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Bio-based solvents — Requirements and test methods

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National foreword

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

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Solvants biosourcés - Exigences et méthodes d'essais

Biobasierte Lösemittel - Anforderungen und Prüfverfahren

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Foreword

This document (CEN/TS 16766:2015) has been prepared by Technical Committee CEN/TC 411 “Bio-based products”, 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/491 [1] given to CEN by the European Commission and the European Free Trade Association, for the development of European Standards for solvents and surfactants in relation to bio-based product aspects. It has been prepared by CEN/TC 411/WG 2 “Bio based solvents”, the secretariat of which is held by the European Solvents Industry Group and NEN.

According to the CEN-CENELEC Internal Regulations, the national standards organizations 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, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Bio-based products from forestry and agriculture have a long history of application, such as paper, board and various chemicals and materials. The last decades have seen the emergence of new bio-based products in the market. Some of the reasons for the increased interest lie in the bio-based products' benefits in relation to the depletion of fossil resources and climate change. Bio-based products may also provide additional product functionalities. This has triggered a wave of innovation with the development of knowledge and technologies allowing new transformation processes and product development.

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;

— Bio-based content determination;

— Life Cycle Assessment (LCA);

— Sustainability aspects;

— 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 from biomass' [3]. Bio-based products (bottles, insulation materials, wood and wood products, paper, solvents, chemical intermediates, composite materials, etc.) are products which are wholly or partly derived from biomass. It is essential to characterize the amount of biomass contained in the product by for instance its bio-based content or bio-based carbon content.

The bio-based content of a product does not provide information on its environmental impact or sustainability, which may be assessed through 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. This Technical Specification has been developed with the aim to fulfil part of a Mandate [1] to describe the technical requirements of bio-based solvents in relation to bio-based product aspects.

Solvents are liquids which have the ability to dissolve, suspend or extract other materials. In Europe, thousands of producers and manufacturers and more than 10 million workers use solvents every day. The solvent producing industry is composed of both small and medium-sized enterprises as well as multinationals. Downstream users generally tend to be SMEs and micro-SMEs.

Solvents are mainly produced from fossil feedstock. The amount of fossil feedstock used for solvent production is however low with less than 1 % of the total world's fossil feedstock consumption (see www.esig.be).

This document describes the approach that can be taken in describing the technical requirements of bio-based solvents in relation to bio-based product for the customers using bio-based solvents. This document examines how to prove the criteria for "bio-based solvents". The purpose of this document is to define how the criteria of performance, health, safety and environment can be determined (measured and calculated) for the

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

bio-based solvent placed on the market. This approach intends to strengthen the reputation of “bio-based solvents” and the confidence of the customer in this product group.

The criteria for "bio-based solvents" published in this Technical Specification are complementary to the other, horizontal standards by CEN/TC 411.

1 Scope

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

— fit for purpose in terms of performance related properties;

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

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

— comply with at least similar sustainability criteria as comparable (regular) solvents.

The criteria of the Regulation for Environmental Assessment of Chemicals (REACH) [2] are included in the discussions that have led to this Technical Specification.

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.

CEN/TS 16640, *Bio-based products - Determination of the bio based carbon content of products using the radiocarbon method*

EN 16575:2014, *Bio-based products - Vocabulary*

prEN 16751:2014, *Bio-based products - Sustainability criteria*

prEN 16760:2014, *Bio-based products - Life Cycle Assessment*

EN ISO 12185, *Crude petroleum and petroleum products - Determination of density - Oscillating U-tube method (ISO 12185)*

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)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 16575:2014 apply.

4 General purpose of solvents

Solvents are liquids which have the ability to dissolve, suspend or extract other materials. They make it possible to process, apply, clean or separate materials. Solvents have significantly changed modern living and are an invaluable solution for industries as diverse as pharmaceuticals and microelectronics to domestic cleaning and printing. In fact, without solvents, many of the products we use and rely on, from penicillin to industrial paint, would not perform to the standards we demand today.

Organic solvents are any organic compound which is typically used, alone or in combination with other agents, and without undergoing a chemical change, to dissolve raw materials, products or waste materials.

