

DIESEL FUEL (HIGH POUR POINT) FOR DEFENCE

K. C. MEHTA* & A. N. NANDY

Defence Research Laboratory (Materials), Kanpur

(Received 22 December 1973 ; revised 23 September 1974)

The paper reviews the studies carried out on diesel fuels produced in Indian refineries and their compatibility with Hycatrol rubber tanks fitted on some 'A' type vehicles. It gives the genesis of latest Defence specification for Diesel Fuel High Pour Point for use in A type vehicles fitted with rubberised tanks. Further improvements required in the Defence specification are also discussed.

Diesel Fuel Grade Special conforming to I.S. 1460-1959 (amended) has been in use in diesel-engined vehicles, both A & B types, for quite some time. In and around 1964 a number of cases of failure of Shaktiman engines due to seizure of pistons and rings were reported. Test bed trials were carried out on some of the engines taken from the assembly lines at Small Arms Factory, Kanpur and it was found during the period of running-in that the compression rings tended to stick due to formation of carbon deposits. It was felt that this failure could be due to substandard fuel or lubricant used. This possibility was discussed by the DIV with the Reprs. of IOC, IIP & DRL(M). During the discussions it transpired that bulk of the diesel fuel being distributed in the country was of high sulphur content upto 1% against maximum of 0.5% recommended by M/s. MAN, West Germany. Excessive sulphur fuels are reported to be the principal source of harder carbon deposits in addition to excessive corrosion within the engine.

Samples of Indian Diesel HPP were also tested by M/s. MAN. They pointed out that the Indian Diesel fuel to specification I.S. 1460 is inferior to German fuel in the following respects : (i) The sulphur content is too high—1% maximum as against 0.5% maximum of the German Diesel fuel. (ii) The final boiling point is much higher in the case of Indian Diesel fuel HPP. The portion of diesel fuel distilling over 370°C may not burn correctly with their engine and may crack and form deposits.

Again in 1967 a number of cases of failure of fuel injection pumps fitted on tanks and other equipments were reported. Technical teams from Czechoslovakia, Russia and England during the investigations of these defects commented that the quality of diesel fuel used by the Indian Army was not in accordance with their standards.

In April 1968, an armoured, regiment reported that the fuel from the rubberised fuel tanks fitted on Vijayanta tanks had become dark brown in colour and they suspected that rubber lining of the fuel tank had been affected. Laboratory tests on the DHPP fuel drained from these fuel tanks revealed that the fuel had undergone the following changes : (i) The colour had changed from pale yellow, to dark brown. (ii) Residue on evaporation (mg per 100 ml at 191°C for 2 hours, air jet) increased from 97 to 1784. (iii) Sulphur percentage by weight increased from 0.2 to 0.87.

Hydrocarbon composition of samples drawn from the supplies at various units revealed that our diesel fuels are rich in aromatics which vary from 15 to 50% approx. Aromatics are known for their high solvency towards rubbers including synthetic. It was suspected that the changes in the fuel drawn from the rubberised tanks could have been due to the effect of diesel fuel on the rubberised tanks fitted on the vehicles.

Studies were, therefore, undertaken at DRL(M) which covered (i) a general survey of diesel fuels ex-Indian refineries, (ii) comparison of requirements stipulated in I.S. specification *vis-a-vis* foreign specifications, (iii) mutual compatibility of hycatrol rubber with Indian diesels.

These studies reveal that our diesel fuel (grade special) as well as our I.S. specification for diesel fuels are deficient in some of the important properties when compared against the diesel fuels in other countries and their controlling specifications. Keeping this in view, a separate Defence specification IND/, SL/, QMG/ 4330 for Diesel fuel HPPA for use in 'A' type vehicles fitted with rubberised tanks has been drawn up.

* Presently Research Officer, Indian Oil Corporation Ltd. (R&D Centre), Faridabad.

