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

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Virginia Institute of Marine Science

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

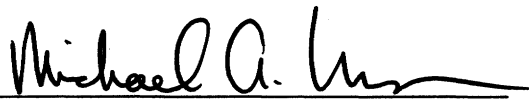May 13, 1999

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:

The Department of Environmental Sciences
The Virginia Institute of Marine Science
The College of William and Mary
Gloucester Point, Virginia 23062

Principal Investigator _____

Michael A. Unger, Ph.D.

Elizabeth River TBT Monitoring
Report on TBT Methodology Detection Limit, Precision and Linearity
May 13, 1999

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:

$$MDL = 0.955 \text{ ng/L}$$
$$LCL = 0.611 \text{ ng/L}$$
$$UCL = 2.10 \text{ ng/L}$$

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

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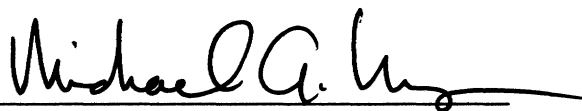
Table 2. Precision Analysis
All samples prepared in 20 ppt artificial seawater
Concentrations reported as TBT Cation (ng/L)
Samples prepared and analyzed April 29-30, 1999

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
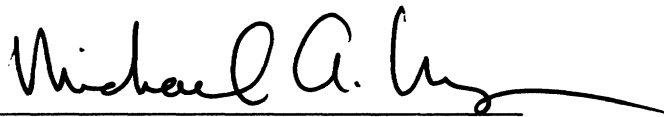
Regression Analysis:

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

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Linearity of TBT Analysis
0, 1, 2, 5, 10, 50, 100 ng/L TBT
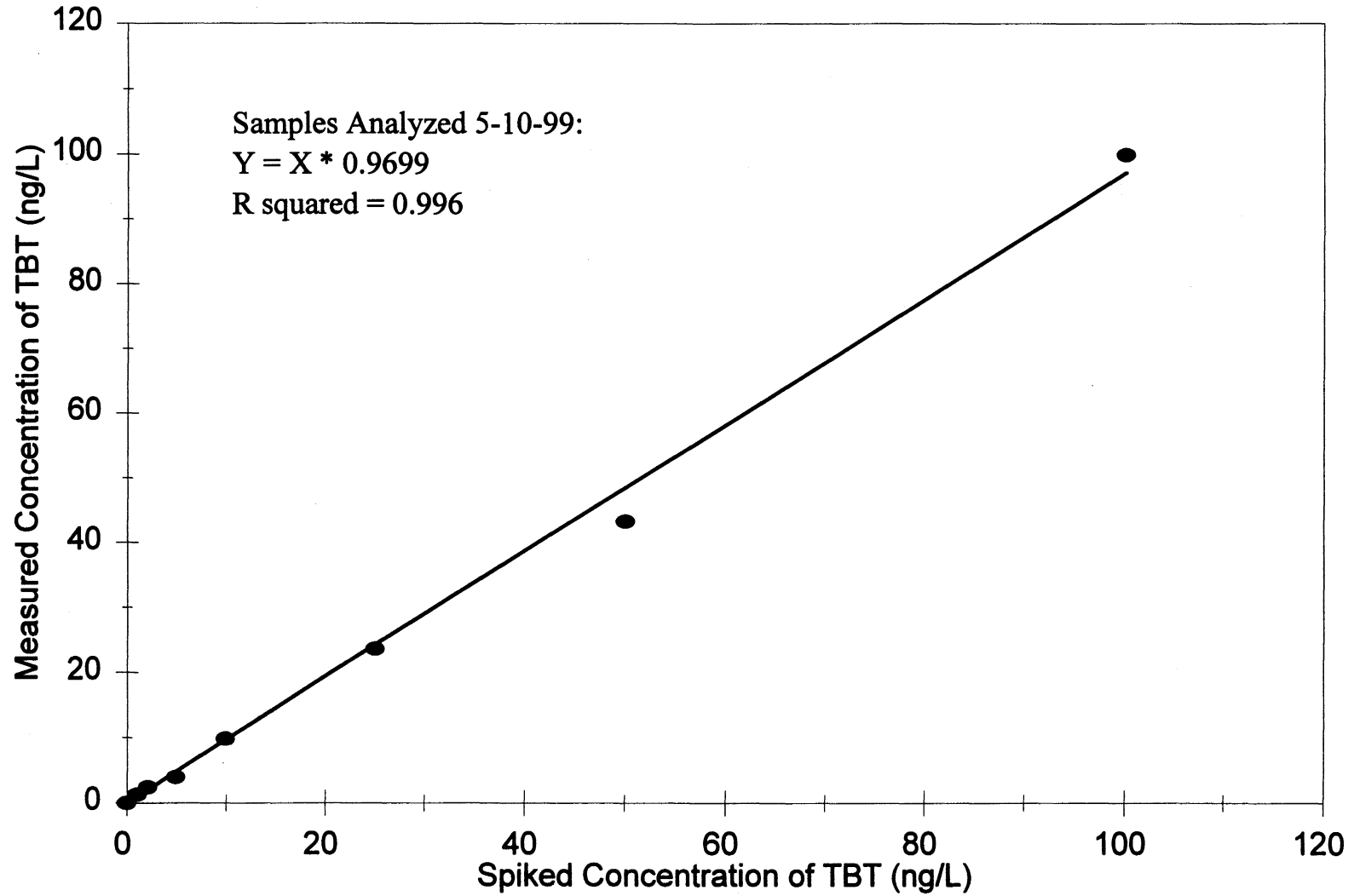
Samples Analyzed 5-10-99:
Y = X * 0.9699
R squared = 0.996

Measured Concentration of TBT (ng/L)

Spiked Concentration of TBT (ng/L)

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Butyltin analysis by GENIE   Page 1

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 $DL = 0.4 \, ng/l$

                SpecialInteg

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]
2	12.13	#12.13	1.0000	25169	FF		HePe3Sn Internal Std	MBT[3]

Total Area   :   48816   Total ng/l   :   45.815

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

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

Spiked Water Blank 05-03-99

Tetrabutyltin

HePe3Sn ISTD

Response

5.70

5.50

5.30

5.10

4.00   6.00   8.00   10.00   12.00   14.00

Retention Time (minutes)

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Butyltin analysis by GENIE   Page   1

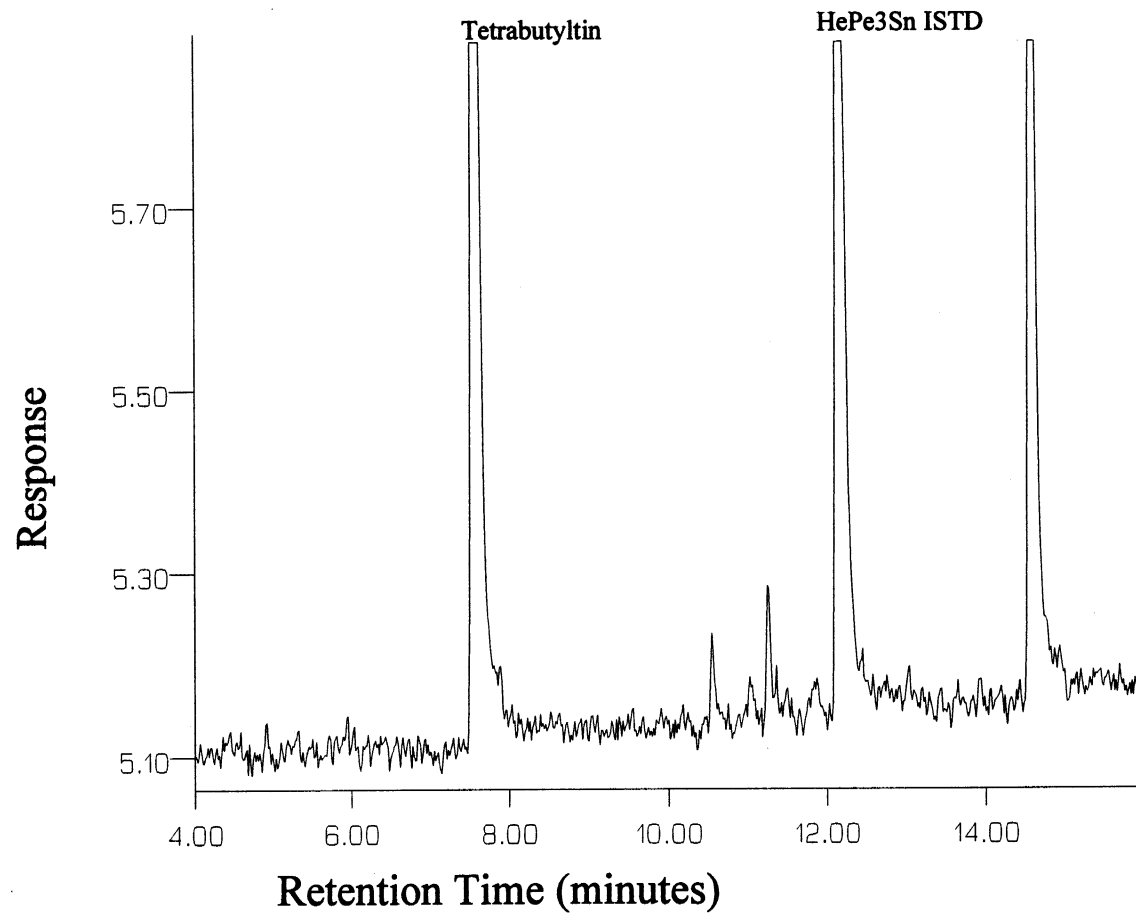Sample Name   : SN27-32A DEQ 1 NG/L A   05-03-99   Report No : 31.02
EXTRACTED ON 5-03-99

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

$DL = 0.5 ng/l$

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+ 1.3 ng/l TBT+
3	11.32	#11.35	.0196	203	FF	.38	DBT++ 0.4 ng/l DBT+2
4	12.10	#12.13	1.0000	17700	FF		MePe3Sn internal std
5	12.99	#13.03	.0148	380	FF	.53	MBT+++ 0.5 ng/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/SMBUTIN.FMT

