BRITISH STANDARD

BS EN
14046:2003

Packaging — Evaluation of the ultimate aerobic biodegradability and disintegration of packaging materials under controlled composting conditions — Method by analysis of released carbon dioxide

The European Standard EN 14046:2003 has the status of a British Standard

ICS 13.030.99; 55.040

National foreword

This British Standard is the official English language version of EN 14046:2003.

The UK participation in its preparation was entrusted to Technical Committee PKW/4, Packaging and the environment, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 22 May 2003

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 22, an inside back cover and a back cover.

The BSI copyright date displayed in this document indicates when the document was last issued.

Amendments issued since publication

Amd. No.	Date	Comments

ISBN 0 580 41854 5

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14046

March 2003

ICS 13.030.99; 55.040

English version

Packaging - Evaluation of the ultimate aerobic biodegradability and disintegration of packaging materials under controlled composting conditions - Method by analysis of released carbon dioxide

Emballage - Evaluation de la biodégradabilité aérobie ultime et de la désintégration des matériaux d'emballage dans des conditions contrôlées de compostage - Méthode par analyse du dioxyde de carbone libéré

Verpackung - Bestimmung der vollständigen aeroben biologischen Abbaubarkeit und Desintegration von Packstoffen unter kontrollierten Kompostierbedingungen - Verfahren mittels Analyse des freigesetzten Kohlenstoffdioxids

This European Standard was approved by CEN on 27 December 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 14046:2003 E

Contents

Foreword

This document (EN 14046:2003) has been prepared by Technical Committee CEN/TC 261 "Packaging", the secretariat of which is held by AFNOR.

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 September 2003, and conflicting national standards shall be withdrawn at the latest by September 2003.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document contains annexes A, B, C, D and E, which are informative.

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

1 Scope

This European Standard specifies a method for the evaluation of the ultimate aerobic biodegradability of packaging materials based on organic compounds under controlled composting conditions by measurement of released carbon dioxide at the end of the test. This method is designed to resemble typical aerobic composting conditions for the organic fraction of mixed municipal solid waste. The packaging material is exposed in a laboratory test to an inoculum which is derived from compost. The aerobic composting takes place in an environment where especially temperature, aeration and humidity are closely monitored and controlled. The test method is designed to yield a percentage and rate of conversion of carbon of the test material to released carbon dioxide.

The conditions described in this European Standard do not necessarily always correspond to the optimal conditions allowing the maximum degree of biodegradation to occur.

2 Normative references

Not applicable.

3 Terms and definitions

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

3.1
test material
packaging materials made from organic compounds normally tested in compact forms at a suitable size

3.2
ultimate biodegradation of a test material
level of biodegradation achieved when the test material is utilised by micro-organisms resulting in the production of carbon dioxide, water, mineral salts and new microbial cellular constituents (biomass)

3.3
total dry solids
amount of solids obtained by taking a known amount of test material or compost and drying at about 105 °C to constant weight

3.4
volatile solids
amount of solids obtained by subtracting the residues of a known amount of test material or compost after incineration at about 550 °C from the total dry solids content of the same sample. The volatile solids content is an indication of the amount of organic matter

3.5
theoretical amount of formed carbon dioxide ($ThCO_2$)
theoretical maximum amount of carbon dioxide formed after oxidising a chemical compound completely, calculated from the molecular formula; expressed in this case as mg carbon dioxide per mg or g test compound

3.6
lag-phase
time from the start of a test until adaptation and/or selection of the degrading micro-organisms are achieved and the biodegradation degree of a chemical compound or organic matter has increased to about 10 % of the maximum level of biodegradation, recorded in days

3.7
maximum level of biodegradation
maximum biodegradation degree of a chemical compound or organic matter in a test, recorded in per cent, above which no further biodegradation takes place during the test

3.8
biodegradation phase
time from the end of the lag phase of a test until about 90 % of the maximum level of biodegradation has been reached, recorded in days

3.9
plateau phase
time from the end of the biodegradation phase until the end of the test, recorded in days

4 Principle

The test method is designed to be an optimised simulation of an intensive aerobic composting process and determines the ultimate biodegradability of a test material under controlled aerobic composting conditions. The inoculum consists of stabilised and mature compost if possible derived from composting the organic fraction of municipal solid waste.

The test material is mixed with the inoculum and introduced into a static composting vessel where it is intensively composted under optimum oxygen, temperature and moisture conditions for a test period of usually 45 or more days.

During the aerobic biodegradation of the test material, carbon dioxide, water, mineral salts and new microbial cellular constituents (biomass) are the ultimate biodegradation products. The carbon dioxide produced is continuously monitored or measured at regular intervals in the test and blank vessels and integrated to determine the cumulative carbon dioxide production. The percentage of biodegradation is obtained by comparing the carbon dioxide produced from the test material to the maximum amount of carbon dioxide which could be derived from the test material and which is calculated from its measured total organic carbon (TOC). This percentage of biodegradation will not include the amount of carbon converted to new cell biomass which is not in turn metabolised to carbon dioxide during the course of the test.

Additionally at the end of the test the weight loss of test material may be determined.

5 Test Environment

Incubation shall take place in the dark or in diffused light in an enclosure which is maintained at a constant temperature of 58 °C 2 °C and which is free from vapours inhibiting to micro-organisms.

