



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

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Introduction

The present document is supplementary to the delivered demonstrator “D2.4 Advanced high-T Half-Mini DMA”, a technology developed in the frame of WP2 “Technology development”, Task 2.4 “Advanced DMA” of the SUREAL-23 project.

The document aims to briefly present the Half-Mini Differential Mobility Analyzer instrument (hereafter referred to as the *Advanced Half-Mini DMA*), developed to be suitable for hot aerosol sampling in the context of sub-23 nm exhaust particle measurements. The document describes the new instrument, summarises its principle of operation and presents the main outcomes of preliminary testing and validation (performed by the technology developer, SEADM) as well as the evaluation of its operation with model and real exhaust aerosols (performed by APTL/CERTH).

Abbreviations list

APTL	Aerosol and Particle Technology Laboratory
CA	Consortium Agreement
CPC	Condensation Particle Counter
D	Deliverable
DMA	Differential Mobility Analyzer
EAG	Electrospray Aerosol Generator
EC	European Commission
ES	ElectroSpray
EU	European Union
GA	General Agreement
GC	Gas Chromatography
HM	Half Mini
HM-DMA	Half mini Differential Mobility Analyzer (also referred at the DOW and hereafter as “Advanced HM-DMA”)
INEA	Innovation Networks Executive Agency
M	Month
MS	Milestone
PC	Project Coordinator
PEEK	PolyEther Ether Ketone
R&D	Research and Development
RDE	Real Driving Emissions
RTD	Research and Technological Development
SESI	Secondary Electrospray Ionization
SMPS	Scanning Mobility Particle Sizer
SoA	State-of-the-Art
THABr	Tetraheptylammonium Bromide
WP	Work Package



Short Project Overview

It is known that a significant proportion of the total number of particles emitted from recently introduced direct injection engines is below 23 nm in diameter. Although the EU aims to regulate those emissions and impose limits on all 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 lack 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 this problem 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 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 for onboard use. The developed instrumentation will assess sub-23 nm particle emissions from both Diesel and GDI vehicles, accounting for the effect of the fuel, lubricants, after-treatment 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 regulatory authorities. The project will deliver systematic characterisation 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 (SMALL REVIEW)

Differential Mobility Analyzers (DMAs) have been used for particle classification since the early twentieth century [1]. Multiple variants have been tried [2], the most popular being the design of Knutson and Whitby [3], commercialised by TSI as part of the Scanning Mobility Particle Sizer (SMPS) system. However, most DMAs have limited performance at very small sizes due to diffusion broadening and to traditional aerodynamic designs unsuited to reach sheath gas flow rates larger than 30 l/min needed to overcome diffusion broadening and particle losses. SEADM and Yale University hold several patents [4] of DMAs for ultra-fine aerosols (in the range of nm), maintaining high resolution at said sizes by using supercritical laminar flow (Reynolds numbers in the order of hundreds of thousands) and compact DMA designs [5].

2 ADVANCED HALF-MINI DMA DESCRIPTION

2.1 Innovation

The Half-Mini DMA (SEADM) is a robust tool for aerosol measurement that offers high resolution for particle sizes in the range 1-15 nm operating at ambient temperature. Two major developments were implemented for the needs of SUREAL-23 to deliver the upgraded model of *Advanced Half-Mini DMA*:

- Particle size range was extended to 30 nm in order to cover the entire sub-23 nm size range but also overlap with current state-of-the-art methods that measure down to 23 nm.
- High-temperature operation was established offering the possibility of measuring aerosol flows up to $T=200$ °C.

Modifications were performed in collaboration with Professor Juan Fernandez de la Mora (Yale University), a SUREAL-23 associate partner from the USA.

The successful modifications, enabling an increased sample/sheath flow rate with nearly ideal response, involved: (i) an inlet ring with many perforations to assure complete axisymmetry of the sample flow as it enters the analyzer region, and (ii) geometrical changes to the sample inlet region directing the sample flow jet into a nearly axial direction, to avoid the formation of flow separation near the inlet. These results have been published in the *Journal of Aerosol Science* [6], acknowledging the support received from the SUREAL-23 project.

