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Solid recovered fuels — Determination of ash content

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National foreword

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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 - Determination of ash content

Combustibles solides de récupération - Détermination de la teneur en cendre

Feste Sekundärbrennstoffe - Bestimmung des Aschegehaltes

This European Standard was approved by CEN on 22 January 2011.

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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 Apparatus **5**
6 Sampling and sample preparation **6**
7 Procedure **6**
8 Calculation **6**
9 Precision **7**
10 Test report **7**
Annex A (informative) **Interlaboratory test results** **8**
Bibliography **11**

Foreword

This document (EN 15403:2011) 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of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

This document supersedes CEN/TS 15403:2006.

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This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document differs from CEN/TS 15403:2006 mainly as follows:

a) use of automatic equipments under specific conditions permitted;

b) results of interlaboratory tests supplemented as an informative Annex A;

c) whole document editorially revised.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, 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

This European Standard covers the determination of ash content of solid recovered fuels. It is primarily geared toward laboratories, producers, suppliers and purchasers of solid recovered fuels but is also useful for the authorities and inspection organizations.

The method specified in this European Standard is based on CEN/TS 14775 as well as ISO 1171.

1 Scope

This European Standard specifies a method for the determination of ash content of all solid recovered fuels.

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

EN 15442, *Solid recovered fuels — Methods for sampling*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15357:2011 and the following apply.

3.1
ash content on dry basis
mass of inorganic residue remaining after ignition of a fuel under specified conditions, expressed as mass fraction in percent of the dry matter in the fuel

4 Principle

The sample is heated in air atmosphere up to a temperature of (550 ± 10) °C under rigidly controlled conditions of time, sample mass and equipment specifications. The ash content is determined by calculation from the mass of the residue remaining after heating.

5 Apparatus

5.1 **Dish**, consisting of inert material such as porcelain, silica or platinum, with a depth from 10 mm to 20 mm and such a size that the sample loading does not exceed 0,1 g/cm^2 bottom area.

5.2 **Furnace**, capable of maintaining a zone of uniform temperature at the levels required in Clause 7 and to reach these levels in the specified heating rates. The ventilation rate through the furnace should be such that no lack of oxygen arises during the heating procedure.

NOTE A ventilation rate from 5 air changes/min to 10 air changes/min should be suitable.

5.3 **Balance**, capable of weighing the dish containing the sample to the nearest 0,1 mg.

5.4 **Desiccator**, without desiccant.

NOTE The use of a desiccator without desiccant is specified in ISO 1171 and emphasised here since ashes from solid recovered fuels are often more hygroscopic than coal ashes.

5.5 Sieve

5.6 Container, sealed airtight.

6 Sampling and sample preparation

The general analysis sample shall be taken and prepared in accordance with EN 15442 and EN 15443. It shall be ground to pass through a sieve with an aperture size of ≤ 1 mm. The general analysis sample shall be received in the laboratory in the container (5.6). The general analysis sample shall either be oven-dried or its moisture content determined in accordance with EN 15414-3. The general analysis sample shall be mixed carefully before weighing (see also Clause 7).

7 Procedure

A minimum of two determinations shall be carried out on the general analysis sample.

Heat the empty dish (5.1) in the furnace (5.2) to (550 ± 10) °C for at least 60 min. Allow the dish to cool down in a desiccator. After the dish is cooled, weigh it to the nearest 0,1 mg and record the mass.

Place about 1 g of the general analysis sample on the bottom of the dish and spread in an even layer over the bottom surface. Weigh the dish plus the sample to the nearest 0,1 mg and record the mass. If the general analysis sample is oven-dried, both the dish and the sample shall be dried at (105 ± 10) °C as a precautionary measure and then weighed.

Place the loaded dish in the cold furnace. Heat the sample in the furnace according to the following heating routine:

a) raise the furnace temperature evenly to (250 ± 10) °C over a period of 50 min (i.e. a rise of 5 K/min). Maintain at this temperature level for 60 min to allow the volatiles to leave the sample before ignition;

b) continue to raise the furnace temperature evenly to (550 ± 10) °C over a period of 60 min (i.e. a rise of 5 K/min) and keep this temperature level for at least 120 min.

Remove the dish with its content from the furnace. Allow the dish and its content to cool on a thick metal plate for 5 min to 10 min and then transfer to a desiccator without desiccant and allow to cool to ambient temperature. Weigh the ash and the dish to the nearest 0,1 mg as soon as ambient temperature is reached and record the mass. Calculate the ash content of the sample as detailed in Clause 8. If there is any doubt of complete incineration (for instance presence of soot at visual inspection), then add droplets of water or ammonium nitrate to the sample before it is reloaded into the cold furnace and reheated to (550 ± 10) °C for a period of further 30 min until the change in mass is lower than 0,2 mg.

