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World Leader in Sub-20nm Particle Measurement

A New Method for Determining the Size Distribution of Particles in CMP Slurries

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Presentation Overview

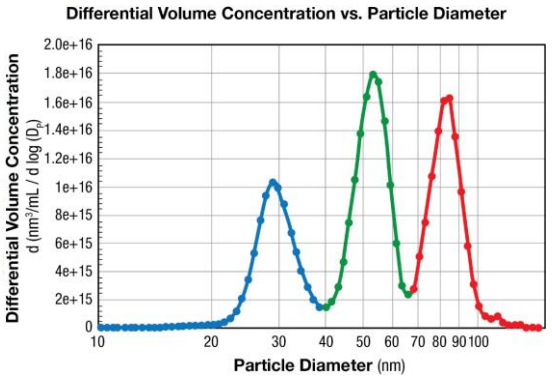
- Liquid Nanoparticle Sizer (LNS) Overview, Specs, and Principle of Operation
- Kanolysis software and parametrized slurry metrics
- LNS Use Cases
  - Engineering - Insight into slurry particle size distributions and direct observation of agglomeration.
  - Operations - Slurry production and quality monitoring tool (outgoing and incoming) with standardized reporting.
- Summary

Liquid Nanoparticle Sizer (LNS)

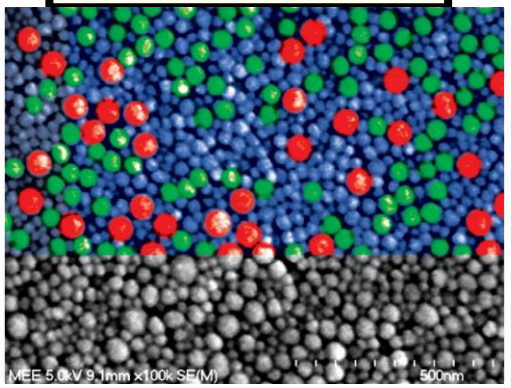
LNS resolves multimodal peaks and provides actual (not relative) concentration

- Current in-situ methods (Dynamic Light Scattering, Laser Diffraction) are limited.
  - Unable to accurately resolve multimodal distributions without a priori knowledge of the sample properties.
  - Cannot provide absolute concentration, only relative concentration.

