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Traceable $PM_{2.5}$ and PM_{10} Calibration of Low-Cost Sensors with Ambient-like Aerosols Generated in the Laboratory

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Abstract: This work builds upon previous efforts at calibrating PM (particulate matter) monitors with ambient-like aerosols produced in the laboratory under well-controlled environmental conditions at the facility known as PALMA (Production of Ambient-like Model Aerosols). In this study, the sampling system of PALMA was equipped with commercial $PM_{2.5}$ and PM_{10} impactors, designed according to the EN 12341:2014 standard, to select different aerosol size fractions for reference gravimetric measurements. Moreover, a metallic frame was mounted around the PM impactor to accommodate up to eight low-cost PM sensors. This sampling unit was placed at the bottom of the 2-meter-long aerosol homogenizer, right above the filter holder for the reference gravimetric measurements. As proof of principle, we used the upgraded PALMA facility to calibrate the new AirVisual Outdoor (IQAir, Goldach, Switzerland) and the SDS011 (InovaFitness, Jinan, China) low-cost PM sensors in a traceable manner against the reference gravimetric method according to the EN 12341 standard. This is the first time that $PM_{2.5}$ and PM_{10} calibrations of low-cost sensors have been successfully carried out with complex ambient-like aerosols consisting of soot, inorganic species, secondary organic matter, and dust particles under controlled temperature and relative humidity.

Keywords: air quality monitoring; calibration; low-cost; optical sensor; particulate matter (PM); ambient-like aerosols; EN 12341 standard

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1. Introduction

High levels of particulate matter (PM) in ambient air are linked to morbidity as well as mortality [1,2]. More precisely, exposure to polluted air is associated with coughing, wheezing, and shortness of breath among children [2], and a recently published study calculated that up to 166,000 premature deaths could be prevented if $PM_{2.5}$ levels were to decrease to the lowest measured concentrations in Europe, i.e., 3.7 $\mu g/m^3$ [1]. Experiments with PM analyzers mounted on cars have shown that spatial concentration differences in cities can be substantial; thus, personal exposure can vary significantly, and the spatial resolution provided by government-operated air quality stations can be considered insufficient [3]. It is no surprise that cities seek to increase spatial resolution in an affordable manner, which has led to an increasing demand for low-cost sensors for air quality monitoring in recent years [4–6].

Sensitivity to composition and particle size of the aerosol, influence of relative humidity (RH), low accuracy, poor repeatability, drifts due to aging, and high unit-to-unit variability are, however, limitations of (some) low-cost optical sensors [7–11]. In theory, each sensor must be regularly calibrated so that users can trust the sensor data. Furthermore, such calibrations aim to compensate for some of the interfering effects—e.g., artefacts due to RH—but can lead to spatial or temporal relocation issues (i.e., a sudden increase in measurement uncertainty) if performed in the field [12–15]. The question arises to what extent governments can rely on such data to make informed policy decisions. Thus, main-

taining so-called "digital trust" with low-cost products during the operation of wireless sensor networks in smart cities remains a challenge [16–19].

As aerosols vary across locations and seasons, examining low-cost PM sensors in laboratory facilities is essential for benchmarking them under different standardized environments, yielding reliable performance assessments independent of the location and time of the year. Recently, a setup known as PALMA (Production of Ambient-like Model Aerosols) was developed for the production of ambient-like aerosols (i.e., mixtures of inorganic salt, mineral dust, soot particles, and secondary organic matter) under controlled environmental conditions, and was successfully applied to the calibration of commercial PM monitors against the gravimetric reference method [20]. However, the setup in its original form was limited to the calibration of total PM mass concentration.

In this work, we report on a further development of the PALMA setup. The sampling system of the gravimetric reference method was equipped with impactors as described in the EN 12341:2014 standard in order to determine $PM_{2.5}$ and PM_{10} mass concentrations, which are more relevant for legislation and air quality assessment. Moreover, the method was scaled up so that a batch of low-cost PM sensors could be calibrated simultaneously, thereby also accounting for unit-to-unit variability. Compared to other setups reported in the literature (see [21] and references therein), this is the first laboratory-based facility that enables the calibration of PM sensors with multicomponent ambient-like aerosols instead of simple model aerosols, such as sodium chloride, sucrose, polystyrene spheres, or dry dust particles. Indeed, ambient PM does not refer to a single pollutant, but to a highly variable mixture of combustion particles, salts, mineral dust, organic substances, and other materials [22,23]. PM sensors based on light scattering are sensitive to particle size, density, and refractive index distribution as well as hygroscopic growth, which can lead to considerable measurement artefacts [24,25]. Thus, complex model aerosols with realistic size distribution and chemical composition are essential for reliable calibration.

