

**IEA**  
**Advanced Motor Fuels**  
**Agreement**

**Annex XVI**

**Biodegradable**  
**Lubricants - Phase 2**

**Diesel Type Vehicles**

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## **Abstract**

The IEA Advanced Motor Fuels Agreement has initiated this project concerning the application of biodegradable lubricants in diesel and gasoline type vehicles. The member countries: Denmark, Finland, Italy, Japan, Sweden and USA have supported the project financially.

Emission measurements on a chassis dynamometer were carried out. The purpose of these measurements was to compare the emissions of CO, CO<sub>2</sub>, NO<sub>x</sub>, THC, PM, lubricant-SOF and PAH from one diesel and one gasoline type vehicle using biodegradable lubricants and conventional lubricants. This report describes the results of the experiments with the diesel type vehicle only. In another report [2] the results of the experiments on gasoline type vehicles are described. Lubricant consumption and fuel consumption are other important parameters that have been evaluated during the experiments.

Both vehicle types were operated on conventional crude oil based fuels and alternative fuels. The diesel vehicles were operated on conventional diesel fuel from a Danish fuel station and biodiesel, which was bought at a fuel station in Germany. The gasoline vehicle was really an FFV (Flexible Fuel Vehicle), which was operated on both gasoline and ethanol.

The driving patterns that were applied in these experiments were the FTP and EU test cycles.

Since the biodegradability of lubricants changes with age, it was necessary to measure this change by driving the vehicles for a number of kilometers in order to obtain a full picture of the environmental impact of implementing biodegradable lubricants. Therefore lubricant samples were taken from the engine crankcase after driving 7500 km on the road. These samples were analyzed in order to evaluate biodegradability of the used lubricant and engine wear.

## 1. Introduction.

Recently there has been increased interest in extending the use of biodegradable vegetable oils in lubricants, driven mostly by environmental as well as health and safety issues, and also arising from changes in economic and supply factors. There is a plentiful supply of vegetable oils in many parts of the world where mineral oils are expensive and in short supply. Biodegradable synthetic esters are used to a wide extent in outboard two-stroke engines, and also for other more specialized engines.

Biodegradable oils are desirable from many environmentally beneficial aspects, being advantageous from the viewpoint of oil spill or illegal waste and improved working environment in workshops.

Lubricants cause parts of the emissions from vehicle engines. From the viewpoint of emissions, biodegradable lubricants are expected to behave differently from conventional lubricants, particularly with respect to SOF emissions (Soluble Organic Fraction of particulate emissions). Vegetable oils do not contain polycyclic aromatic hydrocarbons, which pose a great risk to human health. Vegetable oils are also low in potential pollutants like sulfur containing compounds. Sulfur containing compounds are in many cases environmentally undesired, and may also cause technical problems in connection with catalytic converters.

Since the application of biodegradable lubricants is rapidly growing, several member countries of the IEA Advanced Motor Fuels Agreement (Denmark, Finland, The Netherlands, Sweden and USA) found it appropriate to be equal to the recent development, also foreseeing an extension of the use of biodegradable lubricants to more conventional automotive applications. It was therefore decided to open an annex that looked into the recent lubricant technology, and evaluated the possible future market in the light of advantages and drawbacks of biodegradable lubricants. The result of this investigation was published in 1999 [1].

The investigation revealed that there was a demand for experimental data concerning the behavior of biodegradable lubricants in automotive applications. It was therefore decided to carry out phase 2 of the annex. The purpose of this project was to investigate experimentally the technical and environmental aspects of the application of biodegradable lubricants. The countries: Denmark, Finland, Italy, Japan, Sweden and USA decided to sponsor the project. The results of phase 2 are reported in this document and in another document [2]. This report describes the results of the experiments with diesel type vehicles. In [2] the results of the experiments on gasoline type vehicles are described.

The project was carried out at The Technical University of Denmark, who is also the operating agent for the project. For more information the following address can be used:

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## 2. Biodegradable Lubricants Testing – Overview of the Investigation.

Emission measurements on a chassis dynamometer were carried out. The purpose of these measurements was to compare the emissions of CO, CO<sub>2</sub>, NO<sub>x</sub>, THC, PM, lubricant-SOF and PAH (Polycyclic Aromatic Hydrocarbons) from one diesel and one gasoline vehicle using biodegradable lubricants and conventional lubricants. Lubricant consumption and fuel consumption are other important parameters that have been evaluated during the experiments.

Both vehicles were operated on conventional crude oil based fuels and alternative fuels. The diesel vehicle was operated on conventional diesel fuel from a Danish fuel station and biodiesel, which was bought on a fuel station in Germany. The gasoline vehicle was really an FFV (Flexible Fuel Vehicle), which was operated on gasoline and ethanol.

The driving patterns that were applied in these experiments were the FTP and EU test cycles.

Since the biodegradability of lubricants changes with age, it was necessary to measure this change by driving the vehicles for a number of kilometers in order to obtain a full picture of the environmental impact of implementing biodegradable lubricants. Therefore lubricant samples were taken from the engine crankcase after driving 7500 km on the road.

In the figure below the test matrix is shown:

### Experimental Overview

Biodegradable Lubricants - Phase 2

Fuel	Lubr.	EU	FTP	Driving 7500 km	EU	FTP
Iso-octane	Bio 1	X	X			
	Basis 1	X	X			
Alcohol	Bio 1	X	X	←* →*	X	X
	Basis 1	X	X			
Gasoline	Bio 1	X	X	←* →*	X	X
	Basis 1	X	X			
LSD	Bio 2	X	X			
	Basis 2	X	X			
Diesel	Bio 2	X	X	←* →*	X	X
	Basis 2	X	X			
Biodiesel	Bio 2	X	X	←* →*	X	X
	Basis 2	X	X			

\* Lubr. Sample for biodegradability test

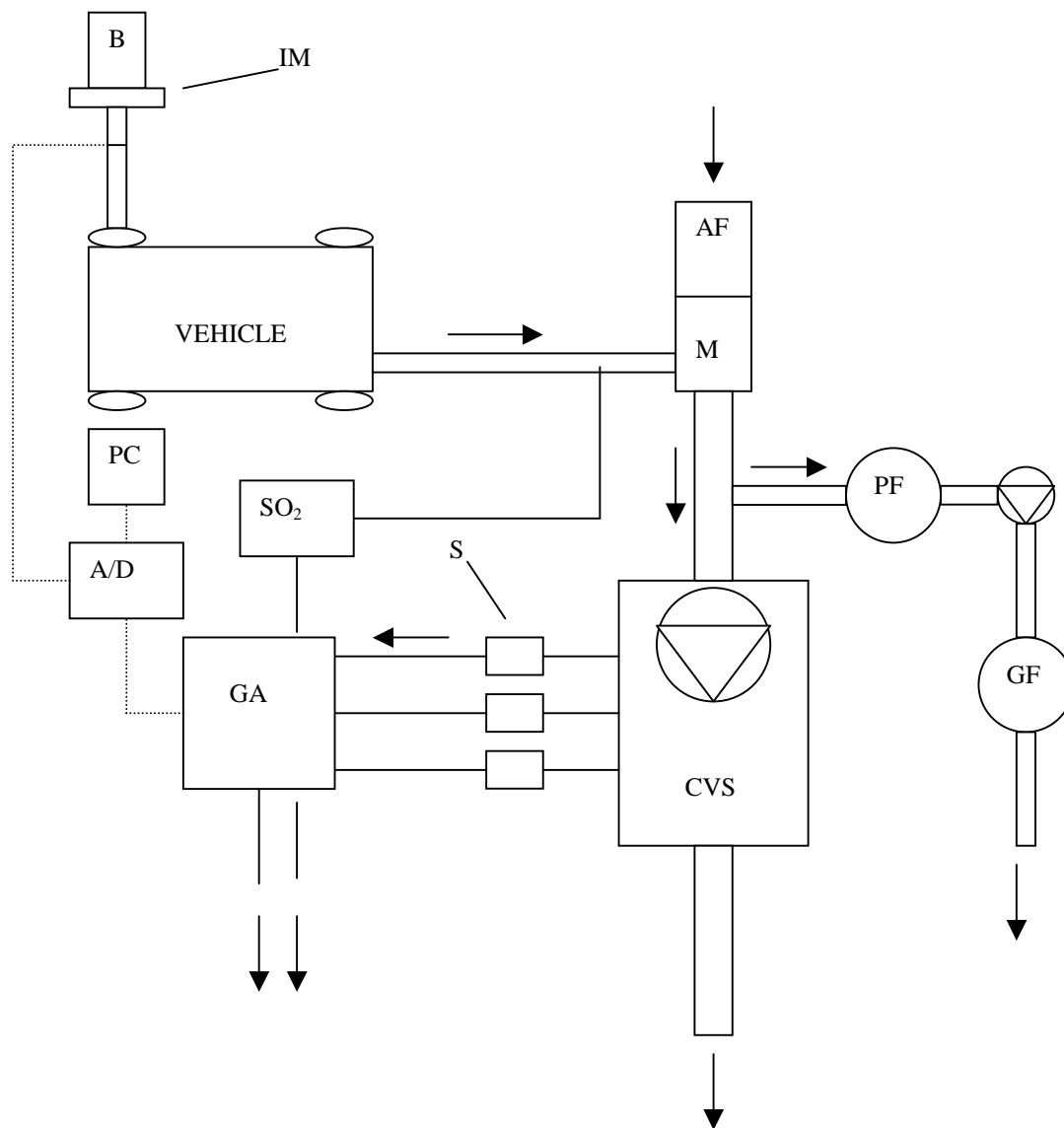
For each fuel/lubricant combination the FTP and EU cycle test were carried out. In every case 2 measurements were carried out. Gaseous emissions were measured, and PM samples were taken for SOF analysis and PAH analysis. In order to measure lubricant-SOF emissions, additional tests were carried out on Swedish low sulfur diesel (LSD) and isooctane. A new diesel vehicle was used for testing on LSD, normal diesel and biodiesel, and two new FFV vehicles were borrowed from Sweden to be tested on isooctane, gasoline and alcohol. After the first two FTP and EU tests the vehicles operating on biodegradable lubricants were run for 7500 kilometers on the roads in order to

age the lubricant. Before carrying out the FTP and EU tests again, samples of the lubricants were taken in order to measure the biodegradability and wear parameters of the aged lubricants.

### 3. Test Procedure.

A schematic picture of the experimental procedure is shown below:

(the following abbreviation applies: B: brake, IM: Inertial Mass, AF: Air Filter, M: exhaust/air Mixer, PF: Particulate sampling Filter, PC: Personal Computer for data collection, A/D: A/D converter, S: Sample bags, GF: Gas Flow meter, CVS: Constant Volume Sampler, GA: Gas Analyzers)



The vehicle is placed on a chassis dynamometer. The chassis dynamometer allows one to simulate real driving conditions in a controlled environment. The chassis dynamometer consists of two rollers driven by the wheels of the vehicle and connected to inertia plates and to a power brake. The car is placed with its driving wheels on the rollers while the other two wheels are anchored to the basement of the dynamometer with adjustable chains. The rotating resistance of the rollers simulates friction losses and aerodynamic resistance. Inertial mass has to be added to simulate the weight of the vehicle. The power absorbed by the rollers can be adjusted by regulating the power brake. The power absorbed by the brake was estimated by calculation of the deceleration time of the rollers. A speed sensor was installed giving an analog signal proportional to the speed of the rollers; the signal was converted from analog to digital. The final output is the instantaneous value of the car's velocity in km/h.

The absorbed power is adjusted at 80 km/h as specified in the FTP test procedure, which says that the power should be calculated according to the formula:

$$P = aA + P + tW$$

where:

$P_a$  = the power in kW

$a = 4,01$

$A$  = the front area of the car

$P$  = a correction factor for projecting parts

$t = 0$  for vehicles with radial tires and  $4,93 \times 10^{-4}$  for other types

$W$  = the reference weight of the vehicle in kg

A short flexible metal hose was attached to the tail pipe of the car and connected to a rigid transfer pipe whose function is to collect the exhaust gases from the diesel car and direct them into the Constant Volume Sampler. The transfer pipe is thermally insulated to minimize the risk of condensation of the water present in the exhaust gas.

In simulating driving conditions on the dynamometer, the volume of the gas emitted from the exhaust pipe of the car is continuously changing with the speed of the engine and of the load. To determine the amount of the emissions a Constant Volume Sampling method was used. The exhaust gas is diluted with a source of filtrated ambient air. The flow is regulated with a constant volume pump. With this system, an increase in exhaust flow means a decrease in the dilution air feed. The dilution ratio can be estimated, knowing the concentration of carbon dioxide before entering and after the tunnel by using the following equation:

$$DR = \frac{CO_{2,exhaust} - CO_{2,air}}{CO_{2,dilutedexhaust} - CO_{2,air}}$$



Where  $CO_{2,exhaust}$  is the concentration of  $CO_2$  in the raw exhaust,  $CO_{2,air}$  is the concentration of  $CO_2$  in the dilution air and  $CO_{2,diluted\ exhaust}$  is the concentration of  $CO_2$  in the diluted exhaust. A constant volume rate of the diluted exhaust gas can be taken and collected in sample bags. The sample bag allows one to collect the gas during different conditions and analyze it later in order to have a mean value of the pollutant concentrations. The bags are always used while running the standard driving patterns. A sufficient ratio of dilution avoids condensation of water that may cause several problems since some compounds can interact with water. Moreover, dilution air inhibits the tendency of exhaust components to react with one another, especially hydrocarbons. With this system ambient air is filtrated before diluting the exhaust. Air flows through a particle filter, an activate carbon filter and a micron filter ( $d < 2\mu m$ ): this allows one to keep the concentration of hydrocarbons under an acceptable level. A heat exchanger is placed before the pump.

The driving patterns were implemented in a program called OPTIMA 2000. This software was installed in a PC that was placed near the driving position: the driver had the keyboard inside the vehicle and was able to follow the driving patterns looking at the monitor of the PC.

#### 4. Measurements of Gaseous Emissions.

Gases sampled through the heated line were analyzed with a CUSSON P7450 Exhaust Gas Analysis System. The system includes the following instruments:

$O_2$  Analyzer: Oxygen concentration is measured with a paramagnetic ADC WA 363 analyzer

CO Analyzer: The instrument used to analyze the Carbon monoxide concentration is an ADC Nondispersive Infrared analyzer.

$CO_2$  Analyzer: Carbon dioxide concentration is measured with a non-dispersive infrared ADC analyzer

Total Hydrocarbon Analyzer: The instrument used to measure the unburned hydrocarbons present in the exhaust is 'Signal' Model 3000 Heated Flame Ionization Detector.

NO/NOx Analyzer: Nitrogen Oxides are measured by a 'Signal' Model 4000 Heated Chemiluminescent Analyzer

Before entering the  $O_2$ , CO,  $CO_2$  analyzers, the sample gas is cooled in a refrigerated water bath dryer to remove excess water vapor. An extra connection to the  $SO_2$  Analyzer was made for the experiments from the output of this cold system.

The mass,  $M_i$ , of a polluting component,  $i$ , in kg/test is determined according to US FTP as:

$$M_i = V_{mix} \cdot \rho_i \cdot kH \cdot C_i \cdot 10^{-6}$$

where:

$V_{\text{mix}}$  = volume of diluted exhaust in norm- $\text{m}^3/\text{test}$

$\rho_i$  = the density of component i, expressed as kg/norm- $\text{m}^3$

$k_H$  = NOx correction factor for humidity

$C_i$  = concentration of component i in the diluted exhaust, corrected for the background concentration, expressed in ppm

If the mass is to be expressed in g/km, the above expression has to be divided by the length of the driving pattern in km.

The concentration of component i in the diluted exhaust is calculated according to:

$$C_i = C_e - C_d \left(1 - \frac{1}{DR}\right)$$

where:

$C_e$  = concentration of component i in the diluted exhaust, expressed in ppm

$C_d$  = concentration of component i in the dilution air, expressed in ppm

DR = the dilution ratio

The dilution ratio is estimated according to a standard formula:

$$DR = \frac{13,4}{C_{CO_2} + C_{THC} + C_{CO}}$$

where:

$C_{CO_2}$  = concentration of  $CO_2$  in the diluted exhaust (%)

$C_{THC}$  = concentration of THC in the diluted exhaust (%)

$C_{CO}$  = concentration of CO in the diluted exhaust (%)

This equation is only valid at near stoichiometric combustion, which is not correct for a diesel engine. However, in our case the correction due to background concentration in the dilution air is negligible.

