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Measurement of Sub-23 nm particles emitted by gasoline direct injection engine with new advanced instrumentation

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ABSTRACT

The research on health effects of soot particles has demonstrated their toxic impact on humans, especially for the smallest ones that can pass through the lungs into the bloodstream and be transferred to other parts of the body. Since the Euro 5b regulation, the total particle number (PN) at the exhaust is limited, but the associated protocol developed by the Particle Measurement Program (PMP) group defined a counting efficiency at the 23 nm cut-off particle diameter to avoid measurement artefacts [1][2]. Recent studies have demonstrated that the last generation Euro 6 engines can emit as many particles in the range 10 – 23 nm as beyond 23 nm [3].

The SUREAL-23 project (Understanding, Measuring and Regulating Sub-23 nm Particle Emissions from Direct Injection Engines Including Real Driving Conditions), funded by Horizon 2020 EU-program, aims to develop sampling, conditioning and measuring instruments and associated methodologies to extend the existing protocol down to at least 10 nm. This measurement setup was evaluated on various light duty direct injection platforms. This communication focuses on a gasoline-DI vehicle with a Euro 6b engine. Tests were conducted on multiple operating conditions (moderate and aggressive driving cycles, hot and cold starts, and several fuel and lubricant formulations). Sampling and conditioning were done with a two-stage dilution system, with a built-in catalytic stripper. The prototype instruments have been compared to commercial reference soot particle analyzers (TSI CPC, Horiba MEXA-2000 SPCS and Cambustion DMS500). A good consistency between all the measurements was demonstrated, with a satisfactory repeatability and robustness of the proposed measurement setup and of the associated methodology.

An on-board version of the proposed setup is currently being developed to allow PN measurement in Real Driving Emissions (RDE) conditions.

INTRODUCTION

A large fraction of the total number of particles emitted by direct injection engines are below the adopted 23 nm diameter threshold [3] and although the EU aims to regulate these emissions and impose limits for new light-duty vehicles, this is not yet possible due to the absence of accurate and reliable quantification methods, especially under real driving conditions. The main reason for this is the lack of adequate knowledge regarding the nature of sub-23 nm particles from different engine/fuel combinations under different engine operating. The EU-funded project SUREAL-23 aims to overcome such barriers by introducing novel technologies for the measurement of sub-23 nm exhaust particle concentration, size and composition.

The objectives of the present work are to evaluate the novel sampling and dilution system, and the advanced particle measurement technologies that were developed in the framework of the project. The

evaluations were conducted at the IFP Energies Nouvelles (IFPEN) lab chassis dyno with a modern Euro 6b turbocharged gasoline direct injection vehicle. In order to investigate a wide variety of aerosol concentrations, particle compositions and engine conditions, the tests were conducted on various driving cycles, with cold or hot start, with different fuel compositions and different lubricants. The response of the prototype instruments has been evaluated under these conditions, and also compared to commercial instruments.

EXPERIMENTAL SETUP

VEHICLE

The vehicle selected for the tests is a Euro 6b, 4 cylinders gasoline engine with turbocharger. It is equipped with a dual injection system that combines direct injection (GDI, wall-guided) with indirect injection (PFI) into the intake manifold. In part-load operation, the indirect injection supplements direct gasoline injection to improve fuel economy and to reduce the output of particulates from the engine, while only direct injection is used at engine start-up and during the warm-up phase to have a more stable combustion. With this system, this vehicle is expected to be compliant with the Euro 6c PN limitation fixed at 6.10^{+11} part/km.

The vehicle has been tested with its base emission control system, i.e. a simple 3-way catalyst.

COMMERCIAL PN SAMPLING AND MEASUREMENT INSTRUMENTS

Commercial instruments were used to establish references from very well-known devices. This setup included the following devices :

- A DMS500 (Differential Mobility Spectrometer) from Cambustion: The DMS500 gives access to the particle size distribution (PSD) and number in real time. The range of electric mobility diameters measured by the DMS500 lies from 5 to 1000 nm. The DMS500 uses its own dilution system, that didn't include any volatile particle remover (VPR).
- A PMP-compliant Horiba MEXA-2000 SPCS. This system includes a volatile particle remover (VPR) to eliminate the volatile organic fraction (VOF) of the particles, and an optical condensation particle counter (CPC) with a cut-off diameter of 50% at 23 nm, as required by the European regulation.
- A second CPC, a TSI CPC3776, was installed downstream of the Horiba VPR, in parallel with the PMP CPC 23 nm cut-off. The CPC3776 has a much lower cut-off diameter of 2.5 nm and then is able to measure the sub-23 nm particles. However, the PMP system is not optimized regarding the small particles losses by diffusion process, so the results can be subjected to this artefact.

PROTOTYPE PN SAMPLING AND MEASUREMENT INSTRUMENTS

Besides these commercial instruments, the prototypes developed in the SUREAL-23 project were gathered to perform their evaluation. The entailed instruments are:

1) The sampling and dilution system from APTL/CERTH:

A prototype Sampling and Conditioning Particle System (SCPS) was designed and constructed by the APTL/CERTH lab. Aerosol flow first passes through a hot porous tube diluter and then through a catalytic stripper (CS), following the same principle that presented in [4]. A downstream ejector diluter creates suction which drives the sampling flow and provides a further dilution (see Figure 1).

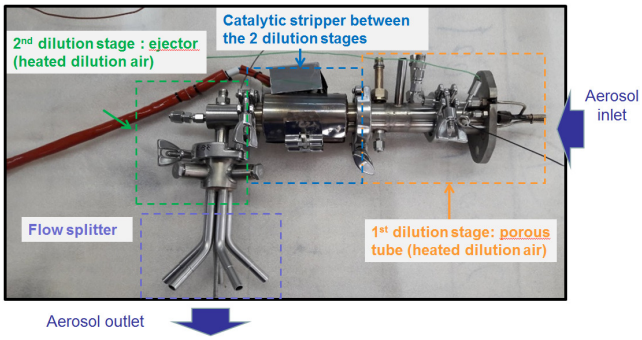

Figure 1: SCPS dilutor by APTL/CERTH

The whole system is fully integrated and flexible in terms of dilution ratio (DR) and temperature. DR is continuously calculated with a differential pressure measurement across an orifice. Moreover, DR is adjustable and varies in the range 30-120. This is particularly useful for accurately measuring at transient exhaust conditions. Furthermore, the system has been designed so that pressure fluctuation in the exhaust tube does not significantly affect the sampling flow and consequently the dilution ratio. To efficiently remove volatile and semi-volatile compounds without creating artefact particles, the system integrates an in-house developed catalytic stripper that oxidizes volatile compounds. DR calculation has shown very good agreement with the CO_2 calculated DR (up to 6% deviation) at steady state engine points. The SCPS was also tested for its tetracontane particle removal efficiency which was higher than 99% for all operating conditions fulfilling the legislation demands. Moreover, solid particle penetration tests showed that the cut-off size d_{50} was at 7.5 nm and goes beyond the current State-of-the-Art. More details of the SCPS diluter are given in the reference [5].

Additionally to this sampling and dilution system, 2 prototypes dedicated to PN measurement were developed in the framework of the SUREAL-23 project. These are the Half-Mini DMA developed by Sociedad Europea de Análisis Diferencial de Movilidad (SEADM) and the Induced Current Aerosol Detector (ICAD) from Fachhochschule Nordwestschweiz (FHNW). These instruments have several common features:

- They are compliant with PN measurement under the current 23 nm diameter cut-off (50%): down to 10 nm for the ICAD and down to 5 nm for the HM-DMA.
- In contrast with the commercial instruments, the sensitive elements of these prototypes are capable of hot measurements, up to 150 °C. This high temperature operation allows reducing the dilution ratio. This has two advantages: the first one is the abatement of undesired artefacts due to the dilution. The second is the simplification of the dilution system, which is particularly useful for embedded measurements.
- They are both build to be light, compact and vibration resistant in order to use them as Portable Emissions Measurement Systems (PEMS).

2) The Half-Mini DMA from SEADM :

The Half-Mini DMA (Differential Mobility Analyzer) is a mobility filtering system that operates by combining an electric field and a perpendicular flow field of gas (called sheath flow) to separate charged particles by mobility which is directly related to the particle size. The separation cell is cylindrical, the flow field is parallel to the axis and the electric field follows radial direction. Particles are introduced from the outer diameter, and only size-selected particles are collected at a slit on the inner electrode [6] [7].

The aerosol sample is charged with a Secondary ElectroSpray Ionization (SESI) system before entering the Half-Mini DMA. The SESI ionizer generates a cloud of ions by means of an electrospray which are mixed with the sample and transfer their charges to the aerosol particles.

These charged particles are measured by means of an electrometer in the range 1 – 30 nm. The Particle Size Distribution (PSD) of the aerosol is measured in about 2 seconds for this this range. The Half-Mini DMA works at high temperature (up to 200 °C) in order to prevent condensation and artefacts (particles created by the dilution system) and this allows its operation with reduced sample dilution needs (in the current work a heated, single-stage ejector-type diluter was used). This is achieved by heating the sheath flow and the SESI chamber, and isolating the separation cell. The sheath flow gas is continuously taken from the room downstream of HEPA filter and released after being used at the Half-Mini DMA by means of a blower installed downstream the separation cell. There is some vacuum at the sample inlet, allowing sampling without additional pumping, directly from the tailpipe. Since the blower cannot work properly at high temperatures, a cooling element is placed before the blower inlet (Figure 2). The Half-mini DMA exhibits an unprecedented resolution at low size and an extremely fast response.

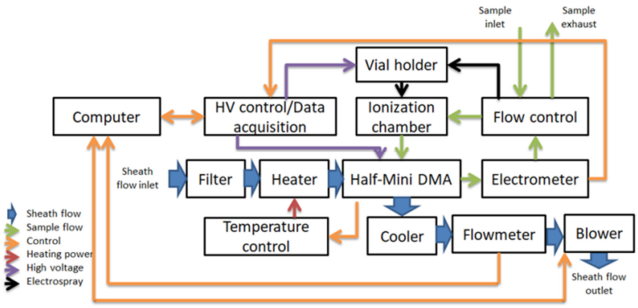

Figure 2: Operating diagram of the HM-DMA

3) The ICAD from FHNW:

The ICAD is a diffusion charging instrument, based on the partector instrument [8]. A scheme is shown in Figure 3.

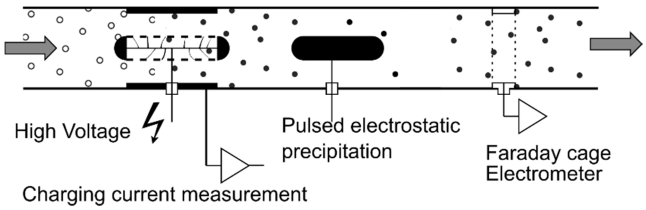

Figure 3: Scheme of the automotive partector

The device consists of a unipolar diffusion charger that is followed by a pulsed electrostatic precipitator to remove a fraction of the particles and finally an induction stage to measure the removal of the particles in the precipitator. The induction cage produces very small signals, on the order of fA – pA, which must be measured by a very sensitive electrometer. Using an induction stage rather than measuring the charge captured in a filter avoids issues with electrometer zero drift and filter clogging [8] as an AC current is measured and no particles are precipitated in the measuring stage. The device has been tuned to achieve a 50% cutoff around 10 nm and can be heated up to 150 °C. This allows an operation with only a very small or even no dilution. Figure 4 shows the counting efficiency as function of particle size.

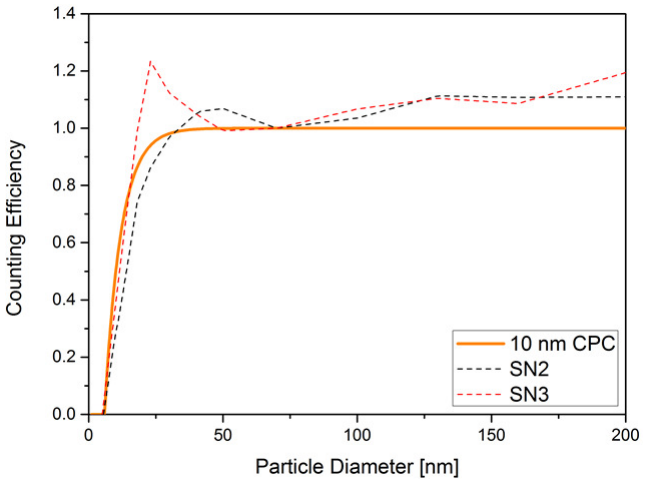

Figure 4: Normalized counting efficiency of the 2 SUREAL-23 prototypes compared to a hypothetical 10 nm CPC

The Figure 5 summarizes the global installation of the sampling and measurement instruments on a scheme.

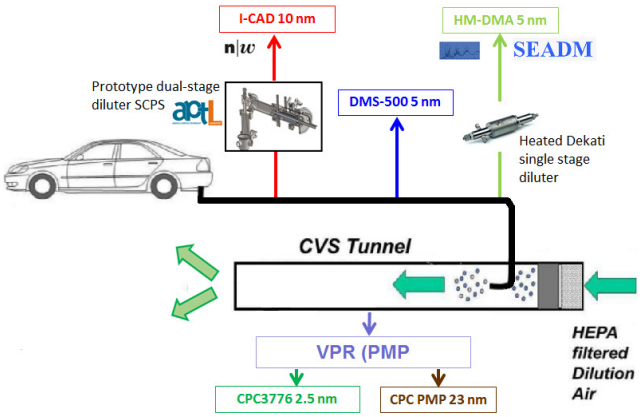

Figure 5: Scheme of the chassis dyno experimental setup

Regarding the temperature conditions, the SCPS diluter used for the ICAD and the Dekati ejector diluter used for the HM-DMA were both heated, so that the gas inlet temperature of the ICAD and of the HM-DMA was about 150 °C.

FUELS

Four different fuels were used in this study:

- An E10 European standard fuel, containing 10 % of bioethanol.
- A specially made fuel with high content of sulfur (150 ppm S), E10 based. This fuel will be noted "High S" in the next.
- A specially made fuel with high content of aromatic compounds, raising the total aromatic content to 39% (E10 based). This fuel will be noted "High Aro" in the next.
- An E25 fuel, obtained by splash blending of pure ethanol in E10 fuel, to reach a 25% of ethanol content.

LUBRICANTS

Two different lubricants were examined in this study:

- The lubricant recommended by the car manufacturer, with low level of Sulfated Ashes Phosphorus and Sulfates (SAPS) of about 0.6%. This lubricant will be called "Standard Oil" in the next.
- An alternative lubricant, Total Quartz Futur 9000 5W30, with full SAPS content (~1.1 %). This last lubricant will be called "full SAPS" in the next

DRIVING CYCLES

Two driving cycle conditions were evaluated: the standard regulated WLTC (Worldwide Harmonized Light Vehicles Test Cycle) cycle, and a more aggressive cycle known as RTS95, meaning that only 5% of the drivers have more aggressive driving than this cycle. Many car manufacturers in Europe are using this RTS95 cycle as a "worse case" of RDE (Real Driving Emission) conditions because of its high level of acceleration up to 2.88 m/s^2 [9]. This kind of aggressive cycle can significantly influence the PN measurement [10].
Both "cold" start and hot start were evaluated. In this paper, "cold" start must be interpreted as an engine at ambient temperature (around 20 °C): the vehicle is start after an overnight cooling-down.

RESULTS

EFFECT OF THE FUELS

The effect of the fuels was evaluated at the chassis dyno on the Euro 6b vehicle outlined in the previous part. During this phase, 4 different fuels were evaluated regarding PN emissions, with the set of instruments described above. The tests were done on the two driving cycles, WLTC and RTS95, both in cold and hot start conditions. The Figure 6 exhibits the results for the case of WLTC with cold start, representative of the European homologation test.

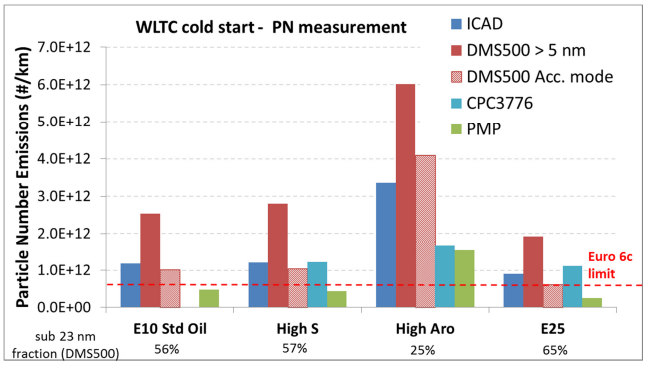

Figure 6: PN measurement on WLTC cycle with cold start with various instruments – 4 fuels tested

The first observation is that the vehicle is compliant with the Euro 6c European regulation when measuring PN emission with the PMP instrument and with the standard E10 fuel. The PN number in this case is 4.7 10^{+11} part/km, below the Euro 6c limit fixed at 6.10^{+11} part/km. The PN number also remains under this limit for the high sulfured fuel and for the E25, but exceeds this limit for the fuel with the high aromatic content.

Two metrics were considered from the DMS500 instrument: the first one is the raw concentration, with a 5 nm diameter cut-off. This value includes all the volatile fractions because no Volatile Particle Remover (VPR) is used upstream of the DMS500. The second one represents the accumulation mode, a numerically calculated value obtained by fitting a log-normal distribution on the right side of the Particle Size Distribution (PSD) curve. Thus, it excludes the nucleation mode from the particle size distribution and can be seen as a "software" volatile particle remover.

In spite of this software correction, the particle concentration of accumulation mode measured by the DMS500 remains from 2.1 times to 2.7 times higher than those measured with the PMP protocol. It tends to demonstrate that the "software" VPR is not so efficient that an actual VPR. It is probable that some volatile particles above 23 nm are taking into account by the DMS500 measurement.

The DMS500 (down to 5 nm) logically displays the highest PN emissions, because it takes into account

the smallest particles without removing the volatile fraction. Then, this metrics is not directly comparable to the others. But it is used to calculate the PN fractions in the range 5 - 23 based on the PSD. These fractions are indicated in the lower part of the Figure 6. The 5 – 23 nm PN fraction lies from 25% with the high aromatic fuel, that created a large part of solid particles, up to 65% for the E25 fuel that tends to create smaller particles, even if this fuel globally produces less PN than the others. These results are consistent with that was found in [1], i.e. that the less the total PN number is, the higher the sub-23 nm PN is.

The prototype ICAD is designed to have a cut-off limit around 10 nm. It is noticeable that the PN emissions measured with the ICAD are over the current Euro 6c threshold, even if this device is installed downstream of the catalytic stripper of the SCPS diluter that removes the volatile fraction of the particles and if the sampling temperature is as high as 150 °C to avoid condensation. For example, the PN value reaches 1.2 10^{+12} part/km in the case of the E10 fuel that is twice the current regulation. It means that if the regulated cut-off limit is reduced to 10 nm, a vehicle that comply with the current PN regulation will probably not meet the new one if the maximum limit allowed it The ICAD downstream the catalytic stripper exhibits particle concentration closed to the DMS500 accumulation mode calculated with the software VPR. It can be explained by the same physical measurement principle (electric charges detection) focused on the solid fraction of the aerosol.

Finally, the CPC3776 measured the PN down to 2.5 nm, downstream of a volatile particle remover. The VPR was the same used by the PMP system because the CPC3776 was installed in parallel with the 23-nm PMP CPC. The CPC3776 counter generally gives higher values than the PMP, by a factor comprised between 3 and 4, except for the fuel with the high aromatic content where the factor is near to 1. It confirms the low relative fraction of sub-23 nm particles for this last fuel. In return, the highest factor is obtained for the E25: it confirms this fuel produces a relatively large fraction of small solid particles in the range 2.5 – 23 nm.

These measurements demonstrated with various instrumentations that the fraction of particles that are not yet took into account by the European regulation can reach 60 – 70 % depending of the fuel, as shown in the Figure 7.

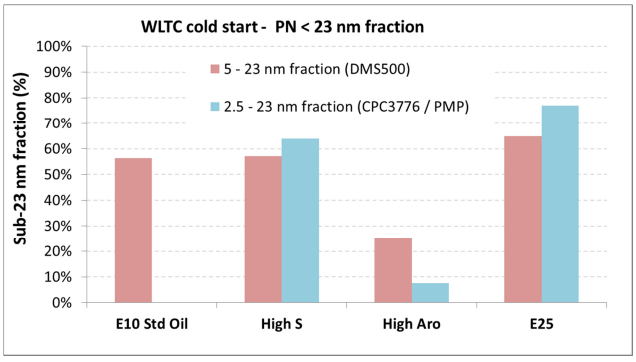

Figure 7: Fraction of PN < 23 nm measured with the DMS500 (range 5 - 23 nm) and the CPC3776+PMP system (range 2.5 - 23 nm) on WLTC cycle (cold start)

Additionally to the total PN measurement, the particle size distribution was measured with one reference instrument (the DMS500 already introduced) and the Half-Mini DMA, a prototype developed within SUREAL-23 project by SEADM. The comparison of these 2 signals is proposed in the Figure 8 for the WLTC cycle in cold conditions.

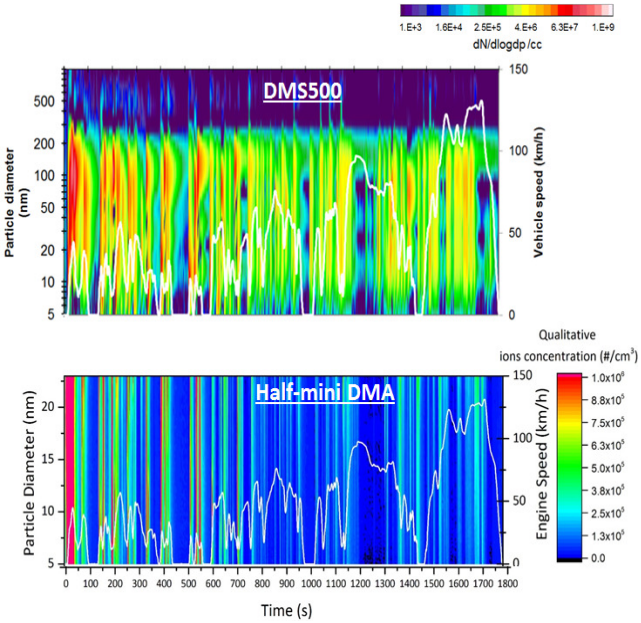

Figure 8: Comparison of the Particle Size Distribution (PSD) vs. time from the DMS500 and the Half-Mini DMA – WLTC cold conditions – Fuel with high aromatic content.

The Half-Mini DMA is able to measure charged particle number concentration in the range 5 – 25 nm with a similar resolution than the DMS500 that uses 22 electrodes to draw the PSD. The graphs from the 2 instruments are consistent, and underline the presence of sub-23 nm particles especially during the cold start and warm-up phase, and also during the accelerations during the first 600 seconds of the WLTC cycle.

EFFECT OF THE DRIVING CYCLE

Additionally to the WLTC, the PN measurements were also done on the RTS95 cycle. This paragraph focuses on this cycle, still in cold conditions, and compares the results with those from the WLTC cycle.

The Figure 9 draws the PN measurement for the 4 fuels during the tests on RTS95 cycle with cold start.

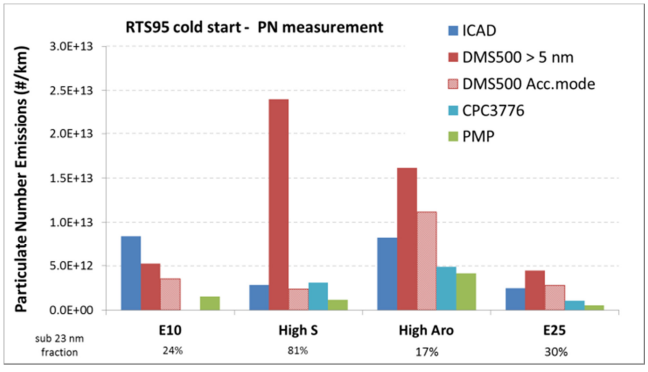

Figure 9: PN measurement on RTS95 cycle with cold start with various instruments – 4 fuels tested

It is noticeable that the global PN emission levels are far higher on this more aggressive cycle than on WLTC. For example, the PMP measurement done on the test with the E10 reference fuel gives a PN value of 1.5 10^{+12} part/km, that is significantly above the Euro 6c limit, and more than 3 times over the score obtained on the WLTC in the same cold conditions.

Like for the previous case, the PN fraction under 23 nm was calculated from the DMS500 measurement. The results are significantly different for the RTS95. The PN fraction now lies from 17% with the high aromatic fuel to 81% for the high sulphur content fuel. The PN fraction significantly goes down for the E10 and E25 of about 30 percentage points, while it substantially increases of 25 percentage points for the high sulfured fuel. It means that the fuel formulation has a direct impact on the sub-23 nm fraction of the aerosol, but the driving cycle brings also its impact on this ratio. The Figure 10 summarizes the sub-23 nm fraction calculated with the DMS500 and the CPC3776/PMP measurements. It underlines the role of the sulphur in the fuel "high S" under aggressive cycle that emits more sulfates particles below 23 nm.

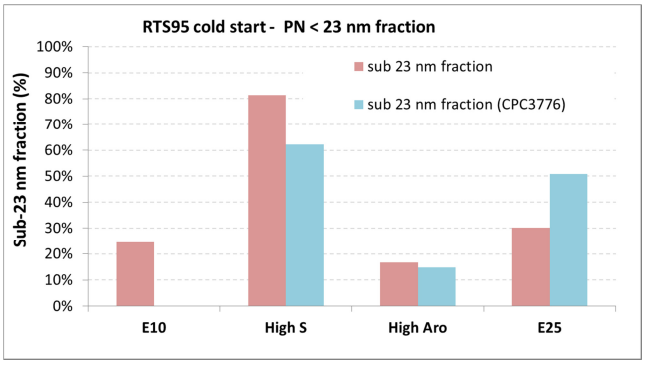

Figure 10: Fraction of PN < 23 nm measured with the DMS500 (range 5 - 23 nm) and the CPC3776+PMP system (range 2.5 - 23 nm) on RTS95 cycle (cold start)

A focus on the PSD for the high aromatic fuel shows a strong PN emission peak just after the engine start and for about 1 minute. This peak is not only visible with the DMS500, but also with the HM-DMA that focuses on the 5 – 25 nm range (see Figure 11). It underlines the presence of these sub-23 nm particles during these stages, even if the fraction is only 17% of the total with this fuel.

EFFECT OF THE LUBRICANTS

As indicated in the previous chapter, two lubricants with 2 different SAPS content were tested; the PN measurement was done at the tailpipe of the vehicle. It is noticeable that not only the SAPS content was changed, but also the global formulation of the oil. This comparison was done with the same reference fuel, i.e. E10 fuel. The Figure 12 displays the results of this comparison.

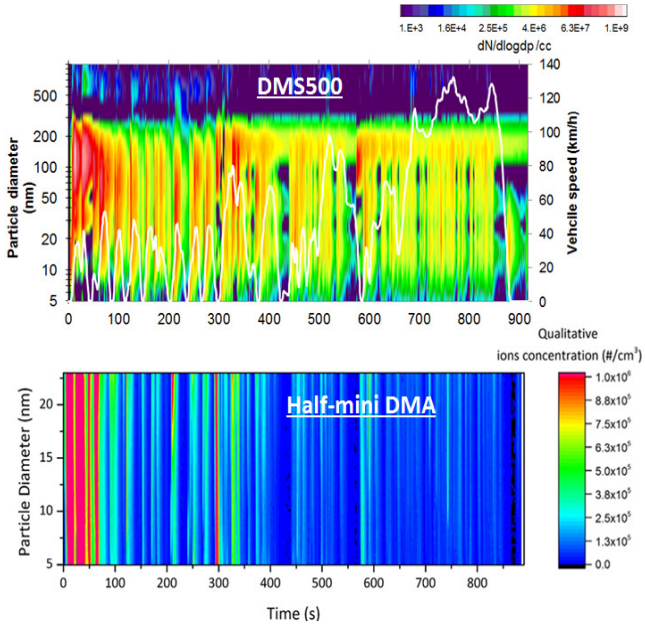

Figure 11: Comparison of the PSD vs time from the DMS500 and the Half-Mini DMA – RTS95 cold conditions – Fuel with high aromatic content.

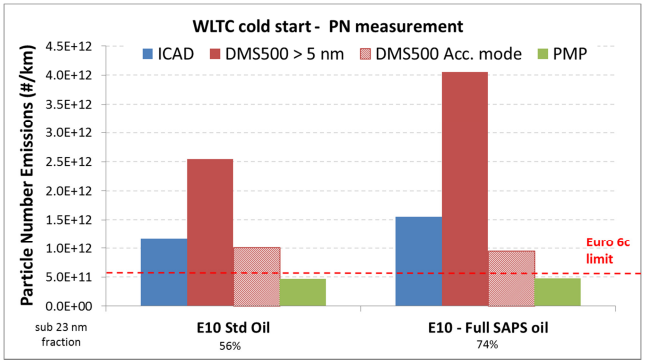

Figure 12: PN measurement on WLTC cycle with cold start with various instruments – 2 lubricants tested

The comparison of PN emissions over 23 nm shows very similar results between the 2 lubricants. So, the lubricant does not appear to play any significant role on these solid particles. In return, the ICAD and DMS 500 >5 nm related higher emissions of sub-23 nm particles: +32% for the ICAD, that has an upstream catalytic stripper, and even +60% for the DMS500>5 nm without any VPR. From these measurements, we can then conclude that the lubricant could be a noticeable contributor to the PN emissions in the range 5 – 23 nm, whether volatile or solid particles.

EFFECT OF THE START CONDITIONS: COLD VS. HOT

The cold start and the warm-up phase are known to be strong contributors to the global particle emissions on driving cycles. In this part, a comparison between total PN emissions with cold start and warm start is done on the WLTC cycle.

All these tests were managed with the reference E10 fuel, and for the 2 lubricants previously mentioned. Regarding the tests conditions, "cold start" means that the engine is started at ambient temperature after overnight stabilization, while "hot start" means that the

engine is started after a conditioning driving phase at high vehicle speeds.

The Figure 13 displays the results for the tests done on the WLTC cycle.

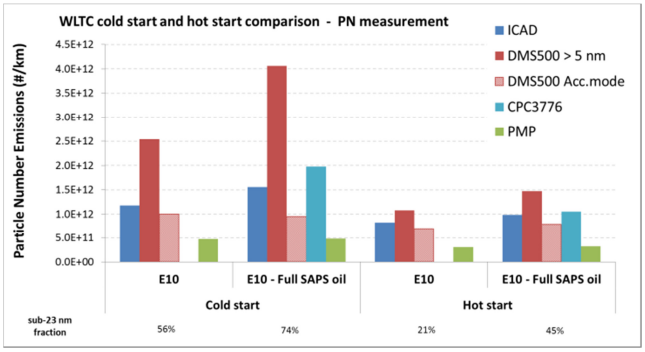

Figure 13: Comparison of PN emissions on WLTC cycle with cold and hot start - Ref. Fuel E10 and 2 lubricants (standard and "full SAPS")

As expected, the PN emissions are far higher when the engine is cold. The increase is about 50% for the measurements with VPR, including ICAD, PMP and CPC3776, and can reach 150% in the case of the DMS500 > 5 nm because of the high sub-23 nm fraction in the aerosol. Indeed, this sub-23 nm fraction strongly increases when the engine is cold, of about 30 percentage points. It means that cold start and warm-up phases are not only strong contributors to the total PN emissions, but that they are also huge emitters of sub-23 nm particles. Based on DMS500 measurement, the number of particles in the 5 – 23 nm range is about 5 times higher in cold conditions than in hot conditions.

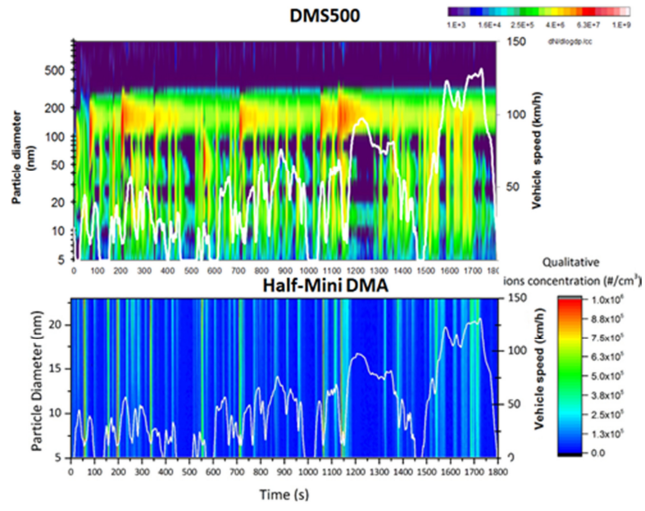

Figure 14: Comparison of the PSD vs time with DMS500 and Half-mini DMA – WLTC hot conditions – Fuel with high aromatic content.

Furthermore, a comparison between the Figure 11 (WLTC in cold conditions) and Figure 14 (WLTC in hot conditions) underlines that the initial cold start contributions of sub 23 nm particles observed by the HM-DMA are vastly larger (and extend to far smaller sizes) than those detected by the DMS500. This striking observation confirms the importance of the development of new instrumentation specially targeting such small particles

CONCLUSION

Numerous PN measurements devices, with different ranges, commercial and prototypes, were evaluated at the tailpipe of a Euro 6b gasoline-DI vehicle on **multiple operating conditions**: **driving cycles**, **cold start and hot start**, **fuels**, **lubricants**. From these tests, several conclusions can be drawn:

- The **fuel formulation** has a strong impact not only on the total PN emissions at the tailpipe, but also on the fraction of the sub-23 nm particles. For example, E25 fuel or high sulfur content fuel can emit a higher fraction of small particles than the reference E10 fuel.

- The **driving cycle** has also an impact, both on the total PN emissions and on the fraction of these sub-23 nm particles. It was demonstrated that the aggressive RTS95 cycle emits 3 times more PN than the reference WLTC with a reference E10 fuel. With the high sulfured fuel, the total PN is 10 times higher when considering particles down to 5 nm, including the volatile fraction.

- Two different **lubricants** were also evaluated. If the total PN > 23 nm, that is the current diameter cut-off imposed by the PMP procedure, is not affected by the lubricant, an increase of SAPS can lead to higher PN emissions in the range 5 – 23 nm, whether volatile or solid particles.

- The **start temperature** is already known to be an important factor for total PN emissions. The tests presented in this paper confirm this trend, and underline that the huge quantities of particles in the range 5 – 23 nm are emitted during cold start and warm-up phases.

In all cases, the use of a catalytic stripper or a volatile particle remover is useful to remove the volatile fraction of the aerosol, that can bring some artefacts in the measurement. The same result is also achieved with the novel approach of sampling from a single hot dilution stage followed by particle measurement systems at higher than the typical temperatures. By using these two approaches, only solid particles are measured, making the results more robust. It is noticeable that even if these devices reduce the volatile fraction of the particles, some particular conditions could lead to a significant contribution of sub-23 nm solid particle emissions.

ACKNOWLEDGMENTS

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ACRONYMS

CPC: Condensation Particle Counter
CS: Catalytic Stripper
DMA: Differential Mobility Analyzer
DR: Dilution Ratio
GDI: Gasoline Direct Injection
HM-DMA: Half-Mini DMA
ICAD: Induced Current Aerosol Detector
PSD: Particle Size Distribution
SAPS: Sulfated Ashes Phosphorus and Sulfates
SESI: Secondary ElectroSpray Ionization
PFI: Port Fuel Injection
VPR: Volatile Particle Remover
WLTC: Worldwide Harmonized Light Vehicles Test Cycle