TBT Spiked Water A: 1 ng/L
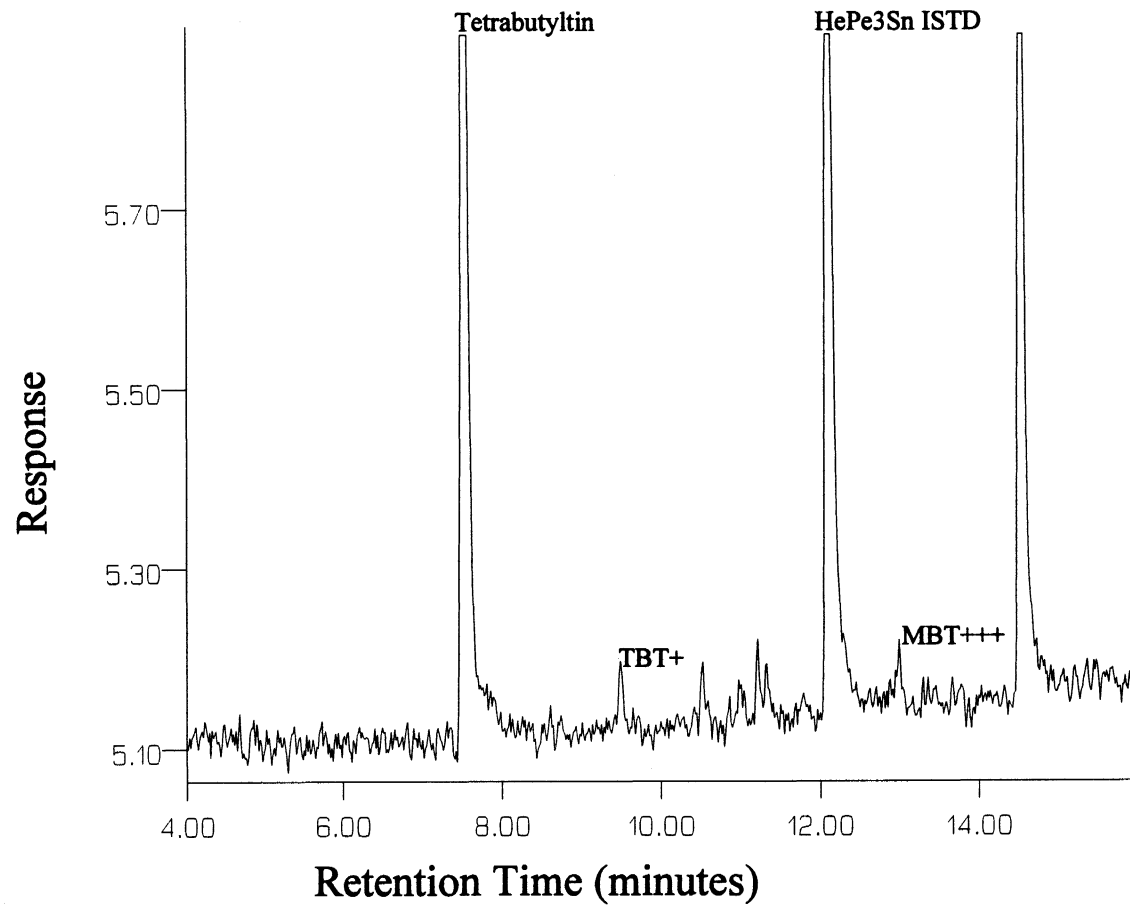
3350 LOOP REPORT - BUTYLTINS

User Modified
Butyltin analysis by GENIE   Page 1

Sample Name   : SN27-328 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+ 1.3 ng/L TBT+
3	11.34	#11.35	.0196	479	FF	.85	DBT++ 0.8 ng/L DBT+2
4	12.12	#12.13	1.0000	18360	FF		HePe3Sn internal std
5	13.02	#13.03	.0149	347	FF	.47	MBT+++ NO MBT*3

Total Area   :   36350   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
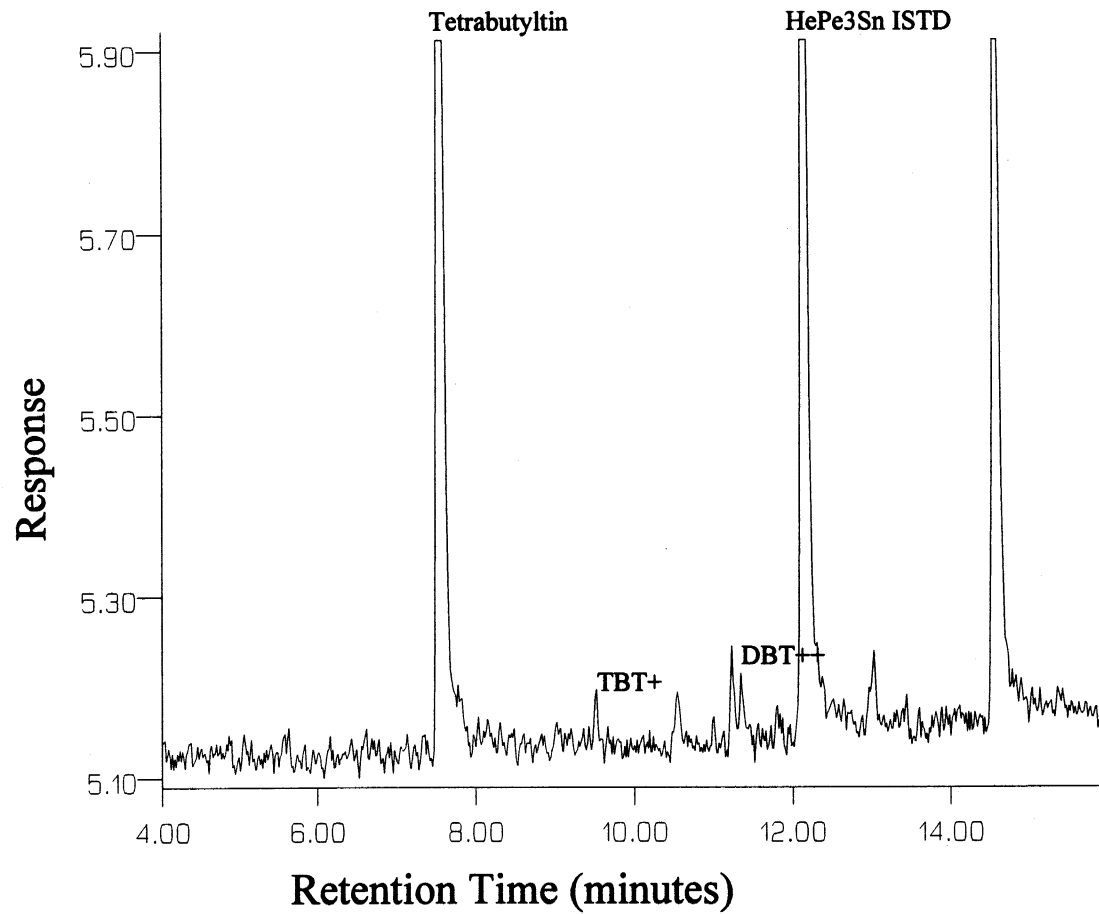
UserModified
Butyltin analysis by GENIE   Page   1

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 $OL = 0.5 ng/l$

| 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^{+1}
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		HePe3Sn 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
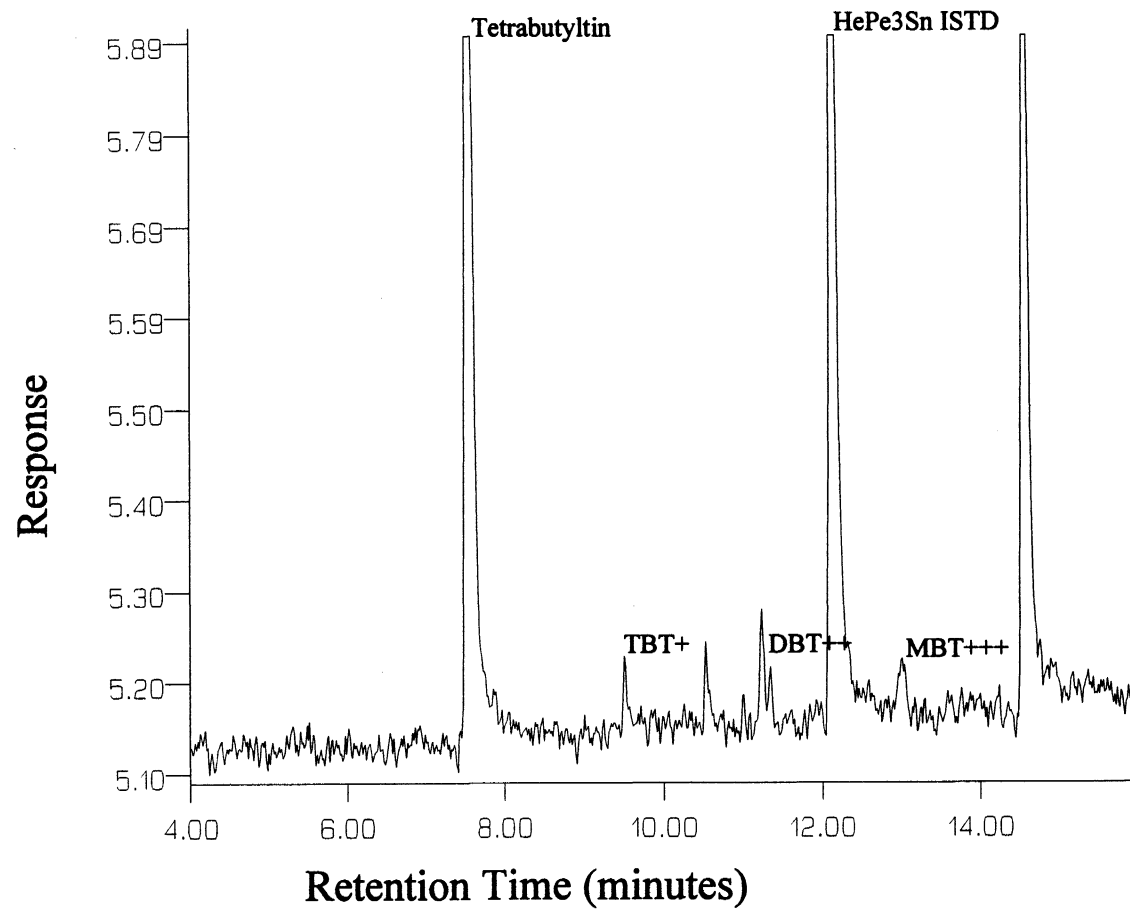
UserModified
Butyltin analysis by GENIE   Page 1

