Revisions summary

The nano-scanning electrical mobility spectrometer (nSEMS)
and its application to size distribution measurements of 1.5-25 nm particles

Revisions in response to Reviewer 1

Reviewer 1 identifies a number of interesting questions, but they are generally beyond the scope of this paper, pointing to alternative ways to assemble an instrument. The reviewer explicitly states:

> They have accordingly no duty to engage in the more extensive discussion I propose. Nevertheless, given their wide experience, their response would be exceedlingly useful to the many colleagues involved in these measurements, as well as on the many possible alternative experimental tools that were not chosen.

We have provided extensive responses to the reviewer's comments in the on-line response. A thorough analysis of each of the points would significantly lengthen the paper, and would detract from the objective of the paper which is to describe an instrument that has already yielded important insights into new particle formation under simulated atmospheric conditions. This may be a useful topic for a review or tutorial paper, one that addresses the philosophy of measurement design. The is always a danger of allowing the desire for perfection to prevent getting the data that is needed to answer the scientific question that one is trying to address. Compromise is often necessary in the interest in having an instrument that will reliably provide the required data when the instrument must be deployed.

We, therefore, decline to add the additional dimensions to this paper that the reviewer suggests, but will certainly consider ways to open the discussion of the important questions that he raises in the literature in the near future.

Revisions in response to Reviewer 2

The nanoscaning electrical mobility spectrometer is a very useful instrument because of its response time and sensitivity for small particles.

Few questions or methods are not discussed in the text or are not clear. I listed few of them below. That will help the readers to have the responses. It will help to precise that the particles used to calibrate the instrument are electrically (positive) charged. Sizing sub 2 nm whith a charger dma cpc is another problem to my opinion. Indeed the chemistry of the particles is changed by the attached ion on the particle. And the chemistry is important for the activation of sub 2 nm particles, see Kangasluoma et al (2013) in Aerosol Sci Technol.; Jiang et al. 2011 in the same journal have used by the way the concept of SMPS to detect neutral particles in the sub 2 nm range. You should cite their work.

Reference to Kangasluoma et al. (2013) has been added with discussions as to how the results from Kangasluoma show that more counts are recorded for "particles" smaller than 1.5 nm than are present in the incoming sample (as measured by mass spectrometer), leading to considerable ambiguity and uncertainty in size distributions below this size. Hence we do not claim to measure size distributions below 1.5 nm. The following text has been added:

> While this CPC can detect particles as small as 1 nm diameter, and the ROMIAC can classify particles of that small size (Mui et al., 2013, 2017), Kangasluoma et al. (2013) observed larger numbers of apparent particles smaller than 1.5 nm diameter than were present in the calibration aerosol that they generated, and attributed the excess particle counts to the activation of gas ions within the CPC. Lacking a definitive method for discriminating between gas ions and particles in the sub-1.5 nm size range, we do not report size distributions below 1.5 nm.

Jiang et al. 2011 in the same journal have used by the way the concept of SMPS to detect neutral particles in the sub 2 nm range. You should cite their work.

We have added at line 67 in the annotated version of the document:

> ..., which was integrated with a DMA to produce the first SMPS system to measure size distributions approaching 1 nm (Jiang et al., 2011a) that agree closely with mass spectrometric data in the overlap region between the two methods (Jiang et al., 2011b).

1°/ Why this 'precise' diameter of 1.5 nm? Is it a limit of the detector or the classifier or the charger. Tetraethylammonium bromide produces 1.11 nm (mobility diameter) has been used by Attoui (2018 Journal of aerosol science).

As noted above, Kangasluoma et al. (2013) performed calibration experiments using particles of known composition in which they observed more counts than there were calibration particles in the aerosol sample flow. In our experiments, we observed such phantom counts as well, interpreted as gas ions. We have cited the Kangasluoma work to support our decision not to claim that we have robust particle size distribution measurements below 1.5 nm where these phantom counts make the measurements ambiguous and uncertain. This ambiguity remains an important challenge for aerosol measurements.

2°/ The charger is the weak point of the study since there is no experimental work on the charging state of sub 3 nm with a bipolar charger. The authors are not giving lot of details about the charger they have used in terms of size of their chamber, concentration of ions nor residence time of the ions in the chamber.

