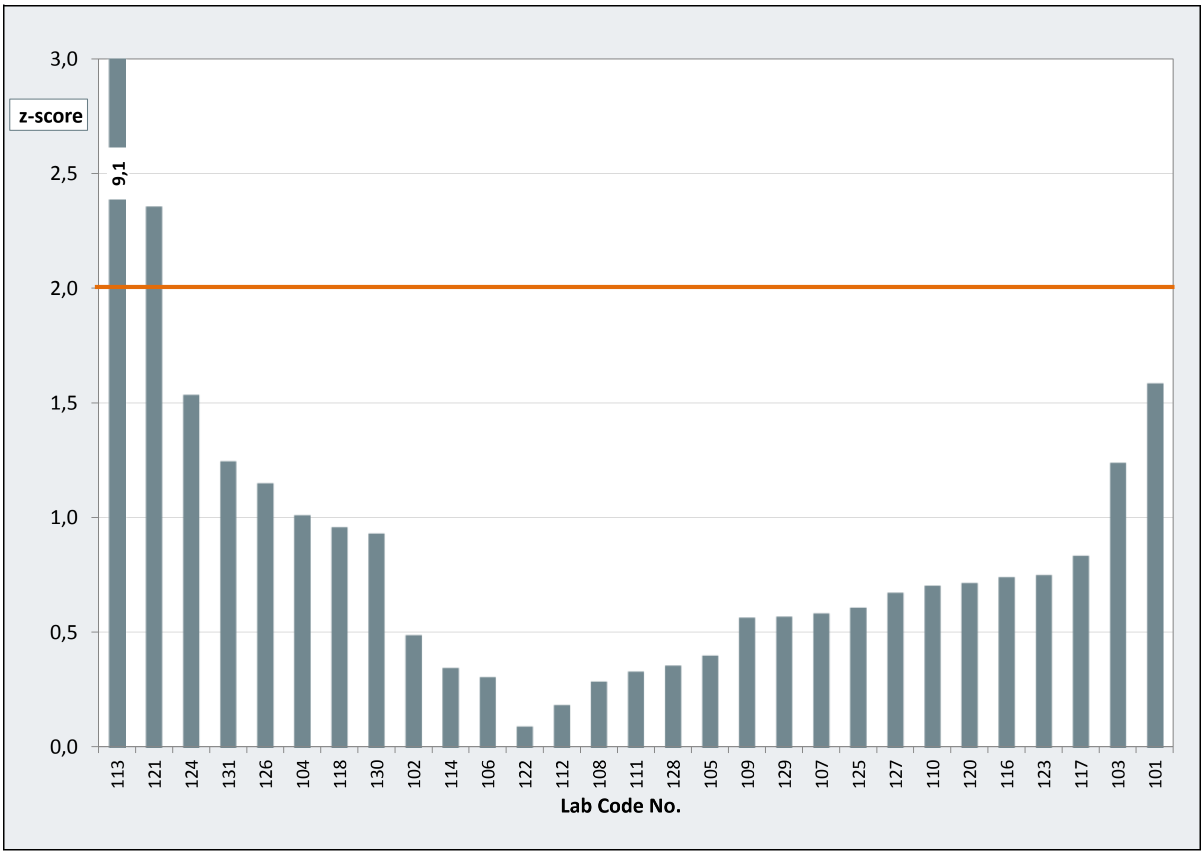
Additional check of the test method accuracy

Do the input data come from a normal distribution ? (The results listed below shall be considered as really justified only if the input data come from a normal distribution)		**NO**	
General mean $\Sigma n_i x_{ik} / \Sigma n_i$	m	20642,5	J/g
Repeatability variance	s_r^2	1873,4272917	
Repeatability standard deviation	s_r	43,28311	J/g
Repeatability coefficient of variation	$CV\%_r$	0,210	%
Between-laboratory variance	s_L^2	23917,8007687	
Between-laboratory standard deviation	s_L	154,65381	J/g
Between-laboratory coefficient of variation	$CV\%_L$	0,749	%
Reproducibility variance s_R^2	$s_r^2 + s_L^2$	25791,2280604	
Reproducibility standard deviation	s_R	160,5965	J/g
Reproducibility coefficient of variation	$CV\%_R$	0,778	%
Repeatability limit	r	**121,2**	J/g
Relative repeatability limit	r_{rel}	**0,59**	%
Reproducibility limit	R	**449,7**	J/g
Relative reproducibility limit	R_{rel}	**2,18**	%
Number of participants included in the accuracy evaluation	p	27	
Number of tests included in the accuracy evaluation	Σn	107	

Performance of individual laboratories in the specific test or measurment

Gross calorific value - q_{gross} - torrefied pellets





RESULTS

SECTOR Round Robin II -

Torrefied Pellets

ASH MELTING BEHAVIOUR

according to CEN/TS 15370

Remarks

Lab. No.:	Remarks
107	Deformation temperature not clearly to evaluate
109	Samples was ashed at 550°C. Four test pieces were tested up to 1450°C, but distinctive shapes were not identified as described in the standard.
110	Over 1500 degrees Celsius for all four different phases and samples
123	Not defined in CEN/TS 15370-1 - and this old ISO 540 term is meaningless regarding a cylinder with a diameter equal to the height (the only shape of the test piece allowed by CEN/TS 15370-1)!
127	Divided on a rotation divider and grinded to 1 mm. Cannot measure SST and ST.

Remarks OFI-RR-Team

All results which were above the detection limit from a laboratory were excluded from the evaluation.

The hemisphere and the flow temperature are above the detection limit from the most laboratories, so a statistical evaluation was not possible.

Ash melting behaviour

SST **Torrefield pellets**

Start shrinking temperature

CEN/TS 15370

Results submitted by participants

i.e., individual results x_{ik}
+ number of the test repetitions made by each lab (n_i)
+ within laboratory means (x_i) and standard deviations (s_i)
+ results of tests for outliers

Number of reporting laboratories p*: 12
Number of reported test results Σn_i: 42

Lab Code No.	Test results in °C — Test replication No. (k) 1	2	3	4	5	6	Statistical evaluation of the submitted test results x_{ik} — n_i	x_i	s_i	Outliers — Cochran	Grubbs	z > 2
107	670,0	660,0	670,0	680,0			4	670,00	8,1650			
123	700,0	690,0	680,0				3	690,00	10,0000			
122	715,0	725,0	710,0	720,0	720,0		5	718,00	5,7009			
128	726,0	727,0					2	726,50	0,7071			
106	733,0	733,0	745,0	751,0	748,0		5	742,00	8,4853			
101	860,0	860,0	860,0				3	860,00	0,0000			
103	1040,0	1060,0	1050,0				3	1050,00	10,0000			
111	1160,0	1170,0	1180,0	1180,0			4	1172,50	9,5743			
130	1247,0	1229,0	1238,0				3	1238,00	9,0000			
131	1264,0	1298,0	1299,0	1312,0			4	1293,25	20,5163	*		
~~104~~	~~1305,0~~	~~1382,0~~	~~1349,0~~				~~3~~	~~1345,33~~	~~38,6307~~	**		
121	1352,0	1351,0	1371,0				3	1358,00	11,2694			
109			no results reported									X
110			no results reported									X
127			no results reported									X