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   DL = 0.5 ng/l

               SpecialInteg

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+ 0.6 ng/l TBT+ +2
3	11.37	#11.35	.0196	537	FF	.85	DBT++ 0.8 ng/l DBT+
4	12.13	#12.13	1.0000	20808	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	288	FF	.34	MBT+++ ND MBT +3

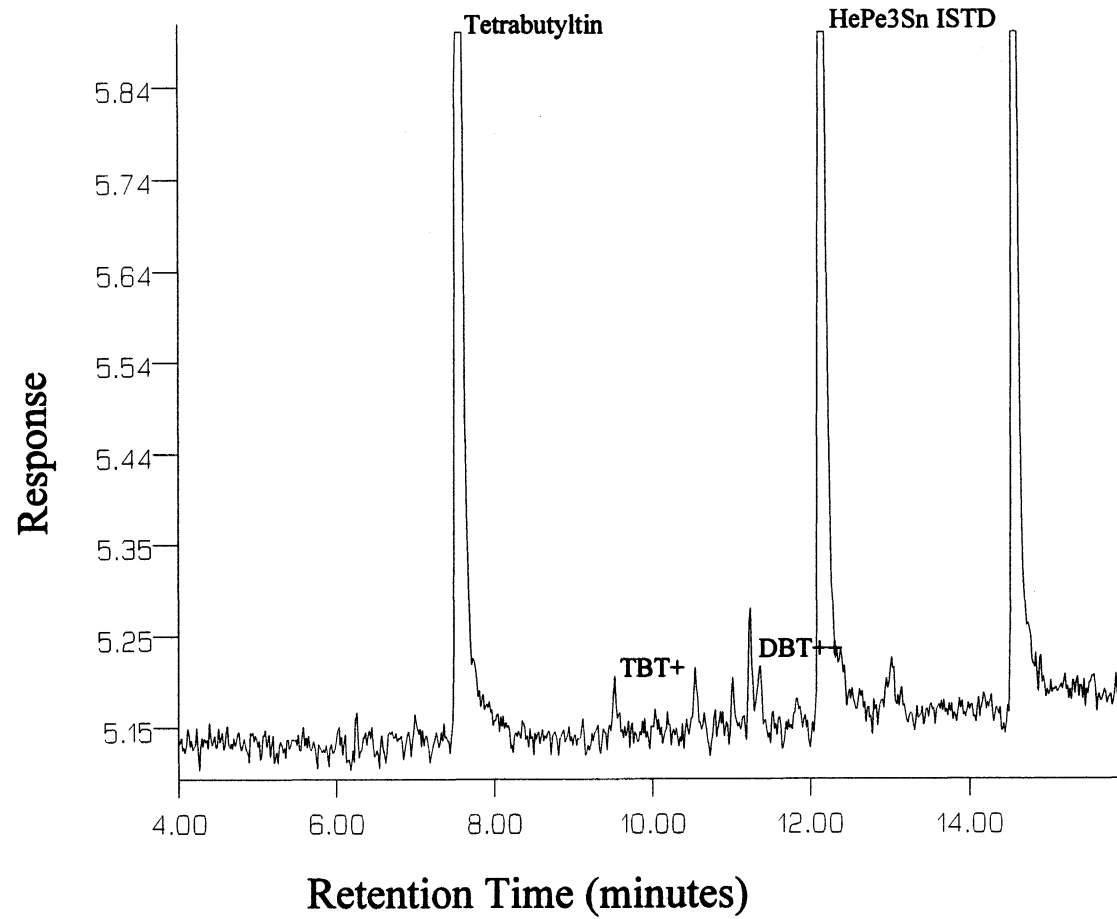Total Area   :   40140   Total ng/l   :   44.449

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

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Butyltin analysis by GENIE   Page   1

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 - 0.4 ng/l

| K#	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	513	FF	.93	TBT+ 0.8 ng/l TBT+
3	11.35	#11.35	.0196	540	FF	.71	DBT++ 0.7 ng/l DBT+2
4	12.13	#12.13	1.0000	25124	FF		HePe3Sn internal std
5	13.02	#13.03	.0149	480	FF	.47	MBT+++ 0.5 ng/l MBT+3

Total Area   :   51271   Total ng/l   :   49.769

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

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

TBT Spiked Water E: 1 ng/L
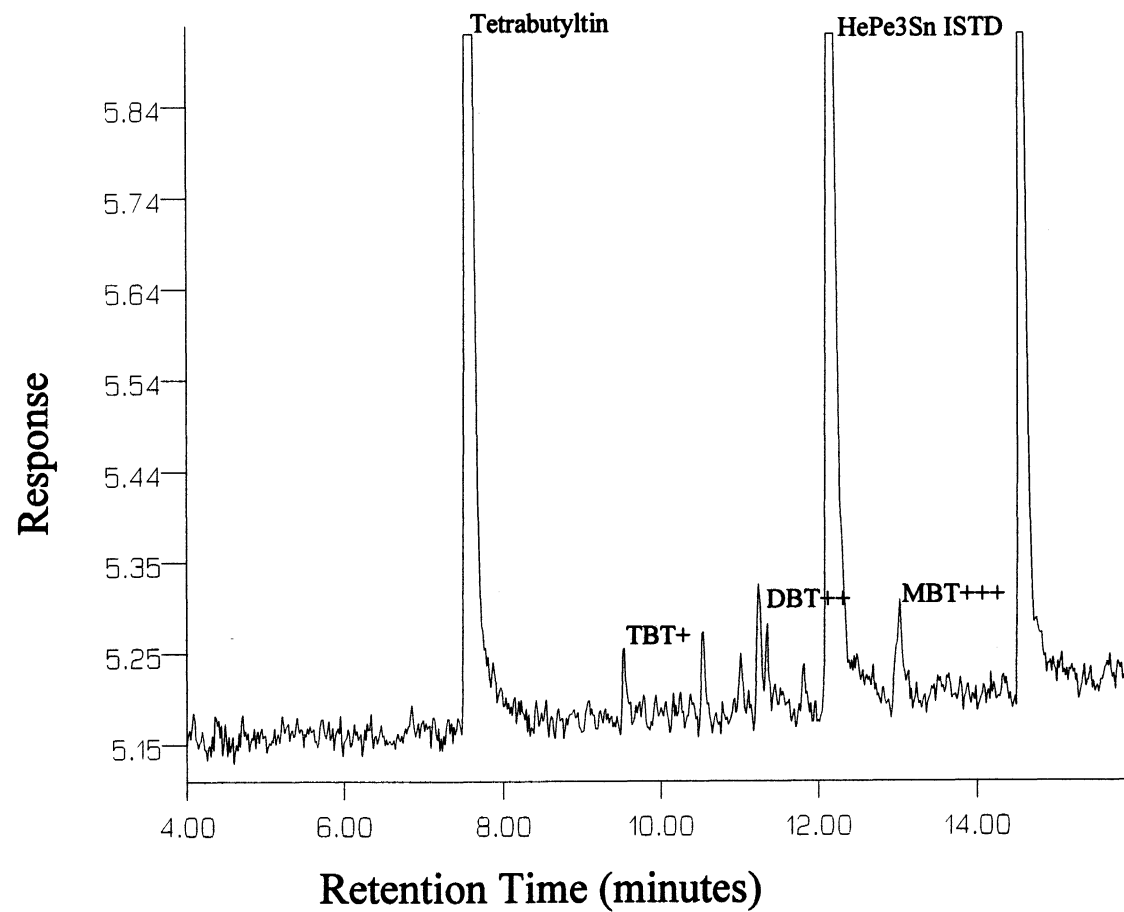
UserModified
Butyltin analysis by GENIE   Page 1

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
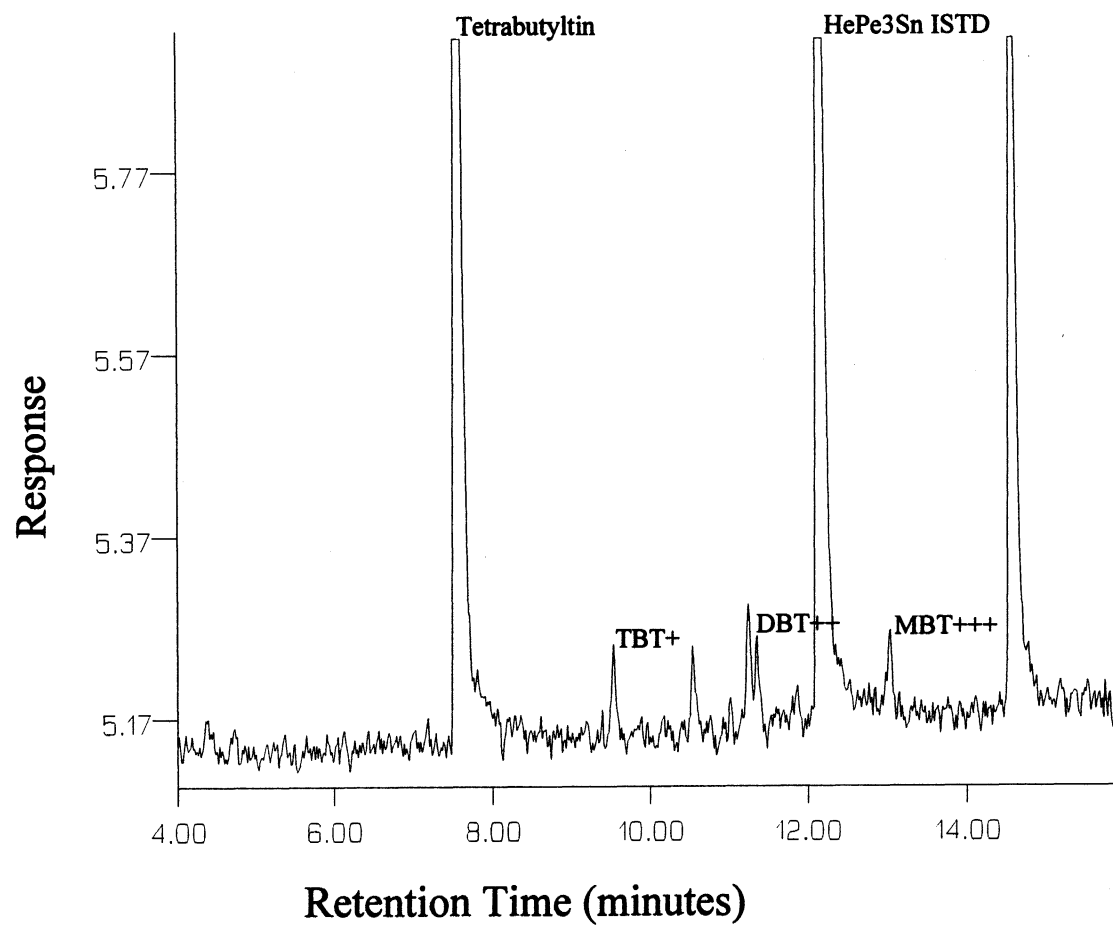
UserModified
Butyltin analysis by GENIE   Page   1

