

# BIODIESEL GUIDELINES

**MARCH 2009** 

From the Worldwide Fuel Charter Committee





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European Automobile Manufacturers Association

Alliance of Automobile Manufacturers Engine Manufacturers
Association

Japan Automobile Manufacturers
Association

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Subject: Worldwide Biofuels Harmonisation

Dear Biodiesel Guidelines Recipient:

On behalf of automobile and engine manufacturers from around the world, we are pleased to present this First Edition of Biodiesel Guidelines from the Worldwide Fuel Charter (WWFC) Committee. We created the WWFC in 1998 to promote greater understanding of the impact of fuel quality on engine and vehicle emissions and performance and to promote harmonization of fuel quality worldwide in accordance with engine and vehicle needs in different markets.

Rapid growth in the use of ethanol and biodiesel prompted the WWFC Committee to address the need for more information about these important renewable fuels. As with conventional gasoline and diesel fuel, biofuel quality must match the needs and capabilities of engine and vehicle technologies, especially as these technologies become more advanced to meet ultra-clean emission standards. This document provides our guidance on biodiesel quality; a companion document provides guidance on ethanol quality.

Given the wide variation in performance and measurement methods of biofuel blends at different blend levels, this guidance focuses on the quality of the blendstock used to make finished biofuel blends, rather than on the finished fuels themselves. Specifically, we created this document to guide blenders who produce and use 100% biodiesel and diesel fuel blendstocks to make finished 5% biodiesel blends. The resulting finished fuels should continue to meet the recommendations contained in the WWFC for the various categories of market fuels.

The use of ethanol and biodiesel fuels is important to help extend supplies of gasoline and diesel fuel. As renewable fuels, they have the potential to help reduce emissions of greenhouse gases. Proper formulation also can help assure lower emissions of conventional pollutants. The key to achieving low emissions is to sustainably produce good quality blendstocks and to blend and distribute the finished fuels in a way that preserves their quality when they reach the consumer.

This document represents our best collective judgment at this time, based on experience with biodiesel produced from conventional feedstocks, such as rapeseed and soy and aided by comments from interested parties. We recognize that its technical information will continue to evolve, so this document will also change as we learn more. We appreciate the efforts of those who provided information and comments, and we are especially grateful to those who expressed support for this endeavor.

We look forward to working with you to support the development and use of high quality renewable fuels for the benefit of consumers and the environment worldwide.

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#### **Supporting organisations:**

Organisation Internationale des Constructeurs d'Automobiles (OICA)

#### **Acronyms**

ABNT NBR Associação Brasileira de Normas Técnicas (Brazilian Standards Number)

ACEA Association des Constructeurs Européens d'Automobiles (European automotive

manufacturers association)

Alliance of Automobile Manufactures

ASTM ASTM International (formerly American Society for Testing and Materials)

Biodiesel This document defines biodiesel as an ester-based fuel, created by reacting vegetable or animal fats with alcohol, meeting certain specifications and able to

be used in diesel engines. Biodiesel is often synonymous with FAME.

BTL Biomass to Liquid

BXX Biodiesel blend containing a specified amount of biodiesel by volume (e.g., B20

contains 20% biodiesel by volume)

Ca Calcium

CEC Coordinating European Council for the Development of Performance Tests for

Transportation Fuels, Lubricants and Other Fluids

CEN/TR Comité European de Normalisation (European Committee for Standardization)

/Technical Report

**DPF** Diesel particulate filter

**EMA** Engine Manufacturers Association

EN European Norm
 FAEE Fatty Acid Ethyl Ester
 FAME Fatty Acid Methyl Ester
 HVO Hydrotreated vegetable oil
 ICP Inductively-coupled plasma

ISO International Organization for Standardization

JAMA Japan Automobile Manufacturers Association

JIS Japanese Industrial Standards

**K** Potassium

**KOH** Potassium hydroxide

Mg Magnesium mod Modified Na Sodium

NaOH Sodium hydroxide ppm Parts per million

**prEN** Provisional European Norm

**PSA DW** A family of PSA Group diesel engines manufactured for use in Peugeot and

Citroën automobiles.

TAN Total Acid Number

WWFC Worldwide Fuel Charter

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#### Introduction

The purpose of the Worldwide Fuel Charter is to promote high quality and harmonized fuels on a global basis, considering the need for optimum engine and vehicle performance and durability and for the cleanest possible operation of engine and vehicle technologies. Meeting this objective will benefit consumers, simplify fuel markets, facilitate trade and help governments meet public policy goals.

