



An intercomparison study of good laboratory practices for aerosol number size distribution measurements using optical spectrometers

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Abstract. An inter-laboratory comparison (ILC) involving optical particle counters (OPCs) was organized at the French national level. The aim of this study was to make an inventory of the metrological capabilities of particle number size distribution (PNSD) measurements using OPCs. This laboratory study took place over a period of 18 months and involved 16 partners and 35 OPCs. Rather than focusing on the actual capability of the tested OPCs, this paper aims to reveal good laboratory practices when using standard OPCs. For that, each partner applied the same pre-defined experimental protocol on the OPC(s) to be tested, operated together with a common control OPC. Three different powder-borne test aerosols were involved, and their PNSDs were measured: (1) - a monodisperse amorphous silica sample, (2) - glass beads and (3) - a green cornstarch powder. This article presents the measured PNSD using the 35 OPCs associated with the description of the experimental set-up, sample preparation protocol and comparison with Scanning Electron Microscopy measurements.

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1 Introduction

Atmospheric aerosols are known to have considerable impacts on human health (Arfin et al., 2023). To control ambient particulate matter (PM) levels, European legislation (European Parliament, 2008) has established limits and target values for annual and daily mean mass concentrations for PM₁₀ and PM_{2.5} fractions. Thus, a dedicated air quality monitoring network has been deployed in France and Europe in order to measure routinely particle mass concentrations through monitoring stations in various locations. Such mass concentrations should be measured using the European gravimetric reference method defined in the Directive and as described in European standards (NF EN 12341, 2023). However, gravimetric analysis is an expensive time-shifted process that involves many steps. This implies difficulty in resolving short-term urban PM episodes temporally and delay in public reporting. Indeed, real-time techniques allow information retrieval with high temporal resolution of around 15 min, while the temporal resolution of gravimetric analysis is longer than one day. This is the primary reason why many European member states are relying on faster techniques by proving their equivalence to the gravimetric reference method (CEN/TS 16450, 2013). Such techniques are mostly based on optical methods, such as photometers (Gebhart, 2001), optical particle counters (Görner et al., 2012), and aerodynamic particle sizers (Pfeifer et al., 2016). However, these optical methods are dependent on particle size, shape, and refractive index. Accurate light scattering theories do not exist for such-complex shaped particles for which the Mie theory appears as an approximation (Mishchenko et al., 2002). For this reason, downstream use of calibration factors is required for converting number concentrations into mass concentrations. Indeed, the response of optical particle counters (OPCs) is based on the light scattered by particles, which occurs when a laser beam of defined wavelength interacts with focused particles. Each resulting detected scattered laser pulse corresponds to the occurrence of a particle, which allows temporal monitoring of the particle number concentration. The intensity of the scattered light at a given scattering angle is then analysed for each pulse to evaluate particle size. The latter is therefore expressed as an optical equivalent diameter (d_{opt}) which corresponds to the diameter of a spherical particle that scatters the same light intensity as the one scattered by a particle of given refractive index, thus applying the Mie theory. As a result, after calibration by applying the Mie theory, OPCs allow particle number size distribution (PNSD) measurements in real-time for particle sizes ranging from ~200 nm up to ~10 μm .

Interestingly, very few OPCs inter-laboratory comparisons were performed over the last 50 years. In their work, (Hindman Ii et al., 1978) performed a field comparison of PNSD measurements involving six OPCs. Systematic differences between the measurements from the various instruments were smallest for sub-micron particles by a factor of 1.5-2.5 and largest for micron particles by a factor of 8-15. More than 40 years later, (Vasilatou et al., 2020) presented a first inter-laboratory comparison for low particle number concentrations dedicated to clean room facilities. Their study was conducted for particle size ranging from 300 nm up to 5 μm and for number concentrations up to 2 particles. cm^{-3} using polystyrene latex spheres and sodium chloride/lactose monohydrate aerosols. Such inter-laboratory comparisons involved non-transportable facilities for the use of