As a proof of concept, we present here the successful calibration of the new AirVisual Outdoor sensor (IQAir, Switzerland) and the SDS011 (InovaFitness, China) sensor. We believe that these advances in the calibration of low-cost PM sensors can make a valuable contribution towards the standardization and SI-traceable calibration of low-cost PM sensors.

2. Materials and Methods

2.1. Experimental Setup

Part of the experimental facility has been described in a previous publication [20]. Briefly, the setup consists of multiple aerosol generators, a 2.1-meter-long flow tube homogenizer placed vertically, a set of custom-made isokinetic sampling probes, and a system for temperature and humidity control. A graphical illustration of the flow tube homogenizer is shown in Figure 1a. Soot, aged soot, ammonium nitrate, ammonium sulfate, and dust particles were aerosolized (see Section 2.2 for a detailed description of the generators) and mixed in the homogenizer by three turbulent air jets. The inorganic salt and dust particles passed through two separate radioactive ^{85}Kr sources (TSI Inc., Shoreview, MN, USA) to reduce the charges of the particles and render the aerosols (as an ensemble) neutral before entering the homogenizer. Experiments with soot and aged soot aerosols have revealed that these are already neutral and do not require any further treatment. Salt particles were dried by passing them through a bed of silica gel beads (2–5 mm, Dry & Safe GmbH, Oensingen, Switzerland). Tests of hygroscopic growth of pure ammonium sulfate particles showed that particles were dried below the efflorescence point before entering the homogenizer.

