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Solid recovered fuels — Determination of the recycling index for co-processing

FDIS stage

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Contents

Foreword

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

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Introduction

When solid recovered fuels (SRFs) are co-processed mainly in the cement industry, simultaneous energy recovery and recycling of mineral components of waste material takes place because the ash is directly incorporated into the clinker. SRF co-processing therefore allows for the replacement of both mineral resources and fossil fuels.

SRF ashes contain various chemical components that are crucial raw materials for cement manufacturers, fulfil specific tasks in cement clinker production or represent clinker phases giving the clinker its specific properties. For example, a major part of SRF ashes from mixed municipal and commercial waste consists of the four main chemical components that are required for cement clinker production: Al_2O_3, CaO, Fe_2O_3 and SiO_2 (see Annex A).Annex A). Additionally, minor ash constituents include MgO and TiO_2, both of which are present in or as clinker phases. K_2O and Na_2O are typical constituents of feldspars that are present in the clay used as a raw material for the process. SO_3, which is also present in SRF ash, or another sulfate carrier is needed in order to convert these alkali oxides into alkali sulfates, a clinker phase that alters the clinker's chemical reactivity with water.

Solid recovered fuels — Determination of the recycling index for co-processing

1 Scope

This document specifies the determination of the share of material recovery in the case of energy recovery (i.e. co-processing) of solid recovered fuels (SRFs), for example, in a cement kiln. This share, called the recycling index (R-index), is calculated on the basis of the ash content and the ash composition.

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/DIS 3884:—[1], *Solid recovered fuels — Determination of elemental content (Al, Ca, Fe, K, Mg, Na, P, Si, Ti, As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Tl, V an Zn)*

ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

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

ISO 21646, *Solid recovered fuels — Sample preparation*

ISO 21656:2021, *Solid recovered fuels — Determination of ash content*

ISO/DIS 3884: —[2] *Solid recovered fuels – Determination of elemental content (Al, Ca, Fe, K, Mg, Na, P, Si, Ti, As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Tl, V an Zn)*

ISO 22940, *Solid recovered fuels — Determination of elemental composition by X-ray fluorescence*

EN 15309, *Characterization of waste and soil. Determination of elemental composition by X-ray fluorescence*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obphttps://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/https://www.electropedia.org/

[1] Under preparation. Stage at the time of publication: ISO/DIS 3884:2024.

[2] Under preparation. Stage at the time of publication: ISO/DIS 3884:2023.

3.1
recycling index
R-index
share of SRF that can be considered as recycled on a material level, expressed as a mass fraction in per cent of the dry matter

3.2
co-processing
use of SRF in manufacturing processes for energy recovery and simultaneously for material recovery of mineral components

4 Symbols and abbreviated terms

A_{db}	ash content at 815 °C on a dry basis
Al_2O_3	aluminium(III) oxide
CaO	calcium oxide
(*d*)	dry (dry basis)
Fe_2O_3	iron(III) oxide
K_2O	potassium oxide
MgO	magnesium oxide
Na_2O	sodium oxide
SiO_2	silicon dioxide
SO_3	sulfur trioxide
TiO_2	titanium dioxide

5 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 **Water**, e.g. deionized (< 0,055 µS/cm).

5.2 **Nitric acid (HNO_3)**, approximately 15 mol/l, mass fraction of 65 % to 70 %.

5.3 **Hydrofluoric acid (HF)**, approximately 23 mol/l, mass fraction of 40 % to 45 %.

5.4 **Hydrochloric acid (HCl)**, approximately 12 mol/l, mass fraction of 35 % to 37 %.

5.5 **Lithium metaborate ($LiBO_2$)**, solid.

5.6 **Binder**, solid or liquid, for example wax. Specifications are given in EN 15309.

6 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

6.1 **Analytical balance**, with an accuracy of 1 mg or better.

6.2 Muffle furnace, for temperatures of 1 050 °C.

6.3 Inductively coupled plasma, normal commercial instrumentation with optical or mass spectrometric detector (ICP-OES, ICP-MS).

6.4 X-ray fluorescence spectrometer, an energy or wavelength dispersion system suitable for qualitative and (semi-)quantitative analysis of the elements listed in this document.

