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Plastics — Determination of the degree of disintegration of plastic materials in marine habitats under real field conditions

Plastiques — Détermination du degré de désintégration des matériaux plastiques dans les habitats marins en conditions réelles

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 14, *Environmental aspects*.

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

Even though plastics that are biodegradable according to established land-based treatment standards are not and never were intended as a solution to marine littering, the United Nations Environment Programme (UNEP) recognizes that "biodegradability in seawater" can be part of the solution (EuBP, 2016; UNEP, 2016). Hence, plastic materials that are biodegradable might be used as a potential alternative option in order to reduce the residence time of plastic waste in case of dispersion. Thus, the degree and rate of disintegration is of interest in order to determine the durability of products when exposed to the marine environment and the physical disappearance of waste in case of dispersal.

This document describes a disintegration test performed in two different marine habitats under real field conditions. The relative durability of plastic materials of the same size and form may vary depending on the location of the exposure, seasonal variations, the climatic conditions, water movement, tides, availability of nutrients, and diversity and density of the competent microbial community. Hence, it is recommended to perform the disintegration test in regions where the plastic material is likely to end up in the coastal environment for accidental or deliberate reasons.

This document describes a disintegration test and not a biodegradation test, as the conversion of the plastic materials is not determined by means of measuring the O_2-consumption or the CO_2-evolution.

The assessment of the intrinsic aerobic biodegradability of plastic materials exposed to marine environment is covered by ISO 22403.

The determination of the degradation and durability of plastic materials floating on the surface of seawater or partially or completely immersed in coastal shallow seawater under real field conditions is covered by ISO 15314.

Plastics — Determination of the degree of disintegration of plastic materials in marine habitats under real field conditions

1 Scope

This document specifies test methods for the determination of the degree of disintegration of plastic materials exposed to marine habitats under real field conditions.

The marine areas under investigation are the sandy sublittoral and the sandy eulittoral zone where plastic materials can either be placed intentionally (e.g. biodegradable fishing nets) or end up as litter due to irresponsible human behaviour. This depends on their physical characteristics, form and size of the materials, and on water currents and tidal movements.

This document specifies the general requirements of the apparatus, and the procedures for using the test methods described.

The determination of the level of disintegration of plastic materials exposed to pelagic zones such as the sea surface or the water column above the seafloor are not within the scope of this document.

This document is not suitable for the assessment of disintegration caused by heat or light exposure.

The described field test is a disintegration test and not a biodegradation test. Therefore, it cannot be used for demonstrating biodegradation or for making unqualified claims such as “biodegradable in marine environment” and similar.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 4591, *Plastics — Film and sheeting — Determination of average thickness of a sample, and average thickness and yield of a roll, by gravimetric techniques (gravimetric thickness)*

ISO 4593, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ASTM E11, *Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves*

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

[SOURCE: ISO 472:2013, 2.1680]

3.2
degradation
irreversible process leading to a significant change in the structure of a material, typically characterized by a change 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

[SOURCE: ISO 472:2013, 2.262]

3.3
disintegration
physical breakdown of a material into small fragments

[SOURCE: ISO 472:2013, 2.1757]

3.4
pelagic zone
water body above the seafloor

Note 1 to entry: Also referred to as the open water or the water column.

Note 2 to entry: The surface of the pelagic zone is moved by wind-driven waves. It is in contact with the atmosphere and exposed to sunlight. With increasing depth pressure increase, temperature decreases, and light and surface wave energy are attenuated.

3.5
sublittoral zone
coastal seafloor that is permanently immersed and extends from the low-water line to the continental shelf edge at 200 m water depth

Note 1 to entry: The seafloor can consist of solid rock, or fragments that form sediments of different particle size, from coarse blocks and pebbles, to permeable sands, silt and clay. Sediments can form from fragmented rock or consist of fragments of biogenic origin (algae, shells, coral, etc.), or be a mixture of these compounds.

3.6
tidal zone
borderline between sea and land that extends from the high tide line, which is rarely inundated with water, to the low tide line, which is typically always covered with water

Note 1 to entry: The tidal zone is frequently a sandy area that is kept constantly damp by the lapping of the waves.

Note 2 to entry: Stony and rocky shorelines also exist.

Note 3 to entry: Synonyms are: eulittoral zone, midlittoral zone, mediolittoral zone, intertidal zone, foreshore.

[SOURCE: ISO 22404:2019, 3.1]

3.7
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

[SOURCE: ISO 472:2013, 2.1872]

3.8
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* (3.7) content of the same sample

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

[SOURCE: ISO 472:2013, 2.1889]

4 Principle

The disintegration test is a real field test performed under natural environmental conditions in two different coastal regions. Concerned habitats are the eulittoral and the sublittoral zone where plastic materials can end up once dispersed in the sea.

NOTE Plastic materials predominately floating in pelagic zone are not covered by this document.

The test material, preferably in the form of a film, is fixed in non-degradable plastic frames and both sides of the material are protected by a plastic mesh with limited defined mesh size (2 mm) in order to avoid sample loss once the disintegration process has started. Several frames with fixed test specimens are exposed to the eulittoral zone where the material is subjected to the tides and severely fluctuating weather conditions, and to the seawater - sediment interface in the sublittoral zone where less rough environmental conditions are likely to be expected.

