

Advances in LPC* Monitoring in CMP Slurries

Statistics are a Bear



Mark Bumiller
Particle Sizing Systems

(*LPC = Large Particle Counts, typically $> 1 \mu\text{m}$)

Particle Sizing Systems

Building solutions one particle at a time.



LPC vs. Defects/Scratches (Yield)*

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

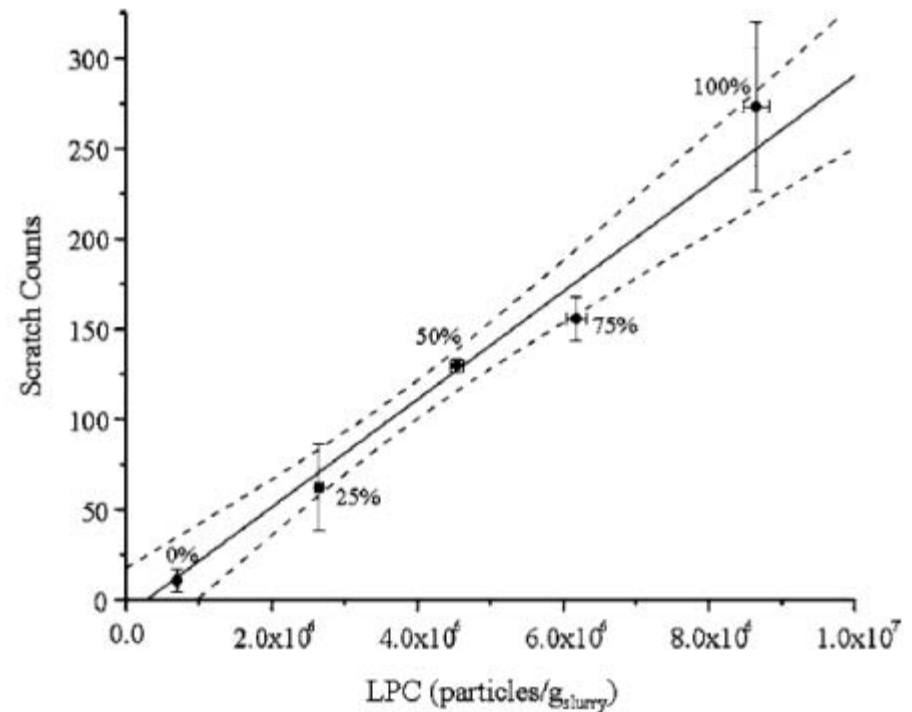
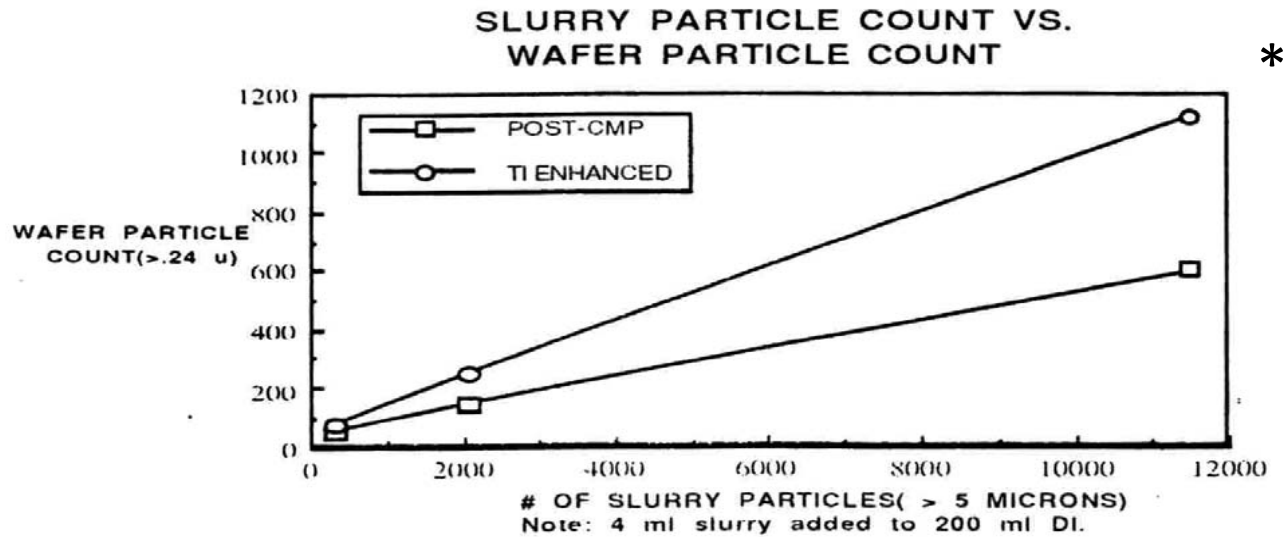


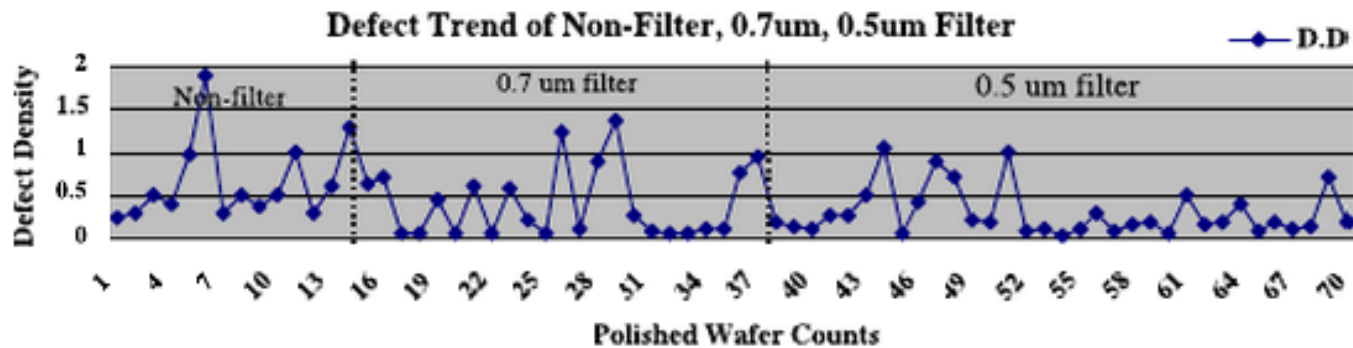
Figure 1. Correlation between scratch counts and LPC determined for particles with diameter $\geq 0.469 \mu\text{m}$ (S03 + S05) with mixtures of slurry A (0 wt % slurry B) and slurry B. Weight% slurry B for the mixtures is labeled at the corresponding data point. The weighted linear regression fit to 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)

LPC Correlates to Yield



*



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* 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

