SLOVENSKI STANDARD
oSIST prEN 17505:2022

01-september-2022

Karakterizacija tal in odpadkov - Diferenciacija celotnega ogljika (TOC400, ROC, TIC900) v odvisnosti od temperature

Soil and waste characterization - Temperature dependent differentiation of total carbon (TOC400, ROC, TIC900)

Boden- und Abfallbeschaffenheit - Temperaturabhängige Unterscheidung von Gesamtkohlenstoff (TOC400, ROC, TIC900)

Caractérisation des sols et des déchets - Différentiation en fonction de la température du carbone total (COT400, COR, CIT900)

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Ta slovenski standard je istoveten z: **prEN 17505**

ICS:

13.030.10	Trdni odpadki	Solid wastes
13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

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2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 17505

July 2022

ICS 13.030.10; 13.080.10

English Version

Soil and waste characterization - Temperature dependent differentiation of total carbon (TOC400, ROC, TIC900)

Caractérisation des sols et des déchets - Différentiation en fonction de la température du carbone total (COT400, COR, CIT900)

Boden- und Abfallbeschaffenheit - Temperaturabhängige Unterscheidung von Gesamtkohlenstoff (TOC400, ROC, TIC900)

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

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

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European foreword

This document (prEN 17505:2022) 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.

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Introduction

Carbon occurs in soils and materials similar to soil in a variety of compounds and forms. When determining carbon in soils or soil-like materials, an overall determination of the different mass fractions is most feasible. The summarized declaration of carbon is yet done by differentiating organic and inorganic carbon (EN 15936, ISO 10694). In the proportion classified as “organic carbon”, a fraction of very stable highly aromatic and highly condensed carbon compounds can be present, sometimes in significant mass fractions. Since this black (pyrogenic) carbon is only very slowly decomposed and released, its environmental relevance has to be differently evaluated than the proportions of organic carbon which are faster chemical-biologically decomposed. The environmental relevance is estimated if e.g. the suitability of soils and soil-like materials for disposal in landfill is assessed. For a differentiated assessment, a separate declaration of the different mass fractions of organic, black (pyrogenic) and inorganic carbon is necessary. Using the specified temperature-gradient method and utilizing the combustion characteristic(s), the various bond types of carbon in soil and soil-like materials can be differentiated.

In respect of the hazard potential, the content of solely organically bonded carbon in solids determined with the described method can be important for disposal and/or recycling.

The method has been validated with the materials listed in Table 1, see also Annex A.

Table 1 — Materials used for validation

Material type	**Materials used for validation**
soils from natural material	**mineral soils** **soil with anthropogenic admixtures (urban soils)**
tailing material (tailings)	**tailing material from coal mining**
sediment	**sediment**
waste	**waste incineration ash** **foundry sand** **construction waste**

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1 Scope

This document specifies a method for the differentiated determination of the organic carbon content (TOC_{400}) which is released at temperatures up to 400 °C, the residual oxidizable carbon (ROC) (including e.g. lignite (brown coal), hard coal, charcoal, black carbon, soot) and the inorganic carbon (TIC_{900}) which is released at temperatures up to 900 °C.

The basis is the dry combustion or decomposition of carbon to CO_2 in the presence of oxygen or non-oxygen conditions using temperatures ranging from 150 °C to 900 °C in dry solid samples of sediment, soil, soil with anthropogenic admixtures and solid waste (see Table 1) with carbon contents of more than 1 g per kg (0,1 % C) (per carbon type in the test portion).

NOTE TIC includes the TIC measured after acid addition e.g. by ISO 10694 or EN 15936. TOC_{400} is the carbon black free portion of TOC measurement e.g. by ISO 10694 or EN 15936.

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:

IEC Electropedia: available at https://www.electropedia.org/

ISO Online browsing platform: available at https://www.iso.org/obp

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3.1
total organic carbon which is released up to 400 °C - TOC_{400}
quantity of carbon which is determined in the range between 150 °C to the 1st signal minimum at (400 ± 20) °C, in the case of dry combustion in the presence of oxygen

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Note 1 to entry: TOC_{400} is the carbon black free portion of TOC measured e.g. by ISO 10694 or EN 15936. This carbon fraction is important regarding the hazard potential for disposal and/or recycling.

