
**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 aérobique 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

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 14855-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 14855 consists of the following parts, under the general title *Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions — Method by analysis of evolved carbon dioxide*:

- Part 1: General method
- Part 2: Gravimetric measurement of carbon dioxide evolved in a laboratory-scale test

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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 part of ISO 14855 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 part of ISO 14855, 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.

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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 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 part of ISO 14855 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 micro-organisms present in the inoculum.

If the test material inhibits micro-organisms in the inoculum, another type of mature compost or pre-exposure compost can be used.

2 Normative references

The following referenced documents are indispensable for the application 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 5663, *Water quality — Determination of Kjeldahl nitrogen — Method after mineralization with selenium*

ISO 8245, *Water quality — Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*

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.

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 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

ThCO₂

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, measured in days, from the start of a test until adaptation and/or selection of the degradation micro-organisms 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

3.8

maximum level of biodegradation

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

3.9

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

3.10

plateau phase

time, measured in days, from the end of the biodegradation phase until the end of the test

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 micro-organisms 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 micro-organisms. 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 equation 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 (ThCO₂).

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 CO₂ 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 2 mm, for CO₂ 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: TLC (thin-layer chromatography) 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

The air-supply system shall be 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

This system shall be 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

These tubes are used to connect the composting vessels to the air supply and the carbon dioxide measurement system.

6.5 pH-meter

The pH-meter is 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

This equipment is 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

The balance is 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

This unit is 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 composting bioreactor is 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 is used as the carbon source. Livestock excrement is added as a source of composting micro-organisms and mineral salt nutrients. This is placed in a container with a volume of about 1 m³ 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 levels can be supplemented using calcium superphosphate. 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 SiO₂. It plays an important role, however, in maintaining an appropriate water content and as a support for microbial growth.