ITRS, SEMI and ASTM Guidelines for Semiconductor Ultrapure Water (UPW) Production and the Consequences for UPW Particle Metrology

David Blackford, Ph.D. Fluid Measurement Technologies, Inc.
St. Paul, Minnesota

University of Arizona TeleSeminar
May 3,2012

The **International Technology Roadmap for Semiconductors (ITRS)** is a set of documents produced by a group of semiconductor industry experts. These experts are representative of the sponsoring organizations which include the Semiconductor Industry Associations of the US, Europe, Japan, South Korea and Taiwan.

Semiconductor Equipment and Materials International (**SEMI**) is a trade organization of manufactures of equipment and materials used in the fabrication of semiconductor devices. Among other activities, SEMI acts as a clearinghouse for the generation of standards and guidelines.

American Society for Testing and Materials (ASTM), is an international standards organization that develops and publishes voluntary consensus technical standards for a wide range of materials, products, systems, and services for all industries.

ITRS Requirements for 2011 and Beyond

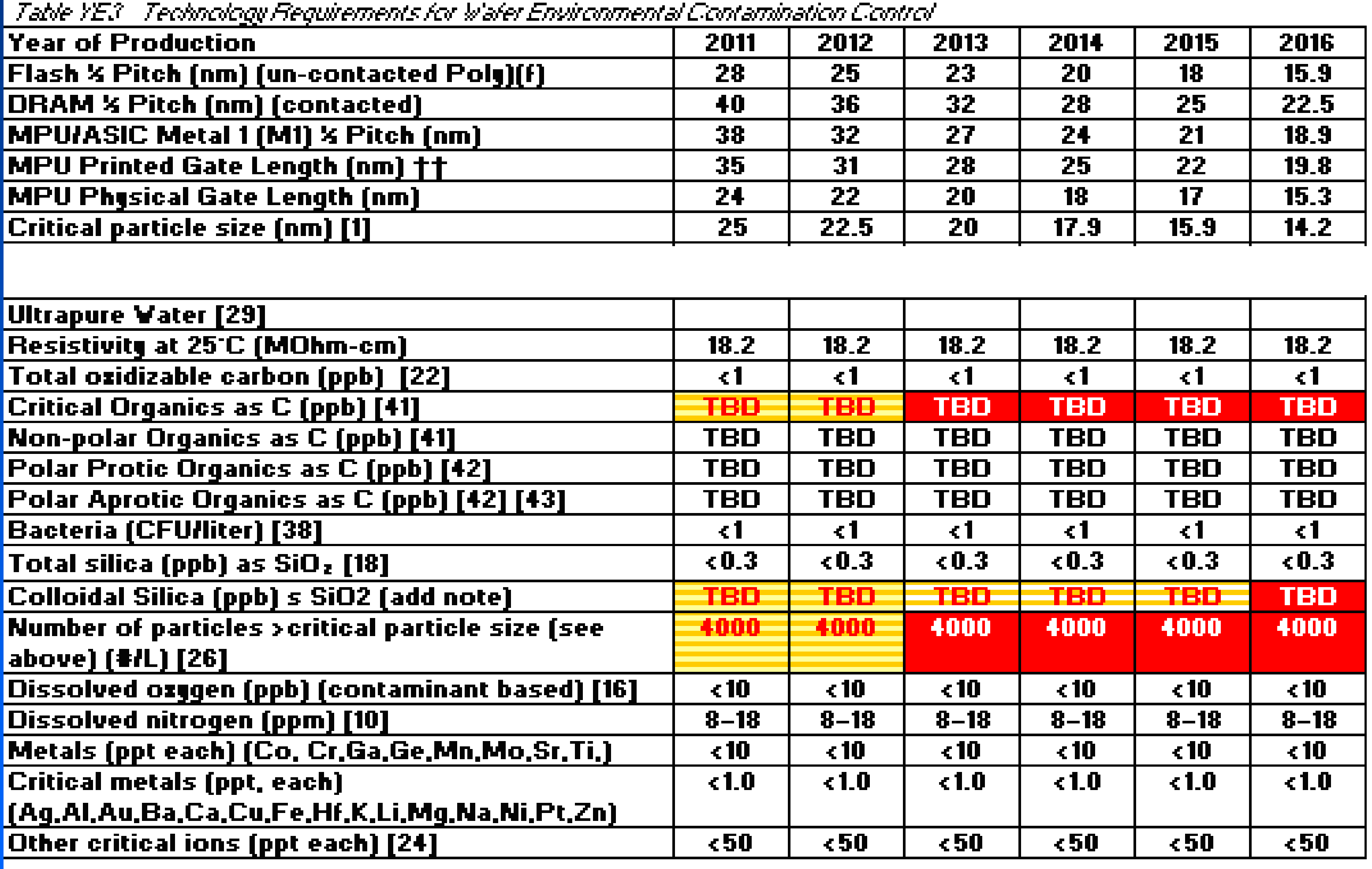
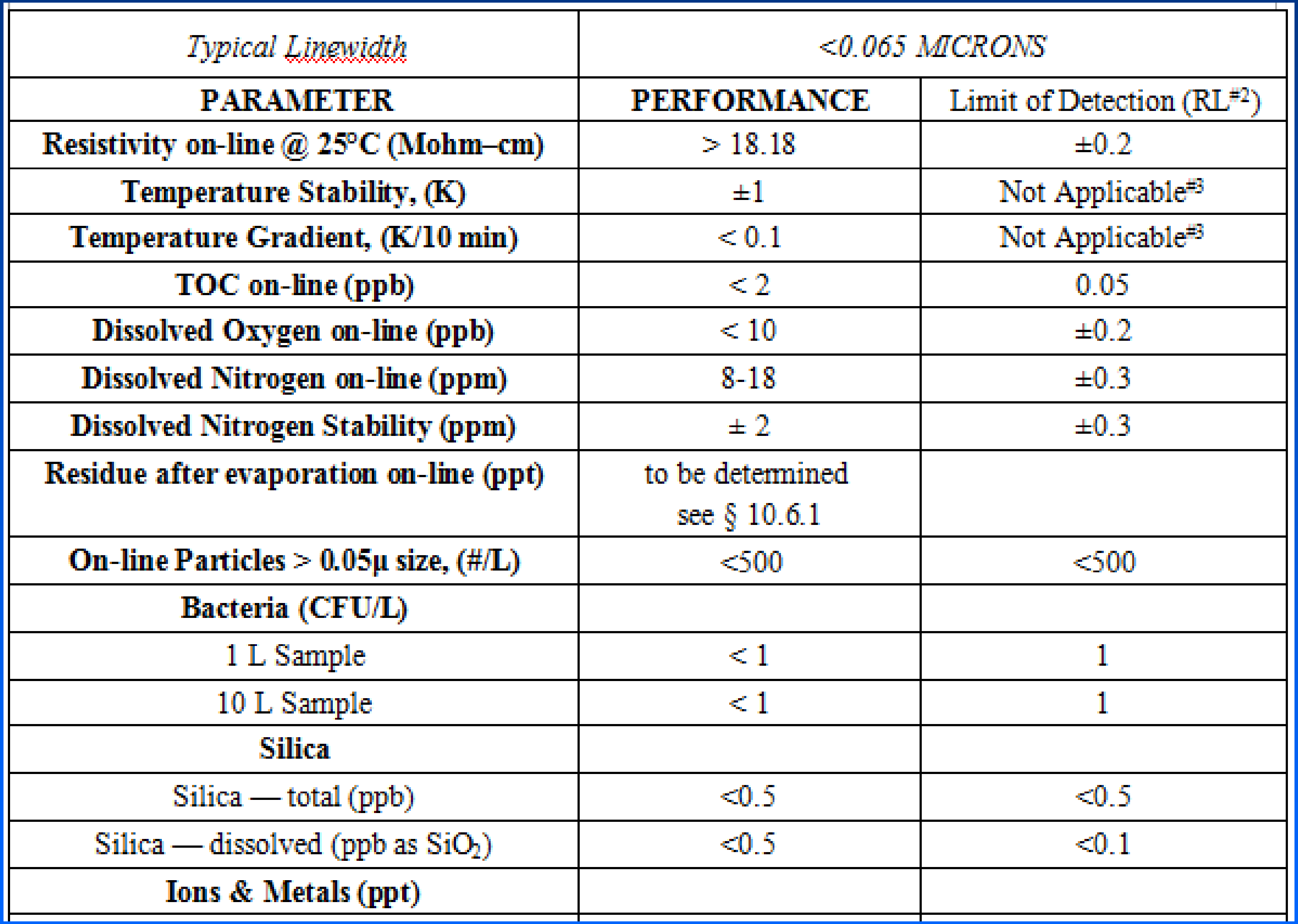


Table YE3 Technology Requirements for Wafer Environmental Contamination Control

Year of Production	2011	2012	2013	2014	2015	2016
Flash ½ Pitch (nm) (un-contacted Poly)(f)	28	25	23	20	18	15.9
DRAM ½ Pitch (nm) (contacted)	40	36	32	28	25	22.5
MPU/ASIC Metal 1 (M1) ½ Pitch (nm)	38	32	27	24	21	18.9
MPU Printed Gate Length (nm) ††	35	31	28	25	22	19.8
MPU Physical Gate Length (nm)	24	22	20	18	17	15.3
Critical particle size (nm) [1]	25	22.5	20	17.9	15.9	14.2
Ultrapure Water [29]						
Resistivity at 25°C (MOhm-cm)	18.2	18.2	18.2	18.2	18.2	18.2
Total oxidizable carbon (ppb) [22]	<1	<1	<1	<1	<1	<1
Critical Organics as C (ppb) [41]	TBD	TBD	TBD	TBD	TBD	TBD
Non-polar Organics as C (ppb) [41]	TBD	TBD	TBD	TBD	TBD	TBD
Polar Protic Organics as C (ppb) [42]	TBD	TBD	TBD	TBD	TBD	TBD
Polar Aprotic Organics as C (ppb) [42] [43]	TBD	TBD	TBD	TBD	TBD	TBD
Bacteria (CFU/liter) [38]	<1	<1	<1	<1	<1	<1
Total silica (ppb) as SiO_2 [18]	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3
Colloidal Silica (ppb) s SiO2 (add note)	TBD	TBD	TBD	TBD	TBD	TBD
Number of particles >critical particle size (see above) (#/L) [26]	4000	4000	4000	4000	4000	4000
Dissolved oxygen (ppb) (contaminant based) [16]	<10	<10	<10	<10	<10	<10
Dissolved nitrogen (ppm) [10]	8–18	8–18	8–18	8–18	8–18	8–18
Metals (ppt each) (Co, Cr,Ga,Ge,Mn,Mo,Sr,Ti,)	<10	<10	<10	<10	<10	<10
Critical metals (ppt, each) (Ag,Al,Au,Ba,Ca,Cu,Fe,Hf,K,Li,Mg,Na,Ni,Pt,Zn)	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Other critical ions (ppt each) [24]	<50	<50	<50	<50	<50	<50

SEMI F63’s Recommended UPW Specifications



Typical Linewidth	*<0.065 MICRONS*	
PARAMETER	**PERFORMANCE**	Limit of Detection (RL[#2])
Resistivity on-line @ 25°C (Mohm–cm)	> 18.18	±0.2
Temperature Stability, (K)	±1	Not Applicable[#3]
Temperature Gradient, (K/10 min)	< 0.1	Not Applicable[#3]
TOC on-line (ppb)	< 2	0.05
Dissolved Oxygen on-line (ppb)	< 10	±0.2
Dissolved Nitrogen on-line (ppm)	8-18	±0.3
Dissolved Nitrogen Stability (ppm)	± 2	±0.3
Residue after evaporation on-line (ppt)	to be determined see § 10.6.1	
On-line Particles > 0.05µ size, (#/L)	<500	<500
Bacteria (CFU/L)		
1 L Sample	< 1	1
10 L Sample	< 1	1
Silica		
Silica — total (ppb)	<0.5	<0.5
Silica — dissolved (ppb as SiO_2)	<0.5	<0.1
Ions & Metals (ppt)		

