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Trace Metal Dynamic Extraction from Levitronix Pump System BPS-3 in Hydrochloric Acid

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Summary

The objective of this test was to determine the type and rate of trace metals extraction from a Levitronix BPS-3 pump system using BOC Edwards' patented DyconE^{x SM} dynamic extraction method in 34% hydrochloric (HCl) acid during a 10-day extraction test. Both surface contamination and bulk contamination can be determined with this method. Surface contamination is defined as the amount of contamination extracted in the first 40 minutes of exposure of the component to chemical. Bulk contamination is defined as the amount of contamination extracted after 40 minutes of exposure to chemical.

The pump was plumbed into the dynamic extraction test apparatus shown in Figure 1. High purity 34% HCl was used as the extractant. Continuous flow of the chemical was maintained through the pump during the test. A background sample was taken from the test system prior to the test and samples were taken at approximately evenly spaced time intervals on a logarithmic scale from a sample port located in the circulation loop. The chemical samples were submitted for analysis of 37 metallic elements. The results of the

analyses were converted to cumulative mass extracted and normalized for the wetted surface area of the pump. The wetted parts of the BPS-3 pump system are the CP-3 pump head consisting of a PVDF pump housing and an ECTFE impeller.

The area normalized surface contamination extracted from all elements was 2.5 ng/cm^2 . These results were well below a major semiconductor equipment manufacturer's surface contamination specification of $< 20 \text{ ng/cm}^2$. Iron accounted for more than 50% of the surface contamination measured in this test. The area normalized mass extraction rate was calculated to be $0.18 \text{ ng/cm}^2/\text{day}$ at 7 days. This extraction rate is about a one-third of a major semiconductor equipment manufacturer's specification of $< 0.5 \text{ ng/cm}^2/\text{day}$ at 7 days. Iron contributed about one-third of the bulk contaminants extracted during the test. A variety of other elements contributed the remainder of the bulk contamination including: potassium, sodium, calcium, aluminium, magnesium, zinc, and boron.

Test System

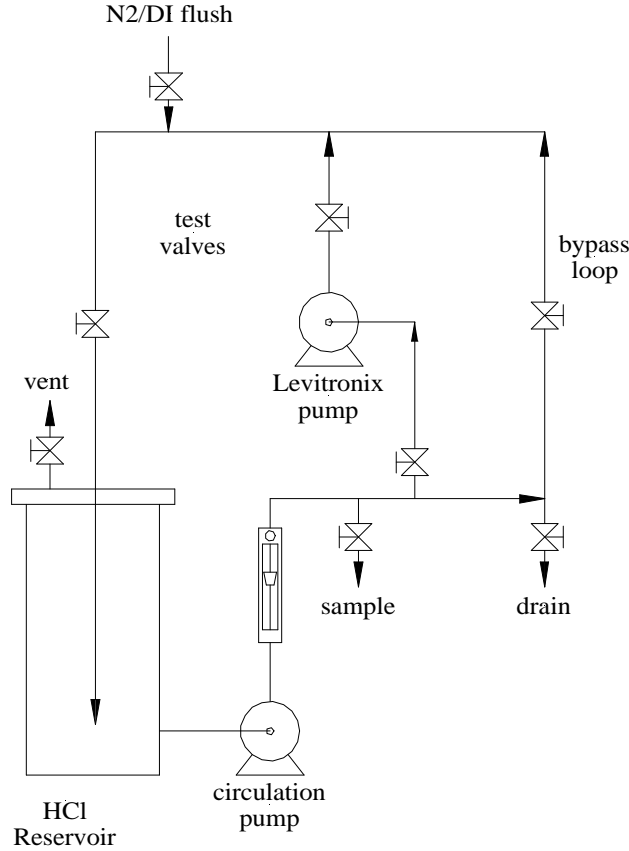
The DyconE^{x SM} dynamic extraction test system is shown in Figure 1. All of the wetted system components are made of fluoropolymers and were preconditioned for a minimum of 6 weeks in 34% HCl. The test system was located in a class 100 cleanroom. Prior to testing, the system shown was cleaned with a 10% hydrochloric acid solution, with a spool piece in place of the test pump. The spool piece allowed the system to be cleaned with all of the plumbing pieces required for the test, without having the test pump in the system. The spool piece simply consisted of a fluoropolymer union. The purpose of cleaning the system was to remove any contamination remaining in the system from the previous test, and to clean the component(s) that were installed to connect to the test pump. The pump was connected to the test system with two PFA female adapters (1" FNPT x $\frac{3}{4}$ " Flaretek) that had been preconditioned in HCl for at least 6 weeks. After cleaning, the system was flushed with ultrapure water and drained completely three times.

