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Uptake of Nitroaromatic Compounds by Polymeric Tubing

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Abstract

The type of polymeric material used in the manufacturing of tubing determines its strength, elasticity, and durability. Tubing made of polymeric material is commonly used for analytical work because it is readily available, inexpensive and can be relatively inert. Polymeric tubing is used in many sampling applications for explosive compounds. A major concern is the uptake of the explosive compounds into or onto the tubing during sampling. Because of the reactive nature of explosives, it is important that as little of the detectable explosive as possible is lost by tubing uptake. It is also important that nothing leaches out of the tubing to interfere with the detection of explosives.

High Performance Liquid Chromatography (HPLC) is commonly used for the analysis of trace levels of explosive compounds in the range of parts per billion (ppb) to parts per million (ppm). This study attempts to determine which types of polymers are most conducive to sampling applications where large volumes of dilute explosive solutions are collected through a length of tubing for analysis. This was determined by analyzing the amount of explosive lost from solution per cm^2 of tubing in solution. It was determined that tubing made of polyethylene, teflon, polypropylene, or KYNAR[®] is recommended for dilute trinitrotoluene (TNT) solution analyses. Tubing made of polypropylene, PHARMED[®], KYNAR[®], or polyethylene is recommended for analyses involving dilute explosive solutions of RDX. Tubing made from polyurethane, TYGON[®], nylon, vinyl, gum rubber, or reinforced PVC are not recommended because they leach contaminants into solution that may interfere with HPLC analysis of explosive peaks.

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Uptake of Nitroaromatic Compounds by Polymeric Tubing

Introduction

The detection of trace amounts of chemicals is often a significant challenge, particularly when the target compound is prone to losses during transfer and handling or when large dilution effects reduce the concentration of the analyte. Recently, Sandia National Laboratories developed methods to detect trace amounts of explosives from marine environments. The explosive contamination in aqueous environments generally originates from one of two sources: environmental runoff or unexploded ordnance. Unexploded ordnance, and other devices containing explosives, leak trace amounts of the explosive into the environment (1,2). This leakage occurs when the explosives contained in the device permeate through polymeric seals or diffuse through joints such as press fit seals and threaded seams (3). Contamination due to environmental runoff occurs where contaminated groundwater discharges into the ocean (4). Upon reaching the ocean, the contaminants experience a significant amount of dilution. Concentrations of explosives in the 10 to 100 parts-per-quadrillion ($1:10^{15}$) range have been observed by this laboratory.

Sandia National Laboratories developed methods to extract and analyze trace amounts of explosives from marine environments. Sample collection and transfer often require seawater to be pumped from depth with polymeric tubing. Once the sample is collected, concentration techniques are used to obtain a sufficient mass of analyte for identification and quantitation. However, it is essential that a minimal amount of analyte is lost during sample collection for this subsequent analysis.

The polymer from which the tubing is made determines its characteristics (5). In addition to possessing sufficient strength, elasticity, and durability for this application, several other qualities should be considered. One important consideration is the possible interaction of the analyte with the tubing polymer. The accurate analysis of seawater contaminants is dependent on the preservation of the analyte during collection. One factor which may contribute to the loss of sample is the interaction of analyte to the polymer surface of the tubing (5). The amount of analyte lost can be minimized by pumping the water at fast flow rates to reduce the contact time of the analyte with the tubing. However, the tubing used to pump seawater is a variable that can be easily manipulated, and the determination of whether the polymer has an affinity for the analyte can be assessed.

In addition to initial detection and quantitative considerations, other consequences of polymer-analyte interactions should be discussed. Depending on the kinetics of these interactions, the polymer may release the absorbed material at a later time, thus contaminating future samples (6). The effect is a carry-over of explosive that results in

its detection at a site where no analyte is actually present. Another consideration that is just as important as analyte loss and/or carry-over is the leaching of plasticizers or other contaminants into the sample stream. For example, polyvinyl chloride (PVC) production includes the addition of plasticizers to decrease the softening temperature, thus making it more pliable (5). If these plasticizers leached into the sample stream, they may introduce false positives during analysis or obscure sample peaks in analytical detection systems.

The purpose of this study is to test a number of commonly used polymer tubings to determine the amount of TNT and/or RDX that is lost from an aqueous solution as a function of time. This information will be used to determine which of the tested polymers are most conducive to this type of sample analysis.