Its details are given at the end of the paper. Some of the stipulations covered in the latest Defence specification for DHPP (A) are reviewed below :

Quality

In the Defence specification, it has been stipulated that the diesel fuel shall be a clear and bright petroleum distillate, stable on storage. It shall not contain any residuum oil. U.K. Defence specification No. DEF 2402 B and ASTM D 975 (T) stipulate a distillate fuel for 47 cetane and 1 D & 2 D fuels respectively. The Canadian specification 3 GP. 15 B calls for 100% straight run distillate. Indian Standard Specification No. 1460-1959 (amended) does not require the fuel to be a straight run distillate. Evidently a 100% straight run distillate devoid of cracked products, coker kerosene etc., would be more stable on storage and is not expected to create filter choking and injector deposit problems. It would, therefore, be preferred by Defence, where stocks of diesel fuels normally have to be stored for long periods without much turn over.

Colour

In the Defence specification colour has been stipulated as 3.5 maximum (ASTM D-1500). The colour shows the degree of refining and the pattern of blending. A straight run distillate or hydro-treated fuel shall be finer and lighter in colour whereas the fuels blended with cracked products and containing more 'higher fractions' would be darker in colour. The survey reveals that samples from Koyali, Cochin and AOC refineries meet the requirement stipulated in the Defence specification. The samples from Barauni, Esso and Burmah Shell refineries are much darker (Table 1).

Flash Point

The flash point in the Defence specification has been stipulated as 55°C minimum. This is in accordance with the original I.S. specification. It is evidently with a view to have a product devoid of naphtha cuts and coker kerosene, which are being added to maximize diesel fuel production. This is also in conformity with foreign specifications like B.S. 2869-1957 for class 'A' fuel. U.K. DEF specification and Canadian 3 GP-15 B specification stipulate flash point 66°C minimum. Most of the samples included in the survey showed flash points above 55°C (131°F).

Carbon Residue

This property is related to combustion chamber deposits. The I.S. specification lays down Ramsbottom carbon residue content as 0.2% maximum, when determined on the fuel as such, whereas foreign specifications like U.K. DEF and Canadian stipulate carbon residue 0.2% maximum, when determined on 10% residuum. The stipulation by the latter method evidently is more representative and realistic in relation to carbon deposits in the combustion chambers. All the samples included in the survey showed carbon residue less than 0.2% as per I.S. specification but when determined on 10% residuum, it was found to be more than 0.2% in a few cases. In a number of cases not included in the survey the carbon residue has been found to be even of the order of 0.4% on 10% residuum.

TABLE I
COLOUR BY LOVIBAND TINTOMETER*, 1 CM CELL

Fuel	Colour units (Y+5R)
Diesel fuel (Ex. Koyali)	1.9
Diesel fuel (Ex. Cochin)	6.5
Diesel fuel (A.O.C.)	11.5
Diesel fuel (Ex. Barauni)	18.5
Diesel fuel (ESSO)	39.0
Diesel fuel (Burmah Shell)	47.5

*Colour equivalent to 17 units on the above scale corresponds to 3.5 units as per ASTM D-1500 Standard.

Distillation Characteristics

The present I.S. specification lays down 90% recovery temperature as 366°C. In the foreign specifications like British standard, U.K. DEF, Canadian, Federal and U.S. Army specifications 90% recovery temperature is stipulated as 357°C. The work carried out in U.K. has shown that the materials less volatile than this give significant increases in carbon lay down on injector tips³. The Russian specification GOST 4749-49 stipulates 350°C maximum as 90% recovery temperature for diesel fuel Grade DL for use in ambient temperatures above 0°C.

Final boiling point : The final boiling point (FBP) of 385°C has been stipulated in the Defence specification in accordance with U.K. DEF and Canadian specifications. The portion of the diesel fuel distilling above 385°C would not burn correctly and could result in increased residues and combustion chamber deposits as well as in increased fuel dilution of the lubricant resulting in lowering of the viscosity of the engine oil and consequently increased wear. M/s. MAN recommended 370°C maximum FBP.

Distillation residue : This also has been limited in the Defence specification as 2% maximum as some supplies have been found to give residues even upto 7-8% which are considered highly injurious to the engine owing to likely increased chamber deposits and fuel dilution.

