SEDIMENT QUALITY ASSESSMENT
for the
TIDAL CHRISTINA RIVER BASIN
VOLUME I: REPORT OF FINDINGS

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Preface

The following document contains summarized and reduced analytical data. Copies of laboratory reports are found in the documents: Sediment Quality Assessment for the Tidal Christina River Basin: Volume II: Analytical Data and Sediment Quality Assessment for the Tidal Christina River Basin: Volume III: PCB Congener Specific Analytical Report. Each of these volumes contains over 500 pages of information and was therefore not included as appendices to this report.

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INTRODUCTION

The Delaware Department of Natural Resources and Environmental Control has been designing and instituting new policies and approaches for the use and protection of State natural resources. To this end, initiatives such as brownfield redevelopment, whole basin management approaches and environmental indicators monitoring have been implemented. The Christina River basin will be impacted greatly by all of these initiatives, since it has been subject to numerous anthropogenic impacts for the past 250 years. Therefore, it became necessary to understand the conditions of the river, as reflected in the sediment quality, for purposes of determining a baseline, possible past impacts, and future basin use implications.

A baseline sediment assessment project was performed in the Christina River basin during the summer months of 1995. The purpose of the project was to more completely discern the presence, distribution, and potential impact of environmental contamination within the basin and to determine potential upland source areas that may be impacting the basin. It was therefore considered appropriate to sample the contaminant sink (area of accumulation) of the basin; which is the sediment of the Christina River and its proximal tributaries and marshes.

The area investigated consisted mainly of the tidal portions of the Christina River, White Clay Creek, Brandywine Creek, Newport Marsh, Nonesuch Creek, and Churchmans Marsh. This area is considered to be the final sink for contaminants discharged in the White Clay, Red Clay, Brandywine, and Christina Watersheds.

The overall goal of the project was the determination of impact by brownfield sites and historical activities on the Christina River Basin, specifically as it applies to potential natural resources impacts. The following objectives were proposed to obtain this goal:

- Perform a baseline investigation of general environmental contaminants in the Christina River sediments and proximal associated sub-basin sediments;
- Determine the general distribution of environmental contaminants in the Christina River;
- Assess the potential for bioavailability and ecological impact of the contaminants;
- Assess the likelihood of upstream loading impacts to downstream sediment quality;
- Identify potential upland areas that may be contributing to sub-basin and river sediment contamination.

SAMPLING PLAN AND ANALYTICAL PARAMETERS

In order to determine the existence and character of any trends and/or spatial distributions, a systematic sampling scheme was selected. Gilbert (1987) recommends even-spaced observation points for estimating trends, cycles, forecasting and distribution. Systematic sampling is easy to implement under field conditions and provides uniform coverage of the target population without involving the unintentional bias of sample collectors. Although unpredicted periodicities and cycles can bias estimates of the population mean and total, the project goal was not solely to estimate a mean and variance.

Samples were collected every one-tenth of a mile from the mouth of the Christina River (excluding the first 0.5 mile due to safety consideration around the marine terminal) to approximately 3.5 miles upstream from the confluence of the Christina and White Clay Creek, and

from the mouth of the White Clay to near its confluence with Red Clay Creek. The total distance was approximately 15 river miles. Samples were generally collected from the inside bank side of the river because of the natural tendency for greater sediment accumulation. The sampling areas and locations are shown in Figure 1a - 1c.

SAMPLING METHOD

Samples were collected using a stainless steel Ponar grab sampler or a stainless steel trowel. The sample depths were approximately 1 to 4 inches deep. The sediment was placed in a stainless steel pan for field measurements and distribution to the sample jars. Although several grabs were generally required to provide enough sediment to fill all the appropriate sample jars, extensive homogenization was not done in order to avoid oxidation and other chemical changes that might be induced by contact with the air. Oxidation-reduction (redox) potential and pH were measured in the field while the sediment was still in the collection pan. Further, during the sampling, surface water measures of temperature, pH, and salinity were performed. These surface water measurements were used to ensure that the environmental conditions were appropriate for the bioassay organisms. The organisms may be sensitive to changes in pH, salinity, and temperature such that positive toxicity results could occur if these parameters are outside specific ranges.

The samples were placed in laboratory supplied and prepared bottleware. Multiple bottles with different preservation methods were required for each sample. The sample jars were placed in 40-gallon coolers and kept on ice for shipment to the laboratory for analysis. All samples were labeled and appropriately documented to maintain chain-of-custody.

ANALYTICAL METHODS

The study focused on contaminants that had previously been identified in the Christina River sediments, or might generally be expected to occur in an urban environment. Also included were physicochemical parameters that may influence the distribution of those contaminants and would aid in assessing the speciation, nature, and/or fate of the contaminants. Specifically, the analytical parameters included in the study were as follows.

- Polychlorinated biphenyls (PCBs) analysis using immunoassay techniques.
- Carcinogenic Polynuclear Aromatic Hydrocarbons (PAHc) analysis using immunoassay techniques.
- Heavy metals included; antimony, arsenic cadmium, chromium, cobalt, copper, lead, mercury, nickel, selenium, silver, tin, and zinc on sample acid digestion extracts using atomic absorption or emission spectroscopy in accordance with U.S. Environmental Protection Agency SW846 methodologies.
- Oxidation-reduction potential (ORP) and pH.
- Total Organic Carbon (TOC) analysis on sample portions passing through a one-eighth inch sieve (to remove large plant debris).
- Grain size distribution by classifying total percentage sands and fines (wt/wt basis) by separating the sample down to a 4.0 phi sieve (63 micrometer (μm)).
- Free ion metal concentrations were approximated by simultaneously extracted metals/acid volatile sulfide (SEM/AVS) analyses.

- Major anion analyses for those anions that can form insoluble metal salts, i.e. carbonate, chloride, sulfide, sulfite, sulfate, and phosphate.
- Bioassay methods included the Microtox®, IQ®, and *Hyalella azteca* 10-day acute methodologies. The toxicity tests were run using three different organisms and employed whole sediment and elutriate assays.
- Pesticides analysis on the 12% confirmation samples for PCBs and PAHc.
- Sequential metals extraction to estimate speciation and distribution of selected metals in the sediment matrix. It was performed on twelve of the samples.
- Confirmation of immunoassay techniques was performed by laboratory analyses of approximately 12 % of the samples for PCBs and PAHc.

Sampling and Analytical Uncertainties and Limitations

Within any project of this nature and scope it is understood and expected that some limitations and uncertainties will arise. Prior to the initiation of the sampling episodes several of these were identified and appropriate measures and controls were set in place to minimize their effects. These are summarized below.

- Weather Conditions - During the sampling episodes, the region was in the time of a 100-year drought. Water levels were generally lower than normal. It is uncertain how this may effect the sediment conditions. Little Mill Creek was scheduled for sampling but the flow was diminished to such a degree that most of the sediments were no longer continually submerged. As a result, Little Mill Creek was removed from the sampling plan and sediments sampled were collected from areas always submerged, even at low tide.

- Sample Oxidation - The sediments collected had not been exposed to the atmosphere and may generally have been in a reduced or moderately reduced condition. Upon retrieval and exposure to air, some oxidation may have taken place. This could have had an impact on the results of several of the analytical procedures selected. Sampling teams were instructed to minimize sample handling, reduce sample container headspace, and place samples into coolers immediately upon collection.

- Sample Homogenization - Because of the need to reduce sample handling to reduce the chances for sediment oxidation; the ability to fully homogenize the samples had to be compromised but not wholly negated. Because several laboratories were involved it was imperative to ensure that each laboratory has a sample of similar physical make-up. The sample similarity was grossly evaluated by comparing the percent solids measured by each laboratory.

