PD CEN/TS 17035:2017

BSI Standards Publication

# Surface Active Agents — Bio-based surfactants — Requirements and test methods

## National foreword

This British Standard is the UK implementation of CEN/TS 17035:2017.

The UK participation in its preparation was entrusted to Technical Committee CII/34, Methods of Test For Surfacee Active Agents.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Published by BSI Standards Limited 2017

ISBN 978 0 580 94368 3

ICS 13.020.55; 71.100.40

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2017.

**Amendments/corrigenda issued since publication**

| Date | Text affected |
|---|---|

# TECHNICAL SPECIFICATION
# SPÉCIFICATION TECHNIQUE
# TECHNISCHE SPEZIFIKATION

**CEN/TS 17035**

March 2017

ICS 13.020.55; 71.100.40

English Version

# Surface Active Agents - Bio-based surfactants - Requirements and test methods

Agents de surface - Agents tensioactifs biosourcés - Exigences et méthodes d'essais

Grenzflächenaktive Stoffe - Biobasierte Tenside - Anforderungen und Prüfverfahren

This Technical Specification (CEN/TS) was approved by CEN on 11 December 2016 for provisional application.

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Ref. No. CEN/TS 17035:2017 E

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# European foreword

This document (CEN/TS 17035:2017) has been prepared by Technical Committee CEN/TC 276 “Surface active agents”, the secretariat of which is held by AFNOR.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document has been prepared under Mandate M/491 [10] of the European Commission, addressed to CEN for the development of European Standards for solvents and surfactants in relation to bio-based product aspects. It has been prepared by CEN/TC 276/WG 3 “Bio-surfactants”, the secretariat of which is held by AFNOR.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

# Introduction

Bio-based raw materials have been used for millennia in the manufacture of surfactants, e.g. the first surfactant used by mankind, was already completely bio-based – soap. With the advent of modern surfactants in the early 20th Century, petrochemical-based raw materials also became of interest. They offered the opportunity to tune the surfactant properties, in a broader sense, to their various applications.

The last decades have seen the emergence of new bio-based raw materials for surfactants. Some of the reasons for the increased interest lie in the bio-based products' potential benefits in relation to the depletion of fossil resources and climate change.

Acknowledging the need for common standards for bio-based products, the European Commission issued Mandate M/492[1)], resulting in a series of standards developed by CEN/TC 411, with a focus on bio-based products other than food, feed and biomass for energy applications.

The standards of CEN/TC 411 "Bio-based products" provide a common basis on the following aspects:

— common terminology[2)];

— bio-based content determination;

— Life Cycle Assessment (LCA)[3)];

— sustainability aspects[4)];

— declaration tools.

It is important to understand what the term "bio-based product" covers and how it is being used. The term "bio-based" means "derived wholly or partly from biomass". It is essential to characterize the amount of biomass contained in the product by, for instance, its (total) bio-based content or bio-based carbon content.

The bio-based content of a product itself does not provide information on its environmental impact or sustainability, which may be assessed through Life Cycle Inventory (LCI), LCA and sustainability criteria. In addition, transparent and unambiguous communication within bio-based value chains is facilitated by a harmonized framework for certification and declaration.

Breaking down the horizontal standards to bio-based products like bio-based surfactants, the European Commission issued Mandate M/491, resulting in standards developed by CEN/TC 276. This Technical Specification has been developed with the aim to fulfil part of the Mandate to describe the technical requirements of bio-based surfactants. The criteria for "bio-based surfactants" published in this Technical Specification are complementary to the horizontal standards by CEN/TC 411.

Surfactants are products which have the ability to reduce interfacial/surface tension, wet surfaces, suspend materials or emulsify oils and fats. In Europe, thousands of producers, manufacturers and

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1) A mandate is a standardization task embedded in European trade laws. The M/492 Mandate is addressed to the European Standardization bodies, i.e. CEN, CENELEC and ETSI, for the development of horizontal European Standards for bio-based products. The M/491 Mandate is addressed to the development of European Standards for bio-solvents and bio-surfactants.

2) EN 16575.

3) EN 16760.

