Understanding, Measuring and Regulating Sub-23 nm Particle Emissions from Direct Injection Engines Including Real Driving Conditions

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Introduction

This report gives an overview of the development of the heated automotive partector (HAP). This device is based on an existing commercial device (the automotive partector (AP) by naneos particle solutions llc.) that is already successfully deployed in one of the leading commercial PN-PEMS systems. The standard AP complies with current regulations (23nm d_{50} cutoff). In this task, the AP was modified so it could be heated and operated at 150°C to reduce requirements for dilution of the exhaust, and optimal settings were found so that the d_{50} cutoff could be shifted to clearly lower diameters (10-15nm). 3 Prototype instruments were built, calibrated and their performance with polydisperse aerosol was verified. These prototypes are now available to be used in further parts of the SUREAL project by other partners.

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Abbreviations list

AP	Automotive Partector
CAST	Combustion Aerosol Standard (soot generator)
CPC	Condensation Particle Counter
DMA	Differential Mobility Analyzer
EU	European Union
HAP	Heated Automotive Partector
PMP	Particulate Measurement Programme
PN	Particle Number
PN-PEMS	Particle Number Portable Emissions Measurement System
RDE	Real Driving Emissions
SMPS	Scanning Mobility Particle Sizer
VPR	Volatile Particle Remover

Short Project Overview

A large proportion of the total number of particles emitted from direct injection engines are below 23 nm and although the EU aims to regulate those emissions and impose limits for new light duty vehicles, this is not yet possible due to the absence of accurate quantification methods, especially under real driving conditions. The main reason for this is the absence of adequate knowledge regarding the nature of sub-23 nm particles from different engine/fuel combinations under different operating conditions. SUREAL-23 aims to overcome such barriers by introducing novel measurement technology for concentration/size/composition measurements. The recently established supercontinuum laser technology will be coupled to photoacoustic analysis and will also be employed for photoelectric ionization aerosol charging to achieve real-time, composition size-specific analysis of the particles. In parallel, state of the art aerosol measurement techniques will be advanced for better compatibility with sub-23 nm exhaust particles as well as on-board use. The developed instrumentation will assess sub-23 nm particle emissions from both Diesel and GDI vehicles accounting for effects of the fuel, lubricants, aftertreatment and driving conditions for existing and near-future vehicle configurations. The most suitable concepts will be developed for PN-PEMS applications and evaluated accordingly. The project will provide measurement technologies that will complement and extend established particle measurement protocols, sustaining the extensive investments that have already been made by industry and regulation authorities. The project will deliver systematic characterization of sub 23-nm particles to facilitate future particle emission regulations as well as to assess any potential trade-off between advances in ICE technology towards increased efficiency and emissions. The consortium consists of European and US organisations, which are leaders in the field of aerosol and particle technology.

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1 State of the Art

The heated automotive partector (HAP) is based on the standard automotive partector (AP) of the Swiss company naneos. We give a short description of the standard AP here and its current application in a PN-PEMS system to present the current state of the art. We also give a very brief overview over other technologies used for particle number counting in automotive exhaust.

1.1 Current emission monitoring regulations and devices

With the emission standard Euro 5b, a particle number (PN) emission limit was introduced, and subsequently several commercial devices became available for measuring PN in automotive exhaust. These devices were first built to be operated on chassis dynamometers for type approval measurements, and consequently they are generally immobile. These devices generally include a volatile particle remover (VPR), a dilution unit and a PN detector, which is usually a condensation particle counter (CPC). The stationary devices are usually called “PMP-compliant PN counters”, named after the working group that established the measurement protocol.

Since September 2017, real driving emissions (RDE) are measured during type approval. On-road testing clearly needs mobile devices, so a new generation of devices has been developed over the last approximately 2-3 years. These devices still measure PN in automotive exhaust, but are generally smaller, lighter and use less power than the stationary PMP devices. This new generation of devices is known as “PN-PEMS” (Particle Number Portable Emission Monitoring System). The PN-PEMS devices must adhere to a specification worked out by the joint research center of the EU. The requirements for the PN-PEMS devices are a little bit less stringent than for the stationary PMP systems.

1.2 Current PN counters in PMP and PN-PEMS systems

The requirements for particle counting in PMP and PN-PEMS systems are specified by setting limits for the particle counting efficiency for some given particle diameters. The requirements do not specify that a certain technology must be used for particle counting, but by far the most common type of detector used is the condensation particle counter (CPC).

