

Sampling Trace-Level Organic Solutes with Polymeric Tubing

Part 2. Dynamic Studies

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Abstract

This is the second part of a study conducted to determine whether polymeric sampling tubing can affect organic analyte concentrations during a sampling event. In this part of the study, we looked for sorption and desorption of trichloroethylene (TCE) and leaching of organic constituents in water pumped through five types of polymeric tubing. The materials tested were a rigid fluoropolymer, a flexible fluoropolymer, low-density polyethylene (LDPE), and two plasticized polypropylene tubings. The effects of tubing length and flow rate were examined.

The least sorptive tubings, both initially and at equilibrium, were the fluoropolymers. However, in some instances the LDPE tubing had little effect on TCE concentrations. This was when a slow flow rate was used to sample relatively shallow wells (50 feet [15 m] or less) or when a faster flow rate (1 L/min) was used to sample wells that are less than 500 feet (152 m). Further testing is recommended using more sorptive analytes.

Using high performance liquid chromatography (HPLC), we were unable to detect any constituents leaching from any of the tubings used in these studies, even when a slow flow rate was used. However, desorption of sorbed analytes is a concern for all the tubings tested, including the rigid fluoropolymer.

Introduction

It is important that the reported concentrations of contaminants in samples taken from ground water monitoring wells accurately reflect in situ values. One concern with sampling methods that involve pumping water to the surface is that the tubing could affect analyte concentrations by either sorbing or leaching inorganic or organic contaminants. This problem would be exacerbated when low-flow sampling is used or in deep wells, where the contact time would be longer. In addition, if the tubing is not dedicated to a particular well, it is possible that tubing used previously to sample a well with high contaminant concentrations could release sorbed contaminants into the next sample, leading to inaccurate high values. In this paper, we focus on the effects tubing materials can have on ground water samples with respect to sorption of organic contaminants, leaching of organic constituents, and desorption of sorbed organic contaminants.

Sorption and Leaching of Organic Contaminants

In part one of this investigation (Parker and Ranney 1997b), we reviewed the existing literature on sorption of organic contaminants and leaching of organic constituents; thus, that material will not be presented here. Also in that study, we compared sorption of eight organic solutes (including six volatile organic compounds [VOCs] and two nitroaromatic compounds at low mg/L concentrations) and

leaching of organic constituents by 20 sampling tubings. The tubings included 13 rigid tubings and seven flexible tubings, of which eight were fluoropolymers. We found that the rigid fluoropolymers, especially the fluorinated ethylene (FEP), FEP-lined polyethylene (PE), and polyvinylidene fluoride (PVDF) tubings, were the least sorptive materials. With respect to leaching, we did not detect any organic constituents leached from the six rigid fluoropolymers, four rigid polyethylene tubings, and one of the flexible plasticized polypropylene tubings.

A logical conclusion to our previous study is that only the rigid fluoropolymers, especially FEP and PVDF, should be used to sample organic analytes. However, we realize that the biases we observed previously might either increase or decrease under dynamic conditions. With respect to sorption of organic solutes, we expect that losses due to sorption would be reduced, or possibly eliminated, with time as equilibrium is approached. There is some evidence in the existing literature to support this hypothesis. Devlin (1987) reported that representative samples could be obtained by flushing PE tubing with contaminated water for five to 10 minutes, and that the equilibration time depended upon the length of the tubing and the pumping rate. However, no data were provided to support this claim. Thus, it may be possible to use a less sorptive material without affecting analyte concentrations if time is allowed for equilibration.

With respect to leaching of constituents, the existing literature indicates that exposure may either increase or decrease with time. Several leaching studies (Packham 1971a and 1971b; Gross et al. 1974; Boettner et al. 1981) have shown that much of the leaching that occurs from rigid PVC pipe decreases with time and is considered a surface phenomenon. This may be true for other polymers. On the other hand, if leaching still occurs with continued flushing, as Junk et al. (1974) observed with flexible PVC, then leaching rather than sorption may dictate which tubings are suitable for sampling ground water.

