इंटरनेट मानक

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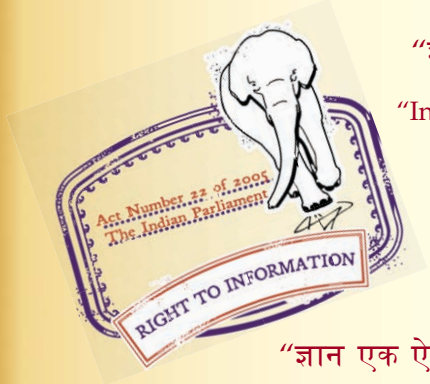
"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 1460 (2005): Automotive Diesel Fuel [PCD 3: Petroleum, Lubricants and their Related Products]



"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

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भारतीय मानक

मोटर वाहन डीजल ईंधन — विशिष्टि

(पाँचवाँ पुनरीक्षण)

Indian Standard

AUTOMOTIVE DIESEL FUEL — SPECIFICATION

(Fifth Revision)

ICS 75.180.20

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 2005

Price Group 6

Petroleum, Lubricants and Their Related Products Sectional Committee, PCD 3

FOREWORD

This Indian Standard (Fifth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum, Lubricants and Their Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The fourth revision of this standard covers the requirements and test methods of High Speed Diesel (HSD) and Light Diesel Oil (LDO) meant for automotive and non-automotive application respectively. Considering the ever increasing stringency in the requirements of automotive diesel fuel to meet the emerging emission norms, it has been decided to split the existing standard in to two separate standards covering specifications for HSD and LDO. Accordingly, it is decided to bring out the fifth revision covering the requirements and test methods of automotive diesel fuel that is HSD only. The requirements and test methods of non-automotive diesel fuel (LDO) are now covered in a separate new specification.

The background and evaluation of the diesel fuel specification is given below.

The high speed 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 high speed diesel fuel and there is an imperative need to maximize its production to meet the requirements of consumers.

In order to meet the increasing demand of middle distillates, especially high speed diesel fuel, almost all the refineries have augmented their secondary processing facilities during last ten years or so. Presently, the requirement of high speed diesel fuel is being met by blending considerable quantities of streams from different secondary processing units, such as FCC, Visbreaker, Coker, etc, with streams from primary distillation unit. It has led to certain inadequacies with regards to fuel stability.

Besides, the import of crude from diverse sources to meet almost 70 percent of the present requirement is also having an impact on the quality of fuels.

While the high speed diesel fuel quality depends not only on the crude source and refining process but also on its demand pattern, at the same time it has to satisfy number of requirements, namely, ignition quality, fuel economy, emission levels, reduced wear and tear, protection against corrosion, reduced noise, low temperature operability, etc. The fuel performance also depends to a large extent, on the design of diesel engines.

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. Flash point requirements of 66°C, *Min* of fishing vessels of 12 metres 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.

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 existing 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* upto 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, maximum for notified areas has been introduced. A requirement of lubricity was introduced for high speed diesel fuel of low sulphur content.

In fourth revision of this specification 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 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.

In the current version of the specification (fifth revision) all the two amendments of its previous version have been 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.

While assessing the quality requirement of high speed diesel fuel, the need was felt for overall improvement in order to meet the vehicle requirements of emerging emission norms. While finalizing these specifications, considerable assistance has been derived from the recommendations of Expert Committee on Auto Fuel Policy headed by Dr. R.A. Mashelkar, Director General, CSIR and comments / data received from all stakeholders was considered. In view of the safety concerns expressed by SIAM, ARAI, Consumer Forums etc and the flash point being specified by various countries, it is decided to review the flash point requirement after receiving the report on hazard study to be conducted by the specially constituted group. Accordingly, the requirements of HSD for vehicles meeting Bharat Stage II and Bharat Stage III Emission 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.

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, 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 high speed diesel fuel at the time and place of delivery.

Nothing in this standard shall, however, preclude observance of the regulations, which may be more restrictive.

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

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

AMENDMENT NO. 3 SEPTEMBER 2012
TO
IS 1460 : 2005 AUTOMOTIVE DIESEL FUEL — SPECIFICATION

(Fifth Revision)

(*Page* 1, *clause* **3.1.3**) — Substitute the following for the existing clause:

'**3.1.3** The use of dyes is permitted.'

(PCD 3)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 MARCH 2010
TO
IS 1460 : 2005 AUTOMOTIVE DIESEL FUEL — SPECIFICATION

(Fifth Revision)

[*Foreword*, *page* (ii), *para* 9] — Add the following at the end:

'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 in Annex C. The date and area for implementation of this specification are as decided by the Competent Authority.'

