Advances in LPC* Monitoring in CMP Slurries

Statistics are a Bear

Mark Bumiller
Particle Sizing Systems

(*LPC = Large Particle Counts, typically > 1 µm)

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LPC vs. Defects/Scratches (Yield)*

- Slurries differing in LPC by a minimum of 1.8×10^5particles/g slurry had statistically different predicted scratch events at the 95% confidence level.
- LPC from PSS AccuSizer 780
  - LE400 (extinction) sensor

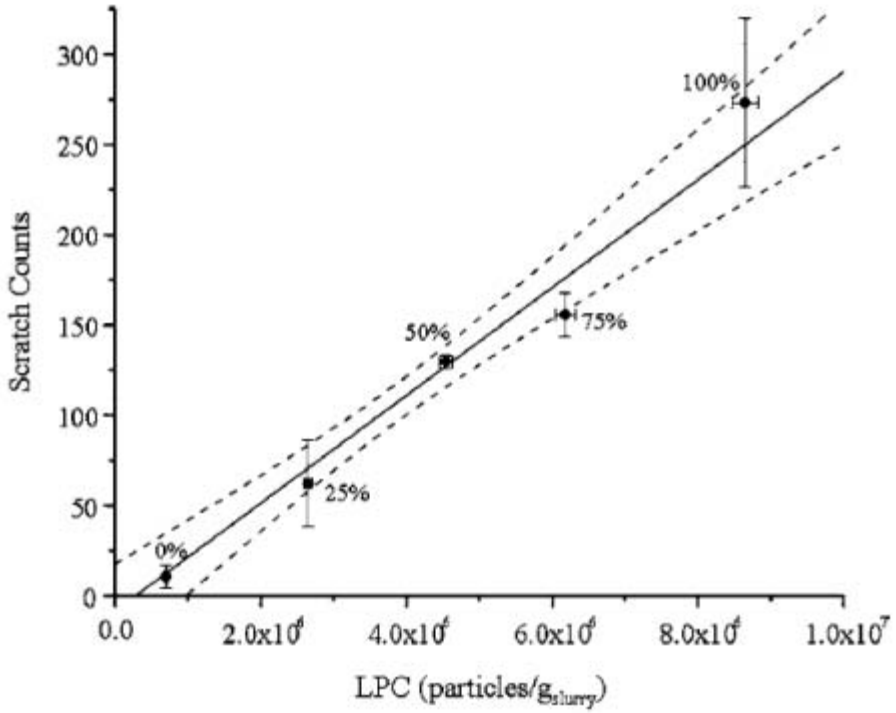

Figure 1. Correlation between scratch counts and LPC determined for particles with diameter ⩾0.469 μm (S03 + S05) with mixtures of slurry A (0 wt % slurry B) and slurry B. Weight% slurry B for the mixtures is labeled at the corresponding data point. The weighted linear regression fit to the data set and 95% confidence limits are represented by the solid line and the dashed lines, respectively. Error bars correspond to ±1 standard deviation.

*Remsen, E. et al., Analysis of Large Particle Count in Fumed Silica Slurries and Its Correlation with Scratch Defects Generated by CMP, Journal of The Electrochemical Society, 153 (5) G453-G461(2006)

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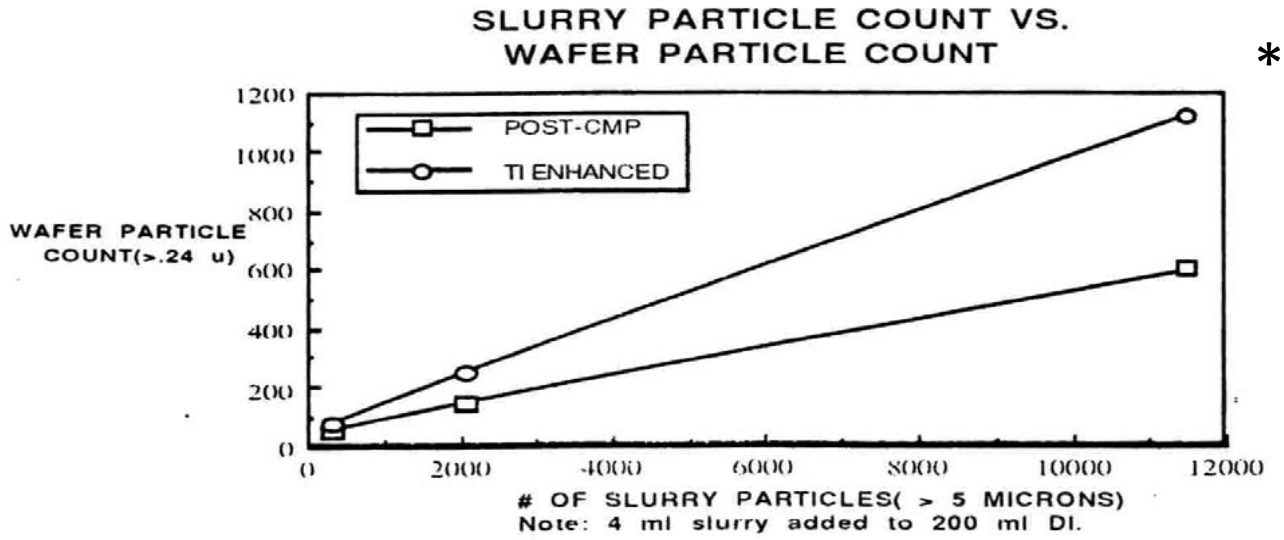
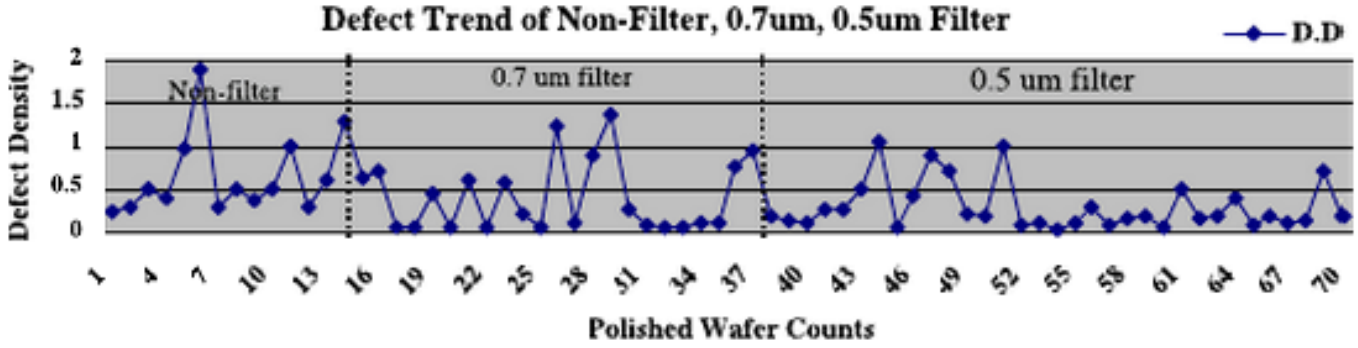
LPC Correlates to Yield





