



SLOVENSKI STANDARD
SIST EN 1484:1998

01-maj-1998

Kakovost vode - Navodila za določevanje celotnega organskega ogljika (TOC) in raztopljenega organskega ogljika (DOC)

Water analysis - Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)

Wasseranalytik - Anleitungen zur Bestimmung des gesamten organischen Kohlenstoffs (TOC) und des gelösten organischen Kohlenstoffs (DOC)

Analyse de l'eau - Lignes directrices pour le dosage du carbone organique total (TOC) et carbone organique dissous (COD)

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ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
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EUROPEAN STANDARD

EN 1484

NORME EUROPÉENNE

EUROPÄISCHE NORM

May 1997

ICS 13.060.30

Descriptors: environmental protection, water tests, potable water, ground water, sea water, surface water, sewage, chemical analysis, determination of content, organic carbon

English version

**Water analysis - Guidelines for the determination
of total organic carbon (TOC) and dissolved
organic carbon (DOC)**

Analyse de l'eau - Lignes directrices pour le dosage du carbone organique total (TOC) et carbone organique dissous (COD)

Wasseranalytik - Anleitungen zur Bestimmung des gesamten organischen Kohlenstoffs (TOC) und des gelösten organischen Kohlenstoffs (DOC)

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom:

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

This European Standard contains three informative annexes.

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 November 1997, and conflicting national standards shall be withdrawn at the latest by November 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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Introduction

It is absolutely essential that tests conducted according to this standard are carried out by suitably qualified staff.

Total organic carbon (TOC) is a measure of the carbon content of dissolved and undissolved organic matter present in the water. It does not give information on the nature of the organic substance.

1 Scope

This European Standard gives guidance for the determination of organic carbon in drinking water, ground water, surface water, sea water and waste water. It deals with definitions, interferences, reagents, and the sample pretreatment of water samples with a content of organic carbon ranging from 0,3 mg/l to 1000 mg/l while the lower value is only applicable in special cases, for example drinking water, measured with instruments capable of measuring these low levels. Higher concentrations may be determined after appropriate dilution. This European Standard does not deal with the instrument-dependent aspects.

In addition to organic carbon the water sample may contain carbon dioxide or ions of carbonic acid. Prior to the TOC determination, it is essential that this inorganic carbon is removed by purging the acidified sample with a gas which is free from CO₂ and organic compounds. Alternatively, both total carbon (TC) and total inorganic carbon (TIC) may be determined and the organic carbon content (TOC) may be calculated by subtracting the total inorganic carbon from the TC. This method is particularly suitable for samples in which the total inorganic carbon is less than the TOC.

Purgeable organic substances, such as benzene, toluene, cyclohexane, and chloroform may partly escape upon stripping. In presence of these substances the TOC concentration is determined separately, or the differential method (TC - TIC = TOC) may be applied. By using the differential method, the value of the TOC *should* be higher than the TIC, or at least of similar size.

Cyanide, cyanate, and particles of elemental carbon (soot) when present in the sample, will be determined together with the organic carbon.

NOTE: In presence of humic material low values may occur when UV-radiation is used.

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2 Normative References

This European Standard incorporates by dated or undated reference provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated reference, subsequent amendment to or revisions of any of these publications apply to this European standard only when incorporated in it by amendment or revision. For undated reference the latest edition of the publication referred to applies.

EN ISO 5667-3 : 1995

Water Quality – Sampling – Part 3: Guidance on the preservation and handling of samples
(ISO 5667-3 : 1994)

3 Definitions

For the purposes of this European Standard the following definitions apply:

3.1 Total carbon (TC)

The sum of organically bound and inorganically bound carbon present in water, including elemental carbon.

3.2 Total inorganic carbon (TIC)

The sum of carbon present in water, consisting of elemental carbon, total carbon dioxide, carbon monoxide, cyanide, cyanate, and thiocyanate. TOC instruments mostly register as TIC only the CO₂ originating from hydrogen carbonates and carbonates.

3.3 Total organic carbon (TOC)

The sum of organically bound carbon present in water, bonded to dissolved or suspended matter. Cyanate, elemental carbon and thiocyanate will also be measured.

3.4 Dissolved organic carbon (DOC)

The sum of organically bound carbon present in water originating from compounds which will pass a membrane filter of pore size of 0,45 μm . Cyanate and thiocyanate are also measured.

3.5 Volatile organic carbon (VOC, POC)

Under the conditions of this method purgeable organic carbon (POC).

3.6 Non volatile organic carbon (NVOC, NPOC)

Under the conditions of this method non-purgeable organic carbon (NPOC).

4 Principle

Oxidation of organic carbon (org. C) in water to carbon dioxide by combustion, by the addition of an appropriate oxidant, by UV radiation or any other high-energy radiation.

The application of the ultraviolet method with only oxygen as an oxidant is restricted to low polluted waters, containing low concentrations of TOC.