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-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	21182	FF	43.22	TETRABUTYLTIN
2	9.52	#9.52	.0244	554	FF	.94	TBT+ 0.9 ng/l TBT'
3	11.35	#11.35	.0196	800	FF	1.19	DBT++ 1.1 ng/l DBT''
4	12.13	#12.13	1.0000	23900	FF		HePe3Sn internal std
5	13.03	#13.03	.0148	842	FF	.87	MBT+++ 0.9 ng/l MBT''

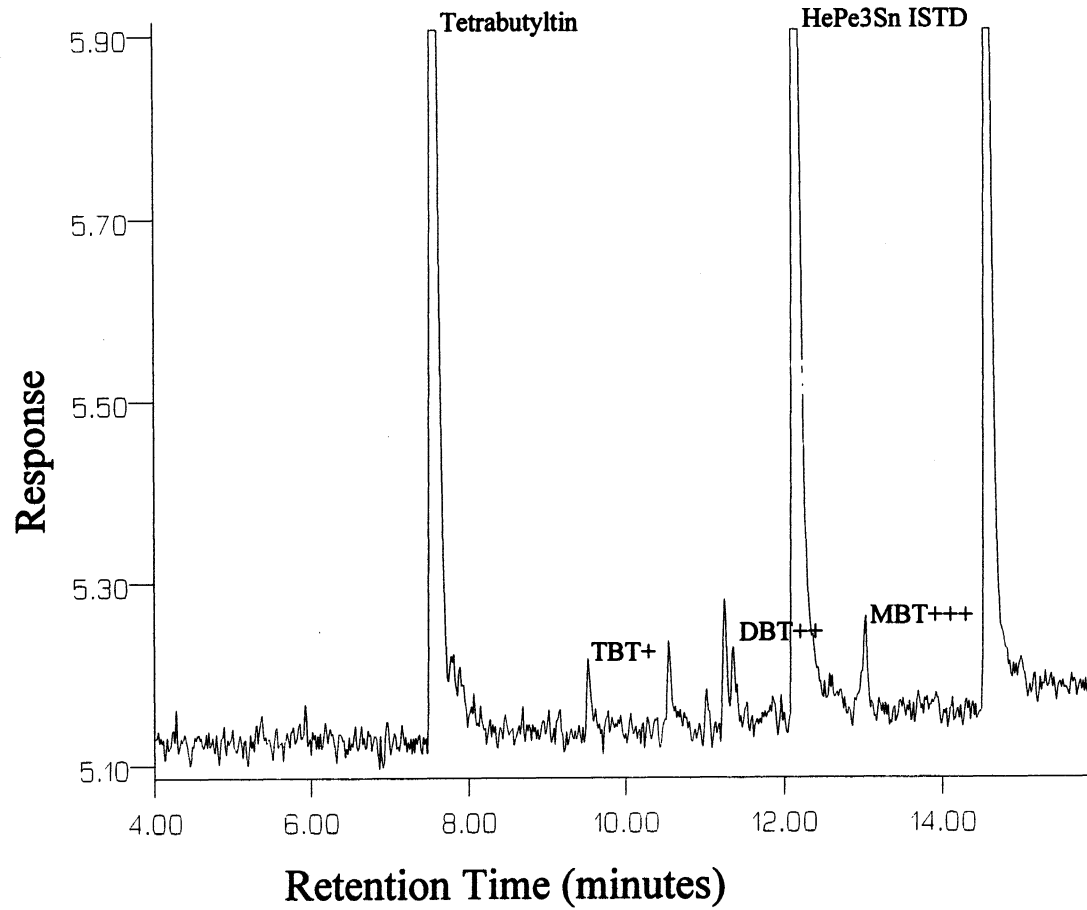Total Area   :   47279   Total ng/l   :   46.129

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

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Butyltin analysis by GENIE   Page   1

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

G 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

K#   RT   ID-tm   Res Factor   Area   Code   ng/l   Name
 1   7.53   #7.53   .0292   16963   FF   39.41   TETRABUTYLTIN   NO TBT & DBT12
 2   12.13   #12.13   1.0000   20986   FF   HePe3Sn internal std
                                                                               MBT+3
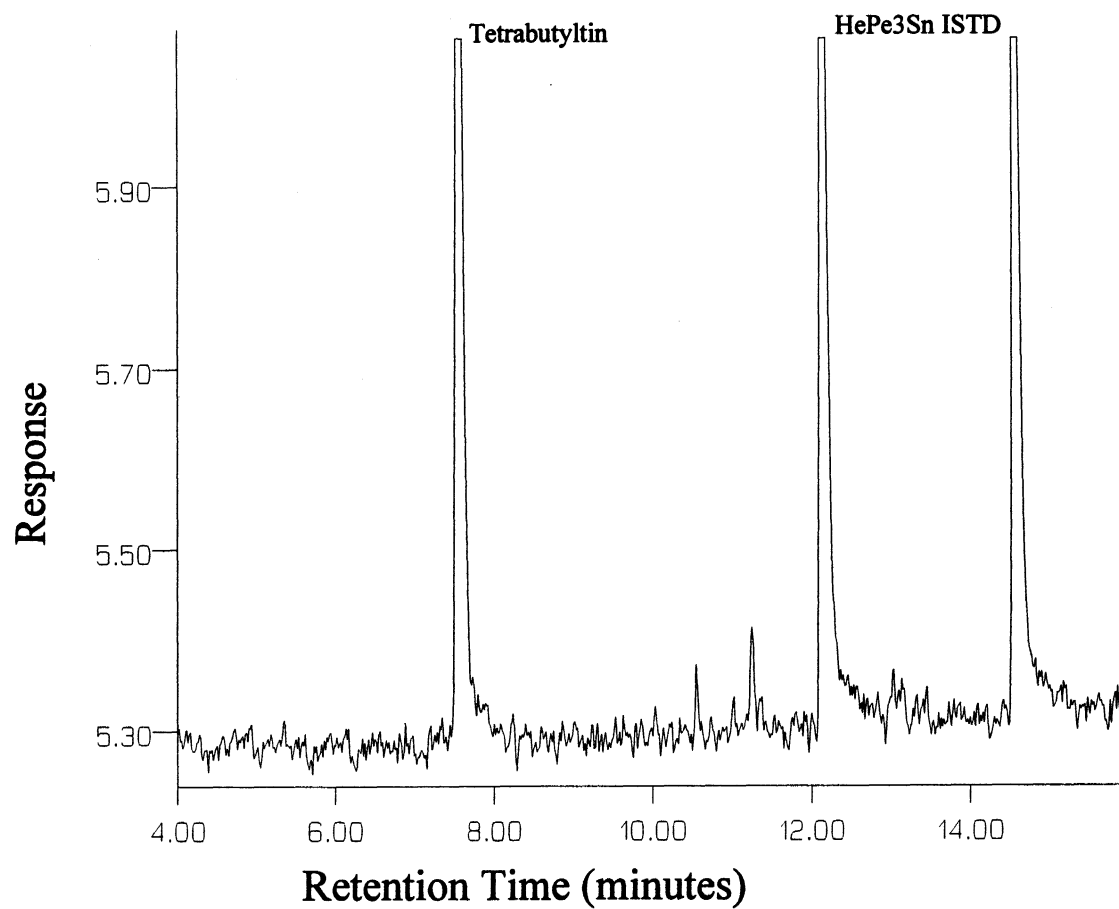
Total Area   :   37949   Total ng/l   :   39.415

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

Tetrabutyltin

HePe3Sn ISTD

Response

Retention Time (minutes)

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Butyltin analysis by GENIE   Page 1

