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Fast pyrolysis bio-oils for industrial boilers — Requirements and test methods

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National foreword

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Schnellpyrolyse-Bioöle für industrielle Kesselanlagen - Anforderungen und Prüfverfahren

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European foreword

This document (EN 16900:2017) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2017, and conflicting national standards shall be withdrawn at the latest by September 2017.

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This document has been prepared under a mandate [1] given to CEN by the European Commission and the European Free Trade Association.

Annex C contains the precision data generated on the test methods, which are the results of inter-laboratory testing, carried out by Working Group 41 of CEN/TC 19. Many of the test methods included in this standard were the subject of inter-laboratory testing to determine the applicability of the method and its precision. In Annex D also the needed modifications to the test methods are presented.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Fast pyrolysis bio-oils (FPBO) or fast pyrolysis liquids are completely different from petroleum fuels both in their physical properties and chemical composition. They are brownish liquids with a distinct and smoky odour. They can be produced from woody[2] biomass and agrobiomass (herbaceous[2]) and there is a wide range of reactor types are suitable for fast pyrolysis bio-oil production. Contrary to fossil fuels, they are highly polar, mainly water-soluble containing typically about 25 % (*m/m*) on wet basis) of water, acidic in nature, dense, and are viscous liquids, very poorly or not miscible with hydrocarbons [3, 6, 18, 19].

1 Scope

This European Standard specifies requirements and test methods for fast pyrolysis bio-oils for boiler use at industrial scale (>1 MW thermal capacity), not for domestic use. Two different grades are specified.

It is recommended to draw attention to differences especially in those properties, which can have an effect on the required flue gas treatment system, such as ash, nitrogen, and sulfur content. National and local regulations determine the requirements for flue gas treatment system.

In addition to the quality requirements and test methods for fast pyrolysis bio-oils, further instructions on storage (Annex A), sampling, and materials compatibility (Annex B) are given.

NOTE For the purposes of this European Standard, the term "% (*m/m*)" is used to represent respectively the mass fraction.

2 Normative references

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

EN 16476, *Liquid petroleum products - Determination of Sodium, Potassium, Calcium, Phosphorus, Copper and Zinc contents in diesel fuel - Method via Inductively Coupled Plasma Optical Emission Spectrometry (ICP OES)*

EN ISO 2719, *Determination of flash point - Pensky-Martens closed cup method (ISO 2719)*

EN ISO 3104, *Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity (ISO 3104)*

EN ISO 3170:2004, *Petroleum liquids - Manual sampling (ISO 3170:2004)*

EN ISO 4259, *Petroleum products - Determination and application of precision data in relation to methods of test (ISO 4259)*

EN ISO 6245, *Petroleum products - Determination of ash (ISO 6245)*

EN ISO 9038, *Determination of sustained combustibility of liquids (ISO 9038)*

EN ISO 12185, *Crude petroleum and petroleum products - Determination of density - Oscillating U-tube method (ISO 12185)*

EN ISO 20846, *Petroleum products - Determination of sulfur content of automotive fuels - Ultraviolet fluorescence method (ISO 20846)*

ISO 3016, *Petroleum products — Determination of pour point*

ASTM E70, *Standard Test Method for pH of Aqueous Solutions with the Glass Electrode*

ASTM E203, *Standard Test Method for Water Using Volumetric Karl Fischer Titration*

ASTM D5291, *Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants*

ASTM D7579, *Standard Test Method for Pyrolysis Solids Content in Pyrolysis Liquids by Filtration of Solids in Methanol*

DIN 51900-1, *Testing of solid and liquid fuels - Determination of gross calorific value by the bomb calorimeter and calculation of net calorific value – Part 1: General information*

DIN 51900-3, *Testing of solid and liquid fuels - Determination of gross calorific value by the bomb calorimeter and calculation of net calorific value - Part 3: Method using adiabatic jacket*

3 Terms and definitions

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

3.1
agrobiomass
biomass obtained from energy crops and/or agricultural by-products (agricultural residues)

[SOURCE: modified from FAO unified bioenergy terminology [UBET]

3.2
fast pyrolysis
thermal treatment of lignocellulosic biomass at short hot vapour residence time (typically less than about 5 s) typically at between 450 °C – 600 °C and at near atmospheric pressure or below, in the absence of oxygen, using small (typically less than 5 mm) dry (typically less than 10 % water) biomass particles

Note 1 to entry: Many fast pyrolysis processes are using fluidised or entrained bed reactor with sand as a heat carrier.

