PD CEN/TR 16227:2011

BSI Standards Publication

Liquid petroleum products — Bio-lubricants — Recommendation for terminology and characterisation of bio-lubricants and bio-based lubricants

bsi.

...making excellence a habit.™

National foreword

This Published Document is the UK implementation of CEN/TR 16227:2011.

The UK participation in its preparation was entrusted to Technical Committee PTI/7, Lubricants and process fluids.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

ISBN 978 0 580 73251 5

ICS 75.100; 75.120

Compliance with a British Standard cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 30 September 2011.

Amendments issued since publication

Date	Text affected

TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

CEN/TR 16227

August 2011

ICS 75.100; 75.120

English Version

Liquid petroleum products - Bio-lubricants - Recommendation for terminology and characterisation of bio-lubricants and bio-based lubricants

Produits pétroliers liquides - Bio-lubrifiants - Recommandations pour la terminologie et la caractérisation des biolubrifiants et des lubrifiants provenant de la biomasse

Flüssige Mineralöl-Erzeugnisse - Bio-Schmierstoffe - Empfehlungen für die Terminologie und Charakterisierung von Bio-Schmierstoffen und bio-basierten Schmierstoffen

This Technical Report was approved by CEN on 14 May 2011. It has been drawn up by the Technical Committee CEN/TC 19.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. CEN/TR 16227:2011: E

Contents

Page

Foreword

This document (CEN/TR 16227:2011) 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.

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

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

The main reason of the recent interest in bio-lubricants is due to the origin (i.e. use of bio-based raw materials) or to the biodegradability of the final products, needed for instance in case of leakages or technically intended losses. The use of bio-based raw materials could be beneficial with reference to two current problems: fossil resources depletion and climate change. Today, regarding the latter issue, we have to manage the carbon in order to avoid its accumulation in the atmosphere. Efficient use of all available resources and responsible utilization of renewable carbon is a way to participate in this reduction.

Lubricants are important materials which contribute significantly to environmental protection: thanks to their tailor-made properties they reduce energy losses and wear in machines and aggregates.

The global manufacture of lubricants in all applications only uses a small part of the entire consumed mineral oil: in Europe, it only makes up around 1 %. The major fraction (> 80 %) of the residual fossil material is used for energy production, predominantly for transportation and heating purposes. Besides crude oil, biomass is an additional raw material source for lubricants.

The currently available biomass is consumed in different segments: food and feed production, power and heat generation, biofuel production and industrial applications (e. g. production of paper, fine chemicals). Due to the limited capacity of ecosystems, the utilization efficiency of renewable resources and availability issues have to be addressed across the whole bio-economy landscape. The eco-efficiency in this competitive use (e. g. energetic use vs. manufacture of goods) should always be in focus.

According to various scientists [1], it would appear appropriate to use agricultural raw materials predominantly in a cascade of uses, instead of burning them directly in furnaces or engines. That would mean, for example, first producing a bio-lubricant from biomass: around 1 t to 2 t of bio-lubricants can be produced per hectare of agriculture land. The bio-lubricant thereby stores CO_2 in the form of vegetable carbon and removes it from the atmosphere. It would be desirable to trap this CO_2 in the lubricant for as long as possible. Finally, after maximum utilization including recycling when achievable and appropriate, the lubricant can then be used either as energy source or – after re-refining – as downshifted base oil – to return the bound carbon to the natural cycle in the form of CO_2.

In order to ensure responsible and environmentally conscious use of natural (fossil and renewable) resources, a clear and unambiguous terminology is of particular importance.

The approach which is published in this report is focused on the view of the customer: *Are the referred criteria for “bio-lubricants” potentially provable for the formulated product?* The statement of this report is: *Every announcement with regard to biodegradability, toxicity and bio-based content should be measurable through the final product in hands of the customer.*

Finally, this approach intends to enhance the reputation of “bio-lubricants” and the confidence of the customer in this product group, even if no official eco-label stands for the correctness of declarations.

The criteria for "bio-lubricants" published in this Technical Report are not contrary to the European Ecolabel for Lubricants, but complementary.

1 Scope

This Technical Report gives information about bio-lubricants and recommendations for bio-lubricant (and bio-based lubricant) related terminology. These recommendations are based on a discussion of commonly used terms in this field.

This Technical Report also briefly describes the current test methods in relation to the characterization of bio-lubricants. It presents recommendations for related standards in the field of biodegradability, product functionality, impact on greenhouse gas emissions and the amount of different renewable raw materials and/or different bio-based contents used during manufacturing of such bio-lubricants forming one product group.

The criteria of the European Ecolabel for Lubricants ("EEL") [2] include the terms discussed in this paper.

NOTE 1 The European Lead Market Initiative ("LMI") [3] defines the term "bio-based" as described in Table 1. It is important to mention that "bio-based" does not imply "biodegradable". In addition, "biodegradable" does not imply the use of "bio-based" material.

NOTE 2 For the purposes of this European Technical Report, the term "% (m/m)" is used to represent the mass fraction.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
renewable resource
resource replenished by natural processes at a rate comparable to its exploitation rate

2.2
biomass
material of biological origin excluding material embedded in geological formations and/or fossilized

NOTE This definition refers to the well-known short-cycle of carbon, i. e. the life cycle of biological materials (e. g. plants, algae, marine organisms, forestry, micro-organisms, animals and biological waste from households, agriculture, animals and food/feed production).

