A comparison of optical particle counting and aerosol-based particle counting technologies for UPW monitoring and diagnostics.

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Introduction

- Liquid optical particle counting (OPC) utilizing light-scattering has been and continues to be a key technology used by the semiconductor industry to count particles in ultrapure water (UPW).
- Demand for detecting particles smaller than the current capabilities of these instruments (<< 20 nm) has resulted in development and introduction of instruments that utilize alternate detection techniques such as acoustic emission[1], laser induced breakdown detection[2] and nebulization/aerosol particle counting[3].
- Each of these techniques deploy technologies that detect particles, and potentially other forms of contamination, that differ from the OPC.
- Correlating the data generated by the new technologies to the historical data base available from OPC's is important for these new technologies to gain acceptance and to understand what new and potentially insightful information may be available using these techniques.

1. Madanshetty, Sameer, Particle profiling of UPW and suspensions, Ultrapure Water Micro 2016, Austin, TX.
2. Boj, Sylvain, et al, "Has the LIBD technique have potential for online Nano-particle detection in UPW?, Ultrapure Water Micro 2015, Portland, Oregon.
3. Blackford, David, et al, Introducing a non-optical 10 nm particle counter for ultrapure water, Ultrapure Water Micro 2014, Phoenix, AZ.

Outline

- Instrumentation overview and capabilities
- Test plan
- Water system description and sampling locations
- Online instrumentation results
- Off-line analysis (SEM/EDS) results
- Key observations and recommendations

Instrumentation

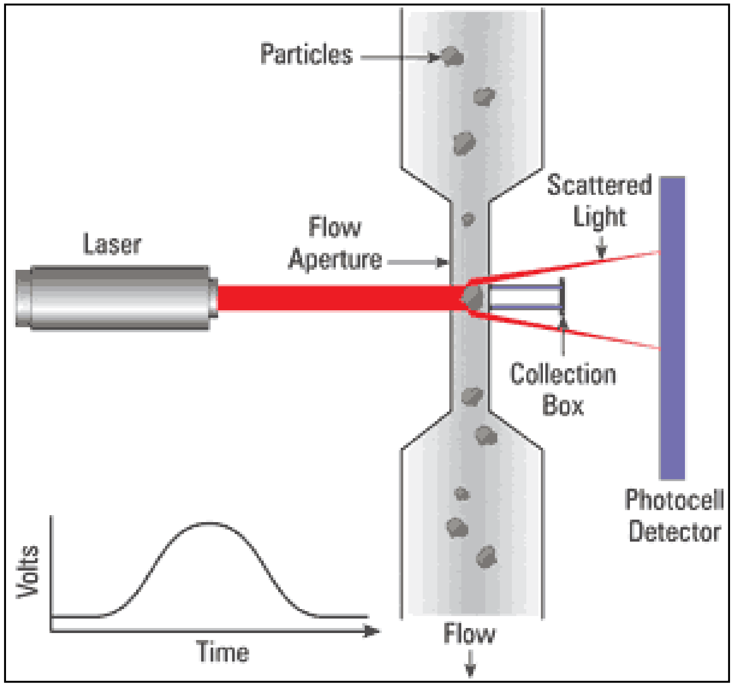
Kanomax FMT
Liquid Nanoparticle Sizer (LNS)

Lighthouse Worldwide
Solutions NC30+ OPC

Aerosol Devices Model SSS110
Sequential Spot Sampler

Kanomax FMT
NanoParticle Nebulizer Model 9110

Theory of Operation and Capabilities - Optical Particle Counting (OPC)

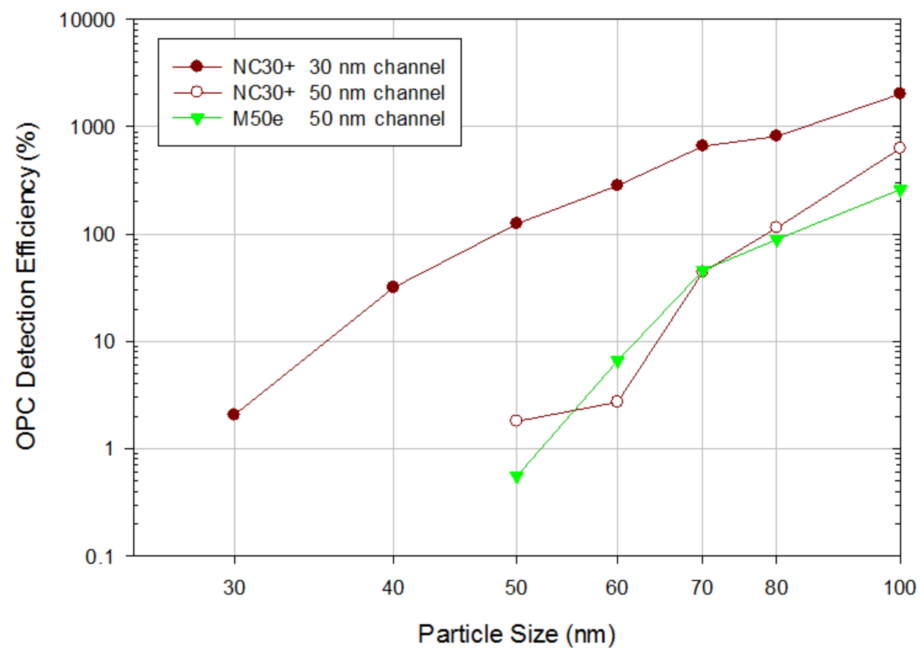


Courtesy of Noria Communications

Lighthouse Worldwide Solutions NC30+

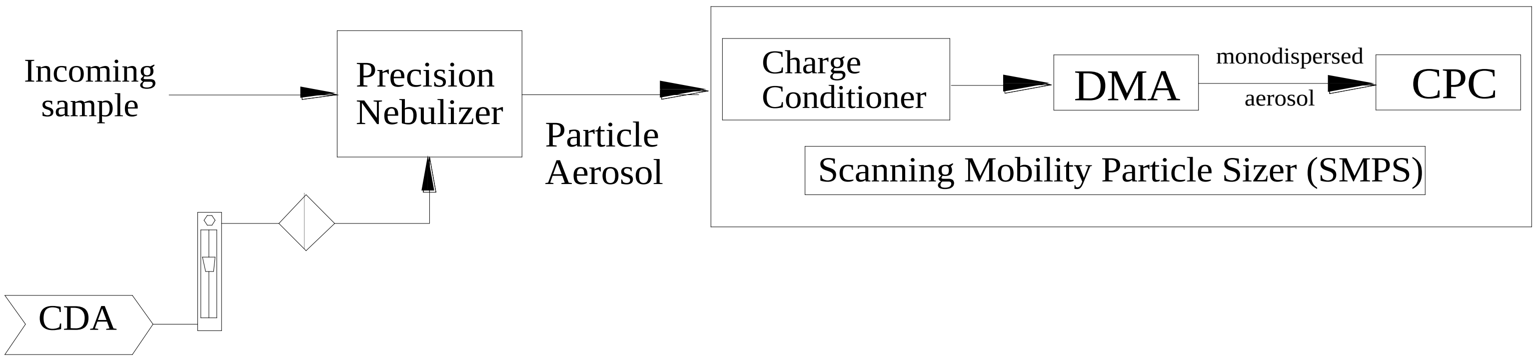
Sizing Channels: 30, 50, 80, 100 nm

OPC response to mono-dispersed PSL

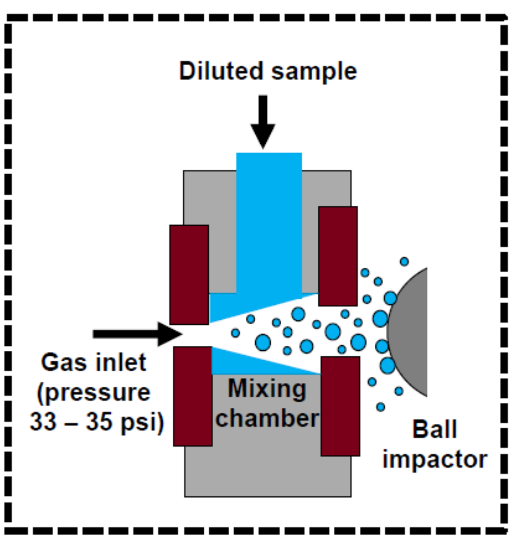


Source: Van Schooneveld, et al., UPW Micro 2013

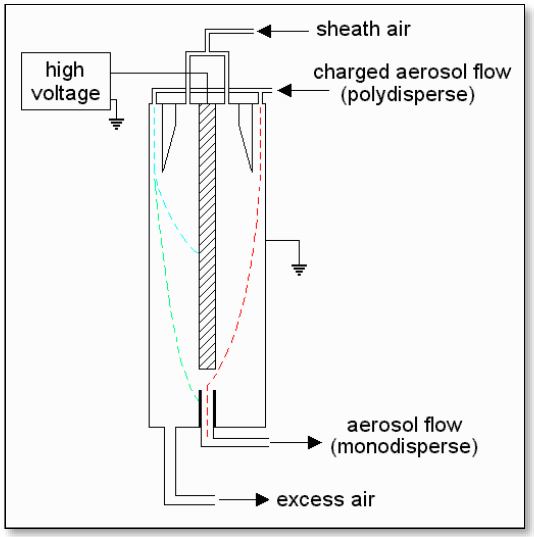
Theory of Operation and Capabilities - Liquid Nanoparticle Sizing (LNS)



