Evaluation of Particle Shedding and Trace Metal Extraction from Centrifugal Pumps

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Introduction

- Semiconductor devices are extremely sensitive to particulate and metal contamination.
- As feature sizes continue to decrease, the need for purity continues to increase.
- High purity pumps are used in many semiconductor processes:
  - bulk chemical delivery
  - ultra pure water systems
  - recirculating etch baths
  - and other high purity process applications
- Particulate contamination can:
  - directly reduce product yield
  - degrade the performance and lifetime of filters
- Metallic contamination can also cause a variety of issues that affect device yield and reliability.

Introduction

Pump Manufacturer	Pump Type	Max Pressure (bars)	Max Flow Rate (L/min)
Levitronix BPS-4000	maglev centrifugal	6.3	280
MagDrive 1 (MD1)	magnetic drive centrifugal	5.4	265
MagDrive 2 (MD2)	magnetic drive centrifugal	5.0	662

Test Procedure – Particle Shedding Tests

- Operating conditions (outlet pressure and flow rate) were chosen such that all pumps were evaluated at similar conditions.
- Particle counters used:
  - PMS HSLIS M50: 4 size channels from ≥ 0.05 to ≥ 0.2 µm
  - PMS LiQuilaz-S05: 15 size channels from ≥ 0.5 to ≥ 20 µm

Flow rate (gpm)	Outlet Pressure (psig)
3	50
5	50
10	50
20	50
50	50
3	70
5	70
10	70
20	70
45	70
3	90
5	90
10	90
20	90
35	90

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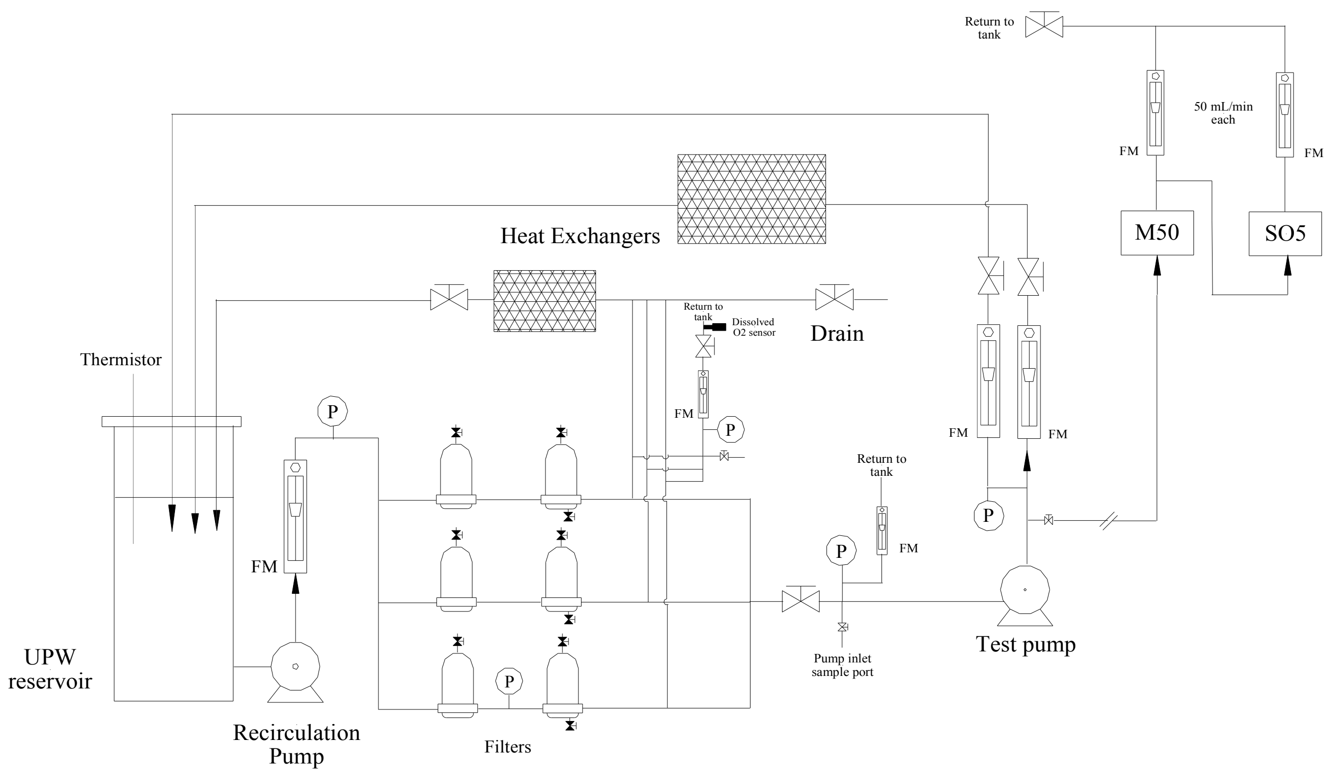
Schematic of particle shedding test system



How many 20 nm particles are needed in 1 liter of water to achieve a contamination level of 1 ppt???

- ~250,000,000
  - Calculations assume that the particle density is 1.0 gram/cm^3

- 2013 UPW purity requirement from ITRS Roadmap:
  - < 4,000/liter $\geq$ 20 nm (or < 0.02 ppq!!!)

