PARTICLE MEASURING SYSTEMS®

Without measurement there is no control

Continuous Monitoring of Particles at 20 nm in Critical Semiconductor Process Chemicals

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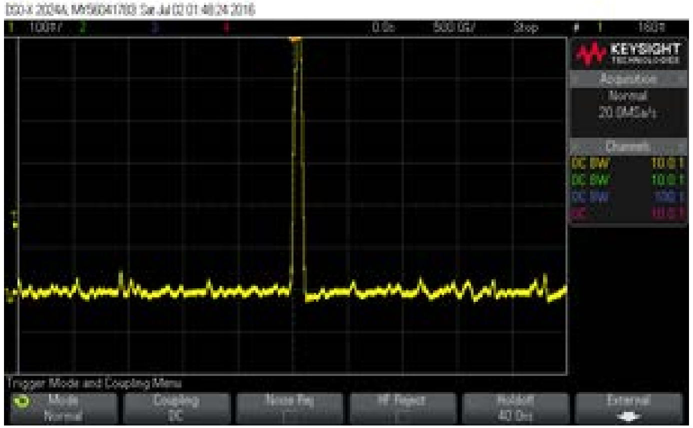
Confidential and proprietary

Monitoring chemicals - SEMI & chemical operations

- Semiconductor Operations
  - Incoming quality measurement
  - On-line monitoring of distribution and blending systems
  - Monitor chemical filtration and recirculation
- Chemical Processing & Packaging
  - Qualification of chemical filters and components
  - In-line testing of packaging systems
  - Outgoing chemical qualification testing

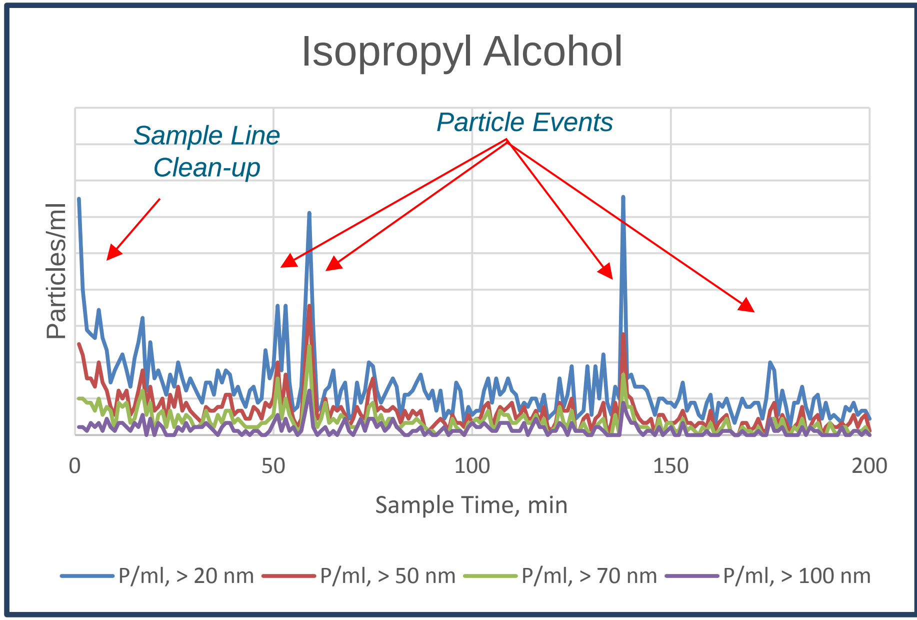
20 nm particle measurement in chemicals

- React quickly to particle excursions before surface scan or yield data are available
- Improved sample population statistics for tighter process control
- Opportunity for statistical analysis
- Optimize chemical delivery systems from the loading dock to point-of-process



