INTERNATIONAL STANDARD

ISO 16968

First edition
2015-05-15

Solid biofuels — Determination of minor elements

Biocombustibles solides — Détermination des éléments mineurs

Reference number
ISO 16968:2015(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

ISO 16968 was prepared by the European Committee for Standardization (CEN) in collaboration with Technical Committee ISO/TC 238, *Solid biofuels*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Introduction

The minor elements present in solid biofuels can, in some cases, be of environmental concern, e.g. it has been shown that certain energy crops will concentrate cadmium and, in polluted areas, other toxic elements may be found at elevated concentrations in the biofuels. This can be a problem if, for example, the ash from the combustion is to be put back in the forest as a fertilizer. Trace elements in biofuels are often present at very low concentrations requiring great care to avoid contamination in the sample preparation and decomposition steps. The typical concentrations of minor elements in solid biofuels can be found in ISO 17225-1. In this International Standard, wet chemical methods are described. Alternative methods such as X-ray fluorescence (XRF) or direct mercury analysers may be used when validated with suitable materials (biomass reference materials).

Solid biofuels — Determination of minor elements

1 Scope

This International Standard is intended for the determination of the minor elements Arsenic, Cadmium, Cobalt, Chromium, Copper, Mercury, Manganese, Molybdenum, Nickel, Lead, Antimony, Vanadium, and Zinc in all solid biofuels. Further, it describes methods for sample decomposition and suggests suitable instrumental methods for the determination of the elements of interest in the digests. The determination of other elements such as Selenium, Tin, and Thallium is also possible with the method described in this International Standard.

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

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 17378-2:2014, *Water quality — Determination of arsenic and antimony — Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)*

ISO 18134-3[1)], *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis simple*

EN 14780, *Solid Biofuels — Sample preparation*

EN 12338, *Water quality — Determination of mercury — Enrichment methods by amalgamation*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

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

1) To be published.

3.2
certified reference material
CRM
reference material, accompanied by a certificate, one or more of which property values are certified by a procedure that 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

As Arsenic

Cd Cadmium

Co Cobalt

Cr Chromium

Cu Copper

Hg Mercury

Mn Manganese

Mo Molybdenum

Ni Nickel

Pb Lead

Sb Antimony

V Vanadium

Zn Zinc

4.2 Abbreviated terms

CV-AAS Cold vapour atomic absorption spectrometry

GF-AAS Graphite furnace atomic absorption spectrometry

HG-AAS Hydride generation atomic absorption spectrometry

ICP-OES Inductively coupled plasma optical emission spectrometry

ICP-MS Inductively coupled plasma mass spectrometry

NBS National Bureau of Standards

NIST National Institute of Standards and Technology[a]

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

5 Principle

The analysis sample is digested in a closed vessel made from a fluoropolymer using nitric acid, hydrogen peroxide, and hydrofluoric acid in a microwave oven or a resistance oven or heating block. The digest is then diluted and the elements are determined with suitable instruments.

6 Reagents

All reagents shall be of analytical grade or better. If the blank level is unacceptably high, i.e. more than 30 % of the determined value, the use of ultra-pure reagents should be investigated.

6.1 Water, containing negligible amount of the minor elements, i.e. amount that do not contribute significantly to the determinations.

NOTE 1 Deionised water or doubly distilled water normally fulfils this requirement.

NOTE 2 The water used for analytical trace metal work is normally produced using a system for production of ultra-pure water for laboratory use conductivity = 0,056 µS/cm.

6.2 Hydrofluoric acid (HF), 40 % (w/w), ρ = 1,13 g/ml.

CAUTION — Hydrofluoric acid may lead to health hazards.

6.3 Hydrogen peroxide (H_2O_2), 30 % (w/w), ρ = 1,11 g/ml.

6.4 Nitric acid (HNO_3), ≥ 65 % (w/w), ρ = 1,41 g/ml.

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

6.6 Certified Reference Materials (CRM or SRM), issued by an internationally recognized authority, to check if the accuracy of the calibration meets the required performance characteristics.

EXAMPLE NBS 1570 spinach leaves, NBS1571 orchard leaves, NBS 1573 tomato leaves, and NBS 1575 pine needles.

When no good recoveries for the certified reference materials can be obtained, due to matrix effects or concentration range limitations, calibration with at least two CRM or SRM materials may 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.