The Effect of Particle Concentration and Face Velocity on the Removal of sub 100 nm Particles from Ultrapure Water

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Introduction

- The 2012 ITRS Roadmap has defined critical particle sizes of 20 nm in 2013 and 10 nm in 2019.
- Particles in UPW with these dimensions and larger can deposit onto wafer surfaces during microcircuit fabrication thereby reducing the yield and performance of the finished devices.
- Concentrations of these small particles in UPW are very difficult to measure but are believed to be ≤ 1E3/mL ≥ 30 nm.
- Microfiltration (MF) and ultrafiltration (UF) devices with ratings below 50 nm are utilized to control particle concentrations.

Introduction (cont'd)

- The particle retention capability of these devices is often measured with challenge concentrations much higher than those in UPW.

- Also, filters are often tested at face velocities other than those used in UPW systems.

- This paper shows a number of examples which indicate that:
  - Particle challenge concentration and face velocity during testing can have a significant effect on particle retention.
  - Filter particle retention measured under conditions other than expected operating conditions can be misleading.
  - Filter test conditions should mimic actual use conditions as closely as possible.

Outline

- Experimental conditions
  - Variables investigated
  - Filter types tested
  - Test methods used
- Effect of variables on performance
  - Filter loading
  - Challenge concentration
  - Face velocity
- Summary
- Implications for filter testing

Operating variables examined

- Particle loading
  - The number of particles removed by the filter per unit of filter area.
  - Will be reported in terms of monolayers of coverage.
    - Coverage is 1 monolayer at the time when the cross-sectional area of the cumulative number of particles in the filter challenge is equal to the filter surface area.
- Challenge concentration
  - The particle concentration at the filter inlet.
- Face velocity
  - Flow rate per unit area of filter.
  - Will be reported as cm/min.
  - The face velocity of a 10” cartridge with 10 ft^2 of membrane area operated at 10 Liters/minute is 1.1 cm/min.

Filter retention definitions

- Retention
  - Retention (%) = 100 $(1 - C_F/C_C)$
    - C_F = Filtrate concentration
    - C_C = Challenge concentration
- Log reduction Value
  - $LRV = \log_{10}(C_C/C_F)$

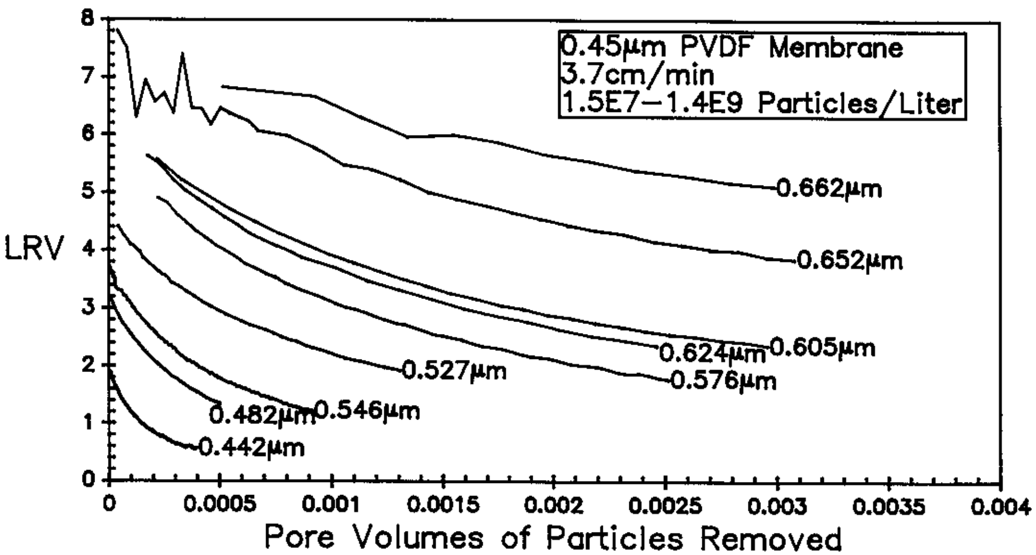
Retention (%)	LRV
99.9	3.00
99	2.00
90	1.00
75	0.60
50	0.30
25	0.12
10	0.05

Particle loading

- Degradation of filter retention performance over time has been cited multiple times and attributed to selective clogging of the smaller pores in the filter.

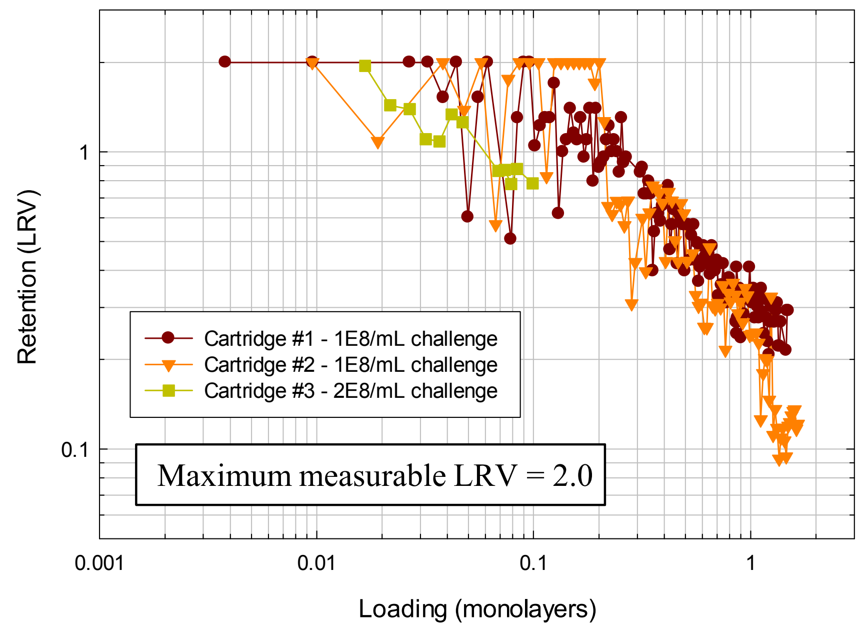
Published examples of retention degradation with loading

Removal of PSL particles
by a PVDF Membrane



From Grant and Liu, *Part. Part. Syst. Chara.*, 8:142-150 (1991)

Removal of 12 nm silica particles
by a high retention membrane



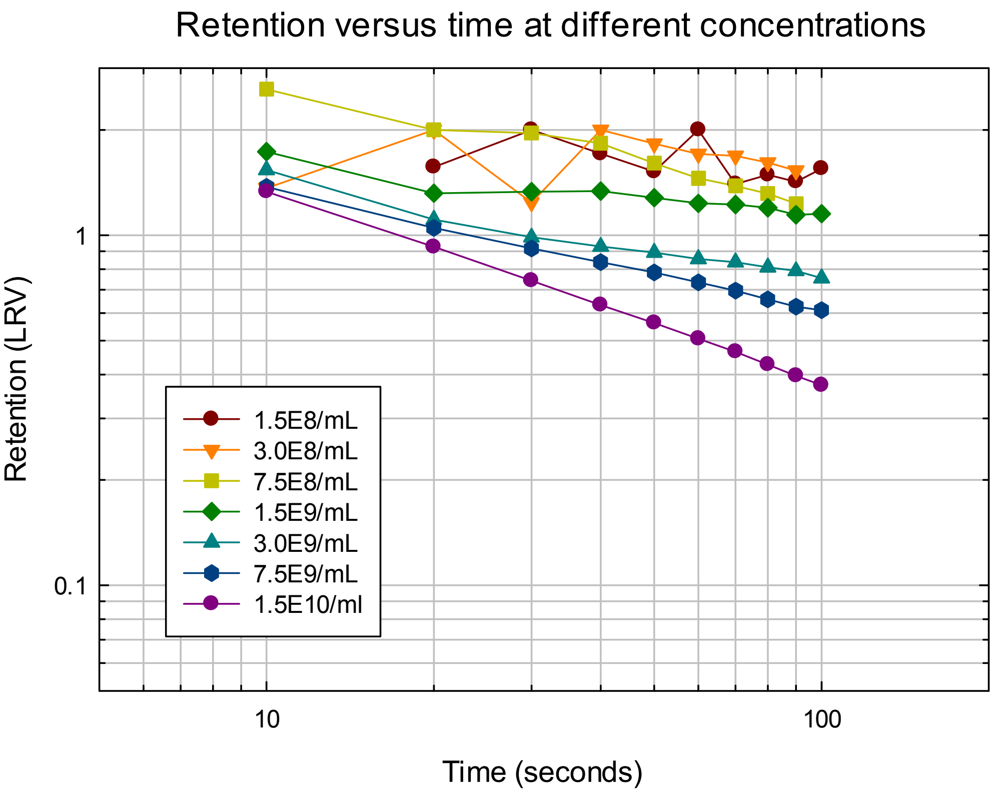
From Grant, Chilcote and Beuscher, UPW Micro 2012.