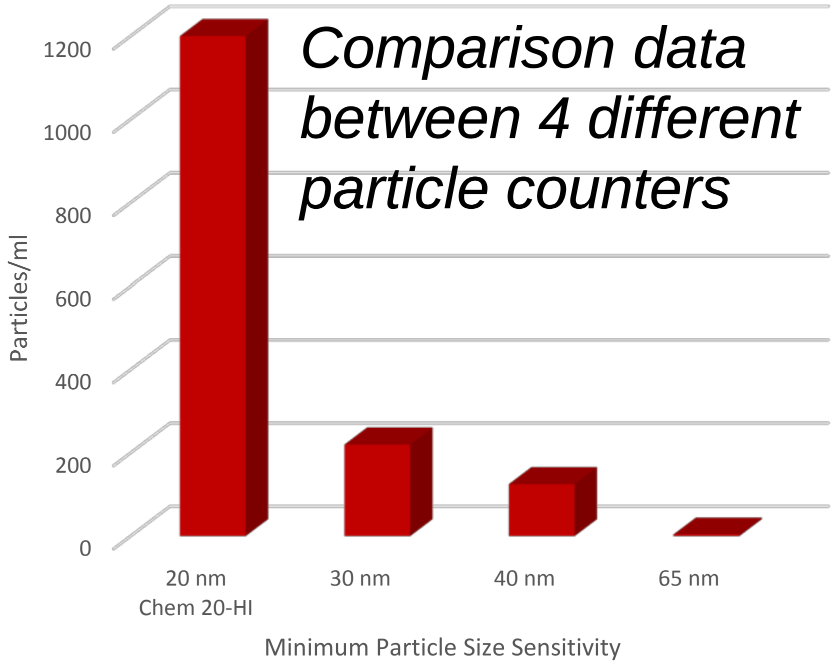
Information from a particle counter

- Level of particle contamination (#/ml)
- Particle contamination events
- Trends in particle contamination
- Particle size distribution
  - Indicate filtration performance
- Poisson’s statistics
  - Indicate a systematic or non-random source of contamination
- Fourier transform analysis of cyclic contamination
- Contamination source isolated/addressed



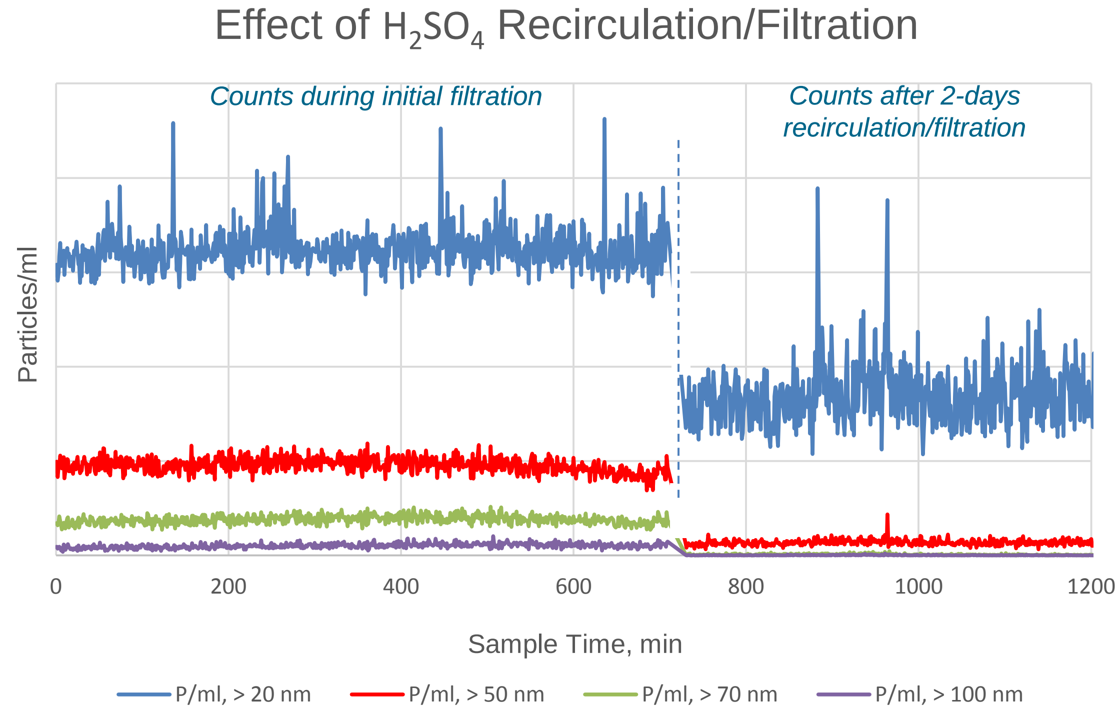
Why 20 nm?

