
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)

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ICS:

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

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EUROPEAN STANDARD
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English Version

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

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

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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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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (prEN 17505:2020) has been prepared by Technical Committee CEN/TC 444 “Test methods for environmental characterization”, 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
tailing material (tailings)	tailing material from coal mining
sludge	dredged sludge
sediment	sediment
waste	waste incineration ash foundry sands recycling material

1 Scope

This document specifies a method for the differentiated determination of the organic carbon content (TOC₄₀₀) 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 (TIC₉₀₀) which is released at temperatures up to 900 °C.

The basis is the dry combustion to CO₂ in a in the presence of oxygen using temperatures ranging from 150 °C to 900 °C in dry solid samples of 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).

Alternatively, the method specified in Annex B may be applied.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

3.1

total organic carbon which is released up to 400° C

TOC₄₀₀

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

Note 1 to entry: The TOC₄₀₀ corresponds to the content of organically bonded carbon excluding ROC. This carbon fraction is important regarding the hazard potential for disposal and/or recycling.

3.2

residual oxidizable carbon

ROC

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

Note 1 to entry: When using the alternative normative method according to Annex B, then the ROC is defined as the carbon determined during dry combustion in a current of oxygen after the TIC₉₀₀ measurement at (900 ± 20) °C.

Note 2 to entry: The *black carbon* is part of the ROC.

3.3

total inorganic carbon which is released up to 900° C

TIC₉₀₀

quantity of carbon present in the sample in the form of organic (TOC₄₀₀), inorganic (TIC₉₀₀) and black carbon (ROC)

prEN 17505:2020 (E)**3.4****total carbon****TC**

quantity of carbon present in the sample in the form of organic (TOC₄₀₀), inorganic (TIC₉₀₀) and black carbon (ROC)

4 Principle

The determination of organic carbon (TOC₄₀₀), residual oxidizable carbon (ROC) and inorganic carbon (TIC₉₀₀) in solid is effected by means of thermal oxidation or decomposition of the different bond types of carbon at different temperatures to CO₂, 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 (TOC₄₀₀), residual oxidizable carbon (ROC) and inorganic carbon (TIC₉₀₀) and the calculation of total carbon (TC) by totalling these contents.

The final analysis of CO₂ can be performed with different methods, e.g. by means of infrared detection or CO₂ sensitive sensors.

5 Interferences**5.1 Interference due to carbides**

Several carbides can interfere with this method.

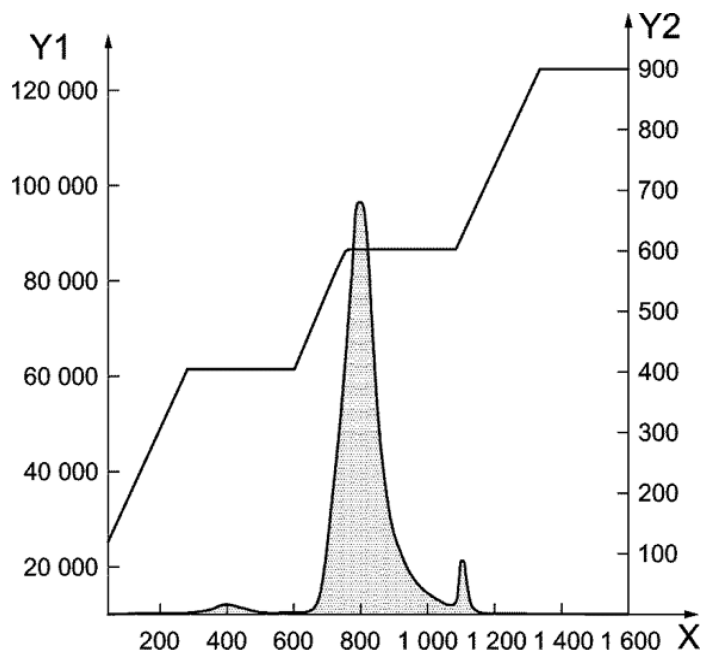
5.2 Interference due to sulfur and nitrogen compounds

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 TOC₄₀₀ peak range and the ROC range. In the presence of certain carbonates or carbonate mixtures which decompose at low temperature ranges, the identification of the TIC₉₀₀ peak is sometimes difficult or impossible. Alternatively, the impact of carbonates on the TOC₄₀₀ analysis can be determined by stripping with acid.

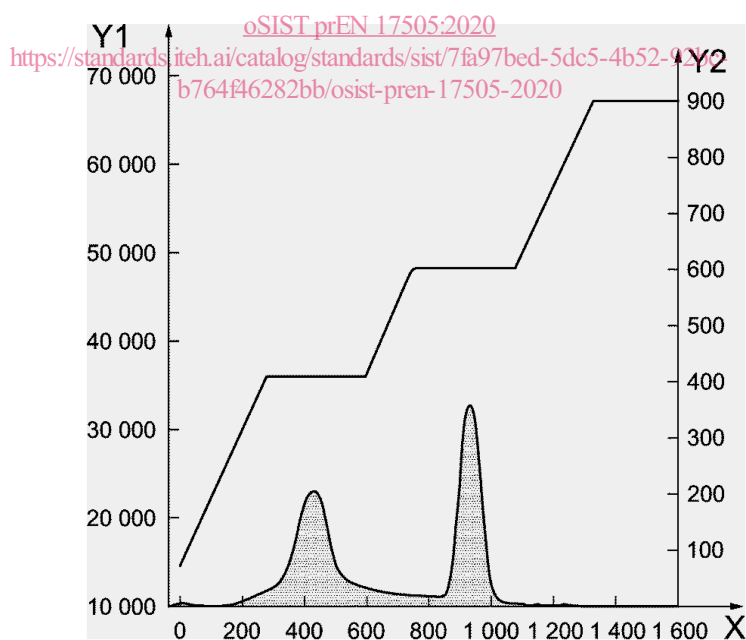
For samples containing the more thermally stable carbonates, e.g. barium carbonate, the liberation of carbon dioxide can be improved by increasing temperature or using additives such as tungsten oxide.

