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Final report

Influence of the incubation temperature on the biological activity of soil improvers using the respirometric method

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SAMENVATTING

In het Vlaamse Gewest van België (of Vlaanderen) is de bepaling van de rijpheidsgraad voor compost (sinds 1998) en de respirometrische test (sinds 2006 voor compost en sinds 2008 voor digestaat producten) reeds verschillende jaren geïmplementeerd om de aërobe biologische acitiviteit te bepalen.

De procedure voor de bepaling van de rijpheidsgraad is in overeenstemming met de Europese standaard EN 16087-2 *Soil improvers and growing media – Determination of the aerobic biological activity –Part 2: Self heating test for compost*, welke was ontwikkeld in 2011 binnen CEN/TC 223 *Soil improvers and growing media*.
Voor de bepaling van de respirometrische waarde verschilt de toegepaste procedure van de Europese standaard, ontwikkeld in 2011 binnen CEN/TC 223 *Soil improvers and growing media*. Deze standaard EN 16087-1 *Soil improvers and growing media – Determination of the aerobic biological activity – Part 1: Oxygen uptake rate (OUR)*[1] beschrijft een vergelijkbare methode als de in Vlaanderen toegepaste compendium methode. Het belangrijkste verschil tussen beide methoden is de gebruikte incubatietemperatuur. In de EN methode 16087-1 wordt de test uitgevoerd bij een temperatuur van 30°C, terwijl bij 20°C in de compendium methode toegepast in Vlaanderen.

Alle Europese standaardmethoden gerelateerd aan zuurstofverbruik / biologische activiteit (EN ISO 9408, EN 1899, EN ISO 17556, EN ISO 10707, EN 16087-2) hebben een incubatietemperatuur van ca. 20°C, met uitzondering van EN 16087-1 welke een temperatuur van 30 ± 2°C voorschrijft. Bijgevolg werd in 2008 in Vlaanderen (België) gekozen voor een (geharmoniseerde) temperatuur van 20°C als referentietemperatuur bij alle procedures met betrekking tot zuurstofverbruik. Vooral, sinds de BZV bepaling bij 20°C een internationaal aanvaarde standaard methode is, zijn in de meeste laboratoria incubatoren en incubatiekamers van 20°C aanwezig. Bovendien houden air conditioning systemen voor laboratoria de thermostaat in het algemeen bij een kamertemperatuur van ongeveer 20°C.

De incubatietemperatuur heeft echter een grote invloed op de resultaten van de stabiliteitstest. In deze studie werd aangetoond dat de matrix (compost of digestaat) een invloed heeft op de grootte van het verschil. Verschillende ratio's van het zuurstofverbruik bij 30°C t.o.v. 20°C worden bekomen voor compost (gemiddelde verhouding ~ 2,5) en voor digestaat (gemiddelde verhouding ~ 1,6).

In verschillende voorstellen van EU kwaliteitscriteria (End of Waste criteria voor biologisch afbreekbaar afval, EU Ecolabel criteria voor bodemverbeteraars en groeimedia) ontbreekt een goede correlatie tussen de respirometrische waarde en de rijpheidsgraad (Rottegrad). Bovendien wordt als voorstel in de nieuwe EU Meststoffenverordening EU een limietwaarde voor de respirometrische index gedefinieerd van 25 mmol/kg organische stof/h. In de praktijk, en rekening houdend met de historische dataset voor de respirometrische methode, is deze waarde niet haalbaar voor de meeste digestaat producten wanneer uitgevoerd bij 30°C.

Op basis van een uitgebreide historische dataset voor compostmonsters, welke bemonsterd en geanalyseerd zijn in Vlaanderen (België), kon een goede correlatie afgeleid worden tussen de respirometrische waarde (bepaald bij 20 ± 2°C) en de rijpheidsgraad (Rottegrad, bepaald bij 22 ± 2°C) en deze is hieronder weergegeven.

Typering	Zuurstofverbruik (bij 20°C)	Rijpheidsgraad T_{max} (°C)	Rijpheidsgraad Klasse
Heel stabiel, weinig actief	< 5 mmol O_2/kg OS/u	≤ 30	V
Stabiel, beperkt actief	5 - 10 mmol O_2/kg OS/u	30.1 –40.0	IV
Matig stabiel, actief	10 - 15 mmol O_2/kg OS/u	40.1 – 50.0	III
Relatief jong, vrij sterk actief	15 - 25 mmol O_2/kg OS/u	50.1 – 60.0	II
Jong, heel actief, onstabiel	> 25 mmol O_2/kg OS/u	> 60	I

Aanbeveling:
Een geaccrediteerd overheidscontrolesysteem voor de rijpheidsgraad (22 ± 2°C) en de respirometrische test (20 ± 2°C) voor de matrices compost en digestaat is reeds meer dan 10 jaar in uitvoering in Vlaanderen (België). Op basis van de verworven kennis enerzijds en de behoefte aan afstemming met de Europese normmethode anderzijds, strekt het tot aanbeveling om een incubatietemperatuur van 20°C op te nemen in de Europese standaard EN 16087-1. Het literatuuronderzoek, de verzamelde gegevens, de vergelijkende studie en de bespreking in dit verslag beschrijft de reden voor dit verzoek.

SUMMARY

In the Flemish part of Belgium, the self-heating test for compost (since 1998) and the respirometric test (since 2006 for compost and since 2008 for digestate products) has been implemented for many years to determine the aerobic biological acitivity.

The procedure applied for the **self-heating** is in accordance with the European standard EN 16087-2 *Soil improvers and growing media – Determination of the aerobic biological activity –Part 2: Self heating test for compost*, which was developed in 2011 within CEN/TC 223 *Soil improvers and growing media*.
For the determination of the **respirometric value** the applied procedure differs with the European standard, developed in 2011 within CEN/TC 223 *Soil improvers and growing media*. The standard EN 16087-1 *Soil improvers and growing media – Determination of the aerobic biological activity – Part 1: Oxygen uptake rate (OUR)*[2] describes a similar method as the compendium method applied in Flanders. The main difference between both procedures is the applied incubation temperature. In the EN method 16087-1 a temperature of 30°C is applied, while in the compendium procedure used in Flanders a temperature of 20°C is applied.

All European standard methods related to oxygen demand/biological activity (EN ISO 9408, EN 1899, EN ISO 17556, EN ISO 10707, EN 16087-2) include an incubation temperature of ca. 20°C, except EN 16087-1 which prescribes a temperature of 30 ± 2°C. Therefore, in 2008, in the Flemish region of Belgium, an (harmonized) temperature of 20°C has been chosen as reference temperature for procedures involving oxygen demand. Especially, since the BOD determination at 20°C is a widely internationally adopted standard method, incubators and incubations rooms at 20°C are available in most laboratories. Besides, air conditioning systems for laboratories keep the thermostat setting generally at a roomtemperature of about 20°C.

The incubation temperature has a large effect on the outcome of the stability test. In the present study, it was demonstrated that the matrix (compost or digestate) affects the magnitude of the difference. Different ratio factors were calculated between the oxygen uptake rate result at 30°C and at 20°C for compost (average ratio ~2.5) and digestate (average ratio ~ 1.6).

In different EU quality criteria proposals (End of Waste criteria for biodegradable waste, EU Ecolabel criteria for soil improvers and growing media) a sound correlation between the respirometric value and the self-heating test (Rottegrad) is apparently lacking. Moreover, as proposal in the new EU Fertiliser Regulation, the limit value for the respirometric index is defined at 25 mmol/kg organic matter/h. In practice, and taking into account the historical dataset for the respirometric test, this value is not achievable for the majority of the digestate products when performed at 30°C.

Based on an extended historical dataset of compost samples, collected and analysed in Flanders (Belgium), a correlation between the respirometric value (determined at 20 ± 2 °C) and the self heating test (Rottegrad, determined at 22 ± 2 °C) could be derived and is presented below.

Description condition	Oxygen uptake rate (OUR) (at 20°C)	Self heating test T_{max} (°C)	Self heating test Class
Very stable, low activity	< 5 mmol O_2/kg OM/h	≤ 30	V
Stable, limited activity	5 - 10 mmol O_2/kg OM/h	30.1 –40.0	IV
Moderately stable, active	10 - 15 mmol O_2/kg OM/h	40.1 – 50.0	III
Relative fresh, high activity	15 - 25 mmol O_2/kg OM/h	50.1 – 60.0	II
Fresh, very high activity, unstable	> 25 mmol O_2/kg OM/h	> 60	I

Recommendation :

An accredited governmental control system for self-heating test (22 ± 2 °C) and respirometric test (20 ± 2°C) for the matrices compost and digestate is in place for more than 10 years in the Flemish region of Belgium. Based on the acquired expertise on the one hand and the need for harmonisation with the EN standard on the other hand, the inclusion of an incubation temperature of 20°C in the European Standard EN 16087-1 should be taken into consideration. The literature review, the compiled data, the comparative study and the discussion given in this report describe the rationale for this request.

