Application of a 10nm particle counter and non-volatile residue monitor for component diagnostics within a UPW system

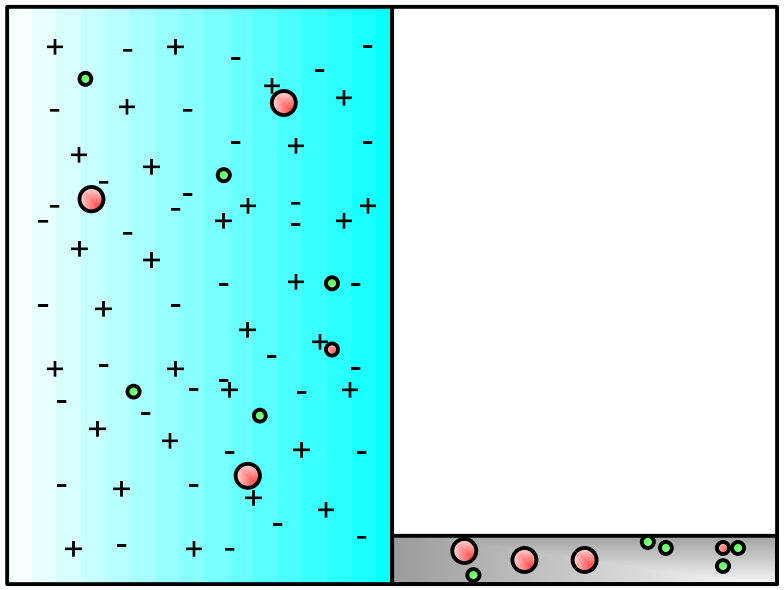
Derek Oberreit, David Blackford
Kanomax FMT, Inc.
Gary Van Schooneveld
CT Associates, Inc.

Outline

- Non-Volatile Residue in Liquids – What is it and how do we characterize it?
- Aerosolization – What does it mean and what factors drive performance?
- Overview of instrumentation
  - Non-Volatile Residue Monitor
  - Scanning Threshold Particle Counter
- System sampling data

Non-Volatile Residue
Definition

- Non-Volatile Residue (NVR) refers to solid material remaining after evaporation of a liquid
- Composed of previously dissolved salts, organics, and solid particles that were present in the liquid (colloidal particles)



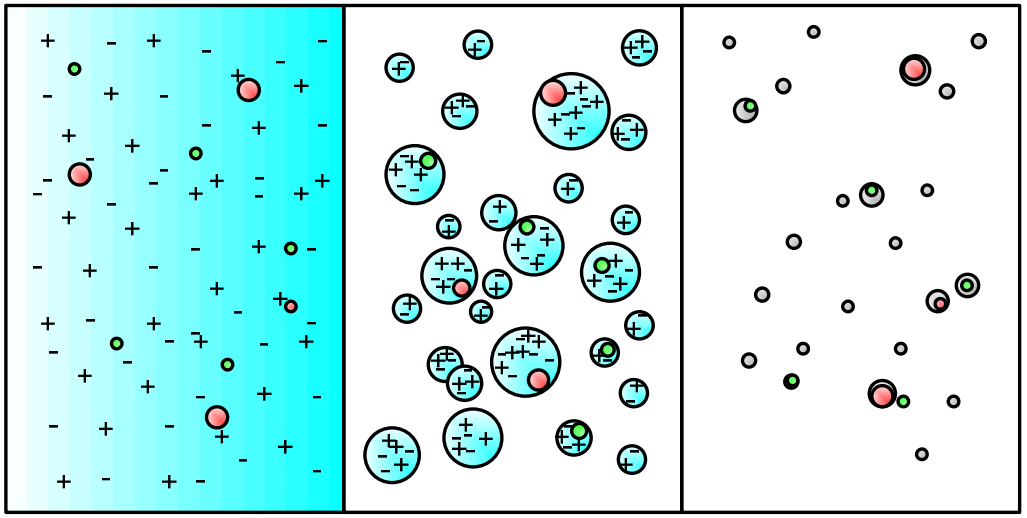
Liquid Sample With NVR

Evaporated Sample

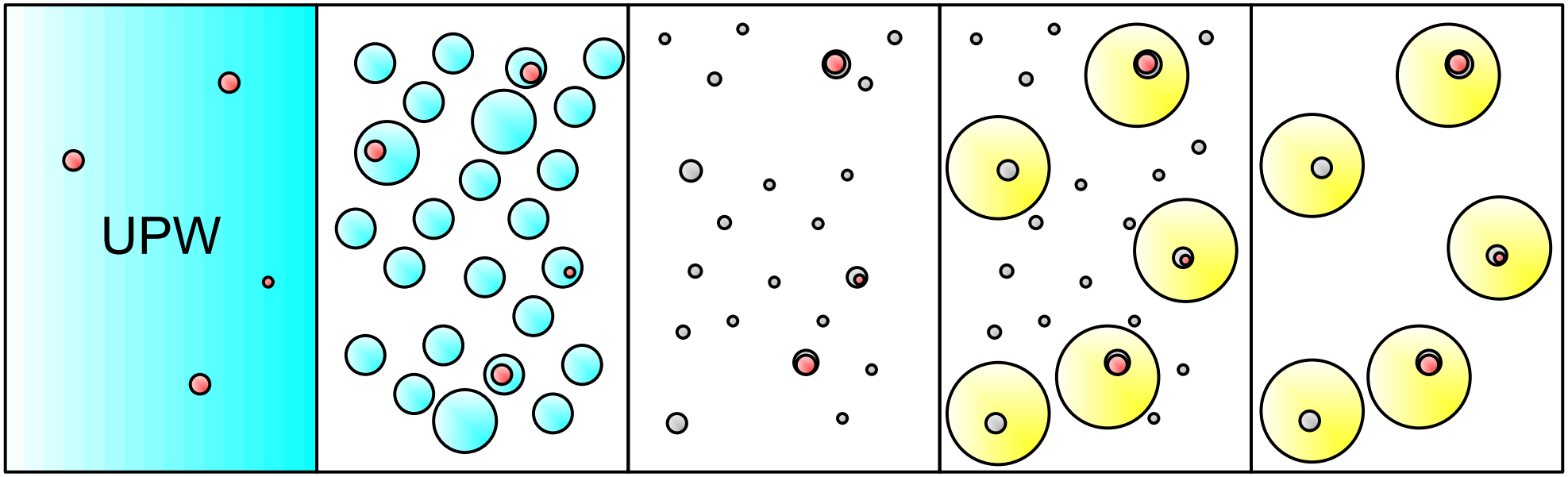
- Insoluble Suspended Particles
- Semi-volatile Organics
- Dissolved Non-Volatile Residue
- Precipitated Non-Volatile Residue

Aerosolization
Definition

- Spray liquid to form a mist of fine droplets (Nebulization)
- Evaporate liquid from the droplets
- Each droplet forms a solid aerosol particle composed of Precipitated Non-Volatile Residue (PNVR) whose size is proportional to the original droplet size
- A small fraction of the aerosol particles will also contain insoluble suspended particles and/or semi-volatile organics found in the original sample



