



BSI Standards Publication

**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)**

National foreword

This British Standard is the UK implementation of EN ISO 3884:2025. It is identical to ISO 3884:2025. It supersedes BS EN 15410:2011 and BS EN 15411:2011, which are withdrawn.

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 committee manager.

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English Version

**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) (ISO 3884:2025)**

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) (ISO 3884:2025)

Feste Sekundärbrennstoffe - Verfahren zur
Bestimmung des Gehaltes an Elementen (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) (ISO 3884:2025)

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European foreword

This document (EN ISO 3884:2025) has been prepared by Technical Committee ISO/TC 300 " Solid recovered materials, including solid recovered fuels " in collaboration with Technical Committee CEN/TC 343 "Solid recovered materials, including 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of an identical text or by endorsement, at the latest by October 2025, and conflicting national standards shall be withdrawn at the latest by October 2025.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 15410:2011 and EN 15411:2011.

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Endorsement notice

The text of ISO 3884:2025 has been approved by CEN as EN ISO 3884:2025 without any modification.

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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 document 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 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).

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)

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 HNO₃; 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 HNO₃; 4 ml HBF₄);
- c) Method B: Microwave assisted digestion with hydrochloric, nitric and hydrofluoric acid mixture (0,5 ml HCl; 6 ml HNO₃; 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 HNO₃; 2 ml HBF₄);
- e) Method C: Microwave assisted digestion with nitric acid, hydrogen peroxide and hydrofluoric acid mixture (2,5 ml H₂O₂; 5 ml HNO₃; 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 H₂O₂; 5 ml HNO₃; 0,8 ml HBF₄);
- g) Method D: Digestion of the ashed SRF sample with fluxing agent lithium metaborate in an oven at 1 050 °C.

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.

XRF can be used for the analysis of major elements (Al, Ca, Fe, K, Mg, Na, P, S, Si, Ti) after ashing (815 °C) of the samples and several major and minor/trace elements in SRF can be analysed by XRF after suitable calibration provided that the concentration levels are above instrumental detection limits of the XRF instrumentation and after proper preliminary testing and validation.

Digestion methods with HF and subsequent boric acid complexation or application of method D are applicable for determination of Si and Ti (better digestion efficiency).

Alternative digestion methods can be applied, if their performance is proved to be comparable with those of the methods described in this document.

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

ISO 22940:2021, *Solid recovered fuels — Determination of elemental composition by X-ray fluorescence*

EN 15309:2007, *Characterization of waste and soil — Determination of elemental composition by X-ray fluorescence*

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 terminology databases for use in standardization at the following addresses:

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

3.1 digestion

mineralization of the organic matter of a sample and dissolution of its mineral part, more or less completely, when reacted with a reagent mixture

3.2 microwave unit

whole microwave digestion system (oven and associated equipment)

4 Symbols and abbreviations

A_{db}	ash content at 815 °C on a dry basis (A_DB)
Al	aluminium (AL)
Al_{ash}	aluminium in ashed SRF (ALASH)
Ca	calcium (CA)
Ca_{ash}	calcium in ashed SRF (CAASH)
DM	dry mass (DM)

Fe	iron (FE)
Fe _{ash}	iron in ashed SRF (FEASH)
K	potassium (K)
K _{ash}	potassium in ashed SRF (KASH)
Mg	magnesium (MG)
Mg _{ash}	magnesium in ashed SRF (MGASH)
Na	sodium (NA)
Na _{ash}	sodium in ashed SRF (NAASH)
P	phosphorus (P)
P _{ash}	phosphorus in ashed SRF (PASH)
S	sulfur (S)
S _{ash}	sulfur in ashed SRF (SASH)
Si	silicon (SI)
Si _{ash}	silicon in ashed SRF (SIASH)
Ti	titanium (TI)
Ti _{ash}	titanium in ashed SRF (TIASH)
As	arsenic (AS)
Ba	barium (BA)
Be	beryllium (BE)
Cd	cadmium (CD)
Co	cobalt (CO)
Cr	chromium (CR)
Cu	copper (CU)
Hg	mercury (HG)
Mo	molybdenum (MO)
Mn	manganese (MN)
Ni	nickel (NI)
Pb	lead (PB)
Sb	antimony (SB)
Se	selenium (SE)
Sn	tin (SN)

Tl	thallium (TL)
V	vanadium (V)
Zn	zinc (ZN)
GF-AAS	Graphite Furnace-Atomic Absorption Spectrometry
HG-AAS	Hydride Generation-Atomic Absorption Spectrometry
CV-AAS	Cold Vapour-Atomic Absorption Spectrometry
ICP-OES	Inductively coupled plasma-Optical Emission Spectrometry
ICP-MS	Inductively coupled plasma-Mass Spectrometry
PTFE	polytetrafluorethylene
PFA	perfluoroalkoxy (e.g. PFA liner)
TFM	tetrafluoroethylene, modified (e.g. TFM liner)
XRF	X-ray fluorescence
SRF	Solid recovered fuels
CRM	certified reference material
RM	reference material
(d)	dry basis
l	number of laboratories after outlier rejection (Annex C : number of outlier-free individual analytical values per level)
n	number of individual test results after outlier rejection (Annex C : number of laboratories after outlier elimination)
o	percentage of outliers (Annex C : percentage of outliers from the replicate determination)
Mean(REF)	mean value (reference laboratories)
Mean	overall mean of results (without outliers)
Recovery rate	recovery rate calculated by division of the overall mean value of results (without outliers) by the mean value of reference laboratories, multiplied by 100 %
η	recovery rate (Annex C)
s_R	reproducibility standard deviation
$C_{V,R}$	coefficient of variation of reproducibility
s_r	repeatability standard deviation
$C_{V,r}$	coefficient of variation of repeatability
RSD	relative standard deviation (Annex C)
CV	Coefficient of variation (Annex D)

HCl	hydrochloric acid
HNO ₃	nitric acid
HBF ₄	Tetrafluoroboric acid
H ₂ O ₂	hydrogen peroxide
HF	hydrofluoric acid

5 Safety remarks

Safety when handling potentially hazardous material is dealt with by the applicable (inter-)national regulations.

