Introducing a 10-nm Particle Counter for Ultrapure Water

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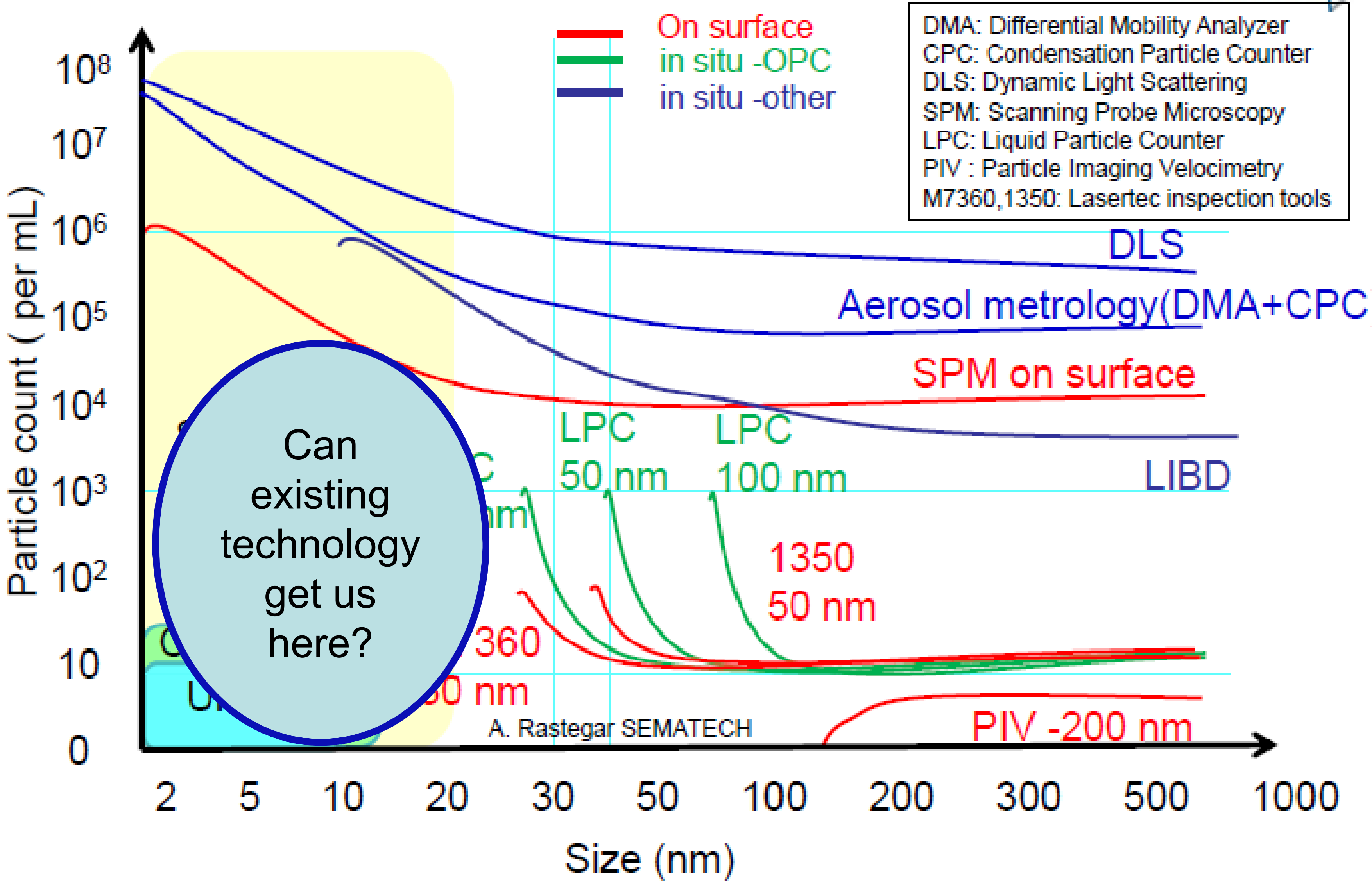
Creative Technology

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

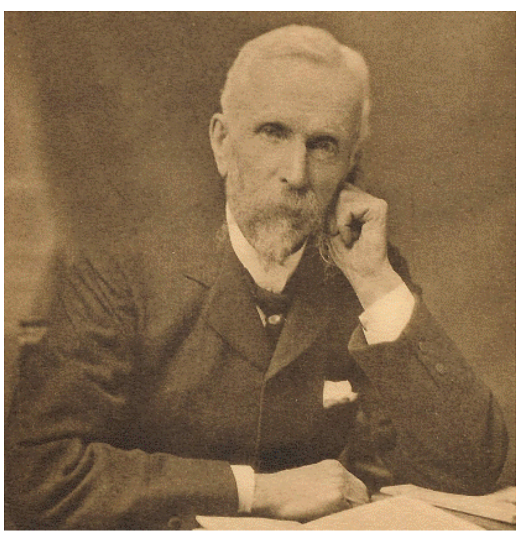


Liquid Particle Counting Technologies

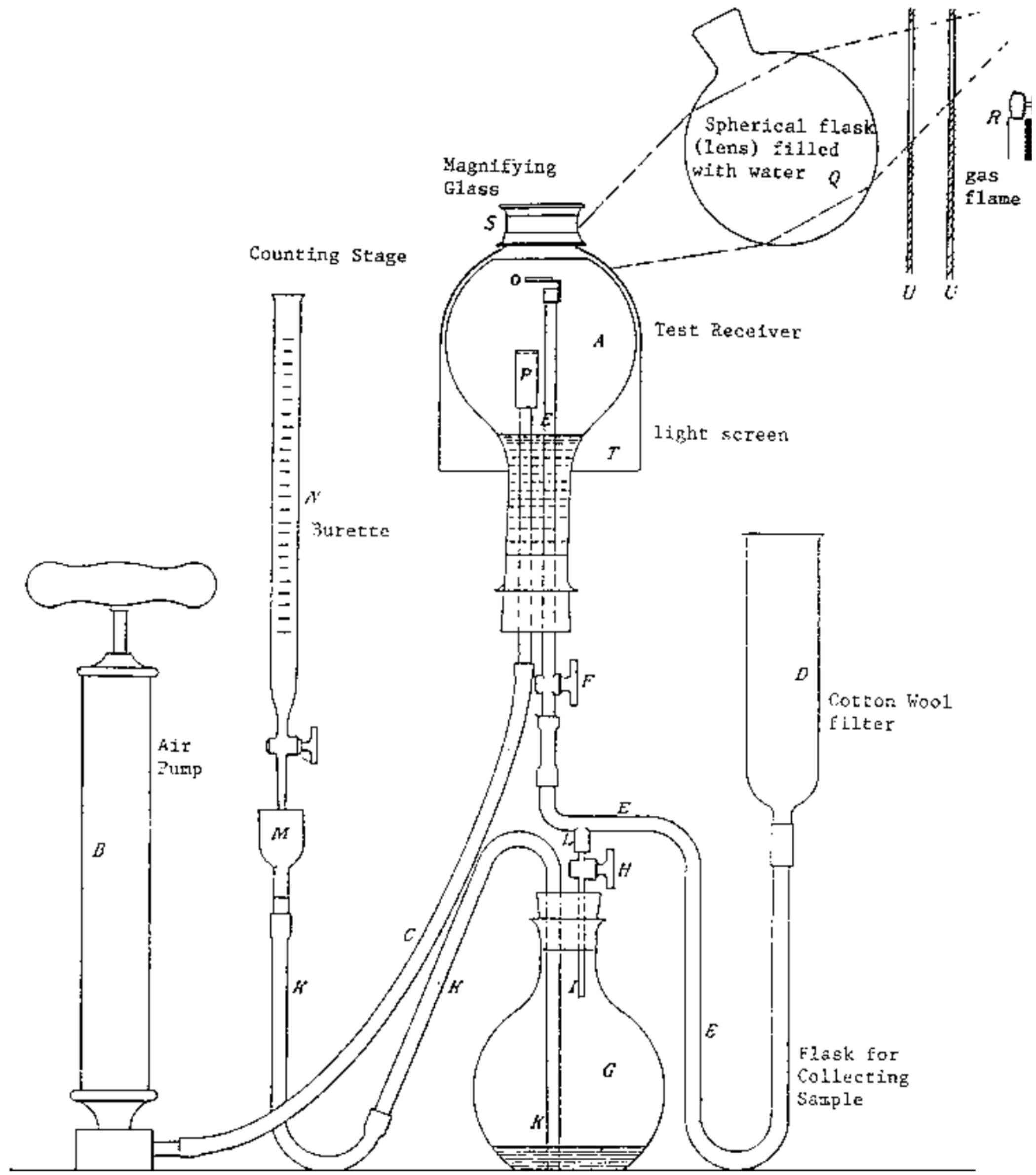
- Dynamic light scattering
  - Requires high concentrations
- Optical particle counting
  - Specified down to 25nm with 3-5% detection efficiency
  - Sensitive to particle composition
- Acoustic Coaxing Induced Microcavitation
  - Able to detect 20nm particles
  - Commercial availability unknown
- Nebulization – 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

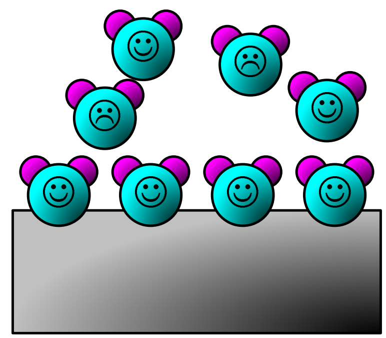


http://www.iara.org/AerosolPioneers.htm

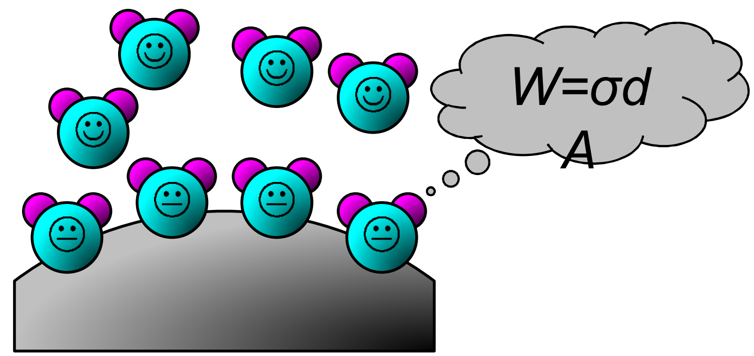


Condensation particle counting…
the science

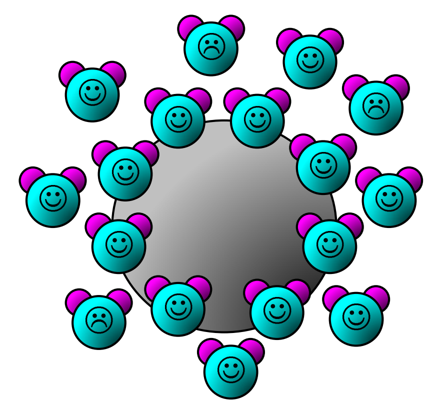
- Supersaturated vapors (relative humidity greater than 100%) are not happy
- The vapor wants to leave the gas phase and condense onto a surface



