BS EN ISO 16967:2015

BSI Standards Publication

Solid biofuels — Determination of major elements — Al, Ca, Fe, Mg, P, K, Si, Na and Ti (ISO 16967:2015)

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National foreword

This British Standard is the UK implementation of EN ISO 16967:2015. It supersedes BS EN 15290:2011 which is withdrawn.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 secretary.

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Biocombustibles solides - Détermination des éléments majeurs - Al, Ca, Fe, Mg, P, K, Si, Na et Ti (ISO 16967:2015)

Biogene Festbrennstoffe - Bestimmung von Hauptelementen - Al, Ca, Fe, Mg, P, K, Si, Na und Ti (ISO 16967:2015)

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Foreword

This document (EN ISO 16967:2015) has been prepared by Technical Committee ISO/TC 238 "Solid biofuels" in collaboration with Technical Committee CEN/TC 335 “Solid biofuels” the secretariat of which is held by SIS.

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of an identical text or by endorsement, at the latest by October 2015, and conflicting national standards shall be withdrawn at the latest by October 2015.

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Endorsement notice

The text of ISO 16967:2015 has been approved by CEN as EN ISO 16967:2015 without any modification.

Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 238, *Solid biofuels*.

Introduction

The elements described as major elements of solid biofuels are in fact major elements of the fuel ashes more than of the fuels. The determination of these elements can be used to assess ash behaviour in a thermal conversion process or to assess utilization of ashes. Moreover, fuel contamination or process additives are indicated by high values of certain elements. Contamination of fuel with sand or soil is indicated by high values of several elements.

In this International Standard, wet chemical methods are described.

Solid biofuels — Determination of major elements — Al, Ca, Fe, Mg, P, K, Si, Na and Ti

1 Scope

This International Standard describes methods for the determination of major elements of solid biofuels respectively of their ashes, which are Al, Ca, Fe, Mg, P, K, Si, Na, Ti. The determination of other elements such as barium (Ba) and manganese (Mn) is also possible with the methods described in this International Standard.

This International Standard includes two parts: Part A describes the direct determination on the fuel, this method is also applicable for sulfur and minor elements, Part B gives a method of determination on a prepared 550 °C ash.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 7980, *Water quality — Determination of calcium and magnesium — Atomic absorption spectrometric method*

ISO 9964-1, *Water quality — Determination of sodium and potassium — Part 1: Determination of sodium by atomic absorption spectrometry*

ISO 9964-2, *Water quality — Determination of sodium and potassium — Part 2: Determination of potassium by atomic absorption spectrometry*

ISO 9964-3, *Water quality — Determination of sodium and potassium — Part 3: Determination of sodium and potassium by flame emission spectrometry*

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

EN 14780[1], *Solid Biofuels — Sample preparation*

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 16993, *Solid biofuels — Conversion of analytical results from one basis to another*

ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements*

ISO 18122[2], *Solid biofuels — Determination of ash content*

ISO 18134-3[2], *Solid biofuels — Determination of moisture content — 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 16559 and the following apply.

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1) To be replaced by ISO 14780.

2) To be published.

3.1
reference material
RM
material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials

3.2
certified reference material
CRM
reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence

3.3
NIST standard reference material
SRM
CRM issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterisations and provides information regarding the appropriate use(s) of the material

4 Symbols and abbreviated terms

4.1 Symbols

Al	Aluminium
Ca	Calcium
Fe	Iron
Mg	Magnesium
P	Phosphorus
K	Potassium
Si	Silicon
Na	Sodium
Ti	Titanium

4.2 Abbreviated terms

CRM	Certified Reference Material
ICP-OES	Inductively Coupled Plasma – Optical Emission Spectrometry
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
FAAS	Flame Atomic Absorption Spectrometry
FES	Flame Emission Spectrometry
SRM	Standard Reference Material

NBS	National Bureau of Standards
NIST	The **National Institute of Standards and Technology** (**NIST**), known between 1901 and 1988 as the **National Bureau of Standards** (**NBS**), is a measurement standards laboratory, also known as a National Metrological Institute (NMI), which is a non-regulatory agency of the United States Department of Commerce.

5 Principle

The sample is digested in a closed vessel by the help of reagents, temperature, and pressure. The digestion is either carried out directly on the fuel (part A) or on a 550 °C prepared ash (part B).

