Using particle effective density to determine SMPS-based aerosol mass concentration: application to airborne carbon and titanium nanoparticles

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Abstract. To avoid multiple instruments to be deployed in field measurement campaigns based on airborne nanoparticles characterization, this study aims at investigating the capability of a Scanning Mobility Particle Sizer (SMPS) to provide accurate data relative to the mass concentration. Two series of test nanoaerosols were produced using a spark-discharge generator equipped with carbon or titanium electrodes (modal number diameters between ~ 50 and ~ 170 nm). The mass concentration was monitored by means of a personal AM520 photometer operated in parallel with the SMPS and closed-face cassette sampling for further off-line analysis: gravimetric analysis, and chemical analysis (thermo-optical analysis for carbon, ICP-OES for titanium). For each operating condition, the average number size distribution stemming from the SMPS was converted into mass size distribution accounting for particle effective density and integrated over the whole range of particle diameters to determine the corresponding mass concentration. Results highlight correlations ($R^2 \sim 0.9$) between SMPS-based and chemical analysis or gravimetric mass concentrations below 1 mg/m$^3$, with relative discrepancies lower than ~ 15% and ~ 25%, respectively. The AM520 photometric response remains dependent on particle physical and light scattering properties, which differ with the test dust used for factory calibration.

1. Introduction
In spite of the absence of consensus on the parameters to be characterized when assessing occupational exposure to airborne nanoparticles, mass concentration shall still be considered as a reference metric. Besides, providing knowledge on the number size distribution as well as the number concentration of submicrometer aerosols is also of great interest [1]. Ideally, a multi-metric approach has to be implemented [2-7].

To allow a given task to be associated with a level of exposure or a specific size distribution, these parameters are to be measured in real-time. However, managing multiple direct-reading instruments in the field might lead to technical issues. To overcome this concern, using devices providing information regarding different metrics constitutes a promising alternative.

The Scanning Mobility Particle Sizer (SMPS) is capable of providing accurate data relative to both the size distribution and total concentration, according to both the number and mass metrics. If SMPS constitutes the reference instrument for the determination of the number size distribution [8-14], determining the mass concentration deduced from SMPS data is more complex. In particular, this approach involves the integration of airborne particle effective density [15-20], which constitutes a
parameter that is in most cases unknown, and whose experimental determination requires specific instrumentation, e.g. the Aerosol Particle Mass analyser (APM) [21, 22], the Centrifugal Particle Mass Analyzer (CPMA) [23, 24] or the Aerodynamic Aerosol Classifier (AAC) [25, 26].

This study aims at investigating the reliability of a SMPS to provide the mass concentration of an aerosol when the number size distribution is combined with the effective density of airborne particles.

2. Materials & methods

2.1. Test facility, test aerosols, measurement and sampling strategy

Two series of test nanoaerosols were produced using the CAIMAN facility [27] equipped with carbon \((n = 7)\) or titanium \((n = 9)\) electrodes (Figure 1). In our setup, the test aerosols were sampled from a homogeneous 4-probes sampling volume.

Figure 1. Test facility and measurement active samplers and direct-reading instruments.

Aerosol mass concentration was monitored in real-time by means of a personal photometer (TSI, model SidePak™ AM520). All the AM520 mass concentrations were measured as raw data, that is using the factory calibration factor \(CF = 1\), obtained with A1 Test Dust / ARD1 Arizona Road Dust. In parallel, the SMPS (DMA, TSI model 3081 & CPC, TSI model 3787) was used to measure the number size distribution.

As depicted in Figure 1, sampling for further off-line analysis were also performed to provide reference mass concentration of the test aerosols:

- a 2-pieces closed-face cassette (CFC), sampling line labeled “A” in Figure 1, equipped with 37-mm calcined quartz fiber filter (Sartorius, Q3400) for thermo-optical carbon quantification at 2 L/min airflow rate (Casella, APEX2);
- a 3-pieces CFC, sampling line labeled “A” in Figure 1, with 37-mm PVC filter (PALL, GLA5000) for titanium quantification by ICP-OES at 2 L/min airflow rate (Casella, APEX2);
- a 2-pieces CFC, sampling line labeled “B” in Figure 1, equipped with 25-mm Gravi-Sert filter capsule (Zefon) for gravimetric analysis (Mettler, XP6U, 1µg resolution) at 5 L/min airflow rate (GSA, SG 10-2).

