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A Quick and Accurate Gas Chromatographic Method or Diesel Dilution in Used Diesel Engine Oils

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Abstract: *In automobiles and power generators, during operation condition mixing of fuel (Diesel) with lube oil are encountered, due to which the lube oil characteristics are changed over a period of time. To establish the level of diesel contamination (diesel dilution) under operating conditions, diesel and lube oil mix have been evaluated by GC-FID using developed markers. The developed method is validated by using a synthetic mixture. The standardized method which does not involve any hazardous chemicals has been applied for the evaluation of used lube oil samples to check the diesel dilution in the mix.*

Key Words: *Diesel dilution, Gas chromatography, Measurement uncertainty*

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I. INTRODUCTION:

Lubricant plays an important role in industry ranging from simple machines to aeroplanes and rockets. Basically it is used to prevent the wear and tear during the operation of the machine or engine. In the present age every human activity ranging from agriculture, medicine, transport industry, Power sector, space applications, aviation industry depend upon petroleum products and lubricants. Various kinds of lubricants are used in the industry like lubricating oil, grease, solid lubricant like molybdenum sulphide and graphite. Around 80 % of transport industry like cars, trucks, public transport system like buses, railways aviation industry, shipping industry uses diesel engines because of its fuel efficiency and life advantages.

Investigations of oil inside the engine have led to the conclusions that changes appear in physical and chemical characteristics of lubricant in engine [1]. These changes directly depend on condition of all elements of tribomechanical system, from their functional characteristics. The important functions of lubricating oil include cooling, reduction in friction, and wear control. But the lube oil is also susceptible to get contaminated with water, dust, solid particles, acids due to the formation of sulphur dioxide of the fuel, soot [2] and finally the fuel (diesel). This will result in the degradation of the lube oil and as a result decrease in the performance of the engine and its breakdown. In case of a trunk piston engine, the unburnt fuel may leak into the crankcase due to blow-by, because of which there is a reduction in flashpoint, viscosity, and load carrying capacity of the lubricating oil [3].

Fuel may find its way into the engine oil during idling, cold running and frequent starts and stops of the engine and causes problems. Poor combustion efficiency, faulty injectors or other leakage also causes fuel dilution. Fuel dilution greater than 2.5 % can drastically alter oil's viscosity (and hence its flow) and can accelerate wear on piston rings, liners, and crankcase bearings. Therefore using motor oil without the monitoring the state of oil can be very risky and can have the serious consequences *viz* mutual attachment of piston rings, burnt and mild covered pistons, quick spending of beds, burnt valves and finally jam engines while on the run.

Hence there is a need to analyse the oil for its contamination. Apart from Viscosity, flash points, water contents which are evaluated using standard methods, diesel (fuel) contamination/diesel dilution also has to be evaluated for trouble free condition of the engine.

Even though some spectrophotometric methods and Gas chromatographic methods [4] found in literature, an attempt has been made to validate the method for diesel contaminations in lube oils from transport vehicles using GC without the use of any hazardous chemicals.

II. MATERIALS AND METHOD

Commercial diesel purchased from market was used as the standard for the analysis of Diesel content. Fresh Lube oil, 15W/40 Lube oil mixed with diesel (5%, 10%, 20 %, 30 % and 50 %) was taken for linearity and recovery studies. Precision study was carried out at 20% diesel dilution. Lube taken out from the engine of the diesel fired power generator at 400 hours of engine operation was considered for diesel evaluation using SHIMADZU GC 2014 gas chromatography with the following experimental conditions:

Column Used : Optima-5-0.5 microns, 60 mx 0.32mm ID
Injector Temperature : 300°C
Column Temperature : Start to 150°C in 2 min, 5 °C increase up to 280 °C, hold for 35 min
Detector Temperature : :320 °C
Carrier gas : Nitrogen gas
Type of Detector : Flame Ionization Detector