The NOx humidity correction factor was calculated according to:

$$kH = \frac{1}{1 - 0,329 \cdot (H - 10,71)}$$

where:

$$H = \frac{6,211 \cdot R_a \cdot P_d}{P_B - P_d \cdot R_a \cdot 10^{-2}}$$

where:

H = the absolute humidity, expressed as g water per kg dry air

R<sub>a</sub> = relative humidity, expressed at atmospheric pressure in %

P<sub>d</sub> = the saturation pressure of water at surrounding air temperature, expressed in kPa

P<sub>B</sub> = surrounding pressure in kPa

## 5. PM/SOF and PAH Measurements.

Special filters were used to collect the Particulate Matter. A separate pump carries a constant volume stream of diluted exhaust from the dilution tunnel before the CVS pump through a filter house where the filter is placed. The filter was a circular quartz fiber filter of the type Palflex TXW40HI20WW with a diameter of 293 mm. The filter was conditioned at 50% relative humidity and 20°C before measuring the mass. After sampling the filters were conditioned again at the same condition and weighed in order to estimate the mass of the emitted PM.

The soluble organic fraction (SOF) was obtained by Soxhlet extraction of the filter using dichloromethane as the extracting solvent. After the filters were extracted for 4h, the extracts were concentrated using rotary evaporation with reduced pressure at 45 °C. The mass of the SOF was determined gravimetrically from the concentrated extract.

The SOF was then used to evaluate the lubricant contribution to the emission of particulate matter and to determine the levels of certain PAH compounds.

PM consist of two major groups of material:

- 1) insoluble material, often just called soot, which mainly consist of solid carbon, water and sulfurous compounds

and

2) soluble material, which is soluble in an organic solvent, and most often just referred to as SOF (Solvent Organic Fraction).

The latter group consists of a wide number of organic compounds, which will have to be separated into individual compounds for identification and quantification. First the group is divided into a certain number of subgroups with similar chemical structure. The number of physical/chemical procedures needed for the grouping of SOF depends on the number of components. One of the groups is the PAH (Poly Aromatic Hydrocarbons) group, which are organic compounds containing several aromatic ring structures. The individual groups are finally separated into individual compounds, in our case by HPLC (High Performance Liquid Chromatography).

The organic compounds of the particulate matter were as earlier mentioned collected in Soxhlet apparatus. The extracted material was divided into 3 fractions by open column chromatography on silica gel. The eluent volumes were:

Fraction I: 7,5 ml hexane  
Fraction II: 5 ml hexane/dichloromethane (1/1)  
Fraction III: 5 ml dichloromethane

The fractions then consist of: I: Non-polar compounds (aliphatics), II: Aromatics, III: Polar components.

The aromatic fraction was analyzed in order to estimate the content of PAH compounds. These compounds were chosen for their presumed health effects and for the fact that they have been widely reported as diesel exhaust components. The individual PAH components were separated and identified (by comparison to known reference standards) using reverse phase HPLC (high performance liquid chromatography) with a fluorescence detector. The HPLC column was 2mm X 250mm long and contained a polymeric C18, 5-micron particle size stationary phase (Vydac 52TP201).

The mobile phases and the time program with respect to mobile phase flow and concentrations are shown in the following table:

Step	Time (min.)	Flow (ml/min.)	% H <sub>2</sub> O	% CH <sub>3</sub> CN
1	3	0,4	50	50
2	15	0,4	0	100 <sup>1)</sup>
3	15	0,4	0	100

1) Linear change in concentrations from step 1 to 2

It is essential for the calibration of the PAH measurements that the calibration standard composition reflects the PAH composition in the exhaust as much as possible. This is a difficult task, since there are hundreds of different PAH compounds in the exhaust. The standard that we chose for the calibration in these experiments was the EPA 610 Polynuclear Aromatic Hydrocarbon Mix, which is often used for this kind of analysis. The content of this standard is shown in the following list:

PAH-compound	Concentration (mg/l)
Acenaphthene	1000
Acenaphthylene	2000
Anthracene	100
Benzo [a] anthracene	100
Benzo [a] pyrene	100
Benzo [b] fluoranthene	200
Benzo [ghi] perylene	200
Benzo [k] fluoranthene	100
Chrysene	100
Dibenz [ah] anthracene	200
Fluoranthene	200
Fluorene	200
Indeno [123-cd] pyrene	100
Naphthalene	1000
Phenanthrene	100
Pyrene	100

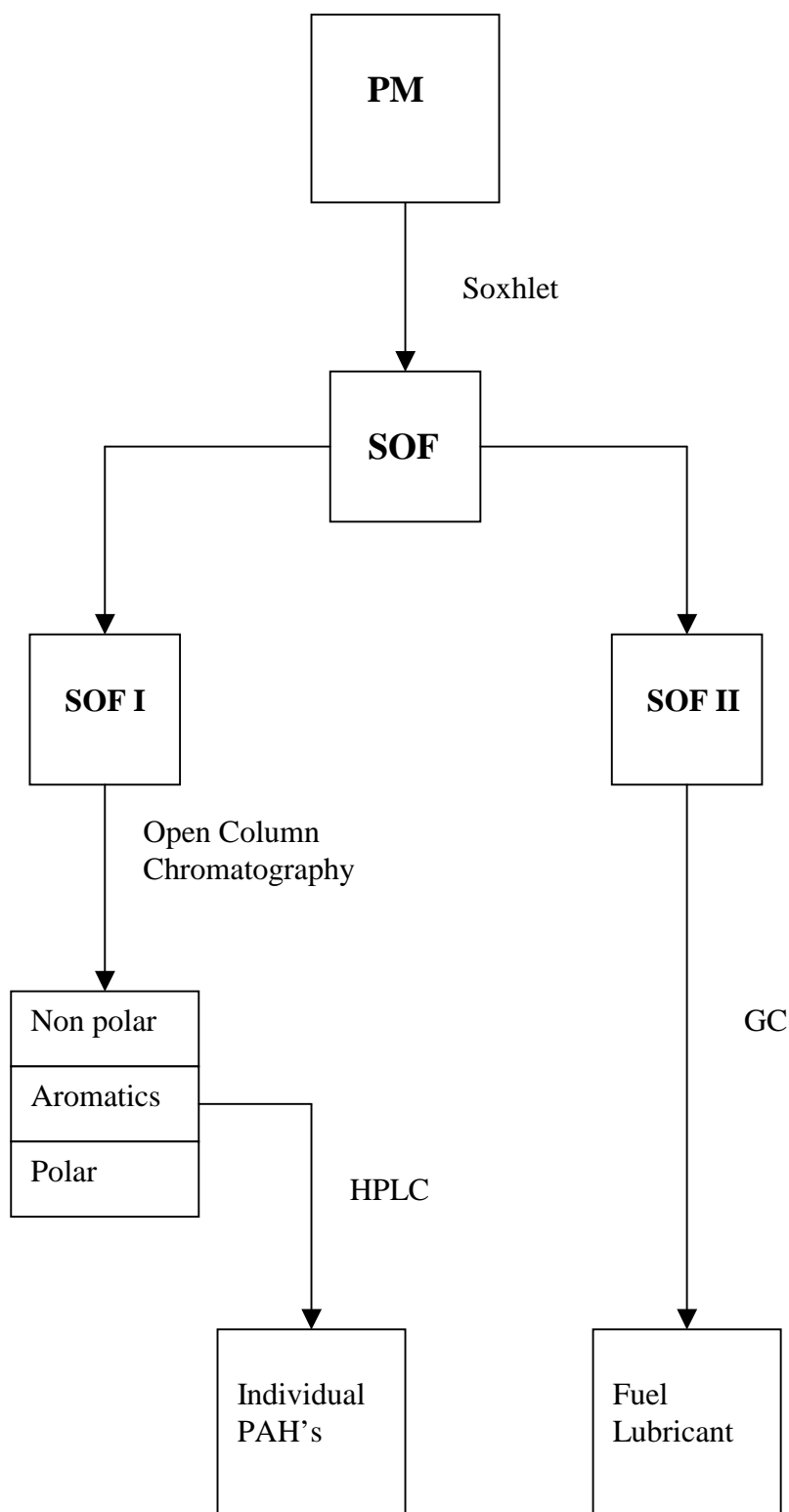
A similar sample of the SOF was used for estimation of lubricant contribution to the particulate matter. In this case the SOF was diluted by dichloromethane and the organic compounds were separated and identified as fuel or lubricant, by comparison to a sample of a mixture of pure fuel and lubricant diluted by dichloromethane, using GC (Gas Chromatography). Capillary columns coated with 100% dimethylpolysiloxane were used. The detector was an FID (Flame Ionization Detector). Helium was used as carrier gas.

The temperature programming are shown in the table below:

Step	Start Time (min.)	End Time (min.)	Start Temp. (°C)	End Temp. (°C)
1	0	3	55	55
2	3	17	55	310 <sup>1)</sup>
3	17	30	310	310

1) Linear increase in temperature from step 1 to 2.

The total analysis schedule is shown in the figure below:



## 6. Lubricant Consumption Measurements.

### Drain Weight Method.

Traditionally lubricant consumption is measured by the drain weight method. In this way the amount of lubricant added to the engine of the vehicle is weighted, and after driving a certain distance the engine is drained for lubricant and the drained weight is measured. The difference between the added amount of lubricant and the lubricant that is left over (drained from the engine) is taken as a measure of the consumed lubricant over the driven distance.

This method is only applicable when the vehicle has been driven for a large number of kilometers. Otherwise the method is too uncertain. In our case the measurements could be carried out only in the cases where the vehicles were driven for 7500 km on the roads in connection with the estimation of the biodegradability of the used lubricants (i.e. the biodegradable lubricants only). Therefore we had to adapt another method for comparison between the different lubricants. For this reason we applied the “S-Tracer Method”. The method is described in the following.

### S-Tracer Method.

Sulfur is assumed to be present in the exhaust mostly as sulfur dioxide. The total amount of SO<sub>2</sub> in the exhaust emissions is deriving from three different contributions:

- Fuel
- Lubricant
- Ambient (air)

The general equation to calculate the amount of SO<sub>2</sub> coming from the lubricant is:

$$(SO_2)_L [\%] = (SO_2)_{tot} [\%] - (SO_2)_F [\%] - (SO_2)_{amb} [\%]$$

where:

- (SO<sub>2</sub>)<sub>L</sub> [%] = amount of SO<sub>2</sub> coming from the lubricant, in volume percent
- (SO<sub>2</sub>)<sub>tot</sub> = total amount of SO<sub>2</sub> present in the sample, in volume percent
- (SO<sub>2</sub>)<sub>F</sub> [%] = amount of SO<sub>2</sub> coming from the fuel, in volume percent
- (SO<sub>2</sub>)<sub>amb</sub> = amount of SO<sub>2</sub> coming from the ambient air, in volume percent.

The concentration of SO<sub>2</sub> is usually measured in ppb (part per billion). The amount of SO<sub>2</sub> coming from the fuel can be determined when the fuel consumption and the mass percent of sulfur in the fuel is known. The molecular formula of the fuel is approximated by the formula: C<sub>a</sub>H<sub>b</sub>. Calling C<sub>F</sub> the fuel consumption, expressed in [g/h], the number of moles of fuel per hour  $\dot{n}_F$  is obtained by dividing the fuel consumption with the molar mass of the fuel:

$$\dot{n}_F = \frac{C_F}{12a + b}$$

where b is the number of H atoms and a the number of C atoms in the hydrocarbon formula of the fuel  $C_aH_b$ .

The number of moles of sulfur per hour coming from the fuel,  $(\dot{n}_S)_F$ , can be calculated as:

$$(\dot{n}_S)_F = \frac{C_F \cdot (M_S)_F}{32}$$

where:  $(M_S)_F$  is the mass of sulfur in the fuel, expressed in grams of sulfur per grams of fuel; 32 is the molar mass of S. In order to calculate the  $SO_2$  content coming from the fuel in ppb it is

necessary to know the total number of moles per hour of exhaust,  $\dot{n}_{tot}$ . The relation between the moles of fuel and the total moles is given by the reaction equation of the combustion:

$$\dot{n}_{tot} = \dot{n}_F \cdot \left[ a + \frac{b}{2} + \lambda \cdot \left( a + \frac{b}{4} \right) \cdot 3,76 + (\lambda - 1) \left( a + \frac{b}{4} \right) \right]$$

where  $\lambda$  represents the excess air ratio. The content of  $SO_2$  [ppb] coming from the fuel can be calculated as:

$$(SO_2)_F = \frac{(\dot{n}_S)_F}{\dot{n}_{tot}} \cdot 10^9$$

In order to calculate the lubricant consumption  $C_L$  it is necessary to know the mass percent of sulfur in the lubricant itself  $(m_S)_L$  and the number of total moles  $\dot{n}_{tot}$  per hour. The number of moles per hour of S coming from the lubricant, called  $(\dot{n}_S)_L$ , and can be calculated as:

$$(\dot{n}_S)_L = (SO_2)_L \cdot \dot{n}_{tot} \cdot 10^9$$

Lubricant consumption  $C_L$ , in grams per hour, can be derived knowing  $(\dot{n}_S)_L$ , the percent of sulfur in the lubricant  $(m_S)_L$  and the molar mass of S:

$$C_L = \frac{(\dot{n}_S)_L \cdot 32}{(m_S)_L} \cdot 100$$

where 32 is the molar weight of S. Combining the previous two equations yields:

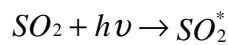


$$C_L = \frac{(SO_2)_L \cdot \dot{n}_{tor} \cdot 10^{-9} \cdot 32}{(ms)_L} \cdot 100$$

In order to maximize the sulfur content in the lubricant compared to the fuel source, a low sulfur diesel fuel (LSD) was used. The fuel and lubricant data are listed in chapter 8.

The SO<sub>2</sub> Analyzer.

The instrument used for SO<sub>2</sub> measurements was a fluorescent SO<sub>2</sub> analyzer. In this analyzer the ultraviolet light emitted from a UV lamp is filtrated through a 214 nm filter and absorbed by SO<sub>2</sub>. According to the following reaction:



where SO<sub>2</sub><sup>\*</sup> is the excited state of SO<sub>2</sub>.

When excited SO<sub>2</sub> decays to ground state a characteristic fluorescence is emitted:



The fluorescence is detected and measured by a photomultiplier tube with a 250 – 390 nm filter.

## 7. Lubricant Biodegradability Measurements.

Biodegradability tests of the used lubricants in this project were carried out according to the CEC L-33A-93 procedure. This is a method specially developed for measuring biodegradability of lubricants. More information about this method and other methods for biodegradability testing can be obtained from an earlier project report [1].

## 8. Fuels and Lubricants Applied.

The main purpose of this study was to apply a biodegradable lubricant in a diesel type vehicle, and investigate the applicability of this, compared to traditional lubricants. Therefore a commonly sold biodegradable lubricant was chosen from the European market. There were only very few products available at the start of this project [1], so the possibilities were very limited. The chosen one was a synthetic based lubricant. The available data on the lubricant are shown in Table 8.1 and denominated “BioLube”.

As a reference we chose a mineral oil based lubricant. This is due to the fact that this type of lubricant represents the traditional type of lubricant and was a very commonly sold lubricant at Danish fuel stations. The available data on this lubricant are shown in Table 8.1 and denominated “Ref.Lube”.

Lubricant		BioLube	Ref.Lube
Base Oil		Synthetic	Mineral Oil
SAE Classification		5W-40	10W-40
Density at 15°C	kg/m <sup>3</sup>	925	880
Viscosity at 40°C	mm <sup>2</sup> /s	77	99,1
Viscosity at 100°C	mm <sup>2</sup> /s	14,0	14,4
Pour Point	°C	-60	-36
Flash Point	°C	205	220
Sulfur	Wt-%	0,39	0,52

*Table 8.1. Lubricant Data.*

Three fuels were applied. These were LSD (Low Sulfur Diesel), Ref.D (reference diesel) and BioD (biodiesel). The reference diesel was chosen because this was the most commonly sold diesel at Danish fuel stations. The biodiesel was chosen because this is an alternative fuel made from biomass. This was expected to have a good influence on the biodegradability of the used BioLube. Finally the low sulfur diesel was chosen because we needed a fuel with very low sulfur content in order to be able to carry out the lubricant consumption measurements by the S-tracer method. Furthermore this fuel is “lighter” than the reference diesel, i.e. it consists of lower carbon number hydrocarbons. The relevant data on the fuels are shown in Table 8.2.

Fuel		LSD	Ref.D.	BioD
Density at 15°C	kg/m <sup>3</sup>	813	845	875-900
Boiling Point	°C	186-280	150-390	302-318
Viscosity at 40°C	mm <sup>2</sup> /s	1,86	1,9-3,7	2,8
Cetane Number		51,5	49	49
Calorific Value	MJ/kg	43,4	42,6	36,8
Sulfur	Wt-ppm	<1	30	-

*Table 8.2. Fuel Data.*

## 9. The Test Vehicle.

The test vehicle was a Citroën Saxo 1,5D. Important vehicle/engine data are given in the table 9.1.

