How Many Particles to Measure

AccuSizer® system

The results generated by the AccuSizer® are particle size distributions. The properties of distributions are governed by the rules of statistics. An important question when using a particle size analyzer is how much of a sample needs to be measured to properly define the resultant size distribution. This technical notes addresses this question both mathematically and experimentally.

INTRODUCTION

If all of the particles in a sample are the exact same size, then measure one particle and report the result. If the sample has a narrow distribution, let's say from 10 – 25 µm, then perhaps measuring only a small number of particles could define the distribution. But if the sample has a broad distribution, then it may become necessary to measure many thousands of particles to fully define the actual distribution.

We also need to ask which result is of interest. If we are only interested in a central value, such as the median fewer particles, it must be measured as if we are focusing on the edge of the distribution, such as the D90 or D95.

Like so many other aspects of particle size analysis, it is best to first define what is required and how the data will be used before beginning the measurement process. In the case of determining the amount of sample to measure (how many particles to analyze when using a counting technique such as the AccuSizer), some understanding of the width of the distribution is required before it is possible to define the required experimental parameters, such as number of particles to size/count or how long to run the experiment.

STANDARD ERROR APPROACH

Let's begin by considering how many particles to size in order to find a mean value in a Gaussian distribution. The fact that there are several different mean values used in the world of particle size analysis will be discussed later in this document. The standard error of a sample with sample size is the standard deviation, of the sample divide by the square root of n as shown in equation 1.

$$SE = s/(\sqrt{n}) \qquad \text{(Equation 1)}$$

where

SE = standard error
n = number of particles counted

$$\text{So} \qquad n = s/SE^2 \qquad \text{(Equation 2)}$$

If the sample has a standard deviation of 2 and we can live with a 2% standard error, then the number of particles, that needs to be measured is

$$n = 2/(0.02)^2 = 5000$$

This value of 5000 particles to measure is probably good to begin with – but only if we only have interest in a mean value.

ISO 13322-1 APPROACH

The topic of how many particles should be sampled to achieve satisfactory results is well described in the standard ISO 13322-1, Particle Size Analysis – Image Analysis Methods – Part 1: Static Image Analysis Methods.[1] The approach described in the ISO standard is based on the work published by Masuda and Innoya.[2] A short summary of this approach if given below. The number of particles to be analyzed, n, is given by:

$\log n = -2 \log \delta + K$ (Equation 3)

δ = the relative error

K = numerically determined by the confidence limit, particle distribution, and other parameters

Next:
$\log n^* = -2 \log\delta + \log\varpi$ (Equation 4)

where

$\varpi \equiv u^2\alpha^2\sigma^2(2c^2\sigma^2+1)$ (Equation 5)

where:

n* = number particles required in a measurement to attain a certain level of confidence.

δ = relative error

σ = population standard deviation

$c = \beta+\alpha$

β = basis number

α = constant

The probability, P, can be related to u as shown in Table 1 below:

Probability	u
50%	0.67
75%	1.15
80%	1.28
90%	1.64
95%	1.96
97.5%	2.24
99%	2.58
99.5%	2.81
99.8%	3.09
99.9%	3.29

Table 1. Relationship between probability percentage and u

Table 2 shows the number of particles required, n*, analyzed using equation 4 with admissible error of 5% as a function of the geometric standard deviation, σGSD, of the sample. Here, the probability, P, is taken as P = 0.95 (u = 1.96 in Table 1).

Geometric Standard Deviation	n*(DMM)	n*(Sauter)	n*(DMV)
1.1	585	389	131
1.15	1460	934	294
1.2	2939	1808	528
1.25	5223	3103	843
1.3	8526	4920	1274
1.35	13059	7355	1750
1.4	19026	10504	2363
1.45	26617	14457	3096
1.5	36007	19295	3956
1.55	47358	25093	4952
1.6	60811	31919	6092

Table 2. Number of particles n as a function of geometric standard deviation, σGSD*

The first column in Table 2 is the width of the sample distribution, expressed at the geometric standard deviation, σGSD. It should be noted that the maximum σGSD of 1.6 is actually not very wide, so many samples would exceed this value. Therefore the last row should be considered for samples with an unknown width. The second through fourth column show the number of particles to be analyzed, n*, depending on which mean value is of interest. The mass median diameter (DMM) is commonly used, so the second column should be referenced for most samples.

Therefore, a good rule of thumb is that when using the AccuSizer and converting to a volume distribution, it is best to analyze approximately 60,000 particles to achieve a high confidence level in the results.

