

Institute of Environmental Sciences and Technology

IEST-RP-CC014.2

Contamination Control Division
Recommended Practice 014.2

Calibration and Characterization of Optical Airborne Particle Counters



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1 SCOPE AND LIMITATIONS

1.1 Scope

This Recommended Practice (RP) covers procedures for calibrating and characterizing the performance of optical particle counters (OPCs) that detect and measure the size of single particles in air and other gases. These procedures are intended for use by OPC manufacturers, specialized test houses, and OPC users who maintain calibration and testing facilities to determine the sizing and counting accuracy of these instruments.

1.2 Limitations

The procedures for characterization and calibration described in this document use monodisperse particles. The response of actual contamination particles, typically with refractive indices different from the monodisperse particles used for calibration, will differ slightly from the results obtained from the procedures in this document. Also, the differences will vary for particle counters with different optical designs.

This RP does not discuss calibration of condensation nucleus counters (CNCs). The document assumes CNCs used as reference counters have 100% counting efficiency in the range of interest for OPCs.

NOTE: This RP does not purport to address all of the safety issues that may arise from its use. The user is responsible for assessing potential safety issues associated with a given method at its point of use. Before using the methods described herein, the user should consider all general laboratory safety precautions. In particular, the user should identify and implement suitable health and safety measures and comply with all applicable regulations.

2 REFERENCES

The following documents are incorporated into this RP to the extent specified herein. Users should apply the most recent editions of the references.

2.1 Reference Documents

IEST-RD-CC011.2: A Glossary of Terms and Definitions Related to Contamination Control

IEST-RP-CC013: Calibration Procedures and Guidelines for Select Equipment Used in Testing Cleanrooms and Other Controlled Environments

ISO 21501-4: Determination of particle size distribution—Single particle light-interaction methods—Part 4: Light-scattering airborne particle counter for clean spaces

2.2 Sources and Addresses

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3 TERMS AND DEFINITIONS

The following terms have special meaning in the context of this RP. See also *IEST-RD-CC011*.

calibration

The comparison of a measurement standard or instrument of unknown accuracy with another standard or instrument of known accuracy to detect, correlate, report, or eliminate by adjustment any variation in the accuracy of the unknown standard or instrument.

coefficient of variation

A measure of the width of a distribution, expressed as a percentage, obtained by dividing the standard deviation of the distribution by the mean of the distribution.

coincidence

The presence of two or more particles in the sensing volume of the instrument at the same time, causing the instrument to interpret the combined signal erroneously as resulting from one larger particle.

concentration

The number of particles per unit volume at ambient temperature and pressure.

concentration limit

The particle concentration specified by the manufacturer at which the error due to coincidence is 10% or less. (Manufacturers may specify concentration limits at error levels other than 10%.)

counting efficiency

The ratio, expressed as a percentage, of a detected concentration of monodisperse particles divided by the actual concentration of particles of a given size or range of sizes.

false count

The maximum particle count indicated by a particle counter, in a given volume, that is sampling particle-free air. This value is specified by the manufacturer, and commonly is also referred to as false call rate, false count, noise, or noise level.

inlet flow

The gas that enters the optical particle counter (OPC) through the gas inlet. Flow rate is expressed as volume per unit time, at ambient temperature and pressure. (See also **sample flow**.)

lower counting limit

The smallest particle size, specified by the manufacturer, at which the OPC is capable of 50% counting efficiency.

monodisperse particles

Polymer spheres composed of polystyrene or a similar polymer, sometimes abbreviated “PSL,” having a

refractive index of 1.58–1.61 and a distribution in which the coefficient of variation is 5% or less when suspended in liquid. When dry particle standards are used, the allowed coefficient of variation should be as low as possible, but may be as high as 15%. [See also polymer microspheres (PSL).]

most penetrating particle size (MPPS)

The particle size at which a given filter has its highest penetration (or lowest efficiency); i.e., the worst-case particle size with respect to filtration efficiency. From filtration theory, efficiency is higher for particle sizes smaller or larger than the MPPS. In practice, the MPPS of a filter is considered to be within a measured size range typical in commercial instruments rather than at one unique particle size.

