



# SLOVENSKI STANDARD

## SIST EN 15936:2022

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**Tla, odpadki, obdelani biološki odpadki in blato - Določevanje celotnega organskega ogljika (TOC) s suhim sežigom**

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

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

Sols, déchets, biodéchets traités et boues - Dosage du carbone organique total (COT) par combustion sèche

**Ta slovenski standard je istoveten z: EN 15936:2022**

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13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

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## Soil, waste, treated biowaste and sludge - Determination of total organic carbon (TOC) by dry combustion

Sols, déchets, biodéchets traités et boues - Dosage du carbone organique total (COT) par combustion sèche

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
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<b>Contents</b>	<b>Page</b>
European foreword.....	4
Introduction .....	5
1 Scope .....	6
2 Normative references .....	6
3 Terms and definitions .....	6
4 Principle .....	6
4.1 Method A (indirect procedure) .....	6
4.2 Method B (direct procedure) .....	7
5 Interferences .....	7
6 Reagents .....	7
7 Apparatus.....	8
8 Sample pre-treatment .....	8
9 Procedure – Method A (indirect method) .....	9
9.1 Determination .....	9
9.1.1 General.....	9
9.1.2 Determination of the TC .....	9
9.1.3 Determination of the TIC.....	9
9.2 Calibration .....	10
9.3 Control measurements.....	10
9.4 Calculation and expression of results.....	11
10 Procedure Method B (direct method).....	12
10.1 Determination .....	12
10.1.1 General.....	12
10.1.2 Removal of the inorganic carbon and determination of the TOC .....	12
10.2 Calibration .....	12
10.3 Control measurements.....	13
10.4 Calculation and expression of results.....	13
11 Performance data.....	14
12 Expression of results.....	14
13 Test report.....	14
Annex A (informative) Repeatability and reproducibility data .....	15
A.1 Materials used in the interlaboratory comparison study .....	15
A.2 Interlaboratory results .....	16
Annex B (informative) Factors influencing dry combustion methods.....	19
B.1 Influence of temperature and modifiers on the decomposition of barium carbonate as an example for a refractory compound .....	19
B.2 Recovery of the control mixture A .....	19

<b>B.3</b>	<b>Influence of aluminium oxide or sodium sulfate used for sample preparation for the recovery of TOC .....</b>	<b>20</b>
<b>B.4</b>	<b>Influence of TIC:TOC ratio on the recovery and the coefficient of variation.....</b>	<b>21</b>
<b>B.5</b>	<b>Method B: Influence of the temperature during the removal of inorganic carbon on the recovery of TOC.....</b>	<b>22</b>
	<b>Bibliography .....</b>	<b>23</b>

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SIST EN 15936:2022

<https://standards.iteh.ai/catalog/standards/sist/5db04676-3683-4c77-b352-96f6523541e1/sist-en-15936-2022>

**EN 15936:2022 (E)****European foreword**

This document (EN 15936:2022) has been prepared by Technical Committee CEN/TC 444 “Environmental characterization of solid matrices”, 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 August 2022, and conflicting national standards shall be withdrawn at the latest by month August 2022.

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 supersedes 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.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations 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, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## 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). The results in this document are expressed in % C in relation to the dry mass (dm).

**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.

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

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**EN 15936:2022 (E)****1 Scope**

This document specifies two methods for the determination of total organic carbon (TOC) in sludge, treated biowaste, soil and waste samples containing more than 0,1 % carbon in relation to the dry mass (dm).

NOTE This method can also be applied to other environmental solid matrices, provided the user has verified the applicability.

**2 Normative references**

There are no normative references in this document.

**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/>
- IEC Electropedia: available at <https://www.electropedia.org/>

**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

Note 1 to entry: 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.



## 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.

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

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.  $\geq 10$ ).

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.

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

- Volatile organic substances (e.g. volatile hydrocarbons from sludge of oil separators) can be lost during sample preparation especially during the acidification. If necessary, the carbon content resulting from volatile organic substances shall be determined separately.
- side reactions between the sample and the acid take place (e.g. decarboxylation, volatile reaction products).

