SLOVENSKI STANDARD
SIST EN 17351:2020

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Bioizdelki - Določevanje kisika z uporabo elementarnega analizatorja

Bio-based products - Determination of the oxygen content using an elemental analyser

Biobasierte Produkte - Sauerstoffgehalt - Bestimmung des Sauerstoffgehaltes unter Verwendung eines Elementaranalysators

Produits biosourcés - Teneur en oxygène - Détermination de la teneur en oxygène à laide dun analyseur déléments

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Bio-based products - Determination of the oxygen content using an elemental analyser

Produits biosourcés - Détermination de la teneur en oxygène à l'aide d'un analyseur élémentaire

Biobasierte Produkte - Bestimmung des Sauerstoffgehaltes unter Verwendung eines Elementaranalysators

This European Standard was approved by CEN on 9 October 2019.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (EN 17351:2020) has been prepared by Technical Committee CEN/TC 411 “Bio-based products”, the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by July 2020, and conflicting national standards shall be withdrawn at the latest by July 2020.

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 has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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Introduction

Bio-based products from forestry and agriculture have a long history of application, such as paper, board and various chemicals and materials. The last decades have seen the emergence of new bio-based products in the market. Some of the reasons for the increased interest lie in the benefits of bio-based products in relation to the depletion of fossil resources and climate change. Bio-based products may also provide additional product functionalities. These developments have triggered a wave of innovation with the development of knowledge and technologies allowing new transformation processes and product development.

Acknowledging the need for common standards for bio-based products, the European Commission issued Mandate M/492[1)], resulting in a series of standards developed by CEN/TC 411, with a focus on bio-based products other than food, feed and biomass for energy applications.

The standards of CEN/TC 411 “Bio-based products” provide a common basis on the following aspects:

— common terminology;

— bio-based content determination;

— life Cycle Assessment (LCA);

— sustainability aspects;

— declaration tools.

It is important to understand what the term bio-based product covers and how it is being used. The term ‘bio-based’ means 'derived from biomass'. Bio-based products (bottles, insulation materials, wood and wood products, paper, solvents, chemical intermediates, composite materials, etc.) are products which are wholly or partly derived from biomass. It is essential to characterize the amount of biomass contained in the product by, for instance, its bio-based content or bio-based carbon content.

The bio-based content of a product does not provide information on its environmental impact or sustainability, which may be assessed through LCA and sustainability criteria. In addition, transparent and unambiguous communication within bio-based value chains is facilitated by a harmonized framework for certification and declaration.

This document has been developed with the aim to specify the method for the determination of oxygen content in bio-based products using an elemental analyser. This document provides the reference test methods for laboratories, producers, suppliers and purchasers of bio-based products. It may be also useful for authorities and inspection organizations.

Part of the research leading to this document has been performed under the European Union Seventh Framework Programme OpenBio (see https://www.biobasedeconomy.eu/).

[1)] A mandate is a standardization task embedded in European trade laws. Mandate M/492 is addressed to the European Standardization bodies, CEN, CENELEC and ETSI, for the development of horizontal European Standards for bio-based products.

1 Scope

This document specifies a direct method for the determination of the total oxygen content in bio-based products using an elemental analyser. The scope is limited to products containing elements carbon, hydrogen, oxygen, nitrogen, chloride, bromide and iodide without fluoride, representing at least 95 % of the composition of the product to be analysed.

NOTE 1 Bio-based materials can contain both organic and inorganic components. The oxygen content might originate both from the organic and/or the inorganic components. The inorganic components are not bio-based but will nevertheless contribute to the amount of oxygen determined by the following prescribed methods and therefore influence the results in terms of oxygen content. According to the current state of the art, it is not possible by isotopic measurements to establish a distinction between oxygen originating from biomass and oxygen originating from non-biomass.

NOTE 2 Although this document has been drafted for the purpose of the determinations dealing with bio-based content, it can be also used as a standalone standard for determination of oxygen in organic compounds.

NOTE 3 For the purposes of this document, the unit "% (*m/m*)" is used to represent the oxygen content of a material.

NOTE 4 The method specified in this document involves a direct measurement method for the determination of oxygen content. This method contains many similarities with the ASTM D5622 [1] standard for gasoline and methanol fuels. The method specified in this document is specifically developed and validated for bio-based products. In addition, this method provides more accurate and unadulterated measured values for oxygen in contrast to indirect measurement methods for the determination of oxygen (e.g. ASTM D 3176 [2]).

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.

