

**Impact of FAME on the performance of three Euro 4 light-duty diesel vehicles.
Part 2: Unregulated emissions**





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Prepared for the Concaawe Fuels and Emissions Management Group by its Special Task Force FE/STF-25:

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ABSTRACT

In 2009, EU legislation mandated that 10% renewable energy must be used in road transportation by 2020, primarily through the use of bio-blending components. For diesel fuel blending, Fatty Acid Methyl Esters (FAMES) manufactured from different natural oils are the most likely to be used in significant volumes over this time period. FAME products have been used in Europe for many years, both as blends and as neat fuels in some niche markets.

Concaawe has completed a vehicle study in which one hydrocarbon-only diesel fuel and three blends of this fuel with FAME (from a single batch of Rapeseed Methyl Ester (RME)) were tested in three Euro 4 light-duty passenger cars. All vehicles used exhaust gas recirculation and were equipped with some type of diesel oxidation catalyst. One vehicle did not have a Diesel Particulate Filter (DPF) while the other two vehicles were equipped with DPFs that were regenerated using two different approaches. The FAME contents of the test fuels varied from 0% to 50% v/v in order to accentuate the effect of RME on the energy content of the blended fuels and on the emissions from the vehicles. The study was statistically designed and completed using a robust and repeatable testing schedule during which fuel consumption and emissions data were collected over different regulatory and transient driving cycles. These cycles included the New European Driving Cycle (NEDC), the ARTEMIS cycle, and two constant speed conditions. The effect of RME on fuel consumption was reviewed in Part 1 of this study.

In addition to the gaseous tailpipe emissions, particulate matter (PM) and particle number (PN) emissions were measured using the Particle Measurement Programme (PMP) regulatory procedure. Total PM, total PN, and solid PN emissions were measured as a function of RME content. In addition, the PM samples were chemically analysed for their soluble organic fraction, the fuel and lube contributions to this organic fraction, ionic species (nitrates and sulphates), and elemental carbon (by difference). Several carbonyl compounds were also collected from the tailpipe exhaust and characterized.

KEYWORDS

Exhaust emissions, diesel fuel, engine technology, vehicles, fuel quality, Euro 4, unregulated emissions, SOF, VOF, carbonyl, PM, PN, FAME, RME

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CONTENTS		Page
1.	INTRODUCTION	1
2.	EXPERIMENTAL	3
2.1.	TEST VEHICLES	3
2.2.	TEST FUELS	3
3.	METHODOLOGY	4
3.1.	TEST PROTOCOL	4
3.2.	DATA ANALYSIS	5
3.3.	PM ANALYSIS PROCEDURES	5
3.3.1.	Determination and breakdown of soluble organic fraction	5
3.3.2.	Anionic species	7
3.3.3.	Carbonyl compounds in the vehicle exhaust	7
3.4.	PN SAMPLING PROTOCOL	7
3.5.	STATISTICAL ANALYSIS	8
4.	RESULTS AND DISCUSSION	10
4.1.	PARTICLE EMISSIONS	10
4.1.1.	PN Emissions	10
4.1.2.	Particle size distribution	12
4.1.3.	PM composition	14
4.2.	CARBONYL COMPOUNDS	15
5.	CONCLUSIONS	18
6.	GLOSSARY	20
7.	ACKNOWLEDGEMENTS	23
8.	REFERENCES	24
APPENDIX 1	DIESEL VEHICLES	27
APPENDIX 2	TEST FUELS	28
APPENDIX 3	FLOWCHART FOR VEHICLE TEST PROTOCOL	29
APPENDIX 4	SAMPLING PROTOCOL	30
APPENDIX 5	PM ANALYSIS PROCEDURES	34
APPENDIX 6	STATISTICAL DATA ANALYSIS	36
APPENDIX 7	AVERAGE PM AND PN (TOTAL AND SOLID) EMISSIONS	39
APPENDIX 8	CARBONYL COMPOUNDS	41
APPENDIX 9	PARTICULATES: MEANS AND STANDARD DEVIATIONS	43
APPENDIX 10	PHOTOS OF PARTICULATE FILTERS	46

SUMMARY

The use of bio-components as alternative energy sources has been considered for many years around the world. Within the European Union, more use of energy from renewable sources, especially through the use of bio-derived blending components in transport fuels, has been mandated by the Renewable Energy Directive (RED, 2009/28/EC [1]) while reductions in greenhouse gas (GHG) emissions have also been mandated for fuels (Fuel Quality Directive (FQD, 2009/30/EC [2]).

Of special interest from a fuel demand, air quality, and human health perspective is the potential impact that these changes may have on vehicle fuel consumption and both regulated and unregulated emissions. To contribute data to this area, Concaawe has completed a vehicle study on biodiesel fuels to improve our understanding of performance issues on light-duty diesel vehicles that are of importance to the European marketplace.

Three Euro 4 light-duty diesel passenger cars were tested to evaluate the effect of Fatty Acid Methyl Ester (FAME) in diesel fuel on fuel consumption and emissions. These vehicles were equipped with common rail turbocharged engines and used different exhaust aftertreatment technologies to achieve the Euro 4 emissions requirements. Two of the vehicles were equipped with Diesel Particulate Filters (DPFs) and one was not. In the two vehicles equipped with DPFs, two different schemes were used to regenerate the particulate filters. All test work was carried out in facilities located in the Laboratory of Applied Thermodynamics (LAT) of the Aristotle University of Thessaloniki, Greece.

Four fuels were evaluated in this study. One base diesel fuel (B0 complying with the EN 590 specification [3]) was blended with commercially sourced Rapeseed Methyl Ester (RME), complying with the EN 14214 specification [4], to give diesel/RME blends of 10% v/v (B10), 30% v/v (B30), and 50% v/v (B50) RME. These concentrations were selected to anticipate future increases in biofuel requirements and to accentuate the impact of RME on fuel consumption and vehicle emissions.

Measurements were performed according to the New European Driving Cycle (NEDC), which is the European regulatory test procedure. Additionally, the ARTEMIS transient cycle was used to simulate more 'real world' driving operation and two constant speed conditions at 50 and 120 km/h were also evaluated. All fuel/vehicle combinations were tested in a rigorously controlled and statistically designed test programme.

The results on fuel consumption and regulated emissions have been reported previously [8,9]. This report focuses on the unregulated emissions from the same vehicle study including total particulate matter (PM), total particle number (PN), solid PN, PN size distribution, and the emissions of selected carbonyl compounds. In addition, the tailpipe PM emissions were collected on filter papers and analysed for chemical composition, including the fuel and lube contributions to the soluble organic fraction (SOF) of the PM, the nitrate and sulphate anionic species, and the elemental carbon (EC), measured by difference.

Not surprisingly, the absolute PM emissions were more than an order of magnitude higher from the non-DPF-equipped vehicle compared to the two DPF-equipped vehicles. For the vehicle that was not equipped with a DPF, increasing the RME content in the diesel fuel blend reduced the total PM emissions and increased the percentage SOF in the filtered PM sample. The fuel-derived SOF was consistently higher than the lube-derived SOF. In general, the percentage EC of the PM

emissions decreased with increasing RME content while the nitrates slightly increased.

The PN emissions from the two DPF-equipped vehicles were up to four orders of magnitude lower than from the non-DPF-equipped vehicle and were very close to the testing facility's background PN levels. For the DPF-equipped vehicles, the very low emissions made it difficult to draw firm conclusions on the effect on RME on PN emissions. For the non-DPF-equipped vehicle, a clear trend towards lower solid PN emissions with increasing RME content was also observed although this effect was small.

Carbonyl-containing compounds were also trapped from the tailpipe emissions and analysed by gas chromatography (GC). Formaldehyde was the major carbonyl compound observed in the tailpipe emissions, with average values ranging from 0.2 to 1.0 mg/km over the NEDC but there was no clear dependence on the FAME concentration in this study. For two vehicles, one that was equipped with a DPF and one that was not, slightly higher average values (up to 1.3 mg/km) were observed for formaldehyde over the ARTEMIS Urban driving cycle. Much higher average values (up to 6.3 mg/km) were observed for the third vehicle that was also equipped with a DPF. Tailpipe emissions of other measured carbonyl compounds were at or near the detection limit and did not depend strongly on the FAME concentration.

1. INTRODUCTION

The Renewable Energy Directive (RED, 2009/28/EC) [1] requires that 10% renewable energy must be used in road transport fuels by 2020 with simultaneous reductions in GHG emissions to be achieved primarily by blending bio-derived products into fuels (FQD, 2009/30/EC [2]). Changes to European gasoline and diesel fuel specifications are being made to enable these requirements.

In the near term, these requirements will largely be addressed by using ethanol for gasoline blending and Fatty Acid Methyl Esters (FAME) for diesel fuel blending. In the longer term, bio-components from biomass and other sources may become more readily available and will undoubtedly be required in order to achieve the RED ambitions. Although this is likely, the current test programme has focused on FAME bio-components only and alternatives to FAME, such as Hydrogenated Vegetable Oils (HVO) and Biomass to Liquids (BTL), may be considered in later studies.

Today, FAME is most frequently used in low level blends with diesel fuel from crude oil refining. Currently, the CEN EN 590 [3] specification allows up to 7% v/v FAME, meeting the EN 14214 [4] specification, to be blended into diesel fuel and all European diesel vehicles are fully compatible with these blends. FAME blending components manufactured from different vegetable and animal waste products are widely available but Rapeseed Methyl Ester (RME) is used most frequently and has been chosen for this study.

Biodiesel blends based on FAME are routinely used in most EU Member States. In some countries, neat FAME (B100) is also allowed by national legislation and some captive fleets have been specially adapted to operate routinely on FAME/diesel blends in the range 20-30% v/v. As a result, any potential impact of FAME on vehicle emissions is already affecting vehicle fleet pollutant emission levels and air quality. Such impacts will gain significance in view of tighter emissions standards (Euro 5 and 6) [5,6].

This study on three light-duty passenger cars was designed to investigate the effect of FAME on fuel consumption and emissions. These Euro 4 vehicles were equipped with common rail turbocharged engines and different exhaust aftertreatment technologies. Two of the vehicles were equipped with Diesel Particulate Filters (DPFs) while one was not. Two different schemes were also used on the vehicles to regenerate the DPFs.

Four fuels were evaluated in this study. One hydrocarbon-only diesel fuel (B0, complying with the EN 590 specification) was blended with commercially sourced Rapeseed Methyl Ester (RME) (complying with the EN 14214 specification) to produce RME/diesel blends of 10% v/v (B10), 30% v/v (B30), and 50% v/v (B50) RME. These RME concentrations were selected to anticipate future increases in renewable energy requirements and to accentuate the impact of RME on fuel consumption and vehicle emissions.

Measurements were performed according to the New European Driving Cycle (NEDC), which is the European regulatory test procedure. Additionally, ARTEMIS [7] transient cycles were used to simulate more 'real world' driving conditions as well as two constant speed conditions at 50 and 120 km/h. All fuel/vehicle combinations were tested in a rigorously controlled and statistically designed test programme.

Because FAME has a slightly lower energy content compared to fossil diesel fuel, it was of interest to know how significantly higher FAME contents would impact

vehicle fuel consumption and emissions. The results on fuel consumption and regulated emissions have been reported by Concaawe [8] and elsewhere [9] while the impact of RME/diesel blends on other emissions is reported in this study.

In this report, the total particulate matter (PM), total particle number (PN), solid PN and PN size distribution are addressed as well as the emissions of different carbonyl compounds from the tailpipe. By chemically extracting the total PM captured on filter paper and analysing the extracted fractions, the effects of fuel, vehicle and driving cycle on the soluble organic fraction (SOF), anionic species, and elemental carbon comprising the PM were also investigated.

2. EXPERIMENTAL

2.1. TEST VEHICLES

Three modern light-duty diesel passenger cars equipped with direct injection, high injection pressure, common rail and turbocharged diesel engines were selected for this test programme. All vehicles featured exhaust gas recirculation (EGR) and some type of Diesel Oxidation Catalyst (DOC) and were homologated to the Euro 4 emissions standard. Vehicles 1 and 3 were also equipped with Diesel Particulate Filters (DPF) for PM emissions control. While the DPFs in both vehicles were regenerated by means of periodically injecting additional fuel at the end of the combustion cycle, the DPF in Vehicle 1 used a catalyzed filter while Vehicle 3 used a soluble metallic additive injected into the fuel at the time of DPF regeneration. The vehicles and their characteristics are summarized in **Appendix 1**.

2.2. TEST FUELS

Four fuels were tested in this study: a hydrocarbon-only diesel fuel (B0) and 3 blends of this diesel fuel with a single batch of Rapeseed Methyl Ester (RME). The B0 diesel fuel complied with the CEN EN 590 specification [3] and had less than 10 mg/kg sulphur content.

The RME blending component complied with the CEN EN 14214 specification [4] and contained 1,000 ppm of a commercial antioxidant additive (BHT) to ensure oxidation stability. The RME was blended into the diesel fuel at three different concentrations, resulting in the following four test fuels:

- B0: Base diesel fuel
- B10: 10% v/v RME in B0
- B30: 30% v/v RME in B0
- B50: 50% v/v RME in B0

An ester-type lubricity additive and a fully-formulated diesel performance additive package that is widely used in Europe were also added to all test fuels at the same concentration in order to ensure fuel system lubricity and cleanliness throughout the vehicle test programme.

The properties of these four test fuels are summarised in **Appendix 2**.

3. METHODOLOGY

Based on the existing literature, it was apparent that a sound experimental design would be required in order to make repeatable, reliable and consistent measurements. Although this was especially important for the fuel consumption and regulated emissions measurements reported in [8,9], a robust test design was also important for the unregulated emissions effects reported here.

3.1. TEST PROTOCOL

The test protocol is described in detail in [8] while **Appendix 3** summarises the daily procedure for vehicle preparation and testing. After equilibrating the vehicle and fuel overnight, each day's testing started with a cold NEDC test followed by ARTEMIS hot start cycles and steady-state conditions. The test protocol was strictly followed so that all vehicle preparation, fuel changes, and measurements were carried out in the same way for each vehicle/fuel combination, thus eliminating many potential sources of experimental error.

