Ethanol fuel content impact on regulated and non-regulated emissions on EU6c and EU6d-Temp vehicles

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Abstract: Vehicle transportation is under fast transformation to ensure low greenhouse emissions. The European Union aims at a carbon neutral industry by 2050. To ensure reaching this goal, several technological solutions are to be implemented with both short and long term impact. The integration of advanced low carbon biofuels has been studied as a prospective path towards such goal. Ethanol is the predominant biofuel that has been developed and integrated into the transportation sector to date. Under this context, this study aims at analysing the impact of fuels' ethanol content over regulated and non-regulated emissions, and recent EU6 vehicle technology. Testing has been carried out over 2 Eu6d-TEMP vehicles adapted with E85 conversion system, and a flexi-fuel vehicle EU6c. Two complementary non-regulated emissions measurements systems have been implemented, through laboratory techniques and temporal resolution ionic cyclotronic resonance mass spectrometry (BTrap). Results indicate that the ethanol fuel content has an impact over regulated and non-regulated emissions for high concentrations of 50 and 85 % vol. Mainly, an increase of unburned ethanol, methane and aldehydes is observed at 50 % vol ethanol or above. Unburned aromatics are reduced with increasing fuel's ethanol content. For fuel E85, regulated emissions remain within regulation levels, with the exception of NO_x emissions increase for the EU6d-TEMP direct injection thermal vehicle with conversion Kit. The impact of ethanol fuel content over CO_2 emissions is variable, and a clear tendency is only observed for E85, inducing a reduction ranging from 3 to 7 %. These tendencies have been noticed independently of the vehicle or conversion system technology.

Keywords: Flexi-fuel, E85 conversion kit, non-regulated emissions, aldehydes, volatile organic carbon.

1. Introduction

European fuel regulations for spark-ignition engines allow the integration of ethanol as a biofuel. It is present up to a level of 10 % vol. in Unleaded 95-E10, and at a level between 65 and 85 % vol. in Superethanol-E85. The use of Superethanol-E85 is authorised on original flex-fuel vehicles, or on certain vehicles fitted with approved E85 conversion kits. The installation of conversion kits has been authorised since 2017, for specific engine technologies, and conditions of use and approval.

Ethanol's physical and thermal properties are similar to those of gasoline. Its composition grants it lower CO_2 emissions, and it's high octane rating allows for higher combustion efficiency and performance [1–3]. These characteristics have popularised the use of ethanol in recent years. However, its lower heat of combustion, reduced vaporisation under cold operation and risk of oil dilution are disadvantages associated to the use of ethanol [4–6].

There are many studies on the influence of ethanol on the emissions of flexi-fuel spark-ignition vehicles. However, very few have studied the impact of Superethanol-E85 on hybrid vehicles, or on vehicles equipped with adapted E85 conversion kits. The need for more data on emissions associated with the use of Superethanol-E85 fuel is mentioned, for example, in a CEN report [7]. This report highlights the need to know the emissions of unburned hydrocarbons both qualitatively and quantitatively for new fuels. One of these issues is related to cold emissions when the catalyst is not active. These emissions can be higher with fuels containing high levels of ethanol. Qualitatively, it is interesting to know what types of molecules are counted in the unburned hydrocarbons. Indeed, it would appear that some studies count ethanol in the exhaust as a hydrocarbon, whereas chemically it is an alcohol. It is important to know whether emissions of "real" hydrocarbons (of the formula CxHy) increase or decrease with the level of ethanol in the fuel by chemical analysis of the exhaust gases. The impact of fuels on particulate emissions is not always clear either. As a consequence, more recent works on EU6c vehicle technology suggested the feasibility of the introduction of ethanol up to 20 % vol. at reduced regulated emission impact. The study also suggested that if non-regulated emissions increased with ethanol, these remained at low level [8].

