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Metrology for Detection of Particle Precursor and Sub-10 nm Particles In Liquids and On-Wafer

CT Associates, Inc.

Gary Van Schooneveld - CT Associates, Inc

Dr. Derek Oberreit - Kanomax Holdings

Dr. Marie Tripp – UNISERS AG

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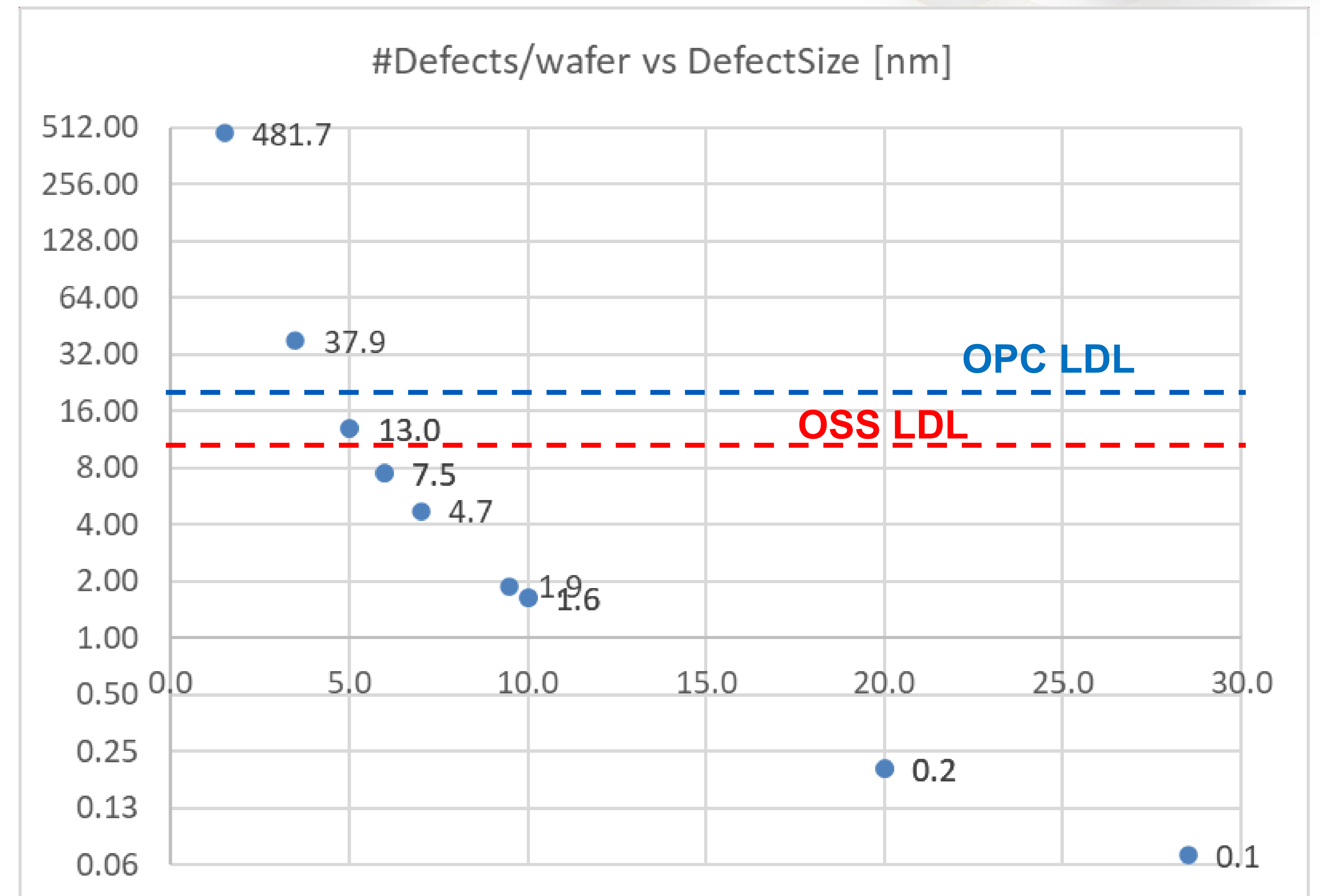
Problem Statement – Device design continues to challenge particle metrology capabilities

Year 2025 MM edition	Defect Size (nm)	Pitch (nm)	Dx/wafer	Defect Mechanism	Air	Process Type
Front-End+Mz+RDL	28.5	114	0.1	Patterning, Gate stack		Wet,Dry
Gate-(XY-direction)	1.5	45	481.7	Patterning, Gate stack	Yes	Wet,Dry
Gate-(Z-direction)	7.0	45	4.7	Patterning, Gate stack	Yes	Wet,Dry
Nanosheet	3.5	26	37.9	Gate stack, EPI		Wet,Dry
Contact (VD - Via for drain)	9.5	45	1.9	Patterning, Metal		Dry
Via0	5.0	28	13.0	Clean		Dry
Metal0	5.0	20	13.0	Patterning, Metal		Dry
Viax	6.0	34	7.5	Clean		Dry
Metalx	6.0	24	7.5	Patterning, Metal		Dry
Viaxt	10.0	57	1.6	Clean		Dry
Metalxt	10.0	40	1.6	Patterning, Metal		Dry
Viay	20.0	113	0.2	Clean		Dry
Metaly	20.0	80	0.2	Patterning, Metal		Dry

More Moore models are pushing critical particle size to less than 2 nm

Source – IRDS 2024 Yield White Paper



OPC – Optical Particle Counting
OSS – Optical Surface Scan

Presentation Outline

- Measurement of sub-10 particles in high-purity liquids by nebulization and condensation particle counting – Derek Oberreit
- Measurement of on-wafer sub-10 nm particle on wafer by surface enhanced particle sizing – Marie Tripp
- IRDS Collaborative Research Overview and Relevant SEMI Standards – Gary Van Schooneveld

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Liquid Based Particle Counting by Nebulization and Aerosol Measurement

Derek Oberreit, Chief Technology Officer

Kanomax Holdings

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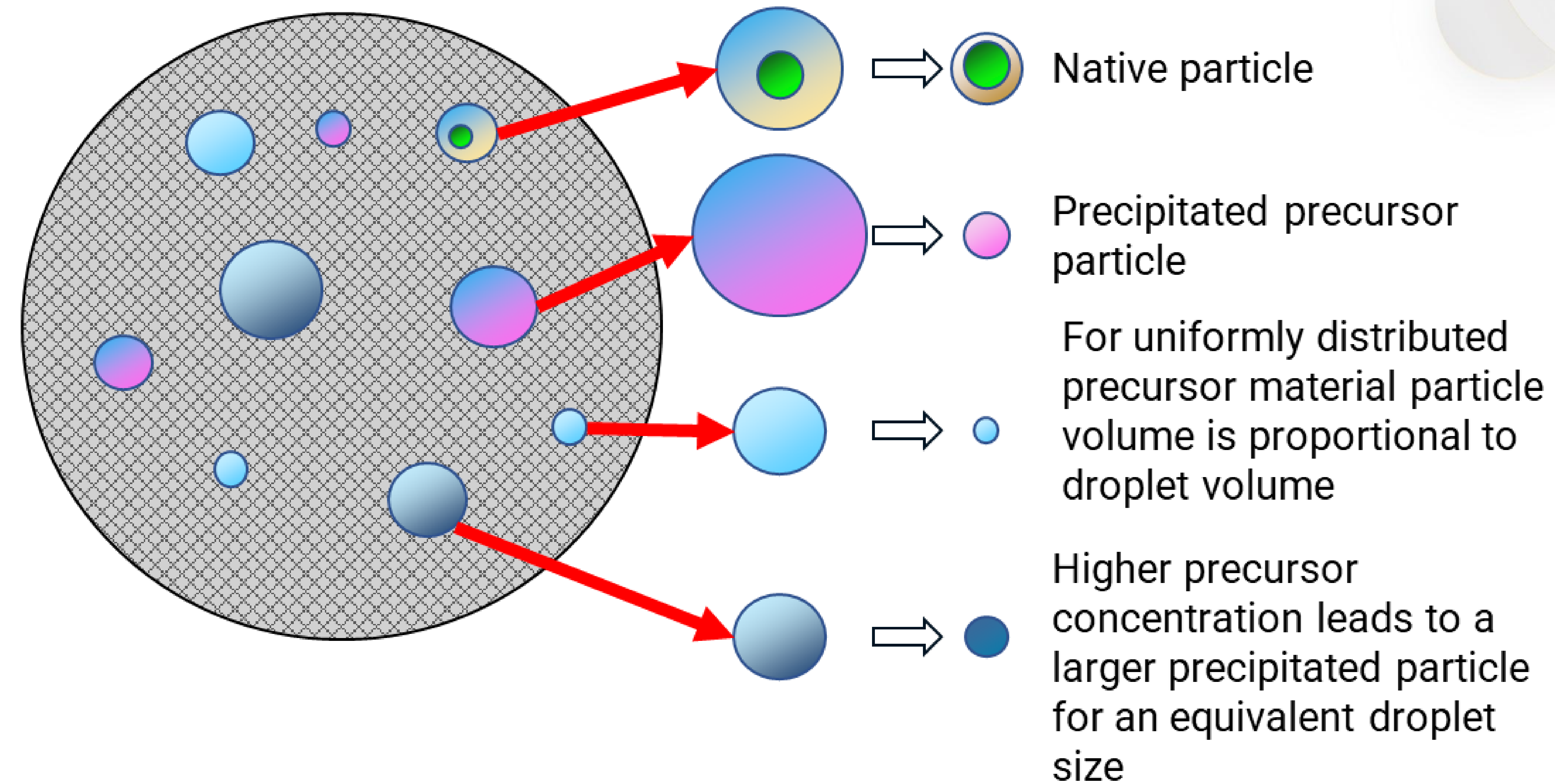
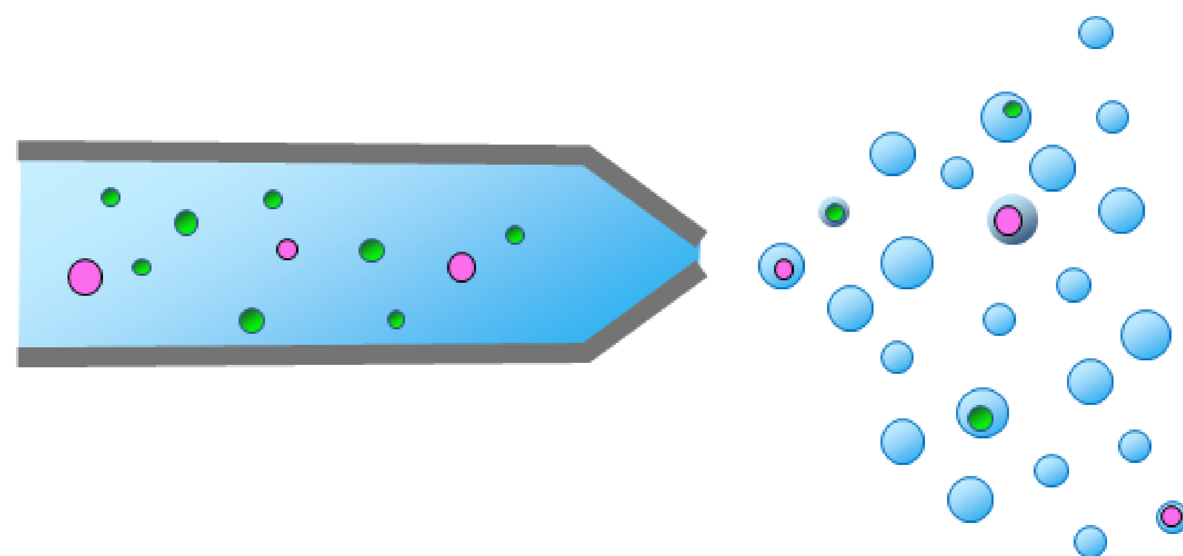
Defect Formations From Liquid Contaminates

- Ultrapure water and chemicals are used to rinse wafers. Wafer is spin dried after rinsing
- Following Spin Drying, small droplets remain on the wafer, which then evaporate



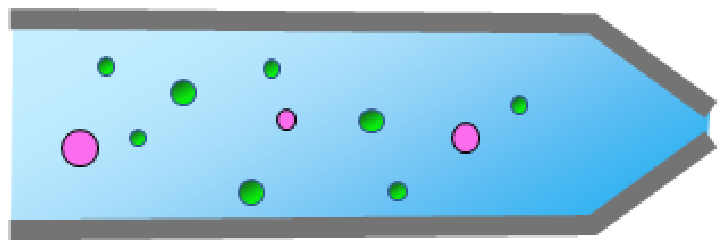


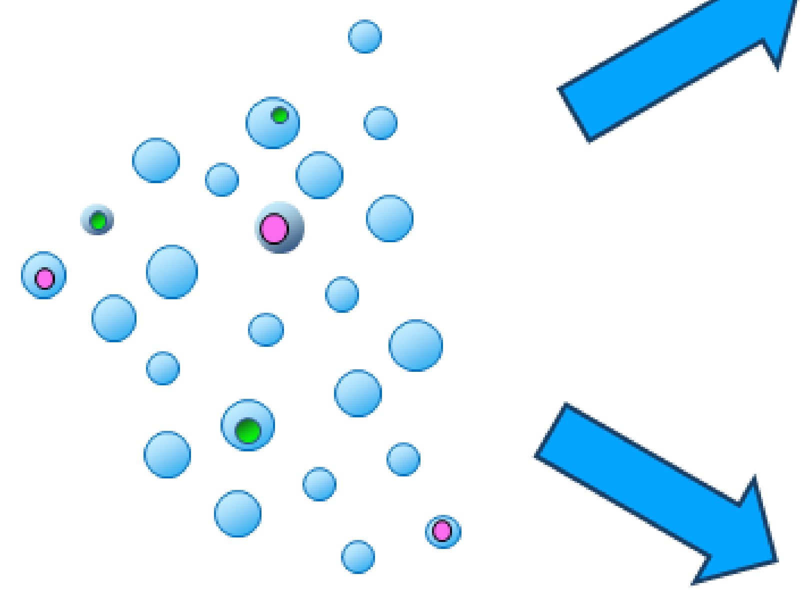
Nebulization mimics formation of localized droplets remaining on wafer after spin drying

Source: Oberreit, et al, Solid State Phenomena, vol. 346.

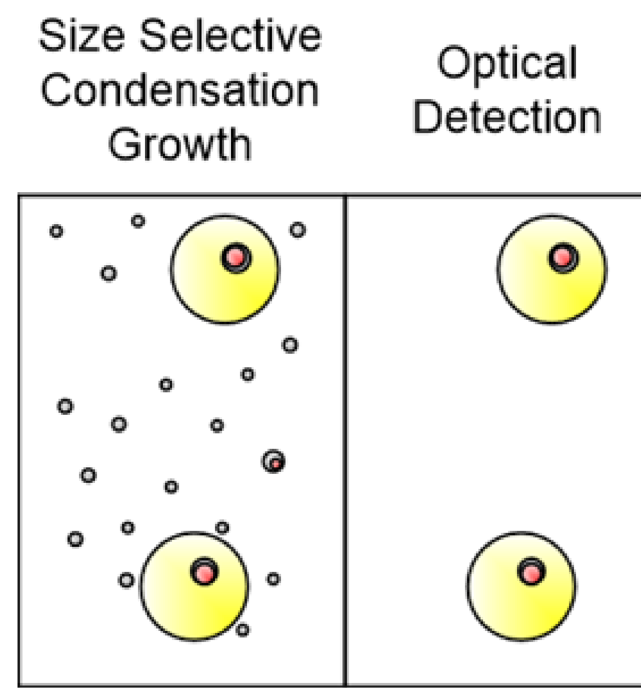
Aerosolization-Based Analytical Methods

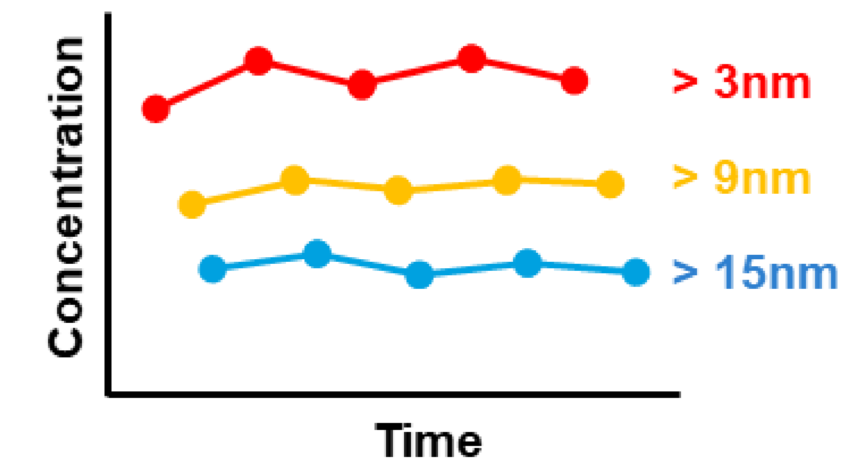
Nebulizer
Converts liquid sample into individual droplets of pure liquid and particle containing droplets



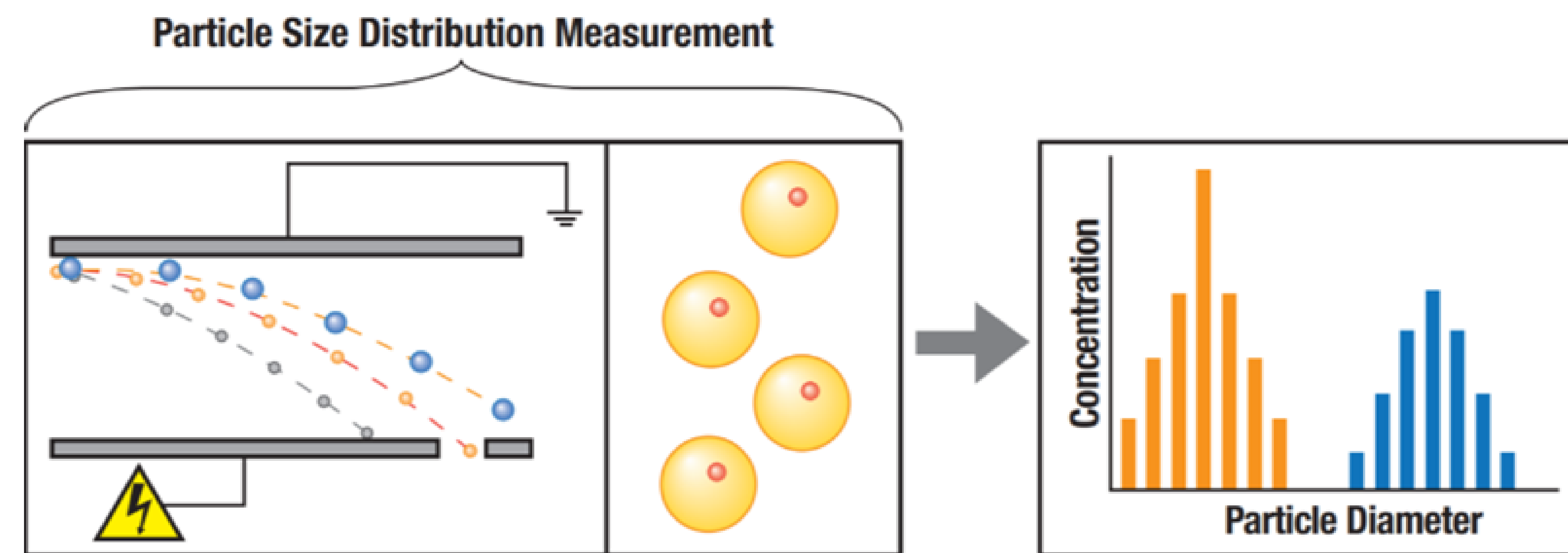


Counter
Counts particles above the size threshold

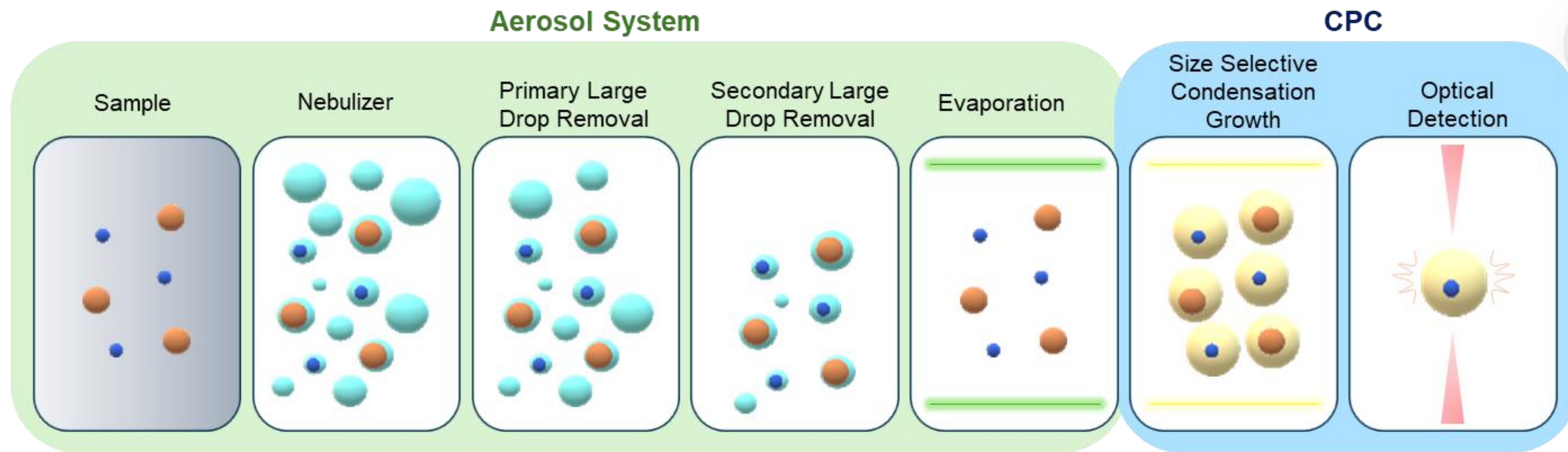


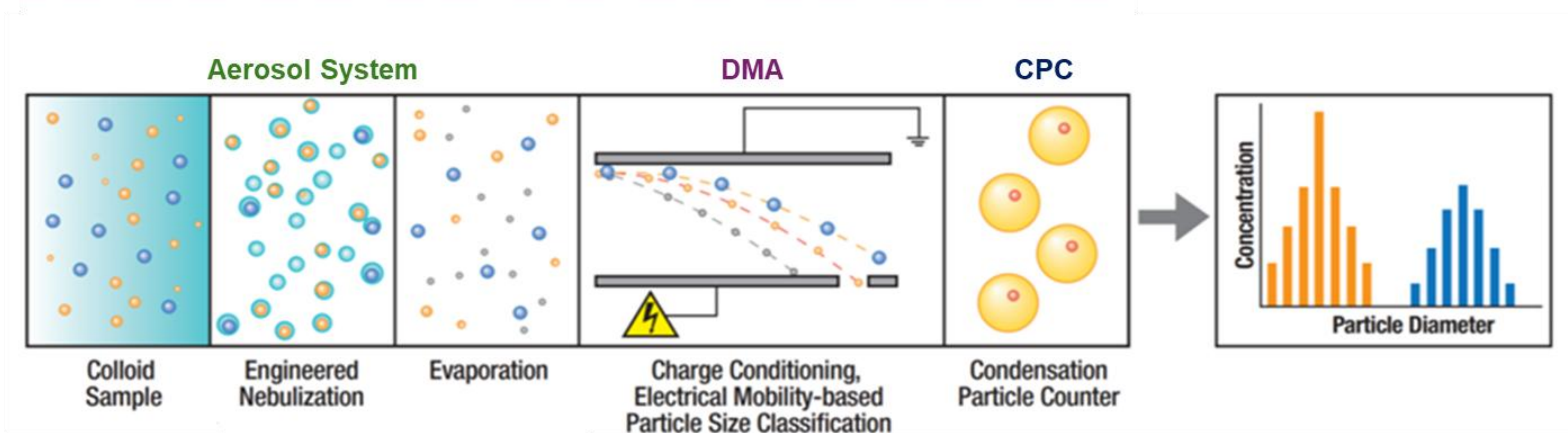


Sizer
Separates the particles based on diameter and then counts particles to provide a histogram



Principles of Operation
Aerosolization-Based Measurement Methods

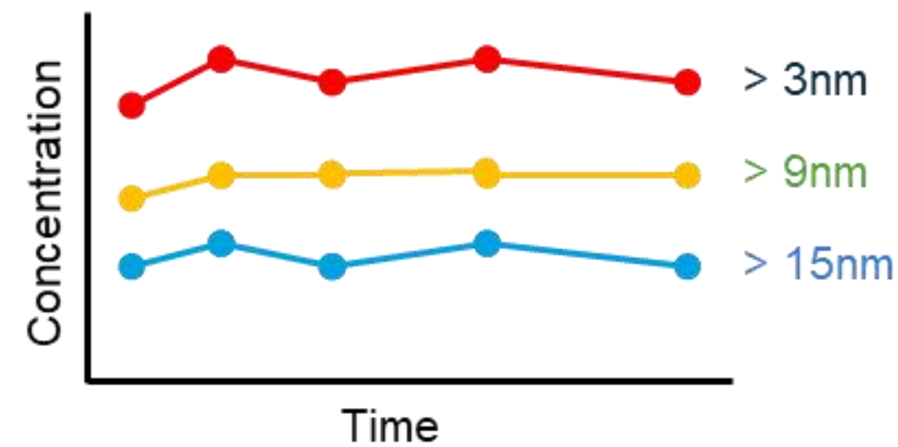




Kanomax FMT Aerosolization-Based Instruments

Scanning Threshold Particle Counter 3

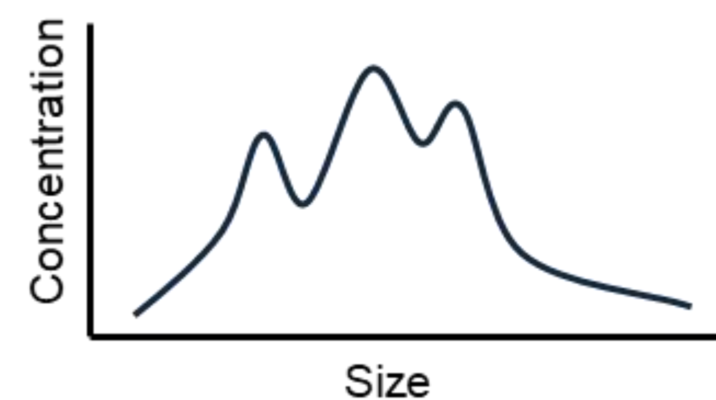
Counts particles above 3 selectable threshold diameters



- Count concentration measurement using CPC alone, without DMA
- Fast event detection in process environments (for example, microelectronic fabrication facilities)
- Real-time monitoring instrumentation
- Lower detection limits
- Compatible with several chemicals of interest
- DL – 1e3 to 1e4 #/mL