- 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



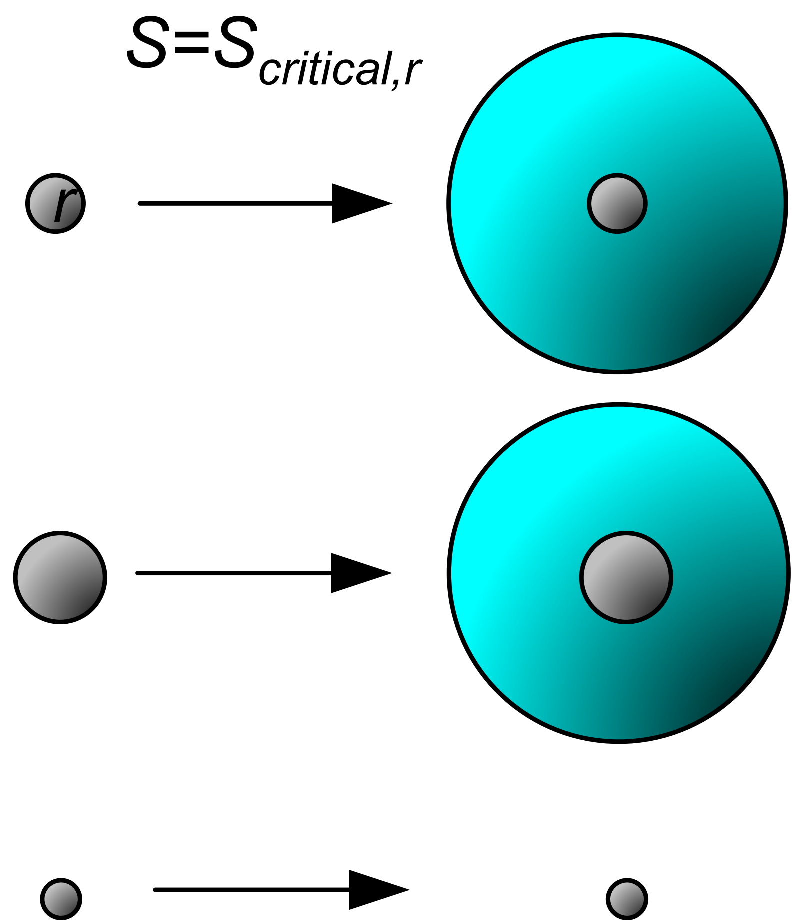
Condensation particle counting

more science

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

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

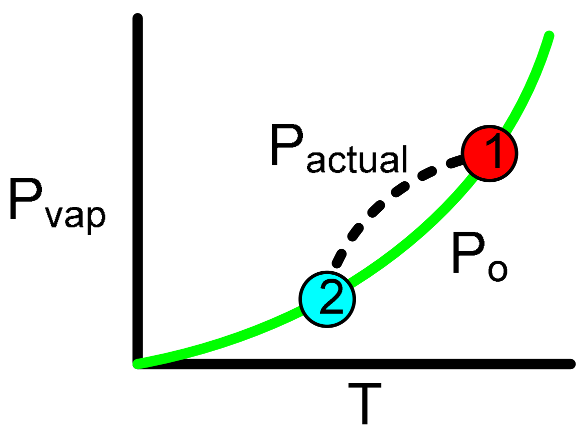
  where γ is the surface tension and V_m is the molecular volume of the condensing liquid. P_o is the vapor pressure above a flat surface of the condensing vapor and P is the actual vapor pressure
- P/P_o is the Saturation Ratio, S
- Varying the degree of S changes the minimum enlarged particle size

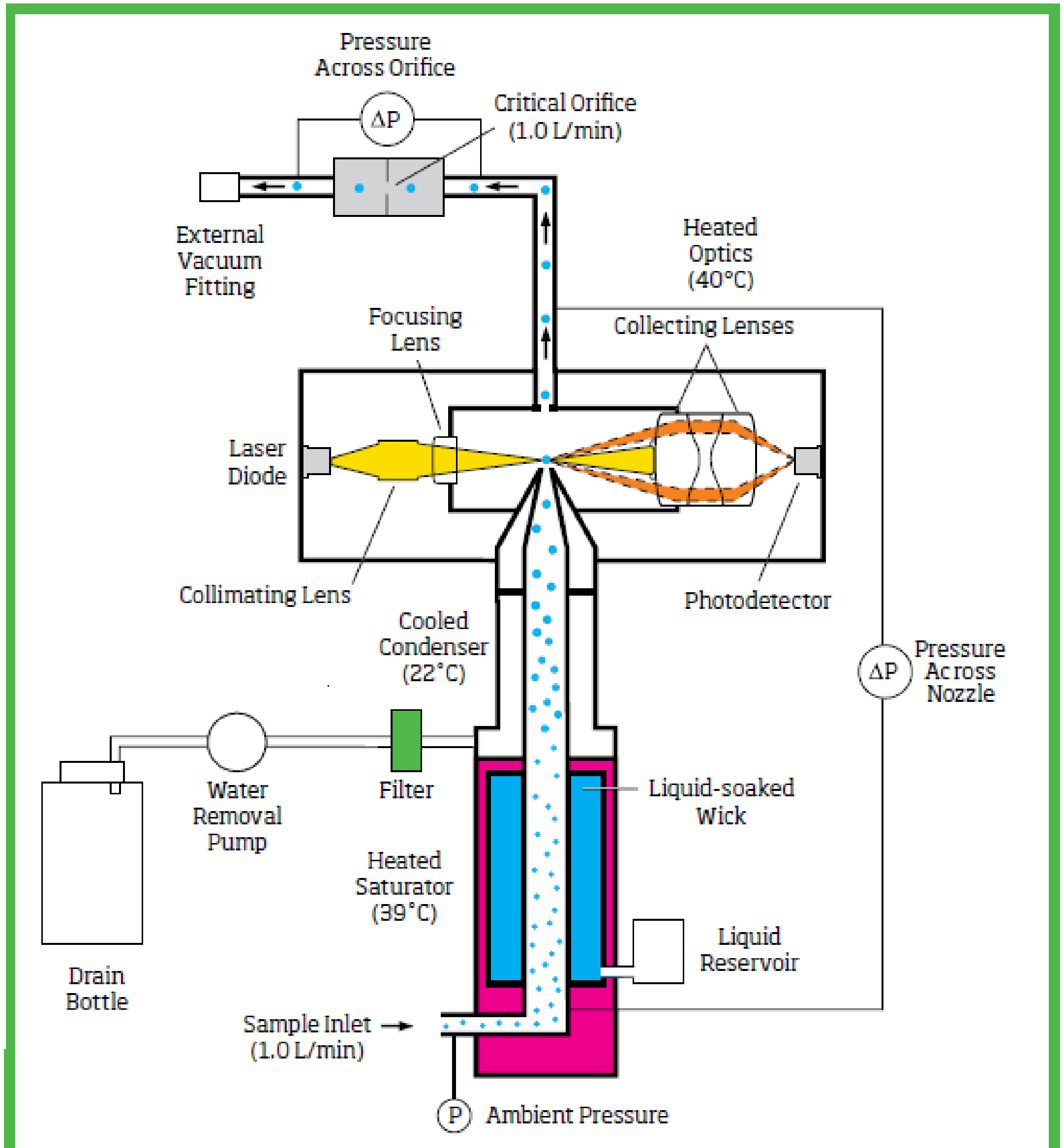


Condensation Particle Counting

how it is implemented

- Lewis number is the ratio of mass diffusivity of a vapor *D* to thermal diffusivity of a gas *λ*, $Le = \lambda / D$
- $Le > 1$ (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