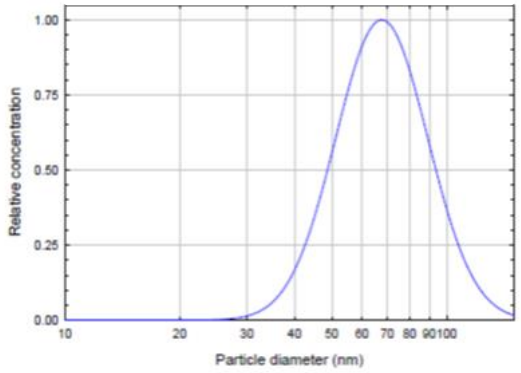
LNS



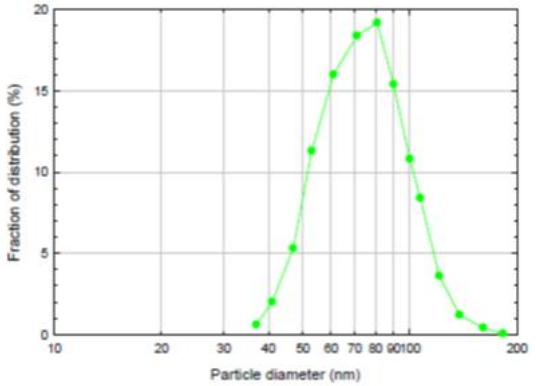
CMP Slurry SEM Image



Dynamic Light Scattering



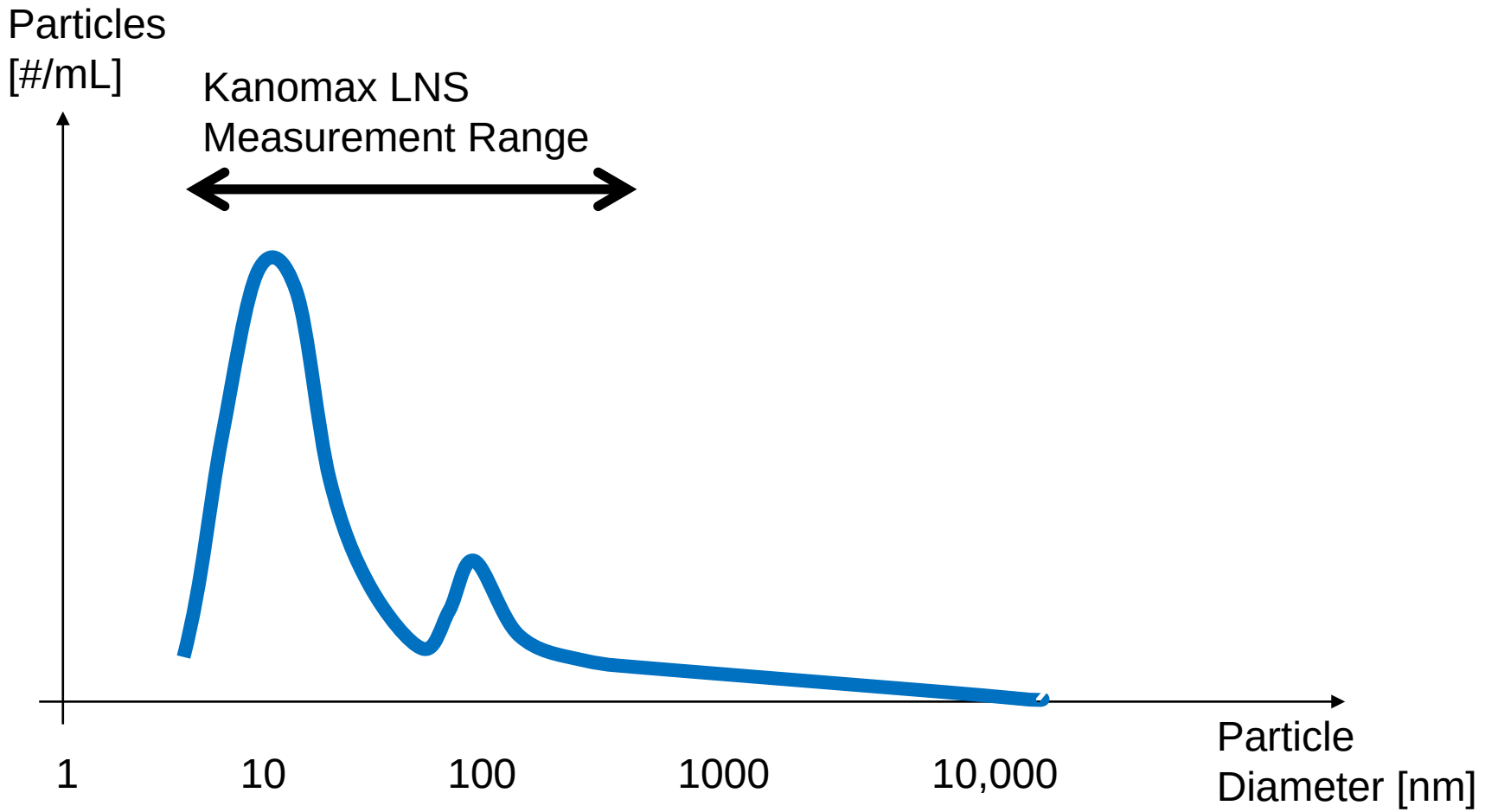
Laser Diffraction



Reference: Litchy, M. et.al.: Pittcon 2012

LNS measures main slurry distribution, not tails

6 - 360 nm for LNS



Kanomax Model 9310

Liquid NanoParticle Sizer System

- 6 nm to 360 nm particles are individually measured regardless of shape or composition.
- 64 size bins per decade are sequentially characterized.
- Complete concentration vs. particle size distribution in about 5 minutes.
- No *a priori* assumptions about the particles.

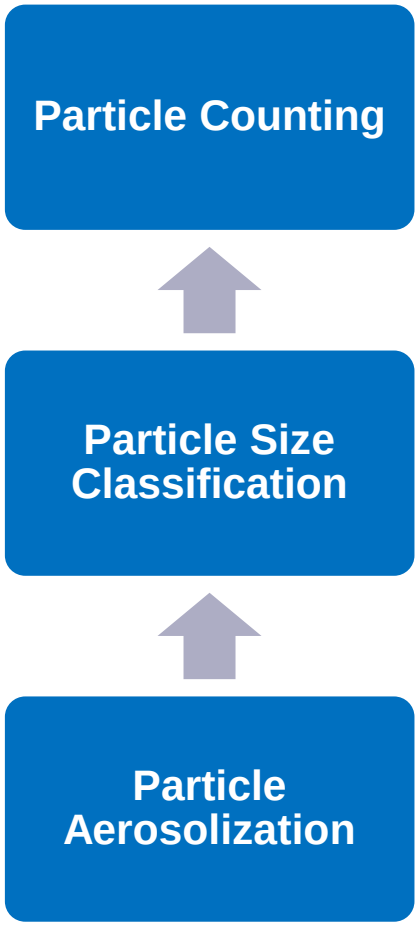
Particle Counting

Particle Size Classification

Particle Aerosolization

US Patents 8,272,253 and 8,573,034 cover this technology and are licensed to Kanomax FMT.

Many Advantages of the LNS System

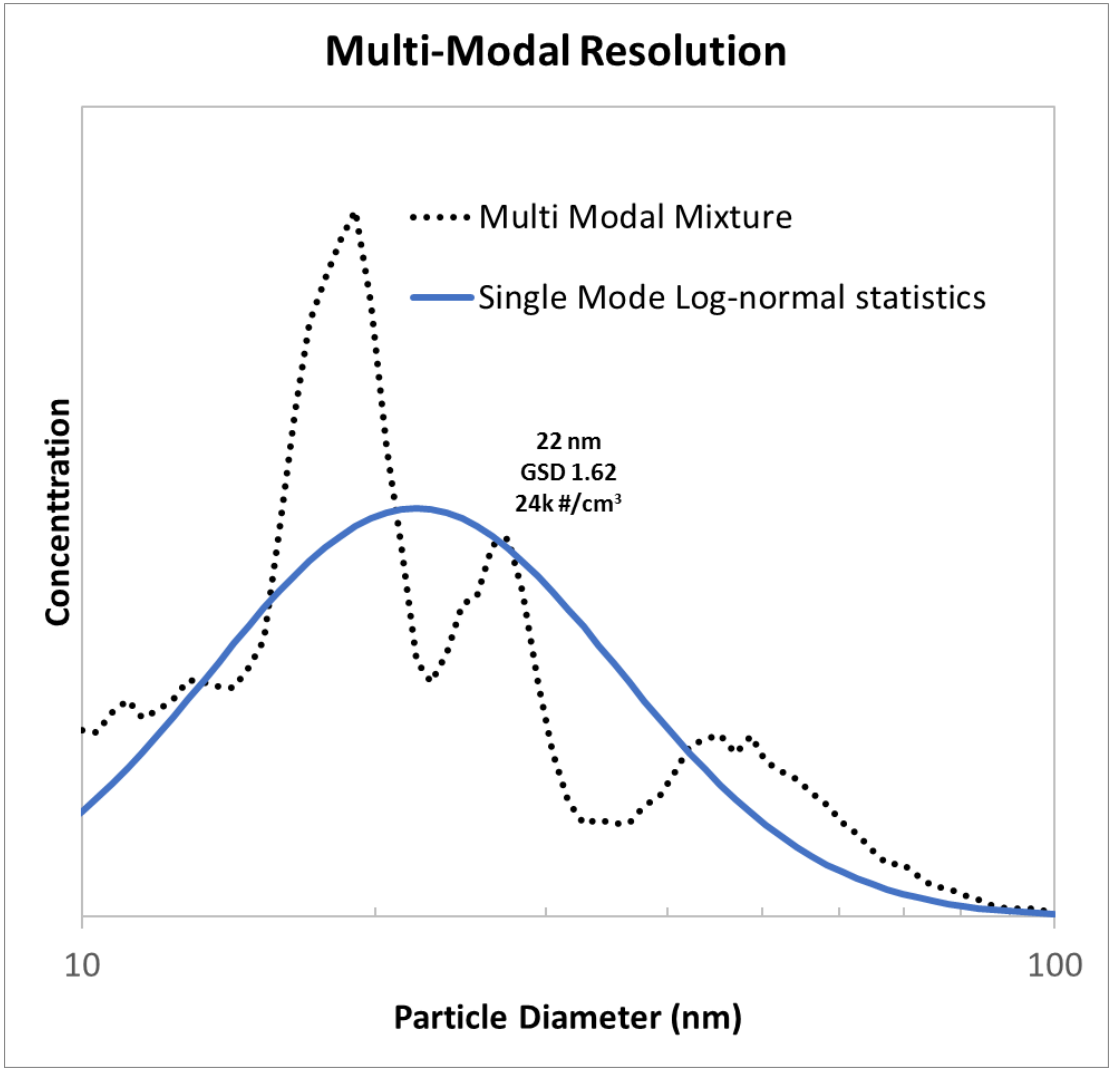


- Highly sensitive since individual particles are counted.
- Actual particle concentrations, not relative concentrations, are measured directly.
- Shape of the particle size distribution is not assumed.
- Technique is independent of optical properties of the particles.
- Suitable for on-line or off-line measurements.