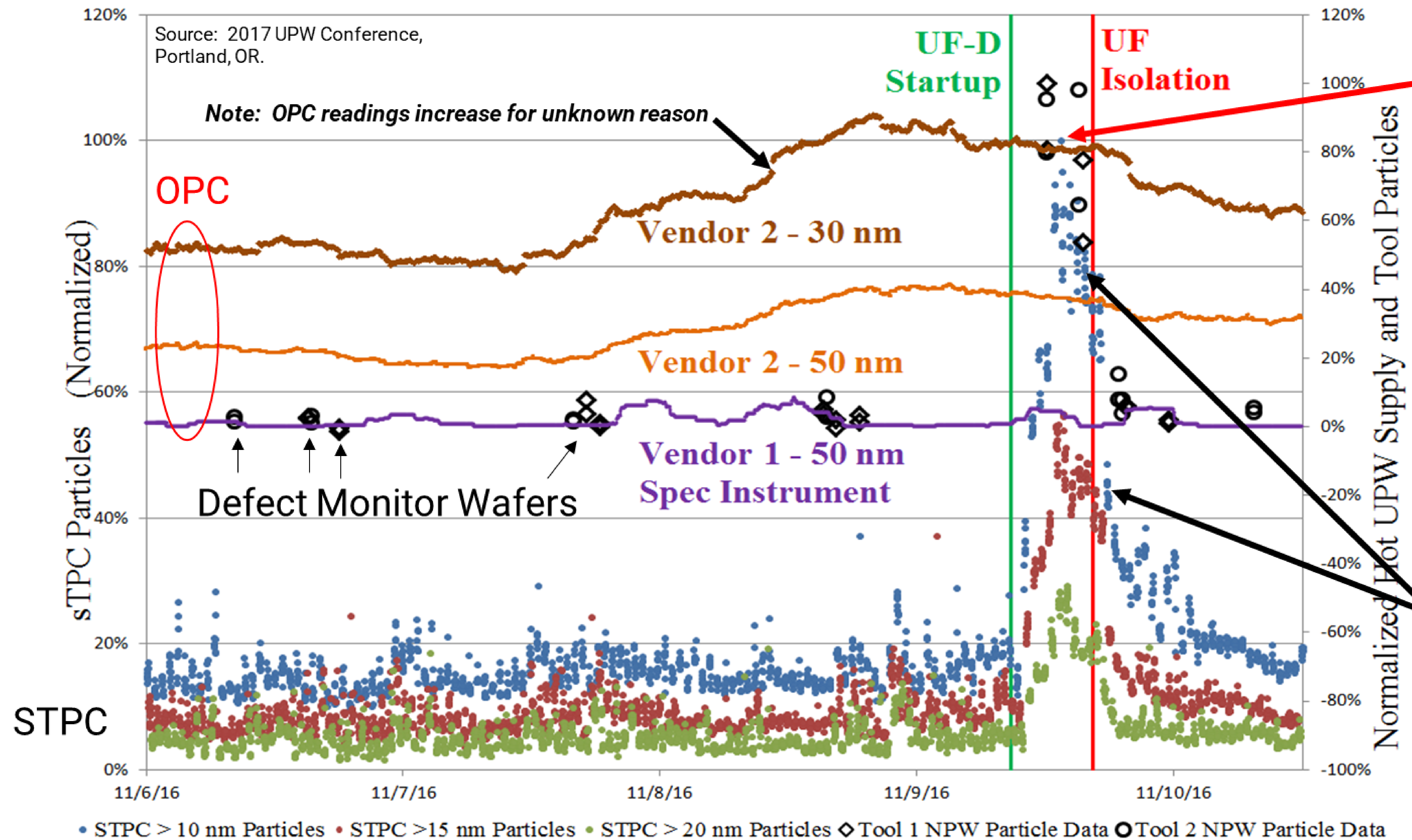
Liquid Nanoparticle Sizer

Measures particle size distributions from 5 to 500 nm



- Particle size distribution analysis based on electrical mobility (DMA)
- Suitable for high-precision analysis in laboratory environments
- Higher resolution for sizing
- Suitable when added particles are present (CMP slurries, filter testing, extracts of critical components like IEX, filters etc.)
- DL – 3e7 to 3e8 #/mL

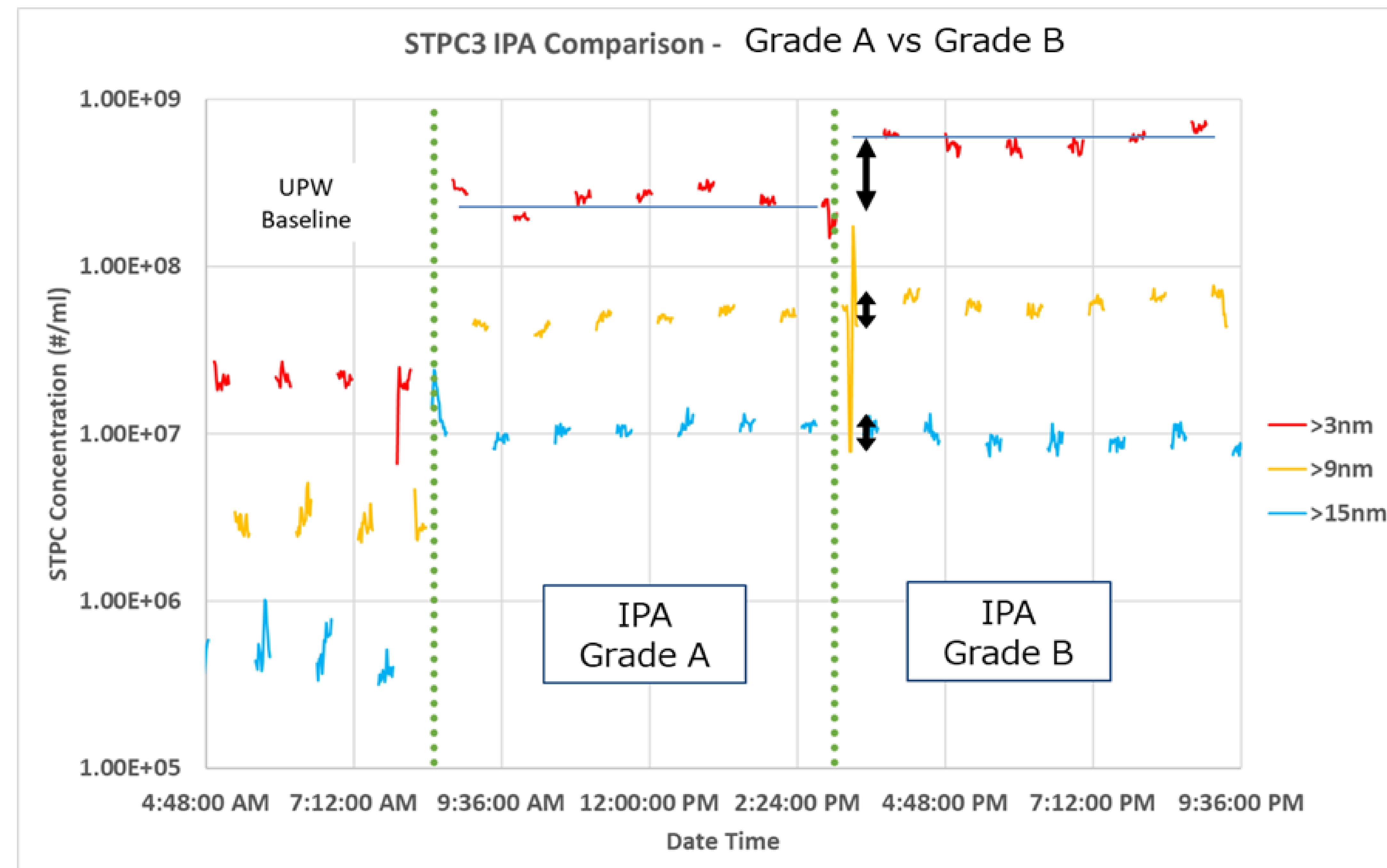
STPC and OPCs in a Sustaining Engineering Role
Optical Particle Counters (OPCs) were blind to UltraFilter (UF) Startup Yield Event



OPC did not detect these yield killing particles

STPC detects particle excursion in real time, before test wafers on mulit-$M wafer inspection tool.

STPC3 for distinguishing IPA

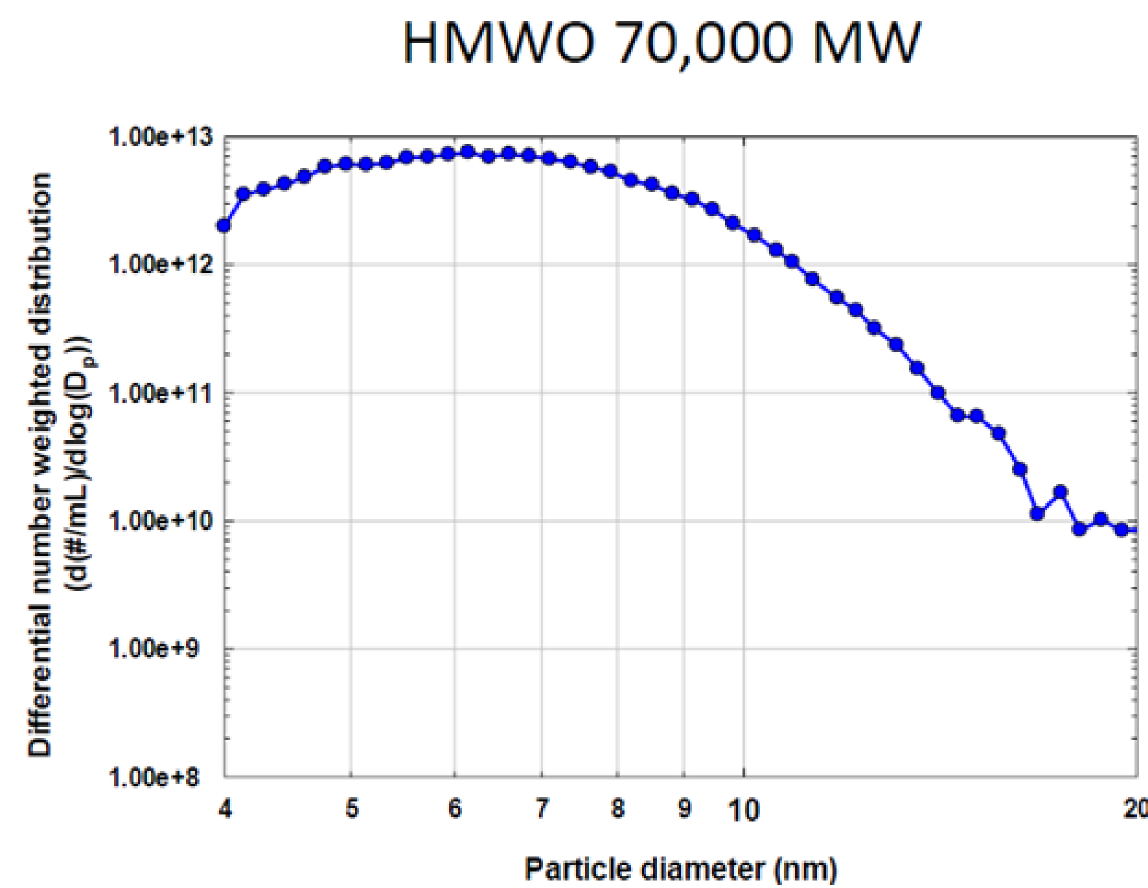


STPC3 is used for continuous monitoring of IPA quality

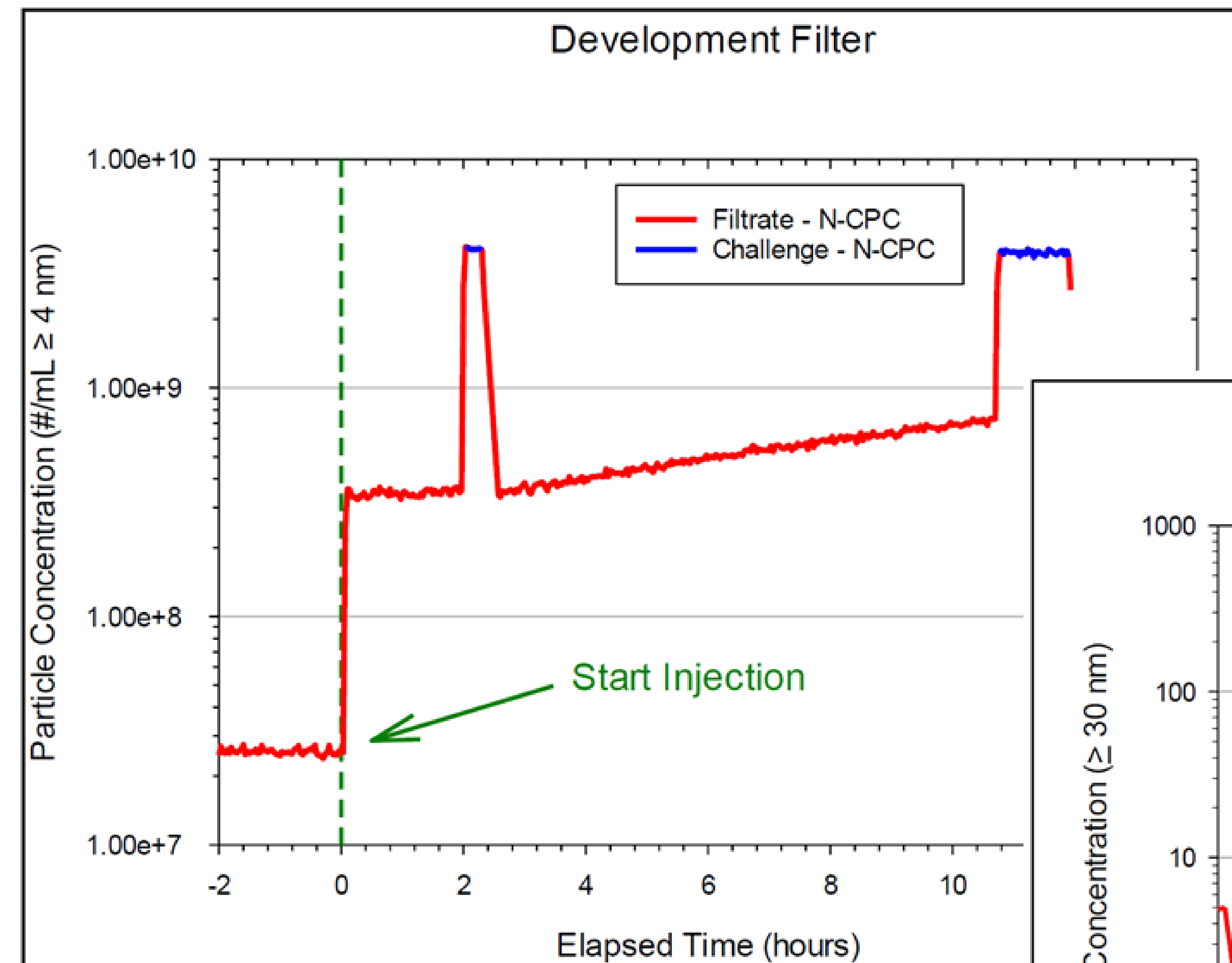
Aerosolization + Threshold Particle Counting Filter Testing

- TPC allows for testing of modern filters using real-world challenges

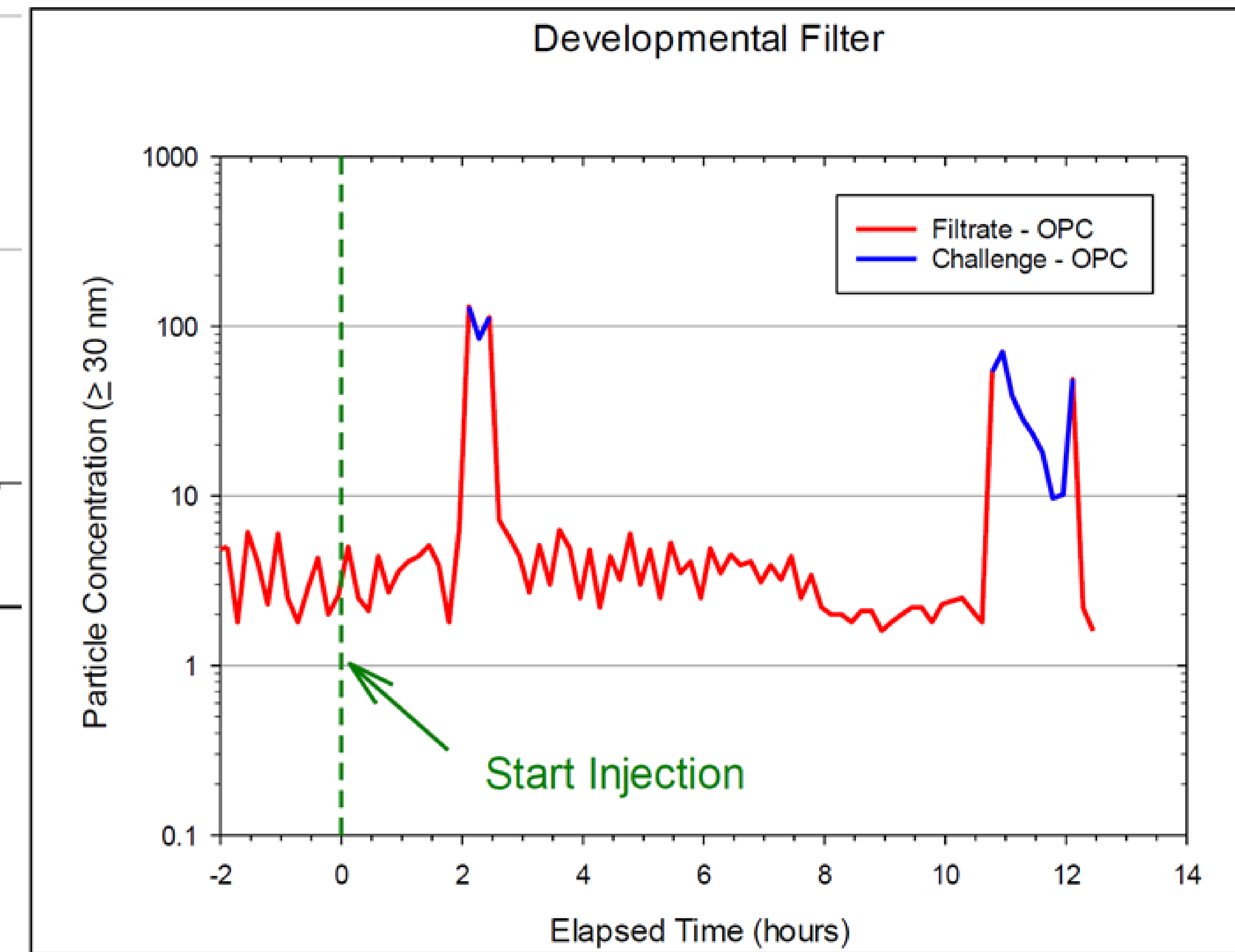
HMWO 70,000 MW



Aerosolization + TPC > 4nm



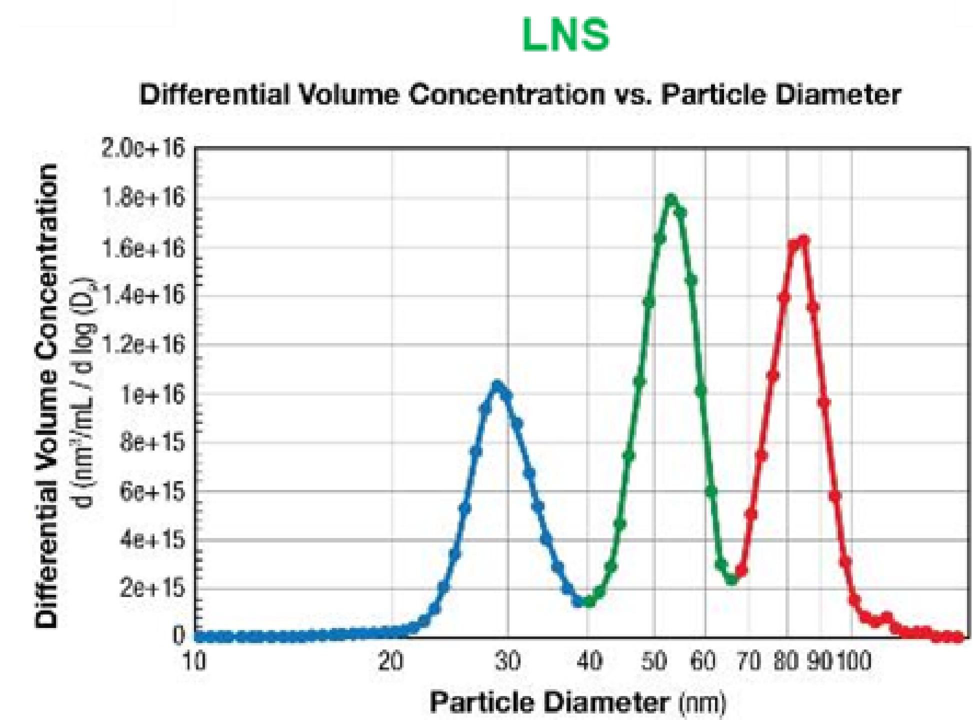
Lighthouse NC30 OPC

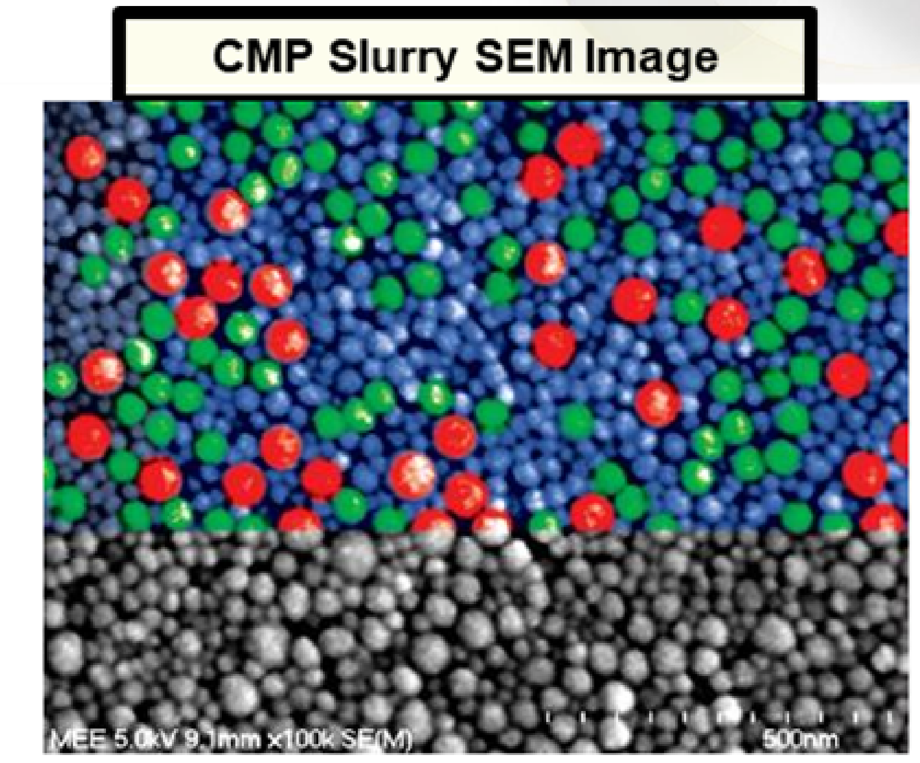


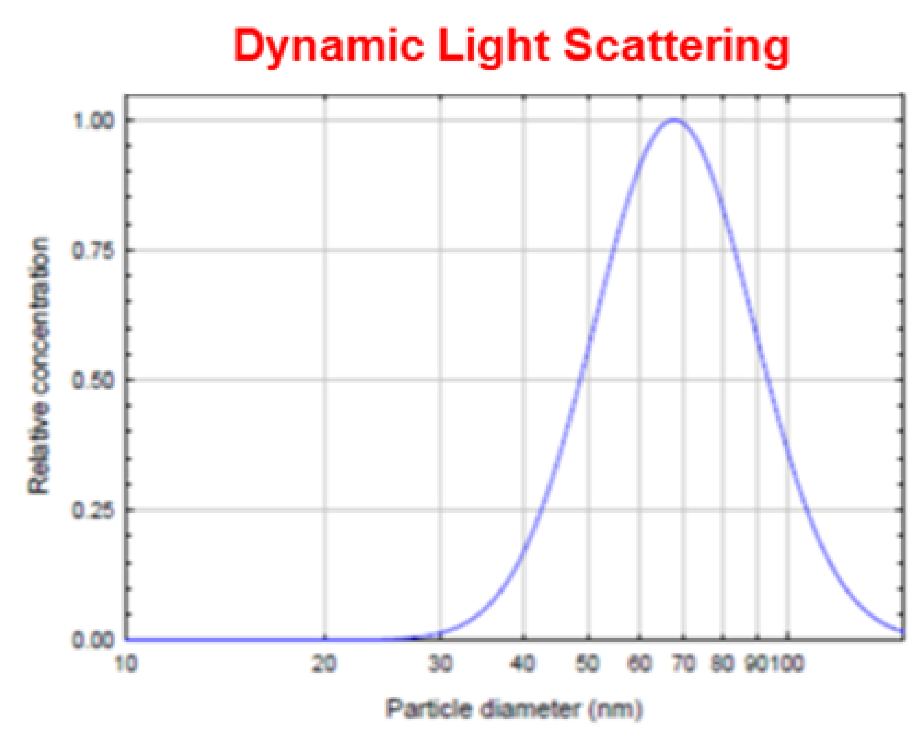
Van Schooneveld et al, Ultrapure Micro 2020

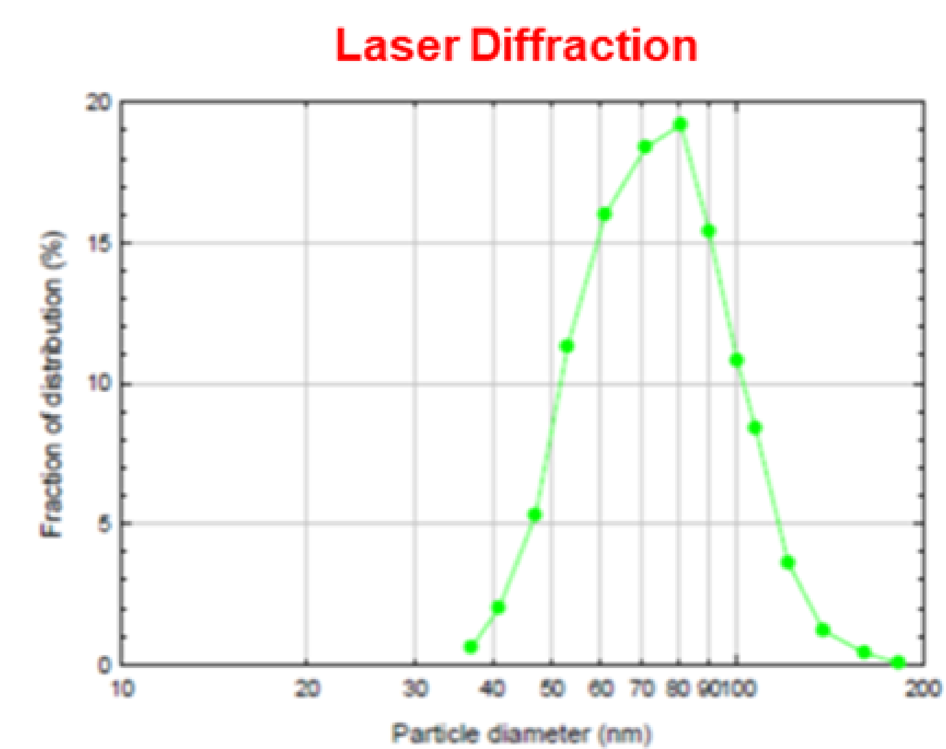
Liquid Nanoparticle Sizer (LNS) Competitive Methods