** ... statistical outlier (99%) * ... straggler (95%)
X ... $z > 2$

Results of robust statistics

Convergence assumed at iteration number: 2

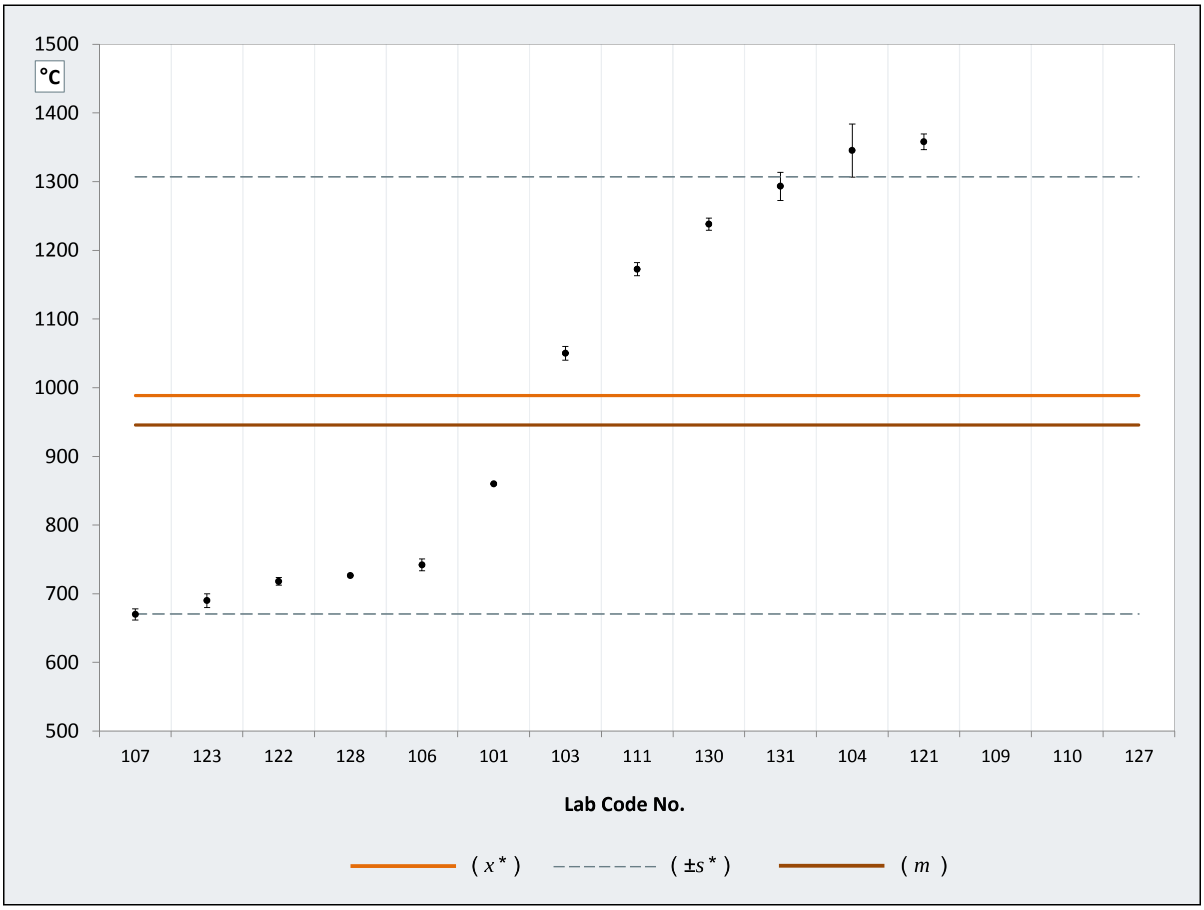
Robust average: x^* = **988,6**	assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: s^* = **318,23**		
Number of repeate measurements necessary due to s_r/s^*-ratio: n' = 1	**OK**	see page 4 for the meaning of **NOT OK**
Standard uncertainty of the assigned value: u_x = 114,83091	**NOT OK**	

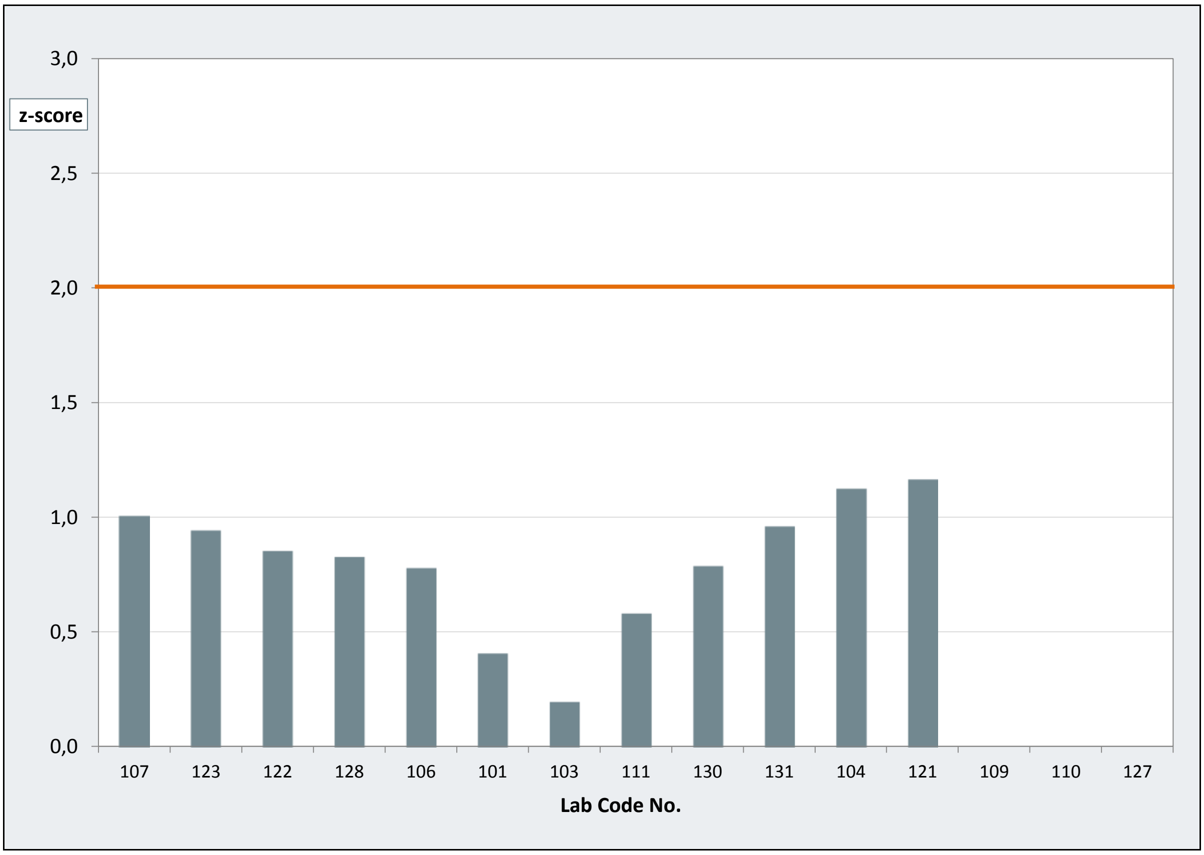
Additional check of the test method accuracy

Do the input data come from a normal distribution ? (The results listed below shall be considered as really justified only if the input data come from a normal distribution)			**NO**
General mean $\Sigma n_i x_{ik} / \Sigma n_i$	m	**945,7**	°C
Repeatability variance	s_r^2	106,1517857	
Repeatability standard deviation	s_r	10,30300	°C
Repeatability coefficient of variation	$CV\%_r$	1,089	%
Between-laboratory variance	s_L^2	72998,9076081	
Between-laboratory standard deviation	s_L	270,18310	°C
Between-laboratory coefficient of variation	$CV\%_L$	28,568	%
Reproducibility variance s_R^2	$s_r^2 + s_L^2$	73105,0593938	
Reproducibility standard deviation	s_R	270,3795	°C
Reproducibility coefficient of variation	$CV\%_R$	28,589	%
Repeatability limit	r	**28,8**	°C
Relative repeatability limit	r_{rel}	**3,05**	%
Reproducibility limit	R	**757,1**	°C
Relative reproducibility limit	R_{rel}	**80,05**	%
Number of participants included in the accuracy evaluation	p	11	
Number of tests included in the accuracy evaluation	Σn	39	

Performance of individual laboratories in the specific test or measurment

Start shrinking temperature - *SST* - Torrefield pellets





Ash melting behaviour

Deformation temperature

DT

Torrefield pellets

CEN/TS 15370

Results submitted by participants

i.e., individual results x_{ik}
+ number of the test repetitions made by each lab (n_i)
+ within laboratory means (x_i) and standard deviations (s_i)
+ results of tests for outliers

Number of reporting laboratories p *: **11**
Number of reported test results Σn_i: **39**

Lab Code No.	Test results in °C – Test replication No. (k) 1	2	3	4	5	6	n_i	x_i	s_i	Cochran	Grubbs	z > 2
106	880,0	865,0	889,0	884,0	902,0		5	884,00	13,4722			X
130	1205,0	1178,0	1186,0				3	1189,67	13,8684			
131	1257,0	1258,0	1240,0	1246,0			4	1250,25	8,7321			
~~107~~	~~1370,0~~	~~1190,0~~	~~1160,0~~	~~1350,0~~			4	~~1267,50~~	~~107,8193~~	**		
121	1283,0	1264,0	1270,0				3	1272,33	9,7125			
128	1274,0	1301,0					2	1287,50	19,0919			
122	1320,0	1295,0	1290,0	1315,0	1320,0		5	1308,00	14,4049			
103	1360,0	1380,0	1380,0				3	1373,33	11,5470			
111	1500,0	1500,0	1495,0	1490,0			4	1496,25	4,7871			
104	1526,0	1527,0	1526,0				3	1526,33	0,5774			
101	1540,0	1530,0	1550,0				3	1540,00	10,0000			
123			no results reported									X
110			no results reported									X
127			no results reported									X
109			no results reported									X