<b>Citroën Saxo 1,5 D Vehicle/Engine Data</b>	
Model Year	2001
Engine Size	1527 cc
Engine Type	Pre-Chamber Engine
Oxidation CAT	Yes
Exhaust Gas	Yes
Recirculation	
Bore	77 mm
Stroke	82 mm
Maximum Power	42 kW at 5000 rpm
Maximum Torque	95 Nm at 2250 rpm

*Table 9.1. Vehicle/Engine Data.*

## 10. Results.

In order to make the report more readable all the figures are shown last in this chapter. All data are presented in tables in appendix 1. In the following presentation the results are shown in figures and tables in order to interpret the result for the reader.

### 10.1. Lubricant Consumption.

S-Tracer Method.

The S-tracer method was applied to the vehicle running on LSD fuel and both reference lubricant and biodegradable lubricant.

Very early it was discovered that there was a problem with NO interference, using the S-Tracer Method. This phenomenon is well known. The estimated lubricant consumption appeared unusually high.

In order to quantify the NO interference experiments were carried out with calibration gas, containing different concentrations of NO and no SO<sub>2</sub>. The correlation between the instrument reading of SO<sub>2</sub> concentration and NO concentration in the calibration gas is shown in Figure 10.1. A regression line was calculated as shown in the figure.

Several test were carried out with different speeds and loads, applying the LSD fuel and both reference lubricant and biodegradable lubricant.

The values of  $(SO_2)_{tot}$  were corrected by subtracting the NOx interference from the measured  $SO_2$  value:

$$(SO_2)_{tot} = (SO_2)_{meas} - (SO_2)_{interf}$$

where  $(SO_2)_{meas}$  is the value of  $SO_2$  measured by the instrument and  $(SO_2)_{interf}$  is the contribution of the positive NOx interference.

If we introduce the regression equation found for the NOx interference the equation becomes:

$$(SO_2)_{tot} \text{ [ppb]} = (SO_2)_{meas} \text{ [ppb]} - (7.538 \cdot \text{NOx [ppm]} + 29.37)$$

A test was carried out with reference lubricant. The procedure consisted of four phases:

1. Warming up. The car was started and let idle at minimum for 20 minutes
2. Idle Sampling. Samples of the exhaust emissions were taken upstream of the dilution tunnel at different engine speeds: 1500 rpm, 2500 rpm, 3500 rpm. This phase was called "idle cold".
3. First Gear Sampling. The car was run at 1st gear at different speeds: 1500 rpm, 2500 rpm, 3500 rpm. Samples were taken as in the previous phase.
4. Idle Sampling. The procedure was the same as the one in the 1st phase. This phase was called "idle hot", since the values of temperature measured in the exhaust line, one meter before the dilution tunnel, were higher than the one in the 1st phase: in the 1st phase the temperature increased from about 35°C at minimum to about 70°C at 3500 rpm; in the last phase the range was from about 60°C to about 90°C.

The four phases were run without interruption between each other. Particular attention was paid in taking the measurements after having waited for the same time in each test, in order to let the  $SO_2$  result stabilize and to have the same temperature conditions inside the pipe line. The test was repeated three times. Lubricant consumption for these values was calculated, subtracting the NOx interference. The different contributions to  $(SO_2)_{meas}$  are shown in Figure 10.2.

The calculated results showed a realistic increase in lubricant consumption with increasing engine speed. Figure 10.3 illustrates the values of lubricant consumption, expressed in grams per hour, calculated for the idle hot phase.

In Figure 10.4 results are shown for different engine speed with the engine running in 1<sup>st</sup> gear. In this picture we get an idea of the difficulty in measuring lubricant consumption in this way. The st.dev. of the measurements seems to be quite big, but still there is a certain tendency towards higher lubricant consumption with higher engine speed, which is very realistic.

The results of calculations of NO<sub>x</sub> interference together with values of SO<sub>2</sub> measured at idle and at 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> gear for different engine speeds, comparing the biodegradable lubricant with reference lubricant, are shown in the figures 10.5-10.10.

It is seen that in the case of 2<sup>nd</sup> and 3<sup>rd</sup> gear operation at 2500 rpm the NO<sub>x</sub> interference contribution to the SO<sub>2</sub> is bigger than the measured SO<sub>2</sub>. This illustrates the uncertainty of this method. However, from the results it is evident that the lubricant consumption is higher with reference lubricant compared to biodegradable lubricant.

#### Drain Weight Method.

After carrying out EU and FTP emission test on the used biodegradable lubricant the lubricant was drained from the engine. In this way we found that the lubricant consumption had been 1964 g pr. 7928 km. The vehicle had been driven on the roads for this distance on ref. diesel fuel in order to measure biodegradability of used lubricant. This corresponds to an average lubricant consumption of 0,25 g/km. Since the average driving speed was about 50 km/h this corresponds to about 12 - 13 g/h which is a little higher, but still the same order of magnitude as estimated from the S-tracer method in the figures 10.3-4. However, the S-tracer method should give a higher lubricant consumption, since this method was carried out during steady-state operation on reference lubricant. Furthermore, as the result from the SOF measurements indicated (chapter 10.3), the lubricant consumption from biodegradable lubricant is higher during transient operation (FTP) compared to reference lubricant. This is a positive finding, confirming the results from the rather difficult S-tracer method.

Both methods seem to indicate that the lubricant consumption from this particular vehicle is a little higher than normal.

The measured lubricant consumption was a little lower in the case of driving on biodiesel fuel with biodegradable lubricant. Here the lubricant consumption was 1260 g pr. 7850 km which corresponds to an average consumption of 0,16 g/km. Assuming again that the average speed was 50 km/h this corresponds to 8 g/h.

The large difference in consumption of biodegradable lubricant from applying different fuels indicates an interaction between these two parameters. Measurements of lubricant contribution to particulate SOF emissions support this theory, since we found much higher lubricant emissions with reference diesel fuel compared to biodiesel. This is discussed later and shown in Figure 10.21. The same measurements, as Figure 10.21 illustrates, did not show any remarkable influence of the fuel on the lubricant emissions with reference diesel lubricant.

One possible mechanism, responsible for this phenomenon could be that more reference diesel fuel dissolves in the lubricant compared to biodiesel, thus lowering the viscosity and increasing the lubricant consumption. However, as seen in chapter 10.4 the viscosity on the contrary was higher in the case of lubricant used with reference diesel fuel. Another explanation could be that the fuel consumption increases with reference diesel fuel compared to biodiesel. In this case we might suspect that the lubricant on the cylinder liner would be more diluted with fuel and thus consumed more. As discussed in chapter 10.2 this is actually seen.

## 10.2. Gaseous Emissions and Energy Consumption.

CO emissions are shown in Figure 10.11. Each diagram in the figure corresponds to each of the three fuels applied, respectively: Low Sulfur Diesel (LSD), Reference Diesel Fuel (Ref.D) and Biodiesel (BioD). In each diagram results are shown for biodegradable lubricant (Biolube) and reference lubricant (Ref.Lube). In the case with reference diesel and biodiesel long term driving on the roads for 7500 km were carried out and the measurement for these cases are denominated “Used Biolube”.

From the result we see a tendency to lower emissions with the FTP test and lower emissions with reference lubricant than biodegradable lubricant. In any case the emissions are lower than current permitted emission level in Europe.

HC emissions are shown in Figure 10.12. It is difficult to conclude anything from the figure. Apparently HC emissions are varying from time to time.

NO<sub>x</sub> emissions are shown in Figure 10.13. NO<sub>x</sub> emissions seem to be lower with low sulfur fuel. NO<sub>x</sub> emissions are generally seen to increase a few percent with the application of biodegradable lubricant. This could be due to increased mechanical loss with the biodegradable lubricant. If this is the case it should be followed by an increase in energy consumption and CO<sub>2</sub> emissions.

CO<sub>2</sub> emissions are shown in Figure 10.14. If we assume that energy consumption is proportional to CO<sub>2</sub> emissions, this figure confirms the suspicion about increased friction and energy consumption from application of the biodegradable lubricant. It is remarkable that in the cases with used biodegradable lubricant that the CO<sub>2</sub> emissions increase with FTP driving but decreases with EU driving. We also notice that the fuel consumption at FTP driving, which is a more realistic driving pattern than the EU, because it reflects the more transient driving phases of real driving, is higher with reference diesel fuel compared to both biodiesel and LSD. As discussed in chapter 10.1 we were looking for this effect in order to explain the higher lubricant consumption on the road with reference diesel.

## 10.3. Particulate Emissions.

Particulate emissions are shown in figure 10.15-16. The emissions are separated into SOF (Soluble Organic Fraction) and SOLID, which is the rest of the total particulate matter. In this way the SOLID fraction covers all the insoluble material, i.e. solid carbon, water and other inorganic compounds.

From the figures it is clear that the FTP test gives higher particulate emissions than the EU test. It is also evident that the SOF emissions are larger with biodiesel than with other fuels. This is clearly seen in Figure 10.17, where the SOF contribution in percent of the total particulate matter is shown. At the same time biodiesel results in a generally lower emission level due to very low SOLID emission.

From the figure it is also seen that during more transient driving (FTP) the biodegradable lubricant gives a higher emission of SOF, whereas the more steady-state EU driving pattern result in a lower SOF emission with the biodegradable lubricant. This latter tendency was, as earlier mentioned, confirmed by the lower lubricant consumption from the biodegradable lubricant during steady state driving, measured by the S-tracer method. This means that there probably, as expected, is a connection between lubricant consumption and lubricant contribution to SOF emissions, again indicating that the lubricant, as many other investigations have pointed out [3,4], is an important contributor to particulate emissions.

In order to investigate this further the GC analysis of the SOF could give some more information. GC pictures of the SOF are shown in the figures 10.18-19. For comparison the GC pictures of the three fuels and the two lubricants are shown in Figure 10.20. The horizontal axes in the figures are the retention times and the vertical axes gives the detector response to the compounds found in the sample. The detector is an FID (Flame Ionization Detector), which gives a signal proportional to the mass of carbon in the individual compounds.

If we look at the fuels first in Figure 10.20, we see that LSD and Ref.D consists of two groups of hydrocarbons that are relatively light, i.e. low carbon numbers. We also notice that LSD hydrocarbons are a little lighter than Ref.D. hydrocarbons. BioD., on the other hand, consists of two major “humps”, or groups of hydrocarbons, that both consists of higher carbon number compounds compared to the other fuels.

The lubricants are also principally different, because the Ref.Lube consist of one major “hump” with a retention time larger than BioD, i.e. higher carbon number, whereas BioLube has two major “humps”, the first one with a retention time similar to Ref.Lube and the second one with a larger retention time.

If we look at the GC pictures of the SOF we clearly recognizes the images of the lubricants in all the samples. It is also clear that the LSD fuel and the Ref.D. fuel does not appear in the SOF, whereas BioD. clearly appears in the SOF from driving with BioD fuel.

This seems to support the theory about the importance of lubricant in connection with particulate emissions. In fact only BioD fuel seems to give a contribution to the SOF of the particulate matter, obviously because the carbon numbers of BioD compounds are higher, compared to LSD and Ref.D. compounds. This would mean that BioD compounds would have a higher tendency to condense when the exhaust is cooled down, and thus associate with the particulate matter.

In Figure 10.21 is shown the amount of lubricant in the SOF estimated from the chromatograms. The measurements reveal an increased amount of lubricant emission with the biodegradable lubricant during transient driving (FTP) and a lower lubricant emission with the biodegradable lubricant during EU driving. This again indicates that the lubricant consumption increases with the biodegradable lubricant as the driving pattern gets more transient, and the opposite effect is seen at more steady-state driving conditions. An exception from the rule is the case with LSD fuel and FTP driving. Here the reference lubricant gives the highest lubricant emission. An explanation for this could be uncertainty in the measurement, unusual variation in the lubricant consumption or an unknown fuel/lubricant interaction during the engine cycle.

### 10.3.1. PAH Emissions.

The measured PAH emissions are shown in Table 10.1-2. Relative measurements are shown in Table 10.1., in order to make the table more easily readable. Absolute values are given in Table 10.2. In the latter case only PAH compounds with more than 2 rings are shown. This is because the standard very badly reflected the composition of PAH's with 2 or less rings, and because this fraction, as the definition says, contain a lot of monoaromatics, which are not really considered as PAH's. In any case the fraction with more than 2 rings made up the major part of the total PAH, and furthermore the most harmful PAH's are the heaviest.

As can be seen from the measurements the PAH emissions are strongly dependent on the fuel type. Reference diesel gives by far the highest PAH emissions and Biodiesel gives the lowest PAH emissions. With respect to driving pattern and lubricant type the picture is quite unpredictable. However, if we remember the CO<sub>2</sub> or (fuel consumption) measurements from Figure 10.14 it seems as if there is a connection between fuel consumption and PAH emissions. This again means that the fuel effect drowns any other effects.

In order to show the connection between fuel consumption and PAH emissions, all measurement of PAH are shown vs. CO<sub>2</sub> in Figure 10.22. The value of the correlation coefficient (R) is actually quite high when we consider the normal accuracy that we can expect on PAH emission measurements.

Some investigations have considered the possibility of the lubricant to dissolve fuel related hydrocarbons [5,6]. According to the theory, dissolved hydrocarbons should be emitted with the lubricant or just be desorbed during the expansion after the combustion, and thus entering the exhaust more or less unburned. Therefore the PAH content of the fuels and both fresh and used lubricants were measured. The results are shown in Table 10.3.

From the table we see, that the biodegradable lubricant does not dissolve any PAH, on the contrary PAH compounds seem to disappear from the lubricant. This could be due to evaporation when the lubricant is heated during engine operation. From the same figure we notice as expected that the PAH content of the Ref.D fuel is far higher than the others, and that BioD has the lowest PAH content. The Ref.Lube has a higher PAH content than the BioLube, but as earlier mentioned, the possible effect on PAH emissions is drowned by the fuel effect.

If we look at the order of magnitude of the results we notice that other investigations have shown PAH emissions of the same level [7,8]. This is encouraging.



<b>Relative PAH Emissions</b>						
	<b>≤2-rings</b>					
	BioLube		Ref.Lube		BioLube - 7500km	
	EU	FTP	EU	FTP	EU	FTP
LSD	66	59	64	47		
Ref.D	150	119	104	100	84	122
BioD	44	38	59	32	51	36

	<b>&gt;2 rings</b>					
	BioLube		Ref.Lube		BioLube - 7500km	
	EU	FTP	EU	FTP	EU	FTP
LSD	46	81	78	65		
Ref.D	143	162	94	100	56	177
BioD	58	54	53	35	85	80

Table 10.1. Relative PAH-emissions (FTP driving with Ref.D and Ref.Lube is set to 100).

<b>Absolute PAH Emissions</b>						
	<b>&gt;2 rings</b>					
	BioLube EU	FTP	Ref.Lube EU	FTP	BioLube - 7500km EU	FTP
LSD	2957	5215	5043	4236		
Ref.D	9277	10490	6083	6471	3649	11460
BioD	3741	3489	3460	2246	5524	5190

Table 10.2. PAH emissions in ng/km.

<b>PAH Content</b>					
<b>≤2 rings</b>	<b>Fuels</b>			<b>Lubricants</b>	
	LSD	Ref.D	BioD	Ref.Lube	BioLube
Fresh	0,13	0,68	0,11	0,76	0,49
Used with Ref.D					0,28
Used with BioD					0,20
<b>&gt;2 rings</b>					
Fresh	3,61	100,00	1,65	11,13	3,28
Used with Ref.D					2,21
Used with BioD					2,98

Table 10.3. Relative PAH content in fuels and lubricants (PAH with more than 2 rings for fresh Ref.D is set to 100).

#### 10.4. Engine Wear.

Engine wear is evaluated based on the analysis of fresh and used biodegradable lubricant. The results of the lubricant analysis are shown in Table 10.4.

No unusual wear is noticed in any of the cases, but it is obvious that wear is more noticeable in the case of lubricant used with Reference Diesel fuel compared to lubricant used with BioDiesel. This is seen in the increased values of metal content (Fe, Cu, Sn, and Mn). Since the engine was new (not broken in), and the Biolube/Ref. D test was run first, followed by the Biolube/BioD test, the differences in wear metals could readily be explained by the fact that the engine was more broken in during the second test. we do notice a decrease in viscosity with BioDiesel, maybe reflecting that BioDiesel is more readily dissolved in the lubricant. However, increased fuel content was not indicated by the measurements. The higher viscosity of BioLube used with Ref.D could also reflect the higher content of particulate matter that was discovered.

The lower content of “particulates” in the lubricant used with BioDiesel also supports the earlier finding that BioDiesel emits less SOLID particulate matter (chapter 10.3). In this way the lubricant is not contaminated with solid particulate matter to the same extend as with Reference Diesel.