- Current in-situ methods are often unable to accurately resolve multimodal distributions
- Concentration information is not provided
- Microscopy methods are costly and time consuming
- Liquid Nanoparticle Sizer system overcomes these limitations





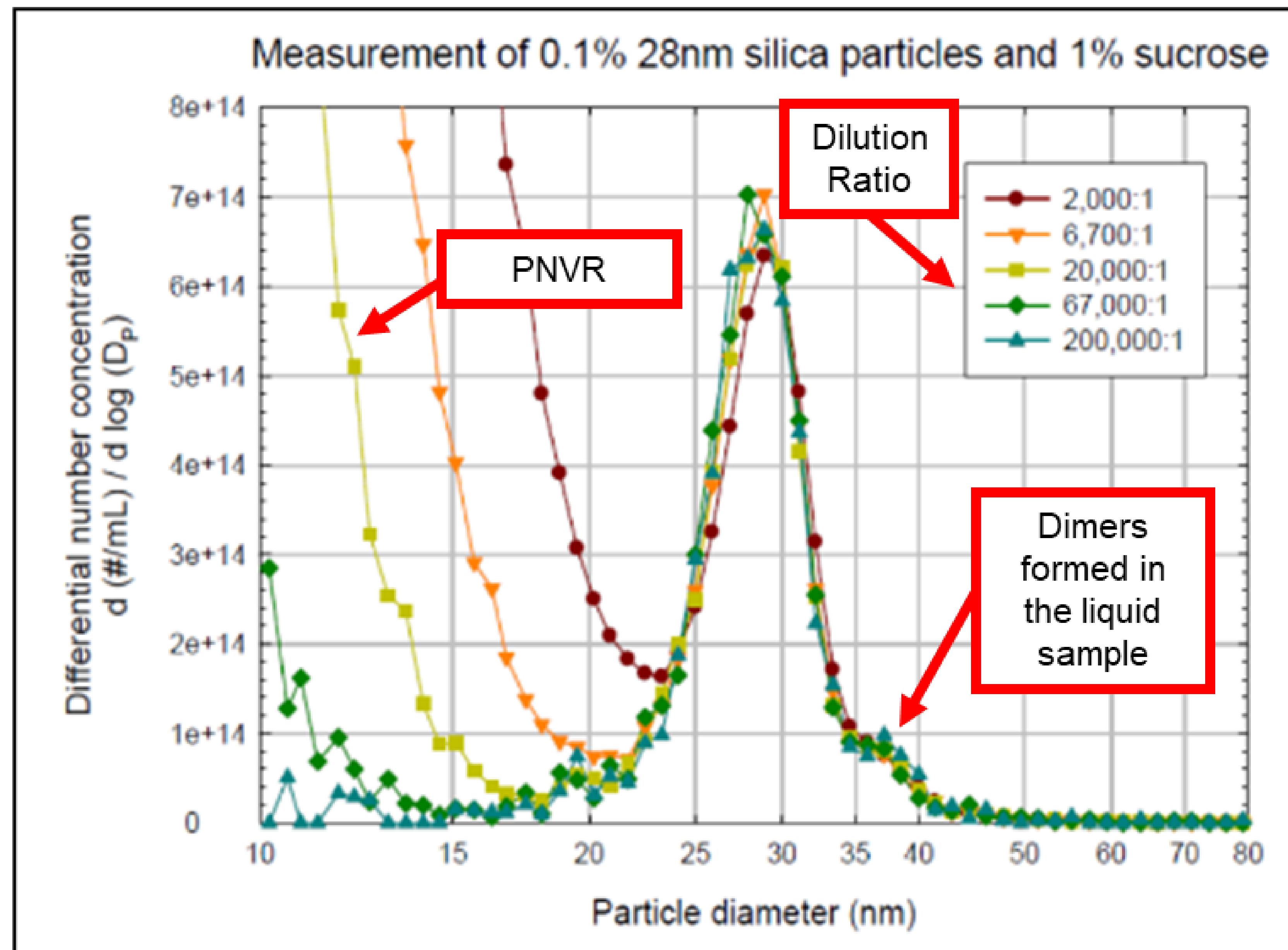




Reference: Litchy, M. et.al.: Pittcon 2012

DLS: PSS NICOMP 380ZLS LD: Horiba LA950

Liquid Nanoparticle Sizer (LNS)
Concentration Effects– Differentiating Particle and Residue Peaks

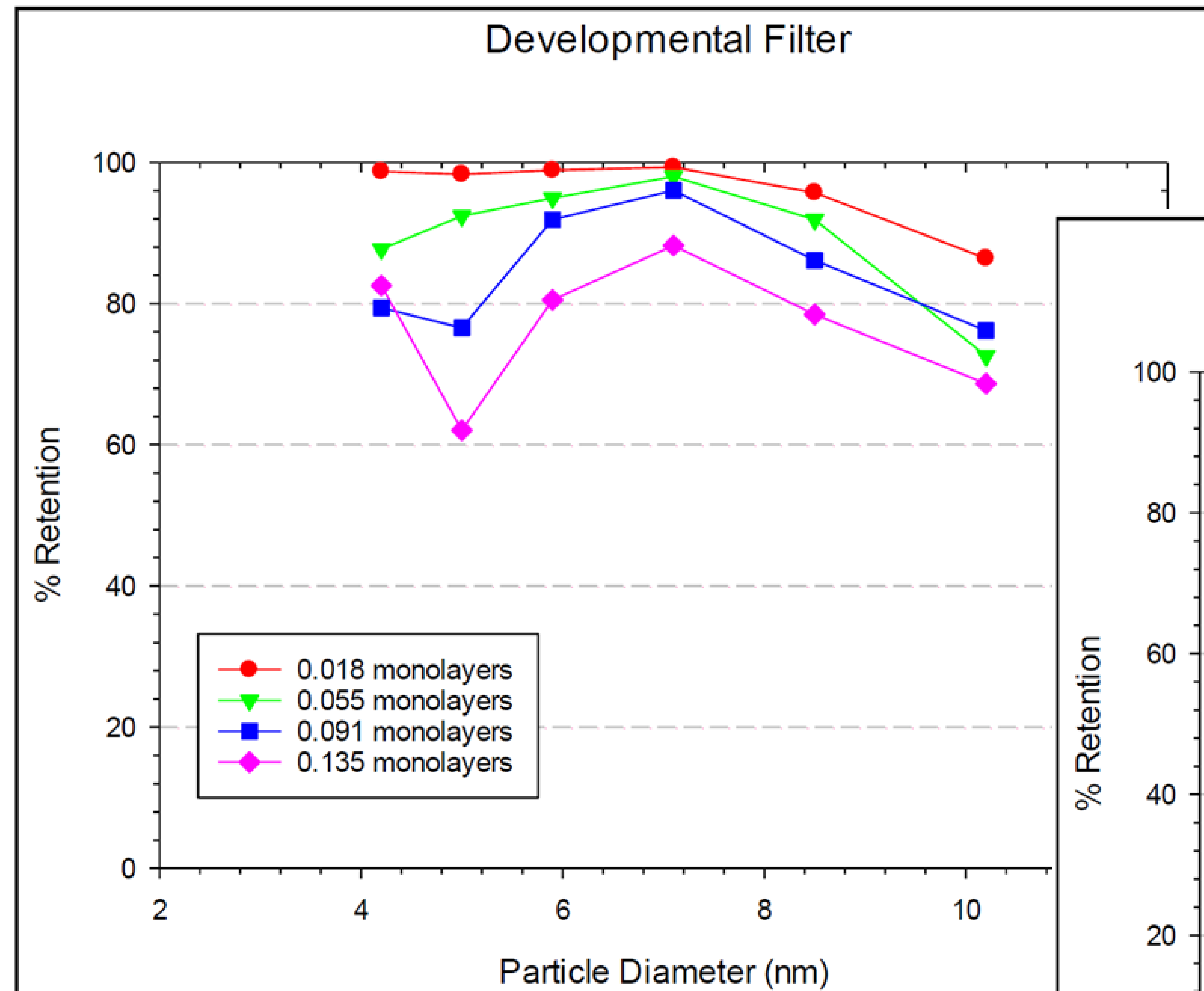


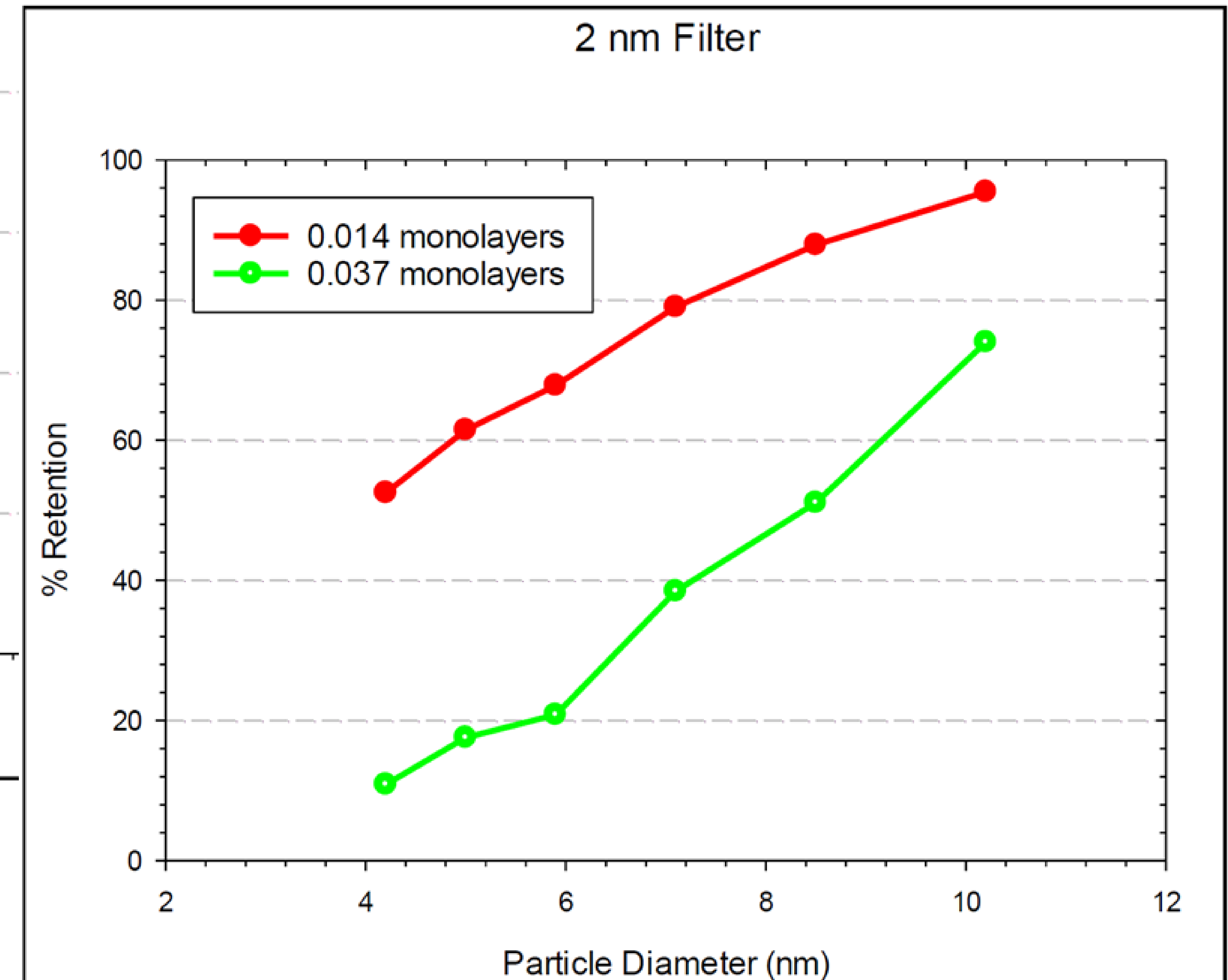
Litchy, M. et.al.: Pittcon 2012

- Dilution allows for discrimination between Precipitated Nonvolatile Residue (PNVR) and liquid borne colloid particles
  - PNVR aerosol particle size distribution will change in size but not total concentration
  - Colloid particle size distribution will change in total concentration but not size
- Dilution also allows for identification of coagulation in liquid or during aerosolization
  - Liquid coagulation will not change with dilution ratio
  - Coagulation due to aerosolization (from two particles present within a droplet) will change as a function of dilution ratio

Liquid Nanoparticle Sizer (LNS) Filter Testing

- LNS allows for high size resolution measurement of filter retention as a function of particle size





Van Schooneveld et al, Ultrapure Micro 2020

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UNIVERSAL CONTAMINATION ANALYZER

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Surface Enhance Particle Sizing (SEPS) for On-Wafer Detection of sub-10nm Particles

Marie Tripp, VP Customer Relations

UNISERS AG

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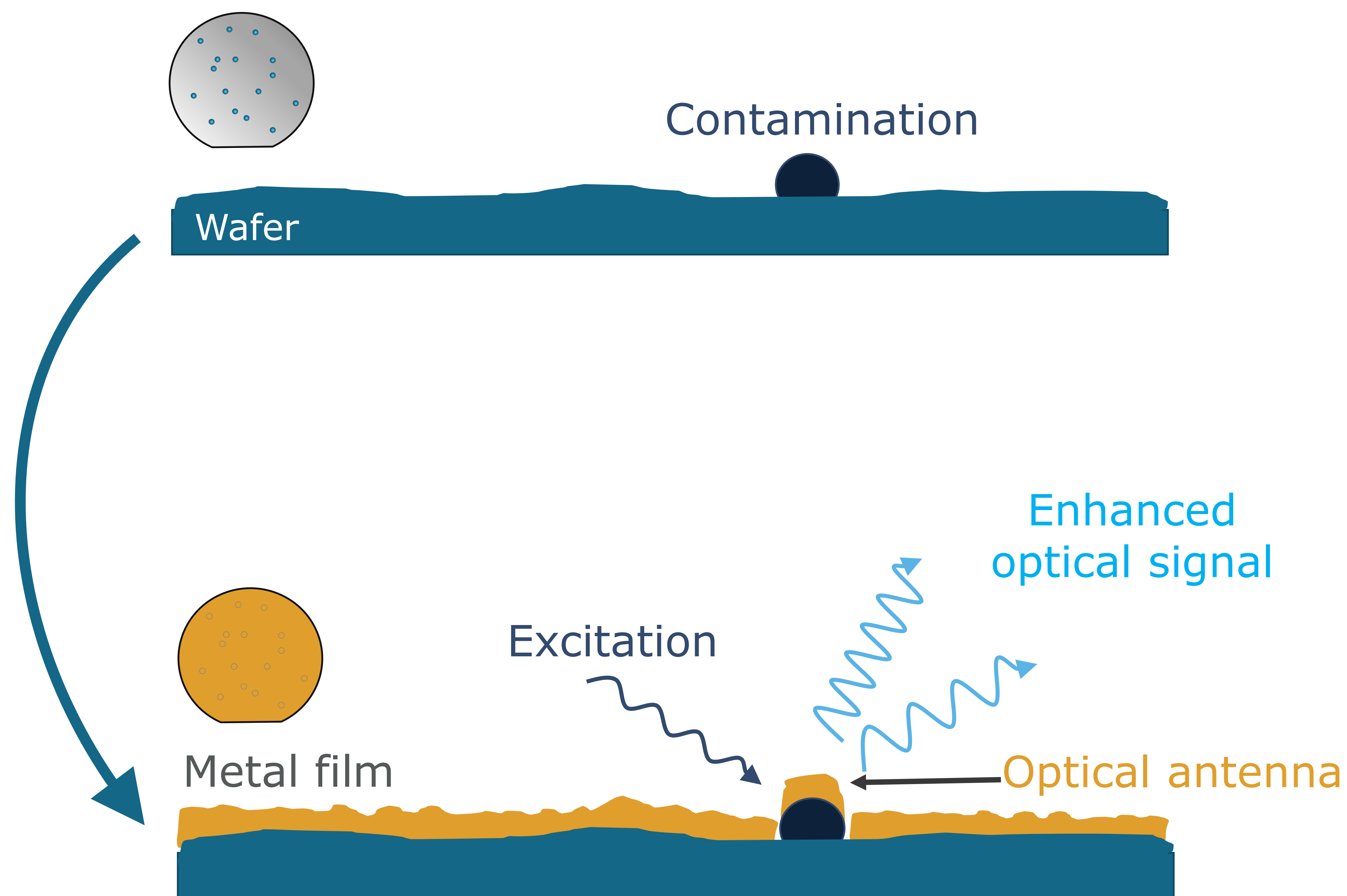
UNISERS Core Technology

- Enhanced **Rayleigh** scattering for Surface Enhanced Particle Sizing (**SEPS**)
- Enhanced **Raman** scattering for Surface Enhanced Raman Speciation (**SERS**)

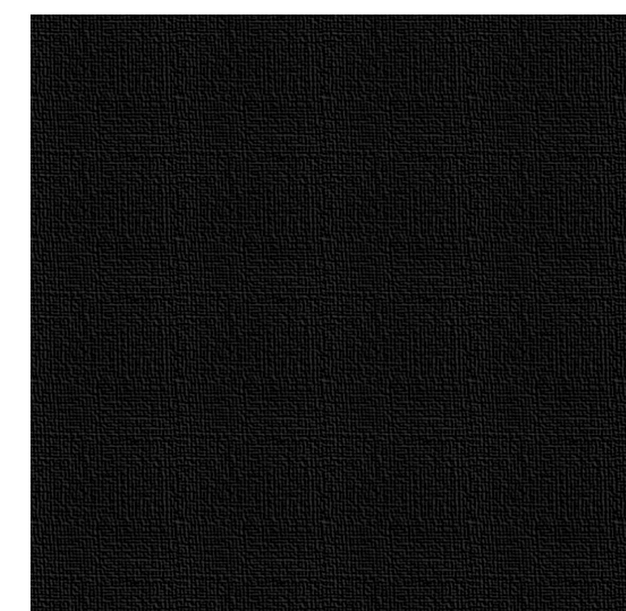


"Let the dirt shine"

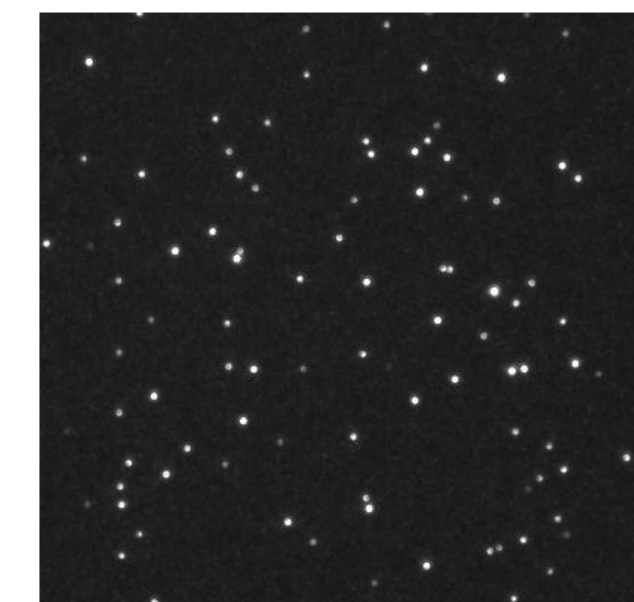
Step 1: UNISERS coating
Unique growth and morphology of a special metal film leads to the formation of an optical antenna on every defect.

Step 2: UNISERS analyzer
Upon illumination the optical antennas lead to strongly enhanced optical signals from the contamination. The UNISERS analyzer provides:

- Size
- Location on wafer
- Morphology
- Index of refraction
- Molecular fingerprint



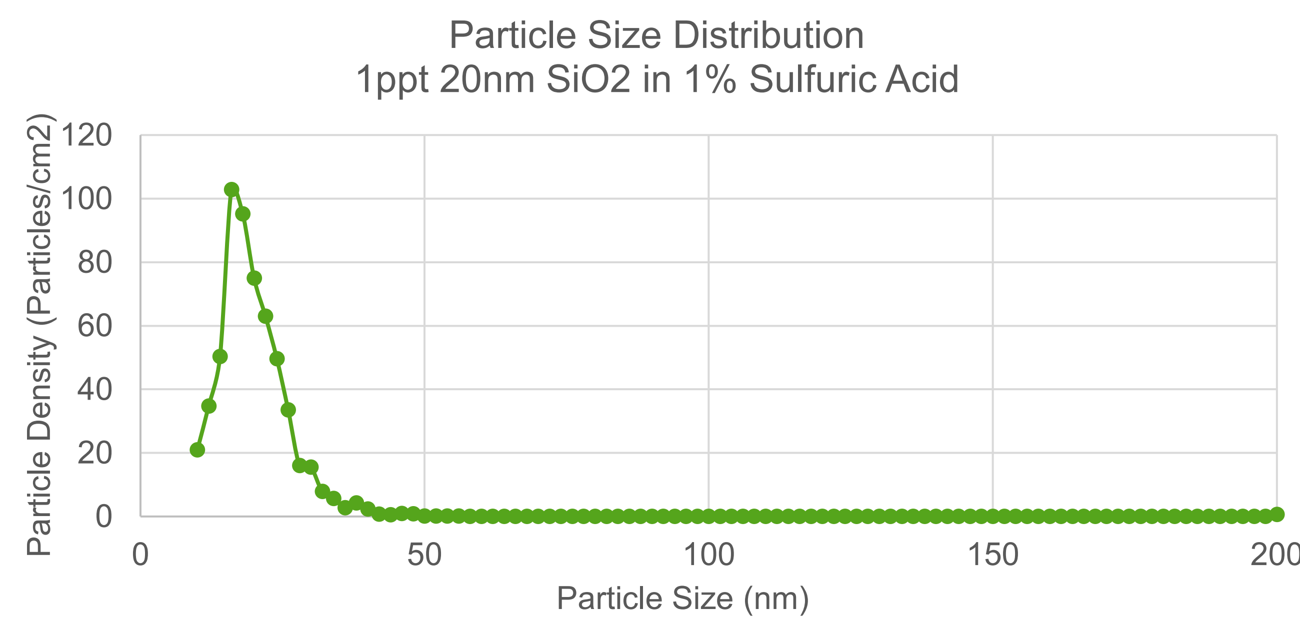
Before enhancement



After enhancement

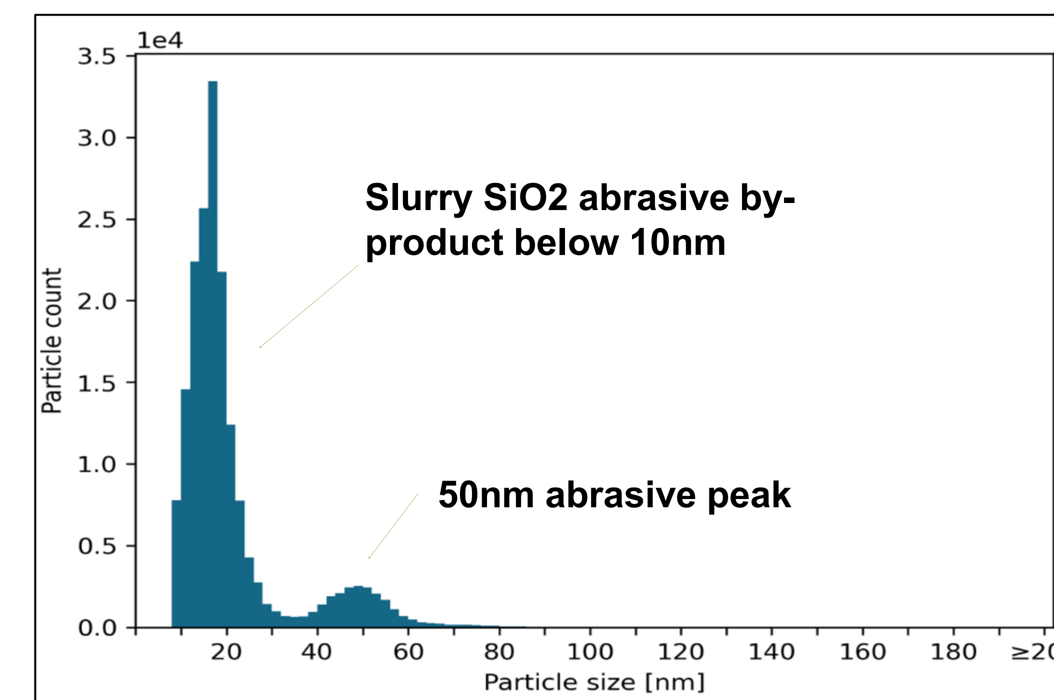
UNISERS Application Overview

On wafer metrology for liquid micro-contamination



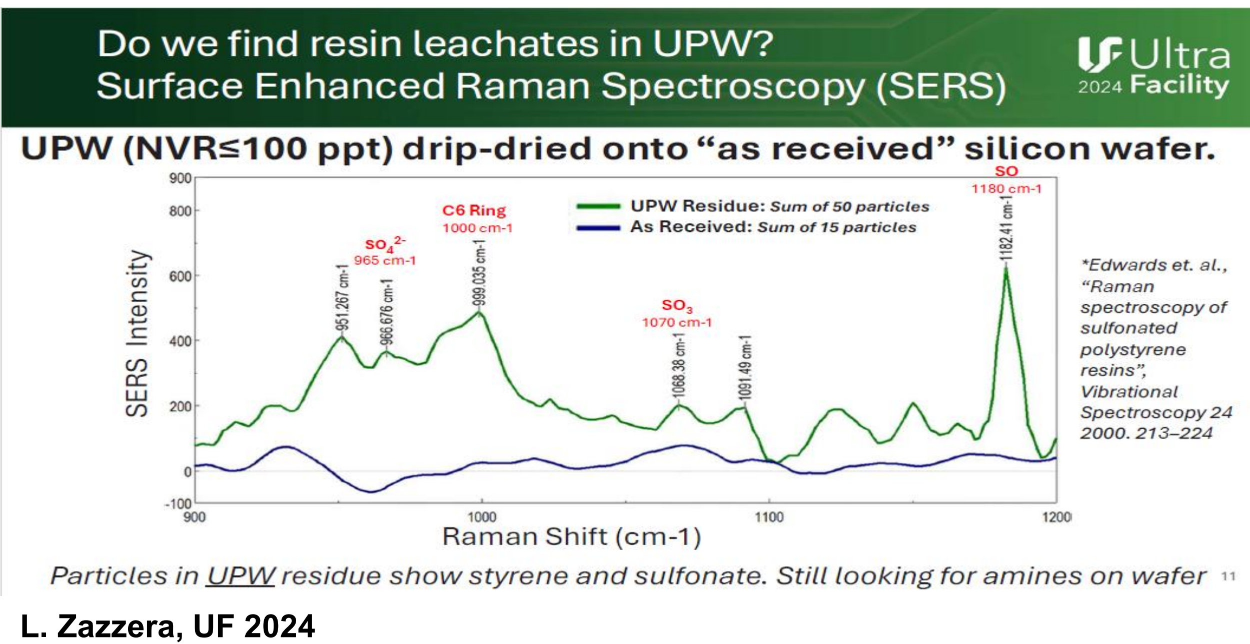
Fully resolved peak from 20nm SiO2 particles detected on wafer from sulfuric acid at 1ppt concentration