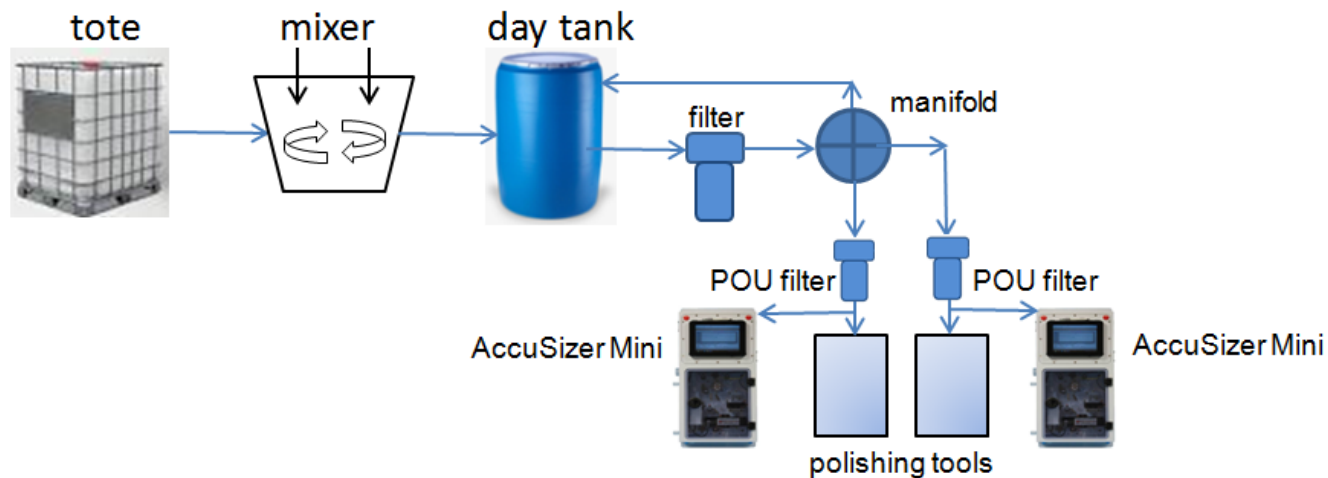
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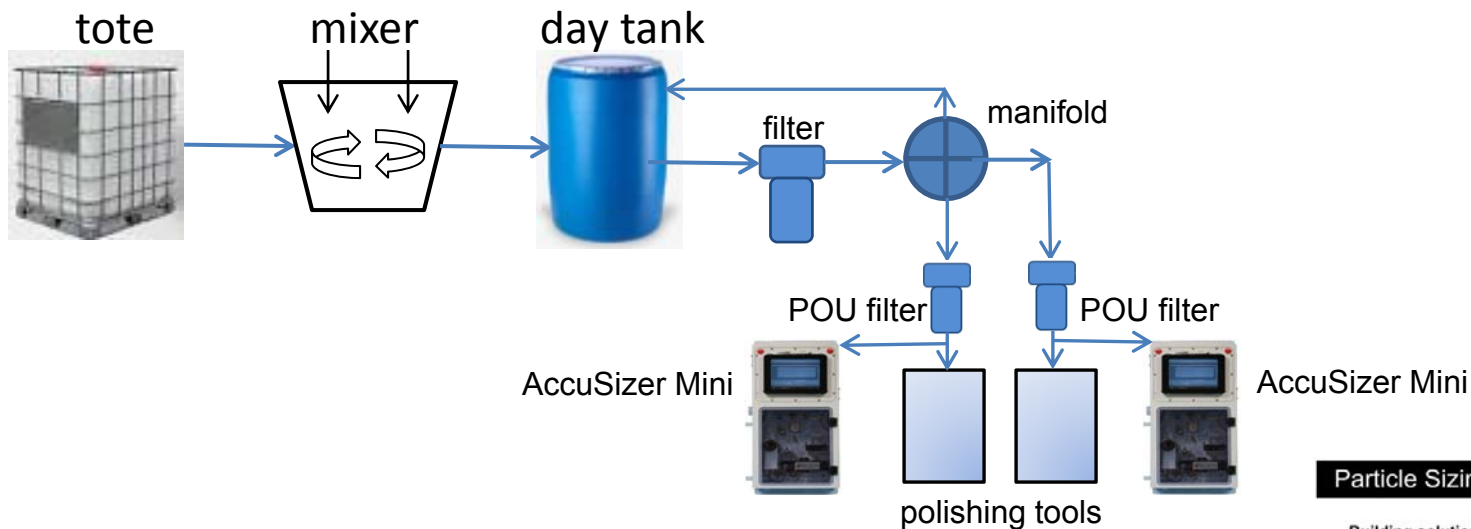
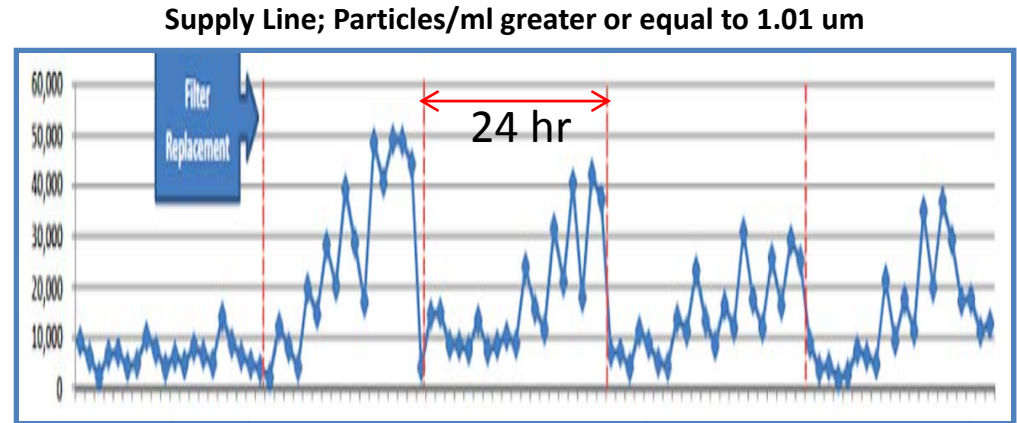
The Wish

