COMPARING PARTICLE COUNTER PERFORMANCE CHARACTERISTICS FOR ANALYZING HIGH-PURITY WATER

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INSTRUMENTS

COMPARING PARTICLE COUNTER PERFORMANCE CHARACTERISTICS FOR ANALYZING HIGH-PURITY WATER

State of the art high-purity water systems are now so clean that quantifying the particle concentrations is difficult. When measuring the cleanliness of the water the scarcity of particles can lead to statistical variations that can be as large or larger than the actual particle concentrations. A thorough understanding of the statistics of particle counting is needed to mitigate these variations and effectively measure the particulate contamination.

There are three major determinants of the statistical performance of a particle counter. First is the particle size distribution, second is the sample volume, and third is the size sensitivity. In addition, the sample interval used will also affect the variation in the measurements. All of these factors, as well as appropriate alarm levels, must be considered when determining which particle counter to choose.

This article will discuss the size distribution of particles found in high-purity water systems, explain the statistics of particle counting, and show how sensitivity and sample volume both affect the statistical performance of particle counters. Finally, a spreadsheet has been developed that compares the statistical performance of different particle counters, shows the variability in measurements that can be expected, and estimates the number of times a day a given alarm level will be exceeded due to statistical variations.

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Particle Size Distributions

Knollenberg and Veal (1) did an extensive study of particle size distributions in clean water systems. They examined 17 systems and the results are shown in Figure 1. The distributions they measured could best be represented by a power law expression of as seen in Equation 1.

$$N = c(\text{particle diameter})^{P} \qquad \text{Eq. 1}$$

Where N is the total number of particles larger than the diameter, and c and P are empirically derived constants. The average of the 17 systems was P = 3.01. That is, the number of particles in these water systems was proportional to (diameter)$^{-3}$.

A subsequent study by Mitchell (2) showed that this power law distribution holds for all liquid systems. Further, the (diameter)$^{-3}$ relationship holds true for chemicals, as well as water, that are filtered and in a steady state. Significant deviation from this relationship occurs only when particles are actively being created, such as during etching processes or during ultrasonic cleaning.

Based on this evidence, it is reasonable to use the (diameter)$^{-3}$ relationship to estimate the number of particles at different sizes. That is, we can assume that if we have 8 particle per milliliter (p/mL) at 0.05 micron (µm) there will be only 1 p/mL at 0.1 µm.

Basic Particle Counting Statistics

The statistics of liquid borne particle counters are relatively simple and well known. It has been established that particles are counted in time in accordance with a Poisson process with rate l (3). l is the expected rate or the long-term average rate, and is often established empirically. The expected number of particles counted for any period of time t is then lt. So if a large number of measurements are made during time period t, the average value of these measurements is lt. The standard deviation of these measurements will be the

