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(सातवा पुनरीक्षण)

Draft Indian Standard
AUTOMOTIVE DIESEL FUEL — SPECIFICATION

(Seventh Revision of IS 1460)

(ICS 75.160.20)

Petroleum and their Related Products of Synthesis or
Biological Origin Sectional Committee, PCD 03

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FOREWORD

(Formal clause will be added later)

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

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. Nothing in this standard shall, however, preclude observance of national 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 is applicable to automotive diesel fuel 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 also included in the second revision to ensure the stability of the fuel.

The third revision of the standard was carried out in 1995, which incorporated three amendments published in 1980, 1981, and 1985. The following major changes were made in the third revision:

- a) Requirement of cetane number modified from 42, *Min* to 45, *Min*;

- b) Kinematic viscosity limit specified as 1.8 to 5.0 cSt;
- c) UOP 413 prescribed as test procedure for total sediments;
- d) Quartz tube method for sulphur determination removed;
- e) Limit prescribed for cold filter plugging point test; and
- f) Pour point prescribed separately for winter and summer grades.

The fourth revision of the standard was carried out in 2000, which incorporated four amendments published in 1997, 1999, and 2000. The following major changes were made in the fourth revision:

- 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/m³;
- h) Total sulphur reduced to 0.25 percent, maximum to meet the requirement given in the notification issued by MOEF; and
- i) Cold Filter Plugging Point (CFPP) tightened upto 6 °C, *Max* for winter and 18 °C, *Max* for summer.

The 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. 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/m³;
- e) Total sulphur reduced to 350 ppm maximum for BS III and 50 ppm maximum for BS IV.

The sixth revision of the standard was carried out in 2017, which incorporated two amendments published in 2007 and 2010. The following major changes were made in the sixth revision:

- a) Requirements for Bharat stage II (BS II) and Bharat Stage III (BS III) automotive diesel fuel were deleted;
- b) Requirements for distillation recovery at 350 °C, distillation recovery at 370 °C and sediments were deleted; and
- c) 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.

In this seventh revision, two amendments published in 2018 and 2021 have been incorporated. Additionally, the following major changes have been made:

- a) Requirement for Bharat stage IV (BS IV) automotive diesel fuel have been deleted;
- b) Requirement of acidity, inorganic deleted;
- c) Requirement of lubricity corrected wear scar diameter (wsd 1.4) changed to wear scar diameter (wsd); and
- d) Limit for chlorinated compounds introduced;

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.

For some requirements in Table 1, Indian Standards do not exist for the test methods, hence EN standards are referred. Once Indian Standards are formulated for these tests, the references will be modified accordingly. Also, alternate test methods are provided below for few characteristics and in case of dispute, the referee methods prescribed in Table 1 shall be followed.

<i>Characteristic</i>	<i>Alternate Method of Tests</i>
Total Acid Number (TAN)	ASTM D664, IP 139
Ash	ASTM D482, IP 4
Carbon residue	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 D73451, IP 123, IP 596, EN 17306
Flash point, Abel	ISO 3679, IP 170, IP 523
PMCC flash point	ASTM D93
Kinematic viscosity	ASTM D445, ASTM D7042, IP 71
Total contamination	IP 440
Density at 15°C	ISO 12185, ASTM D 4052, ASTM D1298, IP 160
Total sulphur	ISO 13032, ISO 20884, ASTM D5453, ASTM D2622, ASTM D7220
Water content	ASTM D6304
Cold Filter Plugging Point (CFPP)	ASTM D6371, IP 309
Oxidation stability	ASTM D2274, IP 388
Polycyclic Aromatic Hydrocarbon (PAH)	ASTM D6591, IP 391
Lubricity, wear scar diameter (wsd) at 60 °C	ASTM D6079
FAME content	ASTM D7371, EN14078