Examples of applications for solvents are (as presented in the Solvents Emissions Directive [4]):

— cleaning agent,

— dissolver,

— dispersion medium,

— viscosity adjuster,

— surface tension adjuster,

— plasticiser, or

— preservative.

NOTE For more examples of applications, see the website of the European Solvents Industry Group, www.esig.org.

5 Performance

5.1 Generalities around performance

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

Solvents are used in a wide variety of applications and it is not convenient to evaluate their performance with respect to each application. Therefore, a practical approach is to define a set of measurable solvent properties which enable technical specialists to select appropriate solvents and guide their evaluation.

A set of seven properties has been selected which describe essential properties associated with a solvent.

5.2 Technical performance properties

5.2.1 Chemical composition

While chemical composition is not a property in itself, it provides essential information regarding the suitability of a solvent in process and applications.

Quantitative chemical composition of the product should be provided. To identify substances or mixtures as relevant, use of the nomenclature as in EU CLP [5] shall be used.

5.2.2 Solvency power

Very different ways have been defined to measure solvency power, generally experimental measurements such as solubility in water, solubility in oil, Aniline point, Kauri Butanol index, polarity, etc. However, such methods are not able to describe the full extent of the solubility properties of products exhibiting a wide variety of polarities.

A well-known quantitative assessment of solvency power has been developed through the means of the Hansen Solubility parameters [6], which provide a suitable description of solvency properties. Annex A gives a detailed description on how to determine these solubility parameters.

Other scales may be used to characterize solvency power as for example the Kamlet-Taft parameters, as long as they are correlated to Hansen Solubility.

Values of the three Hansen solubility parameters shall be given and should be reported to one decimal place and in units of $MPa^{1/2}$.

The source of solubility parameter values shall be reported and whether they are estimated or experimentally determined.

5.2.3 Distillation characteristics

Numerous methods are available, based upon actual distillation measurements or on correlations, especially from gas chromatography.

NOTE Some formulations of solvents might not be suitable for actual distillation measurement, for example some emulsions.

A selection of recommended test methods is listed below:

— EN ISO 3405 [22], which determines distillation characteristics at atmospheric pressure used for petroleum products of distillation points above 0 °C and end points below approximately 400 °C and is equivalent to ASTM D86 [7, 8].

— ISO 918 [25], which determines distillation characteristics of organic liquids used for non petroleum products of boiling points in the range of approximately 30 °C to 300 °C in atmospheric conditions and which are stable under these conditions.

— ASTM D1078 [29], which is a test for the distillation range of volatile organic liquids used for distillation range of organic liquids boiling between 30 °C and 350 °C and which are stable during the distillation process.

— EN 15199-1 [20], describes the determination of boiling range distribution of materials with initial boiling points (IBP) above 100 °C and final boiling points (FBP) below 750 °C, it is equivalent to ASTM D5399 [9].

When reported, distillation characteristics of bio-based solvents shall be expressed by providing an initial boiling point and an end point (Dry Point or Final Boiling Point) expressed in °C with a reporting precision in compliance with the rules of the test method standard selected. The reference of the test method standard shall be indicated also.

5.2.4 Evaporation rate

The evaporation rate of a solvent is a useful indication of the speed at which a solvent will dry. Depending upon the types of solvents, different scales may be used. Three different methods are applicable to various solvent types: ASTM D1901 [31], ASTM D3539 [32] and DIN 53170 [35].

Results of measure for the evaporation rates should be provided in accordance with the relevant test standard. The reference to the test method standard shall be indicated also.

Such methods are used on a comparative basis against a reference volatile solvent such as Di-ethyl-ether or *n*-butyl-acetate. Another potentially useful property, related to the evaporation rate, is the vapour pressure measured or calculated at a specific temperature. The vapour pressure is a property indicated in the EU CLP Safety Data Sheet in section 9 [5].

The value of the vapour pressure may be provided in place of the evaporation rate. For an overview of test methodologies, see CEN/TR 16569 [10]. In that case, the value of the vapour pressure shall be indicated with a proper unit (e.g. Pa, kPa or mm Hg) and the temperature at which the value refers. A reference shall be indicated (i.e. measured or calculated).

