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Indian Standard
Automotive fuels — Paraffinic diesel fuel from synthesis or hydrotreatment — Specification

ICS 43.060.01;75.160.20

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FOREWORD

(Formal clause will be added later)

Synthetic or hydrotreated paraffinic diesel fuel is a complicated combination of hydrocarbons that changes depending on the feedstock and production technique. By synthesis techniques, it is difficult to determine the precise content of paraffinic diesel fuel. As a result, this standard has grown mainly as a performance specification rather than a compositional definition. As a result, the standard restricts vehicle fuels to those derived from traditional sources.

This draft standard defines Paraffinic diesel fuel specification derived from synthesis gas or hydrotreated oils, bio-oils, or –fats like cyclo-paraffins, aromatics, and iso-paraffins. It can be used in all diesel fuel vehicles/engines. This process involves converting fatty acids in triglycerides into linear and iso-alkanes (paraffinic diesel). The reactions involved in the vegetable oil conversion to hydrocarbons fuel are hydrodeoxygenation, decarbonylation, decarboxylation, isomerization, and hydrocracking. Hydrotreated vegetable oil diesel, also known as green diesel or paraffinic diesel, may be produced in a single step/multiple processes using a catalyst. The fuel derived from hydrotreatment of oils, bio-oils, or -fats are mostly paraffinic, and it may not meet the density requirement of conventional crude-based diesel defined in IS 1460. Also, due to more paraffin, the cetane number for paraffinic diesel is higher than that of the crude diesel.

Based on the production processes reported so far around the globe, the paraffinic diesel fuel produced may be classified into two broad categories. (a) Paraffinic Diesel fuel with (Class A) improved ignition quality compared to automotive diesel fuel meeting IS 1460 specifications (b) Paraffinic Diesel fuel with average cetane number (Class B). Paraffinic diesel fuel blending with fatty acid methyl ester (FAME) is also covered in this document.

There are specific test methods for paraffinic diesel fuel, which need to be checked for applicability as required. The product is now no longer limited to captive fleet usage, but the scope needs to check the product use with the vehicle manufacturer. This document is based on current knowledge at the time of publishing but may require review based on different experiences using paraffinic diesel fuel or when the specification for either regular automotive diesel fuel or FAME has been revised.

A limit was stipulated for the cold filter plugging point test to ensure a smooth operation at low ambient temperatures. Because of improved ignition quality compared to automotive diesel fuel, Paraffinic diesel fuel can also be used as a blending component in automotive diesel fuel defined in IS 1460. In that case, the fuel blends shall meet the requirements of the automotive diesel fuel standard, IS 1460.

The following parameters that differ from its BS-VI performance of IS 1460 are mentioned below in this specification.

a) Cetane number for high cetane paraffinic diesel (class A) has been increased to 70 from 51 compared to the BS-VI limit. However, for class B (low cetane paraffinic diesel), the limit is unchanged.
b) Cetane index is removed as insufficient data is available for the cetane index to limit the same.
c) Pout point has been removed as the cold flow properties have been defined by CFPP and cloud point for different fuel grades.
d) Distillation, the limit for 95 percent, *maximum* volume recovered at 360°C.
e) Flashpoint modified from 35 to 55°C, *min*;
f) Density range has been modified from 765-800 to 780-810 kg/m^3;
g) Cold Filter Plugging Point (CFPP) tightened up to 6°C, *Max* for winter and 18°C, *Max* for summer for normal climates. More stringent limits (5 grades A-E) has been set for the severe climate conditions (-44, -38, -32, -36 and -20 °C)
h)Manganese content (mg/L max.) has a limit of 2.0

Increased demand for cleaner fuel due to environmental concerns and depleting petroleum reserves in the world, coupled with the deteriorating quality of crude oil, has led to a surge of research for renewable and clean fuel sources. Oils originating from vegetables and animals cannot be used directly used in the engine due to the problems inherent with these oils, such as higher viscosity and oxygen content and poor atomization and lubricity.

