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Observation of systematic deviations between Faraday cup aerosol electrometers for varying particle sizes and flow rates—results of the AEROMET FCAE workshop

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Abstract

Condensation particle counters (CPCs) are widely used for the measurement of aerosol particle number concentrations in the size range from approximately 3 nm to 3 µm. For an SI-traceable calibration of the size-dependent counting efficiency, which is advisable on a regular basis and required in several applications, Faraday cup aerosol electrometers (FCAEs) are considered to be a suitable SI-traceable reference. While the volumetric aerosol inlet flowrate and the electrical current measurement in FCAEs can be related to respective SI references, inter-comparison exercises for FCAEs are still performed on a regular basis to establish reliable uncertainty budgets and to further investigate the influences of designs and operational parameters on comparability. This is strongly demanded in the international community of metrological institutes and aerosol calibration facilities around the world, which provide CPC calibrations. In the present study, the performance of FCAEs was investigated, using Ag test aerosol particles with a 30 nm particle diameter by varying the inlet flowrates from 0.5 l min⁻¹ to 4 l min⁻¹. From our experimental results, significant deviations were observed in FCAE currents at sample flowrates smaller than 1.5 l min⁻¹. It is recommended that these discrepancies should be quantified before an FCAE is used for CPC calibration at low sample flowrates and small particle sizes in the sub-30 nm size range.

Keywords: Faraday cup aerosol electrometer, CPC calibration, inter-comparison

(Some figures may appear in colour only in the online journal)

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1. Introduction

Faraday cup aerosol electrometers (FCAE) are considered as the simplest form of electrical aerosol measurement instrument used in the field of aerosol science [1]. FCAE instruments measure the electrical current of charged aerosol particles in an airflow [2]. FCAEs have been widely used (a) for calibrating condensation particle counters (CPCs) [3–10], which are used to determine the particle number concentration (PNC) directly or as a detector in a mobility particle size spectrometer (MPSS), measuring the particle number size distribution (PNSD) [11], (b) for direct measurements of net electrical charges of charged aerosol particles, or (c) for measurements of atmospheric ions [12–14]. FCAEs have been frequently used as a primary standard of the PNC, since the measurement depends only on the electric current induced by electrically singly charged particles and the aerosol sample volumetric flow, which are both traceable to primary standards [2,15,16].

The major components of an FCAE instrument are a Faraday cup (FC) and an electrometer along with a flow meter or flow controller [2,15]. In the FCAE, an electrically conducting enclosure (of the same electric potential as the filter) acts as a shield to cover the sensing element, which consists of a conducting high-efficiency particulate filter (not necessarily conductive) [17]. The inner cup is connected to ground potential via a sensitive amplifier including a current-to-voltage converter, meaning an electrometer. An equal and opposite electric charge (also known as an image charge) passes through the electrometer after the collection of a charged particle in the filter, thus making the net charge on the FC itself zero. Therefore, the electrical current in the electrometer's ampere meter is directly proportional to the charge collection rate on the filter. FCAEs can be used to measure electrical currents down to the femtoampere (fA) range. FCAEs used for CPC calibration must have a stable zero baseline. According to the ISO 27891:2015 standard, the zero-corrected absolute arithmetic mean electric current must be less than 1 fA with a standard deviation below 0.5 fA, when no charged particles are present [17,18]. For measurements of the PNC by an FCAE, any losses of particles in the inlet should be avoided and must be minimal. In addition, the efficiency of the particle filter must be ideally 100%. However, some of the electrically charged particles that enter an FCAE inlet may not reach the filter and thus do not contribute to a current at the electrometer. The FCAE's detection efficiency, which would ideally be unity for an 'ideal' FCAE, accounts for the above two factors of underestimating the PNC. For particles above 5 nm and sample flowrates above 1 l min⁻¹, the detection efficiency of an FCAE is typically expected to be better than 99% [18]. One of the challenges regarding FCAE instruments is still the quantification of particle losses in the inlet, which may be influenced by different settings, having the potential to exceed 1%. If so, this would certainly have—besides electrical current measurement noise—a significant impact on the key characteristics of an FCAE, namely the current measurement uncertainty and the lower current detection limit [17]. When an FCAE is used as a primary reference for CPC calibration, the lower

limit of approximately several hundred (singly charged) particles per cm³ inhibits its use near to the CPC's typical lower concentration limit of 0.1 particles per cm³.

