

8 Referências Bibliográficas

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Glossário de Abreviaturas

Compostos orgânicos de estanho

BTs – compostos butílicos de estanho

DBT – dibutilestanho

MBT – monobutilestanho

OTs - compostos orgânicos de estanho

TeBT – tetrabutilestanho

TPrT – tripopilestanho

TBT – tributilestanho

Reagentes

AgNO₃ – nitrato de prata

Al₂O₃ – óxido de alumínio, alumina

APDC – amônio pirrolidino ditiocarbamato

HCl – ácido clorídrico

Na₂SO₄ – sulfato de sódio

PeMgBr – brometo de pentil magnésio

Técnicas

GC – cromatografia gasosa

MSD – detecção por espectrometria de massas

PFPD – detector fotométrico de chama pulsante

SIM – monitoramento de íon selecionado

Outras abreviações

BTS – Baía de Todos os Santos

CRA – Centro de Recursos Ambientais – Baía de Todos os Santos

CRM – material de referência certificado

Eh – potencial redox

GPS – Global Positioning System

LD – Limite de detecção

NRCC – National Research Council of Canada

Apêndice

As concentrações dos diversos compostos organoestânicos nas curvas de calibração estão mostradas na tabela no abaixo.

Tabela A.1. Concentrações dos padrões mixtos organoestânicos nas curvas de calibração dos diversos compostos.

Sn ng mL ⁻¹	TPrT	TBT	DBT	MBT
10	201,53	150,24	150,14	151,00
2	161,22	120,19	120,12	120,81
8	120,92	90,14	90,09	90,60
7	80,61	60,09	60,06	60,40
6	64,49	48,08	48,05	48,32
5	56,43	42,07	42,04	42,28
4	48,37	36,06	36,04	36,24
3	40,31	30,05	30,03	30,20
2	32,24	24,04	24,02	24,16
1	24,18	18,03	18,02	18,12

Na tabela abaixo são mostrados os rendimentos do sub-rogado TPrT que foi introduzido nas amostras em duplicata.

Tabela A.2. Rendimento do sub-rogado.

Amostras em duplicata	$[\text{TPrT}_{\text{obtido}}/\text{TPrT}_{\text{original}}]\times 100$
Estação 1 001	70,35
Estação 1 002	75,60
Estação 2 001	122,55
Estação 2 002	116,59
Estação 3 001	116,86
Estação 3 002	141,91
Estação 4 001	167,53
Estação 4 002	119,51
Estação 5 001	76,07
Estação 5 002	84,78
Estação 6 001	126,31
Estação 6 002	127,19
Estação 7 001	87,61
Estação 7 002	103,65
Estação 8 001	99,59
Estação 8 002	84,96
Estação 9 001	75,13
Estação 9 002	70,34
Estação 10 001	93,80
Estação 10 002	104,04
Estação 11 001	148,57
Estação 11 002	132,34
Estação 12 001	98,71
Estação 12 002	109,63
Estação 13 001	103,47
Estação 13 002	80,60
Estação 14 001	105,32
Estação 14 002	114,28
Estação 15 001	102,98
Estação 15 002	105,01
Estação 16 001	83,86
Estação 16 002	92,88
Estação 17 001	89,58
Estação 17 002	104,00

Nas figuras abaixo são mostradas as curvas de calibração com as respectivas faixas lineares utilizadas para quantificação da batelada de amostras.

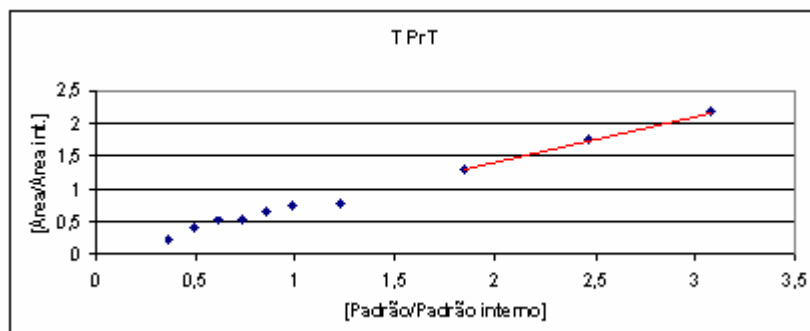


Figura A.1. TPrT (ng mL⁻¹ como Sn)