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CHAPTER 1 INTRODUCTION

In Flanders (Belgium) the self-heating test for compost is already used for many years to determine the aerobic biological acitivity. It involves a measurement of self-heating in a Dewar vessel and measurement of the maximum temperature, where the temperature is an indicator of the state of aerobic biological activity. The results allow to divide the compost in different classes depending on their biological activity, as shown in Table 1. The applied method in Flanders is described in the compendium method CMA/2/IV/22[4] and is already online available since 1998. The described procedure is in accordance with the European standard EN 16087-2 *Soil improvers and growing media – Determination of the aerobic biological activity –Part 2: Self heating test for compost*, which was developed in 2011 within CEN/TC 223 *Soil improvers and growing media*.

Table 1 Classification as a function of T_{max}

T_{max} (°C)	Class
> 60	I
50.1 – 60.0	II
40.1 – 50.0	III
30.1 – 40.0	IV
≤ 30	V

Note: T_{max} = maximum test temperature (sum of ambient temperature and maximum rise of temperature during test).

Additional to the self-heating test, there was a need to monitor the biological stability of other types of materials. For that purpose, the respirometric method was evaluated and a compendium method was developed in 2007 based on a research project performed by Organic Waste Systems (OWS, Ghent) and validated by a round robin test with the recognised laboratories in Flanders (Belgium)[3]. The compendium method CMA/2/IV/25[4] *Stabiliteit met gesloten respirometer* describes the use of an OxiTop® measuring system for the determination of the respiration rate and N-mineralisation rate of organic matter in soil improvers (compost,…) and was derived from the method described by Veeken *et al*.[5] The respirometric method, as described in CMA/2/IV/25, is performed at a temperature of 20°C and has been implemented in the Flemish recognised laboratories since 2008.

In 2011 an European standard was developed within CEN/TC 223 *Soil improvers and growing media*. The standard EN 16087-1 *Soil improvers and growing media – Determination of the aerobic biological activity – Part 1: Oxygen uptake rate (OUR)*[6] describes a similar method as the CMA method. The main difference between both procedures is the applied incubation temperature. In the EN method 16087-1 a temperature of 30°C is applied, while the CMA method applies a temperature of 20°C. The relationship between biological activity and temperature has a so-called Q10 factor of about 2 which means that the degradation at 30°C proceeds twice as fast as at 20°C. This means that the results obtained at 20°C or at 30°C will result in different measurement values.

In this study, it was examinated whether an increase of the incubation temperature with 10°C results in an effective increase of the oxygen uptake rate with a factor of 2. The oxygen uptake rate (OUR) is expressed as mmol/kg OM/h (of which OM = organic matter). For that purpose, VLACO collected 50 samples (different matrix compositions, i.e. compost and digestates). Of each sample the biological activity was measured at a temperature of 20°C (CMA method), as well as at a temperature of 30°C. All analyses were performed in duplicate and conducted by an accreditated laboratory (OWS). Moreover, on these samples the standard parameters such as moisture content, pH, volatile solids,… were determined. The obtained analytical results were delivered to VITO, who performed the evaluation of the obtained data.

In Flanders (Belgium) a significant amount of data from compost samples and digestates are available of both the self-heating test and the OUR test (performed at 20°C). The obtained results need to meet dedicated quality criteria, as prescribed by VLACO, for both parameters. With the comparative tests of the OUR at 20°C and 30°C, performed in this study, we were able to indicate the correlation between both methods. Moreover, an evaluation was performed of the feasibility of the End of Waste criteria for biodegradable waste and the EU Ecolabel Criteria for soil improvers and growing media, as defined for the self heating test and for the OUR determined at 30°C for both compost and digestates.

CHAPTER 2 LEGAL STANDARDS AND QUALITY OBJECTIVES

2.1. STANDARD VALUES AND QUALITY OBJECTIVES – FLANDERS, BELGIUM

2.1.1. INTRODUCTION

For the various types of end products produced by biological treatment processes legal standard values and quality objectives are described.

At first there are legal chemical composition requirements for secondary raw materials (fertilizers or soil improvers) as described in VLAREMA[a] Appendix 2.3.1.A. There are also terms of use for maximum allowable contaminant dosage to the soil that must be respected, as described in VLAREMA Appendix 2.3.1.C. Both conditions are for each product (secondary raw material) applicable. With respect to the parameter oxygen uptake rate (Oxitop ®) no legal requirements are defined in VLAREMA.

In addition, specific standards (reference values) and quality objectives are defined by VLACO in order to obtain a VLACO-label, which are more stringent than the global conditions described in VLAREMA, but which allowed a broader application range of the end products. These reference values, used to assess the quality of the end products, are applicable to the individual results, while the quality objectives are target values which has to be fulfilled "on average", and for which a series of analytical results is assessed. With respect to the parameter oxygen uptake rate (OxiTop®), both reference values and quality objectives are defined as a function of the matrix type. In paragraph 2.1.2 an overview is presented.

As part of the fertilizer legislation (Relevant Authority: Federal Public Service (FPS) - Health, Food Chain Safety and Environment) also product standards are imposed for trading. Compost and digestate are not included in the list of authorized products, therefore one have to apply for an exemption to the FPS Health, Food Chain Safety and Environment. The FPS decides on the trade applications of compost and digestate, and they may impost additional conditions. With respect to the parameter oxygen uptake rate (OxiTop®) no products standards are included in the list of authorizes products, but the stability of these products (oxygen uptake rate (OxiTop®) or self-heating test) is evaluated based on the reference values/quality objectives as defined by VLACO.

2.1.2. STANDARD VALUES AND QUALITY OBJECTIVES – FLANDERS, BELGIUM

In the period 2003-2006 a study was performed by Organic Waste Systems (OWS, Ghent) in commission of VLACO to evaluate the OUR (OxiTop®) method in comparison with the self heating test. From the obtained data reference values and quality objectives are derived, as presented in Table 2. A further classification is described in Table 3.

[a] VLAREMA: Besluit van de Vlaamse Regering tot vaststelling van het Vlaams reglement betreffende het duurzaam beheer van materiaalkringlopen en afvalstoffen – https://navigator.emis.vito.be/mijn-navigator?woId=43991

Table 2 Reference values and quality objectives

Matrix	Parameter	VLACO Quality objective	VLACO Reference value	FPS* Product standard
Vfg-compost** and compost after digestion with post composting	OUR (OxiTop®) (mmol O_2/kg OM/h)	10	15	-
	Self heating test - T_{max}***	< 40 °C (IV)	< 45 °C	< 40 °C (IV)
Green compost	OUR (OxiTop®) (mmol O_2/kg OM/h)	10	15	-
	Self heating test - T_{max}	< 30 °C (V)	< 40 °C (IV)	< 40 °C (IV)
Digestate	OUR (OxiTop®) (mmol O_2/kg OM/h)	-	50	-
	Self heating test (°C)	-	-	-

*FPS: Federal Public Service;
**Vfg-compost: vegetables-fruit and garden compost;
*** T_{max} : maximum test temperature (sum of ambient temperature and maximum rise of temperature during test). Between brackets: classification.

Table 3 Classification for the stability of compost

Oxygen uptake rate (OUR)	Description condition
< 5 mmol O_2/kg OM/h	Very stable, low activity
5 - 10 mmol O_2/kg OM/h	Stable, limited activity
10 - 15 mmol O_2/kg OM/h	Moderately stable, active
15 - 25 mmol O_2/kg OM/h	Relative fresh, high activity
> 25 mmol O_2/kg OM/h	Fresh, very high activity, unstable

These quality objectives and reference values are derived based on OUR results obtained at an incubation temperature of 20°C.

On a yearly base VITO organises round robin tests within Flanders to evaluate the performance of the recognised laboratories. Since 2008 also compost samples are distributed to measure the parameter OUR. Between 2008 and 2011 on the same compost sample (young green compost) as wel as the self heating test (T_{max}) as the oxygen uptake rate (OUR) was determined and, therefore, data of both measurements methods are available on the same sample. The obtained results are shown in Table 4.

Taken into account the proposed reference values and quality objectives between both analytical methods corresponds well with the obtained results in the round robin test. For green compost the quality objective of OUR amounts 10 mmol O_2/kg OM/h and for the self heating test T_{max} is < 30°C, while for the reference value the OUR amounts 15 mmol O_2/kg OM/h and for the self heating test T_{max} is < 40°C.

Additionally the round robin results of 2012-2014 are presented in Table 4, but then the self-heating test and the oxygen utake rate was determined on a different sample.

