Introducing a 10-nm Particle Counter for Ultrapure Water

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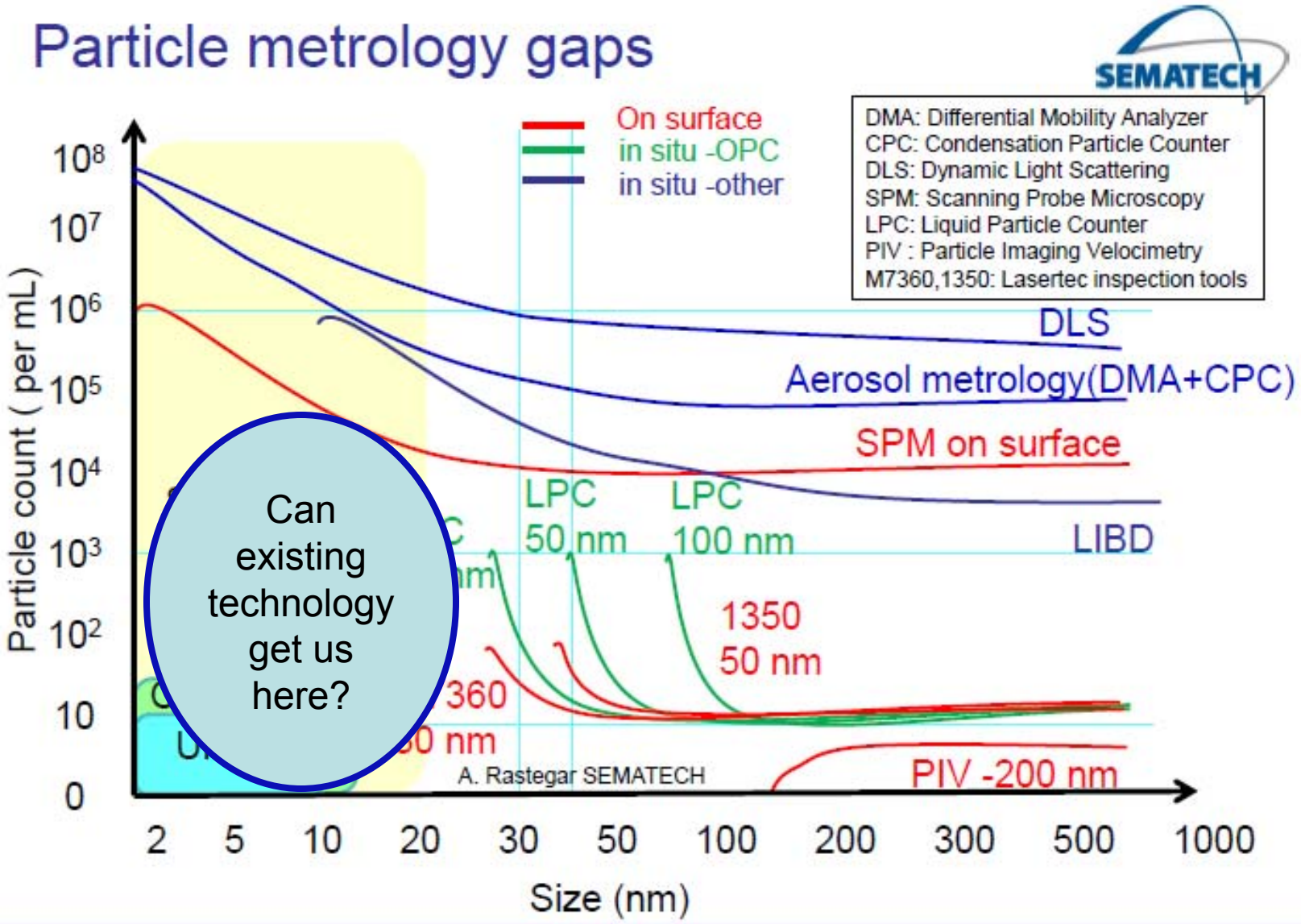
Outline

- Overview of the state of the art in in-situ particle counting
- Overview of particle counting in the gas phase
- Description of threshold particle counting
- Description of the Scanning Threshold Particle Counter (Scanning TPC)
- Review of sample data

Particle metrology gaps

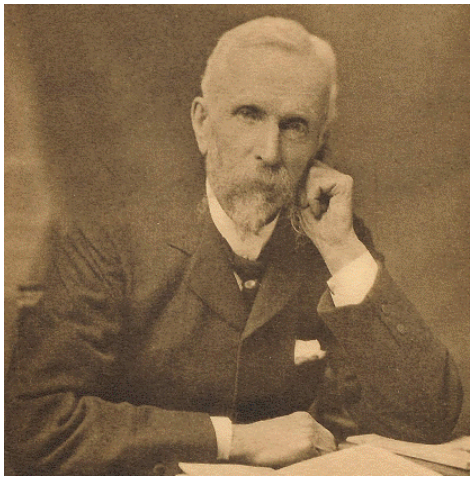


Liquid Particle Counting Technologies

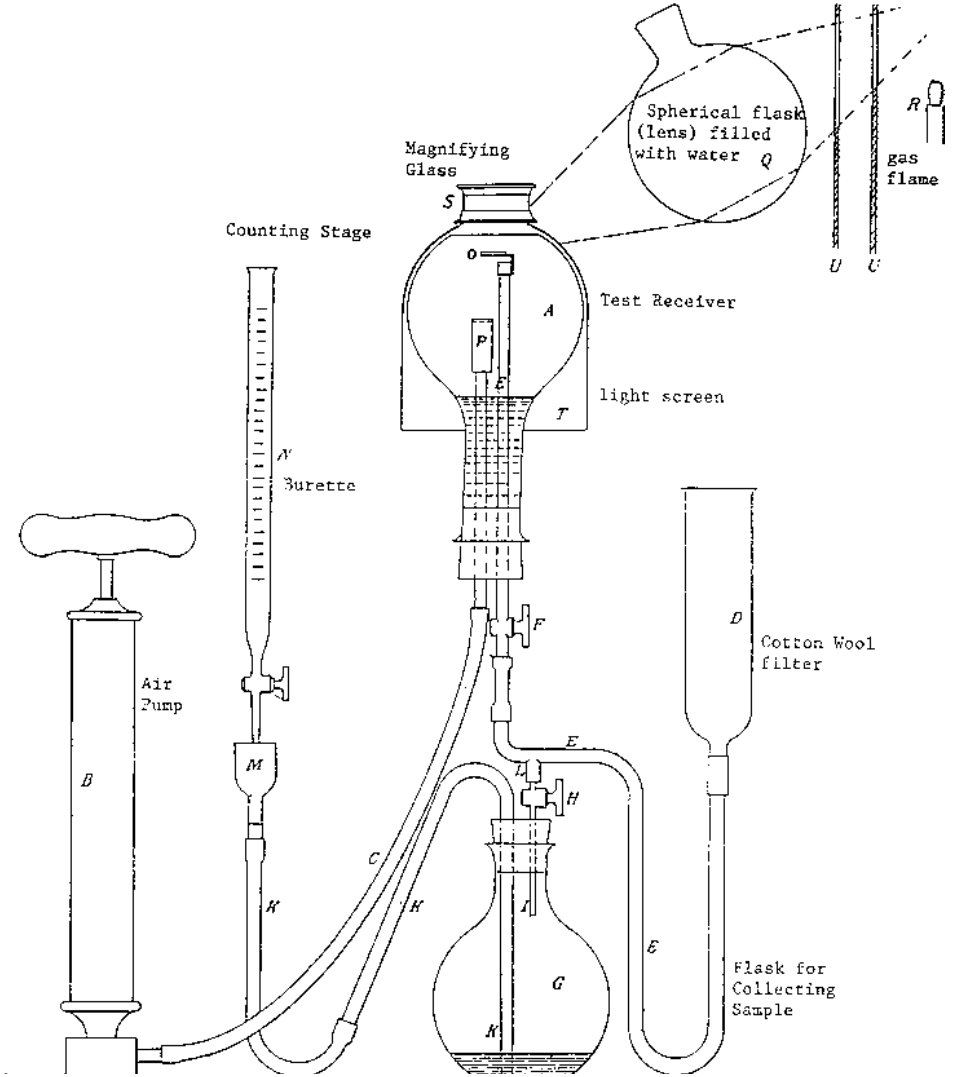
- Dynamic light scattering
  - Requires high concentrations
- Optical particle counting
  - Specified down to 25nm with 3-5% detection efficiency
  - Sensitive to particle composition
- Acoustic Coaxing Induced Microcavitation
  - Able to detect 20nm particles
  - Commercial availability unknown
- Nebulization – Aerosolization – Condensation Particle Counting
  - Promising new technology to provide measurements at previously unattainable size thresholds

Particle analysis in the gas phase

- John Aitken, in 1888, showed a method for detecting and counting optically invisible, nanometer size aerosol particles by enlarging them via heterogeneous condensation of a supersaturated vapor

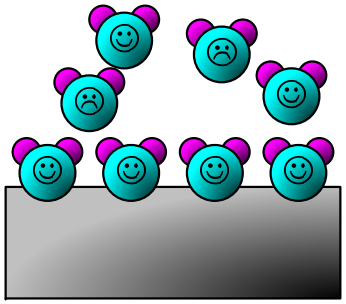


http://www.iara.org/AerosolPioneers.htm

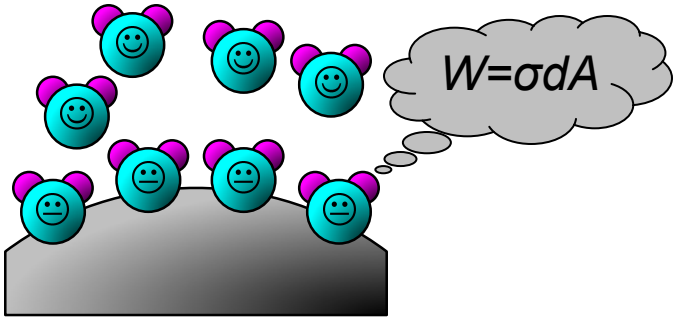


Condensation particle counting…
the science

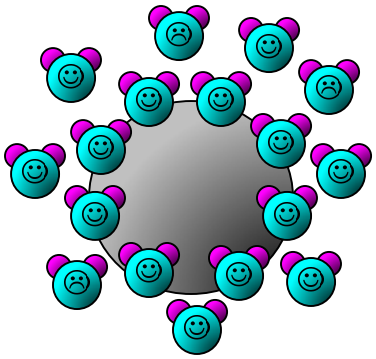
- Supersaturated vapors (relative humidity greater than 100%) are not happy
- The vapor wants to leave the gas phase and condense onto a surface