6 Reagents

Use only reagents of recognised analytical grade.

Use as reference substance for a positive test control cellulose for thin-layer chromatography with a particle size of less than 20 μm.

7 Apparatus

7.1 General

Ensure that all glassware is thoroughly cleaned and, in particular, free from organic or toxic matter.

7.2 Composting vessels

Glass flasks or bottles allowing an even gas purge in an upwards direction.

NOTE A minimum volume of 2 l should fulfil the prerequisites mentioned in 8.2 and 8.3. For screening purpose, depending on the test material also smaller volumes may be used. If the weight loss of the test material is determined weigh each empty composting vessel.

7.3 Air circulation system

Capable of supplying each composting vessel with dry or humidified, if required carbon dioxide free, air at a pre-set flow rate which shall be high enough to provide truly aerobic conditions during the test (example see annex A).

7.4 Apparatus for the determination of carbon dioxide

Device for direct carbon dioxide determination or after complete absorption in a basic solution and determination of the dissolved inorganic carbon (DIC) (example see annex A). If the exhausted air is directly be measured e.g. with a continuous infrared analyser or a gas chromatograph an exact dosing or measurement of the gas flow is required.

7.5 Gas tight tubes

Which prevent diffusion of oxygen and carbon dioxide to connect the composting vessels with air production and carbon dioxide determination system.

7.6 pH meter

7.7 Analytical instruments

For determination of dry solids (at 105 °C), volatile solids (at 550 °C), total organic carbon (TOC) or elemental analysis of compact material and dissolved inorganic carbon (DIC) in the case of DIC measurement.

7.8 Balance

To measure the weight of test vessels including compost and test material which is normally in a range between 3 kg and 5 kg.

7.9 Analytical instruments (optionally)

For the optional determination of oxygen in the air, moisture, volatile fatty acids and total nitrogen (e.g. by the Kjeldahl method ISO 5663).

8 Procedure

8.1 Preparation of the inoculum

Take as inoculum well aerated compost from a properly operating aerobic composting plant.

NOTE 1 It is recommended that compost from a plant composting the organic fraction of municipal solid waste is used in order to get a sufficient range of micro-organisms which may be adapted to packaging materials. The age of the compost should be preferably between 2 and 4 months. If such a compost is not available also compost from plants treating green or yard waste or mixtures of green waste and municipal solid waste (bio-waste) may be used. Such a compost may be less adapted to packaging material.

The compost inoculum should be homogeneous and as free from larger inert materials such as glass, stones or metals. Remove them manually and then sieve the compost on a screen of about 0,5 cm to 1 cm.

NOTE 2 It is recommended to use compost with sufficient porosity to enable as aerobic conditions as possible. Addition of structural material such as small wood particles or persistent or poorly biodegradable inert material may avoid sticking together and clogging of the compost during the test.

Determine the total dry solids and the volatile solids content of the compost inoculum. The total dry solids content should be between 50 % and 55 % of the wet solids and the volatile solids more than about 15 % of the wet or 30 % of the dry solids. Adjust the water content, if necessary, before the compost is used by adding water or gently drying, e.g. by aerating the compost with dry air.

Prepare a mixture of 1 part of compost inoculum with 5 parts of de-ionised water. Mix by shaking and measure immediately the pH value which should be between 7,0 and 9,0.

NOTE 3 For further characterisation of the compost inoculum suitable parameters such as the content of total organic carbon, total nitrogen or fatty acids can be determined at the beginning and the end of the test (optionally).

Check the activity of the compost inoculum during the test by means of a biodegradable reference substance (see 6) and by measuring the carbon dioxide evolution in the blank vessels. The reference substance should be degraded at > 70 % at the end of the test (see 10). The inoculum in the blank control should produce between 50 and 150 mg of carbon dioxide per g of volatile solids over the first 10 days of the test (see 10). If the production of carbon dioxide is too high, the compost should be stabilised by aeration for several days before it is used in a new test. If the activity is too low an other compost inoculum should be used.

8.2 Preparation of the test material and the reference substance

Determine the total organic carbon (TOC) of the test material and the reference substance cellulose (see 6), using e.g. ISO 8245 and indicate it preferably as g TOC per g total dry solids. Alternatively, if the materials do not contain inorganic carbon it is possible to determine the carbon content by elemental analysis. The test material shall have sufficient organic carbon to yield carbon dioxide in an amount suitable for the determination. Normally a minimum of 50 g total dry solids containing 20 g TOC per vessel is required.

Determine the total dry solids of the test material and the volatile solids if the weight loss shall be determined (see Note hereafter).

NOTE The loss of test material and reference substance during the test can be determined optionally as an additional information. In the example given in annex C the amount of volatile solids of the test material is determined and compared with the amount at the end of the test.

Investigate the test material in the form of films, formed articles like tensile bars, granules or powder. The maximum surface area of a compact test material used should be about 2 cm 2 cm. In case the original test material is larger, reduce it in particle size.

8.3 Start-up of the test

Set up a sufficient number of composting vessels (7.2) so that the test includes at least the following:

a) 3 vessels for the test material ;

b) 3 vessels for the reference substance ;

c) 3 vessels for the blank control.

