ULTRAPURE MICRO 2020

Measurement of Particle Precursors in Ultrapure Water

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CT Associates, Inc., Eden Prairie, Minnesota

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Creative Technology

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Outline

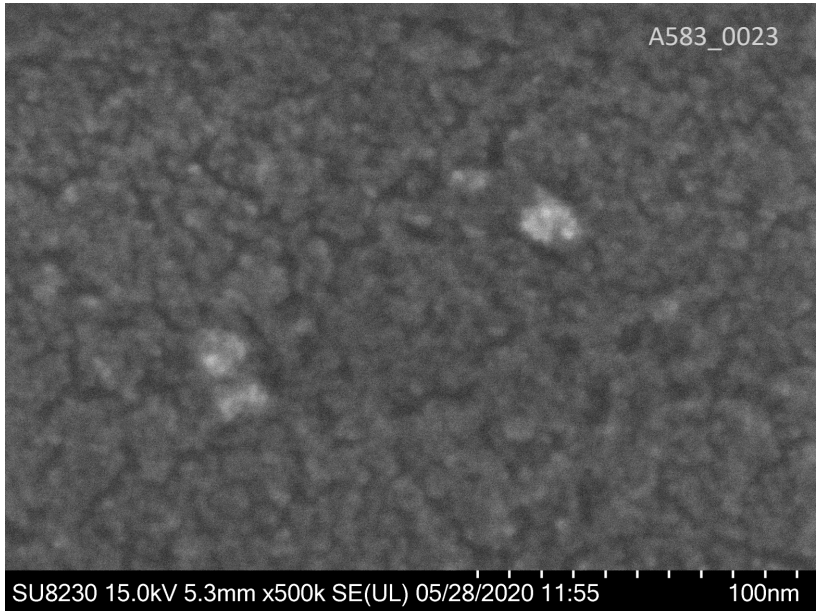
- Potential sources of sub-10nm particle and particle precursors (FAD examples)
- Experimental Plan
- Particle detection instrumentation
- Candidate high molecular weight organics (HMWO) reference material.
- Instrument detection efficiency and response to HMWO
- Retention of HMWO via filtration
- Key findings

Introduction and Problem Statement

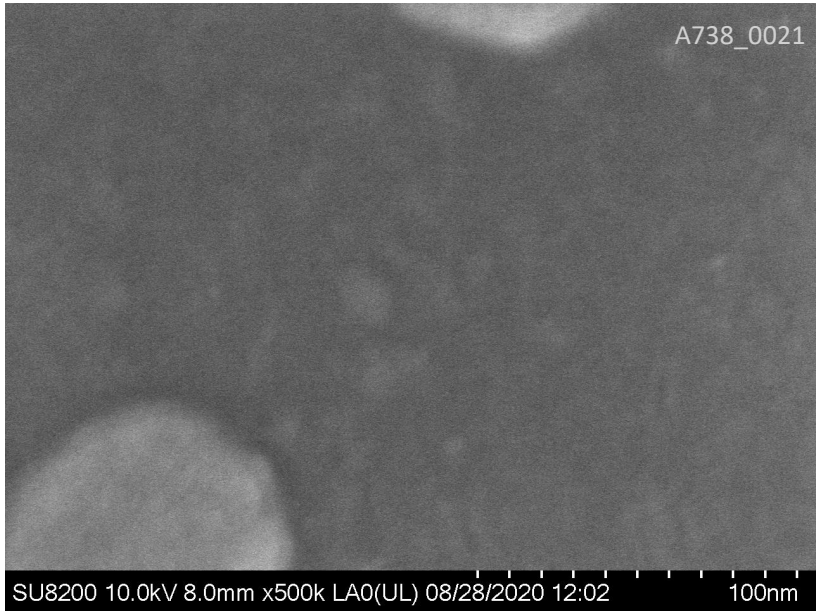
- Particle precursors are dissolved or molecular materials in a fluid smaller than a “critical size” that may form a particle of “critical size” when a droplet dries or precipitates under changing conditions. Examples – high molecular weight organics (HMWO) and silicates.
- There are potentially multiple sources of particle precursors in UPW systems including ion exchange resins, tubing and piping, fluid handling components and polymeric filter and membrane materials.
- Conventional light-scattering particle detection technologies are challenged to measure particles below 20nm or particle precursors.
- Liquid nebulization in conjunction with aerosol-based condensation particle counting has the proven ability to detect and count particles as small as 2.5nm and may serve as a useful tool to measure particle precursors.