Particle loading

- Degradation of filter retention performance over time has been cited multiple times and attributed to selective clogging of the smaller pores in the filter.
- Both particle concentration and face velocity affect the rate of loading with time.
  - Higher concentrations and faster face velocities result in faster loading rates.

Retention versus time and loading

(50nm PSL; 1.2 cm/min)



Retention versus time and loading

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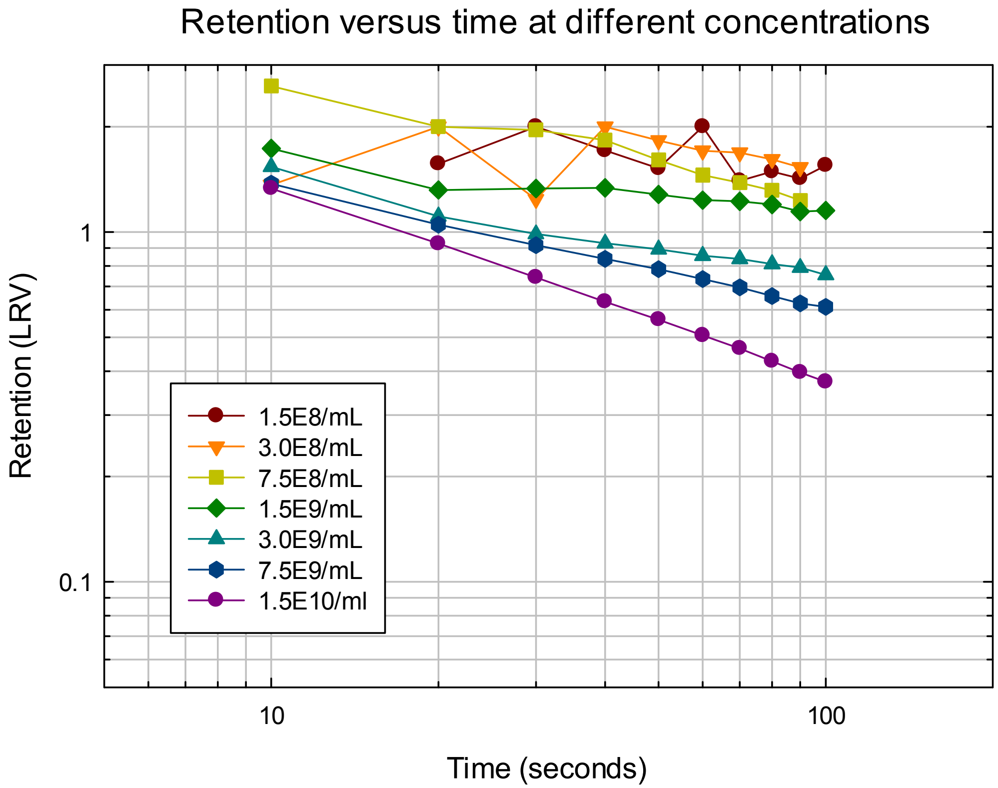
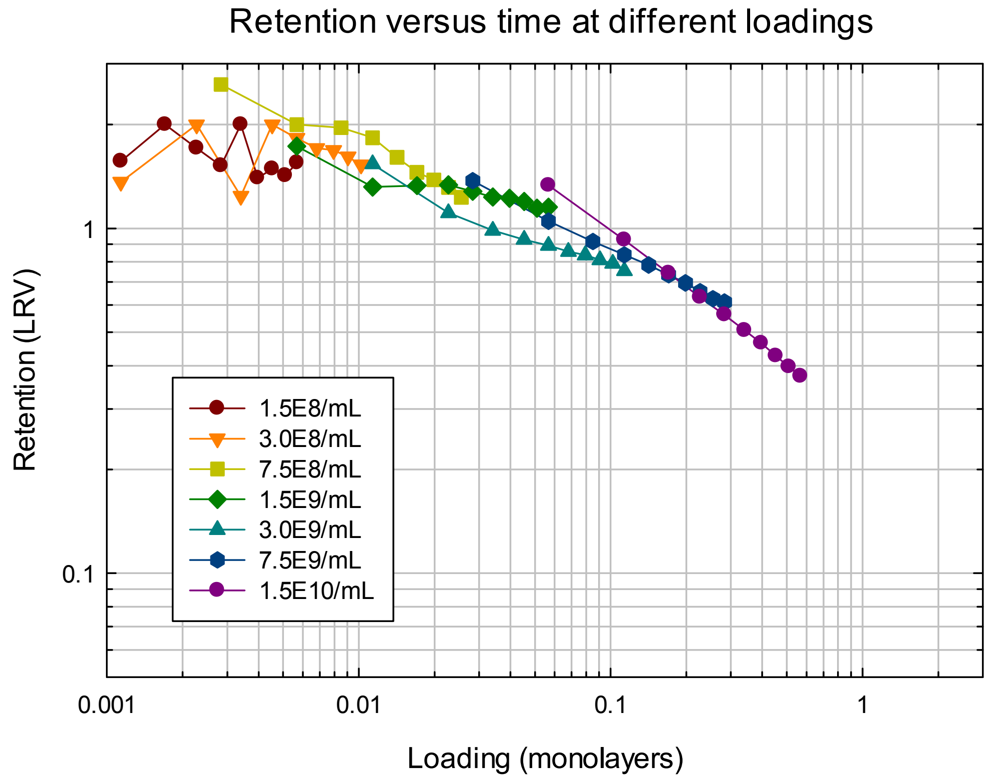




Particle loading

- Degradation of filter retention performance over time has been cited multiple times and attributed to selective clogging of the smaller pores in the filter.
- Both particle concentration and face velocity effect the rate of loading with time.
  - Higher concentrations and faster face velocities result in faster loading rates.
- Filter retention in this presentation will be presented in terms of loading rather than time.

Particle loading

- If UPW contains 1E3 particles/mL that are 30nm in diameter and the 10" filter is operated at 1 cm/min, the loading rate will be 0.004 monolayers/year.

