भारतीय मानक
Indian Standard

IS 15607 : 2016

बायोडीजल (बी100) — फैटी एसिड मिथाइल एस्टर (प्रसिद्धि) — विशिष्टि

(पहला पुनरीक्षण)

Biodiesel (B100) — Fatty Acid Methyl Esters (Fame) — Specification

(First Revision)

ICS 75.160.20

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May 2016

Price Group 2

Petroleum and their Related Products of Synthesis or Biological Origin Sectional Committee, PCD 3

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum and their Related Products of Synthesis or Biological Origin Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

India is a growing economy and the energy is a critical input for socio-economic development. Petro-based fuels meet about 95 percent of the requirement of transportation fuels and the demand has been steadily rising. The domestic crude oil is able to meet only about 20 percent of the demand, while the rest is met through imports. Moreover, the fossil fuels resources are limited, non renewable, polluting and therefore, need to be used prudently. On the other hand, renewable energy sources are indigenous, non polluting and virtually inexhaustible and India is endowed with abundant renewable energy sources. Therefore, their use is required to be encouraged in all possible ways. Biodiesel is one such renewable and alternative fuel which can be used as standalone or its blend in diesel fuel as clean fuel. Biodiesel is non-toxic and biodegradable. This would also help to improve our energy security as well as to reduce exhaust emissions. Moreover, in order to meet the National Policy targets on Biofuels, use in transport sector, use of high concentration of Biodiesel in diesel fuel would play a major role.

Biofuels — Ethanol and Biodiesel — are being increasingly used in many countries as renewable fuels and is better option from environmental aspects also.

Biodiesel is made from virgin or used vegetable oils (both edible and non-edible), waste vegetable oils, by-products of edible oil manufacture like, fatty acids, stearin, etc, and animal fats through trans-esterification or esterification. It is finding use as an automotive fuel for diesel engines at 100 percent concentration, or as an extender. Diesel engines require very little or no modification to use the biodiesel up to 20 percent blend in normal diesel fuel and minor modification for higher percentage blends. The use of biodiesel results in substantial reduction of un-burnt hydrocarbons, carbon monoxide and particulate matter compared to conventional diesel. It has almost no sulphur, no aromatics and about 10 percent built-in oxygen which help in ensuring complete combustion. Its higher cetane number also improves combustion. Biodiesel besides helping in reducing the air pollution, improves lubricity of low sulphur diesel fuels required for meeting Euro III, Euro IV and Euro V emission norms.

While sunflower and rapeseed are the raw materials used in Europe for manufacturing biodiesel, soya bean is mostly used in USA. Thailand uses palm oil. Ireland uses frying oil and animal fats. In India, edible oil is not expected to be used for production of biodiesel as its demand being higher than its domestic production. Keeping in view the climatic conditions and availability of considerable under stocked forest land and non-forest land, it is envisaged that Jatropha Curcas and Pongamia Pinnata ('Honge' or 'Karanja') and such plants may be cultivated profitably in India. The seeds of these plants bear rich in oil which is expected to be used for production for biodiesel through trans-esterification in combination with methanol.

The stability and usability of biodiesel after long term storage is being studied. Annex A provides guidance to consumers of biodiesel (B 100) who may wish to store quantities of fuels for extended periods.

The standard was first published in 2005, wherein requirements for biodiesel were stipulated for its as blend stock up to 20 percent in automotive diesel fuel. The feedstock defined was very limited and both fatty acid alkyl esters of both methanol and ethanol were allowed.

In this revision title and scope have been modified to include use of standalone biodiesel as pure fuel. Further it will cover only fatty acid methyl esters (FAME). Requirements of residuum oil and dye/marker has been deleted. Requirement of glycerides, phosphorus have been included for a check on feedstock. More variety of feedstock that can be used to manufacture biodiesel have been included in line with international practices. This revision is formulated keeping in view of the end use application, production and feed stock availability.

Considerable assistance has been drawn from ASTM D 6751-15 and EN 14214-14, while formulating this standard.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant placed retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BIODIESEL (B100) — FATTY ACID METHYL ESTERS (FAME) — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and tests for biodiesel (B100) — fatty acid methyl esters (FAME) for use in compression ignition engines designed for using as stand-alone fuel and as a blend stock for diesel fuel. B 100 stand-alone can also be used for heating applications and industrial engines.

