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AUTOMOTIVE DIESEL FUEL — SPECIFICATION

(*Seventh Revision* of IS 1460)

(ICS 43.060.01; 75.160.20)

Petroleum and their Related Products of Synthesis or Biological Origin Sectional Committee, PCD 03	Last date for receipt of comment is **26 July 2022**

FOREWORD

(*formal clause will be added later*)

This Indian Standard of automotive diesel fuel refers to Bharat stage VI.

The automotive diesel fuel continues to be the main fuel in India for both public as well as commercial transport and this trend is expected to continue for a long time to come because of favourable economic benefits associated with its use. The fuel demand pattern in our country is, therefore, heavily tilted towards automotive diesel fuel and there is an imperative need to maximize its production to meet the requirements of consumers. Accordingly, the requirements of automotive diesel fuel for vehicles meeting Bharat Stage VI Norms are furnished in Table 1. The date and area for implementation of these specifications are as per the notification issued by the Competent Authority from time-to-time. Nothing in this standard shall, however, preclude observance of the regulations, which may be more restrictive.

Automotive diesel fuel is a complex mixture of hydrocarbons that varies depending on crude source and manufacturing process. Consequently, it is impossible to define the exact composition of automotive diesel fuel. This specification has therefore evolved primarily as a performance specification rather than a compositional specification. It is acknowledged that this largely relies on accumulated experience, therefore the specification limits automotive fuels to those made from conventional sources.

This standard was first published in 1959 and subsequently revised in 1968 and amended in 1971. It was again revised in 1974 by taking into consideration the requirement of diesel fuel and the supply and demand pattern of middle distillates at that time in the country. In view of lowering of cetane number of ‘Grade Special’ from 45 to 42, it was felt unnecessary to retain Grade A and names of Grade Special and Grade B were also changed to High Speed Diesel Fuel (HSD) and Light Diesel Oil (LDO), respectively. Further, as a result of lowering of flash point of HSD from 55°C to 38°C, the Pensky Martens test method was replaced by Abel Flash Point test method. For determination of sulphur content, an alternate method, namely, Quartz tube method was included. An additional requirement for total sediment determination was included in the second revision to ensure the stability of the fuel.

Through Amendment No. 1 in February 1980 an additional requirement for ‘Cold Filter Plugging Point’ (CFPP) was introduced in the standard to take into account the performance of the high speed diesel oil while in operation in the low temperature areas, CFPP being more realistic indicator of filter clogging than pour point which can be reduced by doping. A flash point requirement of 66°C, *Min* for fishing vessels of 12 m and above was also introduced in the standard through Amendment No. 2, October 1981. Further, on a proposal received from

the Ministry of Petroleum & Natural Gas (MoP&NG), to absorb the surplus stock of heavy naphtha into high speed diesel oil with a view to tackle the imbalance in the production of various petroleum products and to meet the increasing demand of HSD, the flash point requirement of HSD was relaxed from 38°C to 32°C through Amendment No. 3, October 1985.

Third revision of this standard was published in 1995. In third revision, the requirements of several characteristics of High Speed Diesel Fuel were upgraded since diesel of this quality could be obtained with good yield without much difficulty by using multifunctional additives and/or carrying out minor process changes, wherever required. The requirement of cetane number was modified from 42, *Min* to 45, *Min*, which resulted into improved fuel economy, cold startability, engine life, reduced engine noise and exhaust emissions.

The kinematic viscosity was specified at a standard temperature of 40°C and accordingly its requirement was changed to a narrower limit of 1.8 to 5.0 cSt. The test procedure for total sediments was replaced by UOP 413 (Modified) as it appeared to be known for better correlation with regard to fuel stability in actual field storage conditions. The requirement of sulphur content was retained as 1.0 percent by mass, *Max* and it was envisaged that due to detrimental effects of the combustion products of sulphur on the life of engine and environment, more stringent requirement would be stipulated in future as the refineries equip themselves with necessary processing facilities. Since the method for determination of sulphur by quartz tube had become obsolete, the reference to this method had been dropped from this standard. A limit was stipulated for the first time on cold filter plugging point test to ensure a smooth operation at low ambient temperatures. Pour point was specified separately for winter and summer grades.