The detection of the elements can be done by ICP-OES, ICP-MS, FAAS, or FES.

6 Reagents

All reagents should be of analytical grade or better. If minor elements are also to be determined, the best qualities should be used.

6.1 Water, containing negligible amounts of major elements, i.e. amounts that do not contribute significantly to the determinations. Deionised water will normally fulfil this requirement.

6.2 Nitric acid (HNO_3), ≥65 % (w/w), $\rho = 1{,}41$ g/ml.

6.3 Hydrogen peroxide (H_2O_2), 30 % (w/w), $\rho = 1{,}11$ g/ml.

6.4 Hydrofluoric acid (HF), 40 % (w/w), $\rho = 1{,}13$ g/ml.

CAUTION — Hydrofluoric acid might lead to health hazards.

6.5 Boric acid (H_3BO_3), 4 % (w/w).

6.6 Use of certified reference materials (CRM or SRM).

Use certified reference materials, issued by an internationally recognized authority, to check if the accuracy of the calibration meets the required performance characteristics. Examples of certified reference materials are: NBS 1570 spinach leaves, NBS1571 orchard leaves, NBS 1573 tomato leaves, and NBS 1575 pine needles.

When, due to matrix effects or concentration range limitations, no good recoveries for the certified reference materials can be obtained, calibration with at least two CRM or SRM materials can solve these problems. In that case, CRM or SRM materials other than used for the calibration shall be used for verification purposes.

NOTE A CRM or SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

7 Apparatus

7.1 Heating oven or heating block suitable for the decomposition system in use, resistance heated oven or heating block that can be used at a temperature of at least 220 °C with an accuracy of ±10 °C.

7.2 Microwave oven, intended for laboratory use and equipped with temperature control.

7.3 Sample digestion vessels, intended for the heating system used, normally made of a fluoro plastic.

7.4 Balance.

7.4.1 Part A, balance with a resolution of at least 1 mg.

7.4.2 Part B, balance with a resolution of at least 0,1 mg.

7.5 Plastic volumetric flasks.

8 Preparation of the test sample

The test sample is the general analysis test sample with a nominal top size of 1 mm or less, prepared in accordance with EN 14780[3].

The moisture content of the test sample shall be determined as described in ISO 18134-3.

9 Procedure

9.1 Digestion

9.1.1 Part A: Direct determination on the fuel

The decomposition shall be carried out in closed vessels. It can be done in a heating oven, a heating block or in a microwave oven.

- Mix 500 mg of ground and homogenized sample, weighed to the nearest 1 mg, with 3,0 ml H_2O_2 (30 %), 8,0 ml HNO_3 (65 %), and 1,0 ml HF (40 %) in a closed digestion vessel. A reaction time of minimum 5 min shall be kept before closing the vessel. Closing the digestion vessel too early can result in a fast pressure build up, sometimes exceeding the maximum pressure limit of the vessel.

If the sample is expected to have an ash content above 10 %, 2,0 ml HF (40 %) should be used.

- The heating of the vessel shall not be too fast. Heat the sample according to the following heating programmes for digestion:

Resistance heating[4]: Step 1: Ramp to 220 °C over 1 h

Step 2: Hold for 1 h at 220 °C

Microwave heating[5]: Step 1: Ramp to 190 °C over 15 min

Step 2: Hold for 20 min at 190 °C

If the maximum pressure limit of the vessel is exceeded during the digestion and by that an opening of the relief valve has occurred, the digestion should be discarded due to possible loss of Si (in form of gaseous SiF_4).

NOTE Some available digestion bomb systems use fluoropolymer vessels, which cannot withstand temperatures above 170 °C. In such cases, this lower temperature can be used, provided that the sample is held longer at this temperature and that comparable results can be obtained, e.g. by the use of equivalent biomass reference materials.

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[3] To be replaced by ISO 14780.

[4] The stated temperature refers to heating device (e.g. oven).

[5] The stated temperature refers to digest solution.

— After cooling to room temperature, HF is neutralised by adding 10 ml H3BO3 (4 %).

If 2,0 ml HF (40 %) was used for the digestion, 20 ml H_3BO_3 (4 %) should be used for the neutralization.