- Detect particles you can't see with lower sensitivity
- Monitor effectiveness of advanced particle filtration
- Identify changes in batch-to-batch chemical purity
- Characterize and eliminate particle sources
- Protect wafer cleaning processes from upsets in chemical quality


Comparison data between 4 different particle counters

More particles detected at 20 nm—better process control

Impact from Recirculation/Filtration - 96% sulfuric acid

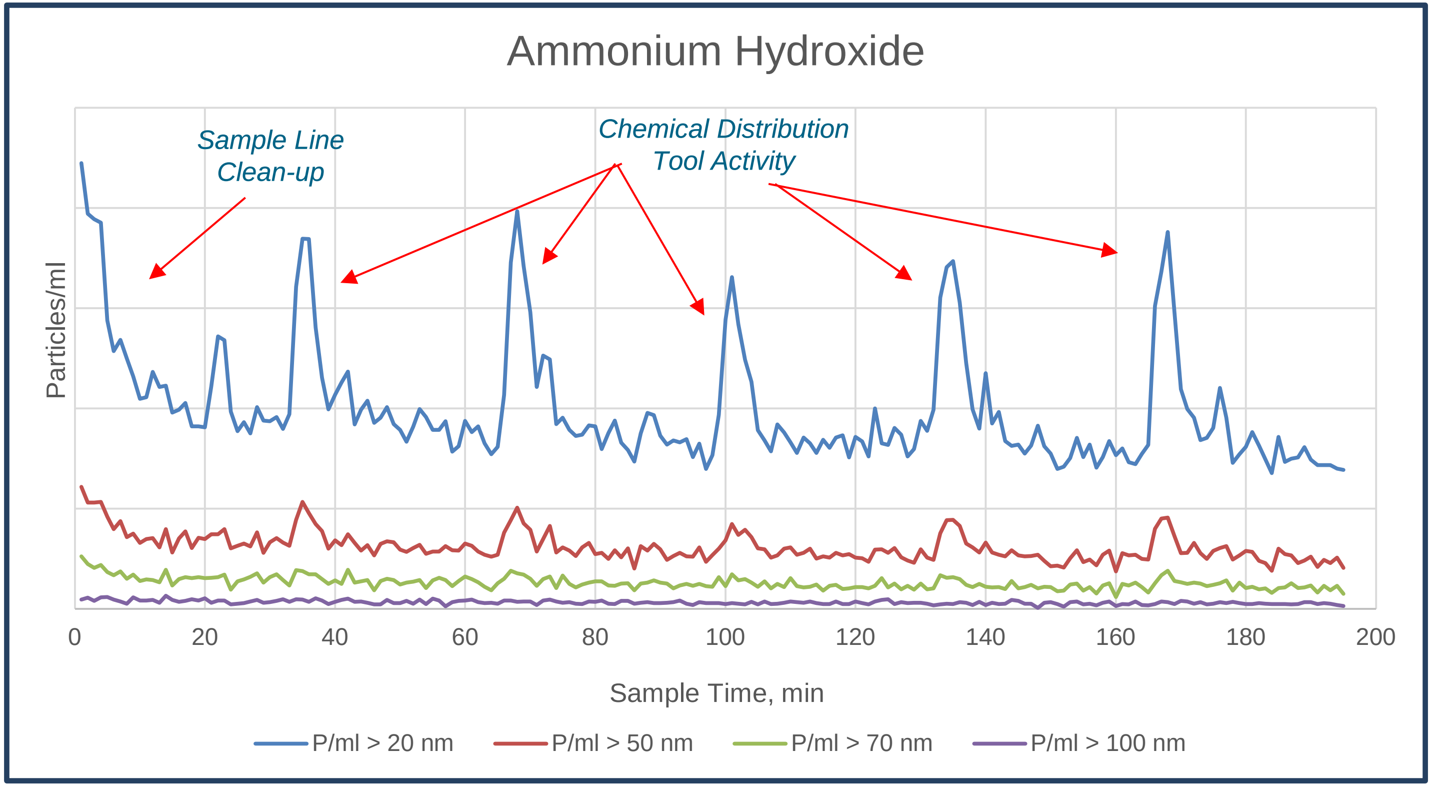


Size, nm	% Reduction
20	47.5%
50	85.6%
70	97.5%
100	98.0%

Particle Reduction after Recirculation

