ELIZABETH RIVER TRIBUTYLTIN MONITORING PROGRAM:

1999-2006

A Report to:
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PURPOSE: The purpose of this project was to implement a study in 1999/2000 that would document the current levels of tributyltin (TBT) in the Elizabeth River and provide baseline data for future efforts to determine the trend of TBT concentrations found in the Elizabeth River Watershed. Subsequent years of sampling have documented spatial and temporal trends in TBT and are described in this report.

BACKGROUND: The VA DEQ-TRO, in collaboration with the Elizabeth River Project, has prepared an Elizabeth River Monitoring Program (ERMP) that is designed to describe the trend in environmental conditions relating to five areas of concern to stakeholders. The five primary areas of concern are: sediment quality; water quality; habitat; living resources; and quality of life.

This TBT monitoring project has evaluated the potential for TBT impacts on water quality in the Elizabeth River, its three branches, the Lafayette River and the lower James River.

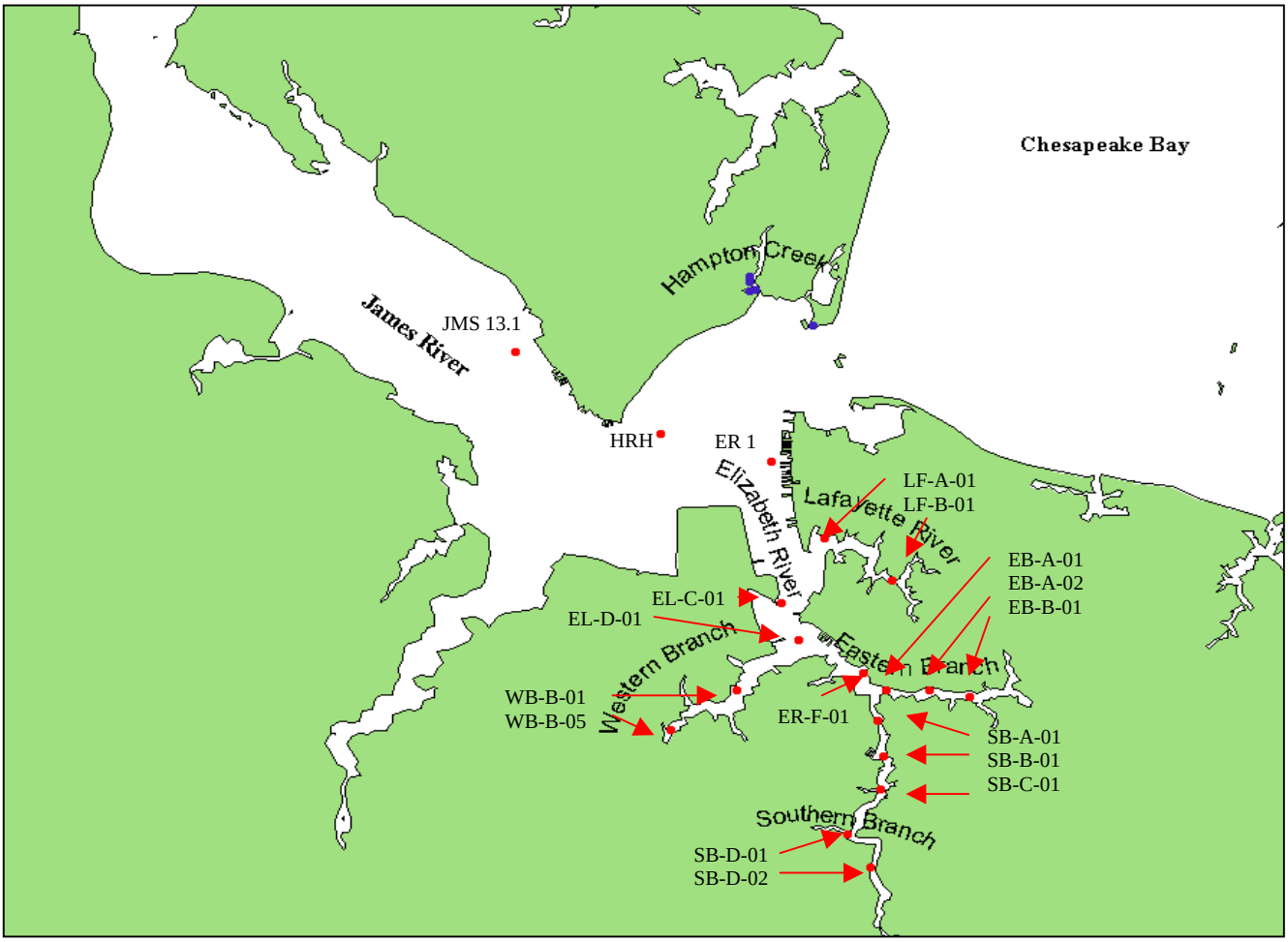
Coordination with other monitoring projects

Concern over adverse environmental effects from TBT use in the 1980's led to regulatory actions restricting the use of TBT in France, the United Kingdom and the United States. The Virginia General Assembly in 1987 enacted TBT legislation limiting use of TBT paints to vessels over 25 m in length (*Code of Virginia*, 1987). To assess the effectiveness of these regulations, the Department of Environmental Sciences at VIMS began analyzing water samples and biota from various locations around southern Chesapeake Bay. Samples collected as early as 1985-1986 showed elevated TBT concentrations in the vicinity of shipyards in the Elizabeth River (Huggett et al, 1986). Water column monitoring was performed at nine stations near marina areas every month from 1985-2002 and the resulting data set has shown elevated TBT concentrations in the vicinity of marinas and has documented the variability in spatial and temporal trends. Concentrations decreased by at least a factor of five at the marina monitoring stations over the period but were still above detection limits (1 ng/L) in most samples in 2002. VIMS continued monitoring TBT in water samples from nine locations in Hampton Creek and Sarah Creek, Virginia until 2002. This allowed a comparison of TBT concentration trends in water from marina areas with trends determined for the Elizabeth River over the same period.

METHODS

Water Column Monitoring

Environmental water samples were collected by VIMS personnel bimonthly at 18 Elizabeth River station locations selected by the VADEQ. These sites are shown in Figure 1. Utilizing the same individuals to collect and analyze water samples eliminated sample custody issues. Once collected, all environmental samples were maintained in locked storage prior to analysis. All vessels used for collecting samples were free of TBT containing antifoulants and water samples were collected in sample storage containers to avoid cross contamination of samples via sampling gear. Samples were collected as near to high slack water as possible and the sampling information recorded on each container and in a field notebook.



Stations	Latitude		Longitude	
	Deg	Min	Deg	Min
JMS13.1	36	59.400	76	27.600
HRH	36	57.300	76	23.500
ER1	36	56.516	76	20.291
LF-A-01	36	54.506	76	18.822
LF-B-01	36	53.350	76	16.848
EL-C-01	36	52.900	76	20.166
EL-D-01	36	51.889	76	19.728
ER-F-01	36	50.938	76	17.898
EB-A-01	36	50.417	76	17.214
EB-A-02	36	50.366	76	15.934
EB-B-01	36	50.167	76	14.748
SB-A-01	36	49.627	76	17.502
SB-B-01	36	48.750	76	17.448
SB-C-01	36	47.970	76	17.562
SB-D-01	36	46.746	76	18.600
SB-D-02	36	45.900	76	18.000
WB-B-01	36	50.633	76	21.648
WB-B-05	36	49.667	76	23.664

Figure 1. Tributyltin sampling stations in the Elizabeth River and adjacent waters.

Water was collected from the top meter of the water column with care to exclude the surface microlayer. Sample bottles were rinsed twice with ambient water prior to collection of the sample. Field blanks were carried on each sampling trip and the samples were kept in the dark on ice in the field for transport back to the laboratory. To document sampling variability, duplicate 2 L samples were collected at four stations during each sampling trip (>20% replication). Once returned to the laboratory, all the samples were preserved with HCL to below pH 2 and kept at 4^{o}C in the dark in a locked cold room prior to analysis.

Tributyltin analysis

Water samples were analyzed for butyltins by an adaptation of the methodology published earlier (Unger et al, 1986) which has been described in detail in a manual prepared for the Virginia DEQ (Unger, 1996). This analytical method has also been recently published as part of Method 6710 (Tributyltin) in Standard Methods of Wastewater Analysis (Rodigari et al., 2005). This same analytical method was used in all previous TBT monitoring and assured comparability with historical data. Analysis of extraction blanks (>10%), sample duplicates (>10%), matrix spikes and matrix spike duplicates documented the accuracy and precision of these analyses to assure project data objectives were met. Method detection limits (1 ng/L) have been determined using the procedures recommended by the VA DEQ and were described in detail previously (Unger, 1999).

Data Quality Objectives

Overall precision (sampling and analytical) was assessed through field replicate measurements/analyses. Four stations were selected for collection of replicate field samples. For the first three sampling periods, the four stations were kept constant but randomly selected replicate locations were used in all subsequent sampling trips. Sampling precision was evaluated by comparing overall precision to measurement/analytical precision obtained through the analysis of multiple matrix spiked samples. Overall accuracy was assessed through field sample matrix spike analyses, and is evaluated as percent recovery. Sampling completeness is calculated based on the ratio of samples collected to samples that were planned, and is expressed as percent completeness.

RESULTS AND DISCUSSION: 1999-2006

Overall Summary and Concentration Trends

Samples were collected successfully at all 18 stations for each sampling date over the 1999-2006 period of the study. A general trend was evident with the highest concentrations located near the confluence of the eastern and southern braches of the Elizabeth River with lower concentrations in the main stem, the western branch, Hampton Roads, and the Lafayette River. This is evident when the average TBT concentrations for the period 1999-2006 for all stations are compared (Figure 2). Average TBT concentrations also tended to decrease at stations that were further upstream in the tributaries. The highest concentrations measured during the monitoring period (>60 ng/L) occurred on September 20, 2001 and are shown in Figure 3. The spatial trend in concentrations mentioned previously is also evident in the figure.

The yearly average TBT concentrations measured at the Elizabeth River stations showed a general increasing trend in the southern and eastern branches of the Elizabeth River over the first three years of the monitoring period and then a decreasing trend from 2003-2006 at all stations (Figure 4).

