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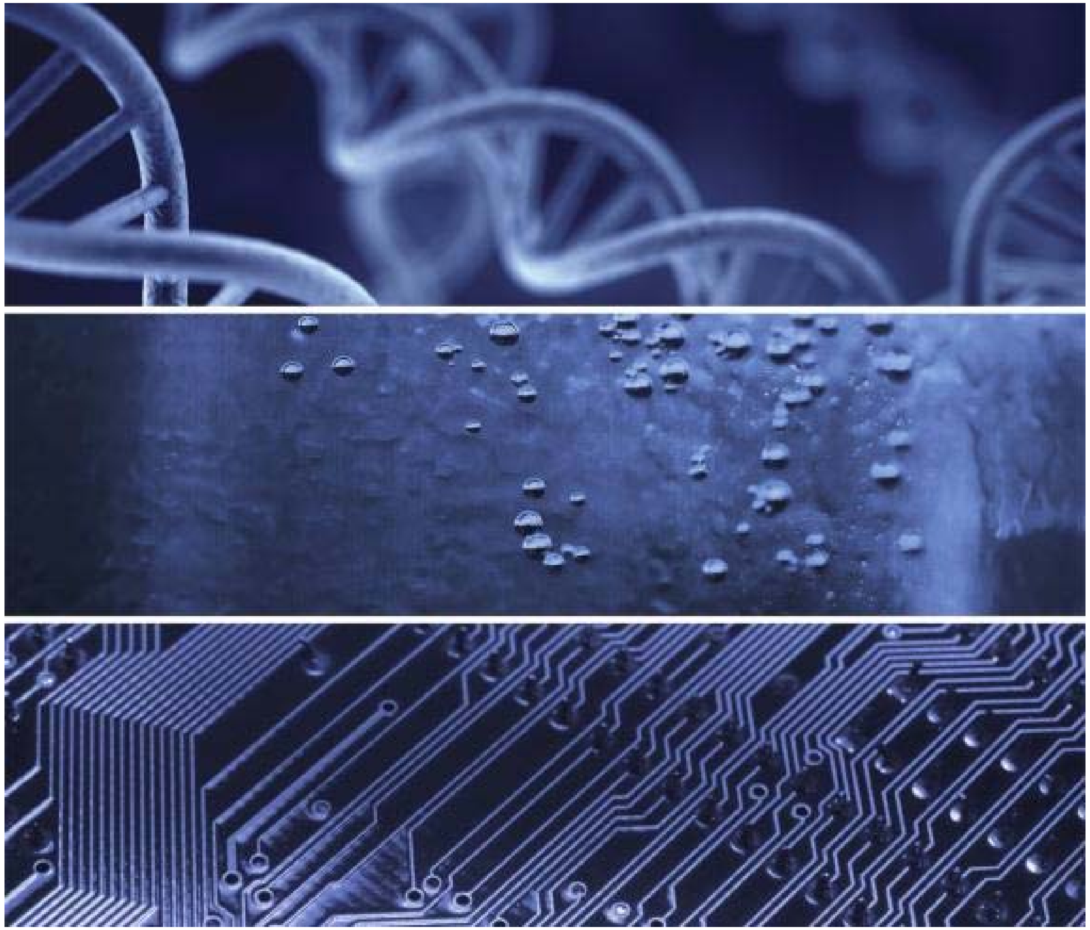

Particle counter utilization for Filter-clean up characterization under unfavorable conditions

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April 2018

WET User Meeting, Amsterdam

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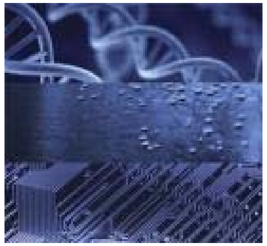
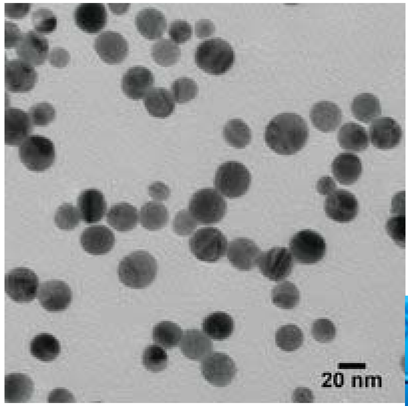
Outline

- Introduction and Motivation
- Particle Counting Techniques
- Background of testing
- Test conditions
- Light scattering vs. acoustic results
- Conclusions, Path Forward



Introduction and Motivation

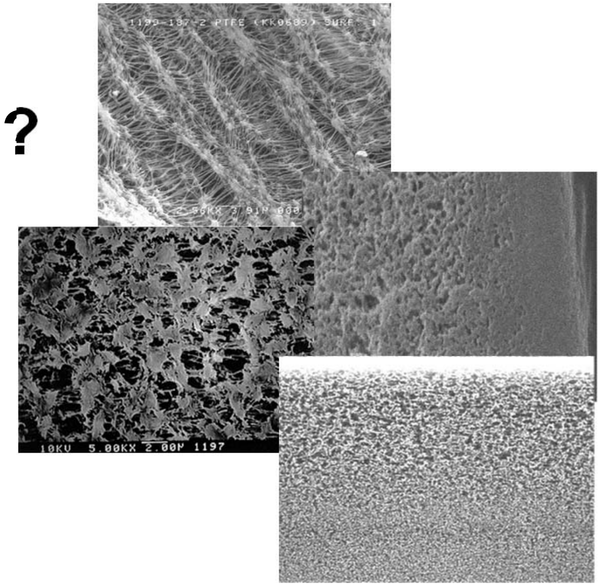
- Particles remain the most critical and dominating risk in Chemicals
- Particle monitoring methods have reached their physical limitation and new techniques are not fully established



How to count

How to differentiate?

