
**Solid biofuels — Determination of
off-gassing and oxygen depletion
characteristics —**

Part 1:
**Laboratory method for the
determination of off-gassing and
oxygen depletion using closed
containers**

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Contents

	Page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Apparatus	3
5.1 General	3
5.2 Test containers	3
5.3 Gas sampler	5
5.4 Ovens	5
5.5 Gas chromatograph (GC) analyser	5
6 Biomass sampling and sample preparation	6
6.1 General	6
6.2 Test sample characterization	6
6.3 Test sample size	6
7 Procedure	6
7.1 Determination of porosity in biomass test sample	6
7.2 Filling of test containers	7
7.3 Test container arrangement and test gas sampling volume	7
7.4 Operation of temperature-controlled ovens	8
7.5 Gas sampling procedure	8
7.6 Gas analysis	9
8 Calculation	9
9 Test report	13
Annex A (normative) Quantification of gas species using chromatography	14
Annex B (informative) Estimation of ventilation requirements for enclosed spaces	16
Annex C (informative) Determination of gas species concentration in open storage space	19
Bibliography	20

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 238, *Solid biofuels*.

A list of all parts in the ISO 20048 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

There is a continuous global growth in production, storage, handling, bulk transport and use of solid biofuels especially in the form of pelletized biofuels.

The specific physical and chemical characteristics of solid biofuels, their handling and storage can lead to a risk of fire and/or explosion, as well as health risks such as intoxication due to exposure to carbon-monoxide, asphyxiation due to oxygen depletion or allergic reactions.

Emission from pellets or biomass stored in enclosed space represents a significant health risk due to exposure to carbon-monoxide (CO) and oxygen depletion. It is important to be able to assess the risk by quantifying the emission of CO in combination with oxygen level. This document describes a method for estimating the propensity of a particular quality of pellets or biomass to emit CO, CO₂, CH₄ as well as the depletion of oxygen within the stored environment. In a confined space, the gas composition can result in a toxic as well as explosive atmosphere.

Biomass species, age of the material as well as the ambient temperature impacts the dynamics of the gas emissions. Unless the level of CO and oxygen levels are well understood in an operating environment, there are inherent risk for workers, which have implications for liability.

This document specifies the methodology for measuring the emission and depletion factor and emission and depletion rate of off-gassing in combination with oxygen depletion for permanent gases emitted in an enclosed storage for biomass.

NOTE A method to be used in preliminary screening of CO for operational planning is currently under development within ISO/TC 238/WG 7. Stage at the time of publication ISO/CD 20048-2:2018.

The method described in this document uses highly sensitive gas chromatography to be able to identify the spectrum of gases and their relative concentration to predict the potential for unhealthy conditions during indoor storage of biomass. The sensitivity for detection of gas species and concentrations is only limited by the sensitivity of the chromatographic instrument. The method allows for estimation of emission and depletion factor and emission and depletion rate for each gas species of biomass at different storage temperatures.

The gas instrument analysis part of the method also allows for identification of gas species and determination of concentrations of gases sampled in open storage spaces for occupational hygiene purposes ([Annex C](#)).

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Solid biofuels — Determination of off-gassing and oxygen depletion characteristics —

Part 1:

Laboratory method for the determination of off-gassing and oxygen depletion using closed containers

1 Scope

This document defines a method for determination of off-gassing (permanent gases) and oxygen depletion from woody as well as non-woody biomass, including densified materials such as pellets and briquettes, as well as non-densified materials such as chips. The method is also applicable for thermally treated materials, including torrefied and carbonized materials.

The emission and depletion factor and emission and depletion rate for various gas species emitted from sample within a closed test container is determined by means of gas chromatography.

The emission and depletion factor and emission and depletion rate provide guidance for ventilation requirements to keep gas concentrations below Permissible Exposure Levels (PEL) in spaces where workers can be exposed to the enclosed atmosphere.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 18135, *Solid biofuels — Sampling*

ISO 14780, *Solid biofuels — Sample preparation*

ISO 17827-2, *Solid biofuels — Determination of particle size distribution for uncompressed fuels — Part 2: Vibrating screen method using sieves with aperture of 3,15 mm and below*

ISO 17828, *Solid biofuels — Determination of bulk density*

ISO 18134-1, *Solid biofuels — Determination of moisture content — Oven dry method — Part 1: Total moisture — Reference method*

ISO 18134-2, *Solid biofuels — Determination of moisture content — Oven dry method — Part 2: Total moisture — Simplified method*

ISO 18846, *Solid biofuels — Determination of fines content in quantities of pellets*

ISO 18847, *Solid biofuels — Determination of particle density of pellets and briquettes*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

emission factor

concentration in percent of a gas species relative to other gases in a volume and expressed in gram per kilogram of the substance emitting at a given temperature