Potential sources of sub-10 nm particles and particle precursors

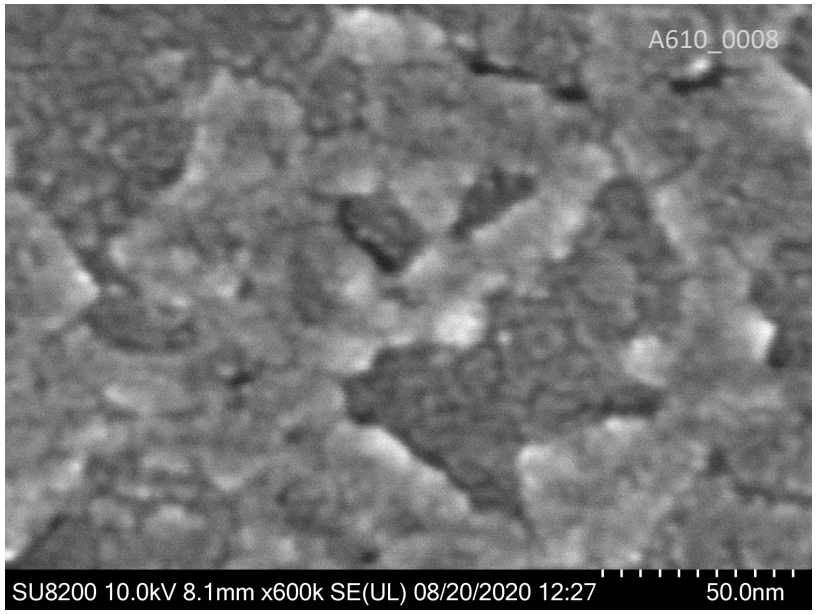
Ion Exchange Resin



Hot UPW Fluoropolymer Piping Extract



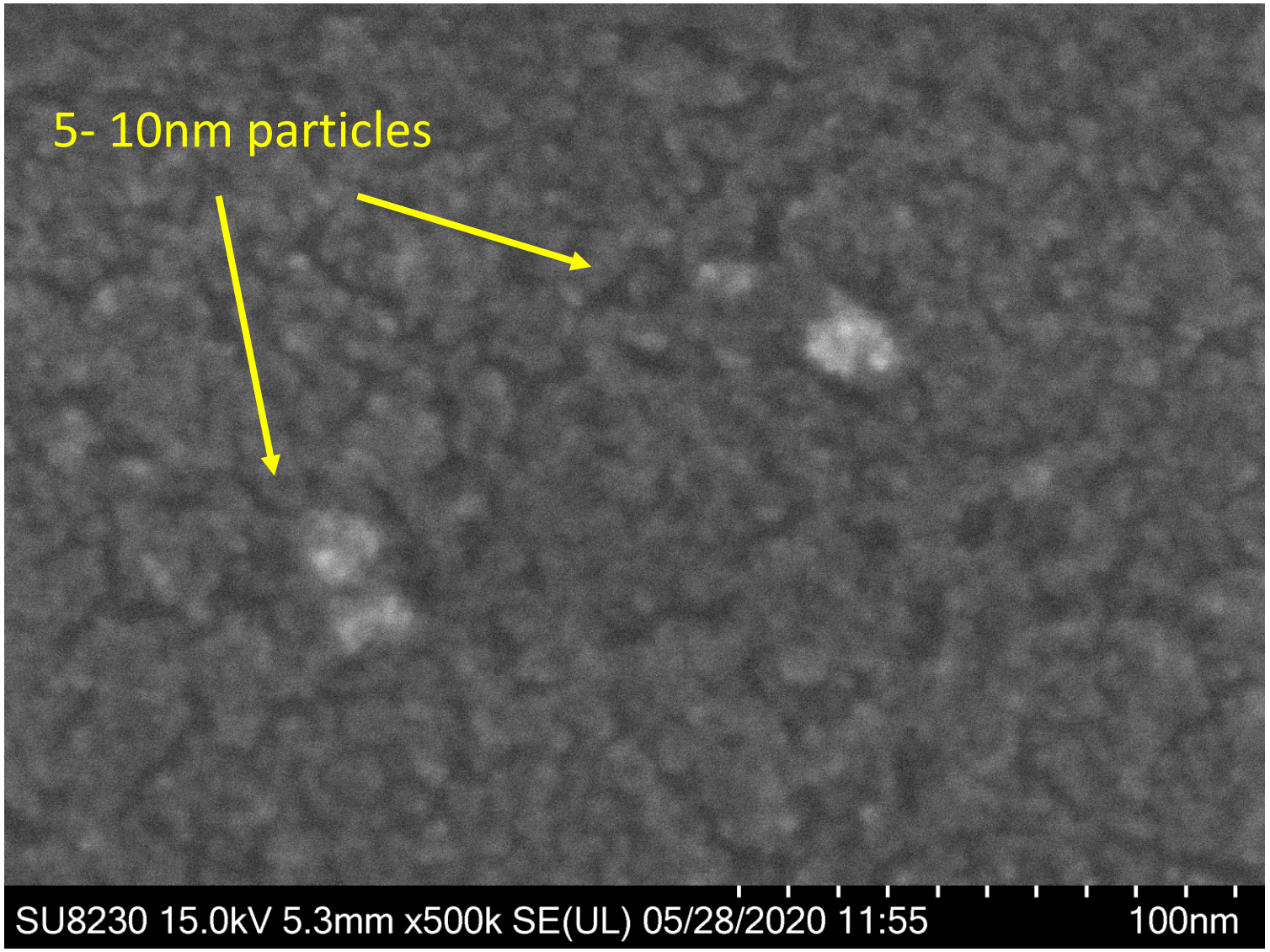
Isopropyl Alcohol



- Sample collection by Focused Aerosol Deposition (FAD) (CT Associates, Inc)
- Sample analysis by FESEM (Hitachi SU8230)

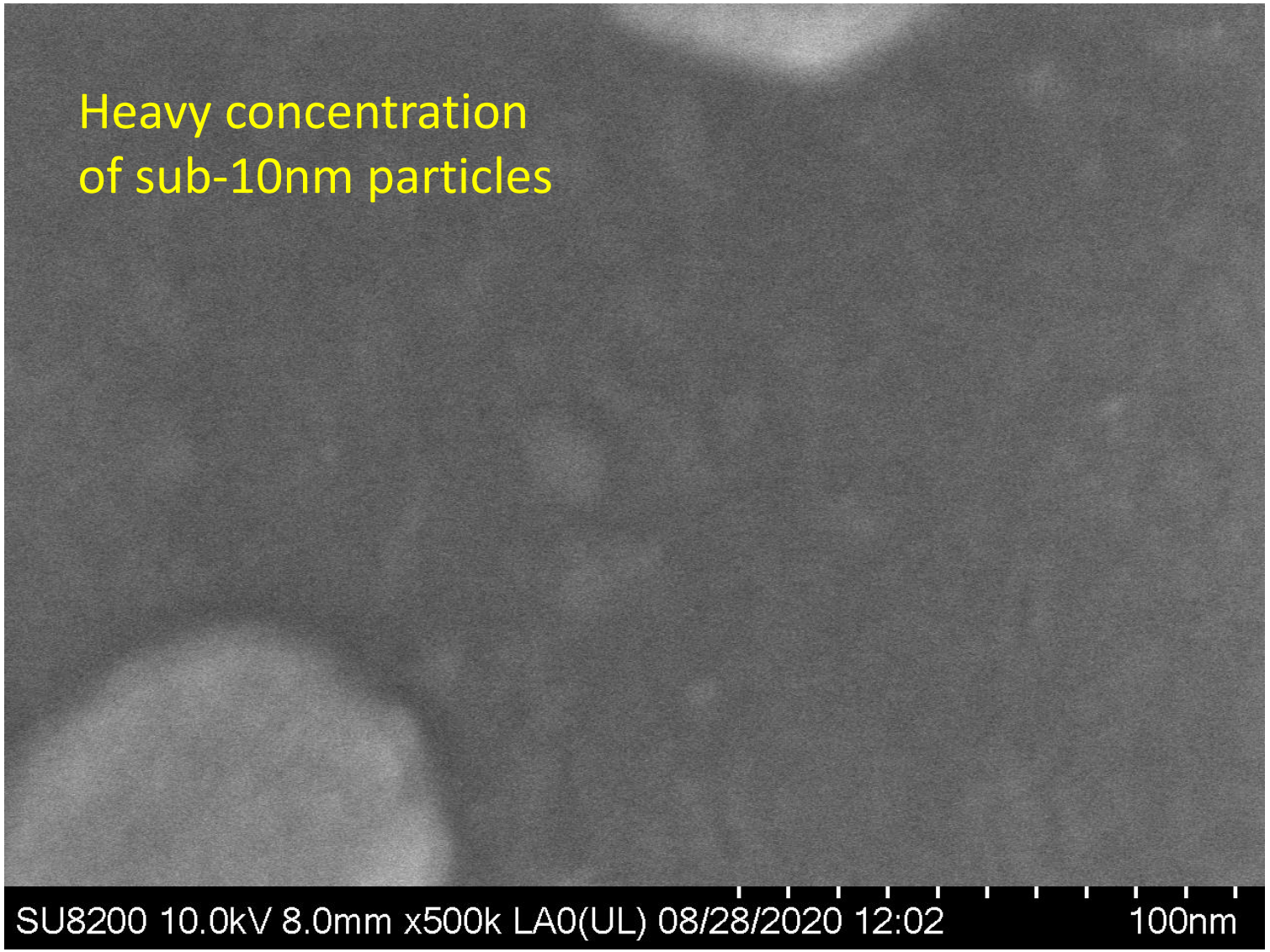
Ion Exchange Resin Effluent

- Sample collection by Focused Aerosol Deposition (FAD) (CT Associates, Inc)
- Sample analysis by FESEM (Hitachi SU8230)



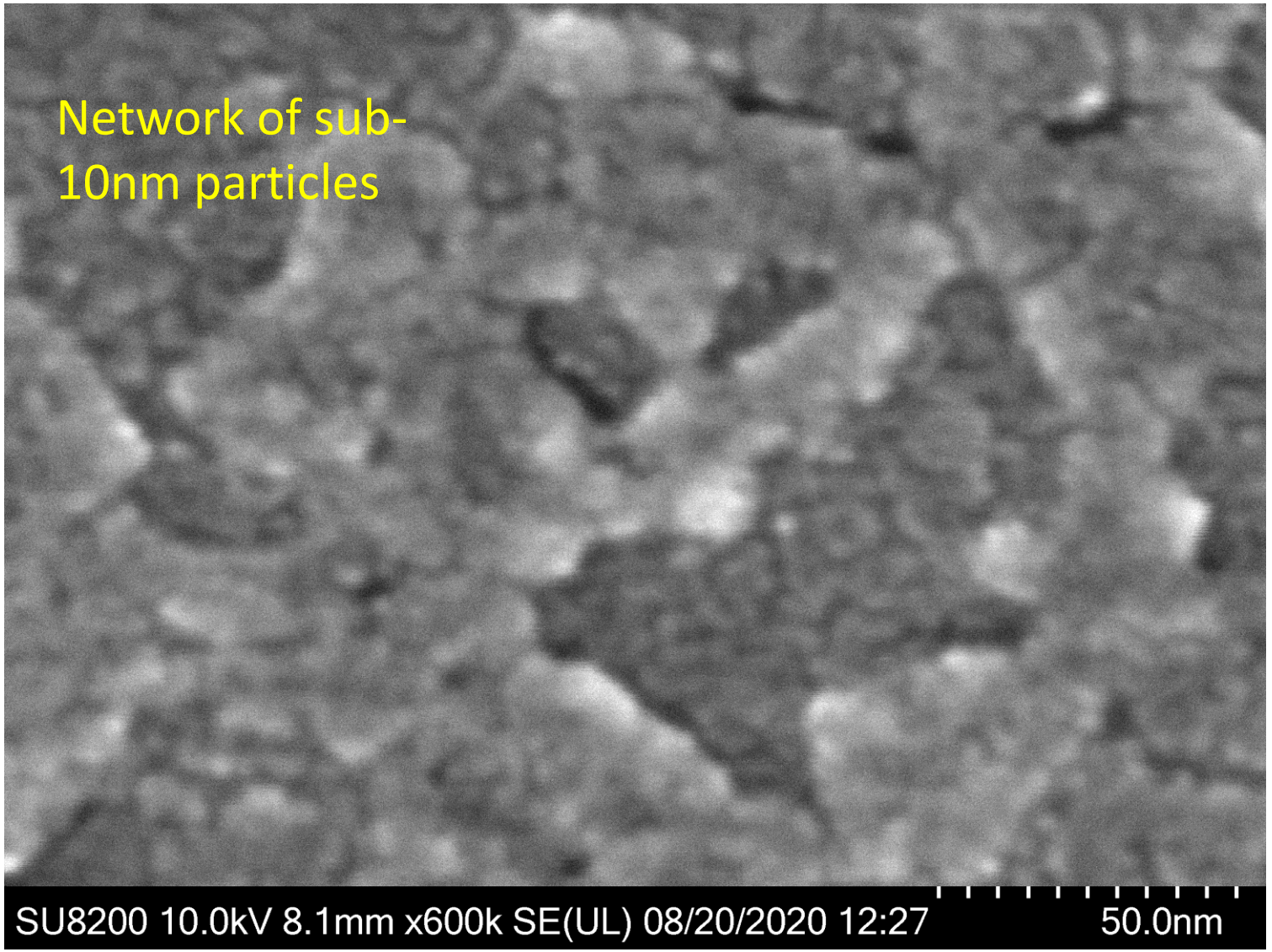
Hot UPW Fluoropolymer Piping Extract

- Sample collection by Focused Aerosol Deposition (FAD) (CT Associates, Inc)
- Sample analysis by FESEM (Hitachi SU8230)



