

SLOVENSKI STANDARD
oSIST prEN ISO 17556:2018
01-julij-2018

Polimerni materiali - Ugotavljanje končne aerobne biorazgradljivosti polimernih materialov v zemlji z merjenjem porabe kisika v respirometru ali količine nastalega ogljikovega dioksida (ISO/DIS 17556:2018)

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 (ISO/DIS 17556:2018)

Kunststoffe - Bestimmung der vollständigen aeroben Bioabbaubarkeit von Kunststoffmaterialien im Boden durch Messung des Sauerstoffbedarfs in einem Respirometer oder der Menge des entstandenen Kohlendioxids (ISO/DIS 17556:2018)

Plastiques - Détermination de la biodégradabilité aérobie ultime des matériaux plastiques dans le sol par mesure de la demande en oxygène dans un respiromètre ou de la teneur en dioxyde de carbone libéré (ISO/DIS 17556:2018)

Ta slovenski standard je istoveten z: prEN ISO 17556

ICS:

83.080.01	Polimerni materiali na splošno	Plastics in general
-----------	--------------------------------	---------------------

oSIST prEN ISO 17556:2018

en,fr,de

DRAFT INTERNATIONAL STANDARD

ISO/DIS 17556

ISO/TC 61/SC 14

Secretariat: DIN

Voting begins on:
2018-04-23Voting terminates on:
2018-07-16

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

Plastiques — Détermination de la biodégradabilité aérobie ultime des matériaux plastiques dans le sol par mesure de la demande en oxygène dans un respiromètre ou de la teneur en dioxyde de carbone libéré

ICS: 83.080.01

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN ISO 17556:2019

<https://standards.iteh.ai/catalog/standards/sist/ea12624b-3c19-4fa2-962a-f50c947f9532/sist-en-iso-17556-2019>

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING



Reference number
ISO/DIS 17556:2018(E)

© ISO 2018

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN ISO 17556:2019

<https://standards.iteh.ai/catalog/standards/sist/ea12624b-3c19-4fa2-962a-f50c947f9532/sist-en-iso-17556-2019>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2018

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Principle	3
5 Test environment	4
6 Materials	4
7 Apparatus	4
8 Procedure	5
8.1 Preparation of the test material	5
8.2 Preparation of the reference material	5
8.3 Preparation of the test soil	6
8.3.1 Collection and sieving of soil	6
8.3.2 Preparation of standard soil	6
8.3.3 Measurement of soil characteristics	7
8.3.4 Adjustment of the water content and the pH of the soil	7
8.3.5 Handling and storage of the soil	7
8.4 Start-up and execution of the test	7
9 Calculation and expression of results	9
9.1 Calculation	9
9.1.1 Percentage biodegradation from oxygen consumption values	9
9.1.2 Percentage biodegradation from carbon dioxide evolved	9
9.2 Expression and interpretation of results	10
10 Validity of results	10
11 Test report	11
Annex A (informative) Principle of a manometric respirometer (example)	12
Annex B (informative) Example of a system for measuring the amount of carbon dioxide evolved	13
Annex C (informative) Examples of methods for the determination of evolved carbon dioxide	15
Annex D (informative) Theoretical oxygen demand (ThOD)	17
Annex E (informative) Example of a determination of the amount and the molecular mass of water-insoluble polymer remaining at the end of a biodegradation test	18
Annex F (informative) Examples of long-term tests	19
Annex G (informative) Round-robin testing	23
Bibliography	27

ISO/DIS 17556:2018(E)

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 on 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 the following URL: www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 17556:2012), which has been technically revised. The main changes are as follows:

- a) unit for BOD, COD and DIC has been corrected (see [Clause 3](#));
- b) the formula for calculating the percent biodegradation was modified (see [Subclause 9.1.1](#));
- c) the test period was revised to two years at the longest (see [Clause 4](#));
- d) the number of replicates was corrected to three (see [Subclause 9.2](#)).

