SLOVENSKI STANDARD
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Določanje aerobne biološke razgradnje popolnoma formuliranih maziv v vodni raztopini - Preskusna metoda na podlagi maziv, ki porabljajo O2 - Poročilo o študiji

Determination of aerobic biological degradation of fully formulated lubricants in an aqueous solution — Test method based on O2-consumption Lubricants – study report

Bestimmung des aeroben biologischen Abbaus von fertig formulierten Schmierstoffen in wässriger Lösung - Prüfverfahren auf der Grundlage des O2-Verbrauchs von Schmierstoffen - Studienbericht

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (FprCEN/TR 18172:2025) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

This document is currently submitted to the Vote on TR.

This document has been prepared under a Mandate M/430 of the European Commission, addressed to CEN for the development of European standards for bio-lubricants in relation to bio-based product aspects. It has been prepared by CEN/TC 19/WG 33 “bio-lubricants”, the secretariat of which is held by DIN.

Introduction

Mandate M/430 [1] of the European Commission addressed to CEN the development of a definition standard for bio-lubricants. Currently, EN 16807 [2] is the active standard to define a bio-lubricant.

EN 16807 defines a bio-lubricant as a lubricant fulfilling a minimum set of environmental requirements, namely biodegradability, aquatic toxicity and content of renewable resources together with minimum technical requirements depending on the application of the lubricant.

The aim of the definition standard was to create a transparent definition for a bio-lubricant and easy-to-test parameters to enable customers to monitor products claiming to be a bio-lubricant, to ensure they fulfil its requirements, independent of their composition or from third parties' control.

For this reason, it is necessary to establish test methods which allow for the testing of these parameters on finished formulated products.

Regarding the content of renewable resources and aquatic toxicity, existing standard test methods are already suitable for finished formulated products. Due to the broad variety of water-soluble, emulsifiable and poorly water-soluble organic substances these standard test methods offer various preparation procedures enabling the testing of complex mixtures such as finished formulated products. In contrast, existing test methods for measuring aerobic biodegradation are optimised for organic substances rather than finished formulated products.

In TC19/WG 33 a new test method for measuring the biodegradation of finished formulated products, namely EN 17181 [3], has been developed based on ISO 9439 [4] (itself based on OECD 301B [5]). The new method includes improvements to sample preparation in order to overcome the difficulties of testing poorly water-soluble organic substances, and aims to provide a standard method for measuring aerobic biodegradation of finished formulated products with the best possible precision.

Additionally, it has been noted that most existing test methods lack a published precision statement. Based on this new standard test method EN 17181, and with significant financial support from industrial partners, an interlaboratory study was performed to determine the reproducibility (R) and the repeatability (r) of this new test method.

Annex A provides an example of biodegradation curves

1 Scope

This document describes the background of test method EN 17181, its improvements compared to the existing standard test methods for measuring aerobic biodegradation of organic substances and the planning and execution of the interlaboratory study .

A variety of data collected in the development of the method and the interlaboratory study is provided in this report, thereby supporting future activity associated with bio-lubricants and the definition of standard requirements.

Aerobic biodegradation resulting in mineralisation of the organic material can be determined by measuring released CO_2 using an appropriate analytical technique.

EN 17181 specifies a procedure for determining the degree of aerobic degradation of organic material in fully formulated bio-lubricants. This organic material is exposed in a synthetic aqueous medium under laboratory conditions to an inoculum prepared from activated sludge. In contrast to existing test methods measuring released CO_2 this new method uses a precise sample preparation procedure for poorly water-soluble organic material. Nevertheless, EN 17181 is also applicable to measuring the aerobic degradation of organic material present within fully formulated lubricants which are water-soluble or emulsifiable.

NOTE In general, EN 17181 can be used to determine biodegradation of finished formulated lubricants, which contain biobased material of unknown amount or do not contain biobased material at all.

All results and statistical data evaluation in this document are based on fresh water as test medium. Tests in sea water are currently not part of EN 17181.

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.

3.1
organic material
total amount of all organic compounds present in a fully formulated lubricant.

3.2
aerobic biodegradation
consumption of organic materials by microorganisms in a biochemical process using oxygen resulting in cleavage of chemical bonds and carbon dioxide (CO_2) production providing energy and/or new biomass.

3.3
primary biodegradation
structural change (transformation) of an organic chemical compound by microorganisms resulting in the loss of a specific property.