Biofuels are of particular interest today due to their potential to help reduce the use of petroleum-based fuels, improve energy security and reduce greenhouse gas emissions. Biodiesel is one of these fuels that is viable and in use today, along other promising alternative diesel fuels such as renewable diesel fuel (hydrotreated vegetable oil (HVO)) and biomass to liquid (BTL) fuels. Good biodiesel quality is fundamental to its continued success as a fuel. The guidance presented here contains manufacturer recommendations regarding the biodiesel quality needed for proper engine and vehicle operation.

The recommended limits in these guidelines are specifically established for 100% biodiesel (B100) blendstock intended for blending with petroleum-based diesel fuel to make a blend containing a maximum of 5% biodiesel by volume (B5) suitable for use in vehicles with compression ignition engines. Higher level blends may require different specifications, labeling, and other controls to adequately protect consumers. Finished biodiesel-diesel fuel blends, as well as finished petroleum-based diesel fuel, should continue to meet the requirements of the appropriate diesel fuel category in the Worldwide Fuel Charter.

Biodiesel quality changes over time due to the inherent nature of the fuel. Oxidation reactions begin to affect the fuel as soon as the biodiesel is created. Storage conditions, especially temperature, exposure to water and exposure to oxygen which is naturally present in ambient air, influence the rate of oxidation. Anti-oxidant additives can help slow this degradation process and improve fuel stability up to a point. Their effectiveness depends on their addition to the biodiesel during or as soon after production as practical. The overuse of anti-oxidants can lead to the additional formation of sludge. Thus, care must be taken in choosing the antioxidant type and amount for maximum effectiveness. It is also critical for biodiesel blendstocks to meet the limits specified in this guidance. After blending, distributors and retailers should avoid storing blends for excessive time periods or under adverse conditions, and they should monitor the blends' quality at least until the fuel is transported or sold.

These guidelines are based on engine and vehicle manufacturer experience with biodiesel fuels made from feedstocks commonly used today in various markets around the world. The guidelines are performance-based and feedstock-neutral. As new feedstocks are identified, the properties and limits specified here may require revision to ensure their continued suitability.

Biodiesel fuel and biodiesel fuel blends must have uniform properties throughout, to ensure a consistent quality both prior to and after blending. High speed injection blending is preferred to splash blending as a means to ensure uniform quality throughout the final fuel blend. The blending process and confirmation of uniform properties is very important, especially when splash blending is used. Blenders and consumers should check with their suppliers and request confirmation of feedstock and finished product uniformity.

Biodiesel fuel and biodiesel fuel blends must comply with all legal requirements regarding labeling. Both blenders and consumers will benefit from having accurate information about biodiesel content. Engine and vehicle manufacturers will continue to evaluate these Guidelines for ongoing applicability to increasingly sophisticated vehicle systems and components, which must comply with numerous government regulations. The WWFC Committee will review and revise these recommendations as necessary to reflect changes in engine and vehicle technologies, biodiesel production and marketing practices and test methods.

#### Guidelines for B100 Blendstock for use in up to B5 Blends

Property	Limit	Units	Test Methods
Ester content	96.5 min	% m/m	EN 14103 mod Other: ABNT NBR 15342
Linolenic Acid Methyl Ester	12.0 max	% m/m	EN 14103 mod
Polyunsaturated acid methyl ester (≥ 4 double bonds)	1 max	% m/m	prEN 15779

#### **Ester content**

The fuel's ester content indicates the amount of FAME in the fuel, and therefore, it is an indicator of the fuel's quality. A low amount of ester may indicate that unreacted compounds, such as tri-glycerides, or process related compounds, such as catalysts (KOH/NaOH) or methanol, remain in the fuel. Low levels also may indicate contamination with non-FAME compounds. These impurities may cause fuel filter plugging, engine deposits or other problems. Local law may require measuring the fuel's ester content; ester measurement also can help prevent or minimize fraud under incentive programs.

#### **Linolenic Acid Methyl Ester**

This ester, which is inherent to certain feedstocks, contains three double bonds which make the molecule highly unstable. Thus, these molecules oxidize or polymerize easily, thereby creating acids or sludge.

#### Polyunsaturated acid methyl ester (≥ 4 double bonds)

These esters will rapidly polymerize and should be absent from the fuel to protect against sludge. Limiting other polyunsaturated molecules with three or more double bonds will improve fuel stability.

#### Notes regarding test methods:

EN 14103 is valid for both methyl and ethyl esters but has a limited range when used for ethyl esters. This test is being improved to cover more esters. Methods for measuring large esters with four or more double bonds are under development.