Therefore, while we know that concentrations of some organic analytes can be affected when contaminated water is pumped through some polymeric tubings, we do not know the full extent of this effect with time. We anticipate that losses would decrease with time. However, we do not know how long it would take to reach equilibrium or what would be the extent of losses at equilibrium. In addition, we do not know what effect flow rate has on this process. With respect to leaching, little is known about the effect of flow rate or continued pumping.

Desorption of Sorbed Organic Contaminants

Although our previous literature review and study examined sorption and leaching of organic compounds, we did not consider desorption of sorbed organic contaminants. Because all types of polymeric tubing have been shown to sorb some organic contaminants, desorption of these compounds could bias samples if the tubing is used for more than one sampling event,

and especially if it is moved from a well containing high analyte concentrations to one with low concentrations. However, only a few studies have addressed this issue.

Desorption of sorbed organic compounds by polymers proceeds in the opposite order of sorption (i.e., diffusion through the polymer matrix to the surface followed by partitioning/dissolution back into the aqueous solution).

Barcelona et al. (1985) studied the release of organics from five flexible tubings: PP, PE, PTFE, flexible PVC, and silicone rubber. These tubings had been previously exposed to two test solutions, one containing ppb levels of chloroform and one containing ppb levels of four chlorinated organic solvents. Conditions for these tests were static, and desorption was followed for one hour. They reported that most (80 to 90%) of the desorption occurred during the first five minutes but, by the end of the hour, most (>50%) of the sorbed organic solutes had not been recovered. They also found that, while the rate and extent of sorption was greatest for the PVC and silicone rubber tubings, the percent desorbed relative to the amount sorbed was greatest for PP and PE tubings, intermediate for PTFE, and lowest for the PVC and silicone rubber tubings.

Devlin (1987) found that the PE tubing was more sorptive of ppb levels of 15 VOCs than Teflon® tubing and also desorbed the VOCs over a longer period. However, no information was provided relative to which tubing desorbed the analytes most relative to the amount sorbed or what flow rate was used in the study. Devlin concluded that carryover of organic contaminants may be a problem unless a fluoropolymer tubing is used.

Thus, little is known about the kinetics of desorption, especially when water is pumped through the tubings. Furthermore, the effect that flow rate has on this process is unclear.

Objective

The purpose of these studies was to determine whether, under dynamic conditions, polymeric tubings sorbed TCE from contaminated well water, leached organic constituents, or desorbed sorbed TCE. In our sorption/leaching studies, we quantify losses when the flow rates are moderately fast (1 L/min) and slow (100 mL/min), determine if equilibration occurs and when, and investigate whether organic constituents are leached. In our desorption study, we determine if TCE is released from contaminated sampling tubings when organic-free water is pumped through them at a slow flow rate, and again look for leaching of organic constituents.

Materials and Methods

The five tubing materials used in this study were LDPE, one rigid fluoropolymer (PVDF), one flexible fluoropolymer [P(VDF-HFP)], and two formulations of plasticized polypropylene (PP1, PP2). We selected these tubings because, in our previous study (Parker

and Ranney 1997b), these tubings leached little or no organic constituents and ranged from relatively highly sorptive (PP1 and PP2) to least sorptive (PVDF). P(VDF-HFP) was one of the least sorptive flexible tubings. All the tubings had an inside diameter of 0.25 inches (6.4 mm) and were not cleaned prior to the study.

Water contaminated with trichloroethylene (TCE) was obtained from an artesian well that is located at our facility. This well can supply up to 380 gal/min (1440 L/min).

Experimental Design for the Sorption Studies

The outflow pipe from the well was tapped and water was drawn off at flow rates that varied from 100 mL/min to 3 L/min. The water was diverted from the outflow pipe to a water distribution system consisting of four components: (1) a master flow valve; (2) an electronic flow meter connected to a linear 555 chart recorder; (3) a three-port valve, which allowed samples to be siphoned off and yet still allowed flow to continue through the tubings; and (4) a bank of five multiturn needle valves, where up to five tubings could be connected (Figure 1). The materials used in the distribution system consisted primarily of copper, brass, and glass. Small amounts of Teflon, Perfluoroalkoxy, and Kel-F were used in the O-rings, three-port valve, and needle valve body. To flush the system, the well water was pumped through the system for several hours prior to connecting the tubings. Strip chart recordings showed that the flow rate into the distribution system varied by approximately 10%.

