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MINUTES OF PMP 8th & 9th OCTOBER MEETINGS

1. Discussions on 8th October focussed on presentation and discussion of the results of the calibration measurement exercise, amendments to the Volatile Particle Remover (VPR) calibration procedure and amendments to the Particle Number Counter (PNC) calibration procedures. On the 9th October discussion on the PNC calibration procedure were concluded and the proposal to amend Regulation 83 was discussed section by section.

Calibration Measurement Results

2. EMPA presented the results of calibration measurements at both EMPA and METAS on the same 2 PNCs and EMPA's homemade, fixed dilution VPR. AEA presented secondary calibration measurement data on one PNC and calibration data on a Matter MD19 VPR across its full range of dilution settings. The chairman presented an overall summary of the data received.

3. EMPA and METAS made both primary and secondary PNC calibration measurements although noting that the reference electrometers available to them were not calibrated for this purpose. Primary calibrations at the two labs agreed well, giving gradients of just over 0.9, although the counters did not feature coincidence correction which would have increased gradients to around 0.96. EMPA noted that their measurements gave a truer indication of repeatability than those of METAS, which involved repeat measurements at each aerosol concentration before moving on to the next rather than completing one full set of calibration measurements at all concentrations before repeating measurements.

4. EMPA also checked the PNC cut-off characteristic noting repeatability within the D50 tolerance but a shallower than expected cut-off gradient. TSI noted this was probably due to the aerosol material used (Palladium rather than emery oil). OICA also noted that recent data suggested that cut-off performance was material dependent. This suggests it will be necessary to more closely define acceptable aerosols for demonstrating the cut-off performance.

5. Secondary calibration results showed better repeatability, and if the reference PNC is calibrated to the electrometer reading (as proposed), very good agreement with the primary method.

6. EMPA and METAS both obtained extremely good repeatability on VPR particle concentration reduction factors (f_r), but around a 10% offset between the two labs. EMPA suggested this may have been due to the different aerosol material

and generation method, hypothesising that there may have been a small proportion of volatile particles in the CAST aerosol used by METAS. EMPA reported only slightly higher (~5%) f_r (and hence particle losses) at the 30nm size than the 50 and 100nm sizes.

7. EMPA measurements on VPR volatile particle removal efficiency were consistently well above the 99% requirement, although they noted that if large volatile particles (e.g. 50nm) were used that the efficiency was lower.

8. EMPA suggested areas for further improvement included correction for multiply charged particles, specifying a tolerance on particle sizes used for the cut off characteristic check (to account for tolerance of the electrostatic classifier), clarifying data acquisition methods (Matter suggested that the 'D command' should be used to download the point by point concentration data from the PNC) and specifying pressure conditions at the PNC inlet.

9. AEA presented repeat calibration data for a PNC by the secondary (reference PNC) method. Repeatability was similar to EMPA's measurements. Cut off characteristic validation checks were made at the 23nm (50%) point showing good repeatability.

10. AEA measurements of the VPR f_rs showed repeatability of 2% or better for f_rs up to 1150, worsening at higher settings in part due to very low downstream particle concentrations at the high dilution settings. It was noted that the maximum dilution setting used in the PMP inter-lab for GDI vehicles was around 600 (i.e. well within the range for which repeatable calibration was demonstrated), with DPF dilution settings being much lower. Matter commented that in performing the calibration potentiometer settings should be read electronically.

Accumulation of Errors Analysis

11. NPL presented their accumulation of errors analysis report. They explained that the method used was the method defined in the ISO Guide to the Expression of Uncertainty in Measurement (GUM). This is the standard approach used by metrology organisations. The analysis was intended to represent realistic rather than worst case differences between measurement systems.

12. They noted that the main contributor to uncertainty was the PNC counting efficiency and that total measurement system uncertainty was around 15%. Accurate measurement of the VPR volatile removal efficiency was difficult using the 2 PNC method due to the low outlet concentrations, the hot-cold method being better, but absolute accuracy was not essential given that VPR performance appeared to be well above the 99% requirement. EMPA commented that the hot-cold method was subject to error due to drift in source aerosol concentration.

13. Concawe asked how significant an error multiply charged particles might introduce into calibrations. NPL responded that their experience was that this was up to 5% but could be controlled by controlling the aerosol GSD. TSI added that selection of the aerosol generation method could also be used to control this, aerosols produced by evaporation and condensation had low numbers of multiply charged particles.