“Gigabit” Isopropyl Alcohol

- Sample collection by Focused Aerosol Deposition (FAD) (CT Associates, Inc)
- Sample analysis by FESEM (Hitachi SU8230)



Experimental Plan: Overall

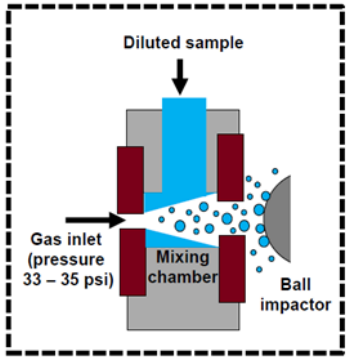
1. Measure UPW monitoring instrument responses a high-molecular weight organic (HMWO) molecule representative of one expected to be found in a UPW system
2. Utilizing the methodology defined in SEMI C79 filter testing guideline, measure filter retention of HMWO particles and particle precursors by cartridge filters.

Particle Detection Instrumentation

Lighthouse Worldwide Solutions NC30+ OPC

Kanomax FMT STPC3 3nm

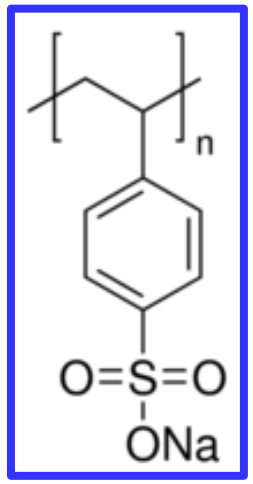
+

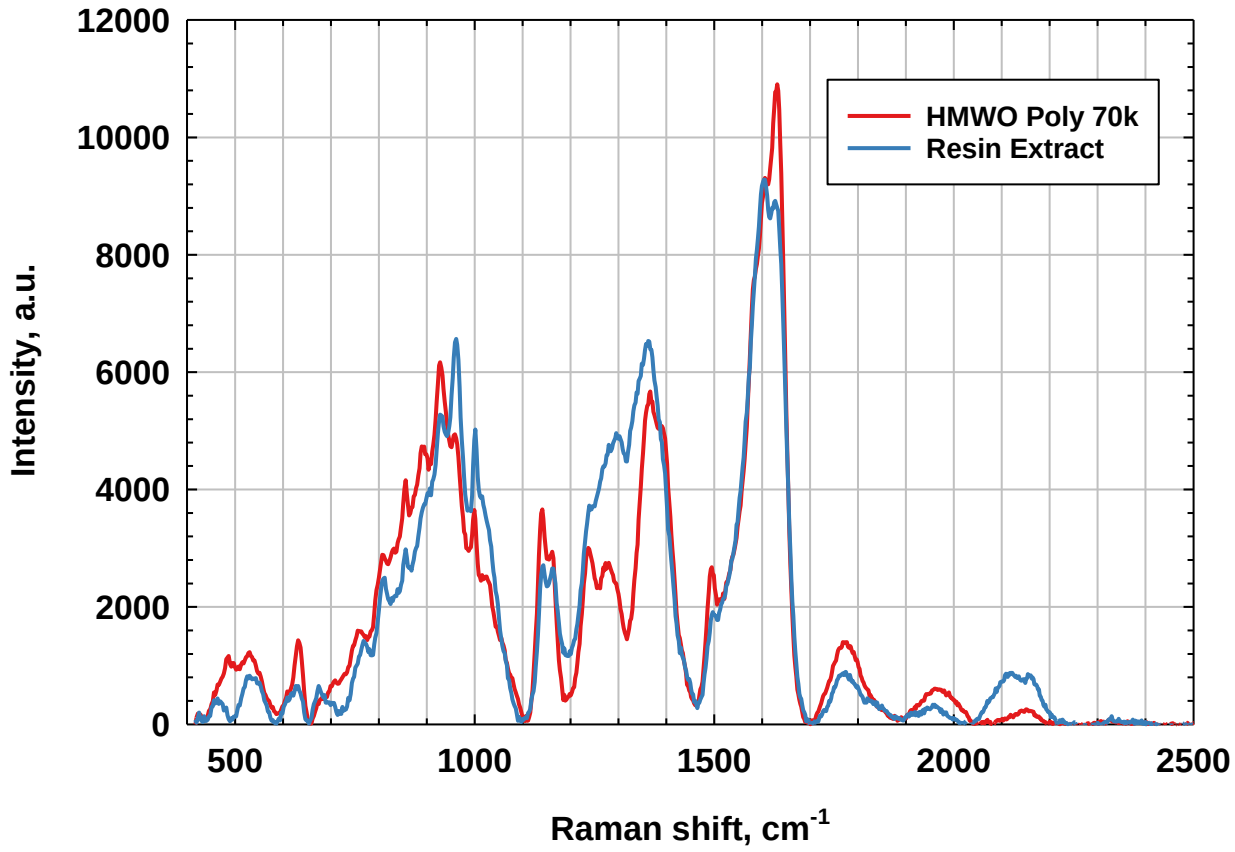


Ultrafine Nebulizer with TSI Model 3775 CPC

Candidate High Molecular Weight Organics (HMWO) Reference Material.

- Commonly used cation exchange resins (CER) are crosslinked polymers of 4-styrensulfonate.
- Species derived from CER can therefore be modeled by poly(sodium 4-styrensulfonate - PS3), which is water soluble and commercially available in multiple molecular weights.





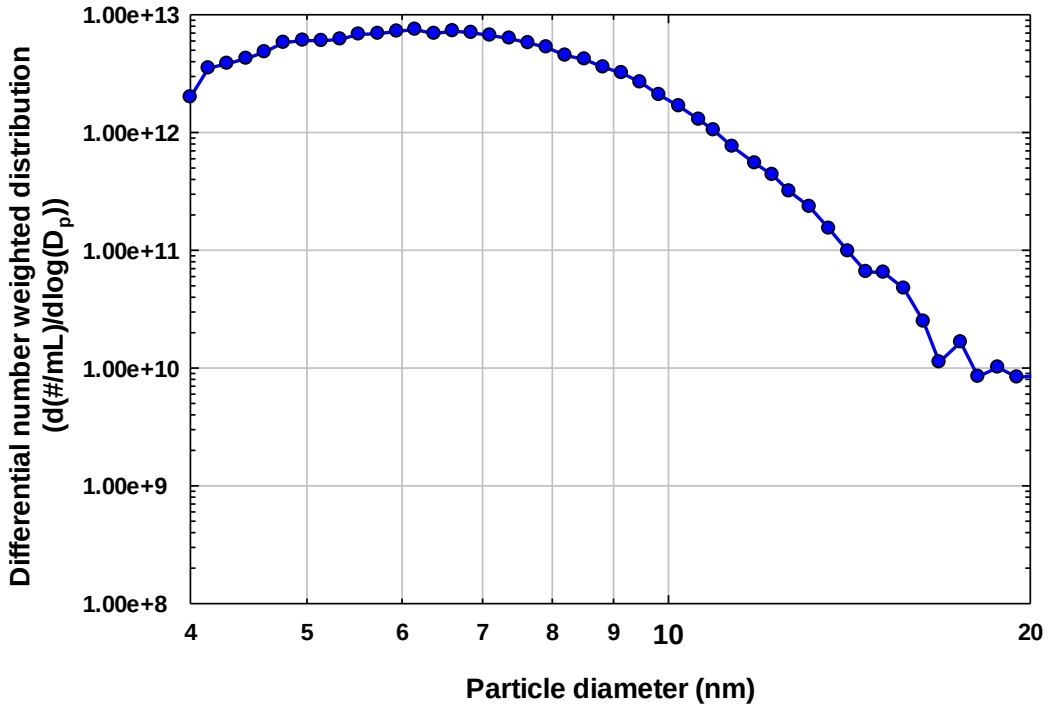
SERS analysis by UNISERS, Zurich, Switzerland