Amendment No. 1, March, 1997 provided a relaxation for diesel fuel processed from Assam Crude with respect to the requirement of cetane number as 42, *Min* up to December, 1999. The requirement of sulphur content was tightened to align with Notification No. GSR 176(E) dated 2.4.1996 issued by MOEF.

Amendment No. 2, February, 1999 was issued to notify the HSD specification for the year 2000 A.D. and to tighten the requirement of total sediment as 1.5 mg/100 ml, *Max*.

Amendment No. 3, March, 2000 the requirement of cetane index, 46, *Min* (43, *Min* for product from Assam crude) was introduced as an alternate to cetane number. The requirement of total sulphur for supplies to Indian Navy for defence use was stipulated as 0.20 percent, by mass, *Max*.

Through Amendment No. 4 the requirement of sulphur content as 0.05 percent by mass, *Max* for notified areas has been introduced. A requirement of lubricity was introduced for high speed diesel fuel of low sulphur content.

Fourth revision of this standard was published in 2000. In fourth revision, all the four amendments of its previous version were incorporated and some of the following requirements were modified and incorporated.

a) Acidity, total, restricted from 0.30 to 0.20 mg of KOH/g, *Max*;
b) Carbon residue reduced from 0.35 to 0.30 percent by mass, *Max*;
c) Cetane number tightened from 45, *Min* to 48, *Min*;
d) Distillation has been brought into two categories, namely volume recovered at 350°C, 85 percent, *Min* and volume recovered at 370°C, 95 percent, *Min*;
e) Flash point modified from 32 to 35°C, *Min*;
f) Kinematic viscosity brought to a narrower range 2.0 to 5.0 cSt at 40°C;

g) Density range tightened from 820-880 to 820-860 kg/m^3;
h) Total sulphur reduced to 0.25 percent, maximum to meet the requirement given in the notification issued by MOEF; and
j) Cold Filter Plugging Point (CFPP) tightened upto 6°C, *Max* for winter and 18°C, *Max* for summer.

Fifth revision of this standard was published in 2005. Considering the ever increasing stringency in the requirements of automotive diesel fuel to meet the emerging emission norms, two separate standards covering specifications for HSD and LDO were published in 2005. In fifth revision of this specification two amendments of fourth revision version were incorporated. These amendments include the incorporation of 5 percent (*v/v*) bio-diesel in the high speed diesel and a target specification for high speed diesel fuel for the vehicles meeting Bharat Stage III (EURO III equivalent) Emission Norms. The nomenclature of high speed diesel was also changed to automotive diesel fuel. The following requirements were modified in the fifth revision:

a) Cetane number tightened from 48, *Min* to 51, *Min*;
b) Distillation temperature for 95 percent volume recovered was brought down from 370°C to 360°C *Max*;
c) Kinematic viscosity brought to narrower range from 2.0 to 5.0 cSt to 2.0 to 4.5 cSt at 40°C
d) Density range tightened from 820-860 to 820-845 kg/m^3;
e) Total sulphur reduced to 350 ppm (mg/kg) maximum for BS III and 50 ppm maximum for BS IV.

Through Amendment 1, September, 2007 few editorial corrections made in the test method number(s) mentioned in tables.

Through Amendment 2, March 2010, reference of ISO 4259 for interpretation of test results based on the precision statements of respective test methods was made. The specification for automotive diesel fuel for the vehicles meeting Bharat Stage IV/Euro IV Vehicular Emissions Norms based on the Auto Fuel Policy, issued by the Ministry of Petroleum and Natural Gas, Government of India, has been incorporated. Amendment 3, September 2012, resulted in deletion of provision for inclusion of marker.

Sixth revision of this standard was published in 2017. In sixth revision, requirements for Bharat stage II (BS II) and Bharat Stage III (BS III) automotive diesel fuel were deleted. Also, contents of Amendment No.1 issued in September, 2007 and Amendment No. 2 issued in March, 2010 were incorporated in this revision. With the deletion of BS II automotive diesel fuel, the requirements for distillation recovery at 350°C, distillation recovery at 370°C and sediments were also deleted. The oxygen content requirement for BS IV grade was replaced with FAME content in line with BS VI grade specification. For formulating the specification for automotive diesel fuel for the vehicles meeting Bharat Stage VI Vehicular Emissions Norms considerable assistance was derived from the report submitted by the Expert Committee to the Government of India, on Auto Fuel Vision & Policy 2025 and from the Extraordinary Gazette Notification No. 651 dated 16th September 2016 Central Motor Vehicles (11th Amendment) Rules, 2016 issued by Ministry of Road Transport and Highways. Comments/data received from all stakeholders were considered.

