Challenges in Metrology & Analysis

for next-generation semiconductor nodes

Advanced Metrology for Particle Sizing and Identification in Ultrapure Liquids

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Creative Technology

CT Associates, Inc.

CONNECT - COLLABORATE - INNOVATE - GROW - PROSPER

Outline

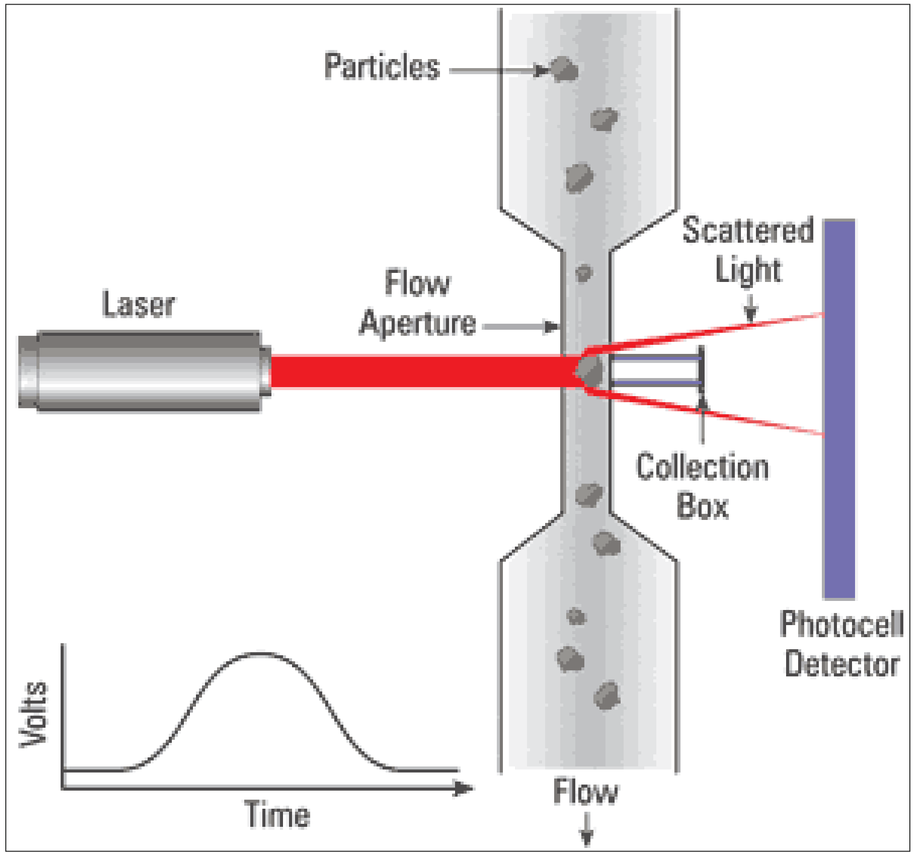
- Optical/Liquid Particle Counting (OPC/LPC)
- Emerging Technologies
  - Liquid to Aerosol Conversion
  - Aerosol Particle Sizing and Counting
  - Focused Aerosol Deposition and Analysis
- Related SEMI Specifications

Optical Particle Counting

The OPC has been the semiconductor industry's particle workhorse for over 40 years.

How does an optical particle counter work?

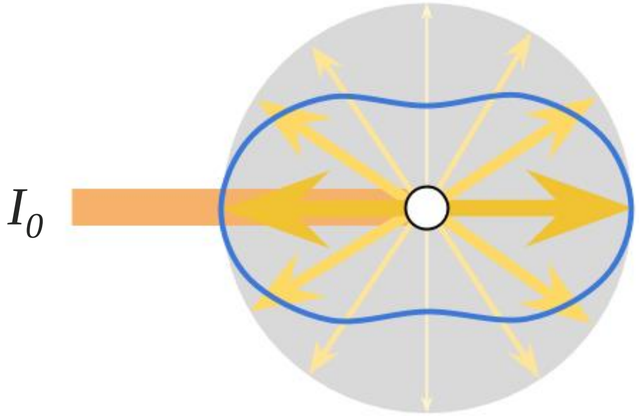
Optical Particle Counting



Courtesy of Noria Communications

1. **An optical counter is a scattered light event and intensity detector.**

Optical Particle Counting

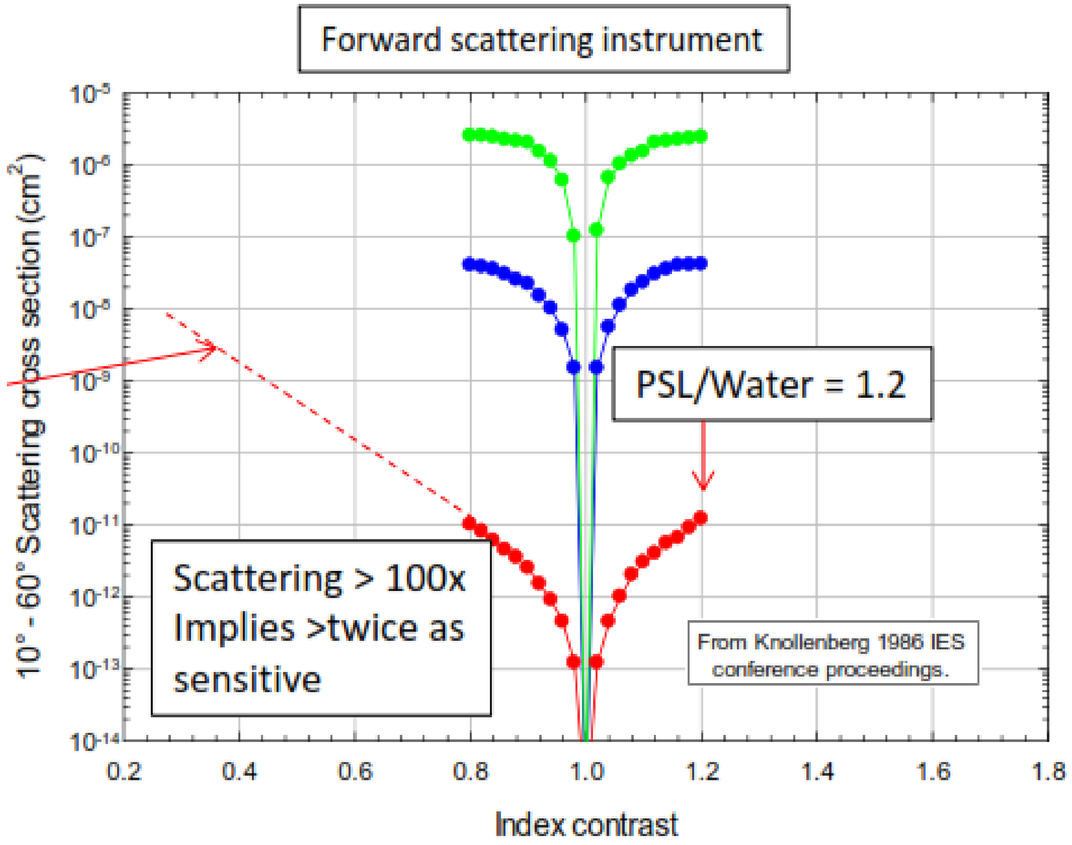


1. An optical counter is a scattered light event and intensity detector.
2. **The intensity of the scattered light is proportional to "size" to 6th power ($I \propto D_p^6$).**

Rayleigh Scattering - Intensity of Light

$$I = I_o \cdot \frac{1 + \cos(\theta)^2}{2 \cdot R^2} \cdot \left(\frac{2 \cdot \pi}{\lambda}\right)^4 \cdot \left(\frac{n^2 - 1}{n^2 + 2}\right)^2 \cdot \left(\frac{d}{2}\right)^6$$