Experimental Design

Thirteen types of polymer tubing were studied. Gum rubber, NORPRENE[®], teflon, polyurethane, TYGON[®], KYNAR, polyethylene, C-FLEX[®], nylon, vinyl, reinforced PVC (polyvinylchloride), polypropylene, and PHARMED[®] were supplied by Cole-Palmer[®] (Vernon Hills, IL). After measuring the pieces of tubing to determine the exposed surface area, SA, they were submerged in three different aqueous explosive solutions: 1.97 mg/L or parts-per-million (ppm) RDX, 1.62 ppm TNT, and a mixed solution containing 0.81 ppm TNT and 0.985 ppm RDX. Table 1 describes the types and measurements of the tubing used in this study. Equation 1 was used to determine the exposed surface area of tubing used in this study.

Table 1. Tubing Measurements

Tubing	Inner Diameter (mm)	Outer Diameter (mm)	Water SA (cm ²) exposed*	Mix SA (cm ²) exposed*	TNT SA (cm ²) exposed*	RDX SA (cm ²) exposed*
Gum Rubber	4.45	7.39	5.44	11.44	11.42	10.78
NORPRENE [®]	4.74	7.69	5.88	11.84	11.72	12.44
Teflon	3.11	6.34	4.32	10.50	9.16	9.57
Polyurethane	6.38	9.48	7.92	16.05	15.10	16.50
TYGON [®]	6.35	9.24	6.50	16.09	15.12	15.55
KYNAR [®]	6.08	9.43	7.69	17.53	16.18	16.82
Polyethylene	6.22	9.43	7.99	16.90	15.79	14.03
C-FLEX [®]	4.70	6.35	5.96	10.48	9.77	9.86
Nylon	6.91	9.48	7.64	15.40	16.13	16.72
Vinyl	6.45	9.38	6.79	17.14	14.23	16.87
Reinforced PVC	6.65	11.48	8.53	21.99	20.06	21.23
Polypropylene	6.18	8.05	6.18	14.04		
PHARMED [®]	6.53	8.96	7.58	13.80		

*The exposed surface area, SA, was calculated by the following:

$$SA = (R_0h + R_1h + R_0^2 - R_1^2) * 2\pi$$

R₀ = outer radius

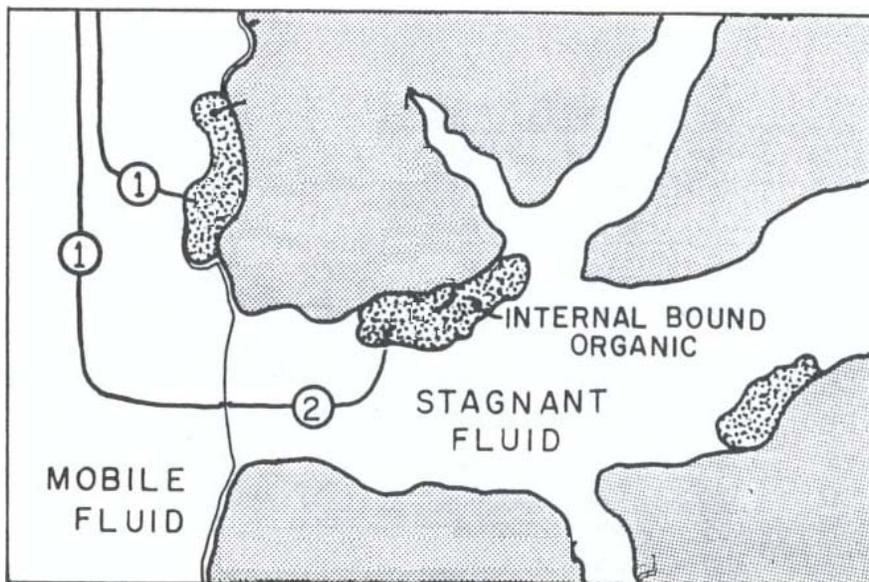
R₁ = inner radius

h = height

(Equation 1)

Because the pieces of tubing were not symmetric, each measurement—inner and outer diameter, length, and thickness—was taken four times and averaged to produce the numbers used in these calculations.

For these calculations to be valid, the polymer surfaces are assumed to be smooth (see Figure 1). If the polymer surface is rough or porous, the actual surface area will be much greater than the calculated surface area. An increase in surface area may affect the amount of analyte uptake by the tubing.



*This figure was modified from L Liang and JJ Morgan, 1990 (7).