<b>Lubricant Analysis</b>			
<b>Analysis</b>	<b>Fresh BioLube</b>	<b>BioLube Used 7500 km with Ref.D Fuel</b>	<b>BioLube Used 7500 km with BioD Fuel</b>
Viscosity (CST 100°C)	15,2	14,9	14,4
Viscosity (CST 40°C)	74	74	71
Flame Point (°C)	180+	180+	180+
Fuel Content (%)	<2	<2	<2
TBN (mgKOH/g)	12,0	13,3	14,7
Water (%)	<0,1	<0,1	<0,1
Silicium (ppm)	6	15	11
Particulates (% wt)	<0,2	0,77	0,30
Fe (ppm)	1	43	23
Al (ppm)	1	4	4
Cr (ppm)	<1	1	<1
Mo (ppm)	<1	<1	<1
Cu (ppm)	<1	5	2
Pb (ppm)	1	3	3
Sn (ppm)	<1	8	3
Ni (ppm)	<1	<1	<1
Ag (ppm)	<1	<1	<1
Mn (ppm)	<1	2	1
V (ppm)	<1	<1	<1

*Table 10.4. Lubricant Analysis.*

### **10.5. Biodegradability of Lubricants.**

The classification of a lubricant as biodegradable by the different environmental labels only covers fresh unused engine oils. However, according to different authors e.g. [9] the biodegradability of a lubricant may change considerably after only a few hours of use due to oxidation and thermal and hydrolytic reactions. Accumulation of dust, combustion products, wear particles and especially heavy metals might inhibit biodegradation as well. The amount of investigations done in the past on the biodegradability of used engine oils is very limited, but Boehme et al [10] presented a paper where they had tested the performance of a synthetic ester based biodegradable engine lubricant. Part of the test program was a test of the biodegradability of the oil before and after use, and the results showed that the degradability of the fresh oil was above 90% (CEC L33-T82), and the biodegradability of the used oil was between 70 and 80%.

The results of the tests carried out in this investigation are seen in Table 10.5.

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**Biodegradability according to CEC L-33A-93 test.**

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Fresh BioLube	BioLube used 7500 km with Ref.D	BioLube used 7500 km with BioD
90%	76%	85%

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*Table 10.5. Biodegradability test results.*

This means that after 21 days of biological activity, 90%, 76% and 85% respectively were degraded. As we expected, the biodegradability of used lubricants are not the same as for new one's. If we assume a linear degradation decrease with time and an average lifetime of a lubricant to be 15.000 km, then the results for the used lubricants in the table reflects the average biodegradability of lubricants in use.

The results agree very well with the earlier reported results [10]. Furthermore, the results show that the fuel is important in this context, since the lubricant used with BioDiesel is more biodegradable than the lubricant used with Reference Diesel. The reason for improved result with BioDiesel could very well be due to less accumulation of dust, combustion products, wear particles and heavy metals which was discussed in chapter 10.4 and shown in Table 10.4.

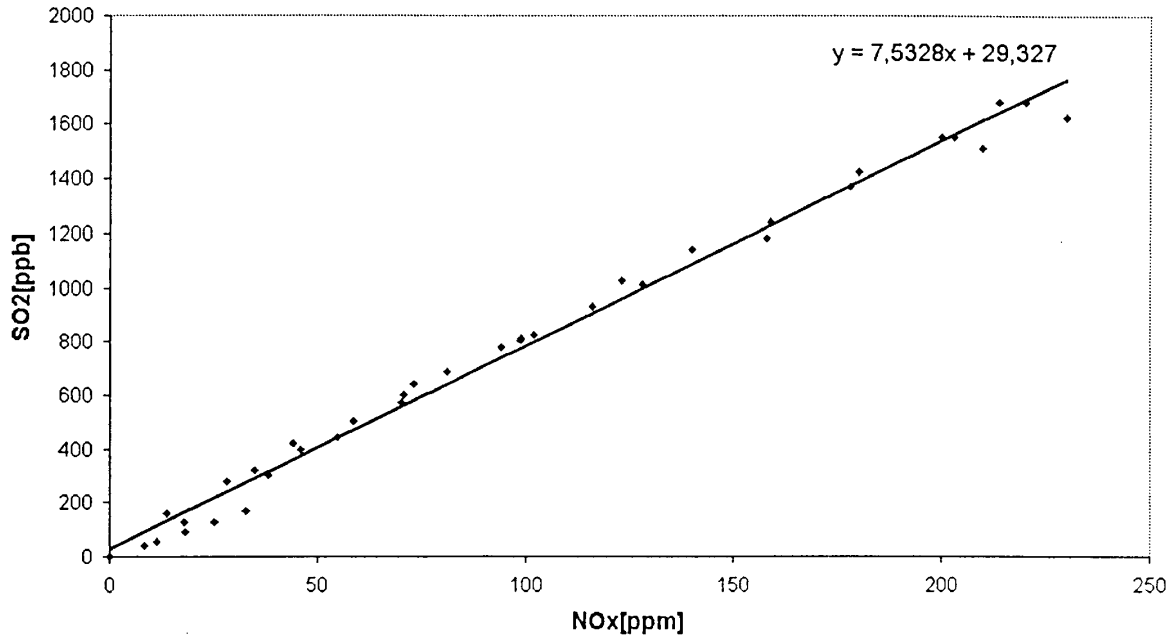


Figure 10.1. Instrument reading of  $SO_2$  for calibration gases with varying NO content.

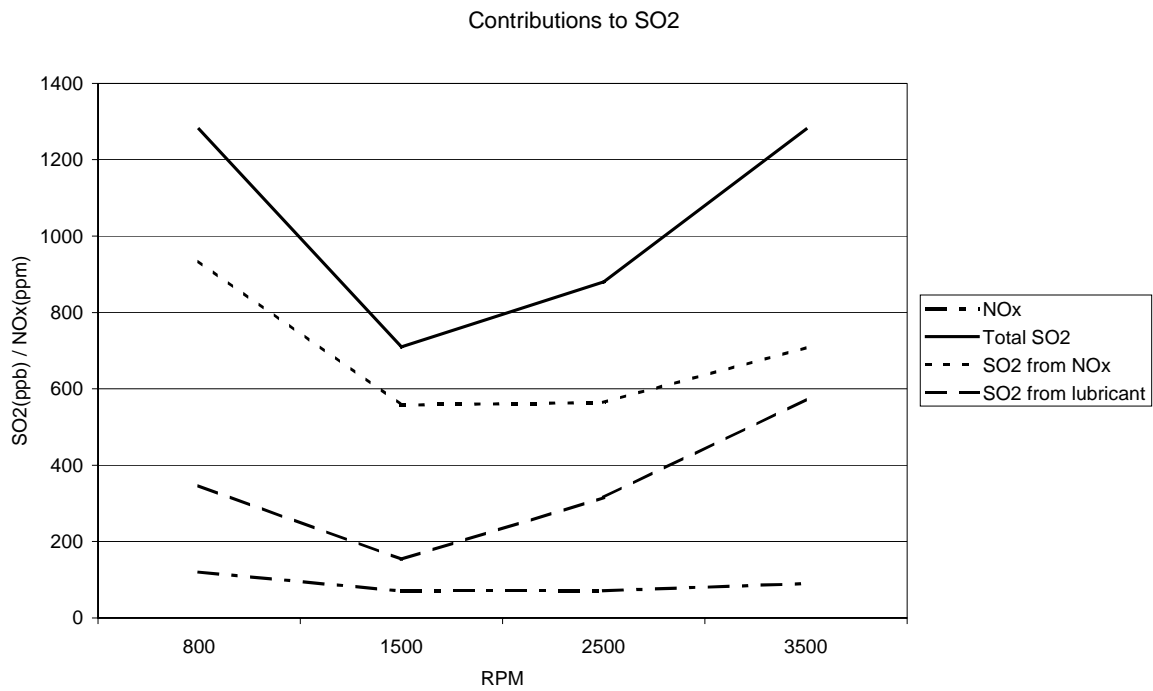


Figure 10.2. Contributions to  $(SO_2)_{meas}$ .

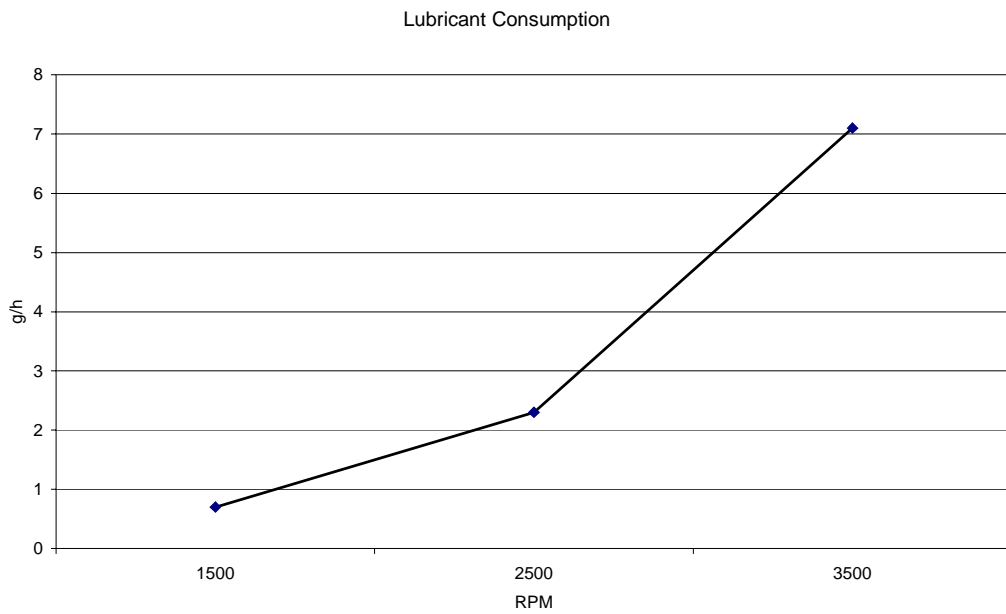


Figure 10.3. Lubricant consumption for reference lubricant and LSD fuel at idle condition.

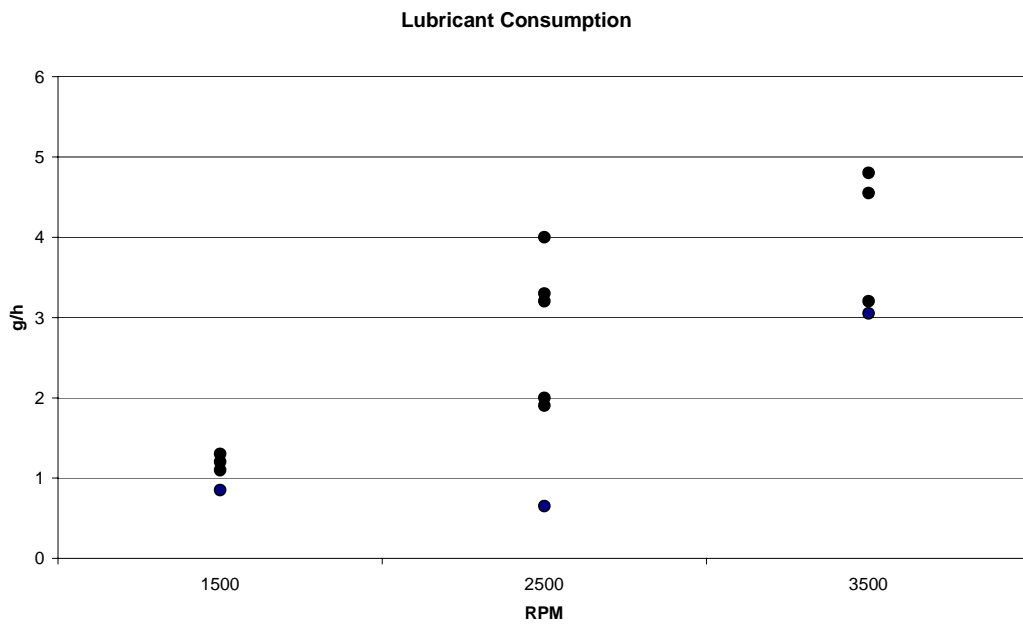


Figure 10.4. Lubricant consumption for reference lubricant and LSD fuel running the engine in 1<sup>st</sup> gear for different engine speeds.

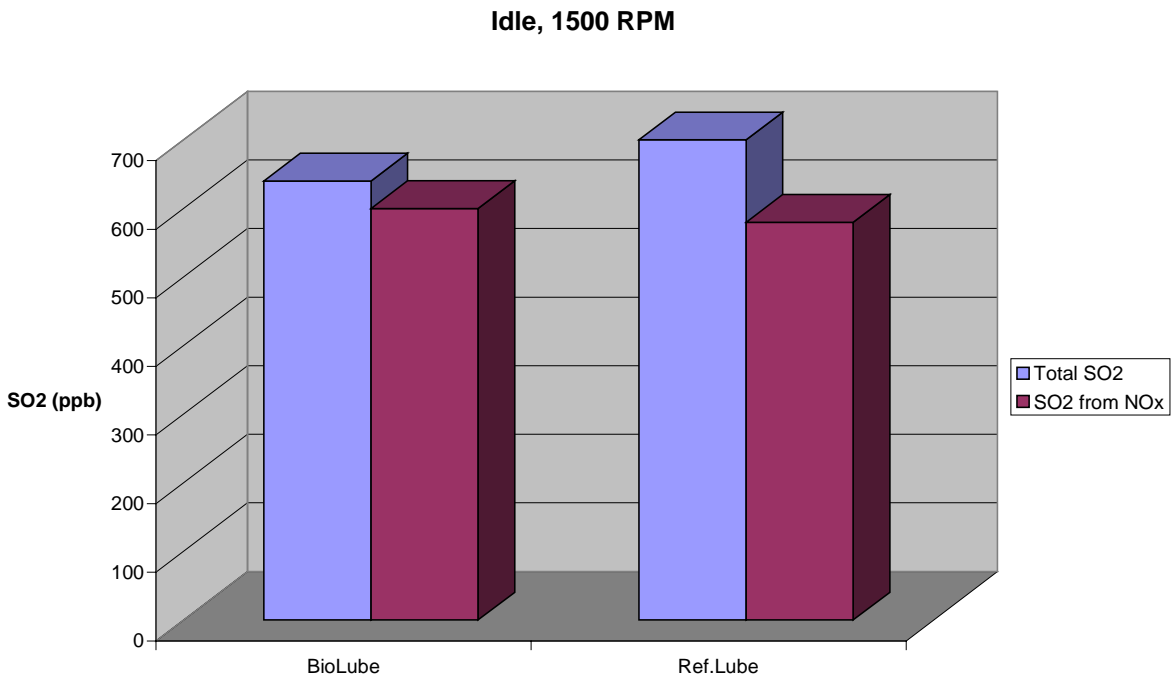


Figure 10.5. SO2 measured and NOx interference at idle and 1500 rpm.