Best on-wafer sensitivity



CMP SiO2 abrasive by-product sub-10nm detected on SiN wafer

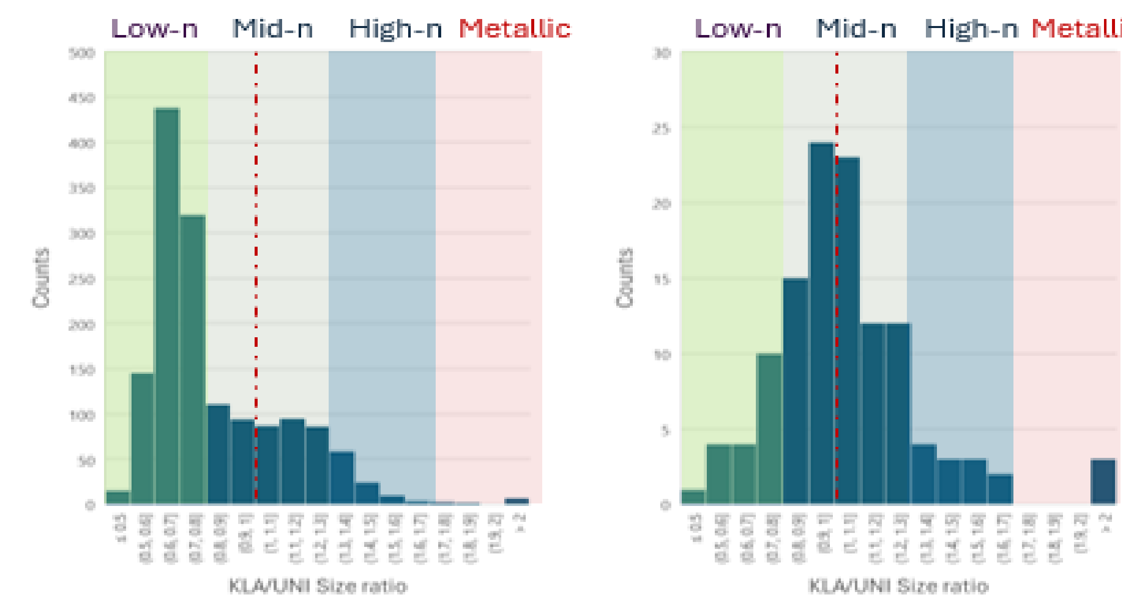
Organic defect speciation



L. Zazzera, UF 2024

Styrene and sulfonate identified in UPW residues by SERS

Metal particle excursion detection



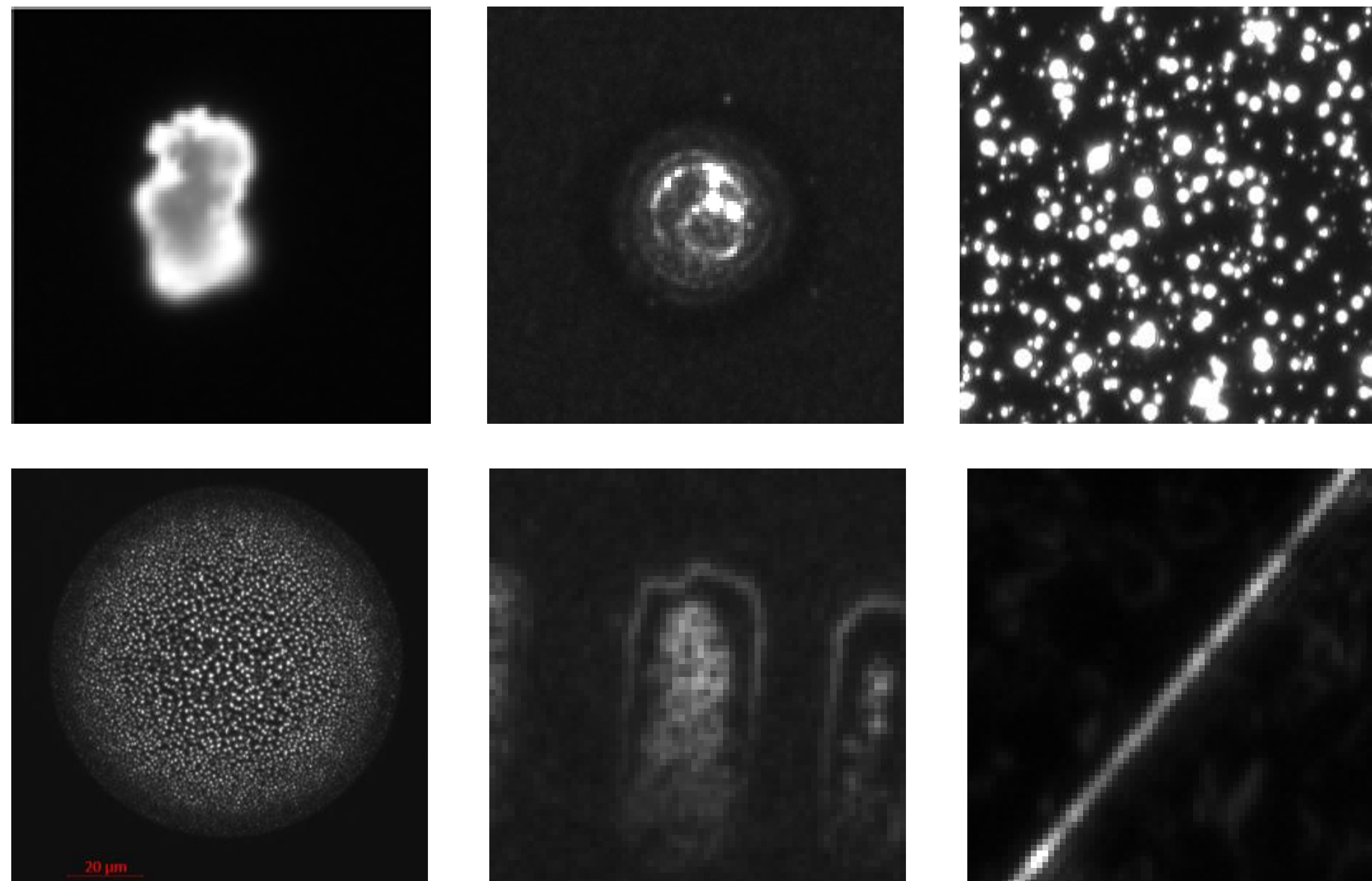
KLARF-based automated defect review for index of refraction classification

SEPS for Silica in Ultrapure Water

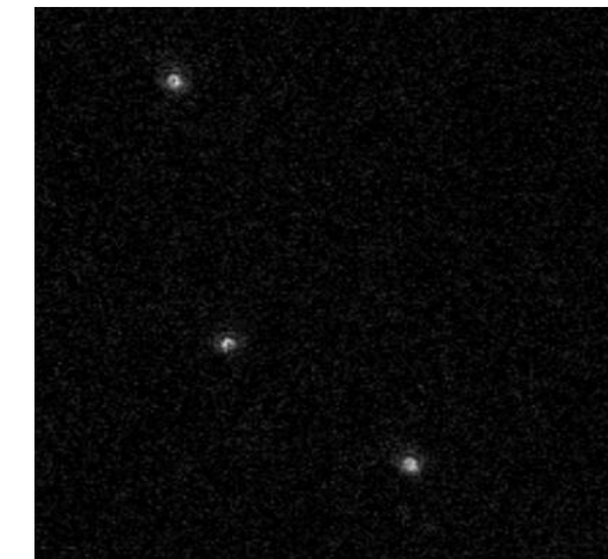
Surface-enhanced particle sizing characterized using Silica particles in UPW

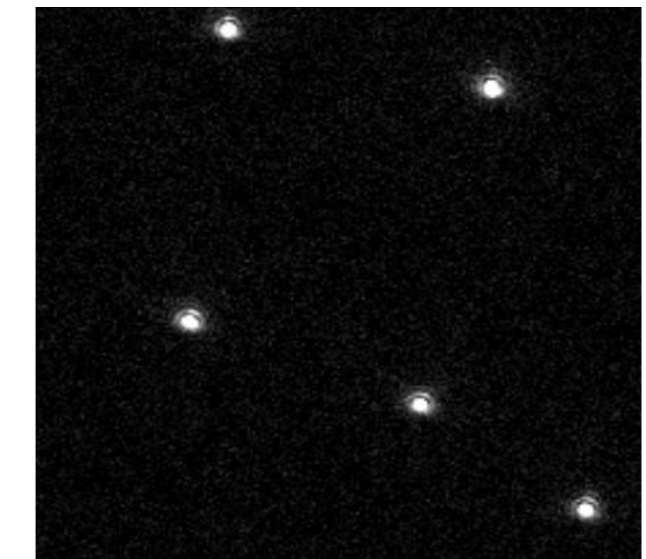
Acquire images with dark field illumination and apply image analysis to locate, size, and classify particles.

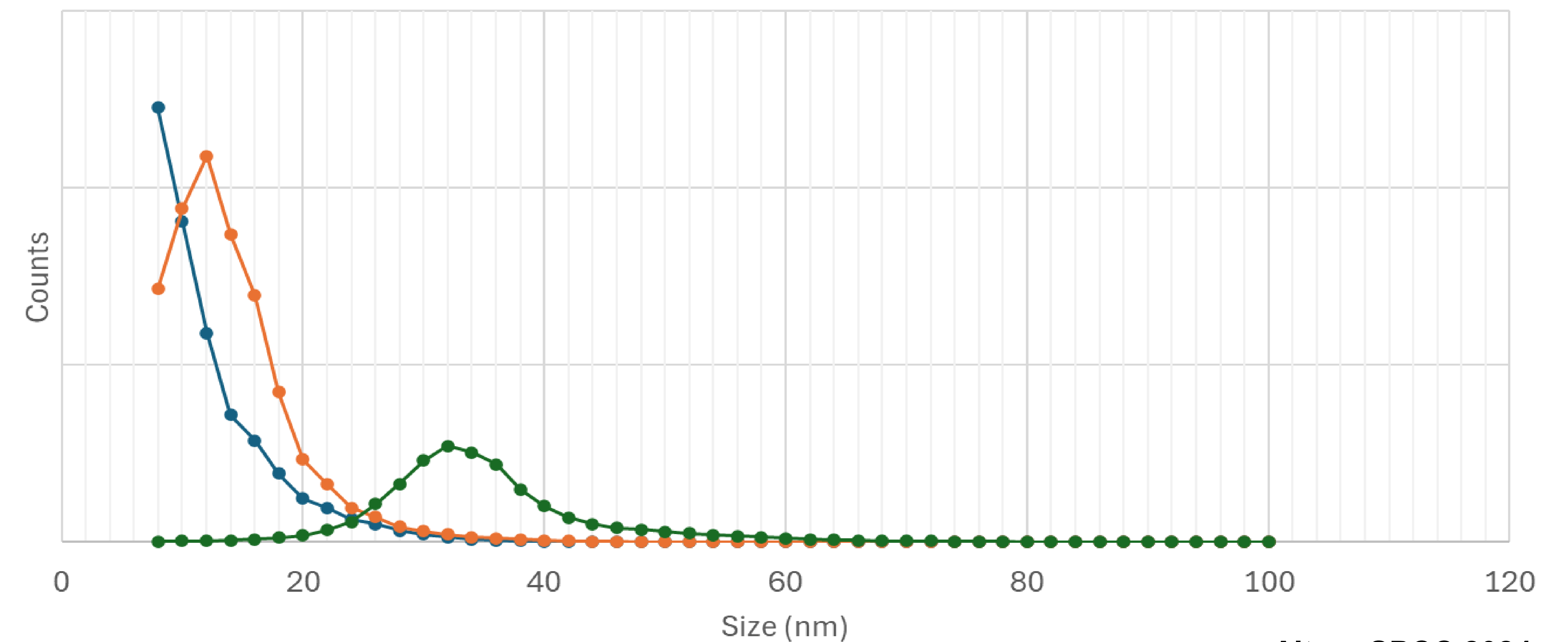
- **detection below 10nm (*particle size*)**
- **independent of material and substrate**



~8nm particles **~15nm particles** **~30nm particles**



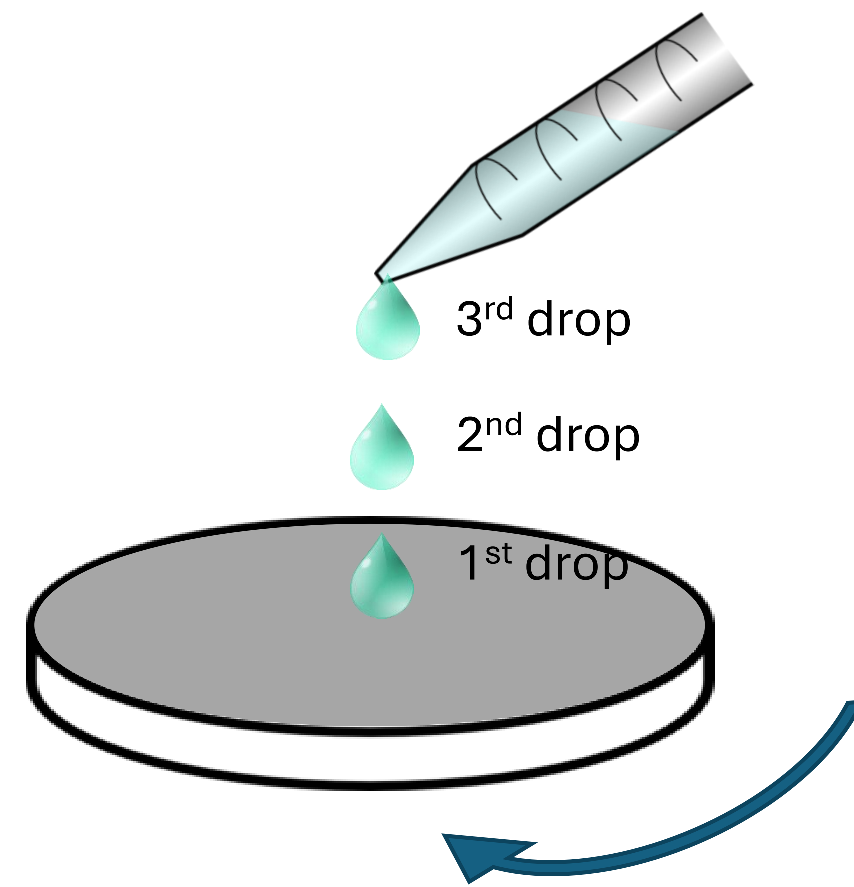


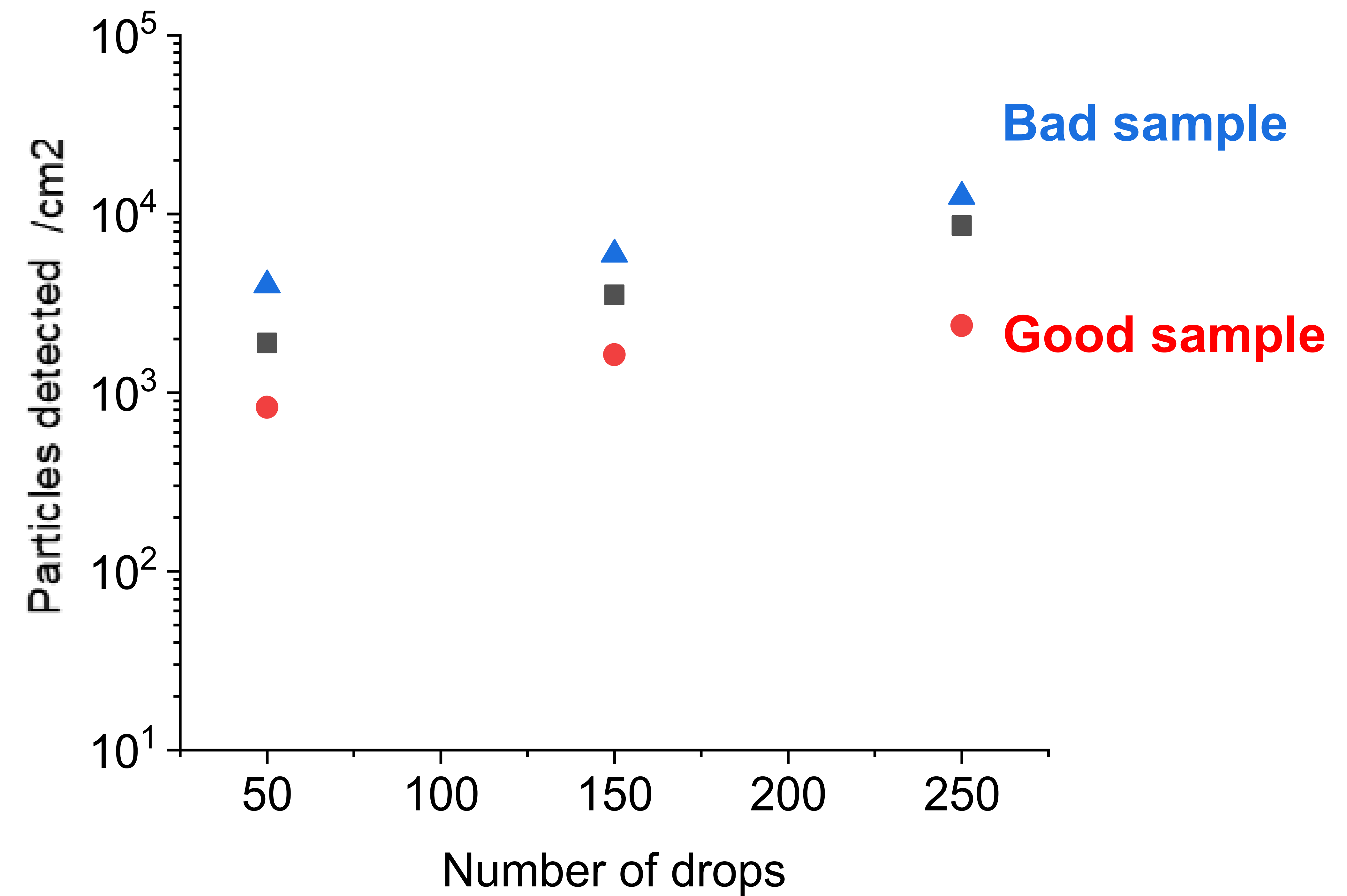


Altun, SPCC 2024

UNISERS Sequential Spin Coating (SSC)

Transitioning from Liquid Metrology to Wafer Metrology





- **Wafer size: 50mm to 300mm**
- **Automated droplet generator**
- **Up to now: UPW, IPA, TMAH, Hydrogen Peroxide, dilute Sulfuric Acid**

UNISERS Products

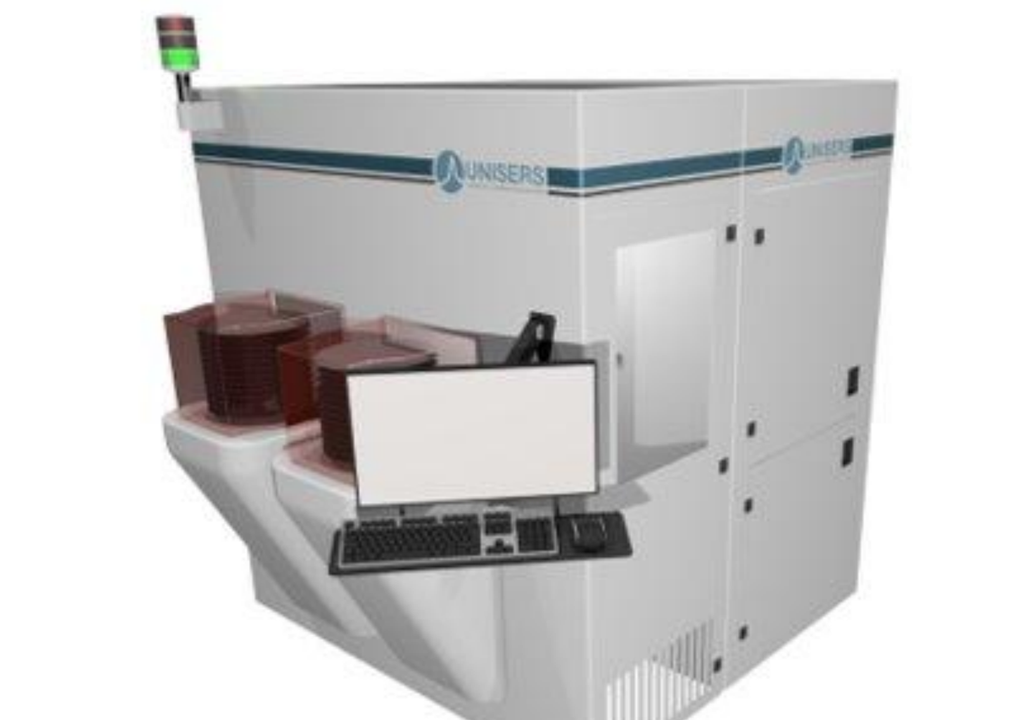
Lab suite for process development, material testing and trouble shooting

Explorer G1 for defect review, quality control and in-fab equipment monitoring

UNISERS SSC

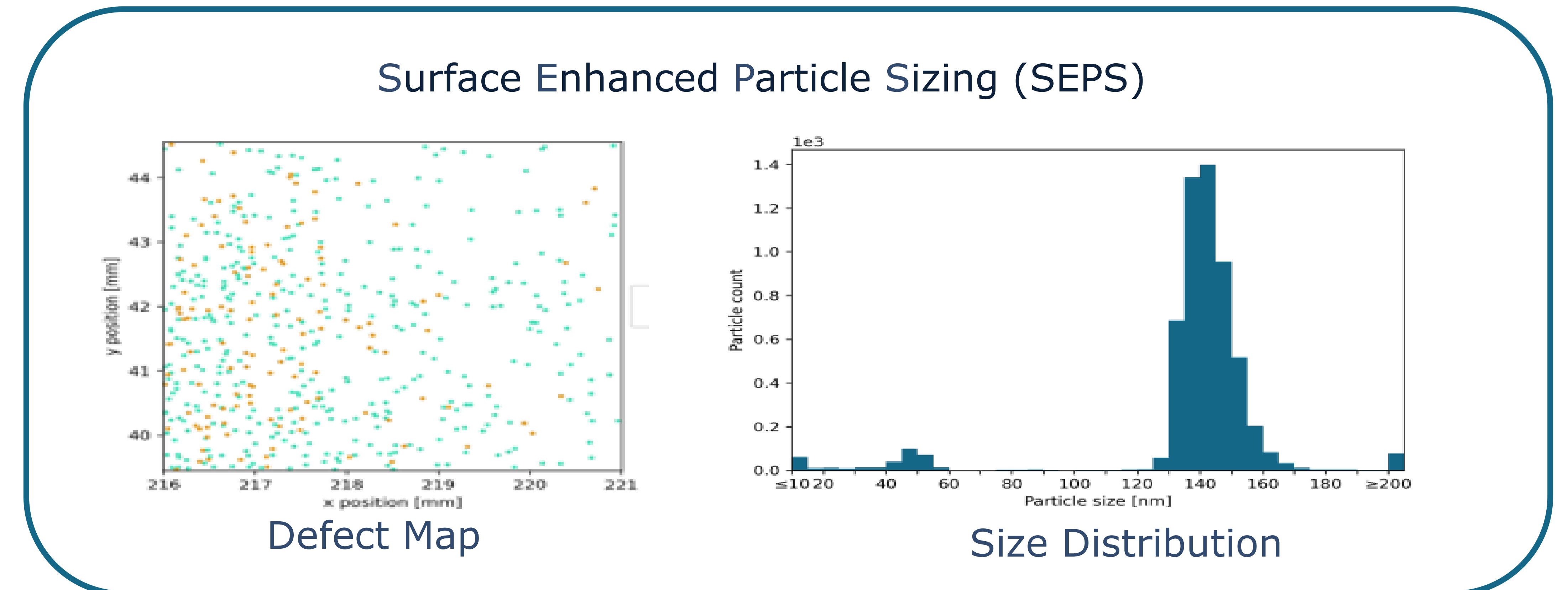
UNISERS Lab Suite

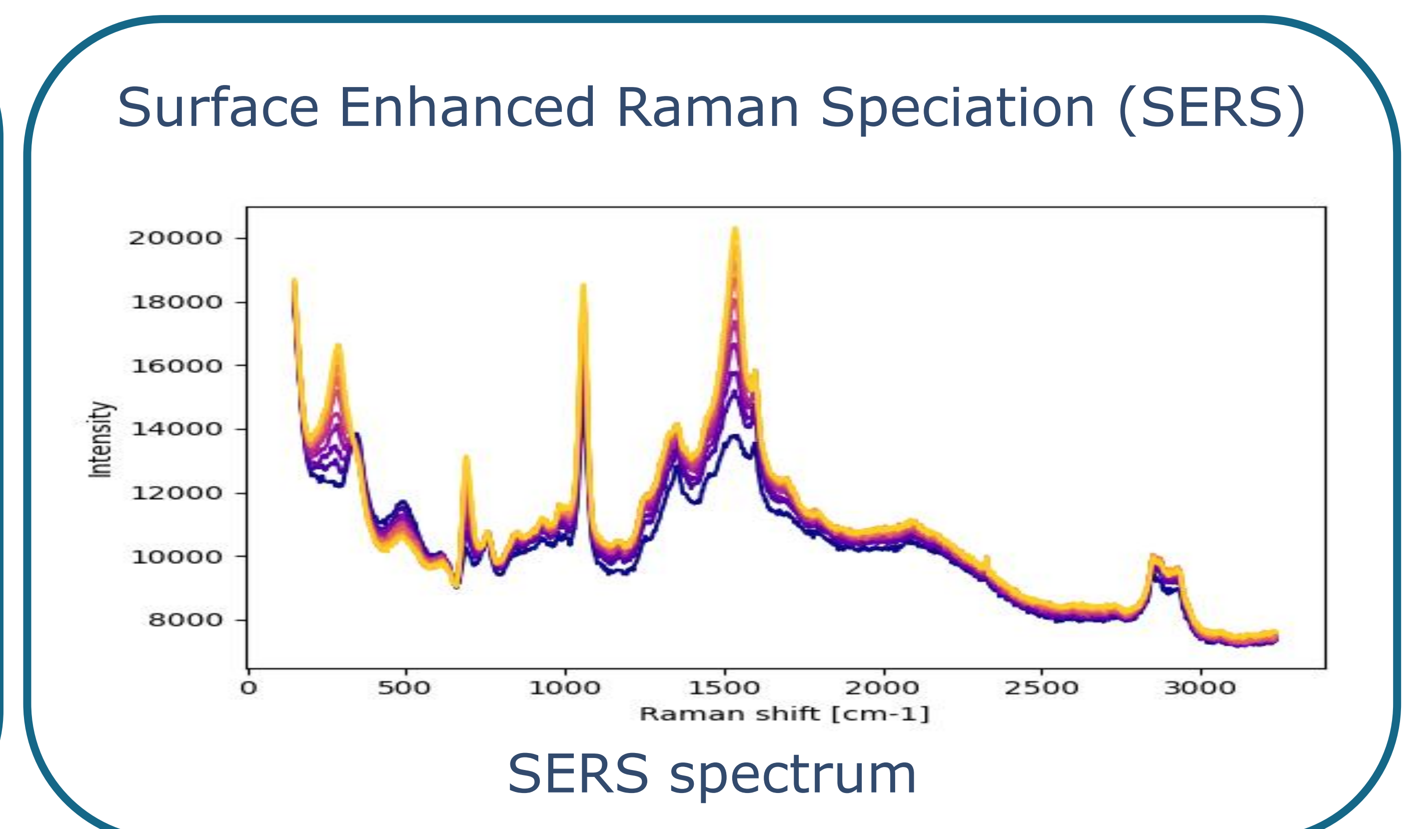
- Automatic
- Versatile
- Liquid and Wafer samples (2-12”)
- Small footprint