2.3
bio-based
derived from biomass

NOTE "Biomass based", "bio-sourced", "biogenic" and "from renewable resource" are equivalent terms to bio-based.

2.4
bio-based product
product wholly or partly bio-based

NOTE The bio-based product is normally characterised by the biomass content. For the time being 25 % (*m*/*m*) is recommended as a minimum content of biomass in the final product formulation.

2.5
bio-based carbon content
amount of carbon in a sample that is of recent origin, as evidenced by its ^{14}C isotope content

NOTE 1 Materials contained in a sample are carbon-based compounds in which the element carbon is attached to other carbon atoms, hydrogen, oxygen, or other elements in a chain, ring, or three-dimensional structure.

NOTE 2 The amount of bio-based carbon in the material or product is often expressed as a percentage of the mass of the total organic carbon of the product.

NOTE 3 For developing the market for bio-based products, there is an obvious need for ways to distinguish bio-based products from non-bio-based products. As bio-based products can be made with a mix of bio-based and non-bio-based (e. g. from fossil oil) components, the bio-based content criteria is of high importance. Test methods used at present for that matter are almost always based on ^{14}C measurement as specified in the US standard test method ASTM D 6866 [12]. In Europe, such methods are currently being developed further for applications such as solid recovered fuels (EN 15440 [24]). However, these methods have not yet been applied to the whole range of bio-based products, such as liquids, and assembled products. A horizontal standard that can be adequately applied to measure bio-based carbon content in all sorts of products is hence needed.

2.6
biomass content
mass fraction of bio-based material in a sample, including all molecular ingredients of biomass, besides carbon f.e. oxygen, nitrogen or hydrogen

NOTE Claims of biomass content are difficult to verify due to lack of standards. Effectively, with ASTM D6866 [12] only the content of ^{14}C content can be measured. In contrast, no standard is actually known for the determination of the oxygen content.

2.7
biocompatible
compatible with human, animal and vegetable tissues and interface with biological systems without having toxic or negative physiological effects

2.8
biodegradable
high amount of the final formulated product will be biodegraded after a certain time

NOTE According to well accepted test methods (like OECD 301 or adequate ISO standards) a high amount of the final formulated product will be biodegraded after a certain time (in case of OECD 301 more than 60 % after 28 days).

2.9
bio-based lubricant
lubricant wholly or partly bio-based

2.10
possible impact to the environment
end-of-life aspects connected with total loss or waste

NOTE 1 Especially for the end-of-life management of bio-lubricants, it is important to differentiate between total-loss lubricants and collectable lubricants. For this reason, an indication of biodegradability via an appropriate marking is a good contribution towards more clarity.

NOTE 2 The numbers shown in Figure 2 can give an impression of the different end-of-life scenarios of lubricants.

NOTE 3 For the time being, recycling of lubricants is focussed on mineral oil; due to relatively low volumes, vegetable oils or ester oils within the collected used oils are tolerable, but not treated individually to get back the single component.

2.11
fit for purpose
fit for use
judged as usable in a specific application

NOTE In this sense, the term 'fit for purpose' describes the legal responsibility of the manufacturer, as well as the responsibility of the user. In cases where specific standards are available and accepted, those criteria should be used; the best example of this would be the International Standard ISO 15380 [14] for bio hydraulic fluids

3 Public perception

The “bio-“ prefix is often considered as a synonym of good for the environment, or in another situation, good for health. The prefix "bio", when associated with lubricants, can be perceived as an indication of biodegradability by the consumers. In other words, a “bio-lubricant” is expected to biodegrade (to break down in the environment). On the other hand, the term bio-lubricant also strongly conveys the idea of natural origin, as “bio” is taken as an indication of the biological world. An analogy is the term “biofuel” – universally taken as implying a fuel derived from renewable resources.

However, as we have seen before, all the different classes (Table 1) are actually present in the marketplace. This is a cause for concern, as it can be the source of misleading information and confusion for the final consumers.

The dissemination of confusing, ambiguous or misleading information should be prevented in order to not jeopardize the success of such schemes as well as the credibility of industry itself. Claims of biodegradability should be supported by appropriate standards.

In some cases, bio-lubricants refer to biocompatible lubricants that interface with biological systems having toxic or negative physiological effects.

Often bio-lubricants are perceived as low performance lubricants. It should be recognized, that modern high performance bio-lubricants can meet and even exceed the performance of conventional lubricants in the market.

Table 1 — common use of the term “Bio” with regard to lubricants

Origin of material	**Biodegradability**	**Example**	**The meaning of the prefix "bio-"**
Renewable	Rapidly biodegradable	Rapeseed oil, tri-methylol-propane-trioleate (TMP-O)	Biodegradable and bio-based
Non-renewable	Biodegradable	Di-isotridecyl-adipate (DITA)	Biodegradable
Renewable	Non-biodegradable	Hydrocarbons from process "Biomass-to-Liquid" (BtL)	Bio-based
Non-renewable	Non-biodegradable	White oil for food grade lubricants	Biocompatible

4 Commonly used terms

4.1 General

Ecological aspects are gaining importance in our society. Bearing in mind that our environment is becoming increasingly contaminated with all kinds of pollutants, any reduction is welcome. From an environmental point of view and compared to a number of other chemical products, lubricants are not particularly problematic. However, a large proportion of the lubricants is released into the environment either during or after use. This may be technically desired (total-loss lubrication) or a result of mishaps such as leaks, emissions, spillages or other problems.

