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Solid recovered fuels — Determination of ash content

Combustibles solides de récupération — Détermination de la teneur en cendres

Reference number
ISO 21656:2021(E)

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Contents

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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This document was prepared by Technical Committee ISO/TC 300, *Solid recovered fuels*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 343, *Solid recovered fuels,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

This document 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 A specified in this document is based on EN 15403[5].

For information about environmental aspect, see Annex B.

Solid recovered fuels — Determination of ash content

1 Scope

This document specifies methods for the determination of ash content of all solid recovered fuels.

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 — Vocabulary*

ISO 21645, *Solid recovered fuels — Methods for sampling*

ISO 21646[1], *Solid recovered fuels — Sample preparation*

ISO 21660-3, *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 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
ash
ash content on dry basis
total ash
A
mass of inorganic residue remaining after combustion of a fuel under specified conditions, typically expressed as a percentage of the mass of dry matter in fuel

Note 1 to entry: Depending on the combustion efficiency the ash may contain combustibles.

Note 2 to entry: If a complete combustion is realized, ash contains only inorganic, non-combustible components.

[SOURCE: ISO 16559:2014, 4.13, modified — “Note 1 to entry” was removed and the following ones renumbered, and symbol "A" was italicized.]

3.2
total ash content
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, which also includes removed ash contributors

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[1] Under preparation. (Stage at the time of publication: ISO/DIS 21646:2021.)

3.3
removed ash contributors
rac
coarse inert material (i.e. metals, glass, stones, tiles etc.) removed from the pre-dried sample before preparation, in order to avoid damage to the preparation equipment

Note 1 to entry: Removed ash contributors are included in the total ash content calculations.

[SOURCE: ISO 21637:2020, 3.62, modified: Note 1 to entry was added]

3.4
total organic matter
combustible part of solid recovered fuels, which consists of the sum of volatile matter and fixed carbon

Note 1 to entry: It is calculated as: 100 - moisture content - ash content.

Note 2 to entry: It is the mass fraction of the matter lost by ignition, also known as “Loss of Ignition" (LOI).

3.5
volatile matter
relative part of the analysed sample, after moisture removal, that is lost when material is heated up under specific conditions of temperature, time and in a reduced atmosphere (anoxic conditions)

3.6
fixed carbon
relative part of carbon contained in a material that can only be degraded in oxic conditions and high temperature

Note 1 to entry: It is calculated as: 100 - moisture content - volatile matter content - ash content.

4 Principle

The sample is heated in air atmosphere up to a temperature of (550 ± 10) °C for Method A or (815 ± 10) °C for Method B 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.

NOTE Difference in the ash content if determined at 815 °C compared to 550 °C is explained by decomposition of carbonates forming CO_2, losses of volatile inorganic compounds (see also 3.5) and further oxidation of inorganic compounds[1]. In common standard practise, 550 °C is used for the determination of ash content in SRFs with a high content of biomass. 550 °C can also be used for major elemental determination (see also EN 15410[6]) and trace elemental determination (see also EN 15411[7]).

Automatic equipment (such as thermogravimetric analysers) may be used as long as the equipment is validated by parallel measurements to the reference method. The automatic equipment shall fulfil all the requirements regarding sample size, heating procedure, temperature, atmosphere and weighing accuracy. Deviations from this paragraph shall be reported and justified.

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 are 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, with an aperture size of ≤1 mm (according ISO 3310-1 or ISO 3310-2).

5.6 Container, sealed airtight.

6 Sampling and sample preparation

6.1 General

The general analysis sample shall be taken and prepared in accordance with ISO 21645 and ISO 21646. 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 container (5.6). The general analysis sample shall either be oven-dried or its moisture content determined in accordance with ISO 21660-3. The general analysis sample shall be mixed carefully before weighing (see also Clause 7).

6.2 Pre-drying

Pre-drying shall be performed according to ISO 21646.

