Heavy-duty Engine Particulate Emissions: Application of PMP Methodology to measure Particle Number and Particulate Mass

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ABSTRACT
During a test programme on a modern heavy-duty engine, measurements were made at engine-out and tailpipe of particle number and particulate mass using the draft heavy-duty inter-laboratory correlation exercise guide prepared by the UN-ECE Particle Measurement Programme (PMP)\(^1\). In addition to the PMP measurements, the elemental carbon content of the particulate matter from this programme was analysed using thermogravimetric analysis of separate filters.

The particle number measurement system proved to provide a reliable and repeatable measurement procedure. Test results over a variety of operational cycles showed a reduction in particle numbers of some 3 orders of magnitude. Particle number emissions were of similar magnitude regardless of the test cycle. Background-corrected particulate mass emissions results using the partial flow dilution method showed emissions levels below 5mg/kWh over all the transient cycles tested.

INTRODUCTION
Since 1992 when particulate mass (PM) emissions limits were first introduced in Europe for heavy-duty engines, PM emissions and the associated limits have been reduced to less than 10% of the original levels. However the European particulate measurement procedures have been substantially unchanged, and there is a concern about the accuracy of the current measurement procedures at such low emissions rates. Both AECC and Ricardo have been involved with the UN-ECE PMP (Particle Measurement Programme). Therefore additional measurements were made during a project to examine the emissions of a modern medium heavy-duty engine fitted with an emissions control system including a wall flow particulate filter and a urea-SCR system. The additional measurements covered particulate mass according to revised procedures developed in the PMP working group, and particle numbers in accordance with proposed PMP procedures. In order to determine the efficiency of the emissions control system, engine-out emissions of particulate mass and particle number were determined so as to complement the main tailpipe emissions measurement. Supplementary analyses were made to determine the elemental carbon content of the particulate emissions.

METHODOLOGY

TEST ENGINE
The test engine was supplied directly by the manufacturer and was designed to meet US2007 emissions levels with low engine-out NOx. The engine was a turbocharged and aftercooled medium heavy-duty engine with a swept volume of 7.5 litres, equipped with cooled EGR and a high pressure Common Rail fuel injection system. In its US2007 production configuration, it is fitted with a Diesel Particulate Filter (DPF). This was removed and replaced by the AECC system\(^2\) which comprised a diesel oxidation catalyst (DOC), catalysed wall flow DPF and a urea-based Selective Catalytic Reduction (SCR) with an ammonia slip catalyst. The engine calibration was not modified for this programme, and the regeneration strategy for the original-equipment DPF was used without modification.

Standard European low-sulfur (10ppm max.) diesel reference fuel CEC RF-06 was used for all tests. A low-ash 10w-40 engine lubricant specified by the manufacturer was used throughout the programme.

EMISSIONS MEASUREMENT
The test work was conducted on a transient testbed equipped with a full flow CVS system. Emissions were measured after the emissions control system (tailpipe)
and, in most cases, engine-out. Triplicate tests were used for the tailpipe emissions, separate single tests for engine-out. Regulated emissions were measured using certification standard equipment appropriate for each gas concerned. Emissions were measured over a number of different cold-start and hot-start cycles.

Particulate mass:

Three distinct methods of measuring particulate mass were employed during the test work in this programme using both full flow and partial flow dilution approaches. Both full and partial flow dilution methods are now permitted for steady-state and transient cycle type-approvals in Europe. The 3 methods used were:

- **Standard European method** – full flow dilution. This method used 70mm Pallflex TX40 primary and back-up sample filters to collect material from a secondary dilution tunnel. Sample flow rate was ~100l/min.
- **Standard European method** – partial flow dilution. This used a Horiba Micro Dilution Tunnel (MDLT) system with the sample collected on 47mm Pallflex TX40 primary and back-up filters. Sample flow rate was ~50l/min.
- **PMP method** – full flow dilution. This method used a single 47mm Pallflex TX40 sample filter to collect material from the secondary dilution tunnel, simultaneously with the standard method. This system also uses a preclassifier cyclone which provides a cut-point at <10μm. Sample flow rate was ~50l/min.

