BS ISO 16929:2013

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Plastics — Determination of the degree of disintegration of plastic materials under defined composting conditions in a pilot-scale test

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National foreword

This British Standard is the UK implementation of ISO 16929:2013. It supersedes BS ISO 16929:2002 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics.

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

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Plastics — Determination of the degree of disintegration of plastic materials under defined composting conditions in a pilot-scale test

Plastiques — Détermination du degré de désintégration des matériaux plastiques dans des conditions de compostage définies lors d'un essai à échelle pilote

Reference number
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Foreword

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

ISO 16929 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 16929:2002), of which it constitutes a minor revision.

Introduction

The biological treatment of biodegradable plastic materials includes aerobic composting in well-operated, municipal or industrial biological waste treatment facilities. Determining the degree of disintegration of plastic materials in a pilot-scale plant is an important step within a test scheme to evaluate the compostability of such materials.

INTERNATIONAL STANDARD ISO 16929:2013(E)

Plastics — Determination of the degree of disintegration of plastic materials under defined composting conditions in a pilot-scale test

WARNING — Compost can contain potentially pathogenic organisms. Therefore, appropriate precautions should be taken when handling it.

1 Scope

This International Standard is used to determine the degree of disintegration of plastic materials in a pilot-scale aerobic composting test under defined conditions. It forms part of an overall scheme for the evaluation of the compostability of plastics as outlined in ISO 17088. The test method laid down in this International Standard can also be used to determine the influence of the test material on the composting process and the quality of the compost obtained. It cannot be used to determine the aerobic biodegradability of a test material. Other methods are available for this (e.g. see ISO 14851, ISO 14852 or ISO 14855-1 and ISO 14855-2.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 5663, *Water quality — Determination of Kjeldahl nitrogen — Method after mineralization with selenium*

ISO 7150-1, *Water quality — Determination of ammonium — Part 1: Manual spectrometric method*

ISO 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

ISO 10390, *Soil quality — Determination of pH*

ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

3 Terms and definitions

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

3.1
biological treatability
potential of a material to be aerobically composted or anaerobically biogasified

3.2
degradation
irreversible process leading to a significant change in the structure of a material, typically characterized by a loss of properties (e.g. integrity, molecular mass or structure, mechanical strength) and/or by fragmentation, affected by environmental conditions, proceeding over a period of time and comprising one or more steps

3.3
biodegradation
degradation caused by biological activity especially by enzymatic action leading to a significant change in the chemical structure of a material

3.4
disintegration
physical breakdown of a material into very small fragments

3.5
compost
organic soil conditioner obtained by biodegradation of a mixture principally consisting of various vegetable residues, occasionally with other organic material, and having a limited mineral content

3.6
composting
aerobic process designed to produce compost

3.7
compostability
property of a material to be biodegraded in a composting process

Note 1 to entry: To claim compostability, it shall have been demonstrated that a material can be biodegraded and disintegrated in a composting system (as can be shown by standard test methods) and completes its biodegradation during the end-use of the compost. The compost shall meet the relevant quality criteria. Quality criteria include low content of regulated metals, no ecotoxicity, no obviously distinguishable residues.

3.8
maturity of compost
assignment of the maturity of a compost based on the measurement of the maximum temperature in a self-heating test using Dewar vessels

Note 1 to entry: It is expressed in terms of the so-called “Rottegrad” (see 6.2.3.1).

3.9
total dry solids
amount of solids obtained by taking a known volume of test material or compost and drying at about 105 °C to constant mass

3.10
volatile solids
amount of solids obtained by subtracting the residues of a known volume of test material or compost after incineration at about 550 °C from the total dry solids content of the same sample

Note 1 to entry: The volatile solids content is an indication of the amount of organic matter present.

4 Principle

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

The test material is mixed with fresh biowaste in a precise concentration and introduced into a defined composting environment. A natural ubiquitous microbial population starts the composting process spontaneously and the temperature increases. The composting mass is regularly turned over and mixed. Temperature, pH-value, moisture content and gas composition are regularly monitored. They have to fulfil certain requirements to ensure sufficient and appropriate microbial activity. The composting process is continued until a fully stabilized compost is obtained. This is usually the case after 12 weeks.

The compost is visually observed at regular time intervals to detect any adverse effect of the test material on the composting process. At the end of the test, the maturity of the compost is determined and the mixture of compost and test material is sieved through 2 mm and 10 mm mesh sieves. The

disintegration of the test material is evaluated on the basis of the total dry solids by comparing the fraction of test material retained by the 2 mm sieve and the amount tested. The compost obtained at the end of the composting process may be used for further measurements, such as chemical analyses and ecotoxicity tests.

