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AUTOMOTIVE BIODIESEL (B100) – Specification

ZAMBIA BUREAU OF STANDARD

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This Draft Zambian Standard was prepared by the…… Technical Committee, (TC…), upon which the following organizations were represented:

Biofuel Association of Zambia
Copperbelt Africa Investment
CFAO Motor Zambia Limited
Energy Regulation Board
INDENI Petroleum Refinery Company Limited
Ministry of Energy - Department of Energy
Puma Energy Zambia PLC
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FOREWORD

The preparation of this Standard has been undertaken by the …….. Technical Committee (TC…), in accordance with the procedures of ZABS. All users should ensure that they have the latest edition of this publication as standards are revised from time to time.

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The revision of this standard was necessary to ensure that the technological advancements in the sector are taken into consideration.

In the preparation of this standard, the following publication was consulted:

SANS 1935: 2004 *Automotive Biodiesel Fuel*, published by the South African Bureau of Standards

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ZAMBIA BUREAU OF STANDARDS

Draft Zambian Standard

AUTOMOTIVE BIODIESEL (B100) - Specification

1.0 SCOPE

This Zambian Standard specifies requirements and test methods for marketed and delivered Biodiesel to be used either as automotive fuel for diesel engines at 100 % concentration, or as an extender for automotive fuel for diesel engines. At 100 % concentration it is applicable to fuel for use in diesel engines designed or subsequently adapted to run on 100 % Biodiesel.

NOTE: In Zambia the standard for automotive fuel for diesel engines is ZS 369.

2.0 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. All standards are subject to revision and, since any reference to a standard is deemed to be a reference to the latest edition of that standard, parties to agreements based on this standard are encouraged to take steps to ensure the use of the most recent editions of the standards indicated below.

ZS ASTM D1160	Standard test method for distillation of petroleum products at reduced pressure
ZS EN 116	Diesel and domestic heating fuels - Determination of cold filter plugging point.
ZS EN 12662,	Liquid petroleum products - Determination of contamination in middle distillates.
ZS EN 14103	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of ester and linolenic acid methyl ester contents.
ZS EN 14104	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of acid value.
ZS EN 14105	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of free and total glycerol and mono-, di- and triglyceride content (Reference method).
ZS EN 14106	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of free glycerol content.
ZS EN 14107	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of phosphorus content by inductively coupled plasma (IPC) emission spectrometry.
ZS EN 14108	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of sodium content by atomic absorption spectrometry.
ZS EN 14109	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of potassium content by atomic absorption spectrometry.
ZS EN 14110	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of methanol content.
ZS EN 14111	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of iodine value.
ZS EN 14112	Fat and oil derivatives - Fatty acid methyl esters (FAME) - Determination of oxidation stability (Accelerated oxidation test).
ZS 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
ZS ISO 2160	Petroleum products - Corrosiveness to copper - Copper strip test.

ZS ISO 3104	Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity.
ZS ISO 3170	Petroleum liquids - Manual sampling.
ZS ISO 3171	Petroleum liquids - Automatic pipeline sampling.
ZS ISO 3675	Crude petroleum and liquid petroleum products - Laboratory determination of density or relative density - Hydrometer method.
ZS ISO 3679	Determination of flash point - Rapid equilibrium closed cup method.
ZS ISO 3987	Petroleum products - Lubricating oils and additives - Determination of sulphated ash.
ZS ISO 4259	Petroleum products - Determination and application of precision data in relation to methods of test.
ZS ISO 5165	Petroleum - Diesel fuels - Determination of the ignition quality of diesel fuels – Cetane, engine method.
ZS ISO 10370	Petroleum products - Determination of carbon residue - Micro method.
ZS ISO 12185	Crude petroleum and petroleum products - Determination of density – Oscillating U-tube method.
ZS ISO 12937	Petroleum products - Determination of water - Coulometric Karl Fisher titration method.
ZS ISO 13759	Petroleum products - Determination of alkyl nitrate in diesel fuels – Spectrometric method.
ZS ISO 14596	Petroleum products - Determination of Sulphur content - Wavelength-dispersive X-ray fluorescence spectrometry.
ZS ISO 20846	Petroleum products - Determination of Sulphur content of automotive fuels – Ultraviolet fluorescence method.
ZS ISO 20884	Petroleum products - Determination of Sulphur content of automotive fuels - Wavelengths- dispersive X-ray fluorescence spectrometry.
SANS 10368	Transport of low-hazard goods in bulk - Emergency information for road vehicles.
ZS 718	Low Sulphur Gas Oil: Specification

3.0 DEFINITIONS

For the purposes of this standard, the following definitions shall apply:

3.1. **Additive:** A compound added to the Biodiesel fuel to improve either the performance of the Biodiesel or its storage stability or both.