** ... statistical outlier (99%)   * ... straggler (95%)
X ... z > 2

Results of robust statistics

Convergence assumed at iteration number: 12

Robust average: x^* = **1326,5**	assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: s^* = **164,40**		
Number of repeate measurements necessary due to s_r/s^*-ratio: n' = 1	**OK**	see page 4 for the meaning of **NOT OK**
Standard uncertainty of the assigned value: u_x = 61,96032	**NOT OK**	

see page 4 for the meaning of **NOT OK**

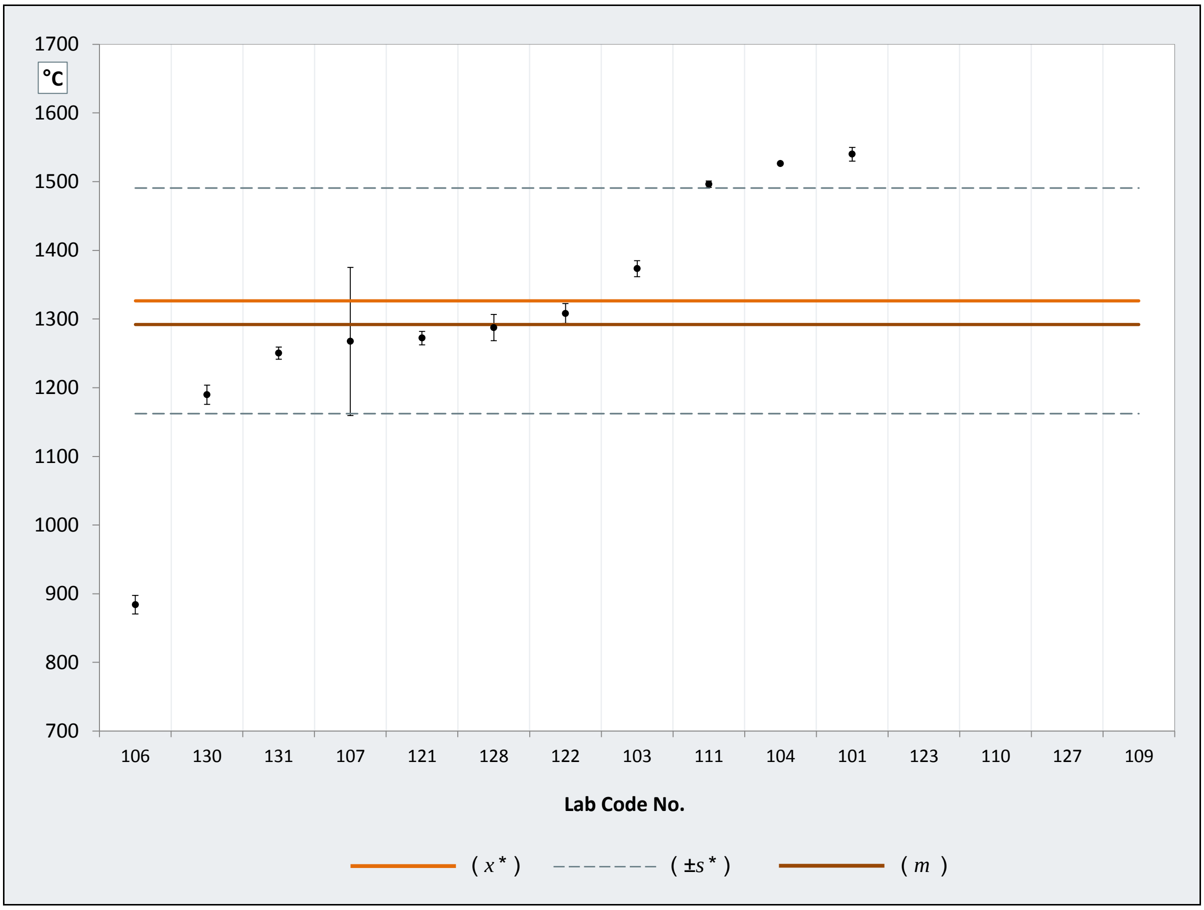
Additional check of the test method accuracy

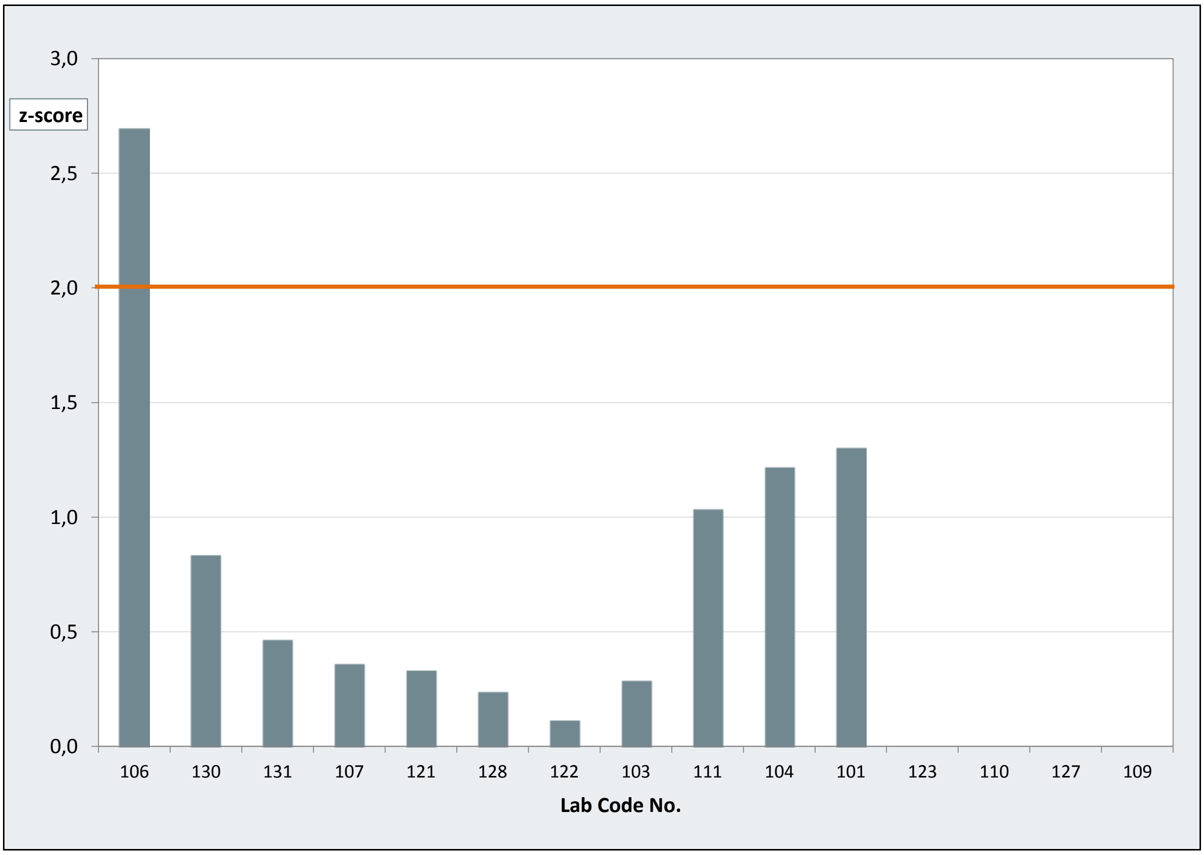
Do the input data come from a normal distribution ? (The results listed below shall be considered as really justified only if the input data come from a normal distribution) — **NO**

General mean $\Sigma n_i x_{ik} / \Sigma n_i$	m	**1292,2**	°C
Repeatability variance	s_r^2	130,3466667	
Repeatability standard deviation	s_r	11,41695	°C
Repeatability coefficient of variation	$CV\%_r$	0,884	%
Between-laboratory variance	s_L^2	45045,1018891	
Between-laboratory standard deviation	s_L	212,23831	°C
Between-laboratory coefficient of variation	$CV\%_L$	16,425	%
Reproducibility variance s_R^2	$s_r^2 + s_L^2$	45175,4485558	
Reproducibility standard deviation	s_R	212,5452	°C
Reproducibility coefficient of variation	$CV\%_R$	16,449	%
Repeatability limit	r	**32,0**	°C
Relative repeatability limit	r_{rel}	**2,47**	%
Reproducibility limit	R	**595,1**	°C
Relative reproducibility limit	R_{rel}	**46,06**	%
Number of participants included in the accuracy evaluation	p	10	
Number of tests included in the accuracy evaluation	Σn	35	

Performance of individual laboratories in the specific test or measurment

Deformation temperature - *DT* - Torrefield pellets





RESULTS

SECTOR Round Robin II -

Torrefied Pellets

DIAMETER AND LENGTH

according to ISO/DIS 17829 or EN 16127

Remarks

Lab. No.:	Remarks
104	Standard deviation (%) is expressed as %RSD.
106	For the length determination 100 pellets each have been used. Amount of sample corresponds with the length determination. For the diameter measurement 20 single pellets have been used-
112	Standard uncertainty of caliper = 0,06 mm
124	Für die Pellet-Länge ist die relative Standardabweichung (%) angegeben. Die Standardabweichung in mm beträgt 6,19 mm bzw. 6,22 mm. Für den Pellet-Durchmesser ist die relative Standardabweichung (%) angegeben. Die Standardabweichung in mm beträgt 0,05 mm für beide Wiederholungen.

