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Plastics — Assessment of the intrinsic biodegradability of materials exposed to marine inocula under mesophilic aerobic laboratory conditions — Test methods and requirements

Plastiques — Évaluation de la biodégradabilité aérobie inhérente et de la sécurité environnementale des matériaux non flottants exposés à des inocula marins dans des conditions de laboratoire et mésophiles — Méthodes d'essai et exigences

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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

Biodegradation (i.e. biodegradation level and biodegradation rate) of a plastic product in any given environment is basically the result of three conditions:

a) the intrinsic (i.e. potential) biodegradability of the material;

b) the available surface and the shape of the product;

c) the environmental conditions where the product is located.

A material's intrinsic biodegradability provides that its chemical structure is susceptible to enzymatic attack so that enzymes can cleave its chemical bonds. Under aerobic conditions (in the presence of O_2) the ultimate biodegradation of a material only leads to the formation of CO_2, H_2O, mineral salts and biomass.

Biodegradation of plastic materials is generally a bio-erosion process happening at the interface between the solid phase and the liquid phase where microbes live. It is a heterogeneous reaction. It is the surface rather than the concentration that controls the biodegradation rate. Thus, the higher the available surface the higher the biodegradation rate.

Environmental conditions determine the biodegradation rate as well. Temperature, nutrient availability, pH and the existing microbial population affect the biodegradation rate. Biodegradation can be slowed or even stopped if environmental conditions are not favourable, even if the material is intrinsically and ultimately biodegradable.

This document covers condition a) mentioned above.

The fate of plastics in the environment is considered important information. The contamination of seas with plastic waste is a relevant problem that should be controlled not least by means of leakage prevention measures such as mandatory collection of plastic items used in marine environments and environmental education. However, in some cases, the dispersal of plastic (waste) is almost unavoidable. For example, plastics are used to make fishing gears and products for fish, mussels, and oysters farming which are prone to be left or lost in the sea. In these cases, the possibility of using products made with biodegradable plastics might be contributing to reducing the risk linked with the dispersion of solid waste. In order to carry out a proper product design and in order to assess impact and risk of leakage, it is important to know whether a plastic material is intrinsically biodegradable when exposed to marine inocula.

Plastics — Assessment of the intrinsic biodegradability of materials exposed to marine inocula under mesophilic aerobic laboratory conditions — Test methods and requirements

1 Scope

This document specifies test methods and criteria for showing intrinsic biodegradability in marine environments of virgin plastic materials and polymers without any preliminary environmental exposure or pre-treatment.

Test methods applied in this document are carried out at temperatures in the mesophilic range under aerobic conditions and are aimed to show ultimate biodegradability, i.e. conversion into carbon dioxide, water and biomass.

This document neither assesses the constituents, such as regulated metals or substances hazardous to the environment, nor potential ecotoxic effects but intrinsic biodegradability only. These aspects will be considered in a separate standard covering the overall environmental impact of products intentionally or accidentally released in the marine environment.

This document does not cover the performance of products made from biodegradable plastic materials and biodegradable polymers. Lifetime and biodegradation rates in the sea of products made with biodegradable plastic materials are generally affected by the specific environmental conditions and by thickness and shape.

Although results might indicate that the tested plastic materials and polymers biodegrade under the specified test conditions at a certain rate, the results of any laboratory exposure cannot be directly extrapolated to marine environments at the actual site of use or leakage.

This document is not applicable for “marine biodegradable” claims of biodegradable plastic materials. For such purpose, see relevant product standards, if available.

The testing scheme specified in this document does not provide sufficient information for determining the specific biodegradation rate (i.e. the rate per available surface area) of the material under testing. For such purpose, see relevant standards about specific biodegradation rate, if available.

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 10210, *Plastics — Methods for the preparation of samples for biodegradation testing of plastic materials*

ISO 18830, *Plastics — Determination of aerobic biodegradation of non-floating plastic materials in a seawater/sandy sediment interface — Method by measuring the oxygen demand in closed respirometer*

ISO 19679, *Plastics — Determination of aerobic biodegradation of non-floating plastic materials in a seawater/sediment interface — Method by analysis of evolved carbon dioxide*

ISO 22404, *Plastics — Determination of the aerobic biodegradation of non-floating materials exposed to marine sediment — Method by analysis of evolved carbon dioxide*

ISO 23977-1:—[1)], *Plastics — Determination of the aerobic biodegradation of plastic materials exposed to seawater — Part 1: Method by analysis of evolved carbon dioxide*

ISO 23977-2:—[2)], *Plastics — Determination of the aerobic biodegradation of plastic materials exposed to seawater — Part 2: Method by measuring the oxygen demand in closed respirometer*