Inorganic carbon is removed by acidification and purging or is determined separately.

The carbon dioxide formed by oxidation is determined either directly or after reduction, for example, to methane (CH_4).

The final determination of CO_2 is carried out by a number of different procedures, for example: Infrared spectrometry, titration (preferably in non-aqueous solution), thermal conductivity, conductometry, coulometry, CO_2 -sensitive sensors and flame ionization detection - used after reduction of the CO_2 , among others, to methane.

5 Reagents

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5.1 General remarks

Use only reagents of recognized analytical grade.

In this European Standard only those chemicals and gases are listed which are used with the majority of TOC methods. Reagents should be used according to the manufacturer's instructions, and should be pretreated, if necessary.

The TOC of the water being used for dilution and for preparation of the calibration standards should be negligibly low, compared with the lowest TOC concentration to be determined.

The method for water treatment depends on the concentration range under investigation as shown in table 1.

NOTE: For measurements of a TOC concentration < 0,5 mg/l, it is preferable to prepare water for blanks and the calibration solutions immediately prior to analysis (see table 1).

Table 1: Dilution Water Specifications

TOC of the sample (mg/l C)	Maximum acceptable TOC of the dilution water (mg/l C)	Example of water treatment methods
< 10	0,1* 0,3	UV treatment condensation
10 to 100	0,5	double distillation with $\text{KMnO}_4/\text{K}_2\text{Cr}_2\text{O}_7$
> 100	1	Distillation
* only for ultrapure water		

5.2 Potassium hydrogen phthalate, stock solution, $\rho(\text{org. C}) = 1\,000\text{ mg/l}$

Dissolve in a 1 000 ml one-mark volumetric flask 2,125 g of potassium hydrogen phthalate ($\text{C}_8\text{H}_5\text{KO}_4$) (dried for 1 h at a temperature between 105°C and 120°C) in 700 ml of water, and make up to volume with water.

The solution is stable for about 2 months if stored in a tightly stoppered bottle in a refrigerator.

5.3 Potassium hydrogen phthalate, standard solution, $\rho(\text{org. C}) = 100\text{ mg/l}$

Pipette 100 ml of the potassium hydrogen phthalate stock solution (5.2) into a 1 000 ml one-mark volumetric flask, and make up to volume with water.

The solution is stable for about 1 week if stored in a tightly stoppered bottle in a refrigerator.

5.4 Standard solution for the determination of inorganic carbon, $\rho(\text{inorg. C}) = 1\,000\text{ mg/l}$

Dissolve in a 1 000 ml one-mark volumetric flask 4,415 g of sodium carbonate (Na_2CO_3) ,dried for 1 h at $(285 \pm 5)^\circ\text{C}$ in approximately 500 ml of water.

Add 3,500 g of sodium hydrogen carbonate (NaHCO_3) (dried for 2 h over silica gel), and make up to volume with water.

This solution is stable at room temperature for about 2 weeks.

5.5 Substances, not readily oxidizable

A standard solution shall be used to check the operational performance of the system.

NOTE: In the interlaboratory trial, copper phthalocyanine has been used for this purpose. A suitable test solution of copper phthalocyanine, $\rho(\text{org. C}) = 100\text{ mg/l}$, may be prepared as follows:

In a 1 000 ml volumetric flask 0,256 g of copper phthalocyanine-tetrasulfonic acid (tetrasodium salt) ($\text{C}_{32}\text{H}_{12}\text{CuN}_8\text{O}_{12}\text{S}_4\text{Na}_4$) are dissolved in 700 ml of water, and made up to volume with water.

The solution is stable for about 2 weeks.

WARNING: This reagent is toxic.

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5.6 Further reagents

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5.6.1 The reagents 5.2, 5.4 and 5.5 may be replaced by other reagents provided these are stable titrimetric substances.

5.6.2 Non volatile acids for expelling the carbon dioxide, such as phosphoric acid, $c(\text{H}_3\text{PO}_4) = 0,5\text{ mol/l}$, if necessary, more concentrated.

5.7 Gases

Air, nitrogen, oxygen, free from carbon dioxide and organic impurities. Use other gases in accordance with the instrument manufacturer's specifications.

6 Apparatus

Usual laboratory apparatus and

6.1 Apparatus for the determination of organic carbon

6.2 Homogenization device, for example a magnetic stirrer with adequate performance for the homogenization of dispersed matter, a suitable ultrasonic apparatus or a high speed stirrer.

7 Sampling and samples

7.1 Sampling

See also EN ISO 5667-3.

When sampling ensure that the samples being collected are representative (particularly in the presence of undissolved substances), and take care not to contaminate the samples with organic substances.

