



Particle Precursors – a Nonobvious Source of Defectivity Issues



Based on Yield Enhancement IRDS and SEMI Standards Studies



Gary Van Schooneveld, CT Associates
Slava Libman Ph.D., FTD Solutions



IRDS and SEMI Collaborative R&D Supporters

IRDS UPW Task Force

- Slava Libman – Co-Chair
- Gary Van Schooneveld – Co-Chair

IRDS Critical Components Task Force

- Bob McIntosh – Co-Chair
- Archita Sengupta – Co-Chair

Integrated Device Manufacturers – Global Foundries, Intel, Micron, Samsung

Equipment Suppliers - ASML, Screen

Component Suppliers – Asahi, Daiken, Entegris, Georg Fischer, Pall, Saint Gobain

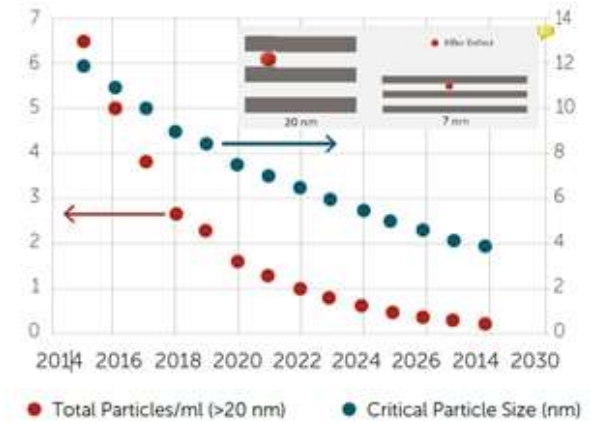
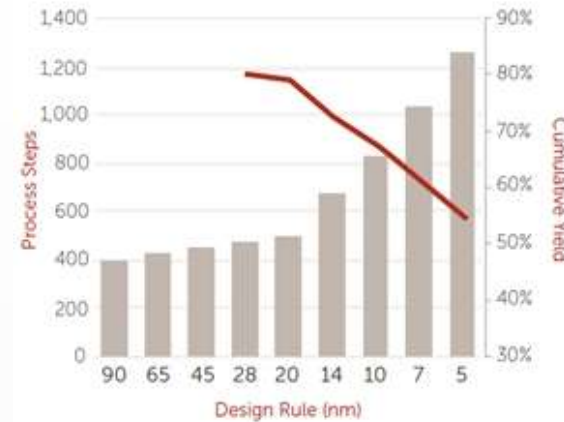
Material and System Providers – Evoqua, Ovivo, Kurita, Lanxess, Solvay, 3M

Technical and Experimental Services – Balazs, CT Associates, Environmental Energy Services, FTD Solutions, Page/imes, Unisers

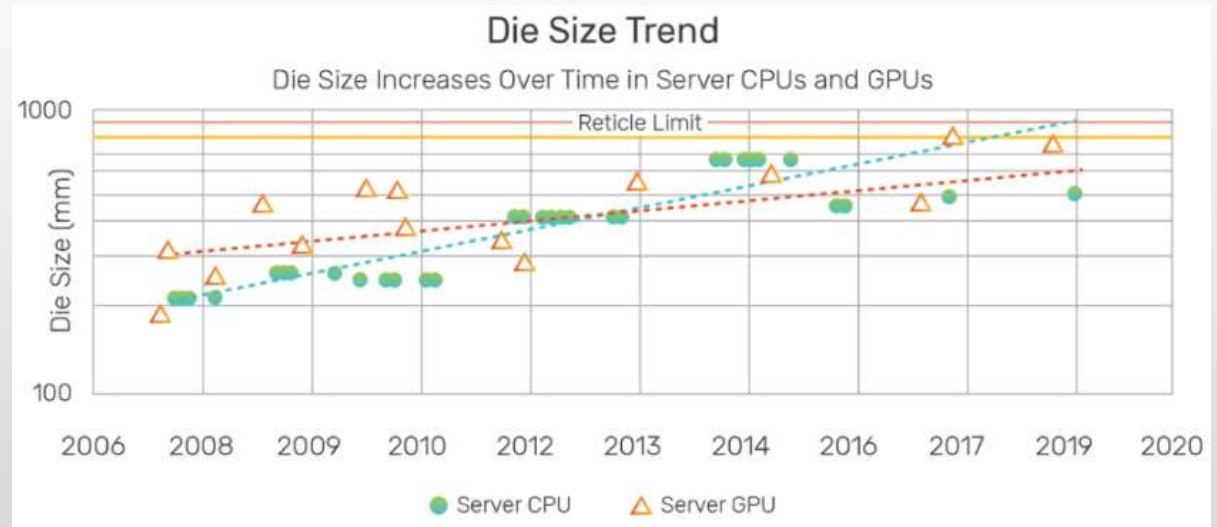
Why is Collaborative R&D Necessary?

Defectivity and Reliability

- Chips getting more complex
 - Yield Risks
 - More process steps
 - New materials
 - Higher aspect ratio
 - Increasing die size due to memory and machine learning
 - >100mm² for high-end AI
 - Reliability Risks
 - High temperature (energy) of HP devices
- Stringent operating range
 - 24/7 continuous operation of HP chips
 - >5-10 years for server/AV chips at >115-125C
- Reliability specs getting hard to meet
 - Critical defect sizes are at the single-nm scale
 - Molecular contamination is in priority
- Time-to-market implications
 - Faster ramp
 - Heterogeneous integration



Source: Semiconductor Digest 2019



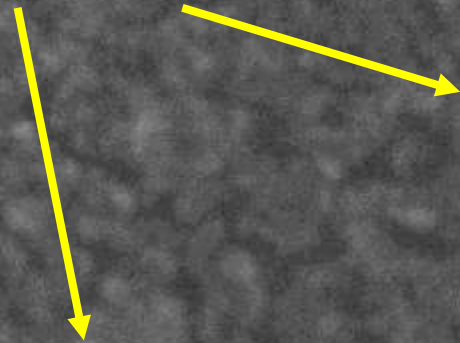
Source: CadenceLIVE Americas 2020

What is a Particle Precursor?

- UPW IRDS, in communication with the National Institute of Standards and Technology (NIST), defined particle precursor as "*a dissolved molecular compound, which may form particles when dried on the wafer surface*".
- Examples – Medium to high molecular weight organics and silicates.