Filters tested

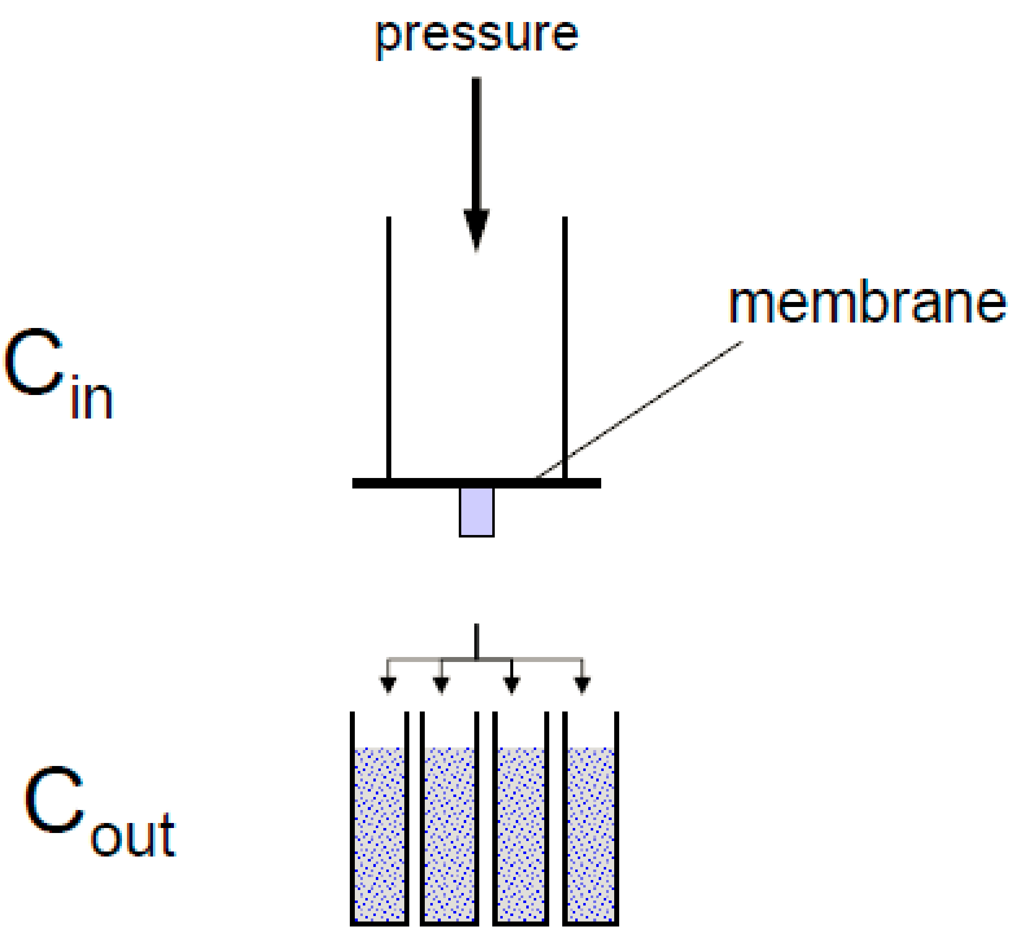
- Filters from a number of manufacturers with retention ratings between 10nm and 50nm were tested.
  - Rating methods are manufacturer dependent.
  - Ratings for each filter tested will not be disclosed.
- Filter membrane materials of construction
  - Polyethylene
  - Polysulfone
  - Fluoropolymer
- All are commercially available
- Most were tested as cartridges; some as flat sheets.

Particle types tested

- Polystyrene latex (PSL)
  - 50 and 70 nm
  - With and without surfactant added.
- Colloidal silica
  - 3 different sizes.
  - Median diameters of 12, 18 and 28 nm.

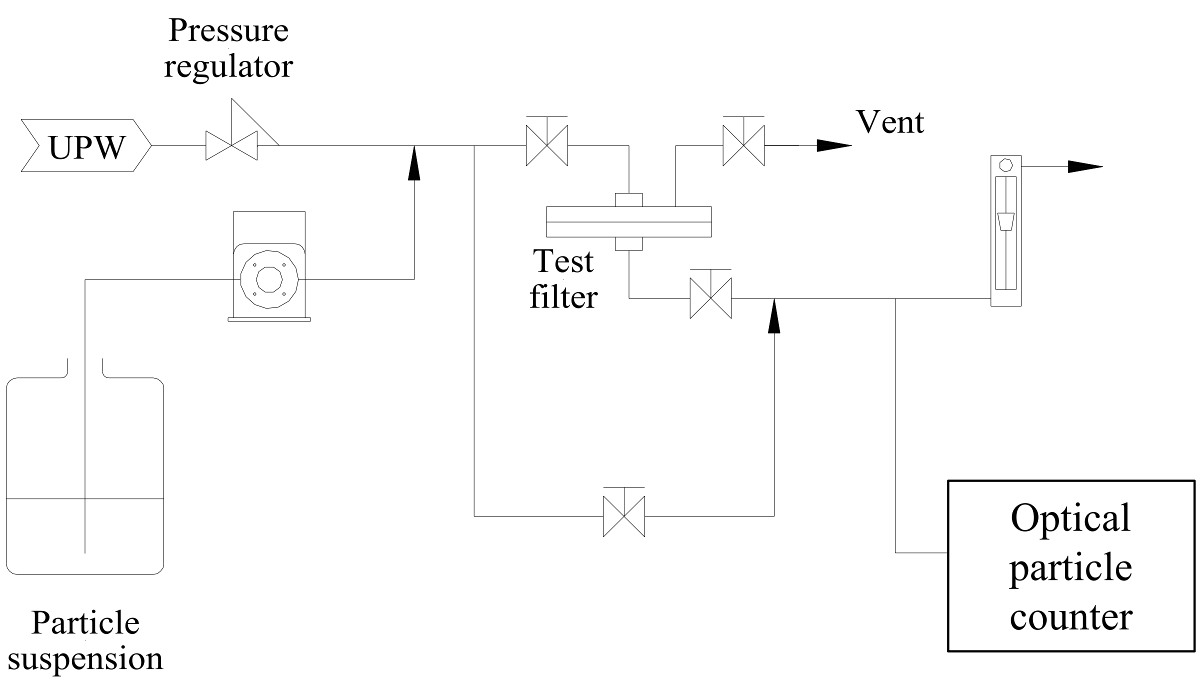
Test Methods

Disc filter test method 1 – Fluorescent PSL beads



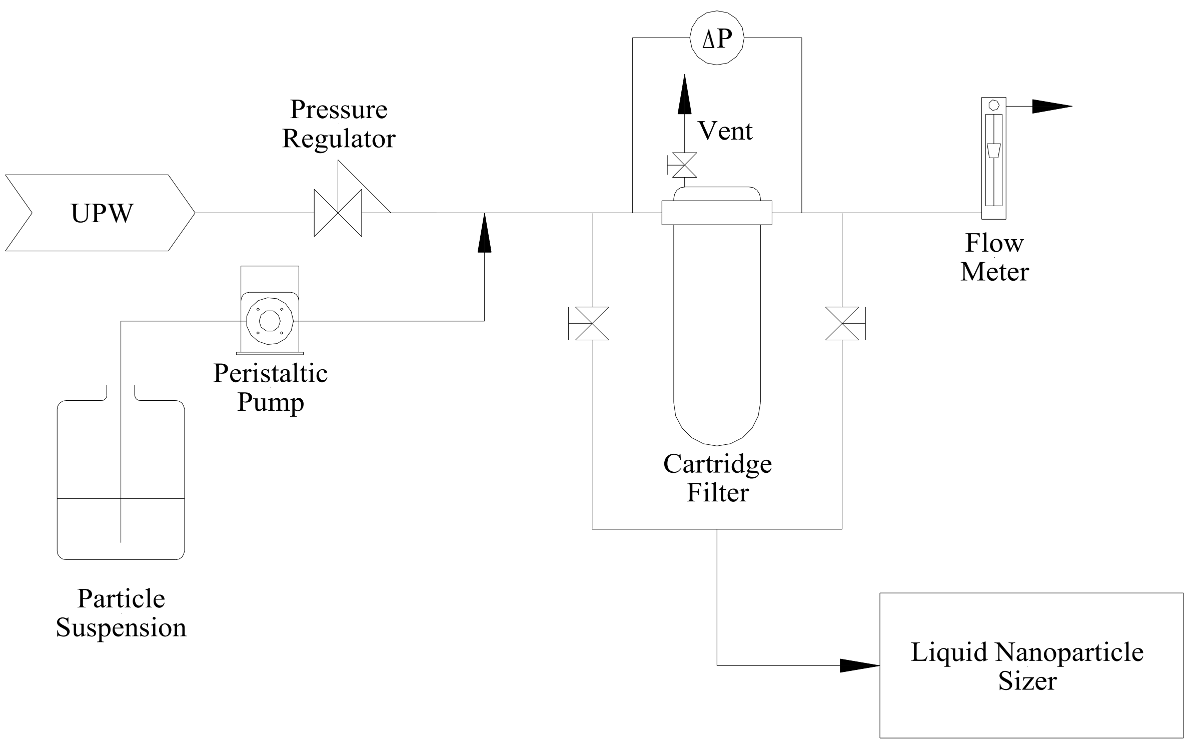
- 1 ppm suspension of 50nm polystyrene latex beads.
- 250 mL challenge suspension.
- Filter into ten 25 mL sample vials at a constant face velocity.
- Concentration measurement by spectrophotometry.