3.2

depletion factor

concentration in percent of a gas species relative to other gases in a volume and expressed in gram per kilogram of the substance depleting at a given temperature

3.3

emission rate

concentration in percent of a gas species relative to other gases in a volume and expressed in gram per kilogram per day of the substance emitting at a given temperature

3.4

depletion rate

concentration in percent of a gas species relative to other gases in a volume and expressed in gram per kilogram per day of the substance depleting at a given temperature

3.5

ppmv

parts per million on volume basis

3.6

gas chromatograph

GC

instrument used in analytical chemistry for separating and analysing compounds that can be vapourized without decomposition

3.7

Permissible Exposure Level

PEL

regulatory limit on the amount or concentration of a substance in the air

Note 1 to entry: This is usually based on an eight-hour time weighted average, but some are based on short-term exposure limits.

4 Principle

One or more test container(s) sealed with an air-tight lid and partly filled with biomass test sample are placed in oven with controlled temperature such as 20 °C, 30 °C, 40 °C or 50 °C. Gas samples are drawn by means of a syringe through the sampling port of the container(s) and the relative concentration of gas species is quantified by means of a gas chromatograph. The concentration is converted from a volume fraction in % relative to other gases in the test container and expressed as emission and depletion factor in gram per kilogram of biomass at a given temperature. The emission and depletion rate are expressed as gram of gas species per kilogram of biomass per day at a given temperature.

A method for converting emission and depletion factor (ppmv) concentration and calculating the number of air exchanges in a space with controlled ventilation is provided in [Annex B](#).

5 Apparatus

5.1 General

All equipment holding biomass samples and gas samples extracted during the determination shall be free of any contaminants, well ventilated and dry before the off-gassing test starts.

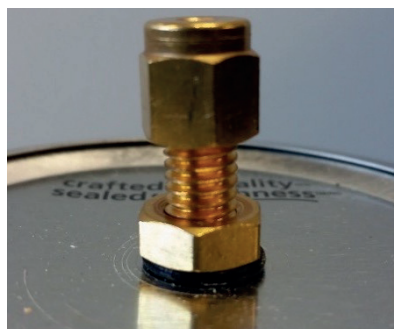
NOTE Containers and fittings can be dried overnight at low temperature around 30 °C.

5.2 Test containers

The test container(s) shall preferably be made of glass, not plastic, due to the risk of contaminating gases from plastic materials at higher temperatures. Since the containers shall only be filled to 75 % with biomass to be tested, it is an advantage to be able to see the level of biomass from the outside. [Figure 1](#) a) to [1 c\)](#) show photos of the test container with sampling port and [Figure 2](#) shows a schematic of the test container and sampling port.



a) Test container of glass with sampling port

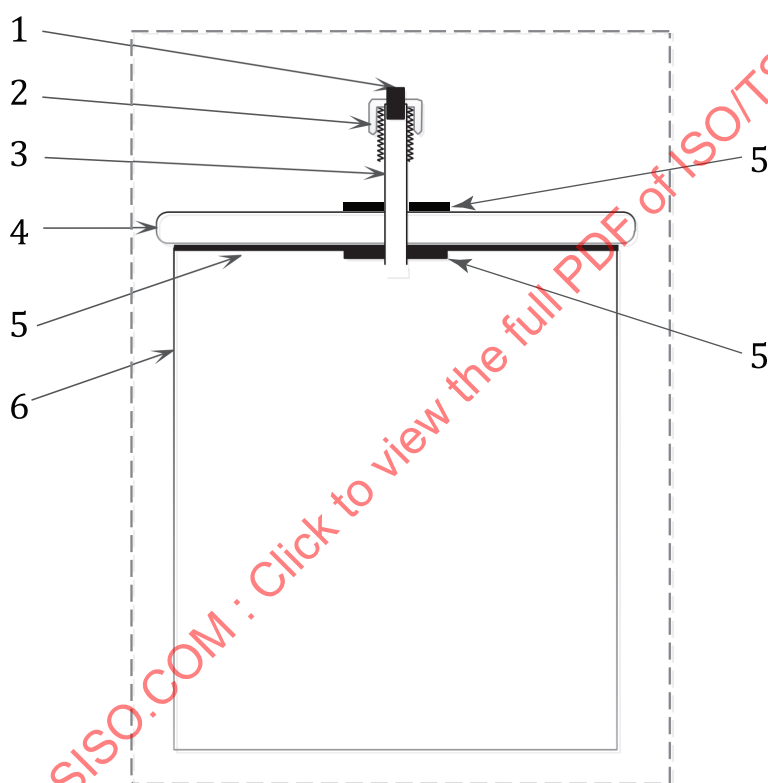


b) Sampling port, from the side



c) Sampling port, from above

Figure 1 — Example of test container of glass with sampling port



Key

- 1 septum
- 2 nipple
- 3 sampling port
- 4 container lid
- 5 air-tight seal
- 6 test container

Figure 2 — Schematic of test container with sampling port

The headspace in an enclosed container shall contain sufficient oxygen to sustain oxidation of test sample to reach a peak (plateau) and allow determination of the emission and depletion factor^{[1][2]}. The 25 % headspace of enclosed air volume under roof in a typical large-scale storage facility such as a silo when fully loaded is typical and is therefore selected for this test method.

