On-line Characterisation of Aerosols – Comparability and Combination of Selected Measuring Devices

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Abstract

A scanning mobility particle sizer (SMPS, TSI Model 3934) and a white-light optical particle counter (OPC, as described by Umhauer 1983) are combined. With these two instruments, fast and non-intrusive on-line measurements of particle size distributions in a range of 15 nm up to 15 μm are possible. A filter provides the possibility of taking samples in order to analyse the morphology and chemical composition of the aerosol. Both liquid and solid airborne particles in diluted suspensions can be measured. The complete system is able to cope with particle concentrations of up to \(10^7\) particles/cm³ without dilution of the aerosol. Since the measuring principles of the combined devices are based on different physical properties, careful calibration is necessary. Both theoretical calculations and experimental methods are used for calibration purposes. The measuring results of different devices are compared during simultaneous characterisation of several aerosols. The performance of the combined measuring system is demonstrated by means of comparison with a common cascade impactor in the laboratory as well as during industrial application.

Introduction

Particulate emissions are often still characterised solely by mass concentration. In modern environmental technology and with regard to particle separation, however, more detailed data are necessary. Health hazards cannot be quantified solely by respirable mass, but by the physical and chemical properties of the inhaled particles. Fibrous particles of certain dimensions and aspect ratios, for example, are suspected of being carcinogenic with little influence of the bulk material. On the other hand, very fine particles that neither consist of dangerous bulk material nor have a critical shape are able to adsorb relatively high amounts of hazardous substances due to their relatively high specific surface area. Thus, dangerous components can be transported by harmless particles into the deepest regions of the human lung where they may become injurious to health.

Detailed information on particle size distributions and chemical composition is of growing interest, not only concerning human health. The latest developments in particle technology increasingly show the tendency to operate in the submicron and nanometer size range. Especially with new materials, e.g. ceramics, nanoparticles have interesting qualities. The production and handling of nanoparticles as well as the characterisation and design of separators and qualified description of emissions, however, call for high-performance measuring systems.

Optical particle counters that determine particle sizes and concentrations by analysing scattered light have become a common tool in aerosol measurement technology. Devices of the type described by Umhauer (1983) are able to measure particle sizes and concentrations fast, non-intrusively, and on-line. The measurable range of concentrations amounts to between 10 and \(10^6\) cm⁻³, which means for spheres of unit density and a diameter of 1 μm, mass concentrations of between approximately 0.005 mg/m³ and 5 g/m³.

An important reason not to use white-light optical particle counters in modern emission control systems and during material processing is their relatively high detection limit of approximately 0.5 μm. However, in many emissions most of the emitted particles are
smaller than this limit. Under environmental aspects especially, these small particles are dangerous to human health.

The goal of our work was to develop a measuring system which is suitable for measurements over a wide range of both particle sizes and concentrations. In order to meet these requirements, the white-light optical particle counter was combined with a scanning mobility particle sizer, the detection limit of which amounts to 15 nm. Thus, a size range from 15 nm up to 15 μm is covered by the combination of the chosen devices.

The main problem with this combined system is uniform calibration. As the scattered light intensity depends on both the bulk material and the particle shape, the optical particle counter had to be calibrated carefully.

Experimental results, e.g. for polystyrene latices and glycerine droplets, were confirmed by Mie calculations for spherical particles (Sachweh, 1991). Calibration methods for “aerodynamic” size measurements (Büttner, 1983, 1990; Heidenreich et al., 1995; Friehmelt and Heidenreich, 1999) were applied for non-spherical particles such as quartz dust. The non-sphericity of particles severely affects the shape of the calibration curve.

Experimental results show that the scattered light intensities depend on particle shape as well as on particle orientation (Bottlinger, 1989). Zerull and Killinger (1987) found that the forwards and sideways scattering of larger particles is enhanced by a non-spherical particle shape in comparison with spheres. Experiments with quartz particles have shown that the enhancement of scattered light intensity by particle shape starts at equivalent volume diameters of around 0.5 μm (Friehmelt and Heidenreich, 1999).

The scattered light intensities caused by non-spherical particles in a 90° white-light optical particle counter are simulated by means of a discrete dipole approximation (DDA) code (DDSCAT, Draine, 1998). Calculations were carried out for particle diameters ranging from 0.1 μm to 2 μm in order to cover the size range in which particle shape becomes important to light scattering. DDA simulations with spherical particles were verified by Mie calculations which proved the method to be applicable.