In order to operate at high temperature, it was necessary not only to design a suitable heating system but also to perform research on materials that could be integrated into the new high-temperature DMA. High resistivity glass and PEEK¹ were used for electrical insulation parts to achieve operating temperatures of 200°C without melting problems, at SEADM facilities. Also, as there is no commercially available radioactive ioniser able to operate at high temperature, the Secondary ElectroSpray Ionization (SESI) technique (which is commonly used for molecular species, rather than aerosols) was used to charge the exhaust sample particles. SESI can be implemented at high temperatures and does not use ionising radiation as its working principle. The SESI ioniser and interface with the DMA was designed at SEADM, whereas the SESI performance characterisation was carried out at APTL. Furthermore, additional innovations were also implemented such the re-design of the control modules and the development of new

¹ Polyether ether ketone polymer material.



control software.

Table 1 presents the specifications of the *Advanced Half-Mini DMA*.

Table 1. *Advanced Half-Mini DMA Specifications*

Classifier	
Aerosol flow rate	1.5 - 10 lpm
Sheath flow rate	15 - 500 lpm
Aerosol temperature range	10 - 200 °C
Voltage	-5000 to 5000 V
Particle size range	1 - 30 nm
Length	2 cm
Secondary ElectroSpray Ionization (SESI)	
Maximum Voltage	2000 V
Operation temperature	50 – 100 °C
Electrometer	
Measurement range	0 - 2 V
Aerosol flow	1 - 5 lpm



2.2 Principles of Operation

A DMA operates by combining an electric field and a perpendicular flow field of gas (called sheath flow) to separate charged particles (Figure 1). In particular for the Half-Mini DMA, the separation cell is cylindrical, the flow field is parallel to the axis, and the electric field follows radial direction. Particles are introduced from the outer diameter, and only selected particles are collected at an exit slit on the inner electrode. The outer electrode is grounded; while the inner electrode is swept from -5000 to +5000 V. (1) (Knutson and Whitby, 1975) relates mobility (Z) with operating voltage (V),

$$Z = \frac{Q \ln \frac{R_2}{R_1}}{2\pi L |V|} \quad (1)$$

where Q , R_2 , R_1 and L are the sheath flow, the outer and inner radii and the classification length, respectively. Particle diameter, d , can be calculated from mobility by means of the following equation, based on the Epstein formula (2),

$$d = \sqrt{\frac{4e^{-\lambda}}{8.39\mu\xi Z}} \quad (2)$$

where e^- , ξ , λ , and μ are the particle charge, an empirical constant (1.36), and the carrier gas mean free path and dynamic viscosity, respectively.

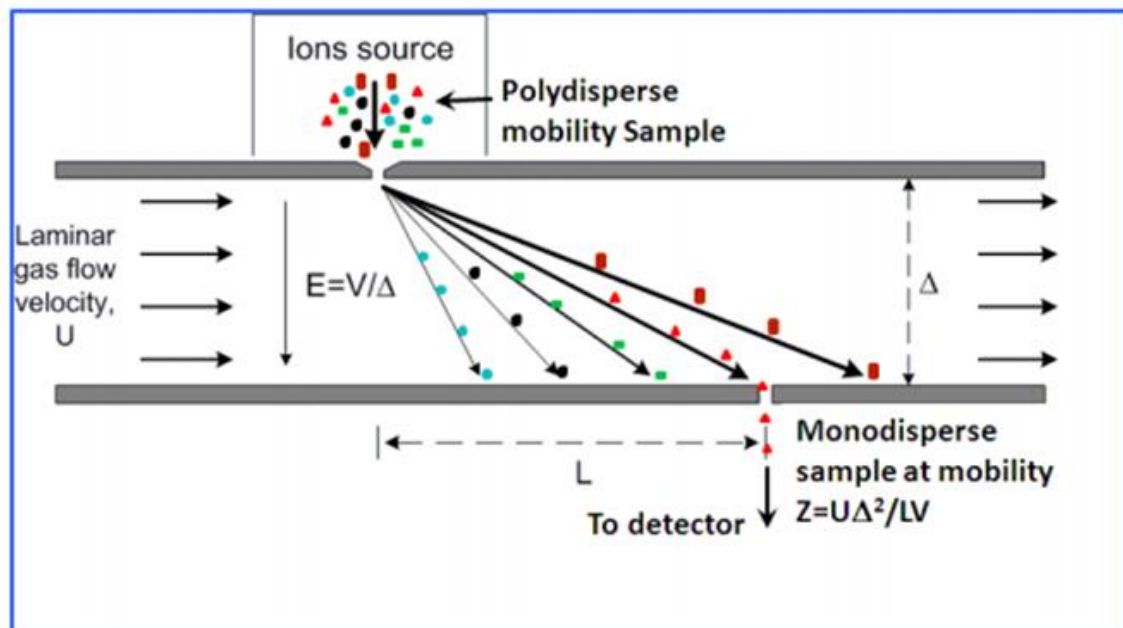


Figure 1 DMA operation principle

While natural charged aerosols do not need any pre-treatment, neutral aerosols must be charged prior the Half-Mini DMA, so the system includes a SESI chamber prior the Half-Mini DMA inlet. The SESI ioniser generates a cloud of ions using an electro spray. These ions are mixed with the sample and transfer their charge to the neutral aerosol particles.