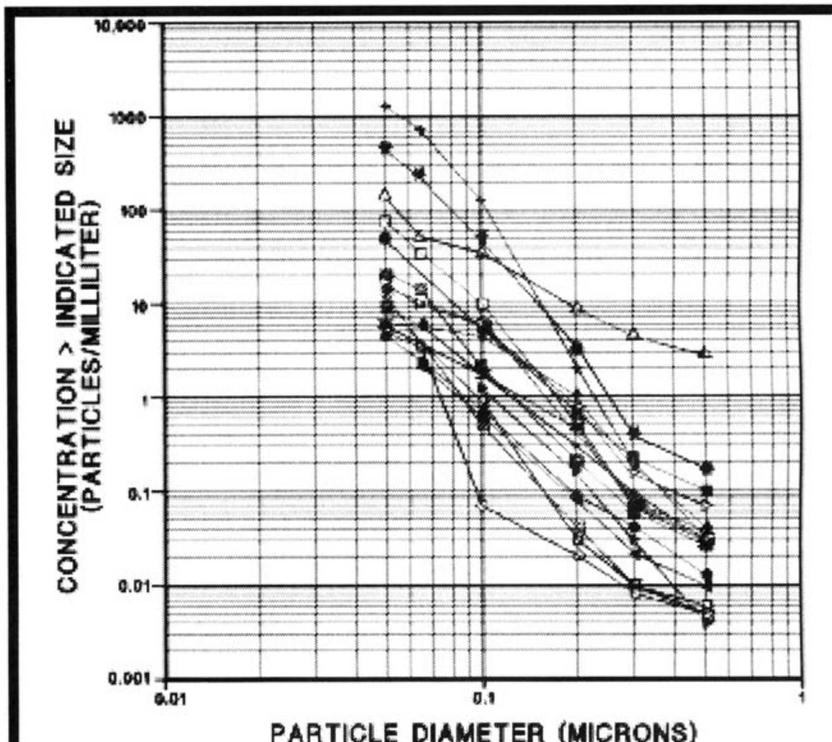

Figure 1. Particle size results from examining 17 clean water systems.

square root of the expected value (4) as seen in Equations 2 and 3.

$$m = \lambda t \qquad \text{Eq. 2}$$

$$s = (\lambda t)^{0.5} \qquad \text{Eq. 3}$$

Given l, Poisson statistics also allow us to calculate the probability that we will count x particles during time period t, where x is any number of interest, and t is any desired time period (Equation 4).

$$P(x, \lambda t) = e^{-\lambda t}(\lambda t)^x / x!\ , \quad x = 0,1,2,\ldots, \qquad \text{Eq. 4}$$

Two important observations can be made from Equations 2 and 3. First, as more particles are counted the statistical variation decreases relative to the measurement average. That is, the ratio of the average to the standard deviation, or the signal-to-noise ratio (s/n) on the measurements, increases as more particles are counted. Relative to particle counters, this means that the more particles an instrument counts per unit time the better the statistical performance. Second, longer measurement intervals also increase the signal-to-noise ratio, and the improvement is proportional to the square root of the time increase. If the sample interval is increased by a factor of 4, the s/n will increase by a factor of 2. If the sample interval is increased by a factor of 10, the s/n will improve by a factor of 3.16.

If the long-term average is known, Equation 4 provides the ability to estimate the number of times a certain measurement value will be seen. This is important because we can now estimate the number of times a particular value will be exceeded and this can be essential in setting appropriate alarm levels and sample intervals.

Statistics and the Real World

An example of the impact of statistical variation is shown in Figure 2. These measurements were made on the water system at a major semiconductor facility. This facility had a specification of less than 4 counts/mL, the sample volume of the instrument was 0.25 mL/min, and they were using 4-minute sample intervals. The long-term average of the measurements is 3.2 counts/mL, so the system was meeting specifications, but the statistical variation in the 4-minute measurements caused many of these measurements to exceed their specification. The customer was concerned

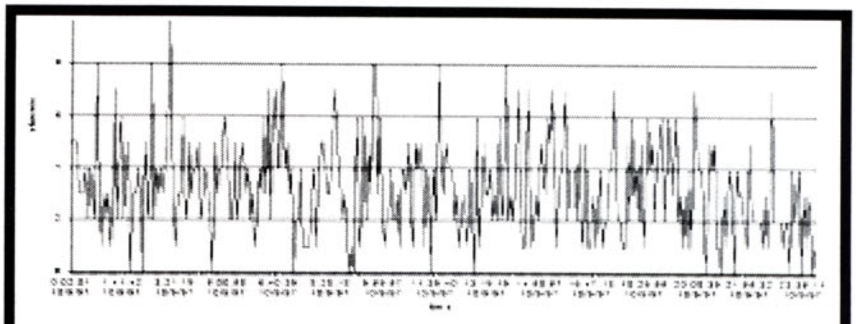

Figure 2. The impact of statistical variation on a water system at a semiconductor plant.

