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# Particle Measurement Programme Heavy Duty Inter-Laboratory Correlation Exercise: Final Report

# Submitted by the expert from the United Kingdom \*

The text reproduced below was prepared by the expert from the United Kingdom on behalf of the Working Group on the Particle Measurement Programme (PMP). It is submitted to the Working Party on Pollution and Energy (GRPE), for consideration at its June 2010 session.

In accordance with the programme of work of the Inland Transport Committee for 2006–2010 (ECE/TRANS/166/Add.1, programme activity 02.4), the World Forum will develop, harmonize and update Regulations in order to enhance the performance of vehicles. The present document is submitted in conformity with that mandate.



## **Executive Summary**

The Heavy Duty Inter-Laboratory Correlation Exercise has conducted testing at 5 test laboratories in the Europe in order to demonstrate the practicality, robustness, repeatability and reproducibility of the particle emissions measurement techniques proposed by the Particle Measurement Programme (PMP). The exercise involved testing a golden engine (a Euro III Iveco Cursor 8, equipped with a wall-flow Diesel Particulate Filter), at all participating laboratories to allow the inter-laboratory reproducibility of measurements to be assessed. Each laboratory tested the engine over multiple repeats of the heavy duty World Harmonized Transient Cycle (both cold and hot start) and World Harmonized Steady state Cycle (WHSC) as well as current European Union (EU) regulatory cycles the European Transient Cycle (ETC) and European Steady state Cycle (ESC). Measurements of solid particle number emissions, particulate mass and regulated gaseous emissions were taken over each test. Particle emissions measurements were taken from both full flow (CVS) and partial flow (PFDS) dilution systems at each laboratory. Two 'golden' particle number measurement systems were circulated between the test laboratories, one for use in CVS measurements one for use in PFDS measurements. In addition laboratories made particle number measurements using several alternatives, PMP type systems to compare the performance of different measurement systems. The golden measurement systems performed reliably at all laboratories and agreed with one another to within 5 per cent when making measurements in parallel.

Particulate Mass (PM) emissions levels from the golden engine using CVS sampling systems were below 6 mg/kWh across all test cycles after exclusion of outlying test results. High tunnel background contributions in some laboratories' CVS systems resulted in higher outlying results. PFDS systems returned slightly lower PM results (below 4 mg/kWh). In this exercise tunnel background PM measurements were generally found to be similar to engine measurements, although ESC cycle results sampled from PFDS systems could be discriminated from tunnel background levels by all laboratories. PM measurement repeatability from PFDS was 20-30 per cent for all test cycles and rather higher, 35-56 per cent, for CVS measurements after exclusion of outliers. Reproducibility between laboratories was 35-45 per cent for PFDS measurements and 35-55 per cent for CVS measurements.

Particle Number (PN) emissions levels from the golden engine varied significantly from cycle to cycle. The cold start World Harmonized Transient Cycle (WHTC) gave the highest PN levels, approximately  $4x10^{11}$ /kWh from both CVS and PFDS dilution systems. At these levels tunnel background PN concentrations did not significantly influence measurements. Hot start WHTC and ETC cycles gave PN levels around 5-9 x 10<sup>9</sup>/kWh, steady state cycles gave higher results (2-3 x  $10^{10}$ /kWh on the WHSC and 6-8 x  $10^{10}$ /kWh on the ESC) possibly due to higher exhaust temperatures resulting in some passive regeneration and reduction in filtration efficiency as the soot cake on the Diesel Particulate Filter is reduced. On these test cycles tunnel background levels were found to have a significant impact in the case of some laboratories' CVS systems, PFDS tunnel background levels however were significantly lower and did not influence PN results. Where tunnel background concentrations were low, correlation between CVS and PFDS measurements was excellent.

PN repeatability levels across the different test cycles ranged from 20-60 per cent for CVS sampling, with best repeatability being on the cold WHTC (where PN levels were highest), and worst on the WHSC, where partial passive regeneration of the Diesel Particulate Filter may result in less stable PN emissions from the engine. PFDS repeatability ranged from 20-70 per cent, with best and worst results again on cold WHTC and WHSC respectively, however, as noted above, on the lower emissions cycles PFDS measurements were less

influenced by tunnel background levels than was the case for CVS measurements. PN reproducibility between laboratories was generally similar to repeatability. Across the different test cycles PN reproducibility ranged from 30-80 per cent for CVS sampling and 50-86 per cent for PFDS sampling.

Results from alternative PN measurement systems conforming to PMP principles generally correlated to the golden system measurements within 15 per cent although some systems showed greater offsets. Additional experiments conducted during the exercise for investigative purposes showed that, contrary to results from some US research, reported concentrations of solid particles of less than 23 nm diameter were low relative to those of larger than 23 nm particles. This confirms the suitability of the 23 nm lower size cut-off for the particle number counter. Although further investigation across a broader range of engines and Diesel Particulate Filters may be of value.

The results of this exercise demonstrate that the PM measurement method is suitable to confirm that engine emissions are below 10 mg/kWh. PM measurements of an engine equipped with an efficient wall-flow Diesel Particulate Filter were similar across all test cycles. In this exercise PM measurements could not generally be discriminated from tunnel background PM measurements. The PN emissions measurement method was able to discriminate between the emissions levels on different test cycles of an engine equipped with an efficient wall-flow Diesel Particulate Filter. PN was also able to discriminate engine emissions from tunnel background levels in this exercise except in the case of high tunnel background sampling systems during testing on cycles with lower emissions levels.

# Particle Measurement Programme Heavy Duty Inter-Laboratory Correlation Exercise: Final Report

## I. Introduction

1. The effect of exhaust emissions from road vehicles on public health has long been a concern. Legislation limiting the pollutant emissions of new vehicles is well established in many regions of the world. One emission of special concern is particulate matter. In vehicle exhaust this consists of tiny solid particles and liquid droplets ranging in size from a few nanometres to up to around one micrometre in diameter. Current legislative emissions standards regulate particle emissions in terms of the total mass of particulate matter emitted per kilowatt hour. This is effective at controlling emissions of larger size particles, but particles at the smaller end of the size range contribute little to the total mass of particulate matter emitted.

2. There is a growing consensus amongst health experts that particles in the ultrafine (< 100 nm diameter) size range may be those which are having the greatest adverse effect on human health. The main driver behind Particle Measurement Programme (PMP) is the impact of particles on human health. The PMP has no medical expertise and does not seek to pre-judge the advice that may emerge from medical experts with respect to the most crucial particle characteristics affecting human health. Nonetheless, current medical opinion suggests that reductions in particle emissions will lead to improved air quality and health and the PMP has therefore moved forward on the basis of the precautionary principle. This and the potential limitations of current regulatory procedures at forcing technology that would control these particle emissions led to the setting up of the PMP as an informal working group of the UNECE Working Party on Pollution and Energy (GRPE). PMP is essentially a collaborative programme of Government sponsored research projects. However, the informal group, chaired by the United Kingdom, exists to co-ordinate the research and ensures that the programme is open to contributions from a wider audience. National Governments, individual laboratories, exhaust after treatment, automotive industry and fuel industry representatives have all provided significant input to the programme.

3. The mandate given by GRPE to the PMP working group was to develop new particle measurement techniques to complement or replace the existing particulate mass measurement, with special consideration to measuring particle emissions at very low levels. These techniques should include a detailed specification of test procedures and equipment, be suitable for light duty vehicle and heavy duty engine type approval testing and be suitable for use in transient testing. Since, within the European Union (EU), type approval tests which could take place at one of many laboratories, good repeatability and reproducibility from laboratory-to-laboratory are key requirements for regulatory measurement techniques. PMP has therefore sought to demonstrate the repeatability and reproducibility of the proposed techniques. PMP was also tasked with accumulating data on the performance of a range of engine/vehicle technologies when tested according to the proposed procedures.

# II. Nature and scope of the Heavy Duty Inter-Laboratory Correlation Exercise

## A. Background to the PMP

4. In 2001, the French, German, Netherlands, Swedish and United Kingdom Governments agreed to a collaborative programme aimed at developing new methods and procedures to facilitate the control of ultrafine particles within a regulatory framework. This programme was designed to deliver a regulatory procedure that would either replace or complement the existing procedure used for particulate mass measurement. The resulting Particle Measurement Programme (PMP) working group, chaired by the United Kingdom's Department for Transport, operates under the auspices of the UNECE, where the government of Switzerland joined the consortium. Japanese and Korean governments have also contributed.

5. The PMP working group devised a three-phased approach to the PMP Programme. In the first two phases of the programme, a wide range of measurement instruments and sampling systems were assessed over standard regulatory tests.

6. In the PMP Phase 1 study, measurement systems addressing several key particle properties including mass, number, active surface and chemistry were evaluated along with appropriate dilution methods, sample conditioning and consideration of cost and logistical aspects.

7. Phase 2 subjected the best performing systems from Phase 1 to more rigorous evaluations. Aims were to confirm the results of Phase 1 and determine fundamental levels of repeatability within a single laboratory during a variety of steady state and transient tests with both engine-out and post-DPF (i.e. Diesel Particulate Filter) exhaust aerosols. The testing from Phase 2 enabled the conclusions that a revised filter mass measurement method and a particle number method, both based upon sampling from a standard dilution system, best met the original objectives of the programme. The two recommended systems were:

- (a) A filter method based broadly upon those currently used in Europe and the United States (US) and that proposed for the US for 2007 type approvals;
- (b) A particle number method using a Particle Counter (PC), a selected size range and sample pre-conditioning to eliminate volatile particles.

8. Draft revised versions of the light duty vehicle (Regulation No. 83  $[^1]$ ) and heavy duty engine (Draft Regulation No. 49  $[^2]$ ) particulate regulatory sampling annexes were prepared from the existing regulatory documents: Regulation No. 83  $[^3]$  and Regulation No. 49  $[^4]$ .

9. The new documents integrated the PMP particulate and particle number approaches into the existing regulatory framework and also formed the bases for two test protocol documents written as laboratory guides for testing. The PMP Phase 3 "Inter-Laboratory Correlation Exercises" (ILCE) for light duty vehicles (ILCE\_LD) and heavy duty engines (ILCE HD) then commenced with the light duty vehicles' exercise.

## B. PMP Phase 3 – Inter-laboratory exercise for light duty vehicles

10. The light duty vehicles' exercise circulated an Euro 4 light duty diesel vehicle equipped with an OEM fit Diesel Particulate Filter (DPF) plus a reference "Golden" Particle Measurement System (GPMS) between laboratories. In addition, each lab was invited to employ other particle measurement systems constructed to meet the design criteria of the GPMS, and to test other Euro 4 vehicles. Testing followed the procedures described in the inter-laboratory guide for light duty vehicles (ILG LD) [<sup>5</sup>] and comprised the measurement

of regulated gaseous emissions, particulate mass and particle number from repeat NEDC tests. To ensure maximum consistency of testing between laboratories, the golden engineer and project manager visited the participating laboratories to advise on facility modifications, how to undertake the test protocols and installation and operation of the GPMS. Low sulphur fuel and lubricant from the same batches were also used at all laboratories. The ILCE\_LD has now completed, with the final report published in June 2007 [<sup>6</sup>] and extended data analyses published in the scientific literature [<sup>7</sup>], [<sup>8</sup>].

- 11. The general conclusions of the ILCE LD are presented below:
  - (a) The revised PMP mass method provides repeatable measurements at well below 2.5 mg/km, but the method collects a large gaseous volatile fraction that may be 20 times the mass of the solid particles collected.
  - (b) Both mass and number measurement approaches appear to have detection limits low enough to discriminate between a highly efficient wall-flow DPF equipped diesel and non-DPF equipped diesel vehicles. In this testing, the mass method proved unable to discriminate a porous wall-flow DPF from a more efficient one.
  - (c) The PMP Particle Number method proved to be less variable than the PMP mass method for Euro-4 non-DPF diesel cars, with repeatability levels from 6 vehicles at  $\geq$  5 per cent.
  - (d) Comparing the lowest emissions of the non-DPF diesels and the highest emissions of the efficient wall-flow DPF equipped diesels, the number method showed a difference of > 300 times and the mass method a difference of  $\sim 18$  times. This can be expressed as a difference in discriminating power approximately 20 times greater for the number method than for the mass method.
  - (e) Mass and number measurement equipment presented no significant functional challenges during the 2 year programme. Minor maintenance issues did occur but these were dealt with as normal service issues.
  - (f) The PMP number method presents improvements over the PMP mass method in terms of limit of detection, accuracy, discrimination power and variability when measuring a stable particle source. For these reasons, the number method is a superior alternative to the existing or a revised mass method for future regulatory procedures.

12. The conclusions of the final report, and consultations with stakeholder groups including national Governments, the European Commission, the automotive industry, Tier 1 suppliers and the test houses were used to finalise a new annex for Regulation No. 83 which introduced the particle number procedure for certification testing. Modifications to the particulate mass measurement procedure were also integrated. The new procedures came into force with the official publication of the procedures during February 2009 [<sup>9</sup>].

# C. Brief overview of the inter-laboratory correlation exercises for heavy duty engines

13. Following the successful completion of the ILCE\_LD, the PMP working group determined the scope of the heavy duty exercise. This essentially comprises three parts:

(a) Investigative work to develop and finalise a robust inter-laboratory guide for heavy duty engines testing. Experiments included identifying background PM and PN levels, effects of different filter media, impacts of filter face velocity changes, exhaust and engine preconditioning effects, comparisons of different particle number systems and investigation of the golden instruments. A full report of the experimental work has been published previously  $[1^{10}] [1^{11}]$  and an overview is given in Chapter II, Section K. The final inter-laboratory guide  $[1^{12}]$  is included in this report as Appendix 1.

- The validation exercise: analogous to the ILCE LD, this programme (b) investigated particle number repeatability and reproducibility by transporting a golden engine to each test laboratory in turn. Along with the engine, two Golden Particle Measurement Systems (GPMS) were shipped to permit particle number measurements to be made simultaneously from both full flow Constant Volume Sampler (CVS) dilution and Partial Flow Dilution (PFDS) systems. As in the ILCE LD, participating laboratories were also invited to test their own particle measurement systems, or other commercially available particle numbers systems. PM and gaseous emissions were also measured. The golden engineer and the project manager ensured that participating laboratories correctly followed the measurement protocols defined in the inter-laboratory guide. Low sulphur fuel and lubricant from the same batches were used at all laboratories. The participating laboratories were JRC (the Joint Research Centre in Ispra, Italy), AVL-MTC (the motor test centre in Sweden), Ricardo (United Kingdom), UTAC (the Union Technique de l'Automobile du motocycle et du Cycle in France), and EMPA (Eidgenössische Materialprüfungs- und Forschungsanstalt in Switzerland). Each test laboratory was funded by its respective national government. JRC undertook duplicate measurement campaigns - at the start and end of testing to monitor consistency of emissions through the programme. Testing in the validation exercise completed at JRC in October 2009.
- (c) The round robin exercise: adhering to the principles of an automotive industry round-robin exercise, this programme is complementary to the validation exercise, but subtly different. Its objective is purely the evaluation of particle number repeatability and reproducibility using different measurement systems. In the round robin, a reference engine is circulated, but each lab uses its own particle number systems from full or partial flow dilution tunnels. All laboratories will use fuel and lubricant of the same types (but not necessarily from the same batches). PM and regulated gaseous emissions will also be measured. Laboratories from the EU, Japan, Korea and Canada are all participating in the programme. While testing in the round-robin exercise has completed at several laboratories, work is on-going, with completion anticipated during 2011.

14. This report describes the procedures, results and conclusions of the PMP Phase 3 validation exercise in detail. Once it has completed, the round-robin exercise will be reported separately.

#### D. Test engine and emissions control system

15. The engine employed in the test programme was a series production IVECO Cursor 8 engine in Euro III specification; a modern 7.8 litre, 6-cylinder engine used in heavy duty vehicle and bus applications. Further details can be found in Table 1.

TVECO Cursor 8 (Euro III)			
Details	7.8 litre, 6 cylinder, 4 valves/cylinder		
Compression ratio	17:1		
Maximum power	295 kW @ 1900 to 2400 rpm		
Maximum torque	1280 Nm @ 1000 to 1900 rpm		
After-treatment	Continuous Regenerating Trap (CRT)		
Oxicat	Pt-based: 10.5x3" catalyst section; approximately 4.25 litres		
DPF	Wall-flow DPF: 11.25x14"; approx 24 litres		

#### Table 1 Test engine and specification

16. Figure 1 (below left) illustrates a typical installation of the engine, in this case the first test laboratory, JRC. The right-hand part of Figure 1 illustrates the exhaust system layout at JRC which was used to devise a set of benchmark dimensions for installations at all the other test laboratories.

17. A guide to installation and commissioning was supplied with the test engine and a support engineer visited each test laboratory to facilitate these processes. After testing at JRC this guide was updated to include the exhaust system layout and sampling positions for the PFDS, raw gas analysers and temperature and pressure sensors. A schematic representation of the engine and exhaust layout is given in Figure 2.

#### Figure 1

Typical engine and emissions control system installations



18. A degree of variability, due to the constraints of test cell size and orientation, was expected during engine, exhaust system and PM/PN measurement system installations. Differences are summarised in Table 2. Generally, differences between laboratories were small and are not expected to have impacted results.



## Figure 2 Schematic of exhaust and emissions control system layout

Numbers in Figure 2 indicate component lengths (and diameters in parentheses) in cm. Table 2

Sampling Dimensions Length in cm diameter (cm)	AVL-MTC	JRC	Ricardo	UTAC	EMPA
Engine-CRT	250 (15)	270 (15)	165 (15)	100 (15)	299 (10)
CRT-PFDS	700 (15)	500 (15)	395 (15)	350 (15)	934 (12.5)
CRT-CVS	1100 (15)	950 (15)	930 (15)	750 (15)	1469 (12.5)
CRT-CVS Insulated	1100 (15)	600 (15)	200 (15)	450 (15)	1045 (12.5)
PFDS-SPCS20	150	150	400	150	320
CVS sampling point - CVS mixing point	500 (50)	470 (47)	500 (45)	575 (45)	470
CVS-SPCS19	400	400	360*	400	202

Exhaust system installation variability at the test laboratories

Ricardo used a heated line at 47°C to extend the 1m Horiba sampling line to ~4m.

## E. CRT: Pt-based oxidation catalyst and wall-flow DPF

19. The DPF employed in the test programme was a cordierite wall flow filter of approximately 24 litres volume and originally supplied by Johnson-Matthey. The ratio of DPF volume to engine size is therefore approximately 3, which is larger than the 1.5 to 2.5 typically employed in current HD applications. The DPF is preceded in the exhaust system by a close-canned Pt-based oxidation catalyst (Eminox) of approximately 4.25 litres volume.

## F. Fuel and lubricant

20. Fuel and lubricant were supplied to the PMP programme by members of the Oil Companies European Organization for Environment, Health and Safety (CONCAWE). The test fuel was provided by energy group Total, who isolated a large batch of the certification reference fuel RF06-03 and nominated it RF06-03-PMP. Participating laboratories purchased quantities of this batch directly from the supplier. This fuel fully complies with Annexes III and IV of Directive 2003/17/EC describing fuel specifications to be employed after 1 January 2009 (i.e. sulphur content of < 10 ppm). Selected properties are given in Table 2 and the detailed specifications can be found on the final page of Appendix 1.

#### Table 3 Fuel specifications

Properties	Units	Value
Cetane number	[-]	53.1
Density	[kg/m <sup>3</sup> ]	834.9
Sulphur	[ppm] or [mg/kg]	7
Polycyclic aromatics	[per cent] by mass	5.1

21. The test lubricant (Table 4: Lubricant Properties) was a BP Vanellus E8 fully synthetic, 5W/30 PAO (polyalphaolefin) based oil with < 0.2 per cent sulphur content. Defined oil change and conditioning procedures were employed at each laboratory to standardise oil conditioning and eliminate this as a source of variability in the results.

#### Table 4

#### Lubricant properties

Density @ 15 °C	0.860kg/litre
Kinematic viscosity @ 100 °C	12.03mm2/s
Viscosity index	163
Viscosity CCS @ -30 °C	5260 CP
Total base number	15.9 mg KOH/g
Sulphated ash	0.19 per cent

## G. Gaseous emissions measurement systems

22. During emissions tests at all laboratories selected gaseous emissions were measured on a continuous basis from both raw and diluted exhaust. In addition, some laboratories supplied cumulative 'bagged' sample results. Raw exhaust samples were drawn directly from the exhaust line, while diluted samples and bagged analyses were made from the fullflow dilution system. Regulated exhaust gases and their methods of analysis are given below:

- (a) Total hydrocarbons (THC): performed using a heated Flame Ionisation Detector (FID).
- (b) Oxides of Nitrogen (NOx): conducted using a Chemiluminescence Analyser (CLA). CLA detects photons that are emitted by excited NO<sub>2</sub> molecules generated in the instrument reaction chamber from NO. Excited NO<sub>2</sub> emits photons of a specific wavelength. The light generated in the reaction is proportional to the NO present in the sample. All the NO<sub>2</sub> in the sample gas is reduced to NO prior to the reaction chamber. The combined concentration of NO+NO<sub>2</sub> is measured. As most oxides of nitrogen are generally in one of these two forms, this measurement is expressed as NOx.
- (c) Carbon monoxide (CO) using a Non-Dispersive Infra-Red (NDIR) instrument.
- (d) Though currently unregulated, carbon dioxide emissions were also measured: using NDIR instrument.

23. During the validation exercise, instrumentation provided by the following analyser suppliers was used for gaseous emissions analysis:

- (a) Horiba
- (b) AVL (both own branded and Pierburg)

## H. Dilution approaches

24. <u>Principles of the dilution systems</u>: In Europe since the implementation of Euro IV legislation, two dilution approaches have been considered equivalent for the measurement of particulate mass during certification testing. These full and partial flow dilution approaches are shown in schematic form in Figures 3 and 4 respectively.

Figure 3

#### Schematic of full flow, double dilution system for PM measurements



#### Figure 4

Schematic of partial flow dilution system for PM measurements



25. <u>Full flow dilution</u>: In a full flow dilution system, the entire exhaust is sampled and diluted but the total flow through the dilution system is maintained at a constant level. This is known as constant volume sampling and the dilution tunnel is often referred to as the Constant Volume Sampler (CVS). Since the exhaust flow varies with engine operation but the total flow through the CVS is fixed, the dilution ratio varies during a test.

26. Heavy duty dilution systems in Europe tend to be twin stage systems with a small secondary dilution system in series from the main CVS. This secondary dilution system takes a fixed proportion of the flow from the CVS and dilutes it by a preselected ratio. The main aim of this step is to reduce the temperature of the diluted exhaust. Diluted exhaust is drawn from the secondary dilution system through a filter. The sampled flow rate must be virtually constant and, by definition, proportional to the total flow through the CVS.

27. In the validation exercise, all the full-flow dilution systems were employed with secondary dilution systems for mass measurements, but particle number measurements were drawn directly from the primary CVS. The main issues with CVS dilution systems are:

- (a) The transfer time between engine emission and measurement of real time diluted gases in the CVS makes the identification of real time emissions effects complex
- (b) Hydrocarbons and PM are known to deposit and release from the transfer system between the exhaust manifold and CVS
- (c) Finally, the full flow CVS is a large, often ceiling-mounted, tube which is difficult to remove and consequently to clean. Carryover of PM emissions from previous tests may result in high background levels.

Full flow dilution systems provided by Horiba and AVL were tested in this work.

28. <u>Partial flow dilution</u>: Partial flow dilution systems (PFDS) are simpler, more compact and less expensive than CVS. In a PFDS, a fraction of the raw exhaust (a partial flow) is sampled and diluted. However, the transfer flow from exhaust to tunnel must be proportional to the total flow through the exhaust: In Figure 5; Q1 must constantly change during the transient cycle and this is achieved by varying the flow of dilution air that is added (Q3). As in the CVS, the total flow, Q2, remains constant, but unlike the CVS the entire tunnel flow is drawn through the PM filter. If an additional flow (Q4) is drawn for further mass or number measurements, an identical increase in the transfer flow, Q1 occurs. This reduces the dilution ratio in the tunnel and would increase the measurement device

(which is positioned downstream of the PM filter) or the changes in dilution corrected automatically by the software.

29. During preliminary work for this programme, JRC performed a comparison of PN measurements from partial flow systems which had both physical correction for the removal of Q4 (i.e. the flow was replaced) and software correction for the removal of Q4  $[^{11}]$ . These two approaches were shown to give equivalent results, but during the actual test programme only the software correction approach was employed.

## Figure 5 **PFDS – Principle of dilution**



30. To facilitate the changes in exhaust flow rate and dilution flow (Q3), real-time fast flow measurement and rapid changes in flow rate are required.

31. The main challenge for partial flow dilution systems is maintaining proportionality with the exhaust flow rate. Measurement procedures for particulate emissions using partial flow dilution systems and of gaseous emissions from raw exhaust gases under transient test conditions are defined in an International Standards Organisation (ISO) publication ISO16183:2002 [<sup>13</sup>].

32. Partial flow dilution systems provided by Horiba [<sup>14</sup>], AVL [<sup>15</sup>] and Control Sistems [<sup>16</sup>] were tested in this work. A PFDS system is also available from Sierra [<sup>17</sup>]. All these systems are fully compliant with ISO16183:2002 and in principle operate almost identically. The main differences between systems are the methods by which flow is measured and controlled.

#### I. Particulate mass measurements

33. <u>Full flow measurements</u>: In the PMP ILCE\_LD the filter-based PMP particulate mass measurement method was employed as the reference method. For conventional diesels this has been shown to give results consistent with the current regulatory particulate mass measurement method [<sup>18</sup>].

34. The development philosophy of the PMP particulate mass measurement system was to adapt the readily achievable elements of the mass method used for heavy duty approvals in the US for 2007, along with selected amendments to improve data quality, to create an enhanced European light duty procedure. Consequently, the approach developed for light duty vehicles in the ILCE\_LD was considered directly transferrable to heavy duty engines' full flow dilution system sampling and the main additions to the standard European method are described in the following sections.

Table 5

- (a) Application of highly efficient dilution air filters for particles and hydrocarbons that reduce mass contributions from the dilution air to near zero.
- (b) The application of a cyclone pre-classifier with a 50 per cent cut-size at between 2.5  $\mu$ m and 10  $\mu$ m to limit the contribution of re-entrained and wear materials to the filter mass.
- (c) External heating of the filter holder and transfer tubing to permit aerosol stabilisation of > 0.2 s at 47 °C  $\pm$ 5 °C prior to sampling and to ensure close control of the filter face temperature to 47 °C  $\pm$ 5 °C. External heating was achieved by either direct surface heating (most laboratories) or by situating the cyclone, transfer tubing and filter holder in an enclosed vessel. In the second case, the sample probe in the CVS was also heated.
- (d) The use of a single 47 mm filter rather than primary and back-up filters to eliminate weighing errors and the back-up filter as a source of volatile artefact
- (e) The filter medium provides at least 99 per cent filtration efficiency for 0.3 μm particles at 35 l/min (~50 cm/s filter face velocity).
- (f) Controlled filter face velocity range (50 cm/s to 80 cm/s) to improve reproducibility

35. Definition of PMP Particulate Mass (PM): Despite the changes introduced to the method, the PM definition remains broadly unchanged from that used previously: all materials sampled from a dilution tunnel using the prescribed method on to a single filter at between 42  $^{\circ}$ C and 52  $^{\circ}$ C.

36. Test facilities: The particulate measurement equipment employed by the participating test laboratories was constructed to meet the requirements of the ILG\_HD. All laboratories used full flow dilution systems equipped with secondary dilution tunnels. The dimensions of these systems, flow rates and residence times were subject to some differences as shown in Table 5.

37. CVS tunnel residence times were controlled to 1.6 s to 2.3 s range, but there was a much larger range in secondary tunnel dimensions and residence time (0.4 s to 7.8 s). As discussed later, these differences did not have a measurable impact on observed PM levels.

	AVL-MTC	JRC	Ricardo	UTAC	EMPA
CVS low ate [Nm <sup>3</sup> /min]	72	80	60	80	80
CVS length [cm]	500	470	450	575	470
CVS diameter [cm]	50	47	45	45	47
CVS Heat exchanger	No	Yes	Yes	Yes	Yes
Preclassifier cutpoint [um]	2.5	2.5	2.5	2.5	2.5
Secondary tunnel flowrate [lpm]	50	50	60	50	40
Secondary tunnel DR	2:1	2:1	2:1	2:1	2:1
Secondary tunnel length [cm]	30	64	100	30	20
Secondary tunnel diameter [cm]	8	8.6	10	8	8

Principal differences between CVS systems – Test laboratories

38. For physical collection of PM, all laboratories used TX40 filters, but some laboratories used current PM holders without a back-up and other laboratories used US07 style holders. Different filter holders did not have a measurable impact on observed PM levels.

39. <u>Partial flow dilution measurements</u>: Partial flow dilution measurements of PM were not undertaken in the ILCE\_LD, but development work was undertaken to refine the procedure in the working group that developed the ISO standard [<sup>13</sup>]. This procedure has many parallels with the PMP full flow method:

- (a) Efficient dilution air filtration;
- (b) Filter face temperature control is permissible;
- (c) 47 mm filters are permitted;
- (d) The same filter media are mandated.

40. In these bases, it was considered wise to conduct partial flow testing during the HD\_ILCE according to the requirements of ISO16183, but to align sampling parameters where possible with the full flow method. Parameters to be matched were prescribed in the inter-laboratory guide, but these included: filter face velocity, filter medium, filter diameter and dilution air quality. The definition of PM sampled from a partial flow dilution system: all materials sampled from a dilution tunnel using the prescribed method on to a single filter at 47 °C  $\pm$ 5 °C.

41. Test facilities: The particulate measurement equipment employed by the participating test laboratories was constructed to meet the requirements of ISO16183, with operating parameters specified in the ILG\_HD. All laboratories used commercially available PFDS systems. The dimensions of these systems, flow rates and residence times were subject to only minimal differences as shown in Table 6.

	AVL-MTC	JRC	Ricardo	UTAC	EMPA
System	Smart Sampler	Smart Sampler & PSS-20	Horiba Mini Dilution Tunnel	PSS-20	Smart Sampler
PM flow rate[g/s]	1.08	1.08	1.205	1.08	1.08
Split ratio [per cent]	0.06	0.06	0.09	0.06	0.06

Table 6 Principal differences between PFDS systems – Test laboratories

As seen with the CVS data, using different filter holders did not result in measurable differences in PM levels.

#### J. Particle number measurement systems

42. Principle of the measurement system: The particle number measurement systems employed within the exercise are known as the Golden Particle Measurement Systems (GPMS). The system is described as 'golden' only in that it represents an internal standard providing a link between testing at the various laboratories and continuity within the test programme.

43. The development philosophy of the particle number measurement system was to enable the accurate, repeatable and reproducible sampling of a well-defined particle sample from a very low background environment. It was also considered desirable to minimise required changes to the current type approval facilities, to employ an understandable metric and for the system to be simple to operate.

44. Measurement system elements: The particle number measurement system comprises a number of fixed elements; instrument manufacturers are free to employ their own technological solutions to meet the requirements of each.