The tubings were wound around a 55-gallon (208 L) metal drum in an upward spiral fashion; this expelled any air from the tubings as they filled with water. The rate of flow through each tubing was routinely moni-

tored by collecting a volume of water from the outflow of the tubing in a graduated cylinder and measuring the time with a stopwatch. Flow through each tubing was regulated by turning the appropriate multi-turn needle valve at the end of the distribution system.

In our first study, all five tubings were tested: PVDF, P(VDF-HFP), LDPE, PP1, and PP2. The contaminated well water was pumped through three lengths—10, 50, 100 feet (3, 15, 30 m)—of tubing at a slow flow rate (100 mL/min). In our second study, the well water was pumped through 500 feet (152 m) of LDPE at the same flow rate (100 mL/min). In our third study, the water was pumped through 100-foot sections of three tubings (PP1, LDPE, and PVDF) at a much faster flow rate, 1 L/min.

In all three studies, samples were collected after 10 and 30 minutes; one, four, and eight hours; and one, two, and three days. Additional samples were collected as needed. For a complete listing of sampling times, see Parker and Ranney 1997a. For each tubing material and sampling time, three samples were collected by filling 1.8-mL HPLC autosampler vials directly from the outflow of the tubing. To determine the initial concentration of TCE in the well water for a given time, three control samples were obtained by collecting water from the three-port valve. The samples were analyzed immediately after collection.

Experimental Design for the Desorption/Leaching Study

The three tubings (PVDF, LDPE, and PP1) that had been contaminated by pumping contaminated well water through them at a flow rate of 1 L/min for seven days (168 hours) in the previous study were used in this study. Flow was stopped from entering the tubings by closing the needle valves at the distribution system, and

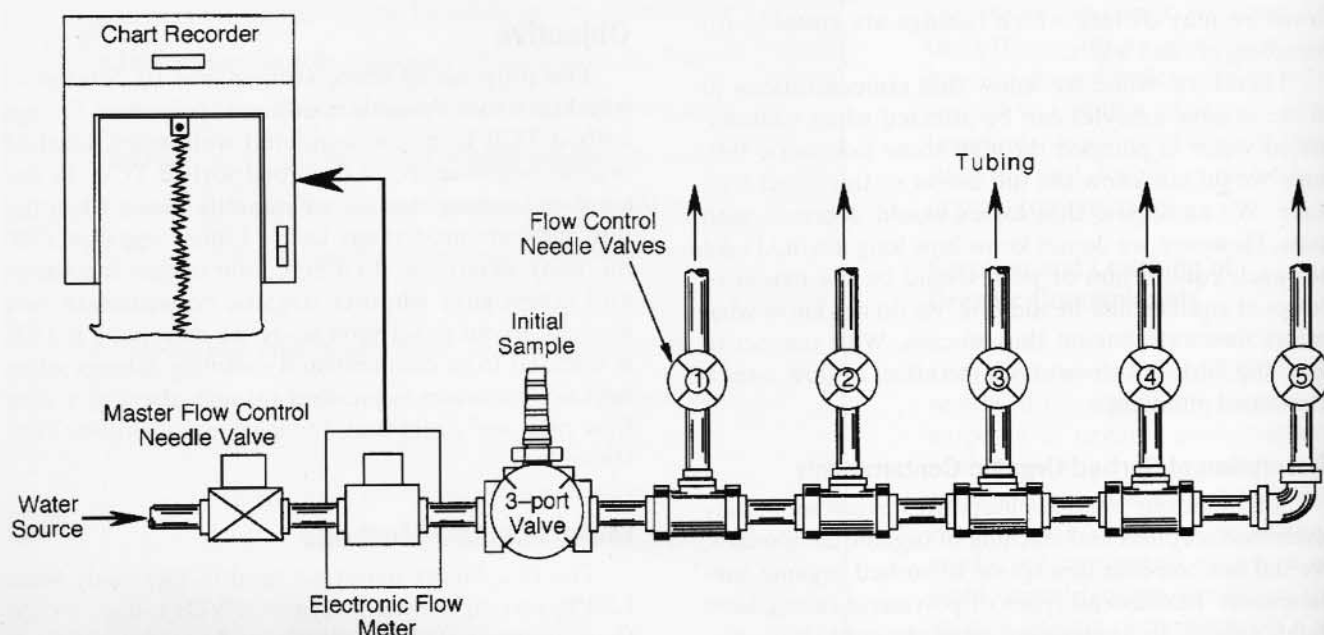


Figure 1. Diagram of distribution system from water source to tubings.

