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

Part 2: Operational method for screening of carbon monoxide off-gassing

Biocombustibles solides — Détermination des caractéristiques de dégagement gazeux et d'appauvrissement en oxygène —

Partie 2: Méthode opérationnelle d'analyse d'un dégagement de monoxyde de carbone

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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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 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.

Emissions from pellets or biomass stored in enclosed spaces represent 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 levels. This document describes a method for estimating the propensity of a particular quality of pellets or biomass to emit CO, CO_2 and CH_4, as well as the depletion of oxygen within the stored environment. In a confined space, the gas composition can result in a toxic and explosive atmosphere.

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

The ISO 20048 series specifies a methodology to measure the emission of off-gases and depletion of oxygen. ISO/TS 20048-1 specifies a method for measuring the emission and depletion factor and emission and depletion rate of off-gassing in combination with oxygen depletion for gas species emitted in an enclosed storage for biomass. This document specifies a method to be used in preliminary screening of CO for operational planning. The results of the determination method described in this document should only be used for preliminary screening for operational planning. To analyse the potential for off-gassing and oxygen depletion of a densified biofuel, this document presents a standardized operational method which can assess the potential for CO emission.

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

Part 2:
Operational method for screening of carbon monoxide off-gassing

1 Scope

This document specifies an operational method for screening of carbon monoxide off-gassing from solid biofuel pellets. It provides requirements for sampling and establishes procedures for sample handling of solid biofuel pellets prior to the analysis of off-gassing. This document specifies the applicability and use of the method. Guidance on the applicability and use of the data is given.

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 14780, *Solid biofuels — Sample preparation*

ISO 18135, *Solid Biofuels — Sampling*

ISO 21945, *Solid biofuels — Simplified sampling method for small scale applications*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
test sample
laboratory sample after an appropriate preparation made by the laboratory

Note 1 to entry: The test sample is here typically a representative sample from a batch of pelletized biofuels.

[SOURCE: ISO 16559:2022, 3.199, modified — note to entry replaced.]

3.2
test portion
sub-sample either of a *laboratory sample* (3.4) or a *test sample* (3.1)

[SOURCE: ISO 16559:2022, 3.198, modified — note to entry removed.]

3.3
sample
quantity of material (all increments), representative of a larger quantity for which the quality is to be determined

[SOURCE: ISO 16559:2022, 3.168, modified — note to entry removed.]

3.4
laboratory sample
sample (3.3) delivered to a laboratory

[SOURCE: ISO 16559:2022, 3.120, modified — note to entry removed.]

3.5
off-gassing
spontaneous emission of condensable gases (e.g. terpenes) and non-condensable gases (e.g. carbon-monoxide, carbon-dioxide, methane) from biomass

[SOURCE: ISO 16559:2022, 3.141]

3.6
specific maximum CO concentration
maximum measured CO concentration divided by the mass (in kg) of the test portion in the test container

4 Principle

The concentration of CO is measured over time at a given set temperature using an enclosed heating apparatus (e.g. an incubator or an oven) with a fan and the sample container suspended to allow the even distribution of heated air. The method is designed to measure two sets of replicates.

NOTE The sample material is subject to auto-oxidation. The oxygen level in the container gradually decreases and the CO level increases until the oxygen is depleted.

Additionally, off-gassing potential (and self-heating) declines with time, therefore the sample shall be analysed without any time delay.

The sample material may be processed either in an in-house company laboratory or by an external laboratory.

5 Apparatus

5.1 Sample container, nominal volume 1 l, cylindrical container made of glass with a sealed glass or metallic lid for an airtight fit (see Figure 1). Containers without a neck should be used.

The nominal volume of 1 l containers can vary significantly. Limit the actual volume of the containers to 1 045 ml to 1 130 ml.

Figure 1 — Example of sample container

5.2 Heating apparatus (e.g. an incubator or an oven), with an automatic temperature control, capable of keeping the temperature stable at 40 °C (±1 °C). The heating apparatus shall be insulated and equipped with a heating device (approximately 80 W) and a fan.

The heating apparatus shall have an elevated perforated floor to allow the heat to be distributed equally around the containers.

5.3 CO data logger that records CO levels in the range 0 ppm to 1 000 ppm and is small enough to fit into the container. The CO data logger, when placed in the pellet bulk, shall not reduce the container volume by more than 5 %. Data recording should be possible at time intervals of at least one value every 5 min. The CO data logger should have a USB interface for simple data download.

