SLOVENSKI STANDARD
SIST EN 15104:2011

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Trdna biogoriva - Določevanje celotnega ogljika, vodika in dušika - Instrumentalne metode

Solid biofuels - Determination of total content of carbon, hydrogen and nitrogen - Instrumental methods

Feste Biobrennstoffe - Bestimmung des Gesamtgehaltes an Kohlenstoff, Wasserstoff und Stickstoff - Instrumentelle Verfahren

Biocombustibles solides - Détermination de la teneur totale en carbone, hydrogène et azote - Méthodes instrumentales

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Solid biofuels - Determination of total content of carbon, hydrogen and nitrogen - Instrumental methods

Biocombustibles solides - Détermination de la teneur totale en carbone, hydrogène et azote - Méthodes instrumentales

Feste Biobrennstoffe - Bestimmung des Gesamtgehaltes an Kohlenstoff, Wasserstoff und Stickstoff - Instrumentelle Verfahren

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Contents

Page

Foreword 3

Introduction 4

1 Scope 5

2 Normative references 5

3 Terms and definitions 5

4 Principle 5

5 Reagents and calibration substances 6
5.1 General 6
5.2 Carrier gas 6
5.3 Oxygen 6
5.4 Additional reagents 6
5.5 Calibration substances 6
5.6 Use of Certified Reference Materials (CRM or SRM) 7

6 Apparatus 7

7 Preparation of the test sample 7

8 Procedure 8
8.1 Preparation of the test portion 8
8.2 Calibration of the apparatus 8
8.3 Analysis of test samples 8

9 Expression of results 9

10 Performance characteristics 9

11 Test report 9

Annex A (informative) Performance data 11

Bibliography 13

Foreword

This document (EN 15104:2011) has been prepared by 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of an identical text or by endorsement, at the latest by August 2011, and conflicting national standards shall be withdrawn at the latest by August 2011.

This document supersedes CEN/TS 15104:2005.

In the pre-normative project BIONORM I&II a robustness test has been performed to find out if all critical parameters in the standard were addressed. Based on the results of that test it has been concluded that all critical parameters were covered. Only minor technical changes were necessary which have been implemented in the revised text. The revision also includes a change of deliverable from Technical Specification to European Standard and updated normative references.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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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 summarised in the bibliography.

1 Scope

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

2 Normative references

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

EN 14588:2010, *Solid biofuels — Terminology, definitions and descriptions*

EN 14774-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

FprEN 14780, *Solid biofuels — Sample preparation*

EN 15296, *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 EN 14588:2010 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

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

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

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

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 recognised authority, to check if the accuracy of the calibration meets the required performance characteristics. Examples of certified reference materials are: NBS 1570 spinach leaves, NBS 1571 orchard leaves, 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: (1) to help develop accurate methods of analysis; (2) 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; and (3) 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 ranges 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 non-linear 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 FprEN 14780.