- Recirculated flow with filtration very effective at removing larger particles

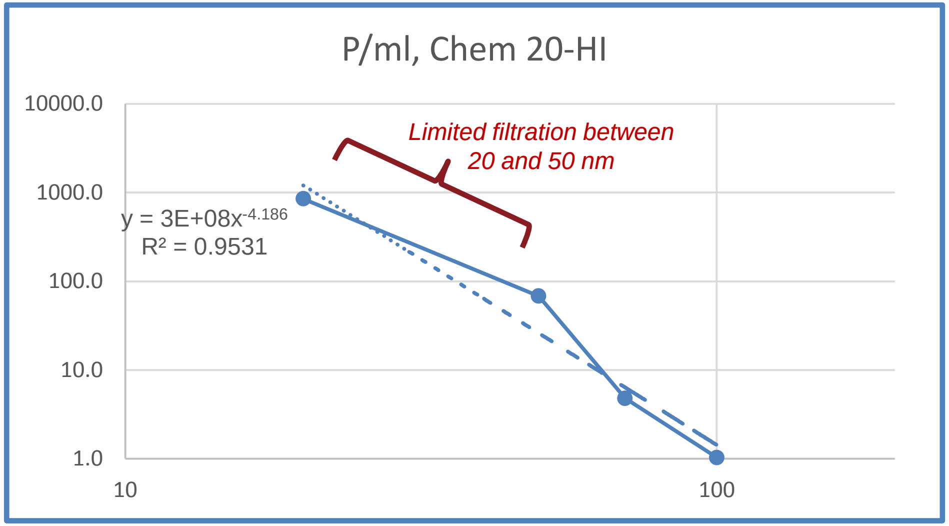
Example: 29% Ammonium hydroxide



- Activities in the distribution system can cause significant surges in contamination

Particle size distribution

- Particle size distribution—plot on log-log and look at slope (-4.186)
- In H_2SO_4, PSD slope ranges from -2.5 to -4.5
- In filtered chemistry, filtration mainly determines PSD slope
  - Filter removal efficiency relative to particle detection efficiency



Poisson Statistics Review

- Useful for calculating the probability that a particle will be detected in a sample interval