Figure 1. Smooth versus Porous Surface

In addition to the three explosive solutions, two sets of controls were used. Twenty milliliters of each of the three solutions were placed in vials without tubing to ensure that any analyte loss was due to interactions with the polymer and not degradation of the explosive or adsorption onto the interior surface of the glass vials. For the second set of controls, each type of tubing was submerged in Millipore water to determine if contaminants, such as plasticizers, leach into water over time.

One set of tubing samples were submerged in 20.0 mL of explosive solution and another set in 10.0 mL of Millipore water. Because only half as much water was used as explosive solution, only half as much of the tubing was submerged in this set of controls in an attempt to preserve the same surface area to volume ratio. A 0.75 mL aliquot of each solution was withdrawn periodically over a 77 hour period as shown in Table 2.

Table 2. Submergence Period Between Sampling

TNT (hours)	RDX (hours)	Mix (hours)	Water (hours)
4	4	1	1
5	5	2	4
6	6	3	24
7	7	4	48
8	8	5	73
9	9	6	144
24	24	7	
28	28	24	
32	32	28	
48	48	48	
77	77	75	

The samples were analyzed using High Performance Liquid Chromatography, HPLC. A Waters™ system (Milford, MA) which included a Model 996 PDA (photodiode array) detector, a Waters™ 717plus Autosampler, and a Waters™ 600E System Controller. Data analysis was performed using Millennium 2010 Chromatography Manager software from Waters™ (Milford, MA).

A Supelco Supelcosil™ LC-18 reverse phase column, dimensions 25 cm x 4.6 mm x 0.5 µm, (part number 58298, Bellefonte, PA) at ambient temperature was used for the separation. A 0.9 mL/min isocratic flow of 30:70 water/methanol at ambient temperature was used for the mobile phase. Sample injection volumes of 150 µL were analyzed from 230nm to 700nm.

Results

Analysis

A one-point calibration was used. The amount of explosive absorbed by the tubing was calculated by subtracting the amount of explosive left in solution from the original solution concentration, as shown by Equation 2.

For example, after submerging Teflon in RDX solution for 24 hours, the concentration of RDX left in the sample can be determined by comparing it with the peak area count of the 1.97 ppm RDX standard after 24 hours. The amount of analyte lost to the tubing is then assumed to be the initial concentration less the concentration analyzed in the sample aliquot. The amount of explosive lost per cm² of tubing submerged is then calculated by converting the concentration (mg/L) to nanograms (ng) of explosive and comparing it to the amount of surface area exposed to solution.

$$A_{\text{RDX}} = \frac{[C_I - (A_{\text{sample}}/A_{\text{STD}}) * (C_{\text{STD}})] * V_I}{\text{SA}} * 10^6 \quad \text{(Equation 2)}$$

A_{RDX} = absorbed ng RDX per cm²

C_I = initial concentration of RDX

A_{sample} = HPLC area counts of the RDX sample

A_{STD} = HPLC area counts of the RDX standard

C_{STD} = concentration of the RDX standard

V_I = injection volume

SA = surface area

There is an inverse relationship between the amount of time during which the polymer is submerged and the concentration of explosive in solution. As the amount of time the tubing spends in solution increases, the concentration of the explosive in solution decreases.

Controls

Analysis of the control RDX and TNT solutions did not show decreasing concentrations over time. Therefore, it is assumed that all analyte loss from the samples is due to interactions with the polymer tubing. It is also assumed that the small aliquots removed for sampling did not perturb the system significantly because each piece of tubing remained submerged in solution.

Analysis of the tubing controls, which consisted of tubing submerged in Millipore water, showed that half of the polymers released one or more contaminants into the water. The identities of the contaminants were not determined. They may have been plasticizers or low molecular weight polymers (8). Gum rubber, polyurethane, TYGON[®], nylon, vinyl, and reinforced PVC leached contaminants that co-eluted with TNT and/or RDX. It was

possible to separately quantitate the contaminant peaks if they absorbed at a sufficiently different wavelength than the analyte peak. TNT absorbs light at approximately 228 nm and RDX absorbs light at approximately 260 nm. Ideally, it is possible to determine the amount of explosive from the co-eluted peak by extracting the chromatogram at the appropriate wavelengths (Figures 2-4), and this method was used to separate the explosive peak from the contaminant peak for gum rubber submerged in RDX solution. In these instances, HPLC peak area counts were determined from a single wavelength as opposed to a range of wavelengths used to analyze other samples