Disc test method 2 – PSL beads



- The filter was flushed until the filtrate approached the system background concentration (<1/mL > 50 nm).
- The filter was challenged with a constant challenge concentration and face velocity.
- Filter inlet and outlet concentrations were measured using an M50 optical particle counter (Particle Measuring Systems).

Cartridge test method



- The cartridge was flushed until the filtrate approached the system background concentration (~10^6/mL > 10 nm).
- The filter was challenged with a constant challenge concentration and face velocity.
- Filter inlet and outlet concentrations were measured using a liquid nanoparticle sizer [1].

[1]. Grant and Beuscher , Ultrapure Water Journal, 26(11):34-40.

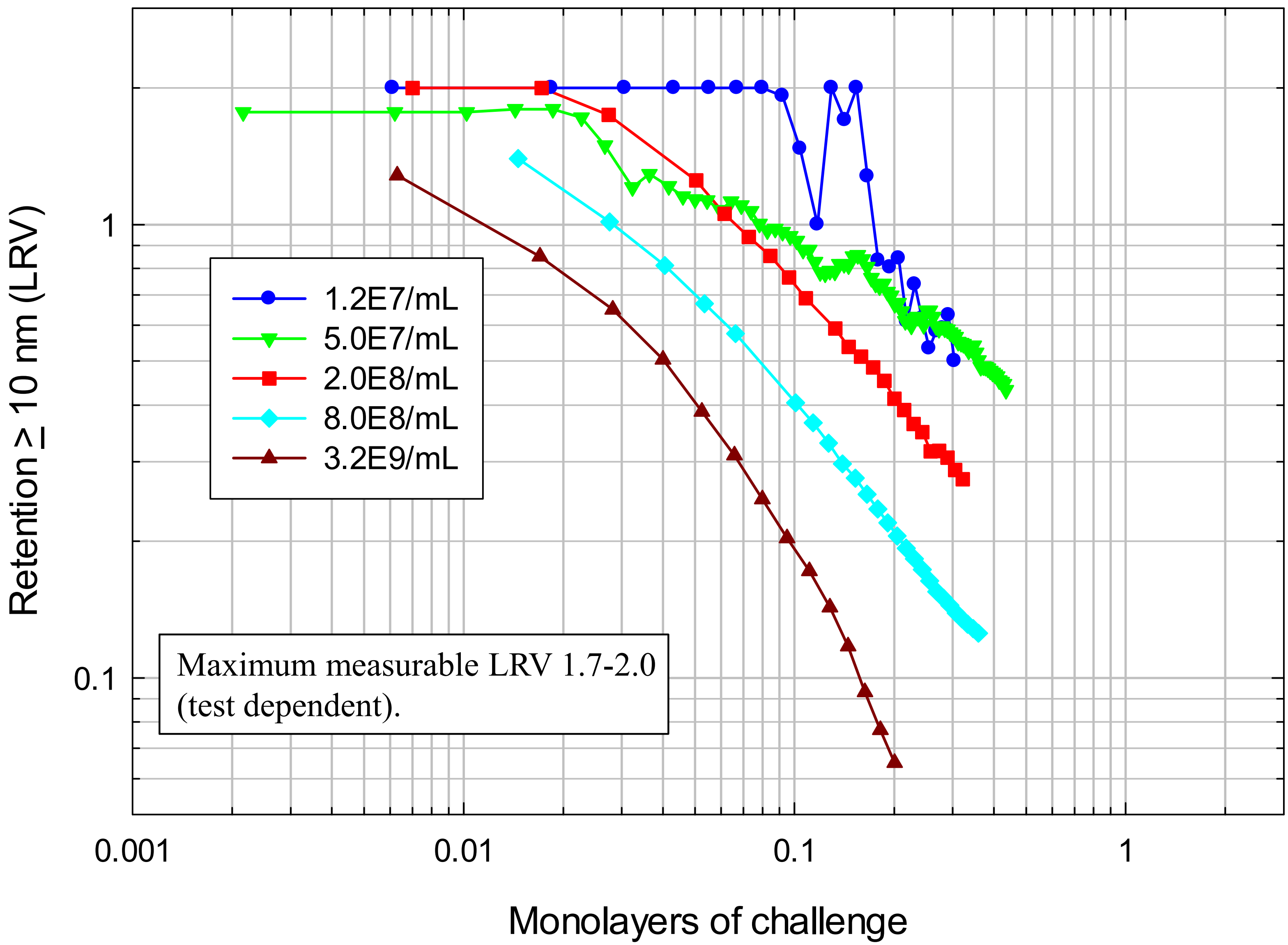
Filter “naming”

- 5 types of filters were evaluated in the “Effect of challenge concentration” tests
  - Labeled A through E
- 3 types of filters were evaluated in the “Effect of face velocity” tests
  - Labeled A through C
- Labels assigned in the two series of tests were independent of each other.

The effect of challenge concentration on retention

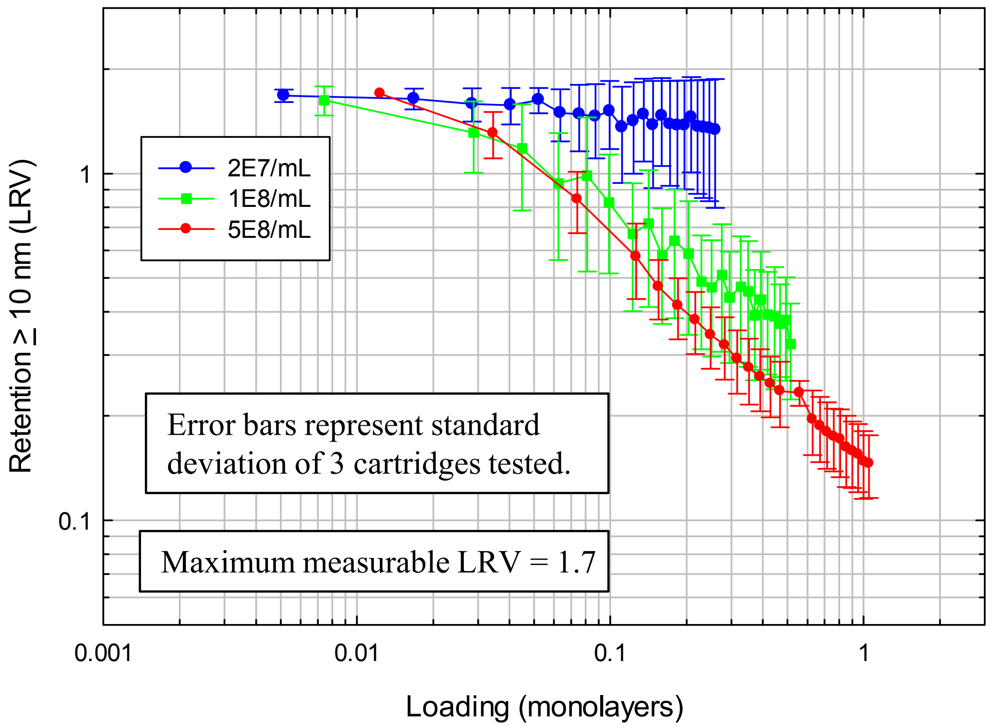
The effect of concentration on retention