$$P(k \text{ events in interval}) = \frac{\lambda^k e^{-\lambda}}{k!}$$

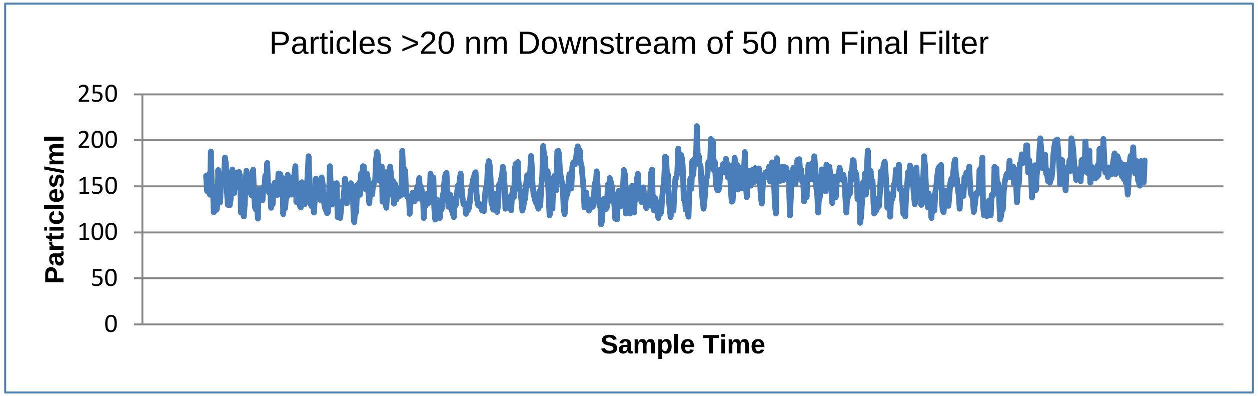
  - k is number of times a particle can be detected in an interval; 0, 1, 2, …
    - Applies to raw particle counts
  - λ is the average number of particles per sample interval
  - Standard deviation = square root of the mean, $\lambda^{1/2}$
- Key assumptions
  - Particle detection events occur randomly and independently
  - Events in one sample interval do not affect the probability of particle detection events in the next sample interval
  - The average rate of particle detection events is constant
  - Probability of detection in a sample interval is proportional to sample interval length

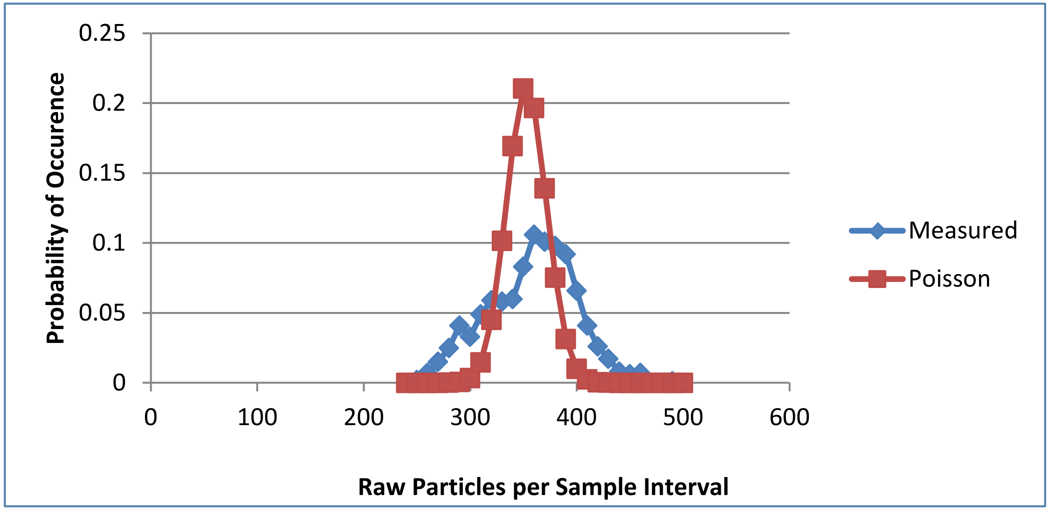
Implications of Poisson Statistics

- Ideal particle count rate occurs with Poisson distribution
- Actual particle rate deviates from Poisson distribution when:
  - Particle detection rate does not vary in a random manner
  - Contamination source is not random
- Deviations from Poisson statistics indicates a systematic contamination source
  - Identify & eliminate contamination sources in systems
  - Examples—valve switching, pressure pulses, changes in usage or flow.
- Quantify the deviation from Poisson as:
  - **DFP = $[(\sigma_{measured}/\lambda^{1/2}) - 1] \times 100\%$**

