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Sizing Accuracy of a LiQuilaz-S Particle Counter

Abstract

The need to verify the performance of a volumetric Optical Particle Counter (OPC) is necessary in many applications. This paper describes the correct method to verify the performance of a LiQuilaz-S series OPC by calculating the sizing accuracy. In order to calculate the sizing accuracy, you must precisely collect particle count data and know the instrument's resolution. These instructions apply to collecting data in Facility Net software using LiQuilaz-S series volumetric spectrometers. The sizing calculator referred to is available on the Particle Measuring Systems website.

Introduction

Accurate sizing and counting have implications in qualifying chemical delivery systems, accepting incoming chemicals, or qualifying incoming parts. Therefore, the need for OPCs to match and a way to qualify their performance is thus very important to many users.

To verify an OPC, one must use appropriate methods to ensure time and or money is not wasted. These methods include good laboratory technique, proper equipment set up, data collection, and using the sizing calculator, as described in this paper.

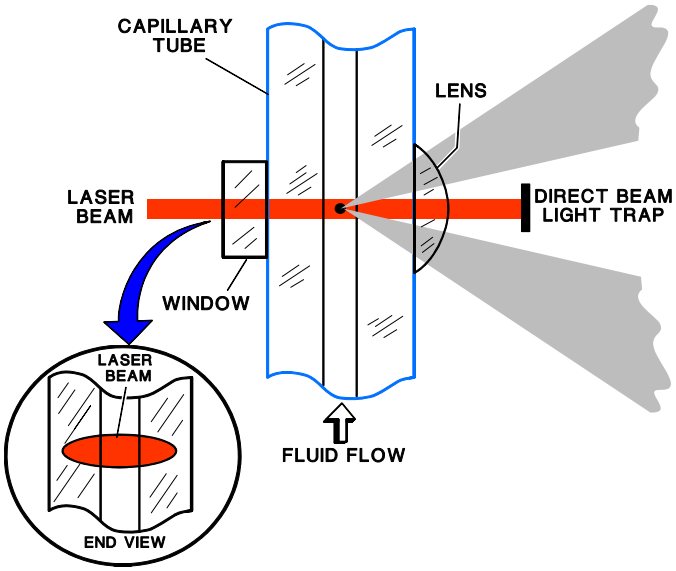
Particle Measuring Systems uses sizing accuracy to verify the performance of our LiQuilaz-S OPCs.

LiQuilaz-S OPC Design

The LiQuilaz is a volumetric instrument that counts 100% of the particles passing through the sensor. 100% of the fluid passing through the capillary is analyzed because the capillary is fully illuminated by the laser beam, as shown in Picture 1. Therefore, the LiQuilaz will always count 100% of the particles eliminating the need to perform count standard tests.

Counting Accuracy refers to the instruments ability to count the correct concentration of particles in a solution.

Sizing Accuracy refers to the instruments ability to correctly determine the size of a Polystyrene Latex Sphere (PSL).



Picture 1: Volumetric OPC Capillary Illumination

If, for example, there is something wrong or the LiQuilaz is misaligned then the LiQuilaz particle counter would not size accurately; therefore as long as the sensor is sizing accurately, it is counting accurately.

Size Standards

Polystyrene Latex Spheres (PSLs) are industry recognized size standards that are NIST traceable and used for instrument calibration. They are readily available from Duke Scientific in various sizes. Particle Measuring Systems uses size standards from Duke Scientific Corporation for calibration and verification. Table 1 lists the recommended PSL sizes for verification.

Picture 2: Polystyrene Latex Spheres

Test Setup

To properly verify the performance of an OPC you must correctly design the test and set up the test equipment. Picture 3 shows a typical particle injection system. Laboratory technique is critical. Individuals performing the sizing accuracy test must have experience measuring particles in ultra-pure liquids, preparing laboratory glassware, particle counter operation, and possess overall good laboratory techniques and practices. The following requirements must be met to determine the sizing accuracy of the OPC.