— Reheat the sample according to the following heating programmes for neutralization:

Resistance heating[4]):	Step 1: Heat rapidly to 180 °C
	Step 2: Hold for 15 min at 180 °C
Microwave heating[5]):	Step 1: Heat rapidly to 150 °C
	Step 2: Hold for 15 min at 150 °C

— After cooling, transfer the digest to a volumetric flask. Rinse the digestion vessel carefully and transfer the rinse solution to the volumetric flask. Add deionised water to the digest to an appropriate volume, depending on the detection method to be used.

9.1.2 Part B: Determination on a prepared 550°C ash

— Heat the sample according to the procedure described in ISO 18122 to obtain ash. Make sure that the ashing procedure is performed exactly according to this procedure as deviations in ashing temperature, time, and air refreshing rate will influence the results. In deviation of ISO 18122, only crucibles made of platinum or graphite can be used for the preparation of the ash, but larger types of crucibles can be used. The use of the stated additives in ISO 18122 to ensure complete combustion is not allowed in the preparation. Also a continuous ashing by refilling of the sample on the previous ash in the crucible is not allowed.

To prepare a sufficient amount of ash for the digestion of larger amounts of sample, compared to the procedure given in ISO 18122, often will be necessary. The ash percentage on dry basis obtained for the prepared ash, thus, shall be calculated and compared to obtained results for the ash content on dry basis determined exactly according to ISO 18122. If the ash content for the prepared ash is also known, the results for major elements determined for the prepared ash can be calculated to fuel basis.

— Homogenize the prepared ash in an agate mortar and reignite the homogenized ash at 550 °C for 30 min.

NOTE 1 The weighing of the test portion of the ash for the digestion has to be carried out immediately after the preparation.

For the digestion of the ash similar working steps, as for the digestion of the fuel, are evident:

— Mix 50 mg of ground and homogenized ash, weighed to the nearest 0,1 mg, with 2,0 ml H_2O_2 (30 %), 3,0 ml HNO_3 (65 %), and 2,0 ml HF (40 %) in a closed decomposition vessel. A reaction time of minimum 5 min shall be kept before closing the vessel.

— Digest the sample following one of the heating programmes described in 9.1.1 for digestion.

If the maximum pressure limit of the vessel is exceeded during the digestion and by that an opening of the relief valve has occurred, the digestion should be discarded due to possible loss of Si (in form of gaseous SiF4).

— After cooling to room temperature, the HF is neutralized by adding 20 ml H_3BO_3 (4 %) and 10 ml deionised water.

NOTE 2 The water is necessary to keep K in solution for bio-ashes with high KCl content.

— Reheat the sample according to the heating programmes for neutralization described in 9.1.1.

- After cooling, transfer the digest to a volumetric flask. Rinse the digestion vessel carefully and transfer the rinse solution to the volumetric flask. Add deionised water to the digest to an appropriate volume, depending on the detection method to be used.

9.2 Detection methods

For the detection of the concentrations of Al, Ca, Fe, Mg, P, K, Si, Na, Ti in the digests, the following methods can be used:

- ICP-OES according to the principles of ISO 11885;
- ICP-MS according to the principles of ISO 17294-2;
- AAS according to the principles of ISO 7980, ISO 9964-1, and ISO 9964-2;
- FES according to the principles of ISO 9964-3.

9.3 Calibration of the apparatus

When the analytical system is evaluated for the first time for this application, establish a calibration function for the measurement in accordance with the manufacturers' instructions. Adjust the established calibration function during the analysis, if necessary. Check the performance of the instrument using the accepted standard procedures like replicate analysis, use of SRM and/or CRM, control samples and control charts. The calibration and quality control scheme shall be organized and maintained in such a way that the required uncertainty of measurement can be obtained. The results of the validation study of BioNorm2 (Annex B) demonstrates what is achievable with commercial instruments that are used by experienced laboratories.

9.4 Analysis of digests

Analyse test portions of the digests in accordance with the manufacturer's instructions.

9.5 Blank test

Carry out a blank test, using the same procedure and methods as described in 9.1.1, 9.1.2, 9.2, 9.3, and 9.4 but omitting the test portion. This assesses both the contents of the elements in the reagents and any contamination from equipment and the laboratory atmosphere. This contribution shall not be quantitatively significant.