Ultrapure water system #1

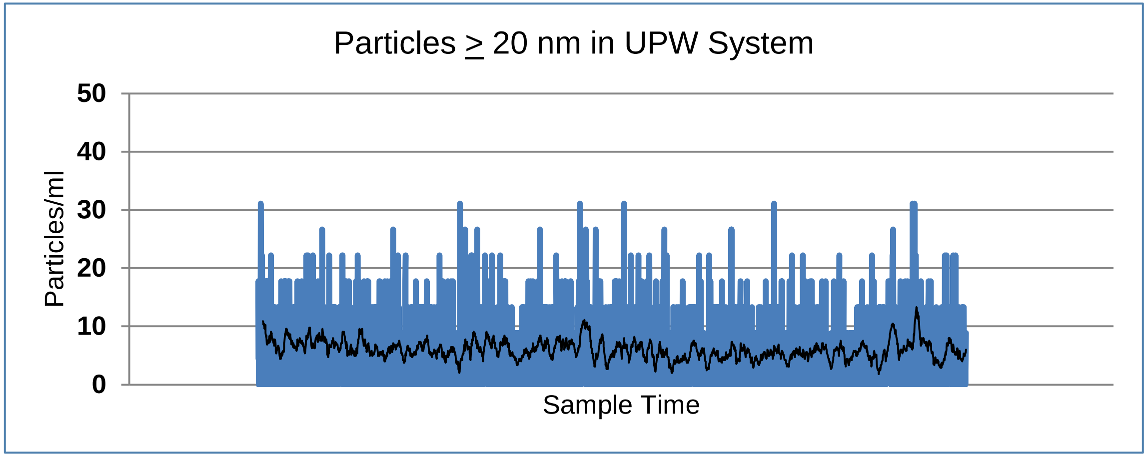
- $\lambda = 353$
- $\sigma_{measured} = 40.8$
- $\lambda^{1/2} = 18.8$
- **DFP = 117.4%**
- System not very clean
  - No ultra-filter
- Definitely not Poisson behavior

