KANOMAX
The Ultimate Measurements

Liquid Nanoparticle Sizer (LNS) System Model 9310

The new Model 9310 Liquid Nanoparticle Sizer (LNS) system provides a complete solution for measuring high-resolution size distributions of nanometer-sized colloidal suspensions with particle sizes from 5 – 600 nm.

Liquid Nanoparticle Sizer (LNS) system.

An optional auto-sampler can be used instead of the peristaltic pump.

Benefits of the LNS

- Fully integrated system maximizes response time and accuracy.
- Measures actual concentration, not relative concentration.
- Measures high-resolution size distributions (64 channels per decade) and has a broad size-measurement range of 5 – 600 nm.
- Measurement is independent of sample viscosity, temperature, particle material, particle shape, and particle surface properties.
- Performs discrete particle sizing and counting.
- Measurements can be made with minimal sample preparation in a fast time (<5 minutes).
- Requires no modifiers to adjust sample conductivity.
- Ideally suited for measuring colloid particles <30 nm in size where dissolved residue may interfere with the sample.
- Offers stable long-term operation.
- No influence from particle-particle interactions.
- Requires no complex inversion routines or data analysis.
- Multimodal size distributions.

Applications

- Chemical mechanical polishing slurries
- Filter testing
- Protein analysis
- Environmental nanoparticle contamination
- Pharmaceutical colloids
- Coagulation studies
- Particle synthesis monitoring

Kanomax's new LNS system has been cited as the measurement technique in two SEMI Guides:

C79-0113 Guide to Evaluate the Efficacy of Sub-15 nm Filters Used in Ultrapure Water (UPW) Distribution Systems.

C93-0217 Guide for Determining the Quality of Ion Exchange Resin Used in Polish Applications of Ultrapure Water System

How the LNS Works

Dilution

A colloid sample can be introduced to the NanoParticle Nebulizer (NPN) Model 9110 with either a peristaltic pump or an auto-sampler. The NPN has the ability to inject a colloidal sample directly into the nebulizer, or you can dilute the sample with ultrapure water by choosing the diluted sample option. Using the dilution option offers the following advantages:

- Stabilizing agents are reduced leading to lower levels of dissolved non-volatile residue (DNVR)
- Limited time for coagulation to occur
- Particle shedding from the sample introduction device and contamination from pipettes and sample vessels are reduced
- Ability to identify native dimers and remove their influence

Nebulization

The Nanaoparticle Nebulizer generates a fine mist of droplets from a water sample using a patented dual-orifice pneumatic sprayer. The volume of large droplets is limited by an inertial impactor. The spray generated by the nebulizer is then heated to promote evaporation of the water and volatile residue. The resulting aerosol is composed of particles that were present in the water sample as well as particles formed by DNVR. The aerosol is mixed with clean dry air and the particle size distribution is measured by using an ion mobility classification and condensation particle counting system. Figure 1 is a diagram of the Dilution and Nebulization process, before measurement by Aerosol Measurement System.

Larger droplets contain more DNVR than smaller droplets and therefore cause either a thicker coating onto sample particles or larger residue-based aerosol particles. Limiting the size of the droplets and using online dilution to reduce the amount of DNVR present in the nebulized sample mitigates the influence of DNVR on the true particle size and overall distribution.

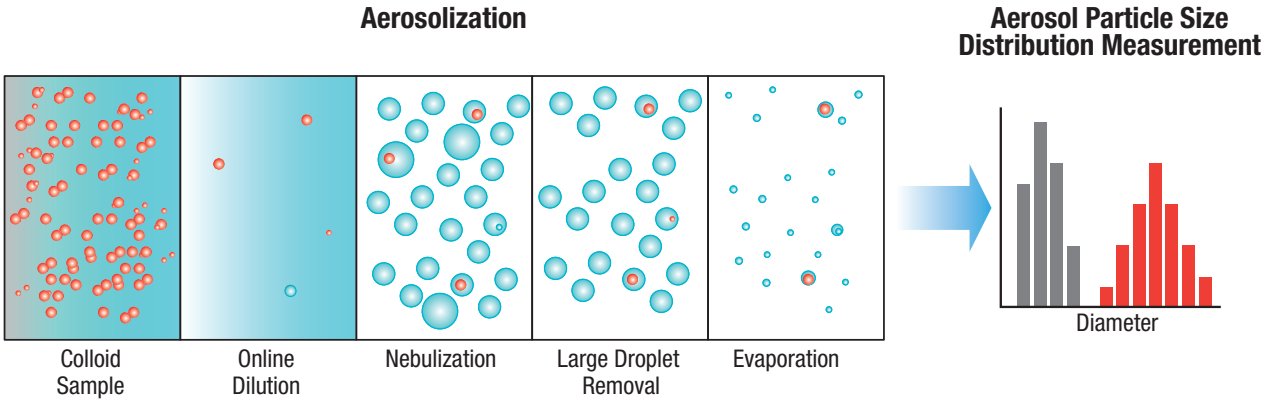

Figure 1: Dilution and Nebulization process.