We agree that the lack of characterization of the charge leads to an incomplete description of the full measurement system employed in the the measurements shown in Fig. 11. The emphasis of this paper is on the use of the opposed migration classifier in voltage-scanning mode to determine particle size distributions in the low nanometer size range. All of the instrument characterization experiments and data begin with a charged aerosol. The reference concentrations for all of those measurements are the concentrations of particles that can be classified and detected, i.e., mobility-classified particles that were charged either by the soft x-ray charge conditioner, or were produced as charged particles from the hot-wire source. The instrument-characterization/calibration data are, therefore, not compromised in any way by the lack of calibration data on the charge conditioner. The charge conditioner has shown no clear differences from other radioisotope or soft x-ray charge conditioners in preliminary tests, but completion of those tests was delayed by laboratory closure due to COVID-19. The charge distribution is consistent with the Wiedensohler (1988) fit to the Hoppel and Frick (1988) calculations using Fuchs (1964) theory. We have used that distribution in the analysis of data for Figure 11, which is the only place where the charge distribution enters the analysis in this paper.

The charge-state of the aerosol is the biggest source of uncertainty in mobility measurements of particle size distributions in general, even for conventional DMAs. We are, therefore, undertaking detailed characterization of this charge conditioner, as well as others to which we have access. This will be the subject of a future paper.

Rather than dwelling on the causes for delay, we have redefined the nSEMS as consisting of the ROMIAC classifier and the two-stage CPC, and pointed out that the soft x-ray source provides the required charged aerosol for the calibration studies reported in this paper. This has required a number of edits throughout the paper and the abstract. Key revisions include:

The Abstract now states:

The nSEMS consists of a novel differential mobility analyzer, and a two-stage condensation particle counter (CPC). The mobility analyzer, a radial opposed migration ion and aerosol classifier (ROMIAC), can classify nanometer-sized particles with minimal degradation of its resolution or diffusional losses.

The soft x-ray charge conditioner is mentioned at the end of the Abstract as:

Using a soft x-ray bipolar ion source in a compact housing designed to optimize both nanoparticle charging and transmission efficiency as a charge conditioner, the nSEMS has enabled measurement of the contributions of both neutral and ion-mediated nucleation to new particle formation.

The final paragraph of the introduction now reads:

In this work we show the development of a nano-scanning electrical mobility spectrometer (nSEMS) that features a fast-scanning OMAC, and a two-stage CPC, to acquire fast and accurate particle size distributions in the range of 1.5 - 25 nm. Here we describe the design and characterization of the scanning OMAC in detail as that is the unique component of the instrument which enables a sizing range that is not possible with a constant-flow DMA.

A radial-flow version of the OMAC that is capable of classifying charged particles or ions as small as 1 nm in diameter (Mui et al., 2013, 2017) is used in this instrument. The two-stage CPC includes a fast-mixing activation stage using DEG as working fluid, followed by an eco-friendly, fast-response, water-based CPC (Hering et al., 2019). Other CPCs, either two-stage ones like the one we have employed, or single stage CPCs that are capable of counting particles as small as 1 nm could be integrated into the nSEMS, so we limit our discussion of the 2-stage CPC to its essential features, its integration into the nSEMS, and the resulting performance. The nSEMS analyzes a charged aerosol, which can be naturally charged or one that is processed through a charge conditioner (CC). The present paper focuses on nanoparticle sizing and detection downstream of the CC. The integrated transfer function of the nSEMS system was derived based on both experimental results and finite-element modeling using COMSOL Multiphysics^{TM}. The nSEMS has been intensively used in the Cosmics Leaving OUtdoor Droplets (CLOUD) experiments at CERN, in which its size resolution and fast response have made it possible to follow very rapid growth of freshly nucleated nanoparticles, and to identify a new mechanism for new particle formation in a highly polluted atmosphere (Wang et al., 2020). A comparison of nSEMS data with measurements from other well-calibrated particle sizing instruments at CLOUD confirms its capacity to provide reliable size distribution in the low-nanometer size regime.