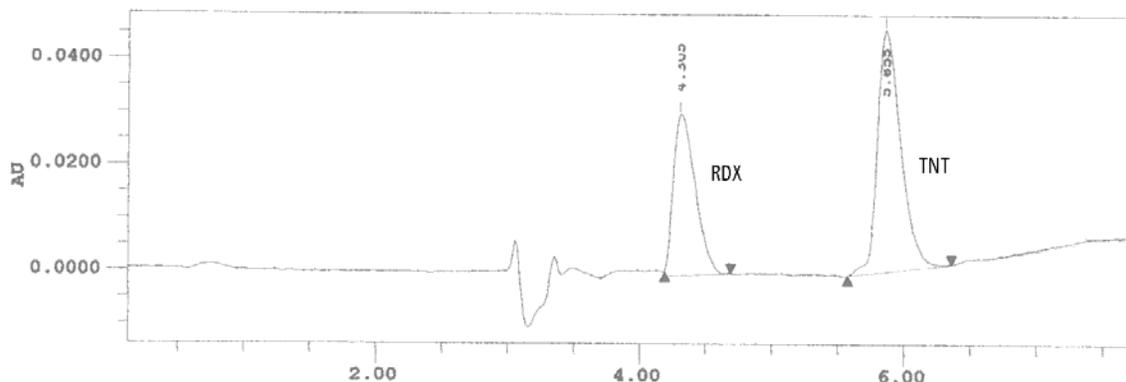


Figure 2. TNT and RDX Standards

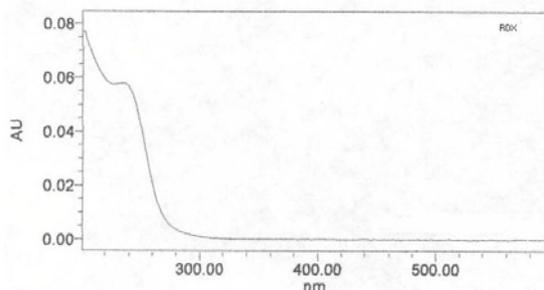


Figure 3. Extracted Spectrum for RDX

These spectra were scanned from 200 nm to 600 nm while the standards and samples in this study were analyzed from 230 nm to 700 nm.

This method of analysis may produce differences in the results seen, but they are assumed to be minimal.

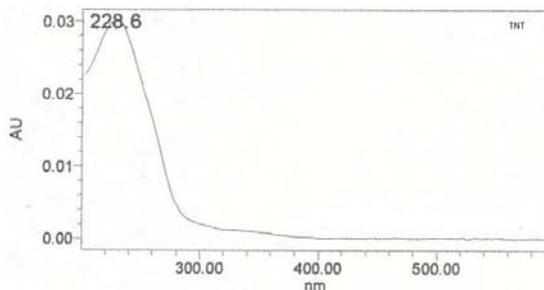


Figure 4. Extracted Spectrum for TNT

Reinforced PVC leaches three contaminants into water that elute close enough to the analytes to interfere with accurate peak analysis (Figures 5 and 6). Although the contaminants from reinforced PVC can be differentiated from the explosive peaks, this is not true for all contaminants. This becomes a problem when contaminant peaks are not considered. Under the assumption that the analytes are the only components eluting at their respective retention times, the presence of co-eluting contaminants will either generate false positives or indicate a higher concentration of analyte than is actually present.