3.2
residual oxidizable carbon measured at 600°C - ROC_{600}
quantity of carbon which is determined between the signal minima at (400 ± 20) °C and at (600 ± 20) °C, in the case of dry combustion in the presence of oxygen following method A (procedure see 8.4)

3.3
residual oxidizable carbon measured at 900°C - ROC_{900}
quantity of carbon which is determined during dry combustion in the presence of oxygen after the completed carbon release for the TOC_{400} and TIC_{900B} measurement at (900 ± 20) °C following method B (procedure see 8.5)

3.4
total inorganic carbon which is released up to 900 °C C in the presence of oxygen TIC_{900A}
quantity of carbon which is determined between the signal minima at (600 ± 20) °C and at (900 ± 20) °C, in the case of dry combustion in the presence of oxygen following method A (procedure see 8.4)

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3.5
total inorganic carbon which is released up to 900 °C during non-oxidizing conditions - TIC_{900B}
quantity of carbon which is determined during non-oxidizing conditions between the signal minima at (400 ± 20) °C and at (900 ± 20) °C before the ROC_{900B} measurement following method B (procedure see 8.5)

3.6
total carbon
TC
quantity of carbon present in the sample representing the sum of organic (TOC_{400}), inorganic (TIC_{900A} and TIC_{900B}) and residual oxidizable carbon (ROC_{600} or ROC_{900})

4 Principle

The determination of organic carbon (TOC_{400}), residual oxidizable carbon (ROC_{600} and ROC_{900}) and inorganic carbon (TIC_{900A} and TIC_{900B}) in solids is affected by means of thermal oxidation or decomposition of the different bond types of carbon at different temperatures to CO_2, if necessary, supported by changing between oxidizing and non-oxidizing carrier gases.

The application of the gradient method with a suitable temperature program allows the determination of organic carbon (TOC_{400}), residual oxidizable carbon (ROC) and inorganic carbon (TIC_{900}) and the calculation of total carbon (TC) by totalling these contents.

The final analysis of CO_2 can be performed with different methods, e.g. by means of infrared detection or CO_2 sensitive sensors.

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5 Interferences

5.1 Interference due to carbides

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Several carbides can interfere with this method.

5.2 Interference due to sulfur and nitrogen compounds

Depending on the measuring technique used, high contents of sulfur or nitrogen compounds can result in overestimations or underestimations. This can be controlled by means of selected standard samples (e.g. potassium sulfate, potassium nitrate). Furthermore, the information provided by the equipment manufacturer shall be considered.

5.3 Interference due to carbonates

The thermal stability of carbonates exhibits a great bandwidth (for examples see Figures 1, 2 and 3). Therefore, carbonates might be detected in both the TOC_{400} peak range and the ROC_{600} range. In the presence of certain carbonates or carbonate mixtures which decompose at low temperature ranges, the identification of the TIC_{900A} peak is sometimes difficult or impossible. Alternatively, the impact of carbonates on the TOC_{400} analysis can be determined by stripping with acid (e.g. Scheibler method EN ISO 10693).

For samples containing the more thermally stable carbonates, e.g. barium carbonate, the liberation of carbon dioxide can be improved by increasing temperature or using additives such as tungsten oxide.

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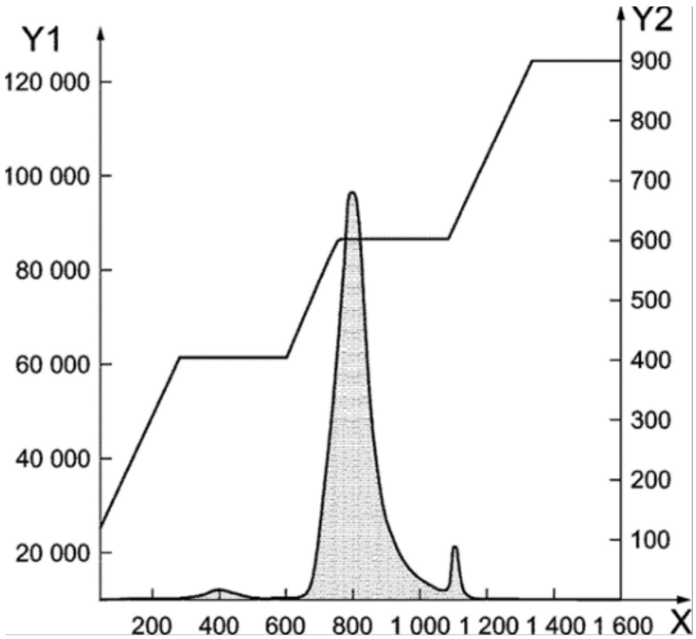