Dilution factors from the CVS plus secondary dilution system ranged from ~6 (3 primary x 2 secondary) to in excess of 40. Partial flow DFs were generally lower, commencing at ~3.

Figure 1 provides a diagram of the layout used for the three simultaneous sampling systems.

**Particle number:**

In the PMP methodology, the particles measured – 'PMP Solid Particles' are defined by upper and lower limits (d50) particle sizes of approximately 25nm and 2.5μm and by their volatility: they must survive the heating and evaporation processes which removes volatile materials. A cyclone pre-classifier provides a 50% cut-point at 2.5μm whilst a strictly controlled counting efficiency curve for the particle number counter (PNC) sets the nominal lower (d50) limit of 23nm+/-3nm. Measurements were undertaken according to the PMP group's heavy-duty inter-laboratory correlation exercise guide with sampling from the primary CVS dilutions system. The system elements were calibrated by the manufacturer to be in compliance with the developmental procedures of the PMP programme. The system is shown in Figure 2.

**Particulate mass compositional analysis:**

Additional samples were taken from both the full flow and partial flow standard systems onto glass-fibre (GF/A) filters to permit chemical analysis of the collected particulate matter. Thermogravimetric analysis was used to determine the elemental carbon content.

A number of simple checks were conducted on a daily basis to ensure correct operation of the measurement system. These included:

- A zero check of the particle number counter, achieved by placing a HEPA filter on the inlet of the PNC
- A flow check of the particle number counter
- A zero check of the entire measurement system, achieved by placing a HEPA filter between the cyclone and the entrance to the hot diluter
- Close monitoring of system LEDs to ensure correct temperature and flow operation.

These checks were also performed during the light-duty PMP programme and proved sufficient to ensure consistent operation over a substantial period. Typical results of these tests have been published previously. These requirements are now integrated within the draft regulation of particle numbers for light-duty vehicles in Europe.

Additional tests were run in most cases to determine engine-out particle number emissions. The PMP particle number measurement equipment was employed to sample from the partial flow dilution system for these tests. Samples were drawn from the partial flow system above the filter holder but after the dilution tunnel. This required that the additional flow drawn by the mass-flow...
controlled particle number system (~1.5litre/min) was corrected since the draw of additional flow leads to an increase in transfer flow from the raw exhaust into the partial flow system. The MDLT software includes a function to permit an additional flow to be drawn and the mass flow corrected. This function was employed in tests where both particulate mass and particle number tests were sampled simultaneously from the MDLT.

In all cases data were logged throughout on a second-by second basis, the particle number trace time-aligned and the relevant data extracted on a mode-by-mode basis for steady states such as the ESC or averaged across the cycle for transient tests. Particle number data were drawn from the CVS (tailpipe measurements) and from the MDLT (engine-out measurements).

**Preconditioning and test regime:**

Previous experience of particulate number measurements for DPF-equipped light-duty vehicles suggested that the sensitivity of the method resulted in some variation in emissions results depending on the fill state of the DPF. To minimise these differences, specific preconditioning procedures, within the relevant legislative test requirements, were undertaken to ensure that the results were as consistent as possible. Pre-conditioning was carried out a minimum of 12 hours before the emissions tests. The schedule consisted of 15 minutes warm-up at ESC Mode 4 (2130 rev/min, 560 Nm), followed by 60 minutes at maximum power (2575 rev/min, 700 Nm) giving a temperature at the DPF inlet of 520~540°C, and then low temperature (>250°C) operation at 1300 rev/min, 150 Nm for 60 minutes.

