

# THE EXTRACTION OF SURFACE AND BULK TRACE METAL IMPURITIES FROM TYPICAL FLUOROPOLYMERS

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## Biography

Dennis Chilcote, the presenting author, is a consulting engineer with CT Associates. His particular areas of expertise include biotechnology, water treatment, and environmental engineering. His broad experience in process design has been applied to the leaching of metals in aqueous solutions, the development of clinical instrumentation, and the design and fabrication of a variety of water treatment processes. He received his PhD in chemical engineering from the California Institute of Technology, with emphasis on bioengineering. He also holds a BS in chemical engineering from the University of Minnesota.

## Abstract

Design specifications for metals contamination from components in chemical handling systems are being established to improve the quality of high-purity semiconductor process chemicals that these systems deliver. The program to develop specifications requires some concomitant methodology to determine whether or not a given component meets the specification, such as the DyconE<sup>x SM</sup> technology that was developed and patented by FSI International. The objective of this study was to evaluate which acids should be used in this type of test method.

The extraction of trace metal impurities from both perfluoroalkoxy (PFA) and fluorinated ethylene propylene (FEP) bottles was evaluated over a 100-day period using concentrated hydrofluoric, hydrochloric, nitric and sulfuric acids. Each type of bottle was filled with a single ultrapure acid, except for the hydrofluoric acid tests, which were run in triplicate. A sample of acid from each bottle was analyzed at

0.01, 0.54, 3, 18, and 100 days. The analytical techniques used for the analysis of trace metals were inductively coupled plasma/mass spectroscopy (ICP-MS) and graphite furnace atomic absorption spectroscopy (GFAAS). The extraction of trace metals during the first 15 minutes (the 0.01-day sample) of each test was designated as surface extraction, whereas extraction between 0.01 and 100 days was designated as bulk extraction.

Although there is sufficient scatter in the triplicate data to suggest caution in drawing hard conclusions on certain comparisons, many general trends were observed. The major leachable trace metals from both types of bottles included Al, Ca, Fe, Mg, K and Na. Surface extraction was comparable to bulk extraction from the FEP bottles for all acids. Except for one hydrofluoric acid replicate, bulk extraction significantly exceeded surface extraction from the PFA bottles for all acids. For both bottle types, sulfuric acid was the least aggressive extractant. There did not appear to be any significant shift in the fingerprint of trace metals between surface extraction and bulk extraction for either type of bottle. Significant extraction of certain trace metals occurred during the time interval between 18 and 100 days. Except for sulfuric acid, the data do not support the conclusion that one acid is superior to all others.

Since samples were taken over time, rates of extraction could be estimated based on a power law model of the extraction of trace metals from a flat surface of bulk fluoropolymer. Rates of extraction for hydrofluoric, nitric and hydrochloric acid are of the same order of magnitude, but are an order of magnitude lower for sulfuric acid. Given the limited data of this study, the similarity in the rate data for

hydrofluoric, nitric and hydrochloric acids suggests that any one of these acids could be effectively used to evaluate the extraction of trace metals from fluid handling components. The clear conclusion from this study is that sulfuric acid should not be used as a test acid.

## Data

### Introduction

Semiconductor manufacturers are increasingly concerned about the levels of metallic impurities in semiconductor process fluids. The obvious sources of metal contamination in these fluids are the various process components that comprise a chemical handling and delivery system. To address this concern, manufacturers of chemical delivery systems are developing methodologies to evaluate the extraction of metals from the individual process components by process fluids that will contact these components. One such example is the DyconE<sup>x SM</sup> technology that was developed and patented by FSI International (1). The wetted surfaces of these components are almost exclusively some type of fluoropolymer. The ultimate objective of this effort to develop methodologies will be to establish design specifications for the extraction of trace metals from the components in the system. Since it is not practical to evaluate all potential process fluids, a preferred approach would be to determine the most aggressive chemical from the standpoint of metal extraction and use this chemical in the development of subsequent extraction protocols.

Many different types of process fluids are encountered in the semiconductor industry. However, related experience from the mineral processing industry and the sample preparation area of a typical analytical chemistry laboratory suggests that concentrated mineral acids are superlative in their ability to dissolve metals from a variety of matrices. In many cases, the matrix itself is dissolved. Thus, this study focused on four ultrapure mineral acids, hydrofluoric (HF), hydrochloric (HCl), nitric (HNO<sub>3</sub>) and sulfuric (H<sub>2</sub>SO<sub>4</sub>). The fluoropolymers chosen for this study were perfluoroalkoxy (PFA) and fluorinated ethylene propylene (FEP). The test components were 125-ml PFA and FEP bottles. The procedure was simple: fill a bottle of each fluoropolymer with each acid and monitor the change in concentration of 32 metallic species in the acid over time.

