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Plastics — Soil biodegradable materials for mulch films for use in agriculture and horticulture — Requirements and test methods regarding biodegradation, ecotoxicity and control of constituents

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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).

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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

Biodegradable mulch films are used worldwide in agriculture and horticulture for many years. They enhance growing conditions and contribute to increased yields and improved crop quality by, for example:

— inhibiting the development of weeds;

— reducing significantly the consumption of water and other resources;

— control of soil temperature;

— reduction in leaching of mineral elements and other fertilizer;

— reduction in soil compaction;

— protecting the crops from soil.

Biodegradable mulch films are not designed to be recovered from soil at the end of the intended service life. Therefore, it is no longer necessary for farmers to retrieve the biodegradable mulch film from the field for disposal or recycling after the harvest. Farmers can simply plow it under along with what remains from the plants so that it is incorporated into soil.

This document defines the standard specification to be met for biodegradable mulch films to be used in agriculture and horticulture. It is suited to characterize both the plastic materials which are used to manufacture mulch films and the mulch films itself with respect to characteristics such as biodegradation, adverse effects on terrestrial organisms and control of constituents.

Plastics — Soil biodegradable materials for mulch films for use in agriculture and horticulture — Requirements and test methods regarding biodegradation, ecotoxicity and control of constituents

1 Scope

This document is applicable to biodegradable plastic materials used to produce mulch films or biodegradable mulch films ready to be used for mulch applications in agriculture and horticulture.

This document specifies test methods and evaluation criteria by addressing the following characteristics:

a) control of constituents;

b) biodegradation;

c) negative effects on terrestrial organisms.

NOTE This document is construed in a way that it can be used to assess other soil biodegradable plastic products that do not qualify as mulch films. For example: drip tape, twine, clips, and plant pots.

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 10390, *Soil quality — Determination of pH*

ISO 11268-1, *Soil quality — Effects of pollutants on earthworms — Part 1: Determination of acute toxicity to Eisenia fetida/Eisenia andrei*

ISO 11268-2, *Soil quality — Effects of pollutants on earthworms — Part 2: Determination of effects on reproduction of Eisenia fetida/Eisenia andrei*

ISO 11269-2, *Soil quality — Determination of the effects of pollutants on soil flora — Part 2: Effects of contaminated soil on the emergence and early growth of higher plants*

ISO 11274, *Soil quality — Determination of the water-retention characteristic — Laboratory methods*

EN 14582, *Characterization of waste — Halogen and sulfur content — Oxygen combustion in closed systems and determination methods*

ISO 15685, *Soil quality — Determination of potential nitrification and inhibition of nitrification — Rapid test by ammonium oxidation*

ISO 17556, *Plastics — Determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved*

OECD. (2006), Test No. 208: Terrestrial Plant Test: Seedling Emergence and Seedling Growth Test, OECD Guidelines for the Testing of Chemicals, Section 2

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
mulch film
film made from thermoplastic material intended to be used in agriculture and horticulture to cover the ground in order to improve growing conditions of crops and depending on the colour to control weeds

Note 1 to entry: It is assumed that a transparent mulch film does not allow to control weeds.

[SOURCE: EN 17033:2018, 3.1.1]

3.2
material
homogenous preparation of biodegradable polymer and additives, as necessary, such as carbon black and colour pigments

Note 1 to entry: Additives are usually introduced under the form of masterbatches using as carrier resin a biodegradable polymer.

[SOURCE: EN 17033:2018, 3.1.2]

3.3
ultimate aerobic biodegradation
breakdown of an organic compound by microorganisms in the presence of oxygen into carbon dioxide, water and mineral salts of any other elements present (mineralization) plus new biomass

[SOURCE: ISO 17088:2021, 3.9]

3.4
mineralization
decomposition of organic matter or organic substances into carbon dioxide, water and the hydrides, oxides or other mineral salts

[SOURCE: ISO 11074:2015, 3.3.19]

3.5
disintegration
physical breakdown of a material into fragments

[SOURCE: ISO 18606:2013, 3.3]

3.6
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]

3.7
total dry solid
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 17088:2021, 3.8]

3.8
volatile solids
amount of solids obtained by subtracting the residue of a known volume of test material or compost after incineration at about 550 °C from the *total dry solids* (3.7) of the same sample

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

[SOURCE: ISO 17088:2021, 3.10]

3.9
organic constituent
chemical constituent that contains carbon covalently linked to other carbon atoms and to other elements, most commonly hydrogen, oxygen or nitrogen

Note 1 to entry: Inorganic carbonates, carbides, cyanides and simple oxides such as carbon monoxide and carbon dioxide are not considered as organic constituent.