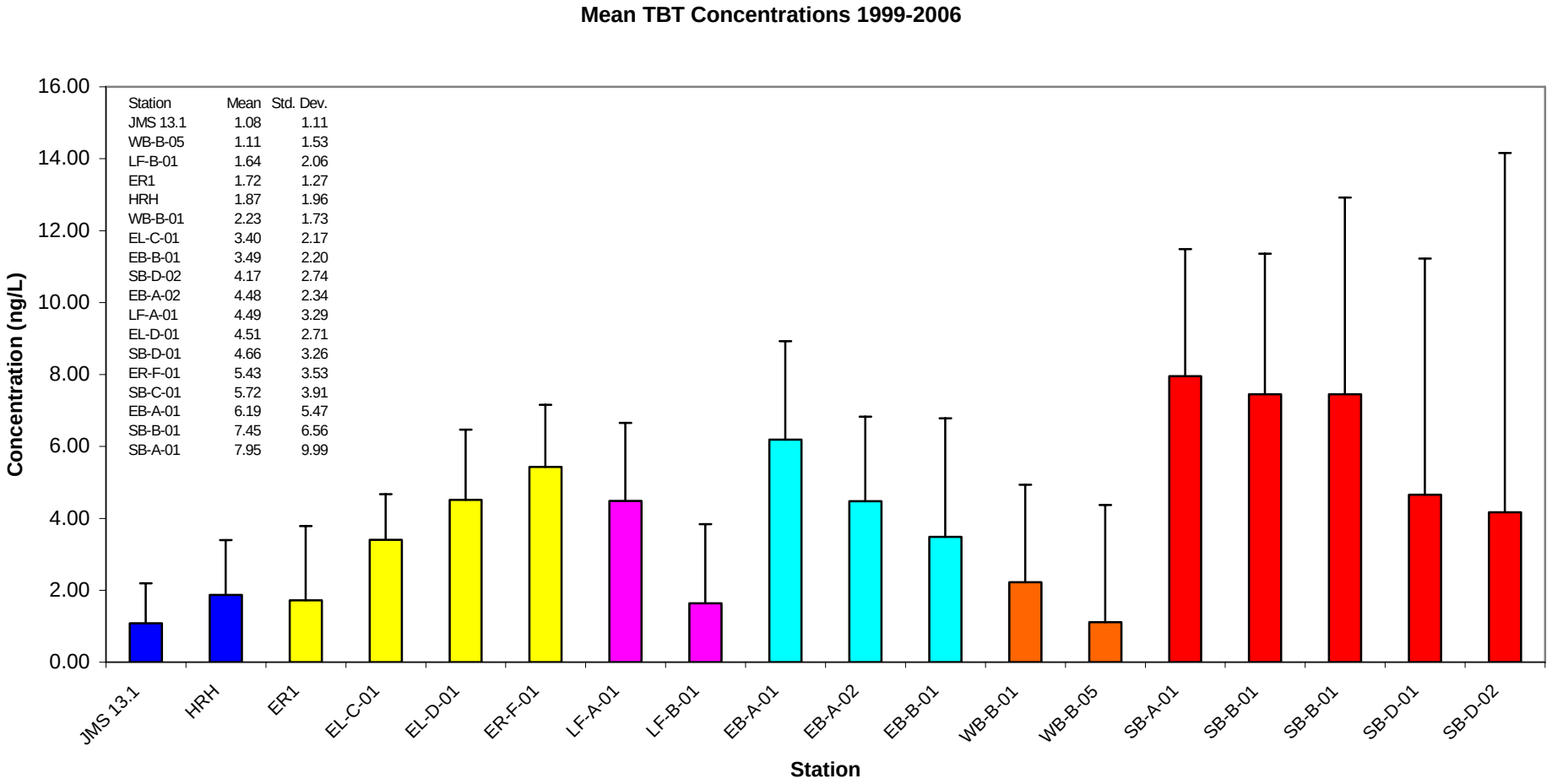
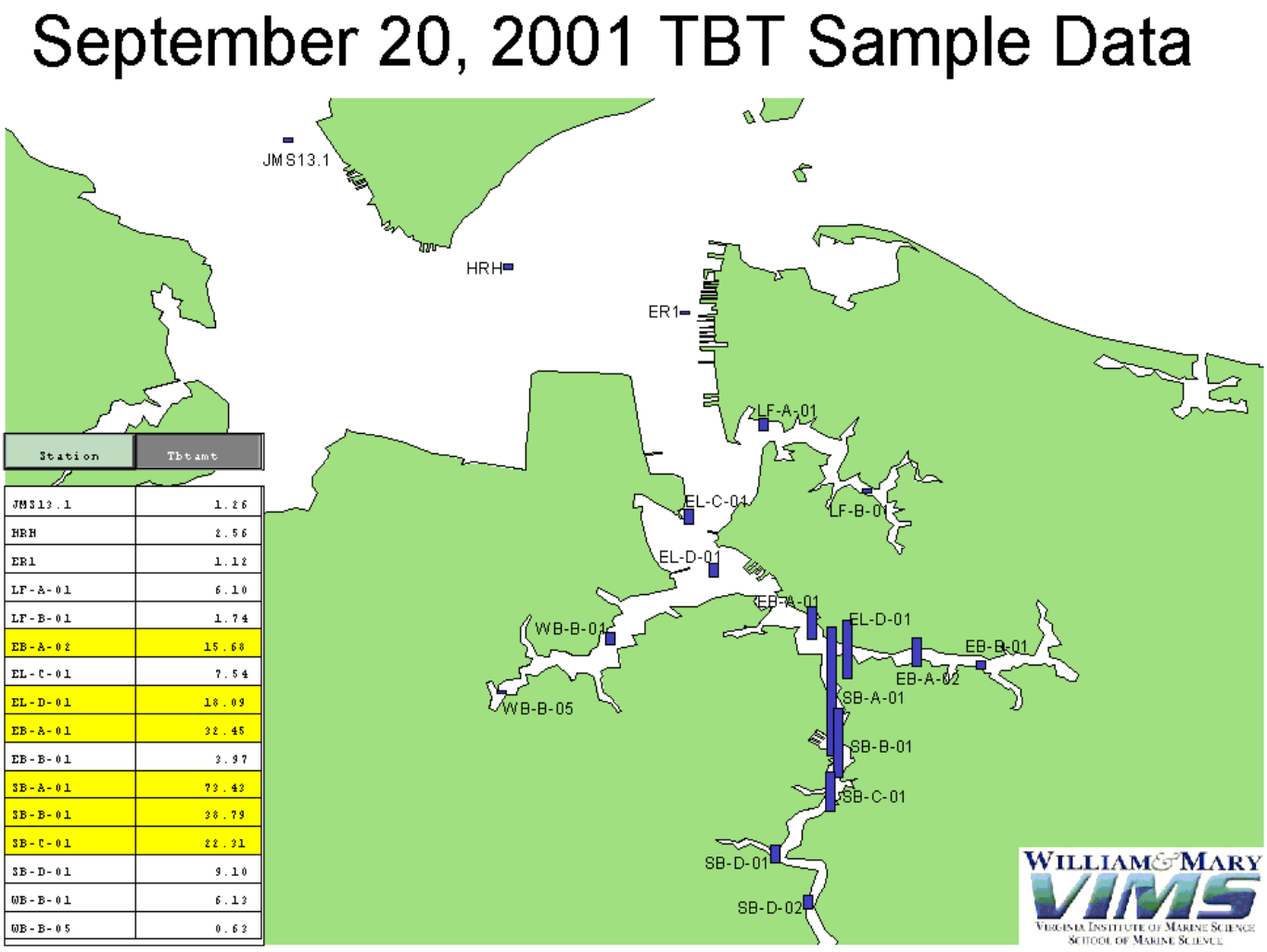


Figure 2. The average TBT concentrations measured for the period July 1999- June 2006 for all 18 stations used in the monitoring program. Highest average concentrations were found near the confluence of the eastern and southern branches of the Elizabeth River.



Station	Tbtamt
JMS13.1	1.26
HRH	2.56
ER1	1.12
LF-A-01	6.10
LF-B-01	1.74
EB-A-02	15.68
EL-C-01	7.54
EL-D-01	18.09
EB-A-01	32.45
EB-B-01	3.97
SB-A-01	73.43
SB-B-01	38.79
SB-C-01	22.31
SB-D-01	9.10
WB-B-01	6.13
WB-B-05	0.63

Figure 3. TBT Concentrations measured at stations in the Elizabeth River and Hampton Roads on September 20, 2001. This represent the highest concentration measured (>60 ng/L) and also demonstrates the spatial trends in TBT concentrations that were repeated at other dates but at lower concentrations.

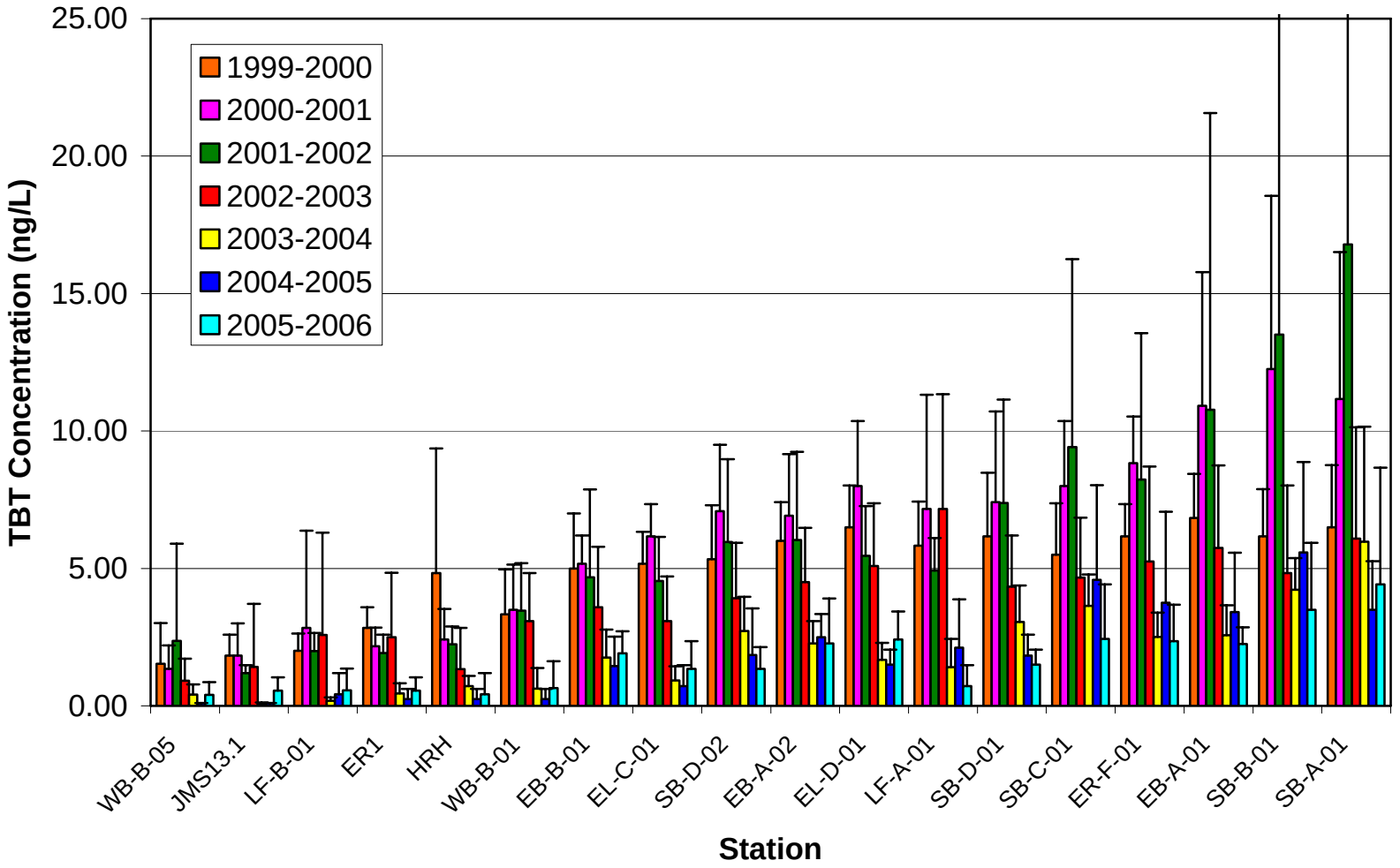


Figure 4. Average annual TBT concentrations measured at 18 stations over seven years in the Elizabeth River and Hampton Roads, Virginia. Error bars represent +/- 1 std. dev., n = 6. Mean TBT concentrations show a significant decreasing trend from 2003 to 2006.

