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Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions — Method by analysis of evolved carbon dioxide —

Part 2: Gravimetric measurement of carbon dioxide evolved in a laboratory-scale test

Détermination de la biodégradabilité aérobie ultime des matériaux plastiques dans des conditions contrôlées de compostage — Méthode par analyse du dioxyde de carbone libéré —

Partie 2: Mesurage gravimétrique du dioxyde de carbone libéré lors d'un essai de laboratoire

Reference number
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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 document 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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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 14, *Environmental aspects,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 14855-2:2018), which has been technically revised.

The main changes are as follows:

— the NDIR method to the measurement of evolved carbon dioxide in addition to the conventional gravimetric method has been added;

— a schematic diagram of the measurement device and an example of biodegradability measurement using an NDIR sensor have been added to Annex D.

A list of all parts in the ISO 14855 series can be found on the ISO website.

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.

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Introduction

Management of plastics waste is a serious problem in the world. Plastics recovery technologies include material recovery (mechanical recycling, chemical or feedstock recycling, and biological or organic recycling) and energy recovery (heat, steam or electricity as a substitute for fossil fuels or other fuel resources). The use of biodegradable plastics is one valuable recovery option (biological or organic recycling).

Several ISO standards for determining the ultimate aerobic/anaerobic biodegradability of plastic materials have been published. In particular, ISO 14855-1 is a common test method that measures the amount of carbon dioxide evolved using methods such as continuous infrared analysis, gas chromatography or titration.

Compared with ISO 14855-1, the amounts of compost inoculum and test sample used in this document are one-tenth the size. In order to ensure the activity of the compost inoculum, inert material that gives the mixture the same texture as soil is mixed into the inoculum. The carbon dioxide evolved from the test vessel is determined by absorbing it in a carbon dioxide trap and carrying out gravimetric analysis of the absorbent. The method described in this document, which uses a closed system to capture the carbon dioxide evolved, can also be used to obtain valuable information, by means of isotopic-labelling studies, on the way in which the molecular structure of co-polymers degrades.

In addition to traditional gravimetric methods, this test method adds a new way to measure carbon dioxide using spectroscopic non-dispersive infrared (NDIR) technology, significantly reducing the routine work and chemical use required for quantitative measurement without compromising the quantitative accuracy of chemical methods.

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Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions — Method by analysis of evolved carbon dioxide —

Part 2: Gravimetric measurement of carbon dioxide evolved in a laboratory-scale test

WARNING — Sewage, activated sludge, soil and compost can contain potentially pathogenic organisms. Therefore, appropriate precautions should be taken when handling them. 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 under controlled composting conditions by gravimetric measurement of the amount of carbon dioxide evolved. The method is designed to yield an optimum rate of biodegradation by adjusting the humidity, aeration and temperature of the composting vessel.

The method applies to the following materials:

— natural and/or synthetic polymers and copolymers, and mixtures of these;

— plastic materials that contain additives such as plasticizers or colorants;

— water-soluble polymers;

— materials that, under the test conditions, do not inhibit the activity of microorganisms present in the inoculum.

NOTE If the test material inhibits microorganisms in the inoculum, another type of mature compost or pre-exposure compost 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 11721-1, *Textiles — Determination of resistance of cellulose-containing textiles to micro-organisms — Soil burial test — Part 1: Assessment of rot-retardant finishing*

ISO 14855-1, *Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions — Method by analysis of evolved carbon dioxide — Part 1: General method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
compost
organic soil conditioner obtained by biodegradation of a mixture principally consisting of various vegetable residues, occasionally with other organic material and having a limited mineral content

3.2
composting
aerobic process designed to produce compost

3.3
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

3.4
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 of the same sample

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

3.5
ultimate aerobic biodegradability
breakdown ratio by expressed as percentage 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 14852:2021, 3.1]

3.6
theoretical amount of evolved carbon dioxide
$ThCO_2$
maximum theoretical amount of carbon dioxide evolved after completely oxidizing a chemical compound, calculated from the molecular formula and expressed as milligrams of carbon dioxide evolved per milligram or gram of test compound

3.7
lag phase
time from the start of a test until adaptation and/or selection of the degradation 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

Note 1 to entry: It is measured in days.

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

Note 1 to entry: It is measured as a percentage.

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3.9
biodegradation phase
time from the end of the lag phase of a test until about 90 % of the maximum level of biodegradation has been reached

Note 1 to entry: It is measured in days.

3.10
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.

3.11
pre-exposure
pre-incubation of an inoculum in the presence of the chemical compound or organic matter under test, with the aim of enhancing the ability of the inoculum to biodegrade the test material by adaptation and/or selection of the micro-organisms

3.12
pre-conditioning
pre-incubation of an inoculum 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 test by acclimatization of the microorganisms to the test conditions

3.13
water-holding capacity
WHC
mass of water that 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

4 Principle

This method is designed to yield the optimum rate of biodegradation of a plastic material in mature compost by controlling the humidity, aeration ratio and temperature in the composting vessel. It also aims to determine the ultimate biodegradability of the test material by using a small-scale reactor. The degradation rate is periodically measured by determining the mass of the evolved carbon dioxide using an absorption column filled with soda lime and soda talc on an electronic balance.

The test material is mixed with an inoculum derived from mature compost and with an inert material such as sea sand. The sea sand plays an active part by acting as a holding body for humidity and microorganisms. Examples of suitable test arrangements are presented in Annexes A and B. The amount of carbon dioxide evolved is measured at intervals on an electronic balance and the carbon dioxide content is determined using the following method. The derivation of the formula used to calculate the degree of biodegradation from the amount of carbon dioxide evolved is given in Annex C. In this method, the degree of biodegradation, expressed as a percentage, is calculated by comparing the amount of carbon dioxide evolved with the theoretical amount ($ThCO_2$).

The test is terminated when the plateau phase of biodegradation has been attained. The standard time for termination is 45 days, but the test can be continued for up to six months.

5 Reagents

Use only analytical-grade reagents. Use only deionized water.

5.1 **Soda lime**, particle size between 2 mm and 4 mm, for CO_2 absorption.

5.2 **Anhydrous calcium chloride**, particle size between 2 mm and 3 mm, for water absorption.

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