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BRITISH STANDARD

BS EN
14045:2003

Packaging — Evaluation of the disintegration of packaging materials in practical oriented tests under defined composting conditions

The European Standard EN 14045:2003 has the status of a British Standard

ICS 13.030.99; 55.040

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National foreword

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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;

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

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 14 May 2003

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14045

March 2003

ICS 13.030.99; 55.040

English version

Packaging - Evaluation of the disintegration of packaging materials in practical oriented tests under defined composting conditions

Emballage - Evaluation de la désintégration des matériaux d'emballage lors d'essais à usage pratique dans des conditions de compostage définies

Verpackung - Bewertung der Desintegration von Verpackungsmaterialien in praxisorientierten Prüfungen unter definierten Kompostierungsbedingungen

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

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Foreword

This document (EN 14045:2003) 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of an identical text or by endorsement, at the latest by September 2003, and conflicting national standards shall be withdrawn at the latest by September 2003.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.

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Introduction

Organic recovery, which includes aerobic composting and anaerobic biogasification, of packaging in well operated, especially municipal or industrial biological waste treatment facilities are important alternatives for reducing and recycling packaging waste in a biological way. Using these technologies, the aims of the European Directive 94/62/EC on Packaging and Packaging Waste can be met.

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1 Scope

This European Standard is used to evaluate the disintegration of packaging materials in a pilot-scale aerobic composting test under defined conditions. Other methods should be used to measure the biodegradability of the packaging materials. Packaging materials are mixed with biowaste and spontaneously composted for 12 weeks in practical oriented composting conditions. At the end of the composting cycle the disintegration is measured by sieving of the compost and the calculation of a mass balance. The influence of the tested sample on the quality of the compost can be studied by using the compost obtained at the end of the composting process for further measurements such as chemical analyses and ecotoxicity tests.

Additionally this method can be used for visual perception and photographic documentation of the disintegration of packaging materials and for evaluating the effect of their addition on the composting process.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 13193: 2000, *Packaging – Packaging and the environment – Terminology.*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate.*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions and those given in EN 13193:2000 apply.

3.1
maturity of compost
stage of development of compost

3.2
total dry solids
amount of solids obtained by taking a known mass of test material or compost and drying at 105 °C to constant weight

3.3
volatile solids
amount of solids obtained by subtracting the residues of a known mass of test material or compost after incineration at about 550 °C from the total dry solids content of the same sample

4 Principle

The disintegration test under defined composting conditions on a pilot-scale level is a standardized composting process.

The test material is mixed in a precise concentration with fresh biowaste and introduced in a defined composting environment after which the biological composting process spontaneously starts. A natural ubiquitous microbial population will start the composting process and temperature increase will happen spontaneously. The composting mass is regularly turned over and mixed. The temperature, pH, moisture content and gas composition within the composting material are regularly monitored and have to fulfill certain requirements in order to ensure sufficient and appropriate microbial activity. The composting process is continued till fully stabilized compost is obtained (after 12 weeks).

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At the end of the composting process, the compost / test material mixture is sieved over 2 and 10 mm. Disintegration of the packaging material is evaluated after sieving procedure (6.1.3.2.). If possible a mass balance is calculated on the basis of wet and dry weight. The compost obtained at the end of the composting process can be used for further measurements such as chemical analyses and ecotoxicity tests.

5 Apparatus

5.1 Composting environment

5.1.1 General

Composting environment may be either a pilot-scale composting bin or nets buried in a pilot-scale composting bin. The volume of each bin shall be high enough for natural self-heating process to occur. Sufficient aeration shall be provided by an air providing system and air distribution should be even.

NOTE To standardize conditions for the test the composting trials can be run in bins, which are placed in a climatic chamber with a constant chamber temperature or in insulated bins.

If during the spontaneous thermophilic phase the compost mass reaches temperatures higher than 65 °C the microbial species diversity could be reduced. To restore a full array of thermophilic microbes, it is possible to re-incoculate the mass with mature compost (1 % of total initial biowaste mass) of recent origin (max. 3 months).

Reinoculationcan be done after the temperature peak at start. Additionally, it is possible to restore a full array of mesophilic microorganisms by reinoculating with mature compost (1% of total initial biowaste mass) of recent origin (max.3 months)after the thermophilic period in the test is over (or in other words : at the start of the mesophilic period in the test, e.g. after 4 or 6 weeks).

