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

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

Combustibles solides de récupération - Méthodes 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)

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Contents

Page

Foreword

This document (EN 15407:2011) has been prepared by Technical Committee CEN/TC 343 “Solid recovered fuels”, the secretariat of which is held by SFS.

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of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

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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 European Standard specifies 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 document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15357:2011, *Solid recovered fuels — Terminology, definitions and descriptions*

EN 15413[1)], *Solid recovered fuels — Methods for the preparation of the test sample from the laboratory sample*

EN 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 document, the terms and definitions given in EN 15357:2011 apply.

4 Safety remarks

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

In addition the following information is given:

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

[1)] To be published.

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 1 000 °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 Table 1.

Table 1 — Calibration standards

Name	Formula	C %	H %	N %
Acetanilide	C_8H_9NO	71,1	6,7	10,4
Atropine	$C_{17}H_{23}NO_3$	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 EN 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 EN 15413.

For some types of instruments it is necessary to carry out the determination of hydrogen on dried analysis samples. For some other types of instruments it is necessary to carry out the determination of carbon on analysis samples that are not completely dried. Working with samples that has been dried at 105 °C and then equilibrated with the moisture in the air on the lab where the CHN analysis are handled, is a good compromise for the C and H moisture artefact, but then extra moisture determinations on the air-dry sample will have to be done.

The nominal top size of the test sample shall be 1 mm or less. 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 shall be determined by validation experiments.

Whereas the determination is carried out on dry basis, the moisture content shall be determined according to EN 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 may 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:

$$C_d = C_{ad} \times \frac{100}{100 - M_{ad}} \quad (1)$$

for the nitrogen content:

$$N_d = N_{ad} \times \frac{100}{100 - M_{ad}} \quad (2)$$

for the hydrogen content:

$$H_d = H_{ad} - \frac{M_{ad}}{8{,}937} \times \frac{100}{100 - M_{ad}} \quad (3)$$

where

$_d$ is dry basis;

$_{ad}$ is as determined;

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

10 Performance characteristics

Data on performance characteristics of the present method are given in Annex B which presents the results of the QUOVADIS validation project (Quality Management, Organisation, Validation of standards, Developments and Inquiries for SRF) [9].

11 Test report

The test report shall contain at least the following information:

a) name, address and location of any laboratory involved in the analysis;

b) description and identification of the laboratory sample;

c) date of receipt of laboratory sample and date(s) of performance of test;

d) a reference to this European Standard, i.e. EN 15407;

e) reference to the analytical standard used for the determination for each element;

f) the analytical results, referring to Clause 9;

g) any details not specified in this European Standard or which are optional, and any other factors which may have affected the results;

h) unique identification of report (such as serial number) and of each page and total number of pages of the report.

The laboratory should keep a trace of any analytical steps and intermediate results (chromatograms, raw data and calculation details) that should be kept available in case of specific requirements (e.g. accreditation).

Annex A
(normative)

Guidelines - Characteristics of the laboratory sample for chemical analysis of SRF

The following requirements apply when preparing the laboratory sample for the chemical characterisation of SRF samples according to this European Standard.

NOTE Equivalent requirements apply in all chemical test method specifications for SRF, i.e. this European Standard, EN 15408, EN 15410, EN 15411, CEN/TS 15412 and EN 15413.

A maximum amount of laboratory sample of 10 kg and maximum particle size of 1 cm is established on the basis of number and type of parameters to be determined, sample representativeness and practical reasons for handling samples. In the Table A.1 the requirements are summarised both for single or grouped chemical parameters.

Table A.1 — Requirements for the laboratory sample for the analysis of SRF

Parameter (single or group)	Minimum laboratory sample amount (g) [a]	Short term storage conditions before delivery to the lab	Long term storage condition before delivery to the lab	Container material
C, H, N	100	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Cl, S, Br, F	100	In the same condition of SRF production	refrigeration 4 °C	non-PVC plastic bottle or bag
Metallic Al	200	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Major elements	400	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Trace elements excluding Hg	200	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Hg	100	In the same condition of SRF production	refrigeration 4 °C	glass or PFA bottle
C, H, N, Cl, S, Br, F	150	In the same condition of SRF production	refrigeration 4 °C	non-PVC plastic bottle or bag
Major elements + trace elements excluding Hg	500	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Major elements + trace elements + Hg	600	In the same condition of SRF production	refrigeration 4 °C	glass bottle (100 g) + plastic bottle or bag

Table A.1 — (*concluded*)

Major elements + trace elements + Hg + metallic Al	700	In the same condition of SRF production	refrigeration 4 °C	glass bottle (100 g) + plastic bottle or bag
Complete analysis	800	In the same condition of SRF production	refrigeration 4 °C	glass bottle (100 g) + non-PVC plastic bottle or bag
[a] The maximum particle size (mm) is related to the laboratory sample amount (g) in order to guarantee sample homogeneity. It is established following the rules reported in EN 15413.				