Additional information

Lab. No.:	Amount of sample (g)			Method used	
				ISO/DIS 17829	EN 16127
101	98,7	98,7	75		X
102	51,41	53,41	56,11	X	
103	168,2	1153,1			X
104	50	50			X
105	124,68	110,29	144,77		X
106	68,9	70,8	58,5		X
108	134,04	133,89	136,26		X
109	100,5	100,8			X
110	116,43	123,89			X
111	66,55	-			X
112	109,99	110,77	111,19		X
113	87,0	81,2			X
115	51,3	54,0		X	
117	m.d	m.d	m.d		X
118	101,4	100,2	100,7		X
121	98,24	99,47			X
123	45,8	43,3		X	
124	100	-		X	
126	122,48	-		-	-
127	126,04	135,82	144,79		X
128	-	-	-		X
129	80	80			X
130	99,25	101,12		X	
131	115	112,5	112		X

Diameter and length — *length* — Torrefield pellets

ISO/DIS 17829 or EN 16127

Results submitted by participants

i.e., individual results x_{ik}
+ number of the test repetitions made by each lab (n_i)
+ within laboratory means (x_i) and standard deviations (s_i)
+ results of tests for outliers

Number of reporting laboratories p *: 24
Number of reported test results Σn_i : 58

Lab Code No.	Test results in mm — Test replication No. (k) 1	2	3	4	5	6	n_i	x_i	s_i	Outliers: Cochran	Grubbs	z > 2
103	11,7	11,8					2	11,75	0,0707			
131	10,3	13,1	12,4				3	11,91	1,4388			
121	11,9	12,2					2	12,05	0,2079			
127	12,0	11,5	12,7				3	12,06	0,5962			
112	12,8	12,2	11,6				3	12,20	0,6107			
102	10,4	13,1	13,2				3	12,25	1,5698			
113	12,0	12,5					2	12,26	0,3889			
115	13,8	10,8					2	12,30	2,1213			
105	12,0	12,3	12,7				3	12,33	0,3676			
110	12,5	12,6					2	12,55	0,1061			
106	13,1	13,5	11,4				3	12,67	1,1495			
111	13,0	13,0					2	12,98	0,0283			
109	13,6	12,5					2	13,06	0,7495			
108	13,1	13,0	14,2				3	13,40	0,6643			
130	13,4	13,7					2	13,55	0,2758			
123	13,9	13,6					2	13,76	0,2404			
124	14,2	14,1					2	14,14	0,1061			
101	13,8	14,6	14,3				3	14,23	0,4041			
128	14,8	15,6					2	15,22	0,5798			
129	15,0	15,9					2	15,46	0,5869			
~~118~~	~~20,8~~	~~16,7~~	~~16,3~~				~~3~~	~~17,92~~	~~2,4859~~		**	X
~~104~~	~~18,8~~	~~18,8~~					~~2~~	~~18,81~~	~~0,0071~~		**	X
~~126~~	~~20,6~~	~~20,6~~					~~2~~	~~20,60~~	~~0,0071~~		**	X
~~117~~	~~22,5~~	~~22,1~~	~~21,3~~				~~3~~	~~21,97~~	~~0,6110~~		**	X

** ... statistical outlier (99%) * ... straggler (95%)
X ... z > 2

Results of robust statistics

Convergence assumed at iteration number: 17

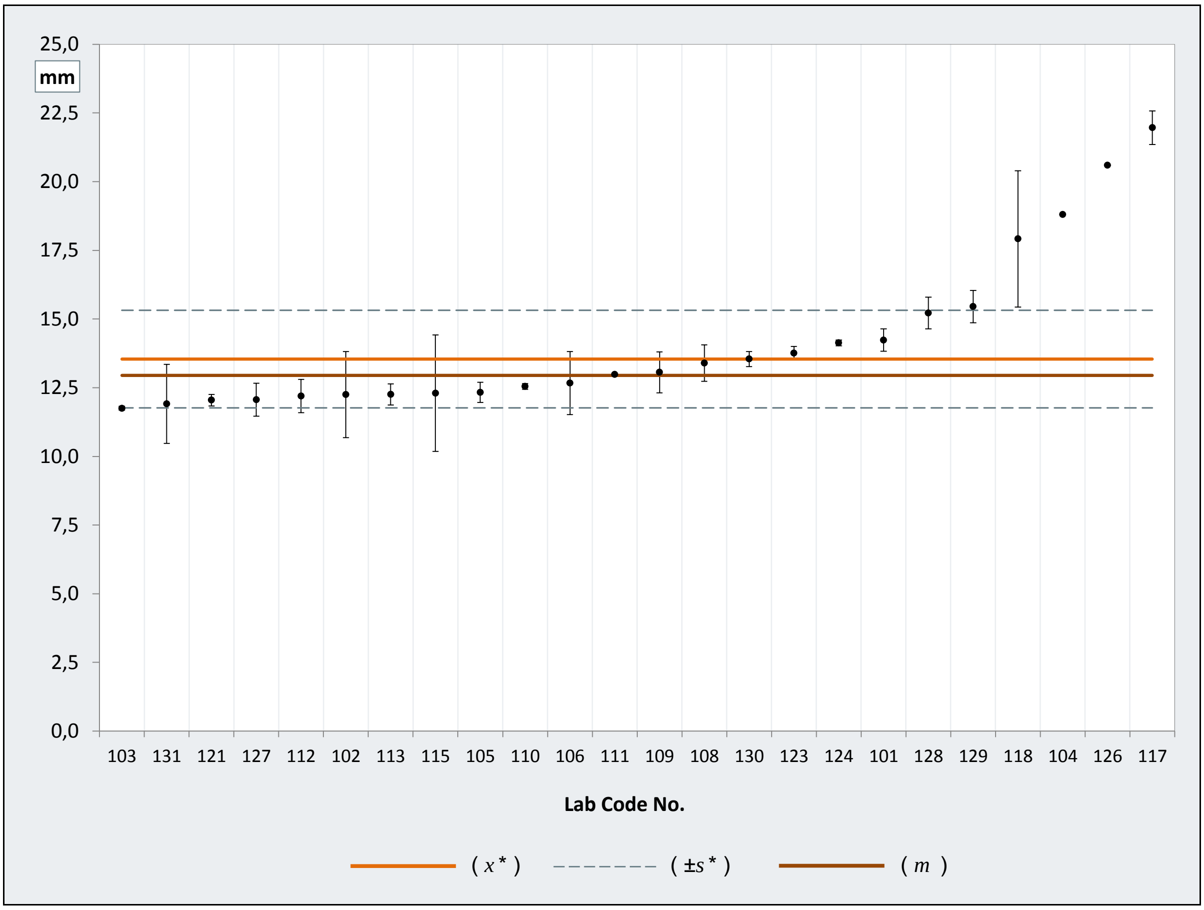
Robust average: x^* =	**13,5**	assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: s^* =	**1,78**		
Number of repeate measurements necessary due to s_r/s^*-ratio: n' =	3	**NOT OK**	see page 4 for the meaning of **NOT OK**
Standard uncertainty of the assigned value: u_x =	0,45301	**OK**	

