BS EN 16718:2015

BSI Standards Publication

Wood and wood based products — Dosage of the total organic carbon (TOC) in wood and wood based products

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National foreword

This British Standard is the UK implementation of EN 16718:2015.

BSI, as a member of CEN, is obliged to publish EN 16718 as a British Standard. However, attention is drawn to the fact that during the development of this European Standard, the UK committee voted against its approval as a European Standard.

Accurate and internationally accepted methods of calculating the carbon content of wood and wood products, including those that have been subject to further treatment or manufacturing processes, are already available. In the opinion of the UK committee, this renders the test method described in the standard unnecessary. The usefulness of this standard is limited to those products which, owing to the method of manufacture, have had carbon added or removed in an uncontrolled manner so that carbon content cannot easily be calculated from the knowledge of wood content and of manufacturing parameters.

The UK committee has observed that the German source word 'Bestimmung' in the title of the standard has been translated as 'Dosage' rather than 'Determination'. However, the word 'Determination' is used in the Scope of the standard (Clause 1). Users should be aware of this discrepancy, which occurs throughout the standard.

The UK participation in its preparation was entrusted to Technical Committee B/515, Wood preservation.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

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Wood and wood based products - Dosage of the total organic carbon (TOC) in wood and wood based products

Produits de préservation du bois et matériaux à base de bois - Dosage du carbone organique total (COT) dans les bois et matériaux à base de bois

Holz und Holzprodukte - Bestimmung des gesamten organischen Kohlenstoffs (TOC) in Holz und Holzprodukten

This European Standard was approved by CEN on 12 September 2015.

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

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European foreword

This document (EN 16718:2015) has been prepared by Technical Committee CEN/TC 38 “Durability of wood and wood-based products”, the secretariat of which is held by AFNOR.

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 May 2016, and conflicting national standards shall be withdrawn at the latest by May 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Bio-based products from forestry and agriculture have a long history of application. The last decades have seen the emergence of new bio-based products in the market. Acknowledging the need for common standards for bio-based products, the European Commission issued mandate M/492, resulting in a series of standards developed by CEN/TC 411.

For business to business transactions, claims which are relevant to describe characteristics of bio-based products in a business to business environment will be given in the near future. Data are by consequence required to generate and transfer information in the industrial chain and/or as an input for product specific standards and certification schemes.

The work to be done by the CEN/TC 411/WG 3 concerns the determination of the bio-based carbon in order to determine the level of bio-based content of a product or materials. A document (CEN/TR 16721) has been prepared by Technical Committee CEN/TC 411 "Bio-based products", and describes a list of methods and an "overview of methods to determine the bio-based content and related methods" for Bio-based products.

As part of the task force of CEN/TC 175, devoted to carbon foot printing and LCA, a European Standard was published on the simplified calculation of the amount of biomass carbon stored in wood (using 50 % of the anhydrous wood mass): EN 16449.

This standard EN 16718 describes the methods based on analytical measurements. These methods can be considered as complementary to the radiocarbon based method and methods based on evaluation by calculation (mass balance approaches). One of these analytical methods is a method based on measurement of stable isotopic ratio present in biomass in order to determine the biomass content of the product.

The development of this method described in this report is ongoing with close collaboration between FCBA and the "Institute des Sciences Analytiques" CNRS in order to determine the bio-based content of wood raw materials, glues and panels made with these raw materials for end use manufactured products with this new method. The objective is to propose correlated analysis (with the TOC method proposed by FCBA) to determine the carbon content to purpose a quick and low cost method easy to handle.

References:

— http://www.biobasedeconomy.eu/standardisation/cen-tc411/

— http://www2.afnor.org/espace_normalisation/structure.aspx?commid=86489

The tests that have resulted in the specification of this document were performed in the context of work conducted by the FCBA [timber certification body] Technological Institute aimed at determining a method for supplying data on organic carbon contents that could be used to calculate carbon balances.

The storage of biomass carbon in wood-based products is the preservation of the carbon absorbed by the tree from atmospheric CO_2 through photosynthesis.

The carbon thus captured in the material is of benefit to the climate throughout the lifespan of the product, which can be several dozen years for a construction product, for example. The French Standard NF P01-010 (2004), which lays out the format of environmental and health statements (FDES) for construction products, provides the option of indicating the following supplementary information, in addition to the "Climate change" indicator, which is calculated from the flows of greenhouse gases associated with the product life cycle: "for some construction products (e.g. plant-based products), CO2 storage during the "service life" stage can be given if measurements are taken based on standardized test methods."

Furthermore, the Guide to Best Practices on environmental labelling of mass-market consumer products (BP X30-323) includes in Annex G: “Carbon accounting integrating time lag” which also requires knowledge of the biomass carbon contents of the products.

The purpose of this document is therefore to propose a laboratory measurement method of the amount of biomass carbon that will provide values of carbon or CO_2 equivalent stored in wood-based products, with the aim to integrate this information in the environmental statements of these products according to the texts referenced above.

While measurement is not systematically necessary for solid wood products, for example, given the common knowledge on the densities of the various wood species and on the proportion of carbon contained in wood, this experimental measurement may prove to be necessary for products made of wood-based composite materials.

The organic carbon contained in wood and wood-based materials is found in several different forms. Cumulative measurements, such as total organic carbon (TOC), need to be used. Isotopic ratio enables the differentiation between synthetic and natural products. IRMS (Isotope Ratio Mass Spectrometer) is a complementary method to the TOC method by an identification of the isotope ^{13}C: both techniques are necessary to give reliable data on a bio-based content on a wood based material such as panel, board, and woods containing chemicals in general. A study is currently in progress in France on wood based materials: the results will enable to improve this present document and to give data with multi-isotopic determinations (^{13}C, ^{15}N, ^{2}H, ^{18}O).

1 Scope

This European Standard describes a method for determining total organic carbon by calculating the difference between the results of measurements of total carbon (TC) and total inorganic carbon (TIC). The identification of the bio-based content given by the stable isotopes such as ^{13}C is described also.

This method is applicable to all wood species, wood-based materials (panels, plywood, wood-polymer, etc.) and woods containing chemicals in general.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 13183-1, *Moisture content of a piece of sawn timber — Part 1: Determination by oven dry method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
total carbon
TC
amount of carbon found in waste, in organic, inorganic and elemental-state forms

3.2
total inorganic carbon
TIC
amount of carbon released as carbon dioxide through acidification

3.3
total organic carbon
TOC
carbon that is transformed into carbon dioxide through combustion and not released through acidification as carbon dioxide

Note 1 to entry: The definitions given above are only applicable in this document and only partly overlap the scientific definitions of TC, TOC and TIC.