Non-Volatile Residue Monitor (NRM)
Principle of Operation



Sample containing NVR

Nebulization

Evaporation

Size Selective Condensation Growth

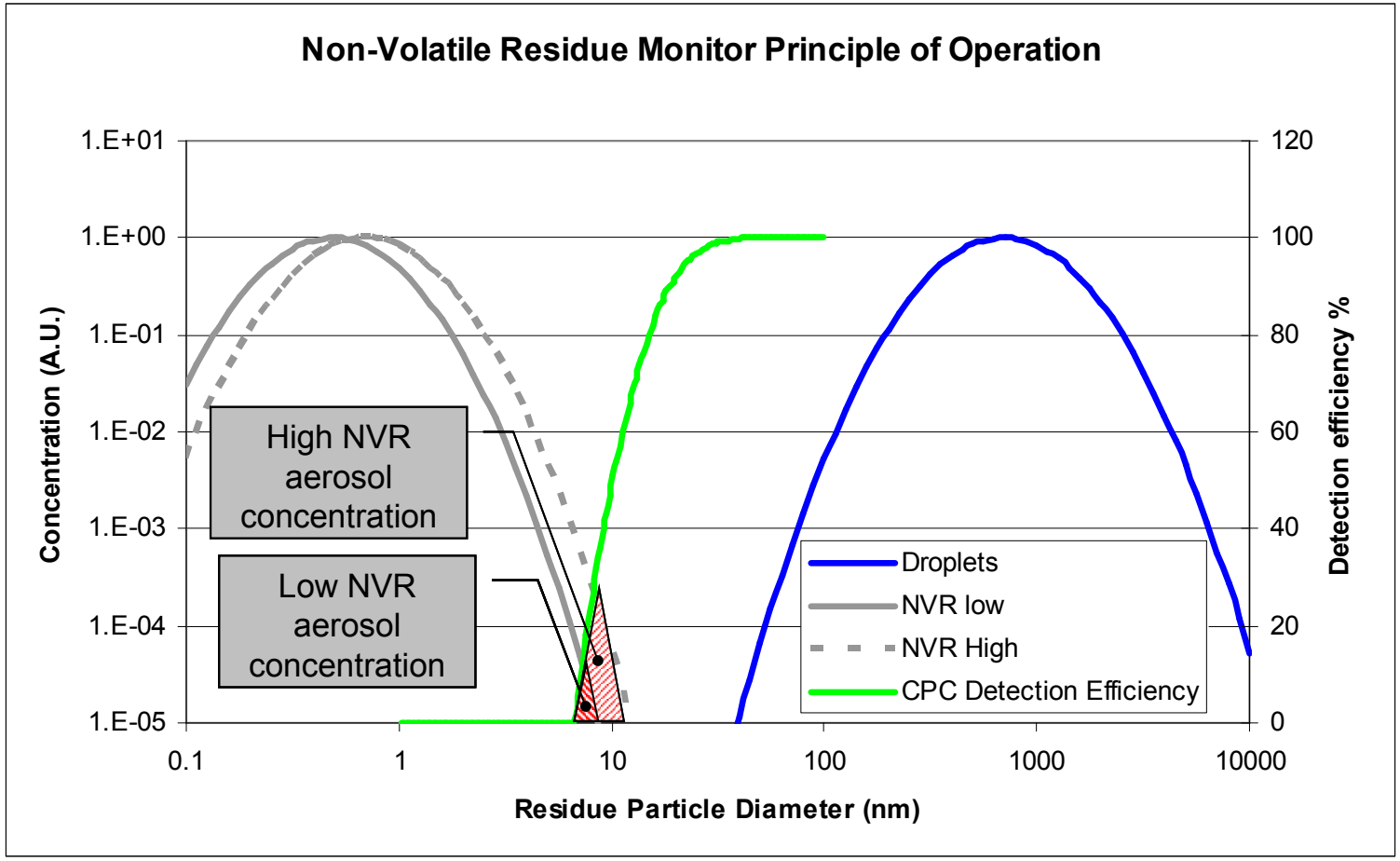
Optical Detection

Condensation Particle Counter

ULTRAPURE WATER MICRO 2015

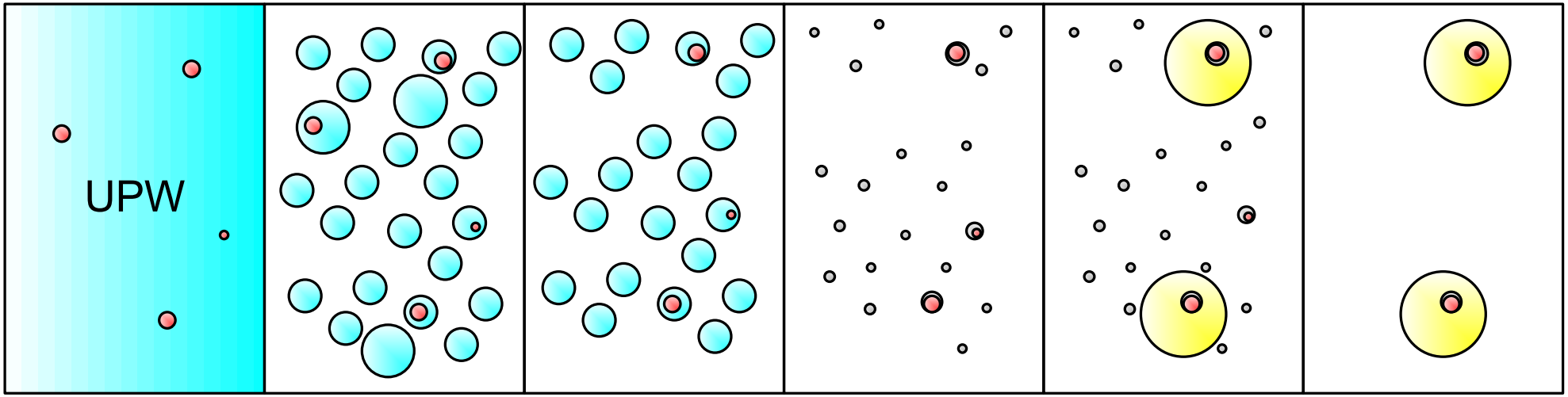
Non-Volatile Residue Monitor

- Condensation Particle Counter (CPC) detects more particles with increasing NVR concentration



ULTRAPURE WATER MICRO 2015

Scanning Threshold Particle Counter (ScanningTPC)

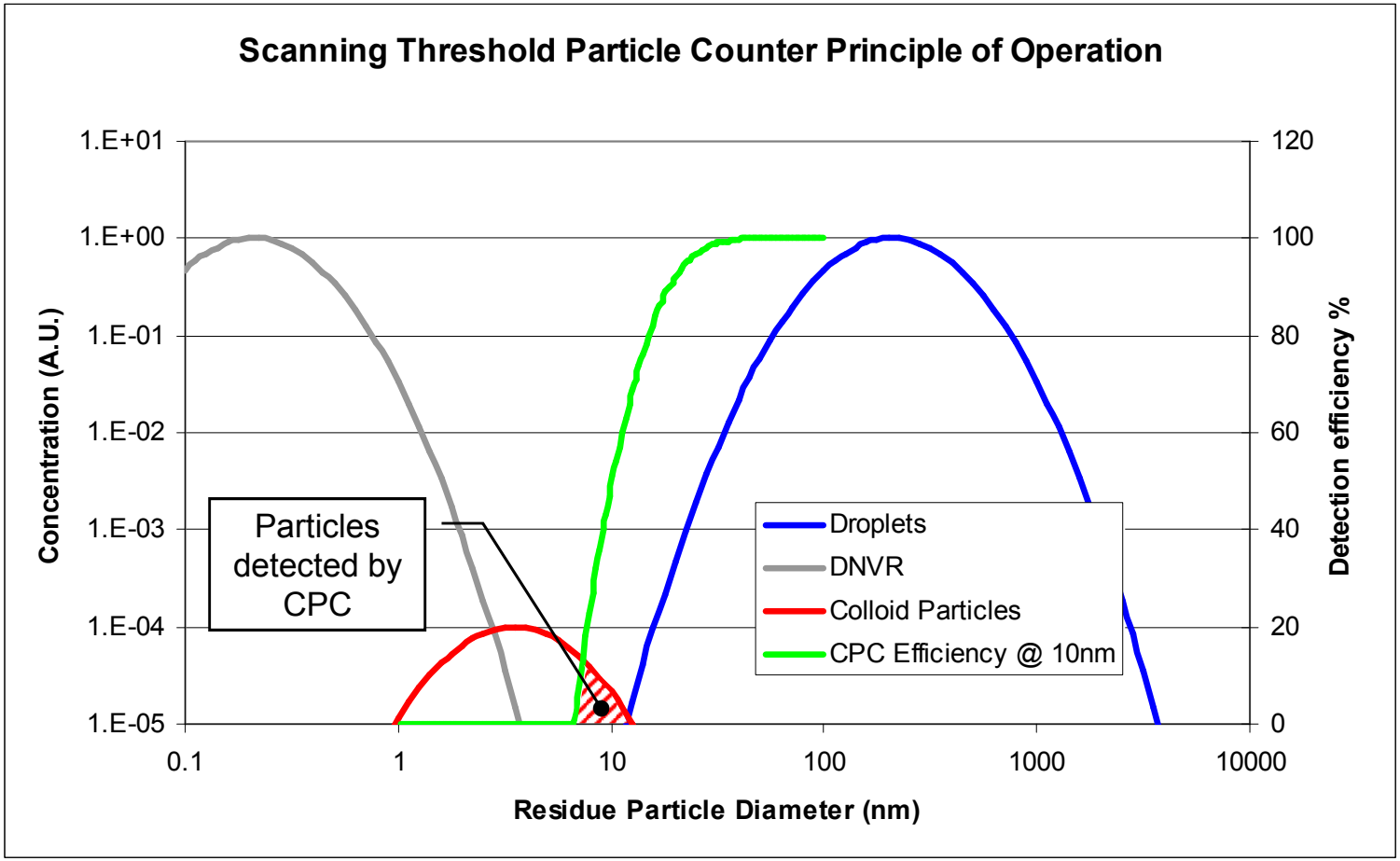


Limits large PNVR particles otherwise detected by the NRM

Only counts aerosol particles that contain insoluble liquid borne particles

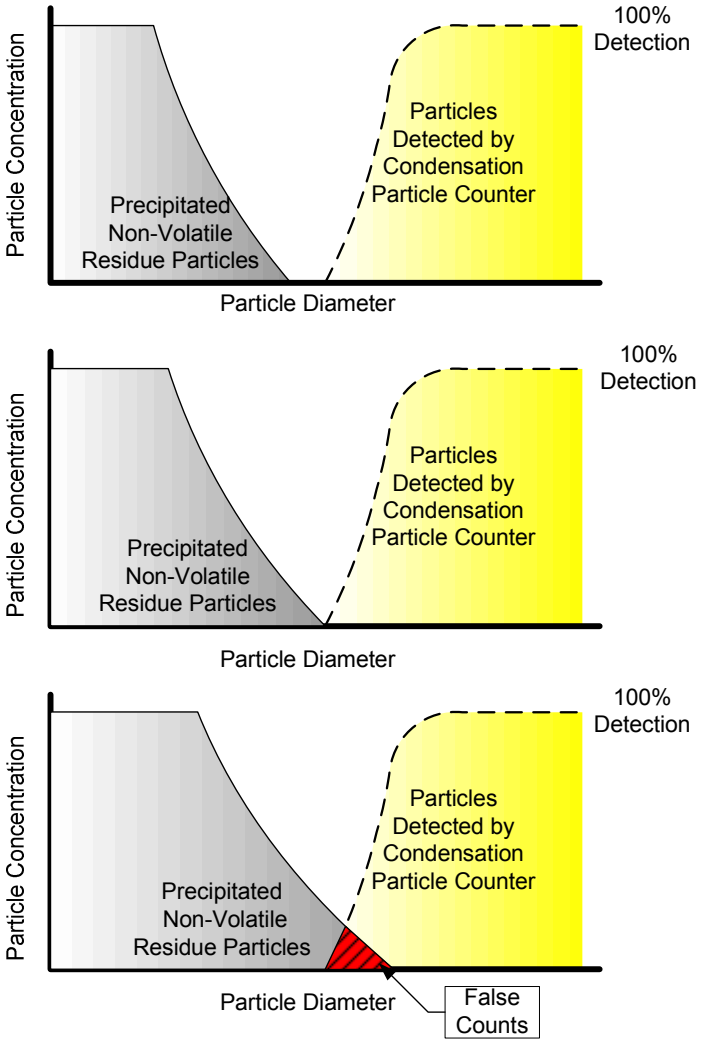
Scanning Threshold Particle Counter (ScanningTPC)

