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MINUTES OF 7TH AUGUST PMP CALIBRATION PROCEDURES MEETING

1. OICA queried the reasons for the maximum PNC concentration of 10,000cm-3. Whilst representative of the limit to which most PNCs operate in single particle count mode, it was felt that this restricted development and use of PNCs with a higher single particle counting limit.

2. The METAS and NPL suggested approaches of calibrating for particle losses by measuring reductions in particle concentrations from VPR inlet to outlet was discussed. It was agreed that this was desirable as it reduced discrepancies between systems and involved measurement of the full system rather than individual components. Initial primary calibration would be required across the full range of dilution settings, periodic validation checks would be conducted at a typical dilution setting for DPF exhaust measurement. As a consequence gas calibration requirements can be deleted.

3. It was discussed whether the aerosol for the volatile removal efficiency check shouldn't specify a minimum GSD to ensure the VPR was challenged with sufficient mass of volatile material. However it was felt that the specified minimum concentration and minimum particle size were sufficient to achieve this.

4. OICA queried whether sodium chloride aerosol was suitable for use in solid particle penetration efficiency measurement. The chairman commented that AEA had reported similar results with back-back measurements using sodium chloride and CAST soot particles. Horiba confirmed that sodium chloride was an acceptable aerosol as did Matter with the proviso that the dilution air should be dry.

5. Grimm commented that additional references on use of aerosol generators would be helpful to users.

6. The potential for error in using 2 different PNCs for simultaneous up and downstream measurement was discussed. It was agreed that in this case the 2 PNCs should be compared at all particle sizes and concentrations prior to their use for calibrating/validating the VPR. During the calibration/validation the measurements from one PNC should be adjusted to the level of the other to eliminate this error. Where a single PNC is used alternately up and downstream a tolerance needs to be added to the repeat upstream measurement to ensure no drift in the calibration aerosol concentration during the measurement.

7. The following amendments will be made to the VPR calibration procedure document as a result of discussions.

- The text shall be clarified regarding where calibration is required and where validation is required.
- Where particle diameters are referred to it should be clarified that these are electrical mobility diameters.
- References to a maximum particle concentration of 10,000 cm^{-3} at inlet to the PNC will be replaced by a comment to the effect that the maximum concentration at PNC inlet must be below the PNC's full scale deflection in single particle count mode (e.g. in sections 1 & 3.2.1).
- VPR penetration efficiency measurement will be replaced by a requirement to calibrate the VPR for its 'particle concentration reduction factor' (f_r). At each dilution setting the overall factor will be defined as the mean of the factors for the sizes 30nm, 50nm and 100nm with a requirement that the particle concentration reduction factor at 30nm and 50nm is within 20% of that at 100nm. For primary calibration, f_r must be determined at 5 dilution settings across the full range of the VPR. In the case of VPRs with a variable PND_2 dilution factor, the calibration must be performed across a matrix of 15 dilution settings, the 5 PND_1 settings at each of 3 PND_2 nominal dilution factors including 10, 15 and an intermediate setting.
- Periodic validation will require determination of f_r at one dilution setting typical for DPF exhaust measurement (using all three particle diameters). The validation acceptance criteria will provisionally be that the validation measurement is +/-10% of f_r from the primary calibration. This will be reviewed following the calibration measurement exercise taking place over August/September.
- Low VPR downstream particle concentration measurements at high dilution settings will be subject to a Poisson statistical assessment over a specified time period to determine the concentrations to be used in calculating f_r. This is the approach currently used for measuring HEPA filter performance with CPCs. **TSI to propose text.** A check on the stability of the aerosol generator will also be conducted, **AEA to review data from ILCE measurements and propose a tolerance**.
- Gas dilution factor measurements (section 3.1.1, 4.1 & 4.4) will be deleted along with the associated equipment listed in Table 1.
- In section 3.1.2 the volatile removal efficiency should be evaluated with PND_2 at its fixed dilution factor or, if variable, at a nominal dilution factor of 10. The Total DF should be the lowest setting (worst case). The ET should be at its normal operating temperature.
- A relative humidity spec for dilution air used in conjunction with sodium chloride calibration aerosol will be added to section 3.2.1 along with some references regarding use of different aerosol generators. **Horiba to supply relative humidity spec and Grimm to supply aerosol generation references**.
- A general requirement will be added to 3.2.1 that any calibration aerosol must be stable at VPR operating conditions i.e. it should be physically and chemically

stable and its aerodynamic behaviour should not change at ET operating temperatures.