Process Risk Calculation for each Individual Ion or Metal

Severity of Deposition

1 unlikely, 2 minor, 3 moderate, 4 major, 5 severe

Occurrence

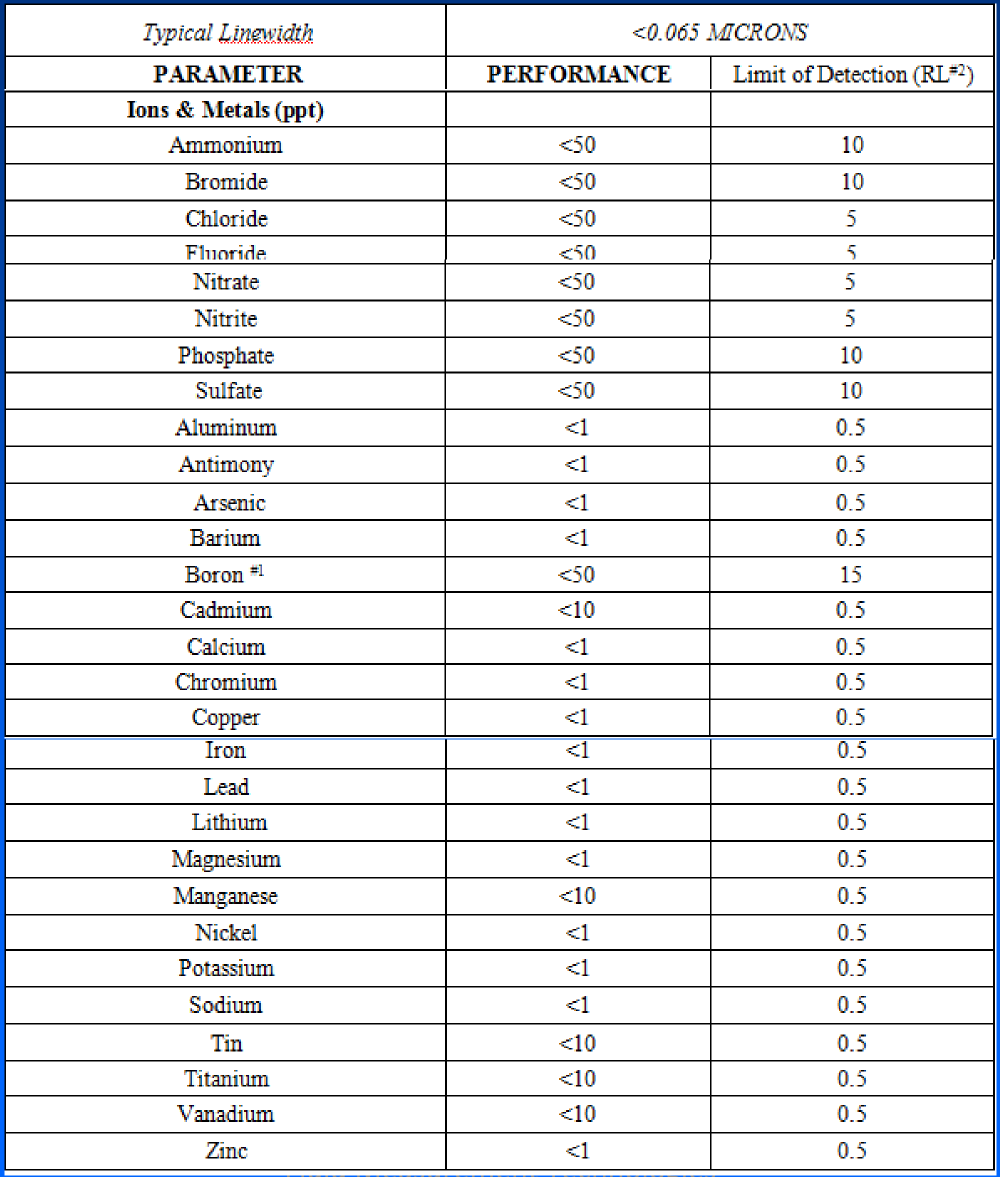
1 yearly, 2 quarterly, 3 monthly, 4 weekly, 5 daily

Detection

1 continuous, 2 hourly, 3 daily, 4 weekly, 5 monthly

🟨 **Low risk**	🟧	🟧	🟥 **High risk**

SEMI F63's Recommended UPW Specification for Ions and Metals



Typical Linewidth	*<0.065 MICRONS*	
PARAMETER	**PERFORMANCE**	Limit of Detection (RL$^{\#2}$)
Ions & Metals (ppt)		
Ammonium	<50	10
Bromide	<50	10
Chloride	<50	5
Fluoride	<50	5
Nitrate	<50	5
Nitrite	<50	5
Phosphate	<50	10
Sulfate	<50	10
Aluminum	<1	0.5
Antimony	<1	0.5
Arsenic	<1	0.5
Barium	<1	0.5
Boron $^{\#1}$	<50	15
Cadmium	<10	0.5
Calcium	<1	0.5
Chromium	<1	0.5
Copper	<1	0.5
Iron	<1	0.5
Lead	<1	0.5
Lithium	<1	0.5
Magnesium	<1	0.5
Manganese	<10	0.5
Nickel	<1	0.5
Potassium	<1	0.5
Sodium	<1	0.5
Tin	<10	0.5
Titanium	<10	0.5
Vanadium	<10	0.5
Zinc	<1	0.5

ASTM Standard Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industries

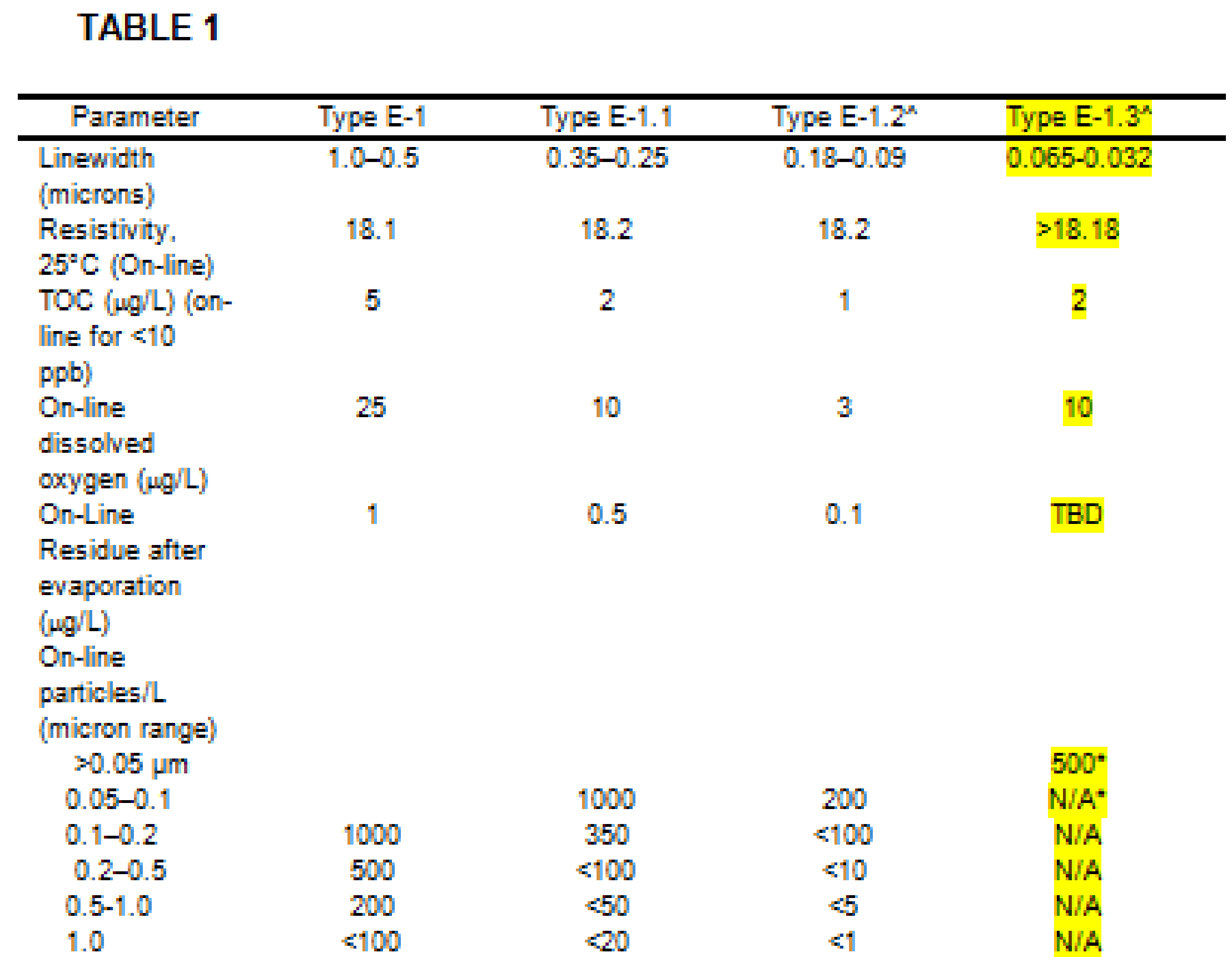
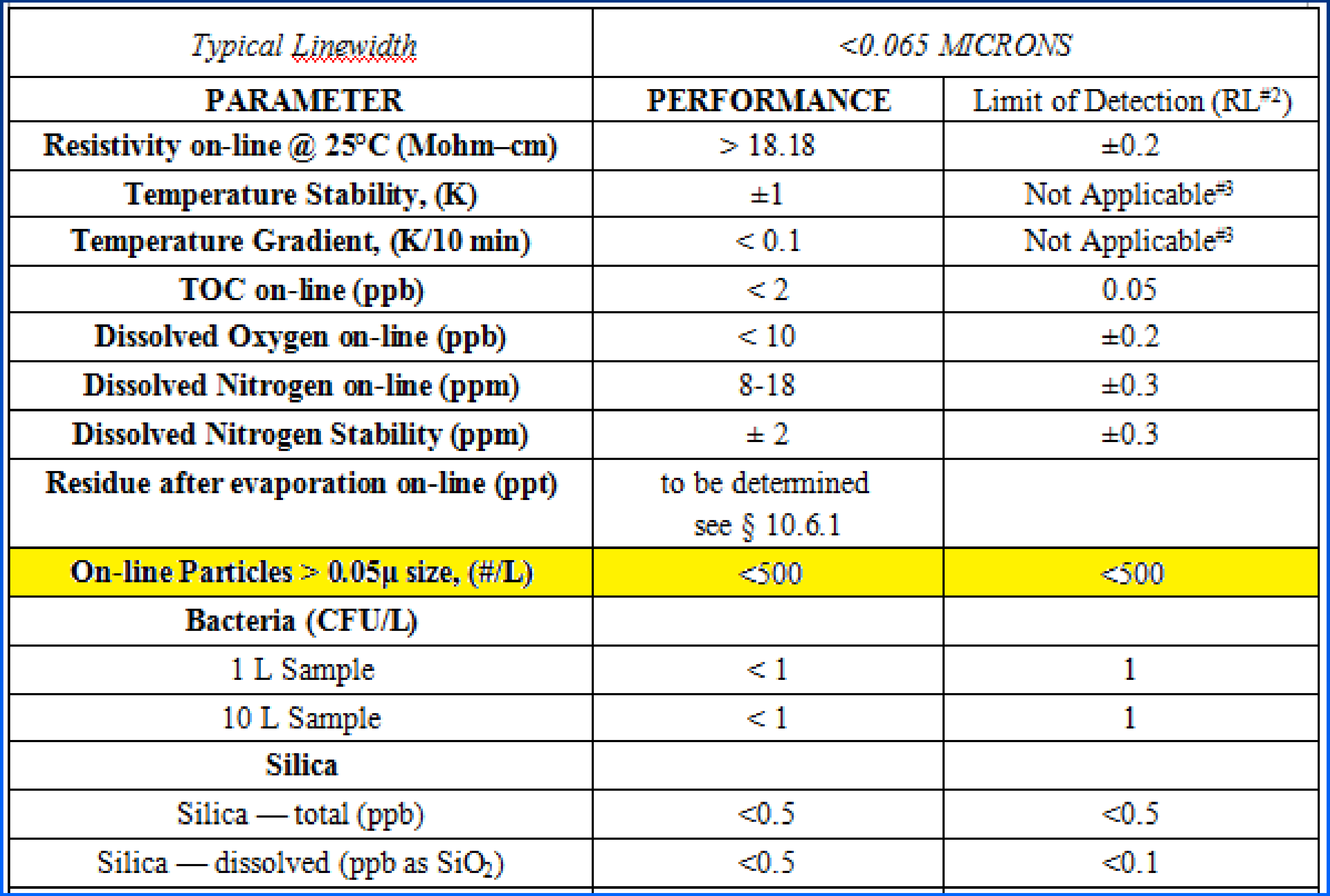