Automatic equipments may be used if the method is validated with biomass reference samples of an adequate biomass type. These equipments shall fulfill all the requirements given in this clause regarding sample size, heating procedure, atmosphere, temperatures and weighing accuracy. Deviations from this paragraph shall be reported and justified.

8 Calculation

The ash content on dry basis, A_{db}, of the general analysis sample, expressed as mass fraction in percent, shall be calculated by Equation (1):

$$A_{db} = \frac{(m_3 - m_1)}{(m_2 - m_1)} \times 100 \times \frac{100}{100 - M_{ad}} \quad (1)$$

where

m_1 is the mass of the empty dish, in grams;

m_2 is the mass of the dish plus the general analysis sample, in grams;

m_3 is the mass of the dish plus ash, in grams;

M_{ad} is the mass fraction of moisture of the general analysis sample on wet basis, in percent.

The result shall be reported as the mean of duplicate determinations to the nearest 0,1 %.

9 Precision

9.1 Repeatability limit

The maximum difference to be expected between two independent single test results of one laboratory at a confidence level of 95 % will not exceed the repeatability limit in more than 5 % of cases when measuring the same measurand in the same medium, using the same facilities and fulfilling all requirements of the test method (interlaboratory testing).

Precision data derived from the interlaboratory test are given in Annex A.

9.2 Reproducibility limit

The maximum difference to be expected between two independent single test results of different laboratories at a confidence level of 95 % will not exceed the reproducibility limit in more than 5 % of cases when measuring the same measurand in the same medium, each laboratory using their own facilities and fulfilling all requirements of the test method (interlaboratory testing)

Precision data derived from the interlaboratory test are given in Annex A.

10 Test report

The test report shall include the following information:

a) identification of the laboratory and the testing date;

b) identification of the sample tested;

c) a reference to this European Standard, i.e. EN 15403;

d) description of experimental set-up used;

e) test results and the basis which is reported on, e.g. „on dry basis“ or „on wet basis“;

f) any deviation from this European Standard;

g) any unusual features observed during the determination which may have affected the test result and details of any operations not included in this European Standard or regarded as optional.

Annex A
(informative)

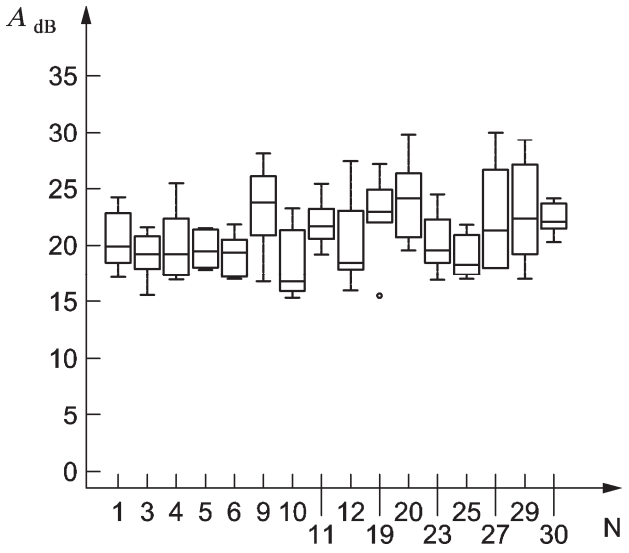
Interlaboratory test results

The statistic evaluation of the interlaboratory test results was carried out in accordance with ISO 5725-5. The precision data obtained are shown in Table A.1.