The vast majority of studies show a decrease in particulate matter when the ethanol content of fuels is increased [9–12] and others show the opposite effect [13, 14], notably due to an increase in fine particulate matter (below 30 nm) [15]**Erreur ! Source du renvoi introuvable.**. Furthermore, the observed effects on particulate matter in number and mass are not always comparable. This may indeed depend on the technology (direct or indirect injection, lean or stoichiometric mixing...) and the driving conditions. Ethanol in fuel may also lead to a reduction of polyaromatic hydrocarbons (PAH) [12], and increased emissions of certain aldehydes [10, 16], under certain operations conditions or ethanol blending. Again, this effect needs to be quantified for high ethanol fuels.

For these reasons, this study analyses the impact of fuel's ethanol content on regulated emissions, ultra-fine particles from 10 nm, and non-regulated emissions, over recent EU6c Flexi-fuel, and non-flexi-fuel EU6d-TEMP technologies.

2. Test installation

2.1 Fuel Matrix

The tested fuel matrix aims to cover a set of different formulation cases that may be encountered when standard commercial fuels E10 and E85 are mixed in the tank. The blends are listed in Table **1**. The ethanol content varies from 10 to 85 % v/v.

Fuel	Formulation	Standard
E10	10% v/v of ethanol (fuel that meets Euro 6 certification specifications)	EN 228
E20	20% v/v of ethanol (mixture of E10 and E85)	EN 228[1]
E50	50% v/v of ethanol (mixture of E10 and E85)	EN 228[2]
E85	85% v/v of ethanol (fuel that meets Euro 6 certification specifications)	En 15293

Table 1: Fuel matrix

2.2 Test equipment

The study focused on three vehicles with different recent engine technologies and representatives of the French market. The vehicles selected were:

- Vehicle A, a EURO 6d-temp gasoline passenger car (GDI, fitted with particle filter).
- Vehicle B, a EURO 6d-temp gasoline hybrid passenger car (GII)
- Vehicle C, a EURO 6c flexi-fuel passenger car (GDI)

These vehicles were fitted with E85 conversion kit and the four matrix's fuels were evaluated. This variation was cross tested over a second conversion kit technologies, over E10 and E85 fuels. The full fuel matrix was also evaluated on the flexi-fuel vehicle EU6c. The tests thus allowed the assessment of the impact of ethanol fuel-content over regulated and non-regulated emissions for different technological solutions and recent vehicle technologies. Table 2 shows an overview.

Vehicle	Configuration tested		
		E85 conversion kit #1	E85 conversion kit #3
Vehicle A GDI + GPF	E10	E10	E10
	E20	E20	
		E50	
		E85	E85
		E85 conversion kit #2	E85 conversion kit #4
Vehicle B Hybrid GII	E10	E10	E10
	E20	E20	
		E50	
		E85	E85
Vehicle C Flexfuel GDI	E10, E20, E50, E85		

Table 2: Configurations tested

The tests were carried out on a roller bench over WLTC and Artemis Urban, Rural, Highway driving cycles (Table **3**). The chassis dynamometer is located into a conditioned chamber maintained at 23°C±1°C. The exhaust gas emissions were collected and measured according to the Constant Volume System (CVS) based on a full flow dilution tunnel. Figure 1 shows the schema of the analytical apparatus.

Power (kW)	55
Speed (km/h)	160
Type	Bi-roller
Ventilation maximum speed	120 km/h
Temperature	23 °C ± 1 °C
Hygrometry	45 % ± 10 %

Table 3: Roller bench technical characteristics

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[1] All specifications are met except for ethanol content.

[2] All specifications are met except for ethanol content.

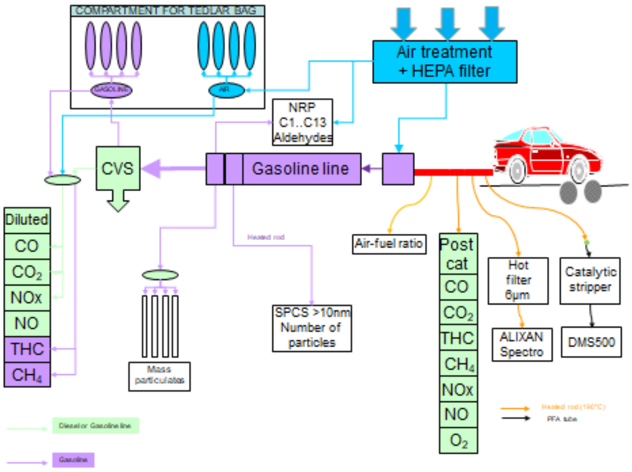


Figure 1: Exhaust gas conditioning and measuring apparatus available at roller bench.