Note 2 to entry: Under REACH it is defined as "lignocellulosic biomass, at short hot vapour residence time (typically less than about 10 seconds) typically at between 450-600 C at near atmospheric pressure or below, in the absence of oxygen"

3.3
fast pyrolysis bio-oil
FPBO
liquid produced by fast pyrolysis from biomass

Note 1 to entry: The typical yield of bio-oil is 60 % (*m/m*) - 75 % (*m/m*) on wet basis (energy basis) and 55 % (*m/m*)– 65 % (*m/m*) of organic matter. Other products are char and non-condensable gases.

3.4
solids
solid particles which are not soluble in methanol-dichloromethane (1:1), possibly containing inorganic elements including sand, char, and additional insoluble organic material

Note 1 to entry: The solids will in time settle to the bottom or raise up to the surface depending on their density and the fast pyrolysis bio-oil composition.

3.5
stability
situation in which physico–chemical properties remain unchanged during handling and storage

Note 1 to entry: FPBOs are not chemically or thermally as stable as conventional petroleum fuels due to the high content of reactive oxygen containing compounds and low-boiling volatiles. The instability of FPBOs can be observed via an increase in viscosity ("ageing") and possible phase-separation by time and temperature. A stability test based on viscosity increase at 80 °C in 24 h may be used to predict if the bio-oil will stand for a year's storage at room temperature without phase-separation [3].

4 Sampling and sample handling

Samples shall be taken as described in EN ISO 3170:2004 and/or in accordance with the requirements of national standards or regulations for the sampling of fast pyrolysis bio-oil. The national requirements shall be set out in detail or shall be referred as a National Annex to this European Standard.

It is strongly advised to review all intended test methods prior to sampling to understand the importance of sampling technique, and special handling requirements.

There is some information in EN ISO 3170:2004 that is not relevant with fast pyrolysis bio-oils: fast pyrolysis bio-oil is mostly water-soluble (approximately 80 %) and hence does not include any free water:

— sampling methods described in EN ISO 3170:2004, Clause 8 are not relevant for FPBO;

— for verification of mixing efficiency application of the procedure as described in EN ISO 3170:2004, 9.3.2 is not recommended;

— water content determination should only be carried out according to ASTM E203.

Even though the separation of extractives is very slow, the samples shall be taken immediately after mixing (see Annex A for further instructions).

If bio-oil samples are not analysed immediately, samples should be stored in a freezer [3, 4, 5, 14, 15].

It is pointed out that the sampling devices, sample bottles, and other devices in contact with bio-oil have to be compatible with bio-oil (see Annex B). Bio-oil shall be well mixed when transferring from the primary sampling process and/or container to another container and/or analytical apparatus. Minimum of two samples should be taken and the maximum difference of the viscosity shall not exceed ± 5 % at 40 °C [5]. A minimum of 0,1 L sample size is recommended.

The bio-oil shall be properly mixed and analysed according to the recommended standard methods. The bio-oil shall not be filtered or preheated above 40 °C for more than 30 min even though mentioned in some of the analysis standards. Fast pyrolysis bio-oils can typically be analysed like single-phase bio-oils because the separation of extractive-rich layer is very slow. However, the sampling and analyses should be carried out immediately after sample homogenization.

5 Requirements and test methods

5.1 Additives

In order to improve the storage stability, the use of additives, like alcohols, is allowed. Suitable fuel additives without known harmful side-effects are recommended in the appropriate amount, to help to avoid aging reactions in the fast pyrolysis bio-oil.

5.2 Generally applicable requirements and related test methods

When tested by the methods indicated in Table 1, fast pyrolysis bio-oils shall be in accordance with the limit values specified in the Table 1. The properties listed in the Table 1 have been assessed for application in boiler use [6, 7]. Precision data from inter-laboratory test programmes are given in Annex C.

Table 1 — Generally applicable requirements and test methods for fast pyrolysis bio- oils for boiler use

Property	Unit	Test Method	Limit value (minimum or maximum)
Net calorific value, on wet basis [a]	MJ/kg	DIN 51900–3	≥ 14,0
Water content, on wet basis	%(*m/m*)	ASTM E203	≤ 30
pH		ASTM E70	≥ 2,0
Density at 15 °C	kg/m^3	EN ISO 12185	≤ 1 300
Pour point	°C	ISO 3016	≤ - 9
Nitrogen content, (d.b.[b]).	% (*m/m*)	ASTM D5291	report
[a] Net calorific value on wet basis is calculated from the gross calorific value according to DIN 51900–1. [b] d.b. is on dry basis.			

