



SLOVENSKI STANDARD
SIST EN 15297:2011

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Nadomešča:

SIST-TS CEN/TS 15297:2006

Trdna biogoriva - Določevanje mikro elementov - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V in Zn

Solid biofuels - Determination of minor elements - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V and Zn

Feste Biobrennstoffe - Bestimmung von Spurenelementen - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V und Zn

Biocombustibles solides - Détermination des éléments mineurs - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V et Zn

Ta slovenski standard je istoveten z: EN 15297:2011

ICS:

75.160.10 Trda goriva

Solid fuels

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EUROPEAN STANDARD

EN 15297

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English Version

Solid biofuels - Determination of minor elements - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V and Zn

Biocombustibles solides - Détermination des éléments mineurs - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V et Zn

Feste Biobrennstoffe - Bestimmung von Spurenelementen - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V und Zn

This European Standard was approved by CEN on 25 December 2010.

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Foreword

This document (EN 15297:2011) has been prepared by 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of an identical text or by endorsement, at the latest by August 2011, and conflicting national standards shall be withdrawn at the latest by August 2011.

This document supersedes CEN/TS 15297:2006.

In the pre-normative project BIONORM I&II a robustness test has been performed to find out if all critical parameters in the standard were addressed. Based on the results of that test it has been concluded that all critical parameters were covered. Only minor technical changes were necessary which have been implemented in the revised text. The revision also includes a change of deliverable from Technical Specification to European Standard and updated normative references.

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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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 EN 14961-1 [1]. In this European 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).

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1 Scope

This European Standard is intended for 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 specifies 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 as Selenium, Tin and Thallium is also possible with the method described in this European Standard.

2 Normative references

The following referenced documents are indispensable for the application 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.

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

EN 14588:2010, *Solid biofuels — Terminology, definitions and descriptions*

EN 14774-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

FprEN 14780, *Solid biofuels — Methods for sample preparation*

EN 15296, *Solid biofuels — Conversion of analytical results from one basis to another*

EN ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885:2007)*

EN ISO 11969, *Water quality — Determination of arsenic — Atomic absorption spectrometric method (hydride technique) (ISO 11969:1996)*

EN ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements (ISO 17294-2:2003)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 14588:2010 and the following apply.

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 realization 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

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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 abbreviations**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

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4.2 Abbreviations

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

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 determined with suitable instruments.

6 Reagents

6.1 General

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.

6.2 Water

Water containing negligible amounts of the minor elements i.e. amounts that do not contribute significantly to the determinations. Deionised water or doubly distilled water will normally fulfil this requirement.

NOTE 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 $\mu\text{S}/\text{cm}$.

6.3 Hydrofluoric acid (HF)

40 % (w/w), $\rho = 1,13 \text{ g/ml}$

CAUTION — Hydrofluoric acid may lead to health hazards.

6.4 Hydrogen peroxide (H_2O_2)

30 % (w/w), $\rho = 1,11 \text{ g/ml}$

6.5 Nitric acid (HNO_3)

≥ 65 % (w/w), $\rho = 1,41 \text{ g/ml}$

6.6 Boric acid (H_3BO_3)

4 % (w/w)

6.7 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, NBS 1571 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 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.

7 Apparatus

7.1 Heating oven or heating block suitable for the decomposition system in use.

A resistance heated oven or heating block that can be used at a temperature of at least 220 °C and an accuracy of ± 10 °C.

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