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Application of a New SEMI F121 Guide for Evaluating Particle Precursor Metrology

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KANOMAX FMT A Kanomax Company

Creative Technology CT Associates, Inc.

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Outline

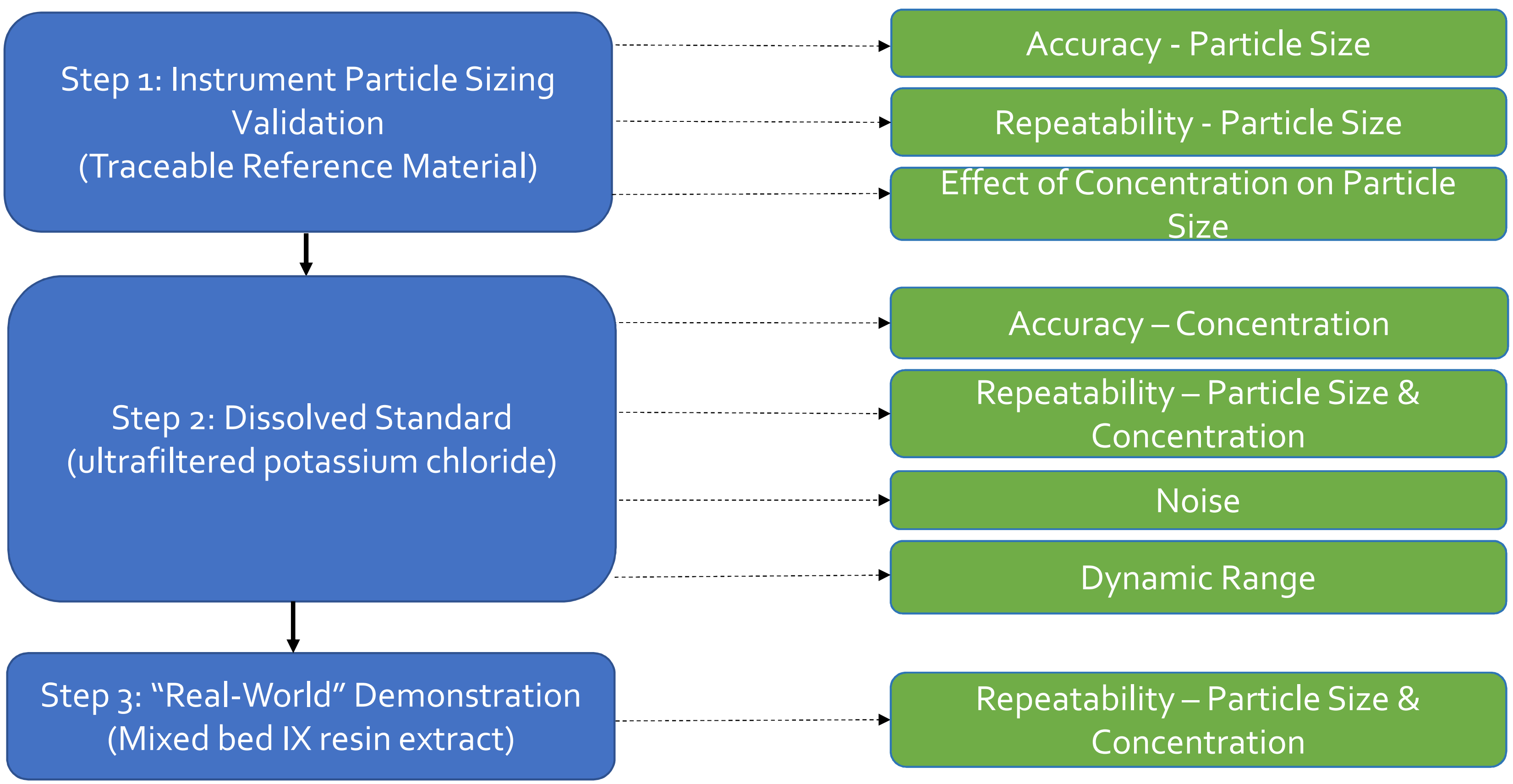
- Introduction to Particle Precursors
- SEMI F121-0923 Guide for Evaluating Particle Precursor Metrology
- Instrumentation used in this evaluation
  - CTA Liquid Nanoparticle Sizer (LNS1 and LNS2)
  - Kanomax FMT sTPC3
- Measurement of dissolved standard (ultrafiltered (UF) KCl)
- Real-world validation with measurement of ion exchange and polymer tubing extracts
- Summary

Introduction

- What are particle precursors (PP)?
  - Dissolved compounds that can form particles when dried on a wafer surface.
- Given the high use of polymers in liquid delivery systems, these contaminants are believed to be primarily organic in nature.
- There are potentially multiple sources of particle precursors in UPW systems including ion exchange resins, tubing and piping, fluid handling components and filter membranes.
- Conventional light-scattering particle detection technologies are challenged to measure particles below 20nm much less particle precursors, which do not form particles until dried.
- Liquid nebulization in conjunction with aerosol-based condensation particle counting has the proven ability to not only detect and count particles as small as 2.5nm, but are a potential tool for measuring particle precursors in liquids.

SEMI F121 Guide for Evaluating Particle Precursor Metrology

Test Material | **Criteria Evaluated**



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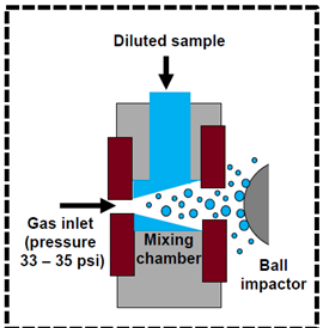
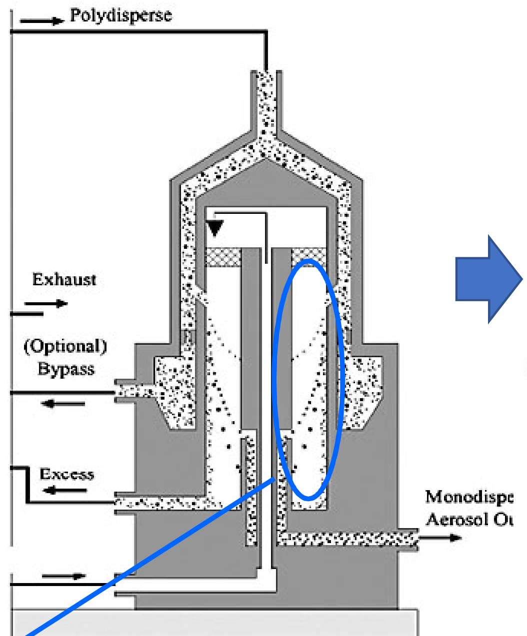
CTA Liquid Nanoparticle Sizing System (LNS)

