To:	PMP Working Group	From:	Chris Parkin
			Cleaner Fuels & Vehicles 4
		Location:	
		Tel:	2958
		Fax:	2605
		Ref: (Paper)	
		Date:	13 June 2007
		Copies:	

MINUTES OF 19TH PMP WORKING GROUP MEETING (6TH JUNE 2007)

Morning Session – Calibration Procedures

1. The morning session was dedicated to a detailed discussion of the draft calibration procedure documents for Particle Number Counters and Volatile Particle Removers.

2. France noted that ISO TC 22 have only recently been consulted on TC24's work on PNC calibration procedures. TC 22 have yet to take a view on this work, but could possibly reject application of the procedures for automotive use.

3. The representativeness of the 'emery oil' used for the primary calibration procedure was discussed. A full specification of the emery oil was given. Ricardo commented that TSI use it as it provides monodisperse, spherical particles of chemical composition representative of synthetic lube oil particles. Cambustion added that it has the advantage over CAST particles of containing few double charged particles.

4. It was agreed that the Chairman would seek data from Grimm on their primary calibration methods for inclusion in the background.

5. OICA commented on the lack of PNC calibration between zero and 2000cm-3. The chairman and AEA commented that there were no reasons why a CPC would behave in a linear manner between 2000-10000cm-3 and at zero but non-linearly between 0 and 2000cm-3. OICA commented that they had seen 18% differences in comparing TSI and Grimm CPCs, however it transpired that this was at high concentrations. Ricardo commented that TSI's calibrations were traceable to NIST standards. EMPA commented that an error of this magnitude was likely to be a flow rate error and that it was important to check the CPC flow rate with a separate flow rate meter (as per the daily checks in the proposed Reg 83 amendment and CPC calibration procedure).

6. On the secondary ("transfer standard") procedure the importance of ensuring the same sample to both CPCs was emphasised. EMPA commented that a neutraliser should be used both up and downstream of the electrostatic classifier. Cambustion commented that the need for this would depend upon the aerosol generation method, but that it was a sensible precaution.

7. OICA expressed the view that we should await the outcome of the ISO work on CPC calibration procedures. AEA explained that the proposed procedures were a

subset of those being developed by ISO and were not incompatible. The chairman commented that since regulators had no control over the timing of ISO's activity the group could not simply await the outcome of their work.

8. France commented that for use as a reference in secondary calibration a CPC should be demonstrated to be 2-3 times better than the normal CPC requirements in terms of linearity and repeatability. TUV agreed. Cambustion commented that the tolerance applied to CPC's should equal the sum of the potential variability in electrometer and reference CPC. JRC commented that there were statistical methods for accounting for errors when comparing two instruments of equivalent variability and that these had been demonstrated in the Inter-Laboratory Correlation Exercise (ILCE), however the slope and R^2 of the regression analysis (if both close to 1 e.g. <+/-5%) are adequate to show that the CPC that is being tested functions properly.

9. OICA queried the lack of calibration requirements for the CPC cut off characteristic. The chairman responded that CPCs generally included diagnostics on all of the parameters that affected the cut off curve and hence periodic calibration was unnecessary. It was agreed however that a requirement to check the 23nm D50 performance would be added in the case of CPCs with no such diagnostics.

10. It was highlighted that the penultimate reference on p.15 was a 'Private Communication'. AEA believe that this has now been published and will update the reference accordingly.

11. It was agreed that diluter calibration procedures needed adding to the Volatile Particle Remover (VPR) calibration procedures. Horiba had commented that NO was the best gas to use since (unlike CO) the analyser is inherently linear and gives good accuracy at concentrations of a few ppm. Using propane with an FID also permits wide-range linear response although there can potentially be issues with background concentrations of hydrocarbons in the dilution air.

12. It was discussed whether an additional CPC with a lower size cut-off was needed to perform the 30nm particle penetration efficiency measurements. It was agreed that the text will be amended to clarify that use of a single PNC with the standard PMP cut-off characteristic measuring sequentially up and downstream was acceptable.

13. The properties of the aerosol for volatile removal efficiency measurement were discussed in detail. It was concluded that a monodisperse aerosol GSD <1.2 of 30nm size with an upstream concentration of >10,000cm-3 should be used.