Through Amendment No. 1, September 2018, title of ISO 20846 : 2011 was modified in reference clause.

In this seventh revision, requirement for Bharat stage IV (BS IV) automotive diesel fuel have been deleted from Table 1. Also, content of Amendment No. 1 issued in September, 2018 has been incorporated in this revision.

It is recognized that there are some applications where for technical or other reasons, limits may be different from those specified in this standard or additional requirements may be necessary. This standard does not cover such special applications, which are subject to agreement between the purchaser and the supplier. This standard, unless otherwise, provided by agreement between the purchaser and the supplier, prescribes the required properties of automotive diesel fuel at the time and place of delivery.

Reference to various other overseas standards like ISO, ASTM, IP, etc. has been given as presently there is no Indian Standard available for them. Once the Indian Standards are formulated, the references will be modified accordingly.

Bibliographical references for this standards have been given in Annex B.

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 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

1.1 This standard prescribes the requirements, sampling procedure and test methods for automotive diesel fuel (earlier also known as High Speed Diesel Fuel, HSD). It is applicable to automotive diesel fuel for use in diesel engine vehicles and stationary diesel engines, designed to run on automotive diesel fuel.

1.2 This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions ofthis 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 below:

IS / ISO No.	*Title*
1260 (Part 1) : 1973	Pictorial marking for handling and labeling goods Part 1 Dangerous goods (*first revision)*
1447 (Part 1) : 2021	Methods of sampling of petroleum and its products Part 1 Manual sampling (*second revision*)
1448	Method of test for petroleum and its products
(Part 2) : 2007/ISO 6619 :1988	Petroleum products and lubricants — Neutralization number potentiometric titration method (*second revision*)
(Part 4/Sec 1) : 2021	Part 4/Section 1 Determination of ash (*fourth revision*)
(Part 8) : 2012/ISO 4262 : 1993	Determination of carbon residue — Ramsbottom method (*second revision*)
(Part 9) : 2019/ISO 5165 : 2017	Determination of the ignition quality of diesel fuels — Cetane engine method (*second revision*)

(Part 10/Sec 2) : 2021/ISO 3016 : 2019	Petroleum and related products from natural or synthetic sources Section 2 Determination of pour point (*third revision*)
(Part 15) : 2004/ISO 2160 : 1998	Petroleum products — Corrosiveness to copper strip test (*third revision*)
(Part 16) : 2014/ISO 3675 : 1998	Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method (*fourth revision*)
(Part 18) : 2020	Distillation of petroleum products (*third revision*)
(Part 20) : 2019/ISO 13736 : 2013	Determination of Flash Point — Abel Closed-Cup Method (third revision)
(Part 21) : 2019/ISO 2719 : 2016	Determination of flash point by Pensky-Martens closed cup apparatus
(Part 25/Sec 1) : 2018/ISO 3104: 1994	Transparent and opaque liquids Section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (*second revision*)
(Part 32) : 2019/ISO 3838 : 2004	Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary stoppered pyknometer and graduated bicapillary pyknometer methods (*third revision*)
(Part 34) : 1979	Determination of sulphur in petroleum products (lamp method) (*second revision*)
(Part 110) : 1981	Cold filter plugging point of distillate fuels
IS 1448 (Part 149) : 2020/ISO 12156-1 : 2018	Diesel fuel — Assessment of lubricity using the high-frequency reciprocating rig (HFRR) — Test method (second revision)
(Part 154) : 2012/ISO 12205 : 1995	Determination of oxidation stability of middle distillate fuels
IS 1448 (Part 160) : 2017/ISO 20846 : 2019	Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method
IS 1448 (Part 161) : 2017/ISO 13032 : 2012	Determination of low concentration of sulfur in automotive fuels — Energy dispersive X-ray fluorescence spectrometric method
IS 1448 (Part 167) : 2018/ISO 12185 : 1996	Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method
IS 1448 (Part 174) : 2020/ISO 4264 : 2018	Petroleum products — Calculation of cetane index of middle-distillate fuels by the four- variable equation
IS 1448 (Part 182) : 2020/ISO 12937 : 2000	Petroleum products — Determination of water —Coulometric Karl Fischer titration method
IS 1448 (Part 186) : 2021/ISO 23581 : 2020	Petroleum products and related products — Determination of kinematic viscosity — Method by Stabinger type viscometer
IS 1448 (Part 188) : 2021/ISO 6618 : 1997	Petroleum products and lubricants — Determination of acid or base number — Colour-indicator titration method
IS 1448 (Part 189) : 2021/ISO 10370 : 2014	Determination of carbon residue — Micro method
IS 15607 : 2022	Biodiesel, Diesel Fuel Blend (B 8 to B 20) — Specification (*first revision*)