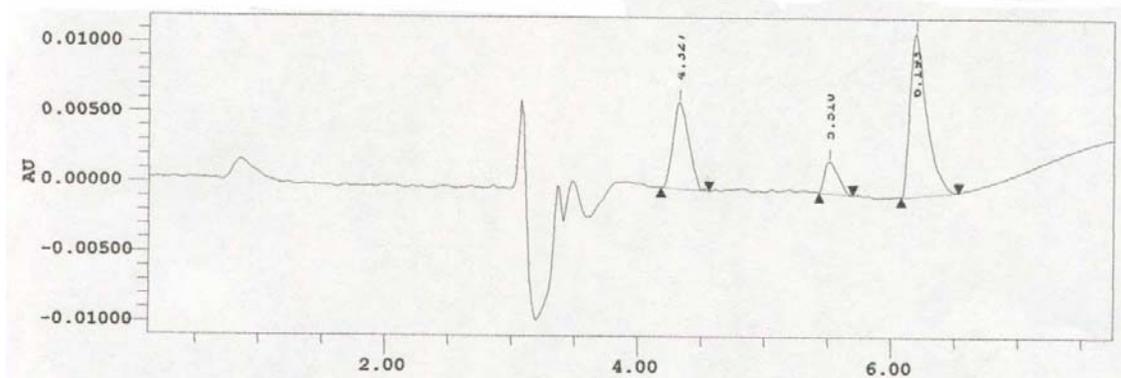


Figure 5. Reinforced PVC after 75 Hours in Millipore Water

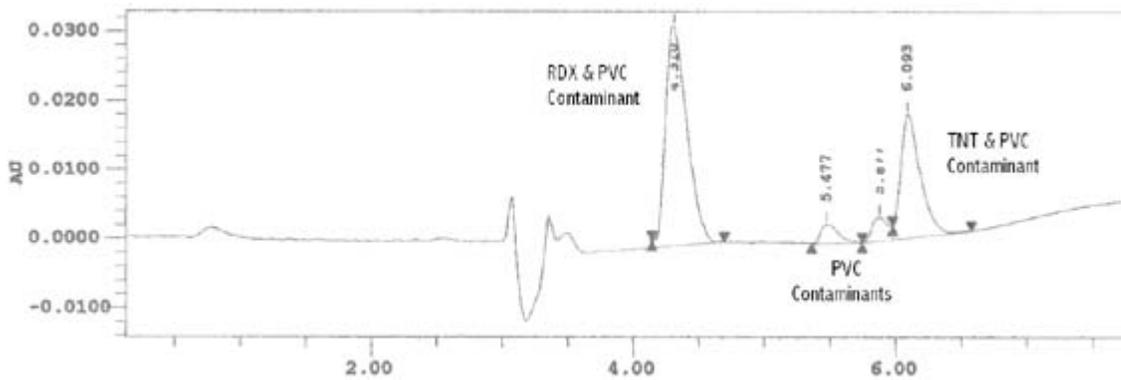
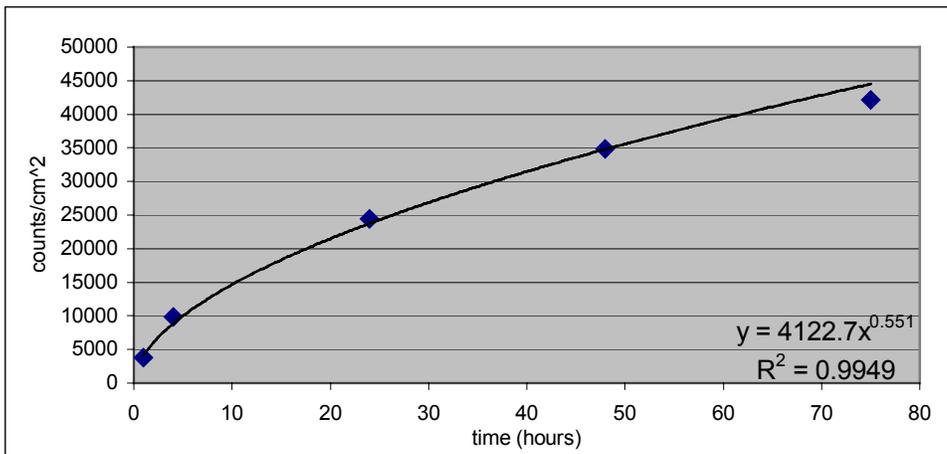


Figure 6. Reinforced PVC after 75 Hours in Mixed RDX and TNT Solution

When the contaminants absorbed light at similar wavelengths as the explosives, this method of deconvolution could not be used. Instead, a computer-generated model of contaminant elution was created from the Millipore water control samples. Because the sampling times for tubing submerged in water did not coincide with those for tubing submerged in explosive solution, an extrapolation was produced using Microsoft Excel. The sampled points were plotted on a graph (ng/cm² explosive vs. time) and an equation was generated from a best-fit line (Figures 7 and 8).

The equations were used to calculate the amount of contaminant leaching into the samples at the corresponding sampling times. It was assumed that the amount of contaminant released from the tubing in water was similar to the amount of contaminant



released from the tubing in explosive solution. With this assumption, the amount of contaminant released was subtracted from the co-eluted contaminant-explosive peak to determine the HPLC area counts for TNT and RDX.

Figure 7. Vinyl Contaminant in Water Over Time

The vinyl contaminant co-elutes with the TNT peak. Figure 7 shows the amount of contaminant leaching into water over a 75 hour period.

