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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

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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).

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 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, *Plastics and environment*.

This second edition cancels and replaces the first edition (ISO 14855-2:2007), which has been technically revised. It also incorporates the Technical Corrigendum ISO 14855-2:2007/Cor.1:2009.

The main changes compared to the previous edition are as follows.

— The correct values for the particle size of soda talc given in the Technical Corrigendum 1 ISO 14855-2:2007/Cor.1:2009 have been adopted.

— The following numbers of composting vessels have been provided:

a) three test vessels for the test mixture;

b) three vessels for blank controls;

c) three vessels for checking inoculum activity using a reference material.

— The next criterion has been added to the list of validity criteria in Clause 10:

c) the inoculum in the blank has produced more than 50 mg but less than 150 mg of carbon dioxide per gram of volatile solids (mean values) after 10 days of incubation.

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.

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.

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 may 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.

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.

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

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.

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 could 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.

5.3 Sodium hydroxide on a talc support (commonly known as soda talc), particle size between 2 mm and 3 mm, for CO_2 absorption.

5.4 Silica gel (with moisture indicator), particle size between 2 mm and 4 mm, for water absorption.

5.5 Sea sand, particle size between 20 mesh and 35 mesh.

5.6 Reference material: thin-layer chromatography (TLC) grade microcrystalline cellulose with a particle size of less than 20 µm, for use as the reference material in the positive control.

6 Apparatus

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

6.1 Air-supply system, capable of supplying each composting vessel with carbon-dioxide-free, water-saturated air.

The air can be prepared by supplying compressed air through a carbon dioxide trap and a humidifier (see examples in Annexes A and B), i.e. columns filled with soda lime and water, respectively. The air flow rate shall be controlled with a flow controller so that it is high enough for aerobic conditions.

6.2 Composting vessels

Use bottles or columns that ensure a supply of water-saturated, carbon-dioxide-free air to the contents. A suitable volume is 500 ml. If the loss in mass of the test material is to be determined, weigh each composting vessel empty before starting the test.

6.3 System for the determination of carbon dioxide, capable of determining carbon dioxide directly from the change in mass of a carbon dioxide trap. The carbon dioxide trap shall consist of columns filled with soda lime, soda talc and anhydrous calcium chloride. The calcium chloride should preferably be in a separate column from the soda lime and soda talc (see examples in Annexes A and B). An ammonia trap (dilute sulfuric acid) and a water trap (silica gel and calcium chloride) are required between the composting vessel and the carbon-dioxide-absorbing column.

6.4 Gas-tight tubes, used to connect the composting vessels to the air supply and the carbon dioxide measurement system.

6.5 pH-meter, used for measurement of the pH of the test mixture. It shall be accurate to 0,1 pH-units or better.

6.6 Analytical equipment, used for the determination of the dry solids (at 105 °C), volatile solids (at 550 °C) and total organic carbon (TOC), for elemental analysis of the test material and, if required, for the determination of dissolved inorganic carbon (DIC), volatile fatty acids, oxygen in the air, water content and total nitrogen.

6.7 Balance, used to periodically measure the mass of the carbon-dioxide-absorbing column, in order to determine the amount of carbon dioxide evolved, and also to measure the mass of the composting vessel containing compost and test material. A top-loading electronic balance with a display reading down to 10 mg and a capacity greater than 500 g is preferred.

6.8 Thermostatic-control unit, required to maintain the temperature of the composting vessels at a controlled temperature during the test (see examples given in Annexes A and B). It shall be capable of maintaining the temperature of the composting vessels constant to within ±2 °C.

6.9 Composting bioreactor. A box, made from polypropylene or another suitable material, having a size that allows the contents to be stirred easily with a spatula. The box shall be provided with a tightly fitting lid to avoid excessive water loss. Three holes with a diameter of about 1 cm shall be made at equal distances along the centreline of the lid. These holes allow air to enter and gases to leave the box, as well as the gradual evaporation of excess water.

7 Procedure

7.1 Preparation of the inoculum

Well-aerated compost from a properly operating aerobic composting plant shall be used as the inoculum. The inoculum shall be homogeneous and free from large inert objects such as glass, stones or pieces of metal. Remove such items manually and then sieve the compost on a screen of about 3 mm mesh.

Compost can be made as follows. Wood shavings, sawdust, used mushroom beds, chaff or rice straw can be used as the carbon source. Livestock excrement is added as a source of composting microorganisms and mineral salt nutrients. This is placed in a container with a volume of about 1 m^3 and mixed well. It is recommended that the compost be adjusted to a carbon/nitrogen (C/N) ratio of 15 and a carbon/phosphorous (C/P) ratio of 30. Insufficient phosphorous and nitrogen levels can be supplemented using calcium superphosphate and ammonium magnesium phosphate hexahydrate or urea respectively. Water is added to reach a water content equal to 65 %. The C/N, C/P and water-content values may also be adjusted to other values, determined by experience, depending on seasonal variations and climatic differences. The compost should be removed from the container once a week to turn it and add water if necessary, before returning it to the container to continue the composting process. The age of the compost should preferably be between two and four months.

Normally, non-exposed inoculum is preferred, especially in the case of standard tests simulating biodegradation behaviour in real composting facilities. Depending on the purpose of the test, however, pre-exposed compost may be used, provided that this is clearly stated in the test report (e.g. percent biodegradation = X %, using pre-exposed compost) and provided the method of pre-exposure is detailed in the test report.

Determine the total dry solids and volatile solids content of the compost inoculum. The total dry solids should be between 35 % and 55 % of the wet solids and the volatile solids more than 30 % of the dry solids. Adjust the water content, if necessary, before the compost is used by adding water or drying gently, e.g. by aerating the compost with dry air.

Prepare a mixture of 1 part of inoculum to 5 parts of deionized water. Mix by shaking and measure the pH immediately. It should be between 7,0 and 9,0.

For further characterization of the inoculum, suitable parameters such as the content of total organic carbon, total nitrogen or fatty acids can optionally be determined at the beginning and the end of the test.

Check the activity of the inoculum during the test by means of a biodegradable reference material and by measuring carbon dioxide evolution in the blank vessels. The reference material shall be degraded by 70 % or more at the end of the test. The inoculum in the blank should produce between 50 mg and 150 mg of carbon dioxide per gram of volatile solids over the first 10 days of the test. If the production of carbon dioxide is too high, stabilize the compost by aeration for several days before using it in a new test.

7.2 Preparation of the sea sand

Dip the sea sand in tap water. After removing floating impurities by decantation, rinse the sand sufficiently, drain off the water and dry the sand at about 105 °C.

NOTE Sea sand is an inert product that contains more than 90 % of SiO_2. It plays an important role, however, in maintaining an appropriate water content and as a support for microbial growth.

7.3 Preparation of test material and reference material

Determine the total organic carbon (TOC) of the test material and the reference material using, for example, ISO 8245 and report it preferably as grams of TOC per gram of total dry solids. Alternatively, provided that the materials do not contain inorganic carbon, it is possible to determine the carbon content by elemental analysis. For this, the test material should contain sufficient organic carbon to yield carbon dioxide in an amount suitable for determination. Normally, a minimum of 10 g of total dry solids containing 4 g of TOC is required per 500 ml-vessel.