III. RESULTS AND DISCUSSION:

Diesel fuel is composed of C10 to C19 hydrocarbons. It consists of 64% aliphatic, 1-2% oleifinic and 35 % Aromatic hydrocarbons. Diesel fuel boils between 165 °C to 360 °C compared to the lube oil which boils at higher temperatures

Hence the different components of diesel hydrocarbons gets eluted from around 4 minutes to 24 minutes in the order of increasing their boiling point, whereas the oil components gets eluted at a later time interval [Fig 1]. The diesel components were identified by markers. In our study since the diesel elution is distinct from the oil peaks, for calculations area integration method has been adopted for the standard diesel and the corresponding integrated areas diesel in the lube oil. It can be seen that this method can be adopted for estimation of diesel in used [Fig 2] oil up to 100% with an average recovery percentage of 95%. Method validation was done using standard techniques[Fig 3] such as method precision[Table 2] , linearity, recovery studies[Table 1] and method uncertainty[Table 3].

IV. Conclusion:

The viscosity measurements and flash point determination of the oil gives only a rough idea of the extent of diesel dilution. Considering the immediate needs of the power industry and automobile industry this provides a fast and accurate, economical validated method [6] for evaluating the diesel dilution in oils from diesel engines, without the use of any hazardous chemicals or solvents. This also gives scope for further research on recovery of diesel from the used lube oil.

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Table 1: Linearity and Recovery

Sample Identity	Sample diesel Area(A)	Std Diesel Area(S)	% Diesel	% Recovery
Ref lube oil	1333304	72227965	NA	
5% diesel spike in ref lube oil	4823815	72227965	4.8	97
10% diesel spike in ref lube oil	8076116	72227965	9.3	93
20% diesel spike in ref lube oil	15385291	72227965	19.5	97
30 % diesel spike in ref lube oil	21746829	72227965	28.3	94
50 % diesel spike in ref lube oil	35104438	72227965	46.8	94
Oil 15W/40 400 hours	2723174	72227965	1.9	NA

Table 2 : Method Precision @ 20% Diesel dilution

Trial	Diesel area for sample	Std diesel Area	Blank area	% Diesel
1	15242245	72227965	1333304	19.3
2	15081731	72227965	1333304	19.0
3	14690126	72227965	1333304	18.5
4	14520339	72227965	1333304	18.3
5	14995156	72227965	1333304	18.9
6	15260771	72227965	1333304	19.3

Average	18.9 %
Standard Deviation	300719.0
RSD, %	2.01
Recovery, %	94.4
Limit of Detection	1.2%

Table3: Uncertainty Calculations

Uncertainty component	**Uncertainty(ui)**	**ui^2**	**ui^4**	**ui^4/degree of freedom(vi)**
Type A (as Precision)	0.0245	0.00060025	3.603E-07	7.206E-08
Temperature	0.37	0.1369		0
Weighing	0.00078	6.084E-07		0
Sum		0.137500858		0
Sum of degrees of freedom				7.206E-08
Combined Uncertainty		0.370811082	0.018906486	
Effective Degrees of freedom			262371.3958	262371.3958
k			2	
Expanded Uncertainty @ 95% C.L				**0.74**
Conclusion : MU for diesel content = ± 0.74% @ 18.9%				