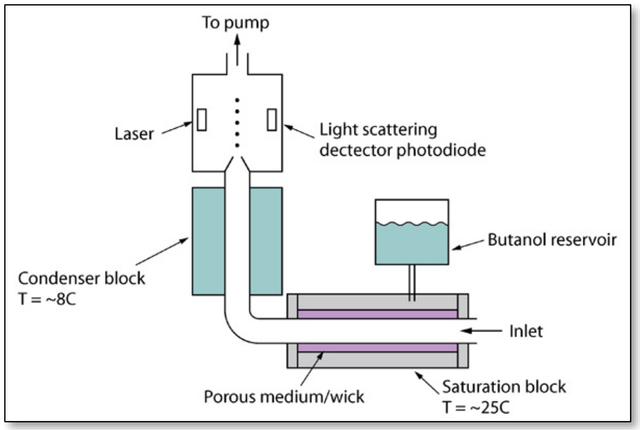
Precision Nebulizer



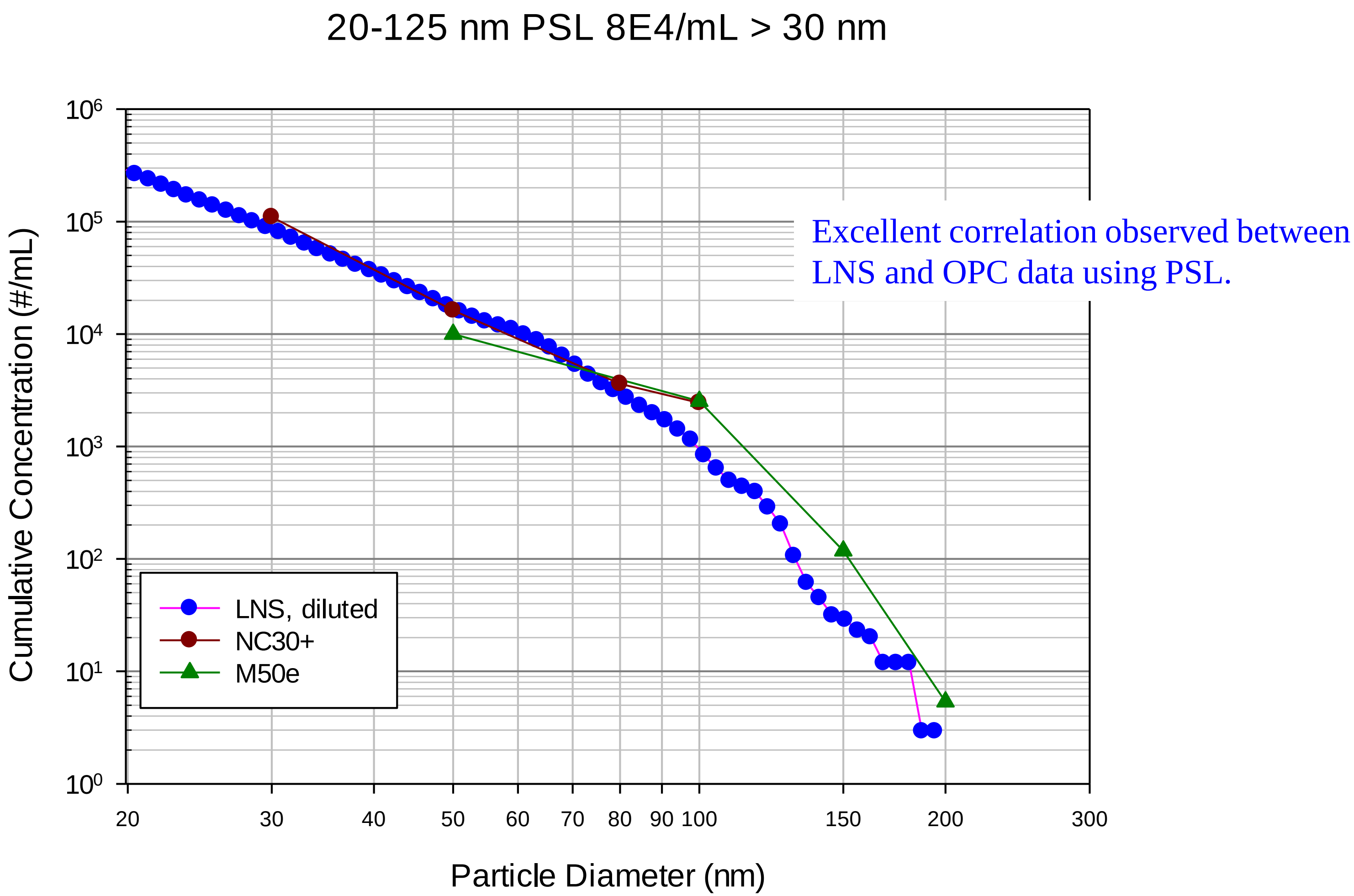
Differential Mobility Analyzer (DMA)



Condensation Particle Counter (CPC)

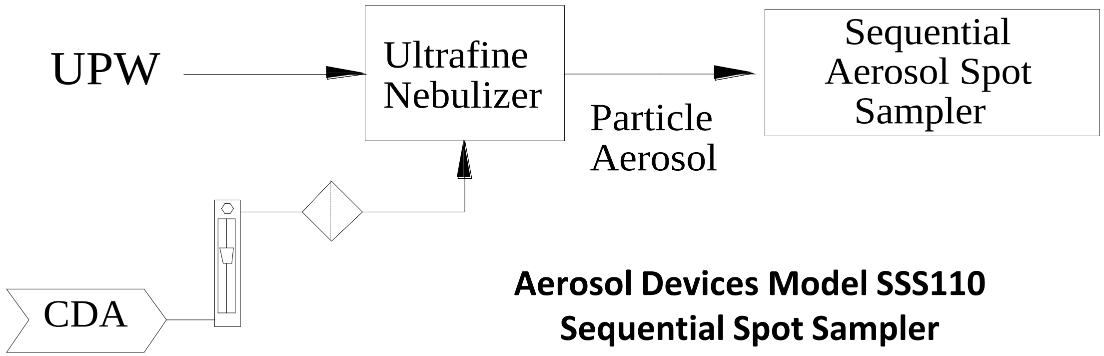


Theory of Operation and Capabilities - Liquid Nanoparticle Sizing (LNS)

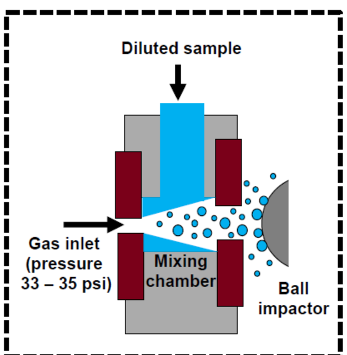


Source: Van Schooneveld, et al., UPW Micro 2013

Theory of Operation and Capabilities - Focused Aerosol Deposition (FAD)



Kanomax FMT NanoParticle Nebulizer Model 9110



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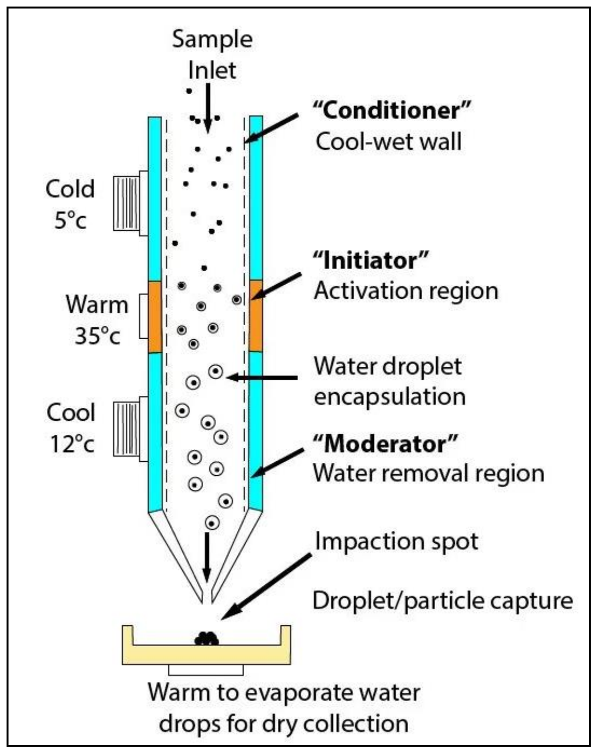
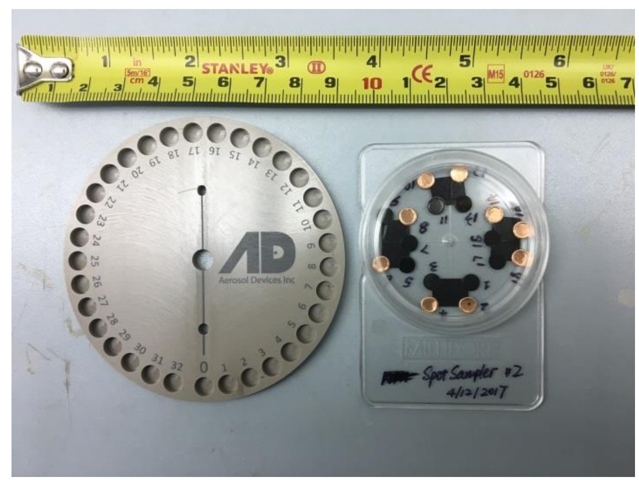
Aerosol Devices Model SSS110 Sequential Spot Sampler



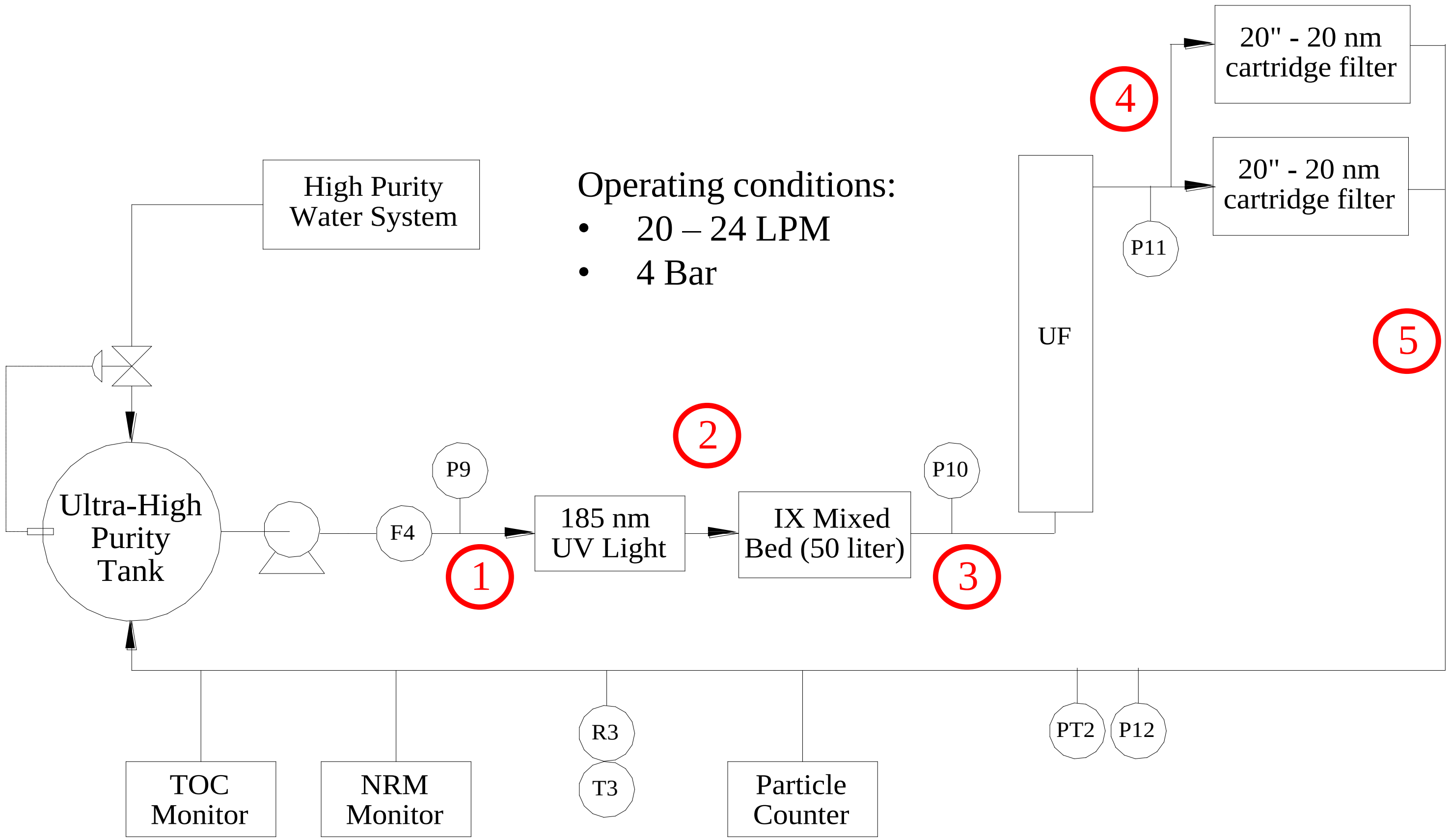


Test Plan

- Measure selected points on CTA's UPW system after major cleaning, new component install and installation of new final resin.
- Measure selected points for 12-24 hours before moving to new location.*
- Eliminate first two hours of data from data analysis.
- Analyze focused aerosol deposition samples via SEM/EDS.

