Elizabeth River TBT Monitoring
Report on TBT Methodology Detection Limit, Precision and Linearity

May 13, 1999

A Report to:
The Virginia Department
of Environmental Quality
Tidewater Regional Office
5636 Southern Blvd.
Virginia Beach VA 23462
Project Officer: Bert Parolari, Jr.

Submitted by:

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Michael A. Unger, Ph.D.

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Purpose

The purpose of this project was to determine the accuracy, precision, linearity and Method Detection Limit (MDL) for tributyltin (TBT) analytical techniques available at the Virginia Institute of Marine Science prior to implementing an environmental monitoring program in the Elizabeth River, Virginia. This precursory work is to document the ability of analytical techniques to accurately detect TBT in ambient water samples at concentrations of 1 ng/L and greater. The methodology used for this work was described in detail in:

Unger, M.A., 1996. **A Manual for the Analysis of Butyltins in Environmental Samples,** prepared for the Virginia Department of Environmental Quality by the Virginia Institute of Marine Science. VIMS Special Report in Marine Science and Ocean Engineering #338.

Preparation of Saline Water Matrix

All samples for the determination of accuracy, precision, linearity and method detection limits were made up in 20 parts per thousand (ppt) seawater to represent the sample matrix found in Chesapeake Bay waters. The artificial seawater mix, Instant Ocean©, was used to prepare sample blanks at 20 ppt for this purpose. Analysis of these prepared blanks showed that TBT, DBT, and MBT were detected above the detection limit at; 1.1, 1.0 and 2.8 ng/L which was unacceptable. In an effort to find a suitable material for preparation of samples, the commercial mixes Hawaiian Marine Mix© and Forty Fathoms© were used to produce additional 20 ppt blank samples for analysis. Blanks prepared from these mixes did not contain detectable levels of butyltins and we selected Hawaiian Marine Mix© to use for preparation of all additional samples.

Determination of Method Detection Limit

In guidance with EPA 40CFR Part 136 *APPENDIX B-DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT-REVISION 1.11* a series of seven samples were prepared at 1 ng/L TBT in 20 ppt seawater matrix. This target MDL concentration was based on previous experience and known signal/noise for the instrument (Step 1, 40CFR Part 136, Appendix B). These samples were extracted and processed on 05-03-99 and analyzed by gas chromatography on 5-4-99 and 5-5-99. The MDL was then calculated based on the procedure 40CFR Part 136, Appendix B, step 6:

$$MDL = t_{(n-1,\ 1-\alpha = 0.99)} * S$$

where: t = 3.143 for the student's t value with n-1 degrees of freedom (6 for 7 aliquots)
S = the standard deviation of the replicate analyses

95% confidence interval estimates (LCL = lower, UCL = upper) for the MDL were calculated based on the chi square over degrees of freedom distribution for the seven aliquots analyzed.

The mean concentration for the seven replicates was 1.03 ng/L with a Standard Deviation (S) of 0.304. The calculated Method Detection Limit is:

MDL= 0.955 ng/L
LCL = 0.611 ng/L
UCL = 2.10 ng/L

Precision Analysis

To demonstrate the precision expected for the methodology, a series of five replicate samples were prepared by spiking 5 ng/L TBT into 20 ppt seawater matrix. These samples were prepared and extracted on 04-29-99 and analyzed by gas chromatography on 04-30-99. The results of these analyses were a measured concentration range of 3.8 to 5.2 ng/L with a mean of 4.5 ng/L. The standard deviation for these replicates was 0.61 (13.5%). The mean recovery for TBT in these replicate samples was 90%.

Demonstration of Linearity of TBT Analyses

To demonstrate the range of linearity possible for the method, a series of water samples (20 ppt) were spiked at TBT concentrations of 0, 1, 2, 5, 10, 25, 50 and 100 ng/L. These samples were prepared and extracted on 05-10-98 and analyzed by gas chromatography on 05-10-99 and 05-11-99. The results of these analyses have been plotted to show the measured vs. spiked concentrations. The slope of the resulting regression was 0.9699 with a R^2 value of 0.996. This demonstrates that the method is capable of producing a linear response through the range of 1 to 100 ng/L.

Tables, Data Reports, Chromatograms

Tables of all data produced are included with this report and copies of gas chromatograms and data system reports are appended.

Table 1. Determination of Method Detection Limit
All samples prepared in 20 ppt artificial seawater
Concentrations reported as TBT Cation (ng/L)
Samples prepared and analyzed May 3-5, 1999

Sample	Spiked Concentration	Measured Concentration
Blank	0	0.0
Replicate A	1.1	1.3
Replicate B	1.1	1.3
Replicate C	1.1	0.9
Replicate D	1.1	0.6
Replicate E	1.1	0.8
Replicate F	1.1	1.4
Replicate G	1.1	0.9

Mean Concentration = 1.03 ng/L
Standard Deviation (S) = 0.304
Calculated MDL = 0.955 ng/L
Calculated LCL = 0.611
Calculated UCL = 2.10

Principal Investigator Michael A. Unger

Michael A. Unger, Ph.D.

Table 2. Precision Analysis
All samples prepared in 20 ppt artificial seawater
Concentrations reported as TBT Cation (ng/L)
Samples prepared and analyzed April 29-30, 1999

Sample	Spiked Concentration	Measured Concentration
Blank	0	0.0
Replicate A	5.0	4.7
Replicate B	5.0	5.2
Replicate C	5.0	3.9
Replicate D	5.0	3.8
Replicate E	5.0	4.8

Mean Concentration = 4.5 ng/L
Standard Deviation = 0.61 (13.5%)
Mean Recovery of TBT = 90%

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Table 3. Demonstration of Linearity of TBT Analysis
All samples prepared in 20 ppt artificial seawater
Concentrations reported as TBT Cation (ng/L)
Samples prepared and analyzed May 10-11, 1999

Sample	Spiked Concentration	Measured Concentration
Blank	0	0
1 ng/L	1.1	1.3
2 ng/L	2.1	2.5
5 ng/L	5.0	4.0
10 ng/L	10.0	9.9
25 ng/L	25.1	23.7
50 ng/L	50.1	43.3
100 ng/L	100.2	100.0

Regression Analysis:

$Y = X * 0.9699$
$R^2 = 0.996$

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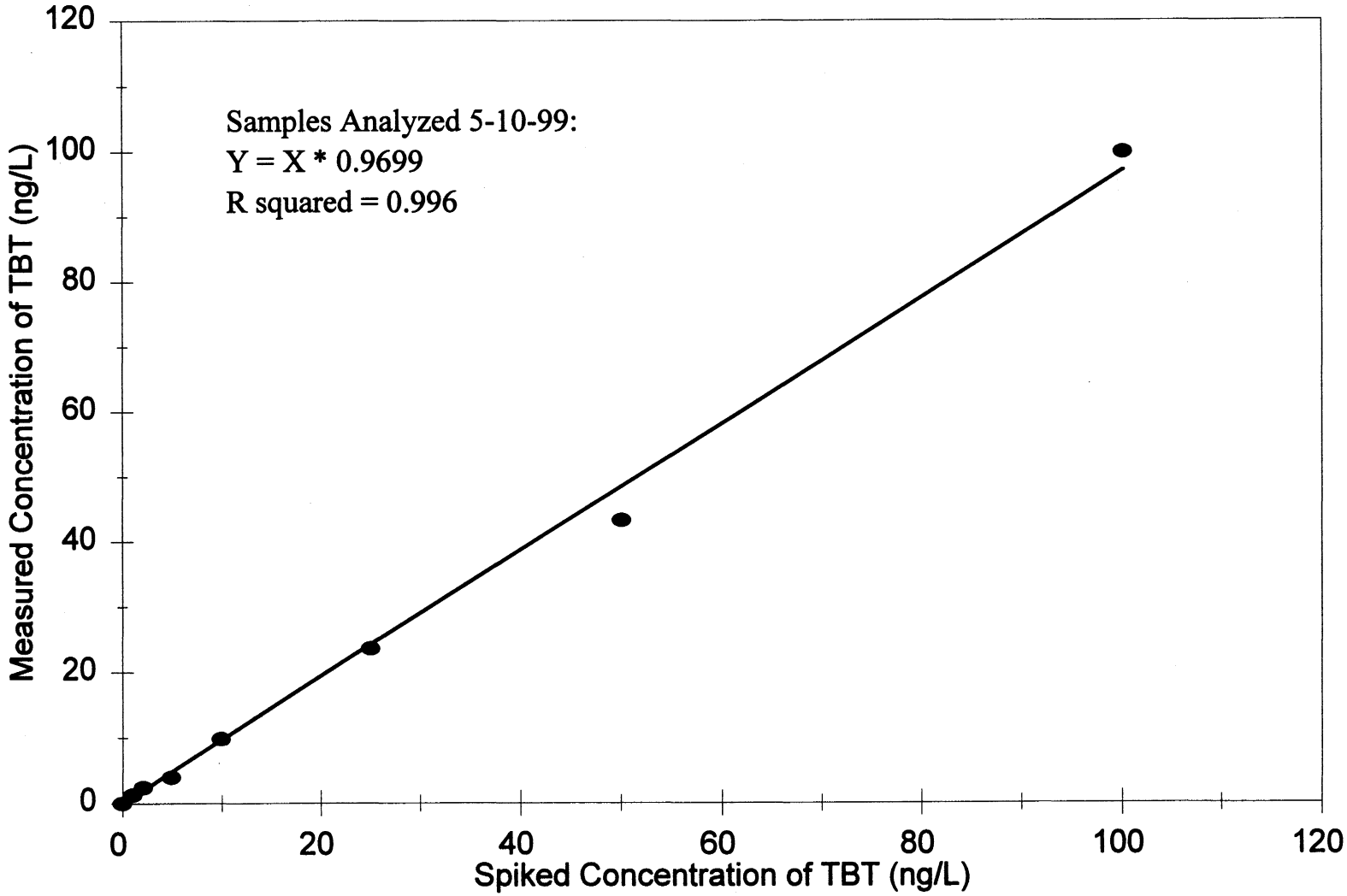
Linearity of TBT Analysis

0, 1, 2, 5, 10, 50, 100 ng/L TBT



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-31D DEQ BLANK 05-03-99 Report No : 30.01
EXTRACTED ON 5-03-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG946L.RES

Run Time : 16.02 Minutes Injected on Tue May 4, 1999 1:15:17 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL = 0.4ng/l

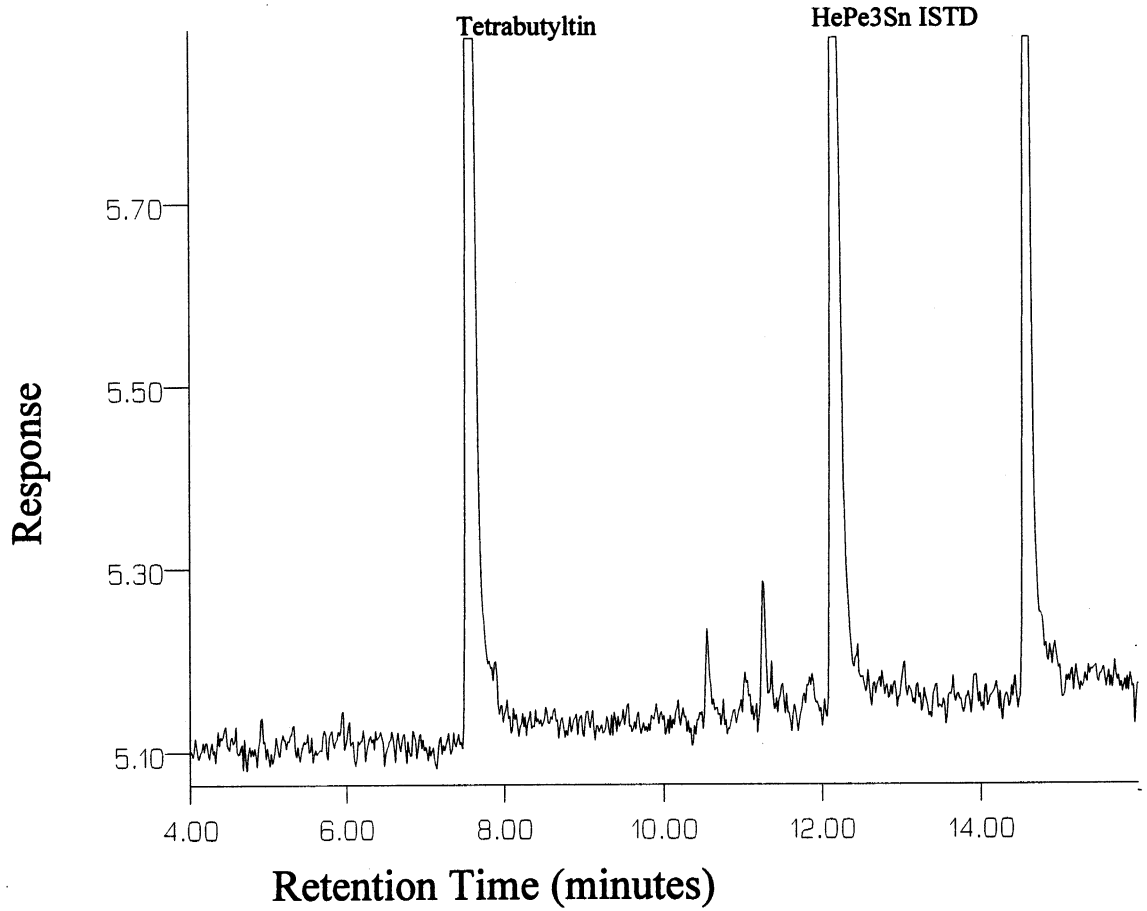
Pk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.55	#7.53	.0292	23647	FF	45.82	TETRABUTYLTIN NOTBT[1], DBT[2], MBT[3]
2	12.13	#12.13	1.0000	25169	FF		HePe3Sn internal std