In addition, the following information is given:

- Only experienced personnel can use the microwave apparatus, following the operating and safety instructions described in the manufacturer manual;
- The microwave unit cavity shall be built in a way that even in case of leakage or explosion of the vessels the safety of the operators can be guaranteed. Household instruments are not suitable for laboratory use.
- Most of the reagents used within this document are strongly corrosive and toxic. Safety precautions are absolutely necessary due to strong corrosive reagents, high temperature and high pressure;
- All procedures have to be performed in a hood or in closed force-ventilated equipment. By the use of strong oxidising reagents, the formation of explosive intermediates is possible, especially when dealing with samples with a high organic content. Do not open pressurised vessels before they have cooled down. Avoid contact with the chemicals and the gaseous reaction products.

6 Principle

The test portion is digested using one of the proposed methods with a suitable acid mixture or by addition of a fluxing agent (ashed SRF). The digested sample is then analysed by the most appropriate spectrometric technique, such as inductively coupled plasma with optical or mass detection (ICP-OES, ICP-MS) or atomic absorption (GF-AAS, CV-AAS, HG-AAS). Alternatively, specific direct methods can be applied after validation (e.g. for determination of mercury or sulfur or for direct determination of elements by XRF).

For XRF analysis of major elements in the ashed SRF, the sample is ashed at 815 °C (ISO 21656 [3]) and the ash is homogenized in a ball mill to obtain a uniform size dimension of the particles. The ash is then pressed in the form of pellets (ISO 22940, EN 15309) or fused with lithium metaborate (EN 15309). Both techniques are suitable for the analysis by XRF. Coal ash and other ashes of various origins can be used for instrument calibration.

7 Reagents

7.1 General

All reagents and acids used shall be of recognized analytical grade or verify the content of the elements to be analysed to avoid high blank values for subsequent analytical measurements. Throughout the procedure, a test blank solution shall be used applying all steps with the same amount of acids or reagents, but without a sample.

7.2 Water, < 0,055 µS/cm, (e.g. deionized water)

7.3 Nitric acid (HNO₃), approximately 15 mol/l, 65 % to 70 % (mass/mass).

- 7.4 Hydrofluoric acid (HF)**, approximately 23 mol/l, 40 % to 45 % (w/w).
- 7.5 Hydrochloric acid (HCl)**, approximately 12 mol/l, 35 % to 37 % (w/w).
- 7.6 Diluted Hydrochloric acid (HCl)**, approximately 2 mol/l. Dissolve 166,5 ml of HCl 37 % (7.5) in water and dilute to 1 l with water (7.2).
- 7.7 Boric acid (H_3BO_3)**, solid.
- 7.8 Boric acid (H_3BO_3) solution**, e.g. 4 % (w/w). Dissolve 40 g of boric acid (7.7) in water and dilute to 1 l of water (7.2).
- 7.9 Hydrogen peroxide (H_2O_2)**, 30 % (w/w).
- 7.10 Lithium metaborate (LiBO_2)**, solid.
- 7.11 Tetrafluoroboric acid (HBF_4)**, approximately 6 mol/l, 38 % to 50 % (w/w).

8 Apparatus

8.1 General

Usual laboratory apparatus along with the following shall be used. All volumetric flasks and digestion vessels shall be adequately cleaned and stored in order to avoid any contamination, particularly with respect to low concentration of the elements of interest.

8.2 Microwave unit, temperature and/or power-controlled, closed vessels

8.2.1 Digestion vessels, for pressurized microwave digestion of appropriate volume, reagent-, temperature- and pressure-resistant and capable of containing the mixture of sample and digest solution. The vessel shall be suitable for the safe application in the temperature and pressure range applied, capable of withstanding pressures of at least 3 000 kPa.

Digestion vessels made of modified polytetrafluorethene (PTFE), e.g. TFM or PFA liner, and equipped with a safety pressure releasing system to avoid explosion of the vessel shall be used. The inner wall of the vessel shall be inert and shall not release contaminations to the digest solutions.

NOTE It can be necessary to periodically clean the digestion vessels with a suitable surfactant to remove persistent deposits.

8.2.2 Microwave unit, temperature-controlled, corrosion resistant and well ventilated.

All electronics shall be protected against corrosion for safe operation. Use a laboratory-grade microwave oven with temperature feedback control mechanisms.

The microwave digestion system shall be able to control the temperature with an accuracy of at least $\pm 5^\circ\text{C}$ and automatically adjust the microwave field output power within 2 s of sensing. Temperature sensors shall be accurate to $\pm 2^\circ\text{C}$, including the final reaction temperature of $190^\circ\text{C} \pm 10^\circ\text{C}$. Temperature feedback control provides the primary performance mechanism for the method. Due to the variability in sample matrix types and microwave digestion equipment (i.e. different vessel types and microwave designs), control of the temperature during digestion is important for reproducible microwave heating and comparable data.

The accuracy of the temperature measurement system shall be periodically tested at an elevated temperature according to the manufacturers' instructions. If the temperature deviates by more than 2°C from the temperature measured by an external, calibrated temperature measurement system, the microwave temperature measurement system shall be re-calibrated.