Lubricants and functional fluids are omnipresent due to their widespread use and they thus enter the environment in small, widely-spread amounts and rarely in large, localized quantities. The terminology used in connection with "environmental compatibility" shall be split between subjective and objective criteria:

a) Subjective criteria (non-measurable):

1. environmentally friendly;
2. environmentally compatible;
3. environmentally acceptable;
4. environmentally adapted;

b) Objective criteria (measurable or provable), for example:

1. biodegradability;
2. use of renewable raw materials;
3. water solubility, water pollution;
4. ecological toxicity and physiological safety;
5. performance, approvals, oil change intervals;
6. efficiency improvements, lower energy consumption, emission reduction in use;
7. environmental awards (EEL).

Additionally, criteria of Life Cycle Assessments (LCA) are to be considered.

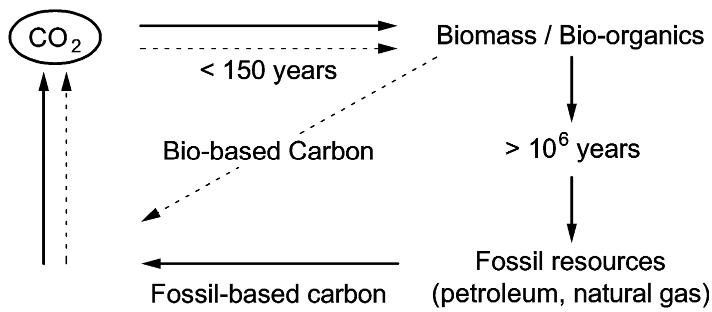
As a consequence of the current ambiguity, the same word is used to designate lubricants, products with very different properties, where all the possible combinations are present (see Table 1).

The bio-lubricants' base oils can be made from both biomass and fossil resources. Lubricants made from biomass can be rapidly, slowly, or not biodegradable; their base oils can be natural (unchanged renewable material) or synthetic (chemically modified biomass). Bio-lubricants can be a combination of both natural and synthetic base oils.

The term bio-lubricant then identifies lubricants which are derived from organic matter constituting living organisms and their residues [4]. Biomass is considered as a renewable resource. A renewable resource is replenished by natural processes at a rate comparable to its exploitation rate. The carbon content of such lubricants is derived from the so-called short carbon cycle (expected time of less than 150 years; see Figure 1, [5]). Most industrial lubricants are presently produced starting from fossil resources which are non-renewable as they cannot be replenished at a rate comparable to the exploitation rate (long carbon cycle, expected time frame to convert biomass to petroleum, gas and coal: > 10^6 years).

4.2 Current situation

Worldwide mineral oil and its derivatives are dominating the lubricants market. But this triumphant progress is limited to the last century. Historically, the friction and wear decreasing properties of natural oils and fats were well known and used in many different ways. In this respect, the development in the last 30 years of biodegradable lubricants based on natural oils is a return to traditional materials – even if the market share today only amounts to a few percent.



Key

Long carbon cycle (> 10^6 years)

Short carbon cycle (< 150 years)

Figure 1 — Global carbon cycling

The market share of “bio-lubricants” amounts to approximately 1 % in Europe. In some countries in Western Europe (Netherlands, Austria, Switzerland, Scandinavia, Germany) the market share is significantly higher, it is published for Germany at approximately 4.3 % [6].

It is assumed that about 40 % to 50 % of the lubricants sold in Europe are lost in the sense that they are not collected/not collectable. The environmental impact is largely caused by this amount of lubricants which is not properly disposed of. This figure includes total-loss applications, the residual oil in millions of oilcans and oil filters, spillages during topping-up, leaks, drips from separated oil-line and hydraulic couplings, accident losses and all manner of emission losses. Concawe Report No. 5/96 [7] gives an overview of the different kinds of loss (see Figure 2).