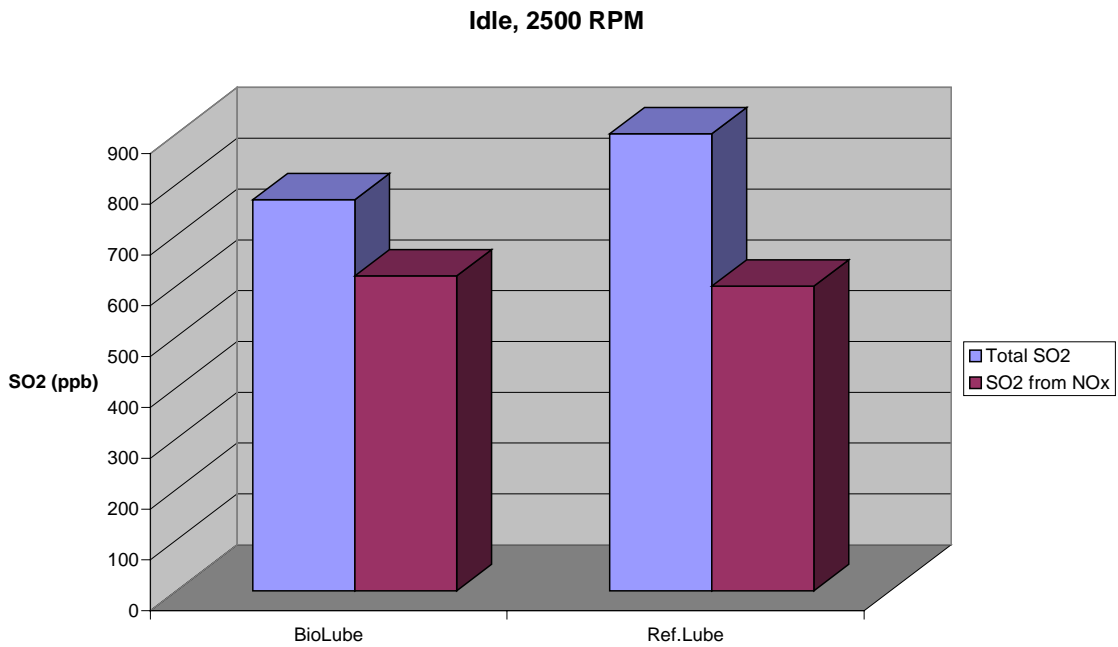
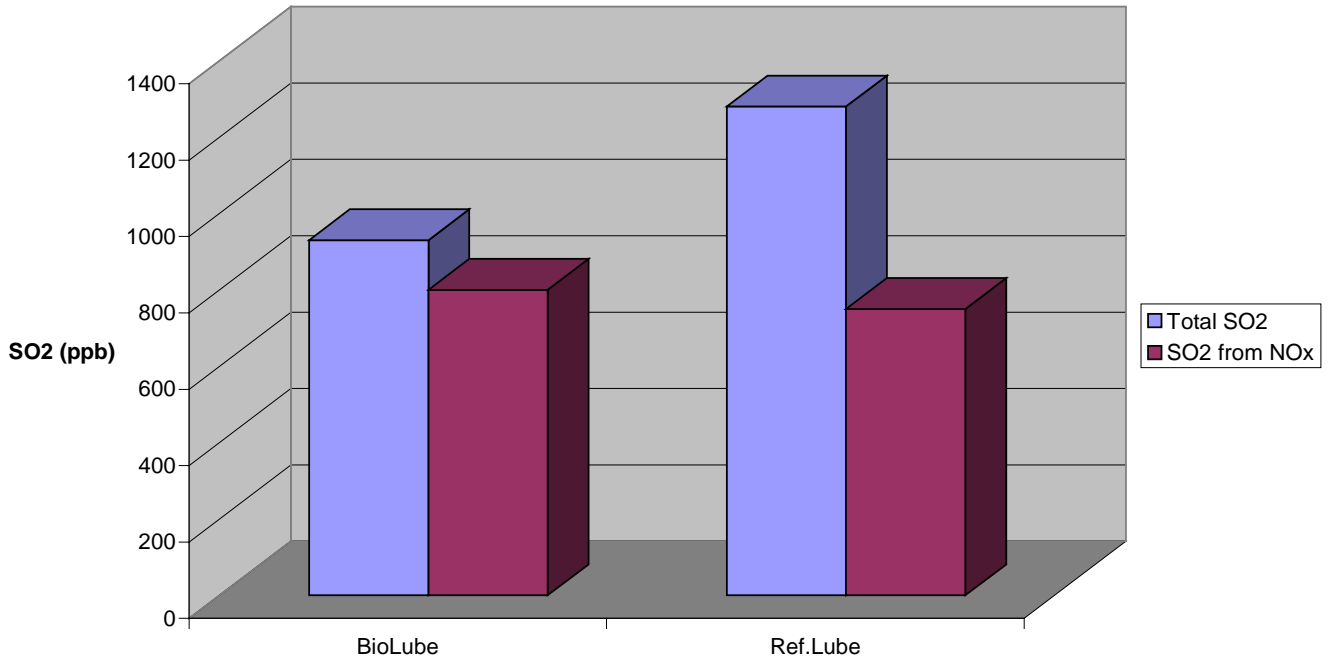


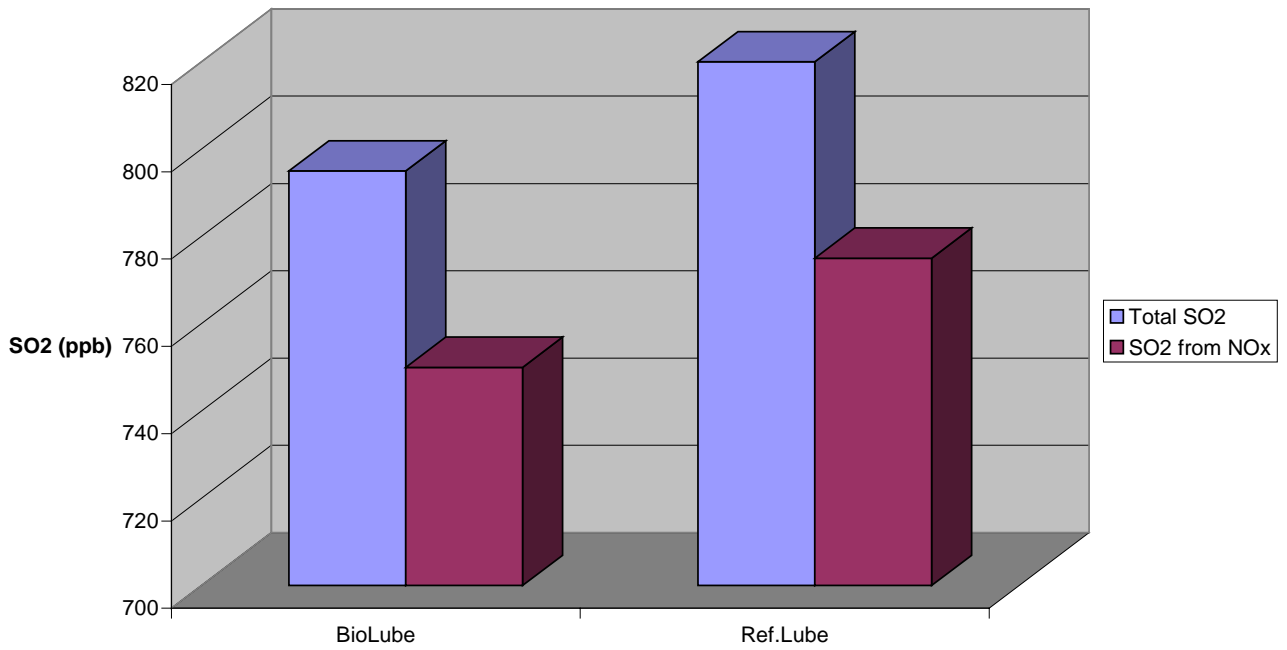
Figure 10.6. SO2 measured and NOx interference at idle and 2500 rpm.

**Idle, 3500 RPM**



*Figure 10.7. SO2 measured and NOx interference at idle and 3500 rpm.*

**1st gear, 2500 RPM**



*Figure 10.8. SO2 measured and NOx interference in 1<sup>st</sup> gear and 2500 rpm*



### 2nd Gear, 2500 RPM

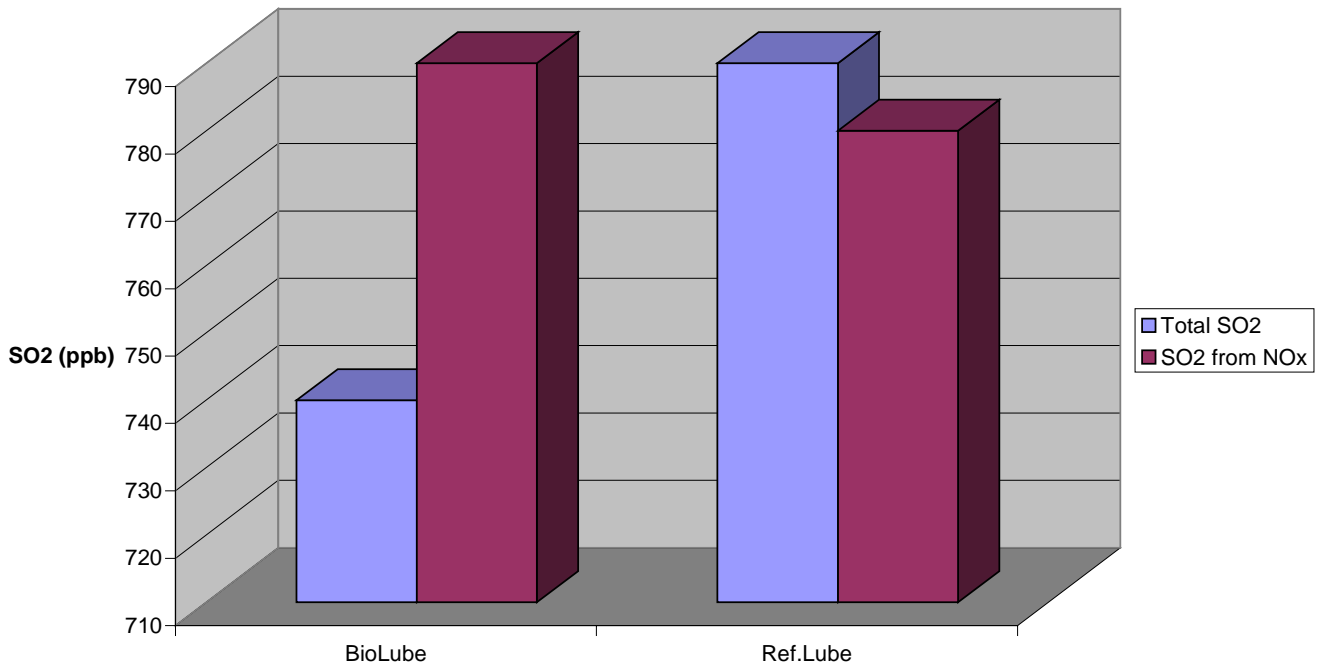


Figure 10.9. SO2 measured and NOx interference in 2<sup>nd</sup> gear and 2500 rpm.

### 3rd Gear, 3500 RPM

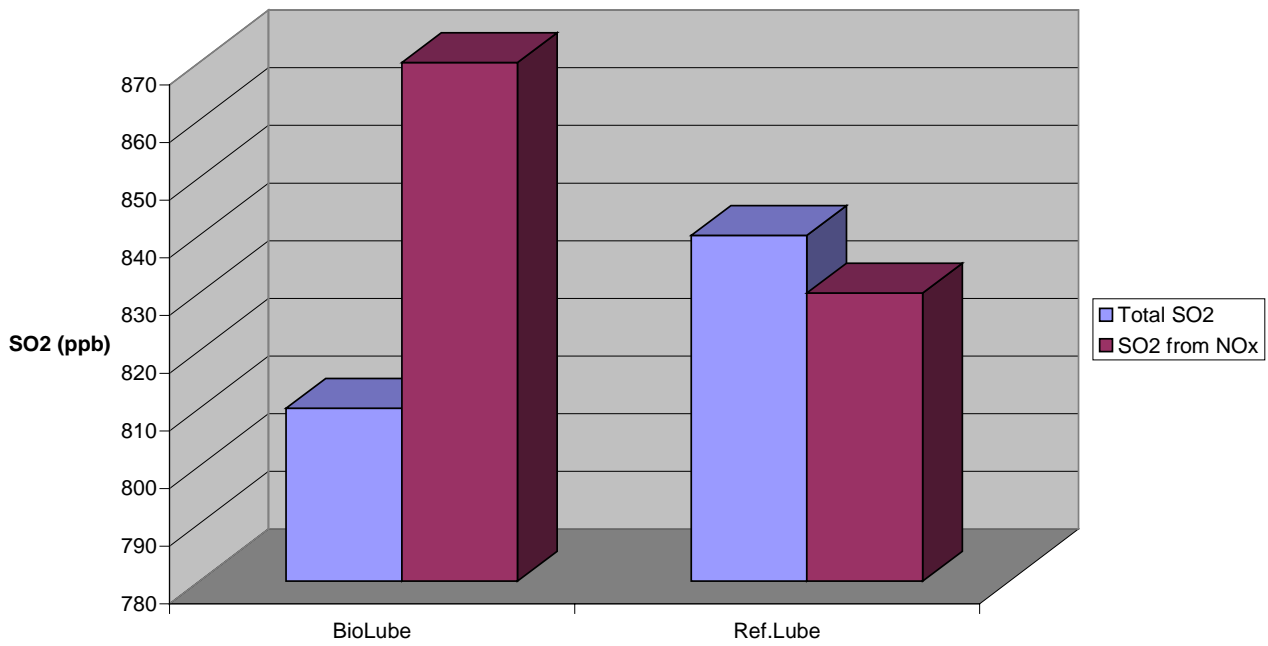


Figure 10.10. SO2 measured and NOx interference in 3<sup>rd</sup> gear and 2500 rpm.

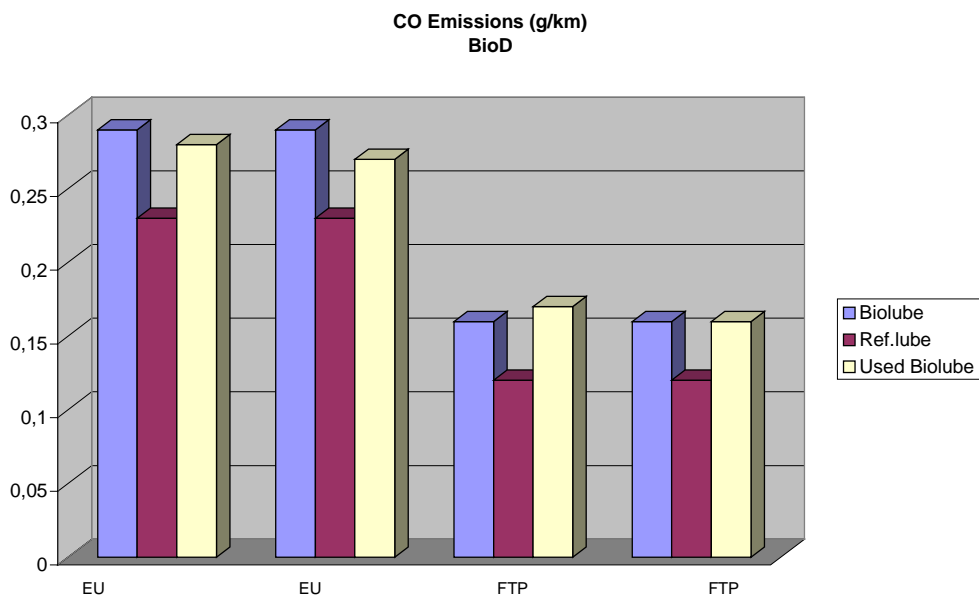
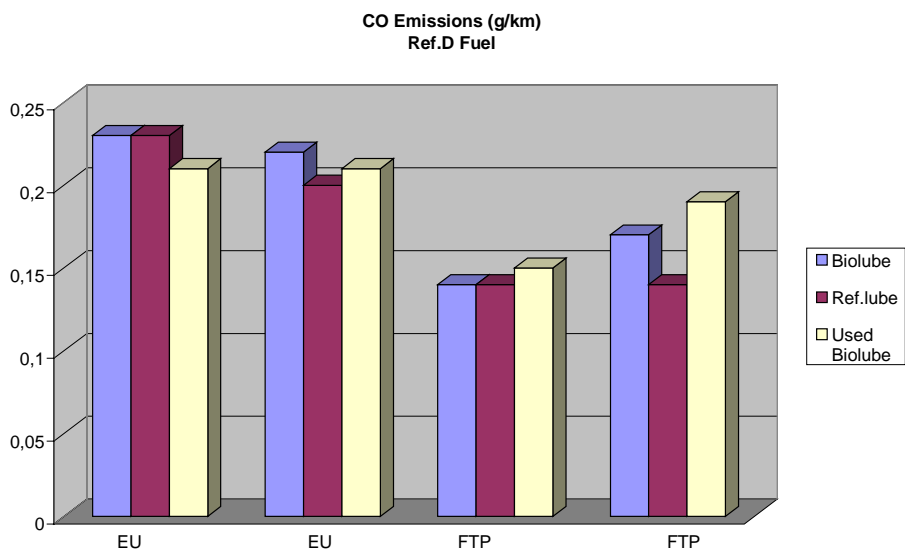
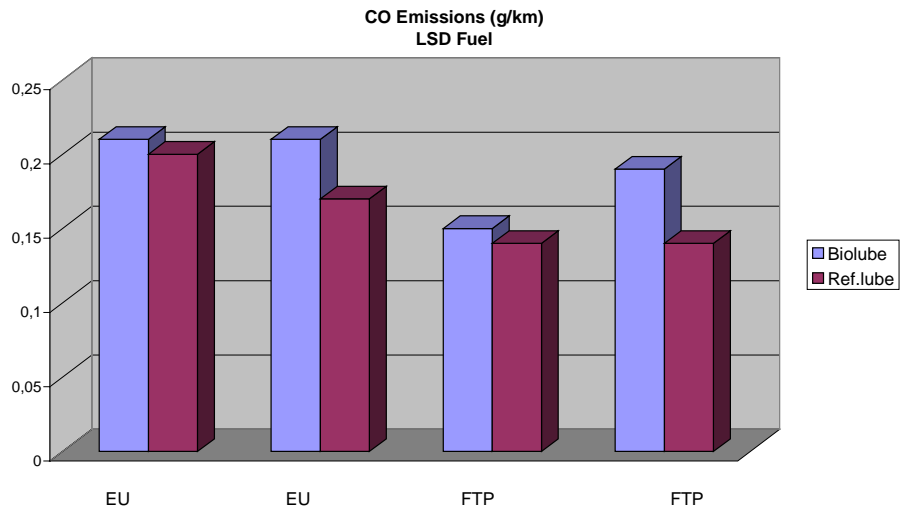


Figure 10.11. CO emissions from the diesel vehicle.