Pre-drying of wet samples is carried out to minimize moisture loss in the subsequent sample-division processes, to facilitate the sample preparation processes, and to minimize biological activity. If it is necessary to dry a sample by heating, it shall be dried in an oven at a temperature not exceeding the temperature according ISO 21646.

6.3 Removed ash contributors (rac)

Coarse inert material (i.e. metals, glass, stones) can be removed from the pre-dried sample before preparation, in order to avoid damage to the preparation equipment.

Removed ash contributors (rac) from pre dried samples shall be considered as dry.

A_{rac} is weighed separately, calculated as mass percent and added to the determined ash content according to calculation in 8.2.

7 Procedure

7.1 General

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

7.2 Method A – ash content at 550 °C

For the determination of the biomass fraction, use the temperature of 550 °C for all cases.

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 against water absorption 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 °C/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 °C/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 equipment may be used if the method is validated with biomass reference samples of an adequate biomass type. This equipment shall fulfil 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.

7.3 Method B – ash content at 815 °C

Heat the empty dish (5.1) in the furnace (5.2) to (815 ± 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 against water absorption 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 °C/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 (815 ± 10) °C over a period of 60 min (i.e. a rise of 15 °C/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 (815 ± 10) °C for a period of further 30 min until the change in mass is lower than 0,2 mg.

Automatic equipment may be used if the method is validated with biomass reference samples of an adequate biomass type. This equipment shall fulfil 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

8.1 General analysis sample

The ash content on wet basis, A_{ad}, of the general analysis sample "as analysed", expressed as mass fraction in percent, shall be given by Formula (1):

$$A_{ad} = \frac{m_3 - m_1}{m_2 - m_1} \cdot 100 \tag{1}$$

The ash content on dry basis, A_{db}, of the general analysis sample, expressed as mass fraction in percent, shall be calculated by Formula (2):

$$A_{db} = \frac{m_3 - m_1}{m_2 - m_1} \cdot 100 \cdot \frac{100}{100 - M_{ad}} = A_{ad} \cdot \frac{100}{100 - M_{ad}} \tag{2}$$

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 %.

8.2 Calculation of total ash content including removed ash contributors, on an as received and dry basis

The total ash content on an as received basis, $A_{total,ar}$, of the sample, expressed as mass fraction in percent, shall be calculated by Formula (3):

$$A_{total,ar} = A_{ad} \cdot \left(1 - \frac{M_p}{100} - \frac{A_{rac,ar}}{100}\right) + A_{rac,ar} \tag{3}$$

where $A_{rac,\,ar}$ is according to Formula (4):

$$A_{rac,ar} = \frac{m_{rac}}{m_{ar}} \cdot 100 \tag{4}$$

The total ash content on a dry basis, $A_{total,\,db}$, of the sample, expressed as mass fraction in percent, shall be calculated by Formula (5):

$$A_{total,db} = A_{total,ar} \cdot \frac{100}{100 - M_T} \tag{5}$$

where the mass fraction of moisture content on an as received basis, M_{ar}, corresponding to M_T, in percent, is according to Formula (6):

$$M_T = M_p + M_{ad} \cdot \left(1 - \frac{M_p}{100} - \frac{A_{rac,ar}}{100}\right) \tag{6}$$

where

A_{ad}	is the ash content of the general analysis sample, in mass percent on wet basis, as analysed;
$A_{rac,ar}$	is the ash content of removed ash contributors after pre-drying on an as received basis, in mass percent;
M_{ad}	is the mass fraction of moisture loss in the sample caused by second drying (until constant mass after pre-drying) of the (general) analysis sample (see definition in ISO 21646), in percent;
M_p	is the mass fraction of moisture loss in the sample caused by pre-drying of the as the received sample, in percent;
M_T	is the total mass fraction of moisture in the as received sample, in percent;
m_{ar}	is the mass of the sample as received (on wet basis), in grams;
m_{rac}	is the mass of the removed ash contributors after pre-drying step, in grams.