1.2.1 Condensation particle counters

The CPC counts particles by growing them in a supersaturated vapor of a working fluid (e.g. Butanol or Isopropanol), and then detecting a pulse of scattered light for each particle. CPCs are well-established workhorses of aerosol science, and generally reliable and accurate devices. They do have some drawbacks though, especially for mobile applications; the drawbacks are mainly related to the necessary working fluid. The most common working fluids are flammable which presents a (controllable) hazard, the working fluid has to be replenished regularly by the user, and the working fluid may spill in the device if the device is tipped, or if large accelerations occur. One further issue with the working fluid is that it has different wetting properties for different materials, so that CPCs usually exhibit a slight material dependence of their counting efficiency. Furthermore, the temperatures in the saturator and condenser of the CPC must be accurately controlled to create repeatable conditions, which may present a challenge for

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an on-road measurement where ambient temperatures may change rapidly. Finally, the common working fluids used in CPCs condense at temperatures lower than typical automotive exhaust temperatures, so that the exhaust has to be cooled which also necessitates a dilution to prevent condensation of the water vapor in the exhaust gas. The dilution is also necessary because the CPCs usually operate in concentration ranges of about 0-5·10^4 pt/cm^3. Consequently, a rather high dilution factor (100x – 1000x) has to be applied, which needs accurate dilution control.
Overall, devices based on CPCs are accurate and use a well-known and proven technology, but have some associated complexities (Temperature control, working fluid, dilution) that present challenges in the RDE application.

1.2.2 Automotive partector

Because of the drawbacks of the CPCs outlined above, one of the leading automotive instrumentation manufacturers (AVL List GmbH) chose to use an electrical aerosol detector, the automotive partector, in its PN-PEMS. A scheme of the AP is shown in Figure 1.

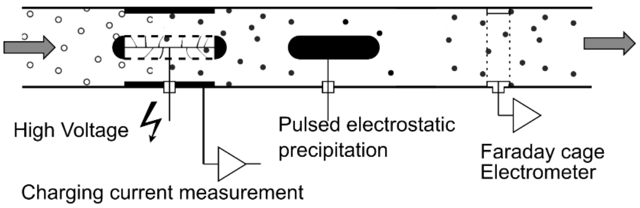

Figure 1: scheme of the automotive partector

The AP is based on the general observation by Burtscher and Schmidt-Ott that the conductivity of an aerosol after unipolar charging is approximately proportional to particle number concentration (Burtscher & Schmidt-Ott, 2004). The device consists of a unipolar charger that is followed by a pulsed electrostatic precipitator to remove a fraction of the particles (to detect the conductivity) and finally an induction stage to measure the removal of the particles in the precipitator. The induction cage produces very small signals, on the order of fA – pA, which must be measured by a very sensitive electrometer. Using an induction stage rather than measuring the charge captured in a filter avoids issues with electrometer zero drift and filter clogging (Fierz et al., 2014).

One of the main advantages of the AP is that it is very small and simple compared to a CPC. An image of the device is shown in figure 2.

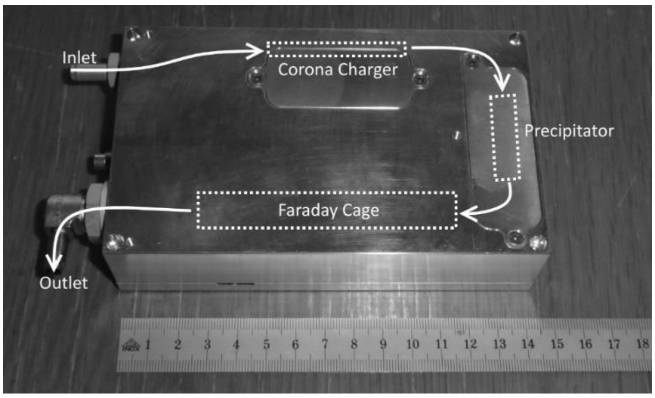

Figure 2: an image of the AP showing the scale, the main components and the flow path

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The counting efficiency of the automotive partector can be adjusted by adjusting several parameters, namely the charging current, the deposition voltage and the flow rate in the device. Figure 3 shows one such example where the deposition voltage in the precipitator is varied, leading to different instrument responses.