2 REFERENCES

The standards listed in Annex B contain provisions, which through reference in this text constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex B.

3 TERMINOLOGY

For the purpose of this standard the following terms shall apply.

3.1 Biodiesel — Fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100. The term 'Biodiesel' refers to mono alkyl methyl esters of vegetable oils like rapeseed, soya bean, sunflower, jatropha curcas, karanja, palm stearin, etc, used cooking oils, waste vegetable oils, by- products of edible oil manufacture like free fatty acids, stearin, etc, and other fats.

3.2 B6 to B20 — Fuel blend consisting of 6 volume percent to 20 volume percent of biodiesel (B100) conforming to the requirements of this standard with the remainder being a light middle or middle distillate grade diesel fuel (*see* IS 1460) and meeting the requirements of this specification.

4 REQUIREMENTS

4.1 General

The biodiesel (B100) shall be mono-alkyl methyl esters of long chain fatty acids from vegetable oil and animal fats.

4.2 Additives

It may contain small amounts of hydrocarbon or non-hydrocarbon additives (*see* Annex A) to improve ignition or other characteristics.

In order improve the performance, use of additives is allowed. Suitable fuel additives without known harmful side effects are recommended in the appropriate amount to avoid deterioration of drivability and help emission control durability. Other technical means with equivalent effect may also be used.

In order to improve the oxidation stability of FAME, it is recommended to add suitable oxidation stability enhancing additives to FAME at the production stage and before storage to obtain maximum benefits.

Biocides or biostats may be added which destroy or inhibit the growth of fungi and bacteria, which can grow at fuel water interfaces to give high particulate concentrations in the fuel.

4.3 The materials shall be free from grit, suspended matter and other visible impurities.

4.4 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods as given in col 4 and col 5 of Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable airtight sealed containers as agreed to between the purchaser and the supplier. The use of airtight sealed containers, such as drums or totes, can enhance the storage life of biodiesel. Use of copper and copper-containing alloy materials should be avoided for packing and transportation of biodiesel as such material may cause increased sediment and deposit formation detrimental to the performance. Contact with lead, tin, and zinc can also cause increased sediment levels that can rapidly plug filters and should be avoided.

5.2 Marking

5.2.1 Each container shall be marked with the following information:

a) Name and grade of the material;
b) Indication of the source of manufacture, initials or trade-mark, if any;
c) Volume of the contents, in litre;
d) Batch No./Month and year of manufacture or packing; and
e) Any other information, if required.

5.3 BIS Certification Marking

The container may also be marked with the standard Mark.

5.3.1 The use of the Standard Mark is governed by th provisions of the *Bureau of Indian Standards Act*, 198 and the Rules and Regulations made thereunder. Th details of conditions under which the license for th use of the Standard Mark may be granted t manufactures or producers may be obtained from th Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawi as prescribed in IS 1447 (Part 1).