The amount of the test mixture, containing compost inoculum and test material, used in the test depends on the quality of the test material (see 8.2) and the size of the composting vessels (see 7.2). The relation between the dry weight of compost inoculum and dry weight of test material should be about 6 : 1. Be sure that the same amount of compost is in each vessel. Inert material if added is not considered in this relationship (see Note 2 in 8.1). About 3/4 of the volume of the compost vessel should be filled with the test mixture. Sufficient of headspace is required in order to provide enough space for manual shaking of the test mixture.

In a typical case prepare composting vessels which have a volume of about 3 l, weigh out 600 g of total dry solids of inoculum (7.2) and 100 g dry solids of the test material and mix well. The test mixture should have the same water content of about 50 % as the inoculum (see 8.1). It should feel somewhat sticky and have some free water available when gently pressed by hand. Adjust the moisture content of the mixture, if required, by adding water. Introduce the mixture into the vessels.

NOTE 1 It is recommended that the ratio between organic carbon and nitrogen (C/N-ratio) of the test mixtures is optimised so as to ensure a good composting process. The C/N ratio for the test mixture should be between 10 and 40, it may be adjusted with urea, if necessary. The organic carbon content can be calculated from the TOC of the compost inoculum and the test material. The total nitrogen content can be measured in a representative sample of the test mixture e.g. by using the Kjeldahl method ISO 5663.

Place the composting vessels in the test environment at 58 °C 2 °C (see 5) and initiate aeration using air which is free from carbon dioxide and normally water saturated. Both can be obtained e.g. by leading the air through wash-bottles filled with sodium hydroxide solution (see annex A).

NOTE 2 If the direct measurement of carbon dioxide concentration in the exhausted air is applied, also normal air can be used rather than air free from carbon dioxide. In this case measurement of the carbon dioxide concentration in the inlet and outlet of the test vessels is recommended. For correction subtract the inlet concentration from the outlet concentration. This is applicable because under the conditions of this test the concentration in the outlet is much higher than in the inlet.

Use flow rates which are sufficiently high to ensure that aerobic conditions are maintained during the test throughout the complete composting vessel. Check the air flow regularly at the outlets e.g. by using wash-bottles, assuring that no leaks are present in the complete system.

NOTE 3 A possibility to ensure aerobic conditions is the regular measurement of the oxygen concentration in the exhaust air of the compost vessels. The oxygen concentration should not drop below about 6 %. Oxygen levels should closely be controlled during the first week e.g. by measuring at least twice daily, afterwards the measurement frequency can be reduced. Adjust air flow rates as needed.

Handle the reference substance in the same way as the test material. The vessels for the blank control contain only compost inoculum with the same amount of total dry solids as in the vessels with test material.

8.4 Incubation period

Measure the amount of carbon dioxide released from each composting vessel at intermediate time intervals in the exhaust air either direct and intermittently using a gas chromatograph, a TOC or an infrared analyser or accumulative as dissolved inorganic carbon (DIC) after absorption in sodium hydroxide solution (see annex A). The frequency of measurements depends on the measuring system, the desired precision of the degradation curve and the biodegradation activity of the test mixtures. If momentaneous measurement is used measure during the biodegradation phase at least twice per day with time intervals of about 6 hours and later on in the plateau phase once per day. If an accumulative method is use measure DIC in the biodegradation phase once per day and during the plateau phase about twice per week.

Shake the composting vessels weekly to prevent extensive channelling and to provide uniform attack of the micro-organisms on the test material.

NOTE 1 For shaking a disconnection from the air production and the carbon dioxide determination system from the compost vessels is recommended.

Ensure that the humidity of the test mixtures in the compost vessels is neither too high nor too low by observing the test vessels. No free-standing water nor clumps of material should be present. Too dry conditions are typically revealed by the absence of condensate in the headspace of the composting vessel. Moisture can also be measured by suitable instruments (optionally). Moisture should be kept in a range of about 50 % (see 8.1). It can be influenced preferably by aerating with tempered humidified or dry air. A more drastic change can be obtained by adding water or by drainage via air inlet. The weekly shaking is helpful to get an even distribution of moisture. If adjustments are made monitor carbon dioxide release closely.

At the weekly shaking and at the end of the test record visual observations with regard to the appearance of the compost quality such as structure, moisture content, colour, fungal development, smell of the exhaust air of the test material.

The composting vessels are incubated for a period of normally 45 or more days at a constant temperature of 58 °C 2 °C which is representative for full-scale composting. The incubation time can be extended if a significant biodegradation of the test material is still observed up to the reaching of an almost constant plateau phase or shortened if the plateau phase is reached earlier.

NOTE 2 To fulfil the requirements for packaging recoverable through composting and biodegradation of a total test duration of 6 months should not be exceeded.

Measure in regular intervals the pH value as at the start of the test (see 8.1).

NOTE 3 If the pH is less than 7,0 0,2 biodegradation could be inhibited due to acidification of the compost by rapid degradation of a easily degradable test material. In this case measurement of the volatile fatty acids spectrum is recommended to check souring of the contents in the composting vessel. If more than 2 g of volatile fatty acids per kg of total dry solids has been formed, then the test should be regarded as invalid due to acidification and inhibition of the microbial activity. To prevent acidification add more compost to all vessels or repeat the test using e.g. less test material or more compost.

