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Solid recovered fuels — Methods for the determination of carbon (C), hydrogen (H), nitrogen (N) and sulphur (S) by the instrumental method

Combustibles solides de récupération — Méthodes de détermination de la teneur en carbone (C), hydrogène (H), azote (N) et soufre (S) par la méthode instrumentale

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 300 *Solid recovered fuels*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The determination of total content of carbon, hydrogen, nitrogen and sulfur is usually performed using instrumental methods. Depending on the amount of test portion used two different types of instrumental methods can be used: micro methods require few milligrams of sample; macro methods use grams of sample. Micro methods require a very careful preparation of the test sample for Solid Recovered Fuel (SRF) analysis.

Solid recovered fuels — Methods for the determination of carbon (C), hydrogen (H), nitrogen (N) and sulphur (S) by the instrumental method

1 Scope

This document specifies the determination of total content of carbon, hydrogen, nitrogen and sulfur in solid recovered fuels by instrumental method. Depending on the amount of test portion, micro or macro instrumental apparatus are used.

This method is applicable for concentrations on dry matter basis of C > 0,1 %, N > 0,1 %, H > 0,1 % and S > 0,05 %.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 21637, *Solid recovered fuels — Terminology, definitions and descriptions*

ISO 21660-3[1], *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

ISO 21646[2], *Solid recovered fuels — Sample preparation*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 21637 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1
coefficient of variation
estimate of the standard deviation of a population from a *sample* (3.10) of n results divided by the mean of that sample

Note 1 to entry: Frequently stated as a percentage.

Note 2 to entry: Adapted from Eurachem/Citac Guide CG 4.

3.2
dry basis
calculation basis in which the material is considered free from *moisture* (3.6)

[SOURCE: ISO 21637, 3.20]

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[1] Under preparation. Stage at the time of publication ISO/FDIS 21660-3.

[2] Under preparation. Stage at the time of publication ISO/DIS 21646.

3.3
dry matter

material remaining after removal of *moisture* (3.6) under specific condition

[SOURCE: ISO 21637, 3.22]

3.4
general analysis sample

sub-sample of a laboratory sample having a *nominal top size* (3.7) of 1 mm or less and used for a number of chemical and physical analyses

3.5
laboratory sample

combined sample received by the laboratory on which sample preparation procedures are undertaken

Note 1 to entry: When the laboratory sample is further prepared (reduced) by subdividing, mixing, grinding, or by combinations of these operations, leading to a nominal top size ≤ 1mm, the result is the general analysis sample. A test portion is removed from the general analysis sample for the performance of the test or for analysis. When no preparation of the laboratory sample is required, the laboratory sample may become the test portion.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection, but it is the initial sample from the point of view of the laboratory.

Note 3 to entry: Several laboratory samples are prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

3.6
moisture

water removable under specific conditions

[SOURCE: ISO 21637, 3.46]

3.7
nominal top size

smallest aperture size of the sieve used for determining the particle size distribution of solid recovered fuels through which at least 95 % by mass of the total material passes through the sieve

[SOURCE: ISO 21637, 3.48]

3.8
particle size

size of the fuel particles as determined in a solid fuel

Note 1 to entry: Different methods of determination can give different results.

3.9
precision

closeness of agreement between independent test/measurement results obtained under stipulated conditions

[SOURCE: ISO 21637, 3.57]

3.10
sample

quantity of material, from a larger amount for which the quality is to be determined

Note 1 to entry: See also increment (3.39), stratified sample (3.78), stratified random sample (3.80), stratified arbitrary sample (3.79), sub-sample (3.82) and test sample (3.84) in ISO 21637.

[SOURCE: ISO 21637, 3.63, modified — Notes 1 - 3 to entry were removed and new note 1 to entry was added]

3.11

test portion

sub-sample either of a laboratory sample or a *test sample* (3.12) required for the specific measurement

[SOURCE: ISO 21637, 3.83]

3.12

test sample

laboratory sample (3.5) after an appropriate preparation made by the laboratory

[SOURCE: ISO 21637, 3.84]

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:

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

5 Principle

A known mass of sample is treated with oxygen, or in an oxygen/carrier gas mixture, under conditions such that it is converted into gaseous products of combustion or decomposition. These consist mainly of carbon dioxide, water vapour, elemental nitrogen and/or nitrogen oxides, oxyacids and oxides of sulfur and hydrogen halides. The products are treated to ensure that any hydrogen associated with sulfur or halides are converted, through a catalytic process, into water vapour. Nitrogen oxides are reduced to nitrogen, and the products of combustion which can interfere with the subsequent gas-analysis procedures are properly removed. The carbon dioxide, water vapour and nitrogen mass fractions in the gas stream are then determined quantitatively by appropriate instrumental gas analysis procedures after separation by trapping or suitable chromatographic column.

The samples are held in a suitable container (tin or other crucible) and then dropped inside the quartz tube furnace at about 1 250 °C in an oxygen stream for complete oxidation in the presence of a catalyst layer.

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 % volume fraction or other gases as specified by the instrument manufacturer.

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

6.3 Additional reagents: as specified by the instrument manufacturer.

6.4 Calibration standards

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

Examples of certified biomass sample with metrological traceability are given in Table 2.

The two tables refer to different types of materials: Table 1 refers to pure substances to be used primary calibration standards while Table 2 refers to biomass materials which can be used as controls since the content depends on the batch.

Table 1 — Calibration standards

Name	Formula	C %	H %	N %	S %
Acetanilide	C_8H_9NO	71,1	6,7	10,4	
Antipyrine	$C_{11}H_{12}N_2O$	70,19	6,43	14,88	
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	26,7
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	18,6
Sulfanilic acid	$C_6H_7NO_3S$	41,6	4,1	8,1	18,5
Thiourea	CH_4N_2S	15,78	5,3	36,8	42,12
TRIS	$C_4H_{11}NO_3$	39,7	9,1	11,6	
2,5-Bis(5-tert-butyl-benzoxazol-2-yl)thiophene (BBOT)	$C_{26}H_{26}N_2O_2S$	72,5	6,0	6,5	7,4

Table 2 — Examples of certified biomass sample with metrological traceability

Name	C %	H %	N %	S %
Alfalfa	45,05		3,6	0,38
Oat meal	45,8	6,7	2,8	0,25
Rye Flour	45	6,4	1,7	0,14
Wheat Flour	45,3	6,4	2,7	0,19

NOTE Certified value for biomass samples is only indicative, depending on the batch.

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, nitrogen and sulfur are converted to carbon dioxide, water vapour, nitrogen oxides or elemental nitrogen and sulfur oxides;

b) for the determination of sulfur, the instrumental configuration shall be able to reach adequate conditions of temperature and combustion time to allow to convert all the chemical compounds that can be present in the sample (e.g. metal sulphides require a temperature of 1 000 °C and calcium sulfate a temperature > 1250 °C);

c) Tungsten wire may not be used for the determination of sulfur because it is a sulfur trap. For instance, copper wire can be used as an alternative;

d) one or more separation steps are usually included to commercial devices, depending on the detector used, to reduce or eliminate any possible interference during the subsequent determination;

e) nitrogen oxides shall be converted to the nitrogen before detection;

f) hydrogen associated with sulfur or halides are converted, through a catalytic process, into water vapour