Ion Exchange Resin Effluent

5- 10nm particles

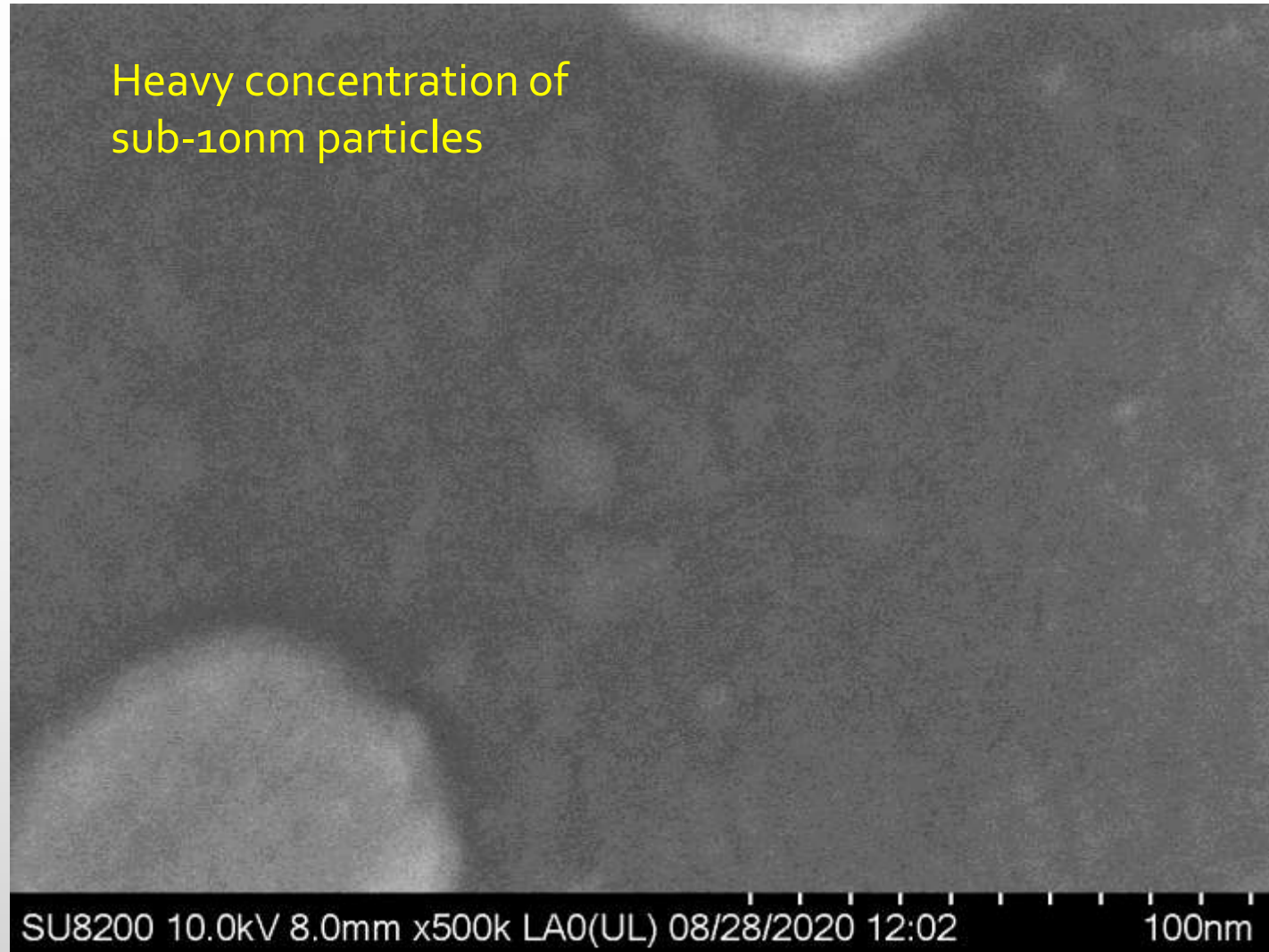


SU8230 15.0kV 5.3mm x500k SE(UL) 05/28/2020 11:55 100nm

- Sample collection by Focused Aerosol Deposition (FAD)
- Sample analysis by FESEM (Hitachi SU8230 – U of MN CharFac)

Hot UPW Fluoropolymer Piping Extract

Heavy concentration of
sub-10nm particles



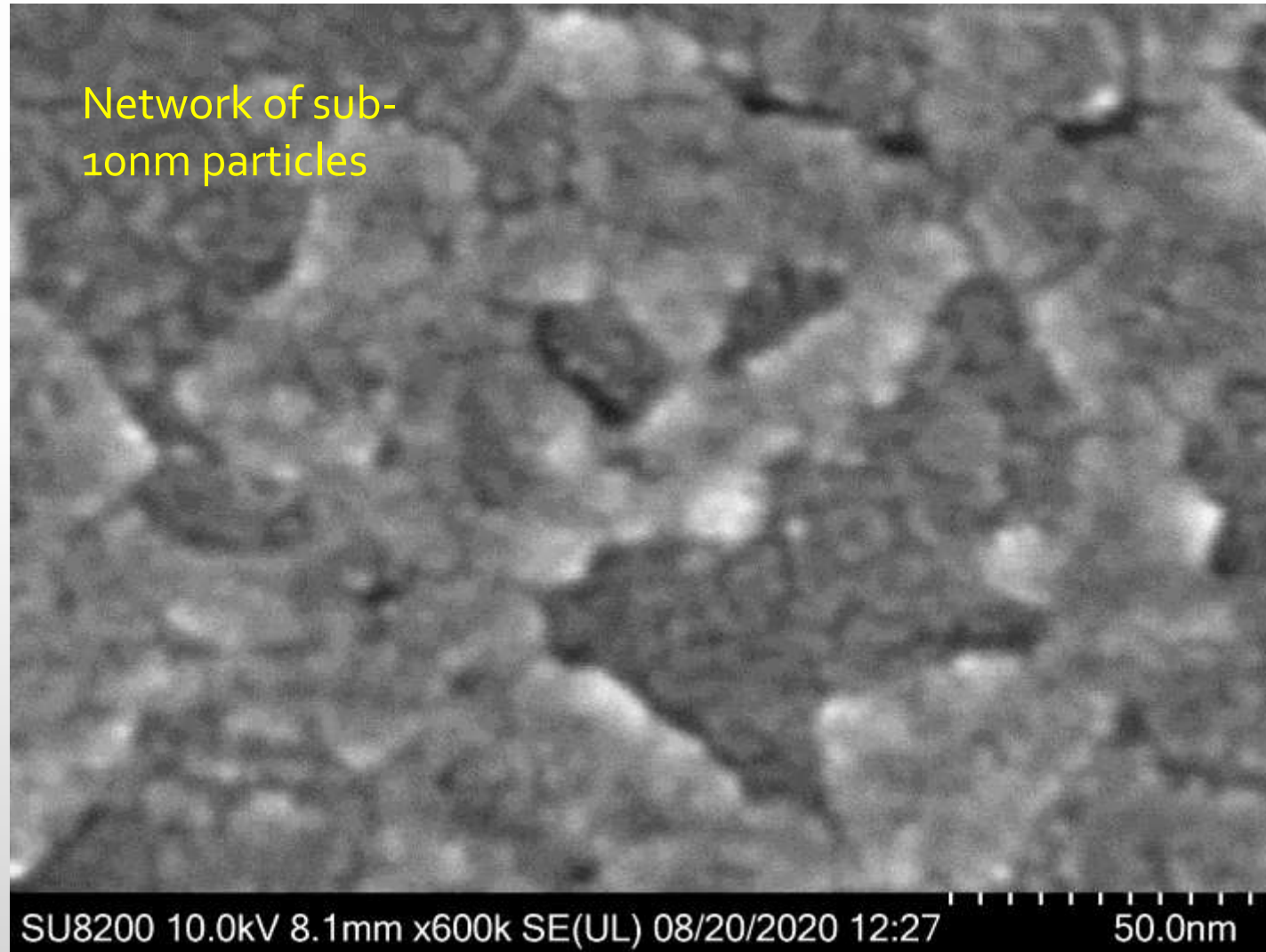
SU8200 10.0kV 8.0mm x500k LA0(UL) 08/28/2020 12:02

100nm

- Sample collection by Focused Aerosol Deposition (FAD)
- Sample analysis by FESEM (Hitachi SU8230 – U of MN CharFac)

Gigabit Isopropyl Alcohol

Network of sub-10nm particles



SU8200 10.0kV 8.1mm x600k SE(UL) 08/20/2020 12:27 50.0nm

- Sample collection by Focused Aerosol Deposition (FAD)
- Sample analysis by FESEM (Hitachi SU8230 – U of MN CharFac)

Why are particle precursors becoming a significant risk?

- Evolving chip design and yield constraints.
- Limited effectiveness of filtration and alternate mitigation options.
- Particle precursor are invisible to traditional particle measuring technologies based on light-scattering such as optical particle counting (OPC) and dynamic light scattering (DLS).
- Online metrology is limited, and interpretation of results can be challenging.

Chip design and yield constraints

2020	Width (nm)	2020	2022	2025	2020	2022	2025	Defect Mechanism	Process Type for predominant defect mechanism
		Defect Size (nm)	Defect Size (nm)	Defect Size (nm)	Dx/wafer	Dx/wafer	Dx/wafer		
Gate	20	10.0	9.0	7.0	5.7	5.0	6.6	Patterning, Gate stack	Dry etch, Wet Etch (GAA), Wet Clean
Fin	7	3.5	3.0	3.5	133.5	136.1	52.8	Gate stack, EPI	Dry etch, Dry Clean (SiCoNi) or Wet Cleans
VC	16	8.0	9.0	9.0	11.2	5.0	3.1	Clean	Dry etch, Wet Clean, Wet Fill (Electroplating), Wet Clean
MetalC	16	8.0	9.0	9.0	11.2	5.0	3.1	Patterning, Metal	Dry etch, Wet Clean, Wet Fill (Electroplating), Wet Clean
Via0	15	7.5	6.0	5.0	13.6	17.0	18.1	Clean	Dry etch, Wet Clean, Wet Fill (Electroplating), Wet Clean
Metal0	15	7.5	6.0	5.0	13.6	17.0	18.1	Patterning, Metal	Dry etch, Wet Clean, Wet Fill (Electroplating), Wet Clean
Viax	18	9.0	8.0	6.0	7.9	7.2	10.5	Clean	Dry etch, Wet Clean, Wet Fill (Electroplating), Wet Clean
Metalx	18	9.0	8.0	6.0	7.9	7.2	10.5	Patterning, Metal	Dry etch, Wet Clean, Wet Fill (Electroplating), Wet Clean
Viay	40	20.0	20.0	20.0	0.7	0.5	0.3	Clean	Dry etch, Wet Clean, Wet Fill (Electroplating), Wet Clean
Metaly	40	20.0	20.0	20.0	0.7	0.5	0.3	Patterning, Metal	Dry etch, Wet Clean, Wet Fill (Electroplating), Wet Clean