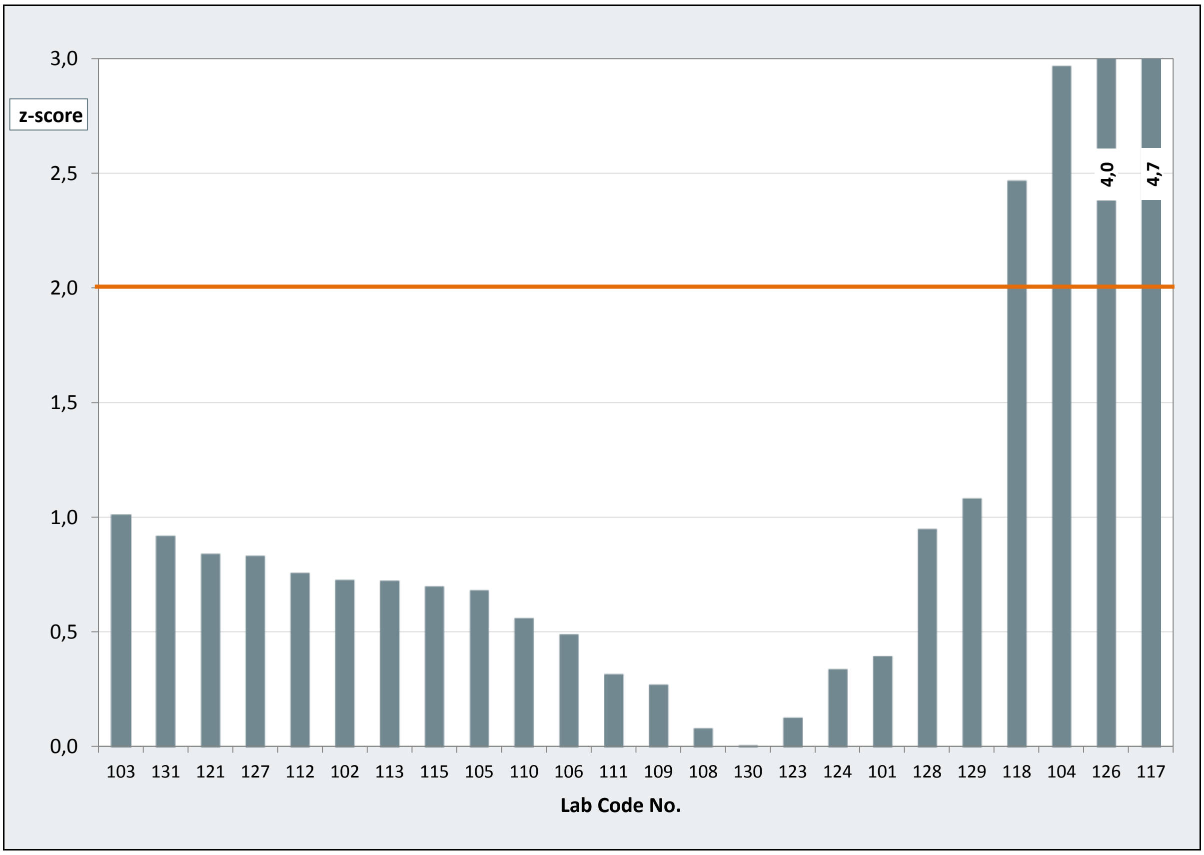
Additional check of the test method accuracy

Do the input data come from a normal distribution ? (The results listed below shall be considered as really justified only if the input data come from a normal distribution)		**YES**	
General mean $\Sigma n_i x_{ik} / \Sigma n_i$	m	12,9	mm
Repeatability variance	s_r^2	0,7409477	
Repeatability standard deviation	s_r	0,86078	mm
Repeatability coefficient of variation	$CV\%_r$	6,651	%
Between-laboratory variance	s_L^2	0,7906911	
Between-laboratory standard deviation	s_L	0,88921	mm
Between-laboratory coefficient of variation	$CV\%_L$	6,870	%
Reproducibility variance s_R^2	$s_r^2 + s_L^2$	1,5316387	
Reproducibility standard deviation	s_R	1,2376	mm
Reproducibility coefficient of variation	$CV\%_R$	9,562	%
Repeatability limit	r	**2,4**	mm
Relative repeatability limit	r_{rel}	**18,62**	%
Reproducibility limit	R	**3,5**	mm
Relative reproducibility limit	R_{rel}	**26,77**	%
Number of participants included in the accuracy evaluation	p	20	
Number of tests included in the accuracy evaluation	Σn	48	

Performance of individual laboratories in the specific test or measurment

Diameter and length - length - Torrefield pellets





Diameter and length

diameter **Torrefield pellets**

ISO/DIS 17829 or EN 16127

Results submitted by participants

i.e., individual results x_{ik}
+ number of the test repetitions made by each lab (n_i)
+ within laboratory means (x_i) and standard deviations (s_i)
+ results of tests for outliers

Number of reporting laboratories p *: 24
Number of reported test results Σn_i: 58

Lab Code No.	Test results in mm – Test replication No. (k) 1	2	3	4	5	6	Statistical evaluation of the submitted test results x_{ik}: n_i	x_i	s_i	Outliers: Cochran	Grubbs	$z>2$
101	7,9	7,9	7,9				3	7,90	0,0000		*	X
106	7,9	7,9	7,9				3	7,92	0,0100			X
104	8,0	8,0					2	7,99	0,0000			
103	8,0	8,0					2	8,00	0,0000			
113	8,0	8,0					2	8,01	0,0424			
102	8,0	8,0	8,0				3	8,02	0,0153			
111	8,0	8,0					2	8,03	0,0141			
131	8,1	8,1	8,0				3	8,08	0,0458			
130	8,1	8,1					2	8,09	0,0212			
105	8,1	8,1	8,1				3	8,09	0,0058			
123	8,1	8,1					2	8,10	0,0071			
118	8,1	8,1	8,1				3	8,10	0,0058			
121	8,1	8,1					2	8,11	0,0134			
108	8,1	8,1	8,1				3	8,11	0,0100			
112	8,1	8,1	8,1				3	8,11	0,0208			
110	8,1	8,1					2	8,12	0,0212			
124	8,1	8,1					2	8,12	0,0354			
128	8,1	8,1					2	8,12	0,0000			
126	8,1	8,1					2	8,12	0,0141			
127	8,2	8,2	8,1				3	8,14	0,0436			
115	8,1	8,2					2	8,15	0,0707			
109	8,2	8,2					2	8,16	0,0071			
117	8,2	8,2	8,2				3	8,16	0,0012			
129	8,1	8,2					2	8,18	0,0849			

** ... statistical outlier (99%) * ... straggler (95%)
X ... $z > 2$

Results of robust statistics

Convergence assumed at iteration number: 6

Robust average: x^* =	8,1	assigned value for the proficiency assessment	
Robust standard deviation for the proficiency assessment: s^* =	0,07		
Number of repeate measurements necessary due to s_r/s^*-ratio: n' =	2	OK	see page 4 for the meaning of **NOT OK**
Standard uncertainty of the assigned value: u_x =	0,01736	OK	

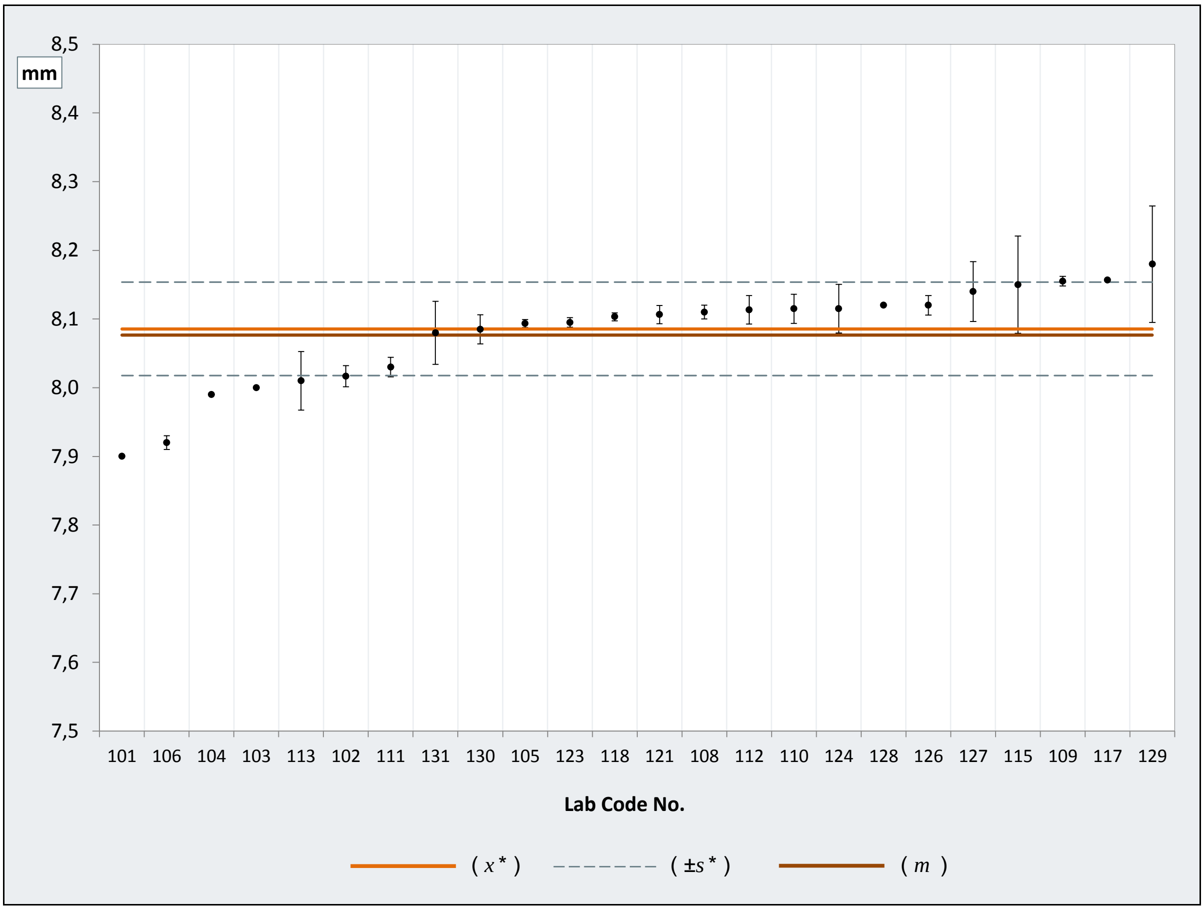
Additional check of the test method accuracy