Collected particles are measured by means of an electrometer [7]. The *Advanced Half-Mini DMA* works at high temperature to prevent condensation of volatiles and the associated measurement artefacts. This is achieved by heating the sheath flow and thermally insulating the separation cell. The sheath flow gas is continuously taken from the room and released after being used in the *Advanced Half-Mini DMA* using a blower installed downstream the separation cell. This

architecture creates some vacuum at the sample inlet, allowing sampling atmospheric aerosols without additional pumping. Since the blower cannot work correctly at high temperatures, a cooling element is placed before the blower inlet.

Figure 2 shows the architecture of the system (including control elements). Figure 3 shows some photographs of the *Advanced Half-Mini DMA* when being assembled at SEADM test bench, while SESI ioniser is shown in detail in Figure 4.

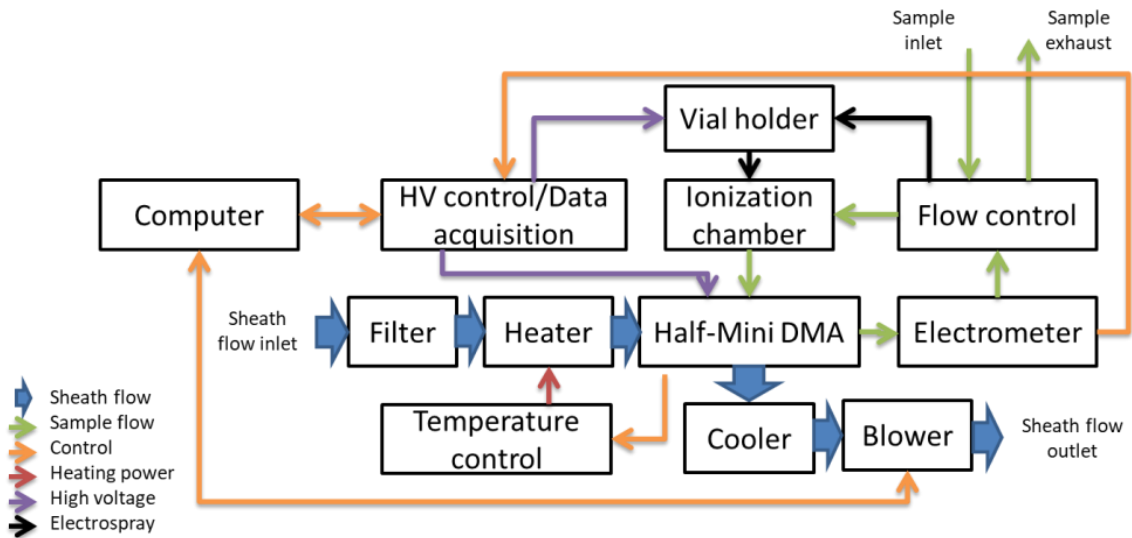


Figure 2. Diagram of the Advanced Half-Mini DMA, showing control signals, flows and electrical connections.

