

# Troubleshooting For Liquid Particle Counting

This paper discusses how to ensure that quality data is being obtained from optical particle counters (OPCs) in liquid applications.

## Abstract

Ensuring that your liquid optical particle counter is operating within its designed specification is critical to obtaining quality data. As data from OPCs becomes increasingly important to maintaining process control, it becomes more critical than ever for users to have confidence in the data being generated. OPC data is frequently used in qualifying process improvements, in control during chemical manufacturing, and in quality control for production of complex parts. It is not uncommon for users to question the results from their particle counter. Discussed in this paper are common misunderstandings and other performance issues that would lead one to question their OPC.

## Introduction

Users can take several easy steps to ensure that quality data is collected. All OPCs have defined and easily controlled specifications. These specifications are flow rate, background scatter, and concentration limit. Also important is the particle size distribution (PSD). Unless all of these parameters are correct, the data should be considered suspect. Even if one parameter is incorrect, the data quality has been compromised. This paper discusses these parameters and their effects on each other, and it also discusses ultimately ensuring that quality data is achieved when analyzing ambient particle distributions in process chemicals and DI water.

## Normal Particle Distributions

Thirty years of particle counting experience shows that most ambient particle distributions in continuously filtered liquid systems follow a  $D^{-3}$  distribution ( $D$  = diameter) on total cumulative counts. Figure 1 shows a normal particle distribution. In extremely clean DI water systems, the distribution can be steep-

er ( $D^{-4}$ ), and in dirty systems, the distribution can be flatter ( $D^{-2}$ ). It is equally important that whatever the distribution is, it remains consistent from sample to sample.

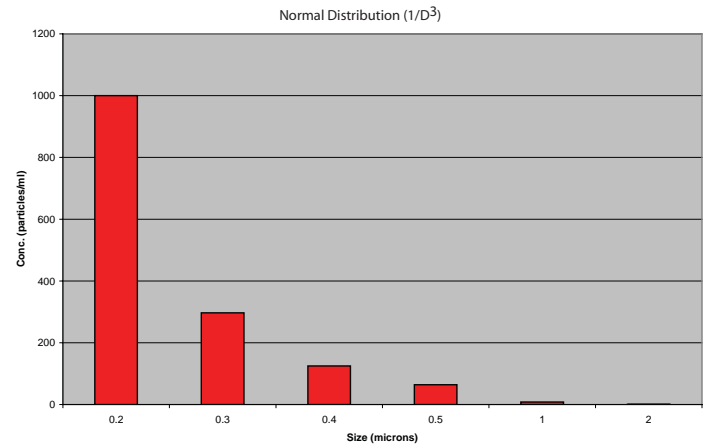


Figure 1: Normal Particle Distribution,  $D^{-3}$

It is essential to ensure that you have a normal particle distribution. If not, it should immediately alert the user to a potential problem. Things that can lead to abnormal distributions or poor data:

- Incorrect flow rate
- Excessive background scatter
- Exceeding the OPCs upper concentration limit
- In addition, real particle events such as pump or valve failures, o-ring problems, damaged tubing, or other process upsets can cause abnormal particle distributions. This is why it is critical to ensure that that quality OPC data is collected. Otherwise, one would not be able to identify a real particle excursion from a poorly performing OPC.

## Flow Rate

All OPCs have a flow rate specification. The specification is most commonly listed as the set flow rate plus or minus a certain percentage. For example, 50 mL/min. +/- 10%. The actual flow rate varies by model.

OPCs are calibrated at a specific flow rate. This

means that real world particles are intended to pass through the particle counter at the calibrated flow rate. If the proper flow rate is not maintained, both sizing and counting accuracy are compromised. As a particle travels through an OPC, it scatters light. The amount of scattered light is converted to an electrical signal which is counted and measured to determine the particle concentration and size.