EN 16575, *Bio-based products - Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 16575 and the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

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

3.1
laboratory sample
sub-quantity of a sample suitable for laboratory tests

3.2
sample
quantity of material, representative of a larger quantity for which the property is to be determined

3.3
sample preparation
action taken to obtain representative analysis samples or test portions from the original sample

4 Principle

One of the major components of bio-based products is oxygen. The amount of oxygen is determined using an elemental analyser. The dried sample is pyrolyzed in an inert gas stream (e.g. Helium, Argon, Nitrogen) in a reaction column at a suitable temperature. The gaseous pyrolysis products are exposed to nickel activated carbon, carbon black or glassy carbon at typical temperatures as shown in Table 1. The oxygen present in the pyrolysis gases is transformed to carbon monoxide. The formed water is removed using a column filled with magnesium perchlorate. The carbon monoxide is then separated on a column and measured using a thermal conductivity detector or a NDIR CO detector.

Table 1 — Reactant and pyrolysis temperatures

Reactant	Pyrolysis temperatures
Nickel activated carbon	1 060 °C
Carbon black	1 100 °C
Glassy carbon	1 400 °C

NOTE There is a restriction to components containing fluorine. Fluorine is forbidden in oxygen analysis, it attacks the glass according the following reaction: $F+SiO_2 \rightarrow SiF_2 + O_2$. As a result of that reaction, the oxygen concentration determined by the measurement will be higher than the actual value.

Some inorganic compounds containing oxygen will also contribute to the measured oxygen concentration in the sample (e.g. carbonates). The influences of these compounds should be assessed by analysing the pure inorganic compound.

5 General requirements for the determination of the oxygen content

One of the major components of bio-based products is oxygen. The amount of oxygen can be determined using an elemental analyser. The oxygen present in water (as part of the bio-based product) is also analysed with this method and, therefore, the sample shall be dried before analyses when possible. When drying is not possible, the oxygen content shall be corrected for the water content.

Solid samples that are air-dried usually have a water content below 5 %. Analyses of the air-dried sample is possible. However, the analyses result shall be corrected for the water content in the air-dried sample. Formula (2) in Clause 10 shall be used for this correction.

For liquid samples with a water content below 5 %, analysis may be done directly on the as-received sample as well. A correction shall then be done according to Formula (2). Water content determination of liquid samples may be done using standard techniques (e.g. Karl Fischer titration, Dean and Stark distillation in toluene).

For samples with a water content above 5 %, the water shall be removed.

CAUTION Samples containing volatile organic compounds cannot be dried without losses of these volatiles.

If there is no method available to remove the water content from the sample or when the sample contains volatile organic compounds, the oxygen containing components in the individual raw materials present in the product shall be analysed separately. The oxygen content of the product may be calculated by recombination of the individual results of the raw materials.

6 Reagents

6.1 Standard reagents with different oxygen content shall be used to check calibration. See Table 2 for examples.

Table 2 — Examples of reagents with different oxygen content

Reagent	Molecular formula	Oxygen content
Benzoic acid	C_6H_5COOH	26,20 *% (m/m)* O
Tris (hydroxymethyl) aminomethane	$C_4H_{11}NO_3$	39,62 *% (m/m)* O
Acetanilide	C_8H_9NO	18,40 *% (m/m)* O
Valine		27,31 *% (m/m)* O
Tryptophan		15,67 *% (m/m)* O
Salycilic acetic acid		35,52 *% (m/m)* O

6.2 Helium, Argon or Nitrogen gas, with a purity of 99,995 %

7 Apparatus

7.1 Elemental analyser, suitable for oxygen determination;

IMPORTANT A number of elemental analysers are equipped with a sample storage system that is flushed with a dry gas. With these analysers, absorption of adherent water to the sample container and dried sample is prevented. However, if the sample storage system is in direct contact with the lab air and the sample containers are not sealed tight, the water content of a recently air-dried sample shall be used for water correction.

7.2 Chromatographic hardware and (optionally) software ;

7.3 Packed pyrolysis tube;

7.4 Separation column (e.g. Porapak™ [2] QS 1,0 m x 6 mm OD);

7.5 Sample containers, as per manufacturers recommendations (for example silver capsules pressed, smooth wall silver capsules, silver foil discs)

7.6 Microbalance, resolution 0,001 mg;

7.7 Milling device.

[2] PoraPak is a trademark of Waters. Porapak is a family of polymer-based chromatography products for clean-up of synthetic reactions. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.