* In subsequent testing, instrumentation collected data for several days at each sample point.

UPW System Sample Points



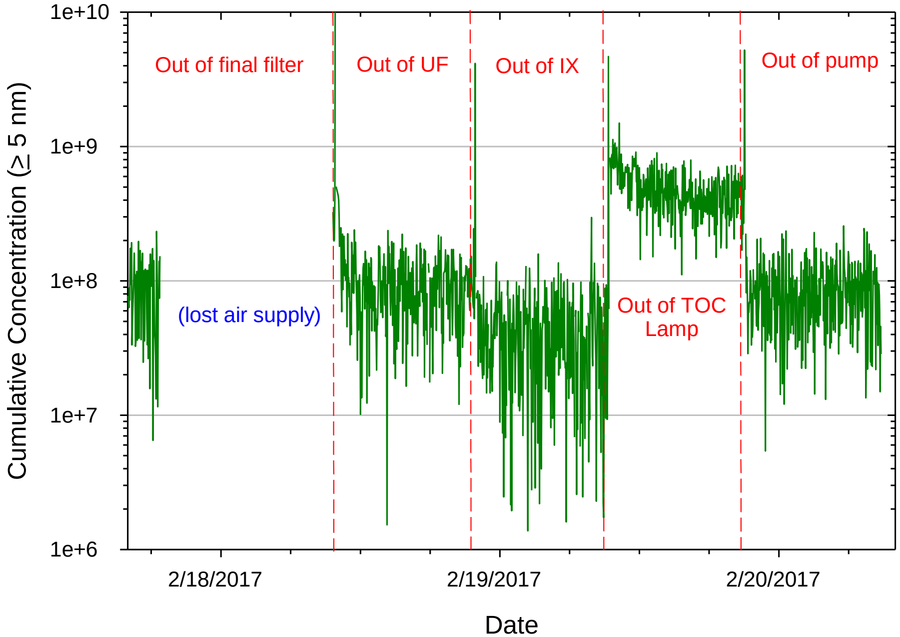
Relevant Dates and Instruments

- January 26-27, 2017
  - Sanitized system with Minncare® cold sanitizer.
  - Installed UF Module
  - Installed 2-20” filters
  - Replace final polish resin.
- February 17-19, 2017
  - CTA LNS#1
  - Lighthouse NC30+
  - Tandum KFMT Nanoparticle Nebulizer with AD Spot Sampler.
- April 5 – May 4, 2017
  - CTA LNS#1
  - Tandum KFMT Nanoparticle Nebulizer with AD Spot Sampler.

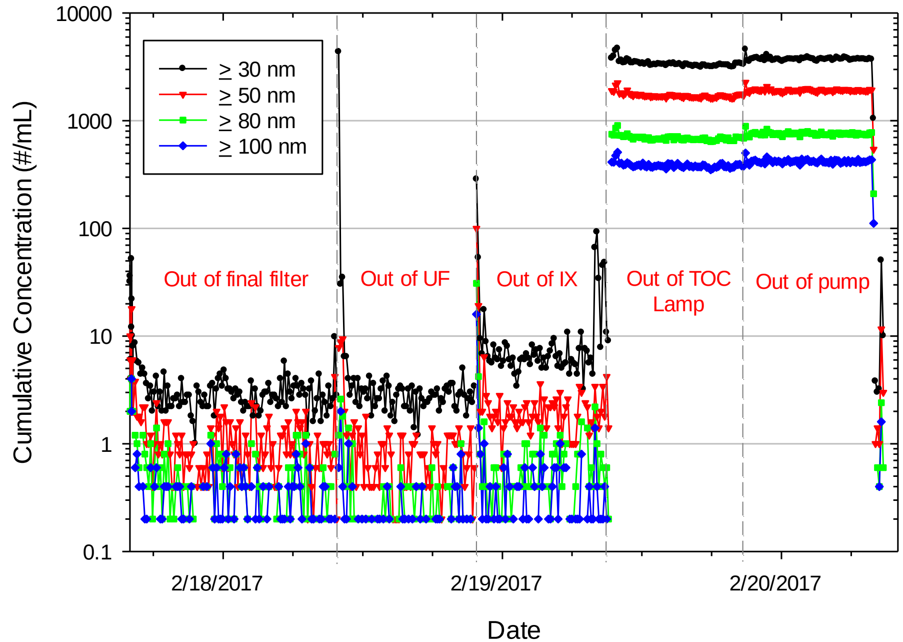
Test Results

Test Results – LNS vs OPC

Liquid Nanoparticle Sizing System



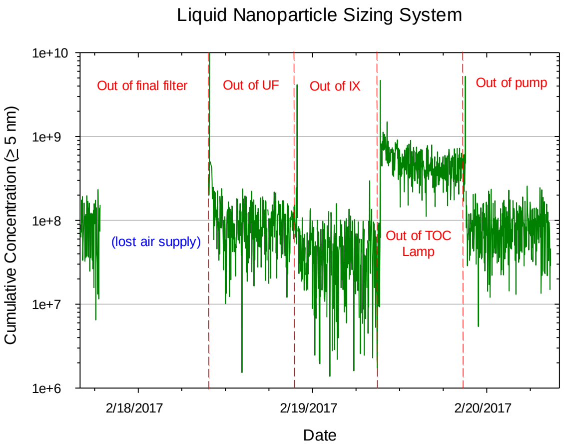
Lighthouse NC30+

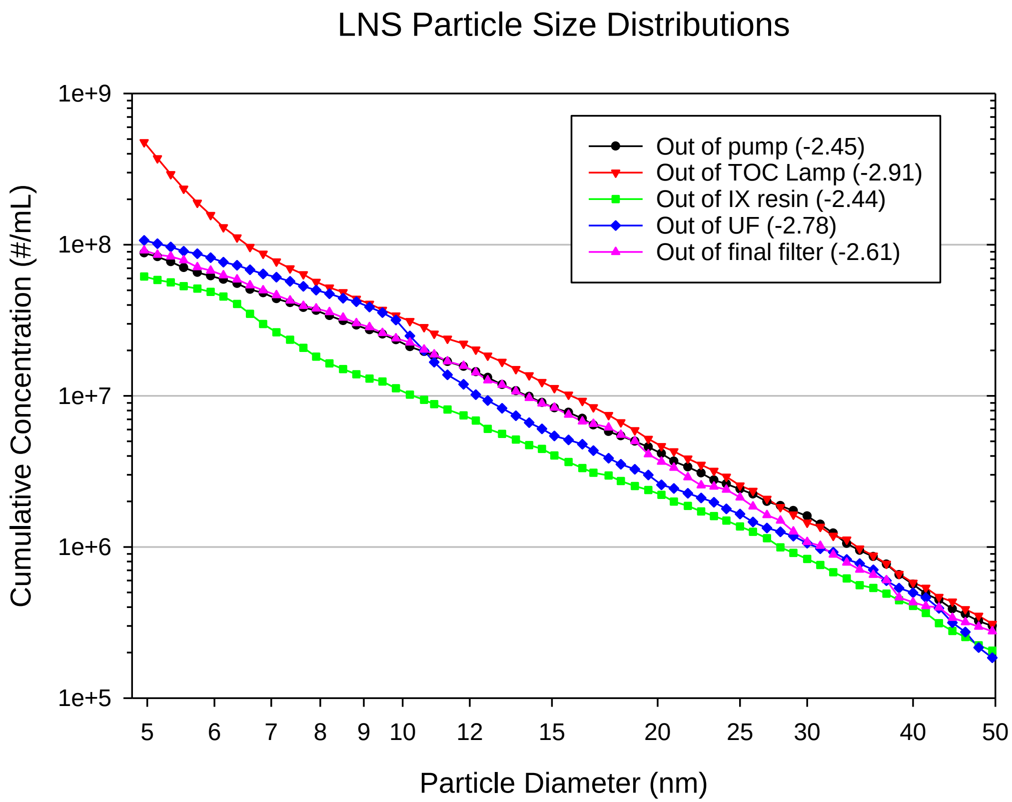


Observations:

1. Highest LNS and OPC concentrations are out of the TOC lamp.
2. Lowest OPC concentration is out of the UF/MF.
3. Lowest LNS concentration is out of the ion exchange resin