A compact and mobile calibration unit was developed in order to be able to calibrate measuring devices on-line. The separation characteristics of the cyclone configurations, which are used in this calibration unit, were determined experimentally with glycerine droplets. A universal grade efficiency curve was specified by applying the dimensionless representation proposed by Büttner (1999). Thus, the calibration unit is applicable to any aerosol.

In order to verify the counting and sizing performance as well as the resolution of the measuring devices used in the combined system, simultaneous measurements were carried out with several aerosol analysers such as an aerodynamic particle sizer, a laser diffraction device, a laser aerosol spectrometer, a condensation particle counter, and a cascade impactor.

The mobility analyser and optical particle counter proved to have high resolution and good classification accuracy. The decreasing counting efficiency of the optical particle counter is taken into account when measuring in combination with the mobility analyser.

Instrumental set-up

The complete system for the characterisation of aerosols is shown in figure 1. The collection probe includes a cyclone in order to separate coarse particles which are larger than the upper detection limit of the measuring devices. With the help of an optional ejection dilution system, the sample can be conditioned. Thus, condensation can be avoided and concentrations adapted to meet the requirements of the individual measuring devices.

The optical particle counter used in our investigations is of the type that was first described by Umhauer (1983). The aerosol is illuminated by a white light source. The scattered light intensity of single particles that pass the sensing volume is collected around a scattering angle of 90° and is converted to an electrical pulse by a photomultiplier tube. The pulse height depends on particle size, shape and bulk material, especially the refractive index of the particles, and is used to determine the particle size. Since the
Sensing volume is optically defined and the aerosol is not aerodynamically focused, high concentrations can be measured without any dilution (Sachweh et al. 1998). However, the measurable concentration is limited by the counting principle, which rules out the possibility of more than one particle being present in the sensing volume at the same time.

The lower detection limit of the OPC is 0.2–0.5 μm, depending on the specific device. Therefore the scanning mobility particle sizer (SMPS, TSI Model 3934) was chosen to extend the lower detection limit of the combined system down to 15 nm. In the SMPS, the aerosol is electrostatically classified. The concentration in the individual size classes is then determined by a condensation particle counter.

The aerosol sample stream is taken isokinetically and completely drawn through the OPC. Downstream of the OPC, another sample is taken and fed to the SMPS. A filter provides the possibility to either determine the total mass concentration or to take samples for the scanning electron microscope or the X-ray diffractometer in order to analyse the morphology and chemical composition of the aerosol.

The whole set-up was designed to be variable to permit adaptation of the complete system to a wide field of applications as described above. Before the designed set-up could be applied, a lot of problems had to be investigated carefully. The different sensors had to be calibrated uniformly and their accuracy has to be controlled constantly. Concentration measurements had to be verified, and especially the influence of coincidences and the counting efficiency of the SMPS, the aerosol particles between the classifier and the condensation particle counter.

The application of the concept was proved to be linear. This means that the differential scattering cross-section, which is calculated from Mie theory, can be converted to a representative pulse height by multiplication with a constant. If this constant has been determined once, the OPC can be calibrated without any further experiment for any spherical particles of known refractive index (Friedmelt and Heidenreich 1999).

Figure 2 shows the results of experimentally derived and calculated calibration curves for PSL-spheres and glycerine droplets. For spherical particles, good agreement between Mie theory and experiment is achieved.

In the case of the experimental calibration with PSL-spheres, several monodisperse fractions of PSL were measured and the mode of the intensity distribution was correlated to the given particle size.

With the glycerine droplets, an experimental calibration method was applied which had been proposed by Heidenreich et al. 1995. The particle size distribution of an aerosol, for which the measuring device is to be calibrated, is determined by an aerodynamic particle sizer (APS, TSI Model 3310) and the measured shape factor which is defined as the relation of the drag force acting on a particle to the drag force on a volume equivalent sphere of the same velocity.

In a first step, both measuring devices were calibrated by monodisperse fractions of polystyrene latex (PSL) spheres. The accuracy of the SMPS is improved by choosing longer scanning times and depends on the correct determination of the so-called delay time, which takes into account the residence time of the aerosol particles between the classifier and the condensation particle counter.