- Curved surfaces are energetically less favorable due to added surface tension work (Kelvin effect)



- Condensation onto particles is a function of the surface radius and the degree of supersaturation

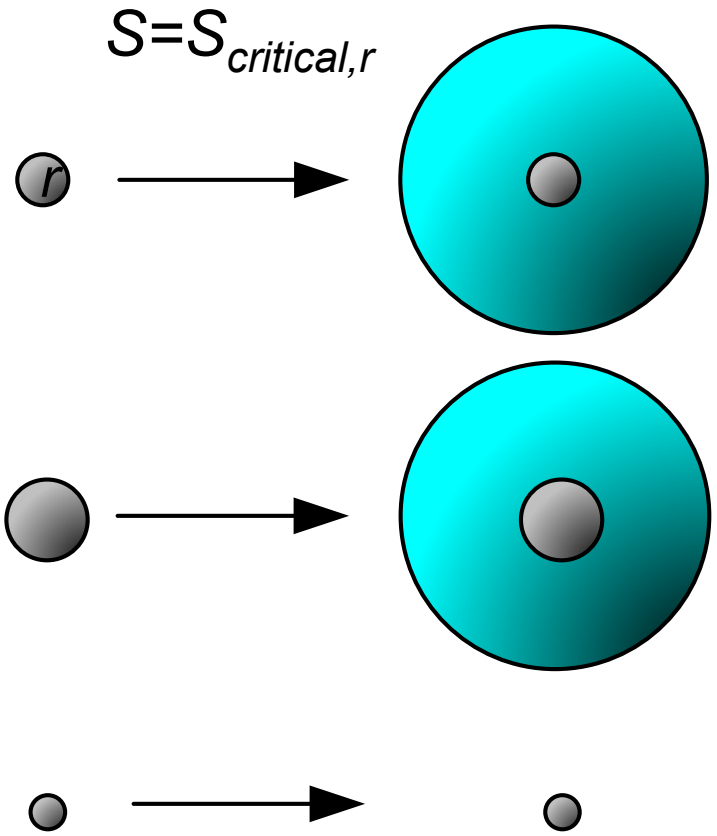


Condensation particle counting
more science

- The particle radius threshold at which a supersaturated vapor will condense onto its surface is given by

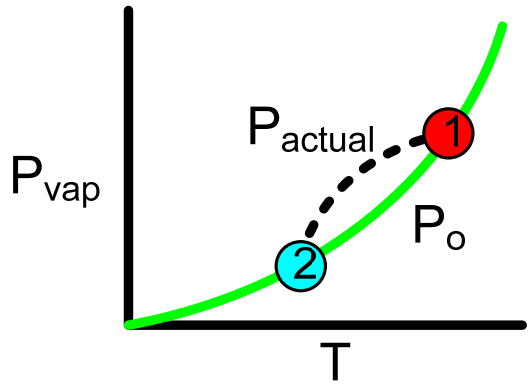
$$r = \frac{-2\gamma V_{vap}}{k_B T} \ln\left(\frac{P_o}{P}\right)$$

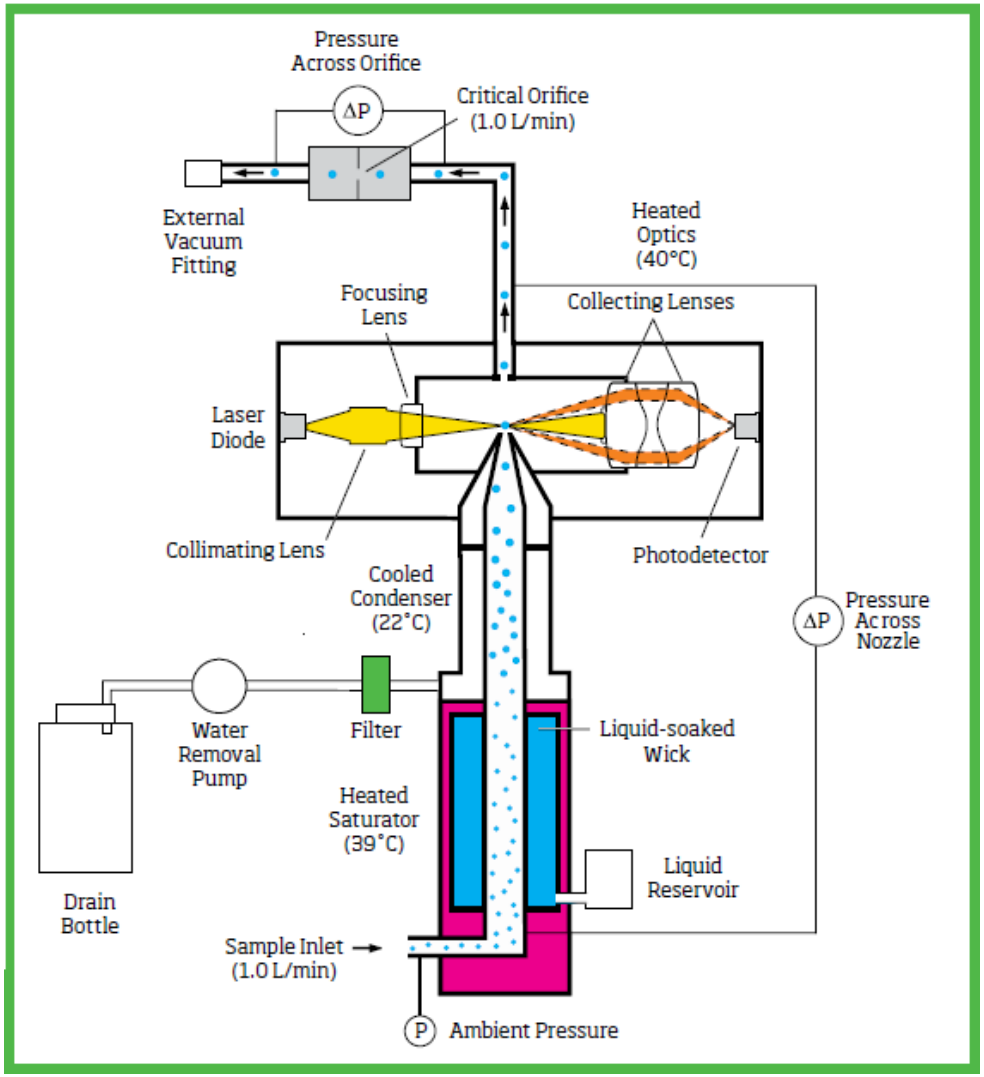
  where γ is the surface tension and V_m is the molecular volume of the condensing liquid. P_o is the vapor pressure above a flat surface of the condensing vapor and P is the actual vapor pressure
- P/P_o is the Saturation Ratio, S
- Varying the degree of S changes the minimum enlarged particle size



Condensation Particle Counting
how it is implemented

- Lewis number is the ratio of the thermal diffusivity of a gas *λ, to the* mass diffusivity of a vapor *D, Le= λ/ D*
- *Le* < 1 (e.g. water in air), cold saturated gas introduced to warm wet walls will drive vapor into gas faster than heat, leading to *S>1*
- *Le* > 1 (e.g. butanol in air), warm saturated gas introduced to cold walls will pull heat out of the gas faster than butanol vapor, leading to S>1

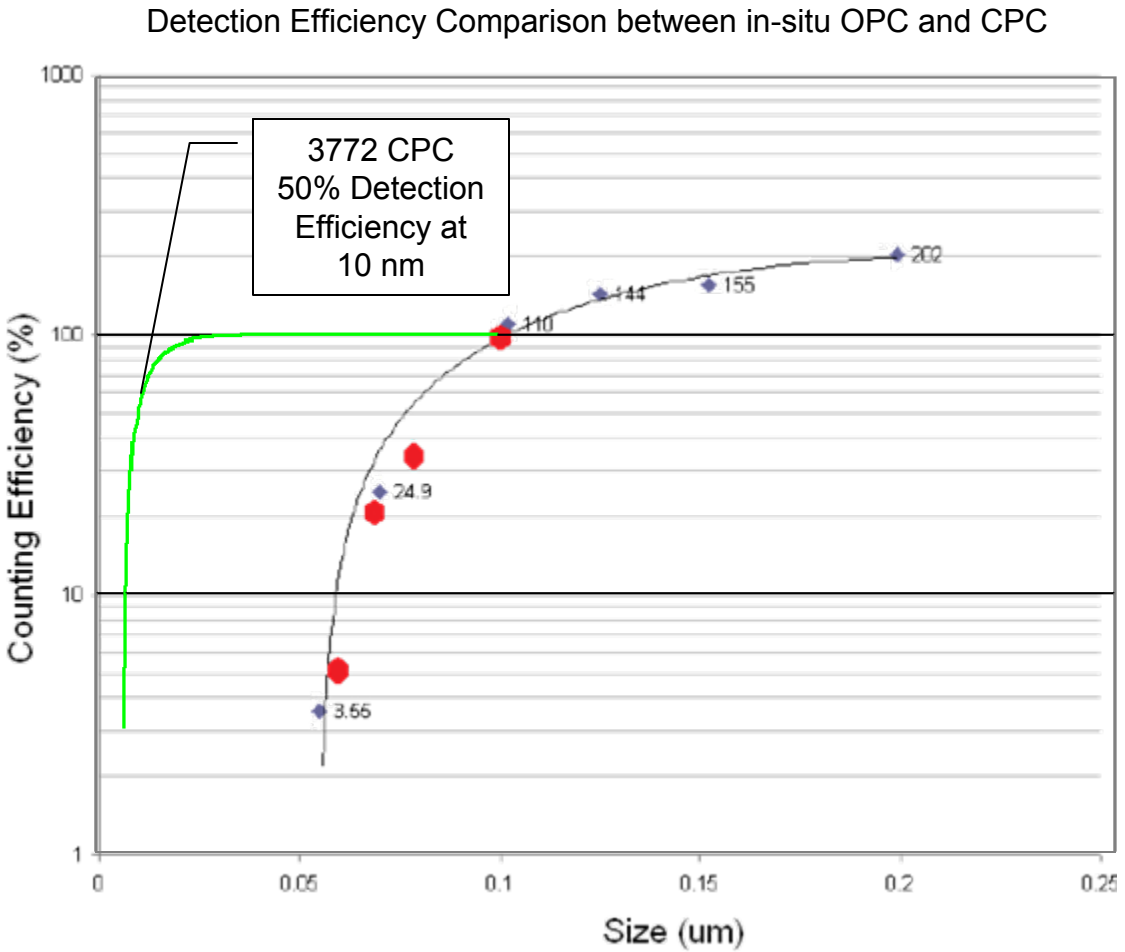




TSI Incorporated 3772 CPC Brochure

A note on detection efficiencies

- OPCs have a shallow detection efficiency curve
- Many OPCs are specified at less than 5% detection efficiency
- Condensation Particle Counters (CPCs) are specified at their 50% cutoff

