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Plastics — Test methods for determination of degradation rate and disintegration degree of plastic materials exposed to marine environmental matrices under laboratory conditions

Plastiques — Méthodes d'essai pour l'évaluation de la vitesse de dégradation et du degré de désintégration des matériaux plastiques exposés aux matrices environnementales marines dans des conditions de laboratoire

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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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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

Plastics are potentially susceptible to ultimate biodegradation, i.e. to be decomposed by the actions of microorganisms under aerobic conditions into CO_2, water and biomass as can be proven with specific test methods. In most cases, biodegradation occurs at the surface of the plastics materials, i.e. at the solid-liquid interface. Microbes and enzymes cannot penetrate the solid plastic item, thus only the exposed surface is generally available to biodegradation. The physical effect of biodegradation on a solid plastic item is erosion leading to a thinning and weakening of the item. This process leads the item to lose mass, physical properties, and ultimately physical integrity by fragmentation into biodegradable particles whose ultimate fate is to be biodegraded. The term disintegration is used when the degradation process is extended until a total fragmentation of the original item into particles below a defined size is reached. When microorganisms cause degradation processes *bio*degradation, *bio*fragmentation, *bio*disintegration are the proper terms, etc. as suggested by CEN/TR 15351. However, when the physical breakdown rather than the chemical breakdown is measured, the generic term "degradation" is preferably used, reserving the term "biodegradation" to the assessment of the ultimate biodegradation, i.e. the conversion into CO_2, H_2O and biomass.

The assessment of specific degradation rates occurring when plastics materials are exposed to marine matrices is needed for designing products intended for marine applications (e.g. biodegradable plastic fish and mussel farming, floating devices) and for assessment of the risk caused by leakage of products into the sea.

In this document three test methods for testing degradation are described. Plastics samples can be exposed to three different test conditions and different marine matrices:

— buried into a wet sandy marine sediment;

— at the interface between a marine sandy sediment and the water column;

— to seawater.

The conditions applied in these test methods are designed to determine the degradation rates of plastics materials and give an indication of their propensity to physical degradation and disintegration in natural environments.

Degradation rates considered in this document are mass loss rate, erosion rate, and mechanical properties loss. Disintegration, i.e. physical breakdown of a sample into very small fragments (<2mm), can also be assessed.

Plastics — Test methods for determination of degradation rate and disintegration degree of plastic materials exposed to marine environmental matrices under laboratory conditions

1 Scope

This document specifies test methods for the measurement of the physical degradation of samples made with plastics materials when exposed to marine environmental matrices under aerobic conditions at laboratory scale.

This document is not suitable for the assessment of degradation caused by heat (thermo-degradation) or light exposure (photo-degradation).

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 527-1, *Plastics — Determination of tensile properties — Part 1: General principles*

ISO 527-2, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics*

ISO 527-3, *Plastics — Determination of tensile properties — Part 3: Test conditions for films and sheets*

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

ISO 16012, *Plastics — Determination of linear dimensions of test specimens*

ASTM D 638-14, *Standard Test Method for Tensile Properties of Plastics*

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 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 very small fragments

[SOURCE: ISO 14855-1:2012, 3.3]

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

[SOURCE: ISO 13975:2019, 3.5]

3.5
volatile solids
amount of solids obtained by subtracting the residues of a known volume of test material or inoculum after incineration at about 550 °C from the *total dry solids* (3.4) 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 13975:2019, 3.6]

4 Principle

This document describes three laboratory test methods:

a) Method A: Sand burial degradation test;

b) Method B: Sediment/seawater interface degradation test; and

c) Method C: Seawater degradation test.

These three test methods are based on the exposure of plastic samples to environmental matrices taken from the sea and on the measurement of physical degradation.

These three test methods differ for the exposure conditions.

In Method A, the plastic samples are buried in a wet sandy sediment (a condition similar to the sandy shoreline where beach is maintained wet by the waves and tides).

In Method B, the plastic samples are laid at the interface between a sandy sediment bed and a water column (a condition similar to the seabed where most debris sinks, accumulates, and undergoes degradation).

In Method C, the plastic samples are exposed to seawater.

The degradation rate of the plastic material can be measured as:

a) mass loss and/or

b) erosion and/or

c) tensile properties decay.

The three test methods can also be used to determine the time for disintegration, i.e. the time needed to get the plastic samples fragmented into pieces below 2 mm, as determined by the surface area loss and/or mass loss determination.

The three test methods can be performed together or independently.

Claims of performance shall be limited to the numerical result obtained in the test and not be used for unqualified "biodegradable in marine environment" claims and similar. The results obtained are solely referred to the propensity to physical degradation caused by exposure to environmental matrices. The results do not give information regarding the ultimate biodegradability in the marine environment.

The test design (i.e. the total number of tested samples, the number of replicates and of repeated measurements) of the test methods is flexible. The complexity of test design and the cost of testing can be modulated according to the requests and purposes of the client. For example, tests planned for results delivered under statistically optimal conditions can be arranged for certification purposes, while simpler tests can be arranged for screening purposes.

5 Reagents

5.1 Distilled or deionized water, free of toxic substances (copper in particular) and containing less than 2 mg/l of DOC.