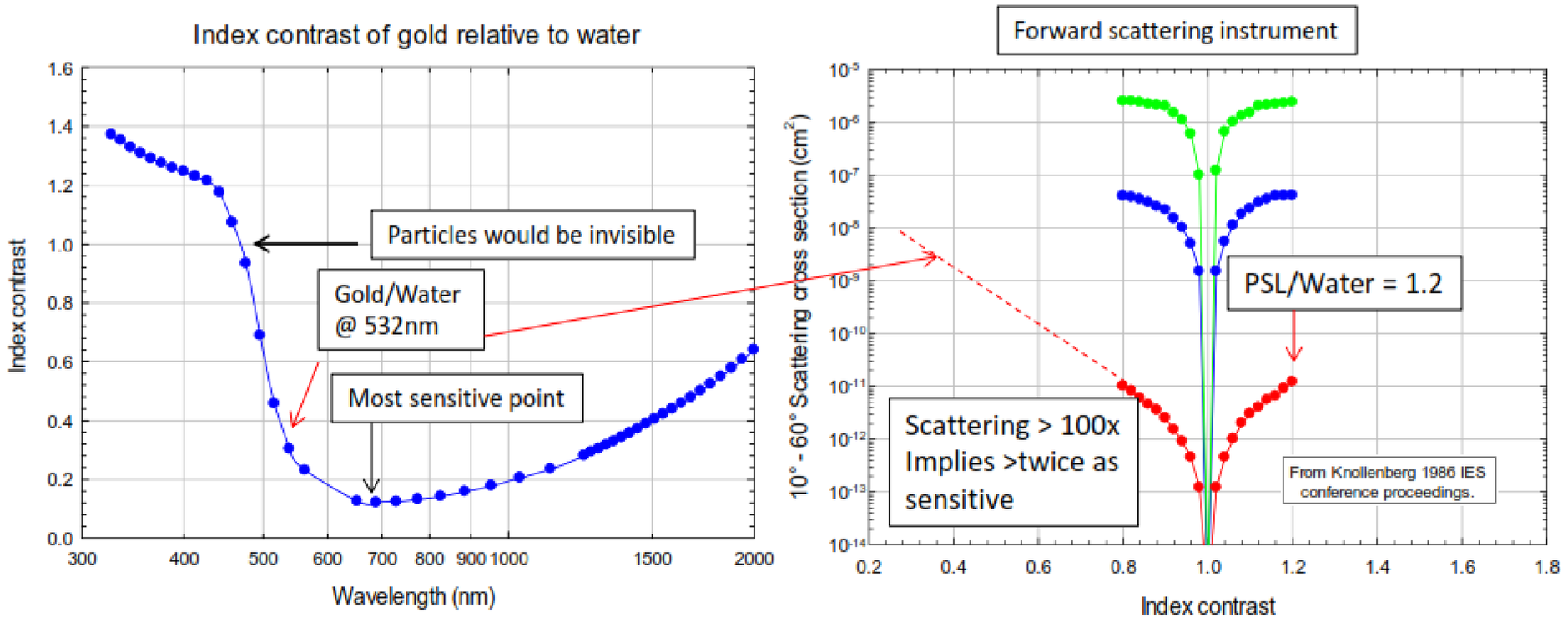
Optical Particle Counting



1. An optical counter is a scattered light event and intensity detector.
2. The intensity of the scattered light is proportional to “size” to 6th power ($I \propto D_p^6$).
3. **The actual size of the particle may be different that the reported size due to material composition and liquid.**

$$\text{Index contrast} = \frac{\text{Particle refractive index}}{\text{Medium refractive index}}$$

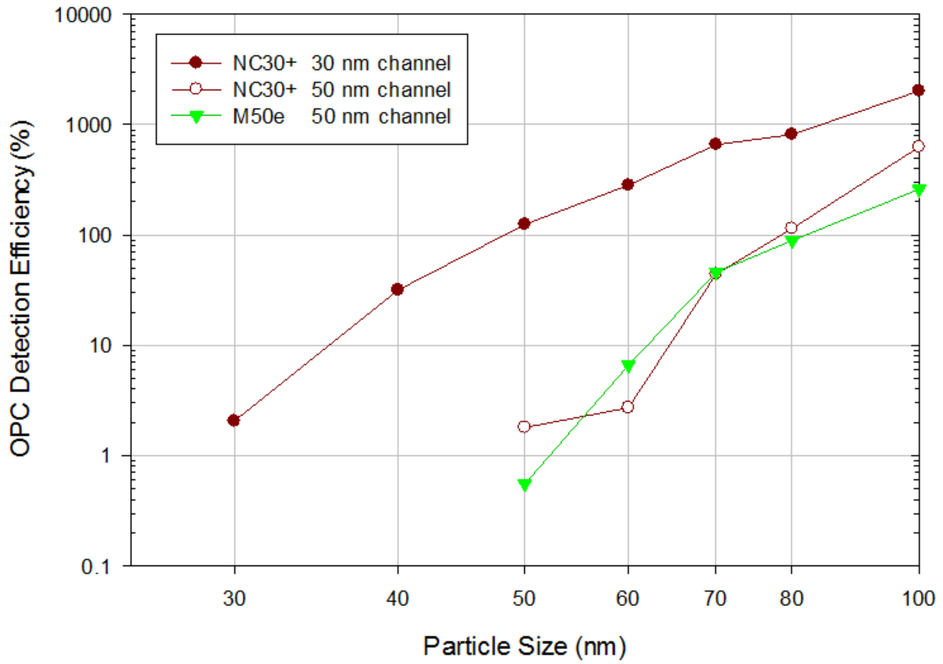
Optical Particle Counting



A 20 nm gold particle in water has roughly the same light-scattering cross-section of a 45 nm PSL particle. However, a 20 nm PTFE particle in water would size closer to 10 nm

Optical Particle Counting

OPC response to mono-dispersed PSL



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

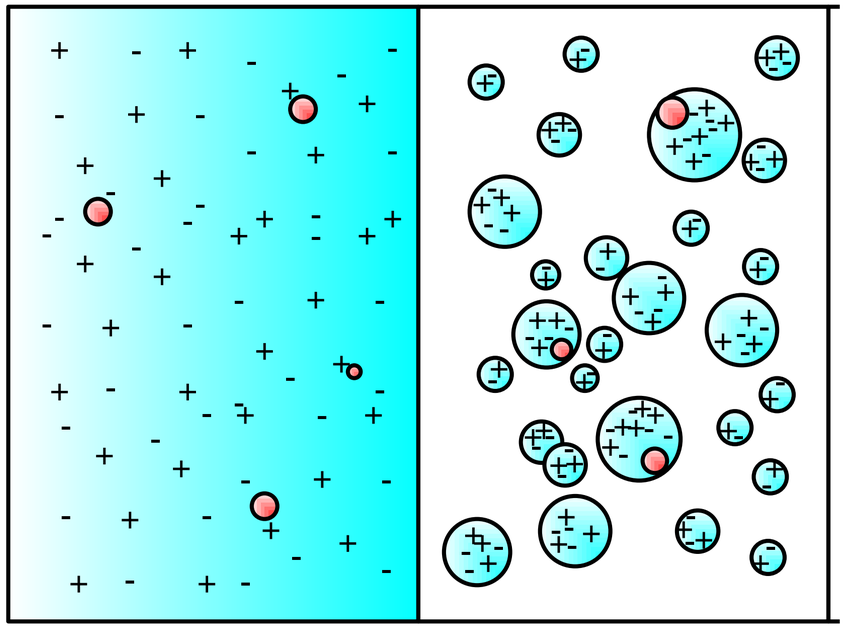
1. An optical counter is a scattered light event and intensity detector.
2. The intensity of the scattered light is proportional to “size” to 6th power ($I \propto D_p^6$).
3. The actual size of the particle may be different that the reported size due to material composition and liquid.
4. **Detection efficiency varies significantly as a function of size.**

Liquid to Aerosol Conversion

Liquid to aerosol conversion was first introduced to the semiconductor industry in 2008 for CMP slurry PSD and UPW particle measurement.

- How does liquid to aerosol conversion work?