Each day’s test regime started with a cold-start test cycle such as the World-Harmonised Transient Cycle (WHTC) followed by the hot-start version of the cycle after the relevant soak time and then other hot-start cycles such as the current European Transient and Steady State cycles (ETC and ESC respectively). Before each of the latter cycles the engine was operated at ESC Mode 4 (2130 rev/min, 530 Nm (=75% load)) for 7.5 minutes to provide a consistent pre-conditioning for all hot-start cycles. For the WHSC test only, the engine was operated at WHSC Mode 9 (1816 rev/min, 373 Nm) for 10 minutes, followed by 5 min soak with the engine at rest, before starting up for the test. In the case of the WHTC, hot-start tests were run after soak times of both 5 and 20 minutes, which are the two options permitted in the Global Technical Regulation (gtr), and after a compromise time of 10 minutes.

**TEST RESULTS**

**PARTICLE NUMBER**

**Transient cycles:**

Figure 3 shows tailpipe particle number emissions from European (ETC), US (FTP) and Japanese (JE05) regulatory cycles, the World Harmonised Transient Cycle (WHTC) and the Non-road Transient Cycle (NRTC). For those cycles with cold- and hot-start portions, results are shown for the cold- and hot-start cycles separately. In addition results are shown for the hot-start WHTC with the 5, 10 and 20 min. soak periods. All tailpipe emissions measurements are the average of at least three tests and the error bars shown are ±2 standard deviations.

Both the ETC and WHTC cycles gave mean tailpipe emissions levels of ~4 x 10^{11}/kWh, while the JE05 and FTP levels were directionally higher at ~7 x 10^{11} and >8 x 10^{11}/kWh respectively, but still of the same order of magnitude. The WHTC soak periods of 5, 10 or 20 minutes duration had no significant impact on particle number emissions. The highest particle number emitting cycles tended to be those with the lowest mean cycle power (FTP ~ 12kW and JE05 ~11.5kW). Since the ETC and WHTC are higher mean power cycles (~24.5kW and ~17kW respectively), this suggests that the post-DPF particle number concentrations are similar from all cycles.

These results should be compared with the engine-out emissions shown in Figure 4. (Engine-out particle number emissions tests were not run for the JE05 cycle or the 10- and 20-minute soak hot WHTC tests). Engine-out particle number emissions were measured over single tests.
Engine-out ETC, WHTC and FTP emissions levels were around $4 \times 10^{14}$/kWh. These levels are consistent with total particle number emissions emitted by a Euro I engine using ultra-low sulfur diesel fuel and at a mass emission of about 0.18g/kWh. Emissions over the NRTC were of the same order of magnitude, but closer to $5 \times 10^{14}$/kWh, suggesting that engine-out emissions from this cycle may be marginally higher than from the other transient cycles. Engine-out particle number emissions tended to be slightly higher from cold-start than from hot-start cycles, but all these differences may be within test-to-test repeatability. The results indicate that the emissions control system provided a reduction of some 3 orders of magnitude for each of the cycles.

**Transient cycle particle production:**

The WHTC cycle was studied in most depth during the test programme. Each WHTC test comprised a cold-start phase lasting 1800s, a soak period lasting either 5 minutes (300s), 10 minutes (600s) or 20 minutes (1200s) followed by a second, hot-start, phase. Excepting the cold start, the hot phase was identical to the cold test. Particle production profiles from typical cold-start WHTC cycles are shown in Figure 5. Note that the particle number scale is 3 orders of magnitude lower in Figure 5a than in Figure 5b.

![Figure 5a: Continuous tailpipe particle number traces for the WHTC cold-start test.](image)

Engine-out particle emissions (lower chart) track the torque profile closely. Particle emissions after the emissions control system (Fig. 5a) are some 3 orders of magnitude lower and can also be seen to track with engine torque. The substantially reduced emissions are both smoothed and slightly offset by their passage through the DPF and changes are most significant when large changes in torque occur. The same particle number measurement system was used for both engine out and post-DPF measurements but drawing from the MDLT and CVS respectively. So, any sample offset difference will come from the transit time from exhaust sample point to the MDLT, the same point through the exhaust to the CVS sampling point and from a physical lag through the DPF and other exhaust componentry. Calculations show the CVS transit time was ~1s and the MDLT time ~0.75s. Residence time in PMP equipment is ~2s. The close similarity between these, and prior experience with other DPFs which have shown no lag, suggests that in this case the lag is a real DPF effect.