14. OICA commented that the procedures were control procedures rather than calibrations. It was agreed that this was the case and that no calibration factor was being derived or applied, indeed this is explicitly prohibited in the Reg 83 proposal.

15. The correct concentrations at which to measure solid particle penetration efficiency were discussed. It was felt that a concentration representative of typical post DPF levels was appropriate in view of the intended application of the measurement procedure to vehicles at or around this level of emissions. Ricardo commented that based on experience from the ILCE this equated to a downstream concentration of

around 1000cm^{-3}. However this is based on a polydisperse exhaust aerosol. At the monodisperse sizes used for penetration measurement inlet concentrations of near 10000cm-3 would be similar to DPF exhaust levels. Therefore inlet concentrations for the penetration efficiency measurements should be as close to 10000cm^{-3} as possible, but below in order to be within the CPC single particle counting measurement range. The VPR should be operated at settings typical of those used for DPF exhaust measurement.

16. OICA queried whether Matter and SPCS systems used in the ILCE met the revised penetration efficiency requirements. The chairman understood that the penetration efficiency data provided by Horiba was for systems of the specification used in the ILCE but agreed to ask Horiba how the production SPCS compared to those used in the ILCE in terms of specification and penetration efficiency. Matter agreed that their dilution factors were calculated using 91nm particles so some particle losses were effectively corrected out in their penetration efficiency data. However they have also performed gas calibrations of their diluters with CO2 showing <5% difference from particle dilution factors so, even adjusted for this, their system does meet the revised penetration efficiency requirement. AVL commented that their development system penetration efficiency data was similar to that shown by Matter.

17. The option of using only downstream measurements for volatile particle removal efficiency with a cold, then heated VPR was discussed and will be added. It was noted that both hot and cold dilution factor calibration will be required in this instance.

18. In summary the following amendments to the draft calibration procedures were agreed.

<u>General Amendments</u>

- Add statement of system performance requirements at start of procedures
- Redraft documents as procedures with examples moved to an Annex

<u>Amendments To PNC Calibration Procedures</u>

- Amend Section 4, para 4 to allow/require coincidence correction in line with Reg 83 proposal
- Consideration will be given to adding an upper limit on allowable coincidence correction factors.
- Add definition of ‘emery oil’ used in primary calibration and GSD of aerosol used
- Alternative primary calibration aerosol material and production should be allowed provided they meet a GSD of <u>≤</u>1.2.
- Specify use of a neutraliser up and downstream of the electrostatic classifier.
- Consider how requirements can be tightened for reference CPCs used for secondary calibration or statistical methods used to account for similar variability in reference PNC and the PNC under calibration.
- 23nm D50 performance check to be added for PNCs not featuring diagnostics on parameters affecting cut-off characteristic.
- Replace reference to ‘Private Communication’ with published reference.

Amendments To VPR Calibration Procedures

- Diluter calibration procedures will be added based on gas calibration using NO.
- It will be clarified that the system description in section 2 is the recommended system and that alternative VPRs meeting the performance specification are allowed.
- Section 3.1.1 will be updated to reflect the revised penetration efficiency characteristic specified in Informal 10.
- The minimum particle concentration in section 3.1.1 would be increased to as close to (but below) 10000cm^{-3} as possible upstream of the VPR to reduce errors in penetration efficiency calculation and provide for calibration at typical concentrations for DPF exhaust. Penetration efficiency measurements should be made with the VPR operating at typical settings for DPF vehicle measurement
- Minimum concentrations and a requirement that the VPR be calibrated at typical operating settings for DPF vehicle measurement as specified in the Reg 83 proposal will be added to 3.1.2
- The calibration aerosol in 3.1.2 will be specified as monodisperse (GSD <1.2), 30nm particles of >10000cm^{-3} concentration.
- Volatile particle removal efficiency check will be modified to allow use of downstream measurements only using the VPR unheated and then heated. Dilutor calibration under both hot and cold conditions will be required in this case.
- Section 4.4 will be modified to accommodate cold v hot downstream measurements as a means of determining volatile removal efficiency.