5.2 Artificial seawater

Dissolve:

Sodium chloride (NaCl)	22 g
Magnesium chloride hexahydrate ($MgCl_2 \cdot 6\,H_2O$)	9,7 g
Sodium sulfate (Na_2SO_4)	3,7 g
Calcium chloride ($CaCl_2$)	1 g
Potassium chloride (KCl)	0,65 g
Sodium hydrogen carbonate ($NaHCO_3$)	0,20 g

in water (5.1) and make up to 1 000 ml

6 Environmental matrix

6.1 Sampling

Take a sample of a sandy sediment with a shovel beneath the low-water line at the shoreline and/or seawater with a bucket. Record location and date of sampling. The wet sediment together with seawater is transferred into sealed containers for transport and fast delivered to the laboratory. After delivery, conserve the sediment and seawater at low temperature (approximately 4 °C) until use. The seawater/sediment sample should preferably be used within 4 weeks after sampling. Record storage time and conditions. More detailed instructions about sampling, preservation, handling, transport and storage of marine matrices are given in ISO 5667-3.

Measure the total dry solids, total organic carbon [(TOC) or, as an alternative, ashes and volatile solids], pH, and nitrogen content of the sediment and of the natural seawater.

The pH can be measured by applying ISO 10523 with seawater or ISO 10390 with marine sediments. ISO 10694 can be applied to determine the TOC and ISO 11261 can be applied to determine nitrogen content. A description on how to measure total dry solids, volatile solids and ashes of a solid environmental matrix (e.g. marine sediment or compost) is given in ISO 20200 and ISO 16929.

6.2 Preparation of the sediment and seawater

Remove, manually or by sieving, stones, pebbles and other materials until a clean marine sediment is obtained .

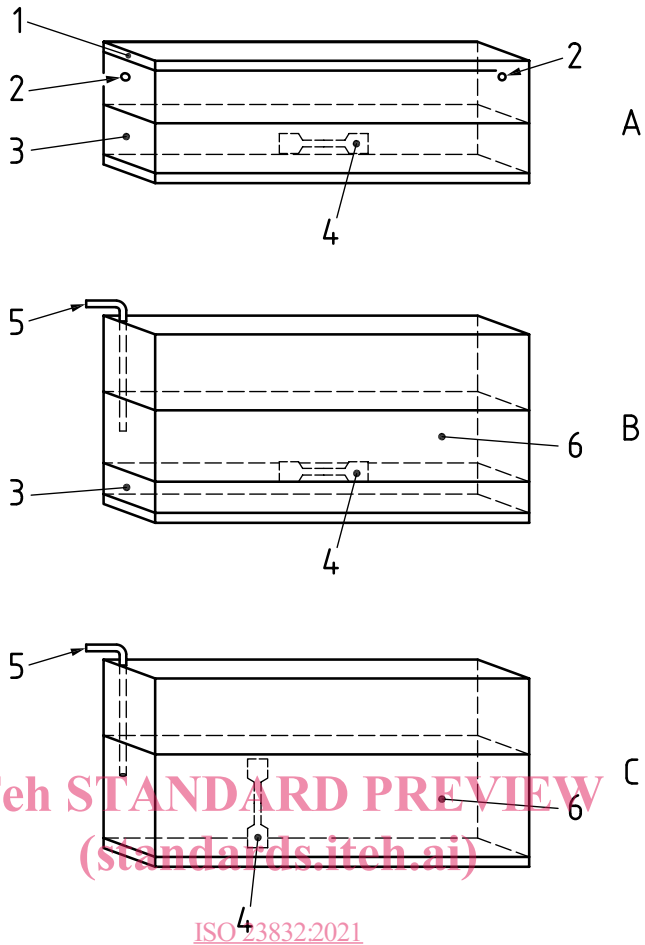
Filter the sediment in a funnel with a coarse filter paper to eliminate excess seawater. Sediment is ready for testing when seawater dripping stops. Sediment after filtering is named “wet sediment” hereafter and ready for Method A and Method B.

Natural seawater is directly used without filtration.

7 Apparatus

7.1 General

Polypropylene boxes or domestic aquariums (i.e. fish tanks for hobbyists) are suitable for the purposes of this document. However, if the test material is made with plastics expected to have degradation properties similar to the plastics used in the aquarium, glass should be used. See Figure 1 for a schematic representation of the tanks, which can be used to carry out the test methods.



Key

A	sand burial degradation test	3	wet sediment
B	sediment/seawater interface degradation test	4	plastic sample
C	seawater degradation test	5	aeration system
1	lid	6	seawater
2	5 mm hole for gas exchange		

Figure 1 — Apparatus

7.2 Test Method A (Sand burial degradation test)

Polypropylene (or other suitable material) boxes with a minimum dimension approximately of 30 cm × 20 cm × 10 cm (length, width, height) are appropriate. Alternatively, test devices as described in 7.3 can be used to perform the sand burial degradation test. Each box shall be provided with a lid assuring a tight closing to avoid an excessive vapour release. The closing between box and lid can be sealed with an adhesive tape to limit the water evaporation. In the middle of the two 20 cm wide sides, a hole of 5 mm of diameter shall be done at a height of about 6,5 cm from the bottom. The two holes provide gas exchange between the inner atmosphere and the outside environment. Attention shall be paid not to cover them with the adhesive tape, or in other way.

7.3 Test Method B (Sediment/seawater interface degradation test)

Tanks/aquariums (made with polypropylene or other suitable materials) with a minimum volume of 12 l (e.g. with dimension of 30 cm × 20 cm × 20 cm length, width, height) are appropriate. The dimension of the aquarium should be decided on the basis of the experimental design established by the operator, i.e. form and size of the plastic material to be tested and the number of replicates.