TABLE 1

Parameter	Type E-1	Type E-1.1	Type E-1.2^A	Type E-1.3^A
Linewidth (microns)	1.0–0.5	0.35–0.25	0.18–0.09	0.065-0.032
Resistivity, 25°C (On-line)	18.1	18.2	18.2	>18.18
TOC (μg/L) (on-line for <10 ppb)	5	2	1	2
On-line dissolved oxygen (μg/L)	25	10	3	10
On-Line Residue after evaporation (μg/L)	1	0.5	0.1	TBD
On-line particles/L (micron range)				
>0.05 μm				500*
0.05–0.1		1000	200	N/A*
0.1–0.2	1000	350	<100	N/A
0.2–0.5	500	<100	<10	N/A
0.5-1.0	200	<50	<5	N/A
1.0	<100	<20	<1	N/A

SEMI F63's Recommended UPW Specifications



Typical Linewidth	*<0.065 MICRONS*	
PARAMETER	**PERFORMANCE**	Limit of Detection (RL[#2])
Resistivity on-line @ 25°C (Mohm–cm)	> 18.18	±0.2
Temperature Stability, (K)	±1	Not Applicable[#3]
Temperature Gradient, (K/10 min)	< 0.1	Not Applicable[#3]
TOC on-line (ppb)	< 2	0.05
Dissolved Oxygen on-line (ppb)	< 10	±0.2
Dissolved Nitrogen on-line (ppm)	8-18	±0.3
Dissolved Nitrogen Stability (ppm)	± 2	±0.3
Residue after evaporation on-line (ppt)	to be determined see § 10.6.1	
On-line Particles > 0.05µ size, (#/L)	<500	<500
Bacteria (CFU/L)		
1 L Sample	< 1	1
10 L Sample	< 1	1
Silica		
Silica — total (ppb)	<0.5	<0.5
Silica — dissolved (ppb as SiO_2)	<0.5	<0.1

ITRS Requirements for 2011 and Beyond

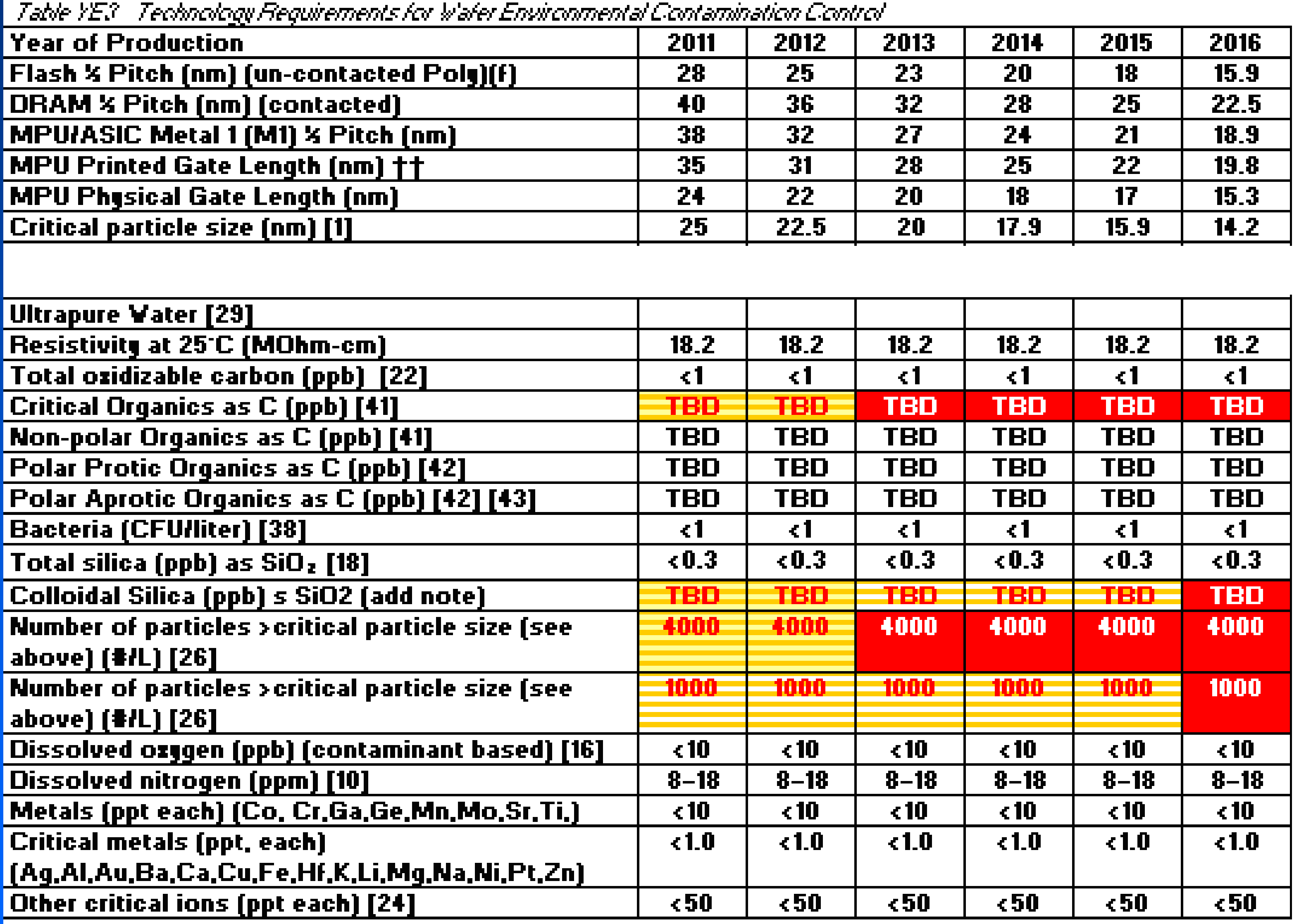


Table YE3 Technology Requirements for Wafer Environmental Contamination Control

Year of Production	2011	2012	2013	2014	2015	2016
Flash ½ Pitch (nm) (un-contacted Poly)(f)	28	25	23	20	18	15.9
DRAM ½ Pitch (nm) (contacted)	40	36	32	28	25	22.5
MPU/ASIC Metal 1 (M1) ½ Pitch (nm)	38	32	27	24	21	18.9
MPU Printed Gate Length (nm) ††	35	31	28	25	22	19.8
MPU Physical Gate Length (nm)	24	22	20	18	17	15.3
Critical particle size (nm) [1]	25	22.5	20	17.9	15.9	14.2
Ultrapure Water [29]						
Resistivity at 25°C (MOhm-cm)	18.2	18.2	18.2	18.2	18.2	18.2
Total oxidizable carbon (ppb) [22]	<1	<1	<1	<1	<1	<1
Critical Organics as C (ppb) [41]	TBD	TBD	TBD	TBD	TBD	TBD
Non-polar Organics as C (ppb) [41]	TBD	TBD	TBD	TBD	TBD	TBD
Polar Protic Organics as C (ppb) [42]	TBD	TBD	TBD	TBD	TBD	TBD
Polar Aprotic Organics as C (ppb) [42] [43]	TBD	TBD	TBD	TBD	TBD	TBD
Bacteria (CFU/liter) [38]	<1	<1	<1	<1	<1	<1
Total silica (ppb) as SiO_2 [18]	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3
Colloidal Silica (ppb) s SiO2 (add note)	TBD	TBD	TBD	TBD	TBD	TBD
Number of particles >critical particle size (see above) (#/L) [26]	4000	4000	4000	4000	4000	4000
Number of particles >critical particle size (see above) (#/L) [26]	1000	1000	1000	1000	1000	1000
Dissolved oxygen (ppb) (contaminant based) [16]	<10	<10	<10	<10	<10	<10
Dissolved nitrogen (ppm) [10]	8–18	8–18	8–18	8–18	8–18	8–18
Metals (ppt each) (Co, Cr,Ga,Ge,Mn,Mo,Sr,Ti,)	<10	<10	<10	<10	<10	<10
Critical metals (ppt, each) (Ag,Al,Au,Ba,Ca,Cu,Fe,Hf,K,Li,Mg,Na,Ni,Pt,Zn)	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Other critical ions (ppt each) [24]	<50	<50	<50	<50	<50	<50

Light-Scattering Event Monitors (also called Optical Particle Counters, OPCs)

For two decades the semiconductor industry has relied on OPCs to detect particles in UPW and chemicals

OPCs have always had limitations:

A Particle Counter's response is based on how it measures white, spherical, PSL (plastic) beads.

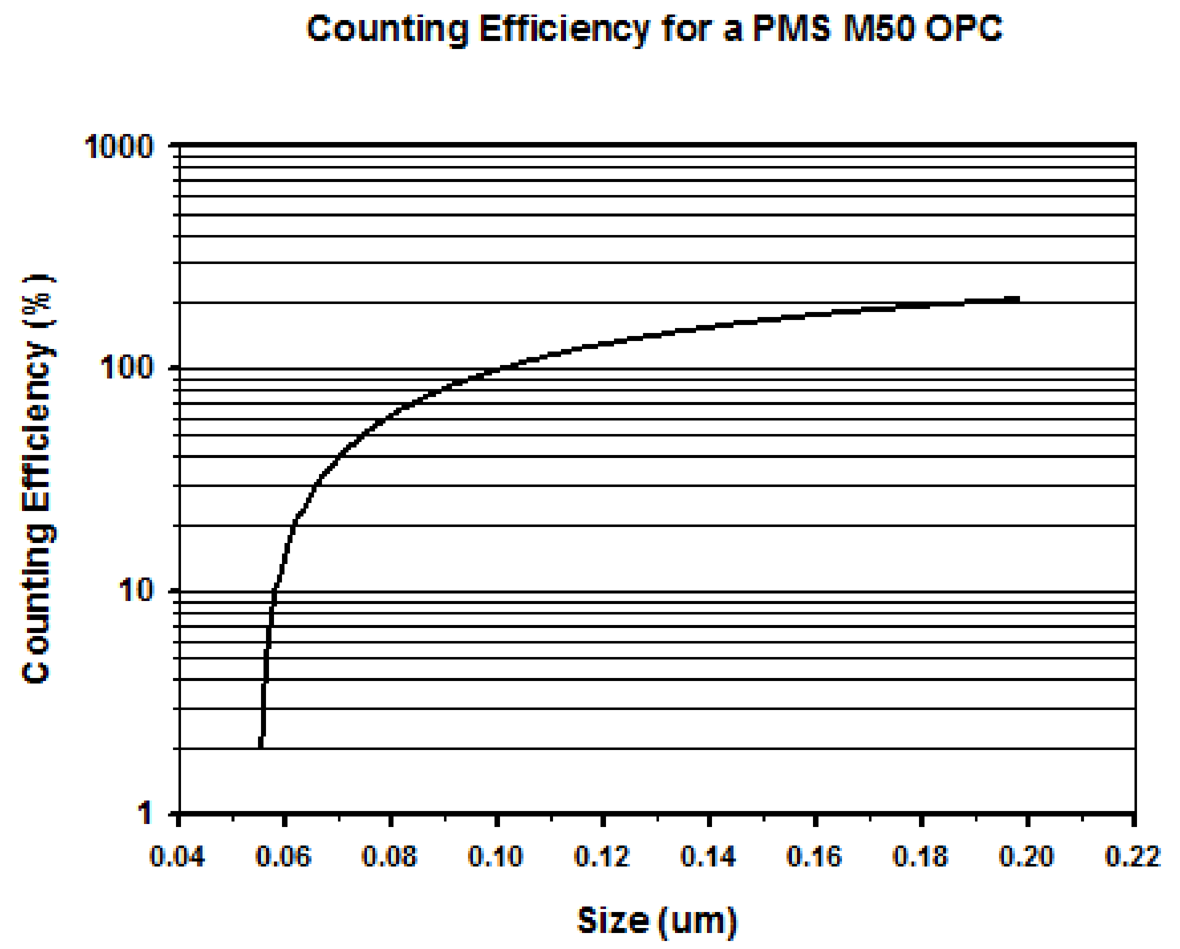
Different manufacturers use different wavelengths, different angles of collection and different definitions of illuminated area. Two OPC's rarely agree with each other.

All Particle Counters measure an equivalent optical Diameter.