3 REQUIREMENTS

3.1 General

3.1.1 The material shall be clear, bright and free from sediments, suspended matter and undissolved water at normal ambient fuel temperature.

3.1.2 *Composition*

The material shall be hydrocarbon oils derived from petroleum. The use of fuel additives is permitted in order to improve the performance quality. Suitable fuel additives without known harmful side-effects are recommended in appropriate concentration to help to avoid deterioration of drivability and emissions control durability.

3.1.3 This fuel shall not contain any residuum oil.

3.1.4 Bio-diesel (Fatty Acid Methyl Ester, FAME) conforming to IS 15607 may be blended up to 7 percent(*v/v*) with automotive diesel fuel [*see* Table 1, Sl No. (xxi)] by authorized agents. Stabilizing agents, as required, shall be incorporated. Percentage of bio-diesel blended shall be ensured & declared by suppliers using a certification process.

3.1.5 The use of dyes or markers is permitted.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the appropriate methods prescribed in col 4 of Table 1.

4 SAMPLING

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

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable containers prescribed by Petroleum and Explosives Safety Organization (PESO) from time to time.

5.2 Marking

5.2.1 The material shall be supplied in accordance with the marking and shipping regulations laid down by Petroleum and Explosives Safety Organization (PESO) from time to time.

5.2.2 Each container shall be marked with the following information:

a) Name and grade of the material;
b) Indication of the source of manufacturer, initials or trade-mark, if any;
c) Volume of the contents, in litres;
d) Year and month of manufacturing or packing; and
e) Any other statutory requirements.

5.2.3 Each container shall also be marked with the caution label 'Highly Flammable' together with the corresponding symbol for labeling dangerous goods [*see* IS 1260 (Part 1)].

5.2.4 *BIS Certification Marking*

The container may also be marked with the Standard Mark.

5.2.4.1 The use of Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act,* 2018 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 QUALITY ASSUARANCE

6.1 Automotive diesel fuel quality assurance is based on batch certification during production at refineries by one set of test results. It is essential that refineries ensure batches are homogenous so that test results are representative of the product supplied.

6.2 At point of manufacture, the refinery shall issue a Certificate of Quality to certify that the batch of fuel complies with all of the requirements of this standard.

6.3 To certify compliance with Table 1 limits, representative samples shall be drawn using appropriate procedures such as those outlined in IS 1447 (Part 1). Each homogeneous batch of the finished product released from manufacturing point shall be tested against the requirements of Table 1. Results shall be reported on the appropriate batch certificate of quality. This requirement is not satisfied by averaging on-line analysis results.