Test Results – LNS Particle Size Distribution

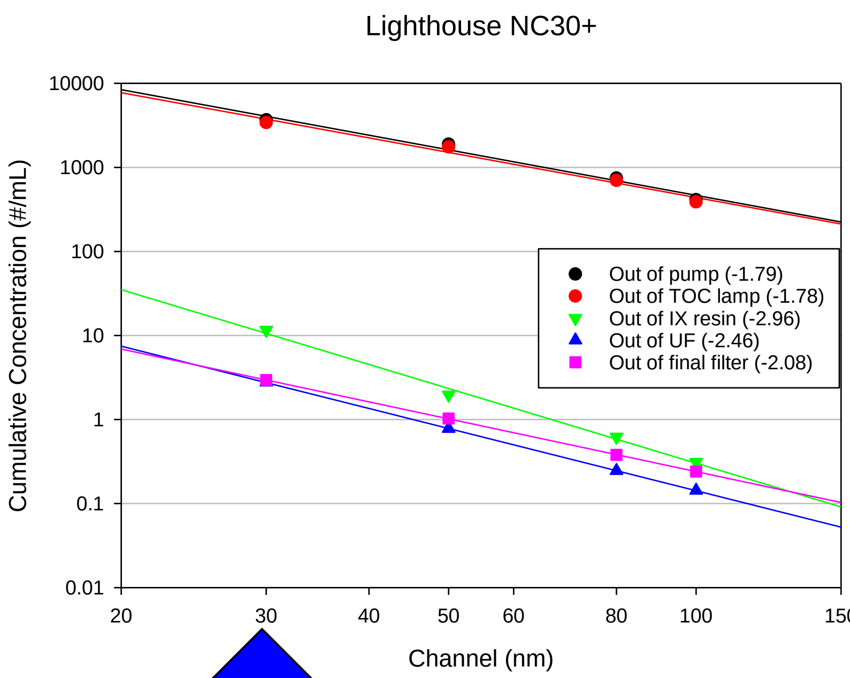


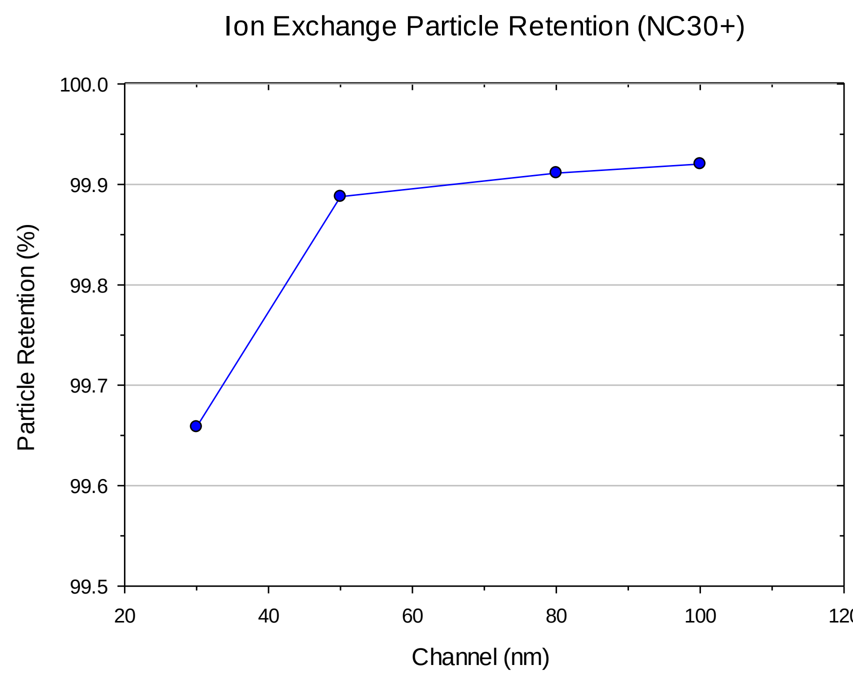


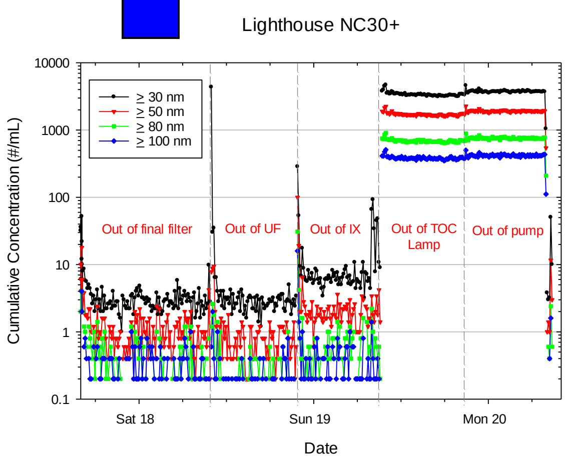
Observations:

1. Increase in small particle size distribution out of the TOC reduction lamp. Could be small particle formation/generation in the TOC lamp and/or increased level of dissolved species (organic or silica).
2. Sharp drop in concentration after UF at 10 nm.
3. Lowest LNS concentration is after IX, not UF or MF

Test Results – OPC Particle Size Distribution



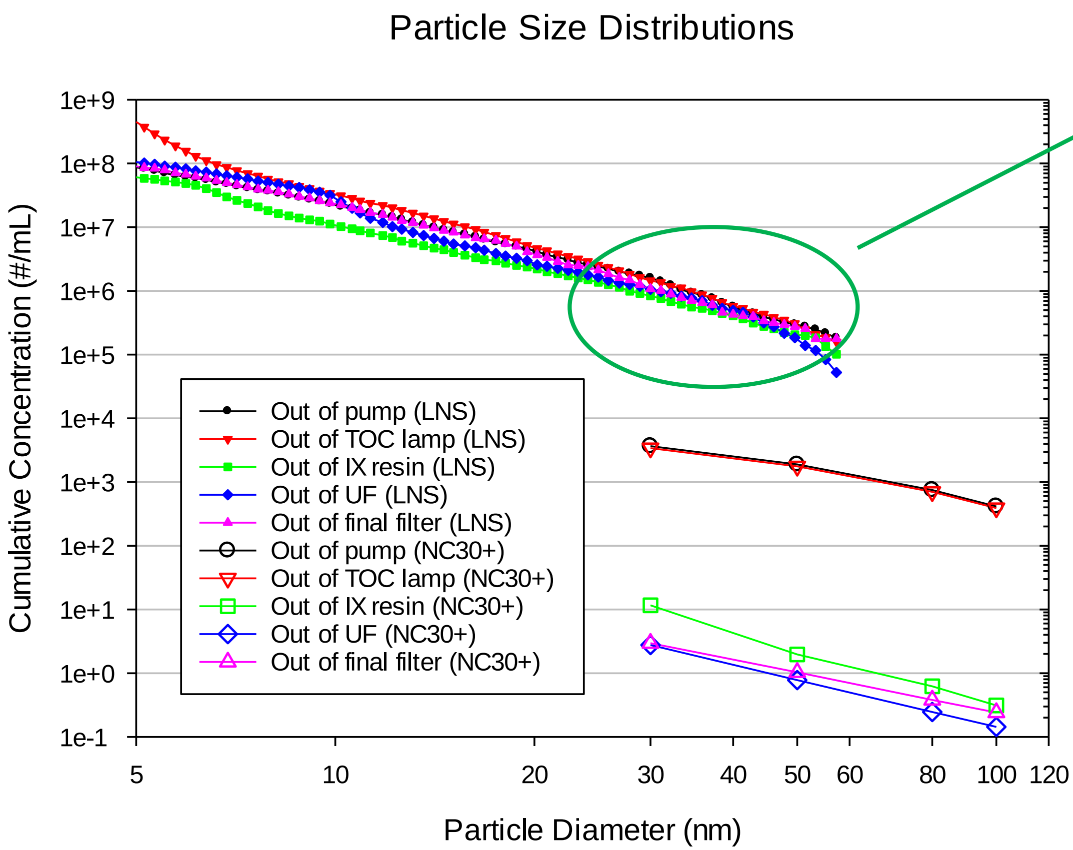


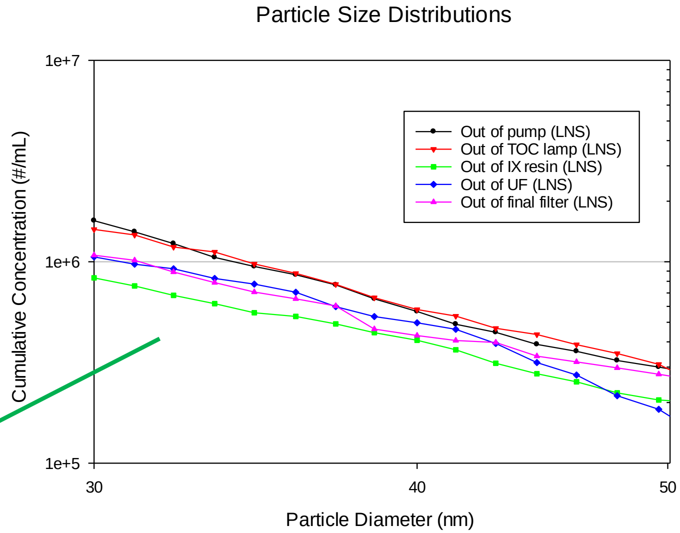


Observations:

1. The ion exchange resin exhibited significant particle retention, > 99% of particles detected by OPC ≥ 30 nm.
2. Unusually flat slope (log-log < -2) out of pump and TOC lamp. Possibly consequence of the sanitizing.
3. Flat slope out of UF/MF is also unusual. Would expect -3.5 to 4. Possibly shedding from the UF and MF's.