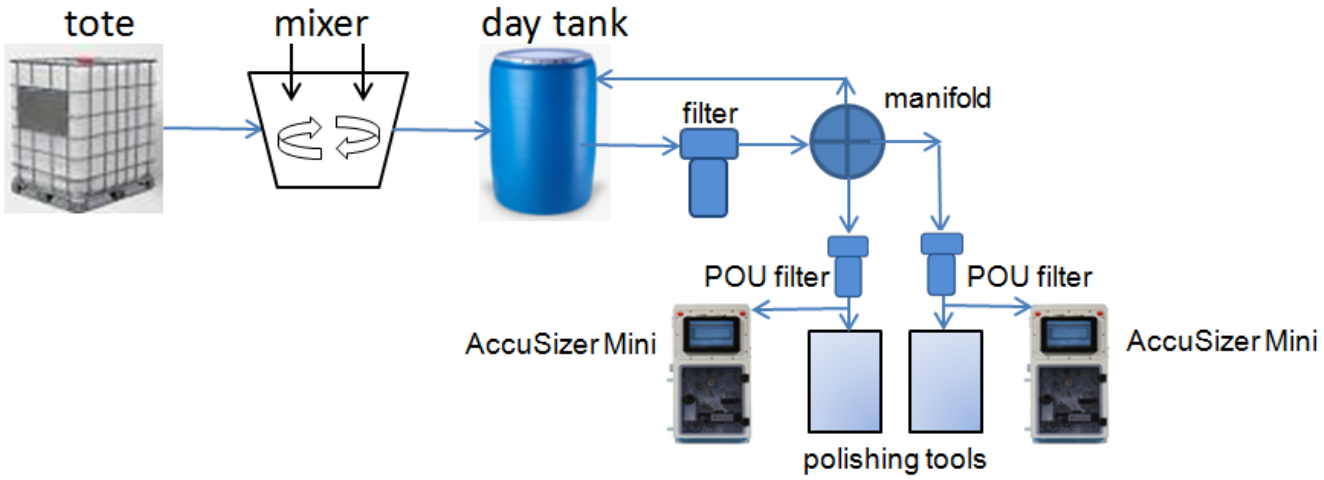
* R. Nagahara *et al*, VMIC-MIC, 1996

**from Y.-J.Seo et al., Effects of slurry filter size on the chemical mechanical Polishing (CMP) defect density, Materials Letters, 58(2004) 2091-2095

The Wish

- Please give me an ideal LPC monitor:
- Inexpensive
- Point of Use?
- No Dilution?
- Finds LPC's

- Yield ROI?
- Yes, LPCs generated
- Coincidence (statistics)
- Statistics matter



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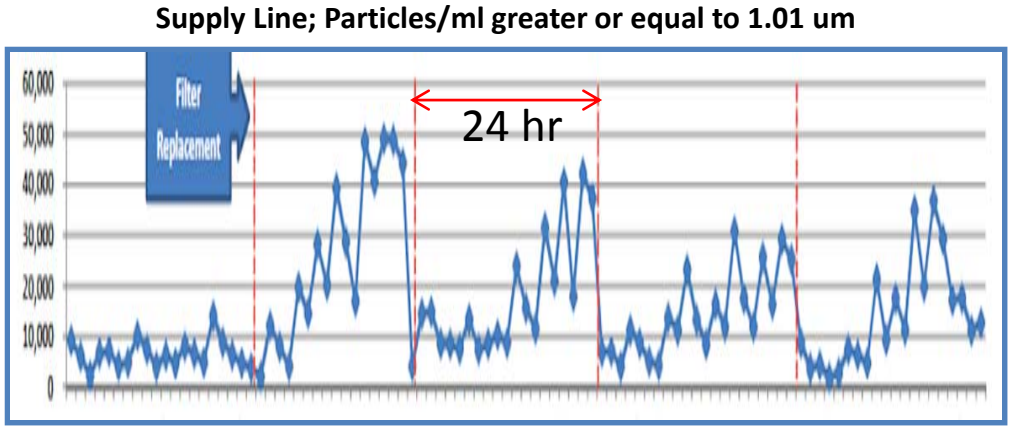
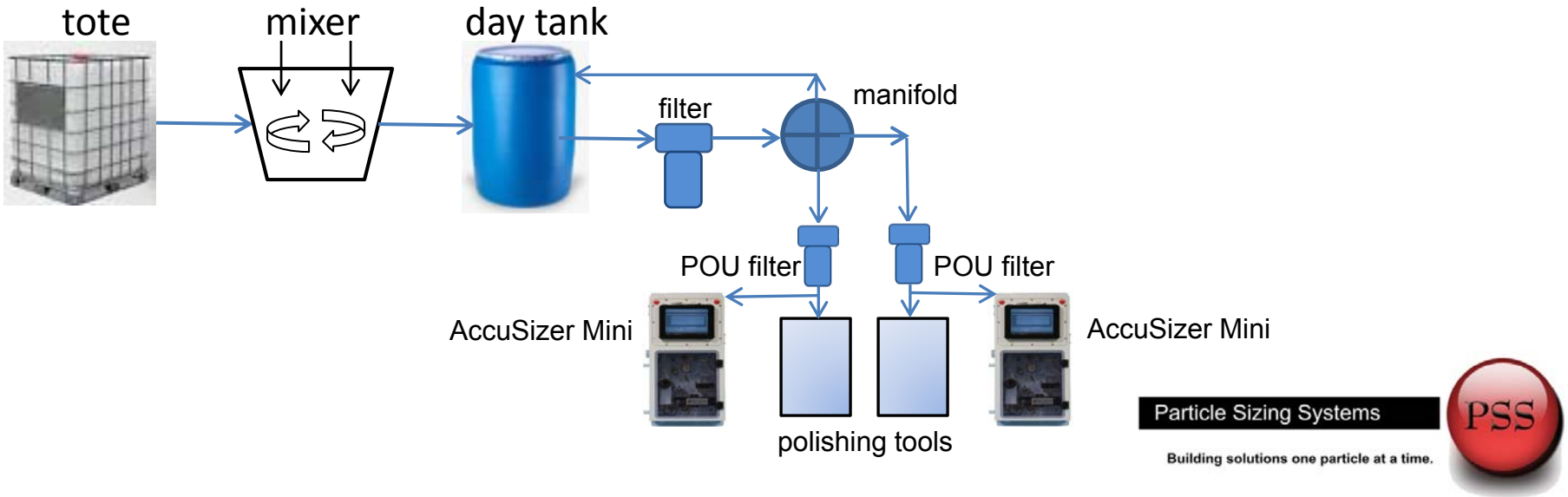
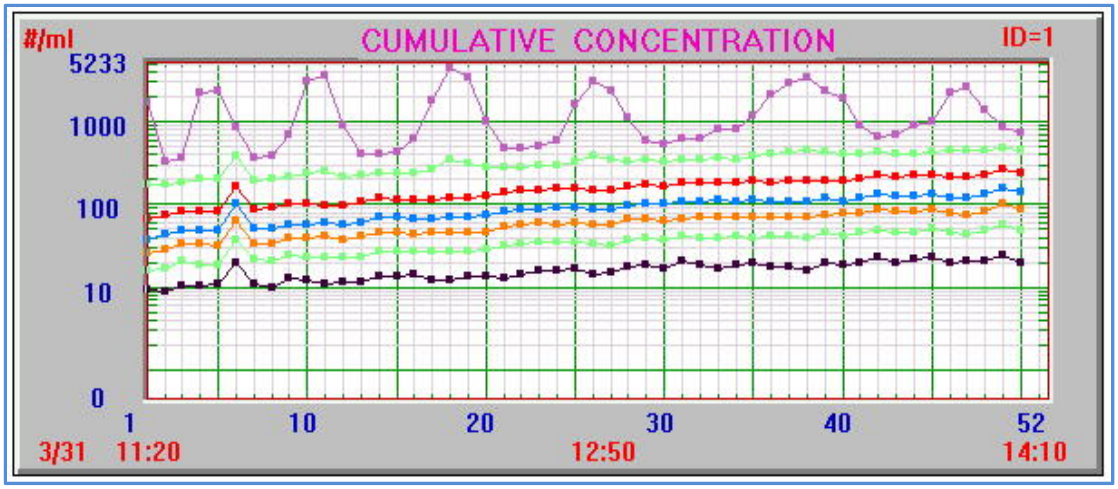
CMP Filtration Monitoring

