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Water quality — Guidelines for the determination of total organic carbon (TOC)

Qualité de l'eau — Guide pour le dosage du carbone organique total (COT)

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8245 was prepared by Technical Committee ISO/TC 147, *Water quality*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Water quality — Guidelines for the determination of total organic carbon (TOC)

0 Introduction

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 about the nature of the organic substance. Inorganic carbon must be eliminated before analysis or determined together with the TOC and subsequently subtracted. The method is instrumental and the selection of the analyser should take into consideration the carbon content of the samples to be analysed. The carbon content of organic substances may vary widely.

1 Scope and field of application

This International Standard gives guidelines for the measurement of TOC concentrations in all kinds of water. It covers definitions, interferences, reagents and pretreatment of water samples containing 0,1 to 1 000 mg/l organic carbon. Higher concentrations may be determined after appropriate dilution. Under certain circumstances it may be necessary to pretreat the sample, for example by the separation of larger particles contained in the water in order to avoid clogging of the apparatus.

This International Standard does not deal with the instrumental side of the determination.

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

Volatile organic substances such as benzene, toluene, cyclohexane, and chloroform may escape during stripping of CO₂. The TOC of these substances must therefore be determined separately or the difference method may be applied.

Particles of elemental carbon (soot), carbide, cyanides, cyanates and isocyanates when present are determined together with the organic carbon.

NOTE — Bibliographic reference 11.2 gives a comparison of the oxidation methods. TOC levels of different types of water are indicated.

2 Reference

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

3 Definitions

For the purpose of this International Standard the following definitions apply.

3.1 total carbon (TC): The quantity of carbon present in water in the form of organic, inorganic and elemental carbon.

3.2 total inorganic carbon (TIC): The quantity of carbon present in water in the form of elemental carbon, total carbon dioxide, carbon monoxide, carbides, cyanates, cyanides and thiocyanates.

3.3 total organic carbon (TOC): The quantity of carbon present in water in that organic matter which is dissolved or suspended in the water.

3.4 dissolved organic carbon (DOC): The quantity of carbon present in water in that organic matter which passes a filtration step through a membrane filter of pore size 0,45 µm.

4 Principle

Oxidation by combustion, addition of suitable oxidant, or ultra-violet irradiation, of organic carbon in water to carbon dioxide.

The application of the ultra-violet method with only oxygen as an oxidant is restricted to unpolluted waters free from particulate matter. Inorganic carbon is eliminated through acidification and purging or is determined separately.

The carbon dioxide formed by oxidation may be determined either directly or after reduction to methane (CH₄).

Among others, the following principles for the ultimate determination of CO₂ are applied: infra-red spectrometry, titration (preferably in non-aqueous solution), thermal conductivity (TCD), conductometry, coulometry, CO₂-sensitive electrodes and, after reduction of the CO₂ to CH₄, flame ionization (FID).

5 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity. In this International Standard, only those chemicals are mentioned that are used in most TOC methods. Further chemicals may be used, and, if necessary, pretreated, according to the manufacturer's instructions.

The TOC of the water used for dilutions and for preparation of the calibration standards shall be negligible in comparison to the TOC of the sample.

The method for water pretreatment depends on the concentration range to be measured. The pretreatment methods to be used in accordance with the TOC of the sample are given in table 1.

NOTE — Water with a TOC content <0,5 mg/l cannot be stored.

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

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 at a temperature not exceeding 120 °C for 2 h), in about 700 ml of water and dilute to the mark with water.

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

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

Pipette 100 ml of the potassium hydrogen phthalate stock solution (5.1) into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

This solution is stable for about one week, if stored in a tightly stoppered bottle in a refrigerator.

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

Dissolve in a 1 000 ml one-mark volumetric flask 4,41 g of sodium carbonate (Na_2CO_3) (dried at 285 °C for 1 h) in about 500 ml of water. Add 3,50 g of sodium hydrogen carbonate (NaHCO_3) (dried over silica gel) and dilute to the mark with water.

This stock solution is stable at room temperature.

5.4 Further reagents

5.4.1 The reagents 5.1 and 5.2 may be replaced by other solutions of stable compounds as long as they meet the requirements of titrimetric substances.

