Technical Paper 3
Verifying Particle Counter Calibration: Size Verification

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Abstract
While water particle counter manufacturers currently perform both size and count calibrations during in-house testing, only size calibration is readily verifiable in the field.

Initially, particle counter calibration seems a simple matter of injecting a size and count standard fluid and adjusting the sensor counts to match. However, differing technologies factors such as electrical zone, light scatter, light blocking or diffraction can result in drastically different field counts, making calibration more complex. Even using identical size calibration spheres from different manufacturers can produce markedly different results.

Current sensor calibration standards don't account for these technical variables, though manufacturers, users, regulators and researchers are rapidly developing both size and count calibration standards for the potable water industry. Manufacturers are also developing better field verification equipment that will verify count calibration in the field. (A future paper will describe these developments.)

While calibrating particle counters with the same standard does not ensure all particle counters will count the same fluid the same way, *field verification* of calibration does ensure the sensor is stable and functioning to the manufacturer's standards.

This paper describes a size and operational verification procedure that assures basic sensor operation for field deployed particle counters.

The Study
The potable and wastewater industries increasingly use particle counters to monitor filtration efficiency and process control. Particle counters provide the necessary single particle visibility required for optimizing the filtration process in order to combat growing contamination of potent and disinfection-resistant microorganisms in drinking water.

Until recently, turbidimeters were used to monitor water quality. While optimum turbidity measured less than 0.1 NTU, these instruments were not necessarily effective. For example, particle concentration of even very clean water can differ remarkably while yielding the same turbidity reading.

Explaining water sensor design
The primary particle sensor design for today's potable water and wastewater applications is the light blocking, laser diode sensor. This sensor is extraordinarily rugged and reliable. It's laser diode lasts up to tens of thousands of hours (years) and is electronically controlled to produce a constant light output over that full lifetime.

Earlier water sensor models used incandescent lamps and multiple lenses, which were subject to misalignment with filament sagging and lens shifting. The newer sensor design uses fewer lenses, which simplifies mounting and reduces the complexity of alignment for increased stability and reliability. As a result, the verification procedure described in this paper assures proper sensor/counter operation to a high degree of confidence.

Describing size and operational verification procedure
Proper verification of a sensor's size response in the field is important. It assures that the sensor is operational and closely matches the manufacturer's original calibration.

Size verification is both conceptually and operationally simple. With the sensor in normal operation, and with few background counts in the size ranges of interest, a size calibration fluid is introduced into the sensor inlet stream and the resultant particle counts are recorded. The location and magnitude of these counts demonstrate if the sensor is assigning the counts to the proper size bins.

Count validation is much harder to perform in the field and will require further development of calibration materials and test equipment.

Introducing the test procedure
Test procedures for verifying size calibration consist of four basic steps:

1. Establish normal particle sensor operation as defined by the manufacturer for:
2. online sensors
3. pressurized batch samplers
4. grab samplers
5. Record the 'clean' background particle count.
6. Introduce the size calibration challenge fluid into the sensor.
7. Record and analyze the results.

Traditionally, the most difficult step is number 3, introducing the size calibration challenge fluid. However, recent accessories provide simple and effective means for introducing calibration spheres directly to the normal flow. Each step is discussed below.

Establishing normal particle sensor operation

Normal particle sensor operation using online sensors
For online sensors, a simple 'tee' fitting can be inserted into the sensor inlet line. Then, using a syringe, the size calibration challenge fluid is injected into the sensor.

Many on-line systems use a counting duty cycle of perhaps only eight to 10 percent. Therefore, synchronizing the fluid injection with the next counting cycle is vital. If the counting period cannot be determined, then sufficient volumes of sample fluid must be injected for the full count cycle, which could last up to five minutes.

Note that with some care and practice an appropriate particle 'burst' of both magnitude and duration for the particle counter is produced. As mentioned above, simple timed released particle accessories are now available to facilitate the injection of particles.

Establishing standards using pressurized batch samplers
Particle counters can sample fluids directly from beakers and bottles using a pressure chamber to force the sample fluid from the bottle into a sample tube and through the sensor.

Since the system is designed for batch sampling, it's easy to introduce the sample fluid into the sensor. However, using pressurized systems in the water treatment laboratory is relatively uncommon because of its large size and AC power requirements. Pressurized systems are also expensive because the sampler requires an external counter and a pump.

Establishing standards using grab samplers
Grab samplers use a slight suction to pull the fluid sample through the particle sensor. This system is completely self-contained because it's often compact, portable, battery operated and includes both the particle sensor and counter.

The grab sampler system is easy to use for size verification, so it is widely used in the potable water industry. Even though it is more expensive than an individual sensor, grab samplers are much less expensive than batch systems. In addition, the grab sampler can introduce the size challenge fluid to an online sensor.