The current detection limit is 50nm, with the possibility of 40nm and 30nm in the near future

OPCs have little chance of meeting the future requirements of the ITRS Roadmap.

The counting efficiency for 50nm rated Optical Particle Counters is only a few percent at best at 50nm (data courtesy of Particle Measuring Systems)



Task Force Rational:

- Optical Particle Counters (OPCs) have reached a practical measurement limit of 40 nm, with a counting efficiency of only a few percent at this size.
- The ITRS Roadmap has a critical particle size of 25 nm for 2011.
- Lack of particle metrology and marginal filtration efficiency at 25nm substantially increases the risk to the next generation of the wafer manufacturing technology.
- UPW ITRS has suggested a risk mitigation strategy based on off-line validation of the filter performance. Until the particle metrology gap is solved, this approach mitigates risks and gets users closer to ITRS requirements.
- Current methods of filter performance characterization using 50-200nm PSL spheres and OPCs, and extrapolating performance to 5-15nm is inadequate for guaranteeing filter performance at the extrapolated size.
- A **new method** of quantifying filter performance is required for filters with a pore size of 5-15 nm.

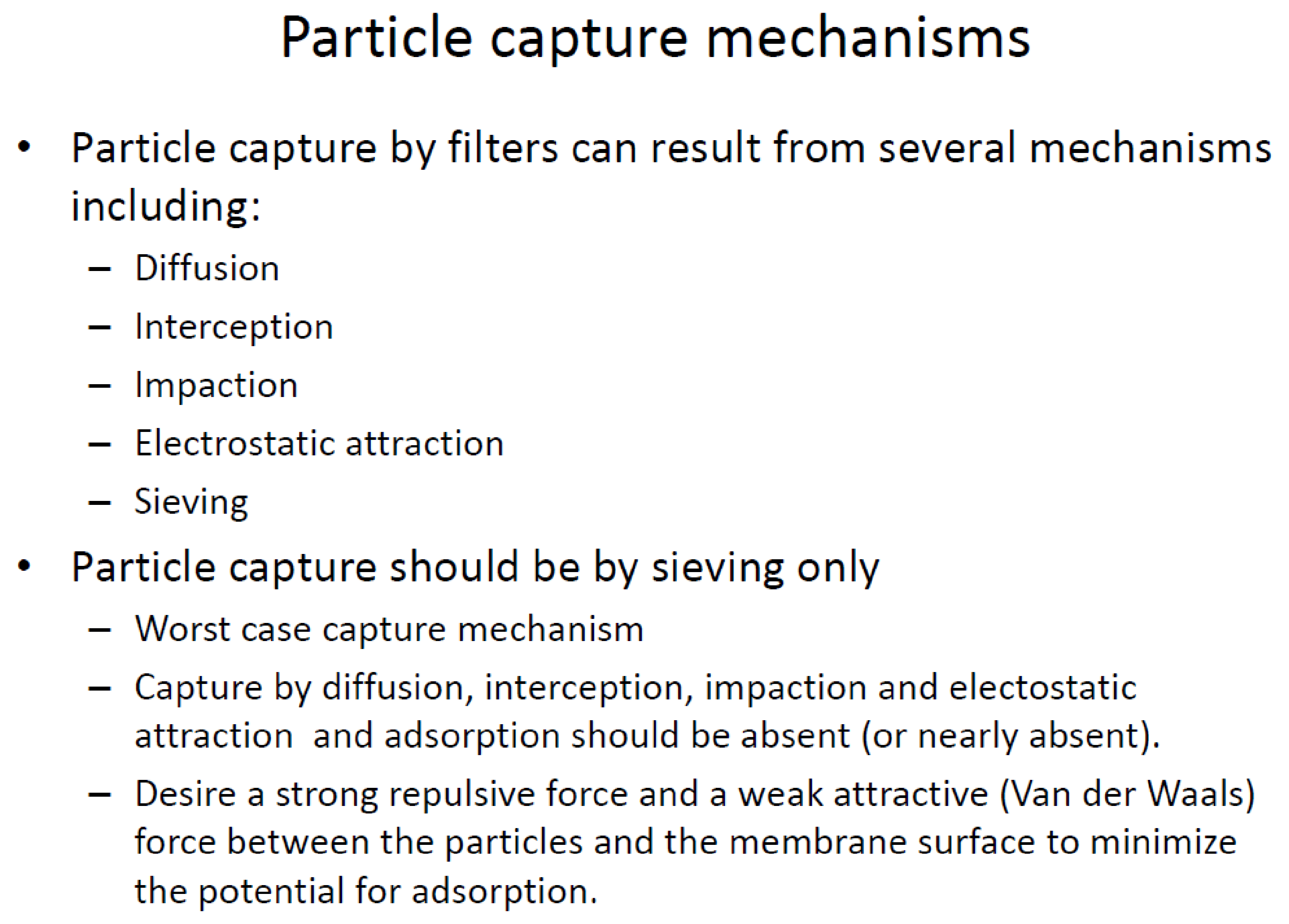
STANDARDS NEW ACTIVITY REPORT FORM (SNARF)

Date Prepared: April 4, 2011 *Revised (if Applicable):* Rev.1

SNARF for: Test Method for Validation of the Efficiency of Final Filtration in Ultrapure Water Production Used in Semiconductor Processing

Originating Global Technical Committee: Liquid Chemicals
Originating Technical Committee Region: NA Liquid Chemicals
Task Force in which work is to be carried out: UPW Filtration Efficiency

Submitted by: Slava Libman ***Company:*** Balazs AirLiquide
e-mail: slava.libman@airliquide.com
Phone: 415-312 4416 ***Fax:***



Particle capture mechanisms

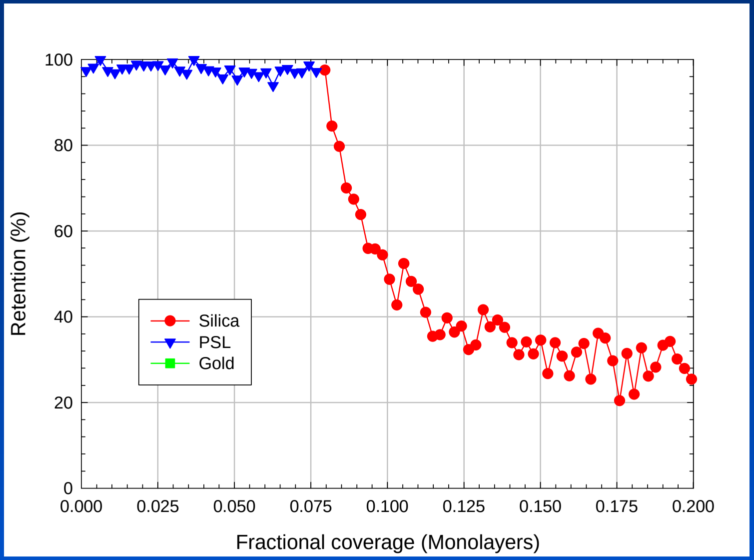
- Particle capture by filters can result from several mechanisms including:
  - Diffusion
  - Interception
  - Impaction
  - Electrostatic attraction
  - Sieving
- Particle capture should be by sieving only
  - Worst case capture mechanism
  - Capture by diffusion, interception, impaction and electostatic attraction and adsorption should be absent (or nearly absent).
  - Desire a strong repulsive force and a weak attractive (Van der Waals) force between the particles and the membrane surface to minimize the potential for adsorption.

Courtesy of CT Associates

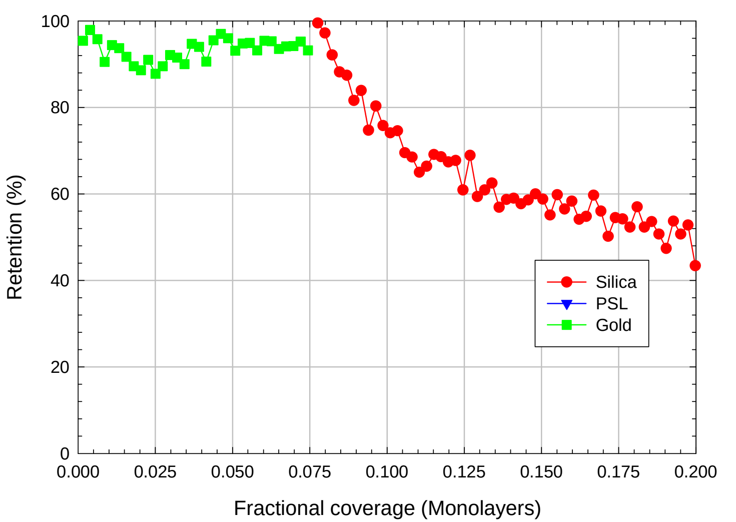
Methodology: Three Phases of Sieving Filtration

- Initial
  - Retention of an un-loaded filter
- Loading
  - Retention decreases as smaller pores in the filter are selectively clogged
  - Retention begins to decrease almost immediately
- Cake filtration
  - When filters are sufficiently loaded the retained particles begin to retain smaller particles
  - This is expected to increase the differential pressure
  - Does not occur until the filter is heavily loaded
    - Based on historical data, UPW filters do not show increase in the differential pressure => unlikely to occur

Particle chemistry can have a significant effect on measured filter retention

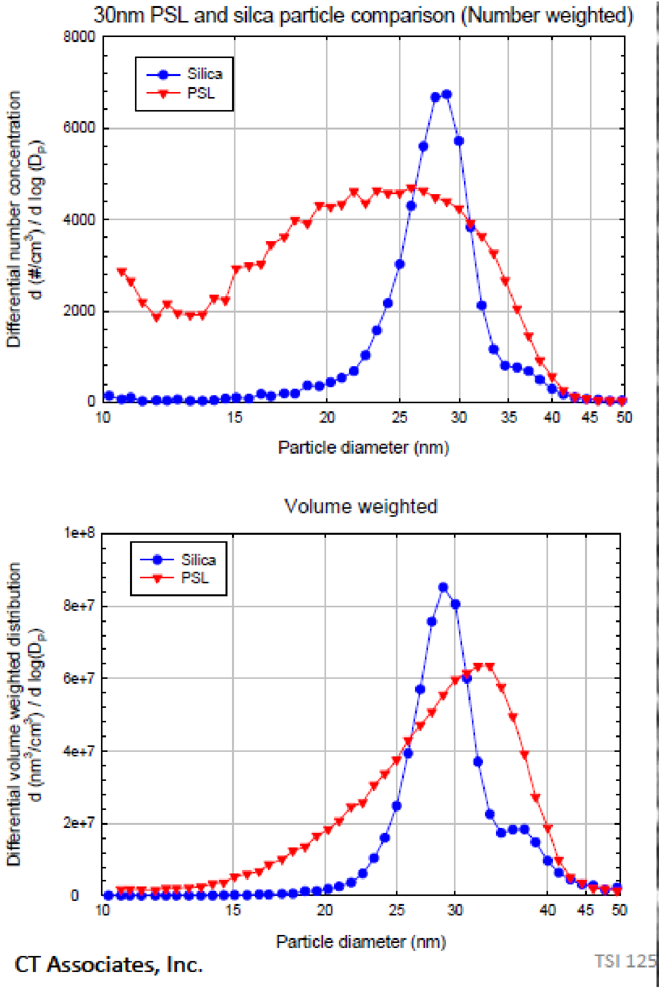


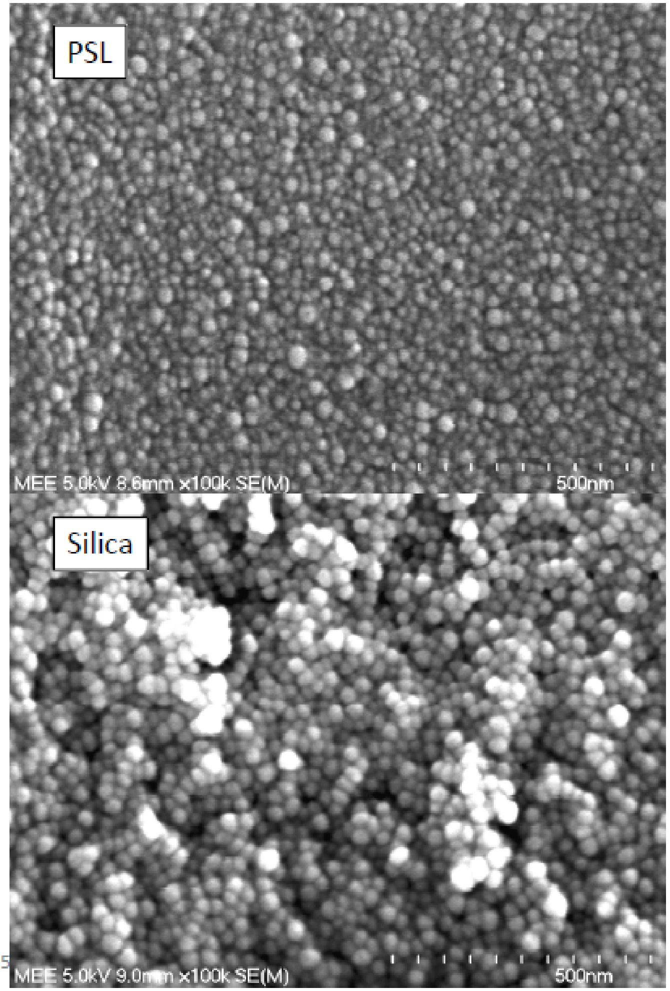
Retention of different 30nm particles types by a commercially available UPW filter cartridges



- Each filter was tested with a sequence of particle types.
- Si particle curves display sieving mechanism
- In all cases the challenge concentration was 2E8/mL.