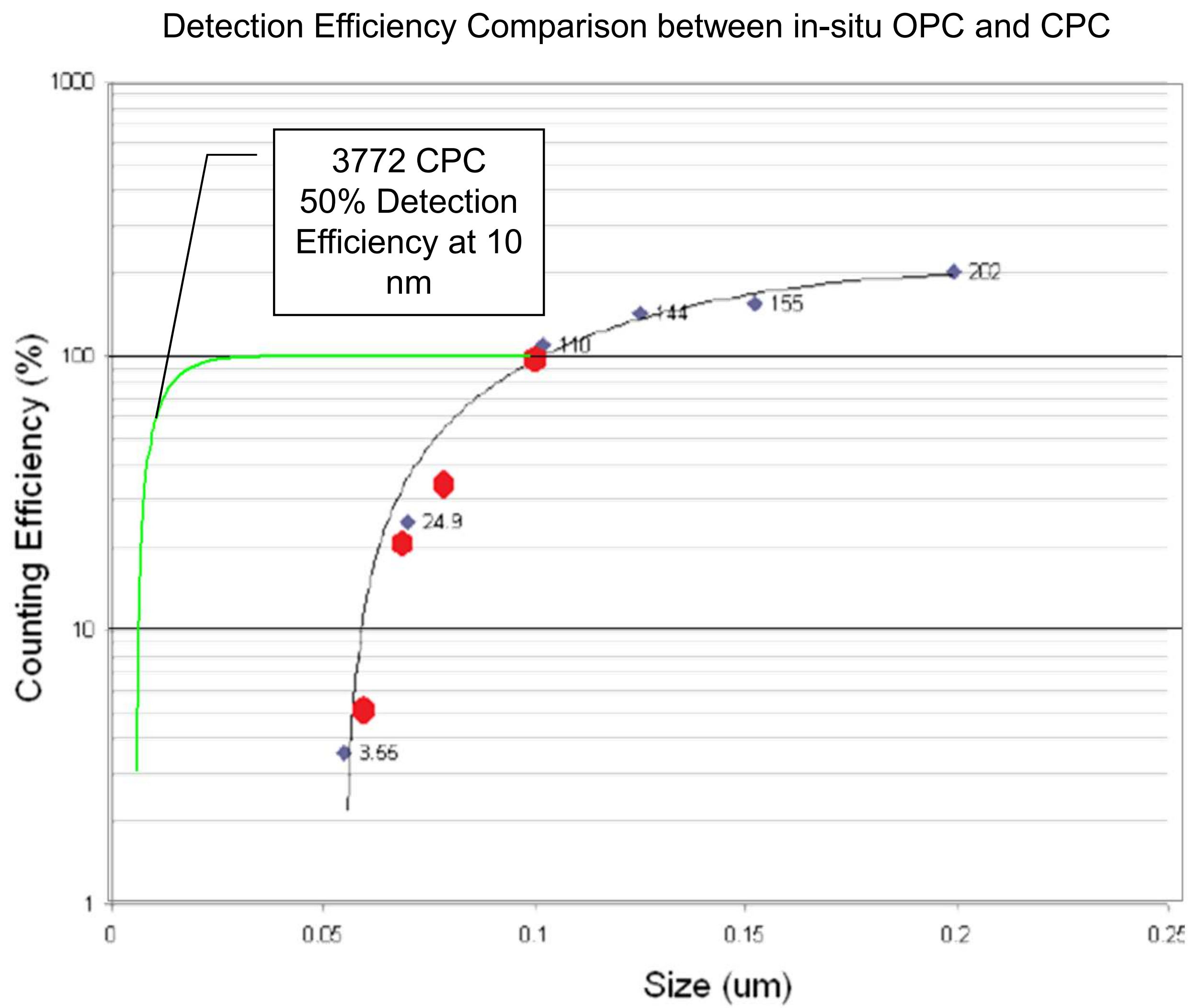




TSI Incorporated 3772 CPC Brochure

A note on detection efficiencies

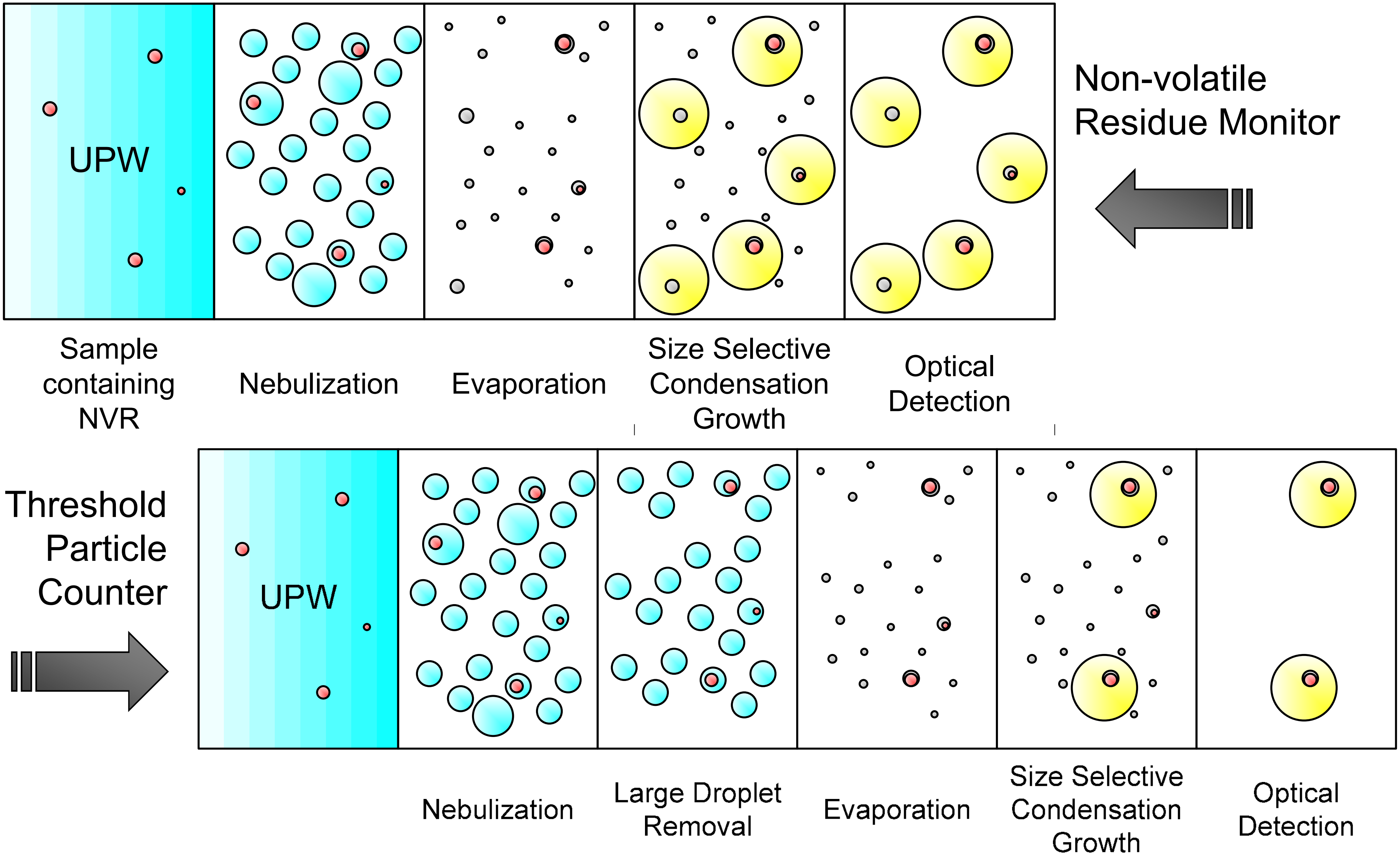
- OPCs have a shallow detection efficiency curve
- Many OPCs are specified at less than 5% detection efficiency
- Condensation Particle Counters (CPCs) are specified at their 50% cutoff



OPC data provided by Particle Measuring Systems.
3772 CPC Data by CT Associates

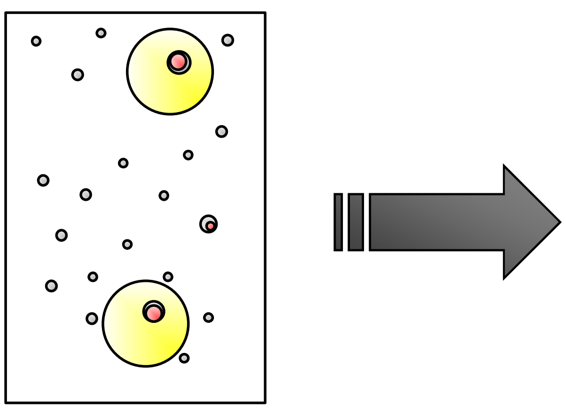
Scanning Threshold Particle Counter (Scanning TPC)