8.2.3 Microwave unit, power-controlled, corrosion resistant and well ventilated.

All electronics shall be protected against corrosion for safe operation. A laboratory-grade microwave oven with temperature feedback control mechanisms shall be used.

The microwave unit shall be able to provide programmable power which can be programmed to within ± 10 W of the required power. Typical units provide a nominal power of 600 W to 1 200 W. If necessary (referring to manufacturers' specifications), calibration of the microwave unit shall be performed (see [Annex A](#)).

The microwave unit shall be designed in a way that guarantees homogeneous heating of the samples.

The microwave unit shall include a temperature and/or pressure control system.

8.3 Sample containers, plastic containers or glass containers (when no free hydrofluoric acid is present)

8.4 Filter, usually with a pore size of 0,45 μm and resistant to the employed acid mixture and of adequate purity

8.5 Centrifuge, minimum 3 000 r/min

8.6 Volumetric flasks, usually of nominal capacity of 50 ml (microwave digestion) or 100 ml, or 250 ml (fluxing agent)

8.7 Magnetic stirrer with heating function and PTFE stirring bone

8.8 Press, in accordance with ISO 22940:2021, 7.2 or EN 15309:2007, 6.3

8.9 Analytical balance, with an accuracy of 0,1 mg or better

8.10 Muffle furnace for temperatures of 1 050 $^{\circ}\text{C}$

8.11 Platinum crucible, e.g. Pt/Au (5 % (mass/mass))

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

8.13 X-ray fluorescence Spectrometer. Energy or wavelength dispersion system suitable for qualitative and (semi-)quantitative analysis of the elements listed in this document (with the exception of Beryllium and Mercury).

8.14 Atomic Absorption Spectrometer. Normal commercial instrumentation, equipped with graphite furnace or hydride generation systems or cold vapour (GF-AAS, HG-AAS, CV-AAS).

9 Interferences and sources of errors

9.1 General information

The use of glassware shall be excluded when free hydrofluoric acid is present.

In the case of filtration of the digested solution, it is necessary to take care that the filtration procedure does not introduce contaminants.

Ensure that the entire test portion is brought into contact with the acid mixture in the digestion vessel.

Some elements of interest can be lost due to precipitation with ions present in the digest solutions, e.g. low soluble chlorides, fluorides and sulphates.

9.2 Closed vessel system for microwave digestion

The upper limits of mass of the test portion according to the manufacturer's specifications shall be taken into account.

Very reactive or volatile materials that can create high pressures when heated can cause a venting of the vessels with potential loss of sample and analytes.

After digestion, the vessels shall be cooled to room temperature before opening.

10 Preparation of the test sample

The sample preparation for heterogeneous materials as SRF is critical. The test sample is the general analysis test sample with a nominal top size of 0,5 mm or less, following the procedure according to ISO 21646^[4] and shall be selected in a way that it is representative for the laboratory sample.

Depending on the used digestion method, the amount of test portion ranges between 0,2 g and 0,5 g SRF-sample or 0,1 g ashed SRF-sample.

NOTE A nominal top size of 1 mm or less can be sufficient, when a significantly higher amount of sample intake for the digestion step is feasible (at least 1 g).

Determine the moisture content of separate portions of the general analysis test sample of SRF in accordance with ISO 21660-3 at 105 °C. Whereas the determination is carried out on a dry basis (as for ashed SRF-samples), the moisture content shall be determined during the drying step in accordance with ISO 21660-3 at 105 °C.

11 Procedure

11.1 General

The mass of the test portion for a single digestion and the set temperature or pressure-programme shall comply with the specifications of the manufacturer of the digestion unit. Use TFM and PFA liners (after a cleaning run) for total digestion and check the power and temperature calibration of the microwave digestion unit regularly to ensure functionality and reproducible results.

If the required amount of representative test portion exceeds the manufacturer's specifications, the test portion shall be divided into smaller quantities and digested separately. The individual digests shall be combined prior to analysis.

When hydrofluoric acid has been used for digestion, a subsequent complexation run can be necessary to complex free fluorides.

Addition of 11 ml cold-saturated boric acid solution (approx. 4 % H_3BO_3) per ml used hydrofluoric acid is recommended.

11.2 Blank test

A reagent blank test digestion shall be carried out in parallel with the samples, using the same procedure and the same quantities of all the reagents as in the determination of the samples, but omitting the test portion. The laboratory shall define acceptable limits.

NOTE The measurement of a blank solution is needed to determine the contribution of the digestion acid mixture, digestion vessels and filter used to the measured value.

11.3 Method A (general method for SRF and major elements in ashed SRF) - Microwave assisted digestion with hydrochloric, nitric and hydrofluoric acid mixture (6 ml HCl; 2 ml HNO₃; 2 ml HF)

- a) Weigh between 0,2 g to 0,5 g of the SRF sample or 0,1 g of ashed SRF sample, to the nearest of 0,1 mg, prepared according to [Clause 10](#) and transfer it into the vessel ([8.2.1](#)). If necessary, the sample can be moistened with a minimum amount of water ([7.2](#)).

The amount of the test sample depends on the amount of organic matter. The maximum amount of organic carbon shall not exceed 0,15 g when 8 ml of HCl and HNO₃ is used. Per additional 0,1 g organic carbon (more than this 0,15 g), 1 ml of additional concentrated HNO₃ ([7.3](#)) shall be added before the digestion process is started.

Referring to the manufacturer's instructions, the upper limits of mass of the test portion shall be taken into account.