- SEM/AVS Application - Generally the application of the SEM/AVS ratio method is considered valid only when the AVS is greater than the laboratory detection limit. This puts great limitation to the use of SEM/AVS on oxidized, and possibly moderately reduced, sediments. Further, samples having similar SEM/AVS ratios but different concentrations of SEM and AVS are not similar; i.e. a sample with low AVS and low SEM may not be similar to a sample with high AVS and high SEM even though the ratios are the same. Also, other complexing agents such as organic matter and other anions are not considered in the ratio. Therefore, an SEM/AVS ratio of <1 indicates that metals are not considered to be

bioavailable but a ratio >1 does not necessarily mean the metals are absolutely bioavailable. As a result, the following considerations were employed:

1. The molar or concentrations of both the SEM and AVS need to be evaluated when interpreting the SEM/AVS ratio.

2. SEM/AVS ratios of samples were AVS is not detected (and subsequently reported at the detection limit by the laboratory) should be considered highly questionable. If used at all it should be noted that the ratio is a "greater than" value.

3. Other complexing anions should be considered in determining the potential for the metals to be bioavailable, specifically in oxidized and moderately reduced sediments.

- Sequential Extraction - This methodology will not identify a specific form that a metal will have in the sediment but may aide in categorizing the metals into geochemical fractions. The limitations to a sequential extraction pertain mainly to the effectiveness of the individual extractions and efficiency of metal removal. The method is not entirely or solely specific to each geochemical fraction and individual steps may remove some metals from another fraction. Also, metal re-adsorption onto the remaining solid phase may occur, leading to lower results in one fraction and potentially increased results in latter extracts. These limitations cannot readily be controlled and the uncertainties need to be accepted.

- Immunoassay Soil Methodologies Application to Sediments - The three immunoassay test kits used were designed for use on soils but were applied to sediments. Although no literature was found relating the performance of the soil methods to oxidized and/or reduced sediments, the methods were previously employed by the DNREC in near-saturated soils and responded similar to unsaturated soil conditions. Laboratory confirmation analyses were used on at least 10% of the sediment samples to evaluate the results.

- Immunoassay Methodology Detection Limits - Because the immunoassay soil methodologies have not previously been applied to sediments and unknown matrix interference potentials may occur, the detection of the sample set will need to be evaluated. This is particularly true for the PCB analysis where the extraction procedure was modified to reduce the standard detection limit of 500 µg/kg to 12.5 µg/kg. To check the sediment data set detection limit, a cumulative probability plot of ranked concentration groups was performed. This method is generally employed to determine the median for data sets where high percentages of non-detectable results and/or several anomalous measures make direct numerical determinations inappropriate. But an extrapolation of the cumulative probability curve (or line if semi-logrithmically plotted) to the x-axis was considered to approximate the working detection limit for the sample data set.

- Immunoassay Reporting Units - Virtually all conventional environmental laboratory results are reported on a dry weight basis. The sample aliquots subjected to the immunoassay tests were not dried and therefore the results are wet weight concentrations. This must be taken into consideration if these results are compared to other studies, guidelines, or laboratory results were dry weight measures are used.

- Field Measurements - The pH and ORP measurements will be obtained using hand held instruments. Many field conditions can adversely effect the response of these instruments. Therefore, only readings that are stable and relatively unchanging will be recorded.

RESULTS and DISCUSSIONS

The presentation and discussion of the results are summarized in four sections: 1) data usability, data manipulations and statistical methods; 2) physiochemical characteristics; 3) analytical results; 4) toxicity results; and, 5) sediment quality assessment. Graphical representations of the analytical results are provided in the appendix.

data usability, data manipulations and statistical methods

Prior to the interpretation and evaluation of the analytical data, including field readings and screening methods, the results were checked for usability and summary statistics reviewed. More specifically, this included: 1) determining the comparability of results between the different laboratories, i.e. agreement between percent solid; 2) determining how to incorporate non-detection results in the data set; 3) identifying and using outliers; 4) preparing general summary statistics, and; 5) determining if normal distributions occur in the raw data sets.

Due to potential depositional differences; i.e., marsh and river environments, the sample results obtained from Churchman's Marsh, Nonesuch Creek, and Newport Marsh were handled separately from the Christina River, Brandywine Creek, and White Clay Creek.

The immunoassay test pesticides results had apparent matrix interference at the low end of the calibration scale and was considered to be invalid. The twenty samples sent for PAHc, PCB, and pesticide laboratory confirmation analyses were the only pesticide results obtained.

laboratory comparability

The analytical results supplied by NET, Thorofare and EA Laboratories were assimilated extensively during the data interpretation. Therefore, it was imperative to generally assess the aliquots sent to each of the two laboratories to ensure comparability of the results. The reported percent solid was used for this purpose. Of the total data set, 65 samples had percent solids performed by each laboratory. Samples that varied greater than 20% were considered marginally comparable and variation of 25% and greater were considered not directly comparable. Seven of the 65 samples had percent solids report at greater than 20% difference. These were sample numbers 5, 17, 21, 85, 101, 105, and 109. Of these seven, samples 17, 101, 105, and 109 varied by greater than 25%. The correlation coefficient between the percent solid analyses between the laboratories was 93%.

Two samples from the marshes were found to be marginally comparable. These were samples CM-5 from Churchman's Marsh and NS-15 from Nonesuch Creek. The correlation between the laboratories for the marsh samples was 98%.

It was therefore concluded that the majority of the data between labs could be commingled with no adjustment. The noncomparability of the nine samples was considered to be due to the inability to adequately homogenize the samples during collection. Caution was used in applying assimilated data in the marginally comparable samples.

The sample 17 aliquot supplied to EA Laboratories was reported to contain only 9% solids. This low percentage of solids made the results unusable. Therefore, the TOC, sulfide, AVS, and SEM values were not used in the study.

use of non-detectable data

The main decision in evaluating the non-detectable results was to determine whether data replacement or data deletion methods were to be employed. The replacement option chosen, if

applicable, was to use one half the reported sample detection limit in data interpretation. It should be noted that detection limits varied between samples for many of the analytical parameters and may have an influence on any statistical evaluation. If the deletion of data was chosen, the detection limit value preceded by a less than symbol (<) would be shown in all appropriate data tables but would not be included during any statistical interpretation.

non-detected metals data

The technique derived to determine whether replacement or deletion was to be used was a nonparametric ranking with a subsequent cluster evaluation. The technique was run several times using different ranking factors. The ranking factors used were geographical location, grain size distribution, selected analytes, and TOC. Arsenic, chromium, copper, iron, manganese, nickel, and zinc were detected in all samples and therefore were not subject to the technique but were used as the selected analytes. Antimony contained very few detectable results therefore deletion was selected. Of the remaining metals (cadmium, cobalt, lead, mercury, selenium, and tin) only one cobalt and two mercury values were detected in the White Clay Creek samples. Therefore, these samples were not included in the technique and the deletion option was chosen. For consistency in handling the tributaries the Brandywine Creek samples were also not included in the technique. Further, due to the low number of samples per analyte (3 to 6) from the Brandywine Creek, deletion was used when all samples were reported as non-detect for an analyte whereas the replacement option was chosen for analytes when the majority of the samples had detection.

The analyses showed a very strong relationship of non-detectable results with geographical location for cobalt, mercury, selenium, and tin. All of the nondetected values for cobalt, mercury, and tin occurred in samples #69 and greater (upstream samples beyond the Newport area). No selenium was detected in samples #69 and greater and was detected 50% of the time in samples #1 through 65. Deletion was selected for these metals except for selenium. The replacement option was used for the samples 1 through 65.

Cadmium and lead were found correspond strongest with the zinc concentration. Cadmium was detected in only two samples when the zinc concentration was less than 475 ppm. Lead was not detected when zinc was less than 125 ppm, except for one sample. As further evaluation, a regression was performed between the grain size adjusted cadmium and grain size adjusted zinc and between the grain size adjusted lead and grain size adjusted zinc. The correlation coefficients were 0.90 and 0.98, respectively. Also, predicted Cd and Pb concentrations calculated by the regression equation were at or below the reported sample detection limits for the samples with laboratory nondetected values. Therefore, due to the significant correlation between Cd and Zn and between Pb and Zn and the predicted values agreeing with the sample detection limits, it was justified to replace the non-detected values with a value of one half the detection limits.