NMI

A term referring to a national metrology institute (e.g., NIST (US), NPL (United Kingdom), PTB (Germany), or AIST (Japan)).

optical particle counter (OPC)

A light-scattering instrument with display or recording means to count and size discrete particles in air and other gases.

particle size

For calibration, the mean diameter of the monodisperse particles. For applications, the diameter of a reference sphere having known properties and producing the same response in the OPC as the particle being measured.

polymer microspheres (PSL)

A term referring to a common type of highly uniform spheres (or a suspension thereof) used for characterizing and calibrating an OPC. (See also **monodisperse particles**.)

resolution

A measure of the ability of a particle counter to differentiate between particles of different sizes; also, the range of sizes an instrument would ascribe to a particular particle if its size were determined repeatedly.

response rate

A measure of the time a particle counter takes to recover from exposure from a high-particulate concentration to that of a particle-free condition. NOTE: If the airflow movement characteristics through the optics chamber are poor, residual particles may result in an extended response rate. Sedimentation of residual particles may cause contamination of the sensor.

sample flow

The gas that passes through the sensing volume of the particle counter. The sample flow may be a portion of the inlet flow. Flow rate is expressed as volume per

unit time, at ambient temperature and pressure. (See also **inlet flow**.)

sensing volume

The portion of the illuminated region through which the aerosol passes and from which scattered light is collected by the detector.

4 BACKGROUND AND PURPOSE

This RP provides standardized test procedures to calibrate and characterize the performance of optical particle counters. OPCs are used to define airborne particle size distribution and concentrations in air and other non-hazardous gases. The data obtained from these instruments are used to verify cleanroom and clean device classification, to characterize air filter performance, to verify installed filter system integrity, and to characterize particle emissions from potential contamination sources. It is often necessary or desirable to use different OPCs operating at different locations or different OPCs at the same location at different times. OPCs of different design may not produce the same data from identical gas samples. Such discrepancies may occur even with similar OPCs if calibrated differently. For these reasons, a standardized calibration methodology should be established to minimize variability among different OPCs. The procedures in this RP are voluntary, not required, and should be used in conjunction with an agreement between customer and supplier considering the requirements of ISO 21501-4.

The procedures defined in this RP include:

- Calibration of inlet flow rate (section 6)
- Particle size calibration (section 7)
- False count (section 8)
- Counting efficiency (sections 9 and 10)
- Response rate (section 11)
- Concentration (coincidence) limit (section 12)
- Resolution (section 13)

5 REQUIREMENTS

5.1 Materials and equipment

The following equipment may be needed to perform the measurements described in this document. Forms for recording test data may be found in Appendix A.

Section 6—Calibration of inlet flow rate

- Flowmeter
- Tubing, hose clamps, or fittings
- Stop watch

- Thermometer
- Barometer

Section 7—Particle size calibration

- Monodisperse particles (NMI traceable)
- Diluent
- Aerosol generator
- Oscilloscope
- Pulse height analyzer

Section 8—False count

- Filter

Section 9—Counting efficiency; Method 1

- Monodisperse particles
- Aerosol generator
- Aerosol size separator
- Mixing chamber
- Tubing
- Reference counter/CNC

Section 10—Counting efficiency, Method 2

- Monodisperse particles
- Aerosol generator
- Mixing chamber
- Tubing
- Reference counter—OPC certified by the primary method (section 9)

Section 11—Response rate

- Filter
- Monodisperse particles
- Diluent
- Aerosol generator

Section 12—Concentration (coincidence) limit

- Same as for section 9

Section 13—Resolution

- Same as for section 7

5.2 Concentration of particles

For all tests described in this RP (unless otherwise noted), the concentration of particles should be no more than 25% of the concentration limit specified by the manufacturer of the instrument.

5.3 Environment

The calibration and characterization of the OPC should be performed in a controlled environment (temperature, humidity, vibration). The degree of environmental control necessary should take into

account the operational specification of all instruments used.

5.4 Initial setup and warm-up of the OPC

Before performing any procedure, confirm that the OPC is turned on and the operating parameters of the OPC are set to the standard settings or the values recommended in this document. Allow time for the instrument to warm up as specified by the manufacturer or for a minimum of 30 min.

6 CALIBRATION OF INLET FLOW RATE

6.1 Apparatus and materials

The following equipment may be needed to perform the measurements described in this document. Forms for recording test data may be found in Appendix A.