Table 4 Results of the round robin test for the parameters T_{max} and OUR

Year	Sample	Parameter	Incubation T°	average value	%RSD	N
2008	Sample 1	Tmax (°C)	22 ± 2°C	39.5°C (IV)	20%	8
	Sample 1	OUR (mmol/kg OM/h)	20 ± 2°C	15.2	23%	8
2009	Sample 2	Tmax (°C)	22 ± 2°C	27.2°C (V)	12%	7
	Sample 2	OUR (mmol/kg OM/h)	20 ± 2°C	10.2	32%	8
2010	Sample 3	Tmax (°C)	22 ± 2°C	40.6°C (III)	9%	6
	Sample 3	OUR (mmol/kg OM/h)	20 ± 2°C	16.0	28%	5
2011	Sample 4	Tmax (°C)	22 ± 2°C	27.2°C (V)	9%	8
	Sample 4	OUR (mmol/kg OM/h)	20 ± 2°C	9.48	8%	6

Year	Sample	Parameter	Incubation T°	average value	%RSD	N
2012	Sample 5	Tmax (°C)	22 ± 2°C	27.1°C (V)	13%	7
	Sample 6	OUR (mmol/kg OM/h)	20 ± 2°C	8.91	23%	5
2013	Sample 7	Tmax (°C)	22 ± 2°C	52.3°C (II)	11%	5
	Sample 8	OUR (mmol/kg OM/h)	20 ± 2°C	12.9	8.6%	5
2014	Sample 9	Tmax (°C)	22 ± 2°C	32.6°C (IV)	12%	5
	Sample 10	OUR (mmol/kg OM/h)	20 ± 2°C	38.8	17%	5

Samples 1 -4, 6, 8, 10: young green compost
Samples 5, 7, 9: Vfg-compost: vegetables-fruit and garden compost

2.2. STANDARD VALUES AND QUALITY OBJECTIVES – EUROPE

For testing the stability of soil improvers, two EN standard methods are available:

- EN 16087-1 Soil improvers and growing media - Determination of the aerobic biological activity - Part 1: Oxygen uptake rate (OUR)
- EN 16087-2 Soil improvers and growing media - Determination of Aerobic biological activity - Part 2: Self heating test for compost

Note that for EN 16087-1 an incubation temperature of 30 ± 2°C is prescribed, while for EN 16087-2 an incubation temperature of 22 ± 2°C is prescribed.

In the report[7], describing the End of waste criteria for biodegradable waste, product quality requirements for compost and digestate are included. Product quality criteria are needed to check:

1) for elements that can result in direct environmental and health risks, and
2) that the product is suitable for direct use (on land, for production of growing media, etc).

With respect to the mimimum stability of compost and digestate the following criteria and comments are formulated:

- **Compost**: Unless an eligible alternative method has been specified by the competent authorities, the producer must demonstrate to meet at least one of the following two stability criteria for compost:
  - Respirometric index of maximum 25 mmol O_2/kg organic matter/h, measured according to standard EN 16087-1.

- Minimum Rottegrad III, IV or V (self-heating test temperature rise of maximum 30 °C above ambient temperature), measured according to standard EN 16087-2.

If a Member State already has an official method in place that differs from the two methods above, together with an associated limit value, the Member State competent authorities may complement or replace the two methods described above with its existing method and associated limit value as an eligible alternative.

- **Digestate**: Unless an eligible alternative method has been specified by the competent authorities, the producer must demonstrate to meet at least one of the following three stability criteria for digestate:
  - Respirometric index of maximum 50 mmol O_2/kg organic matter/h, measured according to standard EN 16087-1.
  - Organic acids content of maximum 1500 mg/l
  - Residual biogas potential of maximum 0.25 l/ g volatile solids.

  As an eligible alternative, the competent authorities of a Member State may complement or replace the three methods described above with another method and associated limit value providing equivalent stability guarantees.

With respect to the minimum stability of compost and digestate the following comments are formulated:

- A minimum stability should avoid unwanted emissions during transport and storage and prevent materials from entering the market without proper treatment.
- Materials being produced in one Member State and used or put on the market in a different Member State shall meet the requirements of both Member States for the stability criterion unless the receiving Member State recognizes the method of the producing Member State.

At European level also a technical report was written related to '*Revision of the EU Ecolabel Criteria for Soil Improvers and Growing Media - Technical report and draft criteria proposal*'[8]. In Annex B the full text related to the Stability criterion is taken over. Below the most important features are highlighted.

Abstract of the report: This technical report is aimed at providing a sound base to the revision process of the EU Ecolabel criteria for Soil improvers and Growing Media. Currently, separate sets of EU Ecolabel criteria exist for Soil improvers (Decision 2006/799/EC) and Growing media (Decision 2007/64/EC). The revision process spanned both product groups; thus common criteria for both Soil improvers and Growing media are developed, only distinguishing between technical product characteristics where necessary. Another objective of this revision is addressing the possibility to broaden the current scope to the product mulch, as it has been identified as a differentiated sub-product within the product group 'soil improvers'. To achieve those objectives, a Preliminary Report was produced together with the first version of this technical report as a working document. Hence, this document was developed to undergo the stakeholder consultation, which is crucial to come up with criteria adapted to the market reality while being able to select the best environmental performance products available on the market. After an extensive stakeholder's consultation process, this current version of the Technical Report supporting the draft criteria for growing media, soil improvers and mulch, constitutes the support of the criteria set voted positive by the Regulatory Committee.

The proposed criteria for stability applies to growing media, soil improvers and mulch, with the exception of mulch totally composed of lignocellulosic constituents and mineral growing media.

Soil improvers and mulch for non-professional applications and growing media for all applications, shall meet one of the following requirements:

Stability parameter	Requirement
Maximum Respirometric index	15 mmol O_2/kg organic matter/h
Minimum Rottegrad, where applicable	IV (self-heating test temperature rise of maximum 20°C above ambient temperature)

Soil improvers and mulch for professional applications shall meet one of the following requirements:

Stability parameter	Requirement
Maximum Respirometric index	25 mmol O_2/kg organic matter/h
Minimum Rottegrad, where applicable	III (self-heating test temperature rise of maximum 30°C above ambient temperature)

The applicant shall provide the test reports conducted in accordance with the following testing procedures:

- EN 16087-1 Soil improvers and growing media - Determination of the aerobic biological activity - Part 1: Oxygen uptake rate (OUR)
- EN 16087-2 Soil improvers and growing media - Determination of Aerobic biological activity - Part 2: Self heating test for compost

Note that for EN 16087-1 an incubation temperature of 30 ± 2°C is prescribed, while for EN 16087-2 an incubation temperature of 22 ± 2°C is prescribed.

The criteria for the self heating test are derivered from Brinton *et al.* (1995) [9], who described that the compost stability can be classified based on their Rottegrad as shown in the table below:

Temperature rise above ambient (°C)	Official class of stability	Descriptors of class or group	Major group
< 10	V	Very stable, well-aged compost	Finished compost
10 - 20	IV	Moderately stable, curing compost	
20 - 30	III	Still decomposing, active compost	Active compost
30 - 40	II	Immature, young or very active compost	
> 40	I	Fresh, raw compost, just mixed ingredients	Fresh compost

Note that Brinton *et al.* describes that the method in the laboratory is carried out at ambient conditions and that it may be utilized under field conditions where a relatively stable room temperature of 20 – 25°C (but no more than 25°C) can be maintained around the vessel.

Brinton *et al.* (1995) also proposes end use categories derived from experience with composted source-separated residential food residues blended with yard-waste, and active compost (Rottegrad III) is applicable to fruity crops, while for general purpose gardening, Rottegrad IV compost is recommended.

The criteria for the oxygen uptake rate are derived from Veeken *et al.* (2003)[5], who proposed the following scheme for classification of biowaste and green waste compost based on specific oxygen uptake rate together with equivalent CO_2 production values.

Category of compost product	Oxygen uptake rate (mmol O_2/kg OM/h)	Oxygen uptake rate (mg O_2/kg OM/h)	Equivalent CO_2 evolution rate (mg CO_2/kg OM/day)
Very unstable	> 30	> 960	> 32
Unstable	15 – 30	480 – 960	16 – 32
Stable	5 – 15	160 – 480	5 – 16
Very stable	< 5	< 160	< 5

Note that the method applied by Veeken *et al.* was performed at 30°C.

2.3. INCUBATION TEMPERATURE IN EUROPEAN STANDARDS RELATED TO OXYGEN DEMAND

In the following paragraph an overview of European/International standards is given in relation to incubation temperatures for the determination of oxygen demand:

1. EN ISO 9408 1999 Water quality - Evaluation of ultimate aerobic biodegradability of organic compounds in aqueous medium by determination of oxygen demand in a closed respirometer (ISO 9408:1999)
   Incubation shall take place in the dark or in diffused light, at a temperature within the range **20°C to 25°C** which shall not vary by more than ± 1 °C during the test.

2. EN 1899-1 1998 Water quality - Determination of biochemical oxygen demand after n days (BODn) - Part 1: Dilution and seeding method with allylthiourea addition (ISO 5815:1989, modified)
   Incubator, capable of being maintained at (**20 ± 2**) °C.

3. EN ISO 17556 2012 Plastics - Determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved (ISO 17556:2012)
   Incubation shall take place in the dark or in diffused light in an enclosure which is free from vapours toxic to microorganisms and is maintained at a temperature constant to within ± 2 °C in the range between **20°C and 28°C**, preferably 25°C

4. EN ISO 10707 1998 Water quality - Evaluation in an aqueous medium of the "ultimate" aerobic biodegradability of organic compounds - Method by analysis of biochemical oxygen demand (closed bottle test) (ISO 10707:1994)
   Incubation shall take place in the dark in an anclosure which is maintained a constant temperature within ± 1 °C between **20°C and 25°C**.