ASTM D6691-17, *Standard Test Method for Determining Aerobic Biodegradation of Plastic Materials in the Marine Environment by a Defined Microbial Consortium or Natural Sea Water Inoculum*

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
intrinsic biodegradability
ability of a polymer or plastic material to be biodegraded established under controlled laboratory conditions

3.2
ultimate biodegradability
breakdown of an organic chemical compound by microorganisms in the presence of oxygen to carbon dioxide, water, and mineral salts of any other elements present (mineralization) and new biomass or in the absence of oxygen to carbon dioxide, methane, mineral salts, and new biomass

[SOURCE: ISO 18606:2013, 3.5]

3.3
constituent
every pure chemical material or substance of which a polymer or plastic material is composed

4 Requirements

4.1 Test material

Plastic materials, polymers, and organic constituents shall be tested in the form specified by the applied standard test method. Refer to ISO 10210 for preparation of powder from plastic materials if milling is required. Test samples shall not be subjected to any pre-treatment (e.g. by heat and or an exposure to radiation exposure) nor naturally aged.

4.2 Reference material

The reference material shall be cellulose (i.e. microcrystalline cellulose or cellulose filter paper).

4.3 Negative control

A negative control shall be tested in parallel with test and reference material. Virgin polyethylene shall be used as negative control.

1) Under development. Stage at the time of publication: ISO/DIS 23977-1:2020.

2) Under development. Stage at the time of publication: ISO/DIS 23977-2:2020.

4.4 Biodegradation test methods

The plastic material, polymer or each organic constituent separately shall be tested together with reference material and negative control according to one of the following marine biodegradation test methods: ISO 18830, ISO 19679, ISO22404, ASTM D6691-17, ISO 23977-1:—, ISO 23977-2:—.

The test may be stopped when a plateau phase is reached. As a guidance, the plateau phase is considered to be reached when the average biodegradation rate of test material of at least 3 consecutive measuring points is less than 3 % within 2 months. After one-year testing, carefully monitor test conditions in order to ensure reliable conditions in the long term. Follow the instructions provided by the applied standard test methods. Results shall be taken into consideration only if validity criteria of the chosen standard test methods are met.

4.5 Requirements

For whole test material or each individual constituent, organic carbon shall mineralize into carbon dioxide for at least 90 % or for the same extent of the reference material within 2 years. For the purposes of this document, this requirement is proven if mineralisation relative to reference material is at least 90 %. Both the reference material and the test sample shall be tested for the same period and the results compared at the same point in time after e.g. the activity of both has reached a plateau.

For organic constituents which are present in the material at a concentration between 1 % and 15 % (by dry mass) the level of biodegradation shall be determined separately. Constituents that turned out to be readily biodegradable in a ready biodegradation test according to an OECD test guideline (OECD 301, Methods A to F); OECD 310 are considered biodegradable in the context of this document.

As an alternative, the level of biodegradation of an organic constituent may be determined using an artificial blend of the same material. This artificial blend shall consist of at least 15 % of the respective organic constituent [by total organic carbon (TOC) content]. The chemical composition and the structure of the material shall remain the same, but the amount of the organic constituent under consideration shall be increased to a minimum of 15 % [by total organic carbon (TOC) content]. The artificial blend shall be produced following the same processing conditions (e.g. extrusion) as used for the production of the original material containing less than 15 % (by dry mass) of the respective constituent. In case the artificial blend meets the criteria specified above, the constituent is considered biodegradable in the context of this document. The constituent can then be used at the same (15 %) or lower concentration (<15 %; by dry mass) in a material that also contains the same co-substrate as the tested material.

NOTE 1 The objective of testing an artificial blend is to verify a synergistic effect demonstrating that a constituent, which is suspected to be non-biodegradable when tested alone, becomes biodegradable in combination with another biodegradable constituent of a material.

NOTE 2 The concentration of the constituent in the artificial blend is set at a minimum of 15 % in order to avoid false-positive results, as theoretically a material with, for example, 10 % of a non-biodegradable constituent can still reach the required pass level for biodegradation.

Chemically unmodified materials and constituents of natural origin (such as wood, wood fibre, cotton fibre, starch, paper pulp, bagasse, jute) shall be accepted as being intrinsically biodegradable without testing.

In principle, a positive biodegradation result obtained with one of the test methods indicated in 4.4 is sufficient to show that the test item is susceptible to biodegradation by marine microorganisms. A test item that fails the test cannot be claimed to be intrinsically biodegradable. However, a negative result could be the consequence of an inoculum with a qualitative or quantitative (or both) ineffective microbial population. Thus, it cannot be excluded that the test item could show biodegradation if test is repeated using different inoculum.