5.1.2 Composting bin

5.1.2.1 Volume and fitting

The bins shall :

¾ have a volume of minimum 140 l ;

¾ consist of a sturdy, heat resistant up to 120 °C and non-biodegradable material ;

¾ not effect the compost process and the quality of the compost.

5.1.2.2 Drainage

A draining system shall be constructed such that the compost will not flood.

5.1.3 Sample nets

The sample nets, if used, shall consist of mesh-like material with a mesh width of 1 mm made of non-degradable plastic, which is resistant to temperatures up to 120 °C.

The minimum volume shall be 20 l.

5.2 Apparatus for temperature measurement

Measuring range (0°C - 100°C) ± 1°C

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5.3 pH meter

5.4 Apparatus for oxygen analysis (e.g. gas chromatography)

5.5 Sieves with screens of 2 and 10 mm (according to ISO 3310-2)

6 Procedure of the test

6.1 Actions during incubation

6.1.1 Start-up of the test

6.1.1.1 Preparation of biowaste

The carrier matrix for the composting test consists of biowaste. It is important that for all test series a homogeneous biowaste of the same age and origin is used. The biowaste shall be reduced to particle sizes of maximum 50 mm (e.g. by shredding or sieving). Depending on the type of waste 10 % to 60 % of bulking agent of structurally stable components such as wood chips, bark, etc. with a size between 10 mm and 50 mm shall be added. In order to ensure a good composting process the biowaste shall fulfil the following criteria :

- C/N ratio of the fresh biowaste/bulking agent mixture shall be between 20 and 30 ;
- moisture content shall be above 50 % (*w/w*) not exceeding its water holding capacity;
- volatile solids content shall be above 50 % (*w/w*) ;
- pH shall be above 5.

NOTE 1 A representative sample of the biowaste should be mixed with demineralised water in a ratio of 1:5 (wet weight based), it should be left to it settle for 5 min and then the pH in the liquid phase should be measured.

NOTE 2 In order to correct the C/N-ratio, urea can be added.

The biowaste is obtained from the input material from a composting plant treating predominantly source separated organic municipal waste. Or, less satisfactorily, directly from households, grocery stores etc.

Alternatively a representative artificial (self-composed) biowaste can be used.

NOTE 3 As an example the following ingredients can be used :

- fresh mixed fruit and vegetable waste ;
- rabbit feed (seeds and dried extruded vegetables) ;
- matured compost ;
- urea (to adjust the C/N-ratio) ;
- plus sufficient water to attain a good moisture content ;
- plus bulking agent (e.g. woodchips or bark).

NOTE 4 After sieving at 50 mm, biowaste (especially urban biowaste) should not bring any packaging pieces which may be included in the disintegration results of the tested packaging.

6.1.1.2 Preparation of the test material

a) If the purpose of test is only measurement of disintegration, composting process and compost quality :

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the test material shall be tested in an identical form (e.g. shape, thickness) as for the intended final use. Large materials can be reduced in size to 10 cm x 10 cm.

NOTE 1 As option a coloring agent may be added to the test material for easier resegregation (e.g. TiO_2 or Fe_2O_3) ;

NOTE 2 Prior to start-up, the test material can be prewetted by adding water and mixing. Excess water is discarded again. The weight of water added should be noted.

b) If the purpose of test includes production of compost for ecotoxicity tests :

For ecotoxicity tests, additional to a), test material shall be added under the form of fine powder or granulate. The fine form shall prevent the mixture of biowaste/test material of getting too bulky.

NOTE It is recommended to use powder of test material with diameters < 500 μm.

6.1.1.3 Number of test series

The test shall include at least :

a) 2 series for the biowaste control and 2 series for the test material disintegration and compost analysis;

b) 2 series for the test material (b1) disintegration and compost analysis plus optionally (b2) ecotoxicity tests ;

c) 2 separate series only for ecotoxicity tests (optional to (b2)).

NOTE Some materials may not be suitable for combined disintegration and ecotoxicity test series. Their powder or granulate form (9 %) may clot, stick or melt to the test material (1 %), which would interfere with the disintegration process and results. Therefore, if such a risk is identified, 2 separate test series should be performed.