70 primary methods for measuring particle number concentrations in full requirements of the ISO 21501-4 standard (Horender et
al., 2019; ISO 21501-4, 2018). For that reason, the authors used OPCs as transfer standards that were shipped to all participants.
They showed that all particle sizes agreed with the reference value within 7%, and were therefore compatible with the stated
uncertainties. Meanwhile, (Iida and Sakurai, 2018) presented a new methodology for evaluating the OPCs counting efficiencies
75 based on an ink jet aerosol generator which allowed producing monodisperse particles at a constant rate with lactose
monohydrate, ionic liquid, and sodium chloride. Nevertheless, and as stated by (Vasilatou et al., 2020), the metrological basis
of this study remains incomplete since the degree of equivalence was investigated by means of inter-laboratory comparisons.
As a result, to the best of our knowledge, no inter-laboratory comparison exists involving OPCs for measuring PNSD, while
such optical counters are used every day worldwide and precision is needed when precise air quality assessment is sought.

80 In this context, the present work aims at presenting the methodology and the results of an inter-laboratory comparison involving
several OPCs (35) for measuring PNSD by focusing on the same three test aerosols. We seized the opportunity of the French
network, built through previous cooperations and conferences, to carry out this inter-laboratory comparison at a national scale,
thus involving 16 research groups. The overall objective of this study was to make an inventory of the metrological capabilities
of various measurement techniques in France, with the idea to identify good laboratory practices. Experiments were carried
85 out using three aerosols samples: (1) - a monodisperse amorphous silica sample, (2) - glass beads and (3) - a green cornstarch
powder. To minimize biases, the dry-based aerosol generator involved in this work was identical for each partner and
completed with a common running controlled OPC, hereafter called the reference OPC; both devices were added to the
experiments performed by each partner.

2. Materials and methods

90 2.1 Experimental set-up

Inter-laboratory comparisons are usually constrained by the difficulty to move instruments at the same location in the same
time. The strategy chosen in this study was to use of a unique laboratory experimental set-up, involving a common dry-aerosol
generator and a reference OPC, which were sent to each participant prior to the experiments. Sixteen partners took part in this
inter-comparison exercise and received every part in order to build the experimental set-up in their own laboratories, as
95 presented in Figure 1.

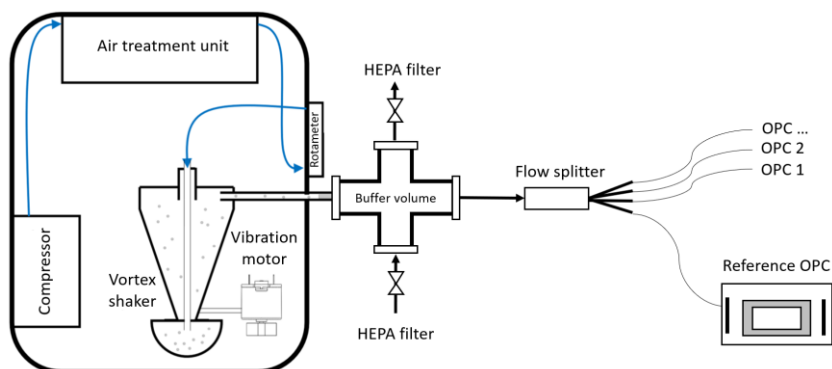


Figure 1. Experimental set-up used by each partner for this study.