6.1.1 Flowmeter

A flowmeter allows the determination of the flow rate in the sample inlet of the OPC. The accuracy of the flowmeter should be $\pm 3\%$ of full scale or better. The flowmeter should have been calibrated within the time schedule specified in IEST-RP-CC013. The pressure drop of the flowmeter should be sufficiently small that it does not introduce an error greater than 1% in the nominal flow rate due to additional pressure drop.

NOTE: This procedure requires measurement of the volumetric flow rate. Different flowmeters require different parameters to be measured to convert their readings to volumetric flow rate. Appendix B discusses the use and precautions applicable to different types of common flowmeters. Not all of the apparatus described in this section are necessary for use with all flowmeters.

6.1.2 Tubing

Tubing couples the flowmeter to the OPC inlet. The inside diameter should be large enough that it does not restrict the flow. The tubing should be sufficiently conductive that it does not build up a static charge during use such that particles collect. Tubing made from materials such as stainless steel and polyester has been found to perform satisfactorily.

6.1.3 Tubing fittings and connections

Hose clamps or fittings are used as necessary to make a leak-free connection between the flowmeter and the inlet of the OPC.

6.1.4 Stop watch

A stop watch with a resolution of 0.01 sec is used.

6.1.5 Thermometer

A calibrated thermometer with an accuracy of $\pm 0.2\text{ }^{\circ}\text{C}$ ($\pm 0.5\text{ }^{\circ}\text{F}$) of reading is used.

6.1.6 Barometer

A calibrated barometer with an accuracy of $\pm 1\text{ mm Hg}$ ($\pm 0.04\text{ in. Hg}$) of reading is used.

6.2 Procedure

a) Connect the flowmeter to the inlet of the OPC using as short a length of tubing as possible. The gas flows first through the flowmeter and then into the inlet of the OPC.

b) Turn on the OPC and allow enough warm-up time to obtain uniform, stabilized gas flow through the inlet.

c) Record the reading of the flowmeter on Form 1, Conditions for Characterizing Optical Particle Counter (Appendix A).

NOTE: The volumetric flow rate is required for this procedure. If a mass flowmeter is used, convert the reading to a volumetric flow rate.

d) Additional flow measurements may be taken. If the OPC allows the user to change the flow rate, the flow controller can be characterized by measuring the flow rate at different nominal flow rates in accordance with the procedures of steps b and c. Operate the OPC only within the manufacturer-suggested range of flow rates, and measure flow rates within the desired operating range.

e) If more than one flow rate was measured, plot a calibration curve showing the sample flow rate indicated by the instrument versus the measured flow rates.

f) Report the volumetric flow rate (volume per unit time) at local ambient operating conditions. Do not correct to standard conditions.

7 PARTICLE SIZE CALIBRATION

NOTE 1: Specific procedures for calibrating different OPCs can vary considerably, not only among manufacturers but also among instrument models. Some OPCs require calibration using specialized equipment not generally available to the typical OPC user. For these reasons, this RP cannot provide detailed procedures for calibrating every OPC. This section presents general concepts that are important for performing a particle size calibration on any OPC. For detailed procedures for calibrating a specific OPC, contact the manufacturer.

NOTE 2: This procedure requires that either the particle counter can be operated as a pulse-height analyzer, or the voltage pulses from the OPC are available for connection to an external pulse-height analyzer.

NOTE 3: The response of some OPCs is not monotonic over the entire range. That is, size ranges can exist within the operating range of the OPC where voltage output both increases and decreases as particle size increases (see figure 1). Calibration particle sizes should be chosen to permit definitive description of any such reversals.

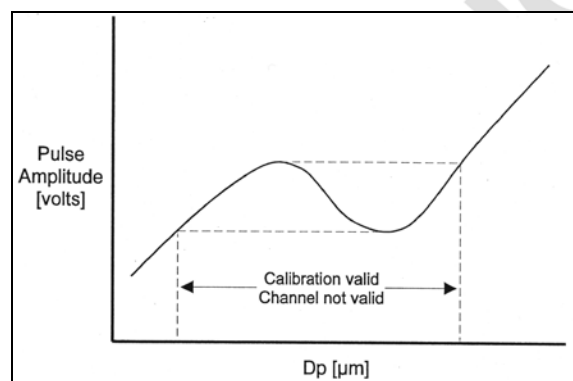


Figure 1—Non-monotonic region of an OPC calibration curve.

