

Using Count Standards to Check Operation of a LiQuilaz[®] Particle Counter

Abstract

Verifying the operation of a volumetric Optical Particle Counter (OPC) is necessary in many applications. This paper describes the proper use of count standards to check the operation of a LiQuilaz[®]-S or E type OPC. The following instructions are for collecting data in SamplerSight software using a syringe sampler paired with a LiQuilaz-S or E type volumetric spectrometer. In order to fully verify the performance of an OPC, it is important to first check the sizing accuracy as described in the paper, [Sizing Accuracy](#)¹.

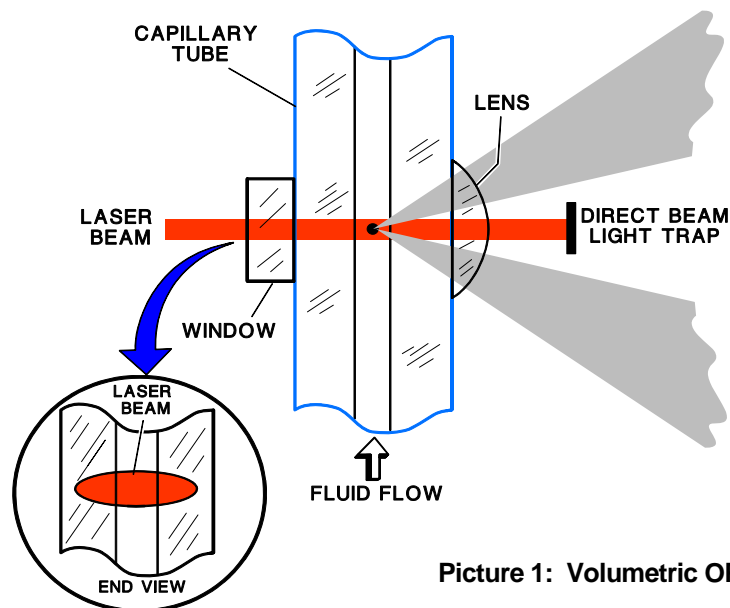
Introduction

Accurate particle sizing and counting have implications in qualifying chemical delivery systems, accepting incoming chemicals, or qualifying incoming parts. The need for particle counters to match and a way to qualify their performance is, therefore, significant.

To accurately and effectively check the operation of an OPC, you must use appropriate procedures; using a shortcut method will waste your time and money. Proper methods include good laboratory technique, proper equipment set up, and data collection. Count standards, when properly applied, may only be used to check the operation of a LiQuilaz particle counter, not verify its performance. Count standards provide insufficient data to calculate the sizing accuracy, thus its performance cannot be verified

Particle Measuring Systems uses sizing accuracy to verify the performance of a LiQuilaz-S OPC. Count standards are not used in the calibration of the LiQuilaz OPC.

LiQuilaz OPC Design



Picture 1: Volumetric OPC Capillary Illumination

The LiQuilaz particle counter is a volumetric instrument that counts 100% of the particles passing through the sensor; that is, 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. Because the LiQuilaz counts 100% of the particles, the need to perform count standard tests is eliminated. Counting accuracy refers to the instrument's ability to count the correct concentration of particles in a solution.



As long as the sensor is sizing accurately, it is counting accurately. For example, if there is something wrong or the LiQuilaz particle counter is misaligned it would not size accurately. Sizing accuracy refers to the instruments ability to correctly determine the size of a Polystyrene Latex Sphere (PSL) (Picture 2).

Picture 2: Polystyrene Latex Spheres (PSLs).

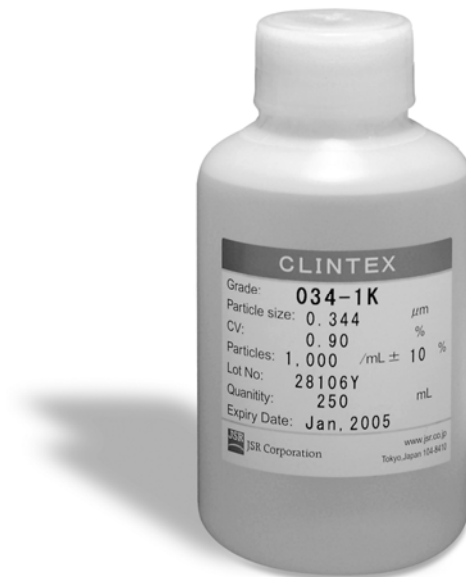
What are Count Standards?