- ☐ The OPC should be installed online with a particle injection system (i.e. PMS VSPI) capable of injecting PSLs into the water. Offline sampling requires careful serial dilutions of the size standard to achieve the appropriate concentration of particles for the sensor.
- ☐ The flow rate must be precisely set to the calibrated flow rate using a flow controller calibrated with a stop watch and graduated cylinder. Typical flow rates are 20, 50, or 80 mL/min. The flow rate should be verified for each instrument.
- ☐ The DI water being used in the test must be below 100 cts/mL for the most sensitive size channel of the OPC under test.
- ☐ The total concentration of PSLs counted by the OPC should be 3000 +/- 500 particles/mL.
- ☐ The DC light must be within specification (S02 <0.5V, S03/S05 <0.05V).
- ☐ If multiple units are under test, they are connected properly in parallel.

Picture 3: Particle Injection System

Data Collection

Data collection is equally important in properly verifying the performance of the OPC. Particle size channels must be defined with the exact particle size of interest. The exact particle size can be found on the PSL bottle as shown in Picture 2. The exact particle size is listed as Diam: 0.350 microns (µm). The exact size must be used, not the nominal size.

The first channel will be the 1st channel of the particle counter. Next, enter the particle size channel that is the exact particle size on the PSL bottle. Enter an equal number of channels above and below the particle of interest using the channel spacing listed in table 1. Channel spacing is defined under the instrument configuration in Facility Net software.

Table 1: Channel Spacing for Sizing Calculator

S02		S03		S05	
Nominal Size	**Channel Spacing**	**Nominal Size**	**Channel Spacing**	**Nominal Size**	**Channel Spacing**
0.35	0.02	0.6	0.05	2.0	0.1
0.6	0.05	0.8	0.05	5.0	0.5
1.0	0.1	3.0	0.2	15.0	1.0
1.5	0.1				

The following example in Figure 1 shows proper channel configuration for a LiQuilaz S02 OPC using the nominal 0.35 micron PSL. The first channel of the LiQuilaz S02 is 0.2 microns. The exact PSL size is 0.350 microns. Channels are set up above and below 0.350 µm using 0.02 micron channel spacing. PSLs are injected showing a clearly defined peak around the particle size of 0.350 microns. You should notice with very clean water there is zero to relatively few particles in the smallest size channels.

Channel	Differential	Cumulative
0.200	10.20	3217.80
0.210	15.00	3207.60
0.230	11.40	3192.60
0.250	23.40	3181.20
0.270	55.50	3157.80
0.290	137.40	3102.30
0.310	447.90	2964.90
0.330	1380.00	2517.00
0.350	1029.90	1137.00
0.370	60.90	107.10
0.390	24.60	46.20
0.410	1.20	21.60
0.430	7.50	20.40
0.450	7.80	12.90
0.470	5.10	5.10

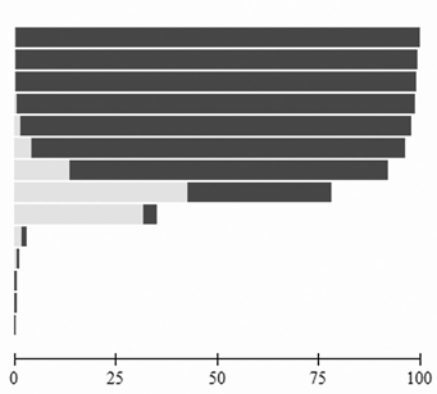


Figure 1: LiQuilaz 0.350 Micron PSL Data

It is critical that clean water is used during these tests. As one will see, collecting the particle count data requires summing the particles above and below the PSL size. If there is contamination in the samples, it will artificially increase the counts below the PSL size thus skewing the data towards a negative sizing error. Figure 2 shows high numbers of counts in the smallest particle channels due to contamination.

Channel	Differential	Cumulative
0.200	288.30	3507.00
0.210	406.20	3218.70
0.230	212.10	2812.50
0.250	214.20	2600.40
0.270	184.50	2386.20
0.290	228.60	2201.70
0.310	450.60	1973.10
0.330	687.00	1522.50
0.350	586.80	835.50
0.370	50.70	248.70
0.390	36.30	198.00
0.410	1.50	161.70
0.430	13.20	160.20
0.450	30.90	147.00
0.470	116.10	116.10