Experimental Plan: Online HMWO injections

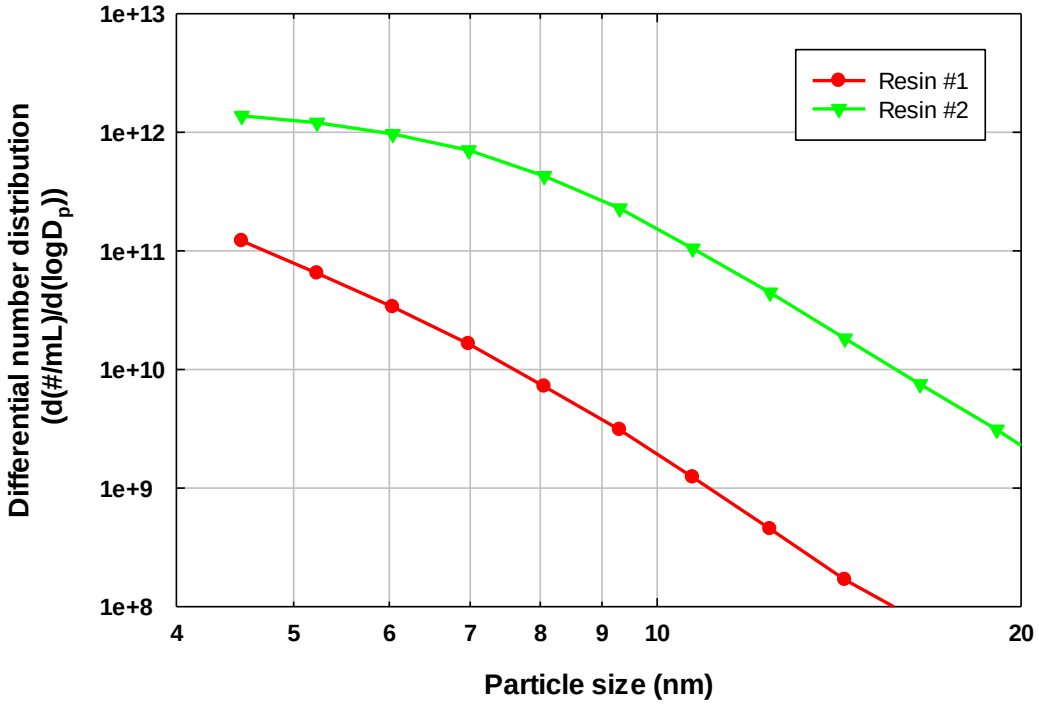
1. Calibrate instruments via 30 nm silica calibration standard.
2. Measure PSD of HMWO poly 70k size distribution using LNS.
3. Measure monitoring instrument response by injecting characterized HMWO into ~ 400 mL/min UHPW. Target concentrations at instruments: b/g, 1 ppt, 10 ppt, 100 ppt.

High Molecular Weight Organics Particle Size Distributions

HMWO 70,000 MW

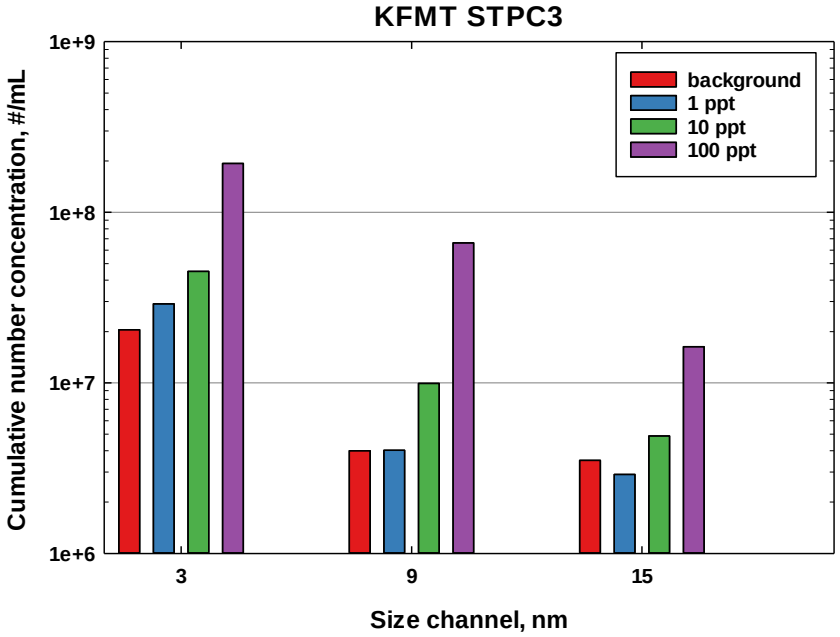


Ion Exchange Resin Extract
(by SEMI C93 grab sample procedure)

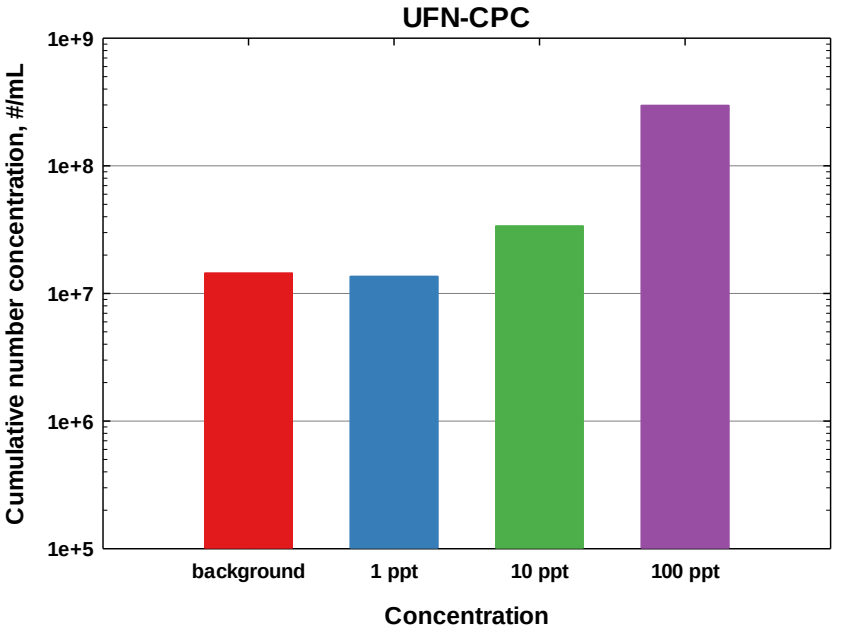


Analysis by Liquid Nanoparticle Sizing (LNS) by CT Associates

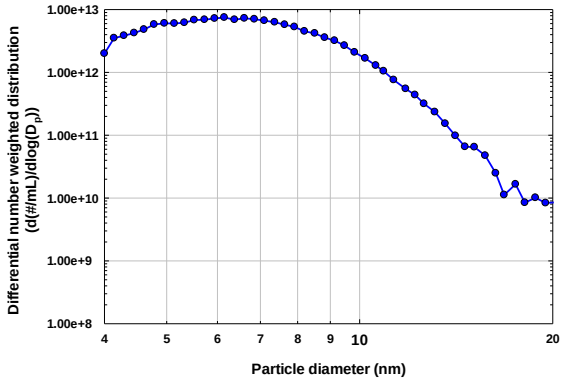
Instrument Response to 70K HMWO

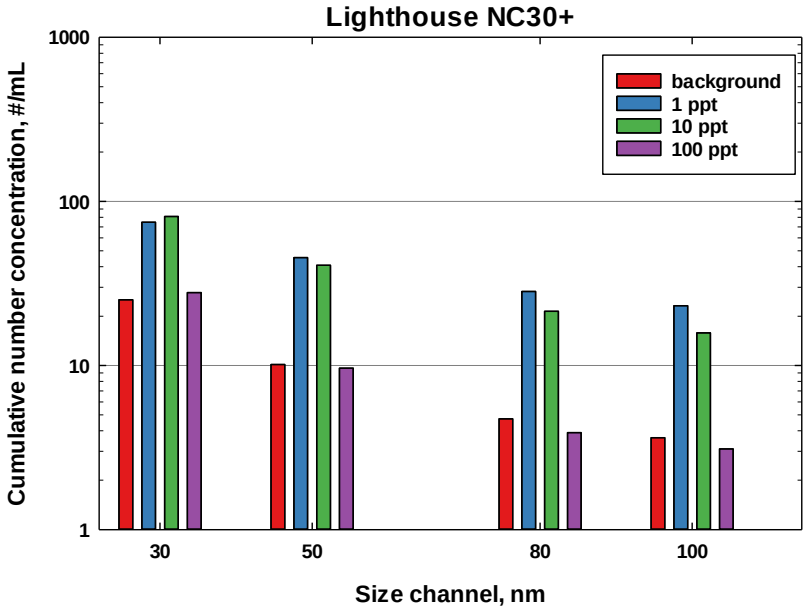


- LNS analysis projects that 1 ppt added HMWO corresponds to ~ 1e6 particles/mL (> 3 nm).
- STPC3 and UFN-CPC correlate well with HMWO concentration changes.
- OPC response is not correlated to HMWO concentration changes.