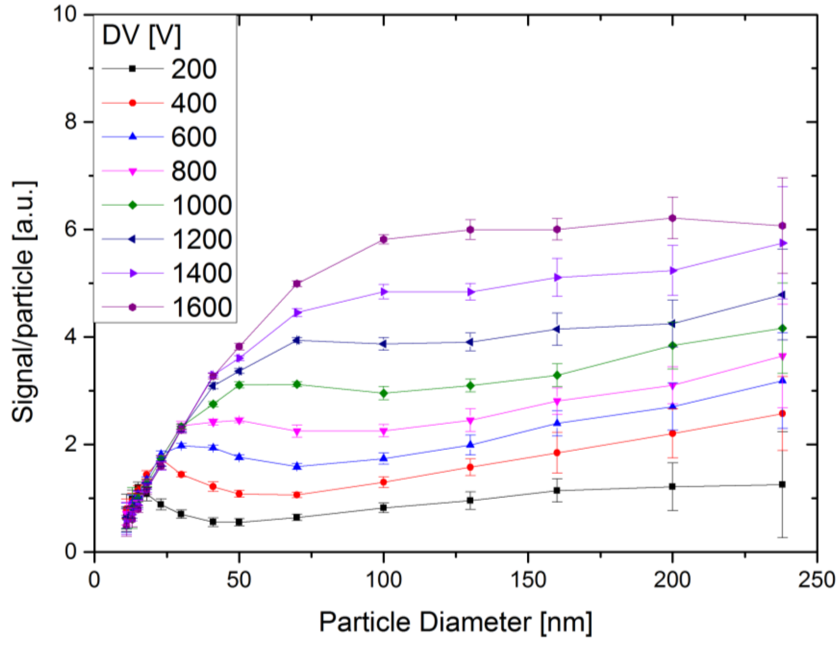

Figure 3: Variation of the deposition voltage DV in the standard AP

Figure 3 shows that the counting efficiency curves that can be obtained with the AP resemble those of a CPC – an initial increase of the counting efficiency is followed by a plateau region. However, the plateau is not perfectly flat as for the CPC, but usually slopes slightly upwards for larger particle diameters. From figure 3 we can also see that the d_{50} of the counting efficiency can be decreased by reducing the precipitator voltage, at the price of a lower signal overall.

In conclusion, the AP has some advantages but also some drawbacks compared to the CPC:

Advantages compared to a CPC

- Smaller
- Lower power consumption
- No working fluids
- Insensitive to orientation
- Low sensitivity to temperatures
- Larger possibilities of adjusting settings

Drawbacks compared to a CPC

- Less accurate
- Baseline noise of ~500 pt/cm^3
- Larger morphology dependence
- Charging may depend on carrier gas composition
- Dependent on initial charge state of the aerosol

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- Less nice counting efficiency curve
- Higher sensitivity to flow rate

Perhaps the most important point in the list above for our project is that particle morphology is relevant in the AP. The charge acquired by the aerosols is higher for fractal particles than for compact particles of the same mobility diameter. Therefore, these particles have a higher drift velocity in the precipitator (proportional to charge q). The total charge removed in the precipitator is proportional to q^2 (one q for the drift velocity, the other for the removed charge), and thus the signal in the induction stage is proportional to q^2. The more fractal a particle is, the higher the instrument response for the particle. For automotive exhaust this is relevant since the soot particles are fractal, and therefore, the AP has to be calibrated with soot particles.
Another important point is the dependence of the corona charging on the carrier gas composition – normally, the AP is operated in air. In automotive exhaust, the Oxygen is (partially) replaced by Carbon Dioxide, which may influence the corona charger. Any effect of this can be reduced by dilution with air.

The particle number range that can be measured with the standard AP is approximately 500 – 500’000 pt/cm^3. It is therefore closer to the measurement range necessary for automotive exhaust and only needs a low dilution (10:1 or similar), which is easier to implement than the high dilutions necessary for the CPCs. The standard AP is operated at a temperature of about 50°C to avoid issues with condensation.

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2 Heated Automotive Partector Description

2.1 Innovation

The AP was designed and optimized to be used as a PMP-compliant particle number counter, i.e. the d_{50} of the device is around 23nm. In its current application in the PN-PEMS, it is used with a dilution of about 10:1.

For SUREAL-23, the first goal was to move the d_{50} to a smaller particle diameter, ideally to around 10nm. As explained in the section above, the AP has many settings that can be changed and we tested a lot of different settings to find those that fit the new application best. From our previous experience with the AP it was already clear that we would have to use a lower deposition voltage (compare figure 3) which would lead to a lower instrument response; therefore, we increased the charging current to increase the absolute signal level by about a factor 2. This “parameter optimization” was an important and labor-intensive step, but not really innovative.

The second and larger change was to enable the operation of the AP at much higher temperatures, up to 150°C. The entire electronics PCB of the AP had to be removed from the housing and replaced with a new PCB with better insulation properties. Also, all temperature-critical components had to be removed from the new PCB, i.e. most electronic components. The separation of the electronics from the mechanical part allowed us to heat the mechanical part of the device to 150 °C. The only part that could not be removed from the new PCB was the electrometer, which needs to be close to the Faraday cage detection tube. The electrometer is cooled by a blower so that it remains at tolerable temperatures ($T \approx 60°C$).

While the instrument response is about 2-3x lower than in the standard AP due to the lower deposition voltage, the operation at the high temperature probably means that the device could be used with raw exhaust without dilution, thus making it more sensitive than a 10:1 diluted standard AP.

2.2 Principles of Operation

The principle operation of the AP has already been explained earlier, and the heated automotive partector (HAP) is essentially identical to the original AP. The big difference compared to the standard AP is that we have built an entire instrument around the AP, with the necessary power supplies, pumps, flow controls and heating controls. Figure 4 shows a picture of the device:

In figure 4, we can see the AP in the right-hand compartment; it is heated to a selectable temperature up to 150°C by the large heating resistor that is visible. Also visible are the preheater on the inlet (connection at top right), and to the left of it, the heated critical orifice used for the flow control. The left-hand compartment contains all the electronics that is necessary for the devices, such as power supply, pump, blower for electrometer cooling, electronics for the AP, and a temperature controller.