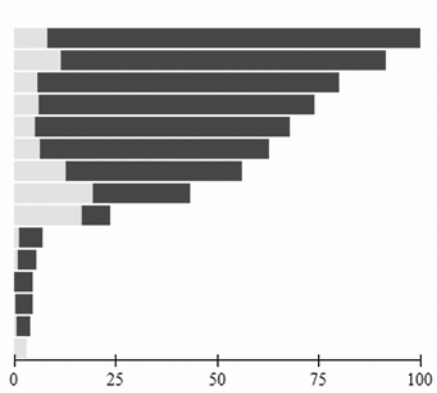


Figure 2: LiQuilaz 0.350 Micron PSL Data with Contamination

Particle Count Data and Sizing Accuracy

Next, particle count data must be collected and entered into the sizing calculator. Once data is entered into the sizing calculator, the sizing accuracy of the OPC can be determined. The Sizing Calculator requires the user to input the exact PSL size, the total number of counts (Total Particle Counts), and the number of counts above the PSL size (High Particle Counts). The calculator then calculates the sizing error. Instrument resolution is the historical average instrument resolution from newly calibrated OPCs.

Using the same example in Figure 1, you would enter 0.350 µm into the *Mean PSL* size cell of the Particle Calculator. Enter 3217.8 into the *Total Particle Count* cell and 1137.0 into the *High Particle Count* cell. The sizing calculator in spreadsheet 1 shows that this OPC has a sizing error of -1.88%.

ISO standards suggest that instruments of this type should have sizing errors of +/-10% or less [1]. If the sizing error is greater than 10% one should review their procedures. If it is determined that the sizing error is indeed greater than 10%, the LiQuilaz will require service and calibration by an authorized technician.

It is important to note the size and/or shape of the peak is not indicative of sizing accuracy, nor are the actual splits. *Splits* are commonly defined as the ratio of counts below and above the particle size of interest. The split in this example is 65% - 35% or there are 65% of the particles below 0.35 µm and 35% of the particles above 0.35 µm as noted in the sizing calculator in the *Split Ratio* cell.

Spreadsheet 1: Sizing Calculator

Instrument Model :	LiQuilaz S02
Instrument Serial Number:	
Date:	
Particle Sizing Accuracy Calculator	
Sizing accuracy test should be performed at a calibration particle size (error at time of calibration is near zero). The exact PSL mean size should be used. ALL CALCULATOR INPUTS ARE IN YELLOW CELLS. Enter the exact PSL size into the calculator. After performing the split test, enter the total number of the counts observed into the Total cell and the total number of counts observed above the particle size of interest into the High cell. The program calculates Relative Size Error.	
	Channel 0.35
Inputs	
Mean PSL particle size (EXACT)	0.350
Channel Resolution	5%
Total Particle Counts	3217.80
High Particle Counts	1137.00
Split Ratio (high size counts /total counts)	0.353
Outputs	
Standard Deviation	0.018
Cumulative proportion	0.65
Calculated PSL Size	0.343
Relative Size Error	**-1.88%**
Calculated Threshold Position	0.357
Counting Error has the same sign as the Relative Size Error	
A negative counting error means that the sensor is under-counting	
A positive counting error means that the sensor is over-counting	

Counting Error

After the sizing error of the OPC is known, you can calculate the actual counting error for a real world sample. Calculating the counting error assumes a normal ambient distribution of particles with a distribution of D^-3 (where D is the particle diameter) [2]. Continuing with our previous example, the counting error for total cumulative counts would be -5.8% as shown in spreadsheet 2. This means that in this example, the OPC is counting 5.8% less total cumulative particles 0.35 µm and greater than a perfectly calibrated OPC would be counting.

Spreadsheet 2: Counting Error

Counting Error Estimate						
Particle Counter	PSL Mean Size (microns)	Calculated PSL Size (microns)	PSD	Relative Size Error	Calculated Threshold (microns)	Counting Error for Ambient Fluids
LiQuilazS02	0.350	0.343	-3	-1.87%	0.36	-5.81%

Conclusion

Verifying the sizing accuracy of a LiQuilaz particle counter is useful to determine if the particle counter is working properly. If it is sizing correctly, then it is counting correctly and ultimately will provide the best instrument to instrument matching as is often required by users. The techniques and methodology for determining the sizing accuracy as described in this paper are the best methods to verify instrument performance. Particle Measuring Systems utilizes sizing accuracy to verify the performance of a LiQuilaz-S OPC.

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Roger Carlone is an Applications Engineer with Particle Measuring Systems. He has been with Particle Measuring Systems for seven years specializing in liquid and parts cleanliness applications. He has a Bachelors degree in Chemical Engineering from the University of Texas at Austin.

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