4) EN 16751.

nearly every inhabitant use surfactants every day in consumer or industrial applications. The surfactant-producing industry is composed of mainly multinationals. Downstream users are found in multinationals as well as SME's.

Surfactants may be produced from both fossil and renewable carbon feedstock (ref. EN 16575 - vocabulary). The amount of crude oil used for surfactant production is, however, low with less than 1 % of the total world's crude oil consumption.

Finally, the approach for these Technical Reports/Specifications/Standards intends to strengthen and harmonize the reputation of “bio-based surfactants” and the confidence of the customer in this product group.

# 1 Scope

This Technical Specification sets requirements for bio-based surfactants in terms of properties, limits, application classes and test methods. It lays down the characteristics and details for assessment of bio-based surfactants as to whether they:

— are fit for purpose in terms of performance related properties;

— comply with the requirements regarding the health, safety and environment which apply to general surfactants;

— are derived from a certain minimum percentage of biomass; and

— comply with at least similar sustainability criteria as comparable (non-bio-based) surfactants.

The criteria of the regulation on Registration, Evaluation, Authorization and Restriction of Chemicals (REACH) [11] also apply to bio-based surfactants.

NOTE EN 16575 defines the term “bio-based” as derived from biomass and clarifies that “bio-based” does not imply “biodegradable”. In addition, “biodegradable” does not necessarily imply the use of “bio-based” material.

# 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1772, *Surface active agents - Determination of wetting power by immersion (ISO 8022:1990 modified)*

EN 1890, *Surface active agents - Determination of cloud point of non-ionic surface active agents obtained by condensation of ethylene oxide*

EN 12458, *Surface active agents - Determination of stability in hard water*

EN 12728, *Surface active agents - Determination of foaming power - Perforated disc beating method*

EN 13955, *Surface active agents - Determination of Krafft point and solubility of ionic surface active agents*

EN 13996, *Surface active agents - Foaming power and antifoaming power - Turbine stirring method*

EN 14210, *Surface active agents - Determination of interfacial tension of solutions of surface active agents by the stirrup or ring method*

EN 14370, *Surface active agents - Determination of surface tension*

EN 14371, *Surface active agents - Determination of foamability and degree of foamability - Circulation test method*

EN 16640, *Bio-based products — Bio-based carbon content — Determination of the bio-based carbon content using the radiocarbon method*

EN 16575, *Bio-based products - Vocabulary*

EN 16751, *Bio-based products - Sustainability criteria*

EN 16760, *Bio-based products - Life Cycle Assessment*

EN ISO 14040, *Environmental management - Life cycle assessment - Principles and framework (ISO 14040)*

EN ISO 14044, *Environmental management - Life cycle assessment - Requirements and guidelines (ISO 14044)*

DIN 53902, *Testing of surface active agents; determination of foaming power, modified Ross-Miles-method*

# 3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 16575 and the following apply.

**3.1**
**surfactant**
organic substance possessing surface activity which, dissolved in a liquid, particularly water, lowers the surface or interfacial tension, by preferred adsorption at the liquid/vapour surface, or other interfaces

Note 1 to entry: "Substance" as defined in REACH [11].

[SOURCE: ISO 862:1995, Definition 1, modified — The term originally defined was "surface active agent" and "a chemical compound" is replaced here with "organic substance" at the beginning of the definition.]

**3.2**
**bio-based surfactant**
surfactant wholly or partly derived from biomass (based on biogenic carbon)

**3.3**
**bio-surfactant**
surfactant wholly based on biomass (based on biogenic carbon) produced either by chemical or biotechnological processing

**3.4**
**degradation**
transformation of a compound into smaller component parts by means of physico-chemical processes, which can occur due to abiotic processes such as oxidation and UV adsorption

**3.5**
**biodegradation**
transformation of a compound into smaller component parts by means of biological processes

**3.6**
**ultimate biodegradation**
breakdown of organic matter by micro-organisms in the presence of oxygen to carbon dioxide, water and mineral salts of any other elements present (mineralization) or in absence of oxygen to carbon dioxide, methane and mineral salts, and in both cases the production of new biomass

# 4 Generalities on surfactants

Surfactants are products which have the ability to reduce interfacial/surface tension, wet surfaces, suspend materials, or emulsify oils and fats. They make it possible to process, apply, clean or separate materials. Surfactants are widely used in consumer and professional products and for industrial applications. Surfactants are typically used on their own or in combination with other surfactants and other agents to fulfil the requirements of the respective applications.