- Incoming slurry supply line
- At point of use
- Change filters based on particle count, not just on expected life time = better yield, lower cost

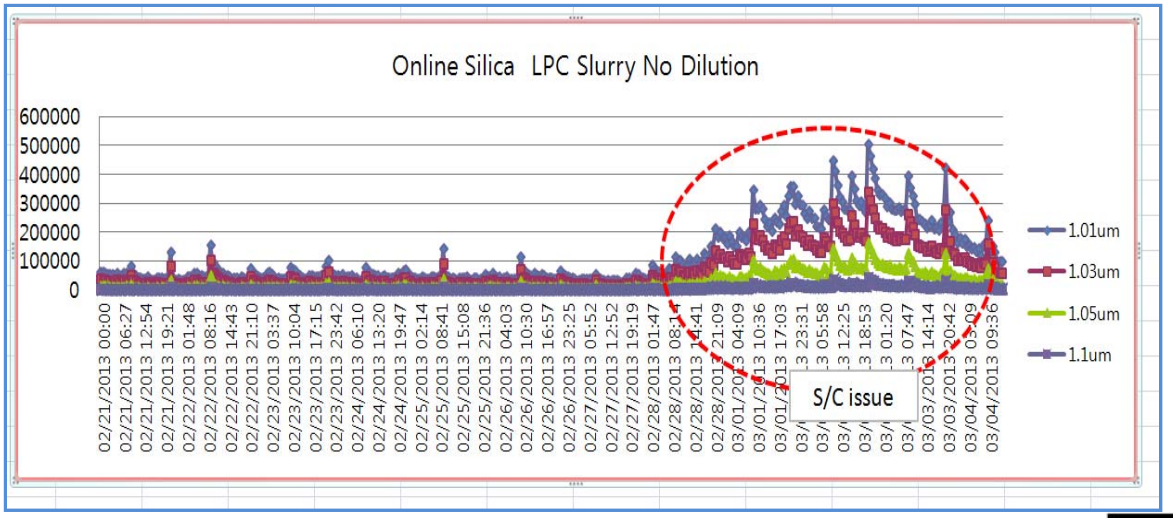




Effect of Pumping (Shear Stress)



Increased circulation



S/C = scratches

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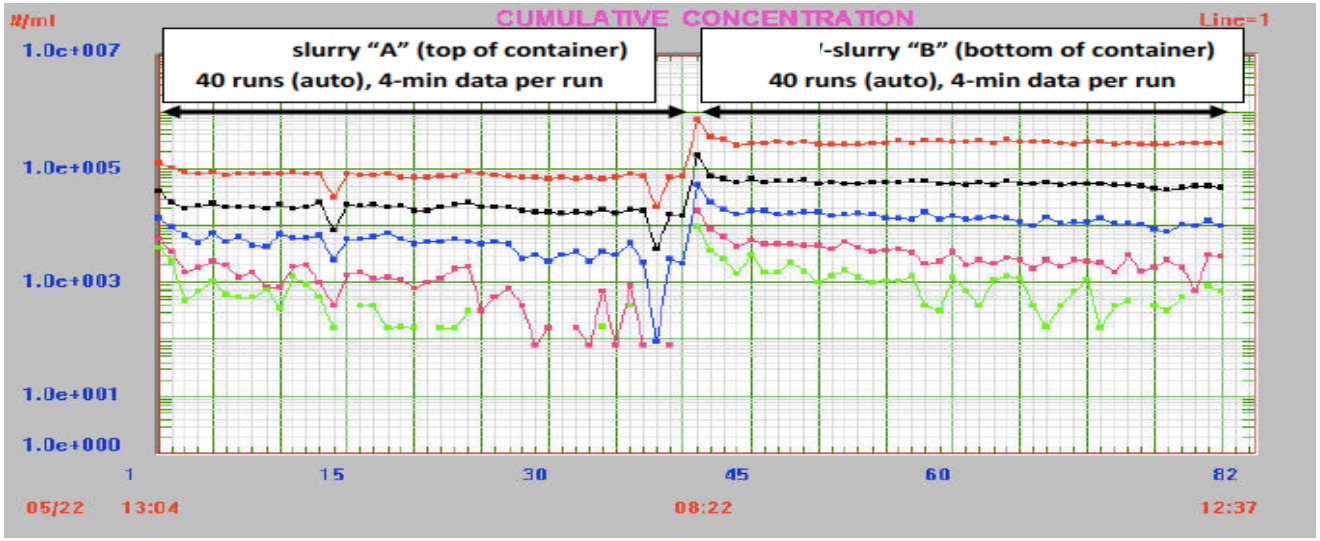
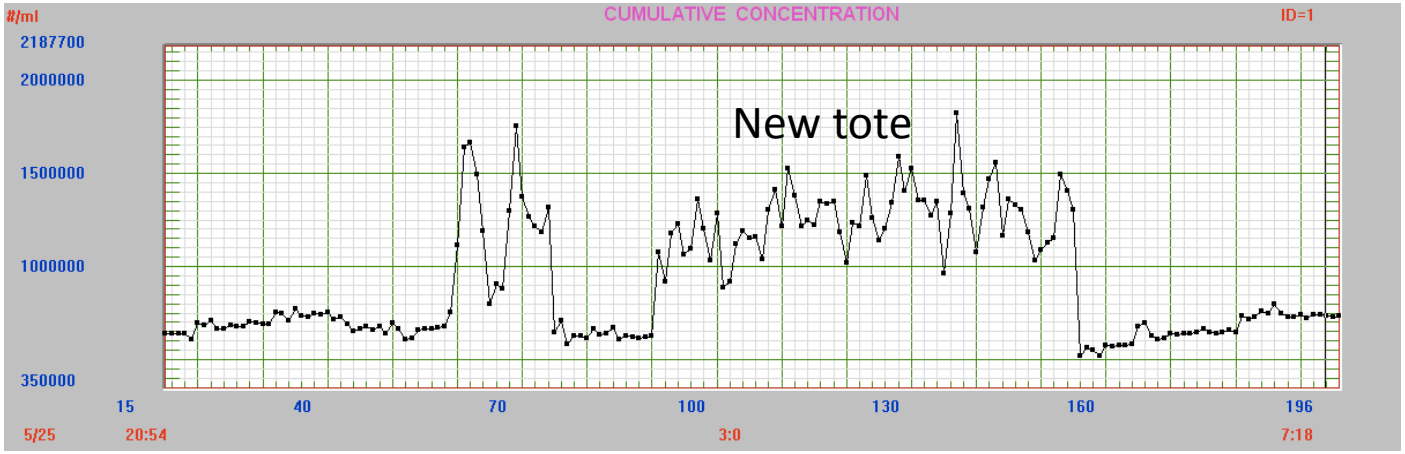
Effect of Pumping (Sedimentation)