The seal between the lid and the container as well as the sampling port nipple (septum) shall be made of polytetrafluoroethylene (PTFE) or neoprene, which are non-reactive materials at the temperatures recommended for the off-gassing tests. Gas samples shall be drawn using a syringe (see 5.3) piercing through the septum.

The effective gas volume in a test container can be expressed in accordance with [Formula \(1\)](#).

$$V = V_h + V_v = 0,25 \times V_c + V_v \quad (1)$$

where

V is the effective gas volume in test container when filled with biomass;

$V_h = 0,25 \times V_c$ is the selected headspace volume;

V_v is the volume of void between the biomass particles;

V_c is the volume of empty test container.

EXAMPLE

The effective gas volume (V) for a test container with a volume of 3 500 ml (V_c) loaded to 75 % with wood pellets and with a volume of void of 50 % can be calculated as follows:

$$V = 0,25 \times 3\,500 \text{ [ml]} + 0,75 \times 3\,500 \text{ [ml]} \times 0,5 = 3\,500 \text{ [ml]} \times 0,625 = 2\,188 \text{ ml}$$

Guidance for selecting container size in relation to gas sample size required by the GC for a selected gas depletion volume is provided in [7.3](#).

5.3 Gas sampler

A gas-tight GC syringe shall be used for drawing gas test samples through septum in the container sampling port nipple. It is recommended that the capacity of the syringe be at least 3 times the volume of the sampling tube and sampling loop of the GC or as recommended by the manufacturer of the GC (see 5.5). The syringe shall have a scale with a resolution of 1 ml and a valve to secure the sample after drawing. It is best to use needles that have a hole on the side rather than the tip to prevent silicone or neoprene material blocking the hole while sampling.

The gas sample is injected directly from the sampler syringe into the GC sample port.

5.4 Ovens

The temperature within the test containers shall be controlled by placing the containers in ovens automatically controlling the temperature in the range of 20 °C to 50 °C $\pm$ 1 °C. A separate oven is required for each temperature selected for testing. The ovens shall be able to hold the size of containers required to achieve the necessary accuracy of the off-gassing determination.

Since temperature of biomass under test has a propensity to self-generate heat^[4] at testing temperatures above 40 °C, particularly if the moisture in the material is high, a thermocouple should be placed inside the material in one of the containers. A thermocouple in the centre of the test volume will help monitoring the uniformity of the temperature.

5.5 Gas chromatograph (GC) analyser

The detection limit for each gas species and related concentrations is determined by the type of column in the GC. The manufacturer of the GC should be consulted. GC with thermal conductivity detector (TCD) shall be used to detect and quantify permanent gases and light hydrocarbons. Packed and capillary

columns could be used with TCD to measure permanent gases. A combination of TCD and FID (flame ionization detector) could also be used for gas measurements depending on the GC configuration.

NOTE 1 Helium (He) is usually used as carrier gas but, e.g., nitrogen or argon are other possible alternatives.

NOTE 2 PEL for CO is in the range of 25 ppmv to 100 ppmv depending on jurisdiction and on the duration of the exposure. Gas chromatography allows identification of a large number of non-condensable like CO, CO₂, CH₄, N₂, H₂, and O₂. The PEL for those compounds can be found in occupational hygiene databases. The occupational health lower limit for oxygen is 19,5 %.

[Annex A](#) provides a generic orientation of operation and calibration of a GC.

6 Biomass sampling and sample preparation

6.1 General

Sampling and sample preparation of biomass shall be done in accordance with ISO 18135 and ISO 14780 respectively.

6.2 Test sample characterization

The test sample characterization shall be done in accordance with the following international standards;

- | | |
|-------------------------------|----------------------------|
| a) Moisture | ISO 18134-1 or ISO 18134-2 |
| b) Particle size distribution | ISO 17827-2 |
| c) Fines content | ISO 18846 |
| d) Bulk density | ISO 17828 |
| e) Particle density | ISO 18847 |

If available, note the origin, species and age of the test sample in the test report ([Clause 9](#)).

6.3 Test sample size

The total sample size depends on the test container configuration selected ([7.3](#)). At least three test sample fractions shall be prepared; one for test sample characterization ([6.2](#)) and the others for off-gassing/oxygen depletion tests.