The following test cycles were included:

- NEDC on the cold vehicle consisting of:
 - an Urban Driving Cycle (UDC, also called the ECE-15)
 - and an Extra Urban Driving Cycle (EUDC),
- ARTEMIS Urban and Road transient cycles [7], and
- 120 km/h steady-state mode with some measurements also collected at a 50 km/h steady-state condition.

In addition to the regulated emissions reported previously [8], the following additional measurements were completed:

- Total PM, captured from the tailpipe exhaust and weighed on filter paper;
- Analysis of the PM composition from selected test conditions. This compositional analysis included the soluble organic fraction (SOF) as measured by gas chromatography (GC), anionic species by ion chromatography (IC), and elemental carbon (EC) obtained by difference;
- PN, both total and solid, over all cycles and the particle size distribution over selected steady state tests;
- Analysis from selected cycles of carbonyl compounds, both aldehydes and ketones. These compounds were captured from the tailpipe exhaust using filter cartridges and analyzed, following extraction from the cartridges, by High Performance Liquid Chromatography (HPLC);
- Photos of the PM filter papers.

3.2. DATA ANALYSIS

The PM emissions from the vehicle tests were captured on filter papers as per the regulatory procedure and photos of these filters are shown in **Appendix 10**. With these photos, it is easy to observe that Vehicles 1 and 3 were equipped with DPFs which were very effective in capturing the PM emissions within the exhaust aftertreatment system.

Selected filter papers from both the NEDC and ARTEMIS Urban tests were subjected to additional chemical analyses as described in **Section 3.3**. The results from these analyses allowed each measured component on the filters to be calculated in terms of mg/km from the individual particle loading on that filter and the average 'mg/km' calculated from the analysed filters for each vehicle/fuel combination.

To prepare the PM composition graphs shown in **Section 4.1.3**, the mean PM mg/km values measured during the indicated test procedure were used as the basis for the chemical analysis of the PM samples. EC contents were calculated by difference and the percentage compositions of the PM samples were calculated from the combined values. Because the EC was calculated by difference, it is not possible to show error bars for each component.

Measured sulphate values were corrected to include bound water. The factor used for this calculation has been under discussion over the years¹ and the impacts of DPF and filter paper type have been discussed [10]. Following these discussions, a factor of 0.9 has been used here to account for the contribution of bound water.

3.3. PM ANALYSIS PROCEDURES

3.3.1. Determination and breakdown of soluble organic fraction

The total SOF in the PM of the tailpipe emissions was determined according to IP 443/99 standard [11]. The hydrocarbons extracted from the SOF were also apportioned to fuel and lubricant boiling ranges using a GC analysis as described in IP 442/99 [12].

¹ Historically a value of 1.3 x [SO₄²⁻] has been used to account for the contribution of bound water

The basis of this IP method is the separation or ‘cut-off’ that is found between the GC traces of the highest-boiling fraction of the fuel fraction and the lubricant used in the vehicles. The ‘topped’ fuel (according to IP123/94 [13]) was prepared by distilling the fresh B0 fuel in order to remove all components boiling below 315°C. The chromatograms from the B0 ‘topped’ fuel fraction and the lubricant were then overlaid (**Figure 1**) and the cut-off point at about 20 minutes was determined. This cut-off point and the GC conditions were then fixed and not changed in subsequent measurements.

Figure 1 Overlapping GC traces of ‘topped’ fuel and lubricant

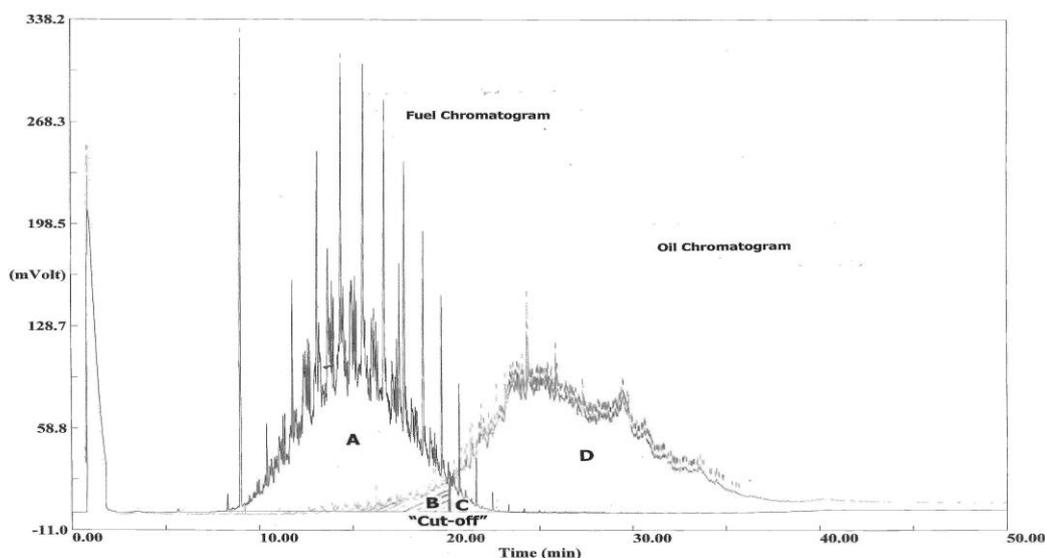
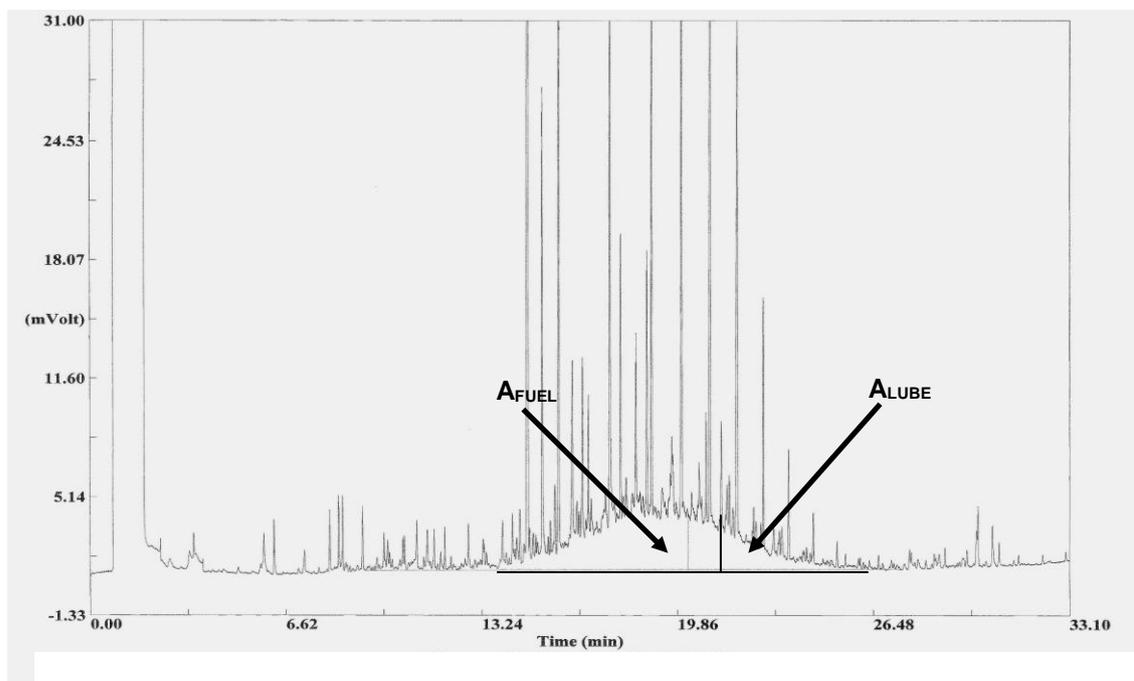


Figure 2 shows an example of a typical GC trace for an SOF extract. This GC was obtained on the SOF extracted from the PM filter paper for Vehicle 1 running on the B0 fuel over the ARTEMIS Urban driving cycle. The fractions of fuel- and lube-derived SOF were then calculated from the integral values of the GC trace before and after the cut-off point determined from **Figure 1**. Clearly, the cut-off point determined as described above only provides an estimate of the fraction of SOF which may have originated from the uncombusted fuel and from the vehicle lubricant. This is because of the significant overlaps that exist in the GC trace between the lower-boiling (predominantly fuel) and higher-boiling (predominantly lubricant) components in the GC analysed fraction.

The impact on the Redox activity of the soluble oil fraction was also studied. That work is not described here but is included in a paper published in FUEL [39].

Figure 2 GC of the SOF extracted from the PM emissions for Vehicle 1 running on the B0 fuel over the ARTEMIS Urban driving cycle



3.3.2. Anionic species

Anionic species, specifically nitrates and sulphates, in the PM were determined by means of ion chromatography (IC) coupled with a conductivity detector. The details of the analytical method are described in **Appendix 5**.

3.3.3. Carbonyl compounds in the vehicle exhaust

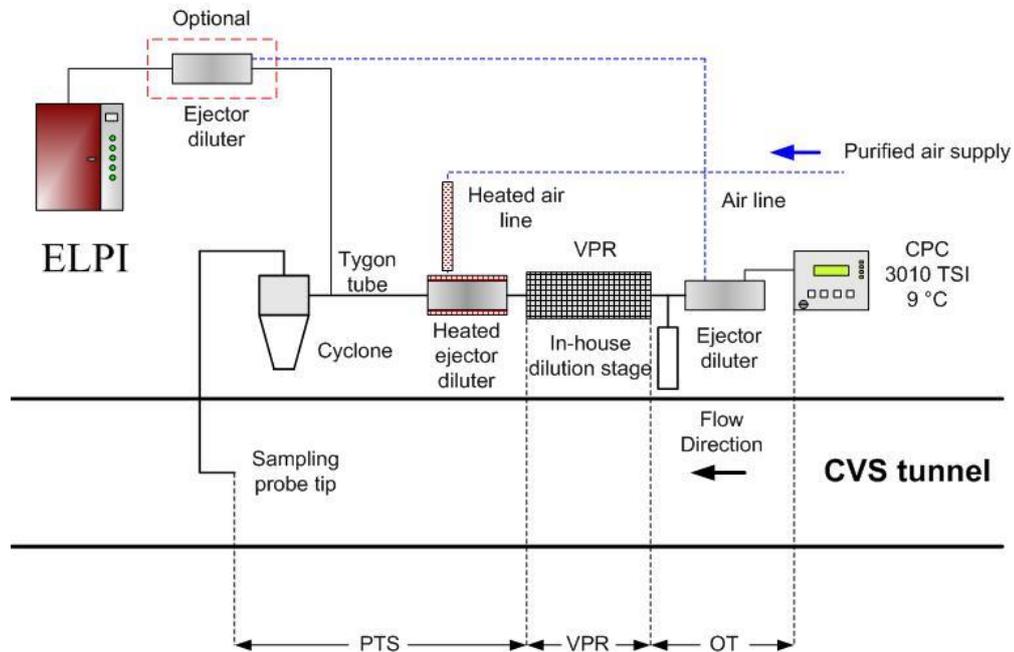
Carbonyl compounds from the vapour phase of the vehicle exhaust were collected using DNPH-coated cartridges, one for each vehicle test. After each test, the cartridges were stored at -18°C until the solvent extraction step could be completed. The determination of carbonyl compounds was based on the US EPA TO-11A standard method [14] and the test protocol is described in **Appendix 5**.

3.4. PN SAMPLING PROTOCOL

The number of solid particles was measured according to Particle Measurement Programme (PMP) protocol [15]. A schematic and description of the sampling equipment are given in **Figure 3**.

In addition to the measurements of solid PN, the particle size distributions were also measured during the steady state test conditions. For these measurements, an Electrical Low Pressure Impactor (ELPI) was used and the total emitted particles were calculated from a summation over the 13 stages of the ELPI instrument.

Figure 3 PN sampling according to the PMP protocol including the ELPI



The ELPI was used to measure the total PN downstream of the cyclone and in parallel to the PMP system, as shown in **Figure 3**. The heated ejector diluter that was upstream of the ELPI was only used for measurements on the non-DPF-equipped Vehicle 2 because of the higher ultrafine particle emissions emitted by this vehicle. The ELPI classifies particles according to their aerodynamic diameter and, as a result, data on the particle size distribution can be obtained in addition to the total number of solid particles.

A Scanning Mobility Particle Scanner (SMPS) was also used consisting of a Condensation Particle Counter (CPC 3010, manufactured by TSI) and a Long Differential Mobility Analyzer (LDMA) classifier. The LDMA produces a mono-disperse aerosol of diameters ranging from approximately 10 to 300nm and the CPC counts the respective number of these aerosol particles. In this way, the distribution of particle size as a function of aerodynamic diameter can be measured. This approach was used during the steady-state tests in order to acquire more information about the physical characteristics of the emitted particles. The SMPS always sampled in the same position as the CPC, according to [15].

3.5. STATISTICAL ANALYSIS

This section describes the statistical methods that were used to analyse the unregulated emissions data from this study. These methods are similar to those used to analyse the regulated emissions in the Part 1 report [8] and in earlier C[] & ^ studies [16,17,18,19]. More details can be found in **Appendix 6**.

PM and PN Emissions

Each particle emission measurement (PM, total PN, and solid PN) was examined separately on a vehicle-by-vehicle and cycle-by-cycle basis.

In previous Concaawe studies [16,17,18,19], the variability in particle measurements has typically been found to follow a lognormal distribution with the degree of scatter increasing as the particle emission level increases. The standard deviation vs. mean plots in **Appendix 9** show that PM, total PN, solid PN measurements all behaved in this way in those vehicles and driving cycles where there were noticeable differences in emissions between fuels. Therefore, all subsequent statistical analyses are based on the assumption of lognormality of particle emissions.

The data were examined for outliers by inspecting studentized residuals (residuals divided by their standard errors) and less than 2% of the measured emission values were rejected based on this analysis.

A significant time trend (at $P < 1\%^2$) was found in just one of the 39 PM, total PN, and solid PN data sets (representing a total of 3 vehicles \times 3 measurements \times up to 5 cycles). This one set of data points was adjusted to where they might have been had all of the tests been conducted halfway through the test programme. Both the uncorrected and corrected means are shown for each vehicle, fuel and driving cycle in **Appendix 7**.