8.5 Termination of the test

If the loss of test material shall be determined (see Note in 8.2) weigh the compost vessels with the test mixtures. Take samples of the test mixtures from all vessels. Determine the total dry solids and the volatile solids if weight loss shall be calculated (see Note in 8.2 and annex C). Measure the pH value as at the start of the test (see 8.1).

Record visual observations with regard to the appearance of the test material to determine its disintegration.

NOTE It is recommended to perform further investigations with the remaining test material samples if possible and required such as weighing, measuring suitable physical properties, chemical analysis or photographing.

9 Calculation and expression of results

9.1 Calculation of the theoretical amount of carbon dioxide

Calculate the theoretical amount of carbon dioxide ($ThCO_2$) (in g per vessel) which can be produced by the added test material by equation (1) :

$$ThCO_2 = M_t \quad C_t \quad \frac{44}{12} \tag{1}$$

where

M_t total dry solids of the test material introduced in the composting vessels at the start of the test (g) ;

C_t relative amount of total organic carbon in the total dry solids of test material (g/g) ;

44 and 12 are the molar and atomic masses of carbon dioxide and carbon.

9.2 Calculation of the percentage of biodegradation

Calculate from the measured, accumulated values of the carbon dioxide production the percentage degree of biodegradation (D_t) of the test material for each measuring interval using equation (2) :

$$D_t = \frac{(CO_2)_t \quad (CO_2)_b}{ThCO_2} \quad 100 \qquad (2)$$

where

$(CO_2)_t$ is the accumulated amount of carbon dioxide released by each compost vessel (g/vessel) ;

$(CO_2)_b$ is the mean accumulated amount of carbon dioxide released by the blank controls (g/vessel) ;

$ThCO_2$ is the theoretical amount of carbon dioxide of the test material in the test vessels (g/vessel).

Calculate the average percentage if the deviations of the single measurements are less than 20 %. If this is not the case use the values of each compost vessel separately.

Use the same equation to calculate the degree of biodegradation of the reference substance.

9.3 Calculation of weight loss

An example for the optional calculation of weight loss based on volatile substances is given in annex C.

9.4 Expression of results

Compile tables containing the measured and calculated data of the test material, the reference substance and the blank controls for each day of measurement. Examples of form sheets are given in annex E.

Plot the accumulated amount of carbon dioxide released for each composting vessel with blank, test material and reference substance as a function of time (example see annex B). Plot a biodegradation curve in percent as a function of time for the test material and the reference substance (example see annex B). Use mean values if the the deviations of the single curves are less than 20 %. If this is not the case plot biodegradation curves for each composting vessel.

Read from the plateau phase of the biodegradation curve the mean degree of biodegradation and indicate it as final test result.

Describe qualitatively the disintegration of the test material in the case of a compact test material. Add further information such as photographs or measured physical values if available.

10 Validity of results

The test is considered as valid if :

a) the percentage of biodegradation for the reference substance is more than 70 % after 45 or more days ;

b) the deviation of the percentage of biodegradation for the reference substance in the different vessels is less than 20 absolute points at the end of the test ;

c) the compost inoculum in the blank control has produced after 10 days of incubation more than 50 and less than 150 mg of carbon dioxide per g of volatile solids (mean values).

11 Test Report

The test report shall provide all pertinent information, particularly the following :

a) testing institute and address;

b) reference to this European Standard ;

c) any information necessary to identify and describe the test material such as dry or volatile solids, organic carbon content, shape or visual appearance ;

d) any information necessary to identify and describe the reference substance and its organic carbon content ;

e) volume of compost vessels, the amount of compost inoculum, test material and reference substance and the main characteristics of the device used to determine carbon dioxide and the carbon analyser employed ;

f) any information on the compost inoculum and the analytical results of final compost residues such as source, age, date of collection, storage, handling, stabilisation, total dry solids, volatile solids, pH value of a suspension, and total nitrogen content or volatile fatty acids if appropriate ;

g) results obtained on carbon dioxide release and biodegradation percentage for each composting vessel and the averages in tabular form and as biodegradation curve and the final biodegradation degree for the test material and the reference substance and the activity of the inoculum (CO_2-production after 10 days in the blank control) ;

h) results on the visual observations of the compost inoculum and the test material during and at the end of the test such as moisture content, fungal development, structure, colour and smell and results of disintegration, eventual physical measurements or photographs ;