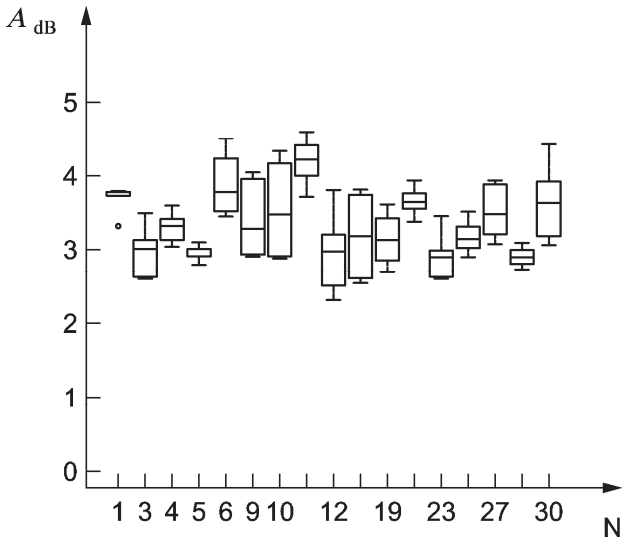
Table A.1 — Precision data

Designation	Shredded tyre	Demolition wood	Dried sludge	Municipal waste	Plastic/ paper fluff
Number of laboratories participating	16	16	16	16	16
Total number of values (without outliers)	96	102	90	96	102
Mean value, in % mass fraction	20,98	3,38	60,80	14,89	23,39
Laboratory effect, in % mass fraction	0,83	0,23	0,73	0,87	1,00
Sample effect, in % mass fraction	1,44	0,43	0,26	0,33	0,28
Repeatability standard deviation, s_r , in % mass fraction	3,04	0,24	0,27	0,76	1,51
Repeatability limit, r: ($r = 2{,}8 \times s_r$) in % mass fraction	8,51	0,67	0,76	2,13	4,23
Reproducibility standard deviation, s_R in % mass fraction	3,15	0,33	0,78	1,16	1,81
Reproducibility limit, R: ($R = 2{,}8 \times s_R$) in % mass fraction	8,82	0,92	2,18	3,25	5,07

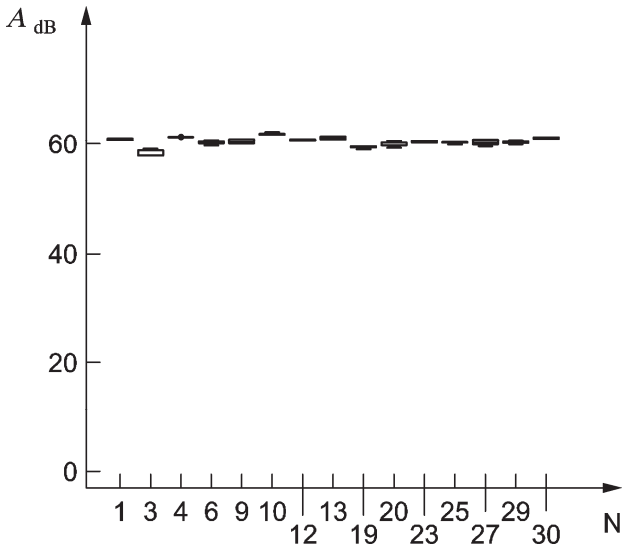
The deviations of the test results between the individual laboratories for each sample type are shown in Figures A.1 a) to A.1 e).



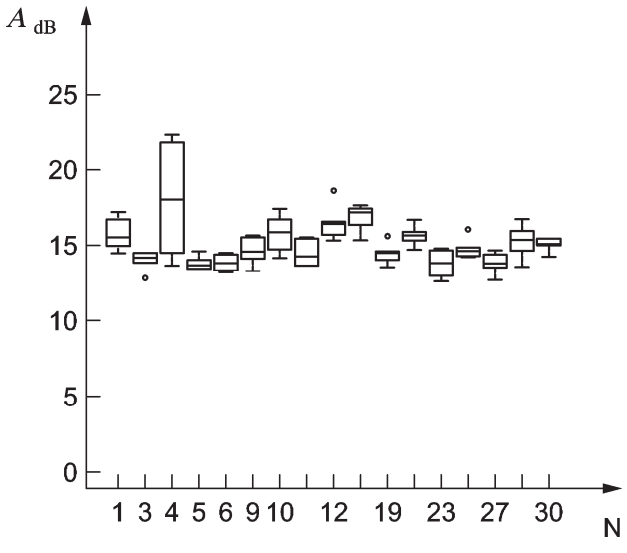
a) Shredded tyre



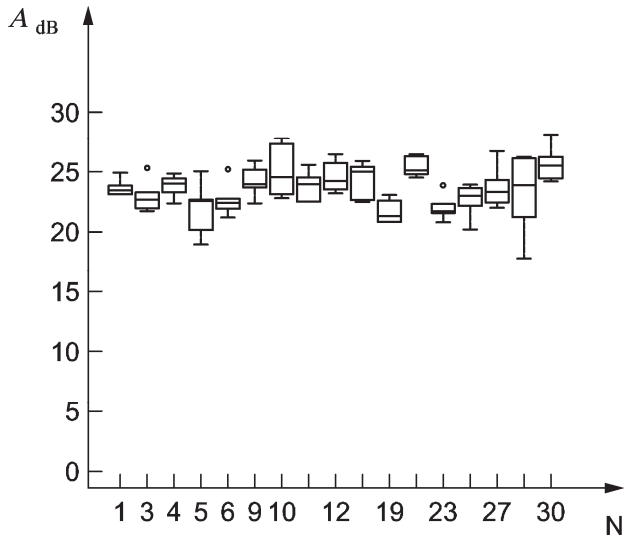
b) Demolition wood



c) Dried sludge



d) Municipal waste



e) Plastic and paper fluffs

Key

A_{db} mass fraction of ash in %
N number of the individual laboratory

Figure A.1 — Deviations of the test results between the individual laboratories

Bibliography

[1] EN 14775, *Solid biofuels — Determination of ash content*

[2] ISO 1171, *Solid mineral fuels — Determination of ash*

[3] 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*

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