- Nebulizer produces droplets that form DNVR particles smaller than smallest size detected by CPC



Scanning Threshold Particle Counter (ScanningTPC)

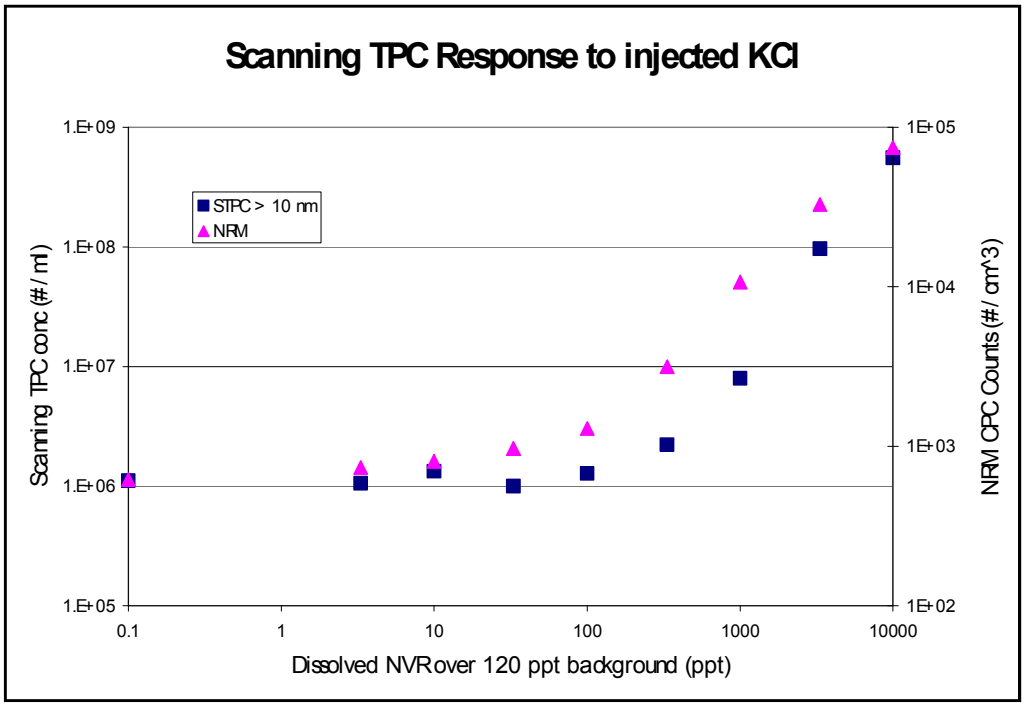
- False counts
  - CPC noise
  - Large droplets forming large precipitated residue particles
  - Coagulation of aerosol particles
  - Coincident particles within a droplet
- Total NVR ideally monitored in parallel

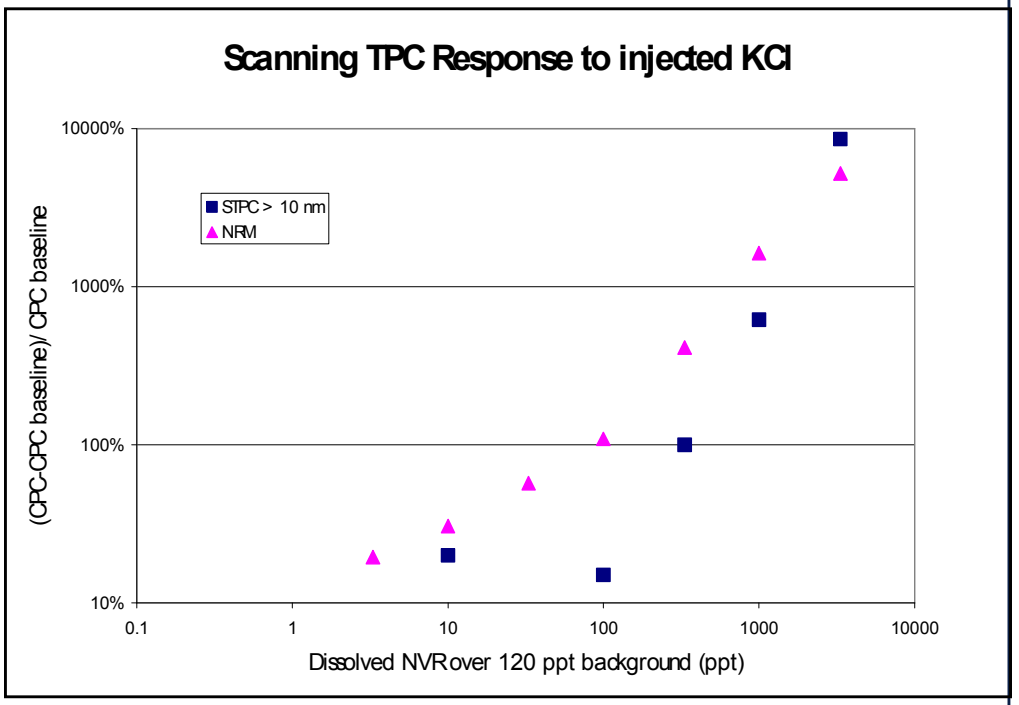


Scanning Threshold Particle Counter (ScanningTPC)

Dissolved Non-Volatile Residue Interference

- ScanningTPC does not show response when injected with low levels of DNVR



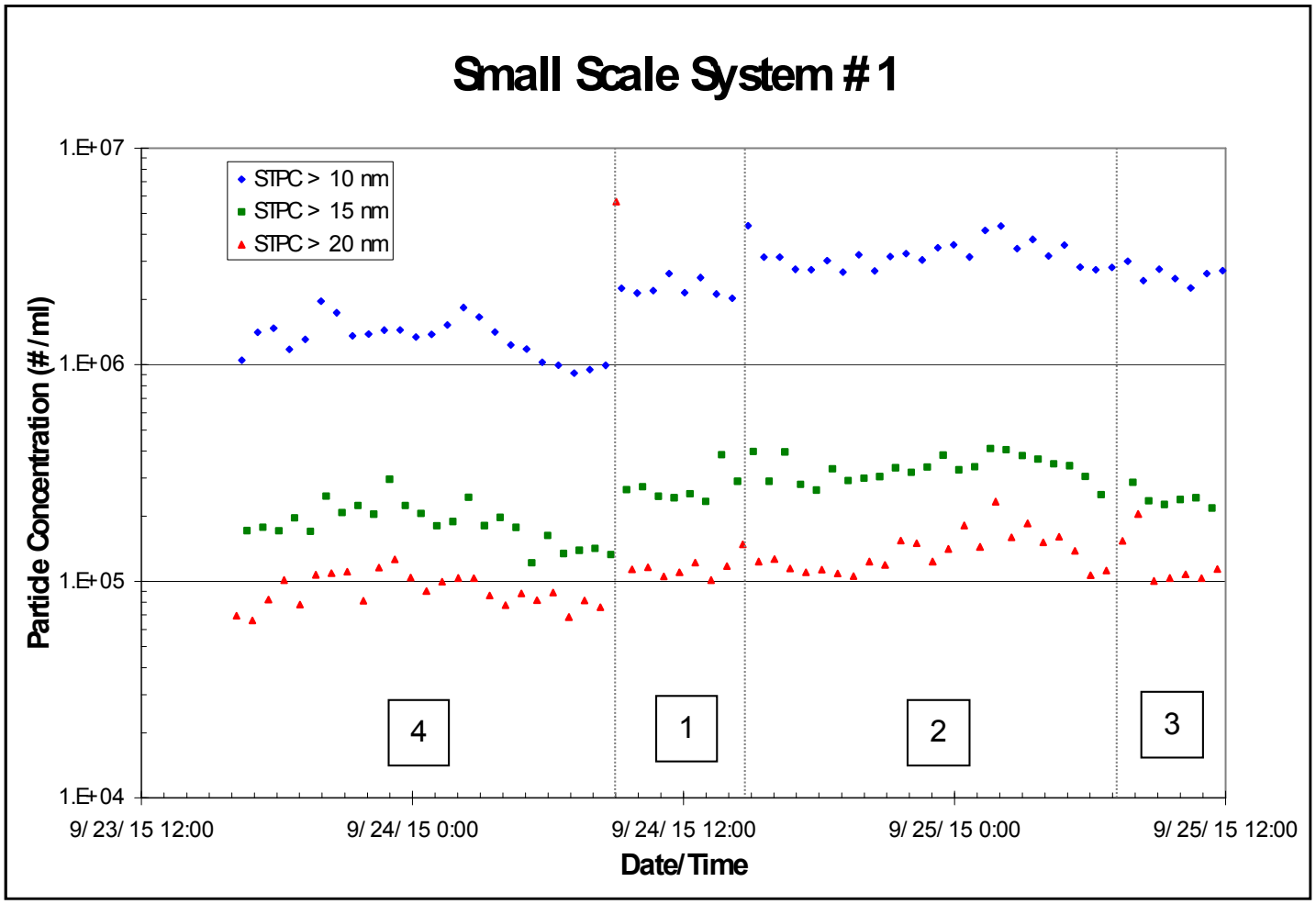


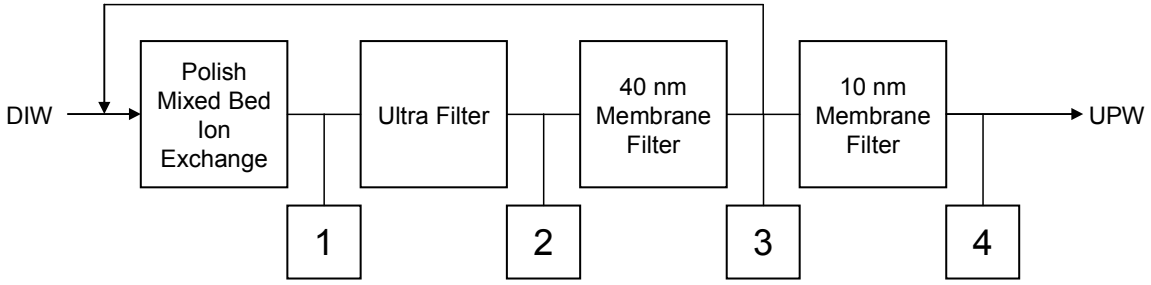
- ScanningTPC response is combination of insoluble particles in the challenge solution and precipitated NVR larger than the threshold diameter
- A portion of the response is likely due to particles in the KCl solution