Sulphur

Sulphur content has been stipulated as 0.5% maximum as against 1% maximum stipulated in the I.S. specification. Obviously high sulphur fuels would result in more corrosion and wear and hard carbon deposits in combustion chamber. M/s. MAN manufacturers of Shaktiman engines recommend maximum of 0.5% sulphur. Russian specifications lay down 0.2% sulphur. Czechoslovakian specification lays down 0.4% sulphur. U.K. and Canadian specifications, however, lay down sulphur limit as 1% maximum, presumably taking into consideration the nature of crudes refined and the high cost of desulphurisation units required for reducing sulphur content and also the fact that higher detergency level oils could mitigate the adverse effects of high sulphur fuels. Evidently the adverse effects of high sulphur fuels (1% maximum) could be mitigated by the use of supplement 1 level oil or higher detergency level oils. In the trials carried out on the Shaktiman engine using 1% diesel fuel and supplement 1 level or MIL-L-2104B level engine oil like Delvac 1130, the performance of the Shaktiman engine has been found to be quite satisfactory. The engine even after completing 50,000 km run in the road trials conducted by CIV in collaboration with DRL(M) did not show any malfunctioning. But there are always some sulphur sensitive engines⁴ due to their peculiar design and metallurgy in which even the better quality of engine oils do not overcome the adverse effect of high sulphur fuels. Those engines would evidently require low sulphur fuels. The number of such engines may not be very high and it may be possible to meet their requirements from the Indian refineries producing low sulphur fuels.

Aromatics

The ill effects of aromatics generally are :

(i) Increase in the combustion chamber deposits as concentration and molecular weights of aromatics increase.

(ii) If sulphur is also in excess besides high aromatics such deposits become harder and more abrasive causing increased wear³.

(iii) If fuels containing high percentage of aromatics are used in rubber tanks incompatibility may occur due to solvent action of the aromatics on rubber. This may cause deterioration of the fuel and increased injector and combustion chamber deposits². The aromatic content in the Indian Defence specification has been stipulated as 20% maximum keeping in view the fact that the hycatrol rubber tanks are also fitted on aircrafts using A.T.F. as fuel (Kerosene type) which contains aromatics 20% maximum.

Detailed studies were later on carried out on the mutual effect of hycatrol rubber and Indian Diesel and short term storage trials were also conducted. From these studies it is revealed that some of the properties

TABLE 2
DATA ON CHANGE IN VISCOSITY AND CARBON RESIDUE OF DIESEL FUEL AFTER SWELLING TEST ON TANK RUBBER

Sample code No.	Viscosity at 100°F (C.S.)		Carbon residue (Ramsbottom % wt)	
	Original sample	After rubber swelling test at 50°C for 100 hours	Original sample	After rubber swelling test at 50°C for 100 hours
A	3.41	3.54	0.11	0.16
B	3.30	3.36	0.11	0.13
C	3.24	3.41	0.09	0.12
D	2.67	2.71	0.03	0.09
E	4.01	4.19	0.12	0.17
F	5.93	5.93	0.08	0.12
G	3.38	3.54	0.10	0.13
H	2.56	2.81	0.10	0.11
I	2.87	3.04	0.06	0.11
J	3.79	2.92	0.09	0.10
K	2.71	2.97	0.02	0.05

of the diesel fuels change when they come in contact with the hycatrol rubber (Tables 2 & 3). It has also been found that

(i) Diesel fuel with low sulphur (0.15%) and aromatic content about 25% causes excessive rubber swelling and deterioration in tensile strength as compared to diesel fuels with high sulphur and similar aromatics content (Table 4).

(ii) Diesel fuels with aromatics content higher than 30% cause excessive rubber swelling and sufficiently high decrease in tensile strength of the tank rubber. The Barauni product with aromatic content 33.5% and with sulphur 0.17% shows decrease in tensile strength by 38.3% as against 25% maximum stipulated in A.S.T.M. D 735 for oil resistant type rubbers class SR (Table 4). From data in this table it would also be seen that high sulphur fuels with aromatic content as high as 29.4%, the decrease in the tensile strength of the hycatrol rubber is within the requirements stipulated in the above A.S.T.M. standard. Summarising, in the case of low sulphur diesel fuels, aromatic content of the order of 25% could be tolerated by the hycatrol rubber tanks, now in use on sophisticated hardware while in the case of high sulphur diesel fuels 30% aromatics could be considered as acceptable without much adverse effect.

