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Solid recovered fuels — Determination of the current rate of aerobic microbial activity using the real dynamic respiration index

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National foreword

This British Standard is the UK implementation of EN 15590:2011. It supersedes DD CEN/TS 15590:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/17, Solid biofuels.

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

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English Version

Solid recovered fuels - Determination of the current rate of aerobic microbial activity using the real dynamic respiration index

Combustibles solides de récupération - Détermination du taux d'activité microbienne utilisant l'indice de respiration dynamique

Feste Sekundärbrennstoffe - Bestimmung des aktuellen Grades aerober mikrobieller Aktivität mittels des realen dynamischen Respirationsindexes

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Contents

Page

Foreword

This document (EN 15590:2011) has been prepared by Technical Committee CEN/TC 343 “Solid recovered fuels”, the secretariat of which is held by SFS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2012, and conflicting national standards shall be withdrawn at the latest by March 2012.

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This document supersedes CEN/TS 15590:2007.

The following changes have been introduced:

— title and scope change; potential microbial self-heating is revised by current rate of aerobic microbial activity;

— results of inter-laboratory tests supplemented as an informative Annex C.

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

Introduction

This document specifies the method used for the determining the current rate of aerobic microbial activity of SRF using the real dynamic respirator index.

The current rate of aerobic microbial activity measures the biological stability under the actual chemical and physical properties of the SRF. The biological stability determines the extent to which readily biodegradable organic matter has decomposed. Therefore, the *RDRI* identifies the actual point reached in the decomposition process and represents a gradation on a recognised scale of values.

1 Scope

This European Standard specifies a method to determine the current rate of aerobic microbial activity of a solid recovered fuel. The methods indirectly estimate the potentiality of odours production, vectors attraction etc. The current rate of biodegradation can be expressed in milligrams O_2 kg^{-1} dm h^{-1}.

WARNING — SRF can contain potentially pathogenic organisms. Take appropriate precautions when handling them and those whose properties are unknown.

2 Normative references

The following referenced documents are indispensable for the application 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 15357:2011, *Solid recovered fuels — Terminology, definitions and descriptions*

EN 15443, *Solid recovered fuels — Methods for the preparation of the laboratory sample*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15357:2011 and the following apply.

3.1
easily biodegrable organic compounds
organic substances available for decomposition by micro-organisms within a real dynamic respiration test

3.2
hourly real dynamic respiration index
value of respiration index calculated every hour

3.3
lag or latency phase
interval of time required for the microbial flora to acclimatize during the course of the real dynamic respirometric test

3.4
mean particle size
aperture size of the sieve used for determining the particle size distribution of solid recovered fuels through which at least 50% by mass of the material passes

3.5
respiration index
rate of oxygen uptake expressed as milligram oxygen per kilogram total dry matter (dm) per hour

3.6
real dynamic respiration test
test measuring the respiration index under specific conditions including forced air flow

3.7
real dynamic respiration index
RDRI
average value of the respiration indexes representing 24 h showing the highest aerobic microbial activity

NOTE See Figure A.1.

4 Symbols and abbreviations

This European Standard uses the following symbols and abbreviations:

RDRI Real Dynamic Respiration Index

$RDRI_h$ hourly Real Dynamic Respiration Index

d_m dry matter in kg

5 Principle

The method for determining the current rate of aerobic microbial activity specified in this European Standard is based on measuring the oxygen uptake rate by micro-organisms to biodegrade easily degradable organic matter of the sample itself under defined continuous airflow and adiabatic conditions.

The test involves keeping the sample under observation in the respirometer (dynamic test system) for 1 day to 4 days according to the duration of the lag phase (if present), taking the index value at hourly intervals ($RDRI_h$) (Clause 8). Moreover, if at the end of the fourth day, the *RDRI* trend is constant or growing, the respirometric test is prolonged with the acquisition of at least others 24 values ($RDRI_h$) (see Figure A.1).