For the determination of the self heating test according to EN 16087-2 (Soil improvers and growing media - Determination of the aerobic biological activity - Part 2: Self heating test for compost), the room temperature shall be maintained at (**22 ± 2**)°C for the entire duration of the test.

Therefore, in the Flanders region of Belgium, an (harmonized) temperature of 20°C has been chosen as reference temperature for procedures involving oxygen demand. Especially, since the BOD determination at 20°C is a widely internationally adopted standard method, incubators and incubations rooms at 20°C are available in most laboratories. Besides, air conditioning systems for laboratories keep the thermostat setting generally at a roomtemperature of about 20°C.

CHAPTER 3 ANALYTICAL RESULTS – OUR AT 20°C AND AT 30°C

3.1. DESCRIPTION OF THE SAMPLES

In the period between March and September 2016, 50 samples were collected by VLACO and delivered at the accreditated laboratory (OWS, Ghent). The sample composition was as follows:

- Compost
  - Green compost – 8 samples
  - Vegetable, fruit and garden waste compost (vfg) – 12 samples
- Digestate
  - Liquid fraction – 6 samples
  - Solid fraction – 8 samples
  - Dried digestate – 6 samples
  - Raw digestate – 7 samples
- Organic biological waste - manure (OBA-manure) – 3 samples

All samples were analysed in duplicate to determine the oxygen uptake rate (OUR) at both temperatures (20°C and 30°C). Additionally, the standard parameters (moisture content, pH, volatile solids) were determined on these samples. All relevant data of the individual samples are presented in Annex A.

3.2. OXYGEN UPTAKE RATE (OUR) RESULTS

3.2.1. RELATION BETWEEN THE OXYGEN UPTAKE RATE AT 20°C AND AT 30°C (ALL DATA INCLUDED)

For each sample the OUR was determined at 20°C and at 30°C, always in duplicate. In Figure 1 the relation is shown between the results obtained at an incubation temperature of 20°C and at 30°C. Always the average value of the duplicate results was taken into account.

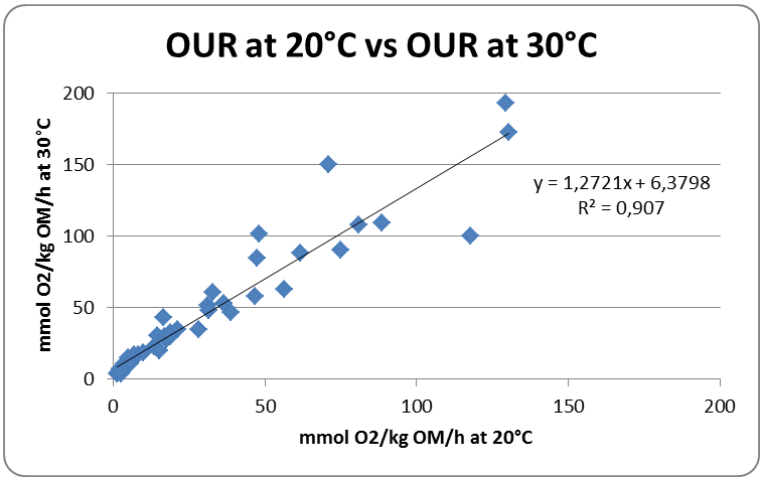

Figure 1 Relation between the oxygen uptake rate (OUR) at 20°C and at 30°C for all data

Alternatively, for each individual sample the ratio factor was calculated between the OUR result at 30°C and at 20°C (Ratio = $OUR_{30°C}/OUR_{20°C}$). Always the average value of the duplicate results was taken into account. The average of all factors (n=50) amounted 1.96, the median 1.85, minimum 0.85 and maximum 3.11. As shown from these results, the calculated factor varies between a broad range (from 0.85 up to 3.11). The obtained data also show that the calculated ratio factor is matrix dependent. A further evaluation of this factor per matrix type is performed in paragraph 3.2.3.

3.2.2. EVALUATION OF THE DUPLO MEASUREMENTS

All samples were analysed in duplicate, for both incubation temperatures. The obtained coefficient of variation (CV_R) amounts 10% for the measurements at 20°C, and 6.9% for the measurements at 30°C. The observed difference is limited and can mainly be attributed to the higher concentration range for the samples measured at 30°C. In general, it can be stated that the obtained CV_R values are within acceptable ranges for these type of analysis.

When evaluating the CV_R by matrixtype (Table 5), slight differences are observed, but these can be mainly assigned to normal measurement uncertainties.

Table 5 Calculation of the CV_R of the duplo samples as a function of matrix type

	N	CV_R (%) at 20°C	CV_R (%) at 30°C
ALL DATA	50	10.0	6.9
Compost-vfg	8	13.6	10.9
Compost-green	12	7.7	2.5
Digestate-solid fraction	8	14.7	5.4
Digestate-liquid fraction	6	5.3	9.7
Digestate - dried	6	4.6	6.2
Digestaat - raw	7	8.7	5.4
OBA-manure	3	10.0	5.6

3.2.3. RELATION BETWEEN THE OXYGEN UPTAKE RATE AT 20°C AND AT 30°C (AS A FUNCTION OF MATRIX TYPE)

In Table 6 the ratio factor is calculated between the OUR result at 30°C and at 20°C (Ratio = $OUR_{30°C}/OUR_{20°C}$). Always the average value of the duplicate results was taken into account. For all sample types, the average value, the median, the minimum and the maximum value is presented. In Figure 2 the obtained factors are graphically presented in box plots.

The obtained data clearly show that there is a significant difference in factor depending on the type of matrix. For all data the average factor is 1.96, which approaches the factor of 2 (which is generally considered as the applicable factor). But when looking into more detail, there is a differentiation between the different matrices.
For the compost samples, there is no significant difference in factor between the green compost and the vfg-compost. An average factor of 2.50 has to be considered, but still the factor can range between 1.45 and 3.11. Based on the coefficient of variation (CV_R) in Table 5, a 95 % confidence interval of ± 20 % can be estimated on the ratio accounting for the measurement uncertainty, i.e. 2.5 ± 0.5.

The digestate can be divided into 2 groups. The solid fraction and the dried digestate are more correlated with an average factor of 1.85 and 1.62, respectively. The liquid fraction and the raw digestate have a significant lower average factor of 1.35 and 1.41, respectively.
Still within the different matrix groups, deviations in the calculated factor are observed.

Table 6 Ratio factor between the oxygen uptake rate (OUR) at 30°C vs at 20°C as a function of matrix type

	N	Min	Max	Average	Median
ALL DATA	50	0.85	3.11	**1.96**	1.85
Compost-vfg	8	1.99	3.01	**2.52**	2.47
Compost-green	12	1.45	3.11	**2.50**	2.44
Digestate - solid fraction	8	1.23	2.62	**1.85**	1.80
Digestaat - liquid fraction	6	0.85	2.12	**1.35**	1.26
Digestaat - dried	6	1.26	1.86	**1.62**	1.63
Digestaat - raw	7	1.21	1.68	**1.41**	1.43
OBA-manure	3	1.24	2.11	**1.72**	1.79

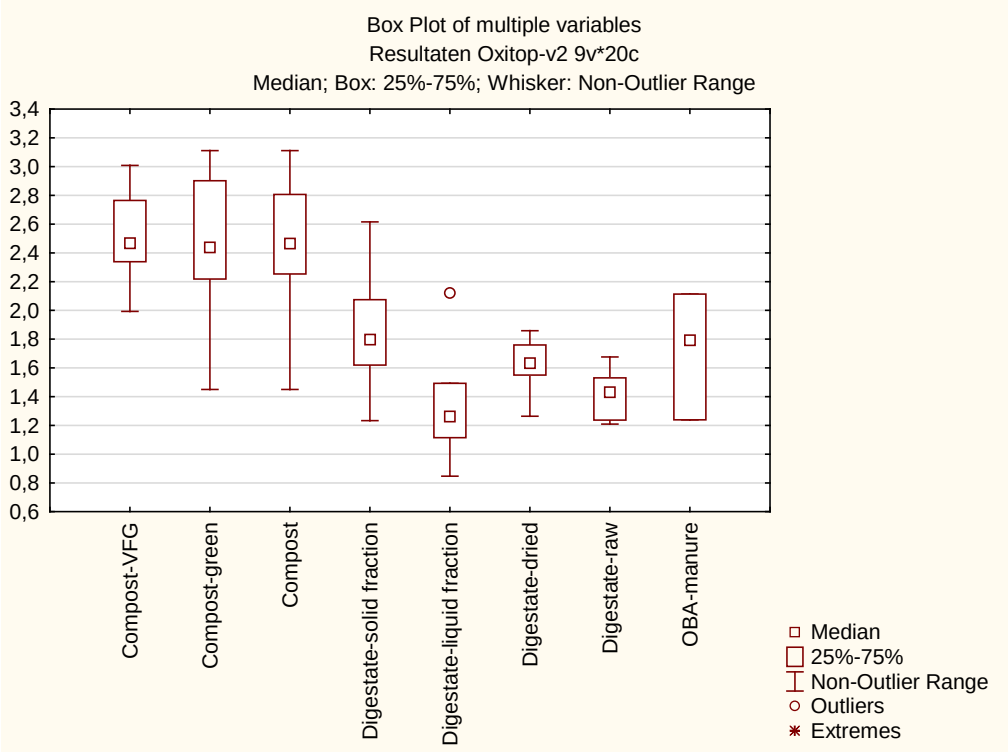

Figure 2 Grafical view of the ratio factor ($OUR_{30°C}/OUR_{20°C}$) asa function of matrix type.

