BS EN ISO 16948:2015

BSI Standards Publication

Solid biofuels — Determination of total content of carbon, hydrogen and nitrogen

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National foreword

This British Standard is the UK implementation of EN ISO 16948:2015. It supersedes BS EN 15104:2011 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/17, Solid biofuels.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Biogene Festbrennstoffe - Bestimmung des Gesamtgehaltes an Kohlenstoff, Wasserstoff und Stickstoff - Instrumentelle Verfahren (ISO 16948:2015)

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Foreword

This document (EN ISO 16948:2015) has been prepared by Technical Committee ISO/TC 238 "Solid biofuels" in collaboration with Technical Committee CEN/TC 335 “Solid biofuels” the secretariat of which is held by SIS.

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of an identical text or by endorsement, at the latest by November 2015, and conflicting national standards shall be withdrawn at the latest by November 2015.

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The text of ISO 16948:2015 has been approved by CEN as EN ISO 16948:2015 without any modification.

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Foreword

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

For the purposes of research on instrumental methods for the determination of total carbon, hydrogen and nitrogen contents in solid biofuels standards, users are encouraged to share their views on ISO 16948:2015 and their priorities for changes to future editions of the document. Click on the link below to take part in the online survey:

ISO 16948 online survey

Introduction

Instrumental methods for the analysis of carbon, hydrogen and nitrogen are now in widespread and in regular use, often in preference to formerly developed chemical methods for which International Standards exist.

The reliable determination of carbon, hydrogen and nitrogen is important for quality control and the results can be used as input parameters for calculations applied to the combustion of solid biofuels. The environmental importance of the nitrogen content is linked to emissions of NO_x (formation of fuel NO_x). Hydrogen content is important for calculation of the net calorific value. Carbon content is required for the determination of CO_2-emissions.

It is recognized that the Kjeldahl method is most reliable for determining nitrogen contents with a concentration lower than 0,1 %. Possible suitable methods are summarized in the bibliography.

Solid biofuels — Determination of total content of carbon, hydrogen and nitrogen

1 Scope

This International Standard describes a method for the determination of total carbon, hydrogen and nitrogen contents in solid biofuels.

2 Normative references

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

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 14780[1], *Solid Biofuels — Sample preparation*

ISO 16993, *Solid biofuels — Conversion of analytical results from one basis to another*

3 Terms and definitions

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

3.1
reference material
RM
material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials

3.2
certified reference material
CRM
reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence

3.3
NIST standard reference material
SRM
CRM issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterisations and provides information regarding the appropriate use(s) of the material

Note 1 to entry: The National Institute of Standards and Technology (NIST), known between 1901 and 1988 as the National Bureau of Standards (NBS), is a measurement standards laboratory, also known as a National Metrological Institute (NMI), which is a non-regulatory agency of the United States Department of Commerce

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[1] To be prepared.

4 Principle

A known mass of sample is burnt in oxygen, or in an oxygen/carrier gas mixture, under conditions such that it is converted into ash and gaseous products of combustion. These consist mainly of carbon dioxide, water vapour, elemental nitrogen and/or oxides of nitrogen, oxides and oxyacids of sulfur and hydrogen halides. The products of combustion are treated to ensure that any hydrogen associated with sulfur or halides products of combustion are liberated as water vapour. Oxides of nitrogen are reduced to nitrogen, and those products of combustion which would interfere with the subsequent gas-analysis procedures are removed. The carbon dioxide, water vapour and nitrogen mass fractions of the gas stream are then determined quantitatively by appropriate instrumental gas analysis procedures.

5 Reagents and calibration substances

5.1 General

WARNING — Care should be exercised when handling reagents, many of which are toxic and corrosive.

Unless otherwise stated, use only reagents and calibration standards of recognized analytical grade for the analysis.

5.2 Carrier gas

The carrier gas used is helium or another suitable gas as specified by the instrument manufacturer.

5.3 Oxygen

Oxygen is used as specified by the instrument manufacturer.

