

Technical Paper 1 : Guidelines for Selecting an Optical Particle Counter (Edited)

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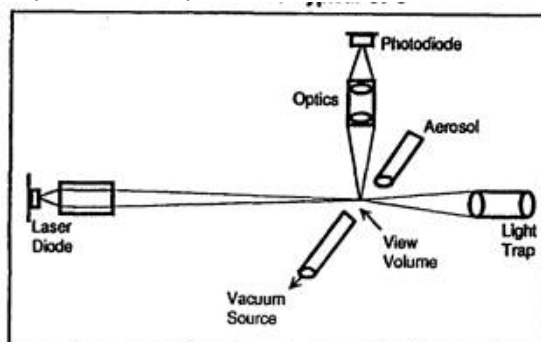
Manufacturers of particle counters tend to emphasize the areas of their best performance. For example, one manufacturer might stress sensitivity and counting efficiency and completely ignore long-term calibration stability (reproducibility). The purpose of this application note is to suggest guidelines for specifying all the important parameters: sensitivity, counting efficiency, accuracy, and reproducibility. This paper emphasizes the importance of instrument performance in a day-to-day working environment.

Selecting an optical particle counter (OPC) appears deceptively simple. Typically, the specification focuses on sensitivity, flow rate, size range and coincidence loss. Secondary requirements are number of channels, the sample/hold periods and alarm limits. There are some serious hazards when selecting an OPC on the basis of these parameters only. For example:

- Specifying sensitivity without considering the counting efficiency curve could mean good sensitivity (ability to sense small particles) but extremely poor resolution (ability to detect small differences in particle size). Poor resolution can cause large errors in the particle count
- Relying exclusively on sensitivity or counting efficiency measurements based on ideal (transparent and spherical) test particles can result in wrong answers when counting particles in the real world. These particles occur in a wide variety of shapes and refractive indices, causing large errors in particle sizing.
- Failure to recognize the difference between a "highly tuned" lab instrument (which can easily slip out of calibration with normal handling) and a "ruggedized" field instrument (which holds its calibration month after month) can be costly. Poor calibration stability causes sizing drift, nonrepeatability, random spikes and ultimately, loss of user confidence.

OPC Theory

A light source (typically a plasma laser or laser diode) is collimated to illuminate a sample volume of aerosol flowing out of a nozzle. As shown below, a photodetector, off-axis from the light beam, measures the amount of light scattered from single particles by refraction, reflection and diffraction. Both the size and the number of particles are measured simultaneously. The size of the particle is deduced from the intensity of the scattered light. Although this report focuses on aerosol OPCs, the guidelines presented are equally applicable to liquid counters. In the case of a liquid OPC the fluid is constrained to a channel inside a transparent (e.g. quartz) cell. The curve below shows the relationship between particle size and the amount of scattered light intensity when testing a typical OPC with monodisperse latex spheres