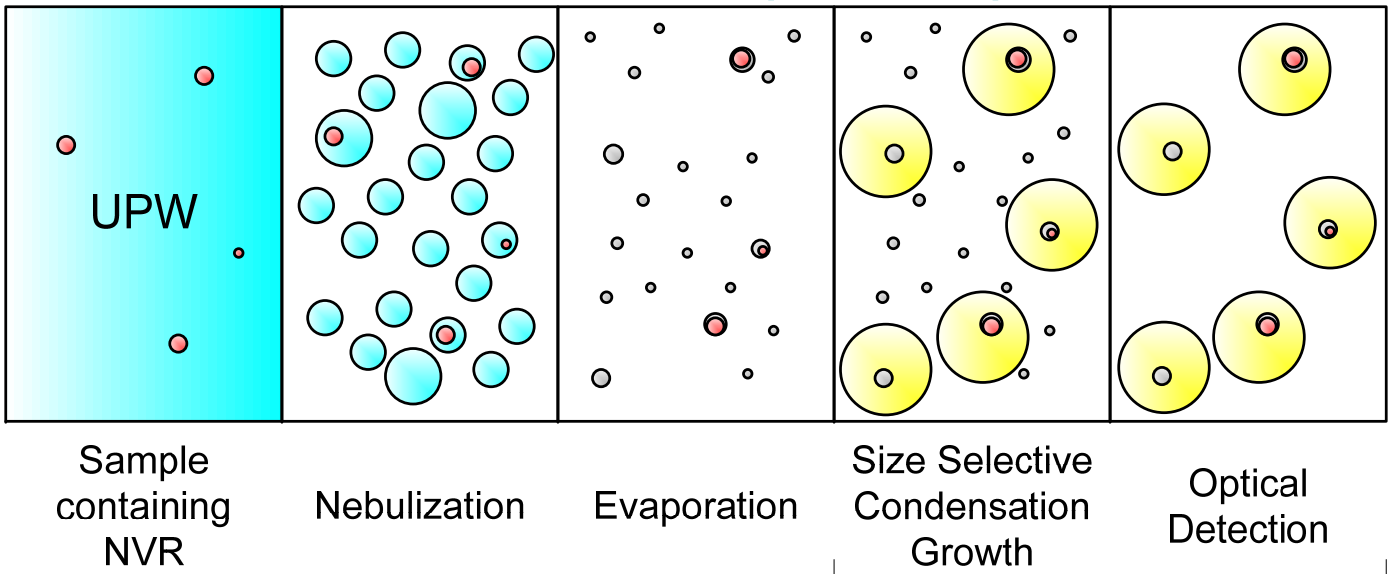


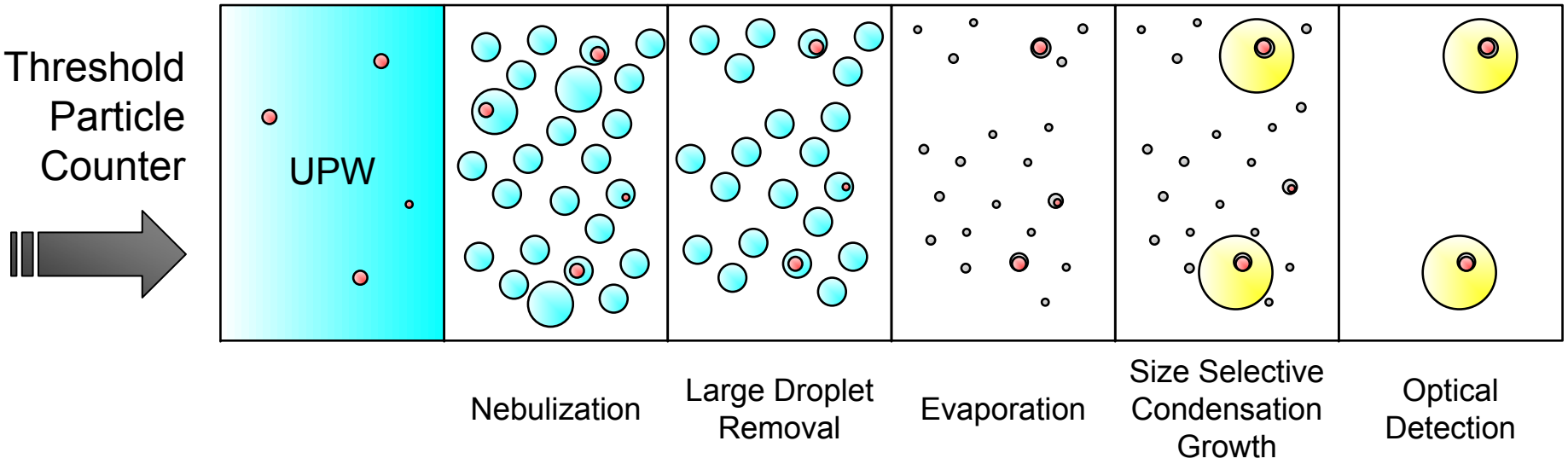
OPC data provided by Particle Measuring Systems.

3772 CPC Data by CT Associates

Scanning Threshold Particle Counter (Scanning TPC)

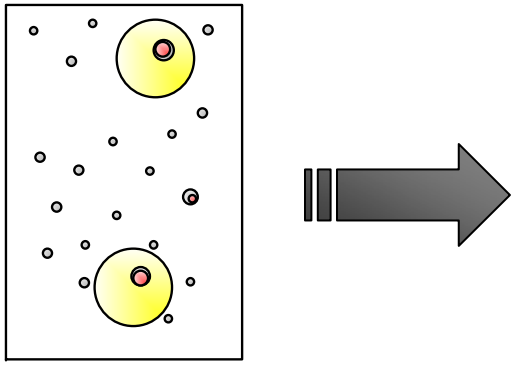
Principle of Operation



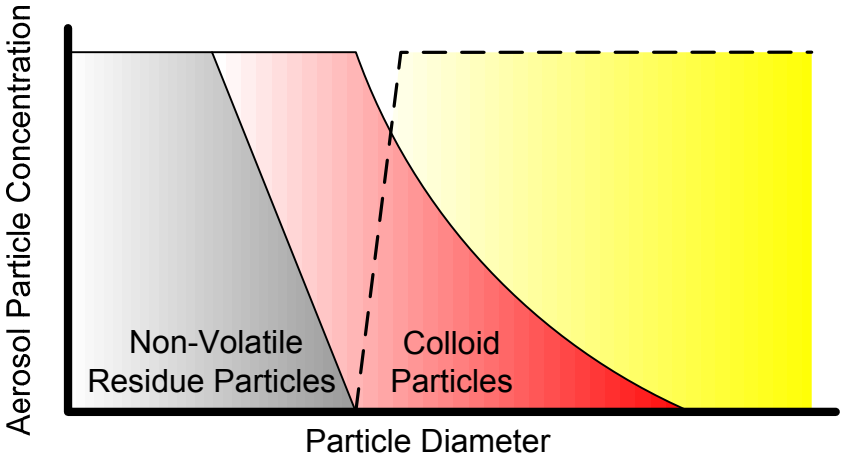


ScanningTPC
Principle of Operation

- Size selective growth
  - All particles entering Condensation Particle Counter (CPC) are optically ‘invisible’
  - Particles larger than a threshold size will grow by orders of magnitude through vapor condensation
  - Threshold size can reach as low as 1 nm!
  - Scanning through different threshold diameters leads to a cumulative size distribution
- Non-volatile residue limits lowest threshold diameter setting

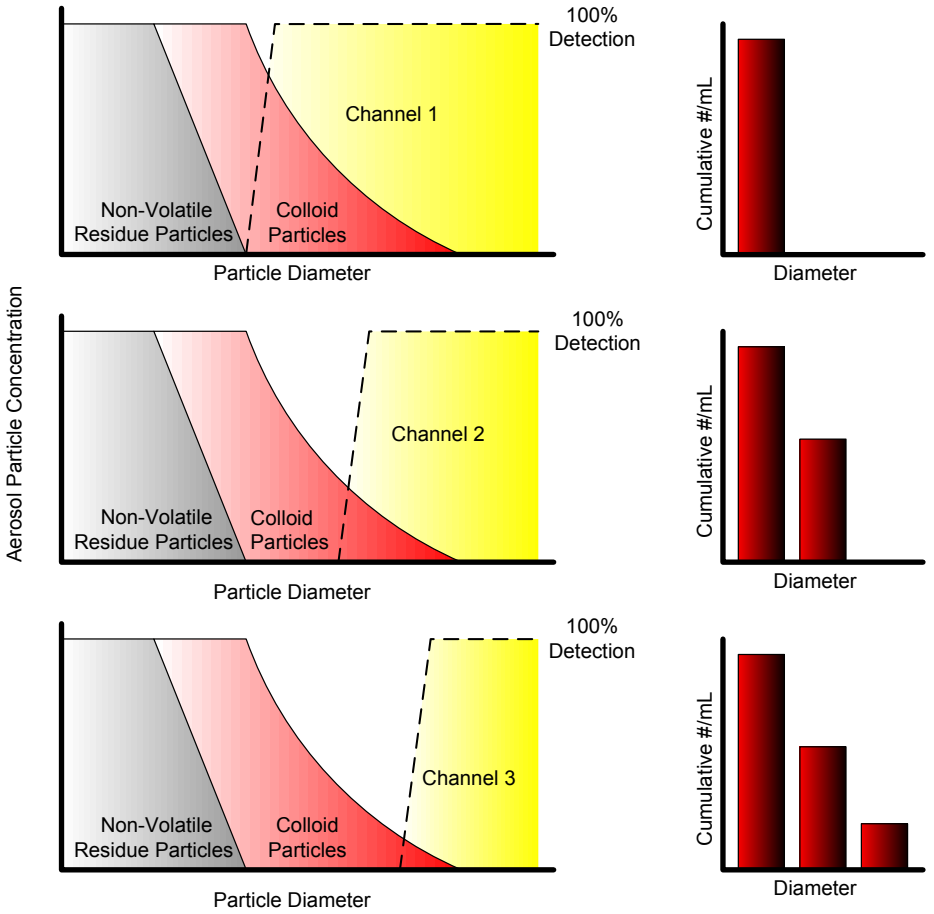


Size Selective Condensation Growth



ScanningTPC
Principle of Operation

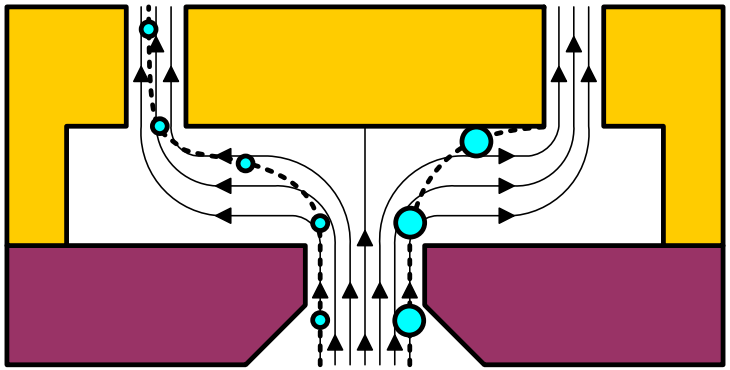
- Stepping threshold diameter range currently 10 nm to 30 nm
- Lower threshold diameters possible with low residue UPW
- Higher threshold diameters also possible