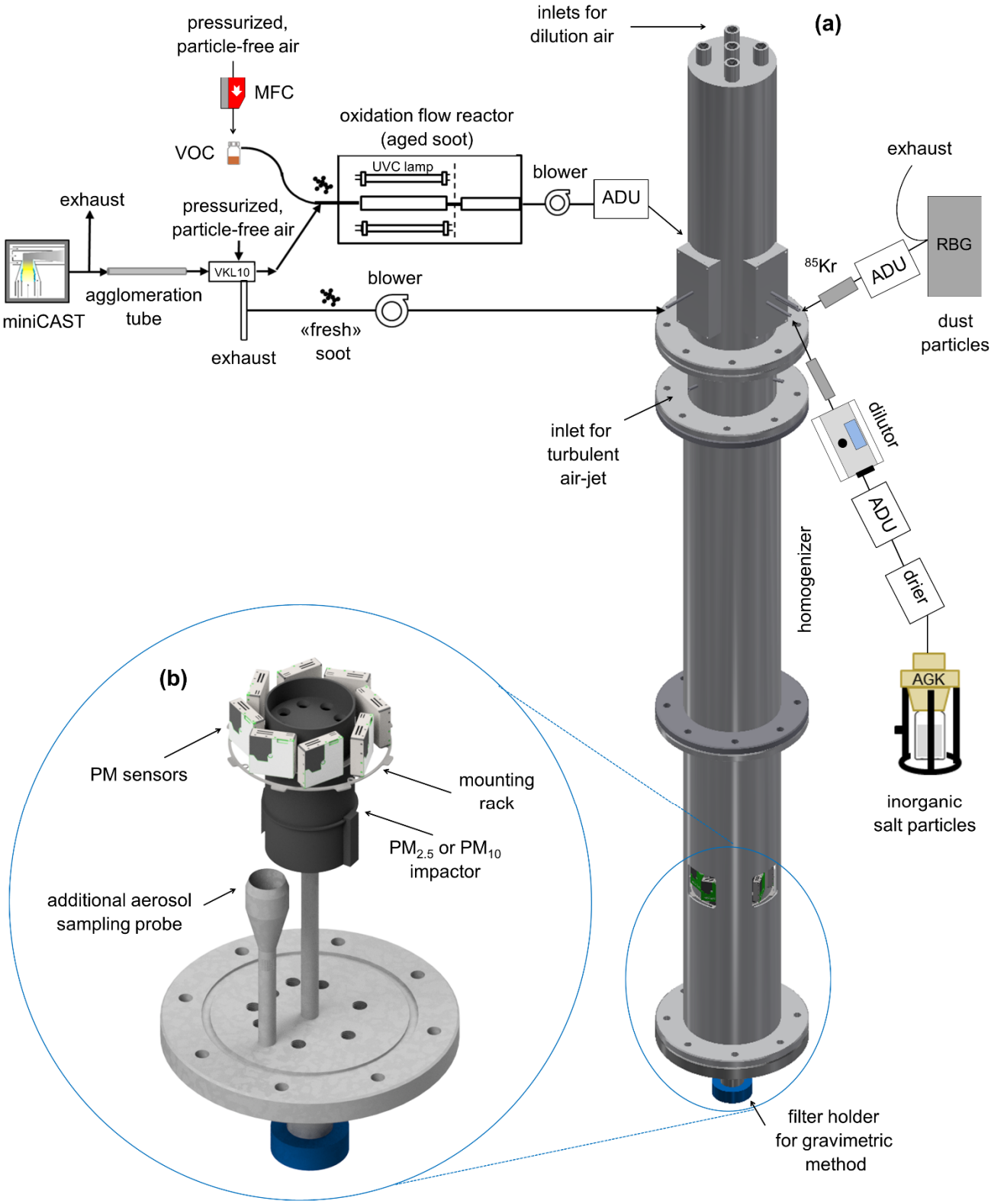


Figure 1. (**a**) CAD illustration of the homogenizer (see [20] for more information). (**b**) Enlarged view of the aerosol sampling system. An impactor selects $PM_{2.5}$ or PM_{10} size fractions for the gravimetric reference method. The low-cost PM sensors are mounted on a rack surrounding the impactor. Aerosols can be also sampled through additional isokinetic sampling probes for further aerosol characterization. For simplicity, only one such probe is displayed. ADU: adjustable dilution unit (custom-made); VOC: volatile organic compound.

In this study, we designed an entirely new sampling system that enables $PM_{2.5}$ and PM_{10} calibration of multiple low-cost PM sensors simultaneously based on the selection of different particle size fractions by standardized impactors. A computer-aided design (CAD, Inventor Professional 2019, Autodesk, San Rafael, CA, USA) of the sampling system is displayed in Figure 1b. The central component is the $PM_{2.5}$ or PM_{10} impactor (Digitel, Zürich, Switzerland). A metallic rack, which can accommodate several PM sensors (the exact number depends on the sensor's dimensions), is placed around the impactor at the height of the impactor's aerosol inlet. The sensors are placed upright, so that the aerosol inlet is on the top, right next to the impactor. The impactor is connected to the filter holder of the gravimetric reference method through a straight metallic tube. Up to eight additional isokinetic sampling probes for the aerosol monitoring instruments—e.g., TEOM (tapered element oscillating microbalance) and SMPS (scanning mobility particle sizer)—and/or PM monitors under test can be attached to the bottom metallic plate of the homogenizer, depending on the experimental needs. The aerosol spatial homogeneity at the sampling region is within 2.6% (k = 2; 95% confidence interval) in particle number concentration [20].

2.2. Aerosol Generation and Characterization

The primary aerosols were generated as described below:

- Inorganic salt: aqueous solutions of ammonium nitrate (>99%, Acros Organics, Thermo Fisher Scientific, Geel, Belgium) and ammonium sulfate (>99.5%, Acros Organics, Thermo Fisher Scientific, Geel, Belgium) were nebulized with an AGK 2000 atomizer (PALAS, Karlsruhe, Germany). The aerosol had a GMD_{mob} (geometric mean mobility diameter, number-based) of 160 nm and a GSD_{mob} (geometric standard deviation) of 1.9. The mode of the mass-based aerodynamic size distribution was expected to lie above 300 nm [20];
- Dust particles (ISO A3 test dust, 98% SiO_2, DMT, Longmont, CO, USA) were dispersed with an RBG 1000 rotating brush generator (PALAS, Germany). The aerosol had a GMD_{mob} of 300 nm and GSD_{mob} of 1.7. The mode of the mass-based aerodynamic size distribution was expected to be in the lower micrometer range [20];
- Soot by propane combustion with a miniCAST 6204 burner (Jing Ltd., Zollikofen, Switzerland), with a GMD_{mob} of 120 nm and a GSD_{mob} of 1.6 (see [26,27] and references therein);
- Photochemically aged soot by ozonolysis of α-pinene (>97%, Sigma-Aldrich, Buchs, Switzerland) in a prototype micro smog chamber ([28,29]); the size distribution had a GMD_{mob} of 180 nm and a GMD_{mob} of 1.2.

The mobility measurements were performed with an SMPS (SMPS+C with L-DMA and CPC model 5.403, Grimm GmbH, Hochdorf, Germany), which was traceably calibrated. The SMPS mobility size range extended to about 1 µm. If needed, the aerosols were diluted before injection in the homogenizer by using custom-made adjustable dilution units (ADU) or commercial dilutors (DDS 562, Topas GmbH, Dresden, Germany).

Reference gravimetric measurements were carried out according to the EN 12341:2014 standard, i.e., particulate matter was passed through a $PM_{2.5}$ or PM_{10} impactor and then sampled on filters and weighed by means of a balance. More specifically, the model aerosols were drawn through 47 mm PTFE-coated glass fiber filters (Measurement Technology Laboratories, Bloomington, MN, USA) placed in a metallic filter holder (C806 standard aerosol filter holder, Merck Millipore, Darmstadt, Germany). The aerosol flow was controlled with a needle valve and measured with a calibrated mass flow meter (Natec Sensors GmbH, Garching bei München, Germany) connected to an aerosol pump (VTE8, Gardner Denver Thomas GmbH, Memmingen, Germany) in such a way that the volumetric flow corresponded to 2.3 m^3/h at ambient conditions. The flow measurement was traceable to national standards.

The connecting tube between the isokinetic sampling and the filter holder was made of inert, electrically conducting rubber material, and was kept as short as possible (≈5 cm) without bends to minimize deposition losses of particulate matter via kinetic processes,

as well as losses due to thermal, chemical, or electrostatic processes. Before and after sampling, the filters were conditioned for 48 h at 20 °C and 50% relative humidity, and then weighed with a calibrated balance (Model UMT5, Mettler Toledo, Albstadt, Germany), which was traceable to national standards. Temperature, pressure, and relative humidity inside the sampling region were also recorded (ALMEMO FHAD 46x with sinter filter SK6, Ahlborn Mess- und Regelungstechnik GmbH, Holzkirchen, Germany).

For quality assurance, the mass concentration of the model aerosols was monitored with a TEOM 1405 (Thermo Scientific, Waltham, MA, USA) operated at a temperature of 30 °C to minimize losses of semivolatile material. The TEOM used a custom-made isokinetic sampling probe for sampling (see Figure 1b). Moreover, the optical size distribution of the model aerosols was measured using a Fidas Frog monitor (Palas, Germany) or an 11-D dust monitor (Grimm, Germany).

The composition of the two model aerosols is displayed in Figure 2. The percentage (%) PM mass fraction of each chemical component was determined gravimetrically by switching off all other aerosol generators and simultaneously recording the particle number density with a calibrated CPC (condensation particle counter). This correlation of mass and particle number density was then used to set the concentration of each aerosol for the calibration measurements. The mass of elemental carbon (EC) and organic carbon (OC) was measured by EC/OC analysis (Lab OC-EC Aerosol Analyzer, Model 5, Sunset Laboratories Inc., USA). The mass of organic matter (OM) was then calculated by multiplying the OC mass by 1.8 [30]. For the EC/OC analysis, aerosols were sampled on quartz filters (Advantec, Japan, QR-100, 47 mm, preconditioned by heating at 500 °C for 1.5 h). The relative expanded uncertainty in the determination of the OC and OM mass concentrations in the model aerosols was estimated to be 12–16%. In the case of inorganic salt and dust particles, the relative expanded uncertainty in the determination of the mass concentration was 10%.