Test Results – LNS vs OPC

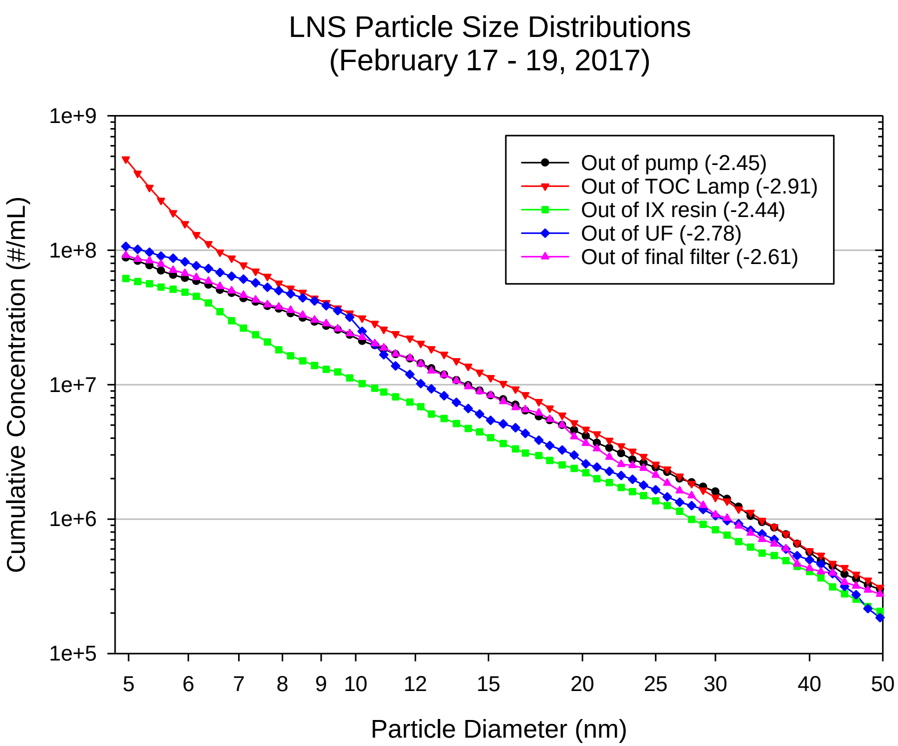


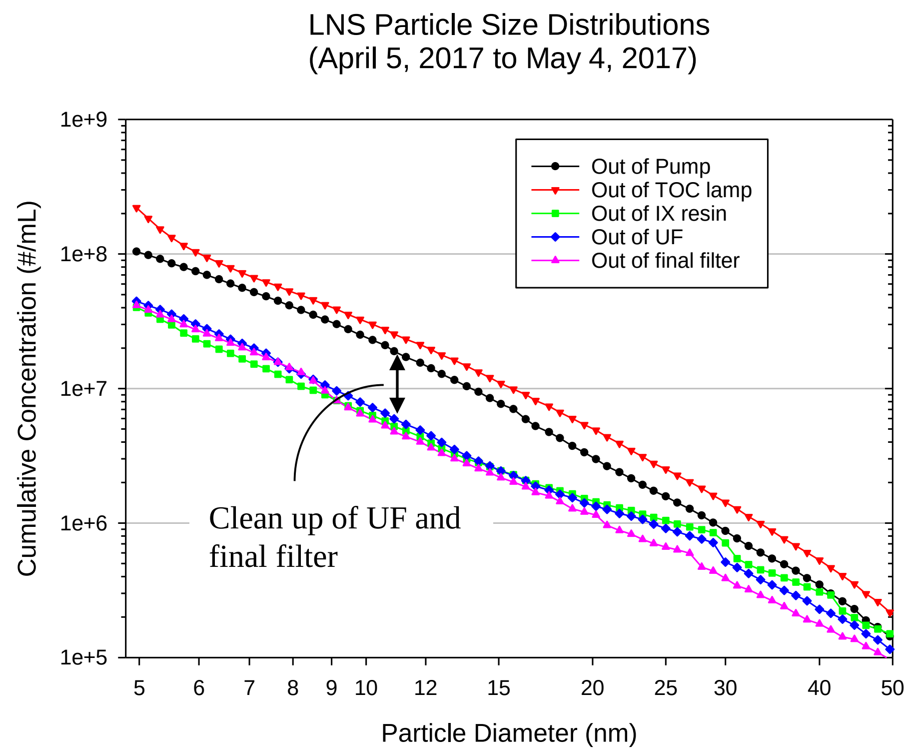


Observations:

1. Sizing order different between two techniques out IX resin. Measuring different particles?
2. Large drop (3 orders) in concentration after IX observed with OPC but not observed by LNS. Why??
3. Concentration measured by LNS are 3 to 6 orders of magnitude higher than OPC. Why??

PSD change with run-time (6 weeks)



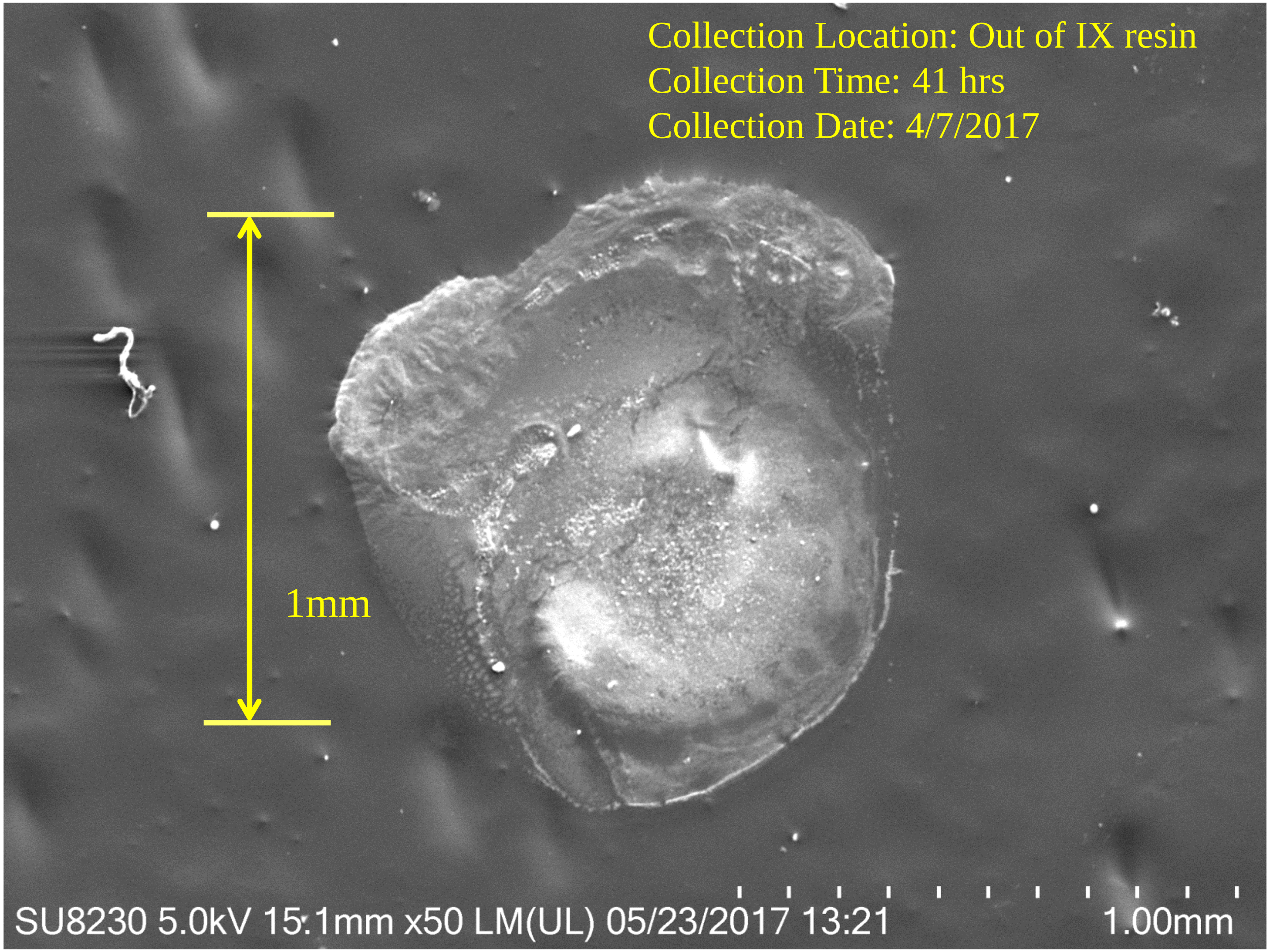


Observations:

1. Little change in PSD prior to IX (after pump and TOC lamp).
2. Cleanup observed in IX, UF and final filter. Small particle (< 20 nm) concentration are similar post IX.
3. Trend in large particles (> 20 nm) are consistent with prior OPC data.

Focused Aerosol Deposition
SEM/EDS Analysis

Spot Sampler SEM/EDS Analysis



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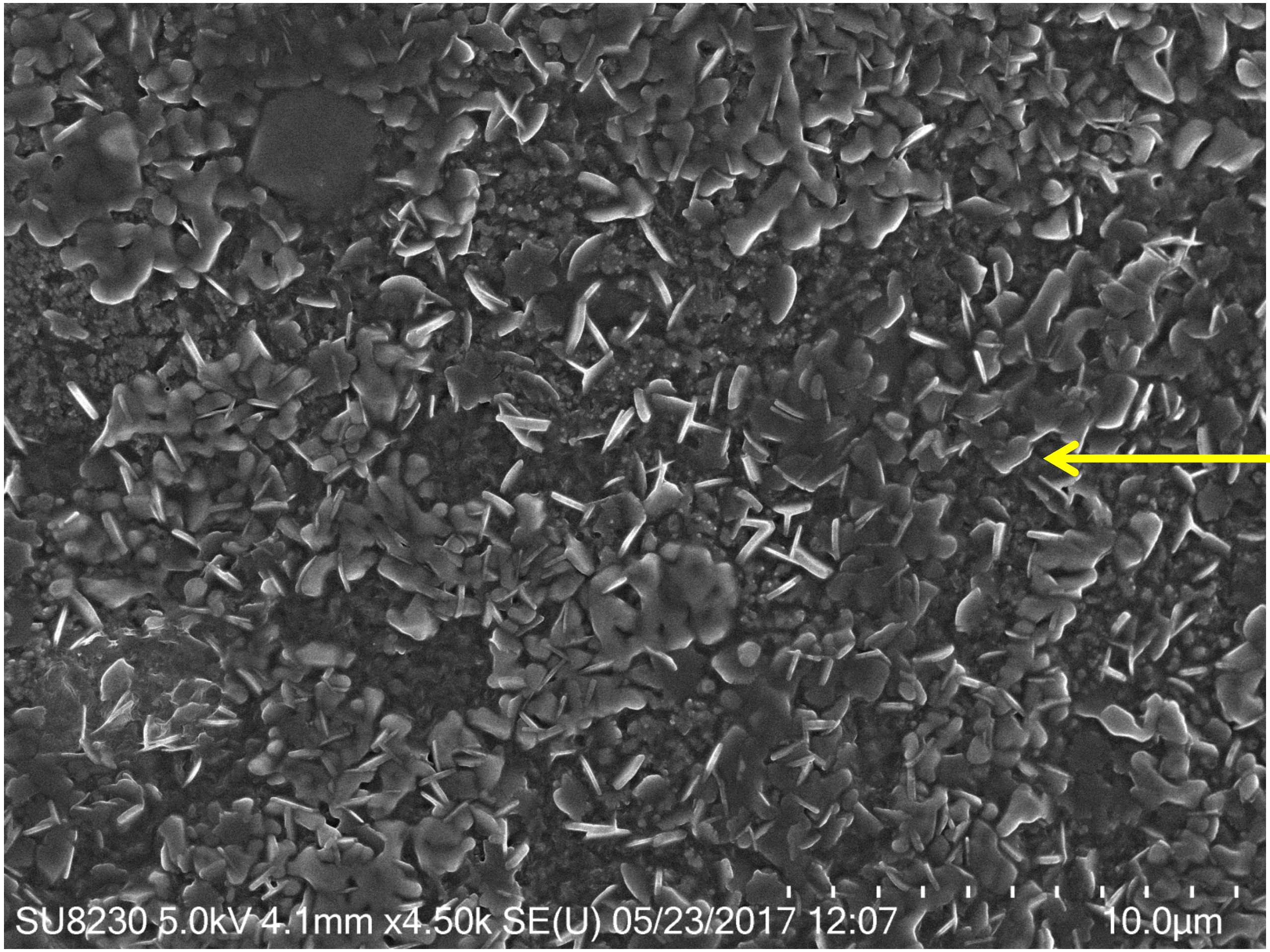