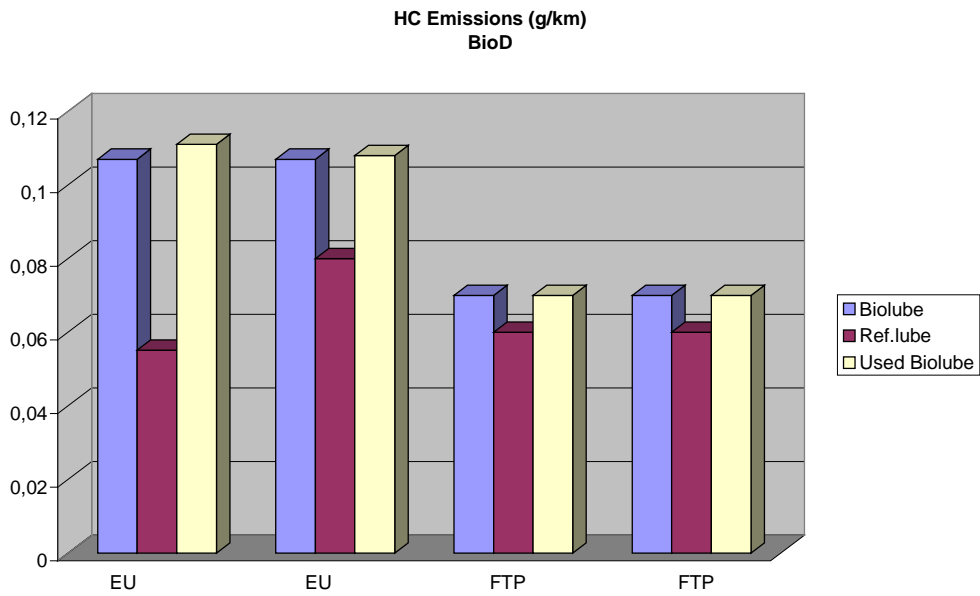
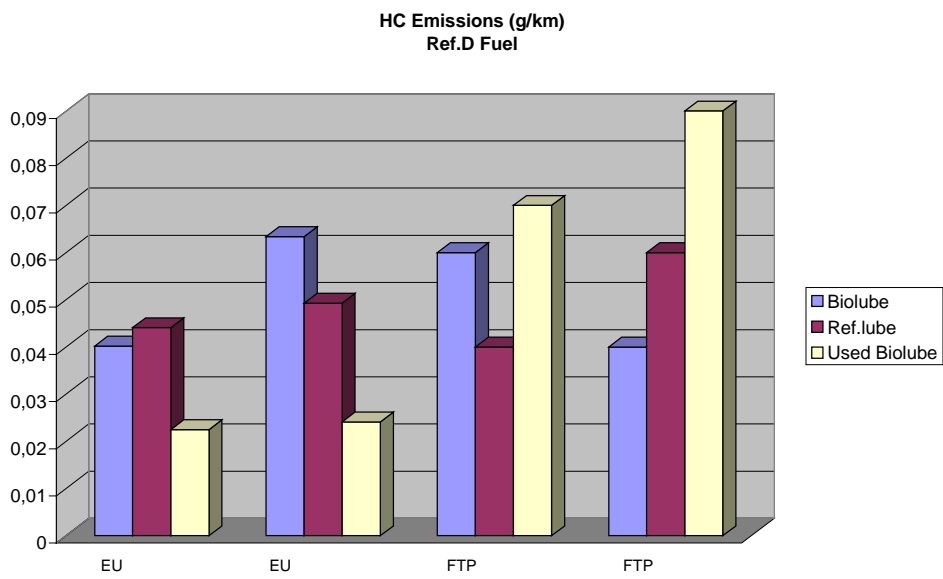
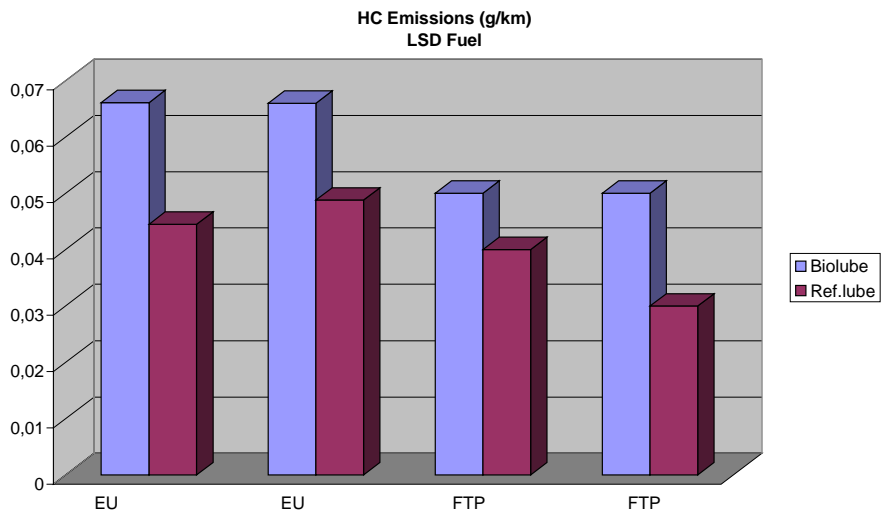


Figure 10.12. HC emissions from the diesel vehicle.

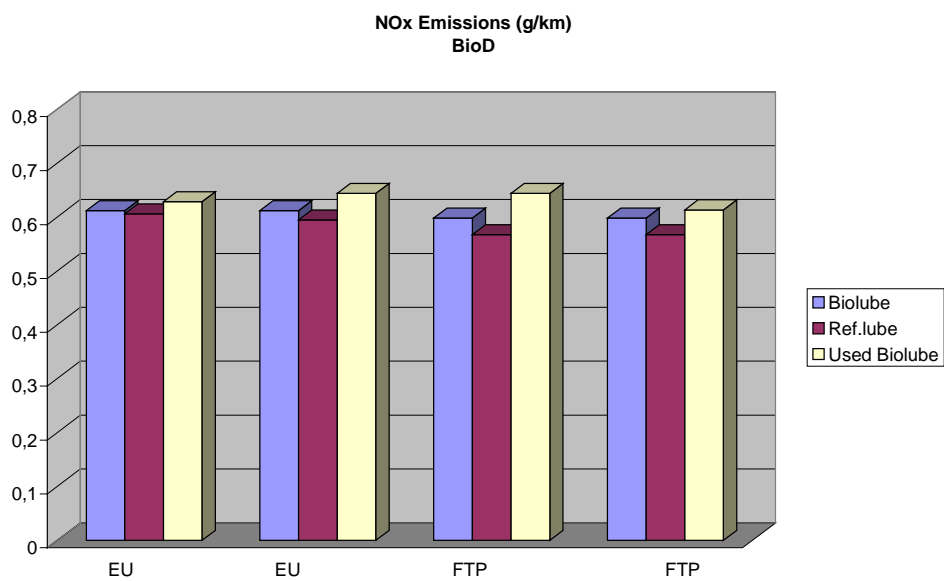
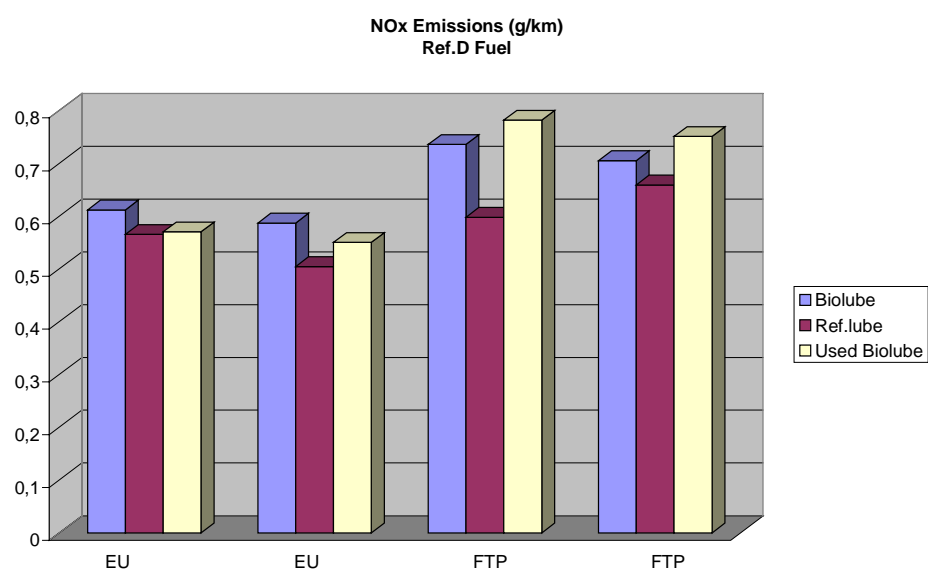
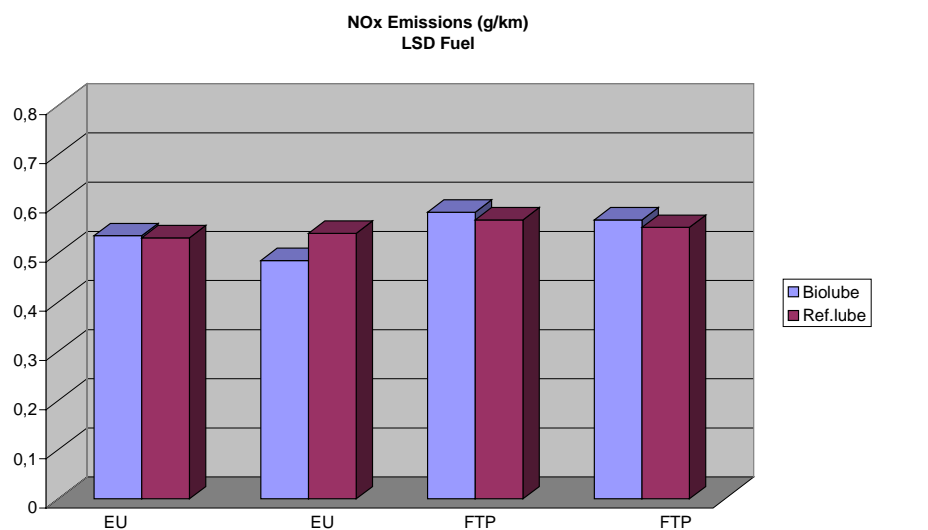


Figure 10.13. NOx emissions from the diesel vehicle.

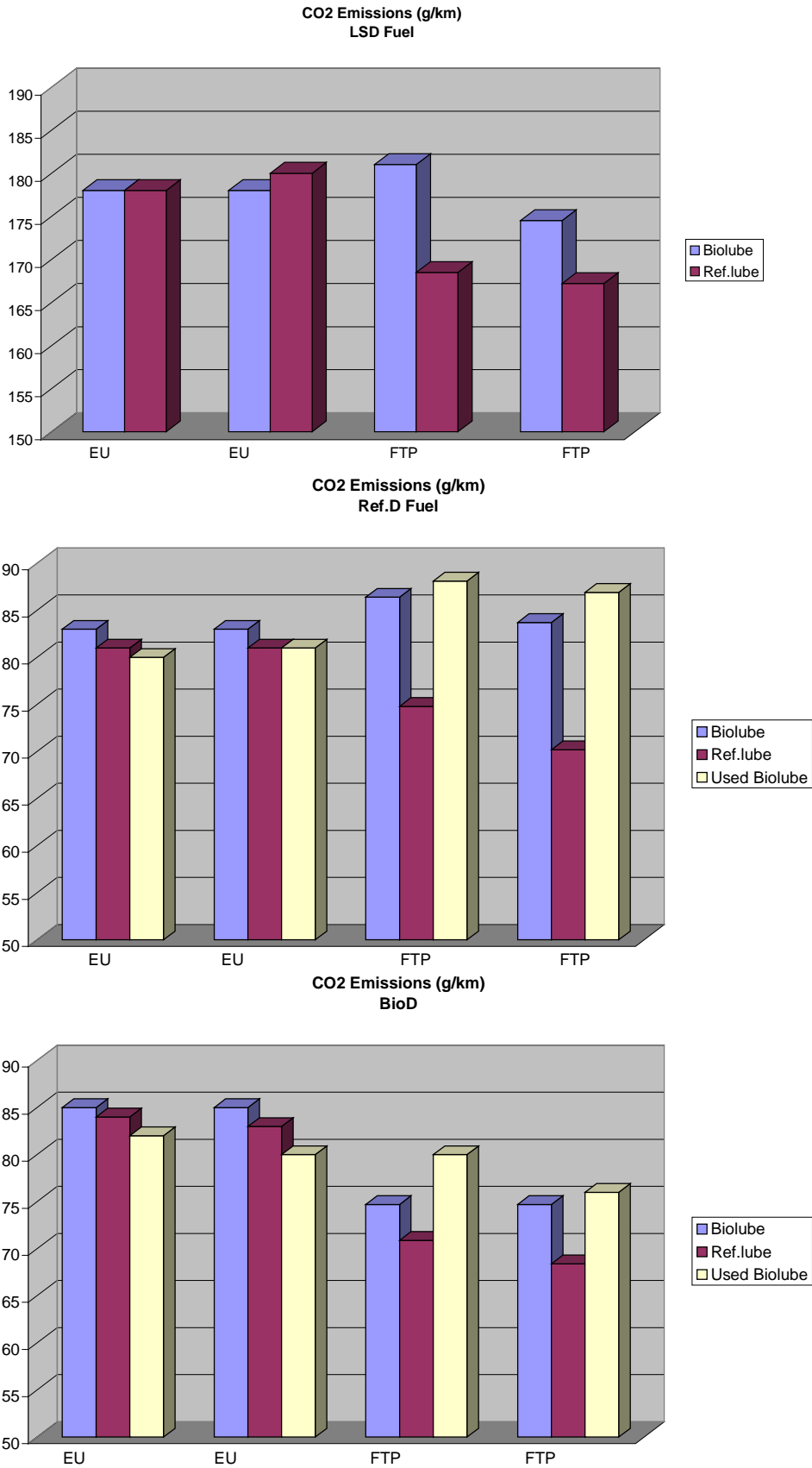


Figure 10.14. CO<sub>2</sub> emissions from the diesel vehicle.

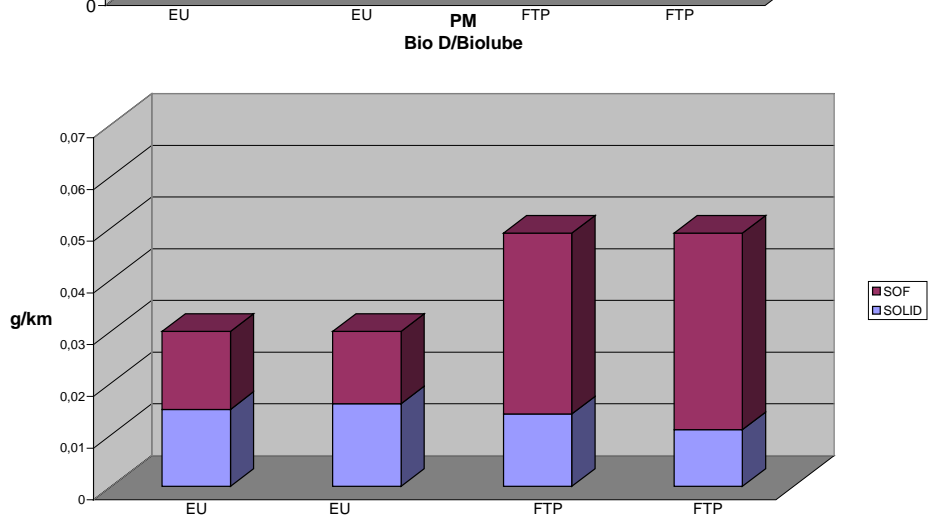
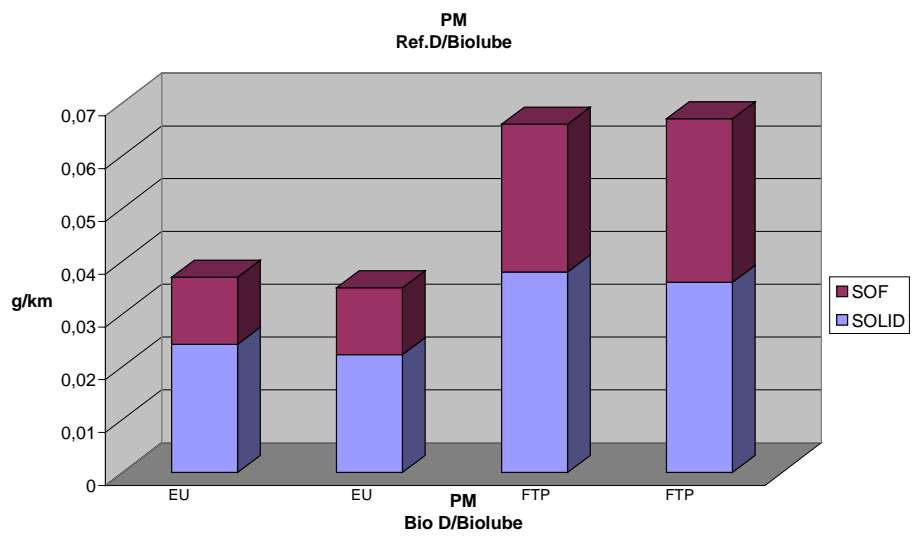
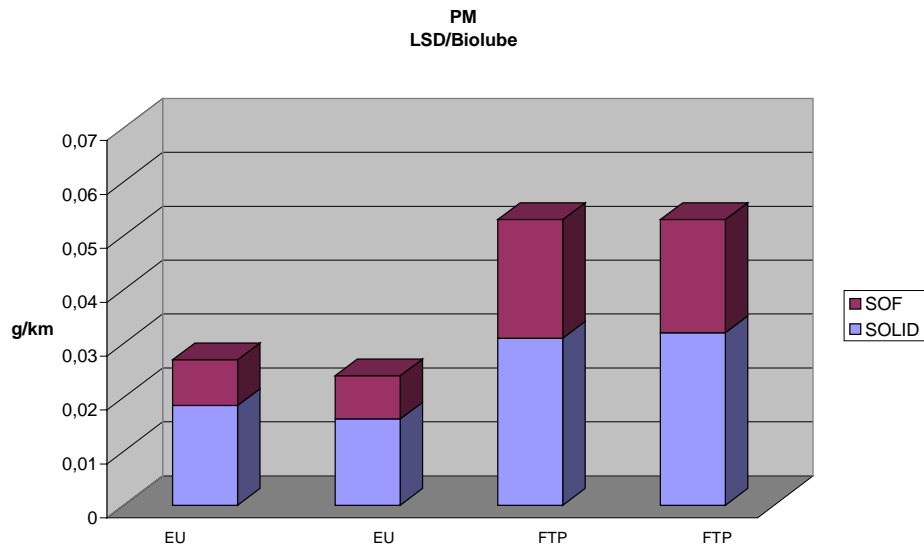


Figure 10.15. Particulate emissions from the diesel vehicle using biodegradable lubricant.