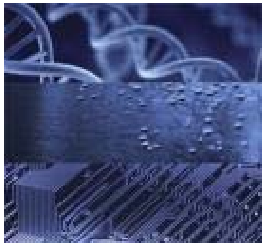
Material influence?



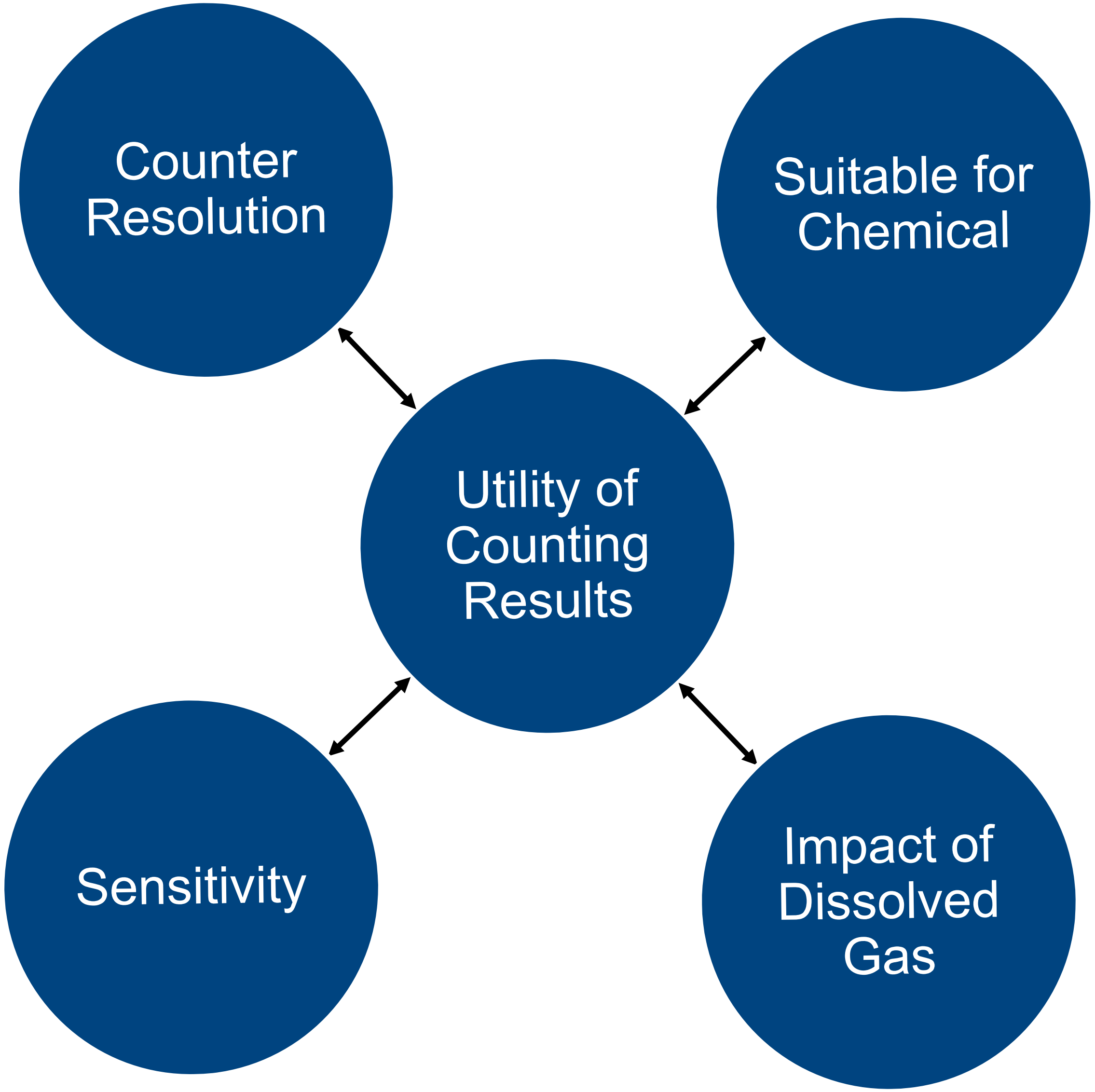
Rationale for Study

- Particle behavior of filters can be affected by the chemical in which they are used
- Evaluation (particle counting) in actual chemical is needed to show actual performance
- Particle sizes of interest for latest technology nodes are much finer than 30 nm
- Many process chemicals are prone to gas bubble formation
- Gas bubbles are counted as particles → **false negative results**
- Technologies that are less sensitive to bubbles could be the solution
- This test is not intended for re-qualifying filters!