While some of the abovementioned sources of measurement uncertainties can be minimized using careful design, the quantification of uncertainties is usually done in comparison studies [2].

Until now, only a few inter-laboratory comparison exercises have been conducted as far as FCAEs are concerned. A comprehensive inter-comparison on PNC and PNSD measurements was performed for size ranges from 50 nm to 170 nm using combustion aerosol particles. The comparison, however, did not cover the sub-50 nm particle size range, which is relevant for PNC measurements using CPCs [19]. The first FCAE campaign compared one reference FCAE directly to other FCAE instruments. The primary purpose for this direct comparison was to show the accurate and within reasonable error consistent determination of the electrical current. This is seen to be a prerequisite for the realization of reliable PNC measurements with FCAEs (www.euramet.org). This comprehensive comparison revealed $\pm 5\%$ relative deviation of the PNC means among eight FCAEs for sub-20 nm particles with 1 l min⁻¹ sample flowrate [2]. Although uncertainties are expected to increase at smaller particle sizes and lower PNCs, the results provided experimental evidence that the requirements of the ISO 27891 standard can practically be met. For the measurements, performed with sub-20 nm particles, it remained unclear whether the observed increased deviations could be attributed to size-dependent losses inside the FCAEs ([2], CCQM K150/P189 2017).

In this study, we investigated the comparison of four FCAEs during a first workshop (11–15 March 2019) and two FCAEs during a second workshop (7–9 September 2020), which were conducted at the World Calibration Center for Aerosol Physics (WCCAP). During the first workshop, the particle size-dependent effect of diffusional losses in the FCAE inlets of different designs on electrical current measurements was investigated using monodisperse silver particles ≤ 30 nm. The second workshop aimed at the measurement of sample flow-dependent effects on electrical current measurements such as diffusional losses at varying flow rates (from 0.5 l min⁻¹ to 4 l min⁻¹) using monodisperse 30 nm silver particles. Both experiments can be considered as a first attempt to quantify the effects of diffusion losses in FCAEs at small particle sizes and low sample flows on the uncertainty budget.

2. Method

2.1. Experimental setup

The experimental setup and the measurement scheme were carefully designed, following in many details the WCCAP setup for CPC calibration [20]. The generation of monodisperse silver particles for the FCAE comparison exercise was done using a condensation particle generator [21]. Here, a tube furnace with three separately temperature-controlled heating zones (model FRH-3-70/750/1250, Linn HighTherm GmbH, Germany) was used for the generation of particles from molten

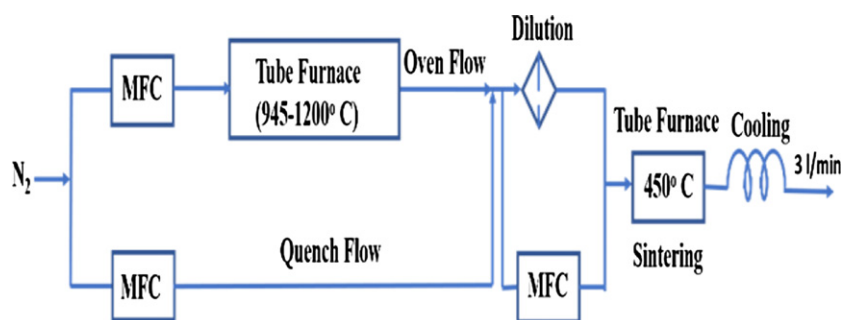


Figure 1. Schematic diagram of the WCCAP Ag aerosol generator. Here, MFC represents mass flow controller. The flow rate at the exit of the generator (after cooling) is 3 l min^{-1} . [10] Taylor & Francis Ltd. <http://tandfonline.com>.