5.4.2 **Non-volatile acid** for expulsion of carbon dioxide, for example phosphoric acid, $c(\text{H}_3\text{PO}_4) = 0,5\ \text{mol/l}$.

5.5 Gases

Air, nitrogen, oxygen, free from carbon dioxide and organic impurities.

6 Apparatus

Usual laboratory apparatus and

6.1 Apparatus for TOC determination.

6.2 **Homogenization device**, for example ultrasonic instrument or magnetic stirrer with sufficient efficiency for homogenization of dispersed matter.

7 Sampling and samples

7.1 Sampling

See also ISO 5667-3.

When sampling, ensure that representative samples are collected (this is especially essential when undissolved substances are present) and be careful not to contaminate the samples with organic substances. Water samples shall be withdrawn in clean glass bottles which shall be filled completely. If storage is necessary, store at 4 °C in a refrigerator. If bacteriological activity is to be expected, the sample shall be acidified to pH 2 [e.g. with phosphoric acid (5.4.2)] in case of storage.

Samples shall be analysed within one week.

7.2 Preparation of the test sample

If, in case of inhomogeneity, the withdrawal of a representative sample is not possible even after thorough shaking, the samples shall be homogenized, for example by ultrasonic treatment with sufficient efficiency. The homogeneity shall be checked.

Table 1 — Water preparation methods

TOC of sample (mg/l carbon)	Maximum acceptable TOC of dilution water (mg/l carbon)	Example of water treatment method
< 10	0,1	— UV treatment — steam method — condensation
10 to 100	0,5	— double distillation with $\text{KMnO}_4/\text{K}_2\text{Cr}_2\text{O}_7$
> 100	1	— distilled water

If only the dissolved organic substances (DOC = dissolved organic carbon) are to be determined, carry out filtration using a membrane filter with pores of 0,45 µm, previously washed with hot water until no further organic substances are released.

8 Procedure

8.1 Calibration

A calibration curve is essential whenever comparative methods are used; in the case of absolute methods, for example acidimetry or coulometry, it is useful for checking the system.

Obtain the calibration curve by analysing the potassium hydrogen phthalate stock or standard solution of appropriate concentration.

For a TOC mass concentration range of 10 to 100 mg/l, proceed as follows.

Prepare from the potassium hydrogen phthalate stock solution (5.1) a series of at least five calibration solutions. Pipette into 100 ml volumetric flasks, for example 1; 2,5; 10 and 25 ml of potassium hydrogen phthalate solution (5.1). Make up to volume with water. Analyse each solution and the blank according to the instructions given by the manufacturer. Prepare a calibration curve by plotting the mass concentrations in milligrams per litre against the instrument response. The slope of the resulting line is the calibration factor f in milligrams per litre.

8.2 Control experiments

Check that the applied method delivers correct values by running standards with each series of samples. Depending on the concentration of the test solution, the following deviations from the true value can be tolerated:

1 to 10 mg/l $\pm 10\%$

> 10 mg/l $\pm 5\%$

If higher deviations occur, check the following sources of error:

- interferences in the instrumental device (for example in the oxidation or detection system, leakages, errors in temperature and gas regulation);
- changes in concentration of the test solution;
- contamination of the system.

In order to confirm the efficiency of the oxidation system it is advisable to choose test solutions (5.4) as similar to the sample as possible regarding oxidizability.

The whole measuring range shall be checked once a week.

These control experiments are in addition to the instrumental control tests as described in the operating instructions of the manufacturer.

8.3 Determination

Determine the TOC value as instructed by the manufacturer of the instrument. In the case of direct determination of TOC, the carbon dioxide present shall be removed prior to analysis. Take care that the loss of volatile organic substances is minimized.

The TOC of the sample should be in the working range of the instrument. This can be achieved by diluting a volume (V_p) of the sample to 100 ml (V). Prior to TOC determination, care shall be taken that the function control experiments are carried out at least at the intervals recommended by the manufacturer.

The total system shall be checked regularly for leaks.

9 Expression of results

9.1 Calculation

Depending on the TOC instrument used, different types of readings are obtained from which the TOC concentrations are to be evaluated. In the case of discontinuous measurements, these values may represent, for example, peak heights, peak areas, or the volume of the base needed for titration. Normally use peak areas. Peak heights shall be used only in cases where peak heights are proportional to concentrations.

