Modeling of component lifetime based on accelerated life tests and gas permeation measurements

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Introduction

- Metallic parts in chemical handling systems are subject to corrosion by acid gases like hydrogen chloride (HCl) and hydrogen fluoride (HF).
  - Examples include springs in valves and magnets in mag drive and maglev pumps
- Polymers, like perfluoroalcoxy (PFA), are often used to isolate these parts from acid gas containing liquids to prevent corrosion.
- Acid gases in these liquids can permeate through polymers and corrode the parts.
- Prior work has been conducted to measurement of HCl and HF permeation rates through polymers and develop a life prediction model based on permeation rates.
- This paper discusses the on-going accelerated life tests of pumps and impellers in HCl.

Outline

- Diffusion theory
- The effect of operating conditions on permeation rate thru PFA
  - Concentration
  - Temperature
  - PFA thickness
- Permeation rates of HCl and HF thru various polymers
- Life testing and accelerated life testing
  - BPS-3 at room temperature
  - BPS-1,3 and 4 elevated temperature testing
  - BSP-3 impeller elevated temperature testing
- Predicted relative lifetimes under different operating conditions
- Summary

Steady-state permeation

$$M = \frac{\mathrm{P} P_V A}{T}$$

Where M = Mass flow rate

P = Permeability coefficient

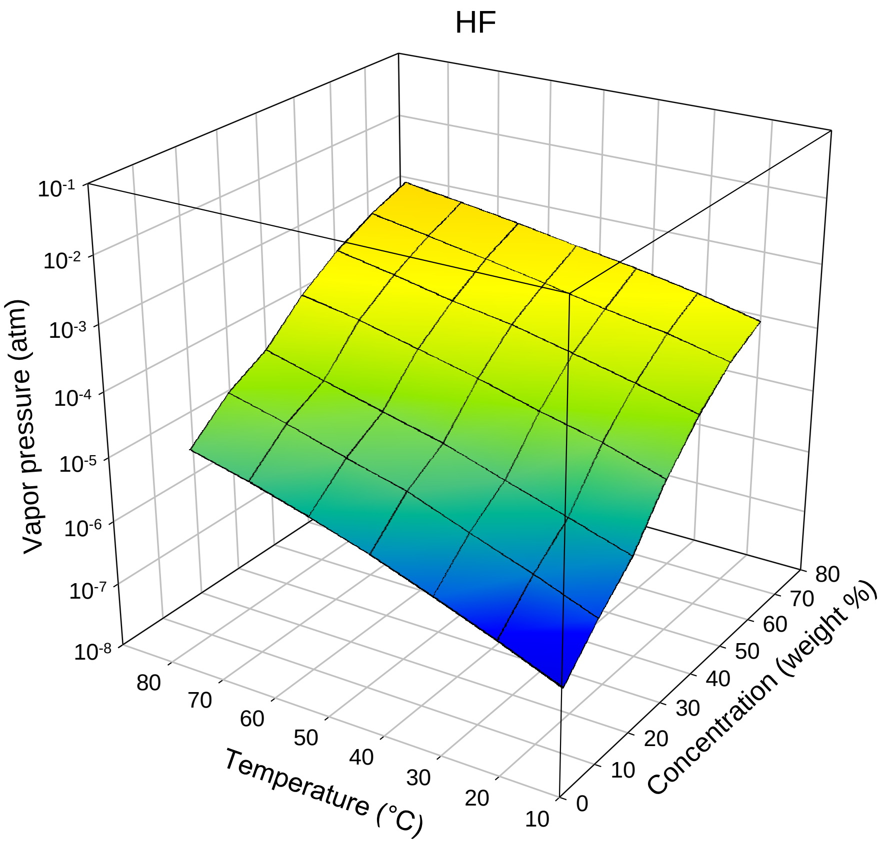
P_V = Gas vapor pressure

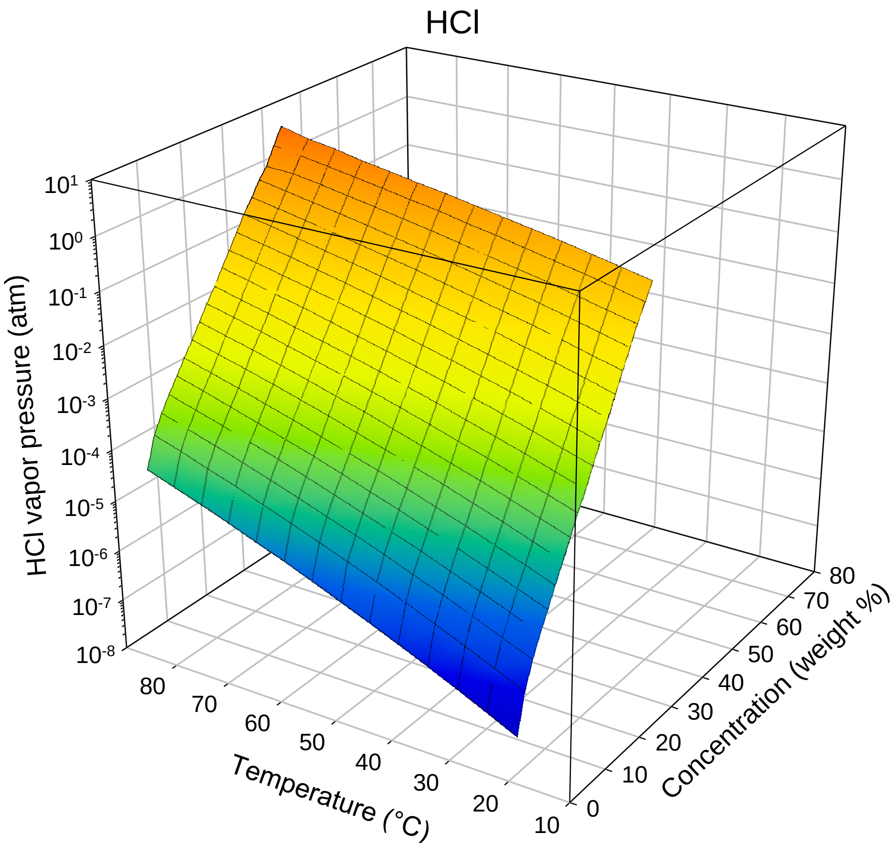
A = surface area available for diffusion

T = material thickness

Note: The mass flow rate is proportional to the gas vapor pressure; not the acid concentration.