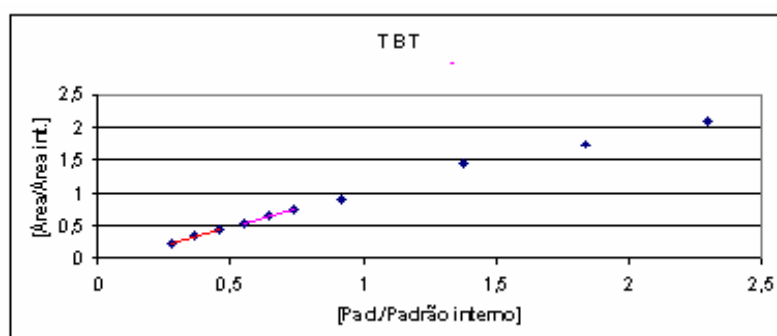


Figura A.2. TBT (ng mL⁻¹ como Sn)

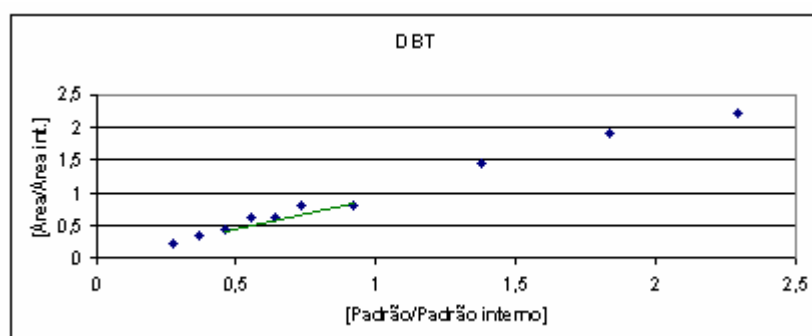


Figura A.3. DBT (ng mL⁻¹ como Sn)

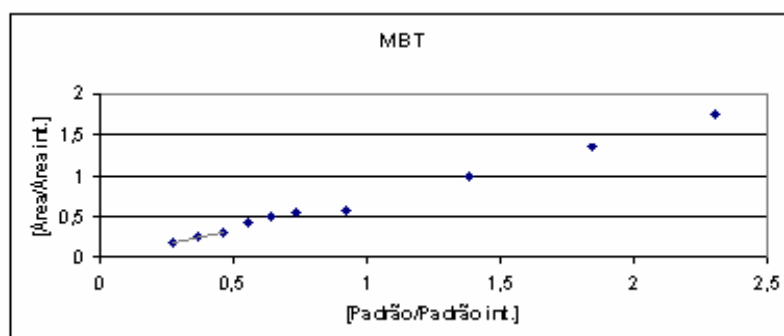


Figura A.4. MBT (ng mL^{-1} como Sn)

Cromatogramas das Estações de Amostragem.

Os cromatogramas estão em escalas diferentes para melhor visualizar os picos de cada estação.