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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

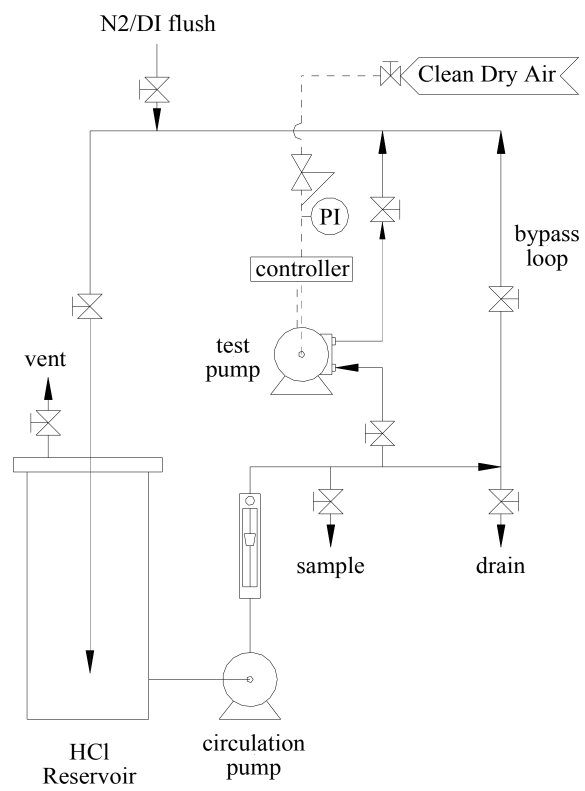
Problems associated with optical particle counters

- Sensitivity:
  - Current technology: 30 nm particle detection
- Low inspection flow rates
  - PMS HSLIS M50: 0.25 ml/min
  - PMS Ultra DI 50: 3.75 ml/min
- False counts
  - Cosmic rays, electronic noise, stray scattering, molecular scattering, etc.
- Gas bubbles
- Index contrast
- Particle coincidence

Sources of metallic contamination

- Component raw material
  - Often distributed throughout the bulk of material and associated with raw resin
- Manufacturing tool, assembly or packaging
  - Often present as a surface and/or near surface contaminant
  - Importance of surface contamination depends on component use
- Component failure
  - Occurs when a non-wetted material (often protected by a polymer coating fails) is exposed to an incompatible chemical.

Schematic of trace metal extraction system



Trace metal extraction tests

- A 10 day dynamic extraction test in 35% HCl was used.
  - Test pumps installed in a pre-extracted test system
  - Test pumps operated continuously during test
- Samples were withdrawn during the test and analyzed for 38 metallic elements.
- Measure both surface and bulk contamination.
- Rate of trace metal extraction over time was calculated for each pump.

Advantages of dynamic extraction test

- Sample contamination minimized since multiple samples taken and contaminant concentrations build up over time.
- Sensitive to detect very low levels of extraction since sample volume minimized and multiple samples taken.
- Chemical flowing during test may result in higher extraction rates than during stagnant tests.
- Can differentiate between surface and bulk contamination and predict mass extraction rates.
- Extraction data can be used to predict a component's contribution to process chemical stream.