5.4 Additional reagents

Additional reagents are of types and qualities as specified by the instrument manufacturer.

5.5 Calibration substances

Examples of pure organic substances suitable for calibration are given in Table 1.

Table 1 — Examples of suitable calibration substances and their theoretical C, H and N contents

Name	Formula	% C	% H	% N
Acetanilide	C_8H_9NO	71,1	6,7	10,4
Atropin	$C_{17}H_{23}NO_3$	70,6	8,0	4,8
Benzoic acid	$C_7H_6O_2$	68,8	5,0	0,0
Cystine	$C_6H_{12}N_2O_4S_2$	30,0	5,0	11,7
Diphenyl amine	$C_{12}H_{11}N$	85,2	6,6	8,3
EDTA	$C_{10}H_{16}N_2O_8$	41,1	5,5	9,6
Phenylalanine	$C_9H_{11}NO_2$	65,4	6,7	8,5
Sulfanil amide	$C_6H_8N_2O_2S$	41,8	4,7	16,3
Sulfanilic acid	$C_6H_7NO_3S$	41,6	4,1	8,1
TRIS	$C_4H_{11}NO_3$	39,7	9,2	11,6

The materials shall be dry and of high purity, i.e. more than 99,9 %. For calibration purposes, the contents of C, H and N according to the certificate of the materials shall be used, not the theoretical contents. Other pure materials can be used provided that they meet the requirement of this standard.

5.6 Use of Certified Reference Materials (CRM or SRM)

Use certified reference materials, issued by an internationally recognized authority, to check if the accuracy of the calibration meets the required performance characteristics. Examples of certified reference materials are: NBS 1573 tomato leaves and NBS 1575 pine needles.

When, due to matrix effects or concentration range limitations, no good recoveries for the certified reference materials can be obtained, calibration with at least two CRM or SRM materials, may solve these problems. In that case CRM or SRM materials other than used for the calibration shall be used for verification purposes.

NOTE A CRM or SRM is prepared and used for three main purposes:

a) to help develop accurate methods of analysis;

b) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit;

c) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

6 Apparatus

No specific design of systems is presented here because there are a range of components and configurations available, which can be used to carry out the test method satisfactorily.

The apparatus shall, however, meet the following functional requirements:

a) The conditions of combustion of the sample shall be such that all of the carbon (including that in mineral carbonates), the hydrogen (including that in the water of constitution of the minerals), and the nitrogen present, shall be converted into carbon dioxide, water vapour (except for hydrogen associated with oxyacids of sulfur and volatile halides), and gaseous nitrogen and/or oxides of nitrogen respectively.

b) The combustion gases, or a representative aliquot, shall be treated to remove and/or separate out any components which would subsequently interfere with the detection and measurement of the carbon dioxide, water vapour or nitrogen in the gas stream.

c) Hydrogen present as hydrogen halides or sulfur oxyacids shall be liberated, as water vapour, into the gas stream prior to determination of water vapour content.

d) Any nitrogen oxides produced by the combustion process shall be reduced to nitrogen prior to presentation to the detection system.

e) The detection systems shall provide responses that correlate directly with the concentrations of the combustion gases, over the full range applicable and preferably in a linear manner.

f) If a nonlinear response is provided by a detection system, it shall include provisions for evaluating that response in a manner which correlates accurately with the concentration of the combustion gas.

g) It shall include a means of displaying the detector responses or of calculating and presenting the concentrations of carbon, hydrogen and nitrogen in the sample following the input of other appropriate data as necessary.

7 Preparation of the test sample

The test sample is the general analysis sample with a nominal top size of 1 mm or less, prepared in accordance with ISO 14780.

The moisture content of the test sample shall be determined concurrently by the method described in ISO 18134-3, using another portion of the test sample.

NOTE 1 For some instruments it may be necessary to prepare a test sample with a lower nominal top size than 1 mm, e.g. 0,25 mm, in order to keep the desired precision. For "new products" an adequate particle size should be determined by validation experiments.