Oxidation Stability:			
Induction Period	10 min	hr	prEN 15751 or EN 14112 as alternative

Oxidation stability is very important. In addition to oxidation stability as measured by Induction Period, see discussion below related to change in Total Acid Number.

Oxidation stability is one of the most important properties because FAME oxidizes easily compared with petroleum diesel fuel and produces reaction products that can damage the engine or vehicle. Fuels with a high number of molecules with methylene groups adjacent to double bonds are particularly susceptible to oxidation. The oxidation process begins as soon as the vegetable oil is created and continues until the beginning of re-esterification. Oxidation produces peroxides (hydro-peroxides)

which undergo further reaction to form acids, which are themselves oxidizing agents. Molecules may also polymerize and form gums, sludge or other insoluble compounds; unlike peroxides, which usually disappear at some point during re-esterification, polymers that form during oxidation do not disappear and remain in the mixture. The oxidation reaction continues until the reactive sites or available oxygen are depleted. The "Rancimat" test method, EN 14112 (or prEN 15751), provides an indication of the amount of time (induction period) the fuel can be stored before the production of acids indicates the fuel is becoming unstable.

Peroxides can damage or degrade plastics and elastomers, particularly at high temperatures. The acids corrode metals used in vehicle and distribution fuel handling systems. The impact of acids on metal fuel tanks is especially severe. Even light corrosion from carbonic acid salts will cause sticky deposits inside the fuel pump and injectors. These metal and carbonic acids react again to form salts. These salts, which are soluble in the fuel, pass through the fuel filter but then stick to the surfaces of the fuel pump and fuel injectors. The salts also can form sludge-like injector deposits. Polymers, sludge and other insoluble materials formed during oxidation can cause fuel filter blockage.

Historically, petroleum diesel fuels have been successfully stored for extremely long periods without oxidizing, but even low concentrations of FAME can reduce the stability of finished blends. A few cases of lower oxidation stability than expected have been observed with biodiesel blends made with very low sulfur petroleum diesel fuels; some theorize this change may be related to the type of processing required to produce the very low sulfur levels.

Depending on storage conditions (e.g., temperature, humidity in the air space, presence of water, and other factors), FAME that meets the specified limit at the time of retail sale should provide six months of storage capability before unacceptable degradation of the FAME or blends occurs.

To reduce the potential for market problems, the oxidation stability of the blendstock must exceed the recommended minimum induction period, and the finished fuel also must meet its own oxidation stability requirement. If the oxidation stability of the blendstock is poor, the oxidation stability of the finished fuel will be poor. Antioxidant additives can help achieve the recommended limit; research is continuing into antioxidant types, effectiveness and dosing levels. When using antioxidants to improve the oxidation stability, producers should add the antioxidants to freshly made FAME—or even during FAME production—for maximum effectiveness. Due to fuel changes over time, fuel providers should monitor the fuel quality and take steps to minimize degradation and avoid the use of degraded fuel.

#### Notes regarding test methods:

The provisional method prEN 15751 is known as the modified Rancimat test method for B100 and biodiesel blends. When testing B100, test methods to measure insolubles such as polymers, sludge and other substances (EN 12205 or ASTM D2274) must be modified to use glass fiber filters to avoid degrading the filter media.

Iodine Number	130 max*	g l <sub>2</sub> /100 g	EN 14111	

Iodine number indicates the total number of double bonds (i.e., level of saturation) in the mixture of molecules. Opinions differ regarding whether the iodine number helps indicate oxidation stability, where a higher iodine number represents lower oxidation stability. To the extent it does help indicate oxidation stability, the iodine number may provide information about the fuel's tendency to form sludge, affect lubricant quality and/or cause corrosion.

\*Limiting the iodine number to 130 max can preclude the use of certain feedstocks that are prominent in different regions, especially soybean, sunflower and other unsaturated oils, but control may be

unnecessary if the fuel has good oxidation stability as measured by other methods. Measuring oxidation stability only by the Rancimat method (EN 14112) may be inadequate, however, because this test only evaluates the fuel's acid forming tendency and cannot evaluate its tendency to form sludge. To bypass the iodine number limit, FAME producers and providers must take additional steps, such as testing for and limiting sludge formation or using methods to evaluate sludge forming tendencies, to prevent problems. There are indications that exhaust aftertreatment technologies utilizing late post injection for active PM-trap regeneration may need a more stringent limit.

The limit recommended here represents the current opinion of vehicle and engine manufacturers, but further research is needed to understand the relation of this property to engine performance and to determine what iodine limits, if any, are necessary and sufficient for adequate stability.