Test Procedure

After the system was cleaned, the test pump was installed into the system, but was isolated by closing isolation valves located on both sides of the test pump. A known volume of 34% Baseline Grade Seastar hydrochloric acid was added to the reservoir. To maximize the sensitivity of the test, the volume of hydrochloric acid was minimized. The initial volume included enough chemical to fill the system plumbing and test pump plus an additional 750 mL for chemical samples. A background sample of this chemical was taken just prior to initiating circulation of the chemical through the test pump. Five additional samples of chemical were removed from the system during 12 days of circulation through the pump. During the test, the Levitronix pump was energized to levitate the impeller, but was not operating. The sample times were approximately evenly spaced on a logarithmic scale (see Table I) to improve the accuracy of the data analysis. After the 12-day sample was taken, the samples were submitted as a group to Seastar Chemicals, Inc. (Sidney, BC, Canada) for the trace metals analysis using ICP-MS (inductively coupled plasma-mass spectroscopy) and GFAAS (graphite furnace atomic

absorption spectroscopy). Finally, the system was flushed with water and the pump was removed.

Figure 1. Schematic of Dynamic Extraction Test System



Miscellaneous Test Specifics

Specifics to this test are shown in Table I.

Table I. Test specifics

Test Conditions	
Component(s) Tested	Levitronix BPS-3 pump system
Component Surface Area	355 cm ² (internal wetted surface area)
Chemical Manufacturer and Grade	Seastar Chemical Company – Baseline Grade
Chemical Volume	1.19 liters
Circulation Rate	~ 0.7 liters/min
Temperature	16 – 20 C
Sample Times	Background, 40 minutes, 2 hours, 8 hours, 2 days, 12 days

Since the present specifications for metallic extractables are normalized by the wetted surface area of the component, the internal wetted surface area of the pump was required.

The internal wetted surface area of the pump was estimated to be about 355 cm². This wetted surface area estimate was obtained from Levitronix.

Results

Surface contamination

The values for the cumulative mass extracted for the 37 metallic constituents at all sample times were normalized for the wetted internal surface area of the pump. The raw data are found in Appendix A. Based on these data, the primary contributors to surface contamination (as defined as the amount of mass extracted during the first 40 minutes of the test) were iron and calcium. The area normalized surface contamination from all elements extracted from the pump was 2.5 ng/cm² as shown in Table II, which is well below a major semiconductor equipment manufacturer's specification of < 20 ng/cm².

Table II. Surface contamination (ng/cm²) measured from test

Surface Contamination		
Element	Normalized Mass Extracted (ng/cm ²)	Percentage of Total (%)
Fe	1.44	57.6
B	0.24	9.6
Ca	0.18	7.2
K	0.12	4.8
Na	0.12	4.8
Al	0.09	3.6
Zn	0.09	3.6
Misc.	0.22	8.8
Total	2.50	100.0

Bulk contamination

Figure 2 shows the mass of selected elements extracted from the bulk material of the pump over time after normalizing for the wetted surface area of the pump. The data are plotted on a log-log scale as a function of time. Figure 3 shows the normalized total mass extracted from all measured elements over time. Figures 2 and 3 exclude surface contamination.

Iron contributed about one-third of the bulk contamination (as defined as the amount of mass extracted after the first 40 minutes of the test) extracted during the test. Other significant contributors were potassium, sodium, calcium, aluminium, and magnesium. The total area normalized bulk contamination from all elements extracted during the test was 5.6 ng/cm².

Data for the normalized mass extracted from the bulk material of the pump over time for all measured elements are presented in Figure 3 on a log-log plot. Since theory predicts that the extraction data can be fit by a power law equation, the data should follow a

straight line when plotted in this format [1]. A simple regression analysis of the data in this format is also presented in Figure 3.

Figure 2. Surface area normalized mass extracted for the primary contributing elements from the pump (excluding surface contamination)