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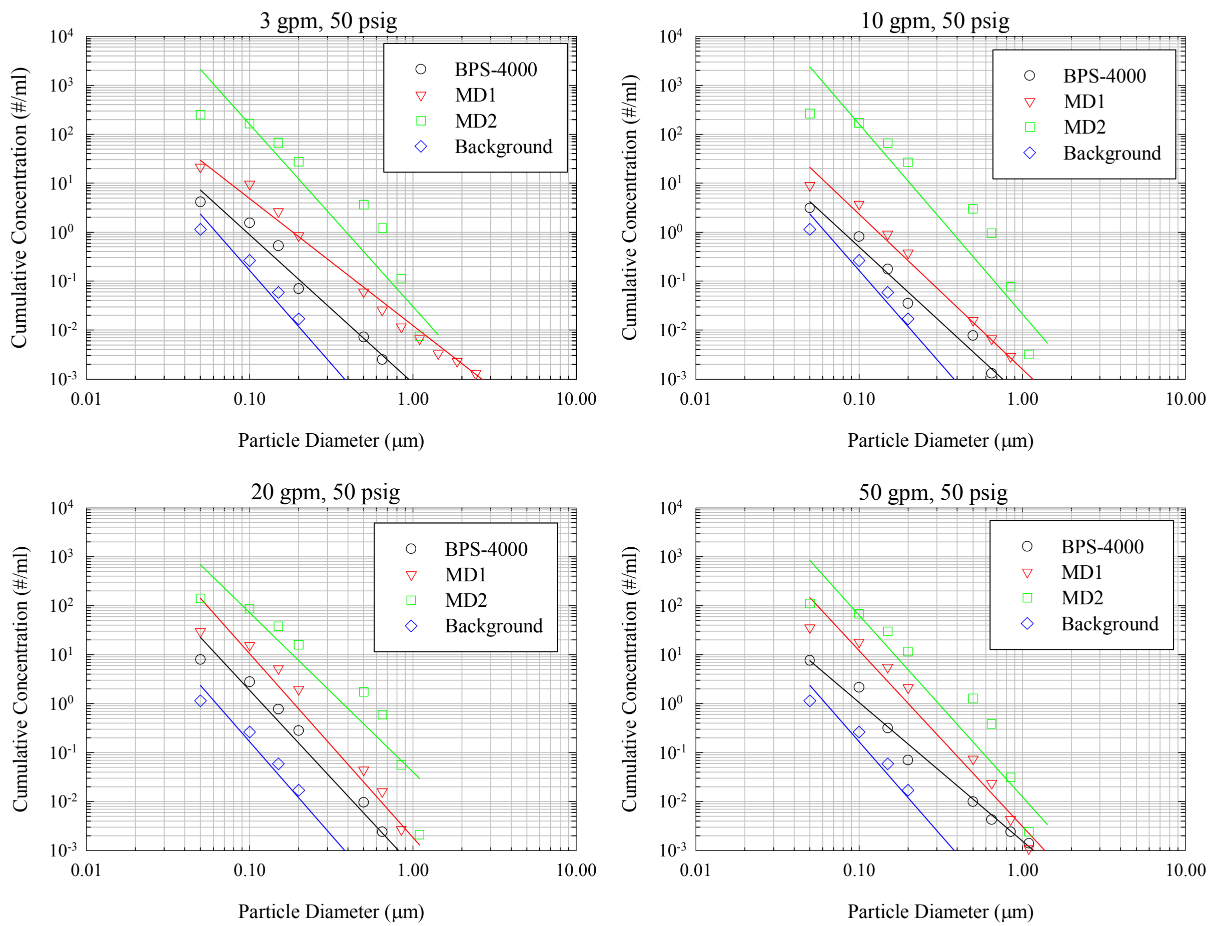
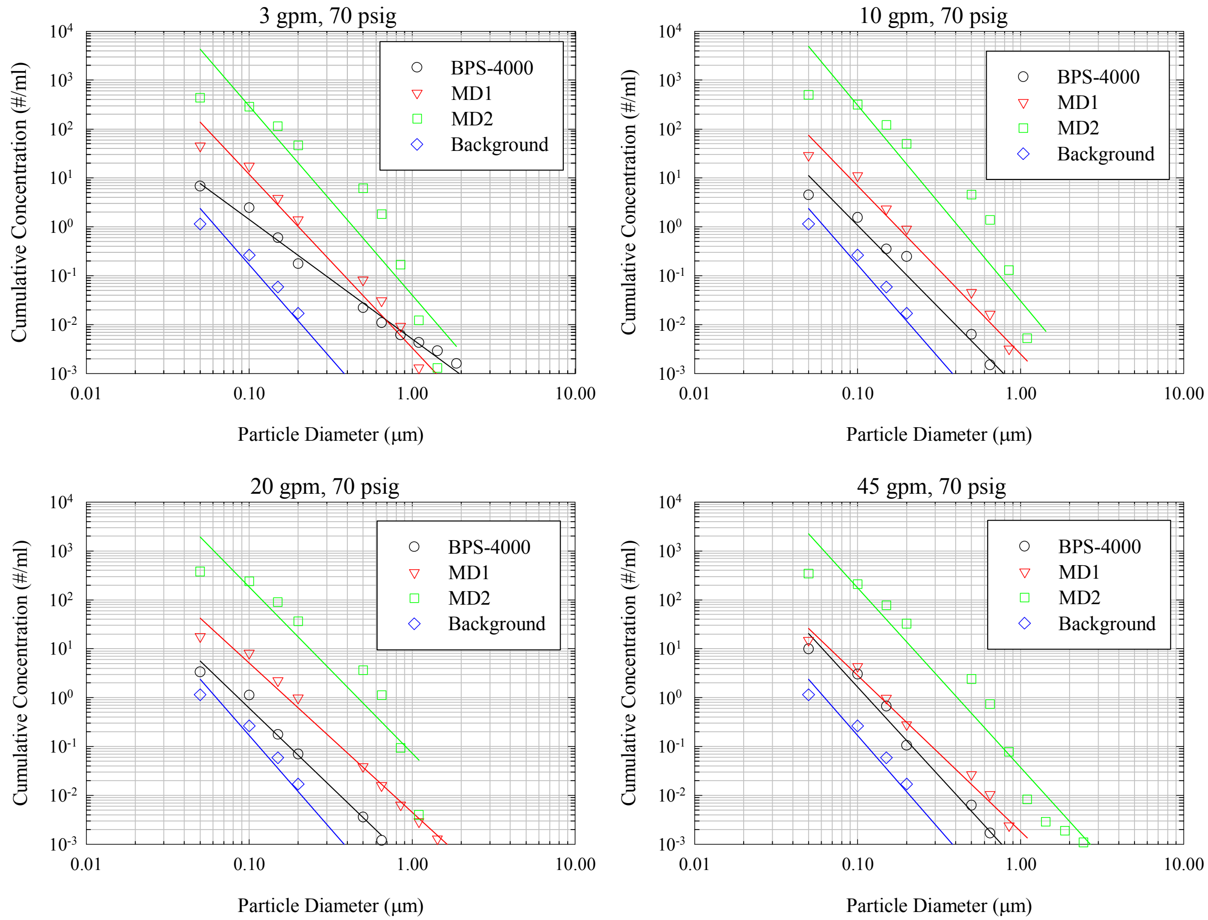
Results