Sample dilution reduces the size of precipitated non-volatile residue particles by reducing the dissolved residue concentration. The online sample dilution feature of the LNS provides the highest purity dilution available while also limiting dilution artifacts such as coagulation. An additional benefit of online dilution is the ability to quickly identify artifact dimers by varying the dilution ratio.

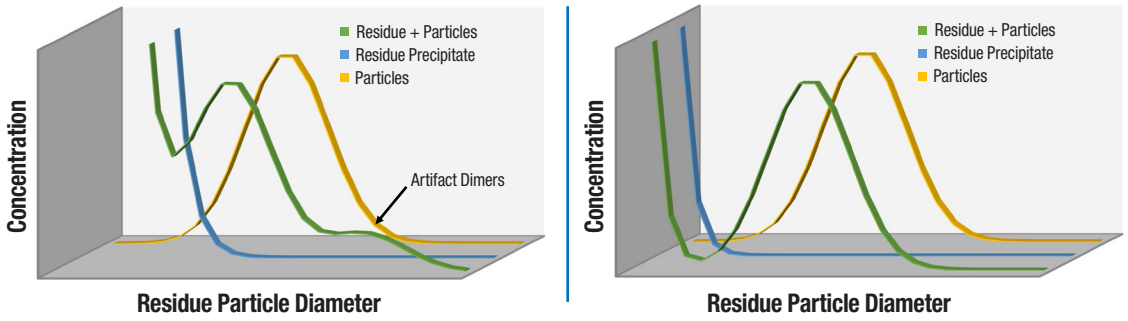

Figure 2: LNS on-line dilution improves the colloid particle size distribution measurement by minimizing DNVR and dimer artifacts.

Aerosol Measurement

The aerosol size distribution measurement system contains:

- A soft x-ray aerosol charge conditioner that places a known low-level distribution of charge on the particles.
- An Annular Flow Ion Mobility Classifier (Model 3660 AFIMC) that acts as a particle diameter "bandpass filter." The AFIMC spatially separates aerosol particles based on their electrical mobility (a function of particle diameter). Only particles within a narrow size band can leave the AFIMC. The selected size is adjusted by varying the electric field (voltage) within the AFIMC and a size distribution can then be generated by scanning through a range of voltages.
- A Fast Condensation Particle Counter (Model 3650 FastCPC) to measure the number concentration of the particles leaving the AFIMC.
- LNS software to rapidly invert the concentration measurement to a particle size distribution. The intuitive software guides the user through measurement and data analysis. Figure 3 shows a sampling of the software screens.