When flow rate is set too high, the particles travel through the OPC too fast. Since the transit time is decreased, the electronics do not have sufficient time to fully integrate the signal, compromising sizing accuracy. Counting accuracy is compromised because the resulting signal is now too small to exceed the particle size threshold. Further complicating the issue is that the accompanying software is normalizing the data to the specified flow rate.

Conversely, if the flow rate is too slow, the transit time is increased and the particle appears to be larger than its actual size. It is critical that the OPC be operated at the correct flow rate.

Table 1 shows the effects of changing the flow rate of an OPC. The specified flow rate is 50ml/min.

Flow Rate	Deviation from Spec.	Cts./mL
40 mL/min.	-20%	3346.5
45 mL/min.	-10%	3217.5
50 mL/min.	0%	3091.9
55 mL/min.	10%	2957.0
60 mL/min.	20%	2841.2

Table 1: Effects of Flow Rate on Counting Accuracy

Flow rate is controlled in the following ways, depending on the application and equipment used:

- Syringe samplers require the user to input the correct syringe volume and sample speed. This offers extremely accurate flow control.
- Online applications require a flow controller downstream of the OPC set to the proper flow rate. It is important to calibrate these flow controllers with a stop watch and graduated cylinder. The accompanying software has no means to set the flow rate nor does it know if the OPC is being used at the correct flow rate.
- Compression samplers have a flow rate adjustment on the instrument to set the flow rate. In this case, the

software will report the actual flow rate at the end of a sample.<sup>1</sup> A detailed explanation of operating a compression sampler can be found in Particle Measuring Systems' Application Note 56, A Counting Challenge.

## Background Scatter

A small amount of background noise or scattered light in an OPC that originates from the laser interacting with the optical path. The scatter from the capillary walls or optical windows can be measured.

Normally, the amount of scatter from the capillary walls is minimal and has no effect on the data. As the capillary becomes contaminated or, in severe cases, damaged, the amount of scatter increases. In other designs, the optical windows from which the sample is viewed can become contaminated. Eventually, the amount of scatter coming from contaminated optics equals the amount of scatter from the smallest detectable particle size. In most cases, simple cleaning of the capillary can resolve this issue<sup>2</sup>.

Excessive background scatter can be recognized in the following ways:

- Accompanying software reports the amount of background scatter. Each OPC has a specification for this value. Once this value has been exceeded, the collected data should be considered suspect.
- Often the data will indicate excessive scatter. If the amount of scatter from contamination equals the amount of scatter from the smallest detectable particle size, there will be an excessive number of counts in the first channel. One will often see hundreds to thousands of counts in only the first channel on known clean DI water with zero to little counts in the other size channels, as shown in Figure 2.