6 Apparatus

The apparatus consists of:

6.1 Dessicator.

6.2 Continuous flow aerobic respirometer, composed of (see Figure 1):

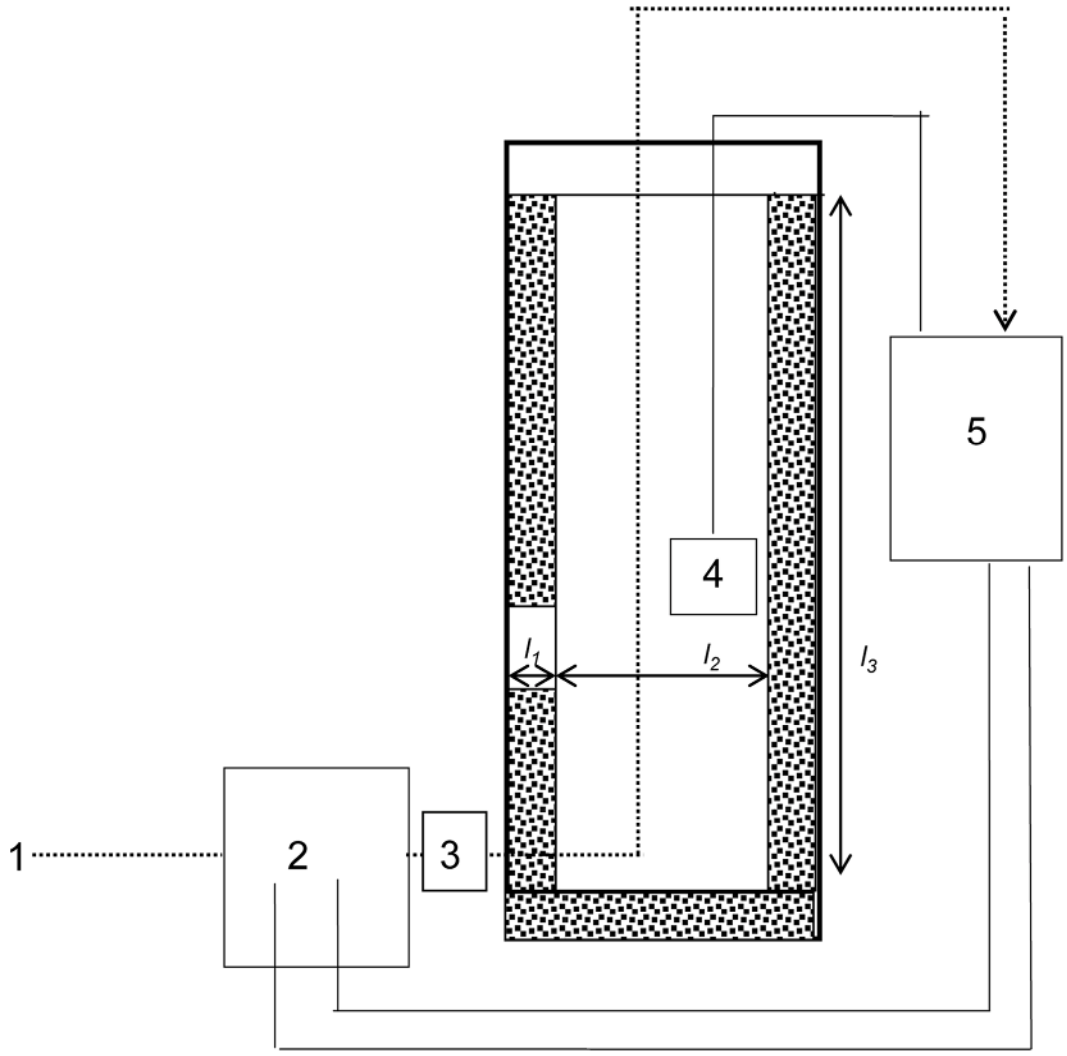
— hermetically sealed adiabatic reactor with the minimum operating volume expressed in litres, equal to or less than the average sample size expressed in millimeters and not greater than 30 mm (for example, for a sample of average size less than 10 mm, the reactor volume is 10 l); the reactor structure must force the input air to cross the entire sample before leaving the reactor, avoiding mixing the of input air and exhaust air;

— reactor air-tightness verification system;

— aeration system provided with flow regulator and capacity gauge;

— system for sampling oxygen concentration in exhaust air (% $^{v}/_{v}$);

— system of data acquisition continuously memorizing the measured parameters at 1 h intervals; the data memorized must be the average of all values read (at least 60) during the interval considered.