In the discussion of the nSEMS system, we now state:

The aerosol enters the nSEMS after passing through a soft x-ray CC at a relatively high flow rate of 4.6 L min^{-1}. A smaller, 1.2 L min^{-1} polydisperse aerosol sample flow, from the core of the larger flow is introduced into a ROMIAC, while the remainder is exhausted. The high flow rate through the CC and the core-sampling flow spitter are designed to minimize losses of the highly diffusive nanoparticles. While the ROMIAC was originally designed to operate at constant voltage, the voltage is continuously varied in an exponential ramp in the nSEMS. Particles transmitted through the ROMIAC are counted using a two-stage CPC to capture the particle size distribution. Detailed operating parameters and default settings are summarized in Table 1.

The CC used in this study employs a soft x-ray source to produce ion pairs from the gas molecules in order to bring the aerosol to a steady-state Fuchs charge distribution that enables estimation of the size distribution whose initial charge state is uncertain. The soft x-ray source has two advantages: (*i*) fewer regulatory hurdles than radio-isotope sources used for the same purpose, and (*ii*) the ability to turn off the ion source in order to measure those particles in the sample that carry charge due to gas ionization by galactic or simulated cosmic rays. It was designed to minimize losses of the smallest particles. Preliminary characterization of the CC, and data from numerous experiments in which it has been applied on conventional DMAs in parallel with other DMAs using conventional CCs, and on the nano-radial differential mobility analyzer (Brunelli et al., 2009) yield results consistent with the Wiedensohler (1988) approximation to the Fuchs charge distribution, with no apparent biases. Since mobility classified, and, therefore, charged particles are used to determine the integrated instrument transfer function that is reported here, the key results from this paper are not affected by any minor deviations from the Fuchs charge distribution that is assumed in inverting data from the nSEMS. Details of the design and quantitative calibration of the soft x-ray CC will be presented in a separate paper.

Wording has been changed slightly in a number of other places, including the conclusions, to keep the message consistent with excluding the soft x-ray charge conditioner from the bounds of the nSEMS as we now define it.

3°/The important flowrate of 4.6 lpm is coming without any explanation about the charging state nor residence time.

The 4.6 L/min flow is designed to minimize losses in the soft x-ray charge conditioner. This will be discussed in the detailed paper on that device.

4°/ Same thing about the losses versus the particles size in the charger, in the conditioner after the dma?

These losses do not affect the calibration studies that are the focus of this paper. They could affect the data presented in Fig. 11, but the high concentration seen at the smallest reported sizes suggest that the losses are not excessive, as do design simulations that will be part of the paper describing the charge conditioner. The losses in the soft x-ray charge conditioner will be quantified in the separate paper that we will write on that device.

5°/ Likewise about the mixing chamber of the mixing type cpc activator. There are no details on terms of size nor activation nor losses.

The discussion of the turbulent mixing activation chamber within the two-stage CPC has been enhanced somewhat, and now reads:

Classified particles transmitted through the ROMIAC are subsequently detected by a two-stage CPC that enables particle counting approaching 1 nm in size (Iida et al., 2009; Jiang et al., 2011a). The first stage employs a fast-mixing condensational activation and growth reactor (Wang et al., 2002) that uses DEG as the working fluid to activate the nanoparticles. This activation stage is based upon the particle size magnifier (PSM) (Kousaka et al., 1982; Okuyama et al., 1984; Gamero-Castano and de la Mora, 2000; Sgro and de la Mora, 2004) in which a cool aerosol sample flow undergoes rapid turbulent mixing with a warm flow that is saturated with vapor to produce the supersaturated state that will activate and grow particles larger than a critical size. The detailed design incorporates modifications reported by Shah and Cocker (2005) that reduce the size of the mixing volume, while maintaining the small growth tube, with a residence time of ~ 10 ms. In the first stage used in this study, supersaturation is produced by turbulent mixing of a 0.3 L min^{-1} flow of hot (e.g., 70 °C) DEG vapor with a 1.2 L min^{-1} cold (20 °C) particle-laden flow. The downstream growth tube is cooled to 10 °C to accelerate particle growth and remove excess vapor. In contrast to the Airmodus particle size magnifier (PSM) and the CPC of Sgro and de la Mora (2004), on which the PSM is based, the mixing time in the activation stage of the present CPC has been minimized (~ 0.12 s) to speed instrument response, as compared with ~ 0.7 s. The design and initial experiments were performed a TSI Model 8210 CPC operating at a flow rate of 1.5 L min^{-1} as the second detection stage. Owing to the slow response of that CPC, it was replaced with a modified, water-based ADI MAGIC^{TM} CPC that serves as the second stage to grow particles sufficiently large for optical detection (Hering et al., 2019). Particle counts are recorded over the nSEMS size distribution scan at 5 Hz. The sample flow rate of the CPC is 1.00 L min^{-1}. Between the activation and booster stages, the flow is split between the water-CPC and a smaller (0.5 L min^{-1}) excess flow to minimize deposition of excessive DEG vapor in the intervening plumbing, and to match the flow to the water CPC.