- Top vs. bottom of tank
- Supply line LPC > 1.01 µm
- Online FX results



- From day tank



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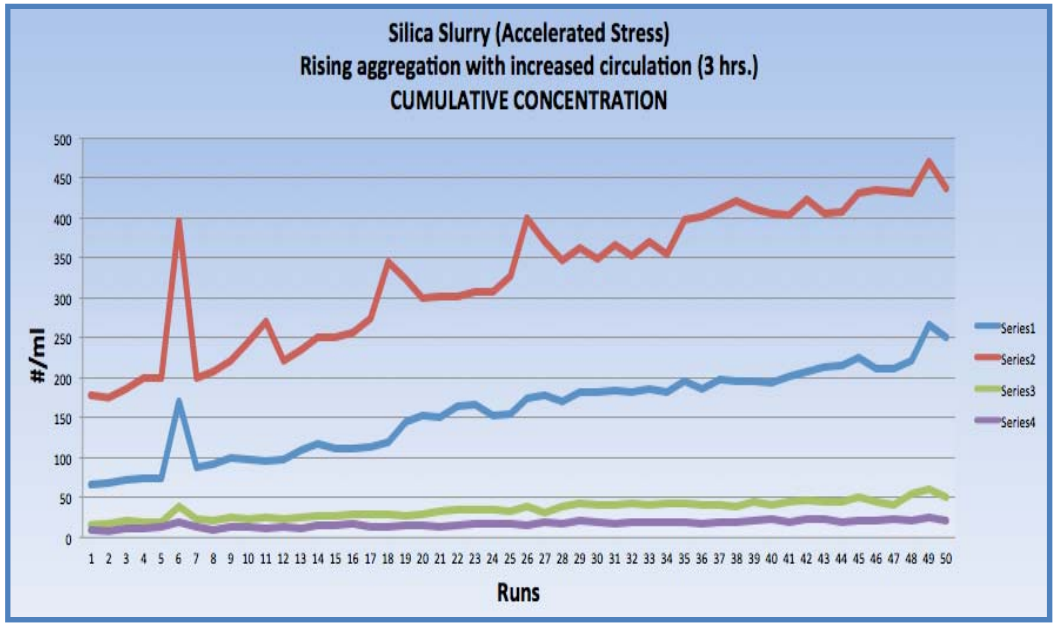
Effect of Slurry Pumping (in the Tool)

- Real time monitoring of LPC counts downstream of a peristaltic pump provides feedback on pump performance and generated LPCs.



Monitoring LPC Counts of Silica Slurry in a Loop

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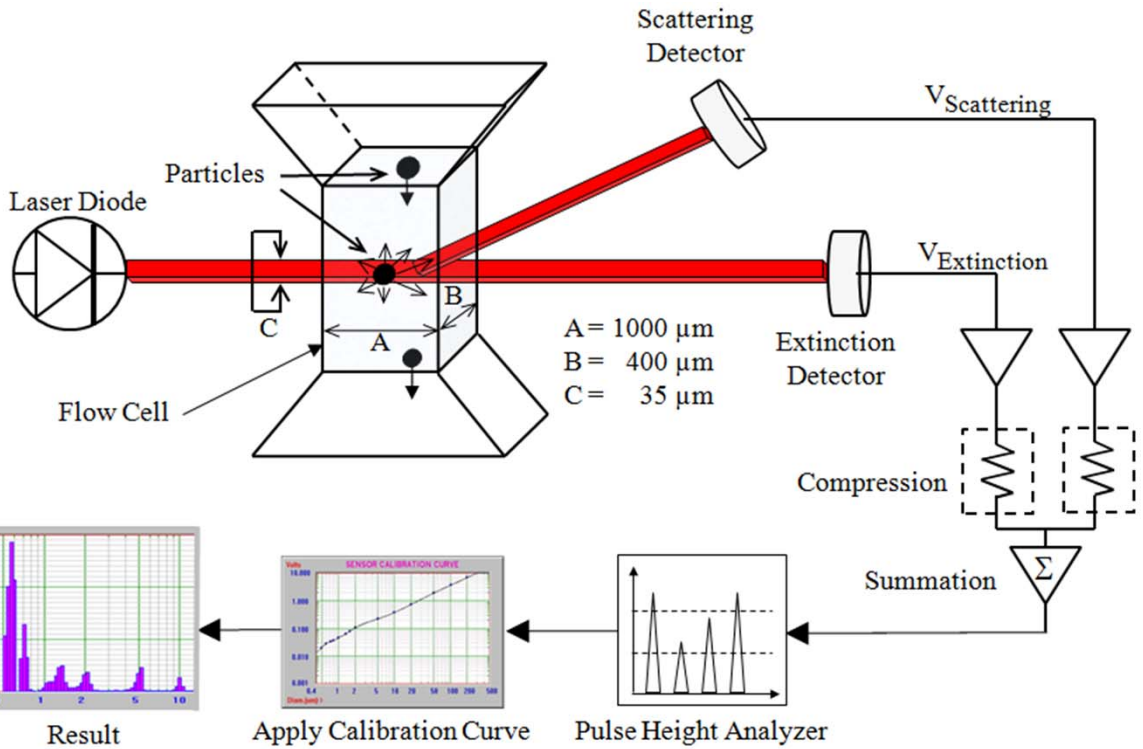
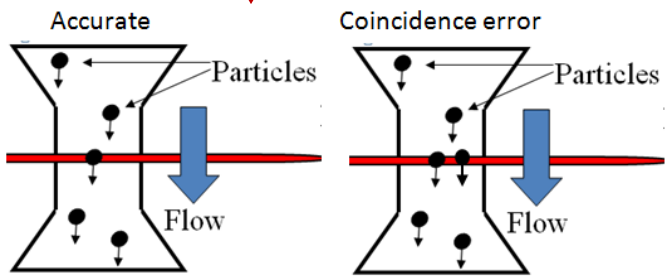
No Dilution? Avoid Coincidence Errors

- Must avoid coincidence error for best data
- Only one particle in measurement zone
- Collimated beam: 0.5 - 400 µm 60 ml/min
- 10,000 particles/mL