- 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



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

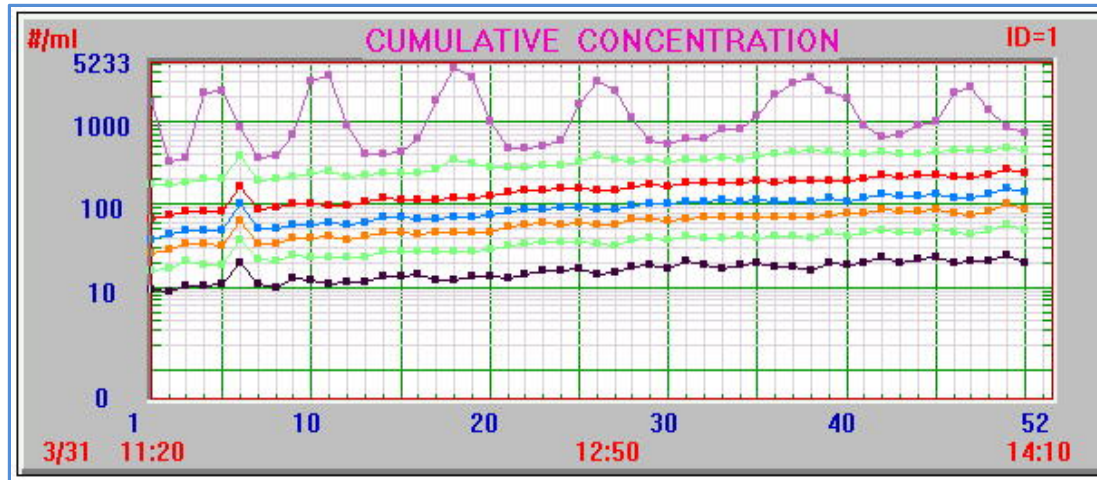


Particle Sizing Systems

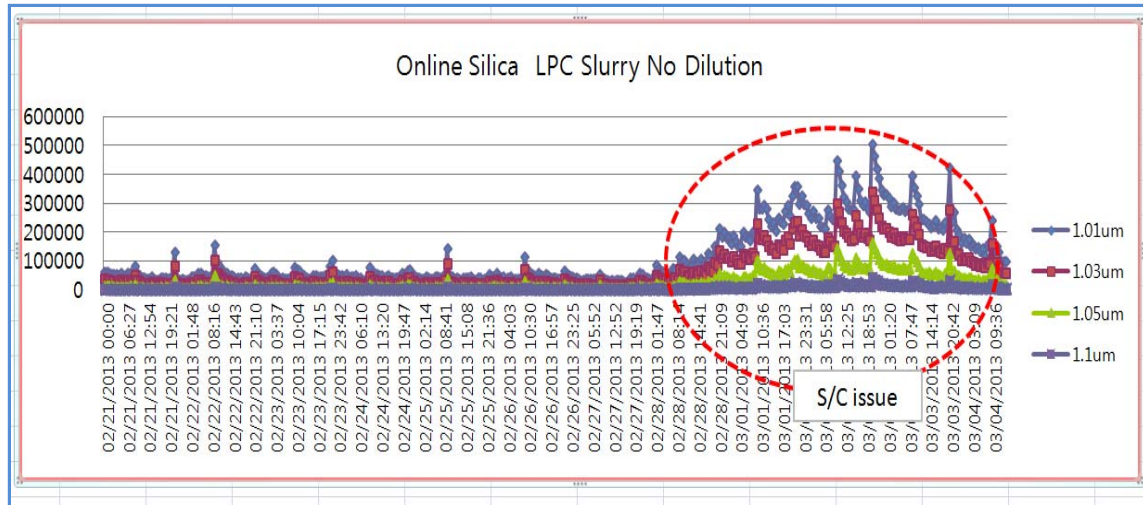
Building solutions one particle at a time.



Effect of Pumping (Shear Stress)



Increased circulation

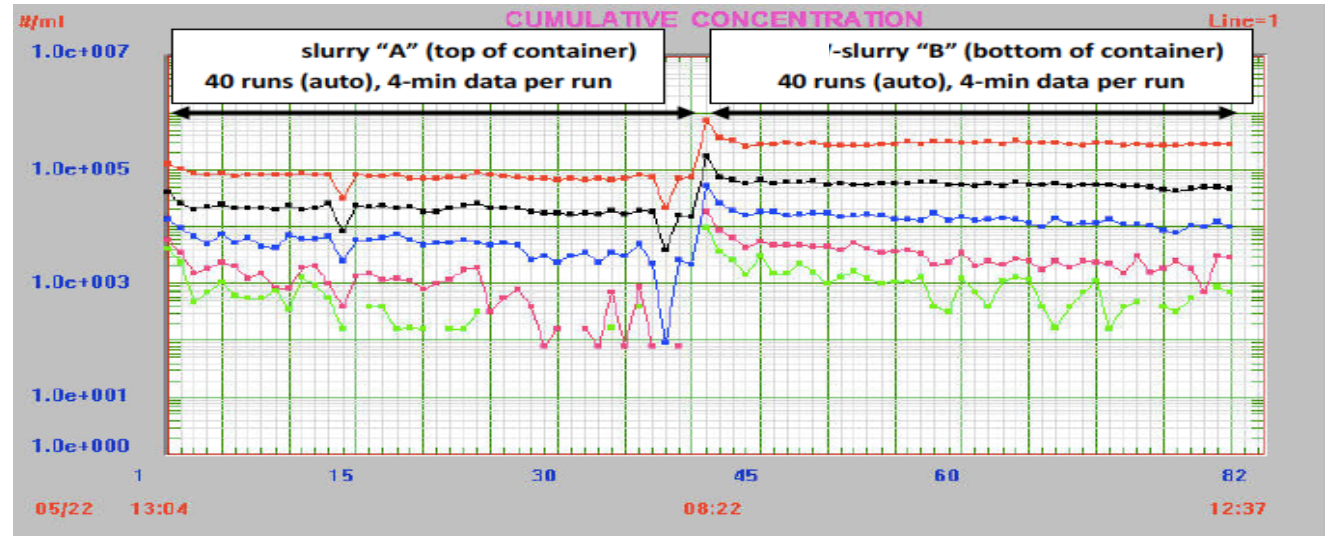


S/C = scratches

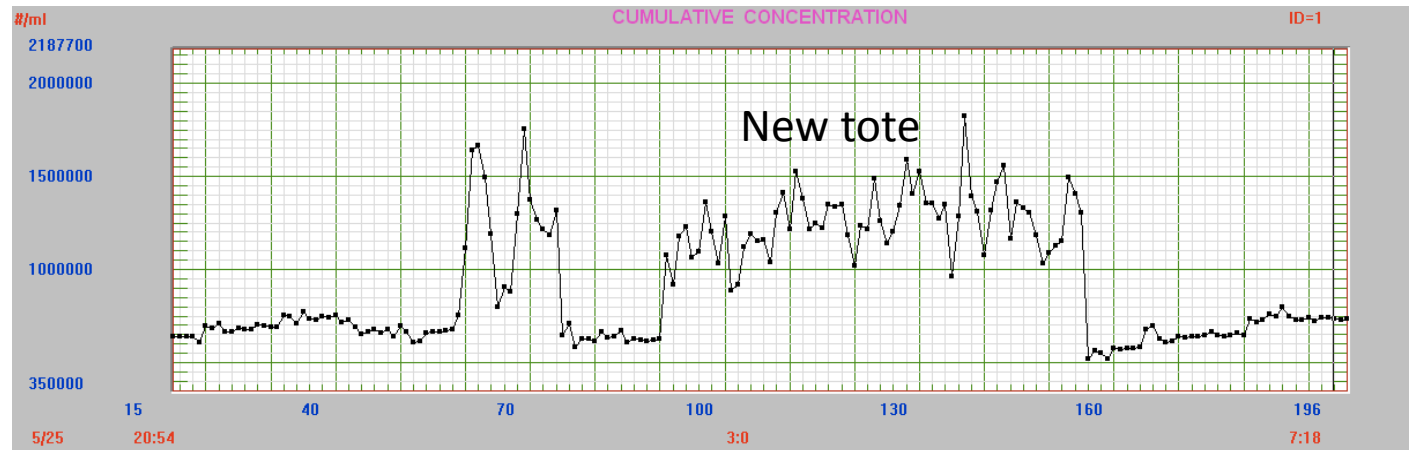


Effect of Pumping (Sedimentation)