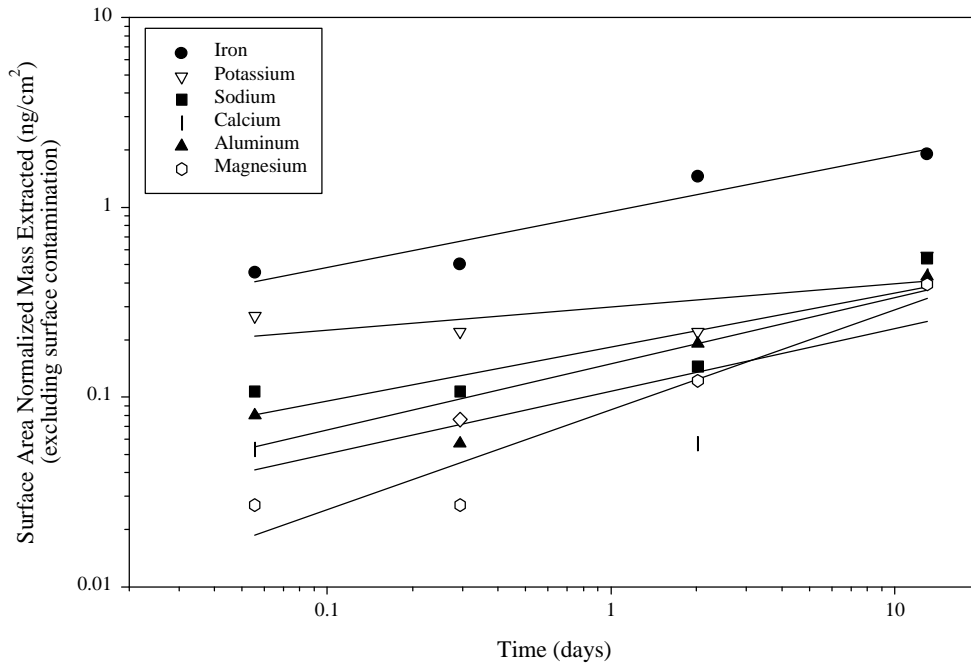
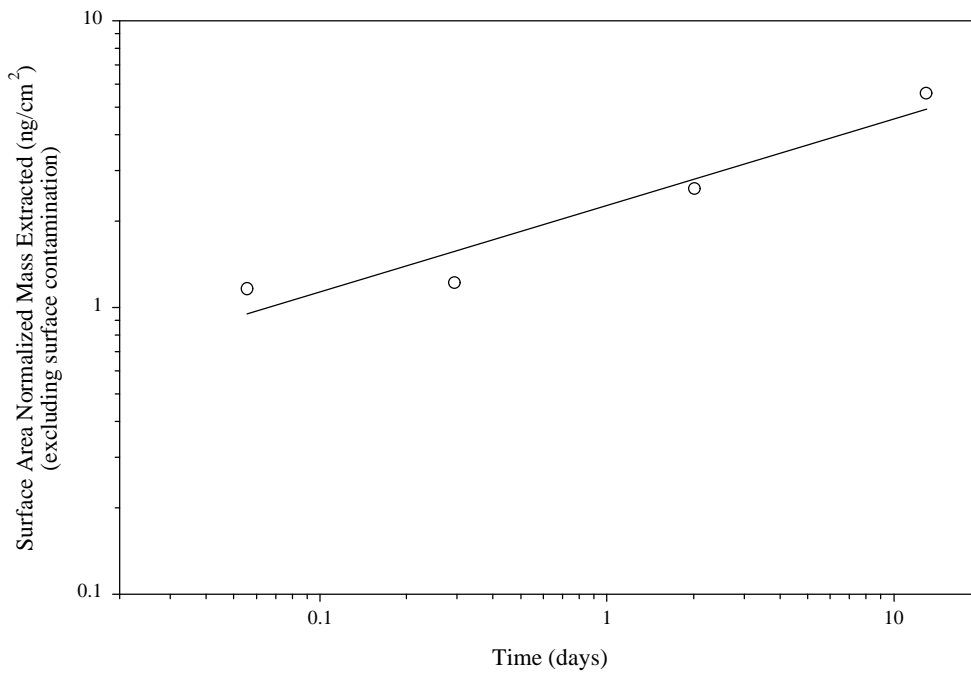


Figure 3. Total surface area normalized mass extracted for all elements from the pump (excluding surface contamination)



The mathematical model used to predict the cumulative mass extracted as a function of time is an equation of the type:

$$m = k * t^n \tag{1}$$

where:

- t = time (days)
- m = normalized cumulative mass extracted (ng/cm²) at time t
- k = proportionality constant
- n = exponent

Taking the log of both sides of the equation gives:

$$\log(m) = \log(k) + n * \log(t) \tag{2}$$

The parameters in equation (1) for total metals are given in Table III. These values were obtained from the linear regression analysis illustrated in Figure 3, where n is the slope and log k is the intercept as shown in equation (2).