Key

1 air flow
2 air pump and probe for measuring of the temperature of the air inlet
3 flow adjustment and flow meter (0-200 l h^{-1})
4 probe for measuring of the temperature of the SRF
5 oxygen analyser and control and evaluation equipment
l_1 thickness of the external walls of the reactor[1)] (70 mm ± 5 mm)
l_2 internal diameter of reactor
l_3 internal height of reactor
l_3/l_2 1,344 ± 0,002

[1)] An insulating materials shall be employed (i.e Polypropylene).

Figure 1 — Diagram of the continuous flow aerobic respirometer

7 Procedure

7.1 Step 1 – Procedure sample preparation (if required).

The size reduction procedure shall be done following EN 15443.

7.2 Step 2 – Instrumentation calibration

Step 2 consists of the following procedures:

— Calibrate the oxygen probe in air as described in the instruction manual;

— Check if the reactor closes properly avoiding air losses.

7.3 Step 3 – Loading the reactor

Step 3 consists of the following procedure:

— Introduce a known weight of untreated sample filling the reactor completely (the exact amount depends by the reactor size (see Clause 6), avoiding formation of aggregates or compacting the SRF.

7.4 Step 4 – Analysis set up

Step 4 consists of the following procedures:

— Set up the data acquisition system and measure the parameters (O_2 and volume of air) for at least 4 days. Whatever the *RDRI* trend is at the end of the fourth day, whether it be constant or growing, continue the acquisition until at least 24 values have been recorded ($RDRI_h$) (see Figure A.1).

— Set up an initial flow of air and if necessary, adjust the flow during the analysis to guarantee that values of oxygen concentration in the exhaust air are within the 14 % v/v to 16 % v/v interval.

8 Calculation of the *RDRI* results

The measure of the volume of oxygen consumed by aerobic biological activity is deduced from the difference in oxygen concentration between the air input into the respirometer and the air output from it (Equation (1)), and calculated as the average of the hourly Real Respirometric Indices ($RDRI_h$) in the 24 h during which the microbial respiration is highest (Equation (2)).

The final value of the *RDRI* will therefore be calculated by using the following procedure:

— identify the maximum $RDRI_h$ value (Equation (1)) reached during the course of the test;

— identify the next 23 highest consecutive $RDRI_h$ values below the maximum $RDRI_h$;

— calculate the average of the 24 $RDRI_h$ values identified (See Equation (2) and Figure A.1).

$$RDRI_h = \frac{Q \times (O_{2i} - O_{2o}) \times 31.98}{V_g \times d_m} \quad (1)$$

$$RDRI = \frac{\sum_{h=1}^{24} RDRI_h}{24} \text{ for } t_c, \quad (2)$$

where

$RDRI_h$ is the Hourly Real Dynamic Respiration Index (calculated every h); $RDRI_h$ is expressed as mg O_2 kg^{-1} dm h^{-1};

Q is the air flow in l h^{-1};

($O_{2i} - O_{2o}$) is the difference between the concentration of the oxygen of the air in input (O_{2i}) and the concentration of the oxygen in the air output (O_{2o}) from the respirometer in ml l^{-1};

V_g is the volume occupied by a mole of gas in l, where

$$V_{g,2} = V_{g,1} \times \frac{T_2}{T_1} \quad (3)$$

where

$V_{g,1}$ is the gas volume in standard conditions for temperature and pressure which are 273,15 K and P is 1 atm; $V_{g,1}$ is equal to 22,4 l mol^{-1};

T_1 is the temperature in standard conditions; T_1 is equal to 273,15 K;

T_2 is the temperature of the air in input to the respirometer in K;

31.98 is the molecular weight of oxygen molecule (O_2) in g mol^{-1};

d_m is the mass of dry matter, in kg, where

$$d_m = \frac{w_{dm,r} \times m_s}{100}$$

where

$w_{dm,r}$ is the mass fraction of dry matter, in percent;

m_s is the sample mass, in kg;

t_c is the time period (24 h) in h during which the highest consecutive values of $RDRI_h$ are recorded (see Figure A.1 – phase C).