Note 2 to entry: Allotropes of carbon, such as diamond, graphite, carbon black, fullerenes, and carbon nanotubes are also not considered as organic constituent.

[SOURCE: ISO 17088:2021, 3.15]

3.10
natural soil
soil collected from the surface layer of fields and/or forests

[SOURCE: EN 17033:2018, 3.3.1]

3.11
poly- and perfluoroalkyl substances
PFAS
organofluorine compounds containing only carbon-fluorine bonds and carbon-carbon bonds but also other heteroatoms

4 Basic requirements

4.1 General

In order to be identified as a biodegradable mulch film in accordance with this document, the mulch film or the material of the mulch film under investigation shall fulfil all requirements specified in Clause 4 and Clause 5, respectively.

If not all requirements are met, no reference to this document shall be made.

4.2 Control of constituents

The mulch film or the material of the mulch film shall be identified and characterized prior to testing including:

— determination of the presence of regulated metals and other elements;

— determination of the presence of organic [poly- and perfluoroalkyl substances (PFAS)] and inorganic fluorine (determined as fluorine);

— evaluation of the presence of other hazardous substance as specified in Annex B;

taking legal compliance into consideration.

In addition, volatile solids shall be determined.

4.3 Ultimate aerobic biodegradation

The ultimate level of aerobic biodegradation shall be established by testing under controlled conditions as laid down in 5.2.1.

4.4 Negative effects on terrestrial organism

The mulch film or the material of the mulch film shall have no adverse effects on terrestrial organisms.

5 Detailed requirements

5.1 Control of constituents

5.1.1 Regulated metals and other elements

The concentrations of regulated metals and other elements in a mulch film or material of the mulch film shall be less than 50 % of those prescribed for sludges, fertilizers and composts in the country where the final product will be placed on the market or disposed of (see Annex A for examples).

Regulated metals and other elements shall be determined and reported.

5.1.2 Organic and inorganic fluorine

5.1.2.1 Organic fluorine (PFAS)

From a precautionary perspective, poly- and perfluoroalkyl substances (PFAS) shall not be intentionally added to the mulch film or to a material of the mulch film.

NOTE Most of poly- and perfluoroalkyl substances (PFAS) are extremely persistent in the environment and in addition, certain PFAS are suspected to have bioaccumulative properties and adverse effects for environment and human health.

5.1.2.2 Inorganic fluorine

The concentration of inorganic fluorine in the mulch film or in a material of the mulch film shall be less than 100 mg fluorine/kg material (dry matter) (see Annex B).

5.1.3 Other hazardous substances

From a precautionary perspective, hazardous substances as specified in Annex B shall not be intentionally added to a mulch film or material of the mulch film.

Information on the use of hazardous substances shall be recorded and can be based on a self-declaration.

5.1.4 Volatile solids

A mulch film or material of a mulch film shall contain a minimum of 60 % of volatile solids.

Volatile solids shall be determined and reported.

5.2 Ultimate aerobic biodegradation

5.2.1 Test method and evaluation criteria

The ultimate aerobic biodegradability shall be determined for the whole material or for each organic constituent.

Test samples shall not be subjected to conditions or procedures, such as a pre-treatment by heat and or an exposure to radiation exposure, designed to accelerate biodegradation prior to testing according to ISO 17556.

The material is considered to have demonstrated a satisfactory rate and level of biodegradation in soil if, when tested in accordance with ISO 17556, it achieves a minimum biodegradation percentage as specified hereunder:

a) 90 % of the organic carbon shall have been converted to CO_2 by the end of the test period (relative to a reference material). Both the reference material and the test item shall be tested for the same length of time and the results compared at the same point in time;

b) as an alternative, 90 % (in absolute terms) of the organic carbon shall have been converted to carbon dioxide by the end of the test period.

If the level of biodegradation exceeds 90 % (relative to a reference material or in absolute terms), then the biodegradation test can be terminated. However, the test period shall be no longer than 2 years.

The biodegradation test shall be performed at a temperature constant to within ±2 °C in the range between 20 °C and 28 °C, preferably 25 °C.