NOTE A content of the elements in the digests of the blank experiment at 20 % or less of the content of the elements in the digests can be considered as not quantitatively significant.

10 Calculations

The content of an element in the sample on dry basis, w_i, expressed in mg/kg, is calculated from the mean of duplicate determinations using the Formula (1):

$$w_i = \frac{\left(c_i - c_{i,0}\right) \times V}{m} \times \frac{100}{\left(100 - M_{ad}\right)} \tag{1}$$

where

w_i is the concentration of the element in the sample, on a dry basis, in mg/kg;

c_i is the concentration of the element, in the diluted sample digest, in mg/l;

$c_{i,0}$ is the concentration of the element, in the solution of the blank experiment, in mg/l;

V is the volume of the diluted sample digest solution, in ml;

m is the mass of the test portion used, in g;

M_{ad} is the moisture content in the analysis test sample in % m/m.

The results can be calculated to other bases, e.g. to as received basis according to ISO 16993.

If the determination has been carried out on a prepared ash (Part B), the results can be calculated to the fuel basis using Formula (2):

$$w_{i,fuel} = w_{i,ash} \times \frac{A_d}{100} \tag{2}$$

where

A_d is the obtained ash content, concerning the prepared ash used for the digestion, in % m/m, dry basis;

$w_{i,\ fuel}$ is the concentration of the element in the fuel sample, on a dry basis, in mg/kg;

$w_{i,\ ash}$ is the concentration of the element in the prepared ashed sample, on a dry basis, in mg/kg.

11 Performance characteristics

The achievable performance of the method is given in Annex B, showing the results obtained by a European inter comparison study carried out for a sample of wood chips and a sample of an exhausted olive residue. These two samples represent the extremity of the method. The wood chip sample represents samples with low contents of most of the elements and the olive residue samples with high amounts of most of the elements.

12 Test report

The test report shall contain at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (sample) tested;

c) a reference to this International Standard, ISO 16967;

d) applied digestion procedure and test method used for determination;

e) results of the test including the basis in which they are expressed, as indicated in Clause 10;

f) any unusual features noted during the test procedure;

g) any operation not included in this International Standard, or regarded as optional.

Annex A
(informative)

List of conversion factors

The following list gives conversion factors for the calculation on the composition on an oxide basis in the case of determination on a prepared 550 °C ash.

$Al \rightarrow Al_2O_3$	1,89
$Ca \rightarrow CaO$	1,40
$Fe \rightarrow Fe_2O_3$	1,43
$Mg \rightarrow MgO$	1,66
$P \rightarrow P_2O_5$	2,29
$K \rightarrow K_2O$	1,20
$Si \rightarrow SiO_2$	2,14
$Na \rightarrow Na_2O$	1,35
$Ti \rightarrow TiO_2$	1,67

Annex B
(informative)

Performance data

The round robin was carried out by laboratories in Austria, Belgium, Denmark, Finland, Germany, Ireland, Italy, The Netherlands, Spain, Sweden, and the United Kingdom. The variety of instruments and other analytical conditions were used in accordance with the quality parameters specified in the method.

The tests were carried out using two samples, wood chips, and exhausted olive residues produced in the EU-project BioNorm according to prEN 14780:2006. The sample “wood chips” was made of German coniferous wood chips; the chips were dried and milled to 1 mm by means of cutting mill. The sample “exhausted olive residues” was obtained from olive oil industry in Spain from a typical outdoor storage facility. In the original sample, stones and other natural impurities were present. These impurities and stones were removed manually and the sample was prepared from the residues in two steps using a coarse cutting mill equipped with a 10 mm sieve and a laboratory cutting mill equipped with WC cutting tools and a 1 mm sieve.

All data are reported on dry basis.

The performance data according to ISO 5725-2 are presented in Tables B.1 to B.9.

NOTE 1 See Table B.1 for definition of the symbols used in the Tables B.1 to B.9.

NOTE 2 Guidelines can be found in ISO 16993:2015, Annex C on how to use these validation parameters.