Key

X time
Y1 signal intensities
Y2 temperature in °C

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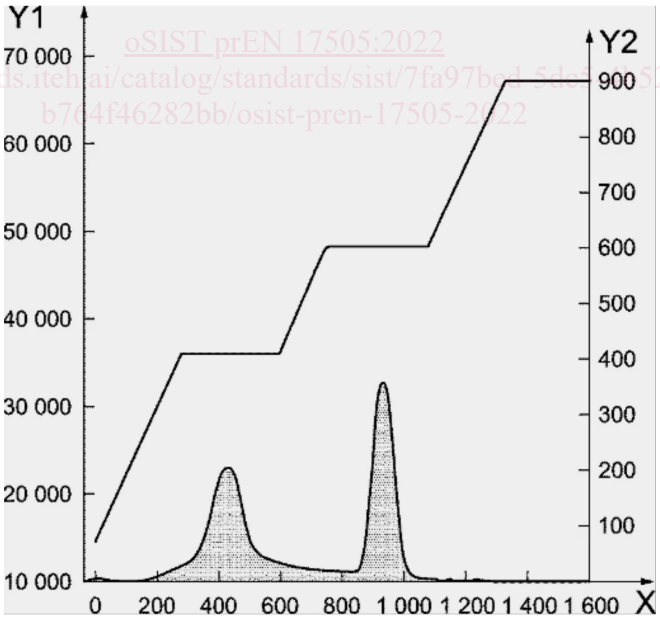
Figure 1 — Example diagram $FeCO_3$



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Key

X time
Y1 signal intensities
Y2 temperature in °C

Figure 2 — Example diagram $MnCO_3 \cdot fH_2O$

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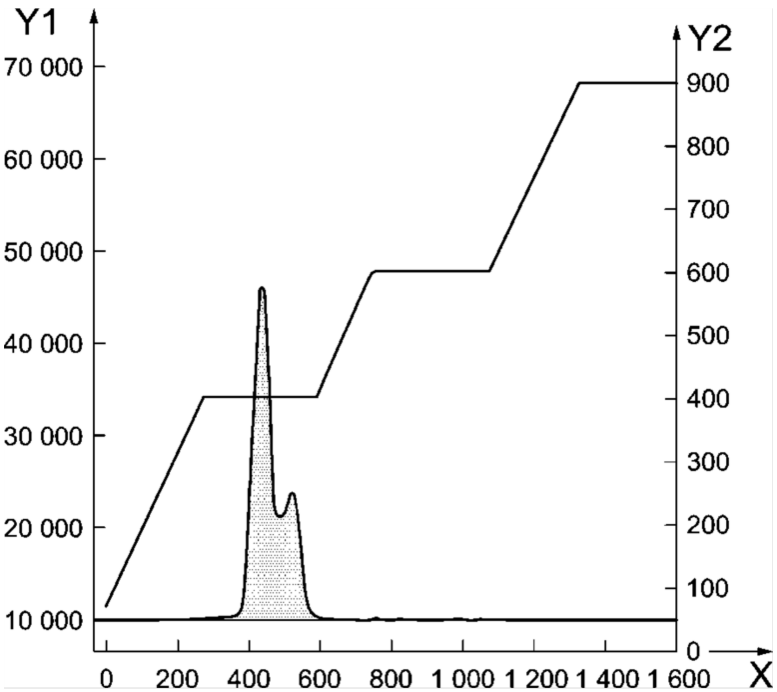

Key

X time
Y1 signal intensities
Y2 temperature in °C

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Figure 3 — Example diagram $PbCO_3$

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5.4 Peak does not reach the baseline

For some materials, the temperature plateau according to the temperature ramp does not last long enough and the peak does not reach the baseline (see Figure 4). A reasonable prolongation of the plateau at the temperature level can improve the result in terms of a significantly better return of the signal to the baseline (see Figure 5).

NOTE A homogeneous distribution of the sample in the combustion vessel optimizes the reaction with oxygen.