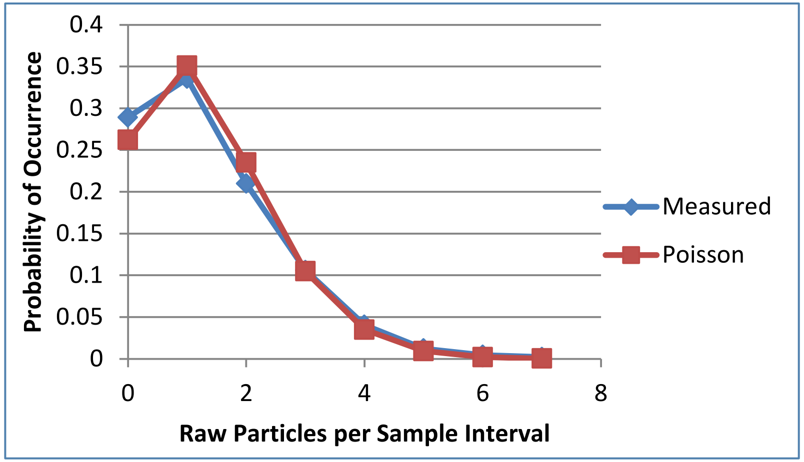




Ultrapure water system #2

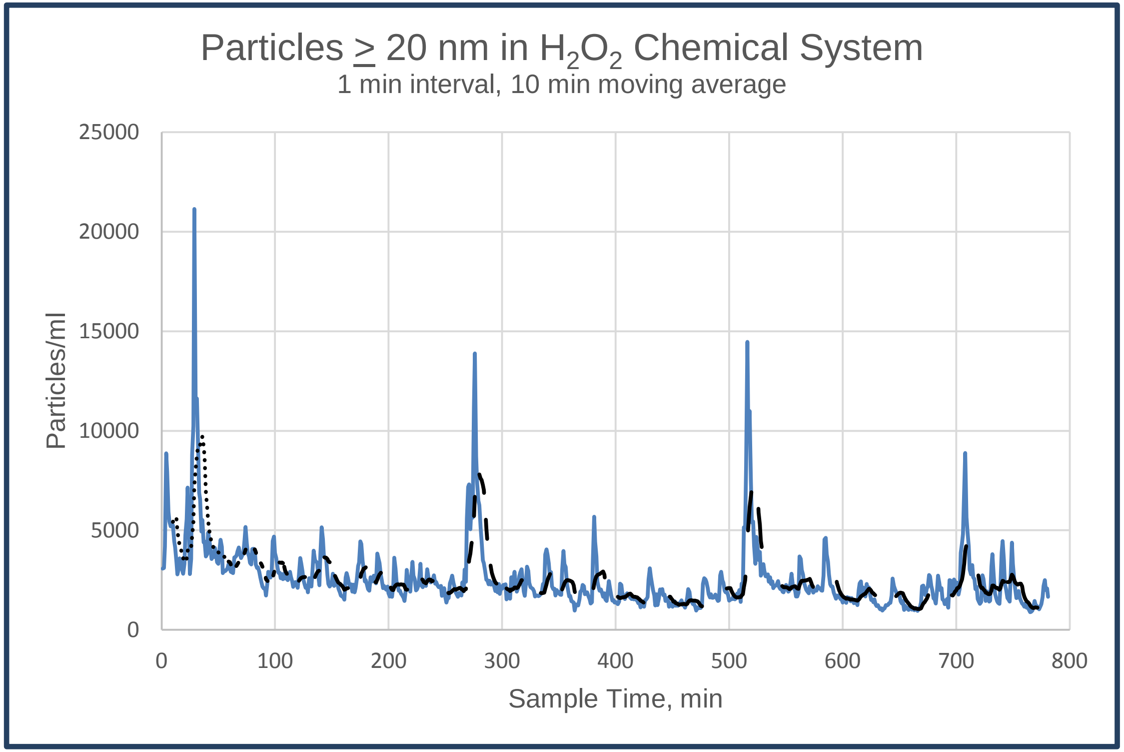
- $\lambda = 1.34$
- $\sigma_{\text{measured}} = 1.25$
- $\lambda^{1/2} = 1.16$
- **DFP = 8%**
- Not the cleanest
- Very stable/well controlled
- Minimal non-random contamination





Example: Chemical System--31% H_2O_2

- $\lambda = 1165$
- $\sigma_{measured} = 768.9$
- $\lambda^{1/2} = 34.13$
- **DFP = 2153%**
- Definitely not Poisson behavior
- Indicates systematic contamination sources