the open ends of the tubings were plugged with short pieces of solid glass rod. The distribution system was disconnected from the well's pump and these ends were plugged with short pieces of solid glass rod. The complete system was then moved from the well house to the laboratory. The distribution system was connected to the laboratory's deionized (DI) water source, and DI water was pumped through each tubing at a flow rate of 100 mL/min. Samples were collected after 10 minutes, 30 minutes, one hour, four hours, eight hours, 24 hours, 48 hours, 72 hours (three days), and 96 hours (four days).

Analyses

Analytical determinations were performed using reversed-phase HPLC (RP- HPLC). A modular system was employed consisting of a Dynatech LC-241 autosampler with a 100- μ L injection loop, a Spectra Physics SP8810 isocratic pump, a Spectra Physics SP100 variable-wavelength UV detector set at 215 nm, and a Hewlett-Packard 3396 series II digital integrator. Separations were obtained on a 25-cm by 0.46-cm (5- μ m) LC-18 column (Supelco) and eluted with 1.5 mL/min of 65/35 (V/V) methanol/water. The detector response was obtained from the digital integrator operating in the peak height mode. A primary TCE standard (3000 mg/L) was made by weighing neat TCE into methanol in a glass volumetric flask. This standard was kept in the freezer. Each day a series of aqueous standards were made from the primary standard by serial dilution using glass pipets and volumetric flasks. These standards ranged in concentration from 3.0 to 0.006 mg/L.

The method detection limit (MDL) for TCE (0.0026 mg/L) was determined by using the protocol described in the *Federal Register* (1984).

Data Analysis

For each time, analysis of variance (ANOVA) tests were conducted on the concentration data to determine if the tubing had any significant effect (at the 95% confidence level) on the TCE concentrations when compared with control values. When significant differences were found, Fisher's protected least significant difference test was performed to determine which tubing materials were significantly different from the controls and each other.

The mean normalized concentrations of TCE were obtained by taking the mean concentration of a sample exposed to a tubing for a given sampling time and dividing it by the mean concentration for the control samples for the same time. Thus, a mean normalized value of 1.00 represents no loss of TCE for a given tubing and time.

The total amount desorbed (mg) from each tubing was determined in three steps: (1) plotting the concentration desorbed vs. time; (2) taking the sum of the areas under lines drawn from time 1 to time 2, time 2 to time 3, etc.; and (3) multiplying the total area by the flow rate.

The total amount sorbed (mg) by the PP1 tubing was determined in four steps: (1) subtracting the concentration (mg/L) after passing through the tubing from the control concentration (mg/L); (2) plotting the difference (mg/L) vs. time; (3) taking the sum of the areas under the lines drawn from time 1 to time 2, time 2 to time 3, etc.; and then (4) multiplying the total area by the flow rate.

The raw data and the results of the statistical analyses for these studies can be found in Parker and Ranney (1997a).

Results and Discussion

Sorption/Leaching Studies

Sorption Findings for the First Study

Figures 2, 3, and 4 show the mean normalized concentration of TCE in well water that was pumped through 10-, 50-, and 100-foot lengths of PVDF, P(VDF-HFP), PP1, PP2, and LDPE tubings at a flow rate of 100 mL/min. The two fluoropolymers, PVDF and P(VDF-HFP), were clearly the least sorptive tubings tested. Sorption of TCE by these two fluoropolymers was always 5% or less, even for the 100-foot sections. For the other polymers, sorption was minimal (<10%) for the 10-foot sections of tubing but became substantial when longer tubings (50 and 100 feet) were used. Losses were greatest initially but then approached some type of equilibrium value. The large losses observed initially were most likely due to sorption on the polymer surface. The residual losses that were found at equilibrium most likely resulted from diffusion through the polymer matrix.