6.1.1.4 Mixing ratio of biowaste and test material

Each composting test series shall be conducted with roughly the same amount of biowaste (wet weight min. 60 kg). In order to get the same mass in each bin, the addition of test material to the biowaste in the test bins shall be followed by the addition of a same mass of biowaste in the blank control bins. Water content of each bin shall be adjusted after addition of the test material or biowaste to meet the requirements of 6.1.1.1. The amount of test material to be added shall be as follows :

a) For measurement of disintegration and compost analysis only :

1 % on wet weight basis under final form of test material ;

b) For measurement of disintegration, compost analysis and ecotoxicity tests in one test series :

1% on wet weight basis under final form of test material plus

9 % on wet weight basis as powder or granulate ;

c) For ecotoxicity tests in separate test series :

As option :

1) 1% on wet weight basis under final form of test material plus

9 % on wet weight basis as powder or granulate ;

or :

2) 10 % on wet weight basis as powder or granulate.

6.1.1.5 Preparation of samples

The biowaste used shall be a homogeneous and representative sample (randomly taken).

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Each test series shall be prepared and filled separately. For the series with test material, biowaste and test material are weighed precisely and mixed before introduction in the bin.

If sample nets are used in the composting bins the input of biowaste of each sample shall be put into a container, weighed and subsequently mixed thoroughly with the packaging which shall be added in the above mentioned ratio (see 6.1.1.4). Sample nets which hold the biowaste/packaging mixture shall be tied up tightly with a non-degradable, heat resistant plastic string and marked appropriately.

6.1.2 Turning

The biowaste mixture has to be turned regularly to break down clumps and to remix water, microorganisms and substrate. This mixing is done weekly during the first 4 weeks and every 2nd week until the end of the test. If sample nets are used, the nets have to be opened and the content is mixed.

6.1.3 Termination of the test

6.1.3.1 Duration

The duration of the incubation is 12 weeks.

6.1.3.2 Sieving procedure

The whole compost obtained from each composting test series at the end of the test shall be screened through a 10 mm sieve. The overflow has to be carefully searched for large packaging particles (clods have to be broken up) while the sieved material < 10 mm is partitioned further by standard sieves into two portions, comprising the particle sizes 2 - 10 mm and < 2 mm. The particles or pieces which do not differ from the compost for colour, structure, dimension, moisture feeling, brightness/gloss are considered to be compost.

If using :

- the whole bins for the test from each of these fractions a homogeneous sample (preferably the whole amount but at least 50 %) shall be taken for further segregation and analysis ;

- nets in bins for the test the whole content of the nets has to be sieved.

For each fraction all extracted packaging particles are put into a 2 mm sieve and carefully cleaned, if possible under a running tap. Cleaned packaging particles shall be dried (at 105 °C, at 40 °C for test materials with melting temperatures under 105 °C, until constant weight is reached), subsequently weighed and analysed for organic matter (volatile solids). Finally the registered dry weight (in g) has to be brought into relation to the dry weight (in g) of packaging added at the beginning of the trial.

The sieving scheme is given in Table 1.

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Table 1 — Sieving Scheme

Compost (at end of pilot scale test)	
Sieving > 10 mm	Particles > 10 mm: Segregation and determination of the dry mass of residual test material
Fraction < 10 mm	10 mm > Particles > 0 mm: Gently mixed: Separation of a part of the compost for chemical analysis according to 6.2.3.1 Option: Ecotoxicity testing
Sieving > 2 mm	10 mm > Particles > 2 mm: Segregation and determination of the dry mass of test material
Fraction < 2 mm	Use for optional analysis

NOTE Extracting possible residual packaging particles from the 2-10 mm fraction may be facilitated by dividing it into fractions with a narrower particle size distribution (e.g. 2-5 mm and 5-10 mm).

A large sample should also be taken from the "< 10 mm" fraction for compost quality analyses and eventual ecotoxicity tests.

The whole final compost sample is sieved through a 10 mm grid, the fraction is gently mixed and divided into two parts. One part is used for the segregation of material residues > 2 mm. The other part is used for chemical analysis (6.2.3.1).

If ecotoxicity tests should be applied, they are also done from the sieving fraction < 10 mm.

6.1.3.3 Visual observation (optional)

If the test material does not disintegrate completely after 12 weeks it is strongly recommended to carry out a visual observation as described below.