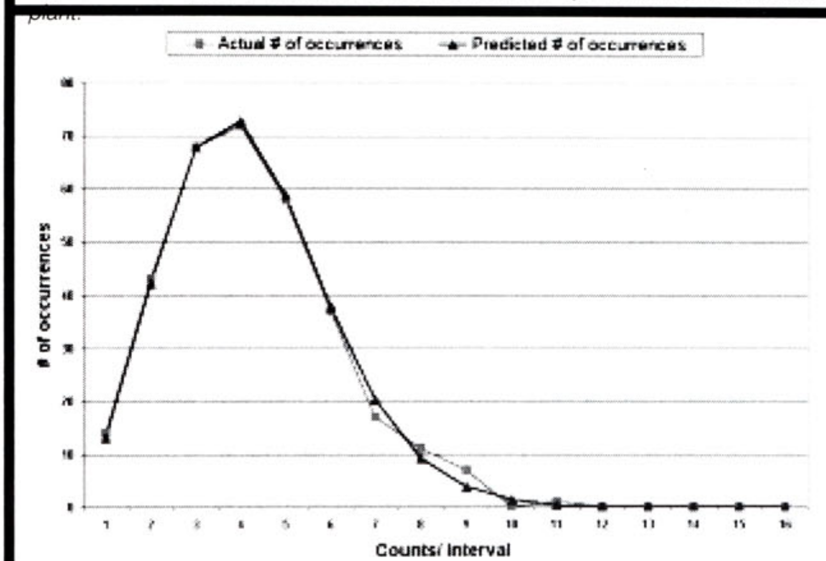

Figure 3. A plot showing the particle count results as reported in Table A.

because there are several data points at 6 counts or higher even though the mean for the day was only 3.2 counts. Based on the mean and using Poisson statistics, we can calculate how many times in a day we would expect see each number of counts. The results are shown in Table A and plotted in Figure 3.

As shown in Table A, using the Poisson distribution, one would expect 6 counts, 20 times over the day, but only 17 actually happened. The results are plotted in Figure 2, and as can be seen, the agreement between theory and measurement is very good. Therefore, the occasional high counts seen in Figure 1 are simply the result of the statistical variation, and reacting to these high counts is inappropriate.

Estimating Particle Counter Performance

Assuming a (diameter)$^{-3}$ distribution, the user can now compare the statistical performance of different particle instruments based on their sample volume and sensitivity. For example, we can compare two volumetric particle instruments (instruments that measure 100% of the sample flow) that both have sample volume of 50 mL/min, with Instrument #1 having 0.1 µm sensitivity while Instrument #2 is limited to 0.2 µm. Instrument #1 will see 8 times as many particles in a given period of time based on (diameter)$^{-3}$ distribution as shown by Equation 5.

$$\lambda t_1 = 8\lambda t_2 \ \ or\ \ 8 = \lambda t_1/(\lambda t_2) \qquad \text{Eq. 5}$$

From Equation 3, we know that the standard deviation of the data is proportional to the square root of lt (see Equation 6).

$$s_1/s_2 = (\lambda t_1/\lambda t_2)^{0.5} = (8)^{0.5} = 2.8284 \qquad \text{Eq. 6}$$

Therefore, the standard deviation for Instrument #1 is 2.8 times larger in absolute terms. However, as a fraction of the number of particles counted, it is 8 / 2.8 = 2.8 times smaller. This reduces the

TABLE A
Actual Number of Occurrences versus Predicted

Counts per 4 Minute Interval	*Actual # of Occurrences*	*Predicted # of Occurrences*
0	14	13.07
1	43	42.12
2	68	67.87
3	72	72.91
4	58	58.74
5	37	37.86
6	17	20.33
7	11	9.36
8	7	3.77
9	0	1.35
10	1	0.44
11	0	0.13
12	0	0.03
13	0	0.01
14	0	0.00
15	0	0.00
Total	328	328.00

affect noise has on the data and the user will be able to detect smaller changes in the particle concentration.

