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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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 14, *Environmental aspects,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 16929:2019), which has been technically revised.

The main changes compared to the previous edition are as follows:

— in 6.1.1, the minimum amount of biowaste has been changed to 15 kg from 30 kg due to the smaller size of composting bins;

— in 6.2.2.3, a separate temperature profile has been added to cover tests including also production of compost for ecotoxicity tests.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 industrial compostability of such materials.

To claim industrial compostability, a material not only has to disintegrate in a composting system, it also has to biodegrade in a composting system (as can be shown by standard test methods) and has to complete its biodegradation during the end-use of the compost. Furthermore, the compost has to meet the relevant quality criteria, including low content of regulated metals, no ecotoxicity, and no obviously distinguishable residues.

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

1 Scope

This document defines a test method 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 industrial compostability of plastics as outlined in ISO 17088.

The test method laid down in this document is also used to determine the influence of the test material on the composting process and the quality of the compost obtained. This test method cannot be used to determine the aerobic biodegradability of a test material.

NOTE Other methods are available for this test (for example, see ISO 14851, ISO 14852 or ISO 14855-1 and ISO 14855-2).

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
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

3.2
biodegradation
degradation (3.1) caused by biological activity especially by enzymatic action leading to a significant change in the chemical structure of a material

3.3
disintegration
physical breakdown of a material into very small fragments

3.4
compost
organic soil conditioner obtained by *biodegradation* (3.2) of a mixture principally consisting of various vegetable residues, occasionally with other organic material, and having a limited mineral content

3.5
composting
aerobic process designed to produce *compost* (3.4)

3.6
compostability
property of a material to be biodegraded in a *composting* (3.5) process

3.7
maturity of compost
assignment of the maturity of a *compost* (3.4) 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).

3.8
total dry solids
amount of solids obtained by taking a known volume of test material or *compost* (3.4) and drying at about 105 °C to constant mass

3.9
volatile solids
amount of solids obtained by subtracting the residues of a known volume of test material or *compost* (3.4) after incineration at about 550 °C from the *total dry solids* (3.8) 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 should 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 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 based on 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. 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 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 10 g/kg 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, 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, of suitable shape with screens of 2 mm and 10 mm mesh (as specified, for example, in ISO 3310-2).

6 Test procedure

WARNING — Compost can contain potentially pathogenic organisms. Therefore, appropriate precautions should be taken when handling it. Aspergillosis, farmer's lung, histoplasmosis, Legionnaire's disease, paronychia and tetanus are some of the more common physical ailments that can result from unprotected contact with compost. The following general safety precautions should be followed in order to avoid transmission of dangerous fungi, bacteria and other pathogens found in compost.

— **Always wear gloves to avoid direct contact with the skin.**

— **Always wear protective footwear that covers your skin adequately.**

— **When stirring and tilling the compost, which is required on a regular basis in order for it to process and break down, always wear a nose and mouth guard or dust mask to avoid inhaling the various spores that will become airborne during tilling and turning.**

— **Do not store compost in fully closed or airtight containers.**

— **Always wash your hands after dealing with compost.**

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 can 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 (such as 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 100 g/kg to 600 g/kg 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 1 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.

NOTE 2 The fine powder or granulates is added with the sole purpose to increase the input concentration at start of the composting test (see 6.1.1.4). The fine powder or granulates, if reduced in particle size to <500 µm, will not affect the disintegration of the test material.

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 15 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)]:

   — 10 g/kg of test material in its final form.

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

   — 10 g/kg of test material in its final form;

   — and 90 g/kg of test material as powder or granules.

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

   — 100 g/kg 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 according 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 l/kg of total dry solids per hour.

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

The temperature profile during the test shall follow a regime typical for industrial composting and consisting of an initial thermophilic phase and a mesophilic continuation. The following minimum and maximum temperatures shall be respected:

a) For measurement of the degree of disintegration and compost analysis [see 6.1.1.2 a) and 6.1.1.4 a)]:

— Days 2 to 7: between 60 °C and 75 °C

— Days 8 to 28: between (55 ± 5) °C and (65 ± 5) °C

— Days 29 to 56: between (50 ± 5) °C and (60 ± 5) °C

— Days 57 to 70: below 50 °C

— Days 71 to 84: below 45 °C

If the test includes production of compost for ecotoxicity tests, 100 g/kg of test material is to be added at start of the pilot-scale test [see 6.1.1.2 b)]. This converts to approximately 300 g/kg on biodegradable volatile solids basis, resulting in a more intensive (and longer) thermophilic phase. As a result, a slightly higher maximum for the period day 8 to 28, day 29 to 56 and day 57 to 70 is allowed.

b) For measurement of the degree of disintegration, compost analysis and ecotoxicity tests in one test series [see 6.1.1.2 a), 6.1.1.2 b) and 6.1.1.4 b)]:

— Days 2 to 7: between 60 °C and 75 °C

— Days 8 to 28: between (55 ± 5) °C and (70 ± 5) °C

— Days 29 to 56: between (50 ± 5) °C and (65 ± 5) °C

— Days 57 to 70: below 55 °C

— Days 71 to 84: below 45 °C

c) For the optional ecotoxicity tests in separate test series [see 6.1.1.2 b) and 6.1.1.4 c)]:

— Days 2 to 7: between 60 °C and 75 °C

— Days 8 to 28: between (55 ± 5) °C and (70 ± 5) °C

— Days 29 to 56: between (50 ± 5) °C and (65 ± 5) °C

— Days 57 to 70: below 55 °C

— Days 71 to 84: below 45 °C

Measure the temperature in the middle 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} of > 60 °C (fresh biowaste);

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

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

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

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

For details of the method, see Reference [12].

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.