Calibration

The different measuring devices and varying calibration techniques lead to size distributions which are related to different equivalent diameters. A uniform particle size distribution for the combination of the measuring devices is derived from calculating the equivalent volume diameter \( d_v \) of both the mobility diameter \( d_m \) and the aerodynamic diameter \( d_a \). The equivalent volume diameter is given by Hinds (1982):

\[
d_v = \frac{d_m C_u(d_a)}{\chi C_u(d_m)} \quad d_a = \frac{\chi C_u(d_a) \rho_p}{C_u(d_m) \rho_s}^{0.5}
\]

where \( C_u \) is the slip correction, \( \rho_p \) is the particle density, \( \rho_s \) is unit density (1 g/cm³), and \( \chi \) is the dynamic

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**Fig. 2** Experimental calibration of the OPC for PSL-spheres and glycerine droplets in comparison with theoretical calibration curves.
The comparison of the different cumulative distributions leads to a calibration curve for aerodynamic particle sizes. This method is also suitable to calibrate the OPC for non-spherical particles. However, since the results of the APS are also influenced by particle shape, the APS has to be calibrated as well.

Figure 3 shows the calibration of the APS for quartz particles. For calibration purposes, a quartz standard (BCR) with certified size distribution was used. By reference, the given and measured cumulative mass were compared and the measured diameter is plotted against the correlating reference diameter. In comparison with our experimental results, some calculated data of Cheng et al. (1990) and measurements published by Marshall et al. (1991) are plotted, which are both valid for a dynamic shape factor of around $\chi=1.19$, while the dynamic shape factor of quartz is approximately $\chi=1.36$. Our experimental results seem to be in reasonable agreement with literature. The error bars are caused by experimental uncertainties:

- The quartz standard has to be dispersed prior to the measurement. If this redispersion is not complete, the certified size distribution will not be reached.
- The sample includes particles smaller than the lower detection limit of the APS, which may cause "phantom particles".
- The counting efficiency of the APS decreases towards its lower detection limit.
- The certified size distribution of the quartz standard includes some variation.

Another method of calibrating measuring devices for "aerodynamic" size measurements is calibration by means of sampling cyclones (Büttner 1983, 1990), which is based on the fact that the median of the grade efficiency of a cyclone is uniquely related to the aerodynamic diameter. The comparison of measured medians of grade efficiency curves with the known separation characteristic of the particular cyclone yields the calibration curve. This method is of special interest because it is mobile and measuring devices can be calibrated on-line. A schematic diagram of a portable calibration unit and a photo of our technical design is shown in figure 4.

The results of a calibration of the OPC for quartz dust are shown in figure 5. Both calibration methods yield similar results. However, the calibration curve gained by the characterisation of cyclones seems to be shifted to smaller particle sizes.

Irregularly shaped monodisperse particles will cause an intensity spectrum when measured by the OPC, since the scattered light intensity depends on particle orientation in the sensing volume. In the case of quartz particles, this intensity spectrum will be asymmetric (Bottlinger 1989). These asymmetric intensity spectra lead to a broadening and a shift of measured grade efficiencies (Friehmelt 2000).
symmetric spectra, the median of the grade efficiency would be measured correctly, and perfect agreement between the different calibration techniques would be achieved.

Due to the irregular particle shape, the scattered light intensity when compared to the calculations for quartz particles under the assumption of spherical shape is increased for larger particles and reduced for smaller particles. In contrast to spherical particles (see figure 2), the use of theoretical calibration curves derived by Mie calculations will severely falsify the measured size distributions of irregularly shaped particles. In order to simulate the scattered intensities caused by non-spherical particles, numerical methods have to be applied. In our investigations, a discrete dipole approximation code (DDSCAT, Version 5a9, by Draine 1998) was used.

Discrete dipole approximation means that the investigated particle is replaced by an array of point dipoles. Any particle can thus be approximated by a finite number of discrete dipoles. The scattering problem for this array of dipoles is then solved exactly (Draine and Flatau 1994).