Regulated emissions. Standard instrumentation used for characterisation of regulated pollutants from Euro 6 vehicles was used, with the exception of the extension of particle size down to 10 nm:

- Diluted gas analyser allowing the characterisation of HC, CO, CO_2, NO_x emissions,
- Gravimetric sampling system for the determination of the mass of particles emitted (PM),
- Standardised counting system for the determination of the total number of particles emitted (diameter > 10 nm) (PN).

The analytical methods employed to measure gaseous emissions, particles mass and number are described in Table **4**.

Compound	Analytical method
CO / CO_2	Non Dispersive Infra-Red (NDIR)
NO / NOx	Chemiluminescence Analysis (CLA)
HC	Flame Ionisation Detector (FID)
Particles mass	Pallflex® filter
Particles number	SPCS (Solid Particle Counting System)

Table 4: Analytical methods

Three different families of non-regulated compounds were analysed in the framework of this study: aldehydes and ketones, polycyclic aromatic hydrocarbons (PAHs) and volatile organic compounds (VOC).

Analysis of aldehydes and ketones. Exhaust gas samples were collected over cartridges containing silica grafted with DNPH (Dinitrophenylhydrazine). Aldehydes and ketones are thus transformed into hydrazone derivatives and retained on the solid phase. Cartridges are then eluted with a mixture of organic solvents (water/acetonitrile 35/65, HPLC grade) to recover the analytes. The extract are analysed with a high-performance liquid chromatograph (Prominence LCD-20ADXR from Shimadzu) coupled to a UV detector (SPD-20A from Shimadzu) operated at 365 nm.

Analysis of PAHs. PAHs sorbed to particles present in the exhaust gases are sampled over PallFlex® filters which are extracted with dichloromethane using a ThermoFisher Automated Solvent Extractor (ASE 350) at 100°C and 100 bars. Extracts were spiked with an internal standard and injected in a gas chromatograph (Agilent 7890) coupled to a mass spectrometer (Agilent 5978C). The 16 PAHs targeted by the US EPA are monitored in Selected Ion Monitoring (SIM) mode.

Analysis of VOCs (C1 to C12). Exhaust gases are sampled in Tedlar® bags and injected in a custom configuration consisting of two gas chromatographs (Varian 450) equipped with three different columns and three FID detectors to ensure the quantification of methane, C2 to C6 and C6 to C12. A pre-concentration system with adsorbents cooled with liquid nitrogen enables to greatly improve the sensitivity of the technique (around ppb).

Real time analysis of VOCs. The BTrap instrument is a Chemical Ionization Mass Spectrometer [17]. This instrument is a Fourier Transform Ion Cyclotron Resonance Mass Spectrometer (FTICR-MS) in which the magnetic field is produced by a cylindrical permanent magnet assembly. It features a high mass resolution enabling the identification of the organic molecule by their molecular formula. The ionization method used was the proton transfer reaction from H_3O^e precursor ions which ionizes only the Volatile Organic Compounds present in the gas exhaust, and not the main component of air. A mass spectrum is obtained every few seconds, giving a measurement in real time of the vehicle engine emission. The sample line of the BTrap mass spectrometer is connected directly to the exhaust, without any dilution, through a heated line.

All of the gas analysers were calibrated with certified reference standards. Blank levels are determined by regular analyses of the dilution air used in the roller test bed. The overall amounts of non-regulated compounds determined by analysis (expressed in ppm) are then converted into mg/km thanks to several experimental factors from the roller test bed (volume sampled, distance, dilution factors…).