45. Efficient dilution air filtration: Highly efficient dilution air filters for particles and hydrocarbons that reduce particle contributions from the dilution air to near zero.

46. Size pre-classification: A sampling inlet which serves to protect the downstream measurement system components from particulate contamination and set a nominal upper size limit for the particle size measured of  $2.5 \,\mu\text{m}$ .

47. Hot dilution: A first particle number diluter (PND1) which heats the sample while diluting it, in order to evaporate volatile particles and reduce the partial pressures of the gas phase species to prevent recondensation at the diluter exit.

48. Evaporation and cold dilution: A low particle loss externally heated Evaporation Tube (ET) in which the sample is heated to a fixed point between 300 °C and 400 °C and held for ~0.2 seconds while semi-volatile particles are evaporated. Any particles that remain in the aerosol after this point are considered to be 'solid' particles. This definition of 'solid' particles is analogous to the definition of regulatory gaseous hydrocarbons: defined as those materials that are measured by Flame Ionisation Detector (FID) downstream of a filter heated to 192 °C. Immediately after exiting the ET the sample enters a second particle number diluter (PND2), where it is cooled by dilution: the partial pressures of the gas phase species are further reduced to prevent recondensation, the concentrations of particles present are controlled such that they are within the single particle count mode of the PNC and thermophoretic losses are minimised.

49. Particle number counting: A particle number counter with a strictly controlled counting efficiency curve receives the sample as it exits PND2. This sets a nominal lower limit of  $\sim$ 23 nm to the size range measured. The strictly controlled counting efficiency curve is considered necessary to exclude the possible confounding of measurement data by low volatility hydrocarbons manifesting as a nucleation mode below 20 nm, while including the primary carbon sphere size of  $\sim$ 20 nm.

50. <u>Correction for particle losses</u>: In the light duty PMP inter-laboratory correlation exercise, differences between particle number results from measurement systems of discrete manufacturers were of the order  $\pm 15$  per cent [<sup>5</sup>]. These systems were subject to a calibration process which corrected for dilution only. In order to normalise these differences, the light duty legislation [<sup>4</sup>] and draft heavy duty legislation requires that both dilution factors and particle losses are corrected. A calibration process, defined in the legislation, determines the Particle Concentration Reduction Factors (PCRF). PCRF correction accounts for the full change in particle concentration, from inlet to particle counter, of a completely non-volatile aerosol with defined properties. PCRF are retrospectively applied to the measured particle numbers as part of the data processing step. The GPMS used in this study were not subjected to a full PCRF calibration until after completion of the inter-laboratory exercise. However, a comparison with other systems used in various participating laboratories, which had full PCRF calibrations, was conducted retrospectively (Chapter VII, Section E).

51. <u>Golden Particle Measurement Systems (GPMS)</u>: Two nominally identical particle number measurement systems were circulated across the participating laboratories for the concurrent determination of the particle number emissions from a full flow CVS tunnel and a partial flow system. These are referred to as GPMS, as they served as an internal standard providing a link between testing at the various laboratories.

52. The GPMSs selected for this study were two prototype Solid Particle Counting Systems (SPCS) developed by Horiba [<sup>19</sup>]. The selection of this particular system was not

based on its performance in terms of the criteria specified in the light duty regulations, but rather on the intention to evaluate alternative candidate systems to the GPMS that had been employed in the PMP Light Duty Inter-Laboratory Correlation Exercise [<sup>7</sup>].

53. SPCS description. The SPCS unit consists of a hot-diluter (PND1), an evaporation tube (ET), a cold diluter (PND2) and a condensation particle counter (PNC). A flow schematic of the SPCS unit is shown in Figure 6. The aerosol first enters a temperature controlled cabinet where it is diverged into a bypass flow, the sole purpose of which is the decrease of the residence time in the sampling line, and the sample flow. The sample mass flow rate is measured in real time by an orifice flowmeter (CFO), taking into account the temperature and the pressure of the sample as determined with a thermocouple and a pressure transducer, respectively. The sample is then diluted in a temperature controlled mixer (HD) with heated – filter dilution air supplied at an adjustable flowrate by means of a mass flow controller (MFC1).

54. A small fraction of the diluted aerosol exiting the PND1 passes through an orifice flowmeter and then enters an externally heated evaporation tube (EU) whose wall temperature is controlled in the range of 300 to 400 °C. During the  $\sim$ 0.5 s residence of the aerosol inside the EU, the volatile particles are vaporized to gas phase. Immediately after exiting the EU the thermally treated aerosol enters a mixer (CD) where it is cooled by filtered-dilution air supplied at an adjustable flowrate by means of another mass flow controller (MFC3). The concentration of the aerosol exiting this secondary diluter is then measured in real time in a TSI 3010D condensation particle counter (CPC). The excess flow from the two dilution stages is sampled with a pump. The dilution ratio of the two diluters is kept constant by supplying make-up air in the two excess lines. Two mass flow controllers (MFC2 and MFC4) continuously adjust the make up air to account for small fluctuations of the sample flow rates measured in real time with the two flowmeters.

55. Operating parameters: All laboratories participated in this study operated the two golden SPCS units at the same settings. The temperatures at the units were set at:

- (a) Cabinet temperature: 47 °C
- (b) Hot dilution air temperature for PND1: 170 °C
- (c) Mixer temperature (HD): 170 °C
- (d) Evaporation tube (ET): 350 °C

56. In these prototype units, the user has to specify the desired dilution ratio of each diluter as well as the dilution air flow rates and the bypass flow. The values employed in the campaign were:

- (a) Primary dilution ratio (PND1): 10
- (b) Primary dilution air flow rate (MFC1): 11.5 lpm
- (c) Secondary dilution ratio (PND2): 15
- (d) Secondary dilution air flow rate (MFC3): 10.5 lpm
- (e) Bypass flow rate: 2 lpm

57. These values were selected after preliminary experiments conducted at JRC. The dilution ratios fulfill the specifications laid down in R83 and the resulting sample flow rate ( $\sim$ 1.3 lpm) is high enough for pressure fluctuations to have an insignificant effect on the stability of the achieved dilution ratios.



58. <u>Other PMP type systems</u>: A number of alternative candidate systems operating on the pre-conditioning and measurement principles required by PMP have also been used in parallel by the participating laboratories. However, calibration data demonstrating compliance with PMP performance requirements has not necessarily been provided for all of these systems. These are briefly described below.

59. Nanomet: This system consists of a primary rotating disk diluter heated at 150 °C, a 1m transfer line, an evaporation tube operating at 300 °C and a secondary simple mixer diluter. Two Nanomet systems were tested at JRC (one of which was the GPMS employed in the ILCE\_LD) and one at Ricardo. The LD\_GPMS system used a TSI 3010D CPC as did the Nanomet employed at Ricardo. The second Nanomet system tested at JRC employed a TSI 3010 CPC modified to replicate the performance of a 3010D (operating at evaporator - condenser temperatures that provided 50 per cent detection efficiency at 23 nm) in some tests, and a TSI 3790 CPC in other tests.

60. APC: This system consists of a primary chopper diluter, a 2 m transfer line heated at 150 °C, an evaporating tube operating at 350 °C and a secondary dilution stage operating with dilution air at ambient temperature. One APC system was tested at JRC during the second measurement campaign and one at AVL MTC. These systems utilize TSI 3790 CPCs.

61. Dekati dual ejector and evaporating tube system: In this system the first dilution is applied by a Dekati ejector diluter equipped with a heating mantle (150 °C) using heated, conditioned (dehumidified, HEPA filtered) dilution air at 150 °C. The diluted sample is then thermally treated in an evaporation tube heated to 330 °C and then further diluter in a secondary Dekati ejector using conditioned dilution air at ambient temperatures. This system was employed at JRC during both measurement campaigns, and was tested with three different CPC models: a TSI 3010D, a TSI 3010 and a Grimm 5.404 (the latter two modified in order to shift the 50 per cent detection efficiency to 23 nm).

62. EMPA's dual ejector and evaporating tube system: This system consists of a Palas ejector diluter (Palas VKL-10E) heated at 150 °C (by means of a heating mantle) operating on conditioned dilution air at 150 °C, an evaporation tube operating at 350 °C, and a secondary Palas ejector diluter (Palas VKL-10) operating on conditioned air at ambient temperature. Two nominally identical systems were employed at EMPA, one of them

sampling from the CVS tunnel (using a TSI 3790 CPC for particle detection) and the other directly from the exhaust (using a TSI 3010 CPC for particle detection).

63. <u>PMP like systems</u>: Some tests were also conducted at JRC using sampling systems employing a Dekati thermodenuder operating at 300°C for the thermal treatment of the aerosol. This replaced the evaporation tube of the PMP type systems. The carbon absorbent section of this particular thermodenuder has an annular design. During the tests, the inner cylinder was cooled by forced convection (using compressed air) and the outer one by natural convection. The thermodenuder operated at a flow rate of 10 lpm which corresponds to a residence time of 0.3 seconds in the heating section and 2.7 seconds in the denuder. The thermodenuder sampled from the CVS tunnel either directly or via a heated (heating blanket at 150°C) Dekati ejector operating with heated dilution air at 150°C. A TSI 3790 CPC was employed for particle detection.

64. <u>Additional instrumentation</u>: In addition to the aforementioned PMP type systems, the participating laboratories have also employed additional aerosol instrumentation in order to get a better insight into the characteristics of the emitted particles. These included:

65. EEPS: A 3090 Engine Exhaust Particle Sizer Spectrometer (EEPS) (TSI Inc.) was used during a limited number of tests. EEPS measured particle size distributions with a maximum data rate of 10 size distributions per second (although averages over 1 second were used in the graphs of this study). It measured particle sizes from 5.6 to 560 nm with a sizing resolution of 16 channels per decade (a total of 32 channels). At the instrument's inlet there was a cyclone with a 50 per cent cut-size at 1  $\mu$ m (inlet flow rate 10 lpm).

66. Dekati Mass Monitor: A Dekati Mass Monitor (DMM) was employed at JRC during some tests conducted after the second measurement campaign. The DMM sampled from the CVS tunnel via a Dekati thermodenuder operating at 300 °C, and was used to measure the mass concentration of non-volatile particles in real time.

67. AVL Soot Sensor: An AVL 483 soot sensor was employed during some of the formal tests conducted during the second measurement phase at JRC, measuring the mass of soot in real time. The soot sensor sampled directly from raw exhaust at a constant dilution ratio of about 2.

68. TSI SMPS: A TSI 3936L SMPS (consisting of a TSI 3080L DMA and a TSI 3010 CPC) was employed during some preliminary tests at JRC operating at sample/sheath flowrates of 0.9 and 9 lpm respectively.

69. TSI 3025A CPC: During some of the preliminary tests conducted at JRC two TSI 3025A CPC units having a 50 per cent counting efficiency at 3 nm were employed. One of them was used to sample directly from the CVS tunnel, and the other to sample from the GPMS connected to the CVS tunnel, and in parallel to the golden CPC (50 per cent efficiency at 23 nm). These tests allowed for the determination of the number concentrations of volatile and non-volatile particles in the 3-23 nm size range. This provided the means to investigate whether significant emissions of non-volatile particles smaller than 23 nm, suggested by recent studies [<sup>20</sup>], are also observed with the golden engine.

## K. Test programme

70. Participating Laboratories: Four laboratories in EC member states, and one in Switzerland, were participants in the test programme. The test laboratories, timing and final test order are given in Table 7. JRC also conducted additional experiments prior to and following the formal testing, these experiments are not included in the assessment of measurement repeatability and reproducibility.. Preliminary experiments, undertaken to refine the inter-laboratory guide are discussed in the next section.

1 est Laboratories and	1 1 menne		
Dates	Test Laboratory	Location	Testing
Jan–Feb 2008	JRC	Ispra, Italy	Preliminary Experiments
Mar-Apr 2008	AVL-TC	Sweden	Formal Testing Lab#1
May–Jun 2008	JRC	Ispra, Italy	Formal Testing Lab#2
Dec 2008 – Jan 2009	Ricardo	United Kingdom	Formal Testing Lab#3
Feb-Apr 2009	UTAC	France	Formal Testing Lab#4
Apr–Aug 2009	EMPA	Switzerland	Formal Testing Lab#5
Aug–Oct 2009	JRC	Ispra, Italy	Formal Testing Lab#2rpt
Nov 2009	JRC	Ispra, Italy	Additional Experiments

Table 7 Test Laboratories and Timeline

71. <u>Summary of preliminary experiments</u>: A number of experiments were conducted in order to better define the measurement approaches described in the inter-laboratory guide. These were based upon concerns regarding the possible differences in emissions between the light duty vehicles that the measurement procedures were developed for, and the heavy duty engines to be the subject of the next phase of PMP work. Further details can be found elsewhere [<sup>10</sup>]. In particular, the differences in PM chemistry, the aftertreatment devices to be used, characteristics of different dilution systems and possible differences in the nature of particles: their sizes and origins, were of concern. These experiments can be classified into four groups as follows:

- (a) Experiment 1: Background and filter tests. These experiments considered the CVS and partial flow systems' background levels for mass and the effect of the filter medium and filter face velocity (ffv) on the PM emissions
- (b) Experiment 2: Sampling parameters. These experiments considered the impact and necessity of using a pre-cyclone with the Horiba SPCS systems. In addition determined
- (c) Experiment 3: Pre-conditioning and continuity protocol. In the ILCE\_LD, it was determined that to improve the repeatability of particle number results a purge of pre-existing particles from the exhaust system and a standardised DPF fill-state was required. Experiments were undertaken to determine the minimum required pre-conditioning for the exhaust and after treatment for repeatable measurements.
- (d) Experiment 4: Real time PN emissions. Concerns have been raised in the US that high levels of solid particles may be present, in the exhaust from HD diesel engines, in the size range below the PMP cut-off (d50) of ~23 nm. Measurements were undertaken to determine the presence and magnitude of < 23 nm solid and < 23 nm volatile particles from the golden engine.</p>

72. All experiments were conducted on the golden engine and emissions control system and using the fuel and lubricant described in Chapter II, Section E.

73. Experiment 1 - Background and sampling parameters for PM:

A schematic of the sampling system used in these experiments is shown in Figure 7.



Figure 7 PM S

74. Evaluations of filter media and face velocity effects were undertaken during repeat WHSC testing. In all cases tests were conducted using a primary and back-up filter to enable the magnitude of volatile 'slippage' from the primary filter to the secondary filter to be quantified. The following measurements were made from the CVS, with results shown in Figure 8.

- (a) (i) One background (BG) PM measurement followed by 3 hot WHSC repeats
  - (ii) 70 mm TX40 filters, PM sample flow of 60l/lmin (ffv = 43 cm/s)
- (b) (i) One background PM measurement followed by 3 hot WHSC repeats
  - (ii) 47 mm TX40 filters, PM sample flow of 601/lmin (ffv = 103 cm/s)
- (c) (i) One background PM measurement followed by 3 hot WHSC repeats
  - (ii) 47 mm TX40 filters, PM sample flow of 401/lmin (ffv = 69 cm/s)
- (d) (i) One background PM measurement followed by 1 hot WHSC
  - (ii) 47 mm TX40 filters, PM sample flow of 401/lmin (ffv = 69 cm/s)
  - (iii) PM filters baked in an oven at 47 °C for 3 hours to remove residual volatiles
- (e) (i) One background PM measurement followed by 1 hot WHSC
  - (ii) 47 mm Teflon membrane filters, PM sample flow of 40l/lmin (ffv = 69 cm/s)

Figure 8



Experiment 1 - Background and sampling parameters for PM

75. The conclusions of these experiments were:

- (a) PM emissions were slightly higher than background levels
- (b) Highest PM mass was collected on 70 mm filters with a sample flow of 60 l/min (ffv of ~40cm/s)
- (c) Similar masses were collected on 47 mm filters at ffvs of ~70 to 100 cm/s (40 to 60 l/min)
- (d) Baking TX40 filters has no beneficial effect
- (e) Teflo filters appear to collect less mass than 47 mm filters
- (f) Secondary (backup filters) collect ~30 per cent of primary filter mass from sample filters
- 76. Contributions to test protocol
  - (a) No substantive changes to light duty PM protocol
  - (b) Employ 50 l/min flow rate for PMP tests (for full flow and partial flow systems with 47 mm TX40 filters).

77. <u>Experiment 2 – PN Sampling parameters:</u> In these experiments 2 SPCS systems were used in parallel during various periods of transient engine operation.

- (a) Transfer line effects (Figure 9)
  - (i) One SPCS sampling directly from the CVS with a 1m insulated line
  - (ii) One SPCS sampling directly from the CVS with a 4m heated line
- (b) Cyclone effects (Figure 10)
  - (i) One SPCS sampling directly from the CVS with a 1m insulated line
  - (ii) One SPCS with 4 m heated line sampling from the CVS via a cyclone operating with a 4 m cut

#### Figure 9 Minimal impact of heated line on PN Results



Figure 10 Minimal impact of cyclone on PN



78. The conclusions of these experiments were:

(a) The differences in PN emissions measured with a 4 m heated line (at 47 °C, with 1.8 s residence time) and a 1m insulated line (0.5 s residence time) were minimal (< 5 per cent). This means that one SPCS can be connected to the CVS through 4 m line (heated at 47 °C), while the other SPCS can be</p>

connected to the partial flow system with a short (insulated) line without adjustment of results for transport losses.

- (b) The cyclone had a negligible effect on the particle number emissions, so for number measurements it is not necessary. However, it is recommended to use one in order to protect instruments' primary diluters from contamination through deposition of larger particles. Insulation of the cyclone and transfer tubes is required to limit thermophoretic losses during high temperature operation.
- 79. Contributions to test protocol
  - (a) Cyclone mandatory for CVS sampling
  - (b) Cyclone optional for partial flow sampling
  - (c) Insulation of cyclone and external sampling system to limit thermophoretic losses
  - (d) Dedicated SPCS (Serial no. 19) for full flow sampling (with heated line)
  - (e) Dedicated SPCS (Serial no. 20) for partial flow sampling (with 1 m insulated line)

80. Experiment 3 – Preconditioning protocols: A daily preconditioning protocol was required that first used a high exhaust temperature steady state ( $\sim 600$  °C) to passively regenerate the DPF, and then used a lower temperature non-regenerating condition to add a standardised quantity of soot to the DPF. This protocol was used at the end of each day to re-baseline the loading state of the DPF prior to the next day's run-through of the test matrix. To avoid very long test days, it was desirable that the entire process was shorter than 2 hours.

(a) 15 minutes at mode 10 was determined as the suitable engine operation and minimum time required to passively regenerate the DPF, eliminating the stored soot. This was determined by running ESC 10 and monitoring how long it took for post DPF solid particle emissions to stabilise (Figure 11, green line, 400 s to 900 s).

Mode 10 operation for 15 minutes showed particle number levels consistent with those seen during the 2 hours of mode 10 running used to condition the lubricating oil, thus indicating that the DPF was indeed 'emptied' by the 15 minutes period (Figure 11, red and blue lines).

(b) ESC Mode 7 has a stabilised exhaust temperature of well below 300 °C, so passive regeneration at this temperature, even with an oxidation catalyst, is minimal. 30 minutes of operation was chosen to add soot to the DPF (Figure 11, green line, 1500 s to 3300 s)

81. The daily test matrix comprised both cold and hot start tests and it was desirable that each lab tested the cycles with the exhaust and emissions control system at the comparable temperatures. This would reduce variability due to pre-conditioning effects and allow a better assessment of the repeatability of gaseous and particle emissions measurement procedures. To ensure this, cycles without regulatory defined warm-ups were preceded by the continuity protocol.

82. The hot WHTC was preceded only by the cold WHTC and a 10 minute soak while the WHSC, was preceded by the hot WHTC and the mandatory 10 minutes at WHSC Mode 9. No continuity protocols were required for these cycles. The continuity protocol comprised 5 min at Mode 7 of the ESC and 3 min at idle. Mode 7 was selected after repeat size distribution measurements at this condition showed no evolution in magnitude or size –

indicating stability of both volatile and solid particles (Figure 12). By comparison a slightly hotter steady state, ESC Mode 4 showed obvious evolution, related to some passive regeneration. Idle followed the Mode 7 operation to permit preparation of the exhaust emissions analysers and to limit emissions and fuel consumption without stopping the engine.



DPF Preconditioning - regeneration (Mode 10) and fill (Mode 7)

Figure 11





- 83. Contributions to test protocol:
  - (a) 15 minutes ESC Mode 10 plus 30 minutes ESC Mode 7 for the daily preconditioning
  - (b) 5 minutes ESC Mode 7 plus 3 minutes at idle for the continuity protocol

84. Experiment 4 – Real-time PN emissions: Concerns have been raised in the US [<sup>22</sup>] that high levels of solid particles may be present, in the exhaust from HD diesel engines, in the size range below the PMP cut-off (d50) of ~23 nm. Measurements were undertaken to determine the presence and magnitude of < 23 nm solid and < 23 nm volatile particles from the golden engine. A variety of steady state and transient emissions tests were performed measuring solid particles from the CVS with an SPCS system equipped with both a TSI 3010D CPC (measuring particles > 23 nm) and a TSI 3025A CPC (measuring particles > 3 nm). The difference between the results of these two particle counters indicated the presence of solid particles in the size range 3 nm to 23 nm. In addition, the same 3025A CPC was used to measure particles directly from the CVS. This permitted the number of volatile particles smaller than 23 nm to be determined. Results are summarised in Figure 13.

#### Figure 13 Particle number emissions < 23 nm



- 85. The implications of this figure can be summarised as follows:
  - (a) For the cycles examined, the concentration of total particles > 3 nm measured from the SPCS was generally 50-95 per cent higher than the > 23 nm nonvolatile particles. The difference appears to be higher over the hot WHTC cycle, measuring particle concentrations direct from the CVS (300 per cent). CVS measurements include both solid and volatile particles and this result is therefore likely to be indicative of a higher proportion of volatile particles over this cycle
  - (b) For the cycles examined the concentration of the non-volatile particles < 23 nm was 15-45 per cent higher than the non-volatile particles > 23 nm (85 per cent for the cold WHTC).

86. Impact of Experimental Result on the Test Protocol for the Validation Exercise:

While there was some evidence that solid particles < 23 nm were present, the levels seen were not consistent with the orders of magnitude increases relative to >23nm particles reported from US engines. On this basis, it was considered reasonable to retain the size and volatility range of particles measured in the ILCE\_LD for measurements from heavy duty engines.

87. <u>Daily Protocol</u>: Following the completion of the preliminary experiments, the test protocol for the inter-laboratory exercise was finalised and used to update the ILG\_HD. The baseline test matrix comprised at least 8 repeats of each of the following tests:

- (a) Cold WHTC
- (b) Hot WHTC
- (c) WHSC
- (d) ETC
- (e) ESC

88. Test order followed the defined matrix (Table 8), with preconditioning for each cycle set as the regulatory requirement, or the continuity protocol if no regulatory requirement exists. The continuity protocol was defined as 5 minutes operation at ESC Mode 7 plus 3 minutes at idle (as described in experiment 3 of Chapter II, Section K).

#### Table 8

## **Matrix for Emissions Testing**

Previous lab	Day 0	Days 1-7	Day 8		
	Oil change	IFV	IFV		
	2h ESC Mode 10	Cold WHTC	Cold WHTC		
		10 min at WHSC Mode 9	10 min at WHSC Mode 9		
		WHSC	WHSC		
		СР	СР		
		ETC	ETC		
		СР	СР		
		ESC	ESC		
2 hours at ESC Mode 10*	Precon	Precon	2 hours at ESC Mode 10*		
ESC - European Steady State Cycle for emissions measurement [30 minutes]					
ETC - European Transient Cyc	le for emissions measu	rement [30minutes]			
WHSC - World Harmonized St	eady State Cycle for en	nissions measurement [30 min	nutes]		
WHTC - World Harmonized Transient Cycle for emissions measurements [30 minutes]					
IFV - Instrument Functional Verification					
CP - Continuity Protocol					
Density 16 minutes DOC and b					
Precon - 15 minutes ESC mode	e 10, 30 minutes ESC n	node 7			

## **III.** Statistical Analyses

89. The variability of the results collected in the PMP Heavy Duty Validation Exercise was quantified using the "random effects analysis of variance" model  $[^{23}]$ . This analysis provides the means for a separate quantification of the repeatability and reproducibility of the measurements.

90. On the other hand, the equivalency between the results (PM and particle number) obtained from the CVS tunnel, the partial flow system and the alternative particle number systems employed, was investigated by means of calculating the average value and the standard deviation of the percentage differences of each individual result. It has been decided not to employ paired t-tests for this type of check as the particular methodology is prone to identifying statistically significant differences when in fact the result are practically equivalent [<sup>24</sup>], with the ability to discriminate between statistically and practically significant differences strongly affected by the sample size.

#### A. Definitions

91. <u>Basic statistical concepts</u>: Before describing the statistical analysis used for the evaluation of the PMP methodology it is important to distinguish between the statistical concepts of accuracy, precision and trueness.

92. Accuracy represents the degree of agreement between the results obtained from a test method and the true or 'accepted' true value. On the other hand, precision refers to how closely the independent measurements agree with each other, while trueness refers to the closeness of agreement between the arithmetic mean of a large number of test results and the "accepted" reference value. In most cases, however, the true value is unknown and therefore only precision statements can be developed. Precision is a qualitative concept which can be expressed numerically only in terms of its opposite, that is the variance or standard deviation.

93. The variance in the results obtained from a test method is due to some random variations of the properties being measured, but also due to the fluctuation of some factors affecting the outcome of the test method. These factors are generally the equipment used, the calibration of the equipment, the operators using the equipment and the environmental variables.

94. When the test method is performed in one laboratory in the shortest practical period of time, by the same operators, using the same equipment on – ideally – materials taken from a single quantity of homogeneous material, then the aforementioned factors remain reasonably constant and the variance in the results is referred to as the within laboratory (intra-laboratory) variability ( $\sigma^2$ ). However, when a test method is performed at different laboratories these factors vary considerably, leading to even greater variability. The variability induced because of performing the tests in different physical environments is the between laboratories (inter-laboratory) variability.

95. The results obtained from one laboratory are said to be satisfactory if they are both repeatable and reproducible. Repeatability is ensured when the above mentioned factors remain reasonably constant. This would be reflected where inter-laboratory variance was similar to intra-laboratory variance. Furthermore, the results obtained from one laboratory are said to be reproducible if the divergence of their mean value is not significantly greater than the variability range which would be expected on the basis of the inter-laboratory variability.

96. <u>Intra- and inter-laboratory variance estimators</u>: The random effects analysis of variance model provides the means for the quantification of the intra-laboratory and interlaboratories variances  $\sigma^2$  and  $\sigma_T^2$ , respectively. If  $y_{ij}$  represents the j<sup>th</sup> result obtained from the i<sup>th</sup> laboratory participating in an inter laboratory correlation exercise conducted at p different laboratories, and n<sub>i</sub> is the number of results provided from the i<sup>th</sup> laboratory then the estimators of  $\sigma$  and  $\sigma_T^2$  are [<sup>23</sup>]:

$$s_{r}^{2} = \frac{\sum_{i=1}^{p} \sum_{j=1}^{n_{i}} y_{ij}^{2} - \sum_{i=1}^{p} \frac{y_{i.}^{2}}{n_{i}}}{N - p}$$

$$s_{L}^{2} = \frac{\left(\frac{\sum_{i=1}^{p} \frac{y_{i.}^{2}}{n_{i}} - \frac{y_{..}^{2}}{N}\right)}{\left(p - 1\right) - s_{r}^{2}}}{\frac{1}{p - 1} \left[N - \frac{\sum_{i=1}^{p} n_{i}^{2}}{N}\right]}$$
Where:  $N = \sum_{i=1}^{p} n_{i}$ ,  $y_{i.} = \sum_{j=1}^{n_{i}} y_{ij}$  and  $y_{..} = \sum_{i=1}^{p} \sum_{j=1}^{n_{i}} y_{ij}$ 

97. <u>Statistical outliers</u>: One of the basic assumptions of the analysis of variance model is that the results obtained from each laboratory are equally variable (a situation known as homogeneity of variance). If the variability in one particular laboratory is significantly different from the rest of the laboratories the particular laboratory is said to have repeatability problems and can be characterized as an outlier. The ASTM 691-99 and the ISO 5725-2:1994 standards provide the means for identifying the laboratories having repeatability problems, by utilizing the repeatability index (k). The k statistic for the results obtained from a laboratory z is defined as

$$k_z = \frac{s_z}{s_r}$$

where  $s_z$  represents the standard deviation of the results obtained from laboratory z, while  $s_r$  is the estimated intra-laboratory standard deviation (square root of  $s_r^2$ ).

98. Furthermore, the two standards suggest the use of the reproducibility index (h) in order to identify laboratories measuring significantly higher or lower results. The h statistic for the results obtained from laboratory z is defined as:

$$h_z = \frac{\overline{x}_z - \mu}{s_0} = \frac{\overline{x}_z - \mu}{\sqrt{ns_L^2 + s_r^2}}$$

where  $x_2$  represents the average value of the results obtained from laboratory z,  $\mu$  is the pooled mean value from all laboratories and  $s_0$  represents the range where the difference is expected to vary due to the intra and inter-laboratory variabilities ( $s_r^2$  and  $s_L^2$ , respectively).

99. The statistical analysis underlying the definition of these two statistics is the hypothesis testing of two variances and two means respectively, and therefore some critical h\* and k\* values can be derived by assuming a type-I error (a). Any h or k value greater or equal to the corresponding critical h\* or k\* is indicative of reproducibility or repeatability problems, respectively. There are also additional patterns indicating problems like one lab having positive (or negative) h values and all the rest negative (or positive), or one lab having too high or too low k values for all tests compared to the rest of the laboratories.

100. The two aforementioned standards provide the equations and critical values for the case of balanced samples (equal number of results from each laboratory). However, in this exercise not all laboratories performed exactly 8 repetitions of the test protocol. Additionally, for various technical reasons, some of the data were excluded and therefore the final sample ended up statistically unbalanced. The following equations apply in the more general case of unbalanced samples [ $^{25}$ ]:

$$k_{z} = \frac{\sqrt{\frac{\sum_{j=1}^{n_{z}} \left(y_{zj} - \frac{y_{z}}{n_{z}}\right)^{2}}{(n_{z} - 1)}}}{s_{r}}$$

$$h_{z} = \frac{\frac{\overline{x_{z}} - \frac{\sum_{i=1}^{p} \overline{x_{i}}}{p}}{\sqrt{\frac{\sum_{i=1}^{p} \left(\frac{y_{i}}{n_{i}} - \frac{y_{..}}{N}\right)^{2}}{p - 1}}}{\sqrt{\frac{n_{z} - 1}{\sum_{i=1}^{p} (n_{i} - p)}} + \frac{1 - \frac{(n_{z} - 1)}{\sum_{i=1}^{p} n_{i} - p}}{F}}$$

where t is the  $\alpha/2$  percentage point of the Student's t-distribution with p-2 degrees of freedom, and F the  $\alpha$  percentage point of the F-distribution for  $n_z$ -1 degrees of freedom for the numerator and [N-p-( $n_z$ -1)] degrees of freedom for the denominator. Following the recommendations of the ISO standard, the critical h and k statistics were calculated at a significance level ( $\alpha$ ) of 1 per cent.