The specific design follows from Wang et al. (2002), with modifications that we first proposed, but that were first implemented and reported by Shah and Cocker (2005). The residence time in the growth region (condenser) is now specified as requested.

6°/ Likewise about the growth tube (if there is a growth tube) of the mixing type CPC. In page 7 line 47, the authors are talking about the growth tube used by Sgro and Fernandez de la Mora indeed. There are no details about the residence time the inner diameter nor length. Is the growth tube what they call 'condenser'? What is its length and inner diameter?

What was originally labeled the condenser is now identified as the growth tube; it has a residence time of 120 ms, which is the relevant quantity for assessing its effect on data analysis. In this paper, we look at the two-stage CPC as a unit, rather than characterizing each sub-component. The larger second stage CPC dominates the time response. The counting efficiency of the entire two-stage CPC is reported in Fig. 6.

7°/ Same thing for the flow rate of '1, 5 lpm'. Why this particular value?

The 1.5 L/min flow rate was a design decision based upon components we had available. We initially used a butanol CPC as the second stage, but replaced it with a more reliable and faster response water CPC, which is the instrument that we report here.

8°/ A hot wire is used for the calibration of the instrument but there are no information nor reference of this method. Is it the same method used by Peineke et al 2009 (Journal of aerosol science)? If yes why the authors are using a charger in the figure 5. Peineke and Schmidt Ott 2007 (Journal of Aerosol Science) claim that the particles are self-charged in negative and in positive mode.

Hot wire sources for ultrafine partices have been used since before DMAs became available commercially. The device that we used in this study is similar to that of Peineke et al. (2006), so we have cited that work.

9°/ Why the charger (neutralizer) is not used any more in the figure 5 for the same hot wire generator?

The hot wire source generates an abundance of charged particles, so we used them directly.

10°/ The PSM as a sizer has been introduced by Gamero and Fermandez de la Mora (paragraph 3.2 in Gamero 2000 Journal of aerosol science). Not by Sgro and Fernandez de la Mora (2004) as said the authors. Citation: https://doi.org/10.5194/amt-2021-62-RC2

We have included the Gamero-Castano reference as requested, but for consistency have given proper credit to Okuyama and collaborators who introduced the method and the name of particle size magnifier. (There is an 1960 paper written in Russian, by Kogan; the reference that I have for it seems to be incorrect so I have not cited it.) The citation was for the use of the PSM to detect particles, not to size them, but this is a minor point, and not worth quibling over. We have kept the Sgro reference since her much simpler design is the one that is used in the Aermodus Particle Size Magnifier, and that determines the response time of that instrument as discussed in this paper.

Other revisions:

In the course of revising this paper, we found that the discussion of the data inversion method could be confusing, so we revised it to make the method that we used clearer. The revised text is:

Data inversion is required to retrieve particle size distribution of the source particles, $n(\log d_p)$, from the particle counts measured by the CPC, $\mathbf{R}_{\text{nSEMS}}$, which can be represented in matrix form as

$$\mathbf{R}_{\text{nSEMS}} = \mathbf{A}_{\text{nSEMS}}\mathbf{N}, \tag{1}$$

where $\mathbf{A}_{\text{nSEMS}}$ is often referred to as the inversion kernel for the instrument, and $\mathbf{N}$ is the vector of weights for the discretized representation of the particle size distribution, for which we use linear splines on $x = \log(d_p)$ (Mai et al., 2018). The time-series instrument response can be written as $\mathbf{R}_{\text{nSEMS}} = [R_{\text{nSEMS},1}, R_{\text{nSEMS},2}, \cdots, R_{\text{nSEMS},I}]^{\text{T}}$. With the default nSEMS voltage ramp time, $t_{\text{ramp}} = 50$ s, and the CPC data recording frequency, $t_{\text{c}} = 0.2$ s, the vector length for one complete scan is $I = 250$. The particle number counts recorded by the CPC in the ith time bin, $R_{\text{nSEMS},i}$, can be represented as the integral of the total number of particles transmitted over the time interval