NOTE 2 For some types of instruments it is necessary to carry out the determination of hydrogen on dried analysis samples to prevent erroneous low results. For a determination on dry sample, the analysis sample is dried as described in ISO 18134–3 immediately before the determination. When using dried samples there is a risk of obtaining erroneous low carbon results.

8 Procedure

8.1 Preparation of the test portion

Weigh, to the nearest 0,1 % (relative), a quantity of the test sample recommended by the instrument manufacturer as appropriate to the type of instrumentation and the expected concentrations of carbon, hydrogen and nitrogen. The test portion shall be weighed directly into the sample capsule in the case of a micro- or semi-micro analyser. Otherwise it may be weighed directly or transferred from a suitable weighing container.

8.2 Calibration of the apparatus

When the analytical system is evaluated for the first time, establish a calibration function for the measurement in accordance with the manufacturers' instructions. Adjust the established calibration function during the analysis if necessary. Check the performance of the instrument using the accepted standard procedures like replicate analysis, use of CRM and or Standard Reference Material (SRM), control samples and create control charts. The calibration and quality control scheme shall be organized and maintained in such a way that the required uncertainty of measurement can be obtained. The results of the validation study of BioNorm2 (Annex A) demonstrate what is achievable with commercial instruments that are used by experienced laboratories.

8.3 Analysis of test samples

Analyse test portions (8.1) of the test samples in accordance with the manufacturer's instructions.

At intervals between batches of samples, analyse a calibration substance (5.5), CRM (5.6) or an internal laboratory control sample.

The control sample shall have a carbon, hydrogen and nitrogen content comparable to the samples.

Example of an analysis run on an automated CHN analyser:

a) 2 dummy samples to condition the instrument

b) 3 samples of calibration substance to check or carry out the calibration

c) 1 laboratory control sample, to ensure the instrument performance for the actual type of samples

d) 9 samples (in duplicate)

e) 2 samples of calibration substance to check the calibration

f) Repetition of d) to e) until all samples are processed

g) 1 laboratory control sample

Calibration checks during an analysis run may be used for adjustment of the established calibration function cf. 8.2. Adjustments should only compensate for small drifts in the instrument performance; relative differences of more than 10 % usually will be an indication of possible malfunction of the instrument.

9 Expression of results

The total carbon, hydrogen and nitrogen contents of the biofuel sample, as analysed, shall be recorded as a percentage by mass.

Report the results on dry basis, as the mean of duplicate determinations. The following equations shall be used for the calculations to dry basis:

For the carbon content: $$C_d = C_{ad} \times \frac{100}{100 - M_{ad}} \tag{1}$$

For the nitrogen content: $$N_d = N_{ad} \times \frac{100}{100 - M_{ad}} \tag{2}$$

For the hydrogen content: $$H_d = \left(H_{ad} - \frac{M_{ad}}{8,937} \right) \times \frac{100}{100 - M_{ad}} \tag{3}$$

where

d is dry basis;

ad is as determined;

M_{ad} is the moisture content of the general analysis sample when analysed.

The constant factor 8,937 is for calculating the hydrogen concentration in the water that is present in the sample. The factor is obtained from the molar formula of water (H_2O) and the atomic weight of hydrogen (1,008) and oxygen (15,999 4).

The results may be calculated to an as received basis according to ISO 16993.

10 Performance characteristics

The achievable performance of the method is given in Annex A showing the results obtained by a European intercomparison study carried out for a sample of wood chips and a sample of an exhausted olive residue. The content of carbon and hydrogen of both samples represents the typical range for solid biofuels. Concerning the content of nitrogen in solid biofuels the samples of wood chips and exhausted olive residues represent the lowest and highest values expected, respectively.

11 Test report

The test report shall contain at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (sample) tested;

c) reference to this International Standard (ISO 16948:2015);

d) method for the determination;

e) the results of the test including the basis in which they are expressed, as indicated in Clause 9;

f) any unusual features noted during the test procedure;

g) any operation not included in this International Standard, or regarded as optional.