Use, as reference material, a well-defined biodegradable polymer (microcrystalline-cellulose powder or ashless cellulose filters). If possible, the physical form and size of the reference material should be comparable to that of the test material.

The validity criteria as stated in ISO 17556 shall be fulfilled.

NOTE Biodegradability is assessed by measuring the mineralization level, i.e. the conversion of the organic carbon of a product or a material into CO_2 with the consumption of O_2 under aerobic conditions, or into CO_2 and CH_4 under anaerobic conditions. During biodegradation, part of the organic carbon is also assimilated as biomass. This biomass yield typically ranges from 10 % to 40 %, depending on the substrate. As a consequence, the mineralization level rarely reaches 100 % also when the biodegradation is 100 %, because of biomass formation. Standard test methods for the accurate determination of product´s or material's carbon assimilated in biomass during biodegradation are not available yet.

5.2.2 Requirements regarding constituents

Organic constituents which are present at concentrations of less than 1 % (dry mass) do not need to demonstrate biodegradability. However, the sum of such constituents shall not exceed 3 % (dry mass).

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 and meet the criteria specified in 5.2.1. Organic constituents at a concentration between 1 % and 15 % (by dry mass) that turned out to be readily biodegradable in a ready biodegradation test according to an OECD test guideline (OECD 301, Methods A to F[30]; OECD 310[31]) are considered as biodegradable in the context of ISO 23517.

As an alternative to testing the single organic constituent used between 1 % and 15 % (by dry mass), the level of biodegradation of that organic constituent can be determined using an artificial blend of the same material consisting of at least 15 % by total organic carbon (TOC) content. In case this artificial blend meets the criteria specified in 5.2.1, then the organic constituent in question is considered to be biodegradable in the context of ISO 23517 and can be used at the same or lower concentration in a material on the condition that the co-substrate is present as tested in the artificial blend.

Carbon black which is frequently used in mulch film is an inert solid. Therefore, it is not considered as an organic constituent and shall not be accounted in the calculation of the degree of biodegradation.

NOTE The objective of testing an artificial blend is to demonstrate that a constituent which does not meet the biodegradability requirements of ISO 23517 when tested alone, may become biodegradable in combination with another biodegradable constituents of a material. The concentration of the constituent in the artificial blend was set at a minimum of 15 % in order to avoid false-positive results, as theoretically a material with, for example, 10 % of a non or moderately biodegradable constituent may still reach the pass level for biodegradation specified in 5.2.1.

5.3 Negative effects on terrestrial organism

5.3.1 Ecotoxicity testing scheme

Ecotoxicity tests shall be performed in order to investigate possible adverse effects caused by degradation products resulting from the degradation of the material of a mulch film in soil at the end of the intended service life.

The test scheme takes into account:

— all relevant terrestrial organism groups as plants, earthworms (invertebrates) and microorganisms;

— important ecological processes critical due to their role in maintaining soil functions as breakdown of organic matter, formulation of soil structure and cycling of materials;

— all relevant exposure pathways as soil pore water, soil pore air and soil material.

The link between soil organism groups of major ecological importance covering all significant soil exposure pathways and suitable test methods for the evaluation of ecotoxicity of the materials of mulch films and their degradation products is shown in Table 1.

Table 1 — Test scheme for the assessment of ecotoxicity

Organism group	Ecological process	Soil exposure pathway	Test methods
plants: - higher plants	primary production	mainly soil pore water (by root uptake)	plant growth test according to OECD 208 or ISO 11269-2 with the modifications specified in Annex C
invertebrates: - earthworms	breakdown of organic matter; formation of soil structure	diverse and multiple up-take routes: - soil pore water; - ingestion of soil material; - soil air	acute earthworm test according to ISO 11268-1 with the modifications specified in Annex D or alternatively chronic earthworm toxicity test according to ISO 11268-2 with the modifications laid down in Annex E
microorganisms: - bacteria	recycling of nutrients	mainly soil pore water	nitrification inhibition test with soil microorganisms according to ISO 15685 with the modifications specified in Annex F

Ecotoxic effects on terrestrial organism shall be determined by comparing soils produced with or without the addition of a test material.

5.3.2 Preparation of soils for ecotoxicity tests

The ecotoxicity of degradation products shall be evaluated according to the test methods specified in 5.3.3, 5.3.4 and 5.3.5 using test soils prepared according to Annex G.