Total Area : 48816 Total ng/l : 45.819

Report Time : Wed May 5, 1999 10:46:54 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

Spiked Water Blank 05-03-99



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-32A DEQ 1 NG/L A 05-03-99 Report No : 31.02
EXTRACTED ON 5-03-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG947L.RES

Run Time : 16.02 Minutes Injected on Tue May 4, 1999 1:39:07 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.5ng/l

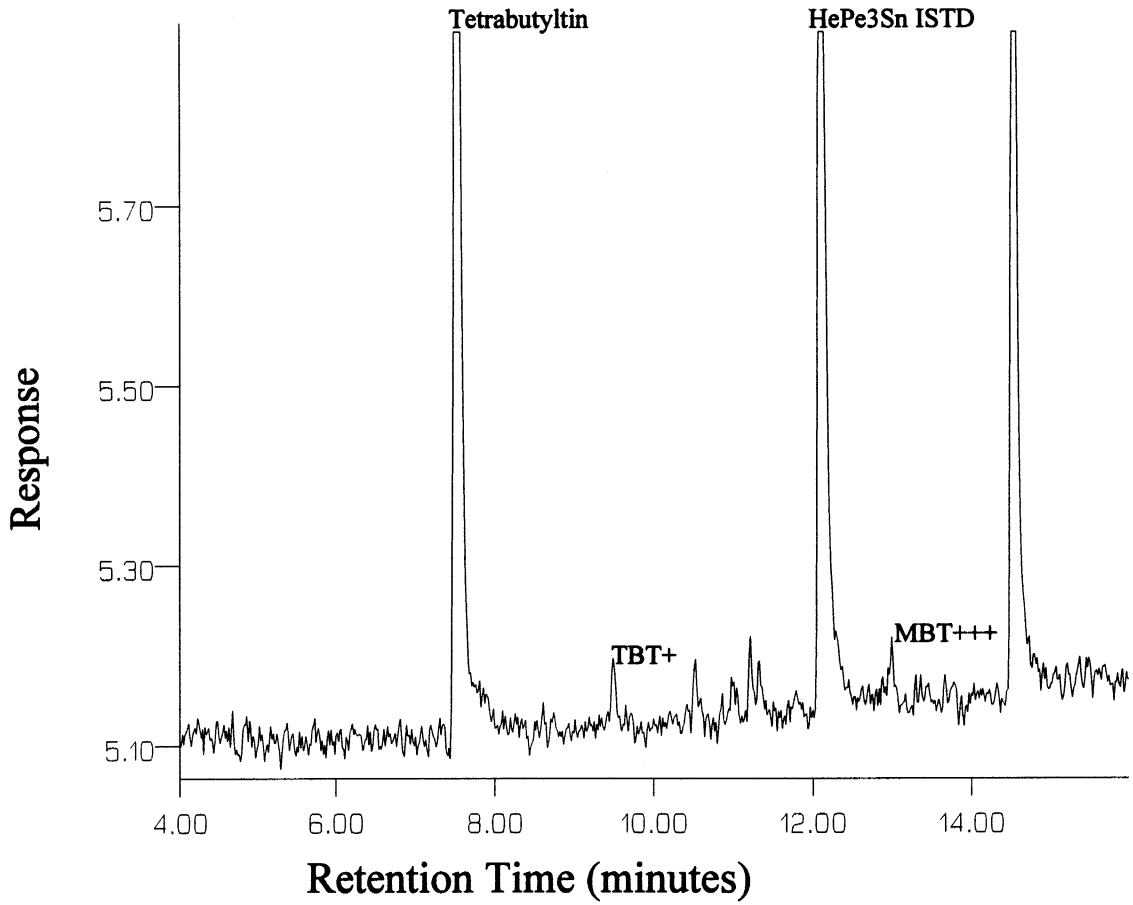
PK#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.50	#7.53	.0292	17011	FF	46.87	TETRABUTYLTIN
2	9.48	#9.52	.0244	571	FF	1.32	TBT+ 1.3ng/l TBT+
3	11.32	#11.35	.0196	203	FF	.38	DBT++ ~~0.4ng/l~~ DBT+2
4	12.10	#12.13	1.0000	17700	FF		NePa3Sn internal std
5	12.99	#13.03	.0148	380	FF	.53	MBT+++ 0.5ng/l MBT+2

Total Area : 35865 Total ng/l : 49.087

Report Time : Wed May 5, 1999 10:52:49 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water A: 1 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-32B DEQ 1 NG/L B 05-03-99 Report No : 32.02
EXTRACTED ON 5-03-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG948L.RES

Run Time : 16.00 Minutes Injected on Tue May 4, 1999 2:01:47 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.5 ng/l

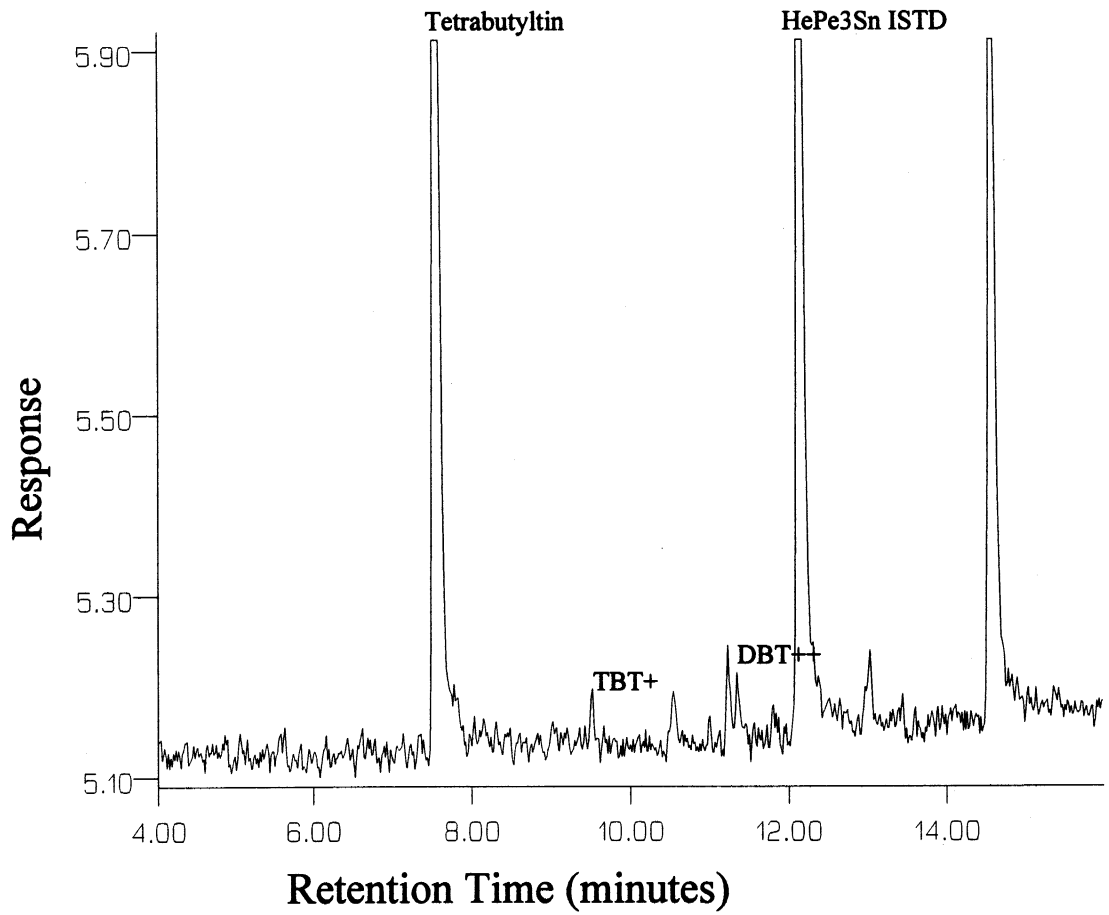
Pk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.52	#7.53	.0292	17098	FF	45.41	TETRABUTYLTIN
2	9.52	#9.52	.0244	566	FF	1.26	TBT+ 1.3ng/l TBT+
3	11.34	#11.35	.0196	479	FF	.85	DBT++ 0.8ng/l DBT++
4	12.12	#12.13	1.0000	18360	FF		NaPe3Sn internal std
5	13.02	#13.03	.0148	347	FF	.47	MBT+++ NO MBT+3

Total Area : 36850 Total ng/l : 47.990

Report Time : Wed May 5, 1999 10:56:48 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water B: 1 ng/L



3350 LOOR REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-32C DEQ 1 NG/L C 05-03-99 Report No : 33.02
EXTRACTED ON 5-03-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG949L.RES

Run Time : 16.00 Minutes Injected on Tue May 4, 1999 2:24:51 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL = 0.5 ng/l

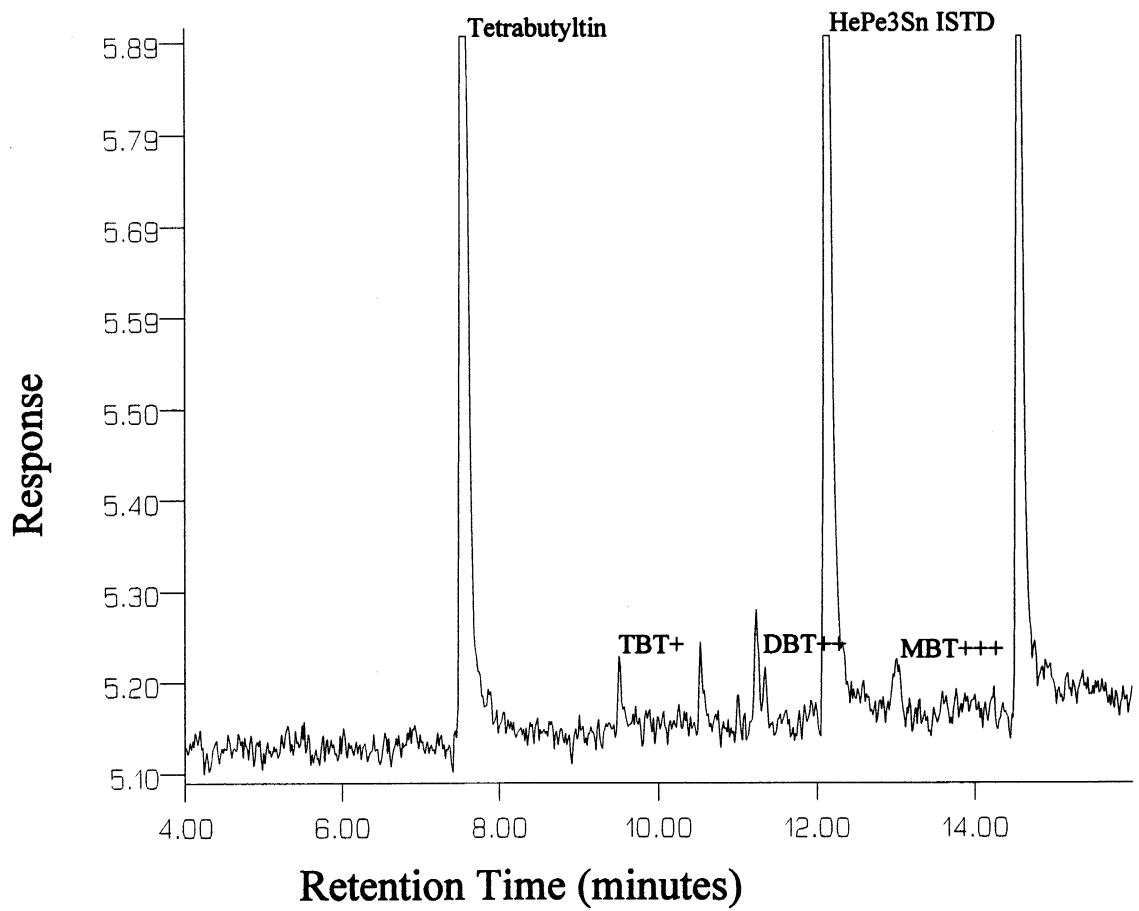
Pk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.52	#7.53	.0292	19510	FF	47.82	TETRABUTYLTIN
2	9.50	#9.52	.0244	423	FF	.87	TBT+ 0.9 ng/l TBT+
3	11.33	#11.35	.0196	450	FF	.74	DBT++ 0.7 ng/l DBT+2
4	12.12	#12.13	1.0000	19896	FF		H4Pe3Sn internal std
5	13.00	#13.03	.0148	457	FF	.57	MBT+++ 0.6 ng/l MBT+3