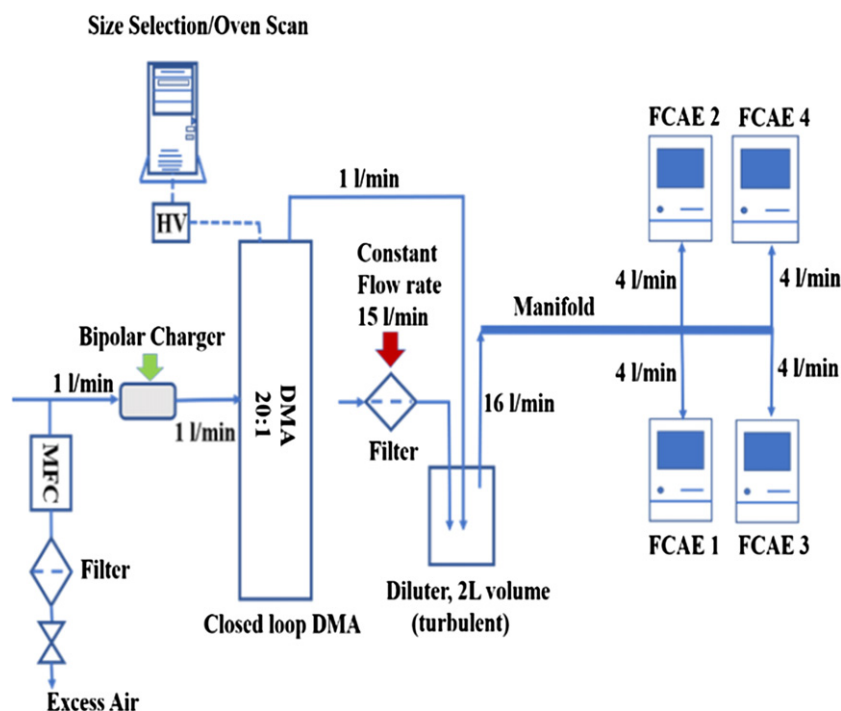


Figure 2. Size selection and inter-comparison setup for FCAEs at the WCCAP. Here, all four FCAEs were run at 4 l min^{-1} at variable sizes. The flow rate through manifold was 16 l min^{-1} . Dashed and solid lines represent electrical connections and tubes, respectively. [10] Taylor & Francis Ltd. <http://tandfonline.com>.

silver. The oven has a heated length of about 1000 mm. All three oven zones were set to similar temperatures. The center of a ceramic tube holds an inner diameter of 19 mm where the sample material for evaporation is located in a ceramic boat. The WCCAP silver aerosol particle generator (as shown in figure 1) features a quench flow to minimize and then stop the coagulation of primary particles after their generation.

Both the oven and quench flowrates are controlled by mass flow controllers (MKS model 1179AX14CS1BV, MKS Instruments Deutschland GmbH, Germany) to ensure a stable PNSD of the silver particles along with a better repeatability of calibration settings. The PNC adjustment is done via a dilution system. After dilution, the particle agglomerates are sintered at $450 \text{ }^\circ\text{C}$ for about 0.3 s to more spherical particles. The aerosol is then cooled down to room temperature using a cooling metal spiral of 1 m length. A custom-made 370 MBq Kr^{85} bipolar charge conditioner is used to generate a known bipolar charge distribution [22] on the freshly generated polydisperse

airborne Ag particles, which are then fed into the WCCAP custom-made Hauke-type DEMC [23] at an aerosol flowrate of 1 l min^{-1} and a sheath air flowrate of 20 l min^{-1} for particle size selection (figure 2), leading to a narrow monodisperse aerosol.

While selecting the monodisperse aerosol, the DEMC high voltage is continuously adjusted to the temperature and pressure in the DEMC. This adjusting was done using a custom-made LabView program. The whole process ensures the production of a quasi-monodisperse aerosol with high repeatability in particle size and quantity under varying ambient conditions. In order to select a monodisperse particle size fraction, the oven PNSD is determined first, using the DEMC as an MPSS. After this, the DEMC is adjusted to the respective voltage for the selection of the envisaged monodisperse aerosol fraction. The particle size is selected from the right side of the oven PNSD in order to avoid multiply charged particles. Downstream of the DEMC, filtered makeup air is added with