In **Appendix 7** and in the bar charts in **Section 4**, arithmetic means are shown for PM results while geometric (i.e. logarithmic) means are used for PN results. This is also consistent with previous studies. Geometric means give excellent comparisons between fuels on a percentage basis but have the disadvantage of underestimating total emissions to the atmosphere. Arithmetic means give better estimates of total emissions to the atmosphere but can be inflated unduly by isolated high results for measurements like PN which can vary by one or more orders of magnitude.

In the bar charts presented in **Section 4**, the error bars show the mean value $\pm 1.4 \times$ the standard error of the mean value. The 1.4 factor was selected for consistency with the EPEFE study [20] and with previous Concaawe studies. Emissions from two fuels will not be significantly different from one another at $P < 5\%$ unless there is a sizeable gap between their error bars.

Carbonyl compounds

Many of the measurements of carbonyl compounds described in **Section 3.3.3** were at or below the detection limit with some results reported to be zero. As a consequence, most of the measured variables were not of sufficient consistency to permit the use of the statistical methods described above.

Therefore carbonyl emissions have been summarized more simplistically. Arithmetic means of the raw data are presented with error bars showing the minimum and maximum measured values over the 4 or 5 repeat tests. The min-max error bars have been plotted in grey in **Figures 10 to 11** in order to distinguish these from the more usual kind representing statistical error.

² $P < 1\%$ = the probability that such an event could be observed by chance when no real effect exists is less than 99%. In other words, we are 99% confident that the effect is real.

4. RESULTS AND DISCUSSION

The particle emissions measurements from the three vehicles are described in this section. The results include total and solid PN emissions, particle size distributions, PM composition, and carbonyl-containing compounds, as aldehydes and ketones.

4.1. PARTICLE EMISSIONS

4.1.1. PN Emissions

PN emissions are an increasing concern because of their potential impact on human health. There have been many investigations into the measurement of the number (and type) of particles emitted from automotive vehicles. Under the auspices of the UNECE GRPE, the Particle Measurement Programme (PMP) was put in place to develop a standard protocol [15]. The PMP procedure focussed on the measurement of dry (solid) particles following the removal of volatile material from the vehicle exhaust stream. Limits on vehicle PN emissions have also been added to light duty diesel vehicle specifications and a compliance limit of 6×10^{11} solid particles/km has been adopted in the EU's Euro 5b technical regulation [6]. Similar compliance limits have been established for gasoline direct injection vehicles and heavy-duty vehicles. In addition to the solid PN, total PN (including any volatile particles) were measured directly on diluted exhaust using an ELPI as described in Section 3.4.

Figures 4 and 5 show the total and solid PN emissions for all vehicles and driving cycles.

Figure 4 Total PN emissions for all vehicles and driving cycles (geometric means)

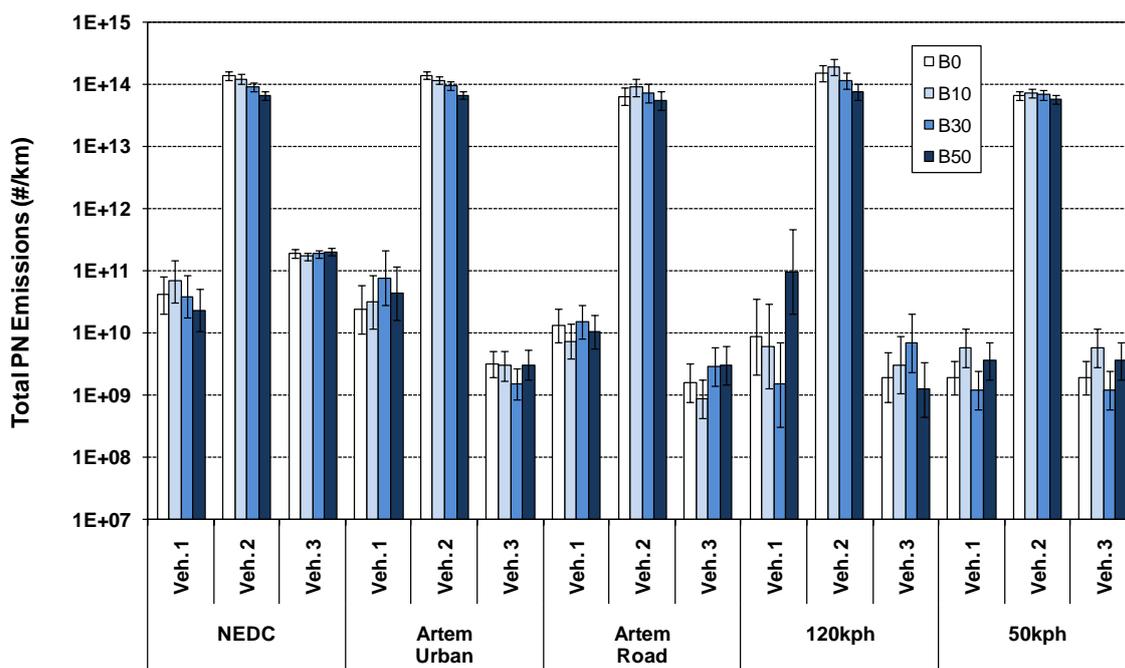
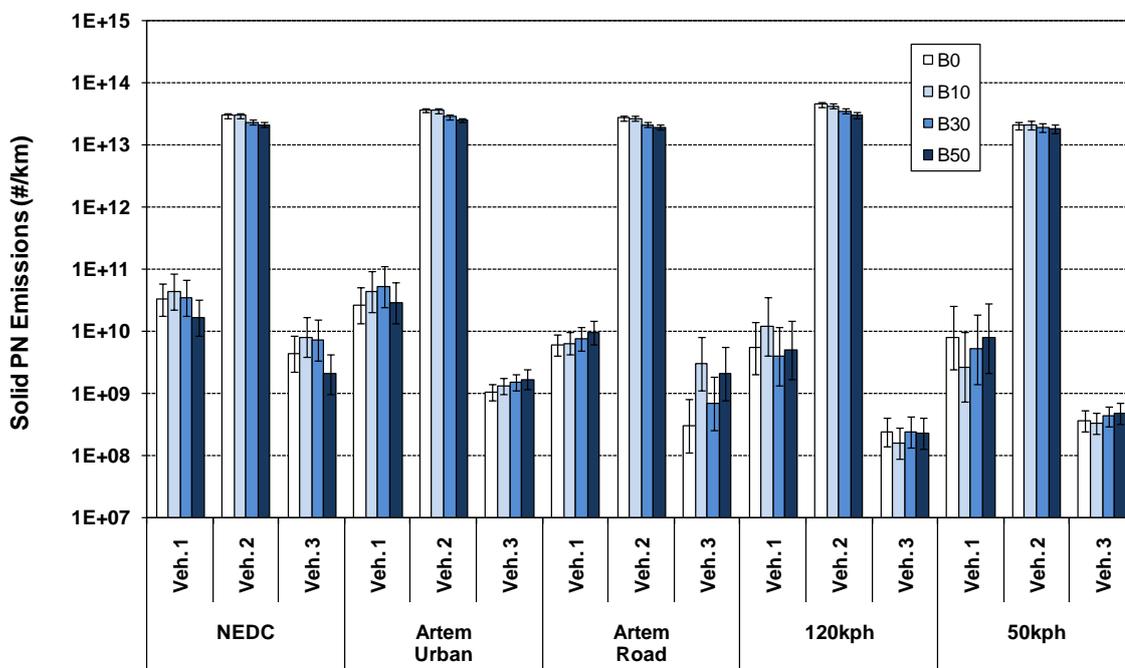


Figure 5 Solid PN emissions for all vehicles and driving cycles (geometric means)



PN levels less than about 1×10^9 /km were considered to be within the background PN levels of the test laboratory and some of the PN results for the DPF-equipped Vehicles 1 and 3 were at or below this background level. It is clear that the DPFs successfully reduce PN at the same time that they reduce the PM levels and there is little evidence that RME has an impact at these low levels of PN emissions.

For Vehicles 1 and 3, there are some differences in absolute PN levels which generally decrease from cold to hot driving cycles. The overall differences between cycles are not large, however, and are close to the background level.

For Vehicle 2, emissions of both total and solid PN are up to four orders of magnitude greater than for Vehicles 1 and 3. For both the total and solid PN emissions, the concentrations are almost the same across the different test cycles. The total PN emissions are higher than the solid PN emissions by about a factor of three or four.

An increase in RME concentration also results in reductions in both total and solid PN emissions across all test conditions. There were two exceptions: the 50 km/h steady-state condition and the total PN over the ARTEMIS Road cycle. A similar observation has been reported previously [21]. Although they are statistically significant, these reductions are small compared to those due to the presence or absence of the DPF device.

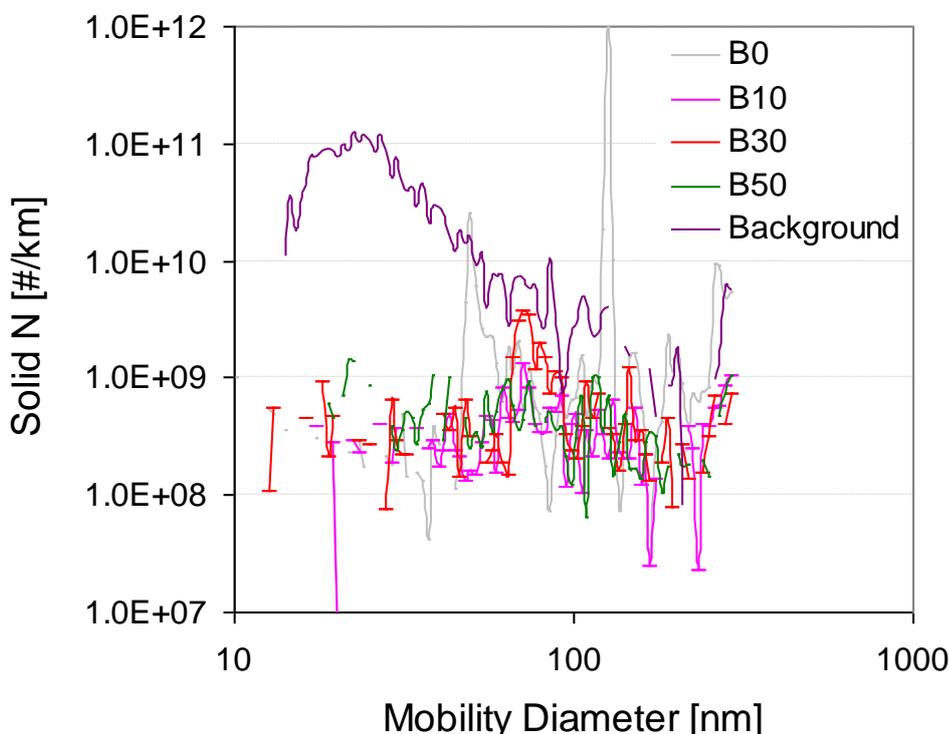
Because the measurement of total and solid PN is comparatively new, there are few publications that relate to the effect of biodiesel concentration on PN emissions. The results reported here are not consistent with the decreases in total PN observed by one group [22] while they are consistent with those reported by another group [21]. However, the different vehicles, driving cycles, and FAME types may well account for the observed variations in these studies.

One might have expected to see an increase in total PN emissions with increasing RME concentration. An increase, for example, could have occurred because of an increase in the semi-volatile components on the PN due to the higher molecular weight components comprising the RME but this was not observed. Instead, the reduction in total PN with increasing RME appears to reflect a reduction in solid PN. It can be concluded that the reduction of solid particles occurs to a larger extent than the creation of any semi-volatile components, at least within the size range measured here.

4.1.2. Particle size distribution

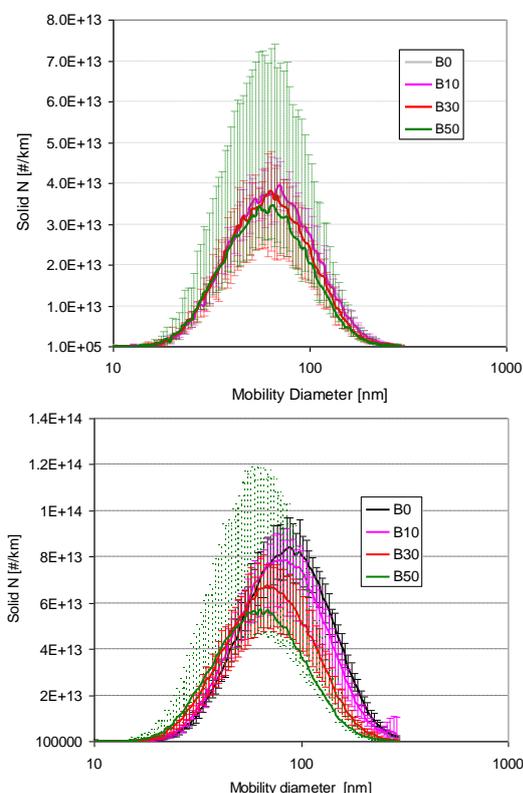
At the steady state test conditions, particle size distribution measurements were also conducted on the solid particle emissions in addition to the solid PN measurements. These measurements were made using the SMPS as described in **Section 3.4**. For the DPF-equipped Vehicles 1 and 3, the concentrations of PN emissions were found to be at the same (or even lower) concentrations than the background levels. As an example, **Figure 6** shows the particle size distributions recorded for Vehicle 3 at 50 km/h. Because of the very low concentrations, it is therefore not possible to draw any firm conclusions from these data.

Figure 6 Indicative particle size distributions from Vehicle 3 at 50 km/h



On the other hand, the particle size distributions measured from the non-DPF-equipped Vehicle 2 are shown in **Figure 7** at both 50 km/h and 120 km/h. In these charts, the bold lines show the average particle distributions while the dashed vertical lines show the variation in measured values at a given particle mobility diameter.

Figure 7 Solid particle size distributions at 50 km/h (left) and 120 km/h (right) from Vehicle 2 for all fuels. Note that the vertical scales are slightly different between the left and right hand charts.



