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Solid recovered fuels — Methods for the determination of the content of elements (Al, Ca, Fe, K, Mg, Na, P, S, Si, Ti, As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Sn, Tl, V, Zn)

Combustibles solides de récupération — Méthodes de détermination de la teneur en éléments (Al, Ca, Fe, K, Mg, Na, P, S, Si, Ti, As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Sn, Tl, V, Zn)

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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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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

Accurate determination of the element content in solid recovered fuels is important for environmental and technical reasons both in the production and combustion stage. Some of the elements determined by the application of one of the methods in this document are included in ISO 21640 [1] while other elements can have environmental implications both for emissions and for the bottom and fly ashes disposal or recovery. Furthermore, the determination of elements such as Al, Ca, Fe, Mg, P, K, Si, Na and Ti can be helpful to predict the melting behaviour and slagging of the ash.

The methods described in this document provide multi-element digestions for a wide range of solid recovered fuels. The elements that are extractable and determined by these procedures can in many instances be described as 'total element contents', although this will be matrix dependent. After digestion, a number of analytical techniques can be used for the accurate determination of major and minor element contents, e.g. inductively coupled plasma with optical or mass detection (ICP-OES, ICP-MS), graphite furnace atomic absorption spectrometry (GF-AAS) and specific direct methods (e.g. for mercury, sulfur).

Alternatively, X-ray fluorescence can be used as a fast method for a qualitative overview of ash forming elements and impurities of solid recovered fuels. After suitable calibration, X-ray fluorescence is very useful for determining major elements or even minor elements (except mercury and beryllium) in solid recovered fuels according to ISO 22940. For calibration of X-ray fluorescence, it is important to use several solid recovered fuel reference materials or solid recovered fuel samples that were carefully characterized after total digestion and measurement by ICP-OES, ICP-MS, GF-AAS or by other techniques such as elemental analysis using combustion technology on sulfur (see ISO 21663 [2]).

After ashing of solid recovered fuels, X-ray fluorescence allows the simultaneous determination of major elements (Al, Ca, Fe, Mg, P, K, Si, Na, Ti) in the ashes after matrix-based calibration (procedures for this are described in ISO 22940 and EN 15309).

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1 Scope

This document specifies methods for the determination of major and minor element concentrations in solid recovered fuels after digestion by the use of different acid mixtures and by addition of a fluxing agent for solid recovered fuel (SRF) ash.

a) Method A: Microwave assisted digestion with hydrochloric, nitric and hydrofluoric acid mixture (6 ml HCl; 2 ml HNO_3; 2 ml HF) followed by boric acid complexation;

b) Method AT: Microwave assisted digestion with hydrochloric, nitric and tetrafluoroboric acid mixture (6 ml HCl; 2 ml HNO_3; 4 ml HBF_4);

c) Method B: Microwave assisted digestion with hydrochloric, nitric and hydrofluoric acid mixture (0,5 ml HCl; 6 ml HNO_3; 1 ml HF) followed by boric acid complexation;

d) Method BT: Microwave assisted digestion with hydrochloric, nitric and tetrafluoroboric acid mixture (0,5 ml HCl; 6 ml HNO_3; 2 ml HBF_4);

e) Method C: Microwave assisted digestion with nitric acid, hydrogen peroxide and hydrofluoric acid mixture (2,5 ml H_2O_2; 5 ml HNO_3; 0,4 ml HF) and optional boric acid complexation;

f) Method CT: Microwave assisted digestion with nitric acid, hydrogen peroxide and tetrafluoroboric acid mixture (2,5 ml H_2O_2; 5 ml HNO_3; 0,8 ml HBF_4);

g) Method D: Digestion of the ashed SRF sample with fluxing agent lithium metaborate in an oven at 1 050 °C.

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This document is applicable for the following major and minor/trace elements:

— Major elements: aluminium (Al), calcium (Ca), iron (Fe), potassium (K), magnesium (Mg), sodium (Na), phosphorus (P), sulfur (S), silicon (Si) and titanium (Ti).

— Minor/trace elements: arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), mercury (Hg), molybdenum (Mo), manganese (Mn), nickel (Ni), lead (Pb), antimony (Sb), selenium (Se), tin (Sn), thallium (Tl), vanadium (V) and zinc (Zn).

Method A is applicable for general use for SRF and ashed SRFs, but the amount of the test portion can be very low in case of high concentration of organic matter. Method AT can be used if an alternative to HF is necessary.

Method B with a higher volume of nitric acid is applicable for SRFs with high organic matter (e.g. suitable for high plastic content) that can be difficult to digest with less nitric acid or as a substitute for method A if appropriate equipment is not available. Method BT can be used if an alternative to HF is necessary.

Method C with combination of nitric acid and hydrogen peroxide and addition of hydrofluoric acid is applicable for wood based SRFs (e.g. demolition wood) or when there is a need for comparability to solid biofuel standards. Method CT can be used if an alternative to HF is necessary.

Method D is specifically applicable for determination of major elements in ashed SRF samples.

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