

# PMP – Particle Measurement Program Informal Working Group

## Task Force 2– Brake Dust Sampling and Measurement

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Meeting #22 – Thursday 18 JULY 14:00 – 15:00

### Minutes of Meeting – Final Version

**1. Tour de Table:** AUDI-(SG) Sebastian GRAMSTAT; AVL-(TM) Thanasis MAMAKOS; AVL-(MA) Michael ARNDT; BMW-(RL) Rasmus LEICHT; BMW-(KK) Katharina KOLBECK; BREMBO-(FR) Francesco RICCOBONO; BREMBO-(MA) Mattia ALEMANI; DEKATI-(MM) Mikko MOISIO; Ford-(JG) Jarek GROCHOWICZ; Ford-(MM) Marcel MATHISSEN; GM-(MR) Matt ROBERE; ITT-(SA) Simone ANSALONI; JARI-(HH) Hiro HAGINO; JRC-(TG) Theodoros GRIGORATOS; LINK-(CA) Carlos AGUDELO; LINK-(QO) Quinn O'HARA; TMD Friction-(AP) Andreas PAULUS; TSI-(RA) Bob ANDERSON; TSI-(SP) Stephan PERCOT; TU ILMENAU-(DH) David HESSE; TU ILMENAU-(TF) Toni FEISSEL.

**2. TF1 activities:** TG briefly described on-going TF1 activities. Important on-going topic is the preparation of a proposal for a methodology to regulate the incoming cooling air flowrate for each setup. The proposed method will be based on vehicle data collected from different projects. TG will share the proposed method with TF2 members when is finalized and not later than late August. Another on-going task is the preparation of the TF1 RR report. The report is being drafted and it is expected that it will be shared with TF2 before the next PMP Meeting. At the same time the provisions for the correct application of the WLTP-based cycle will be drafted. These will include the regulation of incoming cooling air flowrate, temperature measurement by means of embedded TC, application of soak time, considerations regarding the applied script, etc. These will become part of the final report to be released by TF2.

**3. AVL-TUI presentation:** TM described AVL activities regarding measurement of brake emissions. First set of results were related to solid PN emissions. Tests were performed with the TF1 brake pads over the WLTP-based cycle. ***PN concentrations were found to be 200 times lower than the current PN exhaust limit ( $6 \times 10^{11}$  #/km).*** PN concentration measurements were found to be very repeatable (coefficient of variation of about 10% over the 5 repetitions). Very few (or no) particles between 10 nm and 23 nm were measured.

A very important discussion related to the background concentrations followed. TM demonstrated that background concentrations can be as high as PN emissions over some of the trips of the cycle (i.e. trips 6 and 9). ***Overall, background can have a significant effect on PN emissions and therefore it should be kept as low as possible.*** It is expected that the background contribution on measured concentrations will increase as incoming cooling air flowrate increases. Finally, it was demonstrated that electrical size classification techniques (i.e. EEPS) come with much higher noise level and therefore higher measurement uncertainty.

TM presented total PM results over each one of the five repetitions of the cycle. Total PM refers to all particles that were captured to the filters (TSP) and not to PM<sub>10</sub> or PM<sub>2.5</sub> fractions. Total PM EFs of approximately 4.8 mg/km for each brake were reported. ***This corresponds to approximately 14.0 mg/km per vehicle with approximately 30% being PM<sub>2.5</sub>.*** PM measurements were found to be very repeatable (coefficient of variation of about 4% over the 5 repetitions). It was estimated that 45% of total PM emissions are lost somewhere in the system and never reach the filters. **Details regarding**

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the calculation of the losses and its attribution to different parts of the system are provided to the attached presentation of TU Ilmenau.

Data regarding brake particles size distribution were also presented. *This data is based on previous AVL-TUI experimental campaigns. All instruments measuring either total or solid PN gave practically identical results. This shows that no volatile particles are emitted under the testing conditions with the tested pads.* The noise levels of some instruments (i.e. EEPS and DMS) were above the background levels of the tunnel resulting in significant overestimation of PN emissions. It was concluded that great care needs to be taken when interpreting size distribution data. A discussion regarding the accuracy of various instruments and their capabilities followed.