The disintegration is determined and reported after an exposure period of three years. However, the disintegration can be investigated in additional samples with exposure periods below or beyond three years, provided that the test procedure and the test evaluation are in accordance with this document.

At the end of the exposure period, the disintegration of the test material is measured by means of removing the protection mesh and sieving the remaining material through 2 mm mesh sieve. The disintegration of the test material is evaluated by comparing the residual material (total dry solids) retained by the 2 mm sieve by the amount introduced (total dry solids).

Alternatively, the disintegration of the test material can be determined as area loss (%) by means of image analysis (photogrammetry). Images of sampled test material specimen are analysed for the ratio between the disintegrated area versus total area of exposed film.

Even if results from different exposure periods are available indicating a constant increase of the disintegration of a test material, it is not allowed to extrapolate the degree of disintegration beyond the maximum exposure period.

5 Test procedure

5.1 Test material

Use the test material preferably in the form of film in an identical form (e.g. shape, thickness) as for the intended final use. The thickness of a film shall be either determined according ISO 4591 or ISO 4593.

Other forms than films, for instance articles such as foams or plates, can also be tested if test procedure and test evaluation are in accordance with this document.

5.2 Reference material

A poly(3-hydroxybutyrate-co-hexanoate) (PHBH) film[1)] of 25 µm to 30 µm thickness shall be used as a positive control. As a negative control a low-density polyethylene (PE-LD) film of 25 µm to30 µm thickness shall be used.

5.3 Preparation of test and reference materials

Test samples shall not be subjected to conditions or procedures, such as a pretreatment by heat and/or an exposure to radiation, designed to accelerate disintegration prior to testing according to this document.

A plastic material preferably in the form of a film is cut into pieces of 260 mm × 200 mm in size. A test specimen is covered with a non-degradable plastic mesh with a 2 mm × 2 mm mesh size on both sides to prevent eventual fragments from being lost. Use meshes of suitable shape with a screen of 2 mm as specified for instance in ASTM E11. The specimen covered by the meshes is then fixed between two non-degradable plastic frames of 260 mm × 200 mm and 200 mm × 160 mm external and internal dimension, respectively (see Figure 1). Typical non-biodegradable meshes are made of polyamide, polyethylene or polypropylene. The surface area of the film specimen which is exposed to the marine habitats is 320 cm^2.

Film specimen that is 200 mm × 160 mm in size (surface area 320 cm^2) can be used as an alternative fitting in the inner part of the plastic frame not fixed between two non-degradable plastic frames. The film is still covered by non-degradable plastic mesh preventing the loss of material during the exposure period.

Figure 1 — Film specimen covered with a non-degradable plastic mesh and fixed between two non-degradable plastic frames

5.4 Number of replicates

Provide a sufficient number of samples prepared according to 5.3, at least:

— five frames for the test material (F_T 1-5)

— five frames for the positive reference material (F_{pR} 1-5)

1) Supplier of PHBH-pellets: www.kaneka.be/documents/PHBH-brochure-11-2017.pdf. Use pellets to blow a film of 25 µm to 30 µm thickness. PHBH-pellets from Kaneka are an example of a suitable reference material. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the supplier named.

— five frames for the negative reference material (F_{nR} 1-5)

The frames shall be marked by appropriate means to ensure a clear and permanent allocation of each sample even after an exposure period of three years (see Figure 2).

Figure 2 — Code identifying that the sample is attached with a cable tie to the frame

The same number of replicates is requested if other forms than film, e.g. formed articles such as foams or plates, are tested.

The above outlined number of replicates is sufficient for the determination of the disintegration at the end of the exposure period after three years. Prepare the same number of replicates per sampling if samples with exposure periods below or beyond three years are planned, e.g. to better characterize the disintegration of a plastic material over time.

As this is a field test in different marine habitats, mechanical damage of test samples fixed between two plastic frames and protected by a 2 mm × 2 mm protection mesh cannot be excluded during the exposure period, possibly due to the erosive power of tides and sediment and activity of animals. Hence, it is recommended to increase the number of replicates for each material to compensate any loss of test specimens.

5.5 Exposure to marine habitats

The exposure of test specimens (see 5.3) to the seawater — seafloor interface at the sublittoral zone shall be performed according to the procedure described in Annex A.

The exposure of test specimens (see 5.3) to the eulittoral zone shall be performed according to one of the two procedures described in Annex B.

5.6 Termination of the field test

The disintegration is determined and reported after an exposure period of three years.

If test results from samples with exposure periods below three years are available revealing that no more than 10 % of the original mass (dw) of the exposed surface area (see Figure 1) remains in the oversize fraction after sieving through a 2 mm sieve (see 6.2), then the field test can be terminated before the three-year exposure period has been reached and the degree of disintegration can be determined. This also applies if the disintegration is determined by means of image analysis (photogrammetry) and more than 90 % of exposed surface area is lost.

Samples are carefully removed from the eulittoral and the sublittoral zone, rinsed in ambient seawater from the same exposure site, packed singly in zip-lock bags under wet conditions using the seawater from the same exposure site and stored in sealed containers for the transportation to the laboratory. After delivery, conserve the samples at low temperature (approximately 4 °C) until processing. It is recommended that the samples are analysed within 2 days after sampling. Record storage time and conditions.