[*Foreword*, *page* (iii), *para* 4] — Substitute 'Annex D' *for* 'Annex C'.

(*Page* 1, *clause* **2**) — Add the following at the appropriate place:

[P : 34] : 1979	Determination of sulphur in petroleum products (lamp method) (*second revision*)
[P : 83] : 1974	Determination of sulphur by Wickbold oxyhydrogen method
15607 : 2005	Bio-diesel (B 100) blend stock for diesel fuel — Specification

[*Page* 2, *Table* 1, *Sl No.* (xv), *col* 5] — Substitute 'D 4294/ [P : 34][9)]' *for* 'D 4294[9)].

[*Page* 2, *Table* 1, *Sl No.* (xv), *col* 5] — Delete 'D 2785/'.

[*Page* 2, *Table* 1, *Sl No.* (xviii), *col* 5] — Substitute 'ISO 12205' *for* 'ISO 11205'.

[*Page* 3, *Table* 1, *footnote* 9] — Substitute 'IS 1448 [P : 34]' *for* 'ASTM D 4294'.

[*Page* 3, *Table* 1, *footnote* 11] — Substitute the following for the existing footnote:

'[11)] Shall be applicable only for Automotive Diesel Fuel blended with 5 percent (*v/v*) Bio-diesel conforming to IS 15607 and the limit shall proportionately vary as and when the different blending percent of bio-diesel is permitted.'

[*Page* 3, *footnotes*, *Table* 1 (*see also Amendment No.* 1)] — Insert the following new footnote after footnote 12:

'[13)]All the 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.'

(*Page* 8, *Annex* B, *Table Reference*) — Substitute '[*Table* 1, *Sl No.* (xxii); and *Annex* C, *Sl No.* (xxi)]' *for* '[*Table* 1, *Sl No.* (xxii)]'.

(*Page* 10, *Annex* C) — Insert the following new Annex C and redesignate the existing Annex C as Annex D:

ANNEX C
(*Foreword*)

EURO IV/BHARAT STAGE IV EMISSION NORMS COMPLIANT — SPECIFICATION FOR AUTOMOTIVE DIESEL FUEL

Sl No.	*Characteristics*	*Requirements*	*Test Method [P:] of IS 1448/ISO/ASTM*
(1)	(2)	(3)	(4)
i)	Acidity, inorganic	Nil	[P : 2]
ii)	Acidity, total, mg of KOH/g, *Max*	To report	[P : 2]
iii)	Ash, percent by mass, *Max*	0.01	[P : 4]/ISO 6245
iv)	Carbon residue (Ramsbottom) on 10 percent residue[1)], percent by mass, *Max*	0.30	[P : 8]/ISO 10370

v)	Cetane number, *Min*	51[2)]	[P : 9]/ISO 5165
vi)	Cetane index, *Min*	46[2)]	D 4737/ISO 4264
vii)	Pour point[3)], *Max*:		[P : 10]/D 5949 or D 5950 or D 5985
	a) Winter	3°C	
	b) Summer	15°C	
viii)	Copper strip corrosion for 3 h at 50°C	Not worse than No. 1	[P : 15]/ISO 2160
ix)	Distillation, percent *v/v*, recovered at 360°C, *Min*	95	[P : 18]/ISO 3405
x)	Flash point*:		
	a) Abel, °C, *Min*	35	[P : 20]
	b) Pensky Martens closed cup[4)], °C, *Min*	66	[P : 21]
xi)	Kinematic viscosity, cSt, at 40°C	2.0 to 4.5	[P : 25]/ISO 3104
xii)	Sediment, percent by mass, *Max*	–	[P : 30]
xiii)	Total contamination, mg/kg, *Max*	24	EN 12662
xiv)	Density at 15°C[5)], kg/m^3	820-845	[P : 16] or [P : 32][6)]/D 4052/ ISO 3675 or ISO 12185
xv)	Total sulphur[7)], mg/kg, *Max*	50	ISO 20846 or ISO 20847 or ISO 20884/ [P : 83]/D 5453/ D 2622/D 4294 /[P : 34][8)]
xvi)	Water content, mg/kg, *Max*	200	ISO 12937
xvii)	Cold Filter Plugging Point (CFPP)[3)], *Max*:		[P : 110]/D 6371
	a) Winter	6°C	
	b) Summer	18°C	
xviii)	Oxidation stability[9)], g/m^3, *Max*	25	ISO 12205 or ASTM D 2274[9)]
xix)	Polycyclic Aromatic Hydrocarbon (PAH), percent by mass, *Max*	11	IP 391 or EN 12916
xx)	Lubricity corrected wear scar diameter (wsd 1.4) at 60°C, microns, *Max*	460	ISO 12156-1/Cor 1
xxi)	Oxygen content[10)], percent by mass, *Max*	0.6	Annex B

[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)] For Fuel processed from Assam crude, Cetane number and Cetane index is relaxed by 3 units.