### Questions (Black) and Answers (Green)

1. It is mentioned that *"For calculations cooling periods were discarded"*; however, this cannot be the case for PM concentrations. Do you have any rough estimation regarding the influence of soak times on PM concentrations (or it is absolutely negligible)?

That is true, although from our experience when we used the MSS, the background mass is insignificant. During the measurements the 10 nm PN background was higher from the 23 nm PN background which means that these are very small particles so mass-wise it should be insignificant.

2. Slide 3. Temperature profile – How was the temperature measured? Embedded or rubbing TCs?

Both embedded and sliding thermocouples were employed for temperature measurement. Results in slide 3 refer to embedded TCs.

3. Could you please define air velocity just to get comparison with results from other groups?

There is no clear definition of air velocity. It is easy to calculate an average speed in the tubing but not inside the enclosure.

4. Slide 3. Temperatures are quite higher and do not match very well Ford RDE temperatures. Low air velocity leads to increasing disk temperature. Why did you use this air velocity 170m<sup>3</sup>/h if it does not match well Ford profile?

There are several targets in the TF, and one of these was to allow for parallel PM<sub>2.5</sub> and PM<sub>10</sub> measurements. The tunnel was especially designed to increase penetration of particles larger than 2.5 µm. This was also the reason that the enclosure was made relatively large, as it was observed that by reducing its size the particle losses were increasing.

Furthermore, Horiba's data is somehow in agreement with what Ford presented in the last PMP meeting and what we have observed at TUI. The peak disc temperatures which are of more relevance (as these correspond to the actual braking/emission events) are little affected by the air velocity. It is also interesting that despite the higher flows Horiba employed and the lower disc temperatures compared to AVL tests, the number concentrations you measured are considerably higher (on average three times higher) than what we observed with the circulated disc/pads. This reinforces AVL position that we put too much emphasis on the disc temperature. Apparently, there are some other influences more critical in PN measurements.

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Flow could be increased but (in addition to having limited effect on peak temperature and increase PM<sub>10</sub> losses,) this would come at the expense of higher dilution and thus lower number concentrations. Therefore, this would further increase the contribution of background. AVL did not see much change in background concentrations when increasing the flow from 170 m<sup>3</sup>/h to 270 m<sup>3</sup>/h but concentrations of emitted particles would be decreased by ~60%.

**[TG: We need to wait Ford's feedback regarding the actual temperatures of the cycle measured by means of embedded TCs.]**

5. Regarding background correction did you subtract background from every measurement point or calculated mean particle concentration per/test(section)?

Since a constant background concentration was subtracted the two approaches are equivalent.

6. Slide 7 - How did you get data for the graph? Why you are assuming increasing background level with increasing air flow? How was the background level measured? Horiba never saw problems with increasing background level as a function of air flow. From Horiba's observation background level was always stable within 200 #/cm<sup>3</sup>. If it is higher following reasons can be taken to account (1. Leakage, 2. Dirty H13 Filter, 3. Short time between cycle repetition, 4. Changing of air velocity for new cycle)

These were just calculations, based on the assumption that background concentration is constant. If you increase the tunnel flow, you dilute the true concentrations and therefore the relative contribution of background increases.

The main point is that PN emissions are extremely low (as confirmed by all available data). We are talking about more than 2 orders of magnitude below the exhaust PN limit. That is in striking difference from PM mass emissions being more than one order of magnitude higher than the exhaust PM of late technology vehicles. Having a significant fraction above than 2.5 μm, we find it crucial that we focus our work on means to increase penetration of super-micron particles. That is also why we stated that with the existing evidence, it would be an overkill to start discussing about a new PN procedure. If future investigations (i.e. HD applications) reveal the formation of large number of nano-sized particles, then we can perform dedicated measurements to gain better insights and define a more appropriate PN measurement methodology.

7. It is mentioned that "**Electrical size classification techniques have inherently higher noise level (i.e. 1680 #/cm<sup>3</sup> for EEPS/DMS)**". Does this mean that practically only CPC can be applied for measuring PN concentrations?

Definitely high noise is critical. Most critical though is the accuracy of inversion. Even with the noise subtracted, the results are not matching the CPCs and can be off by more than 200%. I showed e.g. that by changing the inversion matrix in the EEPS we get completely different number concentrations and size distributions. These techniques are only suitable for qualitative information so I do not see them as of relevance for this work group. This should become evident once their calibration topic is raised.