Comparison between vapor pressures of HF and HCl over hydrofluoric and hydrochloric acids





Source: Brosheer at all, *Ind and Eng Chem* 39(3):423-427, 1947 and Hydrofluoric Acid Properites, Honeywell (2002).

Source: JH Perry, Chemical Engineers' Handbook, 4th Edition, McGraw Hill (1963) p 3-61

Comparison of HCl and HF vapor pressures

Vapor pressure of HCl over selected hydrochloric acid solutions

HCl concentration (% by weight)	Temperature (°C)	Vapor pressure (atm)
5	20	5.5×10^{-7}
6.3	75	2.1×10^{-4}
37	20	0.17
37	40	0.55
32	75	0.66
37	60	1.51

Vapor pressure of HF over selected hydrofluoric acid solutions

HF concentration (% by weight)	Temperature (°C)	Vapor pressure (atm)
0.5	20	1.2×10^{-5}
5.0	20	7.5×10^{-5}
49	20	0.018
49	60	0.15

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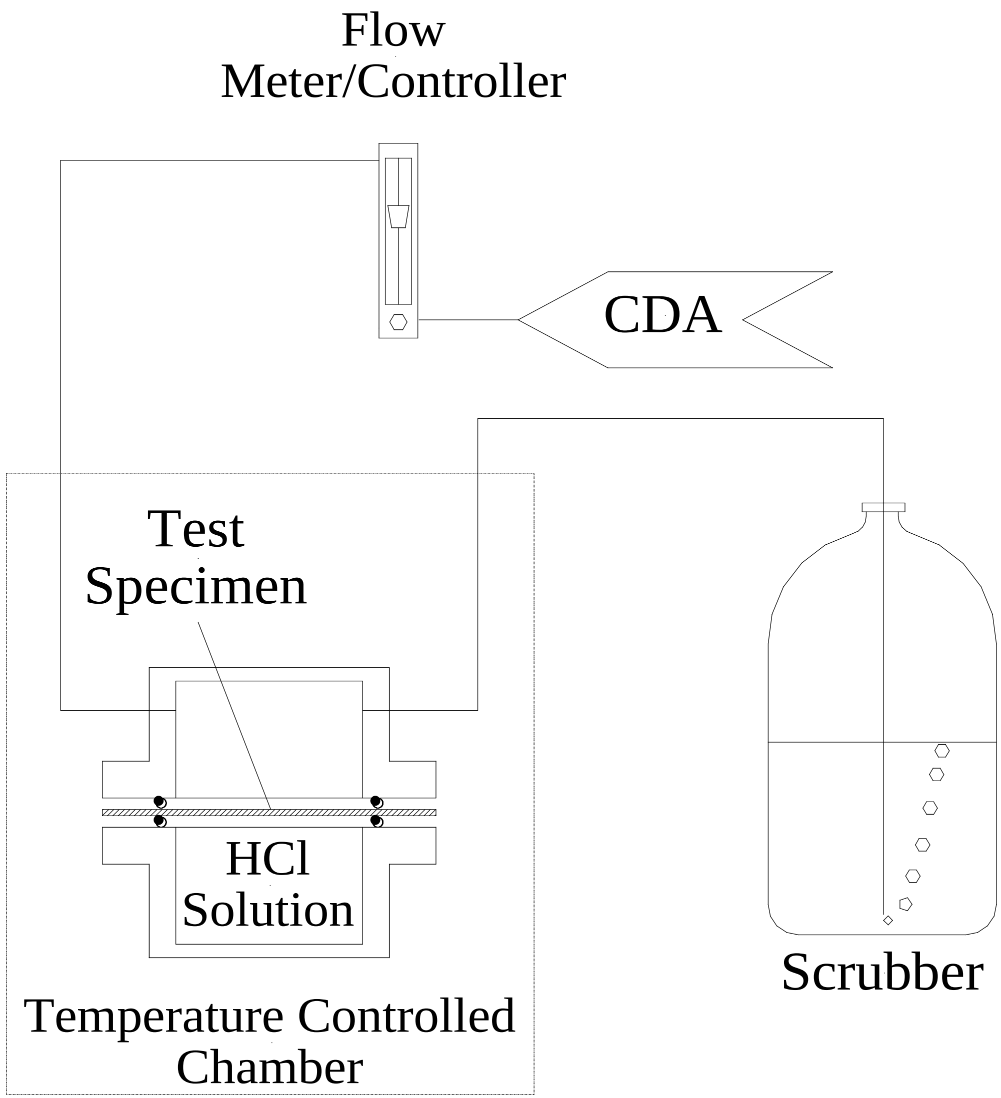
The effect of vapor pressure, temperature, and thickness on the permeation rates of HCl and HF through PFA