EXAMPLE

Volume of material required per temperature test is $V_c \times 4$ plus required volume for characterization depending on the selected test under [6.2](#). Material for each additional test temperature requires $V_c \times 4$.

7 Procedure

7.1 Determination of porosity in biomass test sample

The characteristics of the biomass test sample can vary depending on shape and size of the material as well as amount of entrained dust.

For pellets the bed porosity (or bulk porosity of the bed) is determined using [Formula \(2\)](#)^[7]:

$$\varepsilon = 1 - \frac{\rho_b}{\rho_d} \quad (2)$$

where

ρ_b is the bulk density (ISO 17828) and

ρ_d is the single particle density (ISO 18847) of wood pellet.

7.2 Filling of test containers

Mark the test container at the 75 % level.

Fill each container to the 85 % level with the biomass test material to be tested. In order to achieve a representative packing density, expose the test container to shock by means of dropping the container 5 times from a height of 50 mm onto a wooden board on an even horizontal and hard workbench or floor. Make sure the test container hits the wooden board in a vertical position and that the level of test material reaches the 75 % level. Refill or remove material if necessary, to reach the 75 % level. This procedure shall be repeated until the packing density is stable.

If more than one test container is used, mark the containers with different letters A, B, C etc., and weigh the content of each container to make sure each container has the same weight within 1 % of weight.

If there is dust remaining on the rim of the test container, swipe it off with a cloth.

Apply the lid assembly with the sampling port to the test container and seal it. Tighten all connections. To make it air-tight, use an extra sealant, such as silicone, around the fittings on the side of the lid that is exposed to air when the container is closed. No extra sealant shall be used on the inner side of the lid. Ensure that the sealant chosen can be used in the temperatures it will be exposed to during the test.

The container called PEAK is sampled only once when the sampling is completed. It is to verify the peak emission value at the end of the test period.

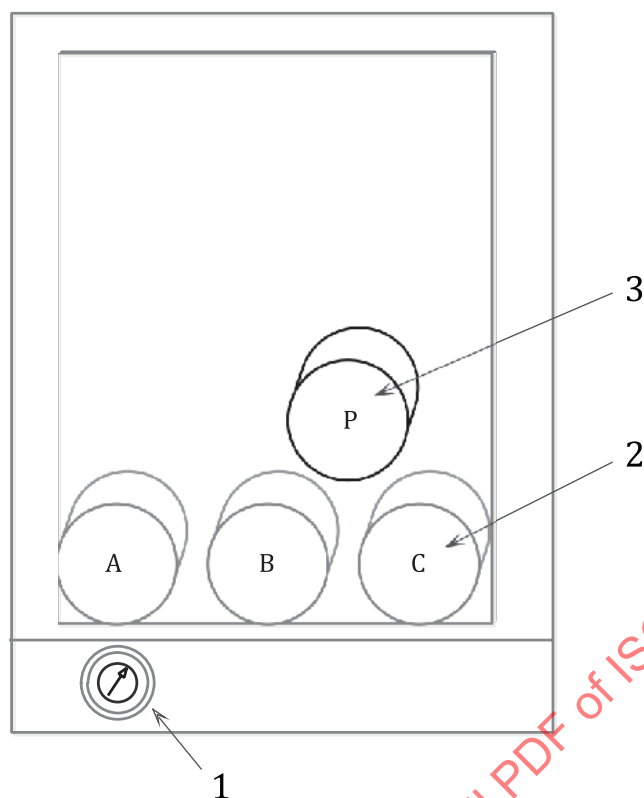
7.3 Test container arrangement and test gas sampling volume

The different gas species evolve at different rates, which mean that the relative ratio of gases can change slightly over time as the oxygen is consumed. Also, the temperature can affect the relative rate of evolution of the various gas species. The oxygen content in the containment is depleted as a function of oxidation of components of the biomass. In order to obtain a representative profile of emissions for long term storage, emission and depletion factors and emission and depletion rates for gas species shall be defined over an extended period of time such as 28 to 30 days. Due to this relatively long period of time, considerations shall be given to testing more than one biomass test material at a time. For example, reference material and several test samples can be processed in parallel. If testing is done at various temperatures such as 20 °C/30 °C/40 °C/50 °C there shall be one oven per temperature.

NOTE Using more than one oven per temperature is not recommended since there will be some difference in the temperature which could affect the results.

Consideration shall be given to maximum allowable gas depletion due to sampling and the number of days samples should be taken.

[Figure 3](#) illustrates arrangements of four test containers in an oven.