Our study explores the use of an alternative counting technology enabling counting in chemicals down to 20 nm (and below) with diminished impact of bubbles



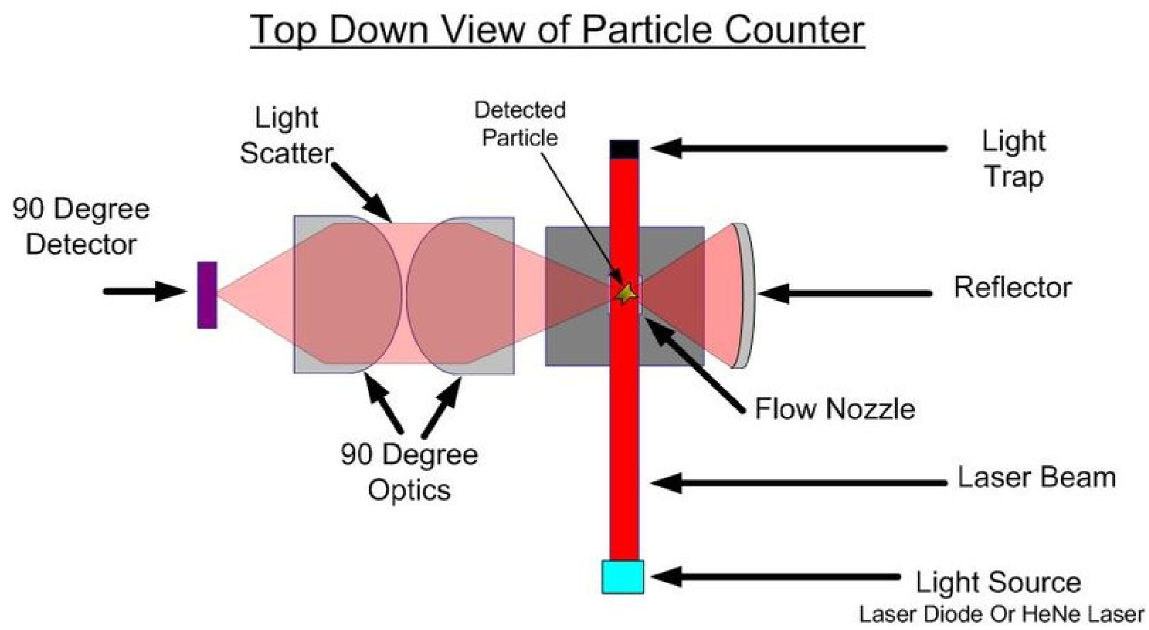
Factors Impacting Particle Counting Operations



Light scattering particle counter 30 nm

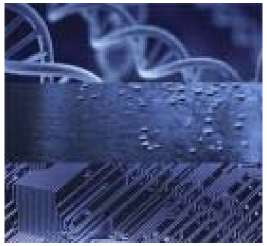
- Present-day light scattering particle counters are limited to a minimum size resolution of 30* nm
- Particle sizes of interest for latest technology nodes are much finer

- Particle passes through the light path (Laser)
- Redirected light is detected by a photo detector.
- The signal is subsequently converted from an analog form into digital form for classification by a microprocessor.



*current available commercial available counter for use in aggressive chemicals, to the best of the authors knowledge