In the case of continuous TOC measurement, the CO₂ concentrations are recorded, for example as a line on a strip chart. The distance of this line from the zero line is proportional to the TOC concentration. Calculate the TOC mass concentration $\rho(\text{TOC})$ using the calibration graph.

The mass concentration in milligrams per litre can also be calculated from the following equation:

$$\rho(\text{TOC}) = \frac{I \times f \times V}{V_p}$$

where

$\rho(\text{TOC})$ is the TOC mass concentration, in milligrams per litre;

I is the instrument-dependent response;

f is the calibration factor evaluated as in 8.1, in milligrams per litre;

V is the diluted sample volume (100 ml);

V_p is the volume of the sample which is diluted to V , in millilitres.

9.2 Precision

Results are expressed in milligrams of carbon per litre. The expression of results is dependent on the random error (precision) of the measurement. Report to two or three significant figures. Report the standard deviation with the degrees of freedom or the confidence interval as achieved within the laboratory that carried out the measurement.

Example:

$$\rho(\text{TOC}) = 0,76 \text{ mg/l or}$$

$$\rho(\text{TOC}) = 530 \text{ mg/l}$$

$$\rho(\text{TOC}) = 6,32 \times 10^3 \text{ mg/l}$$

State whether the sample has been filtered or centrifuged prior to analysis or if the analysis has been made on a sample that had been allowed to settle (method of sample pretreatment, time for settlement, filter used, etc.).

Information regarding repeatability and reproducibility, obtained from the results of an inter-laboratory test, is given in the annex.

10 Test report

The test report shall include the following information:

- a) a reference to this International Standard;

- b) all details required for the complete identification of the sample;

- c) details concerning the storage of the laboratory samples before analysis;

- d) the TOC of the sample, expressed in milligrams of carbon per litre;

- e) details of any deviation from the procedure specified in this International Standard or any other circumstances that may have influenced the result.

11 Bibliography

11.1 DÜRR, W. and MERZ, W., Evaluation of the TOC — ISO inter-laboratory trial and discussion of results. *Vom Wasser* **55**, 287-294 (1980).

11.2 *Methods for the examination of waters and associated materials. The instrumental determination of total organic carbon, total oxygen demand and related determinants.* Her Majesty's Stationery Office, London, 1979.

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Annex

Results of an inter-laboratory test on the TOC determination [11.1]

(This annex does not form an integral part of the Standard.)

Table 2 – Summary of the TOC inter-laboratory test

Sample	True value	Arithmetic mean value	Deviation from true value rel. %	Standard deviations				Number of participants		Number of eliminations	
				repeatability		reproducibility		total	valid	<i>a priori</i>	Cochran DI
				abs.	rel. (%)	abs.	rel. (%)				
4	1,63	1,260	-22,73	0,165	13,06	0,577	45,80	77	52	4	20
1	1,71	1,718	0,47	0,190	11,04	0,434	25,24	77	59	4	14
7/1	1,71	1,678	- 1,90	0,141	8,40	0,289	17,22	20	12	2	4
5	16,3	15,233	- 6,55	0,624	4,10	2,935	19,27	79	64	2	13
2	23,1	22,948	- 0,66	0,946	4,12	2,141	9,33	80	69	2	8
7/2	23,1	22,286	- 3,53	0,649	2,91	1,560	7,00	38	36	0	1
6	493	474,329	- 3,79	21,721	4,58	86,154	18,16	79	68	3	7
3	468	447,027	- 4,48	7,957	1,78	27,425	6,14	80	64	2	13
7/3	468	444,239	- 5,08	6,049	1,36	31,175	7,02	21	15	2	3

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Samples Nos. 1-3 : Potassium hydrogen phthalate

Sample No. 4 : Tetrapropylbenzene sulfonate

Sample No. 5 : Copper phthalocyanine
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Sample No. 6 : Urea/potassium hydrogen carbonate

Sample No. 7 : Prepared by adding aliquots of samples 1, 2 and 3 to a typical water sample most often dealt with by the laboratory

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