Liquid to Aerosol Conversion

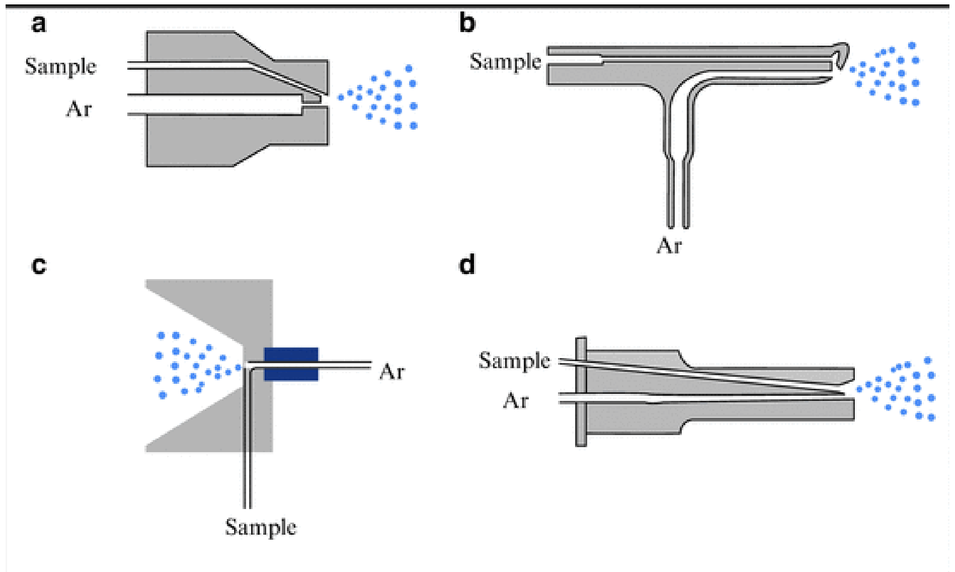


Liquid Sample With NVR

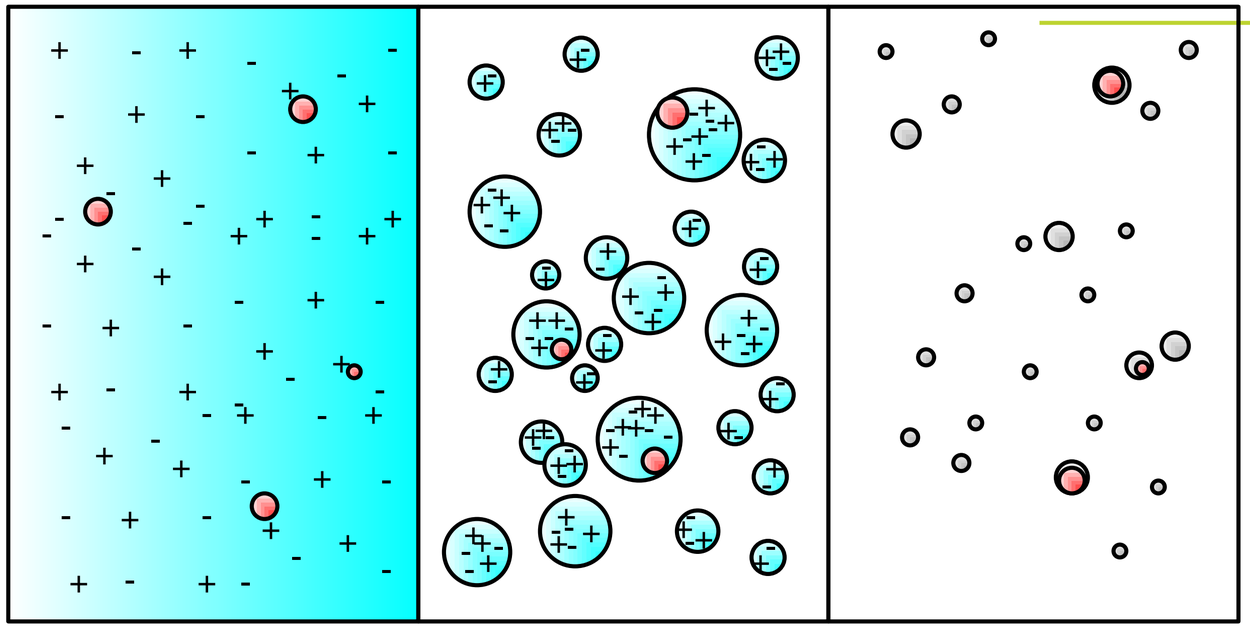
Nebulized Sample

Courtesy of Kanomax FMT

1. **Objective is to convert liquid into small, uniform droplets.**



Liquid to Aerosol Conversion



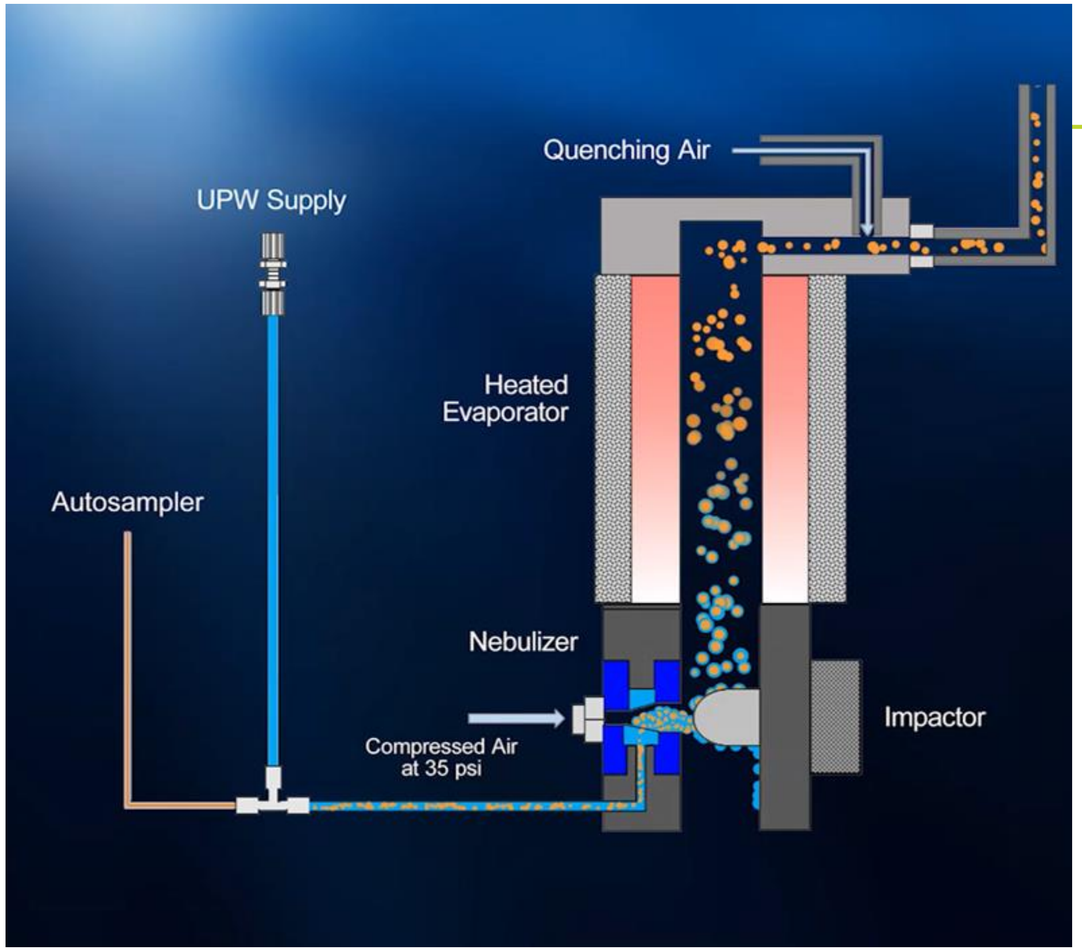
Liquid Sample With NVR | Nebulized Sample | Aerosolized Sample

Courtesy of Kanomax FMT

1. Objective is to convert liquid into small, uniform droplets.
2. **Remove liquid from the droplets leaving "native" and "formed" particles from particle precursors*.**

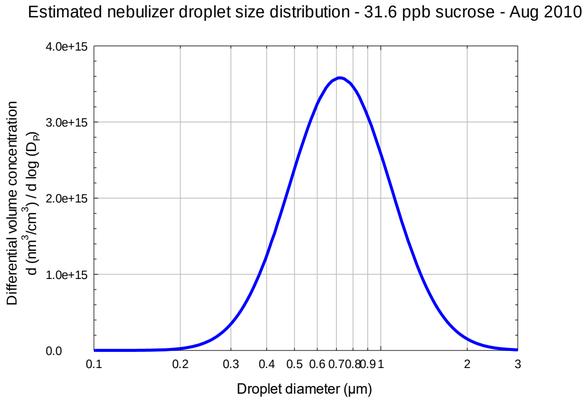
* The risk of particle precursors forming particles of critical dimension during manufacturing is currently being study in the IRDS UPW and Critical Components Task Forces.

Liquid to Aerosol Conversion



Courtesy of Kanomax FMT

1. Objective is to convert liquid into small, uniform droplets.
2. Remove liquid from the droplets leaving “native” and “formed” particle (particle precursors).
3. **Achieving small (<1 μm), uniform (geo SD < 1.2) droplet distribution is key to the success of this technique.**