While initial losses of TCE were substantial (20 to 35%) for the 50-foot and 100-foot lengths of LDPE, they were reduced to only 5% after four hours of pumping for the 50-foot lengths or 24 hours for the 100-foot lengths. However, losses were much greater for the two plasticized polypropylene tubings (PP1, PP2), both initially and at equilibrium. We are not certain what accounts for the pronounced dip in the

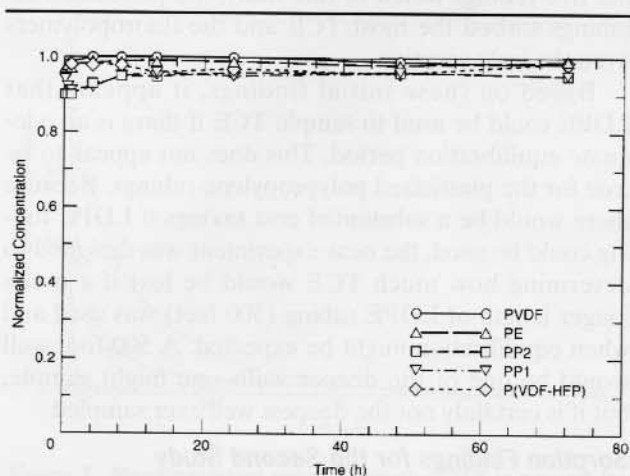


Figure 2. The normalized concentrations of TCE in water pumped through 10 feet of tubing at 100 mL/min.

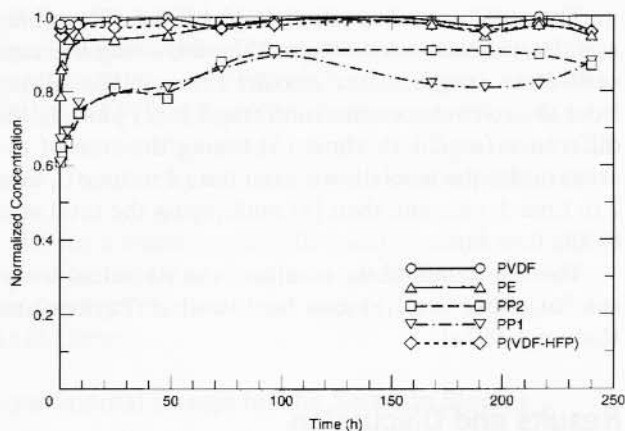


Figure 3. Normalized concentrations of TCE in water pumped through 50 feet of tubing at 100 mL/min.

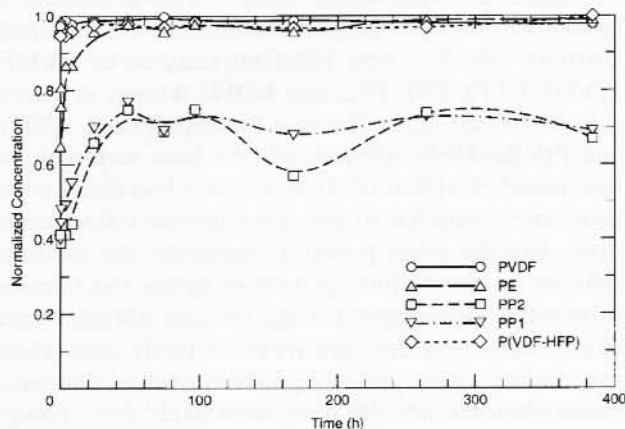


Figure 4. Normalized concentrations of TCE in water pumped through 100 feet of tubing at 100 mL/min.

curves around 150 to 200 hours for the 50-foot length of PP1 or 100-foot length of PP2. It may be due to breakthrough of the TCE through the tubing walls to the atmosphere.

These results agree well with the results from our static study (Parker and Ranney 1997b). That is, among the five tubings tested in this study, the plasticized PP tubings sorbed the most TCE and the fluoropolymers were the least sorptive.