Green diesel/paraffinic diesel is entirely compatible with the conventional blend of petroleum-derived diesel fuels, giving refiners a substantial advantage. The technique for making paraffinic diesel is kind to the environment and works well with current petroleum refineries. It is virtually indistinguishable from regular diesel fuel and may be used as a direct replacement or a useful blendstock to improve the quality of existing diesel pools. Paraffinic diesel may be utilized in today's tanks, pipelines, trucks, pumps, and vehicles without modification since it is chemically identical to regular diesel fuel.

Indigenously, CSIR-IIP uses different tree-borne oil/algae oil to produce paraffinic hydrotreated fuel in a single reactor. The product obtained during the process is separated and distilled into various fractions. The catalyst and process conditions are optimized in such a way that maximum diesel is obtained. The green diesel/paraffinic diesel obtained by this process has a high cetane number (>75). The pilot plant at CSIR-IIP has 200 kg/day feed processing (120-150 kg of paraffinic diesel).

Accordingly, paraffinic diesel fuel requirements for vehicles meeting Class A or Class B norms are furnished in Table 1. The date and area for implementing these specifications are as per the Competent Authority's notification from time to time. However, nothing in this standard shall preclude observance of the regulations, which may be more restrictive.

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

The draft is prepared in line with the European standard "Automotive fuels - Paraffinic diesel fuel from synthesis or hydrotreatment — Requirements and test methods, BS EN 15940:2016+A1:2018". The draft has been prepared considering the requirement of renewable diesel fuel and its supply and demand pattern of diesel fuel other than petroleum origin.

There are references to other international standards such as ISO, ASTM, IP, EN, and others because there is no Indian standard for them. The references will be updated after the Indian Standards have been finalized.

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

The final number, observed or computed, indicating the outcome of a test or analysis, should be rounded off following IS 2: 1960 'Rules for rounding off numerical values (updated)' to determine whether a specific requirement of this standard has been met. The number of significant places in the rounded-off value should be the same as the number of significant places in the given standard value.

1. SCOPE

This draft standard prescribes the requirements, sampling procedure, and test methods for paraffinic diesel fuel and contains up to 7.0 % (V/V) fatty acid methyl ester (FAME). Paraffinic diesel fuel originates from synthesis or hydrotreatment processes. It is intended for use in diesel engine cars and stationary diesel engines powered by automotive diesel fuel and vehicles compatible with paraffinic diesel fuel. It distinguishes between two types of paraffinic diesel fuel: high cetane and regular cetane.

NOTES:

1) Paraffinic automotive diesel fuel may require a validation process to ensure fuel compatibility with the vehicle for general diesel engine warranties, which may still require some older engines (also see Foreword). The vehicle manufacturer needs to be consulted before use.
2) For this document, the terms "% (m/m)" and "% (V/V)" are used to represent the mass fraction and the volume fraction, respectively.

WARNING: 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 before 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 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
1447 (Part 1) :2021	Petroleum and its products ─ Methods of sampling: 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: 2008 /ISO 6245:2001	Petroleum products – Determination of ash (*third revision*)
Part 8: 2012 /ISO 4262:1993	Carbon residue by 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: 2013 /ISO 3016:1994	Cloud point and pour point- Section 2 Determination of pour point (*second revision*)
Part 15: 2004/ ISO 2160: 1998	Petroleum products ─ 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	Determination of flash point - Pensky - Martens closed cup method (*fourth revision*)
Part 25/Sec 1 :2018/ISO 3105	Transparent and opaque liquids section 1 determination of kinematic viscosity and calculation of dynamic viscosity (*second revision*)
Part 32:2019/ISO 3838	Capillary stoppered pyknometer and graded bicapillary pyknometer techniques for determining density or relative density of crude petroleum and liquid or solid petroleum products (*third revision*)
Part 34:1979	Determination of sulfur in petroleum products (lamp method) (*second revision*)
Part 110:1981	Cold filter plugging point of distillate fuels
Part 149:2020/ ISO 12516-1:2018	Diesel fuel lubricity assessment utilizing the high-frequency reciprocating rig (HFRR) Part 1: Test method
Part 153: 2012/ISO 20847:2004	Determination of sulfur content of automotive fuels – Energy-dispersive X-ray fluorescence spectrometry
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	Determination of Sulphur Content of Automotive Fuels — Ultraviolet Fluorescence Method
Part 161:2017/ ISO 13032	Determination of low concentration of sulfur in automotive fuels — Energy - dispersive x-Ray