Ponto 2 – Itapagipe/ fora

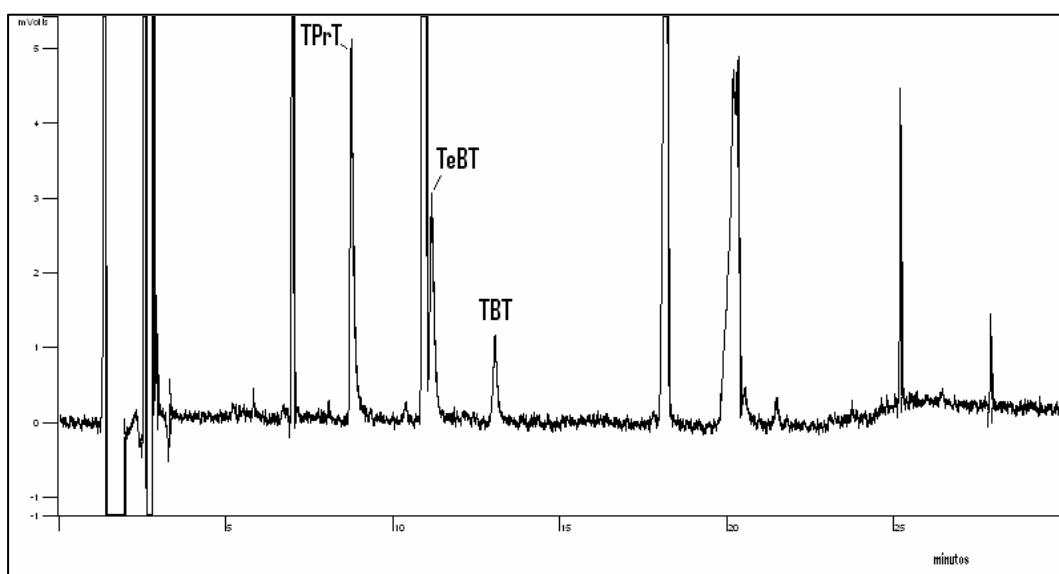


Figura A.5. Cromatograma do ponto 2.

Ponto 3 – Tainheiros

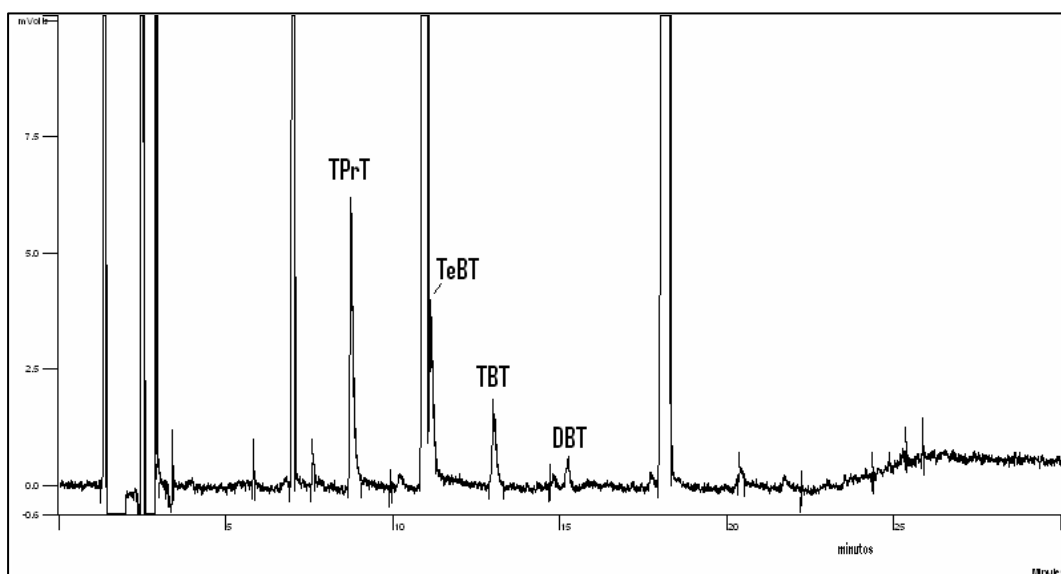


Figura A.6. Cromatograma do ponto 3.

Ponto 4 – Paripe

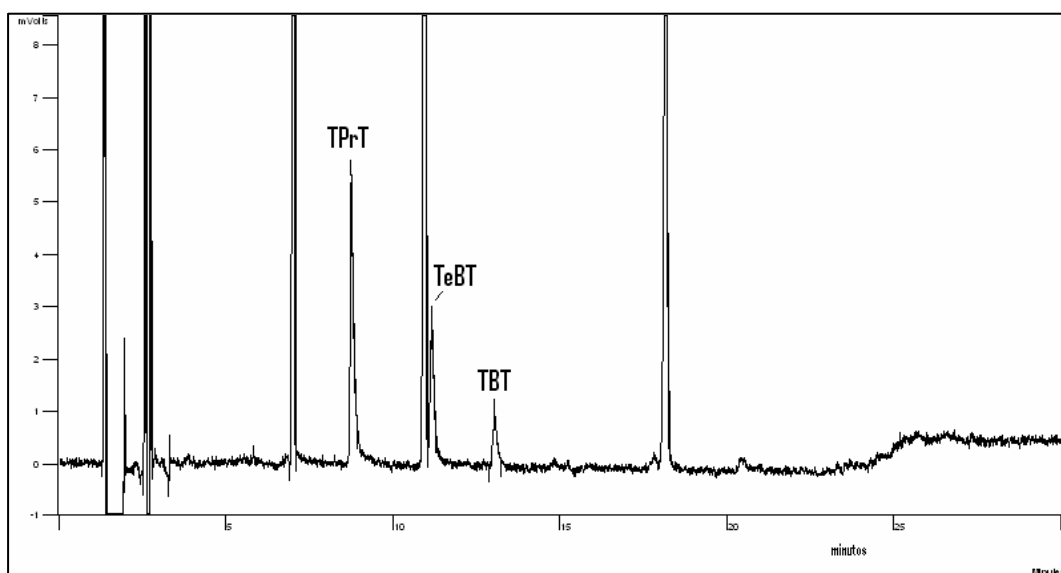


Figura A.7. Cromatograma do ponto 4.