Summary of Poisson Analysis

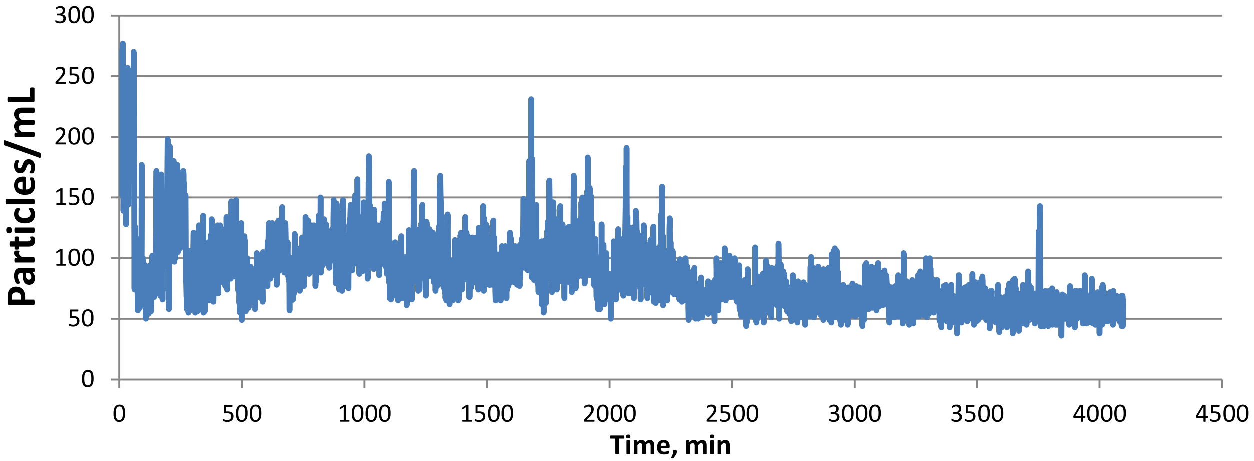
Parameter	**UPW System #1**	**UPW System #2**	**Chemical System-- H_2O_2**
P/ml, $\geq$ 20 nm	157	5.96	1165
DFP	117.4%	7.8%	2153%
Configuration	Old 50 nm Filter, no UF	Electronic System, with Ultra Filtration	50 nm Filtered H_2O_2 Distribution System
Cleanliness	Not very clean	Nominal Cleanliness	Very Unclean
Stability	Very Unstable	Very Stable	Extremely Unstable

- Ultrapure water systems
  - Significantly cleaner and better-controlled than chemicals at 20 nm
- Chemical distribution systems
  - Comparatively higher particle levels
  - More systematic, non-random particle sources
    - Large opportunities for improvements!
  - Not as stable as ultrapure water systems

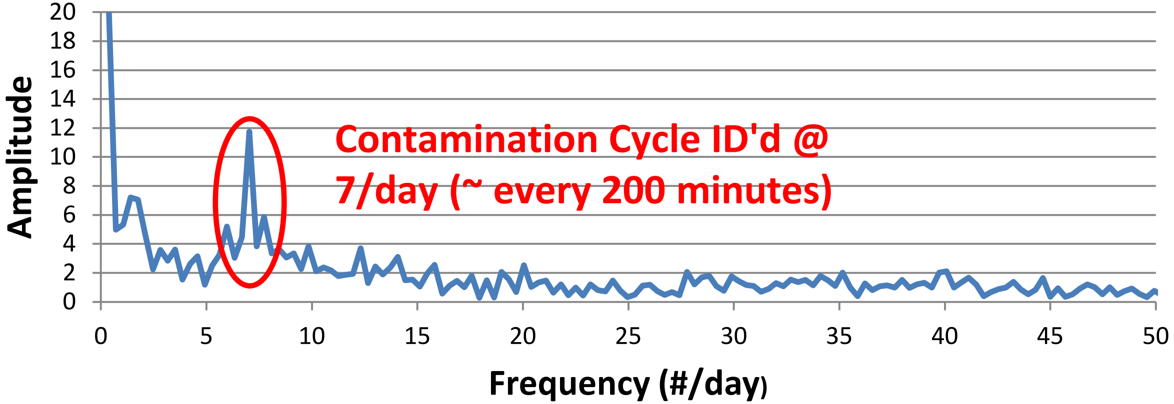
Next: Fourier Transform Analysis of Particle Contamination

- Use FFT to analyze frequency domain of contamination
- Isolate cyclical contamination sources

Particle Contamination in Sulfuric Acid



Fast Fourier Transform Analysis Particle Contamination



Contamination Cycle ID'd @ 7/day (~ every 200 minutes)

Summary

- Continuous, real-time data - react quickly to nanoparticle excursions
- Measure both the contamination level and the deviation from Poisson statistics
  - Quantify impact of system improvements (filtration, pumps, etc)
- Chemical distribution systems generally have large systematic contamination sources
  - Easy to see in 20 nm data
  - Fourier transform analysis to identify cyclic contamination
  - Measure it – control it