Count standards are pre-mixed bottles of particles that contain a specified concentration of particles at a certain size. Common sizes of count standards used to check the performance of a LiQuilaz OPC are the 0.35, 0.6, and 15.0 microns (μm) EZ cal.

When using count standards the instructions on the bottle must be followed explicitly. There are detailed instructions on how to mix the standards, how to handle them, the expiration date, and that they are single use.

The certificate of analysis provides four pieces of information. It shows the name, lot number, and volume of the standard. The particle size specifies average diameter, uncertainty and CV of the size distribution. The concentration provides the specification and actual measured particles.

It also provides a date of certification and expiration date 6 months from certification.



Picture 3: Clintex Count Standards

CERTIFICATE OF ANALYSIS

Product name: CLINTEX-034-1K
Lot number: 28106Y
Volume: 250 ml

Particle size
Average diameter: 0.344 μm
Expanded uncertainty:
(k=2) 0.010 μm
CV of size distribution: 0.90%

Number concentration:
Specification: 1,000 \pm 100 particles/ml
Measured: 1,019 particles/ml

*: particle count obtained by a particle counter is divided by the counting efficiency.

Counting efficiency: 85%
Range of measurement: 0.30 μm and above

Date of Certification: July 22, 2004
Expiry date: Six months after the date of certification

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TECHNICAL INFORMATION

Specific gravity: 1.055
Reflective index: nD = 1.594 at 20°C

Picture 4: Certificate of Analysis

For example, the 0.344 μm standard specifies the average diameter is 0.344 μm with 0.01 μm uncertainty with a CV of 0.9%. The concentration specifies that there are 1000 \pm 100 particles/mL and the actual measured concentration is 1019 particles/mL. Further, it states that the counting efficiency at 0.3 μm is 85%. This means that when measuring the count standard, the reported counts 0.3 μm and greater could be between 765 $((1000-100)*.85)$ and 1100 $(1000+100)$ cts/mL.

The 0.6 μm count standard specifies the total concentration above 0.3 μm s. This means that in order to properly measure this count standard and report the full number of counts specified on the bottle, one needs to use an OPC with at least 0.3 μm sensitivity. Similarly, the 15 μm EZ Cal specifies measuring particles 10 μm and greater.

Test Setup

To properly check the operation of an OPC you must correctly design the test and then set up the test equipment. Picture 4 shows a typical syringe based sampling system. Proper laboratory technique is critical. Individuals performing the counting accuracy test must have experience measuring particles in ultra-pure liquids, preparing laboratory glassware, particle

counter operation, and posses overall good laboratory techniques and practices. The following requirements must be met to check the performance of the OPC.

- The OPC must be paired with a syringe sampler (i.e. LS200) capable of directly sampling from the count standard bottle.
- The sampler settings must be properly set with the correct syringe volume, flow rate, sample volume, tare, and number of samples.
- Prior to testing, the sampler and particle counter must have a background with DI water less than 10 cts/mL for the most sensitive size channel specified by the count standard.
- The DC light must be within specification. (S02 <0.5V, S03/S05 <0.05V, E20 > 8.25V).
- The count standard must be properly prepared according the instructions provided by the manufacturer.
- The count standard must be a new, unused standard within the expiration date specified by the manufacturer.



Picture 5: Syringe Sampling System

Data Collection

Data collection is equally important in checking the operation of the OPC. Default particle size channels should be used with the addition of the particle size specified by the count standard. For two common Clintex count standards, the certificate of analysis specifies 0.3 μm s. Table 1 shows the recommended size channels when using Clintex count standards for individual LiQuilaz OPCs. Channel spacing is defined under the instrument configuration in SamplerSight.