EXPERIMENTAL

The previous example of standard error and the approach taken in ISO 13322-1 suggest that between 5,000 and 60,000 particle should be analyzed to have a high confidence level in the calculated mean value. An experiment was performed to compare actual measurements to the theoretical limits. The sample was a hydrated alumina powder dispersed in water and measured on the AccuSizer SIS system using the LE400 sensor; dynamic range 0.5 – 400 µm. The sample volume analyzed was varied from 0.05 to 2 mL in order to vary the number of particles analyzed. Table 3 and Figure 1 display the D10, D50, and D90* as a function of number of particles analyzed.

#Sized	D10	D50	D90
442	8.5 µm	13.82 µm	21.65 µm
2579	8.3 µm	13.82 µm	20.99 µm
5213	8.73 µm	13.82 µm	21.71 µm
11364	9.47 µm	15.39 µm	25.63 µm
13196	10 µm	16.24 µm	25.02 µm
16748	17.41 µm	24.07 µm	44.11 µm
37688	17.14 µm	29.42 µm	50.45 µm

Table 3. Number particles sized vs. D10, D50, and D90

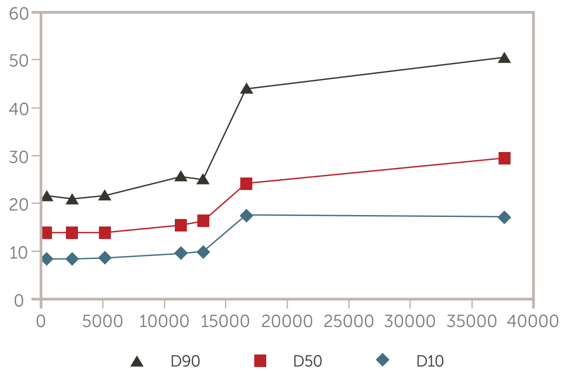


Figure 1. Number particles sized vs. D10, D50, and D90

Several observations and conclusions can be drawn from the data shown in Figure 1, including:

- The results at Number Sized ≤ 11,364 appear to be in error with all values underreported.
- At least 16,748 had to be analyzed to begin achieving close to accurate results.
- The error in D90 is greater than the error in D10 or D50 when analyzing too few particles.
- It would have been easy to stop this experiment at 5,213 particles analyzed and convince ourselves that the results were accurate – and been quite wrong.

The magnified error in the D90 is worth additional consideration. In many industries it's the few large particles that cause the biggest problems. In the microelectronics industry, a few large particles in a CMP slurry can cause surface defects, lowering yields and profits. Detecting the large particle count (LPC) in CMP slurries requires adequate statistics at the tail of the distribution, to the right of the D90. Therefore, even more particles should be analyzed than suggested, by either the theoretical or experimental results, discussed in this document to accurately define the tail of the distribution.

CONCLUSIONS

It is attractive but quite dangerous to analyze just a few particles and believe that the distribution has been accurately defined. It might be possible to analyze just a few particles, and report a mean value for very narrow distributions. But for wider distributions very many particles (tens of thousands) should be analyzed to accurately define the true particle size distribution. The number analyzed should be even higher when the D90, or tail, of the distribution is a primary interest. The AccuSizer remains the best analytical instrument available to easily analyze large numbers of particles both to define the distribution accurately, and to detect tails of distributions.[3]

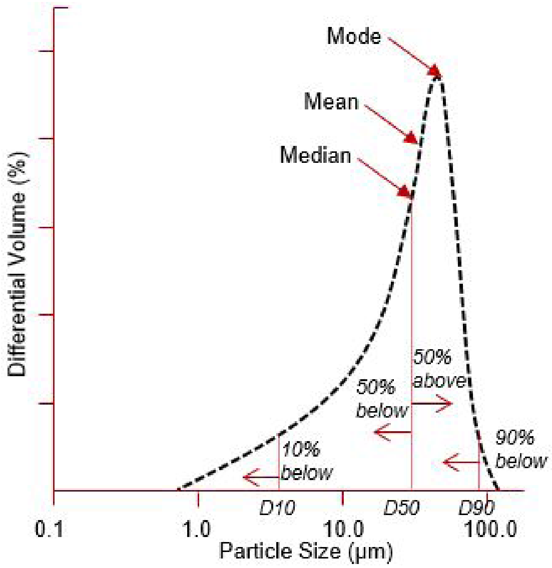
References

[1] ISO 13322-1, Particle Size Analysis—Image Analysis Methods—Part 1: Static Image Analysis Methods, available at www.iso.org

[2] H. MASUDA & K. GOTOH, *Study on the sample size required for the estimation of mean particle diameter, Advanced Powder Technol.,* 10(2), 1999, pp. 159-173

[3] Entegris Application Note - Tails in CMP Slurries

**90% of the distribution lies below the D90, 50% below the D50, and 10% below the D10, see below*



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Corporate Headquarters
129 Concord Road
Billerica, MA 01821
USA

Customer Service
Tel +1 952 556 4181
Fax +1 952 556 8022
Toll Free 800 394 4083