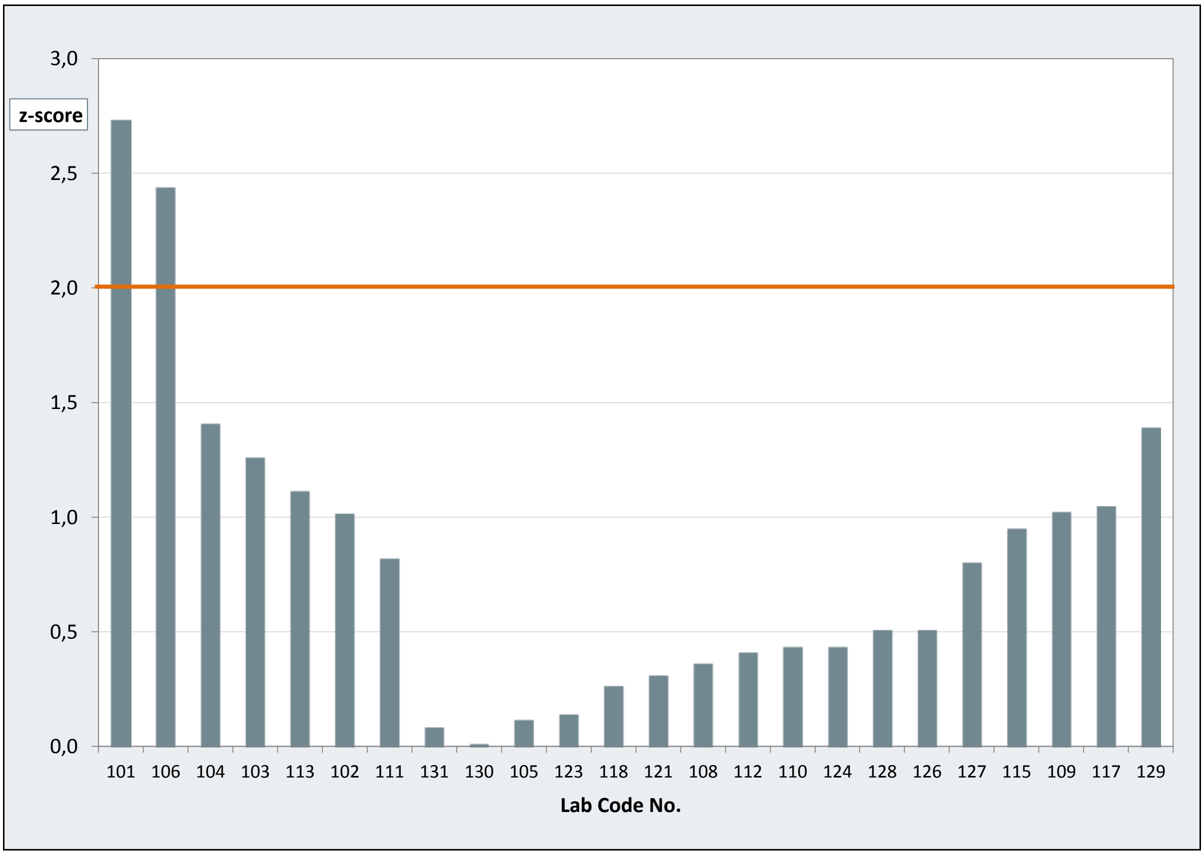
Do the input data come from a normal distribution ? — NO
(The results listed below shall be considered as really justified only if the input data come from a normal distribution)

General mean $\Sigma n_i x_{ik} / \Sigma n_i$	m	8,1	mm
Repeatability variance	s_r^2	0,0007853	
Repeatability standard deviation	s_r	0,02802	mm
Repeatability coefficient of variation	$CV\%_r$	0,347	%
Between-laboratory variance	s_L^2	0,0054430	
Between-laboratory standard deviation	s_L	0,07378	mm
Between-laboratory coefficient of variation	$CV\%_L$	0,913	%
Reproducibility variance s_R^2	$s_r^2+s_L^2$	0,0062282	
Reproducibility standard deviation	s_R	0,0789	mm
Reproducibility coefficient of variation	$CV\%_R$	0,977	%
Repeatability limit	r	**0,1**	mm
Relative repeatability limit	r_{rel}	**0,97**	%
Reproducibility limit	R	**0,2**	mm
Relative reproducibility limit	R_{rel}	**2,74**	%
Number of participants included in the accuracy evaluation	p	24	
Number of tests included in the accuracy evaluation	Σn	58	

Performance of individual laboratories in the specific test or measurment

Diameter and length - diameter - Torrefield pellets





5 Summary and Conclusions

In the following chapter the most important results of the statistical evaluations will be presented. If there are data available the results will be compared with the results of the SECTOR Round Robin 1. However, only the relative repeatability and the relative reproducibility limit (in per cent) are comparable. The repeatability limit and the reproducibility limit are given as absolute number and relative in reference to the general mean.

5.1 New methods

5.1.1 Grinding energy

On the first view the results for the “Grinding energy” looks very well, there is no Z-score above 2 and also no outliers. However the results from the individual participants vary in a very broad range, between 4,9 and 30,7 Wh/kg. This broad range and the slow increase of the values, is the reason that no Z-score is above 2. These huge variations results in a very high reproducibility limit of 123,48%. The repeatability limit of 11,06% is acceptable for a method which has never been tested before in a round robin. This indicates that every participant handle the method on every test on the same or nearly the same way. However an adjustment is still missing.

With a view to the remarks of the participants the main problem for the high reproducibility limit is easily to identify. Nearly every laboratory used different cutting mills and cannot reach the required parameter (e.g. feeding rate), also different metering units were used (per hand, gravity, etc.). Some participants have the problem to exactly determine the starting and ending point of the measurement. Also some other factors could influence the results, for example how sharp-edged the blades of the cutting mill are.

The method description was written very exactly and is rich in details. On the one hand this is very important to ensure a high quality of the method and good results in comparative tests. On the other hand for laboratories it is more difficult to participate in such test and exactly meet the method. The OFI as the organizer of the round robin, decided to allow some deviations from the method description, otherwise the number of participants would have been too small to allow an accurate statistical analysis. Also the remarks from participating laboratories are very important and useful for the further development of the method. So this reproducibility limit is to handle with care.

Table 5: performance data "Grinding energy"

Grinding Energy		SECTOR RR 2	
Robust average	x*	16,4	Wh/kg
Robust standard deviation	s*	8,25	Wh/kg
General mean	m	16,7	Wh/kg
Repeatability limit	r	1,8	Wh/kg
		11,06	%
Reproducibility limit	R	20,6	Wh/kg
		123,48	%
Number of participants	p	11	-
Number of tests	Σn	27	-

5.1.2 Water absorption

The method "Water absorption" was split up into two parts. First the immersion test itself and second a conventional mechanical durability test. Both tests were separately evaluated in the round robin test. Similar to the "Grinding energy" the repeatability limit of the immersion test is, for a new method, acceptable. The results are comparable within the laboratory. On the high reproducibility limit of 35,76% it is obvious, that the method description is not exactly enough formulated. Used equipment has to be defined more accurate and also the procedure itself could be formulated to open. Very important points are the definition of the hole size of the sieve, the temperature for the immersion water and it is also not exactly defined how to consider residues in the soaking pan.

The big advantage that the method description was not written too constricted is that a lot of laboratories can participate in this method and give valuable response.

Table 6: performance data part 1 – "Water absorption"

Water absorption		**SECTOR RR 2**	
Robust average	x*	37,17	%
Robust standard deviation	s*	4,875	%
General mean	m	36,77	%
Repeatability limit	r	2,57	%
		6,990	%
Reproducibility limit	R	13,15	%
		35,760	%
Number of participants	p	23	-
Number of tests	Σn	102	-

The repeatability and the reproducibility limit of the mechanical durability test as conventional test method are very high. With a look on the additional information – "original mechanical durability", it is obvious that the method in general works. Also here, a better defined method description would improve the results. An improvement could be to define the moisture content after drying / before mechanical durability tests. If the pellets are very dry (after drying), the mechanical durability is much worse than after stabilisation on ambient atmosphere.