For the marsh sediment samples correlation was also found with zinc, and iron, similar to that of the river samples. Analytical parameters with less than 40% nondetected results were subject to replacement of the nondetected results with one half the sample detection limit. The parameters with greater than 40% nondetected results had the nondetected results removed. The analytes subject to the removal method were antimony, cadmium, mercury, and selenium.

non-detected organic data

Similar nonparametric techniques were applied to the PAHc and PCB results as the metals data. No significant correlations were revealed for the non-detected results. However, the

highest concentrations of organic compounds did coincide with geographical locations suspected to contain source areas. Due to the widespread presence of the PAHc and PCB compounds, it was decided to replace the non-detected data with one-half the detection limit. The cumulative probability plots for determining the validity of the desired detection limits revealed a sample data set detection for PAHc of 41.5 µg/kg wet weight and a PCB detection limit of 3 µg/kg wet weight. Therefore, the reported detection limits of 60 µg/kg and 12.5 µg/kg for PAHc and PCBs, respectively was considered valid. Only twenty samples were laboratory analyzed for pesticides. Due to the small data set, deletion was used for nondetected pesticides.

use of outliers

Considering the size of the data set, it was expected to contain statistical outliers. However, it would not be possible to initially determine whether individual outliers were present due to natural variation, anthropogenic contamination, or sample handling. Therefore, potential outliers were kept within the data set and removed only on an individual basis if the causation of the outlier was determinable.

physiochemical characteristics

The physiochemical parameters evaluated for the sediment samples included pH, ORP, TOC, sulfide, sulfite, sulfate, total phosphate, chloride, carbonate, percent solid, and percent grain size at 2mm (for removing debris), 500µm, 125µm, 63 µm, and < 63µm. The grain size distribution and pH measurements were not evaluated on the marsh sediments. The physiochemical characteristics are summarized as follows.

- The sediment pH measurements were between 6.3 and 7.7 and were considered neutral.
- The redox potential, as measured in millivolts (mV), was generally negative, indicating reduced or moderately reduced conditions. Positive results were seen in samples two river samples and three White Clay Creek.
- The TOC values showed a distinct trend. From the mouth of the Christina River to the mouth of Brandywine Creek, samples 1 - 11, the TOC increased. The percentages declined until sample 85, along Artesian Marsh, where the percent drastically increased and then continued to decrease upstream. Percent TOC in the Brandywine Creek decreased with distance upstream. White Clay Creek had low TOC levels.
- The sulfide and sulfate, being oxidation-reduction couples, generally had inverse trends. The sulfate concentration was greater than the sulfide concentration near the mouth of the river, significantly greater from sample 20 - 69, Wilmington through Newport, and up the White Clay Creek. As expected the marsh samples contained higher sulfide concentrations than the river and creek samples.
- The chloride concentrations contained some slightly elevated values in samples 1 - 20 and 80 - 100 with one anomalous peak at sample 53, which was one order of magnitude greater than the surrounding samples.
- The total phosphate appeared to be higher in the areas of Wilmington and further upstream past Churchman's Marsh. White Clay Creek and the Newport Area exhibited lower phosphate concentrations. The marsh samples exhibited the highest phosphate concentrations.
- The Grain Size distribution indicated that 72.5 percent of the samples (29 of the 40) exhibited greater than 50% total fines (as defined by the sum of the percent 63µm and the percent

<63µm for each sample). The samples containing less than 50% fines from the Christina River were 29, 61, 73, 81; from Brandywine Creek B13, B16, B19; and all four samples from White Clay Creek, 129 - 145. Grain size distributions were not determined for the marsh samples, however they were clearly composed mainly of fine grained material.

- Interrelationships between TOC, ORP, grain size, and the major anions were identified. Generally, increased TOC and major anion concentrations coincided with decreased grain size and ORP measurements.

As stated above, several empirical relationships are exhibited in the data. The most prominent relationships are the noted between sulfide, sulfate, ORP, and TOC and the findings are briefly discussed below.

Since sulfide and sulfate are redox couples, their concentrations should be dependent on the redox potential of the surrounding environment and trend inversely; i.e. in reducing conditions the amount of sulfur as sulfide should be greater than the amount in sulfate and as the redox potential becomes more oxidized the sulfide concentration will decrease and the sulfate will increase. This relationship was demonstrated in the samples and is graphically expressed in Figure 2. In this figure the ORP range was divided into 5 equal increments of 40 mV each. The ratios of sulfide to sulfate in each ORP increment were averaged and plotted versus ORP. The river and marsh samples were plotted as separate series.

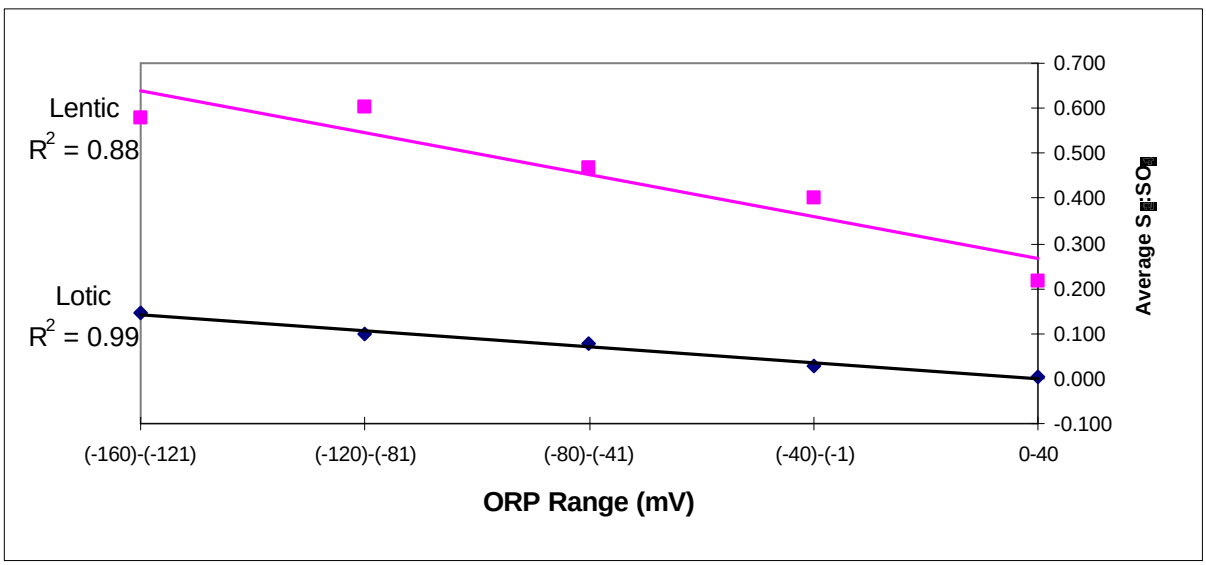

Figure 2. Incremental Average Plot of Sulfide:Sulfate Ratio vs. Redox Potential.
(lentic refers to marsh environs and lotic to river environs)

As is clearly demonstrated, the sulfide:sulfate ratio decreases as the redox potential became more positive. The best-fit linear expression indicates the high degree of correlation. Because of the high goodness-of-fit for the ratio as compared to the ORP range, the sample handling must not have caused significant redox changes overall. These empirical and general relationships may not be applicable at individual sample locations such that one measure is a predictor of the other parameters, but general sediment conditions may possibly be deduced if not all of the parameters are known.

The percent TOC was also shown to be associated with the sediment sample redox potential. Figure 3a shows spatial distributions of both TOC and ORP (as a measure of the redox

potential) versus the sample location. The White Clay Creek samples (#s 129 - 145) are also included in this plot. A regression plot of TOC versus ORP is provided as Figure 3b. Although the TOC and ORP are not directly predictive of each other, the plot visually demonstrates the association of the two parameters.