Particle Shedding

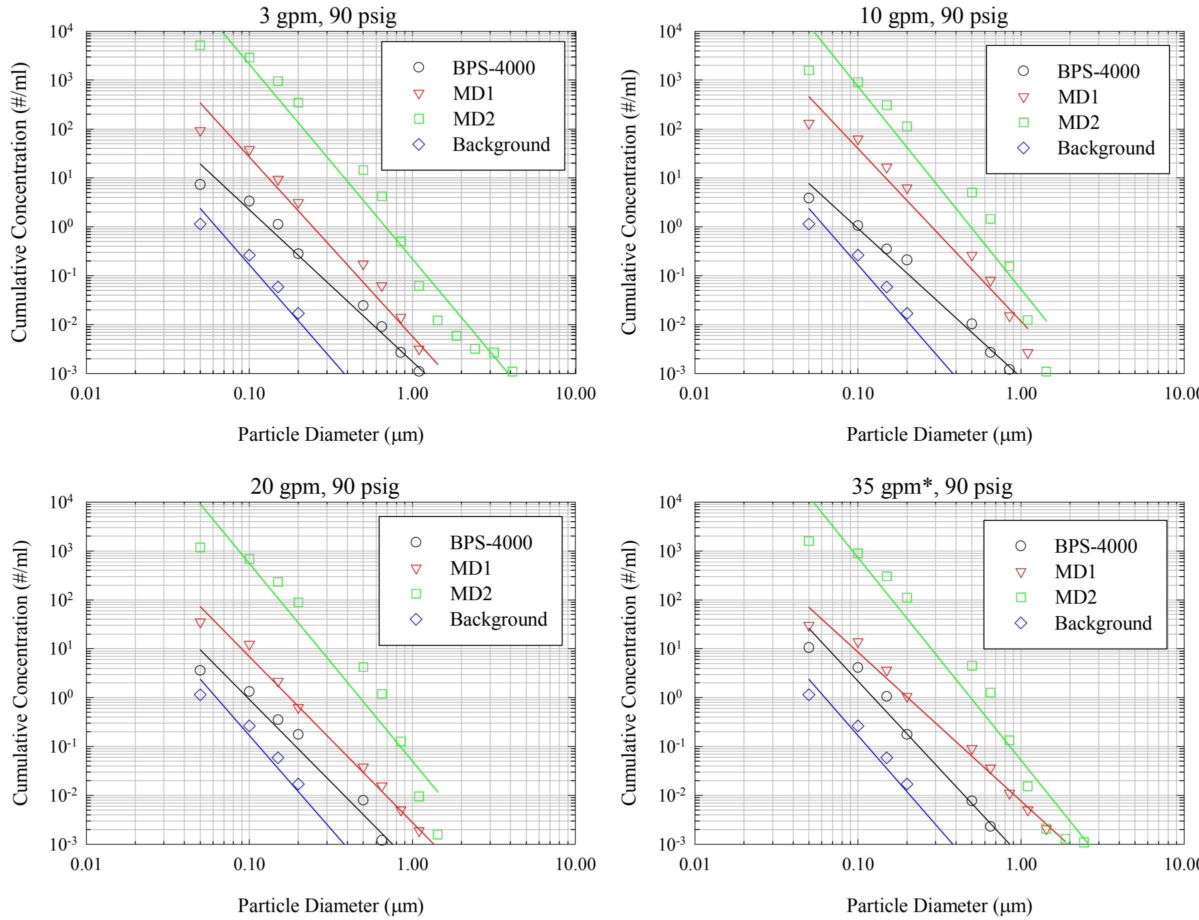
PSDs @ 50 psig (3.4 bars)



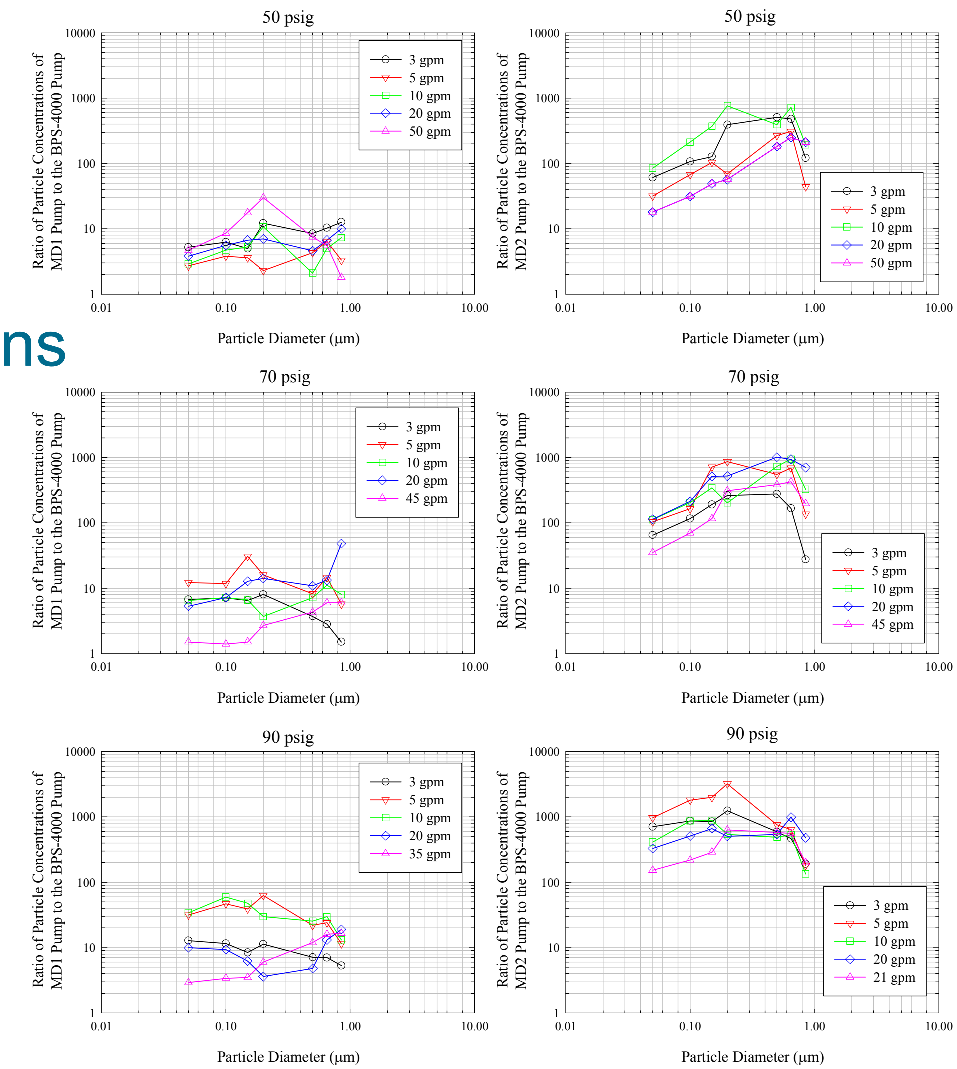
PSDs @ 70 psig (4.8 bars)