ULTRAPURE WATER MICRO 2015

Scanning TPC Monitoring
Small Water System 1

- UF shows slight increase in particle counts, likely due to dirty sampling port
- High performance membrane filter shows reduction of particles at all sizes





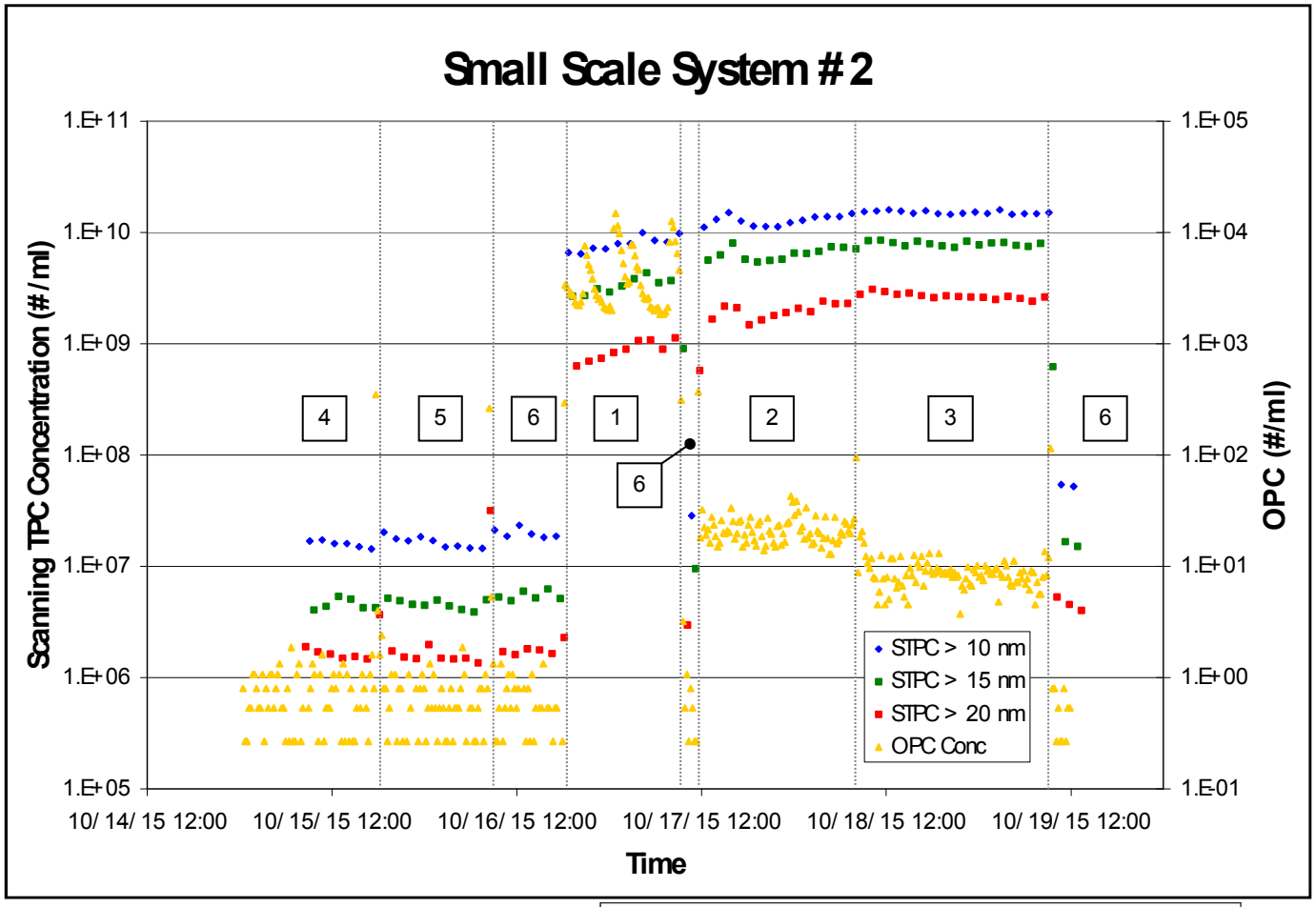
ULTRAPURE WATER MICRO 2015

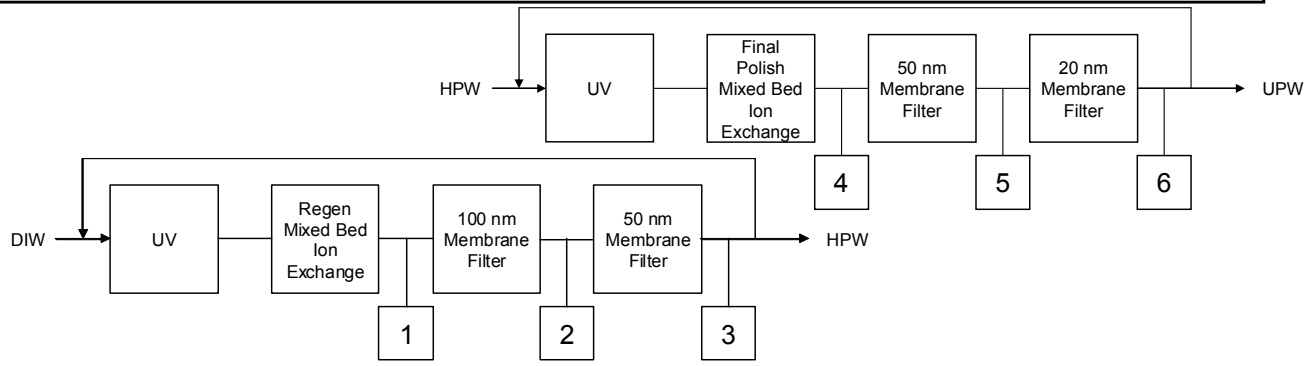
What did we learn?
Small Scale System #1

- Particle removal by 10 nm rated membrane filter measured by ScanningTPC
- Port testing should be done upstream of the mixed bed ion exchange modules to optimize UF placement
  - Upstream of polish mixed bed ion-exchange may increase resin life

Scanning TPC and OPC Monitoring
Small Water System 2

- High particle concentration observed downstream of regenerated IX resin
- 100 and 50 nm filters on HPW loop do not show strong reduction in sub 50 nm particles
- LRV of > 50 nm particles out of HPW filter set #2 is 2.5 to 3.
- A significant portion of the sub 50 nm particles appear to be removed by the final polish ion exchange resin.