Total Area : 40736 Total ng/l : 49.993

Report Time : Wed May 5, 1999 11:02:27 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water C: 1 ng/L



950 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-32D DEQ 1 NG/L D 05-03-99 Report No : 34.02
EXTRACTED ON 5-03-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG950L.RES

Run Time : 16.00 Minutes Injected on Tue May 4, 1999 2:49:26 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL = 0.5 ng/l

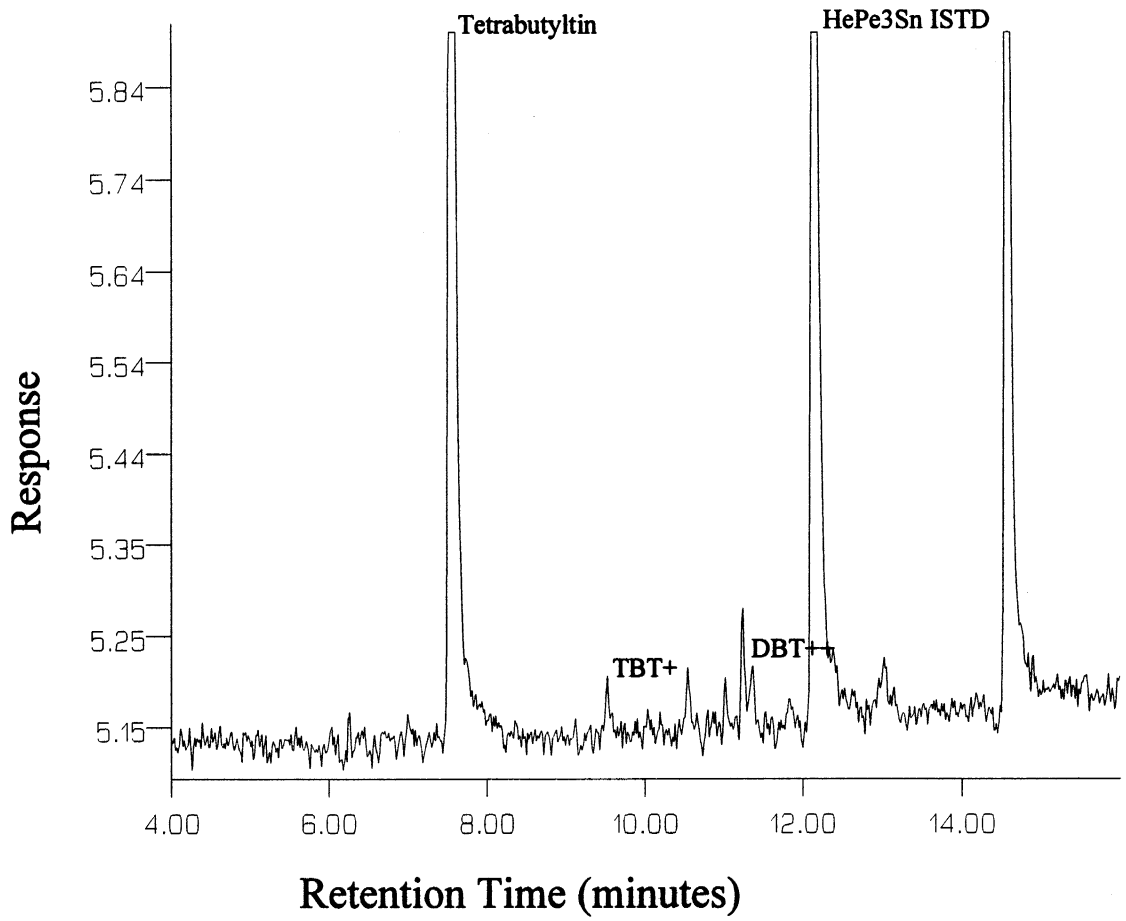
R#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.53	#7.53	.0292	18225	FF	42.71	TETRABUTYLTIN
2	9.51	#9.52	.0244	282	FF	.55	TBT+ 0.6ng/l TBT+
3	11.37	#11.35	.0196	537	FF	.85	DBT++ 0.8ng/l DBT+2
4	12.13	#12.13	1.0000	20808	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	288	FF	.34	MBT+++ NDMBT+3

Total Area : 40140 Total ng/l : 44.449

Report Time : Wed May 5, 1999 11:09:04 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water D: 1 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-33A DEQ 1 NG/L E 05-03-99 Report No : 36.02
EXTRACTED ON 5-3-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG951L.RES

Run Time : 16.00 Minutes Injected on Wed May 5, 1999 9:27:27 am

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialIntag

DL ~ 0.4ng/l

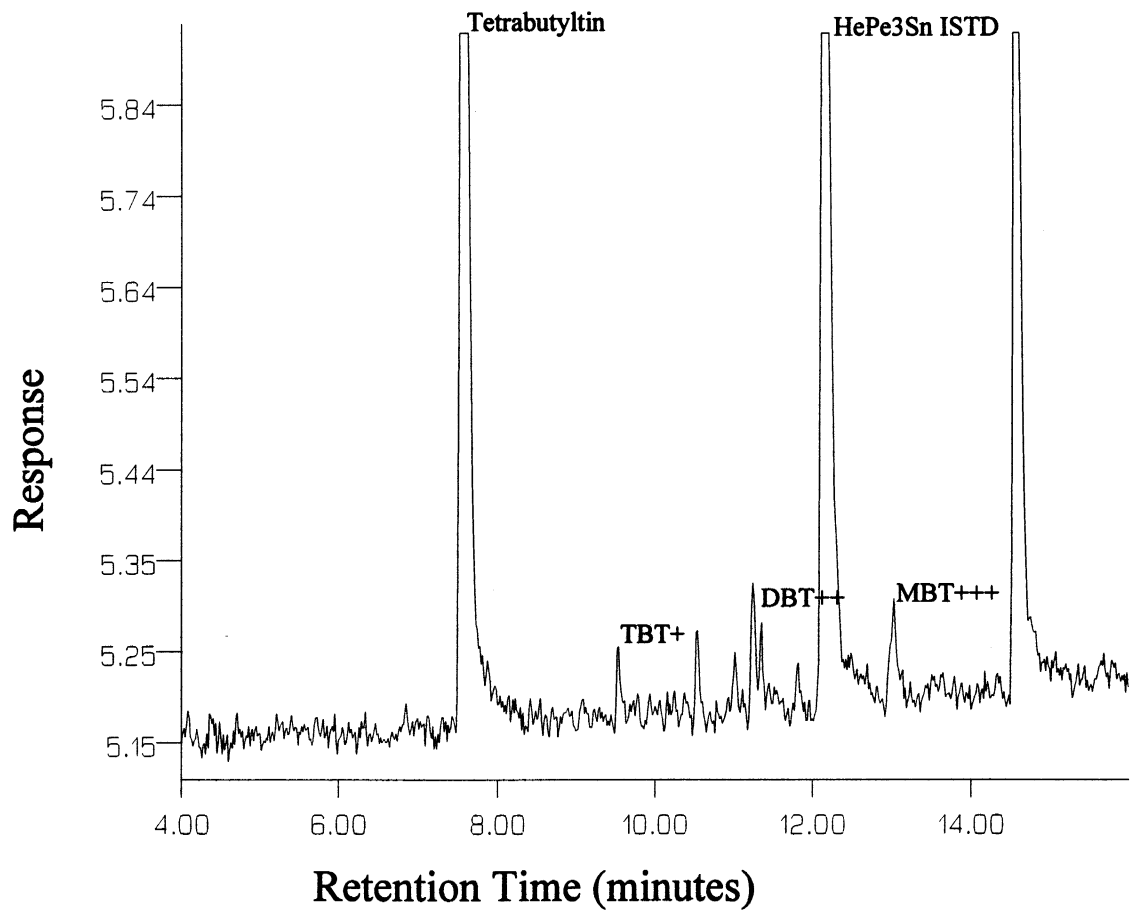
R#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.54	#7.53	.0292	24604	FF	47.75	TETRABUTYLTIN
2	9.52	#9.52	.0244	913	FF	.83	TBT+ 0.8ng/l TBT+
3	11.35	#11.35	.0196	548	FF	.71	DBT++ 0.7ng/l DBT+2
4	12.13	#12.13	1.0000	25124	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	480	FF	.47	MBT+++ 0.5ng/l MBT+3

Total Area : 51271 Total ng/l : 49.769

Report Time : Wed May 5, 1999 11:13:54 am

Method : /METHOD/TIN3/MTIN3S.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water E: 1 ng/L



EXTRACTED OK 3350 LOOP REPORT #BUTYLTINS
UserModified
Butyltin analysis by GENIE

Sample Name : SN27-33B DEQ 1 NG/L F 05-03-99 Report No : 37.03
EXTRACTED ON 5-3-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG952L.RES