Current rate of aerobic microbial activity is expressed as *RDRI* (see Annex B).

The precision of the *RDRI* is reported in Annex C.

9 Storage and labelling samples

Samples shall be stored in tightly-closed containers at 4°C for not more than 7 days before the analysis. Each sample shall be labelled with a unique identification containing the identification of the sample from which it was obtained.

10 Test reports

The test report shall include at least the following information:

a) reference to this Standard, EN 15590;

b) all necessary information for the identification of the test compound;

c) all the measured and calculated data (for example in tabular form) obtained as well as the degradation curve;

d) receipt date of the laboratory sample and the beginning and end dates of the test;

e) complete identification of the laboratory sample;

f) storage conditions;

g) identification of the test equipment and instruments used;

h) *RDRI* from the calculations as milligrams of oxygen for kilograms of SRF dry matter for hour (mg O_2 kg^{-1} dm h^{-1});

i) the reasons, in the event of rejection of the test;

j) any alteration of the standard procedure or any other circumstance that may have affected the results;

k) any deviation from the test method and the reason for this deviation together with all circumstances that have influenced the results.

Annex A
(informative)

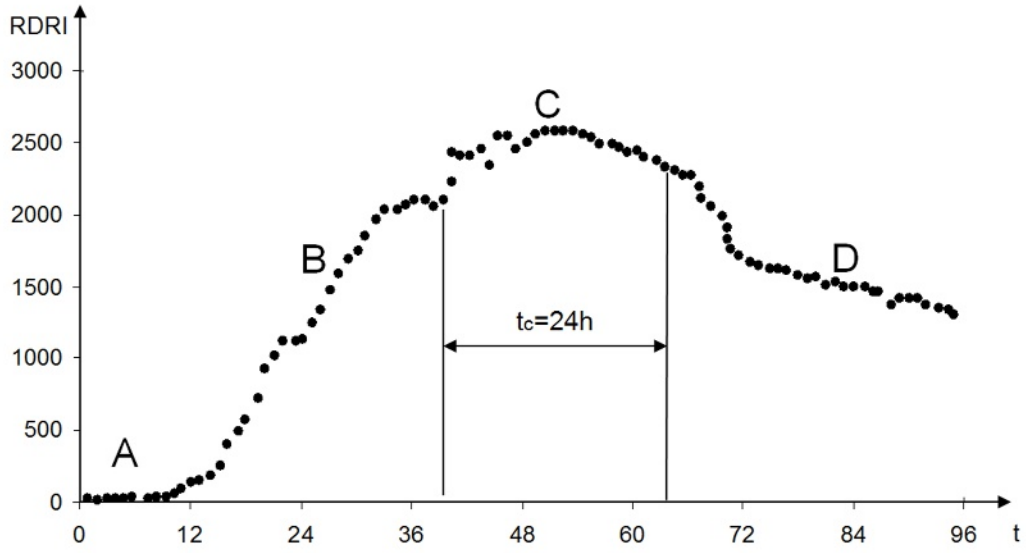
RDRI trend

The typical trend of a Real Dynamic Respiration Index graph (see Figure A.1) is characterized by an initial lag or latency phase (see Figure A.1 – phase A), which when present can continue for several days. Following phase A, if the internal chemical-physical conditions in the sample favour the development of microbial flora, in agreement with the consequent multiplication of the micro organisms, the *RDRI* graph trend becomes exponential (see Figure A.1 – phase B).

The third phase (see Figure A.1 – phase C), starts with the progressive diminution of the rapidly biodegradable compounds, whose reduction causes a slowing of the microbial degradation activity, and so the factors of multiplication and deaths of the micro organisms come into equilibrium. In this case the *RDRI* graph is more or less constant.

The fourth and final phase (see Figure A.1 – phase D) describes a progressive diminution of the *RDRI* values revealing the slowing down of degradation phenomenon because of the reduction of the easily biodegraded substrate.

Negative interference can be caused by toxic substances or conditions inhibiting metabolic activity of aerobic micro-organisms.



Key

A	Lag phase (see 3.3)
B + C + D	Active phase

Figure A.1 — RDRI analysis trend as a function of time

Annex B
(normative)

RDRI interpretation

The current rate of aerobic microbial activity of an SRF is determined by the *RDRI* and shall be evaluated on a qualitative basis as reported in Table B.1.