In Figure 3 and Figure 4 the ratio factor (= $OUR_{30°C}/OUR_{20°C}$) is expressed as a function of the OUR at 20°C and 30°C, respectively. Each matrix type is indicated with a certain color. The results show that for the compost samples (red color) the results for the oxygen uptake rate is less than 10 mmol O_2/kg OM/h at 20°C and less than 20 mmol O_2/kg OM/h at 30°C, and the ratio factor is high (generally between 2.0 and 3.0). Due to the low reactivity of the compost samples, an increase in temperature of 10°C, will have a more significant impact on the oxygen uptake rate compared to

the more reactive digestate samples. Therefore, the ratio factor will also be higher for compost samples compared to digestate samples.

For dried digestates and the solid fraction of digestates the OUR at 20°C varies between 10 and 30 mmol O2/kg OM/h, while at 30°C these values are situated between 20 and 60 mmol O2/kg OM/h. The raw digestate and the liquid fraction of the digestate the obtained OUR results are much higher and situated above 20 mmol O2/kg OM/h and 40 mmol O2/kg OM/h, respectively.

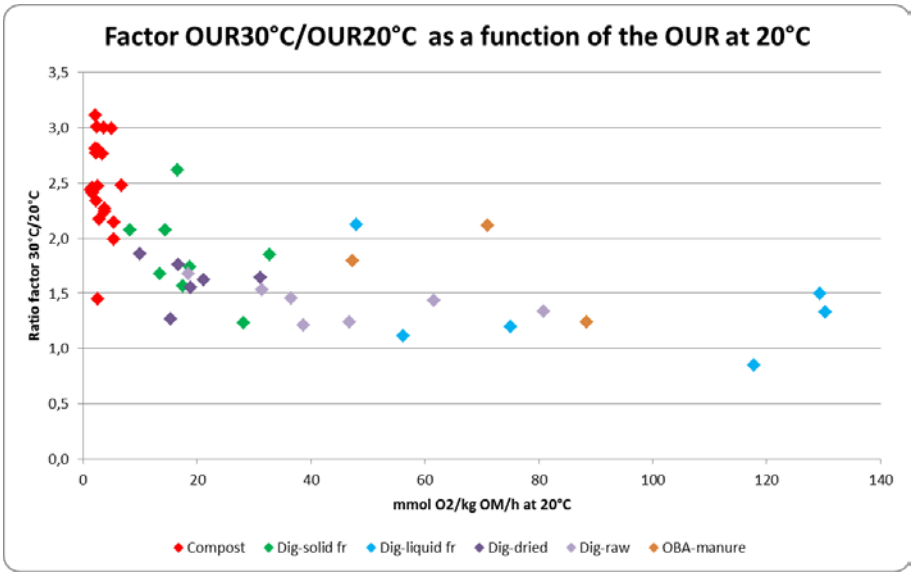

Figure 3 Factor $OUR_{30°C}/OUR_{20°C}$ as a function of the OUR at 20°C

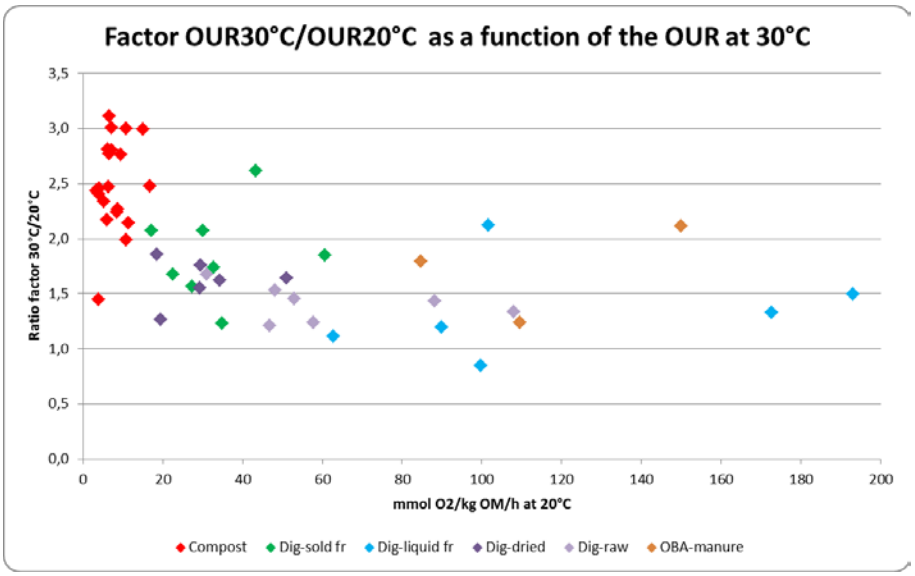

Figure 4 Factor $OUR_{30°C}/OUR_{20°C}$ as a function of the OUR at 30°C

In Figure 5 the factor is expressed as a function of the moisture content. The different groups of matrices can clearly be differentiated.

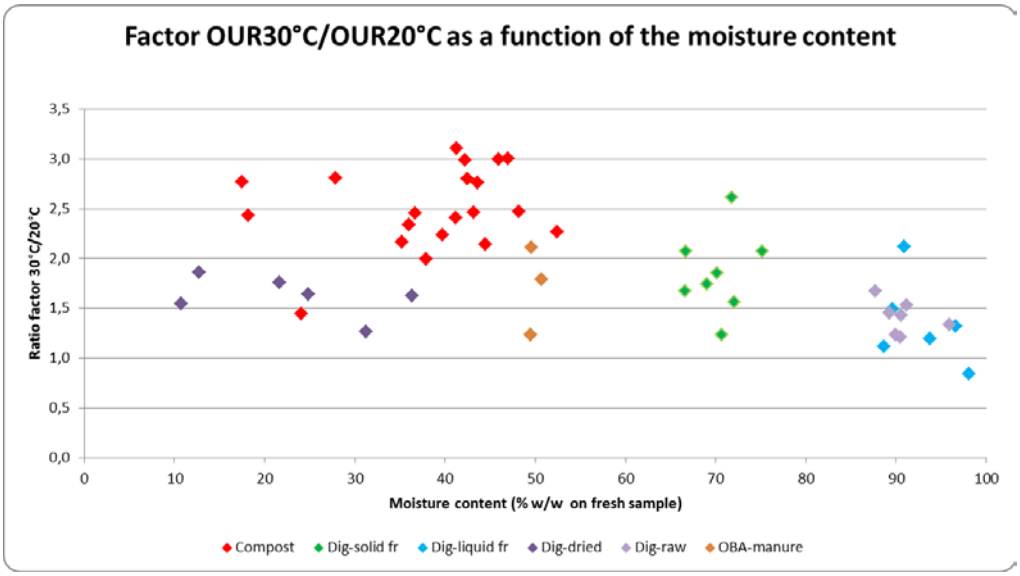

Figure 5 Factor $OUR_{30°C}/OUR_{20°C}$ as a function of the moisture content

3.3. CONCLUSIONS

Taken into account the results for the oxygen uptake rate (OUR) at 20°C and 30°C for the 50 samples analysed (compost and digestates), an average factor (= $OUR_{30°C}/OUR_{20°C}$) of 1.96 (varying between 0.85 and 3.11) is achieved, which is in line with the so-called Q10 factor.

Due to the variation in the ratio for the different matrices, it is recommended to use different factors for compost and digestate to correct the OUR results obtained at 20°C to OUR results at 30°C.

CHAPTER 4 DISCUSSION OF RESULTS AND FEASIBILITY OF EU QUALITY CRITERIA PROPOSAL

In this chapter, the results for the self-heating test and the respirometric test at 20°C and 30°C will be discussed in relation to the proposed EU quality criteria for the aerobic biological stability. Relying on our expertise and historical data with respect to these type of measurements, the feasibility of the proposed limit values is also commented.

4.1. EXPERIENCE AND QUALITY REQUIREMENTS IN FLANDERS

The analytical method to determine the biological stability using the respirometric method (OxiTop® measuring system) was added to the Compendium[4] back in 2008. Over 10 years of experience with this method strengthens the position that this method is applicable for all type of matrices (from very liquid materials of <2% dry matter up to dried products up to >90% dry matter. The available historical dataset is composed of measurements of the respiration rate or oxygen uptake rate (OUR) (mmol/kg organic matter/h), performed at 20°C, on different compost and digestate matrices. Moreover, for compost-like products, there has always been, up to now, a reference of the measured OUR with the stability measured with the self heating test (Rottegrad Method). Although there is a clear relationship between these two methods, in practice, some differences still occur. This is why the Belgian Federal Public Service Health, Food Safety and Environment allows the use of both methods to indicate the stability of fertilisers and soil improvers. In general, for compost samples a correlation between both methods can be derived, as presented in Table 7.