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 in Europe from 2008 (for method A) and for Method B obtained during international comparison studies in 2013, 2014 and 2015 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 in Europe from 2008 (for method A) and for Method B obtained during international comparison studies in 2013, 2014 and 2015 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 document, i.e. ISO 21656:2021;

d) description of method or temperature used (Method A or Method B);

e) test results and the basis which is reported on, e.g. "on dry basis“ or "as received basis“ (see Clause 8);

f) any deviation from this document;

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

h) content of removed ash contributors, in mass percent.

Annex A
(informative)

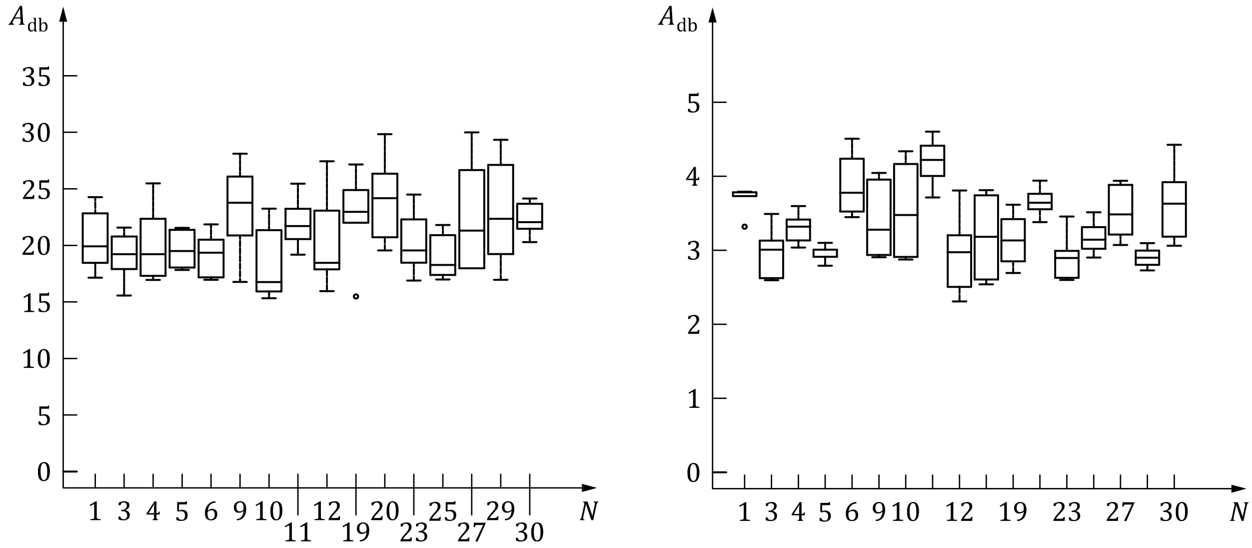
Interlaboratory test results

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 for Method A

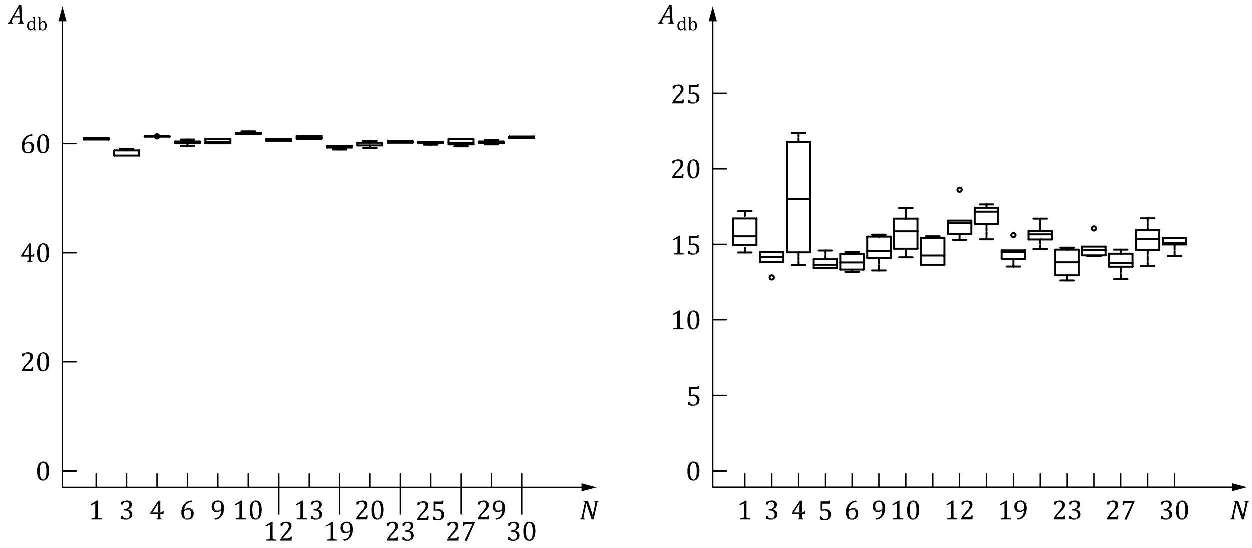
Designation	Shredded tyre	Demolition wood	Dried sludge	Municipal waste	Plastic/ paper fluff
Number of laboratories participating	16	17	15	17	17
Total number of values (without outliers)	96	102	90	96	102
Mean value, in % mass fraction	20,98	3,38	60,80	14,89	23,39
Laboratory effect, in % mass fraction	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 e).



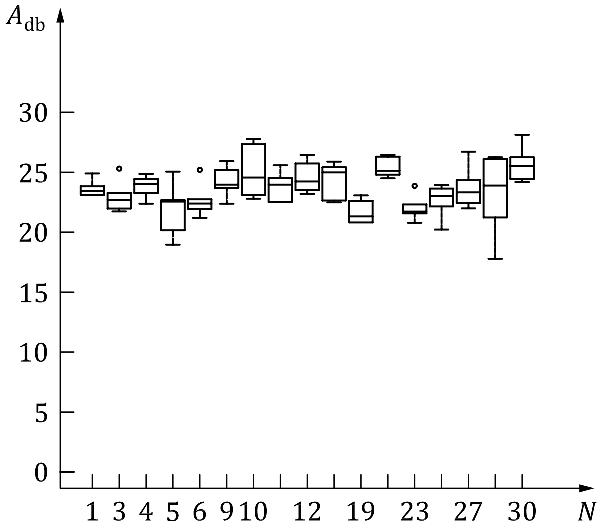
a) **Shredded tyre**

b) **Demolition wood**