Another example of sample volume's effect on data is seen by comparing three particle instruments designed specifically to monitor deionized (DI) water systems. Table B shows the relevant specifications of these instruments, with all three sensitive to 0.05-µm particles, but having different sample volumes. Very clean water systems now have particle levels of 0.2 p/mL > 0.05 µm, and for this example we will assume this to be the "true" particle concentration and/or the long-term average. A sample interval of 30 minutes, typical for monitoring DI water systems, will be assumed.

As shown in Table B, the standard deviation decreases as the sample volume increases, which in turn produces fewer alarm conditions as the statistics are dependent on the number of particles detected and not the concentration. Therefore, all calculations are based in particle numbers, with the effect of sample volume on standard deviation being expressed as shown in Equation 7.

$$s = \frac{(\text{particles counted per sample})^{0.5}}{\text{volume per sample}}$$

Eq. 7

As part of this article, a spreadsheet has been developed to perform this type of comparison previously seen in Table B. The spreadsheet requires the user to input the particle concentration for a user-defined size, the instrument specifications of sample volume and sensitivity, and the sample interval. In addition, the user can specify an allowable variation in the measurement or alarm level. The calculations then assume the (diameter)$^{-3}$ relationship and statistical nature of how the particles arrive in time.

Table C shows another comparison between three instruments that are designed to monitor DI water systems. The user first enters in the assumed particle concentration and in this case the concentration is 0.8 p /mL at 0.05 µm. Next, the sample interval is entered in seconds, here 600 seconds or 10 minutes. Finally, an alarm level is entered. The alarm level is relative to the average concentration, and in this case the alarm limit is specified 50% higher than the long-term average, meaning the alarm level is 1.2 counts/mL. Next, the sample volume and first channel sensitivity of the particle counters are entered. Remember that the sample volume is sometimes not specified directly and may be calculated by multiplying the sample flowrate by the percent sampled.

In this example, all three instruments have the same sensitivity and differ only in the sample volume. Instrument 3 outperforms the other two because of its relatively large sample volume as seen in the number of particles detected per measurement, the standard deviation, and the number of alarms per day. The number of alarms per day is particularly important because this demonstrates how better statistical performance affects the ability of an instrument to monitor the water system. With Instrument 3, a slight increase in concentration will be detected, while the statistical variation on the other two instruments creates 54 false alarms for Instrument 1 and about 34 for Instrument 2. To limit the number of false alarms to less than 1 a day, the alarm level must be close to 389% of the average concentration or 3.91 particles /mL for Instrument 1 and 174% or 2.19 p /mL for Instrument 2. Increases in particle levels must be relatively large or will be lost in the noise for Instruments 1 and 2. On the other hand, Instrument 3 with the larger sample volume will be able to recognize small increases in concentration.

Many users have requested instruments with sensitivity to smaller particles, but the best commercially available instrument is sensitive to 0.05 µm. To examine the benefit of increased sensitivity, the user can specify different sensitivities and sample volumes as shown in Table D. The particle concentration is assumed to be 0.4/mL at 0.05 µm and the alarm 50% above the average.

As can be seen, the instrument with the best sensitivity, Instrument 1, has the poorest statistical performance, and the instrument with the poorest size sensitivity, Instrument 3, has the best statistical performance. Under the assumed conditions, Instrument 1 has approximately 20 alarms because of statistical variation while Instrument 3 has less than one. In the end, the most important factor determining statistical performance is the total number of particles measured in a sample interval, not the size of the particles. A particle instrument's sample volume and sensitivity must be considered together to estimate particle counter performance.

