SLOVENSKI STANDARD
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Bioizdelki - Ugotavljanje deleža 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 l□aide d□un analyseur d□éléments

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

Produits biosourcés - Teneur en oxygène - Détermination de la teneur en oxygène à l¿aide d¿un analyseur d¿éléments

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

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

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Contents

Page

European foreword

This document (prEN 17351:2019) has been prepared by Technical Committee CEN/TC 411 "Bio-based products", the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

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

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 bio-based products' benefits in relation to the depletion of fossil resources and climate change. Bio-based products may also provide additional product functionalities. This has 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 product materials and 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 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 method for the determination of the oxygen content in bio-based products using an elemental analyser. The scope is limited to products containing elements C, H, O, N, Cl, Br and I without F, representing at least 95 % of the composition of the product to be analysed.

NOTE 1 Bio-based materials can contain inorganic components. Oxygen in these inorganic components is 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.

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 term "% (m/m)" is used to represent the mass (μ) of a material.

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 http://www.iso.org/obp

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
all the actions 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 1 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 measurement is 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 is also measured, the sample should therefore when possible be dried. 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 %. Analysing the air-dried sample is possible, the correction for the water content of the air-dried sample shall be calculated with Formula (2) in Clause 10.

For liquid samples with a water content below 5 %, analysis may be directly done as well. A correction shall 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 higher water content the water present shall be removed.

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

When no options to remove the water content from the sample are available, 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	C6H5COOH	26,20 % O (m/m)
Tris (hydroxymethyl) aminomethane	C4H11NO3	39,62 % O (m/m)
Acetanilide	C8H9NO	18,4 % O (m/m)
Valine		27,31 % O (m/m)
Tryptophan		15,67 % O (m/m)
Salycilic acetic acid		35,52 % O (m/m)

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 software (if necessary);

7.3 Packed pyrolysis tube;

7.4 Separation column (e.g. Porapak 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.

8 Sampling

Take a homogeneous and representative sample, if possible dried in advance. This laboratory sample shall be representative of the product under consideration. If available, product sampling procedures for the determination of the elemental composition shall be used and the details shall be documented.

NOTE An informative list of suitable standardized sampling methods is given in EN 16785-1.

9 Measurement procedure

9.1 Sample preparation

Mill a solid laboratory sample to a particle size less than 0,25 mm.

Prepare the analyser for analysis according the manufacturer’s procedure.

9.2 Calibration

Calibrate the elemental analyser using traceable standards, possibly chosen among the ones having an oxygen content as close as possible to the one of the sample under test. (see examples in Table 2).

Weigh between 1 to 3 mg of the calibration standards in the sample containers with an accuracy of ± 0,01 mg. Analyse at least one calibration standard four times and determine the response factor. The relative standard deviation of these measurements shall be less than 5 %. If the relative standard deviation is higher, try to find the cause of the deviation and repeat the calibration procedure until the desired repeatability is obtained. Analyse two calibration standards at the end of the sample run.

9.3 Measurement

9.3.1 Generic measurement

Weigh between 1 to 3 mg of the homogeneous laboratory sample in the suitable sample container with an accuracy of ± 0,01 mg and tightly close the container using a pair of tweezers. Put the sample containers in the auto sampler. Add a calibration standard after every 10 samples. A deviation of response factor of less than 5 % is acceptable. If the deviation is higher, try to find the cause of this deviation and repeat the analysis run.

Analyse each sample and calibration standard(s) after 10 samples in duplicate.

If the instrument has not been used for more than 30 min, then start a new run with two calibration samples. Analyse a quality control material regularly throughout the sequence to monitor instrumental performance, especially drift.

9.3.2 Hygroscopic material

For hygroscopic samples, the following procedure is recommended.

Weigh the samples in the sample containers (1-3 mg ± 0,01 mg) and loosely close the sample containers using tweezers. Place the sample containers into a desiccator containing phosphorous pentoxide as drying agent and evacuate the desiccator.

After a period of 7 days, remove the sample containers from the desiccator, crimp the sample containers completely closed and immediately transfer them to the sealed carousel autosampler.

Flush the autosampler with a dry gas (e.g. helium) to ensure complete removal of atmospheric water introduced during sample loading (alternatively the autosampler may enable evacuation using a vacuum pump to remove water).

Each sample should be prepared in duplicate. Analyse a calibration standard, again in duplicate after 10 samples.

If the instrument has not been used for more than 30 min, then start a new run with two calibration samples. Analyse a quality control material regularly throughout the sequence to monitor instrumental performance, especially drift.

9.4 Blank determination

Use the blank procedure as given by the manufacturer.

If no procedure is available, then determine the blank value of the elemental analyser using an empty sample container at the beginning and end of each sequence of analyses. Additionally, a new blank value shall be determined for each batch of sample containers.

If it can be demonstrated that the blank value of the used sample containers are always neglectable, blank value analysis may be limited to determination when a new batch of sample containers is used.