Table 1 Requirement for Biodiesel

(*Clause* 4.4)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			ISO/ASTM D/EN	[P :] of IS 1448
(1)	(2)	(3)	(4)	(5)
i)	Density[1], at 15°C, g/m³	860 - 900	ISO 3675 / ISO 12185/ D 4052	[P : 16/P : 32]
ii)	Kinematic viscosity at 40°C, cSt	3.5 - 5.0	ISO 3104	[P : 25]
iii)	Flash point (PMCC)[2] °C, *Min*	101	ISO 2719	[P : 21]
iv)	Sulphur[3], mg/kg, *Max*	10.0	D 5453/ISO 20846/ ISO 20884/ISO 13032	
v)	Carbon residue (Ramsbottom)[4], percent by mass, *Max*	0.05	D 4530 /ISO 10370	
vi)	Sulphated ash, percent by mass, *Max*	0.02	ISO 3987	
vii)	Water content[5], mg/kg, *Max*	500	ISO 3733/ ISO 6296/ISO 12937	[P : 40]
viii)	Total contamination, mg/kg, *Max*	24	EN 12662	
ix)	Copper corrosion, 3 h at 50°C, *Max*	1	ISO 2160	[P : 15]
x)	Cetane No., *Min*	51	ISO 5165	[P : 9]
xi)	Acid value, mg KOH/g, *Max*	0.50	EN14104/ASTM D 974	[P : 1/Sec 1][11]
xii)	Methanol[2], percent by mass, *Max*	0.20	EN 14110	
xiii)	Ester content, percent by mass, *Min*	96.5	EN 14103	
xiv)	Monoglycerides[6]content, percent by mass , *Max*	0.7	D6584/EN14105	
xv)	Diglyceride[6]content, percent by mass, *Max*	0.2	D6584/EN14105	
xvi)	Triglyceride[6]content, percent by mass, *Max*	0.2	D6584/EN14105	
xvii)	Free glycerol[7], percent by mass, *Max*	0.02	D 6584/EN14105/ EN14106	
xviii)	Total glycerol[6], percent by mass, *Max*	0.25	D 6584/EN14105	
xix)	Phosphorous[8], mg/kg, *Max*	4.0	D 4951/EN14107	
xx)	Sodium + Potassium[9], mg/kg, *Max*	5	EN 14108/ EN 14109/ EN14538	
xxi)	Calcium + Magnesium[9], mg/kg, *Max*	5	EN 14538	
xxii)	Iodine value[10], g iodine/100 gm, *Max*	120	EN 14111/EN16300	
xxiii)	Oxidation stability, at 110°C, h, *Min*	8	EN 14112/EN15751	
xxiv)	CFPP, °C *Max*:		EN16329/D6371	[P:110]
	a) summer	18		
	b) winter	6		
xxv)	Linolenic acid methyl ester, percent m/m, *Max*	12	EN 14103	
xxvi)	Polyunsaturated (≥ 4 double bonds) methyl ester percent m/m, *Max*	1	EN 15779	

[1] In case of dispute ISO 12185 shall be the refree method.
[2] In case flash point is more than 130°C then testing and reporting of methanol content may not be required.
[3] In case of dispute ISO20846 or ISO 20884 shall be the refree method.
[4] Carbon residue shall be run on 100 percent sample.
[5] In case of dispute ISO 12937 shall be the refree method.
[6] In case of dispute EN14105 shall be the refree method.
[7] In case of dispute EN14105 shall be the refree method.
[8] In case of dispute EN14107 shall be the refree method.
[9] In case of dispute EN14538 shall be the refree method.
[10] In case of dispute EN14111 shall be the refree method.
[11] In case of dispute [P:1/Sec 1] shall be the refree method.

ANNEX A

(*Foreword*)

LONG-TERM STORAGE OF BIODIESEL

A-1 PRINCIPLE

This annex provides guidance to consumers of biodiesel (B 100) who may wish to store quantities of fuels for extended periods. Consistently successful long-term fuel storage requires attention to fuel selection, storage conditions, and monitoring of properties prior to and during storage. This Annex is directed towards biodiesel (B 100) and may be more or less applicable to blends of biodiesel with petro-diesel.

Normally produced biodiesel has adequate stability properties to withstand normal storage without the formation of troublesome amounts of insoluble degradation products, although data suggests some biodiesel may degrade faster than petro-diesel. The selected biodiesel that is to be stored for prolonged periods should be incorporated with suitable concentration of antioxidants to avoid formation of sediment, high acid number, and high viscosities that can clog filters, affect fuel pump operation or plug combustors nozzles or injectors. The selection of biodiesel should result from supplier user discussions.

These suggested practices are general in nature and should not be considered substitutes for any requirement imposed by the warranty of the distillate fuel equipment manufacturers or by federal, state, or local government regulations. Although they cannot replace knowledge of local conditions or good engineering and scientific judgment, these suggested practices do provide guidance in developing an individual fuel management system for the biodiesel fuel user. They include suggestions in the operation and maintenance of existing fuel storage and handling facilities and for identifying where, when and how fuel quality should be monitored.

A-2 TERMINOLOGY

A-2.1 Bulk Fuel — Fuel in the storage facility in quantities over 50 gallon.