Test System Schematic



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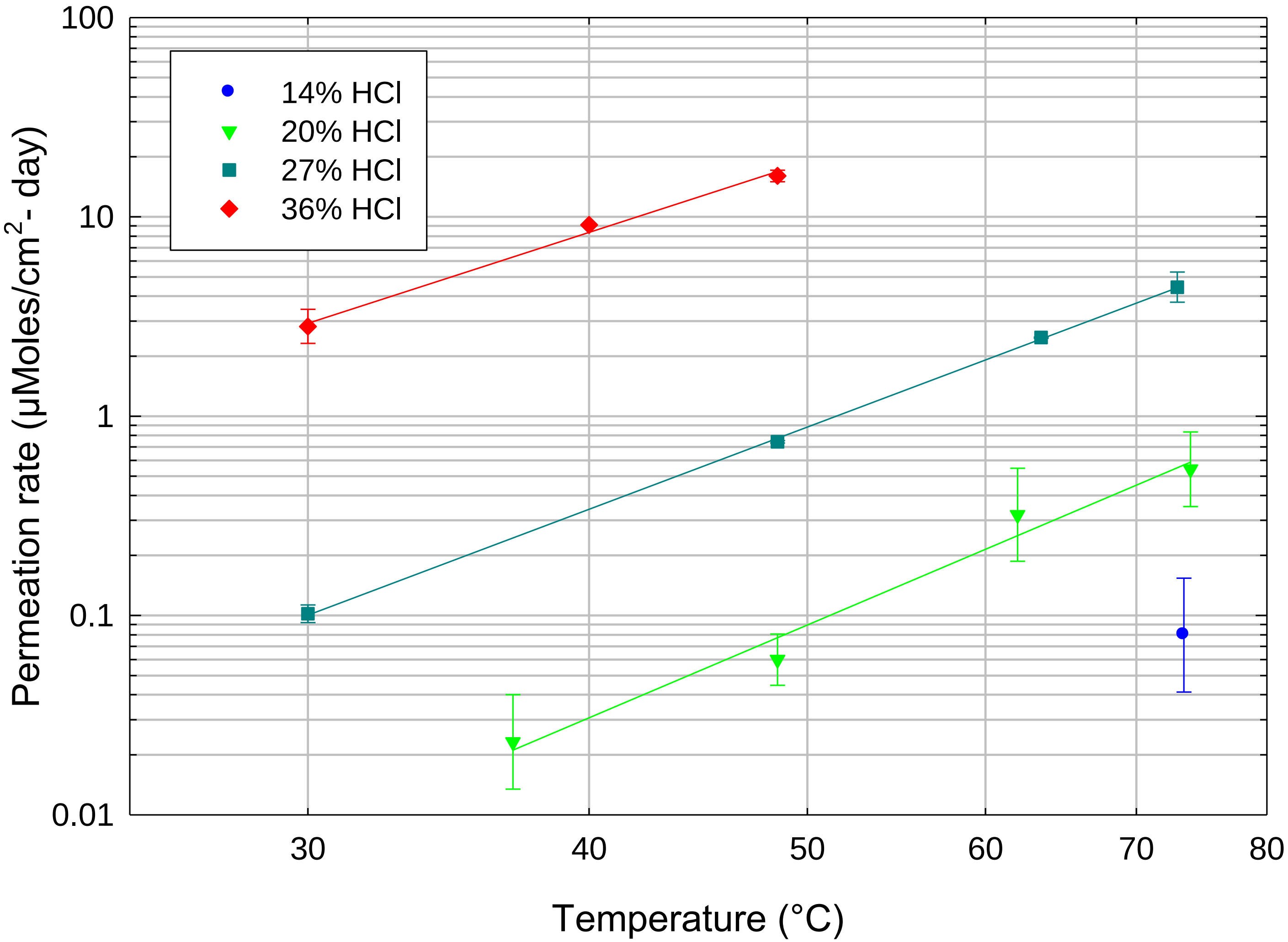
The effect of temperature and hydrochloric acid concentration on HCl permeation rate through 1.5mm thick PFA samples



Permeability coefficient

$$P = \frac{MT}{P_V A}$$

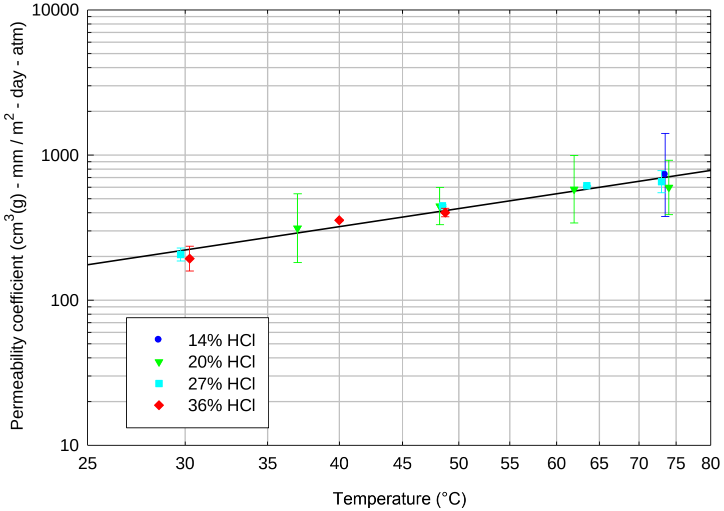
Where **P = Permeability coefficient**
M = Mass flow rate
T = material thickness
P_V = Gas vapor pressure
A = surface area available for diffusion

Permeability coefficient units:

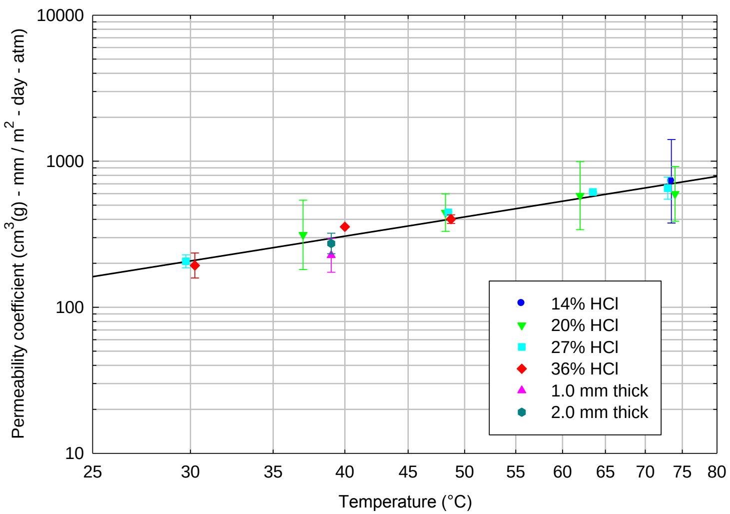
$$\frac{\text{Gas volume – thickness}}{\text{Area in contact with gas– Time - Vapor pressure}}$$