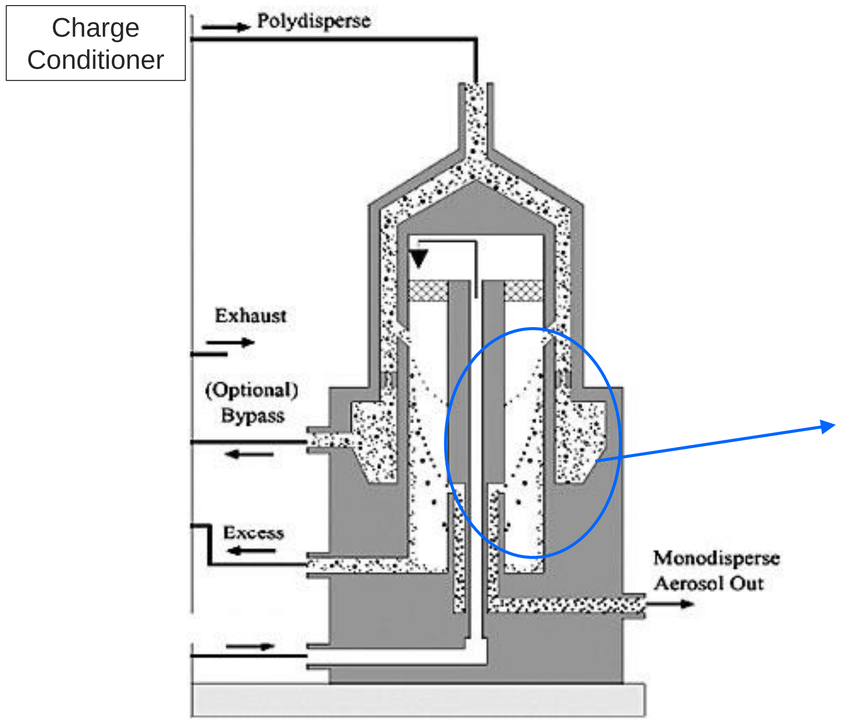


Liquid to Aerosol Particle Sizing and Measurement

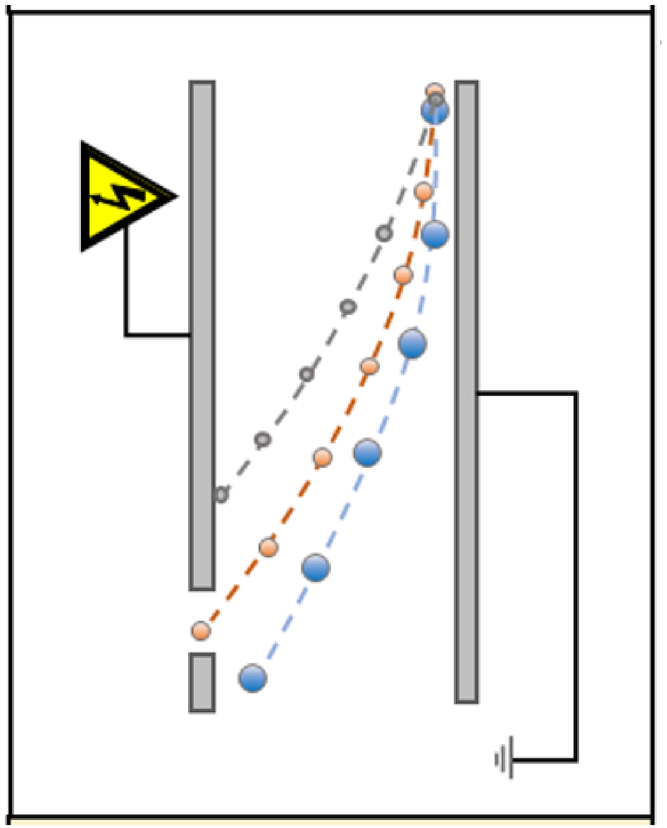
Aerosol classification (sizing) and counting as small as 10 nm was demonstrated in the mid-1970's and commercialize in the early 1980's.

- How does aerosol particle sizing and counting work?

Liquid to Aerosol Particle Sizing and Measurement



Differential Mobility Analyzer



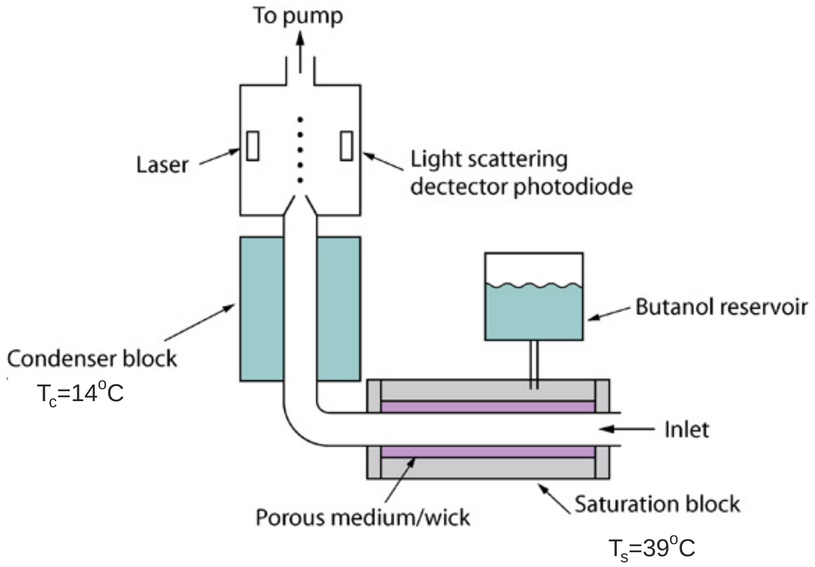
Electrical mobility $\propto D_p$

1. **Electrical mobility classification - sizing**

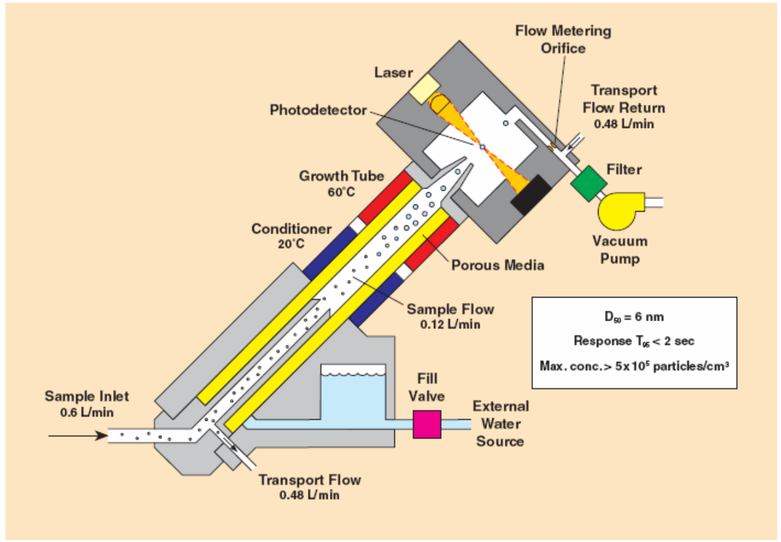
Sizing technique used in Liquid Nanoparticle Sizing (LNS)

Liquid to Aerosol Particle Sizing and Measurement

Detection to < 2 nm available



Butanol Based CPC



Water Based CPC

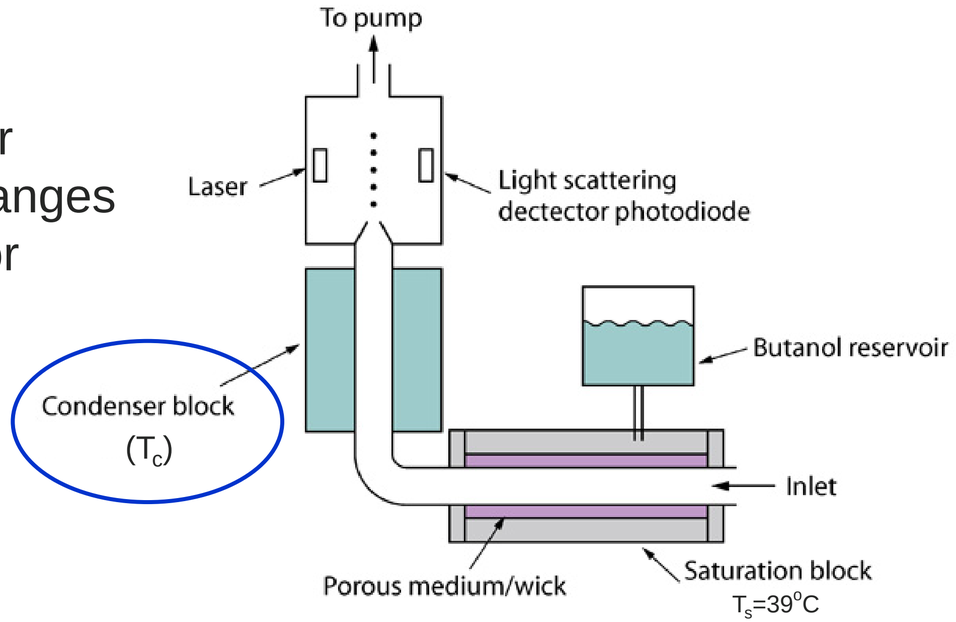
1. Electrical mobility classification – sizing
2. **Condensation particle counting (CPC)**

Liquid condenses on particle and grows large enough for 100% detection efficiency by light scattering.