Examples of applications for surfactants are:

— cleaning agent;

— foaming/defoaming agent;

— wetting agent;

— emulsifier;

— viscosity modifier;

— surface tension reducer;

— process aid;

— fabric softener.

NOTE For many applications to perform effectively, surfactants are essential (see for examples the website of the TEGEWA e.V. (**TE**xtilhilfsmittel" (textile auxiliaries), "**GE**rbstoffe" (tanning agents) and "**WA**schrohstoffe" (detergent raw materials))[5].

# 5 Performance of surfactants

## 5.1 Generalities related to performance

Performance and properties of any molecule, including surfactants, is determined by its chemical structure and not by the origin of its raw materials.

This section gives a common set of technical properties characterizing the performance of surfactants including bio-based surfactants. Due to the absence of international surfactant specification standards, it is necessary to provide to potential users the means to qualify the bio-based surfactant products, especially for its technical performance. Additionally, there are a number of other factors which will determine the acceptance of a surfactant such as the Health, Safety and Environmental properties which are treated in another section of this document.

Surfactants are used in such a wide variety of applications that it is not convenient to evaluate separately their performance with respect to each application. Therefore, a practical approach is to define a set of measurable surfactant properties which enable technical specialists to select appropriate surfactants for their applications.

The following six intrinsic properties characterize the basic performance of a surfactant.

5) See www.tegewa.de for a more detailed brochure about typical use of surfactants.

## 5.2 Technical performance properties

### 5.2.1 Chemical composition

The chemical composition determines the suitability of a surfactant in processes and applications.

The chemical composition is described according to the EU CLP Regulation [14].

### 5.2.2 Solubility

Solubility of surfactants in water depends very much on their hydrophobicity (HLB value[6)]), their ionic character and water hardness. Whereas ionic surfactants exhibit a lower critical solution temperature (Krafft temperature), which can be determined by EN 13955, non-ionic surfactants show an upper critical solution temperature (cloud point, EN 1890).

NOTE Some non-ionic surfactants show also a lower critical solution temperature which can also be assessed by EN 13955.

Additionally, the solubility of surfactants, especially ionic surfactants, is influenced by water hardness. EN 12458 describes a suitable test method.

### 5.2.3 Surface and interfacial tension

The chemical nature of a surface active agent and its concentration in a solvent (typically water) determines its behaviour at liquid-air interfaces (surface tension) and at liquid-liquid interfaces (interfacial tension), e.g. water and hexadecane. The degree of surface or interfacial tension reduction by addition of the surface active agent indicates the use in different applications.

EN 14370 shall be used to measure the surface tension whilst EN 14210 describes one method for evaluation of interfacial tension.

### 5.2.4 Foaming power

One key performance property of a surfactant is its foaming power. Foaming itself depends not only on the type of surfactant, but also on the conditions of application. There are also many applications where foam is contradictory for the use.

A list of standardized test methods is shown below:

— EN 12728 which determines the foaming power under high shear conditions;

— EN 13996 which determines the foaming power of surfactants under rotation conditions;

— EN 14371 which determines the foaming power by means of circulation, also useful for defoaming power;

— DIN 53902 which determines the foaming power under free-flow conditions.

NOTE There are many other application-specific foaming tests available[7)].

### 5.2.5 Wetting performance

When applying a surfactant, the first step is often the wetting of substrates. This could be either spreading across substrates like metals or penetration into substrates like textiles.

6) Griffin, W.C., J. Soc. Cosmet. Chem. 1 (1946) 311-326 ; McGowan, J.C., Tenside Surf. Det. 27 (1990) 4, 229-230

7) Foam guideline, 2007, http://www.tegewa.de/en/publications/foam-guideline.html.

A suitable method to measure the wetting power for textiles like cotton is described in EN 1772. The spreading of surfactant solutions can be determined by contact angle measurements.