	fluorescence spectrometric method
Part 167:2018/ ISO 12185	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
17315 (Part 1) :2019/ ISO 4259-1	Petroleum and Related Products — Precision of Measurement Methods and Results Part 1 Interpretation and Application of Precision data concerning Methods of Test
17315 (Part 2) :2019/ ISO 4259-2	Petroleum and Related Products — Precision of Measurement Methods and Results Part 2 Interpretation and Application of Precision data concerning Methods of Test
ISO 3405:2011	Determination of distillation characteristics at atmospheric pressure
ISO 3679:2015	Determination of flash no-flash and flash point ─ Rapid equilibrium closed cup method
ISO 3924:2016	Determination of boiling range distribution - Gas chromatography method
ISO 6618:1997	Petroleum products and lubricants ─ Determination of acid or base number ─ Colour-indicator titration method
ISO 10370:2014	Determination of carbon residue – Micro method
ISO 12662:2014	Determination of total contamination in middle distillates, diesel fuels, and fatty acid methyl esters
ISO 12916- 1	Determination of aromatic hydrocarbon types in the middle distillates — High-performance liquid chromatography method with refractive index detection.
ISO 13759:1996	Determination of alkyl nitrate in diesel fuels – Spectrometric method
ISO 14078:2014	Determination of fatty acid methyl ester (FAME) content in middle distillates – Infrared spectrometry method
ISO 14214:2012+A1:2014	Fatty acid methyl esters (FAME) for use in diesel engines and heating applications - Requirements and test methods
ISO 15195:2014	Determination of ignition delay and derived cetane number (DCN) of middle distillate fuels by combustion in a constant volume chamber
ISO 15751:2014	Fatty acid methyl ester (FAME) fuel and blends with diesel fuel -Determination of oxidation stability by accelerated oxidation method
ISO 16576:2014	Determination of manganese and iron content in diesel - Inductively coupled plasma optical emission spectrometry (ICP OES) method

ISO 16329:2013	Determination of cold filter plugging point – Linear cooling bath method
ISO 16906	Determination of the ignition quality of diesel fuels — BASF engine method

3 REQUIREMENTS

3.1 General

3.1.1 At typical ambient fuel temperature, the material must be clear, brilliant, and devoid of sediments, suspended debris, and undissolved water.

NOTES
1. Any adulterant or pollutant in paraffinic diesel fuel that may make it unfit for use in diesel engines must be removed.
2. Any intentional addition of non-paraffinic material, other than FAME, additives and dyes or markers, is not allowed.

3.1.2 *Composition*

The material shall be hydrocarbon as such or combined with fatty acid methyl esters derived from synthesis or hydrotreatment of renewable feedstock. The use of fuel additives is permitted to improve 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 IS 15607 biodiesel (Fatty Acid Methyl Ester, FAME) can be mixed with paraffinic diesel fuel up to 7% (v/v) [*see* Table I, SI No. (xxi)]. Stabilizing agents, as required, shall be incorporated.

3.1.5 The use of dyes or markers is permitted.

3.1.6 *Methylcyclopentadienyl Manganese Tricarbonyl (MMT*)

When methylcyclopentadienyl manganese tricarbonyl (MMT) is used, specific labeling is required. The presence of the MMT is limited via a manganese content limit as in Table 1.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the appropriate methods specified in col 5 of Table 1.

4. SAMPLING

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

NOTE - Because some of the test methods referred to are sensitive, special attention must be made to comply with any sampling container instructions provided in the test method standard.

5 PACKING AND MARKING

5.1 Packing

The fuel shall be packaged in appropriate containers as specified by the Petroleum and Explosives Safety Organization (PESO) from time to time.

5.2 Marking

5.2.1 The material shall be supplied following 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) If applicable, an indication of the manufacturer's source, initials, or trade-mark;
c) The content's volume, in liters;
d) Manufacturing or packaging year and month; and
e) Any other statutory requirements.

5.2.3 Each container must also be labeled with the warning label "Highly Flammable" and the matching symbol for hazardous products labeling [*see* IS 1260 (Part 1)].

