SLOVENSKI STANDARD
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Trdna biogoriva ï Doloļ evanje celotnega ogljika, vodika in duġika - Instrumentalne metode

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

Feste Biobrennstoffe - Verfahren zur Bestimmung des Gehaltes an Gesamtkohlenstoff, Wasserstoff und Stickstoff - Instrumentelle Verfahren

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

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English Version

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 - Verfahren zur Bestimmung des Gehaltes an Gesamtkohlenstoff, Wasserstoff und Stickstoff - Instrumentelle Verfahren

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The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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Foreword

This Technical Specification (CEN/TS 15104:2005) has been prepared by Technical Committee CEN/TC 335 “Solid biofuels”, the secretariat of which is held by SIS.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

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

It was considered that the procedures adopted in these instrumental methods should be standardised as far as possible. As a consequence an ISO Technical Specification has recently been developed for solid mineral fuels.

The procedures to be followed when determining the mass fractions of total carbon (including that present as carbonates), total hydrogen (including that present as water) and nitrogen by instrumental methods are specified in ISO/TS 12902:2001 and underlie this Technical Specification.

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 Technical Specification describes 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.

CEN/TS 14588, *Solid Biofuels – Terminology, definitions and descriptions*.

CEN/TS 14774-3, *Solid Biofuels – Methods for the determination of moisture content – Oven dry method – Part 3: Moisture in general analysis sample.*

CEN/TS 14780, *Solid Biofuels – Methods for sample preparation.*

CEN/TS 15296, *Solid Biofuels – Calculation of analyses to different bases.*

3 Terms and definitions

For the purposes of this Technical Specification, the terms and definitions given in CEN/TS 14588 apply.

4 Principle

A known mass of the 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 is liberated as water vapour. Oxides of nitrogen are reduced to elemental 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 standards

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 standards

Examples of suitable calibration standards are given in Table 1.

Table 1 — Calibration standards

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	6,5	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	9,1	11,6

5.6 Certified reference materials (CRM)

Solid biofuels, coal or coke are issued by an internationally recognised authority.

NOTE For quality control, use certified reference materials, issued by an internationally recognised authority. Examples are: BCR 181 coking coal, BCR 182 steam coal, BCR 100 beech leaves, BCR 101 spruce needles, NBS 1570 spinach leaves, NBS1571 orchard leaves, NBS 1573 tomato leaves and NBS 1575 pine needles.

6 Apparatus

No specific design of systems is presented here because there is 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 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.

NOTE The provisions for the evaluation of the detector response and for the performance of any subsequent calculations required may be integral with the instrument or provided by appropriate auxiliary systems.

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 CEN/TS 14780.

The moisture content of the test sample shall be determined concurrently by the method described in CEN/TS 14774-3, using another portion of the test sample.

NOTE 1 For some types of instruments it is necessary to carry out the determination of hydrogen on dried analysis samples. Before carrying out direct analyses of moist analysis samples it therefore should be controlled, that identical results can be achieved compared with analyses carried out on dried samples. For a determination on dry sample, the analysis sample shall be dried at 105 °C as described in CEN/TS 14774-3 immediately before the determination.

NOTE 2 The nominal top size of the test sample shall be 1 mm or less. For some instruments/solid biofuels 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 stated precision (Table 2). For “new products” an adequate particle size should be determined by validation experiments.

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.