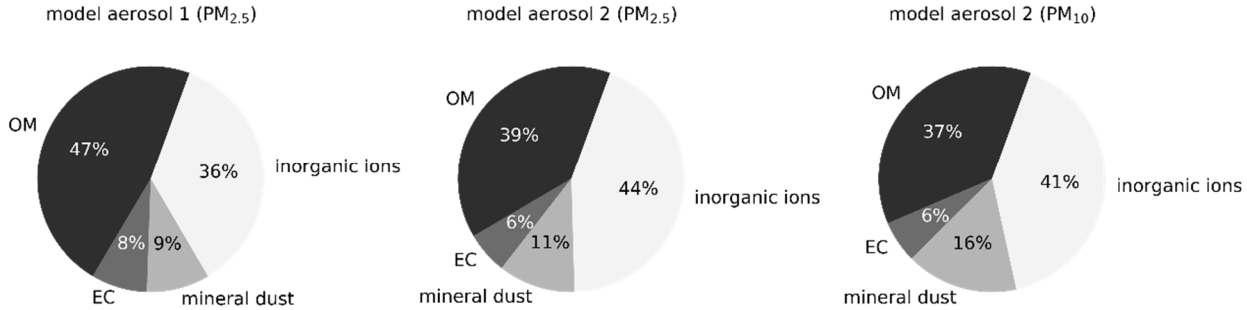


Figure 2. PM composition (%) of the two model aerosols.

3. Results and Discussion

3.1. $PM_{2.5}$ Calibration

An AirVisual Outdoor sensor unit (comprising two PM sensors) was mounted on the rack surrounding the PM impactor (see Figure 1b) throughout all experiments with model aerosols 1 and 2 (see Figure 2). Due to limited availability, the SDS011 PM sensor (InovaFitness, China) was only mounted during the experiments with model aerosol 2. The AirVisual Outdoor is a photometer recording the light scattered from all particles present in the measurement cell, whereas the SDS011 is based on light scattering from individual particles. Note that both devices report $PM_{2.5}$ and PM_{10} measurements as 1 min averages.

The AirVisual Outdoor sensor was calibrated against the gravimetric reference method with the two different model aerosols presented in Section 2.2. As an example, a $PM_{2.5}$ mass concentration measurement is shown in Figure 3. The green line corresponds to the mass concentration according to the gravimetric reference method, while the shaded green area designates the expanded uncertainty (k = 2; 95% confidence interval) thereof. A very good agreement between the AirVisual Outdoor sensor (purple dots) and the gravimetric reference method was observed. The sensor reported an average $PM_{2.5}$ mass

concentration C_{DUT} of 83.4 µg/m^3, thus underestimating the $PM_{2.5}$ mass concentration by ~6% compared to the reference measurement ($C_{ref.}$ = 88.5 µg/m^3). The calibration was repeated at various mass concentrations, as shown in Figure 4a and summarized in Table 1. The AirVisual Outdoor sensor performed very well with both model aerosols, in most cases exhibiting a measurement efficiency ($E_{DUT} = C_{DUT}/C_{ref}$) of 0.9–1.1. The only exception was the measurement of model aerosol 1 at high relative humidity, where the measurement efficiency was around 1.3, presumably due to hygroscopic growth of the ammonium nitrate and ammonium sulfate particles.

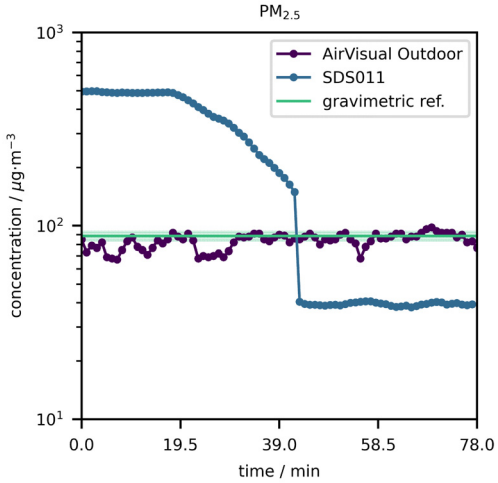


Figure 3. $PM_{2.5}$ mass concentrations reported by the AirVisual Outdoor and SDS011 as a function of time for model aerosol 2. The green line refers to the average mass concentration over the whole measurement period according to the gravimetric reference method, while the shaded green area designates the expanded uncertainty (k = 2; 95% confidence interval) thereof.