**Stationary cycles:**

The steady-state cycles examined were the European and World-Harmonised steady-state cycles (ESC and WHSC) and the Non-road steady-state cycle (NRSC). Tailpipe particle number emissions from the steady-state cycles are shown in Figure 6 and engine-out emissions in Figure 7 (engine-out emissions were not measured on the NRSC). Emissions from the ESC and NRSC (~$8 \times 10^{11}$/kWh and $1.2 \times 10^{12}$/kWh respectively) were directionally somewhat higher than from the ETC and WHTC (~$4 \times 10^{11}$/kWh), while emissions from the WHSC were directionally lower than ETC/WHTC levels. It is possible that the ESC and NRSC show higher particle number emissions (despite high power levels of ~92kWh and ~98kWh respectively) than the lower-power ETC and WHTC for two reasons:

- higher exhaust temperatures may lead to passive regeneration of the DPF and/or
- high exhaust temperatures lead to the thermal release of low volatility materials seen by the PMP equipment as solid particles.

Table 1 shows selected exhaust system temperatures for the three stationary cycles:

<table>
<thead>
<tr>
<th></th>
<th>ESC</th>
<th>NRSC</th>
<th>WHSC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Turbine-out</td>
<td>395</td>
<td>407</td>
<td>301</td>
</tr>
<tr>
<td>SCR-out</td>
<td>385</td>
<td>383</td>
<td>286</td>
</tr>
<tr>
<td>Tailpipe</td>
<td>362</td>
<td>367</td>
<td>264</td>
</tr>
</tbody>
</table>

Table 1: Exhaust temperatures, stationary cycles.
Figure 6: Tailpipe particle numbers: steady-state tests.

Figure 7: Engine-out particle numbers: steady-state tests.

The engine-out and tailpipe results for these tests are summarized in Table 2. The repeatability levels of this engine and DPF were consistent with those of several efficient wall-flow DPF equipped light-duty vehicles tested with similar equipment previously.

<table>
<thead>
<tr>
<th>Cycle</th>
<th>Tailpipe Average</th>
<th>2*STDEV</th>
<th>CoV</th>
<th>Engine-out Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>ETC</td>
<td>3.85E+11</td>
<td>2.40E+11</td>
<td>31.2%</td>
<td>4.38E+14</td>
</tr>
<tr>
<td>WHTC_cold</td>
<td>3.74E+11</td>
<td>2.39E+11</td>
<td>31.9%</td>
<td>3.30E+14</td>
</tr>
<tr>
<td>WHTC_hot[5]</td>
<td>4.94E+11</td>
<td>2.24E+11</td>
<td>22.7%</td>
<td>3.96E+14</td>
</tr>
<tr>
<td>WHTC_hot [10]</td>
<td>3.80E+11</td>
<td>2.04E+11</td>
<td>26.9%</td>
<td></td>
</tr>
<tr>
<td>WHTC_hot [20]</td>
<td>4.04E+11</td>
<td>2.80E+11</td>
<td>34.6%</td>
<td></td>
</tr>
<tr>
<td>FTP_cold</td>
<td>8.60E+11</td>
<td>4.66E+11</td>
<td>27.1%</td>
<td>3.28E+14</td>
</tr>
<tr>
<td>FTP_hot [20]</td>
<td>8.45E+11</td>
<td>2.20E+11</td>
<td>13.0%</td>
<td>4.45E+14</td>
</tr>
<tr>
<td>NRTC_cold</td>
<td>5.86E+11</td>
<td>2.72E+11</td>
<td>23.2%</td>
<td>4.66E+14</td>
</tr>
<tr>
<td>JE05</td>
<td>7.08E+11</td>
<td>5.20E+11</td>
<td>36.7%</td>
<td></td>
</tr>
<tr>
<td>ESC</td>
<td>7.35E+11</td>
<td>1.79E+11</td>
<td>12.1%</td>
<td>3.32E+14</td>
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<tr>
<td>WHSC</td>
<td>2.25E+11</td>
<td>6.87E+10</td>
<td>15.3%</td>
<td>2.39E+14</td>
</tr>
<tr>
<td>NRSC</td>
<td>1.24E+12</td>
<td>5.82E+11</td>
<td>23.5%</td>
<td></td>
</tr>
</tbody>
</table>