Key

- 1 oven with temperature regulator
- 2 test containers marked A, B and C
- 3 reference test container marked PEAK

Figure 3 — Schematic of oven and test container configuration

7.4 Operation of temperature-controlled ovens

The temperature of the ovens has to be stabilized at the selected temperature before the test container(s) are loaded into the ovens. Ideally there should be multiple ovens operated in parallel with temperatures controlled at 20/30/40/50 °C or other selected temperatures to run the tests in parallel to save time and to make sure the material is of the same age.

NOTE If the room temperature is fairly stable, the 20 °C test can be done without an oven.

7.5 Gas sampling procedure

Remove the test containers from the oven during the sampling and rotate it along its axis at least once before the syringe is inserted to draw the gas sample. This will mix the gases and minimize the potential for un-even distribution of gases within the test container. During sampling the door of the oven shall be open as short time as possible and the test containers shall not be outside the oven more than necessary.

After completion of sampling, place the test containers in the oven. Use gloves, protective glasses and other safety gear when handling the test containers, especially at higher temperatures.

NOTE The GC described in this document can also be used for determining concentration of gas spectrum and concentrations in open spaces where personnel are present and where occupational health can be an issue^[6]. The procedures for gas sampling in that case is described in [C.2](#).

7.6 Gas analysis

Transfer the gas sample from the GC syringe to the sample port of the GC as soon as possible after sampling is completed. The syringe shall not be used as a storage device for the gas sample. The sample should be injected in accordance to instructions for the GC and injected slowly to ensure effective purging of the GC sampling tube and loop.

Before conducting the tests, calibrate the GC preferably with three levels of standard gases as specified in [Annex A](#). The standard gases shall contain known concentrations of the most prominent gas to be quantified such as CO, CO₂, CH₄, O₂, N₂ and H₂ and other gases of interest in various concentrations to develop a calibration range. For each day of testing, inject one sample of standard gas before the test and another sample of standard gas after the test gas sample so as to ensure steady and accurate readings.

8 Calculation

The concentration of off-gasses can be derived from the kinetic reaction equation in [Formula \(3\)](#).

$$f_i(t) = f_{i\infty} [1 - \exp(-k_i t)] \quad (3)$$

where

$f_i(t)$ is the instantaneous emission and depletion factor for gas species i (g/kg);

$f_{i\infty}$ is the asymptote Emission Factor (g/kg);

k_i is the kinetic reaction rate constant (d⁻¹) (emission and depletion rate);

t is the time (d).

A GC is usually calibrated with standard gases mixed on a volumetric basis. The results from GC readings are in volume fraction of a specific gas in relation to other gases in the containment and shall be converted to mass fraction at a given temperature.

[Formula \(4\)](#) expresses the emission and depletion factor f_i in gram of gas per kilogram of test material as a function of volumetric gas concentration C_i at constant temperature T (in K) and pressure P (pressure P does not change very much at the temperature range used in this test procedure and can be approximated to be constant)^[3].

$$f_i = \frac{P(C_i V) M_i}{RTm} \quad (4)$$

where

R is the gas constant (8,31 J/mol.K);

T is the temperature (K);

M_i is the molar mass (g/mol);

m is the mass of material in the container (kg);

V is the effective gas volume in the reactor (m³), [Formula \(1\)](#);

P is the absolute pressure of the container (Pa);

C_i is the volumetric concentration of a particular off-gassing specimen measured by GC, expressed as fraction volume of the specific gas in relation to other gases in the container.

EXAMPLE This example illustrates the procedure of converting the C_i value as established by a GC for carbon monoxide (CO) and converting it to emission factor f_i for a sample of biomass. This value does not account for the accumulation of off-gas over a period of time in an unventilated space.

$$\begin{aligned}
 R &= 8,31 \text{ J/mol.K);} \\
 T &= 20 \text{ °C} = 293,15 \text{ K;} \\
 M_{\text{CO}} &= 28,01 \text{ g/mol;} \\
 m &= 1 \text{ kg;} \\
 V &= 1 \text{ l} = 0,001 \text{ m}^3, \text{ Formula (1);} \\
 P &= 1 \text{ atm} = 101,325 \text{ kPa} = 101\,325 \text{ Pa;} \\
 C_i &= 0,1 \% = 0,001 \text{ as established by the GC analysis procedure;} \\
 f_i &= 101\,325 \times 0,001 \times 0,001 \times 28,01 / (8,31 \times 293,1 \times 1) = 0,00\,168\,7 \text{ g/kg, Formula (4).}
 \end{aligned}$$

The results shall be calculated to two decimal places. Table 1 illustrates C_i and the calculated emission and depletion factor f_i in g/kg using Formula (1) and Formula (3).