Principle of Operation

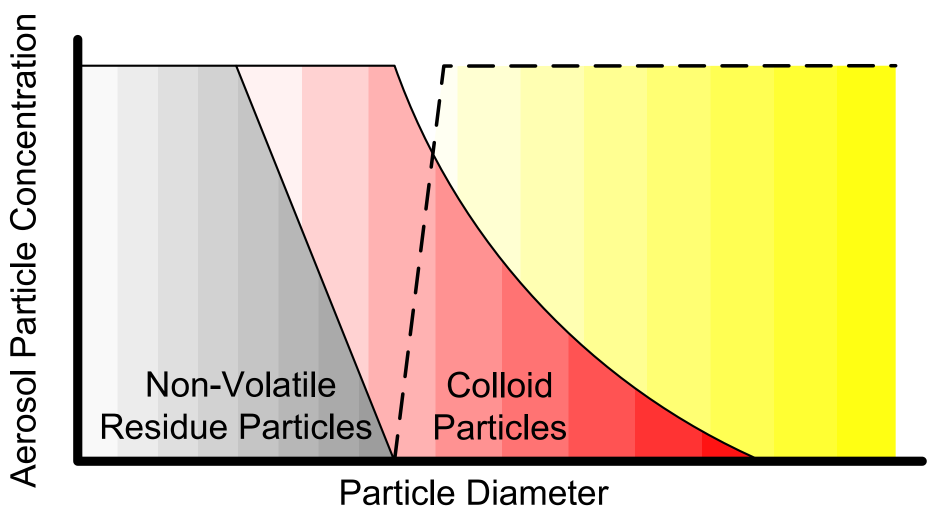


ScanningTPC
Principle of Operation

- 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

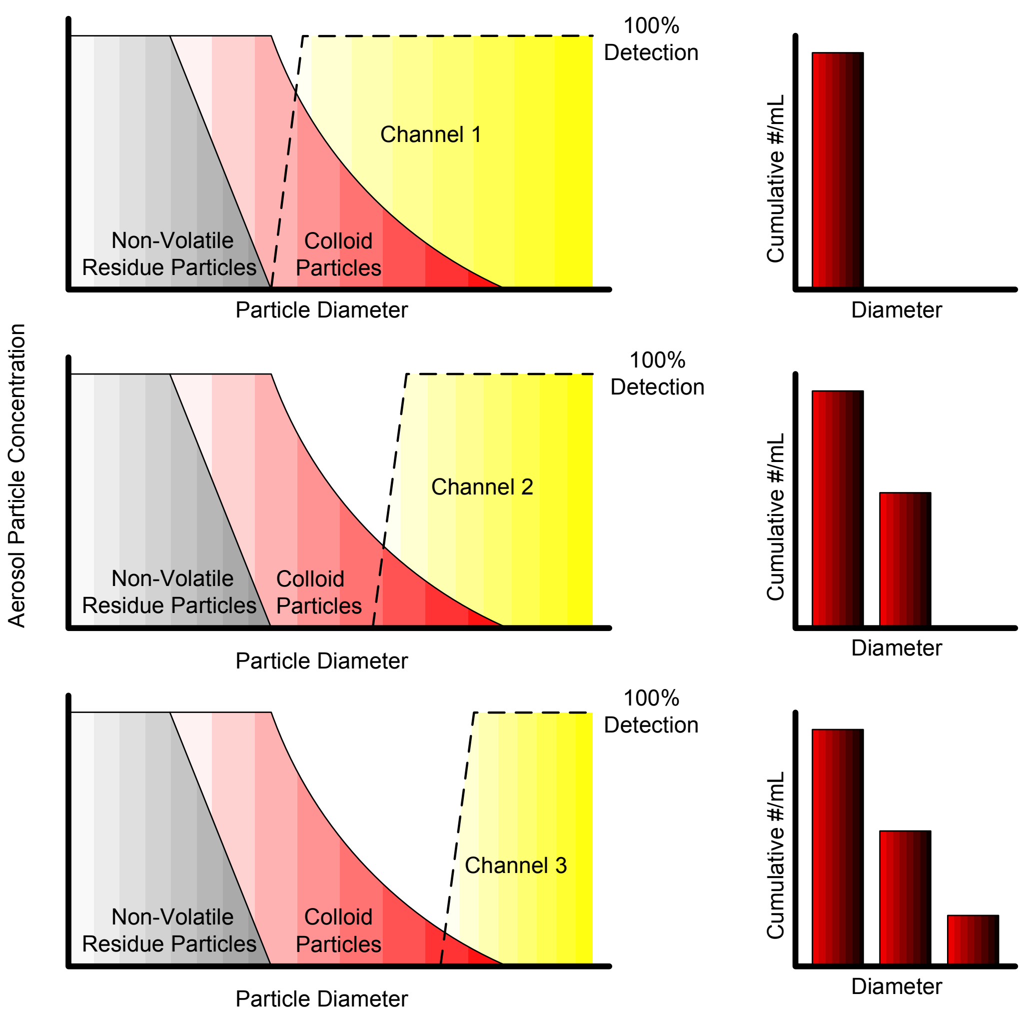


Size Selective Condensation Growth



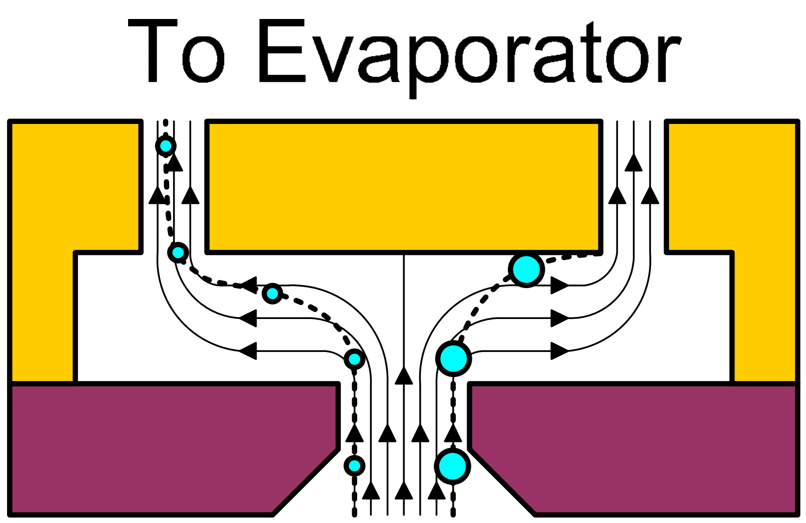
ScanningTPC
Principle of Operation

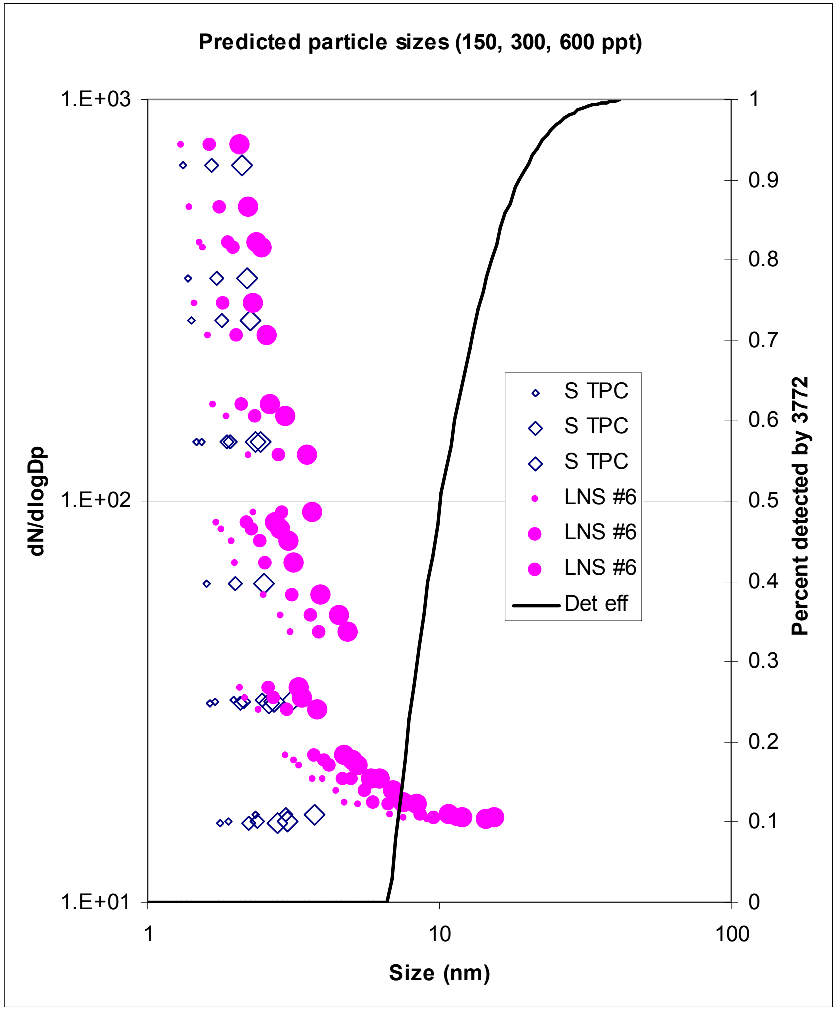
- Stepping threshold diameter range currently 10 nm to 30 nm
- Lower threshold diameters possible with low residue UPW
- Higher threshold diameters also possible



Scanning TPC Nebulizer Design

Secondary large droplet removal incorporated downstream of nebulization region

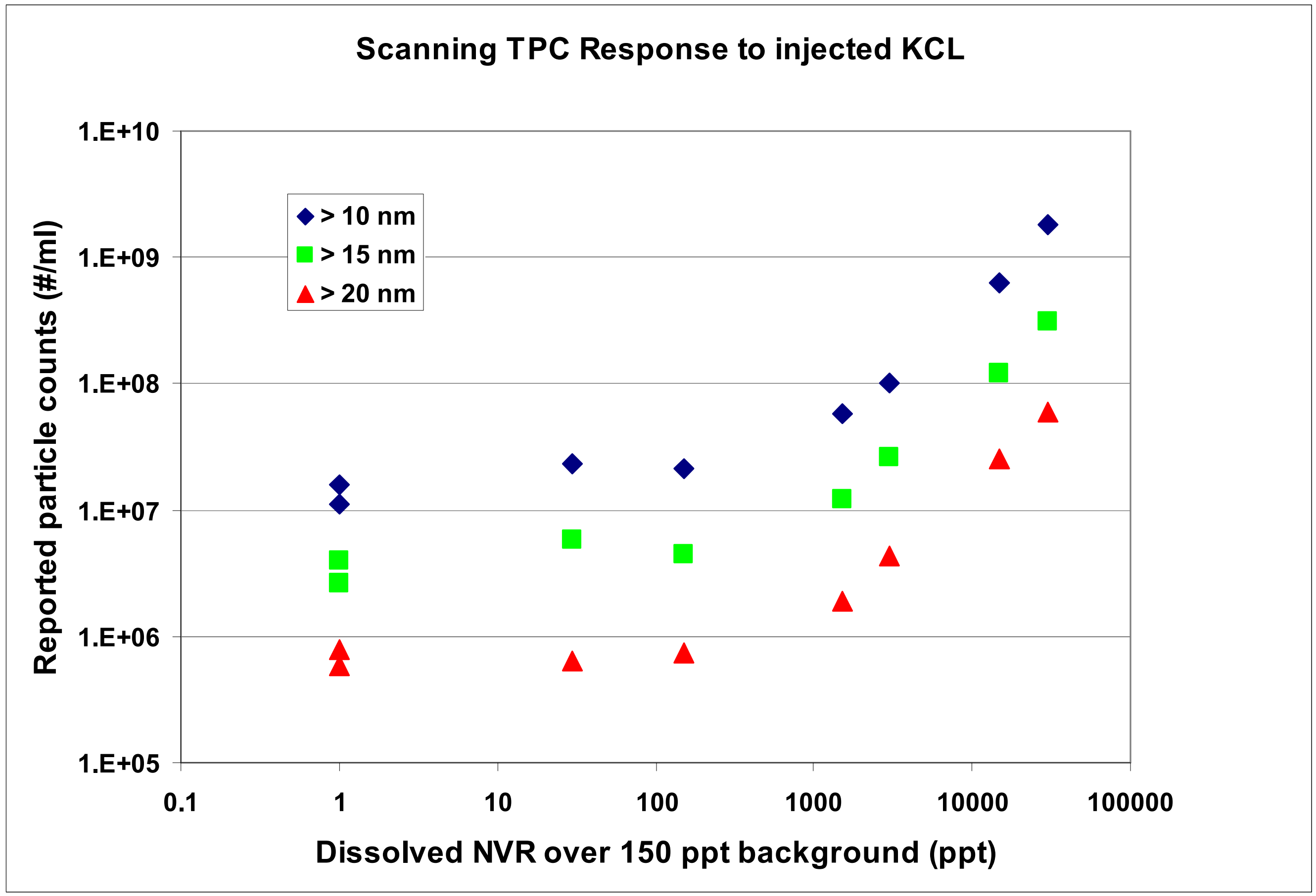




Upper tail of droplet distribution removed to minimize dissolved non-volatile residue particle counts

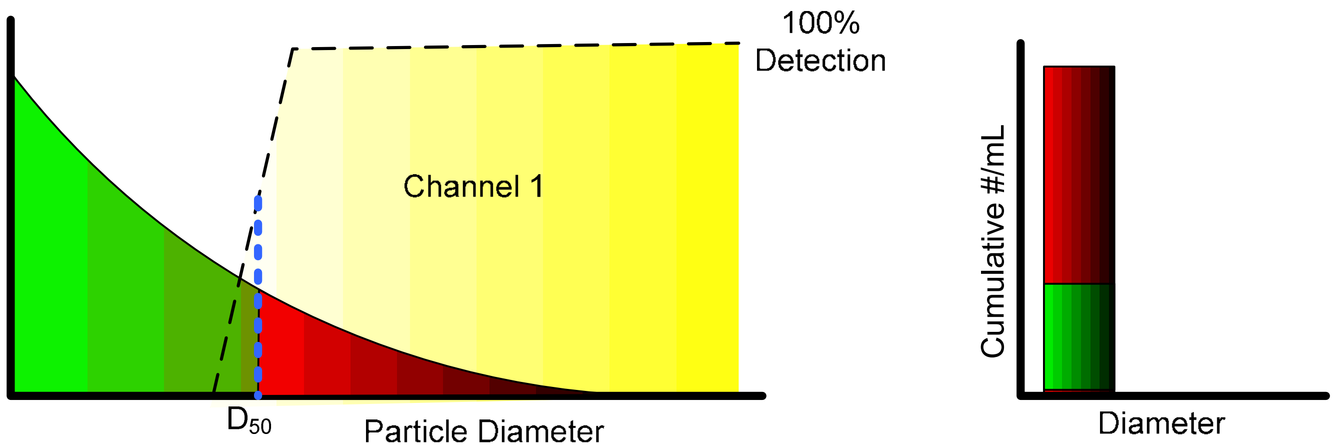
Scanning TPC
Response to dissolved NVR

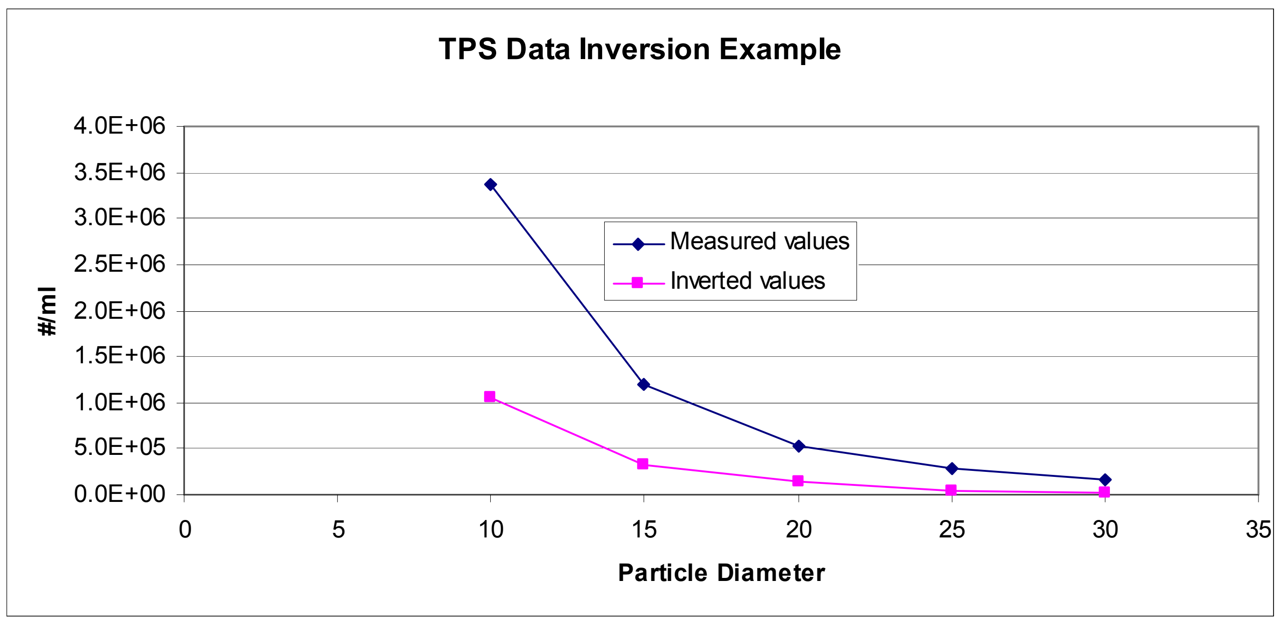
- High levels of DNVR will lead to false particle counts
- Plot shows little sensitivity to low levels of added NVR (some true colloidal particles will be present due to injection system and impurities in the KCL)