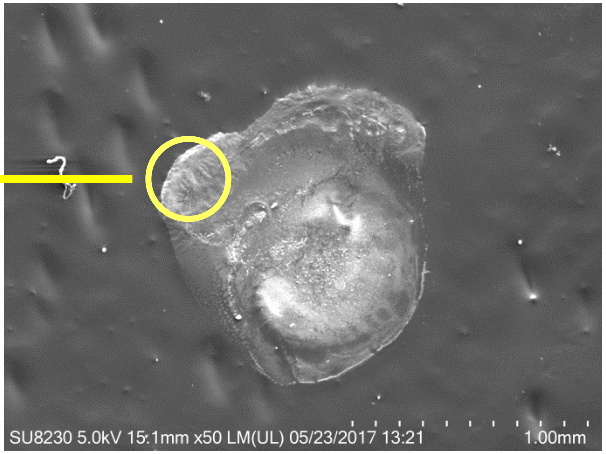

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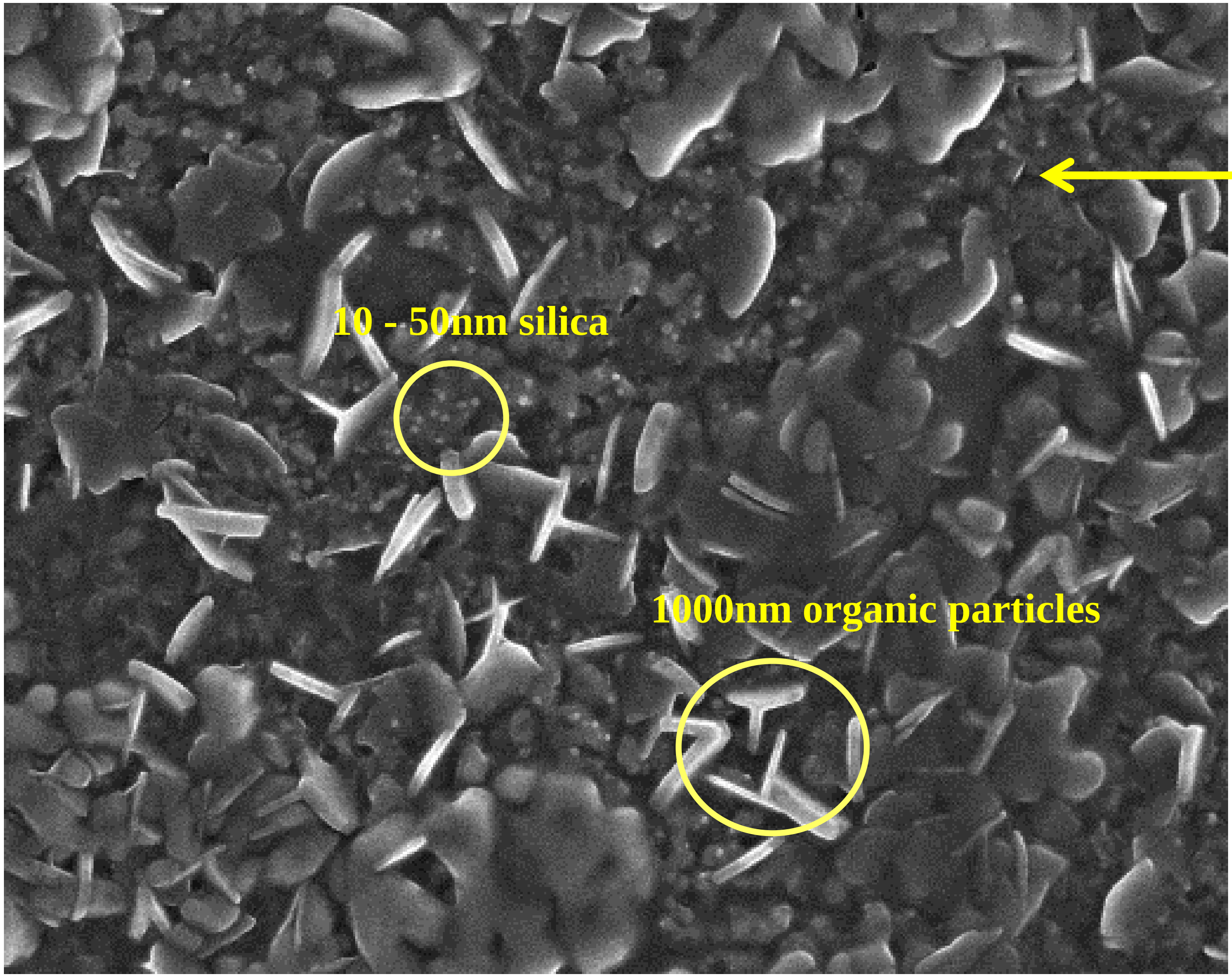
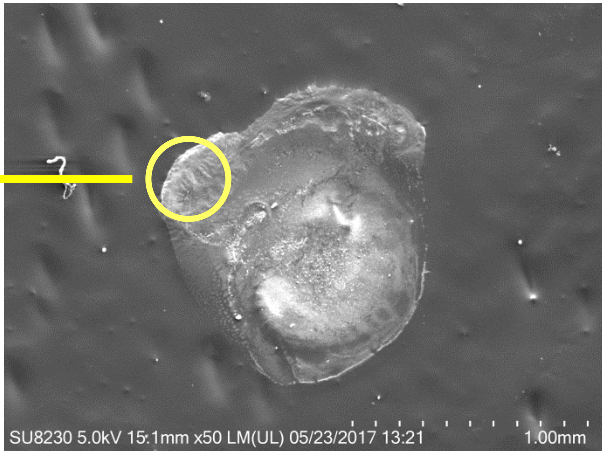
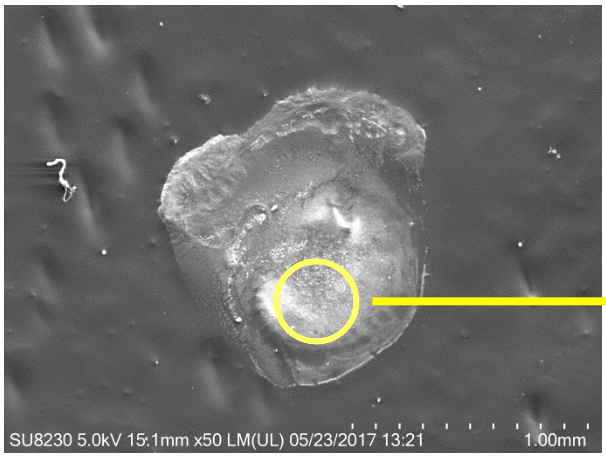
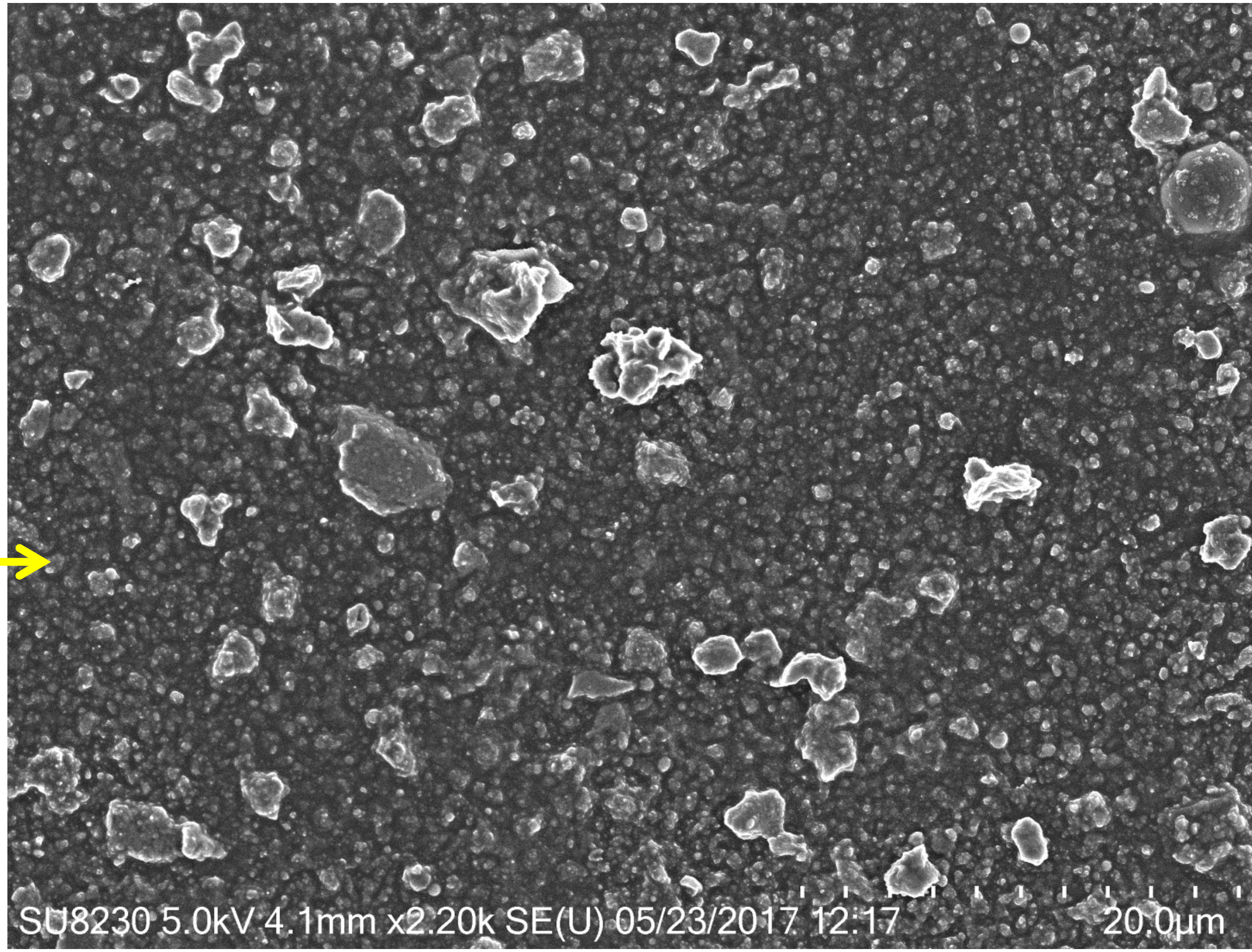