For Vehicle 2, the PN emissions were significantly higher than in the other two vehicles and the particle size distributions were clearly distinguished from background particle concentration. As shown in **Figure 7**:

- At 50 km/h, higher RME concentrations appear to have little effect on the measured size distribution which is consistent with the solid PN results shown in **Figure 5**.
- At 120 km/h, the solid PN decreases with increasing RME content which is also consistent with the results in **Figure 5**. This observation has also been reported previously [23].
- A shift towards lower mobility diameters is observed with increasing RME content. This has also been reported by other workers [21,23,25].
- Increasing the RME concentration up to 50% v/v appears to increase the variability in the PN emissions measurement as observed by the variation at a given mobility diameter.
- Other studies have suggested that an increase in nucleation particles can be seen [21,25] with increasing RME concentration while another study [23] reports a decrease in particles. Some of this earlier work has been carried out on heavy-duty engines where a nucleation mode is more discernible. In light-duty engine testing, a nucleation mode is not always apparent and measurements carried out in this study showed no indication of a nucleation peak within the particle size range measured.

4.1.3. PM composition

The chemical compositions of the PM from these vehicle tests (in mg/km) are shown in **Figure 8** for the NEDC and in **Figure 9** for the ARTEMIS Urban cycle. These figures show the PM composition, including the fuel- and lube-derived hydrocarbons, nitrate and sulphate anions plus water, and EC determined by difference. The composition analysis was completed as described in **Section 3** and in **Appendix 5**.

Pie charts showing the percentage composition (rather than the absolute values) of the PM sample have also been included in these figures.

Figure 8 Total PM emissions (mg/km) and their chemical composition for all vehicles over the NEDC

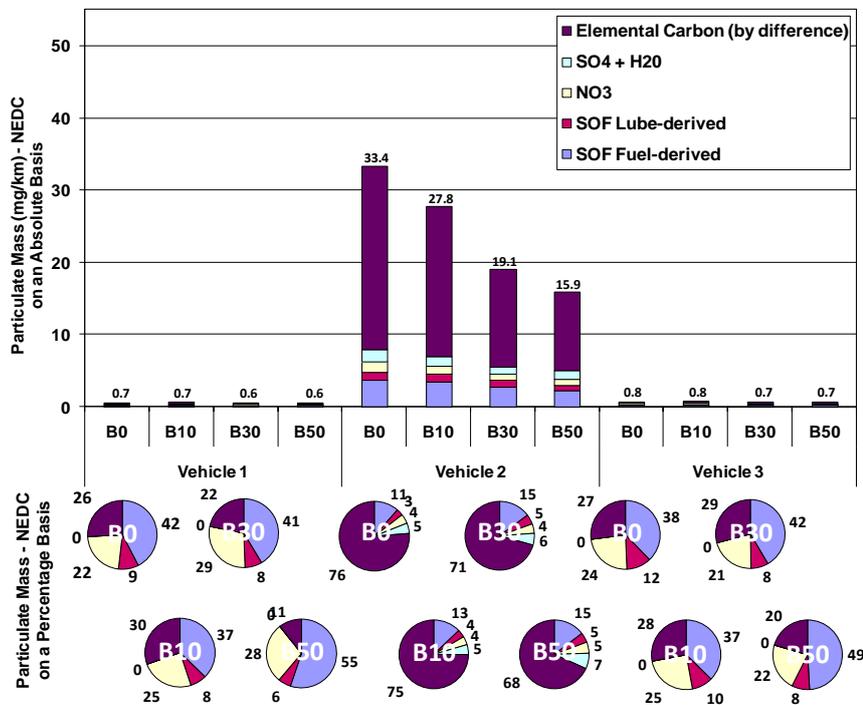
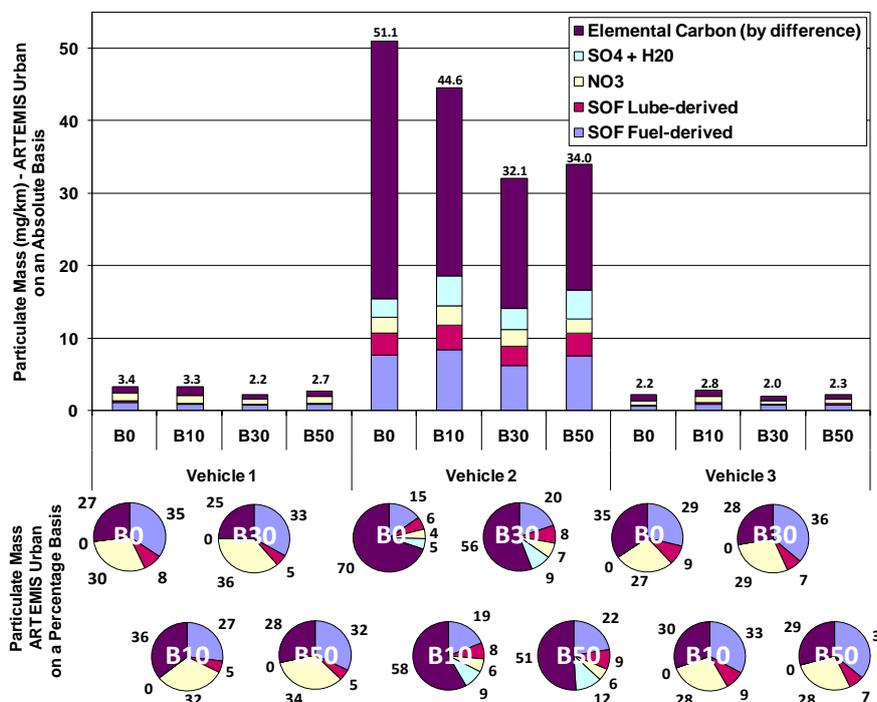


Figure 9 Total PM emissions (mg/km) and their chemical composition for all vehicles over the ARTEMIS Urban cycle



As the total PM decreases and the chemical composition changes, it is not always clear which changes can be specifically ascribed to the RME content of the test fuel. For the non-DPF equipped Vehicle 2, the reduction in total PM (in mg/km) is largely due to the decrease in EC with increasing RME. As a consequence, there is a trend toward an increase in fuel-derived SOF with increasing RME. These effects are directionally the same in the NEDC and ARTEMIS Urban cycle and, because of the higher total PM in the ARTEMIS cycle, the effect is more evident in this case. Published literature from other studies have reported similar findings (e.g., [23,25,26,27]). A reduction in EC with increasing RME in the test fuel is consistent with both the reduction in regulated PM weights and the reduction in solid PN emissions (Figure 5).

It must be remembered that the absolute PM weights for the DPF-equipped Vehicles 1 and 3 are very low resulting inevitably in more variability in the composition measurements. Thus, some care should be taken in over-interpreting the PM composition results from these vehicles. Nevertheless, some observations can be made when reviewing the relative graphs of the PM chemical composition. Compared to Vehicle 2, no sulphate was measured in the PM from either Vehicle 1 or Vehicle 3 over either driving cycle. In addition, the measured nitrate was also a greater percentage of the PM for both driving cycles than for the PM from the non-DPF equipped Vehicle 2.

4.2. CARBONYL COMPOUNDS

Several publications refer to the impact of different biofuels on carbonyl emissions (e.g., [21,26,28,29,30,31,32,33]) from both light- and heavy-duty vehicles. Hansen [28] states that measured carbonyls from biofuel blends are predominantly

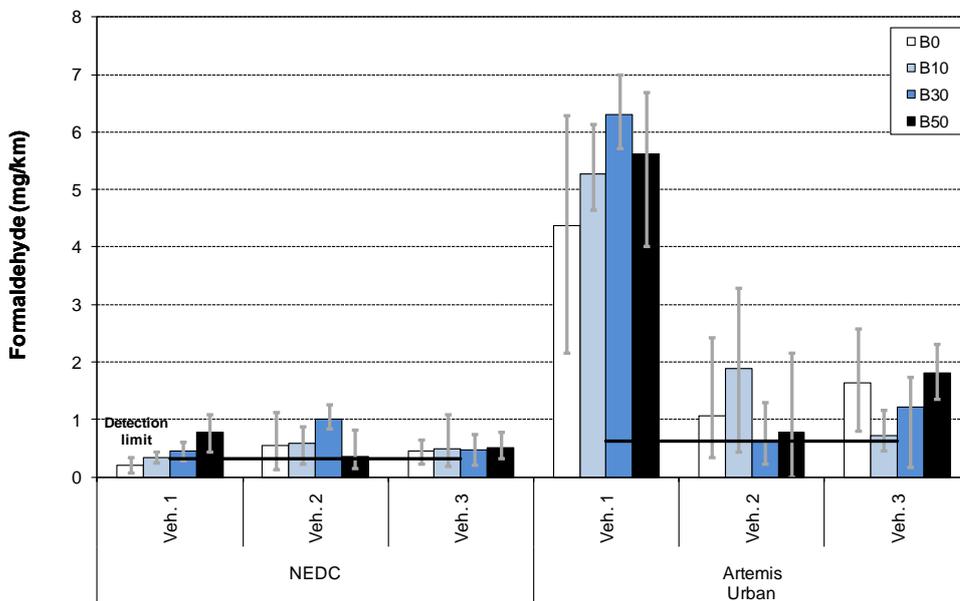
formaldehyde and acetaldehyde (which agrees with [21,30]) which are assumed to be synthesised during the combustion process. Some workers also mention the presence of acrolein [21,32,33] in tailpipe emissions but it has also been mentioned [32] that this could be due to residual glycerine from the FAME product and not from partial combustion.

Although many workers report that carbonyl emissions increase when FAME is used, other studies [30,31,32] saw little change while one study [26] reported lower emissions of carbonyl compounds as the FAME concentration increased.

The carbonyl compounds measured in this study included formaldehyde, acetaldehyde, acetone and propionaldehyde in both the NEDC and ARTEMIS Urban driving cycles. Acetaldehyde values were generally at or slightly below the detection limit for most vehicles, fuels and cycles.

The average formaldehyde emissions are shown in **Figure 10**. The ARTEMIS Urban driving cycle produced more formaldehyde than did the NEDC for almost all vehicle and fuel combinations. Vehicle 1 also showed significantly higher emissions over the ARTEMIS Urban driving cycle compared to the NEDC and compared to the other two vehicles.

Figure 10 Formaldehyde emissions (in mg/km) for all vehicles over the NEDC and ARTEMIS Urban cycle (where the grey bars represent min and max measurements). The detection limit for formaldehyde, estimated from the analytical measurements, is also shown by the horizontal black line.



Average acetaldehyde emissions are shown in **Figure 11** while the averages of the combined acetone plus propionaldehyde emissions are shown in **Figure 12**.

Figure 11 Acetaldehyde emissions for all vehicles over the NEDC and ARTEMIS Urban driving cycle (grey bars represent min and max measurements)

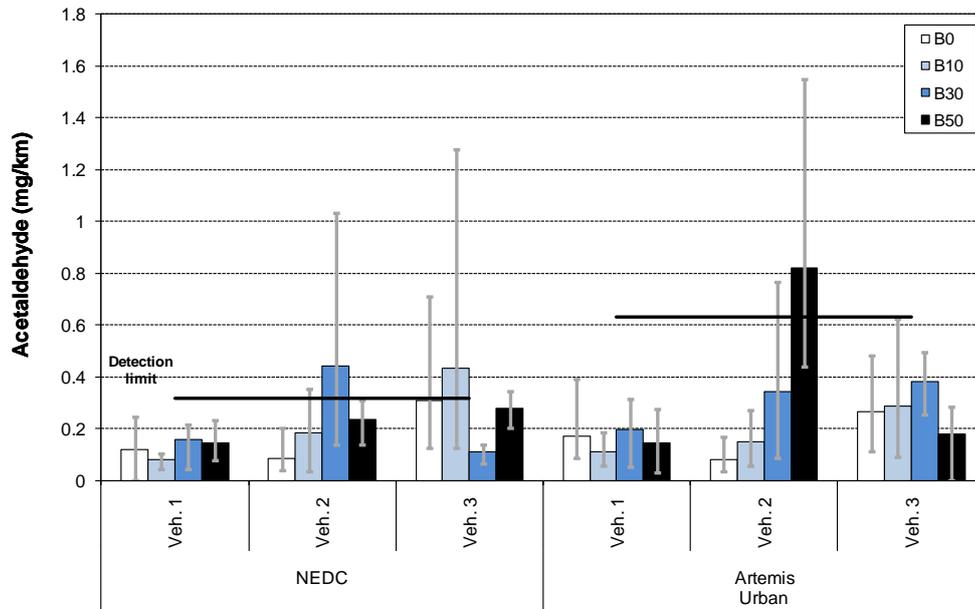
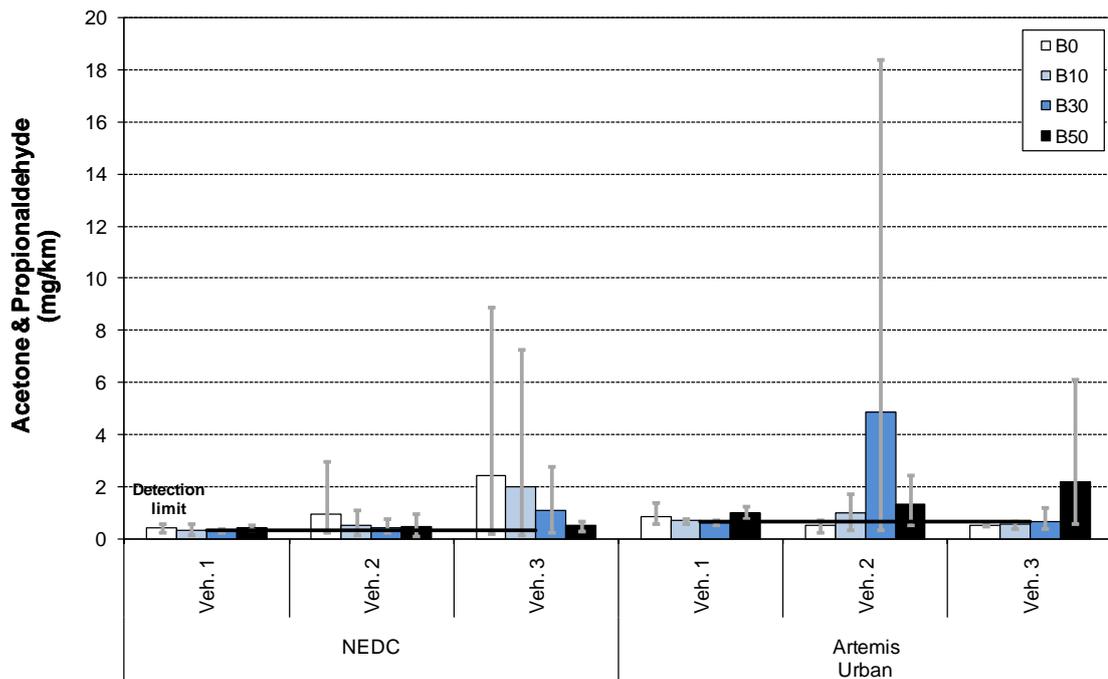


Figure 12 Combined acetone plus propionaldehyde emissions for all vehicles over the NEDC and ARTEMIS Urban driving cycle (grey bars represent min and max measurements)



The mean values for all carbonyl compounds are summarized in **Appendix 8**.