Example 1: Filter A, 12nm silica, 0.95 cm/min

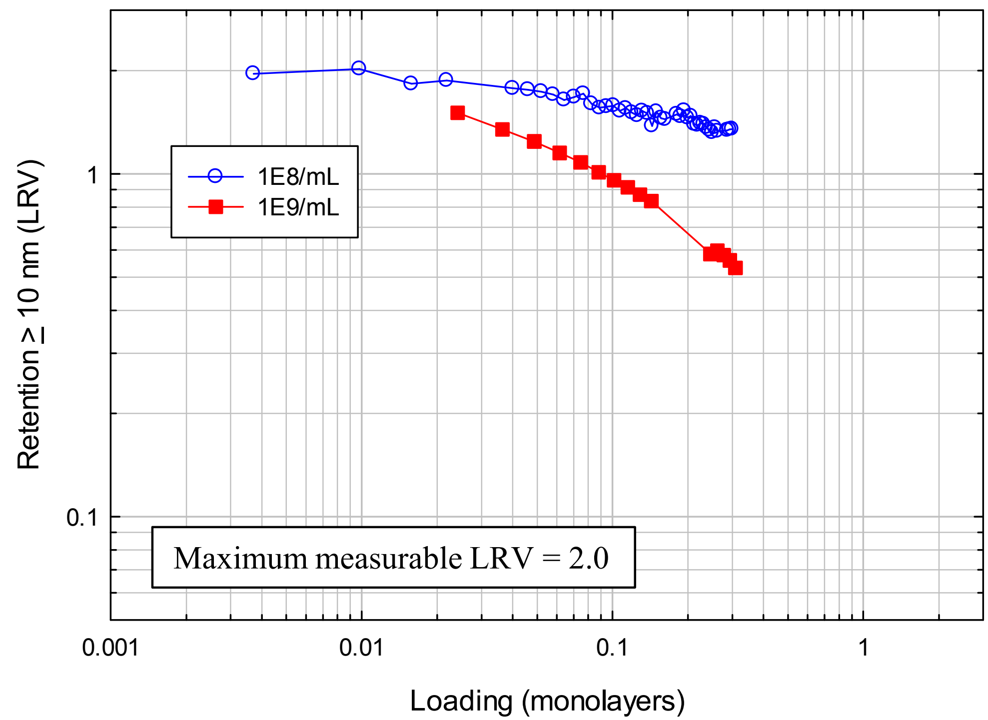


The effect of concentration on retention

Example 2: Filter B; 10-30nm silica particles, 0.95 cm/min



Example 3: Filter C; 30nm silica particles; 1.5 cm/min



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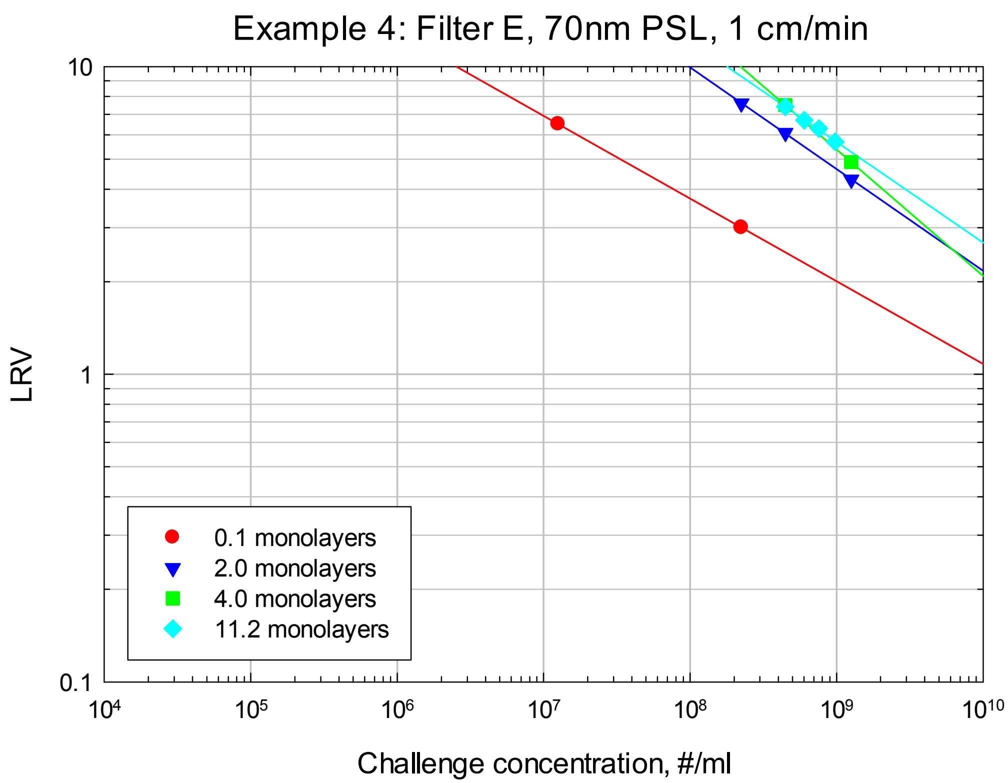
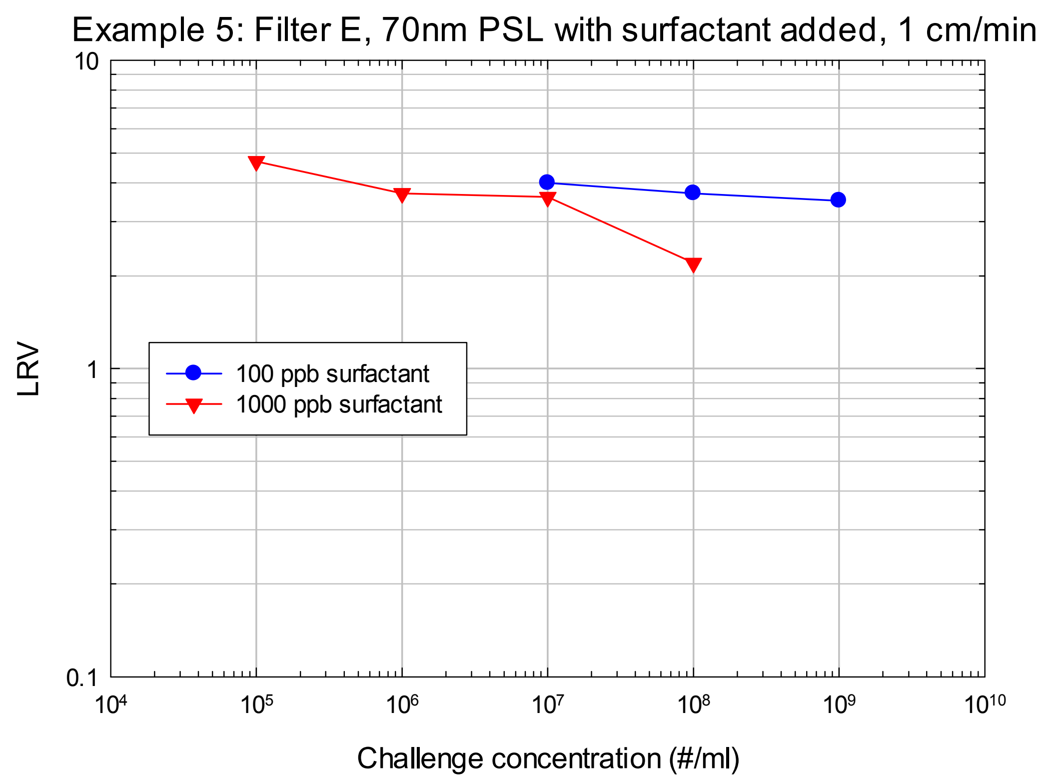
The effect of concentration on retention