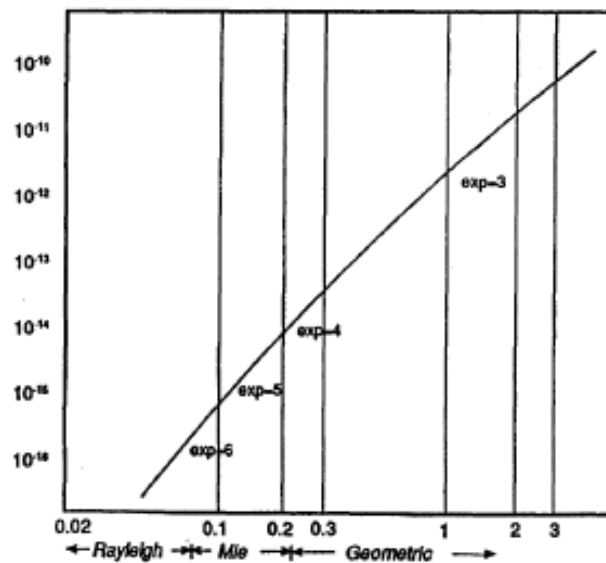
**Pictorial View of a Typical
OPC**

In the Rayleigh region, where particles are smaller than the light wavelength, light is scattered equally in all directions (isotropically) from the particle. Its intensity varies as a function of the 6th power of particle size in this region ($\text{exp} = 6$). In the Mie region, where particles are nearly the same size as the light wavelength, the light pattern surrounding the particle becomes scalloped. The forward lobe (pointing in the same direction as the laser beam) becomes larger as the particle size increases. The exponential relationship changes inversely with particle size. In the Geometric region where particles are much larger than a wavelength, classical optical theory takes over. Light scattering from a particle can be calculated from the physical effects of diffraction, reflection, refraction, and absorption. At Pacific Scientific Instruments, we prefer to address openly the topic of counting real world particles. This information will enable the customer to use the OPC as a most effective tool in the cleanroom.

Definition of Terms

In this section the important parameters are defined in terms of measurements based on PSL spheres. Next, these parameters are examined for comparison to actual particles found in the cleanroom. For convenience, OPC measurements are based on the introduction of aerosol with suspended polystyrene latex (PSL) particles of highly monodispersed sizes over the range of approximately 0.1 to 3 microns. The one-sigma dispersion is typically $\pm 1\%$. If we introduce PSL particles much larger than the specified lower detection limit, say 0.5 microns, the readings will be dispersed about a center value, as shown in the following bell-shaped curve. Accuracy is the difference between the true value and the center value of the interval.

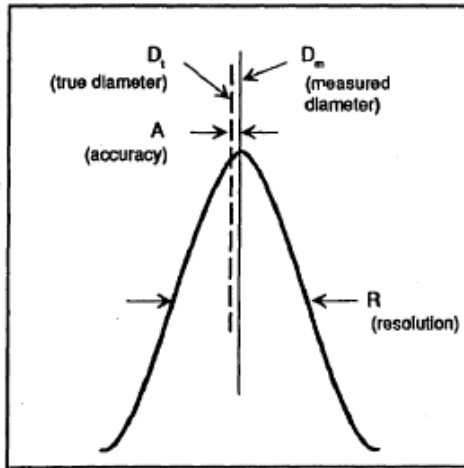
Accuracy - the "correctness" of the size measurement; expressed as a percentage: $A = (D_M - D_1) \times 100\% / D_1$ where D_M is the measured diameter and D_1 is the true diameter (see curve below)



Energy (Joules)

Number of Particles

Accuracy/Resolution (Bell) Curve



Particle Size vs. Energy Curve

An Inexact Science

OPC manufacturers generally specify sensitivity and counting efficiency on the basis of ideal test particles that are transparent and spherical. Most often, polystyrene latex (PSL) spheres, with a refractive index of about 1.59, are used for testing. In the particle counting industry there is a tendency to emphasize PSL sphere sensitivity and skirt the issue of OPC sizing accuracy and sensitivity with particles found in the real world. Unfortunately, real world particles come in a wide variety of shapes and refractive indices, leading to a significant degradation of sensitivity, resolution and accuracy. Sizing real world particles is an inexact science.

Particle Diameter

Resolution - the smallest detectable particle size difference. It is the ratio of the standard deviation (σ) to the diameter (D) expressed as a percentage:

$$\% \text{ Res} = \sigma \times 100\% / D$$

Resolution is a function of the width of the bellshaped curve. It is also referred to as "coefficient of variation, relative precision and relative standard deviation".

Precision - the standard deviation (d) of repeated measurements of the same size monodispersed spheres:

where: D_1 = the i -th measurement of particle diameter (arithmetic mean of N measures)
 N = total number of measurements

Reproducibility (also called repeatability and calibration stability) - the extent to which an OPC will give the same sizing and counting response to the same diameter PSL spheres over a long period of use.