101. The standard deviations contain information on the absolute level of each property tested. In order to compare the variability of properties differing by orders of magnitude it is necessary to normalize the standard deviation with respect to the average emission levels. For this purpose, the within-laboratory  $(s_r/\mu)$  and between variability  $(s_L/\sigma)$  coefficients of variation (CoV) are used instead in this document. For convenience these quantities are also referred to as within and between laboratories variability.

# **IV.** Calibration and validation

## A. Mass systems

102. Filter mass measurement procedures in the weighing room (or chamber) were conducted according to the requirements of the ILG\_HD. During the entire exercise, no substantive issues were reported with the weighing procedures of the HD\_ILG at any of the test laboratories.

103. Figure 14 shows the results of weighing environment validation exercises undertaken at Ricardo. During a calendar month there are typically no deviations from temperature range (19 °C to 25 °C), relative humidity range (37 per cent to 53 per cent) or in the performance of the balance (50.002  $\mu g \pm 5 \mu g$ ). However, several borderline results from reference filter weighings suggests that variability (of 47 mm TX40 filters) increases such that  $\pm 10\mu g$  range could be required instead of the  $\pm 5 \mu g$  used in Regulation No. 83. This is in line with the requirements of the WHDC.



#### Weighing room validation exercises - Ricardo



#### **B.** Number systems

104. Prior to the commencement of the ILCE\_HD, the two SPCS systems and their PNCs (3010D) were calibrated by their manufacturers. These calibrations were performed prior to the finalisation of the PMP calibration methodologies, including that for particle concentration reduction factor (PCRF). The calibrations performed are outlined in the following sections.

105. <u>SPCS Calibration</u>: The two SPCS systems used as golden instruments during the SPCS-20 was subjected to a comprehensive characterisation, while SPCS-19, built in parallel with identical componentry, was subjected to a dilution ratio gas calibration only but compared with the SPCS20 within the main PMP programme and shown to be highly similar. The calibration approach used for the SPCS-20 has been published by Horiba [<sup>26</sup>] and is briefly outlined below. This included determination of VPR penetration using poly disperse aerosol and monodisperse aerosols and dilution ratio validation using a propane

span gas and a flame ionisation detector. The removal efficiency of the VPR for 30 nm tetracontane (n-C<sub>40</sub>) particles was determined according to the procedure previously described by Horiba [<sup>27</sup>].

106. <u>Removal efficiency for tetracontane particles</u>: The apparatus used by Horiba to determine the volatile particle removal efficiency (RE) of SPCS-20 is shown in Figure 15. The C40 aerosol generator heats up to the boiling point of tetracontane and the neutraliser and DMA are used to select monodisperse 30 nm particles. The concentration of these particles is reduced by a room temperature ejector diluter to below 10,000/cm<sup>3</sup> and they then enter the evaporation unit (EU) at 300 °C. Upstream and post-EU concentrations are compared to evaluate the RE.

Figure 15

Apparatus for the evaluation of volatile particle RE



A typical result for this particle removal test, showing efficiency of 99.99 per cent for 30nm  $n-C_{40}$  particles is given in Figure 16.





Solid particle penetration using polydisperse aerosol: The apparatus used by Horiba 107. to determine the solid particle penetration of SPCS-20 using polydisperse NaCl is shown in Figure 17. Temperatures of the PND1 dilution air, mixer, EU, and sample flow were controlled to 150 °C, 150 °C, 320 °C, and 47 °C, respectively. These temperatures were used for normal SPCS operation at the time of these experiments. Subsequently, higher evaporation tube temperatures and other parameters have entered standard use. It is not expected that this will have significantly impacted the penetration performance or results of other calibration activities. Sodium Chloride (NaCl) aerosol is generated with the atomizer and residual moisture in the aerosol is then removed with an efficient diffusion dryer. A bypass is placed upstream of the diffusion dryer to vent excess aerosol flow. In the neutralizer, the aerosol is charged to Boltzmann equilibrium. An ejector diluter is used to provide an aerosol at the correct concentration level and to enable the flow to be controlled by the SMPS. The concentration and the size distribution then remain constant while the upstream (raw) and the downstream (diluted) size distributions of the VPR are measured. Comparisons of these distributions for specific size ranges (Figure 18) and at different dilution ratios provide the penetration efficiencies (Figure 19). At the typical dilution factor used in the PMP work (~150), the polydisperse penetration was > 98 per cent.

#### Figure 17

Apparatus for evaluation of solid particle penetration (polydisperse)











108. Solid particle penetration using monodisperse aerosol: The apparatus used by Horiba to determine the solid particle penetration of SPCS-20 using monodisperse NaCl is shown in Figure 20. Temperatures of the PND1 dilution air, mixer, EU, and sample flow were controlled to 150 °C, 150 °C, 320 °C, and 47 °C, respectively. These temperatures were used for normal SPCS operation at the time of these experiments. Sodium Chloride (NaCl) aerosol is generated with the atomizer and residual moisture in the aerosol removed with an efficient diffusion dryer. A by-pass is placed upstream of the diffusion dryer to vent excess aerosol flow. In the neutralizer, the aerosol is charged to Boltzmann equilibrium. An ejector diluter is used to provide an aerosol at the correct particle concentration level (less than 10,000/cm<sup>3</sup> upstream of the VPR for each monodisperse particle size) and to enable the flow to be controlled by the CPC. The DMA is employed to select particles of 30 nm, 50 nm and 100 nm electrical mobility diameter. Pre and post VPR concentrations are compared to determine penetration levels. Results of these comparisons at several dilution ratios are shown in Figure 21, Figure 22 and Figure 23 for 30 nm, 50 nm and 100 nm particles respectively. Figure 20

#### Compressed Air By - Pass Downstream Polydisper tomizer By-pass VPR CPC Drye L . Eiector Compressed Air Upstream Monodisperse DMA Neutralize

#### Apparatus for evaluation of solid particle penetration (monodisperse)

Figure 21 SPCS Penetration of 30 nm particles





Figure 22 SPCS Penetration of 50 nm particles

Figure 23 SPCS Penetration of 100 nm particles



109. These data convert to a range of PCRF values from 1.06 (taking all the highest results irrespective of dilution ratio) to 1.20 (all the lowest penetrations). This compares reasonably well with the value of 1.25 obtained at JRC (Table 13) using the finalised PCRF procedure. Since real-world usage of PMP PN systems will always be for the measurement of polydisperse aerosols, the development of a robust calibration procedure using polydisperse particles is clearly desirable and remains an on-going research objective. It is currently unclear why penetration efficiencies for monodisperse and polydisperse particles differ: this too is an appropriate topic for future research.

110. <u>Dilution ratio calibration using propane span gas</u>: Comparisons of HC concentrations, corrected for ambient levels, were made upstream and downstream of the VPR at a range of dilution ratios, using a flame ionisation detector. One series of experiments was performed with a fixed primary dilution ratio and another with a fixed secondary dilution ratio. Results are shown in Figure 24 and Figure 25. Results showed that dilution ratio settings were within  $\pm 4$  per cent of the nominal value.




Figure 25 Dilution ratio calibration – fixed primary dilution



111. <u>3010D Calibration</u>: Particle number counters used with the two SPCS systems were calibrated according to the procedures described by TSI [<sup>28</sup>]. This uses the primary method in which particle counting is verified by comparison with the response of a electrometer. The counting efficiencies of the PNCs were determined through the use of an electrospray generated polyalpholefin aerosol classified in a DMA. Certificates of conformity for the two PNCs used with the SPCS systems are given in Appendix 2. These data were supplied directly by TSI [<sup>29</sup>].

112. <u>Validation exercises</u>: No substantive issues were observed with the PN validation procedures of the HD\_ILG at any of the test laboratories during the programme. These included zero checks of the PNC and SPCS systems and flow checks of the PNC. Functional checks (indicative monitoring of flows and temperatures) were undertaken on a continuous basis and any issues resolved via routine maintenance.

### V. Emissions Results

### A. Full flow and partial flow PM

113. Phase II of PMP made recommendations for introducing a solid particle number measurement procedure and also for improving the current EU and ECE regulatory particulate mass measurement procedure. The aim of Phase III of PMP was to validate the measurement techniques recommended in Phase II. The ILCE LD validated both measurement techniques for use in light duty vehicle emissions measurement. The ILCE HD also included measurements to evaluate the revised particulate mass measurement technique recommended by PMP Phase II when applied to heavy duty engine emissions measurement. However, international agreement on improvements to particulate mass measurement techniques for heavy duty engine emissions testing has already been reached within global technical regulation (gtr) No. 4. Many of the particulate mass measurement improvements adopted in gtr No. 4 are consistent with the PMP Phase II recommendations. For these reasons it is not the intention of the PMP informal group to propose amendments to regulatory, heavy duty particulate mass measurement procedures. However the results and conclusions of the measurements made using the revised particulate mass measurement procedures during the ILCE HD are reported here for completeness. Particulate mass data from full and partial flow dilution systems are both discussed in this section.

114. <u>PM – Repeatability</u>: Repeatability levels for all laboratories are expressed as single CoV values that express overall intra-lab variability for each emissions cycle (see Chapter III, Section A). Figure 26 shows the repeatability of the 5 test matrix cycles and the composite weighted WHTC result for the CVS-based PM method. Three results are shown for each cycle, and these include:

- (a) PMCVS1: All data from all laboratories (excepting tests excluded for technical reasons)
- (b) PMCVS2: Outlier analysis iteration 1
- (c) PMCVS3: Outlier analysis iteration 2



### **Repeatability of the CVS PM method**



38

115. <u>Outlier analyses found no exclusions</u>: The best repeatability of ~34 per cent was seen from the cold start WHTC cycle, with all other cycles showing between 50 per cent and 56 per cent. Filters from emissions tests revealed that the cold start WHTC showed both the highest sample masses and visible grey staining indicating the presence of some elemental carbon. Other cycles' filters did not show the same discoloration. Figure 27 shows the repeatability of the 5 test matrix cycles and the composite weighted WHTC result for the PFDS-based PM method. Figure 27



Repeatability of the PFDS PM method

- 116. Three results are shown for each cycle, and these include:
  - PMPFDS1: All data from all laboratories (excepting tests excluded for technical reasons)
  - (b) PMPFDS2: Outlier analysis iteration 1
  - (c) PMPFDS3: Outlier analysis iteration 2

117. Outlier analyses led to the exclusion of all PM results from UTAC which showed PM levels significantly higher than PFDS results from any other lab. Interestingly, this lab's PFDS results showed very similar levels to its CVS-measured results. Following this analysis, the resulting repeatability levels were typically 20 per cent to 30 per cent across all emissions cycles.

118. <u>PM – Reproducibility</u>: Reproducibilities are given as single CoV values that express overall inter-lab variability for each emissions cycle (see Chapter III). Reproducibility levels for the CVS and PFDS PM methods are shown in Figure 28. Data generated following two rounds of outlier iterations are shown. CVS PM reproducibility levels were typically in the range 35 per cent to 55 per cent, averaging 42.7 per cent for the 5 emissions cycles in the test matrix. PFDS PM reproducibility levels ranged from ~30 per cent to ~45 per cent, averaging 36.1 per cent. The lower CoVs from the PFDS systems probably reflect the greater consistency of tunnel background levels in the partial flow system compared to the CVS.



Figure 28 **Reproducibility of PM methods** 

119. <u>PM – Filter Weights, Background Levels and Background Subtraction</u>: Testing at Ricardo included both CVS and PFDS PM measurements, and a comparison has been made of the filter masses collected from background and cycle testing.

120. <u>Filter Weights</u>: In all cases filter masses proved to be higher from the CVS than from the partial flow system (Figure 29). The greatest differential between measurement systems proved to be from the WHTC cycles, with results between the systems closest from the ESC cycle. Filter loadings seldom exceeded 50  $\mu$ g with partial flow sampling (collected masses ranged from a high of 59  $\mu$ g ESC) to a low of zero (also ESC)). CVS levels were both higher and covered a wider range: from 346  $\mu$ g over one cold WHTC down to 31  $\mu$ g (ESC).

Figure 29 Sampled filter masses – various cycles, both measurement systems



121. Figure 30 puts measured filter masses into context of the tunnel background filter levels recorded:

- (a) It is clear that from the CVS, the highest sample mass is higher than the highest tunnel background, but the lowest sample mass is lower than the lowest tunnel background.
- (b) From the partial flow system, the highest and lowest sample masses are roughly equivalent to the highest and lowest tunnel background masses.
- 122. The observations from this testing were as follows:
  - (a) Mass emissions measured by the partial flow system may be indistinguishable from the dilution tunnel background. In which case, mass emissions from all cycles are effectively zero.
  - (b) Mass emissions measured by the CVS system were in some cases higher than the tunnel background levels, but it is also possible that the CVS tunnel background levels at this laboratory may be unrepresentative.

123. If the first observation and comment (all mass emissions measured by the partial flow system were effectively zero) is correct, and the partial flow results are valid, then the highest CVS results must have been seeing a greater tunnel background contribution during the test than from the pre-test measurement.

Figure 30





124. This testing suggested that tunnel background subtraction of filter masses from partial flow and a substantial number of full flow tests is likely to give net PM results of 0 mg/km. This may be a true indication of the mass emissions of this engine with DPF when the resolution of the mass method is considered. Tunnel background PM was also frequently measured at EMPA and JRC. Tunnel background PM filters were drawn prior to the cold start WHTC emissions test each day. The mass on each tunnel background filter was then treated as a cycle emissions result (masses were adjusted for differences in sample times between cycles) and the corresponding mg/kWh emissions figure calculated using engine data from that day's emissions tests.

125. <u>CVS PM Tunnel Background</u>: As Figure 31 shows for CVS-sampled PM data from both EMPA and JRC, only measurements from ESC cycles were above tunnel background levels. The ESC cycle has a substantial period of operation at high exhaust temperatures and this may lead to emissions of low volatility compounds that are efficiently collected and

then retained by the filter. Filters from other cycles collect higher volatility materials from the exhaust and dilution air, but these can be released following acquisition through volatilisation or through a washing effect as further aerosol is drawn through the filter. It is worth noting that except for hot start WHTC measurements at EMPA, both samples and tunnel backgrounds during the ILCE\_HD results were above the limits of detection (3 standard deviations of the blank measurement) for the various cycles.

### Figure 31



Tunnel background and sample PM levels - CVS

126. These data suggest that the CVS PM method is capable of resolving PM emissions from ESC tests from tunnel background levels. Results from other cycles, including the cold WHTC are subject to high uncertainty and would reduce to zero if tunnel background subtraction was undertaken.

127. <u>PFDS PM Tunnel background</u>: Tunnel background and sample filter comparisons were also made from partial flow dilution systems at JRC and EMPA (Figure 32). EMPA results reflected the CVS results, where all cycles' data except ESC were similar to the tunnel background levels. JRC results, conversely, showed that it is possible to discriminate PM samples from the tunnel background, but this discrimination is poorest from the hot and cold WHTC cycles. Tunnel background correction of the JRC PFDS results would reduce the emissions levels from cold start WHTC to (generally) < 1 mg/kWh, hot start WHTC to 0.5 g/kWh or less, WHSC to ~1.2 g/kWh, ETC to 0.5 g/kWh or less and ESC to < 2 mg/kWh.







### Table 9

Limits of Detection – PM Methods at JRC and EMPA (mg/kWh)

	EMPA CVS	EMPA PFDS	JRC CVS	JRC PFDS
C_WHTC	4.9	1.2	0.18	0.10
H_WHTC	13.8	1.7	0.24	0.15
WHSC	6.4	0.9	0.11	0.07
ETC	1.7	1.0	0.07	0.05
ESC	2.2	0.2	0.03	0.03

129. <u>PM – Emissions Levels</u>: Figure 33 (CVS) and Figure 34 (PFDS) show the maximum to minimum ranges of PM emissions levels seen from each emissions cycle at each laboratory. Ranges are shown as error bars above and below the mean values. These data are not corrected for tunnel backgrounds.

130. <u>PM emissions from CVS Systems</u>: Emissions levels from the CVS (Figure 33) showed the largest ranges from Ricardo and EMPA, where tunnel background levels were substantially higher than other laboratories. These laboratories also showed some of the lowest emissions values as well as some of the highest, but were sampled according to the prescribed protocols using compliant equipment and thus were not eliminated as outliers by simple statistical techniques. The contribution of high and variable tunnel background PM to the results from EMPA and Ricardo is believed to be responsible for the variable PM results from these two laboratories and this may be related to the recent test history of the

facilities. In particular, Ricardo had undertaken testing on high bio-content fuels, active regeneration strategies for DPF regeneration and substantial amounts of non-DPF testing on low NOx calibration engines. All these types of testing would be expected to contribute substantially to both volatile and carbonaceous CVS tunnel backgrounds.

131. The test protocols were designed to help purge the CVS system of residual tunnel backgrounds, but it is clear that in some cases extreme measures may be required to eliminate historical PM from full flow dilution systems. For this reason it may be necessary to permit the subtraction of a tunnel, rather than dilution air, tunnel background for regulatory PM purposes. Tunnel background levels of PM in other laboratories were very low (typically < 1 mg/kWh).

132. Generally speaking, and excepting some results from Ricardo and EMPA which were higher, PM emissions from all cycles were < 6 mg/kWh, with no obvious difference in emissions between the cold and hot start WHTC cycles. These levels are substantially below the 10 mg/kWh limits set for the weighted WHTC and WHSC at Euro V and expected for Euro VI. Tunnel background correction of the PM results from Ricardo [<sup>30</sup>] (which showed the highest emissions of all laboratories) brought them in line with other laboratories: reducing Cold WHTC results to ~8 mg/kWh, hot WHTC results to ~7 mg/kWh, WHSC to ~4 mg/kWh, ETC to ~1 mg/kWh and ESC to < 1 mg/kWh.

133. <u>PM Emissions from PFDS Systems</u>: PM emissions measured by PFDSs (Figure 34) showed narrower ranges than those measured from CVS systems. Statistical analyses identified PM results from UTAC as systematically higher, with mean values from all cycles at between 4 mg/kWh and 7 mg/kWh. It is possible that the PFDS used by UTAC had been employed for non-DPF testing and was contributing particles during emissions tests. This is analogous to the high tunnel background PM levels observed by Ricardo from their CVS. By contrast, PFDS emissions levels from other laboratories, and considering all emissions cycles, rarely exceeded 4 mg/kWh.

134. As seen in the CVS PM data, the emissions levels from cold and hot start WHTC do not appear to differ. Comparisons between CVS and PFDS mass data are discussed in Chapter VII, Section E, but emissions from the same engine measured simultaneously from CVS and PFDS appear, in general, to be lower from the PFDS. This is likely to be related to the fact that almost all the PFDSs tested were relatively new and one, tested at Ricardo, had never been used before. Newer dilution systems are less likely to have been exposed to old technology higher PM engines. Tunnel background contributions to PM are therefore likely to be low.

135. It is widely assumed that dilution systems reach a deposition and entrainment equilibrium where losses to the dilution tunnel walls are balanced by re-suspension. This follows a period where particle deposition to tunnel walls is favoured. 'Clean' systems may be within the deposition-dominated phase and this may explain the directionally lower PM results seen with the PFDS data from Ricardo.



# Figure 33 Maximum and minimum ranges of emissions – CVS PM

# Figure 33 (cont'd) Maximum and minimum ranges of emissions – CVS PM





# Figure 34 Maximum and minimum ranges of emissions – PFDS PM

UTAC

EMPA JRC#2

WHTC Cold

PMCVS [mg/kWh]

10

Ŧ

AVLMTC JRC#1 Ricardo

AVLMTC JRC#1 Ricardo UTAC EMPA JRC#2



Ŧ

WHTC Hot

Ŧ

25

20

PMCVS [mg/kWh]



WHTC Combined

Ŧ

UTAC

Ŧ

EM PA JRC#2

30

25

20

15

1

Ŧ

AVLMTC JRC#1 Ricardo

PMCVS [mg/k/Mh]

Ŧ

136. PM – Daily Trends. The trends in PM emissions across the day's test sequence are shown for CVS PM in Figure 35 and for PFDS PM in Figure 36. Data are shown from all test laboratories, with concurrently sampled data from the CVS and PFDS shown.

137. From the CVS (Figure 35), profiles from the JRC, UTAC and AVL-MTC tests are relatively similar, with PM levels remaining relatively flat through the test sequence. The laboratories with the high tunnel backgrounds show different profiles. These results suggest that if any changes in emissions levels occur due to cycle-to-cycle variations or changes to DPF fill state occur with passive regeneration within individual cycles, CVS PM is insensitive to them. Alternatively, the results suggest that in this testing any effects that do occur have been masked by tunnel background levels even in the laboratories with low emissions.



CVS PM Emissions through the test sequence – All laboratories

138. From the partial flow system (Figure 36), PM levels were relatively consistent through the test sequence from all laboratories except UTAC where the tunnel background levels were higher. It is possible that the PM method detects a reduction in cycle PM between the cold and hot WHTC cycles, but as with the CVS data, the PFDS PM measurements in this testing were either insufficiently sensitive to detect changes in DPF fill state through the day's test sequence, or the effects were masked by tunnel background levels.



138. Overview of PM Results

- CVS PM results, after elimination of the high tunnel background laboratories (a) data, showed emissions levels of < 6 mg/kWh across all cycles
- (b) Emissions levels from the PFDSs were generally lower at 4 mg/kWh or less from all cycles, with the exception of UTAC's results which were in the range 4 mg/kWh to 7 mg/kWh. This may have been due to a higher PFDS tunnel background than other laboratories, but there is insufficient data to draw a firm conclusion
- CVS PM tunnel backgrounds were generally at the same level as samples (c) except from ESC cycles, as ESC PM contains low volatility HCs which, once collected, remain on the filter. As a consequence, the CVS PM measurements in this testing only appeared capable of resolving engine emissions from the tunnel background for ESC tests.
- (d) Tunnel background PM levels in PFDS systems were at the low end of levels seen from CVS systems. Even so in only one of 3 systems, in which tunnel background levels were determined, was it possible to discriminate data from any more cycles than the ESC.
- In the one PFDS system that enabled discrimination between tunnel (e) background and sample levels, emissions from all cycles were < 2 mg/kWh and specifically  $\sim 1.2$  mg/kWh from the WHSC and < 1 mg/kWh from the weighted WHTC.

#### В. Full flow and partial flow PN

140. PN - Tunnel background levels: PN tunnel background levels appear to vary substantially between CVS systems in different laboratories, but appear highly similar between partial flow systems. While data are not available for tunnel backgrounds in all laboratories, comparisons of hot transient cycle results from different laboratories, where particle emissions are low, clearly shows the offsets due to tunnel backgrounds.

141. As Figure 37 shows, particle number emissions measured from the CVS from different laboratories across the hot WHTC can vary by a factor of 100 or more. In contrast (Figure 41), particle number emissions from PFDSs sampled simultaneously to the CVS data appear to overlay.

142. JRC, AVL and Ricardo (RCE) also supplied particle number data from 30 minutes dilution system tunnel background acquisitions taken prior to the cold start WHTC on each test day. A typical tunnel background from each of the PFDS and CVS facilities at these laboratories was calculated to particles/kWh and the values obtained were compared. These data are shown in Figure 38.

143. The CVS tunnel background at Ricardo was > 4 x  $10^{10}$ /kWh. This was ~60 times higher than the CVS tunnel background at AVL-MTC and ~140 times higher than the CVS tunnel background at JRC. These differences are in line with the differences in emissions levels seen from the hot start transient test shown in Figure 37, and indicate that from Ricardo and to a lesser extent AVL, the tunnel background predominates in the particle numbers measured on these tests.

144. The tunnel background levels from the 3 PFDS systems are almost identical: at  $< 3 \times 10^8$ /kWh. This indicates that the small variations in particle number levels seen in Figure 38 are probably related to real engine or DPF variability rather than tunnel background contributions.

145. As noted in Chapter V, Section A, where PM tunnel background was discussed, the Ricardo facility is a working facility which had recently experienced testing on high biocontent fuels, work on active regeneration strategies for DPF regeneration and substantial amounts of non-DPF testing on low NOx calibration engines. An elevated PM tunnel background was seen from this laboratory (see Figure 39) and it is apparent the tunnel background also contributes substantially to PN.



Tunnel background impacts PN results - CVS Systems at 3 laboratories







146. <u>PN – Repeatability as transient particle production</u>: Figure 40 to Figure 44 inclusive show real-time repeatability traces for cold WHTC, hot WHTC, WHSC and ESC tests. Each figure shows PN results from a laboratory with the highest CVS particle number tunnel background (upper), results from the laboratory with the lowest CVS PN tunnel background (middle) and typical results from a partial flow system (lower). The bottom and middle charts' data are drawn from the same test laboratory.

147. Data from the cold start WHTCs shown in Figure 40 (upper) covers approximately two orders of magnitude, but the range seen in the middle and lower figures covers more than 4 orders of magnitude. It is also clear that data in the top figure shows little similarity to the engine speed after the first 700 s, but the middle and lower figures reflect changes in engine operation throughout the emissions cycle.

148. The high levels of tunnel background seen in the CVS (Figure 40, upper) do not have a substantial effect on repeatability, because the overall emissions levels from the cycle are dominated by those of the first 700 s. Repeatability levels from the data shown, based upon mean cycle results, would be relatively similar to those seen for Figure 40, middle and lower.

149. The contrast between the upper and middle parts of Figure 40 is clear: while the profiles of emissions coincide at the peaks, the less transient parts of the emissions cycles can be masked if the tunnel background is high. Results from the lower figure are highly similar to those of the middle figure, indicating that there are not fundamental differences in

either emissions levels or transient measurements from full and partial flow systems as long as tunnel background levels are similar.

150. The sensitivity of the measurement is obviously affected by the tunnel background, but as mentioned above, the dominance of the peaks seen in the first 700 s makes even a high tunnel background almost irrelevant to results from the cold WHTC emissions cycle.

151. Results from the hot start WHTC (Figure 41) were substantially affected by the high CVS tunnel background. A comparison between high (upper figure) and low (middle figure) tunnel background CVS facilities shows that the transient traces from the high tunnel background lab bear little or no relationship to the transient events of the cycle and span less than a factor of 10. Conversely, the emissions of the low tunnel background CVS (middle figure) and from the partial flow system (lower figure) indicate transient events corresponding to engine operation and emissions ranges that span 2 to 3 orders of magnitude. Once again, results from the flow tunnel background CVS and partial flow systems appear very similar.

152. Peak emissions levels from the partial flow system (and low tunnel background CVS) were  $\sim$  1000 times lower than the peaks from the cold start WHTC, but in the high tunnel background CVS, cold WHTC emissions only appeared to be 10 times higher than the hot start WHTC. Clearly, the hot WHTC repeatability from the high tunnel background CVS system reflects little more than the repeatability of the tunnel background levels.

153. Emissions from the WHSC cycle (Figure 42) were at a similar level to those seen from the hot WHTC (Figure 41), but the emissions profile from the high tunnel background CVS (upper figure) tracks the engine speed trace well. This suggests that the tunnel background is less significant during this cycle than in the hot WHTC, so running the previous two cycles in the test matrix may have had a 'cleaning' effect on the CVS.

154. From all three dilution tunnels, it appears that particle number emissions are very low from the WHSC until ~1200 s. The exhaust temperature of the WHSC is sufficient to enable passive regeneration (where NO<sub>2</sub> reacts with soot on the DPF to produce N<sub>2</sub> and CO<sub>2</sub>) during ~60 per cent of the emissions cycle (Figure 43, left).

155. It is possible that after ~1200 s of the WHSC, the soot loading of the DPF has reduced sufficiently to have a substantial impact on filtration efficiency. The exhaust temperature has also increased to ~450 °C at this point in the cycle (Figure 43, right) and this might lead to the thermal release of low volatility components from the exhaust system. Both these mechanisms could increase particle number emissions and variability.

156. The ETC cycle is tested following the WHSC in the test sequence. Results, emissions levels and repeatability from this cycle were very similar to those from the hot WHTC. The ETC runs at relatively low exhaust temperatures, so during this cycle additional soot is added to the DPF following passive regeneration in the WHSC. This may have the effect of increasing filtration efficiency for the start of the ESC.









53

Figure 41 Real-time data – cycle-to-cycle repeatability, hot WHTC







54











Figure 43 Passive regeneration during the WHSC

157. Emissions during the 4 minutes of idle at the start of ESC cycle (Figure 44 upper, middle and lower) were very low and probably indistinguishable from tunnel background in all dilution systems. As with the WHSC cycle, most of the modal transitions of the ESC are visible even in the high tunnel background data (upper figure), but the range of emissions seen in this system (~100 from low to high) compares poorly with the 4 orders of magnitude from the low tunnel background CVS and PFDS and indicates lowered sensitivity of this measurement system.

158. The ESC shows the highest variability levels on a cycle-to-cycle basis, and these appear to worsen after 1000 s of the cycle: at this point exhaust temperatures rise substantially, reaching > 600 °C after ~1300 s. Passive regeneration in this cycle may eliminate the soot replaced during the ETC cycle and further eliminate soot from the DPF. Any variations in initial soot loading during the previous night's preconditioning may manifest as variability in the ESC.









159. <u>PN - Repeatability as CoV</u>: Repeatability levels for all laboratories are given as single CoV values that express overall intra-lab variability for each emissions cycle (see Chapter III).

160. Figure 45 shows the repeatability of the 5 test matrix cycles and the composite weighted WHTC result for the CVS-based and PFDS-based PN methods. Three results are shown for each cycle, and these include:

- (a) PNCVS1: All data from all laboratories (excepting tests excluded for technical reasons)
- (b) PNCVS2: Outlier analysis iteration 1
- (c) PNCVS3: Outlier analysis iteration 2

161. Outlier analyses excluded the CVS PN results from both Ricardo and EMPA on the first pass iteration excepting: cold WHTC data from both laboratories and ESC data from EMPA.

162. On the second pass analysis, the ETC data from the first batch of tests at JRC were excluded.

163. Considering all cycles (Figure 24), and following the outlier iterations, repeatability levels were broadly similar: CVS CoVs ranged from  $\sim$ 20 per cent to  $\sim$ 60 per cent and PFDS CoVs from  $\sim$ 20 per cent to  $\sim$ 70 per cent. Focusing on the Euro VI legislative cycles in isolation shows that the CVS approach has better repeatability over the weighted WHTC (21.1 per cent vs. 22.8 per cent) and over the WHSC (59.2 per cent vs. 74.4 per cent) than the PFDS approach.

Figure 45 **Repeatability of Particle Number measurement systems** 





164. <u>PN – Reproducibility</u>: Reproducibility levels for all laboratories are given as single CoV values that express overall inter-lab variability from each emissions cycle (see Chapter III). Figure 46 shows the reproducibility of the 5 test matrix cycles and the composite weighted WHTC result for the CVS-based and PFDS-based PN methods.