Introduction

A number of plastic materials and products have been designed for applications ending up in or on soil. They have been developed for applications where biodegradation is beneficial from a technical, environmental, social or economic standpoint. Examples can be found in agriculture (e.g. mulching film), horticulture (e.g. twines and clips, flower pots, pins), funeral items (e.g. body bags), recreation (e.g. plastic “clay” pigeons for shooting, hunting cartridges), etc. In many cases, recovery and/or recycling of these plastic items is either difficult or not economically viable. Various types of biodegradable plastics have been developed which have been designed to biodegrade and disappear *in situ* at the end of their useful life. Several International Standards specify test methods for determining the ultimate aerobic or anaerobic biodegradation of plastic materials in aqueous or compost conditions. Considering the use and disposal of biodegradable plastics, it is important to establish a test method to determine the ultimate aerobic biodegradation of such plastic materials in soil.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN ISO 17556:2019

<https://standards.iteh.ai/catalog/standards/sist/ea12624b-3c19-4fa2-962a-f50c947f9532/sist-en-iso-17556-2019>

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

WARNING — Appropriate precautions should be taken when handling soil because it might contain potentially pathogenic organisms. Toxic test compounds and those whose properties are unknown should be handled with care.

1 Scope

This document specifies a method for determining the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a closed respirometer or the amount of carbon dioxide evolved. The method is designed to yield an optimum degree of biodegradation by adjusting the humidity of the test soil.

If a non-adapted soil is used as an inoculum, the test simulates the biodegradation processes which take place in a natural environment; if a pre-exposed soil is used, the method can be used to investigate the potential biodegradability of a test material.

This method applies to the following materials:

- natural and/or synthetic polymers, copolymers or mixtures of these;
- plastic materials which contain additives such as plasticizers or colorants;
- water-soluble polymers.

It does not necessarily apply to materials which, under the test conditions, inhibit the activity of the microorganisms present in the soil. Inhibitory effects can be measured using an inhibition control or by another suitable method. If the test material inhibits the microorganisms in the soil, a lower test material concentration, another type of soil or a pre-exposed soil can be used.

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 10381-6, *Soil quality — Sampling — Part 6: Guidance on the collection, handling and storage of soil under aerobic conditions for the assessment of microbiological processes, biomass and diversity in the laboratory*

ISO 10390, *Soil quality — Determination of pH*

ISO 10634, *Water quality — Guidance for the preparation and treatment of poorly water-soluble organic compounds for the subsequent evaluation of their biodegradability in an aqueous medium*

ISO 10694, *Soil quality — Determination of organic and total carbon after dry combustion (elementary analysis)*

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

ISO/DIS 17556:2018(E)

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:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1
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

3.2
biochemical oxygen demand
BOD
 mass concentration of dissolved oxygen consumed under specified conditions by the aerobic biological oxidation of a chemical compound or organic matter

Note 1 to entry: It is expressed as milligrams of oxygen uptake per kilogram of test soil.

3.3
dissolved organic carbon
DOC
 that part of the organic carbon in water which cannot be removed by specified phase separation

Note 1 to entry: It is expressed as milligrams of carbon per litre.

Note 2 to entry: Typical means of separation are centrifugation at $40\,000\text{ m}\cdot\text{s}^{-2}$ for 15 min or membrane filtration using membranes with pores of diameter $0,2\text{ }\mu\text{m}$ to $0,45\text{ }\mu\text{m}$.

3.4
theoretical oxygen demand
ThOD
 maximum theoretical amount of oxygen required to oxidize a chemical compound completely, calculated from the molecular formula

Note 1 to entry: It is expressed as milligrams of oxygen uptake per milligram or gram of test compound.

3.5
theoretical amount of evolved carbon dioxide
ThCO₂
 maximum theoretical amount of carbon dioxide evolved after completely oxidizing a chemical compound, calculated from the molecular formula

Note 1 to entry: It is expressed as milligrams of carbon dioxide evolved per milligram or gram of test compound.

3.6
lag phase
 time, measured in days, from the start of a test until adaptation and/or selection of the degrading microorganisms is achieved and the degree of biodegradation of a chemical compound or organic matter has increased to about 10 % of the maximum level of biodegradation

3.7
biodegradation phase
 time, measured in days, from the end of the lag phase of a test until about 90 % of the maximum level of biodegradation has been reached

3.8**maximum level of biodegradation**

degree of biodegradation of a chemical compound or organic matter in a test, above which no further biodegradation takes place during the test

3.9**plateau phase**

time from the end of the biodegradation phase until the end of the test

Note 1 to entry: It is measured in days.