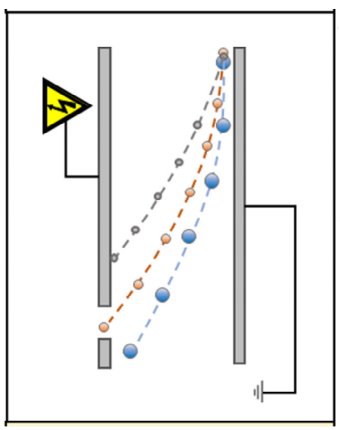
Nebulizer



Charge Conditioner

DMA – Differential Mobility Analyzer

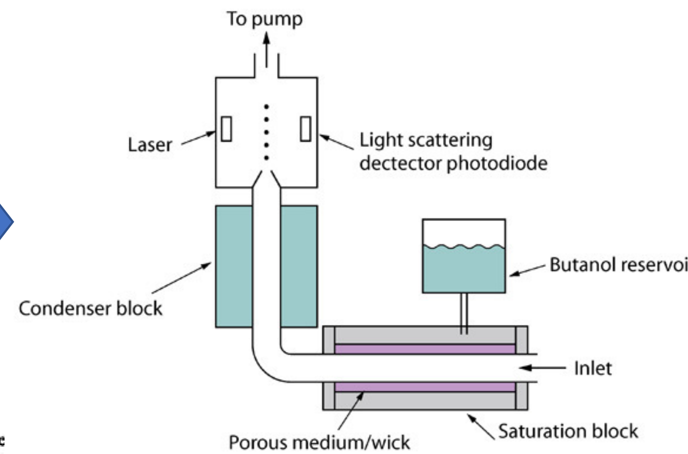




Electrical mobility ∝ particle size

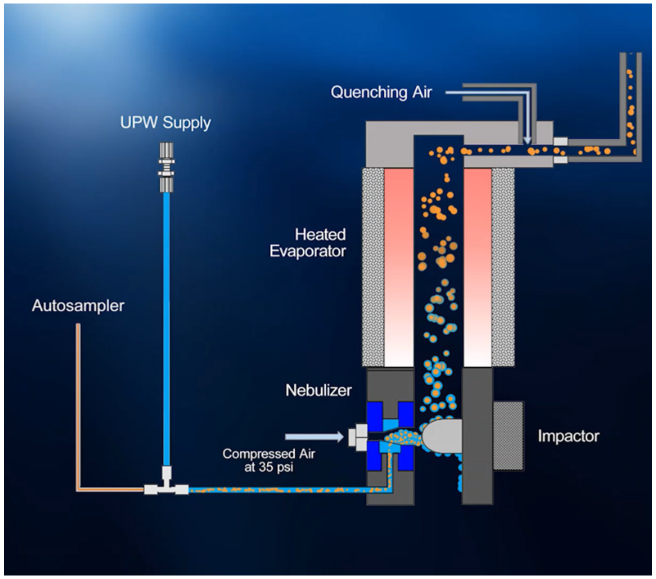
Source: TSI 3936 SMPS Manual

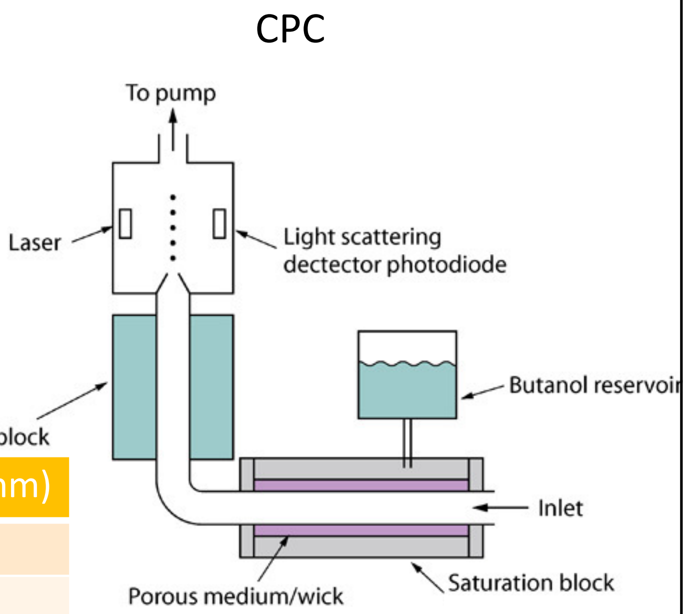
CPC – Condensation Particle Counter



Source: TSI 3775 Condensation Particle Counter Manual

Kanomax FMT 3nm Scanning Threshold Particle Counter (STPC3)