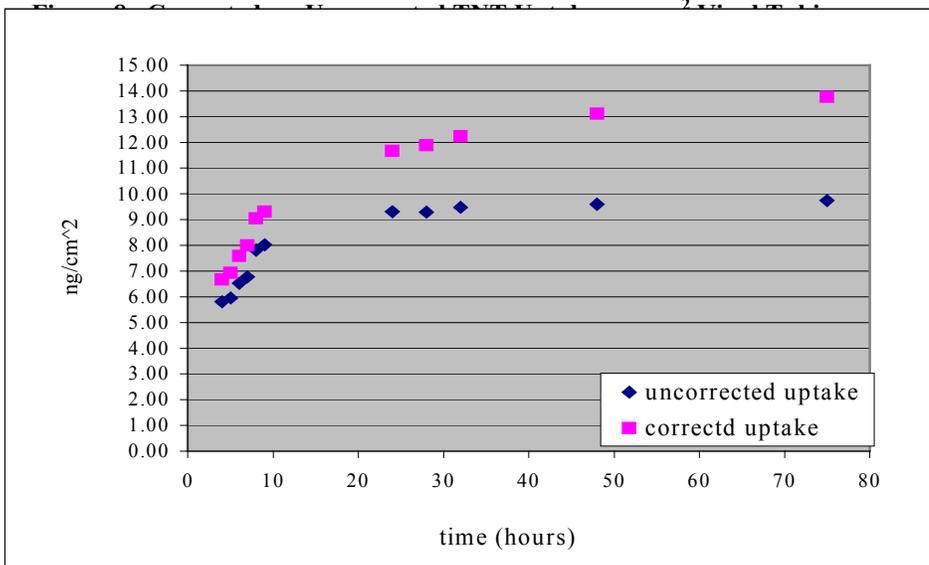


Figure 8 shows the amount of TNT lost from solution for vinyl tubing. Data that has been corrected for the contaminant contribution are shown with data that have not been corrected for contaminant contribution.

The only RDX peak interferences observed were from gum rubber and reinforced PVC. The interference peak was separable from the RDX peak by wavelength for gum rubber (271nm) so there was no need to derive an extrapolated equation to correct for this interference. However, equations from computer-generated extrapolations were used to calculate RDX uptake by reinforced PVC.

The TNT peaks co-eluted with contaminant peaks leached from tygon, vinyl and reinforced PVC. Again, computer-generated extrapolations were used to calculate TNT uptake by these polymers because the co-eluting peaks absorbed light at or near 228nm.

Table 3. Corrected Uptake Equations for TNT and RDX

The first four rows are for TNT and the last row is for RDX. “Y” is the corrected uptake (HPLC counts/cm² and “x” is the sampling time.

Tubing	Equation	Uncorrected Uptake (HPLC counts/cm ³)	Corrected Uptake (HPLC counts/cm ²)
Tygon	$Y=(1.873*10^3)x^{0.1744}$	3720	2254
Vinyl	$Y=(4.123*10^3)x^{0.5510}$	18951	8572
Reinforced PVC	$Y=(1.781*10^2)x^{0.9222}$	5157	2227
Reinforced PVC	$Y=(5.096*10^1)x^{0.7777}$	14464	14428

The amount of contaminant released is not linear over time (Figures 7 and 8). The tapering off of contaminant leaching over time possibly indicates that the solution is approaching equilibrium.

Nylon and vinyl only have one interference peak while polyurethane, tygon, and reinforced PVC released multiple contaminants. Polyurethane and tygon did not leach anything significant into the water before 24 hours. Nylon, vinyl, and reinforced PVC began to leach contaminants into the water within one hour after being submerged.

In addition to the indirect measurement of explosive loss necessitated by these contaminants, other problems may be encountered such as possible reactions with analytes. However, this was not detected in this study. Therefore, it is not recommended that these tubes be used for dilute explosive studies over an extended period of time. The highest concentration of explosive used for this study was 1.97 ppm. The saturation point was not determined for each of the polymers because it would have required a higher solution concentration and more time. Since a maximum loss was not determined, it is assumed that a dilute solution is less than 10 ppm.

Sample Data

Table 3 lists the percent of TNT lost to the polymer tubing after 75 hours. Of the types of polymers presented, polyurethane, Tygon, nylon, vinyl, and reinforced PVC are not recommended for use with dilute TNT explosive solutions because they leach contaminants that co-elute with this analyte.