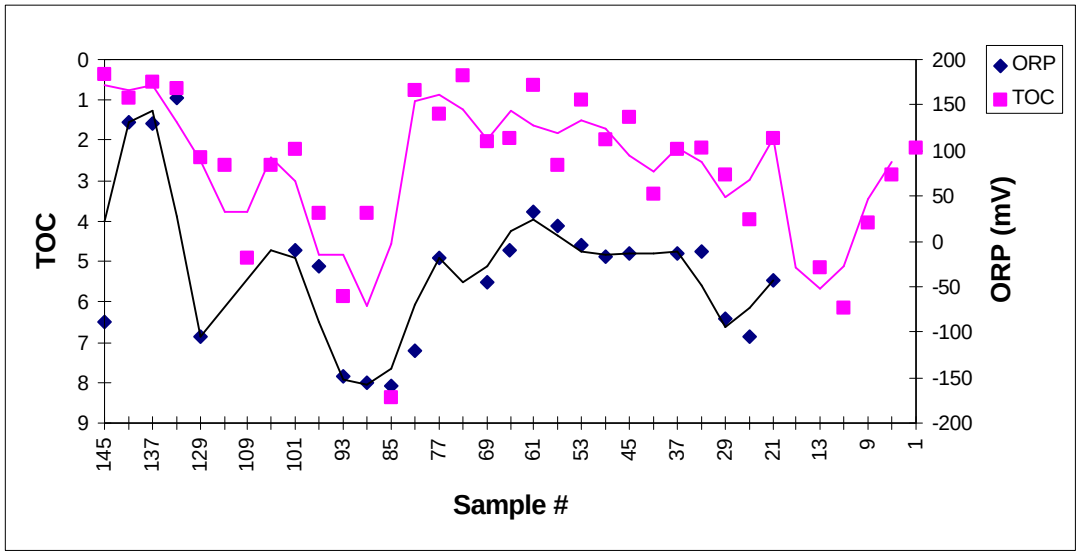

Figure 3a. Trends of Percent TOC and Redox Potential (as measured by ORP) using 2 point moving average. (Note: TOC axis is reversed for clarity)

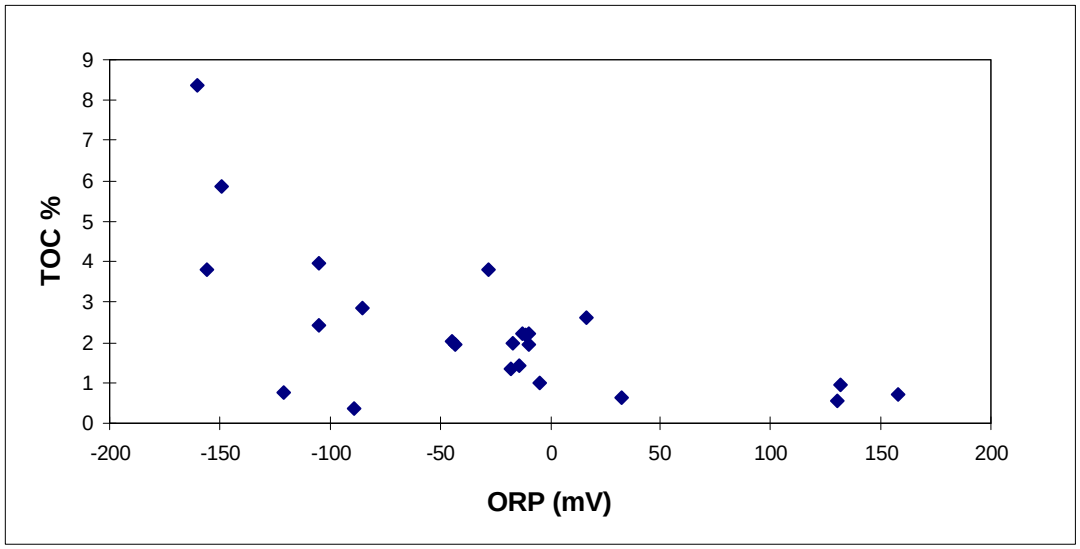

Figure 3b. Scatter diagram of Percent TOC and Redox Potential (as mV ORP).

analytical results

The analytical parameters evaluated for the sediment samples included: antimony, arsenic, cadmium, chromium, cobalt, copper, iron, lead, manganese, mercury, nickel, selenium, tin, zinc, pesticides, PCBs, and PAHc. Further, a sequential extraction and corresponding metals analyses was performed in an attempt to identify the speciation and/or partitioning of the metals in the sediments.

Overall, the marsh samples had a larger percentage of nondetected analytes except for cobalt and PAHc. The mean and median values between the river and marsh samples appear to be similar and are within the same orders of magnitude. However, considering the greater percentage of nondetected results and the fewer number of samples, the statistical significance of

the similarity was not determined. Further, the three marshes may not be comparable and may be significantly different from each other. As mentioned previously, within the river and creek samples arsenic, chromium, copper, iron, manganese, nickel, and zinc were detected in all samples. Iron, zinc, and PAHc were detected in all marsh samples.

heavy metals results

The following tables (Tables 3 and 4) contain the summary statistics for the analytical results. Table 3 includes the riverine samples, from the Christina River, Brandywine Creek, and White Clay Creek, while Table 4 summarizes the marsh samples of Nonesuch Creek, Newport Marsh, and Churchman's Marsh.

Table 3. Summary Statistics of Sediment Analytical Results for the Christina River, Brandywine Creek, and White Clay Creek.

Analyte	Units	Mean	Median	Min.	Max.	%nd*
Sb	mg/kg	26.82	26.45	18.2	46.6	73
As	mg/kg	9.88	7.78	0.73	43.8	0
Cd	mg/kg	2.0	2.0	0.5	5.24	46 R
Cr	mg/kg	34.2	37.9	6.76	86.7	0
Co	mg/kg	13.2	13.1	4.87	24.3	38
Cu	mg/kg	37.35	33.6	3.83	161	0
Fe	mg/kg	17346	16300	4549	32900	0
Pb	mg/kg	67.5	79.4	36.5	171	27 R
Mn	mg/kg	539	473	95.6	1520.	0
Hg	mg/kg	0.46	0.44	0.053	1.6	22
Ni	mg/kg	22.5	22.0	6.09	41.4	0
Se	mg/kg	0.64	0.58	0.25	1.4	47/24 R**
Sn	mg/kg	18.8	18.0	13.0	32.9	46
Zn	mg/kg	440	296	34.7	1140	0
Methoxychlor	ug/kg	170.25	150.0	1	430	29
DDE	ug/kg	29.62	14.00	1	130	36
PCBs	ug/kg	57.5	17.8	6	3115	42 R
PAHc	ug/kg	1987	957	30	21600	11 R

1 - not applicable

2 - replaced w/ 1/2 detection limit

* - 1 sample equals approx. 3%

** - 47% nd / 24% replaced

Table 4. Summary Statistics of Sediment Analytical Results for the Churchman's March, Nonesuch Creek, and Newport Marsh.

Analyte	Units	Mean	Median	Min.	Max.	%nd*

Sb	mg/kg	nd	nd	nd	nd	na[1]
As	mg/kg	4.37	3.98	0.4	12.0	39 R[2]
Cd	mg/kg	3.73	3.73	2.2	5.25	93
Cr	mg/kg	31.4	31.6	2.5	55.6	4 R
Co	mg/kg	9.1	9.3	1.2	17.4	25 R
Cu	mg/kg	28.0	27.0	1.2	68.6	7 R
Fe	mg/kg	19717	20785	747	36220	0
Pb	mg/kg	78.1	71.2	11.0	300.0	29 R
Mn	mg/kg	324	321	21.4	637	4 R
Hg	mg/kg	0.202	0.200	0.026	0.380	89
Ni	mg/kg	16.4	17.4	2.3	31.5	14 R
Se	mg/kg	nd	nd	nd	nd	na
Sn	mg/kg	64.1	64.1	64.1	64.1	96
Zn	mg/kg	493	370.5	13.6	1610	0
PCBs	ug/kg	11.3	6	6	60.4	75R
PAHc	ug/kg	2118	1683	103	6024	0

1 - not applicable

2 - replaced w/ 1/2 detection limit

* - 1 sample equals approx. 4%

Six river and creek samples and six marsh samples were subjected to a sequential extraction and metals analysis. In the procedure, the sediments were filtered and sequentially digested and filtered with three acids of increasing strength. The liquid filtrate and the three extracts were analyzed for arsenic, selenium, cadmium, chromium, copper, lead, nickel, and zinc. The extracts are considered to represent adsorbed, oxide, and organic geochemical fractions. The river and creek samples were numbers 5, 25, 49, 77, 105, and 133; while the marsh samples were N-1, N-3, N-6, NS-8, CM-1, and CM-3. The average percentages of the metals extracted from each geochemical fraction are provided in Tables 5 and 6. The filtrate results are not shown since few samples contained adequate amounts of pore water for analysis. Further, the samples yielding enough water had few detectable results. In the river samples, only sample 105 had metal detection in the filtered water. This was zinc at 17 µg/l. In the marsh samples, the Nonesuch Creek sample NS-8, had nickel detected at 9 µg/l and zinc at 10 µg/l in the filtrate.