- PNC zero check criteria should be adjusted to <0.5cm^{-3} (VPR leak check criteria should be maintained at 1cm^{-3}). Both of these will be reviewed based on experience accumulated in the August/September calibration measurements.
- In the final sentence of paragraph 2 of section 4.2 'small diameter particles' should be replaced by '30nm diameter particles'. Where 2 PNCs are used the responses at all particle sizes and concentrations should be compared and measurements taken with one PNC adjusted to align with the other in order to avoid introducing errors due to offsets between the PNCs.
- In the final set of 3 bullet points in section 4.2 the procedure should be amended to take measurements with one particle size at all dilution settings before varying the particle size.
- In section 4.3 (volatile removal efficiency) Method 1, where 2 PNCs are used, the measurements from one will need to be adjusted to the level of the other.
- Aerosol stability tolerances will be added to section 4.3 based on AEA experience from the ILCE.
- Equations in 4.5 and 4.6 Method 1 will need to be amended to use f_r. Dilution can be omitted from the equation in 4.6 Method 2, the criterion need merely specify that hot concentration must be less than 1% of the cold concentration.
- Annex 1 should be changed to give an example of the new procedure. Annex 2 should be deleted.

8. OICA restated their concern that there was no PNC primary calibration below 1000cm^{-3} . Once again it was stated that there was no reason to suspect non linearity between zero and 1000cm^{-3} if the PNC was accurate at these two points and linear across the 1000-10,000cm^{-3} range. In addition sub 1000cm^{-3} concentrations make only a small contribution to the overall cycle result. The discrepancy between primary and secondary calibration method concentrations (secondary calibration including points between zero and 1000cm^{-3}) was also discussed, but retained as providing some additional reassurance regarding OICA's concern.

9. PNC T90 response time and data logging frequency were discussed. Instrument manufacturers provided helpful clarification explaining that high T90s and/or low logging frequency would not affect the integrated drive cycle result. High T90 would result in a flattening but broadening of instantaneous peaks giving the same integrated result. The logging frequency is the frequency at which the PNC reports concentration rather than the frequency at which it measures it.

10. Low size cut off performance was discussed. Foreseeable faults would result in a shifting of the curve to the right or left, but not a change of gradient. It was therefore concluded that a D50 check would be sufficient. At OICA's request this will be required for all PNCs regardless of whether they feature internal monitoring.

11. OICA queried why emery oil was used for PNC calibration. TSI explained that they found this to be convenient and give good repeatability but that any low volatility oil

could be used. Grimm preferred to use an aerosol generated from a hot wire. TSI and Grimm were requested to make and compare measurements with both emery oil and particles generated from a hot wire to contribute to the August/September calibration programme.

12. UTAC expressed reservations about the secondary PNC calibration procedure potentially introducing an additional error beyond that of the primary procedure. It was agreed that when used for secondary calibration the reference CPC measurements should be adjusted according to the results of its primary calibration in order to remove this error.

13. The necessity for a second neutraliser after the electrostatic classifier was queried. TSI commented that this had been the subject of some debate, but that a number of recent studies suggested that it was not necessary. AEA commented that they would be investigating this as part of their measurement in the August/September calibration programme.

14. References to the standards to which electrometers and electrostatic classifiers were requested.

15. The following amendments will be made to the PNC calibration procedures.

- The text shall be amended to clarify that PNC performance is validated rather than calibrated.
- References to a maximum particle concentration of 10,000 cm^{-3} at inlet to the PNC will be replaced by a comment to the effect that the maximum concentration at PNC inlet must be below the PNC's full scale deflection in single particle count mode.
- Where particle diameters are referred to it should be clarified that these are electrical mobility diameters.
- PNC T90 response time will be reduced to 5s, PNC averaging time and data logging frequency will also be amended. **TSI & Grimm to advise on suitable figures**.
- Section 4 paragraph 2 penultimate sentence 'It may be necessary…' should be deleted. It is not practical to compensate for this nor is it appropriate, the PNC should be considered as a complete unit.
- Section 4 paragraph 4 should be clarified, stating that '…"coincidence correction" is required and should be applied…'.
- A detailed description of the primary calibration procedure will be added to section 4.1 including references to the standards against which electrometers should be calibrated. The procedure should include a check to confirm that multiply charged particles are not present (Grimm suggested a simple check involving doubling the electrostatic classifier voltage).
- The validation check on the cut-off characteristic D50 will be applied to all PNCs regardless of internal diagnostics.
- A maximum permissible coincidence correction, provisionally of 10%, will be specified. **TSI and Grimm to confirm**.