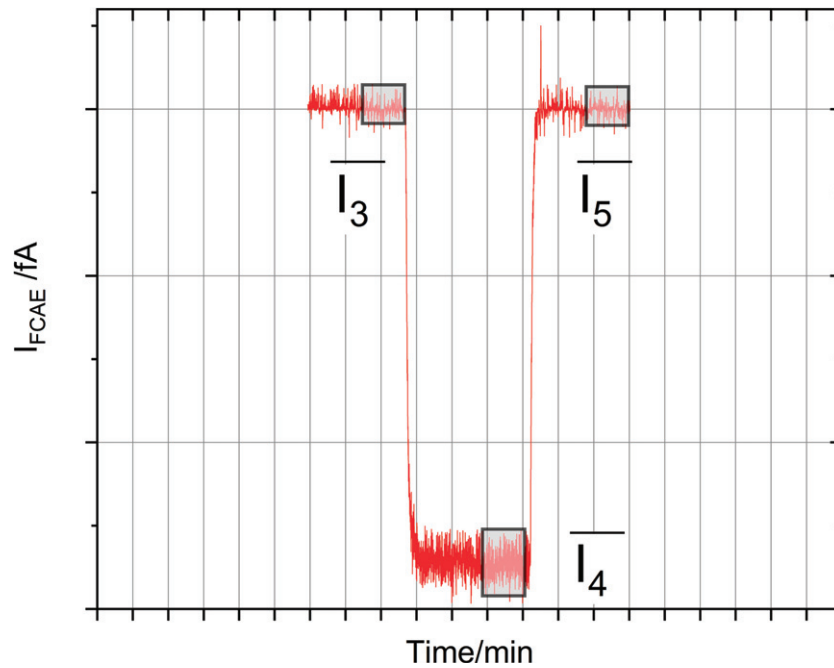


Figure 3. Scheme for the current measurements.

the help of a turbulent diluter and the aerosol is fed into a manifold, which allows parallel providing of four FCAEs under test with identical monodisperse test aerosols. Particle size selecting followed the DEMC setup and adjustment procedure as well as the diameter calculation procedure described in the ISO 15900:2009 standard for diameter calculations. Figures 2 and 4 provide overviews of the experimental setups for the workshops described below.

2.2. First workshop: particle size effects in FCAEs

Four FCAEs were selected for the comparison. Two technically identical instruments were manufactured by TSI (3068BTM, TSI, USA). These devices are labeled in the following as FCAE 1 and FCAE 2. FCAE 3 is a BAM inhouse construction, consisting of a Finnish designed FC with a commercial current amplifier and converter (DDPCA-300TM, FEMTO, Germany) and flow controller (elflowTM, Bronkhorst, Germany). FCAE 4 was manufactured by Palas (model CharmeTM, Palas GmbH, Germany).

One of the TSI FCAEs (FCAE 1) serves as the main WCCAP reference. It is calibrated annually by the National Metrology Institute of Germany [Physikalisch-Technische Bundesanstalt (PTB), Braunschweig] using an SI-traceable fA source. The performance of this instrument was further verified during an international FCAE inter-comparison workshop, where several NMIs participated [2]. FCAE 2 is calibrated against FCAE 1 every six months.

All four FCAEs were connected to the common aerosol source as shown in figure 2 through conductive silicon rubber tubes with 0.7 m length and 6 mm width, branching from a steel manifold with an inner diameter of 19.05 mm (3/4 inch). The setup provided the same PNC, gas pressure and gas temperature in each branch.

The FCAEs were operated at constant volumetric sample flowrates of 3.9 l min⁻¹. Before each measurement the flowrates were adjusted with the help of an external flowmeter (TSI 4146TM, TSI, USA), and then individually kept constant via the internal FCAE flow controllers. By this, possible systematic deviations in flowrate readings from the individual internal flow controllers and their effect on PNC calculations could be omitted.

Hence, in the ideal case of no design-related effects, all FCAEs under test should—for all particle sizes—have within reasonable uncertainty limits the same current readings.

The comparison was performed using monodisperse Ag particles of 30, 20, 10 and 5 nm mobility diameter. The setting of the experimental parameters and the measurements comprised the following steps for each of the particle sizes:

- Setting of the aerosol generator by tuning the differential mobility analyzer (DMA) voltage to the desired particle size, facultatively varying the oven temperature, and finally waiting for a stable PNC.
- Adjustment of the FCAE's inlet flowrates.
- With all flowrates adjusted to the same level and stabilized, the DEMC of the particle generator was set to zero voltage and the zero electrical currents—corresponding to particle-free carrier gas—were measured for approximately 5 min.
- The DMA voltage was reset for the transmission of the monodisperse test aerosol and the FCAEs measured the currents over approximately 5 min.
- Repetition of steps (c) and (d). As comparison parameters from each FCAE, the current means and standard deviations were taken using the formula

$$I_{\text{FCAE } x} = \bar{I}_4 - 0.5 \cdot (\bar{I}_5 + \bar{I}_3) \quad (1)$$

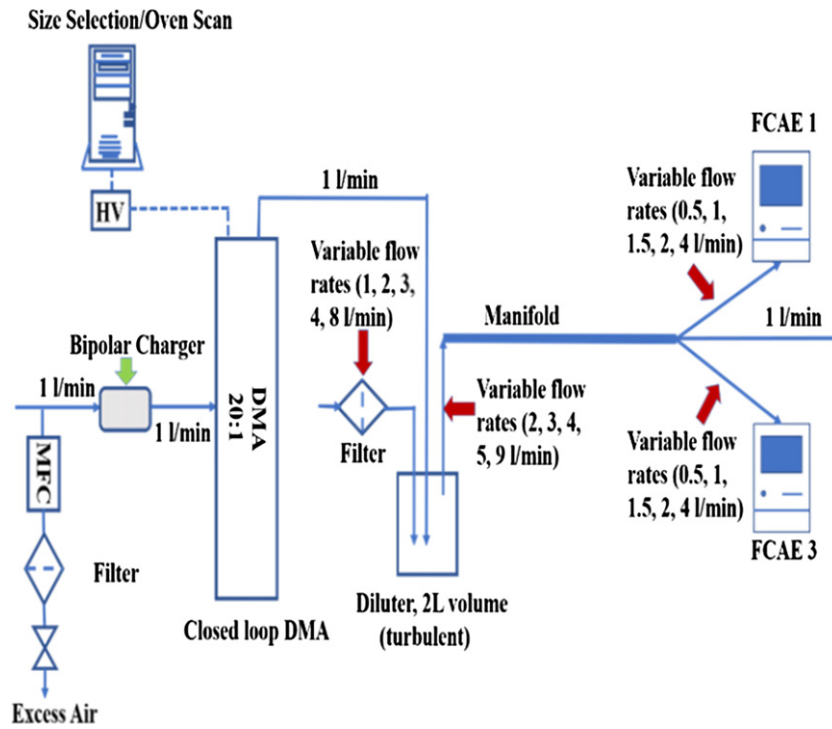


Figure 4. Size selection and inter-comparison setup for FCAEs at WCCAP from September 7–9, 2020. Here, FCAE 1 and FCAE 3 flow rates were varied (0.5 l min^{-1} , 1 l min^{-1} , 1.5 l min^{-1} , 2 l min^{-1} , 4 l min^{-1}) and the CPC was fixed at 1 l min^{-1} . The flow rates through the manifold were variable. Dashed and solid lines represent electrical connections and tubes, respectively. [10] Taylor & Francis Ltd. <http://tandfonline.com>.

where \bar{I}_3, \bar{I}_4 and \bar{I}_5 are the current means in the 2 min intervals in steps (c), (d) and (e) as indicated in figure 3. Negative currents were measured because of the DMA polarity.

2.3. Second workshop: sample flowrate effects in FCAEs

In the second workshop, two of the above-described technically different FCAEs were selected for the comparison: FCAE 1 (3068B™, TSI, USA) and FCAE 3 (BAM, Germany). The setup for this comparison was different from that of the previous workshop. Figure 4 shows that an additional monitoring CPC was incorporated to verify the stability of the particle source. The CPC was placed at end of the line, i.e. downstream. The setup facilitated the operation of both FCAEs simultaneously with identical sample flowrates, which were varied from 0.5 l min^{-1} to 4 l min^{-1} . The tube length from the manifold to each of the FCAEs was again 70 cm and its width remained unchanged. The consecutive steps for current measurements were similar to those in the first workshop, except in step (a) the particle size of 30 nm was checked and maintained and in step (b) the sample flowrates were changed. A flow-dependent reduction in the current signals due to diffusion loss in the tube was taken into account and quantified [24]. The measured electrical currents were been corrected for the tube losses by the following formula:

$$I = I_{\text{measured}} / P_q \tag{2}$$

where P_q indicates the calculated penetration factor for 30 nm particles passing through a tube of 0.7 m length and 6 mm

Table 1. FCAE sample flows and calculated penetration factors P_q for 30 nm Ag particles.