6.5 Microwave unit, in accordance with ISO/DIS 3884:—.

6.6 Press, in accordance with ISO 22940 or EN 15309.

6.7 Platinum crucible, e.g. Pt/Au5 %.

6.8 Inert bowl, for example made from porcelain, silicon dioxide or platinum with a depth of 10 mm to 20 mm and with a size selected in a way that the occupancy of the bottom area does not exceed 0,1 g/cm^2.

6.9 Magnetic stirrer, with heating function and PTFE (polytetrafluoroethylene) stirring bone.

6.10 Volumetric flasks, e.g. 250 ml.

7 Procedure

7.1 Preparation of the test sample

The sampling of SRF shall be carried out ~~according to~~in accordance with ISO 21645 and the sample preparation ~~according to~~in accordance with ISO 21646, with the final sample having a nominal top size of 1,0 mm or less. Hard impurities, i.e. inert materials or metals, that cannot be reduced in size with the apparatuses defined in ISO 21646 are sorted out during sample preparation and are not considered in the subsequent analysis steps. Their amount shall be documented. The sample is dried at 105 °C according to ISO 21660-3. Further procedures and analyses are carried out with the dried sample <1,0 mm free of hard impurities.

7.2 Determination of the ash content and preparation of ash sample

The determination of the ash content shall be carried out ~~according to method B of~~in accordance with ISO 21656:2021, method B. If the determination provides a sufficient amount of material to proceed with the analyses, the ashed material shall be used for subsequent analyses. If the determination of the ash content provides insufficient amounts of ash for the subsequent analysis steps (examples of ash content of typical constituents of SRF from mixed municipal and commercial waste are given in ~~Annex A),~~Annex A), the sufficient amount of ash shall be reached by using one or both of the following two approaches:

a) ~~a)~~ The ashing process is repeated and the ashes are collected and united in order to reach the minimum amounts required for subsequent ash analyses. This may be the preferable approach for method A (wet digestion/ICP-MS, requiring approximately 200 mg of ash sample) and method B (fused beads/ICP-OES, requiring approximately 100 mg of ash sample).

b) ~~b)~~ A correspondingly larger inert bowl is used and the amount of sample is increased accordingly, which may be the preferable option for pellet preparation and XRF analyses (method C, approximately 4,5 g to 10 g is required). The bowl shall fulfil the requirements defined by ISO 21656 and its size shall be selected in such a way that the occupancy of the bottom area does not exceed 0,1 g/cm^2.

7.3 Determination of the elemental content

The determination of the elemental content shall be carried out according to ISO/DIS 3884:— or ISO 22940. The element content shall be determined in the ashed sample.

7.4 Methods

7.4.1 Method A – wet digestion followed by ICP-MS or ICP-OES analysis

The ash shall be digested using microwave-assisted acid digestion with hydrochloric acid, nitric acid and hydrofluoric acid and analysed by ICP-MS or ICP-OES as described in ISO/DIS 3884:—.

7.4.2 Method B – preparation of fused beads followed by ICP-OES analysis

100 mg (±20 mg) of the ashed sample shall be thoroughly mixed with 1 000 mg (±10 mg) of the fluxing agent lithium metaborate in a platinum crucible. The mixture shall be melted in a muffle furnace at 1 050 °C ± 10 °C for 20 min. The resulting fused bead is allowed to cool down and dissolved incrementally by adding 80 ml of hydrochloric acid (c = 2 mol/l) into the crucible in small amounts. The dissolution shall be supported by heating (to approximately 60 °C) and stirring with a PTFE stirring bone. The digest solution shall be filled to a final volume of 250 mL with deionised water (< 0,055 µS/cm). This solution shall contain 0,4 g/l of the sample and 4 g/l of the fluxing agent. Only clear solutions shall be subjected to subsequent analyses, turbid digest solutions are discarded. A blank shall be prepared using the same method, but no sample is added. The element content in the digestion solutions shall be determined by ICP-OES ~~according to~~in accordance with ISO 11885.