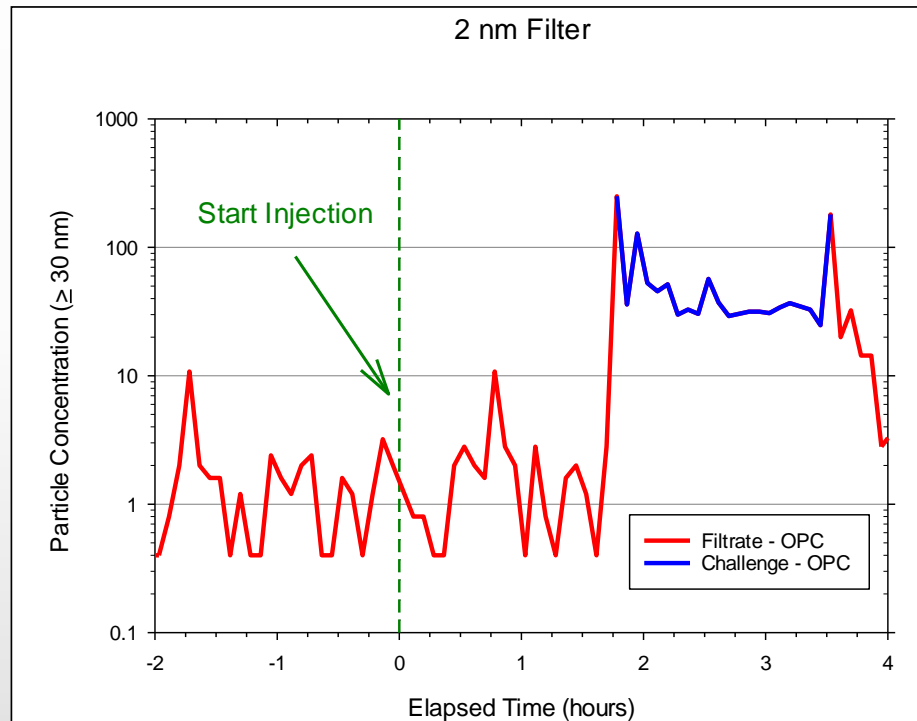
Link required...correlation between particles in the liquid and deposition rate on the wafer

Significant collaborative work is being done by IRDS UPW and Critical Components Teams

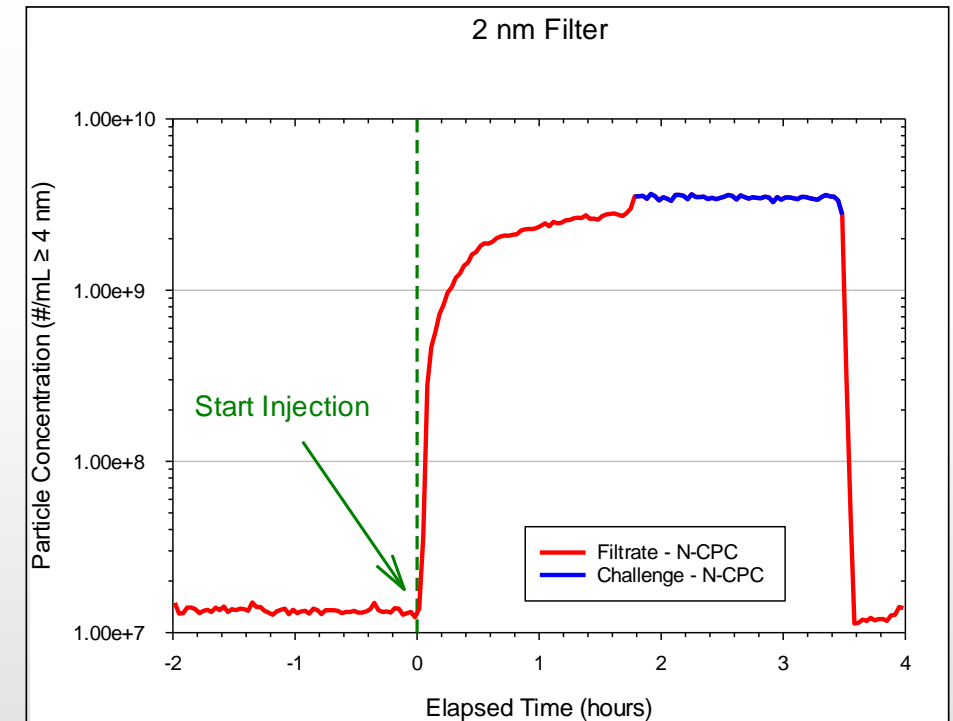
Limitation of filtration and OPC metrology

Retention testing of poly(sodium 4-styrenesulfonate) in UPW (HMWO compound)

30 nm OPC



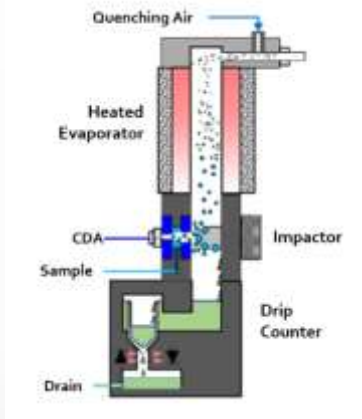
4 nm Nebulization - CPC



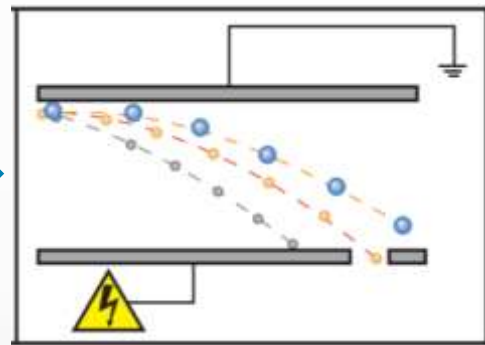
- OPC able to detect large particles > 30 nm but HMWO contaminants are invisible.
- While retention of OPC detectable particle is $> 99.9\%$, PP retention is less than 10%

Particle Precursor Detection Enabling Technology: Nebulization – Aerosol Particle Counting

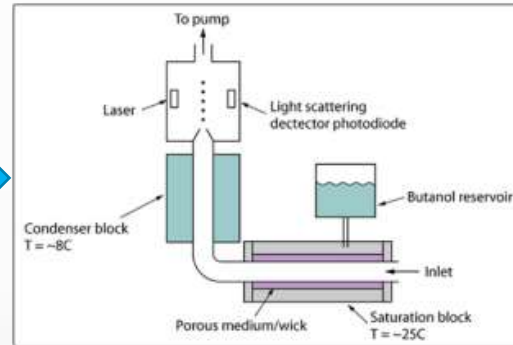
Liquid Nanoparticle Sizing (LNS)



Nebulization

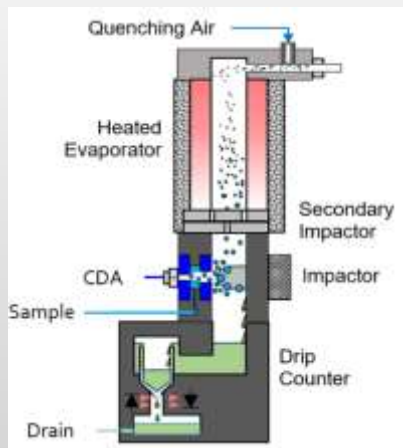
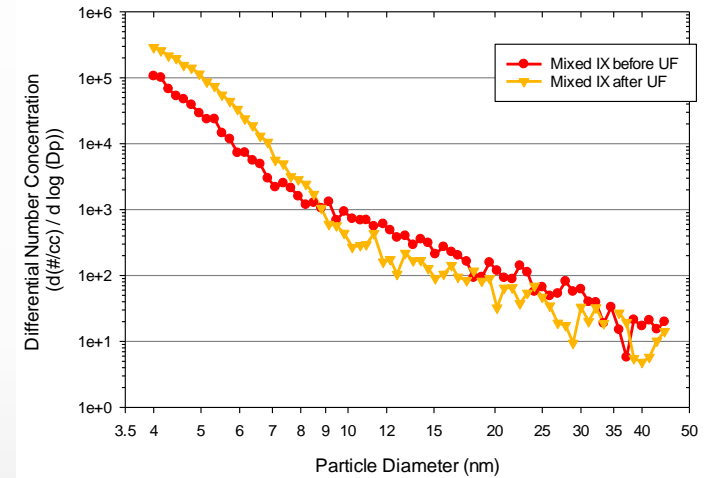


Size Classification by Charge Mobility

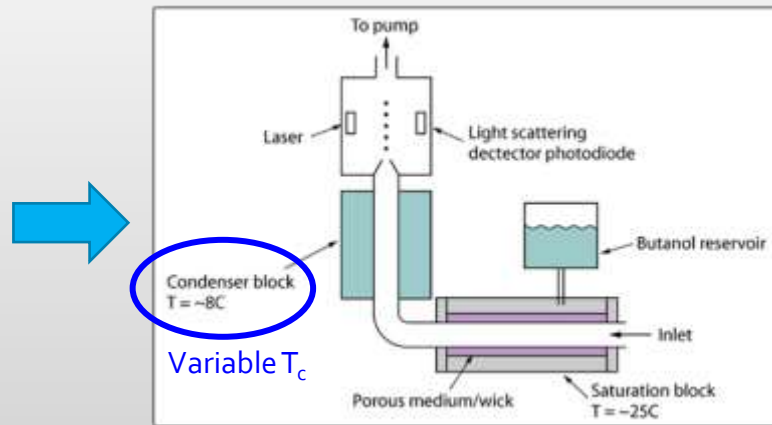


Condensation Particle Counting (CPC)