Ponto 5 – Baía de Aratu sul

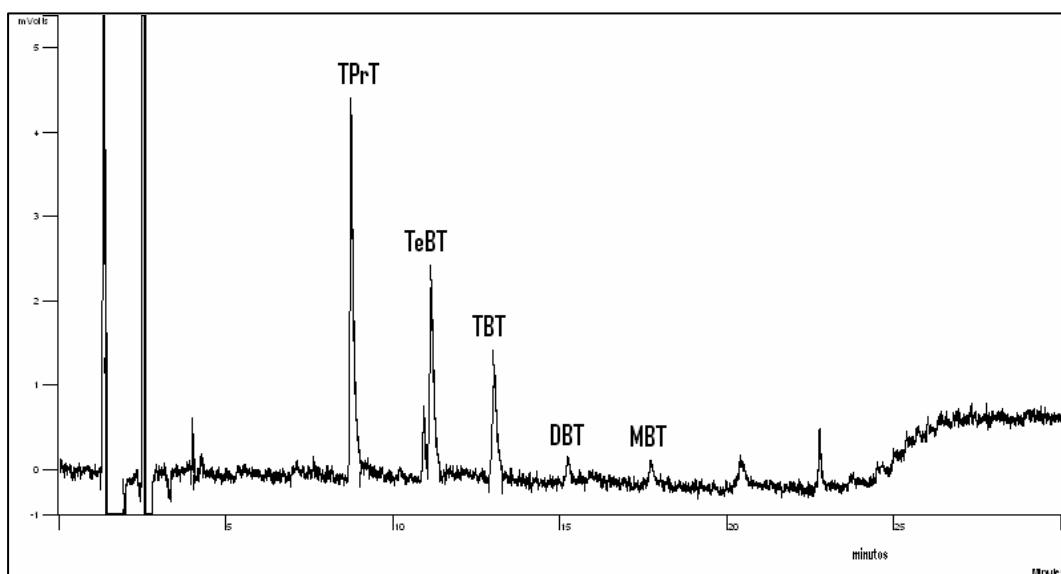


Figura A.8. Cromatograma do ponto 5.

Ponto 6 – Baía de Aratu Norte

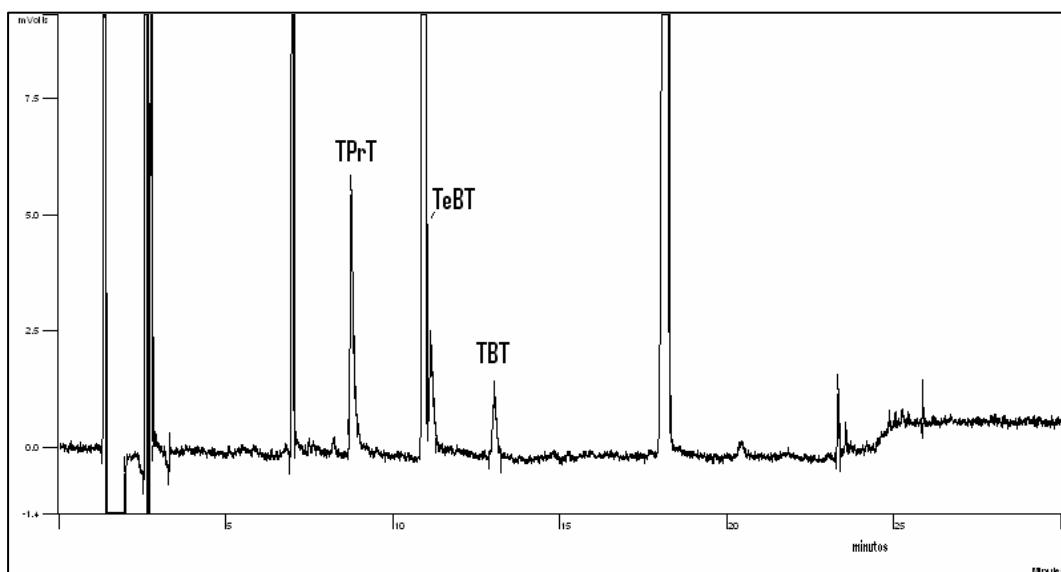


Figura A.9. Cromatograma do ponto 6.