A functional scheme of the device is shown in figure 5. An aerosol preheater is used to make sure that low-temperature gas at the inlet can be brought up to the temperature of the AP at its inlet. A critical orifice is used to create a stable volumetric flow in the instrument; the orifice is temperature-controlled, also at 150°C, because the flow in the critical orifice depends on temperature. Using a constant volumetric flow has one

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drawback: if the inlet pressure is different than ambient (over- or underpressure) the device will see higher signals for overpressure and lower signals for underpressure, because there will be more or less particles per cm^3 in the compressed or expanded gas. Therefore, the HAP should not be operated at high or low underpressures; it will still work, but the results would have to be corrected for the pressure difference, which would also make an external pressure measurement necessary.

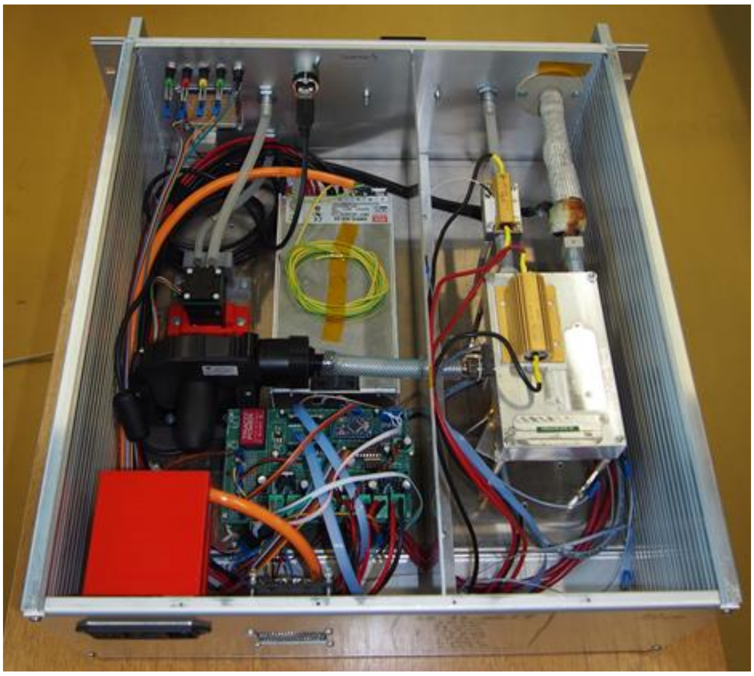

Figure 4: an image of the open SUREAL prototype.

An optional water trap could be used before the pump, which provides the necessary under pressure for the critical orifice.
The AP is controlled by the normal AP electronics PCB, which is placed in the low-temperature zone of the HAP prototype. All temperatures (of the preheater, the AP, and the critical orifice) are regulated by a dedicated temperature controller.

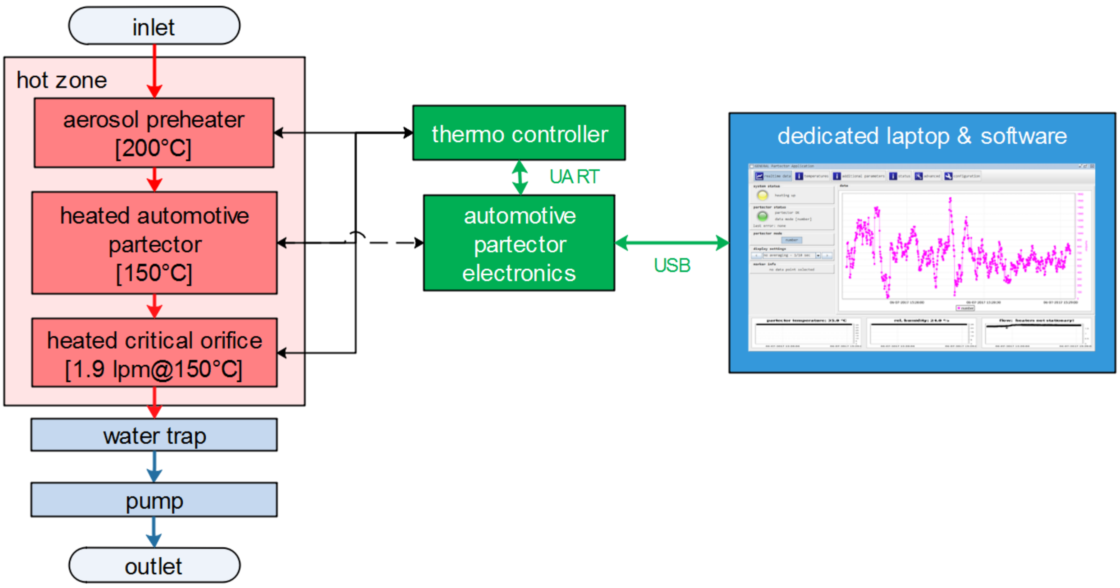

Figure 5: scheme of the SUREAL prototype device

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The SUREAL prototype is controlled by a Java program developed within this project. The program can be used to configure settings of the device, to view all parameters measured by the device online, and to store data. Furthermore, the program also can communicate the data acquired further via Ethernet TCP/IP protocol for integration into a test stand, so that it can easily be used by our project partners.