ISO 6618

ASTM D664, D974

**Total Acid Number** 0.50 max mg KOH/g JIS K2501

Other: ABNT NBR 14448

Acid number is a measure of the acids in the fuel. These acids emanate from two sources: (i) acids used in the production of the biodiesel that are not completely removed in the production process; and (ii) as a byproduct from degradation by oxidation. The acids are measured in terms of amount of KOH required to neutralize a gram of FAME. For biodiesel blends, the acid number will change as a result of the normal oxidation process over time, and recent research has shown that this change is a good indicator of B100 stability. If the fuel is not used immediately after purchase, buyers should monitor their biodiesel fuel blends for changes in acid numbers, as an indicator of fuel degradation. The presence of acids in the fuel can harm injection systems and other metallic components.

#### **Change in TAN**

Research suggests that measuring the change in total acid number is a good way to measure fuel stability. The proposed limit for finished blends (only) is 0.12 mg KOH/g maximum after aging, following the EN 12205/ASTM D2274 protocol at 115°C.

#### *Notes regarding test methods:*

EN 14104 is specifically designed for B100. ASTM D664, a potentiometric method, and ISO 6618/ASTM D974, which are colorimetric methods, can be used on blends but results between the two methods should not be interchanged. JIS K2501 can be used for both B100 and finished blends.

% m/m

EN 14110

0.20 max

Other: ABNT NBR 15343

JIS K2536

#### **Methanol/Other Alcohols**

Methanol

Methanol is a raw material used to produce FAME. Other alcohols, such as ethanol, also may be used, producing fatty acid ethyl ester (FAEE), but there is limited experience in producing FAEE and with selecting and applying an ethanol limit. Ethanol also can enter the product through contamination.

Alcohol remaining in the product, such as from incomplete reaction during production or contamination, can have harmful effects: lowered flash point, decreased lubricity, corroded injectors and degraded materials used in fuel distribution and vehicle fuel systems. Collectively, these potential problems raise handling and safety concerns.

#### Notes regarding test methods:

These test methods do not measure other alcohols that may be present in the ester product, but a preliminary investigation has shown that a small change to EN 14110 will allow ethanol measurement.

Glycerides			EN 14105
			EN 14105
Mono-glyceride	0.80 max	% m/m	ASTM D6584
0.		·	Other: ABNT NBR 15342
			EN 14105
Di-glyceride	0.20 max	% m/m	ASTM D6584
· .			Other: ABNT NBR 15342
			EN 14105
Tri-glyceride	0.20 max	% m/m	ASTM D6584
			Other: ABNT NBR 15342
Glycerin (glycerol)			
			EN 14105/14106
Free glycerin	0.02 max	% m/m	ASTM D 6584
			Other: ABNT NBR 15341
			EN 14105
Total glycerin	0.25 max	% m/m	ASTM D 6584
			Other: ABNT NBR 15344

#### Glycerides: Mono, Di, and Tri

Glycerides, which are high molecular weight reactants in the biodiesel production process, can remain unreacted in the FAME after processing. They are undesirable because they can cause filter plugging, especially at cold temperatures, and injector and engine deposits; they can also limit vehicle operability over a wide range of conditions. Mono-glycerides can lead to increased injector deposits. High monoglyceride content has been linked to solid deposits in B5 where the fuel was stored in tanks under Scandinavian winter conditions. At such low temperatures, a maximum limit of 0.40% m/m is recommended. The limit may be revisited after investigations are complete. Di-glycerides can lead to filter plugging. Tri-glyceride content is a good indicator of unreacted oils or greases in the biodiesel.

Glycerides can be controlled to low levels through the use of good operating procedures during production.

#### Glycerin (glycerol), Total and Free

Glycerin, which is a byproduct of the chemical reaction that produces biodiesel, may remain in the fuel if the ester is inadequately separated or washed. Glycerin also may separate out of the liquid during storage after any methanol, which acts as a solvent, has evaporated. Once separated, the glycerin will fall to the tank bottom and attract such polar compounds as water, mono-glycerides and soaps that can block filters, damage injectors, cause injector coking and other engine deposits, and otherwise make the fuel incompatible with vehicle materials and reduce engine durability.

Glycerin can be controlled through the use of good operating practices during production.

#### Notes regarding test methods:

fuel quality.

Kinematic Viscosity@40°C

In finished fuel blends, petroleum diesel fuel components can interfere with the ability to directly measure glycerin in the fuel. When an acceptable test method for glycerin in a fuel blend is available, a limit value for blends will be established.

A new, simplified cold filtration test procedure related to some glycerides is in development.

Due to the presence of hydroxyl groups in the main ester carbon chain, the only method listed that is suitable for use with castor oil-derived FAME is the Brazilian ABNT NBR 15344.