Table 1 — Illustration of emission factor f_i as a function of C_i for CO₂

d	C_i (%) for CO ₂	$V = 0,25 \times V_c + V_v$ (m ³) [Formula (1)]	f_i for CO ₂ (g/kg) [Formula (4)]
1	0,111 6	0,001 1	0,002 284
2	0,221 0	0,001 1	0,004 524
4	0,355 3	0,001 1	0,007 273
6	0,394 7	0,001 1	0,008 08
8	0,432 1	0,001 1	0,008 846
9	0,433 1	0,001 1	0,008 866
10	0,456 3	0,001 1	0,009 341
12	0,454 4	0,001 1	0,009 302
14	0,463 5	0,001 1	0,009 489
15	0,491 4	0,001 1	0,010 059
17	0,491 0	0,001 1	0,010 052
19	0,491 4	0,001 1	0,010 06
22	0,491 7	0,001 1	0,010 066
24	0,491 6	0,001 1	0,010 064
32	0,496 9	0,001 1	0,010 172

The time period of test shall be long enough for the last three subsequent measurements of C_i to deviate less than 5 % from each other.

The f_i can be plotted as a function of time by curve fitting the f_i data with Formula (3) using one of the many curve fitting programs commercially available. Figure 4 illustrates the plot of the data in Table 1 and provides the equation and the k_i/d value (emission rate in gram gas/kg biomass) for the curve fitted to Formula (4). Also, the peak (asymptote) value of the concentration of CO₂ in an unventilated space can be read from the plot. Figure 5 shows the oxygen depletion for wood pellet with 4 % moisture content (MC) over 62 d at +25 °C, +40 °C and +60 °C.

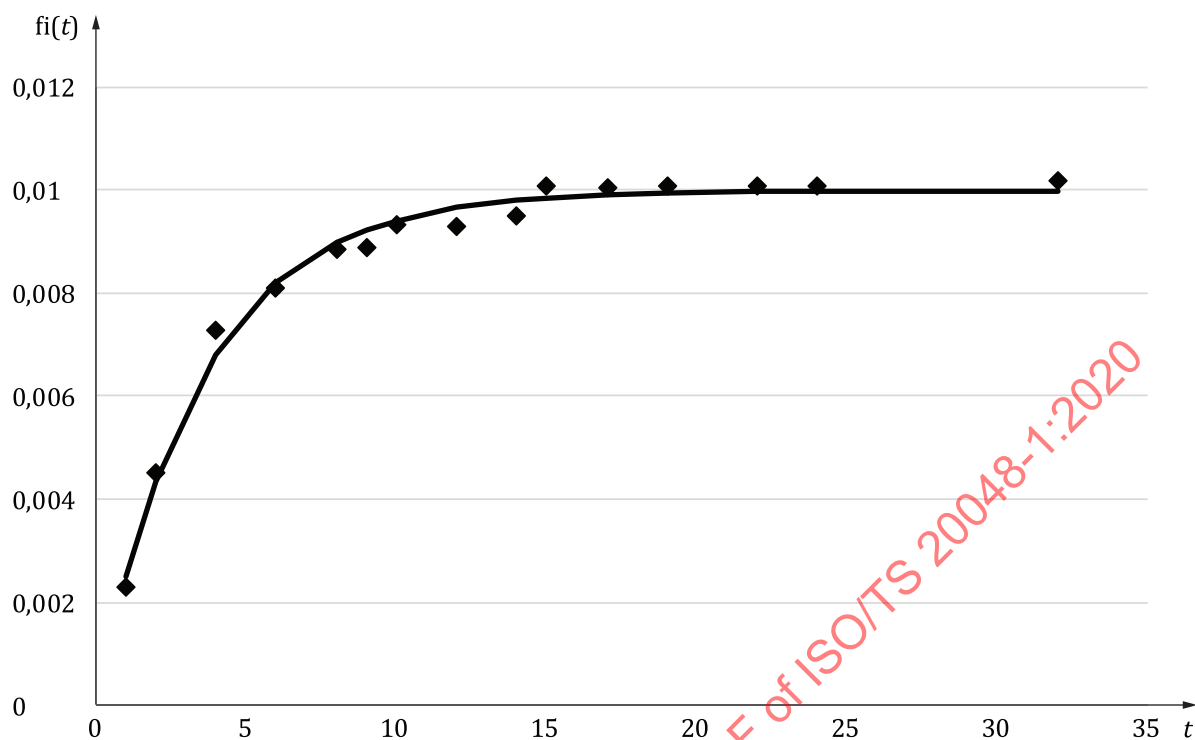


Figure 4 — Plot of emission factor f_i in g/kg of test sample vs. storage time t in days for CO,

$$f_{i\infty} = 9,975\,8E-03 \left(\frac{\text{kg}}{\text{g}} \right), k_i = 0,286\,7 \text{ day}^{-1}$$