8. It is mentioned that "**Noise levels of EEPS (~2000 #/cm<sup>3</sup>) well above the background levels of the tunnel (100-200 #/cm<sup>3</sup>) and concentration spikes during braking events overestimated substantially true particle number emissions**". Does this practically mean that if someone performs total particle measurements with the EEPS and applies to the same sampling line a

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thermal treated CPC will measure a highly overestimated total PN concentration due to background? Does this mean that attributing the whole difference to volatiles will be wrong?

Generally, I would not consider EEPs PN comparable to CPC PN even at the same conditions (solid or total) even at levels well above the instruments. And of course the very low emission levels make comparisons even more troublesome. Also please note that in general volatile background may be more variable and higher (see also slide 8).

9. It is mentioned that **"CPCs of different cut-off sizes agreed within  $\pm 5\%$ , with no evidence of sub-23 nm present"**. Is there a possibility of losing small particles? Do you observe the lack of sub-23 nm for both total and solid only measurements?

Differences of 10% between 10 and 23 nm CPC in an APC suggest that particles are very large even in automotive exhaust. For the limited number of tests we had an optical counter it was measuring the same as the 10 nm CPC, over its concentration measurement range, so these seem to be extremely large particles.

10. Slides 10-12 - As presented during the 50<sup>th</sup> PMP Meeting, Horiba is measuring 9.5 mg/km for total PM that corresponds around 80-85% of total mass losses. Total mass losses disc + pads was 2.28 gr. What was total mass losses disc + pads in AVL case?

It was  $\sim 1.95$  gr.

11. Slide 13 - Did you use same pads and applied a different air flow? If yes, what was the reason?

No, these were results from a previous campaign with different types of ECE pads. At that time, the TF2 pads were not available. We decided to decrease the flow in the final campaign (first slides) as it would improve  $PM_{10}$  penetration. We just wanted to share with the group our experience on total vs solid particles and the performance of sizing instrumentation based on our available data.

12. Slide 14 - Do you have data from CPC measured using VPR as you show on the picture? Could you put to separate graph data for CPC from cold injector (DR25) and hot injector? OR you are comparing data from CPC to APS?

We did not perform many tests with the novel cycle in this specific campaign. The results presented here were from CPCs sampling downstream of the ejector at ambient temperature (i.e. total particles). Tests at different ejector temperatures were performed on different driving conditions, and since this is of no relevance to this work we refrain to presenting this in a dedicated paper.

13. It is mentioned that **"No indication of volatile particles over the novel cycle with pads tested"**. At a next step I think it would make sense to also test NAO pads. They are expected to have different behaviour (lower PM emissions but higher PN concentrations).

We did some tests with NAO over different segments of AK Master. Even there though, I hardly saw volatile besides the fade and generally PN were also lower than ECE. Still I know that there are many different types of pads so I cannot generalize.

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14. It is mentioned that **"Some type of dilution will be required for precise concentration measurements"**. Is that because the background effect will be minimized? What other reason for including further dilution exists?

This is just because individual concentration spikes can be rather high. We measured ~40,000 #/cm<sup>3</sup> in the latest tests and >50,000 #/cm<sup>3</sup> in previous tests. Most full-flow CPCs start having linearity issues above 10,000 #/cm<sup>3</sup>. Coincidence corrections are typically applied, but accuracy of measurements can deteriorate at such high concentrations. The other topic is of course the practical aspects. The end user would want to have a system that can measure also over AK master, and we as a supplier would like to have a system that can cover all conditions. If we only supply the CPC, then the instrument will get saturated (measure flat at the maximum level) as the actual concentrations can be orders of magnitude above the measurement concentration range of the CPC over such conditions.

15. Slide 20 - For which type of measurements did you apply corrections? Was it total, solid or both? It is strange to see that solid PN are higher in comparison to total PN. In this case we cannot reach a safe conclusion about volatile content?

As stated in the slide, it is not entirely surprising, because the particles seem to be very large. Therefore, the default correction for losses (average losses at 30, 50 and 100 nm) overestimates the losses leading in ~10% higher counts. The results though suggest that at least with the specific brake there was not significant volatile content over this cycle (at least not in the form of separate particles or causing a size change that could lead to different particle losses / detection efficiencies by a 23 nm CPC).