Elements Detected:
- Carbon
- Oxygen
- Silicon
- Sulphur
- Zinc
- Magnesium

Spot Sampler SEM/EDS Analysis





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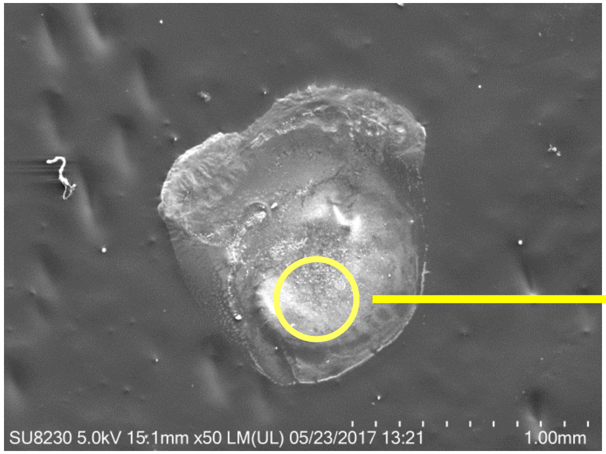
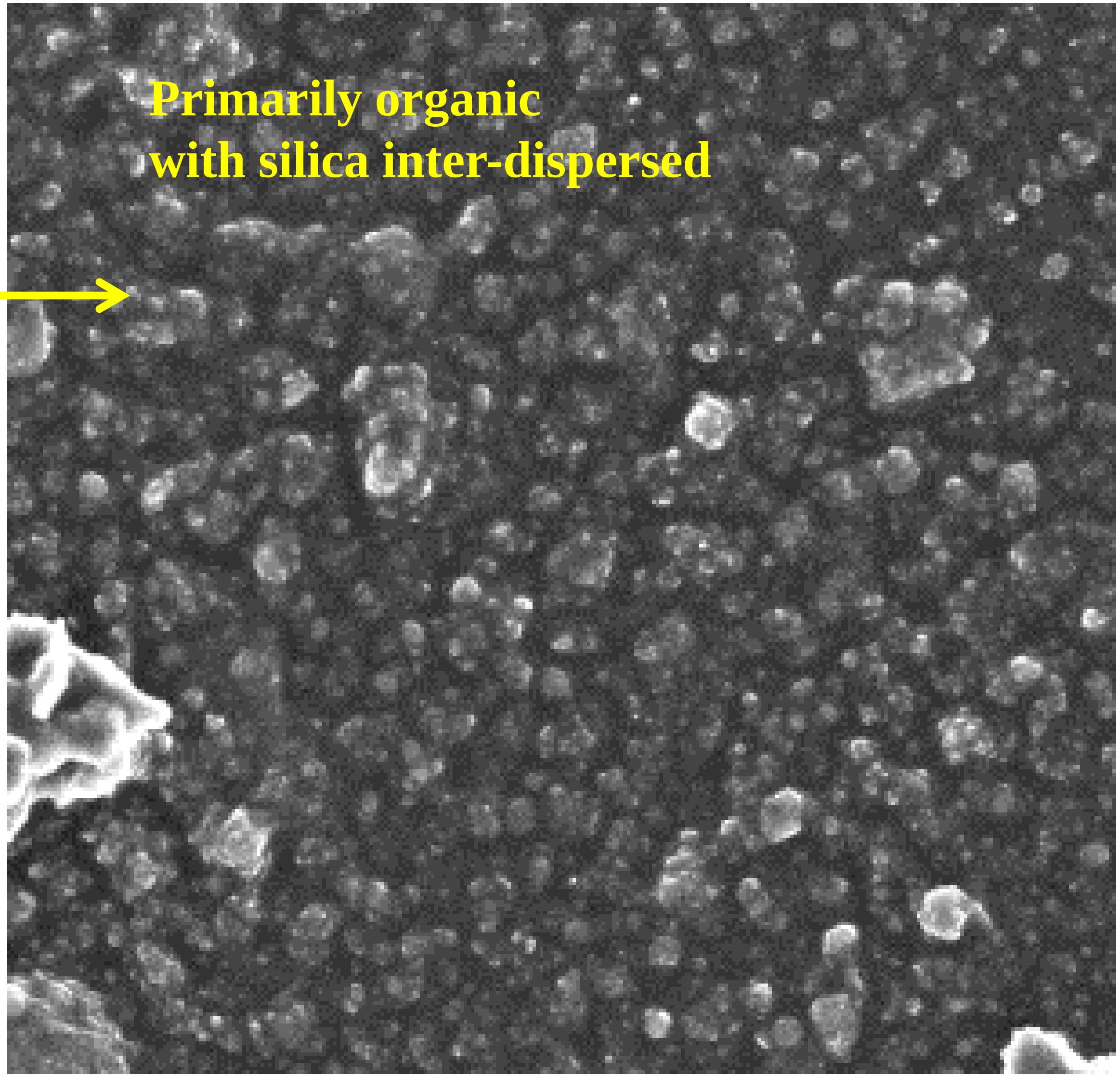
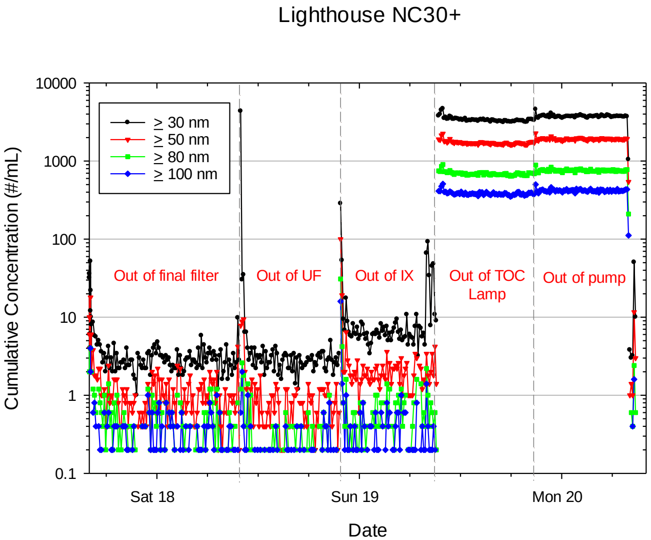


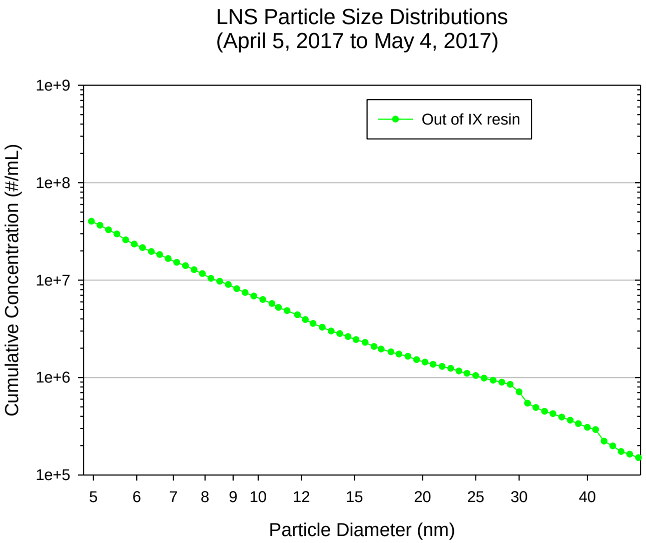


Elements Detected:

- Carbon
- Oxygen
- Sulphur
- Sodium
- Magnesium
- Silicon

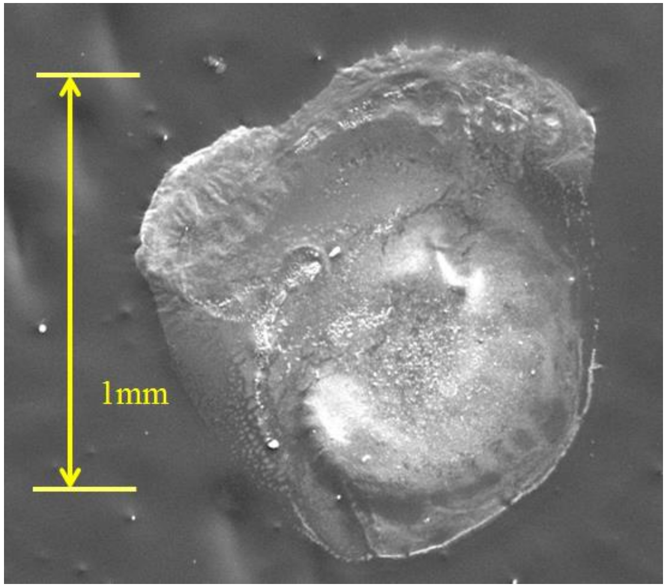
Estimated Spot Size – OPC vs LNS



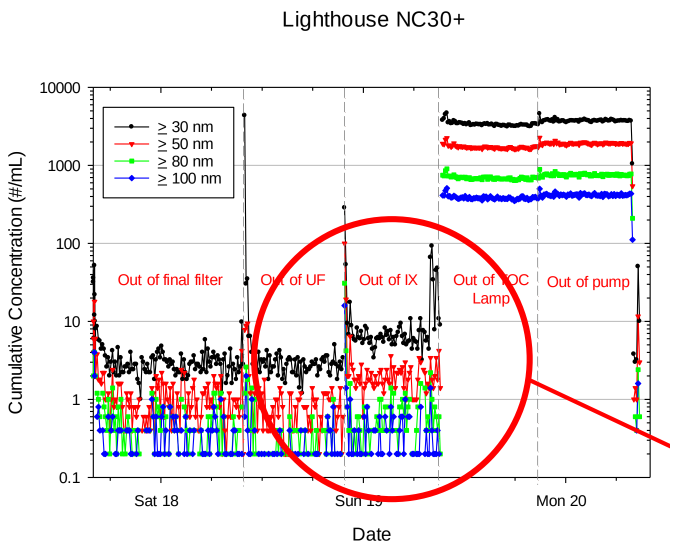


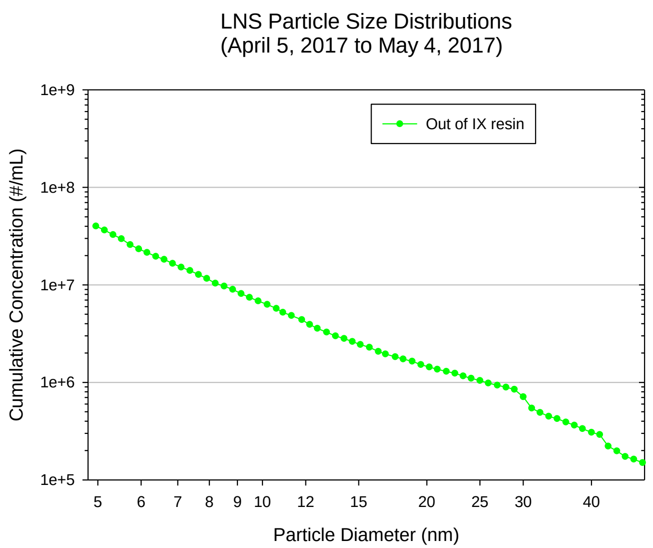
Approach:

- Integrate differential size data.
- Only consider particles > 7 nm.
- Extrapolate OPC PSD to 7 nm using power law: $f(x) = K * (1/\mathrm{d}^{\mathrm{n}})$ where n=3.
- Assume that the OPC measurements from February are still valid.
- Assume 1% packing efficiency.
- Assume 2D packing only.