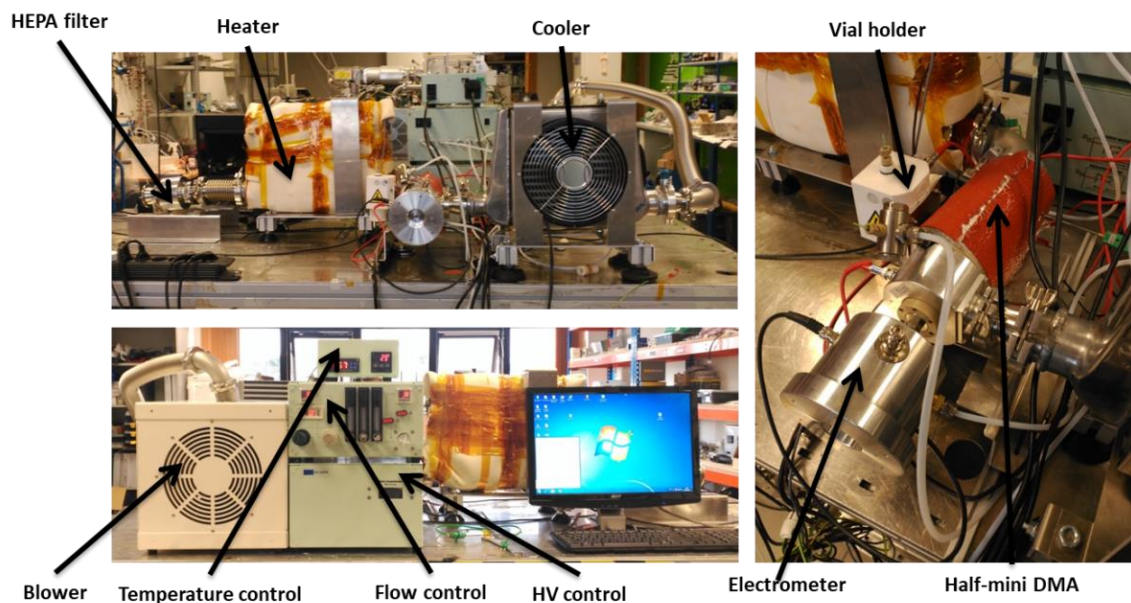


Figure 3. Advanced Half-Mini DMA assembled at SEADM's facilities.

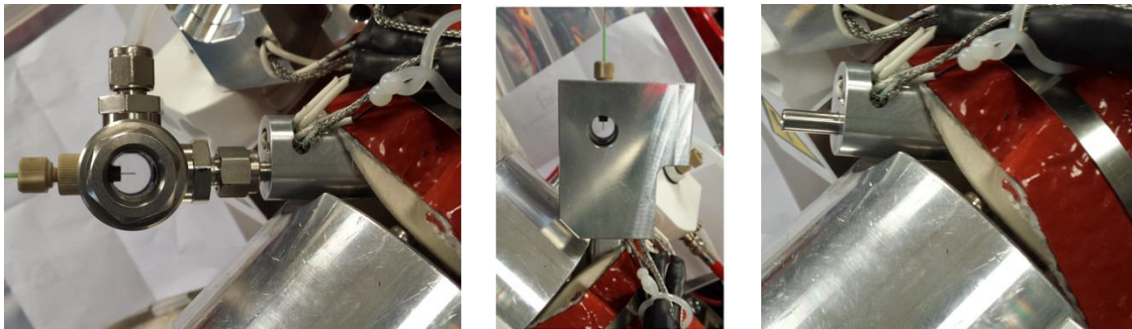


Figure 4. SESI ioniser. Left: SESI chamber; centre SESI chamber with its heating cover assembled; right: detail of the heated transfer line from the SESI chamber to the Advanced Half-Mini DMA.

2.3 Preliminary Experiments

After successful room temperature tests of the aerodynamics of the improved DMA, performed at Yale University, additional modifications and testing were carried out at SEADM to adapt the Half-Mini DMA for high-temperature operation (Figure 5). When raising the temperature, the position of the peaks and the resolving power were kept within acceptable values for the required operating conditions, with a slight downward trend. As the temperature rose, the Reynolds number was lower, and thus the peak voltage slightly drifted to smaller values. The higher diffusion broadening explains the trend in resolving power. In these preliminary tests, the thermal fatigue problem – to be addressed in Section 2.2 – was not noticed yet. The integrated heating power was enough to heat far larger flows than those finally considered for SUREAL-23 while the insulation was found to have sufficient efficiency. For a sheath flow inlet temperature of 210°C, the outlet remained at 187°C, thus eliminating particle nucleation and/or condensation for samples of similar temperature. The electrometer stayed at 30°C, avoiding overheating of the lithium battery.