5.2.4 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the BIS Act, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

5.2.5 *Pump Marking*

The dispensing pumps and nozzles used for delivering paraffinic diesel fuel shall be compatible with paraffinic diesel. Labeling shall be visible, easily legible, and displayed at any point where paraffinic diesel with metallic additives is made available to consumers. In that case, the label shall contain "Contains metallic additives." Further indication on dispensing pumps of "Not suitable for all" shall be mentioned.

6. QUALITY ASSURANCE

6.1 Batch certification during production at refineries/manufacturing units by one set of test results is used to ensure the quality of paraffinic diesel fuel. Refineries and manufacturing facilities must guarantee that batches are homogeneous for test findings to reflect the product provided.

6.2 The refinery/manufacturing unit shall provide a Certificate of Quality at the time of manufacture to confirm that the fuel batch meets all of these standard standards.

6.3 Representative samples must be drawn using suitable techniques, such as those described in IS 1460, to confirm compliance with Table 1 limitations. Each homogenous batch of the completed product issued from the production point must be tested following Table 1's criteria. The results must be documented on the relevant batch quality certificate. Averaging online analytical findings do not meet this criterion.

6.4 The following are the minimal requirements for information to appear on the fuel's batch test certificate of quality at production time.

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 the result of the test;
e) Identification of the signatory certifying the report; and
f) Date of certification.

Table 1 Requirements for Paraffinic diesel fuel from synthesis or hydrotreatment

(*Cl* 3.1.6, 3.2 & 6.3)

Sl No.	**Characteristic**	**Requirement**		**Method of Test, Ref to Part of IS 1448/ Annex/ISO/ ASTM/IP**
		Class A	**Class B**	
(1)	(2)	(3)	(4)	(5)
i)	Appearance	Clear, bright, and free from sediments, suspended matter, and undissolved water at normal ambient fuel temperature	clear, bright, and free from sediments, suspended matter, and undissolved water at normal ambient fuel temperature	Visual
ii)	Acidity, inorganic, mg of KOH/g	Nil	Nil	ISO 6618/ASTM D974[9)]/ IP 139
iii)	Acidity, total, mg of KOH/g, *Max*	0.20	0.20	Part 2 [9)]/ASTM D664/ ASTM D974 / IP 139
iv)	Ash, percent by mass, % *Max (mass)*	0.01	0.01	Part 4:Sec 1 [9)]/ASTM D 482/IP 4
v)	Carbon residue (Ramsbottom or micro) on 10 percent residue[1)], percent by mass, *Max*	0.30	0.30	Part 8 [9)] /ISO 10370/ASTM D 524/IP 14/ASTM D 4530
vi)	Cetane number, *Min*	70[2)]	51[2)]	Part 9 [9)]/ASTM D 613 DIN EN 16906

vii)	Copper strip corrosion for 3 h at 50°C	Not worse than No. 1	Not worse than No. 1	[P : 15] [9)]/ASTM D 130/IP 154
viii)	Distillation, 95 percent *v/v*, recovery, °C, *Max*	360	360	[P : 18] [9)] /ASTM D 86/ASTM D 7345/IP 123
xi)	Flash point, Abel[4)], °C, *Min*	55	55	[P : 20] [9)]/ / IP170 /IP523/ EN13736/
xii)	Kinematic viscosity, mm^2/s, at 40°C	2.0 to 4.5	2.0 to 4.5	[P : 25:Sec 1] [9)]/ ASTM D 445/ASTM D 7042/IP 71
xiii)	Total contamination, mg/kg, *Max*	24	24	EN 12662[9)]/IP 440
xiv)	Density at 15°C, kg/m^3	765-800[5]	780 – 810[5)]	[P : 16] [9)] /[P : 32] / ISO 12185/ ASTM D 4052/ ASTM D 1298/IP 160
xv)	Total sulphur, mg/kg, *Max*	10.0	10.0	ISO 13032 [9)]/ ISO 20884/ISO 20846[9)]/ASTM D 5453/ ASTM D 2622/ ASTM D 7220/[P : 34]
xvi)	Water content, mg/kg, *Max*	200	200	ISO 12937/ASTM D 6304
xvii)	Cold Filter Plugging Point (CFPP)[3)], *Max*: a) Winter b) Summer	 6°C 18°C	 6°C 18°C	[P : 110] [9)]/ASTM D 6371/IP 309
xviii)	Pour point[3)], *Max*: a) Winter **b) Summer**	 3°C **15°C**	 3°C **15°C**	[P : 10] [9)]/ASTM D 5949/ ASTM D 5950/ ASTM D 5985/ASTM D97/ASTM D7346/IP 15
xix)	a) Oxidation stability[6)], g/m^3, *Max* b) Oxidation stability by Rancidity meter[7)], hours, *Min*	25 20	25 20	[P : 154] [9)]/ASTM D 2274 / IP 388 EN 15751
xx)	Polycyclic Aromatic Hydrocarbon (PAH), percent by mass, *Max*	8	8	EN 12916[9)]/IP 391 /ASTM D 6591
xxi)	Lubricity corrected wear scar diameter (wsd 1.4) at 60°C, microns, *Max*	460	460	P 149/ ISO 12156-1/Cor 1
xxii)	FAME content[8)], %v/v, Max	7.0	7.0	Annex A[9)]/ ASTM D7371/ EN14078
xxiii)	Initial boiling point	report	report	ISO 3405
xxiv)	Manganese content mg/l (max)	2.0	2.0	ISO 16576