$q/\text{l min}^{-1}$		P_q
FCAE 1	FCAE 3	
4.00	4.00	0.991
1.98	2.00	0.986
1.48	1.49	0.983
0.98	1.00	0.978
0.50	0.50	0.966

width at flowrate q . The flowrate settings are listed in table 1 along with the respective penetration factors.

3. Results and discussion

3.1. Particle size effects in FCAEs

Silver particles of 5 nm, 10 nm, 20 nm and 30 nm mobility diameter were generated at different PNCs and, correspondingly, the measured absolute currents differ. Figure 5 compares baseline-corrected current readings from the FCAEs under test, normalized to FCAE 1.. The error bars in figure 5 consider the flowmeter’s 2% relative error as a minimum uncertainty. The electrical current mean values of FCAEs 2, 3 and 4 are denoted by open circles and the error is the standard deviation.

All FCAEs correspond quite well within the error margins, especially in the size range $\geq 20 \text{ nm}$. At particle sizes $\leq 20 \text{ nm}$ the FCAEs—even FCAE 2, which is the twin of FCAE

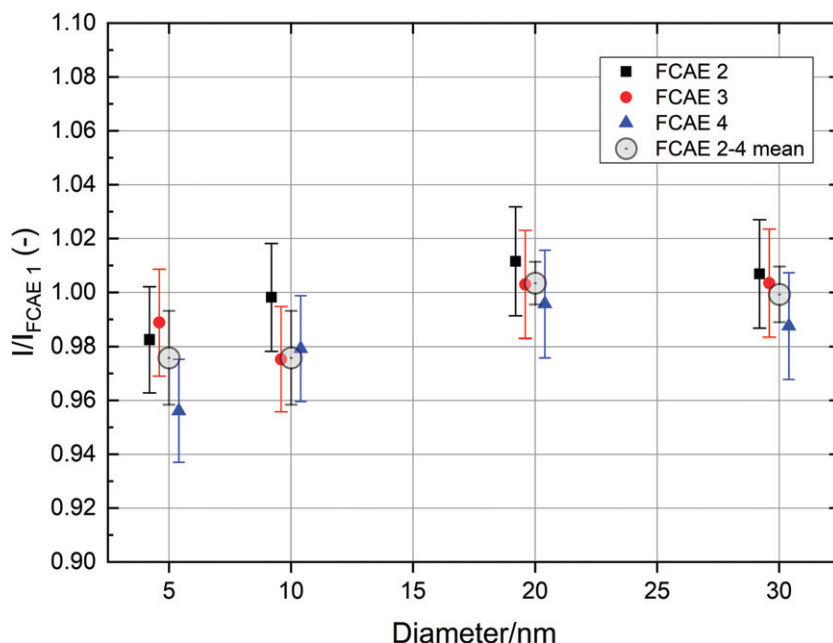


Figure 5. Current values from FCAE 2, 3 and 4 normalized to the current measured in FCAE 1. For each particle size, values have been shifted on the abscissa for better visibility.