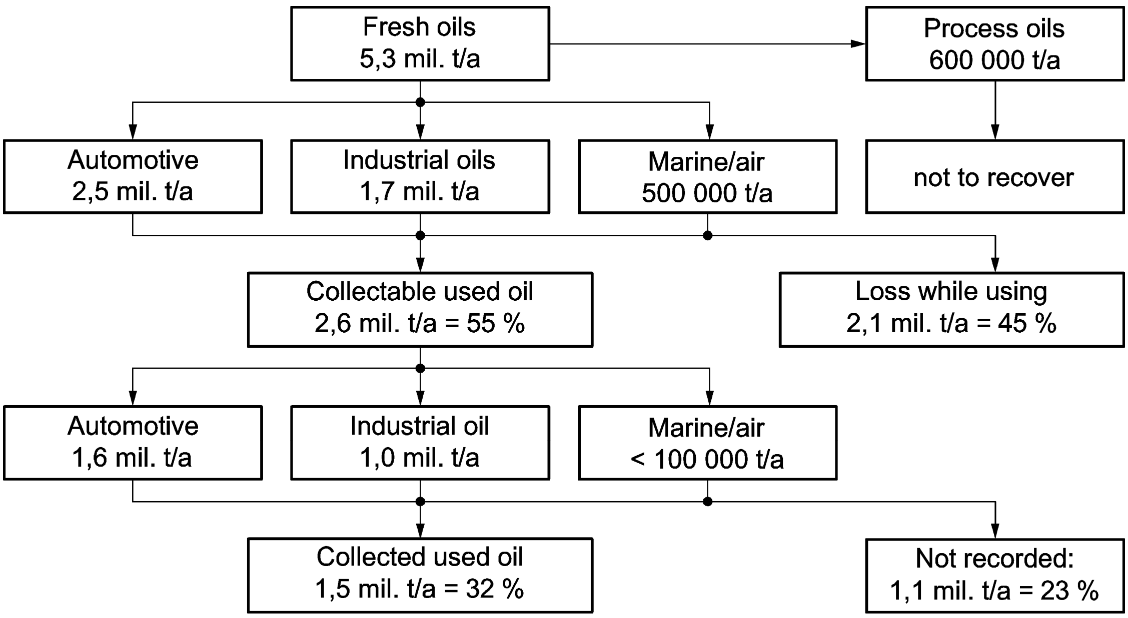


Figure 2 — Lubricants and their different kind of losses

4.3 Recommendation for terminology

4.3.1 General

According to Table 1, the term "bio-lubricant" covers several materials:

a) bio-based lubricants, when referring to raw material sourcing,

b) biodegradable lubricants, when referring to functionality, and

c) biocompatible lubricants, when referring to compatibility with human or animal body (non-toxic properties).

In this document, the term "bio-lubricant" refers to both biodegradability and relevant biomass content. To avoid ambiguity, the use of the terminology as defined in 4.3.2 is recommended.

4.3.2 Standard designation of the term "bio-lubricant" and "bio-based lubricant"

Generic terms such as bioproduct (where "product" can be substituted by e. g. lubricant) frequently appear in everyday language, mainly as marketing tools, and lubricants are far from the only offenders – detergents and plastic products are other examples of the widespread use of the bio- prefix. Without reference to clear and agreed definitions, there are many unsubstantiated claims, which can be deceptive.

Ideally they should be substituted by more accurate and more informative equivalents.

Any claim using a "bio" prefix should refer to an internationally or at least European agreed standard. A clear distinction should be made between the origin of the raw materials and their functionality. Rules should be standardised to define when such claims are justified. This is the only way to bring transparent and non-misleading information to consumers (as defined in EN ISO 14021 [15] on environmental self-claims).

The term “biomass-based”, founded on the test methods described by ASTM D 6866 [12] or equivalent, could be used to indicate products that are partly or totally made from renewable raw materials (i. e. with a biological carbon content).

The umbrella definition for *all* types of bio-lubricants should be properly designed to deliver lubricants for the intended application and for the (changing) needs of the identified market outlets, preferably at minimum costs to society. The outcome could be that a clear identification and a separate collection can be advisable to solve this concern.

Definitions and standards forming the basis for certification are in place for the biodegradability and the toxicity of lubricants. A similar system can be useful to avoid misleading claims on bio-based content.

For the sake of an unequivocal B2C (Business to Consumer) communication and, therefore, to avoid market-hindering confusion, the two pieces of information (bio-based content and biodegradability/toxicity) should be provided together.

Moreover, in order to utilize the technological potential of renewable resources, it is important to have maximum freedom for innovations. Transparency in the market without restrictive quotas and limitation in the raw material applications is a framework required for an efficient and environmentally conscious utilization of resources.

4.3.3 Minimum requirements for "bio-lubricants" and "bio-based lubricant"

For this purpose and in relevant situations where a claim for "bio-lubricants" or "bio-based lubricants" is desired or requested, a European wide standardised marking, such as the following, would be useful:

— Renewability:

Content of renewable raw material 25 % according to ASTM D 6866 [12] (radiocarbon analysis) or equivalent CEN version (to be developed).

— Biodegradability:

≥ 60 % according to OECD 301 B, C, D or F (or adequate ISO or EN standards) for oils;

≥ 50 % according to OECD 301 B, C, D or F (or adequate ISO or EN Standards) for lubricating greases;

— Toxicity:

Not to be labelled as "Dangerous to the environment" (Symbol N) according to CLP Directive 1272/2008/EC [8] (Classification, Labelling, and Packaging). This may be proven for the fully formulated product by testing according to OECD test no. 201/202/203 [13]: EC50/LC50/IC50 > 100mg/l

— Performance:

"Fit for purpose" or "Fit for use" (see 2.11). The lubricant manufacturer and the customer using the product both need to ensure that the recommended lubricant is suitable for a specific application; in other words, the appropriate specifications have to be fulfilled, including well-accepted special test procedures for ester-based lubricants. The most well known example for a special test for bio-lubricants is the '*Dry TOST*' for bio-hydraulic fluids.

It is suggested to relevant working groups in CEN (or national standardisation organisations) to consider in future revisions a "green line" into the general scheme of standards for mineral oil based lubricants. For example, the (wet) TOST (as in DIN 51524-1, -2 and -3) could be alternatively fulfilled by a 'Dry TOST' plus a hydrolytic stability test – such modified standards designated to mineral oil based lubricants could then be also used for bio-lubricants.