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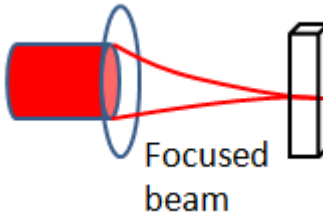
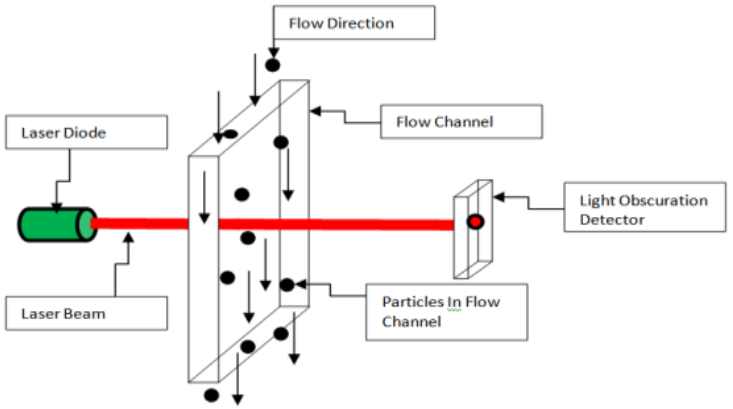
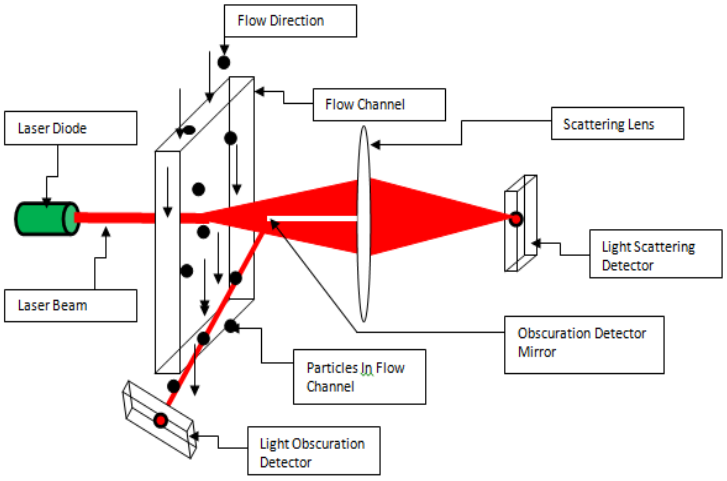
Advancement: Focused Beam

- Focused beam
- ~10^6 particles/mL
- 10^3 improvement
- FX: 0.7- 20 µm
- FX Nano .15 – 10 µm
- But dilution may still be required to avoid coincidence







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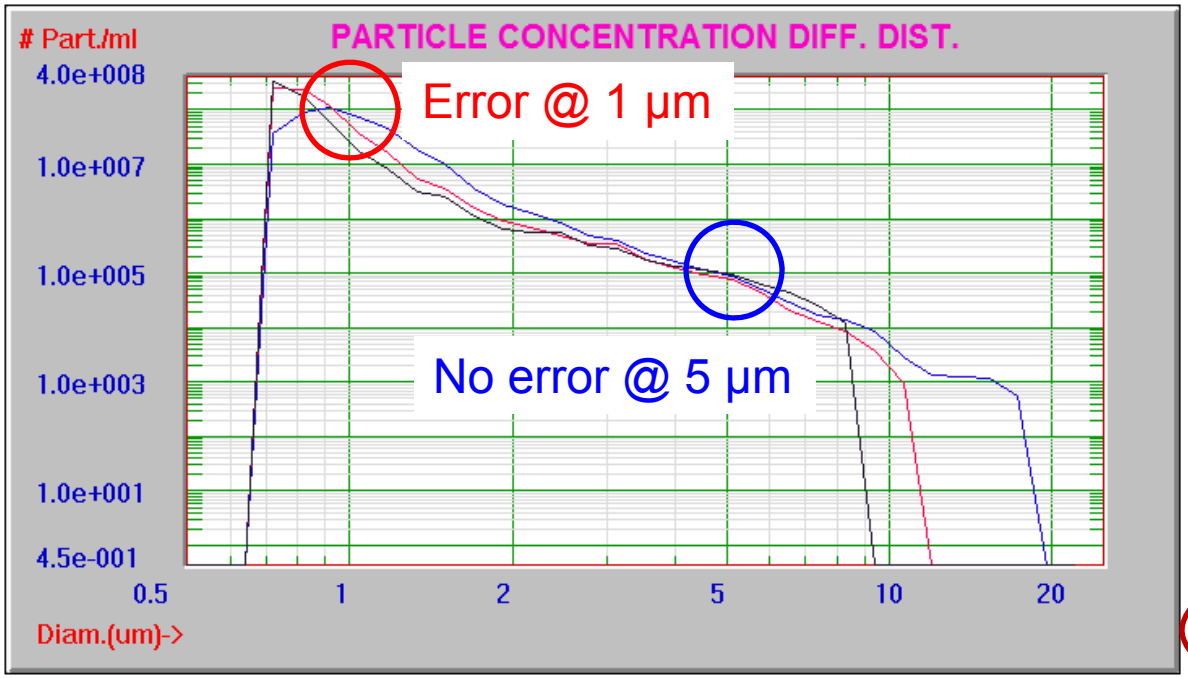
Check for Coincidence Error

- Measure at several different concentrations (dilutions)
- Should have linear response @ size of interest



05/05/2016 13:19

Sensor File:
1211FX42.sns

Sensor Model:
FX20-06

Sensor S/N:
1211FX42

Sample Time:
120 Sec

Fluid Volume:
30.00 ml

Threshold:
0.72 um

Total # Sized:
* 704504

ceriaA_0023.fxe ceriaA_0012.fxe ceriaA_0002.fxe

Mean	Stnd Dev.	Mode	Median	Concentration	D.F./2nd D.F.
0.85 um	0.21 um (25.1%)	0.77 um	0.77 um	6.16E+008 #/ml	267.00 / 267.00