5.3 Transport and general safety requirements and related test methods

The UN transport of goods regulation [16] considers liquids with a flash point of more than 35 °C which do not sustain combustion as flammable liquids. Due to their consistency and water content (see Table 1 and Annex C) FPBO are in general non-flammable liquids and would not sustain combustion at a test temperature above 60,5 °C (as prescribed in EN ISO 9038 [17]).

In line with the above FPBO that fulfils the following requirements are considered to be able to sustain combustion:

— Flash point as measured by Procedure B of EN ISO 2719 is higher than 35 °C; and

— Sustained combustibility passes the test procedure as in EN ISO 9038.

NOTE Results of ILS analysis (Annex C) show that flash point is not suitable analysis method for FPBO. Also, according other research results [17] FPBO is non-flammable liquid.

5.4 Emission and burner dependent requirements and related test methods

For emission and burner dependent requirements [6], options are given to allow grades to be set locally or chosen by the user. The options are two grades, of which Grade 1 requires more flue gas treatment than Grade 2. When tested by the methods indicated in Table 2, fast pyrolysis bio-oils shall be in accordance with the maximum limit specified in the Table 2 for the Grade applicable. The test methods listed in Table 2 have been assessed for application in boiler use.

Table 2 — Emission and burner dependent requirements and test methods for fast pyrolysis bio oil for boiler use

Property	Test method	Unit	Limit value (maximum)	
			Grade 1	Grade 2
Kinematic viscosity at 40 °C	EN ISO 3104	mm^2/s	125	50
Sulfur content	EN ISO 20846	%(*m/m*), d.b. [a)]	0,1	0,05
Solids content	ASTM D7579	%(*m/m*), wet basis	2,5	0,5
Ash content	EN ISO 6245	%(*m/m*), d.b. [a)]	0,25	0,05
Na, K, Ca, Mg	EN 16476	%(*m/m*) d.b. [a)]	-	0,02
[a)] d.b. is dry basis				

5.5 Precision and dispute

For all test methods referred to in this European Standard a precision statement has been developed. In cases of dispute, the procedures described in EN ISO 4259 for resolving the dispute apply, and the precision data from Annex C should be used.

Annex A
(informative)

Storage of fast pyrolysis bio-oil

A.1 Temperature

Recommended storage conditions for large batches of bio-oil are 15 °C to 20 °C and maximum of six months. Storage at a minimum temperature of 15 °C is recommended to maintain adequate fluidity, but not higher than 20 °C for long-term storage. Long time at relatively high storage temperatures accelerate viscosity increase of the fuel [3, 6].

A.2 Mixing

There are always some solids in FPBO but they are not considered as a separate phase in FPBO. After mixing solids are evenly distributed in the bio-oil. An extractive-rich phase may be gradually separated to top layer of the liquid. The stratification to top and bottom phase is slow and can be avoided by constant mixing. Similar type of phenomena is observed also with some agro-biomass. If moderate mixing is provided the fast pyrolysis bio-oil is a homogenous liquid [3, 14].

A.3 Ageing

Ageing can lead to phase separation. All fast pyrolysis bio-oils can separate into aqueous fraction and water insoluble fraction due to changes in chemical composition during ageing or due to other reasons. For example, when the amount of polar content (water, “sugars”) of bio-oil increases to around 60 % (*m/m*) and non-polar (water-insolubles) above 30 % (*m/m*), phase separation might take place [14]. It is also pointed out that fast pyrolysis bio-oils do not contain any free water by the limitation of around 30 %(*m/m*) water within the specification.

Storage in a continuously agitated or circulated container is recommended to maintain homogeneity and easy handling. It is also recommended to limit excessive exposure to air to prevent oxidation. Stored fuel is recommended to be periodically sampled and its quality assessed. Measurement and comparison of the stored fuel’s viscosity can be used to assess viscosity increase. Fuels that have undergone mild to moderate polymerisation can often be consumed in a normal way for example by increasing the fuel pre-heating. Pre-heating temperature is recommended to be between 60 °C and 80 °C at maximum. Filters and other clean-up equipment can require special attention and increased maintenance.