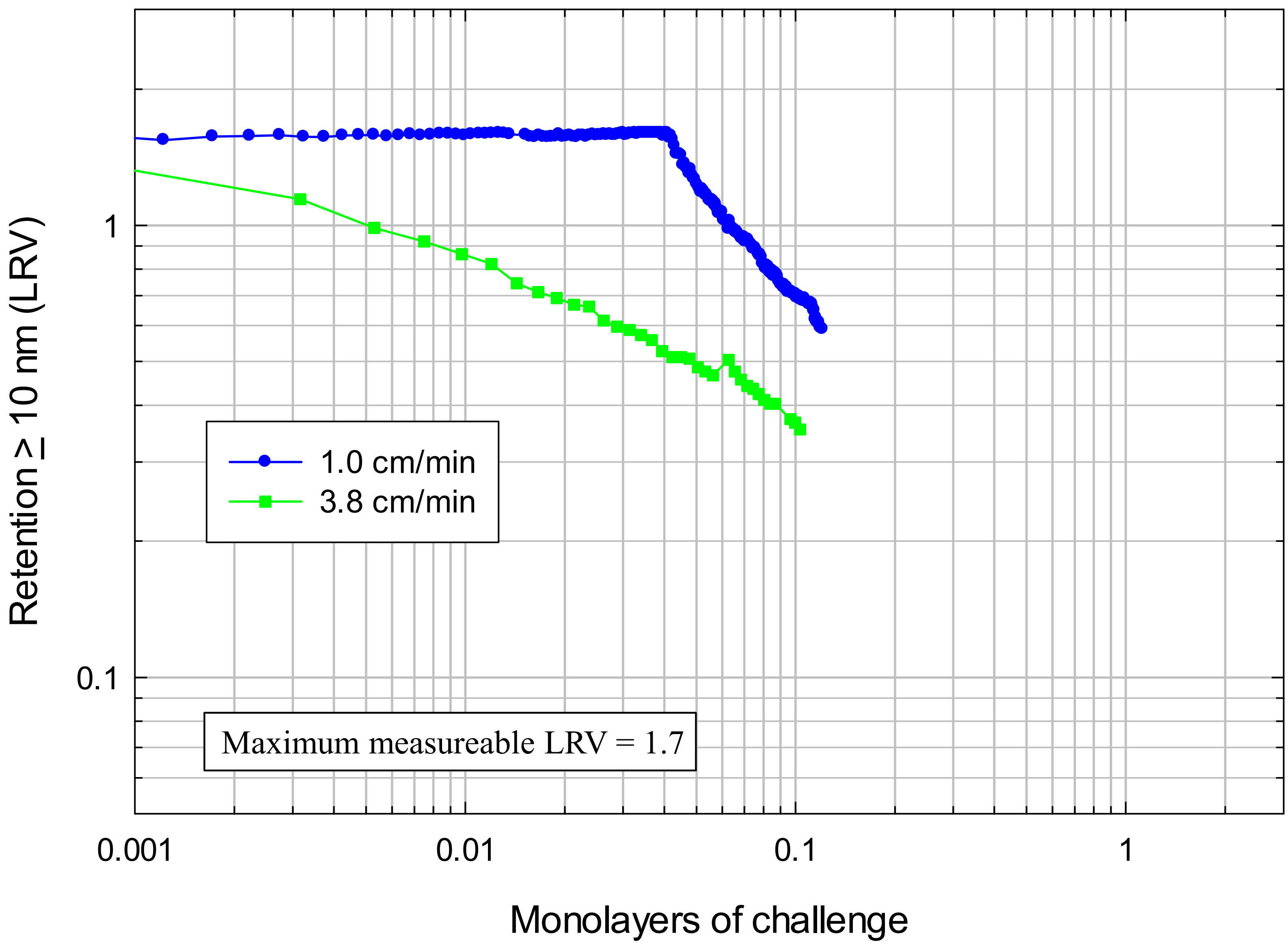




The effect of velocity on retention

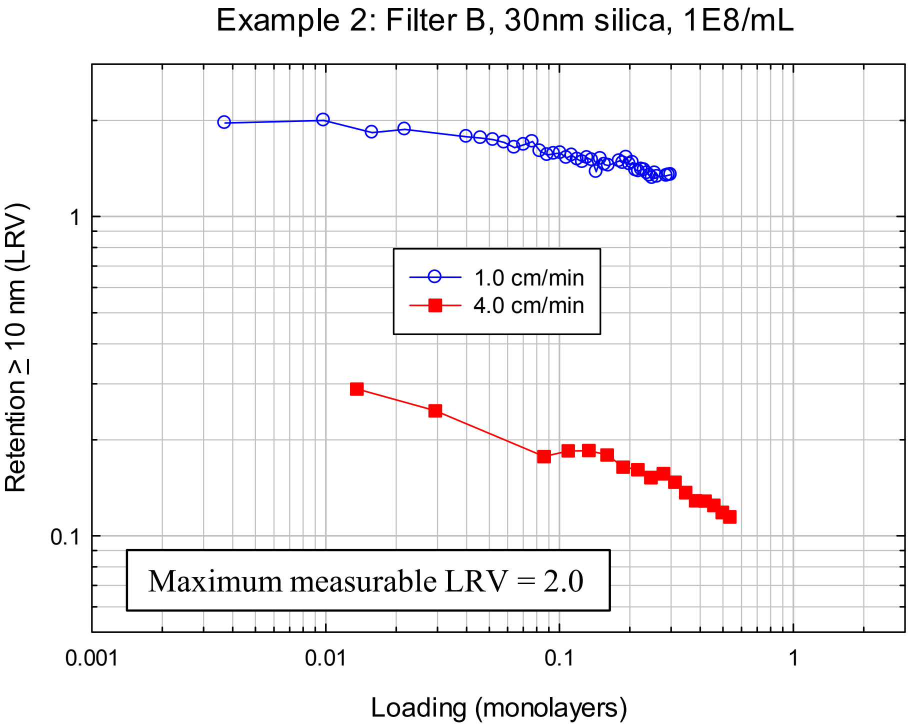
The effect of face velocity on retention

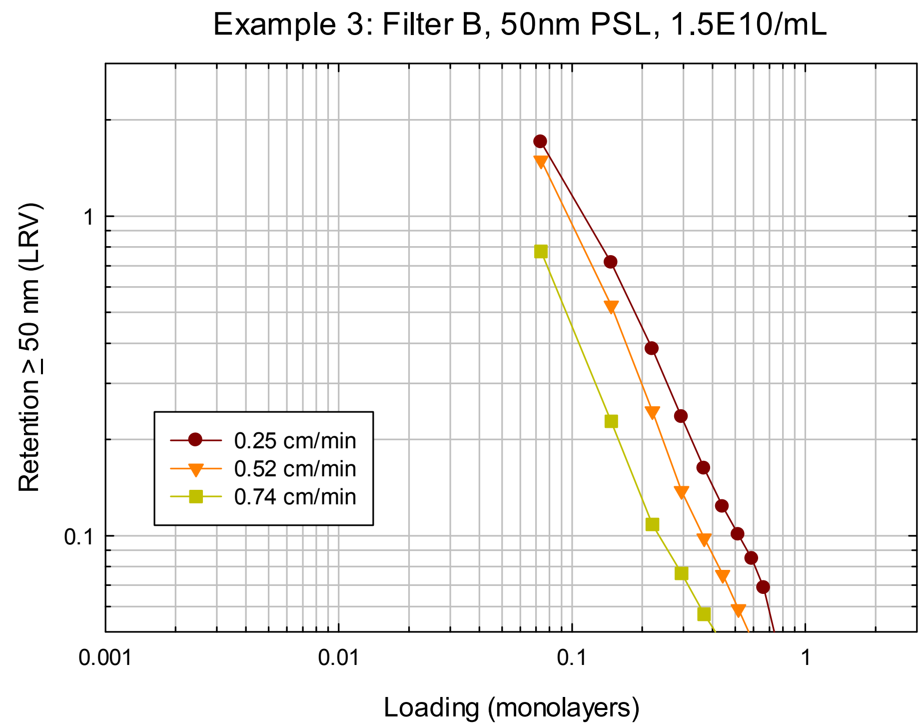
Example 1: Filter A, 12nm silica, 1E8/mL