PSDs @ 90 psig (6.2 bars)



Particle Concentrations Relative to BPS-4000



Comparison of pump mean concentration ratios at all 15 test conditions

Pump	Geometric Mean of the Concentration Ratios of the Following Pumps to the Levitronix Pump	
	MD1	MD2
≥ 0.05 µm	6.4	100
≥ 0.l0 µm	8.0	190
≥ 0.l5 µm	8.5	310
≥ 0.20 µm	9.6	400
≥ 0.50 µm	7.1	430
≥ 0.65 µm	9.5	460
≥ 0.85 µm	7.9	140

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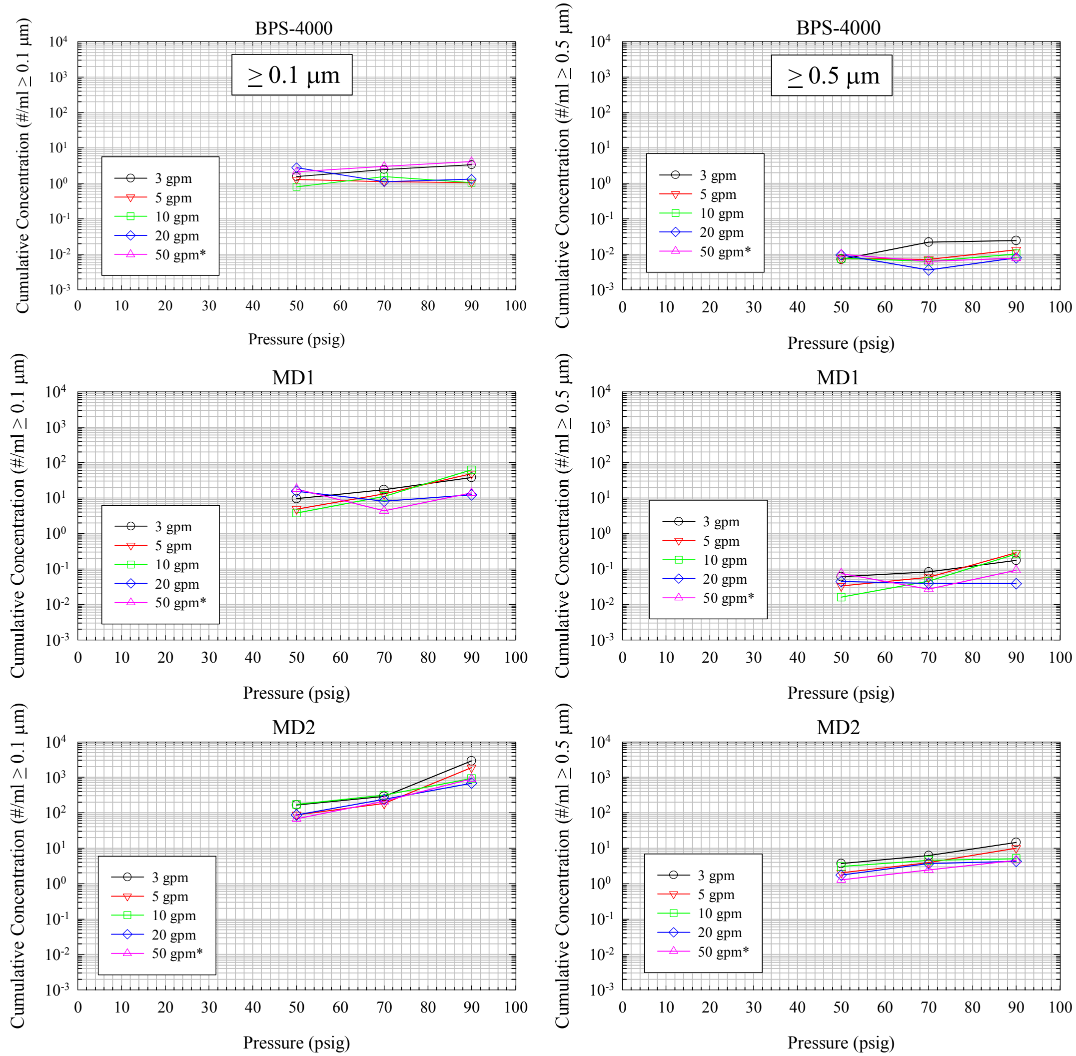
Effect of Operating Conditions



Summary of effect of operating conditions

Pump	Increasing Pressure		Increasing Flow Rate	
	Small Particles	Large Particles	Small Particles	Large Particles
Levitronix	0	0	0	0
MD1	+	+	0	0
MD2	++	+	0	0