A statistical comparison of the pre 2003 and post 2003 mean concentrations was performed for stations SB-A-01, SB-B-01, SB-C-01 and EB-A-01 using a 2-sided t-approximation, Wilcox Rank Sum test. The H_0: The before and after 2003 mean concentrations are the same, was rejected for all stations at $\alpha = 0.05$. The mean TBT concentrations are significantly lower at each of these stations in the period 2003-2006 relative to the mean concentrations measured from 1999-2003.

Current Status of TBT in regions of the Elizabeth River, Virginia

To assess the current status of TBT concentrations in various regions of the Elizabeth River the 18 monitoring stations were further subdivided into the following regions: James River, Main Stem, Western Branch, Eastern Branch, Southern Branch and the Lafayette River. The station/region designations are presented in Figure 5. The fixed station locations of the DEQ TBT monitoring program were designed in 1999 to assess current and future trends in TBT concentrations and is not a probabilistic based design that would be preferred for the statistical evaluation and comparison of regions. Regardless, differences at the specific stations can be compared to assess general trends in the various regions at the station locations. To evaluate the current status, the mean TBT concentrations were calculated for the final two-year interval July 2004- June 2006. This provided 12 measurements per station. Any samples with concentrations below the method reporting limit of 1 ng/L were set to 0.5 ng/L when calculating mean concentrations. Average concentrations calculated from stations with multiple measurements below the reporting limit contain a large amount of censored data so the calculated means should be viewed with caution. Graphs showing the measured concentrations for the entire 1999-2006 monitoring period at each location are provided in Appendix A. A red line on the graphs at 1 ng/L is provided to allow comparison of the concentration trends to the Virginia Water Quality Criteria.

The current average TBT concentrations ranged from 0.5 to 4.5 ng/L (Figure 6). Six of the stations are now, on average, below the Virginia Water Quality Criteria of 1 ng/L. The James River and Western Branch regions are now below 1 ng/L. The Lafayette River is below 1 ng/L average at the upper station but still has measurable TBT on occasions near the mouth of the River. The lower station (LF-A-01) is near a marina and probably is also influenced by input from the main branch of the Elizabeth River while the upper station (LF-B-01) is in an area of the river that is residential. The average concentrations in the Main Stem of the Elizabeth River range from below 1 ng/L at ER1 to 3 ng/L at station ER-F-01 near the confluence of the Southern and Eastern Branches. The Eastern Branch average concentrations ranged from 1.7 to 2.8 ng/L with a decreasing gradient towards the upstream locations. The Southern Branch still has the highest TBT concentrations on average of any region in the watershed but the average concentrations have now decreased to 1.8-4.5 ng/L. Again, the gradient of concentrations decreases towards the upstream stations.

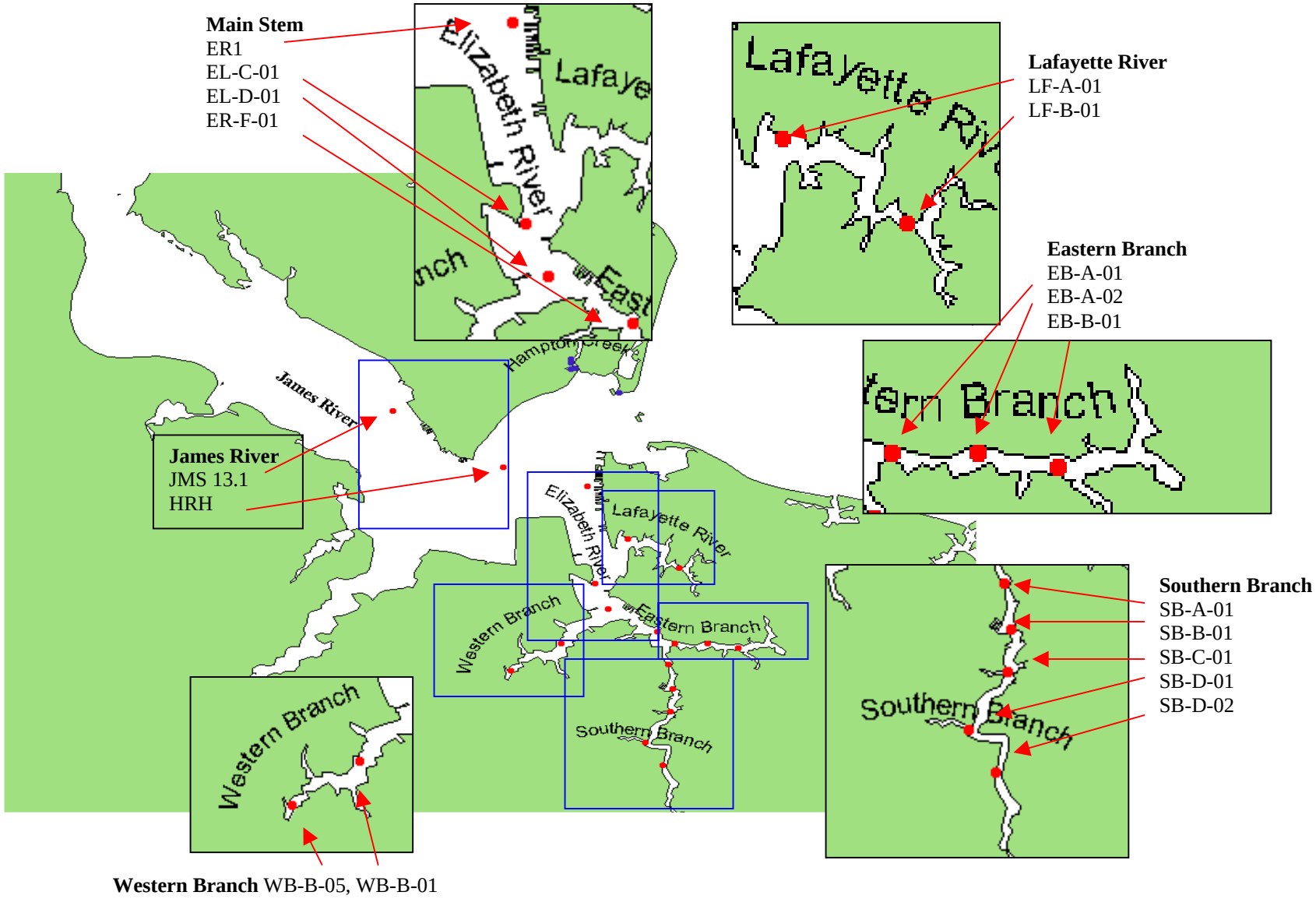


Figure 5. Station groupings for the evaluation of the current status of TBT concentrations in various regions of the Elizabeth River.

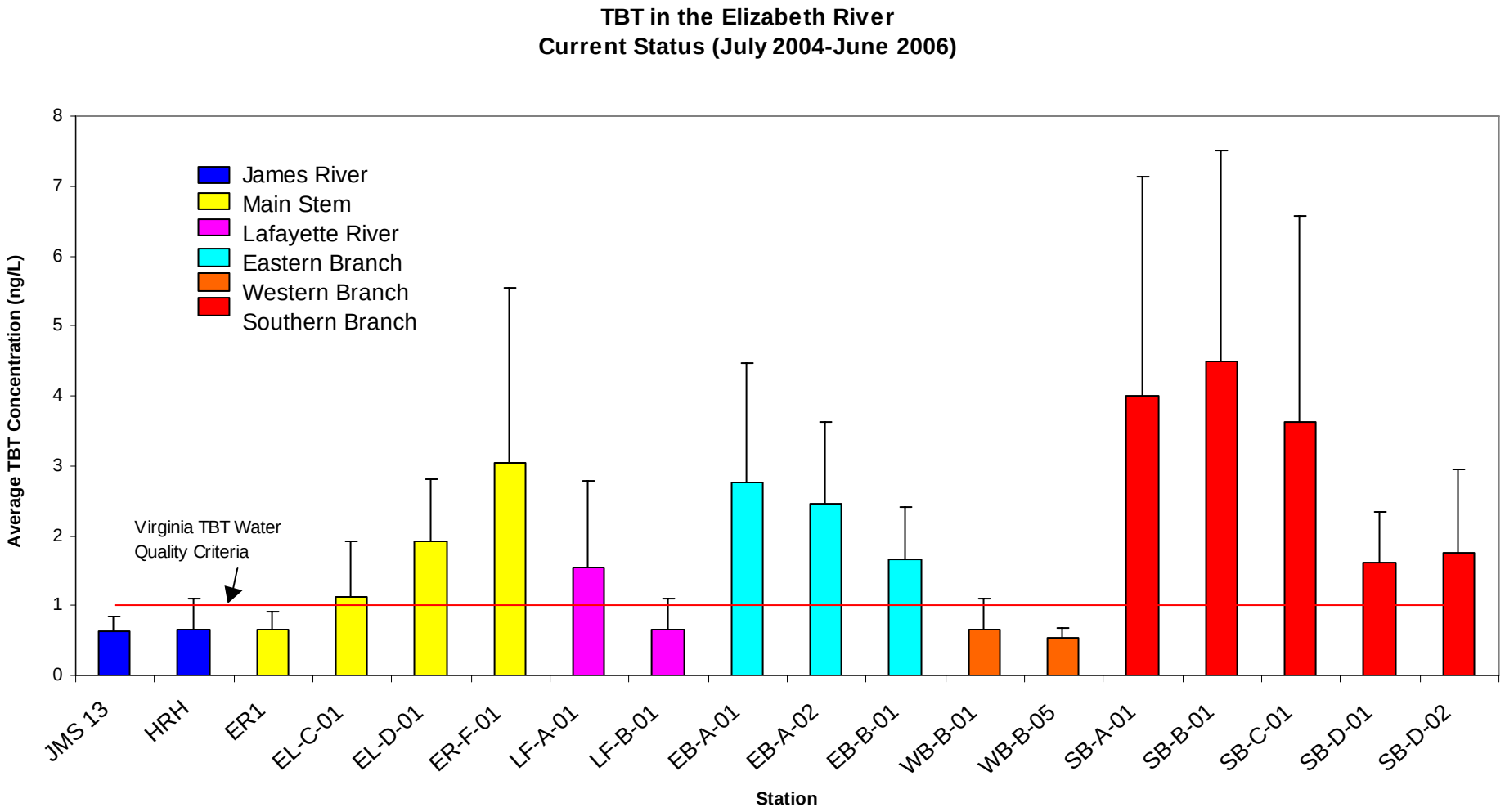


Figure 6. Current Status of average TBT concentrations in the Elizabeth River Watershed (n=12, error bars +/- 1 s.d.). Six stations are now below the Virginia Water Quality Criteria of 1 ng/L.