- In the first bullet point of section 4.1.2 the modal diameter should be specified to be in the range 50-100nm to ensure that the particle size is sufficiently above the PNC's D90 to ensure maximum counting efficiency.
- For the purposes of using a PNC as a transfer standard for secondary calibration, but not for its use for vehicle exhaust measurement, a calibration factor should be applied to its readings to align them with the counting performance of the electrometer against which it was primarily calibrated. This is to ensure no additional errors are introduced relative to the primary calibration measurement.
- In section 4.2.2 Secondary Method (B) R^2 should be calculated from the logs of concentration.
- In section 4.2.2 PNC zero check criteria should be adjusted to $<0.5cm^{-3}$. This will be reviewed based on experience accumulated in the August/September calibration measurements.
- In section 4.2.2, 'Secondary Method B' the text '… to stabilise at each electrostatic classifier voltage…' should be corrected to read '…to stabilise at each dilution setting…'.
- A reference to the standards against which the electrostatic classifier should be calibrated should be inserted.
- A check on the equivalency of losses in the pipework to each PNC in the secondary method should be included involving swapping the connections. A check at a single high concentration is sufficient.

The following complementary amendments to the Regulation 83 amendment will be required.

- Where particle diameters are referred to it should be clarified that these are electrical mobility diameters.
- References to a maximum particle concentration of 10,000 cm^{-3} at inlet to the PNC will be replaced by a comment to the effect that the maximum concentration at PNC inlet must be below the PNC's full scale deflection in single particle count mode.
- A maximum permissible PNC coincidence correction, provisionally of 10%, will be specified. **TSI and Grimm to confirm**.
- VPR maximum dilution factors will be deleted and replaced by a requirement that these be sufficient to reduce particle concentrations at VPR outlet to below the full scale deflection of the PNC in single particle count mode.
- VPR penetration efficiency requirements will be replaced by a requirement to calibrate the VPR for its particle concentration reduction factor. At each dilution setting the overall factor will be defined as the mean factor for the sizes 30nm, 50nm and 100nm with a requirement that the particle concentration reduction factor at 30nm and 50nm is within 20% of that at 100nm.
- Diluter calibration requirements will be deleted

- PNC T90 response time will be reduced to 5s, PNC averaging time and data logging frequency will also be amended (TSI & Grimm to advise on suitable figures)
- It will be clarified that the data logging frequency is the frequency at which the PNC reports particle concentrations rather than the frequency at which it measures them.
- A requirement will be added that the VPR and PNC should be repeat calibrated after any major maintenance will be added.
- A requirement for periodic validation to include a check on the cut-off characteristic will be added.
- References to the calibration procedures will be added as footnotes.

16. The Chairman explained the objectives of the forthcoming August/September calibration measurement exercise to be threefold, to gather data on the repeatability of the calibration procedures, to demonstrate their practicality for use in a type approval lab and to identify any areas where calibration/system tolerances could be tightened. AEA will conduct 5 sets of repeat measurements in-house and two at Ricardo. They will use the secondary PNC calibration method. JRC will also be conducting repeat measurements, these will include measurements on TSI's new 3790 PNC which is their production spec PMP PNC. Other members of the working group, in particular test labs and instrument manufacturers, are also requested to contribute sample calibration measurements.

17. The Chairman will circulate a proforma for the reporting of results from the calibration exercise. Grimm identified ambient conditions under which calibration was conducted as parameters of interest for inclusion on the proforma. The deadline for submission of results will be 28th September.

18. NPL gave a summary of the work they are doing on analysis of accumulation of errors. This analysis includes looking at PNC errors, VPR volatile penetration and solid particle losses. It identifies potential sources of error in each case and attempts to quantify their significance. A draft report has been prepared but will need to be updated in the light of agreed changes to the VPR calibration procedure and treatment of solid particle losses. The draft report will be circulated next week.

19. Under Any Other Business a 10am start time was agreed for the 2 day meeting in October. A reminder of the provisional dates (8th & 9th October) will be sent out along with a request for members to indicate their availability on these or alternative dates.

Chris Parkin
PMP Chairman