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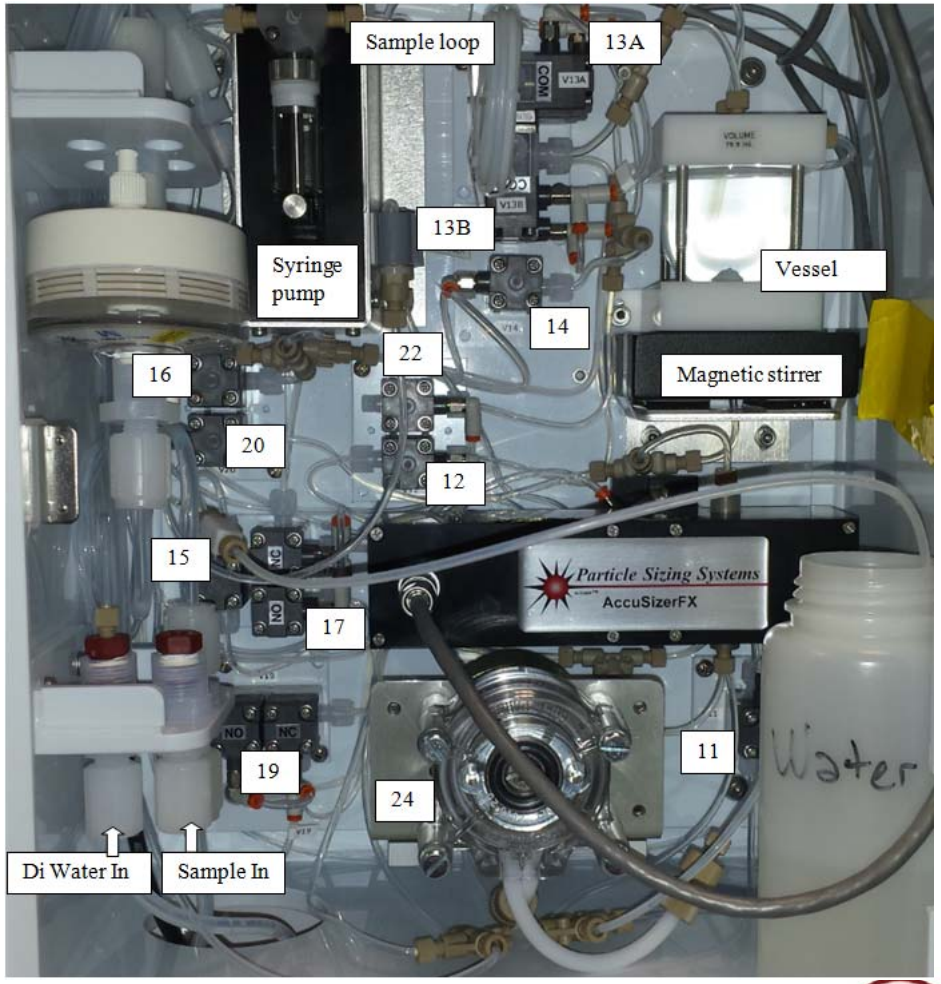
Advancements in Autodilution

2013

2016

Internal Fluidics



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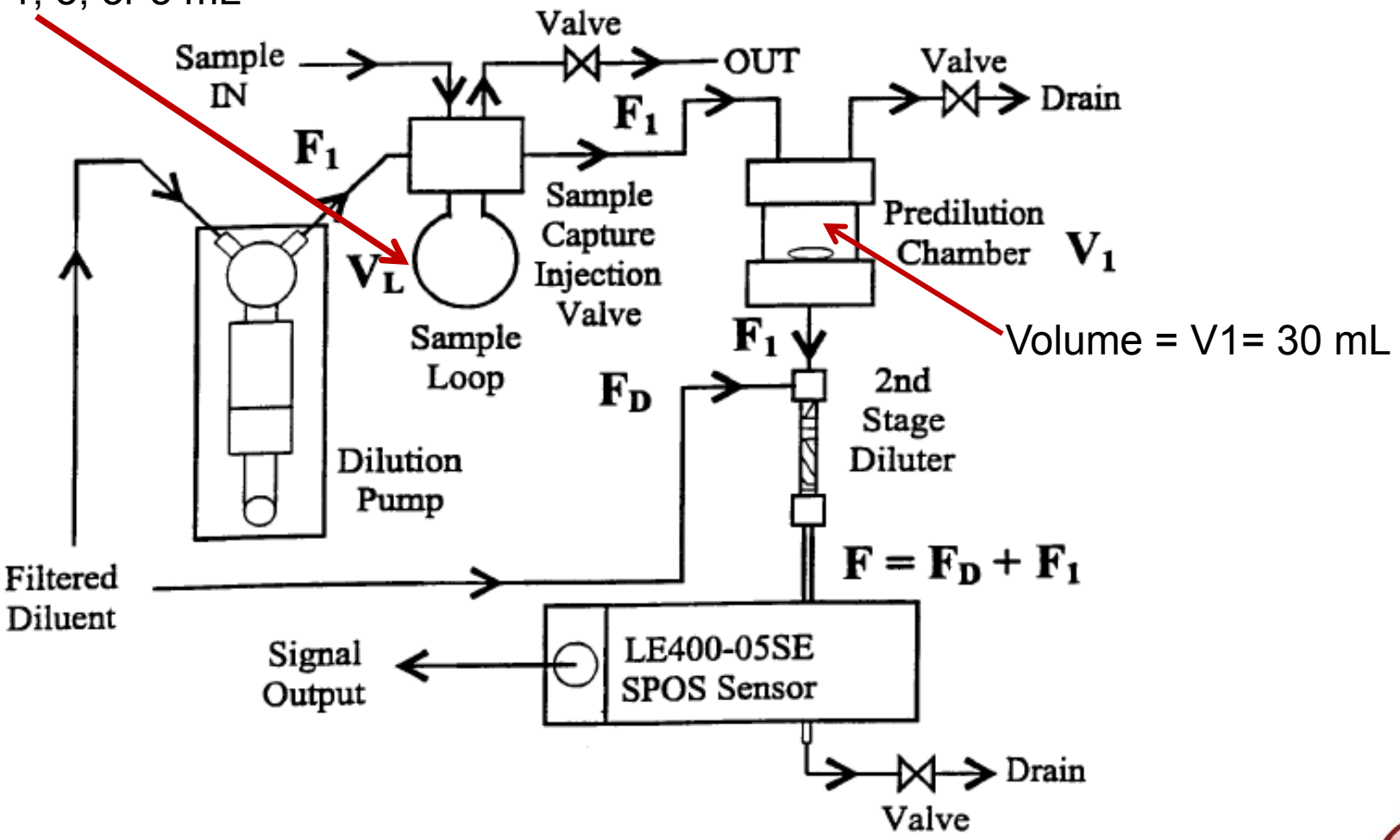
Dilution Fluidics: 2 Stage Dilution