2. Vehicles test protocol

Regulated and non-regulated pollutants, as well as fuel consumption were obtained over four different driving cycles, WLTC and Artemis Urban, Rural and

Highway **Erreur ! Source du renvoi introuvable.**. The protocol to perform the tests was:

- Introduction of the vehicle in the roller bench according to the standard conditions
- Cold soaking vehicle with temperature between 20°C and 25°C
- Driving test according to WLTC cycle or Artemis cycles

The tests were all repeated twice (two chassis dynamometer runs per vehicle and per operating condition). A repeatability criteria was defined using CO_2 emissions as the main parameter. Calculation is based on CO2 measurement over two tests according to the following formula:

$$Repeatability = \frac{2 \times \sigma_{CO2}}{Average \times \sqrt{Nb\_tests}}$$

Where σ_{CO2} is the standard deviation of CO2 global measurements and a validation limit of 2 % maximum deviation was imposed.

2.1 Test cycles

All three vehicles were tested with the WLTC and Artemis driving cycles. André et al.(2004)**Erreur ! Source du renvoi introuvable.** [18] gives a detailed description of the Artemis driving cycles. In brief, the Artemis cycle contains urban (Figure 2), rural (Figure 3) and highway (Figure 4) conditions with average speeds of around 17.6, 57.5 and 96.9 km h–1; sampling times of around 17, 18 and 18 minutes; and driving distances of 4.9, 17.3 and 28.7 km, respectively. WLTC is European and world approved driving cycle with cold start (Figure 5). The average speed, sampling time and driving distance is 47 km h–1, 30 minutes and 23 km, respectively.

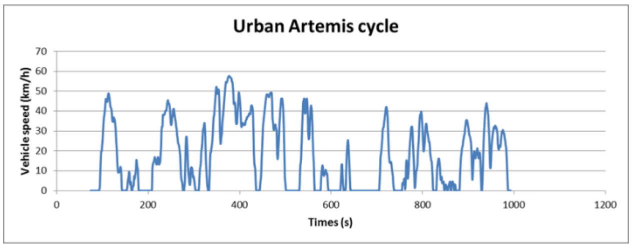


Figure 2: Profile of Urban Artemis cycle

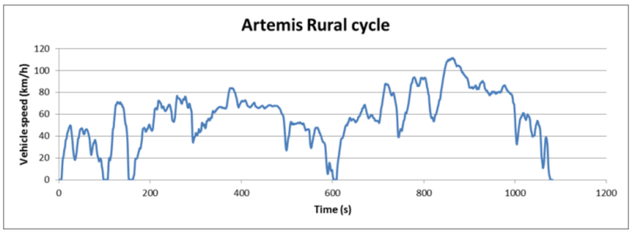


Figure 3: Profile of Rural Artemis cycle

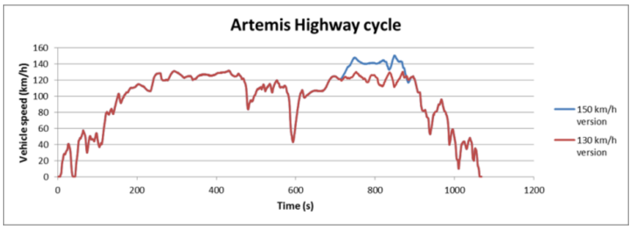


Figure 4: Profile of Highway Artemis cycle

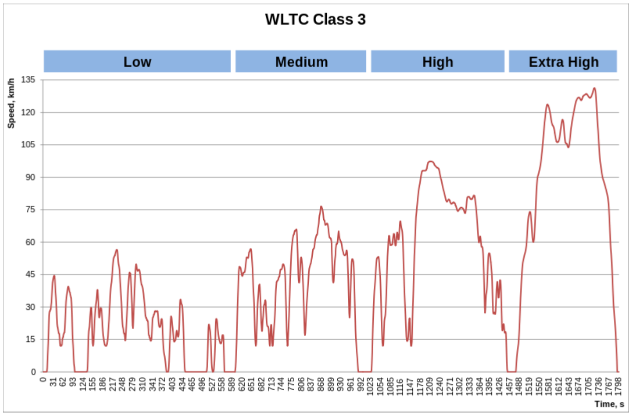


Figure 5: Profile of WLTC cycle (version 7)