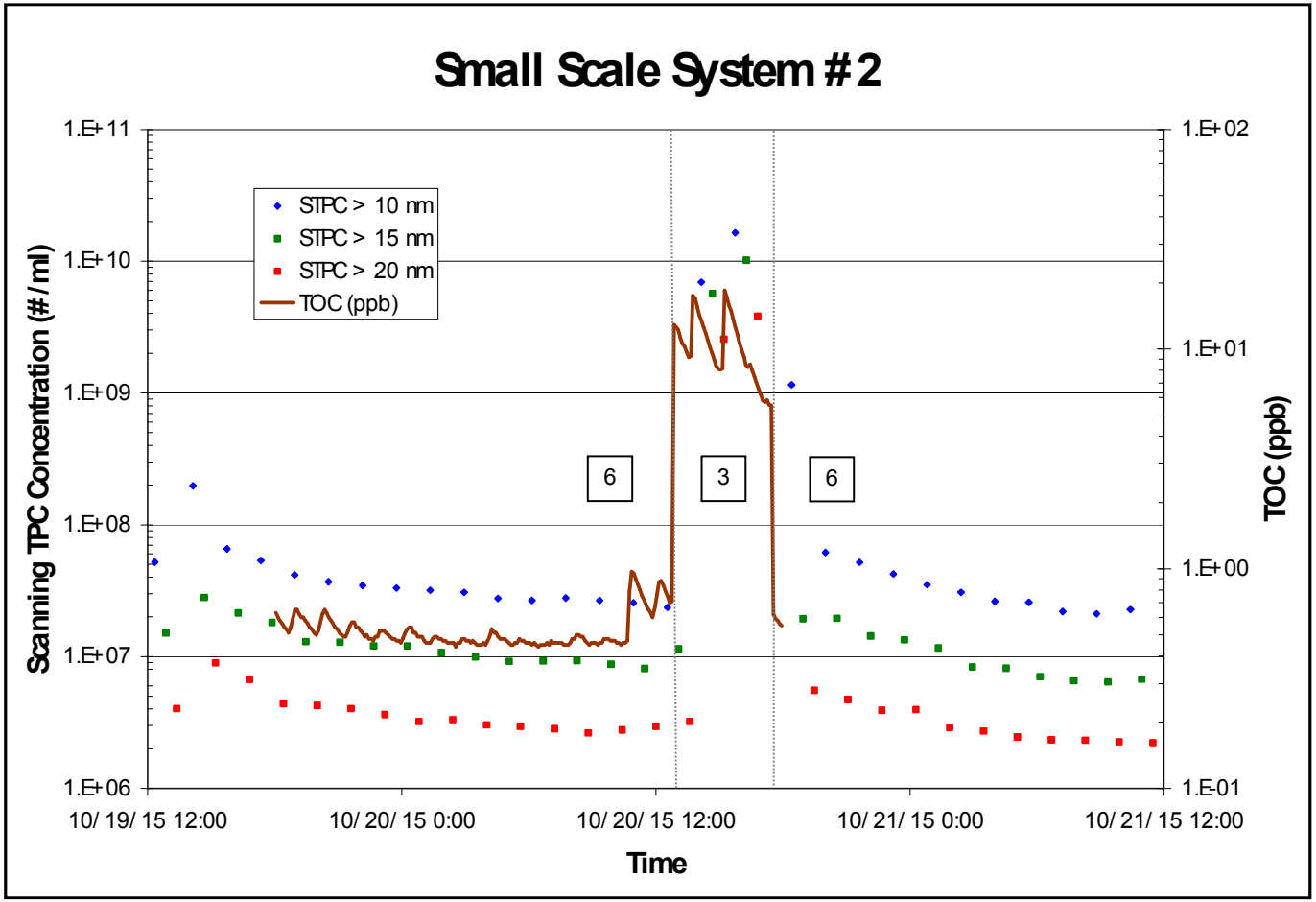


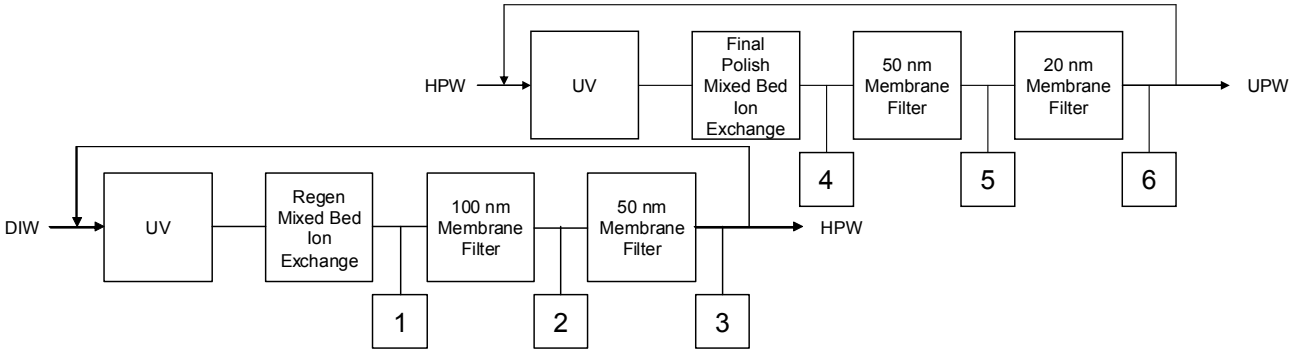


ULTRAPURE WATER MICRO2015

Scanning TPC and TOC Monitoring
Small Water System 2

- Large drop in TOC between HPW and UPW systems
- Unknown if HPW TOC is present as particles or as dissolved material





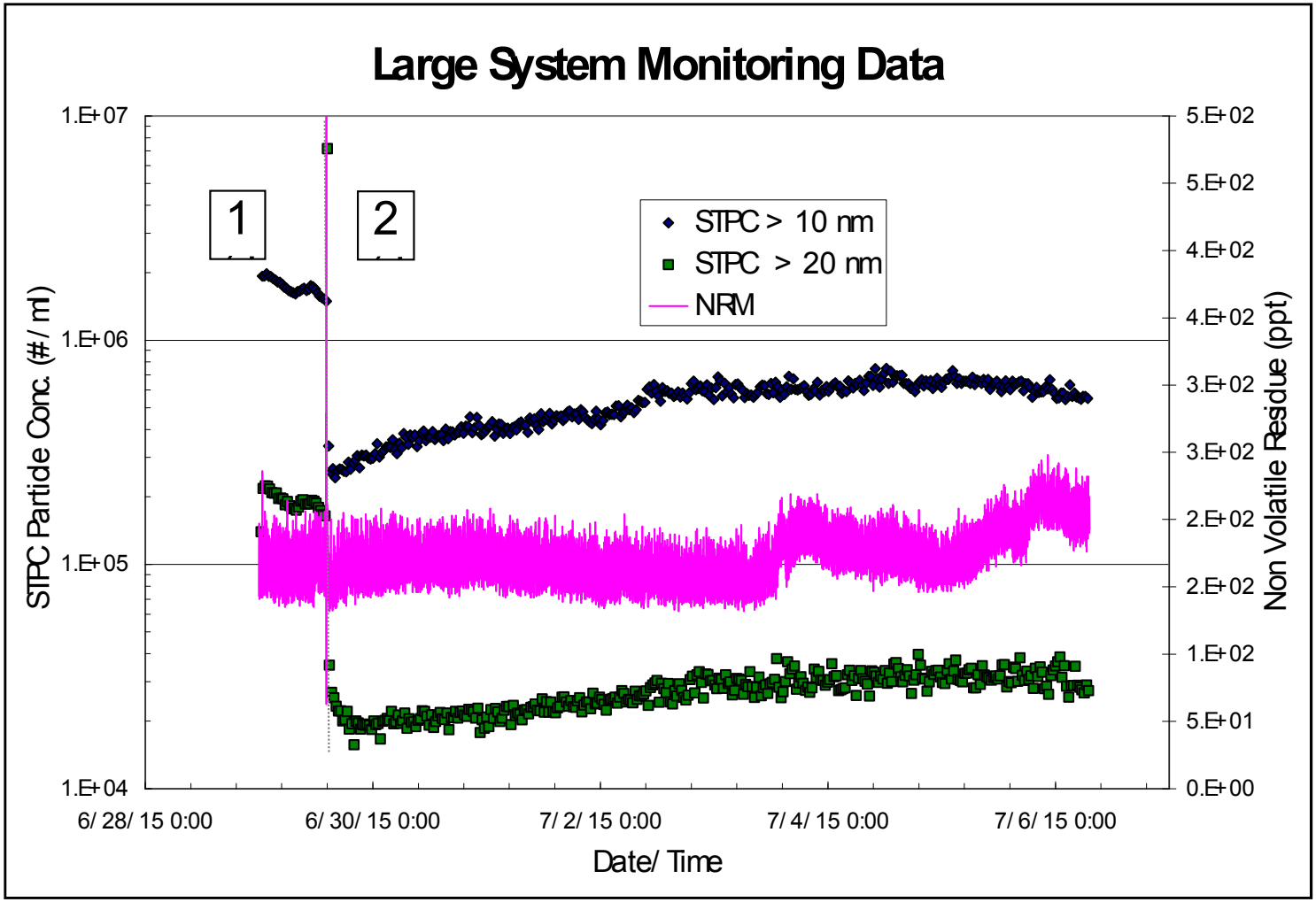
ULTRAPURE WATER MICRO2015

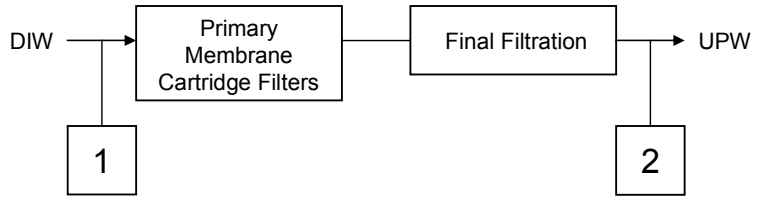
What did we learn?
Small Scale System #2

- Final polish mixed bed ion-exchange module appears to have high particle retention capacity
- UF module with a high loading capacity between the HPW and UPW loops may increase component life, specifically the virgin mixed bed ion exchange resin.
  - Test multiple placement points
  - Particles may be coming from UPW recirculation pump
- Further port testing with TOC monitor will show sources and sinks
  - How much TOC does the regenerated mixed bed resin add
  - Test additional UV module downstream of virgin ion exchange resin

Scanning TPC and NRM Monitoring
Large Water System

- STPC sees a large reduction in counts whereas the NRM does not
- Other NVR features are seen which are not detected by STPC