IX Resin Particle Size Distribution



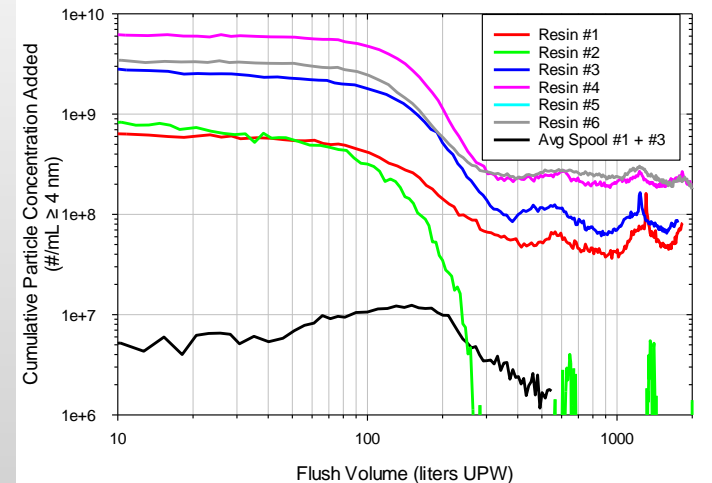
Nebulizer



Condensation Particle Counting (CPC)

Scanning Threshold Particle Counting (STPC)

IX Resin Flush



IRDS Particle Precursor Risk Assessment

Sponsor – IRDS UPW Task Force

@ CTA (USA)

- Liquid sample preparation



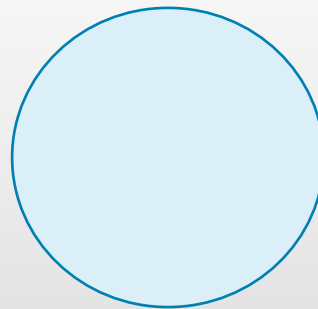
IX Resin PP Extraction

- LNS characterization of liquid particle count (4nm -65nm)



@ Screen (Japan)

- Spin-drying of samples to 12-inch silicon wafer

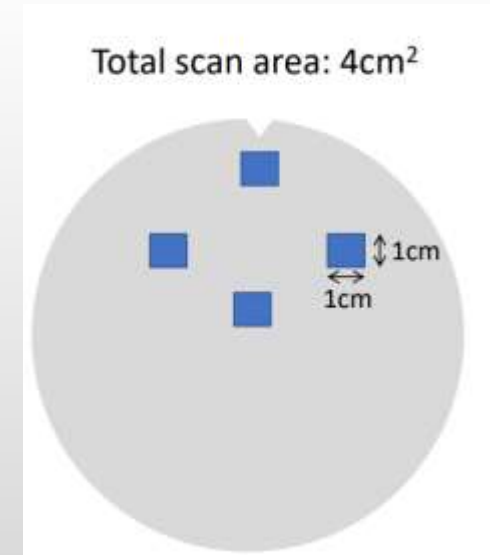


- Full-wafer scan (SP7)



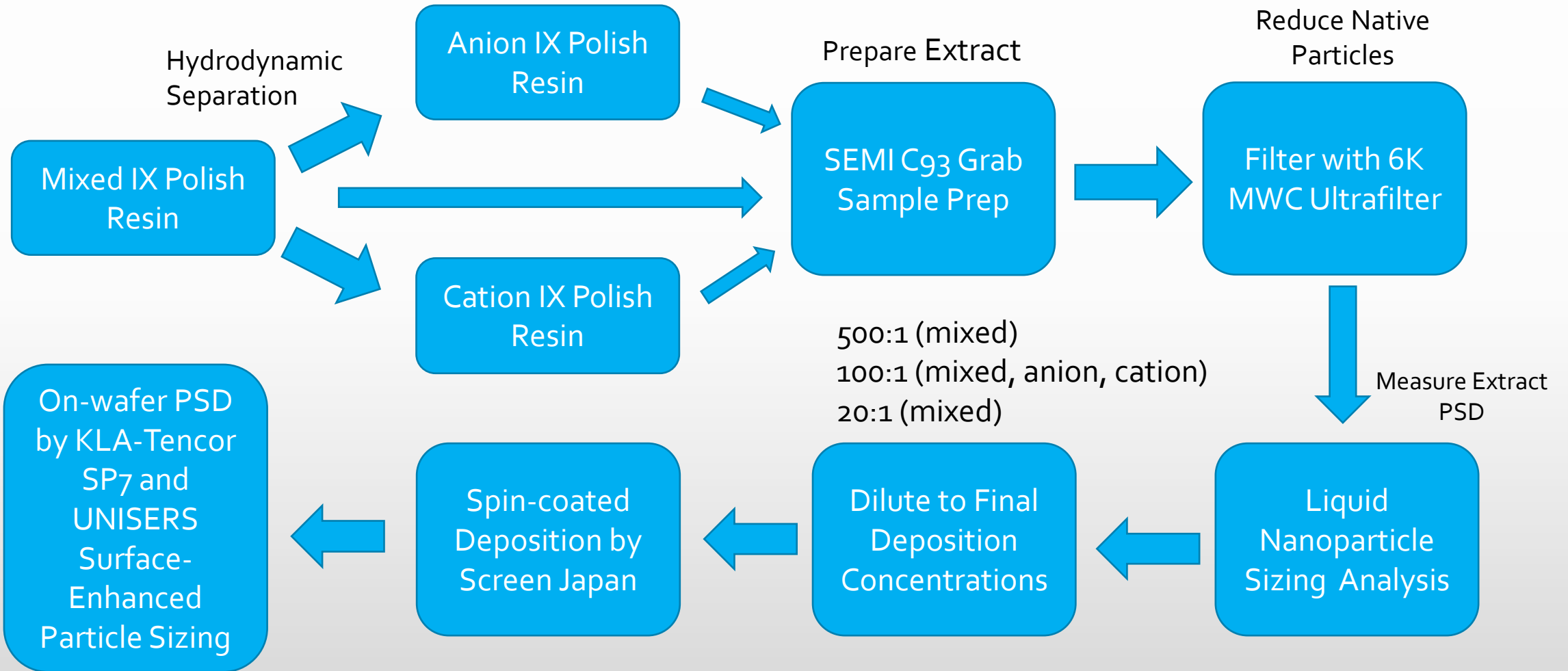
@ Unisers (Switzerland)

- Defect inspection (8nm – 160nm)



- Surface Enhanced Particle Sizing (SEPS)