Sensitivity - the smallest size particle an OPC can detect at a specified counting efficiency, e.g., 0.3 micron at 50% counting efficiency.

Counting efficiency - the detected particle concentration divided by the true concentration (as measured by a hypothetically perfect instrument). This curve provides useful information regarding the sensitivity and resolution of the instrument.

False count rate - the counts per unit volume using perfectly filtered air at a specified flowrate.

Signal - the magnitude of the sensed scattered light produced only by the passing of a particle through the view volume. Size is deduced from the signal magnitude. Noise is the opposite of signal in that it is produced by anything but a particle in the view volume. A high signal-to-ratio implies low false count rate.

Counting Efficiency, Sensitivity and Resolution

As an aid to arriving at a definition of counting efficiency, let us assume the presence of an ideal reference particle counter. See block diagram on next page. This counter can "see" every particle passing through the view volume to a diameter much lower than the lowest detection limit of the UPC under test. Typically, this instrument is a condensation nucleus counter (CNC) or an OPC with a verified counting efficiency of 100% at the lower detection limit of the OPC under test. However, a CNC only counts particles above a given size corresponding to a preset threshold (e.g. 0.01 micron); it cannot size particles. A reference CNC must be used with an electrostatic classifier to analyze particles by controlled deflection in an electrostatic field.

An aerosol carrying monodispersed PSL spheres is generated by the atomizer. The aerosol is mixed with filtered air in the mixing chamber. The OPC under test and the reference counter simultaneously sample the monodispersed spheres at the same concentration. As smaller and smaller monodispersed spheres are introduced, there is a point where the OPC under test fails to detect all the particles that the reference instrument is sensing. Further reduction in particle size results in the eventual loss of particle detection.

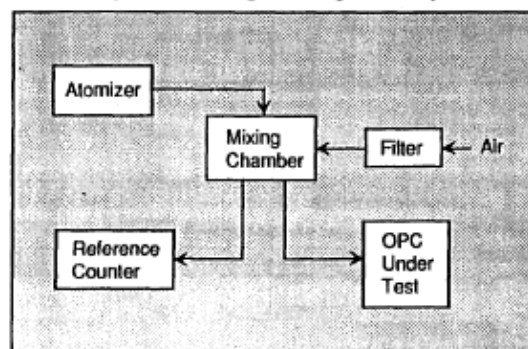
Counting efficiency is expressed as follows:

$$CE = N_m/N, \times 100\%$$

where N_m is the measured concentration and N_1 is the true concentration as measured by the reference instrument.

$$\sigma = \sqrt{\frac{\sum_{i=1}^N (D_i - \bar{D})^2}{N-1}}$$

$$\bar{D} = \frac{1}{N} \sum_{i=1}^N D_i$$



Setup for Measuring Counting Efficiency

In all cases, the threshold of the counter's detector is set to sense those monodisperse particles which fall in the upper half of the bell curve (those particles to the right of the intersection point of the three curves shown below). Smaller particles in the lower half of the curve (those to the left of the intersection point) are intentionally not sensed or counted.

Curve A represents the hypothetical case where the PSL spheres are ideal (exactly the same size with no dispersion) and the particles are sized perfectly by the OPC (also with no dispersion). Here, the 0, 50 and 100% efficiency points lie on the same vertical line. In this case the bell shaped curve is simply a straight line. Unfortunately, particle counters do not exhibit such steep function efficiency curves.