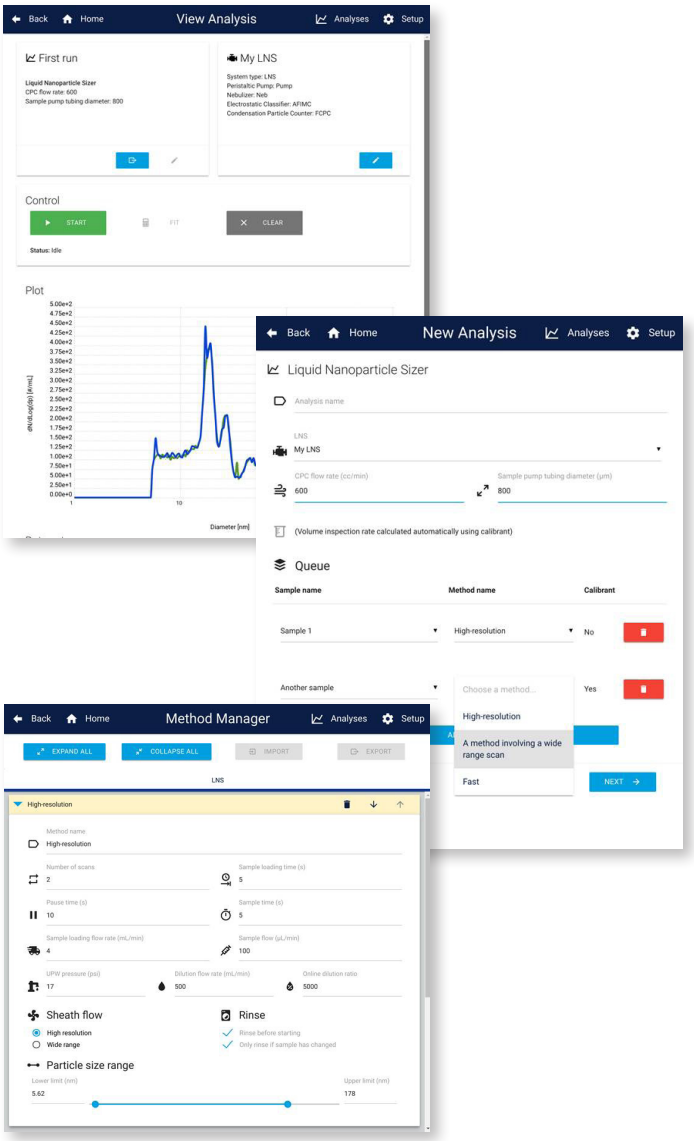

Figure 3: : Modern multi-platform software controls the LNS system and provides data analysis tools with functionality to run methods and protocols.

Outstanding Filter Test Results

The recently published SEMI C79-0113 "Guide to Evaluate the Efficiency of sub-15 nm Filters used in Ultrapure Water Distribution Systems" used the LNS as its particle-size instrumentation because no other particle instrumentation was capable of making the required measurements.

A filter with a nominal pore size of 20 nm was challenged with colloidal silica at 12.6 nm and 18.8 nm. Figures 4 and 5 show the challenge and filtrate concentrations as measured by the LNS. Figure 6 shows the same data plotted as particle retention using a log reduction value (LRV). While the filter shows 99% retention at about 18 nm, there is significant particle penetration at 10 nm.

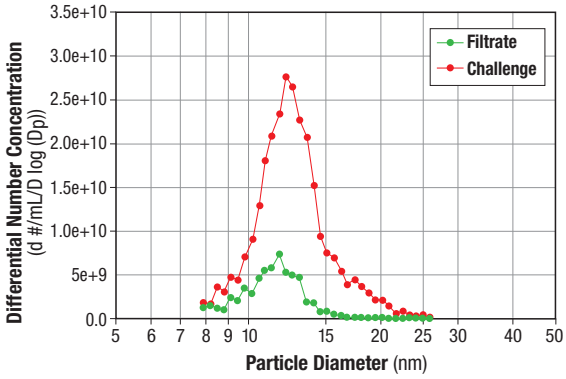

Figure 4: Filtrate and Challenge for 12.6 nm Colloidal Silica.

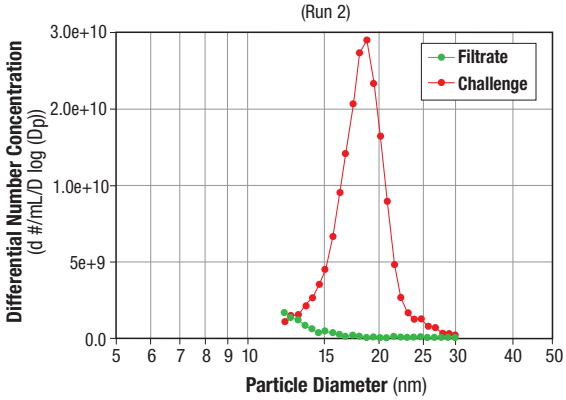

Figure 5: Filtrate and Challenge for 18.8 nm Colloidal Silica.

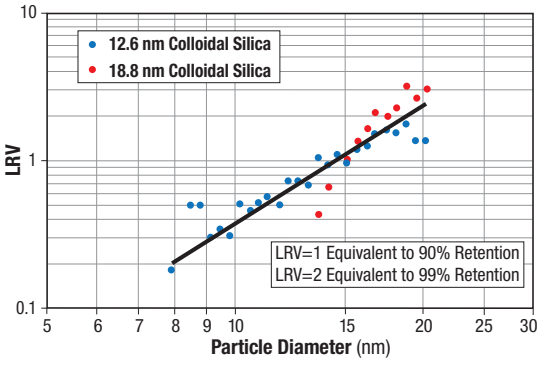

Figure 6: Retention of Two Different Sized Particles.