Table 5. Geochemical Fraction Percentages for River Sediments.
(Fractions that are 10% greater than the other fractions are in bold)

Metal	Adsorbed	Oxide	Organic
As	21%	32%	47%
Se	0%	52%	48%
Cd	21%	**52%**	28%
Cr	9%	27%	**63%**
Cu	0%	**70%**	30%
Pb	18%	**55%**	27%
Ni	33%	36%	30%

Zn	26%	**54%**	21%

Table 6. Geochemical Fraction Percentages for Marsh Sediments.
(Fractions that are 10% greater than the other fractions are in bold)

Metal	Adsorbed	Oxide	Organic
As	11%	26%	**62%**
Se	5%	37%	**57%**
Cd	22%	42%	36%
Cr	6%	28%	**65%**
Cu	2%	**59%**	39%
Pb	22%	**46%**	32%
Ni	22%	35%	43%
Zn	**41**%	**42**%	17%

Within each environmental setting (river and marsh) the samples generally had the same fractional distribution, however, some substantial differences in the partitioning or fractionation were revealed. Within the two Churchmans Marsh samples and the Nonesuch Creek sample the arsenic in the adsorbed fraction only exhibited 0%, 1%, and 2% of the total arsenic extracted. Whereas the three samples from Newport Marsh had 26%, 15%, and 24% arsenic in the adsorbed fraction (similar to the river samples). Also, in the river samples the percentage range for zinc in the oxide fraction was 48% - 69%; two to eight times greater than the other fractions. But in sample 105 (which had zinc in the filtrate) had the greatest percentage of zinc in the organic fraction (47%).

organic compounds results

The organic compound analyses consisted of pesticides, PCBs, and PAHs. All three of these compound groups were to be evaluated by immunoassay techniques with a confirmation laboratory analysis on a sample subset. However, the immunoassay method did not provide usable results for pesticides. Therefore, only the laboratory results were used to evaluate the pesticides.

Twenty samples were analyzed for pesticides; three of which were from the marshes. Only three river samples and one marsh sample had no detectable pesticides. Methoxychlor was detected in 13 of the samples; DDE in 13 of the samples; DDT in 1 sample (#113 at 5.6 µg/kg), and; Endosulfan in 1 sample (#85 at 5.9 µg/kg). For the three marsh samples, the Churchmans Marsh sample, CM-6, had 110 µg/kg methoxychlor and 7.9 µg/kg DDE, Nonesuch Creek sample, NS-5, had 11.0 µg/kg DDE, and Newport Marsh sample, N-1, had no detectable pesticides.

A correlation between the detection and concentration of methoxychlor and DDE was noted. Both pesticides co-occurred in 10 of the 13 samples containing either pesticide and, with the exception of one sample, the concentrations were correlative. Samples up to and including #65 (up to Newport) had methoxychlor and DDE detection of 80% for each pesticide and mean values of 236 µg/kg and 39.2 µg/kg, respectively. Whereas, for samples collected upstream of #65 the detection for methoxychlor and DDE were less and the mean values reduced to 38.3 and 3.97 µg/kg, respectively. Figure 4 shows a plot of the concentrations of DDE and methoxychlor versus sample location.

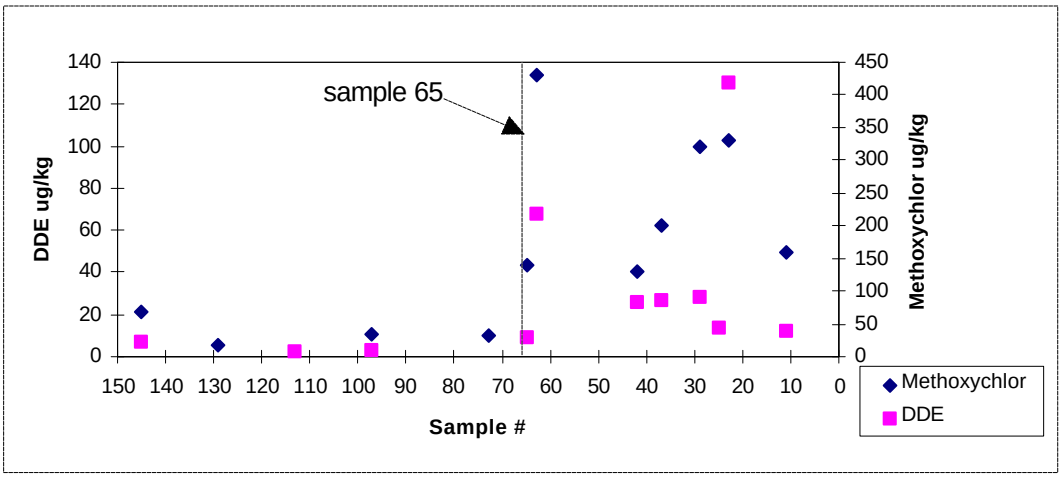

Figure 4. DDE and Methoxychlor Concentration Distribution Plot.

PCBs are of particular concern in the Christina River Basin. They have been found in the tissue of various sport fish species to such a degree that several fish consumption advisories have been issued in the major waterways of the basin. Tables 7 and 8 below provide statistical summaries for PCBs in 20 sample intervals in the river and the breakdown in the individual tributaries and marshes.

Table 7. Summary Statistics of PCB Concentrations in Christina River Sediments Using Immunoassay methods with 12.5 ppb detection limit (in µg/kg wet weight).

Sample # Range	Mean	Median	Range*	% non-detect
1 - 20	32	30	nd - 57	15
21 - 40	96	80	nd - 369	10
41 - 60	197	<12	nd - 3115	65
61 - 80	35	<12	nd - 366	70
81 - 100	25	23	nd - 64	20
101 - 113	27	19	nd - 55	15
Total range (1 - 113)	71	22	nd - 3115	36

* -- nd refers to non-detect (< 12.5 ppb)

Table 8. Descriptive Statistics of PCB Concentrations in Sediments of Reaches Discharging to the Christina River (in µg/kg wet weight).

Sample # Range	# of Samples	Mean	Median	Range*	% non-detect
White Clay Creek	18	< 12	< 12	nd	100
Brandywine Creek	19	26	25	nd - 65	26
Newport Marsh	7	< 12	<12	nd	100
Nonesuch Creek	15	< 12	<12	nd - 60	67
Churchman's Marsh	6	21	< 12	nd - 57	50

* -- nd refers to non-detect (< 12.5 ppb)

PCB's were readily found in the downstream sediments in the Wilmington area (samples 1 - 40). In the mid-stream segments (samples 41 - 80) the presence of PCBs are sporadic or

localized; but the concentrations are much greater than in the downstream sediments. The upstream sediments (samples 81 - 113) had a high percentage of detection like the downstream sediments but the concentrations were slightly less.

The distribution patterns for PAHc presence and concentration distribution were similar to the PCB pattern. The sample areas of highest PAHc concentrations were similar to the highest PCB concentrations with the exception of three areas where the PAHc concentration were much greater relative to the other peaks and four locations where the concentrations were lower than the PCB peaks. The highest wet weight concentrations were greater than 20,000 µg/kg; while secondary peaks were between 5,000 and 10,000 µg/kg. The results and distribution of the PAHc with PCBs are shown in Figure 5. Descriptive statistics for PAHc concentrations are provided in Table 9 for the river and in Table 10 for the tributaries and marshes.