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



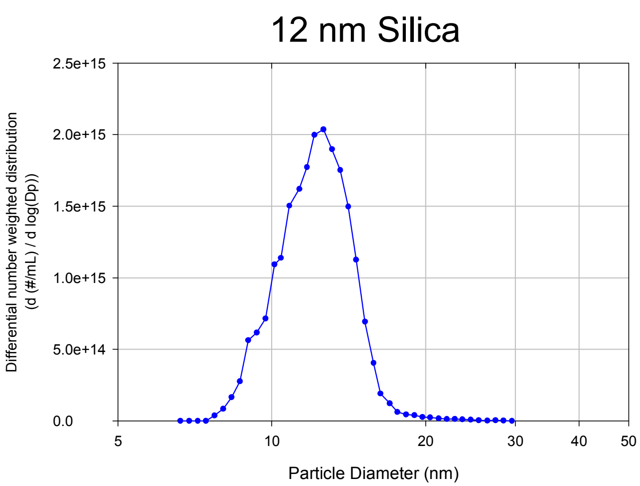


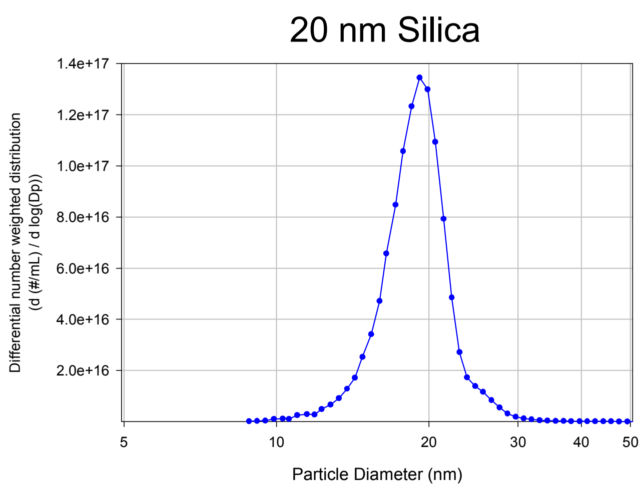
Scanning TPC Sample Data
Colloid Size Standards

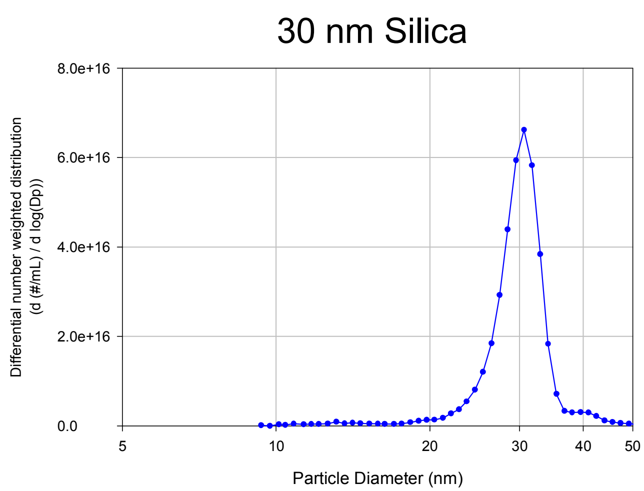
- High concentration solutions of colloidal silica and gold nanoparticles prepared for testing
- Size distributions of each of the solutions has been characterized using Liquid Nanoparticle Sizing (LNS) technology
  - US Patents 8,272,253 and 8,573,034
- Use LiquiTrak ® Precision Diluter to provide high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