The number of dipoles was approximated according to the guidelines given in the user guide for DDSCAT (Draine and Flatau 1998) and checked for each investigated symmetry. The scattered intensity at the detectors of the OPC was calculated from the results for 289 scattering directions which were chosen according to the detection aperture of the OPC. Calculations were carried out under the assumption of parallel incident light. All calculations have to be performed with one discrete illuminating wavelength. The white-light source was approximated by the superposition of seven wavelengths between 300 nm and 600 nm. The spectral composition of the light source and the spectral sensitivity of the photomultiplier tubes were taken into account when calculating the detected intensity. Using 13 instead of 7 wavelengths with a cube of 1 \( \mu m \) effective diameter leads to a difference in averaged intensity of less than one percent.

Calculations were carried out for particle diameters in the range of 0.1 \( \mu m \) up to 2 \( \mu m \) and for 125 orientations of the particles relative to the incident light beam. In figure 6, theoretical calibration curves for some model particles are plotted. The data points are derived from the scattered light intensity averaged over all orientations as an approximation for random orientation of the particles in the sensing volume. Discrete dipole approximation (DDA) with spherical particles is verified by Mie calculations. Both scattering calculations lead to the same result. Cubes and rectangular prisms with square cross-section and a ratio of length to width of two to one ("short prism") and five to one ("long prism") served as non-spherical particles. In the case of the prisms, 250 orientations were averaged. From figures 5 and 6 it is obvious that the tendency of enhanced scattered intensities is modelled correctly by DDA for larger non-spherical particles. No significant difference in scattered intensities between cubes and short prisms was detected. For the long prisms, the scattered intensity seems to be reduced in comparison with cubes and spheres with small particle sizes, which correlates to the observations with quartz particles and calibration by comparison of cumulative size distributions.

Another important quality of particle sizing devices is resolution. A single non-spherical particle may cause different scattered intensities depending on its orientation. Instead of a monodisperse size distribution, a broad distribution will be measured. Bottlinger (1989) showed that the relative standard deviation (standard deviation normalised by mean value) of such broadened distributions increases with particle size and growing deviation from spherical shape. Table 1 shows the relative standard deviations of the model particles, calculated by means of DDA. The relative standard deviations of spheres remains below 0.1 and exhibits no systematic tendency, which confirms that the simulation method yields reasonable results.

### Table 1

| Volume Equivalent Diameter [\( \mu m \)] | 0.1 | 0.3 | 0.5 | 0.7 | 1 | 1.5 | 2 |
|------------------------------------------|-----|-----|-----|-----|---|-----|---|
| Sphere                                  | 0.0014 | 0.0063 | 0.063 | 0.082 |
| Cube                                    | 0.013 | 0.18 | 0.49 | 0.69 | 0.84 | 1.32 | 1.82 |
| Prism 1:2                                | 0.13 | 0.63 | 1.36 | 1.60 |
| Prism 1:5                                | 0.79 | 1.26 | 1.64 | 1.78 | 1.94 |

Fig. 6 Calibration of the OPC for regular non-spherical particles gained by DDA calculations.
small differences in scattered intensities of a sphere under varying orientations are caused by the incomplete approximation of the spherical shape by a finite number of dipoles. The calculations for non-spherical particles confirm Bottlinger's findings. Relative standard deviations increase with growing particle size and from cube to prism and from the short prism to the longer one (see table 1).

DDA calculations with varying detector apertures showed that averaged intensities should not be influenced by neglecting the illumination aperture. However, relative standard deviations of the intensity spectra will be slightly increased by the assumption of parallel illumination.

All the calibration experiments and calculations show that a proper calibration is absolutely essential if different particle sizes are to be compared. Another problem is that size distributions of non-spherical particles are adulterated by the sensing characteristic of almost any aerosol measuring device. Errors caused by the sensing characteristic can be corrected by a suitable inversion algorithm provided that the systematic falsification of measuring results is known. The OPC simulations showed that DDA can serve as a tool to model systematic errors such as the broadening of size distributions caused by non-spherical particles.

**Comparison of different measuring devices**

Simultaneous measurements with different instruments were carried out both in the laboratory and in industry. In figure 7 the detection limits of the devices used in our investigations are illustrated. The cascade impactor (Andersen Inc., Type M III) covers a wide size range but has a weak size resolution, and the gravimetric determination of size distributions is time-consuming, and it is restricted to a narrow concentration range and is susceptible to errors. Due to its robust design and the fact that it yields mass concentrations, which are important in emission control, it is often used in industry. Since the size ranges of AFS and OPC are very similar and the APS has a high sizing accuracy and resolution (Friehmelt 2000), it is suitable for the experimental calibration of the OPC as described above. A high-sensitivity laser aerosol spectrometer (HS-LAS, PMT GmbH) was applied to check the performance of OPC and SMPS near their detection limits. The SMPS itself was chosen to cover the submicron size range. All measuring devices except for the impactor provide measuring results on-line within seconds (at most three minutes).