Run Time : 16.00 Minutes Injected on Wed May 5, 1999 9:52:20 am

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

DL = 0.4 ng/l

SpecialInteg

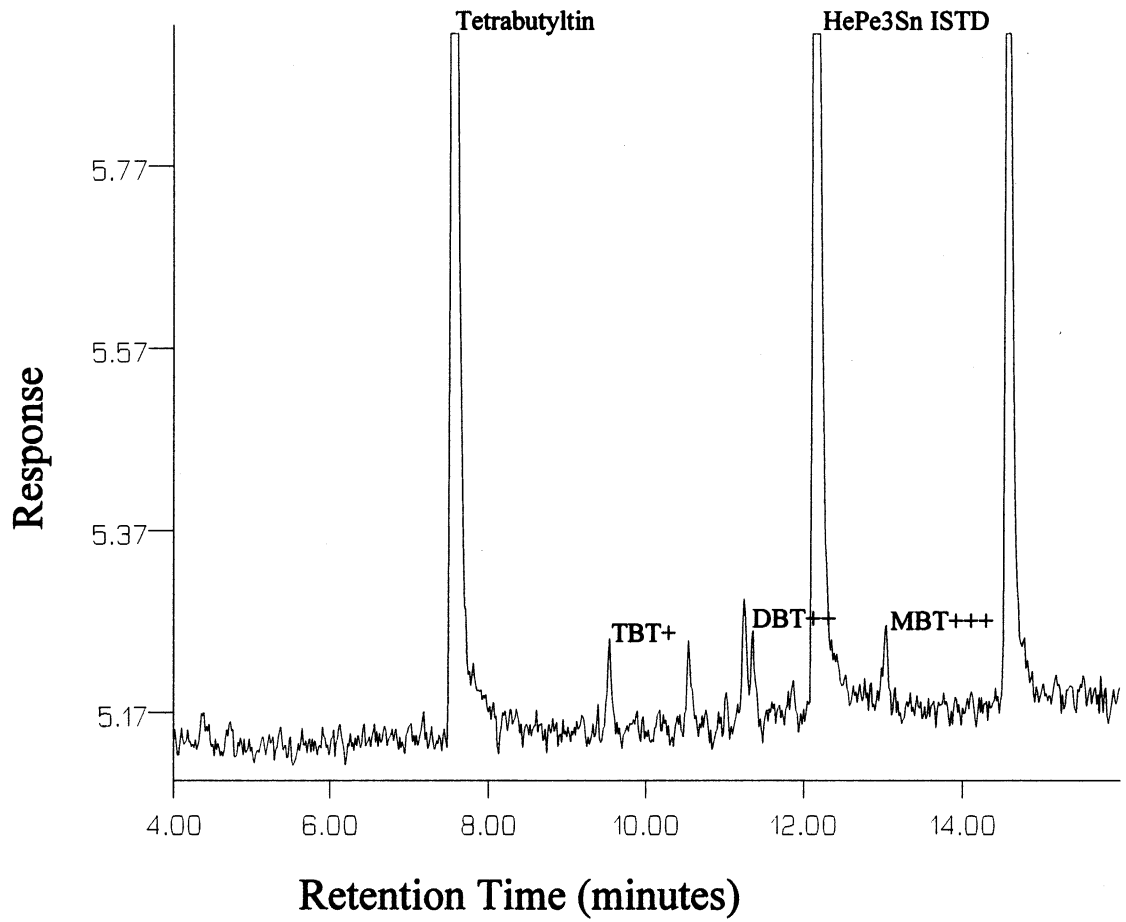
Pk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.54	#7.53	.0292	21584	FF	46.98	TETRABUTYLTIN
2	9.52	#9.52	.0244	771	FF	1.40	TBT+ 1.4 ng/l TBT+
3	11.36	#11.35	.0196	425	FF	.62	DBT++ 0.6 ng/l DBT+2
4	12.13	#12.13	1.0000	22404	FF		HePe3Sn internal std
5	13.03	#13.03	.0148	499	FF	.55	MBT+++ 0.6 ng/l MBT+3

Total Area : 45684 Total ng/l : 49.555

Report Time : Wed May 5, 1999 11:18:45 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water F: 1 ng/L



3350 LOQA REPORT - BUTYLTINS

UserModified

Butyltin analysis by GENIE

Sample Name : SN27-33C DEQ 1 NG/L G 05-03-99 Report No : 38.02
EXTRACTED ON 5-3-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG953L.RES

Run Time : 16.00 Minutes Injected on Wed May 5, 1999 10:15:55 am

% Dil-Faci	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.4ng/l

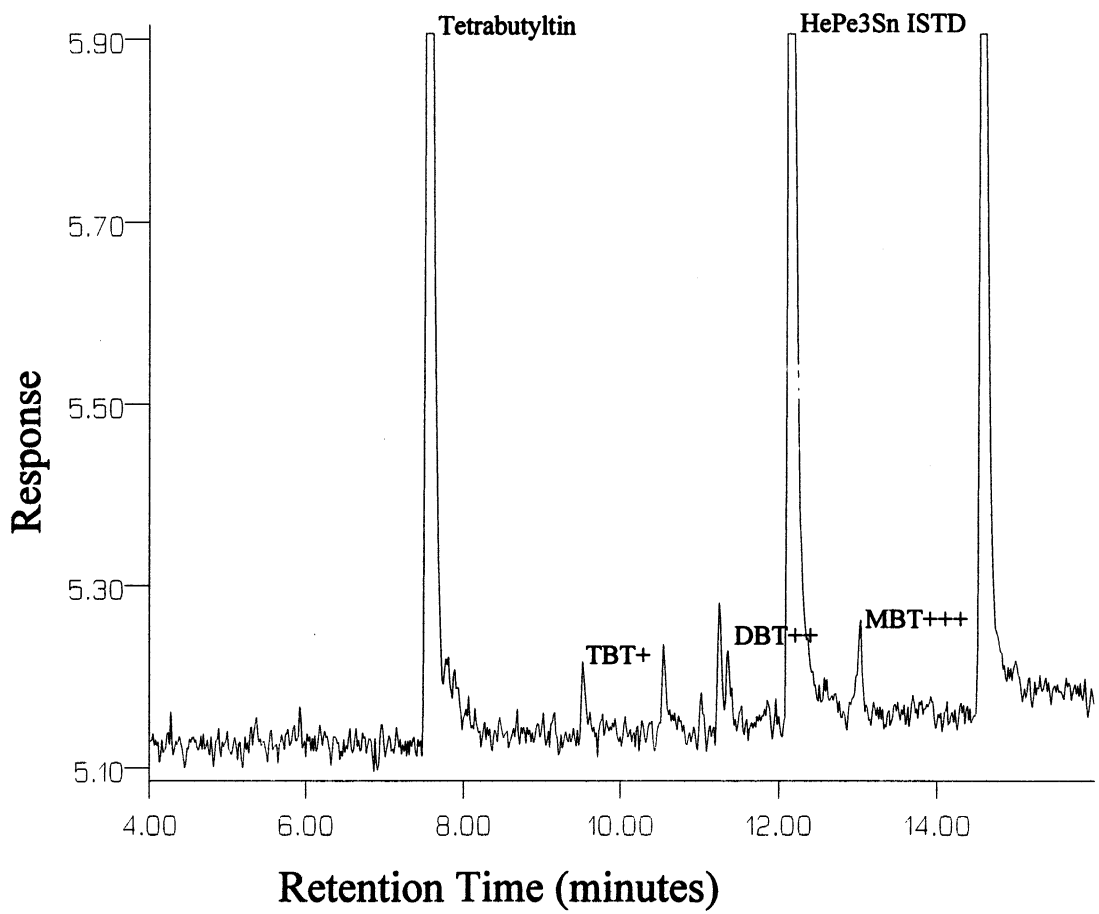
Pk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.54	#7.53	.0292	21182	FF	43.22	TETRABUTYLTIN
2	9.52	#9.52	.0244	554	FF	.94	TBT+ 0.9ng/l TBT+
3	11.39	#11.39	.0196	800	FF	1.10	DBT++ 1.1ng/l DBT++
4	12.13	#12.13	1.0000	23900	FF		HePe3Sn internal std
5	13.03	#13.03	.0148	842	FF	.87	MBT+++ 0.9ng/l MBT+++

Total Area : 47279 Total ng/l : 46.129

Report Time : Wed May 5, 1999 11:23:29 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water G: 1 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-27B DEQ BLANK 04-29-99 Report No : 22.01
EXTRACTED ON 4-29-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG928L.RES

Run Time : 16.00 Minutes Injected on Fri Apr 30, 1999 12:59:17 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.4ng/l

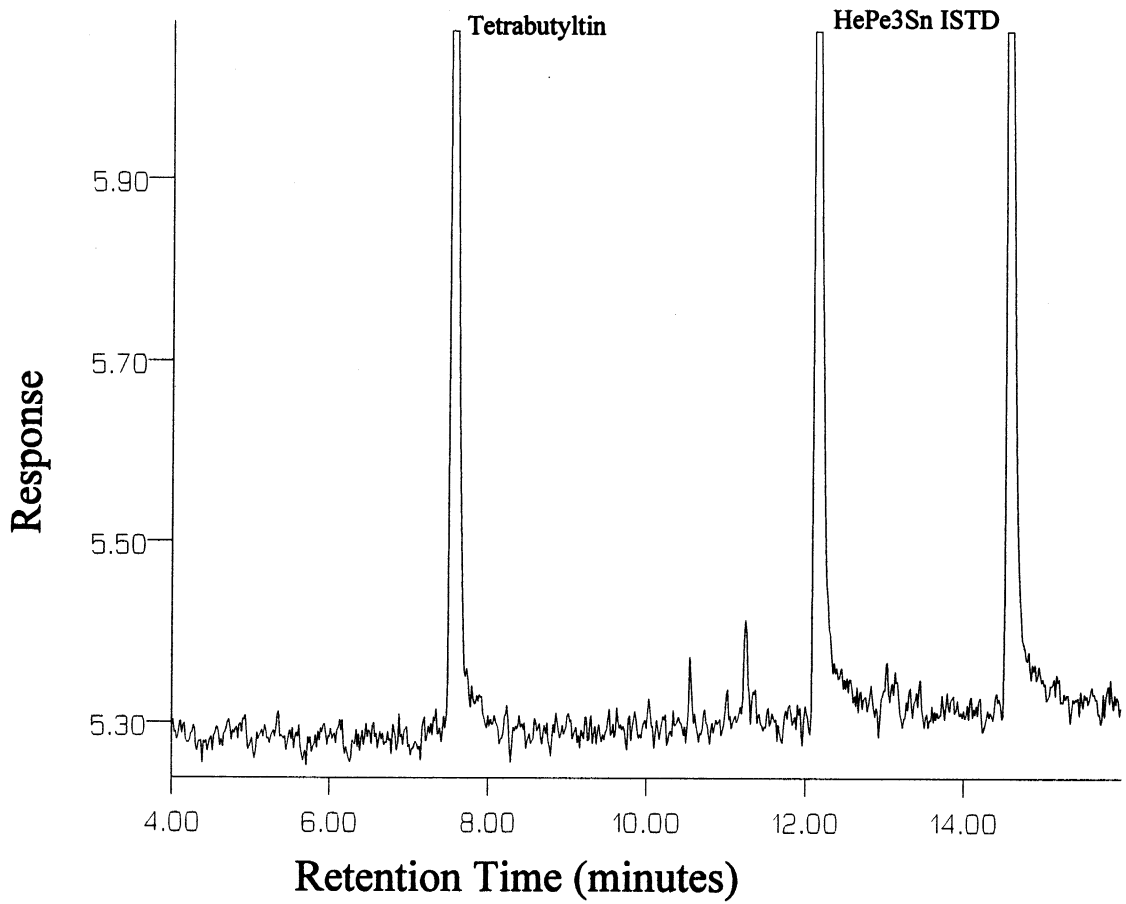
R#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.53	#7.53	.0292	16963	FF	39.41	TETRABUTYLTIN NDTBT+DBT+2,
2	12.13	#12.13	1.0000	20986	FF		HePe3Sn internal std MBT+3

Total Area : 37949 Total ng/l : 39.415

Report Time : Fri Apr 30, 1999 3:45:51 pm

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMOUTIN.FMT

TBT Spiked Water Blank: 04-29-99



3350 LOOP REPORT - BUTYLTINS

Use Modified

Butyltin analysis by GENIE

Sample Name : SN27-27C DEQ 5 NG/L A 04-29-99 Report No : 23.01

EXTRACTED ON 4-29-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG929L.RES

Run Time : 16.02 Minutes Injected on Fri Apr 30, 1999 1:21:53 pm

Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

SpecialInteg

DL ~ 0.5 ng/l

Rk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.53	#7.53	.0292	18729	FF	47.61	TETRABUTYLTIN
2	9.52	#9.52	.0244	2196	FF	4.66	TBT+ 4.7 ng/l TBT+
3	11.34	#11.35	.0196	370	FF	.63	DBT++ 0.6 ng/l DBT+ L
4	12.13	#12.13	1.0000	19182	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	287	FF	.37	MBT+++ NOMBT+3