100 The aerosol generator is based on the principle of a vortex shaker (VS-1000, ADDAIR, patent WO2013092816; (Leglise et al., 2022)). Within the generator, the filtered and dried air is injected from the upper part directly inside the Vortex mixer receptacle containing the powder sample to be aerosolised. The injection rod internal dimension is selected such as the flow in the receptacle is turbulent. The whole structure is connected to a vibrating motor with an eccentric mass to ensure stable and efficient agitation. The second zone, conical in shape, is subjected to an upward flow where the particles are selected according to their size by vertical elutriation. The air injection flow rate was set at 4 L/min for this study and the vibration frequency at 60 Hz (~12 V). A homogenization volume was coupled downstream of the generator in order to smooth temporal instabilities and to dilute the produced aerosols according to the total instrumental flowrate (Figure 1). A 4-way flow splitter (TSI model 3708) was involved to allow the use of multiple instruments in parallel, including the reference OPC. The latter was used as a common control measurement device (FIDAS Mobile, Palas) in order to provide measurements with the same instrument in each laboratory in parallel with the OPCs tested by each partner. Through the sixteen partners involved, 35 optical instruments were included in this inter-laboratory comparison, involving OPCs with different technical specifications, as presented in Table 1. Since there was no intention of including exclusively freshly calibrated instruments, the latter were categorized according to the time delay between their last calibration and the date of the experiments. 40% of the instruments had been calibrated less than one year earlier to the experiments, 26% between 1 and 2 years, and 34% had been calibrated more than two years prior to the experiments.

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Table 1: OPC types implicated in the present intercomparison study.

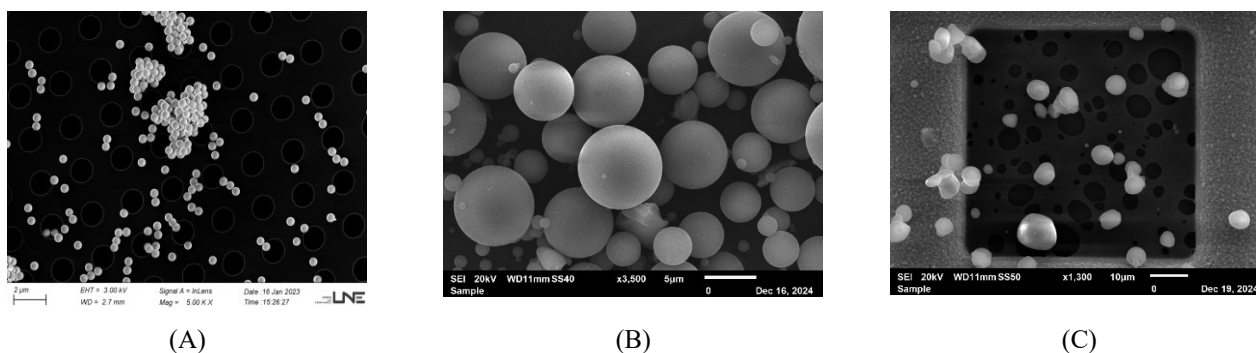
	TSI	Grimm				PALAS			
Models	OPS 3330	1.108	1.109	11D	Mini-Wras	Mobile	Frog	Fidas 200	Promo/Welas
Wavelength (nm)	660	780	655	655	660	White light LED /			Xenon arc lamp 35 W
Detection angle (°)	90 (±60)	90				90 (±5)			
Number of channels (size range, µm)	16 (0.3–10)	15 (0.3–20)	31 (0.25–32)	31 (0.25–35)	31 (0.25–35)	64 (0.18–18)			128 (0.2–10 / 0.3–17)
Flowrate (L/min)	1	1.2				1.4	1.4	4.8	5

125 2.2 Samples, preparation protocol and data acquisition

Three different powder-borne aerosols were investigated:

- (A) a monodisperse amorphous silica, 0.5 µm in size, from Angströmsphäre. This sample was used as a reference sample, allowing to verify the instrumental accuracy and to adjust measurements when required. This silica sample was already studied in the past to determine its refractive index, *i.e.* 1.45 (Hubert et al., 2017). This sample is hereafter labelled as the *A-sample*.
- (B) glass beads, which are named as Spherglass 5000CP00 (Potters) with refractive index in the range from 1.9 to 2.2. This sample is hereafter labelled as the *B-sample*.
- (C) a colored cornstarch powder, called green Holi powder (color people) with refractive index around 1.6. This sample is hereafter labelled as the *C-sample*.

Figure 2 shows a typical scanning electron microscopy (SEM) images of airborne particles sampled on carbon TEM grids from aerosols produced using the experimental setup presented in Figure 1 and using the Mini Particle Sampler (MPS) (R’Mili et al., 2013; Xiang et al., 2021) for each of these three samples.