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

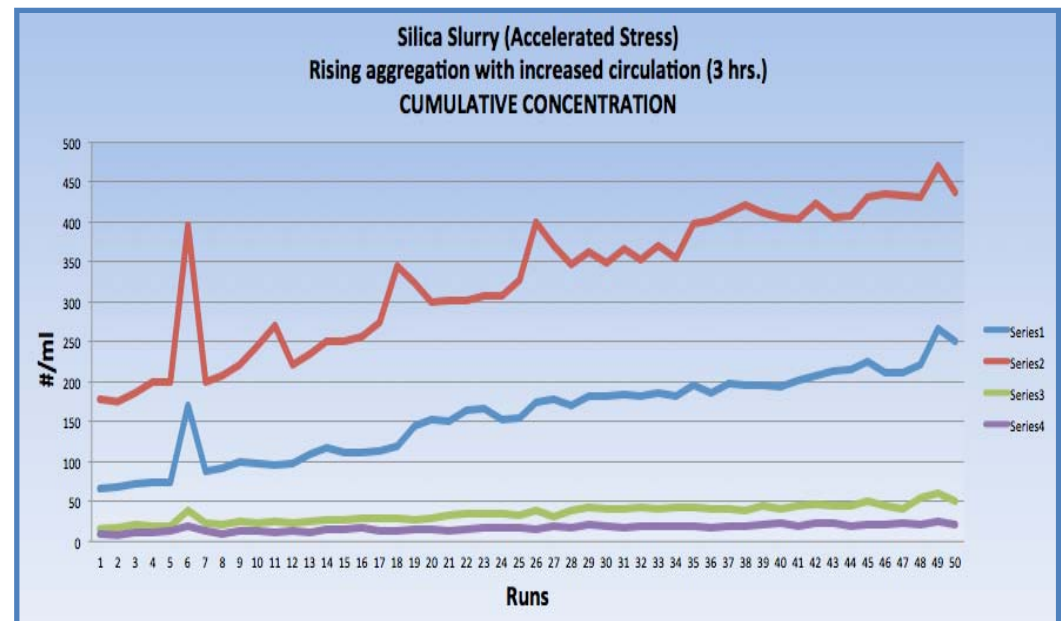


- From day tank



Effect of Slurry Pumping (in the Tool)

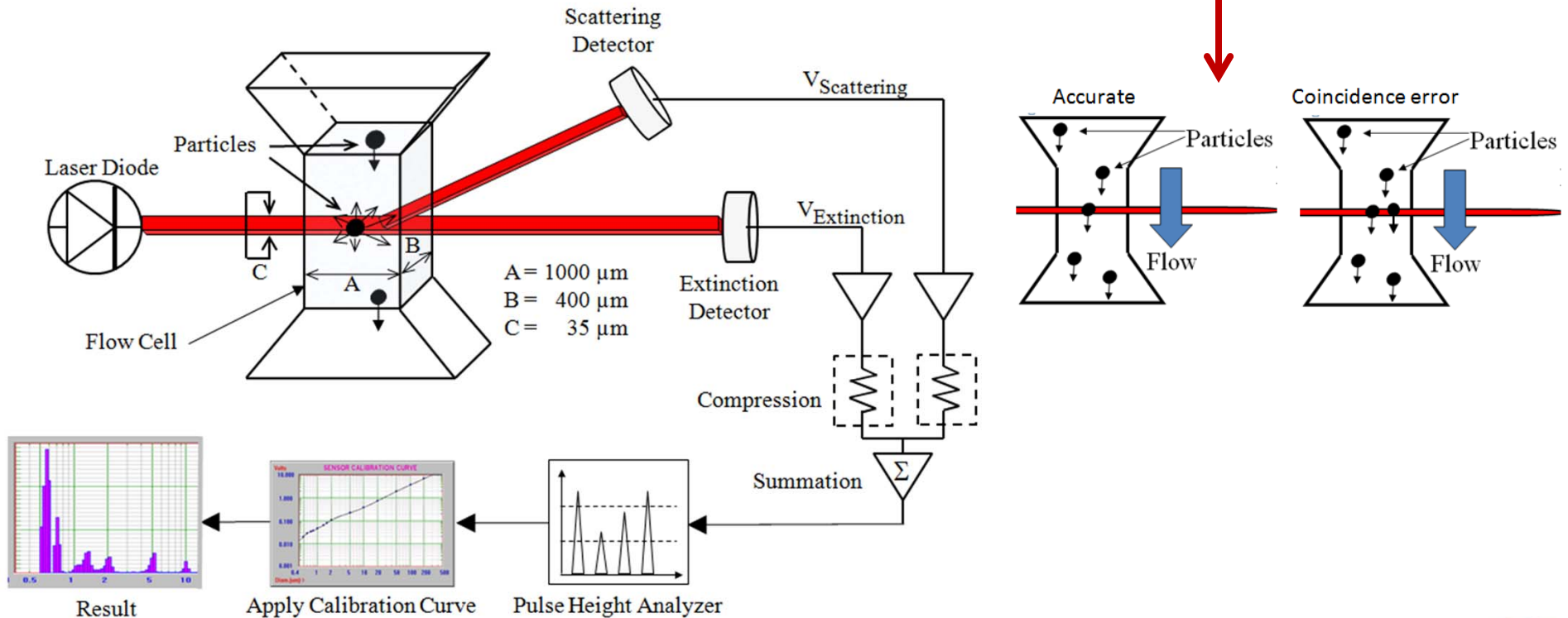
- Real time monitoring of LPC counts downstream of a peristaltic pump provides feedback on pump performance and generated LPCs.