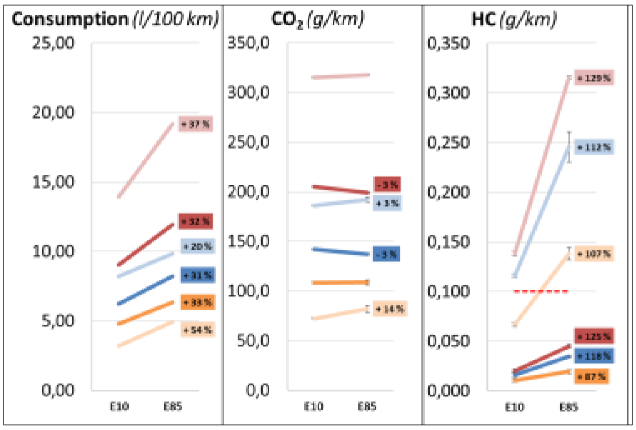
Considering Artemis cycle as the succession of the 3 cycles (Urban, Rural and Highway), this gives a distribution in kilometres of 9.4 %, 33.4 % and 57.2 %. This distribution is very different from that of WLTC cycle. Indeed, combining the slow and medium WLTC phases results in a three phase distribution of 34 %, 31 % and 35 % kilometres respectively. As a result, the cycle referred to in the rest of this paper as "WLTC-weighted CADC" is a weighting of the Urban, Rural and Highway Artemis cycles according to the proportions of WLTC cycle (34 %, 31 % and 35 %) in order not to bias the comparisons when the results are presented for mixed driving.

3. Results

A comparison of the results of the WLTC cycle for E10 and E85 fuels is shown in Figure **6** for the three vehicles. The results for the Low phase are also highlighted. Indeed, this phase represents urban driving and is the phase that emits the most pollutants. The results of the Artemis cycles are summarised in Figure **7** for the E10 and E85 fuels.

The results of the Urban cycle, the most emissive, are also shown.

The fuel consumption increases by 28 to 33 % on WLTC and WLTC-weighted CADC cycles, with similar tendencies found for all vehicles. Greater dispersion is present in warm-up Low phase and Urban cycle. Indeed, the over-consumption can vary from 20 to 54 % in the Low cycle, and from 26 to 33 % in the Urban cycle. Regarding CO_2 emissions, a reduction of up to 3 % on average is obtained in the WLTC cycle at 85 % v/v ethanol. Over Artemis cycles, the reduction in CO_2 emissions is more pronounced, averaging 6 %, and the trend is already present in the Urban cycle. The figure highlights the difference in trend between full cycle CO_2 emissions and Low phase: the Low phase shows an increase on A and B vehicles, between 3 and 14 %, respectively. The original flexi-fuel calibration seems to be favourable to CO_2 emissions under these driving conditions. Results on the Artemis cycle show less variability, and indicate that CO2 emissions show a stronger variations and driving cycle dependency over vehicles equipped with conversion Kit E85. Moreover, only the flexi-fuel vehicle shows a reduction in CO_2 emissions at intermediate ethanol levels. For the A and B vehicles, in E20 and E50, a clear trend in the impact of ethanol content on CO_2 emissions is not found, and the results are within the repeatability range.