16. Slide 22. It is recommended that **"some type of dilution will be required for precise concentration measurements"**. Why would dilution be needed? Is it because CPCs are saturated at certain braking events? Wouldn't that lead to very low overall PN emission levels taking under consideration the already low PN levels? Wouldn't that compromise the credibility of the measurement?

Dilution is needed for two reasons. The most straightforward is that the emissions can temporarily exceed the linearity range of some CPCs. I know that the original TSI full-flow CPCs (some of which we used in our campaigns) have a maximum certified (for linearity) concentration of 10000 #/cm<sup>3</sup>. There is an internal correction in all CPCs (coincidence correction) that increases with measured concentration and at 10000 #/cm<sup>3</sup> reaches 10% for the TSI CPCs (in AVL CPC the 10% correction occurs at 30000 #/cm<sup>3</sup>). Since brake wear measurements in a tunnel are an integrated type of measurement (we accumulate real-time signal) the highest spikes will contribute the most to the final result and are those that need to be precisely measured. We measured up to 40000 #/cm<sup>3</sup>, but of course this may change for different pads and different tunnel flows. Also calibration of CPCs with respect to their linearity is somehow limited due to challenges with the calibration procedures. The second and more practical is that you want to make sure that the technical solution will be working at all conditions. If our concern for PN is that some nucleation particles may be forming, these will lead to orders of magnitude higher concentrations. At such high concentrations, a CPC will just get saturated and will not be able to measure.

Now, the dilution should not compromise the credibility of the measurements, because these are true particles. The CPCs are selected in PN measurements because they have a very low zero

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level. They can detect individual particles, and this typically corresponds to a concentration of  $0.01\#/cm^3$ . So with a dilution of 100 (the one we used), the zero level would be  $1\#/cm^3$ , well below the background levels. Stated differently, CPCs are designed to cover a rather low concentration range ( $0.01$  to  $\sim 10000\#/cm^3$ ). Higher concentrations can be achieved but with compromises in accuracy (either by shifting to non-full flow designs or by moving to photometric modes (i.e. not detection of individual particles)).

17. Slide 23. It is mentioned that **“how reliable is background subtraction and up to what level?”**. Wouldn't be better not to apply subtraction at all but instead report PN emissions and background concentrations? Do you know if this is common in PN measurements at other categories? Wouldn't it make sense to set a limit for acceptable background level in order to consider a measurement valid?

I would say, let's first see what can be achieved at different labs. As I mentioned, we anticipate that there is room for improvement in the TUI tunnel, but still the  $200\#/cm^3$  is not really that high. I remember seeing much higher levels in some presentations in the PMP. It was not my intention to suggest a correction but rather to indicate the issue, and quantify its implications. The main point is that PN emissions are at very low levels. If labs have very different backgrounds, we will end up comparing the backgrounds. There are also other subtle issues, like the apparent indications that we have small particles just because of small background particles.

The R83 methodology describes a correction for PM background but not for PN. The problem with PN is that it is not a conserved property (there is no such thing as conservation of mass). Irrespectively, this is hardly an issue in exhaust measurements because PN emissions are 2 orders of magnitude higher. Cold start emissions even of late technology vehicles can be as high as  $>1e7\#/cm^3$  at tailpipe. However, the situation is quite opposite in exhaust where PM is at noise levels and background can have a strong effect.

18. Slide 24 – Shouldn't we consider coagulation effects by low air velocities as was presented before?

Coagulation at such low concentrations should be insignificant (not sure what concentrations you had but based on your statement “No coagulation problem is expected from theory” it should be too low – we measured max concentration of  $40,000\#/cm^3$ ). I am pretty sure that something else is causing this difference. Background may be a plausible reason. The concentrations of emitted particles will go down due to higher dilution but background level will remain relatively the same. Thus the concentrations will not go down proportionally to the increased dilution. It would be interesting to see how the results would look after subtracting the background.

19. Slide 24 – How sub 23 nm could be introduced to the background if H13 filters are applied? They could come to background just due to previous brake events.