$$\frac{cm^3\text{ (g) – mm}}{m^2\text{ – day - atm}}$$

The effect of temperature on the permeability coefficient of HCl through PFA



The effect of thickness on the permeability coefficient of HCl through PFA

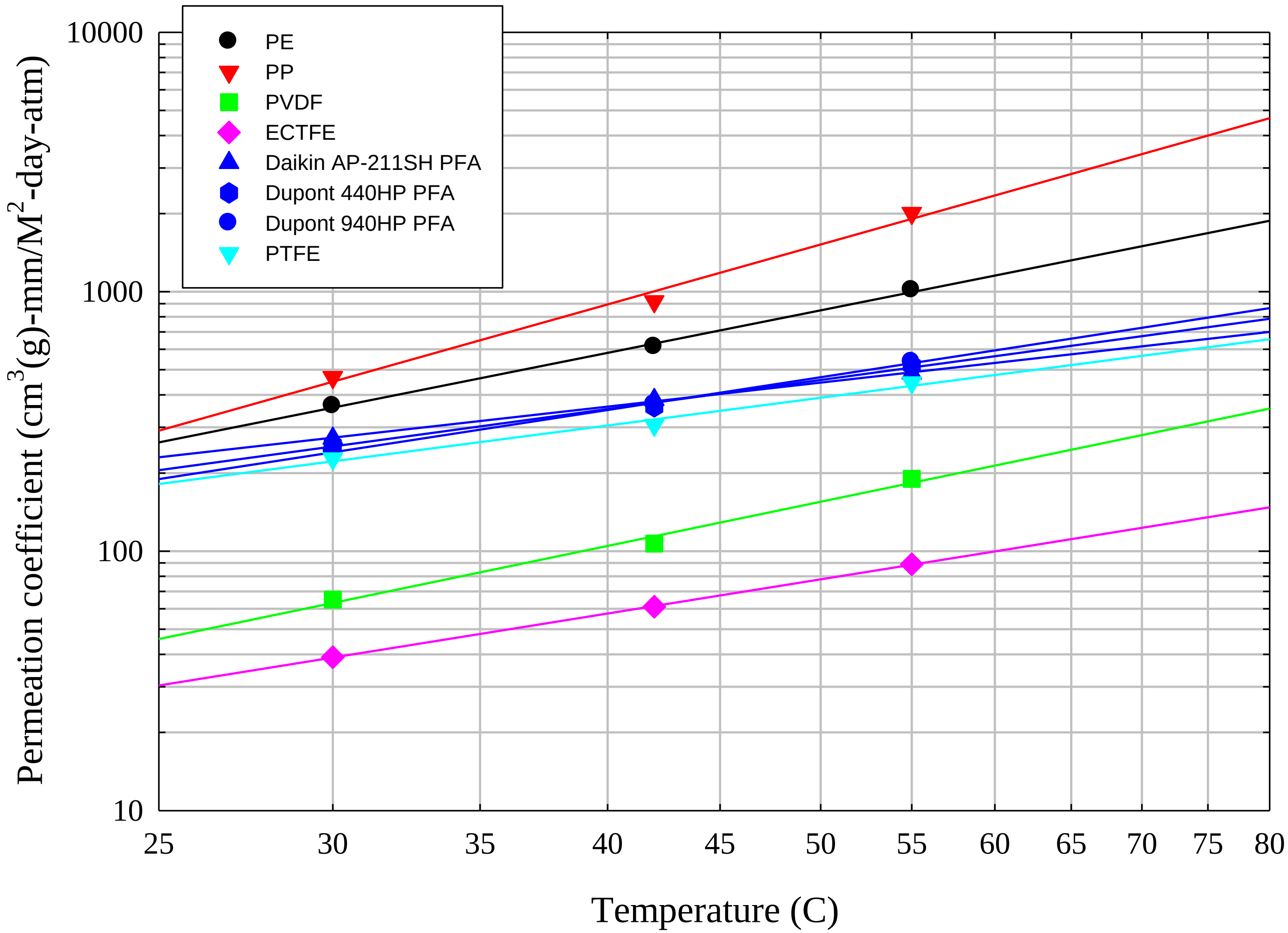


Permeation of HCl through PFA

- Permeation coefficient
  - Increases with temperature
  - Independent of chemical concentration
  - Independent of thickness
- Permeation rate
  - Is proportional to the acid gas vapor pressure
  - Increases with temperature
  - Is inversely proportional to the coating thickness

Polymer permeability comparison

Permeation coefficient of HCl through different polymers



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Comparison between HCl and HF permeation coefficients

Polymer	Permeability coefficient, $cm^3(g)$-mm/M^2-day-atm	
	HCl	HF
PE	430	-
PP	600	4200
PVDF	75	8800
ECTFE	45	3600
PFA	300	2600
PTFE	250	2700

@ 33^{o}C

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Comparison between HCl and HF permeation rates

Polymer	Relative permeability		Vapor pressure		Relative permeation rate	
	HCl	HF	37% HCl	49% HF	37% HCl	49% HF
PE	9.6	-	0.44	0.043	4.3	-
PP	13.3	93	0.44	0.043	5.9	4.0
PVDF	1.7	196	0.44	0.043	0.76	8.4
ECTFE	1.0	80	0.44	0.043	0.44	3.4
PFA	6.7	58	0.44	0.043	3.0	2.5
PTFE	5.6	60	0.44	0.043	2.5	2.6