[3)] Winter shall be the period from November to February in central and northern plains of India (both months inclusive) and rest of the months of the year shall be called as summer.

[4)] Applicable for Naval applications and fishing vessels requiring High Flash Automotive Diesel Fuel.

[5)] For fuel processed from Assam crude, the density range is relaxed to 820-855.

[6)] In case of dispute, IS 1448 [P : 32] shall be the referee test method.

[7)] For Automotive Diesel Fuel supplied to Indian Navy, the limit of sulphur shall be in agreement between the buyer and the supplier.

[8)] In case of dispute, IS 1448 [P : 34] shall be the referee test method.

[9)] This test shall be carried out only at the refinery or manufacturer's end. In case of dispute, ASTM D 2274 shall be the referee method.

[10)] Shall be applicable only for Automotive Diesel Fuel blended with 5 percent (*v/v*) Bio-diesel conforming to IS 15607 and the limit shall proportionately vary as and when the different blending percent of Bio-diesel is permitted.

*Under Review.

(PCD 3)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 SEPTEMBER 2007
TO
IS 1460 : 2005 AUTOMOTIVE DIESEL FUEL — SPECIFICATION

(Fifth Revision)

[*Page* 2, *Table* 1, *Sl No.* (xv), *col* 5] — Substitute 'D 5453' *for* 'D 5433'.

[*Page* 2, *Table* 1, *Sl No.* (xix), *col* 5] — Substitue 'D 2274[12)]' *for* 'D 2274'.

[*Page* 2, *Table* 1 *Sl No.* (xxi), *col* 5] — Substitute 'ISO 12156-1/Cor 1' *for* 'ISO 12156-1'.

(*Page* 3, *Table* 1, *footnotes*) — Include the following as 'Footnote [12)]' after 'Footnote [11)]':

'This test shall be carried out only at the refinery or manufacturer's end. In case of dispute, ASTM D 2274 shall be the referee method.'

Reprography Unit, BIS, New Delhi, India

Indian Standard

AUTOMOTIVE DIESEL FUEL — SPECIFICATION

(Fifth Revision)

1 SCOPE

This standard prescribes the requirements, sampling procedure and test methods for automotive diesel fuel. It is applicable to automotive diesel fuel for use in diesel engine vehicles designed to run on automotive diesel fuel.

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 the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods *(first revision)*
1447 (Part 1) : 2000	Petroleum and its products — Methods of sampling: Part 1 Manual sampling *(first revision)*
1448	Methods of test for petroleum and its products:
[P : 2] : 1967	Acidity *(first revision)*
[P : 4] : 1984	Ash, sulphated ash and water soluble ash *(second revision)*
[P : 8] : 1967	Carbon residue by Ramsbottom method *(first revision)*
[P : 9] :1960	Cetane number
[P : 10] : 1970	Cloud point and pour point *(first revision)*
[P : 12] : 1967	Colour by ASTM colour scale *(first revision)*
[P : 15] : 1976	Detection of copper corrosion from petroleum products by the copper strip tarnish test *(second revision)*
[P : 16] : 1990	Density of crude petroleum and liquid petroleum products by hydrometer method *(third revision)*
[P : 18] : 1991	Distillation *(second revision)*
[P : 20] : 1998	Determination of flash point by Abel apparatus *(second revision)*
[P : 21] : 1992	Flash point (closed) by Pensky Martens apparatus *(second revision)*
[P : 25] : 1976	Determination of kinematic and dynamic viscosity *(first revision)*
[P : 30] : 1970	Sediment in crude and fuel oils by extraction *(first revision)*
[P : 32] : 1992	Density and relative density *(second revision)*
[P : 40] : 1987	Water by distillation *(third revision)*
[P : 110] : 1981	Cold filter plugging point of distillate fuels

3 REQUIREMENTS

3.1 General

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

NOTE — The approximate gross calorific value of the fuel is of the order of 10 500 k cal/kg.

3.1.1 This fuel shall not contain any residuum oil.

3.1.2 Bio-diesel up to 5 percent *(v/v)* may be blended with automotive diesel fuel [*see* Table 1, Sl No. (xxii)]. Stabilizing agents, as required, shall be incorporated.