Annex B
(normative)

Compatible materials

All materials in contact with fast pyrolysis bio-oils should be made of corrosion resistant steel and materials such as AISI 304, AISI 316, PTFE (polytetrafluoroethylene), PP (polypropylene), HDPE (high density polyethylene) and PVC (polyvinylchloride).

Annex C
(normative)

Details of inter-laboratory test programme

Tables C.1 to C.12 present the precision data obtained in inter-laboratory testing programme by CEN/TC 19, partially funded by the European Commission and EFTA. Eighteen laboratories from Europe, USA and Canada participated in the inter-laboratory survey. Eight sets of samples (sets a, b, c, d, e, f, g and h) were prepared. Each set contains two sets of identical sample for double determination. In total 1 078 samples were analysed. Samples in the Tables in this Annex include a sample number and set number.

By using EN ISO 4259 precision equation for bio-oils have been developed. These are given after each respective Table. Not enough data have been returned for the determination of precision for the determination of Na, K, Ca and Mg content using EN 16476. The lab test results returned for pour point (via ISO 3014 or ASTM D97) and flash point (by EN ISO 2719) have been inconclusive for calculating proper repeatability and reproducibility determine.

Definition of symbols used in the Tables:

n is the number of laboratories after outlier elimination;

l is the number of outlier free individual analytical values;

o is the percentage of values from replicate determination;

x is the overall mean of the sample;

C_{VR} is the coefficient of the variation of the reproducibility;

C_{Vr} is the coefficient of the variation of the repeatability;

s_R is the reproducibility standard deviation;

s_r is the repeatability standard deviation;

r_s is the repeatability of that specific sample;

R_s is the reproducibility of that specific sample;

X is the average of the two results being compared.

Table C.1 — Performance data for net calorific value (wet basis), DIN 51900–3

	n	l	o	x	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
Sample			%	MJ/kg		MJ/kg	%		MJ/kg	%
001-a	9	18	0,0	16,962 2	0,860 0	2,809 5	5,070 1	0,182 5	0,596 2	1,075 9
002-a	9	18	0,0	20,586 9	0,855 7	2,795 4	4,156 5	0,124 4	0,397 5	0,604 3
003-a	9	18	0,0	14,671 3	0,910 4	2,974 0	6,205 3	0,140 2	0,458 0	0,955 6
005-a	9	18	0,0	16,189 2	0,937 9	3,063 8	5,793 4	0,106 9	0,341 6	0,660 3
006-a	9	18	0,0	17,919 7	0,921 9	3,011 6	5,144 6	0,057 4	0,183 4	0,320 3
007-a	9	18	0,0	17,384 6	0,947 7	3,096 0	5,451 4	0,092 3	0,301 4	0,530 9
008-a	9	18	0,0	19,154 6	0,937 3	3,062 0	4,893 3	0,150 6	0,491 9	0,786 2

Repeatability r = 0,358 MJ/kg (DIN 51900–3)

Reproducibility R = 2,910 MJ/kg (DIN 51900–3)

Table C.2 — Performance data for water content (wet basis, ASTM E203)

	n	l	o	x	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
Sample			%	%(*m*/*m*)		%(*m*/*m*)	%		%(*m*/*m*)	%
001-a	12	24	0,0	22,475 0	1,963 9	5,763 9	8,738 2	2,174 9	6,705 1	9,677 0
002-a	12	23	4,2	16,026 1	0,806 6	2,379 3	5,033 0	0,689 5	2,145 3	4,302 4
003-a	12	24	0,0	29,433 3	1,227 4	3,593 2	4,170 1	1,194 8	3,683 5	4,059 3
005-a	12	24	0,0	22,366 7	1,673 3	4,910 8	7,481 2	1,509 4	4,653 5	6,748 4
006-a	12	24	0,0	19,416 7	0,900 3	2,584 6	4,636 7	0,694 6	2,141 5	3,577 3
007-a	12	24	0,0	21,300 0	1,024 7	2,999 6	4,810 8	1,010 0	3,113 7	4,741 8
008-a	12	24	0,0	20,108 3	1,696 6	4,870 8	8,437 3	1,307 4	4,030 5	6,501 8

Repeatability $r = 0{,}148\,6\,X + 0{,}573\,7$ %(*m*/*m*) (ASTM E203)