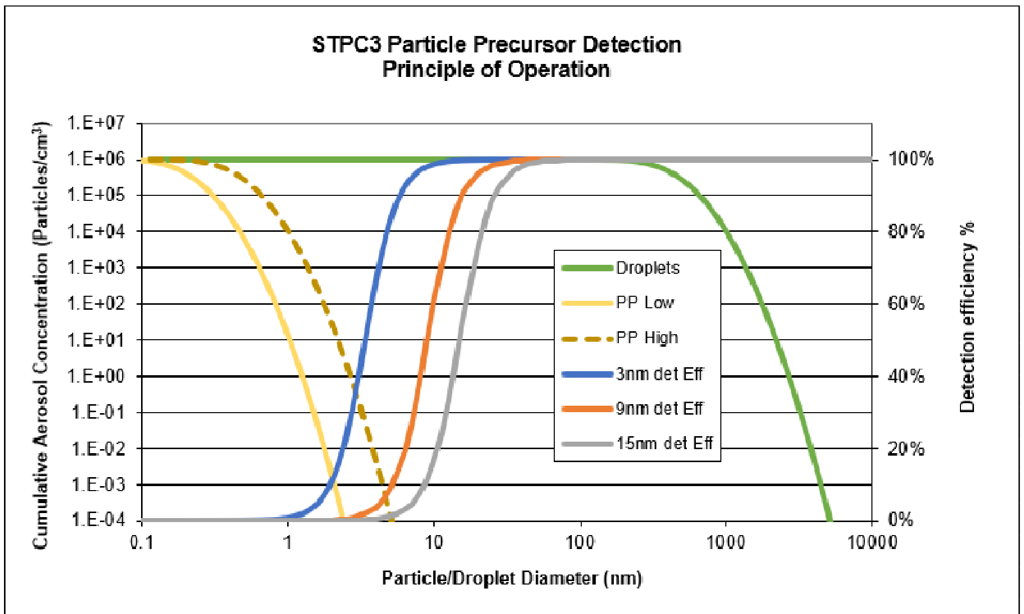


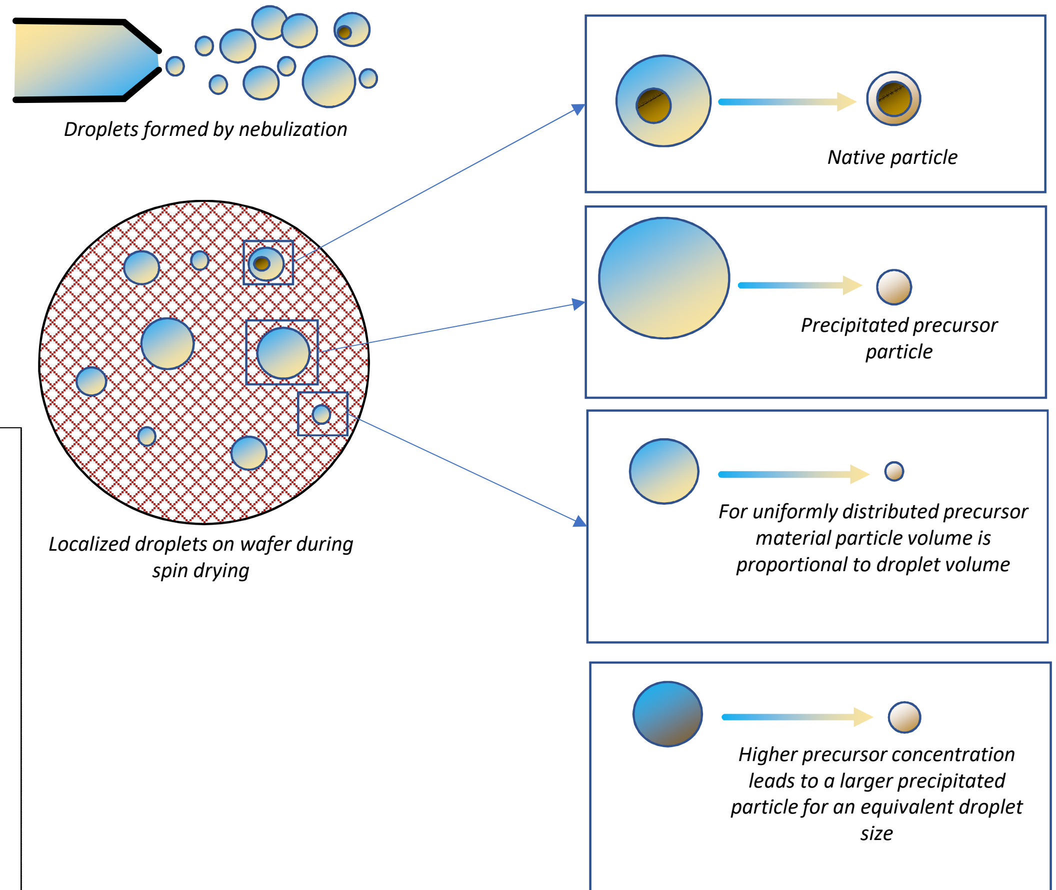
Varying the condenser temperature (T_C) changes the sizing threshold for nucleation

$T_c(^{O}c)$	D_{50}(nm)
15	3
22.5	9
26.5	15

Why nebulization of liquid to form an aerosol?

- When liquid is dried on a wafer surface, native particles in the liquid may adhere to the wafer surface and dissolved compounds may "form" a particle on the surface of the wafer.
- This process is mimicked by nebulizing the liquid sample to form droplets and evaporating the liquid to form an aerosol. This aerosol contains native particles and "formed" precursor particles.

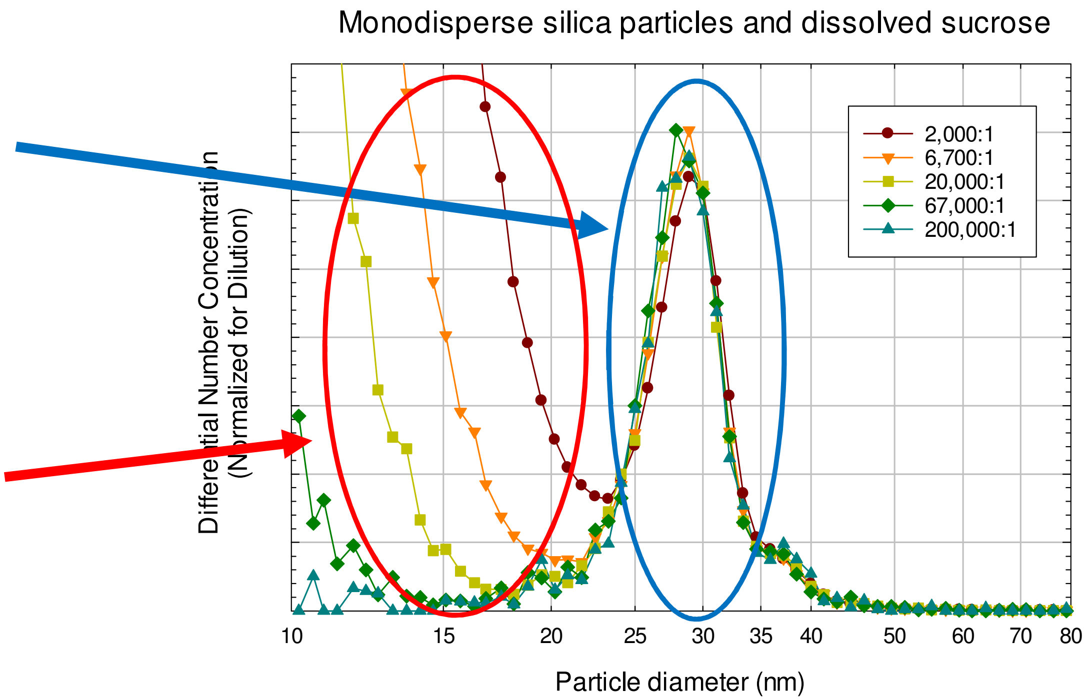




Source: Oberreit, et al, Solid State Phenomena, vol. 346.

How to distinguish between native particles and particle precursors?

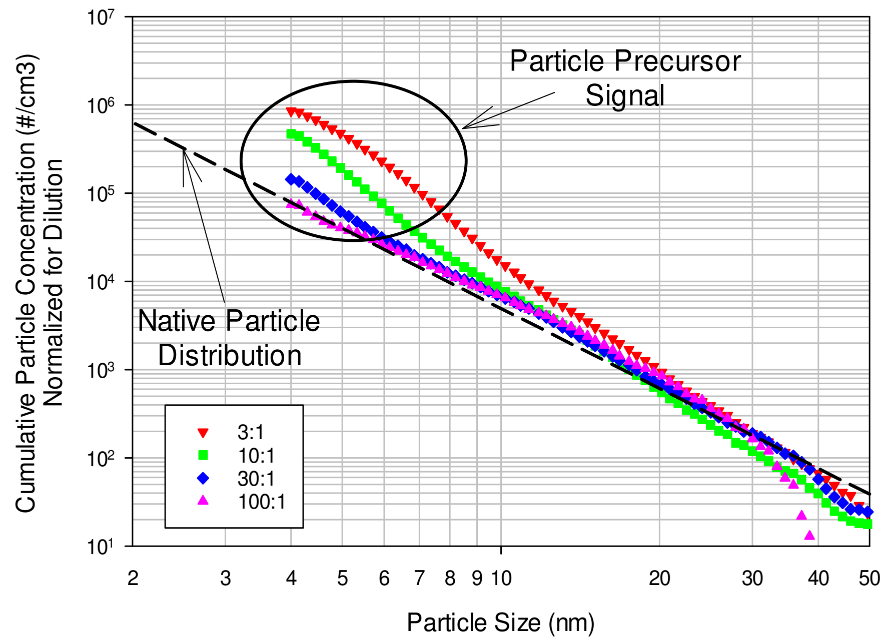
- The native particle concentration changes linearly with dilution ratio. After normalizing for dilution ratio, the native particle concentration in the original sample is unchanged.
- Particle precursor concentration changes non-linearly as dilution ratio is varied. As dilution ratio decreases, the particle precursor signal becomes apparent.



Source: Litchy, et al, Pittcon, 2012.

Example of Native Particles and Particle Precursors in an Extract

- After normalizing for dilution ratio, the native particle concentration in the original sample is unchanged.
- As dilution ratio decreases, the particle precursor signal becomes apparent.