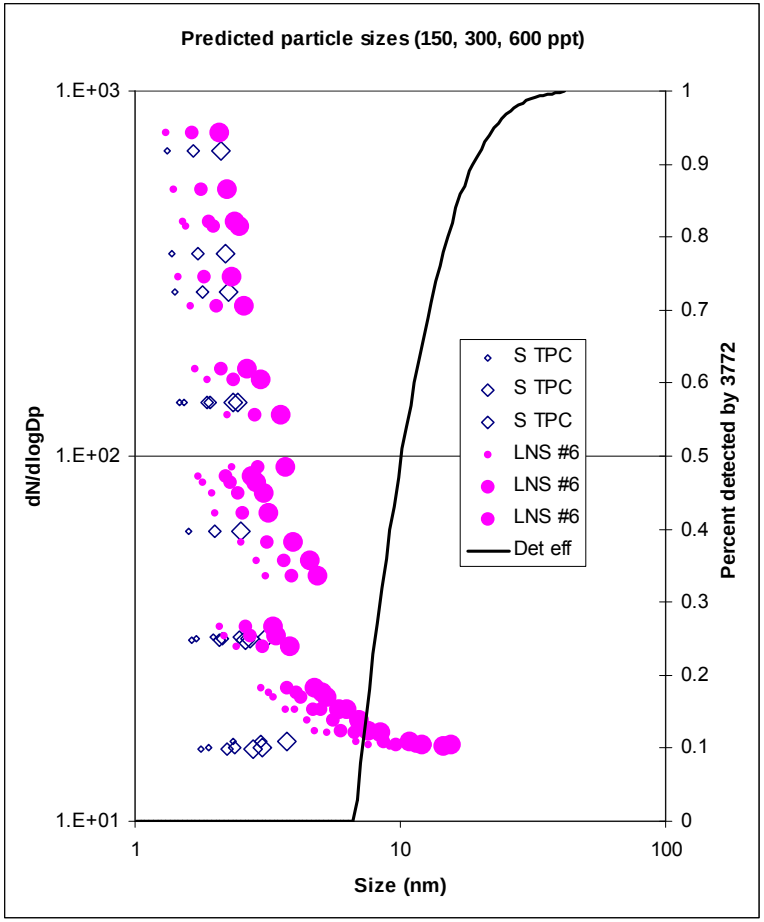


Scanning TPC Nebulizer Design

Secondary large droplet removal incorporated downstream of nebulization region

To Evaporator

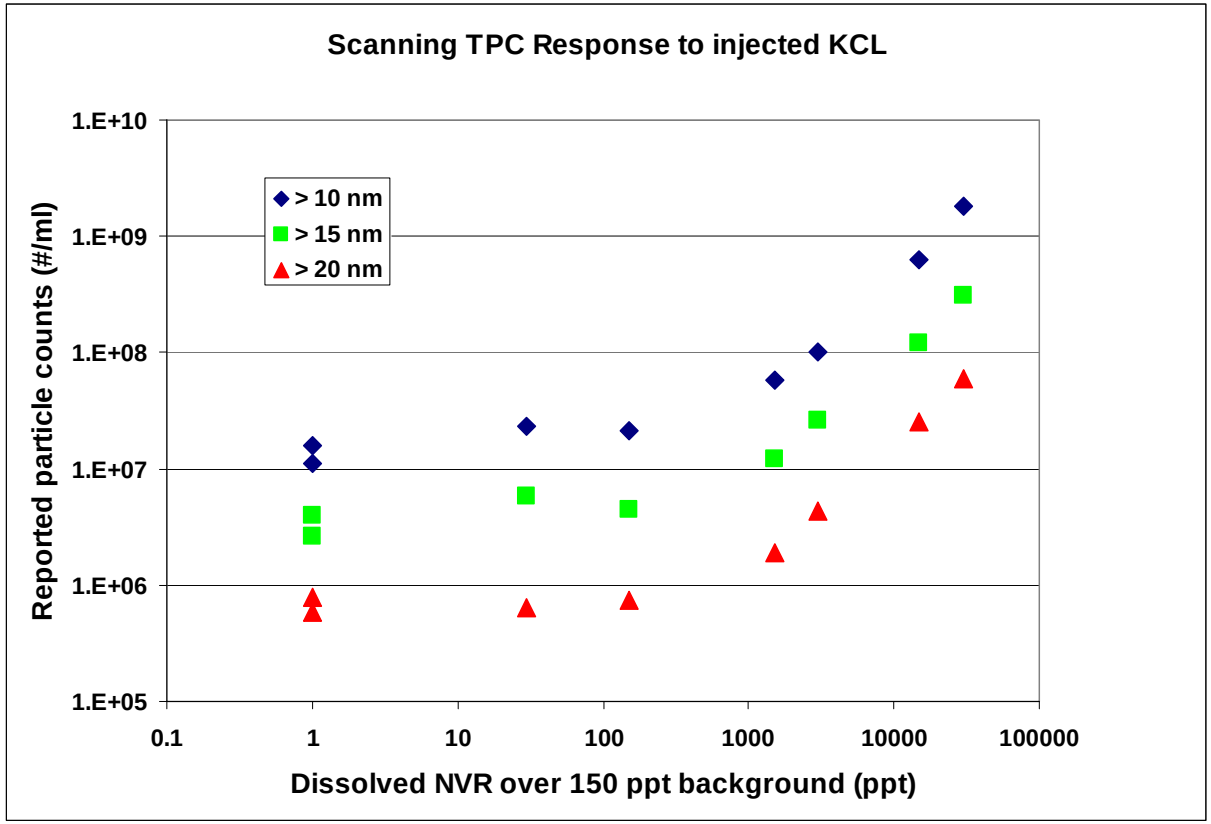




Upper tail of droplet distribution removed to minimize dissolved non-volatile residue particle counts

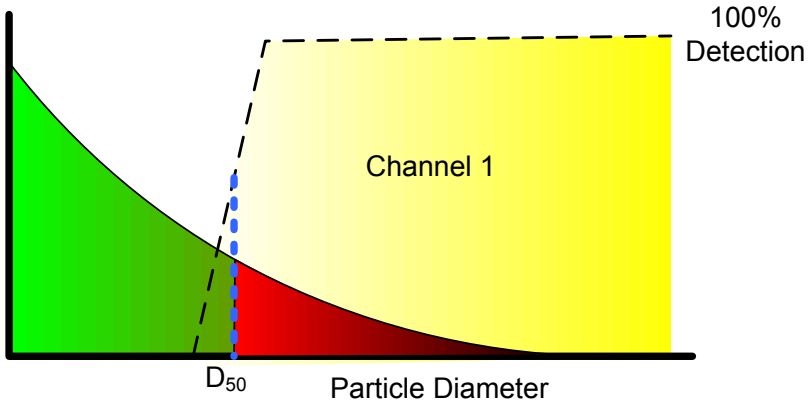
Scanning TPC
Response to dissolved NVR

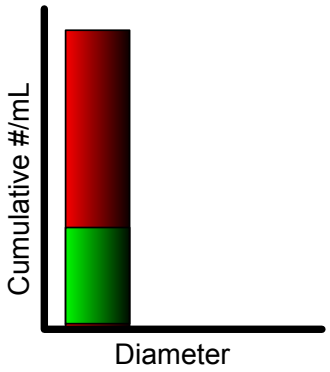
- High levels of DNVR will lead to false particle counts
- Plot shows little sensitivity to low levels of added NVR (some true colloidal particles will be present due to injection system and impurities in the KCL)

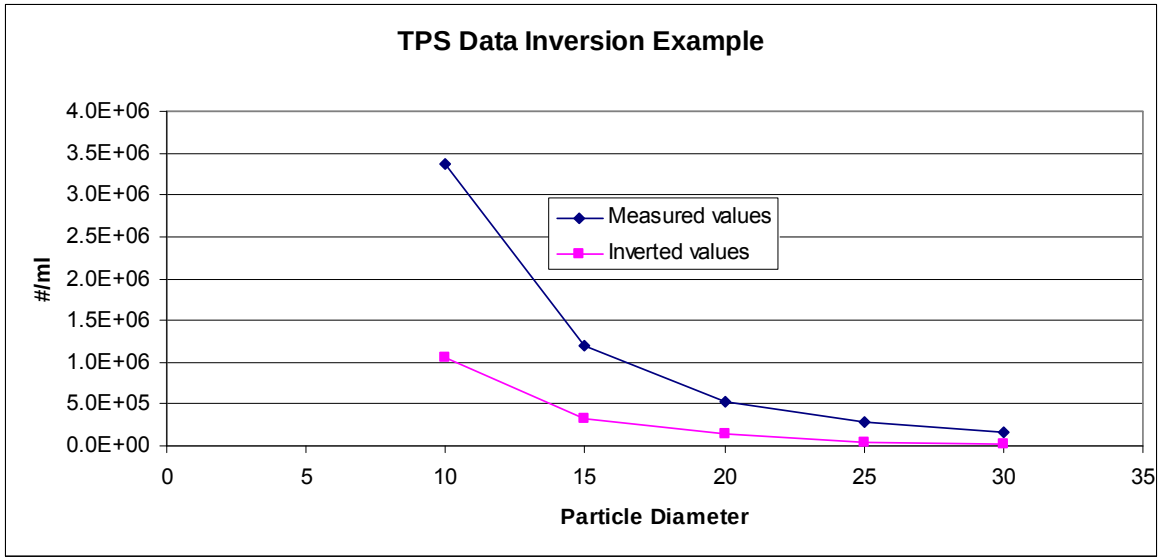


Offline Data Inversion Option

- Monte-Carlo method may be used to estimate the true particle size distribution
- Accounts for shape of detection efficiency curve
- Accounts for shape of droplet size distribution
- Finds best fit of a defined colloid size distribution to match measurements