165. The outlier analysis has a substantial effect on reducing the variability of the CVS system's results (Figure 46 upper), but has little impact on the PFDS results, except in the ETC where the first results from JRC were eliminated from the dataset. Considering just the Euro VI legislative cycles shows that the CVS approach has better reproducibility over the weighted WHTC (41.4 per cent vs. 45.8 per cent) and over the WHSC (81.7 per cent vs. 86.3 per cent) than the PFDS approach.





166. <u>PN – Emissions Levels</u>: Particle number emissions from the various test cycles are shown for CVS measurements in Table 10 and Figure 47 and for PFDS measurements in Table 11 and Figure 49. These Tables and Figures include all test results including those rejected as outliers by the statistical analyses.

167. <u>PN Emissions from CVS Systems</u>: Particle number emissions from the cold WHTC cycle ranged by approximately an order of magnitude across all laboratories – from ~6 x  $10^{10}$ /kWh to ~7 x  $10^{11}$ /kWh with the all-laboratories mean at ~4 x  $10^{11}$ /kWh. Hot WHTC results ranged from  $10^{9}$ /kWh (JRC  $2^{nd}$  campaign) up to ~5 x  $10^{11}$ /kWh at Ricardo, a difference from low to high of 500 times. The all laboratories mean, at ~6 x  $10^{10}$ /kWh, was substantially impacted by the laboratories with high emissions levels.

Table 10

Maximum and minimum ranges for CVS-measured PN							
		WHTC Cold	WHTC Hot	WHTC Combined	WHSC	ETC	ESC
	Average	5.1E+11	8.1E+09	5.9E+10	1.3E+10	4.1E+09	3.0E+10
AVL MTC JRC #1 Ricardo UTAC EMPA	Maximum	6.1E+11	1.9E+10	7.8E+10	3.2E+10	6.3E+09	3.8E+10
	Minimum	3.9E+11	4.7E+09	4.5E+10	5.4E+09	3.2E+09	2.1E+10
	Average	3.9E+11	5.3E+09	4.3E+10	4.0E+10	1.6E+10	3.9E+10
JRC #1	Maximum	4.3E+11	6.2E+09	4.8E+10	6.9E+10	1.8E+10	4.2E+10
	Minimum	3.5E+11	4.3E+09	3.9E+10	red PN           Combined         WHSC           .9E+10         1.3E+10         4.           .8E+10         3.2E+10         6.           .5E+10         5.4E+09         3.           .3E+10         4.0E+10         1.           .8E+10         6.9E+10         1.           .8E+10         6.9E+10         1.           .8E+10         6.9E+10         1.           .9E+10         2.4E+10         1.           .9E+10         2.4E+11         3.           .2E+11         4.2E+11         3.           .3E+11         1.7E+11         1.           .6E+10         1.7E+10         8.           .0E+10         2.3E+10         1.           .3E+10         1.1E+10         4.           .6E+10         1.7E+10         4.           .0E+10         2.3E+10         1.           .3E+10         5.7E+10         1.           .8E+10         5.7E+10         1.           .9E+10         3.3E+09         2.           .5E+10         5.0E+09         3.           .4E+09         1.9E+09         1.           .9E+10         7.7E+10         4. <td>1.3E+10</td> <td>3.6E+10</td>	1.3E+10	3.6E+10
	Average	5.2E+11	2.9E+11	3.1E+11	2.9E+11	2.3E+11	3.5E+11
Ricardo	Maximum	6.3E+11	5.2E+11	5.2E+11	4.2E+11	3.5E+11	5.5E+11
	Minimum	4.3E+11	2.0E+11	2.3E+11	urea F'N           TC Combined         WHSC         ETC         E           5.9E+10         1.3E+10         4.1E+09         3.0           7.8E+10         3.2E+10         6.3E+09         3.8           4.5E+10         5.4E+09         3.2E+09         2.1           4.3E+10         4.0E+10         1.6E+10         3.9           4.8E+10         6.9E+10         1.8E+10         4.2           3.9E+10         2.4E+10         1.3E+10         3.6           3.1E+11         2.9E+11         2.3E+11         3.5           5.2E+11         4.2E+11         3.5E+11         5.5           2.3E+11         1.7E+11         1.3E+11         1.7           5.6E+10         1.7E+10         8.0E+09         1.3           7.0E+10         2.3E+10         1.1E+10         1.8           4.3E+10         1.1E+10         4.9E+09         5.0           1.1E+11         9.8E+10         2.7E+10         7.2           1.4E+11         1.3E+11         4.5E+10         9.4           6.8E+10         5.7E+10         1.5E+10         4.8           1.9E+10         3.3E+09         1.11         7.4E+09         1.9E+09         3.8E+09	1.7E+11	
	Average	4.0E+11	1.8E+10	5.6E+10	1.7E+10	8.0E+09	1.3E+11
UTAC	Maximum	5.1E+11	2.9E+10	7.0E+10	2.3E+10	1.1E+10	1.8E+11
	Minimum	2.9E+11	1.0E+10	4.3E+10	WHSC         ETC           1.3E+10         4.1E+09           3.2E+10         6.3E+09           5.4E+09         3.2E+09           4.0E+10         1.6E+10           6.9E+10         1.8E+10           2.4E+10         1.3E+11           2.4E+11         3.5E+11           4.2E+11         3.5E+11           1.7E+11         1.3E+11           1.7E+10         8.0E+09           2.3E+10         1.1E+10           1.1E+10         4.9E+09           9.8E+10         2.7E+10           1.3E+11         4.5E+10           5.7E+10         1.5E+10           3.3E+09         2.1E+09           5.0E+09         3.8E+09           1.9E+09         1.1E+09           4.2E+11         3.5E+11           1.9E+09         1.1E+09           7.7E+10         4.8E+10           221.4         327.3           1.8E+10         7.6E+09	5.0E+10	
	Average	5.0E+11	6.5E+10	1.1E+11	9.8E+10	2.7E+10	7.2E+10
EMPA	Maximum	7.4E+11	9.7E+10	1.4E+11	1.3E+11	4.5E+10	9.4E+10
	Minimum	3.6E+11	3.1E+10	6.8E+10	N           ined         WHSC         ETC           1.3E+10         4.1E+09           3.2E+10         6.3E+09           5.4E+09         3.2E+09           4.0E+10         1.6E+10           6.9E+10         1.8E+10           2.4E+10         1.3E+11           2.4E+10         1.3E+11           2.9E+11         2.3E+11           3.2E+10         1.3E+11           4.2E+11         3.5E+11           1.7E+10         8.0E+09           2.3E+10         1.1E+10           1.7E+10         8.0E+09           2.3E+10         1.1E+10           1.7E+11         4.9E+09           9.8E+10         2.7E+10           1.3E+11         4.5E+10           5.0E+09         3.8E+09           5.0E+09         3.8E+09           1.9E+09         1.1E+09           4.2E+11         3.5E+11           1.9E+09         1.1E+09           7.7E+10         4.8E+10           221.4         327.3           1.8E+10         7.6E+09	1.5E+10	4.8E+10
	Average	1.7E+11	2.4E+09	1.9E+10	1.7E+11         1.3E+11           1.7E+10         8.0E+09           2.3E+10         1.1E+10           1.1E+10         4.9E+09           9.8E+10         2.7E+10           1.3E+11         4.5E+10           5.7E+10         1.5E+10           3.3E+09         2.1E+09           5.0E+09         3.8E+09           1.9E+09         1.1E+09           4.2E+11         3.5E+11	5.8E+10	
JRC #2	Maximum	3.1E+11	4.5E+09	3.5E+10	5.0E+09	3.8E+09	1.1E+11
	Minimum	6.5E+10	1.0E+09	7.4E+09	1.9E+09	1.1E+09	2.6E+10
	All Min	7.4E+11	5.2E+11	5.2E+11	4.2E+11	3.5E+11	5.5E+11
	All Max	6.5E+10	1.0E+09	7.4E+09	1.9E+09	1.1E+09	2.1E+10
All data	Mean of means	4.1E+11	6.4E+10	9.9E+10	7.7E+10	4.8E+10	1.1E+11
	Range Factor	11.4	500.8	69.7	221.4	327.3	26.8
Outlier labs excluded	Mean of means	4.1E+11	8.6E+09	4.9E+10	1.8E+10	7.6E+09	6.6E+10

- (a) Weighted WHTC results, based upon the cold and hot WHTC data providing 10 per cent and 90 per cent contributions respectively, ranged from  $\sim 7 \times 10^9$ /kWh to  $\sim 5 \times 10^{11}$ /kWh with an all laboratories mean of  $\sim 10^{11}$ /kWh.
- (b) WHSC and ETC results reflected the results of the hot WHTC, with lowest emissions levels at JRC and highest at Ricardo. WHSC emissions levels ranged by more than 200 times: from  $2 \times 10^{9}$ /kWh up to ~4 x  $10^{11}$ /kWh and ETC ranged by a factor of > 300, from ~ $10^{9}$ /kWh up to ~ $3.5 \times 10^{11}$ /kWh.
- (c) ESC results showed a narrower range than any of the emissions cycles except the Cold WHTC. Low to high range covered a factor of <30, from  $\sim 2 \times 10^{10}$ /kWh up to  $\sim 5.5 \times 10^{11}$ /kWh.



# Figure 47 Maximum and minimum ranges for CVS-measured PN

168. As described in Chapter III, statistical methods were used to eliminate individual PN data and a majority of data from several cycles at two specific laboratories in the dataset. Excluded data are:

- (a) Ricardo CVS data from WHTC Hot, WHSC, ETC, ESC
- (b) EMPA CVS data from WHTC Hot, WHSC, ETC
- (c) ETC data from the first set of tests at JRC

169. The effects on the mean of means (the average PN emissions across all laboratories) of eliminating the outlier data is shown in Figure 48. The Cold WHTC data are unaffected since no data are excluded, but the hot WHTC result is substantially reduced, resulting in a weighted WHTC reduction of > 50 per cent to ~5 x 10<sup>10</sup>/kWh. The WHSC result drops to < 2 x 10<sup>10</sup>/kWh, the ETC to below 10<sup>10</sup>/kWh and the ESC to < 7 x 10<sup>10</sup>/kWh.



170. <u>PN Emissions from PFDS Systems</u>: Particle number emissions from the cold WHTC cycle ranged by approximately an order of magnitude across all laboratories – from ~6 x  $10^{10}$ /kWh to ~7 x  $10^{11}$ /kWh with the all-laboratories mean at ~3.7 x  $10^{11}$ /kWh. From the PFDS, cold WHTC emissions levels were substantially higher than from any other cycles.

Maximum and minimum ranges for PFDS-measured PN								
		WHTC Cold	WHTC Hot	WHTC Combined	WHSC	ETC	ESC	
	Average	6.3E+11	4.3E+09	6.7E+10	1.0E+10	2.2E+09	2.8E+10	
AVL MTC	Maximum	7.3E+11	4.9E+09	7.7E+10	2.4E+10	3.3E+09	3.8E+10	
	Minimum	4.7E+11	3.8E+09	5.0E+10	WHSC         ETC           1.0E+10         2.2E+09           2.4E+10         3.3E+09           3.7E+09         1.6E+09           4.4E+10         1.8E+10           7.3E+10         2.1E+10           2.4E+10         1.3E+10           7.3E+10         2.1E+10           2.4E+10         1.3E+10           5.6E+10         5.3E+09           1.4E+11         1.1E+10           2.5E+10         1.0E+09           9.1E+09         9.5E+08           4.4E+10         8.1E+09           9.1E+10         1.0E+10           2.5E+10         4.8E+09           2.6E+09         1.8E+09           3.9E+09         2.9E+09           1.2E+09         1.0E+02           1.4E+11         2.1E+10           3.9E+09         2.9E+09           1.4E+10         1.0E+09           3.9E+09         5.8E+08           2.8E+10         6.2E+09           119.2         22.4	2.0E+10		
	Average	4.0E+11	5.3E+09	4.4E+10	4.4E+10	ETC 2.2E+09 3.3E+09 1.6E+09 1.8E+10 2.1E+10 2.1E+10 2.1E+10 1.3E+10 1.1E+10 1.1E+10 1.1E+10 2.1E+09 2.1E+09 2.1E+09 3.5E+08 3.8E+09 3.8E+08 3.8E+09 3.8E+09 3.8E+08 3.8E+09 3.8E+08 3.8E+09 3.8E+08 3.8E+09 3.8E+08 3.8E+08 3.8E+09 3.8E+08	4.2E+10	
JRC #1	Maximum	4.7E+11	5.8E+09	5.2E+10	7.3E+10	2.1E+10	4.8E+10	
	Minimum	3.3E+11	4.0E+09	3.8E+10	2.4E+10	1.3E+10	3.6E+10	
	Average	2.3E+11	7.4E+09	3.0E+10	ed         WHSC         ETC           1.0E+10         2.2E+09           2.4E+10         3.3E+09           3.7E+09         1.6E+09           4.4E+10         1.8E+10           7.3E+10         2.1E+10           2.4E+10         1.3E+10           2.4E+10         1.3E+10           2.4E+10         1.3E+10           2.4E+10         1.3E+10           2.6E+10         1.0E+09           9.1E+09         2.1E+09           1.5E+10         4.3E+09           2.7E+09         9.5E+08           4.4E+10         8.1E+09           9.1E+10         1.0E+10           2.5E+10         4.8E+09           2.5E+10         4.8E+09           2.6E+09         1.8E+09           3.9E+09         2.9E+09           1.2E+09         1.0E+09           1.4E+11         2.1E+10           2.8E+09         2.8E+09	2.1E+11		
Ricardo	Maximum	3.4E+11	1.0E+10	4.3E+10	1.4E+11	1.1E+10	3.3E+11	
	Minimum	1.4E+11	4.5E+09	1.8E+10	2.5E+10	1.0E+09	1.4E+11	
	Average	4.1E+11	4.8E+09	4.5E+10	9.1E+09	2.1E+09	6.8E+10	
UTAC	Maximum	6.2E+11	7.1E+09	6.4E+10	1.5E+10	4.3E+09	9.9E+10	
	Minimum	2.9E+11	1.9E+09	3.1E+10	ined         WHSC         ETC           1.0E+10         2.2E+09           2.4E+10         3.3E+09           3.7E+09         1.6E+09           4.4E+10         1.8E+10           7.3E+10         2.1E+10           2.4E+10         1.3E+10           2.4E+10         1.3E+10           2.6E+10         5.3E+09           1.4E+11         1.1E+10           2.5E+10         1.0E+09           9.1E+09         2.1E+09           2.7E+09         9.5E+08           4.4E+10         8.1E+09           2.5E+10         4.8E+09           2.5E+10         4.8E+09           2.6E+09         1.8E+09           3.9E+09         2.9E+09           1.2E+09         1.0E+10           1.2E+09         9.5E+08           2.8E+10         6.2E+09           1.4E+11         2.1E+10	2.4E+10		
	Average	4.2E+11	8.5E+09	4.9E+10	2.4E+10 3.3E+09 3.7E+09 1.6E+09 4.4E+10 1.8E+10 7.3E+10 2.1E+10 2.4E+10 1.3E+10 5.6E+10 5.3E+09 1.4E+11 1.1E+10 2.5E+10 1.0E+09 9.1E+09 2.1E+09 1.5E+10 4.3E+09 2.7E+09 9.5E+08 4.4E+10 8.1E+09 9.1E+10 1.0E+10 2.5E+10 4.8E+09 3.9E+09 2.9E+09 1.2E+09 1.0E+09 1.4E+11 2.1E+10 1.2E+09 9.5E+08	6.1E+10		
UTAC	Maximum	6.0E+11	1.6E+10	7.3E+10	9.1E+10	1.0E+10	8.0E+10	
	Minimum	3.1E+11	3.8E+09	3.5E+10	ined         WHSC         ETC           1.0E+10         2.2E+09           2.4E+10         3.3E+09           3.7E+09         1.6E+09           4.4E+10         1.8E+10           7.3E+10         2.1E+10           2.4E+10         1.3E+10           2.4E+10         1.3E+10           2.6E+10         5.3E+09           1.4E+11         1.1E+10           2.5E+10         1.0E+09           9.1E+09         2.1E+09           9.1E+10         1.0E+10           2.5E+10         4.3E+09           9.1E+10         1.0E+10           2.5E+10         4.8E+09           2.6E+09         1.8E+09           3.9E+09         2.9E+09           1.2E+09         1.0E+09           1.4E+11         2.1E+10           1.2E+09         1.0E+09           1.4E+11         2.1E+10           1.2E+09         9.5E+08           2.8E+10         6.2E+09           119.2         22.4	3.9E+10		
	Average	1.3E+11	1.7E+09	1.4E+10	2.6E+09	1.8E+09	5.9E+10	
JRC #2	Maximum	2.3E+11	3.1E+09	2.5E+10	3.9E+09	2.9E+09	1.1E+11	
	Minimum	6.3E+10	8.9E+08	7.3E+09	1.2E+09	1.0E+09	2.6E+10	
	All Min	7.3E+11	1.6E+10	7.7E+10	1.4E+11	2.1E+10	3.3E+11	
	All Max	6.3E+10	8.9E+08	7.3E+09	1.2E+09	9.5E+08	2.0E+10	
	Mean of means	3.7E+11	5.3E+09	4.2E+10	2.8E+10	6.2E+09	7.7E+10	
	Range Factor	11.6	18.4	10.6	119.2	22.4	16.4	

 Table 11

 Maximum and minimum ranges for PFDS-measured PN

171. Hot WHTC results ranged from  $< 10^{9}$ /kWh (JRC 2nd campaign) up to  $\sim 1.6 \times 10^{10}$ /kWh, a difference from low to high of less than 20 times. The all laboratories mean was  $\sim 5 \times 10^{9}$ /kWh, almost 2 orders of magnitude lower than the cold start emissions.

172. The low emissions levels measured from PFDS during the hot WHTC cycle resulted in a substantial reduction in the weighted WHTC result relative to the cold start cycle. Emission ranged from ~7 x 10<sup>9</sup>/kWh to ~8 x 10<sup>10</sup>/kWh with the all laboratories mean at ~4.2 x 10<sup>10</sup>.

173. Emissions from the WHSC showed the greatest range, a factor of ~120, which reflects the substantial period of operation spent in passive regeneration during this cycle and the influence that has on DPF fill-state and filtration efficiency. Low-end emissions levels were close to  $10^9$ /kWh increasing to  $1.4 \times 10^{11}$ /kWh.

174. The range and absolute emissions levels from the ETC were similar to those seen from the hot WHTC:  $\sim 10^{9}$ /kWh up to  $\sim 2 \times 10^{10}$ /kWh with the all laboratories mean at  $\sim 6 \times 10^{9}$ /kWh.

175. ESC data from Ricardo were highest of all the laboratories, but not identified as an outlier. Emissions ranged from  $\sim 2 \times 10^{10}$  to  $\sim 3 \times 10^{11}$ /kWh with the all laboratories mean at just below  $8 \times 10^{10}$ /kWh.

# Figure 49 Maximum and minimum ranges for PFDS-measured PN



176. Figure 50 shows the mean of all laboratories PN emissions from PFDS systems. Data are shown for all emissions cycles.

Figure 50



177. <u>PN Emissions Levels Overview</u>: In both CVS and PFDS cases, and considering mean of means data, emissions were highest from the cold start WHTC at  $\sim 4 \times 10^{11}$ /kWh. At this level of emissions, contributions from the tunnel background, even from laboratories with very high tunnel backgrounds, do not have a substantial impact on emissions.

178. Lowest emissions were observed from hot cycles which do not have substantial periods of passive regeneration: hot WHTC and ETC. These cycles showed emissions levels of  $5-6 \ge 10^9$ /kWh from the PFDS and  $8-9 \ge 10^9$ /kWh from the CVS once outlier laboratories were excluded. Laboratories considered to be outliers reported emissions levels from these cycles substantially above  $10^{11}$ /kWh.

179. Weighted WHTC results were of the order 4–5 x  $10^{10}$ /kWh from the PFDS and CVS (outliers excluded) and ~ $10^{11}$ /kWh from the CVS when all laboratories data were considered.

180. ESC and WHSC cycles results were generally more variable than the hot start ETC and WHTC due to the presence of passive regeneration during these cycles. Passive regeneration may reduce filtration efficiency by reducing or removing the filter cake, but high temperatures may also liberate low volatility HCs which contribute to solid particles: both of these facts result in higher PN emissions from the ESC and WHSC than from the ETC and hot WHTC. The ESC cycle, which has a 2 minutes period of operation at full load, and other modes with very high exhaust temperatures, sees a higher contribution of low volatility HC 'solid particles' than the lower temperature WHSC. Emissions from the WHSC were around 2–3 x  $10^{10}$ /kWh from PFDS and CVS (outliers excluded) and 6–8 x  $10^{10}$ /kWh from the ESC with outliers excluded from the CVS data.

181. <u>Euro VI Certification</u>: Certification testing for Euro VI will include both WHTC and WHSC. From the emissions levels seen in this study a limit set

mandating PFDS and excluding CVS could be lower than a limit set that permits either dilution system. If either dilution system is permitted, the test laboratory or OEM would face substantially higher risk of non-compliance when using a full flow system, unless that CVS was well characterised beforehand.

182. Achievable PN Limit for this Engine: Across this test programme the highest single PN emissions result seen from any cycle across all laboratories was 7.4 x  $10^{11}$ /kWh from a cold WHTC. If this engine and its DPF are considered to have representative PN emissions and both CVS and PFDS dilution approaches are used, a PN limit of 8 x  $10^{11}$ /kWh would be achievable for all emissions cycles tested.

## C. Gaseous emissions

183. <u>General Observations</u>: Gaseous regulated emissions results were generally more repeatable when measured directly from raw exhaust than when measured as dilute emissions. Substantial differences in raw vs. dilute emissions levels also exist, even for 'high emissions' gases such as CO<sub>2</sub>. Evaluations of these differences were not the focus of this programme, but data from this work will be made available for consideration in relevant studies.

184. Some laboratories had high levels of CO and HC backgrounds even when these emissions were measured from raw exhaust. This presents a challenge for the measurement of, in particular, carbon monoxide and hydrocarbons at low levels.

185. It is clear that few laboratories regularly measure 'bagged emissions' from HD testing. This approach of accumulating a dilute sample permits a true, simultaneously sampled, background to be quantified and subtracted. Even so, HC and CO emissions are so low from modern engines that even this approach, since it samples from diluted exhaust, may lack accuracy.

186. <u>Inter-Laboratory Variability</u>: During the ILCE\_LD [<sup>31</sup>], the inter-laboratory variations of regulated gases over the cold start NEDC were found to be  $\leq$  5 per cent for CO<sub>2</sub>, 35–50 per cent for HC and 10–30 per cent for NOx and > 40 per cent for CO.

187. Considering raw emissions measurements during the ILCE\_HD, inter-lab variations were at broadly similar levels from the Cursor 8 engine, with some emissions cycles showing lower variations than others. Highest variations were seen from CO and HC, where actual g/kWh emissions levels were low. Ranges of variation encompassing all test cycles are given below:

- (a) CO<sub>2</sub> emissions across the laboratories: inter-lab variation range (all test cycles) 6 to 13 per cent.
- (b) CO emissions across the laboratories: inter-lab variation range (all test cycles) 63 to 91 per cent. CO emissions range from 13 to 400 mg/kWh.
- (c) NOx emissions differences across the laboratories: inter-lab variation range (all test cycles) 26 to 38 per cent.
- (d) HC emissions differences across the laboratories: inter-lab variation range (all test cycles) 68 to 82 per cent. Emissions range from 6 to 120 mg/kWh.
- (e) Cycle work differences across the laboratories: inter-lab variation range (all test cycles) 2.5 to 12.8 per cent.

188. The reproducibility levels observed in this work were consistent with expected levels. Background contributions to CO and HC measurements influenced the variability of these two gases, despite measurements of raw exhaust

189. Other factors contributing to inter-laboratory variability: some differences in dynamometer settings and exact configurations of how each test bed drives a particular emissions cycle will contribute, and another source of variation will be the difference between analysers from different manufacturers and the different technology levels of analysers used by different laboratories.

190. Overall the gaseous emissions data from this work were considered valid for comparative purposes.

## D. Long-term trends in engine operation

191. <u>Regulated Gases</u>: There were no obvious progressive trends in gaseous emissions across the test programme e.g.  $CO_2$  (Figure 51) and NOx (Figure 52), rather there were some step changes between laboratories that indicate true lab-to-lab variations. Gaseous emissions data from JRC was generally similar between the two measurement campaigns: which demonstrates an absence of drift during the programme.



Figure 51 CO<sub>2</sub> – Consistency across the validation exercise



192. <u>Particle Number Emissions</u>: The programme-wide emissions of particle number measured from the CVS over the cold start WHTC cycle are shown in Figure 53. Similar levels of solid particle number emission rates were determined from all laboratories, with the exception being the final tests conducted at JRC. The levels measured during the second (and last) measurement campaign at JRC were systematically lower. This discrepancy holds for all test cycles and for both CVS and PFDS-measured particle number emissions.



PN from cold-WHTC: Consistency through the test programme



193. A comparison of the real-time particle number emission rates measured during the first (Figure 54) and second campaigns (Figure 55) at JRC suggests that an apparent increase of the filtration efficiency of the DPF filter was seen during the second measurement campaign. Results in the first phase were typically in the range 3 x  $10^{11}$  to 5 x  $10^{11}$ /kWh, while emissions in the second phase dropped to between 1 x  $10^{11}$  and 3 x  $10^{11}$ /kWh. An examination of the back pressure and temperature data revealed only marginal differences.

194. In order to further investigate this issue, the entire daily test protocol was repeated twice after an extended preconditioning consisting of 2 hours operation at ESC mode #10. The intention of this exercise was to passively regenerate the DPF to ensure a complete purge of soot and then, without adding any additional soot, run through the daily matrix.

195. Emissions levels over the cold WHTC from the two days' testing were unchanged after this extended conditioning of the DPF, showing emissions levels of  $\sim 2 \times 10^{11}$ /kWh – in the middle of the band of emissions levels seen in the second formal test phase.

196. If the engine-out PM had increased, this might have led to a more rapid generation of a filter cake, reducing particle numbers. However, since PN was still reduced relative to the first JRC measurement campaign following an extensive passive regeneration, this can be discounted. Consequently there are three possible explanations for the reductions in PN seen.

- (a) Reduction in engine-out PN leading to lower post DPF emissions
- (b) Change in the performance of the SPCS or CVS
- (c) Increase in the filtration efficiency of the DPF leading to lower post-DPF emissions

197. From cold start cycles, PN emissions are generally associated with elemental carbon, which in turn relates to PM. Therefore, in order for engine-out PN to reduce between the two measurement campaigns at JRC, engine-out PM should also reduce. Since NOx and PN trade-off, any reduction in engine-out PN would be accompanied by an increase in engine-out NOx. In fact, NOx is generally lower from tests in the second measurement campaign at JRC. As a consequence, it is unlikely that a reduction in engine-out PN is due to a change in the engine-out emissions.

198. Emissions levels from the PFDS and CVS correlated equally well from the two JRC measurement campaigns. This eliminates issues from the CVS and SPCS as responsible for the change in emissions levels observed.

199. Consequently, the most likely explanation is a change in the filtration performance of the DPF. Soot has been ruled out as the key factor, so it is possible that the ash level in the DPF reached a critical point during preconditioning at JRC prior to the test programme and this resulted in a step change in filtration efficiency.



Figure 54 Real time PN production from the CVS- Cold WHTC (JRC#1)

Figure 55 Real time PN production from the CVS- Cold WHTC (JRC#2)



### VI. Additional Testwork Overview

### A. Mass contributed by EC and by particles

200. During some of the tests conducted in the second measurement campaign in JRC, an AVL 483 [ $^{32}$ ] soot sensor was employed to determine the mass of soot emitted and a Dekati Mass Monitor [ $^{33}$ ] was used to measure the mass of aerosol.

201. The Soot Sensor sampled directly from raw exhaust at a constant dilution ratio of about 2, while the DMM sampled from the CVS tunnel via a Dekati thermodenuder operating at 300 °C. In parallel for these tests, PM was collected on TX40 filters.

202. The mass measured as soot (AVL 483) and contributed by solid particles (DMM) was only a small portion of that determined gravimetrically. Table 12 summarizes the average and the standard deviation of these fractions for the different test cycles while Figure 56 and Figure 57 indicate the percentage contributions of solid particles and soot respectively to measured PM.

203. Over the cold start WHTC, both AVL483 and DMM measured mass concentrations  $\sim 10$  per cent of those determined gravimetrically. This fraction is similar to that determined for cold NEDCs during the light duty inter-laboratory correlation exercise.

204. During hot start test cycles, the DMM signal was at the zero levels of the instrument which is around  $300 \text{ #/cm}^3$ . Consequently, mass estimates from these cycles constitute a maximum level. Concurrently measured SPCS data verifies that the PMP particle number concentrations were also below  $300 \text{ #/cm}^3$  in the CVS. Even though DMM data will have overestimated the contribution of solid particles to PM from hot cycles, the calculated mass emission rates were still only ~0.5 per cent of that determined gravimetrically.

205. The mass concentrations determined with the AVL483 suggest a 7 to 19 per cent soot content in the PM collected on filters from hot start cycles. These figures suggest higher soot fractions than seen from the cold start tests, an observation which is not substantiated by any other measurement method. These high results might indicate that measurements were made below the sensitivity levels of the AVL483 ~5  $\mu$ g/m<sup>3</sup>, but since the concentration range of emissions was 5  $\mu$ g to 125  $\mu$ g/m<sup>3</sup> this is unlikely. It is also possible that there was interference from gaseous components in the AVL483 signal.

 Table 12

 Contribution of soot and solid airborne particles on the PM collected on filters as determined by the soot sensor and the DMM, respectively

	WHTC Cold	WHTC Hot	WHSC	ETC	ESC
	[in per cent]	[in per cent]	[in per cent]	[in per cent]	[in per cent]
Soot content (CVS)	$11.2 \pm (3.81)$	$7.31 \pm (1.13)$	$18.81 \pm (4.79)$	$15.52 \pm (3.03)$	$14.89 \pm (3.39)$
Soot content (PFDS)	$12.33 \pm (2.29)$	$9.53 \pm (1.5)$	$15.2 \pm (1.26)$	$15.76 \pm (1.28)$	$10.22 \pm (1.01)$
Airborne mass (DMM)	$10.4 \pm (3.52)$	$1.38 \pm (1.05)$	$0.93 \pm (0.65)$	$0.54 \pm (0.2)$	$1.51 \pm (1.22)$


206. As Figure 56 shows, in the Cold WHTC, 4 to 20 per cent of PM is contributed by solid particles. These are probably carbonaceous particles. A possible contributing mechanism is through 'blow-off' contributions as seen in the ILCE\_LD during the first 200s of the NEDC [ $^{5}$ ], or transient reduction in filtration efficiency related to filter-cake cracking [ $^{37}$ ].

207. Other cycle emissions show the contribution of mass from particles as  $\le 0.5$  per cent, so in the majority of cycles tested,  $\ge 99.5$  per cent of PM mass from hot start cycles is from volatile particles or gases.

Figure 57 Fraction of mass contributed by EC



208. The elemental carbon fraction of PM (Figure 57) determined by the AVL soot sensor from the Cold WHTC was  $\sim 10$  per cent. This is consistent with the solid particle mass determined by the DMM.