Table III. Mass extraction parameters

Component	k	n
Levitronix pump	0.356	0.302

The values for constants k and n can be used to calculate the total mass extracted from the pump at different times using equation (1). In addition, the rate of extraction can be determined by using the derivative of equation (1) with respect to time:

$$\text{Rate of extraction} = dm/dt = n * k * t^{n-1} \tag{3}$$

Based on equation (3) and the calculated curve fit parameters listed in Table III, the rates of extraction for total metals at 1, 7, and 14 days are shown in Table IV.

Table IV. Calculated rates of extraction for 37 metallic elements

Component	Extraction Rate (ng/cm ² /day)		
	1 day	7 days	14 days
Levitronix pump	0.69	0.18	0.11

The area normalized mass extraction rate for the pump was calculated to be 0.18 ng/cm²/day at 7 days. This extraction rate is about one-third of a major semiconductor equipment manufacturer's specification of < 0.5 ng/cm²/day at 7 days.

Conclusions

A Levitronix BPS-3 pump system was tested for trace metal extraction using BOC Edwards' patented DyconE^{x SM} dynamic extraction method in 34% HCl. Iron accounted for more than 40% of the trace metals extracted during this test. Several other elements contributed at least 5% of the total trace metals including: potassium, sodium, calcium, boron, aluminium, and magnesium. The area normalized surface contamination from all elements extracted was 2.5 ng/cm², which was well below the surface contamination specification of a major semiconductor equipment manufacturer. The metallic extraction rate for the pump was 0.18 ng/cm²/day at 7 days. This extraction rate was about one-third of a major equipment manufacturer's specification of < 0.5 ng/cm²/day at 7 days.

References

Grant D.C., T. Lemke, G. Duepner, D. Wilkes, and N. Powell (1996). "Measurement of Inorganic Contaminant Extraction from Fluid Handling Components by Dynamic Extraction," *Journal of the Institute of Environmental Sciences* 39(2):29-37.

Acknowledgement

DyconE^{x SM} is a procedure patented by BOC Edwards (U.S. Patent No. 5,641,895). BOC Edwards has granted CT Associates, Inc. a license for commercial use of this procedure.