Table 7 Relationship between OUR (20°C) and self heating test derived from historical data in Flanders (Belgium)

Description condition	Oxygen uptake rate (OUR) (at 20°C)	Self heating test T_{max} (°C)	Self heating test Class
Very stable, low activity	< 5 mmol O_2/kg OM/h	≤ 30	V
Stable, limited activity	5 - 10 mmol O_2/kg OM/h	30.1 –40.0	IV
Moderately stable, active	10 - 15 mmol O_2/kg OM/h	40.1 – 50.0	III
Relative fresh, high activity	15 - 25 mmol O_2/kg OM/h	50.1 – 60.0	II
Fresh, very high activity, unstable	> 25 mmol O_2/kg OM/h	> 60	I

This classification is also confirmed by the data collected from the samples analysed in this study. The data (see Annex A) show that all compost samples show a respirometric value (at 20°C) of < 10 mmol O_2/kg organic matter/h and a minimum value for the self heating test of IV or V.

While assessing the legally obliged quality assurance of compost and digestate products, the parameter oxygen consumption (respirometric method) was analysed in 448 samples[b] annually by accredited labs, on behalf of Vlaco. The distribution of these samples was as follows: 143 compost samples, 25 biologically treated manure samples and 280 samples of digestate products. An even larger amount of samples was analysed, as the certified composting and anaerobic digestion plants in the Flemish Region (Belgium) need to have extra analyses carried out on their own behalf (internal quality assurance). This equals a total of over 600 samples/year analysed on this parameter. The respirometric test using the OxiTop® measuring system with incubation temperature of 20°C was used on all fertilising and soil improving products from biological treatment of biowaste (since 2006 for compost and since 2008 for digestate products).

4.2. EVALUATION OF THE PROPOSED END OF WASTE CRITERIA FOR BIODEGRADABLE WASTE

Looking at the product quality requirements for compost and digestate as defined in the End of Waste criteria for biodegradable waste, the following two minimum stability criteria for compost are proposed:

- Respirometric index of maximum 25 mmol O_2/kg organic matter/h, measured according to standard EN 16087-1 (i.e. at 30°C).
- Minimum Rottegrad III, IV or V (self-heating test temperature rise of maximum 30 °C above ambient temperature), measured according to standard EN 16087-2 (i.e. at 22°C).

Here the respirometric index is determined at 30°C. Recalculating the respirometric value to 20°C, a mean factor of 2.5 (see Table 6 for compost) has to be taken into account and will result in a respirometric value (at 20°C) of ~ 10 mmol O_2/kg organic matter/h, which corresponds to the quality criteria as defined in Flanders. But, having in mind the relationship between the respirometric value (at 20°C) and the self-heating test (see Table 7), a minimum Rottegrad of III, IV or V doesn't correspond with the respirometric value of maximum 10 mmol O_2/kg organic matter/h. Therefore, the minimum Rottegrad should be limited to classes IV or V (no addition of class III).

For digestates, a similar evaluation can be performed. The stability criteria for digestates in the End of Waste criteria for biodegradable waste, is as follows:

- Respirometric index of maximum 50 mmol O_2/kg organic matter/h, measured according to standard EN 16087-1 (i.e. at 30°C).

Also here the respirometric index is determined at 30°C. Recalculating the respirometric value to 20°C, a mean factor of 1.6 (see Table 6 for average of -digestates) has to be taken into account and will result in a respirometric value (at 20°C) of ~ 30 mmol O_2/kg organic matter/h. In practice, it will be difficult to meet this quality requirement. After anaerobic digestion, a fraction of the organic material is not totally consumed anaerobically. Although residual biogas potential can be low in a digestate sample, it doesn't necessary include that the respirometric value of this sample is low. In fact, the data, as shown in Annex A for dried and the solid fraction of digestates, are situated in the range < 40 mmol O_2/kg organic matter/h (at 20°C) and exceed the proposed limit value. In Flanders a quality criteria of < 50 mmol O_2/kg organic matter/h (at 20°C) is applicable, and gives a good reflection of the values in the field.

Note: the aerobic biological acitivity of the raw and the liquid fraction of the digestates is still high and often situated above 50 mmol O_2/kg organic matter/h (at 20°C).

[b] Source: Vlaco quality assurance, 2016

4.3. EU ECOLABEL CRITERIA FOR SOIL IMPROVERS AND GROWING MEDIA

In the technical report related to '*Revision of the EU Ecolabel Criteria for Soil Improvers and Growing Media - Technical report and draft criteria proposal*', the following requirements for stability are proposed for soil improvers and mulch for non-professional applications and growing media for all applications:

- Respirometric index of maximum 15 mmol O_2/kg organic matter/h, measured according to standard EN 16087-1 (i.e. at 30°C).
- Minimum Rottegrad IV (self-heating test temperature rise of maximum 30 °C above ambient temperature), measured according to standard EN 16087-2 (i.e. at 22°C).

For soil improvers and mulch for professional applications the following requirements are proposed:

- Respirometric index of maximum 25 mmol O_2/kg organic matter/h, measured according to standard EN 16087-1 (i.e. at 30°C).
- Minimum Rottegrad III (self-heating test temperature rise of maximum 30 °C above ambient temperature), measured according to standard EN 16087-2 (i.e. at 22°C).

A similar deduction can be derived for these prescribed quality criteria.
A respirometric index of maximum 15 mmol O_2/kg organic matter/h, measured at 30°C, corresponds for compost-like products with a value of ~ 6 mmol O_2/kg organic matter/h, measured at 20°C (taken into account of a mean factor of 2.5, see Table 6 for compost). According to the historical dataset (see Table 7), this corresponds with a minimum Rottegrad of class V, and not IV as proposed above.

A respirometric index of maximum 25 mmol O_2/kg organic matter/h, measured at 30°C, corresponds for compost-like products with a value of ~ 10 mmol O_2/kg organic matter/h, measured at 20°C (taken into account of a mean factor of 2.5, see Table 6 for compost). The proposed level corresponds with de quality criteria as defined in Flanders, and is also achievable in practice. According to the historical dataset (see Table 7) this corresponds with a minimum Rottegrad of class IV or V, and not III as proposed above.

4.4. CONCLUSIONS

As proposal in the new EU Fertiliser Regulation, the limit value for the respirometric index is defined at 25 mmol/kg organic matter/h (measured at 30°C). In practice, and taking into account the historical dataset for the respirometric test, this value is not achievable for the majority of the digestate products. After anaerobic digestion, a fraction of the organic material is not totally consumed anaerobically. Although residual biogas potential can be low in a digestate sample, it doesn't necessary include that the respirometric value of this sample is low (or in this case <25 mmol/kg organic matter/h). This experience already showed up after analysing the digestate samples with the respirometric method at an incubation temperatures of 20°C. When the incubation temperature is raised up to 30°C (instead of 20°C), the limit value of <25 mmol/kg organic matter/h is certainly not achievable. This would only be the case for stable compost products with a Rottegrad class IV or even V.

CHAPTER 5 CONCLUSION

In the Flemish part of Belgium, the self-heating test for compost (since 1998) and the respirometric test (since 2006 for compost and since 2008 for digestate products) has been implemented for many years to determine the aerobic biological acitivity.

The procedure applied for the **self-heating** is in accordance with the European standard EN 16087-2 *Soil improvers and growing media – Determination of the aerobic biological activity –Part 2: Self heating test for compost*, which was developed in 2011 within CEN/TC 223 *Soil improvers and growing media*.
For the determination of the **respirometric value** the applied procedure differs with the European standard, developed in 2011 within CEN/TC 223 *Soil improvers and growing media*. The standard EN 16087-1 *Soil improvers and growing media – Determination of the aerobic biological activity – Part 1: Oxygen uptake rate (OUR)*[10] describes a similar method as the compendium method applied in Flanders. The main difference between both procedures is the applied incubation temperature. In the EN method 16087-1 a temperature of 30°C is applied, while in the compendium procedure used in Flanders a temperature of 20°C is applied.

All European standard methods related to oxygen demand/biological activity (EN ISO 9408, EN 1899, EN ISO 17556, EN ISO 10707, EN 16087-2) include an incubation temperature of ca. 20°C, except EN 16087-1 which prescribes a temperature of 30 ± 2°C. Therefore, in 2008, in the Flemish region of Belgium, an (harmonized) temperature of 20°C has been chosen as reference temperature for procedures involving oxygen demand. Especially, since the BOD determination at 20°C is a widely internationally adopted standard method, incubators and incubations rooms at 20°C are available in most laboratories. Besides, air conditioning systems for laboratories keep the thermostat setting generally at a roomtemperature of about 20°C.

The incubation temperature has a large effect on the outcome of the stability test. In the present study, it was demonstrated that the matrix (compost or digestate) affects the magnitude of the difference. Different ratio factors were calculated between the oxygen uptake rate result at 30°C and at 20°C for compost (average ratio ~2.5) and digestate (average ratio ~ 1.6).

