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

AMENDMENT 1: Use of activated vermiculite instead of mature compost

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

AMENDEMENT 1: Utilisation de vermiculite activée à la place de compost mature

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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.

Amendment 1 to ISO 14855:1999 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

Introduction

The method specified in ISO 14855:1999 uses a solid-phase respirometric test system based on mature compost used as a solid bed, a source of nutrients, and an inoculum rich in thermophilic microorganisms. Mature compost is a very heterogeneous and complex material. Therefore, it can be difficult to quantify the residual polymeric material left in the bed at the end of the test, to detect possible low-molecular-mass molecules released into the solid bed by the polymeric material during degradation, and to assess the biomass. As a result, it can be difficult to perform a complete carbon balance. Another difficulty which is sometimes encountered with mature compost is a “priming effect”: the organic matter present in large amounts in the mature compost can undergo polymer-induced degradation, known as the “priming effect”, which affects the measurement of the biodegradability.

To overcome these difficulties and to improve the reliability of the method, the mature compost can be replaced by a solid mineral medium which is used as the composting bed, thus facilitating analyses. The method can be used to measure the biodegradation in terms of CO_2 evolution, to quantify and analyse the biomass and the residues of polymeric material left in the solid bed at the end of the test, and to perform a complete carbon balance. Furthermore, the method is not sensibly affected by the priming effect and can, therefore, be used to assess materials known to cause this problem with mature compost. The mineral bed can also be subjected to an ecotoxicological analysis to verify the absence of any ecotoxic activity in the bed after biodegradation.

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

AMENDMENT 1: Use of activated vermiculite instead of mature compost

Page 1, Clause 1:

Add the following paragraph after the first paragraph:

"Subclauses 8.6 and 8.7 specify a variant of the method, using a mineral bed (vermiculite) inoculated with thermophilic microorganisms obtained from compost with a specific activation phase, instead of mature compost. This variant is designed to yield the percentage of carbon in the test substance converted to carbon dioxide and the rate of conversion."

Page 2, Clause 3:

Add the following definition:

3.11
activated vermiculite
vermiculite colonized by an active microbial population during a preliminary growth phase

Page 2, Clause 4

Add, at the end of this clause, the following text:

Vermiculite should be used instead of mature compost

a) whenever the determination of the degree of biodegradation is affected by a priming effect induced by the test material

and/or

b) when performing a final carbon balance with biomass determination and retrieval of the residual test material.

The vermiculite bed, being inorganic, substantially reduces the priming effect, thus improving the reliability of the method. A further advantage of using vermiculite is the very small amount of carbon dioxide evolved in the blank vessels (nearly zero), because of the low level of microbial activity. This permits low levels of degradation activity to be evaluated precisely.

The mineralization rates obtained with the activated vermiculite are identical, or very similar, to those obtained with mature compost, both in terms of the final degradation level and the degradation rate.

Page 3, Clause 6

Replace the text of this clause with the following:

6.1 TLC (thin-layer chromatography) grade cellulose

Use TLC (thin-layer chromatography) grade cellulose with a particle size of less than 20 µm as the positive-control reference material.

6.2 Vermiculite

Vermiculite is a clay mineral used for building purposes, known to be particularly suitable as a microbial carrier, allowing survival and full activity of microbes. The composition of the native mineral, before heat treatment, is Al_2O_3 10 %, MgO 30 %, CaO 5 %, SiO_2 50 % and combined H_2O 5 %. When the mineral is subjected to heat treatment, it loses the combined water and expands, giving "expanded vermiculite". Expanded vermiculite in flake form shall be used. Expanded vermiculite has a large capacity for water storage, and a water content comparable with that of mature compost can be obtained in the bed.

Vermiculite can be classified into three types, as follows:

"Concrete" type: apparent density 80 kg/m^3 ± 16 kg/m^3 (at the time the material is put into sacks); particle size: 80 % between 12 mm and 4 mm, 2 % passing through a 0,5 mm sieve.

"Medium" type: apparent density 90 kg/m^3 ± 16 kg/m^3; particle size: 80 % between 6 mm and 1 mm, 2 % passing through a 0,5 mm sieve.

"Fine" type: apparent density 100 kg/m^3 ± 20 kg/m^3; particle size: 80 % between 3 mm and 0,7 mm, 5 % passing through a 0,5 mm sieve.

For the purposes of this International Standard, the concrete type is used.[1)]

Page 3, Clause 7

Add, at the end of this clause, the following subclause:

7.9 Bioreactors for activation of the vermiculite: Containers, with a volume between 5 l and 20 l, which are not actively aerated. The containers shall be closed in such a way as to avoid excessive drying out of the contents. Openings shall, however, be provided to allow gas exchange with the atmosphere and ensure aerobic conditions throughout the activation phase.