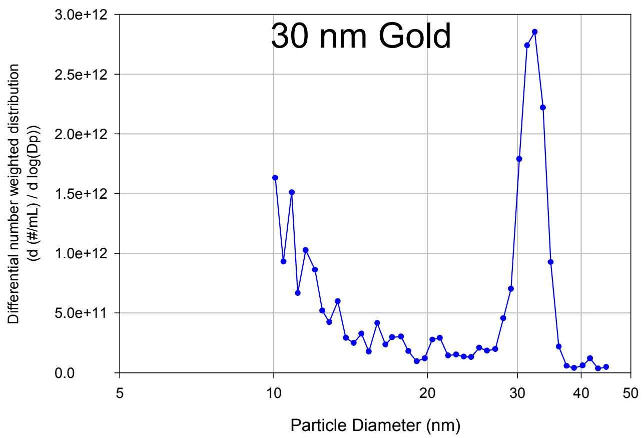
ScanningTPC Sample Data

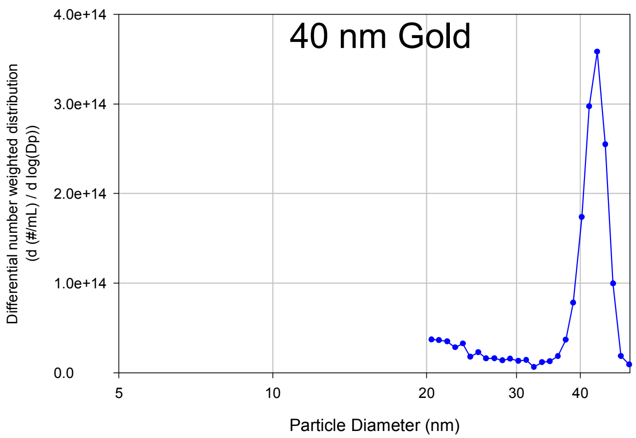
Colloid Size Standards









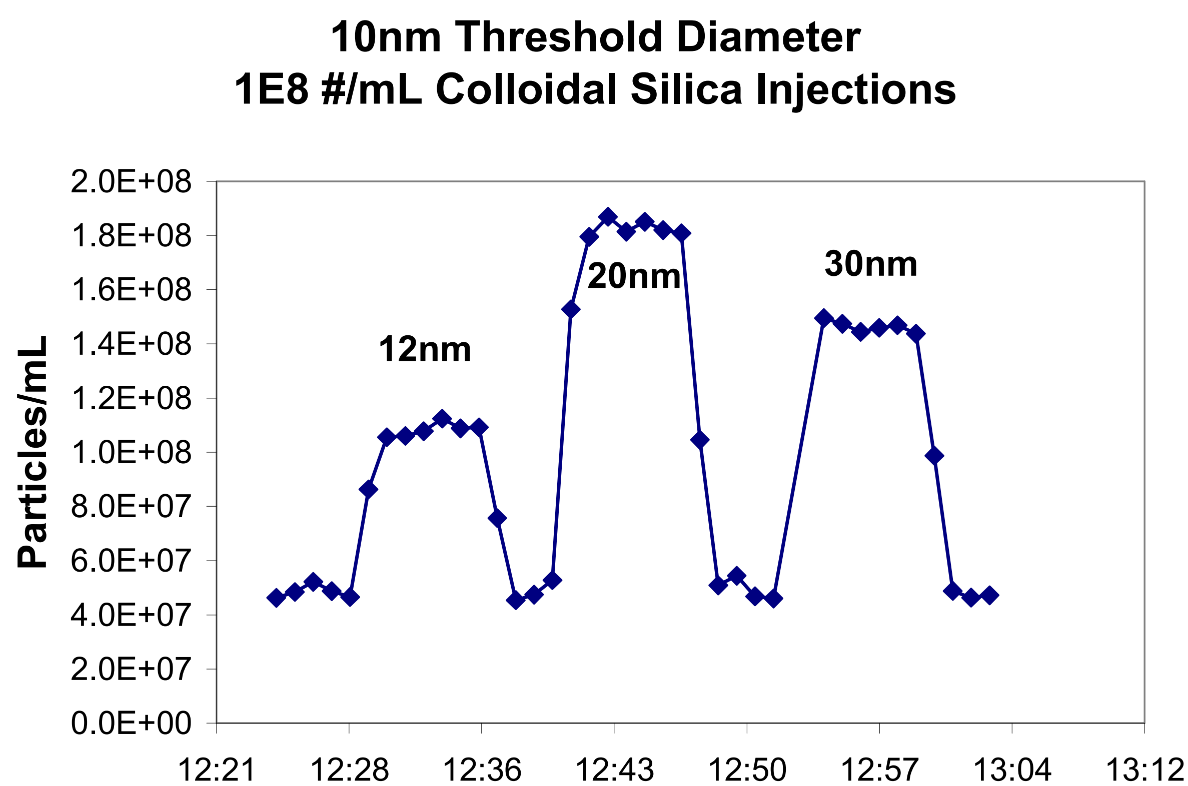


Data provided by CT Associates using Liquid Nanoparticle Sizing methodology

ScanningTPC Sample Data

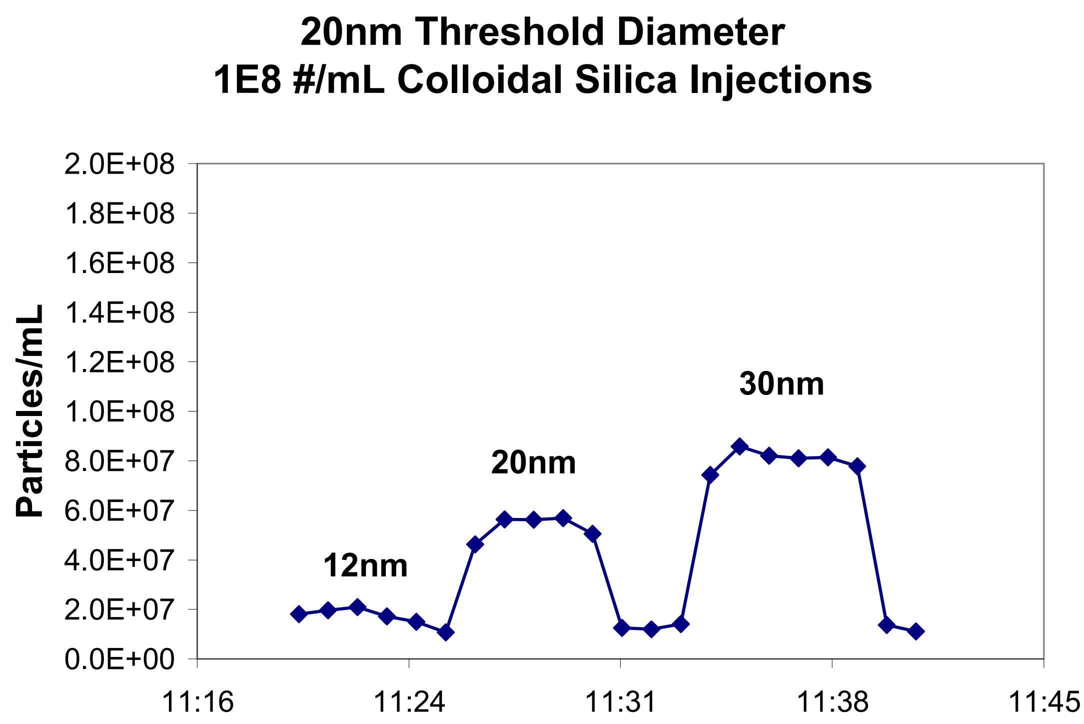
Sensitivity

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



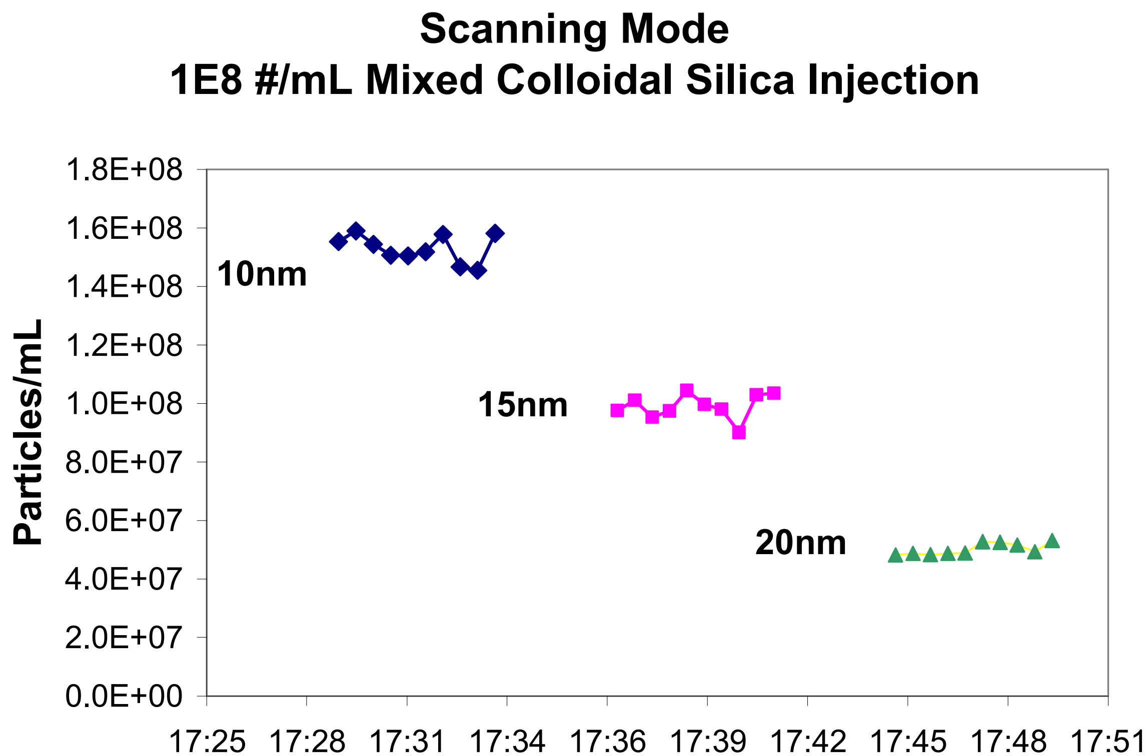
ScanningTPC Demonstration
Sensitivity

- 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.



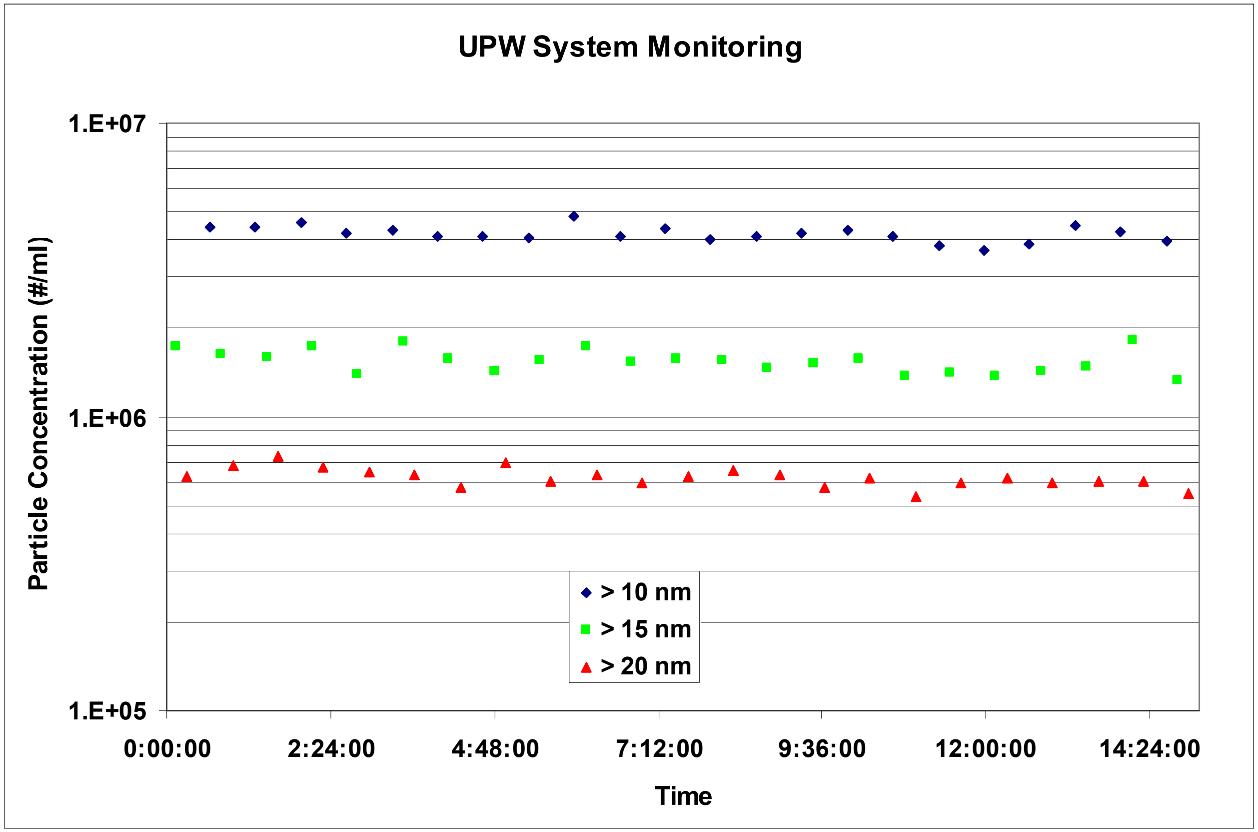
ScanningTPC Sample Data
Scanning Operation