A-2.2 Combustor Fuel — Fuel entering the combustion zone of the burner or engine after filtration or other treatment of bulk fuel.

A-2.3 Fuel Contaminants — Foreign materials that make fuel less suitable or unsuitable for the intended use. Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

A-2.4 Fuel-Degradation Products — Those materials formed in fuel after it is produced. Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (acids and gums) may be more or less volatile than the fuel and may cause an increase in injector and nozzle deposits. The formation of degradation products may be catalyzed by contact with metals, especially those containing copper and, to a less extent, iron.

A-2.5 Long-Term Storage — Storage of fuel for longer than 6 months after it is received by the user.

A-3 FUEL SELECTION

The stability properties of biodiesel are not fully understood and appear to depend on the vegetable oil and animal fat sources, severity of processing, and whether additional production plant treatment has been carried out or stability additives are present.

The composition and stability properties of biodiesel produced at specific production plants may be different. Any special requirement of the user, such as long-time storage, should be discussed with the supplier.

A-4 FUEL ADDITIVES

In order to improve the performance, use of additives is allowed. Suitable fuel additives without known harmful side effects are recommended in the appropriate amount to help avoid deterioration of drivability and emission control durability. Other technical means with equivalent effect may also be used.

In order to improve the oxidation stability of FAME, it is recommended to add suitable oxidation stability enhancing additives to FAME at the production stage and before storage to obtain maximum benefits.

Biocides or biostats destroy or inhibit the growth of fungi and bacteria, which can grow at fuel water interfaces to give high particulate concentrations in the fuel. Available biocides are soluble in the fuel phase or the water phase, or both (*see* ASTM D 6469 for additional information).

A-5 TESTS FOR FUEL QUALITY

Test methods for estimating the long term storage stability of biodiesel (B100) are being developed. Modifications of ASTM D 2274 to use glass fiber filters, varying times and temperatures, and the measurement of pre-test and post-test acid number and viscosity appear promising. However, correlation of this test with actual storage stability is unknown and, may depend upon field conditions and fuel composition.

Performance criteria for accelerated stability tests that ensure satisfactory long-term storage of biodiesel (B100) have not been established.

A-6 FUEL MONITORING

A plan for monitoring the quality of bulk fuel during prolonged storage is an integral part of a successful monitoring program. A plan to replace aged fuel with fresh product at established intervals is also desirable.

Stored fuel should be periodically sampled and its quality assessed. ASTM D 4057 provides guidance for sampling. Fuel contaminants and degradation products may settle to the bottom of quiescent tank although detrimental changes to biodiesel can now occur (rising acid value) without causing sediment formation. A bottom or clearance sample, as defined in ASTM D 4057, should be included in the evaluation along with an all level sample.

The quantity of insoluble fuel contaminants present in biodiesel can be determined using ASTM D 6217 with glass fibre filters and abundant washing although no precision or bias testing has been performed with biodiesel using ASTM D 6217.

The acid value of biodiesel appears to exceed its specified maximum before other deleterious fue property changes occur. A conscientious program o measuring the acid value of biodiesel may be sufficien for monitoring biodiesel stability.

A-7 FUEL STORAGE CONDITIONS

Contamination levels in fuel can be reduced by storing in tanks kept free of water, and tankage should have provisions for water draining on a scheduled basis. Water affects the storage life of biodiesel. Water promotes corrosion, and microbiological growth may occur at a fuel-water interface (*see* ASTM D 6469 for additional information). Underground or isothermal storage is preferred to avoid temperature extremes; above ground storage tanks should be sheltered or painted with reflective paints. High storage temperatures accelerate fuel degradation. Fixed roof tanks should be kept full to limit oxygen supply and tank breathing. The use of airtight sealed containers, such as drums or totes, can enhance the storage life of biodiesel.

Copper and copper-containing alloys should be avoided with biodiesel due to increased sediment and deposit formation. Contact with lead, tin, and zinc can also cause increased sediment levels that can rapidly plug filters and should be avoided.

