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Project / Draft		2021-11-03	**INFO**

Description

Please find enclosed the 1st working draft on WI 00223109, revision of EN 16087-1, Soil improvers and growing media - Determination of the aerobic biological degradation rate of organic matter - Part 1: Oxygen uptake rate (OUR) for your information.

During the last meeting of WG 4 on 2021-10-13, it was agreed that the 1st working draft on WI 00223109 is finalised. The enclosed 1st working draft as well as the forms for the activation of the work item is to be circulated to CEN/TC 223 as soon as the call for tender for the project leaders is completed and the project leaders are known.

CEN/TC 223

Date: 2021-10

prEN 16087-1:2021

Secretariat: NEN

Soil improvers and growing media — Determination of the aerobic biological degradation rate of organic matter — Part 1: Oxygen uptake rate (OUR)

Bodenverbesserungsmittel und Substrate — Bestimmung der aeroben biologischen Abbaurate organischer Substanz — Teil 1: Sauerstoffaufnahme (OUR)

Amendements organiques et support de culture — Détermination du taux de biodégradation aérobie de la matière organique — Partie 1: Cinétique d'absorption de l'oxygène (OUR)

ICS:

CCMC will prepare and attach the official title page.

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European foreword

This document (prEN 16087-1:2021) has been prepared by Technical Committee CEN/TC 223 "Soil improvers and growing media", the secretariat of which is held by NEN.

This document is a working document.

This document will supersede EN 16087-1:2011.

In comparison with the previous edition, the following technical modifications have been made:

- For the balance (5.6) requirements are added;
- Clarification of sample preparation (7.1) is added;
- Formula 3 and 5 are corrected;
- The figures in Annex B have been updated;
- The Bibliography has been corrected;
- The temperature of testing was for all products 30 °C but it was changed. Digestate is to be tested at 20 °C and compost and other materials are to be tested at 30 °C. By nature digestate has a much higher respiration compared to compost. In order to be able to use the same limit value, the test temperature for digestate has been lowered. For the interlaboratory trial both testing temperature, 20 °C and 30 °C are performed for all materials to make a comparison possible.

Kommentiert [FLK1]: May be reconsidered based on results of interlaboratory study.

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

SAFETY PRECAUTIONS — Care should be taken when handling substances of caustic nature or samples that may contain sharps or is of a dusty nature.

1 Scope

This document describes a method to determine the stability of materials (e.g. compost, digestate) used as components (ingredients) in growing media, soil improvers and organic fertilisers by measuring the oxygen uptake rate (OUR). The oxygen uptake rate is a direct parameter for the degradation rate of biodegradable organic matter that is being broken down within a specified time period. The method is not suitable for material with a content of particle sizes > 20 mm exceeding 20 % by volume.

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.

EN 13039:—[1], *Soil improvers and growing media - Determination of organic matter content and ash*

EN 13040-1:—[2], *Soil improvers and growing media - Sample preparation - Part 1: Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory bulk density*

EN 45501, *Metrological aspects of non-automatic weighing instruments*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TS 00223137:—[3] apply.

4 Principle

The material is suspended in water. The respiration rate (i.e. oxygen uptake rate) is indirectly determined by measuring the pressure drop in the headspace (i.e. gas phase in the closed space above the water phase). The produced CO2 (carbon dioxide) is removed by a suitable alkaline absorbent. The measurements are performed under defined conditions.

5 Apparatus

5.1 Testing facility

Temperature controlled room, climate cabinet or water bath, temperature adjustable to (20 ± 2) °C or (30 ± 2) °C, depending on the matrix. Digestate is to be tested at (20 ± 2) °C and compost and other materials are to be tested at (30 ± 2) °C.

Kommentiert [FLK2]: May be reconsidered based on results of interlaboratory study.

5.2 Pressure transducer

Operating range 0 kPa to 20 kPa (accuracy ± 0,1 kPa) and record for measuring 2 to 4 times per hour for seven days.

[1] Under preparation. Stage at the time of publication: EN 13039:20**.