Dissolved Standard Preparation Procedure

- Prepare 1,000 ppm potassium chloride solution using high-purity (ACS reagent grade or better) in ultrapure water (1-gram KCL → 1000 mL water).
- Agitate on shaker table for 12 hours.
- Pre-rinse 4 kDa UF with UPW. Monitor with LNS. Continue rinsing until the concentration of the filtrate is comparable to incoming UPW.
- Filter prepared solution with the clean UF. Collect filtrate in clean HDPE bottle.
- Pull sample for chloride concentration by Ion Chromatography. Calculate KCl concentration.
- Dilute to desired concentrations with UPW.

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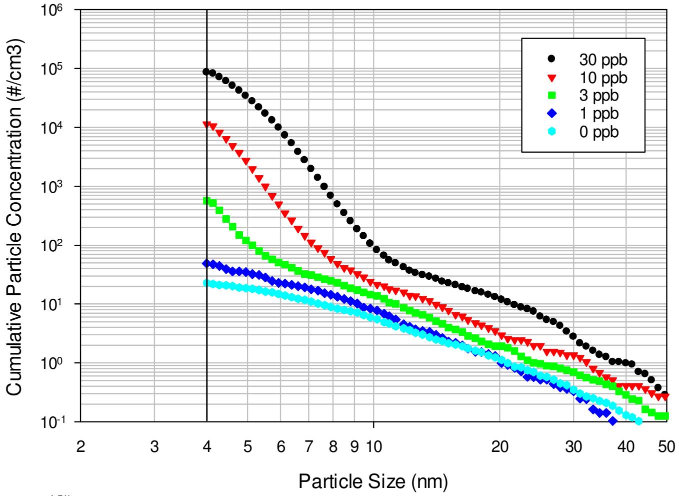
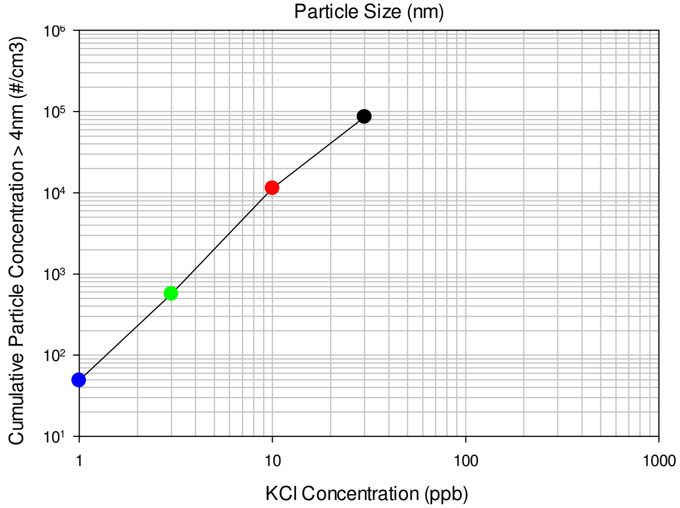
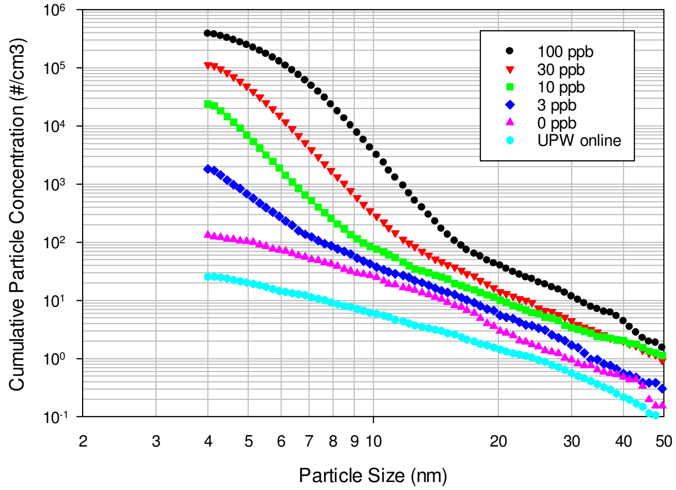
Measurement of dissolved KCl standard:

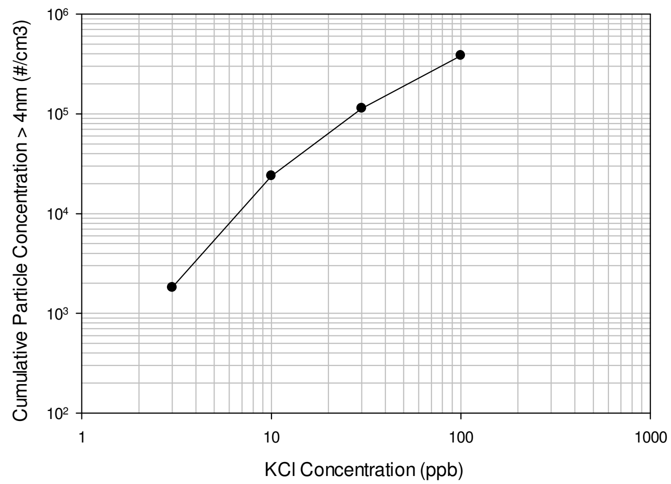
LNS #1: Online dilution method



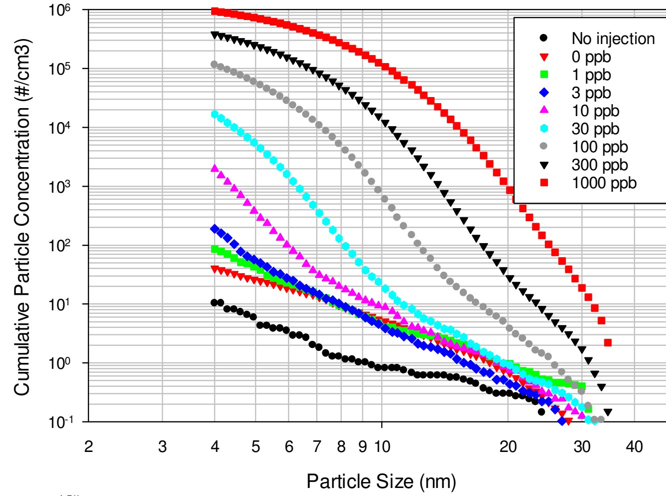


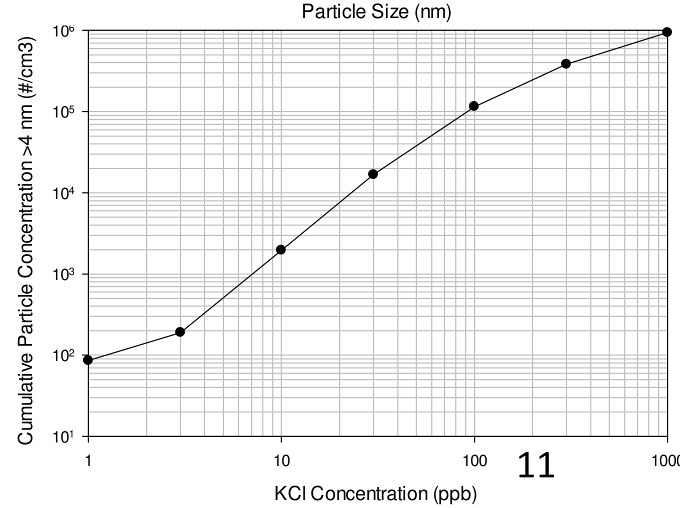
LNS #1: Offline dilution method



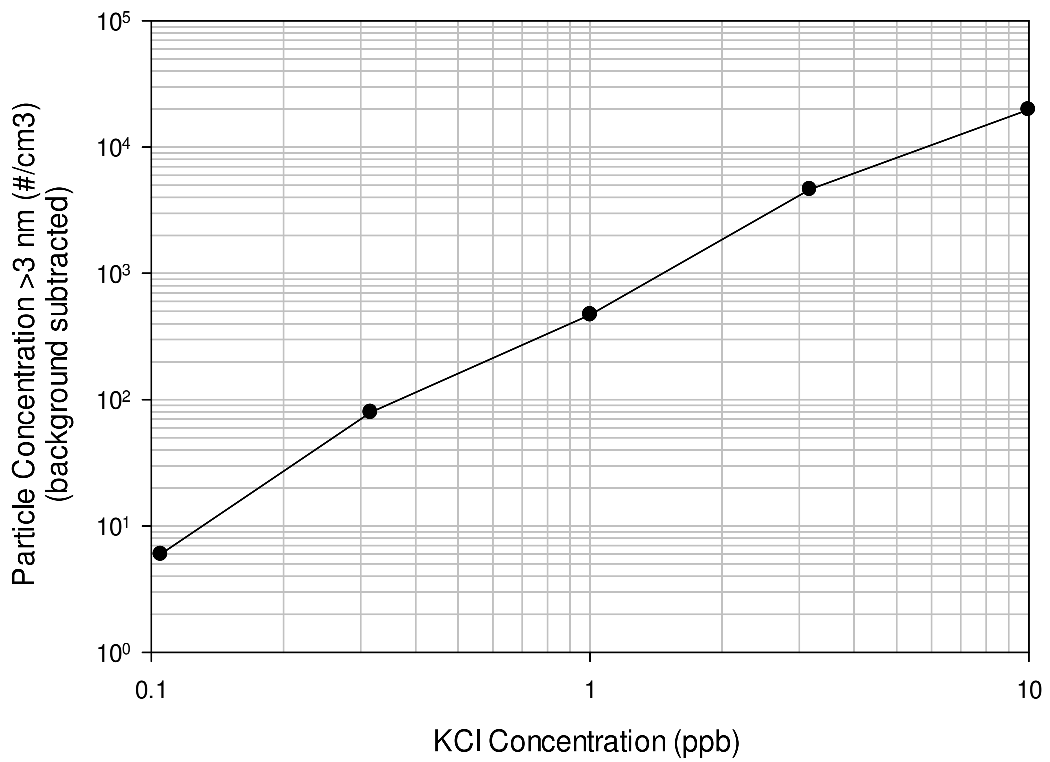


LNS #2: Online dilution method





Measurement of dissolved KCl standard:
Kanomax FMT STPC3 (offline dilution method)



Particle concentration increases non-linearly with increasing KCl concentration.

Real-World Validation

- PFA Extract (from IRDS Critical Components Study)
  - ½” PFA tubing (multiple suppliers) cut to 7-inch lengths
  - Full immersion extraction in pre-conditioned PFA bottles
  - Pre-rinsed with UPW per SEMI F40-0621
  - Extract for 14 days at 85°C
- PVDF Extract (from IRDS Critical Components Study)
  - 1” PVDF tubing (multiple suppliers) supplied in 1.5” lengths
  - Full immersion extraction in pre-conditioned PFA bottles
  - Pre-rinsed with UPW per SEMI F40-0621
  - Extract for 14 days at 85°C
- UPW Mixed Ion Exchange Resin A (from SEMI C79 Task Force PP Study)
  - Prepared per SEMI C93 - Guide for Determining the Quality of Ion Exchange Resin used in Polish Applications of Ultrapure Water System for grab sample analysis (20 bed-volume rinse, 16 hours at 40°C).
- UPW Mixed Ion Exchange Resin B (from SEMI C79 Task Force PP Study)
  - Prepared per SEMI C93 grab sample with elevated and extended hot UPW exposure (20 bed-volume rinse, 7 days at 80°C).

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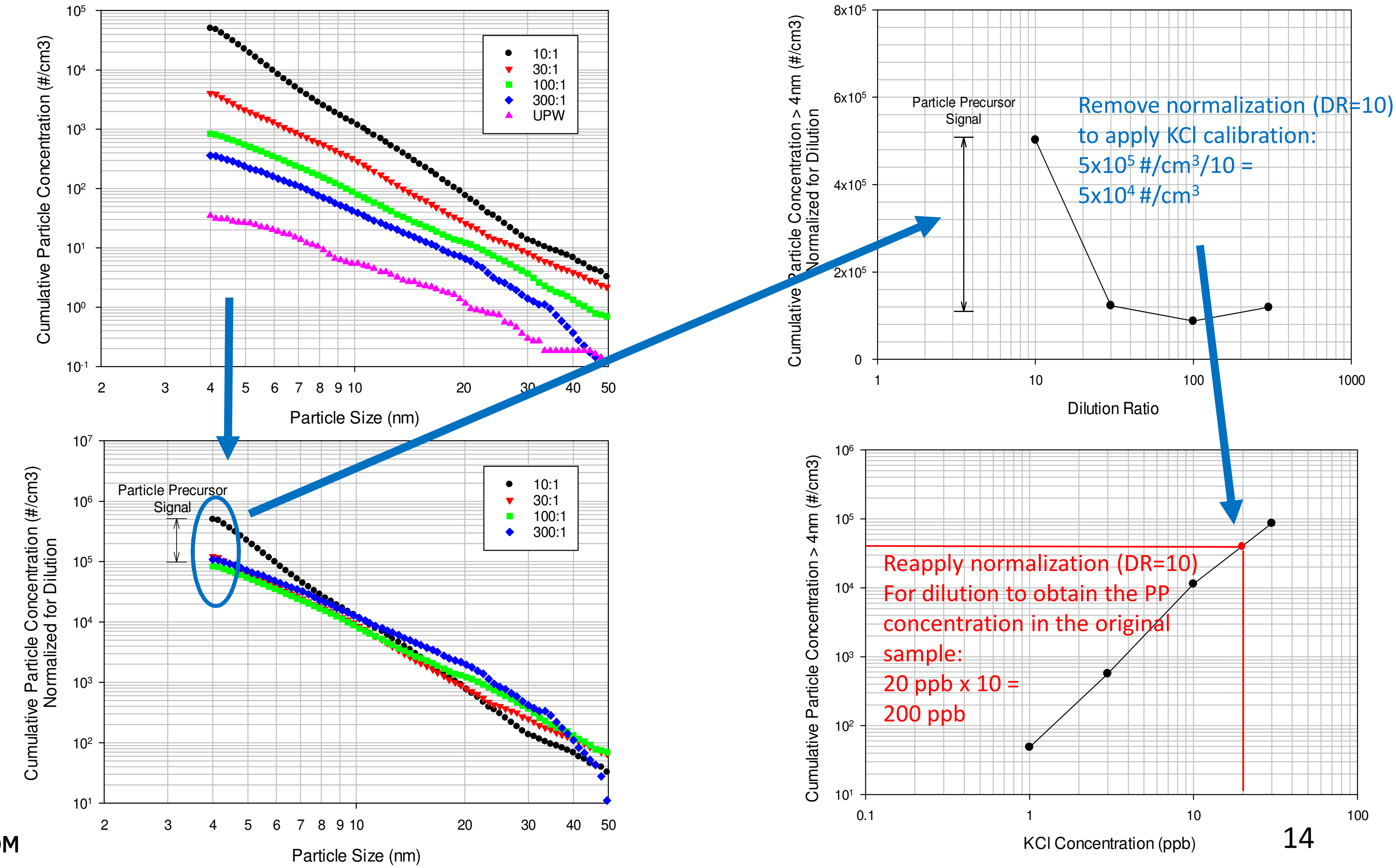
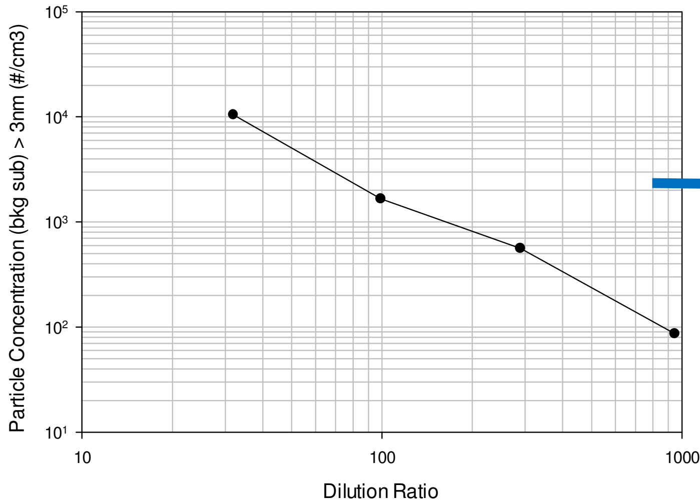
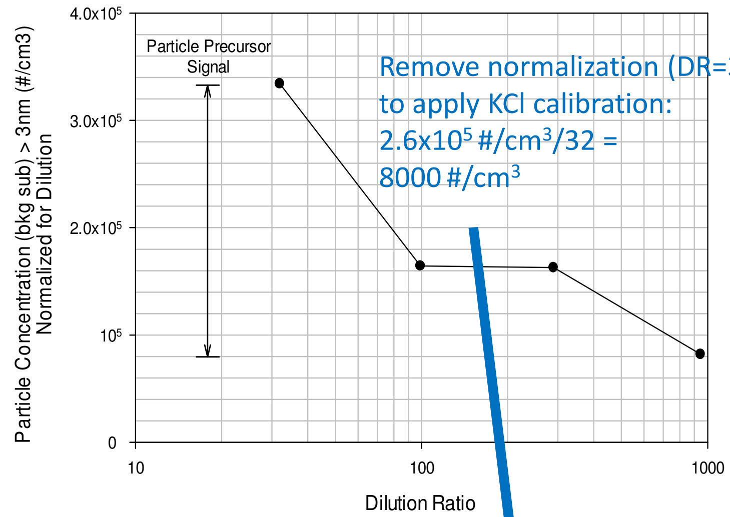
LNS #1: PFA Extract (online): 200 ppb as KCl