3.1.3 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 5 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier and subject to the provision of Red Tariff No. 18 Rules and Rates for the Conveyance by Rail of Explosives and Other Dangerous Goods, issued by the Indian

Table 1 Requirement for Automotive Diesel Fuel

(*Foreword* and *Clauses* 3.1.2 *and* 3.2)

Sl No.	Characteristics	Requirements		Method of Test, Ref to [P:] of IS 1448/ISO/ASTM D/ IP/EN/Annex of this Standard
		Bharat Stage II	Bharat Stage III	
(1)	(2)	(3)	(4)	(5)
i)	Acidity, inorganic	Nil	Nil	[P : 2]
ii)	Acidity, total, mg of KOH/g, *Max*	To report	To report	[P : 2]
iii)	Ash, percent by mass, *Max*	0.01	0.01	[P : 4]/ISO 6245
iv)	Carbon residue (Ramsbottom) on 10 percent residue[1], percent by mass, *Max*	0.30	0.30	[P : 8]/ISO 10370
v)	Cetane number[2], *Min*	48[3]	51[3]	[P : 9]/ISO 5165
vi)	Cetane index[2], *Min*	46[3]	46[3]	D 4737/ISO 4264
vii)	Pour point[4], *Max:*			[P : 10]/D 5949 or D 5950 or D 5985
	a) Winter	3°C	3°C	
	b) Summer	15°C	15°C	
viii)	Copper strip corrosion for 3 h at 100°C	Not worse than No. 1	Not worse than No. 1	[P : 15]/ISO 2160
ix)	Distillation, percent (*v/v*), recovered:			[P : 18]/ISO 3405
	a) at 350°C, *Min*	85	–	
	b) at 360°C, *Min*	–	95	
	c) at 370°C, *Min*	95	–	
x)	Flash point*			
	a) Abel, °C, *Min*	35	35	[P : 20]
	b) Pensky Martens closed cup[5], °C, *Min*	66	66	[P : 21]
xi)	Kinematic viscosity, cSt, at 40°C	2.0 to 5.0	2.0 to 4.5	[P : 25]/ISO 3104
xii)	Sediment, percent by mass, *Max*	0.05	–	[P : 30]
xiii)	Total contamination, mg / kg	–	24	EN 12662
xiv)	Density at 15°C [6], kg/m^3	820-860	820-845	[P : 16] or [P : 32][7]/D 4052/ ISO 3675 or ISO 12185
xv)	Total sulphur [8], mg / kg, *Max*	500	350	IP 336 or D 4294[9] ISO 14596 or ISO 8754/ P: 83/D 2785/D 5433/D 2622/D 3120
xvi)	Water content, percent *(v/v)*	0.05	–	[P : 40] /ISO 3733/ISO 6296
	Water content mg / kg, *Max*	–	200	ISO 12937
xvii)	Cold filter plugging point (CFPP)[4], *Max:*			[P : 110]/D 6371
	a) Winter	6°C	6°C	
	b) Summer	18°C	18°C	
xviii)	Total sediments[10]mg per 100 ml, *Max*	1.5	–	Annex A /ISO 11205/D 2274[10]
xix)	Oxidation stability, g / m^3, *Max*	–	25	ISO 12205 or D 2274
xx)	Polycyclic aromatic hydrocarbon (PAH), percent by mass, *Max*	–	11	IP 391 or EN 12916
xxi)	Lubricity corrected wear scar diameter (wsd 1.4) at 60 °C, microns, *Max*	460	460	ISO 12156-1
xxii)	Oxygen content[11] percent by mass, *Max*	0.6	0.6	Annex B

Table 1 — *Concluded*

[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] Fuel meant for vehicles meeting Bharat Stage II emission norms is required to meet either of these two parameters.

[3] For fuel processed from Assam crude, cetane number and cetane index is relaxed by 3 units.

[4] Winter shall be the period from November to February in central and northern plains of India (both months inclusive) and rest of the months of the year shall be called as summer.

[5] Applicable for Naval applications and fishing vessels requiring high flash HSD.

[6] For fuel processed from Assam crude, the density range is relaxed to 820-870 and 820-855 for Bharat Stage II and Bharat Stage III grades respectively.

[7] In case of dispute [P: 32] shall be the referee test method.

[8] For HSD supplied to Indian Navy, the limit of sulphur shall be in agreement between the buyer and the supplier.

[9] In case of dispute, ASTM D 4294 shall be the referee test method.

[10] This test shall be carried out only at the refinery or manufacturer's end. As an alternative, the test method given Annex A can also be used with a limit of 1.6 mg / 100 ml. In case of dispute, ASTM D 2274 shall be the referee method.

[11] Shall be applicable only for HSD blended with 5 percent (v/v) Bio-diesel and the limit shall proportionately vary as and when the different blending percent of bio-diesel is permitted.

* Under review.