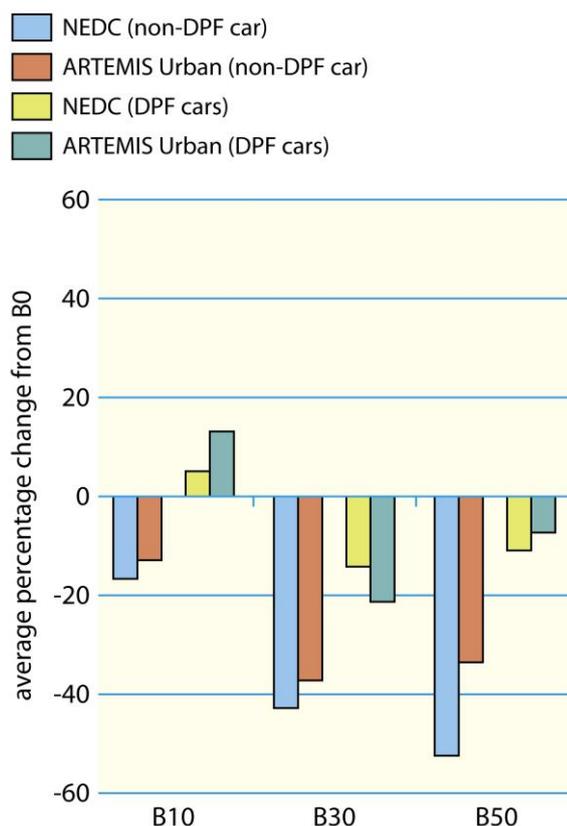
5. CONCLUSIONS

PM Emissions:

- For the non-DPF-equipped Vehicle 2, the PM emissions for the B0 fuel were 18-46 times higher than those from the DPF-equipped vehicles, depending on the vehicle and driving cycle.
- The PM emissions from Vehicle 2 decreased by more than 50% at the highest RME content over the NEDC, consistent with previously published literature.
- The results in **Figure 13** summarize the percentage change in PM emissions from the non-DPF-equipped Vehicle 2 and the DPF-equipped Vehicles 1 and 3 over the NEDC and ARTEMIS Urban driving cycles.

Figure 13 Average percentage change in PM emissions due to the RME content of the test fuel

Average percentage change in PM emissions



PM Composition:

- For the non-DPF equipped Vehicle 2, increasing the RME content in the fuel appeared to increase the % SOF and reduce the percentage of EC over both the NEDC and ARTEMIS Urban cycle. The fuel-derived % SOF was consistently higher than the lube-derived % SOF and the total % SOF values were higher over the ARTEMIS Urban cycle than they were over the NEDC.

- Although the total PM emissions from the DPF-equipped Vehicles 1 and 3 were very low, nitrate anion represented a higher percentage of the PM from these vehicles compared to the PM from Vehicle 2.
- While sulphate anion was measured to be a small percentage of the PM from Vehicle 2, no sulphate was measured in the PM from Vehicles 1 and 3. Because the sulphur concentration of the fuel was very low, it is likely that the sulphur observed in the PM originated with the lubricant.

PN Emissions:

- Solid PN emissions from the DPF-equipped Vehicles 1 and 3 were up to four orders of magnitude lower than from the non-DPF-equipped Vehicle 2 and were close to the testing facility's background levels.
- For Vehicle 2, a trend towards lower solid PN emissions with increasing RME content was observed over most test conditions. This is consistent with the reduction in total PM and the change in percentage EC that was also measured in the study. However, these reductions were very small compared to those due to the presence or absence of the DPF.

Particle Size Distribution:

- Increasing the RME content in the fuel resulted in a shift of particle emissions toward lower mobility diameters and increased the variability in the particle emissions measurement.
- Especially at the 120 km/h steady-state condition, the change in particle size distribution showed that accumulation mode particles are reduced as the RME content increases. This is consistent with the reduction in total PM and solid PN emissions and the change in percentage elemental carbon as described above.
- Nucleation mode particles were not evident in these measurements.

Carbonyl compounds:

- The ARTEMIS Urban driving cycle produced more formaldehyde than did the NEDC for almost all vehicle and fuel combinations. Low levels of acetaldehyde and acetone/propionaldehyde were also measured.
- Vehicle 1 also showed significantly higher formaldehyde emissions over the ARTEMIS Urban driving cycle compared to the NEDC and compared to the other two vehicles.
- The concentrations of these carbonyl compounds were very low in most tests and there was little evidence of an effect due to the concentration of RME in the test fuel.

6. GLOSSARY

ANOVA	Analysis of Variance
ARTEMIS	Assessment and Reliability of Transport Emission Models and Inventory Systems
AUTh	Aristotle University of Thessaloniki
CO	Carbon Monoxide
CO ₂	Carbon Dioxide
CPC	Condensation Particle Counter
CVS	Constant Volume Sampling (system)
DG TREN	Directorate General for Transport and Energy (European Commission)
DI	Direct Injection
DNPH	2,4-dinitrophenylhydrazine
DOC	Diesel Oxidation Catalyst
DPF	Diesel Particulate Filter
DR	Dilution Ratio
EGR	Exhaust Gas Recirculation
ELPI	Electrical Low Pressure Impactor
EN 590	CEN Specification for European Diesel Fuel
EN 14214	CEN Specification for European FAME for fuel blending
EPCL	Environmental Pollution Control Laboratory (at AUTh)
EPEFE	European Programme on Emissions, Fuels and Engine Technologies
ET	Evaporation Tube
EUDC	Extra Urban Driving Cycle
FC	Fuel Consumption
FID	Flame Ionization Detector
FIE	Fuel Injection Equipment
FPS	Fine Particle Sampler

GRPE	Working Party on Pollution and Energy (UNECE)
HC	Hydrocarbon
HD	Heavy-duty
HEPA	High Efficiency Particulate Air filter
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
JRC	Joint Research Centre (of the European Commission)
KV40	Kinematic Viscosity at 40°C
LAT	Laboratory of Applied Thermodynamics
LD	Light-duty
LEPA	Low Efficiency Particulate Air filter
lpm	Litres per minute
NEDC	New European Driving Cycle
NO	Nitric Oxide
NO ₂	Nitrogen Dioxide
NO _x	Nitrogen Oxides
PAH	Polycyclic Aromatic Hydrocarbon
PM	Particulate Matter or Mass
PMP	Particle Measurement Programme
PN	Particle Number
PNC	Particle Number Counter
PND	Particle Number Diluter
RME	Rapeseed Methyl Ester
Significant	Statistically significant at >95% confidence
SMPS	Scanning Mobility Particle Scanner
T10	Temperature (°C) at which 10% v/v diesel is recovered
T50	Temperature (°C) at which 50% v/v diesel is recovered

T95	Temperature (°C) at which 95% v/v diesel is recovered
TC	Turbo Charged
TD	Thermal Denuder
TSI Inc.	Trust, Science, Innovation Incorporated
UNECE	United Nations Economic Commission for Europe
UV	Ultraviolet
VPR	Volatile Particle Remover

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- The Association for Emissions Control by Catalyst (AECC), Brussels, Belgium, for providing Vehicle 3 (the 'golden vehicle' [15]) used in this study.
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While this project was being completed, we were saddened to hear that our collaborator, Professor Stam Stournas at the National Technical University of Athens, had passed away. He was a valued contributor to our project, as well as to European research on fuels and vehicles and his many contributions will be missed.

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APPENDIX 1 DIESEL VEHICLES

Vehicle Characteristics	Units	Vehicle 1	Vehicle 2	Vehicle 3
Model Year	Year	2009	2004	2005
Cylinders		4	4	4
Displacement	cm ³	2148	2200	1997
Maximum Power	kW @ rpm	110	100	98
Injection System		Common Rail Direct Injection	Common Rail Direct Injection	Common Rail Direct Injection
Transmission		Automatic	Manual	Manual
Euro Certification		Euro 4	Euro 4	Euro 4
Exhaust Aftertreatment				
• EGR		Yes	Yes	Yes
• Diesel Oxidation Catalyst (DOC)		Yes	Oxidation pre-catalyst plus 2-stage DOC with DeNO _x characteristics	Yes
• Diesel Particulate Filter (DPF)		Yes, catalyzed DPF that is regenerated by in-cylinder fuel injection	None	Yes, regenerated by in-cylinder injection of fuel-borne catalyst
Mileage at start of testing	km	3,487	62,118	27,603

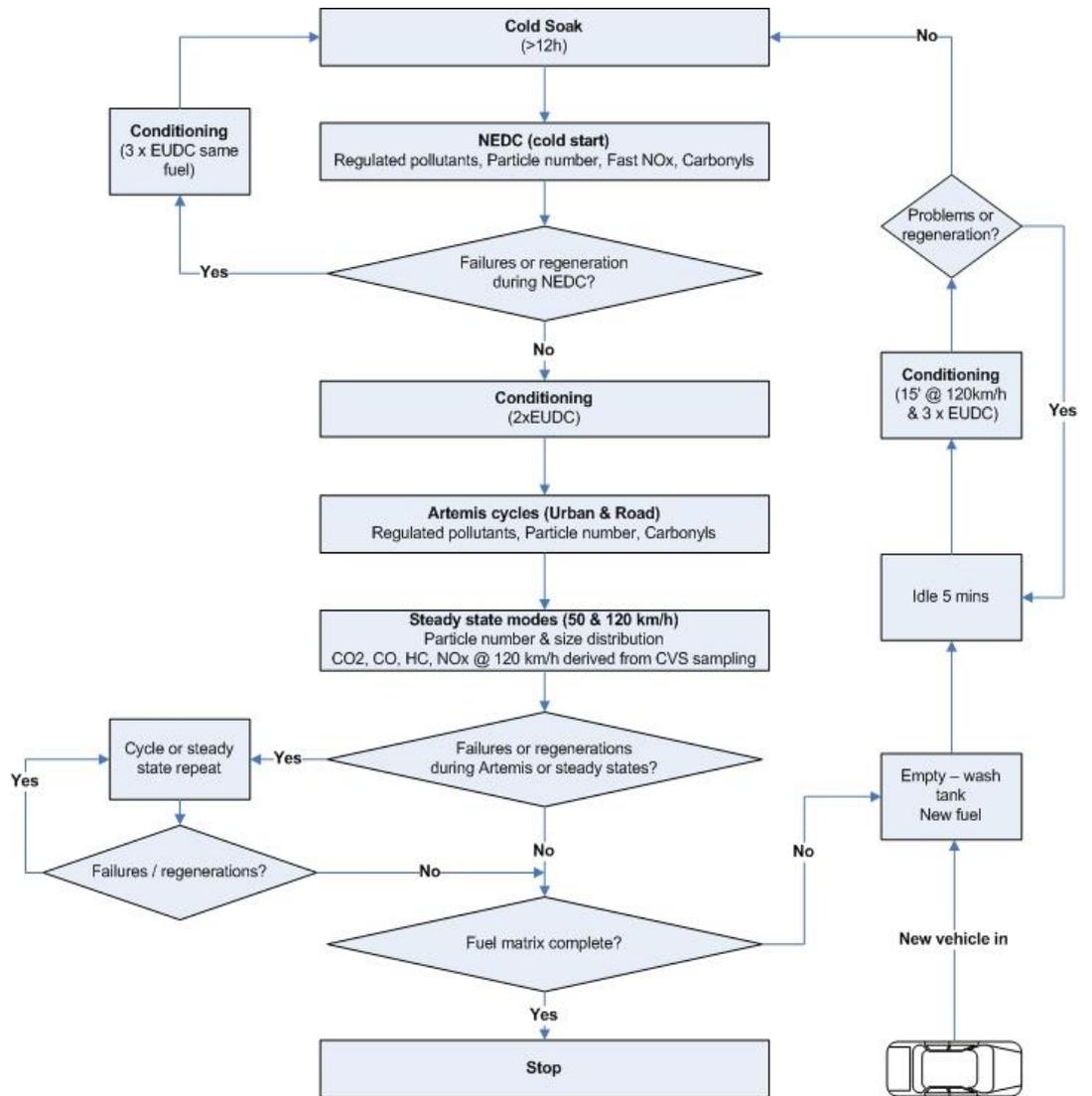
Vehicle 3 is the 'golden vehicle' that was previously used as the round-robin test vehicle in the European Particle Measurement Programme (PMP) [15].