4 Principle

In this procedure, TOC is obtained by subtraction between the measurement results of TC and TIC.

The total carbon (TC) present in the undried sample is transformed into carbon dioxide through combustion in a flow of gas that contains oxygen and is free of carbon dioxide. To ensure combustion is total, catalysers and/or modifiers can be used. The amount of carbon dioxide released is measured using infrared spectrometry, gravimetry, coulometry, conductometry, thermal conductivity detection or flame ionization detection after reduction to methane, or any other appropriate technique.

The TIC is determined separately using another sub-sample, through acidification and purging of the released carbon dioxide, which is then measured using one of the techniques mentioned above.

The ^{13}C identification by IRMS is described in 7.7. This protocol is able also to work on other isotopes: ^{15}N, ^{2}H and ^{18}O, which could be useful for complex materials containing wood.

5 Reagents and products

All the reagents used shall be of analytical grade at least and suitable for their specific uses. Hygroscopic products shall be kept in a dessicator.

5.1 Glucose, $C_6H_{12}O_6$.

5.2 Anhydrous sodium carbonate, Na_2CO_3.

5.3 Non-oxidizing mineral acid used to release the carbon dioxide, e.g. phosphoric acid H_3PO_4, (m = 85 %).

5.4 Synthetic air, nitrogen, oxygen, argon, free of carbon dioxide and organic impurities, according to the instructions supplied by the machine manufacturer.

6 Apparatus

6.1 Homogenization device, such as mixers, stirrers.

6.2 Analytical balance accurate to at least 0,5 % of test portion weight.

6.3 Apparatus for dosing carbon in solid matter, along with its accessories.

6.4 Purging device for dosing TIC.

6.5 Mixer mill.

7 Procedure

7.1 Preparation of the sample

Before the sample preparation, the sampling program shall be properly designed in accordance with the context of the testing and objectives.

The samples to be analysed should be as homogeneous as possible and undried.

The samples of wood or wood-based materials can be directly ground (avoiding any heating) and reduced to powder, preferably with a particle size below 500 µm. The samples are ground in their entire thickness.

The samples that contain negligible concentrations (taking into account the accuracy of the method used) of volatile compounds other than water can be dried at 105 °C before they are homogenized.

7.2 Water content

The determination of moisture content shall be carried out using a different test portion. It can be calculated from the dry matter mass, determined according to EN 13183-1.

NOTE Any other determination method (e.g. with a desiccator's balance) can be used if it has been previously validated.

7.3 Dosage

7.3.1 General

This document does not give any recommendations for the apparatus and its mode of operation. The operating conditions should be selected and verified according to the instructions supplied by the manufacturer.

It is advisable to select a test portion with the greatest mass possible, making sure that the amount of carbon dioxide released is within the apparatus measurement range and the calibration range.

7.3.2 TC dosage

The sample, prepared according to 7.1, is weighed in an appropriate container, i.e. inert and not liable to interact during the carbon content analysis reaction or to contain carbon in any form whatsoever (scoop or crucible made of e.g. ceramic, silica glass, platinum or tin). The container can be previously conditioned by heating (in a muffle furnace or in the analyser itself) to minimize blank carbon values.

The sample is burned or broken down in a carrier gas current that contains oxygen.

The combustion temperature shall be sufficiently high to transform all the carbon into carbon dioxide. For samples containing carbonates that are difficult to break down, such as barium carbonate, carbon dioxide release can be improved by increasing the temperature or using modifiers (e.g. tin, copper).

The temperature range of commercially available devices is between 900 °C and 1 500 °C.

During combustion of the reactive samples, any detonation or production of smoke can be avoided by covering the sample with an inert material (e.g. siliceous sand).

The carbon dioxide released while gas is being discharged is measured using the detection method described in chapter 4, and expressed as carbon.

7.3.3 TIC dosage

The sample prepared according to 7.1 is weighed in the purging device.

The system is closed so as to be impermeable to gases and purged using the carrier gas until the carbon dioxide from the ambient air is eliminated. Then, acid is added and the carbon dioxide is carried out through purging or stirring and/or heating. The carbon dioxide released is transferred to the detector by means of the carrier gas.

The carbon dioxide released while gas is being discharged is measured using the detection method described in Clause 4, and expressed as carbon.

7.4 Calibration

If detection is carried out using a relative method, e.g. infrared detection, calibration is necessary.

Glucose is an example of a standard substance that is appropriate for TC dosage.

Sodium carbonate or calcium carbonate can be used for TIC calibration.

Other standard substances can be used, provided their suitability has been verified.

During calibration, the procedure below should be followed:

— set the preliminary measurement range;

— analyse a series of four calibration samples minimum at least twice, at three different times. The concentration of these master samples shall be regularly distributed over the entire measurement range;

— calculate the mean values for each concentration;

— perform a linear regression analysis using the mean values.

This function should be linear. If this is not the case, the measurement range needs to be reduced to the linear range.

This calibration should not be implemented for initial validation purposes or when major modifications of the apparatus are carried out.

7.5 Control measurements

Control measurements shall be taken to make sure the apparatus is functioning properly. They should be taken every working day. It is deemed sufficient to perform the dosage three times from a point located in the middle of the respective measurement ranges. For TC and TIC, the mean recovery rate shall be between 90 % and 110 % with a variation coefficient ≤ 5 %.

7.6 Evaluation

The TC and TIC masses contained in the samples prepared according to 7.1 are calculated using:

— The calibration function and the sample mass where relative detection methods are used;

— Specific constants and the sample mass where absolute detection methods are used.

TC and TIC contents are the means of at least two measurements each. The respective differences between the two values should be less than or equal to 10 % of the mean value. If this is not the case, at least one other additional dosage needs to be carried out; in such a case, the variation coefficient should be less than or equal to 10 %. If this is not the case, the coefficient shall be recorded along with the result obtained.

The TOC content is calculated using the difference between the mean TC and TIC values with the following formula:

$$\omega_{TOC} = \omega_{TC} - \omega_{TIC} \quad (1)$$

where

ω_{TOC} is the TOC content in carbon dioxide form in the original sample, in grams per kilogram (g/kg);

ω_{TC} is the mean value of the TC content in carbon dioxide form in the sample, in grams per kilogram (g/kg);

ω_{TIC} is the mean value of the TIC content in carbon dioxide form in the sample, in grams per kilogram (g/kg).