Real Slurries often have several modes

Particle populations are generally log-normal

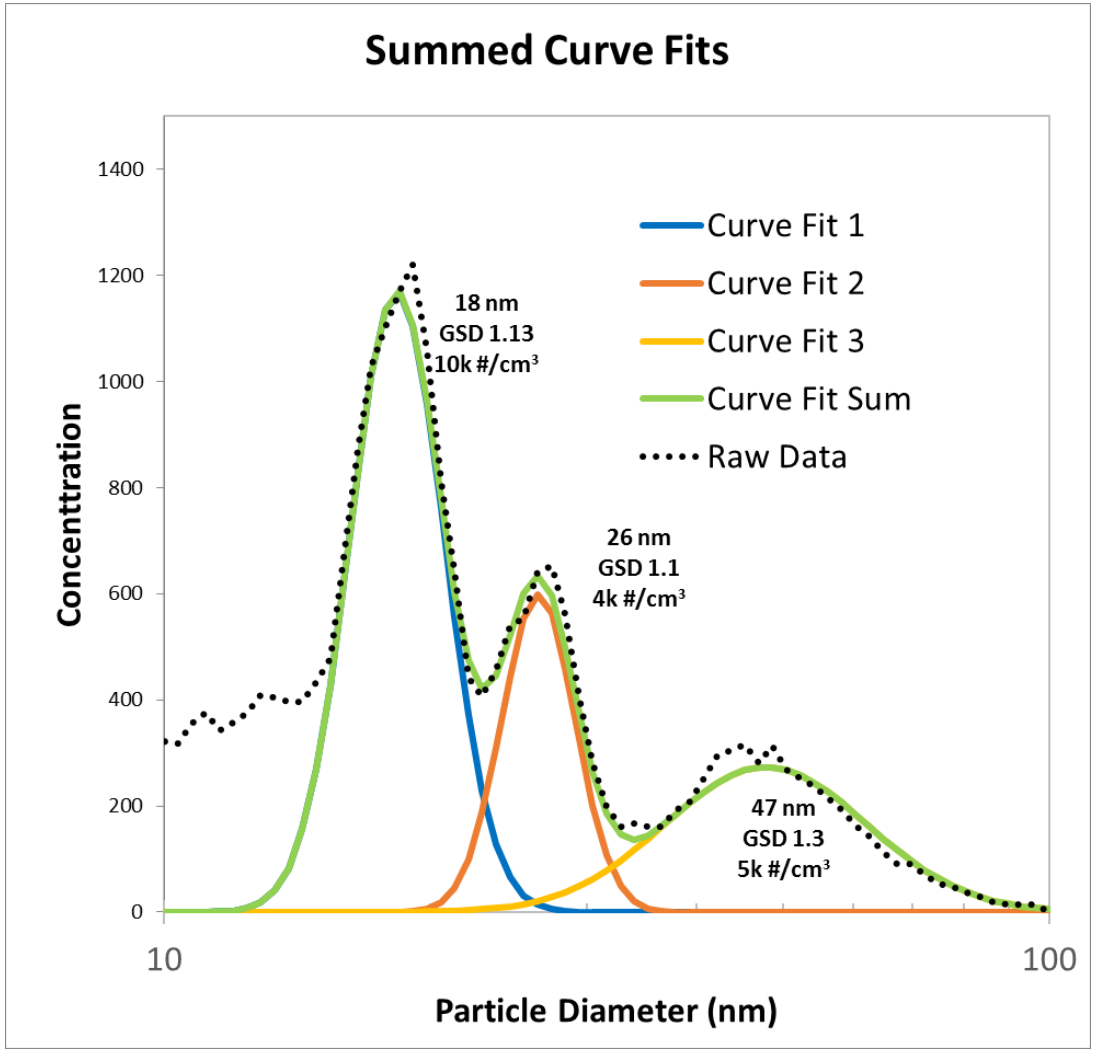
- Particle Populations often follow Log-Normal Distribution
- Log normal distribution defined by three variables
  - *Peak Diameter*
  - *Geometric Standard Deviation*
  - *Total integrated concentration*
- Multimodal and skewed distributions not accurately represented by single mode statistics



LNS handles multi-mode slurries

Multimodal Curve Fitting gives Parametrized Slurry Metrics

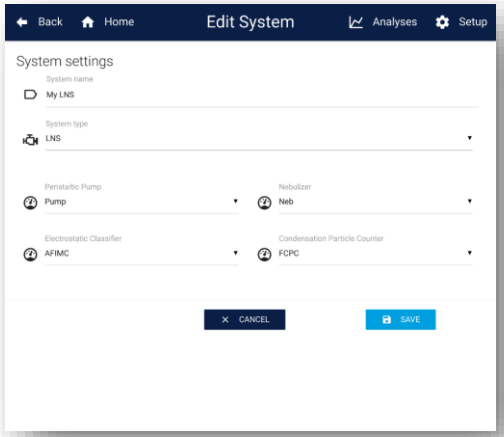
- Automatically generated in Kanolysis
  - Sum of single distributions fit to raw data
  - Individual modes may be defined by separate log-normal distributions
    - *Peak Diameter*
    - *Geometric Standard Deviation*
    - *Total integrated concentration*
- Parametrized slurry metrics are powerful for slurry grading and monitoring.