TBT in Sediments

Surface sediment samples were collected by Ponar grab at 11 stations (LF-A-01, EL-C-01, ER-F-01, EB-A-01, EB-A-02, EB-B-01, SB-A-01, SB-B-01, SB-C-01, SB-D-01, SB-D-02) on 6/21/2005. One sample (SB-A-01) contained interfering compounds that prevented quantification of the TBT so no data is reported for this station. The 10 sediment samples ranged from 9 to 350 ng/g TBT dry weight (Figure 7). Previous research has shown that TBT in sediment samples will range from 10^3-10^4 times higher than ambient water concentrations when water and sediment are in equilibrium (Unger et al., 1996). Sediment concentrations in excess of those predicted by partitioning have been attributed to non-equilibrium conditions, localized high sorption coefficients or ship maintenance areas that included TBT containing paint chips (Unger et al., 1988). A plot of the 1999-2006 average TBT water concentrations at the ten stations vs. the sediment concentrations is shown in Figure 8. The slope of the line through the data represents the apparent partitioning or sorption coefficient (K_p) for the included field data. Two stations (EB-A-01, SB-B-01) contained TBT sediment concentrations in excess of those predicted by equilibrium partitioning alone (Apparent $K_p \cong 10^4$ for the remaining 8 stations). These two stations are in the proximity of ship repair facilities and TBT containing paint chips may be contributing to the elevated TBT concentrations in these sediment samples.

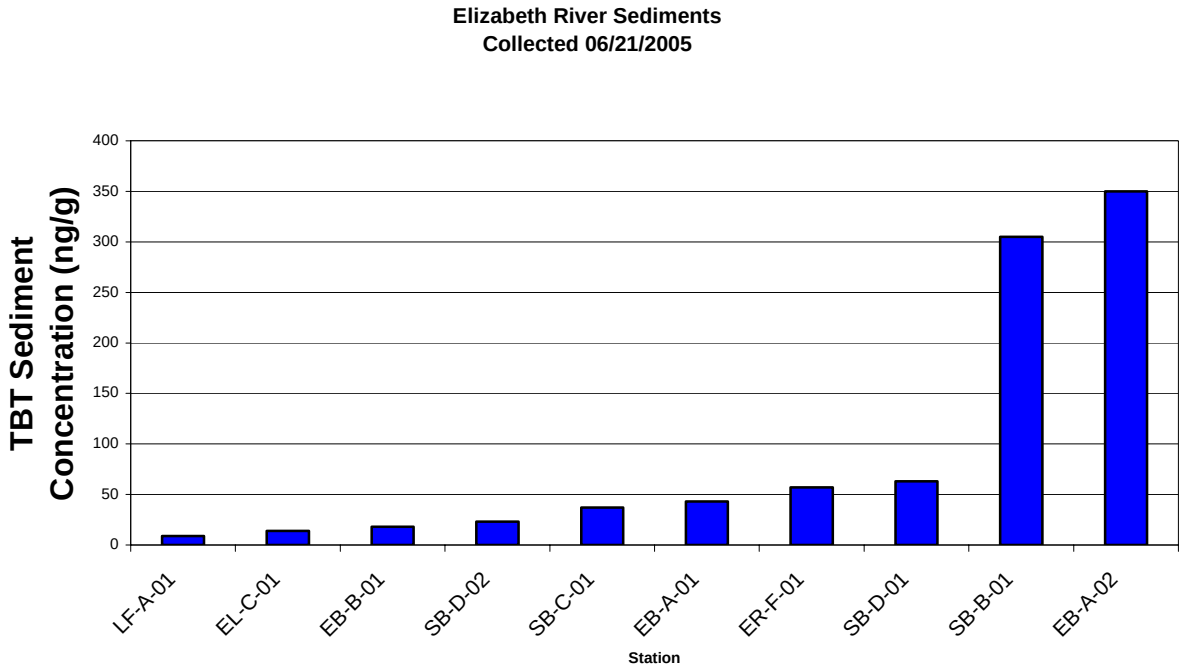


Figure 7. TBT Concentrations Measured at 10 Stations in the Elizabeth River in 2005.

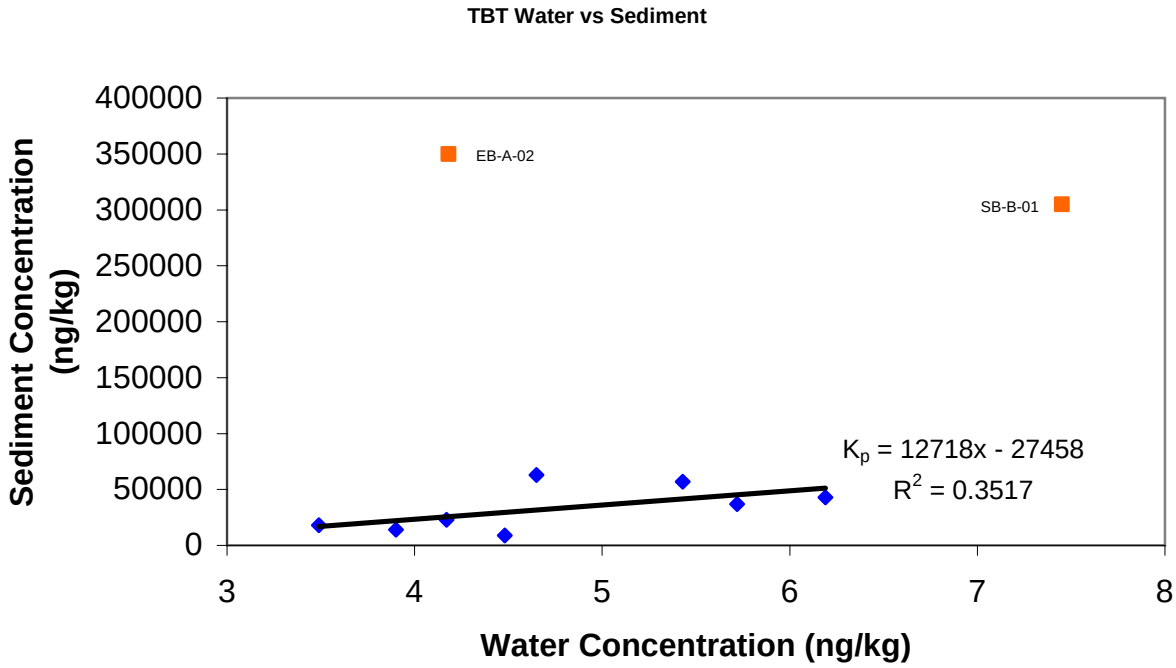


Figure 8. TBT Concentrations in water vs. sediment at 10 stations. Sediment TBT concentrations are elevated at EB-A-02 and SB-B-01 relative to those predicted by equilibrium partitioning alone, suggesting non-equilibrium conditions, higher partitioning coefficients or the presence of TBT paint chips in these samples.

VIMS Marina Monitoring

Samples were collected from the five VIMS monitoring stations in Hampton Creek (Figure 9) at monthly intervals from 1999-2002. The range of TBT concentrations spanned from less than 1 ng/L at station HC5 (Old Point Comfort) to a high of 25 ng/L at HC2 (Hampton Roads Marina #2). Yearly average concentrations ranged from a low of 1.4 ng/L at station HC5 to a high of 8.8 ng/L at station HC2 (Figure 10) and are comparable to concentration measured in the Elizabeth River during the same interval (Figure 4). Error bars illustrate one standard deviation of the calculated mean concentration. There was no obvious temporal trend in average TBT concentrations at the Hampton Creek monitoring sites in contrast to the increasing trends seen for the Eastern and Southern branches of the Elizabeth River during the same time period (Figure 4.) This monitoring program was discontinued in 2002 due to the lack of funding so further comparisons could not be made.

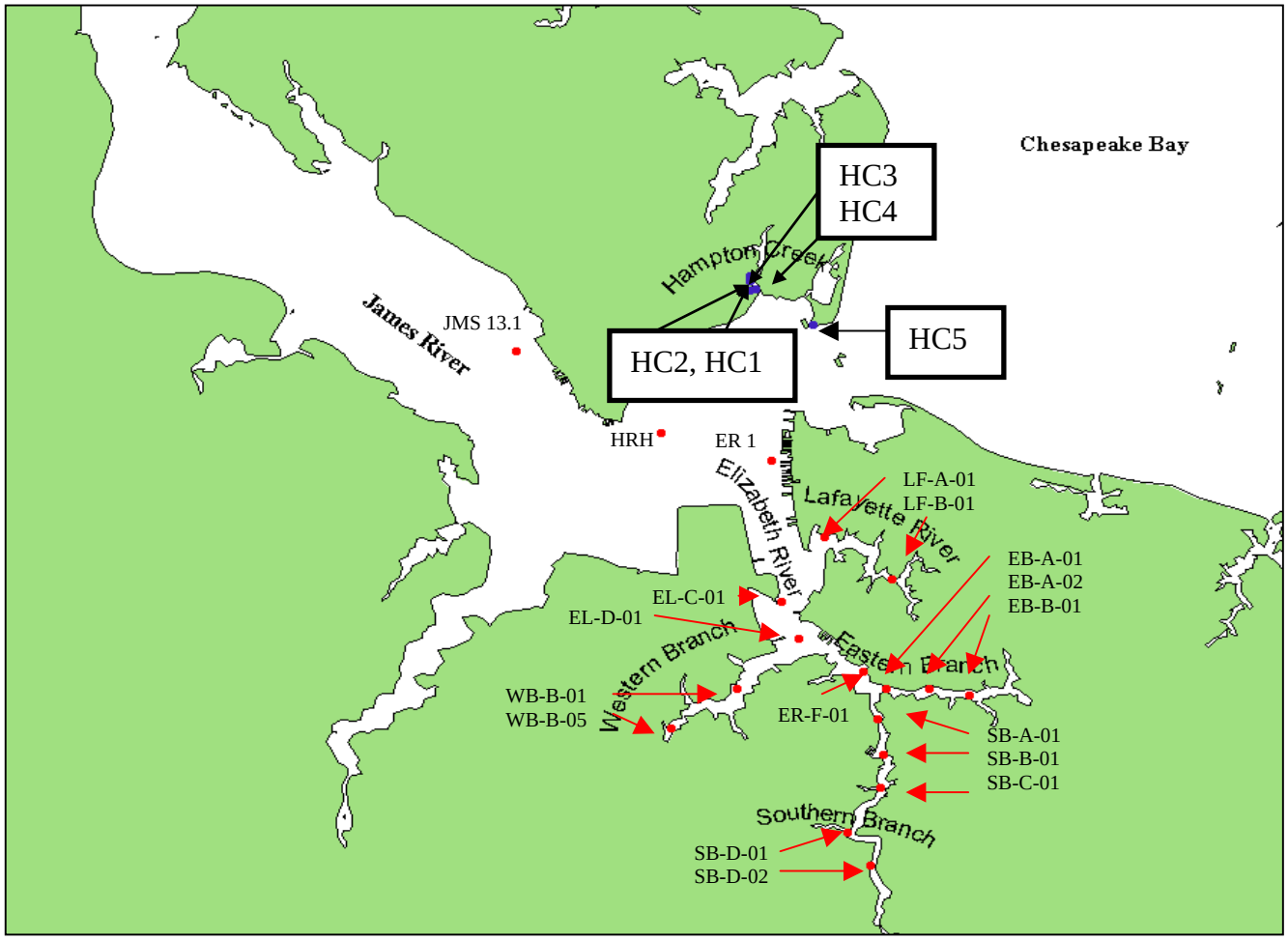


Figure 9. VIMS Marina Monitoring Stations, Hampton Creek, Virginia.