209. Other cycles PM, measured by the AVL soot sensor, comprise on average  $\sim 17$  per cent EC. This is inconsistent with DMM data and may suggest an interference in the soot sensor, at very low mass levels, that reports a gaseous compound as elemental carbon.

210. Observations in this work regarding the contribution of EC and particles to PM are consistent with the findings of the light duty ILCE: the majority of PM mass is not contributed by either solid (including EC) or volatile particles.

## **B** Further filter media evaluations

211. In the second measurement phase conducted at JRC, following completion of the validation exercise, the daily test protocol was repeated three times employing TEFLO filters. Sampling was undertaken from both the CVS secondary tunnel and from the PFDS.

212. The PM results collected during these three sets of tests have been compared to the data from the main measuring campaign (JRC#2) where TX40 filters were used.

213. In both CVS (Figure 58) and PFDS testing (Figure 59) the levels determined using TEFLO filters were systematically lower than results using TX40 filters.

214. Mass emissions were on average 63 per cent (ETC) to 81 per cent (ESC) lower from TEFLO filters than from TX40 filters when samples were taken from the CVS - secondary tunnel. Similar results, showing a range of 31 per cent (WHTC cold) to 88 per cent (ETC) reductions were observed from PFDS samples' results.

215. Particle number emissions recorded with the two golden SPCS instruments (from primary CVS and PFDS) during these tests were found to lie within the range of values recorded during previous TX40 filter sampling. Therefore, the observed difference between TEFLO and TX40 results indicates a true directional effect. As TEFLO filters are less prone to volatile adsorption artefacts , these results suggest that a significant amount of the mass collected on the TX40 filters results from adsorption of gaseous compounds – either from tunnel background or from exhaust emissions - and that the TEFLO filters collect less of this artefact. However, the levels of PM observed even on the TEFLO filters are substantially higher than the mass attributed to solid particles (Chapter VII, Section A), so it is likely that this filter medium still collects some volatile or semi-volatile material.



Figure 59 PM results obtained using TX40 and TEFLO filters – JRC PFDS



# C. Particle Number measurements from PFDS at constant dilution factors

216. There is a possibility that particle number emissions from DPF equipped engines may, especially when the DPF is highly loaded, be delayed in reaching the exhaust due to transit through the filter substrate and filter cake. If this is the case, the dilution ratio in the PFDS, which varies with engine exhaust flow could be incorrect at the time (following transit delay) the particles and PM actually reach the dilution tunnel. If this is the case, there is actually no merit, specifically for post-DPF PN tests, in undertaking proportional sampling. Consequently, a fixed dilution ratio PFDS measurement combined with a real-time exhaust flow measurement would be an inexpensive alternative.

217. In order to investigate the possibility of employing a simpler partial flow system operating at constant dilution ratio, the test protocol was repeated twice at JRC employing constant dilution ratios of 15 and 4, respectively, in the partial flow system (AVL SPC-472 Smart Sampler). These two values correspond to the average and minimum, respectively, dilution ratio in the CVS tunnel over the WHTC cycle. Unfortunately, due to some problems with the control software, the SPC did not sample correctly in some of the tests, but sufficient data were still acquired to perform a comparison.

218. The use of a constant dilution ratio in the PFDS system does complicate the calculations: the particle number emission rate (number of emitted particles per second) requires a second-by-second multiplication of the particle number signal with the measured exhaust flow rate after careful alignment of these two signals.

219. The percentage difference between the calculated PFDS and CVS results for all valid tests are compared to those determined during the main measurement campaign in Figure 60. This shows percentage differences between the cycle average particle number emissions measured from the CVS tunnel and the partial flow system, when the PFDS sampled at a flow proportional to the exhaust flow rate and also at constant dilution ratios of 15 and 4.



Figure 60

220. The limited data available suggests that PN emission levels can be determined with acceptable accuracy (better than  $\sim$ 15 per cent) when sampling from the exhaust at constant dilution ratio. In general though, this approach seems to systematically underestimate the emission levels measured from the CVS tunnel.

221. The good agreement with the CVS tunnel data is also evident in the real time recordings. As an example, Figure 61 shows the real time particle emission rates over ESC measured from the PFDS running at a constant DR of 4 compared with data from the CVS tunnel. This is the test which gave the largest difference, but still the number concentrations measured from the two SPCS units are very similar, as seen in Figure 62.





PN from proportional and constant dilution sampling compared



222. Since PN levels from a PFDS at constant dilution ratio seem to be similar to PN levels drawn during proportional sampling, it is interesting to assess the impact of these approaches on PM. Figure 63 shows a comparison for one-off cold WHTC tests at dilution ratios of 15 and 4 with cold WHTC data from the CVS and PFDS.



223. The mass measured at fixed DR = 4 is substantially lower than seen from either the partial flow or full flow systems (proportionally sampled), but the mass measured at fixed DR = 15 is similar to the low-end results from the CVS.

224. The indications are that mass is underestimated by the constant dilution approach (and it can't be corrected because there are no real time data). However, the mass emissions from many laboratories appear to be indistinguishable from zero if the background is subtracted. On this basis, mass as a metric is of little value and if particle number was the only metric, the constant DR PFDS approach would be a valid and cost-effective approach.

# VII. Comparisons of Measurement Systems

225. This section discusses the relationships between particle and particulate measurements made from full and partial flow dilution systems. Comparisons are made between mass and number metrics from full and partial flow dilution systems.

# A. Comparison of Particle Number measurements from CVS and PFDS

226. Figure 64 shows the correlation between PN emissions measured simultaneously from the full and partial flow systems at JRC only (lhs) and all laboratories (rhs). Data are shown from all emissions cycles. It's clear from the JRC data that during both measurement campaigns, despite the shift observed in the levels, the same correlation between full and partial flow measured PN was present. This correlation seems to hold over almost 3 orders of magnitude.

227. The background levels in the JRC CVS and partial flow systems are known to be low (Chapter V, Section B) and similar. This was not true at all laboratories,

and the differential between the backgrounds of CVS and PFDS systems at the same laboratories leads to poorer correlations, as the right hand figure shows.

228. The differences in background levels can be compared by considering the lowest recorded cycle emissions from each lab, as these must be higher than, or equal to, the background. Levels are overlaid in Figure 64, with the red line indicating the lowest emission at Ricardo ( $\sim 10^{11}$ /kWh), the orange EMPA's lowest emission ( $\sim 10^{10}$ /kWh), violet the lowest levels at UTAC ( $\sim 7 \times 10^{9}$ /kWh) and green UTAC's results ( $\sim 3 \times 10^{9}$ /kWh). The background at JRC was at or below 10<sup>9</sup>/kWh. In general, above  $\sim 5 \times 10^{11}$ /kWh all laboratories data are broadly similar.

229. These data show that full and partial flow dilution systems can provide almost identical results, but that these will be dependent on the level of background present in the system. From the results of this work, partial flow systems seem to have inherently lower backgrounds than full flow systems.



230. Considering the JRC results in more detail, Figure 64 shows the results of cross-plotting the CVS and PFDS sampled real-time data (> 1000 points) from several emissions cycles. These charts demonstrate that the real time responses of the PFDS and CVS, as well as the cycle averaged data, correlate well. This demonstrates that:

- Low background dilution facilities provide almost identical data, irrespective of whether they are CVS or PFDS;
- (b) That the principal differences between CVS and PFDS, such as a PFDS's flow control and response time, do not significantly impact particle measurements sampled at 1 Hz.

231. Figure 65 also shows that as measured particle numbers reduce, the correlation between PFDS and CVS weakens.



Correlations between real-time PN from CVS and PFDS systems

Figure 65

232. Figure 66 illustrates the percentage change in PN emissions from the partial low system relative to the levels measured from the CVS. These are calculated as follows: ((#/km PFDS - #/km CVS) / (#/km CVS)) x 100

233. In this comparison, zero emissions from the PFDS represents -100 per cent difference between systems, the same emissions from the two systems represents zero per cent difference and higher emissions levels from the PFDS appear as +ve per cent differences. With the exception of JRC, which shows a range from -12 to  $\sim 0$  per cent from  $\sim 10^{9}$ /kWh right up to  $10^{12}$ /kWh, and Ricardo, which has a best result of ~40 per cent at  $> 10^{11}$ /kWh, all laboratories results were better than -20 per cent at emissions levels above 10<sup>11</sup>/kWh.



Figure 66 Percentage differences in PFDS emissions compared to CVS – All Laboratories

# B. Comparison of Particulate Mass systems

234. Figure 67 shows the correlation between PM emissions measured simultaneously from the full and partial flow systems at all laboratories (lhs) and all laboratories except Ricardo and EMPA (rhs).

235. In general, PM results from all cycles were between 1 and 10 mg/kWh, except in laboratories with the highest background particle numbers (EMPA and Ricardo, Chapter VIII, Section A) where emissions as high as 18 mg/kWh were observed.

236. Considering only the laboratories with lower backgrounds (Figure 67 rhs), PM levels were broadly similar if not correlated, and in the range 1 mg/kWh to 9 mg/kWh. UTAC's results tended to be towards the high end from both the CVS and PFDS and reasonably well correlated, while JRCs results were generally lower.

237. Overall, both CVS and PFDS are capable of measuring PM emissions at levels below 10 mg/kWh, but as with particle number, background contributions are important and should be minimised.

238. It is worth noting that additional experiments at JRC with TEFLO filters and real-time instruments characterising the contributions of solids and volatiles to PM, revealed that < 10 per cent of the PM mass can be attributed to non-volatile materials from cold start tests and that as much as 99.5 per cent of PM from hot start tests is volatile material. As a consequence, the PM method is quantifying dilution system volatiles — whether these come from the engine or not — rather than particulate emissions from the engine.



239. Figure 68 illustrates the percentage changes in PM emissions from the partial flow system relative to the levels measured from the CVS. These are calculated as follows: ((#/km PFDS - #/km CVS) / (#/km CVS)) x 100

240. In this comparison, zero emissions from the PFDS represents -100 per cent difference between systems, the same emissions from the two systems represents zero per cent difference and higher emissions levels from the PFDS appear as percentages greater than zero.



Figure 68 PFDS PM results: per cent difference to CVS PM data

241. In general, emissions ranged from 50 per cent higher in the CVS than in the partial flow system to 50 per cent higher in the PFDS than in the CVS. Labs either showed one trend or the other and these results were seen in the mass range from  $\sim$ 2 mg/kWh to  $\sim$ 6 mg/kWh.

242. On average, CVS and PFDS PM levels broadly agree, but so do CVS and PFDS backgrounds (Chapter V, Section A). In addition (with the exception of ESC cycles), filter mass levels on both sample and background filters are the same from many dilution systems at several laboratories. On this basis, the fact that CVS and PFDS agree and they can be repeatable, but results are the same as the background, means that both PM methods may just be reporting background that varies in response to the emissions cycle's pressure and temperature transients. The actual PM emission is effectively zero in many cases.

# C. Mass vs. number full flow

243. In the low tunnel background facilities at JRC, partial flow PN and full flow PN correlated well and agreement between solid particle number emissions from the two dilution systems was very good. A comparison between PN and PM from all CVS systems (Figure 69, lhs) shows that highest number samples do have highest mass, but that this relationship is tenuous and, in any case, non-linear. Comparing just JRC data (Figure 69, rhs) this relationship is not apparent. The apparent tenuous mass vs. number relationship would disappear if PM data was background subtracted (all PM data except ESC reduced to zero)

244. Particle number appears to be much more readily measured with high sensitivity than mass (a factor of > 300 covering the PN emission corresponds to a difference of  $\sim$ 5 mg/kWh):

Considering all laboratories' data (Figure 69, rhs), it appears that either:

(a) Participating laboratories show similar results to JRC – a factor of at least 100 range of PN levels in a narrow band of PM values,

or:

(b) The high tunnel background laboratories show a wider range of PM values confined to a narrower (but still factor of 10 or more) band of PN values.

245. In both cases there is no obvious relationship between number and mass from CVS systems.

#### Figure 69

Relationship between mass and number Measurements (CVS)



## D. Mass vs. number partial flow

246. In the low background facilities at JRC, partial flow PN and full flow PN correlated well and agreement between solid particle number emissions from the two dilution systems was very good. The JRC PFDS was the only system in this project in which PM could be definitively discriminated from the PM background, so if this system shows no relationship between mass and number it is highly unlikely that one exists.

247. Comparison between PN and PM (Figure 70) shows that the highest mass samples do not necessarily correspond to the highest particle number results and that individual laboratories appear to occupy discrete, narrow mass emissions bands while spanning a wide PN range.

- (a) The poor relationship between PFDS mass and PFDS number must be related to a variable volatile contribution to PM or to a variable solid and/or volatile PFDS tunnel background.
- (b) There is no mass vs. number relationship apparent for PFDS (Figure 70): generally PN is sensitive but PM only varies slightly across all test cycles and absolute levels are different from different laboratories.

248. It is possible that a relationship could exist between PFDS PM and PN if an accurate background could be subtracted from all PFDS PM results, but this is not achieved even by the low background JRC PFDS data (Figure 71).







250. These data strongly suggest that either there is no relationship between PM and PN downstream of a highly efficient wall-flow DPF, or that substantial improvements in dilution air and tunnel cleanliness will have to be made in order to make any relationship detectable.

# E. Comparisons of PMP type and PMP like systems

251. PMP type and PMP- Like Systems. In addition to the two GPMS systems used with the SPCS, a number of PMP type systems (which operate according to the principles specified in Regulation No. 83), and PMP like systems (which use different concepts for the dilution and thermal treatment of the aerosol) were evaluated in this work.

- 252. The other PMP type systems employed were:
  - (a) Nanomet by Matter Engineering;
  - (b) Dual ejector plus evaporating tube system by Dekat;
  - (c) Homemade dual ejector (Palas) plus evaporating tube from EMPA;
  - (d) AVL Particle Counter (APC).
- 253. PMP like systems were:
  - (a) Dekati thermodenuder (TD): employed sampling from the CVS tunnel either directly or downstream of a Dekati ejector diluter;
  - (b) Dual ejector (Dekati) plus evaporating tube system developed at EMPA: employed sampling aerosol directly from the tailpipe

254. <u>Particle Number Counters</u>: PMP type and PMP-like systems were tested using particle number counters (PNCs) of different models and from different manufacturers. All were condensation nucleus counters (CNCs) from GRIMM or TSI. TSI models are known as condensation particle counters (CPCs). PNCs used were:

- (a) TSI 3010D
- (b) TSI 3790
- (c) TSI 3010
- (d) Grimm's 5.404

255. TSI 3010 CPCs were operated at a condenser-evaporator temperature difference of 9 °C in order to effectively shift the 50 per cent counting efficiency to 23 nm as required by the legislation. Grimm's CPC also operated in PMP mode by appropriate modification of the instrument firmware. It should be stressed though that this particular CPC did not fulfil the requirements of maximum allowable coincidence correction and operation in full flow mode (issues resolved in model 5.430 of Grimm). TSI 3010D and TSI 3790 CPCs are supplied by the manufacturer to be compliant with PMP requirements.

256. Particle Losses. Particle losses within the measurement systems vary, so in order to make fair comparisons, these need to be taken into account. Of all the PMP type and PMP like systems examined, only AVL's APC units were calibrated by their manufacturer for particle losses. AVL incorporate this correction in the reported particle concentration results.

257. To enable particle penetrations through the two golden SPCS units, two of the three different Nanomet systems, Dekati's ejectors and the Dekati TD to be determined, evaluations using NaCl particles were undertaken at JRC before their first measurement campaign. These have been published elsewhere [<sup>35</sup>]. EMPA also calibrated their two homemade systems using NaCl particles. The penetration values determined in these studies, and which were also used for the comparisons shown in this section, are summarized in Table 13.

258. Particle losses inside the Nanomet system tested at Ricardo were not thoroughly investigated. However, preliminary work conducted by AEAT on this system suggested similar losses to those of an identical specification system measured at JRC.

259. The Dekati dual ejector plus evaporating tube system was not calibrated as an entire system, and as a consequence only the particle losses inside the two ejectors can be accounted for in the calculations. Thermophoretic losses in the evaporating tube of this system should be low as the thermally treated sample exiting the evaporating tube is immediately diluted in the second ejector diluter.

	P (30 nm) [in per cent]	P (50 nm) [in per cent]	P (100 nm) [in per cent]	Correction
SPCS	71	83	86	1.25
Nanomet JRC & RCE	68	88	95	1.12*
Nanomet LD GPMS	52	65	90	1.37*
TD	67	73	77	1.38
EMPA's homemade (CVS)	70	71	72	1.41
EMPA's homemade (direct)	61	63	65	1.59
Ejector (heated)	96	98	100	1.02
Ejector (not heated)	100	99	100	1.00

Table 13 Particle penetrations through the various PMP type / like systems used in the PMP HD validation exercise

\* These figures take also into account the penetrations at 80 nm suggested by the manufacturer and included in the reported concentrations. These were indirectly determined by means of dilution factor measurements using gases [<sup>35</sup>].

260. As the different CPC units employed might exhibit different correlations relative to the primary calibration (indicated as the slope of a direct cross-plot between the two) it is also important to account for this difference in the comparisons. Most of the CPCs employed in the study were cross-compared with the golden 3010D PNC employed with the SPCS usually connected to the CVS tunnel in the validation exercise (SPCS19). This was the ideal reference as the slope of this particular unit is 0.99. A direct comparison with this PNC provided the means to determine the slope of the individual CPCs employed with alternative and additional systems, and also with the PNC used with the second SPCS. The results of these comparisons are summarized in Table 14.

#### Table 14

Percentage difference between the different CPCs employed and the	golden	CPC	of SPCS19
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Grimm (JRC)	-5 per cent
TSI's 3010 (JRC)*	-10 per cent
3010D (SPCS-20)	-1 per cent
TSI's 3790	+11 per cent
LD GPMS golden CPC	+5 per cent

\* Operated at a condenser-evaporator temperature difference of 9 °C which effectively resulted in a shift of the 50 per cent counting efficiency to 23 nm.

261. No information is available for the two CPC units employed in EMPA's homemade systems and for the TSI 3010D CPC employed in the Nanomet system tested at Ricardo. Based on the observed differences, an additional  $\pm 10$  per cent uncertainty is to be expected from the results obtained from these systems. The manufacturer's calibration of the APC systems takes the CPC slope into account so there was no need to correct the results of these systems.

# F. Results from golden engine equipped with DPF

262. PMP type systems. Data from all individual tests with other PMP type systems are compared to the GPMS results (SPCS19 or SPCS20 depending on the sampling position of the systems) in Figure 72.



Correlation between the GPMS and other PMP type systems





264. As an example of this effect of elevated system backgrounds, Figure 73 shows a comparison of the real-time particle number concentrations from the SPCS and from a dual ejector plus ET with Grimm CPC. These data are from cold and hot WHTC tests measured from the CVS at JRC.





Comparison of real time PN in the CVS from the GPMS and from a dual ejector plus ET system over Cold and Hot WHTCs

265. It can be seen that the dual ejector system has 20 times higher background levels than the SPCS. However, the levels measured from the two systems are in excellent agreement at levels above the dual ejector system background. Over the cold start WHTC, where the number concentrations are up to three orders of magnitude above the background levels, the cycle average results calculated with the two systems agree to within 1 per cent. From hot start WHTC however, the particle emissions levels are below the dual ejector system background from most of the test cycle, but when the concentrations do rise above the background level of the ejector system, the good agreement is maintained.

266. The cycle average emissions from the dual ejector system over the hot start cycle were more than twice the levels (+111 per cent) of those from the SPCS. The two GPMS units incorporate a much more efficient dilution air filtration system than any alternative systems employed in this study, and therefore the comparisons at the lower range of measured emission rates are affected by this artefact. As a

consequence, only the results obtained over the higher emissions WHTC cold and ESC test cycles are considered in the following analyses, as the emission levels from these were sufficiently high to be relatively unaffected by background particle contributions.

267. Table 15 summarizes the average and standard deviation of the individual percentage differences between the different systems employed and the GPMS. The results obtained with the different PMP type systems over the WHTC cold and the ESC test cycles agreed within  $\pm 30$  per cent and  $\pm 15$  per cent, respectively.

268. All Nanomet systems connected to the CVS tunnel measured systematically higher number concentrations than the GPMS (up to 55 per cent over WHTC Cold and up to 25 per cent over ESC). Both JRC and Ricardo found that the background levels of the Nanomet systems increased during the measurement campaign. This has been attributed to production of wear particles from the diamond-like carbon (DLC) rotating disk of the primary diluter and the manufacturer has developed an alternative disc coating to avoid this. The disc coating deterioration could partly explain the observed overestimation in the particle number emissions determined with those systems. Additionally, the three Nanomet systems employed in the particular study are of an older design that does not take into account the pressure at the sampling point. When the sampling location is from a depression (which is the case in both CVS and PFDS) the indicated dilution ratio may be incorrect and this may also have contributed to the observed overestimation of the particle number emissions. This may also explain the differences observed when the Nanomet was sampled from the CVS and from a PFDS. This discrepancy might also be associated with errors in the setup of the PFDS (e.g. errors associated with the control of the flowrate extracted by the PN measurement system and/or the make up air that compensates for this flow).

269. Particle number results measured by the Dekati dual ejector system were found to be from 0 to 20 per cent lower than the levels seen from the SPCS. This consistent underestimation of the particle number concentrations determined with the Dekati system is probably associated with particle losses inside the evaporating tube (as discussed in Chapter VII, Section E) which have not been accounted for.

270. The results obtained from two APC systems agreed within  $\pm 15$  per cent with the GPMS ones. This can be considered to represent the uncertainty levels from PN measurement systems produced and calibrated in accordance with the requirements of Regulation No. 83.

271. <u>PMP like systems</u>: The PMP like systems investigated were also found to be in good agreement with the GPMS systems (Figure 74).

272. Those systems utilizing Dekati's TD gave about 10 per cent lower emission levels than SPCS over all test cycles. These particular systems utilize lower or even no dilution and this has the advantage of minimizing the effect of particle background. This explains the consistency of the results obtained over all test cycles.

273. EMPA's system connected directly to the tailpipe also gave comparable results to the SPCS, with the differences being on average 1 and -7 per cent over WHTC cold and ESC, respectively. This is a very interesting finding, as the setup employed simplifies the measurement procedure considerably. Care needs to be taken for the accurate calculation of the true particle emissions, as the measured particle number concentration signals need to be precisely time aligned with the exhaust flow rate traces. Additionally, the dilution ratio of the ejector diluter is known to be strongly affected by pressure and temperature variations at the

sampling point [<sup>36</sup>]. The use of a trace gas for a real time determination of the dilution ratio (as performed by EMPA) can resolve this problem, but this approach usually results in a noisy DR signal which introduces some uncertainty in the exact determination of the cycle average results. These two issues might be responsible for the relatively high variability observed in the results (of the order of  $\pm 30$  per cent).

## Figure 74 Correlation between the GPMS and PMP like systems



## G. Engine out and open flow emission levels

274. A limited number of tests were conducted at JRC in which the DPF was replaced with an EMITEC Partial Flow Deep Bed Filter "Open filter" and from engine-out exhaust. This provided the means of comparing some of the other PMP type and PMP like PN measurement systems at higher particle number emission levels.

275. The results obtained with the other PMP type and the PMP like systems are compared with the GPMS units in Figure 75 and Figure 76, respectively. The average and standard deviations of the individual differences determined are summarized in Table 15.

276. Similar trends were observed at these higher emission levels. In particular, the Nanomet systems were found to systematically overestimate the particle number emissions. The dual ejector and the thermodenuder systems gave slightly lower particle number concentrations ( $\sim$ 10 per cent). This consistency in the results over a 4 orders of magnitude variation of the vehicle emissions suggests that the observed differences are associated with errors in the calibration of the systems.



Figure 75 Correlation between the GPMS and other PMP type systems at engine out emission levels

Figure 76 Correlation between the GPMS and PMP like systems at engine out emission levels



## Table 15

Percentage differences in the number concentrations measured with the GPMS, PMP type and PMP-like systems

	0		, <b>, , , , , , , , , , , , , , , , , , </b>	· · · ·		
		WHTC Cold	WHTC Hot	WHSC	ETC	ESC
	Golden engine (with CRT)					
	Nanomet (3010D) JRC	21.87% ± 6.58% (8)	20.73% ± 14.93% (9)	$7.97\% \pm 8.48\%$ (8)	8.59% ± 6.70% (8)	4.46% ± 2.35% (5)
	Nanomet (3010_23nm) JRC	55.67% ± 6.99% (3)	25.29% ± 2.85% (3)	7.2% (1)	0.78% ± 3.66% (2)	-1.40% ± 1.68% (2)
	Nanomet (3010D) Ricardo	21.97% ± 35.77% (4)				
	2xEj+ET (3010_23nm) JRC	$-19.09\% \pm 4.26\%$ (5)	162.99% ± 55.73% (9)	13.43% ± 19.3% (7)	16.62% ± 23.58% (11)	-16.26% ± 9.96% (10)
	2xEj+ET (3790) JRC	-19.37% ± 2.97% (8)	20.81% ± 19.52% (10)	-6% ± 12.37% (8)	-5.46% ± 8.89% (8)	-16.69% ± 5.49% (8)
	2xEj+ET (Grimm) JRC	$-0.03\% \pm 3.7\%$ (9)	118.08% ± 60.64% (10)	70.17% ± 44.62% (9)	74.52% ± 44.06% (9)	$1.54\% \pm 2.33\%$ (11)
/pe	2xEj+ET (3010_23nm) EMPA	7.42% ± 24.62% (8)	-14.28% ± 9.83% (8)	-11.29% ± 10.58% (8)	-19.68% ± 11.09% (8)	-6.46% ± 22.29% (8)
PMP ty	APC AVL MTC	14.99% ± 4.06% (3)	4.87% ± 1.39% (2)	2.13% ± 5.8% (2)	0.67% ± 7.79% (2)	6.32% ± 1.19% (2)
	APC JRC	-16.22% ± 17.77% (6)	49.15% ± 30.68% (5)	21.7% ± 25.93% (5)	30.24% ± 33.74% (4)	-15.79% ± 17.03% (7)
	Nanomet (3790) JRC - PFDS	34.50% ± 18.25% (4)	-11.99% ± 11.49% (5)	0.62% ± 29.45% (4)	-33.73% ± 22.88% (4)	24.90% ± 24.24% (4)
	Golden engine (without CRT)					
	Nanomet LD GPMS (3010D) JRC - PFDS	39.83% ± 15.41% (3)	25.34% ± 10.40% (2)	37.08% ± 3.57% (2)	29.13% ± 1.02% (2)	39.56% ± 1.25% (2)
	Nanomet (3790) JRC - PFDS	52.99% (1)	59.14% (1)	16. 38% ± 30.26% (2)	-4.98% (1)	-9.65% (1)
	2xEj+ET (3790) JRC	-11.45% ± 5.3% (4)	-1.51% ± 10.82% (4)	$-6.23\% \pm 16.68\%$ (3)	-14.62% ± 14.92% (3)	-13.24% ± 12.58% (2)
	2xEj+ET (3010_23nm) JRC	-11.53% ± 12.24% (3)	-14.2% ± 10.49% (3)	-6.08% ± 8.83% (3)	-14.36% ± 4.21% (3)	-12.57% ± 3.95% (3)
	Golden engine (with CRT)					
IP like	2xEj+ET (3010_23nm) EMPA - Direct	0.56% ± 30.42% (7)	-39.31% ± 29.45% (7)	-13.14% ± 32.67% (8)	-45.34% ± 16.26% (6)	-7.67% ± 35.65% (8)
	Ej+TD (3790) JRC	$-12.42\% \pm 3.01\%$ (7)	-8.31% ± 11.88% (9)	-10.2% ± 8.25% (8)	-15.41% ± 8.29% (10)	-13.33% ± 4.2% (10)
	TD (3790) JRC	-25.58% ± 10.94% (2)	-3.38% ± 20.27% (2)	-4.42% ± 4.38% (2)	-3.73% ± 17.28% (2)	-8.29% ± 1.14% (2)
PN	Golden engine (without CRT)					
	Ej+TD (3790) JRC	$-4.\overline{65\% \pm 7.93\%}$ (4)	$4.54\% \pm 9.26\%$ (4)	$-1.33\% \pm 7.44\%$ (4)	$-24.59\% \pm 17.75\%$ (4)	-25.33% ± 16.58% (5)
	Ej+TD (3010D) JRC	$-12.36\% \pm 5.39\%$ (2)	-21.37% ± 23.21% (2)	-14.44% ± 13.75% (2)	-19.68% ± 12.12% (2)	

The percentage values correspond to the average  $\pm 1$  standard deviation of the individual test differences while the numbers in parentheses indicate the number of tests from which these figures were calculated. Results have been corrected for PCRF values for all systems except for the Dekati's 2xEj+ET (shown in red in the table) in which case this information is not available.

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## H. Gaseous Emissions

277. Measurements of gaseous emissions taken directly from raw exhaust appeared to be more repeatable than emissions measured from diluted sources. This included both bagged and continuous dilute measurements, though relatively few laboratories now seem to use the cumulative bagged approach.

278. There were substantial differences in raw vs. dilute emissions even for 'high emissions' gases such as  $CO_2$ . In addition, some laboratories experienced high levels of CO and HC in their background air and this contributed to observed variability.

279. Inter-laboratory variation levels were at similar levels for NOx and  $CO_2$  as seen with the golden vehicle in the light duty PMP exercise. HC and CO emissions levels were slightly more variable, possibly due to contributions of these gases in the background. Overall the data set was considered to be representative of expected lab-to-lab variation levels.

# VIII. Discussion and Overview

280. This section draws together the discussion points from the report and summarises the critical issues for use of the measurement equipment and procedures in legislative procedures.

# A. Particulate Measurements

281. <u>Validation Exercises</u>: Reference filter variation was higher than seen in previous test programmes including the ILCE\_LD, despite stable environmental conditions. Occasionally one filter showed considerably higher variation than the other two, which indicates that baseline differences in filter background rather than environmental changes may be responsible for excessive reference filter variability. If the batch of filters used in this work is representative of all TX40 batches, this confirms that the (larger) permitted reference filter variance of  $\pm 10 \ \mu g$  as specified by gtr No. 4 is appropriate.

282. <u>Tunnel Background Mass Levels – CVS</u>: Tunnel background PM measurements were made in 3 of the 5 test laboratories. In some laboratories PM backgrounds from CVS systems were extremely high – due to the testing history of these systems. Active regeneration and testing fuels with high levels of FAME seem to leave residual carbon and semi-volatile materials in the dilution system that are not readily removable, even by the 2 hour full load operation undertaken by all laboratories before the start of the measurement campaign. These materials do, however, seem to release over time. This suggests laboratories who are testing both high and low emission engines with the same dilution systems may need to take special care in planning their test schedules and in dilution tunnel pre-conditioning, cleaning and maintenance in order to minimise tunnel background contributions.

283. In other laboratories, CVS backgrounds were much lower. However, in this programme, where tunnel background particulate mass from CVS systems was measured, tunnel background filter masses were always equivalent to sample filter masses, with the exception of samples from the ESC which were always higher than the background.