— Any lubricant according to the present criteria of the EU Ecolabel for Lubricants (2005/360/EC [9]) is a "Bio-lubricant" per definition.

NOTE 1 Every claim regarding biodegradability, toxicity and bio-based content should be measurable in the final product by the customer.

NOTE 2 According to the definitions of Clause 2, especially 2.9, the criterion for renewability includes the term “bio-based lubricant”.

5 Bio-lubricants

5.1 Bio-lubricants – base fluids

5.1.1 General

Liquid lubricants are characterized in many different ways. One of the most common ways is by the type of base oil used. The following list mentions the most common types:

— water;

— mineral oils;

— synthetic hydrocarbons (e. g. Polyalphaolefins, PAO);

— vegetable (natural oil);

— synthetic esters;

— polyalkylene glycols (PAG);

— phosphate esters;

- alkylated naphthalenes (AN);
- silicate esters;
- ionic fluids;
- other liquids.

Only vegetable oils and most synthetic esters are normally produced from natural resources; only these base oils are considered as bio-based oils. Other routes of synthesis, e.g. the conversion of biomass to liquid hydrocarbons (BTL), are not yet established and products are not widely available. From a theoretical point of view all chemicals mentioned below can be produced from renewable and petrochemical resources. However, from a cost-driven, practical point of view, the described routes of synthesis are the established ones for the time being – maybe one day new derivatives of renewable raw material will enter the market.

As different types of mineral oils are dominating the lubricants market today, a short overview of these base oils is given: The term “mineral oil” is used to encompass lubricating base oil derived from crude oil. The American Petroleum Institute (API) designates several types of lubricant base oils identified as:

i) Group I – Saturates < 90 % and/or sulfur > 0,03 %, and Society of Automotive Engineers (SAE) viscosity index (VI) of 80 to 120;

ii) Group II – Saturates over 90 % and sulfur under 0,03 %, and SAE viscosity index of 80 to 120;

iii) Group III – Saturates > 90 %, sulfur < 0,03 %, and SAE viscosity index above 120;

iv) Group IV – Polyalphaolefins (PAO);

v) Group V – All others not included above, such as PAG, esters, etc.

Mineral oils can also be classified into three categories depending on the prevailing compositions:

- Paraffinic;
- Naphthenic;
- Aromatic.

Synthetic hydrocarbon oils:

- Polyalpha-olefin (PAO)

Base oils have to fulfil the needs of the applications.

5.1.2 Natural base oils from biomass

These are primarily triglyceride esters derived from plants and animals. Common natural base oils include rapeseed oil, sunflower oil, palm oil and castor oil from vegetable, and tallow from animal sources.

Natural oils have in general particularly good tribological properties. Due to their specific structure, they combine good boundary friction lubricity and general wear protection with good viscosity-temperature behaviour and very low evaporation. However, all their highly positive physical features are countered by a few major limitations, the most important of which is the inadequate resistance to ageing of these base oils.

5.1.3 Synthetic base oils derived from biomass

The scope of oleochemistry allows almost any synthetic ester to be tailor-made. The resulting high-performance lubricants are technologically at least as good, if not, in some cases, much better than mineral oils. The optimization of natural oils refers to specific technical properties such as:

- oxidation and thermal stability,
- hydrolytic stability,

- viscosity,
- foaming,
- evaporation, etc.

Lubricants based on these synthetic esters are subject to performance and cost constraints. Currently the most used base oils for bio-lubricants are based on modification of fatty acids.

The most important modification relates to the carboxyl group of fatty acids, e.g. esterification: Vegetable oils are hydrolyzed to yield the fatty acids which are subsequently combined selectively to form special synthetic esters. As counterpart for the fatty acids, synthetic petrochemical-derived alcohols are often used. Depending on the relation between fatty acids and alcohols, the content of renewable raw material in the ester molecule can be calculated.

With regard to derivatives of fatty acids, different performance levels have to be recognized; traditionally oleochemical ester oils are classified into one of the three groups that have increasing thermal-oxidative stability:

- triglycerides,
- unsaturated esters, and
- saturated esters.

However, due to latest high oleic renewable oils and their derivatives, the three groups are not strongly divided, but show overlaps in some aspects of performance.

Another classification of synthetic esters refer to the number of functional groups in the alcohol and the acid, respectively: poly-ol esters, mono esters, dicarboxylic ester, complex esters. Other synthetic routes, like the biomass-to-liquid process, to produce base oils are also possible, even if not established today.

5.1.4 Biodegradable base oils from non-renewable resources

In principle, very low viscous, clean hydrocarbons can be biodegradable, i.e. degrade more than 60% according to OECD 301 test procedures [13]: Some special types of synthetic hydrocarbons up to a viscosity of 6 mm²/s at 100°C are seen as biodegradable. An example is the synthetic hydrocarbon PAO 2.

Many poly-alkylene-glycols (PAG) are well known as biodegradable; especially types of poly-ethylen-glycols up to PEG 1500 are ranked as biodegradable. Most biodegradable PAG’s are soluble in water and not mixable with mineral oil.

Even if PAG’s are developed with parts from renewable raw materials (up to 30 %), these products are not really discussed as potential base oils for bio-lubricants.

Synthetic esters derived from mineral oil are more compatible with conventional, mineral oil-based products. As mentioned, many synthetic esters contain alcohol components derived from petro-chemistry; i.e. a part of the final product coming from non-renewable resources.