Variable sample loop volume
= VL= 1, 3, or 5 mL



Volume = V1= 30 mL

Goodbye coincidence errors

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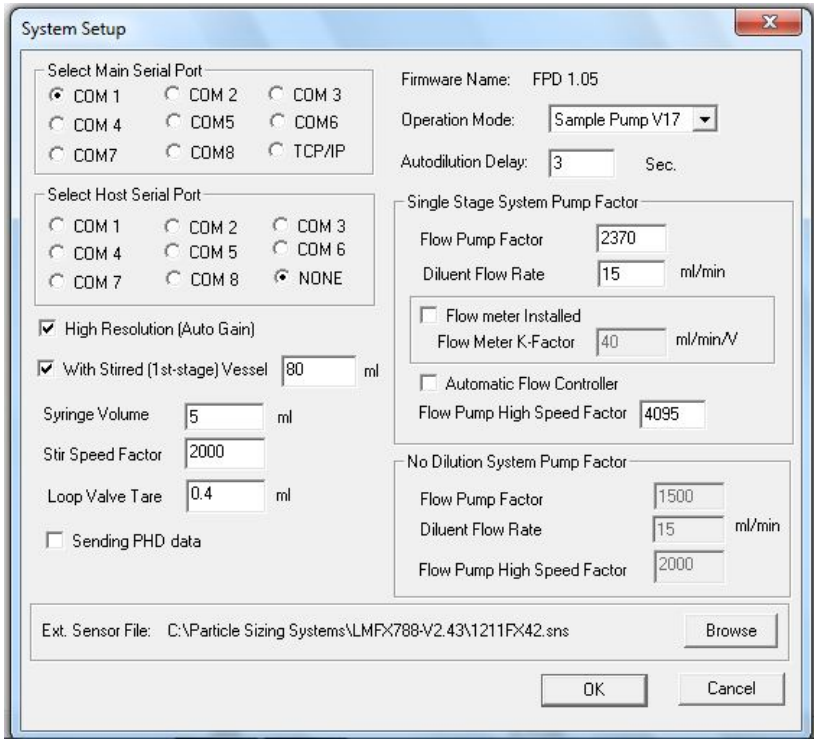
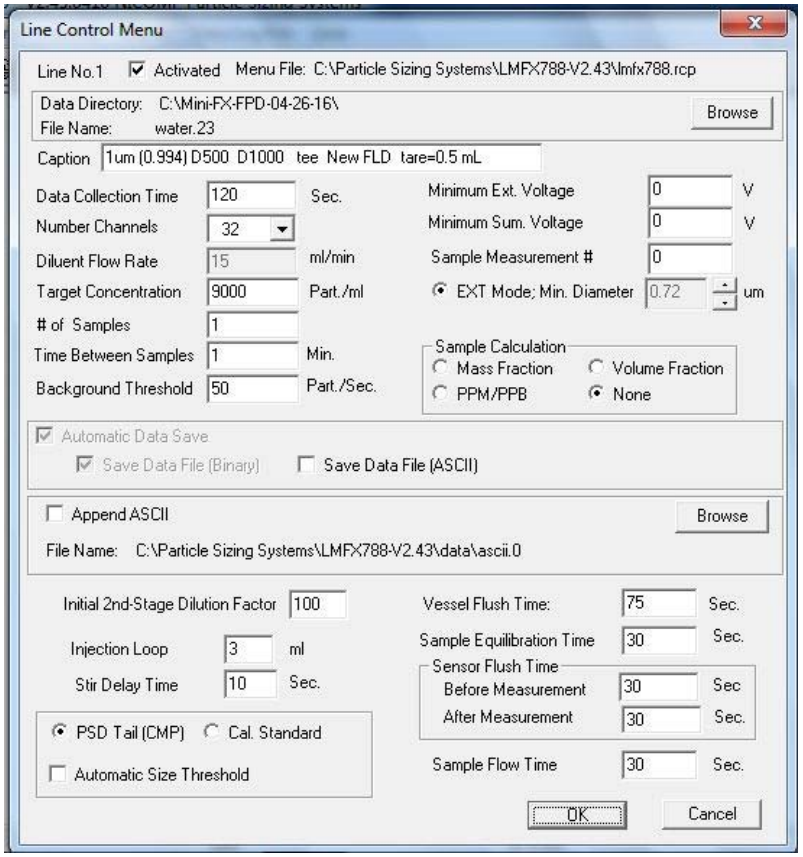
Tailor Measurement to Slurry





Plus many controls fixed in firmware
Parameters saved for each slurry

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Distribution Statistics: Standard Error

$SE = s/(\sqrt{n})$ where

s = standard deviation

n= number of particles counted

So $n = s^2/SE^2$

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

$n = 2^2/ (0.02)^2 = 10{,}000$

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ISO 13322-1 Approach*

Table A.2 — Number of particles required n^*, δ = 0,05, P = 0,95, (u = 1,96)

δ	σ_g	$n^*(D_{MM})$	n^*(Sauter)	$n^*(D_{MV})$
0,05	1,10	585	389	131
	1,15	1 460	934	294
	1,20	2 939	1 808	528
	1,25	5 223	3 103	843
	1,30	8 526	4 920	1 247
	1,35	13 059	7 355	1 750
	1,40	19 026	10 504	2 363
	1,45	26 617	14 457	3 096
	1,50	36 007	19 295	3 956
	1,55	47 358	25 093	4 952
	1,60	60 811	31 919	6 092

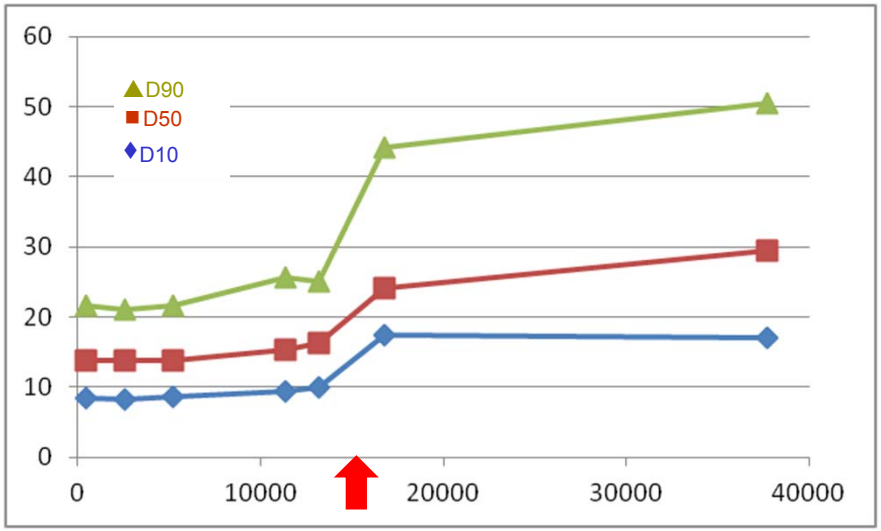
*n** with admissible error of 5% as a function of the geometric standard deviation σGSD of the sample. Here, the probability, *P*, is taken as *P* = 0.95

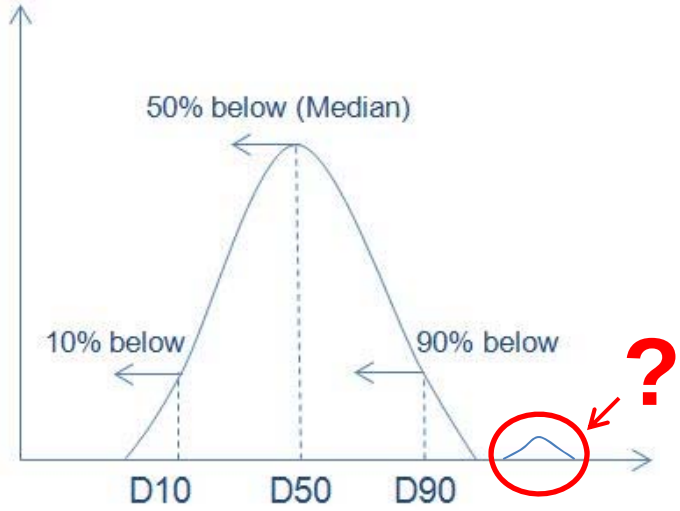
*ISO 13322-1, Particle Size Analysis—Image Analysis Methods—Part 1: Static Image Analysis Methods, available at www.iso.org

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*

Experimental Confirmation

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





NEED to count tens of thousands, preferably hundreds of thousands of particles to achieve statistical accuracy at tails of the distribution (LPC)