5.2.5 Colour

Depending upon the type and intensity of the colour exhibited by the solvent, different scales are used. The following colour test methods and scales should be used.

For clear liquids, the Gardner colour scale as in ISO 4630-1 [26] and ISO 4630-2 [27] or the Platinum-Cobalt scale as in EN ISO 6271-1 [23] and EN ISO 6271-2 [24].

For petroleum type of liquids the Saybolt scale as determined via the chronometer method of ASTM D156 [28], the ASTM scale determined by using ASTM D1500 [30] or the more general techniques as in ASTM D6045 [33] (automatic tristimulus method).

Results of measure for the colour shall be provided in accordance with the relevant test standard. The reference to the test method standard shall be indicated also.

5.2.6 Density

Density of solvents can vary significantly depending upon their chemical nature. Density of solvents provide useful information for the selection of suitable solvents for multiple applications.

EN ISO 12185 shall be used, a method technically equivalent to ASTM D4052 [11].

Results of measure for the density shall be provided in accordance with the relevant test standard and associated with the temperature at which the measurement refers to. The reference to the test method standard shall be indicated also.

5.2.7 Kinematic viscosity

Viscosity measures the internal resistance to the flow exhibited by a fluid. Various measurement methods, direct or indirect are suitable for the determination of kinematic viscosity at 40 ºC.

CAUTION — Some solvents have a boiling point below 40 ºC. In that case, measurement at lower temperature shall be done, preferably the temperature of use, and reported.

The following selection of viscosity test methods should be used:

— EN ISO 3104 [21] for the determination of kinematic viscosity and the calculation of dynamic viscosity, a technique also described in ASTM D445 [12], or

— ASTM D7042 [34] for dynamic viscosity and density of liquids by the Stabinger viscosimeter, a technique momentarily also developed under CEN.

Results of measure for the kinematic viscosity shall be provided in accordance with the relevant test standard and associated with the temperature at which the measurement refers to. The reference to the test method standard shall be indicated also.

6 Health, safety and environmental requirements

As any chemical, the substance(s) composing the bio-based solvent shall be fully in compliance with REACH regulation [2], especially for their registration and classification, and with GHS/CLP regulation [4] for the labelling. In addition, a bio-based solvent shall comply with any other EU regulations related to chemicals.

7 Bio-based content

At the moment of publication of this document, standardized determination technologies for bio-based content are measuring based on carbon content. For determination of bio-based carbon content, CEN/TS 16640 shall be used.

For solvents, a measurement of bio-based content may not be relevant in most cases. Reasons for this are:

— the nature of (bio)chemical processes,

— the different requirements of the applications of solvents,

— the complex supply/value chain of the solvents, and

— the existence of interlinked production systems (co-production, mixture, etc.).

In such cases, methods based on mass balance or material balance are recommended to establish the (allocated) bio-based content of a bio-based solvent.

NOTE Standardized techniques for mass balance and material balance techniques are being developed in CEN.

To inform about the bio-based content, three classes for bio-based solvents are defined, A, B and C, where the highest class will be “A” and the lowest is “C”. Details are given in Table 1.

The determined bio-based content may be reported in addition to the solvent class.

Table 1 — Bio-based solvents classes

Solvent class	Bio-based carbon content [a] % (*m/m*)	Comments
C	$\geq$ 25 [b]	Applicable for solvents, whereas the minor raw material part is bio-based
B	$\geq$ 50	Applicable for solvents, whereas the major raw material part can be considered as bio-based
A	$\geq$ 95 [c]	Applicable for solvents, whereas all raw material part can be considered as bio-based

[a] At the moment of publication of this document, standardized determination technologies for bio-based content are based on carbon content. If other determination techniques become available, the classes may be reviewed.

[b] To allow harmonization with other bio-based initiatives like surfactants, lubricants, polymers, etc. and to be in line with (legal) drivers such as the United States Department of Agriculture, the minimum threshold of 25 % bio-based carbon content has been set.

[c] For classification purposes, 100 % bio-based is not realistic, so an acceptable and reproducibly measurable level is set.

8 Sustainability

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

NOTE Further definition of the sustainability criteria will follow in a future version of this Technical Specification.