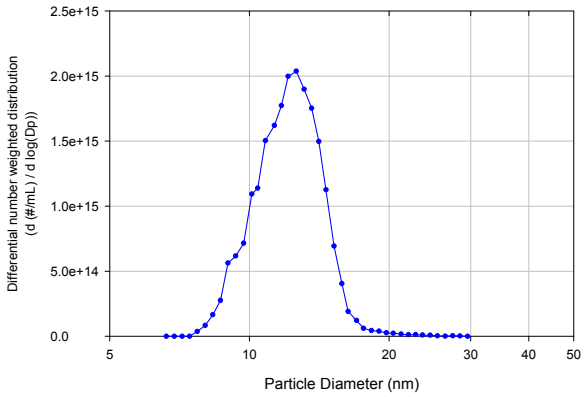


Scanning TPC Sample Data
Colloid Size Standards

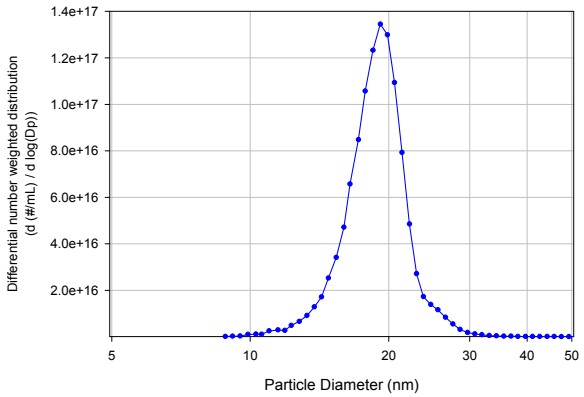
- High concentration solutions of colloidal silica and gold nanoparticles prepared for testing
- Size distributions of each of the solutions has been characterized using Liquid Nanoparticle Sizing (LNS) technology
  - US Patents 8,272,253 and 8,573,034
- Use LiquiTrak ® Precision Diluter to provide high purity, online dilution.

ScanningTPC Sample Data
Colloid Size Standards

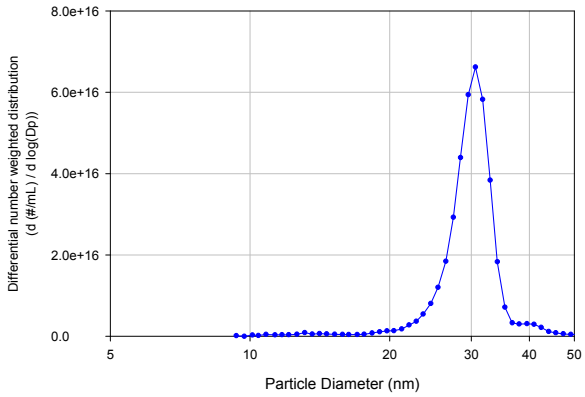
12 nm Silica



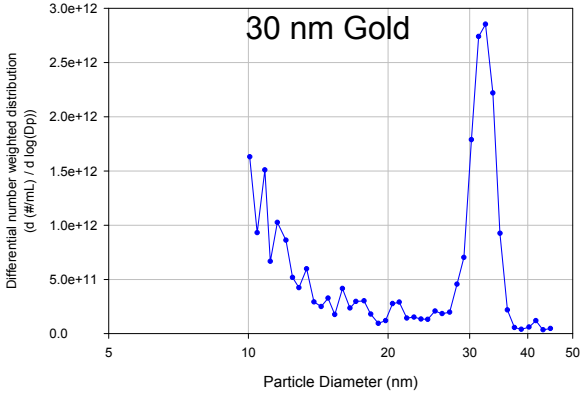
20 nm Silica



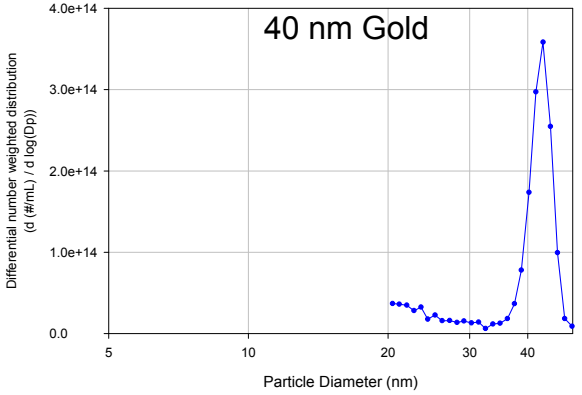
30 nm Silica



30 nm Gold



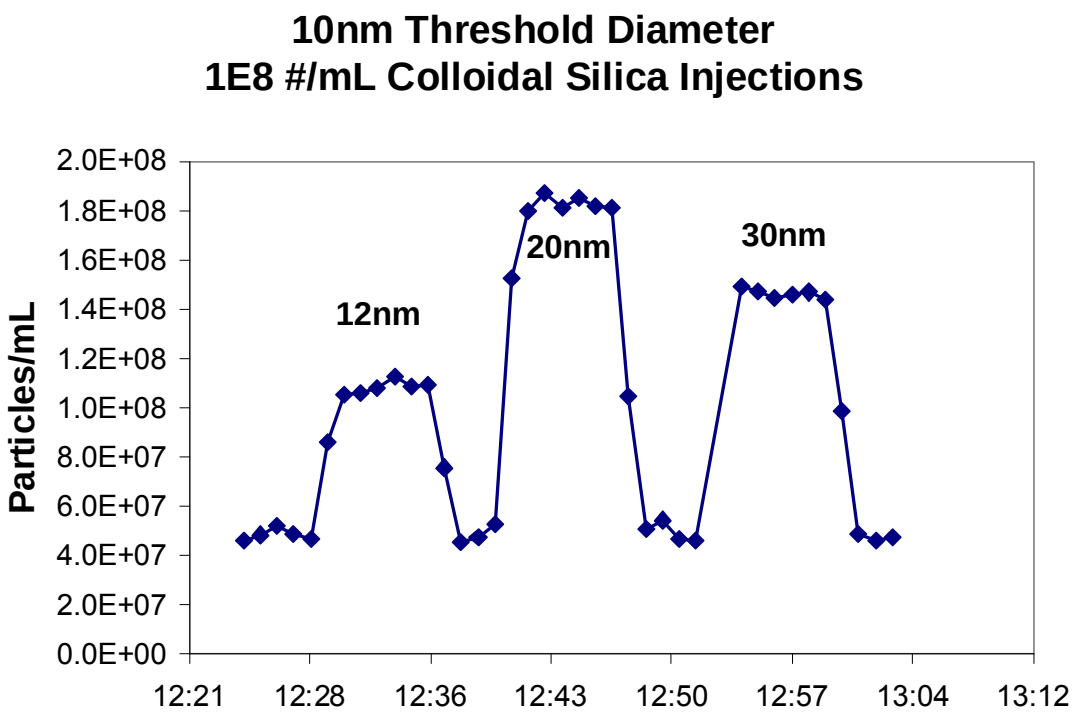
40 nm Gold



Data provided by CT Associates using Liquid Nanoparticle Sizing methodology

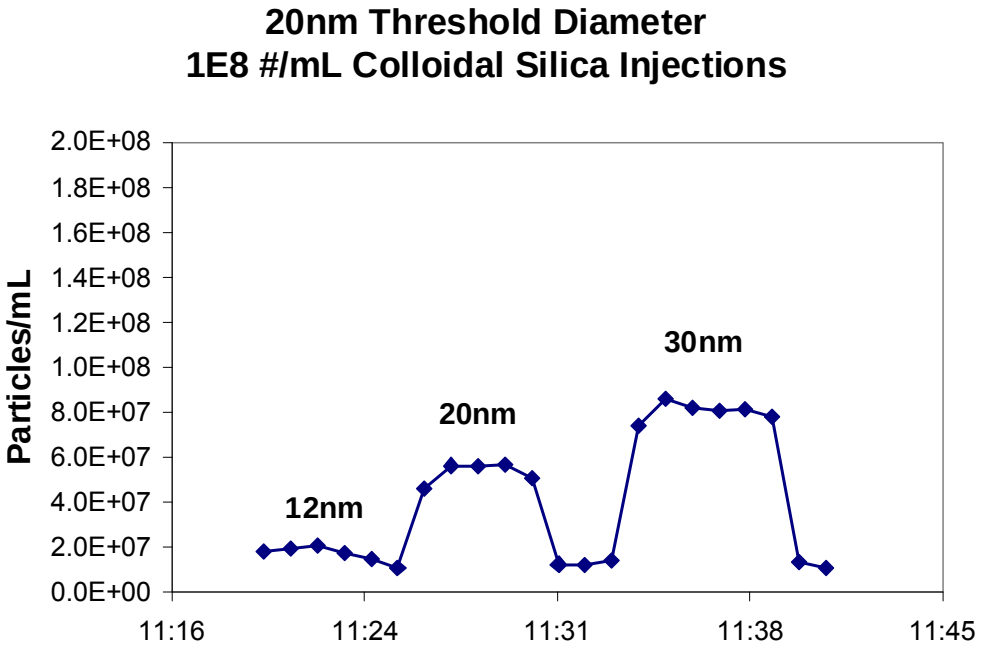
ScanningTPC Sample Data
Sensitivity

- Threshold diameter set to 10nm
- The system responds to all particles with an attenuated response for the 12 nm particles