https://en.wikipedia.org/wiki/Particle_counter#/media/File:Particlecounter.jpg



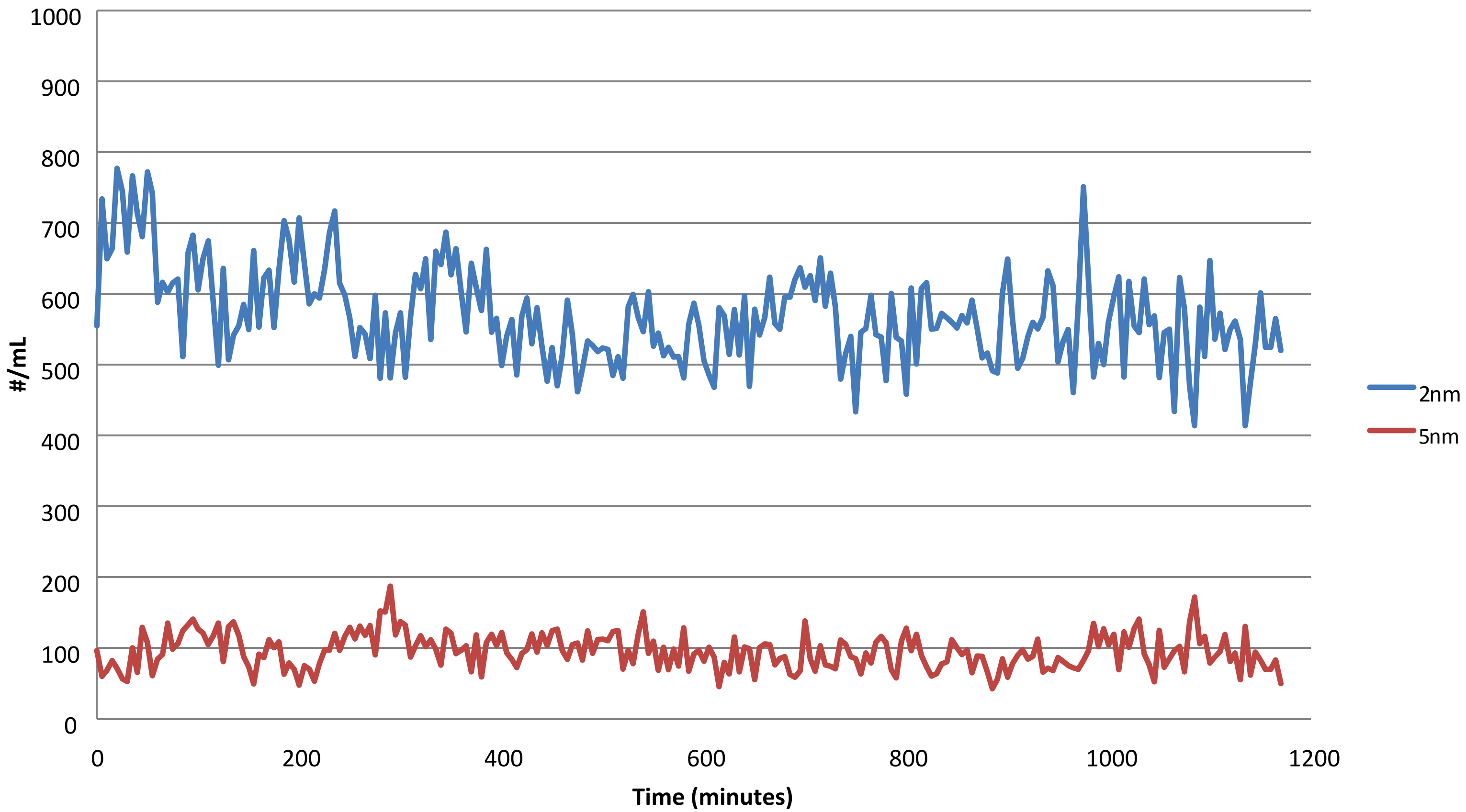
PS 20 Acoustic particle counter ***

- The PS 20 counter is rated to detect particles 20 nm and greater, also in Chemicals**.
- The Particle Scout PS 20 particle counter uses a subtle acoustic interaction, ACIM (Acoustic Coaxing Induced Microcavitation) to facilitate a bubble onto a particle.
- Basically generating a bubble nucleation, acoustic coaxing ACIM transforms the particle into a 10,000 x strong scatterer.
- Liquid particles respond to ACIM fields of adequate intensity by triggering cavitation events.
- There is evidence that bubbles can be discriminated from particles
- Internal data shows the sensitivity of the instrument for even smaller particles

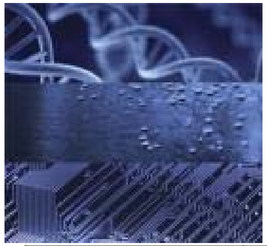
**for extensive chemical compatibility consultation contact Uncopier: Sameer <sameer@uncopiers.com>

***PS 20 is manufactured by Uncopier www.uncopiers.com

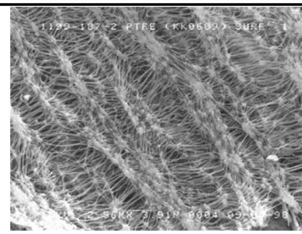
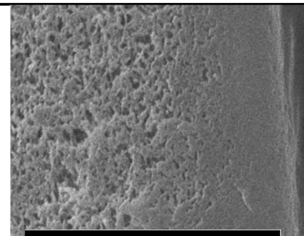
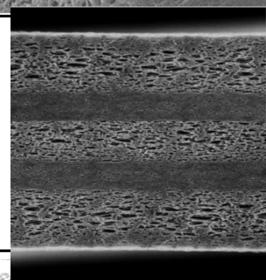
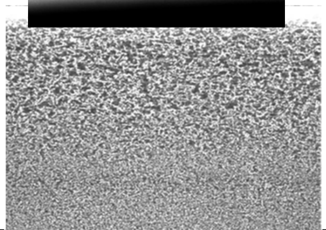
ACIM Sensitivity for 2 and 5 nm Gold particles



- Early testing with an acoustic counter in lithography solvent suggests finer particle detection than previously possible in chemicals
- This was viewed as an indication that this counter ought to be able to allow more discrimination among filters rated 20 nm and finer



Various Filter Media for Chemicals**

Material	Flow-ΔP	General compatibility	Wettability	Min. avail. rating	Used for this Test	Appearance in SEM
HDPE	★★★	★★★★	-phobic	1 nm	5 nm	
Nylon 6,6 asym.	★★★	★★	-philic	5 nm	5 nm	
PTFE	★★★★	★★★★★	-phobic	5 nm	12 nm	
HAPAS*	★★★★★	★★★	-phobic	2 nm	5 nm	

*HAPAS: highly asymmetric polyarylsulfone **for chemical compatibility consult with Pall