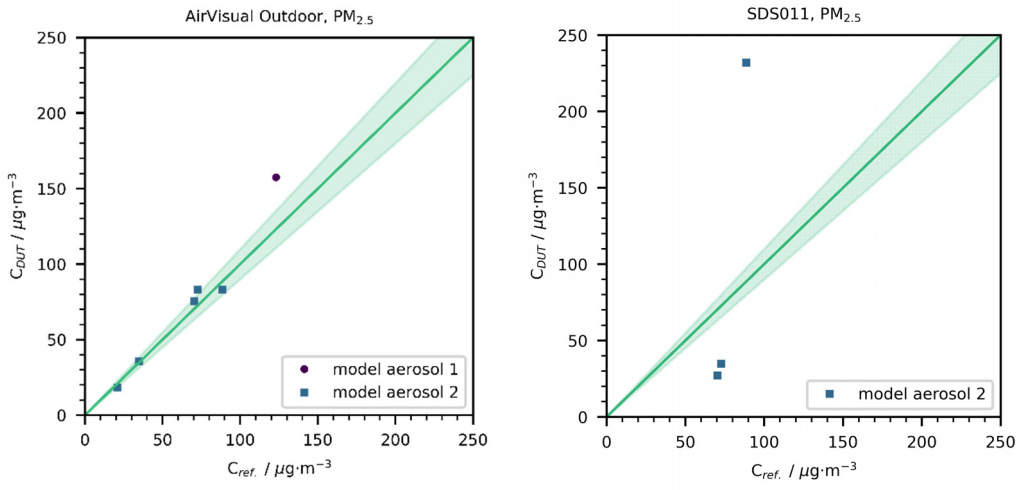


Figure 4. $PM_{2.5}$ mass concentrations reported by (**a**) the AirVisual Outdoor and (**b**) the SDS011 versus the gravimetric reference mass concentration. The green line denotes a perfect agreement, while the shaded green area designates a maximum 10% relative expanded uncertainty (k = 2; 95% confidence interval) of the gravimetric reference method.

Table 1. $PM_{2.5}$ mass concentrations reported by the DUT (device under test) and the reference gravimetric method—C_{DUT} and $C_{ref.}$, respectively—and measurement efficiency of the DUT, E_{DUT}. U denotes expanded uncertainties (k = 2; 95% confidence interval); s is the standard deviation of the mean (i.e., $\frac{\sigma}{\sqrt{n}}$, with n being the number of measurements).

Model Aerosol	T/°C	RH/%	DUT	$C_{DUT} \pm 2\,s$/µg·m^{-3}	$C_{ref.} \pm U$/µg·m^{-3}	$E_{DUT} \pm U$
1	8	82	AirVisual Outdoor	157 ± 7	123 ± 6	1.28 ± 0.09

Table 1. *Cont.*

Model Aerosol	T/°C	RH/%	DUT	$C_{DUT} \pm 2$ s/µg·m^{-3}	$C_{ref.} \pm U$/µg·m^{-3}	$E_{DUT} \pm U$
2	21	50	AirVisual Outdoor	18.4 ± 3.0	20.9 ± 2.2	0.88 ± 0.17
	21	50		35.9 ± 4.2	36.6 ± 2.2	0.98 ± 0.13
	21	50		35.9 ± 4.2	34.6 ± 2.1	1.04 ± 0.14
	21	83		75.5 ± 2.2	70.2 ± 3.5	1.08 ± 0.06
	21	66		83.1 ± 1.4	72.6 ± 5.0	1.14 ± 0.08
	21	50		83.4 ± 1.8	88.5 ± 4.7	0.94 ± 0.06
	21	83	SDS-011	27.3 ± 4.0	70.2 ± 3.5	0.39 ± 0.06
	21	66		34.9 ± 5.4	72.6 ± 5.0	0.48 ± 0.08
	21	50		232 ± 44	88.5 ± 4.7	2.62 ± 0.52