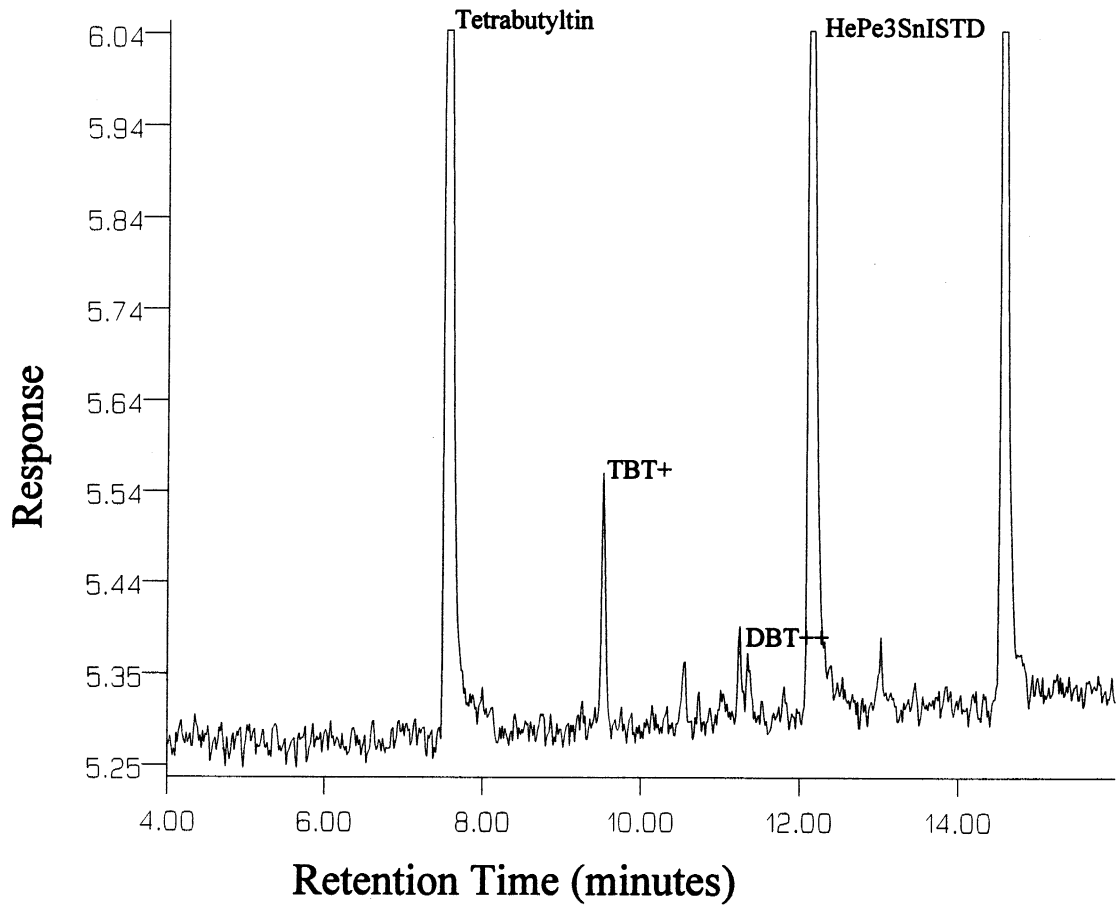
Total Area : 40763 Total ng/l : 53.270

Report Time : Fri Apr 30, 1999 3:47:43 pm

Method : /METHOD/TIN3/MTIN3G.MTH

Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water A: 5ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-27D DEQ 5 NG/L B 04-29-99 Report No : 24.01
EXTRACTED ON 4-29-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG930L.RES

Run Time : 16.02 Minutes Injected on Fri Apr 30, 1999 1:48:38 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL = 0.4 ng/l

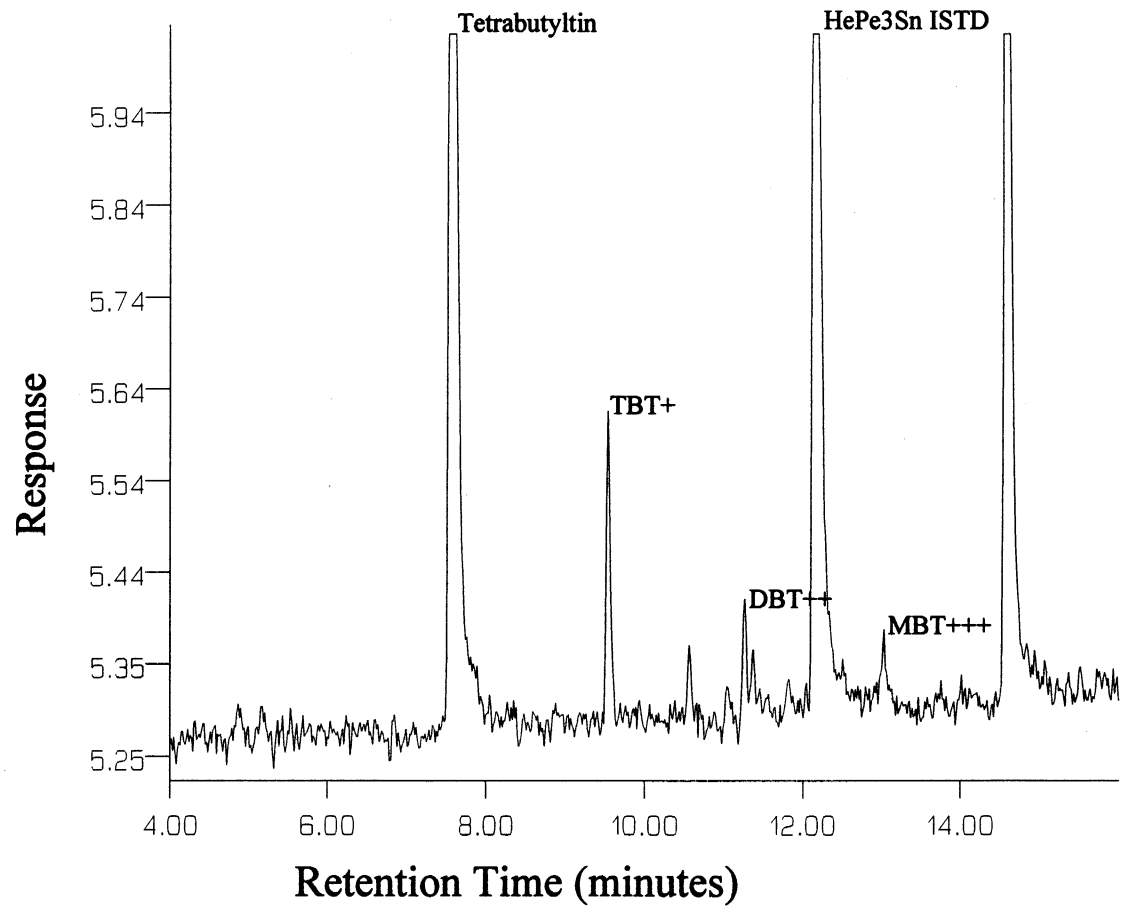
Pk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.55	#7.53	.0292	21615	FF	46.83	TETRABUTYLTIN
2	9.53	#9.52	.0244	2862	FF	5.18	TBT+ 5.2 ng/l TBT+1
3	11.37	#11.35	.0196	292	FF	.42	DBT++ 0.4 ng/l DBT+2
4	12.14	#12.13	1.0000	22509	FF		HePe3Sn internal std
5	13.04	#13.03	.0148	467	FF	.51	MBT+++ 0.5 ng/l MBT+3

Total Area : 47745 Total ng/l : 52.944

Report Time : Fri Apr 30, 1999 3:50:18 pm

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water B: 5 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified

Butyltin analysis by GENIE

Sample Name : SN27-28A DEQ 5 NG/L C 04-29-99 Report No : 25.01

EXTRACTED ON 4-29-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG931L.RES

Run Time : 16.02 Minutes Injected on Fri Apr 30, 1999 2:13:09 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL = 0.5ng/l

Pk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.52	#7.53	.0292	17279	FF	45.25	TETRABUTYLTIN
2	9.52	#9.52	.0244	1801	FF	3.94	TBT+ 3.9ng/l TBT+
3	12.12	#12.13	1.0000	18620	FF		HePe3Sn internal std ND DBT+2
4	13.02	#13.03	.0148	602	FF	.80	MBT+++ 0.8ng/l MBT+3

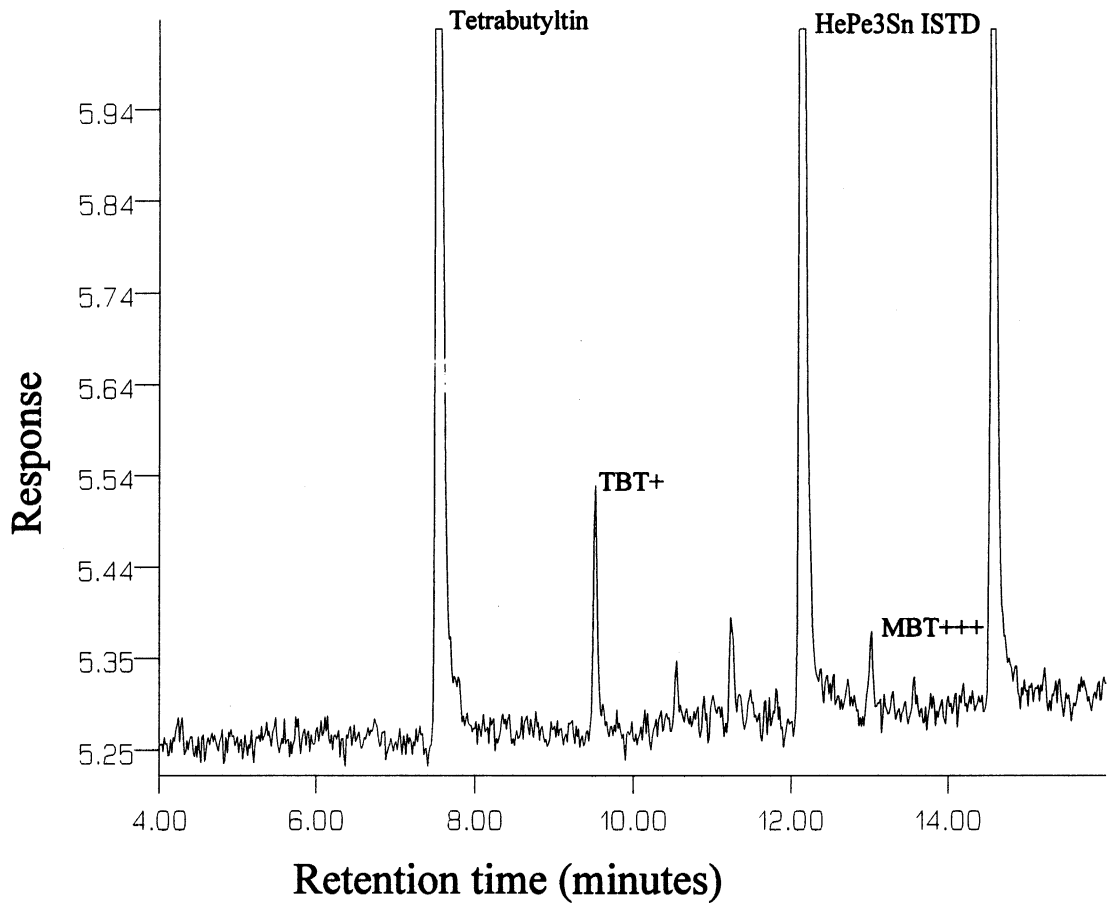
Total Area : 38303 Total ng/l : 49.994

Report Time : Fri Apr 30, 1999 3:51:56 pm

Method : /METHOD/TIN3/MTIN3G.MTH

Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water C: 5 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified

Butyltin analysis by GENIE

Sample Name : SN27-28B DEQ 5 NG/L D 04-29-99 Report No : 26.02

EXTRACTED ON 4-29-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG932LL.RES

Run Time : 16.02 Minutes Injected on Fri Apr 30, 1999 2:37:25 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL = 0.5 ng/l

Ch.#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.52	#7.53	.0292	15460	FF	43.27	TETRABUTYLTIN
2	9.51	#9.52	.0244	1422	FF	3.79	TBT+ 3.8 ng/l TBT+1
3	12.12	#12.13	1.0000	17423	FF		HePe3Sn internal std
4	13.02	#13.03	.0148	313	FF	.44	MBT+++ NO DBT+2, MBT+3