Table 2: Engine-out and tailpipe particle number results (particles/kWh).

PARTICULATE MASS

In general the tailpipe particulate emissions were low for all cycles, and for all of the three measurement methods used. However the measurements from the partial flow MDLT system were more consistent, resulting in smaller error bars, than for the other methods. The tailpipe PM results produced by the three methods are compared for the transient test cycles in Figure 8. Note that for all three methods the error bars intersect with the zero line for all the test cycles and the measurement methods. This indicates that the tailpipe particulates were very low over a wide range of engine operating conditions. Based on the results from the MDLT the removal efficiency for PM was >99%.

The discrepancy between the tailpipe emissions levels recorded by the partial flow system and the high levels recorded by both of the full flow methods was investigated in some detail. Background filter papers taken both before and after tests showed high background levels and sample and background masses were found to be equivalent, again suggesting that tailpipe emissions were extremely low. The investigatory work established that tailpipe elemental carbon (EC) levels from all the PM methods used were close to the detection limit and similar to the blank filter paper. Chromatographic analysis of full flow filter papers gave almost identical hydrocarbon profiles at levels well above the unused blank filter paper (Figure 9). PM filter blanks drawn from the partial flow system were indistinguishable from a used blank filter.

These HC profiles did not, however, appear to reflect the fuel or oil used in the engine. A background sample was taken from the primary tunnel and in this case the high background was eliminated, indicating that the background contribution arises after the primary CVS dilution system. As the dilution air for both the primary and secondary tunnels was HEPA filtered, in line with the recommendations of the PMP protocol, the secondary dilution system was further investigated, including using the MDLT system as the secondary diluter.
The source of contamination was traced to the ‘make-up’ air pump that supplied additional dilution air to the secondary tunnel. This was necessary to permit the two full flow methods to be sampled simultaneously and to control secondary tunnel temperature by supplying hot air to the secondary tunnel. In this case the pump was pushed to the limits of its capability and some of the seals perished. This then permitted lubricating oil from the pump to volatilise and be carried by the air into the secondary dilution system. This pump is situated downstream of the dilution air HEPA filter, so the HEPA filter was unable to capture any contaminants. The problem thus resulted from the decision to take parallel samples to provide a direct comparison of the current European and PMP full flow methods and should not normally present a problem in routine or certification measurement.

Subtraction of background air contribution to PM mass is permitted in the ECE Regulation 49, though, so it is legitimate to subtract a mean tunnel background for the full flow results in this programme. The revisions to UN regulations proposed in association with the PMP programme for light-duty measurements specifically permit tunnel background subtraction in some circumstances.

Although the engine-out particulate emissions were relatively high, due to the high rates of EGR used to control NOx, the tailpipe PM mass emissions were very low. Consequently, the PM conversion efficiency calculated using the MDLT results was 99.6–99.9% for all tests, except the ESC tests, for which the result was 94.3%; possibly due to a reduction in DPF filtration efficiency during and after passive regeneration at mode 10 of the ESC and the additional capture of low-volatility HCs emitted at Mode 10 on the tailpipe PM filter.