Reproducibility $R = 0{,}100\,8\,X + 1{,}694\,2$ %(*m*/*m*) (ASTM E203)

Table C.3 — Performance data for pH (ASTM E70)

Sample	*n*	*l*	*o*	*x*	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
			%				%			%
001-c	7	14	0,0	2,480 0	0,144 3	0,444 8	5,818 5	0,104 9	0,342 8	4,229 8
002-c	7	14	0,0	3,165 6	0,130 3	0,401 6	4,116 1	0,098 1	0,320 4	3,098 9
003-c	6	11	21,4	2,105 4	0,058 0	0,193 4	2,754 8	0,028 0	0,097 0	1,329 9
004-c	6	11	21,4	1,714 6	0,077 6	0,239 3	4,525 8	0,076 5	0,265 2	4,461 7
005-c	7	13	7,1	2,190 7	0,086 8	0,283 4	3,962 2	0,032 4	0,112 3	1,479 0
006-c	7	13	7,1	2,520 0	0,143 1	0,477 8	5,678 6	0,026 6	0,088 8	1,055 6
007-c	7	14	0,0	2,625 3	0,169 9	0,543 0	6,471 6	0,080 4	0,268 3	3,062 5

Repeatability $r = 0{,}087\ 2\ X + 0{,}005\ 0$ (ASTM E70)

Reproducibility $R = 0{,}192\ 2\ X - 0{,}092\ 4$ (ASTM E70)

Table C.4 — Performance data for density at 15 °C (EN ISO 12185)

Sample	*n*	*l*	*o*	*x*	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
			%	kg/m³		kg/m³	%		kg/m³	%
001-b	5	8	20,0	1 187,59	0,910 0	3,300 0	0,076 6	0,300 0	1,190 0	0,025 3
002-b	5	10	0,0	1 219,92	1,280 0	4,080 0	0,104 9	0,970 0	3,350 0	0,079 5
003-b	5	10	0,0	1 180,14	2,740 0	8,510 0	0,232 2	2,570 0	8,910 0	0,217 8
004-b	4	8	20,0	1 223,83	0,600 0	1,980 0	0,049 0	0,520 0	1,910 0	0,042 5
005-b	5	10	0,0	1 245,88	2,920 0	9,220 0	0,234 4	2,700 0	9,350 0	0,216 7
006-b	5	10	0,0	1 225,31	1,460 0	4,890 0	0,119 2	0,860 0	3,000 0	0,070 2
007-b	5	10	0,0	1 229,38	0,880 0	2,760 0	0,071 6	0,760 0	2,630 0	0,061 8
008-b	5	10	0,0	1 205,73	2,210 0	7,080 0	0,183 3	1,790 0	6,220 0	0,148 5

Repeatability $r = -2{,}637 \times 10^{-5}\ X + 4{,}538$ kg/m³ (ISO 12815)

Reproducibility $R = -0{,}008\ X + 15{,}442$ kg/m³ (ISO 12815)

Table C.5 — Performance data for pour point (ISO 3016)

Sample	n	l	o	x	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
			%	°C		°C	%		°C	%
001-d	5	10	0,0	−27,0	9,721 1	32,444 6	36,004 1	7,348 5	26,708 2	27,216 6
002-d	5	10	0,0	−11,4	8,949 9	29,237 7	78,507 5	10,564 1	38,395 5	92,667 5
003-d	5	10	0,0	−7,80	4,449 7	15,417 5	57,047 7	2,683 3	9,752 5	34,401 0
004-d	5	10	0,0	−15,0	8,836 0	28,865 8	58,906 7	7,707 1	28,011 8	51,380 9
005-d	5	10	0,0	−24,1	6,853 8	22,390 3	28,439 1	5,753 3	20,910 4	23,872 4
006-d	5	10	0,0	−27,9	10,744 0	37,224 7	38,507 5	7,035 6	25,571 2	25,217 3
007-d	5	9	10,0	−31,0	5,100 6	17,672 5	16,453 4	3,824 3	15,035 2	12,336 3
008-d	5	10	0,0	−2,40	6,623 8	22,107 3	275,992 5	4,837 4	17,581 5	201,556 0

No repeatability and reproducibility determined.