APPENDIX 2 TEST FUELS³

Fuel Property	Units	Test Method	B0	B10	B30	B50
Cetane Number		EN ISO 5165	53.2	53.6	53.8	53.9
Cetane Index (4 variable)		ASTM D4737	60.3	59.1	57.6	56.1
Density at 15°C	kg/m ³	EN ISO 12185	823.1	829.1	841.0	853.0
Initial Boiling Point	°C	EN ISO 3405	202.8	204.9	206.1	214.3
Distillation T50	°C	EN ISO 3405	271.2	277.2	293.6	312.0
Distillation T95	°C	EN ISO 3405	316.5	330.5	339.3	344.3
Final Boiling Point	°C	EN ISO 3405	326.5	337.3	344.2	352.1
Flash point	°C	EN ISO 2719	80	76	91	97
CFPP	°C	EN 116	-22	-21	-25	-21
Cloud Point	°C	EN ISO 3015	Not Measured	-18	-14	-11
Viscosity at 40°C	mm ² /s	EN ISO 3104	2.661	2.785	3.081	3.410
Total Aromatics	% m/m	EN 12916	22.3	18.3	15.0	10.6
Mono-aromatics	% m/m	EN 12916	20.8	17.1	14.1	9.9
Di-aromatics	% m/m	EN 12916	1.4	1.2	0.9	0.7
Tri-aromatics	% m/m	EN 12916	0.1	0.1	<0.1	<0.1
Polycyclic Aromatic Hydrocarbons	% m/m	EN 12916	1.2	1.2	0.9	0.6
FAME	% vol	EN 14078	<0.1	10.7	30.6	50.9
Sulphur	mg/kg	EN ISO 20846	<3	<3	<3	<3
Copper Corrosion 3h @ 50°C		EN ISO 2160	1A	1A	1A	1A
Oxidation Stability	g/m ³	EN ISO 12205	4	0.2	0.1	0.1
Oxidation Stability	hrs	EN 15751	Not Measured	64.6	31.3	21.5
Water	mg/kg	EN ISO 12937	22	37	138	138
Ash Content	% m/m	EN ISO 6245	0.002	<0.001	<0.001	<0.001
HFRR	Micron	EN ISO 12156	227	156	171	168

³ Single measurements as provided on the Certificate of Analysis from the fuel blender

APPENDIX 3 FLOWCHART FOR VEHICLE TEST PROTOCOL



APPENDIX 4 SAMPLING PROTOCOL

PM sampling

Particulate Matter (PM) sampling was performed following the specifications of the PMP protocol [15]. A separate weighed filter paper was used for each of the three driving cycles (NEDC, ARTEMIS Urban, and ARTEMIS Road) for measuring PM emissions.

In addition to the vehicle tests, six additional blank tests were performed for determining background levels for both PM and carbonyl compounds (see below). During these six tests, the sampling procedure for PM and carbonyl compounds was repeated identically as for vehicle measurements except that the CVS was disconnected from the vehicle tailpipe.

After weighing and calculating PM emissions, the filters were packed in order to be used for determining the soluble organic fraction (SOF) of the PM, the ionic species (anions) and elemental carbon (EC) by difference. The PM filters and cartridges for capturing the carbonyl compounds were stored according to Concaawe's recommendations and were sent for analysis periodically as the measurements progressed. All of the PM filters, including those from the ARTEMIS Road driving cycle, were stored in the same way in case they were needed for future measurements.

The SOF analysis and measurements of aldehyde and ketone emissions were completed by the Environmental Pollution Control Laboratory (EPCL) of the Department of Chemistry at the Aristotle University of Thessaloniki, Greece.

Filter Preparation

Pallflex TX40 Fluorocarbon coated glass fibre filters were used having a filter diameter of 47 mm. The filter batch was always recorded.

The filters, from blank and test runs, were conditioned to the same temperature and humidity in a clean room, under controlled temperature ($22\pm 3^{\circ}\text{C}$) and humidity conditions ($45\pm 8\%$), according to the PMP regulatory procedure. The filters were placed on a grounded aluminium plate during the conditioning period and were covered by a perforated aluminium cover in order to protect them from dust while keeping them in contact with the clean room environment at the same time.

Three reference filters were also kept in the clean room and were weighed at the same time as the blank and loaded filters, in-line with the PMP regulation. Each sample filter, from blank and test runs, was weighed more than once during its conditioning period. The conditioning period was between 2 and 80 h as per the PMP procedure. However, because of the subsequent non-regulated pollutant analyses, the loaded filters were normally kept in the clean room between 24 and 48 h and were then immediately stored, in order to ensure no change of the PM composition.

Microgram balance

The analytical balance used was a Mettler-Toledo UMX2 with 0.1 μg resolution. The balance was grounded by placing it on an anti-static plate. The particle filters were placed on a grounded aluminium mat in order to avoid static charge. A reference weight was weighed during the testing period along with the reference filters. The

balance precision (standard deviation) for the reference weight was 0.9 μg during the entire measuring period.

Filter storage

After their final weighing, the filters were packed in order to be stored and then sent for PM speciation analyses. The filter paper was folded in half with the side containing the PM deposit on the inside. The folded filter was wrapped in aluminium foil and the foil-wrapped filter was placed in a suitably-sized self-sealing plastic bag. The plastic bag was stored in the chilled area of a refrigerator.

Each bag was separately labelled. The bag labelling included the filter code number which identified a unique test and the conditioning time of the loaded filter. The date of the final weighing, the weight of the filter, the batch number of the filter, and the initials of the person preparing the filter sample bag were also recorded.

PN sampling

The particle sampling system follows exactly the latest PMP recommendations (Regulation R83, Annex 4, **Appendix 5**). The general layout is shown in **Figure 4** which consisted of a metallic tube (probe), the dilution tunnel, a particle pre-classifier (cyclone), and a particle measurement system comprising a Volatile Particle Remover (VPR) upstream of the Condensation Particle Counter (CPC 3010, TSI). The probe sampled directly from the dilution tunnel and was one piece with the particle transfer tube.

Sample Probe

The particle sampling probe was installed near the dilution tunnel centre line, 10 tunnel diameters downstream of the gas inlet, facing upstream into the tunnel gas flow with the sampling probe tip axis parallel to the axis of the dilution tunnel. The probe had an inner diameter of 12mm and was not heated or insulated.

The distance from the sampling tip to the point at which the probe left the dilution tunnel was 200mm and the distance from the sampling tip to the entrance to the particle pre-classifier (cyclone) was 500mm, that is, with the total distance less than 1000mm, which is the PMP maximum length. For the flow rate through the sample probe that was used in this study (~ 40 lpm), the Reynolds number was approximately 4300 and the residence time was ~ 0.1 sec.

Particle Pre-classifier

The 50% cut-point of the cyclone particle pre-classifier (*URG-2000-30EP*) was $8\mu\text{m}$ at the flow rate established through the cyclone (~ 40 lpm). The particle sample flow rate was then adjusted to 6 lpm. The residence time into the cyclone was estimated to be ~ 0.5 sec.

Volatile Particle Remover (VPR)

The residence time of the exhaust gas up to the entrance of the VPR was estimated to be 1.9s (PTS: sample probe ~ 0.1 s, cyclone ~ 0.5 s, 1 m Tygon tube ~ 0.8 s, first diluter ~ 0.5 s) ≤ 3 sec, which is the limit according to the PMP protocol specifications.

The VPR consisted of:

First Particle Number Diluter: The exhaust aerosol, after exiting the cyclone, was primarily diluted by an ejector type diluter, manufactured by Dekati Ltd. The diluter body was kept at 150°C by means of an electrically heated blanket. The dilution air was also heated to 150°C before entering the diluter by means of a heated air line. The exact dilution ratio of the diluter was determined at the end of each testing day by measuring the CO₂ concentration upstream and downstream of the diluter. The measured dilution ratio was ~14.5:1, using pressurized air at 2 bar overpressure.

Evaporation tube: The evaporation tube had an inner diameter of 10mm and a length of 25cm. The wall temperature was set at 310°C and was kept constant in the range of +/-5°C. The residence time in the evaporation tube was estimated equal to 1s.

Second Particle Number Diluter: The secondary diluter was also supplied by Dekati Ltd. and was also an ejector type diluter. Its dilution ratio was determined by means of flowrate measurements and was found to be approximately 10:1, varying only slightly from day to day (<0.2:1). The supplied pressurized air was set at 2 bar overpressure.

Particle Counter: A particle counter (TSI 3010 CPC, T90 ~4.5s) was connected downstream of the second diluter. The outlet tube (OT) had a minimum length in order to retain the residence time of the exhaust equal to 0.5s, which is less than the PMP protocol requirement of 0.8sec. The CPC temperature difference (temperature difference between the saturator and the condenser) was set at 9°C, shifting in this way the lower detectable particle diameter from 7nm to 23nm. The total dilution of the PMP system (downstream of the CVS) to the CPC was 145:1 for Vehicles 1 and 3. The particle concentration at the inlet of the CPC never exceeded 10⁴ particles/cm³, avoiding in this way the coincidence correction and any induced errors. For Vehicle 2, more dilution was needed in order to keep the particle concentration within the measuring range of the CPC. This extra dilution stage is described below.

In-house dilution stage: The extra dilution was achieved by installing an in-house dilution stage in series between the evaporator tube and the second diluter. The in-house diluter consisted of a mixing chamber and a HEPA capsule filter and provided an additional dilution ratio of about 10:1. Hence, for Vehicle 2, the total DR downstream of the cyclone was ~1450:1. The total residence time of the exhaust in the sampling system (PTS+VPR+OT) was 3.4s. This residence time plus the T90 of the PNC was less than 20s (~7.9s) as required.

Particle sampling - remark

The PMP sampling system used in this study is identical to the heated version of the "Dual Ejector System" [34]. As the authors of that paper presented, this is probably the best performing PMP system, exhibiting the lowest particle losses, since the PN concentration reduction factor is the lowest of all candidate systems (1.02) and exhibits the lowest variation with particle size (from 1.00 to 1.08). In addition, daily measurements of the dilution factor in the working ranges of both diluters were performed to provide the exact dilution ratio.

Carbonyl compound sampling

For capturing the carbonyl compound from the exhaust, DNPH cartridges were used with one cartridge per NEDC and ARTEMIS Urban test. Diluted exhaust gas was drawn from the CVS through these cartridges at a rate of 150mL/min using a pump. As for PM filters, the used DNPH cartridges were sealed after the test and put into

the refrigerator immediately after each sample was collected. Background measurements were conducted as described above. Cartridges were periodically sent for analysis as the vehicle testing progressed (see **Appendix 5**).

All measurements

Table A4-1 summarizes all of the measurements that were completed in this study at each driving condition.

Table A4-1 Summary of all measurements

	UDC	EUDC	NEDC	ARTEMIS Urban	ARTEMIS Road	50 km/h	120 km/h	Conditioning
ECU data			X	X	X	X	X	X
CO ₂	X	X	X	X	X	*	*	*
CO	X	X	X	X	X	*	*	*
HC	X	X	X	X	X	*	*	*
NO _x	X	X	X	X	X	*	*	*
Modal NO _x			X					
PM			X	X	X			
PM speciation			X	X				
PN total			X	X	X	X	X	
PN solid			X	X	X	X	X	
Particle size distribution						X	X	
Carbonyl compounds			X	X				

* Modal data for these emissions were collected for quality control purposes only and used for calculating indicative emissions over the 120 km/h steady state operating point.

APPENDIX 5 PM ANALYSIS PROCEDURES

Ion chromatography (IC) method

The filter halves left over from the procedure described in **Section 3** were taken out of the refrigerator and conditioned for 24 hours at $20\pm 1^\circ\text{C}$ and $50\pm 5\%$ relative humidity. The filters were then weighed on a balance of $1\mu\text{g}$ readability in order to estimate the fraction of the PM used for anion analysis. The filters were then extracted in an ultrasonic bath for 45 minutes using a mixture of 0.95ml deionized and double distilled water and 0.05ml methanol of analytical grade. The use of methanol reduces the hydrophobicity of the samples resulting from the presence of the elemental carbon [35]. The extracts were then filtered through $0.45\mu\text{m}$ membranes, placed in sealed containers with appropriate labels and stored at the refrigerator below 5°C until the IC analysis. The same procedure was applied to 4 different blank filters in order to ensure that an appropriate blank value was used. All glassware and implements used to handle the samples were cleaned carefully in order to avoid any organic or inorganic contaminants.

An Allsep Anion 7u chromatographic column and a mixture of phthalic acid with 4mM lithium hydroxide were used for the determination of the anions. The quantification of nitrates and sulphates was based on 5-point calibration curves constructed from solutions of known concentrations. The retention time of nitrates was 5.5min and that of sulphates was 12min. Unused filters were also analyzed using the same procedure and the chromatograms of the test samples were corrected based on the unused filter results. The IC method's limits of detection are $1.358\mu\text{g}/\text{filter}$ for nitrates and $2.661\mu\text{g}/\text{filter}$ for sulphates. The relative standard deviations were 4.63% and 3.48%, respectively.

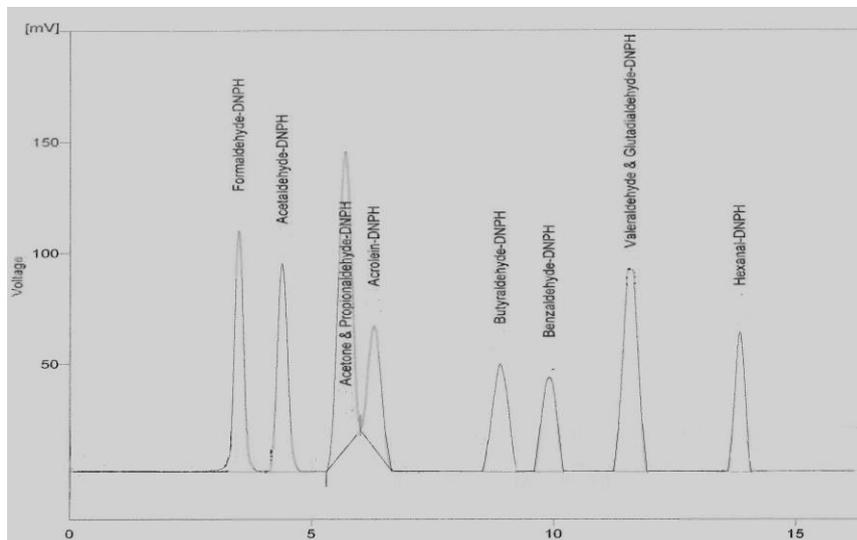
Extraction and analysis procedure for carbonyl measurements

Carbonyl compounds were recovered from the DNPH-coated cartridges using extraction with 5mL acetonitrile (CH_3CN) according to procedures previously developed (EPA Method TO-11A [14]). The extracts were stored at -18°C until they were analyzed by High Performance Liquid Chromatography (HPLC).