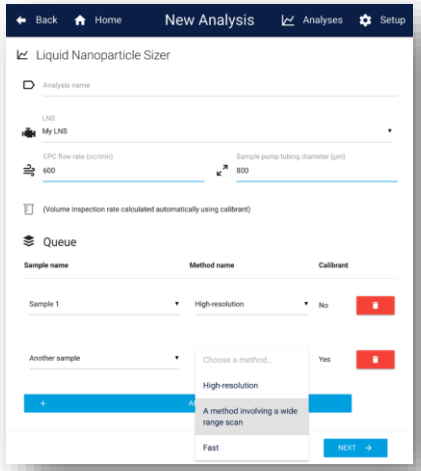


Kanolysis software streamlines measurements

Fully customizable, but can be automated for routine analyses

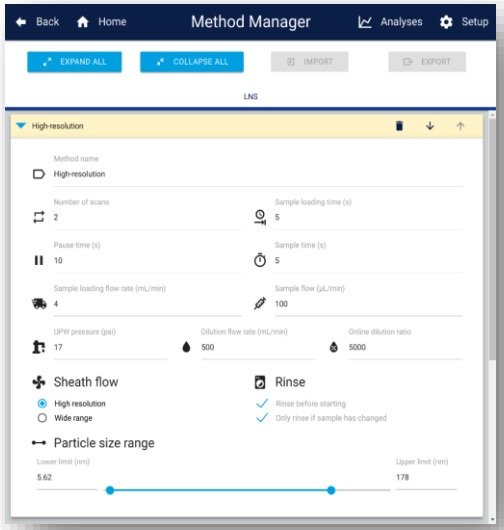
System manager defines the LNS system

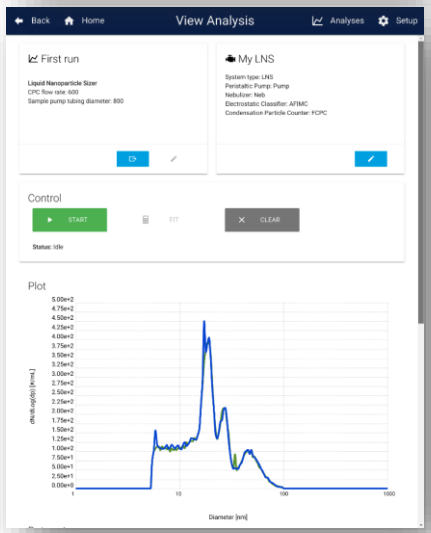




Analysis contains a series of methods used for characterization

Methods define system variables for a measurement



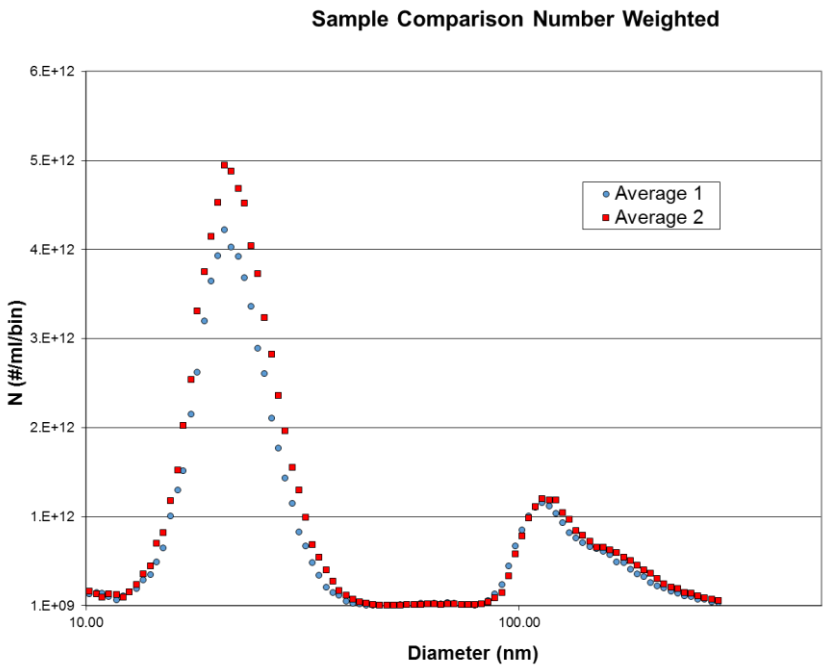


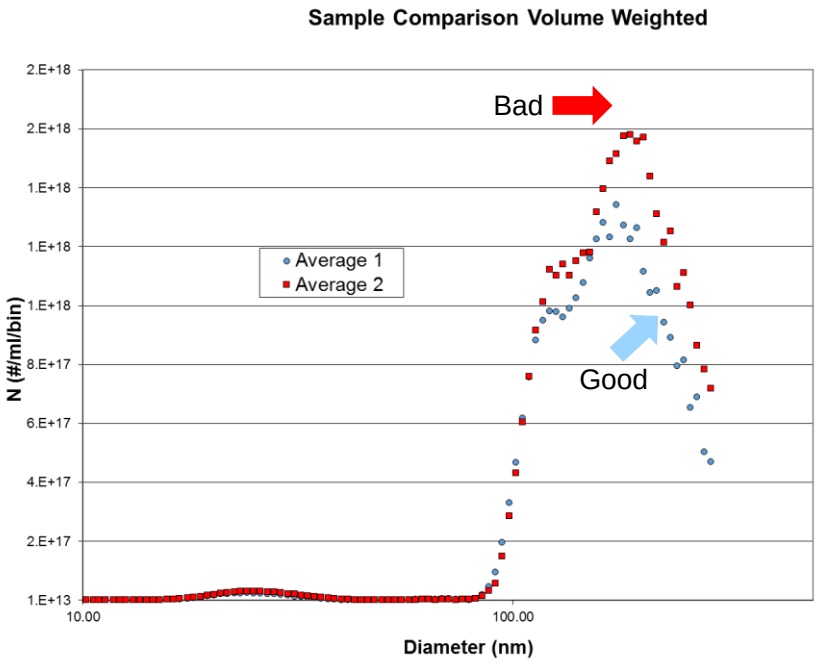
Particle Size Distribution data displayed in real time

Engineering Use: Slurry Turnover Limit

Size profile vs number of slurry turnovers shows onset of agglomeration

- After too many slurry turnovers, the number of large diameter particles increased to an unacceptable “Bad” limit. Peak Diameter also shifted larger. Likely due to agglomeration effects.
- The LNS system was able to resolve this subtle difference
  - Detect small shifts in individual modes
  - Detect changes in ratios of mode concentrations
  - Detect changes in mode shape (e.g. increased dimers)





Operations Use: Standardized Reporting

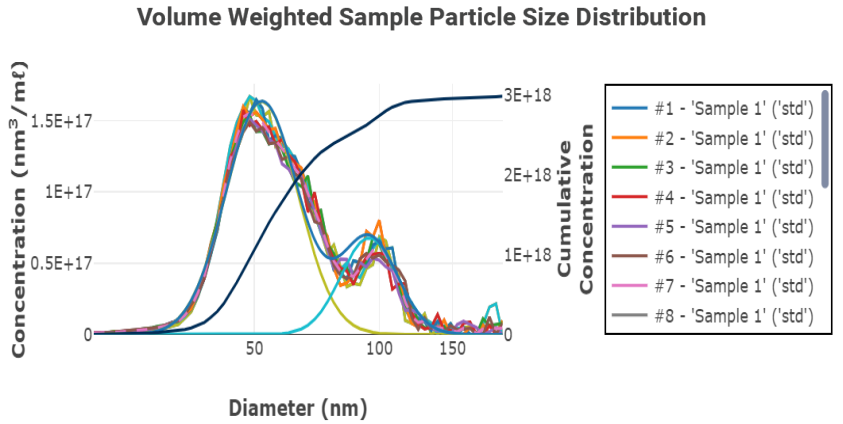
Concise report summarizing the important aspects of the slurry sample

- Many important uses in an industrial setting
  - Engineers can set up standard analyses that technicians can run.
  - Standardized slurry metrics.
  - Outgoing or Incoming Quality Control.
  - Line monitor and SPC.