Based on these initial findings, it appears that LDPE could be used to sample TCE if there is an adequate equilibration period. This does not appear to be true for the plasticized polypropylene tubings. Because there would be a substantial cost savings if LDPE tubing could be used, the next experiment was designed to determine how much TCE would be lost if a much longer length of LDPE tubing (500 feet) was used and when equilibration might be expected. A 500-foot well would be one of the deeper wells one might sample, but it is certainly not the deepest well ever sampled.

Sorption Findings for the Second Study

Figure 5 shows the mean normalized concentration of TCE in water that was pumped through 500 feet of

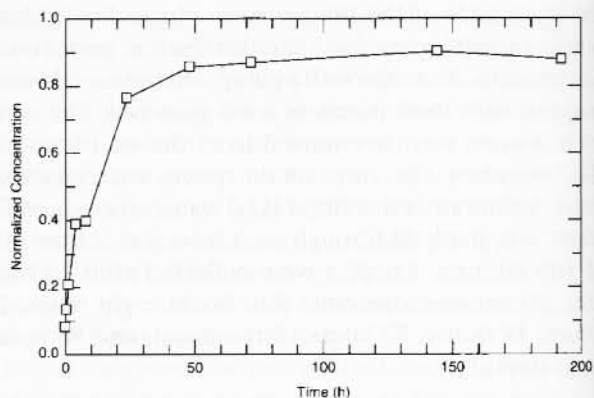


Figure 5. Normalized concentration of TCE in water pumped through 500 feet of tubing at 100 mL/min.

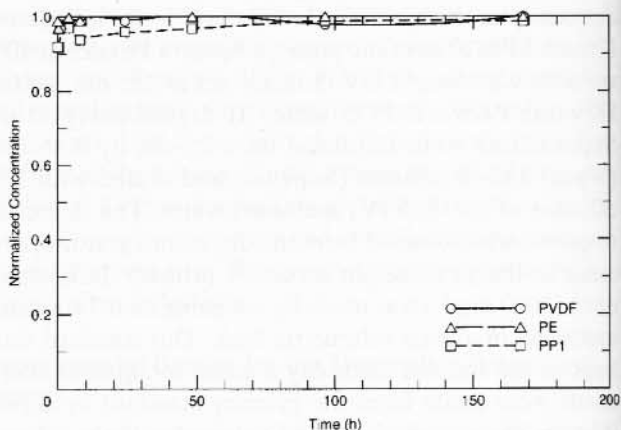


Figure 6. Normalized concentration of TCE in water pumped through 100 feet of tubing at 1 L/Min.

LDPE tubing at a flow rate of 100 mL/min. For this length of tubing, sorption was much more pronounced initially, with 92% loss after 10 minutes. Equilibration appears to occur after 48 hours of pumping with residual losses of 10 to 15%.

When we looked for a relationship between the pumping time (t) and the relative concentration (C_t) for the various length tubings, we found that for the longer tubings or contact times, sorptive losses can be adequately described by a log function (Parker and Ranney 1997a).

Sorption Findings for the Third Study

In this study, we wanted to determine what effect a faster flow rate had on sorption. Because of physical limitations we had with the delivery system, only three tubings could be used in this study. We selected PP1, LDPE, and PVDF. We eliminated PP2 because its performance was similar to PP1 and P(VDF-HFP) because its performance was very similar to PVDF.

Figure 6 shows the mean normalized concentration of TCE in water that was pumped through 100-foot sections of these tubings at a flow rate of 1 L/min. Statistical analyses indicated that the concentrations of TCE in samples pumped through the PP1 tubing were

significantly lower than the controls (Parker and Ranney 1997a). However, these losses were always less than 10%. In general, concentrations of TCE in water pumped through the PVDF and LDPE tubings did not differ significantly from the controls (Parker and Ranney 1997a).

When we related sorption with contact time, we found that there is good agreement between the expression for the relative concentration of TCE in water pumped through 100 feet of LDPE at 1 L/min and water pumped through 10 feet of LDPE tubing at 100 mL/min (Parker and Ranney 1997a). In both cases, the contact time was one minute. Thus, it appears that contact time can be used to predict losses.