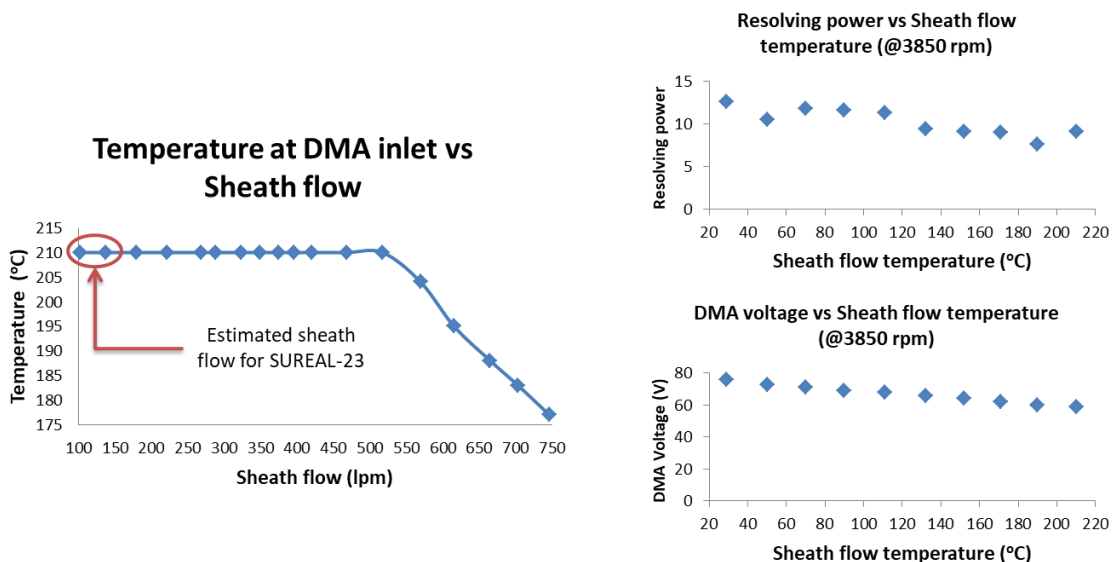


Figure 5. Advanced Half-Mini DMA high temperature tests

Finally, the *Advanced Half-Mini DMA* was tested by analysing a sample aerosol of THABr generated by means of the integrated electrospray. As shown in Figure 6, the results were repeatable. This is a typical test to check and calibrate such DMA devices, where the scanning voltage values of known ions THA^+ (monomer) and $[\text{THA}_2\text{Br}]^+$ (dimer) are taken as references to calculate the mobility Z .

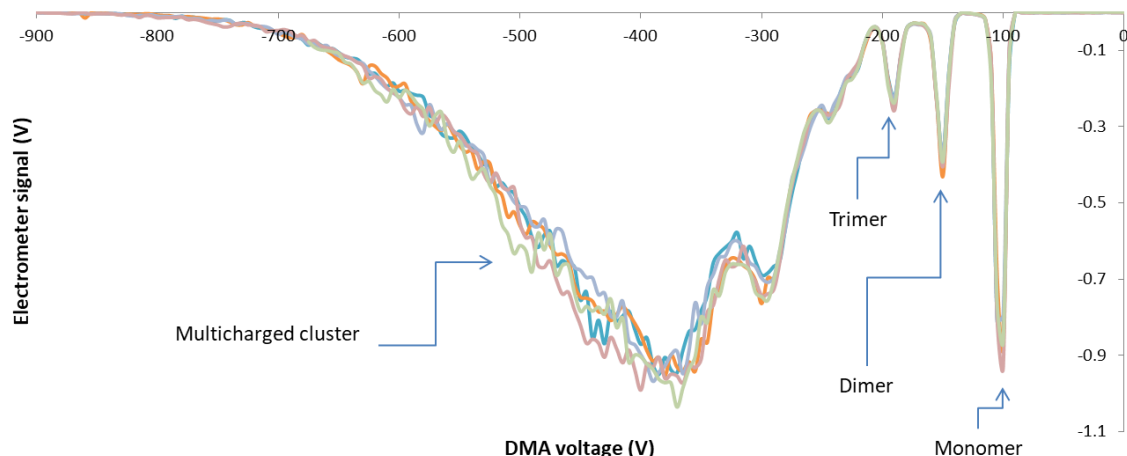


Figure 6. Advanced Half-Mini DMA THABr spectra (blower speed: 5000 rpm; sheath flow temperature: 110°C)

3 EVALUATION of ADVANCED HALF-MINI DMA OPERATION

Following the *Advanced Half-Mini DMA* preliminary experiments performed by SEADM, an evaluation campaign for the system's operation was designed and performed by APTL/CERTH. The objective of the campaign was to determine and evaluate the system's size resolution, detection accuracy under hot operation, as well as to explore the size and concentration range of operation.