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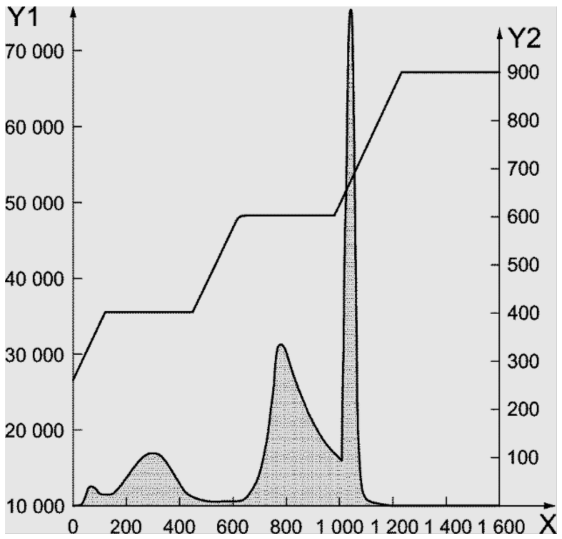

Key

X time in s
Y1 signal intensities
Y2 temperature in °C

Figure 4 — Example diagram for cases where peaks do not reach the baseline

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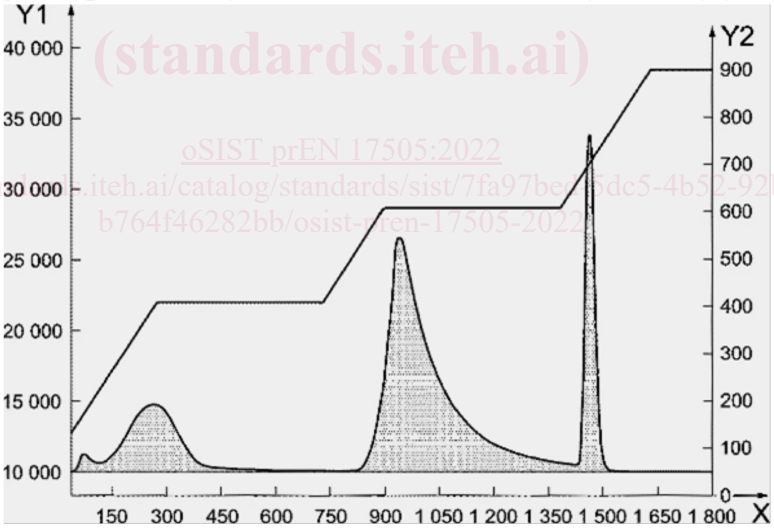

Key

X time in s
Y1 signal intensities
Y2 temperature in °C

Figure 5 — Example diagram for the prolongation of the temperature plateau so peaks can reach the baseline

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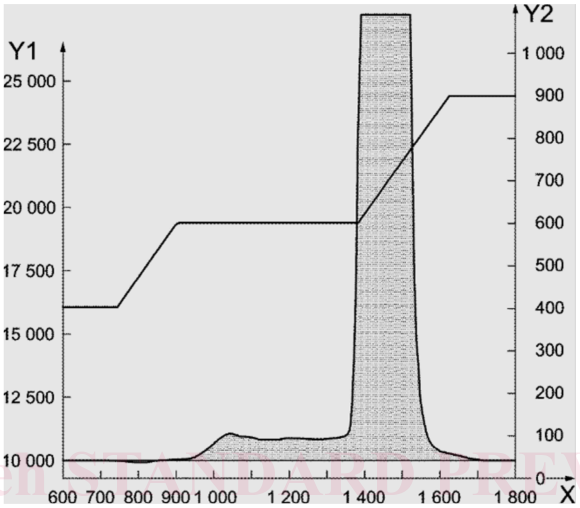
5.5 Difficulties in separating ROC_{600} peak and TIC_{900A} peak

If the temperature ramp does not allow the separation (resolution) of the ROC_{600} peak from the TIC_{900A} peak (see Figure 6), the influence of carbonates on the ROC_{600} analysis or of ROC_{600} on the TIC_{900A} measurement can be determined by stripping with acid (e.g. Scheibler EN ISO 10693). Alternatively, the method specified in 8.6 can be used. The method has to be documented with the measuring result.

In the case of deviating determination of TIC_{900A} by means of acid, the information provided by the equipment manufacturer should be consulted.



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Key

X time
Y1 signal intensities
Y2 temperature in °C

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Figure 6 — Difficulties in separating ROC_{600} and TIC_{900A} peaks

5.6 Interferences due to premature releases and deflagrations

During the combustion of reactive samples, deflagration or carbon black (soot) formation can occur, and it is also known that the remaining carbon might undergo premature ignition resulting in superposition (overlapping) and misidentification. This can be prevented by covering the sample with a layer of inert material, e.g. quartz sand or aluminium oxide.

5.7 Interferences due to catalytic active metal contents in samples

In waste samples from high temperature treatment with catalytic active metal contents can lead to overestimated TOC_{400} values.

6 Reagents

6.1 General

All reagents used shall be at least of analytical grade and shall be suitable for their specific purposes.