We used the relationships derived for the five- and 10-minute contact times at the 100 mL/min pumping rate to predict what the losses might be for TCE-contaminated water pumped through 500 and 1000 feet of LDPE at 1 L/min, after allowing the tubing to equilibrate for various times (Table 1). For 500 feet of tubing, the estimated losses would be 10% after allowing four hours for equilibration. However, for the 1000-foot lengths, a much longer equilibration time would be required to see an equivalent loss, approximately 18 hours. Thus, we predict that long lengths (500 feet and greater) of LDPE cannot be used to sample TCE at a flow rate of 1 L/min.

Table 1
Predicted Normalized Concentrations
of TCE Following Pumping at 1 L/min
for Various Pumping Times

Tubing Length (feet)	Sample Contact Time (min)*	Pumping Time (hour)	Predicted Normalized Conc. TCE
500	5	2	0.88
		4	0.90
1000	10	2	0.81
		4	0.84
		8	0.87
		12	0.89
		18	0.90

*Time for water to pass through tubing.

General Discussion on Sorption

The question that remains is: what would happen to other analytes that are more readily sorbed by the LDPE tubing, such as we (Parker and Ranney 1996) previously observed with tetrachloroethylene and p-dichlorobenzene? Based upon those findings, we expect that losses of those analytes would be substantially greater at equilibrium than what we observed with TCE. However, this is unproven at this time.

Because losses were always much greater for test solutions exposed to the flexible fluoropolymers than the rigid fluoropolymers in Part 1 of this study (Parker and Ranney 1996), we were surprised to find that, in

this study, there was no significant difference between the amount of TCE sorbed by the rigid PVDF tubing vs. the flexible P(VDF-HFP) tubing (Parker and Ranney 1997a). We suspect that we would have detected some differences between these two materials if a longer length of tubing or a more sorptive analyte had been tested. However, this also remains unproven.

Leaching Findings

We did not detect any spurious peaks, using reversed-phase HPLC analysis and a UV detector, in any of the samples that had been pumped through any of the tubings used in these experiments, even the longest lengths or the slower flow rate. However, we should mention that, because the concentration of TCE was in the low mg/L range, these analyses were not highly sensitive. Also, UV detectors are not universal detectors and many organic constituents that might be leached would not be found using this type of detector. UV detectors are most sensitive for conjugated bonds and sulfur-, nitrogen-, bromine-, and iodine-containing compounds (Johnson and Stevenson 1978). Given the brief contact time between the sample and the tubing, we feel that these results agree reasonably well with earlier findings from of this study (Parker and Ranney 1996), where no organic constituents were detected leaching from the PVDF, LDPE, and PP2 tubings and only one constituent was detected leaching from the PP1 and P(VDF-HFP) tubings.

Desorption/Leaching Study

Desorption Findings

Another concern in the monitoring industry is whether there will be any carryover of contaminants if a tubing is used to sample more than one well. Figure 7 shows the mean concentration (mg/L) of TCE that was released into the DI water pumped through three contaminated tubings (PVDF, PP1, LDPE) at a flow rate of 100 mL/min. After 30 minutes, which corresponds to rinsing the tubing with three tubing volumes of water,

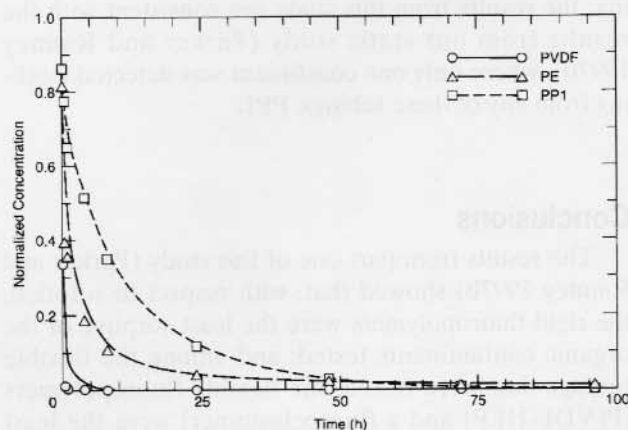


Figure 7. Mean desorbed concentrations of TCE in organic-free water pumped through 100 feet of contaminated tubing at 100 mL/min.