In different EU quality criteria proposals (End of Waste criteria for biodegradable waste, EU Ecolabel criteria for soil improvers and growing media) a sound correlation between the respirometric value and the self-heating test (Rottegrad) is apparently lacking. Moreover, as proposal in the new EU Fertiliser Regulation, the limit value for the respirometric index is defined at 25 mmol/kg organic matter/h. In practice, and taking into account the historical dataset for the respirometric test, this value is not achievable for the majority of the digestate products when performed at 30°C.

Based on an extended historical dataset of compost samples, collected and analysed in Flanders (Belgium), a correlation between the respirometric value (determined at 20 ± 2°C) and the self heating test (Rottegrad, determined at 22 ± 2°C) could be derived and is presented below.

Description condition	Oxygen uptake rate (OUR) (at 20°C)	Self heating test T_{max} (°C)	Self heating test Class
Very stable, low activity	< 5 mmol O_2/kg OM/h	≤ 30	V
Stable, limited activity	5 - 10 mmol O_2/kg OM/h	30.1 –40.0	IV
Moderately stable, active	10 - 15 mmol O_2/kg OM/h	40.1 – 50.0	III
Relative fresh, high activity	15 - 25 mmol O_2/kg OM/h	50.1 – 60.0	II
Fresh, very high activity, unstable	> 25 mmol O_2/kg OM/h	> 60	I

Recommendation :

An accredited governmental control system for self-heating test (22 ± 2 °C) and respirometric test (20 ± 2°C) for the matrices compost and digestate is in place for more than 10 years in the Flemish region of Belgium. Based on the acquired expertise on the one hand and the need for harmonisation with the EN standard on the other hand, the inclusion of an incubation temperature of 20°C in the European Standard EN 16087-1 should be taken into consideration. The literature review, the compiled data, the comparative study and the discussion given in this report describe the rationale for this request.

ANNEX A: ANALYTICAL RESULTS OF THE COLLECTED SAMPLES (COMPOST AND DIGESTATES)

		Oxitop 20° (Meas. 1)	Oxitop 20° (Meas. 2)	Oxitop 30° (Meas. 1)	Oxitop 30° (Meas. 2)		Moisture content	Organic matter	Conductivity (1/5)	pH (H2O)	Density	Self heating test	
	Matrix type	mmol O2/kg OM/h	mmol O2/kg OM/h	mmol O2/kg OM/h	mmol O2/kg OM/h	Ratio factor 30°C/20°C	% (w/w) on fresh sample	% (w/w) on fresh sample	µS/cm on fresh sample		kg/l	Class	Max. Temp °C
2	Compost-VFG	3,6	4,1	8,0	9,1	2,2	39,7	22,1	2460	8,5	0,468	V	23,0
12	Compost-VFG	2,5	2,3	7,2	7,2	3,0	47	23,2	1891	8,1	0,474	V	23,7
22	Compost-VFG	2,8	4,1	9,1	9,9	2,8	43,6	18,8	1136	8,9	0,495	V	25,4
27	Compost-VFG	4,9	6,0	9,0	12,7	2,0	37,9	20,9	1901	9,2	0,473	V	24,8
31	Compost-VFG	6,4	7,1	16,7	16,8	2,5	48,2	17,2	1198	8,9	0,520	IV	33,9
41	Compost-VFG	1,6	1,7	4,0	4,3	2,5	36,7	16,5	1400	8,8		V	21,5
48	Compost-VFG	2,1	2,7	7,1	6,0	2,8	17,5	31,9	3800	8,2		V	25,7
50	Compost-VFG	1,3	1,5	3,1	3,6	2,4	18,2	23,4	3430	8,9	0,556	V	24,1
1	Compost-green	3,7	3,5	10,5	11,1	3,0	45,9	17,4	1246	8,6	0,543	V	22,7
3	Compost-green	4,7	5,3	14,8	15,4	3,0	42,2	23,5	1041	8,6	0,424	V	24,8
4	Compost-green	2,5	2,6	7,2	7,1	2,8	42,5	17,3	1224	8,6	0,584	V	22,7
11	Compost-green	2,4	2,6	6,2	6,3	2,5	43,2	20,5	1128	8,9	0,543	V	23,8
16	Compost-green	2,4	2,0	6,3	6,2	2,8	27,9	23,5	1059	8,6	0,496	V	23,2
28	Compost-green	1,6	1,6	4,0	4,0	2,4	41,2	23,2	573	8,9	0,496	V	24,6
29	Compost-green	2,1	2,2	6,7	6,4	3,1	41,3	19,5	627	8,5	0,512	V	22,5
30	Compost-green	3,9	3,8	8,5	8,9	2,3	52,4	20,5	654	8,8	0,449	V	28,3
36	Compost-green	5,6	5,1	11,6	11,4	2,1	44,5	16,3	1742	8,6	0,597	V	25,5
37	Compost-green	2,1	2,4	5,4	5,2	2,3	36	14,9	643	8,3	0,543	V	21,5
45	Compost-green	2,6	2,6	3,8	3,9	1,4	24,1	25,6	1356	8,7	0,488	V	23,6
49	Compost-green	2,4	3,2	5,9	6,2	2,2	35,2	13,8	1100	7,8		V	23,1
6	Digestate-thick fraction	14,0	15,0	29,7	30,3	2,1	75,2	20,3	2670	9	0,533		
8	Digestate-thick fraction	10,3	6,2	18,4	16,0	2,1	66,7	21	5800	9			
18	Digestate-thick fraction	18,4	16,8	28,0	26,8	1,6	72,1	20	3600	9,1			
20	Digestate-thick fraction	19,0	18,5	32,8	32,6	1,7	69	18,2	4120	8,9	0,553		
25	Digestate-thick fraction	31,3	34,3	61,3	60,0	1,9	70,2	13,9	2970	8,7	0,551		
33	Digestate-thick fraction	18,9	14,2	45,6	41,0	2,6	71,8	17	1700	8,9			
38	Digestate-thick fraction	13,8	13,2	21,6	23,5	1,7	66,6	28,9	1262	8,9	0,256		
43	Digestate-thick fraction	28,0	28,4	33,5	36,0	1,2	70,7	17,3	3750	9	0,68		
10	Digestate-thin fraction	129,1	129,7	188,6	197,8	1,5	89,6	4,9	5200	9			
14	Digestate-thin fraction	46,8	49,2	101,2	102,3	2,1	90,9	5,5	11000	8,6			
21	Digestate-thin fraction	137,9	122,8	157,6	187,9	1,3	96,6	1,3	9240	8,5	1,008		
24	Digestate-thin fraction	119,9	115,6	87,9	111,6	0,8	98,1	0,5	6120	8,3	1,001		
39	Digestate-thin fraction	70,3	79,9	95,3	84,7	1,2	93,8	4	5670	8,4	1,023		
47	Digestate-thin fraction	56,8	55,7	59,7	65,8	1,1	88,7	7	10800	8,6	1,043		
7	Digestate-dried	31,5	30,8	51,1	51,2	1,6	24,8	51,2	7520	9,7	0,682		
15	Digestate-dried	9,6	10,4	18,7	18,4	1,9	12,7	65	4100	9,5			
26	Digestate-dried	19,7	22,6	35,8	32,9	1,6	36,3	29	4010	8,4	0,54		
40	Digestate-dried	16,8	16,6	28,1	30,7	1,8	21,6	51,5	2380	9,4	0,291		
44	Digestate-dried	18,9	18,8	31,8	26,7	1,6	10,7	53,4	3030	8,9	0,475		
46	Digestate-dried	15,6	15,1	19,7	19,1	1,3	31,2	53,4	4730	9	0,451		
5	Digestaat-raw	18,6	18,3	30,9	31,0	1,7	87,7	8,9	5630	8,4	0,969		
9	Digestaat-raw	60,8	62,4	91,2	85,1	1,4	90,6	4,5	11000	8,3			
13	Digestaat-raw	34,4	38,5	55,4	50,6	1,5	89,3	7	12000	8,5			
17	Digestaat-raw	30,4	32,4	50,3	45,8	1,5	91,2	6,4	6200	8,8			
19	Digestaat-raw	44,0	33,3	46,2	47,3	1,2	90,5	5,9	4600	8,3			
23	Digestaat-raw	84,2	77,5	115,2	100,7	1,3	95,9	1,8	5610	8,5	1,004		
42	Digestaat-raw	48,1	45,5	58,7	57,1	1,2	90	5,8	7970	8,9	1,043		
32	OBA-manure	71,1	70,8	148,5	151,5	2,1	49,6	31,4	8870	7,4	0,549	I	63,5
34	OBA-manure	52,4	42,2	79,5	90,0	1,8	50,7	31,6	6160	8,5	0,45	I	65,2
35	OBA-manure	83,3	93,5	112,6	106,5	1,2	49,5	27,6	6310	10,2	0,575	V	23,6

ANNEX B: REVISION OF THE EU ECOLABEL CRITERIA FOR SOIL IMPROVERS AND GROWING MEDIA

At European level a technical report was written related to '*Revision of the EU Ecolabel Criteria for Soil Improvers and Growing Media - Technical report and draft criteria proposal*'[8]. In this annex the full text related to the Stability criterion is taken over.