The TOC content yielded by Formula (1) is correlated to the dry matter using Formula (2). To do so, the water content, determined separately, is used:

$$\omega_{TOCdm} = \omega_{TOC} \frac{100}{100 - w} \quad (2)$$

where

ω_{TOCdm} is the TOC content in carbon dioxide form, correlated to the dry matter base, in grams per kilogram (g/kg);

ω_{TOC} is the TOC content in carbon dioxide form in the original sample, in grams per kilogram (g/kg);

w is the water content of the original sample, expressed as a mass fraction in percentage (%).

The TOC content is generally determined using the undried sample, but it is always recorded as carbon correlated to dry matter. With Formula (2), the results are obtained in g/kg. They can be converted to other units by using the appropriate factors.

7.7 ^{13}C measurement by IRMS (Isotope Ratio Mass Spectrometer)

7.7.1 Material and methods

Isotopic analyses are performed using an elemental analyser (carbon nitrogen analyser or oxygen hydrogen analyser) linked to an isotope ratio mass spectrometer (IRMS). At first the samples are mineralized into gas: carbon and nitrogen are transformed into CO_2 and N_2 by combustion and reduction of nitrogen oxides, and hydrogen and oxygen are converted into H_2 and CO by a pyrolysis reaction.

The determination of the isotopic ratios is carried out by using the different atomic masses of gas as follows:

— CO_2: atomic mass 44, 45 and 46;

— N_2: atomic mass 28, 29 and 30;

— H_2: atomic mass 2, 3;

— CO: atomic mass 28, 30.

As the isotopic variations are very small, a relative scale (value δ) is used to express the isotopic ratio.

The isotopic ratios of samples for carbon, nitrogen, hydrogen and oxygen are expressed as values $\delta\,^{13}C$, $\delta\,^{15}N$, $\delta\,^{2}H$ and $\delta\,^{18}O$ respectively.

The STD ratio is given by the isotopic ratio of an international reference defined for each isotope.

$\delta\,^{13}C$ is the delta value of 13C isotope for the sample, expressed in parts per thousand (‰):

$$\delta^{13}C = \left[\frac{{}^{13}C/{}^{12}C \text{ sample}}{{}^{13}C/{}^{12}C \text{ standard}} - 1 \right] \times 1\,000$$

Isotopic measurements have been done relative to an international standard:

— For ^{13}C Vienna Pee Dee Belemnite (VPDB) is the standard and the percentage is 1,123 7 $^{13}C/^{12}C$ %;

— For $^{15}N/^{14}N$ atmospheric air is the standard and the ratio is 0,367 6 %;

— For $^{2}H/^{1}H$ and $^{18}O/^{16}O$ the isotopic standard is Vienna Standard Mean Ocean Water (V-SMOW), supplied by IAEA (Vienna) a water which the isotopic ratio of $^{2}H/^{1}H$ is 0,015 5 %, and that isotopic ratio for $^{18}O/^{16}O$ is 0,205 2 %.

The isotopic analysis is a fast method (duration less than 10 min) and needs only few milligrams of material.

The analysis error is about ± 0,3 ‰ for ^{13}C isotope and ^{15}N isotope, ± 1 ‰ for ^{18}O isotope and ± 5 ‰ for ^{2}H isotope. Although ^{2}H isotope error is high, the variations between the different origins are also greater than others isotopes. Isotopic values range from −30 ‰ to +20 ‰ for ^{13}C isotope, ^{15}N isotope and ^{18}O and −300 ‰ to +100 ‰ for ^{2}H isotope.

7.7.2 ^{13}C isotope

Three different metabolic pathways for carbon fixation in photosynthesis of plants exist:

a) the Calvin cycle or C_3 cycle (C_3 Plants);

b) the Hatch and Slack cycle or C_4 cycle (C_4 plants);

c) the Crassulacean acid metabolism or CAM cycle (CAM using plants).

Plants are divided in these classes according their different pathways.

In the Calvin cycle (C_3 cycle) different plants from temperate regions such as rice, grape, plants found in honey and trees (hardwood, fir…). Their isotopic values are in the range −20 ‰ to −33 ‰.

In the Hatch and Slack cycle (C_4 cycle) the rapidly growing plants such as cane sugar, maize, and amaranth are comprised. Their isotopic values are in the range −9 ‰ to −14 ‰.

The CAM cycle is reacting between the both C_3 and C_4 cycles and their isotopic values are in the range −10 ‰ to −24 ‰. In this class one can find pineapple and vanillin. Vanillin, which is the most important flavour extracted in the world, has an isotopic value near −20 ‰ for bean origin that is far from the synthesis origin −28 ‰. This technique, which is over 30 years old, is currently used to assess the authentication of vanillin samples.

For the C_4 plants (e.g. maize, cane sugar), isotopic values are in the range −9 ‰ to −14 ‰ that are different from the fossil oil origin where values range from −20 ‰ to −35 ‰. This means that products made from natural origins such as maize or cane sugar could be authenticated using ^{13}C isotopic measurements.

7.7.3 ^{18}O isotope

$^{18}O/^{16}O$ isotopic ratio alone is not very efficient to clarify origin of natural or synthetic molecules. The determination of this isotope is used in addition of ^{13}C and ^{2}H (multi isotopic determinations).

7.7.4 ^{2}H isotope (Deuterium)

Water is the only source of hydrogen for the photosynthesis in natural compounds. Isotopic values coming from natural flavours show lower values than the ones of synthetic origin. In any case $\delta\ ^{2}H$ values coming from synthesis compounds are positive.

Deuterium isotope measurement is regularly used in order to check the origin of raw materials or flavours in order to differentiate between natural or synthetic. Differences between both origins are very high, more than 100 ‰.

7.7.5 ^{15}N Isotopes

^{15}N isotope could not be analysed directly in wood origin because of the lower level of nitrogen contained. However isotopic values obtained in the origin of glue or paint for example could be given indications about the origin of these different compounds.

7.7.6 Multi-isotopic determinations: ^{13}C, ^{18}O and ^{2}H in wood and tree

More recently multi-isotopic determinations have been performed on wood, cellulose and lignin. These investigations have been done with the aim to link the trees growing and the climate (temperature, rainfall...). In that way, samples of cores, rings extracted from different varieties of trees were selected in a dendrochronological study.

Results showed that isotopic values range from −25 ‰ to −21 ‰ for ^{13}C isotope, +22 to +29 for ^{18}O and −141 to −29 for ^{2}H isotope [10] [11].