3 EVALUATION

The SUREAL prototype was evaluated in multiple ways. First, we performed a large number of experiments with monodisperse aerosol, where the counting efficiency as function of particle size was determined for many different settings by comparing the sensor signal with a CPC. This step yielded the optimal settings for the sensor to achieve a low d_{50} cutoff. After this, we next performed a calibration with polydisperse soot particles, since the sensor is supposed to measure polydisperse aerosol rather than monodisperse particles. The reference instrument in this case was an SMPS. A calibration factor was determined from these experiments. In a final step, we verified the instrument performance against a CPC, and corrected the calibration factor obtained in the second step. All experiments are discussed in detail in the next section.

3.1 Counting efficiency for monodisperse particles

A large set of calibration experiments was performed to find the optimal settings for the HAP. In these, NaCl or soot particles were size-selected in a DMA, neutralized, and then sampled in parallel by a CPC (TSI 3775) and the HAP prototypes. Care was taken to keep particle concentrations below about 25'000 pt/cm^3 to remain in the single counting mode of the CPC. Doubly charged fractions were estimated, but we made no attempt to correct for them, since the influence of the doubly charged particles is nearly negligible due to the rather flat instrument response for particle diameters >~30nm.

Initially, exploratory experiments were performed with NaCl particles, because the NaCl particle generator produces a wide spectrum of particle sizes, so that it is possible to measure the counting efficiency as function of particle diameter without changing the particle source. These initial experiments provided a basis for selecting a promising range of parameters to be used for the HAP. Further tests were then performed with soot produced by a combustion aerosol standard generator (CAST), where only the promising parameter range found with the NaCl experiments was explored further. This was necessary, since the fractal particles acquire more charge as discussed earlier, and therefore the HAP must be calibrated with soot. Unfortunately, the CAST generates a relatively narrow particle size distribution, so that the counting efficiency has to be determined with multiple different settings of the CAST, making the measurement effort with the CAST much larger than with NaCl. All soot experiments were performed with 4 different settings of the CAST, corresponding to nominal geometric mean diameters of 27, 48, 88 and 105 nm. For each of these settings, multiple measurements were performed (with different deposition voltages, different charging currents, different flows). The size range that could be covered with the CAST is different for each of the 4 different set points. From previous experience we knew that for particle diameters smaller than about 25nm, NaCl particles and soot particles behave identically in the AP. Because it is hard to generate particles smaller than about 20 nm with the CAST, we simply used the NaCl results for diameters below 25 nm.

Figure 6 shows the absolute counting efficiency of SUREAL prototypes 2 and 3 for the different types of calibration aerosol. For the soot particles, the calibration curve for each CAST diameter only covers a part of the size range investigated, as the CAST size

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distributions are rather narrow.

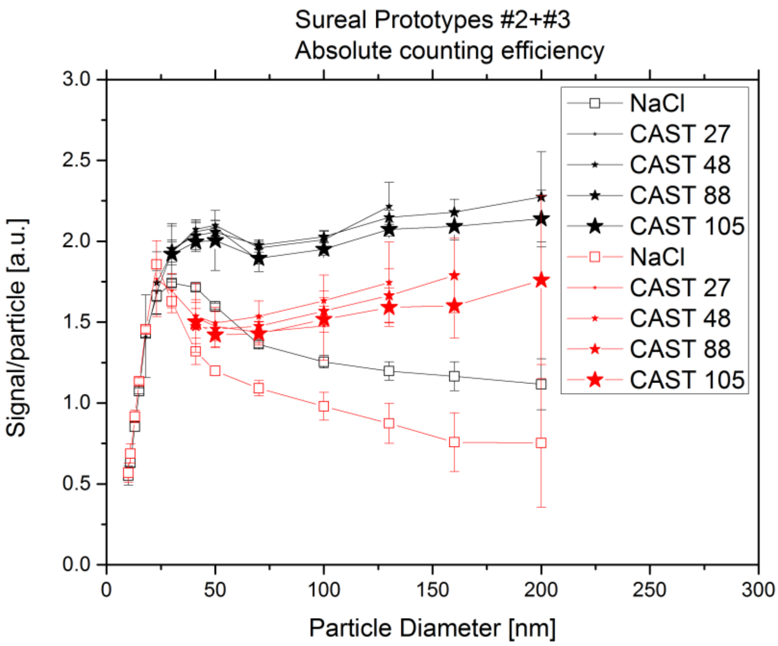

Figure 6: absolute signal levels in SUREAL prototypes 2 and 3 for different particle types and size. The black symbols belong to prototype 2, the red ones to prototype 3. The numbers in the legend (CAST 28,48,88,105) correspond to the nominal diameter of the CAST aerosol.