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

The bio-based solvent shall be based on sustainable biomass feedstock, therefore the economic operator shall demonstrate compliance with a chain-of-custody system that is recognized and widely accepted internationally, regionally or nationally and in line with prEN 16760, thereby ensuring traceability of the biomass along the value chain.

9 Declaration and product labelling

A bio-based solvent shall be labelled with its class as defined in Clause 7.

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

For additional requirements for business-to-business and business-to-consumer declarations, the user is referred to work in CEN/TC 411/WG 5 or to relevant regulations [13].

Annex A
(normative)

Hansen solubility parameters

A.1 Generalities

The solvency power, or polarity, of a solvent is crucial to its performance, dictating the range of compounds it can dissolve. The IUPAC definition of polarity is given as thus [14]: When applied to solvents, this rather ill-defined term covers their overall solvation capability (solvation power) for solutes (i.e. in chemical equilibria: reactants and products; in reaction rates: reactants and activated complex; in light absorptions: ions or molecules in the ground and excited state), which in turn depends on the action of all possible, nonspecific and specific, intermolecular interactions between solute ions or molecules and solvent molecules, excluding such interactions leading to definite chemical alterations of the ions or molecules of the solute.

Occasionally, the term solvent polarity is restricted to nonspecific solute/solvent interactions only (i.e. to van der Waals forces).

There are different approaches to the measurement of polarity. In one method, the forces that contribute towards the total polarity expressed by a solvent are divided into three parameters, and measured as dispersion forces (δ_D), dipole forces (δ_P), and hydrogen bonding (δ_H). Collectively, they are known as the Hansen solubility parameters [6]. The square of each Hansen solubility parameter is a portion of the total cohesive energy density of the solvent, which in turn corresponds to the square of the Hildebrand solubility parameter (δ_T). Cohesive energy density is a measure of the strength of all solvent interactions in a given volume, calculated from the standard enthalpy of vaporization (ΔH_{vap}, in units of $Jmol^{-1}$), and molar volume of the solvent (V_m, in units of cm^3mol^{-1}).

$$\delta_T^2 = \frac{\Delta H_{vap} - RT}{V_m} = \delta_D^2 + \delta_P^2 + \delta_H^2 \qquad (A.1)$$

The three Hansen solubility parameters of a bio-based solvent provide enough information to begin the substitution of petrochemical solvents. Solvents with similar Hansen solubility parameters are expected to exhibit the same solvency power for the same range of solutes. Some examples of Hansen solubility parameters are provided in Table A.1.

Table A.1 — Examples of Hansen solubility parameters

Solvent	δ_D	δ_P	δ_H
Acetic acid	14,5	8,0	13,5
Acetone	15,5	10,4	7,0
Acetonitrile	15,3	18,0	6,1
Benzene	18,4	0,0	2,0
1-Butanol	16,0	5,7	15,8
Chloroform	17,8	3,1	5,7
Diethyl ether	14,5	2,9	5,1
N,*N*-Dimethyl formamide	17,4	13,7	11,3
Ethanol	15,8	8,8	19,4
Ethyl acetate	15,8	5,3	7,2
Ethyl lactate	16,0	7,6	12,5
n-Hexane	14,9	0,0	0,0
Limonene	17,2	1,8	4,3
Tetrahydrofuran	16,8	5,7	8,0

A.2 Calculation of the Hansen solubility parameters for individual solvents

A.2.1 General

Hansen solubility parameters can be calculated using experimental estimations or computational methods. Both methods are widely practiced and preferred to obtain Hansen solubility parameters. Alternatively, experimental data can be used when available or determined directly. Values of the three Hansen solubility parameters should be reported to one decimal place and in units of $MPa^{½}$. The source of the values should be reported, and whether they are calculated or experimentally determined.

A.2.2 Estimated values

The Hansen solubility parameters of many established solvents are already known, mostly through estimations [6]. Conversely, novel bio-based solvents will have limited experimental data available for the determination of polarity, and will rarely feature in datasets of estimated Hansen solubility parameters. In instances where data is available there is no need to recalculate the polarity measurements. The Hansen solubility parameters are routinely calculated from group contribution methods, for the experimental methods are quite complex, and the estimated values generally reliable. Computer software is available with which to perform this task [15], as are published tables of group contribution data for manual calculation [16].