NOTE 4: The particles used for calibrating the OPC should be certified using calibration procedures and equipment traceable to national or international agencies. This certification may be provided by the manufacturer of the monodisperse particles used for calibration.

7.1 Apparatus and materials

7.1.1 Monodisperse calibration particles

The particles used for calibration should be monodisperse particles as defined in this RP. The diameter of

the spheres should be defined with an error no greater than 3% and be NMI traceable. The coefficient of variation of the particles in liquid suspension should be no greater than 5%. When dry particle standards are used, the allowed coefficient of variation should be as low as possible, but may be as high as 15%.

7.1.2 Diluent

Calibration particles are normally supplied in concentrations too high to be used directly in aerosol generators; therefore, dilution may be necessary. Clean water should be used for dilution. The water should be cleaned using a filter with a pore size no more than 10% of the size of the particles being suspended, and stored in a clean container before use.

7.1.3 Aerosol generator

The aerosol generator uses an atomizer that converts the monodisperse particle suspension to an aerosol for calibration. The aerosol generator uses compressed gas to generate the aerosol and transport the resulting particles to the particle counter. The compressed air, or other suitable gas used to generate the aerosol, should be filtered using a filter that meets the requirements of section 8.1, because the same gas supply will typically be used for dilution as well as atomization. Dry dilution gas is used to evaporate the water from the droplets generated by the atomizer, and may be used to reduce the aerosol concentration. A bipolar ion source may be used to reduce electrostatic charge on the particles to minimize agglomeration and losses by deposition in transit to the OPC. Aerosol generators typically can be obtained from the OPC manufacturer.

7.1.4 Oscilloscope

An oscilloscope is used to view the voltage pulses generated by the OPC.

7.1.5 Pulse height analyzer (PHA)

A pulse height analyzer is used to collect and analyze the distribution of voltage pulses generated by monodisperse particles. The required range and speed depend on the performance of the OPC being calibrated. These parameters should be obtained from the OPC manufacturer.

The pulse height analyzer should have at least 64 channels and a resolution of at least 1% of the average voltage that will be measured. If the particle counter includes a pulse height analyzer that meets these criteria, that analyzer can be used for calibration.

7.2 Calibration procedure

7.2.1 Initial setup

- a) Attach the output line of the aerosol generator to the OPC inlet. If the output of the aerosol generator exceeds the flow rate of the OPC, an excess flow bypass vented to ambient air will be required. If the aerosol generator provides less flow than is required, dilution air will be required. The dilution air should be passed through a filter with a pore size of 0.2 μm or less.
- b) Turn on the OPC and confirm that it is set for standard operation. Set the OPC to count in the cumulative mode and set the first channel of the OPC to measure at the lower counting limit of the OPC.
- c) Connect an oscilloscope to the OPC to observe output pulses. Connect a pulse height analyzer to the OPC if the particle counter does not contain an acceptable pulse height analyzer.

7.2.2 Diluent cleanliness verification

- a) Clean the atomizer liquid reservoir and fill it with clean diluent. Turn on the compressed-air flow to the atomizer and to the dilution line, and adjust the atomizer pressure and flow rates, as recommended by the aerosol generator manufacturer.
- b) Turn on the OPC counting system and determine the number of counts reported at the smallest size for which the OPC is specified. Observe the oscilloscope screen to confirm the OPC is generating predominantly individual pulses. Collect counts for three 1-min periods. Determine the average number of particles reported per liter of air. If that number exceeds 1 particle/L (28 particles/ ft^3), the diluent should be refiltered before further use.

NOTE: Diluted monodisperse particles 1 μm and smaller should not be stored for more than one week.

7.2.3 OPC threshold-setting procedure

- a) Select the particle size ranges for which calibration is required. Vigorously agitate the suspensions of calibration particles. Add the well-mixed suspension of monodisperse particles to the atomizer liquid reservoir. Turn on the compressed-air flow to the atomizer and adjust the dilution line flow as described in section 7.2.2.

NOTE: The concentration of monodisperse particles in the atomizer should be sufficiently small that most droplets contain only one or no particles. The amount of concentrated particle solution to be added to the atomizer varies greatly with particle size and solids

concentration in the suspension. Typically, if the diluted suspension in the atomizer is noticeably cloudy, the concentration of particles in the atomizer may be too high. Observe the oscilloscope screen to confirm the aerosol concentration is low enough that the OPC is generating predominantly individual pulses. Adjust the dilution line flow so the counting rate is no more than 25% of the concentration limit of the OPC.