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

| K#	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+
4	12.13	#12.13	1.0000	19182	FF		HePe3Sn internal std
5	13.02	#13.03	.0148	287	FF	.37	MBT+ NO MBT+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
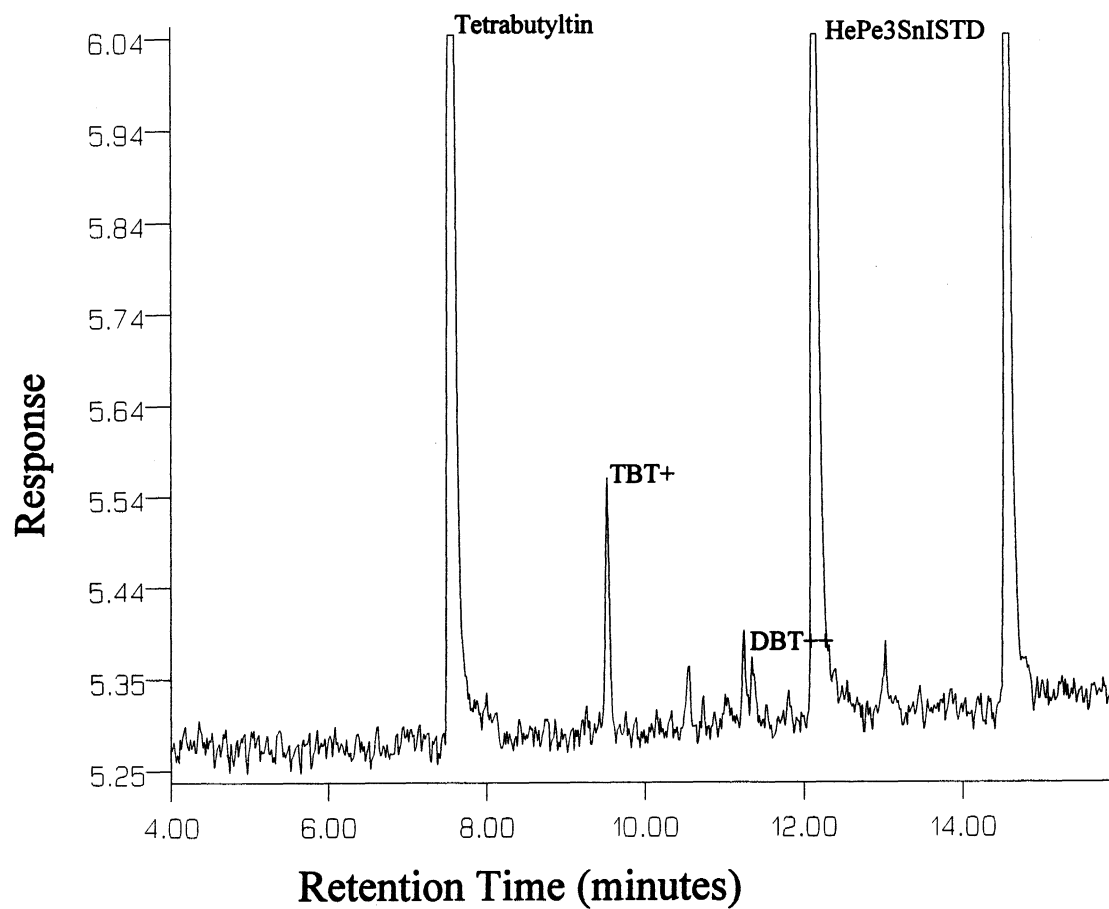
```
              3350 LOOP REPORT - BUTYLTINS
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Butyltin analysis by GENIE                                    Page   1

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
```
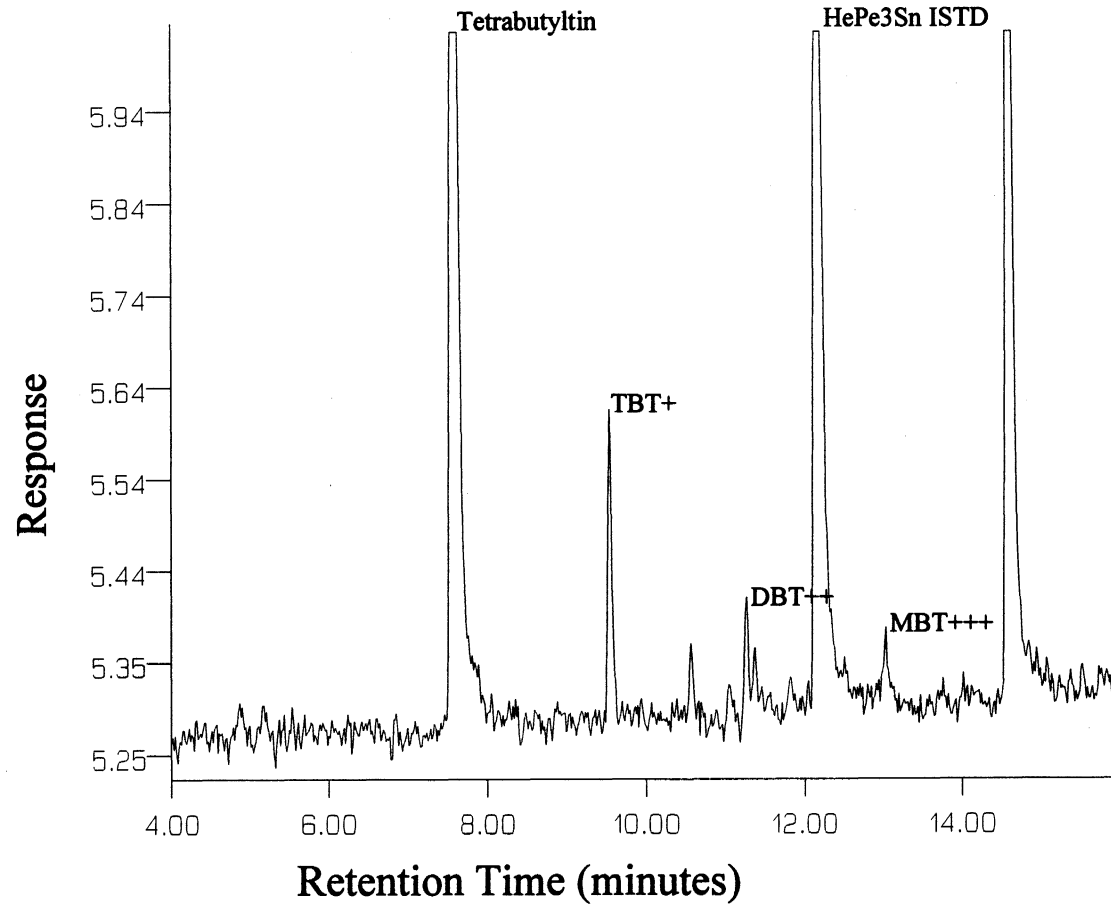
TBT Spiked Water B: 5 ng/L

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Butyltin analysis by GENIE   Page   1

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 $DL = 0.5 ng/l$

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+ $3.9 ng/l \, TBT^+$
3	12.12	#12.13	1.0000	18620	FF		HePe3Sn internal std $NO DBT^{+2}$
4	13.02	#13.03	.0148	602	FF	.80	MBT+++ $0.8 ng/l \, MBT+3$

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
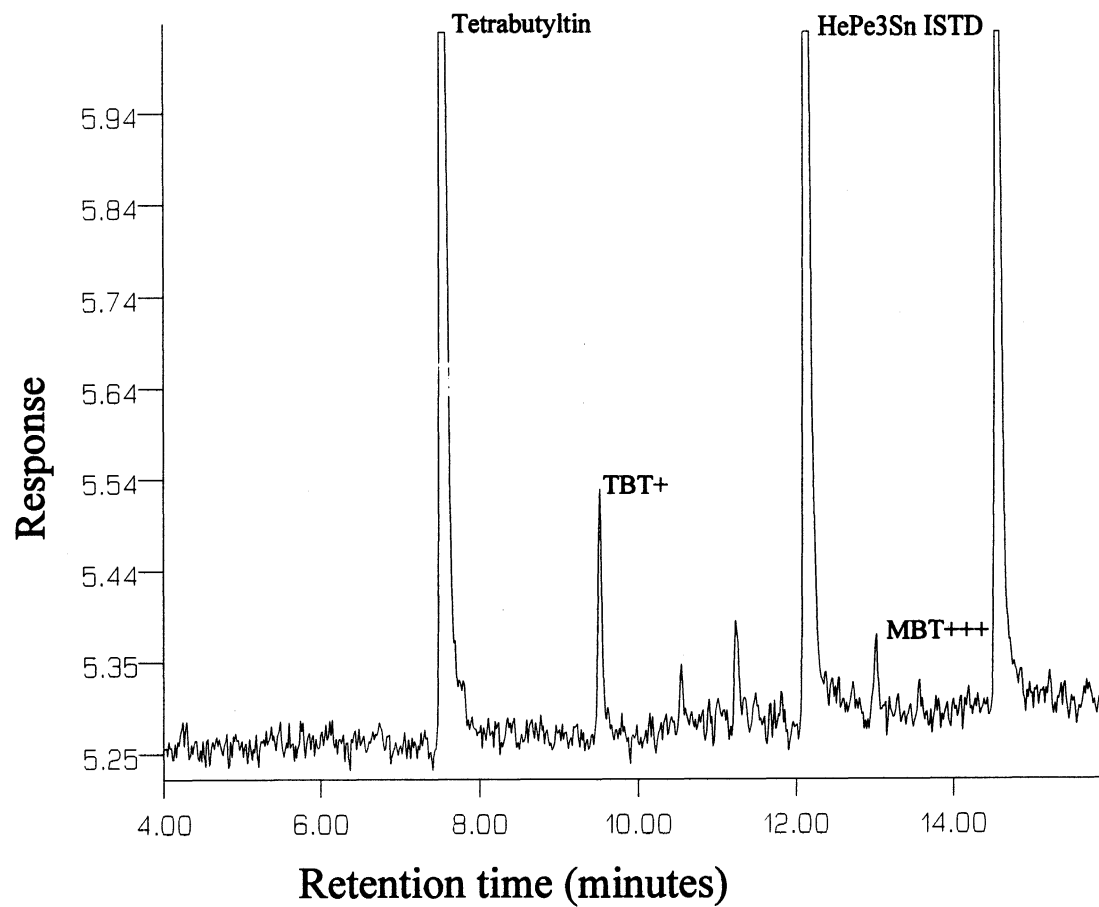
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Butyltin analysis by GENIE   Page   1

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   DL= 0.5 ng/l

                SpecialInteg

#	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
3	12.12	#12.13	1.0000	17423	FF		HePe3Sn internal std
4	13.02	#13.03	.0148	713	FF	.44	MBT+++ B, NO DBT+², MBT+³

Total Area   :   34817   Total ng/l   :   47.508
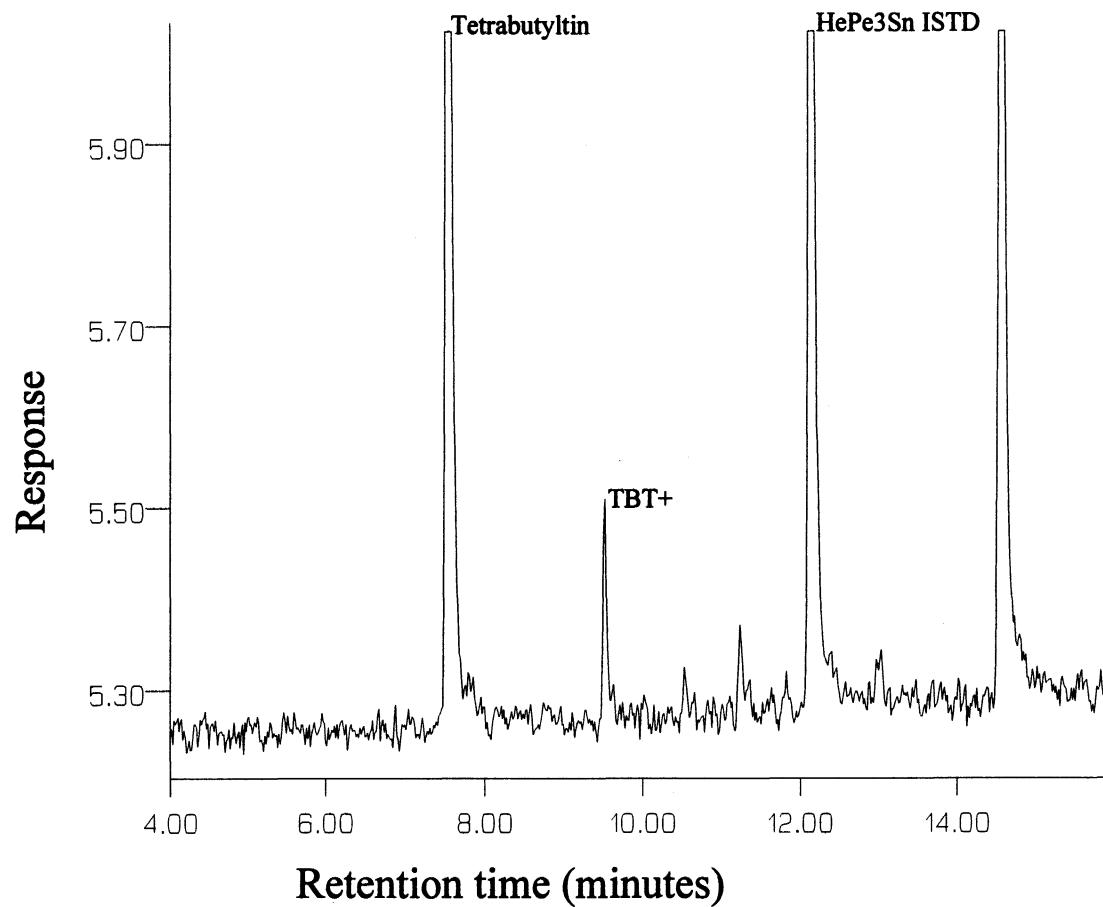
Report Time   : Fri Apr 30, 1999 3:57:06 pm