Another alternative to the experimental determination of the Hansen solubility parameters is to calculate them from observed solubility in a range of solvents, as is done for polymers. A large number of solvents are required to fit the polarity of the solute (in this instance the bio-based solvent) into a three dimensional polarity chart, each axis corresponding to a Hansen solubility parameter. The procedure is described in detail elsewhere [6]. From observation, the bio-based solvent can be positioned on the polarity map according to which solvents it is miscible with, and those it is not. It is likely that a bio-based solvent will be miscible with a great number of other organic solvents and so assignment of Hansen solubility parameters may be difficult using this method, in which case computation estimation is preferable

A.2.3 Experimental dispersion forces

The first Hansen solubility parameter, δ_D, is arrived at by analogy with a solvent's hydrocarbon homomorph (e.g. ethanol becomes propane, acetone becomes iso-butane, etc.) when the comparison is made at the same reduced temperature [17]. To do so the critical temperature may need to be estimated. The vaporisation energy of the homomorph is used as the basis of the measurement of dispersion forces [6]. The δ_D parameter of the solvent can be derived from graphical plots of the vaporisation energy (or cohesive energy density) of hydrocarbon homomorphs as a function of their molar volume. This approach should only be used if the Hansen solubility parameters cannot be calculated computationally or through estimation from observed solubility in other solvents.

A.2.4 Experimental dipole forces

A simple equation for obtaining values of δ_P is available based on the experimental bond dipole moment (μ, in units of Debye) and the molar volume of the solvent at 298 K [6]:

$$\delta_\mathrm{P} = \frac{37{,}4\mu}{(V_\mathrm{m})^{0{,}5}} \qquad \text{(A.2)}$$

Formula (A.2) is not always satisfactory, such as in the case of symmetrical molecules with zero dipole moment, and for large molecules with long hydrocarbon chains. In this instance, computational calculation or estimation from observed solubility in other solvents is preferable. Alternatively, a more complex equation has been reported for use in instances where more physical property data is available [18].

A.2.5 Experimental hydrogen bonding

The δ_H parameter is routinely determined by removing the contributions of δ_D and δ_P from δ_T when reliable experimental data is available. An alternative but related method of obtaining δ_H values from experiment has been described by Bondi and Simkin [19]. Group contribution techniques are available for the estimation of δ_H and are frequently used in preference to experimentation.

Alternatively, δ_H may be calculated by plotting the 3D Hansen chart based on the observed solubility of the bio-based solvent in a large number of other solvents.

A.3 Calculation of Hansen solubility parameters for solvent blends

A mixture of solvents has a combined polarity that is a function of the Hansen solubility parameters belonging to each component (δ), and their respective volume fractions (V). Therefore, the Hansen solubility parameters describing the polarity of a mixture of solvents shall be calculated from information corresponding to each ingredient:

$$\delta = \sum_{i}^{n} \delta_i \cdot V_i \tag{A.3}$$

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Recommended standards in support of this document

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[21] EN ISO 3104, *Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity (ISO 3104)*

[22] EN ISO 3405, *Petroleum products - Determination of distillation characteristics at atmospheric pressure (ISO 3405)*

[23] EN ISO 6271-1, *Clear liquids - Estimation of colour by the platinum-cobalt scale - Part 1: Visual method (ISO 6271-1)*

[24] EN ISO 6271-2, *Clear liquids - Estimation of colour by the platinum-cobalt scale - Part 2: Spectrophotometric method (ISO 6271-2)*

[25] ISO 918, *Volatile organic liquids for industrial use - Determination of distillation characteristics*

[26] ISO 4630-1, *Clear liquids - Estimation of colour by the Gardner colour scale - Part 1: Visual method*

[27] ISO 4630-2, *Clear liquids - Estimation of colour by the Gardner colour scale - Part 2: Spectrophotometric method*.

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[33] ASTM D6045, *Standard Test Method for Color of Petroleum Products by the Automatic Tristimulus Method*

[34] ASTM D7042, *Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)*

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