Figure 10 shows the PM emissions and efficiency for the ETC and WHTC cycles. Average tailpipe emissions were 1mg/kWh on the ETC and 2 mg/kWh on the weighted WHTC (10% cold weighting, 5 minute soak period, as proposed for European application of the WHTC).

**ELEMENTAL CARBON**

Elemental carbon (EC) analyses were performed on the glass-fibre filters drawn from the post-catalysts (tailpipe) sampling point on all cycles and from engine-out PM samples collected from selected cycles. All data were corrected for a system blank, on which a carbon background of less than 1mg/kWh (equivalent) was determined. The highest post-catalysts soot levels observed were 5mg/kWh from the ESC cycle, though in general levels were below 3.5mg/kWh. Engine-out emissions of elemental carbon covered a wide range from 0.1g/kWh over the WHSC cycle to >1g/kWh over the hot FTP cycle. The base engine’s calibration had high rates of EGR to control NOx, and it is probable that, under transient conditions, air-fuel ratios were low, leading to high levels of engine-out elemental carbon.

Filtration efficiencies for elemental carbon were typically 99% or greater for all transient cycles and averaged 99.14%. The ESC result was slightly lower than the average at ~95%. As with the PM results this may be due to passive regeneration at mode 10. However it should be noted that post-DFP masses were very low, elemental carbon levels even lower and even a small background contribution of EC (which is corrected in the analyses) could be responsible for this difference. The filtration efficiencies for elemental carbon are shown in Figure 11.
SUMMARY

The PMP procedures for measurement of particulate mass and particle number emissions were applied to a modern medium heavy-duty engine. The PMP particle number method was sufficiently sensitive and reliable even at near-ambient particle emissions levels, but some difficulties were experienced in the measurement of very low particulate mass levels. These were traced to contamination resulting from the decision to take parallel samples to provide a direct comparison of the current European and PMP full flow methods and should not normally present a problem in normal measurement.

Engine-out particle number data was in the range of 2.5 to 5 x 10^{14}/kWh. All transient cycles data showed tailpipe particle number emissions below 10^{12}/kWh and the range of particle numbers was well within an order of magnitude. Background-corrected PM from the PMP method gave results below 5mg/kWh and PM conversion efficiencies were >99.5% over the ETC and EU-composite WHTC, resulting in PM tailpipe levels of 1 to 2mg/kWh when measured with the partial flow method. The emissions control system reduced elemental carbon emissions by more than 99% over the cycles tested.

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REFERENCES

7. PNC: Particle Number Counter Calibration Procedure, Report to the Department for Transport; ED47382004/PNC, AEA Technology First draft 2006
11. gtr No. 4 - Test procedure for compression-ignition (C.I.) engines and positive-ignition (P.I.) engines fuelled with natural gas (NG) or liquefied petroleum gas (LPG) with regard to the emission of pollutants, United Nations, 15 November 2006.

DEFINITIONS, ACRONYMS, ABBREVIATIONS

CoV: Coefficient of Variation (ratio of standard deviation to the mean)
CVS: Constant Volume Sampler
DOC: Diesel Oxidation Catalyst
DPF: Diesel Particulate Filter
EC: Elemental Carbon
EGR: Exhaust Gas Recirculation
ESC: European Steady-state Cycle
ETC: European Transient Cycle
FTP: (US) Federal Test Procedure
grt: (UN) global technical regulation
HEPA: High Efficiency Particulate Air (filter)
MDLT: Micro-Dilution Tunnel (partial flow)
NRSC: Non-Road Steady-state Cycle
NRTC: Non-Road Transient Cycle
PM: Particulate Mass
PMP: United Nations Economic Commission for Europe (UN-ECE) Particle Measurement Programme
PNC: Particle Number Counter
SCR: Selective Catalytic Reduction
STDEV: Standard Deviation
WHSC: World Harmonised Steady-state Cycle
WHTC: World Harmonised Transient Cycle