140 **Figure 2. SEM images for the three powder samples, *i.e.* (A) – monodisperse silica, (B) glass beads and (C) – Holi powder.**



From these SEM images, particle sizes were found to be $0.486 \pm 0.015 \mu\text{m}$ for monodisperse amorphous silica, $4.06 \pm 2.792 \mu\text{m}$ for Spherglass, and $4.117 \pm 0.899 \mu\text{m}$ for Holi powder. It is worth noting that SEM measurements correspond to the geometrical diameter of particles, which may be different from the equivalent optical diameter reported by OPCs. All stock samples, preliminary prepared for all techniques, were provided to each participant and stored at room temperature and protected from light. Preparation protocols for powder samples to be analyzed were deliberately basic to be performed as simply as possible. Regarding data acquisition, each partner was required to record PNSDs with a 10-second acquisition time per sample and a total duration of 10 minutes. A blank measurement was performed between samples.

3. Results and discussion

3.1 Reference number size distributions

For each of the three test aerosols, all PNSDs measured by each partner with the reference OPC were used to calculate the averaged PNSD resulting from all measurements. Figure 3 presents the corresponding averaged PNSD for the three test aerosols, accompanied with an adjusted lognormal law and 95% confidence interval. The presence of submicron particles for samples B and C, also visible in SEM pictures provided in Figure 2, could be linked to powder synthesis process or physical frictions between grains (attrition).

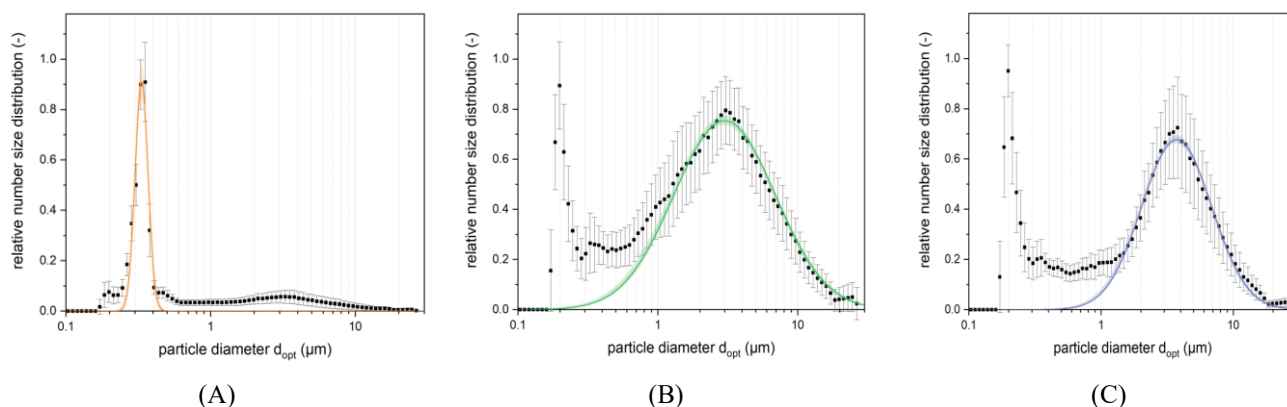


Figure 3. Average particle number size distributions measured by the control mobile Fidas instrument for powder samples A (monodisperse silica), B (glass beads) and C (Holi). Errors bars corresponds to calculated standard deviations.

The characteristics of the measured PNSD, as measured by the reference OPC, are given in Table 2.

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Table 2: Characteristics of the measured PNSD from the reference OPC.