Table 4 lists the percent of RDX lost to the polymer tubing after 75 hours. Gum rubber and reinforced PVC are not recommended for use with dilute RDX explosive solutions because they leach contaminants that co-elute with RDX.

Table 4. Amount of TNT lost from solution after 75 hours

Tubing Type	ng/cm² available	ng/cm² lost from TNT	% lost from TNT	ng/cm² available	ng/cm² lost from Mix	% lost from Mix
Teflon	1.99	0.20	10%	0.85	0.03	4%
Polypropylene	**	**	**	0.60	0.04	7%
Polyethylene	1.03	0.26	25%	0.50	0.06	12%
PHARMED [®]	**	**	**	0.60	0.20	33%
NORPRENE [®]	1.38	0.39	28%	0.69	0.26	38%
KYNAR [®]	1.00	0.31	31%	0.50	0.10	20%
C-FLEX [®]	1.66	0.67	40%	0.81	0.30	37%
Gum Rubber	1.42	0.86	61%	0.71	0.39	55%
Vinyl	1.14	0.88	77%	0.50	0.38	76%
Reinforced PVC	0.76	0.70	92%	0.39	0.33	85%
TYGON [®]	1.07	1.00	94%	0.51	0.50	98%
Polyurethane	1.07	1.05	98%	0.52	0.52	100%

**The TNT values for polypropylene and PHARMED[®] were not collected.

Table 5. Amount of RDX absorbed by the tubing after 75 hours

Tubing Type	ng/cm² available	ng/cm² lost from RDX	% lost from RDX	ng/cm² available	ng/cm² lost from Mix	% lost from Mix
Polypropylene	**	**	**	0.73	0.03	4%
Polyethylene	1.40	0.18	13%	0.61	0.01	2%
KYNAR [®]	1.17	0.16	14%	0.60	0.04	7%
PHARMED [®]	**	**	**	0.73	0.07	10%
Reinforced PVC	0.98	0.28	29%	0.47	0.10	21%
Teflon	2.05	0.38	19%	1.03	0.12	12%
C-FLEX [®]	2.00	0.45	23%	0.99	0.07	7%
Gum Rubber	1.83	0.46	25%	0.86	0.14	16%
Nylon	1.18	0.43	36%	0.66	0.08	12%
TYGON [®]	1.27	0.49	39%	0.62	0.25	40%
Vinyl	1.17	0.48	41%	0.61	0.18	30%
NORPRENE [®]	1.58	0.98	62%	0.84	0.08	10%
Polyurethane	1.19	0.86	72%	0.64	0.50	78%

**The RDX values for polypropylene and PHARMED[®] were not collected.

Conclusion

The type of tubing used does effect the detection of dilute amounts of explosives in solution. Tubing made of teflon, polypropylene, polyethylene, PHARMED[®] or KYNAR[®] is recommended for dilute TNT solution analyses. Tubing made of polypropylene, teflon, polyethylene, PHARMED[®] or KYNAR[®] is recommended for analyses involving dilute RDX solutions. Tubing made of polyurethane, tygon, nylon, vinyl, gum rubber, or reinforced PVC are not recommended because they release contaminants into solution that may interfere with HPLC analysis of explosive peaks.

Although indirect methods could be used to determine the amount of explosive lost during the experiment, this type of analysis is not ideal because it is an inconvenience and may affect the accuracy of the results. The assumption that the amount of contaminant released in water is similar to the amount released in explosive solution may be not be true in all cases.

It is interesting to note the differences between the percent of explosive loss between the Mix solution and TNT and RDX solutions. The data for most of the polymers shows that a lower percentage of explosive is lost to the Mix solution than either of the TNT and RDX solutions. This is logical because the two analytes will be competing for the same adsorption sites on the polymers so less of each analyte will be adsorbed. If the polymer did not show greater affinity for one analyte over another, the amount lost from the Mix solution should be half of that lost from either the TNT or RDX solution.

Two of the polymers, vinyl, TYGON[®] and polyurethane, do not follow this trend. The same percentage of TNT is lost to vinyl from the Mix solution as from the TNT solution. The same is true for both TNT and RDX with respect to TYGON[®] and polyurethane. This suggests that the loss is due to something other than or in addition to a simple adsorption mechanism like that described above.

Further studies are needed to elucidate the mechanism(s) underlying these results.

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