Liquid Nanoparticle Sizer Report **Kanomax Model 9310**

Technician:	Steve Kosier
System ID:	LNS System 14
Method:	Slurry outgoing QA
Sample:	10.15.2018.Slurry45
Offline dilution ratio:	1000:1

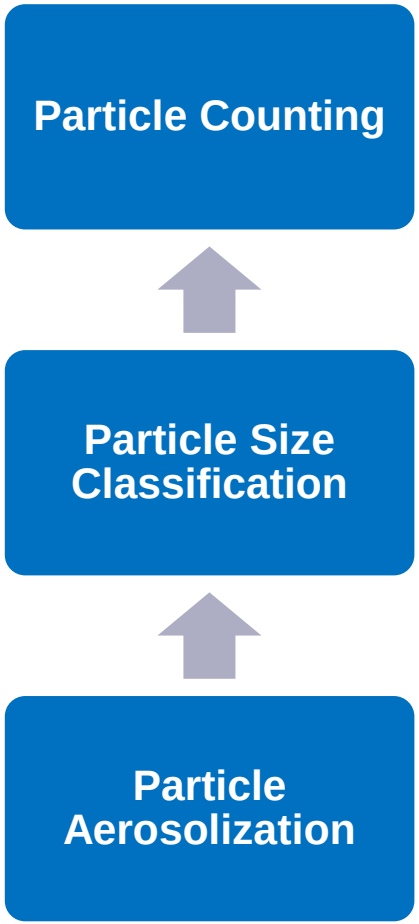
	GM Diameter (nm)	GSD	Concentration ($nm^3/m\ell$)	D_{10}(nm)	D_{50}(nm)	D_{90}(nm)
Curve fit A	48.1	1.22	6.25e+13	40.7	52.3	67.3
Curve fit B	90.0	1.17	6.00e+12	77.7	93.1	116
Total			6.85e+13	42.2	56.2	96.5



Report Date: 2018/10/11

Summary

- The LNS 9310 System from Kanomax FMT is a versatile and easy to use tool for advanced CMP Slurry characterization and control.
  - 6 nm to 360 nm particles are individually measured regardless of shape or composition.
  - Complete concentration vs. particle size distribution in about 5 minutes.
  - No a priori assumptions about the particles.
  - Measures the main slurry distribution, not the tail.
- The LNS System is useful in both engineering and operations environments.
- Kanolysis software provides flexibility and workflow automation to boost productivity.



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Thank you for your attention!

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www.KanomaxFMT.com

Appendix

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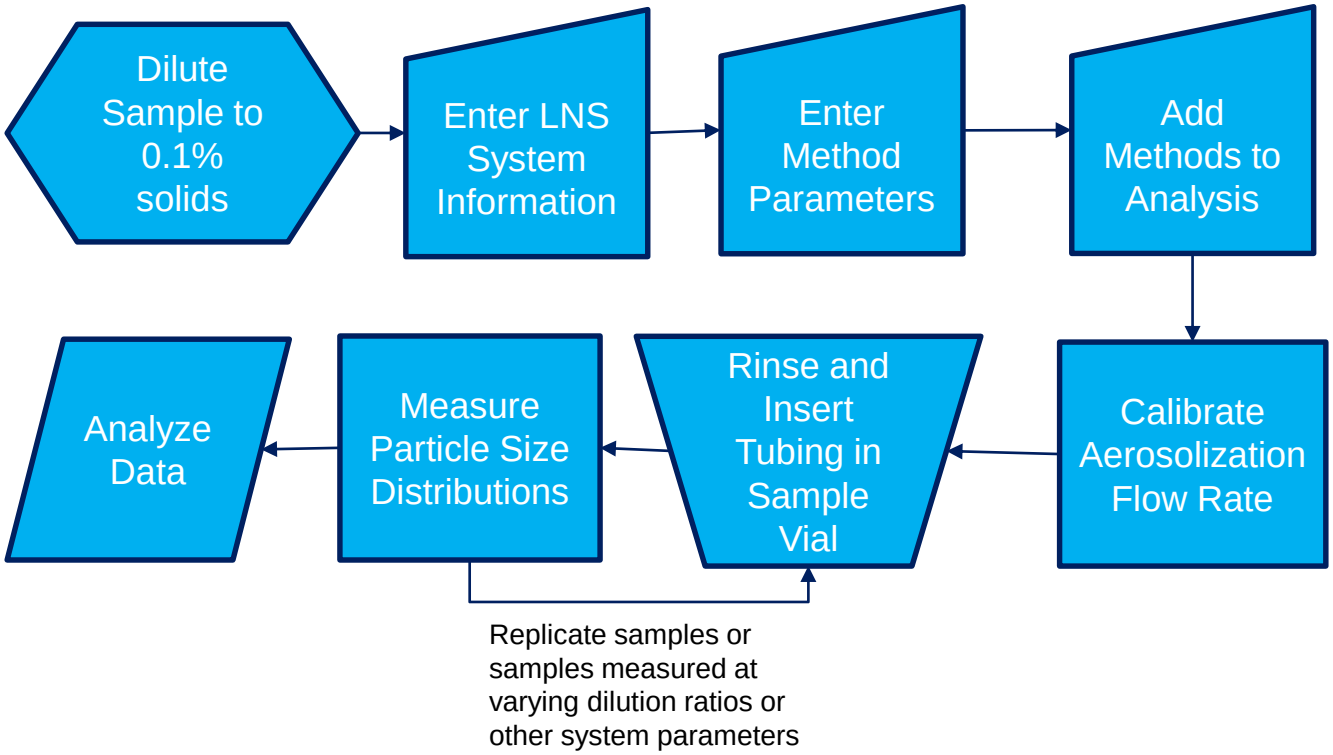
Grant DC, DC Chilcote and U Beuscher (2012). "Removal of 12 nm particles from UPW by a combination of Ultrafiltration Modules and Microfiltration Cartridges," *Ultrapure Water Journal*, May/June 2012.