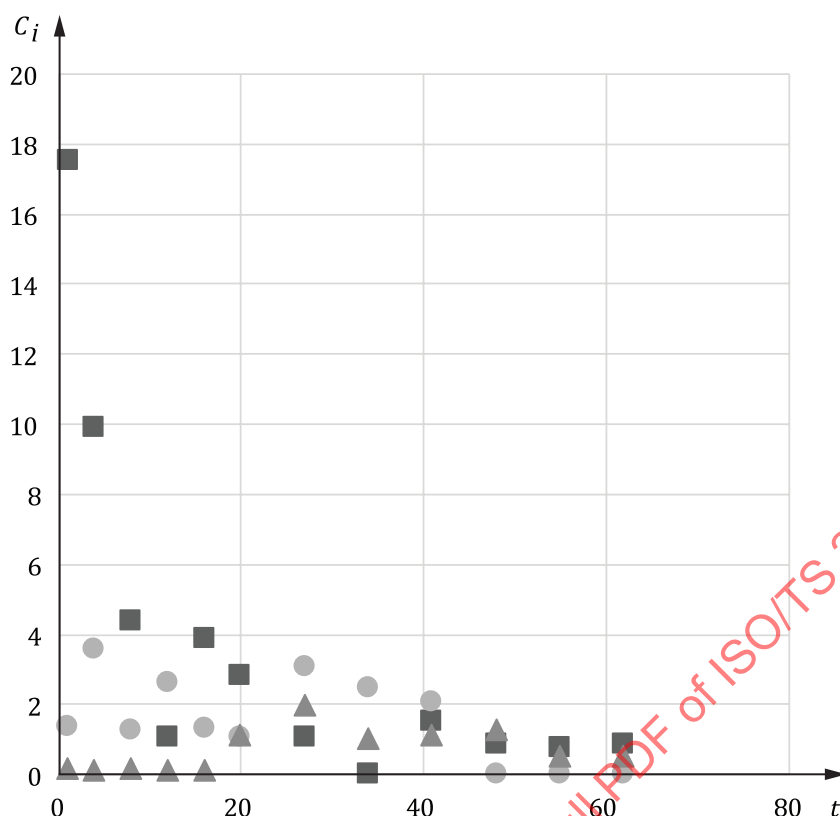
**Key** C_i oxygen concentration in % t storage time [d]■ oxygen concentration after storage in t [d] at 25 °C● oxygen concentration after storage in t [d] at 40 °C▲ oxygen concentration after storage in t [d] at 60 °C

Figure 5 — Oxygen depletion for wood pellet with 4 % moisture content over 62 d at 25 °C, 40 °C and 60 °C

For storage tests conducted at several temperatures, it is recommended to tabulate the test data values of $f_{i\infty}$ and k_i as illustrated in [Table 2](#) for each gas and for each test temperature T .

Table 2 — Illustration of tabulation of emission factor f_i for different gas species and at different temperatures

Gas species	Temperature °C	$f_{i\infty}$ (g/kg)	k_i (d)–1
Gas species 1	T1		
	T2		
Gas species 2	T1		
	T2		
etc.			

NOTE For conversion of concentration in mass fraction (g or mg of gas per kilogram of biomass) to concentration in volume fraction (% or ppmv), see [Annex B](#).

[Annex B](#) illustrates how to estimate the need for ventilation, for example when evaluating gas concentration in a space for a gas species.

9 Test report

The test report shall include at least the following information:

- a) identification of the laboratory performing the test and the date of the test;
- b) characteristics of the biomass tested as well as any reference material, including:
 - moisture content;
 - fines content;
 - particle size distribution;
 - void volume;
 - origin, species and age of the material;
- c) test arrangement, including:
 - number and volume of test containers;
 - testing temperatures;
 - length of test;
 - type and model of GC;
- d) results from testing, including emission factor, emission rate and plot of emission rate versus time;
- e) reference to this document;
- f) any deviation from the instructions in this document, or operations regarded as optional, and
- g) any unusual condition and observation during the testing such as accumulation of condensate in sampler, self-heating in test sample etc.

Annex A (normative)

Quantification of gas species using chromatography

A.1 General

Gas chromatography is a laboratory technique that separates gas mixtures into individual fractions. When a gas is passed through a GC column some gas fractions are retarded more than others and separated in time. The column typically is a fused silica capillary with an inner diameter of 0,1 mm and length 50 m.

The gas stream containing the separated gas fractions passes by detectors such as a Flame Ionization Detector (FID) or Thermal Conductivity Detector (TCD), which will respond to the fractions as they occur by changing its electrical output. The output from the detector becomes the chromatogram.