	Modal optical diameter (μm)	Geometric standard deviation
<i>A-sample</i>	0.34 ± 0.01	1.11 ± 0.01
<i>B-sample</i>	3.05 ± 0.37	2.31 ± 0.03



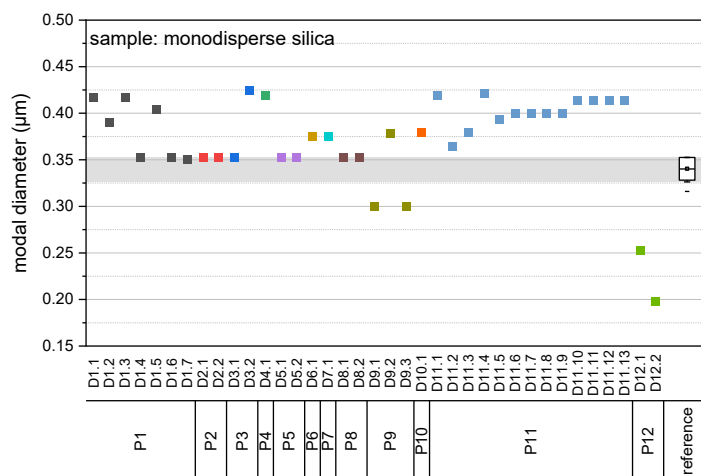
C-sample 3.74 ± 0.38 1.84 ± 0.02

165 It is important to note that default of accordance between SEM-based (Figure 2 and section 2.2) and OPC-based modal diameter measurements can be due to the fact that SEM provide a geometric diameter whereas OPCs return an equivalent optical diameter, *i.e.* the diameter of a particle with given refractive index that diffuses the same light intensity than the particle.

3.2 Modal diameters and Z-Scoring

170 Mean, modal and median diameters are commonly used to describe particle size distributions. In this study, only modal diameters were considered in data processing. This choice is justified because the modal diameter represents the most frequently occurring particle size within the distribution, providing a robust and representative measure of the predominant particle population. The modal diameters obtained by each partner were then normalized with respect to the modal diameter stemming from the measurement performed with the control OPC that was carried out simultaneously. Figures 4-6 present the modal diameters measured by each instrument involved for each powder sample and their ratios calculated in relation to the simultaneous measurement of control OPC, respectively. In these figures, each partner is referred to as “Pi” and device as “Di.j”. However, four partners were unable to provide data, and the following figures present the remaining twelve.

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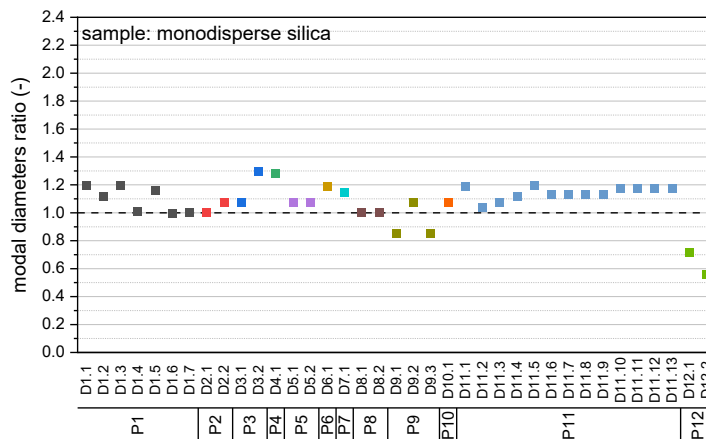
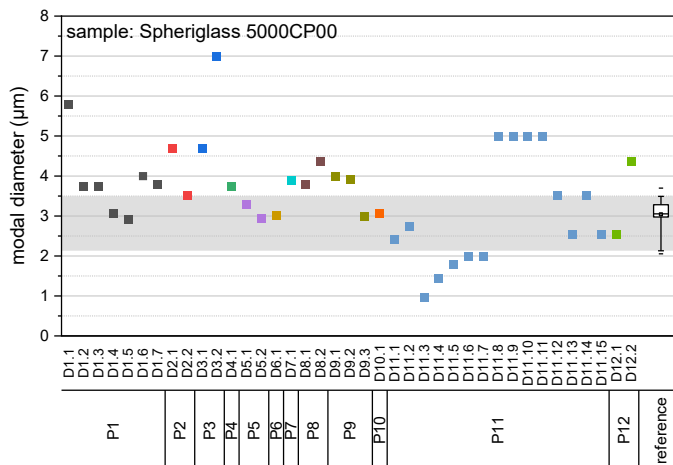
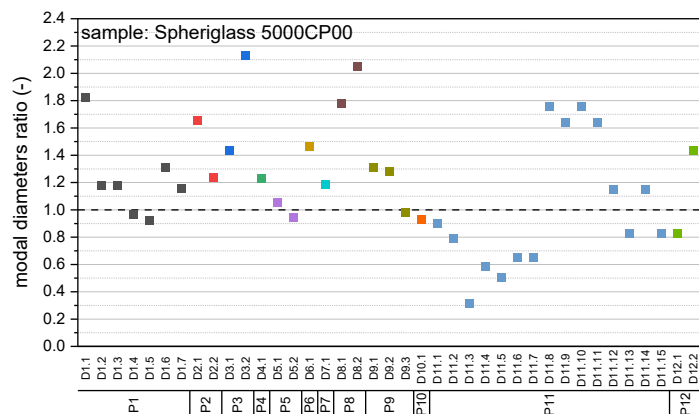