Ponto 8 – Caboto Norte

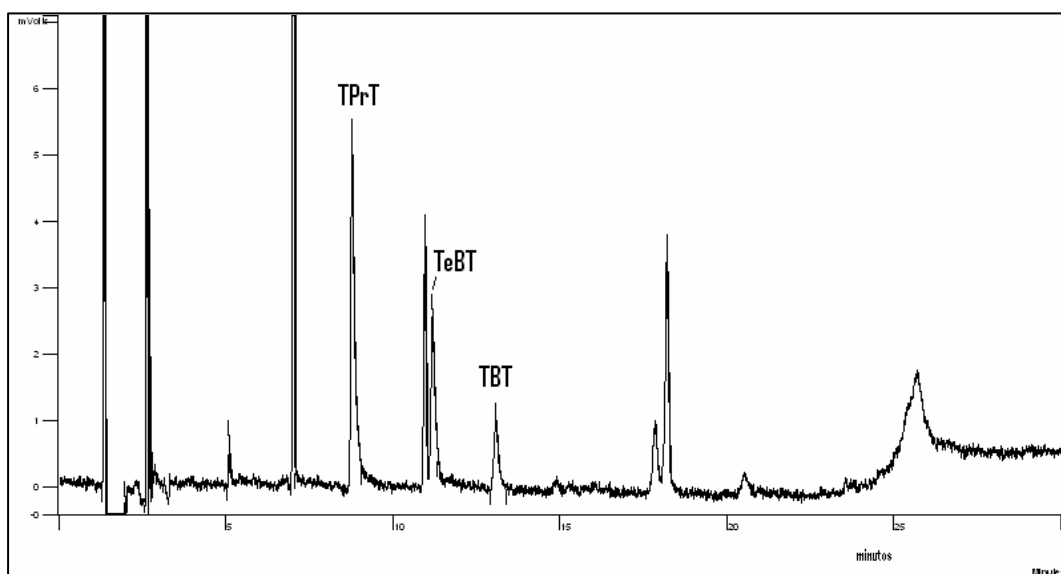


Figura A.10. Cromatograma do ponto 8.

Ponto 9 – Ponto da Cacimba

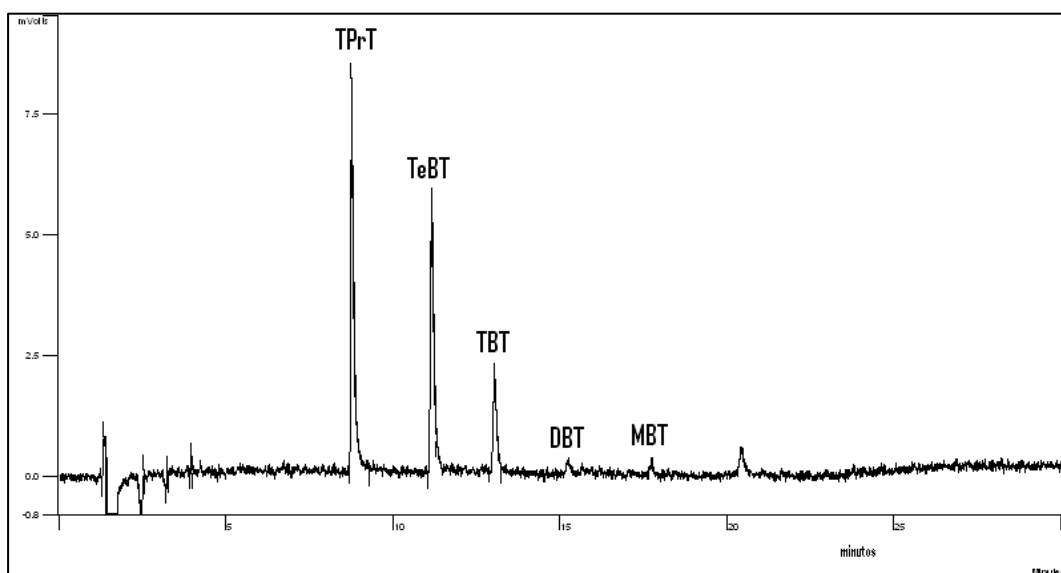


Figura A.11. Cromatograma do ponto 9.