PSL is no longer mono-sized at 30nm





Colloidal gold particles are as mono-sized at Si, but require surface modification to reduce capture by absorption

Particle type comparison

Particle Type	Sizes Available	"Real World"?	Sieving only?	Cost of particles per gram
PSL	Yes	No	Can be achieved by adding surfactant.	$1,800
Colloidal Gold	Yes	No	Can be achieved by surface modification.	$23,000
Colloidal Silica	Yes	Yes	Yes ?	$0.08

Colloidal silica appears to be the best choice.

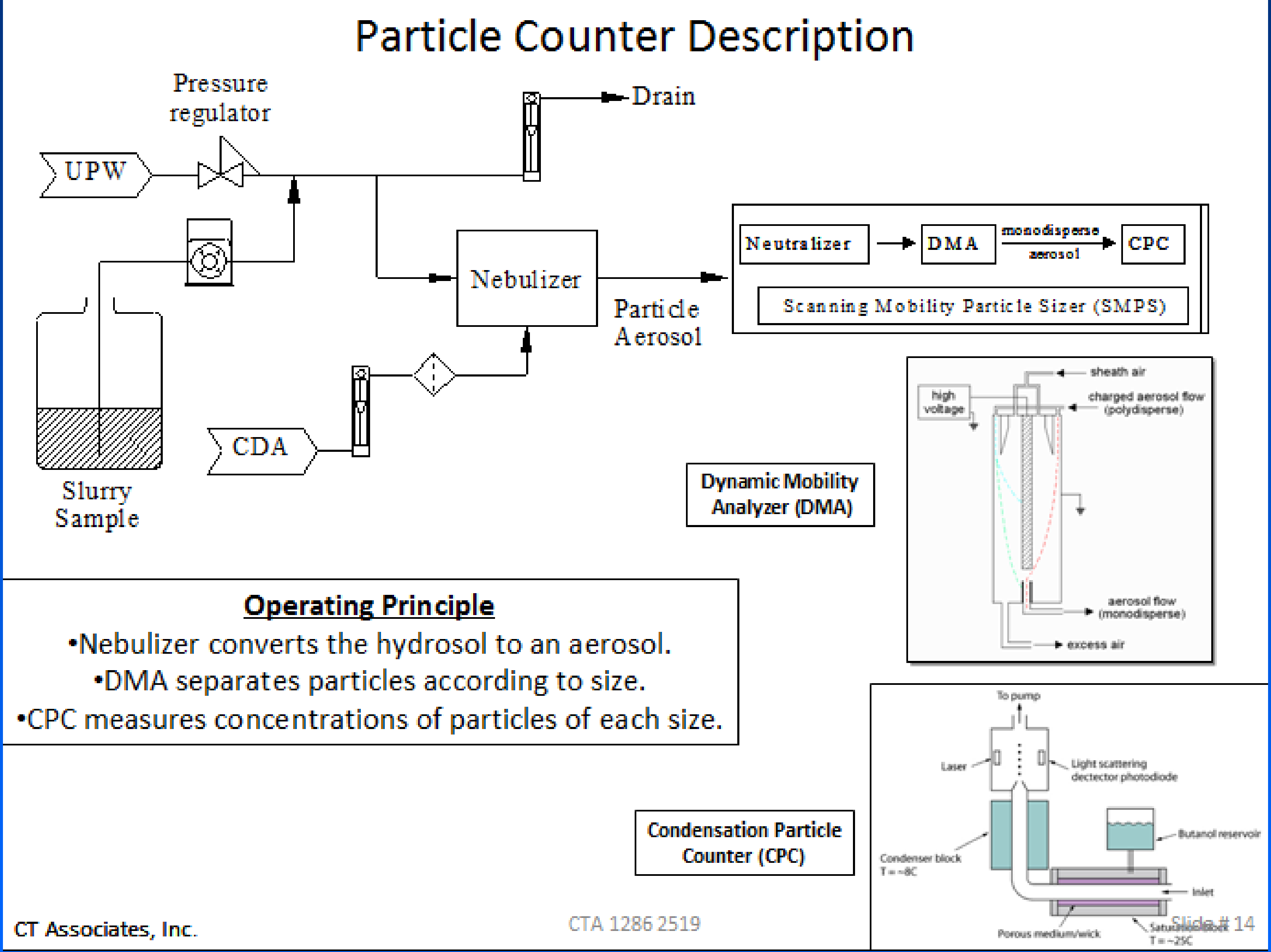
Colloidal Silica

Typical end uses for colloidal silica include coatings for corrosion control, ink receptive papers, metal casting, refractory products, and catalysts.

Commercially available colloidal silica were never intended as a standard for particle counters.

There is no such entity as a NIST or AIST traceable colloidal silica standard, and there probably will not be for the foreseeable future.

New Particle Counter being Developed by CT Associates and TSI



Particle Counter Description

Dynamic Mobility Analyzer (DMA)

Condensation Particle Counter (CPC)

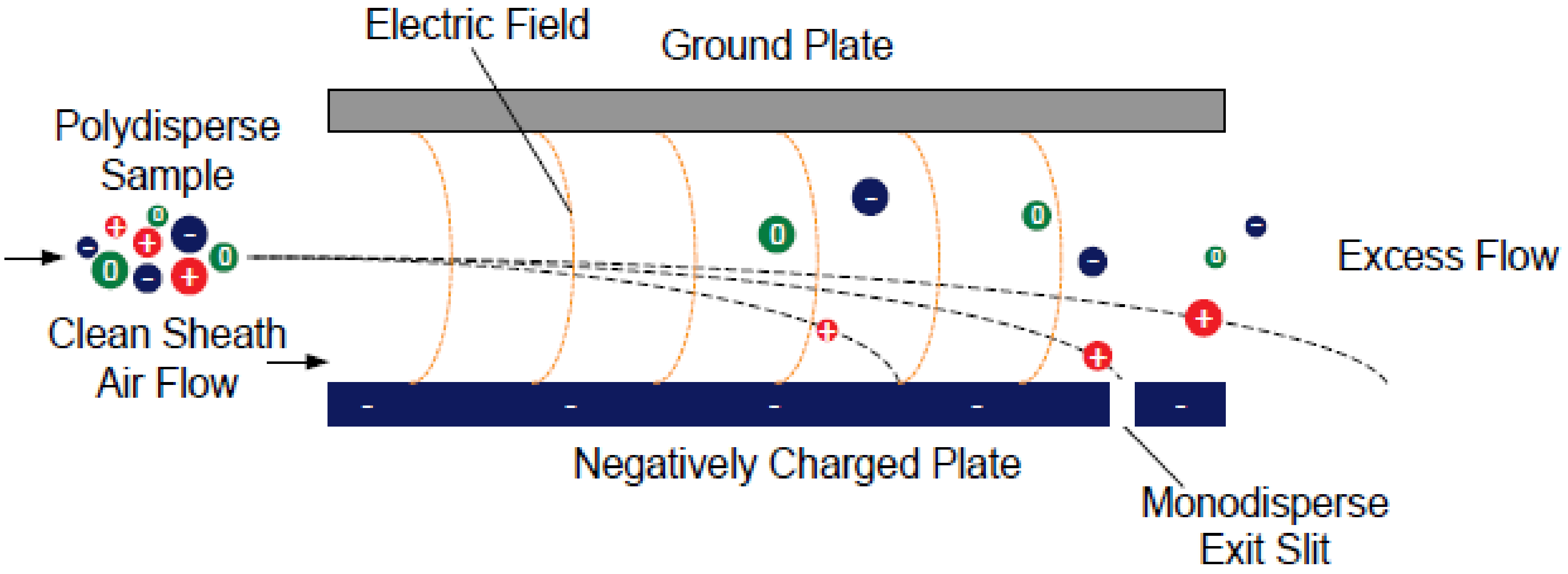
Operating Principle

- Nebulizer converts the hydrosol to an aerosol.
- DMA separates particles according to size.
- CPC measures concentrations of particles of each size.

CT Associates, Inc.

CTA 1286 2519

Slide # 14



(1) $$F_{electric} = n_p e E$$

(2) $$F_{viscous\ drag} = \frac{3\pi\mu D_p v}{C}$$

(3) $$F_{electric} = F_{viscous\ drag}$$

(4) $$Z_p = \frac{v}{E} = \frac{n_p e C}{3\pi\mu D_p}$$

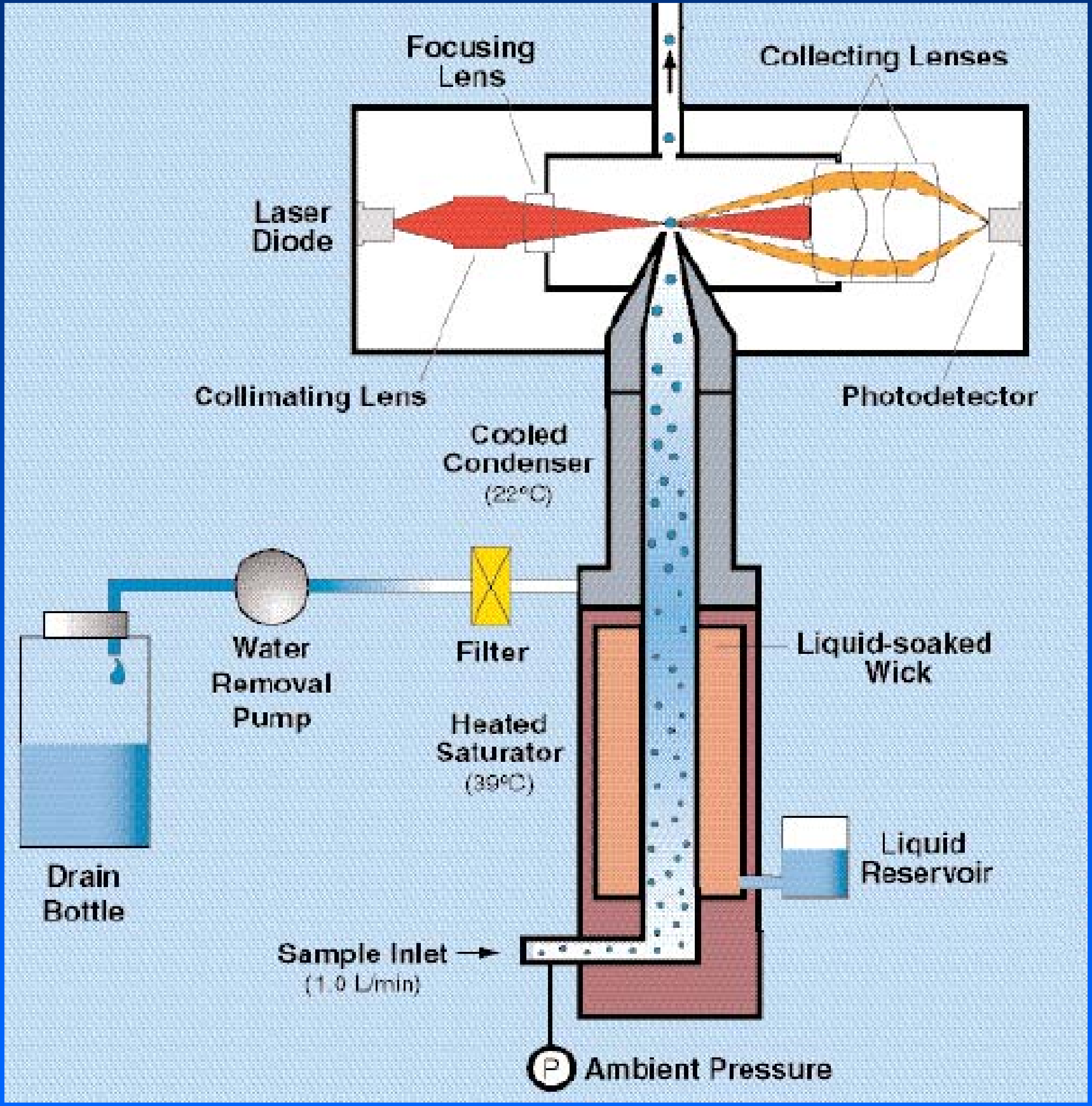
Where:

n_p = number of charges per particle
e = elementary unit of charge
E = electric field strength
μ = viscosity of gas
D_p = particle diameter
C^* = Cunningham slip correction
v = Velocity