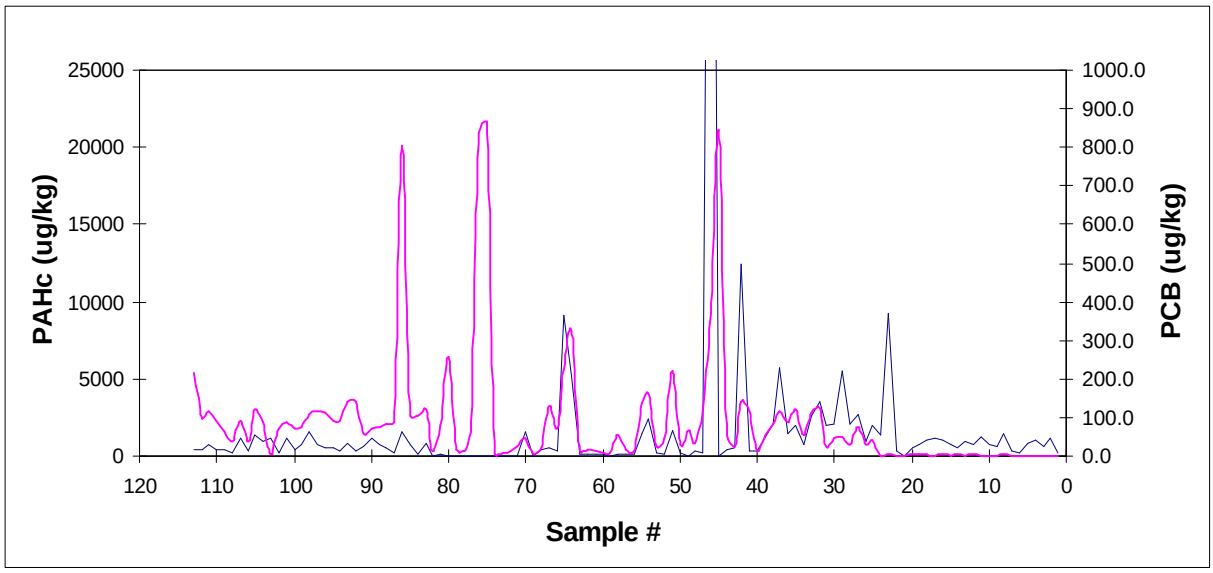

Figure 5. PAHc Distribution Overlay on PCB plot (PAHc- light: PCB - dark).

(Highest PCB not shown at 3115 µg/kg)

Table 9. Summary Statistics of PAHc Concentrations in Christina River sediments using Immunoassay method with 60 ppb detection limit (in µg/kg wet weight).

Sample # Range	Mean	Median	Range*	% non-detect
1 - 20	58	nd	nd - 58	65
21 - 40	1330	1155	nd - 3138	15
41 - 60	3102	1344	216 - 21090	0
61 - 80	3677	582	114 - 21600	0
81 - 100	3176	2325	288 - 20100	0
101 - 113	2158	2172	150 - 5400	0

Total range (1 - 113)	2260	1308	nd - 21600	8

* -- nd refers to non-detect (< 60 ppb)

Table 10. PAHc Concentrations Descriptive Statistics in Tributaries and Marshes (in µg/kg wet weight).

Location	Mean	Median	min.	max.
Newport	749	318	108	3492
Nonesuch	2467	2472	180	5016
Churchmans	2842	2088	1254	6024
Brandywine	1699	972	132	11604
White Clay	577	306	30	2232

toxicity results

The following discussion describes the findings of the toxicity indicator tests and assays. It must be emphasized that the toxicity assays are for determining acute toxicity. Chronic toxicity was not assessed because the need to identify hot spots was considered to be more important in this initial baseline assessment.

The laboratory performing the Microtox® analyses ran the samples in the 1% to 45% dilution range; the results of which were reported as “>45%” for all samples except for six of the Nonesuch Creek samples. Of these six, only one, NS-14, had less than a 45% result, but only on the 5 minute reading and not the 15 minute reading. Therefore, the Microtox® results were considered to be of little comparative value as part of the data set.

The *Hyalella azteca* assay exposes this freshwater amphipod to sample sediment for 10 days. Toxicity is considered significant if organism survival is significantly less than that in a “clean” control sediment. The IQ® assay expresses toxicity as an EC_{50} in terms of organism (*D. magna*) response. The toxicity values are expressed as a dilution factor (percentage) of the pore water. The Microtox® and IQ® bioassays were selected because they are relatively inexpensive, rapid screening methods for acute toxicity which are well-suited to the concept of this study; to provide baseline information over a large area with good coverage.

For the potentially free metals, the ratio of simultaneously extracted metals to acid volatile sulfide (SEM/AVS) was used. When the molar ratio of metals to sulfide is below unity, no acute toxicity would be expected because there is sufficient sulfide present to bind the metals. At ratios above unity toxic effects are possible.

Geographical locations of areas exhibiting acute toxicity as determined by evaluating all toxicity indicator tests are shown in Figure 6.

***Hyalella azteca* results**

A total of 48 samples were subjected to the *H. azteca* toxicity test. The samples were run in 5 different sets with at least one control sample included in each set (one control was used for every ten samples in a set). It was decided to use 80% survival as the demarcation between toxicity and non-toxicity based on the results of the control samples. This value also coincides

with the ASTM recommended low-end value for control samples. Table 11 identifies the samples and their corresponding values for samples considered acutely toxic.

Table 11. Percent Survival Results for Sediments with Lower than 80% by the *H. azteca* assay.

Sample #	% Survival
1	57.5
5	77.5
9	67.5
17	62.5
B1	65.0
B17	25.0
25	72.5
33	70.0
45	35.0
65	70.0
81	77.5
CM-1	65.0
NS-12	77.5

IQ results

The IQ results are reported as the percentage of the pore water sample at which 50% of the test organisms are affected (the EC_{50}). For example, sample NS-13 had a reported EC_{50} of 6.3%, meaning a four-fold serial dilution of the original sample was necessary to affect 50% of the test organisms. The undiluted pore water in sample NS-13 probably affected all of the organisms. Of the 43 samples with reported results 39 samples (90%) were between 2.7% and 35%. Of the remaining 4 samples, two samples were reported as 0.0% (considered to be less than 1% and extremely toxic to *D. magna*); one sample as 107% (considered to be 100% and very non-toxic to *D. magna*); one sample as 51.2%.

Based on general and assumptive correlation evaluations with the other toxicity indicators, the range for confidently indicating toxic effects is estimated to be about 0 - 10% Those IQ® results above 15% can't be assumed to reliably indicate true toxic effects. Table 12 lists the sample numbers and percentage result of the IQ test that was consisted to represent toxicity.

Table 12. IQ Results in Percent for Samples Considered Toxic to *D. magna*.

Sample ID	IQ %
21	10.0
25	10.5
37	3.6
45	4.6
53	10.2
57	10.3
65	5.0
69	4.2
77	6.3
85	3.3
89	4.2
93	7.0
97	6.3
109	6.3

Sample ID	IQ %
113	9.2
145	9.6
B13	0.0
B16	0.0
N-3	2.7
N-6	7.8
NS-11	7.8
NS-12	4.6
NS-13	6.3
NS-14	5.0
NS-2	10.1
NS-6	9.8
NS-8	7.9
NS-9	10.3

SEM/AVS results

The SEM/AVS ratios were generally lower in the marsh samples than the river and creek samples, indicating a lower potential for free metals. Further, the differences in the average SEM/AVS ratios between these environments was found to be due to the greater amount of sulfide present in the marsh environment. The extracted metals concentrations appear to be similar between the marsh and river environments. It must be restated that the SEM/AVS applicability to the less reduced environments, such as those in several of the river samples, may be questionable. The lack of sulfide mathematically produces a large SEM/AVS ratio and therefore the inference of significant free metal. However, other anions, such as sulfate, sulfite, phosphate, phosphite, chromate, chloride, and carbonate can form insoluble complexes with toxic metals. These complexes may or may not be extracted during the SEM/AVS analytical procedures.