Railway Conference Association, with any alternations or additions made thereafter.

4.2 Marking

4.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

4.2.2 Each container shall be marked with the following information :

a) Name and grade of the material;

b) Indication of the source of manufacture, initials or trade-mark, if any;

c) Volume of the contents, in litres;

d) Year of manufacture or packing; and

e) The caution label 'FLAMMABLE' together with the corresponding symbol for labelling dangerous goods as given in Fig. 5 of IS 1260 (Part 1).

4.3 BIS Certification Marking

The container may also be marked with the Standard Mark.

4.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act,* 1986 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 manufactures or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

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

ANNEX A

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

TEST FOR THE DETERMINATION OF TOTAL SEDIMENTS (Adopted from UOP Method 413-82)

A-1 GENERAL

This method is for determining the accelerated storage stability of distillate fuel oils. It is valuable for providing rapid information which indicates the tendency of an oil to form sediment and colour in storage. It is also very useful as a rapid screening test for evaluating various inhibitors that may be added to promote the storage stability of fuel oils.

There is no general agreement on what constitutes satisfactory criteria for determining stability of distillate fuels. Some believe that a filterability test, such as UOP Method 359, should be the important criterion, especially if dispersant type fuel oil additives are used. Others regard total sediment as the only important criterion. Colour and soluble gum are also often regarded as important. Other tests emphasize the light absorptivity of the sediment. This test method takes total sediment and colour as the criteria for stability.

The method employed herein, uses heat to accelerate the degradation of the distillate oil. However, it should be noted that there is considerable indication that heating does not produce results that are directly relatable to what will actually happen with some distillate oils during storage at ambient temperatures. The subject method, adapted from one suggested by Gyrath, *et al.* has been adopted because it is quick and convenient. It also differentiates nicely between the various additives and is usually useful in suggesting the concentrations of inhibitors to be added to fuel oils for long-term storage tests.

A-2 OUTLINE OF THE METHOD

The fuel oil is heated at 100°C in the presence of oxygen for 16 h. The entire sample is filtered and the amount of sediment collected is weighed. The colour or transmittance of the oil is also measured before and after the heating period for comparative purpose.

A-3 APPARATUS

A-3.1 Induction Period Apparatus — Consists of (*see* Fig. 1) an enclosed water bath (1) or a multiple thereof, each containing 4 stainless steel oxygen pressure vessels (2) having inside dimensions of 54 mm (preferred) to 57 mm diameter by 210 mm height, sufficient to hold a 250 ml Pyrex sample bottle (3). The head (4) of each pressure vessel has a square groove joint and fits closely (with a gasket) onto the pressure vessel. The head is held in place by six bolts with hexagonal nuts. The flange at the top of the pressure vessel is tapped and a stainless steel tube connects the pressure vessel with a pressure recorder (5).

The recorder may be operated with 1 to 4 points, allowing a different colour pen for each pressure vessel in the bath. The manifold (6) connects each pressure vessel to the pressure regulated oxygen tank (7) via individual valves (17) and is provided with a 690 kPa relief valve (14), a block valve (15) and a vent valve (16). Heating or cooling is achieved by introducing either steam (18) or cold water (19) into the water bath.

A-3.2 Balance — capable of weighing to 0.1 mg.

A-3.3 Bottles — 250 ml, Pyrex, constructed from 51 mm OD standard wall tubing, 100 mm high with a 22 mm ID neck opening.

A-3.4 Cell — Spectrophotometer 40 mm path length, UV silica.

A-3.5 Colorimeter — According to ASTM D 1500.

A-3.6 Cylinder — 200 ml, graduated.

A-3.7 Desiccators — with aluminium plate.

A-3.8 Dish, Petri — borosilicate glass, 60 mm diameter.

A-3.9 Filter Holder — stainless steel, 'Millipore Hydrosol filter holder'.

A-3.10 Flask — filtering, with side arm, 500 ml.

A-3.11 Gaskets — 0.79 mm thick, asbestos composition for pressure vessels, shall be found suitable when used in conjunction with the Teflon gasket lubricant.