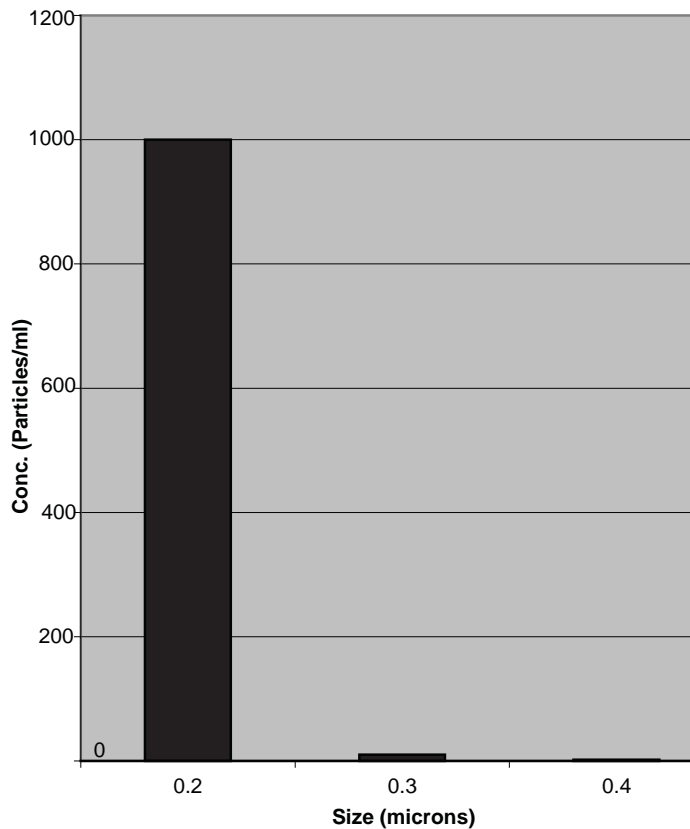


Figure 2: Excessive Background Scatter

Users often ask if they can ignore the first channel. The answer is “No” for two reasons. First, the OPC electronics becomes busy processing the noise signal which prevents it from accurately processing real particle signals. Second, there is a constant amount of light scatter hitting the detector. The OPC is designed to measure total cumulative counts from the smallest detectable size and greater. The electronics and signal processing are not designed to handle such situations, resulting in compromised data across all channels.

### Upper Concentration Limit

All OPCs have an upper concentration limit, which is the maximum allowable concentration of particles that the particle counter should be exposed to. This is typically expressed as <10% coincidence loss at 10,000 particles total cumulative counts. This means that at 10,000 particles, up to 10% of the particles are not being counted.

Optical coincidence occurs as the concentration of particles nears the upper limit, and two or more particles pass through the OPC viewing volume simultaneously. Particles passing side by side and appear larger, or smaller particles can be obscured by

bigger ones. The end result is that, as more and more particles pass through the OPC, the OPC loses the ability to distinguish one particle from another. When this occurs, the distribution of particles changes and typically appears as if there is the same number of counts in the first several particle channels of the OPC as seen in Figure 3.

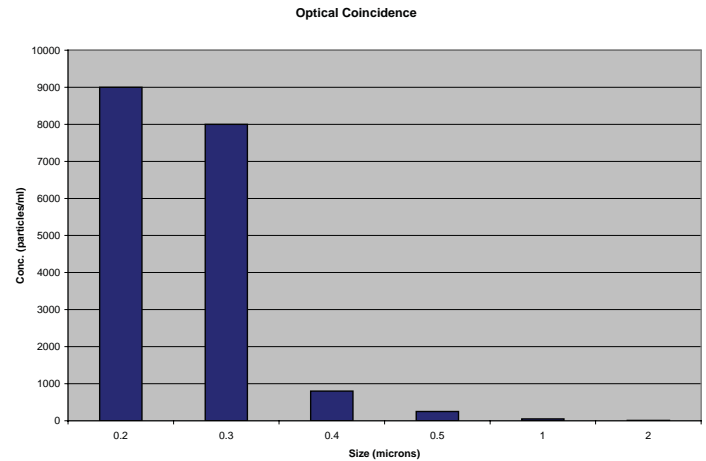


Figure 3: Optical Coincidence

When the concentration limit has been exceeded, there is no guarantee of the actual counts or that two instruments will agree with each other. Coincidence loss is neither predictable nor necessarily a linear response as the concentration increases.

If the particle concentration is extremely excessive, the OPC will saturate. When saturation occurs, the electronics are completely overloaded so that they no longer have the ability to respond to individual particles. The particle distribution will actually invert and show fewer counts in the first channel as seen in Figure 4. In such cases, the OPC can actually read zero counts.