A visual assessment of the criteria described below is carried out at the beginning of the test and also during the entire trial period whenever the test material is turned. Initially :

¾ distribution of particle size of remaining packaging particles has to be estimated and ;

¾ signs of microbial colonisation of the packaging (fungal hyphae, bacterial growth) have to be described and photographed.

At least 10 packaging particles shall be selected with the intention to provide an impression of all visible degradation phenomena, ranging from little decomposition to severe degradation of the packaging particles.

The selected particles shall be carefully cleaned with water and evaluated visually for the following criteria :

¾ consistency and thickness of the material ;

¾ discolouring ;

¾ erosion of the material (holes, tunnels, etc.) and signs of local disintegration ;

¾ ease of discovery.

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Results of each assessment relevant for the test shall be documented in writing and by photographs.

6.2 Analysis and process control

6.2.1 Start-up of the test

a) biowaste :

At the start of the test the biowaste and separately the bulking agent shall be analysed (see 6.1.1.1) . The composition of the waste (share of garden and kitchen waste) shall be characterised and documented ;

b) test material :

The test material shall be described by reporting the type of material, volume to surface ratio or thickness, the C/N ratio, the moisture content and the volatile solids.

6.2.2 During testing

6.2.2.1 Aeration

The aeration is controlled in such a way that the composting process can proceed smoothly.

Oxygen concentration inside the composting material shall be above 10 % and shall be measured regularly in the composting material or in the exhaust air (minimum every working day during the first month of the test and minimum once a week afterwards). If oxygen concentration decreases below 10 %, the biowaste shall be aerated with flowrates of maximum 15 l/kg dry matter and hour.

NOTE The air flow also can be used to control the temperature and the moisture level of the composting bins contents.

The air flows applied to ventilate the bin should be in line with those applied in a real composting plant. If, for practical reasons, higher flows are to be used then an estimation of the ammonia removed by the air flow can be performed and this amount restored by addition of additives (e.g. urea).

6.2.2.2 Moisture content and pH

After turning a sample of each test series shall be taken for measurement of pH and moisture content. If the moisture content is too low for a good composting process to occur (< 40 % (w/w)) water shall be added.

6.2.2.3 Temperature

Temperature in the middle of the composting material shall be measured at least once per working day.

6.2.2.4 Visual observation (optional)

During turning the mixture and the test material are also inspected visually (see 6.1.3.3) with regard to structure, moisture, fungal development and general appearance.

6.2.3 Termination of the test

6.2.3.1 Compost

See ISO 11261, ISO/DIS 14256-1 and ISO 11465.

The characteristics of the compost obtained in the presence of the test material shall be compared to the results for the biowaste control bins.

The wet weight of the total compost before sieving shall be exactly determined.

NOTE The composting bins content can be cooled to ambient temperatures before it is weighed and sieved, otherwise too much moisture may evaporate between weighing and sampling for the determination of the moisture content.

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A homogeneous sample of the "< 10 mm" fraction shall be analyzed for dry matter, volatile solids, pH, NH_4-N, NO_x-N, Kj-N content, Volatile Fatty Acids (VFA) and maturity. The results of these analyses are used to describe the quality of the compost produced. If applicable the "< 10 mm" fraction shall also be used for further ecotoxicity testing.

NOTE The assignment of the maturity of compost takes place on the basis of the temperature maximum (T_{max}).

Rottegrad I : T_{max} = 61 °C – 70 °C (fresh biowaste)

Rottegrad II : T_{max} = 51 °C – 60 °C

Rottegrad III : T_{max} = 41 °C – 50 °C

Rottegrad IV : T_{max} = 31 °C – 40 °C

Rottegrad V : T_{max} = 20 °C – 30 °C (mature compost)

(see : Methods Book for the Analysis of Compost)

6.2.3.2 Test material

The dry weight of the complete fraction > 2mm shall be determined.