Although some work to define the extraction of metals from fluoropolymers has been reported in the literature (2), there is no definitive study on the effectiveness of various extractants for leaching metals from inert solids. This paper describes a study that was undertaken to begin the process of evaluating the effectiveness of certain acids to extract metals from typical fluoropolymers. The purpose of the study was twofold. The first objective was to determine if a particular acid was clearly advantageous as a general extractant of trace metals from fluoropolymers. The second objective was to evaluate the quality of the data that were generated in a typical extraction test and provide some indication of how effectively this data could be fit by a published extraction model.

In addressing the problem of metal contamination of fluoropolymers, there are two areas of concern. The first is metal contamination on the surface of the component due to manufacturing and/or machining and subsequent handling. The second is contamination in the bulk of the fluoropolymer material that was present in the raw materials used to prepare that material. During the analysis of the data generated during this study, an attempt was made to address both issues by arbitrarily dividing the total amount of metal extracted into two groups, a surface extractable fraction and a bulk extractable fraction.

### Experimental Approach

Two types of 125-ml fluoropolymer bottles were selected for this study. These bottles were obtained from a single supplier. The concentrated, ultrapure acids (Seastar Chemicals Inc., Sidney, British Columbia) were used without dilution. The test matrix is shown in Table 1.

Table 1. Number of Bottles Tested

Acid	Bottle Type	
	PFA	FEP
49% Hydrofluoric	3	3
37% Hydrochloric	1	1
70% Nitric	1	1
98% Sulfuric	1	1

Each bottle received from the supplier was sealed in a plastic bag. Each bottle was removed from the plastic bag, labeled accordingly, filled to the mark with acid, and capped. The bottles were not rinsed prior to the test. Once filled, except for sampling, the bottles sat

undisturbed at ambient temperature for 100 days. Each bottle was sampled at 0.01, 0.54, 3, 18, and 100 days. Approximately 15 to 20 grams of acid were removed at each sample time for sample preparation and analysis of 32 metallic species by ICP-MS and GFAAS. All trace metals except for Ca, Fe and K were analyzed by ICP-MS using standard hot plasma conditions. For Ca, Fe and K, either GFAAS or ICP-MS using cool plasma conditions was used. The baseline analysis (control) of each type of acid used in this study was obtained by sampling directly from the original acid bottle purchased from the manufacturer.

All extraction and sample preparation procedures were conducted in a Class 10 cleanhood located in a Class 100 cleanroom. The instruments used for this study were a Perkin-Elmer Elan 6000 ICP-MS configured with a CETAC microconcentric nebulizer (MCN) and a Perkin-Elmer SIMAA 6000 GFAAS. Both instruments were located in a Class 100 cleanroom.

### Data Analysis

It appears reasonable to assume that the surface contamination of fluid handling components with respect to metals could be different in degree and in kind from the metal contamination that is characteristic of the bulk of the material. Given this assumption, the analysis of the data attempted to separate surface extraction from bulk extraction. This attempt assumed that, at the start of the extraction test, the metals that were extracted were preponderantly from the surface contamination pool, and that this source was depleted very quickly. In the present study, metals extracted during the first 15 minutes of the test were defined as surface extraction, and all metals extracted after this time were defined as bulk extraction.

The results of interest are the mass of each metal and the sum of the mass of all metals extracted as a function of time. The measured data were the concentrations of metals in the respective acids at various times, and a data reduction protocol was required to obtain the mass values that were used to compare the effectiveness of the acids for extracting metals from fluoropolymers.

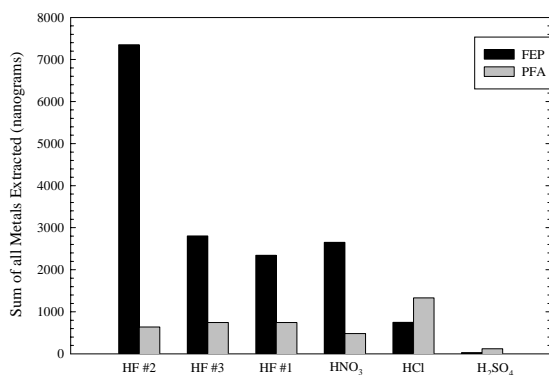
The two sets of tests using HF were run in triplicate to assess the variability of the results between bottles of the same type.

Since the amount of surface exposed to acid did change over time, each increment of mass extracted was weighted by the ratio of original volume to current volume at the time the sample was taken. However, for this approach to be valid the ratio of surface area to volume during the test must remain constant or the results will be biased. Given the geometry of the bottles and the amount of acid removed over the course of the test, the variation in the ratio of area to volume during the study was calculated to be on the order of 10 percent. This degree of variation was considered acceptable, and no correction was applied to the data.