Also, if fatty acids from petrochemical processes are used (e.g. odd-numbered branched fatty acids), the final molecule contains no renewable material. Well known examples for those ester oils are di-iso-tridecyl-adipate, di-2-ethyl-hexyl-adipate, tris-isononyl-trimellitate esters, phosphate esters etc.

5.2 Bio-lubricants – additives

In this context, the “bio“ prefix is used as an indication for lubricants which combine both biodegradability and relevant content of biomass. Due to the wide range of applications of lubricants, the number of additives used to impart performance characteristics to the lubricants is huge. The main families of additives are:

- antioxidants;
- detergents;

- anti-wear;
- metal deactivators;
- corrosion inhibitors, rust inhibitors;
- friction modifiers;
- extreme pressure;
- anti-foaming agents;
- viscosity index improvers;
- demulsifying/emulsifying;
- stickiness improver, provide adhesive property towards tool surface (in metalworking);
- complexing agent (in case of greases).

In principle, the same additives can be used for both conventional and bio-lubricants, as long as the overall environmental aspects are fulfilled.

5.3 Bio-lubricants – functionalities

Bio-lubricants as well as conventional lubricants have to perform the following key functions, depending on the specific application, e.g.:

- keep moving parts apart;
- protect against wear;
- reduce friction;
- transfer heat;
- carry away contaminants and debris;
- transmit pressure energy;
- prevent corrosion;
- reduce noises;
- seal for gases;
- reduce the risk of smoke and fire of objects.

5.4 Bio-lubricants – groups of application

All parts of ISO 6743 [16] establish the general classification system, which applies to lubricants, industrial oils and related products called class L. Within class L 18 families of products are defined to cover all applications for which lubricants are used. Not all families have been issued. The parts 16, 17 and 18 of ISO 6743 are still awaiting issue at this time.

Some of these 18 families and some of their subdivisions are shown below, mainly those product groups with some relevance to bio-lubricants.

From a pure technical point of view, it is accepted that more than 90 % of all present-day applications can be covered by bio-lubricants [10]. Table 2 summarizes nearly all application types of lubricants.

Table 2 — "Bio-lubricant" – groups of application

Product group	Main use today	already existing	anticipated, possible
Industrial	hydraulic oils	yes	yes
	air compressor oils	no	yes
	gas compressor oils	no	yes
	industrial gear oils	yes	yes
	slideway oils	yes	yes
	bearing and circulating system oils	yes	yes
	refrigerator compressor oils	yes	yes
	steam and gas turbine oils	yes	yes
	machine oils	partly	yes
	Insulating liquids	yes	yes
	concrete release oils	yes	yes
	chainsaw oils	yes	yes
Metalworking	cutting fluids – water-miscible	yes	yes
	cutting fluids – not water-miscible	yes	yes
	forming oils	yes	yes
	rust preventives	no / partly	yes
	quenching oils	yes	yes
Automotive	Engine oils — petrol (gasoline) engine oils — diesel engine oils — 2-stroke engine oils — gas turbine engine oils	 yes yes yes yes	 yes yes yes yes
	Automotive gear oils — manual transmission oils — automatic transmission fluids	 no no	 yes yes
	Brake fluids	no	potentially
	Mobile hydraulic fluids	yes	yes

Table 2 (*continued*)

Product group	Main use today	already existing	anticipated, possible
	Air Filter oils	yes	yes
Automotive	Tractor (one lubricant for all systems) — Universal Tractor Transmission Oil – UTTO — Super Tractor Oil Universal – STOU	yes yes	yes yes
	Crosshead cylinder oils	no	potentially
	Crosshead Crankcase oils	no	potentially
	Trunk piston engine oils	no	potentially
	Stern tube lubricants	no	yes
Greases	Roller bearings	yes	Yes
	Cars, trucks, construction vehicles	no	yes
	Steel mill	no	partly
	Mining	no	partly
	Railroad, Railway	yes	yes
	Gears	no	yes
	Food-grade applications	no / partly	partly
	Textile machines	no	partly

6 Standardisation needs

6.1 Standard test methods

It is evident that some effort needs to be made to bring more clarity in the sector of bio-lubricants. The term bio-lubricant should be better defined and the usage connected with defined wording, on the basis of some agreed standards, particularly when referring to the carbon sources and cycle as well as biodegradation.

6.2 Biodegradation

The most important test methods with regard to biodegradation are shown in Table 3 – beyond these methods ISO, ASTM and national procedures with more or less similar methods and restrictions do exist:

The limit for “readily biodegradation” is not exactly defined, but most of legislation and recommendations are working with these numbers.

Table 3 — Test methods for testing the biodegradation of (not water soluble) lubricants

Test method	Short description – relation to other test methods
OECD 301B	"Modified Sturm test", aerobic degradation, ultimate biodegradation
OECD 301C	"Modified MITI-Test", aerobic degradation, measurement of O_2-consumption, for volatile components
OECD 301D	"Closed Bottle Test", aerobic degradation, preferred for water soluble products, but possible for not water solubles substances
OECD 301F	"Manometric Respirometry Test"; for water soluble and not water soluble substances
BODIS test, ISO 10708 [17]	Two phases "Closed Bottle" test, similar OECD 301D
EN ISO 14593 [18]	CO2-Headspace Test

Previously another test method was widely used, the CEC L-33-A-93 [19]. This test method was used especially for 2-stroke oils. Because this test method determines only the initial ("inherent") and not the final degradation, this test method has become obsolete. It used an aerobic degradation, where the water-soluble part of degradation was not considered and it effectively was applicable only for poor water-soluble substances. Due to the difference of initial and final degradation the values are higher than OECD 301 results, up to 20 %.