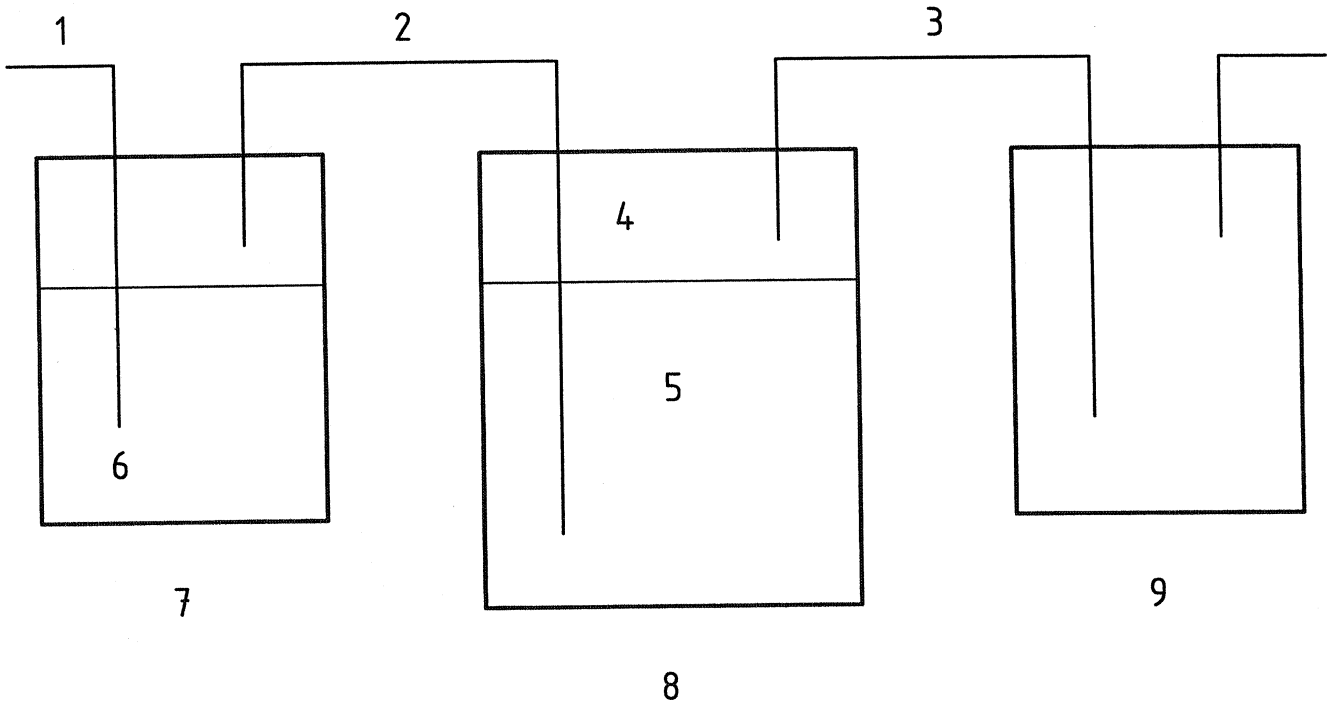
i) results on weight measurements of compost vessels at the start and the end of the test and the weight loss if performed ;

j) validity of test according to 10 or the reasons for rejection of any test results;

k) name of person responsible for the test and signature.

Annex A
(informative)

Principle of a test system with released carbon dioxide



Key

1 Air
2 CO_2 free air
3 Exhausted air
4 Headspace
5 Compost and test material
6 NaOH solution
7 CO_2 removal system
8 Composting vessel
9 CO_2 determination system

Figure A.1 - Principle of a test system with released carbon dioxide

Synthetic air free from carbon dioxide or compressed air is supplied at a constant low pressure. If compressed air is used the carbon dioxide is removed by passing through a suitable carbon dioxide absorption system. If a solution of sodium hydroxide in water is used also a humidification of the air is obtained. A second trap containing barium hydroxide solution can be used to indicate the absence of carbon dioxide.

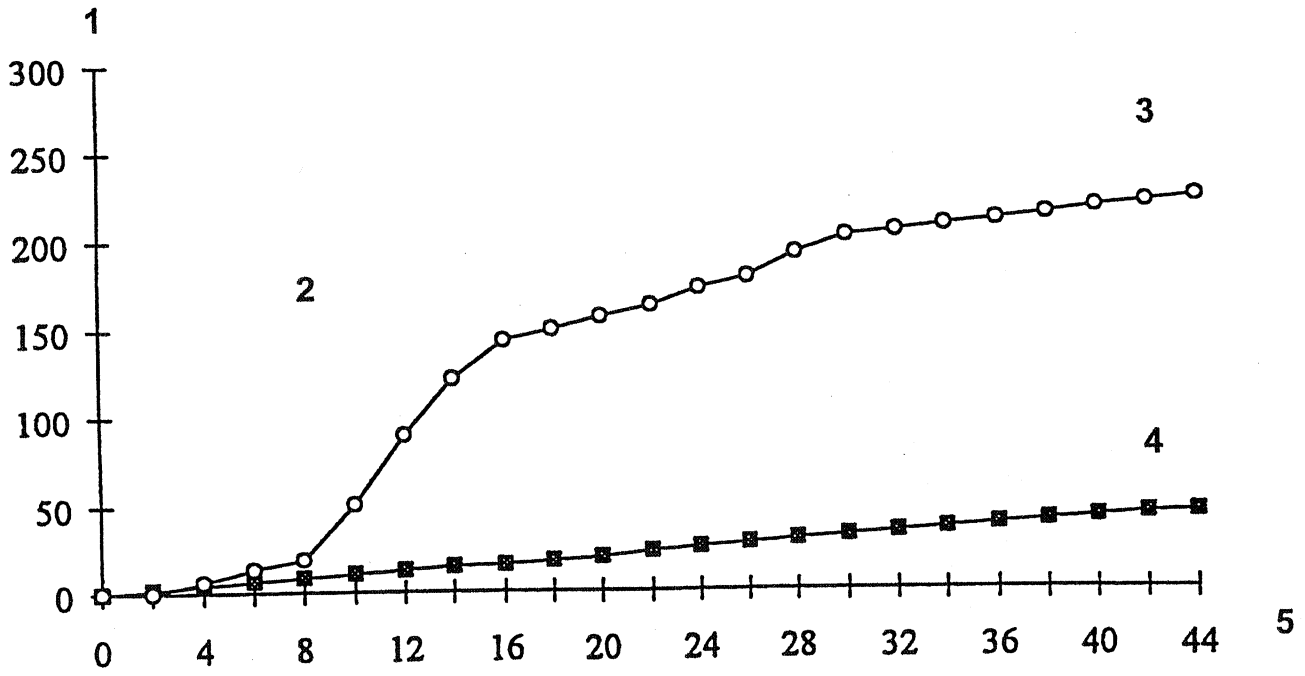
The air, used to aerate the test mixture in the composting vessels, should be introduced to the bottom of the vessels and distributed as even as possible. If biodegradation takes place, carbon dioxide is produced and released with the exhausted air.