### Experimental Results

A graphical comparison of the effectiveness of the various acids to extract metals from PFA and FEP bottles is presented in Figure 1.

Figure 1. Comparison of the Effectiveness of Acids to Extract Trace metals from Fluoropolymers  
cumulative sum of all metals extracted in 100 days

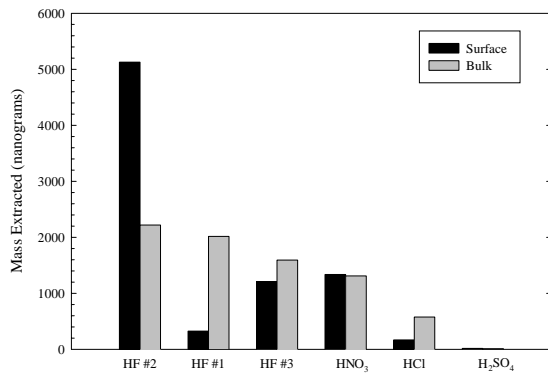


The initial conclusion from this figure is that HCl was most effective for the PFA material, and that HF and HNO<sub>3</sub> were most effective for FEP material. The conclusions for FEP are tempered by the large variability in the results from the replicate HF tests with the FEP bottles, which make ranking the effectiveness of the acids difficult from a statistical point of view. It does appear that H<sub>2</sub>SO<sub>4</sub> is definitely inferior to the other acids as an extractant for trace metals from these materials.

As mentioned previously, the mass of each metal extracted from any given bottle over the course of the test was divided into two fractions: that extracted from the surface of the fluoropolymer, and that extracted from the bulk of the fluoropolymer. All

metals extracted in the first 15 minutes were assigned to the surface fraction, and all metal extracted after this time was assigned to the bulk fraction. A comparison of the surface and bulk fractions of the total mass extracted from the FEP bottles is shown in Figure 2.

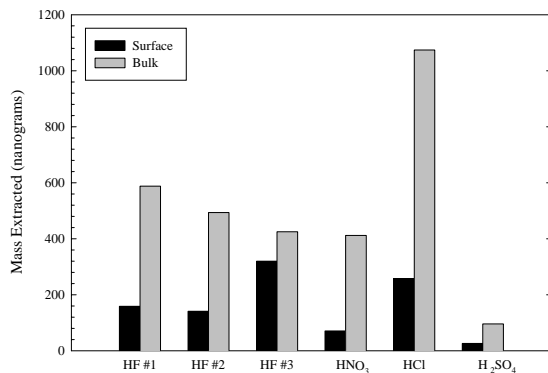
Figure 2. Comparison of Surface to Bulk Extraction sum of all metals extracted from the FEP Bottles



This figure illustrates the differences between surface and bulk extraction in the individual tests, and how these differences compare among the acids that were tested. They show that the mass of all trace metals extracted in the first 15 minutes of the test was on the order of the cumulative mass that was extracted between 15 minutes and 100 days. It is also apparent from these results that a significant portion of the variability observed for the HF replicates was in the surface-extractable fraction. The bulk fractions of total extractable metals for the three HF replicates were very similar.

Complementary results for the PFA bottles are presented in Figure 3.

Figure 3. Comparison of Surface to Bulk Extraction sum of all metals extracted from the PFA Bottles



Compared to the FEP bottles, the PFA bottles showed a much higher ratio of bulk extraction to surface extraction. As was observed for the FEP bottles, the variability was highest in the surface extraction fraction.

Although three replicates do not provide a high degree of statistical resolution, the statistical evaluation of the extraction data is nevertheless informative. Pertinent statistics from the replicate tests are shown in Table 2.

Table 2. Statistical Evaluation of the Mass Extraction Data from the HF Replicates

	Mean (ng)	Standard Deviation (ng)	Coeff. of Variation (%)
FEP, surface	2222	2557	115
FEP, bulk	1190	87	7
PFA, surface	207	98	48
PFA, bulk	288	16	5

The results from Table 2 suggest that the comparison of the effectiveness of the various acids should be based on the bulk extraction results, rather than total extraction. This approach eliminates a significant uncertainty in the cumulative mass extraction data caused by the high variability that occurs during the first 15 minutes of the leaching test.

Results for bulk extraction from all tests are presented in Table 3.