14. The report will be updated to clarify the approach used and include a ‘fishbone’ diagram. The report will also need to take into account changes agreed to the PNC calibration approach and comment on the impact of taking measurements at dilution settings required for GDI exhaust measurement.

VPR Calibration Procedure

15. The VPR calibration procedure document was discussed section by section. The following amendments were agreed;

- In section 2.1 the ‘within 20%’ requirement on particle concentration reduction factors at 30nm and 50nm measurements would be amended to require f_r to be no more than 30% greater at 30nm, no more than 20% greater at 50nm than the 100nm value and no more than 5% less at either size. The -5% requirement was required to allow for measurement uncertainty. Opinions were divided as to whether 30% should be allowed at 30nm. The Chairman ruled that 30% should be allowed since the intention was to set requirements which were demonstrably achieved by the available measurement systems (rather than aspirational requirements for future systems). The available data for Horiba and Matter systems supported the need for a 30% allowance.

- On page.2 the potential for confusion between reference 1 and footnote 1 will be removed

- In section 2.2 “(DFtot)” and the final sentence of the second paragraph “DFtot is calculated……” will be deleted in both the Reg 83 proposal and hence the VPR procedures document.

- It will be clarified that particles of at least 30nm size must be used to demonstrate compliance with the 99% volatile particle removal efficiency requirement, but that it is not required to meet this efficiency for all particle sizes above 30nm.

- In section 2.2 the tolerance on the evaporation tube temperature will be deleted, leaving the requirement that the temperature must be controlled within the range 300-400C.

- In section 3, second bullet the requirement for residence time in pathways to up and downstream instruments to be comparable will be supplemented by a requirement that pathways are ‘as short as possible’ in order to reduce agglomeration losses at high (upstream concentrations). The sentence ‘It may also be necessary … between the instruments.’ Will be deleted.

- In section 3, third bullet, use of conductive tubing shall be required.

- A Glossary will be added, including amongst other terms a definition of particle concentration reduction factor.

- 3.1.1 will be redrafted to account for the possibility that the VPR may have a fixed dilution factor.

- In Table 1 the electrostatic classifier will be moved to the mandatory section.

- OICA will propose some text (having considered the text of the previous draft procedures) on gas dilution factor calibration for the purposes of more frequent 'quality control checks' on the VPR.

- OICA requested that the particle concentration factor validation criteria be tightened from +/-10% of the calibration value, at least for factors up to 1000. This will be revisited when further data is available on the influence of aerosol material on the measurement of particle concentration reduction factor e.g whether this explains the EMPA-METAS offset referred to in paragraph 6 above.

- Use of the second person in section 5.1 will be removed for consistency with the remainder of the procedures.

- In the 2 PNC method in sections 5.1 and 5.2 a requirement shall be inserted to time align up and downstream measurements accounting for VPR residence time.

- PNC zero check and VPR leak check criteria shall be reduced to $\leq 0.2 cm^{-3}$ and $\leq 0.5 cm^{-3}$ respectively throughout the document.

- In the following sections the sampling time shall be reduced to 1 minute or the time required for a cumulative sample of 10,000 particles, whichever is greater.

    - section 5.1 points s & y
    - section 5.2 Method 1 point r and Method 2 points o and r
    - section 5.3
    - section 5.4.1
    - section 5.4.2
    - section 6.1 point u
    - section 6.2, Method 1, points q & r and Method 2 points o & r
    - section 6.3
    - section 6.4.1
    - section 6.4.2
- The Note at the end of 5.1 shall refer to the cumulative number of particles sampled.

- A 35C upper limit on the room temperature in which the calibration is conducted will be added.

- In section 5.3 equation (1) will be amended to refer to $N_{in}(d)$ and $N_{out}(d)$. The meaning of 'd' will be inserted. Inequalities (3) and (4) and the preceding sentence will be amended in line with the particle concentration reduction factor requirements specified above. The

requirement to measure over at least 5 dilution settings will be amended to allow for fixed dilution VPRs.

- Aerosol generation references will be inserted in section 7, Grimm & TSI will supply a list of the most relevant available in English as well as German.

- References to HEPA filters of at least 99.99% efficiency will be replaced by references to HEPA filters of 'class H13 of EN 1822:1998 or better filtration efficiency'.

- A number of typographical corrections were also highlighted as being required.