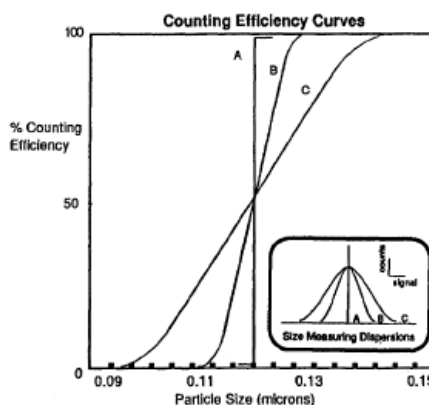
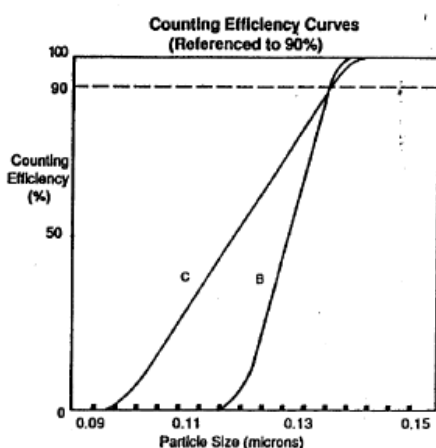
Curve B, exhibiting a relatively steep slope, is typical of a counter with good resolution, whereas curve C is representative of a counter with poor resolution. Size dispersion is much smaller in the case of the superior instrument (see curves in figure inset below).

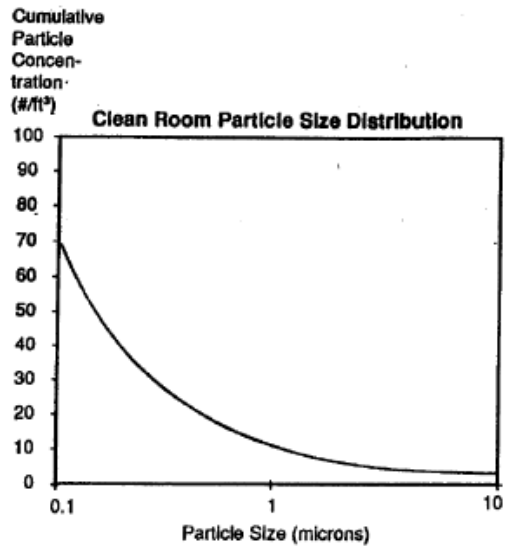
Let's examine the elements that determine the slope in practice. The largest contributor to poor resolution is the lack of uniformity of light intensity across the view volume. With any optical system, it is difficult to collimate a light beam down to a small area to achieve good sensitivity and, at the same time, maintain perfectly uniform intensity across this area. Any non-uniformity causes a discrepancy between sizing a particle passing right through the middle of the view volume and one that passes through one edge of the volume (being mistaken for a smaller particle). Variations in flow rate also contribute to wider dispersions resulting in degraded resolution. Other contributors, such as photodetector and amplifier stabilities are usually negligible in the typical particle counter.

If the counter thresholds corresponding to curves B and C had been set to anything but 50%, the particle counts between the two instruments would be in total disagreement. Only at 50% counting efficiency would two instruments with different resolutions count exactly half the monodispersed particles introduced.

If, for example, the thresholds were set for 90% counting efficiency at 0.12 microns as shown in the figure below, the curve C counter would outcount the curve B counter by a wide margin.

A quick and easy way to assess resolution is to compare the 50 and 100% points. In a typical Met One 0.1 micron counter this difference is about 0.015 microns, corresponding to a resolution of about 5%. This represents good resolution in the OPC industry. A more effective approach is to obtain (or generate) the counting efficiency curve for the instrument in question and determine the size spread between the 10 and 90% points. This provides a solid basis for specifying and comparing OPCS. In a typical cleanroom, the distribution of particles vs. particle size follows a power law function as shown in the next curve. Given this distribution, theory predicts that a poor resolution instrument will actually out-count one with good resolution at sizes below the threshold settings. Multichannel counters have a number of threshold settings, each set for 50% counting efficiency at the designated size. Note in the following set of curves the change in slope (and the apparent degradation of resolution) as particle size increases. This is due to the change in power-law exponent of the intensity of scattered light versus particle size.