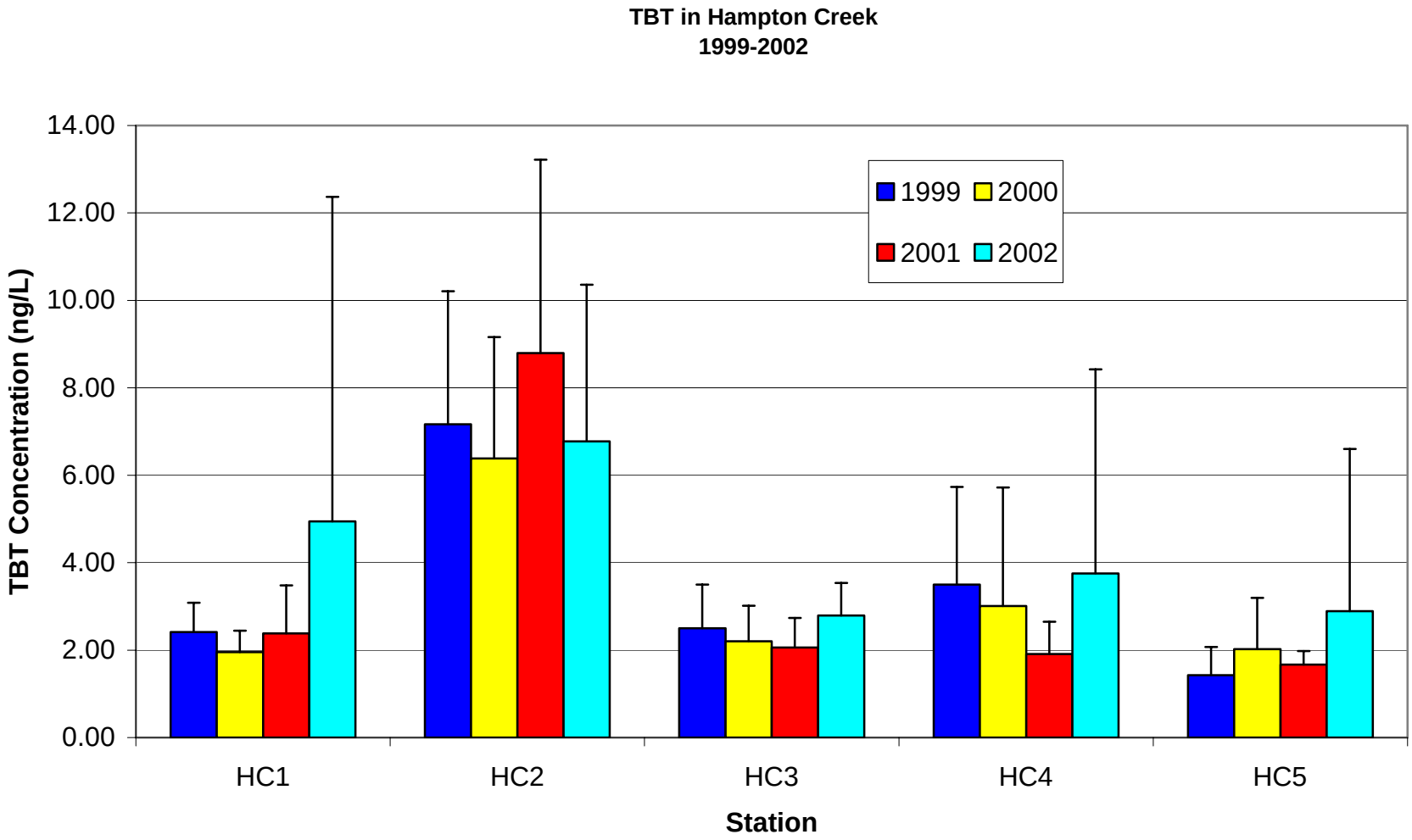


Figure 10. Average yearly concentrations at five VIMS Marina monitoring stations in Hampton Creek, Virginia, 1999-2002

CONCLUSIONS

Current Status of TBT Concentrations and Potential Effects to Biota

The current average TBT concentrations measured in the Elizabeth River Watershed (Figure 6.) shows there is little risk of acute toxic effects from TBT exposure but chronic effects in the most sensitive species are still likely. Acute effects for saltwater species are most likely when concentrations exceed 420 ng/L (Hall et al, 2000), well in excess of concentrations measured in this monitoring program. The study by Hall et al (2000), "A Probabilistic Ecological Risk Assessment of Tributyltin in Surface Waters of the Chesapeake Bay Watershed" used 5 ng/L as a 10^{th} percentile chronic toxicity endpoint for their risk assessment. It is important to note that this endpoint is based on the principal of protecting 90% of the species 90% of the time and is not protective of the most sensitive species in the ecosystem. Based on this principal, the Southern Branch and the rest of the Elizabeth River Watershed has now, on average, decreased to below this 10^{th} percentile chronic toxicity endpoint of 5 ng/L used in previous risk assessments.

The 2003 EPA Ambient Aquatic Life Water Quality Criteria for Tributyltin (TBT) recommends a chronic criteria for salt water organisms of 7.4 ng/l (higher than Hall et al, 2000) and acute value of 420 ng/l (same value as Hall et al, 2000). The EPA criterion also recognizes that there may be sensitive locally important species. The procedures described in the "Guidelines for Deriving Numerical National Water Quality Criteria for the Protection of Aquatic Organisms and Their Uses" indicate that, except possibly where a locally important species is very sensitive, saltwater aquatic organisms and their uses should not be affected unacceptably if the four-day average concentration of TBT does not exceed 7.4 ng/L more than once every three years on the average and if the one-hour average concentration does not exceed 420 ng/L more than once every three years on the average. Discrete water samples were collected every two months during this program and do not represent four-day averages as described in the "EPA Guidelines" but TBT concentrations exceeding the chronic criteria can be used as a general measure to understand how current conditions relate to the EPA chronic criteria of 7.4 ng/L. As described above, no TBT concentrations were measured near the acute value of 420 ng/L during this monitoring program. Over the last three sampling years (July '03-June'06), 18 water samples collected from the Southern Branch, Eastern Branch and Main Stem were 7 ng/L or higher at six different sampling dates. The last samples to exceed the criteria during this period were three samples collected in November 21, 2005 from the Southern Branch of the Elizabeth River. Based on assuming these discrete samples are representative of four-day average concentrations then the EPA chronic criteria is still being exceeded in the Elizabeth River more than once every three years and there is the potential for adverse chronic effects.

Chronic effects in the most sensitive local species are still likely at average TBT concentrations in the 1-5 ng/L range. Tributyltin has been shown to be an endocrine disrupting compound and can induce hormonal changes in sensitive species at low ng/L concentrations. These hormonal effects have been shown to cause sex changes in marine gastropods (imposex) and may hinder hormonal governed development in other sensitive invertebrate species such as copepods. Imposex has been observed in gastropods at environmental concentrations as low as 2 ng/L (Gibbs et al. 1988, Bryan et al. 1989). A recent publication by Mann et al (2006) has shown that the introduced species, veined rapa whelk (*Rapana venosa*), inhabiting the lower James River and Elizabeth River watershed has a preponderance of imposex females over "normal" females. Additional study of this population has shown that both TBT and DBT accumulated in *R. venosa* from Hampton Roads and imposex development is related to butyltin exposure (Jestel, 2003). Mann et al (2000) make note that while 2 ng/L has been shown to be the threshold for imposex

development in other species of gastropods, the threshold for imposex development in *R. venosa* has not yet been determined. Imposex in *R. venosa* is occurring in areas of the Elizabeth River watershed that have current average TBT concentrations below 1 ng/L. Current TBT concentrations in the Main Stem, Southern and Eastern Branches of the Elizabeth River are still likely to produce imposex in sensitive mollusk species.

Marine copepods have also been shown to be sensitive to the effects of TBT. Bushong et al (1990) found a no observable effects concentration (NOEC) of 10-12 ng/L and chronic toxicity (6 d) values of 16-17 ng/L for the estuarine copepod, *Acartia tonsa.* Other studies of the same species found significant development rate effects beginning at 1-5 ng/L TBT level (Kusk and Petersen, 1997) and egg production was reduced at 10 ng/L (Johansen and Mohlenberg, 1987). This copepod species is one of the most important zooplankton species in Chesapeake Bay (Brownlee and Jacobs, 1987). Current average TBT concentrations have now decreased to below the chronic toxicity values reported for *Acartia tonsa* but average concentrations in the Southern Branch of the Elizabeth River still exceed the values that have been shown to inhibit developmental rates in this copepod (Kusk and Petersen, 1997).

ACKNOWLEDGEMENTS

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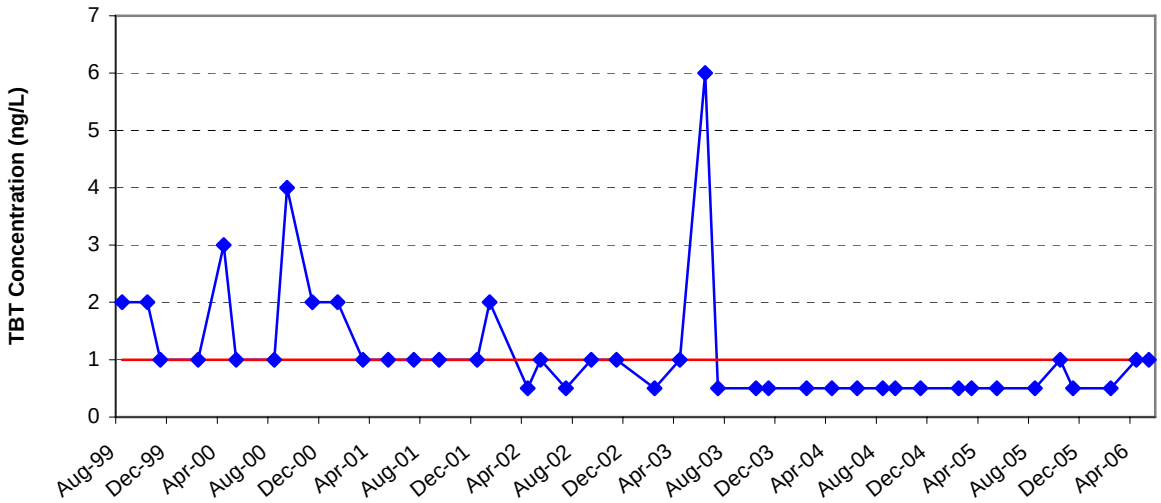
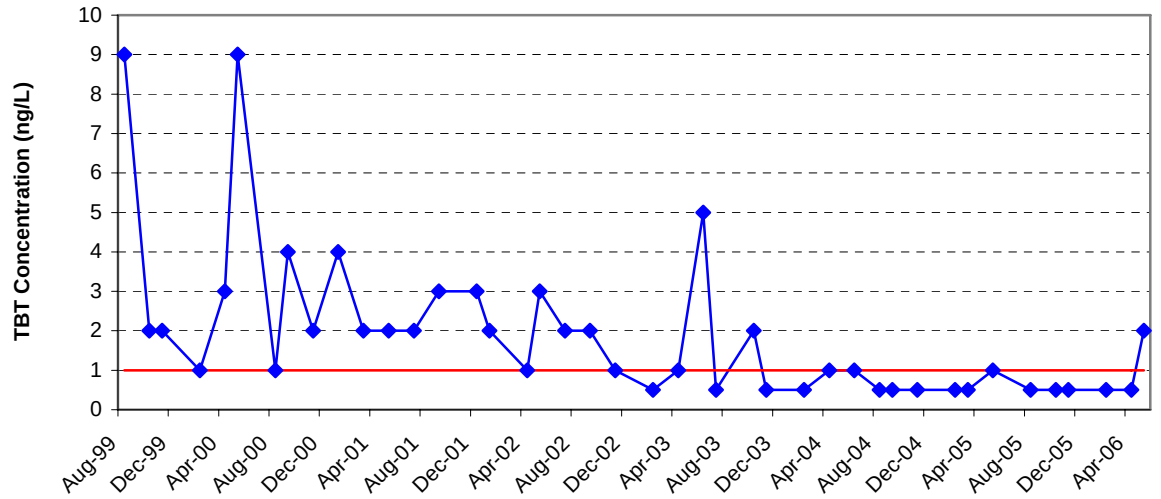
Appendix A
TBT Concentrations Measured at Specific Stations
From 1999 to 2006