- b) Add (6,0 ± 0,1) ml hydrochloric acid ([7.5](#)) followed by (2,0 ± 0,1) ml nitric acid ([7.3](#)) and (2,0 ± 0,1) ml hydrofluoric acid ([7.4](#)).

If a vigorous reaction occurs, allow the reaction to cease before capping the vessel.

- c) Cap the digestion vessel according to the manufacturer's instructions. Place samples in all available positions in the microwave for sample vessels.

If a lower number of samples are available compared to the vessel positions, use vessels filled with the same amount of the acid mixture without sample to fill all the available vessel slots. This is to ensure the same microwave energy absorption occurs during each digestion procedure. This method is an operationally defined method, designed to achieve consistent digestion of samples by specific reaction conditions.

Temperature profile for temperature- or power-controlled microwave unit, closed vessels:

Microwave heating (acid digestion):

- Step 1: Ramp to 190 °C over 15 min to 20 min;
- Step 2: Hold for 20 min at 190 °C;
- Step 3: Cool down (20 min).

NOTE 1 The stated temperature refers to the digestion solution. The high temperature serves to accelerate the digestion reaction and therefore results in a better digestion efficiency.

1) Microwave unit, temperature-controlled, closed vessels: The temperature of the digestion mixture in each vessel shall be raised with a heating rate of approximately 10 °C/min to 15 °C/min to (190 ± 10) °C and remain at (190 ± 10) °C for (20 ± 1) min. Afterwards cool down to room temperature.

2) Microwave unit, power-controlled, closed vessels: According to the manufacturers' manual a suitable power programme is selected for the numbers of vessels and adjusted to ensure consistent reaction conditions to reach at least 190 °C in 15 until 20 min and to remain at maximum temperature for at least (20 ± 1) min. Afterwards, cool down to room temperature.

WARNING — A too high temperature increase in the microwave assisted digestion can cause a vigorous, exothermic reaction in the digestion solution with high pressure increase and blow-off of the security valve. Losses of analytes are then possible. Therefore, it is mandatory to follow manufacturer's instructions.

If necessary due to manufacturer's instruction, the maximum temperature can be decreased and the programme has to be adjusted (e.g. longer hold time when a lower maximum temperature must be set).

A temperature increase of 10 °C leads to a halving of the reaction time, at the same time a higher temperature also leads to an increase in the oxidation potential of the acid mixtures, which has a positive effect on the digestion efficiency.

- d) Verify the different kind of condition for the effectiveness of digestion for SRF samples and for (certified) reference material/quality control samples by comparison of the results with another laboratory that works according to the conditions listed above. Alternative kinds of digestion methods can be applied, if their performance is proved to be comparable with those of the methods described in this document.
- e) At the end of the microwave programme (temperature-controlled or power-controlled, closed vessels), allow the vessels to cool according to the manufacturer's instructions before removing them from the microwave system. Cooling of the vessels can be accelerated by internal or external cooling devices. After reaching room temperature, check if the microwave vessels maintained their seal throughout the digestion.

Temperature profile for temperature- or power-controlled microwave unit, closed vessels:

Microwave heating (boric acid complexation):

- Step 1: Ramp to 150 °C over 10 min - 15 min;
- Step 2: Hold for 10 min at 150 °C;
- Step 3: Cool down (20 min).

NOTE 2 The stated temperature refers to the digestion solution.

WARNING — A too high temperature increase can cause a vigorous, exothermic reaction in the digestion solution with high pressure increase and blow-off of the security valve. Losses of analytes are then possible.

- f) Boric acid complexation:

Carefully uncap and vent each vessel in a well-ventilated fume hood according to the manufacturer's instructions, then add a solution containing 650 mg boric acid for 2 ml of HF (e.g. 22 ml of 4 % m/m (7.8) for 2 ml of HF). Cap the vessels and put them into the microwave unit and start the boric acid complexation programme (see above). The temperature of the boric acid digestion mixture in each vessel shall be raised with a heating rate of approximately 10 °C/min to (150 ± 10) °C and remain at (150 ± 10) °C for (10 ± 1) min. Afterwards allow the vessels to cool down to room temperature according to the manufacturer's instructions.

- g) Transfer quantitatively the solution content of each vessel into a suitable sized volumetric flask (e.g. 50 ml) and add water (7.2) to the volume mark. Alternatively, another procedure can be applied, such that the adjustment to volume (e.g. 50 ml) with the solid residue still present shall be carried out immediately after digestion.

If the measurement solution contains suspended particles which can clog nebulizers or interfere with an injection of the sample into the instrument, the sample can be centrifuged or filtered (8.4).

The composition of the residue is controlled by XRF or any other suitable technique: if a significant amount of the elements of interest is detected, an alternative digestion method for the dissolution of the residual material is necessary. The control on the residue can be avoided if its composition is known from previous analyses of the same kind of materials or on the basis of experience.

The measurement solution is now ready for the determination of the elements of interest using appropriate elemental analysis techniques.

11.4 Method AT (method for SRF and major elements in ashed SRF) - Microwave assisted digestion with hydrochloric, nitric and tetrafluoroboric acid mixture (6 ml HCl; 2 ml HNO₃; 4 ml HBF₄)

- a) Weigh between 0,2 g to 0,5 g of the SRF sample or 0,1 g of ashed SRF sample, to the nearest of 0,1 mg, prepared according to [Clause 10](#) and transfer it into the vessel ([8.2.1](#)). If necessary, the sample can be moistened with a minimum amount of water ([7.2](#)).