Olefins

Out of the main four types of hydrocarbons present in the petroleum products, namely, paraffins, naphthenes, aromatics and olefins, the olefins are most susceptible to oxidation or auto-oxidation processes which are taking place at all temperatures. These olefins get auto-oxidized to form varnishes and lacquers which with passage of time come out in the form of insoluble gums and choke the filters normally provided in the fuel systems. It is therefore, desirable that the olefin content should be as low as possible. In the Defence specification for diesel fuel (DHPPA) the olefins have been limited to 5% by volume. This limit is based on the I.S. specification for A.T.F. (Kerosene type). Some studies have been carried out on the relationship of olefin content and accelerated gum determined by A.S.T.M.D. 2274. The data is reproduced in Table 5. In some cases the accelerated gum content obtained by the A.S.T.M. method is quite excessive, but no direct relationship in these two properties has been observed indicating that other hydrocarbons like aromatics etc. may also be affected in this accelerated test.

It is, however, considered that a suitable storage stability clause and gum limit could easily take care of the olefin requirement stipulated in the existing Defence specification for DHPPA. Addition of suitable antioxidants could also mitigate the adverse effects of olefinic hydrocarbons when present in the diesel fuels.

CONCLUSIONS

In the Defence services since the diesel fuel is required to be stored for long periods, the need for a better and stable diesel is inescapable. Further since large number of sophisticated hardware is fitted with

TABLE 3

STORAGE TRIALS OF DIESEL FUEL (J) IN PANNIER BAGS

Test	Sample as received	Sample drawn after					
		1 month	2 months	3 months	4 months	5 months	6 months.
Colour test by Lovibond tintometer in 1 cm cell (Y+5R)	18.5	20.7	20.7	22.0	24.0	—	—
Specific gravity at 60/60 F	0.876	0.876	0.876	0.876	0.876	0.876	0.876
Cetane index	39	43	42	43	42	42	42
Diesel index	41	41	41	41	41	41	41
Flash point (P&M), °F	168	168	168	168	168	168	168
Viscosity at 100°F in Cs	3.7	3.8	3.8	3.8	3.8	3.9	3.9
Pour Point, °F	40	40	40	40	40	40	40
Carbon residue on 10% residuum (% by wt)	0.17	0.21	0.23	0.25	0.35	0.37	0.41
Residue on evaporation (Steam jet) for 2 hrs, mgm/100 ml)	27	32	33	29	37	69	66
Aromatics (% vol)	33.1	34.3	34.6	35.2	35.4	35.1	35.2
Bromine No.	9.60	8.80	8.50	7.89	7.85	7.98	7.94
Olefins (% vol)	9.9	8.6	8.4	7.8	7.6	7.8	7.8
Total sulphur (%wt)	0.17	0.23	0.24	0.23	0.22	0.22	0.20

TABLE 4

DATA ON CHANGE IN TENSILE STRENGTH, VOLUME AND PER CENT ELONGATION OF TANK RUBBER AFTER ACCELERATED SWELLING TEST (100°C-70 HOURS) (ASTM D-412)

Diesel sample code No.	Aromatics (%vol)	Total sulphur (% wt)	Average tensile strength (T.S.) of rubber kg/sq cm			Average elongation at break (Per cent)			Change in volume (%)
			Original sample	After swelling	Change in T.S. (%)	Original rubber sample	Rubber after swelling	Change in elongation	
E	24.9	0.15	176	131	-25.6	557	543	-2.5	16.9
F	17.2	1.0	176	165	-6.3	557	540	-3.1	10.2
G	29.4	0.93	176	144	-18.2	557	540	-3.1	13.8
H	26.3	1.0	176	143	-18.8	557	517	-7.2	11.5
J	33.5	0.17	176	109	-38.1	557	507	-9.0	24.1
K	22.5	0.05	176	158	-10.2	557	510	-8.4	8.1