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

TBT Spiked Water D: 5 ng/L

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Butyltin analysis by GENIE   Page   1

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 $DL = 0.5 ng/l$

| PK	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+ 4.8 ng/l TBT+
3	11.35	#11.35	.0196	366	FF	.68	DBT++ 0.7 ng/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+++ 1.0 ng/l MBT+3

Total Area   :   36051   Total ng/l   :   48.997

Report Time : Fri Apr 30, 1999 3:59:43 pm

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

TBT Spiked Water E: 5 ng/L
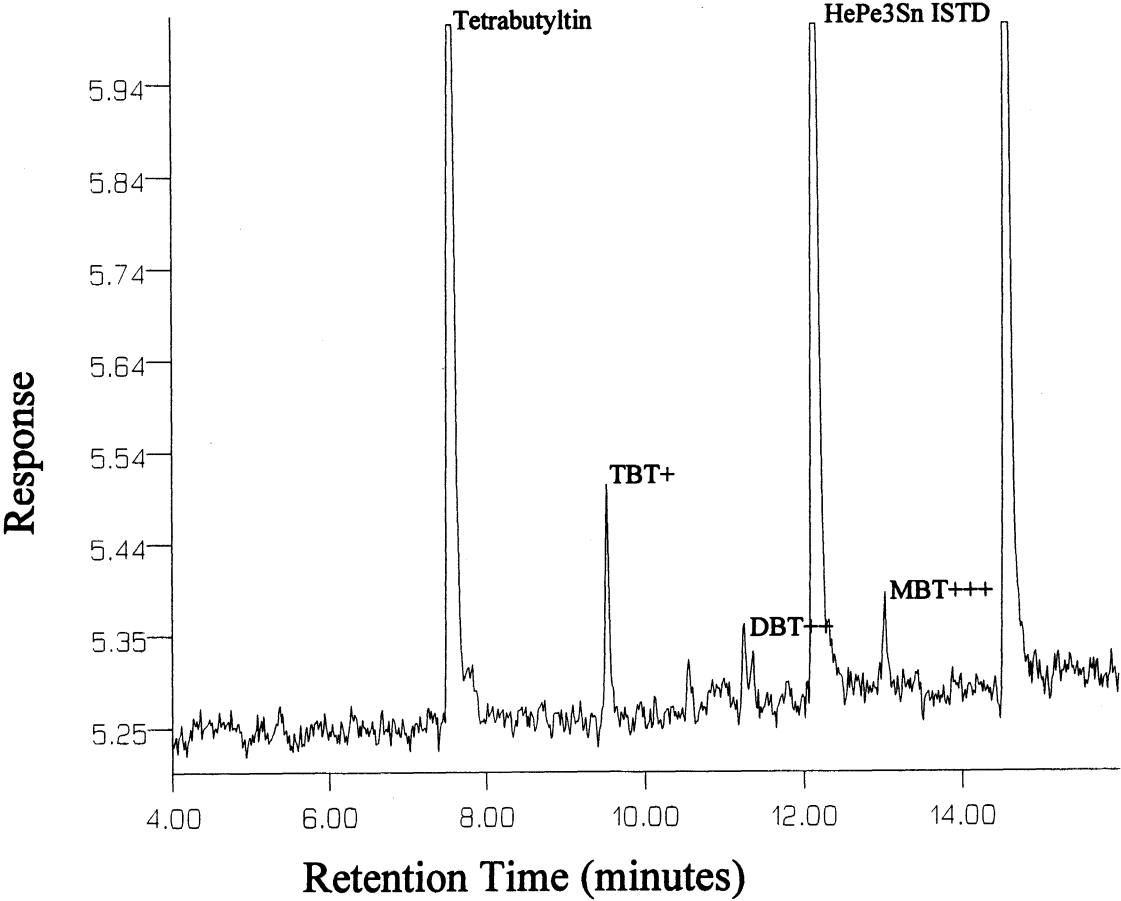
3350 LOOP REPORT – BUTYLTINS

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Butyltin analysis by GENIE   Page   1

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 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= 0.4 ng/l

#	RT	ID-tm	Rea 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	24269	FF		HePe3Sn internal std

(handwritten, right margin): ND TBT+, 0.5 ng DBT+2, NO MBT+3

Total Area   :   50230   Total ng/l :   51.955
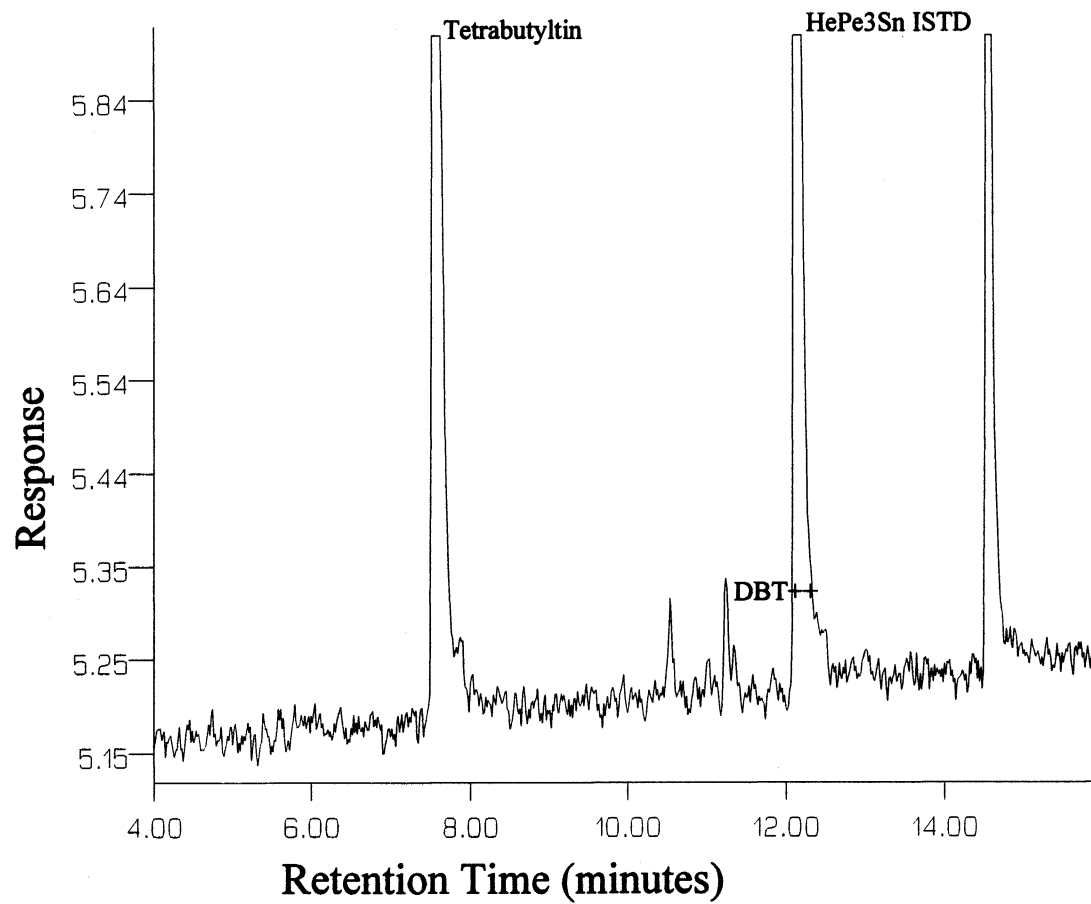
Report Time : Wed May 12, 1999 10:28:44 am

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

TBT Spiked Water Blank 05-10-99

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Butyltin analysis by GENIE   Page   1

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/GRG955L.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   DL= 0.4ng/l