HMWO 70,000 MW

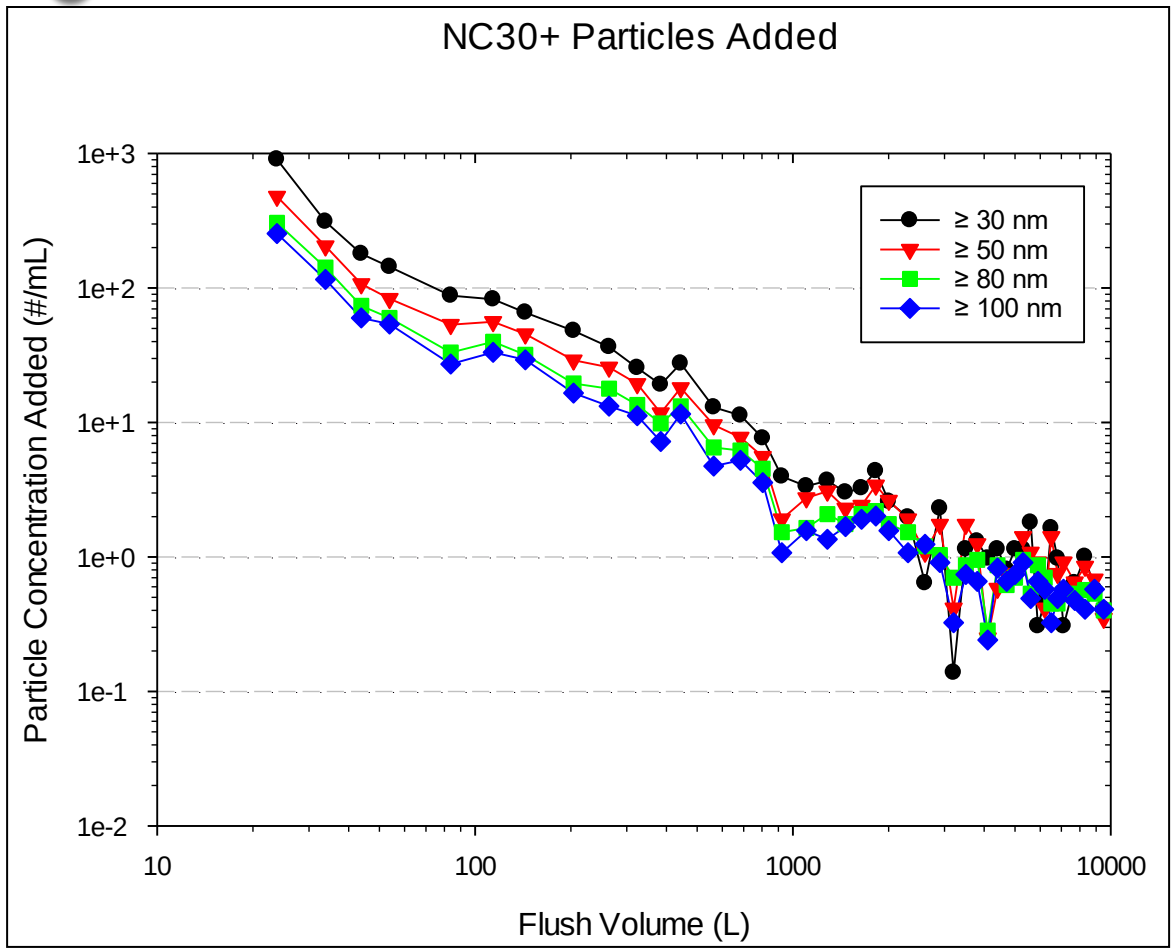


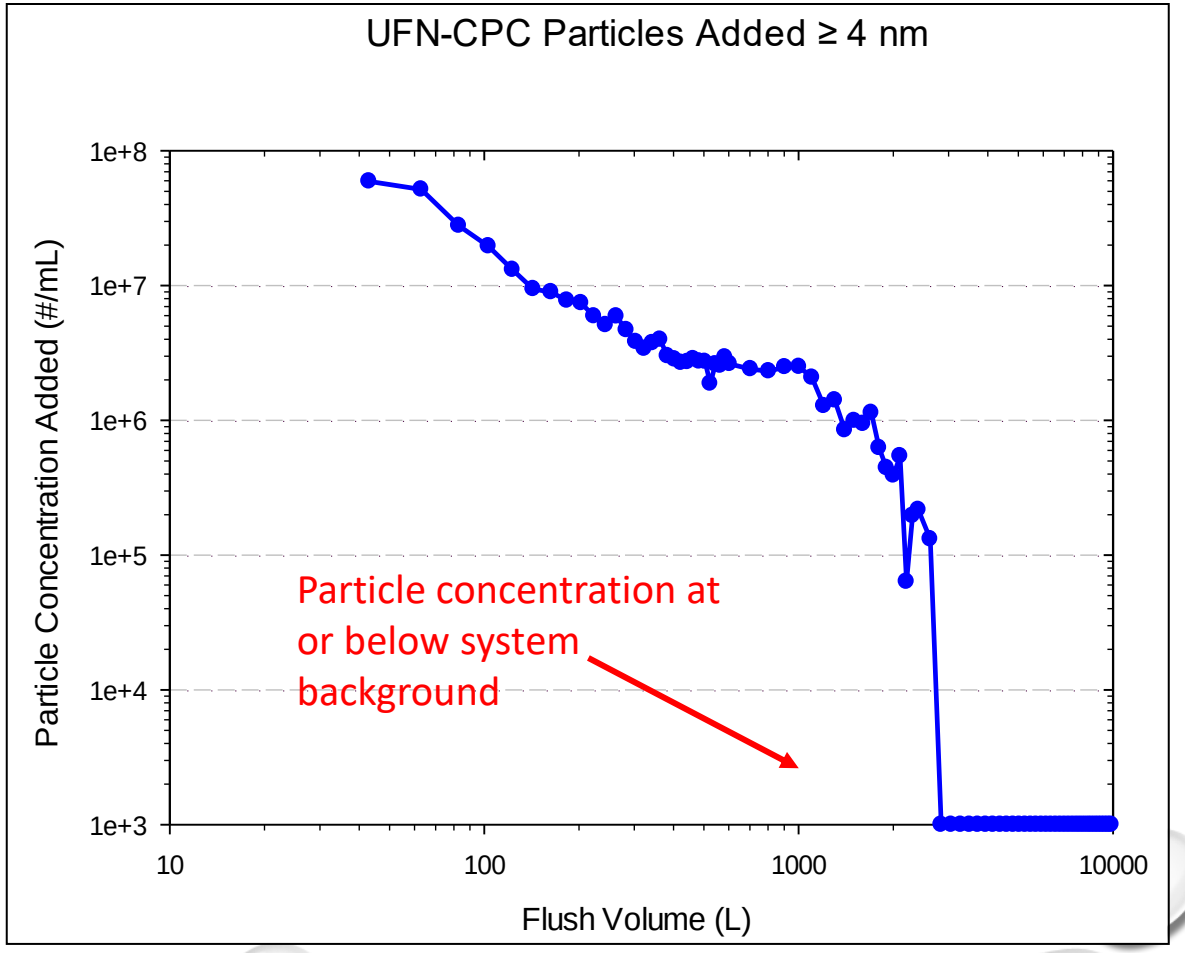


Experimental Plan: Filter Retention Tests

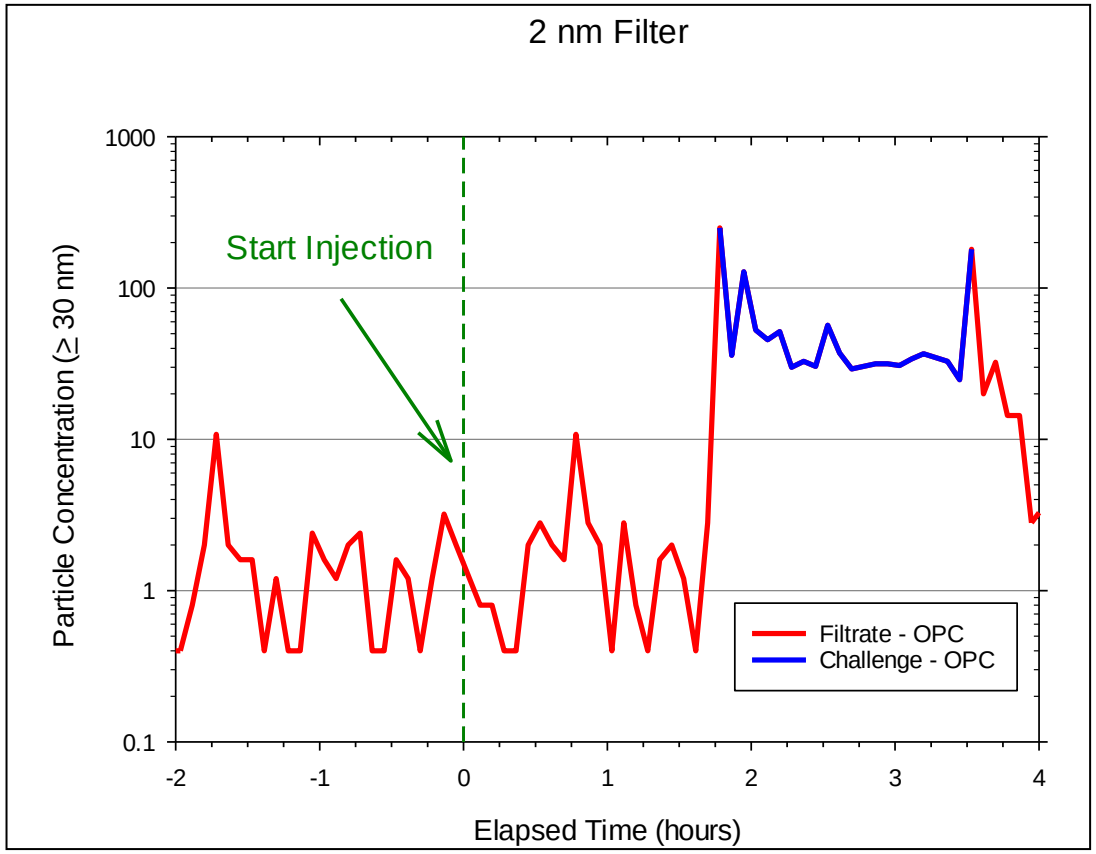
1. Test two 10” cartridge filters:
   - Commercially available 2nm filter
   - Sub-2nm developmental filter
2. Pre-rinse filters to steady-state background.
3. Use face velocity established in SEMI C79 (0.80 cm/min).
4. Target challenge concentration 1.5 e9/mL (> 4 nm).
5. Measure challenge at the end of retention testing.