Rastegar, A (2013). "Particle Control Challenges in UPW ", presented at 2013 UPW Micro Conference

Patents US 8,272,253; US 8,573,034; US 7,852,465; Other patents pending

LNS Workflow

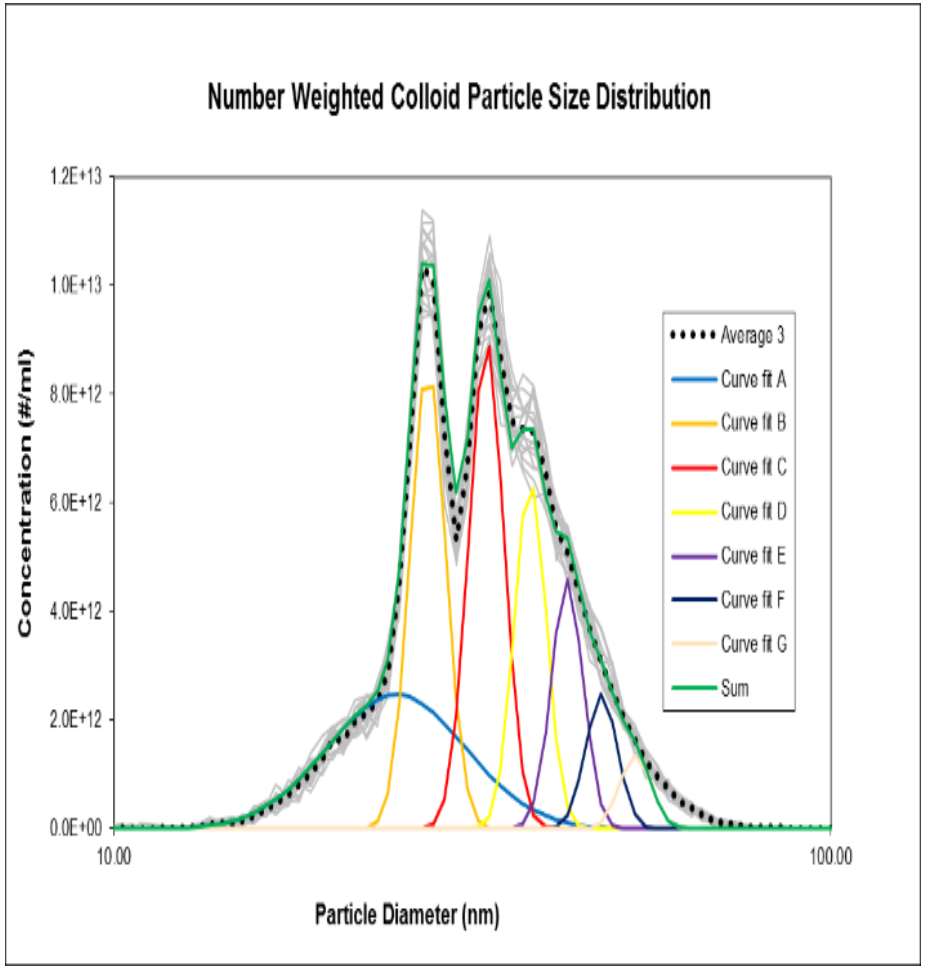
Easy sample preparation and automated analysis routines