NOTE Claims of biodegradability in other environments (e.g. landfill,) currently lack appropriate standards although development work is ongoing.

6.3 Ecotoxicity

In order to develop bio-lubricants, toxicological criteria should be considered. The aim is to protect life in various environments, especially in water (aquatic) and in the non-aquatic area (terrestrial). The following, especially in the eco-labelling systems used test methods are of importance:

— The bacteria test, according to EN ISO 8192 [20], determines acute toxicity through the inhibition of oxygen consumption; results of this test method are EC50 values.

— The algae toxicity test according to OECD 201 [13] is another test method for aquatic systems (measurement of chlorophyll-fluorescence and determination of EC10 and EC50 values).

— One of the most important test procedures concerning the aquatic environment is a test on small living organisms called the “Daphnia test“ (daphnia magna STRAUS, water flea, small crustacean) according to OECD guideline 202. Test method results are EC10, EC20 and EC50 values.

— In the aquatic environment, fish toxicity, according to OECD 203 [13], performed on the Goldorfe, (Leuciscus idus) is of importance. Test method results are the LC0, LC50 and LC100 values.

— Fish toxicity, according to OECD 204 [13], is incorporated in the eco label “Blue Angel“. Possible pollution of the non-aquatic terrestrial environment, i.e. soil and plants, is evaluated with the plant growth test method according to OECD 208 [13] (i.e. wheat, cress and rape seeds).

In general, toxicity testing for environmental protection purposes should also include toxicity for mammals and humans, particularly with regard to the combination of safety-at-work and environmental protection. The lethal dose LD50 is an important measure of toxicity.

For preparations such as lubricants, the EC Dangerous Preparations Directive (DPD – 99/45/EC [11]) is of great importance for assessing the toxic potential. The DPD's prime aims are identifying and labelling of dangerous formulations in finished products. As a consequence, the DPD lubricants might be labelled as “Dangerous for the Environment“ (DfE) and if necessary carry the "dead fish/dead tree" hazard symbol, based on the amount of DfE classified components they contain, or their intrinsic properties.

To avoid this negative labelling for bio-lubricants special toxicity limits are provided.

According to the DPD, the health and environmental hazards of a preparation can be evaluated by either a "conventional" test method (with limits for single components) or by testing the preparation using experimental test methods, e.g. OECD. In general, classification derived using test data of the finished product will override those given by the "calculation method", but there is a number of exceptions to this. Any preparation containing more than the specified amount of a component, which is classified as a carcinogen, mutagen or reproductive toxicant, shall be classified using the conventional test method.

Following the second test method – determination of the acute aquatic toxicity by testing on the preparation –, the test methods are to be carried out on all three species (i.e. algae, daphnia and fish according to OECD 201, 202, 203 with the limits of LC/EC_{50} > 100 mg/l), unless the highest hazard classification relating to acute aquatic toxicity has been assigned to the preparation after testing on one of the species. After successful testing such preparation is not related to a symbol, even if by the calculation scheme it would demand labelling. This procedure can be helpful for evaluation of formulated, finished products.

NOTE The DPD will be followed by the CLP, which is the Classification, Labelling and Packaging Directive 1272/2008/EC [8]. From 1 June 2015, both substances and product mixtures should be classified, labelled and packaged according to CLP.

The above-described evaluation of aquatic toxicity will be continued within CLP.

6.4 Renewable Raw Material (RRM)

The bio-based content in lubricants is covered in principal by standardisation in Europe[1)]. Relevant EN standards will be listed when available. In the USA, the issue of the bio-based content has been addressed by the ASTM with ASTM D 6852 [21], ASTM D 7075 [23], ASTM D 7026 [22] and ASTM D 6866 [12].

ASTM D 6866 is the only test method which can be used to unequivocally determine the amount of renewable raw material in any given product. It is based on the determination of the content of the radioactive isotope ^{14}C present in the material. Some natural radioactivity is present in the biosphere and, as a consequence, natural products always have a known content of ^{14}C. On the other hand, fossil-derived carbon contains negligible amount of ^{14}C, because this isotope has decayed over geological time.

It is therefore possible to measure the bio-based content of any product through a measurement of its ^{14}C content. Well-established methods of measuring ^{14}C exist and a similar standard test method could be developed by CEN.

NOTE All of these test methods relate to the origin of the carbon content of the final product. They do not include any consideration of the origin of the energy used in production, distribution and disposal of the material, which requires a full life cycle analysis if the true environmental impact of any material is to be assessed. More energy can be needed to grow and harvest biomass than to process oil. These effects can only be assessed by an overall LCA.