284. The ESC cycle has a substantial period of operation at high exhaust temperatures and this may lead to emissions of low volatility compounds that are efficiently collected and then retained by the filter. Filters from other cycles collect higher volatility materials from the exhaust and dilution air, but these can be released

following acquisition through volatilisation or through a washing effect as further aerosol is drawn through the filter.

285. <u>Tunnel Background Mass Levels</u> – PFDS: Tunnel background PM levels from PFDSs were, at all laboratories, at the low end of levels from CVS systems. Despite the generally lower tunnel background levels, 2 of the 3 laboratories' partial flow systems tunnel backgrounds were also similar to sample levels, except for the ESC cycle.

286. The third lab, JRC, showed similar tunnel background levels to the other laboratories but slightly higher emissions levels, making it just possible to discriminate samples from tunnel background on all emissions cycles.

287. Since the PFDSs used in this programme were newer than the CVS systems used, they have seen less usage with older, sootier engines. CVS systems may have substantial levels of elemental carbon accumulated on the walls of the dilution tunnel over many years. These dilution tunnels tend not to be cleaned. This carbon may capture volatile materials and further soot more efficiently than the cleaner walls of a newer partial flow system. Subsequent release of these materials will contribute a high and variable CVS tunnel background, while the relatively clean PFDSs had a much lower, more consistent tunnel background.

288. Most PFDS systems have removable dilution tunnels which can be easily cleaned and replaced, further reducing background contributions.

289. <u>Emissions Levels</u>: After exclusion of the results from the two test laboratories that had very high PM tunnel backgrounds as outliers, PM levels measured directly from CVS systems were < 6 mg/kWh from all test cycles, with no obvious difference in emissions between the cold and hot start WHTC cycles. These levels are substantially below the 10 mg/kWh limits set for Euro VI.

290. PM emissions from PFDSs were generally lower than the CVS levels and less than 4 mg/kWh from all cycles except at one lab where emissions from all cycles filled a narrow mass band between 4 mg/kWh and 7 mg/kWh.

291. Subtraction of the low tunnel background JRC PFDS results would reduce the emissions levels from cold start WHTC to (generally) < 1 mg/kWh, hot start WHTC to 0.5 mg/kWh or less, WHSC to  $\sim 1.2 \text{ mg/kWh}$ , ETC to 0.5 mg/kWh or less and ESC to < 2 mg/kWh. The weighted WHTC result would be substantially below 1 mg/kWh.

292. <u>Tunnel Background Subtraction</u>: Due to the high variation in tunnel background masses from both CVS and most partial flow systems, subtraction of tunnel backgrounds would be likely to result in increased variation in corrected PM mass emissions compared with uncorrected results. Nevertheless, PM emissions measured in this programme, using current dilution systems that meet all the regulatory criteria, were in many cases indistinguishable from the background.

293. <u>Partial Flow or Full Dilution</u>: The results of this work indicate that partial flow dilution systems may be preferable to CVS systems for PM measurements. This is purely a consequence of the lower backgrounds present in these systems, their greater ease of cleaning, and the fact that at one laboratory at least, it was possible to discriminate emissions levels from background levels for all cycles tested. As discussed in Chapter VIII, Section A, laboratories using CVS systems for Euro VI type approval testing may need to carefully manage tunnel background levels in order to minimise the risk of background levels resulting in erroneous fail results.

## **B.** Particle Number Measurements

294. <u>Golden systems – PCRF correction</u>: The SPCS systems used throughout this work were not subjected to a manufacturer's PCRF calibration prior to the commencement of the test programme. Subsequent work conducted by JRC indicates that applying a Particle Concentration Reduction Factor (PCRF) correction to the particle number data from these two systems would increase the measured emissions by ~25 per cent. This incremental factor should be considered if results from this work are to be compared with emissions measured by fully PMP type systems in other studies.

295. <u>Particle Number tunnel backgrounds</u>: The high levels of PM tunnel background seen in two laboratories was also reflected as high PN tunnel backgrounds, meaning that there must have been a substantial contribution of either EC or low volatility HCs, or both.

296. At the lab with the highest tunnel background levels, these were the equivalent of  $\sim 4 \times 10^{10}$ /kWh over the ETC. In comparison, two other laboratories had CVS tunnel backgrounds that were 60 and 120 times lower.

297. Tunnel backgrounds from PFDS systems were both lower and much more consistent: three laboratories showed levels equivalent to ETC emissions of between  $2.7 \times 10^8$ /kWh and  $2.9 \times 10^8$ /kWh. This close agreement in solid particle backgrounds from PFDSs indicates that the greater variation in PM tunnel backgrounds from PFDSs must be due to volatiles.

298. <u>Real-time particle emissions elevated at cold start</u>: Emissions from the cold start WHTC were dominated by the first 700 s of the cycle, where particle numbers were several orders of magnitude higher than in the remaining 1100 s. This cold start effect reflects the observations made in the light duty PMP validation exercise. It has been hypothesised [<sup>37</sup>] that these high emissions following cold start are due to reduced filtration efficiency that occurs when the filter cake cracks as it dries and cools following high temperature operation. Particles follow the cracks and escape through the filter substrate until the cracks fill with freshly emitted soot and the filter cake regains its integrity. Emissions from the hot start WHTC show lower emissions from the start of the cycle: as the filter cake is now in place and filtration efficiency is maximised.

299. Transient particle emissions from the WHSC are low from the start but increase after  $\sim$ 1200 s. This may be the point at which on-going passive regeneration within this cycle appreciably reduces the filter cake and it is also where the exhaust temperature is highest. High exhaust temperatures may promote thermal release of low volatility materials which the PN measurement system sees as solid particles.

300. <u>PN Emissions levels compared with backgrounds</u>: Particle number emissions from the cold WHTC were sufficiently high that they were substantially above the tunnel background levels from both CVS and PFDS systems in all the test laboratories. However, CVS tunnel background levels at Ricardo and EMPA were sufficiently high that emissions levels from several cycles (hot WHTC, WHSC, ETC, ESC at Ricardo; hot WHTC, WHSC, ETC at EMPA) could not be discriminated from the tunnel background. These data were excluded as outliers in the statistical analyses.

301. In comparison, tunnel background levels in the partial flow systems were sufficiently low that no laboratories data were identified as outliers based upon high emissions levels attributable to the tunnel background. As with the PM

measurements, it is clear that CVS systems are prone to higher PN tunnel backgrounds than partial flow systems.

302. PN Emissions levels. From both CVS and PFDS cases, mean PN emissions, including all laboratories results, were highest from the cold start WHTC at ~4 x  $10^{11}$ /kWh. At this level of emissions, contributions from the tunnel background, even from laboratories with very high backgrounds, do not have a substantial impact on emissions.

303. Lowest emissions were observed from hot cycles which do not have substantial periods of passive regeneration: hot WHTC and ETC. These cycles showed emissions levels of 5-6 x  $10^9$ /kWh from the PFDS and 8-9 x  $10^9$ /kWh from the CVS once outlier laboratories were excluded. Laboratories considered to be outliers reported emissions levels from these cycles to be substantially above  $10^{11}$ /kWh.

304. Weighted WHTC results were of the order 4-5 x  $10^{10}$ /kWh from the PFDS and CVS (outliers excluded) and ~ $10^{11}$ /kWh from the CVS when all laboratories data were considered.

305. ESC and WHSC cycles results were generally more variable than the hot start ETC and WHTC due to the presence of passive regeneration during these cycles. Passive regeneration may reduce filtration efficiency by reducing or removing the filter cake, but high temperatures may also liberate low volatility HCs, which contribute to solid particles: both of these results in higher PN emissions from the ESC and WHSC than from the ETC and hot WHTC. The ESC cycle, which has a 2 minutes period of operation at full load, and other modes with very high exhaust temperatures, sees a higher contribution of low volatility HC 'solid particles' than the lower temperature WHSC. Emissions from the WHSC were around 2-3 x  $10^{10}$ /kWh from PFDS and CVS (outliers excluded) and 6-8 x  $10^{10}$ /kWh from the ESC with outliers excluded from the CVS data.

## C. Simultaneous particle number and particulate mass measurements

306. For partial flow systems to measure correctly there are two basic requirements:

- (a) Proportionality: The sampled exhaust gas should be a constant ratio of the exhaust gas flow rate
- (b) Accuracy: The value estimated for the sampled exhaust gas should be accurate (i.e. no bias).

307. Proportionality: The quality of the proportionality is checked by applying the regression analysis between sample flow and exhaust flow in accordance with ECE/TRANS/WP.29/2009/114, Annex 4B, paragraph 9.4.6.1. During testing in this programme, there were no proportionality failures.

308. Accuracy: The accuracy of the sample flow is checked according to the requirements of Annex 4B, paragraph 9.4.6.2. For testing in this programme, assuming an average dilution factor of 10, the sample flow was 5 lpm. The extracted flow rate from the golden instrument was 1.3 lpm with a variability/accuracy of <2 per cent. This translates to <0.5 per cent uncertainty for the sampled exhaust gas. For lower dilution ratios this uncertainty is smaller. Although this uncertainty is quite high (0.5 per cent translates to 5 per cent uncertainty in the PN results), there was no indication of significant error for the measurements. Nevertheless it is recommended to decrease this uncertainty to <0.1 per cent by using a digital or analogue signal of the extracted flow rate with a data acquisition rate of at least 1 Hz (and not a constant value) or by feeding back the extracted flow.

309. <u>Required corrections</u>: When particle number and mass are measured simultaneously from partial flow dilution systems, corrections are required to be made for:

- (a) Mass flow removed, which results in an increase in the transfer flow from the raw exhaust. This can be corrected by an automated correction in the instrument software, by physical feedback of the sample flow (if the measurement system allows it) or by a separate feedback of an accurate flow equivalent to that drawn by the measurement system.
- (b) Fractional removal of PM material from upstream of the PM filter, if not replaced by physical feedback of the actual flow taken by the PN measurement system. This can be corrected according to the provisions of ISO 16183 as defined in the appropriate European regulation [<sup>38</sup>].

310. <u>Correction of PM measurement</u>: When a particle number sample flow is extracted from a total sampling partial flow dilution system, the mass of particulates (mPM) calculated in Annex 4B, paragraph 8.4.3.2.1. or 8.4.3.2.2. of ECE/TRANS/WP.29/2009/114 must be corrected as follows to account for the flow extracted. This correction is required even where filtered extracted flow is fed back into the partial flow dilution systems.

$$m_{PM,corr} = m_{PM} \times \frac{m_{sed}}{(m_{sed} - m_{ex})}$$

where:

m <sub>PM,corr</sub>	=	mass of particulates corrected for extraction of particle number sample flow, g/test,
m <sub>PM,corr</sub>	=	mass of particulates corrected for extraction of particle number sample flow, g/test,
m <sub>PM</sub>	=	mass of particulates determined according to Annex 4B paragraph 8.4.3.2.1. or 8.4.3.2.2., g/test,
m <sub>sed</sub>	=	total mass of diluted exhaust gas passing through the dilution tunnel, kg,
m <sub>ex</sub>	=	total mass of diluted exhaust gas extracted from the dilution tunnel for particle number sampling, kg.

## D. Repeatability and reproducibility

311. <u>PM and PN – Repeatability</u>: The repeatability of CVS PM measurements, expressed as CoV, was lowest for the cold WHTC cycles at  $\sim$ 35 per cent with other cycles in the range 50 per cent to 56 per cent. There were no laboratories' data that were considered outliers.

312. PFDS PM measurements showed CoV ranging from 20 per cent to 30 per cent across all cycles, with one lab's data excluded as an outlier by the statistical analysis.

313. By contrast, no laboratories' results were excluded from the PFDS PN data set, but, with CVS measurements, outlier analyses excluded all hot cycle results from Ricardo and all but the cold WHTC and ESC from EMPA.

314. Following the exclusion of outliers, particle number repeatability levels were broadly similar: CVS CoVs ranged from  $\sim 20$  per cent to  $\sim 60$  per cent and PFDS CoVs from  $\sim 20$  per cent to  $\sim 70$  per cent.

315. Focusing on the Euro VI legislative cycles in isolation, shows that the CVS approach has better repeatability over the weighted WHTC (21.1 per cent vs. 22.8 per cent) and over the WHSC (59.2 per cent vs. 74.43 per cent) than the PFDS approach.

316. Taken at face value, and only using the repeatability as the assessment approach, these data might lead one to conclude that PFDS is the favoured dilution system for PM measurements, CVS is the favoured measurement system for PN and PM is the better metric, since it is more repeatable than PN. However, in reality, only the first of these statements is correct. PFDS systems, with their lower backgrounds will produce an emissions result that is lower and therefore closer to the true value.

317. CVS PN results were more repeatable in this programme than PFDS results, but only marginally and after 2 of 5 laboratories were rejected as outliers. CVSs had higher background than the PFDSs, so CVS results were in reality further from the true values.

318. PM measurements in this programme were more repeatable than PN measurements, but again only slightly and, in most cases, PM measurements were indistinguishable from tunnel background, which appears to be at least as repeatable as the emissions from the engine.

319. <u>PM and PN – Reproducibility</u>: CVS PM reproducibility levels were typically in the range 35 per cent to 55 per cent, averaging 42.7 per cent for the 5 emissions cycles in the test matrix. PFDS PM reproducibility levels ranged from  $\sim$ 30 per cent to  $\sim$ 45 per cent, averaging 36.1 per cent. The lower PM CoVs from the PFDS systems probably reflect the greater consistency of background levels in the partial flow system compared to the CVS. Considering just the Euro VI legislative cycles shows that the CVS approach has similar PN reproducibility over the weighted WHTC (41.4 per cent vs. 45.8 per cent) and over the WHSC (81.7 per cent vs. 86.3 per cent) to the PFDS approach. The higher variation in the PN results than the PM results from the WHSC cycle is an indication that the PM method is insensitive to the effects of passive regeneration in this cycle.

320. <u>Repeatability and reproducibility overview</u>: When considering a measurement system it is important to consider more than just the repeatability and reproducibility. The results of this study indicate that the most repeatable and reproducible methods do not necessarily discriminate the emissions from the tunnel background nor determine a true value. However, this work does indicate that PFDS may be preferable for PM determination and that both CVS and PFDS show similar repeatability and reproducibility for the measurement of particle numbers.

### F. Elemental carbon and non-volatile particle contributions to PM

321. Masses collected with TX40 filters were compared with simultaneous mass measurements from real-time instruments. From cold start WHTC cycles, both the Dekati Mass Monitor (which calculates mass from the particle size distribution after evaporating volatile particles) and AVL483 (photoacoustic soot sensor) indicated mass levels ~10 per cent of the filter mass. This suggests that 90 per cent of the filter mass from this cycle is volatile and the other 10 per cent is mostly elemental carbon. At this level of EC content, PM filters appeared slightly grey. From all other (hot start) test cycles the DMM mass was  $\leq 0.5$  per cent of the filter mass, indicating the volatile contribution to PM is ~99.5 per cent. PM filters from hot start tests appeared unused. Clearly, at a maximum of 10 per cent EC in the PM measured on the filter, agreement between mass and number metrics would not be expected.

## G. Filter media effects

322. In experiments at JRC, comparisons were made between PM sampled with 47 mm TX40 and with 47 mm Teflon membrane filters. Collected masses were always lower with Teflo filters, repeating the observations made in the light duty PMP work.

323. Typical masses captured with the Teflon Membrane filters were 30 per cent to 90 per cent lower than recorded with TX40 filters. Considering also the observations in Chapter VIII, Section E, this suggests that even the Teflon filters are still capturing some volatiles.

## H. Other PMP type / like systems

324. VPR systems which were calibrated as a whole unit either by the manufacturer (APC) or in the framework of this study (GPMS, EMPA's homemade system, systems utilizing a thermodenuder), differed by less than  $\pm 15$  per cent when the results were corrected for the average PCRF value as described in Regulation No. 83.

325. The observed differences were not affected by the emitted particle number concentrations (and therefore from the associated uncertainties in the dilution ratio determination) or by the CPC unit employed. Therefore, this is a PCRF related issue. Inaccuracies in the determination of the PCRF values might have contributed to this difference. Most of the instruments used in this study were calibrated against NaCl particles which were not thermally treated. Some preliminary tests suggested a change of structure of NaCl particles when heated which leads to particle shrinkage [<sup>35</sup>].

326. Uncertainties associated with the operation of the DMA used for the production of the monodisperse calibration aerosol might also have contributed in the observed discrepancies. In particular, uncertainties in the charge distribution acquired in the neutralizer and therefore in the contribution of multiply charged (larger) particles in the produced monodisperse aerosol might have affected the calculated PCRF values. The relatively high concentration required for the production of monodisperse aerosol at sufficiently high concentrations might give rise to space charge fields affecting the classified particle size.

327. Another reason for the observed discrepancies between the various systems might be associated with size dependent losses inside the VPR systems. The calculations have been performed using the average PCRF value determined for particle mobility diameters of 30 nm, 50 nm and 100 nm, as suggested by the regulations. The correct approach, however, would be to use the PCRF value corresponding to the geometric mean diameter of the underlying distribution. This simplified approach, which is justified as the true size distributions are not generally known, introduces some error depending on the underlying size distribution of the sampled aerosol, but also on the steepness of the penetration characteristics of the VPR system employed (that is particle losses as a function of particle size). It is worth noting that most of the VPR systems tested exhibited steeper penetration curves than that specified in the regulations. It is therefore important to further investigate the calibration procedure for the VPR systems.

# I. Should solid particles < 23 nm be considered for European PN legislation?

328. Comparisons were made between non-volatile particles > 3nm and non-volatile particles > 23 nm by using different particle counters as the counting elements of the SPCS systems. A number of different steady state and transient (including cold and hot WHTC) cycles were studied.

329. Irrespective of operating conditions, the number of solid particles between 3 nm and 23 nm, measured in the investigations of this programme, was never greater than the level of solid particles above 23 nm. The highest level of particles measured between 3 nm and 23 nm was from the cold WHTC. Levels were ~85 per cent of the number found above 23 nm.

330. No measurements were made of the composition of these 3-23 nm solid particles. At the upper end of the size range primary carbon spheres can be present, however, it is also possible that other solid particles are present. These may derive, for example, from the lubricant as metal oxides. While there was some evidence that solid particles < 23 nm were present, the levels seen were not consistent with the orders of magnitude increases relative to > 23 nm particles reported from US engines. On this basis, it is considered reasonable to retain the size and volatility range of particles measured for light duty vehicles in heavy duty engines' legislation.

## J. Achievable PN limit for this engine

331. Across this test programme the highest single PN emissions result seen from any cycle across all laboratories was 7.4 x  $10^{11}$ /kWh from a cold WHTC. If this engine and its DPF are considered to have representative PN emissions and both CVS and PFDS dilution approaches are used, a PN limit of 8 x  $10^{11}$ /kWh would be achievable from all emissions cycles tested.

332. Alternatively, considering the mean results across all laboratories, the starting point for calculating a PN limit for both WHTC and WHSC is a mean result at or below  $10^{11}$ /kWh.

## K. An alternative approach to sampling for PN measurements

333. There is a possibility that particle number emissions from DPF equipped engines may, especially when the DPF is highly loaded, be delayed in reaching the dilution system due to transit through the filter substrate and filter cake. If this is the case, the dilution ratio in the PFDS, which varies with engine exhaust flow, could be incorrect at the time (following transit delay) the particles and PM actually reach the dilution tunnel. If this is the case, there is actually no merit, specifically for post-DPF PN tests, to undertake proportional sampling. Consequently, a fixed dilution ratio PFDS measurement combined with a real-time exhaust flow measurement has potential to offer an inexpensive alternative approach which may be worthy of further investigation.

334. Only limited tests were undertaken in this programme, but data available suggests that PN emission levels can be determined with acceptable accuracy (better than ~15 per cent) when sampling from the exhaust at constant dilution ratio into a PFDS. In general though, this approach seems to provide directionally lower emission levels than measured from the CVS tunnel.

335. Indications are, that particulate mass is underestimated by the constant dilution approach (and it can't be corrected for real time flow because there are no real time data). However, since particulate mass emissions from many laboratories in this programme appear to be indistinguishable from tunnel background levels this may not be an overriding barrier to this approach.

# IX. Conclusions

## A. Engine operation

336. Engine operation was sufficiently consistent across the test programme to enable comparisons between test laboratories

## **B.** Measurement systems

337. The GPMS systems tested in this work, SPCS systems from Horiba, suffered no significant mechanical failures during the test programme and performed reliably passing all required validation checks in all test laboratories. The two SPCS systems supplied agreed to within  $\sim$ 5 per cent when tested in parallel, enabling direct comparisons to be made when one was used to sample from CVS systems and the other from partial flow systems

## C. PM emissions

338. The PMP mass method collects a large gaseous volatile fraction that may be times the mass of the solid particles collected from the cold WHTC and 200 times the mass of solid particles from hot start cycles.

- 339. Levels:
- (a) PM emissions levels from CVS systems, after exclusion of laboratories with high PM tunnel backgrounds were < 6 mg/kWh from all emissions cycles. However, tunnel background levels from CVS systems in all laboratories were equivalent to drive cycle emissions levels for all cycles except the ESC.
- (b) The chemistry of PM from ESC tests comprises lower volatility HCs which are more effectively retained by the sample filter.
- (c) PM emissions from PFDS systems were generally lower than results from CVS systems – at < 4 mg/kWh from all emissions cycles. From PFDS too, tunnel background levels were similar to or just below sample levels on all cycles except the ESC which could be discriminated from the tunnel background in all laboratories.
- (d) One lab's PFDS was able to discriminate sample levels from tunnel background PM and this revealed all mass emissions to be < 2 mg/kWh. Average emissions, following tunnel background subtraction, were  $\sim 0.6 \text{ mg/kWh}$  from the weighted WHTC, and approximately 1.2 mg/kWh from the WHSC.
- 340. Repeatability / Reproducibility:
- (a) PM repeatability levels from the PFDS were between 20 and 30 per cent for all emissions cycles, while CVS repeatability was best from the cold WHTC (~35 per cent) and 50 to 56 per cent for other cycles.
- (b) Reproducibility levels from the CVS were similar to the repeatability levels, ranging from 35 per cent to 55 per cent. PFDS PM reproducibility levels were slightly better, on average ranging from 35 to 45 per cent.

## D. PN emissions

341. The PMP number method determines real-time emissions that can be related to engine events with high sensitivity.

- 342. <u>Levels</u>:
- (a) PN emissions levels from the Cold WHTC were approximately 4 x 10<sup>11</sup>/kWh from both full and partial flow dilution systems. At these levels of emissions the tunnel background contribution has no substantial impact on emissions.
- (b) Tunnel background PN levels in the CVS were generally higher than in PFDS systems and in two laboratories sufficiently high for those laboratories to be identified as outliers since their tunnel background levels were as high or higher than emissions from many of the hot start cycles. In these cases, emissions from hot start cycles were  $\sim 10^{11}/kWh$ .
- (c) Investigations showed low PN tunnel background levels to be present in several PFDSs, and since very low emissions levels could be measured in all PFDS, it is likely that background levels were low in all PFDS.
- (d) Emissions levels from both PFDS and CVS dilution systems with low PN tunnel backgrounds were 5-9 x  $10^9$  from hot start WHTC and ETC cycles, but higher from WHSC (2-3 x  $10^{10}$ /kWh) and ESC (6-8 x  $10^{10}$ /kWh) where passive regeneration may oxidise the filter cake and reduce the filtration efficiency of the DPF.
- 343. Repeatability / Reproducibility:
- (a) Repeatability levels for the CVS and PFDS were similar, ranging from  $\sim 20$  to  $\sim 70$  per cent, with the cold WHTC most repeatable and the WHSC least repeatable.
- (b) Reproducibility levels between CVS and PFDS systems were also similar at 41-45 per cent for the weighted WHTC and 81–86 per cent for the WHSC.
- (c) With the particle number measurement systems, it is clear that the passive generation present in the WHSC leads to increased variability. This effect is not seen in the PM results.

### E. Relationships between measurement approaches

- 344. Full vs. partial flow mass:
- (a) Emissions levels from PFDS systems were more repeatable and reproducible than measurements from CVS systems.
- (b) One lab's PFDS was able to discriminate mass emissions from tunnel background PM levels. Further research might identify procedures which will enable all laboratories to achieve this with their partial flow systems.
- (c) Mass emissions from PFDS and CVS did not correlate due to variations in tunnel background contributions in this exercise: in most cases this was systems' backgrounds.
- 345. Full vs. partial flow number:
- (a) PFDS systems showed lower tunnel backgrounds than CVS systems, but when CVS system tunnel backgrounds were similar to PFDS tunnel backgrounds the correlation between PN emissions was excellent.
- (b) Higher tunnel background CVS systems still showed similar PN emissions levels to PFDS systems from cold start WHTC tests. However, across other cycles, high CVS tunnel backgrounds weaken the correlation between CVS and PFDS PN measurements as the engine particle number emissions are approach the CVS tunnel background PN level.

- 346. Mass vs. number:
- (a) Mass and number methods did not correlate. The majority of mass comes from volatile materials, which are unrelated to the materials comprising solid particles.

## F. Other PMP type / like systems

- 347. Various PMP type and PMP like systems:
- (a) The majority of the various PMP type and PMP like systems correlated closely with the GPMS, the difference being on average  $\pm 15$  per cent after accounting for the PCRF values and the slopes of the CPCs.
- (b) Systematic differences were observed between the various PMP type PMP like systems employed. These differences hold for emission levels spanning over 4 orders of magnitude as well as when different CPC units are employed. This points towards differences in the calibrations of the PCRF values but could also partly be associated with differences in the penetration curves (penetration as a function of particle size).
- (c) Simplified approaches such as the use of dual ejector systems sampling directly from the tailpipe or operation of the partial flow systems at constant dilution ratios resulted in similar levels of agreement.
- (d) The various PMP type and PMP like systems examined had almost an order of magnitude higher background levels from the GPMS. More efficient conditioning of the dilution air is necessary in order to accurately determine the emission levels over the hot start transient test cycles and the WHSC.

## G. General Conclusions

348. In this work, PM emissions from an engine with an efficient wall-flow DPF measured from both CVS and PFDSs, without any correction for dilution air ground or compensation for tunnel background contribution (which is not permitted in current regulatory procedures e.g. gtr No. 4), were consistently below 10 mg/kWh across all the cycles tested.

349. The results of this work demonstrate that the PM method is suitable to confirm that engine PM emissions levels are below the emissions levels required for Euro VI (10 mg/kWh for the WHTC and WHSC cycles). However, PM measurements in this programme could not generally discriminate between actual emissions levels and dilution tunnel background levels, except in the case of measurements from one PFDS at one lab and for the ESC cycle at all laboratories.

350. In this work, PN emissions from an engine with an efficient wall-flow DPF, measured from both CVS and PFDSs, without any correction for tunnel background, ranged from  $\sim 10^9$ /kWh to  $> 10^{11}$ /kWh across all the cycles tested.

351. Emissions levels from every emissions cycle, with both PFDS and low tunnel background CVS systems, were substantially above tunnel background levels.

352. From all PFDS systems and low tunnel background CVS systems in this work, the PN method is suitable to determine the actual emissions levels from all drive cycles tested, and at levels  $\geq 10^{10}$ /kWh. A threshold of  $10^{10}$ /kWh is quoted, since as shown in Figure 64, the lowest CVS results (at JRC and AVL MTC) were beneath this level and consequently tunnel backgrounds must also have been below this level. This lower emissions threshold accounts for a PCRF correction of 1.25 to PFDS and CVS particle number data from the SPCS systems in this study.

353. Considering WHTC and WHSC results, the PM emissions method may appear more repeatable and reproducible than the PN method, but a direct comparison between the two is inappropriate. In this programme PM measurements were generally indistinguishable from tunnel background levels and consequently the PM repeatability here is more indicative of the repeatability of tunnel background measurement than engine emissions measurement. In contrast the PN figures reflect the variability of emissions from an unstable DPF system.

## X. Recommendations

354. <u>PN tunnel background</u>: High dilution tunnel background PN concentrations can have a significant impact on PN results on some test cycles. Laboratories should monitor tunnel background levels and take steps to minimise them prior to conducting type approval tests. Use of PFDS can make minimising tunnel background contributions easier owing to the greater ease of cleaning of these systems.

355. Where PFDS cannot be used, laboratories should minimise tunnel contamination prior to type approval testing by avoiding testing high particle emissions engines in advance of DPF engine approval testing, tunnel cleaning (where possible) and pre-conditioning.

356. Subtraction of tunnel background PN should not be permitted for type approval testing, but should be allowed in the case of conformity of production testing where tunnel background levels are shown to be significant.

357. <u>PN variability on WHSC</u>: PN repeatability levels on the WHSC test cycle appear to be higher than on other test cycles. This may be due to exhaust temperatures being sufficient for passive regeneration to occur for a significant proportion of the test cycle, reducing the soot cake on the DPF and reducing filtration efficiency. This effect needs to be considered and accounted for in setting regulatory PN limit values on the WHSC.

358. <u>PNC particle size cut off</u>: Numbers of solid particles below the 23 nm PNC cut off size used in Regulation No. 83 were not found to be significant compared to numbers of larger than 23 nm particles. It is therefore recommended that the 23 nm PNC cut off is retained for heavy duty engine emissions testing.

359. <u>Compensating for PN sample flow</u>: Extraction of a sample from a PFDS for PN measurement needs to be accounted for in controlling the proportionality of sampling. It is recommended that where the PN sample flow exceeds 0.1 per cent of the total dilute exhaust gas flow in the PFDS that such compensations be required. This can be achieved either by mathematical correction or physical feedback of the sample flow of (if the measurement system allows it) or by a separate feedback of an accurate flow equivalent to that drawn by the measurement system.

360. PN sample flow should also be mathematically compensated for in calculating PM emissions in the case of total flow type PFDS systems.

# Annex 1

# PMP Phase 3: Inter-Laboratory Correlation Exercise Heavy Duty engines (ILCE-HD) – Validation Exercise (VE\_HD) and Round Robin (RR\_HD)

# I. Introduction

1. This document has been prepared in response to a request from the United Kingdom Department for Transport (UK-DfT) as part of the Particle Measurement Programme (PMP). The document's purpose is to specify the testing guidelines and protocol for an inter-laboratory correlation exercise. This exercise is specifically designed to evaluate the revised particulate mass and particle number measurement techniques proposed by PMP Phase 2. The document also introduces particle number and particulate mass measurements from partial flow dilution systems as integral parts of the PMP Phase 3 work.

2. In Chapter IX, the document contains specific and detailed guidelines on how the testing should be conducted at each laboratory.

# II. Scope

3. This document defines test procedures for the inter-laboratory validation exercise and round-robin exercises to evaluate methods for particulate (all materials collected by the conventional filter method) and particle (exhaust aerosol; solid particles as defined by the measurement system) exhaust emissions measurement from heavy duty engines under transient conditions on a bench dynamometer. It is derived from the light duty inter-laboratory correlation exercise document (Ricardo RD04/04/80801.4), the existing HD type approval procedure and from draft procedures for future HD legislation (Regulation No. 49, ISO 16183 and US 2007). Regulated gaseous emissions will be measured at the same time as particulate and particle emissions, using established regulatory measurement techniques. This document is concerned with two exhaust dilution systems, namely: a full flow primary dilution tunnel with constant volume sampler (CVS) and secondary dilution system and a partial flow dilution system. This document acts as the guide for testing in the validation exercise (VE\_HD) and the round robin (RR\_HD).