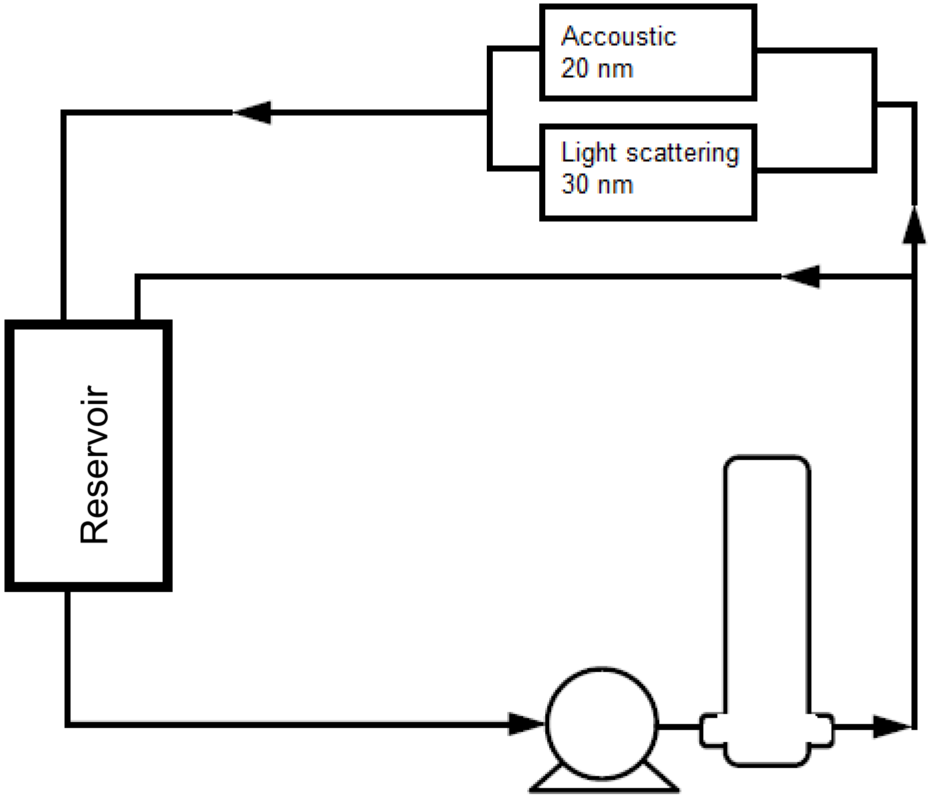
Selection of Candidate Model Chemistry

Tetramethylammonium hydroxide (“TMAH”) selected as model chemical

- Occurs widely for various applications
  - Cleaning chemistries
  - Silicon etchant
  - Photolithography developer
- Also serves as a model for alkaline chemistries in general
- Moderate concentration of 2.5%
- Made up from concentrated (25 %) high grade bottle

Test Set-up and Operation

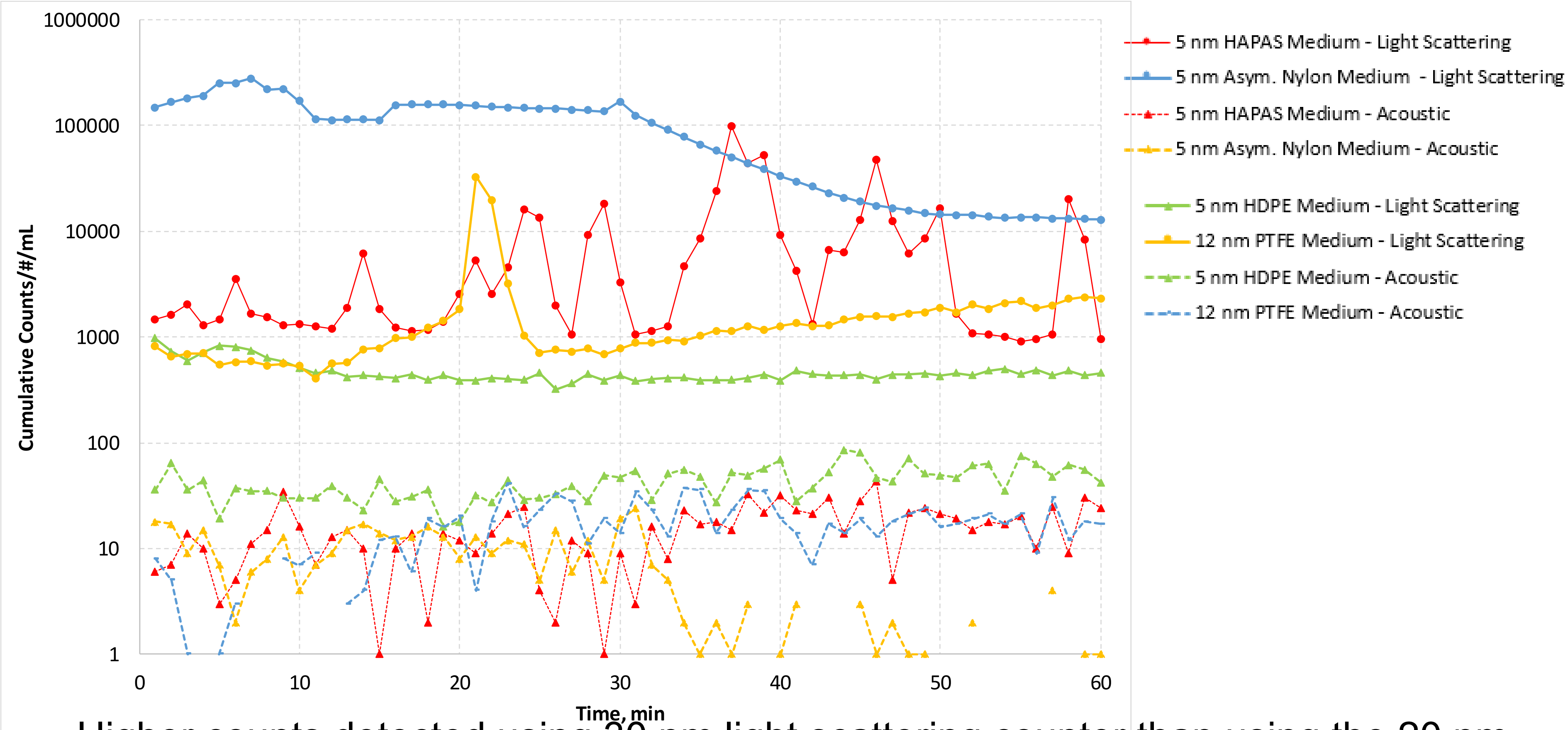
- Recirculative test system set-up with reservoir, pump, insertion point for filter, and side stream to particle counters
  - Effluent sidestream split to two counters
    - Acoustic 20 nm res.
    - Light scattering, 30 nm res.
- No additional challenge particles introduced