Monitoring LPC Counts of Silica Slurry in a Loop

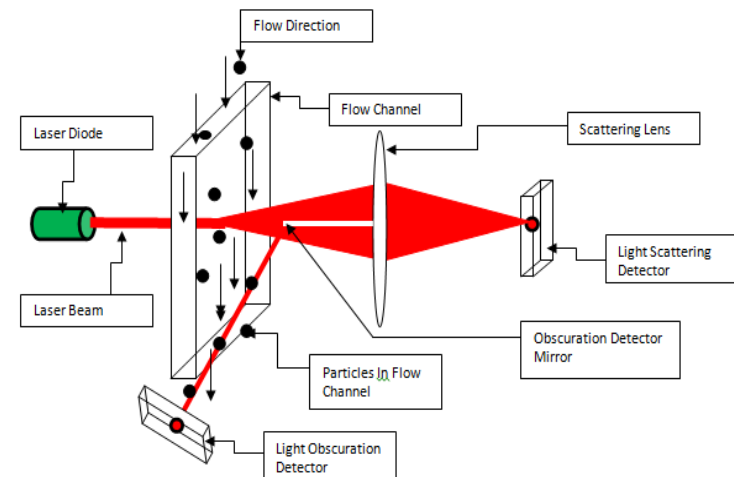
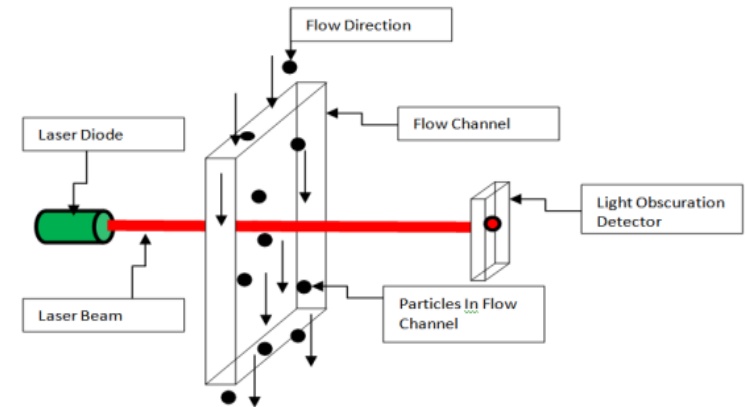
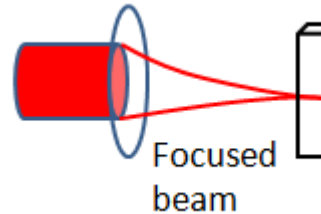
No Dilution? Avoid Coincidence Errors

- 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



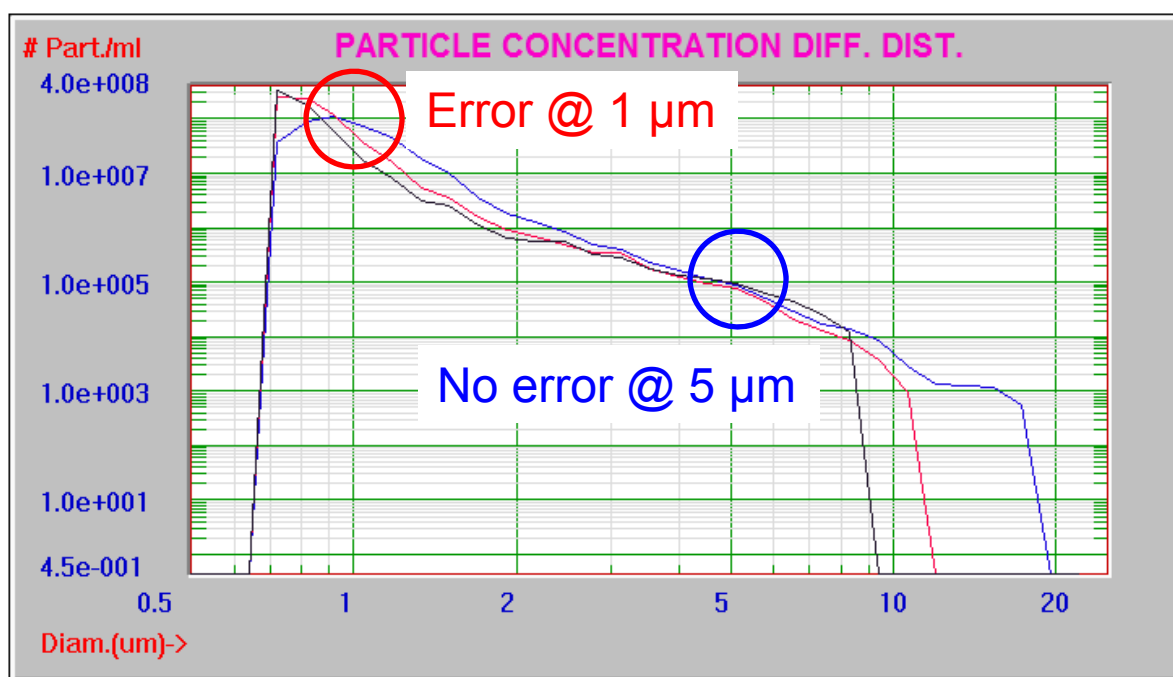
Advancement: Focused Beam

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



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 μm

Total # Sized:
* 704504

ceriaA_0023.fxe ceriaA_0012.fxe ceriaA_0002.fxe

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

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Advancements in Autodilution