Table C.6 — Performance data for nitrogen content (wet basis, ASTM D5291))

Sample	n	l	o	x	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
			%	%(m/m)		%(m/m)	%		%(m/m)	%
001-a	11	18	18,2	0,080 5	0,063 7	0,208 17	79,155 3	0,0123 0	0,039 33	15,280
002-a	11	22	0,0	0,783 2	0,094 3	0,293 49	12,044 5	0,0296 9	0,092 36	3,791 0
003-a	9	14	22,2	0,069 4	0,071 4	0,238 25	102,823	0,0162 5	0,056 31	23,405
005-a	11	21	4,5	0,164 9	0,059 5	0,181 81	36,094 6	0,0316 5	0,099 83	19,193
006-a	9	14	22,2	0,067 9	0,044 0	0,143 70	64,757 8	0,0168 3	0,058 30	24,776
007-a	9	14	22,2	0,068 1	0,060 6	0,197 85	88,875 8	0,0305 5	0,101 96	44,834
008-a	9	17	5,6	0,314 0	0,076 2	0,248 90	24,264 3	0,0136 6	0,044 63	4,350 3

Repeatability $r = 0{,}028\ 3\ X + 0{,}064\ 0$ %(m/m) (ASTM D5291)

Reproducibility $R = 0{,}144\ 0\ X + 0{,}184\ 1$ %(m/m) (ASTM D5291)

Table C.7 — Performance data for kinematic viscosity at 40 °C (EN ISO 3104)

	n	l	o	x	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
Sample			%	mm²/s		mm²/s	%		mm²/s	%
003-b	6	11	8,3	14,010	0,500 1	1,732 7	3,569 6	0,249 8	0,907 8	1,783 0
004-b	6	12	10,0	55,580	1,058 8	3,384 1	1,905 0	0,822 7	2,850 6	1,480 2
005-b	6	11	8,3	78,496	1,802 9	6,246 8	2,296 8	0,632 8	2,299 9	0,806 2
006-b	6	12	0,0	70,663	2,548 2	7,928 2	3,606 1	2,647 1	9,171 6	3,746 1
007-b	6	10	16,7	60,214	1,587 7	5,770 5	2,636 8	0,322 4	1,267 4	0,535 4
008-b	5	10	16,7	20,936	2,408 4	7,867 9	11,503 6	2,227 4	8,095 6	10,639 1

Repeatability $r = 0{,}004\ 7\ X + 3{,}904$ mm²/s (EN ISO 3104)

Reproducibility $R = 0{,}036\ 8\ X + 3{,}656$ mm²/s (EN ISO 3104)

Table C.8 — Performance data for sulfur content (wet basis, EN ISO 20846)

	n	l	o	x	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
Sample			%	%(*m*/*m*)		%(*m*/*m*)	%		%(*m*/*m*)	%
001-d	6	12	0,0	0,007 3	0,001 4	0,004 8	18,715 8	0,000 5	0,001 6	6,420 8
002-d	6	11	8,3	0,062 8	0,022 0	0,080 0	35,070 1	0,001 8	0,006 5	2,836 2
003-d	6	12	0,0	0,058 4	0,007 7	0,026 6	13,155 2	0,002 8	0,009 5	4,710 5
004-d	6	12	0,0	0,007 4	0,001 4	0,004 8	18,573 4	0,000 5	0,001 6	6,191 1
005-d	6	12	0,0	0,032 3	0,005 6	0,022 0	17,333 3	0,001 4	0,005 4	4,279 1
006-d	6	12	0,0	0,483 5	0,053 4	0,194 1	11,041 7	0,007 1	0,024 5	1,460 1
007-d	6	12	0,0	0,020 3	0,003 0	0,011 0	14,926 1	0,000 9	0,003 2	4,532 0
008-d	6	12	0,0	0,020 6	0,004 1	0,013 8	20,126 4	0,002 4	0,008 2	11,424

Repeatability $r = 0{,}043\ 6\ X + 0{,}003\ 8$ %(*m*/*m*) (EN ISO 20846)

Reproducibility $R = 0{,}386\ 4\ X + 0{,}011\ 2$ %(*m*/*m*) (EN ISO 20846)

Table C.9 — Performance data for solid content (wet basis, ASTM D7579)