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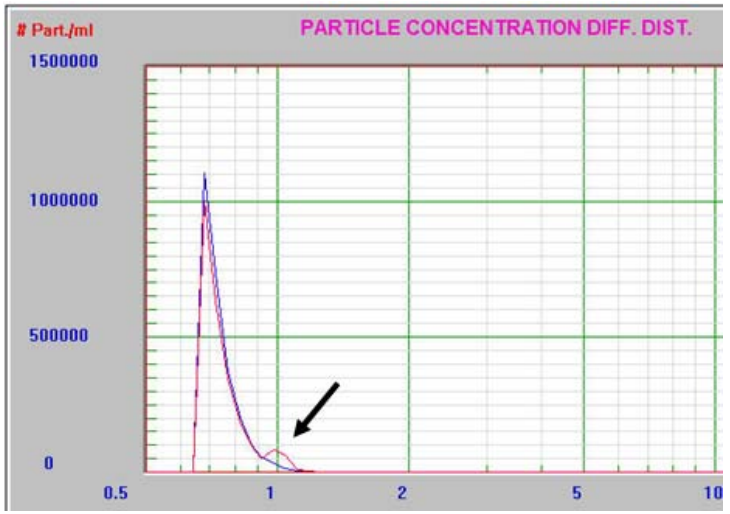
LPC Sensitivity

- Spike silica slurry w/1 µm PSL standard
- Dilute slurry A 200:1
- Add 1.44 mL diluted PSL stock at 1.74 × 10^7/mL
- Measure on AccuSizer FX Point of Use system
- Note increase of ~100,000 particles/mL @ expected size = excellent recovery



Diameter Range (microns)		# Part. Sized (# / ml)	Cum Num >=Diam. (# / ml)
0.70 –	0.72	1107410	2637543
0.72 –	0.77	738493	1530133
0.77 –	0.82	372885	791640
0.82 –	0.87	203005	418755
0.87 –	0.92	100429	215749
0.92 –	0.98	54625	115320
0.98 –	1.04	32665	60695
1.04 –	1.11	13392	28030
1.11 –	1.18	7375	14639
1.18 –	1.25	4147	7264
1.25 –	1.33	1762	3117
1.33 –	1.41	709	1355
1.41 –	1.50	213	645
1.50 –	1.60	60	432
1.60 –	1.70	88	372

Diameter Range (microns)		# Part. Sized (# / ml)	Cum Num >=Diam. (# / ml)
0.70 –	0.72	1005006	2484223
0.72 –	0.77	633343	1479216
0.77 –	0.82	343434	845874
0.82 –	0.87	183631	502439
0.87 –	0.92	96589	318808
0.92 –	0.98	53409	222219
0.98 –	1.04	82906	168810
1.04 –	1.11	64733	85904
1.11 –	1.18	11916	21172
1.18 –	1.25	4192	9256
1.25 –	1.33	2477	5064
1.33 –	1.41	939	2587
1.41 –	1.50	371	1647
1.50 –	1.60	171	1276
1.60 –	1.70	0	1105

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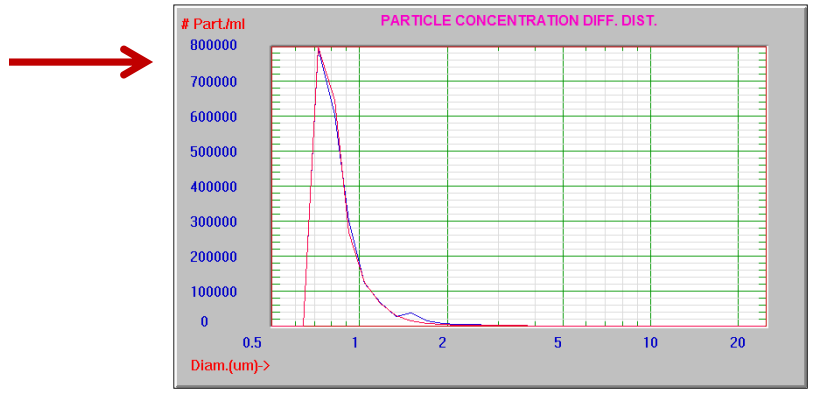
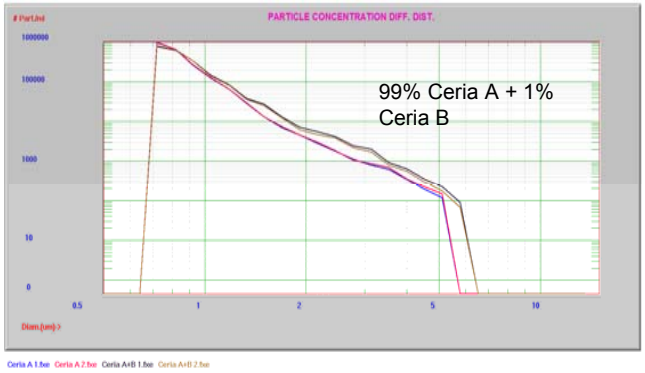
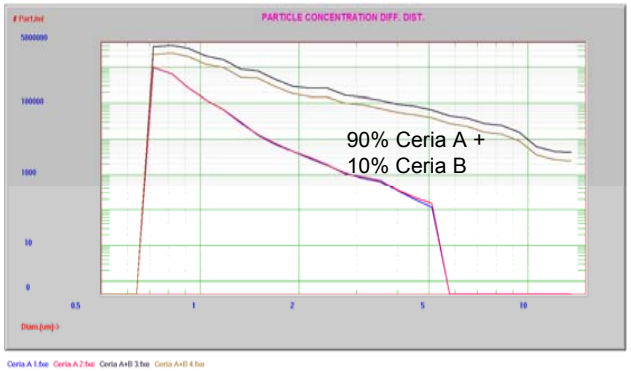
Ceria CMP Slurry

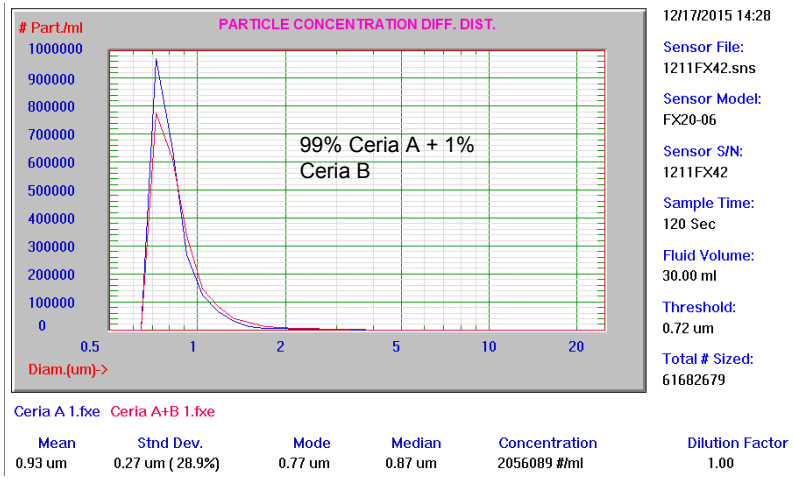
- Spike ceria A slurry w/ 10^6/mL 1 µm PSL



- Mix Slurry A & B







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Conclusions

- It's worth the investment: increased yields
- Point of Use monitors detect LPCs where they cause troubles
- Need autodilution, tailored to the slurry
- Need to measure 10^4 particles to always find LPCs
- Can't beat statistics
  - Sorry, you won't win this jackpot

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