PNC Calibration Procedure

16. Discussions on the PNC calibration procedure began on 8th and were completed on the 9th October. A major change in approach was agreed, this was to apply a calibration factor (restricted to the 0.9-1.1 range) to the PNCs to correct them to the measurement level of the primary calibration reference instrument. This was felt to be appropriate in view of the fact that actual PNC flow rates could differ slightly from nominal, due to the tolerance of the critical orifice, affecting reported concentrations. In addition the following amendments to the procedures were agreed;

    - In section 4, paragraph 3 the pathway equivalence criterion will be $\leq$3%

    - PNC zero check and VPR leak check criteria shall be reduced to $\leq 0.2 cm^{-3}$ and $\leq 0.5 cm^{-3}$ respectively throughout the document.

    - In section 4, paragraph 3 the requirement for residence time in pathways to test and reference instruments to be comparable will be supplemented by a requirement that pathways are 'as short as possible' in order to reduce agglomeration losses at high (upstream concentrations).

    - A reference to the procedures against which the reference electrometer must have been calibrated will be inserted.

    - In section 4, paragraph 4 use of conductive tubing shall be required.

    - Section 4.1.1 Liu and Pui (1974)[8] should be corrected to reference 10.

    - In section 4.1.2 it will be clarified that the concentration should be as close to, but below, $SPCM_{max}$ as possible.

    - The penultimate paragraph of section 4.1.3 will be corrected to refer to the reported concentrations rather than the log of the reported concentrations.

- In the final paragraph of 4.1.3 the R^2 will be clarified as being the Pearson product moment correlation coefficient for consistency with draft R83.

- Figure 3 will be amended to make it clearer that the aerosol electrometer and optical particle counter ranges overlay the CPC range.

- In section 4.1.4 a link to the draft ISO standard will be inserted.

- In section 4.2 the requirement for the reference PNC to be within 5% of the electrometer reading will be deleted. This is no longer relevant given that a calibration factor will have been applied to the reference PNC to correct it to the level of the electrometer.

- In section 4.2.1 the word 'solid' will be deleted from the first sentence.

- In section 4.3 the aerosol material used to determine the cut-off characteristic shall be specified as emery oil. Alternative materials will be allowed subject to it being demonstrated that they give equivalent counting being demonstrated. Explicit inclusion of other materials will be considered following submission of data from TSI and Grimm (who volunteered to supply data within 1 month). In particular CAST and tetracontane particles will be considered.

- A +/-1nm tolerance on the 23nm particle size will be added in section 4.3 to account for the tolerance of the electrostatic classifier in delivering particles of a particular size. The GSD will be specified as $\leq$1.1.

- The need to determine the detection efficiency of the reference PNC used for checking the cut-off characteristic will be clarified in section 4.3.

- References to HEPA filters of at least 99.99% efficiency will be replaced by references to HEPA filters of 'class H13 of EN 1822:1998 or better filtration efficiency'.

- In Table 2 the second neutraliser will be deleted.

- TSI will confirm the details of Reference 17.

- Concawe will check and confirm a specification (as opposed to typical properties) for emery oil.

- Figure 7 axes will be changed to a linear scale.

Regulation 83 Proposal

17. The text of the proposal to amend Regulation 83 was discussed section by section including discussion of written comments from CLEPA and OICA submitted at last GRPE. A number of these comments have been superseded by

the move to calibrating the VPR for particle concentration reduction factor and the PNC for gradient (detection efficiency) against the reference electrometer.

18. In addition to a number of editorial improvements, the following changes of substance to the proposal were agreed;

- Section 6.2.4 of Annex 4a: the point for extraction of a background sample will be specified as being the same as that for vehicle particulate sampling.

- Section 6.2.5 of Annex 4a: background subtraction for particle number will also be allowed for In Service Conformity testing

- Section 6.3.1 of Annex 4a: diesel pre-conditioning procedures will be applied to direct injection petrol vehicles also.

- Section 6.6.8 of Annex 4a: PNC calibration factors and VPR particle concentration reduction factors will be incorporated in the calculation of particle number emissions, dilution ratio (DR_{TOT}) will be deleted. The requirement to sample for 20 seconds after the end of the cycle will be deleted as it unduly complicates test cell procedures and does not significantly effect on the overall cycle result. Coincidence correction will be added to the calculation of mean particle concentrations.

- Section 1.3.2 of Appendix 2: the charcoal scrubber shall be made optional.