Ponto 10 - Maré / São Paulo

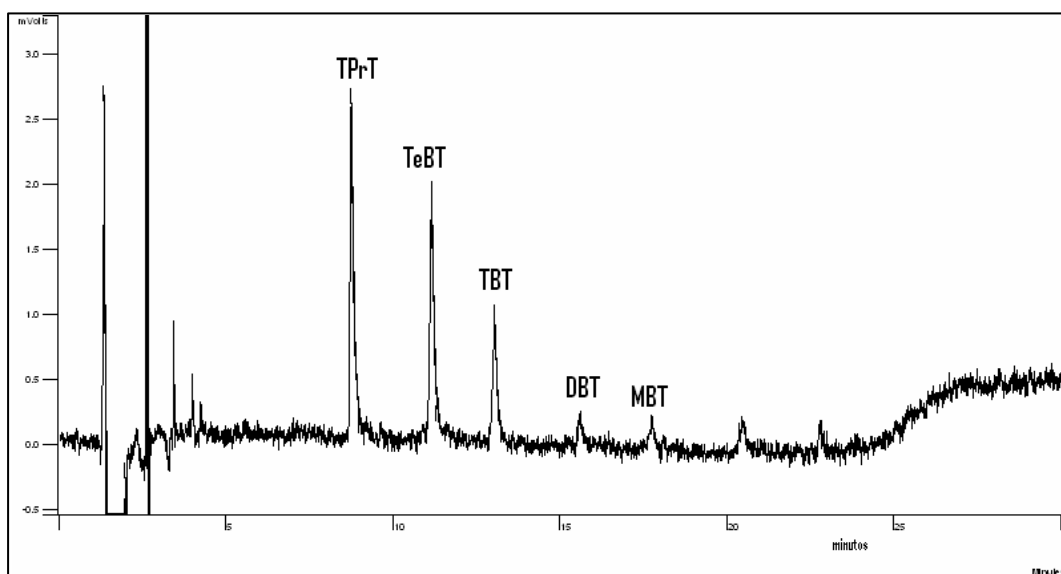


Figura A.12. Cromatograma do ponto 10.