NOTE There is no standard available for contact angle measurement.

### 5.2.6 Emulsion power

The use of surface active agents to stabilize emulsions is essential. As there are many different emulsion types there is no single standard available to determine the emulsion power of a surfactant or a surfactant mixture. The test methods issued are application specific.

NOTE Some examples are given in the following:

— ASTM D3709 for the temperature stability of oil-in-water emulsions,

— ASTM D4317 for certain emulsion adhesives,

— ASTM D7000 for bituminous emulsions.

## 6 Health, safety and environmental requirements

As with any chemical, the substance(s) composing the bio-based surfactant shall comply with REACH regulation [11], and with GHS/CLP regulation [13] for the labelling. In addition, a bio-based surfactant shall comply with any other EU regulations related to chemicals or surfactants.

## 7 Bio-based content

### 7.1 Generalities

Standardized determination technologies for bio-based content are based on measuring the carbon alone.

With regard to the elemental constituents that comprise surfactants, it is principally the carbon atoms that can be sourced from either fossil sources such as oil or gas, on one hand, or from recent biological material such as plants. Carbon atoms form the majority of the backbone of the surfactant molecules and the number of carbons in the molecule will alter its properties such as water solubility. The major part of the molecular weight of most surfactants is derived from the carbon content (e.g. a 12 carbon alcohol ethoxylate with 7 ethoxylate sub-units weighs 494 $g.mol^{-1}$ with 63 % of this derived from the carbon atoms).

Additionally, when processing raw feed stocks, there is the potential for some of the hydrogen and oxygen atoms to exchange with other isotopes of the same elements.

The source of carbon atoms in compounds may be determined through careful analysis of the isotopes present. Although 99 % of all carbon atoms are $^{12}C$ isotope, approximately 1 % are in the stable isotopic form of $^{13}C$. This isotope has one extra neutron in the nucleus but is stable and does not decay with time. As this carbon isotope is ~8 % heavier than $^{12}C$, in some reactions the lighter $^{12}C$ isotopic form is favoured over the heavier $^{13}C$ form and it is possible to distinguish between some sources of raw materials on this basis.

As well as the $^{13}C$ isotope, a very small proportion of the carbon in a molecule will be of the naturally occurring radioactive form, $^{14}C$, also called radiocarbon. The $^{14}C$ atoms are formed in the upper atmosphere due to interactions between cosmic rays and nitrogen atoms. The natural abundance of $^{14}C$ in compounds is around 1 part per trillion. This radioactive carbon isotope decays with a half-life of 5 730 years such that after six half-lives, it is almost undetectable in a sample. Carbon compounds that are derived from fossil sources such as oil or gas will contain no radiocarbon as it will have decayed away during the millions of years needed to make such reserves. This contrasts with recently grown plant-based materials that do contain measureable amounts of $^{14}C$.

Radioactive carbon can be measured using gas proportional counting, liquid scintillation counting, and accelerator mass spectrometry. The latter approach, AMS, is the most sensitive of the three.

There are be a number of confounding factors that make the measurement of $^{14}C$ difficult:

1) The quantity of $^{14}C$ present in a compound is small at the best of times and so a suitable quantity of material shall be available.

2) All analyses shall be conducted in such a manner as to reduce the potential contamination of the sample with other carbon compounds containing $^{14}C$.

3) The cost of such analyses is relatively high due to the small market for such analyses and the expense of the analytical equipment.

4) It can be envisioned that at some stage in the future, carbon dioxide from the combustion of fossil fuels will be captured and used as a feed stock for phytoplankton, the primary producing algae. These unicellular organisms are able to harness the energy of the sun to convert carbon dioxide into more complex compounds such as sugars, carbohydrates and fats. In such a scenario, the carbon compounds formed would have the isotopic signature of the fossil fuel although the chemicals harvested from the algae could be under any other classification called bio-based.

## 7.2 Surfactant classification

A surfactant classification system based on bio based carbon content has been developed to clarify naming used in industry. The surfactant class are shown according to the thresholds shown in Table 1.