The exhausted air can directly be measured e.g. with a continuous infrared analyser or a gas chromatograph. In this case an exact dosing or measurement of the gas flow is necessary. Depending on the measuring instrument it can be necessary to remove water from the air e.g. by cooling. If several composting vessels shall be connected with one measuring instrument a suitable gas switch may be required.

The exhaust air of each composting vessel can also be absorbed in a carbon dioxide trap containing e.g. a solution of 20 g/l sodium hydroxide in water and determined as dissolved inorganic carbon (DIC) e.g. in a suitable TOC analyser (using e.g. ISO 8245).

Annex B
(informative)

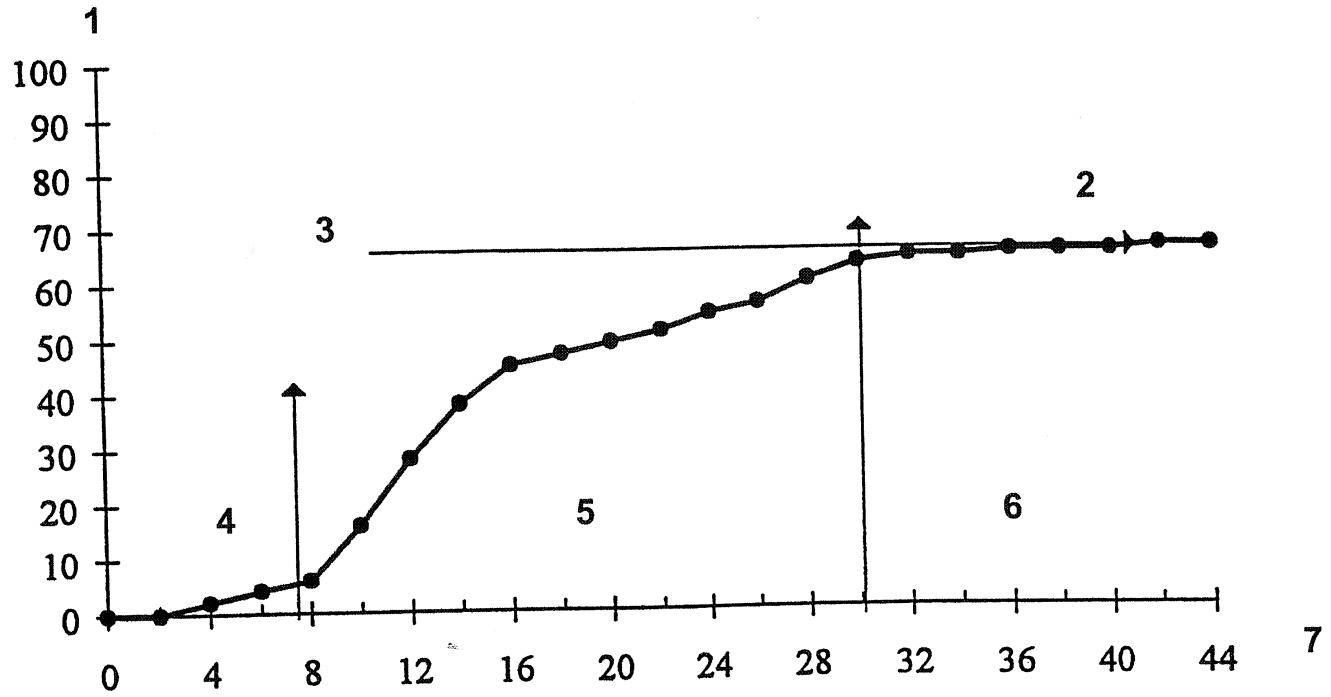
Example of a carbon dioxide release and a biodegradation curve



Key

1 CO_2 production g/vessels)
2 test material
3 CO_2 release curve
4 blank control
5 time (d)

Figure B.1 - Example of a carbon dioxide release



Key

1 biodegradation (%)
2 biodegradation curve
3 biodegradation degree 65 %
4 lag-phase
5 degradation phase
6 plateau phase
7 time (d)

Figure B.2 - Example of a biodegradation curve

Annex C
(informative)

Example of weight loss determination

C.1 General

The determination of the weight loss of the organic matter of a test material during the composting test may provide helpful quantitative information to support the biodegradation degree primarily measured as CO_2 evolution. In the following procedure a possibility is given of calculating this loss on the basis of measured volatile solids of the test material and the compost at the beginning and the end of the test.