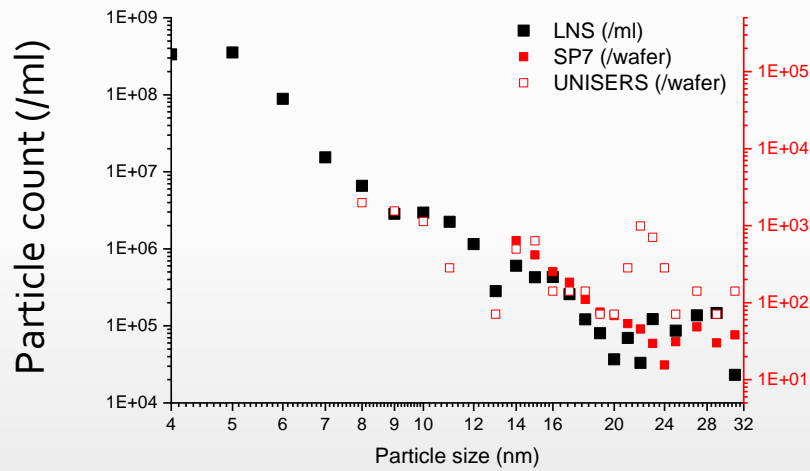
Particle Precursor Test Approach



Particle Precursor Deposition Results

LNS, SP7, UNISERS CORRELATION – Mixed Resin

500:1 dilution, Mixed resin

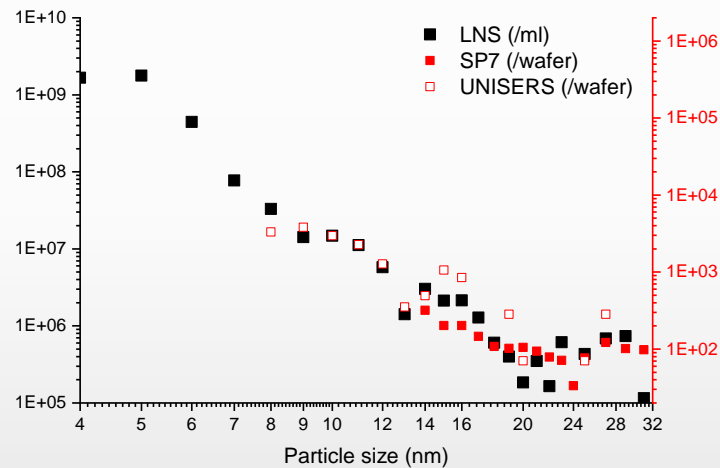


~ 2E3 particles/ml



1 particle/wafer

100:1 dilution, Mixed resin

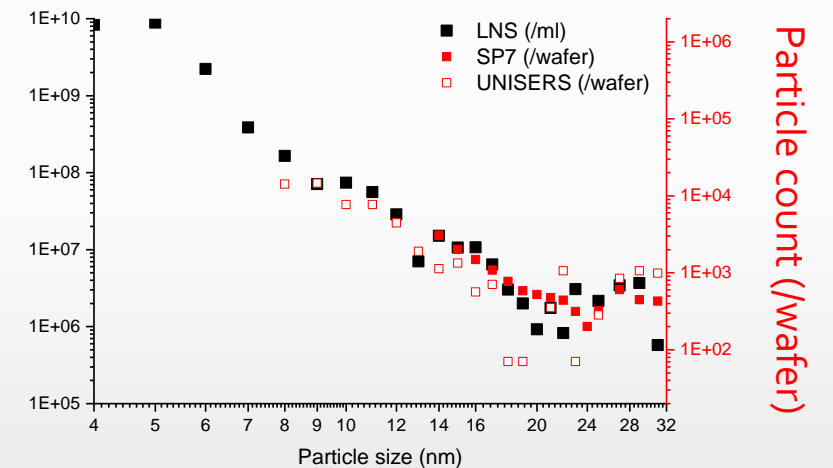


~5E3 particles/ml



1 particle/wafer

20:1 dilution, Mixed resin



~ 5E3 particles/ml

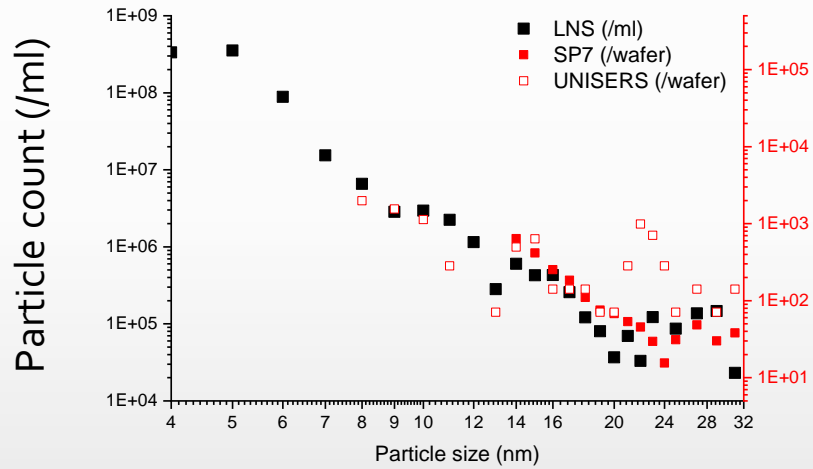


1 particle/wafer

Particle Precursor Deposition Results

LNS, SP7, UNISERS CORRELATION – Mixed Resin

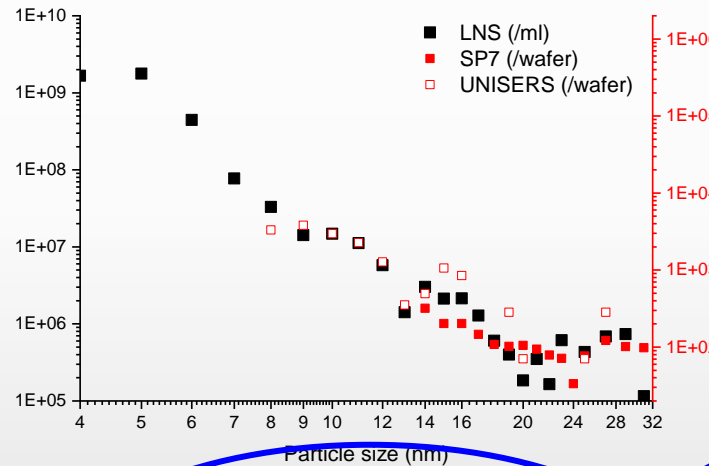
500:1 dilution, Mixed resin



~ 2E3 particles/ml

1 particle/wafer

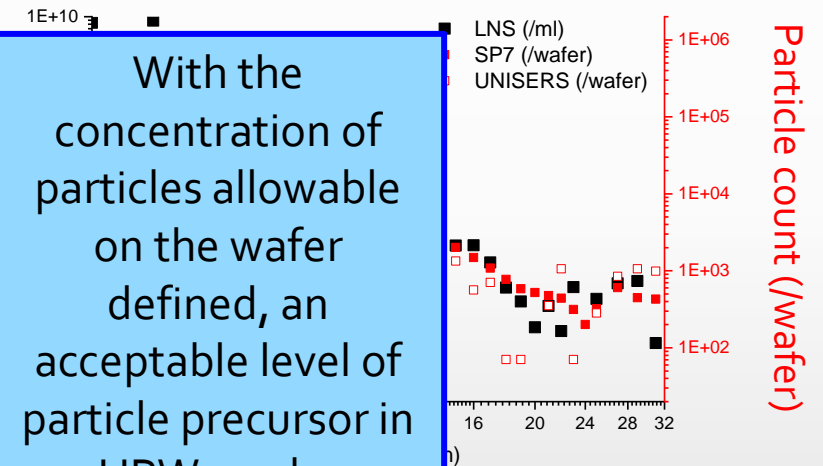
100:1 dilution, Mixed resin



~ 5E3 particles/ml

1 particle/wafer

20:1 dilution, Mixed resin



With the concentration of particles allowable on the wafer defined, an acceptable level of particle precursor in UPW can be established.

1 particle/wafer

International Roadmap for Devices and Systems (IRDS™) 2022 Edition

Table YE3 Technology Requirements for Surface Environmental Contamination Control

Year of Production	2021	2022	2023	2024	2025
Add Pitch designation					
Add Pitch designation					
Logic industry "Node Range" Labeling (nm)	"5"	"3"	"3"	"3"	"2.1"
IDM-Foundry node labeling	i7-f5	i3-f2.1	i3-f2.1	i3-f2.1	i2.1-f1.5
Logic device structure options	FinFet	finFET LGAA	finFET LGAA	finFET LGAA	LGAA
Logic device mainstream device	FinFet	finFET	finFET	finFET	LGAA
MPU/SoC Metafx ½ Pitch (nm) [1,2]	12.0	12.0	12.0	12.0	12.0
Critical particle size non-electrically active (non-EAP) (nm) based on 50% of Logic 1/2 Pitch (nm) (contacted) [1]	10	9	9	9	7
Critical particle size (nm) of Electrically Active particles based on 50% width of fin Logic SiGe Front End or other device critical dimensions for LGAA (>2 monolayers)	3.5	3	3	3	3.5
Particle Precursors, #/ml [56]	this level is expected to produce 5 particles per 300 mm wafer of 10nm particles and greater.	20,000 (see note)	20,000 (see note)	20,000 (see note)	20,000 (see note)

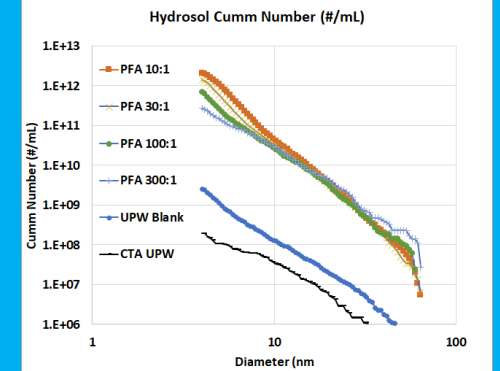
[56] The critical particle size for the manufacture of semiconductors is now below 5 nm. The semiconductor industry is entering a region where particles, particle precursors and molecules in liquids, begin to overlap. A particle precursor is defined as a dissolved molecular compound, which may form particles on the wafer. The ability to distinguish particle precursors from solid particles in UPW is becoming critical. While advanced filtration can remove nanometer sized solid particles, the same filter may have little or no ability to remove particle precursors. The use of combination of Nebulization and Condensation Particle Counting (N+CPC) measurement techniques have shown promise in detecting these particle precursors. This measurement techniques use aerosol particle detection, reconfigured to measure particles in liquids. NIST has extensively used the Nebulization and Particle Counting after Electrical Mobility Size Classification method of measuring particle size. The precursors are measured in the units of the number of particles produced in N+CPC instrument per milliliter of UPW. **It is important to note that the particle precursor parameter cannot be detected with use of currently available metrology at that level defined in the roadmap and therefore should not be used as specification for any purpose other than a target for future technology development around metrology, material purity, and treatment technology.** The available metrology does not have a standard to verify the quality of the data produced. The quality of the results relies on the source specific calibration/verification methods.

IRDS Particle and Precursor of High-Purity Pipe Risk Assessment

Sponsor – IRDS Critical Components Task Force

Technical Objectives:

1. To quantify critical organic extracted from high-purity piping using hot UPW; specifically, PFA and PVDF.
2. Assess the affinity of the organic contaminants to the wafer surface during spin processing.
3. Determine the relative concentrations of organic contamination remaining on the wafer surface as a function of temperature.