Liquid to Aerosol Particle Sizing and Measurement

Varying the condenser block temperature changes the sizing threshold for nucleation

T_c(°c)	D_{50}(nm)
10	2.5
14	4
22	10



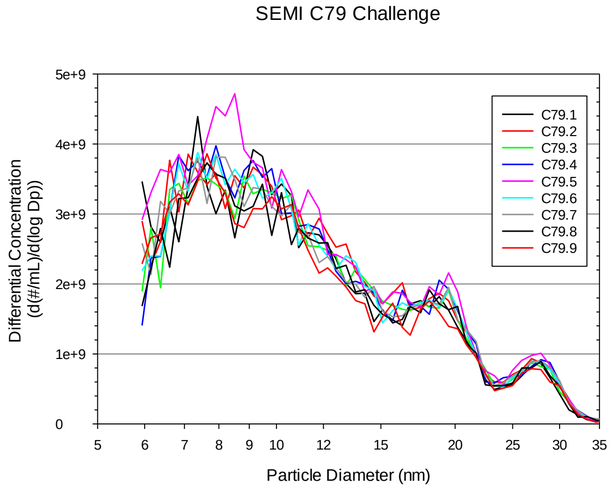
Butanol Based CPC

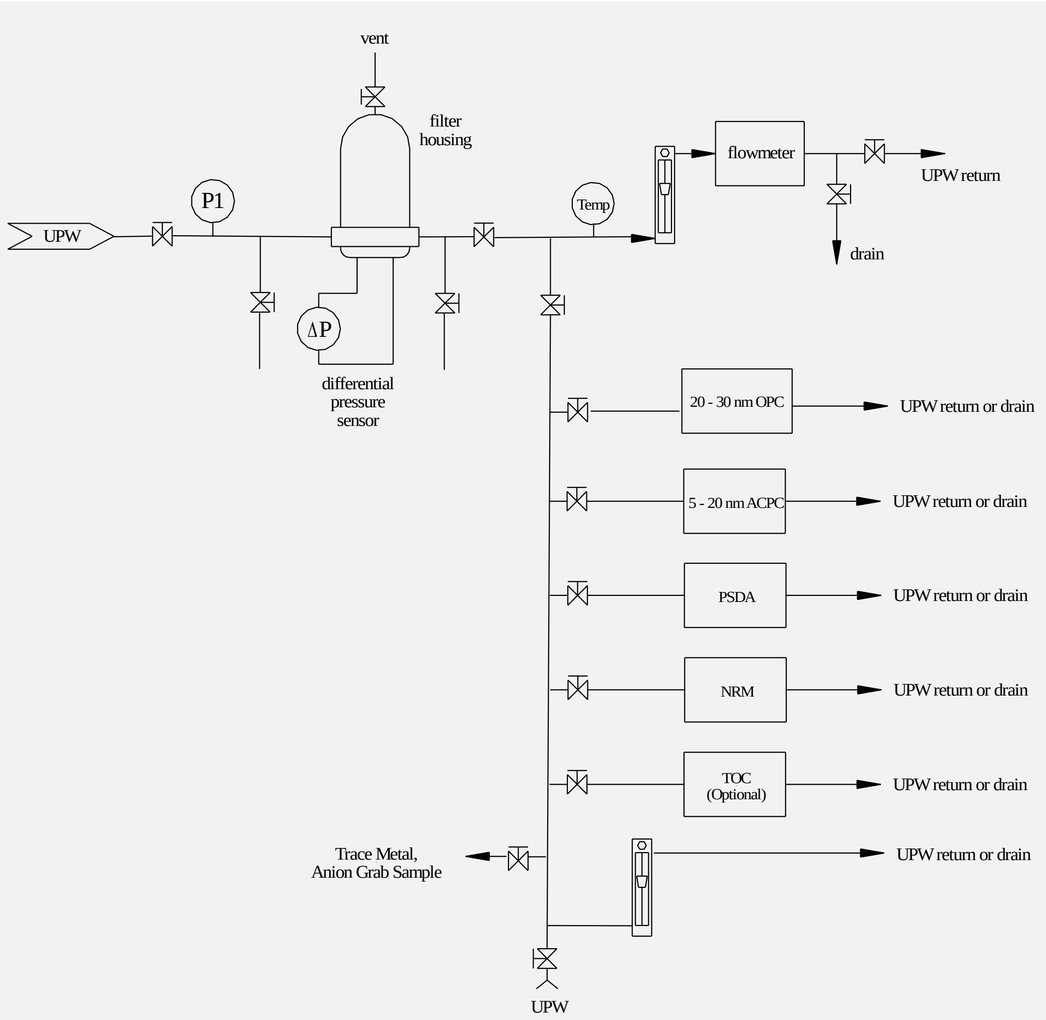
1. Electrical mobility classification – sizing
2. Condensation particle counting (CPC)
3. **Variable D_{50} cutoff CPC's - Sizing**

Sizing technique used in the Scanning Threshold Particle Counter (STPC)

Performance Comparisons – SEMI C79 Filter Testing

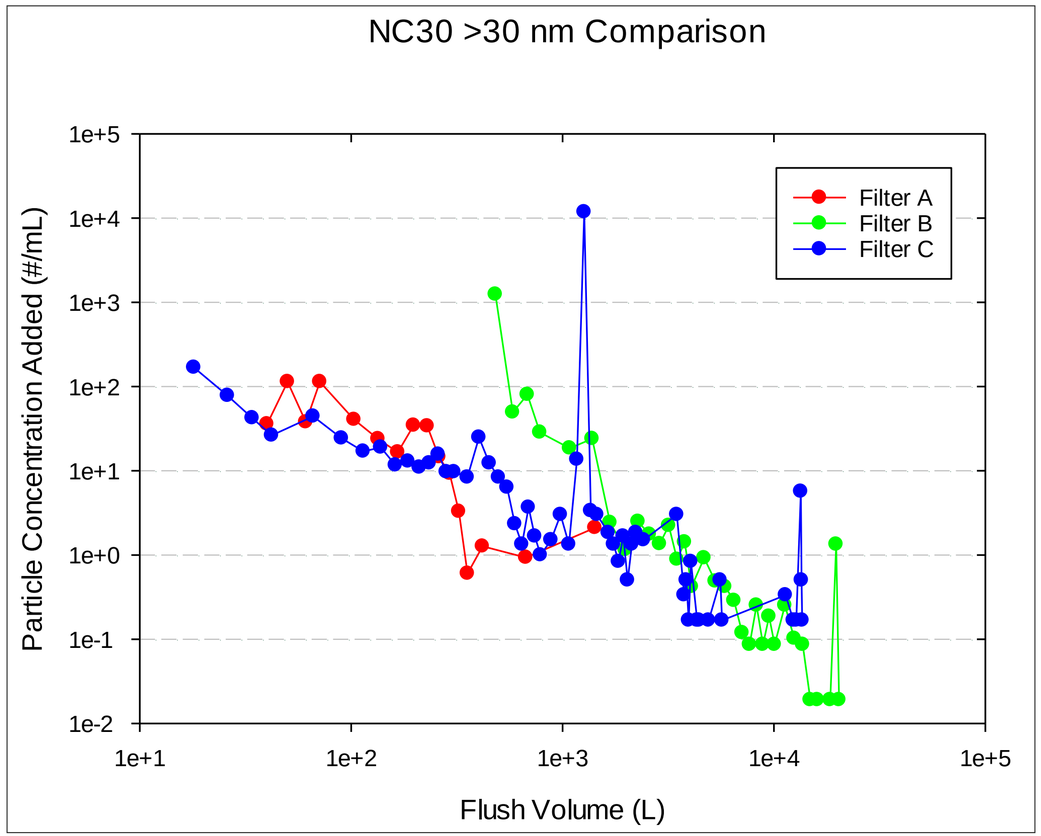
1. 10” POP filter cartridges with similar ratings
2. Three filter manufacturers
3. Test performed in triplicate
4. Rinse testing in UPW (three days)
5. Retention testing with poly-dispersed silica (6 – 35 nm)