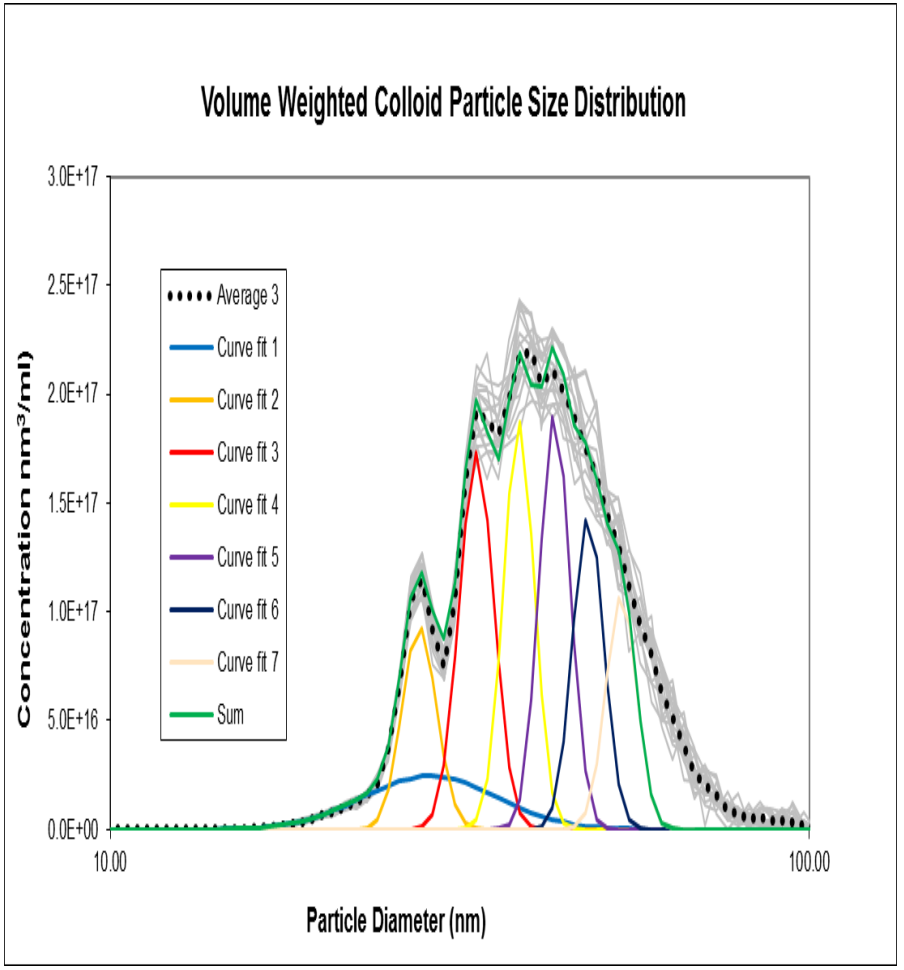


Complex modal distributions are fitted easily

Can use Number, Volume, Surface Area, or Mass Weighting

- Able to resolve multiple overlapping modes

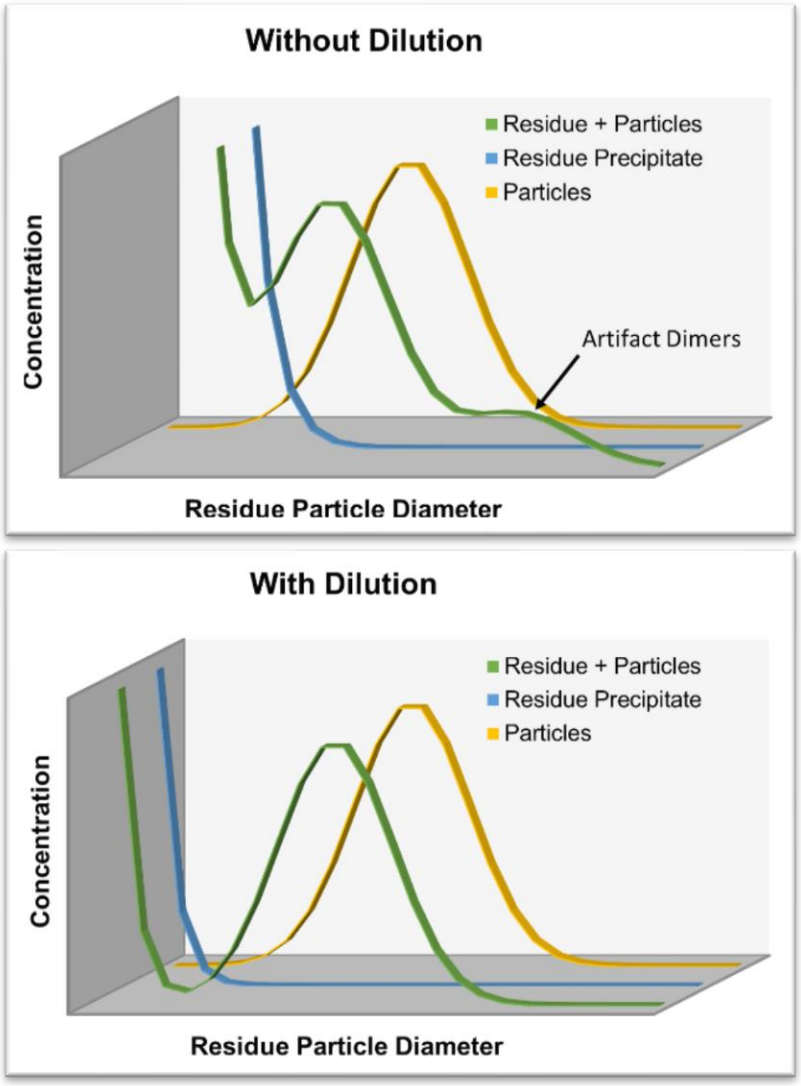




Aerosolization

Sample Concentration Effects

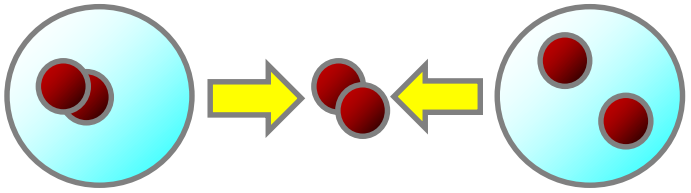
- Each nebulized droplet contains the same concentration of Dissolved Non-Volatile Residue
- Size of Precipitated Non-Volatile Residue particle proportional to the parent droplet size
- High concentration of Dissolved Non-Volatile Residue may overlap native particle size distribution
- Artifact dimers form when two separate particles are coincident in a single droplet



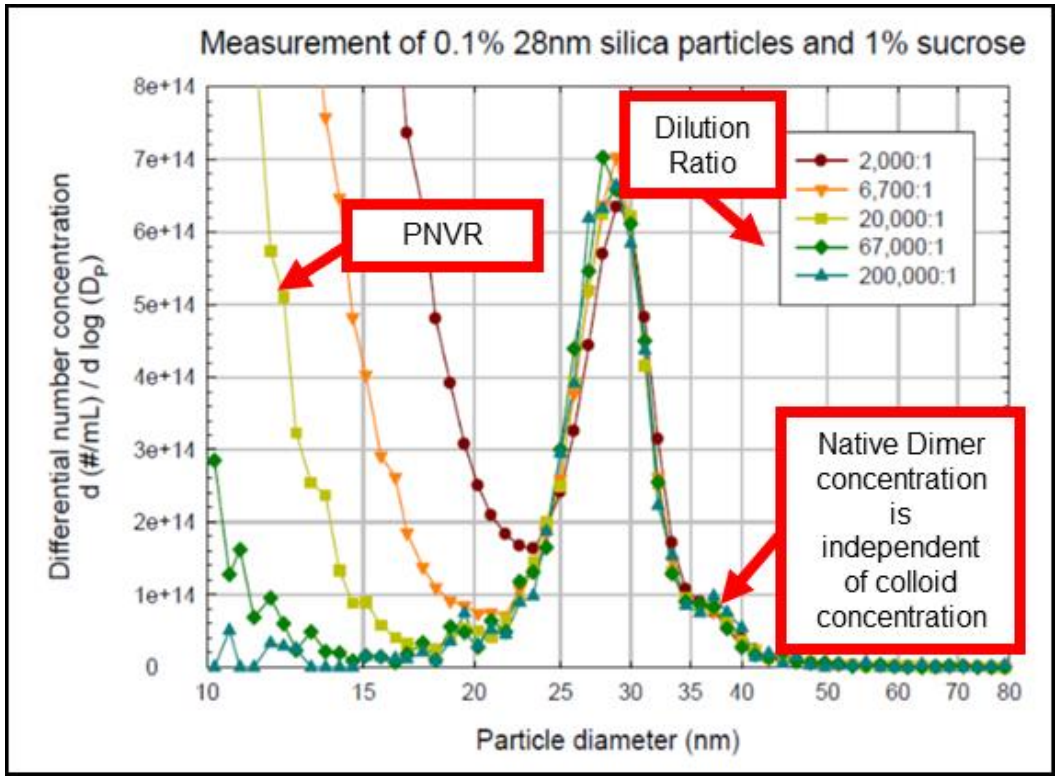
Liquid Nanoparticle Sizer (LNS)

Concentration Effects- Identifying artificial dimers

- Artificial dimers are caused by the presence of two discrete colloid particles within a single droplet
- The probability of a single droplet containing multiple particles is proportional to droplet diameter and colloid concentration: $P_{2+} \propto C \times D_{droplet}^{3}$
- Eliminating large droplets allows for higher concentrations of discrete aerosolized colloid particles