Table 3. Bulk Extraction from FEP and PFA, All Acids

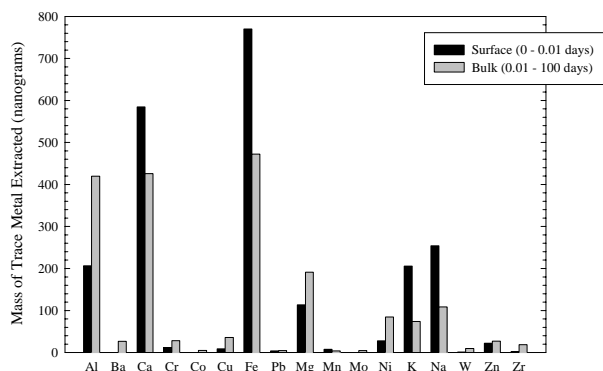
	Bulk Extraction of Total Metals (ng)	
	FEP	PFA
HF, replicate #1	1230	306
HF, replicate #2	1250	282
HF, replicate #3	1090	276
HNO <sub>3</sub>	1037	294
HCl	367	609
H <sub>2</sub> SO <sub>4</sub>	8	57

Based on this set of data, one would conclude that HF and HNO<sub>3</sub> give comparable results for both FEP and PFA, and are the most effective extractants of metals from FEP. HCl, on the other hand, was the most effective extractant for metals from PFA. H<sub>2</sub>SO<sub>4</sub> was an order of magnitude less effective as an extractant for metals than any other acid that was tested. The

replicate bulk extraction data for HF from the FEP and PFA bottles give standard deviations for this set of data that are on the order of 10 percent of the mean values. If these relative standard deviations are typical of all acids, then these initial conclusions are at least conditionally justified.

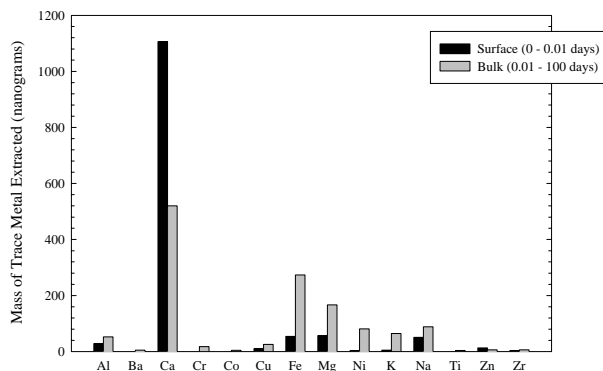
A comparison of the surface and bulk extraction of individual metallic species from the FEP bottles using HF is shown in Figure 4.

Figure 4. HF Extraction of FEP Bottles, Surface vs Bulk  
average of three replicates



This figure again shows the consistent pattern of high extraction of several metals during the first 15 minutes of the test. An interesting observation is that the metallic components that showed significant extraction during the first 15 minutes are also those that showed the highest extraction from the bulk material during the remainder of the test. Thus the fingerprints of metallic species that were extracted from the surface and bulk of the FEP bottles are similar, suggesting that the manufacturing/product handling operations did not add any contaminant to the surface that was not present in the bulk material. The observed differences were in degree, not kind.

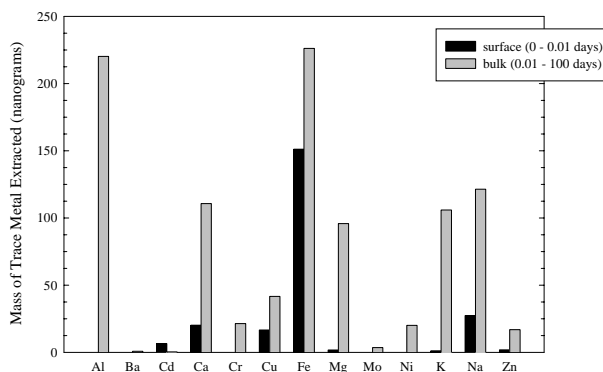
Figure 5. HNO<sub>3</sub> Extraction of FEP Bottles, Surface vs Bulk



Complementary results for FEP with HNO<sub>3</sub> are presented in Figure 5. For both acids, the metals that showed significant extraction were Al, Ca, Fe, Mg, Ni, K and Na, followed by Cr, Cu, Ni and Zn. Although the sums of all metals for bulk (not surface) extraction were similar for both HF and HNO<sub>3</sub>, the ratios of the mass of the individual metallic species that were extracted varied greatly between the two acids. This observed variability on an element-by-element basis reinforces the difficulty in ranking the effectiveness of the acids.

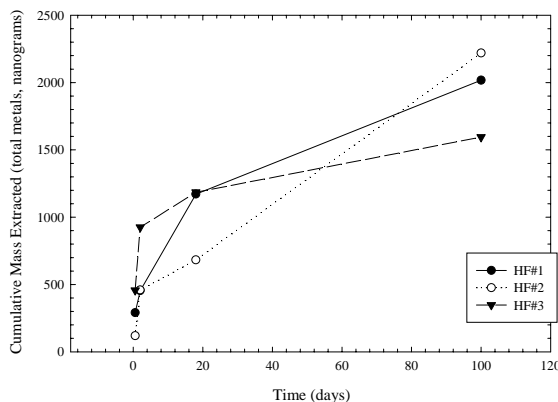
A comparison of the surface and bulk extraction of individual metallic species from the PFA bottles using HCl is shown in Figure 6. In this case, several metals that show significant bulk extraction are absent from the surface.