Figure 6 clearly shows the morphology dependence of the signal, with an increasing difference in signal for compact and fractal particles with increasing particle diameter. This also shows the necessity of calibrating the HAP with soot particles rather than with NaCl particles. We can also observe a generally higher signal level in prototype 2 relative to prototype 3. The origin of this difference is presently still unclear.

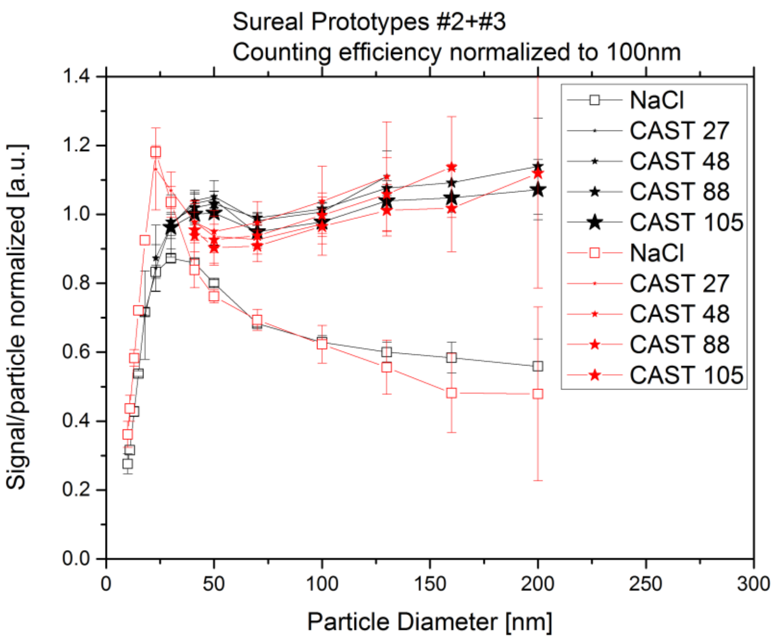

Figure 7: counting efficiencies of SUREAL prototypes 2 and 3, normalized to 100nm. The black symbols belong to prototype 2, the red ones to prototype 3.

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The counting efficiency for monodisperse particles can be used to calculate a particle number from the signal, and for this, we normalized the signal to particle number at the 100 nm data point. The choice to normalize at 100 nm is obviously arbitrary, any point between about 50 and 150 nm would serve just as well, as the counting efficiency is rather flat in this region. Figure 7 shows the normalized counting efficiency for the two devices.

In figure 7, we can see the CPC-like counting efficiency for the HAP prototypes with CAST soot. We can also see a slight difference between the two prototypes around 20-30nm, where prototype 3 exhibits a slight overshoot in its counting efficiency.

Based on these efficiency curves, we can calculate the d_{50} cutoff diameters for the two prototypes for monodisperse aerosols. They are 14.4 and 11.9 nm, respectively, for the prototypes 2 and 3, well within the size range that we hoped to achieve within this project (10-15 nm).

3.2 Theoretical instrument response for polydisperse aerosol

The counting efficiency curves shown in figure 7 are not as nice as typical CPC counting efficiency curves. The plateau region for d >> d_{50} is not perfectly flat but slopes upwards slightly, and there is a little amount of overshoot (counting efficiency > 1) for diameters around 25nm (prototype 3) and 50nm (prototype 2). The HAP is unable to completely mimic the CPC efficiency curve due to its very different principle of operation. To show this more clearly, we have plotted the average normalized counting efficiency for the 2 prototypes against the counting efficiency of a hypothetical 10 nm CPC. For the 10 nm CPC curve, we used a fit for the 3790 CPC with its 23 nm d_{50} cutoff, and rescaled the x-axis of the data so that its d_{50} cutoff lies at 10 nm. The comparison of the SUREAL prototypes with the 10 nm CPC is shown in figure 8.

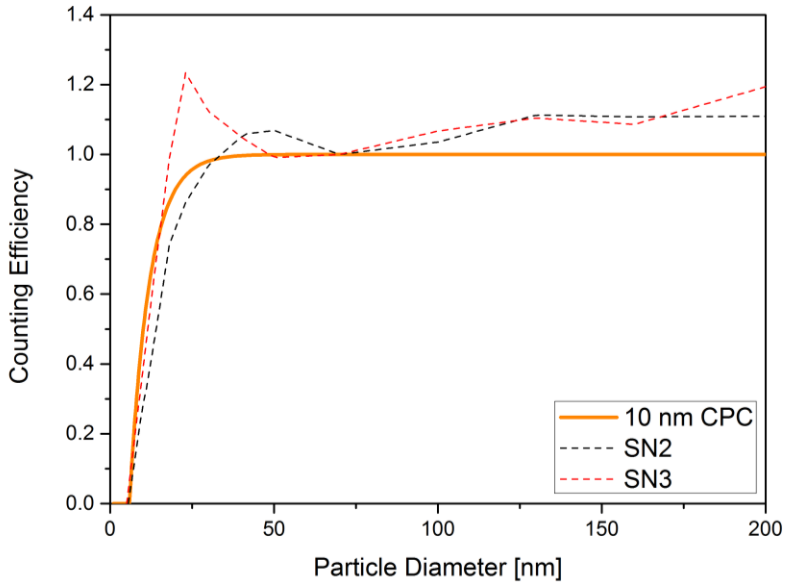

Figure 8: normalized counting efficiency of the 2 SUREAL prototypes compared to a hypothetical 10 nm CPC.