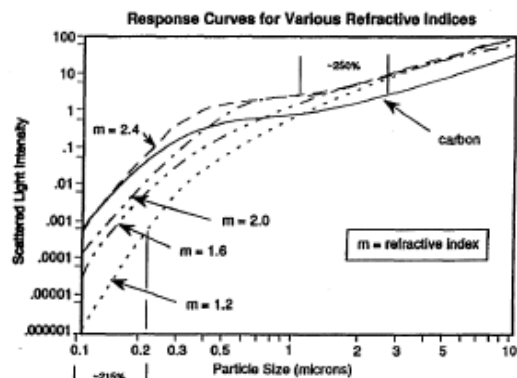
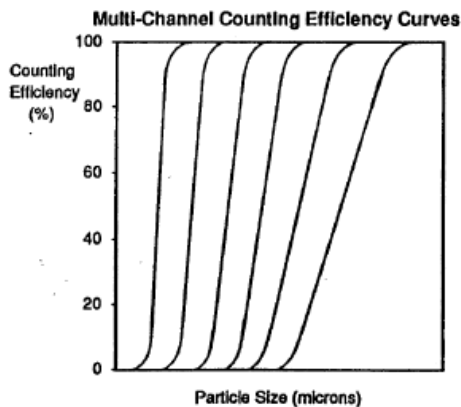
In setting the threshold at the lower sensitivity level, the signal level must be above the noise level. If, in order to stretch sensitivity, the threshold is set too close to the noise level, the false count rate (zero particles) will increase. To avoid this, always make sure that sensitivity and counting efficiency are accompanied by a minimum acceptable false count specification.

Particles in the Real World

Measurements with ideal (PSL) spheres provide us with a powerful tool for assessing the sensitivity, accuracy, resolution and false count level of a counter. This calibration technique serves two purposes: 1) Gives comparative evaluations of a wide variety of counters, 2) provides a measure of how well a counter maintains its calibration (reproducibility).

Parameters such as accuracy, counting efficiency, and resolution are very important in the process of PSL-based measurements. For this reason a somewhat detailed study was given in the "Definition of Terms" section. However these parameters become meaningless when it comes to measuring real world particles. Particles found in the cleanroom have a wide variety of shapes and refractive indices. This leads to a significant degradation of sensitivity, accuracy and resolution.

In an article in the periodical, Microcontamination, (ref. no. 8) Stuart Hoenig makes the point that "At 0.1 microns, optical scattering....is strongly dependent on the particle's shape, color and electromagnetic characteristics" which reinforces the fact that significant error exists in sizing real world particles.



To be able to "see" all of the particles in the view volume, the volume must be totally illuminated. Such a sensor is called "volumetric"; one that is in line with the flow and sees a sample of the aerosol is termed "in-situ". Some of the most sensitive counters available detect, for example, only 80% of the particles introduced and the curves are "normalized" to the 100% point when constructing the counting efficiency curves. All counting efficiency curves published by MET One reflect the true count. This fact must be considered when ordering a counter.

Some elements commonly found in contaminants, the by-products of processes like ionic etching and vacuum deposition, are:

aluminum copper nickel
boron fluoride phosphorus
calcium gold potassium
carbon iron sodium
chlorine lithium sulfur
chromium manganese tin

These elements exhibit a wide range of refractive indices. Some are highly reflective while others absorb most of the incident light energy.

Particle sizing errors due to changes in refractive index encountered with particles from the elements listed above are shown in the curves below. This is a family of response curves for a 90° (off-axis), 24° collection angle, aerosol counter for spherical particles of different diameters (0.1 to 10 μ) and refractive indices (in = 1.2 to 2.4). These curves, derived from reference no. 11, are for nonabsorbing particles only (except carbon). As indicated, a 0.2 μ particle at in = 1.2, scatters about the same amount of light to the detector as a 0.1 μ particle at m = 2.4. This represents an accuracy of about 215% according to the definition of terms presented earlier. This number could change even more dramatically if the effects of absorption and differences in particle shape are considered.