Summary

The statistical behavior of particles in high-purity water systems can be understood and predicted using Poisson statistics. Combined with the (diameter)$^{-3}$ size distribution demonstrated by Knollenberg and Veal (1) and Mitchell (2) the statistical performance of particle counters can be estimated and comparisons of different particle counters can be made. These comparisons show

TABLE B
A Comparison Between Instrument Performances

Criteria				
Smallest detected particle size	0.05 µm			
Particle concentration	0.20 counts/ mL ≥ smallest detected particle size per d-3 size distribution			
Sample interval1,800 seconds				
Alarm level	25.00% percent above particle concentration			

Particle Counter	*Instrument 1*	*Instrument 2*	*Instrument 3*	
Sample volume	0.05	0.25	3.75	mL/min
1st Channel	0.05	0.05	0.05	µm

Results	*Instrument 1*	*Instrument 2*	*Instrument 3*	*Notes*
Expected concentration	0.20	0.20	0.20	1st channel and greater per d^{-3}
Samples per day	48	48	48	
Volume per sample	1.5	7.5	112.5	mL
Detected particles	0.30	1.50	22.50	1st channel and greater per d^{-3}
Standard deviation	0.3651	0.1633	0.0422	
P(sample exceeds alarm level)	0.4455	0.3797	0.1178	
Upper alarm level	0.25	0.25	0.25	1st channel and greater per d^{-3}
Alarms per day	21.39	18.23	5.66	from statistical variation

Common Control Limits	*Instrument 1*	*Instrument 2*	*Instrument 3*
90.0% UCL	0.67	0.41	0.25
95.0% UCL	0.80	0.47	0.27
99.87% UCL	1.30	0.69	0.33

TABLE C
Comparison of Particle Counters Monitoring DI Water

	Criteria	
Smallest detected particle size	0.05 µm	
Particle concentration	0.80 counts/ mL ≥ smallest detected particle size per d^{-3} size distribution	
Sample interval	600 seconds	
Alarm level	50.00%	percent above particle concentration

Results	*Instrument 1*	*Instrument 2*	*Instrument 3*	*Notes*
Expected concentration	0.80	0.80	0.80	1st channel and greater per d^{-3}
Samples per day	144	144	144	
Volume per sample	0.5	2.5	37.5	mL
Detected particles	0.40	2.00	30.00	1st channel and greater per d^{-3}
Standard deviation	1.2649	0.5657	0.1461	
P(sample exceeds alarm level)	0.3759	0.2397	0.0031	
Upper alarm level	1.20	1.20	1.20	1st channel and greater per d^{-3}
Alarms per day	54.13	34.52	0.44	from statistical variation

Common Control Limits	*Instrument 1*	*Instrument 2*	*Instrument 3*
90.0% UCL	2.42	1.52	0.99
95.0% UCL	2.89	1.73	1.04
99.87% UCL	4.59	2.50	1.24

TABLE D
A Comparison of Different Sensitivities and Volumes

Criteria	
Smallest detected particle size	0.05 µm
Particle concentration	0.40 counts/ mL > smallest detected particle size per d^{-3} size distribution
Sample interval	600 seconds
Alarm level	50.00% percent above particle concentration

Particle Counter	*Instrument 2*	*Instrument 3*	*Measure*	
Sample volume	0.25	3.75	50	mL/min
1st Channel	0.03	0.05	0.1	µm

Results	*Instrument 1*	*Instrument 2*	*Instrument 3*	*Notes*
Expected concentration	1.85	0.40	0.05	1st channel and greater per d^{-3}
Samples per day	144	144	144	
Volume per sample	2.5	37.5	500	mL
Detected particles	4.63	15.00	25.00	1st channel and greater per d^{-3}
Standard deviation	0.8607	0.1033	0.0100	
P(sample exceeds alarm level)	0.1410	0.0264	0.0062	
Upper alarm level	2.78	0.60	0.08	1st channel and greater per d^{-3}
Alarms per day	20.30	3.80	0.89	from statistical variation

Common Control Limits	*Instrument 1*	*Instrument 2*	*Instrument 3*
90.0% UCL	2.95	0.53	0.06
95.0% UCL	3.27	0.57	0.07
99.87% UCL	4.43	0.71	0.08

that often sample volume is more important than increased sensitivity.■

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