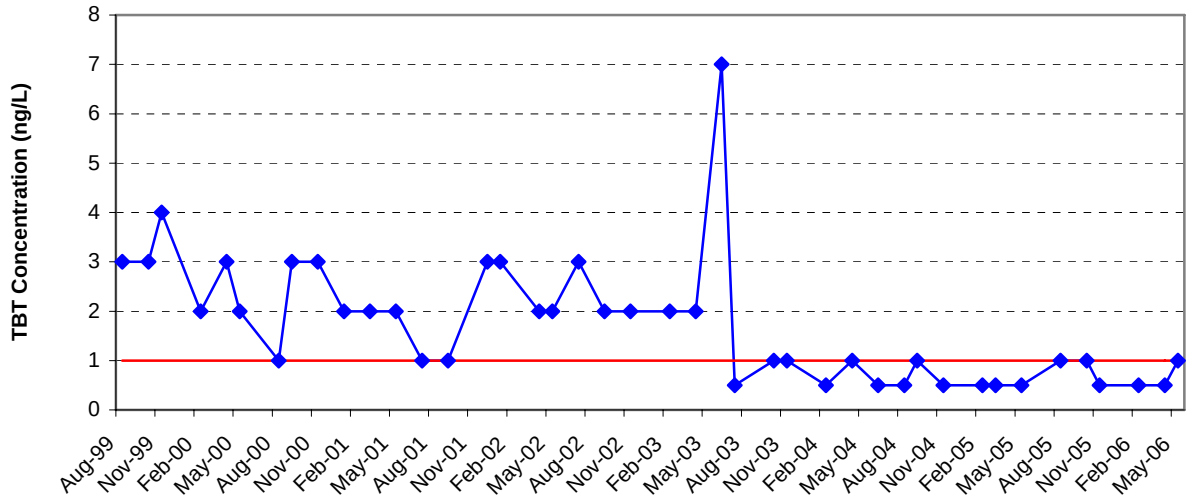
JMS 13.1
James River



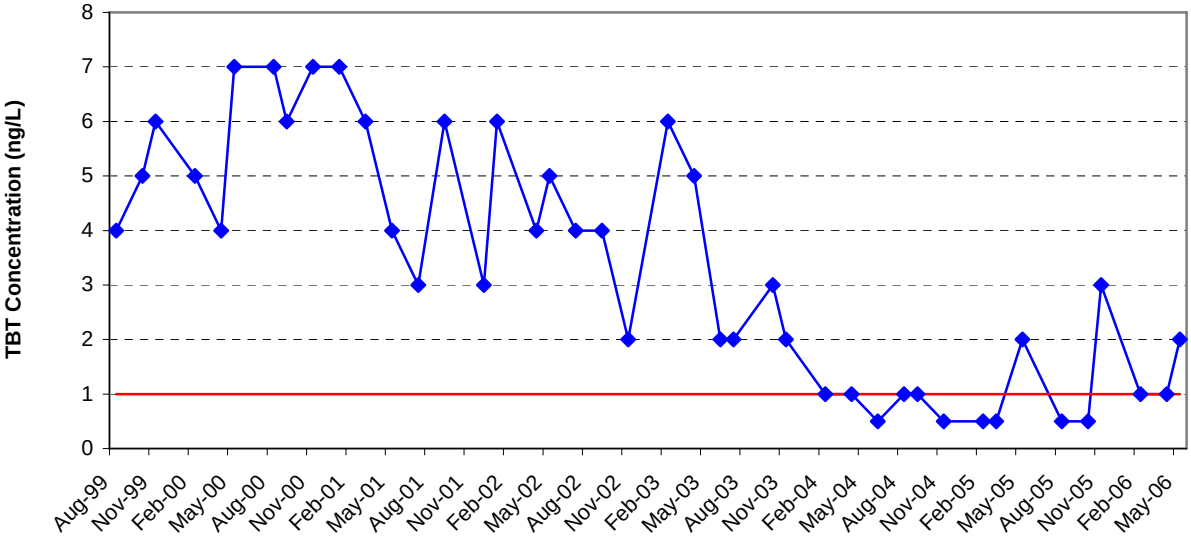
HRH
James River



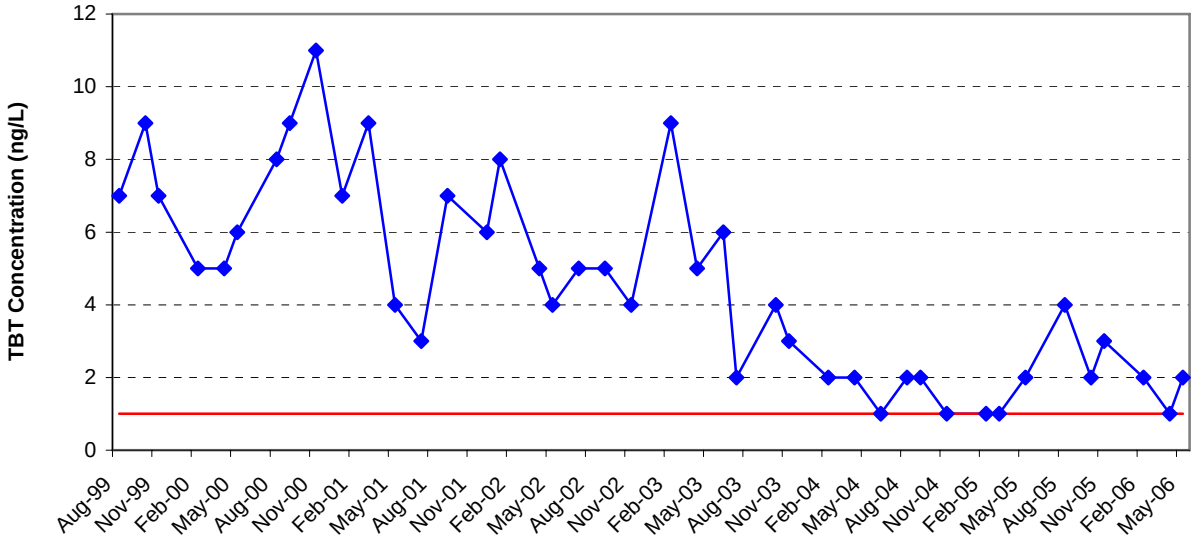
ER1
Main Stem



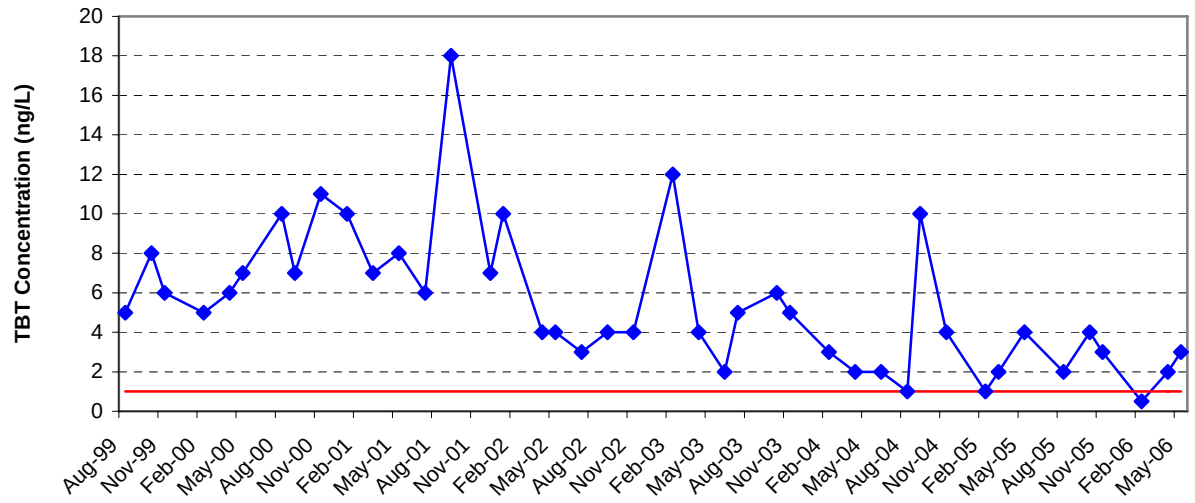
EL-C-01
Main Stem



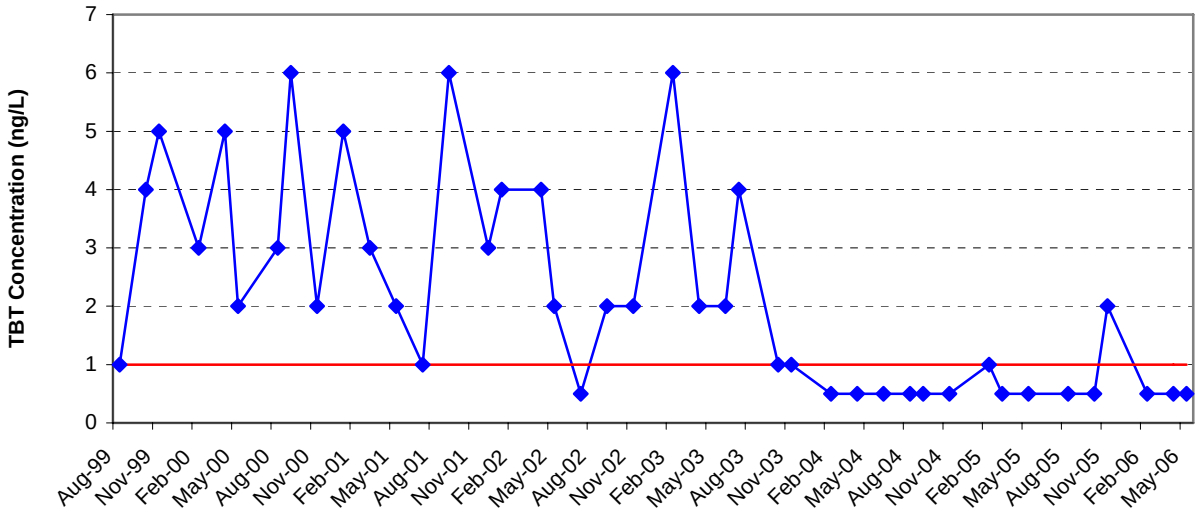
EL-D-01
Main Stem



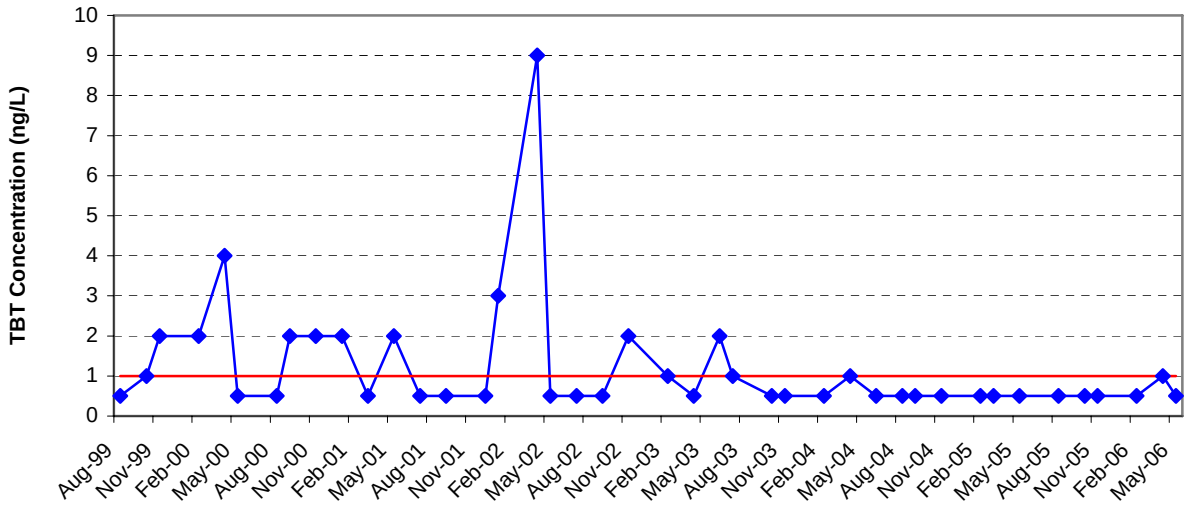
ER-F-01
Main Stem

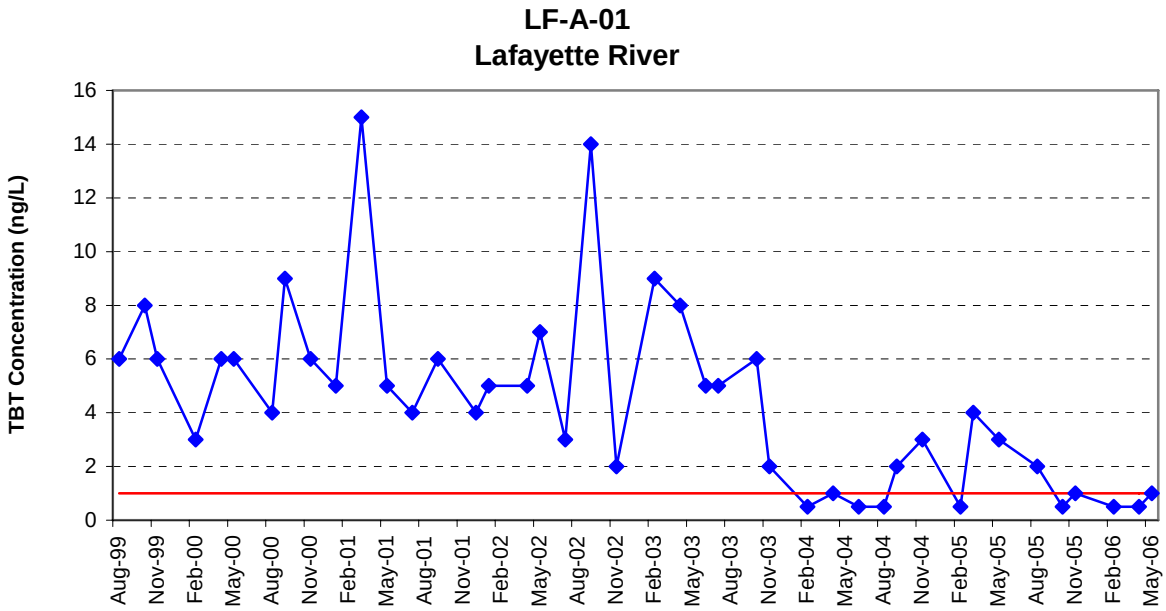


WB-B-01
Western Branch



WB-B-05
Western Branch




LF-A-01
Lafayette River
TBT Concentration (ng/L)
16
14
12
10
8
6
4
2
0
Aug-99
Nov-99
Feb-00
May-00
Aug-00
Nov-00
Feb-01