@ 33^{o}C

$$M = \frac{PP_v A}{T}$$

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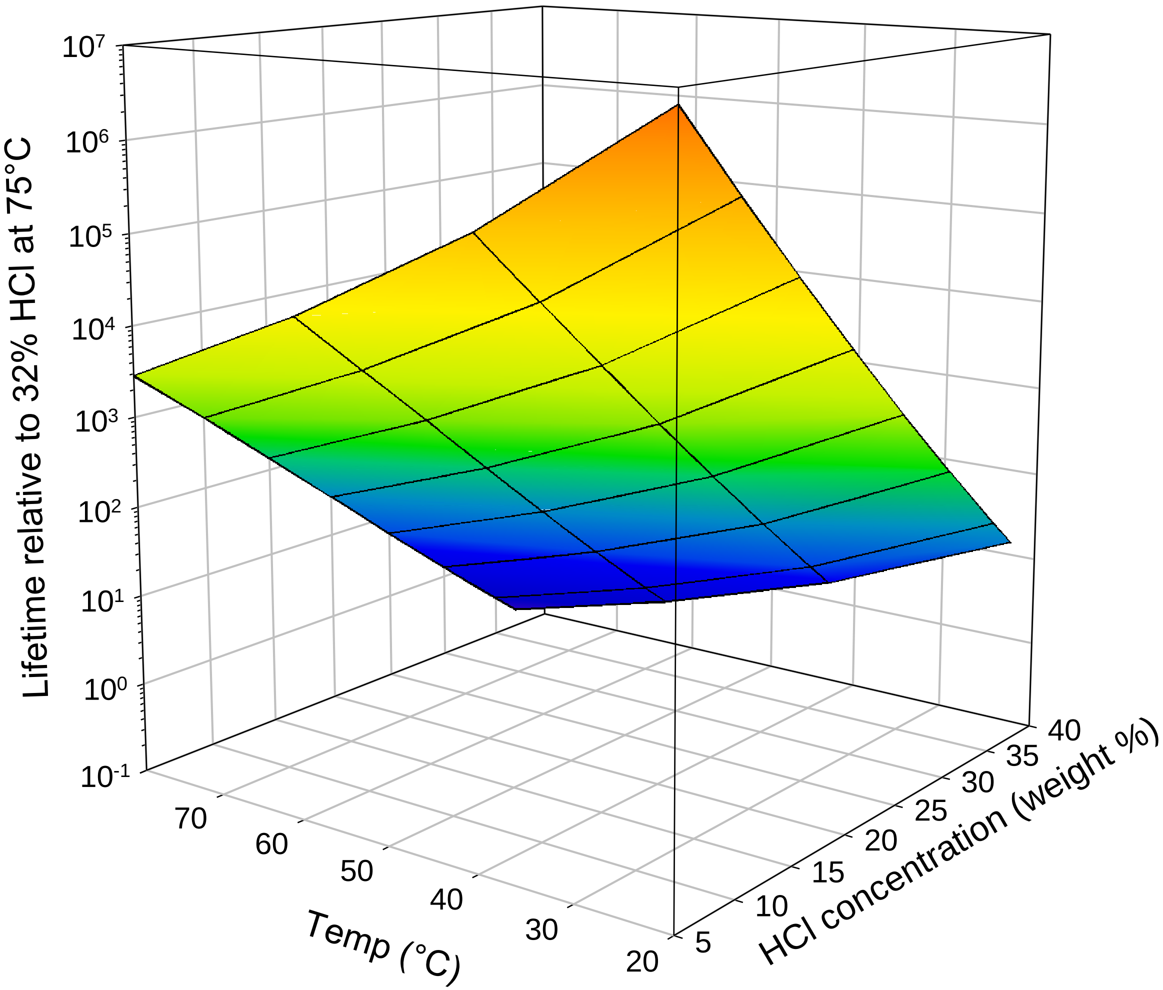
Component lifetime predictions

Assumption: Component lifetime is inversely proportional to the rate at which the acid gas reaches the component.

Predicted PFA coated component life based on HCl permeation rate (assumes that failure rate is proportional to permeation rate)



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Predicted relative lifetimes of PFA coated components under expected use conditions

Acid	Concentration, % by weight	Temperature, °C	Relative lifetime
HCl	6.3	75	3100
	37	20	22
	37	40	2.7
	32	75	1.0
HF	0.5	20	40,000
	5	20	6,400
	49	20	26

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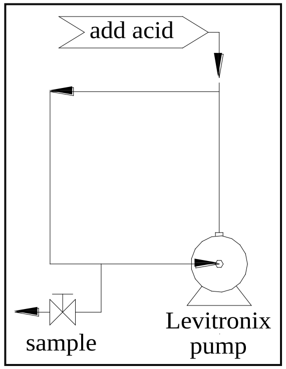
Accelerated life tests in chemical

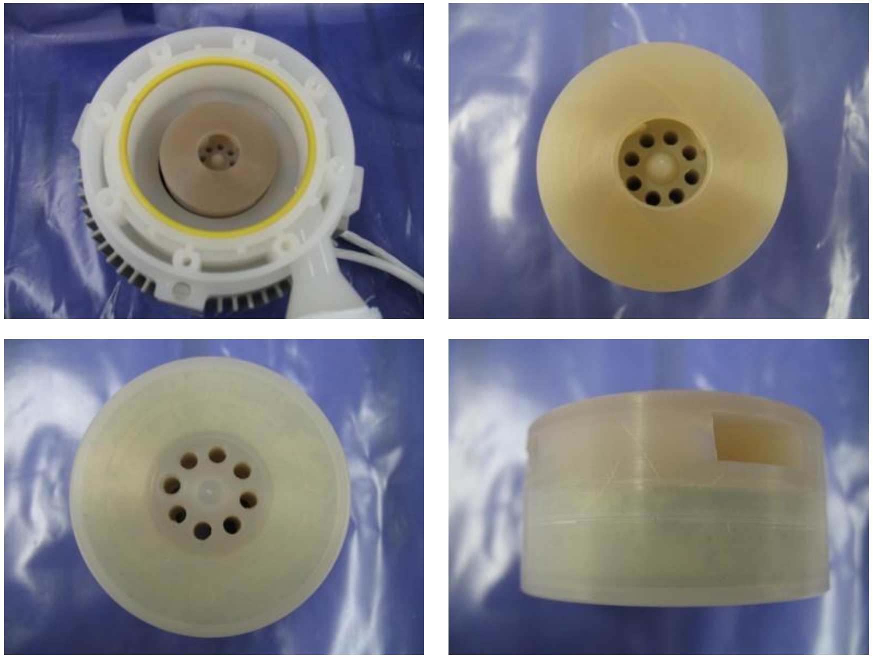
- Three studies on-going
- BPS-3 pump circulating 35-37% HCl at room temperature
- BPS-4 pump circulating in 30-32% HCl at 75°C.
- Static soak of BPS-3 PFA impellers with two encapsulation thicknesses (1.4 mm and 0.7 mm) in 30-32% HCl at 75°C.

BPS-3 Room Temperature Testing

- PVDF housing
- ECTFE encapsulated magnet
- 35-37% by weight HCl
- Room temperature (18-23^{o}C)
- Pump speed: 400 rpm
- Trace metal samples taken every 6 month
- Visual examination of impeller once a year.
- Operating nearly 8 years without a failure.