In contrast, the SDS011 sensor showed a rather erratic behavior, reporting a sudden decrease in the $PM_{2.5}$ mass concentration during measurement (Figure 3). The measurement efficiency ranged between 0.4 and 2.6 (Figure 4b, Table 1). Budde at al. compared 17 SDS011 sensors to a Palas PROMO 2000 reference in a laboratory setting using ambient air, and reported an average $PM_{2.5}$ measurement efficiency E_{DUT} of 0.66 (E_{DUT} ranging from 0.48 to 0.85) [31]. The measurement efficiency decreased even further to an average of 0.49 (0.41–0.63) with pure ammonium sulfate as the test aerosol. Liu et al. also reported underestimation of $PM_{2.5}$ mass concentration in their field campaign, particularly for higher pollution levels (E_{DUT} ranging from 0.7 to 0.9) [32].

3.2. PM_{10} Calibration

Figure 5 displays an example of a PM_{10} calibration of the low-cost sensors as a function of time. A summary of all PM_{10} calibrations in the mass concentration range 20–150 µg/m^3 is provided in Figure 6, and the data are listed in Table 2. A very good agreement between the AirVisual sensor and the gravimetric reference method was observed (E_{DUT} close to 1), while the SDS011 drastically underestimated the mass concentrations. For the PM_{10} readings of the SDS011, Budde et al., reported that particles larger than 5 µm were severely underestimated, resulting in an average E_{DUT} value of 0.65 [31], which is consistent with the trends reported here.

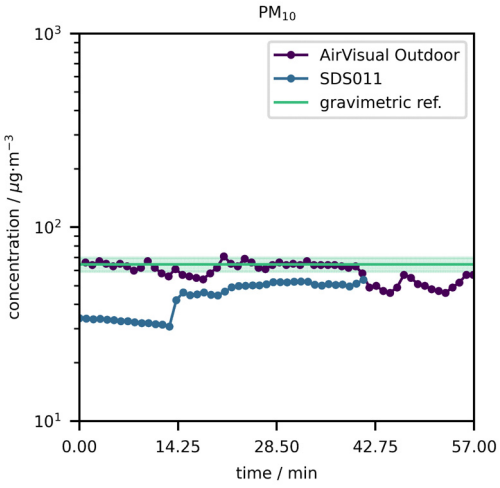
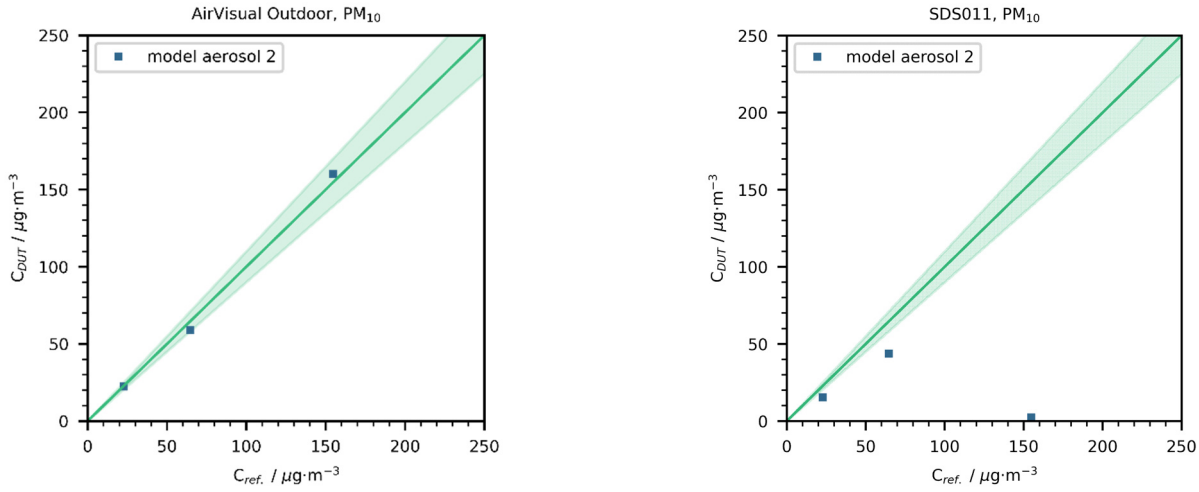


Figure 5. PM_{10} mass concentrations reported by the AirVisual Outdoor and SDS011 as a function of time (model aerosol 2). The green line refers to the average mass concentration over the whole measurement period according to the gravimetric reference method, while the shaded green area designates the expanded uncertainty (k = 2; 95% confidence interval) thereof.