[2] Under preparation. Stage at the time of publication: EN 13040-1:20**.

[3] Under preparation. Stage at the time of publication: prCEN/TS 00223137:20**.

5.3 CO_2-absorbent containing unit

5.4 Reaction vessel

1 000 ml to 2 500 ml with a CO_2-absorbent containing unit (see 5.3) and the pressure transducer (see 5.2) gastight connected (see Figure B.1).

5.5 Mixing device

Shaking table (120 ± 20) rpm or magnetic stirring unit and banded magnetic stirrer (see Figure B.2).

5.6 Balance

Meeting EN 45501 class II tolerances with a verification scale interval of 0,01 g.

5.7 pH meter

With slope adjustment and temperature control.

5.8 Dispenser

Dispensers or pipettes, adjustable units of 0,5 ml.

5.9 Glassware

Beakers and measuring cylinder.

5.10 Sieves

10 mm and 20 mm square aperture size.

5.11 Filter paper

Filter paper, with low ash content and high retentive properties.

6 Reagents

6.1 Water with a specific conductivity not higher than 0,2 mS/m at 25 °C

6.2 pH buffer

86 g/l KH_2PO_4, 89 g/l $Na_2HPO_4 \cdot 2H_2O$, mix ratio of 1:4 for pH 7; the solution is stable for 2 months if stored at 1 °C to 5 °C. When a particulate precipitation is found a fresh solution is to be made.

Commercially available buffers may be used as well.

6.3 Macronutrient solution

Dissolve the following masses of chemical compounds in 1 000 ml water (see 6.1): 4,3 g NH_4Cl,

5,4 g $CaCl_2 \cdot 2H_2O$, 4,3 g $MgSO_4 \cdot 7H_2O$

The solution is stable for 2 months if stored at 1 °C to 5 °C. When a particulate precipitation is found a fresh solution is to be made.

6.4 Micronutrient solution

Dissolve the following masses or volumes of chemical compounds in 1 000 ml water (see 6.1): 5,0 g EDDHA iron chelate (60 g Fe/kg), 1,4 g $MnSO_4$, 1,1 g $ZnSO_4$, 4,2 g $Na_2B_4O_7$, 0,2 g $CuSO_4$, 0,13 g Na_2MoO_4, 1 ml/l HCl (360 g/kg).

The solution is stable for 2 months if stored at 1 °C to 5 °C. When a particulate precipitation is found a fresh solution is to be made.

6.5 Complete nutrient solution

Add 1 ml of micronutrient solution (see 6.4) to 1 000 ml of macronutrient solution (see 6.3). The solution is stable for 2 months if stored at 1 °C to 5°C. When a particulate precipitation is found a fresh solution is to be made.

6.6 Nitrification inhibitor

4 g/l N-Allylthiourea, $C_4H_8N_2S$ (ATU).

Store the solution in a closed container at 1 °C to 5 °C. The solution is stable for not more than 2 weeks.

6.7 CO_2-absorbent

Such as NaOH-pellets, KOH-pellets or soda lime (mixture of $Ca(OH)_2$, NaOH, KOH and water), preferably with colour indicator.

NOTE Based on experience, 4,0 g of pellets (carbon dioxide absorption capacity > 200 g/kg) per container is sufficient.

6.8 NaOH (0,5 mol/l)

6.9 HCI (0,5 mol/l)

6.10 Inoculum (to be added for materials without or low microbial activity)

Prepare an extract by shaking an active compost with water at mass:volume, 1:10 and filter it over a paper filter (5.11).

Kommentiert [FLK3]: To be added: OUR level 25 to 30, respiration level 3

7 Procedure

7.1 Sample preparation for solid materials <10 mm.

Take minimally 1 l of material. Manually, but carefully, break up lumps and agglomerates only that have been caused by, for example, compression during transport. Break up lumps and agglomerates only and pass the sample through a 10 mm sieve (see 5.10), gently agitating the material if required. Particles > 10 mm shall not be broken up and shall be removed. Record the % mass of particles > 10 mm. If this amount is > 20 % of the total fresh mass the procedure for materials <20 mm (7.2) is to be followed. The moist sample shall be stored at 1 °C to 5 °C (max. 2 weeks).