UNISERS Explorer G1

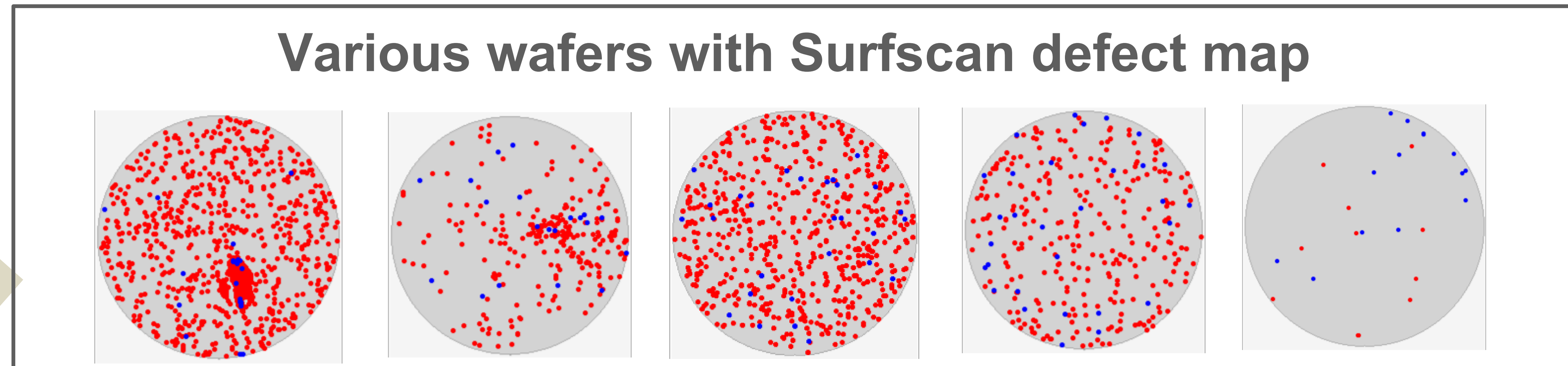
- Fab Integrated
- GEM300 compliant
- KLARF input/output
- Wafer samples (12”)
- Enhanced throughput





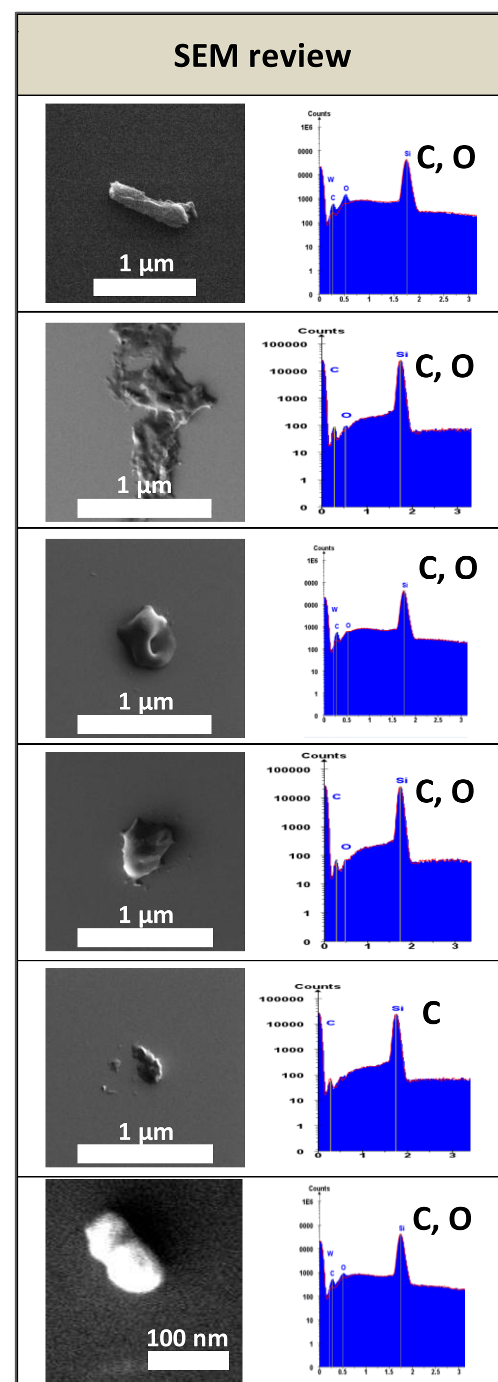
SEPS and SERS for defect review

Various wafers with Surfscan defect map



Source: IRDS, UPW task-force test

SEM defect review



173 defects reviewed.
120 invisible defect.
53 defect images.

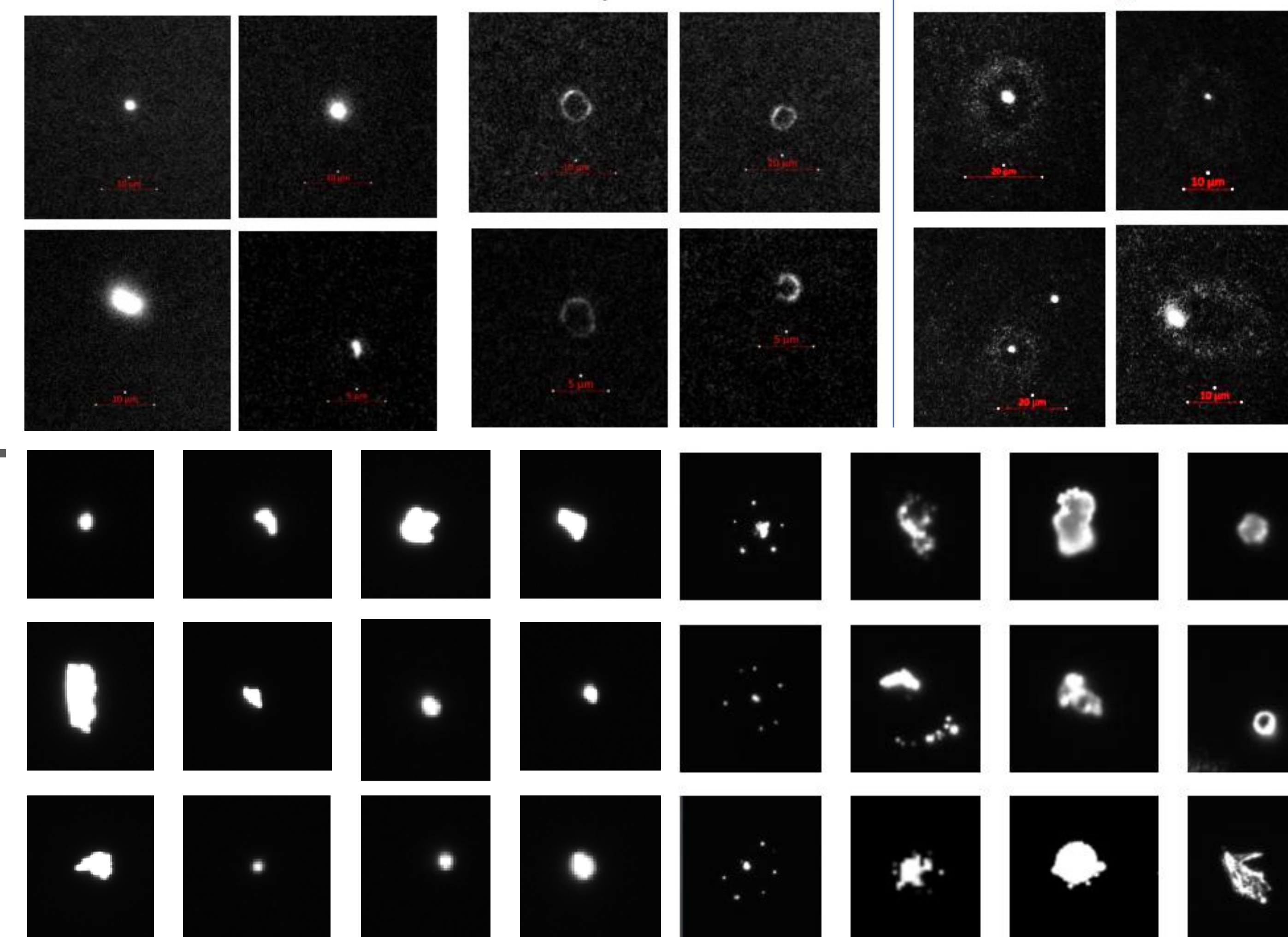
Learnings

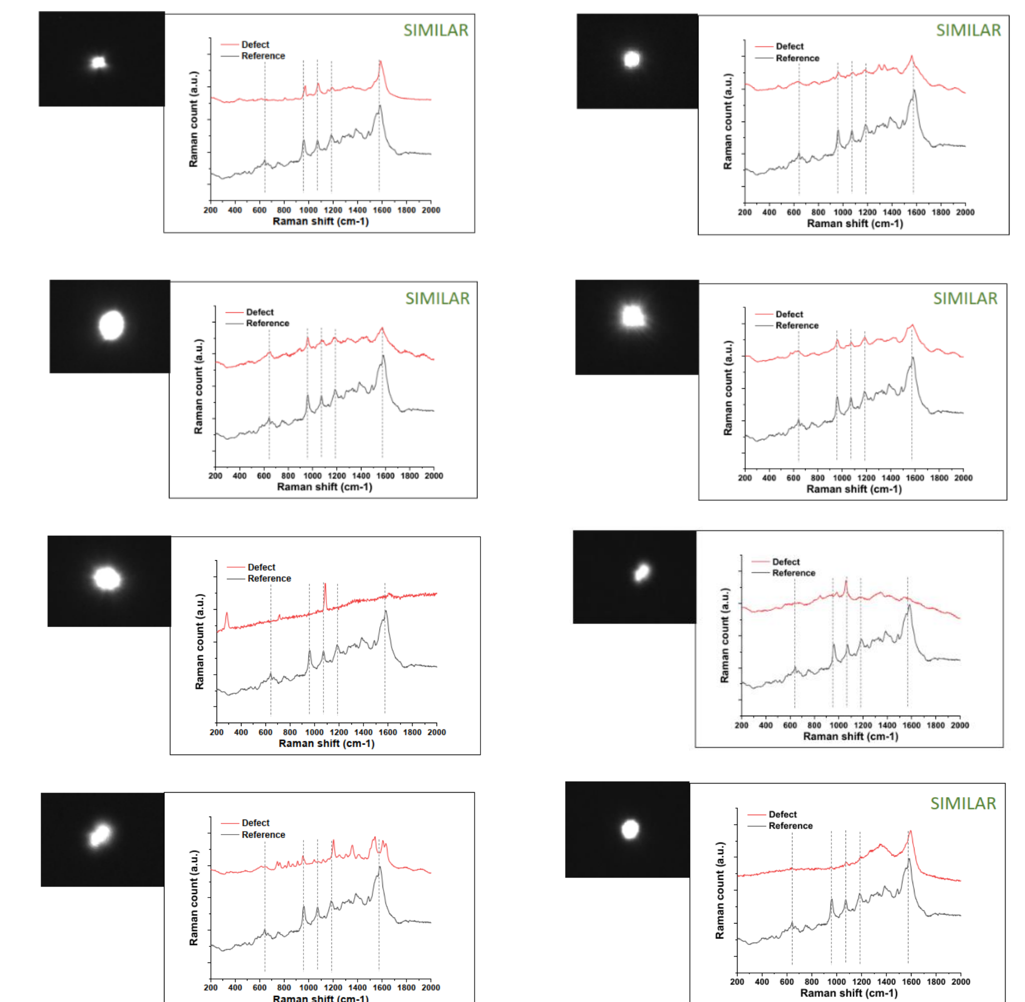
SEM images:
- Particles and agglomerations

EDX analysis:
- Carbon and oxygen

UNISERS defect review

1513 defect images.





Learnings

SEPS images:
- 96% single particles
- 1% evaporation marks
- 3% organic films

SERS analysis:
- 60% polystyrene sulfonate (ion exchange resin extract)
- 27% unspecified organics
- 13% silicates

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IRDS Collaborative Research Overview and Relevant SEMI Standards

Gary Van Schooneveld, President

CT Associates, Inc.

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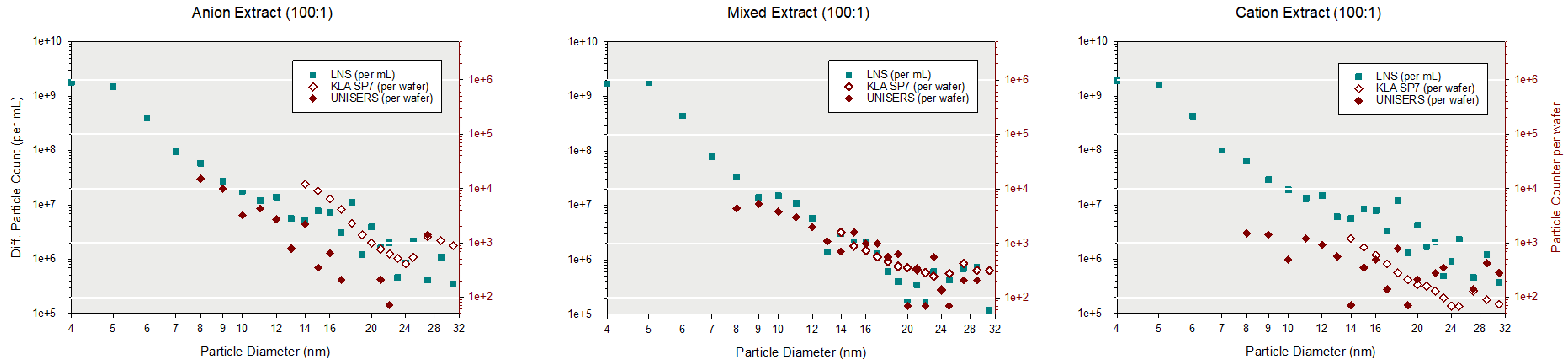
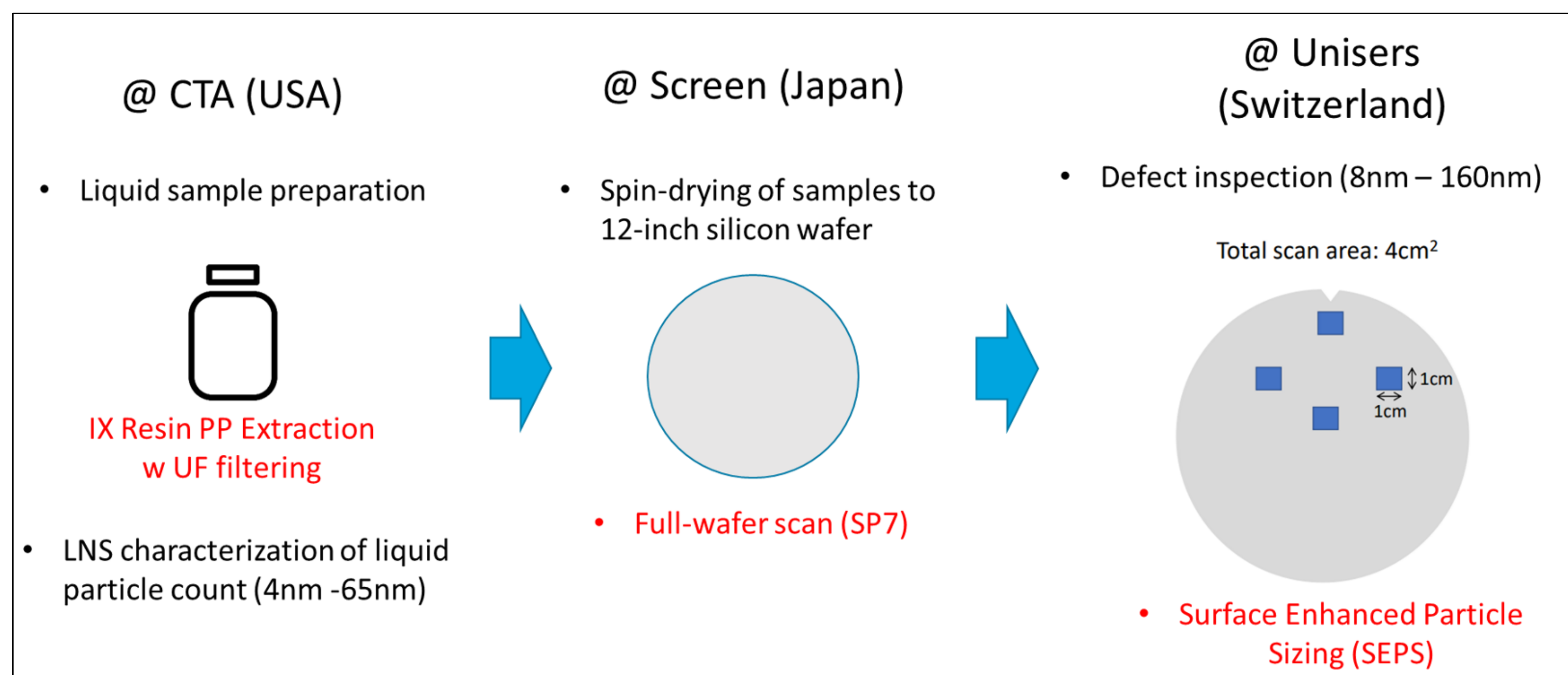
IRDS UPW 2021 Particle Detection Study





- **Early UNISERS data from 2022**
- **Proven ability to detect particles below 10nm**
- **Out-performed classical on-wafer techniques**

Reused with Permission: Presented by Gary Van Schooneveld in the UPM Community Event June 2023

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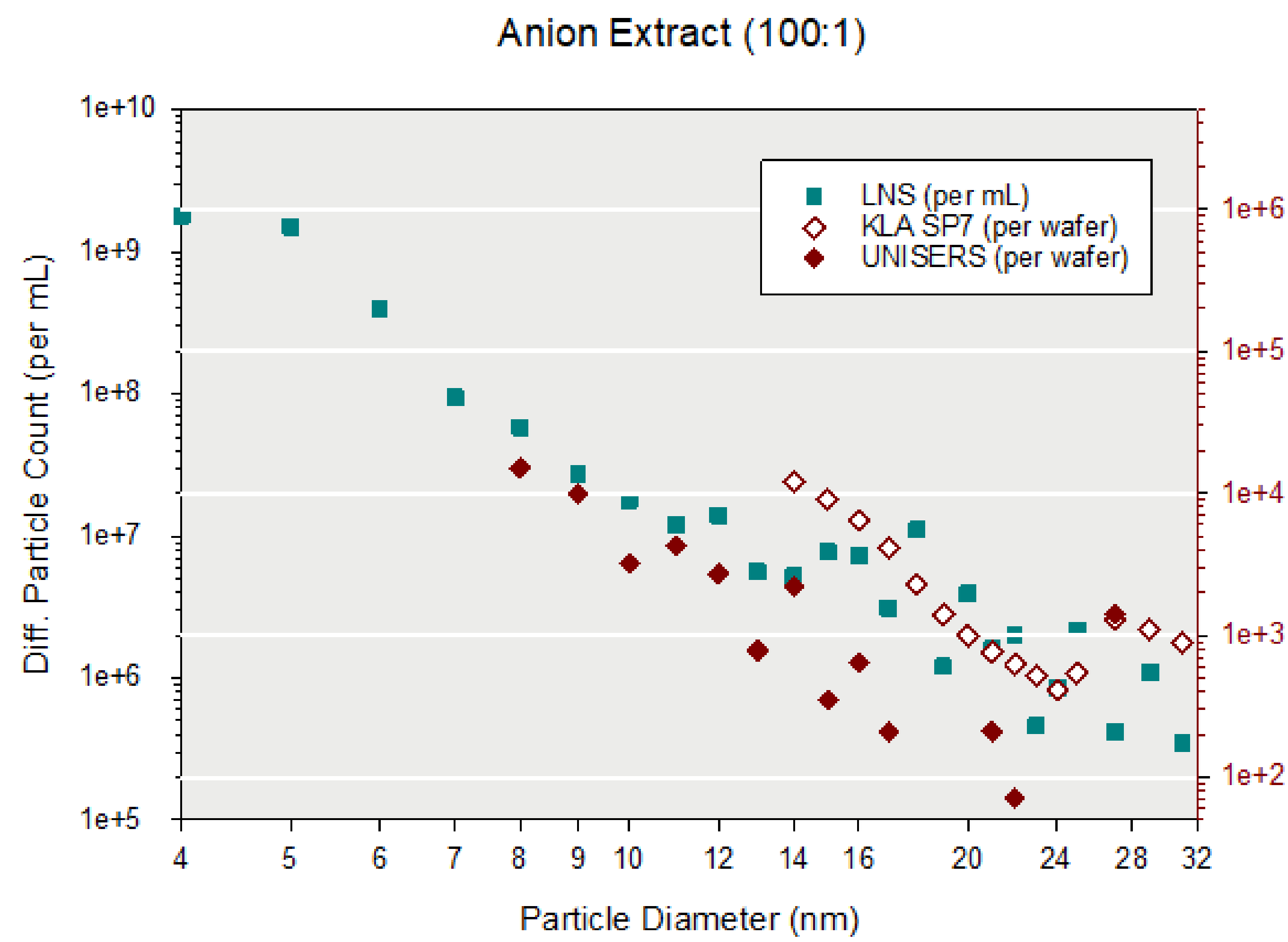
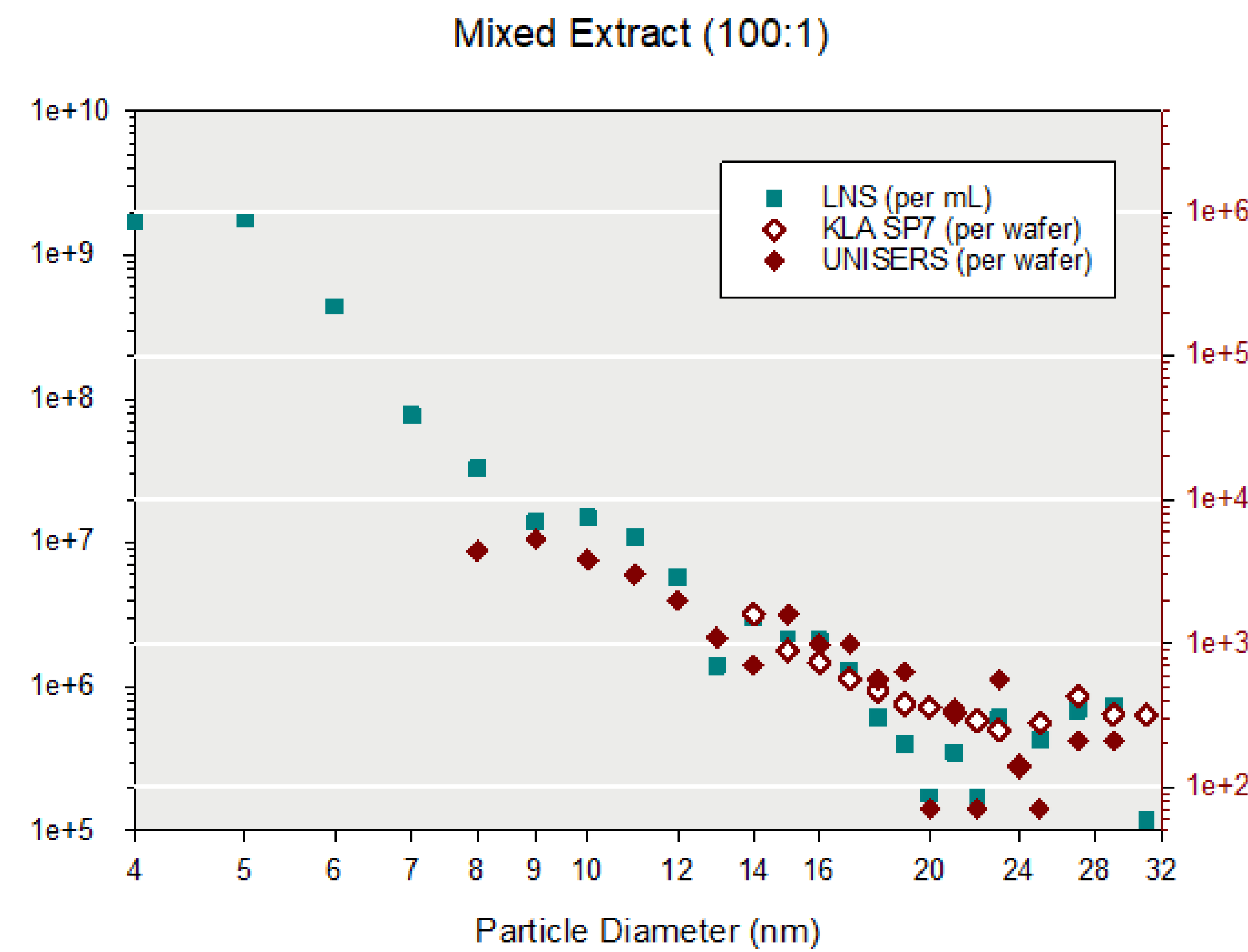
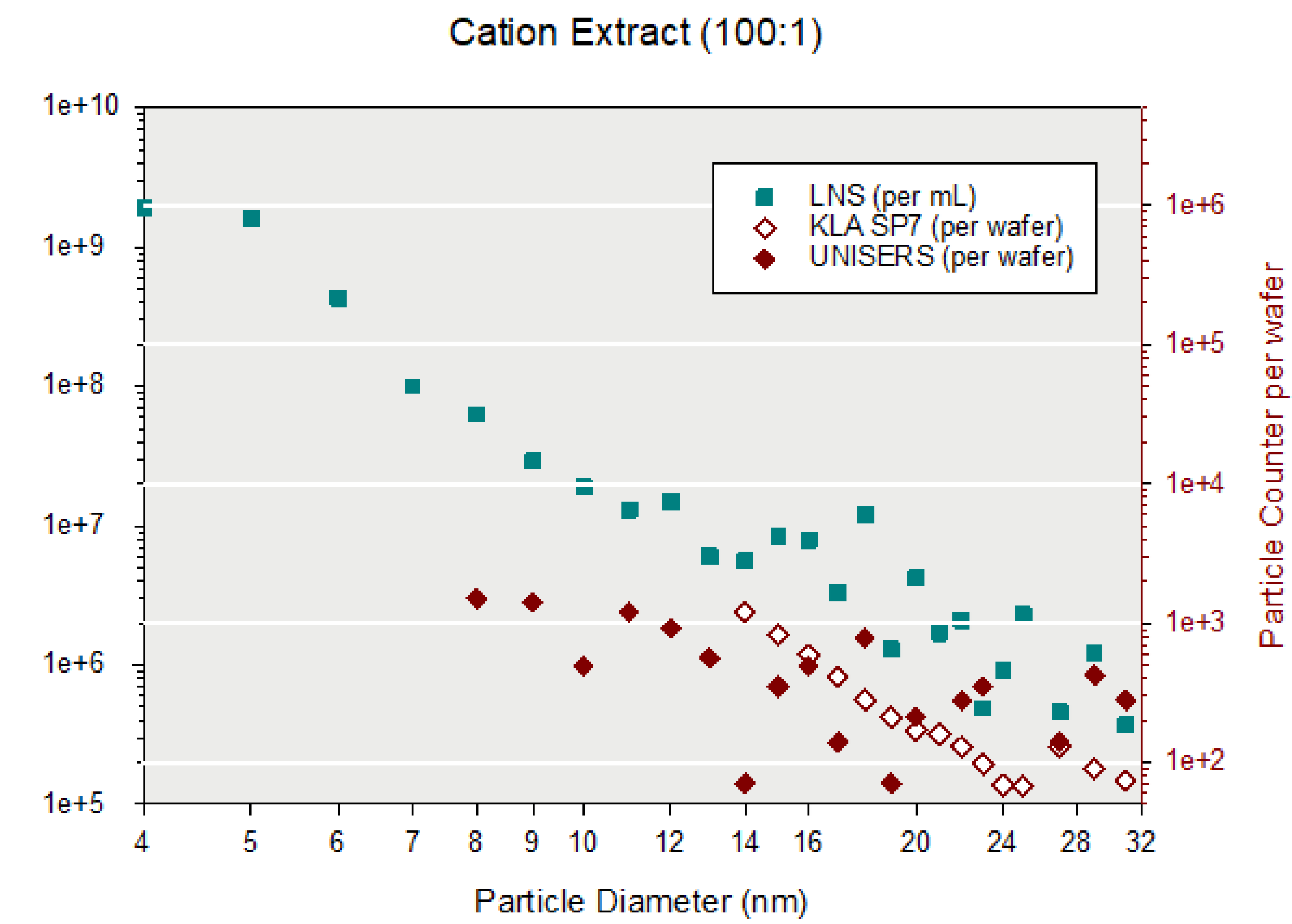
LNS to UNISERS CORRELATION – Anion, Cation, Mixed resin