An example of a suitable bioreactor is a box, made of polypropylene or another suitable material, having the following dimensions: 30 cm × 20 cm × 10 cm (l, w, h). The box shall have a tightly fitting lid in order to avoid excessive loss of water vapour. In the middle of the two 20-cm-wide sides, a hole 5 mm in diameter shall be made at a height of about 6,5 cm from the bottom of the box. It is these two holes which allow gas exchange between the atmosphere inside the box and the outside environment.

1) A possible source of this type of vermiculite is BPB plc, Park House, 15 Bath Road, Slough SL1 3UF, UK (www.bpb.com). This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the source named.

Page 6, Clause 8

Add the following two subclauses:

8.6 Activation of vermiculite

The vermiculite is activated by inoculating it with a solution containing both organic and inorganic nutrients and mature compost. The composition of the inoculum solution used shall be as given in Tables 1, 2 and 3. The ratio of vermiculite to inoculum solution shall be 1:3 (mass/volume).

Prepare the compost extract used in the inoculum solution by mixing mature compost with deionized water (20 % mass/volume) for about half an hour, then filtering the slurry with a strainer (aperture size about 1 mm). A further filtration through filter paper or centrifugation at about 1 000 rpm for 15 min can then be performed.

Table 1 — Composition of 1 l of inoculum solution

Constituent	Mineral solution (see Table 2)	Suitable nutrient broth	Urea	Corn starch	Cellulose	Compost extract
Amount	500 ml	13 g	5,8 g	20 g	20 g	500 ml

Table 2 — Composition of 1 l of mineral solution

Chemical	KH_2PO_4	$MgSO_4$	$CaCl_2$ (10 % solution)	NaCl (10 % solution)	Trace-element solution (see Table 3)
Amount	1 g	0,5 g	1 ml	1 ml	1 ml

Table 3 — Composition of 1 l of trace-element solution

Chemical	H_3BO_3	KI	$FeCl_3$	$MnSO_4$	$(NH_4)_6Mo_7O_{24}$	$FeSO_4$
Amount	500 mg	100 mg	200 mg	400 mg	200 mg	400 mg

Mix the necessary amounts of vermiculite and inoculum solution to give a homogeneous mixture, and dispense the mixture into the bioreactors (about 1 kg of mixture in each). Weigh each bioreactor with its contents and incubate at (50 ± 2) °C for three/four days.

Reweigh the bioreactors daily and, if necessary, bring the mass back to its original value by adding chlorine-free tap water, deionized water or distilled water. In addition, mix the contents of each bioreactor daily with a spatula or an ordinary spoon to ensure aeration.

Vermiculite treated in this way is referred to as “activated vermiculite” and can be placed in the composting vessels for use as a solid bed instead of the mature-compost inoculum (see 8.1). For normal assessments, use 800 g of activated vermiculite in each composting vessel.

The amounts of activated vermiculite and test material used in the test will depend on the size of the composting vessels. The ratio between the dry mass of the activated vermiculite and the dry mass of the test material should preferably be about 4:1. About half of the volume of the composting vessel should be filled with the test mixture. Sufficient headspace is required to be able to manually shake the test mixture.

For normal assessments, use composting vessels which have a volume of about 3 l. Weigh out an amount of activated vermiculite corresponding to 200 g of dry solids and an amount of test material corresponding to 50 g of dry solids, and mix well before introducing the mixture into the vessels.

8.7 Recovery procedure, carbon balance

At the end of the test, the vermiculite beds can be extracted to recover and determine quantitatively the amount of test material remaining and the amounts of degradation by-products and/or biomass present. The bed in each composting vessel can be analysed independently or the contents of all the composting vessels in a series pooled and analysed together. The values obtained for the amount of biomass, the amount of test material remaining and the amount of by-products can be used, along with the amount of carbon evolved as CO_2 during the test, to perform a final carbon balance. The amount of carbon present in the original test material is compared with the amount of carbon evolved as CO_2 during the test, the amount of carbon transformed into biomass, and the amount of carbon in the remaining test material and in the degradation by-products, at the end of the test. In this way, it is possible to validate the result obtained for the degree of biodegradation.

The extractions can be performed in sequence using water and/or organic solvents, depending on the nature of the test material. For this purpose, carry out preliminary solubility trials on the test material to choose a suitable solvent.

Analytical procedures which can be used are spectroscopy (IR, UV-Visible, NMR, etc.), chromatography, gravimetric analysis, elemental analysis, etc. These procedures can be applied directly to the extracts and/or to concentrates of the extracts. The extracts can also be subjected to ecotoxicological testing.

Page 8, Clause 11

Add, at the end of this clause, the following items:

j) information on the source, type and amount of vermiculite used;

k) if carried out, the results of the carbon balance determination.

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Add the following Bibliography:

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