The amount of the test sample depends on the amount of organic matter. The maximum amount of organic carbon shall not exceed 0,15 g when 8 ml of HCl and HNO₃ is used. Per additional 0,1 g organic carbon (more than this 0,15 g), 1 ml of additional concentrated HNO₃ ([7.3](#)) shall be added before the digestion process is started.

Referring to the manufacturer's instructions, the upper limits of mass of the test portion shall be taken into account.

- b) Add (6,0 ± 0,1) ml hydrochloric acid ([7.5](#)) followed by (2,0 ± 0,1) ml nitric acid ([7.3](#)) and (4,0 ± 0,2) ml tetrafluoroboric acid ([7.11](#)).

If a vigorous reaction occurs, allow the reaction to cease before capping the vessel.

- c) Cap the digestion vessel according to the manufacturer's instructions. Place the digestion vessels with samples in all available positions in the microwave.

If a lower number of samples are available compared to the vessel positions, use vessels filled with the same amount of the acid mixture without sample to fill all the available vessel slots. This is to ensure the same microwave energy absorption occurs during each digestion procedure. This method is an operationally defined method, designed to achieve consistent digestion of samples by specific reaction conditions.

- d) Define the microwave programme to achieve the temperature profile cited in [11.3](#) for temperature-controlled or power-controlled, closed vessels and digest the samples in the microwave unit.
- e) At the end of the microwave programme (temperature-controlled or power-controlled, closed vessels), allow the vessels to cool according to the manufacturer's instructions before removing them from the microwave system. Cooling of the vessels can be accelerated by internal or external cooling devices. After reaching room temperature, check if the microwave vessels maintained their seal throughout the digestion.
- f) Finally, transfer quantitatively the solution content of each vessel into a suitable sized volumetric flask (e.g. 50 ml) and add water ([7.2](#)) to the volume mark. Alternatively, another procedure can be applied, such that the adjustment to volume (e.g. 50 ml) with the solid residue still present shall be carried out immediately after digestion.

11.5 Method B (e.g. plastic based SRF and others if method A cannot be applied) - Microwave assisted digestion with hydrochloric, nitric and hydrofluoric acid mixture (0,5 ml HCl; 6 ml HNO₃; 1 ml HF)

- a) Weigh 0,3 g of the sample, to the nearest of 0,1 mg, prepared according to [Clause 10](#) and transfer it into the vessel ([8.2.1](#)). If necessary, the sample can be moistened with a minimum amount of water ([7.2](#)).

Referring to the manufacturer's instructions, the upper limits of mass of the test portion shall be taken into account.

Add (0,5 ± 0,05) ml hydrochloric acid ([7.5](#)) followed by (6,0 ± 0,1) ml nitric acid ([7.3](#)) and (1,0 ± 0,1) ml hydrofluoric acid ([7.4](#)).

If a vigorous reaction occurs, allow the reaction to cease before capping the vessel.

- b) Cap the digestion vessel according to the manufacturer's instructions. Place samples in all available positions in the microwave for sample vessels.

If a lower number of samples are available compared to the vessel positions, use vessels filled with the same amount of the acid mixture without sample to fill all the available vessel slots. This is to ensure the same microwave energy absorption occurs during each digestion procedure. This method is an operationally defined method, designed to achieve consistent digestion of samples by specific reaction conditions.

- c) Define the microwave programme to achieve the temperature profile cited in [11.3](#) for temperature-controlled or power-controlled, closed vessels and digest the samples in the microwave unit.
- d) At the end of the microwave programme (temperature-controlled or power-controlled, closed vessels), allow the vessels to cool according to the manufacturer's instructions before removing them from the microwave system. Cooling of the vessels can be accelerated by internal or external cooling devices. After reaching room temperature, check if the microwave vessels maintained their seal throughout the digestion.
- e) Boric acid complexation

Carefully uncap and vent each vessel in a well-ventilated fume hood according to the manufacturer's instructions, then add a solution containing 325 mg boric acid for 1 ml of HF (e.g. 11 ml of 4 % m/m ([7.8](#)) for 1 ml of HF) and define the microwave boric acid complexation programme to achieve the temperature profile listed in [11.3](#) for temperature-controlled or power-controlled, closed vessels and perform boric acid complexation.

- f) Finally, transfer quantitatively the solution content of each vessel into a suitable sized volumetric flask (e.g. 50 ml) and add water ([7.2](#)) to the volume mark. Alternatively, another procedure can be applied, such that the adjustment to volume (e.g. 50 ml) with the solid residue still present shall be carried out immediately after digestion.

11.6 Method BT (e.g. plastic based SRF and others) - Microwave assisted digestion with hydrochloric, nitric and tetrafluoroboric acid mixture (0,5 ml HCl; 6 ml HNO₃; 2 ml HBF₄)

- a) Weigh 0,3 g of the sample, to the nearest of 0,1 mg, prepared according to [Clause 10](#) and transfer it into the vessel ([8.2.1](#)). If necessary, the sample can be moistened with a minimum amount of water ([7.2](#)).

Referring to the manufacturer's instructions, the upper limits of mass of the test portion shall be taken into account.

- b) Add (0,5 ± 0,05) ml hydrochloric acid ([7.5](#)) followed by (6,0 ± 0,1) ml nitric acid ([7.3](#)) and (2,0 ± 0,2) ml tetrafluoroboric acid ([7.11](#)).

If a vigorous reaction occurs, allow the reaction to cease before capping the vessel.

- c) Cap the digestion vessel according to the manufacturer's instructions. Place samples in all available positions in the microwave for sample vessels.

If a lower number of samples are available compared to the vessel positions, use vessels filled with the same amount of the acid mixture without sample to fill all the available vessel slots. This is to ensure the same microwave energy absorption occurs during each digestion procedure. This method is an operationally defined method, designed to achieve consistent digestion of samples by specific reaction conditions.