6.4 The minimum requirements for information to be shown on the fuel's batch test certificate of quality at point of manufacture are as under:

a) Specification name, issue and any amendment number;
b) Name and address of testing laboratory;
c) Batch number or unique identifier;
d) Properties tested including specification limit, test method and result of test;
e) Identification of the signatory certifying the report; and
f) Date of certification

Table 1 Requirement for Automotive Diesel Fuel
(*Clauses* 3.1.4 and 3.2)

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to Parts of IS 1448 /ASTM/EN/ISO/Annex**
(1)	(2)	(3)	(4)
i)	Appearance	Clear, bright and free from sediments, suspended matter and undissolved water at normal ambient fuel temperature	Visual
ii)	Strong acid number, mg of KOH/g	Nil	(Part 188)/ ASTM D974[9)]
iii)	Total Acid Number (TAN), mg of KOH/g, *Max*	0.20	(Part 2)[9)]
iv)	Ash, percent by mass, *Max*	0.01	(Part 4/Sec 1)[9)]
v)	Carbon residue (Ramsbottom or micro) on 10 percent residue[1)], percent by mass, *Max*	0.30	(Part 8)[9)]/(Part 189)
vi)	Cetane number, *Min*	51[2)]	(Part 9)[9)]
vii)	Cetane index, *Min*	46[2)]	(Part 174)[9]
viii)	Pour point[3)], °C, *Max*: a)winter b)summer	 3 15	(Part 10/Sec 2) [9]
ix)	Copper strip corrosion for 3 h at	Not worse than No. 1	(Part 15)[9)]
x)	Distillation, 95 percent *v/v*, recovery, *Max*	360	(Part 18)[9)]/ISO 3405
xi)	Flash point, Abel[4)], °C, *Min*	35	(Part 20)[9)]

xii)	Kinematic viscosity, cSt, at 40°C	2.0 to 4.5	(Part 25/Sec 1)[9)]/(Part 186)/ ASTM D445
xiii)	Total contamination, mg/kg, *Max*	24	EN 12662[9)]
xiv)	Density at 15°C, kg/m^3	810–845[5)]	(Part 16)[9)]/(Part 32)/ (Part 167)
xv)	Total sulphur, mg/kg, *Max*	10	(Part 160)[9]/(Part 34)/ (Part 161)
xvi)	Water content, mg/kg, *Max*	200	(Part 182)[9)]
xvii)	Cold Filter Plugging Point (CFPP), ^{o}C, *Max*: a) Winter b) summer	 6 18	(Part 110)[9)]
xviii)	a) Oxidation stability[6)], g/m^3, *Max* b) Oxidation stability by Rancidity meter[7)], hours, *Min*	25 20	(Part 154)[9)] EN 15751[9)]
xix)	Polycyclic Aromatic Hydrocarbon (PAH), percent by mass, *Max*	8	EN 12916[9)]
xx)	Lubricity, wear scar diameter (wsd) at 60^{o}C, microns, *Max*	460	(Part 149)[9)]
xxi)	FAME content[8)], percent *v/v*, *Max*	7.0	Annex A[9)]

NOTES

1 This limit is applicable prior to addition of ignition improvers, if used. In case a value exceeding the limit is obtained on finished fuels in the market, ASTM D 4046/ISO 13759 shall be used to establish the presence of nitrate containing compound. In such case the present limit for carbon residue cannot be applied. However, the use of ignition improver does not exempt the manufacturer from meeting this requirement prior to the addition of additives.

2 Cetane number and Cetane index relaxation and time frame, if any, for fuel processed from Assam Crude, may be guided by the notifications issued by Government of India, from time to time.

3 Winter shall be the period from November to February (both months inclusive) and rest of the months of the year shall be called as summer.

4 Whenever Abel flash point exceeds 66°C by IS 1448 (Part 20)/ISO 3679/IP 170/IP 523, PMCC flash point by IS 1448 (Part 21)[9)] is to be used.

5 Density range relaxation and time frame, if any, for fuel processed from Assam Crude, may be guided by the notifications issued by Government of India, from time to time.

6 This test shall be carried out only at the refinery or manufacturer's end.

7 This test is applicable for diesel fuel having FAME content of above 2 percent *v/v*.

8 Requires if blended with Bio-Diesel. Bio-diesel shall conform to IS 15607.

9 In case of dispute, this test shall be the referee test method.

10 No external addition of chlorine based materials and metallic additives are allowed.

11 All test methods referred to in this standard include a precision statement. The interpretation of results based on test method/ precision shall be used whenever applicable. In case of dispute the procedure described in ISO 4259 shall be used.