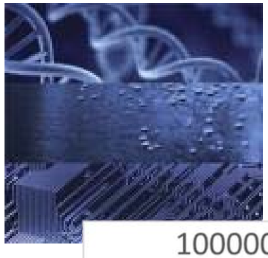
Test Set-up and Operation

- **Low backpressure used**
  - Low backpressure is normally the wrong way to install online counters
- **Low backpressure enhances formation of air bubbles**
  - Air bubbles might impact the light scattering method vs. counting via acoustic signal should show less interference
  - A high amount of bubbles is intended to show differences in the particle counting of both measurement systems
  - Impact of surface energy of filters shall be highlighted
- **Mimic actual operational condition often occurring**
  - Filter capsules used for all tests
  - Flow rates selected based on size, typically in range 0.3 – 1 LPM

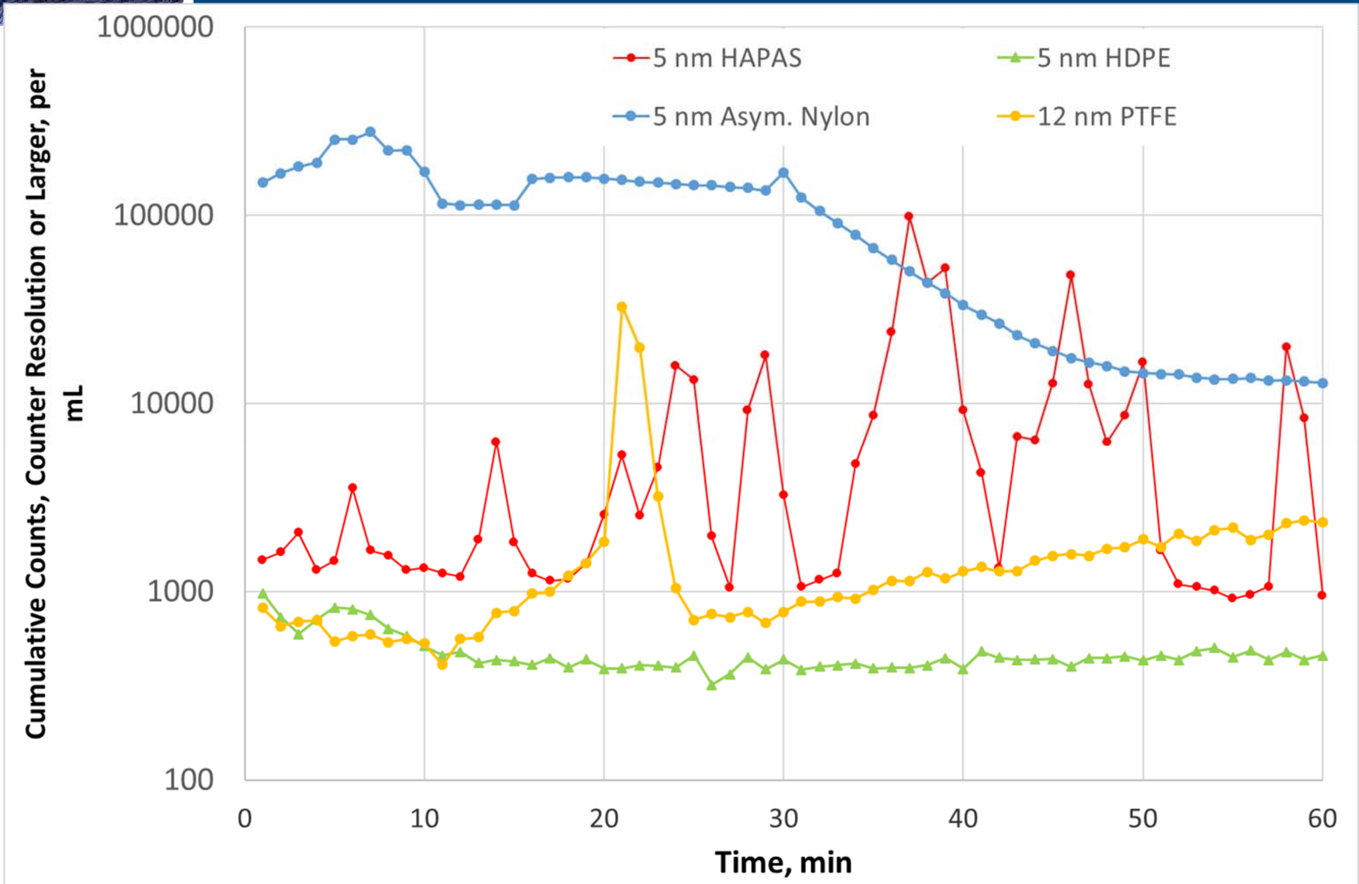
Relative Behavior Found for Various Filters Evaluated with Either Counter Type in TMAH