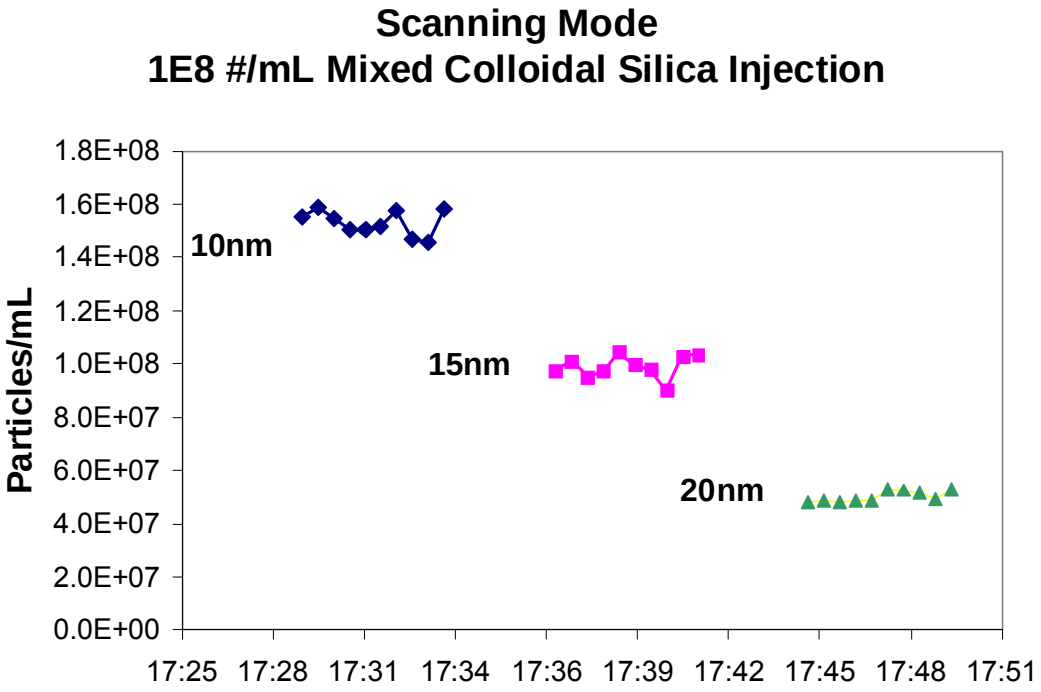
ScanningTPC Demonstration
Sensitivity

- Threshold diameter set to 20nm
- The system shows little or no response to the 12nm particles, attenuated response to the 20nm particles and 80% detection of 30 nm particles.



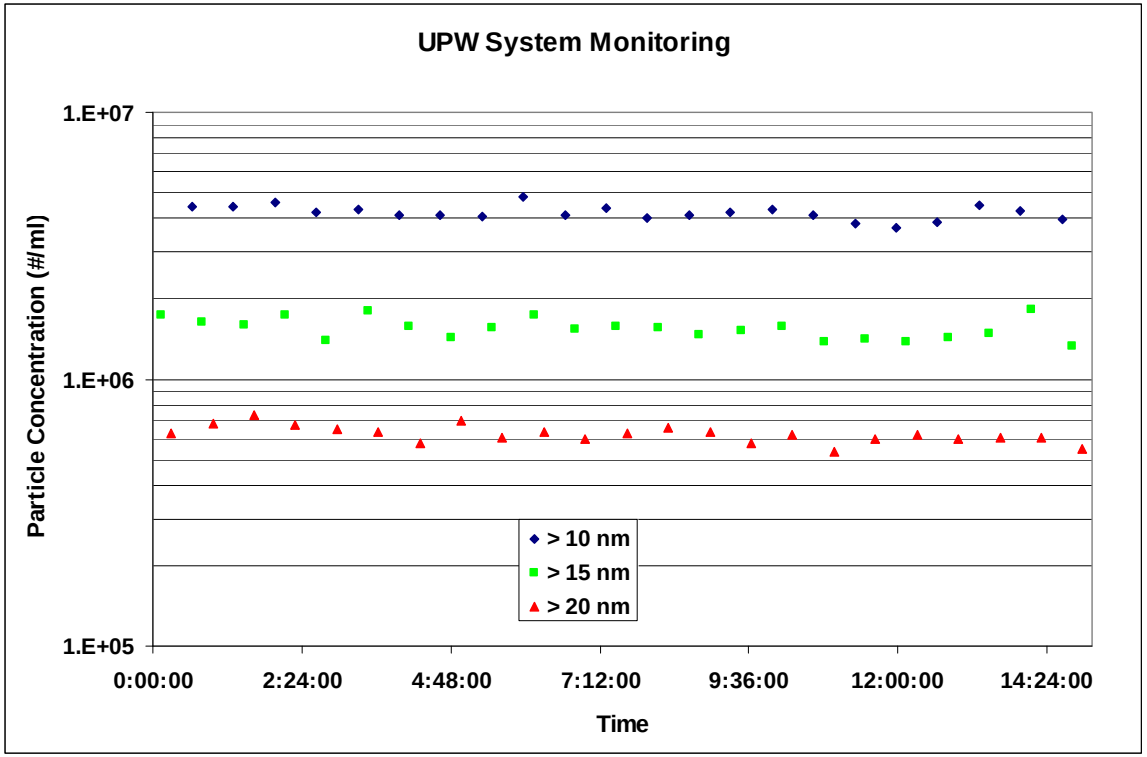
ScanningTPC Sample Data
Scanning Operation

- Operated TPC in mode that steps through threshold particle diameters
- Mixture of 12, 20, and 30 nm particles at equal concentrations



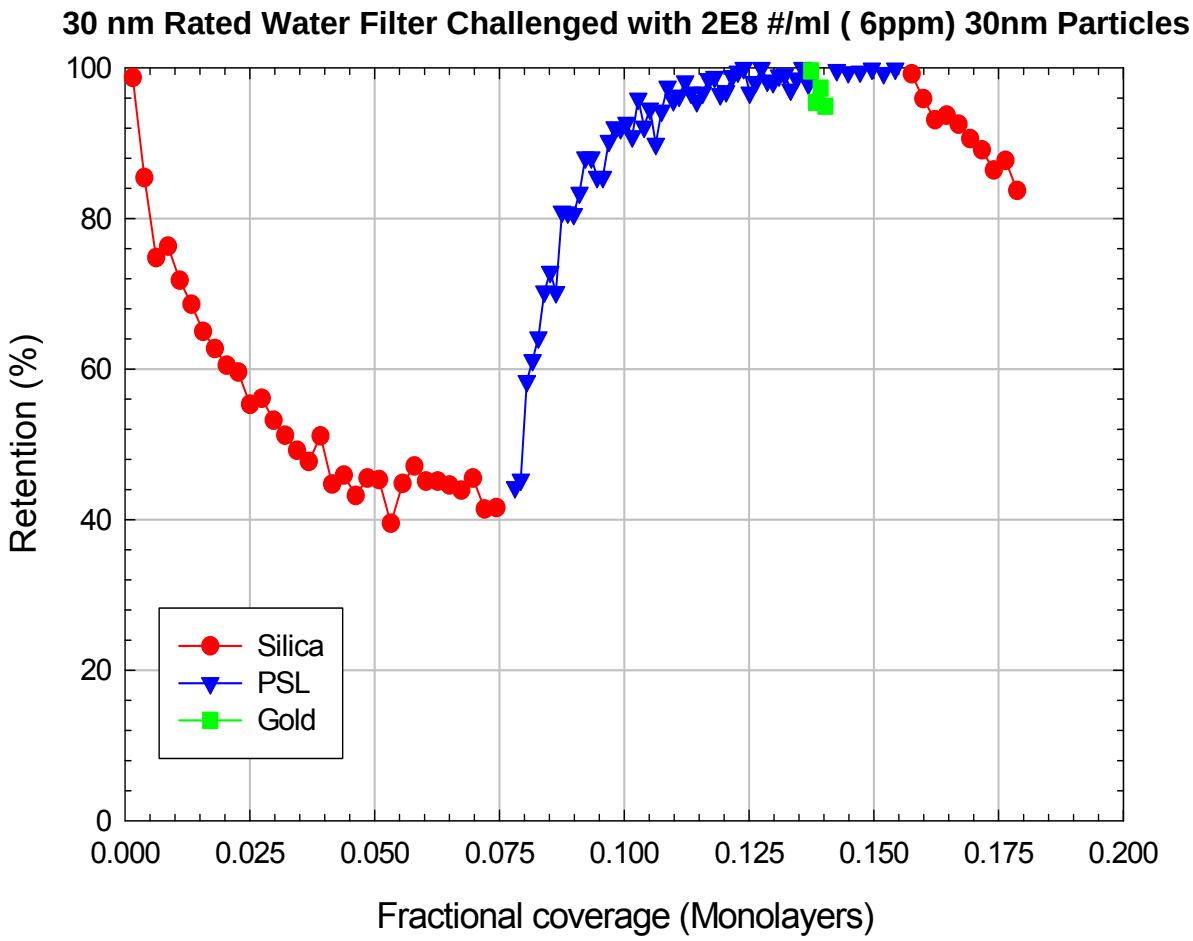
ScanningTPC Sample Data
UPW Monitoring

- Instrument installed on small scale, semiconductor grade UPW system at CT Associates
- Good stability observed
- Distribution of particle sizes apparent
- Large scale UPW systems ~4X lower



Scanning TPC Filter Testing

- Single channel mode >10 nm
- Shows material dependent retention of 30 nm particles



Scanning TPC
Pros and Cons

- Pros
  - Particle composition independence
  - No dependence on refractive index or particle shape
  - Wide dynamic range
- Cons
  - Low sample volume rate (~1 µl / min)
  - Requires Butanol as a consumable
  - Lower threshold limit is sensitive to amount of dissolved non volatile residue

Further development

- Increase inspection volume
- Further characterization of operating parameters
- Refinement and automation of optional offline data inversion software

FMT Model 1000
LiquiTrak® Scanning TPC

- Internal data logging
- Touchscreen display
  - Set nebulizer temperatures
  - Monitor and log pressures and temperatures
  - Adjust minimum detected particle size
  - Display particle counter concentration and trend lines
  - Set scan rates
- Patent pending

Thank you

Questions?

Bibliography

Grant DC (2008). "A New Method for Determining the Size Distribution of the Working Particles in CMP Slurries," presented at the 2008 CMP Users Conference, sponsored by Levitronix.

Don Grant and Uwe Beuscher (2009), "Measurement of Sub-50 nm Particle Retention by UPW Filters", Ultrapure Water Journal, 26(11):34-40.

Blackford D and DC Grant (2009). "A proposal for measuring 20-nm particles in high-purity water using a new technology," Ultrapure Water, January 2009.