Annex B
(informative)

Data on performance characteristics

The method described in this European Standard has been validated within the QUOVADIS project. In this validation project several SRF were selected to provide a reasonable coverage of SRF. They were tested according to the present standardised method. Results and robustness, repeatability and reproducibility data are reported in the document : "EUR 23552 EN - 2008 - Joint Research Centre – Institute for Environment and Sustainability - QUOVADIS Project - Organization of Validation Exercises - JRC Scientific and Technical Reports - ISBN 978-92-79-10396-4 - Luxembourg: Office for Official Publications of the European Communities".

Inter-laboratory trials were carried out by laboratories in Austria, Belgium, France, Germany, Italy, the Netherlands and the United Kingdom. The variety of instruments and other analytical conditions used conformed to the quality parameters specified in the method.

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

The data derive from laboratories participating in the above-mentioned inter-laboratory trials.

Table B.1 — Performance data for carbon

Sample	Matrix	l	n	o %	x_{ref} %	$\bar{\bar{x}}$ %	η %	s_R %	CV_R %	S_r %	CV_r %
A	SRF produced from shredded tyres	81	14	3,6	na	71,6	na	5,12	7,10	5,12	7,10
B	SRF produced from demolition wood	80	14	4,8	na	47,7	na	2,33	4,85	0,26	0,54
C	SRF produced from sewage sludge	84	14	0	na	30,5	na	0,43	1,43	0,43	1,43
D	SRF produced from municipal waste	84	14	0	na	46,4	na	2,23	4,85	1,63	3,54
E	SRF produced from municipal waste (paper and plastic reach)	84	14	0	na	45,4	na	2,97	6,60	1,71	3,80

Definition of symbols

l	is the number of outlier-free individual analytical values per level;
n	is the number of laboratories after outlier elimination;
o	is the percentage of outlying values from the replicate determination;
x_{ref}	is the accepted reference value on dry matter base;
$\bar{\bar{x}}$	is the overall mean on dry matter base;
η	is the recovery rate;
s_R	is the reproducibility standard deviation on dry matter base;
CV_R	is the coefficient of the variation of the reproducibility;
S_r	is the repeatability standard deviation on dry matter base;
CV_r	is the coefficient of the variation of the repeatability;
na	not applicable.

Table B.2 — Performance data for hydrogen

Sample	Matrix	l	n	o %	x_{ref} %	$\bar{x}$ %	η %	s_R %	CV_R %	S_r %	CV_r %
A	SRF produced from shredded tyres	63	12	12,5	na	6,83	na	0,91	13,4	0,30	4,40
B	SRF produced from demolition wood	66	13	15,4	na	6,14	na	0,73	12,0	0,07	1,10
C	SRF produced from sewage sludge	60	12	16,7	na	2,28	na	0,04	1,70	0,04	1,70
D	SRF produced from municipal waste	70	13	10,2	na	6,53	na	0,76	11,7	0,24	3,70
E	SRF produced from municipal waste (paper and plastic reach)	72	13	7,7	na	6,73	na	1,20	17,9	0,43	6,40
Definition of symbols: see Table B.1.											

Table B.3 — Performance data for nitrogen

Sample	Matrix	l	n	o %	x_{ref} %	$\bar{x}$ %	η %	s_R %	CV_R %	S_r %	CV_r %
A	SRF produced from shredded tyres	53	12	26,4	na	0,55	na	0,20	36,4	0,07	12,7
B	SRF produced from demolition wood	64	13	17,9	na	0,71	na	0,18	25,4	0,06	8,5
C	SRF produced from sewage sludge	58	12	19,4	na	1,02	na	0,02	2,0	0,02	2,0
D	SRF produced from municipal waste	62	13	20,5	na	1,83	na	0,12	6,6	0,12	6,6
E	SRF produced from municipal waste (paper and plastic reach)	58	13	25,6	na	0,95	na	0,14	14,7	0,06	6,3
Definition of symbols: see Table B.1.											

Annex C
(informative)

Major results of ruggedness testing

The design of the ruggedness testing was carried out by applying the analytical method to be validated with some controlled variations of analytical parameters (composition and different grain size: 0,5 mm, 1 mm and 1,5 mm) in repeatability conditions, in order to evaluate separately the influence of each varying parameter on the final results.

No significant grain size effect on repeatability are observed for C,H analysis: 1 mm grain size is adequate for most cases for the analysis of SRF samples of different origin, even if better results can be obtained in terms of precision by using larger amounts or lower grain size.

Bibliography

[1] EN 15442, *Solid recovered fuels — Methods for sampling*

[2] EN 15443, *Solid recovered fuels — Methods for the preparation of the laboratory sample*

[3] EN 15410[2)], *Solid recovered fuels — Method for the determination of the content of major elements (Al, Ca, Fe, K, Mg, Na, P, Si, Ti)*

[4] EN 15411[2)], *Solid recovered fuels — Methods for the determination of the content of trace elements (As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Ti, V and Zn)*

[5] CEN/TS 15412, *Solid recovered fuels — Methods for the determination of metallic aluminium*

[6] EN 15408, *Solid recovered fuels — Methods for the determination of sulphur (S), chlorine (Cl), fluorine (F) and bromine (Br) content*

[7] ISO 5725-5, *Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method*

[8] Quality Management, Organisation, Validation of Standards, Developments and Inquiries for SRF (http://quovadis.erse-web.it/)

[2)] To be published.

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