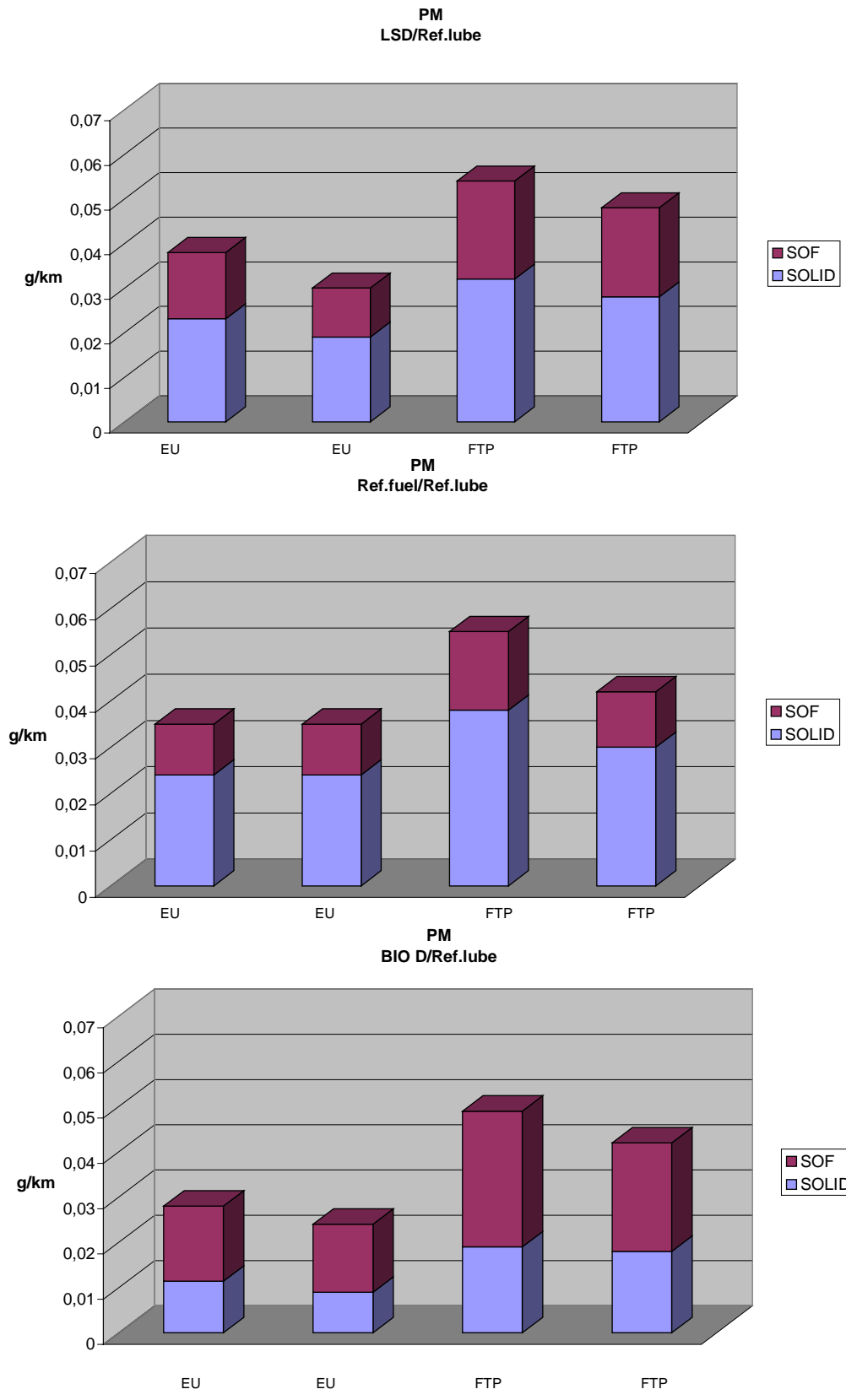


Figure 10.16. Particulate emissions from the diesel vehicle using reference lubricant.

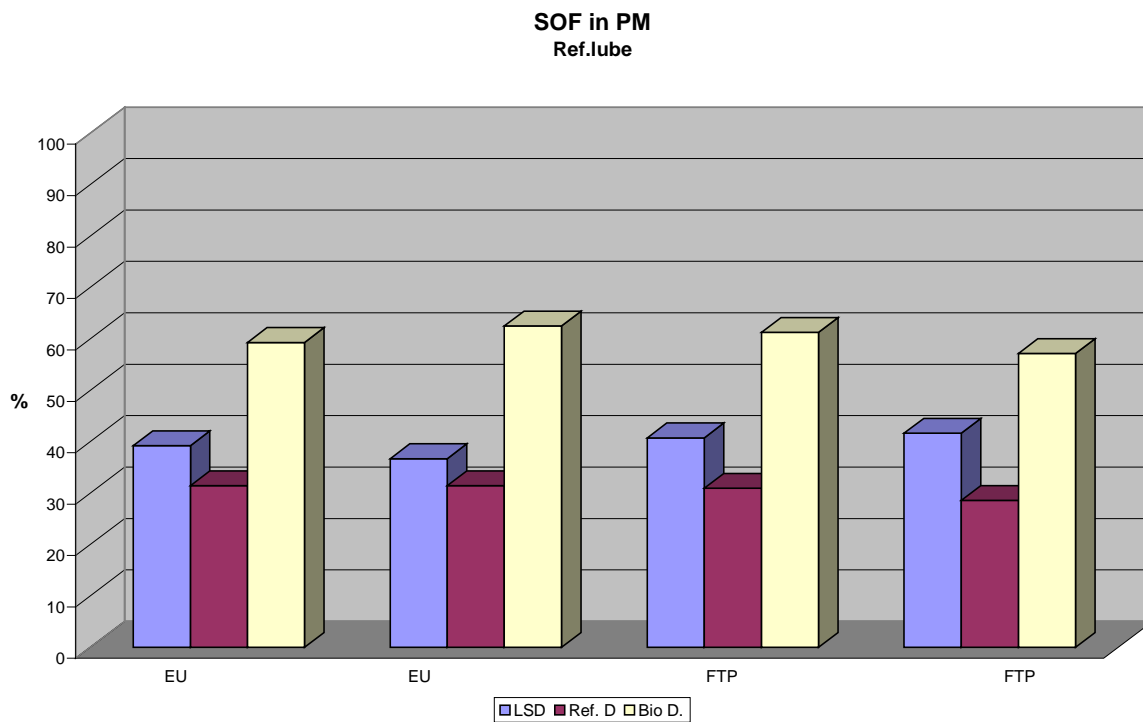
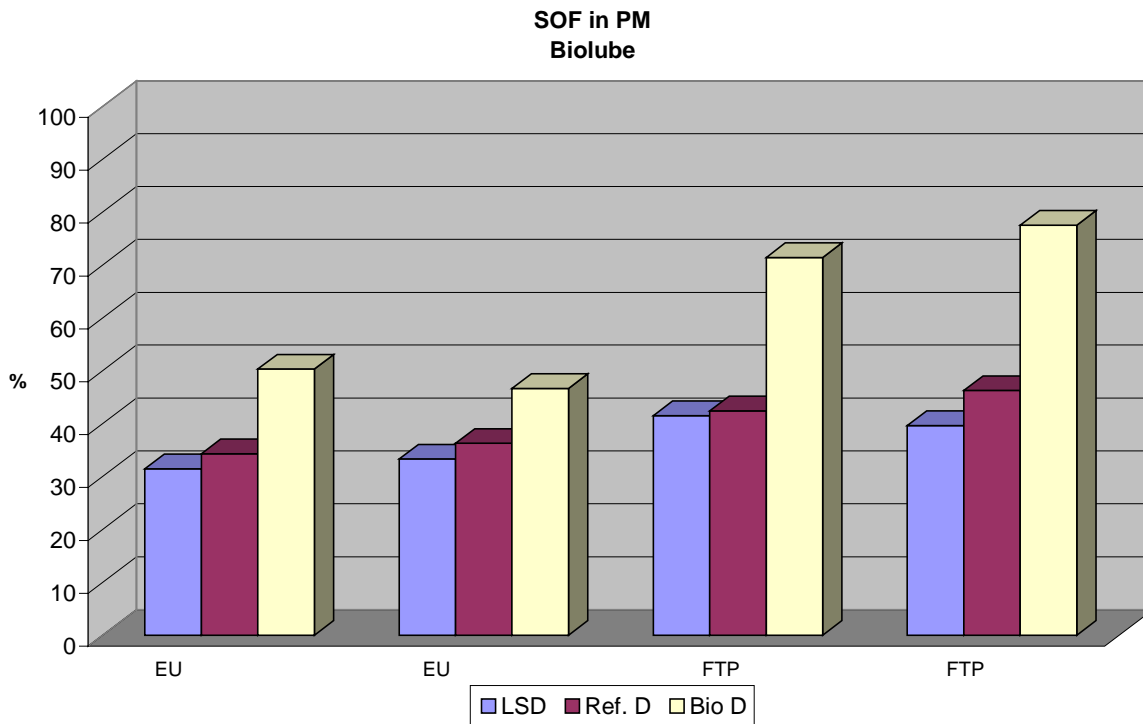


Figure 10.17. SOF contribution to total particulate matter in percent.



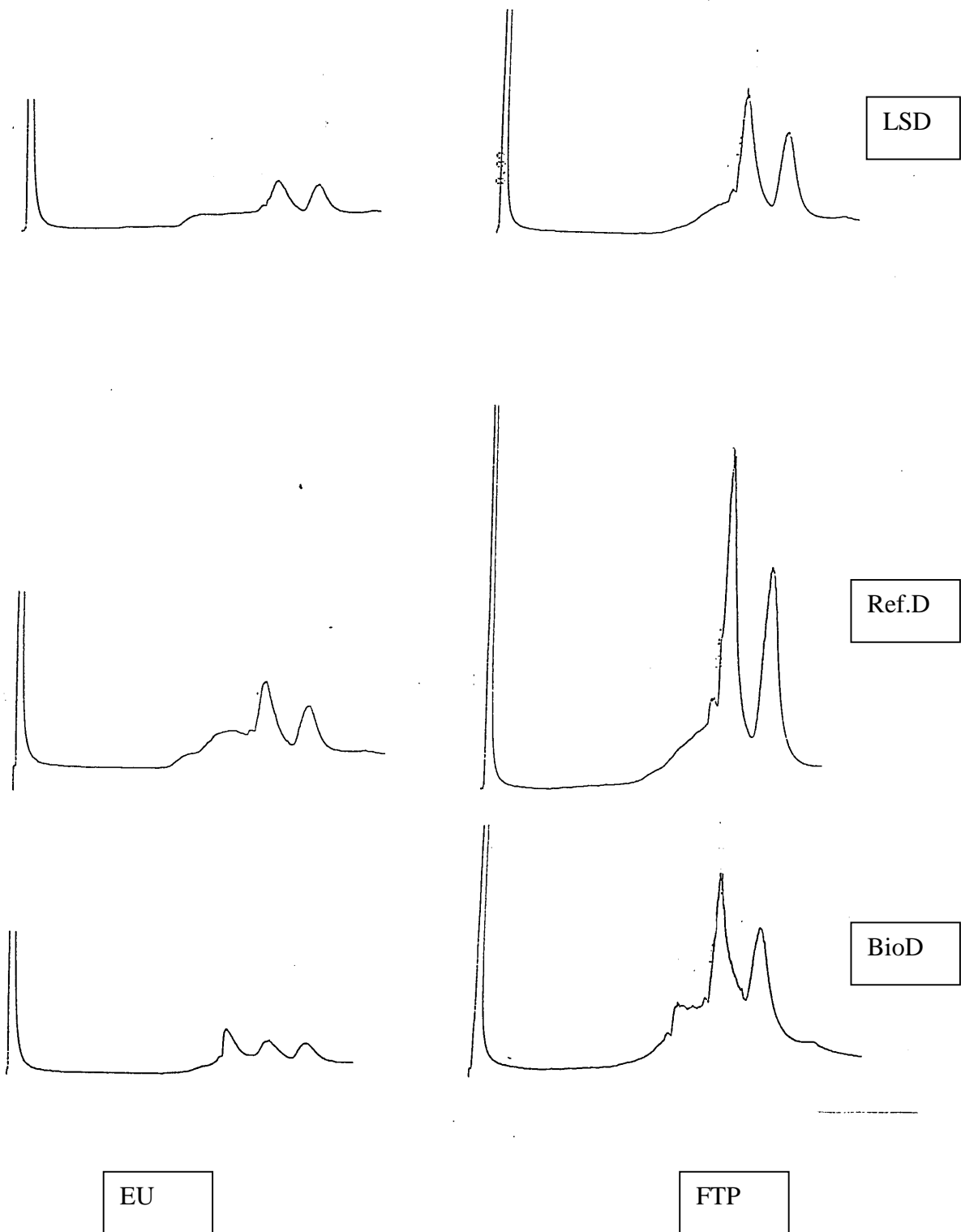


Figure 10.18. Chromatograms of SOF from tests with biodegradable lubricant.

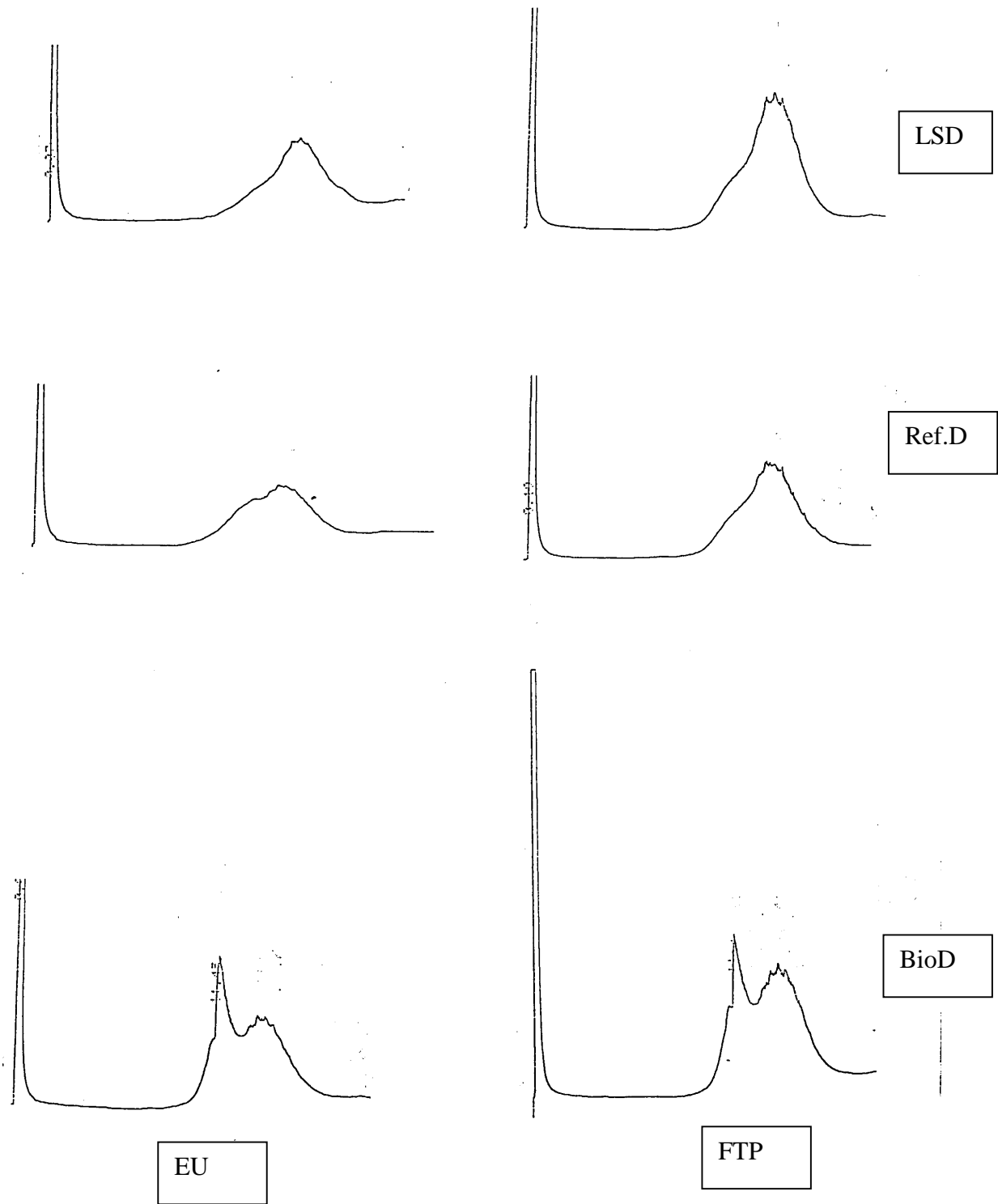


Figure 10.19. Chromatograms of SOF from tests with reference lubricant.