When pure carrier gas (e.g. argon, helium, hydrogen, nitrogen) passes a detector a baseline signal is generated. Each peak in the chromatogram represents a different fraction of the test sample gas mixture. The retention time separates each species and the peak size (height or area) is a measure of the amount of concentration. The carrier gas shall be pure to 99,999 5 %. Contaminants can react with the sample or the column and create spurious peaks or load the detector and raise the baseline. Injection in the sample port of the GC of a high-purity gas free of hydrocarbons and moisture separated by a filter is recommended for verification purposes.

In case the sample contains condensable gases with a dew point temperature lower than the room temperature, there can be moisture or droplets of liquids forming inside the syringe. Samples with liquids shall not be transferred to the GC since it can damage the equipment or corrupt the calibration of the instrument. Caution should be taken with the liquid since the condensate can consist of acidic substances generated by the test sample during decomposition under heat.

The GC should be equipped with a membrane filter between the syringe and the GC sample port to separate moisture.

A.2 Calibration of gas chromatograph

In general, a GC is calibrated with pre-mixed gas on a volumetric basis. Thus, the results of measurements with a GC provide a measure in volume of the gas species under measurement as fraction per volume of composite gas going through the GC.

Standard gas mixtures covering a range of gas species concentrations as illustrated in [Table A.1](#) can be used for calibration. The concentration shall be in the same concentration as expected of the test sample, for example 0,05 % to 2,5 % for CO and 0,1 % to 6 % for CO₂. The following three standard calibration gas mixtures are examples available in the market. At least three injections of each calibration gas should be done to ensure GC accuracy.

Table A.1 — Examples of standard gas mixtures for calibration of GC

Standard gas 1		Standard gas 2		Standard gas 3	
CO ₂	0,1	CO ₂	0,5	CO ₂	6,0
CO	0,05	CO	0,1	CO	2,5
CH ₄	0,5	CH ₄	0,1	CH ₄	1,5
H ₂	0,5	H ₂	1,0	H ₂	0,0

Table A.1 (continued)

Standard gas 1		Standard gas 2		Standard gas 3	
O ₂	21,0	O ₂	21,0	O ₂	10,0
N ₂	79,0	N ₂	79,0	N ₂	3,0
He	0	He	0	He	2
Ar	0	Ar	0	Ar	balance

[Table A.2](#) illustrates data from a typical calibration procedure for CO₂.

Table A.2 — Data from a typical calibration procedure for CO₂.

Sample	CO ₂	Mean area	Calibration formula	a, b and c factors in formula
Standards gas 1	0,1	386 999	$y_{\text{CO}_2} = ax^2 + bx + c$	a = 6,601 51E-14
Standards gas 2	0,5	166 215 9		b = 1,936 281E-7
Standards gas 3	6,0	817 902 1		c = 0 R2 = 0,999 9

NOTE The "y" is the gas concentration and the "x" is the area under the curve of the GC reading.

Annex B (informative)

Estimation of ventilation requirements for enclosed spaces

B.1 General

Smaller storages of biomass such as wood pellets do not have active ventilation which means accumulation of off-gas and risk for exposure to toxic environment during entry. Many of the larger industrial storages on the other hand can have active ventilation, either at the top or at the bottom through louvers or grates for exhaust of off-gas as well as for controlling the temperature. Concentration of off-gas in a storage can be estimated based on emission and depletion factor or emission and depletion rate ([Clause 8](#)).

The following examples illustrates how to convert emission and depletion factor for CO from mass fraction (g/kg or mg/kg) to gas concentration in ppmv and mg/m³.

NOTE The examples are only intended to illustrate the procedure for calculation and the numbers used are highly dependent upon characteristics of the biomass such as species, bulk density, fines content, age and storage conditions, including size of headspace etc.

B.2 Converting gas concentration from mg/kg to ppm on a mass basis

To convert gas concentration from mg/kg to ppm on a mass bases, use [Formula \(B.1\)](#) or [Formula \(B.2\)](#).

$$1 \text{ mg/kg} = 1 \text{ ppm} \quad (\text{B.1})$$

$$1 \text{ g/kg} = 1\,000 \text{ ppm} \quad (\text{B.2})$$

B.3 Converting gas concentration of CO from mg/kg to mg/m³ in un-ventilated storage space

EXAMPLE

For a flat bottom storage space, filled to 75 % with wood pellets with a bulk density, ρ_b , of 675 kg/m³ and a bed porosity, ε of 48 %, the following calculation is made to convert the measured CO concentration in mg/kg to mg/m³:

- d is the diameter of the silo (m) = 10 m
- h is the height of the silo (m) = 10 m
- V_S is the volume of storage space (m³)
- V_b is the volume of the pellet bed (m³)
- ρ_b is the bulk density of the wood pellets (kg/m³) = 675 kg/m³
- m_b is the mass of the pellets bulk (kg)
- ε is the bulk porosity of the bed = 48 %
- V is the effective gas volume in test container when filled with biomass