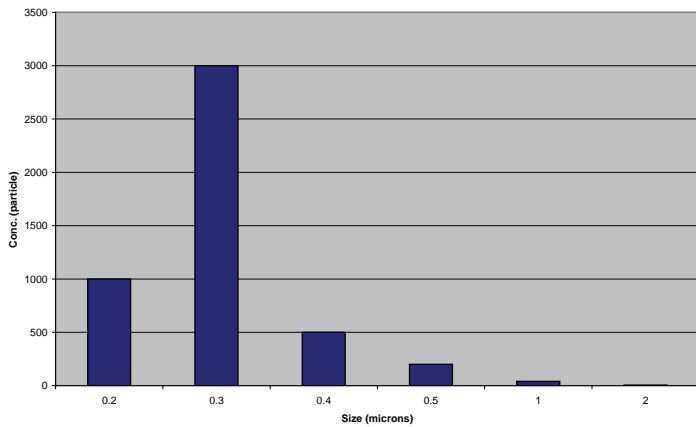
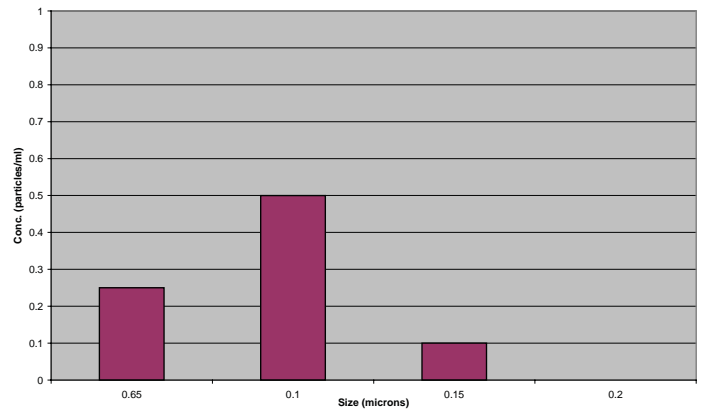


Figure 4: OPC Saturation

## Cosmic Rays

OPCs with less than 0.1 micron sensitivity is false counts due to cosmic rays. Cosmic rays are normally occurring, natural events. When a cosmic ray hits the photo detector, it will register as a particle. The rate of cosmic rays hitting the detector varies by location and environmental factors but generally occurs about once per minute, resulting in less than 1 ct./mL. It is also common for these counts to appear in the second channel of the OPC, which in most cases is 0.1 microns.

In normal applications this is not significant. However, most DI water systems, and even some process chemicals used in semiconductor manufacturing, have normal concentrations of particles less than 1-2 ct./mL. If the OPC is otherwise within specification and the normal concentration of particles is less than 1ct./mL, the resulting distribution of particles can appear as in Figure 5. It is important to note that most OPCs have a zero count spec of 1-4 cts/ml. This means that anything below this limit should be considered noise.



## Conclusion

Proper operation and adherence to published specifications of an optical particle counter is critical to obtaining quality data. Without quality data, one cannot make informed decisions regarding the control of their processes.

A few simple steps can ensure that quality data can be collected. First, one should look for a normal particle distribution. This is a good initial indicator of quality data. Even with a normal distribution, one should check that the OPC is operating with the correct flow rate and within the published specifications of background scatter and particle concentration.

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### Author

Roger Carlone  
Particle Measuring Systems

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Particle Measuring Systems Headquarters  
5475 Airport Blvd., Boulder, CO 80301  
1-303-443-7100 1-800-238-1801 Fax: 1-303-546-7331  
Instrument Service & Support: 1-800-557-6363  
Customer Response Center: 1-877-475-3317

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Particle Measuring Systems Europe  
Tel: +44 1684 581000  
PMSEurope@pmeasuring.com

Particle Measuring Systems Japan  
Tel: +81 3-5298-8175  
PMSJapan@pmeasuring.com

Particle Measuring Systems Singapore  
Tel: +65 68460-500  
PMSAsiaPacific@pmeasuring.com

Particle Measuring Systems China  
Tel: +86 21-6113-3688 751  
PMSChina@pmeasuring.com

Particle Measuring Systems Mexico  
Tel: +52 55-22-71-51-06  
PMSMexico@pmeasuring.com

Particle Measuring Systems Puerto Rico  
Tel: 1-787-740-8582, 1-787-740-1878  
PMSPuertoRico@pmeasuring.com

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