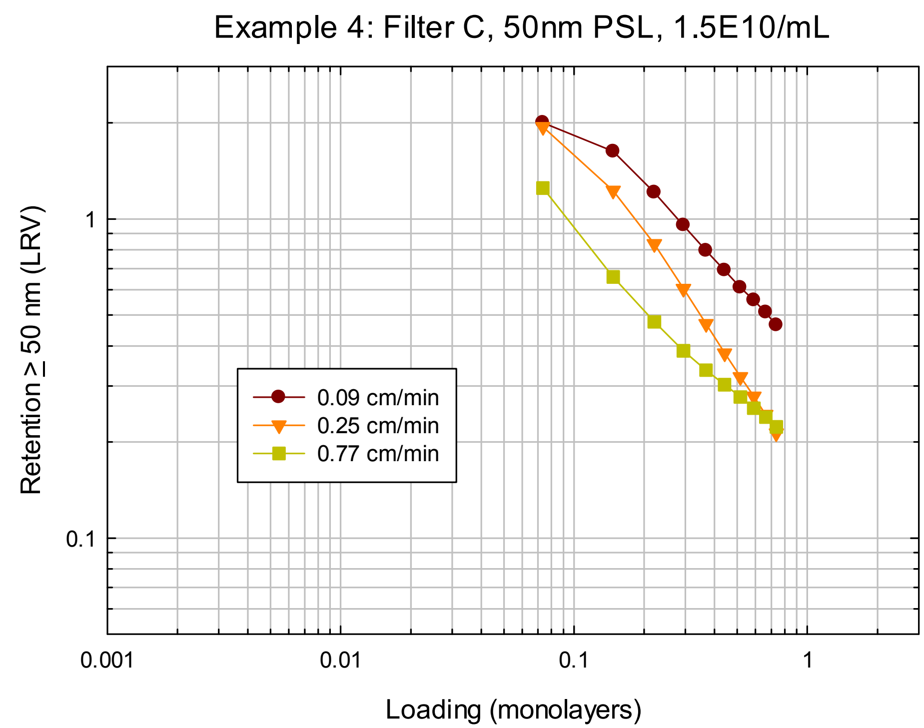


Grant, Beuscher, Van Schooneveld, UPW Micro 2013 (CTA Publication 122)

The effect of face velocity on retention





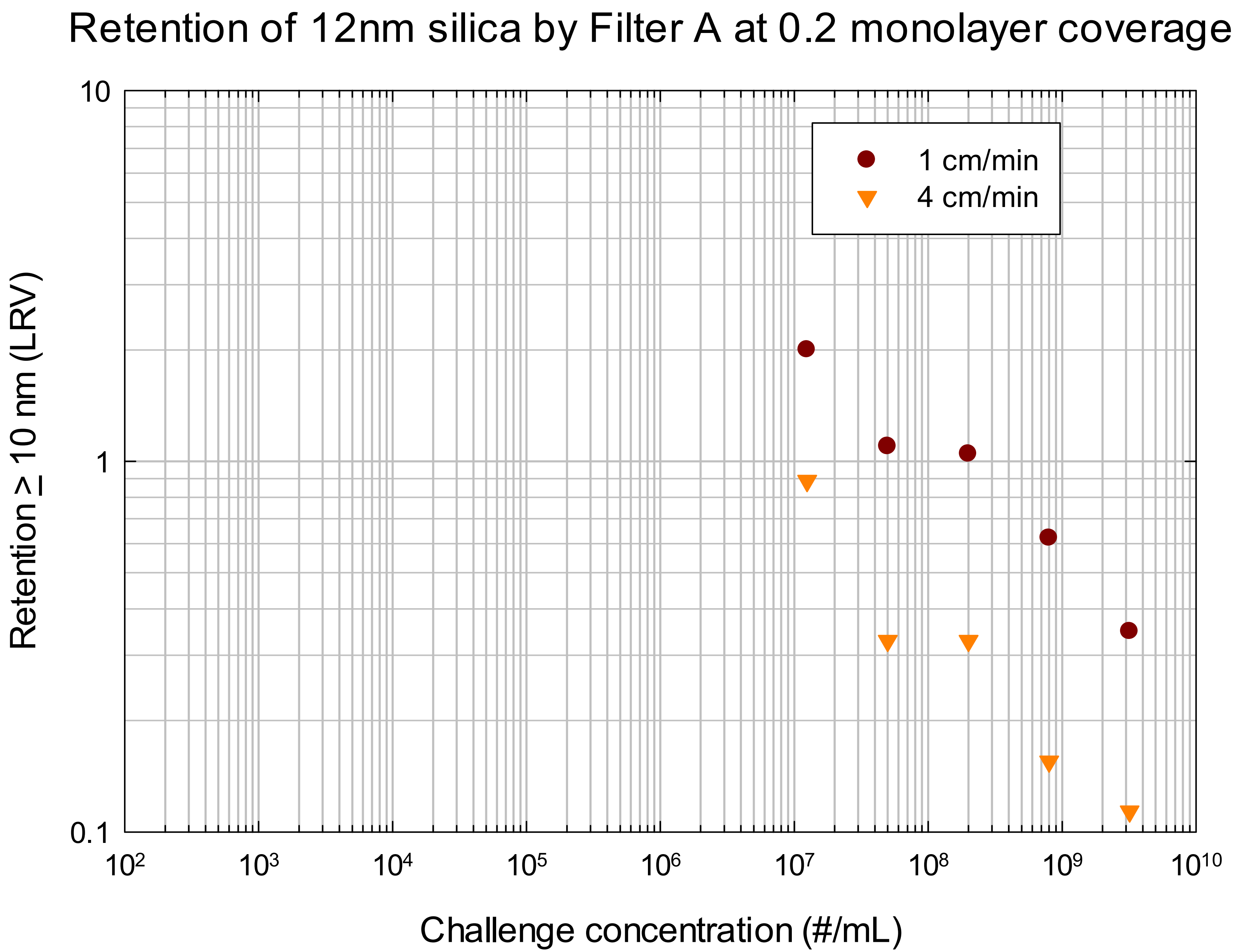


Summary

- Multiple types of filters were tested to determine the effects of challenge concentration and face velocity on their particle retention.

- Many observations were made which indicated that retention:
  - Decreased with loading
  - Decreased with challenge concentration
  - Decreased with face velocity.

What does this mean for UPW filter testing?