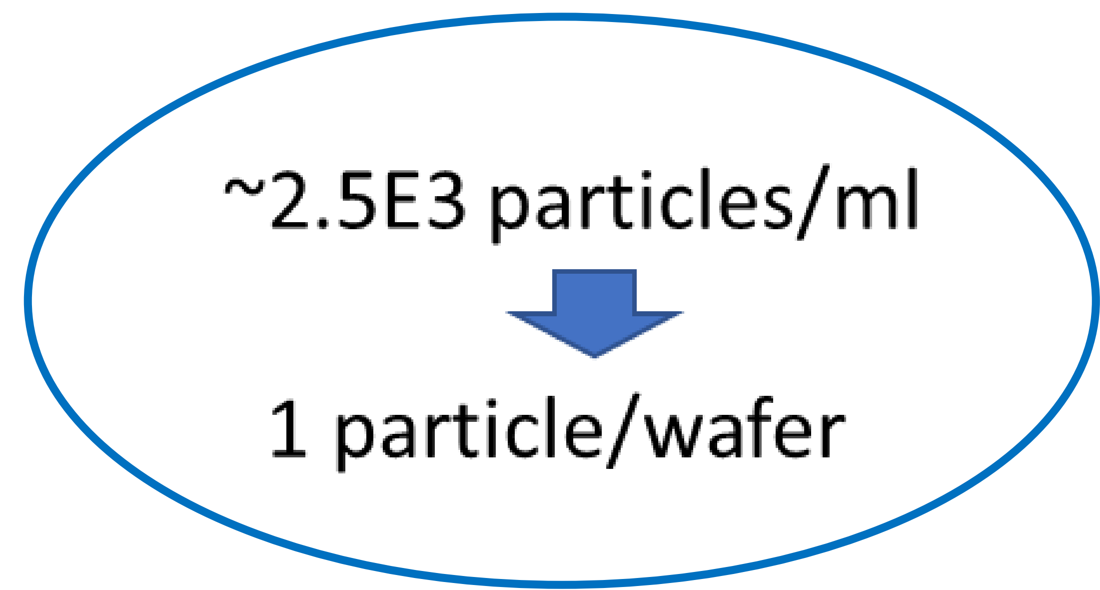






~ 4E3 particles/ml

1 particle/wafer



~2.5E3 particles/ml

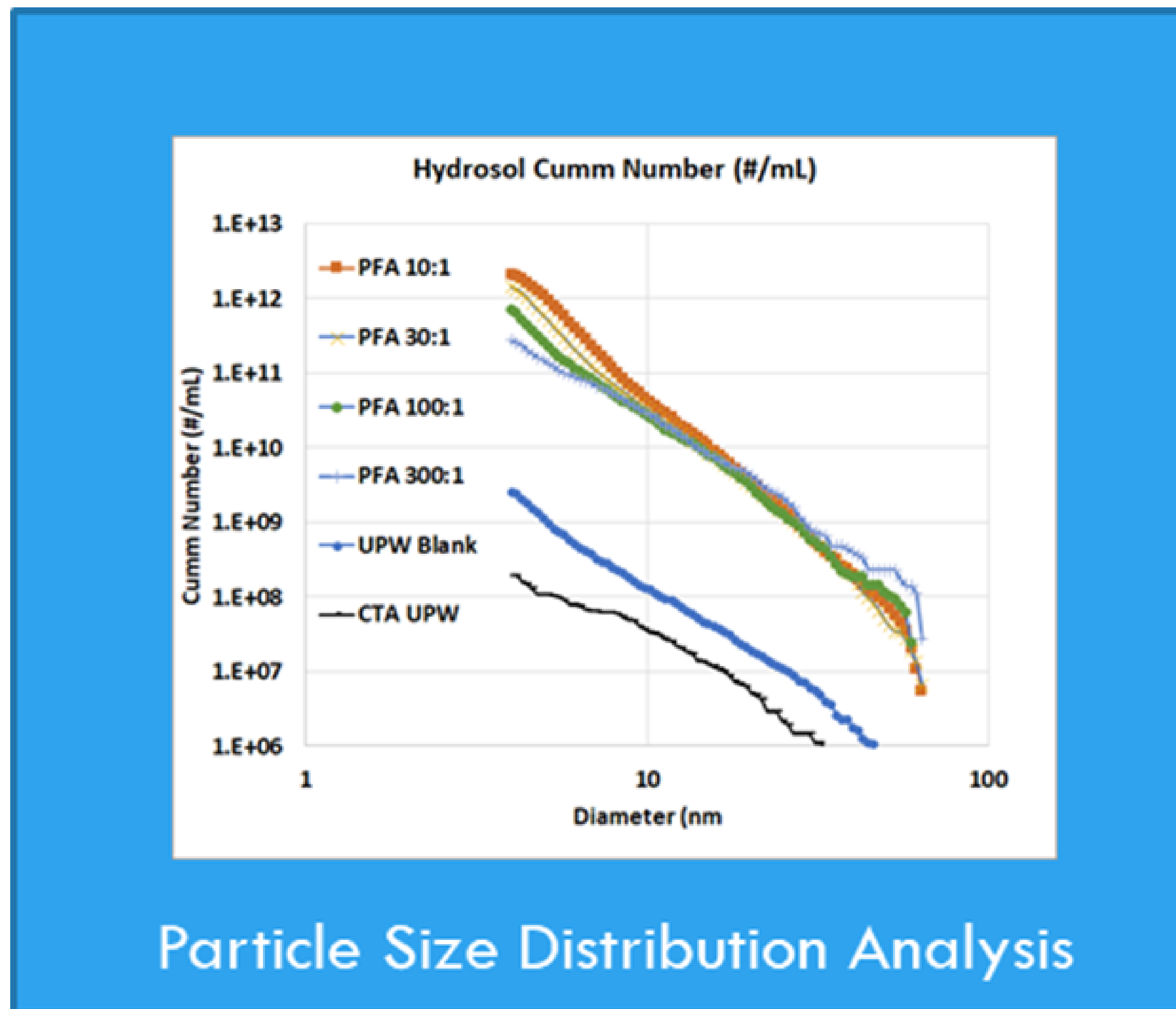
1 particle/wafer

~ 2E4 particles/ml

1 particle/wafer

Used to establish the particle precursor specification in the 2022 IRDS Roadmap

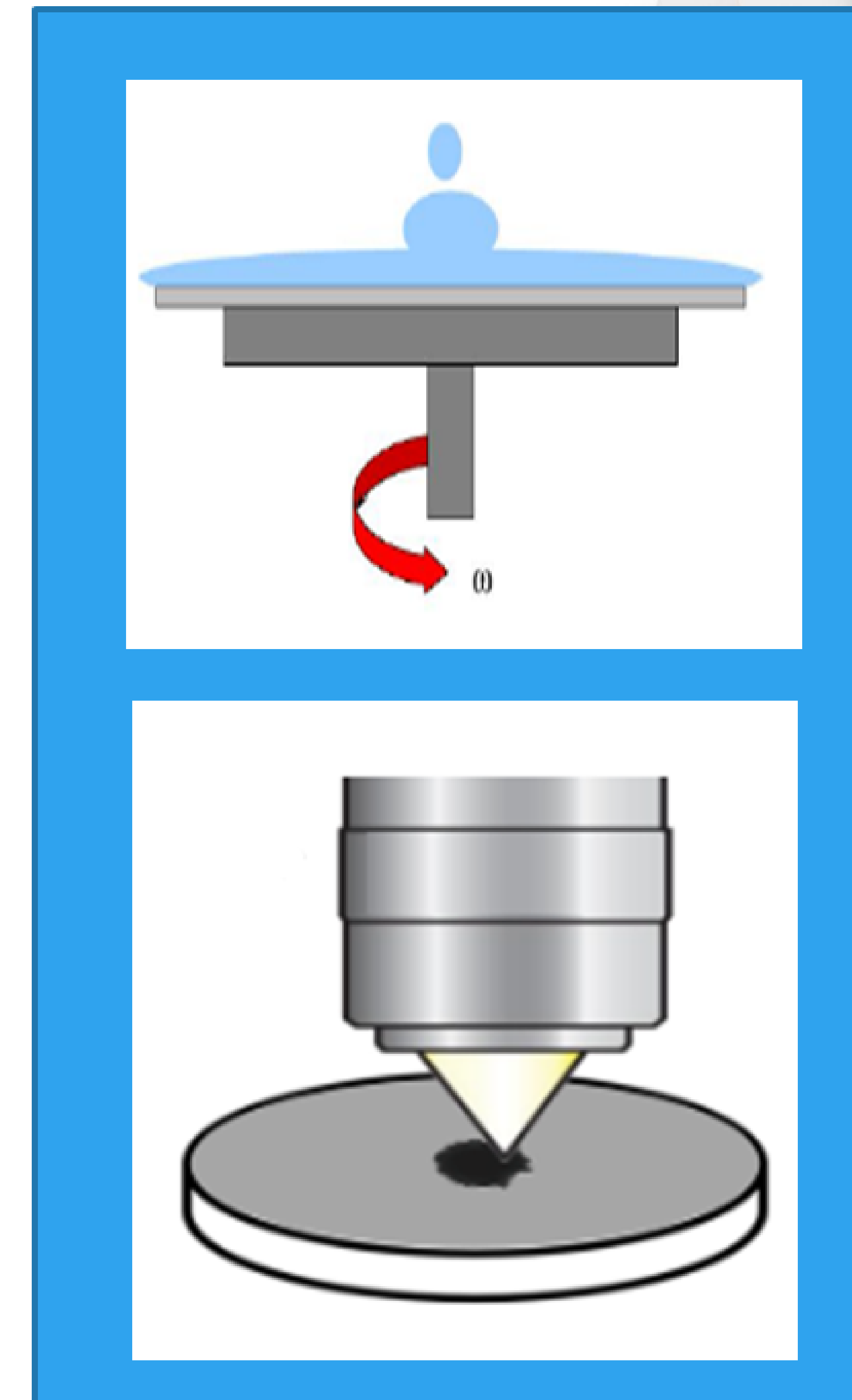
IRDS CC Approach for Determining On-wafer Particle Defect Risk from Native Particles and Particle Precursors



Particle Size Distribution Analysis

In-liquid native particle and particle precursor concentrations

Particle Defectivity Assessment



Sequential Spin Coating (SSC) - Surface Enhance Particle Sizing (SEPS)

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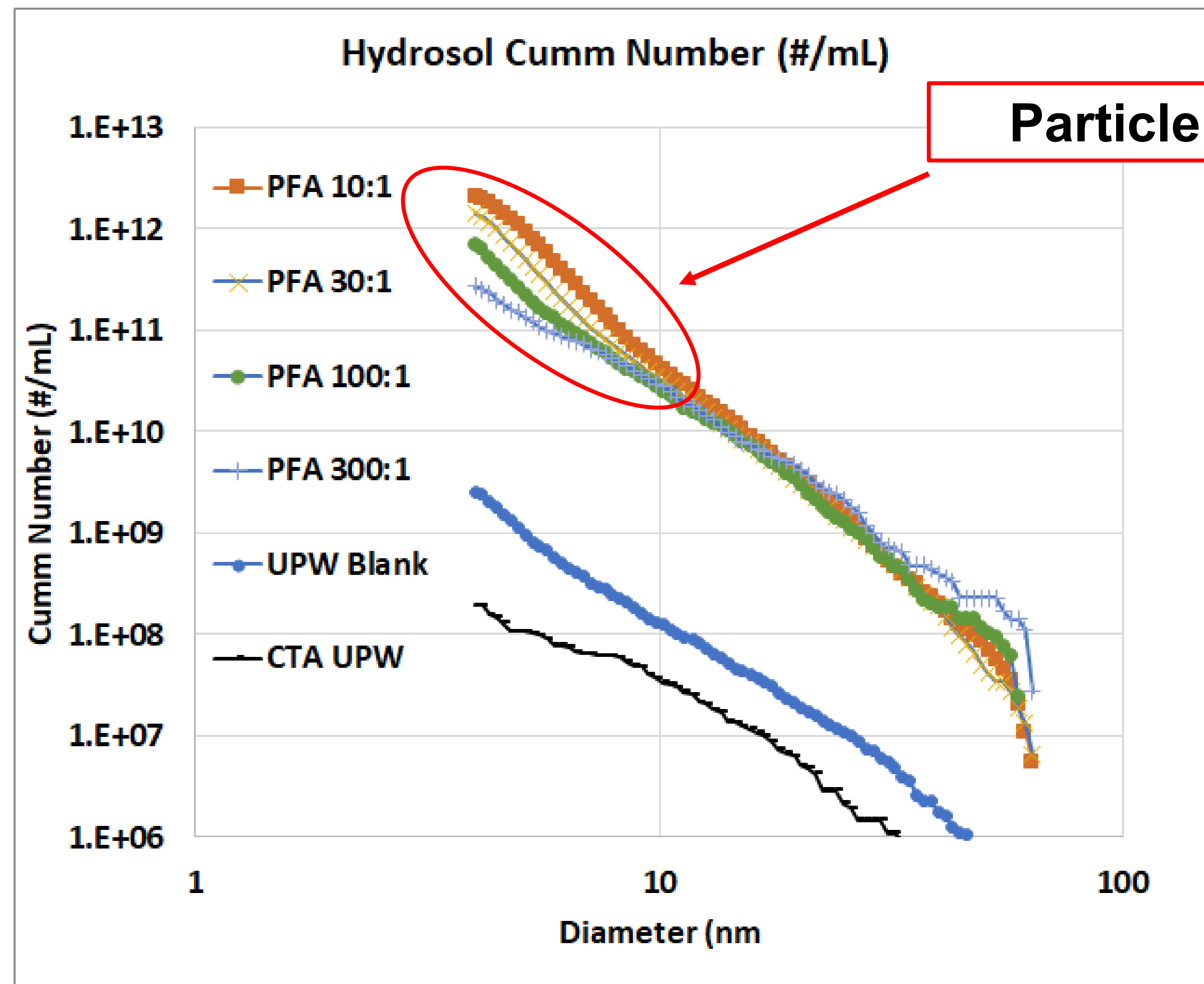
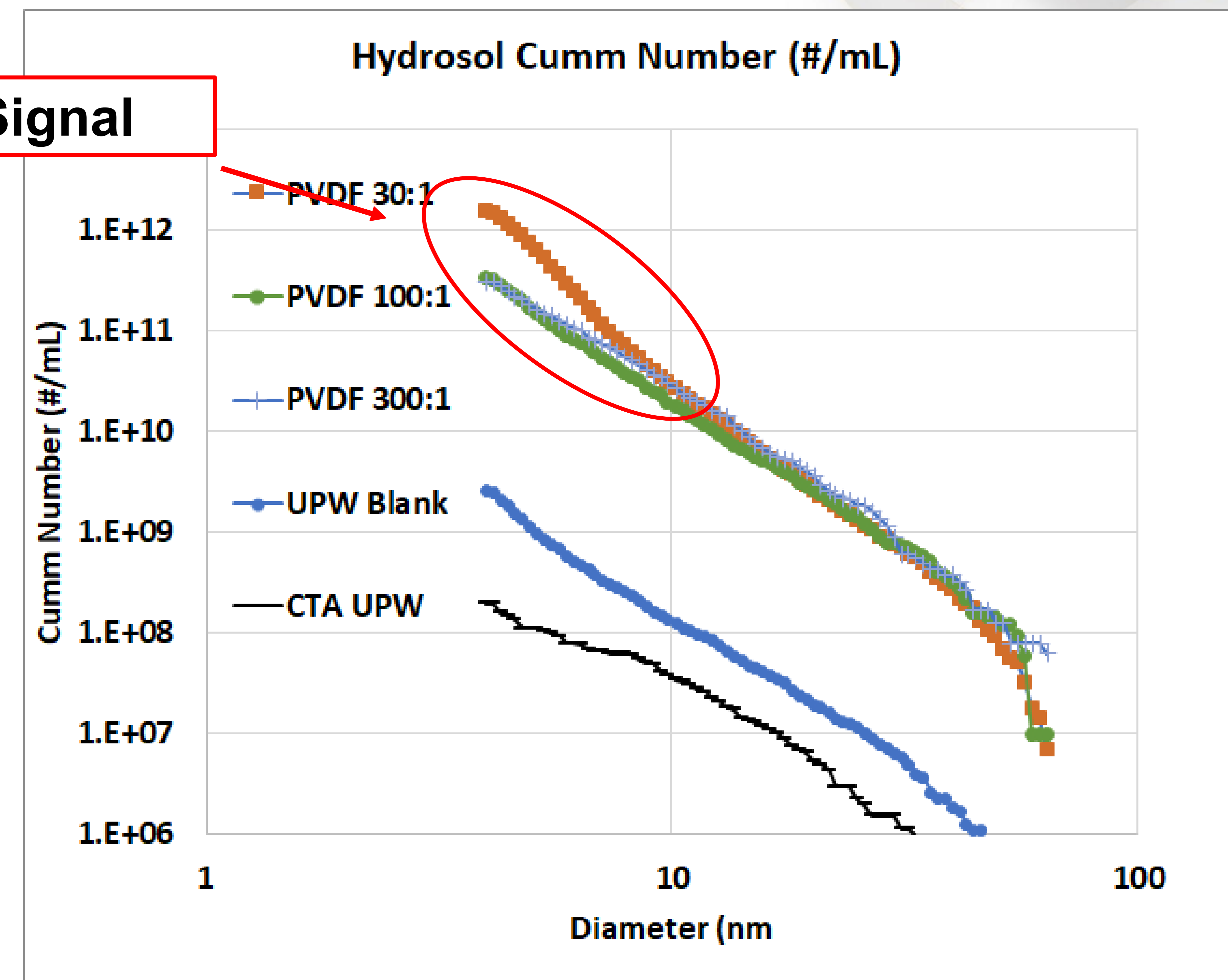
Particle and Particle Precursor Analysis of PFA and PVDF Extract, 14 Day at 85˚C LNS (Dilution Normalized)

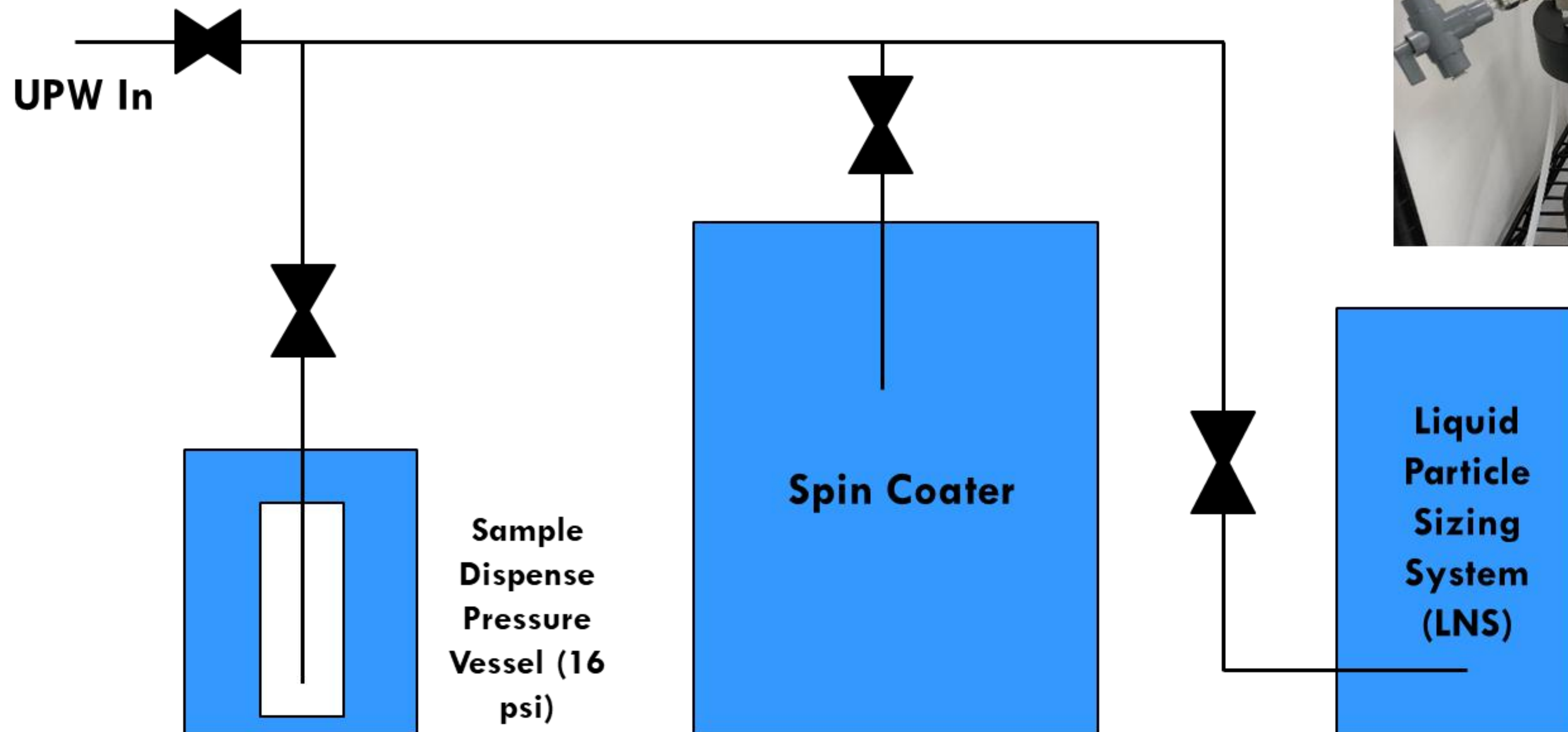


Particle Precursor Signal



Source: Van Schooneveld, et al, Ultrapure Micro 2022

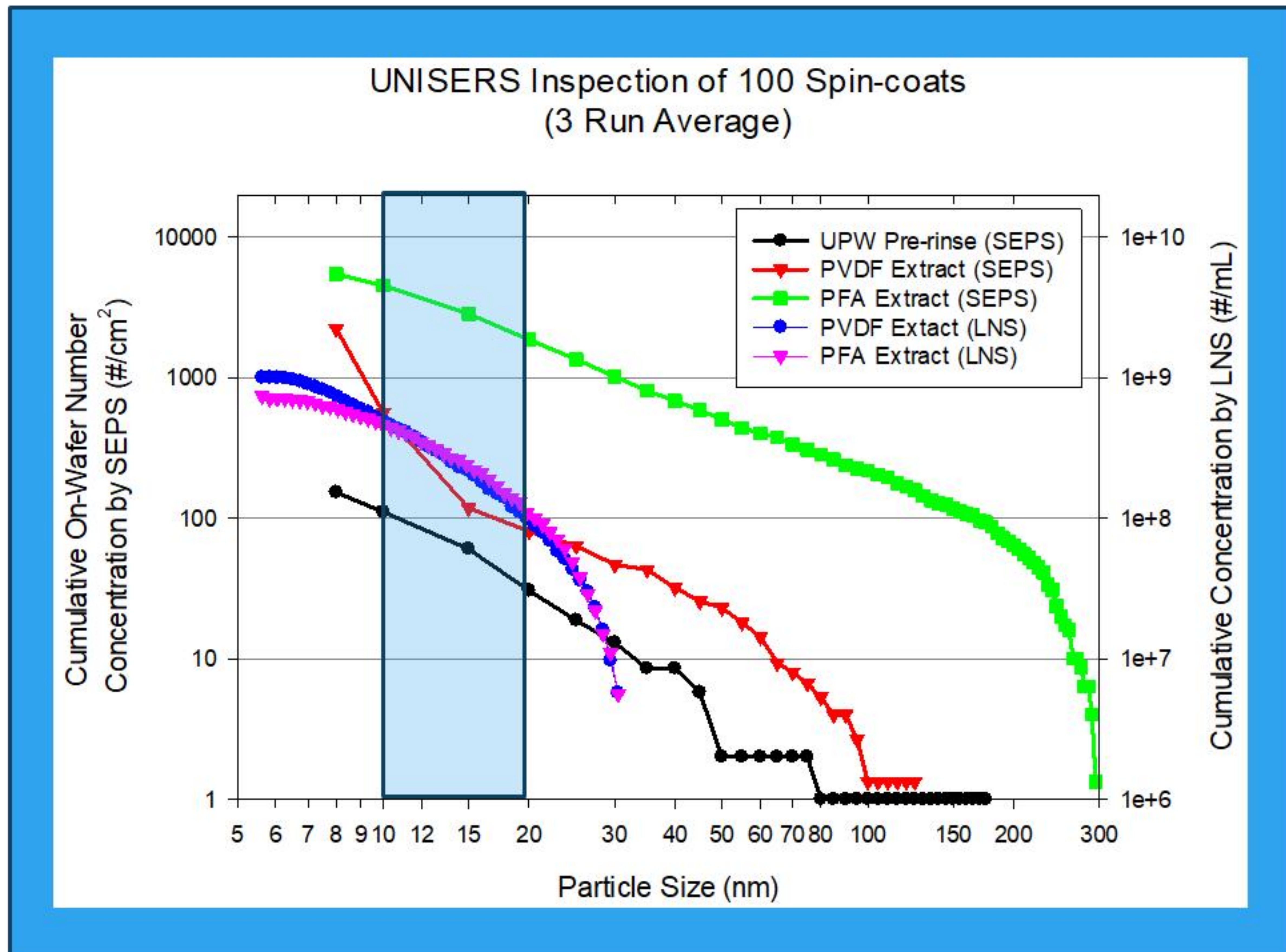
Test Schematic – Sequential Spin Coating



Equipment List:

- Laurell Model WS-650MZ
- KFMT Model 9110 Nebulizer
- KFMT Model 3560 Classifier
- KFMT Model 3650 CPC