Kanomax Holdings, Inc. purchased Fluid Measurement Technologies (FMT) in July 2015 and renamed the company Kanomax FMT, Inc. (KFMT). Dr. David Blackford, the founder of FMT is now the President of KFMT. He has nine issued U.S. patents, three U.S. patents pending, and many technical publications for his innovative technologies.The LNS product development was done in association with CT Associates, Inc. (CTA).

Bibliography

"A Nanoparticle Nebulizer for Generation of Aerosolized Colloid Particles with Reduced Influence by Non-volatile Residue." Authors: Derek Oberreit, David Blackford, Gary Van Schooneveld and Seongho Jeon. Poster presented at 2014 AAAR Conference, Orlando FL.

"A Novel Method for Measuring the Sizes and Concentrations of 5-500 nm Particles in Colloidal Suspensions." Authors: Gary Van Schooneveld, Mark Litchy and Donald Grant Nanotech Conference 2011: Technical Proceedings.

"A New Method for Determining the Size Distribution of Particles in Slurries." Authors: Gary Van Schooneveld, Mark Litchy and Donald Grant. Proc. 2010 International Conference on Planarization/CMP Tech. Pg. 348-351.

"Nanomaterial Size Distribution Analysis via Liquid Nebulization Coupled with Ion Mobility Spectrometry (LN-IMS)." Authors: Seongho Jeon, Derek Oberreit, Gary Van Schooneveld and Christopher J. Hogan Jr. Analyst, 2016, 141, 1363.

Patent numbers 8,272,253 and 8,573,034 have been issued to CTA and licensed by Kanomax. Kanomax has applied for additional domestic and international patents for technology contained within the ScanningTPC. Patent number 7,852,465 has been issued to Kanomax.

CMP Size Distribution Measurement

Figure 7 shows the size distribution of a multimodal CMP slurry. In this example Kanomax's custom software (Kanolysis) has identified three modes within the size distribution. Kanolysis can then assign a cumulative size D10 (diameter at 10%), D50 and D90 to each mode.

As the size of a particular mode changes, it can be monitored with these size statistics.

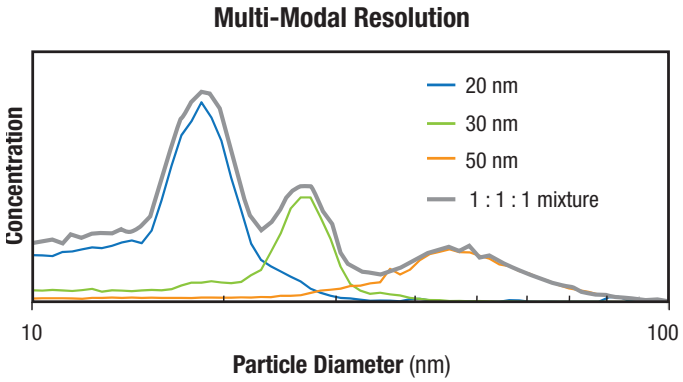

Figure 7: LNS measurement of a CMP multimodal size distribution.

Specifications

Particle Size Range: 5 – 600 nm

Particle Size Resolution: 64 channels per decades of size

Measurement Time: < 5 minutes

Inspection Volume Rate: 0.2 – 1.0 µL/min

Total Liquid Sample Flow Rate (online): 50 – 280 mL/min

Nebulizer Flow Rate: 0.5 – 3.0 mL/min (direct injection)

Dilution Factor Range: 50 – 20,000

Colloid Concentration Range(post dilution): 3E7 - 3E11 number/mL

Response Time to Concentration Change: < 90 seconds

Inlet Water Pressure (online): 200 – 500 kPa (29 – 72 psig)

Compressed Air Flow Rate/Pressure: 2.5 std L/min CDA or Nitrogen 2.8 bar (50-60 psi)

Wetted Surfaces (before nebulization): PFA, PTFE, sapphire, 316L stainless steel, PEEK

CPC Working Fluid: n-butyl alcohol (butanol)

I/O Communications: Ethernet

Power Requirements: 100/115/220/240 VAC; 50–60 Hz

Operating Temperature: 10-30°C

Operating Humidity: 0-90 non-condensing

Storage Temperature: 5-35°C

Dimensions: 9.5 × 8 × 36 inches (D/W/H)

Software – Computer Operating System: Windows 10

Refer to individual product sheets for component details.

Computer not included.

CDA – clean, dry air

Specifications subject to change without notice.

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