**Key**

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

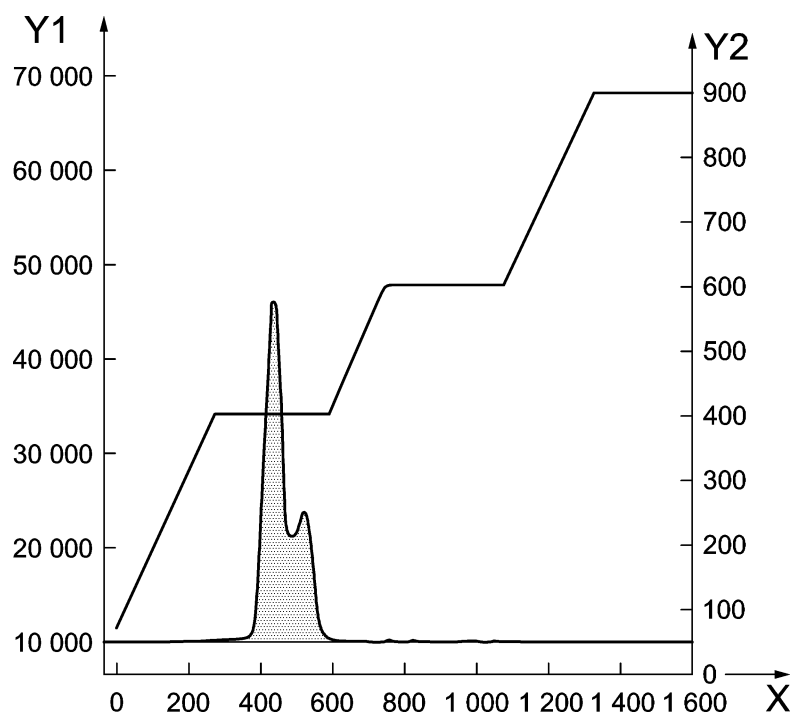
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Figure 1 — Example diagram FeCO_3 **Key**

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

Figure 2 — Example diagram $\text{MnCO}_3 \cdot f\text{H}_2\text{O}$

**Key**

X	time
Y1	signal intensities
Y2	temperature in °C

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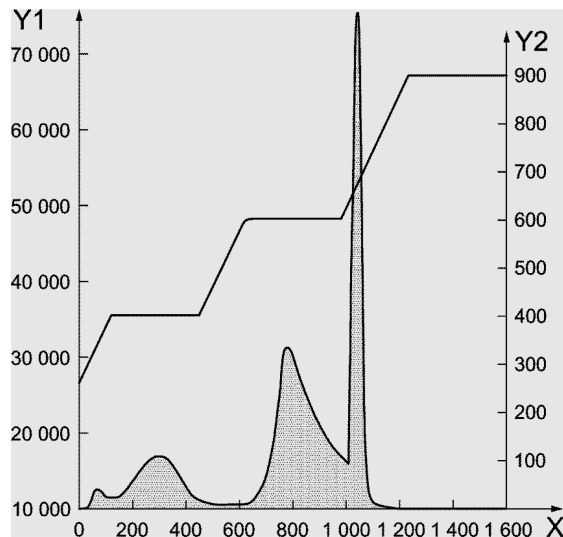
Figure 3 — Example diagram PbCO₃

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5.4 Peak does not reach the baseline

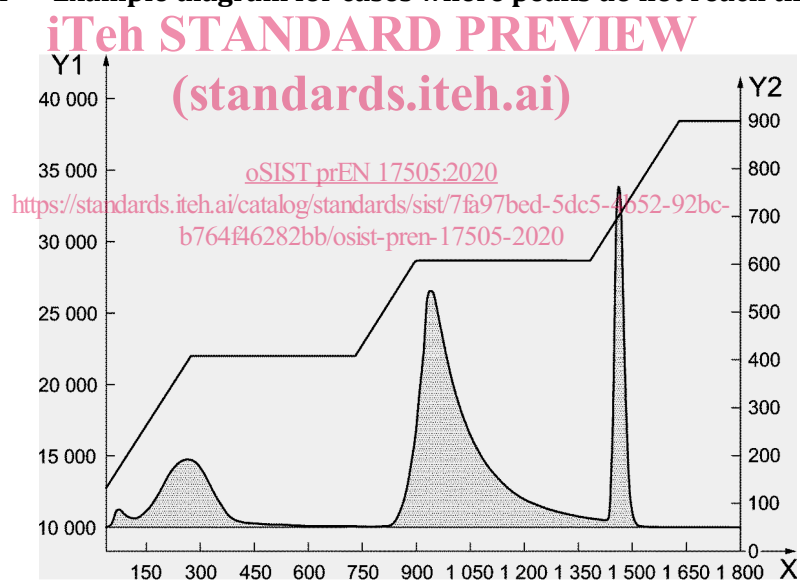
For some materials, the retention time according to the temperature ramp is not sufficient and the peak does not reach the baseline (see Figure 4). A reasonable prolongation of the retention time at the temperature level can improve the result in terms of a significantly better return of the signal to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

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

**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

**Key**

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

Figure 5 — Example diagram for the prolongation of retention time where peaks do not reach the baseline