May-01
Aug-01
Nov-01
Feb-02
May-02
Aug-02
Nov-02
Feb-03
May-03
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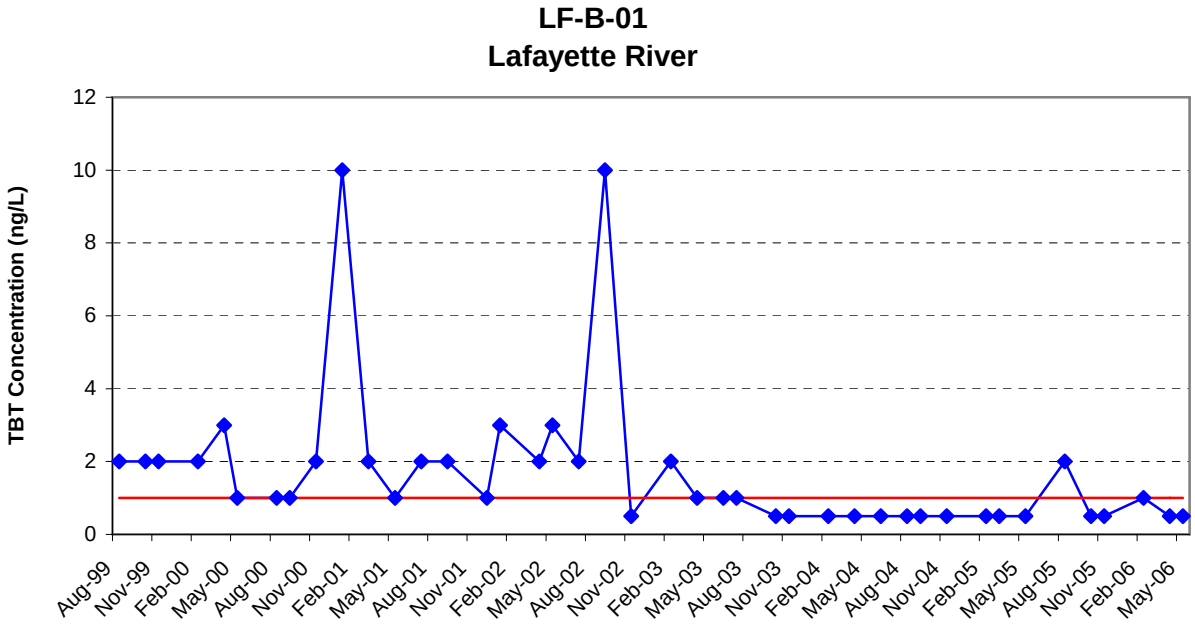
Feb-05
May-05
Aug-05
Nov-05
Feb-06
May-06


LF-B-01
Lafayette River
TBT Concentration (ng/L)
12
10
8
6
4
2
0
Aug-99
Nov-99
Feb-00
May-00
Aug-00
Nov-00
Feb-01
May-01
Aug-01
Nov-01
Feb-02
May-02
Aug-02
Nov-02
Feb-03
May-03
Aug-03
Nov-03
Feb-04
May-04
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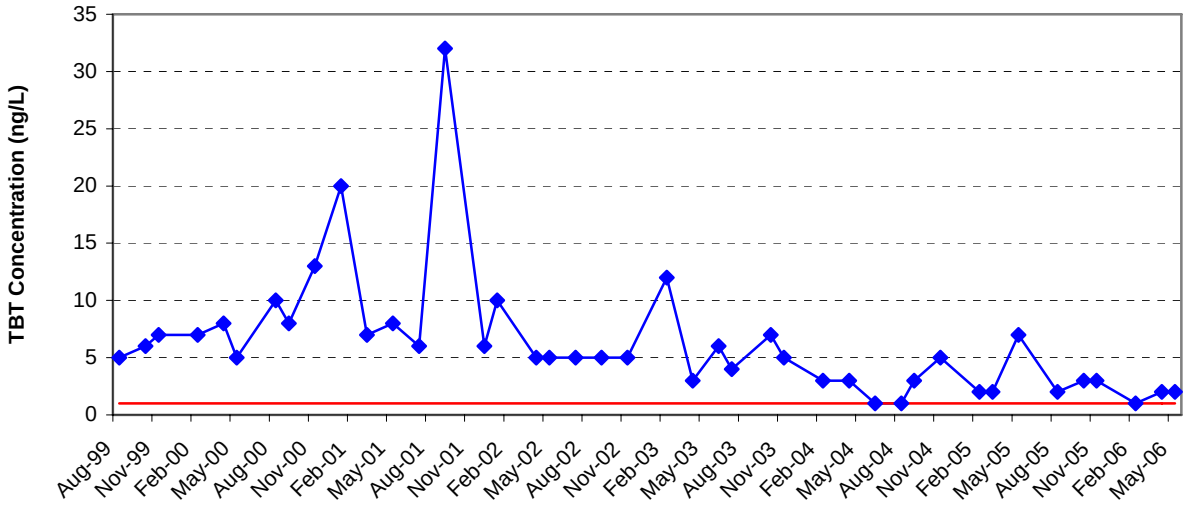
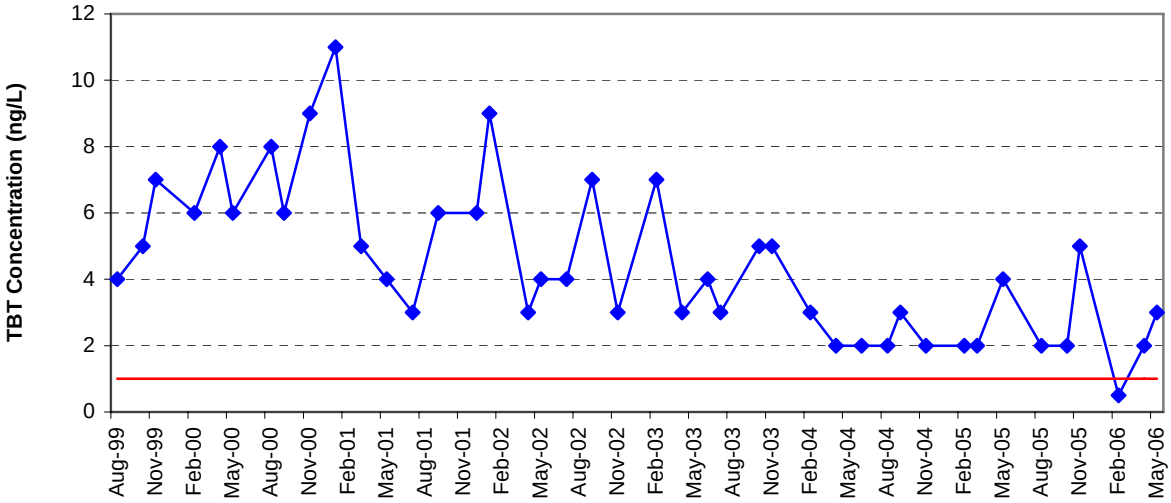
May-05
Aug-05
Nov-05
Feb-06
May-06