Performance Comparisons – SEMI C79 Filter Testing

OPC Data

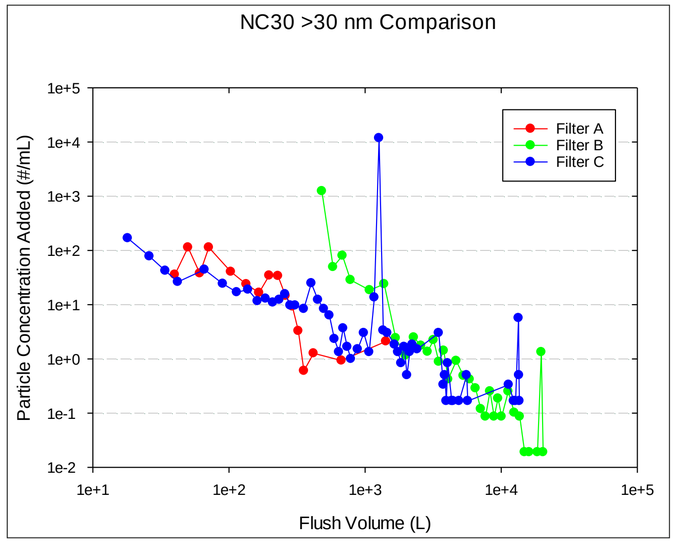


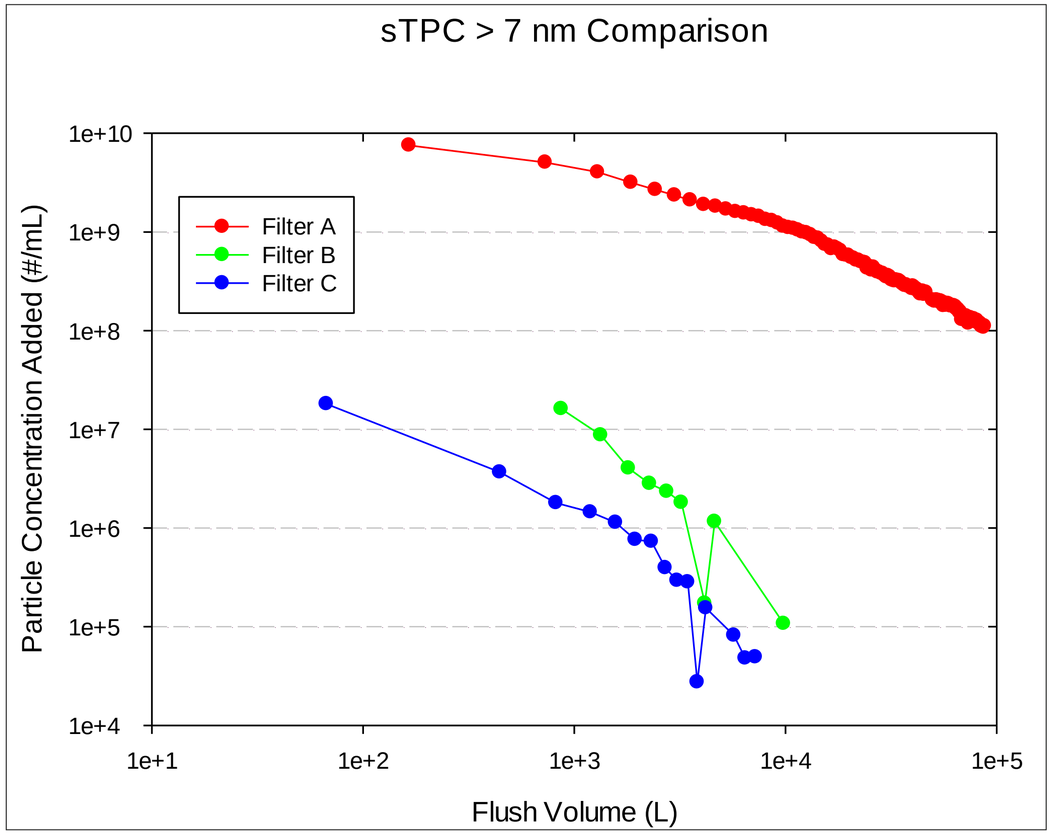
~ 33 hours rinse period

Performance Comparisons – SEMI C79 Filter Testing

Liquid to Aerosol - CPC

OPC Data

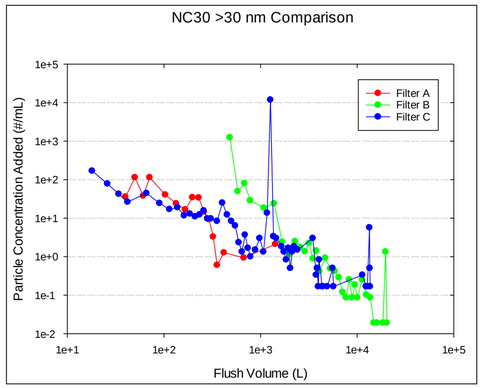


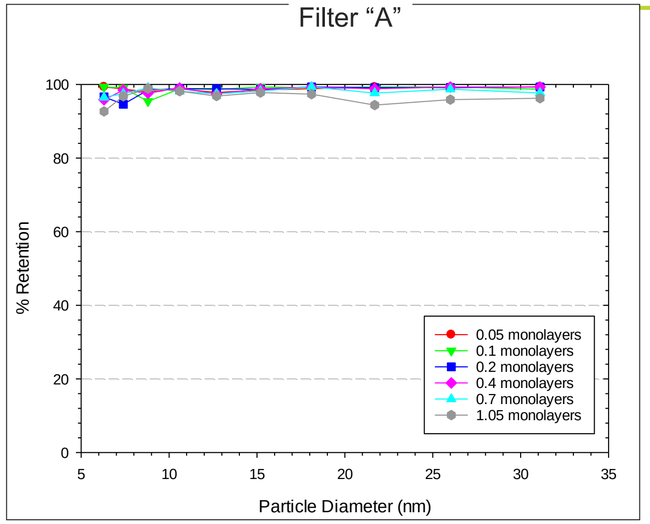


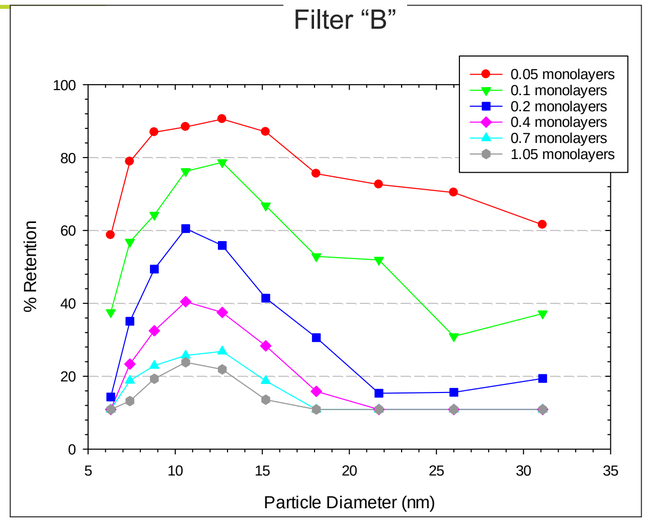
Performance Comparisons – SEMI C79 Filter Testing

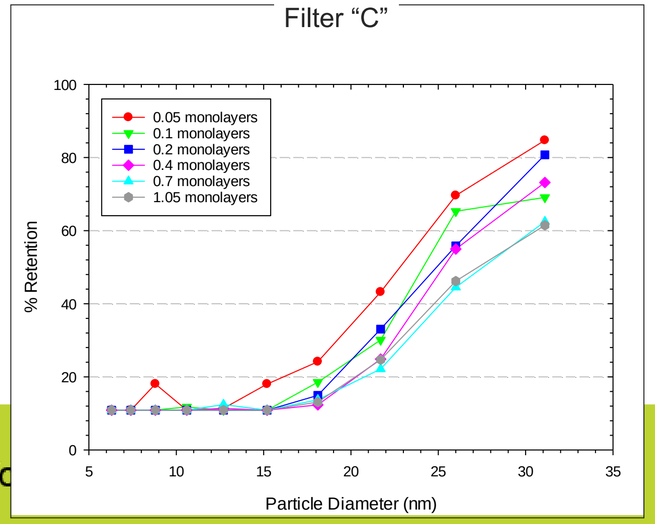
Liquid to Aerosol – EM Classification

OPC Data

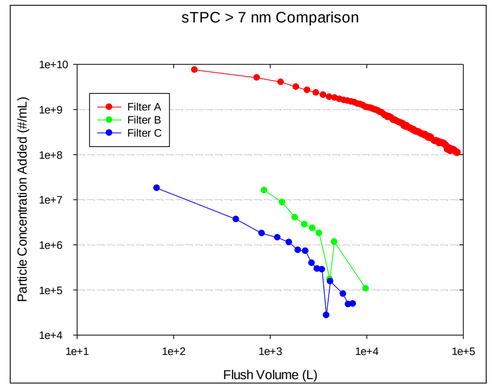








Liquid to Aerosol - CPC



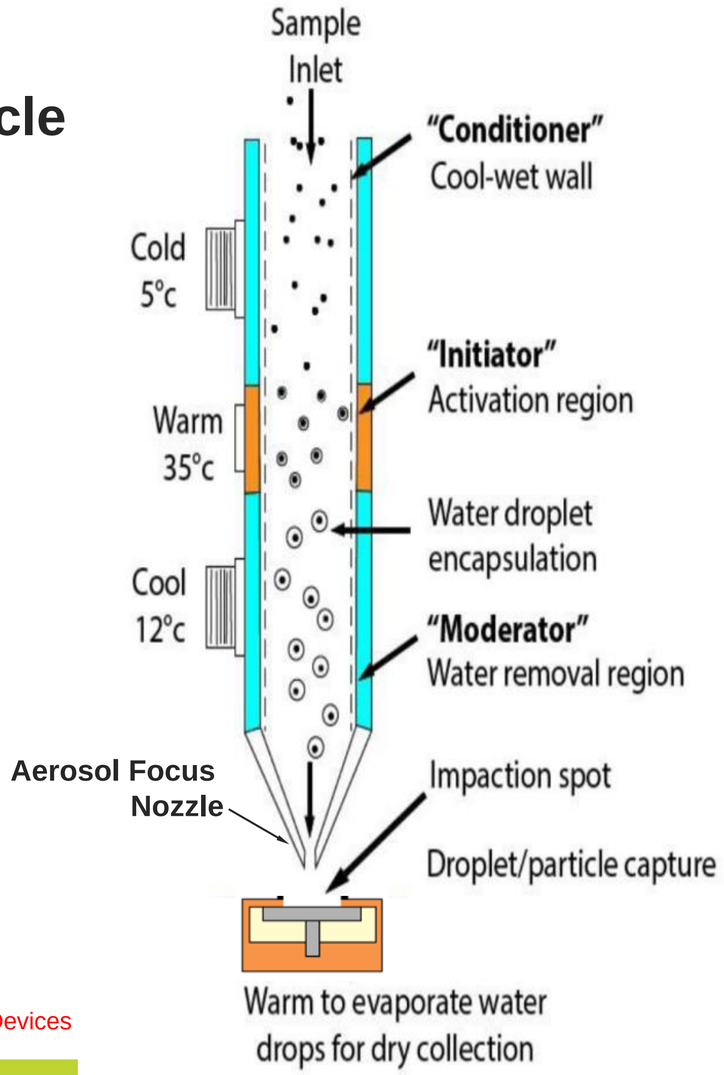
Focused Aerosol Deposition (FAD) and Analysis

Ability to collect, concentrate and deposit extracted nanoparticles as small as 5 nanometers was first demonstrated in 2017.