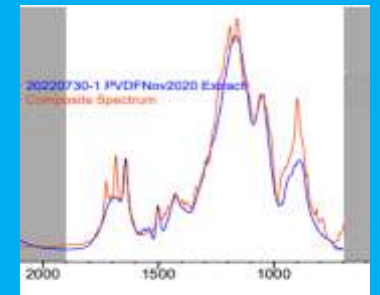
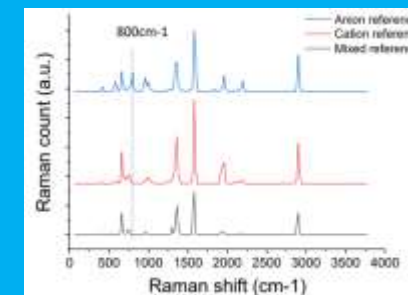
Particle Size Distribution Analysis



Spin Coat

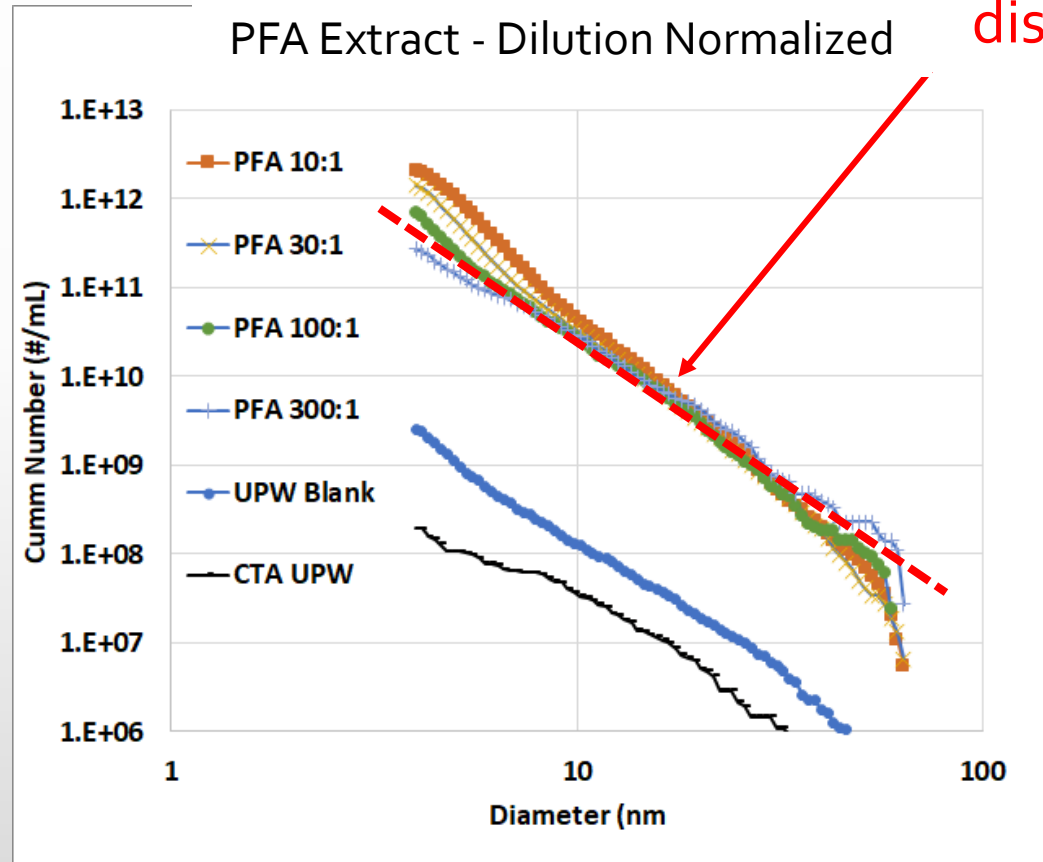


Surface Analysis



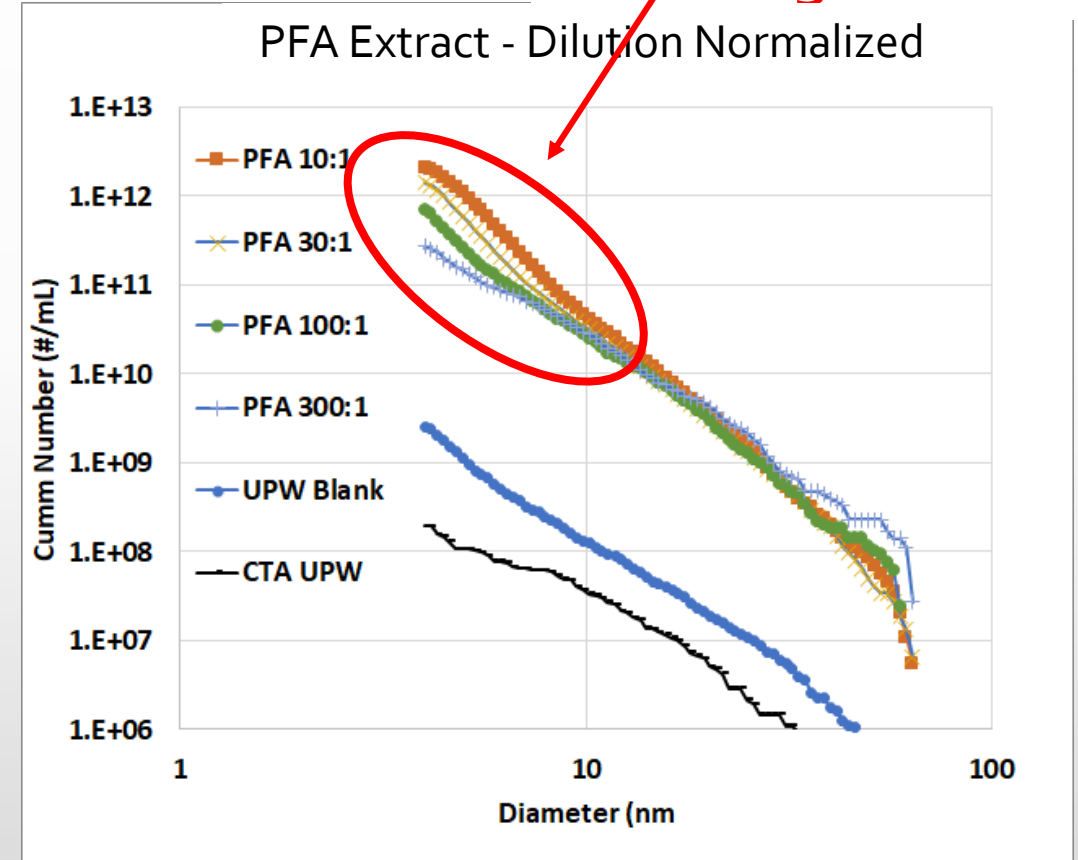
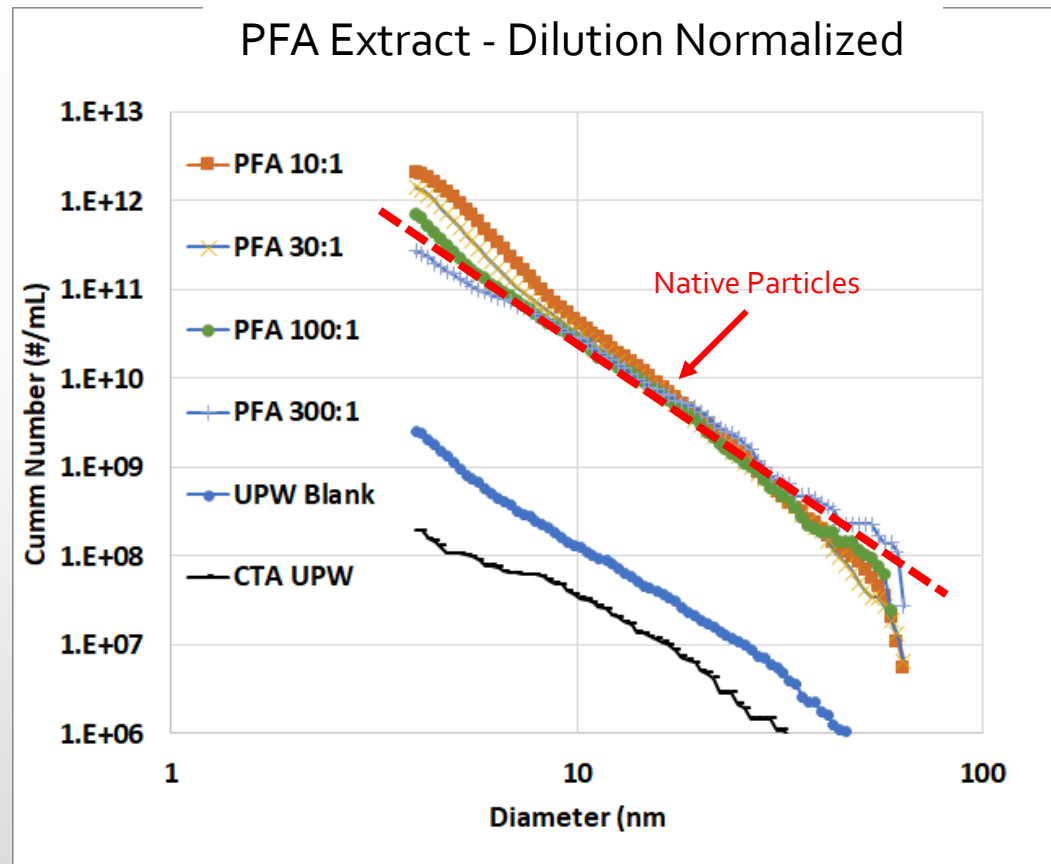
Molecular Finger Printing