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



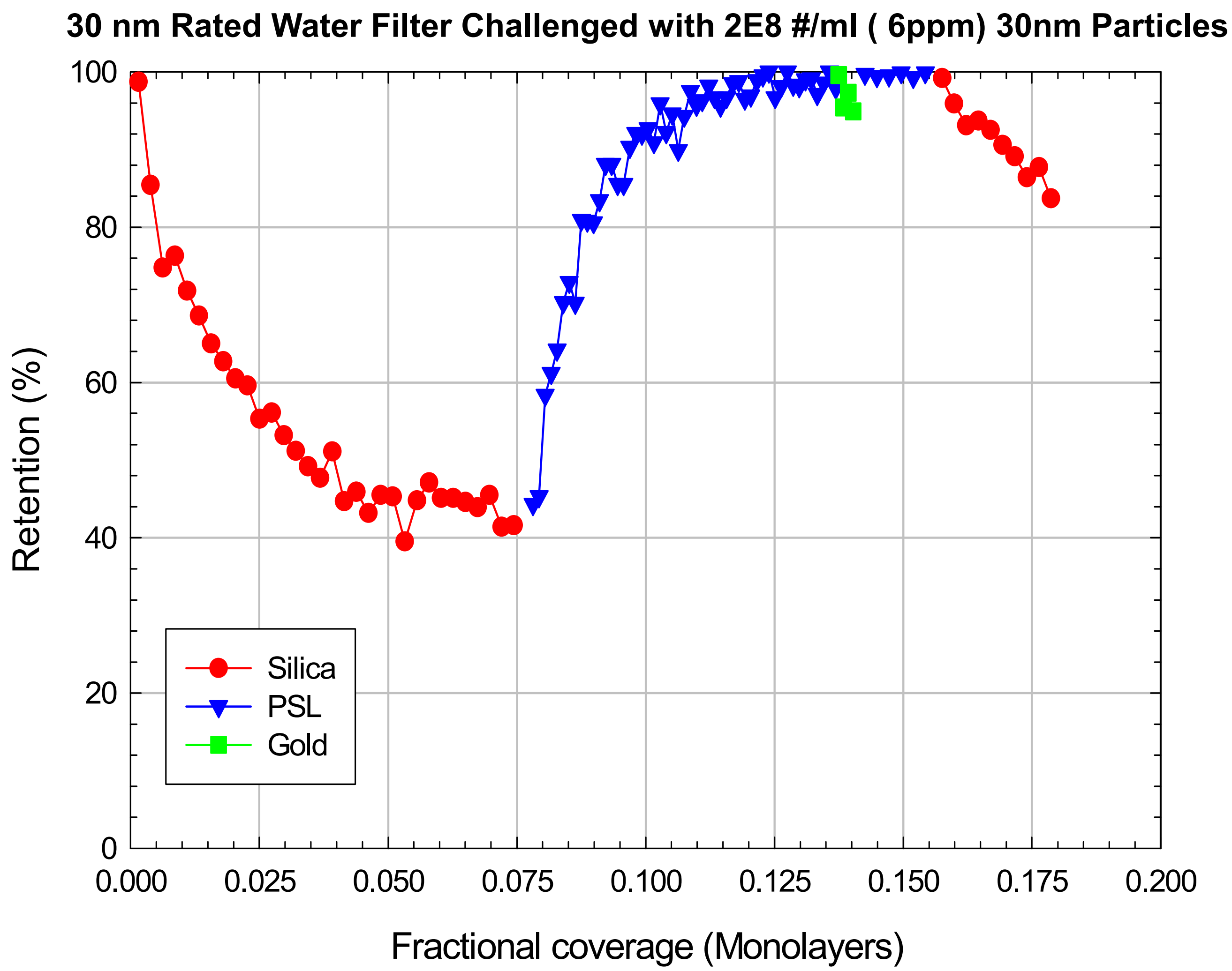
ScanningTPC Sample Data
UPW Monitoring

- 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

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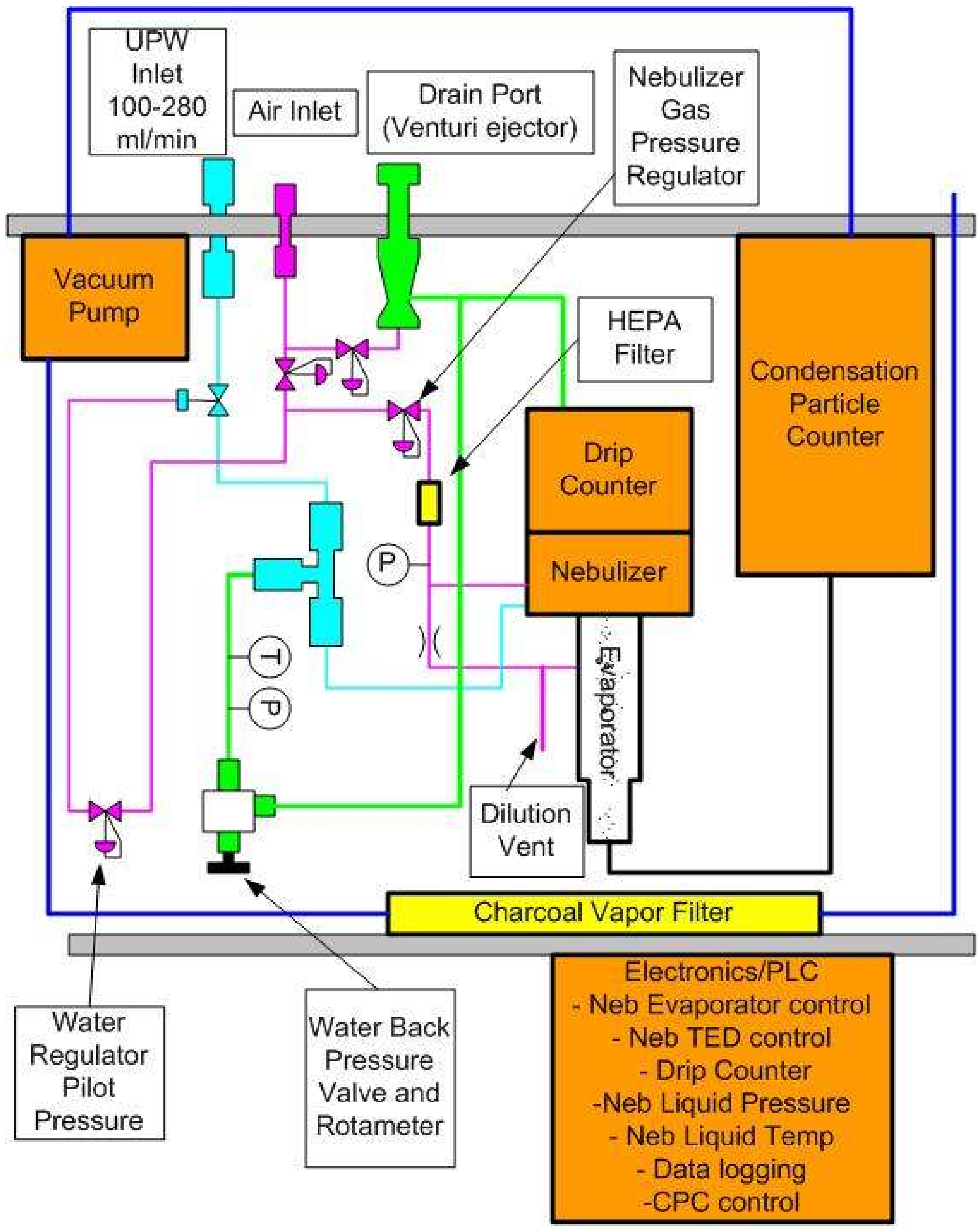
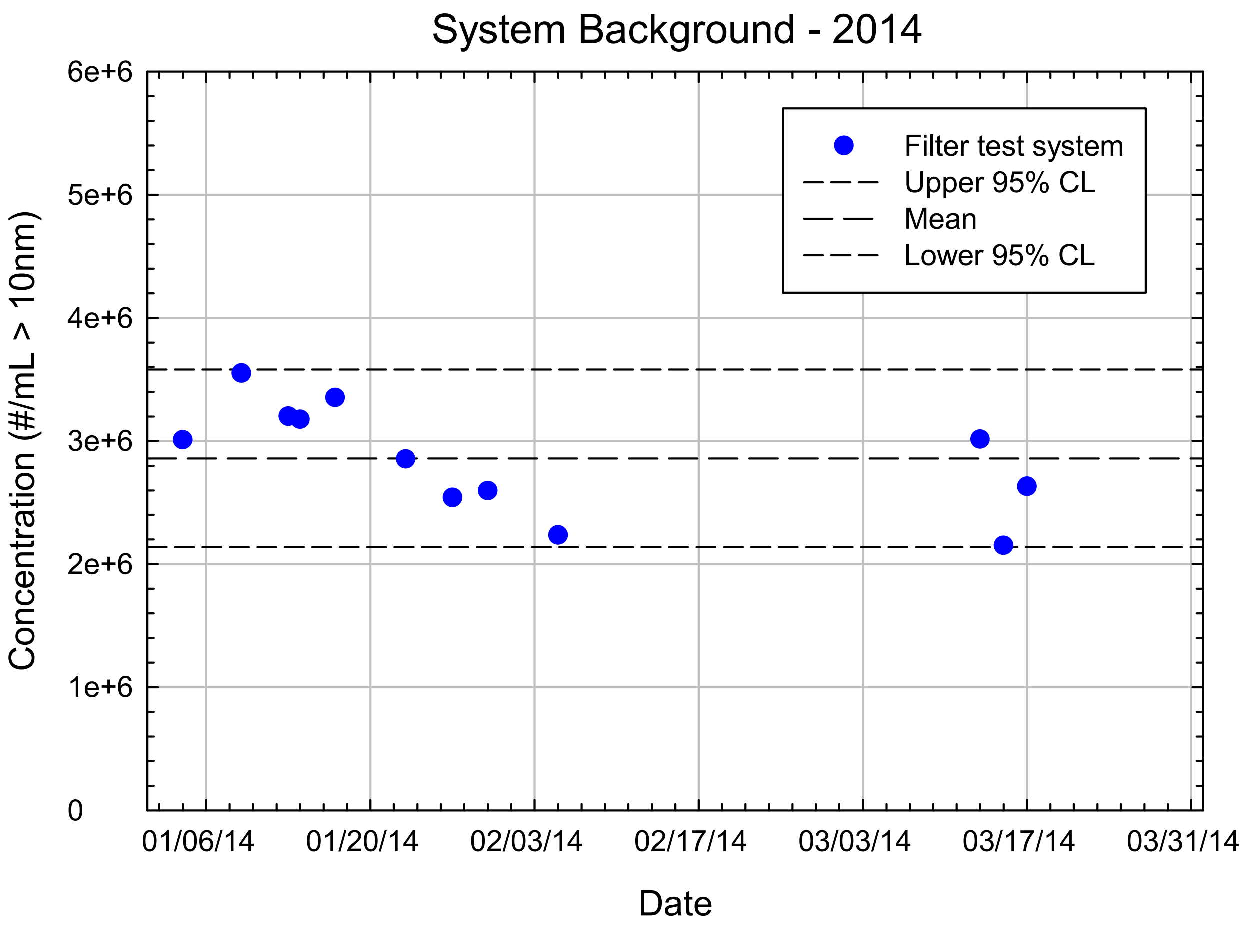
Rastegar, A (2013). "Particle Control Challenges in UPW ", presented at 2013 UPW Micro Conference

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



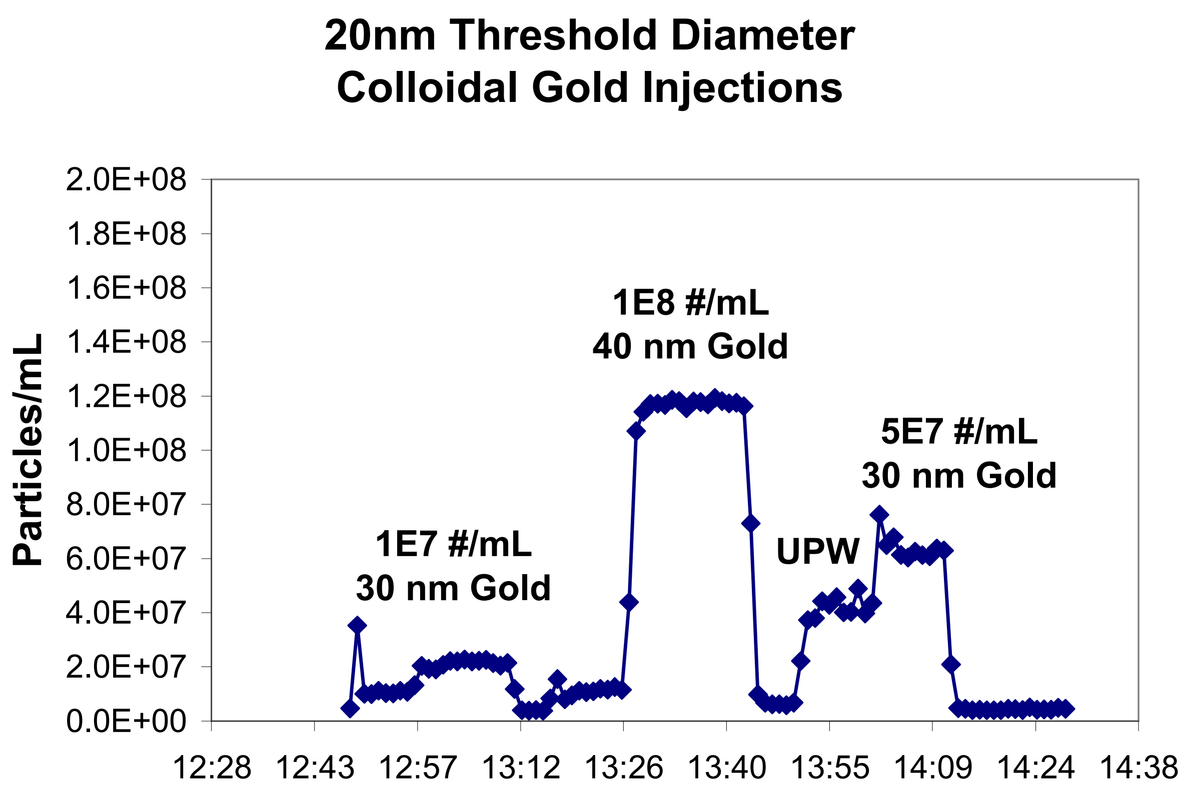
UPW Inlet 100-280 ml/min
Air Inlet
Drain Port (Venturi ejector)
Nebulizer Gas Pressure Regulator
Vacuum Pump
HEPA Filter
Condensation Particle Counter
Drip Counter
Nebulizer
P
T
P
Evaporator
Dilution Vent
Charcoal Vapor Filter
Water Regulator Pilot Pressure
Water Back Pressure Valve and Rotameter
Electronics/PLC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