$(i-1)t_\mathrm{c} \le t < it_\mathrm{c}$:

$$R_{\mathrm{nSEMS},i} = Q_\mathrm{a} \int\limits_{(i-1)t_\mathrm{c}}^{it_\mathrm{c}} \int\limits_{-\infty}^{\infty} n(x) \sum_\phi p_{\mathrm{charge}}(x,\phi)\, \eta_{\mathrm{CPC}}(x,\phi)\Omega_{\mathrm{nSEMS}}(Z_\mathrm{p}(x,\phi),\beta,\delta,t)\, \mathrm{d}x\, \mathrm{d}t$$

$$= Q_\mathrm{a} \int\limits_{(i-1)t_\mathrm{c}}^{it_\mathrm{c}} \sum_j \int\limits_{x_{j-1}}^{x_j} n(x) \sum_\phi p_{\mathrm{charge}}(x,\phi)\, \eta_{\mathrm{CPC}}(x,\phi)\Omega_{\mathrm{nSEMS}}(Z_\mathrm{p}(x,\phi),\beta,\delta,t)\, \mathrm{d}x\, \mathrm{d}t \tag{2}$$

The particle charging probability from the soft x-ray CC, $p_{\mathrm{charge}}(x,\phi)$, was assumed to be that of the Wiedensohler (1988) approximation, and is computed separately for scans of negative and positive polarity. In order to obtain the instrument transfer function, $\Omega_{\mathrm{nSEMS}}(Z_\mathrm{p}(x,\phi),\beta,\delta,t)$, for each time bin, the simulated ROMIAC transfer functions were first fitted as a Gaussian function:

$$\Omega_{\mathrm{ROMIAC}}(Z_\mathrm{p}(x,\phi),\beta,\delta,t) = a\exp\left(-\frac{(t-b)^2}{2c^2}\right) \tag{3}$$

The three fitting parameters, a, b, and c, were then interpolated over the entire time vector with 250 bins. By substituting the interpolated parameters back into Eq.(3), a ROMIAC transfer function, $\Omega_{\mathrm{ROMIAC}}(Z_p(x,\phi),\beta,\delta,t)$, was generated for each time bin. The fitted transfer functions were adjusted by the empirically determined mobility correction factor, f_z. The nSEMS transfer function for each time bin, $\Omega_{\mathrm{nSEMS}}(Z_\mathrm{p}(x,\phi),\beta,\delta,t)$, was then computed by the convolution of the ROMIAC transfer function and the CPC residence time distribution of Eq.12. The inversion kernel matrix for the ith time bin, and j^{th} particle size bin thus becomes

$$A_{nSEMS,i,j} = Q_a t_c \int\limits_{\log d_{p,j-1}}^{\log d_{p,j}} n(x) \sum_\phi p_{\mathrm{charge}}(x,\phi)\, \eta_{\mathrm{CPC}}(x,\phi)\Omega_{\mathrm{nSEMS}}(Z_\mathrm{p}(x,\phi),\beta,\delta,t)\, \mathrm{d}x \tag{4}$$

We then applied a totally nonnegative least squares (TNNLS) algorithm to retrieve the sample particle size distribution from the inversion kernel and the particle number concentrations detected by the CPC, i.e., solving for $N = \mathbf{A}_{\mathrm{nSEMS}}^{-1}\mathbf{R}_{\mathrm{nSEMS}}$ (Merritt and Zhang, 2005; Mai et al., 2018).

Other small revisions have been made to ensure consistency (especially with respect to the treatment of the charge conditioner, correct typographical errors, formatting of units, supply missing units Fig. 6, change the label of the growth tube on Fig. 1, and other details. All revisions (except those in the two figures), are identified in the PDF version of shows the differences between the present manuscript and the one that was originally submitted.

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