3.2. **Biodiesel**: A fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats designated as B100.

3.3. **Vegetable Oil (Edible/Non-Edible):** Oil extracted from plant seeds.

4.0 REQUIREMENTS

4.1. General

4.1.1. The automotive Biodiesel fuel shall contain, principally, mono-alkyl methyl esters of long chain fatty acids derived from vegetable oil. This standard dwells on Biodiesel produced using methanol.

4.1.2. Suitable fuel additives without known side effects may be used to help avoid deterioration of driveability and emissions control durability. Other technical means that exhibit an effect equivalent to that of additives can also be used.

4.1.3. The fuel may contain applicable small quantities of colouring materials which are documented as harmless

to give it a distinctive colour.

4.1.4. The fuel shall be clear and free of visible water, sediment, suspended matter and any other contaminant that is documented as likely to cause malfunctioning of equipment designed to use this type of fuel, either as a blend or in its 100 % concentration form.

4.2. Physical and chemical properties

The fuel shall comply with all the requirements given in Table 1.

NOTE 1: In case of a need for identification of Biodiesel, it is recommended that a method based on the characterization of fatty acid methyl esters by LG/GC, in accordance with EN 14331, be used.

NOTE 2: In case of a need to identify the source oil of Biodiesel, the iodine value of the Biodiesel can be calculated by the method presented in Annex B.

5.0 METHOD OF TEST

5.1. For all tests, use samples taken in accordance with Annex D.

5.2. For all properties, use the applicable test method or, when relevant, one of the applicable test methods listed in column 3 of Table 1.

5.3. The limiting value for the carbon residue given in Table 1 is based on product prior to the addition of ignition improver, if used. If a value exceeding the limit is obtained on finished fuel in the market, use ZS ISO 13759 to determine the presence of a nitrate-containing compound. If an ignition improver is thus proved present, the limit value for the carbon residue of the product under test cannot be applied. The use of additives does not exempt the manufacturer from meeting the requirement of maximum 0.30 % mass fraction of carbon residue prior to the addition of additives.

5.4. Precision and dispute

5.4.1. All test methods referred to in this standard include a precision statement according to ZS ISO 4259.In cases of dispute, use the procedures described in ZS ISO 4259 for resolving the dispute, and interpretation of the results based on the test method precision shall be used. However, the methods currently available for total contamination, ester content, tri-glyceride content, free glycerol and alkaline metals (total of Na and K) do not meet the 2R requirement of ZS ISO 4259 at the limit in Table 1.

5.4.2. Test methods for petroleum products may contain precision data specific to these products. When the same methods are used to test Biodiesel fuel, the precision data might differ; such data are given in Annex A.

5.4.3. In cases of dispute concerning density, use ZS ISO 3675 with the determination carried out at 15°C.

NOTE: Available conversion tables are not applicable to Biodiesel.

5.4.4. In cases of dispute concerning free glycerol, the reference method is ZS EN 14105.

5.4.5. For the determination of Cetane Number alternative methods may also be used in cases of dispute, provided that these methods originate from a recognized method series, and have a valid precision statement, derived in accordance with ISO 4259 and, which demonstrates precision at least equal to that of the referenced method. The test result, when using an alternative method, shall also have a demonstrable relationship to the result obtained when using the reference method.

Table 1 - Requirements for Automotive Biodiesel

Property	Requirements	Test method[a]
Ester content[a], % mass fraction, min	96.5[b]	EN 14103
Density at 15 ºC[c], kg/m^3	860 – 900	ISO 3675, ISO 12185
Kinematic viscosity at 40 ºC[d], mm^2/s	3.5 – 5.0	ISO 3104
Flash point, ºC, min	120	ISO 3679
Sulphur content, mg/kg, max	10.0	ISO 20846, ISO 20884
Carbon residue (on 10 % distillation residue)[f], % mass fraction, max	0.3	ISO 10370
Cetane Number[g], min	51.0	ISO 5165
Sulfated ash content, % mass fraction, max	0.02	ISO 3987
Water content, % mass fraction, max	0.05	ISO 12937
Total contamination[h], mg/kg, max	24	EN 12662
Copper strip corrosion (3 h at 50 ºC), rating, max Class	1	ISO 2160
Oxidation stability, at 110 ºC, h, min	6	EN 14112
Acid value, mg KOH/g, max	0.5	EN 14104
Iodine value, g of iodine/100g of FAME, max	140	EN 14111
Linolenic acid methyl ester, % mass fraction, max	12	EN 14103
Polyunsaturated (> = 4 double bonds) methyl esters[i], % mass fraction, max	1	EN 15779
Methanol content, % mass fraction, max	0.2	EN 14110
Monoglyceride content, % mass fraction, max	0.8	EN 14105
Diglyceride content, % mass fraction, max	0.2	EN 14105
Triglyceride content[j], % mass fraction, max	0.2	EN 14105
Free glycerol[j], % mass fraction,	max 0.02	EN 14105, EN 14106
Total glycerol, % mass fraction, max	0.25	EN 14105
Group I metals (total of Na and K)[k], mg/kg, max	5.0	EN 14108, EN 14109
Group II metals (total of Ca and Mg), mg/kg, max	5.0	EN 14538
Phosphorus content, mg/kg, max	10.0	EN 14107
Cold Filter Plugging Point (CFPP)m Winter, °C, max Summer, °C, max	-4 +3	EN 116