Annex A
(informative)

Performance data

The round robin was carried out by laboratories in Austria, Belgium, Denmark, Finland, Germany, Ireland, Italy, The Netherlands, Spain, Sweden and the United Kingdom. The variety of instruments and other analytical conditions were used in accordance with the quality parameters specified in the method.

The tests were carried out using two samples, wood chips and exhausted olive residues produced in the EU-project BioNorm according to ISO 14780. The sample "wood chips" was made of German coniferous wood chips; the chips were dried and milled to 1 mm by means of cutting mill. The sample "exhausted olive residues" was obtained from olive oil industry in Spain from a typical outdoor storage facility. In the original sample stones and other natural impurities were present. These impurities and stones were removed manually and the sample was prepared from the residues in two steps using a coarse cutting mill equipped with a 10 mm sieve and a laboratory cutting mill equipped with WC cutting tools and a 1 mm sieve.

All data are reported on dry basis.

The performance data according to ISO 5725-2 are presented in Tables A.1, A.2 and A.3.

NOTE 1 See Table A.1 for definition of the symbols used in the tables of A.1 to A.3.

NOTE 2 A guideline can be found in to ISO 16993:2015, Annex C on how to use these validation parameters.

Table A.1 — Performance data for Carbon (C)

Sample	n	l	o %	x % m/m	s_R % m/m	CV_R %	s_r % m/m	CV_r %
wood chips	27	128	1,5	50,3	0,55	1,1	0,14	0,29
exhausted olive residue	26	123	1,6	48,0	0,55	1,2	0,27	0,56
Definition symbols								
n	is the number of laboratories after outlier elimination							
l	is the number of outlier free individual analytical values							
o	is the percentage of outlying values from replicate determination							
x	is the overall mean							
s_R	is the reproducibility standard deviation							
CV_R	is the coefficient of the variation of the reproducibility							
s_r	is the repeatability standard deviation							
CV_r	is the coefficient of the variation of the repeatability							

Table A.2 — Performance data for Hydrogen (H)

Sample	n	l	o	x	s_R	CV_R	s_r	CV_r
			%	% m/m	% m/m	%	% m/m	%
wood chips	28	135	0	6,1	0,36	5,9	0,07	1,2
exhausted olive residues	24	115	0	5,7	0,32	5,77	0,06	1,1

Table A.3 — Performance data for Nitrogen (N)

Sample	n	l	o %	x % m/m	s_R % m/m	CV_R %	s_r % m/m	CV_r %
wood chips	18	84	6,7	0,10	0,04	30	0,01	7,4
exhausted olive residues	25	115	7,3	1,40	0,11	8,1	0,04	3,2

NOTE 1 The high s_R value of 0,04 % m/m concerning the determination of nitrogen in the wood chip sample is obtained due to a contribution from elemental analysers with a small sample intake (typical 2 mg to 5 mg) in combination with a thermal conductivity detector and with an insufficient removal of the air in the sample capsule.

The performance at low levels can be improved using the Kjeldahl method or element analyzers working with higher sample intake and/or a high sensitive nitrogen detection.

NOTE 2 The high s_R value of about 0,36 % m/m concerning the determination of hydrogen could be due to the following conditions concerning the procedures of the participating laboratories:

— determination on the moist (as received) analysis sample using a micro-analyser with the problem stated in the NOTE 2 of Clause 7;

— determination on the moist analysis sample without correction for the moisture-hydrogen included;

— determination on dried analysis sample material which between the drying and the analysis have absorbed moisture.

An s_R value of 0,2 % m/m should be obtainable when using a correct procedure.

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[9] NIST definitions: http://ts.nist.gov/MeasurementServices/ReferenceMaterials/DEFINITIONS.cfm.

[10] Pure Appl. Chem. 1990, **62** (1) pp. 149–162 [Harmonized protocol for the adoption of standardized analytical methods and for the presentation of their performance characteristics]

[11] NIST Technical note 1297:1994, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*

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