All the accompanying results were obtained using a grab sampler.

Recording clean background counts
The ‘zero count’ test is one of the most basic tests used to determine the effectiveness of particle counters. The idea is for all particle sensors to count nearly zero particles per ml when there are no particles present. To achieve a zero count, either filter or stop the sensor flow.

The straightforward way to determine zero count is to filter the sensor flow path and remove all particles large enough to be seen by the sensor. However, filtering requires selecting and purchasing an appropriate filter, disturbs the plumbing connections to the sensors and requires higher pressure to overcome filter head loss.

For these reasons, stopping the sensor flow is often the easiest and most satisfactory way to check zero count. If the counts are zero, or near zero, with the flow stopped, this means that the sensor is not counting noise pulses as particles.

Introducing size calibration fluid
The size calibration fluid, in this case monodisphere Polystyrene Latex (PSL) spheres, should be introduced to the sensor so that it appears as a normal influent. It should not produce out-of-tolerance sensor flow or introduce undue turbulence, bubbles or excessive background counts.

Online sensors
For online sensors still connected to filter effluents, the sample fluid should be introduced when the counter is actually sampling. A simple approach is to inject a moderately concentrated size calibration fluid into the sensor inlet line using a ‘tee’ fitting and a syringe. The injection should be relatively uniform, extending over the sample period if known, or over a full sample cycle.

If the actual particle concentration of the size challenge fluid is known, then the approximate sensor count can be controlled by the injection duration. For example, if a 2,000 particle per ml concentration sizing fluid is used with a 100 ml/min sensor flow, over a 24-second sample period (i.e., 40 ml sample), then choosing an arbitrary 200 particle per ml count of size challenge particles requires a 10 percent concentration.

Sample volume = 40 ml (during 24-second sample period)
Syringe injection volume = 4 ml (during 24second sample period)

Choosing a pre- and post-sample time buffer zone of about 18 seconds each adds another 6 ml of syringe volume. So, a 10 ml syringe steadily injected over 60 seconds would yield about 200 particles per ml. Injecting the size challenge fluid at 10 percent of the normal sample volume will not likely disturb the sensor flow appreciably, or introduce extraneous bubbles or contamination counts. However, due to variables cited above, actual counts will not agree better than 10 to 20 percent, even if the original particle concentration of the size challenge fluid is perfectly known.

Again, for size verification, the actual particle concentration is not significant, as long as it lies between the background counts and well below the coincidence limit of the sensors.

Batch sampler sensors
Since the sample can be run at any time, simply mixing the size challenge with very clean water can achieve the desired particle concentration.

Grab sampler sensors
Like batch samplers, the particle size challenge can simply be added to the sample container of clean water.

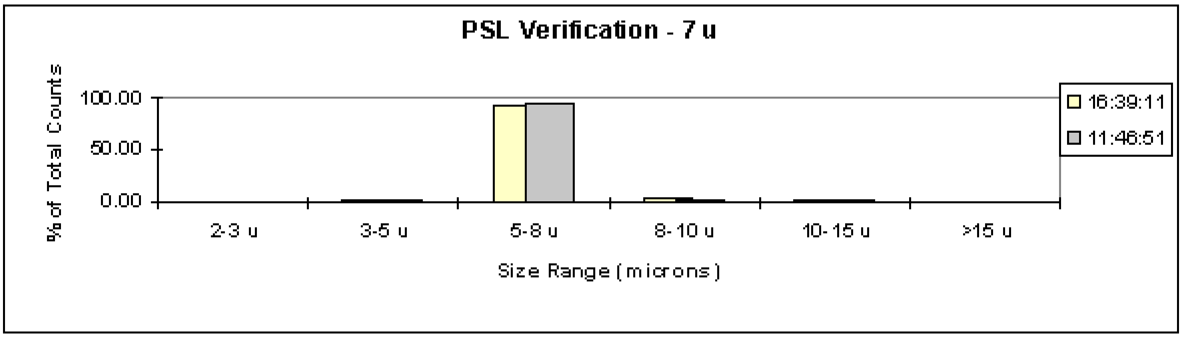
Looking at test results
The following graphs show the results of size verification testing.