A set of standard aerosol generators in conjunction with reference instrumentation for aerosol characterisation was used to generate and characterise selected reference aerosols in the sub-23nm mobility size range. Specifically, an Electro spray Aerosol Generator (EAG) (TSI, 3482) was applied for monodisperse protein particles generation (albumin, ferritin) and solid particles generation (NaCl). Additionally, a standard propane diffusion flame soot generator, the Combustion Aerosol Standard (CAST, Matter Engineering) burner was used for exhaust-like particles generation. It was necessary to modify the typical CAST operation, which provides nanoparticles above 30 nm, by appropriate adjustment of the essential flame and post-flame flows (C_3H_8 fuel, combustion air, N_2 quench flow) in order to generate an aerosol with nominal particle diameters in the sub-23 nm particle size range, well characterized in APTL's recent study [8]. The CAST-generated test aerosol was introduced into an oxidation catalyst maintained at 450°C, oxidizing any volatile compounds and providing a repeatable aerosol of purely solid particles. When needed, reference aerosols were also characterized by the Scanning Mobility Particle Sizer (SMPS) system, consisting of a nano-DMA (TSI, model 3085) and a Concentration Particle Counter (TSI, model 3776).

3.1 Sizing accuracy and resolution

The above described supercritical operation of the *Advanced Half-Mini DMA* can lead to high resolution scanning from -5000V to 5000V. This corresponds to an upper limit of the mobility size range that is either 29nm or 31nm, depending on the system's operation temperature; hot (150°C) or ambient respectively. The lower particle detection limit is constrained by the unavoidable "signature" of the SESI's electro spray solution occupying the size spectrum up to 4 nm. SESI's "scanning signature" depends on the purity level and chemical composition of the ES solutions.

We define system's resolution R as the inverse of the relative width of the mobility peak for a

monomobile aerosol:

$$R = \frac{d}{\Delta d} \quad (3)$$

where d is the particle diameter at the maximum of the mobility peak and Δd is the distribution mode full width at the peak's half height.

Two different monodisperse particle generation methods were used for the *Advanced Half-Mini DMA* resolving power evaluation. Accordingly, monodisperse protein particles and protein complexes (dimers) were generated with the EAG. Two different protein solutions were used: albumin from bovine serum (Sigma Aldrich, A7638) and ferritin from equine spleen (Sigma Aldrich, F4503) in a 20 mM ammonium acetate buffer solution. Figures 7a,b show the particle size distributions of albumin and ferritin, respectively. The first peak that appears at smaller sizes corresponds to the protein particle (monomer) while, when moving to larger sizes, we measure protein complexes (dimer, trimer etc.) created due to the high protein concentrations in the electrospray solution. The protein particle sizes measured with the *Advanced Half-Mini DMA* are in agreement with literature values [10].

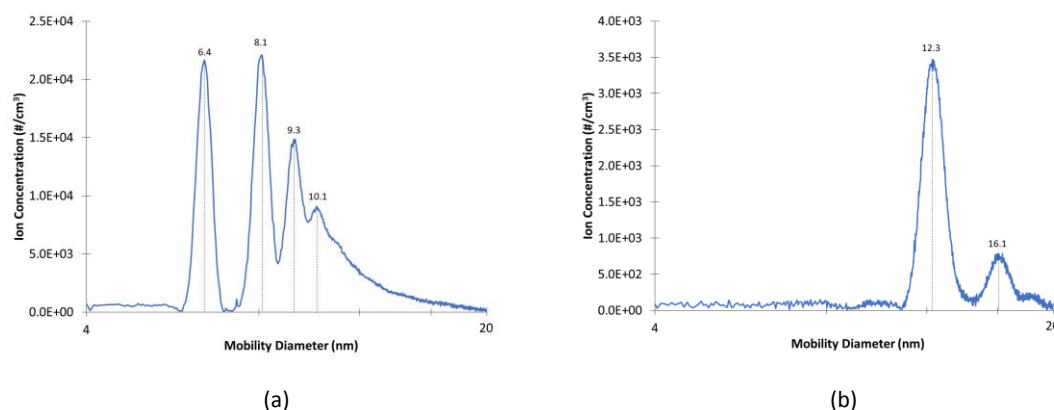


Figure 7. Particle size distribution of a) Albumin protein particles, and b) Ferritin protein particles, measured with *Advanced Half-Mini DMA*.

The system resolution was also evaluated with the tandem DMA method using a nanoDMA (TSI, model 3085) for a first stage of (upstream) size selection. In particular, size selected sub-23 nm solid soot particles, generated by CAST, with mobility diameter of 15 nm, were introduced into the *Advanced Half-Mini DMA* operating at elevated temperatures.