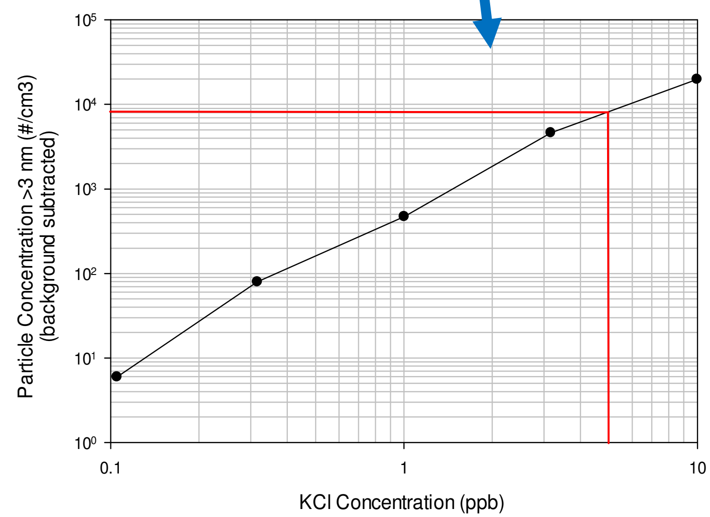


KFMT STPC3: PFA Extract (offline): 160 ppb as KCl





Remove normalization (DR=32) to apply KCl calibration:
$2.6x10^5$ $\#/cm^3/32$ =
8000 $\#/cm^3$



Reapply normalization (DR =32) for dilution to obtain the PP concentration in the original sample:
5 ppb x 32 =
160 ppb

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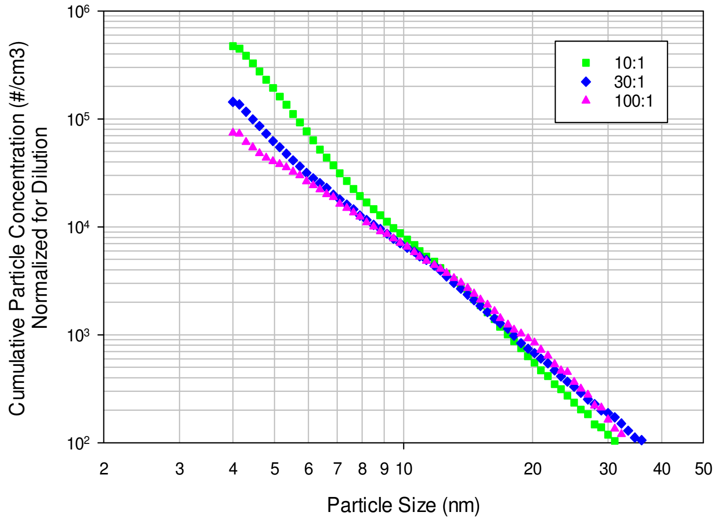
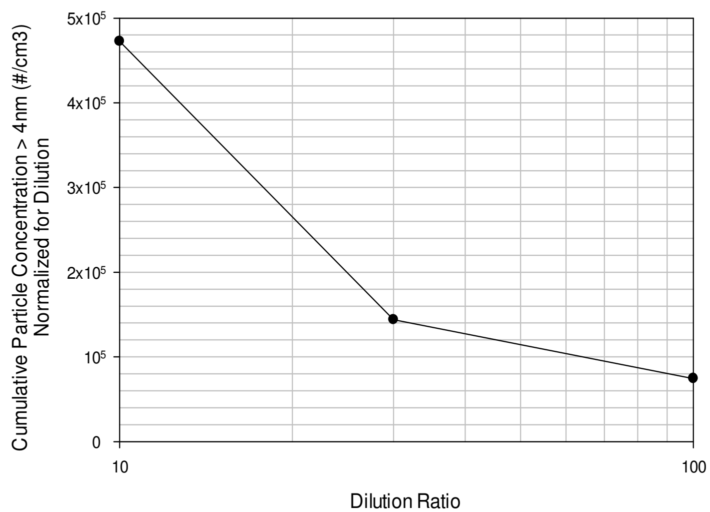
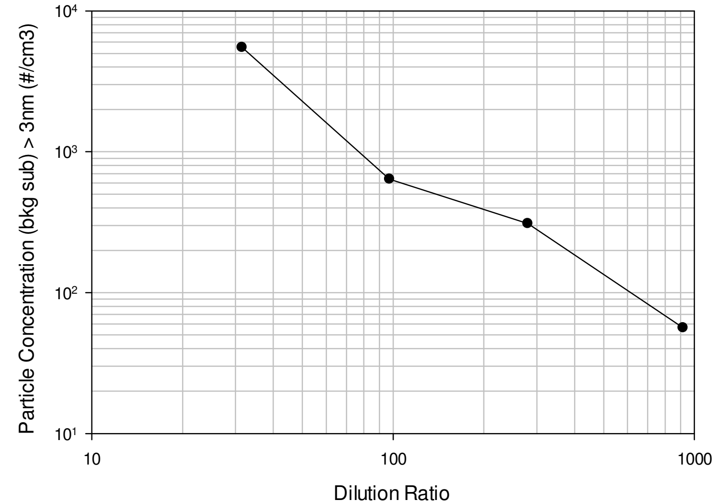
PVDF Extract