6.5 Issues in progress: sustainability, LCA, certification

Bio-based products should include in future (elements of) LCA’s. This point seems to be of special interest due to the proposed demand of minimum content of RRM in bio-lubricants. Thus, the three pillars of sustainable development:

---

1) See e.g. CEN/TC 343 "Solid recovered fuels" work on the determination of biomass content in such fuels (EN 15440 [24]

- economical aspects,
- social aspects, and
- environmental aspects

should have the same weight in any kind of considerations.

A step in this direction is recently done with first certification schemes for bio-based fuels; since 2010 certification labels such as RSPO (Rountable of Sustainable Palm Oil), ISCC ("International Sustainability and Carbon Certification") and REDcert (Renewable-Energy-Directive certification) are officially in place. However, a general adaption of this biofuel-focused approach to bio-based chemistry will be complicated, since most chemicals are derived from different raw material feeds – many single LCA’s would have to be combined.

NOTE Sustainability criteria for bio-energy is under consideration by CEN/TC 383.

Bibliography

[1] Bioplastics — Renewable raw Materials and Climate Protection (Kunststoffe International 2007/10, p. 109-115).

[2] European Ecolabel for lubricants (EEL) — http://ec.europa.eu/environment/ecolabel

[3] 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 final — http://eur-lex.europa.eu

[4] 07/589/EC, Commission Decision of 18 July 2007 establishing guidelines for the monitoring and reporting of greenhouse gas emissions pursuant to Directive 2003/87/EC of the European Parliament and of the Council, OJ EU, 2007, No. L 229, p. 1-85

[5] Narayan, Ramani, Biobased and Biodegradable Materials, Rationale, Drivers & Technology exemplars, ACS (An American Chemical Society Publication) Symposium Ser., 939, Ch. 18, pg 282, (2006).

[6] Nachwachsende Rohstoffe in der Industrie, edited by Fachagentur Nachwachsende Rohstoffe, 3rd revised edition, 2010, no. 228, p.32

[7] Concawe Report No. 5/96, *Collection and disposal of used lubrica- ting oil, Concave Report 5 (1996) CONCAWE, Boulevard du Souverain 165, B-1160 Brussels* – www.concawe.be

[8] 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, OJ EU, 2008, No. L 353, p. 1-1355

[9] 2005/360/EC, Commission Decision of 26 April 2005 establishing ecological criteria and the related assessment and verification requirements for the award of the Community eco-label to lubricants, OJ EU, 2007, No. L 131, S. 27

[10] Mang/Dresel: *Lubricants and Lubrication*, Wiley-VCH (2001), p.117

[11] 99/45/EC, *Directive 1999/45/EC of the European Parliament and of the Council of 31. May 1999 concerning the approximation of the laws, regulations and administrative provisions of the Member States relating to the classification, packaging and labelling of dangerous preparations*, OJ EC, 1999, No. L 200, p. 1-68

[12] ASTM D 6866, *Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis*

[13] *OECD Guidelines for the Testing of Chemicals* — www.oecd.org

[14] ISO 15380, *Lubricants, industrial oils and related products (class L) — Family H (Hydraulic systems) — Specifications for categories HETG, HEPG, HEES and HEPR*

[15] EN ISO 14021, *Environmental labels and declarations — Self-declared environmental claims (Type II environmental labelling) (ISO 14021:1999)*

[16] ISO 6743 (all parts), *Lubricants, industrial oils and related products (class L) — Classification*

[17] ISO 10708, *Water quality — Evaluation in an aqueous medium of the ultimate aerobic biodegradability of organic compounds — Determination of biochemical oxygen demand in a two-phase closed bottle test*

[18] 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 (CO_2 headspace test) (ISO 14593:1999)*

[19] CEC L-33-A-93, *Biodegradability of Two-Stroke Cycle Outboard Engine Oils in Water, http://www.cectests.org*

[20] EN ISO 8192, *Water quality — Test for inhibition of oxygen consumption by activated sludge for carbonaceous and ammonium oxidation (ISO 8192:2007)*

[21] ASTM D 6852, *Standard Guide for Determination of Biobased Content, Resources Consumption, and Environmental Profile of Materials and Products*

[22] ASTM D 7026, *Standard Guide for Sampling and Reporting of Results for Determination of Biobased Content of Materials via Carbon Isotope Analysis*

[23] ASTM D 7075, *Standard Practice for Evaluating and Reporting Environmental Performance of Biobased Products*

[24] EN 15440:2011, *Solid recovered fuels — Method for the determination of biomass content*

[25] 01/77/EC, *Directive 2001/77/EC of the European Parliament and of the Council of 27 September 2001 on the promotion of electricity produced from renewable energy sources in the internal electricity market*, OJ EC, 2001, No. L 283, p. 33-40

[26] 99/31/EC, *Council Directive 1999/31/EC of 26 April 1999 on the landfill of waste*, OJ EC, 1999, No. L 182, p. 1-19

This page deliberately left blank

This page deliberately left blank

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards -based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email bsmusales@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Copyright

Useful Contacts:

Customer Services
Tel: +44 845 086 9001
Email (orders): orders@bsigroup.com
Email (enquiries): cservices@bsigroup.com

Subscriptions
Tel: +44 845 086 9001
Email: subscriptions@bsigroup.com

Knowledge Centre
Tel: +44 20 8996 7004
Email: knowledgecentre@bsigroup.com

Copyright & Licensing
Tel: +44 20 8996 7070
Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

bsi.

...making excellence a habit.™