5 Apparatus

5.1 Composting environment

5.1.1 General

The 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 to occur. Sufficient and even aeration shall be provided by an appropriate air supply system.

NOTE 1 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.

NOTE 2 If during the spontaneous thermophilic phase the compost reaches temperatures higher than 65 °C, the diversity of microbial species can be reduced. To restore a full array of thermophilic bacteria, the compost can be re-inoculated with mature compost (about 1 % of the total initial biowaste mass) of recent origin (maximum 3 months old).

5.1.2 Composting bins

5.1.2.1 Volume and material

The bins shall:

— have a minimum volume of 35 l;

— consist of a sturdy, heat-resistant and non-biodegradable material;

— not affect the composting process or the quality of the compost.

5.1.2.2 Drainage

The drainage shall consist of a layer of drains with a thickness of at least 5 cm at the bottom of the bins.

5.1.3 Sample nets

The sample nets, if used, shall consist of mesh-like material with a mesh size 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

5.3 pH-meter

5.4 Apparatus for oxygen measurement

5.5 Sieves

Use sieves of suitable shape with screens of 2 mm and 10 mm mesh (as specified for instance in ISO 3310-2).

6 Test procedure

6.1 Actions before and during incubation

6.1.1 Start-up of the test

6.1.1.1 Preparation of biowaste

As a carrier matrix, use biowaste, if possible from the input material of a composting plant treating predominantly municipal waste, or, less satisfactorily, biowaste directly from households or grocery stores for example.

NOTE Alternatively, a representative artificial biowaste with, for example, the following ingredients may be used:

— freshly mixed fruit and vegetable waste;

— rabbit feed (seeds and extruded dried-vegetable pellets);

— mature compost;

— sufficient water to attain a good moisture content;

— a bulking agent (e.g. wood chips or bark).

It is important that for all test series a homogeneous biowaste of the same age and origin is used. Reduce the biowaste to particle sizes of maximum 50 mm, for example by shredding or sieving. Depending on the type of waste, add about 10 % to 60 % of bulking agent (structurally stable components such as wood chips or bark with a particle size between 10 mm and 50 mm).

To ensure a good composting process, the biowaste shall meet the following criteria:

— the C:N ratio of the fresh biowaste/bulking agent mixture shall be between 20 and 30;

— the moisture content shall be above 50 % mass fraction, with no free water present;

— the volatile solids content of the total dry solids shall be above 50 % mass fraction;

— the pH shall be above 5.

Adjust the C:N ratio with urea, if required.

6.1.1.2 Preparation of the test material

a) If the purpose of the test is to measure the degree of disintegration of the test material and to determine the effects on the composting process and the compost quality, use the test material in an identical form (e.g. shape, thickness) as for the intended final use. Reduce large materials in size to 10 cm × 10 cm for films and 5 cm × 5 cm for other products.

   NOTE As an option, a colouring agent (e.g. TiO_2 or Fe_2O_3) can be added to the test material for easier re-isolation.

b) If the (optional) purpose of the test includes production of compost for ecotoxicity tests, use in addition to a) the test material in the form of fine powder or granules. The fine form is intended to prevent the mixture of biowaste and test material from getting too bulky.

   It is recommended that the test material be used as a powder with a particle size <500 µm.

6.1.1.3 Number of test series

Provide a sufficient number of composting test series, at least:

a) two series for the biowaste control;

b) two series for the test material for the purpose of 6.1.1.2 a);

c) an optional two series for the test material for the purpose of 6.1.1.2 b).

6.1.1.4 Mixing ratio of biowaste and test material

Conduct each composting test series with roughly the same amount of biowaste (wet mass minimum 60 kg). The amount of test material to be added shall be as follows:

a) For measurement of the degree of disintegration and compost analysis [see 6.1.1.2 a)]:

   — 1 % on wet mass basis of test material in its final form.

b) For measurement of the degree disintegration, compost analysis and ecotoxicity tests in one test series [see 6.1.1.2 a) and b)]:

   — 1 % on wet mass basis of test material in its final form plus;

   — 9 % on wet mass basis of test material as powder or granules.

c) For the optional ecotoxicity tests in separate test series [see 6.1.1.2 b)]:

   — either 1 % on wet mass basis of test material in its final form plus 9 % on wet mass basis of test material as powder or granules;

   — or 10 % on wet mass basis as powder or granulate.