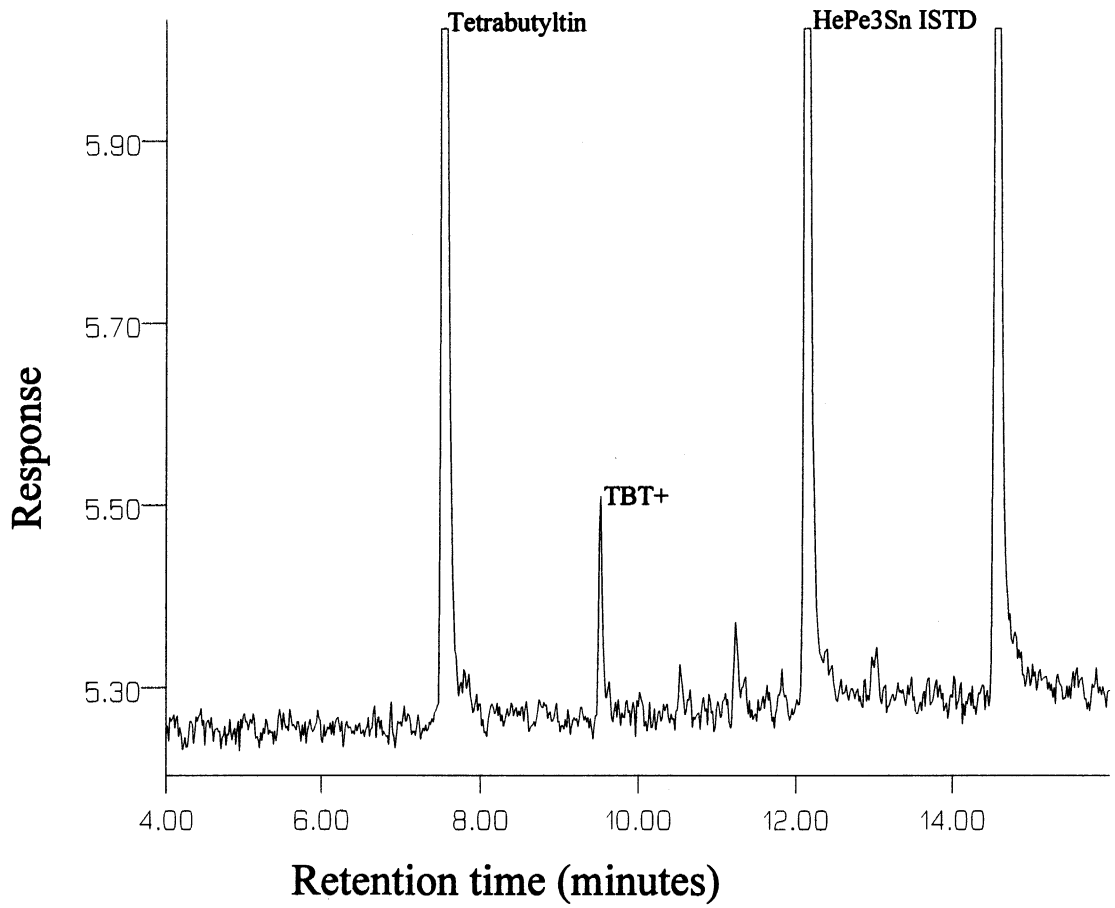
Total Area : 34817 Total ng/l : 47.508

Report Time : Fri Apr 30, 1999 3:57:06 pm

Method : /METHOD/TIN3/MTIN3G.MTH

Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water D: 5 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-28C DEQ 5 NG/L E 04-29-99 Report No : 27.01
EXTRACTED ON 4-29-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG933L.RES

Run Time : 16.00 Minutes Injected on Fri Apr 30, 1999 3:02:01 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.5ng/l

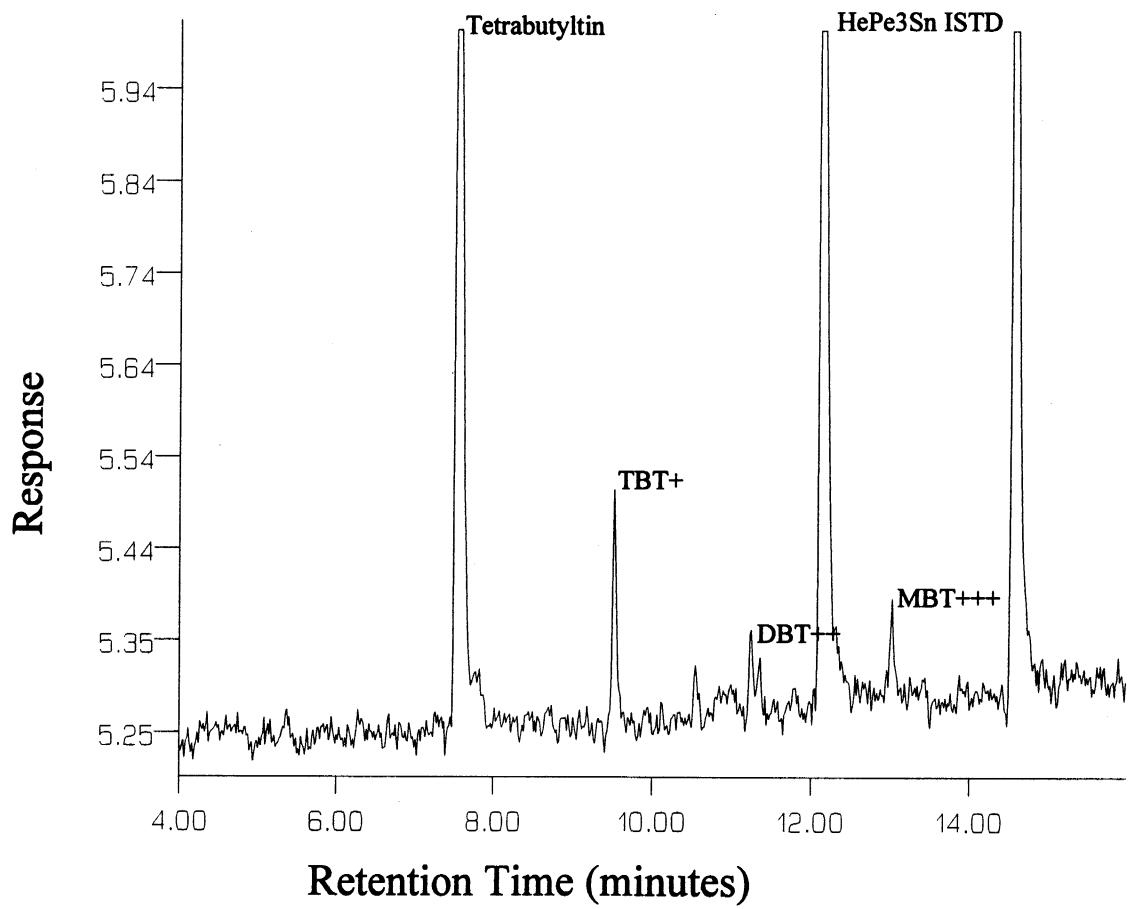
Peak	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.53	#7.53	.0292	15325	FF	42.51	TETRABUTYLTIN
2	9.52	#9.52	.0244	2078	FF	4.82	TBT+ 4.8ng/l TBT+
3	11.35	#11.35	.0196	366	FF	.68	DBT++ 0.7ng/l DBT+2
4	12.13	#12.13	1.0000	17579	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	702	FF	.99	MBT+++ 1.0ng/l MBT+3

Total Area : 36051 Total ng/l : 48.997

Report Time : Fri Apr 30, 1999 3:58:43 pm

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water E: 5 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-33D DEQ BLANK 05-10-99 Report No : 2.01
EXTRACTED ON 5-10-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG954L.RES

Run Time : 16.00 Minutes Injected on Tue May 11, 1999 12:43:27 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.4ng/l

Ki	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.54	#7.53	.0292	25623	FF	51.49	TETRABUTYLTIN
2	11.34	#11.35	.0196	348	FF	.47	DBT++
3	12.13	#12.13	1.0000	24268	FF		HePe3Sn internal std

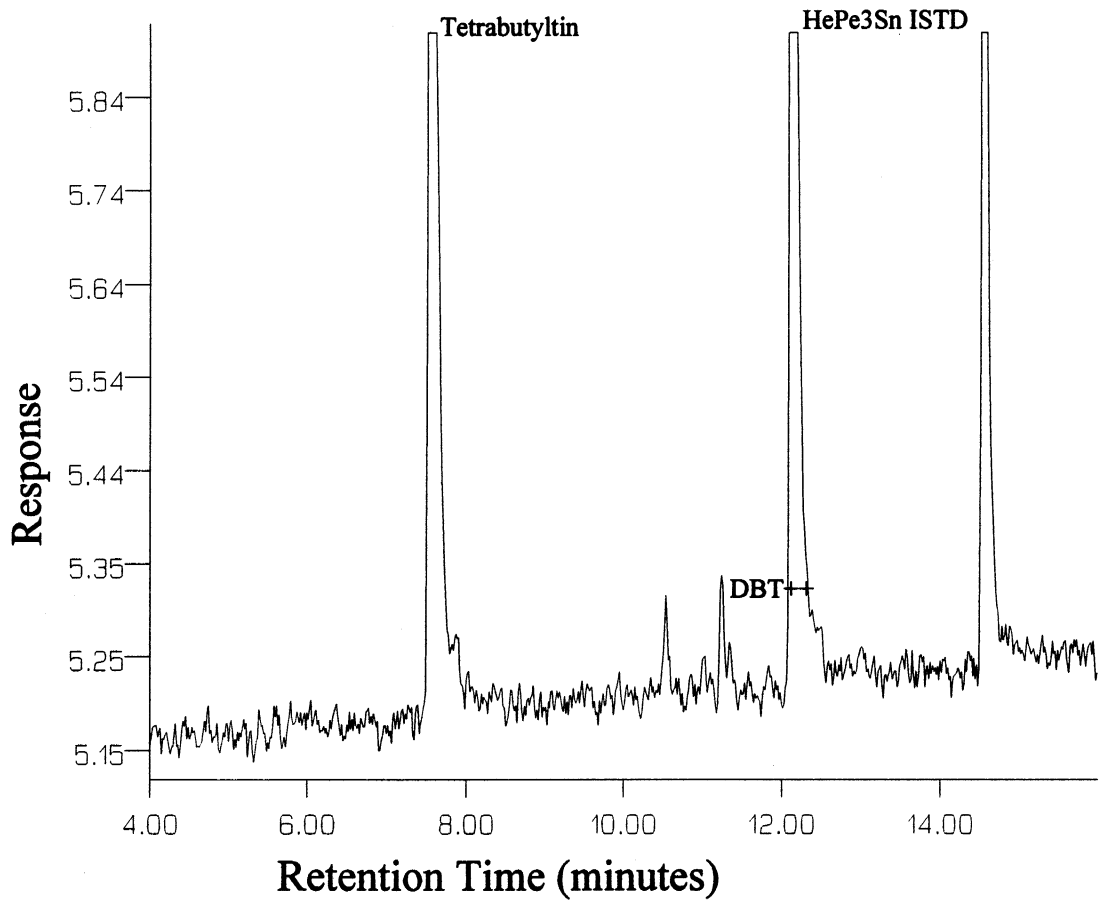
NDTBT+
0.5ng/l DBT+2
NO MBT+3

Total Area : 50230 Total ng/l : 51.955

Report Time : Wed May 12, 1999 10:28:44 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water Blank 05-10-99



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-34A DEQ 1 NG/L 05-10-99 Report No : 3.02
EXTRACTED ON 5-10-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG955L.RES