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.

## 6 Reagents

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

Hygroscopic substances shall be stored in a desiccator.

**6.1 Calcium carbonate**,  $\text{CaCO}_3$ .

**6.2 Sodium carbonate**,  $\text{Na}_2\text{CO}_3$ , anhydrous.

**6.3 Tetrasodium ethylenediamine tetraacetate-tetra-hydrate**,  $\text{Na}_4\text{-EDTA} \cdot 4 \text{H}_2\text{O}$  ( $\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8\text{Na}_4 \cdot 4 \text{H}_2\text{O}$ ), heated at 80 °C for 2 h.

Other forms of  $\text{Na}_4\text{-EDTA}$  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**,  $\text{C}_8\text{H}_5\text{O}_4\text{K}$ .

**EN 15936:2022 (E)**

**6.5 Acetanilide**,  $C_8H_9NO$ .

**6.6 Atropine**,  $C_{17}H_{23}NO_3$ .

**6.7 Spectrographic graphite powder**, C.

**6.8 Sodium salicylate**,  $C_7H_5O_3Na$ .

**6.9 Aluminium oxide**,  $Al_2O_3$ , neutral, granular size < 200  $\mu m$ , annealed at 600 °C.

**6.10 Control mixture A**, prepared from sodium carbonate (6.2),  $Na_4$ -EDTA · 4  $H_2O$  (6.3) and aluminium oxide (6.9) in a mass ratio of 2,34 : 1,00 : 7,28.

The mixture shall be homogenized. It shall contain 2,5 % TIC and 2,5 % TOC (e.g. 22,06 g of sodium carbonate, 9,41 g  $Na_4$ -EDTA · 4  $H_2O$ , 68,53 g of aluminium oxide).

**6.11 Control mixture B**, prepared from sodium salicylate (6.8), calcium carbonate (6.1),  $Na_4$ -EDTA · 4  $H_2O$  (6.3) and aluminium oxide (6.9) in a mass ratio of 1,00 : 4,36 : 1,97 : 8,39.

The mixture shall be homogenized. It shall contain 3,3 % TIC and 6,6 % TOC (e.g. 6,36 g of sodium salicylate, 27,78 g of calcium carbonate, 12,50 g of  $Na_4$ -EDTA · 4  $H_2O$ , 53,36 g of aluminium oxide).

**6.12 Non-oxidizing mineral acid**, used for carbon dioxide expulsion, e.g. phosphoric acid  $H_3PO_4$  ( $w = 85$  %).

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  $P_4O_{10}$  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

The sample should be pre-treated according to EN 16179 or EN 15002, if not otherwise specified. The particle size shall be < 250  $\mu m$ . Foreign bodies or non-comminutable material should be separated from the sample and the weight and nature of the material should be recorded.

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 to a particle size  $< 250 \mu\text{m}$ . 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 requested for the method – negligible amounts of volatile compounds except water, the samples shall 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 shall 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

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

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

The combustion temperature shall 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  $900 \text{ }^\circ\text{C}$  and  $1\ 500 \text{ }^\circ\text{C}$ .

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).

#### 9.1.3 Determination of the TIC

The sample prepared according to Clause 8 is weighed into the purging unit (7.3) or in the sample vessel (7.4).

The system is closed gas-tight and flushed with carrier gas until no more carbon dioxide from ambient air is present. Then acid (6.12) is added and the carbon dioxide is stripped by purging or stirring and/or heating. The released carbon dioxide is transferred to the detector by the carrier gas.

The addition of wetting agents, e.g. surfactants, can improve wetting of the surface of the sample.

The addition of anti-foaming agents, e.g. silicone oil, can be helpful in the case of strongly foaming samples.

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

Samples containing persistent carbonates (e.g. concrete, cement) require treatment with hot acid for complete release of carbon dioxide according to manufacturers' instructions.