Carbonyls compounds were analyzed by HPLC with UV detection (360 nm). Separation was performed on two $5\mu\text{m}$ Macherey Nagel Nucleosil 100-5 C18 HD columns (125 x 3 mm) arranged in series. The chromatographic conditions were based on the EPA Method TO-11A and were further optimized in order to provide adequate separation and resolution. The mobile phase was a $\text{CH}_3\text{CN}-\text{H}_2\text{O}$ gradient comprising 60% CH_3CN over 7 min, 60–85% CH_3CN between 7 and 15 min, 85–100% CH_3CN between 15-16 min, 100% CH_3CN between 16-20 min, 60-40% CH_3CN between 20-21 min, 60-40% CH_3CN between 21-25 min (flow rate 0.8ml/min, injection volume $30\mu\text{l}$). The system was calibrated with a custom solution standard (LGC Standards Promocher), containing the following carbonyl compounds: acetaldehyde, formaldehyde, acetone, propionaldehyde, acrolein, n-butylaldehyde, benzaldehyde, valeraldehyde, glutaraldehyde, hexanaldehyde. Acetone and propionaldehyde, as well as valeraldehyde and glutaraldehyde were not satisfactorily separated and, therefore, were added together.

The carbonyl compounds in the sample were identified and quantified by comparing their retention times and area counts with those of standard DNPH derivatives. A chromatogram of a standard solution is shown in **Figure A5-1**.

Figure A5-1 HPLC of a standard solution of different carbonyl compounds



APPENDIX 6 STATISTICAL DATA ANALYSIS

This Appendix provides additional information on the statistical analysis methods discussed in **Section 3.5**.

Outlier and trend detection

The PM and PN data were checked for possible outliers by examining studentized residuals (residuals divided by their standard errors) in an analysis of (co)variance models fitted to the measured emissions for a particular vehicle and cycle on both the natural and log-transformed scales. In this study, a one-way ANOVA model was fitted to each vehicle × cycle × emission combination with emission or ln(emission) as the response variable and fuel as the classifying factor. Time trends were sought by treating the test order as a covariate.

The studentized residuals were compared against the upper 5% and 1% points as tabulated in [36]. Questionable results were queried with the test laboratory and were not rejected unless there were sound engineering reasons to believe that something unexpected had occurred with that particular test. Following this review of the data, 12 results were rejected, as follows:

Vehicle 1

- Total PN result in ARTEMIS Road on 3 March 2009 on B0 (abnormally low)

Vehicle 2

- PM result in NEDC on 28th April 2009 on B50 (abnormally low)
- Total PN results on 7 (NEDC & ARTEMIS Urban on B0) and 8 April 2009 (NEDC on B10) (all abnormally low)
- Total PN results on 24 April 2009 in the ARTEMIS Road, 50 and 120 km/h on B0 (all abnormally low)

Vehicle 3

- Total PN result over the NEDC on 25 May 2009 on B0 (abnormally low)
- Solid PN result at 50 km/h on 25 May 2009 on B0 (abnormally high)
- Solid PN result in ARTEMIS Urban on 1 July 2009 on B50 (abnormally low)
- Solid PN result in ARTEMIS Urban on 6 July 2009 on B0 (abnormally low)

A statistically significant time trend (at $P < 1\%$) was found in just one of the 39 PM, total PN, and solid PN data sets (3 vehicles × 3 measurements × up to 5 cycles). This was for:

Vehicle 3

- Total PN results at 120 km/h (number of particles decreases with time)

Using statistical “analysis of covariance” techniques, this set of data points was adjusted to where they might have been if all of the tests had been conducted halfway through the test programme. The trend was corrected on the logarithmic scale and the adjusted means were then converted back to the original #/km scale to form corrected geometric means.

It can be seen from the total PN table in **Appendix 7** that the trend correction had little effect on the mean emissions because a statistically robust randomised block design was used to establish the fuel and vehicle testing order. Nevertheless, the trend correction reduced standard errors and error bars and thus helped improve

the discrimination between the different fuels. For this reason, corrected means are shown for this variable in **Figure 4**.

Arithmetic and geometric means and error bars

As in previous Concaawe studies, arithmetic means have been used in this report to summarize PM emissions, even though the standard deviation vs. mean plots in **Appendix 9** suggest that these measurements follow a lognormal distribution. Geometric (i.e. logarithmic) means give excellent comparisons between fuels on a percentage basis but have the disadvantage of underestimating total emissions to the atmosphere.

However, geometric means did have to be used to average both total and solid PN measurements because PN can differ by one or more orders of magnitude. This means that arithmetic means can be dominated by one or two very high results and can be unrepresentative of the main body of data.

Each vehicle × cycle × emission measurement data set was analysed separately. The arithmetic mean PM emissions, and their standard errors, were estimated for the various fuels from a weighted analysis of variance or covariance in which each measurement was assigned a weight equal to:

- weight = $1 / (\text{mean emission for that fuel and vehicle})^2$

to take account of the lognormality in the data (see [36], Annex 05).

In the bar charts presented in **Section 4.1.1**, the error bars show the

- mean value $\pm 1.4 \times$ standard error of mean

The 1.4 factor was chosen for consistency with both the EPEFE [20] and recent Concaawe reports [16,17,18,19]. The original rationale was that when two fuels were significantly different from one another at $P < 5\%$, their error bars would not overlap. This 1.4 factor also gave 84% confidence that the true mean lay within the limits shown.

Error bars based on a factor 1.4 err on the side of being slightly too narrow for determining significant differences in the present study because fewer tests were carried out. Such an interpretation would require error bars based on a factor between 1.52 and 1.56, depending on the exact number of valid tests and whether or not a trend correction has been applied. Therefore, there needs to be a sizeable gap between the error bars for two fuels for their means to be considered significantly different from one another at $P < 5\%$.

Carbonyl compounds

Many of the measurements of carbonyl compounds described in **Section 3.3.3** were at or below the limit of detection with some results being zero. As a consequence, most of the measured variables were not of sufficient consistency to warrant our usual statistical treatment. Analyses based on the usual lognormality assumptions cannot readily be applied to data sets containing very low numbers. Standard outlier and trend correction techniques would not be reliable and error bars based on estimated standard errors would be too sensitive to aberrant values to provide reliable confidence limits for true emission levels.

Therefore carbonyl emissions have been summarized by calculating simple arithmetic means of the raw data. Error bars have been plotted showing the minimum and maximum measured values over the 4 or 5 repeat tests rather than the usual ± 1.4 SE error bars used here for particulate emissions and consistently with other recent Concaawe reports. The min-max error bars have been plotted in grey in **Figures 10 to 12** to distinguish these from the more usual kind.

APPENDIX 7 AVERAGE PM AND PN (TOTAL AND SOLID) EMISSIONS

The tables in this appendix give the average PM and PN (total and solid) emissions for each vehicle, fuel and cycle. These values are the averages of five measurements for the B0 fuel and four measurements each for the B10, B30, and B50 fuels. Arithmetic means are reported for PM emissions while geometric means are reported for PN emissions. Where trend corrections have been made, both the corrected and uncorrected averages are given; see **Appendix 6** for more details.

PM (g/km) – Arithmetic Means

Vehicle	Fuel	NEDC	ARTEMIS Road	ARTEMIS Urban
		Uncorrected	Uncorrected	Uncorrected
1	B0	0.00066	0.00094	0.00337
1	B10	0.00071	0.00103	0.00330
1	B30	0.00055	0.00080	0.00222
1	B50	0.00060	0.00086	0.00274
2	B0	0.03338	0.02693	0.05113
2	B10	0.02780	0.02509	0.04461
2	B30	0.01910	0.02155	0.03201
2	B50	0.01593	0.01800	0.03404
3	B0	0.00078	0.00170	0.00220
3	B10	0.00080	0.00188	0.00282
3	B30	0.00069	0.00126	0.00202
3	B50	0.00068	0.00276	0.00229

Total PN (#/km) - Geometric Means

Vehicle	Fuel	NEDC	ARTEMIS Road	ARTEMIS Urban	Steady State 50 km/h	Steady State 120 km/h	
		Uncorrected	Uncorrected	Uncorrected	Uncorrected	Uncorrected	Corrected
1	B0	4.075E+10	1.309E+10	2.423E+10	1.897E+09	8.738E+09	
1	B10	6.706E+10	7.336E+09	3.144E+10	5.736E+09	6.028E+09	
1	B30	3.814E+10	1.466E+10	7.658E+10	1.166E+09	1.493E+09	
1	B50	2.282E+10	1.043E+10	4.298E+10	3.514E+09	9.538E+10	
2	B0	1.352E+14	6.427E+13	1.408E+14	6.677E+13	1.533E+14	
2	B10	1.226E+14	8.901E+13	1.166E+14	7.305E+13	1.906E+14	
2	B30	9.088E+13	7.209E+13	9.562E+13	6.762E+13	1.166E+14	
2	B50	6.489E+13	5.448E+13	6.683E+13	5.676E+13	7.587E+13	
3	B0	1.892E+11	1.561E+09	3.138E+09	1.897E+09	1.919E+09	1.919E+09
3	B10	1.712E+11	8.521E+08	2.955E+09	5.736E+09	2.811E+09	3.010E+09
3	B30	1.877E+11	2.817E+09	1.494E+09	1.166E+09	7.300E+09	6.817E+09
3	B50	1.990E+11	3.026E+09	3.013E+09	3.514E+09	1.231E+09	1.231E+09

Solid PN (#/km) - Geometric Means

Vehicle	Fuel	NEDC	ARTEMIS Road	ARTEMIS Urban	Steady State 50 km/h	Steady State 120 km/h
		Uncorrected	Uncorrected	Uncorrected	Uncorrected	Uncorrected
1	B0	3.221E+10	5.914E+09	2.625E+10	7.883E+09	5.341E+09
1	B10	4.366E+10	6.365E+09	4.259E+10	2.629E+09	1.213E+10
1	B30	3.434E+10	7.487E+09	5.179E+10	5.113E+09	3.985E+09
1	B50	1.648E+10	9.590E+09	2.819E+10	7.700E+09	4.965E+09
2	B0	2.982E+13	2.711E+13	3.595E+13	2.051E+13	4.478E+13
2	B10	2.971E+13	2.678E+13	3.562E+13	2.058E+13	4.222E+13
2	B30	2.320E+13	2.106E+13	2.812E+13	1.903E+13	3.514E+13
2	B50	2.102E+13	1.918E+13	2.493E+13	1.791E+13	3.006E+13
3	B0	4.271E+09	2.937E+08	1.052E+09	3.539E+08	2.390E+08
3	B10	8.027E+09	3.005E+09	1.322E+09	3.317E+08	1.559E+08
3	B30	7.168E+09	6.766E+08	1.476E+09	4.220E+08	2.342E+08
3	B50	2.037E+09	2.039E+09	1.676E+09	4.723E+08	2.230E+08

APPENDIX 8 CARBONYL COMPOUNDS

Carbonyl Compounds (mg/km) – Arithmetic Means - NEDC

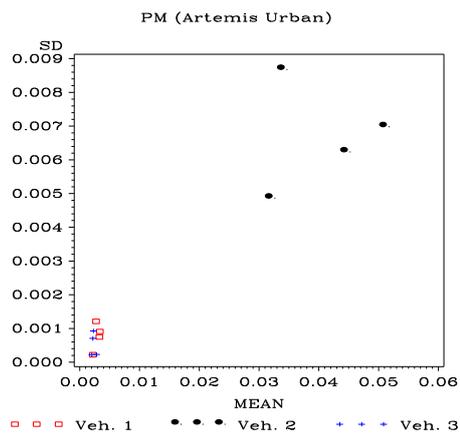
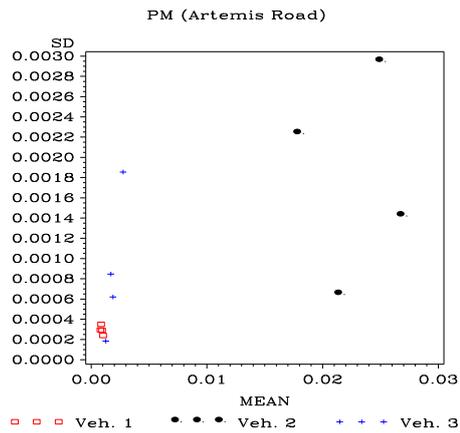
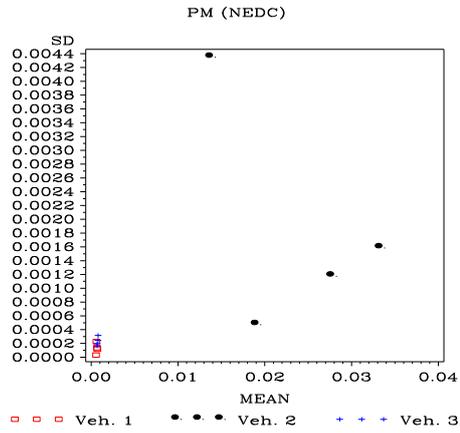
Cycle	Vehicle	Fuel	Formaldehyde (mg/km)	Acetaldehyde (mg/km)	Acetone & Propionaldehyde (mg/km)	Acrolein (mg/km)	Butyraldehyde (mg/km)	Benzaldehyde (mg/km)	Valeraldehyde & Glutaraldehyde (mg/km)	Hexanal (mg/km)
NEDC	1	B0	0.20320	0.12084	0.39237	0.00805	0.05329	0.00156	0.00048	0.00155
	1	B10	0.34380	0.07832	0.32775	0.00654	0.02769	0.00334	0.00038	0.00181
	1	B30	0.45070	0.15816	0.32258	0.01245	0.01620	0.00228	0.00086	0.00795
	1	B50	0.77964	0.14517	0.42784	0.00356	0.00830	0.00106	0.00098	0.00405
	2	B0	0.55604	0.08366	0.94629	0.00708	0.06606	0.00132	0.00130	0.00264
	2	B10	0.58474	0.18271	0.48843	0.01368	0.10935	0.00161	0.00330	0.00318
	2	B30	1.00686	0.44089	0.42622	0.00627	0.08066	0.00559	0.00092	0.00351
	2	B50	0.35829	0.23389	0.44448	0.00506	0.19395	0.00289	0.00262	0.00523
	3	B0	0.45847	0.30665	2.42895	0.02307	0.08778	0.00169	0.00088	0.00255
	3	B10	0.49105	0.43468	2.00201	0.01750	0.08394	0.00611	0.00054	0.00576
	3	B30	0.47685	0.11053	1.08213	0.02539	0.15710	0.00326	0.00140	0.01611
	3	B50	0.51432	0.27760	0.49167	0.03328	0.08613	0.00149	0.00057	0.00166

