



SLOVENSKI STANDARD
oSIST prEN 15936:2020
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Blato, obdelani biološki odpadki, tla in odpadki - Določevanje celotnega organskega ogljika (TOC) s suhim sežigom

Sludge, treated biowaste, soil and waste - Determination of total organic carbon (TOC) by dry combustion

Schlamm, behandelte Bioabfall, Boden und Abfall - Bestimmung des gesamten organischen Kohlenstoffs (TOC) mittels trockener Verbrennung

Boues, bio-déchets traités, sols et déchets - Détermination de la teneur en carbone organique total (COT) par combustion sèche

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Sludge, treated biowaste, soil and waste - Determination of total organic carbon (TOC) by dry combustion

Boues, bio-déchets traités, sols et déchets -
Détermination de la teneur en carbone organique total
(COT) par combustion sèche

Schlamm, behandelter Bioabfall, Boden und Abfall -
Bestimmung des gesamten organischen Kohlenstoffs
(TOC) mittels trockener Verbrennung

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 444.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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prEN 15936:2020 (E)

European foreword

This document (prEN 15936:2020) has been prepared by Technical Committee CEN/TC 444 “Environmental characterization of solid matrices”, the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 15936:2012.

This document combines methods from EN 15936:2012 and EN 13137:2001.

The main changes compared to the previous edition are as follows:

- New composition of the substances in control mixture A [6.10] was defined and the recovery requirement [9.3] was adapted to the results of a lab trial.
- Annex C – “Determination of total organic carbon (TOC) in solid samples using the suspension method” was skipped.
- The text was editorially revised.

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Introduction

This document is applicable and validated for several types of matrices as indicated in Table 1 (see also Annex A for the results of the validation).

Table 1 — Matrices for which this document is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Compost, Fresh Compost
Soil	Sludge amended soil, Agricultural soil
Waste	Filter cake, Bottom ash, Electro-plating sludge, Dredged sludge, Rubble

WARNING — Persons using this document should be familiar with usual laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

prEN 15936:2020 (E)**1 Scope**

This document specifies two methods for the determination of total organic carbon (TOC) in sludge, treated biowaste, soil, waste and sediment samples containing more than 1 g carbon per kg of dry matter (0,1 %).

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 15002, *Characterization of waste - Preparation of test portions from the laboratory sample*

EN 15934, *Sludge, treated biowaste, soil and waste - Calculation of dry matter fraction after determination of dry residue or water content*

EN 16179, *Sludge, treated biowaste and soil - Guidance for sample pretreatment*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

— ISO Online browsing platform: available at <https://www.iso.org/obp/ui>

— IEC Electropedia: available at <http://www.electropedia.org/>

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3.1 total carbon

TC
quantity of carbon present in the sample in the form of organic, inorganic and elemental carbon

3.2 total inorganic carbon

TIC
quantity of carbon that is liberated as carbon dioxide by acid treatment. Typically, the TIC represents the carbonates present in a sample

3.3 total organic carbon

TOC
quantity of carbon that is converted into carbon dioxide by combustion and which is not liberated as carbon dioxide by acid treatment

4 Principle**4.1 Method A (indirect procedure)**

In this procedure, the TOC is obtained by the difference between the results of the measurements of TC and TIC.

The total carbon (TC) present in the sample is converted into carbon dioxide by combustion in an oxygen-containing gas flow free of carbon dioxide. To ensure complete combustion, catalysts and/or

modifiers can be used. The released amount of carbon dioxide is measured e.g. by infrared spectrometry, thermal conductivity detection, or other suitable techniques.

The TIC is determined separately from another sub-sample by means of acidification and purging of the released carbon dioxide. The carbon dioxide shall be measured by one of the techniques mentioned above.

NOTE Alternatively, for soil, the carbon present as carbonate (TIC) can be determined according to e.g. ISO 10693 (volumetric method).

4.2 Method B (direct procedure)

In this procedure, the TIC present in the sample are previously removed by treating the sample with acid. The carbon dioxide released by the following combustion step is measured by one of the techniques mentioned in 4.1 and indicates the TOC directly.

4.3 Applicability of Methods A or B

Methods A and B have the same applicability for the determination of TOC and/or the determination of the TIC to TOC ratio. In samples with relatively high TIC contents method B should be applied.

Method B can lead to incorrect results in the following cases:

- the sample contains volatile substances that evaporate during the acidification (e.g. volatile hydrocarbons from sludge of oil separators);
- side reactions between the sample and the acid take place (e.g. decarboxylation, volatile reaction products).

NOTE The quality of results of Method B is dependent on experience and practice, especially regarding the steps before the determination of TOC. Use of automatic dispensing units regarding removal of TIC prior to determination of TOC can improve the performance of Method B.

5 Interferences

Volatile organic substances can be lost during sample preparation. If necessary, the carbon content resulting from volatile organic substances shall be determined separately.

Depending on the laboratory experience with samples containing high amounts of TIC, the procedures can lead to unreliable TOC results if the TIC to TOC ratio is very high (e.g. ≥ 10).

Depending on the detection method used, different interferences can occur, for instance:

- the presence of cyanide can interfere with the coulometric detection of TIC by modifying the pH value (dissolution of HCN);
- high content of halogenated compounds can lead to an overestimation of TOC when coulometric detection is used; in some cases the classical silver or copper trap can be insufficient to absorb all halides.