Table 2 summarises the calculated resolutions as well as the standard deviation, σ_g , of the particle size distribution. The mean diameter is calculated as the weighted average of five mobility diameters that include the diameter at the highest signal and its four neighbouring diameters that correspond to the following higher signals. A similar approach is followed when DMAs operate in a stepwise or static mode [11].

Table 2 shows that *Advanced Half-Mini DMA* offers high resolution, $R > 7$, when measuring monodisperse aerosol particles in the sub-23 nm size range. This finding applies both to protein particles and complexes as well as to soot particles. Indeed, particle nature is not expected to influence system's measurements due to the use of an electrometer for the particle counting. These findings, in agreement with preliminary tests performed by SEADM and Yale University, confirm the applicability of the modified Half-Mini DMA to a wide particle size range while maintaining the high resolution.

Table 2. Mean diameter D_{mean} , resolution R and standard deviation σ_g of the Advanced Half-Mini DMA

		Step-wise method	De la Mora (2017)	Gaussian distribution fit	
Electrosprayed protein ions	X-mer	D_{mean} (nm)	R (-)	D_{mean} (nm)	σ_g
Albumin	monomer	6.4	14.7	6.5	1.03
	dimer	8.1	13.3	8.1	1.03
Ferritin	monomer	12.4	9.6	12.3	1.05
	dimer	16	10.4	16.1	1.05
Size selected Soot particles		15.2	7.3	15.1	1.06

3.2 Hot operation mode accuracy

Additional sizing accuracy tests at *Advanced Half-Mini DMA's* hot operation mode were performed with sodium chloride particles (NaCl) generated by EAG and catalytically-treated solid CAST soot particles. The SMPS system described previously was used as a reference system. The scope of these experiments was the system's hot operation accuracy validation. Figures 8a,b plot the NaCl and the CAST-generated soot particle size distributions respectively. Note that y-axis is normalized. The excellent agreement between the reference system and the *Advanced Half-Mini DMA* suggests the accuracy of the hot temperature measurements which are of great importance for achieving the goals of the SUREAL-23 project.

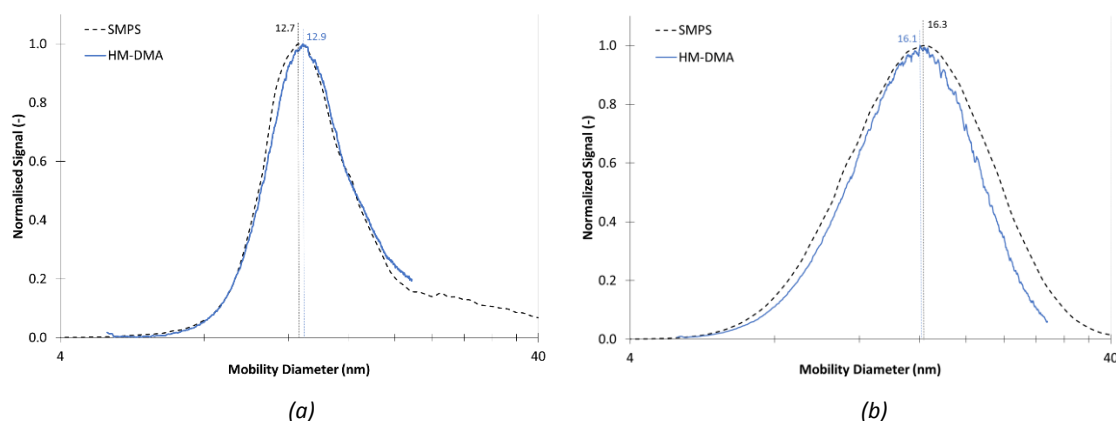


Figure 8. Normalized Particle size distribution of a) NaCl particles, and b) CAST-generated soot particles, measured with *Advanced Half-Mini DMA* at the hot temperature mode and compared with an SMPS (NanoDMA 3085, CPC 3776).

Additionally, hot aerosol sample measurements were performed in the sub-23 nm size region as recently presented [9]. Specifically, solid soot particles with different temperature generated by CAST and treated by an oxidation catalyst were measured by the *Advanced Half-Mini DMA* in the hot operation mode. The inlet temperature was either close to ambient, $T=34^{\circ}\text{C}$, or elevated, $T=140^{\circ}\text{C}$. Note that in typical PMP applications the aerosol flow after the first hot dilution stage is $T=150^{\circ}\text{C}$. Figure 9 plots the particle size distribution measured with the *Advanced Half-Mini DMA* at the two different temperatures. As depicted at Figure 9 detected mobility size distribution was identical for cold and hot inlet aerosol flow having the same $d_m=13.2$ nm, $\sigma_g=1.3$, and total ion concentration (<2% differences). Thus, *Advanced Half-Mini DMA* may measure hot sample aerosol without introducing errors.