LNS Extract from UPW, 14 Day 85°C



Native particle size distribution

LNS Extract from UPW, 14 Day 85°C

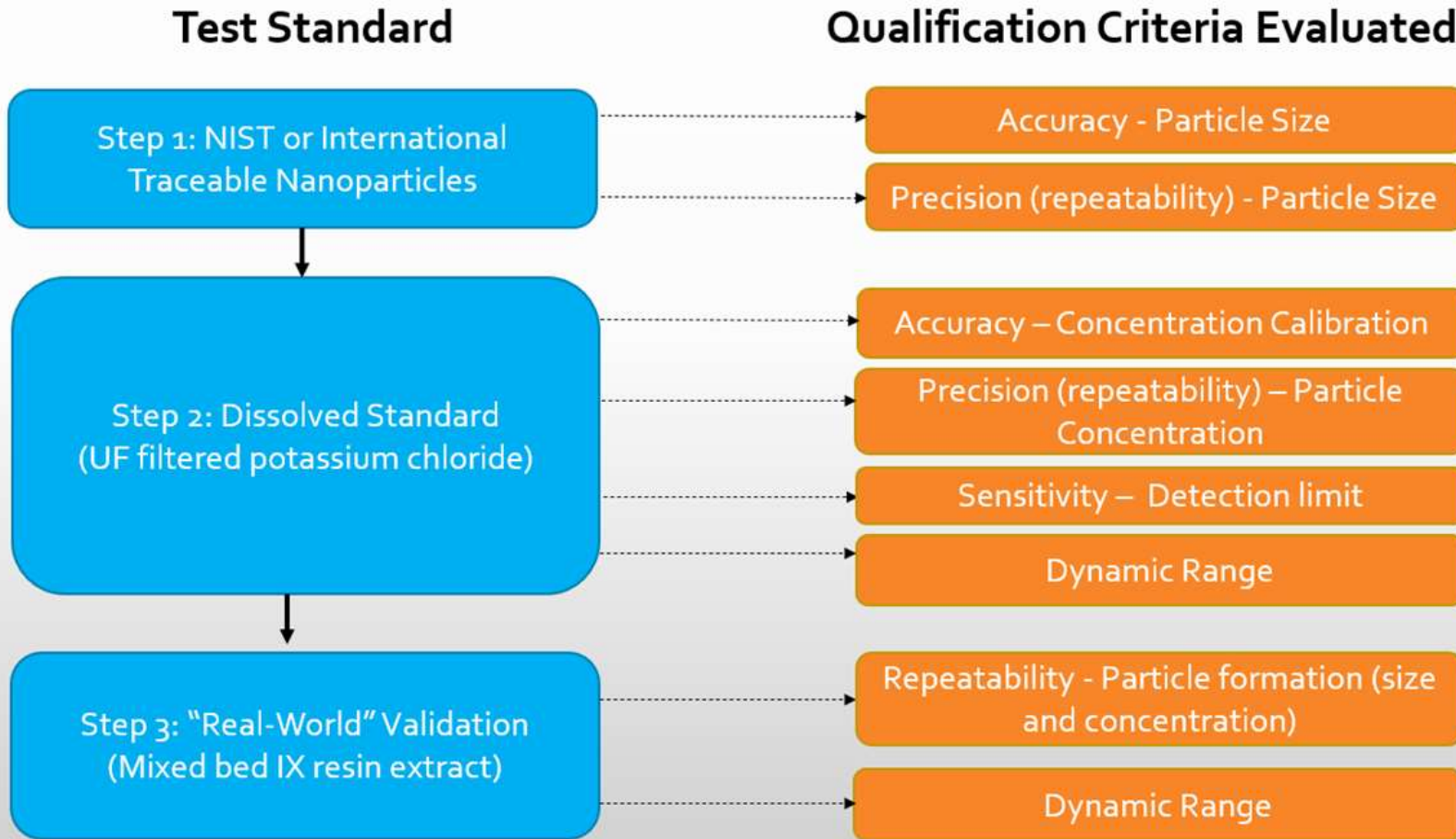


Native particles and particle precursors evident in both hot UPW PFA and PVDF extract

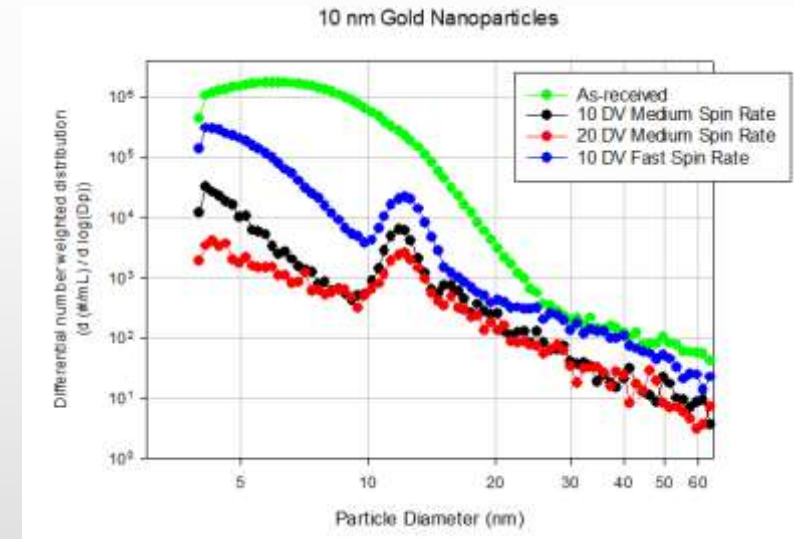
SEMI UPW TF –Addressing Precursor Issues

- New document in work addressing Particle Precursor Metrology
- Revision of SEMI C79 (Filter Cleanliness/Performance)
 - Developing IX resin extract as a filter/membrane challenge
- SEMI C93 (IX Resin Quality) – updated with smaller particle size
- SEMI F61 (UPW System), F63 (UPW Quality), F75 (UPW Metrology)
 - Will incorporate proactive precursor management in next revisions

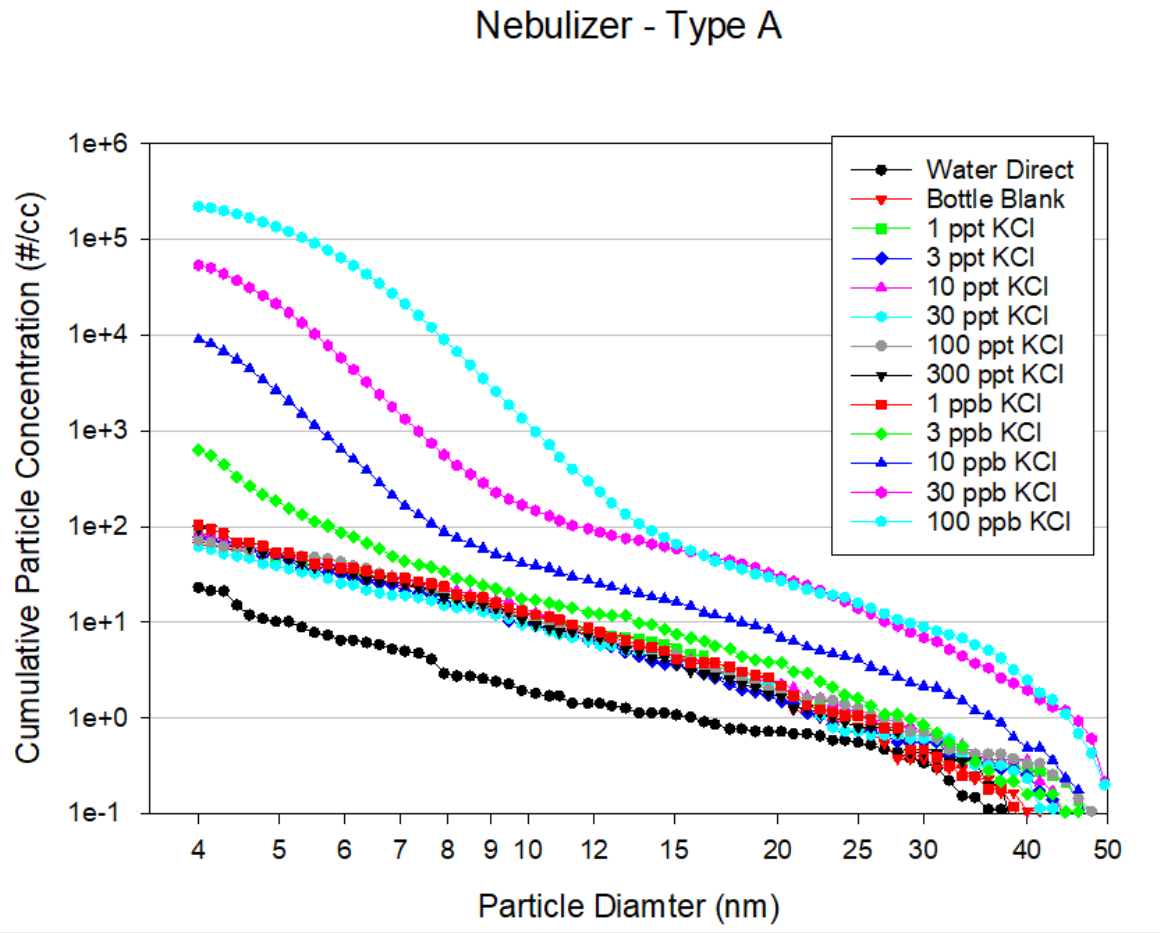
SEMI Draft Document 6715 -Guide for Measuring Particle Precursors in Ultrapure Water