ANNEX B

(*Clause* 2)

LIST OF REFERRED INDIAN/INTERNATIONAL STANDARDS

IS No./International Standards	*Title*
1460 : 2005	Automotive diesel fuel — Specification (*fifth revision*)
1447 (Part 1) : 2000	Petroleum and its products — Methods of sampling (*first revision*)
1448	Methods of test for petroleum and its products
[P : 1/Sec 1] : 2002	Determination of acid number of petroleum products by potentiometric titration (*second revision*)
[P 9] : 2013/ISO 5165	Cetane number (*first revision*)
[P : 15] : 2004/ ISO 2160	Corrosiveness to copper — Copper strip test (*third revision*)
[P : 16] : 1990	Density, relative density or API gravity to crude petroleum and liquid petroleum products by hydrometer method (*third revision*)
[P : 21] : 2012/ ISO 2719	Flash point (closed) by Pensky Martens apparatus (*third revision*)
[P : 25] : 1976	Determination of kinematic and dynamic viscosity (*first revision*)
[P : 32] : 1992	Density and relative density (*second revision*)
[P : 40] : 1987	Water by distillation (*third revision*)
[P : 110] : 1981	Determination of cold filter plugging point of distillate fuels
ISO 3104	Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity
ISO 3675	Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method

IS No./International Standards	*Title*
ISO 3987 : 2010	Petroleum products — Determination of sulfated ash in lubricating oils and additives
ISO 10370	Petroleum products — Determination of carbon residue — Micro method
ISO 12185	Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method
ISO 13032	Methods of test for petroleum and its products: determination of low concentration of sulphur in automotive fuels — energy-dispersive x-ray fluorescence spectrometric method
ISO 20846	Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method
ISO 20884	Petroleum products — Determination of sulfur content of automotive fuels — Wavelength-dispersive X-ray fluorescence spectrometry
ASTM D 2274	'Standard test method for oxidation stability of distillate fuel oil (accelerated method)
ASTM D 4057	Standard practice for manual sampling of petroleum and petroleum products
ASTM D 4530	Standard test method for determination of carbon residue (Micro method)
ASTM D 4951	Standard test method for determination of additive elements in lubricating oils by inductively coupled plasma atomic emission spectrometry
ASTM D 5453	Standard test method for determination of total sulfur in light hydrocarbons, spark ignition engine fuel, diesel engine fuel, and engine oil by ultraviolet fluorescence
ASTM D 6217	'Standard test method for particulate contamination in middle distillate fuels by laboratory filtration'
ASTM D 6371	Standard test method for cold filter plugging point of diesel and heating fuels
ASTM D 6469	Standard guide for microbial contamination in fuels and fuel systems
ASTM D6584	Standard test method for determination of total monoglycerides, total diglycerides, total triglycerides, and free and total glycerin in B-100 biodiesel methyl esters by gas chromatography
EN 14103	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of ester and linolenic acid methyl ester contents
EN14105	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of free and total glycerol and mono-, di-, triglyceride contents
EN14106	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of free glycerol content
EN14107	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of phosphorous content by inductively coupled plasma (ICP) emission spectrometry
EN 14108	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of sodium content by atomic absorption spectrometry
EN 14109	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of potassium content by atomic absorption spectrometry
EN 14111	Fat and oil derivatives. Fatty acid methyl esters (FAME)- Determination of iodine value
EN 14112	Fat and oil derivatives. Fatty acid methyl esters (FAME). Determination of oxidation stability (accelerated oxidation test)
EN14538	Fat and oil derivatives. Fatty acid methyl ester (FAME). Determination of Ca, K, Mg and Na content by optical emission spectral analysis with inductively coupled plasma (ICPAES)
EN15751	Automotive fuels. Fatty acid methyl ester (FAME) fuel and blends with diesel fuel. Determination of oxidation stability by accelerated oxidation method
EN 15779	Petroleum products and fat and oil derivates. Fatty acid methyl esters (FAME) for diesel engines. Determination of polyunsaturated (≥ 4 double bonds) fatty acid methyl esters (PUFA) by gas chromatography
EN16300	Automotive fuels. Determination of iodine value in fatty acid methyl esters (FAME). Calculation method from gas chromatographic data
EN16329	Diesel and domestic heating fuels. Determination of cold filter plugging point. Linear cooling bath method