To ensure reproducibility of data and in case a CO logger is faulty and stops working during the experiment, at least two CO loggers of the same kind are required for each type of sample tested.

5.4 Laboratory balance that can determine the mass of samples to an accuracy of 0,1 % of the sample mass.

6 Sample handling

6.1 Sampling

Sampling shall be carried out according to the procedures prescribed in ISO 18135 or ISO 21945. The minimum mass of the test sample for a material to be investigated shall be 1 500 g. This will allow for a measurement with replication. If two types of materials are to be investigated at the same time, 1 500 g of another material shall be sampled as well.

Sample preparation shall be done according to ISO 14780.

The sample history and the conditions for sample handling should be stated as thoroughly as possible in the test report (Clause 10). This includes temperature at the sampling location.

6.2 Sample transport and storage

Ideally, pellets should be sampled and filled in the test containers without any delay, transport or storage. If, however, the facility where the test is carried out is not at the sampling location the laboratory sample shall be transported in an airtight sample container. The container shall be completely filled with the sample. The time between sampling and analysis shall be minimized and elevated temperatures shall be avoided.

NOTE 1 The requirement of an airtight container is to limit the amount of available oxygen in order to limit oxidation reaction with the sample.

NOTE 2 A completely filled container decreases the amount of air in the container (i.e. amount of oxygen) and further reduces deterioration of the sample from physical wear (i.e. reduces the amount of fine fraction).

NOTE 3 It has been shown that a sample can be stored for 2 to 3 months without any significant change in reactivity if placed in a freezer directly upon receipt from the analysis laboratory.

If the pellets are to be kept for some time before being tested, the filled airtight sample container should be kept in a fridge (at 0 °C to 5 °C) until the day of testing.

6.3 Sample preparation

If the pellets are sampled directly after production, they are hot. Put the test sample on trays for 4 h to 6 h so the pellets reach room temperature (20 °C ± 3 °C) before the test.

If the pellets were kept in the fridge before testing, remove the filled containers from the fridge 4 h to 6 h before the test and put them on trays so the pellets reach room temperature (20 °C ± 3 °C).

The pellets shall be put on trays with a maximum pellet layer thickness of 2 cm during the temperature adjustment.

Take a test portion from the test sample. Fill each container to 75 % of its volume in order to provide sufficient supply of oxygen in the container to oxidize the pellets and generate CO. Weigh the pellets inserted in each container to ensure that the test portion mass is the same for all replicants of the same sample.

7 Procedure

7.1 Preheat the heating apparatus (e.g. an incubator or an oven) to 40 °C. Plan accordingly, as the preheating can take some time, see Figure 2.

NOTE Information on preheating time is given in Figure 2.

7.2 Place the CO logger in the container filled with pellets. This can be done easily by keeping the container inclined and inserting the CO logger. Annex A describes another method to insert the CO logger.

Make sure the CO data logger does not reduce the container volume by more than 5 %.

7.3 Assess if the temperature in the heating apparatus has reached the desired 40 °C (±1 °C) and the temperature has been stable for at least 10 min.

7.4 Ensure that the containers are at room temperature (20 °C ± 3 °C) and the lids are free of wood dust. Seal the containers so they are airtight.

7.5 Place the containers inside the heating apparatus at equal distances from each other and from the sides of the box. Seal the heating apparatus immediately.

7.6 The measurement starts as soon as the sealed containers are placed in the heating apparatus and the heating apparatus door is closed, after the heating apparatus preheating phase (see Figure 2). Note the start time.

7.7 Measure the concentration of CO every 5 min for at least 3 h, up to 10 h if possible. Note the end time and remove the containers from the heating apparatus. Record the value after 3 h as $CO_{max\text{-}3h}$.

NOTE At this volume of pellets and temperature exposure, CO off-gassing in most cases reaches its peak after 48 h and no significant amounts of CO are emitted thereafter.

Since 48 h is too long a measurement interval in an industrial setting (where results can be required within a day), the following duration can be used, acknowledging the reduced accuracy of the shorter measurement time (see values for accuracy in Annex A, Figure A.2):

— 3 h (a reliable trend can be seen and $CO_{max\text{-}3h}$ can be recorded);

— 10 h (reliable prediction of the maximum concentration after 8 h is possible; it is possible that the curve has reached a plateau by 10 h of storage).

Compare only samples with the same duration time and the same number of containers in the heating apparatus, because the time of heating up is influenced by the number of containers.

7.8 Remove the CO data logger from each container and extract the recorded data. The pellet samples may now be disposed of.