Run Time : 16.00 Minutes Injected on Tue May 11, 1999 1:07:47 pm

Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.4ng/l

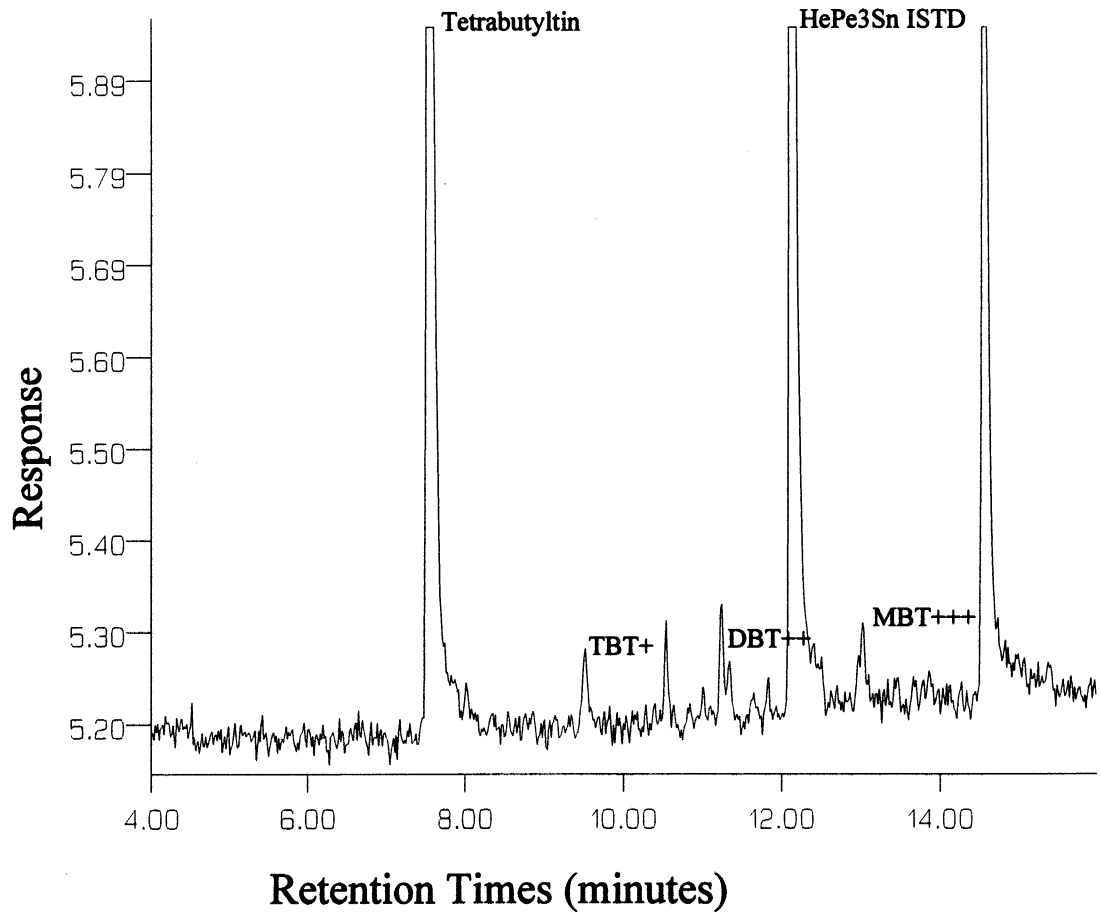
A#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.52	#7.53	.0292	20296	FF	44.88	TETRABUTYLTIN
2	9.52	#9.52	.0244	676	FF	1.31	TBT+ 1.3ng/l TBT+
3	11.34	#11.35	.0196	343	FF	.53	DBT++ 0.5ng/l DBT+2
4	12.12	#12.13	1.0000	21019	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	496	FF	.58	MBT+++ 0.6ng/l MBT+3

Total Area : 42740 Total ng/l : 49.305

Report Time : Wed May 12, 1999 10:32:57 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water: 1 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified

Butyltin analysis by GENIE

Sample Name : SN27-34B DEQ 2 NG/L 05-10-99 Report No : 4.02

EXTRACTED ON 5-10-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG956L.RES

Run Time : 16.02 Minutes Injected on Tue May 11, 1999 1:34:18 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL = 0.5 ng/l

A#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.54	#7.53	.0292	17409	FF	47.89	TETRABUTYLTIN
2	9.52	#9.52	.0244	1071	FF	2.46	TBT+ 2.5 ng/l TBT+
3	11.35	#11.35	.0196	344	FF	.64	DBT++ 0.6 ng/l DBT+2
4	12.13	#12.13	1.0000	17726	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	489	FF	.68	MBT+++ 0.7 ng/l MBT+3

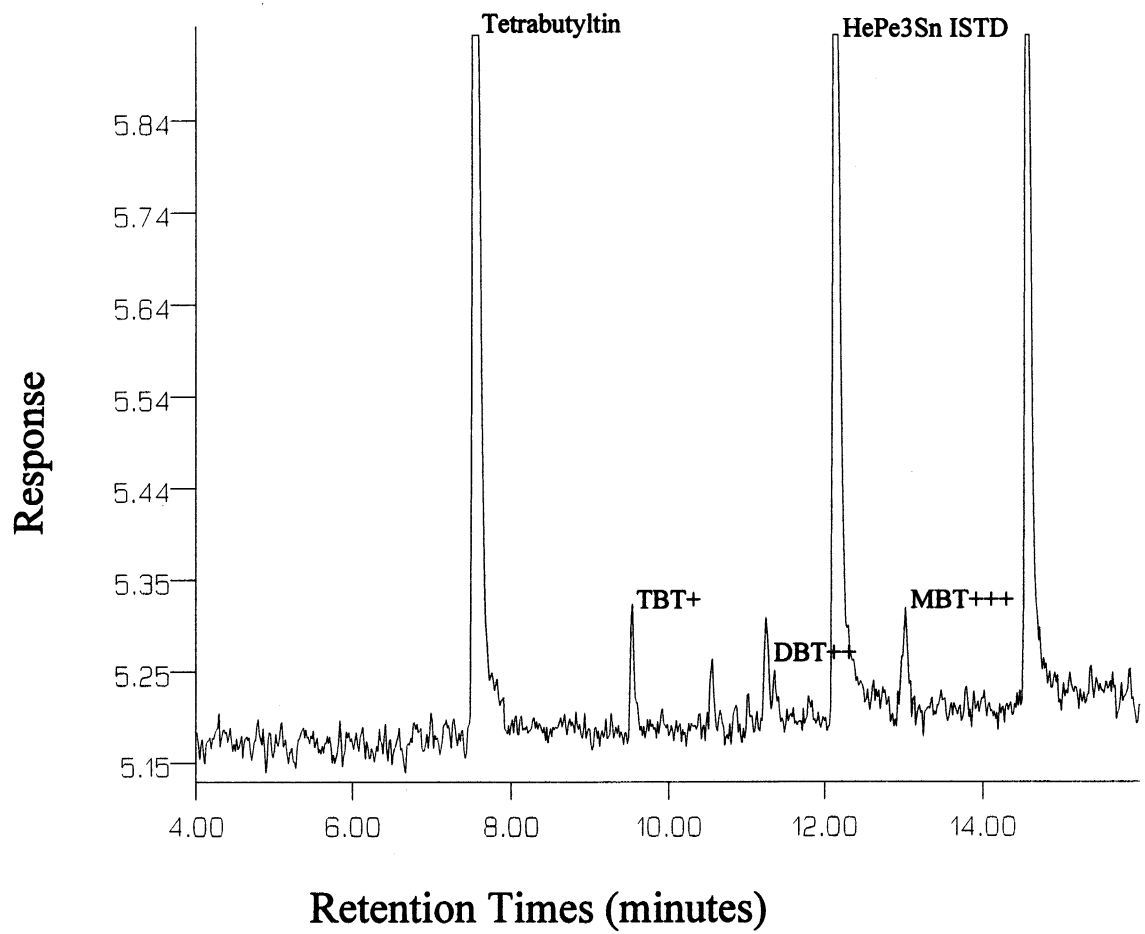
Total Area : 37039 Total ng/l : 51.673

Report Time : Wed May 12, 1999 10:35:04 am

Method : /METHOD/TIN3/MTIN3G.MTH

Report File : /FORMAT/TIN3/CMBUTIN.FMT

TBT Spiked Water: 2 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-34C DEQ 5 NG/L 05-10-99 Report No : 5.02
EXTRACTED ON 5-10-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG957L.RES

Run Time : 16.00 Minutes Injected on Tue May 11, 1999 1:58:55 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.6ng/l

%#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.52	#7.53	.0292	15653	FF	47.57	TETRABUTYLTIN
2	9.52	#9.52	.0244	1569	FF	3.98	TBT+ 4.0 ng/l TBT+
3	12.12	#12.13	1.0000	16045	FF		HePa3Sn internal std

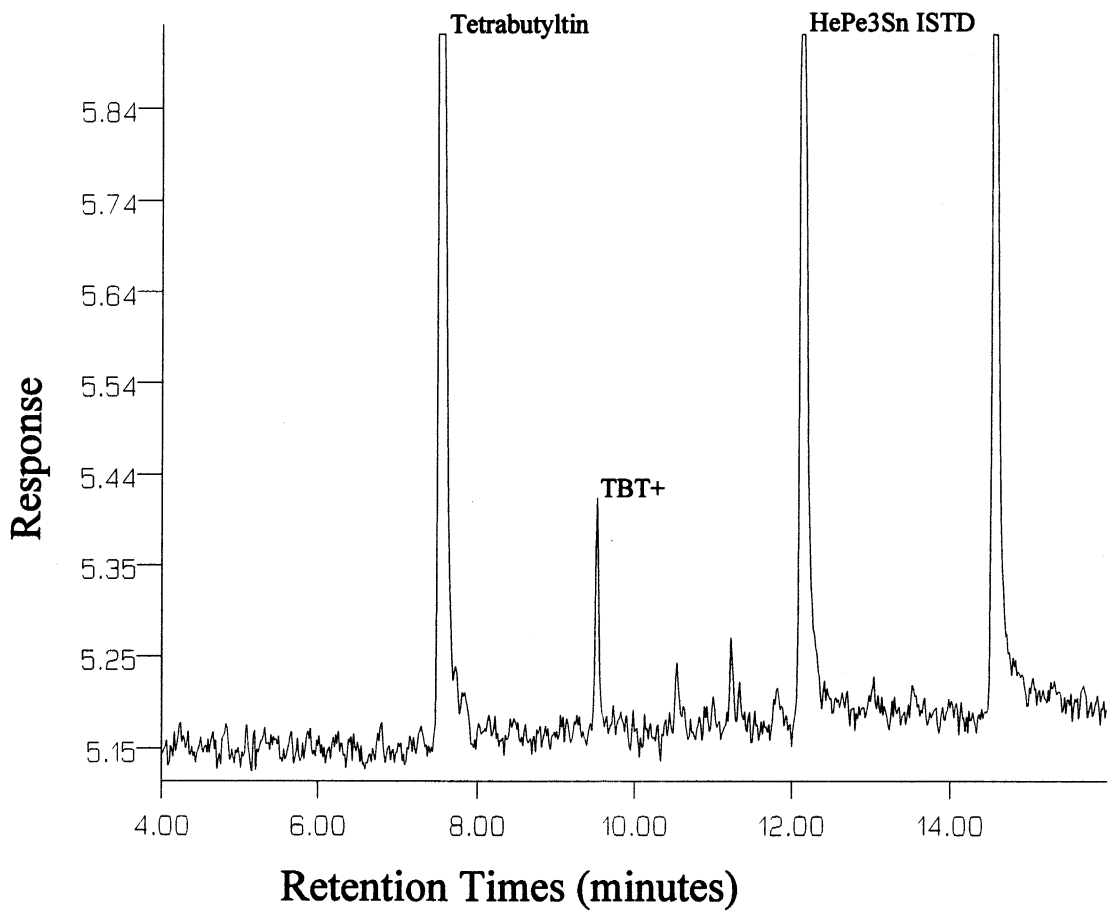
ND DBT+2, MBT+3

Total Area : 33267 Total ng/l : 51.55

Report Time : Wed May 12, 1999 10:37:53 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/SMBUTIN.FMT

TBT Spiked Water: 5 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-34D DEQ 10 NG/L 05-10-99 Report No : 6.02
EXTRACTED ON 5-10-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG958L.RES

Run Time : 16.00 Minutes Injected on Tue May 11, 1999 2:21:45 pm

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL- 0.5 ng/l

Pk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.54	#7.53	.0292	19506	FF	51.38	TETRABUTYLTIN
2	9.52	#9.52	.0244	4513	FF	9.93	TBT+ 9.9ng/l TBT+
3	11.35	#11.35	.0196	392	FF	.69	DBT++ 0.7ng/l DBT+2
4	12.13	#12.13	1.0000	18513	FF		NaPe3Sn internal std