Figure 6. HCl Extraction of PFA Bottles, Surface vs Bulk



Again, no components were identified on the surface that were not present in the bulk. The metallic species that showed significant extraction from PFA were the same as those observed from the FEP bottles.

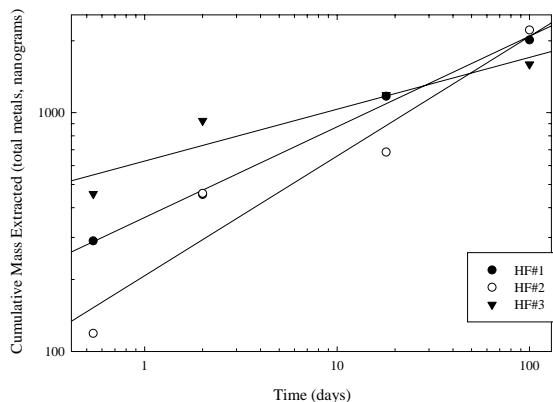
Figure 7. Bulk Extraction of Total Metals from FEP Bottles  
comparison of HF replicates



The replicate HF data for bulk extraction from the FEP bottles are plotted against time in Figure 7, and show considerable differences in the shapes of the extraction curves.

Theory predicts that the extraction data should follow a power law curve when cumulative mass extracted is plotted as a function of time (3). This theoretical derivation assumes no surface contamination, so the analysis of the extraction data must focus on bulk extraction. If the theory holds true, the bulk extraction data, when plotted in log-log space, should result in a series of straight lines, assuming that the surface component of the extraction data has been effectively eliminated. Log-log plots for the bulk extraction data for the HF replicates from the FEP bottles are shown in Figure 8.

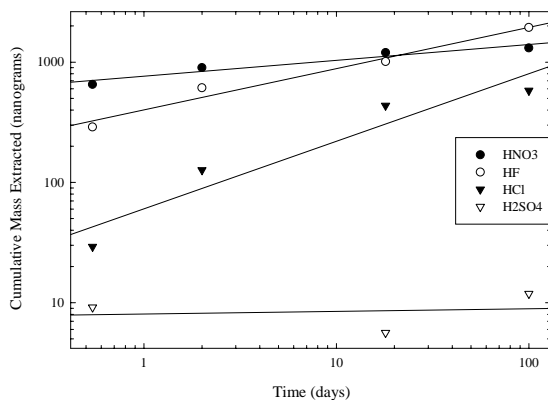
Figure 8. Bulk Extraction of Total Metals from FEP Bottles log-log plot



For at least two of the replicates, there is a fair degree of scatter about the straight-line fit of the data. If the variation in the results from the replicate tests was due solely to differences between bottles, we would expect a series of straight lines with good fits of each data set to its respective regression curve. The initial conclusion from these data sets is that the variation in the data is due to more than mere differences between bottles. Plotting the data for the HF replicates from the PFA bottles gave similar results, reinforcing this conclusion.

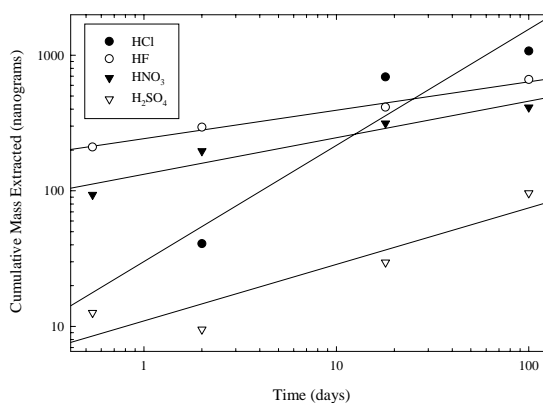
Cumulative bulk extraction from the FEP bottles as a function of time is plotted in Figure 9 for all acids tested. The HF plot is the average of the three replicates. This graph shows a reasonably good fit of the linear regression curve to the extraction data for all acids. Note that the log-log plot exaggerates small differences at low values.

Figure 9. Bulk Extraction of Total Metals from FEP Bottles log-log plot



Bulk extraction data from the PFA bottles are shown in Figure 10.