Estimated Spot Size – OPC vs LNS





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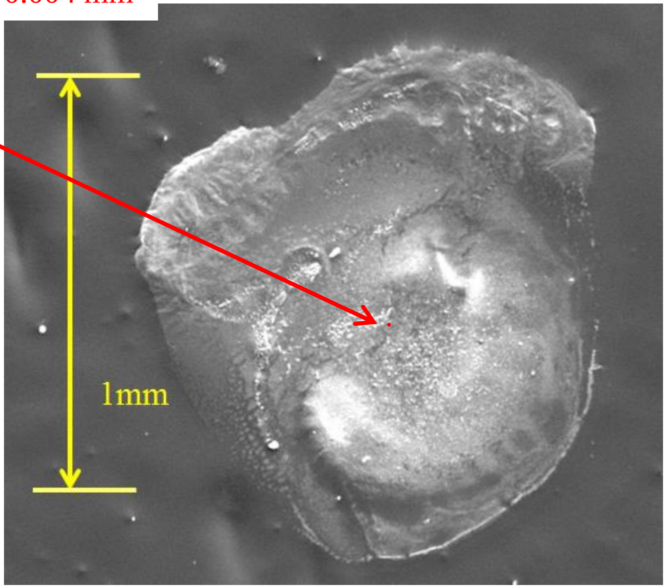
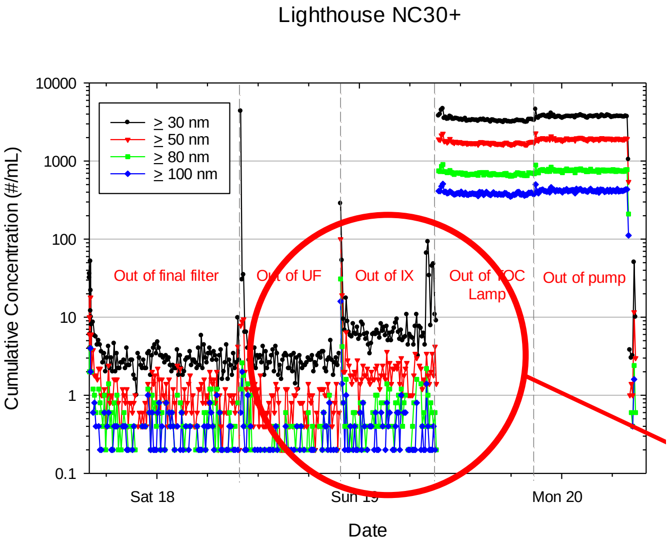
Projected spot diameter based on integrated OPC data – 0.004 mm



Estimated Spot Size – OPC vs LNS



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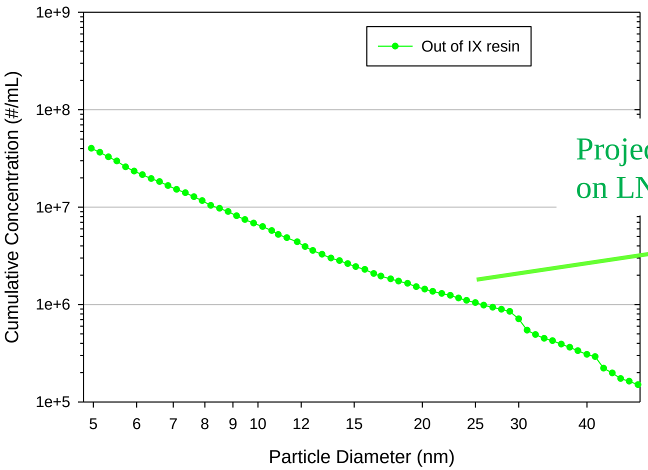
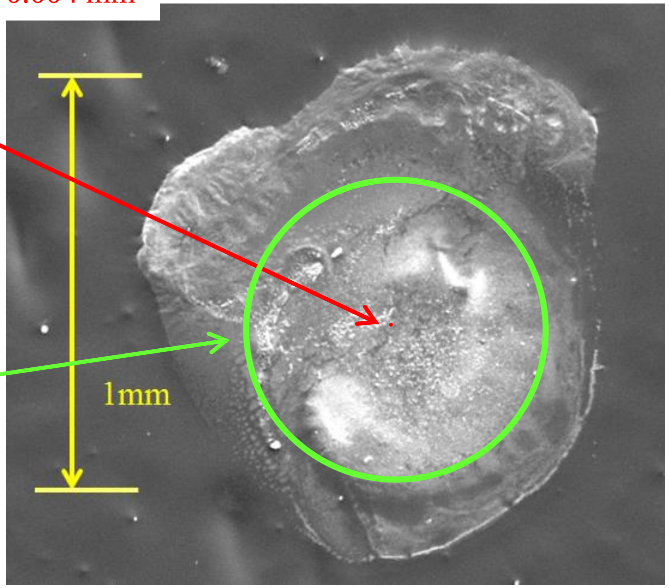


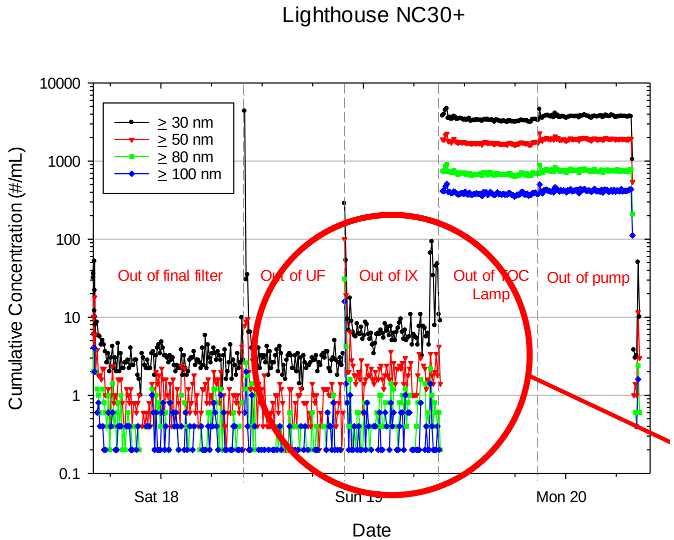
Projected spot diameter based on LNS data – 0.63 mm



Estimated Spot Size – OPC vs LNS

Focused Aerosol Deposition SEM results seem to correlate better with LNS data than OPC data in this particular study.

Additional work will be required to validate this observation in other locations within the UPW system.



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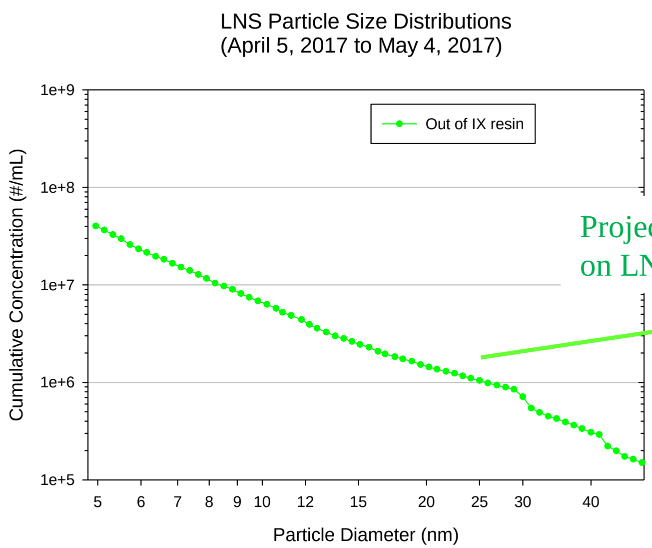

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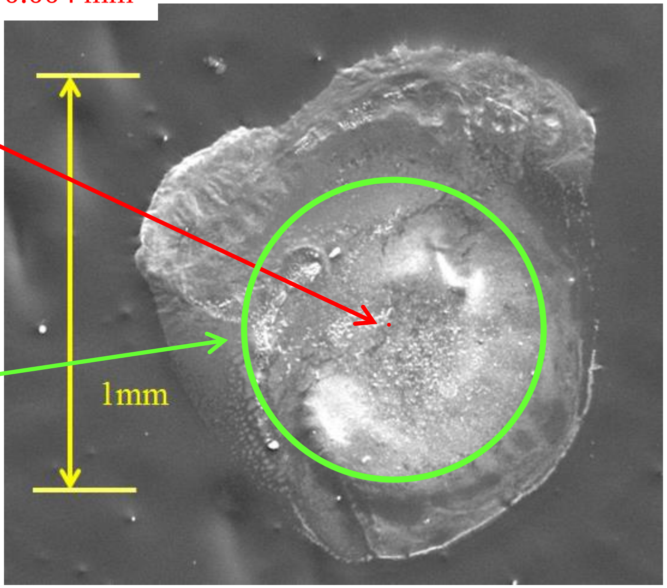

Key Observations

- While particle concentrations varying significantly between the LNS and OPC, trends were consistent except out of the IX resin.
- Focused Aerosol Deposition with SEM/EDS appear to support the higher concentrations measured by the LNS.
- The vast majority of the material deposited by the AD Spot Sampler were organics and silica. Some organics materials are more difficult to detect by light scattering due to their refractive index being close to water. Same is true for silica. This may be one of the reasons for the differences observed between the instruments.
- The TOC reduction lamp was a significant contributor to sub-20 nm contamination. It is uncertain if these are particles, dissolved material or both. Further analysis using Focused Aerosol Deposition may help to determine the nature of these particles.
- The mixed-bed IX resin used in this test has significant particle removal capability, especially greater than 30 nm, except for silica.

Recommendations

- Continue developing our understanding of the differences between aerosol-based and light-scattering particle measurement techniques:
  - Conduct parallel testing with aerosol-based and light scattering instruments.
  - Include alternate technology instruments as they become available.
  - Utilize new tools such as Focused Aerosol Deposition to collect and analyze the nanoparticles found in modern UPW systems.
  - Work to establish a quantitative correlation between FAD, aerosol-based and OPC results.
  - Continue controlled testing to measure the response of the instruments to nanoparticles of varied compositions.

Acknowledgements

- University of Minnesota’s Materials Charactization Lab for the use of the Hitachi SU 8230 FE/SEM and Thermo Noran System 7 Spectral Imaging (EDS) instruments.