Collect water samples in glass or polyethylene bottles, completely filled with the sample, and, if biological activity is suspected, acidify to pH 2 (for example with phosphoric acid (5.6.2)). In some cases the result may be reduced by the loss of volatile substances during to the liberation of carbon dioxide, when the sample is acidified. If volatile organic compounds are suspected, carry out the measurement without acidification and within 8 h of sampling. Otherwise, store the sample in a refrigerator at a temperature in the range of 2 °C to 5 °C, and analyze within 7 days. If this is not possible, the sample can be kept at -15 °C to -20 °C for up to several weeks.

7.2 Preparation of the water sample

If, because of inhomogeneity, the collection of a representative sample is not possible, even after thorough shaking, homogenize the sample, using for example, the apparatus as described in 6.2.

The homogeneity may be checked for example by analyzing a sample from the upper and the lower layer of the bottle.

If only dissolved organic substances (DOC) are to be determined, filter the sample through a membrane filter, pore size 0,45 µm, which has previously been washed with hot water to completely remove adhering organic substances. Nevertheless the carbon content of the filtrate has to be determined and taken into account.

8 Procedure

8.1 Calibration

Comparison methods (for example IR detection) require calibration; in the case of absolute methods, for example acidimetry or coulometry, the calibration serves the purpose of checking the analytical system.

Calibrate the instrument according to the manufacturer's instructions.

Establish a calibration curve by analyzing potassium hydrogen phthalate standard solutions of adequate concentrations. For example for mass concentrations ranging from 10 mg/l to 100 mg/l proceed as follows: Prepare a series of at least 5 calibration solutions from the potassium hydrogen phthalate stock solution (5.2).

To prepare the calibration solutions pipette into each of a series of 100 ml volumetric flasks for example 0 ml (blank), 1 ml, 2 ml, 3 ml, 5 ml, and 10 ml of the potassium hydrogen phthalate stock solution (5.2), and make up to volume with water.

Analyze each solution and the blank solution (from the flask without added potassium hydrogen phthalate) in accordance with the manufacturer's instructions.

Establish a calibration curve by plotting the mass concentrations of TOC, in milligrams per litre of carbon, against the instrument-specific response units (I).

The reciprocal value of the slope of the resulting calibration line is the calibration factor f , in milligrams per litre of carbon.

In the case of the determination of TIC it is necessary to establish a calibration curve by analyzing calibration solutions made from solution (5.4).

In the case of the determination of TOC from the difference TC-TIC, it is necessary to establish a calibration curve by analyzing calibration solutions prepared from a known mixture of the standard solutions 5.3 and 5.4.

8.2 Control Procedures

Analyze the test solutions (either 5.2; or 5.3; or 5.4; or 5.5; or 5.6.1) with each series of samples in order to verify the accuracy of the results obtained by the method.

If the deviations found are higher than intra-laboratory quality criteria allow, then the following causes of error should be investigated:

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- Malfunction of the instrument (for example in the oxidation or detection system, leakages, faults in the temperature or the gas control);
- Changes in concentration in the test solution;
- Contamination of the measuring assembly.

Regularly check the entire measuring system according to the instructions of the manufacturer; also check the entire systems regularly for the absence of leaks.

These control experiments are carried out in addition to the instrumental control as specified in the operating instructions provided by the instrument manufacturer.

8.3 Determination

Determine the TOC concentrations of the samples in accordance with the instrument manufacturer's instructions.

In the case of direct determination of the TOC, remove the total inorganic carbon (ensure that the pH is below 2) prior to analysis. Carefully minimize the loss of volatile organic substances.

The TOC concentration should be within the working range of the calibration. This can be achieved by diluting the sample.

Prior to each batch of TOC determinations (for example 10 determinations) carry out appropriate control experiments at the intervals recommended by the manufacturer or specified by the laboratory.

After acidification, blow a stream of pure inert gas free from CO₂ and organic impurities through the system (for approximately 5 min) in order to remove CO₂.

9 Evaluation of results

9.1 Calculation

Depending on the type of TOC instrument used, different kinds of readings may be obtained from which the TOC or DOC concentration of the analyzed sample is calculated. In the case of discontinuous measurements, these values may be in terms of, for example, peak heights, peak areas or volume of the titrant required. Normally, peak areas are reported. Use peak heights only if they are proportional to the concentration.

In the case of quasi-continuous TOC or DOC measurements, the CO₂ concentration generated by the combustion of the organic matter is recorded, for example as a line on a strip chart recorder. The distance of this line from the zero line is proportional to the TOC concentration.

Calculate the mass concentration using the calibration curve (8.1).

The mass concentration of TOC or DOC, calculated in mg/l, may also be obtained from the following equation:

$$\frac{I \cdot f \cdot V}{V_p}$$

Where:

- I is the instrument specific response;
- f is the calibration factor evaluated as in 8.1, in milligrams per litre of carbon;
- V is the volume of the diluted water sample, in millilitres;
- V_p is the sample volume being diluted to V, in millilitres.