7.2 Sample preparation for solid materials <20 mm.

Take minimally 1 l of material. Manually, but carefully, break up lumps and agglomerates only that have been caused by, for example, compression during transport. Break up lumps and agglomerates only and pass the sample through a 20 mm sieve (see 5.10), gently agitating the material if required. Particles > 20 mm shall not be broken up and shall be removed. Record the % mass of

particles > 20 mm. If this amount is > 20 % of the total fresh mass, the test is not applicable. The moist sample shall be stored at 1 °C to 5 °C (max. 2 weeks).

7.3 Sample preparation for liquid materials

Take minimally 1 l of material. Homogenise the sample by stirring. Break up lumps and agglomerates only and pass the sample through a 10 mm sieve (see 5.10), gently agitating the material if required. Particles > 10 mm shall not be broken up and shall be removed. Record the % mass of particles > 10 mm. If this amount is > 20 % of the total fresh mass, the test is not applicable. The sample shall be stored at 1 °C to 5 °C (max. 2 weeks). Stir the sample just before using it again.

7.4 Determination of moisture content and organic matter content

The moisture content shall be determined according to EN 13040-1:— and the organic matter content according to EN 13039:—, both determination should take place after the sample preparation.

7.5 Starting the procedure

For solid materials <10 mm and liquid materials (7.1 and 7.3), calculate the mass of fresh material (EOM) to be added to the reaction vessel based on 2 g of organic matter per litre according to Formula (1).

$$EOM(g) = \frac{20000}{W_{om} \times W_d} \tag{1}$$

where

W_{om} is the organic matter content, in % mass of the dried sample according to EN 13039:—;

W_d is the dry matter content, in % mass of the fresh sample according to EN 13040-1:—.

Calculate the required mass of sample (W_S) to perform the test according to Formula (2).

$$W_S(g) = EOM \cdot C_V \tag{2}$$

where

C_V is the capacity of the vessel in litres.

Place the calculated quantity of the sample in the clean reaction vessel (see 5.4). Add 180 ml water (see 6.1) and 10 ml complete nutrient solution (see 6.5) using a dispenser (see 5.8). Add 10 ml pH buffer (see 6.2) using a dispenser (see 5.8). Add 2,5 ml nitrification inhibitor (see 6.6) using a dispenser (see 5.8). Place the sample on the mixing device (see 5.5) and start the mixing for 4 h to 8 h in the conditioned room (see 5.1). Digestate is to be tested at 20 °C and compost and other materials are to be tested at 30 °C. Do not close the bottles.

The nitrification inhibitor is added to prevent the use of oxygen for nitrification processes.

For materials without or low microbial activity (e.g. wood fibre), 5 ml inoculum (6.10) can be added. Then, measure the pH of the suspension. The value should be between 6,5 and 7,5. If this is not the case, base or acid should be added (see 6.8 and 6.9).

For solid materials <20 mm (7.2) double the amount of mass of material and liquids (6.1, 6.2, 6.5 and 6.5) are to be taken and the vessel size should be 2 l.

The analyses shall be performed at least in duplicate.

At first instance, an equivalent of 2 g organic matter should be used for analysis. If it appears during the test that the pressure drop during the first three days is not higher than 2 kPa, then the amount of organic matter should be increased but with a maximum of 20 g dry matter however for materials with a very low organic matter content this can be more. If on the other hand the pressure drop during the first three days is more than 5 kPa, the amount of organic matter should be adjusted to 1 g. The amount of material tested will be 1 g to 5 g organic matter depending on the matrix.

NOTE If it appears there is not enough material available, the complete test can be repeated with a new sample.

NOTE In case of very homogeneous materials, a lower amount of organic matter can be taken, with the recommendation to run the test in 3 replicates.