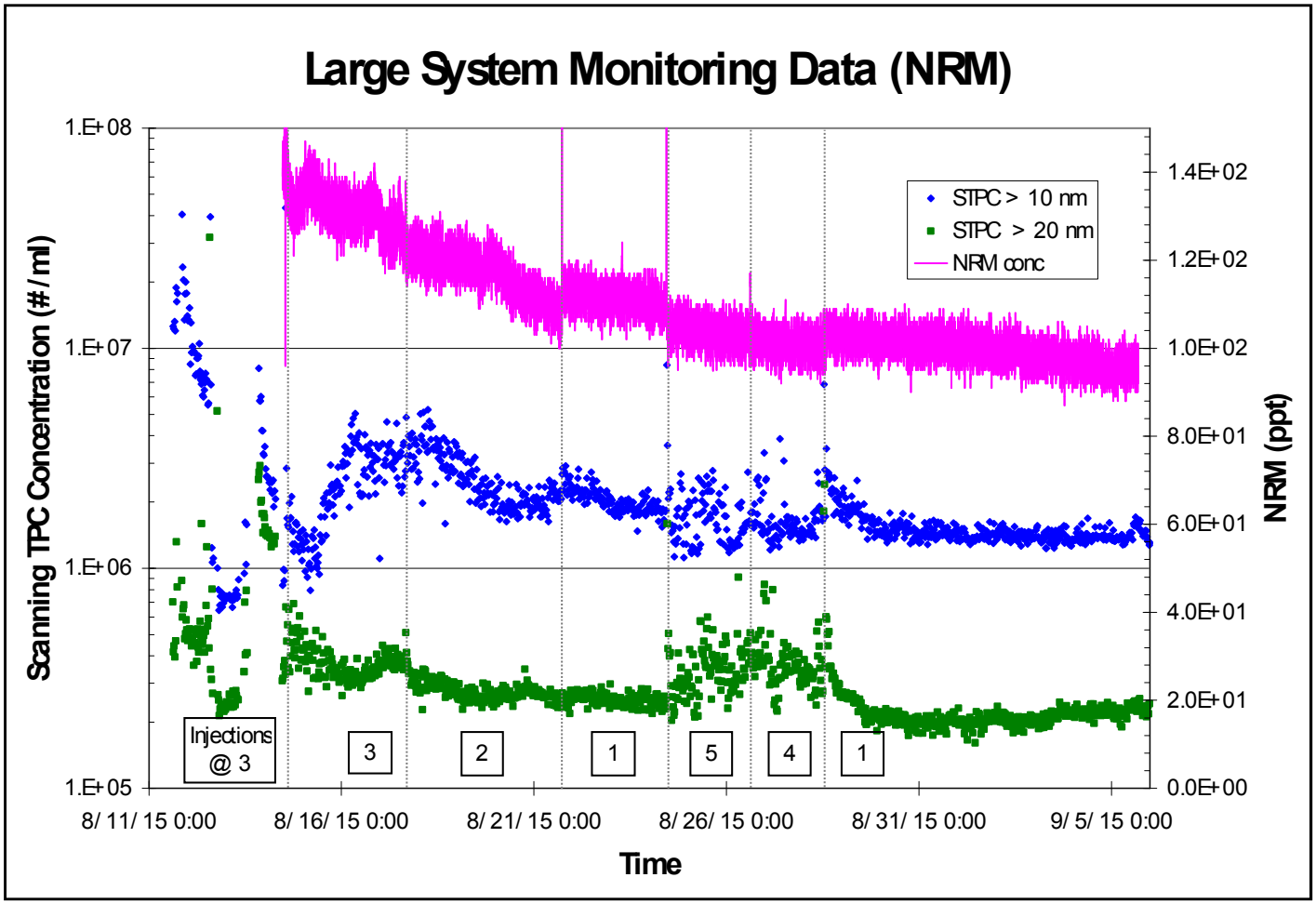


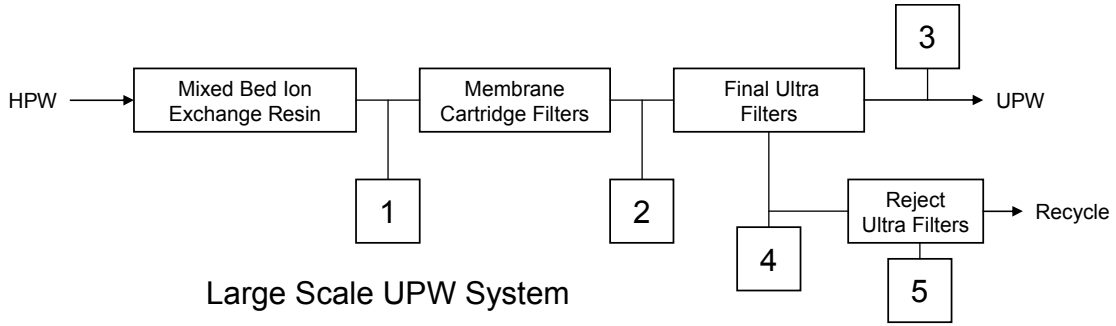
Large Scale UPW System

ULTRAPURE WATER MICRO 2015

Scanning TPC and NRM Monitoring
Large Water System

- NRM shows slight bump on cartridge filter feed
- 10 nm particles appear on 8/16-8/19. Not seen by NRM or 20 nm STPC channel



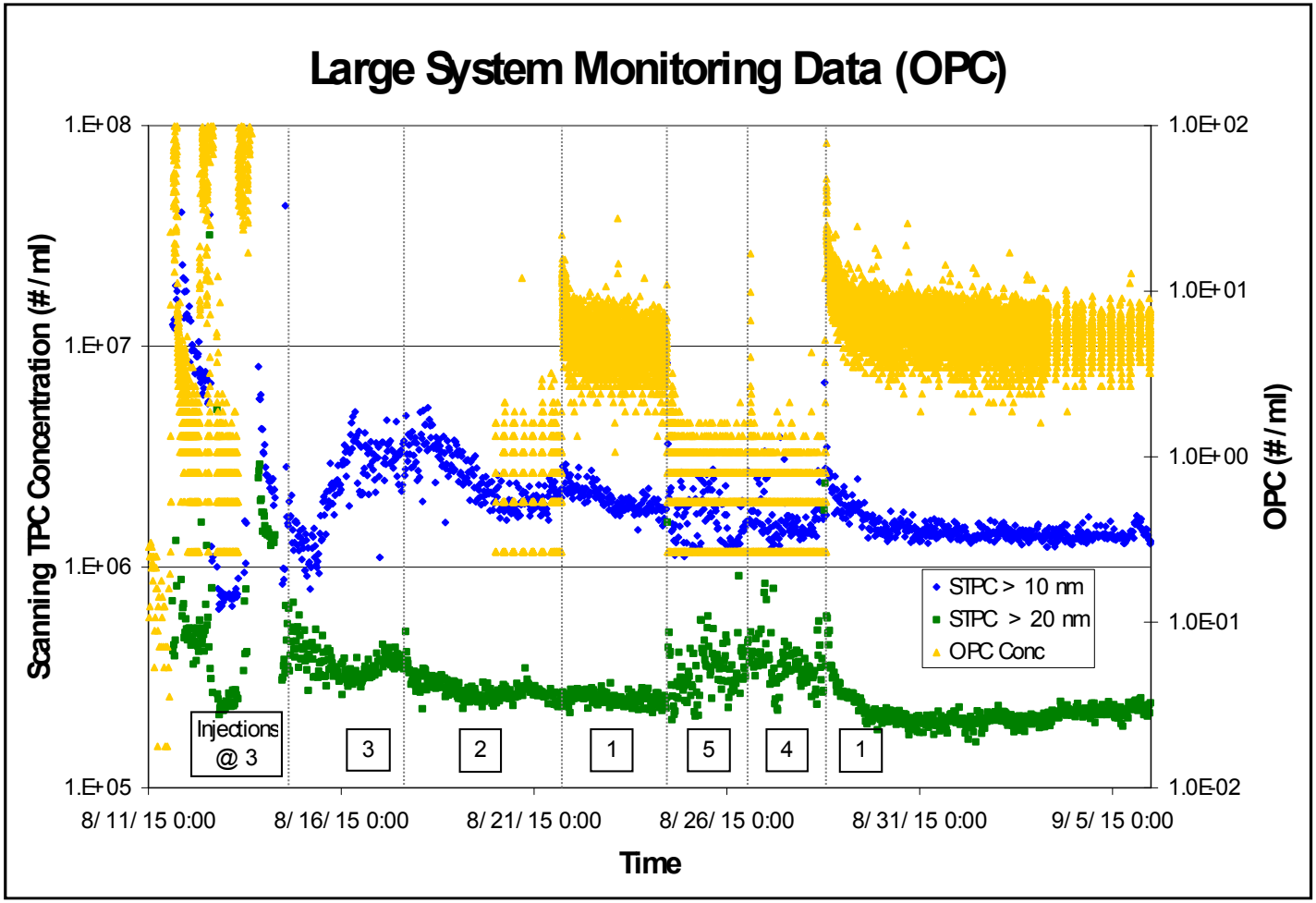


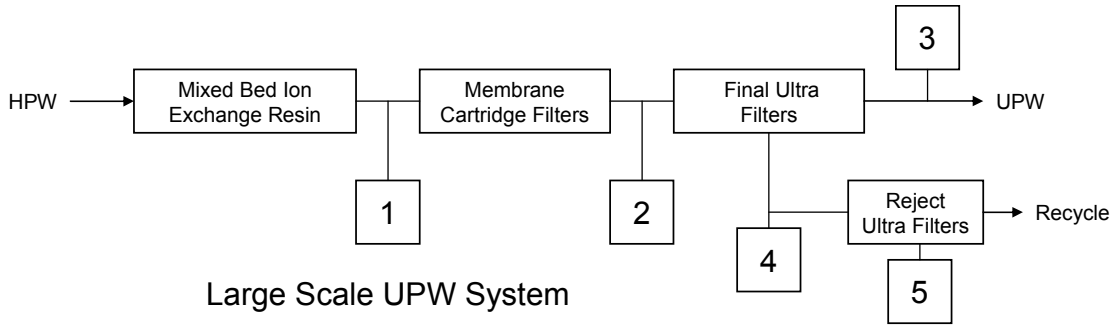
Large Scale UPW System

ULTRAPURE WATER MICRO 2015

Scanning TPC and OPC Monitoring
Large Water System

- OPC Detects change across final cartridge filter
- No difference for UF feed, product, or rejects