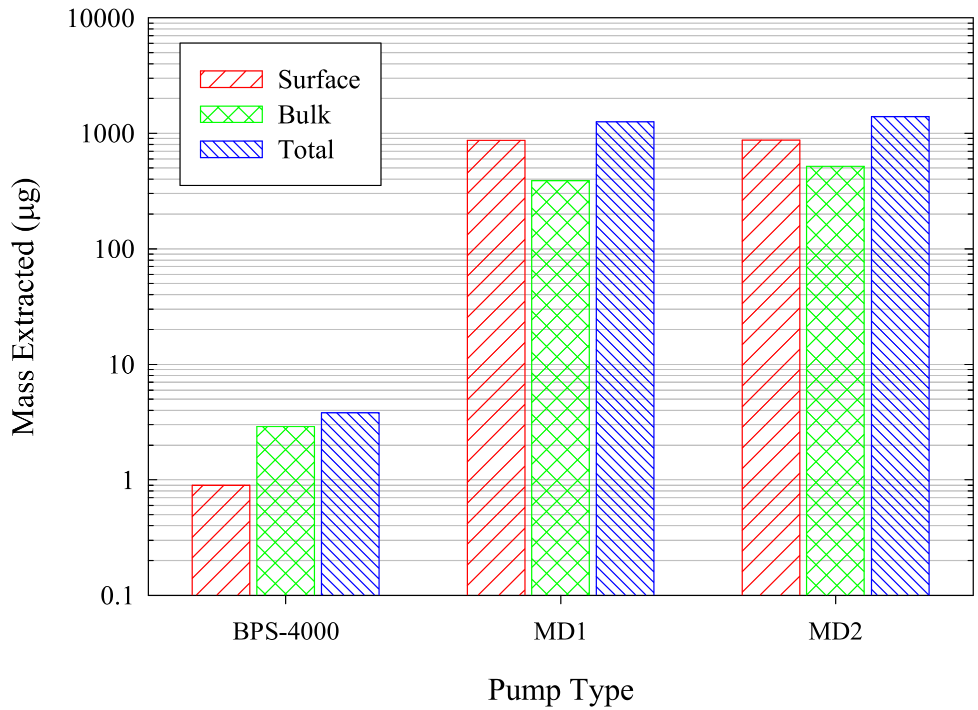
Key:

Concentration Change	Concentration Change due to Increasing Pressure (%/psig)	Symbol	Concentration Change due to Increasing Flow Rate
None	0-5	0	< a factor of 2 increase
Small Increase	5-20	+	> a factor of 2 increase
Large Increase	>20	++	NA

Results

Trace metal extraction

Surface, bulk, and total mass extracted from each pump



Surface Contamination

BPS-4000			MD1			MD2		
Element	Mass Extracted (ug)	% of Total (%)	Element	Mass Extracted (ug)	% of Total (%)	Element	Mass Extracted (ug)	% of Total (%)
Fe	0.35	39.8	Na	371.5	42.7	Fe	534.4	61.0
Al	0.17	19.3	Fe	234.9	27.0	Bi	59.6	6.8
Ca	0.15	17.0	K	97.5	11.2	Ca	45.8	5.2
Cu	0.07	8.0	Ca	52.1	6.0	Cu	40.9	4.7
Na	0.07	8.0	Zn	20.8	2.4	Al	39.4	4.5
Mg	0.05	5.7	Mg	15.7	1.8	Sn	23.9	2.7
Misc.	0.02	2.3	Ti	14.7	1.7	Zn	23.9	2.7
			Cu	12.8	1.5	Ni	23.8	2.7
			Misc.	49.3	5.7	Misc.	83.7	9.6
Total	0.88	100.0	Total	869.3	100.0	Total	875.4	100.0

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Elements with Total Contamination ≥ 1 μg

BPS-4000		MD1		MD2	
Element	Mass Extracted (μg)	Element	Mass Extracted (μg)	Element	Mass Extracted (μg)
Fe	1.1	Na	588	Fe	600
		Fe	235	Bi	223
		K	150	Sn	86
		Ca	56	Al	79
		Ti	52	Ca	69
		B	39	Cu	55
		Mg	26	Mg	51
		Zn	21	Ti	47
		Al	18	Ni	30
		Cu	18	Zn	28
		V	14	Cr	25
		Ba	10	B	24
		Cr	6.8	V	19
		Zr	6.8	Ba	16
		Ni	5.1	Na	12
		Mn	2.6	K	7.0
		Sn	2.1	Zr	5.2
		Pb	1.3	Pb	4.2
		Ga	1.1	Cd	2.0
		Co	1.0	In	1.1
				W	1.1
				Ga	1.1
				Mo	1.1

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Summary of surface and bulk extraction

- Compared to other high purity components tested in past, the metallic extraction from:
  - Levitronix Maglev pump considered low
  - MagDrive centrifugal pumps considered high
- Iron is often the largest contaminant found in these tests.
  - Iron was either 1^{st} or 2^{nd} highest contaminant in each test.
- Neodymium magnets ($Nd_2Fe_{14}B$) are used in these Maglev and MagDrive pumps.
  - The total mass of Boron extracted from MagDrive pumps 1 and 2 was more than 100 times higher than found in any of the Levitronix pumps.
  - Very low levels of neodymium were also present as a surface contaminant with both MagDrive pumps tested.