3.10**pre-conditioning**

pre-incubation of soil under the conditions of the subsequent test in the absence of the chemical compound or organic matter under test, with the aim of improving the performance of the test by acclimatization of the microorganisms to the test conditions

3.11**pre-exposure**

pre-incubation of soil in the presence of the chemical compound or organic matter under test, with the aim of enhancing the ability of the soil to biodegrade the test material by adaptation and/or selection of the microorganisms

3.12**water content**

mass of water which evaporates from the soil when the soil is dried to constant mass at 105 °C, divided by the dry mass of the soil

Note 1 to entry: This is simply the ratio between the mass of the water and that of the soil particles in a soil sample.

3.13**total water-holding capacity**

mass of water which evaporates from soil saturated with water when the soil is dried to constant mass at 105 °C, divided by the dry mass of the soil

3.14**total organic carbon****TOC**

amount of carbon bound in an organic compound

Note 1 to entry: It is expressed as milligrams of carbon per 100 mg of the compound.

4 Principle

This method is designed to yield the optimum rate of biodegradation of a plastic material in a test soil by controlling the humidity of the soil, and to determine the ultimate biodegradability of the material.

The plastic material, which is the sole source of carbon and energy, is mixed with the soil. The mixture is allowed to stand in a flask over a period of time during which the amount of oxygen consumed (BOD) or the amount of carbon dioxide evolved is determined. Provided the CO₂ evolved is absorbed, the BOD can be determined, for example, by measuring the amount of oxygen required to maintain a constant gas volume in a respirometer flask, or by measuring either automatically or manually the change in volume or pressure (or a combination of the two). An example of a suitable respirometer is shown in [Annex A](#). The amount of carbon dioxide evolved is measured at intervals dependent on the biodegradation kinetics of the test substance by passing carbon-dioxide-free air over the soil and then determining the carbon dioxide content of the air by a suitable method. Examples of suitable methods are given in [Annexes B](#) and [C](#).

The level of biodegradation, expressed as a percentage, is determined by comparing the BOD with the theoretical oxygen demand (ThOD) or by comparing the amount of carbon dioxide evolved with

ISO/DIS 17556:2018(E)

the theoretical amount (ThCO_2). The influence of possible nitrification processes on the BOD has to be considered. The normal test period is six months. The test may be shortened or extended until the plateau phase (see [3.9](#)) is reached, but the total test period shall not exceed two years.

Unlike ISO 11266, which is used for a variety of organic compounds, this document is specially designed to determine the biodegradability of plastic materials.

5 Test environment

Incubation shall take place in the dark or in diffused light in an enclosure which is free from vapours toxic to microorganisms and is maintained at a temperature constant to within ± 2 °C in the range between 20 °C and 28 °C, preferably 25 °C.

6 Materials

6.1 Distilled water, containing less than 2 mg of DOC per litre.

6.2 Carbon dioxide absorber, preferably soda lime pellets.

7 Apparatus

Ensure that all glassware is thoroughly cleaned and, in particular, free from organic or toxic matter.

7.1 Closed respirometer, including test flasks and all other necessary equipment, located in a constant-temperature enclosure or in a thermostatically controlled apparatus (e.g. a water-bath). An example is described in [Annex A](#).

Any respirometer capable of determining with sufficient accuracy the biochemical oxygen demand is suitable, preferably an apparatus which measures and automatically replaces the oxygen consumed so that no oxygen deficiency and no inhibition of the microbial activity occurs during the degradation process.

7.2 Apparatus for measuring the amount of carbon dioxide evolved

7.2.1 Test flasks: glass vessels (e.g. conical flasks or bottles), fitted with tubing impermeable to carbon dioxide to allow purging with gas, and located in a constant-temperature enclosure or in a thermostatically controlled apparatus (e.g. a water-bath).

7.2.2 CO₂-free-air production system, capable of supplying CO₂-free air at a flow rate of several ml/min to each test flask, held constant to within ± 10 % (see example of system, including test vessels, in [Annex B](#)). Alternatively, the incubation apparatus shown in ASTM D5988 may be used.

7.2.3 Analytical equipment for accurately determining carbon dioxide. Typical examples are a carbon dioxide IR analyser, a dissolved inorganic carbon (DIC) analyser, apparatus for titrimetric determination after complete absorption in a basic solution (see [Annex C](#)), and apparatus for the gravimetric determination of carbon dioxide in accordance with ISO 14855-2.

7.3 Analytical balance.

7.4 pH-meter.