Figure 10. Bulk Extraction of Total Metals from PFA Bottles log-log plot



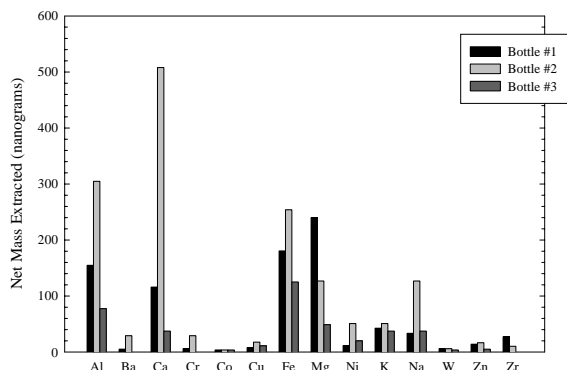
The peculiar data for HCl were apparently due to analytical variability in the first few time samples where analytical sensitivity is most critical. Fitting only the last two data points would give a curve that is much more consistent with the curves for the other acids. Although summing the data over all elements should help to average out variations in the individual analyses, it does not eliminate the analytical component of variability in the final results.

In general, the mass of any metal extracted from a fluoropolymer component is very low. Effective analysis of samples from a typical extraction test requires trace metal analyses that place the highest demands on the capability of the analytical instrumentation. Much of the data that is collected is on the order of the detection limits of the analytical instrument, where the analytical variability is highest. Unfortunately, small differences in results at this level

can significantly influence the slope of the regression curve, which in turn defines the model that is used to predict the rate of extraction at any given time. Ultimately, it is the rate of extraction that is most important for setting specifications.

To better understand the variability between bottles, the bulk extraction data from the HF replicates from day 18 to day 100 was plotted for each element. At day 18, all surface effects should have dissipated, and the increase in concentration between day 18 and 100 should be due solely to extraction of metals from the bulk of the fluoropolymeric material. Data for the HF replicates from the FEP bottles are presented in Figure 11.

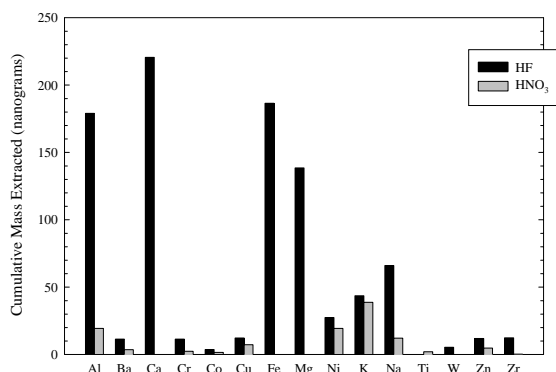
Figure 11. Cumulative Bulk Extraction of Metals from FEP Bottles comparison of HF replicates between day 18 and day 100



Differences between bottles were significant and, except for Mg, the relative ratios are reasonably comparable, which suggests that these differences are real. Similar results were noted for the PFA bottles.

The average results for HF were then compared to those for HNO<sub>3</sub>, as shown in Figure 12.

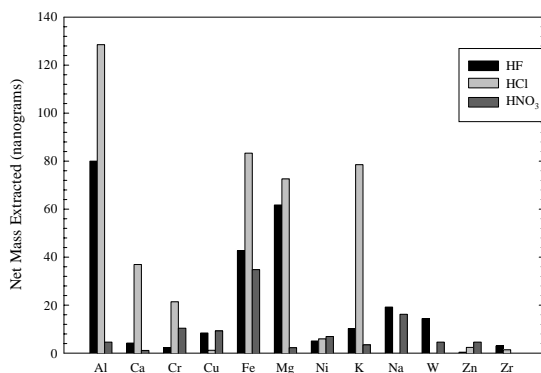
Figure 12. Cumulative Bulk Extraction of Metals from FEP Bottles comparison of HF and HNO<sub>3</sub> between day 18 and day 100



Although Figure 12 would suggest that HF is indeed more effective than HNO<sub>3</sub>, the error bars on the HF data derived from the results in Figure 11 are too large to statistically validate this conclusion.

Comparable data for HCl, HF and HNO<sub>3</sub> for the PFA bottles are shown in Figure 13. In this case, the variations observed between acids are on the order of those observed between bottles for the HF replicates (data not shown), and no relative ranking of acid effectiveness can be surmised from this figure.