7.6 Respiration measurement

Fill the CO_2-absorbent containing unit (see 5.3) with the absorbent (see 6.7). The pellets can be used several times. Before every use, they shall be inspected for colour changes. If the colour has changed, they shall be replaced. Replace the bottle top sensor and ensure a gas-tight fit.

NOTE To overcome oxygen depletion (see also 8.1) the reactor headspace can be purged with pure oxygen. The oxygen tubing is brought to just above the liquid surface (not in the liquid). The headspace must be purged for ± 15 sec. After purging, immediately mount the pressure measuring head with the closing piece on the reaction bottle and tighten it. Take the prescribed safety measures when using oxygen.

Start the shaking table (120 ± 20) rpm or magnetic stirrer (between 180 rpm and 450 rpm) and measure the pressure over seven days. Record the pressure 2 to 4 times per hour over seven days with the connected pressure transducer (see 5.2). The measurement ends, in principle, after seven days but can be ended if the pressure difference between the maximum and minimum value is more than 10 kPa.

NOTE If a magnetic stirrer is used and the amount of sand and gravel in the sample is high, it is important to check that the stirrer is not in contact with the base.

8 Calculations

8.1 Theoretical background

The pressure drop as a function of time in principle looks like Figure B.3. During the first period (0 h to 8 h), the pressure rises by the rising pressure of water vapour in the headspace. Thereafter is a short period (0 h to 12 h) in which the pressure is more or less stable. This is the growing phase of microorganisms and is dependent on the number of active microorganisms initially present. In the third period, the pressure drops linearly and in the fourth phase the pressure drop decreases to become constant. In the final phase, the oxygen runs out.

From the pressure drop in the third period the respiration rate is determined (see Figure B.3). The pressure drop shall not be more than 10 kPa (this is equivalent to a decrease of the oxygen content from 20% to 10% in the headspace), because at a higher pressure drop the oxygen supply to the water can be limiting (see Veeken [2]).

NOTE A sudden and increasing rate of the pressure drop is an indication of nitrification. This can be confirmed by measuring nitrate in the solution. In this case the OUR cannot be determined as nitrification also consumes oxygen. Check the date of the prepared ATU solutions, it should not be older than 2 weeks.

8.2 Calculations

The oxygen consumption (*Oc*, in mmol O_2 per kg *OM*) is calculated from the pressure drop (*ΔP*) in the headspace according to Formula (3):

$$Oc = \frac{\Delta P}{R\left(273,15 + T\right)} \times \frac{V_{gas} \cdot 10000}{W \cdot DM \cdot OM} \tag{3}$$

where

Oc	is the oxygen consumption, in mmol O_2 per kg OM;
ΔP	is the pressure drop in the headspace, in kPa;
R	is the gas constant (8,314 $\mathrm{l \cdot kPa \cdot K^{-1} \cdot mol^{-1}}$);
T	is the temperature the measurement is performed, in °C;
W	is the initial mass of the sample, in kg;
DM	is the dry matter content of the sample, in % mass;
OM	is the organic matter content of the sample, in % DM mass;
V_{gas}	is the volume of the gas phase, in ml.

$$V_{gas} = V_{vessel} - \frac{W \times DM \times 10000}{\rho} - \frac{W \times Wm \times 10000}{1000\,*} - V_{liquid} \tag{4}$$

* - taken as water density ($\mathrm{kg \cdot m^{-3}}$)

where

V_{vessel}	is the total volume of vessel, in ml;
V_{liquid}	all added liquids (water, nutrient solution, pH buffer and ATU solution), in ml;
W_m	is the moisture content, in % mass of the fresh sample according to EN 13040-1:—;
p	is the calculated sample density, in $\mathrm{kg \cdot m^{-3}}$.

$$\rho = \frac{1}{\frac{OM\,/\,100}{1550} + \frac{(100 - OM)\,/\,100}{2650}} \tag{5}$$

The sample density is calculated from OM, W and DM and should not be confused with the laboratory compacted bulk density.