8 Performance characteristics

The performance data of the method are given in Annex A. They were established during a study conducted in 2011.

9 Test report

The test report shall include the following information at least:

a) individual or legal entity that drafted it;

b) date;

c) person who requested the test;

d) all the information needed to completely identify the wood-based material or wood species, including its origin;

e) a reference to this document;

f) the measurement techniques used;

g) the results obtained, including the associated uncertainties if possible;

h) any additional information on the operations that are not prescribed in this standard, as well as the phenomena that may have influenced the result obtained.

Annex A
(informative)

Results of the validation method

A.1 General

The results of the validation method conducted in 2011 by the FCBA Technological Institute are presented below.

a) Apparatus:

1) mixer mill with a heavy metal-free lining, equipped with a 0,5 mm sieve;

2) analytical balance;

3) ceramic crucibles;

4) Shimadzu TOC 5050A TOC analyser + SSM TOC 5000A module.

b) Reagents:

1) glucose $C_6H_{12}O_6$ (TC = 40 %);

2) sodium carbonate: Na_2CO_3 (IC = 11,3 %);

3) phosphoric acid (25 %).

c) Statistical processing software:

1) E-noval version V3.0a PROD;

2) Excel version 2000 9.0 2812.

A.2 Response function

A.2.1 General

The response function establishes the relationship between the response (device signal) and the analyte concentration (amount) in the sample. The calibration curve is the most appropriate response function.

Table A.1 — Experiment design for the response function

Experiment design		
Number of series	Number of levels	Number of repetitions
3	4	2

A.2.2 Total carbon

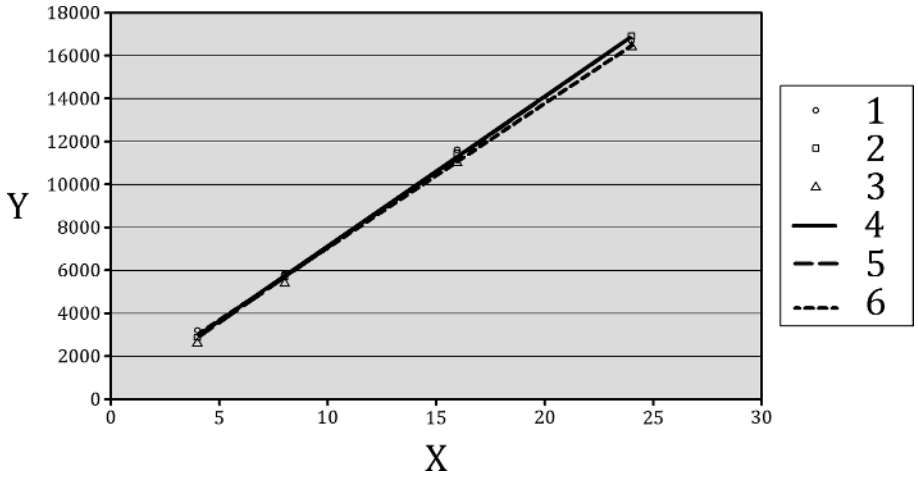
Table A.2 — Measurement for each level (TC)

	Measurement for each level (peak area)							
Series	**Level 1** (4 mg of C)		**Level 2** (8 mg of C)		**Level 3** (16 mg of C)		**Level 4** (24 mg of C)	
	Repetition 1	Repetition 2	Repetition 1	Repetition 2	Repetition 1	Repetition 2	Repetition 1	Repetition 2
1[a]	3,054	3,183	5,559	5,493	11,358	11,587	16,779	16,834
2[b]	2,850	2,727	5,686	5,586	11,260	11,427	16,688	16,856
3[c]	2,953	2,675	5,801	5,453	11,090	11,041	16,413	16,509

a Formula of the series 1 calibration curve: y = 693,19x + 219,42 (R^2 = 0,998 9).

b Formula of the series 2 calibration curve: y = 700,19x + 32,466 (R^2 = 0,999 7).

c Formula of the series 3 calibration curve: y = 681,38x + 133,89 (R^2 = 0,999 5).



Key

Axis: X: nominal carbon mass (mg)

Y: measured area

1 Series1

2 Series2

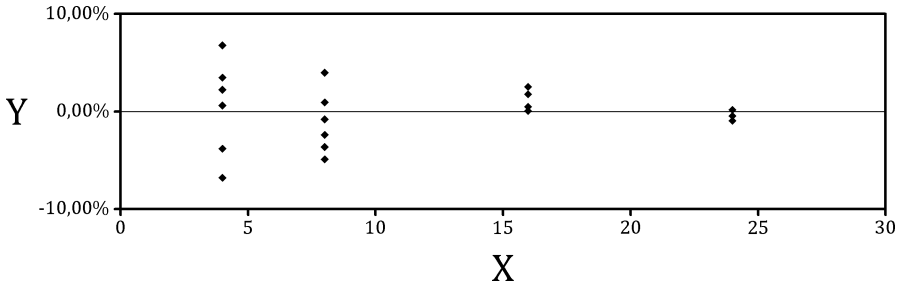
3 Series3

4 Linear (Series 1)

5 Linear (Series 2)

6 Linear (Series 3)

Figure A.1 — Response function (TC)



Key

Axis: X: theoretical quantities

Y: bias in percentage

Figure A.2 — Bias distribution in % according to the level

A.2.3 Inorganic carbon

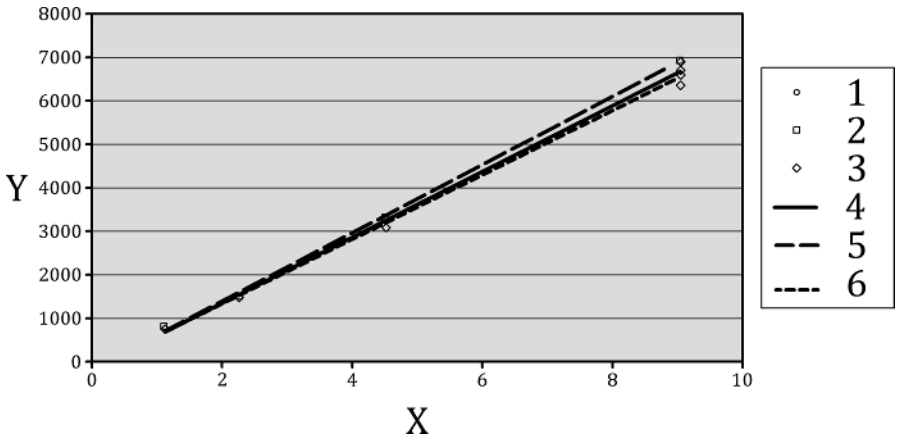
Table A.3 — Measurement for each level (IC)