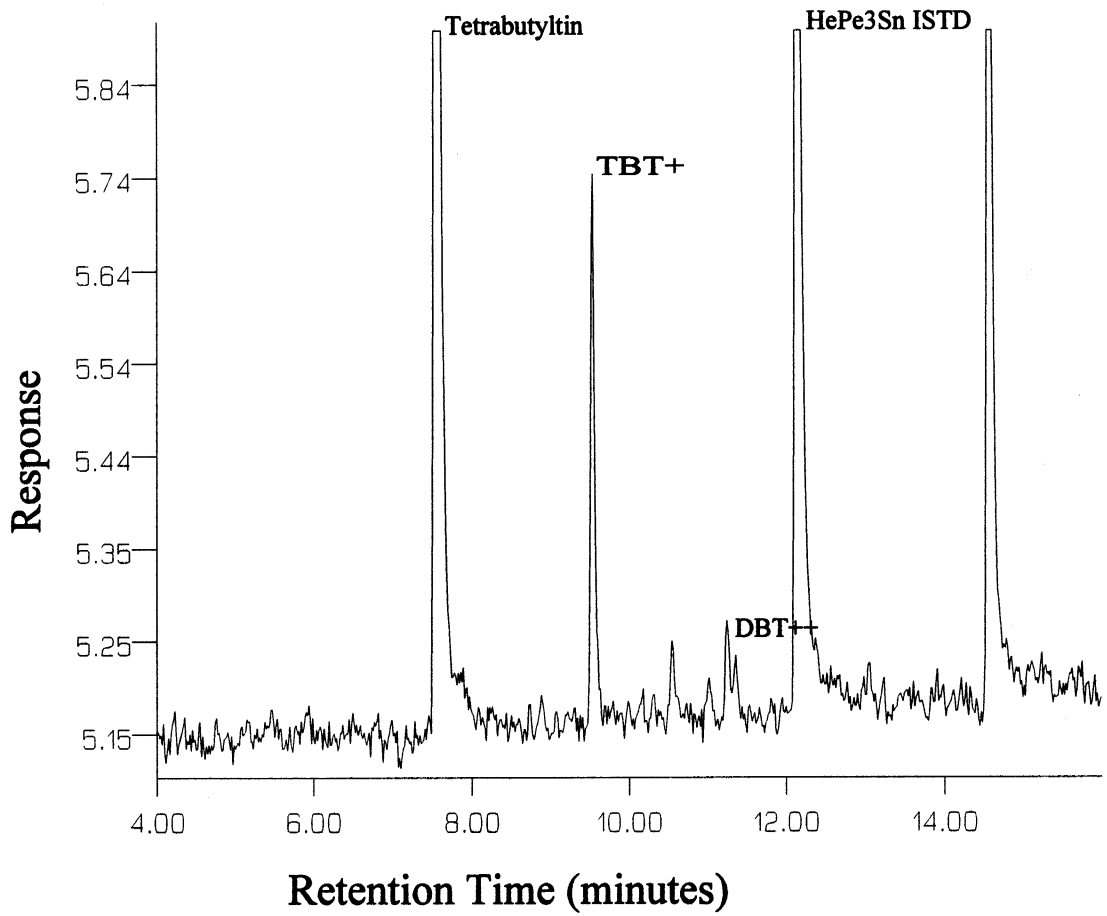
NO MBT+3

Total Area : 42924 Total ng/l : 62.007

Report Time : Wed May 12, 1999 10:40:09 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water: 10 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-35A DEQ 25 NG/L 05-10-99 Report No : 7.02
EXTRACTED ON 5-10-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG959L.RES

Run Time : 16.00 Minutes Injected on Tue May 11, 1999 2:46:26 pm

Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.5ng/l

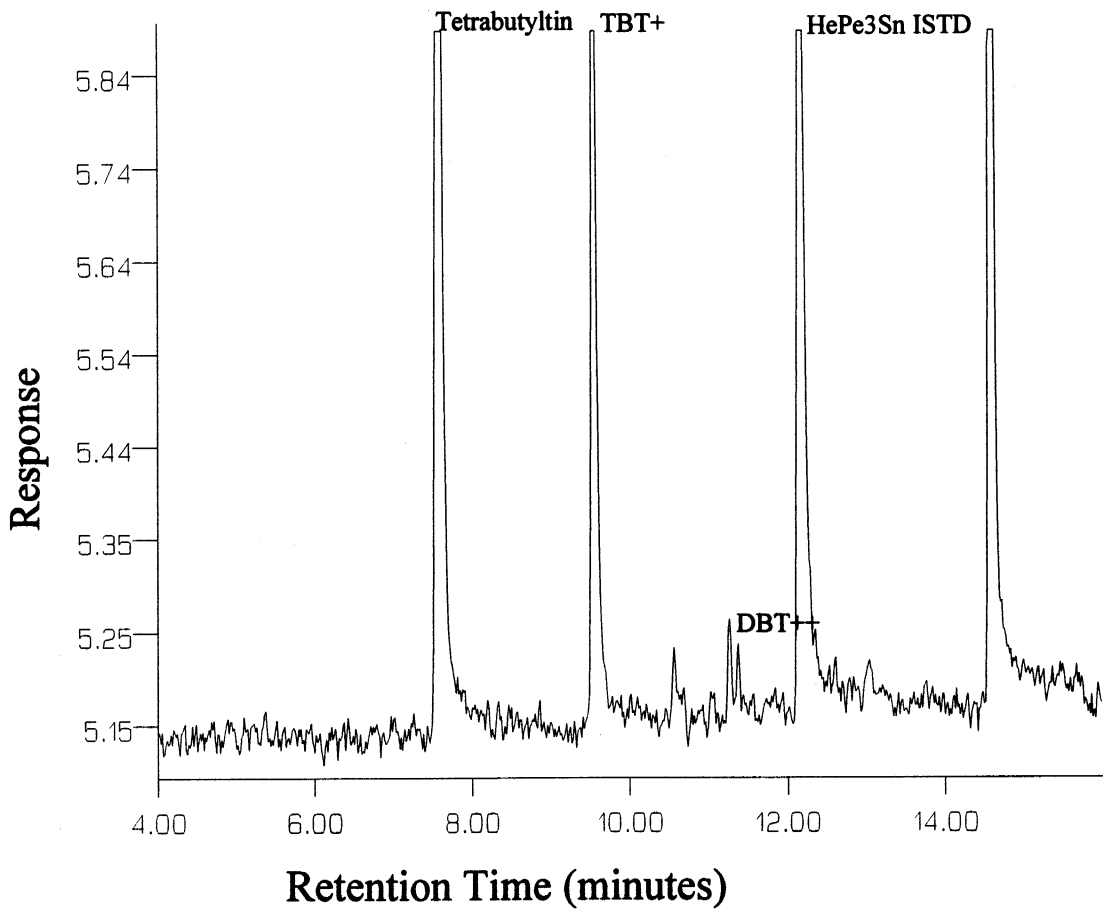
No.#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.55	#7.53	.0292	17931	FF	48.75	TETRABUTYLTIN
2	9.53	#9.52	.0244	10180	FF	27.65	TBT+ 23.7 ng/l TBT+
3	11.37	#11.35	.0196	300	FF	.56	DBT++ 0.6 ng/l DBT+2
4	12.14	#12.13	1.0000	17536	FF		MePe3Sn internal std
							ND MBT+3

Total Area : 45547 Total ng/l : 77.965

Report Time : Wed May 12, 1999 10:42:27 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water: 25 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-35B DEQ 50 NG/L 05-10-99 Report No : 9.02
EXTRACTED ON 5-10-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG960L.RES

Run Time : 16.00 Minutes Injected on Wed May 12, 1999 9:33:13 am

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL = 0.5 ng/l

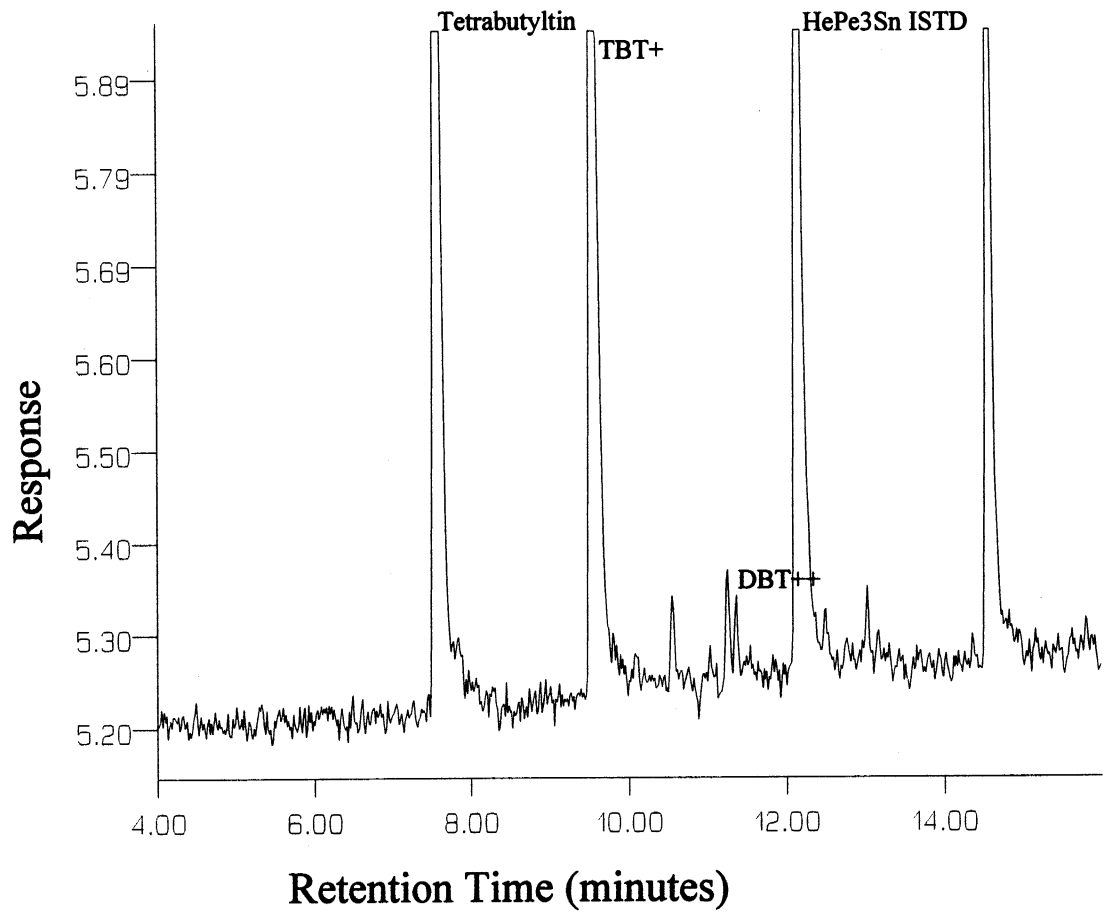
PK#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.54	#7.53	.0292	19999	FF	47.20	TETRABUTYLTIN
2	9.52	#9.52	.0244	21966	FF	43.32	TBT+ 43.3 ng/l TBT+
3	11.36	#11.35	.0196	609	FF	.97	DBT++ 1.0 ng/l DBT+2
4	12.13	#12.13	1.0000	20662	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	346	FF	.41	MBT+++ ND MBT+3

Total Area : 63582 Total ng/l : 91.895

Report Time : Wed May 12, 1999 10:45:47 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water: 50 ng/L



3350 LOOP REPORT - BUTYLTINS

UserModified
Butyltin analysis by GENIE

Sample Name : SN27-35C DEQ 100 NG/L 05-10-99 Report No : 10.02
EXTRACTED ON 5-10-99

Peak Processor : Genie Multilevel : False

Instrument : A/D_3 Calculation : InternalSTD

Result File : /RESULT/TIN3/SRG961L.RES

Run Time : 16.02 Minutes Injected on Wed May 12, 1999 10:02:05 am

% Dil-Fact	Smp-Amt	Std-Amt	% Purity	Inj Vol Ratio
100.00000	1.00000	16.70000	100.000	1.000

Run Status : RunStatusOK

EndOffBaseline

SpecialInteg

DL= 0.8 ng/l

Pk#	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.52	#7.53	.0292	12806	FF	56.62	TETRABUTYLTIN
2	9.51	#9.52	.0244	27053	FF	99.95	TBT+ 100.0 ng/l TBT+
3	11.33	#11.35	.0196	480	FF	1.43	DBT++ 1.4 ng/l DBT+2
4	12.12	#12.13	1.0000	11029	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	415	FF	.93	MBT+++ 0.9 ng/l MBT+3

Total Area : 51784 Total ng/l : 158.922

Report Time : Wed May 12, 1999 10:48:09 am

Method : /METHOD/TIN3/MTIN3G.MTH
Report File : /FORMAT/TIN3/GMBUTIN.FMT

TBT Spiked Water: 100 ng/L