The agreement between the SUREAL prototypes and the hypothetical CPC curve is rather good, although some deviations occur and especially some overestimation in the lower diameter range occurs. However, we should not forget that all these devices are meant to measure automotive exhaust and not monodisperse particles, i.e. they are

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meant to measure particles which usually have a lognormal particle size distribution with a geometric standard deviation of roughly 1.7. Using the experimentally determined counting efficiencies of prototypes 2 and 3 for monodisperse particles, we calculated what the expected instrument response would look like for a polydisperse aerosol, by integrating the counting efficiency over the size distribution, while weighting the result with the particle concentration as function of the particle diameter. Such an averaging over the particle size distribution will average out the overshoot seen in the counting efficiency of prototype SN3. Figure 9 shows the calculated polydisperse counting efficiency for the same 3 devices as in figure 8, calculated for lognormal aerosols with $\sigma = 1.7$.

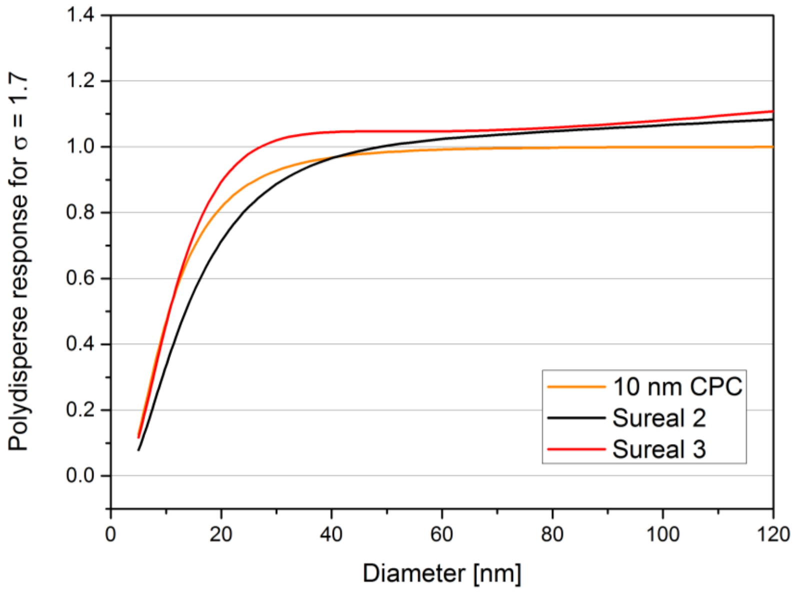

Figure 9: calculated counting efficiency for a polydisperse aerosol with lognormal size distribution with $\sigma = 1.7$

Comparing figures 8 and 9, we can see that the HAP prototypes are expected to perform very similarly to a CPC for a lognormal aerosol; the wiggles in the size distribution are averaged out. We can also re-calculate the d_{50} diameter from the data shown in figure 9 for the 2 devices, and we find 14.1 and 11.0 nm for prototypes 2 and 3 respectively, compared to 14.4 and 11.9 nm for the monodisperse calibration. The slight overshoot of SUREAL prototype number 3 in the monodisperse counting efficiency actually helps to reduce the d_{50} for the polydisperse aerosol a bit.

Figure 9 also shows that when the counting efficiency of the device is measured with monodisperse aerosol and a calibration constant is derived from the 100 nm data point, the device will slightly overcount in the plateau region; for prototype #2, we expect it to overcount by 5% if we set the calibration constant according to the value measured at 100 nm. Therefore, we decided to use polydisperse aerosol to set the actual calibration constant. This is described in the next section.

3.3 Calibration with polydisperse soot aerosol

After measuring the counting efficiency for monodisperse aerosol as described in section 3.1, we initially set the calibration constant of the device to the value derived from the 100 nm data point. The calculations in section 3.2 suggested that the devices would then probably overcount polydisperse aerosol, and therefore we performed further

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comparison measurements. The CAST was used to generate polydisperse soot particles with geometric mean diameters in the range of about 10 to 75 nm. Unfortunately it was not possible to make any larger particles with our CAST.

The HAP prototypes sampled the CAST aerosol in parallel with an SMPS (TSI 3080 with long DMA + CPC 3775, AIM 9.0 software), which was used as a reference instrument. Figure 10 shows the result of these experiments in comparison with the corresponding monodisperse results.