- b) Allow the flow system to stabilize.
- c) Accumulate data to include at least 1000 pulses.
- d) Determine the median OPC pulse voltage amplitude from the pulse height analyzer. The median voltage can be found by defining a “region of interest” that includes the peak that represents the voltage pulses from single calibration particles (see figure 2). The median voltage is that voltage which divides the number of pulses in this region such that half are greater than the median and half are less than the median.

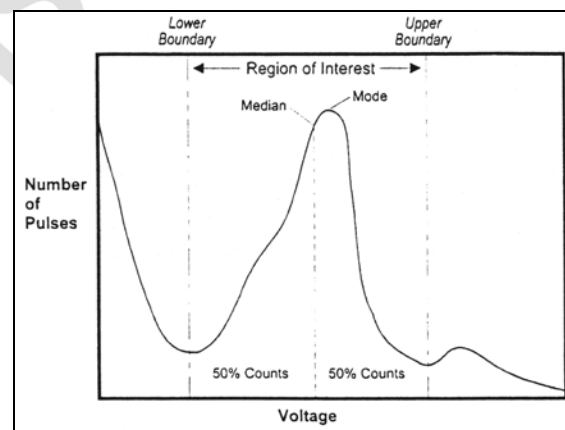


Figure 2—Determination of median voltage using a PHA distribution.

- e) Repeat this procedure for all particle sizes selected. If a single atomizer is used, the atomizer should be rinsed with clean diluent three times before adding a new suspension of particles.

NOTE: Care is required when the procedure is carried out for the smallest size particle for which the OPC is specified. Record the OPC noise level. The average voltage for the smallest size particle should be at least 1.1 times the stated noise-level voltage at which one noise pulse occurs in 1 min. If the threshold voltage for the smallest specified particle size is too close to the stated noise level, the validity of data for that size may be questionable.

7.3 Reporting results

a) Record calibration results on the calibration table [Form 2, Optical Particle Counter Calibration Table (Appendix A)], which should be stored with the particle counter. It may be useful to plot the calibration voltages against particle size on a log-log graph.

b) Note any polytonic response of the calibration curve for the OPC and the size range(s) over which the polytonic response may occur. In this region of the calibration curve, one voltage corresponds to more than one size.

8 FALSE COUNT

The intent of this procedure is to determine whether or not the instrument is performing within its specification, not to measure the actual value of the false count rate. It is assumed that the actual false count rate of a properly operating instrument exceeds its specification such that any failure of the verification test can be attributed to physical failure within the instrument and not to statistical variation in the measurements.

8.1 Apparatus and materials (filter)

The filter should have either a pore size $\leq 0.2 \mu\text{m}$ as measured in liquid or a removal efficiency $\geq 99.97\%$ at the most penetrating particle size (MPPS) as measured in air. The pressure drop across the filter should be small enough that the sample flow rate can be maintained at its nominal value.

8.2 Procedure

The OPC should be calibrated before the false count rate can be verified (see section 7). The false count rate is specified at a 95% upper confidence level of false counts per cubic meter.

a) The minimum acceptable sample time is 10 min. Using a longer sample time will yield more accurate results. The maximum sample time should be limited so that the observed count does not exceed a value of 10.

b) Attach a filter to the inlet of the OPC to purge residual airborne particles from the sensor.

c) Following the purge, begin recording counts for the sample time t determined in step a.

d) Record the sample time and the cumulative counts that occurred on the smallest channel during the sample time.

e) Calculate the volume sampled during the test as follows:

$$V = Q \times t \quad (1)$$

where:

V = volume sampled

Q = flow rate (unit volume/min)

t = sample time (min)

f) Total the number of counts observed during the sample and determine the 95% upper confidence limit (UCL) from table 1.

Table 1—Observed Count and 95% UCL.

Observed Count	95% Upper Confidence Limit
0	3
1	4.7
2	6.3
3	7.8
4	9.2
5	10.5
6	11.8
7	13.1
8	14.4
9	15.7
10	17.0

g) Calculate the false count rate as follows:

$$F = U / V \quad (2)$$

where:

F = false count rate (counts per cubic meter)

U = 95% UCL of observed counts

V = volume sampled during test (cubic meters)

h) Record the information on Form 3, False Count Verification Worksheet (Appendix A).