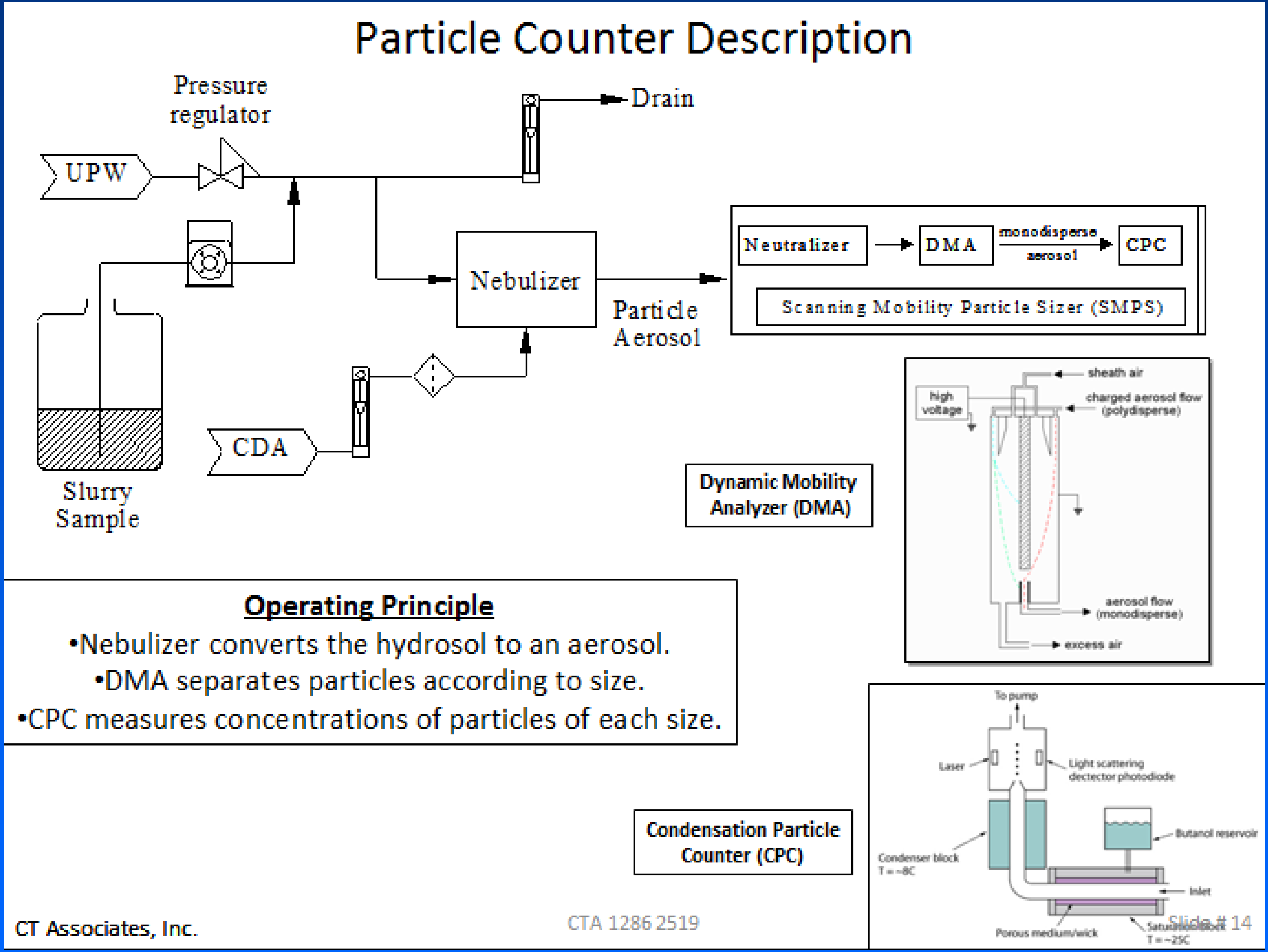
**C is a lower order function of particle diameter*

Only when a particle experiences an equal force from the electric field and the drag due to the gas flow, can the particle exit through the Monodisperse Slit.

Schematic of Condensation Particle Counter



New Particle Counter being Developed by CT Associates and TSI



Particle Counter Description

Operating Principle

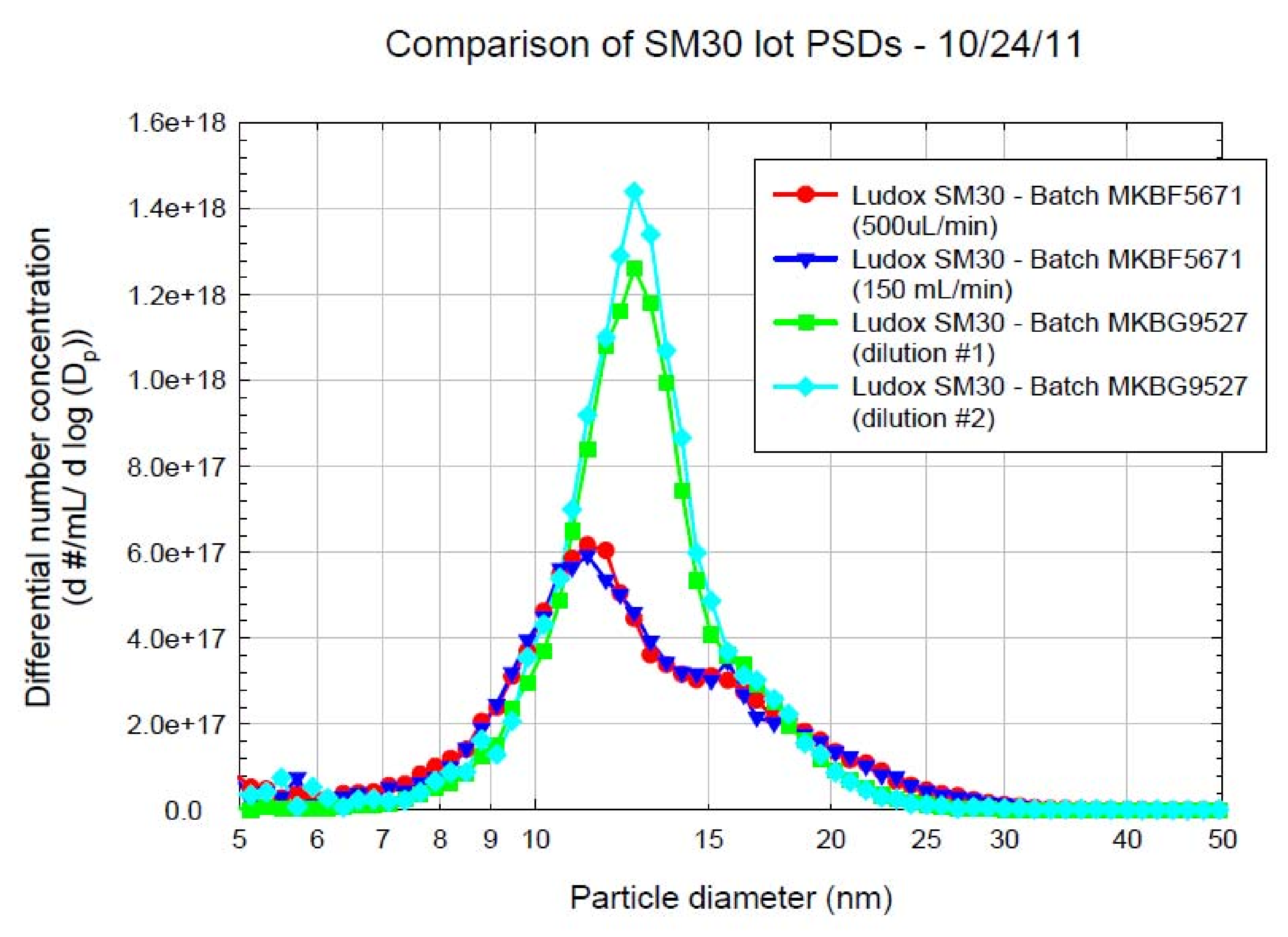
- Nebulizer converts the hydrosol to an aerosol.
- DMA separates particles according to size.
- CPC measures concentrations of particles of each size.

CT Associates, Inc.