Table 1: Recommended OPC Size Channels for Count Standards

S02	S03	S05	E20
Size Channels (µms)	Size Channels (µms)	Size Channels (µms)	Size Channels (µms)
0.2	0.3	0.5	2.0
0.3	0.4	0.6	5.0
0.4	0.5	1	10.0
0.5	0.6	5	15.0
0.6	0.7	10	25
1.0	1.0	20	

The following examples in Figures 1 and 2 show proper channel configuration for a LiQuilaz S02 when measuring the 0.344 µm Clintex count standards.

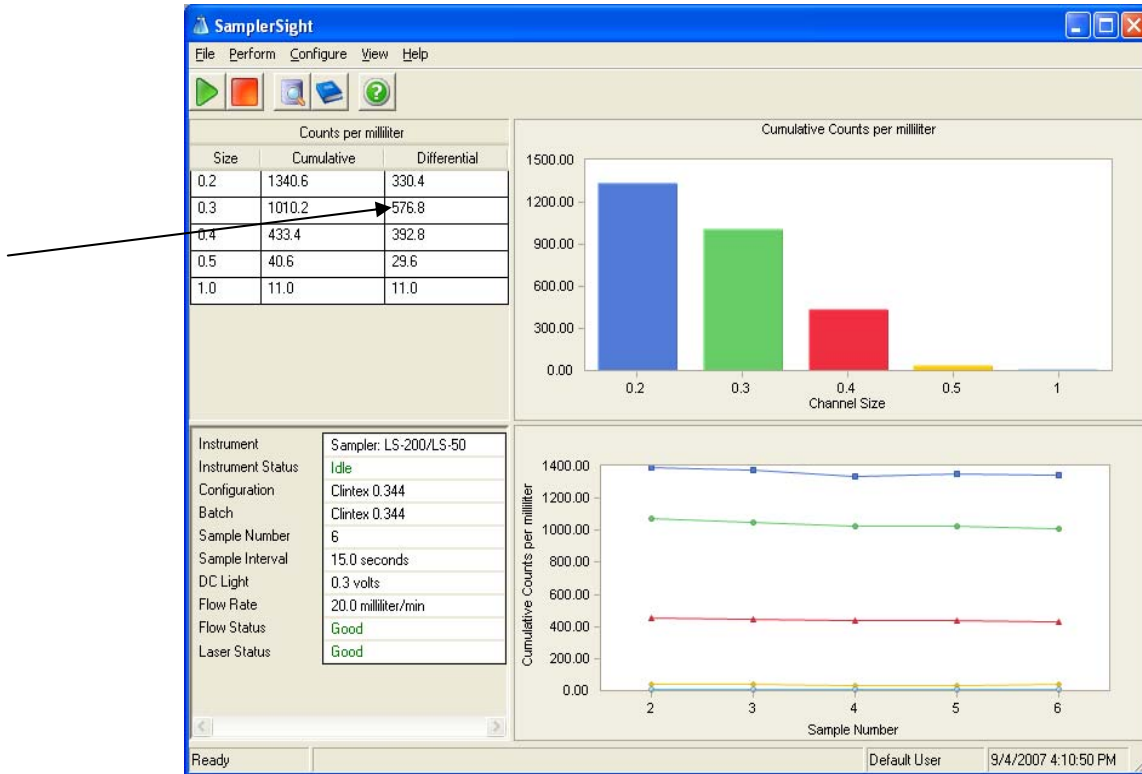


Figure 1: Clintex 0.344 µm Count Standard, Low Background Counts

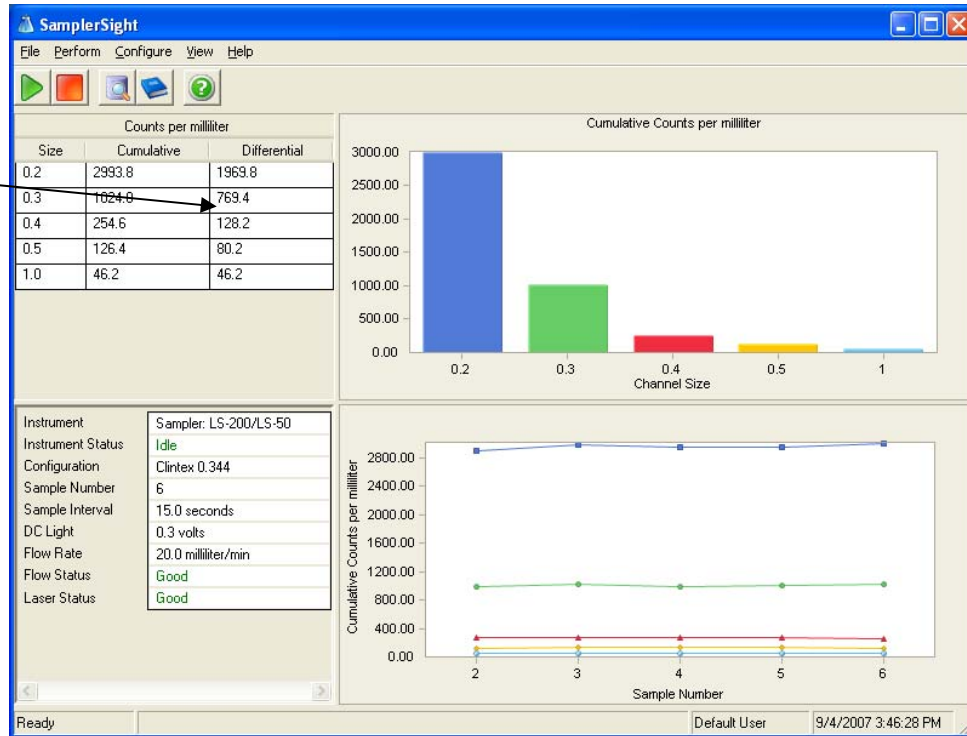


Figure 2: Clintex 0.344 μm Count Standard, High Background Counts

Count Standard Results

Once the count standard has been measured, the data can be analyzed to determine if the LiQuilaz OPC is performing as expected. In the examples shown in figures 1 and 2, both tests indicate the LiQuilaz is performing as expected and passes the count standard test according to the specifications provided by the Clintex count standard. Referring to the Clintex specifications described earlier in the Application Note, the LiQuilaz should report greater than 765 particles/mL 0.3 μm and greater. Figures 1 and 2 show measurements of 1010.2 particles/mL and 1024.0 particles/mL respectively.

Closer examination of the data shows very different results for both tests even though both pass the count standard specification. Figure 1 shows a test with a new count standard that has been properly prepared and tested. The total cumulative particles measured is 1340.6 particles/mL and the differential data shows a larger concentration of 0.3 μm particles (as noted above) as would be expected by measuring a 0.344 μm count standard. Even in this test, there is a small level of contamination demonstrating the difficulty in performing these measurements.