Sample	n	l	o	x	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
			%	%(m/m)		%(m/m)	%		%(m/m)	%
001-e	6	12	0,0	0,038 5	0,055 1	0,171 5	143,170 6	0,015 4	0,0534	39,998 0
002-e	8	16	11,1	0,595 6	0,075 6	0,231 0	12,695 6	0,075 3	0,246 0	12,640 2
003-e	9	18	0,0	7,081 9	0,162 0	0,517 7	2,287 1	0,082 8	0,264 8	1,169 7
004-e	7	12	14,3	0,053 7	0,062 5	0,216 7	116,459 0	0,005 4	0,019 5	9,994 2
005-e	9	18	0,0	0,659 3	0,067 4	0,202 1	10,225 0	0,013 9	0,044 5	2,112 6
006-e	6	10	16,7	0,037 6	0,052 3	0,190 1	139,218 8	0,010 0	0,039 2	26,576 6
007-e	9	17	5,6	0,444 4	0,041 7	0,136 2	9,381 4	0,009 0	0,029 5	2,028 7
008-e	7	14	12,5	0,205 0	0,065 3	0,218 1	31,849 9	0,045 7	0,152 7	22,301 0

Repeatability $r = 0{,}027\ 7\ X + 0{,}074\ 6$ %(m/m) (ASTM D7579)

Reproducibility $R = 0{,}047\ 1\ X + 0{,}181\ 7$ %(m/m) (ASTM D7579)

Table C.10 — Performance data for ash content (wet basis, EN ISO 6245)

Sample	n	l	o	x	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
			%	%(m/m)		%(m/m)	%		%(m/m)	%
001-e	7	14	0,0	0,015 0	0,004 8	0,014 9	32,267	0,004 3	0,014 3	28,600
002-e	7	14	0,0	0,106 8	0,013 0	0,040 3	12,136	0,010 6	0,035 3	9,916 7
003-e	7	14	0,0	1,572 9	0,129 5	0,399 4	8,236 0	0,119 3	0,398 0	7,581 7
004-e	7	13	7,1	0,137 5	0,010 2	0,035 2	7,394 2	0,002 7	0,009 3	1,948 5
005-e	7	14	0,0	0,134 0	0,019 8	0,066 1	14,776	0,007 5	0,024 9	5,574 6
006-e	7	13	7,1	0,016 1	0,005 2	0,016 4	32,027	0,003 2	0,011 2	20,149
007-e	7	12	14,3	0,164 8	0,005 1	0,016 2	3,075 9	0,003 4	0,012 5	2,087 0
008-e	6	11	21,4	0,136 4	0,029 8	0,103 1	21,832	0,013 0	0,047 3	9,548 3

Repeatability $r = 0{,}253\ 3\ X - 0{,}003\ 2$ %(m/m) (EN ISO 6245)

Reproducibility $R = 0{,}243\ 1\ X + 0{,}017\ 1$ %(m/m) (EN ISO 6245)

Table C.11 — Performance data for total hydrogen content (wet basis, DIN 51900–1)

Sample	n	l	o	x^a	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
			%	%(*m*/*m*)			%			%
001-a	11	22	0,0	7,670 0	0,282 7	0,863 5	3,685 8	0,155 0	0,482 4	2,020 9
002-a	11	22	0,0	7,367 3	0,199 5	0,615 0	2,707 9	0,092 0	0,286 4	1,248 8
003-a	10	20	0,0	7,882 5	0,238 5	0,742 1	3,025 7	0,096 9	0,305 7	1,229 3
005-a	11	21	4,5	7,434 8	0,177 5	0,552 4	2,387 4	0,064 1	0,202 3	0,862 2
006-a	9	18	10,0	7,456 7	0,105 9	0,329 4	1,420 2	0,055 7	0,178 0	0,747 0
007-a	9	18	10,0	7,586 1	0,137 7	0,434 1	1,815 2	0,060 5	0,193 4	0,797 5
008-a	9	19	5,0	7,666 8	0,204 6	0,645 1	2,668 6	0,067 1	0,214 5	0,875 2
[a] includes H in water part of the fuel										

Repeatability $r = 0{,}224\ 3\ X - 1{,}434\ 2$ %(*m*/*m*) (DIN 51900–1)

Reproducibility $R = 0{,}565\ 5\ X - 3{,}689\ 5$ %(*m*/*m*) (DIN 51900–1)

Table C.12 — Performance data for flash point (EN ISO 2719)