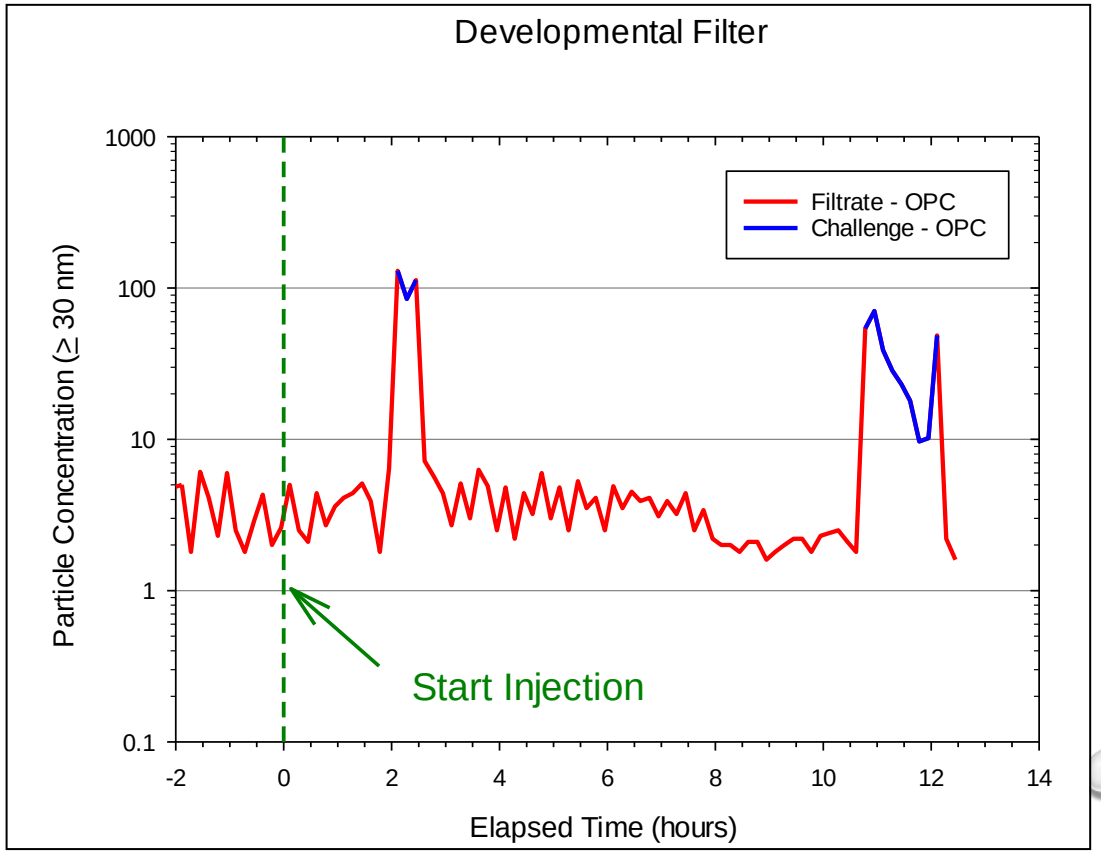
Rinse Monitoring – 2nm Filter





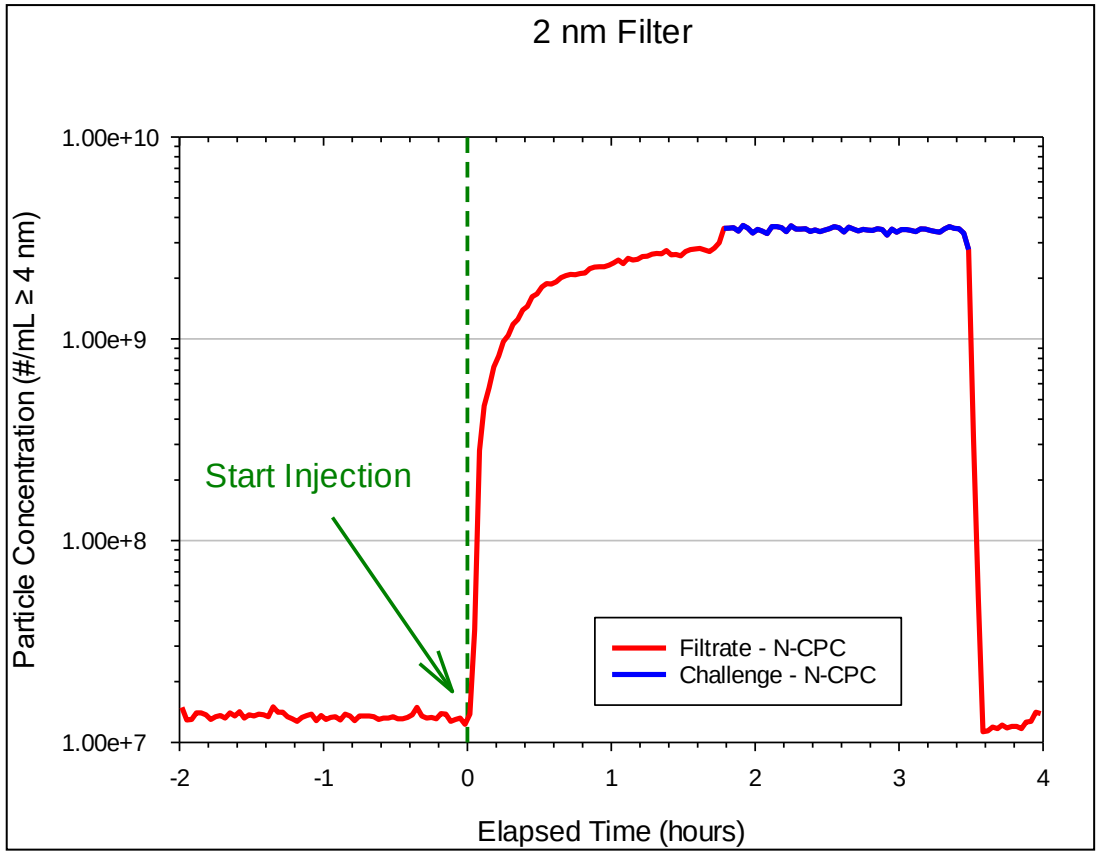
Retention Summary – Lighthouse NC30 OPC

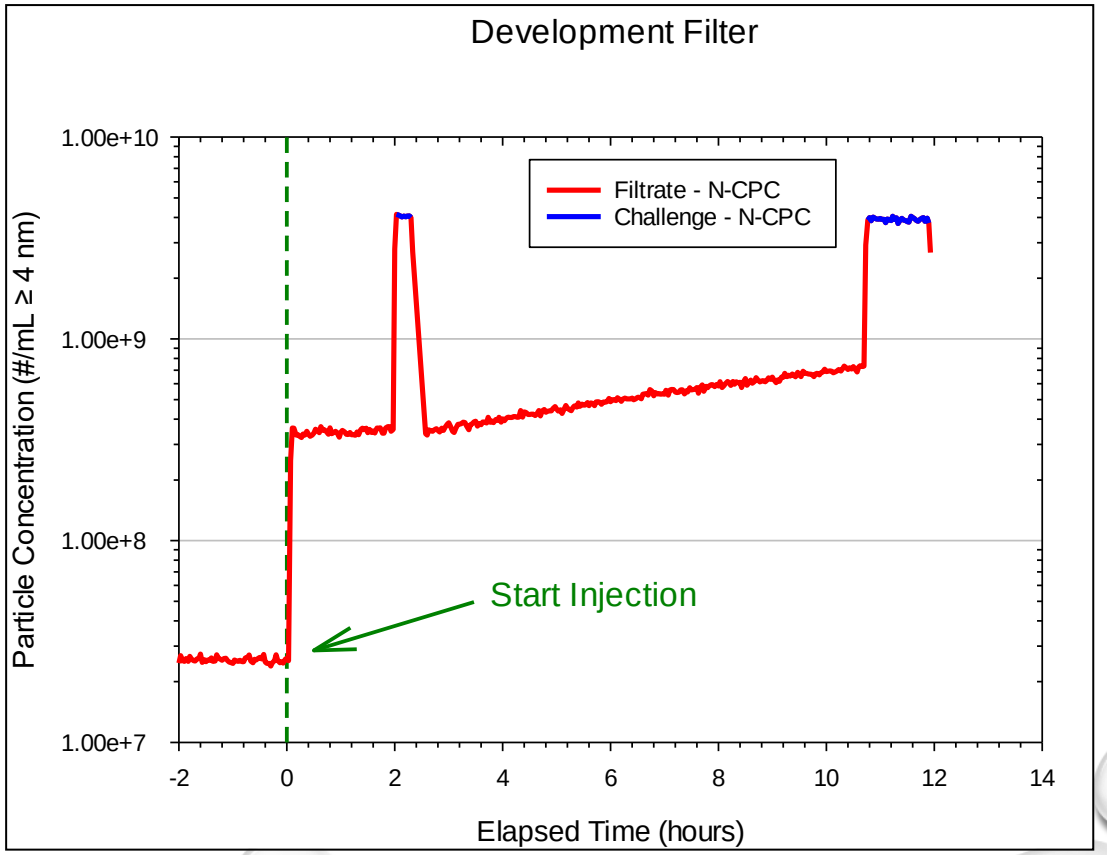