Figure 13. Cumulative Bulk Extraction of Metals from PFA Bottles comparison of acids between day 18 and day 100



### Modeling the Extraction Data

As mentioned previously, theory predicts that the extraction of trace metals from a solid can be modeled by a power law equation:

$$y = k \cdot t^n \quad (1)$$

where:

- $t$  = time
- $y$  = mass extracted at time  $t$
- $k$  = proportionality constant
- $n$  = exponent

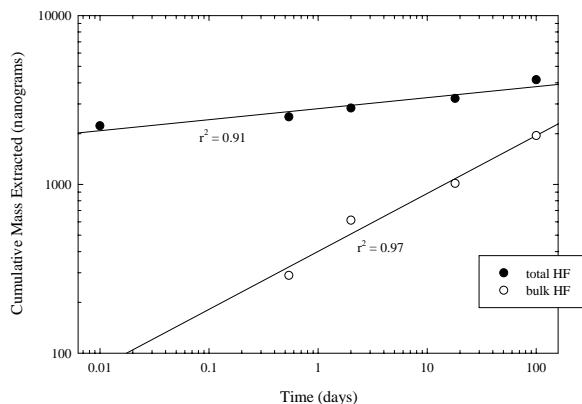
This theoretical derivation also predicts that the exponent would be 0.5. This derivation assumes that all mass extracted from the component comes from the bulk of the material, i.e., there is no pool of surface contamination that would extract rapidly and completely over a short period of time when the extractant first contacts the component.

The question of whether or not there is true surface contamination is not an easy one to answer. We have assumed that this fraction exists and is completely

removed in the first 15 minutes of the test. This assumption, however, is arbitrary. In addition, we know that extraction of material from the bulk phase begins at time zero, and that the rate of extraction is highest at this point. Thus, assigning even the first 15 minutes of extraction entirely to surface skews the bulk extraction data as well. However, a quick evaluation of equation (1) shows that if the exponent is 0.5, the mass extracted in the first 15 minutes of the test is approximately one percent of the total mass extracted in 100 days. The data from Figure 2 show that, for the FEP bottles, nearly 50 percent of the total mass extracted in 100 days appears in the first 15 minutes. Thus, if the model approximates reality even reasonably well, we can conclude that the FEP bottles have a significant level of surface contamination. What remains missing is a method to evaluate the amount of surface contamination from the extraction results.

Figure 14 presents a linear regression of all the averaged HF extraction data (cumulative from day 0) as well as a corresponding fit to the bulk extraction data (cumulative from day 0.01) plotted in log-log space.

Figure 14. Linear Regression of the Average HF Extraction Data  
log-log plot of the FEP bottle data



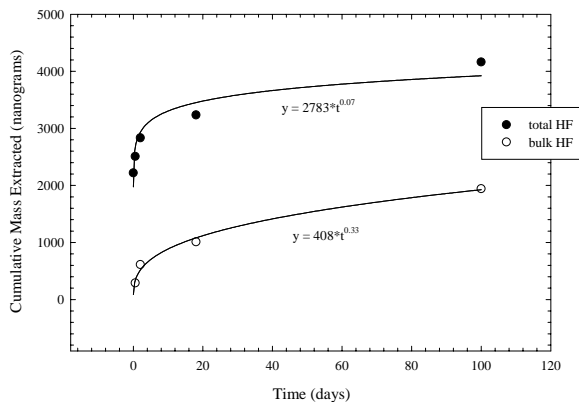
Both sets of data are fit by a linear regression; however, the bulk extraction data give a significantly better fit. In addition, for the total data set, the slope of the regression line is 0.06, whereas for the bulk data set the slope is 0.25. The bulk data set gives a slope much closer to that predicted by theory. Plotting the data in linear space and fitting each data set to equation (1) gives the results shown in Figure 15.

At least visually the fit appears much better for the bulk data in the range of 20 to 100 hours. In addition, fitting all the data with an equation of the form

$$y = y_0 + k \cdot t^n, \quad (2)$$

where  $y_0$  is the “surface” fraction that appears instantaneously at time zero, gives a value of 0.30 for the exponent and a value of 2118 for  $y_0$ . The total mass extracted at 15 minutes was measured as 2222, which is a good match to the predicted surface value given by  $y_0$ . The exponent also matches that obtained from fitting the bulk extraction data. These observations reinforce the assumption of a surface contamination component to the total extraction data.

Figure 15. Power Law Fit of the Average HF Extraction Data  
FEP bottles



Unfortunately, the data sets for the other acids do not behave as well. Using equation (2) to fit the complete set of extraction data for the other three acids that were tested did not consistently provide a reasonable prediction for the surface contamination component of the total mass of metals extracted from any given bottle. The determination of the rate of extraction at any given time is dependent on the model used to fit the data and, in the current approach, on the time chosen to separate surface extraction from bulk extraction. The development of a unified model is currently underway that will utilize all the extraction data, provide a reasonable fit of the data over a wide range of extraction conditions, predict surface contamination, and allow a practical estimate of the rate of extraction.