**Performance of OPC and SMPS near their detection limits.**

The size ranges of OPC and SMPS overlap reasonably. However, especially around the detection limits of the measuring devices, systematic errors may occur. The upper detection limit of the SMPS and the lower detection limit of the OPC were investigated within simultaneous measurements with the HS-LAS. The measurement of particles in ambient air, carried out with OPC, HS-LAS and SMPS simultaneously, are plotted in figure 8. In the case of the SMPS, no systematic error was detected — as figure 8 clearly shows. After proper calibration, HS-LAS and SMPS yield the same result. Good agreement was also achieved with the measuring results of the OPC. In the case of the OPC, however, some additional effects have to be taken into account when optical particle counters are compared with other measuring devices — as figure 9 shows.

Different monodisperse fractions of polystyrene latex (PSL) spheres were investigated in the overlapping size range between OPC and HS-LAS. The measuring results of three discrete fractions are plotted in figure 9. The particle size distributions support agreement and systematic errors. For example, excellent conformity of the particle size distributions measured by OPC and HS-LAS was achieved with 0.356 μm
latex spheres. Near the detection limits of the sensors, however, measured distributions are adulterated. The upper detection limit of the HS-LAS is given by its calibration curve. For particles larger than \( d = 0.4 \ \mu m \) ambiguities appear and measured distributions are broadened, as measurements of 0.48-\( \mu m \) latex spheres in figure 9 show. The lower counting efficiency of the OPC can be observed for the 0.2-\( \mu m \) spheres. In order to quantify the counting efficiency of the OPC, the concentrations of monodisperse aerosols were measured with an OPC and a condensation particle counter (CPC) simultaneously.

**Combination of OPC and SMPS**

After having verified the performance of OPC and SMPS in the size range where both instruments overlap, several simultaneous measurements were carried out with these two devices and different aerosols. As an example, the results for glass microspheres and quartz particles are plotted in figure 11. The measured number concentrations in the overlapping size range agree well, provided that the OPC is properly calibrated and the counting efficiency of the OPC is taken into account. The results of the two measuring devices can be combined to a continuous size distribution which covers a size range of three decades.

**Combination of OPC and SMPS versus cascade impactor**

The advantages of the combined measuring system comprising OPC and SMPS are high resolution both in time and particle size. Reasonable measuring times range from 1 to 3 minutes for the combined system, while an impactor measurement may take hours, depending on the total concentration of the aerosol. Furthermore, results of the combined system are available on-line, while the gravimetric evaluation of impactor measurements is not possible at all on-line.

**Figure 12** illustrates the resolution in time for an aerosol of glass microspheres with moderately changing concentration. Changes in concentration can be observed on-line with the combined system, while the impactor yields a mean concentration with an additional delay caused by the evaluation procedure. The mass density of the same glass aerosol is plotted in figure 13 and demonstrates the higher resolution in particle size of the combined system. In principle, the mass density distribution and total mass concentration agree well, in this case for spherical particles.

Even in the case of non-spherical particles such as
Fig. 12 Resolution in time of cascade impactor in comparison with the combination of SMPS and OPC.

Fig. 13 Resolution in particle size of cascade impactor in comparison with the combination of SMPS and OPC.

Fig. 14 Simultaneous measurements of quartz particles with combination of SMPS and OPC and cascade impactor.

Fig. 15 Diagrammatic view of the experimental set-up for blow-by measurements.

For the combined measuring system, the concentration range described by the error bars amounts to 3.49–9.0 mg/m³.

**Industrial application**

The measurement of the blow-by aerosol from an Otto engine shall serve as an example of the industrial application of the combined particle characterisation system. During ignition the pressure inside the piston of an Otto engine increases rapidly. Although the piston is sealed, the pressure difference between piston and crankcase will cause a leak flow which drags along oil droplets. For environmental reasons, this so-called blow-by aerosol has to be drawn off the engine housing, recirculated and injected again with the combustion air. In order to avoid losses of oil and interference with the combustion process caused by oil droplets, the aerosol has to be separated before injecting the recirculated blow-by gas. The task was to characterise the blow-by aerosol at different locations all around the engine housing in order to find a suitable position to draw off the aerosol and to design the separation step properly.