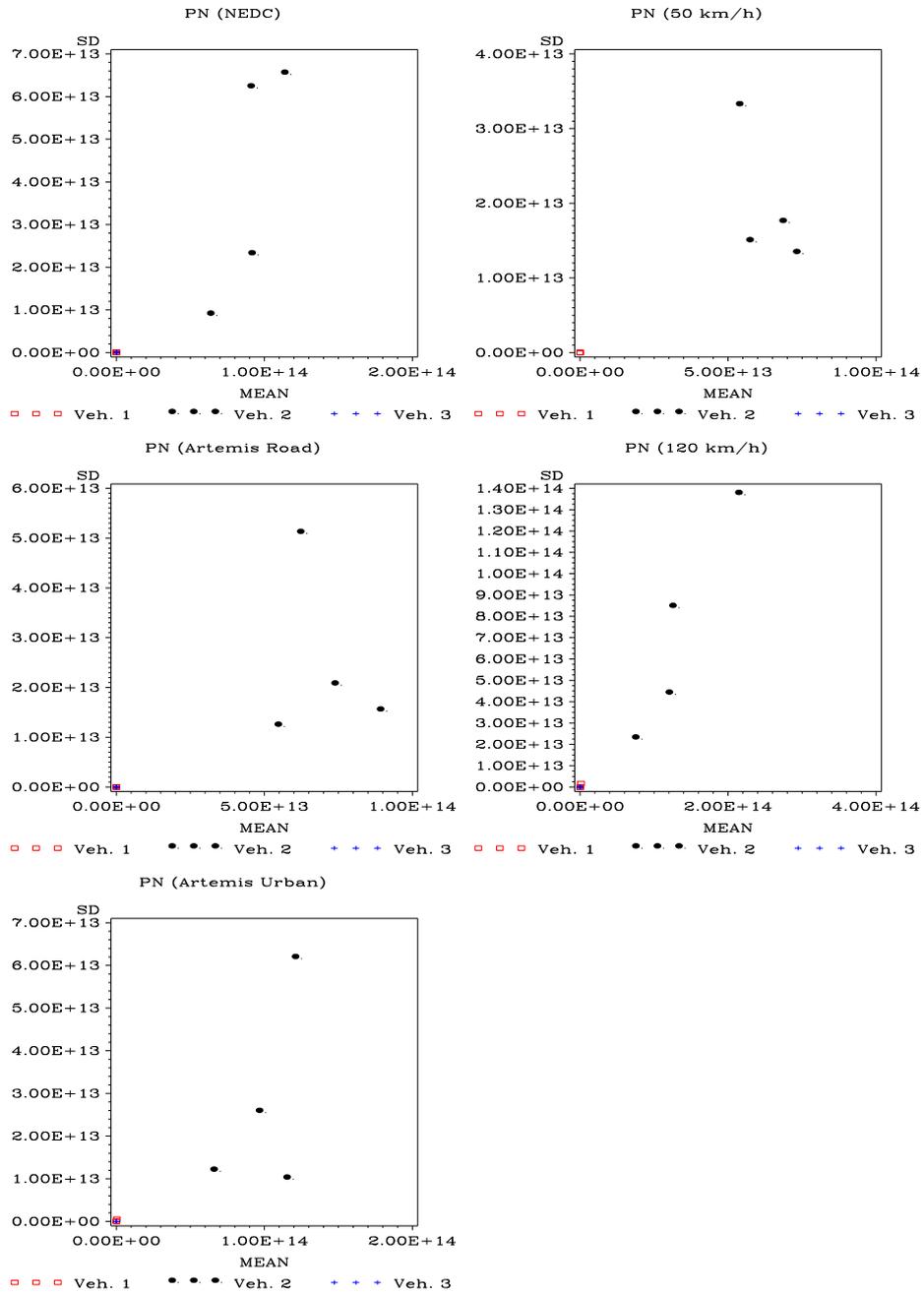
Carbonyl Compounds (mg/km) – Arithmetic Means – ARTEMIS Urban Cycle

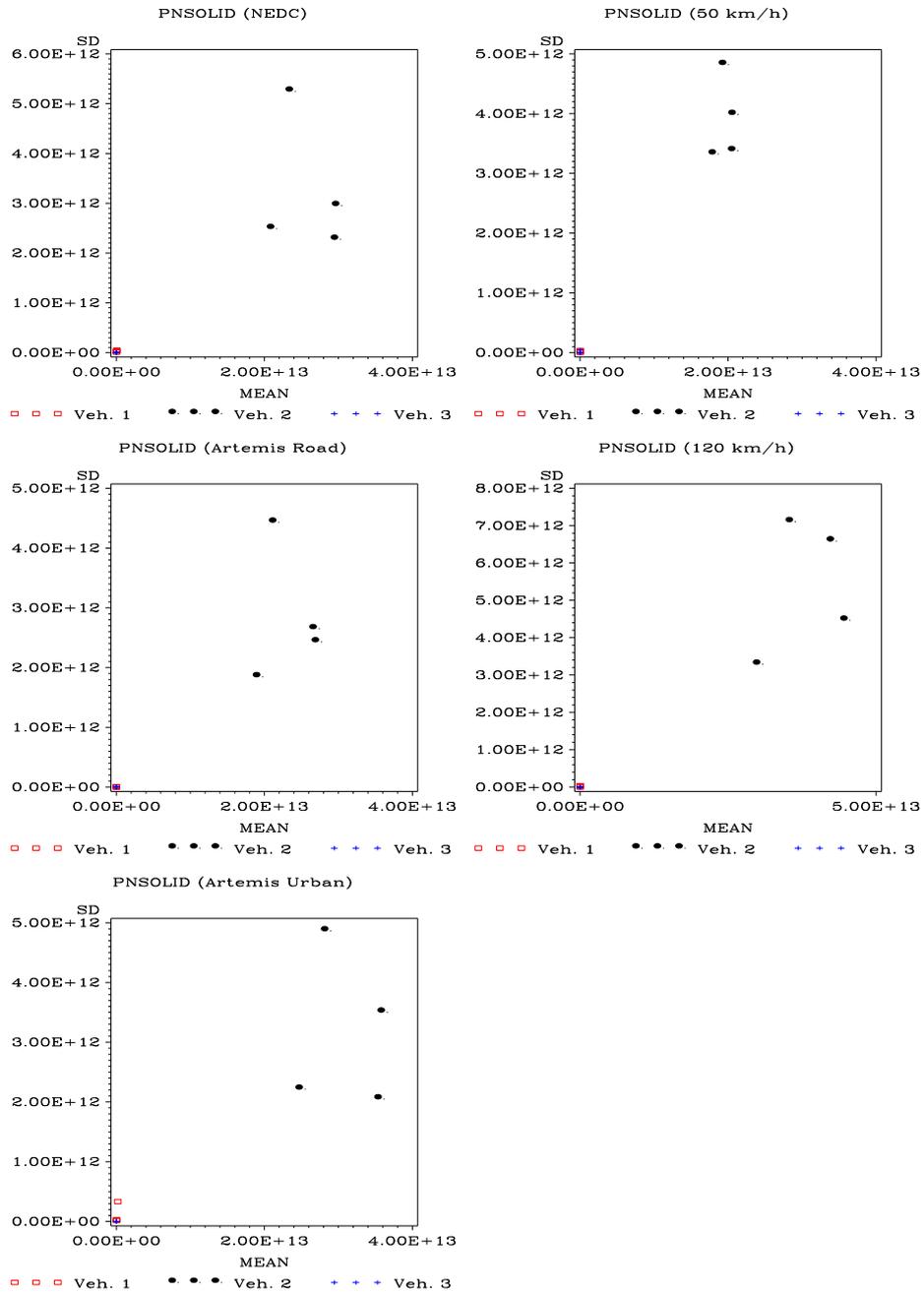
Cycle	Vehicle	Fuel	Formaldehyde (mg/km)	Acetaldehyde (mg/km)	Acetone & Propionaldehyde (mg/km)	Acrolein (mg/km)	Butyraldehyde (mg/km)	Benzaldehyde (mg/km)	Valeraldehyde & Glutaraldehyde (mg/km)	Hexanal (mg/km)
ARTEMIS Urban	1	B0	4.37655	0.17261	0.82352	0.01742	0.12365	0.00613	0.00169	0.00362
	1	B10	5.27189	0.11185	0.70502	0.01366	0.01803	0.00372	0.00155	0.01708
	1	B30	6.29973	0.19658	0.59963	0.01069	0.14637	0.00574	0.00674	0.00558
	1	B50	5.6097	0.14405	0.97958	0.00000	0.11334	0.01249	0.00083	0.02667
	2	B0	1.05501	0.08009	0.52081	0.01776	0.24796	0.00926	0.00114	0.01706
	2	B10	1.88834	0.15126	0.99954	0.00874	0.10441	0.01419	0.00214	0.01717
	2	B30	0.62172	0.34184	4.88025	0.01426	0.13114	0.00285	0.00046	0.01686
	2	B50	0.77286	0.81898	1.31460	0.08011	0.28609	0.00876	0.00081	0.01786
	3	B0	1.64352	0.26427	0.53251	0.03938	0.14338	0.00179	0.00142	0.00262
	3	B10	0.72855	0.28883	0.56978	0.02814	0.17528	0.00766	0.00179	0.01121
	3	B30	1.22011	0.38062	0.65981	0.04977	0.23123	0.00317	0.00254	0.01976
	3	B50	1.81115	0.18125	2.17827	0.01891	0.20545	0.00301	0.00283	0.01045

APPENDIX 9 PARTICULATES: MEANS AND STANDARD DEVIATIONS

These plots are based on all of the measured data before trend correction or the deletion of outliers.

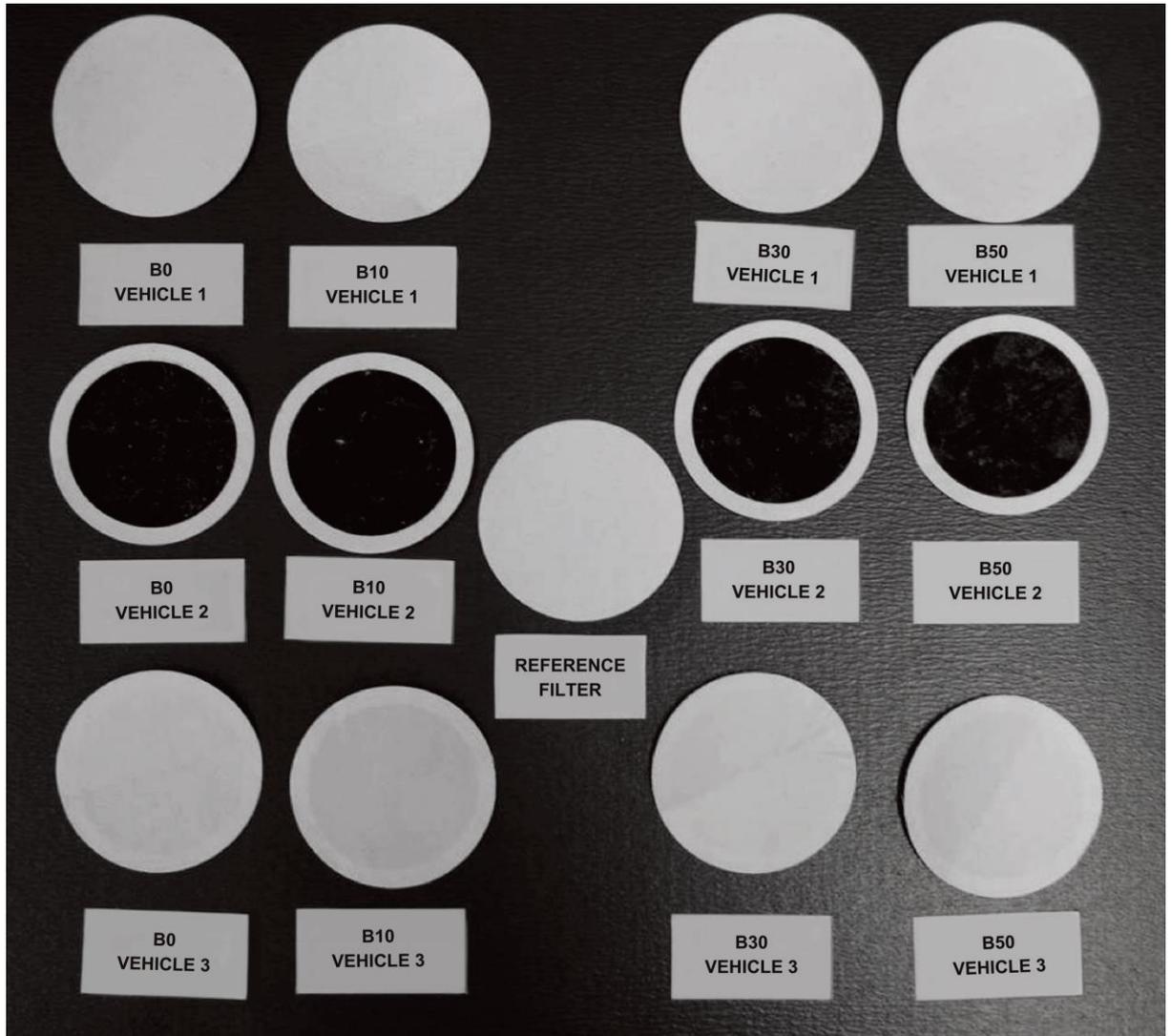






APPENDIX 10 PHOTOS OF PARTICULATE FILTERS

Vehicles 1 and 3 were equipped with Diesel Particulate Filters (DPFs) while Vehicle 2 was not.



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