Approach #1- Correlation Between SEPS and LNS PSD by Differential Analysis



UNISERS Lab Suite at CTA

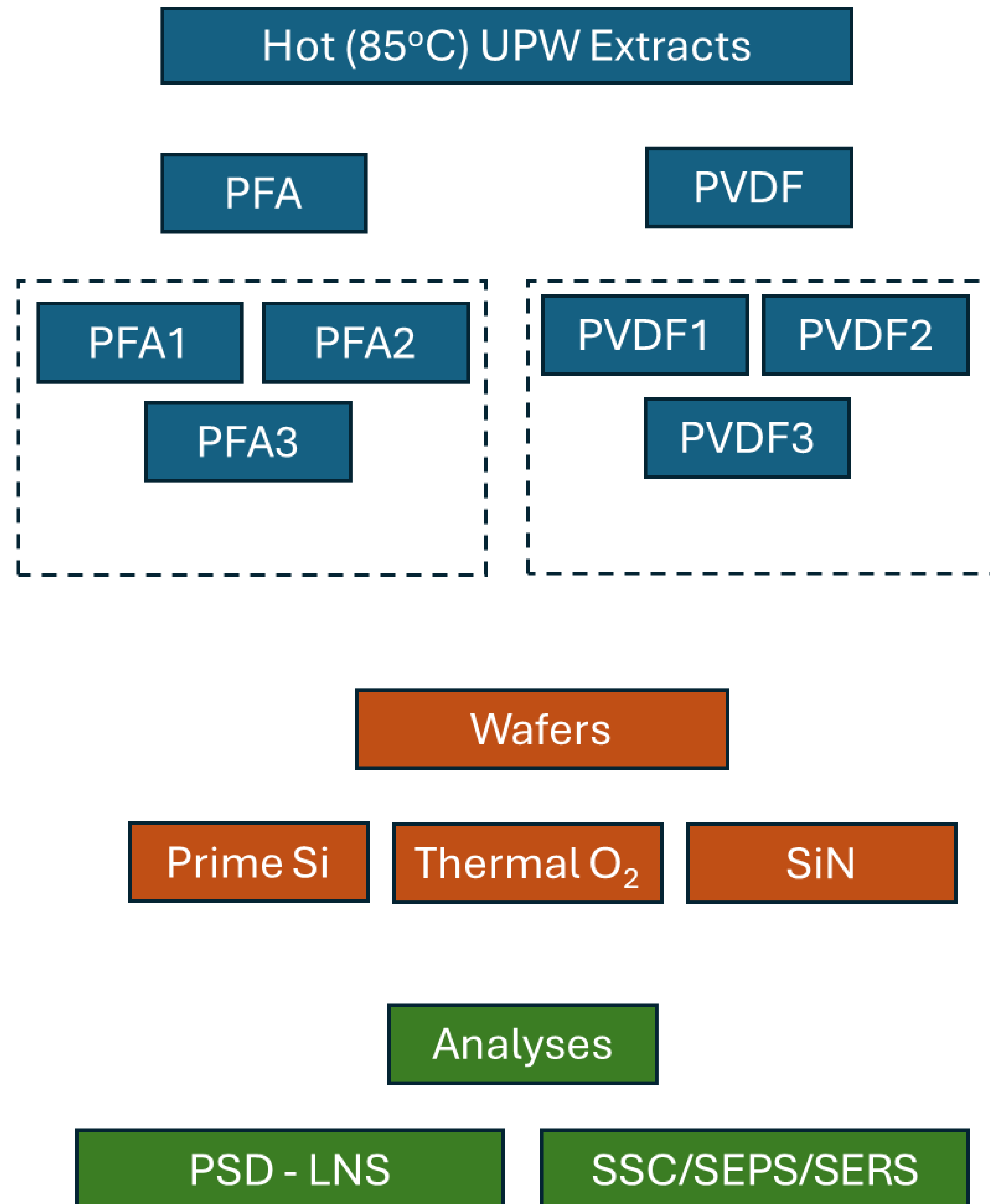
$$\text{Defectivity Coefficient} = \frac{\Delta\,(LNS\ C_{20nm}\ \ LNS\ C_{10nm})}{\Delta\,(SEPs\ C_{20nm}\ _SEPS\ C_{10nm})/100)}$$

Defectivity Coefficients per Coat (10 – 20 nm):*

- PFA – 1.4E7 #/mL per particle/wafer-cm^2
- PVDF – 8.3E7 #/mL per particle/wafer-cm^2

- Not normalize for extraction surface area

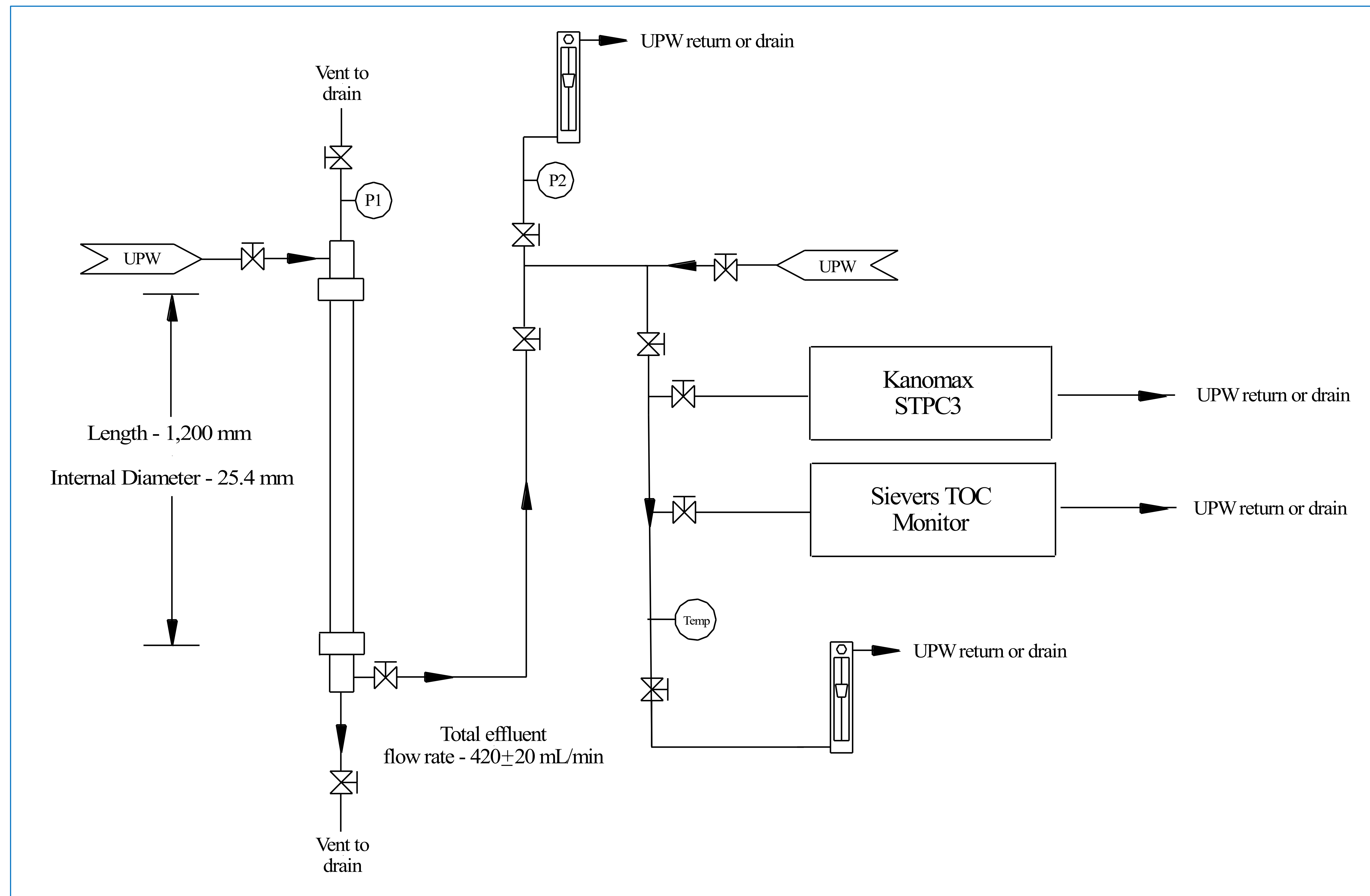
IRDS Critical Components – Pre-SEMI Test Method Development (in process)



Test Objectives:

- **Assess impact of spin coating approach (fixed speed vs ramp to dry) of defectivity coefficient.**
- **Determine optimum number of spin-coats (sensitivity vs size).**
- **Quantify differences in defectivity between wafer surfaces.**
- **Demonstrate repeatability and sensitivity of the method using multiple extracts.**

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



- **Multi-day testing used to simulate initial ion exchange resin installation and rinse-down.**
- **TOC rinse measured continuously with TOC monitor.**
- **Particle and particle precursor rinse down measured continuously with STPC3**

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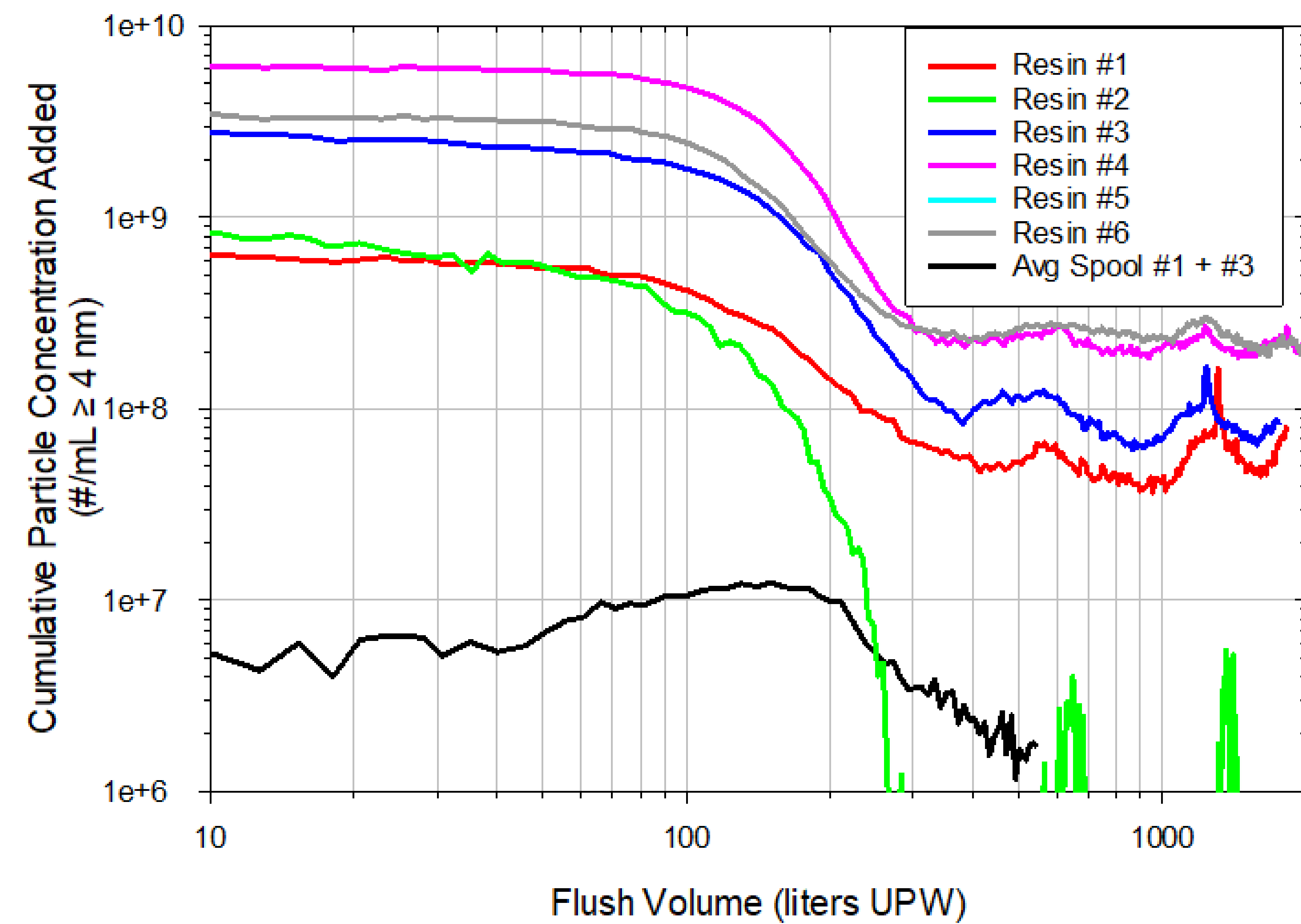
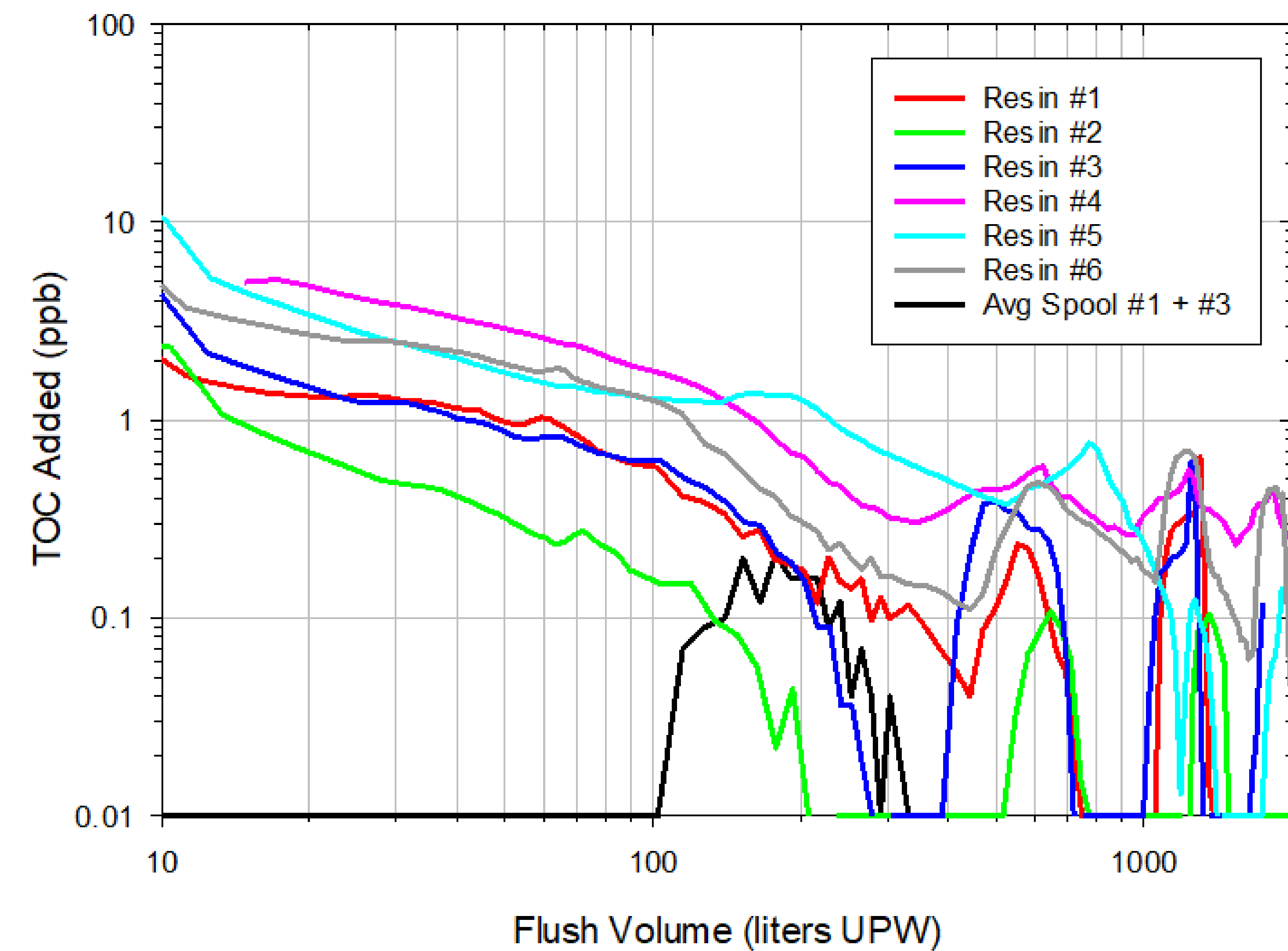
SEMI C93 Dynamic Rinse Test Results

Particle and Particle Precursor Rinse Curves

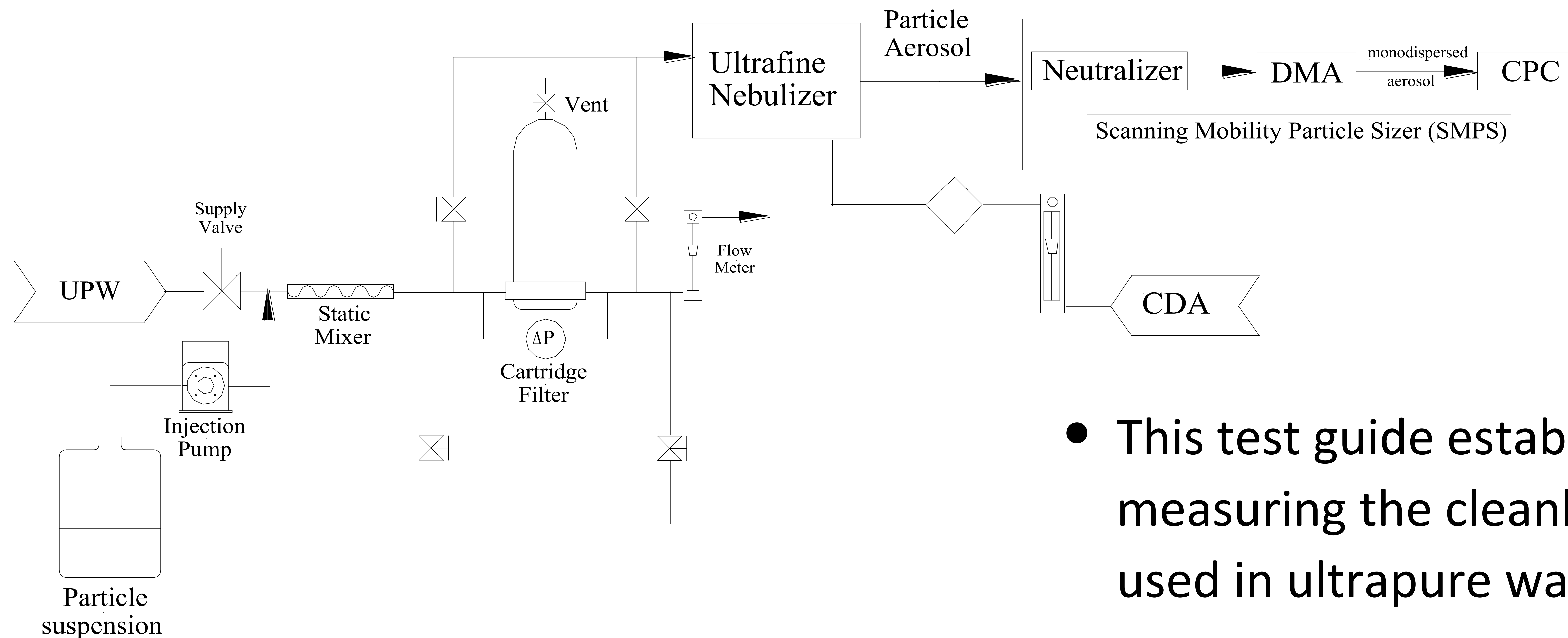


TOC Rinse Curves



Source – SEMI UPW C93 Task Force - 2019

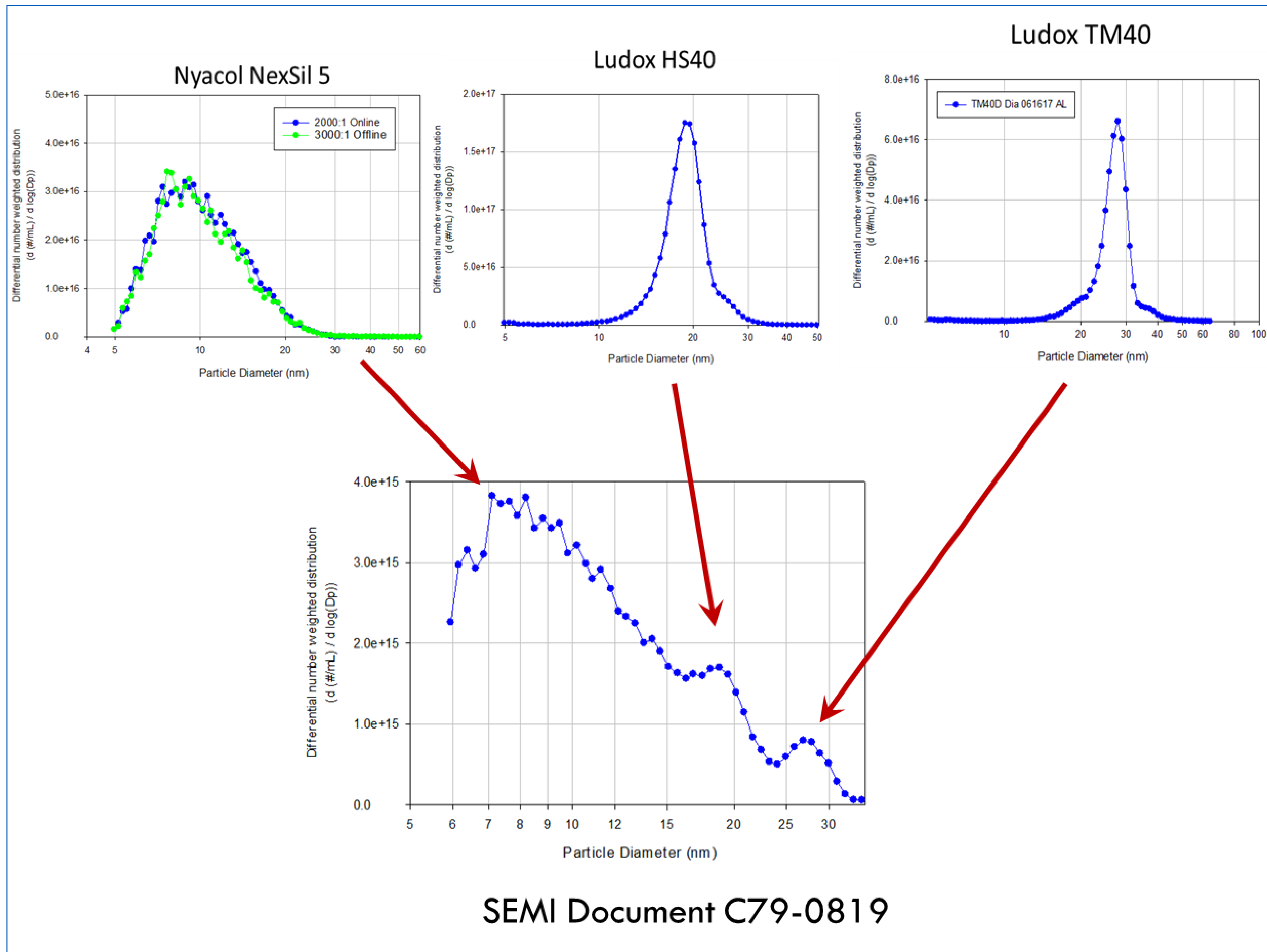
SEMI C79 - Guide to Evaluate the Efficacy of Sub-15 nm Filters Used in Ultrapure Water (UPW) Distribution Systems



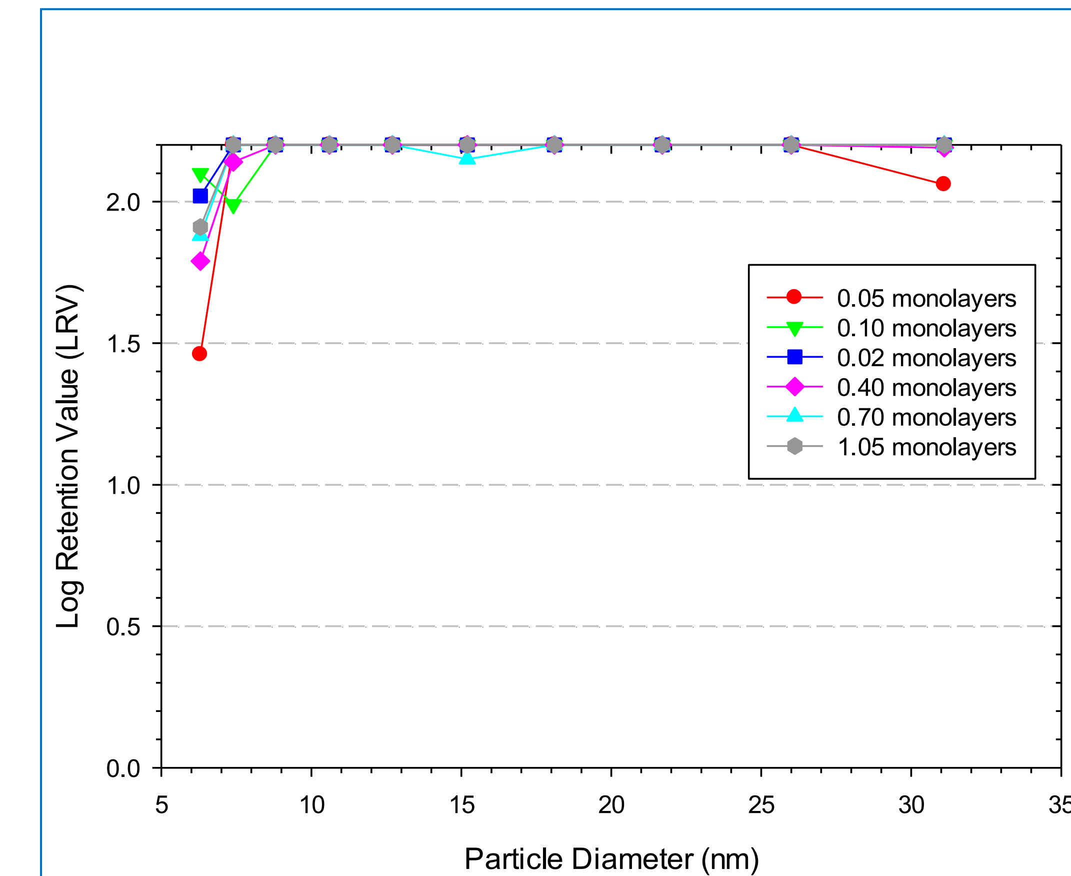
Utilizes Nebulizer-based SCTP3 and LNS for particle measurements

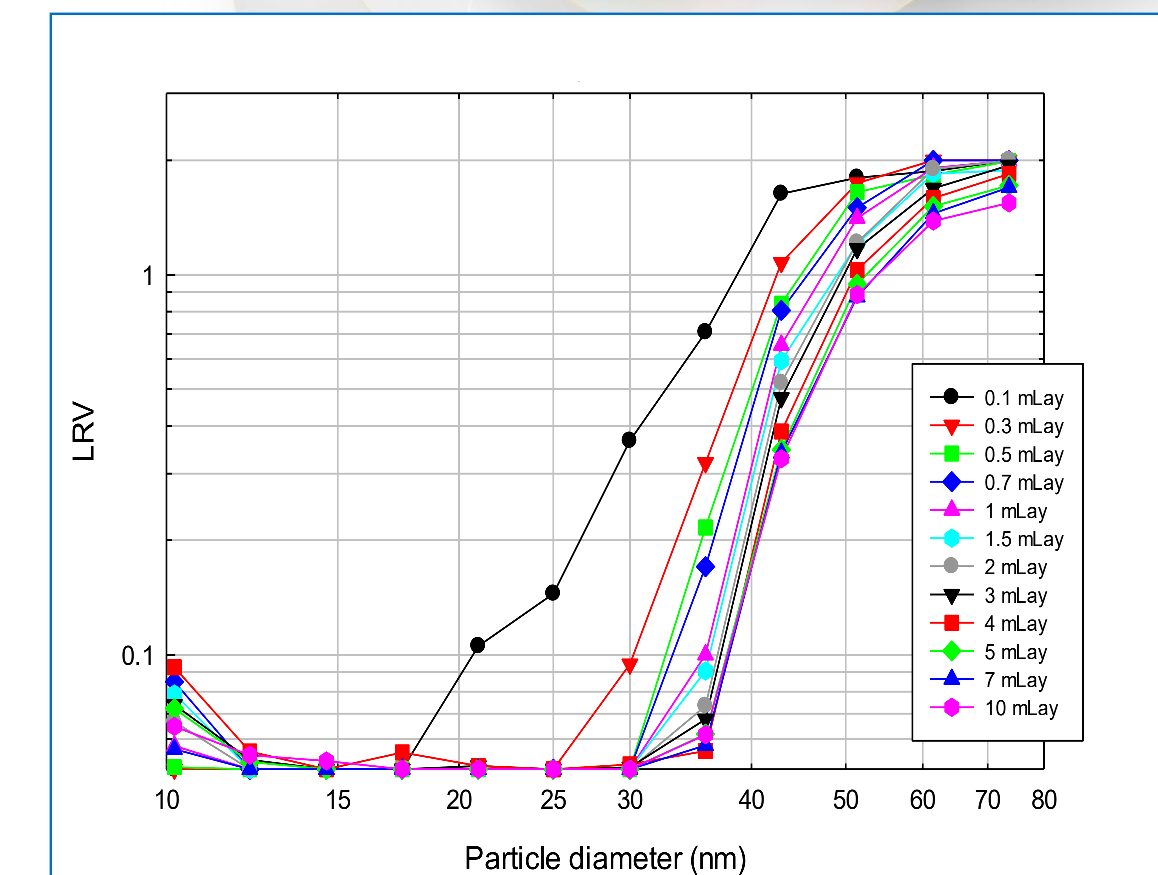
- This test guide establish the test method for measuring the cleanliness and retention for filters used in ultrapure water applications.
- Flush times for particles, particle precursors and TOC are measure on the filters as installed.
- Retention capability of the filter is measured using a colloidal silica with particle sizes ranging from 6 to 35 nm.