12 Alternate methods to the characteristic given in Table 1 are provided in Annex B

ANNEX A

[*Table* 1, *Sl No.* (xxi)]

ESTIMATION OF BIO-DIESEL CONTENT IN BLENDS OF DIESEL AND BIO-DIESEL (FAME) BY FTIR SPECTROSCOPY TECHNIQUE

(Adopted From IOCM 156/2003)

A-1 SCOPE

The method describes the methodology for the estimation of bio-diesel in diesel by using infrared spectroscopy and estimation of oxygen content in bio-diesel.

A-2 SUMMARY OF THE METHOD

The IR spectra of the samples are recorded in a fixed path length cell (0.05 mm) and absorbance area is measured in the region 1 766-1 726 cm^{-1} which is then compared with calibration curve developed usingblends of known concentrations. The amount of bio-diesel in diesel is then calculated using the calibration equation. From the bio-diesel content, the amount of oxygen content is calculated.

A-3 SIGNIFICANCE AND USE

The method can be used for quick quality checks on bio-diesel content estimation. It has specific use in the blends of diesel and bio-diesel being used commercially. The method has been developed on six bio-diesel samples (*karanjia, soyabean, Jatropa, ricehran* and *palm* oil). Since the absorptivilty of all the bio-diesel is found to be almost same (205-217) the method is independent of the nature of the bio-diesel.

A-4 APPARATUS

A-4.1 *Instrument* — Infrared spectrophotometer covering the full range of 4 000-400 cm^{-1} with linear absorbance versus linear wave number recording, with good resolution is required. Ordinate repeatability and accuracy of the instrument should be better than 1 percent of full scale. The instrument should be in a position to calculate the area under the peaks.

A-4.2 *Cells* — Fixed path length cells with KBr windows and PTFE stoppers, having a path length of approximately 0.05 mm.

A-4.3 *Syringe* — 1 ml syringe with luer fitting.

A-5 CHEMICALS AND REAGENTS

A-5.1 *Cyclohexane* — Spectroscopic grade.

A-5.2 *Chloroform* — Spectroscopic grade.

A-5.3 *Bio-diesel Samples*

A-5.4 *Commercial Diesel*

A-5.5 *Benzene* — Spectroscopic grade.

A-6 PROCEDURE

One can develop the calibration equation using known blends of bio-diesel samples as reference as per the procedure given below and use the generated calibration equation for the estimation of bio-diesel content in unknown samples. Alternately, one can use the calibration equation provided for the estimation of bio-diesel content directly from the IR spectra of the unknown bio-diesel samples.

A-6.1 Reference Standards

Prepare standard blends of bio-diesel in a commercial diesel sample in the range of 1-20 percent by weight. Accurately pipette the bio-diesel into 10 ml volumetric flask. Measure the weight of the bio-diesel taken. Make up the volume with diesel and weigh again to calculate the weight percent of the blends.

A-6.2 Determination of Cell Path Length

A-6.2.1 Fill the IR cell with spectroscopic grade benzene and record the infrared spectrum over the whole range (4 000-400 cm^{-1}).

A-6.2.2 Measure the absorbance at 1 960 cm^{-1} for cells having path length less than 0.1 mm.

A-6.2.3 Cell thickness, mm = 0.1 × absorbance.

A-6.2.4 Calculate the cell path length correction factor to make the path length 0.05 mm.

A-6.3 Calibration Equation

A-6.3.1 Record the IR spectra of the known blends in MID-IR region filling the cell using the syringe and taking care that there are no entrapped air bubbles. See that the exterior of the cell does not become contaminated. Fix the PTFE stoppers to the inlet and outlet of the cell.

A-6.3.2 Measure the area under the curve in the region I, 766-1 726 cm^{-1} (in 0.05 mm cell path) and plot these values (in the X-axis) against the known concentrations of bio-diesel in diesel (in the Y-axis) to obtain the calibration curve and the equation.

A-6.4 Record the IR spectra of the diesel sample with unknown concentration of bio-diesel in diesel in the similar manner. Measure the area under the curve in the region I 766-1 726 cm^{-1} (in 0.05 mm path length).

A-6.5 From the calibration curve, determine the concentration of the bio-diesel in unknown sample by using the developed calibration (regression) equation.