Sample	n	l	o	x	s_R	R_s	C_{VR}	s_r	r_s	C_{Vr}
			%	°C		°C	%		°C	%
001-h	5	9	10,0	56,555 6	6,047 3	21,979 2	10,693	2,291 3	9,008 2	15,928 0
002-h	1	2	50,0	95,000 0	-	-	-	-	-	-
003-h	6	10	16,7	57,700 0	5,784 8	21,024 9	10,026	1,457 7	5,731 1	9,932 6
004-h	4	7	12,5	39,785 7	8,542 0	38,414 9	21,470	1,925 7	8,660 3	21,767 4
005-h	6	12	0,0	60,083 3	18,656 7	59,629 0	31,051	14,906 7	51,648 9	85,962 2
006-h	4	5	37,5	77,400 0	25,988 8	102,175 2	33,577	20,506 1	368,300 0	475,839 8

No repeatability and reproducibility determined.

Annex D
(normative)

Information on test method procedures

It is recommended to pay attention on calibration standards used. For example, for analysis of carbon, nitrogen and hydrogen, the calibration standard should contain similar level of nitrogen than the sample.

Table D.1 presents modifications to standard methods that shall be followed. These steps have been used in determining the precision as presented in Annex C. CEN intends to update FPBO in the scope of those analysis methods not yet including FPBO.

Also included in Table D.1 are similar, commonly used standards that may be used as alternative and can be more familiar to some laboratories. It is expected that for these standards the similar precision as in Annex C applies.

Table D.1 — Recommended modifications to the standard analysis methods to analyse FPBO

Property	Test Method	Modification to the test method
Net calorific value	DIN 51900–3 and DIN 51900–1 or ASTM D240 [8] and ASTM D5291	FPBO not included in the scope of the test method.
Water content	ASTM E203	FPBO not included in the scope of the test method. Methanol-Chloroform (3:1) as a solvent. HYDRANAL K reagents (Composite 5K and Working Medium K) in case of a fading titration end point. 50 ml solvent for two determinations. Sample size about 0,25 g (water content > 20 m/m-%). Stabilization time 30 s.
pH	ASTM E70	FPBO not included in the scope of the test method. Checking the pH meter with pH 4 buffer solution. Maximum allowed difference ± 0,05. If larger, calibration is needed, see the standard.
Density at 15 °C	EN ISO 12185 or ASTM D4052 [9]	FPBO not included in the scope of the test method. Careful mixing/rolling of the bottle of foam-prone forest residue liquids in order to avoid air bubbles. The sample is not shaken to avoid the air bubbles, but turned around carefully
Pour point	ISO 3016 or ASTM D97 [10]	FPBO not included in the scope of the test method. No preheating of the sample.
Nitrogen content	ASTM D5291	FPBO not included in the scope of the test method. At least triplicates and representative standards are recommended. Single determination (sample is weighted just before analysis). Use Com-aid, which is aluminium oxide, to prevent from splashing of the sample. Weighed sample is put into the sample crucible, sample is covered with com-aid (no need to weight), and combusted. Maximum sample size 120 mg.

Property	Test Method	Modification to the test method
Flash point	EN ISO 2719 or ASTM D93 [11], B	FPBO not included in the scope of the test method. Elimination of air bubbles before sampling. The sample is not shaken to avoid the air bubbles, but turned around carefully
Sustained combustibility	EN ISO 9038	FPBO not included in the scope of the test method.
Kinematic viscosity at 40 °C	EN ISO 3104 or ASTM D445 [12]	FPBO not included in the scope of the test method. Cannon-Fenske viscometer tubes. No pre-filtration of the sample if visually homogenous. Air bubbles can disturb the determination. The sample is not shaken to avoid the air bubbles, but turned around carefully. Equilibration time 15 min. Maximum allowed difference of duplicates 5 %.
Sulfur content	EN ISO 20846 or ASTM D5453 [13]	FPBO not included in the scope of the test method.
Solids content	ASTM D7579	Proper mixing of the sample prior sampling needed to make the sample homogenous.
Ash content	EN ISO 6245	FPBO not included in the scope of the test method. Size of crucible minimum of 150 ml (width 80 mm, height 55 mm) should be used in order to avoid splashing of the sample. Cool always same time crucible in desiccator.
Na, K, Ca, Mg	EN 16476	FPBO not included in the scope of the test method. In the standard EN 16476 the sample is dissolved in kerosene which shall be replaced by methanol because of the solubility of FPBO. This might require some material changes in ICP-OES. The analysis can also be performed after dissolution of the FPBO in a mineral acid.

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