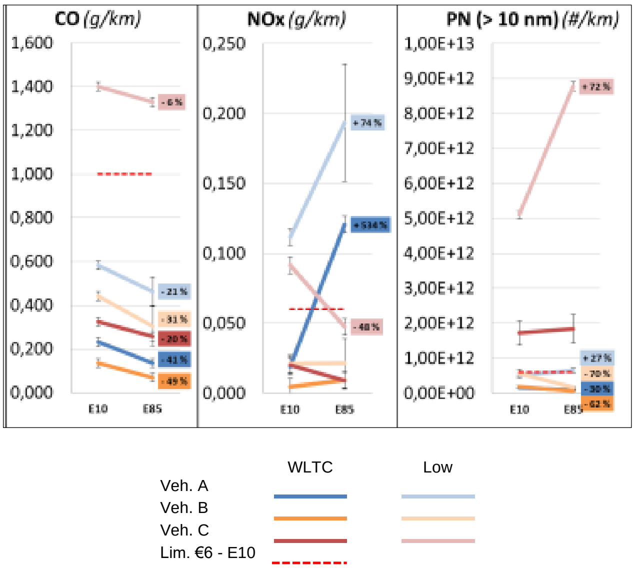
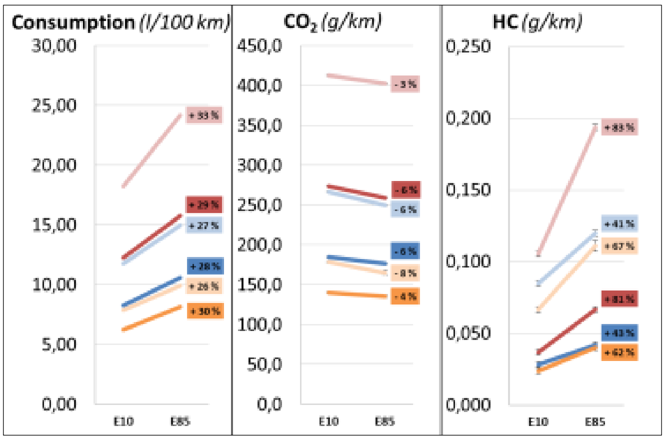


Figure 6: Results of WLTC cycle and Low phase for E10 and E85 fuels

Concerning HC emissions, a clear trend towards the increase of overall emissions is observed, this independent of the vehicle technology, or driving cycle.The increase varies within 40 % to more than doubled, most important increase is found over the Urban and Low cycles, when catalyst has not attained maximum efficiency. However cycle results remain below the regulation limit of 0.1 g/km, and this for both cycles. CO emissions also show a clear reduction tendency, for all vehicle technology and driving cycle.



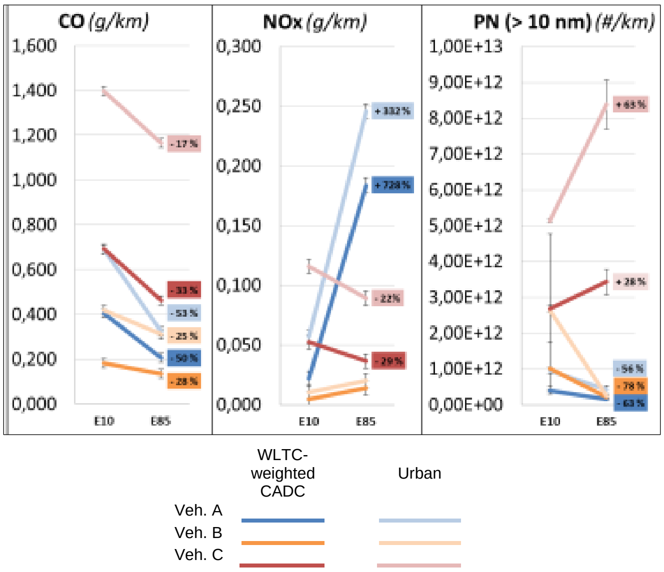


Figure 7: Results of all Artemis cycles and Urban cycle for E10 and E85 fuels

NO_x emissions are variable and dependent on the vehicle technology. Flexi-fuel vehicle C, indicates a reduction of NO_x emissions, ranging between 20 and 50 %, while vehicle B, having an indirect injection, shows little dependency on ethanol fuel content. Contrastingly, vehicle A shows a strong increase in NO_x emissions for both cycles and different phases. The increase has been observed in the warm-up phase, as well as during strong accelerations throughout the driving cycles, indicating a deteriorated of the lambda control with E85 conversion kit and high ethanol fuel content. An escalation is observed throughout the increase of ethanol concentration, and the EU6 regulation limit of 0.060 g/km is exceeded at E85.