For the total 64 SEM/AVS samples from the study area, 33 samples (52%) had SEM/AVS ratios that were greater than 1. However, 17 of these samples had no detectable sulfide concentrations and their greater than unity ratios are therefore questionable. The remaining 16 samples were used in the evaluation. The amount of potentially free metal was calculated by subtracting the μm/g of sulfide from the total SEM μm/g concentration. Table 13 summarizes the results by listing the sample number, in descending order for values calculated. Actual values are not provided in the table because the SEM/AVS test is designed for determining only if there is a potential for free metals to exist and greater values do not necessarily imply greater toxicity. A greater potential for toxicity however may be indicated by higher ratios.

Table 13. Samples Listed in Descending Order for SEM/AVS Ratio and Potentially Free Metals Concentration.

SEM/AVS Ratio	Free Metal
45	45
65	85
B13	97
9	113
B19	93
5	B4
101	65
B4	109
97	B13
93	25
113	105
109	101
25	9
105	5
NS-10	NS-10
85	B19

sediment quality assessment

Comparison to Long and Morgan Guideline Values

Although toxicity assays were performed on the sediments and pore water, the analytical results were compared to the nationally recognized Effects Range - Low (ER-L) and Effects Range - Medium (ER-M) guideline values derived by Long and Morgan (1991) and Long et. al. (1995). Any analytical result under the ER-L was considered to have a "minimal effect" on benthic organisms. Results between the ER-L and ER-M were considered to exhibit "possible effects", and results over the ER-M were considered to have "probable effects" to the benthic community. When an ER-L or ER-M value was not available, the EPA Sediment Quality Benchmark value was used. The following table (Table 14) summarizes the comparison by listing the percentage of the total sample set that exceed the ER-L and ER-M for each analyte.

Table 14. Percentage of Total Samples Exceeding ER-L and ER-M Values.

Analyte or Compound	ER-L (mg/kg)	ER-M (mg/kg)	% Exceeds ER-L	% Exceeds ER-M	Number of Samples
Antimony (Sb)*	2	25	15	6	65
Arsenic (As)	8.2	70	32	0	65
Cadmium (Cd)*	1.2	9.6	35	0	65
Chromium (Cr)	81.0	370	1.5	0	65
Cobalt (Co)	-	-	-	-	65
Copper (Cu)	34.0	270	43	0	65

Lead (Pb)	46.7	218	69	1.5	65
Mercury (Hg)	0.15	0.71	43	3	65
Nickel (Ni)	20.9	51.6	48	0	65
Tin (Sn)	-	-	-	-	65
Zinc (Zn)	150	410	89	45	65
Methoxychlor	19 (EPA SQB)		60		20
DDE	2.2	27	60	20	20
PAHc	4022	44792	10 (32)	0	178
PCBs	22.7	180	39 (54)	4 (6)	178

* Method detection limit above ER-L, therefore all detected results are >ER-L. Sb detection limit ranged from 15 to 50 mg/kg. Cd detection limit ranged from 1.2 to 4.3 mg/kg.

- No ER-L, ER-M or EPA SQB available.

( ) Calculated based on dry weight assuming 50%, moisture which is the study average.

There were numerous heavy metal concentrations that exceeded the ER-L. Only two metals, antimony and chromium, had less than 30% of the samples exceeding the criterion. Four of the metals had concentrations that exceeded the ER-M. The metals that had concentrations exceeding the ER-M or that exceeded the ER-L in over half the study area were decided to be the metals of greatest concern. These are antimony, lead, mercury, and zinc. This should not be misinterpreted to imply that the other metals do not pose potential impacts but only that the antimony, lead, mercury, and zinc are considered to pose the greatest potential for impact due to the concentration and degree of presence. With regards to antimony the detection limits for the samples always exceeded the ER-L and in many samples exceeded the ER-M. The detected results in some samples were less than reported detection limit of other samples. Therefore, the interpretations and conclusion regarding antimony may be questionable. A map indicating the areas where metals concentrations have exceeded the ER-M is provided in Figure 6.

All samples with detectable DDE were over the ER-L with the exception of one sample. Four of the samples are also over the ER-M. The one sample that did not exceed the ER-L of 2.2 μg/kg (sample # 113) had a concentration of 2 μg/kg. This sample also exhibited the only DDT detection at 6 μg/kg and is over the DDT ER-L of 1.58 μg/kg. Because DDE is a breakdown product of DDT, all DDE detections are considered to have originated from a DDT source. Sample #113 is considered to be close to an ongoing DDT release area.

There is no established ER-L or ER-M for methoxychlor but the EPA SQB for methoxychlor is 19 μg/kg. Only one of the samples with methoxychlor does not exceed the SQB of 19 μg/kg, sample #129, but it exhibited a concentration of 17 μg/kg. This data along with DDT and DDE data suggest that DDT and DDT related compounds (DDE and methoxychlor, a.k.a. methoxy DDT) are present in concentrations that could exhibit impacts to the benthic community. The one Endosulfan detection of 5.9 μg/kg exceeded the EPA SQB of 5.4 μg/kg was not considered to be significant due to its low frequency of detection.

The areas of elevated PAHc and PCB concentrations were defined as samples with wet weight concentrations greater than the ER-L for each compound group due to noted prevalence. A total of 17 samples exceeded the PAH ER-L, while 69 samples exceeded the PCB ER-L. Four samples exceeded the ER-M for PCBs. Since all other analytes were reported as dry weight the approximate PAHc and PCB dry weight concentrations were calculated based on the approximate average moisture of 50%. This calculation dramatically increased the percentages of ER-L

exceedances. Figures 7 and 8 highlight the locations of the elevated PAHc and PCB concentrations, respectively.

Toxicity evaluation

The SEM/AVS results were compared to the *H. azteca* and IQ results. Because the SEM/AVS methodology was designed for samples containing sulfide and potential free metals occur if the ration exceeds 1; only results that met these two criteria were used. The average *H. azteca* survival for the SEM/AVS ratio >1 was 69%; while the survival for ratios <1 was 85%. Considering a *H. azteca* control value of 80%, it was concluded that toxicity is greater when the SEM/AVS ratio is >1. Figure 9 shows the regression plot of *H. azteca* versus SEM/AVS. The regression revealed a correlation coefficient (R^2) of 0.89, a probability of 0.007, and an F statistic probability of 6×10^{-7}. The statistical significance of these results indicates a relationship and possibly causation. The average IQ results were 7.9% and 18.7% for the SEM/AVS ratios >1 and <1, respectively which also generally indicates acute toxicity (<10%) for SEM/AVS ratios > 1. However, regression analysis the results did not indicate as strong of a correlation as with the *H. azteca*.

The *H. azteca*: SEM/AVS correlation was evaluated further to ensure that the correlation was true and not due to the high prevalence and concentrations of PAHc and PCBs. Therefore, the *H. azteca* results for the samples used in the SEM/AVS regression were compared to the organic compounds. The PCBs, which ranged from non-detected to 366 µm/kg, did not indicate a correlation with the survival percentages. The PAHc indicated a correlation with the *H. azteca* results in the samples with high SEM/AVS ratios (R^2 value of 0.79). However, further analysis indicated that this was due to the occurrence of PAHc in the samples with the high SEM/AVS results. Also no direct relationship was seen between PAHc and the *H. azteca* toxicity throughout the entire sample set (regardless of the SEM/AVS ratios). It was therefore concluded that the toxicity is more likely related to free metals and that the co-occurring organic compounds may aid in the overall toxic effects. It should be noted that the pesticide values were also compared to the toxicity data and no direct correlation was found. However, the pesticide data set was small and the results were not statistically significant.