Test Schematic



Pump and impeller after 7 years of operation in concentrated HCl

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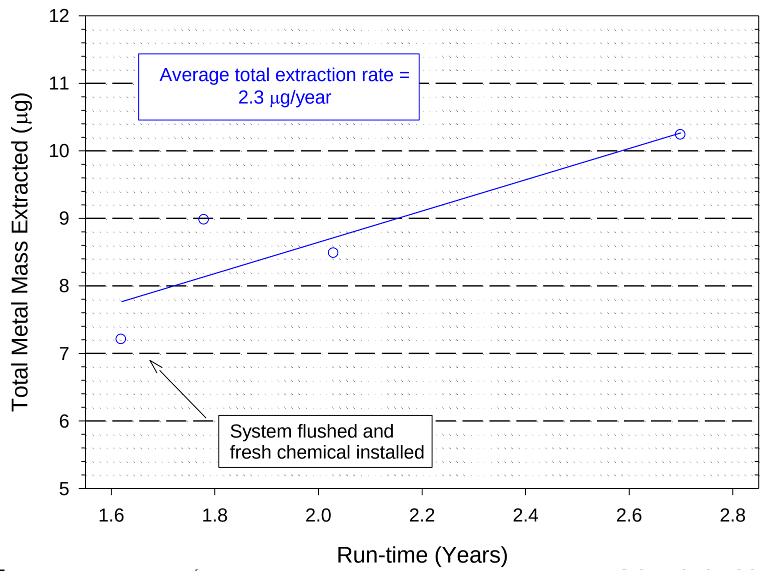
BPS-4 Elevated Temperature Testing

- PTFE pump body
- PFA encapsulated magnet
- Pump speed – 4350 rpm
- Outlet pressure – 14 ± 2 psi.
- Average HCl concentration – 31.3% by weight
- Average operating temperature 72.6^{o}C
- Trace metal samples taken every 6 months
- Pump has running time of more than 4 years without failure.

Test Apparatus

Trace metal measurements



Exposure Time Equivalents

Operating Conditions	Run-time, Years
As Tested (30-32% HCl @ ~70^{o}C)	4.1
37% HCl @ 20^{o}C	64
6.3%, 75^{o}C	9000

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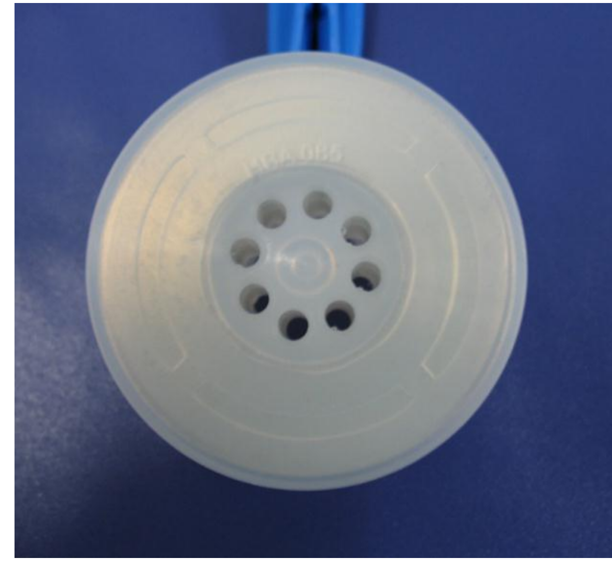
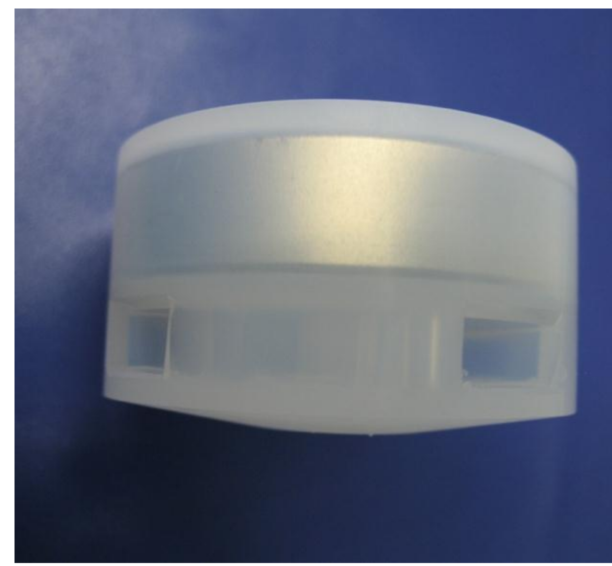
Impeller Testing in Hot HCl

- Static soak of 6 impellers.
- Two PFA encapsulation thicknesses (3 each).
  - 1.4 mm (standard)
  - 0.7 mm (non-standard)
- Average HCl concentration – 31.4% by weight.
- Assay measured at beginning and end of each conditioning period.
- Average conditioning temperature 74.8^{o}C.
- Trace metal samples taken every 3 months including blank.
- Photo examination every 3 months.
- Dimensional changes (diameter) measured at beginning of test and at 16 months.
- Impellers run at Levitronix in a BPS-3 after 16 months exposure.