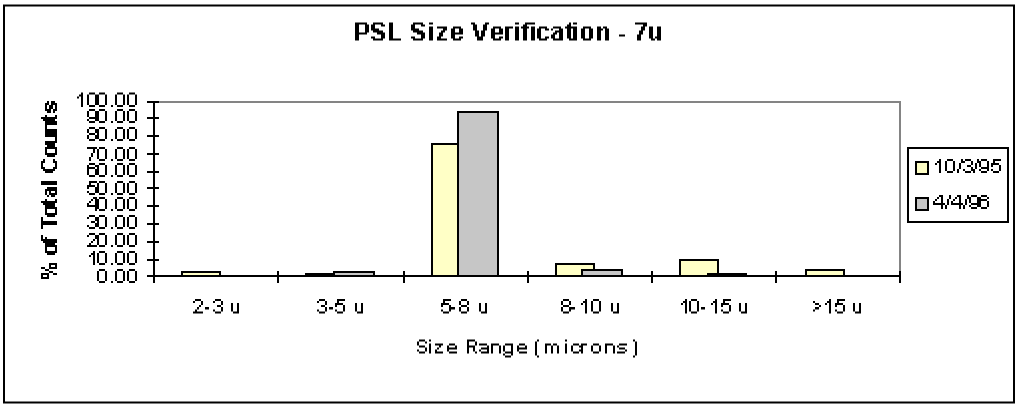
Polystyrene latex (PSL) spherical calibration particles have very tight size tolerances. They can show when counting thresholds drift off by noting the ratio of particle counts in adjacent bins.

For example, using a 10 micron particle size (9.975 +/- 0.061 microns) and noting the relative counts in the 8 to 10 and 10 to 15 micron range, the count ratio of these two size bins can be periodically recorded and compared to detect any drift in sensor calibration.

Note the test particle sizes used are not precisely the same as the particle counter bin sizes. Since PSL particles can't be made arbitrarily close to their nominal sizes, a 10-micron particle standard may actually be labeled as 9.975 microns.

The particle counter calibration curve is generated by interpolating a number of these 'close to nominal' PSL particles to generate a chart for the 'even' values that are usually programmed. This is one reason why calibration particles may not always split their counts equally between adjacent size bins.





Graphs 1A and 1B (particle centered in size bin)

As expected, most counts from a nominal 7-micron PSL calibration particle appear in the 5 to 8 micron size bin. The question is, why are there significant counts in the 8 to 10, 10 to 15, and >15 micron bins as shown in the 1995 data of Graph 1A?

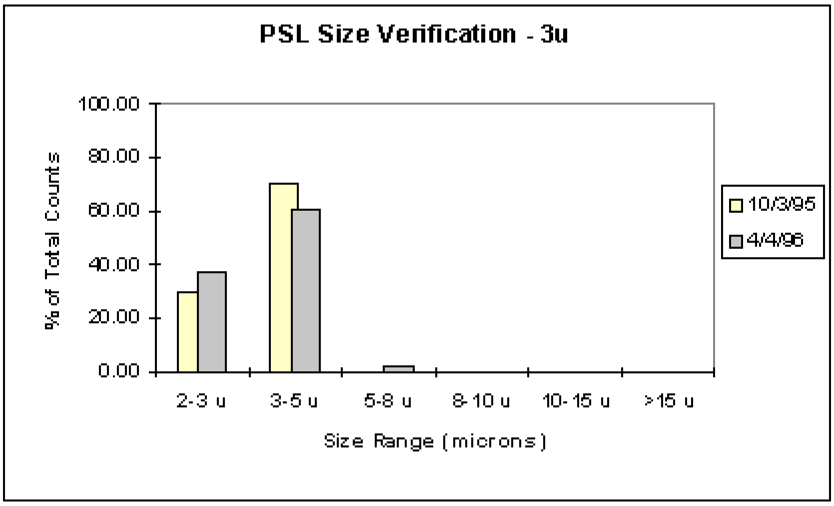
The answer is that these are residual counts produced by 10 micron particles measured just prior to running the seven micron counts. The 1996 data in Graph 1A shows more typical results when sufficient cleanup time has been allotted.

When checking a series of sizes, it is beneficial to start with the largest particles and work down. The larger particles often have lower concentrations and clean up quicker than the smaller particles.

It is also important to record the 'background' counts just prior to introducing the calibration spheres so any discrepancies in binning can be evaluated for validity.

The use of high-test particle counts, when compared to background counts, minimizes count skewness. The 5 to 8µ size bin counts were 79 particles per ml in 1995 and 1,930 particles per ml in 1996.

Graph 1B shows a much closer comparison of counts taken about four hours apart on the same day. These variations may well represent the minimum detectable deviation in counts.



Graph 2 (particle at edge of size bin)

Graph 2 shows the effects of 'splitting' the 3.004-micron counts between adjacent size bins. Note the proportion of counts in the 3 to 5 range is greater than in the 2 to 3 range.