c) **Dried sludge**

d) **Municipal waste**



e) Plastic and paper fluffs

Key

N number of the individual laboratory

A_{db} ash content on dry basis, in %

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

The precision data for Method B obtained during international comparison studies in 2013, 2014 and 2015 are shown in Table A.2 (see also Reference [8]).

Table A.2 — Precision data for Method B

Designation	Plastic I (grinded <0,5 mm)	Plastic II (granulated)	Demolition wood (<1 mm)
Number of laboratories	8	7	11
Mean value, in % mass fraction	3,02	5,23	1,38
Repeatability variance, s_r^2	0,033	0,003 7	0,006 4
Repeatability standard deviation, s_r, in % mass fraction	0,18	0,06	0,08
Variance between laboratories, s_L^2	0,117	0,001	0,026
Standard deviation between laboratories, s_L, in % mass fraction	0,34	0,03	0,16
Reproducibility variance, s_R^2	0,15	0,005	0,032
Reproducibility standard deviation, s_R, in % mass fraction	0,39	0,07	0,18
Repeatability limit, r: ($r = 2{,}8 \times s_r$) in % mass fraction	0,51	0,17	0,22
Reproducibility limit, R: ($R = 2{,}8 \times s_r$) in % mass fraction	1,08	0,19	0,50

Annex B
(informative)

Environmental aspects

For environmental aspects, the solid recovered fuels supply chain relies on training and trained operatives competent to carry out their work. Each aspect of the operations has varying degrees of competency within the scope of the operations.