The duration of the different samplings was between 15 and \(~300\) min.
Figure 2 presents the relative number size distributions of the test aerosols produced in this work to investigate the approach based on SMPS allowing the mass concentration to be determined.

Figure 2 shows that the modal diameters covered by the test aerosols range from ~ 60 to ~ 170 nm for airborne carbon particles, and from ~ 50 to ~ 120 nm for airborne titanium particles, with Geometric Standard Deviations of ~ 1.6. The range of number concentration of the test aerosols was 8.2-22 \times 10^5 \text{n/cm}^3 for carbon electrodes, and 4.7-23 \times 10^5 \text{n/cm}^3 for titanium electrodes; the target mass concentrations were lower than 1 mg/m³.

2.2. Determination of the mass concentration from SMPS data
For each operating condition, the average number size distribution stemming from the SMPS was fitted by a monomodal lognormal distribution law \( C_N(d_m) \) by least-squares optimization, further converted into mass size distribution accounting for particle effective density \( \rho_{\text{eff}}(d_m) \), and integrated a range of particle diameters from 1 nm to 10 \( \mu \text{m} \) to determine the corresponding mass concentration:

\[
C_M = \sum_{d_m} C_N(d_m) \cdot \rho_{\text{eff}}(d_m) \cdot \pi d_m^3 / 6
\]  

(1)

The following particle effective density \( \rho_{\text{eff}}(d_m) \) were used for the determination of the mass concentration from SMPS data (where \( d_m \) is in nm) as characterized earlier by tandem DMA-APM measurements [21]:

\[
\rho_{\text{eff}}(d_m) = 20.135 \cdot d_m^{-1.020}
\]
The resulting concentrations were compared to data coming from the other approaches.

2.3. Quantification of Carbon mass concentration by thermo-optical analysis
To determine the mass concentration of Carbon aerosols, particles were collected on a quartz fiber filter (Sartorius QMA 37) which had previously been decarbonized by heating at 450°C for 48 hours in a ceramic oven designed for this purpose and not polluted by other uses. The mass of carbon was quantified using a specific carbon analyzer, the Thermo optical OC/EC aerosol analyzer (Sunset Laboratory, Inc.).

After sampling, a punch of the filter was heated in a quartz oven according to temperature steps up to 980°C under an inert and then oxidizing atmosphere. Under inert atmosphere, elemental carbon is thermally stable and only organic carbon were volatilized and then oxidized by catalysis into carbon dioxide to be quantified after reduction into methane by flame ionization spectrometry (FID). Second, at high temperature and in the presence of oxygen, elemental carbon burns, thus forming in turn carbon dioxide which will be detected and quantified. Throughout the analysis, the optical properties of a laser beam through and on the surface of the sample, in transmission and reflection are monitored to differentiate the elemental carbon fraction from the organic one.

For this analysis, the thermal protocol used is the EUSAAR 2 protocol (European Super-sites for Atmospheric Aerosol Research) [28, 29], with the last stage at 870°C under oxidizing atmosphere extended by 120 seconds to allow all the elemental carbon to burn out. This protocol leads to a Limit of Quantification of 2 µg/filter, with a linearity range up to 500 µg/filter.

In this study, four punches were analyzed per filter, the maximum relative standard deviation measured between the punches for a given filter was found to be ~ 7 %.

2.4. Quantification of Titanium mass concentration by chemical analysis
PVC membranes were mineralized in TFM tubes with 3 ml of hydrofluoric acid and 2 ml of nitric acid. In order to take into account wall deposits, CFC were rinsed using 5 ml of diluted nitric acid (2%) and put in an ultrasonic bath for 20 minutes. This solution was then transferred to TFM tubes. Samples were put in a single chamber reaction microwave oven (Ultrawave, Milestone®) and were heated to 260°C for 10 min and the temperature maintained at 260°C for 20 min, before cooling. Once digested, the samples were diluted to 15 ml and analyzed, after matrix matching, by inductively coupled plasma optical emission spectrometry (ICP-OES, Agilent® 5110) in axial mode, with five replicates, and at three different wavelengths (308, 336 and 368 nm). Limit of Quantification was determined to 133ng/membrane.