Table B.1 — Current rate of aerobic microbial activity

RDRI **mg O_2 kg^{-1} dm h^{-1}**	**Current rate of aerobic microbial activity of SRF**
< 500	Very low
500 to 1 000	Low
1 000 to 2 000	Moderately high
2 000 to 3 000	High
> 3 000	Very high

Annex C
(informative)

Results of the inter-laboratory test

C.1 Introduction

The precision of a method is represented by its repeatability and reproducibility. The repeatability (r) and reproducibility (R) limits (ISO 5725-2 [4] and [5]) are the maximum admissible difference between two measurements made consecutively by the same laboratory (repeatability) and by two different laboratories (reproducibility) respectively. If the differences registered are higher than r or R, the results become suspect.

The validation process will be carry out considering two different SRF samples that will be analysed in six replicates for three laboratories.

C.2 Laboratory analysis

The analytical activity regarded:

— the dry matter content determination (three determinations for each *RDRI* replicate for each laboratory);

— the current rate of microbial activity of SRF by using the *RDRI* (6 replicates) determinations for each laboratory.

C.3 Statistical analysis

The arithmetic mean (μ), the standard deviation (SD) and the relative standard deviation (RSD) for dm and *RDRI*, for each laboratory (A, B and C) were calculated by considering the data set available. The grand mean (M) was calculated for each parameter.

Straggler and outlier values for each sample and parameter analysed were detected by using the Cochran (for standard deviation) and Grubbs (for means) tests.

Results were then processed to calculate the repeatability (s_r) and the reproducibility (s_R) standard deviation as reported by ISO 5725-2:1994.

Finally, all analytical data were processed to determine the repeatability (r) and reproducibility (R) limits (ISO 5725-2:1994) which were calculated as follows:

$$r = t_{\infty} * \sqrt{2} * s_r = 2.8 * s_r$$

$$R = t_{\infty} * \sqrt{2} * s_R = 2.8 * s_R$$

where

s_r and s_R are the repeatability and reproducibility standard deviations,

t_{∞} is the Student's t (2 tails) for $v = n - 1$ and

Results and descriptive statistics for all the parameters, samples and laboratories considered are reported in Tables C.1 to C.3.

Table C.1 — Total solid content of SRF sample

SRF Sample	Total solid (% w/w)	N	Mean	Standard deviation	Relative standard deviation
1	Lab A	18	70,52	1,96	2,78
	Lab B	18	70,09	1,80	2,57
	Lab C	18	68,8	1,30	1,88
2	Lab A	18	52,36	1,50	2,86
	Lab B	18	51,03	3,56	6,98
	Lab C	18	48,91	1,60	3,27

Table C.2 — *RDRI* of SRF sample

SRFsample	*RDRI* (mg O_2 kg^1 dm^- h^{-1})	N	Mean	Standard deviation	Relative standard deviation
1	Lab A	6	498	99	19,87
	Lab B	6	617	78	12,64
	Lab C	6	462	70	19,39
2	Lab A	6	1 571	207	13,17
	Lab B	6	1 701	374	22,01
	Lab C	6	1 568	238	15,15

Table C.3 — Standard deviation of repeatability and reproducibility, and repeatability and repeatability and reproducibility limit

RDRI **($mgO_2\ kg^{-1}\ dm\ h^{-1}$)**	**M[a]**	s_r[b]	r[c]	s_R[d]	R[e]
SRF 1	526	94	262	126	352
SRF 2	1 613	269	752	282	790

[a] Grand mean

[b] Repeatability standard deviation

[c] Limit of repeatability = Repeatability

[d] Reproducibility standard deviation

[e] Limit of reproducibility = Reproducibility

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[1] CEN/TS 15414-1, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 1: Determination of total moisture by a reference method*

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[3] EN 15442, *Solid recovered fuels — Methods for sampling*

[4] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurements methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[5] Horwitz, W. (1995), "Protocol for the design, conduct and interpretation of method-performance studies", *Pure & Applied Chemistry,* Vol. 67, N° 2, pp. 331-343

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