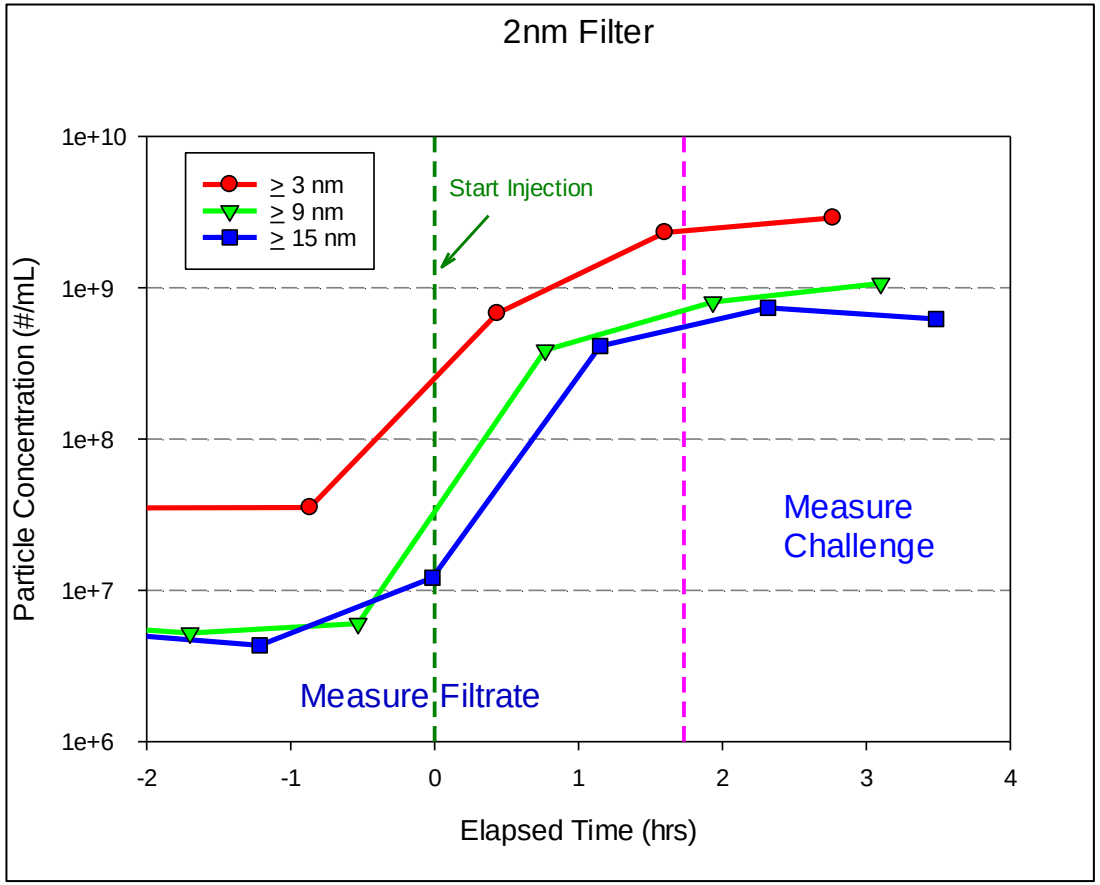
Filter Retention > 99.9% of OPC detected particles ≥ 30 nm for both filters

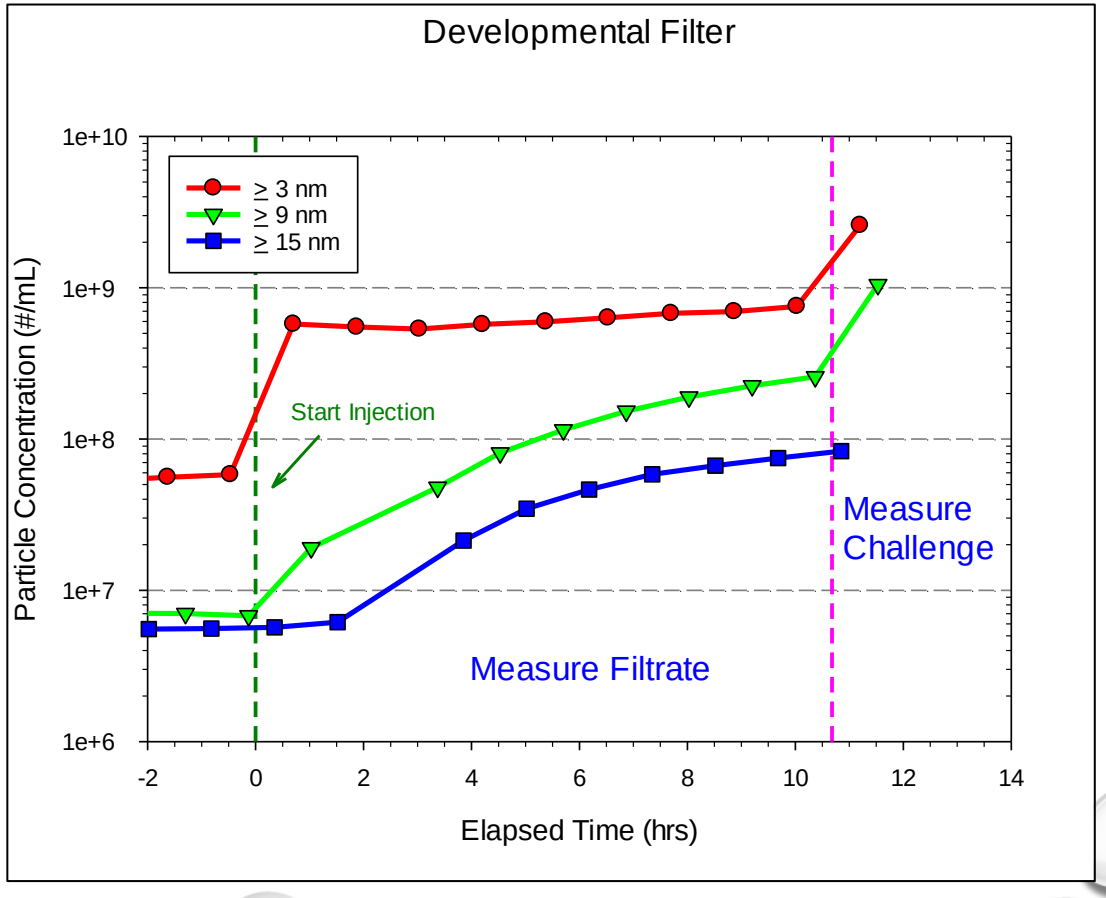
Retention Summary – Ultrafine Nebulizer with 4 nm CPC