- d) Define the microwave programme to achieve the temperature profile cited in [11.3](#) for temperature-controlled or power-controlled, closed vessels and digest the samples in the microwave unit.
- e) At the end of the microwave programme (temperature-controlled or power-controlled, closed vessels), allow the vessels to cool according to the manufacturer's instructions before removing them from the microwave system. Cooling of the vessels can be accelerated by internal or external cooling devices.

After reaching room temperature, check if the microwave vessels maintained their seal throughout the digestion.

- f) Finally, transfer quantitatively the solution content of each vessel into a suitable sized volumetric flask (e.g. 50 ml) and add water (7.2) to the volume mark. Alternatively, another procedure can be applied, such that the adjustment to volume (e.g. 50 ml) with the solid residue still present shall be carried out immediately after digestion.

11.7 Method C (e.g. wood based SRFs) - Microwave assisted digestion with nitric acid, hydrogen peroxide and hydrofluoric acid mixture (2,5 ml H₂O₂; 5 ml HNO₃; 0,4 ml HF)

- a) Weigh 0,3 g of the sample, to the nearest of 0,1 mg, prepared according to [Clause 10](#) and transfer it into the vessel (8.2.1). If necessary, the sample can be moistened with a minimum amount of water (7.2).

Referring to the manufacturer's instructions, the upper limits of mass of the test portion shall be taken into account.

- b) Add (5 ± 0,1) ml nitric acid (7.3) and (2,5 ± 0,1) ml hydrogen peroxide (7.9) and (0,4 ± 0,05) ml hydrofluoric acid (7.4).

If a vigorous reaction occurs, allow the reaction to stop before capping the vessel.

- c) Cap the digestion vessel according to the manufacturer's instructions. Place samples in all available positions in the microwave for sample vessels.

If a lower number of samples are available compared to the vessel positions, use vessels filled with the same amount of the acid mixture without sample to fill all the available vessel slots. This is to ensure the same microwave energy absorption occurs during each digestion procedure. This method is an operationally defined method, designed to achieve consistent digestion of samples by specific reaction conditions.

- d) Define the microwave programme to achieve the temperature profile cited in [11.3](#) for temperature-controlled or power-controlled, closed vessels and digest the samples in the microwave unit.
- e) At the end of the microwave programme (temperature-controlled or power-controlled, closed vessels), allow the vessels to cool according to the manufacturer's instructions before removing them from the microwave system. Cooling of the vessels can be accelerated by internal or external cooling devices. After reaching room temperature, check if the microwave vessels maintained their seal throughout the digestion. Carefully uncap and vent each vessel in a well-ventilated fume hood according to the manufacturer's instructions.

- f) Boric acid complexation

When the instrument used for the analysis is equipped with components resistant to hydrofluoric acid boric acid complexation step can be omitted. Otherwise add a solution containing 130 mg boric acid for 0,4 ml of HF (e.g. 4,4 ml of 4 % m/m (7.8) for 0,4 ml of HF) and define/select the microwave boric acid complexation programme to achieve the temperature profile listed in [11.3](#) for temperature-controlled or power-controlled, closed vessels and perform boric acid complexation.

NOTE For this low amount of hydrofluoric acid, boric acid complexation of the hydrofluoric acid can sometimes be omitted, e.g. for ICP-OES or ICP-MS instruments when nebulizers resistant to hydrofluoric acid are applied.

- g) Finally, transfer quantitatively the solution content of each vessel into a suitable sized volumetric flask (e.g. 50 ml) and add water (7.2) to the volume mark. Alternatively, another procedure can be applied, such that the adjustment to volume (e.g. 50 ml) with the solid residue still present shall be carried out immediately after digestion.

11.8 Method CT (e.g. wood based SRFs) - Microwave assisted digestion with nitric acid, hydrogen peroxide and tetrafluoroboric acid mixture (2,5 ml H₂O₂; 5 ml HNO₃; 0,8 ml HBF₄)

- a) Weigh 0,3 g of the sample, to the nearest of 0,1 mg, prepared according to [Clause 10](#) and transfer it into the vessel ([8.2.1](#)). If necessary, the sample can be moistened with a minimum amount of water ([7.2](#)).

Referring to the manufacturer's instructions, the upper limits of mass of the test portion shall be taken into account.

- b) Add (5 ± 0,1) ml nitric acid ([7.3](#)) and (2,5 ± 0,1) ml hydrogen peroxide ([7.9](#)) and (0,8 ± 0,1) ml tetrafluoroboric acid ([7.11](#)).

If a vigorous reaction occurs, allow the reaction to cease before capping the vessel.

- c) Cap the digestion vessel according to the manufacturer's instructions. Place samples in all available positions in the microwave for sample vessels.

If a lower number of samples are available compared to the vessel positions, use vessels filled with the same amount of the acid mixture without sample to fill all the available vessel slots. This is to ensure the same microwave energy absorption occurs during each digestion procedure. This method is an operationally defined method, designed to achieve consistent digestion of samples by specific reaction conditions.

- d) Define the microwave programme to achieve the temperature profile cited in [11.3](#) for temperature-controlled or power-controlled, closed vessels and digest the samples in the microwave unit.
- e) At the end of the microwave programme (temperature-controlled or power-controlled, closed vessels), allow the vessels to cool according to the manufacturer's instructions before removing them from the microwave system. Cooling of the vessels can be accelerated by internal or external cooling devices. After reaching room temperature, check if the microwave vessels maintained their seal throughout the digestion. Carefully uncap and vent each vessel in a well-ventilated fume hood according to the manufacturer's instructions.
- f) Finally, transfer quantitatively the solution content of each vessel into a suitable sized volumetric flask (e.g. 50 ml) and add water ([7.2](#)) to the volume mark. Alternatively, another procedure can be applied, such that the adjustment to volume (e.g. 50 ml) with the solid residue still present shall be carried out immediately after digestion.