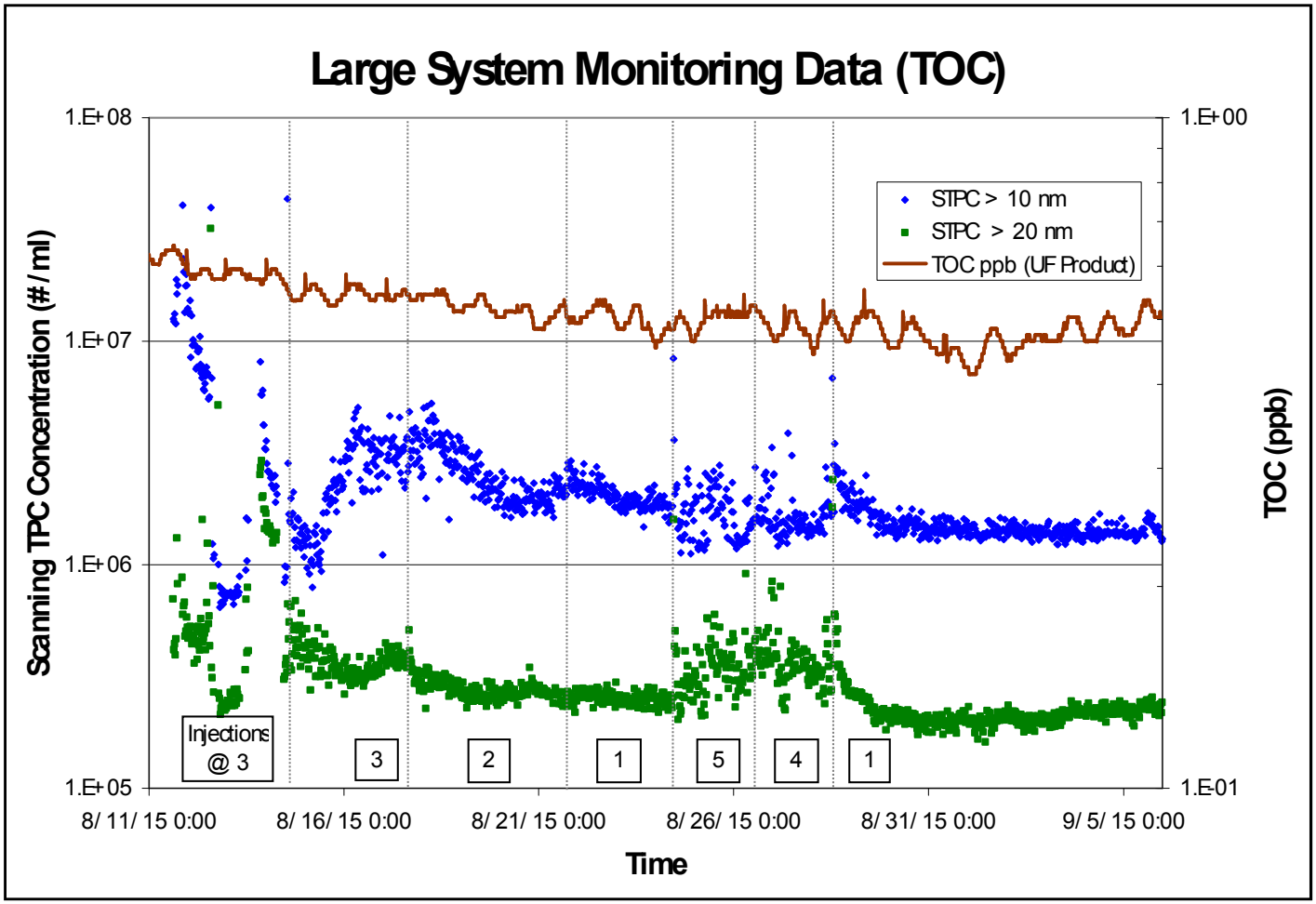


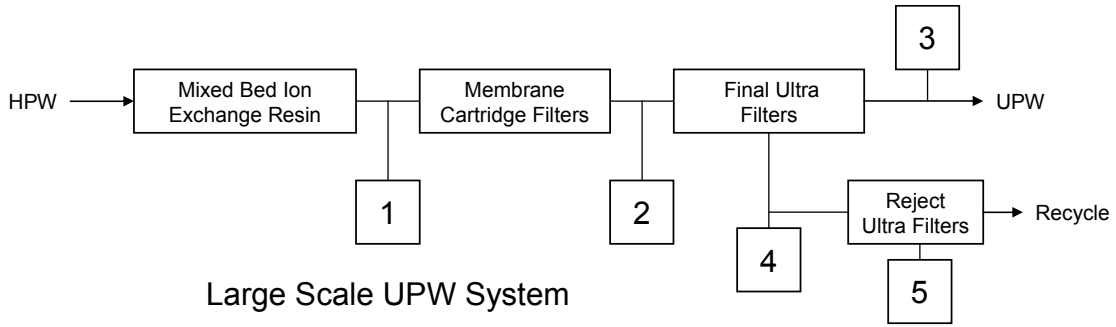
Large Scale UPW System

ULTRAPURE WATER MICRO 2015

Scanning TPC and TOC Monitoring
Large Water System

- TOC has daily cycles not seen by NRM or STPC
- 20 nm STPC channel tends to track the TOC level trend





Large Scale UPW System

ULTRAPURE WATER MICRO 2015

What did we learn?
Large Scale System #1

- Sampling should be repeated with a TOC monitor in parallel
- OPC is effective at showing large, low concentration particle removal across cartridge filters in this system
- Testing should be done downstream of a high performance point of use filter
- Data indicate that the majority of particle removal is accomplished upstream of the final filtration modules

Conclusions

- Scanning TPC and Non-Volatile Residue monitoring are able to distinguish sources of contamination in UPW
- Ensemble of instruments is useful for design and monitoring of UPW systems

The Takeaway

TOC, NRM, and ScanningTPC measurements provide different information for the UPW system engineer. Used together, these instruments aid in UPW system design and maintenance

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.

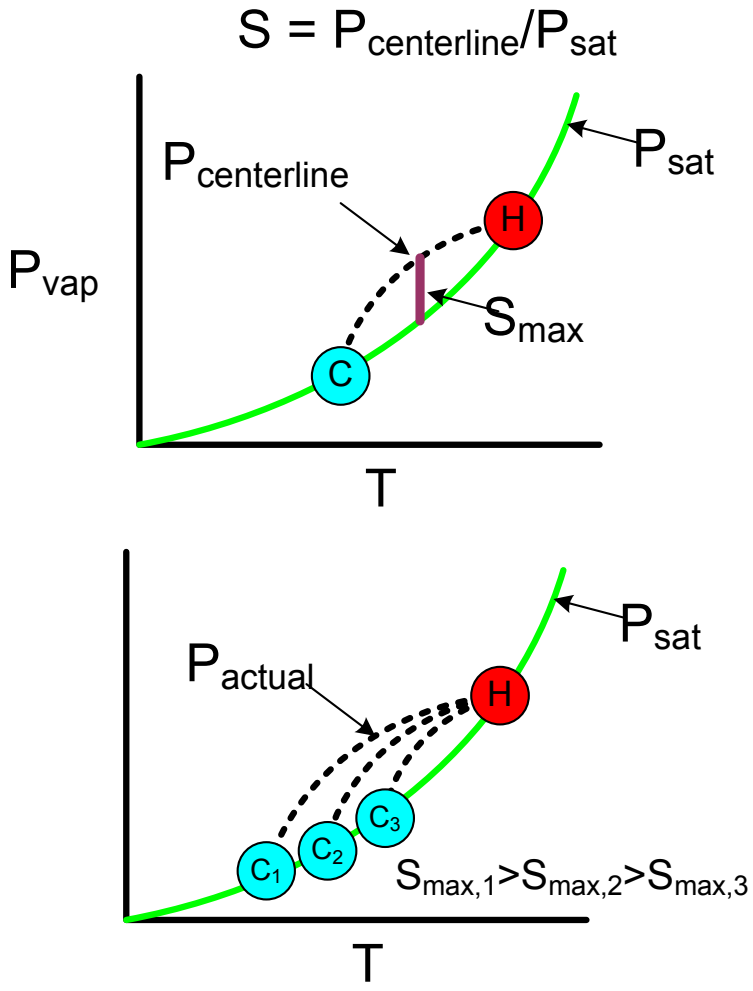
Grant DC, DC Chilcote and U Beuscher (2012). "Removal of 12 nm particles from UPW by a combination of Ultrafiltration Modules and Microfiltration Cartridges," *Ultrapure Water Journal*, May/June 2012.

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

Scanning Threshold Particle Counter (ScanningTPC)

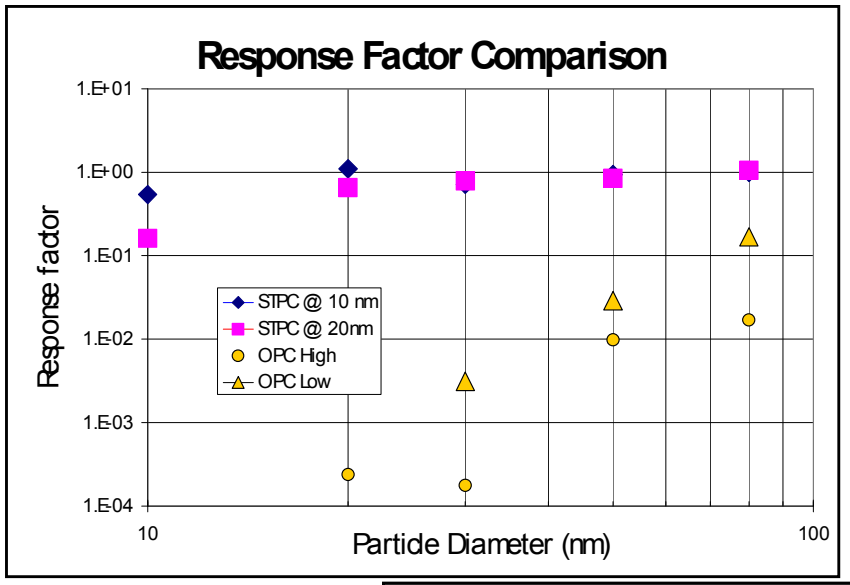
- Condensation Aerosol-Particle Counter detects particles larger than a threshold size
- Threshold size is a function of the saturation ratio, *S* of the condensing vapor
- *S* is varied by changing CPC operating temperatures