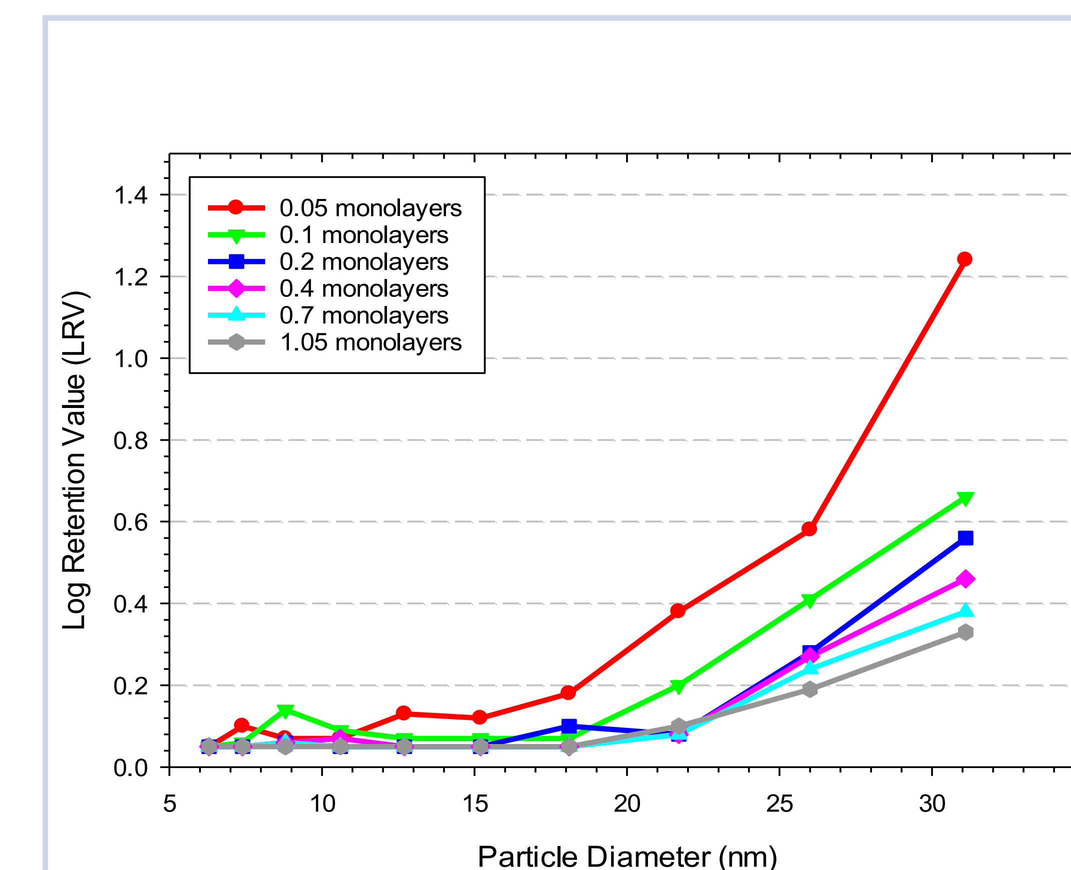
Particle size detection sensitivity reveals significant differences in retention mechanisms in filters

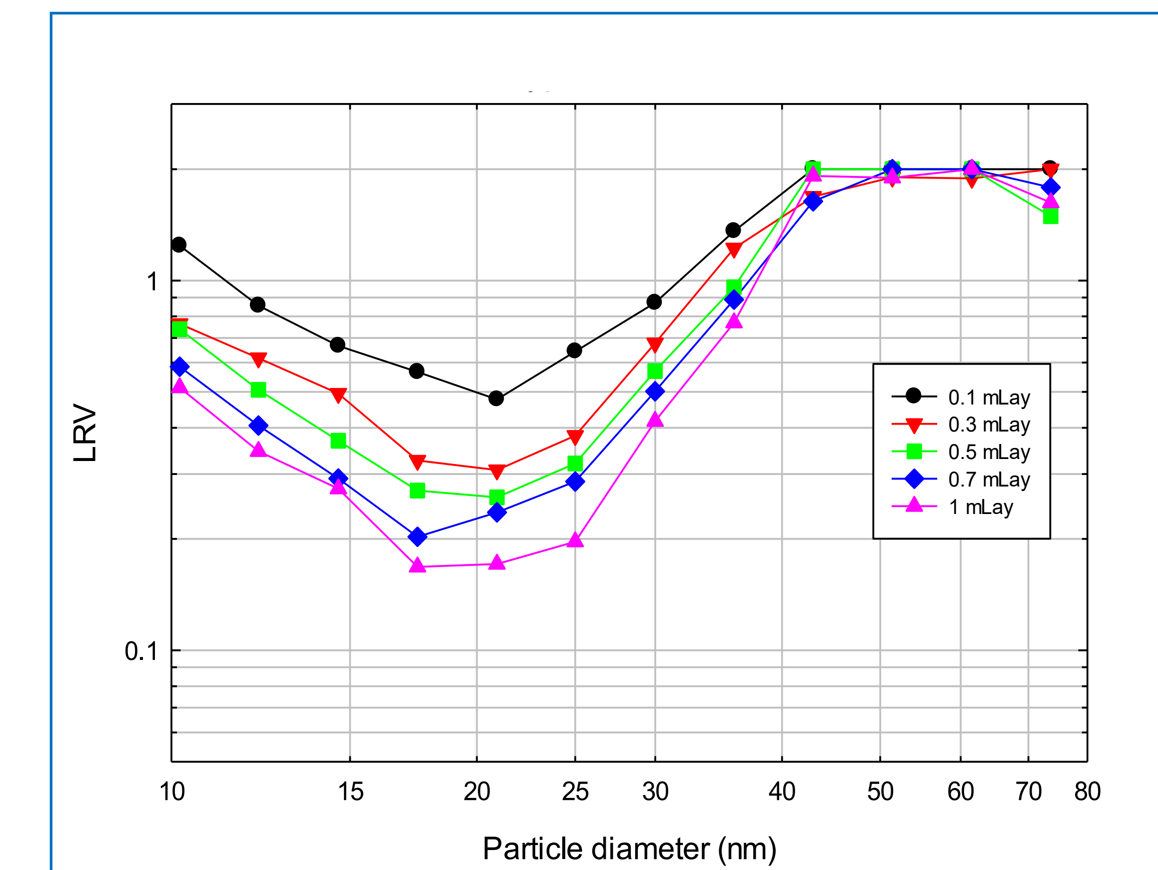


SEMI Document C79-0819

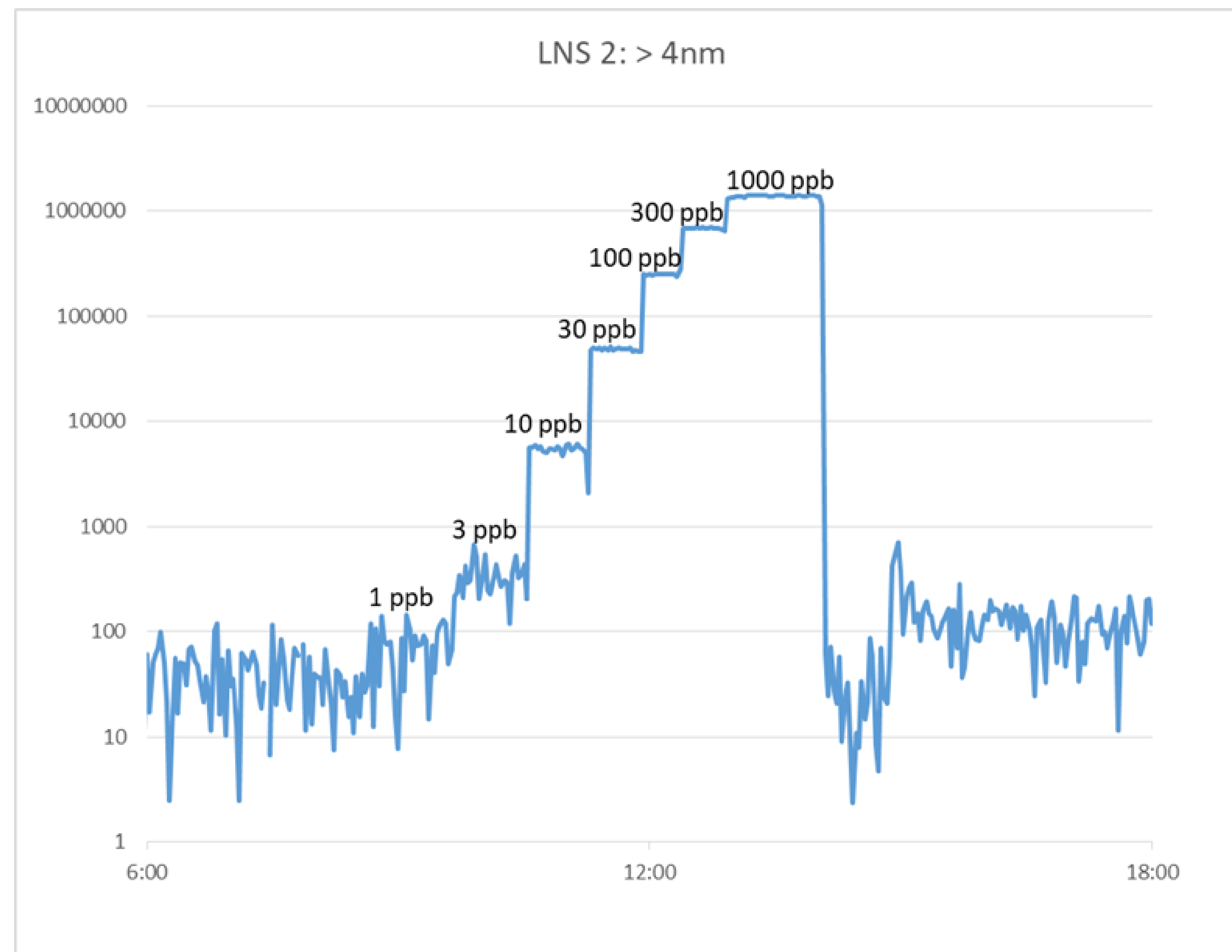


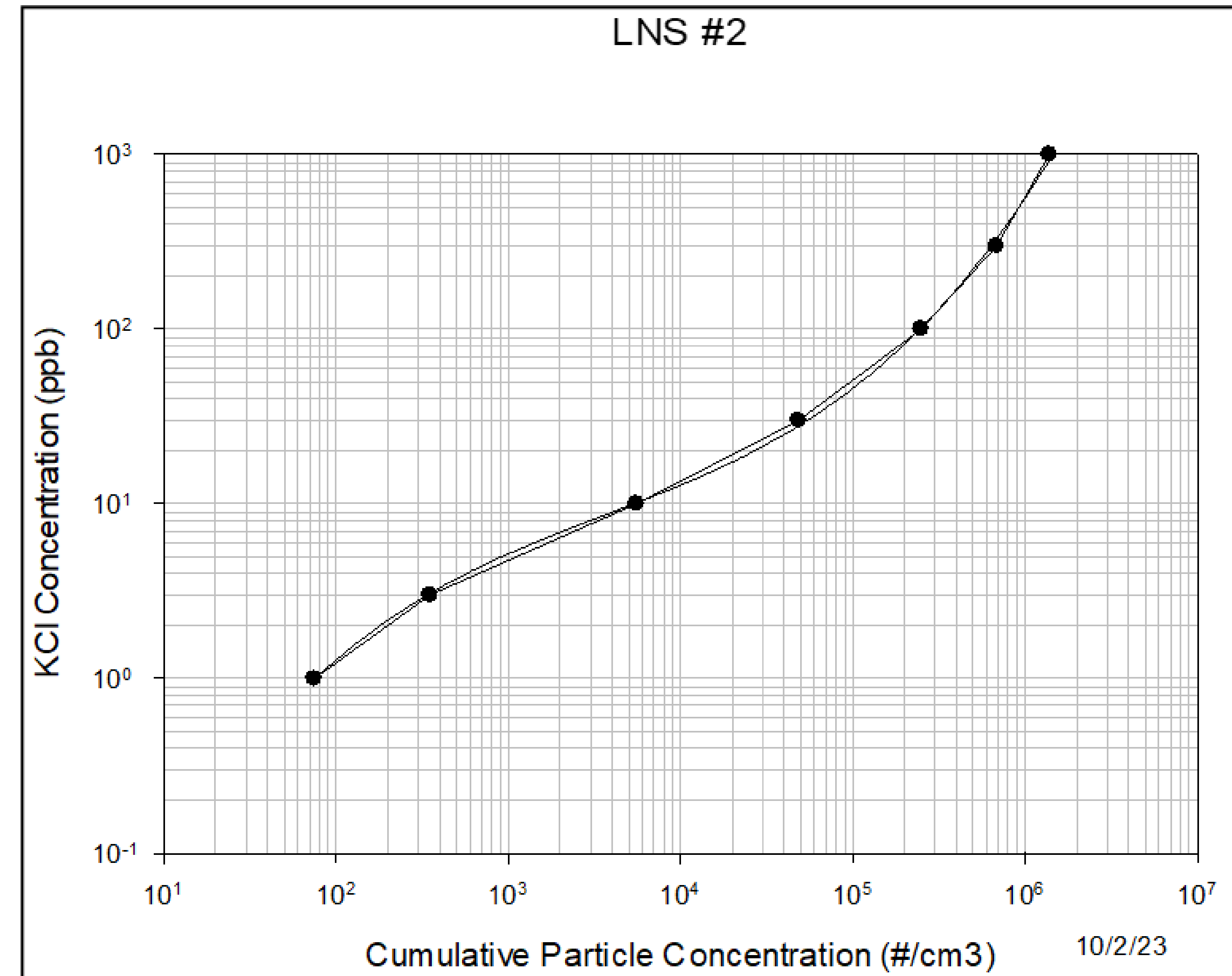






Calibration Curves for SEMI C79 PP Method Development per SEMI F121



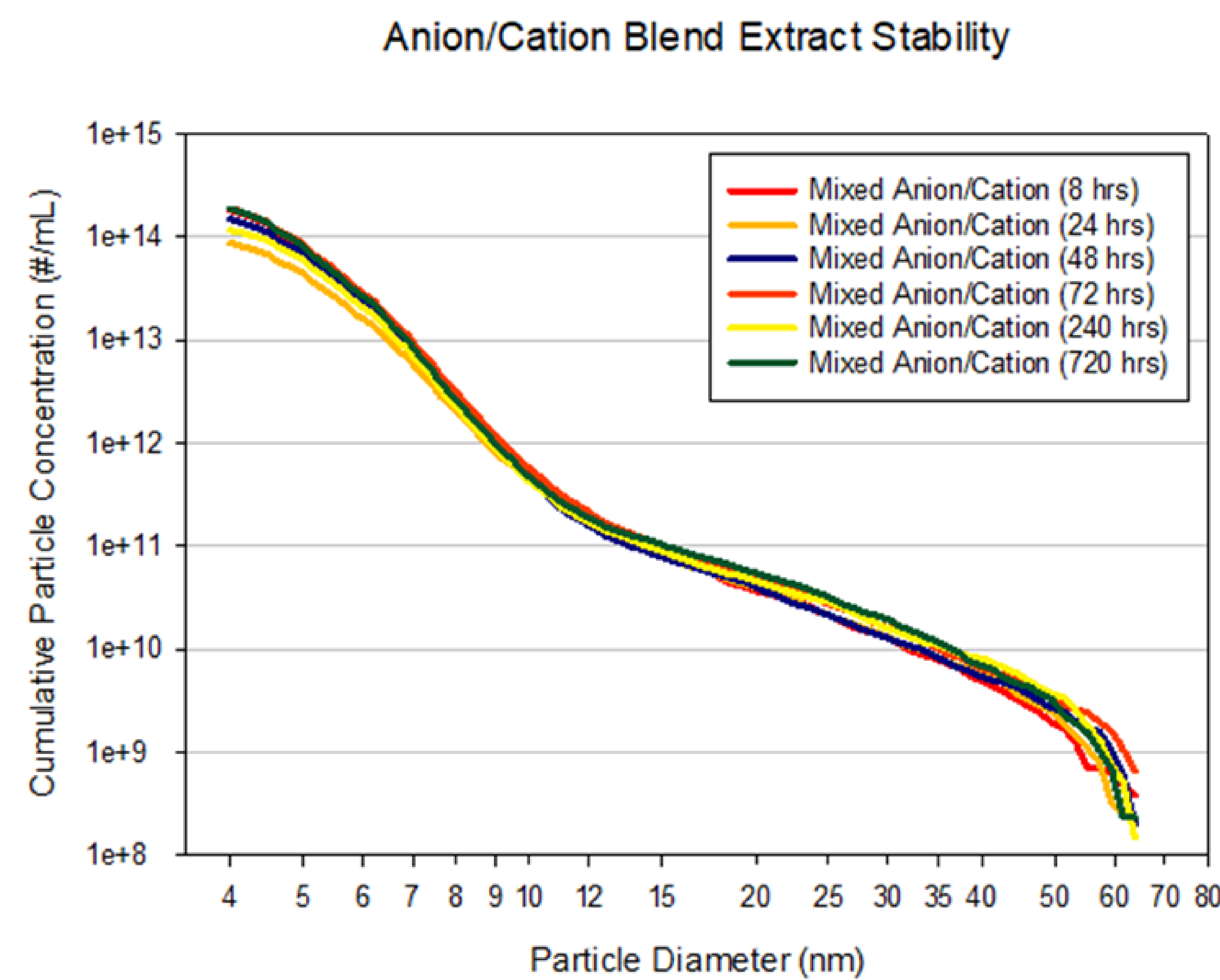


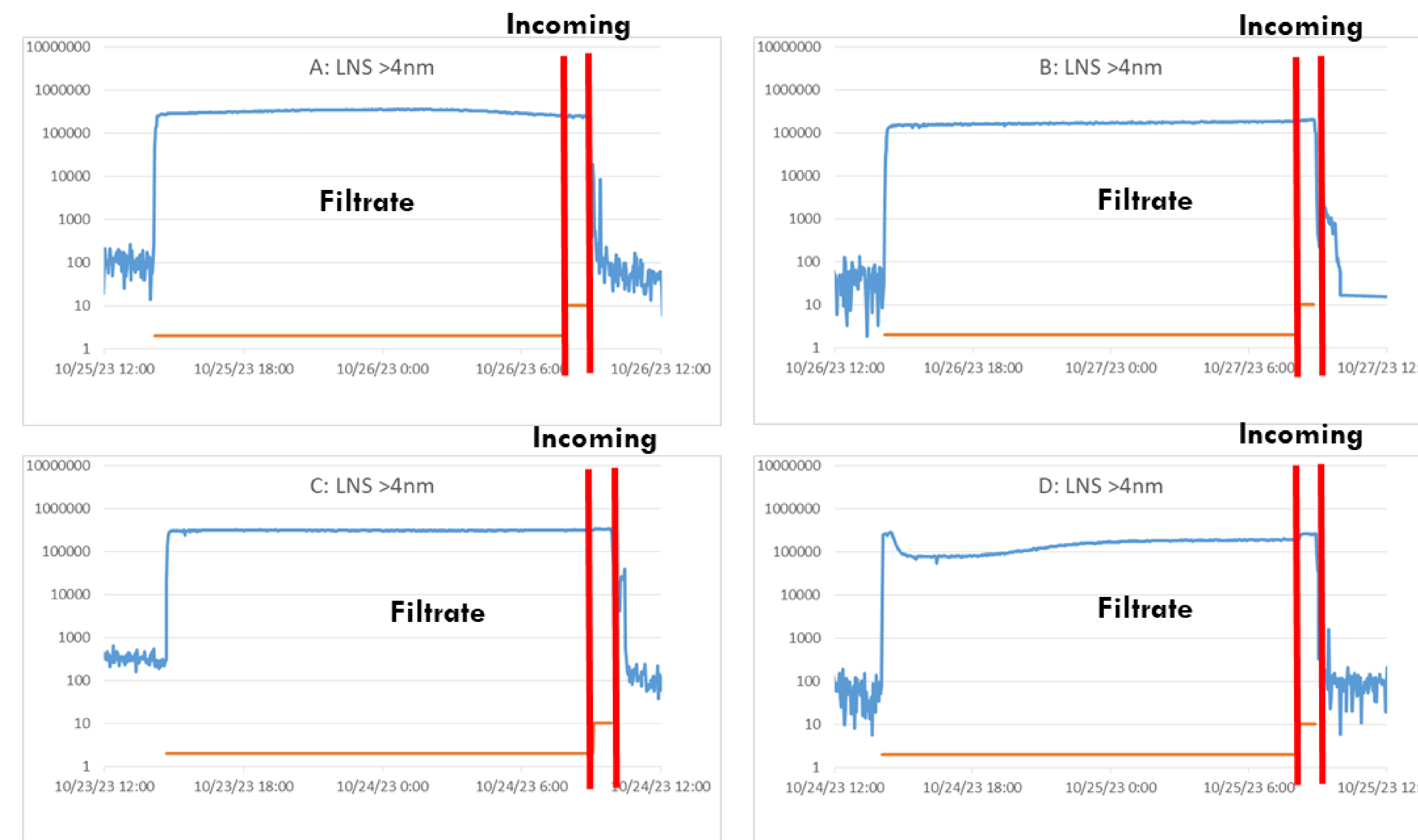
Particle Precursor Definition: *A particle precursor is a dissolved molecular compound that can form particles when dried on a wafer surface.*

Source – IRDS in communication with NIST

New Standard: Guide to Evaluate the Efficacy of Particle Precursor Reduction Devices Used in Ultrapure Water (UPW) Systems

Typical Retention Response with Mixed Anion and Cation Extract





The method was found to be repeatable however, IX resin PP was not retained with current generation filtration media.

Summary and Take-aways

- IRDS more Moore models are pushing the critical particle size to less than 2 nm.
- Techniques for monitoring sub10-nm particles and particle precursors for in-liquid and on-wafer are required to detect critical particles for future technology nodes.
- Kanomax aerosolization-based liquid analyzers enable real-time monitoring of sub-10 nm particle and particle precursor concentrations in high purity process chemicals.
- UNISERS Surface Enhance Particle Sizing (SEPS) provides on-wafer detection of sub-10nm particles. Sequential Spin Coating (SSC) enables the transition from liquid to on-wafer metrology. SEPS can be utilized in a R&D lab environment or a fully-automated fab environment.
- Industry-directed, collaborative R&D is necessary to accelerate the introduction these and other emerging technologies in the semiconductor metrology mainstreams.

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Thank You

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Bios and Contact Details

Gary Van Schooneveld - CT Associates, Inc
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Gary is a leading authority in nano-scale particle detection with over 35 years of semiconductor industry experience. He specializes in sub-10 nm particle measurement in liquids and on silicon wafers, optimizing aerosol-based techniques that extend detection to 1.8 nanometers. His expertise encompasses particle precursor detection methodologies essential for identifying contamination sources that form particles on wafer surfaces, establishing crucial correlations between liquid contamination and on-wafer defects through collaborative studies with major manufacturers. Gary serves as Co-chair of the IRDS Critical Components Task Force and is active member of the IRDS UPW Task Force. Gary also co-chairs the SEMI Ultrapure Water Task Force and is responsible for leading development of multiple industry standards including SEMI C79, C93 and F121. Recent honors include the SEMI Leadership Award (2024) and Semiconductor Review's Advance Nanocontamination Measurement Service Award (2025). He holds BS and MS degrees in Materials Engineering from Rensselaer Polytechnic Institute and an MBA from UT Arlington.

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Derek began his professional career as a development engineer in the particle instruments group at TSI incorporated. In the fall of 2002, He left TSI to join Quant Technologies where he was responsible for the design of aerosol particle counters and aerosol-based liquid analyzers used in the analytical chemistry and semiconductor industries. In the fall of 2009, Derek temporarily left industry and returned to graduate school to earn his doctorate at the University of Minnesota. Derek joined Kanomax FMT, née Fluid Measurement Technologies, in October of 2013 where he was responsible for the development and manufacturing of state-of-the-art instrumentation used for measuring the properties of particles in fluids. In July of 2025, Derek became Chief Technology Officer of Kanomax Holdings; overseeing R&D for Particle, Semiconductor, and Analytical Solutions Worldwide. Derek is the inventor/co-inventor on 11 patents with several patents pending, an author/co-author on over 15 peer reviewed journal publications, and has presented at dozens of conferences throughout the world.

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Dr. Marie Tripp is a seasoned professional in the semiconductor industry, with expertise in supply chain management, sales, manufacturing R&D, and high-volume production. Her technical specialties include semiconductor metrology and process control equipment, focusing on wafer and EUV mask inspection, optical metrology, and analytical failure analysis. Currently, Dr. Tripp serves as VP of Customer Relations at Unisers AG, where her leadership is enabling an early-stage startup to find and eliminate contamination in semiconductor manufacturing. Previously, she held multiple roles at Intel Corporation, including positions in manufacturing R&D and supply chain management. Dr. Tripp holds a PhD in Electrical Engineering from the University of Colorado and has served on advisory boards such as the Semiconductor Research Corporation and the NSF STROBE Science and Technology Center.