7.4.3 Method C – preparation of pellets followed by XRF analysis

The pellet preparation and XRF analyses shall be performed ~~according to~~in accordance with ISO 22940 or EN 15309. The fine ash sample shall be mixed and homogenized with binder at a defined ratio (different ratios can be applied and the dilution factor needs to be considered; a typical sample to wax mass ratio is 10:1) and shall be pressed with an automatic or manual press. For a pellet with a diameter of 40 mm, about 10,0 g of the ash sample is required. For a pellet with a diameter of 32 mm, approximately 4,5 g ash sample is required.

7.4.4 Alternative procedures

Alternative methods can be applied if their performance is proved to be comparable with those listed in the preceding subclauses.

7.5 Calculation

7.5.1 Calculation of element oxides

The results for the concentrations of chemical elements need to be converted into concentrations of element oxides. The conversion is performed by multiplying the concentration of the respective element, expressed as mg/kg *(d)*, with the corresponding conversion factor~~:~~ as shown in Formula (1):

~~$c_{i_2} = CF_i * c_{i_1}$~~ ~~(1)~~

$$c_{i\ 2} = F_{ci} * c_{i\ 1} \quad (1)$$

where

~~c_{i1}~~ $c_{i\ 1}$ is the concentration of a selected element expressed as mg/kg *(d)*;

~~c_{i2}~~ $c_{i\ 2}$ is the concentration of the corresponding element oxide expressed as mg/kg *(d)*, see ~~Table 1;~~Table 1;

~~CF_i~~ F_{ci} is the corresponding conversion factor as listed in ~~Table 1.~~Table 1.

Table 1 — Conversion factors

Element	Element oxide	Conversion factor ~~CF~~F_c
Al	Al_2O_3	1,889 4
Ca	CaO	1,399 2
Fe	Fe_2O_3	1,429 7
K	K_2O	1,204 6
Mg	MgO	1,658 3
Na	Na_2O	1,348 0
S	SO_3	2,496 9
Si	SiO_2	2,139 3
Ti	TiO_2	1,668 5

~~7.5.3~~7.5.2 Calculation of R-index for co-processing

The share of the SRF that is recycled on a material level (i.e. R-index), expressed as mass fraction in per cent of the dry matter [% (*d*)], can either be calculated considering the four main chemical compounds required for the production of cement clinker (R_{i4}) or considering additional elements that are introduced by natural raw materials and are part of clinker phases as well (R_{i9}). The R-indices shall be calculated as follows:

$$R_{i4} = \frac{A_{db}}{100} * \frac{c_{Al_2O_3} + c_{CaO} + c_{Fe_2O_3} + c_{SiO_2}}{10\,000} \quad \frac{A_{db}}{100} * \frac{c_{Al_2O_3} + c_{CaO} + c_{Fe_2O_3} + c_{SiO_2}}{10\,000} \tag{2}$$

$$R_{i9} = \frac{A_{db}}{100} * \frac{c_{Al_2O_3} + c_{CaO} + c_{Fe_2O_3} + c_{K_2O} + c_{MgO} + c_{Na_2O} + c_{SO_3} + c_{SiO_2} + c_{TiO_2}}{10\,000} \quad R_{i9} = \frac{A_{db}}{100} * \frac{c_{Al_2O_3} + c_{CaO} + c_{Fe_2O_3} + c_{K_2O} + c_{MgO} + c_{Na_2O} + c_{SO_3} + c_{SiO_2} + c_{TiO_2}}{10\,000} \tag{3}$$

where

A_{db} is the ash content of the general analysis sample at 815 °C on a dry basis, expressed as mass fraction in per cent of the dry matter;

c_x is the concentration of element oxides expressed as mg/kg (*d*).

8 Performance characteristics

Data for repeatability and reproducibility for predefined solid recovered fuel samples (paper fibre sludge, high calorific waste fraction from non-hazardous municipal solid waste) are shown in ~~Annex B.~~Annex B.

9 Test report

The test report shall contain at least the following information:

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

b) b) description and identification of the laboratory sample;

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

d) d) a reference to this document, i.e. ISO 4349: —:2024;

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

f) f) the analytical results for R-index$_4$ and/or R-index$_9$, referring to % (*d*);

g) g) amount of hard impurities, referring to % (*d*);

h) h) any details not specified in this document or which are optional, and any other factors which couldcan have affected the results;

i) i) 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 (raw data and calculation details) that should be kept available in case of specific requirements.