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Plastics — Determination of the ultimate anaerobic biodegradation of plastic materials in controlled slurry digestion systems — Method by measurement of biogas production

Plastiques — Évaluation de la biodégradabilité anaérobie ultime des matériaux plastiques dans des systèmes de digestion de boue contrôlés — Méthode par mesurage de la production de biogaz

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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).

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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 second edition cancels and replaces the first edition (ISO 13975:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

— at least following numbers of test vessels have been provided;

  — three test vessels for the test mixture;

  — three vessels for blank controls;

  — three vessels for checking inoculum activity using a reference material;

— the Clausius-Clapeyron equation has been collected (see Formula D.1).

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

Biological recycling (biorecycling), together with mechanical recycling and chemical recycling, is a viable option for the recovery of plastic waste. This document specifies a method of evaluating the anaerobic biodegradability of such waste in a controlled anaerobic slurry system. This is a representative anaerobic digestion test method and system for biodegradable plastic waste.

The production of a biogas is observed under anaerobic conditions suitable for the growth of thermophilic or mesophilic microorganisms. The biogas is collected in a bag under atmospheric pressure, and the biogas volume is measured with a syringe or a gas burette. The biodegradability of the test material is evaluated from the sum of the amount of carbon dioxide dissolved in the supernatant and the cumulative quantity of evolved biogas. This document describes a biodegradation test method for plastic materials in a controlled anaerobic slurry system. It differs from ISO 15985 which uses high-solids anaerobic digestion conditions, and ISO 14853 which uses an aqueous system in an anaerobic environment.

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Plastics — Determination of the ultimate anaerobic biodegradation of plastic materials in controlled slurry digestion systems — Method by measurement of biogas production

WARNING — Sewage sludge and other organic waste might contain potentially pathogenic organisms. Therefore, appropriate precautions should be taken when handling such materials. Digestion of organic materials produces flammable gases that present fire and explosion risks. These gases also contain toxic chemicals, including hydrogen sulfide and ammonia, in substantial concentrations. Appropriate safety measures, such as the use of a draft chamber, gas masks and/or well-ventilated laboratory facilities, should be taken. Toxic test chemicals and chemicals whose properties are not known should be handled with care and in accordance with safety instructions. Care should be taken when transporting and storing quantities of organic matter undergoing digestion.

1 Scope

This document specifies a method of evaluating the ultimate anaerobic biodegradability of plastic materials in a controlled anaerobic slurry digestion system with a solids concentration not exceeding 15 %, which is often found for the treatment of sewage sludge, livestock faeces or garbage. The test method is designed to yield a percentage and rate of conversion of the organic carbon in the test materials to carbon dioxide and methane produced as biogas.

The method applies to the following materials, provided they have a known carbon content:

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

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

— water-soluble polymers.

It does not apply to materials which exhibit inhibitory effects on the test microorganisms at the concentration chosen for the test.

NOTE Inhibitory effects can be determined by an inhibition test (e.g. ISO 13641-1 or ISO 13641-2).

2 Normative references

There are no normative references in this document.

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 http://www.electropedia.org/

3.1
ultimate anaerobic biodegradation
breakdown of an organic compound by microorganisms in the absence of oxygen into carbon dioxide, methane, water and mineral salts of any other elements present (mineralization) plus new biomass

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3.2
digested sludge
mixture of the settled sewage and activated sludge which has been incubated in a mesophilic or thermophilic anaerobic digester to reduce the biomass and odour and to improve the dewaterability of the sludge

Note 1 to entry: Digested sludge contains an association of anaerobic fermentative and methanogenic microorganisms producing carbon dioxide and methane.

3.3
slurry
watery mixture of insoluble matter

Note 1 to entry: The suspended-solids concentration of a slurry might be as high as around 15 %, but slurry is fluid and pumpable.

3.4
dissolved inorganic carbon
DIC
carbon dioxide dissolved in water or transformed into carbonic acid, hydrogen carbonate ion and carbonate ion

3.5
total dry solids
amount of solids obtained by taking a known volume of test material or inoculum and drying at about 105 °C to constant mass

3.6
volatile solids
amount of solids obtained by subtracting the residue of a known volume of test material or inoculum after incineration at about 550 °C from the *total dry solids* (3.5) of the same test portion

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

3.7
theoretical amount of evolved biogas
ThBiogas
maximum theoretical amount of biogas (CH_4 + CO_2) which will evolve after complete biodegradation of an organic compound under anaerobic conditions

Note 1 to entry: ThBiogas is calculated from the molecular formula and expressed as litres of biogas evolved per gram of test material under the standard conditions.

3.8
lag phase
time from the start of an anaerobic digestion 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* (3.11)

Note 1 to entry: It is measured in days.

3.9
biodegradation phase
time from the end of the *lag phase* (3.8) of a test until about 90 % of the *maximum level of biodegradation* (3.11) has been reached

Note 1 to entry: It is measured in days.

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3.10
plateau phase
time from the end of the *biodegradation phase* (3.9) until the end of the test

Note 1 to entry: It is measured in days.

3.11
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 in percent.

4 Principle

This test method is designed to determine the biodegradability of plastic materials under anaerobic conditions in a slurry system. The methanogenic inoculum is obtained from an anaerobic digester operating on sewage sludge or, alternatively, on organic waste such as livestock faeces or garbage. The test material mixed with the inoculum is anaerobically incubated in a test vessel at a pre-selected temperature for a period normally of 60 days. The test period may be shortened or extended until a plateau phase is reached, but the total period shall not exceed 90 days. The digestion temperature shall be (55 ± 5) °C in order to simulate thermophilic anaerobic digestion. Alternatively, the digestion temperature may be set at (35 ± 3) °C in order to simulate mesophilic anaerobic digestion.

The volume of the gases produced in the test vessel, carbon dioxide (CO_2) and methane (CH_4), is measured. A considerable amount of CO_2 will also be dissolved in the digested sludge or dissociated to hydrogen carbonate ion and carbonate ion under the conditions of the test. This dissolved inorganic carbon (DIC) is measured at the end of the test. The amount of biogas produced is calculated from the volume of biogas collected and the amount of DIC formed in excess of blank values.

The percentage biodegradation is calculated as the ratio of the sum of the net increase of produced biogas and DIC to the theoretical amount of evolved biogas (ThBiogas). The biodegradation curve can be followed by making intermediate measurements of biogas production.

5 Test and reference materials

5.1 Test material, the test material is normally added directly as solids to give a concentration of volatile solids in the range of 7 g/l to 10 g/l. The test material should preferably be used in powder form or as film.

5.2 Reference material, thin-layer chromatography (TLC) grade microcrystalline cellulose with a particle size < 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. The usual laboratory equipment are required, including the following.

6.1 Digestion vessel.

Use conical or other suitable glass flasks with gastight connectors and gas-impermeable tubing. A minimum volume of 1,5 l is recommended in view of the requirements of 7.3.

6.2 Gas volume measurement system.

Use a gas-sampling bag to collect the biogas evolved. A gastight syringe or a gas burette should preferably be used to measure the volume of the gas in the bag. The water in contact with the gas shall

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