Afternoon Session

19. The chairman presented the revised VPR solid particle penetration efficiency characteristic based on additional measurements from Matter, Horiba and AEA measurements using diesel exhaust (GRPE-PMP-19-1)

20. In response to a question from Germany the chairman reported that the morning session had identified a number of improvements to the draft calibration procedure documents. These would be updated and the chairman anticipated there would be a further meeting to discuss the updated documents once available.

21. OICA gave a presentation (GRPE-PMP-19-2) on comparison measurements using Matter + TSI equipment v the FPS + Grimm CPC. These included vehicle exhaust measurements, and comparison of the two different CPCs using CAST particles. The FPS showed significantly higher results than the Matter system. Comparison of the CPCs showed an 18% difference. Ricardo asked whether the Grimm CPC had been calibrated to the NIST traceable standard employed by TSI (OICA to verify).

22. Finland gave a presentation (GRPE-PMP-19-3) on behalf of Dekati covering the specification and performance of the FPS systems used in the ILCE and subsequent developments. The level of the particle number results from the systems used in the ILCE were unreliable due to the dilutors operating outside their calibrated temperature range. In addition the high residence time in the evaporation tube resulted in excessive smoothing of particle emissions. Dekati have developed a revised system which eliminates these issues, is more accurate and easier to operate.

23. Finland asked whether measurement systems meeting the performance requirements but not the recommended system description were acceptable and asked who would approve such systems for use. The chairman responded that all systems meeting the performance specification would be acceptable and that instrument manufacturers should perform their own calibration measurements to demonstrate their systems meet the performance requirements.

24. The chairman explained that the proposal to insert the PMP measurement procedures into Reg 83 had been revised from GRPE 53 Informal 3 in line with discussions at the meeting on 20th Feb and with the VPR solid particle penetration efficiency specified. The procedures were now proposed for insertion into Reg 83 as an unreferenced Annex.

25. France queried the necessity of adopting this now and felt that the situation differed from that of the adoption of WHDC procedures in Reg 49 as unreferenced Annexes. The chairman did not see a difference. Germany felt that there were outstanding questions that needed to be addressed before the particle number procedures were adopted in Reg 83, but that there was an urgent need to adopt the revised particulate mass measurement procedures. Italy agreed with Germany. The chairman queried why Germany felt that the ILCE was sufficient to demonstrate the readiness of the revised particulate mass measurement procedure but not that of the particle number measurement procedure. Germany responded that PM was simply a development of a well established procedure whereas PN was an entirely new system of measurement.

26. The chairman showed a slide comparing the ILCE results from the Golden System with those from Alternative Systems as a means of showing how the results from a Round Robin with no Golden System might have looked. JRC commented that if the FPS results were removed (due to the system used being known to be inaccurate) the Golden and Alternative system results were within 10%. They also commented that the PM repeatability was favoured by the outlier rejection criteria used in the ILCE.

27. OICA noted that they would submit comments on the Reg 83 proposal dealing with background correction for PN, correction of referencing errors, clarification of filter paper face temperature limits in regeneration tests, use of the charcoal dilution air scrubber and D10 CPC inlet specification.

28. Switzerland supported the proposal to amend Reg 83 now.

29. AECC presented the results of their Heavy Duty particle measurement work (GRPE-PMP-19-4). They saw a 3 order of magnitude reduction in particle numbers across the DPF resulting in levels of $4x10^{11}$ /kWh (ETC) and $5x10^{11}$ /kWh (WHTC). Peak CVS concentrations were 70000cm-3, reducing to around 70cm-3 post DPF.

30. Partial flow PM measurements gave maximum filter masses of 41 μg on the ESC (~12mg/kWh), but background subtraction reduced PM to zero. Typical mass emissions from WHTC and ETC were 1-2mg/kWh. Full flow PM measurements were compromised by contamination from the sample pump, possibly because a

double sample flow was being extracted in order to make regulatory and PMP PM measurements side-by-side.

31. JRC gave an update on the Heavy Duty validation testing (GRPE-PMP-19-5). Their hardware and software upgrades are nearly complete. Testing is expected to start in the second half of June. Delivery of the fuel for the programme is still awaited. Testing will last until the end of August. The Test Protocol will then be finalised and testing can commence at the second Validation lab and first Round Robin lab.

Chris Parkin
PMP Chairman