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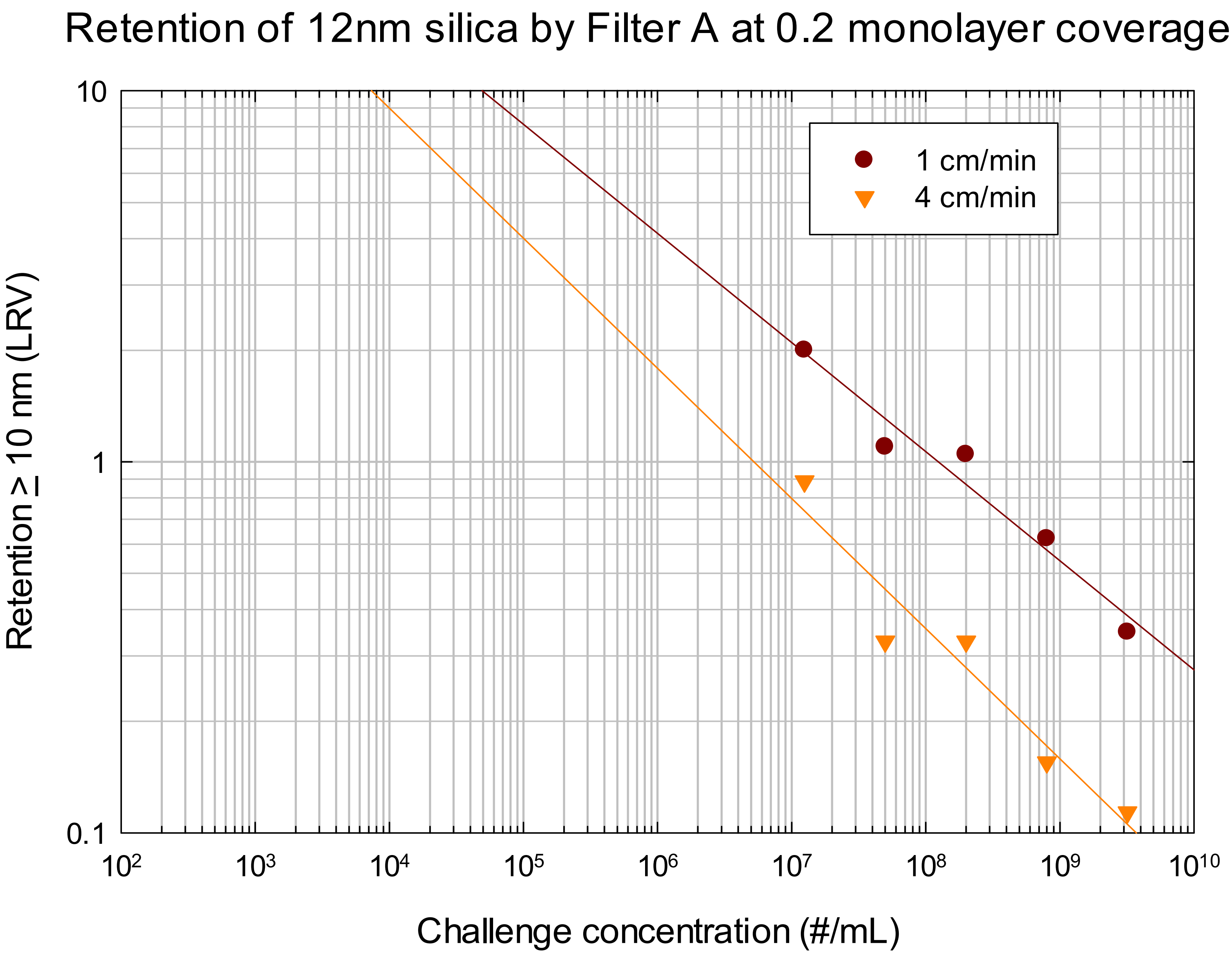

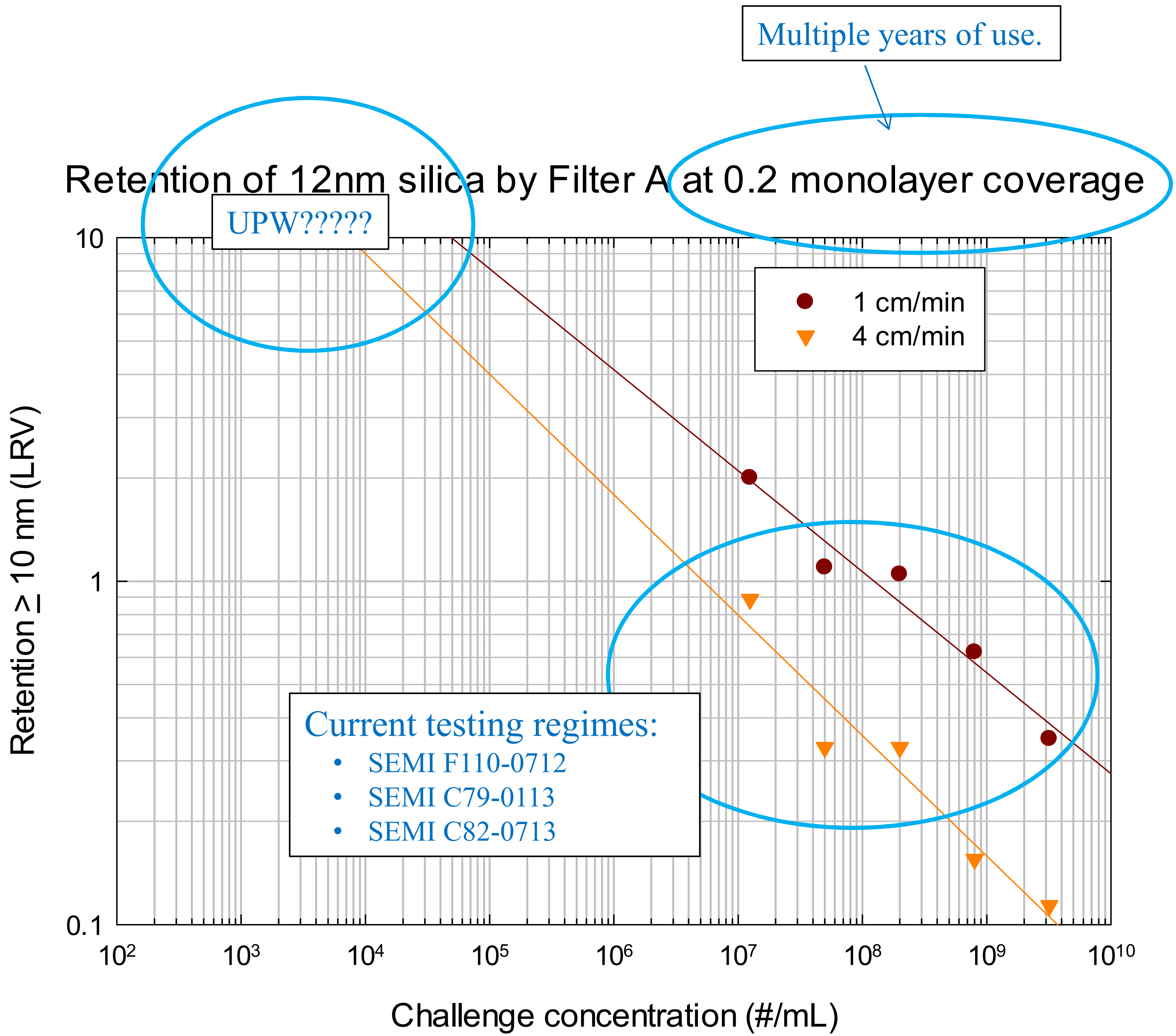

Multiple years of use.
Retention of 12nm silica by Filter A at 0.2 monolayer coverage
UPW?????
1 cm/min
4 cm/min
Current testing regimes:
• SEMI F110-0712
• SEMI C79-0113
• SEMI C82-0713
Retention ≥ 10 nm (LRV)
10
1
0.1
10^2
10^3
10^4
10^5
10^6
10^7
10^8
10^9
10^10
Challenge concentration (#/mL)

Summary

- Multiple types of filters were tested to determine the effects of challenge concentration and face velocity on their particle retention performance.

- Many observations were made which indicated that retention:
  - Decreased with loading
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  - Decreased with face velocity.

- Filter retention testing should mimic actual use conditions as closely as possible.