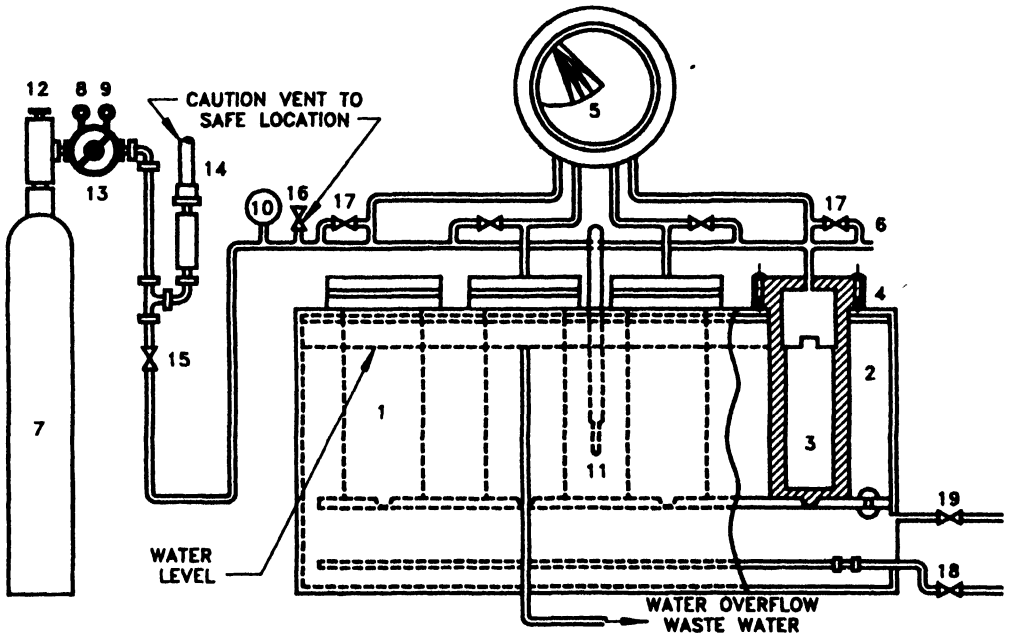
A-3.12 Gasket Lubricant — Teflon aqueous suspension, used to coat gasket and to prevent sticking. Gaskets are soaked for 5 minutes in a 1:1 aqueous solution, then allowed to air dry, hanging from a horizontal rod, for at least 3 h.

A-3.13 Oven — Gravity convection, capable of operation at 150 ± 1°C.

A-3.14 Pipes, Automatic — 150 ml capacity.

A-3.15 Pipette, Mohr — Long tip Pyrex, 2 ml graduated, 0.01 ml divisions.

A-3.16 Pump Vacuum — heavy duty.



1. Water bath
2. Oxygen pressure vessel
3. Sample bottle
4. Pressure vessel head
5. 4-Point pressure recorder
6. Oxygen manifold
7. Oxygen tank
8, 9, 10. Pressure gauges
11. Thermometer
12. Oxygen tank valve
13. Pressure regulator
14. Relief valve
15. Block valve
16. Vent valve
17. Valve
18. Steam supply valve
19. Cold water

FIG. 1 INDUCTION PERIOD PRESSURE VESSEL BATH

A-3.17 Recorder — 4 pen spiral type, range 0-1 380 kPa.

A-3.18 Regulator — Oxygen, 2-stage, delivery pressure range 28-690 kPa.

A-3.19 Shaker

A-3.20 Spectrophotometer — Single beam. Modify cell holder, if required, to accept a 40 mm cell.

A-3.21 Thermometer — Viscosity thermometer No. 22 C, having a range of 95°C to 103°C as prescribed in ASTM Specification E1 or equivalent.

A-4 REAGENTS AND MATERIALS

All reagents shall conform to the relevant Indian Standards when such standards are available, unless otherwise specified. References to water mean deionized or distilled water. Unqualified references to solutions mean aqueous solutions.

A-4.1 Acetone, Dry

A-4.2 Gum Solvent, Toluene-Acetone-Methanol (TAM) — Mixture 1 : 1 : 1 by volume.

A-4.3 *n*-Heptane — 99 percent minimum purity.

A-4.4 Hydrochloric Acid — 1 : 1 by volume.

A-4.5 Membrane Filter Discs — 0.8 micrometer, 47 mm diameter, white, plain, MF-Millipore, or equivalent.

A-4.6 Oxygen, Extra Dry — 99.6 percent minimum purity. CAUTION, use extreme care in the handling of compressed oxygen.

A-5 PREPARATION OF APPARATUS

Thoroughly clean all pressure vessels (2), heads and gasket seats (4) with acetone soaked paper towels so that they are free from any traces of gasoline, oil or gum deposits. Be certain that all traces of acetone are removed from the pressure vessels, etc. Using brief bursts of oxygen from the oxygen tank (7), set at 690 kPa according to gauges (9) and (10) make sure that the oxygen inlet to the pressure vessel via the oxygen manifold (6) is dry and free of any traces of acetone. Clean several pyrex sample bottles to remove any trace of lead deposits, which may remain from leaded fuels, using 1:1 HCl. Following this, remove detergents, oils and gums by using gum solvent (TAM) followed by first an acetone and then a distilled water rinse with subsequent drying at 105°C.