8.3 Acceptance

1) The acceptable count level N is met if the false count rate is less than or equal to the false count rate specified by the manufacturer, Z .

2) If the OPC indicates counts low enough to meet the false count rate specification considering the actual volume sampled, and t is at least 10 min, the test may be terminated early with the OPC passing false count verification.

3) If the OPC fails the initial test, the OPC should be tested two more times as outlined in section 8.2. If the OPC passes both the second and third tests, then the false count specification is verified.

- 4) If the OPC fails either the second or third test, the false count specification is *not* verified.

NOTE: If the counts for consecutive measurements decrease, there may be residual particles contaminating the OPC inlet. In this case, continue recording counts until the counts appear to stabilize, then attempt the false count verification again.

If the counts are far above the acceptance count N on the first test, try one or more of the following before repeating the test:

- 1) Use a new filter on the inlet of the OPC.
- 2) Check the OPC and its connections for leaks.
- 3) Rap the filter and sample lines repeatedly while gas flows through them. Continue to purge until the counts return to a stable value.
- 4) Connect the filter so its source is the gas leaving the OPC; recirculate while observing the false count.
- 5) Isolate the OPC from strong sources of electrical noise.
- 6) Contact the manufacturer for assistance.

9 COUNTING EFFICIENCY—METHOD 1

This section describes the use of a CNC as the reference instrument to verify and measure the counting efficiency of an OPC. This method is used mainly in a laboratory setting. Counting efficiency is determined at the lower counting limit and an additional size that is 1.5 to 2.0 times the lower counting limit size. Counting efficiency should be determined at the following times:

- 1) After realignment of the optics.
- 2) Immediately before the counter is to be used for a critical or high-accuracy measurement.
- 3) Regularly, based on the stability of the counter as determined by replicate counting efficiency measurements over extended lengths of time. See IEST-RP-CC013 for guidelines for establishing calibration schedules.
- 4) Every 12 months.

9.1 Apparatus and materials

9.1.1 Monodisperse particles

The spheres should be of the composition described in section 7 and should have a coefficient of variation of 5% or less. The measured diameter of the particles should be NMI traceable. The precision of the mea-

surement of the mean diameter should be $\leq 3\%$ of the diameter.

9.1.2 Aerosol generator

The aerosol generator consists of an aerosol atomizer, dryer, and neutralizer. The atomizer should output an aerosol concentration that varies by no more than 10% of this time period as measured by the reference counter over periods of 15 min or greater. A dilute suspension of polymer microspheres (PSL) can be created by gently inverting the PSL bottle several times and then immersing it in a low-power sonic bath for 30 sec. The PSL bottle should not be shaken because shaking forms small bubbles that may introduce statistical artifacts. Clear the dropper tip of residue by dispensing two to three drops in a waste container. Then place one drop of PSL in 1 L of clean water. The final PSL-water suspension should be dispersed by inverting and sonicating. This suspension will probably need further dilution to provide a final aerosol concentration within the range of both the reference counter and the counter to be tested. The suspension can be diluted in the mixing chamber (see section 9.1.4).

NOTE: The monodisperse particles in the test aerosol should be thoroughly dry. A diffusion dryer or adequately dry dilution air should be used to dry the particles. Complete drying of the aerosol is important to prevent a water layer that would increase the size of the particles. The diffusion dryer uses silica gel desiccant to remove the moisture. The desiccant should either be new or freshly regenerated.

The particles in the aerosol should be dry enough that their size is not increased measurably by retained moisture. If the available air is not sufficiently dry, it should be dried, most conveniently by a greater than 2-sec contact with a desiccant in a drying tube. Silica gel with water content below 10% is generally adequate as a desiccant.

A radioactive-source aerosol neutralizer should be connected in line with the dryer to reduce the electrostatic charges on the dry PSL particles.

NOTE: Some electrostatic neutralizers may produce large numbers of ultra-fine particles ($<0.05 \mu\text{m}$), which will combine with the PSL aerosol.

9.1.3 Aerosol size separator

An aerosol size separator should be connected in line with the aerosol neutralizer to ensure that the generated aerosol is monodisperse. Many of the droplets generated by the atomizer contain no PSL particles. If the water were absolutely pure, empty droplets would evaporate completely, leaving no residue particles.