# **III.** References

- 4. This specification is based upon or draws from the following documents:
- (a) UN documents related to Regulations Nos. 83 (ECE/TRANS/WP.29/GRPE/2007/8/Rev.1) and 49 (GRPE-PMP-13-03), and global technical regulation (gtr) No. 4 on WHDC.
- (b) Code of Federal Regulations Title 40, Part 86, Subpart N Emission Regulations for new Otto-cycle and diesel heavy duty engines; gaseous and particulate exhaust test procedures (revised July 1, 2001): "US2007".
- (c) ISO 16183 Heavy duty Engines Measurement of gaseous emissions from raw exhaust gas and of particulate emissions using partial flow dilution

systems under transient test conditions. To be used in its final form and referred to as "16183".

- (c) European Union Directives 2005/55/EC, 2005/78/EC and 2006/51/EC.
- (d) Aerosol measurement principles, Techniques and Applications. Ed: Paul A. Baron and Klaus Willeke, 2nd edition 2005, John Wiley & Sons Inc.
- (e) ASTM (1999): American Society for Testing and Materials ASTM E691–99: Standard Practice for Conducting an Inter-laboratory Study to Determine the Precision of a Test Method, West Conshohocken, PA.

# **IV.** Test specifications

## A. Testing Environment

5. The participating laboratories shall provide facilities and resources required to perform heavy duty engine emissions tests according to the Regulation 49, plus additional capability as required for particulate and particle measurements as defined in this document. They will also be required to install the test engine, supply measurement systems, and to liaise with the programme managing agent (PMA) and "golden engineer" (GE).

## **B.** Engine Specifications

6. (Two engines will be employed in the PMP programme. The first (VE-E1), a Euro III compliant Iveco Cursor 8 equipped with a catalyst based uncoated DPF, will be employed in the Validation Exercise (VE\_HD). The second engine (RR-E2), a Euro III compliant Mercedes OM501 equipped with a catalyst based uncoated DPF, will be employed in the round-robin programme (RR\_HD). Laboratories may test an additional engine. This could be:

- (a) A Euro IV compliant conventional diesel (without DPF). This is likely to employ high pressure injection (via CR or EUI) and selective catalytic reduction (SCR) to reach Euro IV NOx and PM levels simultaneously
- (b) Diesel-fuelled Euro IV compliant engine equipped with an OEM system diesel particulate filter (DPF). Ideally this engine will also be equipped with either common rail (CR) or electronic unit injection (EUI) systems
- (c) A Euro III or IV compliant engine equipped with a partial (open) filter
- (d) US 2007/2010 or Euro V, Euro VI development engines
- (e) Laboratories may test further engines as above, but also other possibilities (Euro IV compliant CNG engine)

# C. Lubricating Oil

7. A single lubrication oil shall be employed for VE-E1. This has been supplied by CONCAWE (BP) and is: BP Vanellus E8 ULTRA 5W-30.
#### Table 1 Lubricating oil specification

#### Typical Characteristics

	Test Methods Ui	iits	Grade :
			SAE
Density at 15 °C Kinematic Viscosity at 10 Viscosity Index CCS Viscosity at -30 °C Pour Point Flash Point (COC) Total Base Number Sulphated Ash	ISO3675/ASTM D1298 D °CISO3104/ASTM D445 ISO2909/ASTM D2270 ASTM D2602 ISO3016/ASTM D97 ISO2592/ASTM D92 ISO 3771/ASTM D2896 ISO3687/ASTM D874	kg/m³ mm²/s - mPa.s °C °C mgKOH/g %m	SAE 5W-30 860 12.03 163 5260 -54 220 15.9 1.9

8. A large single batch of lubricant will be shipped to the test laboratories in advance of the arrival of the test engine. The total volume that has been acquired for the whole of VE\_HD is sufficient for a rigorous flush and fill procedure for VE-E1 at each laboratory across the entire inter-laboratory correlation exercise.

9. Lubricating oil requirements for RR-E2 will be defined and provided by OICA. It is not intended that lubricant be changed at each RR\_HD laboratory, but filling with new lubricant may be necessary where RR-E2 is transported to a laboratory by air freight.

10. The flush and fill procedure employed at JRC will be implemented upon arrival of VE-E1 at each test laboratory. An example flush and fill procedure is shown in Appendix 1. If required, a lubricating oil change procedure for RR-E2 will be defined and provided by OICA.

#### D. Test Fuel

11. The diesel fuel to be employed during this programme will be RF06, which also complies with Annexes 3 and 4 of Directive 2003/17/EC describing fuel specifications to be employed after 1st January 2009. This fuel specification was also employed in the ILCE\_LD. A specification is given in Appendix 2.

12. A single batch of fuel for VE\_HD has been arranged by CONCAWE (Total), VE\_HD laboratories must purchase fuel from this batch directly from jean.thiebaut@total.com. With contingency, each laboratory will require 2,000 litres of diesel fuel. RR-E2 will also be tested on RF-06 diesel, but this should be sourced by RR\_HD laboratories and will not be from the VE\_HD single batch.

# V. Test protocol

# A. Delivery and preparation of test engines

13. The test engines shall be inspected for damage on arrival at the laboratory. Any problems shall be reported to the GE (Jon.Andersson@Ricardo.com) and PMA (VE-E1) (Giorgio.Martini@JRC.IT) or to Mr. Stein of OICA (RR-E2) (hj.stein@daimler.com). All engines shall be stored in an appropriate manner prior to installation.

#### **B.** Components and information

14. The following components and information shall be provided with the test engine.

- (a) Engine with test bed compatible ECU and control system
- (b) Diesel Particulate Filter (DPF)
- (c) Other exhaust components: catalysts etc
- (d) Diagnostic system
- (e) Engine mounts and brackets
- (f) After cooler and air-side pipe work (pre-set for correct pressure drop across after cooler)
- (g) Exhaust pipe flange to adapt to test bed system
- (h) Complete dimensions of test cell exhaust system
- (i) Wiring harness and throttle pedal
- (j) Wiring diagram
- (k) Engine operating parameters (e.g. back pressure, coolant and fuel temp, after cooler outlet temperature map etc)
- (l) Full load power curve data
- (m) Instrumentation for critical engine and aftertreatment operating parameters: temperatures, pressures, fuel system etc with suitable quick-fit connectors
- (n) Baseline particulate mass and gaseous emissions data

### C. Installation

15. Care shall be taken to closely replicate the manufactured test cell exhaust system dimensions between laboratories. For example, the distance between exhaust manifold and aftertreatment components shall, as far as possible, be matched between all test laboratories. The critical dimensions of the exhaust system, gas residence times between manifold and catalyst inlet and exhaust system contribution to backpressure will be supplied with the engine following installation at the first laboratory.

#### **D.** Pass-off tests

16. To confirm correct engine operation, 3 ETC cycles will be run on receipt of the engine and the results compared with data from the previous test laboratory.

### E. Test cycles

17. The engine shall be tested over 8 repeats of the cold start World Heavy Duty Transient Cycle (WHTC) along with 8 repeats of a hot-start WHTC following a 10 minute soak. In addition, 8 further tests on each of the World Heavy Duty Steady State Cycle (WHSC), the European Transient Cycle (ETC) and the European Steady State Cycle (ESC) will be undertaken.

### F. Criteria for repeat tests

18. 8 tests of each cycle shall be performed on the engine. Supplementary tests shall be carried out if one or more of the tests does not comply with current type-approval test practices (for example if the cycle does not validate, cold start failure, any malfunction during the tests). Statistical methods based on ASTM (1999) will be employed to identify outliers from the complete VE\_HD and RR\_HD datasets following completion of each programme. No more than 2 additional tests will be required for any single cycle.

#### G. Testing approach

19. The test work shall be carried out according to a pre-defined schedule for engine, exhaust and sampling system conditioning, measurement system checks and test cycles.

#### H. Test order and system preconditioning

20. Test order shall consider the possibility of contamination of test results by a previously tested engine, or from an engine in an adjacent facility which shares the dilution system. Prior to performing any emissions tests, a preconditioning phase shall be completed in order to purge the engine's exhaust system and to stabilise the dilution system with respect to the chemistry of the engine's exhaust.

21. In order to enable close control of both test procedures and test timing, a continuity protocol (Chapter V, Section L of this annex) is included in the test matrix. The continuity protocol controls the time and engine operation between tests specifically, so that testing can be exactly reproduced between laboratories.

22. A shared dilution system, in which one or more other engines are tested during the PMP test period, may not be employed in the VE\_HD. In RR\_HD, where, for facility scheduling reasons, testing of other engines in a shared dilution system during the PMP test period is unavoidable, the fact that other engines have been tested must be reported. In addition RR\_HD laboratories should make every effort to limit other engine testing to wall-flow DPF equipped engines only.

#### I. Catalyst system fill-state consistency

23. <u>Catalyst system fill-state consistency RR-HD</u>: Prior to the first testing in each laboratory, the DPF will be fully regenerated by sustained operation (2h) at ESC Mode 10. This will be the last test activity performed by each test laboratory prior to shipping of the test engine.

24. <u>Catalyst system fill-state consistency VE-HD</u>: Prior to the first testing in each laboratory, the DPF will be fully regenerated by sustained operation at ESC Mode 10. This will be conducted as part of the oil change and conditioning procedure.

#### J. Sampling, measurement and catalyst systems preconditioning

25. Preconditioning at the evening before each test day:

- (a) Preconditioning will include a 15 minutes passive regeneration phase (ESC Mode 10) and a 30 minutes DPF loading phase (ESC mode 7).
- (b) For the 15 minutes duration of the ESC Mode 10 operation and the ESC Mode 7 phase, exhaust will be diverted through the CVS and secondary dilution system.
- (c) The partial flow sampling system shall be operated in bypass during the 15 minutes of the ESC Mode 10 conditioning period and the ESC Mode 7 phase, with operating parameters adjusted to give tunnel temperature of  $\sim$ 70 °C.

#### K. Preconditioning – shared dilution system

26. For RR\_HD laboratories, in a shared dilution system, where a non wall-flow DPF equipped engine's exhaust is passed into a dilution tunnel which is shared between 2 or more cells, the preconditioning detailed in Chapter V, Section J of this annex must be performed the previous evening. A dilution system shared between two DPF equipped diesel engines is acceptable without additional tunnel preconditioning.

#### L. Continuity protocol (CP)

27. Between each transient cycle, the continuity protocol shall be applied. The continuity protocol is employed to ensure identical temperature profiles in the engine and exhaust following each test. This will enable the test work to be closely replicated from facility to facility. The protocol will be similar to that described below:

- (a) Drop to idle for 5 minutes (if the engine was not turned off);
- (b) 5 minutes operation at ESC mode 7;
- (c) Drop to idle for 3 min and commence test sequence (Firstly a zero and span of the analysers and then commencement of the automated part of the emissions cycle);
- (d) If a specific period of engine operation is mandated as preconditioning for a particular drive cycle, this operation may replace one or more stages of the CP for that cycle only.

# VI. Measurement and sampling systems gaseous emissions

#### A. Full flow dilution system

28. The mass of gaseous emissions shall be measured from the dilute exhaust during all tests in accordance with the current Regulation No. 49 / gtr No. 4 on WHDC.

### B. Raw exhaust sampling

29. The mass of gaseous emissions shall be measured from the raw tailpipe exhaust in accordance with Regulation No. 49 / gtr No. 4 on WHDC for steady-state cycles, and ISO 16183 for transient cycles. If possible, engine out raw emissions shall also be measured on a continuous basis throughout the test.

# VII. Measurement and sampling systems for particulates: full flow

# A. Introduction

30. The mass of particulate material emitted by the test engine and for each test will be measured using the system defined in Chapter VII, Sections B to E and Chapter IX. Two possible examples of compliant particulate measurement system configurations are shown in Figures 1 and 2.

Figure 1

Example of particulate measurement system (1)





#### Figure 2 Example of Particulate Measurement System (2)

#### **B.** Dilution systems

31. A full flow double dilution system shall be used for particulate mass measurements.

32. A full flow CVS exhaust dilution tunnel system meeting the requirements of Regulation No. 49 shall be used. The CVS flow rate at each lab (Qi) will be selected such as to ensure similar residence times in the primary dilution tunnel (±25 per cent), according to the equation:

 $Qi = Q \times L / Li \times D^2/Di^2$ 

where

 $Q = 80 \text{ m}^3/\text{min},$ 

D = 0.47 cm,

L = 4.7 m and

Li and Di are the length and inner diameter of the test laboratory CVS

33. The dilution air used for the primary dilution of the exhaust in the CVS tunnel shall be first charcoal scrubbed and then passed through a secondary filter. The secondary filter should be capable of reducing particles in the most penetrating particle size of the filter material by at least 99.95 per cent, or through a filter of at least class H13 of EN 1822; this represents the specification of High Efficiency Particulate Air (HEPA) filters.

34. A secondary dilution system meeting the requirements of Regulation No. 49 shall be used. The dilution ratio in the secondary dilution system shall be fixed such that tunnel temperature is < 52 °C and ideally 47 °C +/- 5 °C. Where possible, the dilution should be one part dilution air to one part sample aerosol. The dilution air for the secondary dilution system shall be subject to HEPA and charcoal filtration.

#### C. Particulate sampling: primary tunnel

35. A sample probe shall conduct materials to the secondary dilution tunnel. It shall be installed near the tunnel centre-line, 10-20 tunnel diameters downstream of the gas inlet and have an internal diameter of at least 12 mm. The sample probe will be sharp-edged and open ended, facing directly into the direction of flow in the primary dilution tunnel.

36. Where the system permits, a sample probe will be installed in the secondary dilution tunnel. It shall be sharp-edged and open ended, facing directly into the direction of flow. For systems which draw the entire secondary tunnel flow through the PM filter this is not necessary.

37. A cyclone or impactor based pre-classifier shall be employed at VE\_HD laboratories. At RR\_HD laboratories use of a cyclone or impactor pre-classifier shall be optional.

38. A pump will draw a sample of dilute exhaust gas proportional to the total tunnel flow through the sample pre-classifier and filter holder.

39. The distance from the entrance to the secondary tunnel to the filter mount shall be at least five probe diameters, but shall not exceed 1,500 mm.

#### D. Sample pre-classifier

40. At VE\_HD laboratories, and optionally at RR\_HD laboratories, in accordance with the recommendations of the draft Regulation No. 49 document (GRPE-PMP-13-3), a cyclone or impactor pre-classifier shall be located upstream of the filter holder assembly. The pre-classifier 50 per cent cut point particle diameter shall be between 2.5  $\mu$ m and 10  $\mu$ m at the volumetric flow rate selected for sampling particulate mass emissions. The pre-classifier shall allow at least 99 per cent of the mass concentration of 1  $\mu$ m particles entering the pre-classifier to pass through the exit of the pre-classifier at the volumetric flow rate selected for sampling particulate mass emissions. Evidence of compliant performance to this specification shall be presented (e.g. manufacturer's calibration certificate).

#### E. Filter sampling

41. <u>Filter face temperature</u>: A temperature of < 52 °C, and ideally 47  $\pm$  5 °C shall be maintained within 20 cm of the filter face:

- (a) This shall be achieved by either direct heating means: the filter holder shall be heated by a mantle or similar, or be mounted inside a temperature-controlled enclosure with the transfer lines to the filter holder heated to enable a residence time of at least 0.2 s at the above temperature to be achieved,
- or
- (b) the temperature of the aerosol within the secondary dilution tunnel shall be controlled to the required temperature by heating of the dilution air. In this case, the temperature of the dilution air shall be modulated in response to the temperature of the transfer gases between the primary and secondary dilution tunnels. Residence time at ~47 °C in the secondary tunnel and at the filter face shall be at least 0.2 s.

42. <u>The filter holder assembly</u>: The filter holder assembly shall be of a design that provides for a single filter only. The shape of the holder shall be such that an even flow distribution of sample across the filter stain area is achieved.

43. <u>Filter medium</u>: Pallflex TX40 Fluorocarbon coated glass fibre filters shall be employed. All filters will be drawn from a single batch procured by the project-managing laboratory for the VE\_HD. Laboratories participating in the RR\_HD may use alternative media if that media meets the performance specifications of the TX40 filters.

44. <u>Filter size and stain area</u>: For VE\_HD laboratories the filter diameter shall be 47 mm and the stain area shall be at least 1075 mm<sup>2</sup>.

45. <u>Filter face velocity / volumetric sample flow rate</u>: Filter face velocity shall be in the range 55 cm/s to 90 cm/s, which corresponds to a flow rate range of 30 l/min to 50 l/min with 47 mm filters. Filter face velocity should be calculated at 47 °C or temperature corrected mass-flow controllers used.

# VIII. Measurement and sampling systems for particulates: partial flow

## A. Introduction

Figure 3

46. The mass of particulate material emitted by the test engine and for each test will be measured using the system defined in Chapter VIII, Sections B to F (para. 60) and Chapter IX. An example of a compliant particulate measurement system configuration is shown in Figure 3. Sampling and measurements will be undertaken according to ISO 16183 except where parameters are explicitly specified in this document.



Example of partial flow measurement system

#### **B.** Dilution system

47. A partial flow, single dilution system meeting the requirements of ISO 16183 shall be used for particulate mass measurements in tandem with the full flow system. Exhaust gas mass flow shall be determined using one of the methods outlined in ISO 16183, and the resulting data shall be used for controlling the sample rate from the raw exhaust into the partial flow dilution tunnel.

48. For transient tests, attention should be given to the dynamic performance of the various measurement and control systems, and good engineering practise should be employed as required to ensure that the sample drawn from the raw exhaust is proportional to the exhaust flow rate at the sample point. The use of a pre-recorded exhaust flow trace for look-ahead control is not allowed.

49. The same flow measurement and control approach as used for transient cycles shall be used for steady state tests.

50. All dilution air supplies for the partial flow dilution system shall be subject to HEPA and charcoal filtration. The dilution system shall be capable of achieving the filter sampling conditions as outlined in Chapter VIII, Section F below.

#### C. Dilution system parameters for golden engine

51. For the golden engine only, dilution system operating parameters such as split ratio, dilution factor and filter flow rate shall be as follows:

Split ratio	Total CVS flow rate	Filter flow rate	
For 80 m <sup>3</sup> /min and 50 lpm or 0.000626	For 80 m <sup>3</sup> /min or 6200 kg/h	50 lpm or 3000 nl/h or 1.08 g/s	

52. These provide maximum tunnel temperature of 52 °C, maximised filter loading and filter face velocity in the permitted range (Chapter VIII, Section F) for all test cycles. These parameters will be reproduced during VE-E1 testing at each test laboratory. Testing in the RR\_HD may employ these or a laboratory's own standard procedures. When particle number and particulate mass samples are taken simultaneously from a partial flow system, the additional flow taken by the particle number measurement system must be either replaced – flow pumped back into the system under mass flow control downstream of the filter but upstream of the measurement element or corrected in the partial flow systems' software.

#### **D.** Particulate sampling point

53. The sampling point for collection of particulate matter from the exhaust system of the test engines shall be determined according to the recommendations of the partial flow dilution system manufacturer and ISO 16183.

54. <u>Particulate sampling point for VE-E1 only</u>: The distance between the exit from the DPF and the sampling point has been determined following preliminary testing at JRC as 5 m ( $d_{in} = 15$  cm), as far as possible the sampling point for particle measurements using the partial flow tunnel shall be reproduced in all subsequent laboratories. However, if different diameter exhaust tubing is employed, sampling after a similar exhaust volume is required. The exhaust volume at the sampling point should be  $0.09 \pm 0.01$  m<sup>3</sup> (80 to 100 litres).

55. <u>Particulate sampling point for additional engine(s) and RR-E2</u>: Sampling may use either the same point as the VE-E1 or an alternative point selected according to paragraph 53.

#### E. Sample pre-classification

56. The sample probe used for raw exhaust sampling shall be the original equipment provided by the partial flow system manufacturer. In accordance with ISO 16183, it is recommended that a pre-classifier is installed immediately upstream of the filter holder.

### F. Filter sampling

57. <u>Filter face temperature</u>: A maximum filter face temperature of 52 °C shall be recorded within 20 cm of the filter face. This temperature will be controlled by selecting the appropriate split / dilution ratios within the partial flow system and according to the manufacturer's recommendations and ISO 16183.

58. <u>Filter holder assembly</u>: The filter holder shall be the original equipment as provided by the partial flow system manufacturer. This may be of a design that provides for either a single filter, or sample and backup filters, but in the latter case only the sample filter should be used.

59. <u>Filter medium</u>: For the VE\_HD 47 mm Pallflex TX40 Fluorocarbon coated glass fibre filters shall be employed. All filters for VE-E1 will be drawn from a single batch procured by the project-managing laboratory. For the RR\_HD, either 47 mm or 70 mm filters of TX40 (or equivalent) may be employed.

60. <u>Filter size and stain area</u>: The filter diameter shall be 47 mm or 70 mm with a stain area of at least  $1075 / 2825 \text{ mm}^2$  respectively.

- 61. Filter face velocity / volumetric sample flow rate:
- (a) <u>VE-E1 Sampling</u>: The filter face velocity conditions selected by the first laboratory testing the golden engine shall be reproduced by all other test laboratories. The filter face velocity shall be in the range 55 cm/s to 90 cm/s, which corresponds to a flow rate range of 72 l/min to 130 l/min with 70mm filters and 30 l/min to 50 l/min with 47 mm filters. Filter face velocity shall be calculated at mean partial flow tunnel temperature. Ideally, temperature corrected mass-flow controllers shall be used. The recommended flowrate is 50 lpm (similar to the full flow secondary tunnel). This corresponds to 3000 nl/h (for PSS) and 1.08 g/s for Smart sampler (47 mm filters).
- (b) <u>RR-E22 and additional engine(s) sampling</u>: For any additional engines, the test laboratory may use any appropriate sampling conditions according to the manufacturer's recommendations and ISO 16183. All conditions must be reported.

# IX. Particulate measurement equipment and environment

62. The following parameters and equipment are common to both partial flow and full-flow particulate mass sampling.

# A. Filter Preparation

63. The particulate sampling filters shall be conditioned (as regards temperature and humidity) in an open dish that has been protected against dust ingress for at least 8 and for not more than 80 hours before the test in an air-conditioned chamber. After this conditioning, the uncontaminated filters will be weighed and stored until they are used. If the filters are not used within one hour of their removal from the weighing chamber they shall be re-weighed.

64. The one-hour limit may be replaced by an eight-hour limit if one or both of the following conditions are met:

- (a) a stabilised filter (filters) is placed and kept in a sealed filter holder assembly with the ends plugged, or;
- (b) a stabilised filter (filters) is placed in a sealed filter holder assembly which is then immediately placed in a sample line through which there is no flow.

#### **B.** Microgram balance

65. The analytical balance used to determine filter weight must have a precision (standard deviation) of better than 2  $\mu$ g for a clean filter; better than 1  $\mu$ g for a reference weight and a readability of 1  $\mu$ g or better. To eliminate the effects of static electricity: the balance should be grounded through placement upon an antistatic mat and particulate filters should be neutralised prior to weighing; this can be achieved by a Polonium neutraliser or a device of similar effect.

66. <u>Balance integrity</u>: At the start of each weighing session a 50 mg weight with a certified value (recertified annually) will be weighed 3 times. The mean of these three weighings shall be  $\pm 5 \ \mu g$  of the certified value. If the mean value is outside this tolerance, the balance shall be recalibrated.

#### C. Weighing chamber parameters

67. The temperature of the chamber (or room) in which the particulate filters are conditioned and weighed must be maintained to within 295 K  $\pm$  3 K (22 °C  $\pm$  3 °C) during all filter conditioning and weighing. The humidity must be maintained to a dew point of 282.5 K  $\pm$  3 K (9.5 °C  $\pm$  3 °C) and a relative humidity of 45  $\pm$  8 per cent. The weighing room parameters should be controlled as tightly as possible.

#### **D.** Calibration requirements

68. <u>Microbalance calibration</u>: The microbalance shall be calibrated according to the manufacturer's specification within 3 months prior to the commencement of the test programme.

69. <u>Reference filter weighing</u>: To determine the specific reference filter weights, at least two unused reference filters shall be weighed within 8 hours of, but preferably at the same time as, the sample filter weighings. Reference filters shall be the same size and material as the sample filter.

70. If the specific weight of any reference filter changes by more than  $\pm 5 \ \mu g$  between sample filter weighings, then the sample filter and reference filters shall be reconditioned in the weighing room and then reweighed.

71. The comparison of reference filter weighings shall be made between the specific weights and the rolling average of that reference filter's specific weights. The rolling average shall be calculated from the specific weights collected in the period since the reference filters were placed in the weighing room. The averaging period shall be at least 1 day but not exceed 30 days. Multiple reconditioning and reweighing of the sample and reference filters are permitted until a period of 80 hours has elapsed following the measurement of gases from the emissions test. If, prior to or at the 80 hours point, more than half the number of reference filters meet the  $\pm 5 \ \mu g$  criterion, then the sample filter weighing can be considered valid.

72. If, at the 80 hours point, two reference filters are employed and one filter fails the  $\pm 5 \mu g$  criterion, the sample filter weighing can be considered valid under the

following condition: the sum of the absolute differences between specific and rolling averages from the two reference filters must be less than or equal to 10  $\mu$ g. In the case that less than half of the reference filters meet the  $\pm 5 \mu$ g criterion the sample filter shall be discarded, and the emissions test repeated. All reference filters must be discarded and replaced within 48 hours.

73. In all other cases, reference filters must be replaced at least every 30 days and in such a manner that no sample filter is weighed without comparison with a reference filter that has been present in the weighing room for at least 1 day.

# X. Particle measurement system and sampling systems

74. For the VE\_HD, the number of particles emitted by each engine technology and for each test cycle shall be determined using two nominally identical 'Golden Particle Measurement Systems' (GPMS). These will be Horiba Solid Particle Counting Systems (SPCS). Particle numbers shall be determined by measurement from the primary dilution tunnel (full flow) and from the partial flow dilution system. The majority of GPMS components will be provided, though certain items indicated in the text shall be provided by the laboratory.

75. Throughout the duration of the VE\_HD, each laboratory participating in the RR\_HD will supply two particle measurement systems and must perform simultaneous measurements from the full flow dilution system and a partial flow system.

76. After completion of testing in the VE\_HD, laboratories participating in the RR\_HD may elect to measure particle numbers from full-flow alone, partial flow alone or both full-flow and partial-flow dilution systems and will supply sufficient particle number measurement systems. All alternative particle number measurement systems [ALT\_SYS] including all systems to be employed in the RR\_HD must be fully certificated to the requirements of the following sections and/or to the relevant sections in the United Kingdom proposal to amend Regulation No. 83 (ECE/TRANS/WP.29/GRPE/2007/8/Rev.1). Calibration reports demonstrating compliance with the above requirements must be provided for all RR\_HD measurement systems and all VE\_HD alternative measurement systems.

#### A. Safety

77. The electrical components of the GPMS supplied as part of the PMP Programme, shall not be modified in any way by employees of the participating laboratories unless the express permission of the Inter-lab manager or golden engineer is given. All modifications, for example: of electrical connectors shall be recorded and the subsequent laboratory informed of changes so that safety checks can be performed prior to further testing.

#### **B.** Particle sampling system

78. The particle sampling system shall be identical for the two GPMS systems with the exception of the sampling tubes. Two sampling tubes will be required: in the primary dilution tunnel for full flow sampling ( $PST_f$ ) and partial flow dilution tunnel for partial flow sampling ( $PST_p$ ). Further elements of the particle sampling system are: a particle pre-classifier (PCF) and the GPMS particle conditioning and measurement system comprising a volatile particle remover (VPR) upstream of the particle number counter (PNC\_GOLD) unit. The particle sampling system is required to draw a sample from the primary or partial flow dilution systems, size classify it,

transfer it to a diluter, condition the sample so that only solid particles are measured, and pass a suitable concentration of those particles to the particle counter.

79. <u>Sample probe – Full flow</u>: The sampling probe tip (PSP) in the CVS and particle transfer tube (PTT) together comprise the particle transfer system (PTS). The PTS conducts the sample from the dilution tunnel to the entrance to the VPR. The PTS shall meet the following conditions:

- (a) It shall be installed near the tunnel centre line, 10 to 20 tunnel diameters downstream of the gas inlet, facing upstream into the tunnel gas flow with its axis at the tip parallel to that of the dilution tunnel.
- (b) It shall have an internal diameter of  $\ge 8 \text{ mm}$
- 80. Sample gas drawn through the PTS shall meet the following conditions:
- (a) It shall have a flow Reynolds number (Re) of < 1700.
- (b) It shall have a residence time in the PTS of  $\leq 3$  seconds.

81. Any other sampling configuration for the PTS for which equivalent particle penetration at 30 nm can be demonstrated will be considered acceptable.

82. <u>Sample probe – partial flow</u>: A sample probe is recommended to be installed in the partial flow dilution tunnel or downstream sampling system but will be placed upstream of the PM sample filter holders. It shall be sharp-edged and open-ended and comprised of stainless steel.

83. <u>Particle pre-classifier – full flow</u>: The upper limit of the particle size range to be measured shall be determined by the use of the cyclone particle size pre-classifier provided. The 50 per cent cut-point of the particle pre-classifier shall lie at between 2.5  $\mu$ m and 10  $\mu$ m. The laboratory will provide a suitable pump to ensure that the upper size limit of particles sampled into the measurement system lies within this range.

84. <u>Particle pre-classifier – partial flow</u>: Optionally, the upper limit of the particle size range to be measured will be determined by the use of an inertial particle size pre-classifier. The 50 per cent cut-point of the particle pre-classifier shall lie at 2.5  $\mu$ m. The laboratory will provide a suitable pump to ensure an upper size limit of particles sampled into the measurement system of 2.5  $\mu$ m. Any flow drawn from the partial flow tunnel must be replaced upstream of the measurement element or taken into account in the partial flow system software.

#### C. Volatile particle remover (VPR)

85. The VPR shall be used to define the nature of the particles to be measured.

86. <u>Description</u>: The VPR provides heated dilution, thermal conditioning of the sample aerosol, further dilution for selection of particle number concentration and cooling of the sample prior to entry into the particle number counter.

- 87. <u>Elements of the VPR</u>: The VPR shall comprise the following elements:
- (a) The first particle number dilution device shall be specifically designed to dilute particle number concentration and operate at a (wall) temperature of 150-400 °C. The wall temperature setpoint should not exceed the wall temperature of the ET (paragraph 2.). The diluter should be supplied with HEPA filtered dilution air and be capable of a dilution factor of 10 to 200 times. For the golden engine, the dilution factor of this diluter; PNDF1 will be provided by the golden engineer and replicated at subsequent sites. For

additional engines, the dilution ratio will be determined by experimentation and agreed with the GE and PMA.