Appearance	Visibly free of suspended or precipitated contaminants (clear and bright)	Visual

a. See Clause 5.4.1

b. The addition of non-fatty acid methyl esters components other than additives is not allowed (see 4.1.1).

c. Density may be measured by ISO 3675 over a range of temperatures from 20 °C to 60 °C. A temperature correction shall be made according to the formula given in Annex C (see 5.4.2).

d. If CFPP is -20 °C or lower, the viscosity measured at -20 °C shall not exceed 48 mm^2/s. In this case, ISO 3104 is applicable without the precision data owing to non- Newtonian behavior in a two-phase system.

e. A 2 ml sample and apparatus equipped with a thermal detection device shall be used.

f. ASTM D 1160 shall be used to obtain the 10 % distillation residue (see 5.3)

g. See 5.4.5.

h. Pending development of a suitable method, EN 12662 shall be used. The precision of EN 12662 is, however, poor for biodiesel.

i. Suitable test method to be developed.

j. See 5.4.1 and 5.4.4.

k. See 5.4.1. See Annex A for precision data for the total of Na and K.

6.0 PACKING, MARKING AND PLACARDING

6.1. Packing

Except when transported in bulk, the Biodiesel shall be packed in sound, clean, dry drums not deleteriously affected by the Biodiesel. The drums shall be so closed that leakage and contamination of the Biodiesel is prevented during normal handling and transport.

6.2. Marking

The following information shall appear in prominent, legible and indelible marking on each drum or, in the case of Biodiesel filled in bulk storage tanks or bulk carriers, in the storage and consignment documents of each bulk carrier:

a. the manufacturer's (or the supplier's) name or the brand name of the product or both;

b. a description of the product, i.e. "BIODIESEL FUEL – B100";

c. batch identification; and

d. the quantity of the contents.

6.3. Placarding

Where biodiesel is transported in road tankers or vehicles carrying portable tanks that exceed 3 500 kg GVM, placarding shall comply with SANS 10368.

ANNEX A
(Normative)

Table A.1 – Table of Precision Data

Property	Test method	Data for pure FAME[a,b,c]
Kinematic viscosity at 40 ºC, mm^2/s	ISO 3104	r = 0.0011X R = 0.018X
Sulphur content, mg/kg	ISO 20846 ISO 20884	r = 0.0285X + 2 R=0.1088X+2 r = 0.026X + 1,356 R = 0.567X + 1,616
Distillation, ºC	ASTM D 1160	r = 2.0 R = 3.0 (90 % volume fraction distilled)
Cetane Number	ISO 5165	r = 2.4 R = 5.0
Sulfated ash content, % mass fraction	ISO 3987	r = 0.06X0.85 R = 0. 142X0.85
Total contamination, mg/kg	EN 12662[d]	r = 2.24 R = 13.6
CFPP, ºC	EN 116	Not available
Total of Na and K, mg/kg	EN 14108 EN 14109	r = -0.017X + 0.512 R = 0.305X + 1.980
[a] r is the repeatability in accordance with ISO 4259. [b] R is the reproducibility in accordance with ISO 4259. [C] X is the mean of two results being compared. [d] The precision of EN 12662 is poor for Biodiesel.		

ANNEX B
(Informative)

Calculation of iodine value

B.1 Scope

This method describes a procedure for calculating the iodine value of 100 % concentration Biodiesel orBiodiesel extracted from blends with diesel fuel. In case of dispute on the iodine value this method shall be used as a substitute for EN 14111.

NOTE This method is adapted for Biodiesel from the AOCS recommended practice Cd 1c - 85 for the determination of the iodine value of edible oil from its fatty acid composition.

B.2 Definition

This method is used to calculate the iodine value expressed in g 12/100 g sample from the percentageby mass of methyl esters as determined by either EN 14103 (neat Biodiesel) or EN 14331 (Biodiesel extracted from blends with diesel fuel).

B.3 Procedure

B.3.1 Check the methyl ester composition of the sample by using the appropriate method as described in B.2.