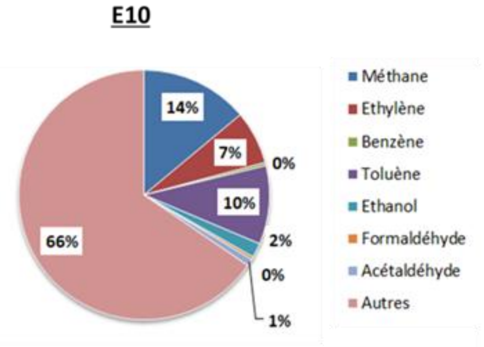
The number of particle emissions show a reduction with increased ethanol content, with the exception of the flexi-fuel vehicle. The increase concerns the warm up phase, over the urban and Low cycles, while no increase is observed during the acceleration phases after warm up. The contrast between vehicle A and vehicle C could indicate the effective control of particle emissions through the integration of the particle filter.

The analysis of non-regulated emissions indicate that the most notable volatile organic compounds VOC pollutants are methane, ethylene, benzene, and toluene. Unburned ethanol has also been detected. Formaldehyde and acetaldehyde are the most significant aldehydes detected. These pollutants have been observed as the most significant mass emissions throughout the fuel matrix tested. Absolute mass emissions are variable and depending on the vehicle technology. However, the impact of ethanol fuel content is consistent throughout the test sequence.

The increase of fuel ethanol content has a significant impact on the compositional distribution of non-regulated emissions. Figure 8 illustrates the non-regulated emissions observed over vehicle A, representative of the mean tendency over the different vehicles and conversion kit technologies. For E10 fuels, a variable composition of VOC embody between 60 to 70 % of total emissions, while dominant compounds are methane (9 to 14 %), toluene (10 to 12 %) and ethylene (3 to 7 %). Acetaldehyde and ethanol represent 1 to 2 % of the total composition. Increasing the ethanol content has a strong reduction impact of the total VOC, to 15-30 % at E85. Concerning specific VOC compounds, methane and ethylene increase with ethanol content. Methane increases to 20-25 % of total emissions, representing an absolute increase of a factor of 2 to 4. However, analysis of greenhouse warming of CO_2 and methane emissions remain below that of reference fuel, as they are strongly dependant on CO_2 emissions.

Results indicate little impact over benzene emissions, while toluene is strongly reduced. Unburned ethanol represent 30 to 40 % of the non-regulated emissions. Acetaldehyde emissions also increase to 5 to 10 %.

The impact of ethanol over aldehyde emissions is most significant for the EU6d flexi-fuel vehicle. Formaldehyde emissions have a tendency to increase for ethanol content of 50 and 85 % vol. EU6d-TEMP vehicles, A and B, show a maximal formaldehyde emission for E50, and above 1 mg/km, while decreasing again at E85. However, flexi-fuel EU6d vehicle C, has a linear increasing tendency, with a maximum value of 4.5 mg/km at E85. Acetaldehyde emissions increase continuously with the ethanol content, with an exception found for vehicle A, for which mean results indicate a maximum emission at E50, and attaining reference values at E85. Overall, aldehyde results indicate an alleviated E85 impact over the total aldehyde emissions for recent vehicle technologies EU6d-TEMP.



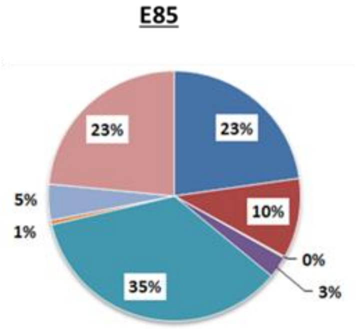


Figure 8: Non-regulated emissions distribution detected for E10 and E85 fuels, vehicle A, Low phase of the WLTC cycle.