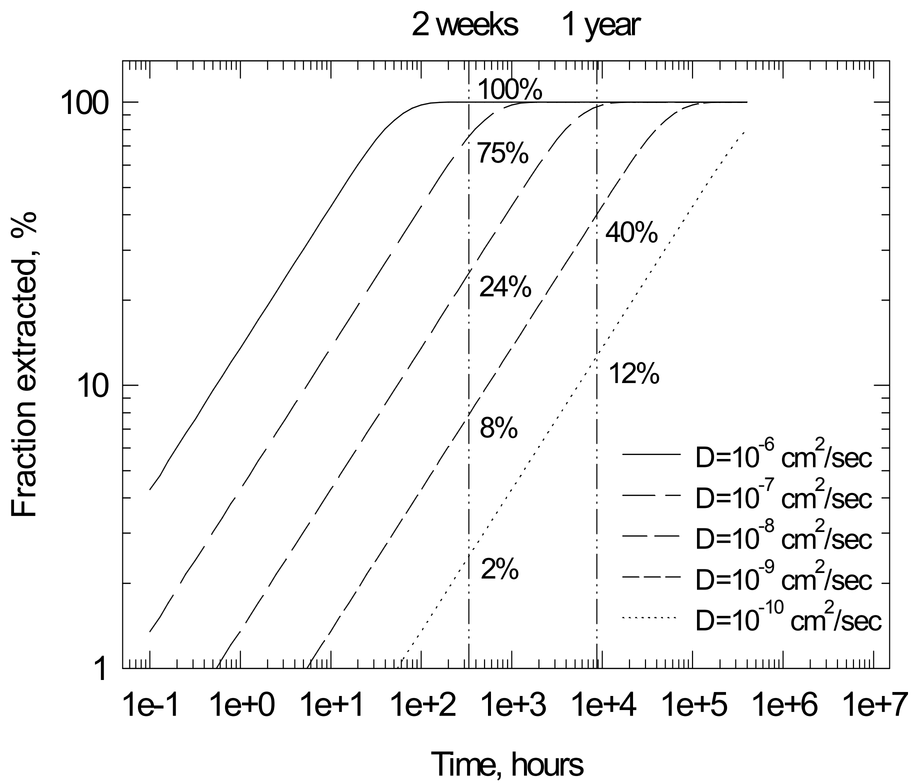
Diffusion Theory

$$\frac{M_t}{M_\infty} = 1 - \sum_{n=0}^{\infty} \frac{8}{(2n+1)^2 \pi^2} \exp\left[-D(2n+1)^2 \pi^2 t / 4l^2\right]$$

- where:
  - M_t = Mass extracted at time t
  - M = Total extractable mass
  - D = Diffusion coefficient of the contaminant in the bulk material
  - t = Time
  - l = Plane thickness
- Diffusion coefficients in solids not well known and hard to measure.
- Diffusion coefficients in solids range from 10^{-7} to 10^{-20} cm^2/sec.

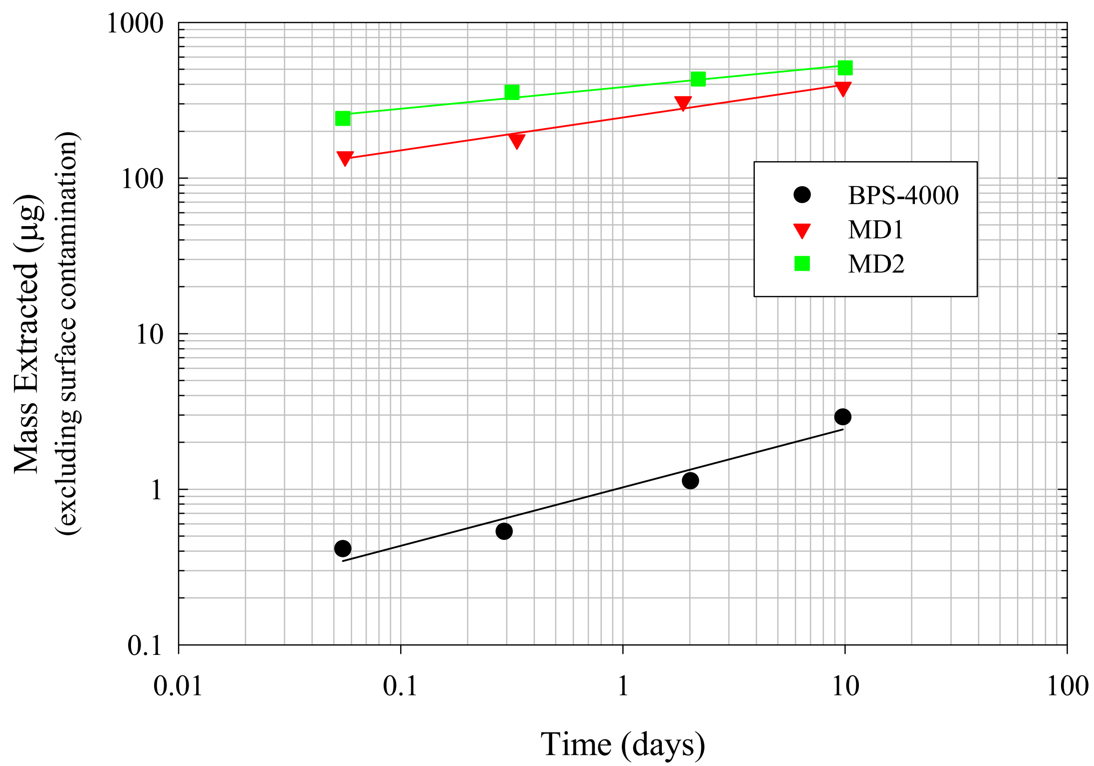
Lemke T and DC Grant (1995). "Dynamic Extraction: A New Technique for Measuring Metallic Extractables from Chemical Delivery System Components," Proceedings of the 13th Annual Technical Symposium: Partnering for Contamination-Free Manufacturing, Millipore Corporation, Bedford, MA.