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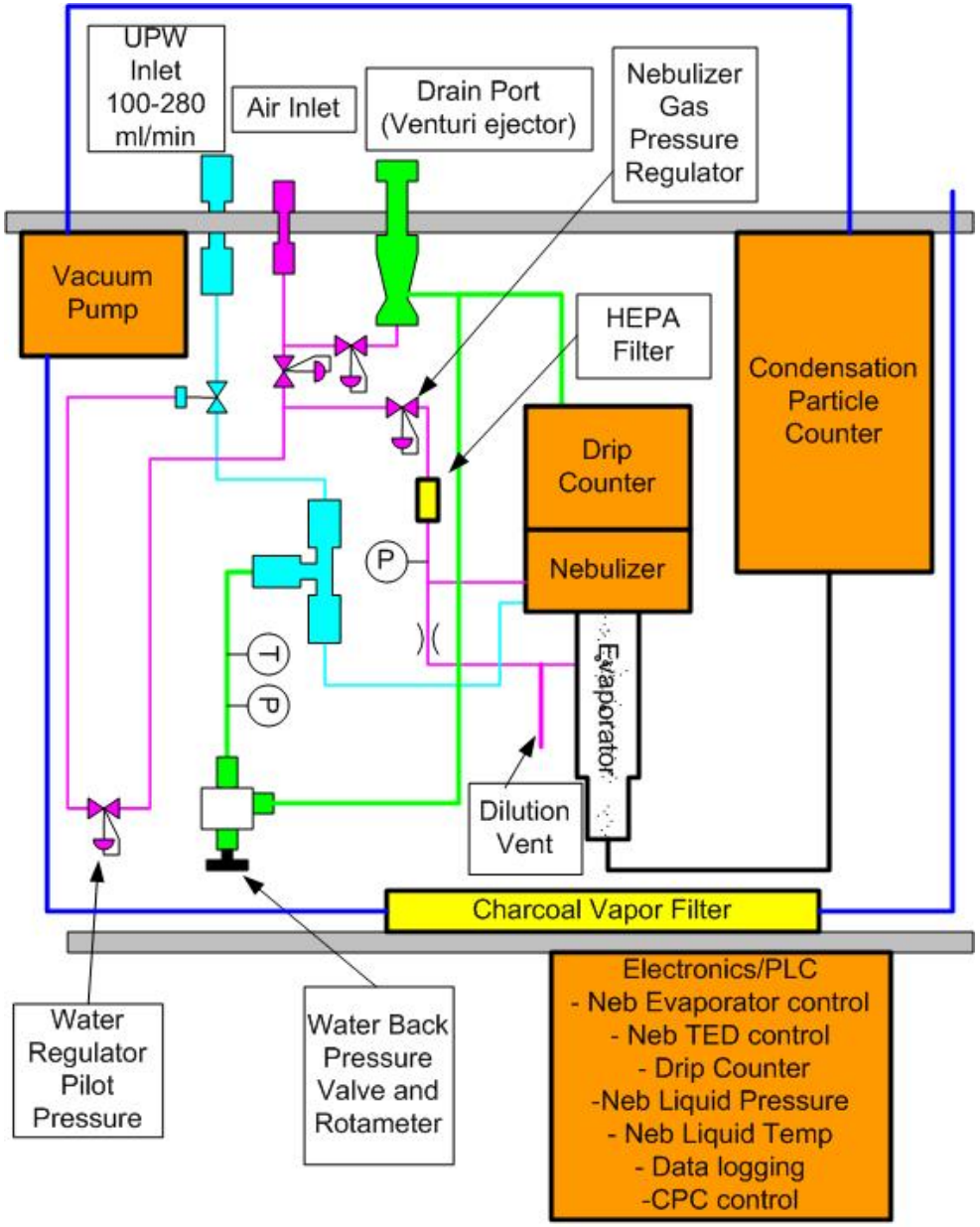
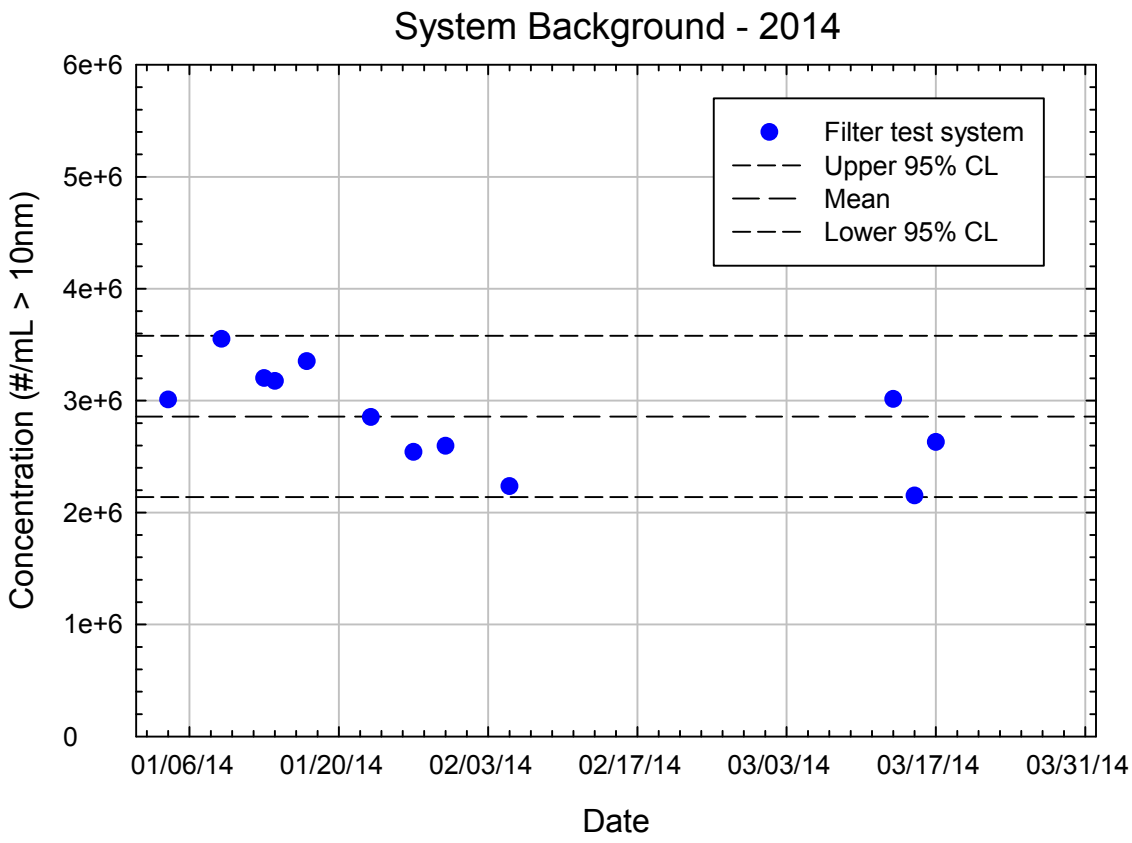
Rastegar, A (2013). "Particle Control Challenges in UPW ", presented at 2013 UPW Micro Conference

Patents US 8,272,253; US 8,573,034; US 7,852,465; Other patents pending

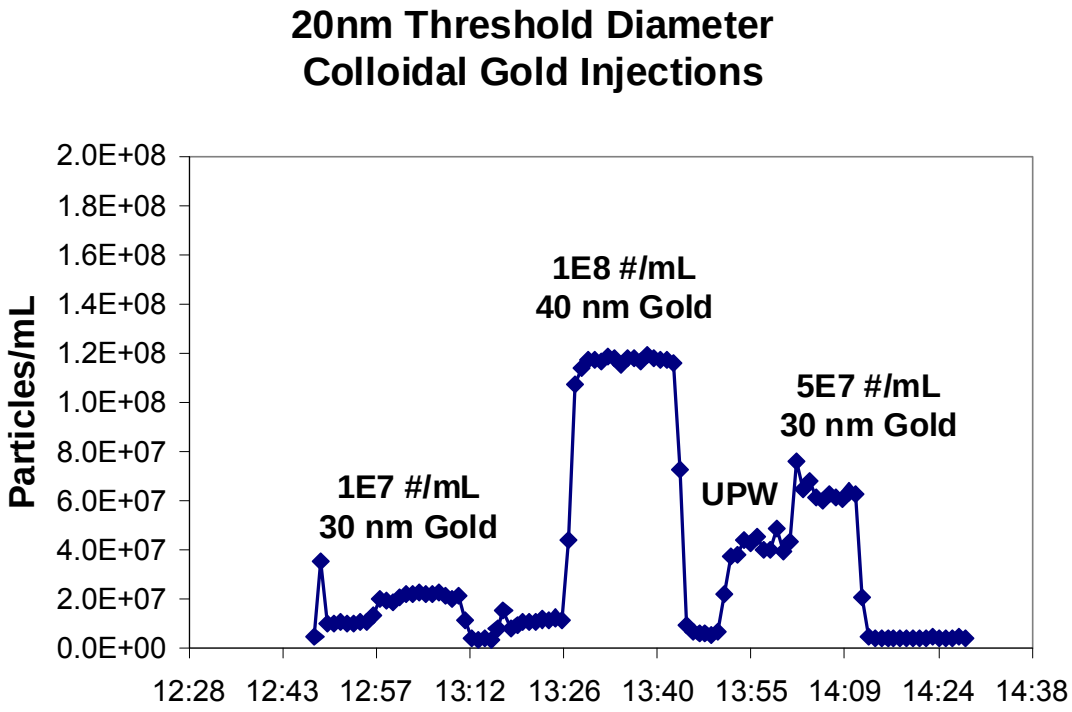

UPW Inlet 100-280 ml/min
Air Inlet
Drain Port (Venturi ejector)
Nebulizer Gas Pressure Regulator
Vacuum Pump
HEPA Filter
Condensation Particle Counter
Drip Counter
Nebulizer
P
T
P
Evaporator
Dilution Vent
Charcoal Vapor Filter
Water Regulator Pilot Pressure
Water Back Pressure Valve and Rotameter
Electronics/PLC
- Neb Evaporator control
- Neb TED control
- Drip Counter
-Neb Liquid Pressure
- Neb Liquid Temp
- Data logging
-CPC control