3. Results
3.1. Overview of all data
The overall data of concentration stemming from SMPS calculations, chemical analysis quantification, gravimetric measurement and those from averaged AM520 raw data are synthetized in Figure 3.

As shown in Figure 3, the mass concentrations reported by the different approaches cover a range from 4.0 to 983 µg/m³. In addition, 7 samples were below the LOQ determined for gravimetric analysis of Gravi-Sert capsules (87 µg as determined according to ISO 15767:2009 [30]) and 2 were even below the associated limit of detection (LOD). On the contrary, all samples analyzed by chemical analysis contained a mass greater than the corresponding LOQ indicated in the previous sections. It is worth noting that for Ti aerosols, only 7 test aerosols out of the 9 corresponding experimental conditions investigated were sampled for chemical and gravimetric analysis.

Figure 3 highlights a good correlation between SMPS-based mass concentration (orange bars) and chemical analysis (green bars), and, to a lesser extent, with gravimetric analysis (purple bars).
correlation between SMPS and photometer (yellow bars) mass concentrations seems satisfying for carbon aerosols, it is worth noting that it is not the case for titanium aerosols.

Figure 3. Overview of parallel mass concentrations from SMPS data, carbon or titanium quantifications, capsule gravimetric measurements and AM520 data.

3.2. SMPS-based mass concentration vs chemical analysis

Figure 4 presents the SMPS-based mass concentrations against those obtained from chemical analysis.

Figure 4. Comparison of the mass concentrations between SMPS and chemical analysis.
Results presented in Figure 4 highlight a satisfactory correlation ($R^2 > 0.9$) between SMPS-based and chemical analysis, with an average relative discrepancies of about 15%. No effect of either the material or the modal number diameter of the aerosol has been demonstrated.

3.3. SMPS-based mass concentration vs gravimetric analysis

Figure 5 presents the SMPS-based mass concentrations against those obtained from gravimetric analysis.

![Figure 5: Comparison of the mass concentrations between SMPS and gravimetric analysis.](image)

Results presented in Figure 5 highlight satisfactory correlations ($R^2 \sim 0.9$) between SMPS-based and gravimetric analysis, with relative discrepancies lower than ~25%. No effect of either the material or the modal number diameter of the aerosol can be clearly demonstrated.

Nonetheless, most of the mass concentrations calculated from gravimetric analysis might not be significant since most of the masses were lower than the LOQ of the collection media used. Therefore, these data are to be considered carefully.

3.4. SMPS-based mass concentration vs AM520

Figure 6 presents the SMPS-based mass concentrations against those obtained from the photometer.

Figure 6 underlines an effect of particle composition in the correlation of SMPS-based mass concentrations with AM520 ones. A strong correlation ($R^2 = 0.98$) and close values (slope = 0.91) are observed between SMPS-based and AM520 photometer mass concentration when total carbon (elementary + organic) is considered, with reasonable biases up to 30%. On the other hand, AM520 underestimates the titanium mass concentrations obtained by SMPS calculation by a factor close to 5.8.
Figure 6. Comparison of the mass concentrations between SMPS and photometer.

3.5. AM520 data vs chemical analysis
As shown in Figure 7, the correlation between raw AM520 mass concentrations and total carbon concentrations measured by thermo-optical quantification presents a slope of $\sim 0.84$. A slope of $\sim 0.19$ characterizes the relationship between raw AM520 mass concentrations and titanium concentrations measured by ICP-OES.

Figure 7. Comparison of mass concentrations measured with AM520 and chemical analysis (thermo-optical quantification for carbon, ICP-OES quantification for titanium).
4. Discussion
The results presented above in Figures 4 to 7 demonstrate high correlations (> 0.87) between the different approaches, with slopes ranging from ~ 0.2 to ~ 5.8. To provide elements for explaining the discrepancies observed, the sources of uncertainty inherent to each of the techniques involved are discussed in this section.

4.1. Sources of uncertainty for the SMPS calculation approach
According to Equation 1, SMPS-based mass concentrations are associated with uncertainties that can be due to either the measurement of the number size distribution itself or the effective density law used to convert it into mass size distribution.