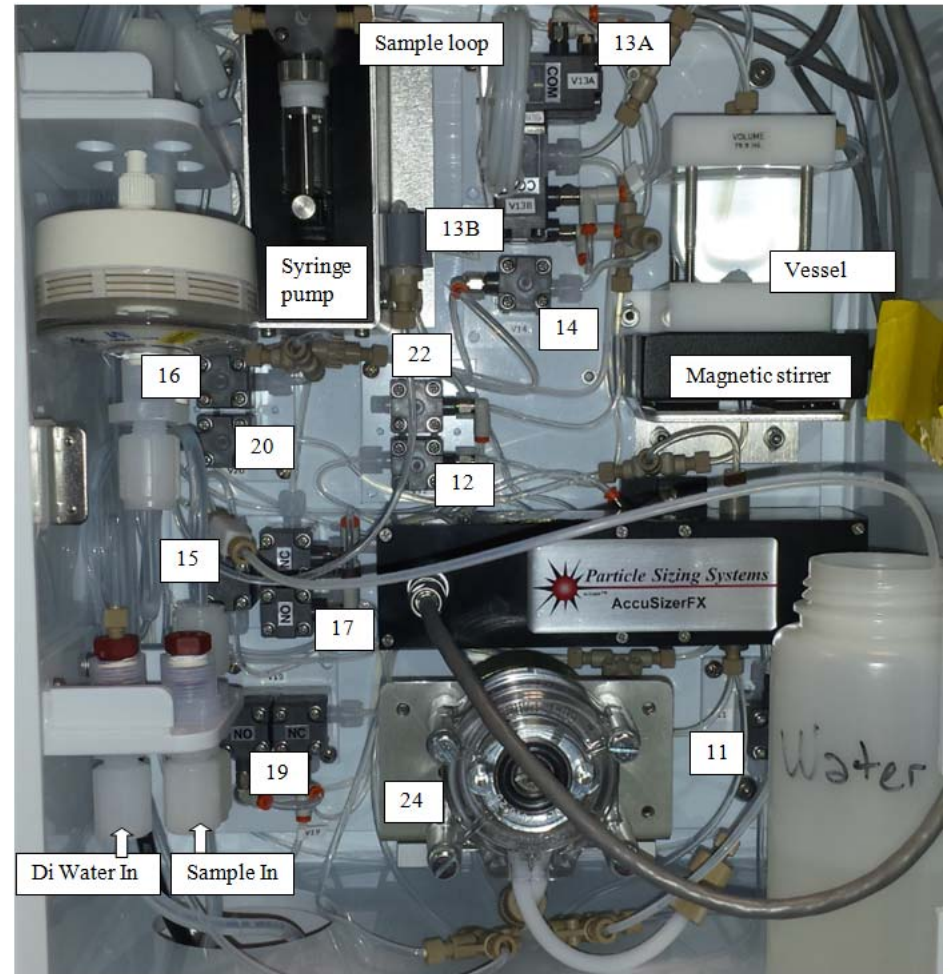
2013



2016



Internal Fluidics



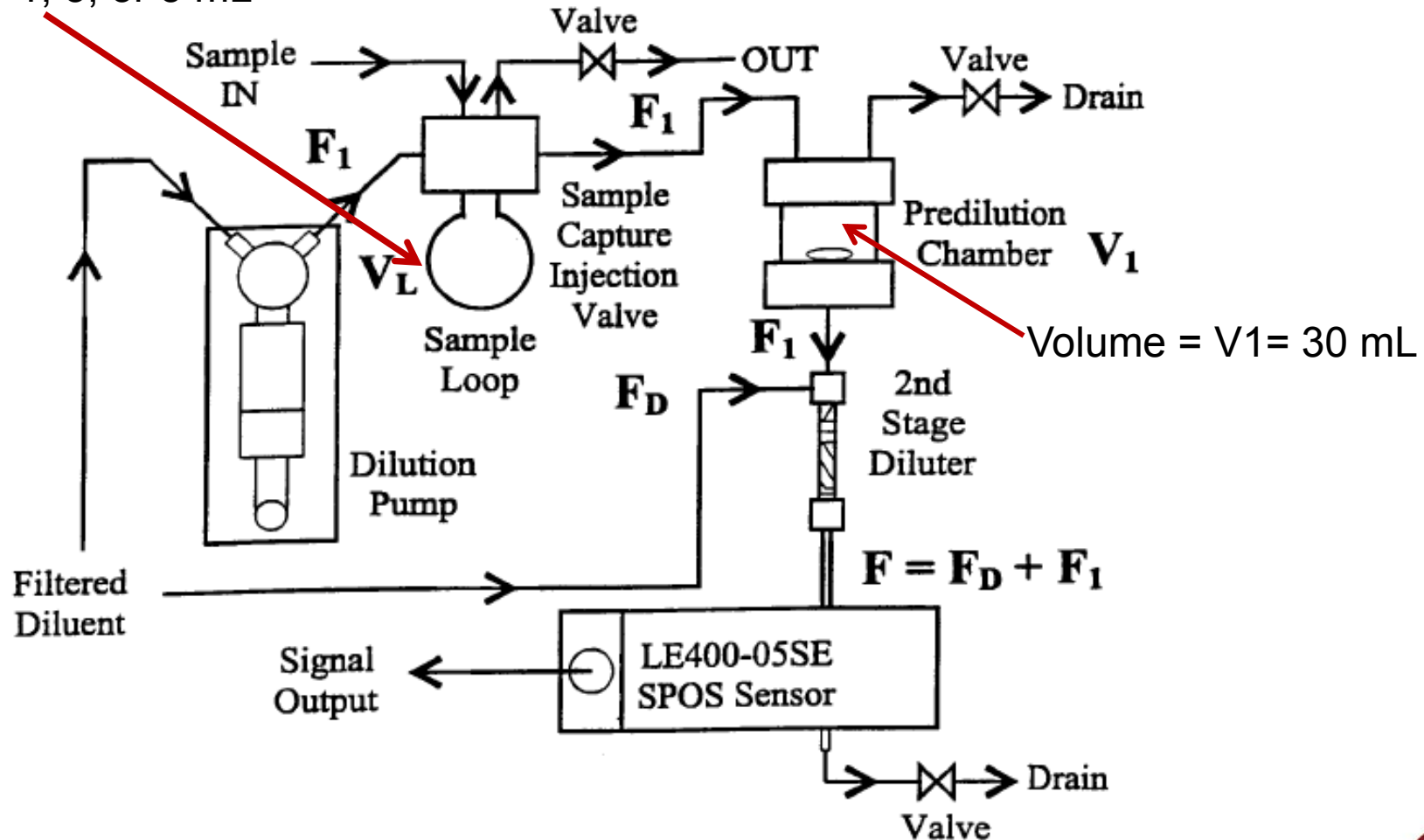
Particle Sizing Systems

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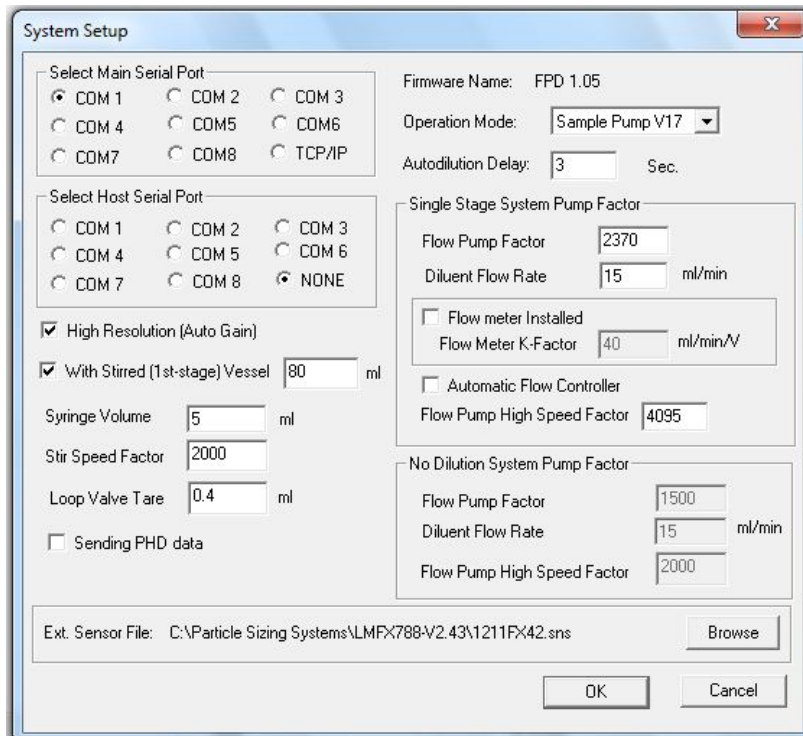
Dilution Fluidics: 2 Stage Dilution