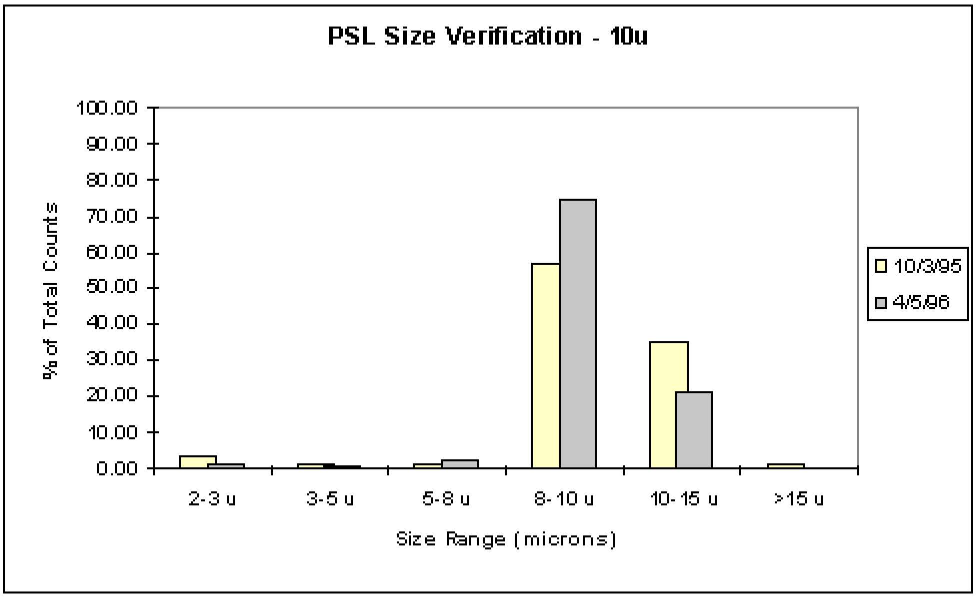
This unequal distribution could be due to the use of particles whose mean size (3.004) is just slightly larger than the bin lower limit (3µ). But more likely, the particle counter size threshold is slightly misadjusted. Mere millivolt can produce this kind of count skewness across a size boundary.

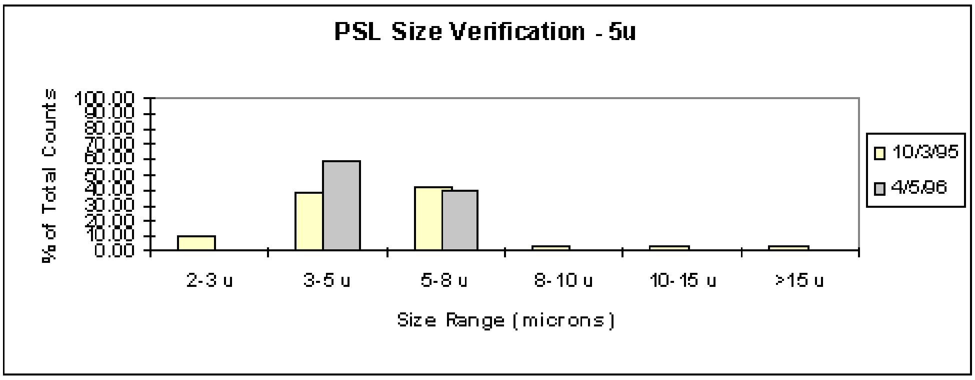
After a year's time, the calibration appears to be evening up, with the 3 to 5 micron counts decreasing and the 2 to 3 micron counts increasing. This could be deceptive, since the same bottle of particles was not used for both tests.

The same manufacturer's part number was used to make both challenge fluids, but the actual challenge fluid bottles were different. To produce a true test of size shifting, the very same bottle of challenge fluid particles should be used each time a bin split test is performed.

The proper mixing and storage of the PSL test spheres is vital to their accuracy and longevity. Both particle clumping and microorganism contamination can have deleterious effects on size verification.

These results indicate the sensor is still close to the initial factory calibration for this size threshold.





Graphs 3 and 4

Similarly, graphs 3 and 4 show the 5µ (5.010µ) and the 10µ (9.975µ) size verification results.

While repeated tests for each calibrated sensor size would rigorously affirm the original calibration, often only a few sizes are of interest and importance.

Of course, the smallest sensor size threshold cannot be used to detect bin shifts because there is no adjacent valid lower size bin due to electrical noise. So, in this case, it would be useless to purchase 2 micron particles to check the 2 micron minimal size bin.

The sizes that absolutely should be checked are those that define the endpoints of any important size range, such as 3 to 5 microns for Cryptosporidium and/or 5 to 15 microns for Giardia.

For more information
The author is available for further discussion and details of the field calibration verification method at Pacific Scientific Instruments by phone (541) 472-6500.