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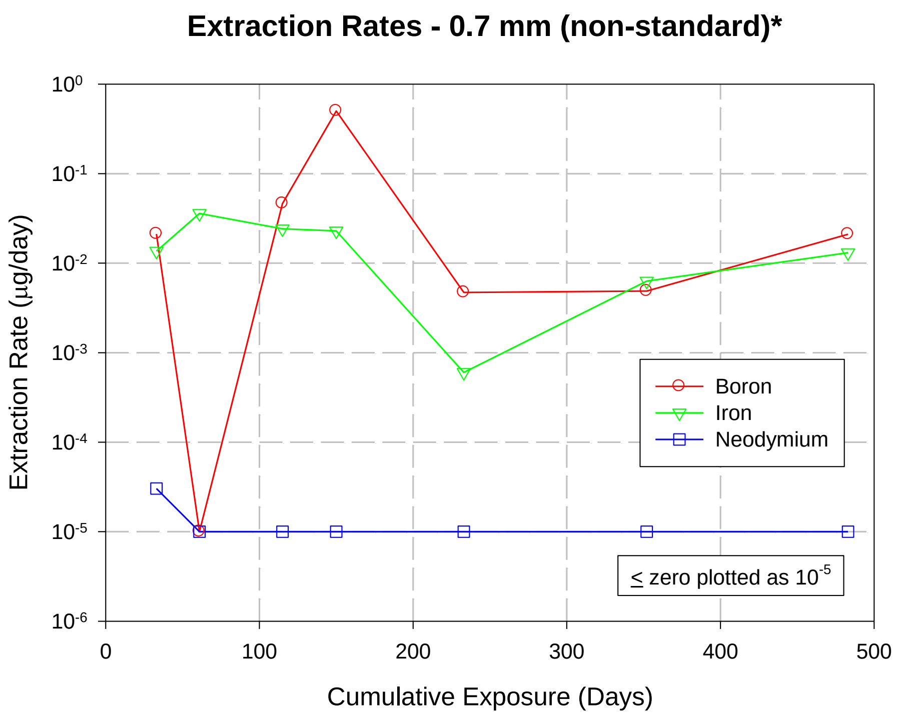
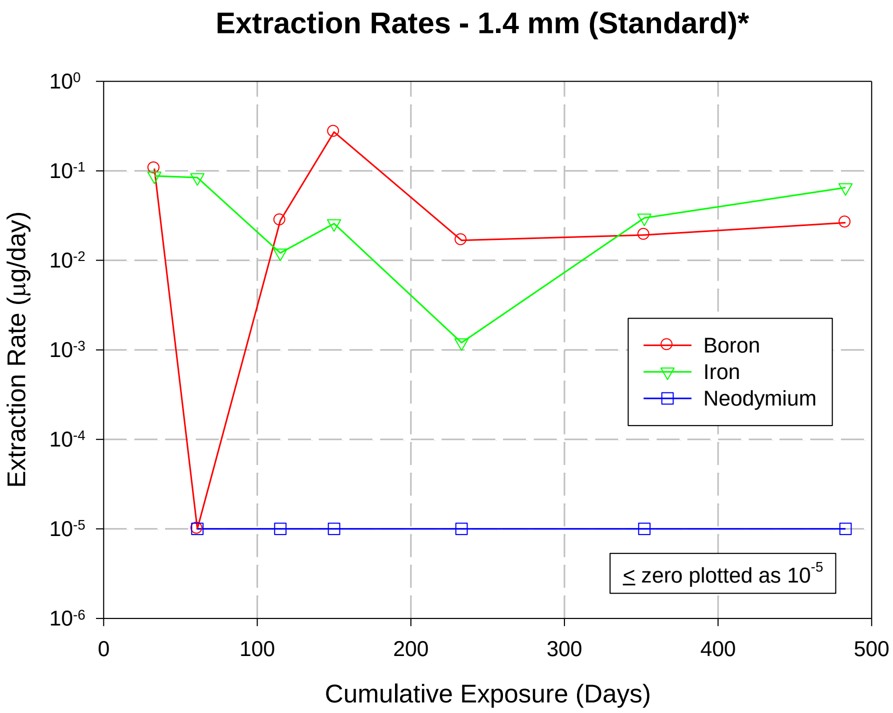
Trace Metal Extraction





* Blank subtracted

Are these extraction rates significant?

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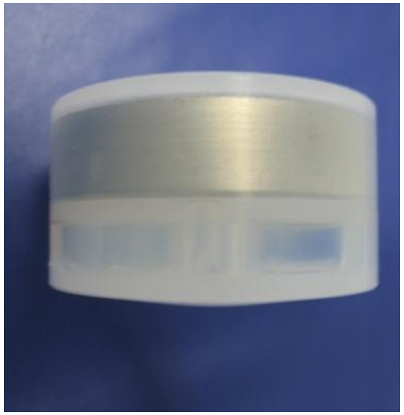
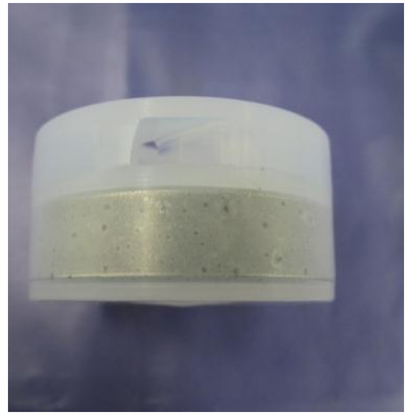
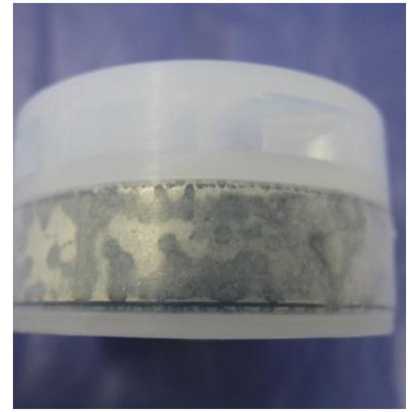
Trace Metal Extraction

- Assume:
  - Recirculation system with a 100 liter tank.
  - Chemical usage rate of 100 liters per day.
  - Worst case extraction rate of 0.02 ug/day iron (0.06 ug/day÷3 impellers) from prior figure.

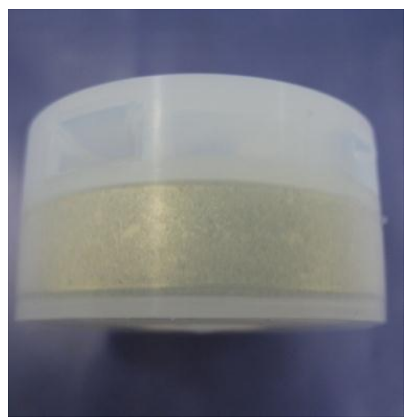
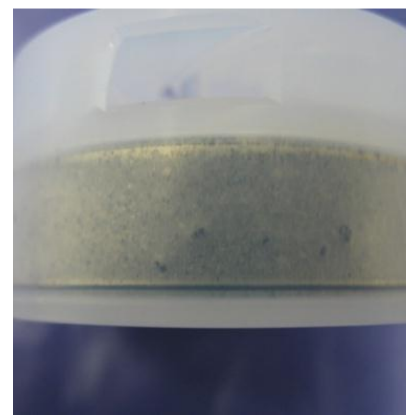
Change in iron concentration in the chemical would be 0.0002 ppb.
(0.02 ug/day÷100 liters/day)

Visual Changes

0.7 mm impeller (non-standard)

		
Before HCl Exposure	**4.9 Months HCl Exposure**	**15 Month HCl Exposure**

1.4 mm impeller (standard)