Theoretical extraction from 0.5 cm thick plane with uniform initial contaminant concentration



Lemke T and DC Grant (1995). "Dynamic Extraction: A New Technique for Measuring Metallic Extractables from Chemical Delivery System Components," Proceedings of the 13th Annual Technical Symposium: Partnering for Contamination-Free Manufacturing, Millipore Corporation, Bedford, MA.

Extraction from the bulk material



Model used

- $M=k*t^n$
  - t = time (days)
  - M = cumulative mass extracted (µg) at time t
  - k = proportionality constant
  - n = exponent
- Taking the log, we get:
  - log(M) = log(k) + n*log(t)
- Values for k and n may be obtained from the linear regression of the curves of mass extracted vs. time
- These values can be used to calculate the rate of extraction at any time
  - Rate of extraction = dM/dt = $nk(t)^{n-1}$
- Extraction from a component will likely occur throughout its life, but the rate of extraction will decrease with time.

Pump	k	n
Levitronix BPS-4000	1.03	0.38
MagDrive 1	245	0.21
MagDrive 2	384	0.14

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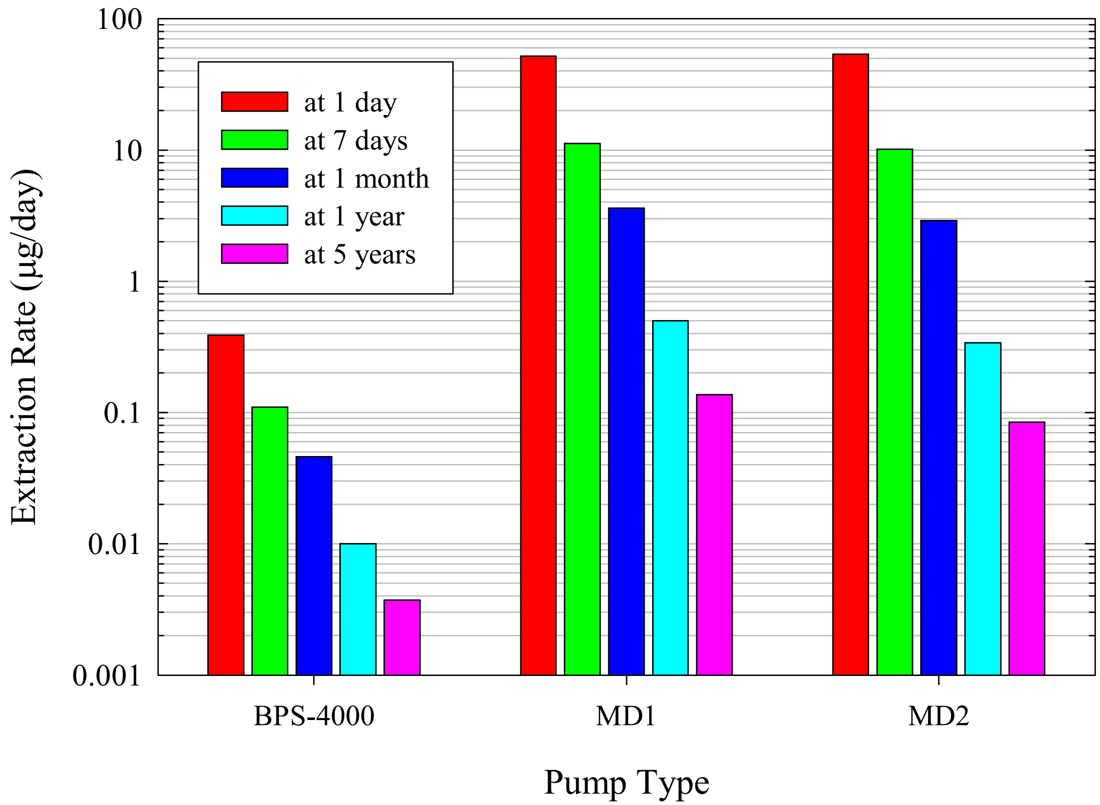
Mass Extraction Rates



Summary of particle shedding results

- BPS-4000 pump shed the fewest particles of the pumps evaluated in this study, regardless of pump operating conditions.
  - In many cases, the particle concentrations measured downstream of the BPS-4000 were very close to the concentrations (within a factor of five) measured without a test pump in the system.
  - MD1 typically shed 5-10 times as many particles as the BPS-4000.
  - MD2 typically shed 100-400 times as many particles as the BPS-4000.
- Operating conditions had small effects on particle concentrations.
  - Particle shedding typically increased as outlet pressure increased for both the MD1 and MD2 pumps. No effect of outlet pressure was observed for the BPS-4000.
  - Flow rate appeared to have little effect regardless of pump type or outlet pressure over the range of flow rates evaluated in this study.

Summary of trace metal extraction results

- Surface contamination measured from MD1 and MD2 was ~1000X higher than BPS-4000
- Bulk contamination measured from MD1 and MD2 was >100X higher than BPS-4000
- Rates of mass extraction @ 7days from the MagDrive pumps were on the order of 100 times higher than BPS-4000
- It would take on the order of 5 years for the rate of mass extraction of the MagDrive pumps to reach the level of the BPS-4000 @ 7 days