The oxygen uptake rate (*OUR*, mmol $O_2 \cdot \mathrm{kg^{-1}} \cdot \mathrm{OM} \cdot \mathrm{hour^{-1}}$) is calculated from *Oc* and the related time period according to Formula (6).

$$OUR = \frac{Oc}{\Delta t} \tag{6}$$

where

Δt	time *ΔP* is taken, in h.

9 Test report

The test report shall contain at least the following:

a) a reference to this document;

b) all data required for a complete identification of the sample;

c) the number of replicates;

d) the mean *OUR*, rounded to one decimal place and the testing temperature;

e) details of all work cycles not contained in this standard or that were considered optional, as well as all factors that may have influenced the results.

Annex A
(informative)

Validation

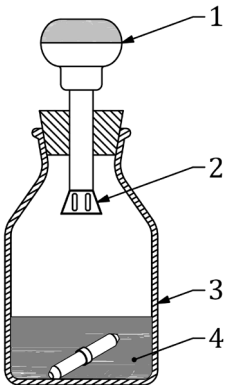
Samples:

- Liquid digestate
- Dried digestate
- Compost: 2x (stable and less stable)
- Coir
- Blend of growing medium

In the interlaboratory trial both 20 °C and 30 °C are to be performed for all materials. The self-heating test (compost) and residual biogas production (digestate) are to be performed parallel on the samples in the interlaboratory trial.

Annex B
(informative)

Specific information on the OUR-test



Key

1 pressure transducer

2 CO_2-absorbent

3 reaction vessel

4 sample suspension, optionally with banded magnetic stirrer (see Figure B.2)

Figure B.1 — Reaction vessel (*OUR*)

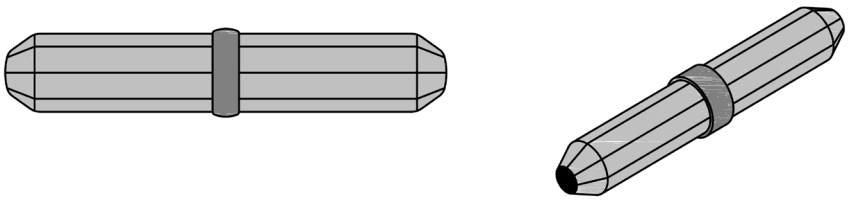
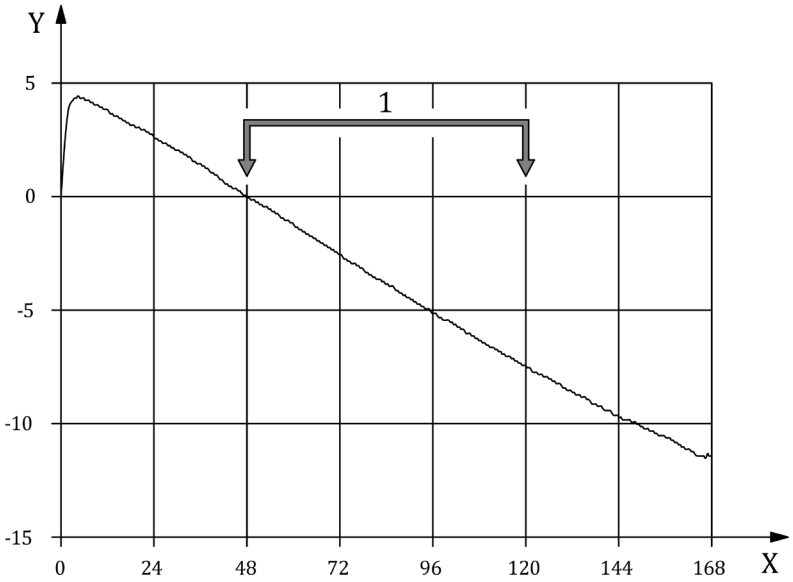


Figure B.2 — Banded magnetic stirrer



NOTE This is an example of a possible curve. Many variations are possible.

Key

1 3rd period, relevant for the measurement

X time (h)

Y pressure drop (kPa)

Figure B.3 — Typical time pressure relation during the OUR-test

Bibliography

[1] CEN/TS 00223137:—, *Soil improvers and growing media — Terminology*

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