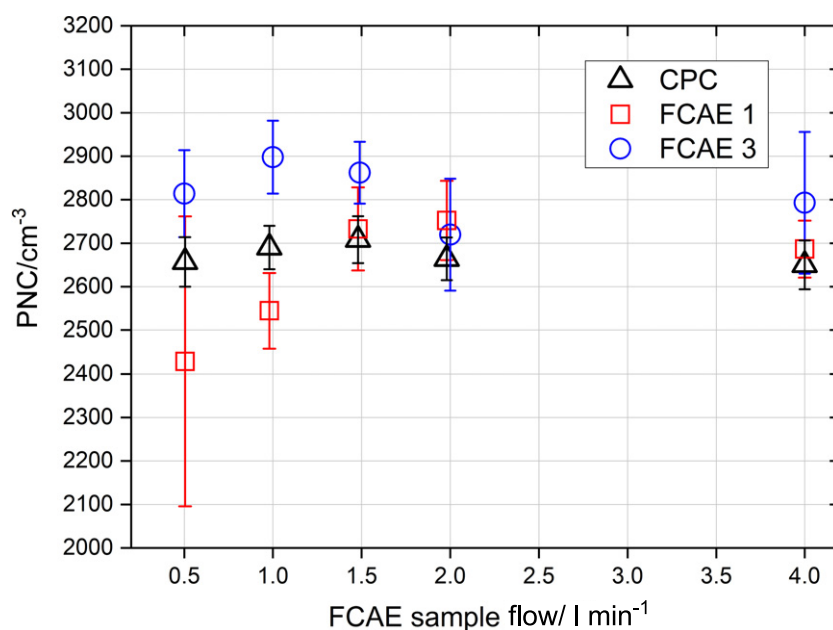


Figure 6. PNCs measured by FCAEs 1 and 3 at different sample flowrates in comparison to the CPC.

1—tend to slightly underestimate and the scatter around the means is slightly increased. These results corroborate preliminary findings from previous workshops at the WCCAP. The following conclusions can be drawn here. At particle sizes larger than 20 nm, different FCAE constructions show no significant effects on the detection efficiency. For particles smaller than 20 nm, particle size-related effects can be estimated not to exceed approximately $\pm 2\%$, which is the maximum deviation of the FCAE 2–4 mean at 5 nm. Because of the very good correspondence, 30 nm particles seem to be a rea-

sonable choice to investigate the sample flow-related effects of FCAEs.

3.2. Flow rate effects in FCAEs

Particles with a diameter of 30 nm were used for this experiment for the reasons pointed out above. The measured sample flowrates and calculated penetration factors from five measurement runs are shown in table 1. Monitoring the particle source by the CPC downstream of the FCAEs revealed that the variation in PNC over time could experimentally be kept

at approximately $\pm 2\%$ only. This allows for an observation of flow effects in FCAEs at almost constant PNC.

As shown in figure 6, the calculated PNC from the FCAEs corresponds for sample flowrates above 1.5 l min^{-1} quite well to that of the CPC. However, at smaller flowrates, each of the FCAEs deviates from the CPC by more than 2%. Within the given measurement uncertainties, this can be regarded as a significant discrepancy well above the suspected 1%. FCAE 3 tends to overestimate while FCAE 1 clearly underestimates the CPC. The difference between the FCAEs and the discrepancies between each of the FCAEs and the CPC tend to increase when lowering the sample flow. For both FCAEs, the enlarged error bars at lower sample flows result from the lower current levels.

4. Conclusion

This investigation describes an inter-comparison of aerosol electrometers for (a) their internal size-dependent diffusional losses at sub-30 nm particle sizes and (b) flow-dependent diffusional losses at sample flowrates from 0.5 l min^{-1} to 4 l min^{-1} .

Four FCAEs—among which two are technically identical—were compared in a first workshop, designed to check their particle size dependency. At a high sample flowrate of 4 l min^{-1} and for Ag particles above 20 nm in diameter, FCAEs 2, 3 and 4 revealed less than 2% deviation from FCAE 1, which served as a reference for comparison. For smaller particles, the FCAEs showed a slight trend to underestimate FCAE 1 but all FCAEs still corresponded within error limits.

In the second workshop, two FCAEs with different designs simultaneously measured 30 nm particles at varying sample flowrates. This comparison revealed no significant deviations at sample flowrates above 1.5 l min^{-1} . However, significant discrepancies occurred below 1.5 l min^{-1} . As the flowrates of the FCAEs were carefully adjusted and the data were corrected for flow-dependent particle losses in the tubes, the deviations are most likely assignable to design-specific losses in the FCAEs, which obviously become effective at low sample flowrates. Therefore, the statement from the beginning—that particle losses in the inlet have the potential to exceed 1%—could be corroborated by our findings for the two investigated FCAE designs, and no general conclusions can be drawn here. It remains for future work to find out whether this sample flow effect is observable in other FCAE designs too.

Most CPCs operate below 1.5 l min^{-1} . It is—for CPC calibration with particles below 30 nm—advisable to carefully check beforehand the possible occurrence of significant sample flowrates effects in a reference FCAE when it is run at the CPC's sample flowrates. The applied protocol and the measurement setup seem well suited to quantify the effect of low sample flowrates in reference FCAEs for a correct estimation of the contribution to the uncertainty budget.

Conflict of interest

The authors declare no conflicts of interest.

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