Diafiltration preparation of gold nanoparticles

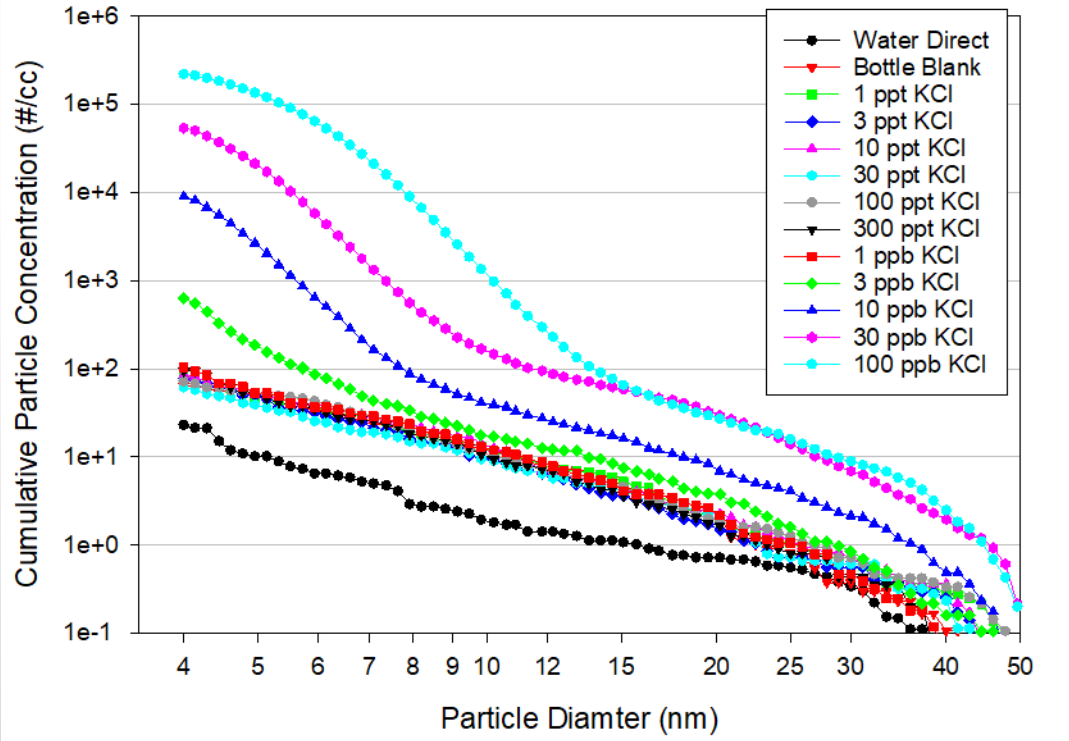


Guide for Measuring Particle Precursors in Ultrapure Water - KCl Calibration Curve Example

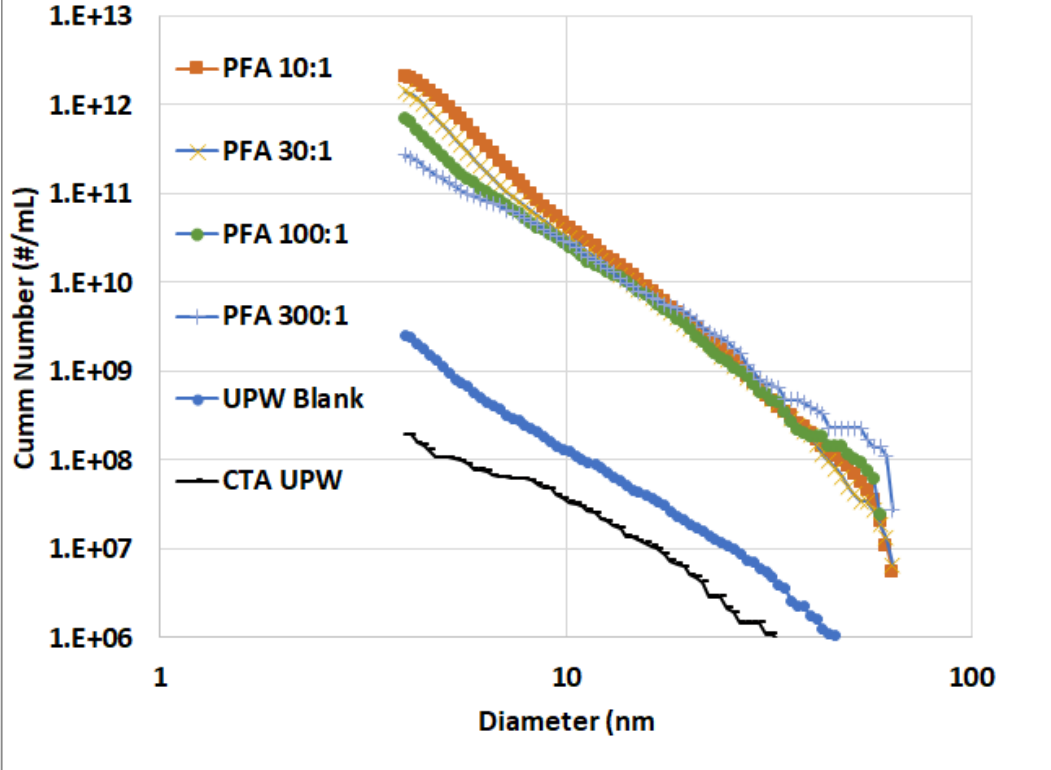


Guide for Measuring Particle Precursors in Ultrapure Water - KCl Calibration Curve Example

Nebulizer - Type A



PFA Extract - Dilution Normalized



Closing Comments

- Particle precursors pose an increasing risk to manufacturing yield.
- Current filtration and purification techniques have limited ability to remove particle precursors necessitating proactive contamination management to minimize these risks.
 - Improvements needed in IX resins
 - Reduced release from high surface area components such as filtration devices, membrane contactors and polymer components is needed.
- Particle precursor contamination is not limited to UPW. Particle precursors have been detected in IPA, sulfuric acid and CMP slurries.
- Online metrology is available that can identify the presence of particle precursor; SEMI UPW TF working on method for validation and quantification.

Questions?

Gary Van Schooneveld
CT Associates, Inc
Eden Prairie, MN
952-470-0166 x24

Email – Gary@CTAssociatesinc.com



IRDS UPW Team

Slava Libman, co-chair	FTD Solutions
Gary Van Schooneveld, co-chair	CT Associates
Bernie Zerfas	Independent
Derek Oberreit	KanomaxFMT
Rick Godec	Independent
Alan Knapp	Evoqua
Marcel Teunissen	ASML
Tim Miller	Purdue
Deena Starkel	Micron
Jim Snow	Screen SPE USA
Andreas Neuber	AMAT
Yoichi Tanaka	Kurita

Abbas Rastegar	AMAT
Jochen Ruth	Pall Corporation
Philippe Rychen	Ovivo
Steven Smith	Texas Instruments
Sung In Moon	Entegris
Dan Wilcox	Page
Varinder Malik	Samsung
Brett Clark	Suez
Glen Slater	Intel
Jason Tewksbury	Intel

IRDS Critical Components Team

Member Name	Organization	Region
Robert McIntosh, Co-chair	Enviro E	US
Archita Sengupta, Co-chair	Intel	US
Albert Lueghamer	Agru	Europe
David Tarantino	3M	US
Matt Orr	Daikin	US
Gary Van Schooneveld	CT Associates	US
James Henry	Arkema	US
Jenell Mccall	Chemours	US
John Leys	Entegris	US
Katrin Wallheinke	Georg Fischer	Europe
Kimberly Farnsworth	Chemours	US

Member Name	Organization	Region
Quynh Le	Air Liquide	US
Stephane Domy	St Gobain	Europe
Steve Powell	Parker	US
Thomas Labour	Arkama	Europe
Tommaso Crisenza	Solvay	Europe
Per Nelson	3M	US
Jim Tu	Entegris	US
Paolo Toniolo	Solvay	Europe
Wim Devos	Solvay	Europe
Nick Dotzenrod	3M	US