Abbreviations used :

- com = compost,
- mat = test material,
- mix = mixture of compost and test material,
- ves = test vessel,
- wat = water.

Subscripts used :

- w = wet material,
- d = total dry solids,
- v = volatile solids,
- d/w = ratio of total dry solids to wet weight,
- v/d = ratio of volatile solids to total dry solids,
- deg = degraded test material,
- f = test vessel,
- s = start of the test,
- e = end of the test,
- y = empty test vessel (tare),
- a = addition control,
- add = added water,
- b = blank (compost only),
- m = mixture of compost and test material,
- $mean$ = mean value.

C.2 Weigh each empty test vessel (ves) to obtain the tare (ves_y) in g.

C.3 Determine the wet weight (mat_w), the total dry solids (mat_d) and the volatile solids (mat_v) of about 10 g of the test material (*mat*) and calculate the ratio of total dry solids to wet weight ($mat_{d/w}$) and of volatile solids to total dry solids ($mat_{v/d}$).

C.4 Use the amount of wet weight of the test material (mat_{wfs}) introduced into the test vessels at the start of the test to calculate the total amount of volatile solids (mat_{vfs}) in each test vessel according to equation (1) and express the result as g/vessel :

$$mat_{vfs} = mat_{wfs} \quad mat_{d/w} \quad mat_{v/d} \tag{C.1}$$

C.5 Determine before the start of the test the wet weight (com_{ws}), the total dry solids (com_{ds}) and the volatile solids (com_{vs}) of about 10 g of the compost used as inoculum (com_s) and calculate the ratio of total dry solids to wet weight ($com_{ds/ws}$) and of volatile solids to total dry solids ($com_{vs/ds}$).

C.6 Use the amount of wet weight of the compost (com_{wfs}) introduced into each test vessel at the start of the test to calculate the total amount of volatile solids of the compost (com_{vfs}) in each vessel according to equation (2) and express the result as g/vessel.

$$com_{vfs} = com_{wfs} \quad com_{ds/ws} \quad com_{vs/ds} \tag{C.2}$$

C.7 Weigh each test vessel with the test mixture of compost and test material and each blank control vessel containing compost only at the start (ves_{ms} and ves_{bs}) and the end (ves_{me} and ves_{be}) of the test and express it in g/vessel.

C.8 Check the correct addition of test material (mat_{wfs}), compost (com_{wfs}) and water added (wat_{add}) to the test vessels using equation (3) for the test mixtures (ves_{am}) and equation (4) for the blank controls (ves_{ab}) :

$$ves_{am} = ves_y + ves_{ms} = ves_y + com_{wfs} + mat_{wfs} + wat_{add} \tag{C.3}$$

$$ves_{ab} = ves_y + ves_{bs} = ves_y + com_{wfs} + wat_{add} \tag{C.4}$$

C.9 Calculate for each test vessel the amount of the mixture of compost and test material at the end of the test based on wet material (mix_{wfe}) using equation (5) and for each blank control vessel the amount of compost (com_{wfe}) using equation (6) and express the result in g/vessel :

$$mix_{wfe} = ves_{me} \quad ves_y \tag{C.5}$$

$$com_{wfe} = ves_{be} \quad ves_y \tag{C.6}$$

C.10 Take representative samples of about 10 g of the mixtures of compost and test material from each test vessel with test material at the end of the test. Determine the wet weight (mix_{we}), the total dry solids (mix_{de}) and the volatile solids (mix_{ve}) of the mixture (mix_e) and calculate the ratio of total dry solids to wet weight ($mix_{de/we}$) and of volatile solids to total dry solids ($mix_{ve/de}$). Use the same procedure to determine the ratio of total dry solids to wet weight ($com_{de/we}$) and of volatile solids to total dry solids ($com_{ve/de}$) in the blank controls of the compost.

C.11 Calculate the amount of volatile solids of each test mixture at the end of the test (mix_{vfe}) using equation (7) and of the compost in each blank vessel (com_{vfe}) using equation (8) and express the result in g/vessel :

$$mix_{vfe} = mix_{wfe} \quad mix_{de/we} \quad mix_{ve/de} \tag{C.7}$$

$$com_{vfe} = com_{wfe} \quad com_{de/we} \quad com_{ve/de} \tag{C.8}$$

C.12 Calculate the mean value of the amount of volatile solids of the compost in the blank vessels at the end of the test ($com_{vfe\ mean}$).

C.13 Calculate the amount of volatile solids of the test material in each test vessel at the end of the test (mat_{vfe}) using equation (9) and express it in g/vessel :

$$mat_{vfe} = mix_{vfe} \quad com_{vfe\ mean} \tag{9}$$

C.14 Calculate the amount of degraded test material based on volatile solids ($mat_{\deg}$) using equation (10) and express the result in g/vessel :

$$mat_{\deg} = mat_{vfs} \quad mat_{vfe} \tag{10}$$

C.15 Calculate the relative weight loss of the test material based on volatile solids for each test vessel using equation (11) and express this degree of biodegradation (D_v) in per cent :

$$D_v = \frac{mat_{\deg} \quad 100}{mat_{vfs}} \tag{11}$$

C.16 Calculate the mean value $D_{v\ mean}$ of the degree of biodegradation.