Glyceride measurements can be used to calculate total and free glycerin.

2.0 - 5.0

EN ISO 3675
ASTM D4052

Density Report g/ml JIS K2249
Other: EN ISO 12185, ABNT NBR
7148/14065

Biodiesel density is usually higher than that of fossil diesel fuel, with the specific values depending on fatty acid composition and purity. Most batches of FAME contain only about ten different molecules with densities usually within a very narrow range. Contamination can significantly affect FAME density, so this property can be used to indicate contamination by some unwanted compounds and to monitor

EN ISO 3104
ASTM D445

mm<sup>2</sup>/s

JIS K2283 Other: ABNT NBR 10441

Kinematic viscosity is an inherent property of FAME's different feedstocks that affects injector lubrication and fuel atomization. Biodiesel fuel blends generally have improved lubricity; however, their higher viscosity levels tend to form larger droplets on injection which can cause poor combustion and increased exhaust smoke under certain operating conditions. At FAME blending levels up to 5 % by volume, the suggested limits provide an acceptable level of fuel system performance for the finished fuel blends and allow blending without changing the viscosity of the base diesel fuel. Adhering to the limits will also help harmonize FAME quality.

For temperatures at or below -20°C, viscosity should be at or below 48 mm<sup>2</sup>/s to avoid potentially dangerous loads on the fuel injection pump drive system.

 
 Flash Point
 100 min
 °C
 ISO 3679 ASTM D93

The flash point temperature is the minimum temperature at which the fuel will ignite (flash) on application of an ignition source under specified conditions. It is used to classify fuels for transport and storage according to hazard level; minimum flash point temperatures are required for proper safety and handling of the fuel.

Flash point varies inversely with the fuel's volatility, and biodiesel's flash point can decrease rapidly as the amount of residual alcohol increases. Thus, the biodiesel flash point measurement helps indicate the presence of methanol. Fuel providers must measure the biodiesel flash point prior to blending with

fossil diesel fuel; since the petroleum component's flash point will be much lower, one cannot rely on the blend's flash point measurement to indicate the presence of methanol.

		ISO 5165	
Cetane Number	51 min	ASTM D613	
		JIS K2280	

Cetane number is a measure of the fuel's ignition and combustion quality characteristics. Fuels with low cetane numbers will cause hard starting, rough operation, noise and increased smoke opacity. Finished blends should meet relevant WWFC category limits.

Water	500 max	mg/kg	EN 12937
Water and Sediment	0.05 max	% v/v	ASTM D2709
<b>Total Contamination</b>	24 max	mg/kg	EN 12662 ASTM D2276. D5452. D6217

#### Water and Sediment / Total Contamination

The fuel should be clear in appearance and free of visible water and sediment/total contamination. The presence of these materials can shorten filter life or plug fuel filters, which can lead to engine fuel starvation.

Water accelerates oxidation, dramatically increases corrosivity and promotes microbial growth. Since FAME is capable of absorbing significantly more water than petroleum diesel fuel, it is especially important to dehydrate FAME during production and to minimize the potential to create free water during blending or distribution. The level of water specified is within the solubility level of water in fuel and, as such, does not represent free water.

Esterification and product refining processes, along with feedstock selection, tend to produce many types of contamination, such as by soap. Poor fuel handling practices can introduce other contaminants or compounds that are not soluble in FAME. Small amounts of such contamination will cause fuel filter plugging and injector deposits and should be well-controlled to prevent problems. If water is measured independently from sediment, the fuel should also meet a separate Total Contamination limit.

Engine and fuel injection equipment manufacturers continue to develop fuel systems to reduce emissions and fuel consumption and to improve performance. Injection pressures have increased dramatically; current injectors operate at pressures as high as 2400 bars, and injectors with even higher pressure capabilities are in development. Such levels of injection pressure demand reduced orifice sizes and component clearances, typically ranging from 2 to 5  $\mu$ m in injectors. Small, hard particles, which may remain in the fuel that are carried into these engine parts, are potential sources of injector and subsequent engine failure.

The recommended limits allow comparing measured results to a maximum level acceptable for proper engine operation.

#### **Notes regarding methods:**

Measuring fuel particle contamination necessarily considers total mass, size and number of particles per size class (i.e., particle size distribution). The ISO 4406 protocol provides a means of expressing the level of contamination by size distribution. ASTM D2276 allows line sampling in the field. Whatever method is used, glass fiber filters should be used instead of a membrane filter.

