SLOVENSKI STANDARD
SIST EN 17181:2019

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Maziva - Določevanje aerobne biološke razgradnje polno formuliranih maziv v vodni raztopini - Preskusna metoda, ki temelji na proizvodnji CO2

Lubricants - Determination of aerobic biological degradation of fully formulated lubricants in an aqueous solution - Test method based on CO2-production

Flüssige Mineralölerzeugnisse - Bestimmung des aerobe biologischen Abbaus von vollständig formulierten Schmierstoffen in wässriger Lösung - Prüfverfahren mittels CO2-Produktion

Bio-lubrifiants - Détermination de la biodégradabilité aérobie, en solution aqueuse, de lubrifiants complètement formulés - Méthode basée sur le dégagement de CO2

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75.100	Maziva	Lubricants, industrial oils and related products

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English Version

Lubricants - Determination of aerobic biological degradation of fully formulated lubricants in an aqueous solution - Test method based on CO2-production

Bio-lubrifiants - Détermination de la biodégradabilité aérobie, en solution aqueuse, de lubrifiants complètement formulés - Méthode basée sur le dégagement de CO2

Schmierstoffe - Bestimmung der aeroben biologischen Abbaubarkeit ausformulierter Schmiermittel in wässrigem Medium - Testmethode basierend auf CO2-Bestimmung

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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Contents

Page

European foreword

This document (EN 17181:2019) 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 European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2019, and conflicting national standards shall be withdrawn at the latest by August 2019.

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Introduction

Existing test methods for determination of aerobic biodegradation of organic substances in an aqueous medium (e.g. numerous ISO and OECD test methods) are widely used. Moreover, all actually existing ecolabels, international and national regulations and recommendations are working with them.

Testing of organic substances requires a broad scope of preparation tools to maintain comparable results for all different materials appearing liquid, solid, water-soluble, non-water-soluble or emulsifiable.

In consequence of this, results may differ regarding the choice of test method, preparation procedure or inoculum thus leading to low levels of reproducibility and repeatability.

Fully formulated lubricants (oils or greases or emulsions) are complex mixtures of mostly organic compounds that are mainly water insoluble. Based on ISO 9439 (resp. OECD 301B) this test method has been aligned by modifying sample preparation and test procedure for such complex mixtures in order to achieve improved significance of test results. The test method described in this document, however, was not assessed for greases. The precision statement therefore only applies for lubricating oils.

Finally, from customer's point of view a biodegradation test method for the final product of improved significance will result in better market transparency, enhanced reputation of "bio-lubricants" and confidence of the customer in this product group.

1 Scope

This document specifies a procedure for determining the degree of aerobic degradation of fully formulated lubricants. The organic material in a fully formulated lubricant is exposed in a synthetic aqueous medium under laboratory conditions to an inoculum from activated sludge.

Biodegradation resulting in mineralisation of the organic material can be determined by measuring released CO_2 in a total organic carbon (TOC) analyser. In contrast to existing test methods measuring released CO_2 this method uses a precise preparation procedure for non-water soluble organic material.

The above mentioned method applies to fully formulated lubricants which

a) are water-soluble, non-water soluble or emulsifiable, and

b) are not toxic and not inhibitory to the test microorganisms at the test concentration.

The presence of inhibitory effects is determined as specified in this document.

This test method is focused on fresh water as test medium. Tests in sea water are currently not included in this method, but may be introduced later.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 9439, *Water quality — Evaluation of ultimate aerobic biodegradability of organic compounds in aqueous medium — Carbon dioxide evolution test*

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:

• IEC Electropedia: available at http://www.electropedia.org/

• ISO Online browsing platform: available at http://www.iso.org/obp

3.1
organic material
total amount of all organic compounds a fully formulated lubricant consists of

3.2
aerobic biodegradation
consumption of organic materials by microorganisms in a biochemical process under use of oxygen, resulting in cleavage of chemical bonds and CO_2 production providing energy and/or new biomass

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

3.4
mineralisation
aerobic biodegradation of organic material by microorganisms to yield carbon dioxide, water and mineral salts of any other elements present 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
concentration of suspended solids of an activated sludge
amount of solids obtained by filtration or centrifugation of a known volume of activated sludge and drying at about 105 °C to constant mass

3.7
inoculum
sample of activated sludge for the purpose of this test method used in degradation procedures described in this method

3.8
reference compound
organic compound of known biodegradability with a degradation degree of more than 60 %

3.9
dissolved organic carbon
DOC
part of the organic carbon in water which cannot be removed by specified phase separation

Note 1 to entry For example by centrifugation at 4 000 rpm for 15 min or by membrane filtration using membranes with pores of 0,2 µm to 0,45 µm diameter.

3.10
total inorganic carbon
TIC
total amount of inorganic carbon in a water sample arising from carbon dioxide and carbonates

3.11
dissolved inorganic carbon
DIC
part of the inorganic carbon in water which cannot be removed by specified phase separation

Note 1 to entry For example by centrifugation at 4 000 rpm for 15 min or by membrane filtration using membranes with pores of 0,2 µm to 0,45 µm diameter.