**Table 1 — Bio-based surfactants classes**

| Surfactant class | Bio-based carbon content [a] [X % ($m/m$)] | Comments |
|---|---|---|
| Wholly bio-based surfactant | X ≥ 95 [b] | Applicable for surfactants, where all raw material are considered to be bio-based |
| Majority bio-based surfactant | 95 > X > 50 | Applicable for surfactants, where the majority of the raw material is bio-based |
| Minority bio-based surfactant | 50 ≥ X > 5 | Applicable for surfactants, where the minor part of the raw material is bio-based |
| Non bio-based surfactant | X ≤ 5 | Applicable for surfactants where no raw material is bio-based |

[a] Standardized determination technologies for bio-based content are based on carbon content. If other determination techniques come available the classes may be reviewed.

[b] To account for analytical precision and accuracy, the threshold is set at 95 % instead of 100 %.

In addition, the bio-based carbon content, as a percentage, may be reported.

## 7.3 Analytical approach

If the bio-based carbon content cannot be demonstrated through other means, the radiocarbon method EN 16640 shall be used.

NOTE When parties agree, a certificate of biomass origin can be accepted, in compliance with a Quality Assurance system.

## 8 Sustainability

To ensure that the bio-based surfactant does not have a negative impact to the environment, economy and society, it is required that the bio-based surfactant pass similar or better sustainability criteria as comparable non bio-based surfactant with similar use. The sustainability criteria shall be based upon EN 16751.

If a life-cycle assessment is part of the determination, it shall be according to EN 16760 or to EN ISO 14040 and EN ISO 14044.

The bio-based surfactant shall be based on sustainable biomass feedstock. Therefore, the economic operator shall demonstrate compliance. The compliance may be demonstrated via certification schemes and/or established traceability (segregation or mass-balance).

NOTE Definition of segregation and mass balance can be found from e.g. Roundtable on Sustainable Palm Oil www.rspo.org.

## 9 End of life

Surface active agents are generally discharged via treated and untreated wastewater into the environment. Consequently, ultimate biodegradability (mineralization) is often required for such substances. There are numerous test methods to measure biodegradability which may be used [14].

These are the recommended methodologies for determining the degradability of the bio-based surfactants. Surfactants will be considered biodegradable if the degree of biodegradability (mineralization) measured according to one of the five following tests is at least 60 % within 28 d:

— EN ISO 14593: pre-adaptation is not to be used. The ten day window principle is not applied. (Reference method).

— Method of the Directive 67/548/EEC Annex V.C.4-C [Carbon dioxide ($CO_2$) Evolution Modified Sturm Test]: preadaptation is not to be used. The ten day window principle is not applied.

— Method of the Directive 67/548/EEC Annex V.C.4-E (Closed Bottle): pre-adaptation is not to be used. The ten day window principle is not applied

— Method of the Directive 67/548/EEC Annex V.C.4-D (Manometric Respirometry): pre-adaptation is not to be used. The ten day window principle is not applied.

— Method of the Directive 67/548/EEC Annex V.C.4-F (MITI: Ministry of International Trade and Industry-Japan): pre-adaptation is not to be used. The ten day window principle is not applied.

Depending on the physical characteristics of the surfactant, one of the methods listed below can be used if appropriately justified. It should be noted that the pass criterion of at least 70 % of these methods is to be considered as equivalent to the pass criterion of at least 60 % referred to in methods listed above. The choice of the methods listed below will depend on efficacy and be decided on a case by case:

— Method of the Directive 67/548/EEC Annex V.C.4-A (Dissolved Organic Carbon DOC Die-Away): pre-adaptation is not to be used. The ten day window principle is not applied. The pass criteria for biodegradability measured according to the test shall be at least 70 % within 28 d.

— Method of the Directive 67/548/EEC Annex V.C.4-B (Modified OECD Screening-DOC Die-Away): pre-adaptation is not to be used. The ten day window principle is not applied. The pass criteria for biodegradability measured according to the test shall be at least 70 % within 28 d.

NOTE 1 The DOC methods give results on the removal and not on the ultimate biodegradation. The Manometric Respirometry and the MITI could not be appropriate in some cases because the high initial test concentration could be inhibitory.