	Measurement for each level (peak area)							
Series	**Level 1** (1,13 mg of IC)		**Level 2** (2,26 mg of IC)		**Level 3** (4,52 mg of IC)		**Level 4** (9,04 mg of IC)	
	Repetition 1	Repetition 2	Repetition 1	Repetition 2	Repetition 1	Repetition 2	Repetition 1	Repetition 2
1[a]	769	756	1,520	1,483	3,152	3,086	6,893	6,595
2[b]	811	733	1,499	1,450	3,297	3,314	6,886	6,947
3[c]	774	742	1,452	1,453	3,334	3,207	6,692	6,357

a Formula of the series 1 calibration curve: $y = 760{,}82x - 192{,}24$ ($R^2 = 0{,}997\ 5$)

b Formula of the series 2 calibration curve: $y = 785{,}48x - 211{,}33$ ($R^2 = 0{,}999$)

c Formula of the series 3 calibration curve: $y = 736{,}29x - 118{,}67$ ($R^2 = 0{,}997\ 7$)



Key

Axis: X: nominal IC value (mg)
Y: measured area

1 Series 1

2 Series 2

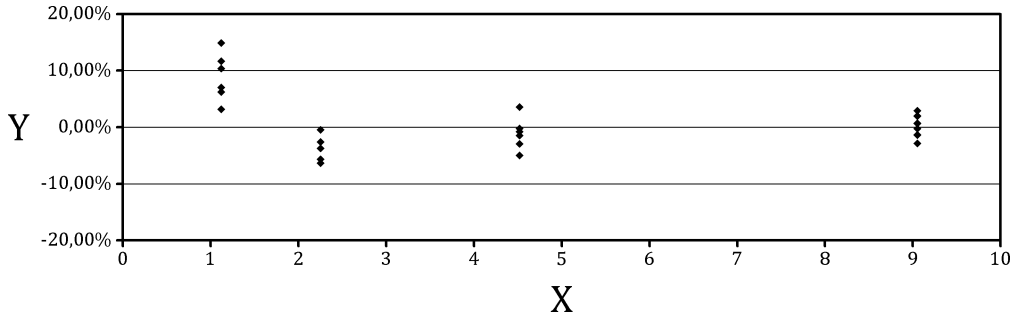
3 Series 3

4 Linear (Series 1)

5 Linear (Series 2)

6 Linear (Series 3)

Figure A.3 — Response function (IC)



Key

Axis: X: theoretical quantities
Y: bias in percentage

Figure A.4 — Bias distribution in % according to the level in relation to the maximum deviation accepted by the laboratory (IC)

A.3 Trueness

A.3.1 General

Bias is the closeness of agreement between the mean value obtained from a large series of test results and an accepted reference value. It provides information on the presence of a systematic error.

Table A.4 — Experiment design for trueness

Experiment design		
Number of series	Number of levels	Number of repetitions
3	4	6

A.3.2 Total carbon

Table A.5 — Results obtained for the trueness criterion (TC)

Level	Amount introduced (mg)	Mean of results (mg)	Absolute bias (mg)	Relative bias (%)	Recovery rate (%)	Confidence interval at 95 % of recovery (%)
1	4	3,916	- 0,083 89	- 2,097	97,90	[95,65 ; 100,2]
2	12	11,93	- 0,068 89	- 0,5741	99,43	[98,37 ; 100,5]
3	24	23,56	- 0,044 06	- 1,836	98,16	[97,28 ; 99,05]

A.3.3 Inorganic carbon

Table A.6 — Results obtained for the trueness criterion (IC)

Level	Amount introduced (mg)	Mean of results (mg)	Absolute bias (mg)	Relative bias (%)	Recovery rate (%)	Confidence interval at 95 % of recovery (%)
1	1,430	1,431	0,000 555 6	0,038 85	100	[98,47 ; 101,6]
2	2,860	2,769	- 0,091 11	- 3,186	96,81	[94,88; 98,74]
3	7,150	6,615	- 0,535 0	- 7,483	92,52	[91,10 ; 93,93]

A.4 Precision

A.4.1 General

Precision is the closeness of agreement between the results of independent tests obtained under specified conditions. It quantifies a random error and can be evaluated on 2 components: repeatability and intermediate precision.

A.4.2 Total carbon

Table A.7 — Results obtained for relative repeatability and intermediate precision (TC)

Relative repeatability and intermediate precision			
Level	Mean of quantities introduced (mg)	Repeatability (VC %)	Intermediate precision (VC %)
1	4	4,535	4,535
2	12	2,132	2,132
3	24	1,659	1,819

Table A.8 — Results obtained for absolute repeatability and intermediate precision (TC)

Absolute repeatability and intermediate precision					
Level	**Mean of quantities introduced (mg)**	**Repeatability** (SD–mg)	**Interseries** (SD-mg)	**Ratio of variance components (Inter/Intra)**	**Intermediate precision** (SD-mg)
1	4	0,181 4	0	0	0,181 4
2	12	0,255 8	0	0	0,255 8
3	24	0,398 2	0,178 9	0,201 8	0,436 5

Table A.9 — Upper confidence level at 95 % (TC)

Upper confidence level at 95 %			
Level	**Mean of quantities introduced** (mg)	**Upper confidence level at 95 % of repeatability** (SD-mg)	**Upper confidence level at 95 % of intermediate precision** (SD-mg)
1	4	0,254 0	0,2540
2	12	0,358 2	0,3582
3	24	0,572 3	1,131

Table A.10 — Recovery rate per series (TC)

Recovery rate per series				
Level	**Series**	**Mean of quantities introduced** (mg)	**Calculated amount** (mg)	**Recovery rate** (%)
1	1	4,000	3,920	98,00
1	2	4,000	3,945	98,63
1	3	4,000	3,883	97,08
1	Mean of all series	4,000	3,916	97,90
2	1	12,000	11,87	98,92
2	2	12,000	12,01	100
2	3	12,000	11,92	99,32
2	Mean of all series	12,000	11,93	99,43
3	1	24,000	23,72	98,84
3	2	24,000	23,68	98,65
3	3	24,000	23,28	97,01
3	Mean of all series	24,000	23,56	98,16