3.12
theoretically released amount of carbon dioxide
$ThCO_2$
theoretical maximum amount of carbon dioxide released from a chemical compound, calculated from TOC content

Note 1 to entry Expressed in this case as milligrams of carbon dioxide evolved per milligram or gram of test compound.

3.13
total organic carbon
TOC
amount of carbon bound in an organic compound

Note 1 to entry Refer to ISO 8245 [17] for further details.

3.14
biochemical oxygen demand
BOD
mass concentration of dissolved oxygen consumed under specified conditions by the aerobic biological oxidation of a chemical compound or organic matter in water

Note 1 to entry Expressed in this case as milligrams oxygen uptake per milligram or gram of test compound.

3.15
chemical oxygen demand
COD
mass concentration of oxygen equivalent to the amount of a specified oxidant consumed by a chemical compound or organic matter when a water sample is treated with that oxidant under defined conditions

Note 1 to entry Expressed in this case as milligrams oxygen consumed per milligram or gram of test compound.

3.16
theoretical oxygen demand
ThOD
theoretical maximum amount of oxygen required to oxidize a chemical compound completely, calculated from elemental analysis

Note 1 to entry Expressed in this case as milligrams of oxygen uptake per milligram or gram of test compound.

3.17
lag phase
time from the start of a test until adaptation and selection of the degrading microorganisms are 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 This is expressed in days.

3.18
maximum level of biodegradation
maximum biodegradation degree of a chemical compound in a test, above which no further biodegradation takes place during the test

Note 1 to entry This is expressed in percentage.

3.19
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 This is expressed in days.

3.20
plateau phase
time from the end of the biodegradation phase when the maximum level of biodegradation is reached until the end of the test

3.21
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

3.22
pre-exposure
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

4 Principle

Biodegradation is followed over a specified period by measuring the production of carbon dioxide CO_2. The evolution of carbon dioxide (CO_2) is determined and compared with the theoretical carbon dioxide evolution ($ThCO_2$) using the CO_2 evolution test as in ISO 9439.

The mineralisation process of a fully formulated lubricant (see also Annex A) is determined using aerobic microorganisms in a static aqueous test system. The test system contains of a mineral medium, activated sludge obtained from a waste water treatment plant of mainly domestic waste as the inoculum and the organic material (the sole source of carbon and energy).

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 will be collected in external flasks, determined using a suitable analytical process, and expressed as a percentage of $ThCO_2$.

If a substance-specific analytical method is available then information on the primary degradability may be obtained additionally for water-soluble, non-volatile lubricants only.

5 Test environment

Incubation shall take place in the dark or diffused light, at a temperature within a range 20 °C to 25 °C which shall not vary by more than ±1 °C during the test.

6 Reagents

Reagents of analytical grade should be used only.

6.1 Water, preferably of distilled or de-ionized quality, with a DOC content of less than 1 mg/l.

6.2 Stock solutions for mineral medium, as follows:

Stock solution (a)	
Potassium dihydrogen orthophosphate, KH_2PO_4	8,50 g
Dipotassium hydrogen orthophosphate, K_2HPO_4	21,75 g
Disodium hydrogen orthophosphate dihydrate, $Na_2HPO_4 \cdot 2H_2O$	33,40 g
Ammonium chloride, NH_4Cl	0,50 g
Dissolve in water and make up to 1 l. The pH of the solution should be 7,4.	
Stock solution (b)	
Calcium chloride, anhydrous, $CaCl_2$ or Calcium chloride dihydrate, $CaCl_2 \cdot 2H2O$	27,50 g 36,40 g
Dissolve in water and make up to 1 l.	
Stock solution (c)	
Magnesium sulphate heptahydrate, $MgSO_4 \cdot 7H_2O$	22,50 g
Dissolve in water and make up to 1 l.	
Stock solution (d)	
Iron (III) chloride hexahydrate, $FeCl_3 \cdot 6H_2O$	0,25 g
Dissolve in water and make up to 1 l.	

NOTE Stock solution a) can be stabilized by the addition of one drop of concentrated HCl per litre. If any precipitate is formed during storage the stock solution is replaced by a freshly prepared one.

7 Apparatus

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

7.1 Test flasks, allowing aeration, containing hose coupling impermeable to carbon dioxide, and a method for agitation of the test sample such as shaking of the flasks or stirring inside the flasks.

7.2 Water-bath or constant temperature room (to comply with Clause 5);

7.3 Equipment for production of CO_2-free air in order to constantly aerate all test flasks with a flow rate of approximately 50 ml/min to 100 ml/min air for a maximum of 3 l medium;

7.4 Equipment for measurement of evolved carbon dioxide, consisting of a suitable instrument or analytical process of sufficient sensitivity, e.g. CO_2- or DIC-analysers or titrimetric determination of CO_2 after absorption in alkaline solution.

7.5 Centrifuge or device for filtration, capable of producing an acceleration of 4 000 rpm.