- How does FAD work?

Focused Aerosol Deposition (FAD) and Analysis

Nanoparticle Collector



Supersaturation levels of 120-140% activate condensation growth on particles as small as 3 nm.

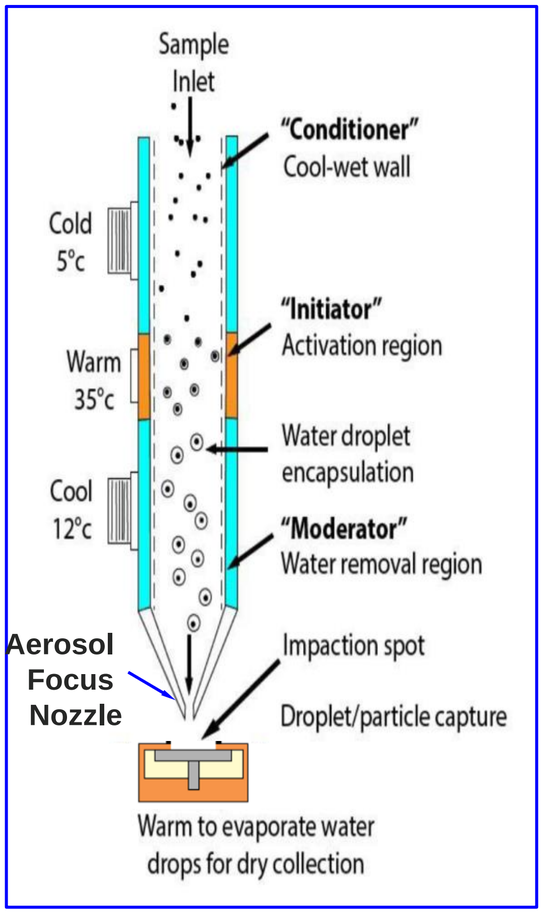
Commercial liquid filters used for particle collection are limited to 20 nm and greater

Droplets grown to nominal 3µm diameter are easily captured by bounce-free, soft inertial impaction.

Courtesy of Aerosol Devices

• Focused Aerosol Deposition (FAD) and Analysis

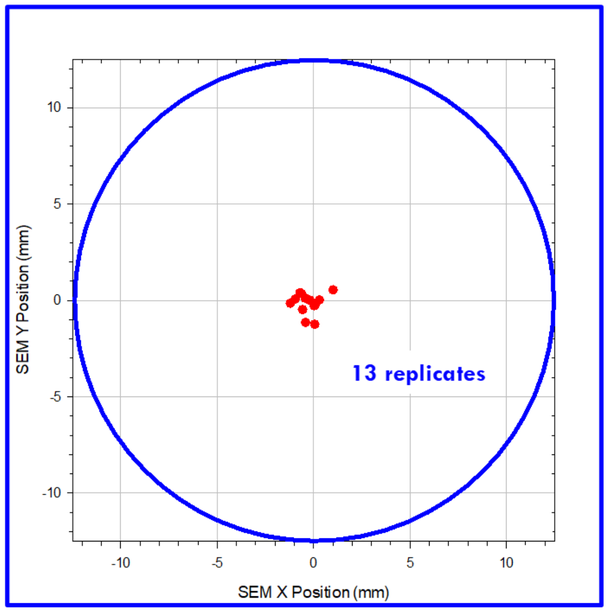
Nanoparticle Collector



Collection Media:

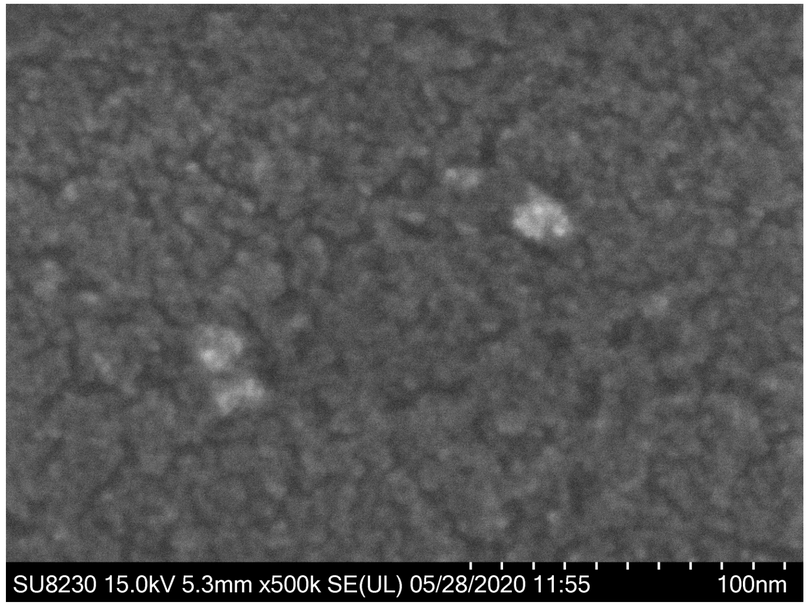
- EPI Silica Wafer
- EPI Germanium Wafer
- SERS Ready Wafer
- TOF-SIMS Ready Wafer