A.4.3 Inorganic carbon

Table A.11 — Relative repeatability and intermediate precision (IC)

Relative repeatability and intermediate precision			
Level	**Mean of quantities introduced** (mg)	**Repeatability** (VC %)	**Intermediate precision** (VC %)
1	1,430	3,037	3,196
2	2,860	3,882	3,882
3	7,150	2,473	2,981

Table A.12 — Absolute repeatability and intermediate precision (IC)

Absolute repeatability and intermediate precision					
Level	**Mean of quantities introduced** (mg)	**Repeatability** (SD–mg)	**Interseries** (SD-mg)	**Ratio of variance components (Inter/Intra)**	**Intermediate precision** (SD-mg)
1	1,430	0,043 42	0,014 26	0,107 8	0,045 70
2	2,860	0,111 0	0	0	0,111 0
3	7,150	0,176 9	0,119 0	0,452 8	0,213 2

Table A.13 — Upper confidence level at 95 % (IC)

Upper confidence level at 95 %			
Level	**Mean of quantities introduced** (mg)	**Upper confidence level at 95 % of repeatability** (SD-mg)	**Upper confidence level at 95 % of intermediate precision** (SD-mg)
1	1,430	0,062 41	0,108 7
2	2,860	0,155 5	0,155 5
3	7,150	0,254 2	0,636 3

Table A.14 — Recovery rate per series (IC)

Recovery rate per series				
Level	**Series**	**Mean of quantities introduced** (mg)	**Calculated amount** (mg)	**Recovery rate** (%)
1	1	1,430	1,415	98,95
1	2	1,430	1,420	99,30
1	3	1,430	1,457	101,9
1	Mean of all series	1,430	1,431	100
2	1	2,860	2,800	97,90
2	2	2,860	2,785	97,38
2	3	2,860	2,722	95,16
2	Mean of all series	2,860	2,769	96,81
3	1	7,150	6,740	94,27
3	2	7,150	6,640	92,87
3	3	7,150	6,465	90,42
3	Mean of all series	7,150	6,615	92,52

A.5 Measurement uncertainty

A.5.1 General

Measurement uncertainty is a parameter associated with a measurement result that characterizes the dispersion of values that could reasonably be attributed to the mesurand.

A.5.2 Total carbon

Table A.15 — Results obtained on uncertainties (TC)

Uncertainty					
Level	**Mean of quantities introduced** (mg)	**Bias uncertainty** (mg)	**Uncertainty** (mg)	**Extended uncertainty** (mg)	**Relative extended uncertainty** (%)
1	4	0,042 76	0,186 4	0,372 8	9,319
2	12	0,060 30	0,262 8	0,525 7	4,381
3	24	0,139 5	0,458 2	0,916 5	3,819

A.5.3 Inorganic carbon

Table A.16 — Results obtained on uncertainties (IC)

Uncertainty					
Level	**Mean of quantities introduced** (mg)	**Bias uncertainty** (mg)	**Uncertainty** (mg)	**Extended uncertainty** (mg)	**Relative extended uncertainty** (%)
1	1,430	0,01314	0,04755	0,09511	6,651
2	2,860	0,02617	0,1141	0,2281	7,977
3	7,150	0,08036	0,2278	0,4556	6,372

A.6 Accuracy

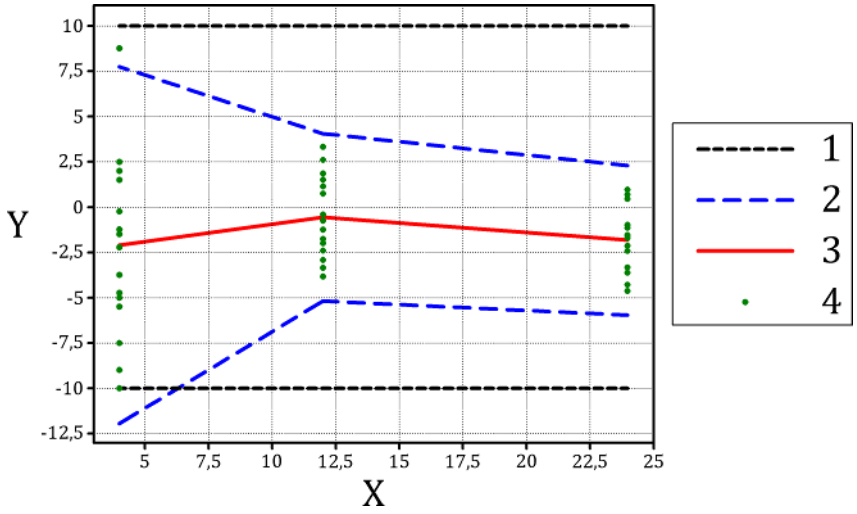
A.6.1 General

Accuracy expresses the closeness of deviation between the test result and the accepted reference value; it takes into account total error and random error. Accuracy is the expression of the sum of trueness and precision.

A.6.2 Total carbon

Table A.17 — Results obtained for the accuracy criterion (TC)

Method accuracy				
Level	**Mean of quantities introduced** (mg)	**Beta-expectation tolerance limit** (mg)	**Relative beta-expectation tolerance limit** (%)	**Risk** (%)
1	4,000	[3,522 ; 4,310]	[- 11,95 ; 7,751]	6,379
2	12,00	[11,38 ; 12,49]	[- 5,203 ; 4,055]	0,03362
3	24,00	[22,57 ; 24,55]	[- 5,972 ; 2,301]	0,04974



Key

Axis: X: amount (mg)

Y: relative error (%)

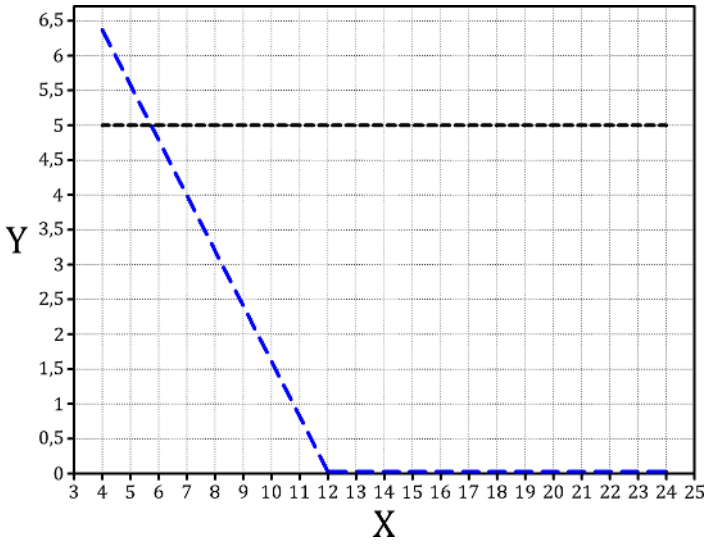
1 acceptance limits (set to 10 %)