Abstract: This technical report is aimed at providing a sound base to the revision process of the EU Ecolabel criteria for Soil improvers and Growing Media. Currently, separate sets of EU Ecolabel criteria exist for Soil improvers (Decision 2006/799/EC) and Growing media (Decision 2007/64/EC). The revision process spanned both product groups; thus common criteria for both Soil improvers and Growing media are developed, only distinguishing between technical product characteristics where necessary. Another objective of this revision is addressing the possibility to broaden the current scope to the product mulch, as it has been identified as a differentiated sub-product within the product group 'soil improvers'. To achieve those objectives, a Preliminary Report was produced together with the first version of this technical report as a working document. Hence, this document was developed to undergo the stakeholder consultation, which is crucial to come up with criteria adapted to the market reality while being able to select the best environmental performance products available on the market. After an extensive stakeholder's consultation process, this current version of the Technical Report supporting the draft criteria for growing media, soil improvers and mulch, constitutes the support of the criteria set voted positive by the Regulatory Committee.

Criterion 7: Stability

This criterion applies to growing media, soil improvers and mulch, with the exception of mulch totally composed of lignocellulosic constituents and mineral growing media.

Soil improvers and mulch for non-professional applications and growing media for all applications, shall meet one of the following requirements:

Stability parameter	Requirement
Maximum Respirometric index	15 mmol O_2/kg organic matter/h
Minimum Rottegrad, where applicable	IV (self-heating test temperature rise of maximum 20°C above ambient temperature)

Soil improvers and mulch for professional applications shall meet one of the following requirements:

Stability parameter	Requirement
Maximum Respirometric index	25 mmol O_2/kg organic matter/h
Minimum Rottegrad, where applicable	III (self-heating test temperature rise of maximum 30°C above ambient temperature)

Assessment and verification

The applicant shall provide the test reports conducted in accordance with the following testing procedures:

- EN 16087-1 Soil improvers and growing media - Determination of the aerobic biological activity - Part 1: Oxygen uptake rate (OUR)

- EN 16087-2 Soil improvers and growing media - Determination of Aerobic biological activity - Part 2: Self heating test for compost

Rationale and discussion

This proposal is not completely new, since the current criterion on Provision of information includes a statement about the stability of organic matter (stable or very stable) by national or international standard.

The stability criterion is proposed based on several comments from stakeholders that pointed out the concerns related to unstable products. A stability requirement can help prevent the introduction of materials that have hardly undergone any treatment (e.g. so-called "shred-and-spread" compost). Furthermore, greenhouse gas emissions may occur during transport and storage of all compost and digestate materials. The limits proposed were the values required to classify a product as 'stable' according to the two referred standards. This criterion is also aimed at retaining and standardizing the current criterion of provision of information, where a statement about the stability of organic matter (stable or very stable) by national or international standard is required.

On the other hand, a maximum C/N ratio was recommended, as indicator of the decomposition of the organic matter in the soil improver and its grade of stability and maturity.

Stakeholders feedback

There was a wide consensus among the experts on the unsuitability of the C/N ratio, as it is not a good indicator for maturity and stability. Therefore, the requirement on C/N ratio is withdrawn.

It was also stressed that the stability criterion is irrelevant for mulches, since they are composed by vegetal by-products (barks, straws, wood chips...) which are very dry and stable.

Some stakeholders highlighted that there is a lack of robust European-wide experience with the proposed test method and validation of the limit values, thus they do not support implementing stability/maturity criteria for the EU Ecolabel for soil improvers and growing media. According to their view, there is no European wide acceptance of the proposed limit values, so, the criterion seems likely to have significant adverse effects on parts of the compost market. For example, with the proposed rotting degree of IV and V, fresh compost which is widely used as soil improver and organic fertilisers in the agricultural sector, will be excluded from the EU Ecolabel. In addition, if a stability criterion is decided, it needs to be based on a European standard and, there is no European validated test method available for digestates.

On the subject of the application of this criterion to digestates, it is important to stress that the minimum stability is meant to comply with the quality expectations of the market, both professional and non-professional. Some concerns about the quality of the products have been raised along the revision process, from the growing media manufacturers and from the agricultural associations. In this regard, some market barriers have been identified related to the level of stability of digestates, i.e. methane and ammonia emissions, unpleasant odour, ammonium content (WRAP, 2013). These barriers may be overcome with a further aerobic stabilization, and thus a minimum stability criterion is recommended to ensure the quality of the product, while enhancing the customers' perception of the waste-derived products.

Some Member States disagreed on the use digestates within the EU Ecolabel. In their view, digestate is a bio-reactive and therefore biologically unstable substance. If digestates could be used we need to set very strict requirements to prevent unstable digestates to be awarded with EU Ecolabel.

Taking into account the input from stakeholders, the criterion is proposed to be split on professional and non-professional applications, with a less strict limit values for the first ones. For non-professional applications of soil improvers and all application of growing media, the limits proposed are the values established to classify a product as 'stable'. According to Brinton *et al.* (1995), the compost stability can be classified based on their Rottegrad as shown in the table below:

Temperature rise above ambient (°C)	Official class of stability	Descriptors of class or group	Major group
< 10	V	Very stable, well-aged compost	Finish compost
10 - 20	IV	Moderately stable, curing compost	
20 - 30	III	Still decomposing, active compost	Active compost
30 - 40	II	Immature, young or very active compost	
> 40	I	Fresh, raw compost, just mixed ingredients	Fresh compost

Brinton *et al.* (1995)[9] also proposes end use categories derived from experience with composted source-separated residential food residues blended with yard-waste, and active compost (Rottegrad III) is applicable to fruity crops, while for general purpose gardening, Rottegrad IV compost is recommended.
Veeken *et al.* (2003)[5] proposed the following scheme for classification of biowaste and green waste compost based on specific oxygen uptake rate, which is shown in the table below, together with equivalent CO_2 production values calculated for this report.

Category of compost product	Oxygen uptake rate (mmol O_2/kg OM/h)	Oxygen uptake rate (mg O_2/kg OM/h)	Equivalent CO_2 evolution rate (mg CO_2/kg OM/day)
Very unstable	> 30	> 960	> 32
Unstable	15 – 30	480 – 960	16 – 32
Stable	5 – 15	160 – 480	5 – 16
Very stable	< 5	< 160	< 5

The limit values proposed for professional purposes are based on the End of Waste criteria for biodegradable waste report, which recommends the following stability criterion for compost:

One of those minimum stability requirements:

- Respirometric index of maximum 25 mmol O2/kg organic matter/h, measured according to standard EN 16087-1.
- Minimum Rottegrad III (self-heating test temperature rise of maximum 30 °C above ambient temperature), measured according to standard EN 16087-2.

The report also covers digestate, for which it recommends one of those minimum stability requirements

- Respirometric index of maximum 50 mmol O_2/kg organic matter/h, measured according to standard EN 16087-1.
- Organic acids content of maximum 1500 mg/l.

- Residual biogas potential of maximum 0.25 l/g volatile solids.

The report set those values to ensure a minimum stability needed to avoid methane and odour emissions during uncontrolled anaerobic conditions after sales (e.g. during storage).

According to the EoW for biodegradable waste report, many Member States already regulate compost stability, whether by imposing certain methods and associated limit values or by requiring a declaration. Most methods are based on a self-heating test or a respirometric index. Studies on the evaluation of the different systems used for stability measurement indicate that the different approaches are actually highly correlated, at least for compost stability. A WRAP study (WRAP, 2009) suggested that there is no clear superiority of any given method. Nonetheless, EN standards exist for oxygen uptake rate and self-heating tests (EN 16087-1 and EN 16087-2) and hence these should be preferable over national standards or commercial measurement tools to provide a level playing field. For digestate stability, it appears that fewer measurement methods are being used at present. Most of them are based on organic acids testing or assessment of remaining biodegradability through an aerobic respirometric test or anaerobic biogas formation potential. Provided that digestate is a less stabilized material than compost, a less strict respirometric index is proposed by the EoW for biodegradable waste report, together with equivalent values based on other tests commonly used for digestates.

The minimum stability for professional uses proposed in the EU Ecolabel criterion is meant to ensure a sufficient level of stability, while preventing the introduction of materials that have hardly undergone any treatment (e.g. so-called "shred-and-spread" compost), despite the fact that these untreated materials might be used in agriculture. The figure proposed ensures that the materials were processed to get a reasonable level of stabilization by means of aerobic stabilization. In the case of digestates, a post-composting process would be needed, to overcome the market barriers identified and to improve the perception of the waste-derived products. This aims to avoiding methane and odour emissions, while it suffices to comply with the market expectations for professional purposes, which often use active compost, for soil improvers or mulch, according to the feedback received from the stakeholder. Nevertheless, the national requirements will supersede these minimum stability requirements, if they are stricter.

For growing media, the manufacturers reported that they use stable/mature compost, and therefore a specific value for professional uses is not needed.

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