A comparison of the results from the mechanical durability and the "Water absorption" (immersion) test shows that laboratories with a high loss of mechanical durability typically have also a high water uptake and laboratories with a low loss of mechanical durability usually have a low water uptake. One the one hand, this demonstrate the connection of these two test methods, and on the other hand it shows that, if the immersion test will be improved also the results of the mechanical durability test will be better.

Table 7: performance data part 2 – "Loss of mechanical durability"

Loss of mechanical durability		SECTOR RR 2	
Robust average	x*	13,39	%
Robust standard deviation	s*	2,342	%
General mean	m	13,44	%
Repeatability limit	r	2,99	%
		22,268	%
Reproducibility limit	R	8,67	%
		64,495	%
Number of participants	p	21	-
Number of tests	Σn	92	-

5.2 Standard methods

5.2.1 Carbon content

The results of the "Carbon content" show a normal distribution, like in SECTOR Round Robin 1. The repeatability limit and the reproducibility limit are a marginal higher in SECTOR Round Robin 2, which might be related with the raw material (forest residues) or some inhomogeneity in the torrefied material. However, the method works for conventional wood pellets and for torrefied pellets as well.

Table 8: performance data "Carbon content"

Carbon content		SECTOR RR 1	SECTOR RR 2	
Robust average	x*		51,77	w-%
Robust standard deviation	s*		1,313	w-%
General mean	m	53	51,47	w-%
Repeatability limit	r	0,43	0,55	w-%
		0,81	1,065	%
Reproducibility limit	R	1,93	4,56	w-%
		3,64	8,859	%
Number of participants	p	32	24	-
Number of tests	Σn	125	102	-

5.2.2 Gross calorific value

The repeatability limit and the reproducibility limit of the EN 14918 are both not met in SECTOR Round Robin 2. The calorific value is extremely sensitive to inhomogeneous torrefaction, and some participants noted that there are some inhomogeneities in the material. However, it is also reasonable to suppose that the method works for torrefied materials.

Table 9: performance data "Gross calorific value"

Gross calorific value		**SECTOR RR 1**	**SECTOR RR 2**	
Robust average	x*		20630,2	J/g
Robust standard deviation	s*		182,95	J/g
General mean	m	19606	20642,5	J/g
Repeatability limit	r	103	121,2	J/g
		0,53	0,59	%
Reproducibility limit	R	484	449,7	J/g
		2,47	2,18	%
Number of participants	p	31	27	-
Number of tests	Σn		107	-

5.2.3 Ash melting behaviour

The most important characteristic of the "Ash melting behaviour" is the deformation temperature. The results show an acceptable repeatability limit and a very high reproducibility limit. This corresponds to the experience made by other round robin tests and analyses. The evaluation of the results of the ash melting behaviour is very subjective, which leads to a low repeatability and a high reproducibility limit.

The hemisphere and the flow temperature are above the detection limit from the most laboratories, so a statistical evaluation was not possible.

Table 10: performance data "Ash melting behaviour" (deformation temperature)

Ash melting behaviour (DT)		SECTOR RR 1	SECTOR RR 2	
Robust average	x*		1326,5	°C
Robust standard deviation	s*		164,40	°C
General mean	m	1394	1292,2	°C
Repeatability limit	r	32	32,0	°C
		2,3	2,47	%
Reproducibility limit	R	293	595,1	°C
		21,0	46,06	%
Number of participants	p	14	10	-
Number of tests	Σn		35	-

5.2.4 Diameter and length

In SECTOR Round Robin 1 the method "Diameter and length" was not tested, so there are no data available to compare the results. The performance data for the determination of the length has to be interpreted with caution. Despite the greatest care, it cannot ensured that some pellets were broken during the transportation. The results show mainly deviations above the mean value and only less below. For the test "Diameter and length" two methods were offered, EN 16127 and ISO/DIS 17829. Most participants chose EN standard and only five the ISO/DIS, so a separate statistical evaluation was not possible.

Table 11: performance data "Diameter and length" (length)

Diameter and length (length)		SECTOR RR 2	
Robust average	x*	13,5	mm
Robust standard deviation	s*	1,78	mm
General mean	m	12,9	mm
Repeatability limit	r	2,4	mm
		18,62	%
Reproducibility limit	R	3,5	mm
		26,77	%
Number of participants	p	20	-
Number of tests	Σn	48	-

Table 12: performance data “Diameter and length” (diameter)

Diameter and length (diameter)		**SECTOR RR 2**	
Robust average	x*	8,1	mm
Robust standard deviation	s*	0,07	mm
General mean	m	8,1	mm
Repeatability limit	r	0,1	mm
		0,97	%
Reproducibility limit	R	0,2	mm
		2,74	%
Number of participants	p	24	-
Number of tests	Σn	58	-

5.3 General

In general it was noted by labs, that the sample was not perfectly eligible for a round robin test. Forest residues are not the best raw material to assure a homogeneous pellet and there were some inhomogenities in the torrefied material. On the one hand, it could be possible that the pellets include some not perfectly torrefied parts and on the other hand it could be happened that the torrefied material was wasted with some untorrefied material during the production. However some methods are extremely sensitive for inhomogeneous materials, for example the calorific value.

For the further development of the new methods, this round robin test was very important and useful. Especially the performance data of the “Water absorption” (immersion and mechanical durability) were for the first test good. With some modifications in the method description and more detail information in some fields the method can be used in practice.

The results of the grindability tests according to the proposed method seem to be reliable at first, however a further development of the method is advisable. Differences in equipment (e.g. mill, feeding system) between the participating laboratories were the largest source of variation. The round robin test was successful in revealing these deviations and was a valuable step towards adopting and perfecting this novel grindability method

6 List of figures

7 List of tables

8 References

- EN 15104 “Solid biofuels - Determination of total content of carbon, hydrogen and nitrogen - Instrumental methods”
- EN 14918 “Solid biofuels - Determination of calorific value”
- CEN/TS 15370 “Solid biofuels - Method for the determination of ash melting behaviour”
- ISO/DIS 17829 “Solid Biofuels - Determination of length and diameter of pellets”
- EN16127 “Solid biofuels - Determination of length and diameter of pellets”
- Method description “Grinding energy”
- Method description “Water absorption”

9 Appendix

- Method description “Grinding energy”
- Method description “Water absorption”

SECTOR Production of Solid Sustainable Energy Carriers from Biomass by Means of TORrefaction

METHOD DESCRIPTION

DETERMINATION OF GRINDING ENERGY

The method description explains how the grinding energy required during the milling process of torrefied biomass pellets is measured. Therefore a certain amount of pellets is grinded in a cutting mill while the power consumption is recorded. Additionally the calculation of the specific grinding energy is explained using the measured values.

If there are any questions to the method or the equipment please contact us.

DBFZ Deutsches Biomasseforschungszentrum
Contact person: **Andreas.Pilz@dbfz.de**

Equipment:

- Power measuring device:

The measuring device should have a measuring range from 1 W to 4 kW and the measuring period should be set to 1 s (e.g. the power quality clamp meter 345 from Fluke).

- Cutting mill with a collecting vessel:

The mill should have a capacity of around 60 l/h and a circumferential speed of approximately 20 m/s. The collecting vessel should have a capacity of minimum 5 l (e.g. cutting mill from Fritsch "Pulverisette 19" or Retsch "SM300" - revolution speed of 3000 rpm, rotor with V-cutting edges and fixed knives, 1 mm sieve with trapezoidal perforation).

- Metering unit

The metering unit is used for dosing the pellets with an uniform mass flow. The metering unit should be equipped with a frequency converter to adjust the dosing to the respective sizes of the pellets. For the analysis a mass flow of 15 – 20 g/s is required. The least possible stress has to be applied for the metering unit, which reduces the pre-comminution effect by e.g. abrasion.

- Laboratory equipment

Laboratory balance and collecting vessel, stop watch: The laboratory balance (measuring range: up to 5000 g with a readability of 0.1 g), the stopwatch and the collecting vessel (container volume of about 10 liters) are used for the determination of the dose rate and the grinding process.

Preparation Procedure

The mill used for testwork has to be firmly attached on the fixed base, checked for operability and the presence of any impurities. The mill is fitted with a 1 mm round-hole sieve.

The fines of the pellets used for the test have to be screened with a 3.15 mm round-hole sieve (according DIN EN 15149-1). Afterwards, a sample of 2.5 kg (with moisture as received) of the test material is poured into the feed hopper of the metering unit.

Before starting the fine grinding it is necessary to adjust the throughput of the metering unit. Therefore the screened pellets are placed into the feed hopper and a collecting vessel is placed in front of the metering device. Subsequently, the metering unit is turned on at a selected frequency and the pellets are fed into the collecting vessel for one minute. After turning off the metering unit, the delivered volume is weighed and the throughput is calculated. This process is repeated until the flow rate is adjusted to a range of 15 to 20 g/s.