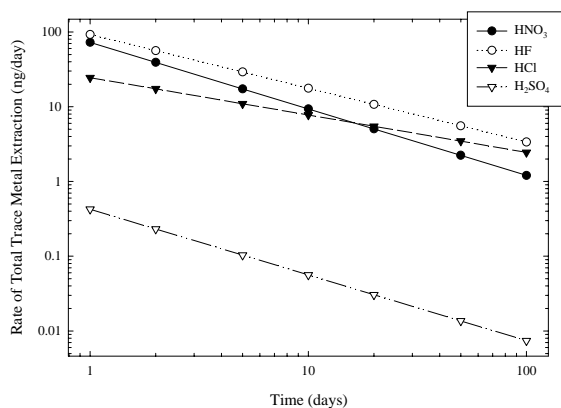


If equation (1) provides a reasonable fit of the extraction data, the rate of extraction can be determined by taking the derivative of this equation:

$$\text{Rate of extraction} = dy/dt = n * k * t^{n-1} \quad (3)$$

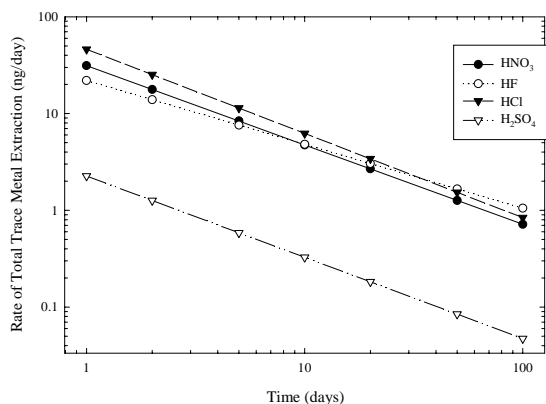
The parameters  $k$  and  $n$ , obtained from fitting equation (1) to the data, are then used to calculate the rate at any time  $t$  via equation (3). Calculated rates of extraction for the FEP bottles based on the regression parameters from Figure 9 are given in Figure 16.

Figure 16. Rate of Bulk Extraction of Total Trace Metals from FEP Bottles



The rates of extraction at any time  $t$  for HF, HCl and HNO<sub>3</sub> were similar, and were two orders of magnitude greater than that for H<sub>2</sub>SO<sub>4</sub>. The corresponding data for the PFA bottles are presented in Figure 17.

Figure 17. Rate of Bulk Extraction of Total Trace Metals from PFA Bottles



For both types of bottles, the rate data show similar patterns. Rather than distinguishing which acid is best, the data show conclusively that H<sub>2</sub>SO<sub>4</sub> is not the acid of choice for extraction studies.

## Conclusion

Four acids, HF, HCl, HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub>, were used to extract trace metals from PFA and FEP sample bottles over a 100-day period. Data from the extraction study showed sufficient variability between the HF replicate tests for both the FEP and PFA bottles to temper any attempt to establish a unique ranking of acids. For both types of bottles, the extraction results were similar for HF, HCl, and HNO<sub>3</sub>. Typically, the variability between HF replicates was on the same order as the variability between acids. H<sub>2</sub>SO<sub>4</sub> was significantly less effective as a trace metals extractant than any other acid, and based on the results of this study, would not be recommended. The results also suggest that the effectiveness of a particular acid in extracting metals from fluoropolymers might depend on the fluoropolymer itself.

Sources of variation in the data appeared to be due to the variability in the analytical data as well as variability between components. Variation between components could be significant, as was the case for the FEP bottles, and the analysis of one component may poorly represent an average of several components.

A power law model was used to evaluate and compare the bulk extraction data from the different tests. The parameters from this model were then used to calculate rates of extraction for the various tests. The rate data again corroborated the initial conclusions from the extraction data, showing that no single acid was significantly better than all others in extracting trace metals from fluoropolymers. The rate data did confirm that H<sub>2</sub>SO<sub>4</sub> was significantly less effective as an extractant for trace metals than the other acids.

The mass of all trace metals extracted during the first 15 minutes of a test was arbitrarily designated as surface extraction, and the mass extracted between 15 minutes and 100 days was designated as bulk extraction. One advantage in separating surface from bulk was the elimination of the high variability associated with the extraction results calculated at the first sampling point. Modeling the data with a power law equation did provide justification of the presence of surface contamination, but the case was not strong. Unfortunately, no alternative method is currently available to provide an estimate of the amount of surface contamination that might be present on a component. Modification of the power law model to include a surface contamination component gave inconsistent results. An effort is currently underway

to develop a reasonable model for the full data set from a typical extraction study that will effectively deal with the issues of surface and bulk extraction.

Based on the results presented in this study, HF, HCl, or HNO<sub>3</sub> could be used as the test medium to evaluate the extraction of trace metals from fluid handling components fabricated from fluoropolymers.

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