Figure 4. PNSD modal diameters of monodisperse silica. (top): measured by each partner. The grey area corresponds to the 90% confidence interval of the modal diameter measurements obtained with the reference instrument, (bottom): normalized with the reference instrument.



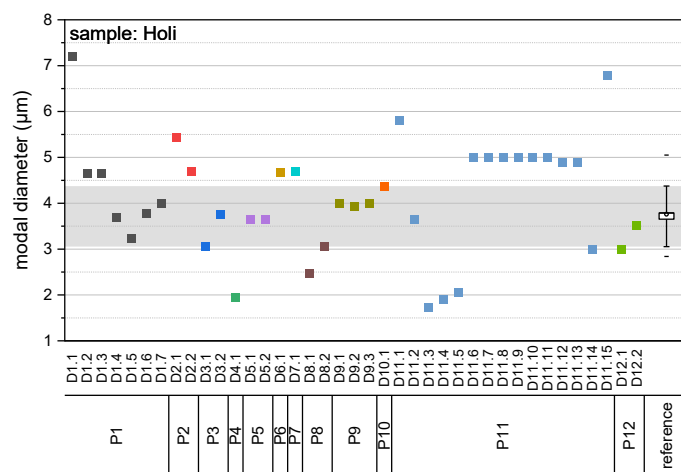
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185 **Figure 5. PNSD modal diameters of Spheriglass. (top): measured by each partner. The grey area corresponds to the 90% confidence interval of the modal diameter measurements obtained with the reference instrument, (bottom): normalized with the reference instrument.**

Concerning the aerosol generated from the monodisperse silica sample (Figure 4), all modal diameters are found within an interval of $\pm 30\%$ compared with the control measurement, except for one measurement. The comparability between the different devices and the reference instrument can be explained by the monodisperse nature of the test aerosol (geometric standard deviation of the distribution ~ 1.11).

190 Besides, a more significant dispersion is observed on the modal diameter ratios obtained on the two other samples, namely the glass beads (Figure 5, ratios between 0.3 and 2.15) and the Holi powder (Figure 6, ratios ranging from 0.4 to 1.9).



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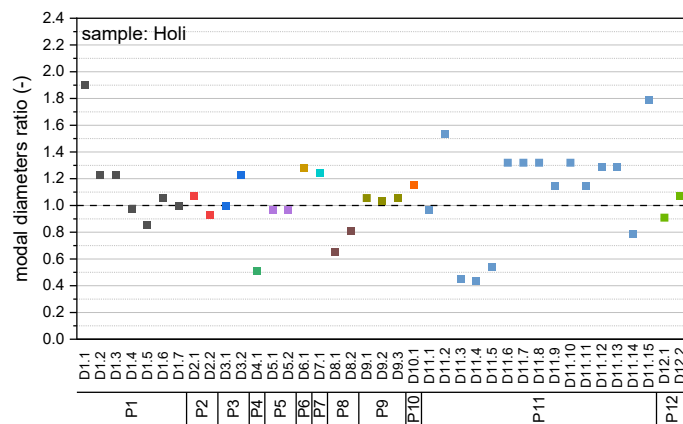


Figure 6. PNSD modal diameters of Holi powder. (top): measured by each partner. The grey area corresponds to the 90% confidence interval of the modal diameter measurements obtained with the reference instrument, (bottom): normalized with the reference instrument.