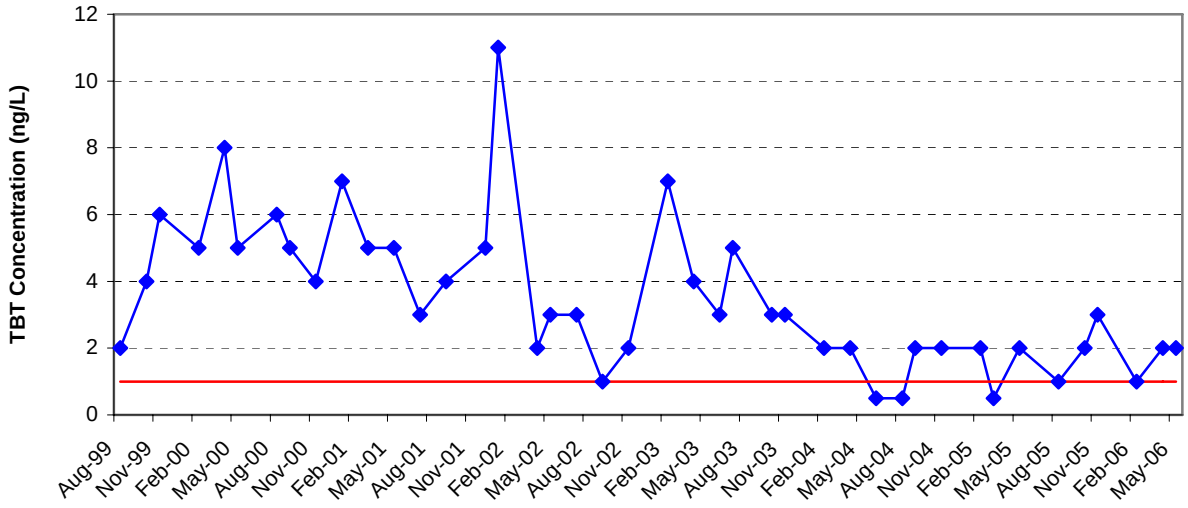
EB-A-01
Eastern Branch



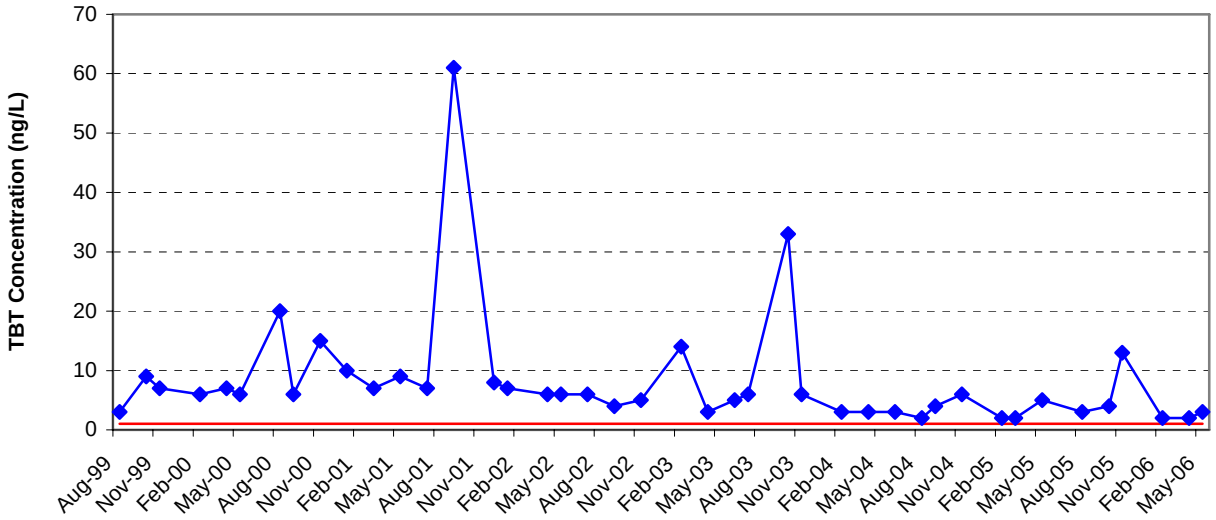
EB-A-02
Eastern Branch



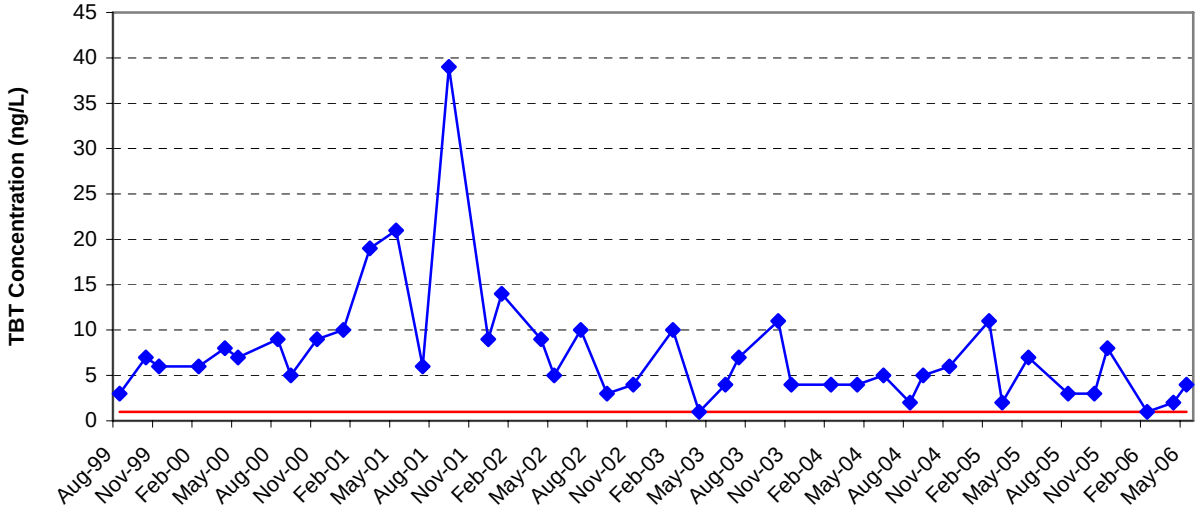
EB-B-01
Eastern Branch



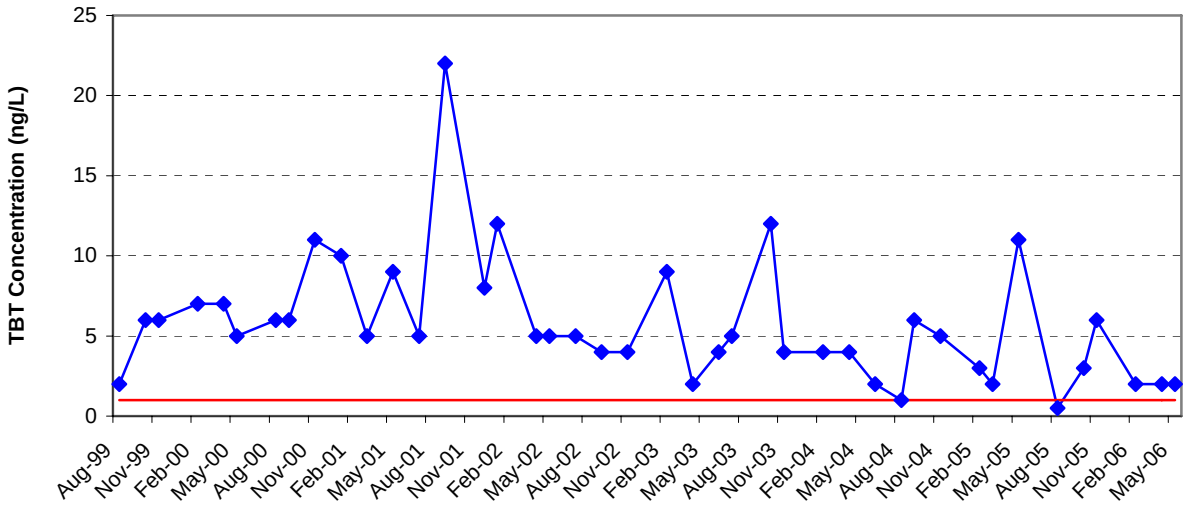
SB-A-01
Southern Branch



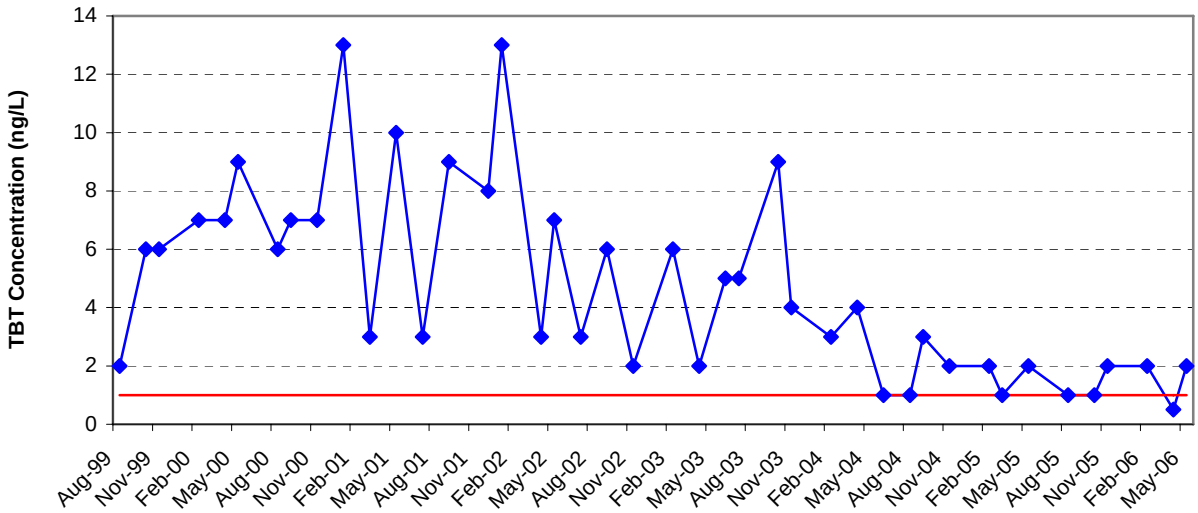
SB-B-01
Southern Branch



SB-C-01
Southern Branch



SB-D-01
Southern Branch



SB-D-02
Southern Branch