When present, elemental carbon, carbides, cyanides, cyanates, isocyanates, isothiocyanates and thiocyanates are determined as organic carbon using the methods described in this document. An interpretation of the measured value can therefore be problematic in cases where the sample contains relevant levels of the above-mentioned components. If needed, these components shall be determined separately by means of a suitable validated method and be recorded in the test report.

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6 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

Hygroscopic substances shall be stored in a desiccator.

6.1 Calcium carbonate, CaCO₃.**6.2 Sodium carbonate, Na₂CO₃, anhydrous.****6.3 Tetrasodium ethylenediamine tetraacetate-tetra-hydrate, Na₄-EDTA · 4 H₂O (C₁₀H₁₂N₂O₈Na₄ · 4 H₂O), heated at 80 °C for 2 h.**

Other forms of Na₄-EDTA hydrates may be used if the water content is exactly known. In these cases, the composition of the control mixtures shall be recalculated accordingly (see also 6.10 and 6.11).

6.4 Potassium hydrogen phthalate, C₈H₅O₄K.**6.5 Acetanilide, C₈H₉NO.****6.6 Atropine, C₁₇H₂₃NO₃.****6.7 Spectrographic graphite powder, C.****6.8 Sodium salicylate, C₇H₅O₃Na.****6.9 Aluminium oxide, Al₂O₃, neutral, granular size < 200 µm, annealed at 600 °C.****6.10 Control mixture A, prepared from sodium carbonate (6.2), Na₄-EDTA · 4 H₂O (6.3) and aluminium oxide (6.9) in a mass ratio of 2,34 : 1,00 : 7,28.**

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The mixture shall be homogenized. It should contain 25,00 g/kg TIC and 25,00 g/kg TOC (e.g. 22,06 g of sodium carbonate, 9,41 g Na₄-EDTA · 4 H₂O, 68,53 g of aluminium oxide).

6.11 Control mixture B, prepared from sodium salicylate (6.8), calcium carbonate (6.1), Na₄-EDTA · 4 H₂O (6.3) and aluminium oxide (6.9) in a mass ratio of 1,00 : 4,36 : 1,97 : 8,40.

The mixture shall be homogenized. It should contain 33,3 g/kg TIC and 66,6 g/kg TOC (e.g. 6,36 g of sodium salicylate, 27,78 g of calcium carbonate, 12,50 g of Na₄-EDTA · 4 H₂O, 53,36 g of aluminium oxide).

6.12 Non-oxidizing mineral acid, used for carbon dioxide expulsion, e.g. phosphoric acid H₃PO₄ (w = 85 %).

NOTE Due to potential corrosion by hydrochloric acid, phosphoric acid is preferred for TIC determination in Method A (9.1.3). Due to potential formation of P₄O₁₀ during combustion, hydrochloric acid is preferred for removal of inorganic carbon in Method B (10.1.2).

6.13 Carrier gas, e.g. synthetic air, nitrogen, oxygen or argon, free of carbon dioxide and organic impurities in accordance with the manufacturer's instructions.

7 Apparatus

7.1 Precision balance, accurate to at least 0,5 % of test portion weight.

7.2 Equipment for determination of carbon in solids, with relevant accessories.

7.3 Purging unit for TIC determination, for Method A only.

7.4 Vessels, made of e.g. ceramic, silica, quartz, silver or platinum.

NOTE Tin and nickel vessels are not acid-resistant. Tin vessels are suitable only for Method A.

8 Sample pre-treatment

Pre-treat the sample according to EN 16179 or EN 15002, if not otherwise specified.

For solid materials, dried samples shall be used.

NOTE 1 The drying method can affect the result.

Moist or paste-like waste samples may be mixed with aluminium oxide (6.9) until granular material is obtained and then be comminuted. In this case, the ratio of aluminium oxide to sample shall be considered in the calculation of TOC (according to 9.4 or 10.4).

If samples contain – depending on the accuracy of the method – negligible amounts of volatile compounds except water, the samples may be dried.

NOTE 2 For waste samples, the homogeneity is important and often the aluminium oxide step is helpful. More information is given in Annex B.

9 Procedure – Method A (indirect method)

9.1 Determination

9.1.1 General

The mass of the test portion should be as large as possible and shall be chosen so that the liberated quantity of carbon dioxide lies within the working range of the equipment/calibration.

9.1.2 Determination of the TC

The sample prepared according to Clause 8 is weighed into a suitable vessel (7.4). To minimize carbon blank values the vessel may be pre-treated by heating (in a muffle furnace or the TC apparatus itself).

The sample is combusted or decomposed in a flow of carrier gas containing oxygen (6.13).

The combustion temperature should be high enough to convert all carbon completely to carbon dioxide.

NOTE For samples containing carbonates, which are difficult to decompose, e.g. barium carbonate, the release of the carbon dioxide can be improved by increasing the temperature or by the use of modifiers, e.g. tin, copper (see B.1)

The temperature range of commercially available instruments is between 900 °C and 1500 °C.

During the combustion of reactive samples, explosion or fuming can be prevented by covering the sample with inert material e.g. silica sand.

The amount of carbon dioxide released during the combustion is measured e.g. by infrared spectrometry, thermal conductivity detection, or other suitable techniques, and is expressed as total carbon (TC).