Collection Focus Repeatability

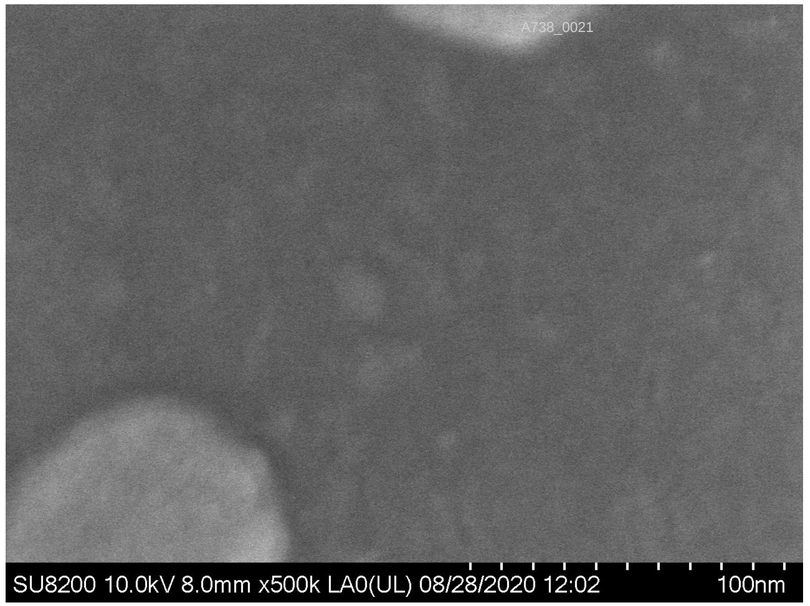


FAD Collections Examples – SEM Analysis

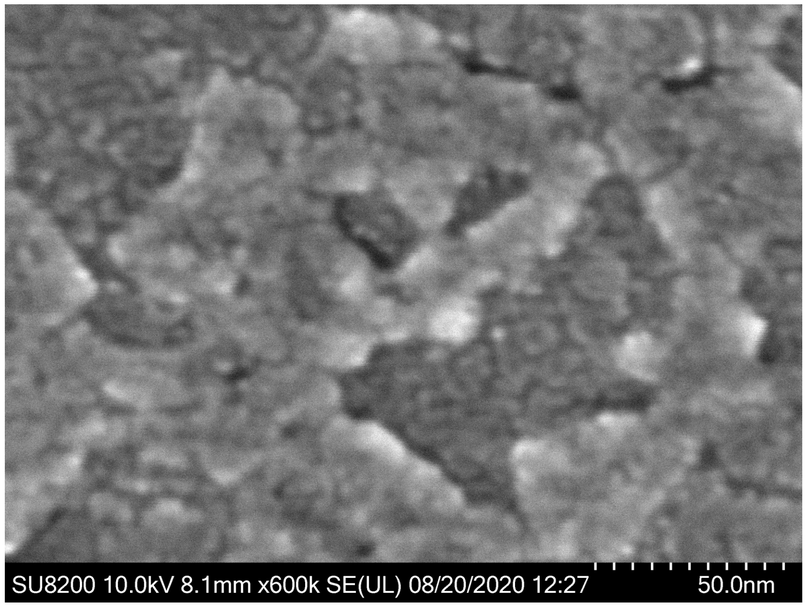
Ion Exchange Resin Effluent



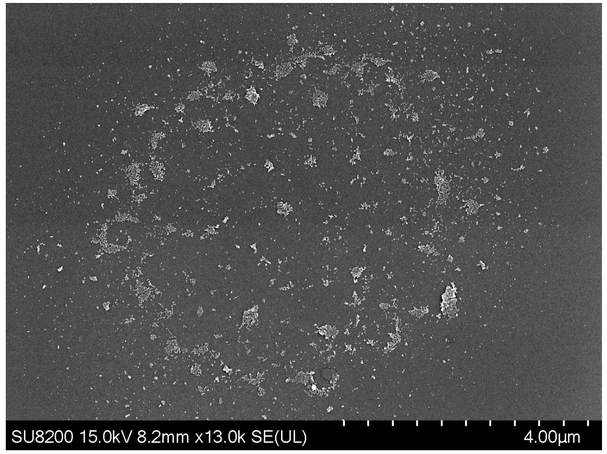
Hot UPW Fluoropolymer Piping Extract

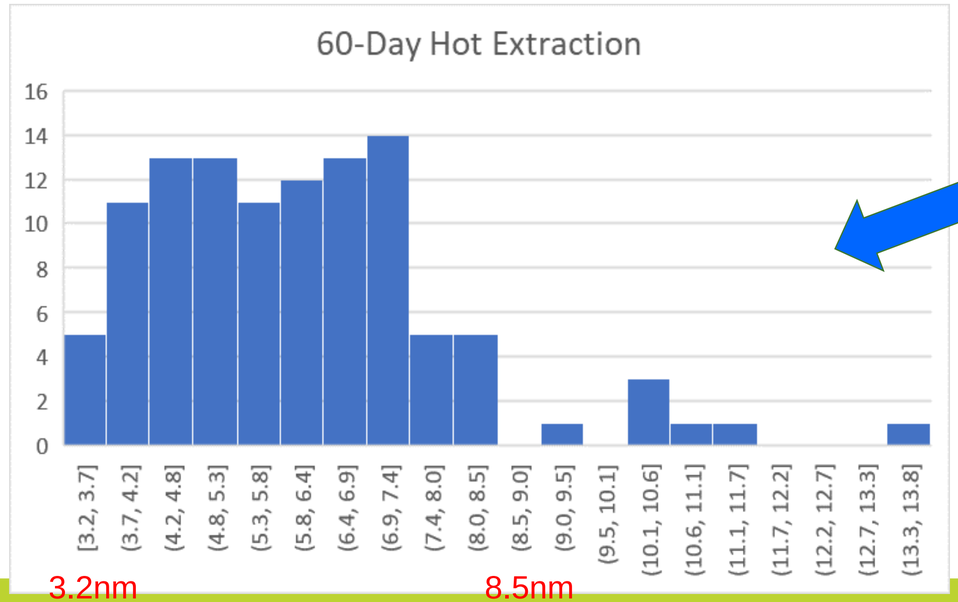


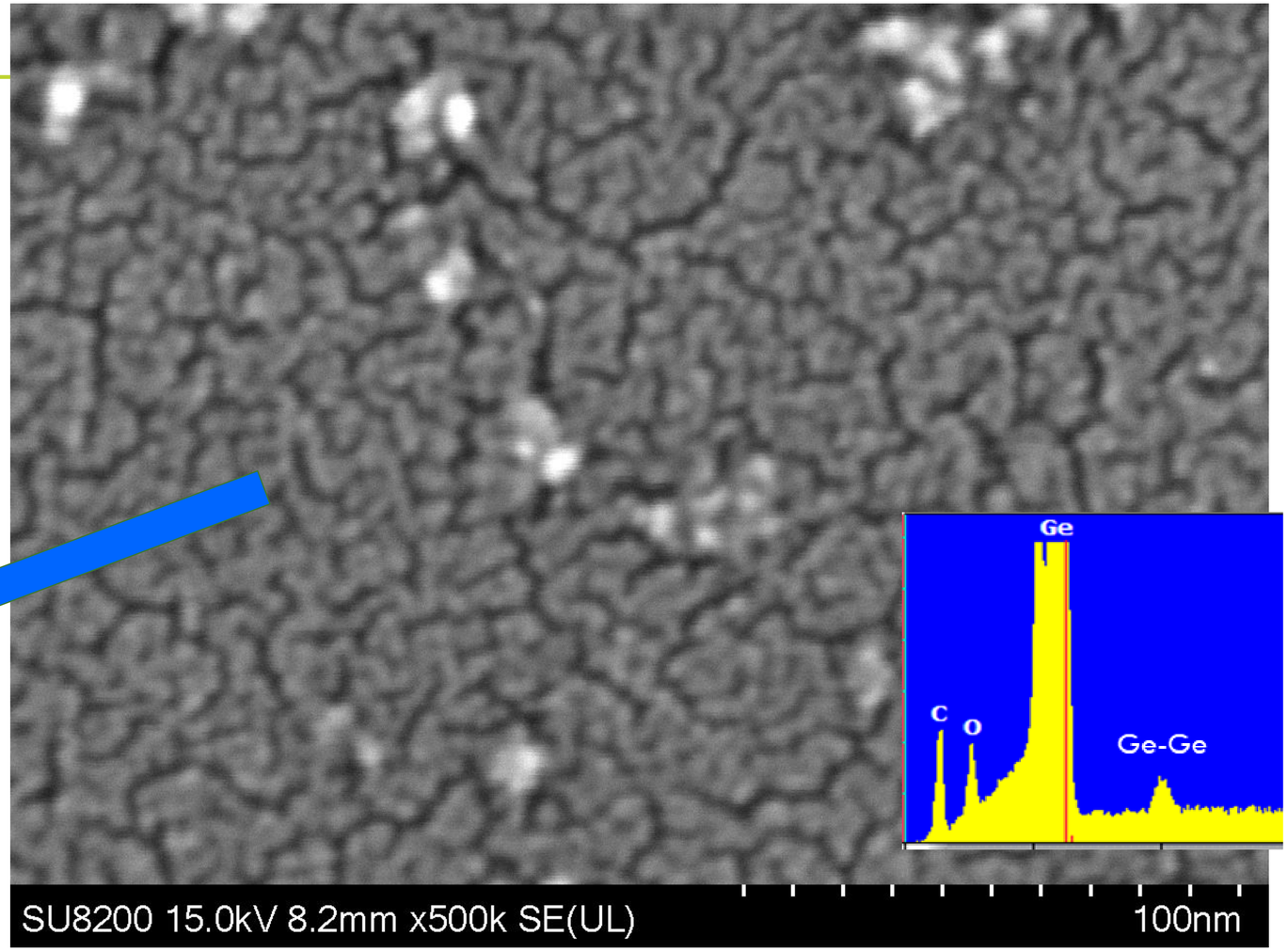
IPA



FAD Collection Example – SEM/EDX/PSD Analysis







Key Takeaways

- Liquid optical particle counting appears to be reaching its practical and economic limit (15 to 20 nm).
- Liquid to aerosol conversion and sizing techniques have demonstrated capabilities to measure particles as small as 2 nm with high detection efficiency.
- Focused aerosol deposition in conjunction with surface analysis techniques provides a tool for determining the composition and source of nano-contamination at sizes previously not possible.

Related Subject Matter- SEMI Specifications

- SEMI C77 - Test Method for Determining the Counting Efficiency of Liquid-Borne Particle Counters for Which the Minimum Detectable Particle Size is Between 30 nm and 100 nm
- SEMI C79 - Guide to Evaluate the Efficacy of Sub-15 nm Filters Used in Ultrapure Water (UPW) Distribution Systems
- SEMI C82 - Test Method for Particle Removal Performance of Liquid Filter Rated 20 to 50 nm With Liquid-Borne Particle Counter
- SEMI C93 - Guide for Determining the Quality of Ion Exchange Resin Used in Polish Applications of Ultrapure Water System
- SEMI C98 - Guide for Chemical Mechanical Planarization (CMP) Particle Size Distribution (PSD) Measurement and Reporting Used in Semiconductor Manufacturing
- SEMI F75 - Guide for Quality Monitoring of Ultrapure Water Used in Semiconductor Manufacturing
- SEMI F104 - Test Method for Evaluation of Particle Contribution of Components Used in Ultrapure Water and Liquid Chemical Distribution Systems
- SEMI F110 - Test Method for Mono-Dispersed Polystyrene Latex (PSL) Challenge of Liquid Filters