3.4
mineralisation / ultimate biodegradation
aerobic biodegradation of organic material by microorganisms to yield carbon dioxide, water and mineral salts of any other elements present (mineralization) and new biomass

3.5
activated sludge
biomass produced in the aerobic treatment of wastewater by the growth of bacteria and other microorganisms in the presence of dissolved oxygen

3.6
inoculum
sample of activated sludge used in degradation procedures described in this method

3.7
reference compound
organic compound of known biodegradability with a degradation degree of more than 60%

3.8
dissolved organic carbon
DOC
part of the organic carbon in water which cannot be removed by specified phase separation, for example by centrifugation at 4000 rpm for 15 min or by membrane filtration using membranes with pores of 0,2 µm to 0,45 µm diameter

3.09
theoretically released amount of carbon dioxide
$ThCO_2$
theoretical maximum amount of CO_2 released from total oxidation of a lubricant, calculated from TOC content, expressed in this case as milligrams of CO_2 evolved per milligram or gram of test compound

3.10
total organic carbon
TOC
amount of carbon bound in an organic compound

Note 1 to entry: Refer to ISO 8245 for further details<

3.11
lag phase
time from the start of a test until adaptation and selection of the degrading microorganisms is achieved and the biodegradation degree of a chemical compound or organic matter has increased to about 10 % of the theoretical maximum biodegradation

Note 1 to entry: It is expressed in days.

3.12
maximum level of biodegradation
Maximum biodegradation degree 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 expressed as a percentage

3.13
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 expressed in days

3.14
plateau phase
time from the end of the biodegradation phase when the maximum level of biodegradation is reached until the end of the test

3.15
pre-conditioning
pre-incubation of an inoculum under the conditions of the subsequent test in the absence of the test chemical compound or organic matter, with the aim of improving the performance of the test by acclimatization of the microorganisms to the test conditions

3.16
pre-exposure / pre-adaption
pre-incubation of an inoculum in the presence of the test 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 microorganisms

3.17
repeatability
r
difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, within a short interval of time, under the same test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, can be calculated using the following function:

$r = fr(x)$

where x is the average of the two test results being compared

3.18
repeatability
R

difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, under different test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, can be calculated using the following function:

$R = fR(x)$

where x is the average of the two test results being compared.

4 Background

Test procedures to detect the elimination of surfactants in waste-waters were developed in the 1970s [6] with the aim of reducing the amount of visible foaming in surface waters. These methods measured the elimination of material by determination of the DOC and only water-soluble material could be determined with sufficient precision.

To enable measurement of the degradation of non-water soluble two-stroke outboard engine oils this method was adapted for lubricants in the so-called “Zürich-Workshops” [7] in 1982 and was published by the Coordinating European Council (CEC) as CEC L-33-T-82. This test method describes the elimination of the test material during the microbiological degradation process and measured primary biodegradation. As an alternative approach to describe biodegradation in the aqueous environment, it was the first test for finished formulated lubricants.

With ongoing discussions about the fate of chemicals in the environment it wasn't sufficient to assume the disappearance of chemical substances by measuring primary degradation. Therefore, in 1981, OECD adopted 5 screening test methods for ready biodegradation to measure the potential of a chemical compound to undergo ultimate biodegradation in aqueous environment. These were OECD Guidelines 301 A to E [8] and another version of these methods, OECD 301 F, was added to this set in 1992.

In 1988, an interlaboratory study was performed. Thirty-seven laboratories took part in an OECD ring-test of methods for determining ready biodegradability. Four different organic substances were tested using six different test methods. Despite ring-test coordinator´s recommendation to develop more reliable methods for the determination of CO_2 and even though no precision statement have been reported, OECD 301 test-methods have since been established in different industries and applications [9].

Other biodegradation test methods have been evaluated later under the regime of ISO following the principles laid down in OECD 301:

— ISO 9408 [10],

— ISO 9439 and

— ISO 14593 [11].

ISO 14593 includes a precision statement.

5 Principle of biodegradation testing

The extent of the mineralisation of organic materials is determined using aerobic microorganisms in a static aqueous test system. The test system consists of a mineral medium, an inoculum and the organic material, which is the sole source of carbon and energy for the microorganisms.

This mixture is stirred in test flasks and aerated using CO_2-free air for a maximum time of 28 days. The amount of carbon dioxide evolved from biological degradation is collected in external flasks, determined using a suitable analytical process, and expressed as a percentage of $ThCO_2$.

In order to correct for the CO_2 production due to the inoculum itself (i.e. endogenous respiration), blank flasks solely containing inoculum and test medium are run in parallel to determine the amount of CO_2 which has to be subtracted from the test run.

Additionally, test flasks with a reference substance are run in parallel in order to verify the activity of the inoculum and possibly, to assess any toxic effects of the test sample on the inoculum.

6 Disturbance variables

6.1 General

The test equipment and the test protocol introduce sources of variance which can influence the precision of the experiment. The testing of replicate samples together with the use of a blank control and a reference substance minimise the impact of some of these variables so that the test result can be considered reliable and precise.

Nevertheless, certain parameters turned out not to be controlled in a sufficient way by the standard procedures.

6.2 Discontinuous measurement

Test methods based on determining CO_2 production typically are not performed using continuously operating analysis equipment, and measurements are therefore taken periodically over the test period often by different individuals. This can introduce variances depending on the type of measurement,