DI water pumped through the PP1 and LDPE tubings contained high $\mu\text{g/L}$ concentrations of TCE (776 and 396 $\mu\text{g/L}$, respectively), while water that was pumped through the PVDF tubing contained only low $\mu\text{g/L}$ concentrations (14 $\mu\text{g/L}$). After eight hours, the TCE concentration in water that was pumped through the PVDF tubing was below the detection level (2.6 $\mu\text{g/L}$). In contrast, both the LDPE and PP1 tubings continued to release high $\mu\text{g/L}$ concentrations of TCE into the DI water for eight to 24 hours and low $\mu\text{g/L}$ concentrations for the remainder of the experiment (four days).

These results show that while contaminants are removed from fluoropolymer tubings much more readily than from other types of tubing, pumping rinse water through the fluoropolymer tubing is not an efficient method for removing the sorbed organic contaminants. Clearly, desorption is a problem for all three materials tested.

For the more sorptive polymers (LDPE, PP1), the relationship between the pumping time and the concentration desorbed for each of the three tubings can be adequately described by a negative log function (Parker and Ranney 1997a).

For the PP1 tubing, we were able to estimate the total amount sorbed after seven days of pumping and the total amount desorbed after four days of pumping. We found that 23% of the total TCE sorbed had been desorbed. While we were able to estimate the total amount desorbed for the PVDF tubing (0.91 mg) and the LDPE tubing (29 mg), we could not estimate the amount sorbed by these two tubings because these losses were not statistically significant.

Leaching Findings

We did not observe any spurious peaks in the DI water that was pumped through either the PVDF, LDPE, or PP1 tubings at 100 mL/min. In this case, the sensitivity of the analyses was much greater because we were looking for low $\mu\text{g/L}$ concentrations. However, our analysis time was not as long in this experiment as in our previous study (Parker and Ranney 1997b) where leaching was observed. Again, given the relatively brief contact time between the sample and tubing, the results from this study are consistent with the results from our static study (Parker and Ranney 1997b), where only one constituent was detected leaching from any of these tubings, PP1.

Conclusions

The results from part one of this study (Parker and Ranney 1997b) showed that, with respect to sorption, the rigid fluoropolymers were the least sorptive of the organic contaminants tested; and among the flexible tubings that were tested, the flexible fluoropolymers [P(VDF-HFP) and a fluoroelastomer] were the least sorptive. The results from these studies show that under dynamic conditions the fluoropolymer tubings

were the least sorptive of TCE, both initially and at equilibrium.

There were instances when a less expensive tubing such as LDPE did not bias the TCE test results. These conditions were when a slow pumping rate (100 mL/min) was used to sample a shallow well (50 feet and less); or when a faster flow rate was used (1 L/min) to sample all but the deepest wells (500 feet and greater). Because earlier findings indicate that some other analytes are more readily sorbed by LDPE, we recommend that there be additional testing with those analytes.

In this study, we did not detect that any organic constituents leached from any of the five tubings tested when either a slow rate (100 mL/min) or a moderately fast rate (1 L/min) was used to pump water through the tubings. Given the relatively brief contact time between the sample and tubing, we feel that these results agree with our previous static study (Parker and Ranney 1997b), where only one constituent was detected leaching from two of these tubings [PP1, P(VDF-HFP)].

The data from this study clearly show that desorption of sorbed organic solutes, such as TCE, can be a problem if slow-flow pumping is used. This is true whether the tubing is relatively nonsorptive (e.g., PVDF) or highly sorptive (e.g., PP1). Furthermore, pumping organic-free water through these tubings at this flow rate is not an effective way to remove TCE contamination.

Recommendations

If sampling an unknown organic contaminant at a slow flow rate (100 mL/min), we would recommend using a fluoropolymer tubing, preferably a rigid fluoropolymer. It may be feasible to use a less expensive tubing, such as LDPE, to sample an unknown organic contaminant if the well is relatively shallow (<50 feet) or if a faster flow rate is used (e.g., 1 L/min). However, further testing with more hydrophobic contaminants is needed to verify this.

To prevent problems with carryover of contaminants when slow-rate sampling is used, the tubing should either be dedicated to the well or else decontaminated prior to using it in another well (assuming a suitable decontamination method is available). Also, if concentrations in a well decrease dramatically, it may be prudent to remove the pump from the well and decontaminate it prior to sampling.

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