11.9 Method D (major elements in SRF ash) – digestion of ashed SRF with fluxing agent lithium metaborate (fused bead)

- a) Thoroughly mix 0,1 g ± 0,02 g of homogenized ashed sample according to Method B of ISO 21656:2021^[3] (815 °C) with 1 g ± 0,01 g of the fluxing agent lithium metaborate ([7.10](#)) in a platinum crucible.
- b) The mixture is melted in a muffle furnace at 1 050 °C ± 10 °C for 20 min.
- c) The resulting fused bead is allowed to cool down and dissolved incrementally by adding 80 ml of hydrochloric acid (c = 2 mol/l) ([7.6](#)) into the crucible in small amounts. The dissolution is supported by heating (to approx. 60 °C) and stirring with a PTFE stirring bone.
- d) The digest solution is filled to a final volume of 250 ml with deionised water ([7.2](#)). This solution contains 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.

- e) A blank is prepared using the same method, but without test sample.
- f) The element content of Al, Ca, Fe, K, Mg, Na, P, S, Si and Ti in the digestion solution of the ash is determined by ICP-OES according to ISO 11885^[5].

With the used procedure the results for major elements are referred to the ashed SRF sample: calculation on dry basis of the initial SRF sample is performed using the equation reported in [Clause 13](#).

12 Analysis

12.1 Preparation of the solution for analysis

If the digested sample contains particles which can clog the nebulizers of the measurement apparatus or which can interfere with the injection of the sample into the instrument, the sample solution can be centrifuged (minimum 3 000 r/min), allowed to settle (overnight) or be filtered (0,45 µm). In the case of filtration, dilute the content of the vessel before filtering, rinse and then fill to the mark of the volumetric flask. The method used has to be reported in the test report.

Solutions containing hydrofluoric acid shall be processed with HF resistant apparatus.

12.2 Analytical step

For the detection of the concentrations of the elements in the digests, the following methods can be used:

- ICP-OES according to the general principles of ISO 11885^[5];
- ICP-MS according to the general principles of ISO 17294-2^[6];
- GF-AAS according to the general principles of ISO 15586^[7];
- CV-AAS according to the general principles of ISO 12846 (mercury)^[8];
- HG-AAS according to the general principles of ISO 17378-2^[9];
- Direct mercury analysis according to the method reported in EPA Method 7473^[10];
- sulfur determination of ashed SRF using a high-temperature tube furnace according to the method reported in ASTM D5016-16^[11];
- total sulfur determination according to ISO 21663^[2] (instrumental method).

Instruments shall be set up and calibrated following the manufacturers' instructions and the used reference methods.

12.3 XRF analysis on ashed samples – sample preparation (major elements only)

The sample is prepared following the general principle of ISO 21656 ^[3] (ashing at 815 °C). A sufficient amount of SRF sample shall be ashed in order to obtain a suitable amount of sample (at least 1 g to 2 g of ash); the total ash content can be very low for some types of samples (5 % or less). The sample preparation is a critical stage for the analysis by X-ray fluorescence. The quality of the sample preparation will strongly influence the standard deviation on repeated measurements.

The available preparation techniques for solids are powder, pressed pellets and fused glass disc.

NOTE 1 The error of the light elements Na to S decreases with pressed pellets in the preparation technique.

NOTE 2 By using a fused glass, particle size effects are eliminated.

NOTE 3 The particle size of the solid sample will strongly influence the standard deviation on repeated measurements.

The resulting ashes are analysed in the form of pressed pellets or fused glass disc. The analysis of the sample prepared as described can be performed following the general principles of several methods.

NOTE 4 Examples of such methods are ASTM D4326-13 ^[12] or EN 15309 or EN ISO 22940.

With the used procedure, the results for major elements are referred to the ashed SRF sample: calculation on dry basis of the initial SRF sample is performed using [Formula \(1\)](#) given in [Clause 13](#).

13 Calculations

The results shall be expressed in milligrams per kilogram on a dry basis.

For method D according to [11.9](#) and for XRF procedure on SRF ash according to [12.3](#), [Formula \(1\)](#) shall be used to express the results for major elements on dry original matter:

$$C_{i,d} = C_{i,ash} \times \frac{A_{db}}{100} \quad (1)$$

where

- $C_{i,d}$ is the concentration of a selected major element i on dry basis in the SRF sample, in mg/kg (d)
- $C_{i,ash}$ is the concentration of a selected major element i in the ash, in mg/kg (d)
- A_{db} is the ash content of the general analysis sample at 815 °C on dry basis according to ISO 21656:2021[3], expressed as mass fraction in percent of the dry matter, in % dm.

14 Quality control

To detect possible contaminations from vessels and/or reagents, blank tests shall be carried out by the same sample preparation procedure, using the same quantities of reagents but without sample intake.

If available, the use of standard reference materials is recommended. The trueness of the analytical method shall be checked by applying it to a certified reference material (CRM), if existing or a reference material, e.g. a well characterized SRF-sample (RM/quality control sample). Calculate the analytical result for the CRM/RM and compare it with the reference or certified value.