7 Calculation

Disintegration of test material (in %) $D = \frac{m_{(1)} \times R \quad m(2)}{m_{(1)} \times R} \quad 100$

with :

$m_{(1)}$ = dry weight of test substance input ;

$m(2)$ = dry weight of retrievable test substance > 2 mm;

$$R = \frac{compost\ screened}{total\ compost} (w/w)$$

NOTE The disintegration of the test material (on dry weight basis) > 2 mm should be calculated on the basis of the results of the analyses and weight determinations of the various sieving fractions. The degree of disintegration is found by adding up the weight of the packaging particles of all portions selected from the particle size categories (see 6.1.4.1) and the subsequent calculation of the ratio between the amount of recovered packaging at the end of the test and added packaging at the test beginning.

8 Validity of the test

The test is considered valid if :

a) for all bins or nets in bins with biowaste and the mixture of biowaste and test material the temperature during composting is below + 75°C

b) for all control bins or nets in bins with biowaste only

1) the temperature is above + 60°C for at least 1 week;

2) the temperature is above + 40°C for at least 4 consecutive weeks;

3) the pH should increase to above 7.0 at the end of the test, but in any case it shall not fall down below 5 during the test;

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4) to ensure a completion of a normal composting process, the compost from biowaste only is mature after 12 weeks, i.e. Rottegrad IV - V and the content of volatile fatty acids (VFA) < 500 mg/kg (wet weight).

9 Test Report

The test report shall provide all relevant 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) biowaste source and results of chemical analyses at start of incubation ;

e) precise description of the set-up for composting (bins, nets in bins for pilot-scale test) ;

f) volume of composting test series, the amount of biowaste and test material ;

g) results obtained for disintegration. Residual amount of test material and degree of disintegration (see 7) after composting and sieving ;

h) results characterising the composting process :

- ¾ representation of temperature profile ;
- ¾ results on analyses of :
  - ¾ pH and moisture content ;
  - ¾ oxygen concentration ;

i) results of compost analyses at end of composting cycle ;

j) results on the sensory observations of the biowaste compost and the test material during and at the end of the test such as, fungal development, structure, colour and smell, eventual photographic documentation (optional) ;

k) reasons for rejection of any test results;

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

The test report should also mention all the corrective measures taken, such as re-inoculation (5.1) addition of urea (6.1.1.1), or air flow changes (6.2.2.1).

Bibliography

- EN 13432:2000, *Packaging - Requirements for packaging recoverable through composting and biodegradation - Test scheme and evaluation criteria for the final acceptance of packaging.*
- ISO 10634:1995, *Water quality - Guidance for the preparation and treatment of poorly water-soluble organic compounds for the subsequent evaluation of their biodegradability in an aqueous medium.*
- ISO 11734:1995, *Water quality - Evaluation of the ultimate anaerobic biodegradability of organic compounds in digested sludge - Method by measurement of the biogas production.*
- ISO 14851, *Evaluation of the ultimate aerobic biodegradability of plastic materials in an aqueous medium - Method by determining the oxygen demand in a closed respirometer.*
- ISO 14852, *Evaluation of the ultimate aerobic biodegradability of plastic materials in an aqueous medium - Method by analyses of released carbon dioxide.*
- ISO 14855, *Evaluation of the ultimate aerobic biodegradability and disintegration of plastics under controlled composting conditions - Method by analysis of released carbon dioxide.*
- ISO 14855 DAM 1, *Determination of the ultimate aerobic biodegradability and disintegration of plastic materials under controlled composting conditions - Method by analysis of evolved carbon dioxide; Amendment 1: Use of a mineral bed instead mature compost.*
- ISO/TR 15462, *Water quality - Selection of tests for biodegradability.*
- ISO 10390, *Measurement of pH.*
- *ISO 11261, Soil quality — Determination of total nitrogen — Modified Kjeldahl method.*
- ISO 11465, *Measurement of dry and volatile solids.*
- *ISO/DIS 14256-1, Soil quality — Determination of nitrate, nitrite and ammonium in field moist soils by extraction with potassium chloride solution — Part 1: Manual method.*
- Methods Book for the Analysis of Compost including :

¾ measurement of pH and moisture content ;

¾ measurement of dry and volatile solids ;

¾ measurement of compost maturity (Rottegrad).

Publisher : Bundesgütegemeinschaft Kompost e.V., Hauptstraße 305, 51143 Cologne ((New Address))

Afvalstoffenanalysen - Compendium. Publisher: OVAM, Kan.De deckerstraat 22 - 26, B-2800 Mechelen - Belgium

Determination of VFA (Bepaling van vluchtige organische zuren).

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