The H13 filter has a finite filtration efficiency and the actual background concentration will also depend on the distribution of background air entering the filter. To my experience the background air can have significant amount of  $<23$  nm particles depending on the measurement location, so a lot of them can exist in the tunnel. We see this when measuring with 10 and 23 nm APC or even the CPCs sampling directly background from the tunnel. It is also possible that some

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background may be originating from the shaft and the bearings. At the end this is something that can be easily checked if you have two CPCs with different cut-off size.

20. Slide 25. It is mentioned that **“size information can only be treated as qualitative”**. Does this mean that it can be used for losses calculation?

Absolutely not. Depending on the assumptions you make in the inversion you can get substantially different size distributions (notice that the axis is in logarithmic scale). Furthermore, a lot of these instruments show artificial modes at small sizes due to the noise in the electrometer (the signal produced by a given number of particles decreases considerably with decreasing particle size thus electrometer noise can give the impression of nanosized particles). I really feel that this is an overkill.

Now, I am not entirely sure I understand which type of losses you refer to. If you refer to PN, then we are mainly talking about losses of small particles. Then, the only reasonable approach I would recommend is the one suggested by Pr. Kittelson in which one uses two CPCs with different cut-off sizes (i.e. 10 nm and 23 nm) and apply a correction that would depend on the relative difference (i.e. when 10 nm CPC measures more than a 23 nm CPC, the emitted particles are smaller so higher losses are expected). This would serve as a very crude size information with the advantage of utilizing two very sensitive detectors with precisely defined sizes (mobility sizes that are of relevance for diffusion losses).

If you refer to PM, then this implies that focus is on larger sizes and aerodynamic diameters. But then, I cannot really recommend something at this stage as we have not even decided on the very basics, like how to define 2.5  $\mu\text{m}$  and 10  $\mu\text{m}$  (i.e. cyclone specifications). Definitely though, if it is to consider something then as in the case on PN (i.e. two CPCs) I would only recommend first principle methods like a cascade impactor, but then again as an option (with some needed work on effect of particle bounce on brake-wear particles). Clear specifications for 2.5  $\mu\text{m}$  and 10  $\mu\text{m}$  cyclones that would allow for a parallel measurement of  $\text{PM}_{10}$  and  $\text{PM}_{2.5}$  would probably suit better. Still correction at large sizes would require a detailed experimental characterization of particle penetration in the tunnel for super-micron particles which is demanding. Our attempts thus far in this area were unsuccessful.

21. Slide 28 – Weighing of components like disk with mass around 6 kg could introduce uncertainty due to measurement limit of balance used for heavy parts. We should interpret such data very carefully. We presented already at the 50<sup>th</sup> PMP Meeting the influence of air velocity on PN and PM for novel cycle as well as for exhaust WLTP.

We certainly realize that this is demanding, although not necessarily impossible. Filter weighing in exhaust measurements is probably more demanding. Nevertheless, we found this approach useful in our research work and wanted to share this with the group.

22. Slide 29. It is mentioned that **“Use low sample flowrates (less than 10 Lpm), and minimize bends in connecting tubes”**. Isn't this quite close to JARI's recommendation? Do you see big differences from what you propose?

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Please note that we refer to the extracted flow from the tunnel for PM filter measurements. Not on the tunnel flow.

### Important conclusions

- 1. PN emissions over the novel cycle were found to be more than 2 orders of magnitude below the exhaust PN limit for the tested pads;*
- 2. Non-volatile PN measurements exhibited a repeatability of better than 13% which is very good taking into account the complexity of the measurement;*
- 3. Background tunnel concentrations can have a significant effect on PN results at low emission levels. Therefore, maintaining background as low as possible is crucial for precise PN measurements;*
- 4. Emitted particles over the novel cycle are much larger than 23 nm. This is also confirmed by previous tests with different type of ECE pads;*
- 5. There was no indication of volatile particle emissions despite the relatively high disc temperatures. Some volatiles are present during sparse events resembling tunnel artifacts;*
- 6. Size distribution information can only be treated as qualitative due to the high level of noise;*
- 7. Total PM emissions with the supplied pad over the novel cycle were at  $4.8 \pm 0.2$  mg/km. 25% of the measured total PM belong to the  $PM_{2.5}$ .*

**4. Next meeting:** The next TF1 meeting will take place beginning of September.