Ash Content	0.001 max	% m/m	ISO 6245 ASTM D482 JIS K2272
Sulfated Ash	0.005 max	% m/m	ISO 3987 ASTM D874 Other: ABNT NBR 984

#### Ash -- Sulfated and Other

Ash is a measure of the amount of metals and other inorganic contaminants contained in the fuel. Ash precursors may be present in three forms—(i) abrasive solids, (ii) soluble metallic soaps, and (iii) residual biodiesel catalyst; when oxidized during combustion, these materials form ash.

Ash has been linked to engine deposits and filter plugging, and metallic soaps can contribute to deposits in the fuel system. Abrasive solids and biodiesel catalyst materials also cause increased wear of internal fuel system engine components exposed to fuel after injection.

Diesel particulate filters (DPF), which began appearing in mid-2000 to achieve extremely low particulate emissions and stringent emission standards, are particularly susceptible to impairment from ash. All ash forming compounds can contribute to the accumulation of material on these filters. Rapid accumulation of ash requires more frequent filter maintenance and will reduce vehicle fuel economy.

The levels specified are considered acceptable for engine performance, and slightly less stringent limits may be acceptable for vehicles without a DPF (see the Worldwide Fuel Charter for limits in Categories 1, 2 and 3). More stringent limits than shown above, however, may be necessary for optimal particulate filter maintenance intervals.

#### Notes regarding test methods:

For the given methods, a large sample size (min 100 g) is needed to obtain precision to three significant digits. More sensitive and precise test methods are needed to determine ash at the lowest levels.

Carbon Residue:			
Ramsbottom, on 100%	0.05 max	% m/m	ASTM D4530
distillation residue			

Carbon residue, which forms on combustion, is a source of particulate matter within the vehicle system. This property serves as a measure of the tendency to form deposits on injectors and in the combustion chamber and should be minimized.

#### *Notes regarding test methods:*

Sample should be distilled under vacuum to prevent polymerization. If distilled at atmospheric pressure, the amount of residue will increase and may produce misleading results.

Corrosion: Ferrous	light rusting, max	Rating	ASTM D665 Procedure A

Corrosion testing indicates potential compatibility problems between fuels and fuel system components made of various metals.

#### Notes regarding test methods:

The rating is made after 3 hours at 50°C by comparing the sample to a standard.

Sulfur	10 max	ppm	EN 20846/20884 ASTM D5453 /D2622 JIS K3541-126 or -7

Many governments regulate sulfur levels in fuel to assure compatibility with emission control systems and to enable these systems to meet emission standards. Biodiesel blends may not exceed the applicable maximum sulfur levels defined for petroleum diesel fuel; fortunately, most biodiesel naturally contains less than 10 ppm sulfur. See the Worldwide Fuel Charter for additional discussion about sulfur's numerous adverse impacts and other limits that may apply in markets with less stringent requirements.

#### Notes regarding test methods:

Use ASTM D5453 for fuels anticipated to have less than 20 ppm sulfur, for the best accuracy when using ASTM test methods.

Phosphorus	4 max	mg/kg	EN 14107	
Pilospilorus	4 IIIdX	ilig/ kg	ASTM D4951, D3231	

Phosphorus will be present in biodiesel at trace levels from phospholipid compounds naturally found in plant oils, and larger quantities can sometimes be found as a result of the use of phosphorus-containing fertilizer to grow the biomass feedstock and inorganic salts that may be contained in used cooking oils. Phosphorus can greatly impair the effectiveness of emission control systems. Its influence is cumulative, which means that even very low levels in the fuel may lead to premature deterioration over time, especially when an engine consumes a significant amount of contaminated fuel. The limit may be lowered when better test methods are available.

#### Notes regarding test methods:

The ISO test method is applicable and accurate down to 4 ppm. ASTM D4951 is precise down to 0.05 % by mass, +/- 10%; lower values may be measurable, depending on the sensitivity of the ICP instrument. ASTM D3231, a method for measuring phosphorus in gasoline, may be acceptable because it uses techniques that decompose the organic materials, so it is independent of the matrix. ASTM D3231 can detect phosphorus at levels below 1 mg/L.

Alkali (Group I) metals (Na+K)	5 max	mg/kg	EN 14108/14109, EN 14538
Alkaline (Group II) metals (Ca+Mg)	5 max	mg/kg	EN 14538

#### Alkali metals (Na + K)

Biodiesel producers who use sodium and potassium to catalyze biodiesel production should remove these metals before allowing the biodiesel to leave the production process. Residual alkali metals can form deposits in fuel injection system components and poison emission control systems. Sodium and potassium are also associated with ash formation.