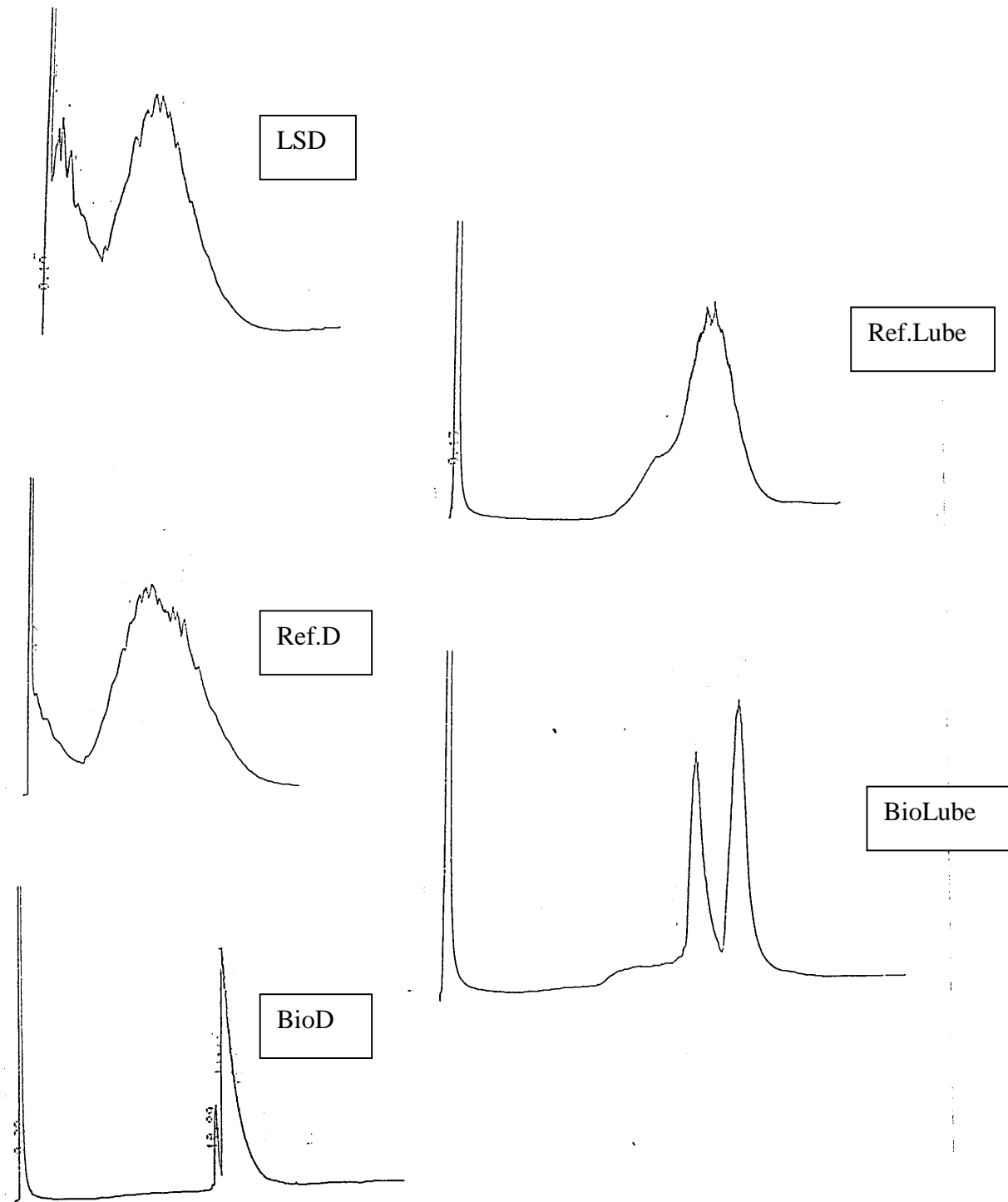


Figure 10.20. Chromatograms of fuels and lubricants.

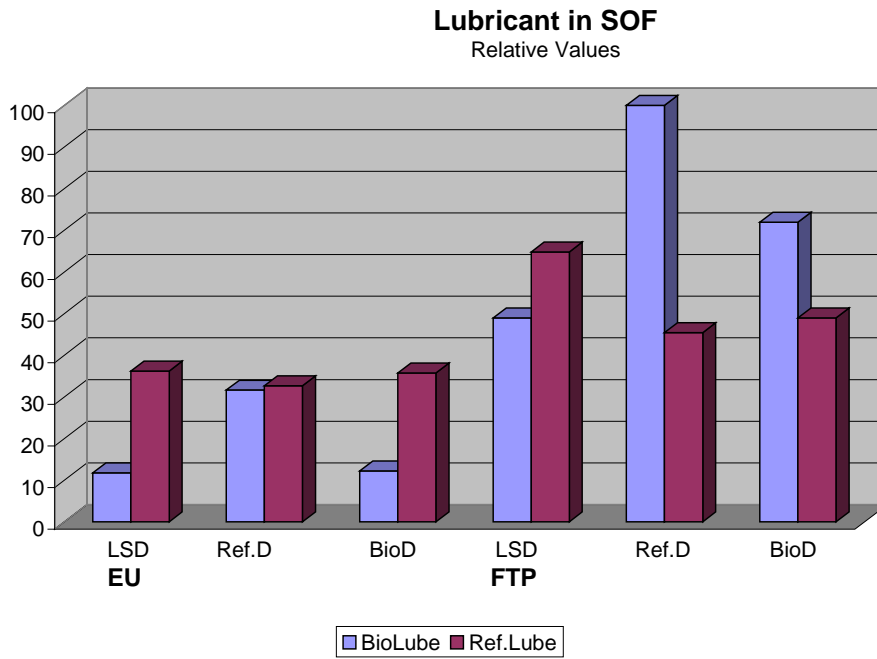


Figure 10.21. Lubricant in SOF estimated amount from chromatograms.

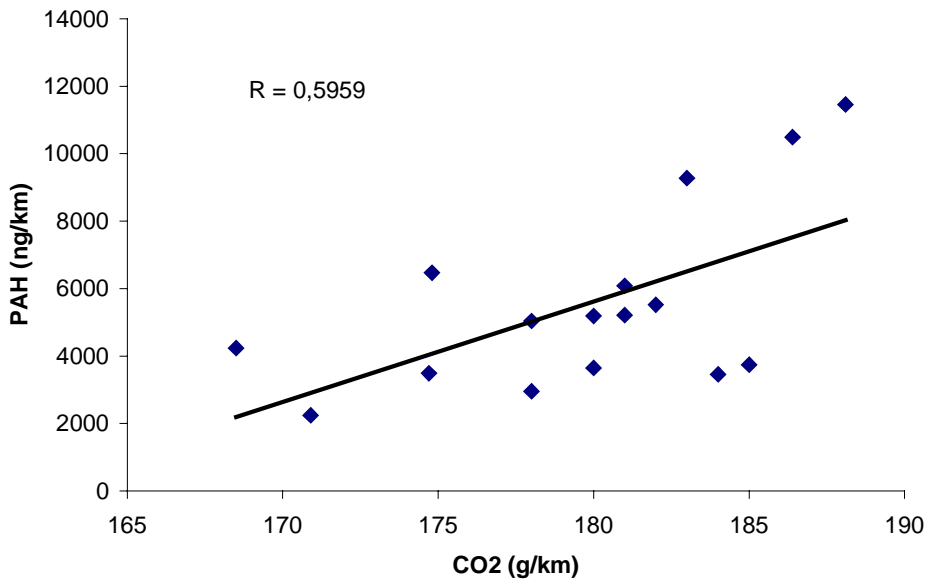


Figure 10.22. PAH emissions vs. CO<sub>2</sub> emissions.

## 11. Conclusions.

The application of a biodegradable lubricant of synthetic ester type to common diesel vehicle technology did not result in unusual wear in the engine. This was verified in long term driving tests on the road with a standard diesel fuel and a biodiesel fuel.

The biodegradability of the fresh lubricant was 90% according to the CEC L-33A-93 test. The results showed that the biodegradability decreases as the lubricant is used. The average biodegradability of the lubricant in use was respectively 76% with the standard diesel fuel and 85% with the biodiesel fuel. The reason for decreased biodegradability is suggested to be due to oxidation of the lubricant and thermal and hydrolytic reactions, furthermore accumulation of dust, combustion products, wear particles and especially heavy metals might also inhibit biodegradation.

The results showed as expected that the application of biodiesel resulted in higher biodegradability compared to standard diesel.

Lubricant consumption measurements showed that application of the biodegradable lubricant resulted in a higher lubricant consumption, compared to a mineral oil reference lubricant, during more transient driving, like the FTP driving pattern, but a lower lubricant consumption for more steady state driving patterns like the EU driving pattern.

This also had the effect that the SOF (Soluble Organic Fraction of particulate matter) emissions were higher during FTP driving with biodegradable lubricant and lower with EU driving. This is because there is a connection between lubricant consumption and lubricant contribution to SOF emissions. The effect is very clearly recognized because the lubricant was found to be the major contributor to SOF emissions.

GC measurements showed that of the fuels it was only biodiesel that contributed to the SOF emissions. The lubricant in every case clearly contributed to SOF emissions. The reason why biodiesel contributed to SOF was suggested to be due to the higher carbon number of biodiesel hydrocarbons, resulting in an increased tendency to fuel condensation in the exhaust.

Biodiesel was found to result in lower emissions of solid particulate matter.

Reference diesel fuel resulted in a higher lubricant consumption during the road test compared to biodiesel. This also resulted in higher SOF emissions. This could be an effect of the higher fuel consumption with reference diesel, which could result in more fuel/lubricant interaction on the cylinder liner.

Application of the biodegradable lubricant generally resulted in a few percent higher fuel consumption. This was followed by a similar increase in NO<sub>x</sub> emissions due to higher combustion temperatures because of increased friction loss.

The higher fuel consumption also resulted in a higher emission of PAH (Polycyclic Aromatic Hydrocarbons) with the biodegradable lubricant. The PAH emissions were found to be very fuel related. The standard diesel fuel had a much higher PAH content than the biodiesel and the LSD

(Low Sulfur Diesel) and biodiesel had the lowest PAH content. Thus standard diesel resulted in the highest PAH emissions.

There was seen no connection between lubricant PAH content and PAH emissions because the effect of the lubricant on the fuel consumption drowned any possible effect.

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## **Abbreviations**

CO	carbon monoxide
CO <sub>2</sub>	carbon dioxide
EU	European test driving cycle
FTP	USA test driving cycle (Federal Test Procedure)
GC	gas chromatography
HPLC	high performance liquid chromatography
LSD	low sulphur diesel
NO <sub>x</sub>	nitrogen oxides
PAH	polycyclic aromatic hydrocarbons
PM	particulate matter
THC	total hydrocarbons
SOF	soluble organic fraction
SOLID	the fraction of PM that is not SOF

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## Appendix 1. Data Tables.

CO	(g/km)		
0 km			
	biolube	ref.lube	
city D	0,2	0,2	EU
	0,2	0,17	EU
	0,14	0,14	FTP
	0,16	0,14	FTP
ref D	0,23	0,23	EU
	0,22	0,2	EU
	0,14	0,14	FTP
	0,17	0,14	FTP
bio D	0,29	0,23	EU
	0,29	0,23	EU
	0,16	0,12	FTP
	0,16	0,12	FTP
7500 km ref D			
	0,21		EU
	0,21		EU
	0,15		FTP
	0,19		FTP
7500 km bio D			
	0,28		EU
	0,27		EU
	0,17		FTP
	0,16		FTP



HC		(g/km)	
0 km			
city D	biolube	ref.lube	
	0,0661	0,0445	EU
	0,066	0,0488	EU
	0,05	0,04	FTP
	0,05	0,03	FTP
ref D	0,0402	0,0441	EU
	0,0634	0,0493	EU
	0,06	0,04	FTP
	0,04	0,06	FTP
bio D	0,107	0,0551	EU
	0,107	0,08	EU
	0,07	0,06	FTP
	0,07	0,06	FTP
7500 km ref D			
	0,0225		EU
	0,0241		EU
	0,07		FTP
	0,09		FTP
7500 km bio D			
	0,111		EU
	0,108		EU
	0,07		FTP
	0,07		FTP

NOx	(g/km)		
0 km			
	biolube	ref.lube	
city D	0,535133	0,530533	EU
	0,484533	0,539733	EU
	0,582667	0,567333	FTP
	0,567333	0,552	FTP
ref D	0,6118	0,5658	EU
	0,587267	0,504467	EU
	0,736	0,598	FTP
	0,705333	0,659333	FTP
bio D	0,6118	0,605667	EU
	0,6118	0,594933	EU
	0,598	0,567333	FTP
	0,598	0,567333	FTP
7500 km ref D			
	0,5704		EU
	0,550467		EU
	0,782		FTP
	0,751333		FTP
7500 km bio D			
	0,628667		EU
	0,644		EU
	0,644		FTP
	0,613333		FTP

CO2		(g/km)	
0 km			
	biolube	ref.lube	
city D	178	178	EU
	178	180	EU
	181	168,5	FTP
	174,5	167,2	FTP
ref D	183	181	EU
	183	181	EU
	186,4	174,8	FTP
	183,7	170,2	FTP
bio D	185	184	EU
	185	183	EU
	174,7	170,9	FTP
	174,7	168,4	FTP
7500 km ref D			
	180		EU
	181		EU
	188,1		FTP
	186,9		FTP
7500 km bio D			
	182		EU
	180		EU
	180		FTP
	176		FTP

PM	(g/km)		
0 km			
	biolube	ref.lube	
city D	0,027	0,038	EU
	0,024	0,03	EU
	0,053	0,054	FTP
	0,053	0,048	FTP
ref D	0,037	0,035	EU
	0,035	0,035	EU
	0,066	0,055	FTP
	0,067	0,042	FTP
bio D	0,03	0,028	EU
	0,03	0,024	EU
	0,049	0,049	FTP
	0,049	0,042	FTP
7500 km ref D			
	0,035		EU
	0,037		EU
	0,081		FTP
	0,07		FTP
7500 km bio D			
	0,029		EU
	0,03		EU
	0,051		FTP
	0,049		FTP

SOF	(g/km)		
0 km			
	biolube	ref.lube	
city D	0,0085	0,0149	EU
	0,008	0,011	EU
	0,022	0,022	FTP
	0,021	0,02	FTP
ref D	0,0127	0,011	EU
	0,0127	0,011	EU
	0,028	0,017	FTP
	0,031	0,012	FTP
bio D	0,0151	0,0166	EU
	0,014	0,015	EU
	0,035	0,03	FTP
	0,038	0,024	FTP
7500 km ref D			
	0,0048		EU
	0,0051		EU
	0,027		FTP
	0,023		FTP
7500 km bio D			
	0,00897		EU
	0,00928		EU
	0,0113		FTP
	0,0109		FTP

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### Absolute PAH Emissions

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(ng/km) PAH	BioLube		Ref.Lube		BioLube - 7500km	
>2 rings	EU	FTP	EU	FTP	EU	FTP
LSD	2957	5215	5043	4236		
Ref.D	9277	10490	6083	6471	3649	11460
BioD	3741	3489	3460	2246	5524	5190

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