Appendix A

Dynamic Extraction Study - Test Results and Data Analysis																			
Test Material:			Levitronix pump					Serial Numbers:											
Model No:			BPS-3 pump system (CP-3 pump head)																
Wetted Area (cm2):			354.7																
Leachate Type:			35% HCl																
Initial Fill Volume:			1.190 liters																
Sample+Rinse Volume 0:			0.123 liters																
Sample+Rinse Volume 1:			0.119 liters																
Sample+Rinse Volume 2:			0.136 liters																
Sample+Rinse Volume 3:			0.135 liters																
Sample+Rinse Volume 4:			0.143 liters																
Date	08/27/02		08/27/02		08/27/02		08/27/02		08/29/02		09/09/02								
Time	7:55		8:35		9:55		15:38		9:05		8:20		Cumulative Metal Extraction (ng/cm2)						
Elapsed Time (hours)	Detection Limit	Control	0.00		1.33		7.05		48.50		311.75		Day>>>		0.056	0.294	2.021	12.990	
			Concentration (ppb)					Surface	% of Surface	Extracted					% of Bulk	Total	% of Total		
Ag	0.01	0.01	0.01	0.01	0.01	0.01	0.01	Ag	0.00	0.0%	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al	0.01	0.02	0.05	0.08	0.07	0.14	0.3	Al	0.09	3.6%	0.08	0.06	0.19	0.43	7.7%	0.52	6.5%		
As	0.05	0.05	0.05	0.05	0.05	0.05	0.05	As	0.00	0.0%	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Au	0.1	0.1	0.1	0.1	0.1	0.1	0.1	Au	0.00	0.0%	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
B	0.05	0.07	0.15	0.16	0.18	0.18	0.33	B	0.24	9.6%	0.03	0.07	0.07	0.30	5.3%	0.54	6.7%		
Ba	0.001	0.006	0.01	0.011	0.012	0.016	0.028	Ba	0.01	0.5%	0.00	0.00	0.01	0.03	0.5%	0.04	0.5%		
Be	0.01	0.01	0.01	0.01	0.01	0.01	0.01	Be	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
Bi	0.001	0.001	0.001	0.001	0.001	0.001	0.001	Bi	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
Ca*	0.02	0.04	0.1	0.12	0.13	0.12	0.37	Ca*	0.18	7.2%	0.05	0.08	0.06	0.43	7.8%	0.61	7.6%		
Cd	0.001	0.001	0.001	0.001	0.001	0.001	0.003	Cd	0.00	0.0%	0.00	0.00	0.00	0.00	0.1%	0.00	0.0%		
Co	0.001	0.002	0.004	0.001	0.004	0.007	0.013	Co	0.01	0.2%	-0.01	0.00	0.00	0.01	0.2%	0.02	0.2%		
Cr	0.01	0.02	0.04	0.04	0.04	0.08	0.12	Cr	0.06	2.4%	0.00	0.00	0.08	0.14	2.4%	0.20	2.4%		
Cs	0.001	0.001	0.001	0.001	0.001	0.001	0.001	Cs	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
Cu	0.01	0.01	0.02	0.03	0.04	0.05	0.08	Cu	0.03	1.2%	0.03	0.05	0.07	0.11	2.0%	0.14	1.8%		
Fe	0.03	0.03	0.51	0.68	0.7	1.2	1.5	Fe	1.44	57.8%	0.45	0.50	1.45	1.91	34.1%	3.35	41.4%		
Ga	0.001	0.001	0.001	0.001	0.001	0.001	0.001	Ga	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
In	0.001	0.001	0.001	0.001	0.001	0.001	0.001	In	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
K*	0.02	0.1	0.14	0.24	0.22	0.22	0.44	K*	0.12	4.8%	0.27	0.22	0.22	0.55	9.9%	0.67	8.3%		
Li	0.001	0.001	0.001	0.001	0.001	0.001	0.001	Li	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
Mg	0.005	0.005	0.01	0.02	0.02	0.07	0.25	Mg	0.02	0.6%	0.03	0.03	0.12	0.39	7.0%	0.41	5.0%		
Mn	0.001	0.001	0.002	0.003	0.003	0.003	0.007	Mn	0.00	0.1%	0.00	0.00	0.00	0.01	0.2%	0.01	0.1%		
Mo	0.001	0.002	0.003	0.003	0.004	0.005	0.04	Mo	0.00	0.1%	0.00	0.00	0.00	0.06	1.0%	0.06	0.7%		
Na	0.01	0.05	0.09	0.13	0.13	0.15	0.41	Na	0.12	4.8%	0.11	0.11	0.15	0.54	9.6%	0.66	8.1%		
Nb	0.001	0.001	0.001	0.001	0.001	0.001	0.001	Nb	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
Ni	0.01	0.04	0.06	0.06	0.08	0.09	0.15	Ni	0.06	2.4%	0.00	0.05	0.06	0.16	2.8%	0.22	2.7%		
Pb	0.001	0.001	0.005	0.006	0.007	0.011	0.019	Pb	0.01	0.5%	0.00	0.00	0.01	0.02	0.4%	0.04	0.5%		
Rb	0.001	0.001	0.001	0.001	0.001	0.001	0.001	Rb	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
Re	0.001	0.001	0.001	0.001	0.001	0.001	0.001	Re	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
Sn	0.01	0.01	0.01	0.02	0.02	0.03	0.08	Sn	0.00	0.0%	0.03	0.03	0.05	0.12	2.2%	0.12	1.5%		
Sr	0.001	0.001	0.001	0.001	0.002	0.003	0.007	Sr	0.00	0.0%	0.00	0.00	0.00	0.01	0.2%	0.01	0.1%		
Ti	0.01	0.01	0.01	0.02	0.01	0.01	0.03	Ti	0.00	0.0%	0.03	0.00	0.00	0.03	0.6%	0.03	0.4%		
Tl	0.001	0.001	0.001	0.001	0.001	0.001	0.001	Tl	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
U	0.001	0.001	0.001	0.001	0.001	0.001	0.001	U	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
V	0.01	0.01	0.01	0.01	0.01	0.01	0.01	V	0.00	0.0%	0.00	0.00	0.00	0.00	0.0%	0.00	0.0%		
W	0.01	0.01	0.01	0.02	0.01	0.01	0.02	W	0.00	0.0%	0.03	0.00	0.00	0.02	0.3%	0.02	0.2%		
Zn	0.03	0.03	0.06	0.07	0.06	0.07	0.25	Zn	0.09	3.6%	0.03	0.00	0.02	0.29	5.3%	0.38	4.8%		
Zr	0.001	0.001	0.005	0.009	0.008	0.005	0.011	Zr	0.01	0.5%	0.01	0.01	0.00	0.01	0.2%	0.02	0.3%		
Total									2.50	1.16	1.22	2.59	5.58	8.08					
Extraction Rates (ng/cm2 - day)																			
Day																			
B0(=log K)										0.356	K =		2.269865	1	0.685				
B1(=n)										0.302	N =		0.302	7	0.176				
											N-1 =		-0.698	14	0.109				