Scanning TPC Long Term Stability



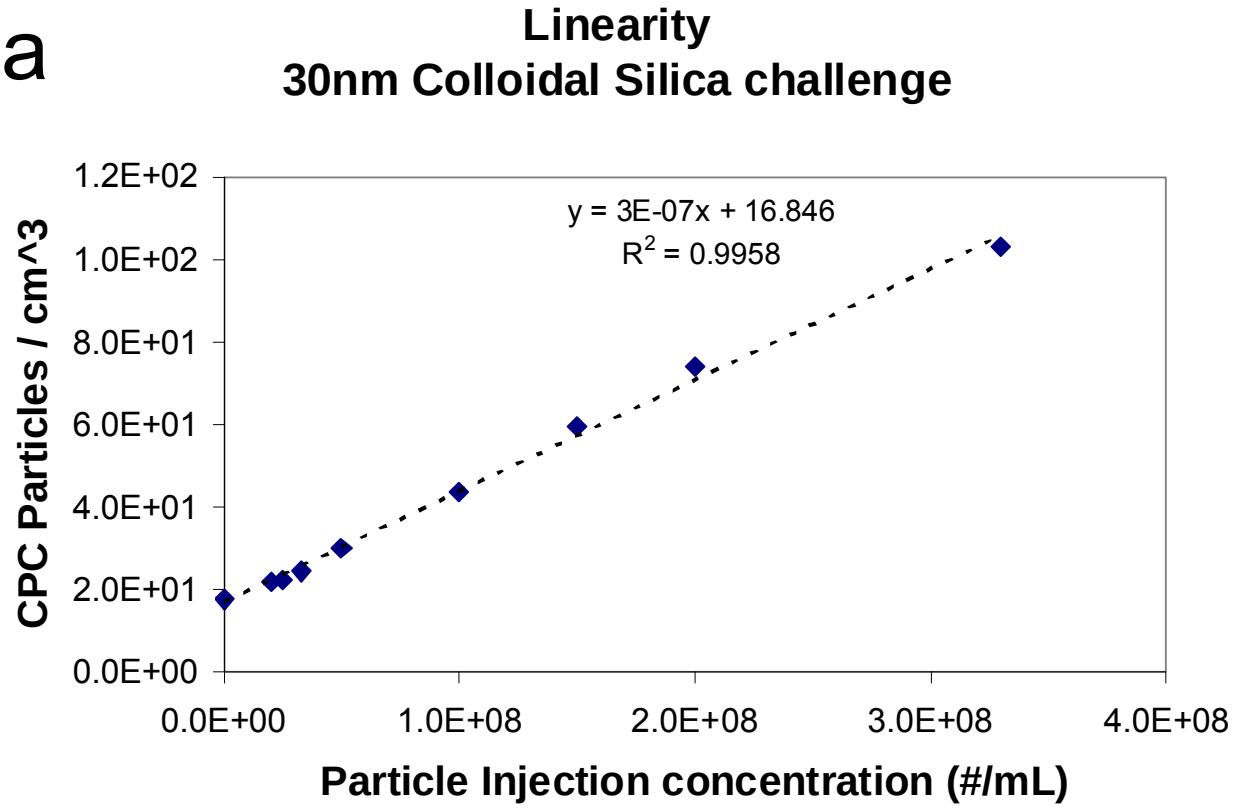
ScanningTPC Sample Data
Material Independence

- Inject 30 and 40 nm colloidal gold nanoparticles
- Response similar to colloidal silica

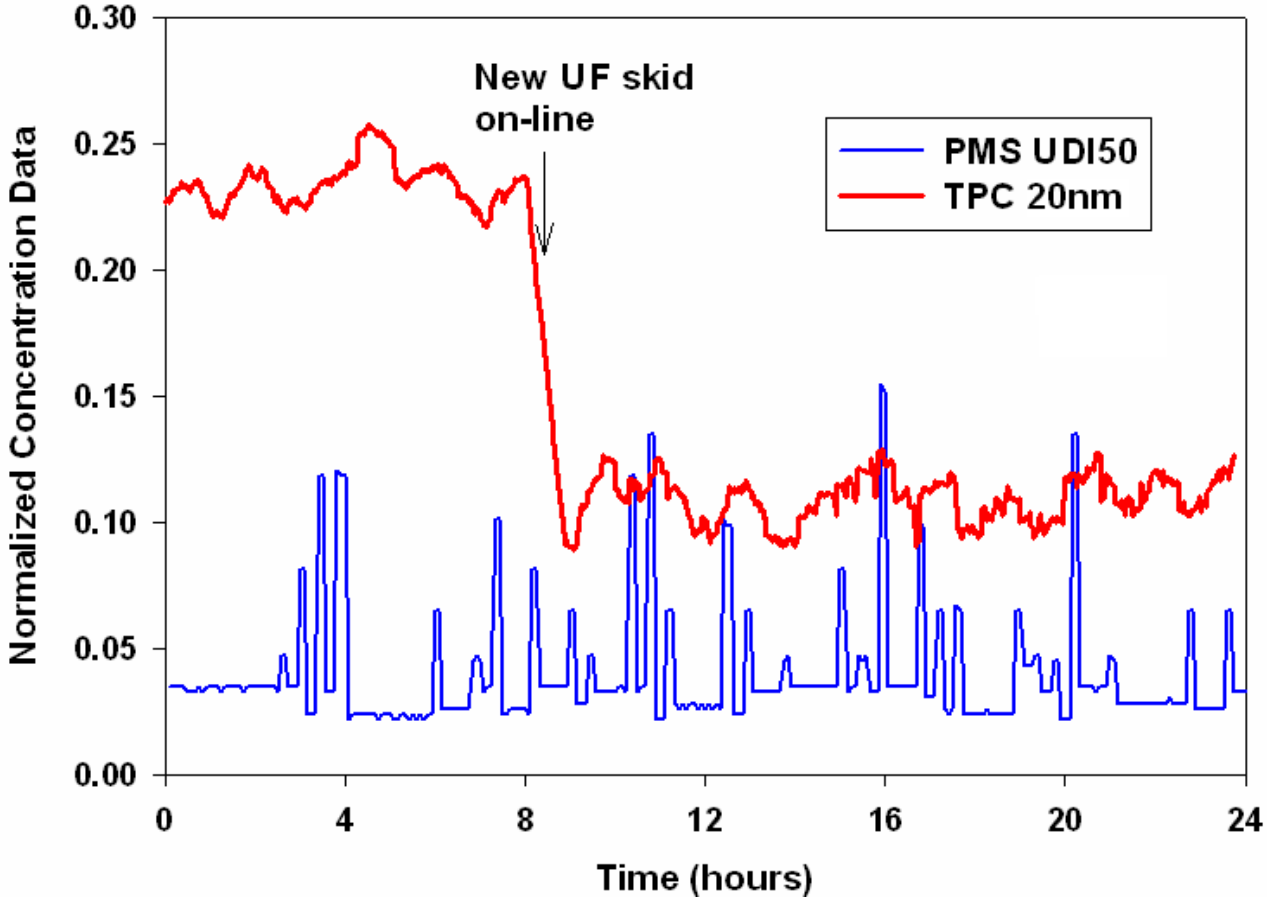


ScanningTPC Sample Data
Linearity

- Dilution ratios varied over a 16X range

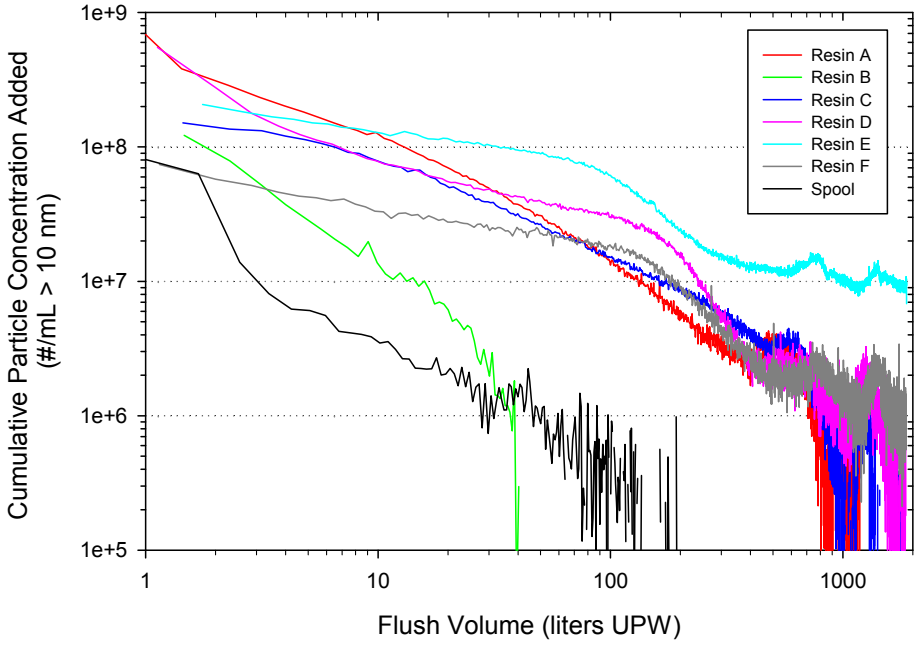


Scanning TPC
Single Channel Performance
Installation of Ultra Filters at a Semiconductor Facility

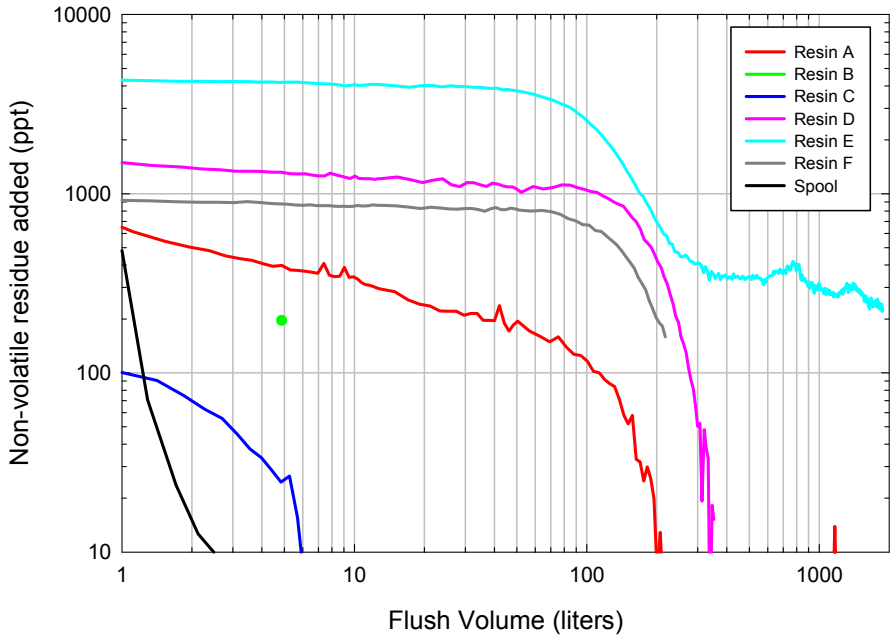


Scanning TPC
Single Channel Performance
Resin Rinse Down

Particle Rinse



Non-volatile Residue Rinse



Data prepared and presented by CT Associates to the SEMI Ion Exchange Task Force on 02/27/2014