- (b) The entire length of the ET shall be controlled to a wall temperature greater than or equal to that of the first particle number dilution device and the wall temperature held at a fixed value between 300 °C and 400 °C.
- (c) PND2 shall be specifically designed to dilute particle number concentration. The diluter shall be supplied with HEPA filtered dilution air and be capable of maintaining a single dilution factor within a range of 10 to 30 times. The dilution factor of PND2 shall be selected in the range between 10 and 15 such that particle number concentration downstream of the second diluter is less than the upper threshold of the single particle count mode of the PNC and the gas temperature prior to entry to the PNC is < 35 °C.</p>

88. <u>Performance</u>: The VPR shall achieve > 99.0 per cent vaporisation of 30 nm tetracontane (CH<sub>3</sub>(CH<sub>2</sub>)38CH<sub>3</sub>) particles, with an inlet concentration of > 10,000 cm<sup>-3</sup>, by means of heating and reduction of partial pressures of the tetracontane. It shall also achieve a particle concentration reduction factor (f<sub>r</sub>) for particles of 30 nm and 50 nm electrical mobility diameters, that is no more than 30 and 20 per cent respectively higher, and no more than 5 per cent lower than that for particles of 100 nm electrical mobility diameter for the VPR as a whole.

89. <u>Performance of other particle sampling and transport system elements -</u> <u>GPMS</u>: The outlet tube (OT) conducting the diluted sample from the VPR to the inlet of the PNC shall have the following properties:

(a) It shall have an internal diameter of  $\geq 4$  mm.

(b) Sample gas flow through the POT shall have a residence time of  $\leq 0.8$  seconds.

90. Any other sampling configuration for the OT for which equivalent particle penetration for particles of 30nm electrical mobility diameter can be demonstrated will be considered acceptable.

#### **D** Particle counter (Particle Number measurement unit, PNC)

91. The particle counter is used to determine the number concentration of solid particles in a diluted sample of engine exhaust aerosol continuously drawn from the CVS.

- 92. PNC Performance Characteristics
- (a) Operate under full flow operating conditions.
- (b) Have a counting accuracy of ±10 per cent across the range 1 cm<sup>-3</sup> to the upper threshold of the single particle count mode of the PNC against a traceable standard. At concentrations below 100 cm<sup>-3</sup> measurements averaged over extended sampling periods may be required to demonstrate the accuracy of the PNC with a high degree of statistical confidence.
- (c) Have a readability of at least 0.1 particles  $\text{cm}^{-3}$  at concentrations below 100  $\text{cm}^{-3}$ .
- (d) Have a linear response to particle concentrations over the full measurement range in single particle count mode.
- (e) Have a data reporting frequency equal to or greater than 0.5 Hz.

- (f) Have a T90 response time over the measured concentration range of less than 5 s.
- (g) Incorporate a coincidence correction function up to a maximum 10 per cent correction, and may make use of an internal calibration factor as determined in paragraph 2.1.3, but shall not make use of any other algorithm to correct for or define the counting efficiency. For the 3010Ds of the SPCSs the correction will be done externally
- (h) Have counting efficiencies at particle sizes of 23 nm (±1 nm) and 41 nm (±1 nm) electrical mobility diameter of 50 per cent (±12 per cent) and > 90 per cent respectively. These counting efficiencies may be achieved by internal (for example; control of instrument design) or external (for example; size pre-classification) means.
- (i) The PNC working liquid shall be replaced at the frequency specified by the instrument manufacturer. At least 2.5 litres of the working fluid, per PNC, shall be provided for the test work.
- (j) The sum of the residence time of the PTS, VPR and OT plus the T90 response time of the PNC shall be no greater than 20 s.

#### E. Sampling lines

93. It is recommended that all sampling lines be of stainless steel composition with conductive silicone tubing and TYGON (specifically R3603) are also acceptable. Sampling lines shall contain smooth internal surfaces and be of minimal length. Sharp bends and abrupt changes in section should be avoided in all sampling lines.

#### F. Calibration of particle number measurement systems

94. Calibration requirements for the PNC and VPR, including the calculation of the particle concentration reduction factor, can be found in the following documents, supplied with this guide:

- (a) PNC: Particle Number Counter Calibration Procedure, Report to the Department for Transport; ED47382004/PNC, AEA Technology Issue 5, December 2007
- (b) VPR: Volatile Particle Remover Calibration Procedure, Report to the Department for Transport; ED47382004/VPR, AEA Technology Issue 5, December 2007
- (c) ECE/TRANS/WP.29/GRPE/2007/8/Rev.1

95. <u>Golden Particle number Measurement Systems</u>: Prior to commencement of the test programme, the GPMSs will be fully calibrated by the instrument suppliers to meet the required specifications.

96. <u>Alternative measurement systems for particles</u>: For Alternative Systems (ALT-SYS) in the VE\_HD and for all systems in the RR\_HD, test laboratories shall propose their own specific particle number measurement systems for measurement from the CVS and partial flow systems. In the VE\_HD, alternative systems installations will be subject to approval by the GE and/or PMA.

97. Alternative Systems shall comply with the sections of this document and provide appropriate certification to ensure performance and calibration of the systems meet the required specification: Chapter X of this annex, Section B, paras. 79-80, 83 and Sections C, D and E (including all sub-sections).

# **XI.** Test procedures

## A. Test matrix

98. Testing shall be undertaken strictly according to the requirements and order stated in the test matrix. A test matrix addressing 8 cold start WHTC (C-WHTC), 8 hot start WHTC preceded by a 10 minute soak (H-WHTC\_10), 8 ETC, 8 WHSC and 8 ESC cycles during two week's testing in a single laboratory is shown in Table 1 below.

Table 1:Example test matrix, heavy duty testing

Previous lab	Day 0	Days 1-7	Day 8		
	oil change	IFV	IFV		
	2h ESC Mode 10	cold WHTC	cold WHTC		
	3 x ETC 10 minute soak		10 minute soak		
		hot WHTC	hot WHTC		
		10 minutes at WHSC mode 9	10 minutes at WHSC mode 9		
		WHSC	WHSC		
		CP	CP		
		ETC	ETC		
		CP	CP		
		ESC	ESC		
*2 hours at ESC Mode 10	Precon	Precon	*2 hours at ESC Mode 10		
ESC - European Steady State Cycle for emissions measurement [30 min]					
ETC - European Transient Cycle for emissions measurement [30 min]					
WHTC - World Harmonised Steady State Cycle for emissions measurement [30 min]					
WHTC - World Harmonised Tran	nsient Cycle for emis:	sions measurement [30 min]			
IFV - Instrument Functional Ver	ification				
CP - Continuity Protocol					
Precon - 15 miuntes ESC mode 10, 30 minutes ESC mode 7					

#### \* DPF regeneration only required if oil change and conditioning not performed

#### **B.** Preparation of the engine

99. Engines shall be prepared in accordance with Regulation No. 49 and good engineering practice for emissions testing. The fuel and lube oil used shall be as specified in Chapter IV, Sections C and D of this annex.

100. <u>Instrumentation</u>: The engine and exhaust system will be suitably instrumented for exhaust and catalyst temperatures, and DPF pressure drop and backpressure. These shall be recorded from each emissions cycle and during the Mode 10 conditioning.

### C. Dynamometer preparation

101. The engine shall be mapped across the speed range according to Regulation No. 49, and the ESC,  $WHSC^1 / ETC / WHTC$  cycle reference speeds ( $n_{lo}$ ,  $n_{hi}$  and  $n_{ref}$ ) once per engine and cycle set-points shall be calculated.

102. The dynamometer control parameters shall be adjusted as necessary to meet the test cycle verification requirements of Regulation No. 49.

<sup>&</sup>lt;sup>1</sup> Where applicable

103. The results of the power map and cycle verification tests shall be reviewed by the project manager and golden engineer.

#### D. Test and conditioning protocols

104. Prior to any testing, the exhaust system, transfer tube and dilution systems shall be thermally purged. This shall be achieved by operating the engine at ESC mode 10 for 15 minutes the previous day. The flow settings on the partial flow system may be adjusted for this procedure, so that an elevated tunnel temperature is achieved (52-70  $^{\circ}$ C) whilst remaining within safe operational limits.

105. Specific requirements for the preconditioning are specified in Chapter V, Section J of this annex. Throughout each day's testing, the engine shall be stabilised between tests through the continuity protocol (Chapter V, Section L of this annex).

106. Warm-up and pre-conditioning procedures shall be carried out on the measurement and sampling systems as appropriate. System verification and calibration checks as required shall be performed daily.

#### E. Test procedures – gaseous emissions

107. For the full flow dilution system, gaseous emissions shall be determined from diluted exhaust according to the procedures described in the Regulation No. 49. Gaseous emissions shall also be determined directly from the raw exhaust according to Regulation No. 49 for steady-state cycles and ISO 16183 for transient cycles.

108. <u>Preparation for the test</u>: Prior to the test the gaseous emissions analysers shall be calibrated using suitable reference gases, on the ranges that will be used during the test. The zero and span readings shall be recorded.

109. <u>Partial flow dilution only</u>: Prior to the test the response times of the gas analysers and exhaust flow measurement devices shall be determined in accordance with ISO 16183.

110. <u>During the test</u>: During each test the data from the gaseous emissions analysers shall be recorded with a logging rate of at least 0.5 Hz for the full flow (dilute) analysers and 2 Hz for the raw emissions analysers.

111. <u>Full flow dilution only</u>: At the start of the test, the bag-sampling unit shall be switched to start filling the sample and ambient bags.

112. <u>Post test – full flow dilution</u>: At the end of the test the bag sampling unit shall be stopped. Following the test the zero and span readings of the gaseous emissions analysers shall be checked and recorded. The analysers shall then be calibrated using suitable reference gases, on the ranges that will be used for analysing bag samples. The emissions concentrations in the bag samples shall then be measured and recorded.

#### F. Test procedures - Particulate emissions: full and partial flow

113. <u>Preparation of the partial flow dilution system</u>: Prior to the test, the flow settings for the partial flow dilution system shall be determined, as required to meet the sampling requirements of Chapter VIII of this annex. If necessary, a pre-test cycle shall be run and the exhaust flow data recorded by the partial flow sampling system.

114. <u>Preparation of the test (filter weighing, switch to bypass)</u>: Prior to the test the test filters shall be conditioned in the weighing room. The initial filter masses shall be measured and recorded on a microbalance with 1  $\mu$ g or better resolution. Temperature and humidity during sample and reference filter weightings shall be recorded. During

Table 2:

the system stabilisation procedure, the particulate sampling systems shall be operated on bypass.

115. During the test (switch to sample): At the start of the test, the particulate sampling systems shall be switched from the bypass to the sample filters.

116. Post test (condition and weigh filters): On completion of the test, the particulate sampling systems shall be stopped. The filter holders shall be removed and the filters returned to the weighing room or chamber for conditioning. After conditioning the filters shall be weighed and the masses recorded. Temperature and humidity during sample and reference filter weightings shall be recorded. It is recommended that the sample filters are not weighed until at least 4 hours have elapsed since they were placed in the weighing room or chamber.

#### G. **Test procedures – particle emissions**

117. The following sections describe the procedures that shall be followed by each laboratory in receiving, installing and operating particle measurement systems.

118. On arrival at the laboratory, all equipment shall be unpacked and inspected for damage. If any components are missing or damaged the golden engineer and project manager shall be informed.

119. The equipment accompanying the VE HD golden engine that will be circulated between laboratories is summarised in Table 2.

Engine Lubricant Filter for the lubricant change TX40 filters SPCS-19 with lap top SPCS-20 with lap top 4m heated line with controller 2 T connections for SPCSs (HEPA/sample) 3790 CPC Cyclone Sampling tube (insulated) Pump with orifice for 90 lpm

Components for circulation around participating laboratories

120. Laboratories should provide filtered pressurised air for SPCS (6 bar, 25 lpm each), LAN and RS232 cables (for each SPCS) of enough length to move the laptops outside the test cell, power for the SPCS units (380V, 32 A supplies fed by 220V, 16A transformers for each SPCS), feedback filtered and HC scrubbed air for the partial flow sampling systems (3.5 lpm) that cannot take into account the extracted flow for particle number measurement. Around 2 l of butanol for the CPCs.

- 121. The instrument functional verification at the beginning of the day includes:
- (a) Warm up of both SPCS 30 min
- (b) Connect HEPA filter at the inlet
- (c) Open laptops and software, CPC check
- (d) File Sample
- (e) Set correct values (dilution air and dilution factor)
- (f) If everything ok, connect SPCS to dilution tunnel or partial flow sampling system

122. More details will be supplied in the "daily check" spreadsheet that will be provided by JRC.

123. Dilution factor settings for PND1 and PND2 have been determined in the first laboratory. These shall be employed for the first test on VE-E1 at each subsequent laboratory. The DFs for the first and second diluters are as follows (for both SPCS units):

- (a) PNDF1 =10, dilution air=11 (11.5 for SPCS-20)
- (b) PNDF2 = 15, dilution air=10.5
- (c) Bypass=2

#### Figure 4 Schematic layout for GPMS



124. <u>Preparation of daily protocol – instrument warm-up and daily verification</u> <u>exercises</u>: The following will be undertaken for GPMS and ALT\_SYS in both the VE\_HD and RR\_HD.

125. First thing each morning all the elements of the particle measurement systems will be activated, and left for at least 30 minutes to stabilise. This includes pumps, heaters, diluters and particle counters. The temperature of heated sections will be

inspected to ensure compliance with the requirements of Chapter VIII, Section B of this annex.

126. Instrument manufacturers of the various elements of the particle measurement systems will provide calibration certification for the diluter(s), evaporation tube and particle counter employed for PMP particle number measurements. These data will be appropriate to address those requirements for primary calibration of instrumentation defined in draft Regulation No. 49. However, it should be noted that the regulations are drafted with the intention that instrument manufacturers will have time to develop entirely suitable equipment and at this time exact compliance of all instrumentation with the draft regulations may not be possible.

127. Therefore the main issues are that operation consistent with the baseline calibrations is ensured, and that repeatable and valid operation can be demonstrated and maintained. In order to ensure this, regular calibration checks shall be performed. These are summarised as follows:

(a) Verification of Free Sample Flow and Flow rate – The particle measurement systems shall be checked for physical blockages and the CPC flow rate checked. The measured flow rate shall be within 5 per cent of the instrument's nominal value.

(b) Verification of Counter Zero – An initial concentration of around  $10000/\text{cm}^3$  (e.g. background number concentration) will be applied to any PNCs via a HEPA filter and using clean, particle free tubing. Testing shall commence if the measured particle count is less than  $0.2/\text{cm}^3$ 

(c) Verification of System Contamination and Leak Integrity – After heating the evaporation tube a HEPA filter will be applied to the inlet of the diluter and particle number concentration through the whole system measured using PNC\_GOLD. Testing can commence providing the measured particle count is less than 0.5/cm<sup>3</sup>.

128. The particle measurement system shall then be fully reassembled. A sample line connected downstream of the particle pre-classifier shall then be connected to the inlet of the VPR. Sampling shall commence.

#### H. Troubleshooting

129. In the VE\_HD any problems encountered during the daily verification exercise should be referred to the golden engineer or project manager who will make a decision on whether to proceed with the test programme.

130. In the RR\_HD, the particle measurement system manufacturer shall be consulted.

#### I. During the test

131. During each emissions test, particle number concentrations from the PNC shall be measured continuously in the particle sampling system with a frequency of  $\ge 0.5$  Hz. The average concentrations shall be determined by integrating the analyser signals over the entire period of the test cycle, with data recorded electronically. The system response time shall be  $\le 20$  s, and shall be co-ordinated with primary tunnel (CVS) flow fluctuations and sampling time/test cycle offsets, if necessary.

## J. Post-test (where specified in the Test Matrix)

132. The following instrument function verification tests will be performed according to the demands to the daily test protocol:

- (a) Verification of Free Sample Flow The particle measurement system shall be checked for physical blockages. (Chapter XI, Section G, para. 27(a) of this annex). The PNC flow rate will be checked.
- (b) Verification of Counter Zero An initial concentration of around 10000/cm<sup>3</sup> (e.g. background number concentration) will be applied to the PNC via a HEPA filter and using clean, particle free tubing. Testing shall commence if the measured particle count is less than 0.2/cm<sup>3</sup> (Chapter XI, Section G, para. 27(b) of this annex).
- (c) Data from each test will be inspected to determine whether instantaneous concentrations at the PNC have exceeded 104 particles/cm<sup>3</sup> during the emissions cycle.

133. If this has occurred, the dilution ratios of PND1 and PND2 may need to be modified. In the PMP, these modifications shall be discussed with and approved by the project manager or golden engineer prior to the next test on that engine.

134. If necessary, the PND1 and PND2 diluters should be cleaned at this stage. It is not anticipated that this will be required with tests on a DPF equipped engine, but laboratories testing conventional diesels may encounter contamination issues.

135. <u>Repeat daily verification exercise</u>: Following the first block of tests, correct VPR functional temperatures will be established and the checks described in Sections Chapter XI, Section G, sub-paras. 27(a, b and c) of this annex inclusive conducted.

## K. VE\_HD only: on completion of the test matrix

136. On completion of all testing, the GPMS and engines will be prepared for despatch to the next laboratory for testing. However, prior to testing at the first laboratory and subsequent to testing at some additional laboratories, the VPR will be returned to JRC for a performance check. This check will determine key performance parameters of the VPR.

137. These performance evaluations will be undertaken during the shipping process for the golden engine and shall not delay the test programme. The decision as to when the VPR will be returned to the calibration facility will depend on the number of participating laboratories and will be at the discretion of the project manager and golden engineer.

# XII. Data capture and presentation in correct format

138. All data will be presented in a format compatible with Microsoft Excel. A standard spreadsheet for these data will be provided, prior to the commencement of testing, by the project manager.

#### A. Regulated emissions

139. Summary regulated gaseous emissions, carbon dioxide and fuel consumption data shall be quoted as g/kWh according to current European regulations. Data will be presented from complete ETC, WHTC cycles as well as WHSC, ESC and steady states where appropriate.

#### **B.** Particulate mass

140. Summary particulate mass data shall be quoted as g/kWh according to current European regulations. Data will be presented from the complete emissions cycles.

#### C. Particle number

141. Summary particle number data shall be quoted as number/kWh and number/s. Data will be presented from individual urban, rural and motorway phases and from the combined, ETC and WHTC cycles. Data from the ESC and WHSC cycle shall be presented per mode in particles/s and per kWh for the weighted cycle. In addition, logged particle number data, time-aligned and synchronised with the regulated gaseous emissions shall be presented in a time-aligned format on a CD-R.

# D. Diagnostic data

142. Testbed data shall be logged continuously throughout each test at a rate of at least 1 Hz in order to provide diagnostic capability if repeatability or reproducibility of engine tests is poor. These data shall be employed to interpret catalytic activity and engine management control. All logged data shall be presented in a time-aligned format on a CD-R. As a minimum these data shall include:

- (a) engine speed and torque
- (b) intake, exhaust and catalyst temperatures and pressures
- (c) coolant and oil temperatures and pressures
- (d) DPF backpressure
- (e) dilute gaseous emissions and CVS flow rate
- (f) raw gaseous emissions and exhaust / air / fuel flow rate
- (g) partial flow dilution system sample flow rate and split ratio

# Appendices

# Appendix 1a

# Lubricant change and flush protocol (VE\_HD)

- (a) Commence oil drain
- (b) Allow drain to continue until flow stops
- (c) Fill with  $15 \pm -1$  litre of replacement oil
- (d) Start engine and idle (ESC Mode 1) for 30 seconds
- (e) Run the engine up to ESC Mode 4
- (f) Allow the engine speed and load to settle for ~15 seconds
- (g) Return to idle and allow to settle ~15 seconds
- (h) Repeat the ESC Mode 4 / Idle cycling 5 times
- (i) Drain the oil down and remove the oil filter
- (j) Refill with 15 litres of new oil and new filter filled with oil

Lubricant preconditioning/ageing:

- (a) 3 times: 10 minutes at Mode 10 (ESC) plus 10 min at low load (800 min<sup>-1</sup>/200 Nm)
- (b) 4 times: 15 minutes at Mode 10 (ESC) plus 5 min at idle.

# Appendix 1b

# Lubricant change and flush protocol (RR\_HD)

(To be added during the programme if required).

# Appendix 2a

# **Certification of PNC performance**

Lower Particle Size Detection Limit Characteristics (Counting Efficiency)



#### D10, D25, D50 and D90 Values

DIAMETER IN NM	3010D CPC BASED ON CURVE FIT EQUATION	PMP CPC RANGE
D10	16.9	16 ± 1
D25	19.4	18 ± 2
Dso	23.5	23 ± 3
Dao	37	37 ± 4





Electrometer Reading, part/cc

# Appendix 2b

# Specification of reference fuel (RF06)

APPELATION : gazole type CEC RF 06-03 PMP			Reference of analysis : 9460		
N° of samples : 0	N° of batch : B7277051		Date: 05/06/2007		
COMPLIANCE CERTIFICATE		BULLETIN OF ANALYSIS		×	
1	DIESEL FUEL	RESULT	S UNITS	METHODS	
PHYSICAL DATA Density 15 °C Viscosity 40°C		834.9 2.654	kg/m3 cSt	EN 190 3676-98 ASTM D 445	
DISTILLATION					
IBP 5 % Vol 10 % Vol 20 % Vol 40 % Vol 50 % Vol 50 % Vol 80 % Vol 80 % Vol 95 % Vol 95 % Vol 95 % Vol 50		171 196 204 224 262 277 291 304 318 334 346 357 33.8 96.1 53.1	ໍດີ ເດີດ ເດີດ ເດີດ ເດີດ ເດີດ ເດີດ ເດີດ ເ	ASTM D 95 ASTM D 95	
COMPOSITION		0,	0	LIN 22710	
Poly-aromatics		5.1	%Mass	IP 391	
Cold BEHAVIOU Cold Filter Plugging Point	R nt (CFPP)	-17	°C	EN 116, NF M 07042	
COMBUSTION Lower Calorific Value %C, %H, %O		46.4 86.7/13.2:	M.Ukg ⊲ <b>0.2</b> %Mass	ASTM D 4858 GC / Calculated	
COMPLEMENTAR' Oxidation stability Copper Strip Corrosion a Sulfur content Conradison Carbon Resi Ash content Neutralisation Number Sediment content Fatty Acid Methyl Ester Water content HFRR 60°C	Y DATA at 50 °C due on 10% Dist.Residue	2 1 7 <0.2 <0.001 <0.02 6 <0.2 30 310	g/m3 merit mg/kg %Pds/%mass %Pds/%mass mg/kg %Mass mg/kg µm	ISO 12205 ISO 2100 ISO 2100 ISO 4260 / ISO 8754 ISO 6245 ASTM D 974 ASTM D 974 ASTM D 974 ISO 1237 ISO 015 12156	

# **Appendix 3**

### References

- [1] Conclusions on Improving Particulate Mass Measurement Procedures and New Particle Number Measurement Procedures Relative to the Requirements of the 05 series of amendments to Regulation No. 83. http://www.unece.org/trans/doc/2004/wp29grpe/TRANS-WP29-GRPE-48-inf11r1e.pdf
- <sup>[2]</sup> A working document for the United Kingdom Department for Transport presenting an updated and restructured version of Regulation No. 49, Annex 4.
  - http://www.unece.org/trans/doc/2004/wp29grpe/PMP-2004-13-03e.doc
- [<sup>3</sup>] Regulation No. 83: Vehicle Emissions Legislation; Light duty vehicles and PCVs (GVW < 3.5 tonnes). http://www.unece.org/trans/main/wp29/wp29regs/r083r3e.pdf
- [<sup>4</sup>] Regulation No. 49: Vehicle Emissions Legislation; Heavy Duty Vehicles (GVW > 3.5 tonnes). http://www.unece.org/trans/main/wp29/wp29regs/r049r3e.pdf
- [<sup>5</sup>] UN-GRPE PMP Phase 3 Inter-laboratory Correlation Exercise: Framework and Laboratory Guide, A Document For The United Kingdom Department for Transport. RD 04/80801.5 Jon Andersson and David Clarke
- [6] http://ies.jrc.ec.europa.eu/uploads/fileadmin/Documentation/Reports/Emissions\_and\_Health/EUR\_2006-2007/EUR\_22775\_EN.pdf
- [<sup>7</sup>] Giechaskiel B., Dilara P., Andersson J. (2008). Particle Measurement Programme (PMP) light duty inter-laboratory exercise: Repeatability and reproducibility of the particle number method. Aerosol Sci. Technology, 42, 528-543
- [<sup>8</sup>] Giechaskiel B., Dilara P., Sandbach E., Andersson J. (2008). Particle Measurement Programme (PMP) light duty inter-laboratory exercise: Comparison of different particle number measurement systems. Measurement Sci. Technology, 19, 095401
- <sup>[9</sup>] http://www.unece.org/trans/main/wp29/wp29regs/r083r3a2e.doc
- [<sup>10</sup>] Exploratory experiments SAE 2009-01-1767
- [<sup>11</sup>] Particle Measurement Programme (PMP) Heavy duty (HD) Inter-laboratory Exercise. Exploratory work at JRC (Oct' 07 -Dec'07). B. Giechaskiel, S. Alessandrini, F. Forni, P. Sinuez, M. Carriero, G. Martini
- [<sup>12</sup>] UN-GRPE PMP Phase 3 Inter-laboratory Correlation Exercise: Updated Framework and Laboratory Guide for Heavy Duty (HD) Engine Testing A Document For The United Kingdom Department for Transport. Ricardo Document RD 07/578701.2a. Jon Andersson and David Clarke
- [<sup>13</sup>] ISO 16183:2002 Heavy duty engines -- Measurement of gaseous emissions from raw exhaust gas and of particulate emissions using partial flow dilution systems under transient test conditions. http://www.iso.org/iso/catalogue\_detail.htm?csnumber=32152
- [<sup>14</sup>] http://www.horiba.com/automotive-test-systems/products/emission-measurement-systems/dilutionsampling-systems/details/mdlt-1300t-876/
- [<sup>15</sup>] AVL SPC 472 Smart Sampler: http://www.avl.com/wo/webobsession.servlet.go/encoded/ YXBwPWJjbXMmcGFnZT12aWV3JiZub2RlaWQ9NDAwMDIwMTc0.html
- [<sup>16</sup>] http://www.controlsistem.it/data/automotive/testrig/pss20.swf
- [<sup>17</sup>] http://sierrainstruments.com/emissions/bg3.html
- [<sup>18</sup>] United Kingdom Particulate Measurement Programme (PMP): A Near US 2007 Approach To Heavy Duty Diesel Particulate Measurements - Comparison With The Standard European Method. Jon D Andersson; David P Clarke; James A Watson. SAE 2004-01-1990
- [<sup>19</sup>] Wei Q., Oestergaard K., Porter S., Asano I, Adachi M. and Rahman M., 2006. Real-time measuring system for engine exhaust solid particle number emission – Design and Performance. SAE 2006-01-0864
- [<sup>20</sup>] Johnson K. C., Durbin T. D., Jung H., Chaudhary A., Cocker D. R., Herner J. D., Robertson W. H., Huai T., Ayala A. and Kittelson D., 2009. Evaluations of the European PMP methodologies during anroad and chassis dynamometer testing for DPF equipped heavy duty diesel engines. Aerosol Science and Technology, 43, 962-969.
- [<sup>21</sup>] Johnson, Kent C., Thomas D. Durbin, Heejung Jung, Ajay Chaudhary, David R. Cocker III, Jorn D. Herner, William H. Robertson, Tao Huai, Alberto Ayala, and David Kittelson, 2009. Evaluation of the

European PMP Methodologies during On-Road and Chassis Dynamometer Testing for DPF Equipped Heavy Duty Diesel Vehicles, Aerosol Science and Technology, 43:962-969, 2009.

- $[^{22}]$ Johnson, Kent C., Thomas D. Durbin, Heejung Jung, Ajay Chaudhary, David R. Cocker III, Jorn D. Herner, William H. Robertson, Tao Huai, Alberto Ayala, and David Kittelson, 2009. Evaluation of the European PMP Methodologies during On-Road and Chassis Dynamometer Testing for DPF Equipped Heavy Duty Diesel Vehicles, Aerosol Science and Technology, 43:962-969, 2009.
- $[^{23}]$ Hines W., Montgomery D., Goldsman, D. and Borror C., Probability and Statistics in Engineering. 4th edition, John Wiley and Sons, 2003.
- $[^{24}]$ Armstrong S., 2007. Significance tests harm progress in forecasting. International Journal of Forecasting, 23 321-327.
- $[^{25}]$ Mamakos A., Ntziachristos L. and Samaras Z., 2004. Comparability of particle emission measurements between vehicle testing laboratories: a long way to go. Measurement Science and Technology, 15, 1855-1866.
- $[^{26}]$ Penetration Calibration and Verification for the Solid Particle Counting System with Polydisperse and Monodisperse Particles. Qiang Wei, Mike Akard, Ichiro Asano, Montajir Rahman. SAE 2008-01-1178
- $[^{27}]$ Real-time measuring system for engine exhaust solid particle number emission - Performance and Vehicle tests. Oiang Wei, Asano Ichiro, Masayuki Adachi, Rahman M. Montajir, Takeshi Kusaka, Yuichi Goto. SAE 2006-01-0865
- $[^{28}]$ Calibration of Condensation Particle Counters. Wei Liu, Brian L. Osmondson, Oliver F. Bischof, and Gilmore J. Sem. SAE 2005-01-0189
- Personal communication between J. Andersson and Hans-Georg Horn (TSI Europe)
- [<sup>30</sup>] Final Report for DfT on testing in the PMP Phase 3 Heavy duty Validation Exercise. Ricardo Document 09\_110201\_2
- $[^{31}]$ Particle Measurement Programme (PMP) Light duty Inter-laboratory Exercise: Repeatability and Reproducibility of the Particle Number Method. Barouch Giechaskiel, Panagiota Dilara, Jon Andersson. Aerosol Science and Technology, Volume 42, Issue 7 July 2008, pages 528 - 543
- $[^{32}]$ http://www.avl.com/wo/webobsession.servlet.go/encoded/ YXBwPWJjbXMmcGFnZT12aWV3JiZub2RlaWQ9NDAwMDM5MzUy.html
- http://www.dekati.com/cms/dmm
- Maricq MM, Podsiadlik DH, Chase RE 1999. Gasoline vehicle particle size distributions: Comparison of steady state, FTP, and US06 measurements. Environmental Science & Technology 33(12):2007-2015.
- $[^{35}]$ Calibration and Validation of Various Commercial Particle Number Measurement Systems. Barouch Giechaskiel, Massimo Carriero, Giorgio Martini, Alois Krasenbrink, Daniel Scheder
- [<sup>36</sup>] Giechaskiel B., Ntziachristos L. and Samaras Z., 2004. Calibration and modelling of ejector dilutors for automotive exhaust sampling. Measurement Science and Technology 15, 2199-2206.
- [<sup>37</sup>] [<sup>38</sup>] Personal communication between J. Andersson and Tim Johnson (Corning Inc.)
- ECE/TRANS/WP.29/2009/114, Annex 4B