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Overall, a moderate spread suggests inter-instrument or inter-laboratory variability. Most measurements cluster near the reference instrument, though a few outliers occur at both lower and higher values, likely reflecting differences in calibration, measurement methods or sample handling. About the ratios of normalized modal diameters to the reference, most measurements are close to unity and fall within an acceptable range (approximately $\pm 10 - 15\%$) confirming an overall satisfying agreement. A few points lie outside this range, highlighting systematic deviations in data provided by certain partners that may require further investigation.

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An analysis based on the Z-score performance criterion (ISO 13528, 2022; Thompson et al., 2006) was conducted in order to consider both the discrepancy between the modal diameters and the variability of the control measurement. Z is defined as:

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$$Z = \frac{d_{mod_{OPC_i}} - d_{mod_{OPC^*}}}{2\sigma(d_{mod_{OPC^*}})} \quad (1)$$

where $d_{mod_{OPC_i}}$ and $d_{mod_{OPC^*}}$ represent the modal diameters from the tested OPC and from the control measurement respectively, and $\sigma(d_{mod_{OPC^*}})$ the repeatability standard deviation calculated from all the control measurements. Depending on the Z value, the measurements can then be classified into performance zones:

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- $|Z| > 3$ are considered to be unsatisfactory values (“warning zone”);
- $2 < |Z| \leq 3$ are considered to be questionable values (“surveillance zone”);
- $1 < |Z| \leq 2$ are coherent values and correspond to acceptable performance;
- $|Z| \leq 1$ are optimal values and correspond to excellent performance.



220 In order to better understand the Z-scoring, each OPC involved in this interlaboratory comparison was classified in function of the technical specification associated to commercial type. The results are presented in Figure 7 for each aerosol sample.

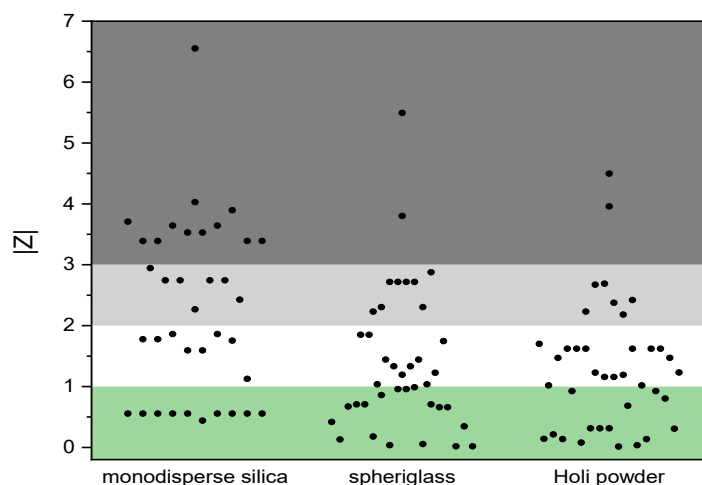


Figure 7. Z-score calculated for each OPC tested in the interlaboratory comparison for the three test aerosols.

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More than three-quarters of the studied OPCs are located in the Z-score zone ranging from 0 to 2 for the aerosols generated from the glass beads and Holi powder samples. This proportion is lower (~ 50%) for the monodisperse silica aerosol, which was characterized by a distribution with a modal diameter below 400 nm and a lower repeatability standard deviation (0.01 μm).