NOTES:

1 This limit is applicable prior to the 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 compounds. In such a case, the present limit for carbon residue cannot be applied. However, the use of an ignition improver does not exempt the manufacturer from meeting this requirement prior to additives.

2 Cetane number and Cetane index relaxation and time frame, if any, for fuel processed from Assam Crude may be guided by India's Government reports from time to time.
3 Winter shall be the period from November to February (both months inclusive), and the rest of the months of the year shall be called summer.
4 Whenever Abel flash point exceeds 66°C by IS 1448 [P 20]/ISO 3679/ IP170 /IP523, PMCC flash point by IS 1448 [P 21][9]is to be used
5 For correction of the determined value for paraffinic diesel fuel, Annex C shall be applied.
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 Bio-diesel shall conform to IS 15607.
9 In case of dispute, this test shall be the referee test method.
11 No external addition of chlorine-based materials and metallic additives are allowed.
12 A precision statement is included in all test methods mentioned in this standard. Whenever possible, the interpretation of results based on the test method/precision will be applied. In the event of a disagreement, the IS 17315 Part 1 and Part 2 shall be followed.
13 Before blending with FAME, paraffinic diesel fuel is expected to contain more than 90 % (m/m) of paraffinic hydrocarbons.
14 As no test method for determining the paraffinic hydrocarbon content is available at the time of publication of this standard, only Polycyclic Aromatic Hydrocarbon are limited in Table 1.

ANNEX A

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

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

A-1 SCOPE

The method describes the methodology for the estimation of bio-diesel in a 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 the absorbance area is measured in the region 1 766-1 726 $cm^{-1,}$ which is then compared with a 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 a 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, ricebran and palm* oil). Since the absorptivity of all the bio-diesel is found to be almost the 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 the 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 a reference to estimate the given below and use the generated calibration equation to estimate the given below use the generated calibration equation to estimate bio-diesel content in unknown samples. Alternately, one can use the calibration equation for the assessment of bio-diesel content directly from the unknown bio-diesel samples' IR spectra.

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 the MID-1R 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 an unknown concentration of bio-diesel in diesel in a similar manner. Measure the area under the curve in the region 1 766 -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 an unknown sample by using the developed calibration (regression) equation.

A-7 Alternately, the combined calibration equation obtained for different bio-diesel samples *is* given below:

$$Y = 1.018\ 2\ X — 0.406\ 5$$

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-7.1 Record the IR spectrum of unknown bio-diesel samples using pre-calibrated fixed path IR cell in 1 766-1 726 cm^{-1} regions and measure the area of the band in the region as described earlier.

A-7.2 Determine the concentration of the bio-diesel in an unknown sample employing the above equation.

A-7.3 determination of percent oxygen content in bio-diesel:

$$\text{Percent Oxygen Content} = CB \times 10.70/100 \text{ in bio-diesel}$$

where,
CB = 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 biodiesel in diesel. The samples are analyzed by two operators in duplicate. 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-9 REPEATABILITY

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