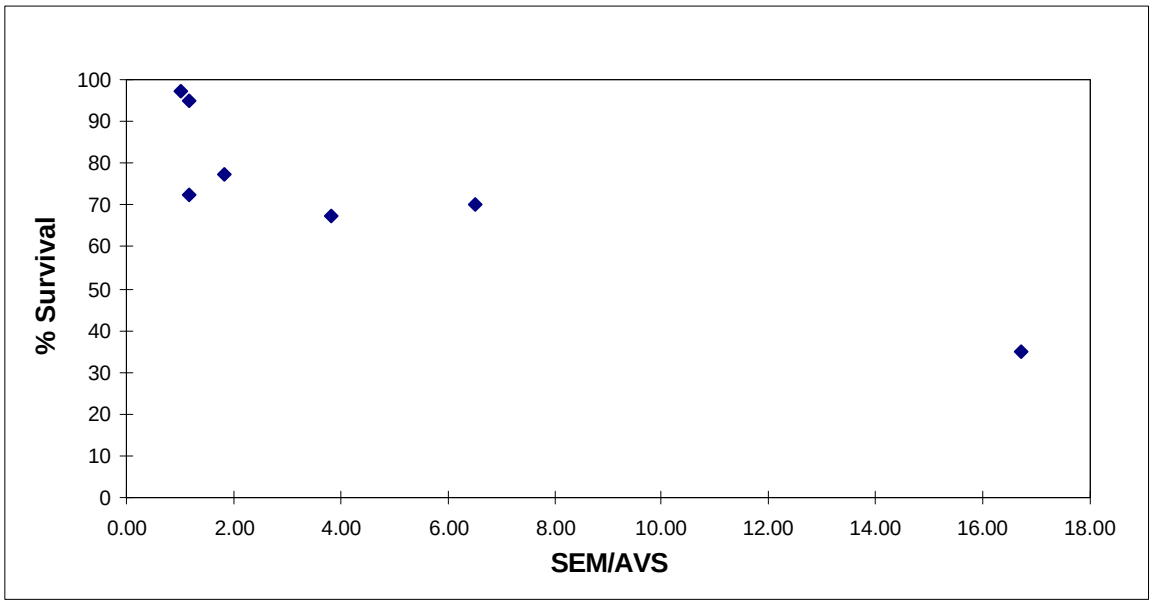

Figure 9. Percent Survival for *H. azteca* vs. SEM/AVS >1 Ratios for Samples with Detectable Sulfide Concentrations.

As mentioned previously, the SEM/AVS ratio does not include other anions that could also bind with the metals rendering them non-bioavailable. In an attempt to explain the IQ results the other anions measured in the sediments that can bind with metals were combined with the sulfide and paired with the total SEM results to produce a SEM/Total Anion ratio. The EC_{50} range (up to 20%) was divided into four equal intervals of 5% each. The mean SEM/Total Anion ratio was calculated for each interval and plotted. Figure 10 shows the result.

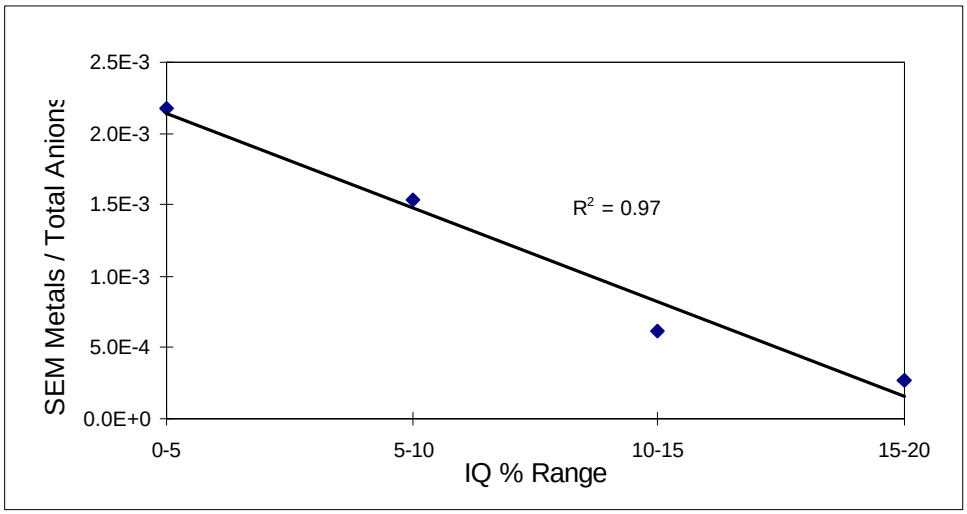

Figure 10. Mean SEM/Total Anion Ratios vs. 5% IQ Intervals.

As evident in the plot, there appears to be a correlation between the metal/anion ratio and the IQ results. The correlation coefficient was 0.97 with a high "goodness of fit". These results also indicate bioavailable metals as being the cause for the acute toxicity results. The areas indicating acute toxicity are shown in Figure 11.

CONCLUSIONS

The results of the baseline assessment indicated many areas in which sediment contamination is present. Several of theses areas have contaminant levels elevated to a degree that impacts are strongly suggested. Further, toxicity assays performed on the sediment confirmed the impact. The areas containing the measured and potential impacts include:

1. near the mouth of the Christina River;
2. the mouth of Brandywine Creek;
3. the Brandywine Creek proximal to Wilmington in the vicinity of the 11th St. bridge;
4. the Christina River along south west Wilmington;
5. the mouth of the Dravo Marsh;
6. the mouth of The Lagoon at Newport;
7. the mouth of Churchmans Marsh and along Interstate 95; and,
8. the Christina River upstream of Churchmans Marsh in the vicinity of Christiana.

Other various isolated locations are indicated but their significance is considered less than the areas mentioned above. Virtually all of these areas appear to be similar in that they are adjacent to discharge points, whether natural or constructed outfalls. The areas of the upper Brandywine Creek and southwest Wilmington appear to coincide with areas of numerous outfalls. The drainage areas contributing to these outfalls should contain the upland areas that served as sources for the contaminants identified. As stated, the data suggests that the acute toxicity is related to the elevated bioavailable metals. Other anthropogenic contaminants such as PCBs, PAH compounds, and pesticides are present throughout the study area. Their concentrations vary greatly but were not shown to directly account for the acute toxicity.

During this study only acute toxicity was assessed. The findings of this project clearly demonstrate the need to assess chronic toxicity in the Christina River sediments. The information presented here can be used in designing a chronic assessment as well as an on-going monitoring plan. Of particular note, sediments with elevated contaminants did not always exhibit acute toxic results in the assays. These same sediments may, due to the elevated contaminants, provide a chronic exposure to aquatic biota. In the case of toxic metals, the reason for non-acute toxicity may probably be due to speciation.

Additionally, the geographical correlation between the presence of elevated PCBs in sediments and elevated PCBs in game fish tissue (Greene, 1996) was noticed. This information aids in documenting biomagnification processes in the river. Further, using health risk extrapolations, PCB concentration criteria in sediment can be established for the protection of human health and piscivorous wildlife. Also, additional contaminants found in game fish tissue can also be extrapolated to aid in setting sediment criteria.

Physiochemical data inferred of presence of several different sediment types. The sediment differences appear to be caused by the physiographic province(s) from which the sediments originate. Upstream sediments, where the river is also shallow, the sediment characteristics are indicative of Coastal Plain materials. It consists of fine to coarse quartz grains that are less chemically reduced, and exhibit lower total extracted metals concentrations than downstream. Downstream sediments are fine grained, reduced material, higher in metals and/or heavy minerals. This may be indicative of materials originating from the Piedmont Province. The importance of this finding is in the comparison of upstream “background” samples to downstream samples in environmental investigations. The comparison may not be justified if the sediments used were not initially similar in character, especially when elements and compounds may react differently with each of the sediment type. Many sampling protocols suggest that tidal sediments should not be used to gauge background or unaffected conditions.

The evaluation of the toxicity assays and ecological predictors was found to be applicable within the basin. Additionally, the usefulness and reliability of field screening and measuring methodologies such as immunoassay techniques were found to be applicable to the Christina River sediment evaluation.

APPENDIX

Graphical Representation of Sediment Data