2 tolerance interval limits

3 bias

4 relative concentration error

Figure A.5 — Accuracy profile (TC)



Key

Axis: X: amount introduced (mg)

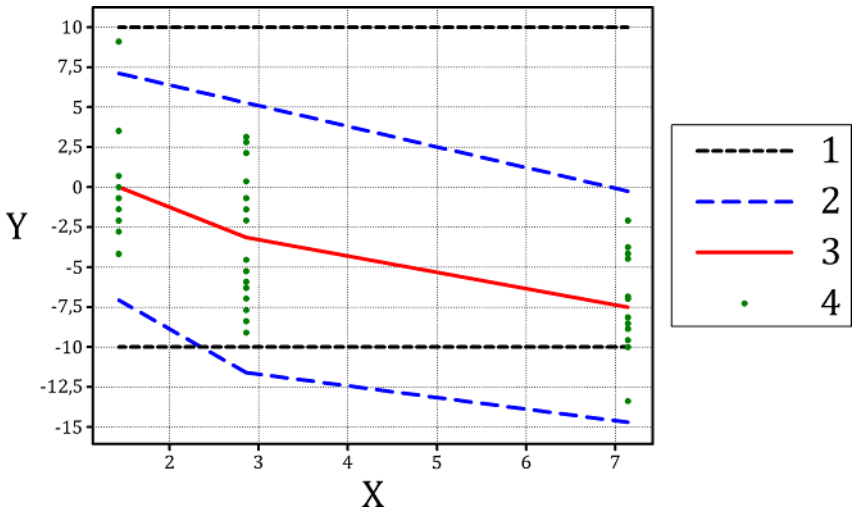
Y: risk of measurement outside acceptance limits

Figure A.6 — Risk profile (TC)

A.6.3 Inorganic carbon

Table A.18 — Results obtained for the accuracy criterion (IC)

Method accuracy				
Level	**Mean of quantities introduced** (mg)	**Beta-expectation tolerance limit** (mg)	**Relative beta-expectation tolerance limit** (%)	**Risk** (%)
1	1,430	[1,329; 1,532]	[- 7,066; 7,143]	0,9056
2	2,860	[2,528; 3,010]	[- 11,62; 5,244]	5,522
3	7,150	[6,098; 7,132]	[- 14,71; −0,2578]	22,52



Key

Axis: X: amount (mg)
Y: relative error (%)

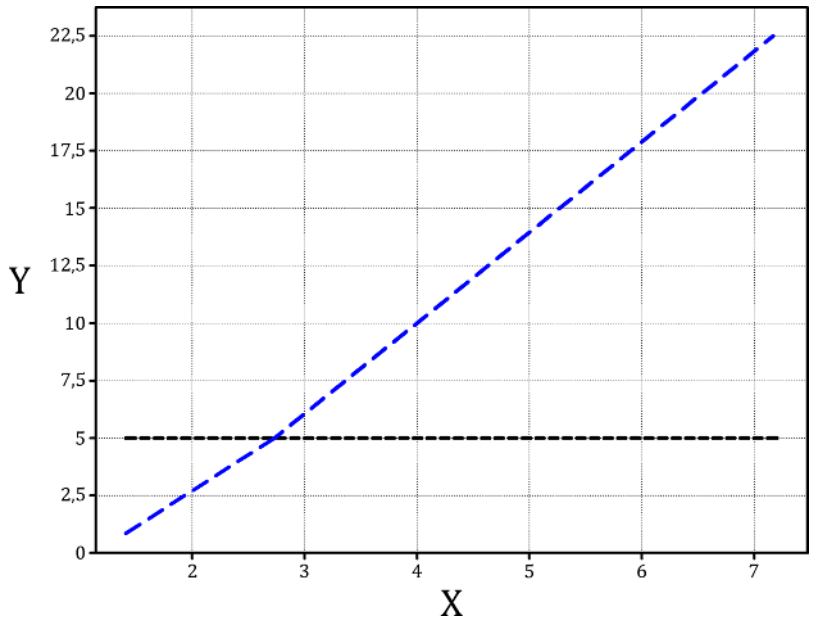
1 acceptance limits (set to 10 %)

2 tolerance interval limits

3 bias

4 relative concentration error

Figure A.7 — Accuracy profile (IC)



Key

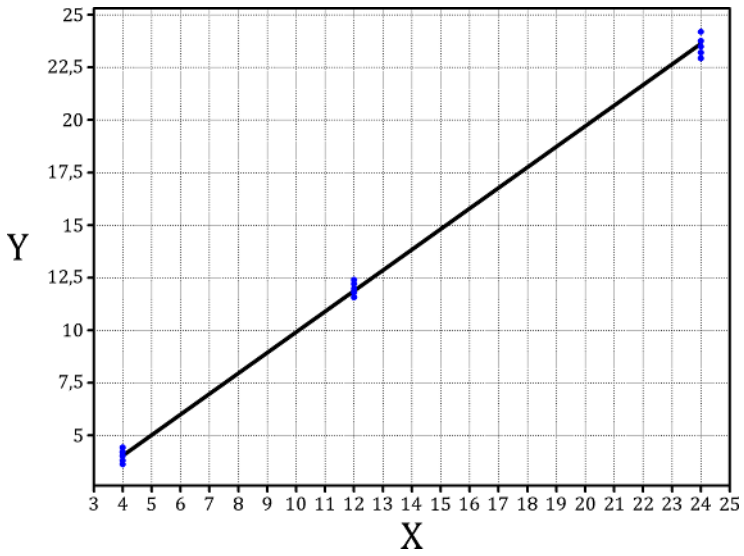
Axis: X: amount introduced (mg)

Y: risk of measurement outside acceptance limits

Figure A.8 — Risk profile (IC)