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 and methods of sampling and test 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 of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS / International Standards</i>	<i>Title</i>
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling goods Part 1 Dangerous goods (<i>first revision</i>)
IS 1447 (Part 1) : 2021	Methods of sampling of petroleum and its products Part 1 Manual sampling (<i>second revision</i>)
IS 1448	Methods of test for petroleum and its products
(Part 2) : 2007 / ISO 6619 :1988	Petroleum products and lubricants — Neutralization number - potentiometric titration method (<i>second revision</i>)
(Part 4/Sec 1) : 2021	Determination of ash (<i>fourth revision</i>)
(Part 8) : 2012 / ISO 4262 : 1993	Determination of carbon residue — Ramsbottom method (<i>second revision</i>)
(Part 9) : 2023 / ISO 5165 : 2020	Determination of the ignition quality of diesel fuels — Cetane engine method (<i>third revision</i>)
(Part 10/Sec 2) : 2021 / ISO 3016 : 2019	Petroleum and related products from natural or synthetic sources Section 2 Determination of pour point (<i>third revision</i>)
(Part 15) : 2004 / ISO 2160 : 1998	Petroleum products — Corrosiveness to copper strip test (<i>third revision</i>)
(Part 16) : 2014 / ISO 3675 : 1998	Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method (<i>fourth revision</i>)
(Part 18) : 2020	Distillation of petroleum products (<i>third revision</i>)
(Part 20) : 2019 / ISO 13736 : 2013	Determination of flash point — Abel Closed-Cup Method (<i>third revision</i>)
(Part 21) : 2019 / ISO 2719 : 2016	Determination of flash point - Pensky-Martens closed cup method (<i>fourth revision</i>)
(Part 25/Sec 1) : 2018 / ISO 3104 : 1994	Transparent and opaque liquids Section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (<i>second revision</i>)
(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 (<i>third revision</i>)
(Part 34) : 1979	Determination of sulphur in petroleum products (lamp method) (<i>second revision</i>)
(Part 110) : 2023	Cold filter plugging point of distillate fuels (<i>first revision</i>)
(Part 149) : 2020 / ISO 12156-1 : 2018	Diesel fuel — Assessment of lubricity using the high-frequency reciprocating rig (HFRR) — Test method (<i>second revision</i>)
(Part 154) : 2012 / ISO 12205 : 1995	Determination of oxidation stability of middle distillate fuels

(Part 159) : 2018 / ISO 20884 : 2011	Determination of sulphur content of automotive fuels - Wavelength - Dispersive x - Ray fluorescence spectrometry
(Part 160) : 2017 / ISO 20846 : 2011	Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method
(Part 161) : 2017 / ISO 13032 : 2012	Determination of low concentration of sulfur in automotive fuels — Energy dispersive X-ray fluorescence spectrometric method
(Part 167) : 2018 / ISO 12185 : 1996	Determination of density — Oscillating U-tube method
(Part 174) : 2020 / ISO 4264 : 2018	Petroleum products — Calculation of cetane index of middle-distillate fuels by the four variable equation
(Part 182) : 2020 / ISO 12937 : 2000	Petroleum products — Determination of water —Coulometric Karl Fischer titration method
(Part 186) : 2021 / ISO 23581 : 2020	Petroleum products and related products — Determination of kinematic viscosity — Method by Stabinger type viscometer
(Part 189) : 2021 / ISO 10370 : 2014	Determination of carbon residue — Micro method
IS 15607 : 2022	Biodiesel B-100 - Fatty Acid Methyl Esters FAME Specification (<i>second revision</i>)
IS 17315 (Part 2) : 2019 / ISO 4259-2 : 2017	Petroleum and Related Products - Precision of Measurement Methods and Results Part 2 Interpretation and Application of Precision Data in Relation to Methods of Test
ISO 3405 : 2019	Petroleum and related products from natural or synthetic sources — Determination of distillation characteristics at atmospheric pressure
EN 12662 : 2014	Liquid petroleum products. Determination of total contamination in middle distillates, diesel fuels and fatty acid methyl esters
EN 15751 : 2014	Automotive fuels. Fatty acid methyl ester (FAME) fuel and blends with diesel fuel. Determination of oxidation stability by accelerated oxidation method
EN 16091 : 2022	Liquid petroleum products. Middle distillates and fatty acid methyl ester (FAME) fuels and blend. Determination of oxidation stability by rapid small scale oxidation test (RSSOT)
EN 12916 : 2019	Petroleum products. Determination of aromatic hydrocarbon types in middle distillates. High performance liquid chromatography method with refractive index detection

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. (xxii)] by authorized agents. Stabilizing

agents, as required, shall be incorporated. Percentage of bio-diesel blended shall be ensured and declared by suppliers using a certification process.

3.1.5 The use of dyes and/or markers as approved by competent authority or the Ministry of Petroleum and Natural Gas 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 or alternate test methods (*see* Foreword).

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 of the material;
- b) Indication of the source of manufacturer, initials or trade-mark, if any;
- c) Volume of the material, 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 labelling 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 the standard, representative samples shall be drawn using appropriate procedures specified in IS 1447 (Part 1). Each homogeneous batch of the finished product released from manufacturing point shall be tested against the requirements prescribed in the standard. Results shall be reported on the appropriate batch certificate of quality (COQ). 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 COQ at point of manufacture are as under:

- a) IS number and year along with amendment, if any;
- b) Name and address of testing laboratory;
- c) Batch number or unique identifier;
- d) Properties tested including specified 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, 3.2 and Foreword)