NOTE: The total methyl esters thus revealed should equal 1.00 after the deduction of the methyl ester C17 used for internal standard in EN 14103.

B.3.2 Use the percentage by mass obtained in B.3.1 to calculate the sample's iodine value, being the sum of the individual contributions of each methyl ester, obtained by multiplying the methyl ester percentage by its respective factor (see table B.2). The factor for each constituent of Biodiesel is given in Table B.1

Table B.1 - Methyl ester factors

Methyl ester	Factor
Methyl ester of saturated fatty acids	0
Methyl hexadecenoate (Methyl palmitoleate) C16:1	0.950
Methyl octadecenoate (Methyl oleate) C18:1	0.860
Methyl octadecadienoate (Methyllineolate) C18:2	1.732
Methyl octadecatrienoate (Methyllinolenate) C18:3	2.616
Methyl eicosenoate (Methyl gadoleate) C20:1	0.785
Methyl decasenoate (Methyl erucate) C22:7	0.723

B.4 Expression of Result

Iodine value (calculated from the methyl ester composition) = Xg Iodine/100g of FAME. The result should be reported to the decimal place.

NOTE 1: In 1994 the AOCS uniform Methods Committee reviewed the coefficients used and concluded that no changes were necessary at that time. The reasoning behind that choice is that triple the molecular weight of methyl ester is almost identical to the molecular weight of the corresponding triglyceride.

NOTE 2: For samples with unsaponifiable content greater than a mass fraction of 0,5 % or those containing a significant additive content, the calculated value tends to be higher than the true value.

NOTE 3: The calculated result tends to be lower than the true value in samples with a lower Iodine value.

Table B.2 – Calculation example

Methyl ester of the following acids	Percentage mass fraction	Factor	Contribution
Myristic C14:0	0,3	0	0
Palmitic C16:0	4,0	0	0
Palmitoleic C16:1	1,1	0.950	1,0
Stearic C18:0	2,0	0	0
Oleic C18:1	60,5	0.860	52.0
Linoleic C18:2	19,8	1.732	343
Linolenic C18:3	9,4	2.616	24.6
Eicosanoic (Euricic) C20:0	0,4	0	0
Eicosanoic (Beheric) C20:1	0,7	0.785	0.6
Docosanoic (Gadoleic) C22:0	0,7	0	0
Docosenoic (AraChidic) C22:1	1,1	0.723	0.8
		Calculated Iodine value	113.3

ANNEX C
(Normative)

Correction factor for calculation of density of Biodiesel

The following equation shall be used for the calculation of density of Biodiesel at 15 ºC:

$\rho_{(15)} = \rho_{(T)} + 0.723\ (T - 15)$

Where,

$\rho_{(15)}$ is density of Biodiesel at 15°C;

$\rho_{(T)}$ is density at any other temperature in the range of 20 °C to 60 °C;T

is any temperature in the range of 20 °C to 60 °C

ANNEX D
(Normative)

Sampling and compliance with this standard

D.1 Sampling

D.1.1 General

The sampling procedure(s) given in D.1.3 shall be applied in determining whether a lot complies with the relevant requirements of this standard. The samples so drawn shall be deemed to represent the lot.

D.1.2 Definitions

D.1.2.1 Non-Compliant
A test sample of the Biodiesel that fails in one or more respects to comply with the relevant requirements of this standard.

D.1.2.2 Lot

That quantity of Biodiesel in containers bearing the same brand name or trade mark, grade designation and batch identification, from one manufacturer, and submitted at anyone time for inspection and testing.

D.1.3 Samples for inspection and testing

After checking for compliance with the relevant requirements of 6.1 and 6.2, use the relevant sampling procedure given in ISO 3170 or ISO 3171 (or both), as appropriate, to determine whether a lot complies with this standard, and deem the samples so taken to represent the lot for the respective properties. Particular attention shall be paid to compliance with any guidance on sampling containers, which is included in the test method standard.

D.2 Compliance with this standard

The lot shall be deemed to comply with the relevant requirements of this standard if, on inspection of the containers in the lot, and on testing of the samples taken in accordance with D.1.3 and 5.1, no non-compliance is found.

ANNEX E
(Informative)

Quality verification of Automotive Biodiesel fuel

When a purchaser requires ongoing verification of the quality of Biodiesel fuel, it is suggested that instead of concentrating solely on evaluation of the final product, the purchaser should also direct their attention to the manufacturer's quality system. In this connection it should be noted that ZS ISO 9001 covers the provision of an integrated quality system.

ANNEX F
(Informative)

Biodiesel may also be derived from transesterification using any other catalyst. The final product shall meet the limiting requirements stipulated in Table 1.