Variable sample loop volume
= $V_L = 1, 3, \text{ or } 5 \text{ mL}$



Goodbye coincidence errors

Tailor Measurement to Slurry



System Setup

Select Main Serial Port:
 COM 1 COM 2 COM 3
 COM 4 COM 5 COM 6
 COM 7 COM 8 TCP/IP

Select Host Serial Port:
 COM 1 COM 2 COM 3
 COM 4 COM 5 COM 6
 COM 7 COM 8 NONE

High Resolution (Auto Gain)
 With Stirred (1st-stage) Vessel 80 ml

Syringe Volume 5 ml
Stir Speed Factor 2000
Loop Valve Tare 0.4 ml
 Sending PHD data

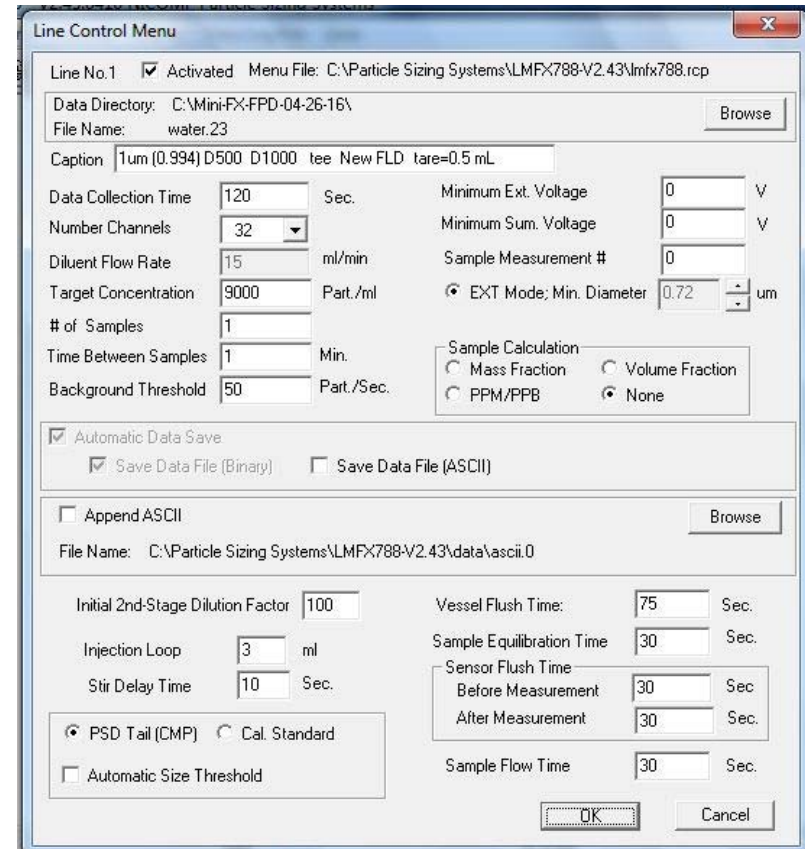
Ext. Sensor File: C:\Particle Sizing Systems\LMFX788-V2.43\1211FX42.sns

Firmware Name: FPD 1.05
Operation Mode: Sample Pump V17
Autodilution Delay: 3 Sec.

Single Stage System Pump Factor
Flow Pump Factor 2370
Diluent Flow Rate 15 ml/min
 Flow meter Installed
Flow Meter K-Factor 40 ml/min/V
 Automatic Flow Controller
Flow Pump High Speed Factor 4095

No Dilution System Pump Factor
Flow Pump Factor 1500
Diluent Flow Rate 15 ml/min
Flow Pump High Speed Factor 2000

OK Cancel



Line Control Menu

Line No.1 Activated Menu File: C:\Particle Sizing Systems\LMFX788-V2.43\lmfx788.rcp

Data Directory: C:\Mini-FX-PPD-04-26-16\
File Name: water.23

Caption: 1um (0.994) D500 D1000 tee New FLD tare=0.5 mL

Data Collection Time 120 Sec. Minimum Ext. Voltage 0 V
Number Channels 32 Minimum Sum. Voltage 0 V
Diluent Flow Rate 15 ml/min Sample Measurement # 0
Target Concentration 9000 Part./ml EXT Mode: Min. Diameter 0.72 um
of Samples 1
Time Between Samples 1 Min. Sample Calculation
 Mass Fraction Volume Fraction
 PPM/PPB None

Automatic Data Save
 Save Data File (Binary) Save Data File (ASCII)

Append ASCII
File Name: C:\Particle Sizing Systems\LMFX788-V2.43\data\ascii.0

Initial 2nd-Stage Dilution Factor 100 Vessel Flush Time: 75 Sec.
Injection Loop 3 ml Sample Equilibration Time 30 Sec.
Stir Delay Time 10 Sec. Sensor Flush Time
Before Measurement 30 Sec.
After Measurement 30 Sec.
Sample Flow Time 30 Sec.

PSD Tail (CMP) Cal. Standard
 Automatic Size Threshold

OK Cancel

Plus many controls fixed in firmware
Parameters saved for each slurry



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:

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



ISO 13322-1 Approach*

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

δ	σ_g	$n^*(D_{MM})$	$n^*(\text{Sauter})$	$n^*(D_{MV})$
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 σ_{GSD} of the sample. Here, the probability, P , is taken as $P = 0.95$

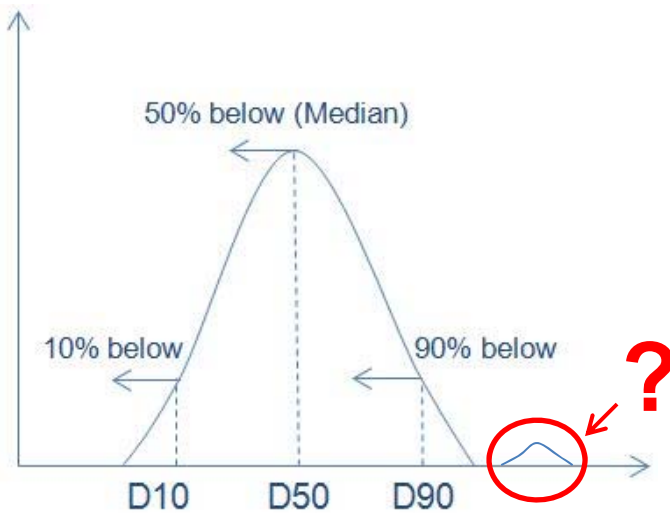
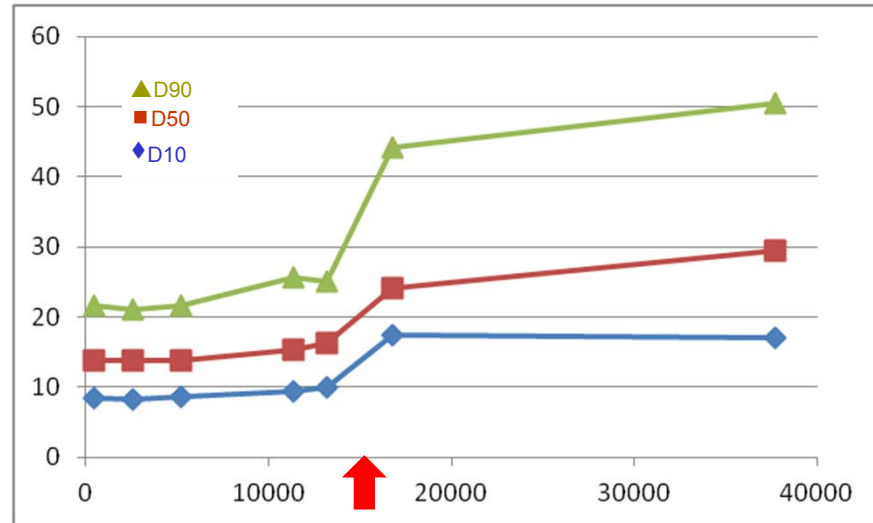
*ISO 13322-1, Particle Size Analysis—Image Analysis Methods—Part 1: Static Image Analysis Methods, available at www.iso.org