6.1.1.5 Preparation of samples

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

Prepare each test series separately. For all series with test material, weigh biowaste and test material precisely and mix well before introducing into the bin.

If sample nets are used in the composting bins, put the input of biowaste from each sample into a container, weigh and subsequently mix thoroughly with the test material which shall be added in the ratios specified in 6.1.1.4. Place the mixture of biowaste and test material in the nets, tie the nets up tightly with non-biodegradable and heat-resistant plastic string and mark them appropriately.

6.1.2 Turning

Turn the biowaste mixture regularly to break down lumps and to remix water, microorganisms and substrate. Do this weekly during the first 4 weeks and then every 2 weeks until the end of the test. If sample nets are used, open the nets and mix the contents.

6.1.3 Termination of the test

6.1.3.1 Duration

The duration of the incubation shall be 12 weeks.

6.1.3.2 Sieving procedure

Screen the compost obtained from each composting test series for residual particles of the test material, as follows.

When using bins for the test, take from each bin a homogeneous sample, preferably the whole content of the bin but at least 50 %. When using nets in bins, use the whole content of the net.

Sieve each of the samples through a standard 10 mm sieve, searching the overflow carefully for large lumps of (sticky) compost in which pieces of test material remain and breaking these up to crumbly particles, which have a more typical particle size distribution for compost and are easier to sieve. Separate the sieved material further by sieving through a standard 2 mm sieve. From the 2 mm to 10 mm fraction thus obtained, pick out all particles of the test material, place them on a separate 2 mm sieve and clean carefully, if possible by washing under a running tap. Dry the cleaned particles at 105 °C (or at 40 °C for test materials with melting temperatures below 105 °C) until constant mass is reached. From the mass of total dry solids thus obtained, calculate the degree of disintegration as indicated in Clause 7. In addition, measure the amount of organic matter present by determining the volatile-matter content.

NOTE Picking out the particles of test material can be facilitated by dividing the 2 mm to 10 mm fraction into fractions with a narrower particle-size distribution (e.g. 2 mm to 5 mm and 5 mm to 10 mm). Losses of particles of test material during the cleaning process can be avoided by using an additional 1 mm sieve under the 2 mm sieve. All particles <2 mm in size are, however, usually neglected.

It is recommended that samples be taken from the compost left after picking out the test material for compost quality analyses and ecotoxicity tests.

6.1.3.3 Visual observations (optional)

Carry out a visual assessment at least at the beginning and the end of the test and, if possible, whenever the test material is turned. Estimate the particle size distribution of the test material and record signs of microbial colonization (e.g. fungal hyphae, bacterial growth) on the test material particles.

For this, select at least 10 particles providing an impression of all visible degradation phenomena, ranging from little decomposition to extensive degradation of the test material. Clean the selected particles carefully with water and evaluate visually with regard to the following:

— consistency and compactness of the material;

— decolourization;

— signs of local disintegration (e.g. the presence of holes);

— how easy (or difficult) it was to pick out the test material.

Return the selected particles to the composting mixture. Note and document the results of each assessment in writing and by means of photographs.

A visual assessment is strongly recommended if the test material does not disintegrate completely after 12 weeks.

6.2 Analysis and process control

6.2.1 Start-up of the test

a) Biowaste

At the start of the test, analyse the biowaste and, separately, the bulking agent (see 6.1.1.1). Characterize and document the composition of the waste (e.g. the proportions of garden and kitchen waste).

b) Test material

Describe the test material (see 6.1.1.2) by reporting, for example, the type of material, the volume to surface area ratio or thickness, the ratio of carbon to total nitrogen (C:N), the moisture content, the total dry solids and the content of volatile solids.

6.2.2 During the test

6.2.2.1 Aeration

Control the aeration in such a way that the composting process can proceed smoothly. Measure regularly the oxygen concentration in the composting material or in the exhaust air, at least every working day during the first month of the test and once a week afterwards. The oxygen concentration inside the composting material shall be above 10 %. If the oxygen concentration decreases to below 10 %, aerate the biowaste, using air flow rates of no more than 15 litres per kilogram of total dry solids per hour.

NOTE The air flow can be used to control the temperature and the moisture level of the composting bins. The air flow used to ventilate the bins is preferably in line with that used in a real composting plant. If, for practical reasons, a higher flow is used, the ammonia removed by the air flow can be estimated. This amount can be restored by the addition of, for example, urea.