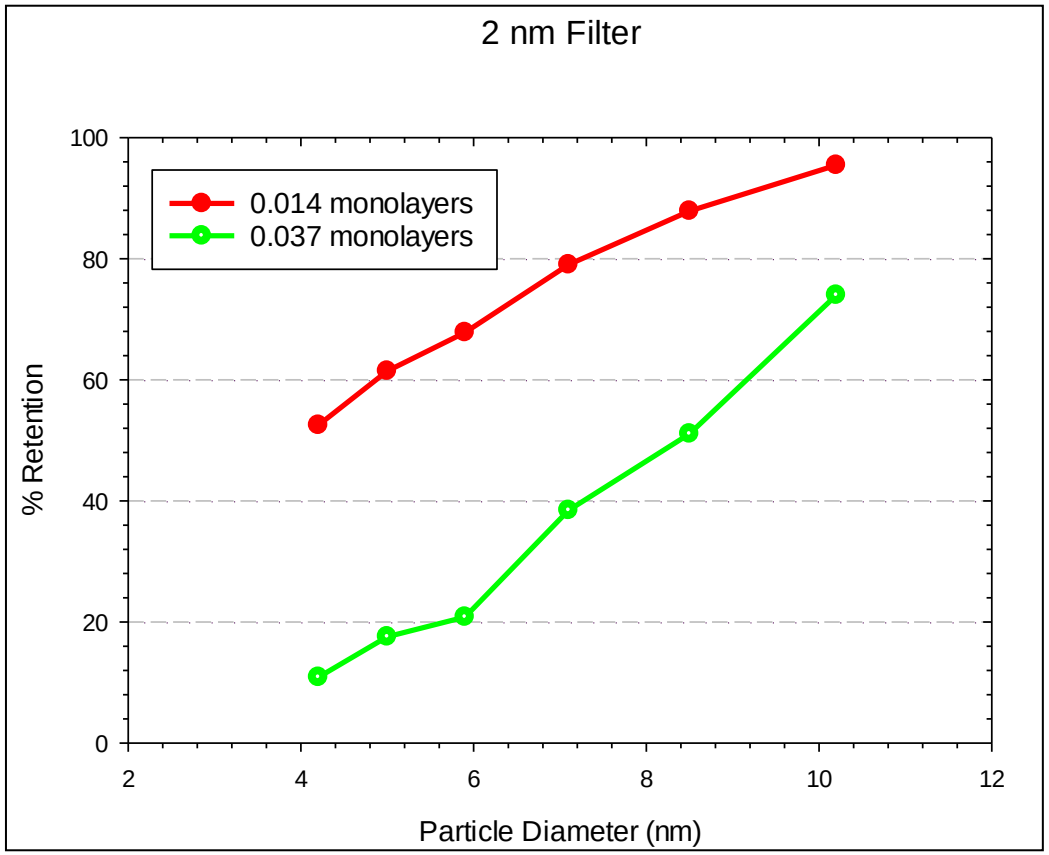


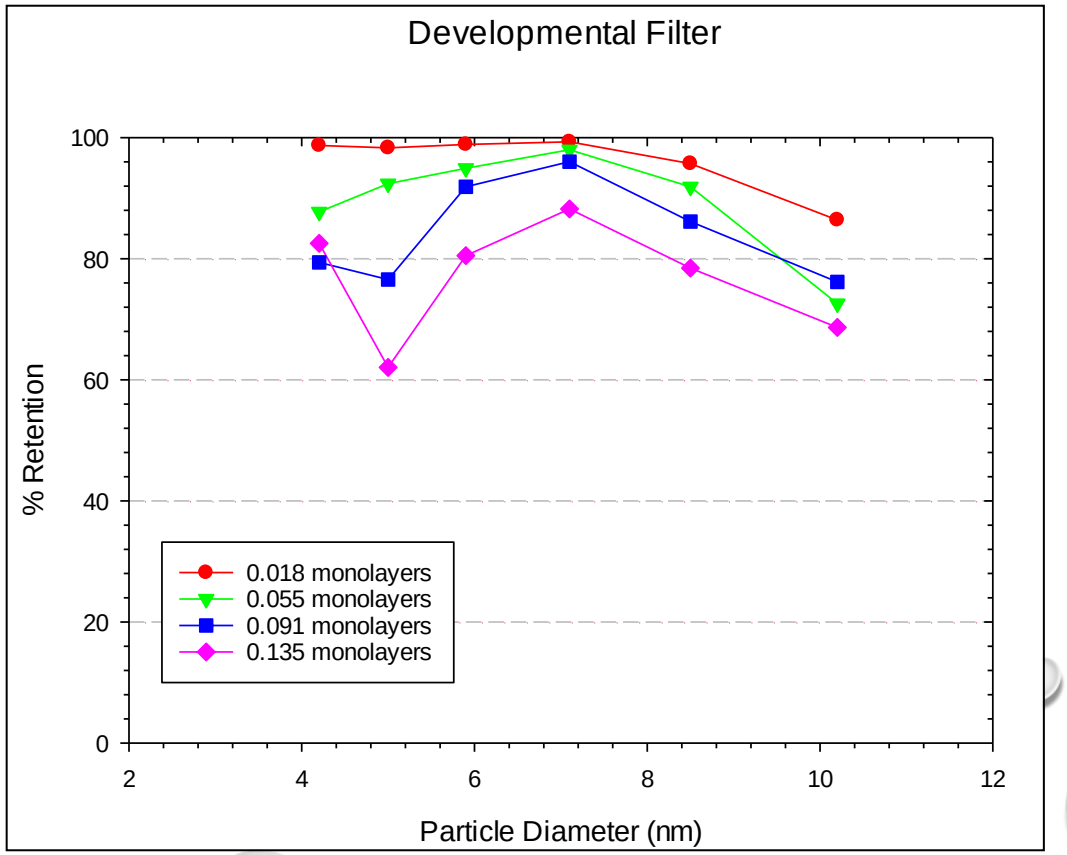
Retention Summary - KFMT STPC3





Filter Retention – Liquid Nanoparticle Sizing





Observations and Recommendations

- HMWO particles and particle precursors as small as 3 nanometers can be measured with high efficiency using ultrafine nebulization in conjunction with aerosol counting instruments (ex. STPC3, LNS, UFN-CPC).
- Use of ultrafine nebulization with aerosol counting appears to be viable candidate for measuring the retention performance of state-of-the-art filters to 3 nanometers.
- A procedure analogous to SEMI C79 using a HMWO challenge appears feasible.
- **The low particle background and high filter retention as measured by liquid optical particle counters did not correlated to the elevated concentration and filter retention of HMWO particle precursors and sub-10 nm particles challenging the "Power Law" assumption.**

Thank you for your time!

Gary Van Schooneveld is President of CT Associates, Inc. Gary has over 30 years of experience with high-purity fluid systems including chemical delivery and ultrapure water and the development and testing of their associated materials and components. He is the author or co-author of more than 45 technical papers and presentations. Gary is an active member of the IRDS UPW and SEMI UPW Task Forces and has been a key contributor in the development and validation of SEMI specification for measuring filter retention below 15 nm (SEMI C79), particle shedding from critical components (SEMI F104) and extractables ion exchange resin rinse performance (SEMI C93). Gary has BS and MS degrees in Materials Engineering from Rensselaer Polytechnic Institute (Troy, NY) and an MBA from the University of Texas (Arlington, TX).

Jikku M Thomas is Staff Engineer and Scientist at CT Associates, Inc., where he has been involved in the application and development of aerosol-based metrology for high-purity liquid systems for over 2 years. He finished undergraduate degrees in Chemical Engineering from IIT Madras, India, and MS and PhD degrees in Chemical Engineering from the University of Minnesota (Twin Cities, MN), where he gained 5 years of research experience focused on sub-20 nm aerosol science. He has authored or co-authored more than 5 technical papers and presentations.

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Product Information

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