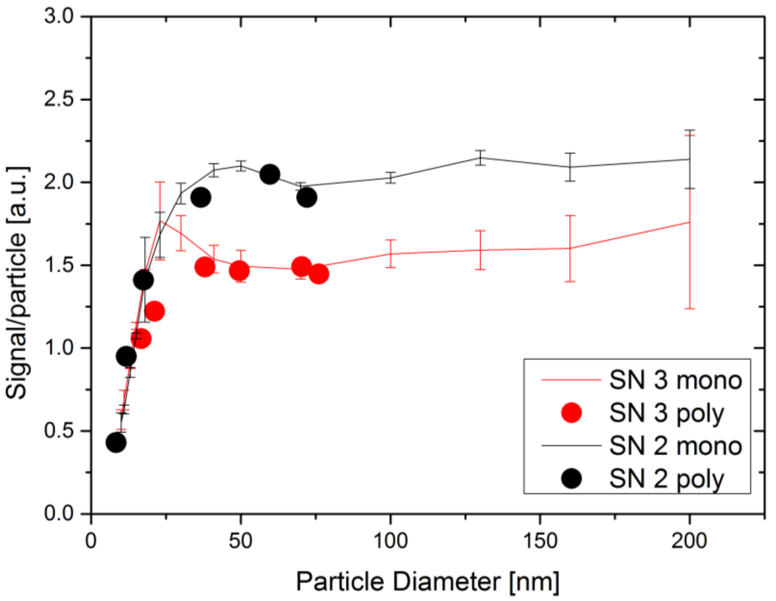

Figure 10: comparison of counting efficiency determined from monodisperse and polydisperse calibration experiments.

From Figure 10, we can see that the polydisperse results agree quite well with the monodisperse calibration points, but that the SMPS-derived results appear to be a bit too low in general. Since the SUREAL sensor will see polydisperse aerosol, we used the average of all data points in the plateau region (>~50nm) to derive a calibration constant for the two sensors.

3.4 Verification with polydisperse aerosol and the CPC

The SMPS uses several assumptions in its data inversion (e.g. the charge distribution on the particles is assumed to follow the published Wiedensohler charging probabilities, whereas real charging probabilities are known to depend on the exact type of neutralizer used and on particle morphology; multi-charge correction + diffusion correction also need some assumptions). The comparison of the HAP prototypes with the SMPS in section 3.3 showed that the prototypes appear to work well for the polydisperse aerosol that will be encountered in real engine emission measurements; and that the features in the calibration curve such as the overshoot in the counting efficiency of prototype 3 are averaged out when measuring polydisperse aerosols. We performed one final experiment where the polydisperse CAST aerosol was diluted to a concentration level of around 25'000 pt/cm^3 so that – in addition to the SMPS - we could compare the HAP prototypes directly with a second 3775 CPC in single counting mode. This test revealed that the SMPS overcounted the particle number compared to the CPC by a factor 1.2. We therefore used this factor to adjust the calibration constant determined in section 3.3 so that the SUREAL prototypes agreed with the 3775 in single counting mode rather than with the SMPS, because we deemed the CPC to be more reliable than the SMPS.

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4 CONCLUSIONS

We have built 3 prototypes of our heated automotive partectors. The devices were characterized, optimal settings were found to reduce the d_{50} diameter, and the performance of the devices was verified with polydisperse soot aerosol similar to that expected from engine exhaust. Calibration constants were derived from a comparison with a CPC for polydisperse soot aerosol.

Table 1 gives an overview of the most important specifications of the sensor.

Table 1: sensor specifications

Size	178x430x460 mm (Standard 4HE 19” rack mount)
Weight	10 kg
Sample flow rate	1.72 lpm (external flow @ 20°C)
d_{50} cutoff	11-14 nm
Concentration range	~ 10^3 – 10^6 pt/cm^3
Pressure range	Δp at inlet should not exceed ± 5% of ambient pressure
Inlet gas temperature	0 – 400° C

From our evaluation experiments, the sensors appear to be working well, with d_{50} cutoffs very close to the 10 nm that we wanted to achieve. The main open question at the moment is related to the large influence of the particle morphology shown in figure 6 and 7. We have shown that our devices behave quite differently for CAST particles than for the compact NaCl particles.

It is presently still unclear how the devices will perform for real automotive exhaust, i.e. whether or not the soot particles in automotive exhaust will give a similar response as the laboratory-generated CAST soot, as different soot particles may have different morphologies. Also, it is still unclear whether any exhaust gas conditioning (e.g. 1:1 or 1:4 dilution or similar) is necessary for real automotive exhaust. Our goal was to build a device that could operate without any dilution thanks to the high internal temperature of 150°C, but this needs to be proven in practice. The exhaust gas composition might be critical for the charger in undiluted exhaust. These open questions will be answered in further testing in the SUREAL project when our partners use the sensors for automotive exhaust.

5 REFERENCES

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