6.2.2.2 Moisture content and pH

After turning, take a sample of each test series to measure the pH and moisture content. If the moisture content is too low for a good composting process to occur (<40 % mass fraction), add water.

6.2.2.3 Temperature

Measure the temperature in the middle of the composting material at least once per working day.

6.2.2.4 Visual observations (optional)

Inspect visually the mixture and the test material during turning with regard to structure, moisture, fungal development and general appearance (see 6.1.3.3).

6.2.3 Termination of the test

6.2.3.1 Compost

Determine the wet mass of the total compost before sieving.

It is recommended that the contents of the composting bins be cooled to ambient temperature before weighing and sieving, otherwise too much moisture can evaporate between weighing and sampling for the determination of the moisture content.

Analyse a homogeneous sample of the <10 mm fraction for total dry solids, volatile solids (e.g. use ISO 11465), pH (e.g. use ISO 10390), ammonium nitrogen (e.g. use ISO 7150-1), nitrite and nitrate nitrogen (e.g. use ISO 10304-1) and total nitrogen (e.g. use ISO 5663). Use for the determination of the maturity of the compost a suitable method such as determination of volatile fatty acids (e.g. by ion chromatography of an aqueous extract) and/or the “Rottegrad”.

NOTE The assignment of a maturity level to compost using the “Rottegrad” scale takes place on the basis of the determination of the maximum temperature (T_{max}) in a self-heating test using Dewar vessels. The measured maximum temperature after about 2 to 5 days is used to classify the compost as follows:

— Rottegrad I: T_{max} > 60 °C (fresh biowaste)

— Rottegrad II: T_{max} 50,1 °C to 60 °C

— Rottegrad III: T_{max} 40,1 °C to 50 °C

— Rottegrad IV: T_{max} 30,1 °C to 40 °C

— Rottegrad V: T_{max}: ≤ 30 °C (mature compost)

For details of the method, see Reference.[6]

Use the results of these analyses to describe the quality of the compost produced. The results may be compared to a known compost of good quality.

If required, use the <10 mm fraction for further ecotoxicity testing.

6.2.3.2 Test material

Determine the total dry solids of the complete >2 mm fraction.

7 Calculation

Add up the mass of the retrieved test material particles of all selected >2 mm fractions (see 6.1.3.2) and compare it to the mass of the initial test material input (see 6.1.1.2). Calculate the degree of disintegration of the test material, D_i, on the basis of the respective total dry solids from Formula (1):

$$D_i = \frac{m_1 - m_2}{m_1} \times 100 \quad (1)$$

where

D_i is the degree of disintegration of the test material, expressed in per cent (%);

m_1 is the mass of total dry solids in the test material input, expressed in grams (g);

m_2 is the mass of total dry solids in the retrieved test material, expressed in grams (g).

8 Validity of the test

The test is valid if, for all bins, or nets in bins, with biowaste and the mixture of biowaste and test material:

a) the maximum temperature during composting remains below 75 °C during the first week and below 65 °C thereafter;

b) the temperature remains above 60 °C for at least 1 week;

c) the temperature remains above 40 °C for at least 4 consecutive weeks;

d) the pH increases to a value above 7 during the test and does not fall below 5;

e) the biowaste compost of the blank control has a maturity (Rottegrad) of IV or V after 12 weeks and/or the volatile fatty acid content is <500 mg/kg. In addition, some other suitable parameter is used to ensure completion of the normal composting process.

9 Test report

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

a) a reference to this International Standard, i.e. ISO 16929;

b) all information necessary to identify and describe the test material, such as total dry or volatile solids content, shape and visual appearance;

c) the source of biowaste and the results of any analyses carried out at the start-up of the test;

d) a precise description of the composting set-up (bins, nets in bins);

e) the volume of the composting test series and the amounts of biowaste and test material;

f) the test results, i.e. the amount of test material remaining and the degree of disintegration after composting and sieving;

g) the values of the parameters characterizing the composting process, such as the temperature profile, pH, moisture content and oxygen concentration;

h) the results of the analyses carried out at the end of composting;

i) (optional) the results of the 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, documented in writing and by means of photographs;

j) the reasons for rejection of any test results.

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[6] *Methods Book for the Analysis of Compost;* Bundesgotegemeinschaft Kompost e. V, Hauptstral\e 305, D-51143 Cologne, Germany

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