#### Alkaline metals (Ca + Mg)

Biodiesel producers who use alkaline metals as absorbents during biodiesel production should remove these metals before allowing the biodiesel to leave the production process. Hard water also can contribute alkaline metals to the FAME, so producers should use soft or deionized water during processing. Residual alkaline metals can form deposits in fuel injection system components and poison emission control systems. Calcium soaps can cause injection pumps to stick.

ntentional addition	ASTM D7111
	ntentional addition

#### **Trace Metals/Elements**

Metal compounds should not be added to the fuel.

#### Notes regarding test methods:

ASTM D7111 can measure trace elements between approximately 0.1 to 0.2 mg/kg in middle distillate fuels (with a boiling range between 150°C to 390°C).

#### **Additional Properties**

Consult the Worldwide Fuel Charter for recommended limits and test methods for additional properties—such as cold temperature operability, injector cleanliness, biological growth and lubricity—that require control in the finished biodiesel blend.

#### **Good Housekeeping Practices**

Blenders should manage blends according to the practices outlined in the technical report CEN/TR 15367. Producers should create B100 fuels that have the minimum properties identified in this guideline at the time of delivery or blending. Blenders should evaluate finished products for uniform blending levels and monitor fuel property changes over time including acid number, appearance of free water, and microbial contamination.

Producers and blenders also should be aware that rags saturated with B100, perhaps during spill cleanup, may spontaneously combust and pose a safety hazard under certain conditions. Handlers should consult their professional associations for further guidance.

#### Labeling

Vehicle and engine manufacturers design their products for compatibility with different concentrations of biodiesel. These guidelines are for B100 used for blending at levels up to B5 only. Vehicle and engine manufacturer concerns about biodiesel compatibility and quality increase as the biodiesel content increases and therefore any blends greater than B5 dispensed from pumps should be labeled to identify the blend.

## **Summary of Guidelines**

Property	Value	Units	Test Methods
Ester content	96.5 min	% m/m	EN 14103 mod Other: ABNT NBR 15342
Linolenic Acid Methyl Ester	12.0 max	% m/m	EN 14103 mod
Polyunsaturated acid methyl ester (≥4 double bonds)	1 max	% m/m	prEN 15779
Oxidation Stability:	10 min	h.	prEN 15751 or EN 14112 as
Induction Period	10 min	hr	alternative
Iodine Number	130 max <sup>1</sup>	g I <sub>2</sub> /100 g	EN 14111
Total Acid Number	0.5 max	mg KOH/g	ISO 6618 ASTM D664, D974 JIS K2501 Other: ABNT NBR 14448
Methanol	0.20 max	% m/m	EN 14110 JIS K2536 Other: ABNT NBR 15343
Glycerides			EN 14105
Mono-glyceride	0.80 max	% m/m	EN 14105 ASTM D6584 Other: ABNT NBR 15342
Di-glyceride	0.20 max	% m/m	EN 14105 ASTM D6584 Other: ABNT NBR 15342
Tri-glyceride	0.20 max	% m/m	EN 14105 ASTM D6584 Other: ABNT NBR 15342
Glycerin (glycerol)			
Free glycerin	0.02 max	% m/m	EN 14105/14106 ASTM D6584 Other: ABNT NBR 15341
Total glycerin	0.25 max	% m/m	EN 14105 ASTM D6584 Other: ABNT NBR 15344
Density	report	g/ml	EN ISO 3675 ASTM D4052 JIS K2249 Other: EN ISO 12185, ABNT NBR 7148/14065
Kinematic Viscosity@40°C	2.0 - 5.0 <sup>2</sup>	mm²/s	EN ISO 3104 ASTMD445 JIS K2283

<sup>&</sup>lt;sup>1</sup> This limit may unnecessarily preclude certain feedstocks. Some engine technologies may need a more stringent limit.

 $<sup>^2</sup>$  For temperatures at or below  $-20^{\circ}$ C, viscosity should be at or below 48 mm $^2$ /s to avoid potentially dangerous loads on the fuel injection pump drive system.