- Section 2.3 of Appendix 4: the reference filter weighing criteria will be clarified and the procedure to follow on the first day after the reference filters have been replaced will be specified.

- Sections 1.2.1 & 1.4.2 of Appendix 5: the flow Reynolds number requirement will be amended to specify Re<1700. It will be clarified that the PND_2 outlet tube (POT) is external to the VPR and not a part of it.

- Section 1.2.4 of Appendix 5: it will be clarified that gradual changes in cross-section are permissible.

- Section 1.3 of Appendix 5: a new requirement will be inserted to state that the sample shall not pass through a pump in advance of the PNC.

- Throughout Appendix 5: references to the maximum concentration at the PNC being 10,000cm^{-3} will be replaced by the concentration being below the upper threshold of the PNC's single particle counting mode.

- Section 1.3.3.2 of Appendix: a minimum dilution factor of 10 upstream of the evaporation tube will be required to ensure effective volatile particle removal.

- Sections 1.3.3.3 and 1.4.4 of Appendix 5: solid particle penetration efficiency requirements will be replaced by particle concentration factor

requirements. These will require factors for 30nm and 50nm particles to be no more than 30% and 20% higher than for 100nm particles.

- Sections 1.3.3.4, 1.4.4 & 2.3.3 of Appendix 5: 'n-C40 alkane' will be replaced by 'tetracontane ($CH_3(CH_2)_{38}CH_3$)'.

- Section 1.3.4.2 of Appendix 5: PNC counting accuracy requirements at low concentrations will be replaced with % accuracy criteria accompanied by confidence criteria. Demonstration of compliance will require Poisson statistical assessment of concentrations recorded over a period.

- Section 1.3.4.3 of Appendix 5: readability shall be amended so as not to require decimal places at high concentrations (or an excessive number of decimal places at nominal zero concentrations).

- Section 1.3.4.8 of Appendix 5: will be amended to require either internal or external application of a calibration factor to correct to the reference electrometer level. Maximum permissible coincidence correction will be specified as 10%.

- Section 1.3 of Appendix 5: a new requirement that the sum of the measurement system delay (i.e. residence) time and the PNC T90 shall not exceed 20s will be added.

- Section 1.4.4.1 of Appendix 5: the maximum dilution factor of the recommended system PND_1 will be reduced to 200. Total (PND_1xPND_2) dilution of 600 is all that is required for measurement on DPF and GDI vehicles, however OICA requested higher dilution factors to enable measurements for other purposes e.g. future OBD calibration.

- Section 2.1 of Appendix 5: a requirement for recalibration of the PNC following major maintenance will be inserted as will a requirement to check the PNC's D50.

- Section 2.1.1 of Appendix 5: PNC calibration shall be amended to require at least 6 concentrations including the nominal zero point. A calibration factor will be applied to the PNC to correct its counting efficiency to the level of the reference instrument. For calculation of both the gradient and R^2 the linear regression shall be forced through the origin. In the reference PNC case the aerosol concentrations shall include 3 points below 1000cm^{-3} with the remaining points linearly spaced across the PNC's range.

- Section 2.2 of Appendix 5: a requirement for recalibration of the VPR's particle concentration reduction factors across its full range of dilution settings when new and following major maintenance will be inserted. Periodic validation will be limited to a check at a single typical dilution setting. Concentration reduction factors at 30nm and 50nm particle sizes shall be within 30% and 20% respectively of the factor at 100nm size. Method of calculation of the factors will be added.

- Section 2.4.1 of Appendix 5: the monthly linearity check will be deleted
- Sections 2.4.2 & 2.4.4 of Appendix 5: PNC zero check and VPR leak check criteria will be reduced to $\leq$0.2cm^{-3} and $\leq$0.5cm^{-3} respectively.
- Section 2.4.3 of Appendix 5: the PNC flow check will be reduced to a monthly frequency.

Any Other Business

19. JRC summarised the status of the Heavy Duty validation programme. Upgrades on JRC's test cell had been completed. Background concentrations were now <1cm^{-3} in the full flow dilution system and <5cm^{-3} in the partial flow system. Initial testing had finalised required dilution settings on the Golden Engine and minimum DPF loading periods required to ensure stability of the engine as a particle emissions source. Delivery of the Golden Systems was expected within the next week.

20. The Chairman thanked everyone for their participation and closed the meeting.

Regards,

Chris Parkin