NOTE 2 For further options, the user is also referred to work according to the Technical Report FprCEN/TR 16957 [3].

## 10 Declaration and product labelling

A bio-based surfactant shall be labelled with its class as defined in Clause 7 and optionally with the percentage of bio-based carbon content.

The performance properties as defined under 5.2 may be reported in separate documentation in case of business-to-business communication.

NOTE For additional requirements for business-to-business and business-to-consumer declarations, the user is referred to work in accordance to EN 16848 [1] and prEN 16935 [2].

# Bibliography

[1] EN 16848, *Bio-based products — Template for B2B reporting and communication of characteristics — Data sheet*

[2] prEN 16935, *Bio-based products — B2C reporting and communication — Requirements for claims*

[3] CEN/TR 16957, *Bio-based products — Guidelines for Life Cycle Inventory (LCI) for the End-of-life phase*

[4] EN ISO 14593, *Water quality - Evaluation of ultimate aerobic biodegradability of organic compounds in aqueous medium - Method by analysis of inorganic carbon in sealed vessels (CO2 headspace test) (ISO 14593)*

[5] ISO 696, *Surface active agents — Measurement of foaming power — Modified Ross-Miles method*

[6] ISO :1984, *Surface active agents — Vocabulary*

[7] ASTM D3709, *Standard Test Method for Stability of Water-in-Oil Emulsions Under Low to Ambient Temperature Cycling Conditions*

[8] ASTM D4317, *Standard Specification for Polyvinyl Acetate-Based Emulsion Adhesives*

[9] ASTM D7000, *Standard Test Method for Sweep Test of Bituminous Emulsion Surface Treatment Samples*

[10] M/491, *Mandate addressed to CEN, CENELEC and ETSI for the development of European Standards and Technical Specifications and/or Technical Reports for bio-surfactants and bio-solvents in relation to bio-based product aspects*, 7 March 2011

[11] *Regulation (EC) No 1907/2006 of the European Parliament and the Council concerning the Registration, Evaluation Authorisation and Restriction of Chemicals (REACH), establishing a European Chemicals Agency, amending Directive 1999/45/EC and repealing Council Regulation (EEC) No. 793/93 and Commission Regulation (EC) No. 1488/94 as well as Council Directive 76/769/EEC and Commission Directives 91/155/EC, 93/67/EEC, 93/105/EC and 2000/21/EC*, 18 December 2006

[12] COMMUNICATION FROM THE COMMISSION TO THE COUNCIL. the European Parliament, the European Economic and Social Committee and the Committee of the Regions, A lead market initiative for Europe, COM (2007) 860, http://eur-lex.europa.eu

[13] Council Directive 1999/13/EC of 11 March 1999 on the limitation of emissions of volatile organic compounds due to the use of organic solvents in certain activities and installations

[14] 1272/2008/EC, Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006. *OJEU.* 2008, **353** (L) pp. 1–1355

Method of the Directive 67/548/EEC Annex V.C.4-C [Carbon dioxide (CO2) Evolution Modified Sturm Test]: Preadaptation is not to be used. The ten days window principle is not applied

Method of the Directive 67/548/EEC Annex V.C.4-E (Closed Bottle): Pre-adaptation is not to be used. The ten days window principle is not applied

Method of the Directive 67/548/EEC Annex V.C.4-D (Manometric Respirometry): Pre-adaptation is not to be used. The ten days window principle is not applied

Method of the Directive 67/548/EEC Annex V.C.4-F (MITI: Ministry of International Trade and Industry-Japan): Pre-adaptation is not to be used. The ten days window principle is not applied

Method of the Directive 67/548/EEC Annex V.C.4-A (Dissolved Organic Carbon DOC Die-Away): Pre-adaptation is not to be used. The ten days window principle is not applied. The pass criteria for biodegradability measured according to the test shall be at least 70 % within twenty-eight days

Method of the Directive 67/548/EEC Annex V.C.4-B (Modified OECD Screening-DOC Die-Away): Pre-adaptation is not to be used. The ten days window principle is not applied. The pass criteria for biodegradability measured according to the test shall be at least 70 % within twenty-eight days

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