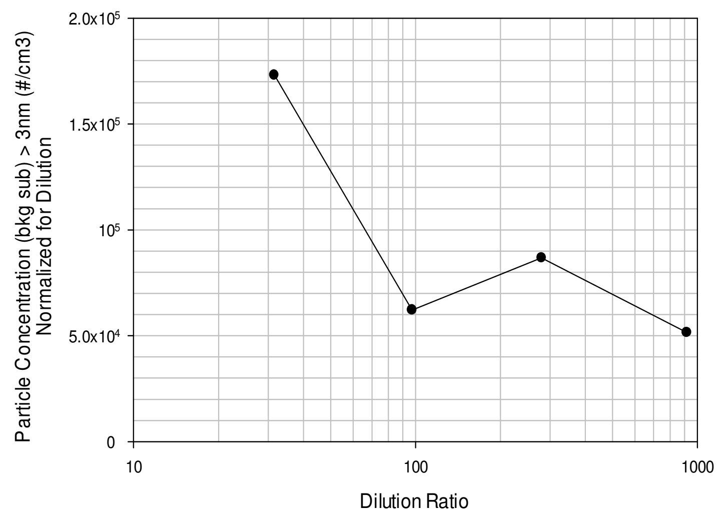
LNS #1 (offline): 150 ppb as KCl





STPC3 (offline): 90 ppb as KCl





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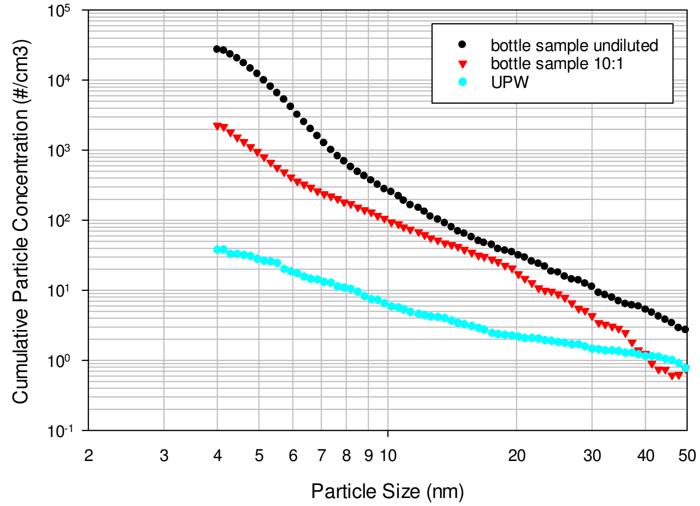
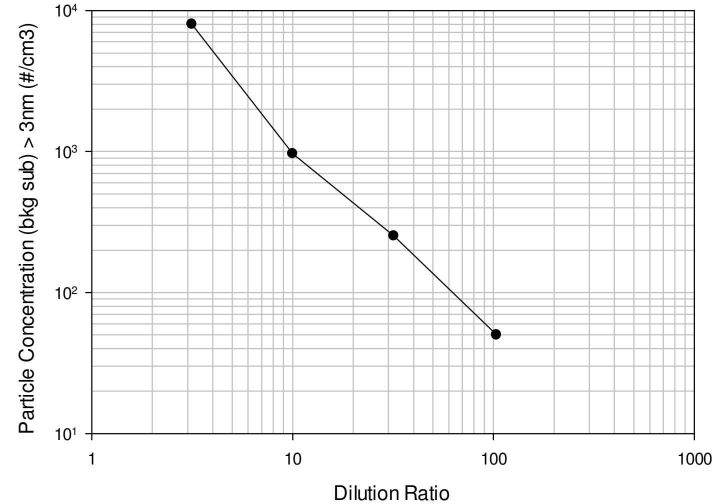
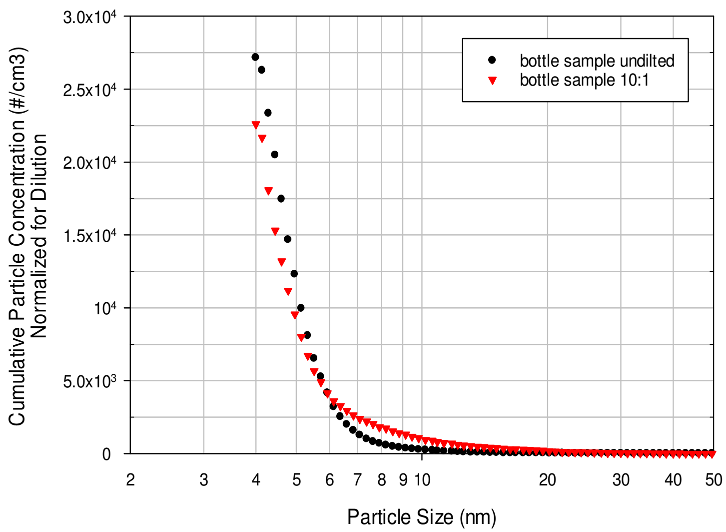
IX Resin A

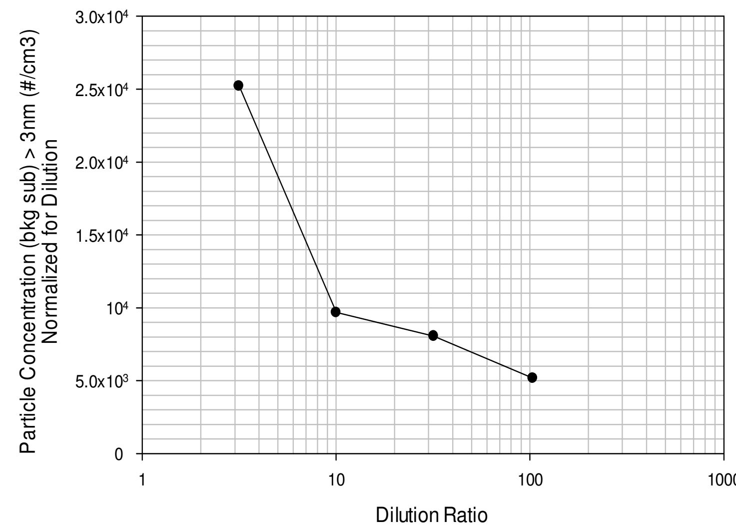
LNS #1 (offline):~7 ppb as KCl (<10 ppb)