- Higher counts detected using 30 nm light scattering counter than using the 20 nm acoustic counter
- High ≥30 nm counts suggest *extreme* presence of air bubbles
- Contrary to expected *lower* counts at larger size

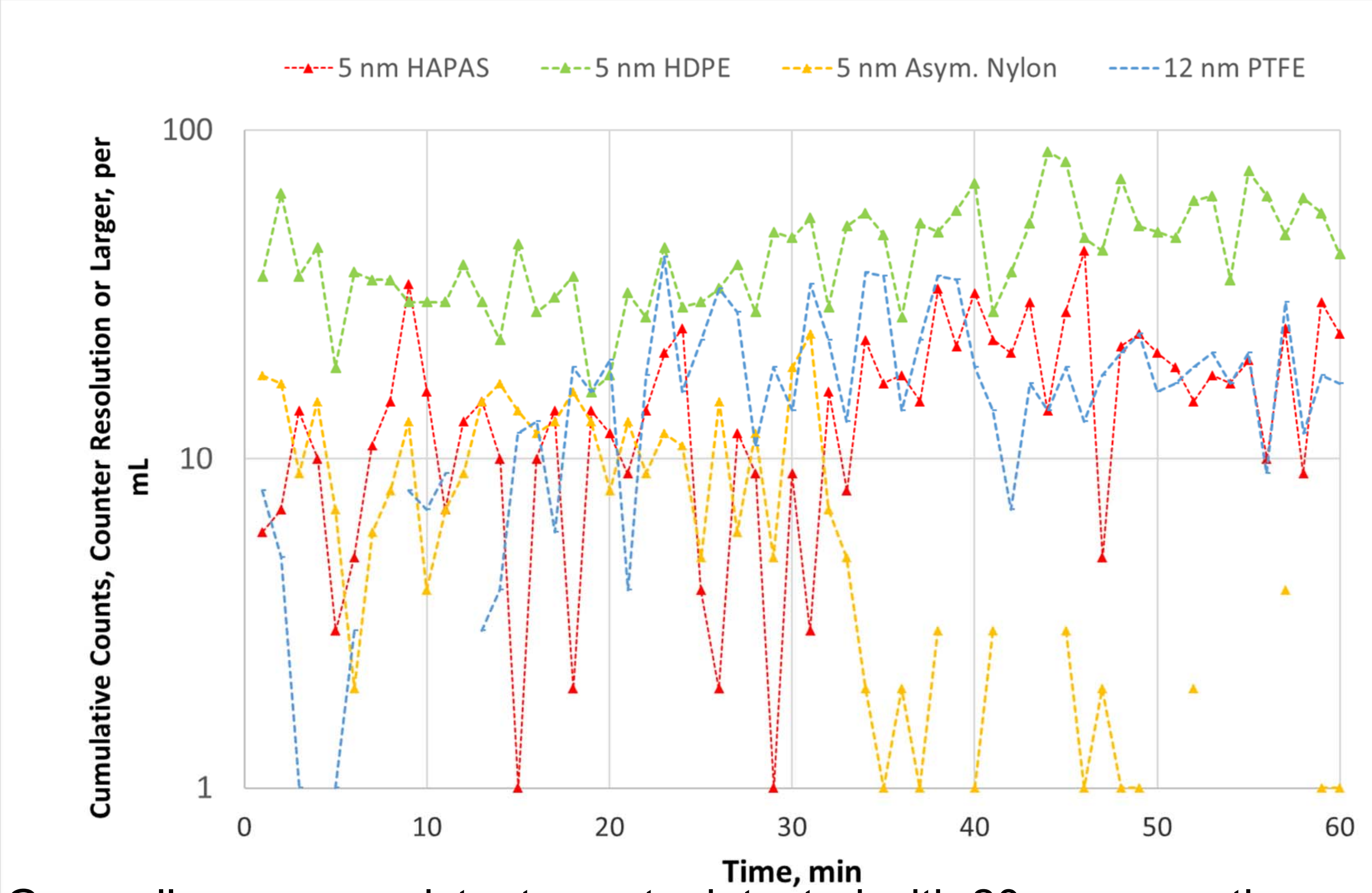


Counting with light scattering



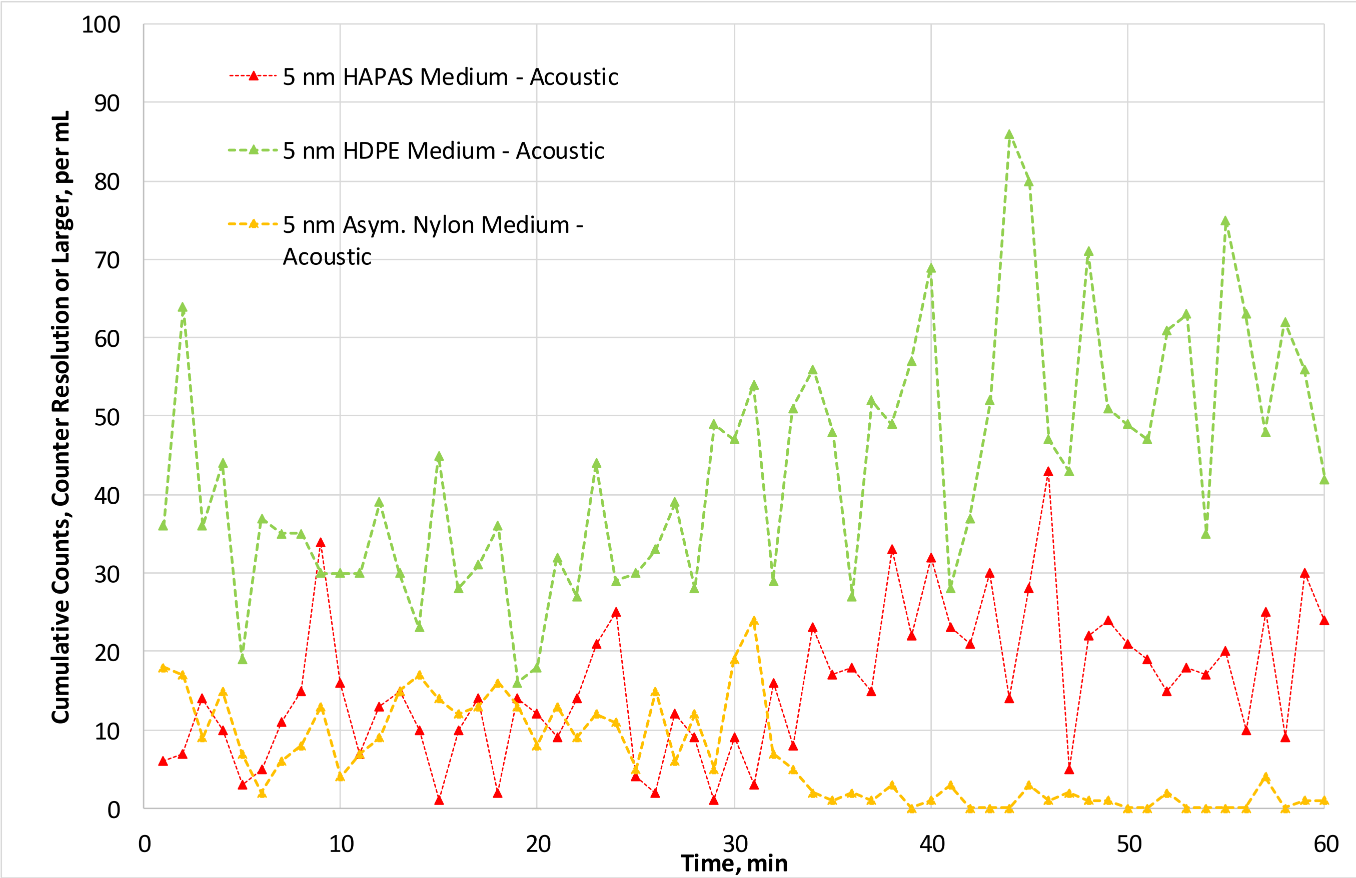
- Higher counts ≥ 1000 detected using 30 nm light scattering counter
- Wide variation among samples
  - 5 nm HDPE presented stable counts over 60 min
  - 5 nm HAPAS presented unstable counts “1000 counts vs. 100000 counts” over 60 min

Counting with acoustic detection



- Generally more consistent counts detected with 20 nm acoustic counter
  - Differences detected among samples but in a narrower range
  - More in line with expectations for filters rated at 12 nm or finer
- Obviously no influence of air bubbles

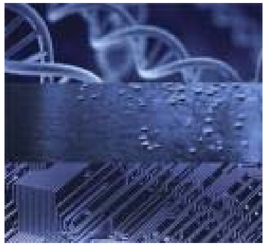
Acoustic Counting Comparison Including Filter with Asymmetric Nylon Medium



- Nylon and HAPAS filter actually shows the lowest ultimate count level
  - Nylon medium filter exhibited *highest* counts with light scattering counter
- Focus shifted to comparison via acoustic counting

Conclusions, Path Forward

- **Testing was conducted in 2.5% TMAH as a suitable model chemical, with four filter types evaluated**
- **Methodology was capable of showing differences among filters with different media**
  - **Obviously the media type has an influence of air bubble formation**
- **Further study using different WEC-relevant chemicals, different filters, and alternate test conditions appears warranted to validate the counter over a broader application space**



Conclusions, Path Forward ct.

- **An acoustic particle counting methodology allows particle counting in chemical at smaller minimum size than for currently available light scattering counters**
- **Acoustic counting allows more realistic counting results even in the case of no/low back pressure**
- **Results very different from those obtained via light scattering counting, likely due to impact of desorbed gas**
- **Acoustic counting has demonstrated the ability of particle detection at 10, 5 and 2 nm particles sizes in UPW**

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