(**a**) (**b**)

Figure 6. PM_{10} mass concentrations reported by (**a**) the AirVisual Outdoor and (**b**) the SDS011 versus the gravimetric reference mass concentration. The green line denotes a perfect agreement, while the shaded green area designates a maximum 10% relative expanded uncertainty (k = 2; 95% confidence interval) of the gravimetric reference method.

Table 2. PM_{10} mass concentrations reported by the DUT and the reference gravimetric method—C_{DUT} and $C_{ref.}$, respectively—and measurement efficiency of the DUT, E_{DUT}. U denotes expanded uncertainties (k = 2; 95% confidence interval); s is the standard deviation of the mean (i.e., $\frac{\sigma}{\sqrt{n}}$, with n being the number of measurements).

Model Aerosol	T/°C	RH/%	DUT	$C_{DUT} \pm 2\ s$/µg·m^{-3}	$C_{ref.} \pm U$/µg·m^{-3}	$E_{DUT} \pm U$
2	21	50	AirVisual Outdoor	22.6 ± 1.4	22.6 ± 1.8	1.00 ± 0.10
	21	50		59.3 ± 1.8	64.5 ± 5.0	0.92 ± 0.08
	21	50		160 ± 18	156 ± 10	1.03 ± 0.14
	21	50	SDS011	15.5 ± 2.0	22.6 ± 1.8	0.69 ± 0.10
	21	50		43.8 ± 2.2	64.5 ± 5.0	0.68 ± 0.07
	21	50		2.40 ± 0.20	154 ± 10	~0

Note that sensors measuring PM mass concentration based on light scattering are very sensitive to the chemical composition and size distribution of the test aerosol. Since the properties of ambient air vary by time and location, it is impossible to define a single test aerosol in the laboratory that would sufficiently simulate all real environmental scenarios. In principle, the generators described in Section 2.2 are versatile, and can easily be used to produce different kinds of inorganic species (e.g., sodium chloride) or mineral dust particles (Arizona test dust, dolomite dust, kaolinite, etc.) to further refine the composition and scattering properties of the test aerosols. However, for simulating remote aerosols, marine aerosols, or specific pollution events (such as wildfires), more dedicated experiments must be carried out, and additional generators (e.g., a stove) would be required. A rigorous calibration of low-cost sensors in the laboratory, akin to that described in this study, would increase the price of the sensors. A more pragmatic approach would be to provide sensor manufacturers, air quality monitoring stations, and other end users with a limited set of calibrated sensors, which could then serve as (mobile) "reference" set in the laboratory or field [33–35].

4. Conclusions

This manuscript describes a novel method for the traceable calibration of low-cost $PM_{2.5}$ and PM_{10} sensors with ambient-like aerosols generated in the laboratory. The setup consisted of multiple aerosol generators for producing soot, aged soot (i.e., coated

with secondary organic matter), inorganic species, and dust particles. The aerosols were mixed in an aerosol flow tube homogenizer and sampled through a custom-made system equipped with $PM_{2.5}$ and PM_{10} impactors for the gravimetric reference method. Up to eight low-cost sensors could be calibrated simultaneously. As a proof of concept, the AirVisual Outdoor and the SDS011 sensors were calibrated against the gravimetric method in the mass concentration range ~20–150 $\mu g/m^3$. The AirVisual Outdoor sensor agreed very well with the reference method, showing measurement efficiencies typically in the range of 0.9–1.1, whereas the SDS011 exhibited a rather erratic behavior, with large deviations from the reference method. We believe that the calibration procedure outlined in this study can contribute to the performance characterization and standardization of low-cost sensors for air quality control.

Author Contributions: S.H. performed the experiments. G.T. analyzed and visualized the data. G.T. and K.V. wrote the manuscript. K.A. and S.H. designed the new sampling system. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare that they have no conflict of interest.

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