A-6 PREPARATION OF SAMPLES

Filter a sufficient amount of the fuel sample through a 0.8 mm filter and measure the colour by IS 1448 [P : 12]. If desired, also determine the percent transmission with a spectrophotometer at 650 nm in a 40-mm cell using water as the reference. Report these data for the unaged fuel. The sample bottle is charged from an automatic pipette, or a 200 ml graduated cylinder, with 150 ml of the filtered fuel to be tested and is placed into the centre of the pressure vessel well.

If the stability of a given fuel is to be tested with an inhibitor additive, which is normally added in the range of 2 to100 weight-ppm, accurately prepare 100 ml of a 1-2 weight/volume percent toluene solution of the additive. Follow this by a secondary toluene dilution in such a way that an aliquot of one to two ml from a graduated 2 ml pipette, when added to 150 ml of fuel to be tested, produces the desired weight-ppm additive concentration. Simultaneously agitate up to 9 sample bottles containing the fuel with the additive by using the Junior Orbit Shaker of a safe rotational speed setting for about 10 min. Place the sample bottles into the pressure vessel wells.

A-7 PROCEDURE

A-7.1 Oxygen Purge

Bring all oxygen pressure vessels containing the test samples to a uniform temperature of 15-25°C by turning on the cold water supply valve at the water bath of the pressure vessels apparatus and allowing the water to exit freely at the stand pipe overflow for approximately 60 min. Install the circular charts in the pressure recorder for later leak testing of the pressure vessels. Position a new Teflon-coated gasket on the pressure vessel head, cover the pressure vessel, with the lid and hand tighten all nuts. Using a manually operated torque wrench at 275 kPa or a pneumatic wrench with an air supply at 620 kPa, first tighten opposite nuts lightly, then firmly to 275 kPa.

Flush out the air in the pressure vessel with oxygen by closing the exhaust vent opening the oxygen tank valve, block valve, needle valves and slowly adjusting the cylinder regulator valve until the gauges read 690-705 kPa. Next, close block valve and slowly release all pressure by opening the exhaust vent, slowly reopen the block valve to repressurize the system with oxygen, and repeat two more purges as described. Finally, leaving the pressure vessels pressurized under 690 ± 4 kPa of oxygen by closing all valves as well as block valve slowly open exhaust vent until the pressure gauge indicates zero kPa, and check for pressure vessel leaks by observing the pens of the pressure-recorder. The recorder pen should show no change over a period of 15 min.

After a leak free system is assured, slowly release the oxygen in each pressure vessel by opening all needle valves until atmospheric pressure is reached. Then close the needle valves for all pressure vessel to 'lock in' the sample.

A-7.2 Start-Up

Close the cold water supply valve. Fully open the steam supply valve to heat up the water bath as rapidly as possible. Record the start-up time (steam valve opened). When the bath has reached a temperature of 100°C in about 20 min time, adjust the steam supply valve so that a reduced steam supply will assure a temperature of 100°C for the duration of the test as indicated by the thermometer. (The maximum obtainable temperature may vary slightly depending on the barometric pressure.) Allow the samples to heat or age for 16 h at 100°C under oxygen at atmospheric pressure.

A-7.3 Shutdown

Record the bath temperature from the thermometer. Turn off the steam supply valve and open fully the cold water valve. Only after the pressure vessel lids are cold to the touch, slowly release all pressure vessel pressures by opening the exhaust vent and all valves, until the pens or recorder indicated zero pressure. Unlock and remove the samples.

A-7.4 Sample Analysis

Briefly swirl the sample bottles and quantitatively vacuum filter the sample through a 0.8 mm filter disc weighed to the nearest 0.1 mg, which is held in the Millipore filter holder. Remove the filtered sample from the filtering flask and, without delay, measure the colour by IS 1448 [P : 12] and, if desired, the percent transmission in a spectrophotometer at 650 nm in a 40 mm cell using water as a reference. Replace the filtering flask rinse the sample bottle with three, 25 ml portions of *n*-heptane, transferring each wash to the filter holder. Thoroughly wash the filter disc and holder with *n*-heptane, using vacuum, to remove all traces of fuel oil. Any residual fuel oil remaining on the filter will influence sediment measurement. Discard the *n*-heptane filtrate.

Place the filter in an appropriately labelled petridish and dry the filter in an oven for 30 min at 95°C (do not exceed 95°C). Remove the dish and filter from the oven and cool in a desiccator for 1 h. Weigh and filter disc to the nearest 0.1 mg. Correct sediment weights for a moisture blank by processing a filter blank in exactly the same fashion, heptane washing and drying, as a sample. A blank should be determined for every four oil samples analyzed.