A-6.6 Alternately, the combined calibration equation obtained for different bio-diesel samples (from *Palm Oil, Jatropa Oil* and *Sunflower Oil) is* given below:

$$Y = 1.0182 \times X - 0.4065$$

where

Y = concentration of unknown bio-diesel, in volume percent; and

X = area under the curve between the region 1 766-1 726 cm^{-1} in 0.05 mm cell path length.

A-6.6.1 Record the IR spectrum of unknown bio-diesel samples using pre-calibrated fixed path IR cell in 1 766-1 726 cm^{-1} region and measure the area of the band in the region as described earlier.

A-6.6.2 Determine the concentration of the bio-diesel in unknown sample employing the above equation.

A-7 Determination of percent oxygen content in bio-diesel:

Percent oxygen = $Y \times 10.70/100$ in bio-diesel content

where

Y = concentration of bio-diesel estimated.

A-8 PRECISION

The precision of the method is estimated employing the standard statistical techniques. Samples are prepared in the concentration range of 1-15 percent bio-diesel in diesel. The samples are analyzed by two operators in duplicate. ANOVA analysis is carried out on the results obtained and the precision statement of repeatability and reproducibility values are found to be 0.8 and 1.8, respectively. One can develop the precision statements up to 20 percent bio-diesel concentration also employing suitable standards and IR cells.

A-8.1 Repeatability

a) 0.0 to 5.0 percent — 0.4
b) 5.1 to 15.0 percent — 0.8

ANNEX B

(*Foreward*)

BIBLIOGRAPHICAL REFERENCES

International/Other Standard No.	*Title*
ASTM D445-21	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
ASTM D974-21	Standard Test Method for Acid and Base Number by Color-Indicator Titration
EN 12662 : 2014	Liquid petroleum products — Determination of total contamination in middle distillates, diesel fuels and fatty acid methyl esters
EN 12916 : 2014	Petroleum products — Determination of aromatic hydrocarbon types in middle distillates — High performance liquid chromatography method with refractive index detection
EN 15751 : 2014	Automotive fuels — Fatty acid methyl ester (FAME) fuel and blends with diesel fuel — Determination of oxidation stability by accelerated oxidation method
ISO 3405 : 2019	Petroleum and related products from natural or synthetic sources — Determination of distillation characteristics at atmospheric pressure

ANNEX C

(*Table* 1, *Note* 12)

ALTERNATE METHODS

Characteristic	*Alternate Method of Tests*
Strong Acid Number	IP 139
Total Acid Number (TAN), mg of KOH/g	ASTM D664, IP 139
Ash, percent by mass	ASTM D482 , IP 4
Carbon residue (Ramsbottom or micro) on 10 percent residue), percent by mass	ASTM D524, ASTM D4530, IP 14
Cetane number	ASTM D613
Cetane index	ASTM D4737, IP 380
Pour point	ASTM D5949, ASTM D5950, ASTM D5985, ASTM D97, ASTM D7346, IP 15
Copper strip corrosion for 3 h at 50°C	ASTM D130, IP 154
Distillation, 95 percent *v/v* recovery,	ASTM D86, ASTM D7345[1)], IP 123, ISO 3405
Flash point, Abel , °C	ISO 3679, IP170, IP523
Kinematic viscosity, cSt, at 40°C	ASTM D7042, IP 71
Total contamination, mg/kg	IP 440
Density at 15°C, kg/m^3	ISO 12185, ASTM D 4052, ASTM D1298, IP 160
Total sulphur, mg/kg	ISO 13032, ISO 20884, ASTM D5453, ASTM D2622, ASTM D7220

Water content, mg/kg	ASTM D6304
Cold Filter Plugging Point (CFPP)	ASTM D6371, IP 309
Oxidation stability, g/m^3,	ASTM D2274, IP 388
Polycyclic Aromatic Hydrocarbon, (PAH), percent by mass	ASTM D6591, IP 391
Lubricity, wear scar diameter (wsd) at 60°C, microns,	ASTM D6079
FAME content , percent *v/v*	ASTM D7371, EN14078

NOTES

1 Results from test method ASTM D7345 shall be corrected for relative bias as described in Test Method ASTM D7345.

2 Results from ASTM D7042 shall be converted to bias-corrected kinematic viscosity results by the application of correction as described in the method ASTM D7042