A.7 Linearity of results

A.7.1 Total carbon



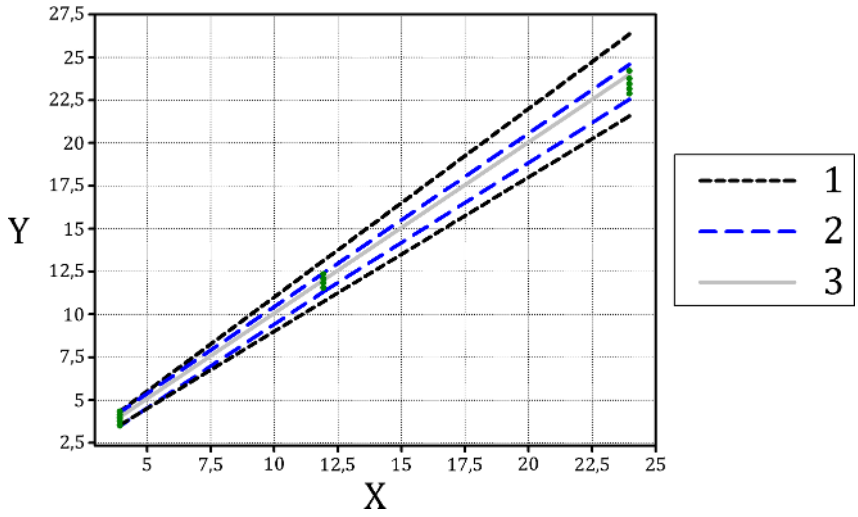
Curve formula: Y = 0,053 83 + 0,981 1 × R^2 = 0,998 6

Key

Axis: X: amount introduced (mg)

Y: results (mg)

Figure A.9 — Linearity study (TC)



Key

Axis: X: amount introduced (mg)

Y: results (mg)

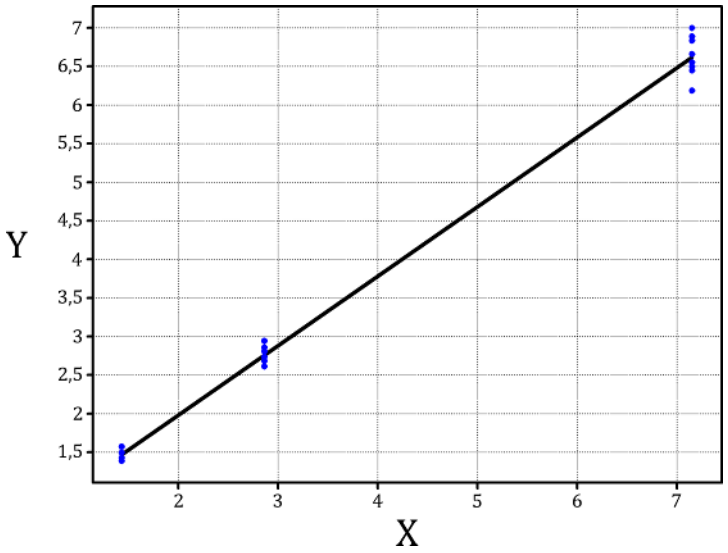
1 acceptance limits (in amount)

2 tolerance interval limits

3 line X = Y

Figure A.10 — Linearity profile (TC)

A.7.2 Inorganic carbon



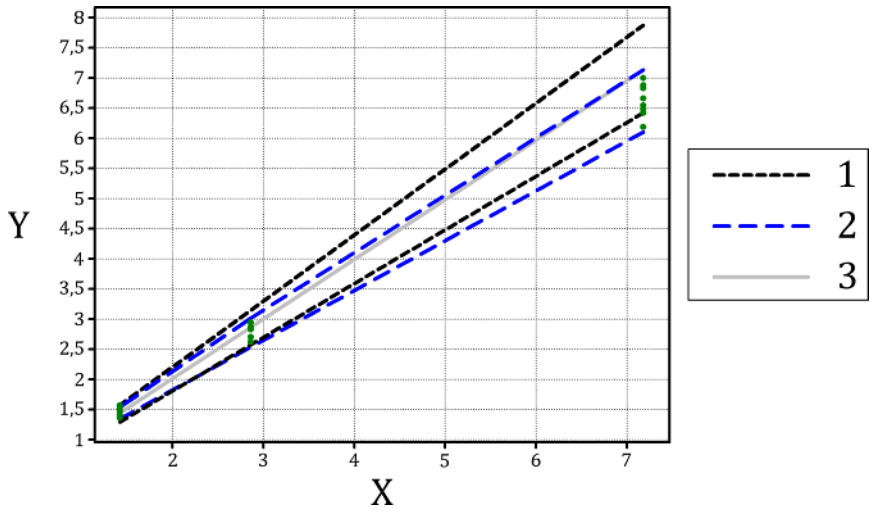
Curve formula: Y = 0,157 2 + 0,904 1 × R^2 = 0,996 3

Key

Axis: X: amount introduced (mg)

Y: results (mg)

Figure A.11 — Linearity study (IC)



Key

Axis: X: amount introduced (mg)
Y: results (mg)

1 acceptance limits (in amount)

2 tolerance interval limits

3 line X = Y

Figure A.12 — Linearity profile (IC)

A.8 Quantification limit and dosage interval

A.8.1 Total carbon

Lower quantification limit = 6,308 (mg).

Upper quantification limit = 24,00 (mg).

A.8.2 Inorganic carbon

Lower quantification limit = 1,430 (mg).

Upper quantification limit = 2,352 (mg).

A.9 Conclusion

The mean recovery rates for TC and TIC are between 90 % and 110 % with a variance coefficient ≤ 5 %. The method is deemed to be validated.

Annex B
(informative)

Measurements on wood species and wood-based materials

By way of example, various different wood species and wood-based materials were measured by the FCBA [timber certification body] Technological Institute on 6 test portions. The results are presented below.

NOTE 1 The exact origin of the various samples below is not known.

NOTE 2 The moisture content of the samples analysed is between 6 and 8 %.

Table B.1

Material/species	TC (%)	TIC (%)	TOC (%)
Oak	46,5	< LOQ[a]	46,5
Douglas	47,4	< LOQ[a]	47,4
Spruce	46,2	< LOQ[a]	46,2
Maritime pine	49,4	< LOQ[a]	49,4
Scots pine	46,9	< LOQ[a]	46,9
Fir	47,3	< LOQ[a]	47,3
Curupixa	46,2	< LOQ[a]	46,2
Tauari	46,9	< LOQ[a]	46,9
Plywood	45,6	< LOQ[a]	45,6
MDF	45,5	< LOQ[a]	45,5
OSB	47,2	< LOQ[a]	47,2
Particle board	46,3	< LOQ[a]	46,3

[a] LOQ: Limit of quantification.

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