The power measuring device has to be installed directly at the power supply line ahead the mill. It has to be guaranteed that neither the power of the metering unit nor any other forces are captured. The measured values have to be stored in the shortest possible interval (best: one value per second).

Measuring process

Initially, the idle power consumption of the cutting mill has to be recorded before starting the fine grinding of the torrefied pellet sample. Thus, the mill equipped only with a sieve and the power measuring device is turned on and the idle power is measured over a period of at least 120 s.

Afterwards, the metering unit which is set to a dosing rate of 15 to 20 g/s is turned on and thus, the grinding process is started. The starting time of the metering unit needs to be recorded to analyze the particular parts of the power measuring after the procedure.

If no more material is fed into the mill, the milling process is completed and the metering has to be stopped. Again this particular time has to be recorded. The mill has to be run continuously for at least another 120 s to obtain further data for measuring the idle power after the load. Afterwards the cutting mill is stopped and subsequently the power measuring device is also switched-off.

The milled material, which is collected in the collecting vessel, has to be weighed.

The determination is repeated two times and the results of the calculated specific grinding energy are averaged.

Interpretation and calculation

To determine the applied grinding energy for milling torrefied pellets, the stored output values have to be divided into three sections. The three sections are called as follows, see also figure 1:

- idle power before grinding $\mathbf{P_{I1}}$
- total active power $\mathbf{P_{total}}$
- idle power after grinding $\mathbf{P_{I2}}$

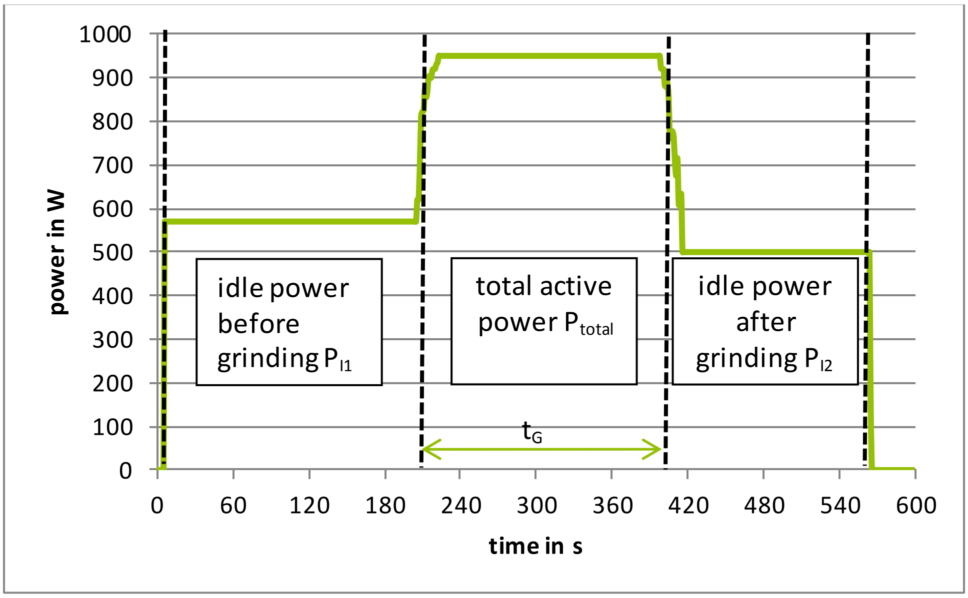


Figure 1: Typical curve of the power measurement including the three sections

The classification is according to the listed times during the experiment.

The idle power $\mathbf{P_{I1}}$ and $\mathbf{P_{I2}}$ (unit: W) are averaged and lead to the total idle power $\mathbf{P_I}$:

$$P_I = \frac{\overline{P_{I1}} + \overline{P_{I2}}}{2}$$

During the grinding process the average total active power $\mathbf{P_{total}}$ is recorded, which is the sum of the energy applied for the grinding $\mathbf{P_G}$ and the idle power of the mill $\mathbf{P_I}$. Thus, $\mathbf{P_G}$ can be determined by subtracting $\mathbf{P_I}$ from $\mathbf{P_{total}}$.

$$P_G = P_{total} - P_I$$

With this result the energy consumption during the grinding process $\mathbf{E_G}$ (unit: Wh) is determined:

$$E_G = P_G \cdot t_G$$

Thereby $\mathbf{t_G}$ is the duration of milling in hours.

This yields to the mass specific grinding energy $\mathbf{E_m}$ (unit: Wh/kg), which is specified with one decimal point:

$$E_m = \frac{E_G}{m_G}$$

METHOD DESCRIPTION

WATER ABSORPTION - IMMERSION TEST

Most biomass materials easily absorb moisture when they directly penetrated by water. This behaviour is especially problematical for storage and transport. Through the water absorption the net calorific value decline and the mass of the material increase. Also the mechanical durability changes, this affects particular compressed biomass (e.g. pellets).

The method description explains how to measure the absolute water absorption of torrefied pellets. Therefore a certain amount of pellets is immersed in water and the water absorption (WA) is measured. Furthermore the mechanical durability (D_U) is determined as well.

If there are any questions to the method or the equipment please contact us.

OFI - Österreichisches Forschungsinstitut für Chemie und Technik
Contact person: **Christoph.Goebl@ofi.at**

Normative references

- EN14774-2, Solid biofuels – Determination of moisture content – Oven dry method – Part 2: Total moisture – Simplified method
- EN15210-1, Solid biofuels – Determination of mechanical durability of pellets and briquettes, Part 1: Pellets

Required Items / Apparatus

- Round hole sieve: hole size of 3.15 mm
- Soaking pan: 3-4x of pellets volume
- Sieve: with sufficient separation of liquid and solid content

Absolute water absorption with mechanical durability

In order to determine the absolute absorbed water, the water content (EN14774-2) of the original sample (WC_{or}) must be known. As a reference data the original mechanical durability ($D_{U\ or}$) (EN15210-1, double determination-mean value) needs to be determined as well.

For the determination of the water absorption, 650g pellets are required. The pellets are sieved with the 3.15 mm round hole sieve.

From the sieved material 600 g are weighed in into the soaking pan (m_{wi}). This is filled up with sufficient water, which means 2-3x of the pellets volume. The water level should be at least 2 cm higher than the pellets. In order to avoid entrapped air and clumping of pellets repeated gentle stirring may be needed. After one hour the test portion is separated from the water by using a sieve appropriate for liquid/solid separation and let the sample drip free of water for 30 minutes.

After the dripping time the wet pellets are weighed out (m_{wa}) and weighed in to drying trays (e.g. aluminium trays) and dried to constant mass (max. 24 hours) at 105°C. From the obtained data the absolute water absorption (WA) can be calculated according to equation no. 1.

The dried pellets are left at ambient atmosphere in order to stabilize for at least 24 hours. Afterwards the water content (EN14774-2) and the mechanical durability ($D_{u\ at}$) (EN15210-1, single determination) is tested. The loss of mechanical durability ($D_{U\ loss}$) is determined according equation no. 2

Absolute water absorption without mechanical durability

In order to determine the absolute absorbed water, the water content (EN14774-2) of the original sample (WC_{or}) must be known.

For the determination of the water absorption, 650 g pellets are required. The pellets are sieved with the 3.15 mm round hole sieve.

From the sieved material 600 g are weighed in (m_{wi}) into the soaking pan. This is filled up with sufficient water, which means 2-3x of the pellets volume. The water level should be at least 2 cm higher than the pellets. In order to avoid entrapped air and clumping of pellets repeated gentle stirring may be needed. After one hour the test portion is separated from the water by using a sieve appropriate for liquid/solid separation and let the sample drip free of water for 30 minutes.

After the dripping time the wet pellets are weighed out (m_{wa}). From the obtained data the absolute water absorption can be calculated according to equation no. 1.

Calculations

All results have to quote at two decimal places.

Equation 1: Determination of the absolute absorbed water:

$$WA = \frac{(m_{wa} - m_{wi})}{m_{wi}} * 100 + WC_{or}$$

WA … Water absorption [%]

m_{wa} … mass of the pellets after dropping time [g]

m_{wi} … mass of the initial weight of the pellets [g]

WC_{or}…original water content [%]

Equation 2: Determination of the loss of mechanical durability:

$$D_{U_{loss}} = \frac{(D_{U_{or}} - D_{U_{at}})}{D_{U_{or}}} * 100$$

$D_{U\ loss}$ … Loss of mechanical durability [%]

$D_{U\ or}$ … mechanical durability of original sample [%]

$D_{u\ at}$ … mechanical durability after stabilisation at ambient atmosphere [%]