15 Performance characteristics

Data about performances of all described digestion methods and direct methods derived by the validation inter-laboratory study SRF22ERI[13] using real SRF-samples are shown in [Annex B](#). Data about ruggedness and performances of methods for SRF by the European QUOVADIS project[14,15] are shown in [Annex C](#) and [Annex D](#).

16 Test report

The test report shall contain at least the following information:

- a) name, address and location of any laboratory involved in the digestion and analysis;
- b) description and identification of the laboratory sample;
- c) date of receipt of laboratory sample and date(s) of performance of test;
- d) the digestion method and acid mixture/reagents used, together with a reference to this document and subclause, e.g. ISO 3884:2025, 11.3, Method A (aqua regia + HF + boric acid complexation) or 11.4 Method AT (aqua regia + HBF₄);
- e) reference to the analytical standard used for the determination of each element;
- f) the analytical results, referring to [Clause 13](#);
- g) any details not specified in this document or which are optional, and any other factors which can have affected the results;
- h) unique identification of report (such as serial number) and of each page and total number of pages of the report.

The laboratory shall keep a record of any analytical steps and intermediate results (raw data and calculation details) for quality reasons.

Annex A (informative)

Calibration of the power adjustment

In order to compare the power adjustment of different apparatus between each other, the actually supplied (effective) power shall be determined. Additionally, the relationship between the supplied power and the adjustment scale shall be controlled. Furthermore, the effective power shall be checked periodically.

Calibration is carried out by heating a known amount of water during a fixed time period (e.g. 2 min) at different microwave power rates. Each raise of temperature has to be measured with a limit deviation of $\pm 0,1$ °C after each heating cycle. The absorbed power is calculated from the raise of temperature (see formula at the end of this annex).

As an example, place 1 kg of water in a plastic beaker (or a beaker made of other material that does not absorb or reflect microwave energy), stir and measure the temperature. Place the beaker in the microwave oven. Always select the same position. Set the microwave during 2 min at full power. Remove the beaker, stir and measure the final temperature. Repeat this procedure also at lower power rates.

The power absorbed is calculated using [Formula \(A.1\)](#):

$$P = \frac{C_p \times m \times \Delta T}{t} \quad (\text{A.1})$$

where

- P is the power absorbed by the water in W (J/s);
- C_p is the specific heat for water J/kg °C (=4 184 J/kg °C);
- m is the mass of the water that is used for the calibration in kg;
- ΔT is the difference between initial and final temperature in °C;
- t is the time period in s.

If in the described procedure 2 min and 1 kg water are used, the formula can be simplified to:

$$P = \Delta T \times 34,87$$

where

- P is the power absorbed by the water in W (J/s);
- ΔT is the difference between initial and final temperature in °C.

Annex B (informative)

Validation and performance data (SRF22ERI)

B.1 General

An interlaboratory comparison study on the determination of element content (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) of solid recovered fuels (SRF) was organized by Umweltbundesamt Austria in 2022 (SRF22ERI)^[13] and 36 participants from 14 countries registered for this part (AT, BE, CH, DE, ES, FR, HU, IT, JP, KR, NL, SI, SK, UK) (sample dispatch: September 12th, 2022; closing time for the submission of results: October 27th, 2022; extension for element content by November 22nd, 2022 at the latest, report 2023).

For validation, four real samples of SRF were selected and distributed to the registered participants. The sampling of SRF1, SRF3 and SRF4 according to ISO 21645^[16] was carried out by Montanuniversität Leoben. The samples of SRF2 were provided by Umweltbundesamt Austria. Sample preparation and homogenization (ISO 21646^[4]) and homogeneity testing was performed jointly by Montanuniversität Leoben and Umweltbundesamt Austria.

- SRF1E: paper fibre sludge (1 bottle ~25 g per sample SRF1E, particle size: < 0,5 mm)
- SRF2E: municipal sewage sludge (1 bottle ~25 g per sample SRF2E, particle size: finely ground)
- SRF3E: waste wood (1 bottle ~25 g per sample SRF3E, particle size: < 0,5 mm)
- SRF4E: high calorific waste fraction from non-hazardous municipal solid waste (1 bottle ~25 g per sample SRF4E, particle size: < 0,5 mm)

All provided samples for the validation were real samples and contained the elements of interest as they occur in their original composition – no spiking was performed.

The participants were advised to follow the procedures according to this document. Four independent determinations per sample and method (e.g. for method A, method B, method C, method D) had to be carried out. Two independent determinations for calculation of dry mass and ash content were recommended. In case of XRF analysis two independent determinations were requested (e.g. preparation of two pellets/fused glass discs and duplicate analysis).

Statistical data evaluation was performed according to ISO 5725-2^[17] using R-Studio. For the statistical evaluation, all results per each method were treated separately. All data sets were tested for outliers by Grubbs test, Hampel test and Cochran test (ISO 5725-2), and after plausibility check, technically confirmed outliers were eliminated for further statistical treatment.

The statistical data of results for each method are presented in the following [Tables B.1](#) to [B.42](#). Values below limit of quantification (LOQ) or below limit of detection (LOD) are expressed as '<LOQ' or '<LOD'.

B.2 Results

Key to [Tables B.1](#) - [B.42](#)

l	number of laboratories after outlier rejection
n	number of individual test results after outlier rejection
o	percentage of outliers
Mean(REF)	mean value (reference laboratories)
Mean	overall mean of results (without outliers)
Recovery rate	Recovery rate calculated by division of the overall mean value of results (without outliers) by the mean value of reference laboratories, multiplied by 100 %
s_R	reproducibility standard deviation
$C_{V,R}$	coefficient of variation of reproducibility
s_r	repeatability standard deviation
$C_{V,r}$	coefficient of variation of repeatability