SI No.	Characteristic	Requirement	Method of Test, Ref to Parts of IS 1448 / ISO / EN / Annex
(1)	(2)	(3)	(4)
i)	Appearance	Clear, bright and free from sediments, suspended matter and undissolved water at normal ambient fuel temperature	Visual
iii)	Total Acid Number (TAN), mg of KOH/g, <i>Max</i>	0.20	(Part 2) ⁸
iv)	Ash, percent by mass, <i>Max</i>	0.01	(Part 4/Sec 1) ⁸
v)	Carbon residue, on 10 percent residue ¹ , percent by mass, <i>Max</i>	0.30	(Part 8) ⁸ / (Part 189)
vi)	Cetane number, <i>Min</i>	51 ²	(Part 9) ⁸
vii)	Cetane index, <i>Min</i>	46 ²	(Part 174) ⁸
viii)	Pour point ³ , °C, <i>Max</i> : a) Winter b) Summer	3 15	(Part 10/Sec 2) ⁸
ix)	Copper strip corrosion for 3 h at 50 °C, <i>Max</i>	1	(Part 15) ⁸
x)	Distillation, 95 percent v/v recovery, °C, <i>Max</i>	360	(Part 18) ⁸ / ISO 3405
xi)	Flash point, Abel ⁴ , °C, <i>Min</i>	35	(Part 20) ⁸
xii)	Kinematic viscosity, cSt, at 40 °C	2.0 to 4.5	(Part 25/Sec 1) ⁸ / (Part 186)
xiii)	Total contamination, mg/kg, <i>Max</i>	24	EN 12662 ⁸
xiv)	Density at 15 °C, kg/m ³	810 to 845 ⁵	(Part 16) ⁸ / (Part 32) / (Part 167)

xv)	Total sulphur, mg/kg, <i>Max</i>	10	(Part 160) ⁸ / (Part 34) / (Part 161) / (Part 159)
xvi)	Water content, mg/kg, <i>Max</i>	200	(Part 182) ⁸
xvii)	Cold Filter Plugging Point (CFPP) ³ , °C, <i>Max</i> :		(Part 110) ⁸
	a) Winter	6	
	b) Summer	18	
xviii)	Oxidation stability ⁶ , g/m ³ , <i>Max</i>	25	(Part 154) ⁸
xix)	Oxidation stability (for diesel fuel having FAME content of above 2 percent v/v), hours, <i>Min</i>	20	EN 15751 ⁸ / EN 16091
xx)	Polycyclic Aromatic Hydrocarbon (PAH), percent by mass, <i>Max</i>	8	EN 12916 ⁸
xxi)	Lubricity, wear scar diameter (wsd) at 60 °C, µm, <i>Max</i>	460	(Part 149) ⁸
xxii)	FAME content ⁷ , percent v/v, <i>Max</i>	7.0	Annex A ⁸

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 D4046 / 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 70 °C by IS 1448 (Part 20) / ISO 3679 / IP 170 / IP 523, PMCC flash point by IS 1448 (Part 21)⁸ 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 Required if blended with biodiesel. Biodiesel shall conform to IS 15607.
- 8 In case of dispute, this method shall be the referee test method.
- 9 No external addition of chlorine based materials and metallic additives are allowed.
- 10 Fuel should be free from chloride or chlorinated materials and metallic additives. In case of any dispute raised by consumer on chloride content in the fuel (above 5 mg/kg), representative fuel sample collected at the point of delivery or point of dispensing can be tested by Wavelength Dispersive X-Ray Fluorescence (WDXRF) or Energy Dispersive X-Ray Fluorescence (EDXRF) or micro coulometric technique by a competent testing laboratory and decided
- 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 IS 17315 (Part 2) shall be used.

ANNEX A
[Table 1, Sl No. (xxii)]

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 sample is recorded in a fixed path length cell (0.05 mm) and absorbance area is measured in the region 1 766 to 1 726 cm^{-1} which is then compared with calibration curve developed using blends 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 (*karanja*, *soyabean*, *Jatropha*, *ricehran* and *palm* oil). Since the absorptivity of all the bio-diesel samples is found to be almost same (205 to 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 to 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

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, 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 to 20 percent by weight. Accurately pipette the bio-diesel into 10 ml volumetric flask and weigh it. 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 (4000 to 400 cm^{-1}).

A-6.2.2 Measure the absorbance at 1960 cm^{-1} for cells having path length less than 0.1 mm.

A-6.2.3 Cell thickness, mm = 0.1 \times 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 1 766 to 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 1 766 to 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 in the region 1 766 to 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 to 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 (Foreword)

BIBLIOGRAPHICAL REFERENCES

<i>International/Other Standard No.</i>	<i>Title</i>
ASTM D4046-14	Standard Test Method for Alkyl Nitrate in Diesel Fuels by Spectrophotometry
ISO 13759 : 1996	Petroleum products — Determination of alkyl nitrate in diesel fuels — Spectrometric method