In the case of carbon particles, which are highly absorptive, the deviation in respect to the in = 2.4 curve increases rapidly for particles above 0.2 μ, with the error approaching 250% above 1 μ (see curves). The foregoing verifies our earlier statement regarding the inexactness of sizing real world particles with an optical particle counter.

For practical purposes, OPCs are calibrated with ideal particles having a refractive index between 1.5 and 1.6. The size measured by the OPC is then an "equivalent PSL diameter" or an "equivalent DOP diameter", depending on the calibrating aerosol used.

The magnitude of error in sizing real world particles with an OPC would appear to be a discouragement in attempting to set up an effective cleanroom quality assurance program. Actually the outlook is not as bleak as might be expected at first glance. It turns out that the optical particle counter can function as a surprisingly effective tool in the cleanroom if used in a protocol that has been evolved by cleanroom professionals over the years.

How To Use An OPC In A Cleanroom

The OPC has two basic functions in the cleanroom. The first is to certify the cleanroom to meet standards established by FED-STD-209E or the ISO 14644 Part 1. The second function is to support a quality maintenance program in the cleanroom.

Cleanroom Certification

Federal Standard 209E and the ISO 14644 Part 1 establishes classes of air cleanliness for airborne particulate levels in cleanrooms. It also prescribes methods for class verification and monitoring of air cleanliness. For classification see table on the next page.

Cleanliness Classification	No of Particles per m ³					
	0.1µm	0.2µm	0.3µm	0.5µm	1.0µm	5.0µm
ISO Class 1 -	10 -	2 -				
ISO Class 2 -	100 -	24 -	10 -	4 -		
ISO Class 3 1	1 000 1 240	237 265	102 106	35 35.3	8 -	- -
ISO Class 4 10	10 000 12 400	2 370 2 650	1 020 1 060	352 353	83 -	- -
ISO Class 5 100	100 000 -	23 700 26 500	10 200 10 600	3 520 3 530	832 -	29 -
ISO Class 6 1 000	1 000 000 -	237 000 -	102 000 -	35 200 35 300	8 320 -	293 247
ISO Class 7 10 000				352 000 353 000	83 200 -	2 930 2 470
ISO Class 8 100 000				3 520 000 3 530 000	832 000 -	29 300 24 700
ISO Class 9 -				35 200 000 -	8 320 000 -	293 000 -

Extract from FS 209E and ISO 14644 Part 1

Air Quality Maintenance

Monitoring air quality with the particle counter to support an air quality maintenance program is more involved than cleanroom certification. The goal is to eliminate "killer defect" particles that can destroy product yield. Particles whose size is about one-tenth (or larger) of the minimum line width on a semiconductor wafer fall into this category.

Here, the user must be more aware of the magnitude of particle sizing errors in the real world, due to variations in shape and refractive index. Instead of trying to size particles precisely, establish particle concentration reference levels and correlate these levels with product yields. At this point, the exact sensitivity (whether it was 0.1 or 0.15 µ), as measured earlier with PSL spheres, becomes insignificant. What is important is the ability of the counter to hold its calibration over the long term.

During a particle-shedding event, particles are generated in a wide distribution of sizes. If there are 0.1 micron particles in the sample, you can be sure there will also be 0.2 micron particles present. Considering this factor, the second decimal place in measuring micron sensitivity with PSL spheres should not be overemphasized. Calibration stability is more important.

Step I in a typical cleanroom monitoring procedure is to- establish a reliable zero count (false count level) using a quality filter on the counter's aerosol inlet port. Repeated checks that show -consistently low counts will give you confidence that you have .attained an acceptable level.

Step 2 is to establish a particle concentration baseline for each station to be monitored in the cleanroom. Even though the exact size of particles counted is unknown because of differences in shape and refractive indices, a reference level can be created. Familiarize yourself with the levels associated with the various processes and determine empirically what level is acceptable and what levels begin to reduce the yield.