Scanning TPC Long Term Stability



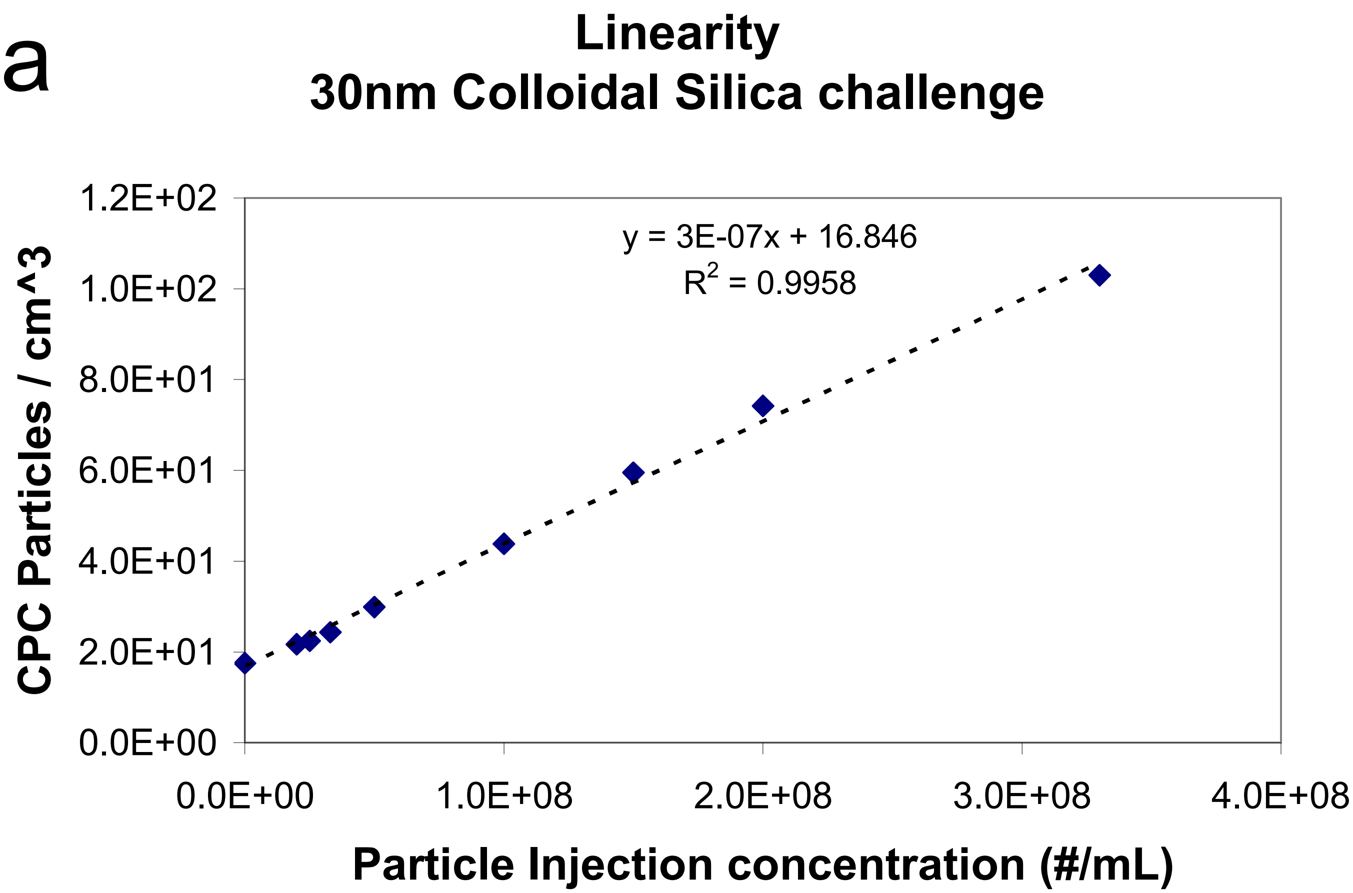
ScanningTPC Sample Data
Material Independence

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

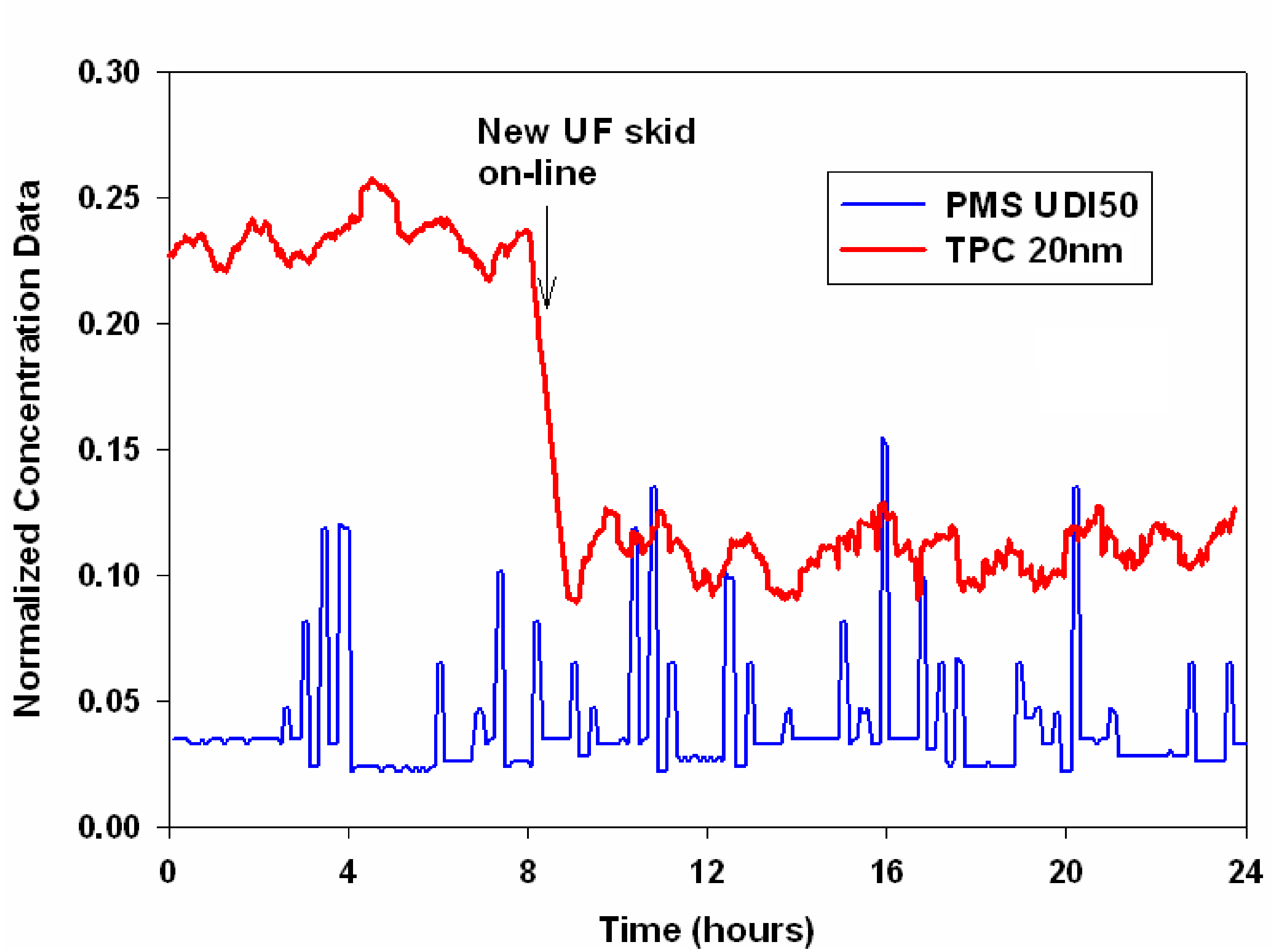


ScanningTPC Sample Data
Linearity

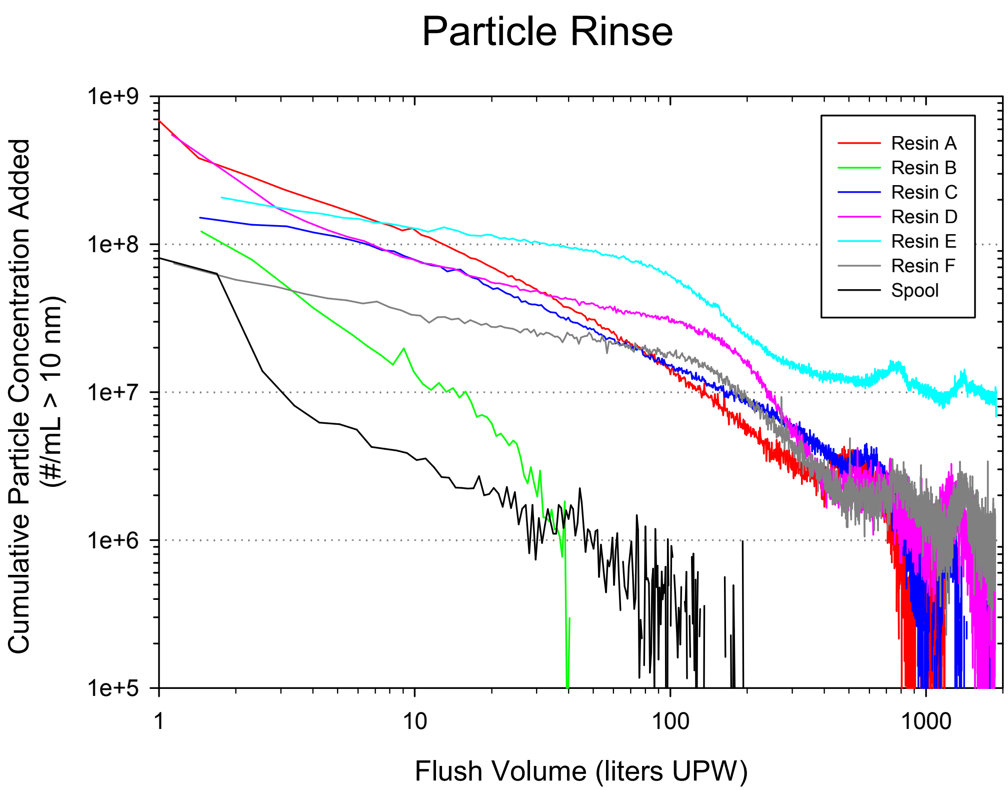
- Dilution ratios varied over a 16X range

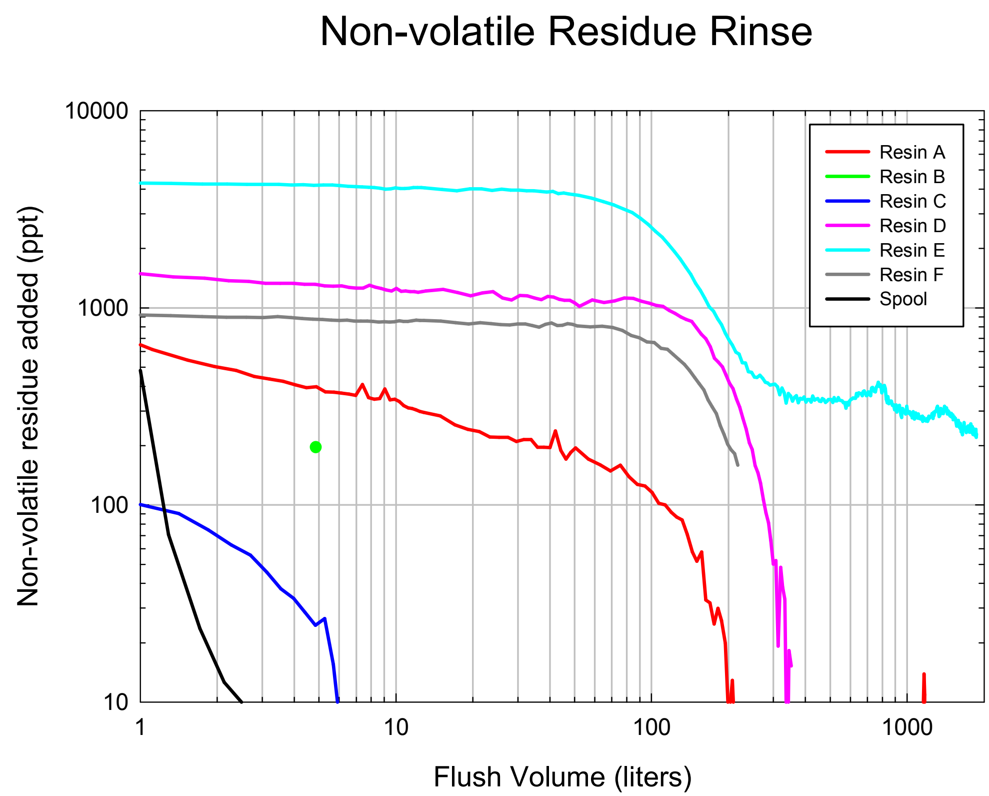


Scanning TPC
Single Channel Performance
Installation of Ultra Filters at a Semiconductor Facility



Scanning TPC
Single Channel Performance
Resin Rinse Down





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