Many of the functions carried out by operatives within the solid recovered fuels supply chain require specialists to carry out the work, such as trained laboratory technicians and mechanical handling machine operatives.

The accessibility requirements for each of the functions will be identified and reviewed by the site owners and their management, within the boundaries of Health and Safety at Work principles, best practice and legislation and regulation.

The standards in and of themselves do not specify the accessibility requirements for any function of the operations.

The environmental checklist (see Table B.1) indicates possible environmental aspects according to the analysis of solid recovered fuels to take into account.

Users may reproduce the form in Annex B without prejudice to the rights of ISO regarding the entire document.

Table B.1 — Environmental checklist

Document number (if available):			Title of standard: Solid recovered fuels — Determination of …					TC/SC/WG number: ISO/TC 300/ WG 4			
Work item number (if available):			Version of the environmental checklist:					Date of last modification of the environmental checklist:			
Environmental Issue	**Stages of the life cycle**										**All stages**
	Acquisition		**Production**		**Use**			**End-of-Life**			
	Raw materials and energy	**Pre-manufactured materials & components**	**Production**	**Packaging**	**Use**	**Maintenance and repair**	**Use of additional products**	**Reuse/ Material and Energy Recovery**	**Incineration without energy recovery**	**Final disposal**	**Transportation**
Inputs											
Materials							X				
Water					X						
Energy		X	X			X					
Land											
Outputs											

NOTE 1 The stage of packaging refers to the primary packaging of the manufactured product. Secondary or tertiary packaging for transportation, occurring at some or all stages of the life cycle, is included in the stage of transportation.

NOTE 2 Transportation can be dealt with as being a part of all stages (see checklist) or as separate sub-stage. To accommodate specific issues relating to product transportation and packaging, new columns can be included and/or comments can be added.

Table B.1 *(continued)*

Document number (if available):			Title of standard: Solid recovered fuels — Determination of ...					TC/SC/WG number: ISO/TC 300/ WG 4				
Work item number (if available):			Version of the environmental checklist:					Date of last modification of the environmental checklist:				
Environmental Issue	**Stages of the life cycle**											**All stages**
	Acquisition		**Production**		**Use**			**End-of-Life**				
	Raw materials and energy	**Pre-manufactured materials & components**	**Production**	**Packaging**	**Use**	**Maintenance and repair**	**Use of additional products**	**Reuse/ Material and Energy Recovery**	**Incineration without energy recovery**	**Final disposal**		**Transportation**
Emissions to air			X		X		X	X				
Discharges to water												
Discharges to soil										X		
Waste			X		X		X					
Noise, vibration, radiation, heat			X		X							
Other relevant aspects												
Risk to the environment from accidents or unintended use			X		X			X	X	X		
Customer information												
Comments:												
NOTE 1 The stage of packaging refers to the primary packaging of the manufactured product. Secondary or tertiary packaging for transportation, occurring at some or all stages of the life cycle, is included in the stage of transportation. NOTE 2 Transportation can be dealt with as being a part of all stages (see checklist) or as separate sub-stage. To accommodate specific issues relating to product transportation and packaging, new columns can be included and/or comments can be added.												

Bibliography

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

[2] ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

[3] ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

[4] 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*

[5] EN 15403, *Solid biofuels — Determination of ash content*

[6] EN 15410, *Solid recovered fuels - Methods for the determination of the content of major elements (Al, Ca, Fe, K, Mg, Na, P, Si, Ti)*

[7] EN 15411, *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, Tl, V and Zn)*

[8] For more information regarding the Reports for Plastic I from 2013, for Demolition wood from 2014 and for Plastic II from 2015, see http://www.bioenergy.co.at/

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