                SpecialInteg

| #	RT	ID-tm	Res Factor	Area	Code	ng/l	Name
1	7.52	#7.53	.0292	20396	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
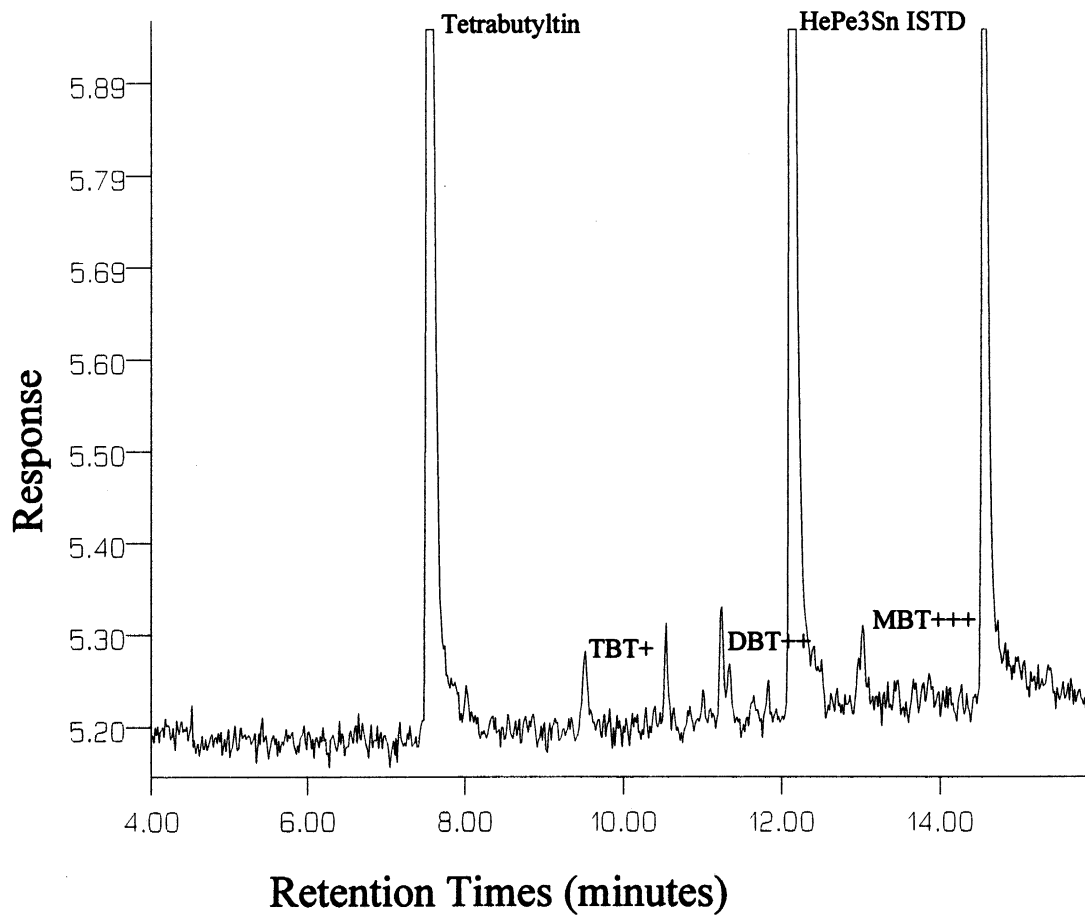
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              3350 LOOP REPORT - BUTYLTINS
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Sample Name  : SN27-34B DEQ 2 NG/L          05-10-99    Report No :  4.02
EXTRACTED ON 5-10-99

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                         DL = 0.5 ng/l

                SpecialInteg


 #     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/TBT+
 3   11.35   #11.35      .0196           344    FF       .64   DBT++  0.6 ng/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/MBT r3