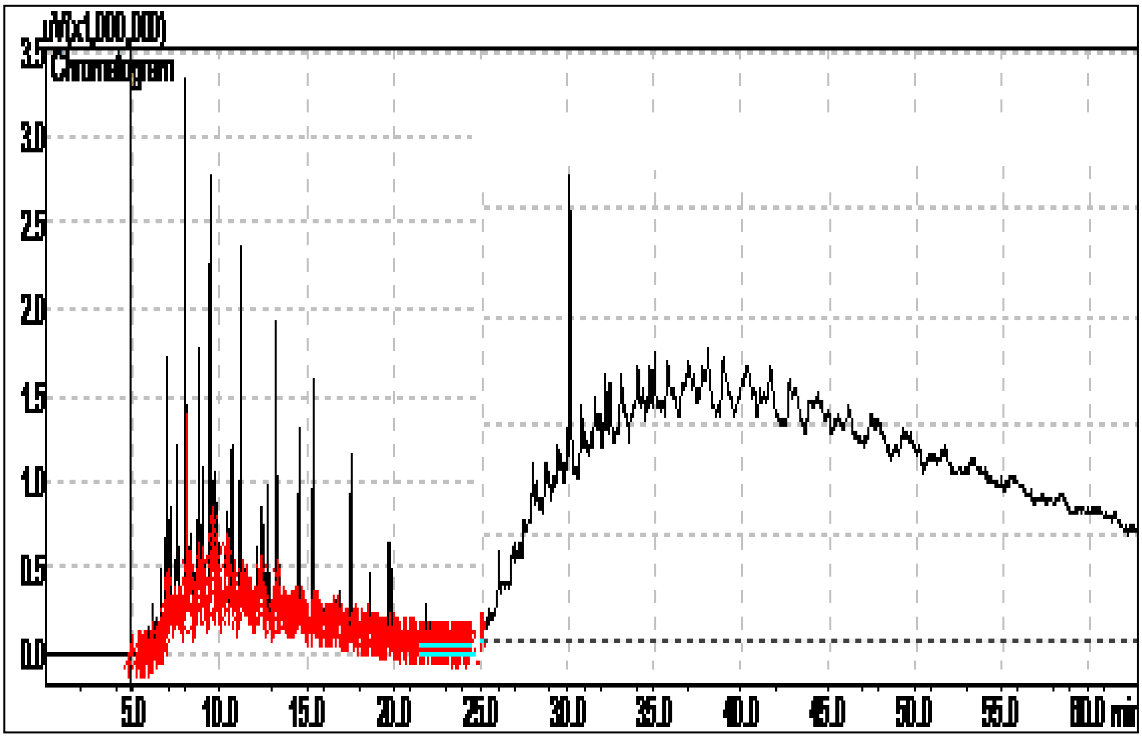

Fig 1: Gas chromatograph showing diesel and Lube oil peaks

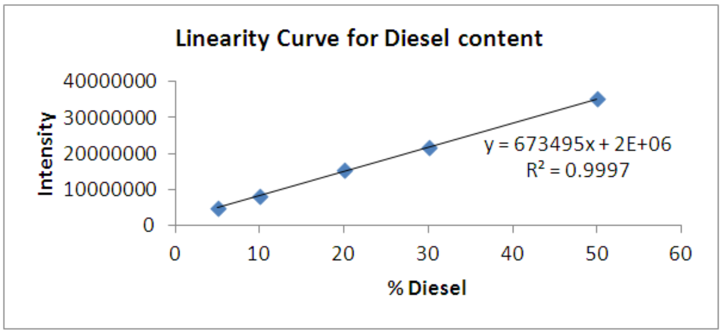

Fig 2: Linearity for Diesel content

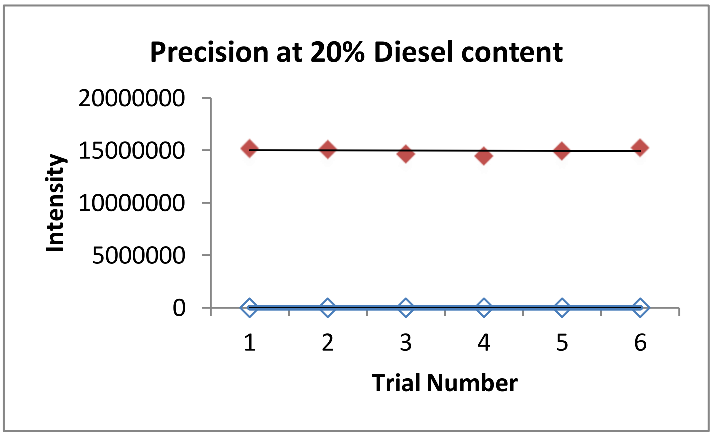

Fig 3: Precision for Diesel content @ 20% diesel dilution

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