Figure 2 shows a test with an expired count standard that has been improperly prepared and tested. The total cumulative particles measured is 2993.8 particles/mL and the differential data does not indicate a higher concentration of 0.3 μm particles (as noted above). This is attributed to a higher than normal level of contamination within the count standard itself or in the test equipment.

The results of these two tests demonstrate why Clintex count standards cannot be used to verify the accuracy of the LiQuilaz OPC. Both sensors pass the count standard specification

with very different results. In addition, insufficient data is available to know if the LiQuilaz is sizing accurately.

Sizing Accuracy

Count standards provide insufficient information to calculate the sizing accuracy of an OPC. As a result, they should not be used to verify the performance of an OPC. Count standards should only be used to check the operation of the OPC.

Counting Error

Count standards only provide enough information to check the operation of an OPC by reporting the total counts observed in the count standard. Count standards do not provide sufficient data to predict the actual counting error on real world samples.

Conclusion

Checking the operation of a LiQuilaz particle counter is a useful way to determine if the particle counter is functioning. Count standards provide a good method to check the operation of a LiQuilaz. Exact procedures must be followed to collect this data. Count standards do not provide sufficient information to properly verify instrument performance.

References

¹Paper: Sizing Accuracy of a LiQuilaz-S Type Particle Counter, Roger Carlone, Particle Measuring Systems, 2008.

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