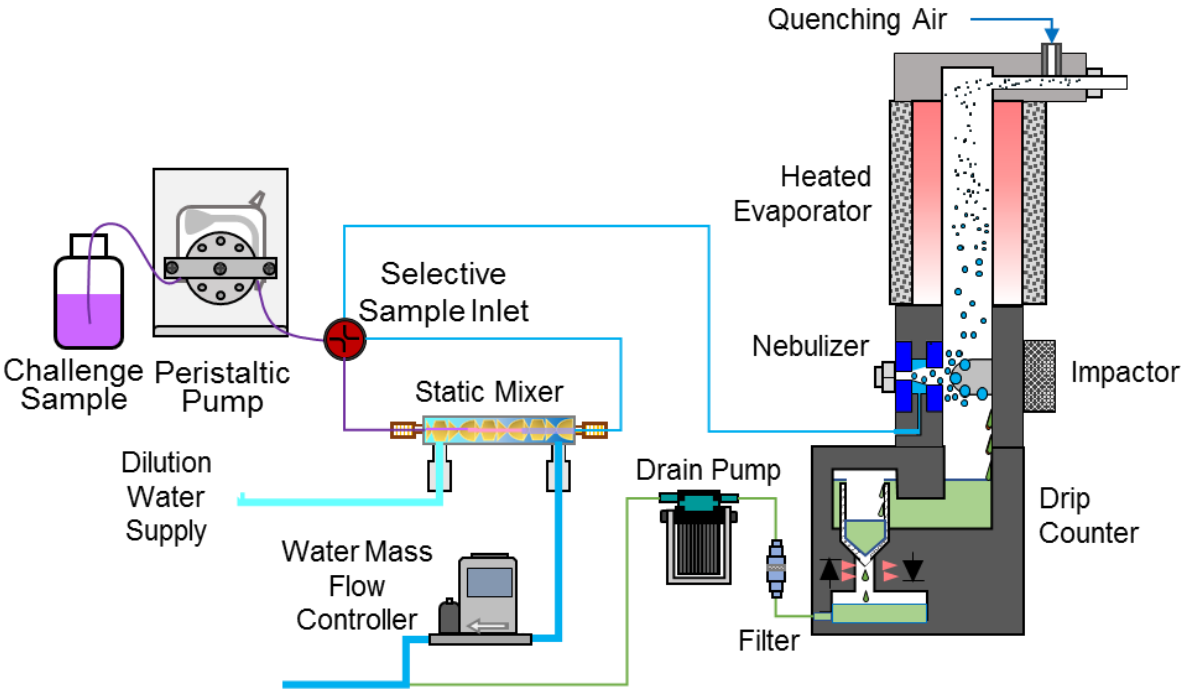
Native dimer Artifact dimer



Liquid Nanoparticle Sizer (LNS)

Principle of Operation – Nanoparticle Nebulizer

- Nanoparticle Nebulizer provides online dilution and sample aerosolization
- Designed to nebulize droplets with a small peak diameter and reduced concentration of large droplets
- Software controls dilution ratio by varying sample and dilution water flow



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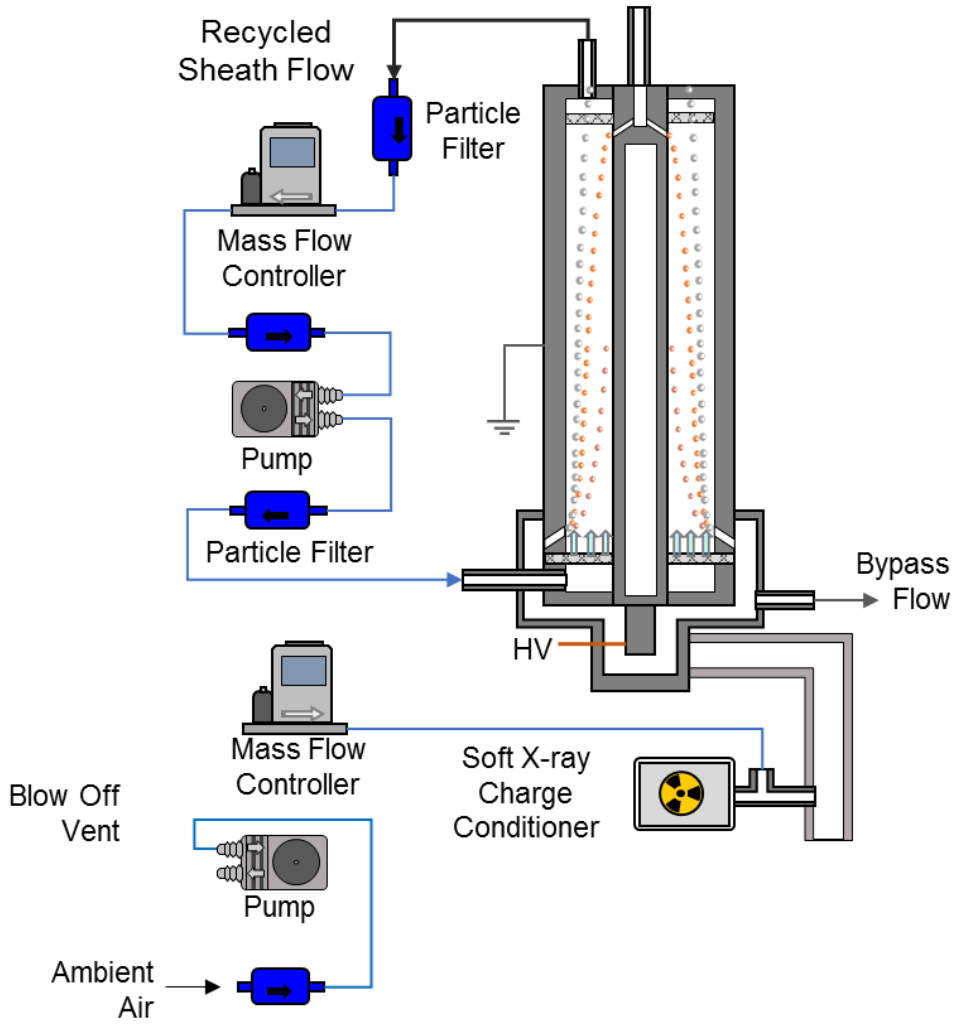
Liquid Nanoparticle Sizer (LNS)

Principle of Operation – Ion Mobility Spectrometer

- Annular Flow Ion Mobility Classifier (AFIMC) acts as a "bandpass" filter based on particle size
- Measurement of particle concentrations over a range of selected sizes provides particle size distribution information
- Data inverted to account for charging and detection efficiency of the aerosol particle counter



Liquid Nanoparticle Sizer (LNS)

Principle of Operation - Condensation Particle Counter

- Used as particle detector at the exit of the AFIMC
- Heated Saturator adds butyl alcohol vapor to the aerosol
- Cooled Condenser causes the butyl alcohol vapor to become supersaturated
- Supersaturated butanol vapor condenses onto particles in the aerosol making large droplets
- Droplets counted optically using light scattering ("Dry" particles are not detected)