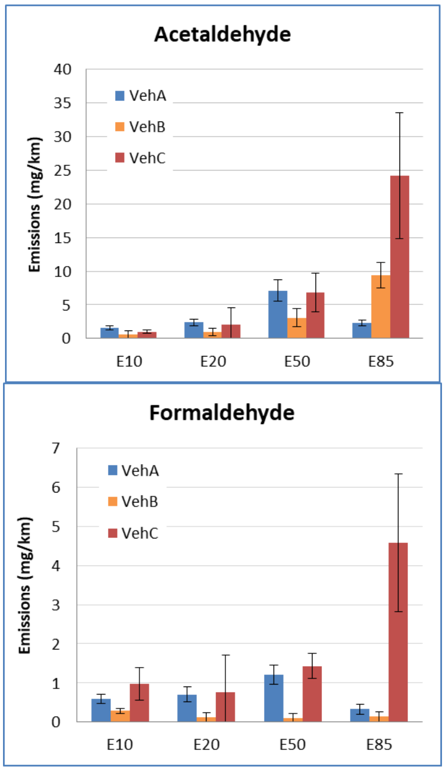


Figure 9: Mean formaldehyde and acetaldehyde emissions, Low phase of the WLTC cycle.

Real time measurements. The main VOCs emissions as measured in real time directly at the exhaust for vehicle A for E50 by the BTrap instrument are shown in Figure 9. The principal observed emission is for ethanol. The compounds are emitted at the beginning (first three minutes) of the low phase of the WLTC cycle, during the warm up of the after treatment system. The temporal structure of the emission differ a little for the different compounds. The beginning of the ethanol detection is somewhat delayed compared to aromatic compounds or acetaldehyde.

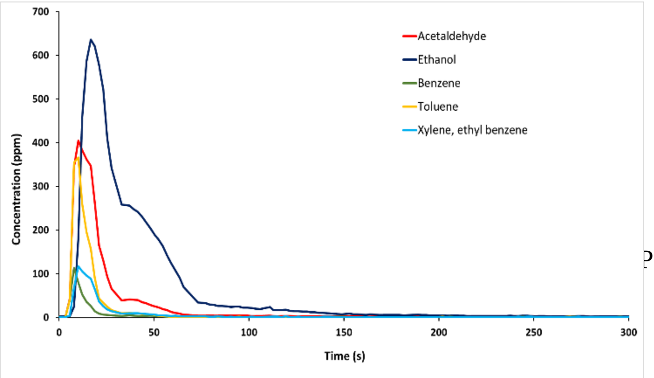


Figure 9: Instant concentrations measured for the main emissions as a function of time (s) for vehicle A with conversion kit #2.

The integration of the area under the curve gives for each compound the total quantity emitted in ppm.s during the cycle. The evolution of the emissions measured for each compound as a function of the fuel matrix and the conversion system are in good agreement with those measured in the laboratory.

4. Conclusion

In this study, the effect of ethanol content on the performance and emissions of EU6b and EU6d-TEMP spark-iginition vehicles were investigated experimentally in order to assess its impact over regulated and non-regulated emissions.

The results show that the impact of high ethanol content is broadly similar amongst the different technologies tested, E85 conversion kits and flexi-fuel. it was concluded that from 50 vol. % ethanol in the fuel, the emissions of total unburnt hydrocarbons increase with an exponential trend. To a lesser extent, the increase in ethanol content results in a reduction in carbon monoxide and particulate matter for EU6d-temp vehicles. However, the results indicate a risk of increased NOx emissions over EU6d-TEMP technologies that would require confirmation over other similar technology vehicles. A positive impact of high fuel ethanol content over CO_2 emissions is observed over E85. This is slightly reduced for vehicles with conversion kit, possibly due to increased CO_2 emissions in the WLTC's Low phase.

The main trends identified for non-regulated emissions were observed in a similar way for all configurations. Ethanol content of 50 % vol and above, tend to result in an increase of aldehyde emissions, ethylene, methane and ethanol in the exhaust gasses. The later represent near 50 % of total non-regulated emissions.

It would be of interest to extend these analysis over a larger vehicle fleet and Real Driving Emissions cycles. Further study toward the optimisation of regulated and non-regulated emissions for high ethanol fuels could be beneficial in an aperture towards the increased of renewable fuels.

5. Acknowledgement

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8. Glossary

VOC: Volatile Organic Compound