Measurement uncertainty associated with SMPS measurements has been reviewed recently [31], highlighting different sources of bias. Among them, the duration of a scan [32], electrostatic effects within the DMA [33], calibration and stability of operating flowrates within the DMA [34] constitute technical factors that might affect the number size distributions reported. Besides, physical parameters such as the difference in charging efficiency for agglomerates and aggregates [35] also lead to an increased uncertainty on SMPS raw data.

Second, the effective density law considered for determining the corresponding mass size distribution to be integrated is a parameter whose measurement involves specific selection devices, which can also be source of uncertainty. In this study, effective densities obtained from DMA-APM measurements were considered. Such data were found to be reliable for PSL particles between 40 and 800 nm [36-38]. Nevertheless, deviations up to ~ 10% were reported for black carbon agglomerates in the range 100–500 nm [15, 39, 40], mainly due to the presence of multiply-charged particles.

4.2. Sources of uncertainty for the thermo-optical carbon quantification
In our study, elementary carbon and organic carbon were measured using a thermo-optical analyzer and total carbon was considered in the results. Though pure graphite electrodes were used, the fraction of organic carbon was higher than 50% in all the samples what might be due to the degradation of the polyamide chamber of the generator [41].

4.3. Sources of uncertainty for the ICP-OES titanium quantification
Only titanium was quantified in the closed-face cassette samples but it cannot be excluded that Ti electrodes used in the spark-discharge generator also contain other compounds that were not analyzed and not taken into account in the determination of the mass concentration. To address this issue, the purity of the Ti electrodes is currently being verified.

A second source of uncertainty is related to the state of oxidation of titanium in our samples. According to the literature, which reports fast oxidation kinetics of Ti nanoparticles [42], we assumed that all the particles collected were 100% TiO₂. Again, this assumption may have led to an unknown bias in the calculation of the mass concentrations of titanium.

4.4. Sources of uncertainty for the gravimetric mass concentration
As previously mentioned, 9 gravimetric Gravi-Sert samples out of 12 were below the LOQ of 87 µg, as determined in our laboratory according to ISO 15767 [30]. Therefore, it is legitimate to interpret with caution the data resulting from gravimetric analysis because they could be associated with significant uncertainties.

4.5. Sources of uncertainty for the AM520 mass concentration
Each light scattering photometric mass measurement instrument is calibrated to a specific test aerosol. The SidePak™ AM520 is factory calibrated to the A1 Test Dust / ARD1 Arizona Road Dust. It is the finest standard dust of ISO 12103-1; it mainly contains silicon (69 to 77 %) but also a mix of different
mineral and metallic oxides [43]. Any aerosol other than this standard one will lead to a different photometric response, i.e. flux of scattered light per unit mass concentration of aerosol. As an example, photometric instrument response may differ from gravimetric mass measurement when the physical and light scattering properties of the sampled aerosol differ from the calibration aerosol, including refractive index, shape, density and size distribution [44, 45]. Therefore, custom calibration factors (CF) are needed for different aerosols.

Our experimental results (Figure 7) show that the custom calibration factor for the carbon test aerosol would be close to 0.8 while that for the titanium test aerosol would be close to 0.2. Even if the main objective of this article is not to discuss in depth the results measured by a photometer such as the AM520, one can notice in Table 1 the differences in properties between ARD1, carbon and titanium particles.

|                        | ARD1  | Black Carbon | Organic Carbon | Titanium |
|------------------------|-------|--------------|----------------|----------|
| density (g/cm³)        | 2.65  | ~ 2.20*      | ~ 4.50         |          |
| refractive index (λ ~ 650 nm) | 1.54  | 2.0          | 1.47           | ~ 2.8    |
| extinction coefficient (λ ~ 650 nm) | 0.03  | ~ 0.60       | 0.00           | 0.00     |
| references             | [46, 47] | [47, 48]     | 49, 50         |

*between ~ 1.3 and ~ 2.2 depending on the organic carbon content

Without going into the details of Lorenz-Mie calculations, O'Shaughnessy et al. [51] proposed to consider particle density, refractive index and size distribution as the most important physical aerosol parameters, that may influence the correction factor of a photometer in comparison to a given calibration dust. Several complementary phenomena will then influence the response of a photometer and lead to bias in the determination of the photometric mass concentration:

- Density of the measured particles is not taken into account in the optical photometer response. Therefore, it is commonly accepted that the mass concentration can be roughly corrected for the difference between the densities of the actual measured dust relative to that of the calibration dust, by simply applying the ratio of the two densities to the measurement;
- Changes in refractive index or extinction coefficient do not produce a linear change in photometer response. So, a compensation based on a straight ratio cannot be simply applied in order to try to determine a predictable correction factor that relates photometer concentration to true gravimetric mass measurements of an aerosol;
- Number size distribution of the actual aerosol to be measured is also a key parameter for two reasons: the particle diameters involved and the number concentration will both influence the photometer response. Photometers underestimate the particle concentration for both very small and very large particles. The ratio of the optical response of a photometer to particle mass concentration is only approximately constant for particles with sizes comparable to the wavelength, reflecting an approximately linear relationship between the two. So, in the case of the AM520, the response curve follows a bell-shaped curve that demonstrates a steep increase with increase in diameter below ~ 0.65 µm, followed by a steep decrease for diameters greater than ~ 0.65 µm (the approximation of 0.65 µm depends on particles properties and the wavelength of the monochromatic laser light). The number concentration will also influence the photometer response as the flux of light collected by the photodetector in presence of the entire distribution of particles in the sensing volume will be considered. The low intensity of light scattered by very small particle diameters, for example, may be counterbalanced by the high number concentrations of these particles.

Last but not least, both the carbon and titanium particles produced by the spark-discharge generator are not spherical or even isometric particles but fractal-like agglomerates [21, 27]. For this specific structure, the Rayleigh-Debye-Gans theory for fractal aggregates [52-54] would be more suitable than
the Lorenz-Mie theory, insofar as it accounts for primary particle size, number of primary particles constituting the agglomerates, fractal dimension, etc. The state of agglomeration of the nanoparticles could also have influenced the response of the photometer.

It remains difficult within the scope of our work to determine the exact contributions of the numerous physical and optical parameters (number size distributions and concentrations in Figure 2; densities and refractive indexes in Table 1; primary particle size, state of agglomeration, fractal dimension, etc.) that will explain that the AM520 response was satisfactory for carbon aerosol concentrations (~0.8) and underestimated titanium aerosol concentrations (~0.2).

5. Conclusion
To investigate the capability of a Scanning Mobility Particle Sizer (SMPS) to provide accurate data relative to the mass concentration, two series of test nanoaerosols were produced using a spark-discharge generator equipped with carbon or titanium electrodes. In the range of mass concentrations from ~20 to ~1000 µg/m³, different approaches were compared, including gravimetric analysis, chemical analysis, and direct-reading measurement by means of a photometer.

The experimental results presented in this paper exhibit that SMPS can be used to determine the mass concentration of airborne nanoparticles, provided that their effective density ρ_eff(d_m) has been preliminarily documented, by means of e.g. DMA-APM data. This approach could be used for other mobility spectrometers, such as FMPS/EEPS. In case particle effective density ρ_eff(d_a) is obtained by means of e.g. AAC-APM, this methodology can also be applied to aerodynamic spectrometers such as ELPI/ELPI+

Indeed, high correlations (R^2 ~ 0.9) between the different approaches were shown, in particular between SMPS-based mass concentrations and those obtained from chemical or gravimetric analysis. In addition, larger discrepancies were highlighted when SMPS-based mass concentrations were compared to photometer data. Therefore, sources of uncertainty were listed for each of the techniques involved to explain the biases observed. From a metrological point of view, it could be of interest to evaluate the contribution of each of the bias sources to the different data.

This preliminary laboratory work is part of a larger project aimed at studying the performances of several optical direct-reading instruments regarding mass concentration of various aerosols, in both laboratory and workplaces:

- Photometers: TSI DustTrak 8533 & 8534, TSI AM 510 & 520, Casella MicroDust Pro, Nephelometer Sensidyne, Respicon II, Thermo Scientific DataRAM pDR-1500, SKC Split 2, etc.
- Optical Particle Counters: TSI OPS 3330, Grimm 11D, Palas Fidas Frog, LightHouse 3016, etc.

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