Table B.1 — Performance data for Aluminium (Al)

Sample	n	l	o %	X mg/kg	s_R mg/kg	CV_R %	s_r mg/kg	CV_r %
wood chips	12	57	5	47	8	18	2	4,1
exhausted olive residues	11	55	0	2360	170	7,2	110	4,7
Definition symbols								
N	is the number of laboratories after outlier elimination							
L	is the number of outlier free individual analytical values							
O	is the percentage of outlying values from replicate determination							
X	is the overall mean							
s_R	is the reproducibility standard deviation							
CV_R	is the coefficient of the variation of the reproducibility							
s_r	is the repeatability standard deviation							
CV_r	is the coefficient of the variation of the repeatability							

Table B.2 — Performance data for Calcium (Ca)

Sample	n	l	o %	x mg/kg	s_R mg/kg	CV_R %	s_r mg/kg	CV_r %
wood chips	11	54	1,8	1 500	100	6,6	24	1,6
exhausted olive residues	13	65	0	14 200	1 040	7,3	607	4,3

Table B.3 — Performance data for Iron (Fe)

Sample	*n*	*l*	*o*	*x*	s_{R}	CV_{R}	s_{r}	CV_{r}
			%	mg/kg	mg/kg	%	mg/kg	%
wood chips	15	69	5,5	54	6	11,7	4	6,6
exhausted olive residues	15	73	1,4	1 600	165	10,3	81	5,1

Table B.4 — Performance data for Magnesium (Mg)

Sample	*n*	*l*	*o*	*x*	s_{R}	CV_{R}	s_{r}	CV_{r}
			%	mg/kg	mg/kg	%	mg/kg	%
wood chips	12	60	0	194	14	7,2	3	1,7
exhausted olive residues	13	65	0	3 140	243	7,7	149	4,7

Table B.5 — Performance data for Phosphorus (P)

Sample	*n*	*l*	*O*	*x*	s_{R}	CV_{R}	s_{r}	CV_{r}
			%	mg/kg	mg/kg	%	mg/kg	%
wood chips	11	53	3,6	74	5	6,7	2	3,4
exhausted olive residues	13	65	0	1 490	127	8,5	58	3,9

Table B.6 — Performance data for Potassium (K)

Sample	*n*	*l*	*O*	*x*	s_{R}	CV_{R}	s_{r}	CV_{r}
			%	mg/kg	mg/kg	%	mg/kg	%
wood chips	13	63	3,1	691	77	11,1	12	1,8
exhausted olive residues	11	52	5,5	24 500	1 560	6,4	468	1,9

Table B.7 — Performance data for Silicon (Si)

Sample	*n*	*L*	*o*	*x*	s_{R}	CV_{R}	s_{r}	CV_{r}
			%	mg/kg	mg/kg	%	mg/kg	%
wood chips	8	39	2,5	320	106	33	36	11,2
exhausted olive residues	10	49	4,0	10 040	1 230	12,2	769	7,7

Table B.8 — Performance data for Sodium (Na)

Sample	*n*	*l*	*o*	*x*	s_{R}	CV_{R}	s_{r}	CV_{r}
			%	mg/kg	mg/kg	%	mg/kg	%
wood chips	9	44	0	13	6	48	2	14
exhausted olive residues	10	49	0	171	40	23	9	5,4

Table B.9 — Performance data for Titanium (Ti)

Sample	*n*	*l*	*o*	*x*	s_{R}	CV_{R}	s_{r}	CV_{r}
			%	mg/kg	mg/kg	%	mg/kg	%
wood chips	9	43	4,4	5,5	0,40	7,4	0,32	5,9
exhausted olive residues	11	54	1,8	136	11	7,9	6	4,4

Table B.10 — Lists of techniques for the round robin

Used method	**No. of laboratories (18 participating laboratories)**
No information	3
Microwave digestion ICP-OES and ICP-MS	13
Microwave digestion and AAS	1
Fusion bead and X-ray fluorescence spectometry	1

Bibliography

[1] ISO 5725-2:2004, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[2] ISO 16996[6], *Solid biofuels — Determination of elemental composition by X-ray fluorescence*

[3] ISO/TS 21748:2004, *Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation*

[4] NIST Technical note 1297:1994 *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results.*

[5] NIST definitions: http://ts.nist.gov/MeasurementServices/ReferenceMaterials/DEFINITIONS.cfm.

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[6] Under development.

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