SLOVENSKI STANDARD
SIST-TS CEN/TS 15407:2007

01-marec-2007

Trdno alternativno gorivo - Metoda za določ evanje ogljika (C), vodika (H) in dušika (N)

Solid recovered fuels - Method for the determination of carbon (C), hydrogen (H) and nitrogen (N) content

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an Kohlenstoff (C), Wasserstoff (H) und Stickstoff (N)

Combustibles solides de récupération - Méthode pour la détermination de la teneur en carbone (C), en hydrogene (H) et en azote (N)

Ta slovenski standard je istoveten z: **CEN/TS 15407:2006**

ICS:

75.160.10	Trda goriva	Solid fuels

SIST-TS CEN/TS 15407:2007 **en**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 15407

September 2006

ICS 75.160.10

English Version

Solid recovered fuels - Method for the determination of carbon (C), hydrogen (H) and nitrogen (N) content

Combustibles solides de récupération - Méthode pour la détermination de la teneur en carbone (C), en hydrogène (H) et en azote (N)

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an Kohlenstoff (C), Wasserstoff (H) und Stickstoff (N)

This Technical Specification (CEN/TS) was approved by CEN on 25 March 2006 for provisional application.

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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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. CEN/TS 15407:2006: E

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Foreword

This document (CEN/TS 15407:2006) has been prepared by Technical Committee CEN/TC 343 “Solid Recovered Fuels”, the secretariat of which is held by SFS.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN 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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

The determination of carbon, hydrogen and nitrogen is usually performed using instrumental methods. The latter can be divided in two groups depending on the amount of test portion used. Micro instrumental methods require few mg of sample; macro methods use grams of sample. If micro methods are used for SRF analysis, a very homogeneous test sample needs to be prepared in order to obtain the required precision.

1 Scope

This Technical Specification describes a method for the determination of total carbon, hydrogen and nitrogen contents in solid recovered fuels by instrumental techniques.

This method is applicable for concentrations on dry matter basis of C over 0,1 %, N over 0,01 % and H over 0,1 %.

2 Normative references

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

CEN/TS 15357:2006, *Solid recovered fuels — Terminology, definitions and descriptions*

CEN/TS 15413, *Solid recovered fuels — Methods for the preparation of the test sample from the laboratory sample*

prCEN/TS 15414-3, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

3 Terms and definitions

For the purposes of this Technical Specification, the terms and definitions given in CEN/TS 15357:2006 apply.

4 Safety remarks

The safety in handling of potentially hazardous materials is dealt with in relevant national and European regulations, which every laboratory should refer to.

In addition the following information is given:

— instruments for carbon, hydrogen and nitrogen determination shall be used only by experienced personnel, following the safety instructions of the manufacturer.

5 Principle

The method is based on the complete oxidation of the sample ("flash combustion" instruments can also be used) which converts all organic substances into combustion products. The resulting combustion gases pass through a reduction furnace and are swept into the chromatographic column by the carrier gas (helium) where they are separated and detected quantitatively by appropriate instrumental gas analysis procedures (for example by a thermal conductivity detector (TCD)). The samples are held in a suitable container (tin or other crucible) and then dropped inside the quartz tube furnace at about 1000 °C in an oxygen stream for complete oxidation in the presence of a catalyst layer. Excess oxygen is removed by contact with copper, while nitrogen oxides are reduced to elemental nitrogen.

6 Reagents and calibration standards

All reagents shall be at least of analytical grade and suitable for their specific purposes.

6.1 **Carrier gas**: Helium, 99,99 % or other gases as specified by the instrument manufacturer.

6.2 **Oxygen**, free of combustion material, purity 99,95 %, or as specified by the instrument manufacturer.

6.3 **Additional reagents**: as specified by the instrument manufacturer.

6.4 **Calibration standards**

Examples are given in the following table.

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
Diphenylamine	$C_{12}H_{11}N$	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

7 Apparatus

Various instrumental configurations are available. The general requirements for a suitable apparatus are:

a) the combustion conditions shall be such that all carbon, hydrogen and nitrogen are converted to carbon dioxide, water vapour and nitrogen oxide or elemental nitrogen;

b) a separation step is included to reduce or eliminate any possible interference during the subsequent determination;

c) nitrogen shall be reduced to the elemental form before the detection;

d) analytical balance, resolution of at least 1 part per thousand of the weighted amount.

8 Procedure

8.1 Sample conservation and pre-treatment

The laboratory samples shall be stored according to guidelines defined in Annex A.

8.2 Sample preparation

The test portion shall be prepared from the laboratory sample according to CEN/TS 15413.

The amount of test portion depends on the particular instrument used. The particle size of the test sample should be related to the amount of sample to be used, according to CEN/TS 15413.

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 prCEN/TS 15414-3 immediately before the determination.

Whereas the determination is carried out on dry basis, the moisture content shall be determined according to prCEN/TS 15414-3.

8.3 Preparation of the test portion

Weigh the appropriate amount of material as recommended by the instrument manufacturer as appropriate for the type of instrument and the expected content 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.

8.4 Calibration

Set up the instrument following the manufacturer instructions.

Stabilize the furnace and analyzer.

Select 3 to 5 reference materials with increasing concentration of nitrogen, hydrogen and carbon. Calibrate the instruments for nitrogen, hydrogen and carbon determination following the manufacturer instructions. Use the same procedure as for sample analysis (see below). Alternatively, different amounts of the same substance can be used to prepare the calibration.

Verify the calibration by analysing as a test sample a portion of a suitable standard, preferably with a different material than that used for the calibration.

The calibration is acceptable if the measured value differs from the standard value by no more than the repeatability limit for the test method. Otherwise repeat the calibration procedure.

8.5 Analysis of samples

Weight the test portion and transfer it into the instrumental apparatus. Start the cycle following then operating instruction for the specific instruments. At least 3 replicates are necessary.

9 Expression of results

The total carbon, hydrogen and nitrogen contents of the solid recovered fuels shall be expressed as a percentage by mass on the dry basis. Most commercially available instruments give the results directly.

The following equations shall be used:

for the carbon content:

$$C_d = C_{ad} \times \frac{100}{100 - M_{ad}}$$

for the nitrogen content:

$$N_d = N_{ad} \times \frac{100}{100 - M_{ad}}$$

for the hydrogen content: