# Measuring automotive exhaust particles down to 10 nm

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## Abstract

The latest generation of internal combustion engines may emit significant levels of sub-23 nm particles. The main objective of the Horizon 2020 “DownToTen” project was to develop a robust methodology and provide policy recommendations towards the particle number (PN) emissions measurements in the sub-23 nm region. In order to achieve this target, a new portable exhaust particle sampling system (PEPS) was developed, being capable of measuring exhaust particles down to at least 10 nm under real-world conditions. The main design target was to build a system that is compatible with current PMP requirements and is characterized by minimized losses in the sub-23 nm region, high robustness against artefacts and high flexibility in terms of different PN modes investigation, i.e. non-volatile, volatile and secondary particles. This measurement setup was used for the evaluation of particle emissions from the latest technology engine and powertrain technologies (including vehicles from other Horizon 2020 projects), different fuel types, and a wide range of exhaust aftertreatment systems. Results revealed that in most cases (non-volatile), PN emissions down to 10 nm ($SPN_{10}$) do not exceed the current $SPN_{23}$ limit of $6\times10^{11}$ p/km. However, there are some cases where $SPN_{10}$ emissions exceeded the limit, although $SPN_{23}$ were below that. An interesting finding was that even in the latter cases, the installation of a particle filter could significantly reduce PN emissions across a wide particle size range, fuels, and combustion technology. DownToTen results are being used to scientifically underpin the Euro 7/VII emission standard development in the EU. The method developed and the results obtained may be used to bring in the market clean and efficient vehicle technologies, improve engine and emission control performance with different fuels, and characterize size-fractionated particle chemistry to identify the formation mechanisms and control those in a targeted, cost-effective fashion.

## Introduction

Over the past decade, the European Union (EU) has taken several steps to reduce the human health impacts of particulate matter (PM) from transport, complementing the control of the mass of PM with limits on the numbers of non-volatile (or solid) particles (PN) initially for diesel vehicles (2011) and later for gasoline direct injection (GDI) vehicles and on-road engines (2014), inland waterway vessels (>300 kW) and rail traction engines since 2017. For light-duty vehicles (LDV), tests have been needed on-road real driving emissions (RDE) since 2017 (Regulation (EU) 2017/1151). A minimum size threshold at 23 nm particle diameter has been set in order to include the smallest soot particles and exclude volatile nucleation mode ones. Concerns have been raised, however, that current cut off size might not be appropriate for some modern combustion engine technologies because high concentrations of solid sub-23 nm particles have been found. In particular, for port-fuel injection vehicles[1], mopeds and motorcycles[2][3] along with diesel particle filter regenerations[1,4], evidence had shown that there is a considerable percentage of particles below 23 nm. Especially, sub-23nm emissions of PFIs during cold start, were up to 4 times higher than SPN23[1]. First, concerns were raised for spark ignition direct injection vehicles where particle emissions were found to be higher than DPF equipped diesels of the same generation [5,6], but further research showed that other vehicles such as CNGs might also emit a significant number of sub-23 nm particles [7]. Such non-volatile nucleation or core mode particles are soot particles originate from incomplete combustion of fuels in fuel rich zones [8,9] or lubricant derived particles [10,11] while the emissions depend on driving behavior or aftertreatment system [5,6]. Responding to these concerns, the EU launched the GV-02-2016 Call for ‘Technologies for low emission light duty powertrains’ with a special emphasis on the sub-23 nm particle characterization. DownToTen (DTT) is one of the three projects that have investigated the topic.

The DTT consortium was established to develop further knowledge on particle emissions from light duty vehicles and a robust and sound method for the measurement of sub -23 nm particles. The main aims of the project were:

- To increase the understanding of the nature and the characteristics of sub-23 nm particle emissions.
- To provide a robust sampling and measurement methodology for laboratory and RDE measurements.
- To assess the effectiveness of technical measures for reducing particle emissions.
- To provide input on emissions factors for particle number emissions from current and future technologies to enhance air quality modelling tools.

The first step was to gather the available data and information on particle emissions from the latest vehicle technologies, including hot exhaust aerosol (solid particles), fresh exhaust aerosol (total particles), and aged exhaust aerosol (secondary particles) from a variety of technologies and fuels. The survey provided critical information to distinguish between particle formation from fuel, lube oil, additives, engine wear, or via storage/release mechanisms in the exhaust of after-treatment systems. Also, the study produced data to select the currently available sampling and instrumentation devices to meet the new targets both in the laboratory and RDE conditions. The identification of the limitations and constraints of the available instrumentation suggested necessary modifications and upgrades that were addressed in the next steps.

## Main objectives and methodology

### *Objectives of the DownToTen project*

The basic target of DownToTen (DTT) was the development of a reliable and robust methodology that should enhance the regulatory approach in the assessment of particle number emissions in the sub 23 nm region (down to at least 10 nm). The size of 10 nm was selected to ensure that sub 23 nm particles are regulated while avoiding measurement artefacts that may arise in the <10nm range (particle losses, re-nucleation of volatiles, or pyrolysis). The focus was on PN emissions of the new generations of internal combustion engines under real world operating conditions. Following a request from the Commission, after the project had commenced, DTT uniquely expanded its focus to study the production of <23 nm PN from ICEs, in detail. In parallel, the project aimed at complementing the in-cylinder particle formation and particle filtration research being undertaken in linked technology development H2020 projects (uPGrAdE, PaREGEn, DiePeR).

To this end, the objectives and targets of DTT were:

- to understand better the nature and the physicochemical characteristics of sub-23nm particles for the facilitation of metrology and evaluation purposes
- to provide a reliable sampling and measurement methodology for both laboratory and real-world conditions
- to use the above to measure a number of current and future engine and vehicle technologies as well as state-of-the-art exhaust aftertreatment systems in the laboratory and in real world conditions
- to develop a model to simulate particle transformation during sampling and sample conditioning processes utilizing experimental evidence from the project
- to develop a PN-PEMS demonstrator with high efficiency in determining PN emissions of current and future engine technologies in the real world.

### *Sampling and measurement setup*

DownToTen has developed a new sampling system that respects the main principles for the measurement of non-volatile particles in the EU but offers enhanced characteristics to enable the measurement of particles below 23 nm (Figure 1). The main principles followed include the sampling dilution under hot conditions (hot dilution air at 150-200degC), the subsequent treatment of the exhaust aerosol in the volatile particle remover (VPR) under high temperature conditions (300degC wall temperature) and, finally, a subsequent dilution to further decrease concentration and bring the temperature to atmospheric conditions. A final ejector dilutor (ED) can be used at times to further decrease concentrations. Dilution ratios at the first and second dilution stages are adjustable but typically in the range of 10:1. The sampling system can be used to sample particles directly from the tailpipe or follow primary dilution in the CVS. In this paper, we do not separate CVS from tailpipe measurements initially, except from investigation on specific vehicles and fuels that were conducted under tailpipe conditions and it will be discussed further below

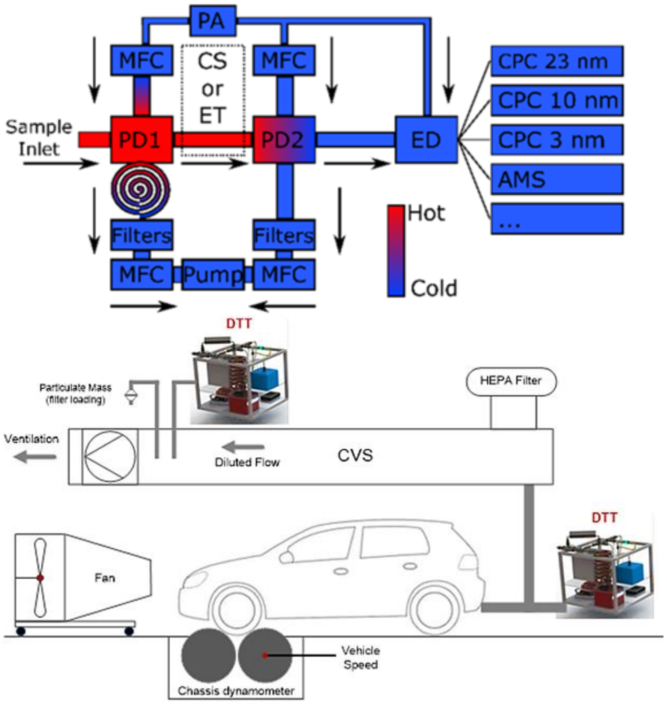

Figure 1 An illustration of EU HORIZON 2020 DownToTen sampling system (above). Dilution on PD1 and PD2 is performed with porous tube diluters. Several particle detection devices can be placed at the end of the sampling. MFC: Mass flow controller, ED: ejector dilutor used to further decrease concentrations, when necessary. The system is suitable for tailpipe and CVS testing (below).

To achieve low particle losses, the dilution in both stages is conducted with porous tube diluters, which have, for long been known to induce low wall losses [12,13]. Moreover, the DownToTen consortium decided to perform most of the measurements utilizing a catalytic stripper (CS) as a VPR to treat the aerosol in-between the two dilution stages. This is not necessarily a recommendation for developing the regulatory protocol and, definitely, not a recommendation when particle chemistry needs to be studied. However, as the initial focus was on non-volatile particles, we expect that the CS will have enhanced capacity to remove volatile and semi-volatile species over an evaporation-tube type of VPR [14–16]. An additional difference from the PMP protocol concerns the sampling flowrates; in the DownToTen device enough diluted sample is provided to serve more than one particle counting and sizing instruments. The sampling system was developed as a first stage prototype in meeting DownToTen's ultimate aim of developing a portable exhaust particle sampling system (PEPS) with the capacity to measure particles <23nm.

Overall, particle losses of the system have been characterized, utilizing monodisperse and polydisperse aerosol [17]. With the porous tube dilutors, thermophoretic losses were limited to less than 5%, regardless of particle size. The majority of size-dependent losses occurred in the CS due to diffusional losses. Optimization of the CS led to constraining overall losses to less to than 40% at 10 nm and 20% at 20 nm. Further optimization of the system could result in a less steep loss curve with particle size if so required for regulatory purposes. In particular, CS optimization is a space-velocity trade-off between semi-volatile oxidation and solid particle losses. Apart from dilution correction all of the measured levels with this sampling system in this paper have been corrected for particle losses. Depending on particle size distribution, total emissions corrected for particle losses which are at 10% or below for $SPN_{23}$, around 30% in the 10-23 nm range, and in the order of 60-70% for particles <10 nm [17]. The average Particle Number Concentration Reduction Factor (PCRF) at 30, 50 and 100 nm values determined at the DTT sampling system, using golden instrumentation with soot generator, agreed within ±5%, with an average value at 8%.

Utilized CS, incorporates a sulfur trap to minimize the risk of potential artefacts caused by $SO_2$ oxidation to sulfate particles. The volatile particle removal efficiency remained in excess of 99.99% for polydisperse emery oil particles at concentration of $5mg/m^3$ , which were substantially higher than required automotive regulations. In addition, Bainschab et al. [17] studied the potential for artefact creation downstream of the utilized CS at different inlet sulphur concentrations. Artefacts due to new particle formation (nucleation) or particle growth within the measured size range due to condensation were very low under all realistic sulphate concentrations established. Still, the capacity of CS for sulfur storage is an additional optimization parameter, especially when low sulfur fuel is not available [18].

Three practically identical prototype sampling systems were built and were used in four different laboratories (AUTH, AVL, JRC, and Ricardo) to perform the different tests. Prior the tests, all the three sampling systems were tested and characterized for particle losses in order to minimize system to system measurement variability. A range of counting and sizing instruments were available in the various laboratories to measure particles of different sizes downstream of the sampling system. As regards the measurement equipment that involved in the tests, each laboratory followed a specific pre and post testing sequence which was based on the gained knowledge from previous research projects in the field (Particulates) as well as the guidelines from the studies of Particle Measurement Program (PMP) and JRC relevant work [19–21].

Specifications of the different devices used are shown in Table 1. The two CPCs were used downstream of the DownToTen sampling system, while the AVL APC was used for comparison in some of the measurements. Warm up, calibration and zeroing of each device was mandatory prior to each test to maintain a high level of measurement validity. When required, CPC coincidence corrections were applied according to the manufacturer for each device. Depending on each lab's configuration, some measurements were conducted at the CVS, and some were conducted at the tailpipe. Although the exact placement of the measurement does have a role to play when certification measurements are conducted, we do not expect significant impacts on the overall emissions general trends that we initially examine in the current paper. However, investigation on specific vehicles and fuels conducted under tailpipe sampling conditions in order to decrease potential test to test uncertainties further.

Table 1 Technical characteristics of the main particle detection instrumentation used in the different measurements of this work

| | CPC TSI 3776 | CPC TSI 3010 | AVL APC |
|---|---|---|---|
| Particle size range (nm) | >2.5 | >10 | SPN>23 (PMP) |
| Concentration range (part./cm$^3$) | $0 – 3\times10^5$ | $0 – 10^4$ | $0 – 5\times10^4$ |
| Time resolution/ acquisition frequency (Hz) | 1 | 1 | 10 |

## *Vehicle samples and tests*

During this study, fourteen passenger cars with the addition of heavy duty, non-road machinery and mopeds of different engine, emission control and fuel technologies were investigated, including different aftertreatment devices and fuels. In addition, one engine concept where combined port fuel injection (PFI) and GDI operation could be commanded is included for comparison. All light duty vehicles were compliant with at least Euro 6b emission standard. Although the latest $SPN_{23}$ limit of $6\times10^{11}$ part/km was not applicable to all technologies tested (such as for example the PFI and CNG ones), it is used as a reference throughout the study to compare the observed emission levels. Figure 2 shows the range of technologies that have been tested, split per fuel. For comparative purposes with real-world emissions, market fuel was used in the current study. With regard to CNG combustion, there starts to appear a large diversity in combustion principles (PFI, GDI, dedicated vs bi-fuel, etc.). CNG monovalent DI vehicles are not commercially available at the moment (2020). Even though such types of vehicles were not involved into the investigation, improvements in CNG combustion may change the final result of this analysis. The current investigation only involved market-available CNG vehicles that are bi-fuelled, running primarily on CNG under PFI mode and on gasoline in GDI mode, when CNG is depleted. By observing the on-board diagnostics (OBD) signals recorded, we identified no gasoline inter-injections for any of the tests conducted. As regards hybrid vehicles those were HEV types in all cases as the Figure 2 shows. A variety of engine and aftertreatment technologies were tested on a wide range of global regulatory cycles, discrete operating conditions and pre-selected speed & load patterns designed to explore particle production events. The passenger vehicles in the Figure 2 are the "core" of tested vehicles that used in the analysis. Apart from the vehicles from Figure 2, heavy duty and road machinery where also investigated in order to acquire a well-rounded knowledge of 10nm particle emission performance with DownToTen system. In general, the results were gathered in a number of categories which include:

- Engine technologies / aftertreatment combinations (including 6d-temp and final light-duty applications)
- Emissions certification standard: At least Euro 5 through to Euro VI-C and Euro 6d-Final
- Regulatory cycles from around the World
- Extreme operation (beyond the velocity, dynamics and temperature boundaries of regulatory cycles)
- Including environmental temperature extremes (down to -10°C, up to 30°C)
- Fuel variations
- SI fuels to >25% Ethanol, and CNG
- CI fuels to 30% biodiesel, and paraffinic diesels

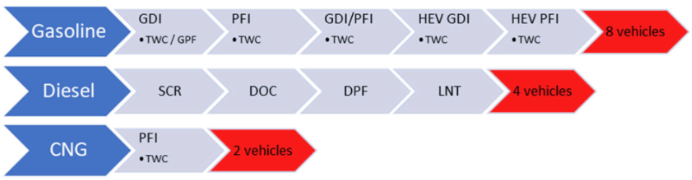

Figure 2 Test matric of passenger cars that included several fuels combustion and exhaust aftertreatment technologies. Fourteen vehicles were investigated under different aftertreatment setups, resulting to 31 individual configurations summarized in this study.

## Results and Discussion

### *Non-volatile particle number down to 23 nm*

Results are shown in a normalized format where the x-axis of the chart shows emissions of $SPN_{23}$ divided by the current regulatory limit values and converted to a percentage. The y-axis shows emissions of non-volatile particles, either with a lower size threshold of 10 nm ($SPN_{10}$) as in (Figure 3), or with a lower size threshold of < 10 nm ($SPN_{<10}$), similarly normalized to limit values as will be seen in later figures. In practice the $SPN_{<10}$ data contains data measured with particle counters with 7 nm, 4 nm and 2.5 nm d50 cut-points. At the highest level, technologies in these figures can be identified by the shape of the marker used. For example: diesel emissions are always shown as circles, GDI as squares and CNG as triangles. The data shown in Figure 3 contains approximately 260 separate results for $SPN_{23}$ and $SPN_{10}$ [covering 5+ orders of magnitude], with data supplied by Ricardo, LAT, TUG, AVL and JRC. There are fewer results in $SPN_{<10}$ data, but these still amount to almost 220 individual results. When calculating percentage emissions, the following limits were employed:

- Light-duty vehicles*6x10$^{11}$ part./km
- Heavy-duty engines' transient testing 6x10$^{11}$ part./kWh
- Heavy-duty engines' steady state testing 8x10$^{11}$ part./kWh
- Non-Road mobile machinery (NRMM) testing 1x10$^{12}$ part./kWh.

* including Euro 6b GDI vehicles that were designed for a 6x10$^{12}$ part./km limit

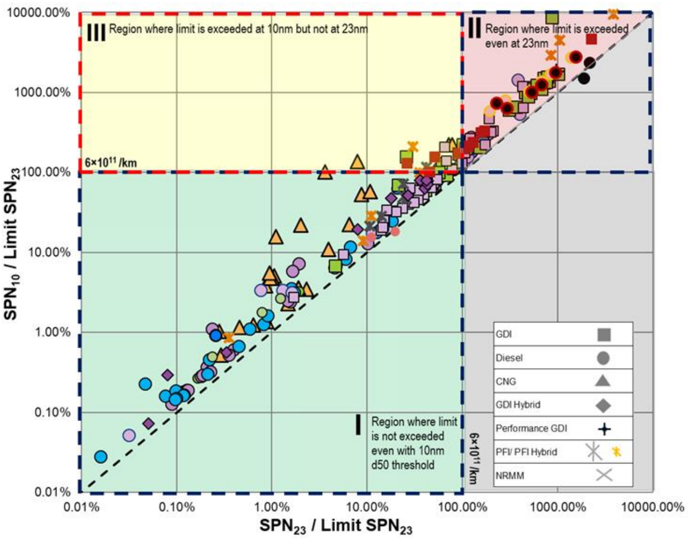

Figure 3 Ratios of particle emissions over the SPN23nm limit for different vehicle configurations. The critical zone is No III where some technologies exceed the limit only for the SPN10nm but not for the SPN23nm measurements

As shown in Figure 3, results can be identified to lie within one of three regions:

I. Region I – the region where emissions of $SPN_{23}$ and $SPN_{10}$ are compliant with current limit values for PN. This region is of limited interest, but it does show where current technology strengths in PN control lie, and where all technologies' emissions should eventually reside. Most technologies are compliant with 6x10$^{11}$ part./km limit for both >23 nm and >10 nm.

II. Region II - the region where both $SPN_{23}$ and $SPN_{10}$ emissions are greater than the current limit value. This region is of interest, as it shows where current technology weaknesses in PN control lie. Emissions above the limit values for both $SPN_{23}$ and $SPN_{10}$ are apparent from non-DPF diesels and a moped, with emissions also high from high-performance port-injection gasoline vehicles, especially for low temperature (-7°C) testing. Some technologies emit between 6x10$^{11}$ (100% of the limit) and 10$^{12}$part./km (167% of the limit): PFI motorcycles (Euro 5), some diesels with DPFs (up to Euro 6dt) due to regeneration, and a few Euro 6dt-GDI vehicles tested over non-regulatory cycles. Three-way catalyst equipped Euro 6b GDI vehicles emit around the earlier PN limit of 6x10$^{12}$part./km for vehicles of this certification standard. Those results above the Euro 6b limit are generally from cycles other than WLTC or NEDC (Figure 3).

III. Region III - the region where emissions of $SPN_{23}$ are compliant with the current limit value, but emissions of $SPN_{10}$ or $SPN_{<10}$ exceed the limit value. This region is of great interest, as it comprises technologies with PN<23 emissions that should be targeted for further development effort to bring results into Region I, if the particle size threshold was to be decreased. According to our research, these technologies include motorcycles and mopeds on standard cycles (WMTC and R47), Euro 6b PFI and GDI vehicles without GPFs on WLTC and DPF-equipped diesels after regeneration. GDI hybrid emissions are at the borderline without the

GPF or fail at $SPN_{23}$ as well as $SPN_{10}$. Euro 6b and 6dtemp GDI vehicles (with retrofitted and production GPFs, respectively) on WLTC and more severe urban cycles are above the limit for $SPN_{10}$, but most GPF-equipped vehicles have 'fresh' GPFs where ash accumulation is minimal and filtration efficiency is at lowest possible levels. The CNG PN emissions are significantly elevated below 10 nm, with 90 times as many particles when extending downwards to 2.5 nm. These particles are thought to be metal oxides derived from additive metals in the lubricant oil that exist independently, due to low in-cylinder soot concentrations. However, retrofitting of a GPF to a CNG vehicle, just for experimentation, has been seen to reduce both $SPN_{10}$ and $SPN_{<10}$ to well below the limit value (Figure 3 and Figure 4).

There is a fourth, unnamed, region shown in grey, in which PN emissions in ranges starting from <23nm would indicate lower levels than PN emissions in the range >23nm. Clearly this is unlikely unless the particle number counters used in parallel are noticeably different in the >23nm region. Very few results were detected in this region during the project.

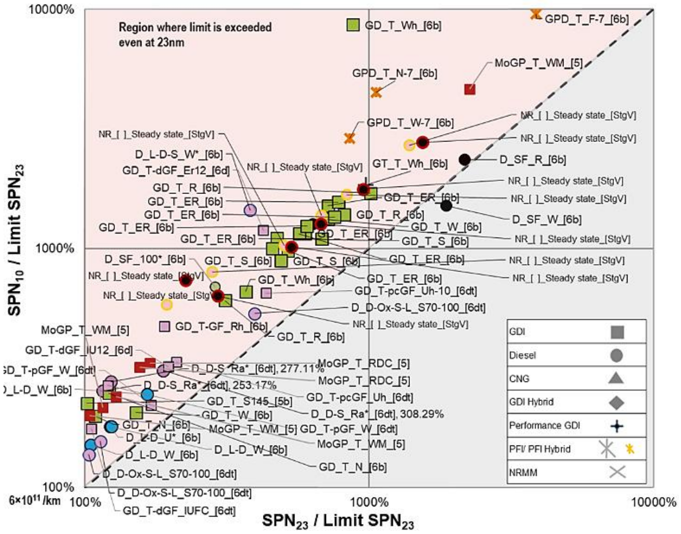

Figure 4 Results from Region II where both SPN23 and SPN10 emission are greater than the current value

As discussed previously, Figure 3 and 4 cover a wide range of engine technologies and vehicle types under various test conditions that include CVS and tailpipe measurements. This mainly happened in order to acquire a holistic approach and understanding of what is the sub-23nm particle emission performance, with respect to the previous parameters and basically the previous figures are the input of the DownToTen project as regards the measurement part. Even though from regulatory point of view, laboratory PN must be measured from diluted exhaust at the CVS, there are reports that suggest tailpipe sampling is less prone to artifacts[22] with CVS PN emissions to be an order of magnitude higher than at the tailpipe [23]. Thus, the following figures and analysis focus on specific vehicle types (passenger cars) at the same test conditions (tailpipe sampling, temperature etc.), so as to draw a solid conclusion as regards the sub-23nm particle emissions behavior at the given powertrain/aftertreatment configuration or vehicle segment.

Hence, figure 5 shows average $SPN_{23}$ levels over cold WLTC tests, grouped by main fuel, aftertreatment and powertrain configuration. In particular for GDI vehicles, a split according to the existence of a GPF or not is shown. All vehicles but some Euro 6 GDI ones appear below the level of the current $SPN_{23}$ limit of $6\times10^{11}$ part/km, even if the limit is not applicable to all vehicles. In particular, a PFI hybrid produces particle concentrations which are much below typical ambient levels and hence total emissions are one order of magnitude below the limit level value. A single GDI hybrid vehicle is also found at very low levels, even without being equipped with a GPF. This shows that advanced hybridized powertrains and latest GDI technologies can achieve very low $SPN_{23}$ emission levels, even if powered by direct injection engines.

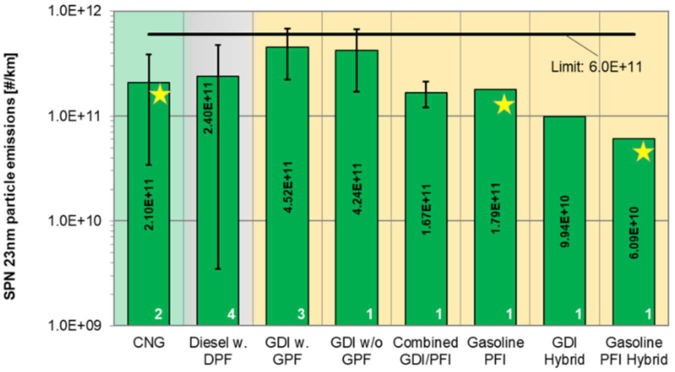

Figure 5 Average (±standard deviation) aggregated SPN23nm particle emissions over cold-start WLTC tests, grouped by major powertrain/aftertreatment configuration. The number of vehicles per configuration is shown on the side of each bar. Also, an asterisk marks those configurations for which the SPN23nm limit is not applicable.

Diesel vehicles and conventional PFI ones are roughly at the same levels, about 3-4 times below the limit. In particular, diesel vehicles exhibit a rather large variation, as shown by the wide error bars, with some of them emitting below $10^{10}$ part./km levels and some closer to the limit. The wide range is mostly observed because some tests were executed immediately following DPF regeneration while some others were executed with the DPF properly loaded with soot. The two PFI CNG vehicles, although on average appear to emit much below the limit, also exhibit the second largest variation following DPF equipped vehicles. The significant difference in the performance of the two CNG vehicles is further discussed below.

Interestingly, the average emission levels between GPF-equipped and non-GPF equipped GDI vehicles were not found to be very different, in fact the rather high emission levels reflects the necessity for a further optimization of GPF technology that would have a positive impact on particle emission reduction especially during specific events (passive regeneration). This contrasts the diesel experience where implementation of a DPF can decrease $SPN_{23}$ emission levels by three orders of magnitude. Latest Euro 6 GDI, such as the hybrid one, show that $SPN_{23}$ could be well below the limit even w/o GPF and that use of a GPF may decrease emission levels, actually not dramatically, but just to retain compliance with the regulatory limit. Additionally, the GPF filtration efficiency is lower that the diesel one due to limited soot cake formation in the GPF compared to the DPF [24,25]. As our measurements were mostly performed with cleaned-up GPF systems, we would expect somehow elevated PN levels downstream of the GPF. The mixed performance for GDI vehicles is hence mostly due to latest low emitting GDI combustion systems without GPF and older, less developed systems, where GPF is used to bring the emissions in line. We should expect even the clean combustion systems to be equipped with GPFs in the end.

Figure 6 extends the findings of Figure 5 by also looking at the emission performance below 23 nm. Depending on the laboratory that performed the measurements, data exist for SPN$_{10}$, that is non-volatile particles down to 10 nm and also non-volatile particles down to 2.5 nm or down to 4 nm.

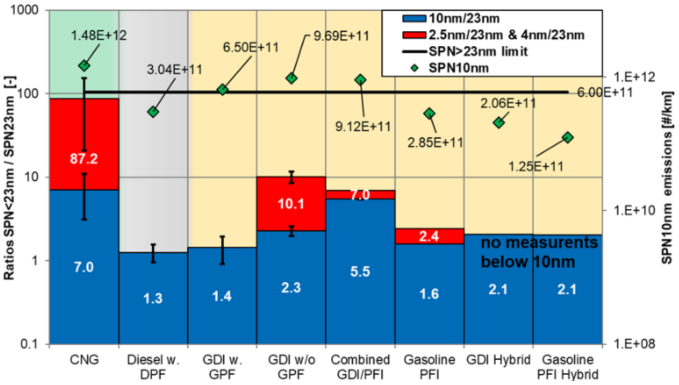

Figure 6 Solid particle emission ratios and aggregated levels for different engine technologies. Measurements were performed using the lab version of DownToTen system. Error bars show the standard error of the mean including 4 to 13 repetitions. Lack of error bar indicates a unique measurement. The horizontal bold line corresponds to current GDI and diesel SPN23nm limit ($6\times10^{11}$ part/km) and is provided here only as a guide to the eye

Some general trends may be observed according to emission control technology. Starting with the GPF / non-GPF GDI comparison, the GPF clearly decreases the number of particles below 23 nm, regardless of whether one looks at SPN$_{10}$ or smaller sizes. In GDI vehicles w/o GPF (hybrid or not), there appear to be more particles in the 10-23 nm range than above 23nm (the SPN$_{10}$/SPN$_{23}$ ratio is above 2). On the contrary, for vehicles with GPF, extending to 10 nm increases SPN by 44%, similarly to the DPF case (26%). This indicates that the GPF is more efficient for <23nm PN than for >23nm PN, a finding most probably driven by diffusion collection of small particle sizes in the GPF.

Two cases significantly stand out in terms of SPN$_{10}$/SPN$_{23}$ ratio. The first is the PFI CNG vehicles and the second is the combined GDI/PFI where the majority of particles seem to reside below 23 nm. With respect to the PFI CNG, it appears that particle size distribution actually extends significantly below 10 nm as well, with 90 times as many particles when extending to 2.5nm. Such small particles are often considered to derive from lube oil and not combustion [26–28] and are considered to be ash or heavy organic species. The DownToTen project will be chemically analyzing particles to validate the nature of these nanoparticles. Regardless of their formation pathway, it seems here that the SPN$_{23}$ limit is not effective to limit total non-volatile PN emissions. For the combined GDI/PFI, the particle size distribution does not substantially extend below 10 nm hence a limit at SPN$_{10}$ would be effective in this case. Finally, one should notice the practically zero number of non-volatile particles below 10 nm (before particle loss correction) for the DPF vehicles, which could be explained by the high DPF efficiency by diffusion at this size range. High DPF efficiency comes with soot cake formation in the filter, and the DPF becomes almost an absolute filter. In our tests, it seems that soot cakes very rarely formed in the GPFs, hence filtration efficiencies even of the best GPFs were below those of DPFs.

## *Investigation on specific vehicles and fuels*

With the reduced PN levels currently achieved by using particulate filter technologies for diesel and GDI vehicles, emission levels of other powertrain technologies become relatively important. Here, we focus on CNG and hybrid vehicles, which are considered as ‘’clean’’ vehicles and as an element on the path towards energy independence from fossil fuels. Both vehicle types are steadily increasing their market share and they are expected to gain higher market share in the years to come, mainly due to their low CO$_2$ emissions, either directly from the tailpipe or over the full lifecycle. As discussed earlier, the following tests were made at the same lab, under the same tailpipe sampling conditions, in order to avoid potential test to test measurements uncertainties, that may arise from sampling conditions at the dilution tunnel (condensation, nucleation)[22,23].

### Natural gas engines

Natural gas is considered more attractive from an atmospheric pollution standpoint and one advantage of the CNG vehicles is their ability to meet stringent standards with less complicated emission control systems. While CNG vehicles based on different combustion principles (PFI, GDI, dedicated vs bi-fuel, etc.) start to appear, the current investigation focuses only on market-available CNG vehicles that are designed as bi-fuel engines and run on CNG and gasoline. Latest research on the field indicates better SPN$_{23}$ particle emission performance of CNG when compared to gasoline and on average, CNG SPN$_{23}$ emissions were comparable with a DPF equipped diesel vehicle [5,6]. However elevated sub-23nm emission results provide a different insight of CNG overall particle emission performance [7].

Figure 6 showed that the number of particles below 23 nm for PFI CNG and GDI w/o GPF is much higher than those above 23 nm. A large number of non-volatile sub-23 nm particles has been also observed in the past with regard to the emissions of GDI vehicles [29]. In some cases, Giechaskiel et al. attributed this to particle formation downstream of the evaporation tube due to the high concentrations of volatile and semi-volatile species, i.e. those particles could be considered as sampling system artefacts [3]. Specific measurements (and all CNG ones) have been conducted directly at the tailpipe without the CVS. Moreover, we have used a catalytic stripper and not an evaporator VPR for enhanced removal efficiency. Hence, the possibility for measurement of particles which are not true vehicle emissions is in principle much lower than in the original PMP setup.

Table 2 Typical characteristics of prototype catalyzed particle filters used in the study. Current filters used during CNG and gasoline measurements with and without additives.

| Filters used | Wall thickness [mils] / Cell density [cpsi] | Mean pore size / Porosity |
|---|---|---|
| PF 1 | 8 /300 | Large / High |
| PF 2 | 10 / 300 | |

Figure 7 show SPN$_{10}$/SPN$_{23}$emissions and SPN$_{2.5}$/SPN$_{23}$ emissions of the three CNG vehicles over a cold WLTC compared to limit. The emission of GDI, PFI with and without GPF along with Hybrids are also shown for further comparison. CNG results are indicative of how sub 23nm particle emissions can be. Despite the fact that CS is used as an enhanced method to remove volatiles, there are some cases that high sub-23nm emissions were observed. Even though CNG SPN$_{10}$ and SPN$_{2.5}$ emissions span to a greater area (Region I and II) than

GDI or PFI respected operation, the addition of particulate filter in the exhaust line reduces significantly (up to two orders of magnitude) the particulate emissions levels.

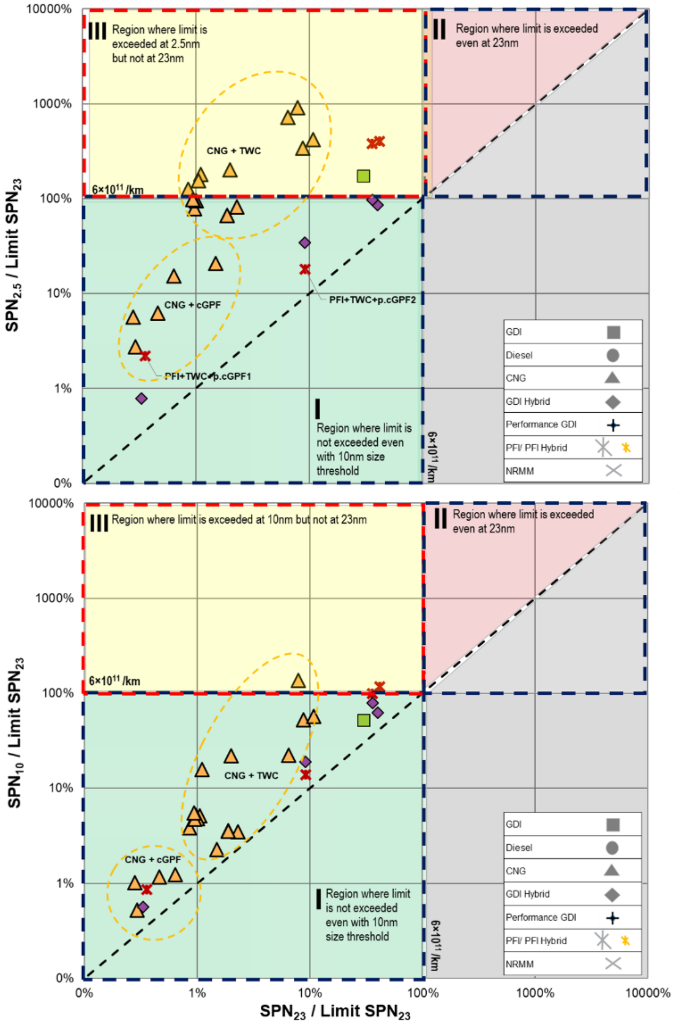

Figure 7 SPN10nm, SPN2.5nm along with SPN23nm compared to the limit. With yellow triangles are all the CNG tests corresponding to WLTC cold measurements. For comparative purposes gasoline PFI as well GDI hybrid results are also depicted.

The $SPN_{10}$ emissions of the CNG vehicles are low. It seems that their particle size distributions extend significantly below 10 nm, with several times as many particles when extending to 2.5 nm (region III in Figure 7). As it was said previously, such small particles are often considered to derive from lube oil and not combustion and are considered to be ash or heavy organic species [26–28,30]. Gasoline PFI without PFs emitted also significant numbers of $SPN_{2.5}$ particles (region III) whereas vehicles equipped with PFs emitted still below limits that were more than 10times lower than non-PF $SPN_{2.5}$ emission results (region I) In particular $SPN_{10nm}$ emission levels with PF where comparable with DPF equipped diesel vehicles. Although there is an improvement in sub-23nm particle emission performance, current PFs, which were used in this study, are prototype ones and a fully optimized filter is expected to reduce sub-23nm particle emissions further, especially during specific engine events (passive regeneration). Figure 8 summarizes the SPN particle emissions of the CNG vehicles. Also shown are the results obtained using standard market gasoline fuel with MMT additive (an octane booster), and from low-sooting alkylate gasoline. In Table 3 are some typical specification of the liquid and gaseous fuels along with the MMT additive used during the test campaign.

Table 3 Typical specifications of the fuels and additive used in this test campaign

| Fuels / Additives | Octane [-] / Mn concentration [mg/$l_{mixture}$] | Lower Heating Value [MJ/kg] |
|---|---|---|
| E10 | 82 | 43.5 |
| Alkylate | 92-95 | 44.2 |
| CNG | 120+ | 48.5 |
| MMT (additive) | 30 mg Mn/$l_{mixture}$ | [-] |

PFs reduce significantly the particle emissions and seem to enhance sub-23 nm filtration. However, high $SPN_{10}$ and $SPN_{2.5}$ emissions are observed even with "clean" aromatic-free fuels (alkylate gasoline). Finally, with the MMT added in the gasoline, the emissions of sub-23 nm particles become the highest. As it was also observed in Figure 7, the addition of particulate filter reduces significantly particle emissions from both gasoline and CNG fuels to at least one order of magnitude. Additionally, the utilization of prototype particulate filter reduces drastically the sub23nm particles in all cases. Further optimization of gasoline particulate filters would further optimize the emission performance.

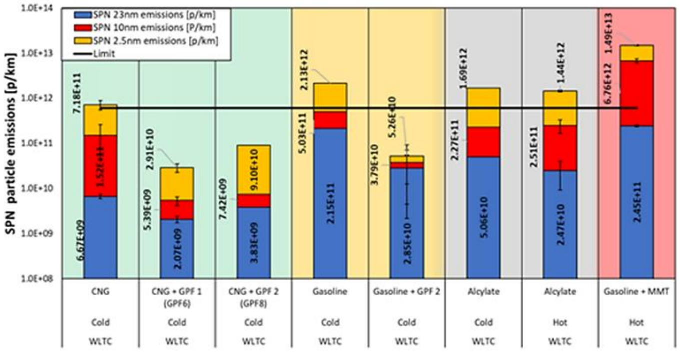

Figure 8 Average emission ratios with different fuels and additives along with different prototype particulate filters. The results are referred to a PFI Euro 6d-temp (CNG/Gasoline) vehicle.

## Hybrid vehicles

Hybrid drivetrains aim to synergistically combine the internal combustion engine and the electric motor to fully capitalize on these fundamental characteristics for both reduced fuel consumption and emissions reduction. Continued cost reductions in electric drivetrains and battery technology combined with further development of ICEs especially designed for hybrid drive can lead to higher efficiency, lower costs and a better driving experience.

Interestingly, PN emissions from hybrid-electric vehicles were found to be higher than their gasoline counterparts in a study performed by Yang et al. [31], but most recent studies focus on engine efficiency or gaseous emissions that excludes particle emissions [32,33]. Thus, a clear picture of particle emissions from all such different powertrain types cannot be provided based on current literature. In Figure 9 the evolution of $SPN_{23}$ along with $SPN_{10}/SPN_{23}$ (red line) ratio during a charge depleting mode with high initial state of charge (SOC) are depicted. Elevated particle emission can be seen during initial cold start. In addition, higher ratios of $SPN_{10}/SPN_{23}$ (red line) can be seen during that period. Also, specific driving events that require high engine demand for a short period of time can initiate the internal combustion engine. Such an event can be seen in the beginning of the test cycle.

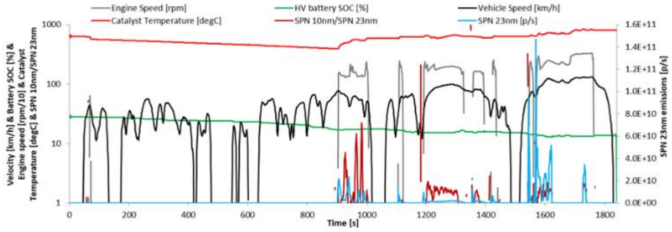

Figure 9 Evolution of SPN23nm emissions along with SPN10nm/SPN23nm ration during WLTC cold with high initial SOC, under Charge depleting battery mode.

Figure 10 show $SPN_{10}/SPN_{23}$ emissions of hybrid vehicle on cold WLTC compared to limit. As shown, the PHEV vehicle in charge depleting mode (EV) has similar emission performance as in charge sustain mode (HEV) at low SOC. However, significant differences between EV and HEV modes are observed with SPN emissions decreasing as the initial state of charge (SOC) rises in EV mode. High $SPN_{23}$ peaks only occur during the cold start in the HEV mode. Both $SPN_{23}$ and $SPN_{10}$ remain within limits in both modes.

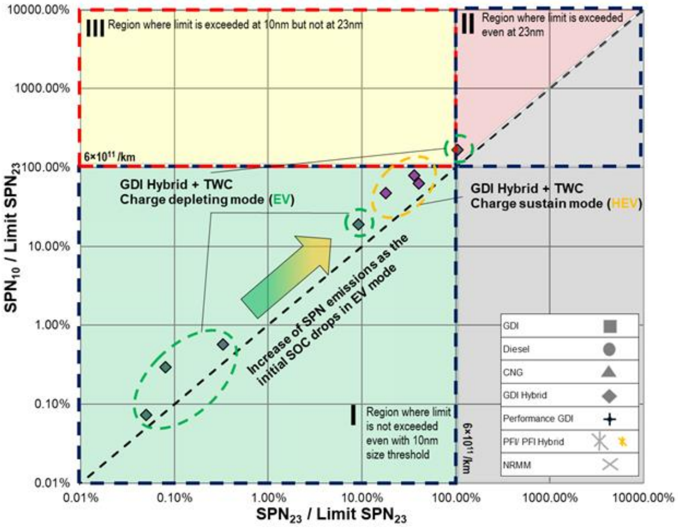

Figure 10 SPN10nm and SPN23nm emissions of PHEV vehicle under different battery modes (Charge depleting and Charge sustain) during WLTC cold measurements

Figure 11 summarizes the SPN particle emissions of the hybrid vehicle. The lower sub-23 nm particle emissions for this technology in the EV mode, as well as the significant increase when the PHEV operates in HEV mode are evident. The big deviation in WLTC hot cycle emissions was due to different initial SOCs from test to test (low, medium and high). In general, current results indicate a specific trend between particle emission performance and PHEVs different battery control modes (HEV or EV). However, future enhanced battery control algorithms, that may include geofencing strategies, may change the overall emission results.

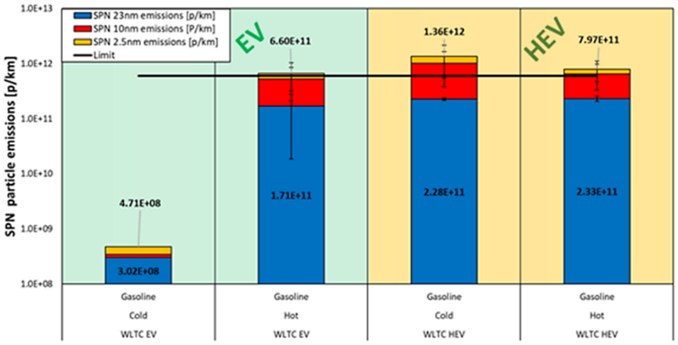

Figure 11 Aggregated average emissions of PHEV for the different battery mode. EV and HEV stands for charge depleting and charge sustain respectively.

## *Total particle emissions*

Although solid particles above or below 23 nm are of high interest for regulatory purposes, total particle number (TPN) emissions are also of interest since they may provide an alternative to the regulated gravimetric PM mass method, which has reached its detection limit in modern vehicles equipped with PFs. In addition, special attention was given to the fresh exhaust aerosol of which vehicles contribute up to 75% to 80% on busy roads[34]. In DTT, total particle number emissions were studied, aiming at evaluating the sampling methodology by implementing different temperature conditions and by removing the CS (Figure 12), as well as by evaluating different engine technologies, fuels and aftertreatment devices (PFs).

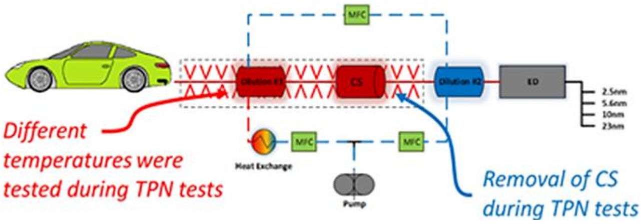

Figure 12 Tailpipe TPN measurement layout. The TPN measurement setup is feasible by removing the CS from the DTT system.

Figure 13 summarizes the TPN over the SPN results great area of interest is the Region II where TPN emissions are more than an order of magnitude higher that SPN respected ones. Recent technologies seem to lead to significant reductions of TPN emissions. Region I in the figure designates the region where TPN emissions are less than an order of magnitude higher than SPN emissions, including CNG, PFI, PHEV in the EV mode, alkylate gasoline and gasoline+MMT. However, special driving events can significantly increase these emissions as can be seen in Region II in the figure, which designates the region where TPN emissions are more than an order of magnitude higher than SPN emissions (CNG with and without PF, PFI with PF and PHEV in the HEV mode). Interestingly, for the hybrid vehicle, both TPN and SPN emissions increase from high to low initial SOC in the EV mode whereas TPN emissions under steady state operation showed very high variability for TPN. Further analysis on TPN sampling conditions showed that an increase of temperature in the 1st dilution stage results to lower TPN emissions, that in some cases were less than an order of magnitude greater than the respected SPN results.

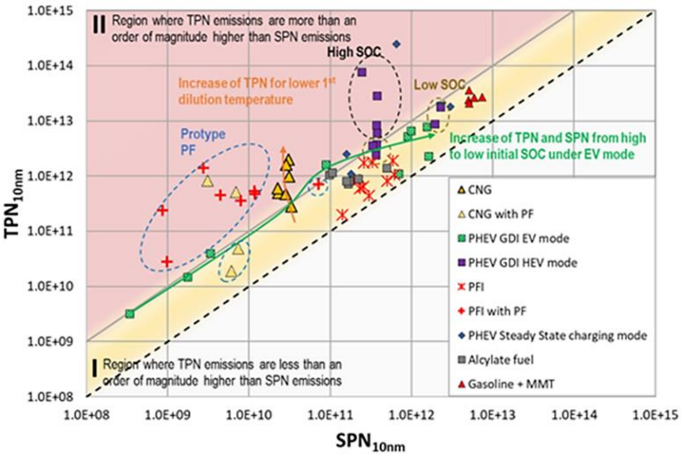

Figure 13 Total particle emission versus Solid ones for the different engine technologies and fuels.

Finally, specific engine events can lead to several times higher TPN emissions. TPN emissions from the hybrid operation have showed a strong relation with engine load (Figure 14). Due to aggressive battery charging at the beginning of the test cycle (100-700s), a higher engine demand is needed. During this period TPN emissions are up to 900 times higher than SPN ones, elevated TPN emissions were also observed from Giechaskiel et.al. but in this case were silicon artifacts due to elastomer connections [1] which are not used in the current work. DTT study on TPN emission give an insight of how TPN and SPN are related and it would be the guideline for further study in that field.

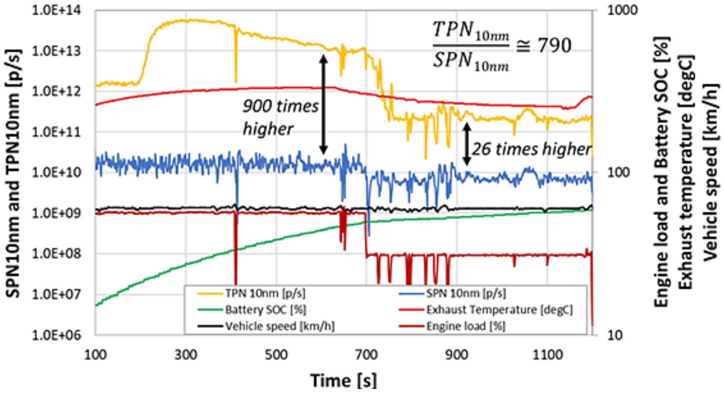

Figure 14 Time series of TPN (yellow) and SPN (blue) emissions during steady state operation of PHEV under charge sustain mode.

# Conclusions

## General

The DTT sampling and measurement system, developed under the H2020 DownToTen project, has been successfully employed to generate a comprehensive dataset on sub-23 nm particle emissions with a wide range of LDVs engines and fuels. Both SPN and TPN emissions have been successfully measured under laboratory operation. The system offers a reasonable penetration efficiency for primary particles and — within normal operation — a reasonably stable DR (100±10). When the CS is in-line, the system is expected to be artefact free. Going down to lower than 10 nm particles increases both the probability of gas-to-particle artefacts and diffusion losses (penetration 40-60%). The method used to assess the SPN emissions of engines and fuels tested graphically, show that emissions lie within one of three regions:

- Region I – the region where emissions of $SPN_{23}$ and $SPN_{10}$ or $SPN_{<10}$ are both compliant with current limit values for PN. This region is of limited interest, but it does show where current technology strengths in PN control lie, and where all technologies' emissions should eventually reside.
- Region II - the region where both $SPN_{23}$ and $SPN_{10}$ or $SPN_{<10}$ emissions are greater than the current limit value. This region is of interest, as it shows where current technology weaknesses in PN control lie.
- Region III - the region where emissions of PN23 are compliant with the current limit value, but emissions of $SPN_{10}$ or $SPN_{<10}$ exceed the limit value. This region is of interest, as it identifies technologies with particle emissions that should be targeted for further development effort to bring results into Region I.

Most technologies fall in Region I, although there are some that fall in Region II (non-DPF diesels and a moped) and in Region III (mopeds, gasoline vehicles without GPF, DPF-equipped diesels after regeneration, CNG). Highest emissions are observed from PFI vehicles that are not currently subject to PN legislation, indicating that adoption of particle filters is likely to be required to meet the current limit for $SPN_{10}$.

## *Specific case with significant number of particles below 23 nm*

- Cold start PN emissions <23nm dominate the overall emissions from SI technologies whereas GDI engines may emit <23nm soot particles under very high load transients. In the case of active DPF regeneration, PN emissions can be dominated by $SPN_{<10}$ for short periods, however, levels are not sufficiently high to exceed $6x10^{11}$part./km even for $SPN_{<10}$.

- Artefacts formed at high exhaust temperatures due to release of material from elastomer connectors could be avoided by using raw measurements, or potentially by adding CVS dilution air directly at the tailpipe upstream of the transfer line. The adoption of CS in sub-23nm particle emission testing should be considered in future regulation, to safeguard against re-nucleation of evaporated species (i.e. sulphates)

- The CNG vehicle emissions are significantly elevated below 10 nm but a simple retrofit of a GPF has been shown to reduce both $SPN_{10}$ and $SPN_{<10}$ to well below the limit value. Measurements from a prototype direct CNG injection technology further improve the <23 nm emission reductions.

- TPN emissions are generally not more that an order of magnitude higher than SPN emissions, although special driving events, such as high accelerations and steady state high vehicle speeds, can significantly increase these emissions

Based on the above observations, potential areas for further development have been identified, including improvements in filtration efficiency of GPFs, development of optimal DPF regeneration strategies that maintain the minimum soot cake for high filtration efficiency, raw vs. dilute sampling, engine/powertrain measures, development of robust calibration procedures for $SPN_{10}$ - with a view to $SPN_{<10}$, gas engine particle emissions, ultra-clean combustion and fuel combinations beyond CNG and chemical/elemental and microscopic analyses of particulate materials from gas engines from various fuels and lubricants aiming to correlate potential precursors with $SPN_{10}$ and $SPN_{<10}$. In addition, the potential areas of future development have been identified, including particle filters for gas engines, ultra-low ash fuels and lubricants, durability of new aftertreatment systems to ash poisoning in the absence of soot emission, measures to avoid and regenerate heavily soot-loaded GPFs and evaluating the need, complexity and challenges of moving from $SPN_{10}$ to $SPN_{<10}$.

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## Definitions/Abbreviations

| | |
|---|---|
| **CNG** | Compressed natural gas |
| **CS** | Catalytic stripper. |
| **DPF** | Diesel particulate filter |
| **GDI** | Gasoline Direct Injection |
| **JRC** | Joint research center |
| **PCRF** | Particle Number Concertation Reduction Factor |
| **PFI** | Port fuel injection |
| **PHEV** | Plug in Hybrid Electric Vehicle |
| **PMP** | Particle Measurement Program |
| **SOC** | State of Charge |
| **SPN** | Solid particle number |
| **TPN** | Total particle number |
| **TWC** | Three way catalyst |
| **VPR** | Volatile particle remover |