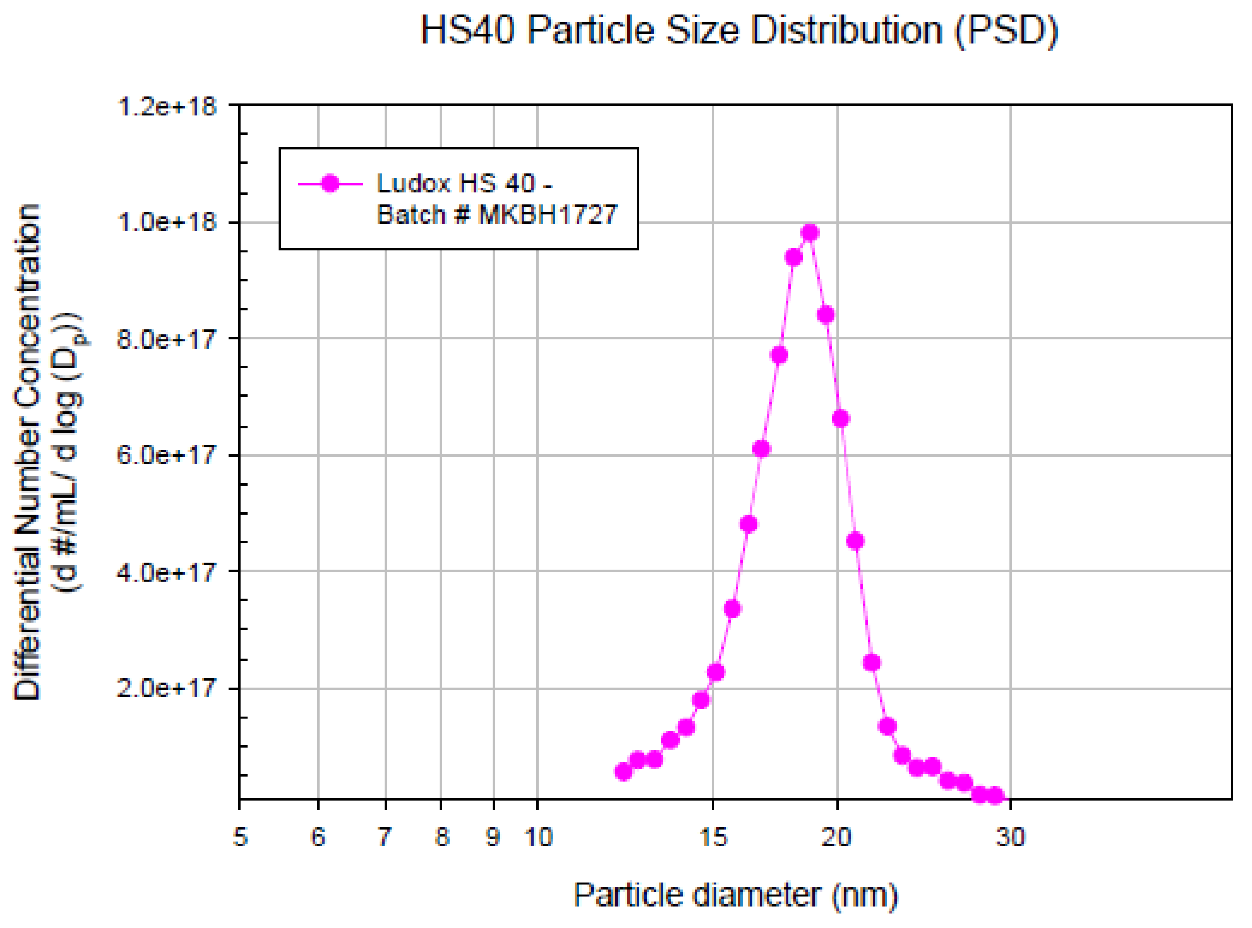
CTA 1286 2519

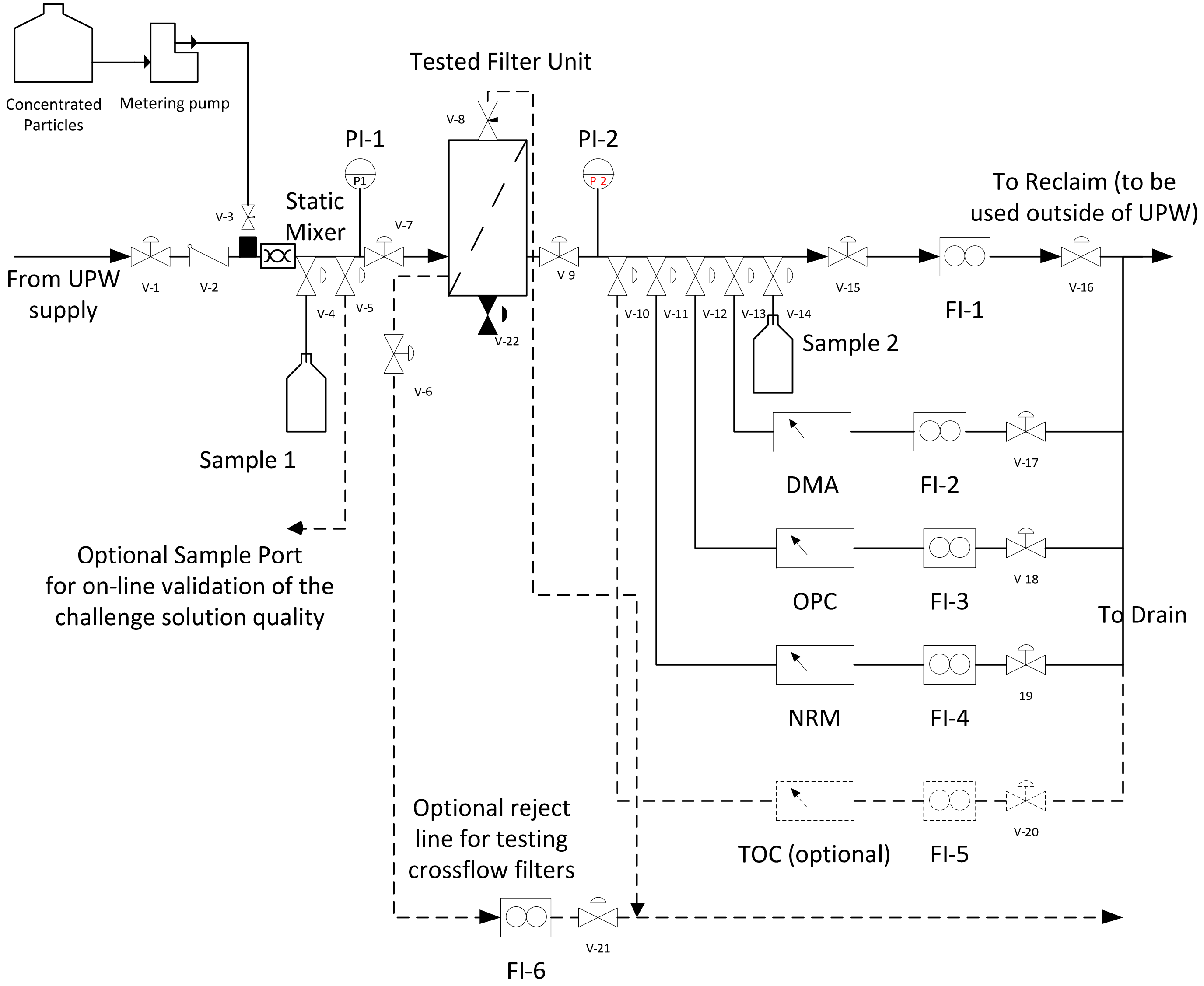
Slide # 14

Particle size distributions for Ludox® colloidal silica suspensions

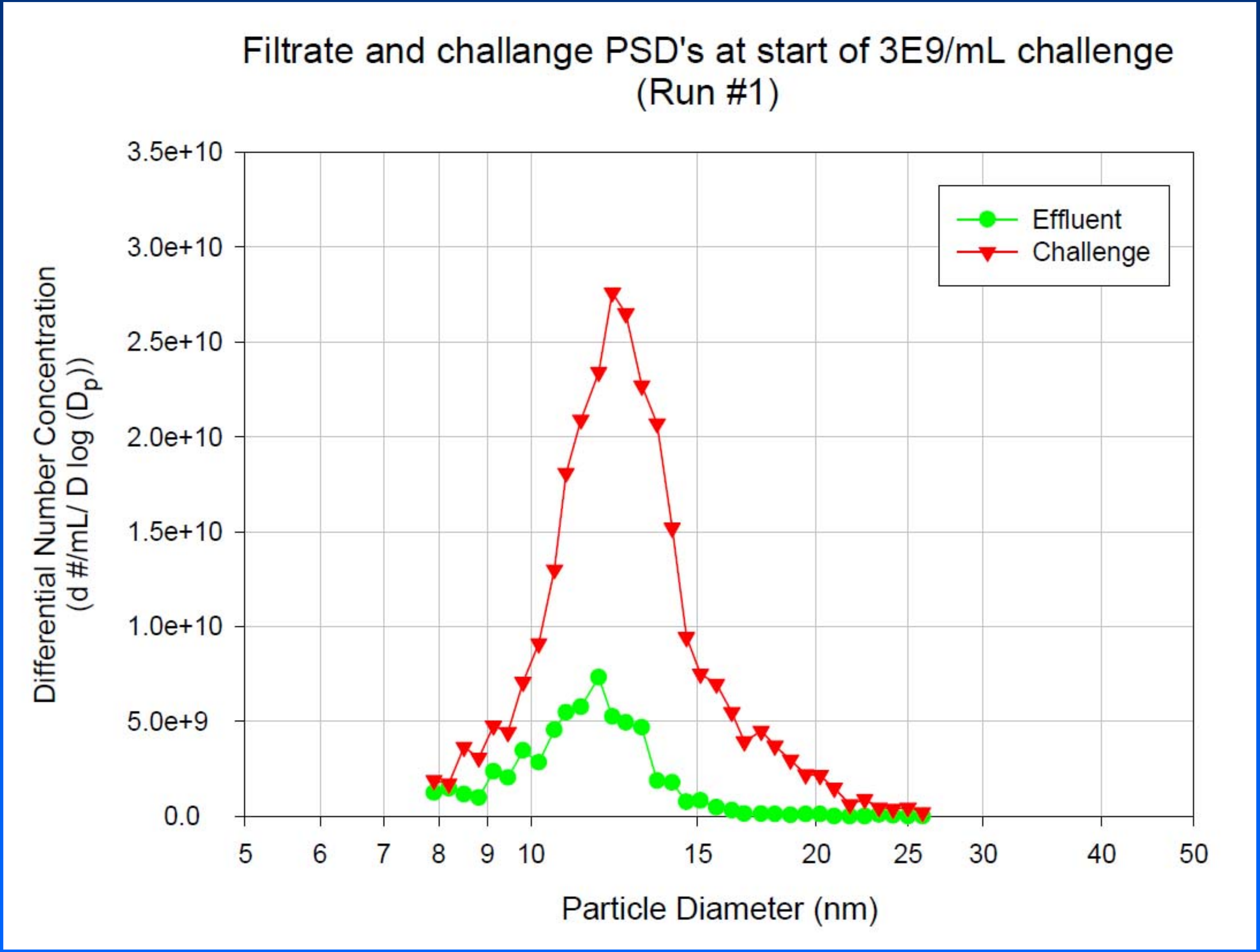


Colloidal silica mean size 18nm

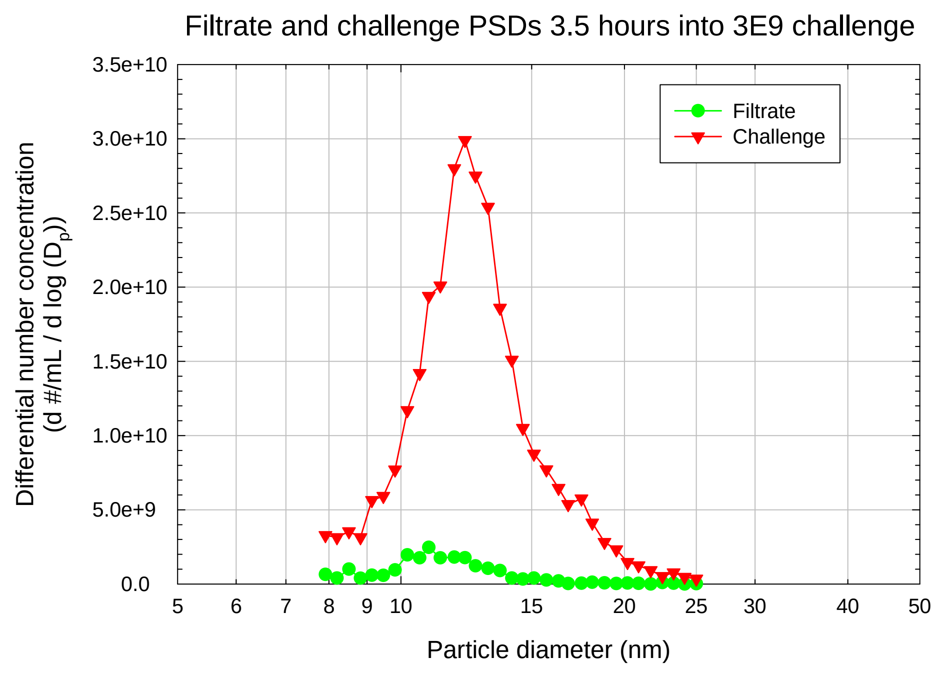


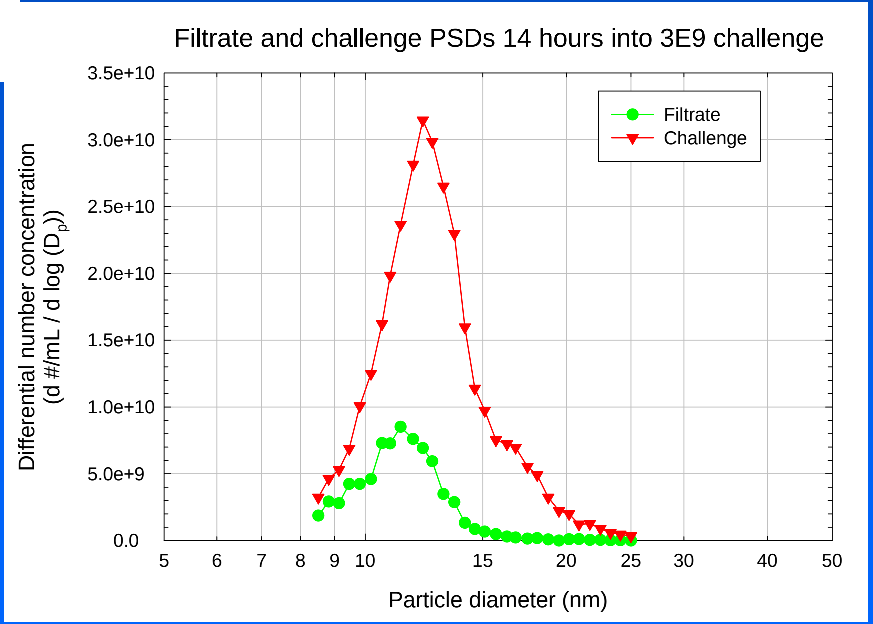


Particle Challenge Test Skid built at Pall Corporation

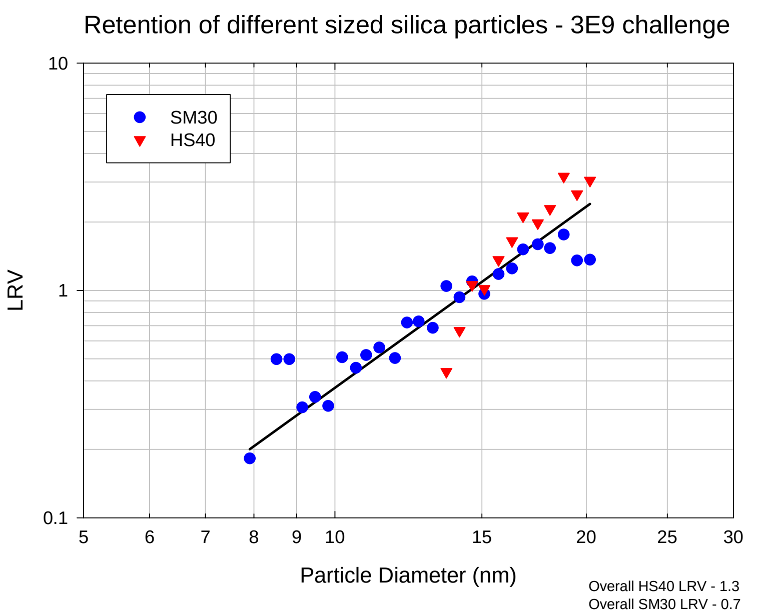

Filtrate and challange PSD's at start of 3E9/mL challenge
(Run #1)
Differential Number Concentration
(d #/mL/ D log (D_p))
3.5e+10
3.0e+10
2.5e+10
2.0e+10
1.5e+10
1.0e+10
5.0e+9
0.0
5
6
7
8
9
10
15
20
25
30
40
50
Particle Diameter (nm)
Effluent
Challenge

Effect of Particle Loading on Filter Retention

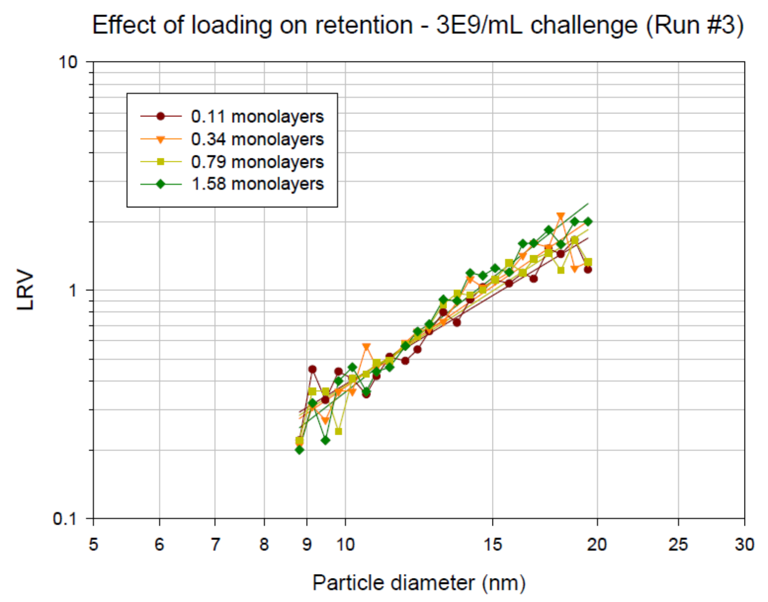


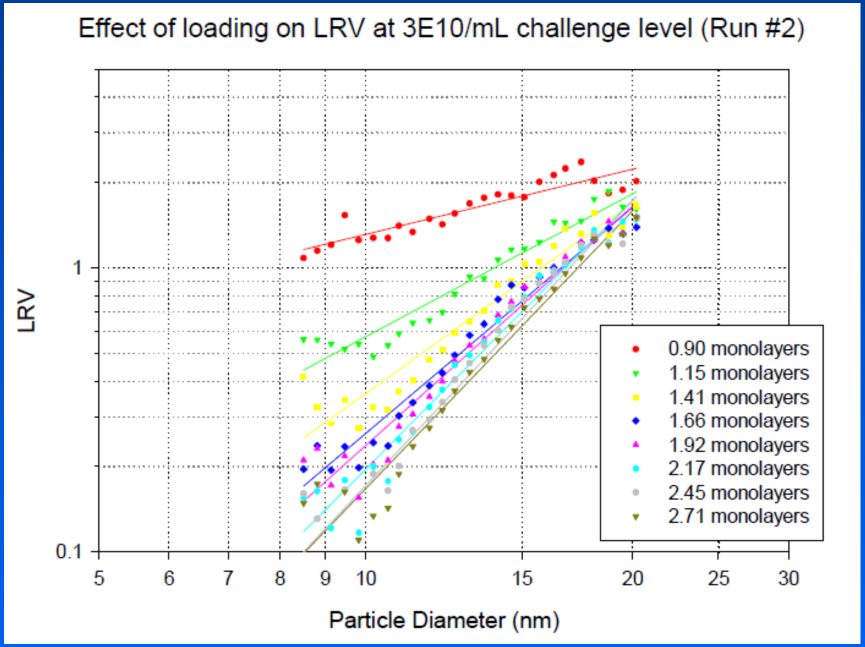


Effect of Particle Diameter and Loading on Filter Performance


Retention of different sized silica particles - 3E9 challenge

Overall HS40 LRV - 1.3
Overall SM30 LRV - 0.7


Effect of loading on retention - 3E9/mL challenge (Run #3)


Effect of loading on LRV at 3E10/mL challenge level (Run #2)

90% efficient = 1 (LRV)

99% efficient = 2 (LRV)

99.9% efficient = 3 (LRV)

ITRS Requirements for 2011 and Beyond

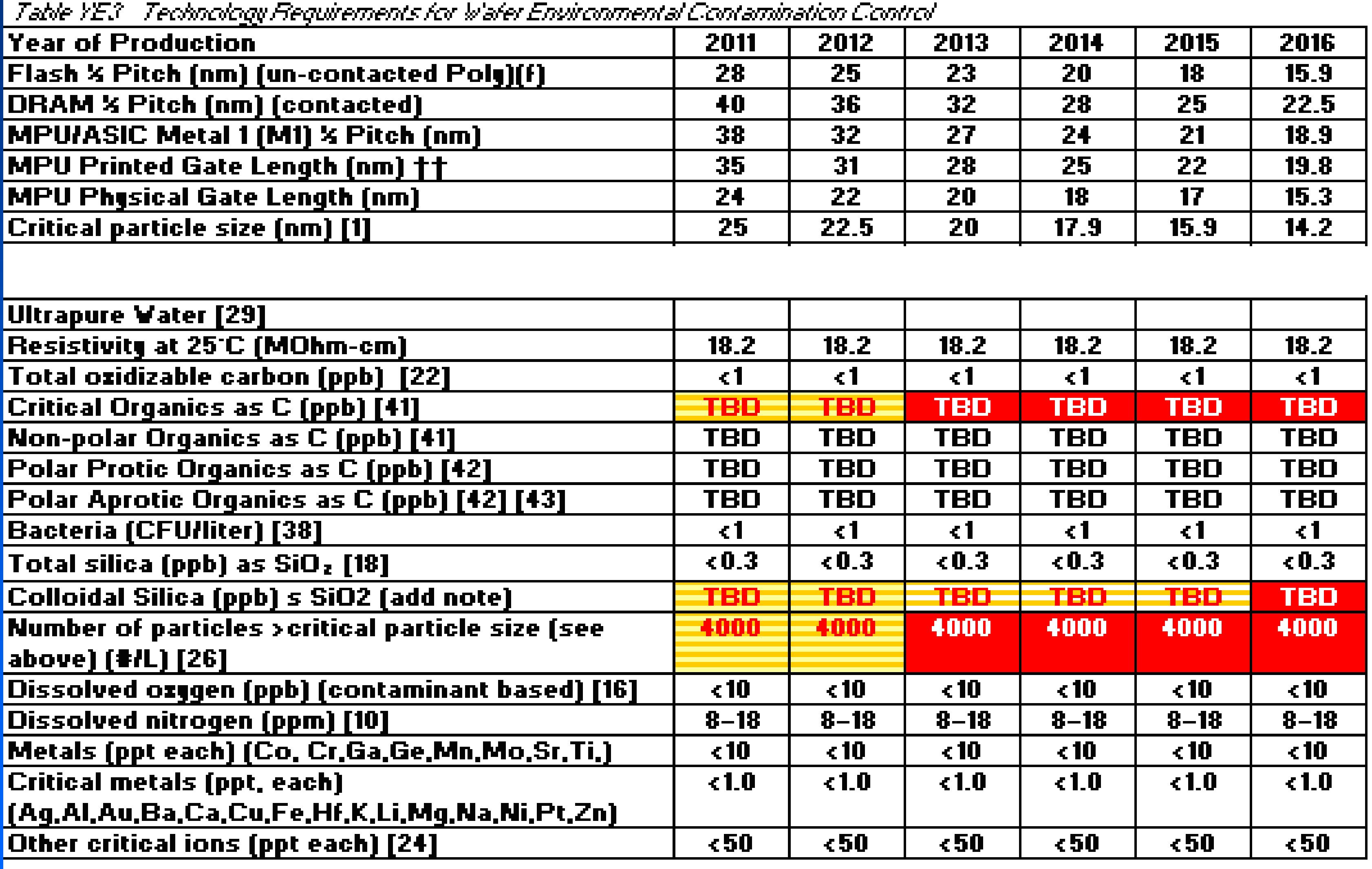


Table YE3 Technology Requirements for Wafer Environmental Contamination Control

Year of Production	2011	2012	2013	2014	2015	2016
Flash ½ Pitch (nm) (un-contacted Poly)(f)	28	25	23	20	18	15.9
DRAM ½ Pitch (nm) (contacted)	40	36	32	28	25	22.5
MPU/ASIC Metal 1 (M1) ½ Pitch (nm)	38	32	27	24	21	18.9
MPU Printed Gate Length (nm) ††	35	31	28	25	22	19.8
MPU Physical Gate Length (nm)	24	22	20	18	17	15.3