The experimental set-up is shown in figure 15. The blow-by aerosol is routed to a chamber which helps to dampen pressure pulsations, unify concentration and size distribution and separate coarse particles. From this chamber, the aerosol is fed to the combined measuring system as described above or to the cascade impactor in alternation. The volume flow through the measuring devices is metered and controlled.

The OPC was calibrated for the oil droplets by means of the mobile calibration unit described above. Additionally, a theoretical calibration curve was gained by Mie calculations, since the refractive index n of the quartz, good agreement between the combined measuring system and the cascade impactor are achieved as demonstrated in figure 14. For the combined system the range of probable size distributions is estimated because of the uncertainty with the calibration of the OPC as described by figure 5. When comparing the total mass concentration, it has to be mentioned that particle losses in the cascade impactor have not been taken into account. For the combined measuring system, the concentration range described by the error bars amounts to 3.49–9.0 mg/m³.
specific oil could be determined. Both coinciding calibration curves are plotted in figure 16. Again, both cascade impactor and the combined measuring system lead to comparable results as demonstrated by figure 17.

However, a set of measurements at seven locations around the engine housing was carried out within less than half a day with the combined system, whereas it took about a week with the impactor. In addition, defects such as a leak in the measuring system will be obvious at once with the combined system, since measuring results are available on-line. Troubleshooting and repetition of the measurement will cause no significant loss of time. If using the cascade impactor, it may take days to become aware of any defects.

The measurements revealed increasing mass concentration with growing speed and load, which could be expected, and significant changes in mass concentration and size distribution depending on the location in the engine housing. However, the investigated locations could be divided into two groups as illustrated in figure 18. Mass concentration and size distribution at several locations near the top of the engine housing are similar as well as at three locations near its bottom, inside or near the crankcase. At the bottom locations, concentrations are higher and the size distribution is shifted to larger particles compared with the top locations. However, the total mass concentration for submicron droplets, which are difficult to separate, is nearly constant throughout the engine.

Conclusion

A measuring system was presented that covers a size range from 15 nm up to 15 μm. The different measuring devices were calibrated uniformly. For spherical particles, the calibration curves of the scattered light detectors can be derived from Mie calculations, whereas for non-spherical particles, empirical aerodynamic calibration is applied. Measurable concentrations exceed \(5 \times 10^6\) particles/cm³.

The combination of an optical particle counter and mobility analyser on the one hand and a common cascade impactor on the other hand were compared under ideal conditions in the laboratory as well as with demanding industrial applications. Reasonable agreement between both measuring systems was achieved in terms of both size distributions and aerosol concentrations. Compared with the cascade impactor, measurements with an optical particle counter are much faster, measuring results are obtained on-line, and a higher resolution both in time and in particle sizes can be achieved.

The limitations of the described combined measuring system are primarily given by the effects of irregular particle shape. Like all common aerosol analysers, especially the optical particle counter is affected...
by non-sphericity and surface structures of the investigated aerosol particles. With experimental calibration methods some uncertainties have to be taken into account. However, a powerful simulation method could be established, which already improved the understanding of shape effects in optical particle sizing and may serve as a basis for further investigations.

Symbols and Abbreviations

\( c_n \) : number concentration \( \text{[cm}^{-3}\text{]} \)

\( C_u \) : slip correction \( [-] \)

\( d_a \) : aerodynamic diameter \( \text{[um]} \)

\( d_e \) : equivalent volume diameter \( \text{[um]} \)

\( d_m \) : mobility diameter \( \text{[um]} \)

\( n \) : refractive index \( [-] \)

\( \chi \) : dynamic shape factor \( [-] \)

\( \rho_o \) : unit density \( [1 \text{kg/m}^3] \)

\( \rho_p \) : particle density \( [\text{kg/m}^3] \)

APS : aerodynamic particle sizer

CPC : condensation particle counter

HS-LAS : high sensitivity laser aerosol spectrometer

OPC : optical particle counter

PSL : polystyrene latex

SMPS : scanning mobility particle sizer

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