A-8 CALCULATIONS

Report the ASTM colour, as well as the percent

transmission, if desired, for the filtered fuel before heating (unaged fuel), and again after heating (aged fuel). An approximation of the ASTM colour may be obtained by converting the percent transmission using the Correlation Table 2. The quantity of sediment formed, in mg, after heating of the aged fuel is calculated for 100 ml of sample as follows:

$$\text{Sediment, mg/100 ml} = \frac{(A - B)}{1.5}$$

where

A = mass of sediment in sample, in mg; and

B = mass of sediment in blank, in mg.

Table 2 Correlation

(*Clause* A-8)

Filter Value	Percent Transmittance 650 nm, 40-mm Cell
(1)	(2)
0.5	88.5-100.0
1.0	82.5-88.4
1.5	76.3-82.4
2.0	70.1-76.2
2.5	64.1-70.0
3.0	58.0-64.0
3.5	51.8-57.9
4.0	45.7-51.7
4.5	39.8-45.6
5.0	33.6-39.7
5.5	27.6-33.5
6.0	21.5-27.5
6.5	15.2-21.4
7.0	9.2-15.1
7.5	3.2-9.1
8.0	0.0-3.1

A-9 INTERPRETATION OF RESULTS

Although a dark colour has no functional effect on a fuel oil, most refiners prefer to market a light-coloured oil. Colour and especially colour degradation, is considered an index of the stability of oil although no direct relationship has been established. When various oils or inhibitors are tested, the one that has the least colour degradation is considered superior. Total sediment formed is taken as an important criterion of fuel oil stability because it correlates roughly with field performance. The physical characteristics of the sediment may be more important than the actual weight of the sediment in causing filter and injector plugging in home furnaces. However, the practice of determining total weight of the sediment is an accepted technique whereas no measurement of other physical properties is generally applied. The sediment formed in long-term storage, then, is usually the criterion of fuel oil stability.

A-10 PRECISION

The estimated standard deviation (esd) for the sediment determination and percent transmission based on the indicated replicates is shown below. Duplicate results by the same operator should not differ by more than the amounts shown in the allowable difference column (95 percent probability).

A-11 TIME FOR ANALYSIS

The elapsed time for one analysis is 19 h. The effective time requirement is 1.25 h.

The test is normally run in duplicate. In groups of 10 determinations the elapsed time is 22 h and the labour requirement, per sample, is 0.55 h.

Sample Type	*Number of Replicates*	*Sediment Level* mg/100 ml	*Sediment esd,* mg/100 ml	*Sediment Allowable Difference,* mg/100 ml
Distillate	7	2	0.05	0.16
Sample Type	*Number of Replicates*	*Range of percent Transmission*	esd	*Allowable, Difference, percent Transmission*
Light Cycle Oil	8	35-43	2.7	9.0

ANNEX B

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

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

(Adopted from IOCM 156/2003)

B-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.

B-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 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.

B-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, ricebran* 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.

B-4 APPARATUS

B-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.

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

B-4.3 Syringe — 1ml syringe with luer fitting.

B-5 CHEMICALS AND REAGENTS

B-5.1 Cyclohexane — Spectroscopic grade.

B-5.2 Chloroform — Spectroscopic grade.

B-5.3 Bio-diesel Samples

B-5.4 Commercial Diesel

B-5.5 Benzène — Spectroscopic grade.

B-6 PROCEDURE

One can develop the calibration equation using known blends of bio-diesel samples as reference as 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.

B-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.

B-6.2 Determination of Cell Path Length

B-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}).

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

B-6.2.3 Cell thickness in mm = 0.1 × absorbance.

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

B-6.3 Calibration Equation

B- 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.

B-6.3.2 Measure the area under the curve in the region 1 766-1 726 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.

B-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 1 766-1 726 cm^{-1} (in 0.05 mm path length).

B-6.5 From the calibration curve, determine the

concentration of the bio-diesel in unknown sample by using the developed calibration (regression) equation.

B-7 Alternately, the combined calibration equation obtained for different bio-diesel samples (from *Palm Oil*, *Jatropa Oil* and *Sunflower Oil*) 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.

B-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} region and measure the area of the band in the region as described earlier.

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

B-7.3 Determination of percent oxygen content in bio-diesel:

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

where

CB = concentration of bio-diesel estimated.

B-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.

B-9 REPEATABILITY

0.0 to 5.0 percent	–	0.4
5.1 to 15.0 percent	–	0.8

ANNEX C

(Foreword)

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This Indian Standard has been developed from Doc: No. PCD 3 (2298).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Simco Printing Press, Delhi