Critical particle size (nm) [1]	25	22.5	20	17.9	15.9	14.2
Ultrapure Water [29]						
Resistivity at 25°C (MOhm-cm)	18.2	18.2	18.2	18.2	18.2	18.2
Total oxidizable carbon (ppb) [22]	<1	<1	<1	<1	<1	<1
Critical Organics as C (ppb) [41]	TBD	TBD	TBD	TBD	TBD	TBD
Non-polar Organics as C (ppb) [41]	TBD	TBD	TBD	TBD	TBD	TBD
Polar Protic Organics as C (ppb) [42]	TBD	TBD	TBD	TBD	TBD	TBD
Polar Aprotic Organics as C (ppb) [42] [43]	TBD	TBD	TBD	TBD	TBD	TBD
Bacteria (CFU/liter) [38]	<1	<1	<1	<1	<1	<1
Total silica (ppb) as SiO_2 [18]	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3
Colloidal Silica (ppb) s SiO2 (add note)	TBD	TBD	TBD	TBD	TBD	TBD
Number of particles >critical particle size (see above) (#/L) [26]	4000	4000	4000	4000	4000	4000
Dissolved oxygen (ppb) (contaminant based) [16]	<10	<10	<10	<10	<10	<10
Dissolved nitrogen (ppm) [10]	8–18	8–18	8–18	8–18	8–18	8–18
Metals (ppt each) (Co, Cr,Ga,Ge,Mn,Mo,Sr,Ti,)	<10	<10	<10	<10	<10	<10
Critical metals (ppt, each) (Ag,Al,Au,Ba,Ca,Cu,Fe,Hf,K,Li,Mg,Na,Ni,Pt,Zn)	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Other critical ions (ppt each) [24]	<50	<50	<50	<50	<50	<50

Voltaire's *Dictionnaire Philosophique* (1764)

“The pursuit for perfection becomes the enemy of good enough”

Conclusions:

- The Task Force has made significant progress in just 12 months
- The boundary conditions for testing nanometer pore size filters with a 5-15nm challenge have been established
- A draft filter test protocol has been written
- A filter test skid has been built at Pall Corporation and the first filters tested
- A draft of the test method has been written and will be submitted for SEMI ballot this month.