C.17 Determine in the same way the degree of biodegradation based on weight loss of the reference substance, if required.

Annex D
(informative)

Information - Ring test

To validate this method a ring test was performed. Test materials were paper and a copolymer of poly-β-hydroxybutyrate and poly-β-hydroxyvalerate. As a positive (degradable) reference substance cellulose with a particle size of less than 20 µm was used. The test results and the experience of the participants showed that the method is suitable and practicable and provides test results of high predictive value. The test results are published in

Pagga, U, Beimborn D.B., J. Boelens and B.De Wilde (1995)

Determination of the Aerobic Biodegradability of Polymeric Material in a Laboratory Controlled Composting Test. Chemosphere 31:4475-4487.

Annex E
(informative)

Example of form sheets

Controlled Aerobic Composting Test – Test Report

Test material : _____ Reference substance : _____

Origin of compost : _____ Age of compost : _____

Volume of test vessels : _____ Mode of CO_2 determination : _____

Test results

	Mean biodegradation based on CO_2 of $ThCO_2$	Mean biodegration based on weight of the organic matter (%)	Test duration (d)	Observations
Test material				
Reference substance				

Validity criteria

Mean $CO_2/ThCO_2$ of the reference substance after 45 days > 70 % O yes O no

Deviation of $CO_2/ThCO_2$ of the reference substance in the parallel vessels at end of test < 20 % O yes O no

Mean CO_2 production in the blank vessels after 10 days: in the range of 50-150 mg/g volatile solids O yes O no

Controlled Aerobic Composting Test – Biodegradation Based on CO_2-Evolution

Test material/reference substance : _____ TOC : _____ g/g $ThCO_2$: ____ g/ves

Date	Day	$(CO_2)_{b1}$ g/ves	$(CO_2)_{b2}$ g/ves	$(CO_2)_{b3}$ g/ves	$(CO_2)_{b\ mean}$ g/ves	$(CO_2)_{t1}$ g/ves	$(CO_2)_{t2}$ g/ves	$(CO_2)_{t3}$ g/ves	D_{t1} %	D_{t2} %	D_{t3} %	$D_{t\ mean}$ %

$(CO_2)_b$ = measured, accumulated CO_2 production of the blank control $(CO_2)_t$ = measured, accumulated CO_2 production of the test material or ref.substance

Calculations :

$$(CO_2)_{b\ mean} = \frac{(CO_2)_{b1} + (CO_2)_{b2} + (CO_2)_{b3}}{3} \qquad D_t = \frac{(CO_2)_t \quad (CO_2)_{b\ mean}}{ThCO_2} \qquad D_{t\ mean} = \frac{D_{t1} + D_{t2} + D_{t3}}{3} 14$$

ves = test vessel $mean$ = mean value

Controlled Aerobic Composting Test – Biodegradation Based on Weight Loss of the Organic Matter

○ Test material : _____ ○ Reference substance : _____

Test material (*mat*) :	mat_w (g)	mat_d (g)	mat_v (g)	$mat_{d/w}$	$mat_{v/d}$
Compost inoculum, start (com_s) :	com_{ws} (g)	com_{ds} (g)	com_{vs} (g)	$com_{ds/ws}$	$com_{vs/ds}$
Test mixture, end (mix_e) :	mix_{we} (g)	mix_{de} (g)	mix_{ve} (g)	$mix_{de/we}$	$Mix_{ve/de}$
Compost inoculum, end (com_e)	com_{we} (g)	com_{de} (g)	com_{ve} (g)	$mom_{de/we}$	$com_{ve/de}$

blank	com_{wfs} g/*ves*	com_{vfs} g/*ves*	wat_{add} g/*ves*	ves_{bs} g/*ves*	ves_{ab} g/*ves*	ves_y g/*ves*	ves_{be} g/*ves*	com_{wfe} g/*ves*	com_{vfe} g/*ves*	
com 1										
com 2										
com 3										
com_{mean}										

test material	mat_{wfs} g/*ves*	mat_{vfs} g/*ves*	wat_{add} g/*ves*	ves_{ms} g/*ves*	ves_{am} g/*ves*	ves_y g/*ves*	ves_{me} g/*ves*	mix_{wfe} g/*ves*	mix_{vfe} g/*ves*	mat_{vfe} g/*ves*	mat_{deg} g/*ves*	D_v %
mat 1												
mat 2												
mat 3												
mat_{mean}												

Abbreviations : *com* = compost, *mat* = test material, *mix* = mixture of compost and test material, *ves* = test vessel, *w* = water

Subscripts : *w* = wet material, *d* = total dry solids, *v* = volatile solids, *m* = mixture of compost and test material, *a* = addition control, *add* = added water, *deg* = degraded material, *s* = start of the test, *e* = end of the test, *y* = empty test vessel (tare)

Calculation of degradation degree based on volatile solids $D_v = mat_{\deg} \quad 100 / mat_{vfs}$

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ISO 8245:1999, *Water quality – Guidelines on the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*

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blank

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