Total Area    :         37039       Total ng/l  :      51.673

Report Time   : Wed May 12, 1999 10:75:04 am

Method        : /METHOD/TIN3/MTIN3G.MTH
Report File   : /FORMAT/TIN3/CMBUTIN.FMT
```
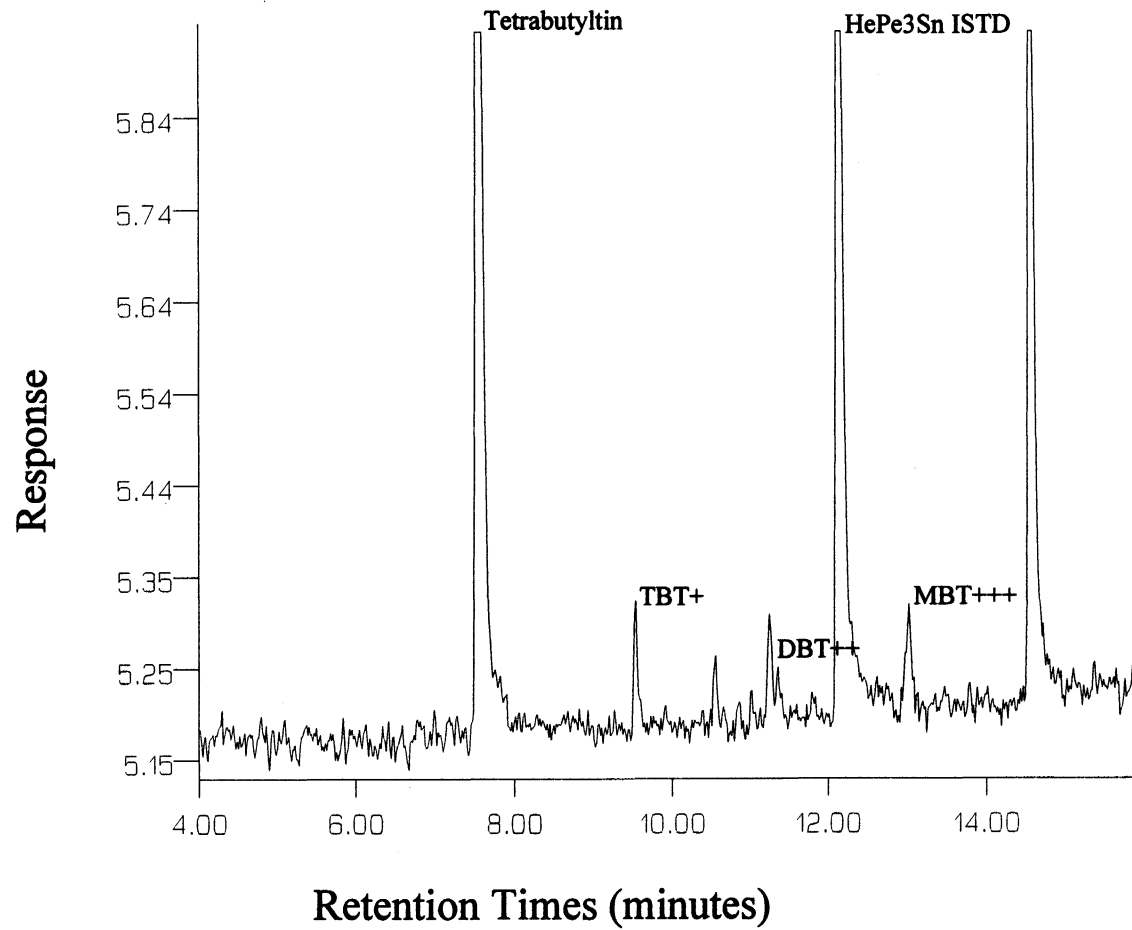
TBT Spiked Water: 2 ng/L

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            3350 LOOP REPORT - BUTYLTINS
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Butyltin analysis by GENIE                                  Page   1

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/SRG952L.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
                                            DL= 0.6 ng/l
               SpecialInteg


#    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       3.99    TBT+  4.0 ng/l TBT+
3   12.12    #12.13   1.0000       16045    FF               HePe3Sn internal std
                                                             NO 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
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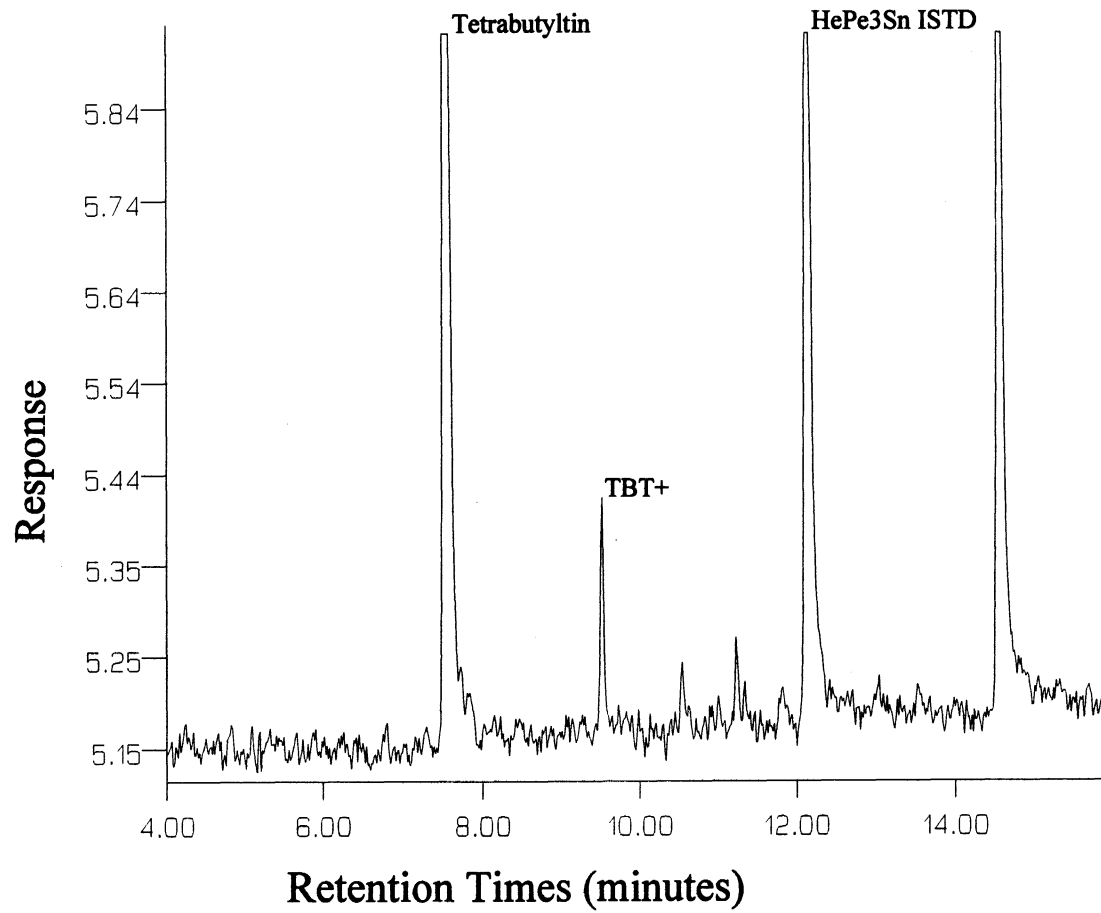
TBT Spiked Water: 5 ng/L

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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		HePe3Sn internal std

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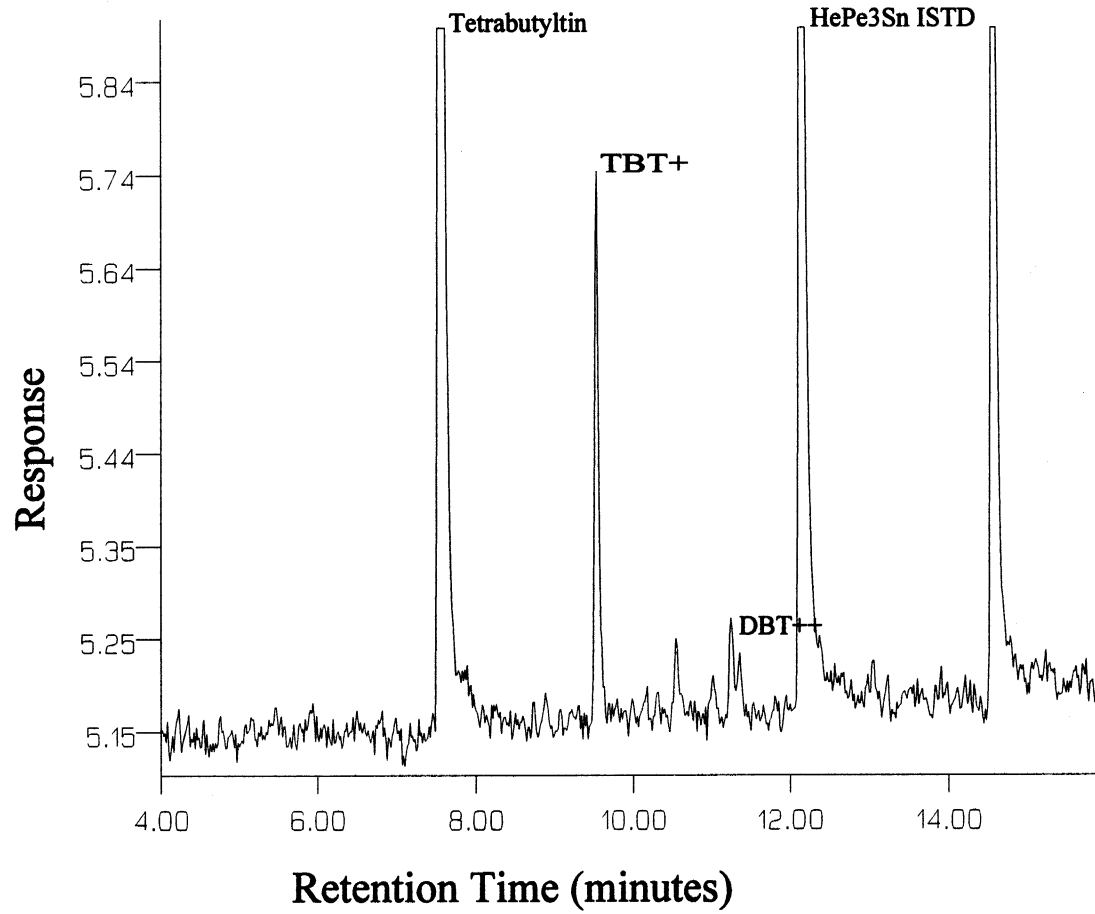
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Sample Name : SN27-35A DEQ 25 NG/L   05-10-99   Report No : 7.02
EXTRACTED ON 5-10-99

Peak Processor : Genie   Multilevel   : False

Instrument   : A/D_3   Calculation : InternalSTD

Result File   : /RESULT/TIN3/SRG959L.REС

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 $DL= 0.5 ng/l$

N	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	23.65	TBT+ *23.7 ng/l TBT+*
3	11.37	#11.35	.0196	700	FF	.56	DBT++ *0.6 ng/l DBT+2*
4	12.14	#12.13	1.0000	17536	FF		MeMe3Sn internal std

NO MBT+3

Total Area   :   45547   Total ng/l   :   72.965
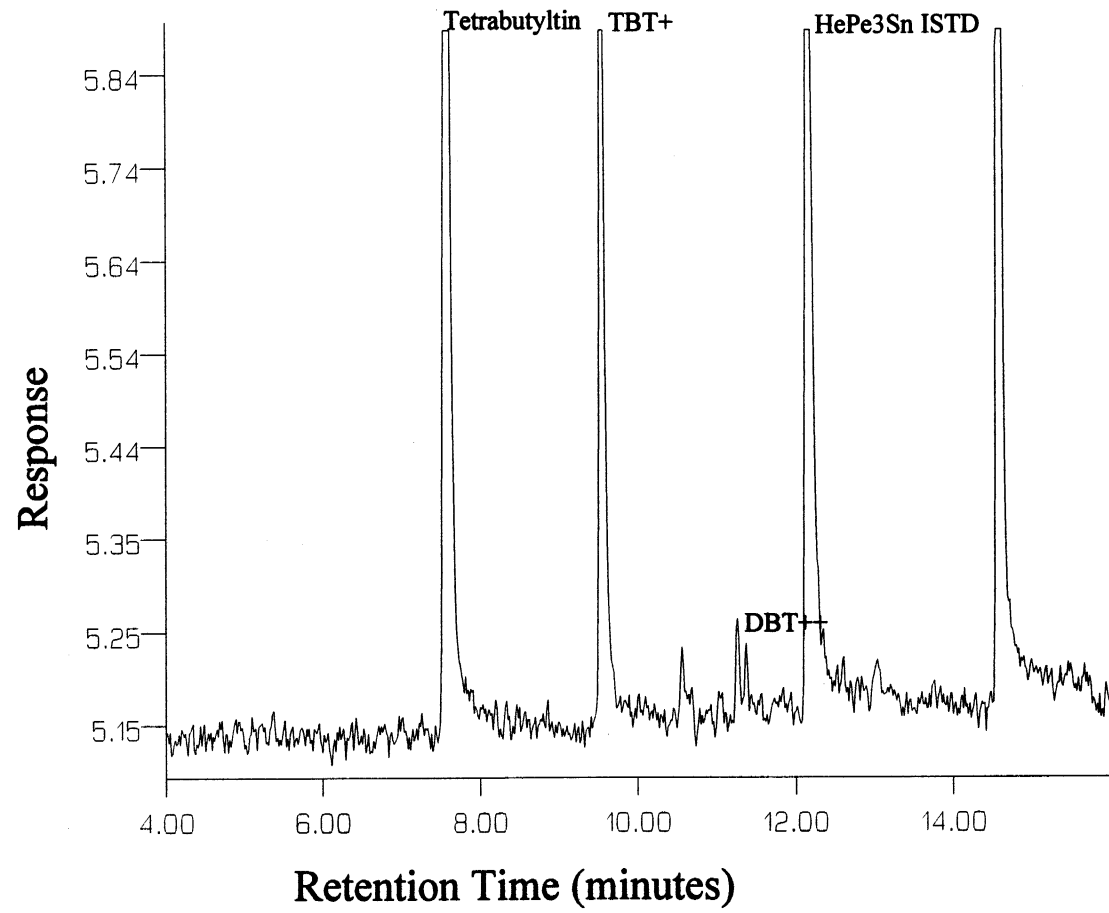
Report Time   : Wed May 12, 1999 10:42:07 am

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

TBT Spiked Water: 25 ng/L

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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/SR6960L.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
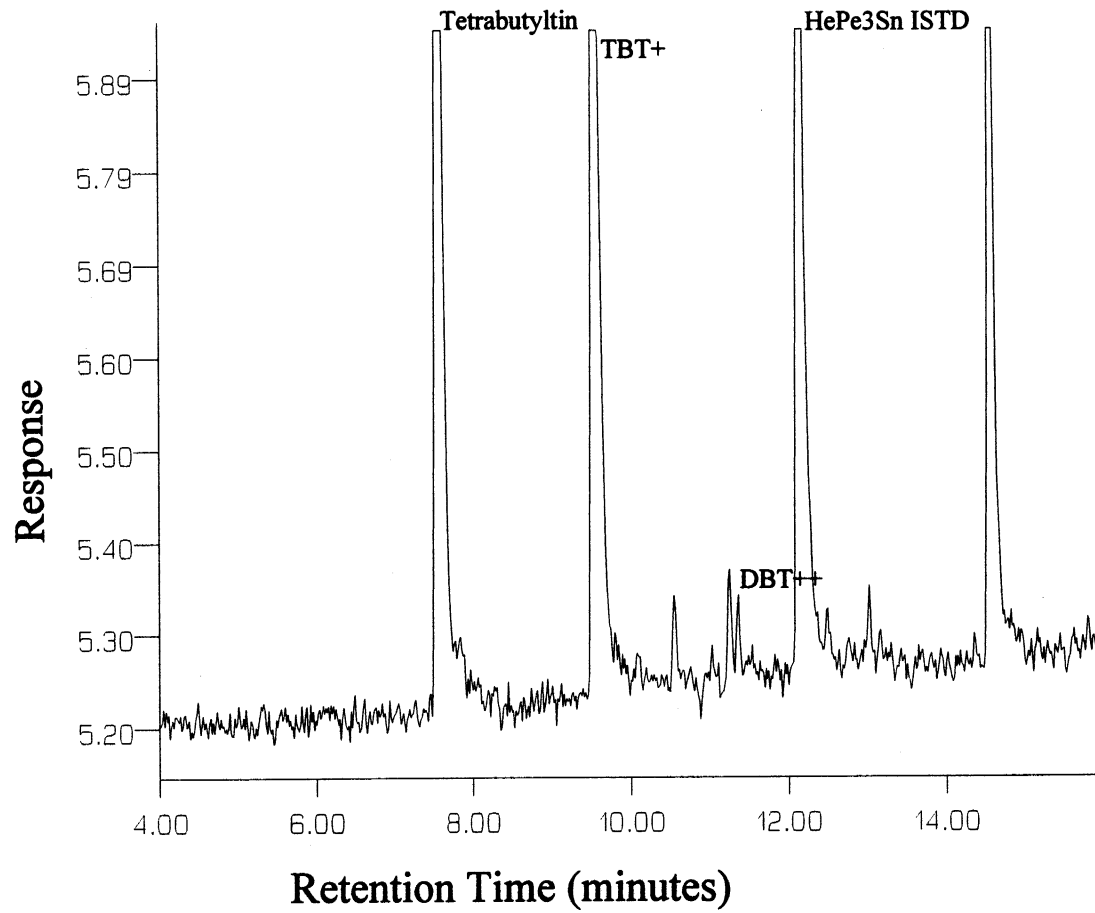
3350 LOOP REPORT - BUTYLTINS

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Butyltin analysis by GENIE   Page   1

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   DL= 0.8 ng/l

               SpecialInteg

k#   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