Property	Value	Units	Test Methods
			Other: ABNT NBR 10441
Flash Point	100 min	°C	ISO 3679
Flasii Poliit	100 111111	C	ASTM D93
			ISO 5165
Cetane Number	51 min		ASTM D613
			JIS K2280
Water	500 max	mg/kg (ppm)	EN 12937
Water and Sediment	0.05 max	% v/v	ASTM D2709
Total Contamination	24 max	mg/kg	EN 12662
Total Containmation	24 111dX	ilig/kg	ASTM D2276, D5452, D6217
			ISO 6245
Ash Content	0.001 max	% m/m	ASTM D482
			JIS K2272
	0.005 max	% m/m	ISO 3987
Sulfated Ash			ASTM D874
			Other: ABNT NBR 984
Carbon Residue:			
Ramsbottom, on 100%	0.05 max	% m/m	ASTM D4530
distillation residue			
Corrosion: Ferrous	light rusting, max	Rating	ASTM D665 Procedure A
			EN 20846/20884
Sulfur	10 max	ppm	ASTM D5453/D2622
			JIS K3541-1, -2, -6 or -7
Phosphorus	4 max	ma/ka	EN 14107
Phosphorus		mg/kg	ASTM D4951, D3231
Alkali metals (Na+K)	5 max	mg/kg	EN 14108/14109, EN 14538
Alkaline metals (Ca+Mg)	5 max	mg/kg	EN 14538
Trace Metals	no addition		ASTM D7111

# Summary of Test Methods<sup>3</sup> (see main text for additional notes)

Units	CEN/ISO	ASTM	JIS	Other
% m/m	EN 14103 mod			ABNT NBR 15342
% m/m	EN 14103 mod			
% m/m				prEN 15779
hr	prEN 15751 or EN 14112 as alternativ	ve		
g I <sub>2</sub> /100 g	EN 14111			
mg KOH/g	ISO 6618	D664, D974	K2501	ABNT NBR 14448
% m/m	EN 14110		K2536	ABNT NBR 15343
% m/m	EN 14105			
% m/m	EN 14105	D6584		<b>ABNT NBR 15342</b>
% m/m	EN 14105	D6584		ABNT NBR 15342
% m/m	EN 14105	D6584		ABNT NBR 15342
% m/m	EN 14105, EN 14106	D6584		ABNT NBR 15341
% m/m	EN 14105	D6584		ABNT NBR 15344
g/ml	EN ISO 3675	D4052	K2249	EN ISO 12185 ABNT NBR 7148/14065
mm²/s	EN ISO 3104	D445	K2283	ABNT NBR 10441
°C	ISO 3679	D93		
	ISO 5165	D613	K2280	
mg/kg	EN 12937			
% v/v		D2709		
mg/kg	EN 12662	D2276, D5452, D6217		
	% m/m % m/m % m/m hr g l <sub>2</sub> /100 g mg KOH/g % m/m  % m/m  % m/m  % m/m  % m/m  % m/m  % m/m  % m/m	% m/m EN 14103 mod  % m/m EN 14103 mod  % m/m  hr prEN 15751 or EN 14112 as alternative g l <sub>2</sub> /100 g EN 14111  mg KOH/g ISO 6618  % m/m EN 14105  % m/m EN 141	% m/m       EN 14103 mod         % m/m       EN 14103 mod         % m/m       EN 14103 mod         % m/m       PrEN 15751 or EN 14112 as alternative         g I <sub>2</sub> /100 g       EN 14111         mg KOH/g       ISO 6618       D664, D974         % m/m       EN 14100       D6584         % m/m       EN 14105       D6584         g/ml       EN 150 3675       D4052         mm²/s       EN ISO 3104       D445         °C       ISO 3679       D93         ISO 5165       D613         mg/kg       EN 12662       D2276, D5452,         mg/kg       EN 12662       D2276, D5452,	% m/m       EN 14103 mod         hr       prEN 15751 or EN 15751 or EN 14112 as alternative         g I <sub>2</sub> /100 g       EN 14111         mg KOH/g       ISO 6618       D664, D974       K2501         % m/m       EN 14110       K2536         % m/m       EN 14105       D6584         % m/m       EN 150 3675       D4052       K2249         mm²/s       EN ISO 3104       D445       K2283         °C       ISO 3679       D93       K2280         mg/kg       EN 12662       D22709       D22709         mg/kg       EN 12662       D2276, D5452,

 $<sup>^{3}</sup>$  Test methods may be used with B100; consult method to determine if also applicable to blends.

Property	Units	CEN/ISO	ASTM	JIS	Other
Sulfated Ash	% m/m	ISO 3987	D874		ABNT NBR 984
Carbon Residue: Ramsbottom, on 100% distillation residue	% m/m		D4530		
Ferrous Corrosion	rating		D665 Procedure A		
Sulfur	ppm	EN 20846/20884	D5453/D2622	K3541-1	, -2, -6 or -7
Phosphorus	mg/kg	EN 14107	D4951, D3231		
Alkali metals (Na, K)	mg/kg	EN 14108/EN 14109, EN 14538			
Alkaline metals (Ca, Mg)	mg/kg	EN 14538			
Trace metals			D7111		