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Plastics — Determination of the ultimate anaerobic biodegradation and disintegration under high-solids anaerobic-digestion conditions — Method by analysis of released biogas

Plastiques — Évaluation de la biodégradation anaérobie ultime et de la désintégration dans des conditions de digestion anaérobie à teneur élevée en solides — Méthode par analyse du biogaz libéré

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 15985 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

Introduction

New types of plastic are being developed in which biodegradability is a specifically sought-for characteristic. These plastics and derived products can be added to or used as feedstock for biological recycling and recovery in aerobic composting plants or anaerobic biogasification plants. To make sure these plastics are fit for biological recycling, their biodegradability must be demonstrated, preferably by standard test methods.

Standard test methods which determine the degree of biodegradation under aerobic, high-solids conditions have been developed (e.g. ISO 14855). However, it is well known from the literature that the degree of biodegradation can differ significantly depending on the environmental conditions such as the presence or the absence of oxygen (aerobic or anaerobic). To have a complete understanding of the biodegradation characteristics of a plastic under these different environmental conditions, various methods are required.

This International Standard specifies a method for the determination of the ultimate anaerobic biodegradation of plastic materials under high-solids conditions. This is representative of systems for the anaerobic biogasification of the organic fraction of municipal solid waste. Another method for determining the degree of anaerobic biodegradation is ISO 11734. However, this method is designed for soluble test materials in aqueous test conditions and at low concentrations (typically detergents) which is not typical of plastics. In addition, it is not possible to determine the degree of disintegration in an aqueous method.

Plastics — Determination of the ultimate anaerobic biodegradation and disintegration under high-solids anaerobic-digestion conditions — Method by analysis of released biogas

1 Scope

This International Standard specifies a method for the evaluation of the ultimate anaerobic biodegradability of plastics based on organic compounds under high-solids anaerobic-digestion conditions by measurement of evolved biogas and the degree of disintegration at the end of the test. This method is designed to simulate typical anaerobic digestion conditions for the organic fraction of mixed municipal solid waste. The test material is exposed in a laboratory test to a methanogenic inoculum derived from anaerobic digesters operating only on pretreated household waste. The anaerobic decomposition takes place under high-solids (more than 20 % total solids) and static non-mixed conditions. The test method is designed to yield the percentage of carbon in the test material and its rate of conversion to evolved carbon dioxide and methane (biogas).

The conditions described in this International Standard may not always correspond to the optimum conditions for the maximum degree of biodegradation to occur.

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

3 Terms and definitions

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

3.2
disintegration
physical breakdown of a material into very small fragments

3.3
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

3.4
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 content of the same sample

NOTE The volatile solids content is an indication of the amount of organic matter present.

3.5
lag phase
time, measured in days, from the start of a 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

3.6
maximum level of biodegradation
degree of biodegradation, measured in per cent, of a chemical compound or organic matter in a test, above which no further biodegradation takes place during the test

3.7
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

3.8
plateau phase
time, measured in days, from the end of the biodegradation phase until the end of the test

4 Principle

The test method is designed to be an optimized simulation of an intensive anaerobic digestion process and determines the ultimate biodegradability and degree of disintegration of a test material under high-solids anaerobic digestion conditions. The methanogenic inoculum is derived from anaerobic digesters operating on pretreated household waste, preferably only the organic fraction.

The test material is mixed with the inoculum and introduced into a static digestion vessel where it is intensively digested under optimum temperature and moisture conditions for a test period of 15 days or longer until a plateau in net biodegradation has been reached.

During the anaerobic biodegradation of the test material, methane, carbon dioxide, water, mineral salts and new microbial cellular constituents (biomass) are the ultimate biodegradation products. The biogas (methane and carbon dioxide) evolved is continuously monitored or measured at regular intervals in test and blank vessels to determine the cumulative biogas production. The percentage biodegradation is given by the ratio of the amount of biogas evolved from the test material to the maximum theoretical amount of biogas that can be produced from the test material. The maximum theoretical amount of biogas produced is calculated from the measured total organic carbon (TOC). This percentage biodegradation does not include the amount of carbon converted to new cell biomass which is not metabolized in turn to biogas during the course of the test.

Additionally, the degree of disintegration of the test material is determined at the end of the test and the loss in mass of the test material may also be determined.

5 Test environment

Incubation shall be in the dark or in diffused light in an enclosure that is maintained at a constant temperature of 52 °C ± 2 °C.

6 Reagents

Use only analytical-grade reagents.

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

7 Apparatus

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

Required is usual laboratory equipment, plus the following:

7.1 Digestion vessels: conical or other suitable glass flasks connected up so that no loss of gas occurs.

A minimum volume of 750 ml is recommended in view of the requirements of 8.2 and 8.3.

Weigh each empty digestion vessel if the loss in mass of the test material is to be determined.

7.2 Gas volume measurement system, comprising an inverted graduated cylinder or plastic column in water or another suitable device for measuring gas volume. The water in contact with the gas shall be at a pH of less than 2 during the whole period of the test to avoid CO_2 loss through dissolution in the water. The gas volume measuring device, as well as the gas tubing, shall be of sufficient quality to prevent gas migration and diffusion between the system and the surrounding air.

7.3 Apparatus for gas analysis (optional), comprising a gas chromatograph, or other apparatus, equipped with a suitable detector and column(s) for measuring the methane and carbon dioxide concentration in the evolved gases.

7.4 Analytical apparatus (optional), for determining volatile fatty acids by aqueous-injection chromatography, as well as total Kjeldahl nitrogen, ammonia nitrogen, dry solids (at 105 °C) and volatile solids (at 550 °C).

8 Procedure

8.1 Preparation of the inoculum

The inoculum shall be obtained from a properly operating anaerobic digester using pretreated household waste as the sole feedstock. The pretreated household waste should preferably come from an existing waste treatment facility that is treating municipal solid waste where, through sorting, shredding, sieving or other means, a fairly homogeneous organic fraction is produced with a particle size of less than 60 mm.

The digester should have been operating for a period of at least 4 months using this organic fraction, with a retention time of a maximum of 30 days under thermophilic conditions (52 °C ± 2 °C). Gas evolution yields should be at least 15 ml of biogas at standard temperature and pressure per gram of dry solids in the digester and per day on average for at least 30 days.

The inoculum should preferably be derived from a digester operating under dry (> 20 % total solids) conditions. It can, however, also be derived from a wet fermentation process, the digested sludge being dewatered by centrifugation, by using a press or by drying at a maximum temperature of 55 °C to a total solids content of at least 20 %.

The prepared inoculum should undergo a short post-fermentation of approximately 7 days at the same operating temperature as that of the facility from which it was derived. This means that the inoculum is not fed but allowed to postferment anaerobically by itself. This is to ensure that large, easily biodegradable particles are degraded during this period and also to reduce the background level of degradation of the inoculum itself.