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

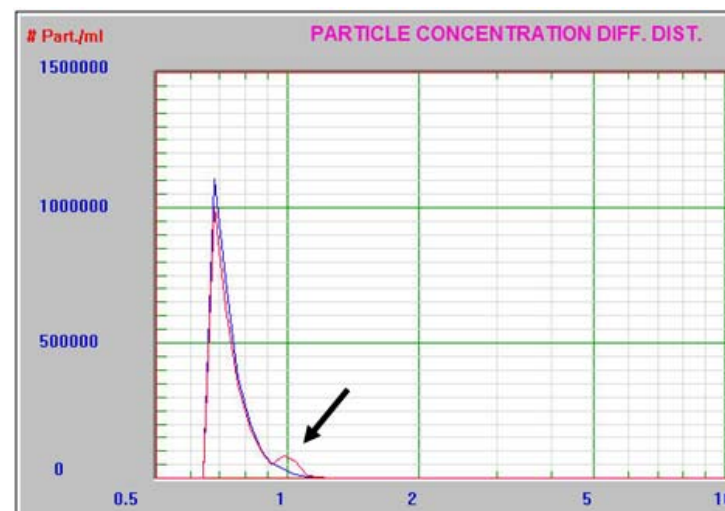
# Sized	D10 (μm)	D50 (μm)	D90 (μm)
442	8.5	13.82	21.65
2579	8.3	13.82	20.99
5213	8.73	13.82	21.71
11364	9.47	15.39	25.63
13196	10	16.24	25.02
16748	17.41	24.07	44.11
37688	17.14	29.42	50.45



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

LPC Sensitivity

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

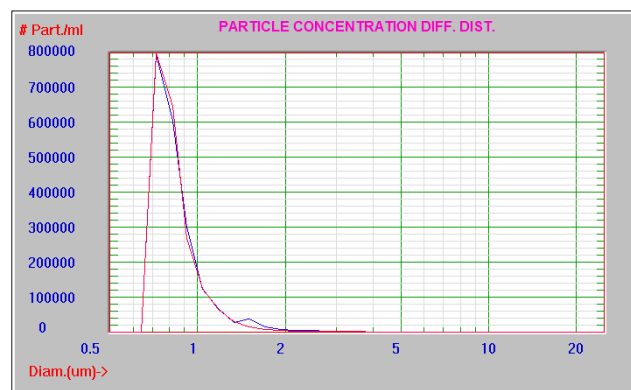


Diameter Range (microns)	# Part. Sized (# / ml)	Cum Num >=Diam. (# / ml)
0.70 – 0.72	1107410	2637543
0.72 – 0.77	738493	1530133
0.77 – 0.82	372885	791640
0.82 – 0.87	203005	418755
0.87 – 0.92	100429	215749
0.92 – 0.98	54625	115320
0.98 – 1.04	32665	60695
1.04 – 1.11	13392	28030
1.11 – 1.18	7375	14639
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

Ceria CMP Slurry

- Spike ceria A slurry w/ $10^6/\text{mL}$ $1 \mu\text{m}$ PSL
- Mix Slurry A & B



12/17/2015 11:15
 Sensor File: 1509FX42.sns
 Sensor Model: FX20-06
 Sensor S/N: 1509FX42
 Sample Time: 120 Sec
 Fluid Volume: 30.00 ml
 Threshold: 0.72 um
 Total # Sized: 64113512
 # > 25.00 um: 163

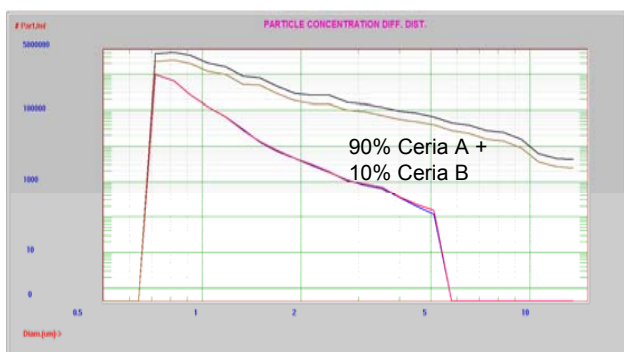
CeriaA +1 μm.fxe Ceria A 1.fxe

Mean	Stnd Dev.	Mode	Median	Concentration
0.89 um	0.29 um (32.1%)	0.77 um	0.87 um	2137117 #/ml

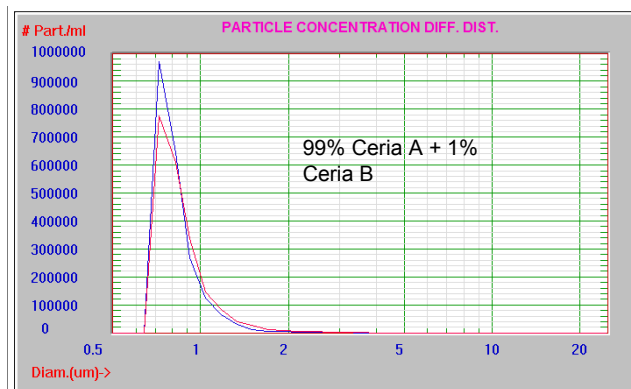
Dilution Factor: 1.00



Ceria A 1.fxe Ceria A 2.fxe Ceria A+B 1.fxe Ceria A+B 2.fxe



Ceria A 1.fxe Ceria A 2.fxe Ceria A+B 3.fxe Ceria A+B 4.fxe



12/17/2015 14:28
 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: 61602679

Ceria A 1.fxe Ceria A+B 1.fxe

Mean	Stnd Dev.	Mode	Median	Concentration
0.93 um	0.27 um (28.9%)	0.77 um	0.87 um	2056089 #/ml

Dilution Factor: 1.00

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Building solutions one particle at a time.



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

Booth 1100
mbumiller@pssnicomp.com



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