STPC3 (offline): 13 ppb as KCl







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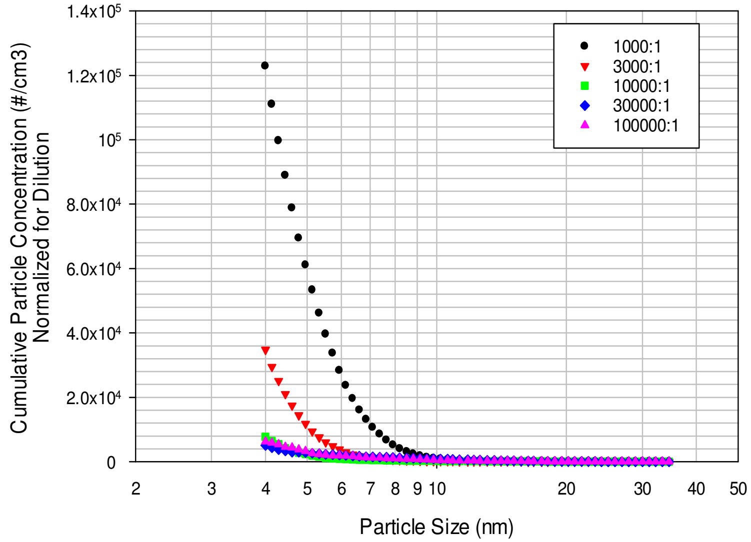
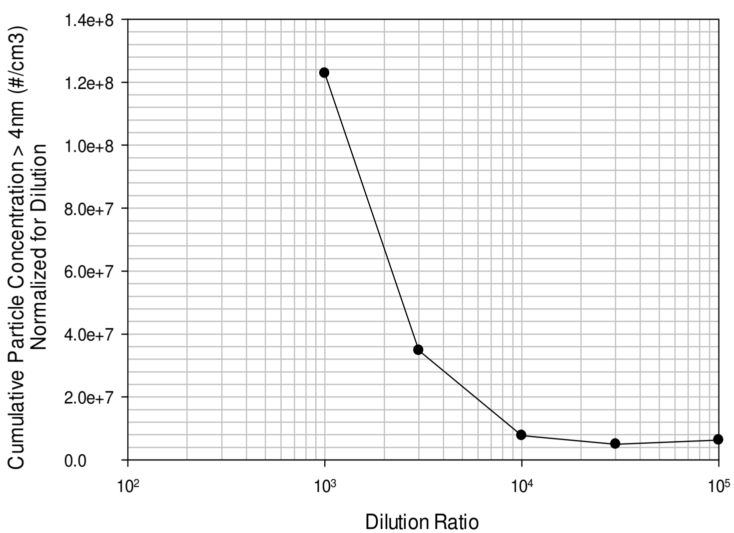
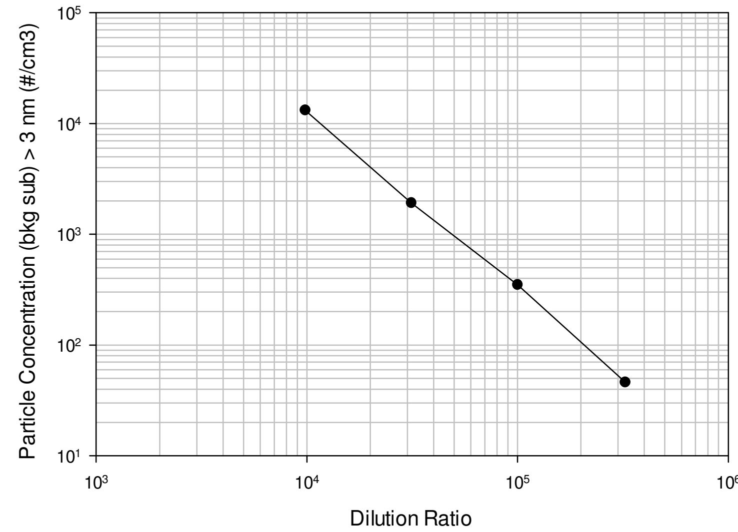
IX Resin B

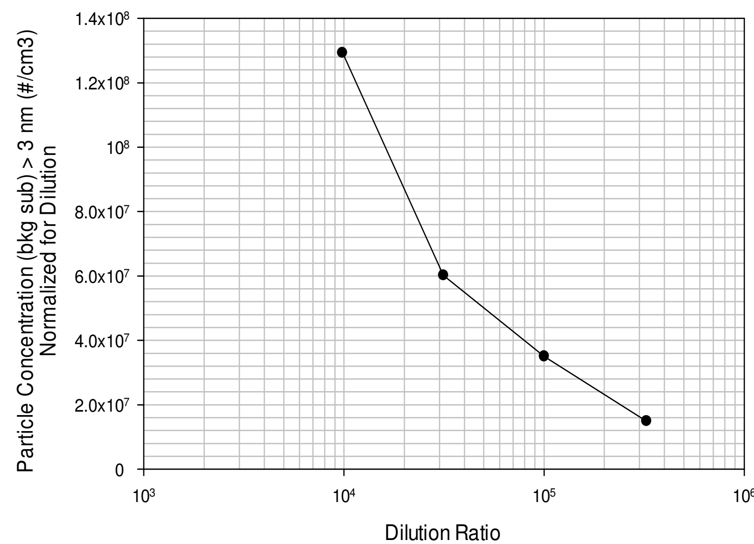
LNS #2 (online): 100 ppm as KCl





STPC3 (offline): 60 ppm as KCl





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Comparison of "Real-World" Extract Sample Results

Mass Concentration (ppb or ppm as KCl)				
	LNS #1		LNS #2	STPC 3 #1
Sample	Online Dilution	Offline Dilution	Online Dilution	Offline Dilution
PFA extract	200 ppb			160 ppb
PVDF extract	100 ppb	150 ppb		90 ppb
IX Resin A		< 10 ppb		13 ppb
IX Resin B			100 ppm	60 ppm

Similar results were obtained for different instruments operating at different locations for each extract measured.

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Criteria for Instrument Qualification: Kanomax FMT STPC3

Step #	Description of Test	Performance Criteria	Result	Meets Criteria?
1 (Instrument Particle Sizing Validation - Traceable Reference Nanomaterial)	Particle size accuracy	7.3 ± 2 nm	N/A	
	Particle size repeatability	RSD < 15%	N/A	
	Concentration effect on particle sizing	± 5%	N/A	
2 (Dissolved Standard Particle Precursor Measurement - Ultrafiltered KCl solution)	Concentration accuracy	± 10%	6.8%	Yes
	Particle size repeatability	RSD < 15%	N/A	
	Particle concentration repeatability	RSD < 15%	10.4%	Yes
	Noise	<30%	2.3%	Yes
	Dynamic range	R^2 >0.95	0.99	Yes
3 (Real-World Demonstration with mixed bed IX resin extract)	Particle size repeatability	RSD < 15%	N/A	
	Particle concentration repeatability	RSD < 15%	11.6%, 8.3%, 3.3%	Yes

Summary

- Demonstrated how to distinguish between native particles and particle precursors in real-world extract samples using 2 different measurement instruments using the SEMI F121 Guide for Evaluating Particle Precursor Metrology.
- Demonstrated the ability to correlate the output of 2 different measurement instruments to various concentrations of a dissolved KCl standard.
- Demonstrated the ability to quantify the particle precursor concentration found in "real-world extracts" as mass concentration of KCl. We found that different instruments measuring the same samples in different locations were able to produce similar results.

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Thanks for your attention.

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