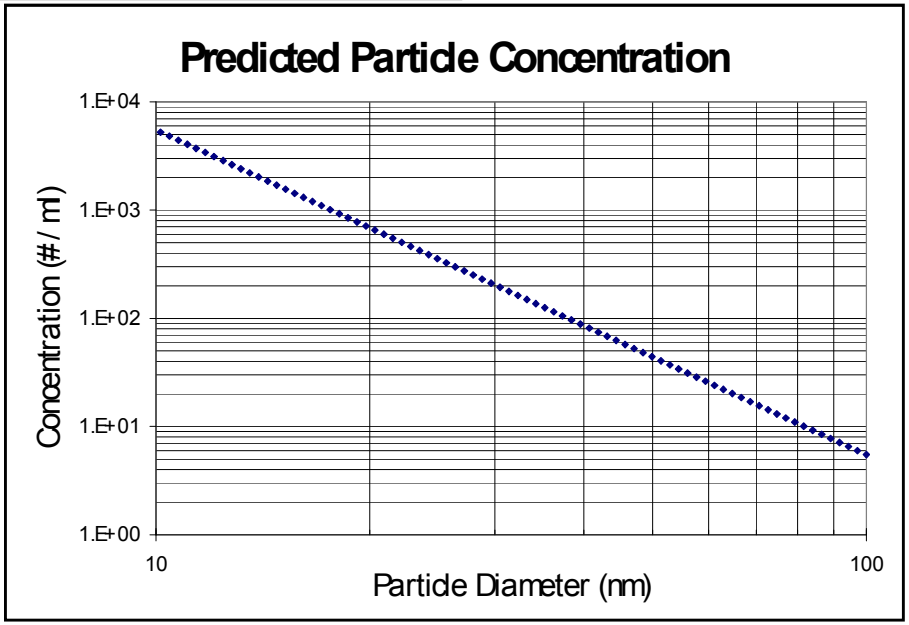


Scanning TPC and Optical Counter Comparison

Detection Efficiency

- OPC has difficulty detecting < 50 nm colloidal silica
- Low OPC background counts leads to high sensitivity for > 50 nm particles
- Background concentration at 10 and 20 nm is likely several orders of magnitude higher than reported by OPC at 50 nm



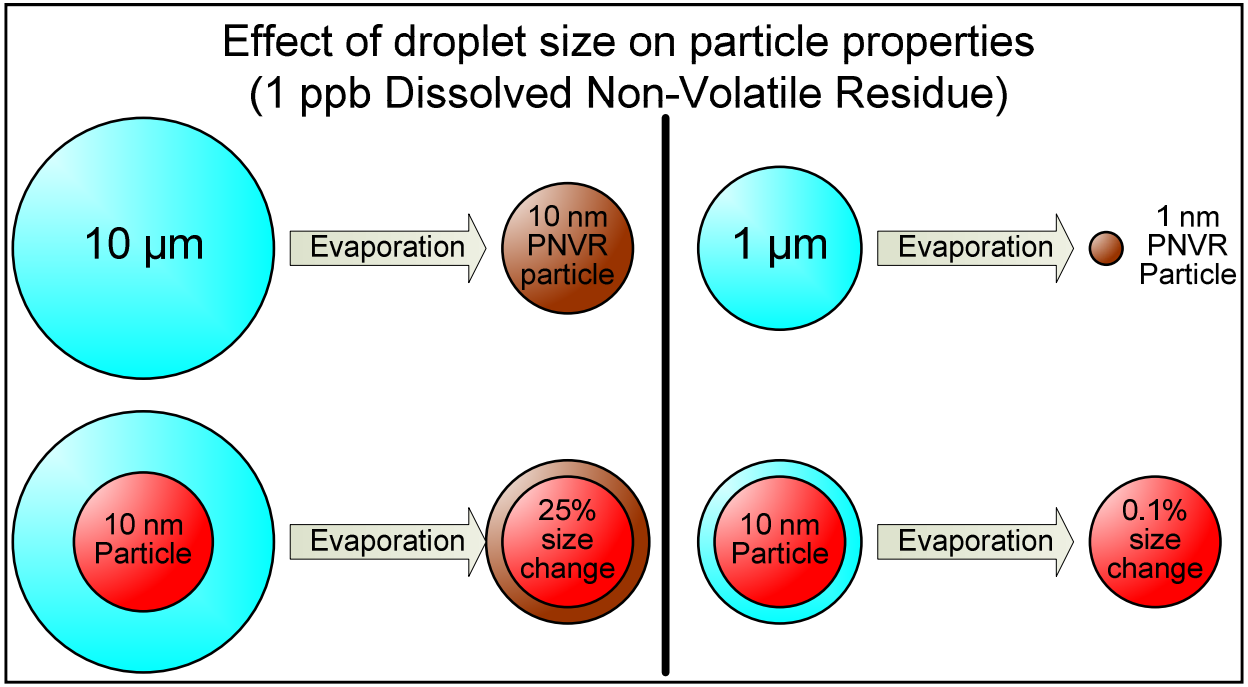


- 20 #/ml @ 65nm (corrected for OPC detection efficiency)
- -3 log-log slope

ULTRAPURE WATER MICRO2015

Droplet size and Non-Volatile Residue

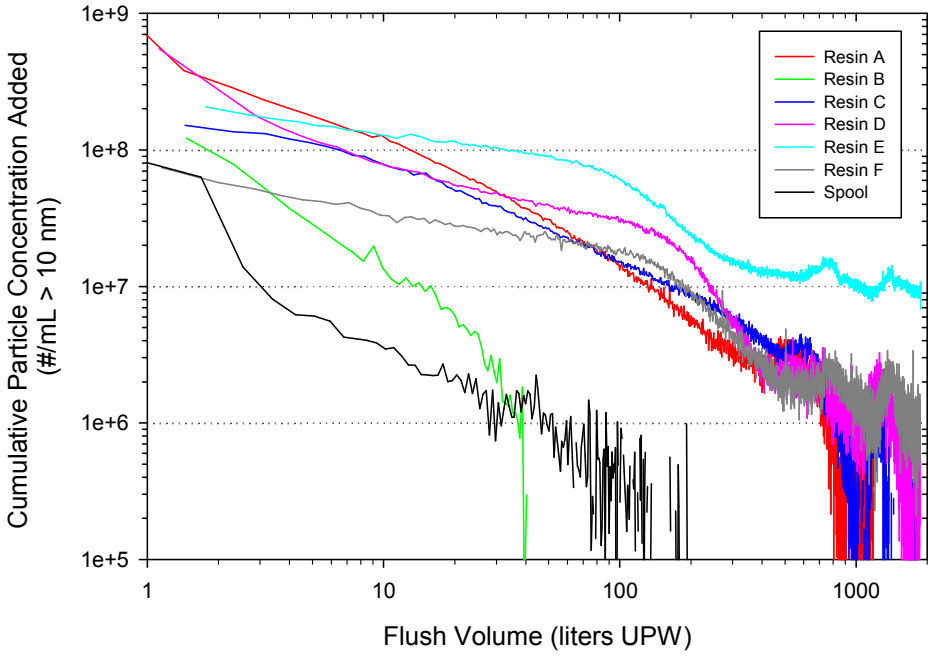
- Size of particle composed of Precipitated Non-Volatile Residue (PNVR) is proportional to its parent droplet size
- Interference of solid particle properties by PNVR is controlled by limiting the size of large droplets and operating on systems with low levels of Dissolved NVR (<500 ppt)



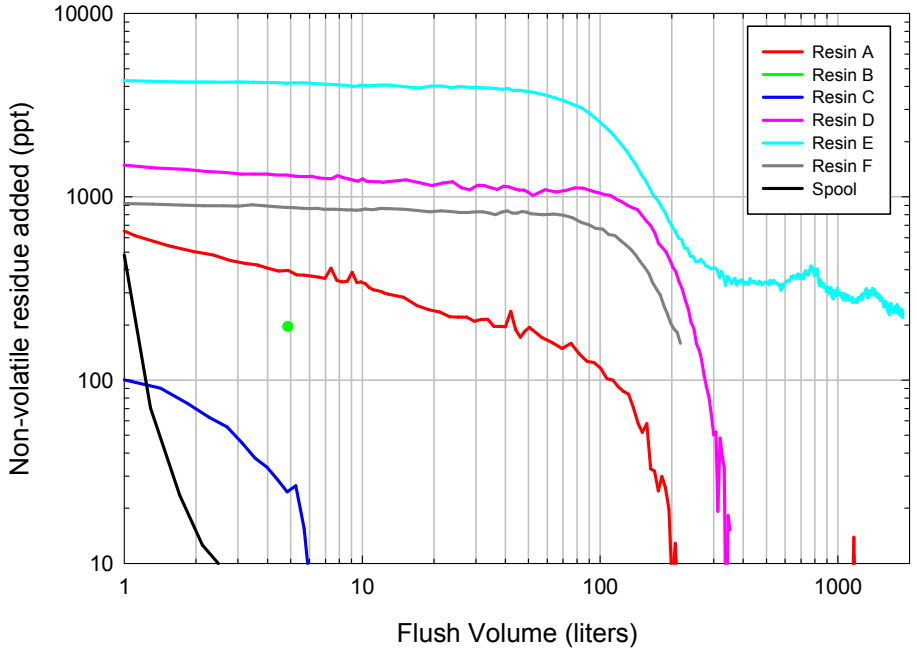
ScanningTPC and NRM Monitoring
Resin rinse

- NVR composition during resin rinse varies by manufacturer

Particle Rinse



Non-volatile Residue Rinse



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

ULTRAPURE WATER MICRO 2015