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Bioolja, pridobljena s hitro pirolizo, za industrijske kotle - Zahteve in preskusne metode

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

Fast Pyrolyse-Öle für die stationäre Wärmeerzeugung - Anforderungen und Prüfverfahren

Huiles de pyrolyse rapide pour application chaudières - Spécifications et méthodes d'analyses

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

Huiles de pyrolyse rapide pour application chaudières - Spécifications et méthodes d'analyses

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of an identical text or by endorsement, at the latest by September 2017, and conflicting national standards shall be withdrawn at the latest by September 2017.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/m³	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.