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By considering the overall results, two thirds of the involved OPCs are in optimal and acceptable zones. More precisely, this fraction corresponds to 66%, 60% and 77% of the types TSI, Grimm and Palas, respectively. It was then intended to seek dependence between Z-scores and instrumental characteristics. However, the correlation matrix did not reveal a significant effect of any parameter tested (number of size channels, time since last calibration, etc.). It is therefore believed that the biases between measured and reference modal diameters are multi-factorial. One question rises about the choice of the mobile Fidas

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as a control instrument, which may have improved the performance evaluation of other Palas devices. This choice was mainly motivated by the higher size-channel resolutions of Palas OPCs compared to the other types (Table 1). The size dependence and variability of the scattered light intensity with particle morphology and refractive index influence the performances of OPCs (Szymanski et al., 2009). Other parameters can also impact the measured pulse height spectrum of OPCs such as the coincidence due to the presence of more than one particle in the sensing volume and the inability of the electronic system to

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process such events. These limitations may affect the comparability of results between different OPC types which are usually calibrated by the manufacturers or, less frequently, by users. Calibration of OPCs can also be used to characterize the instrument correlation to particles with different refractive index and morphology from those of calibration particles (Szymanski and Liu, 1986). In terms of particle size and number concentration, (Marple and Rubow, 1976) calibrated an OPC



with respect to aerodynamic particle size using cascade impactors, which appears as a time-consuming methodology. Other approaches were also developed using cyclones and filters on the OPC inlet. Comparisons to aerodynamic particle sizers, scanning mobility particle sizers and/or electron microscopy measurements were also performed (Binnig et al., 2007; Sousan et al., 2016). In that way, (Sang-Nourpour and Olfert, 2019) developed a new calibration technique involving an aerodynamic aerosol classifier (AAC) coupled to an OPC to be tested for the size calibration and to a condensation particle counter for the number concentration. Their protocol presents the advantage of being not limited to a specific particle material or aerosol generation method. Among the parameters that may be at the origin of the deviations observed in relation to the control measurement, the degree of expertise of the operator in charge of the tests, as well as the time since the last calibration/maintenance of the device are to be considered. It is therefore difficult to pinpoint a single parameter responsible for the observed deviations between instruments and further propose a set of good laboratory practices. Depending on the aerosol concentration used during the experiments carried out by each partner, coincidence effects can have disproportionately affected measurements. Instrument-specific factors, including differences in size-channel resolution, calibration history, operator expertise and time since last maintenance, further complicate comparability and interpretation. Because OPC devices are typically calibrated with reference particles whose optical properties do not match those of the test aerosols, these differences likely introduce significant biases in the particle size measurement, highlighting the challenges of achieving precise inter-laboratory agreement.

260 4. Conclusion and perspectives

This inter-laboratory comparison provides a comprehensive assessment of the metrological performance of OPCs for measuring particle number size distributions across a range of aerosols. The study involved 16 research groups and 35 OPCs of various types, tested on three representative aerosol samples: monodisperse amorphous silica, glass beads, and green Holi powder. For the monodisperse silica sample, measurements were within $\pm 30\%$ of the reference OPC, reflecting the high comparability of instruments for narrow size distributions. Greater variability was observed for the glass beads and Holi powder, which exhibited broader size distributions and complex particle morphologies. Overall, two-thirds of the instruments were in optimal or acceptable Z-score zones with 66%, 60% and 77% for TSI, Grimm and Palas devices, respectively. Most measurements clustered near the reference but outliers highlighted systematic deviations linked to factors such as calibration history, size-channel resolution, particle morphology, and refractive index differences between calibration standards and test aerosols. Coincidence effects due to higher aerosol concentrations during partner experiments were also identified as contributing to measurement variability.

These results confirm that while OPCs provide generally reliable PNSD measurements, the observed inter-instrument deviations emphasize the complexity of ensuring consistent performance across laboratories. Our database does not allow good laboratory practices to be proposed yet. Nonetheless, this study establishes both a methodology and a reference dataset for improving OPC reliability at national and international scales. Therefore, future work focusing on harmonizing calibration



procedures using aerosols that better represent real-world optical properties, correcting for coincidence losses, and standardizing best practices for instrument maintenance and data acquisition is still needed. Expanding inter-laboratory comparisons to include a broader range of particle types, sizes, and concentrations will further strengthen confidence in OPC measurements supporting more accurate and timely air quality monitoring.

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