Step 3 is to recalibrate with PSL spheres from time to time until you acquire confidence in the counter's ability to hold its calibration in the working environment.

Summarizing, instead of an absolute particle sizer and counter, the OPC can be used more effectively as an early warning trend indicator or burst detector. This will allow you to shut down a process if the concentration level exceeds a preset threshold. Thus the OPC can function more as a process tool than an environmental tool in your cleanroom quality assurance program.

Stability Considerations

Stability in the working environment must be considered at least as important as sensitivity. It is extremely important that the counter maintain its calibration over the long term, otherwise, particle concentration baselines become meaningless.

Typically, the wavelength of light used in OPCs ranges from about 0.63 to 0.83 micron. As particles become smaller than a wavelength, the amount of light they scatter into the detector collection optics drops off rapidly. Referring to the Particle Size vs. Energy Curve illustrated earlier, the detected light energy falls off exponentially with decreasing particle size. At 0.3 micron the detected energy drops off as a function of about the 4th power of particle size; at 0.1 micron the detected light energy drops off as a function of about the 5th to 6th power of particle size.

For example, to upgrade the sensitivity of a counter from 0.2 to 0.1 micron requires about a 17-fold increase in light power focused into the view volume. To achieve a sensitivity approaching 0.1 micron requires a well-designed laser/optical system with a narrow optical bandwidth (sometimes referred to as "high Q") in order to develop high light intensity in the view volume. Making the optical bandwidth too narrow (Q too high) in order to achieve high sensitivity can actually lead to calibration instability (in the presence of mild shock or vibration) with the attendant loss of sensitivity.

Each manufacturer is faced with making a trade-off between high sensitivity and reproducibility in the working environment. At MET ONE we put the emphasis on stability and shock-resistant reproducibility in a tough environment at the expense of a slight sacrifice in sensitivity.

If a manufacturer decides to make the trade-off favoring sensitivity at the expense of calibration stability, the result is a sensitive, "highly tuned" laboratory instrument that can easily slip out of calibration with normal use. Simply moving such an instrument from one bench to another can degrade its detection limit performance.

In this industry there is a tendency to tout OPC performance solely in terms of PSL sphere micron sensitivity at 50% counting efficiency (e.g. 0.10 micron @ 50% counting efficiency). In the world of particle counting in a working environment, sensitivity alone is not a meaningful number. It does not give you enough information to determine if you are dealing with a highly tuned laboratory instrument or a durable and reliable workhorse.

Considering the degradation in particle counter sensitivity when dealing with real world particles, MET ONE does not put undue emphasis on the second decimal place of the particle size sensitivity specification. Our position is very clear: a counter that maintains a long-term sensitivity of, for example, 0.12 microns (50% C.E.) in a rough environment is far superior to an instrument that begins with a sensitivity of 0.10 microns (50% C.E.) and slips out of calibration the first time it is moved to a new location.

Summary

- 1) Specify sensitivity in terms of counting efficiency. To assess the inherent resolution, examine at least three points on the counting efficiency curve.
- 2) Make sure the manufacturer has not set the detector too close to the noise level in order to stretch sensitivity beyond allowable limits. To accomplish this, always accompany the sensitivity/counting efficiency specification with a minimum acceptable false count level.
- 3) Be aware of the degradation of sensitivity, resolution, and accuracy when counting real world particles. These particles appear in all shapes and refractive indices. Accordingly, don't put all the emphasis on the PSL sphere sensitivity.
- 4) Specify that the counter must "see" 100% of the particles passing through the counter at the 100% point (and above) on the counting efficiency curve. Some available counters, although extremely sensitive, "see" only about 80% of the particles in the sampled aerosol at the 100% point on the counting efficiency curve.

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NB : This particular paper has been edited by Cesstech in Dec 2001 with respect to the latest version of the FS 209E and the new ISO 14644 Part 1