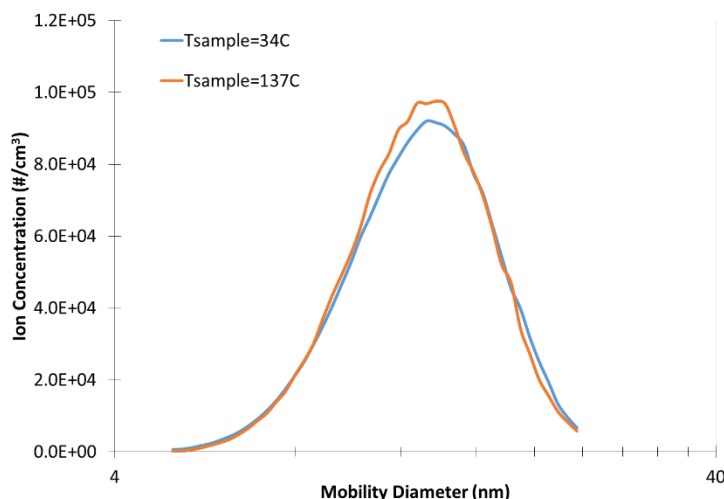


Figure 9. Particle size distributions of CAST solid soot particles, measured with Advanced Half-Mini DMA. Aerosol sample is measured either warm (137 °C) or at close to ambient temperature (34 °C) without affecting system's selection and detection capability.

3.3 Concentration range and accuracy

The supercritical flow *Advanced Half-Mini DMA* is combined with a fast electrometer with 100 ms response time, developed and evaluated by De la Mora *et al.* [7]. Such response capability is 10 times higher than the traditional electrometers, forming a significant advantage of the system, especially in the context of forthcoming Real Driving Emissions requirements for real-time size distribution measurements.

The electrometer's average background signal, 0.005V [7], determines the system's ions concentration lower detection limit which corresponds to 1.25×10^3 ions/cm³. The electrometer's upper detection limit is 2 V and corresponds to 5×10^5 ions/cm³. By accounting the sheath to aerosol flow ratio, the limits change by a factor of approximately 10. These limits are indicative and vary considerably with the electrometer's inlet flow.

The concentration measurement range is restricted compared to a CPC that can count from a particle. However, there are several advantages in the use of an electrometer. Firstly, particle concentrations even in the 10^6 range are accurately measured without the need of approximations. Secondly, there is no particle nature counting efficiency dependence that may influence measurements especially in the sub-23 nm size range. Finally, the electrometer's fast response permits very fast scans which can potentially provide a full-size spectrum per second.

The electrometer's fast response accuracy was validated by comparing the total ion concentration measured by the *Advanced Half-Mini DMA* setup against the total particle concentration measured with the reference SMPS system already described. Test aerosol was salt (NaCl) particles generated by the EAG. The EAG is equipped with an X-ray neutralizer that was used as the only particle charger in this setup in order to avoid charging efficiency influence on the total ion/particle number measurement. Measured concentrations were in agreement with an average difference of 17%.

4 CONCLUSIONS

The *Advanced Half-Mini DMA* system is a supercritical DMA, with a 2 cm working section, initially developed at Yale University and subsequently improved by SEADM to be able to classify aerosol particles in the size range 4 – 30 nm with high resolution [6] and fast spectrum acquisition [7]. The working principle involves particle ionisation (unipolar charging) by a secondary electro-spray and classification under the simultaneous action of the well-controlled axial sheath flow and a strong radial electric field, whereby only the particles of a specific mobility are transmitted, via the DMA outlet, to the electrometer.

The main novelty of the system consists in its ability to maintain a laminar flow even in the presence of high sheath flow rate which is necessary for classifying particles with high resolution in the particle size range of interest. Innovative geometry is used to avoid diffusion broadening and turbulence-related effects.

An additional significant feature of the new instrument is that it can accommodate a hot sample flow by employing a heat-tolerant semiconducting glass tube in the path from the inner electrode to the grounded outlet of the DMA, yielding a device capable of operating at temperatures up to 200 °C. By reducing or eliminating the need for high sample treatment (i.e. the PMP-compliant system), known artefact creation mechanisms are avoided resulting in more reliable solid particle emission measurements.

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