TABLE 5

OLEFIN CONTENT VS ACCELERATED GUM CONTENT OF DIESEL FUELS

Sample Code No.	Olefin content (% Vol)	Accelerated gum content (ASTM D-2274) in mg/100 ml (both insoluble & adherent gum)
C	9.0	2.6
D	2.8	1.4
E	6.1	1.0
F	7.8	1.0
G	9.6	6.0
H	6.7	11.2
I	5.7	0.6
J	9.5	0.6
K	2.5	1.1

rubberised tanks, supply of low aromatic diesel fuel is also unavoidable. In order to avoid failures of very costly engines and also with a view to reduce down time and overhauls to the minimum, a straight run distillate fuel, with colour not more than 3.5 A.S.T.M. standard (D-1500), with carbon residue not more than 0.2% on 10% residuum, with sulphur content preferably less than 0.5% and with aromatics less than 25% v/v (revised safe limits based on compatibility studies) and with 90% recovery temperatures lower than 357°C may be a possible answer. The cetane number requirement stipulated in the present I.S. specification for diesel fuels i.e. 42 minimum is expected to meet the Defence needs satisfactorily, both in A type vehicles as well as B type vehicles.

In view of this the latest IND/SL specification for Diesel fuel HPPA can be further improved upon in respect of aromatic content and cetane number requirements.

ACKNOWLEDGEMENTS

Thanks are due to the authors of Technical Reports "Improvement of the Quality of Diesel Fuel, High Pour Point, Indian Refineries H. P. P. for Defence use" on which introduction matter has been based.

REFERENCES

1. MENON, A.G., MEHTA, K.C., NARANG, J.R., TRIPATHI, V.K., SHARMA, B.R. & AGNIHOTRI, R.K., *LABDEV, J. Sci. Tech.*, 8A (1970) 33-41.
2. MENON, A.G., MEHTA, K.C., NARANG, J.R. & TRIPATHI, V.K., *LABDEV, J. Sci. Tech.*, 8A (1970), 180-185.
3. BAILY, C.L., Diesel Engine Fuels, Modern Petroleum Technology, (Richard Clay & Company Ltd. Suffolk, U.K.), 1962 p 584-599.
4. GEORGI CABL, W., Motor Oil & Engine Lubrication, 1954, 378-380.
5. MEHTA, K.C., NARANG, J.R. & NANDY, A.N., *LABDEV, (J. Sci Tech., 10A (1972).*

APPENDIX

SPECIFICATION FOR DIESEL FUEL—HIGH POUR POINT (DHPPA)

Scope

This specification prescribes the requirements for Diesel Fuel, High Pour Point suitable for use as a fuel in high speed compression ignition engines.

Quality

The Diesel Fuel shall be a clear and bright petroleum distillate, stable on storage, free from grit, suspended matter, water and other visible impurities. It may contain approved additives to improve ignition quality or other characteristics. The contractor shall intimate to the Inspection Authority the nature and concentration of the additives wherever used together with the purpose for which they have been added. No anti-smoke additives will be added.

The material, Diesel Fuel, High Pour Point, shall not contain any residuum oil.

Examination of samples taken from any portion of supply shall conform to the following requirements :

Test	Limits	
	Min.	Max.
Colour ASTM D 1500		3.5
Flash point (Pensky Martens closed °C)	55	—
Kinematic viscosity in centistokes at 37.8°C	2.0	7.5
Carbon residue (Ramsbottom) per cent by weight on 10% residuum of distillation		0.20
Cetane number	45	—
Distillation		
(i) Per cent of recovery upto 357°C	90	—
(ii) Recovery temperature for 50% by volume, °C	Not limited but to be reported	
(iii) Final boiling point °C	—	385
(iv) Residue % by volume	—	2.0
Copper strip corrosion for 3 hours at 100°C	Not worse than No. 1	
Water content, % by volume	—	0.05
Sediment, % by wt	—	0.05
Ash, % by wt	—	0.01
Acidity, inorganic	—	Nil
Acidity, total, mg. of KOH/g.	—	0.50
Sulphur, total per cent by wt	—	0.50
Pour point	—	6°C
Density at 15°C	Not limited but to be reported.	
Olefins, per cent by volume	—	5
Aromatics, percent by volume	—	20

NOTE —In so far as supplies to Indian Navy, the closed Flash Point shall be not less than 65°C.