		
Before HCl Exposure	**4.9 Months HCl Exposure**	**15 Month HCl Exposure**

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Dimensional Changes

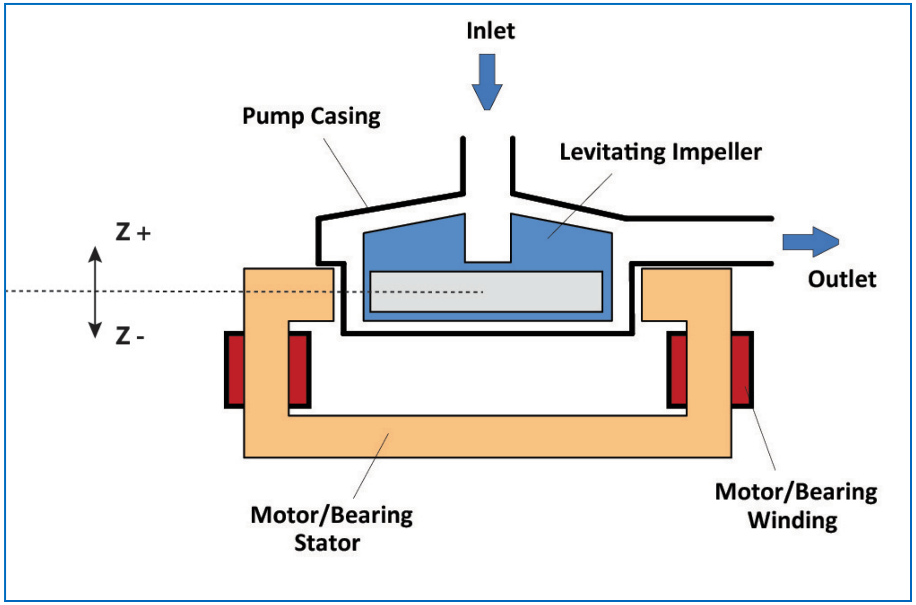
		Diameter as Received			Diameter after HCl Exposure			
Description	**ID**	**Mean (mm)**	**SD (mm)**		**Mean (mm)**	**SD (mm)**		**Diameter Change (mm)**
Standard Impeller (51.4 mm)	HBA 077	51.28	0.025		51.73	0.074		0.45
	GGQ 177	51.24	0.015		51.67	0.136		0.43
	GGQ 170	51.24	0.040		51.67	0.084		0.43
	Average	**51.25**	**0.03**		**51.69**	**0.10**		**0.44**
Special Impeller (50.0 mm)	HBA 085	49.99	0.031		50.49	0.157		0.50
	HBA 398	49.89	0.050		50.44	0.071		0.55
	HBA 320	50.04	0.053		50.70	0.118		0.66
	Average	**49.98**	**0.04**		**50.54**	**0.12**		**0.57**

Levitronix design approach for contamination prevention

- Pumps are designed to sense and respond to changes in impeller size.
- This provides an early indication of impeller swelling due to magnet corrosion.
- These accelerated life tests were designed to determine:
  - when the impeller size changes enough for the pump to sense and respond to impeller swelling (useful service life) and
  - if or when metal ions are released from the impeller (safe life)
- Goal is to have a safe life well beyond the useful service life of the impeller.

Levitronix "in-pump" testing

- Impellers were returned to Levitronix for testing in a BPS-3 pump.
- 3 standard thickness impellers were tested.
- Pumps were operated in water at:
  - 500 rpm, 0 lpm
  - 6000 rpm, 43 lpm
  - 6000 rpm, 79 lpm
- "Z" positions (axial position) were measured.



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In-pump test results

		Production impellers "Z" postion					HCl Conditioned impellers "Z" position		
Pump speed (rpm)	Flow Rate (lpm)	# of Samples	Mean (mm)	Standard Deviation (mm)	Min (mm)	Max (mm)	Mean (mm)	Min (mm)	Max (mm)
500	0	88	0.19	0.057	0.07	0.39	1.98	1.74	2.19
6000	43	88	1.71	0.094	1.49	1.94	4.40	3.69	4.92
6000	79	88	5.78	0.197	5.3	6.16	**7.04**	3.38	**8.23**

Note: "Z" position limit is -2.5 mm to +7.0 mm.

Predicted lifetimes based on pumps and impellers currently under test

Pump Model	Test Speed (rpm)	Run Time (Days)	Average Temp (^oC)	Average HCl Assay (wt%)	Service Life @ 37%/20^oC (Years)	Safe Life @ 37%/20^oC (Years)	Service Life @ 6.3%/75^oC (Years)	Safe Life @ 6.3%/75^oC (Years)
BPS 3	400	2874	RT	35-37	> 7.8	> 7.8	> 1100	> 1100
BPS 1*	4500	546	70.0	28.7	10.5	> 12.0	1,500	> 1,700
BPS 3*	4500	590	74.4	29.9	15.5	> 19.9	2,200	> 2,800
BPS 4	4370	1478	72.6	31.2	> 64	> 64	> 9000	> 9000
BPS 3 Impellers	0	546	74.8	31.4	≤ 26	> 29	≤ 3600	> 4000

* Test Complete

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Summary

- Previous work has shown permeation rates of HCl and HF through PFA to:
  - be proportional to the HCl or HF vapor pressure
  - be inversely proportional to the coating thickness
  - increase with temperature
- The permeation rates of HCl through different polymers is not a good indicator for HF permeation rates through the same polymers (and vice versa).
- A model has been developed to predict component failure rate resulting from HCl and HF acid gas permeation.
- The model, combined with on-going life test data, predicts pump lifetimes with PFA-coated impellers to be >10 years under challenging use conditions.
- No failures resulting in chemical contamination have yet to be observed in any of the exposure tests.
- In-pump impeller position can be used to signal impeller replacement point.

References

- Grant DC (2008). "The Effect of Exposure Conditions on Component Life in HCl solutions," presented at the 2008 Ultra Pure Fluid and Wafer Cleaning Conference, sponsored by Levitronix.
- Grant DC and D Carrieri (2008). "The effect of HCl permeation through PFA on expected component life," *Semiconductor Fabtech*, 37th Edition, pp 101-105.
- Van Schooneveld G, DC Grant, D Carrieri, D Chilcote and M Litchy (2009). "Modeling of component life based on accelerated acid gas permeation measurements," presented at the 2009 Ultra Pure Fluid and Wafer Cleaning Conference, sponsored by Levitronix.