Ponto 12 – Maré / Canal

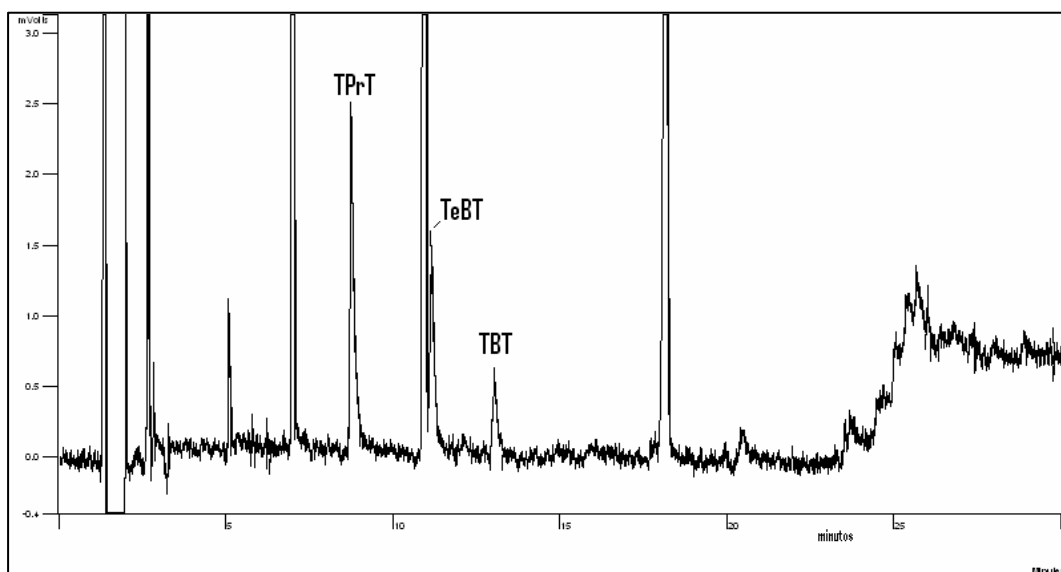
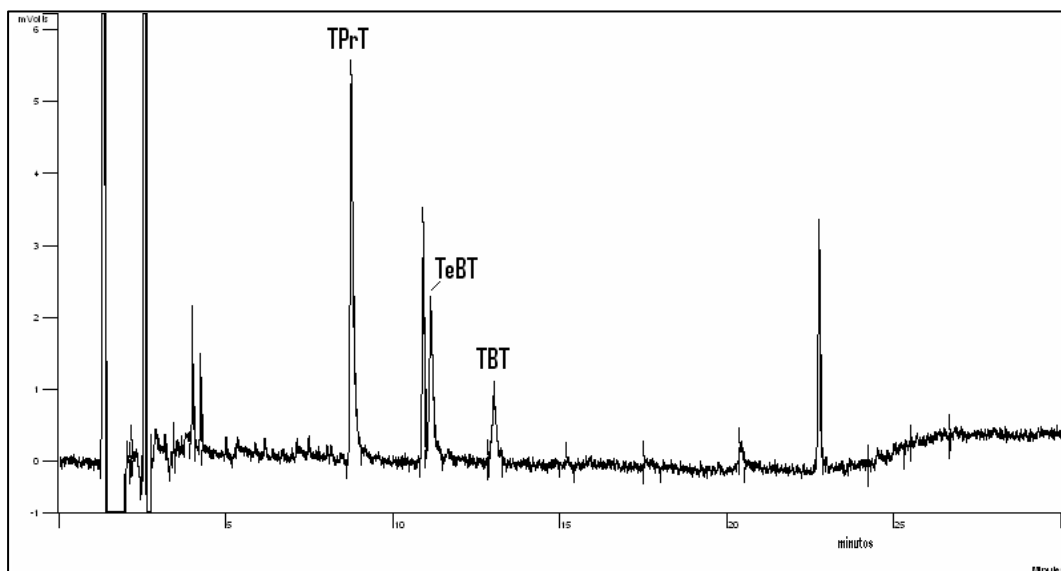
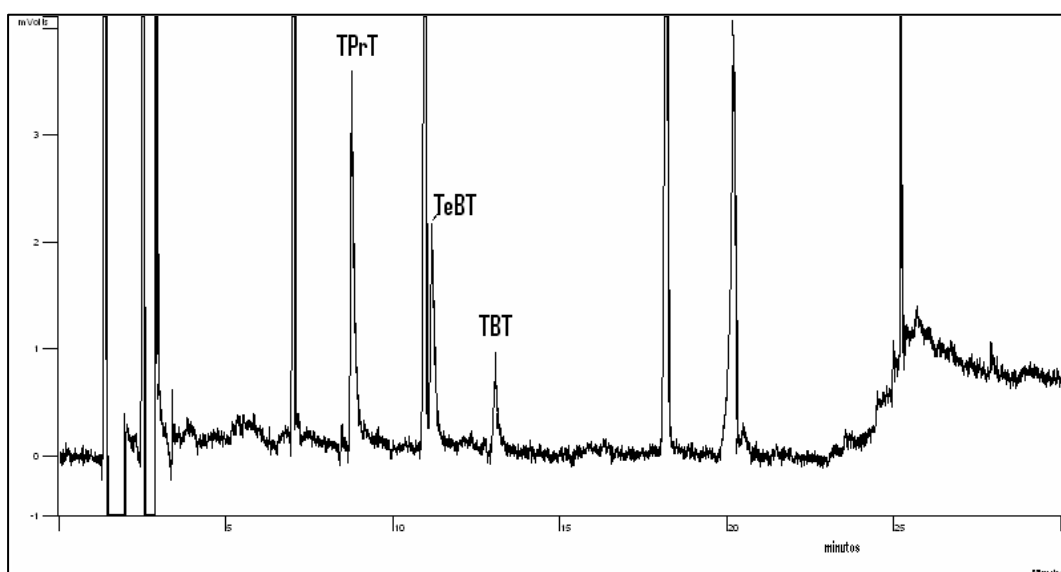


Figura A.13. Cromatograma do ponto 12.

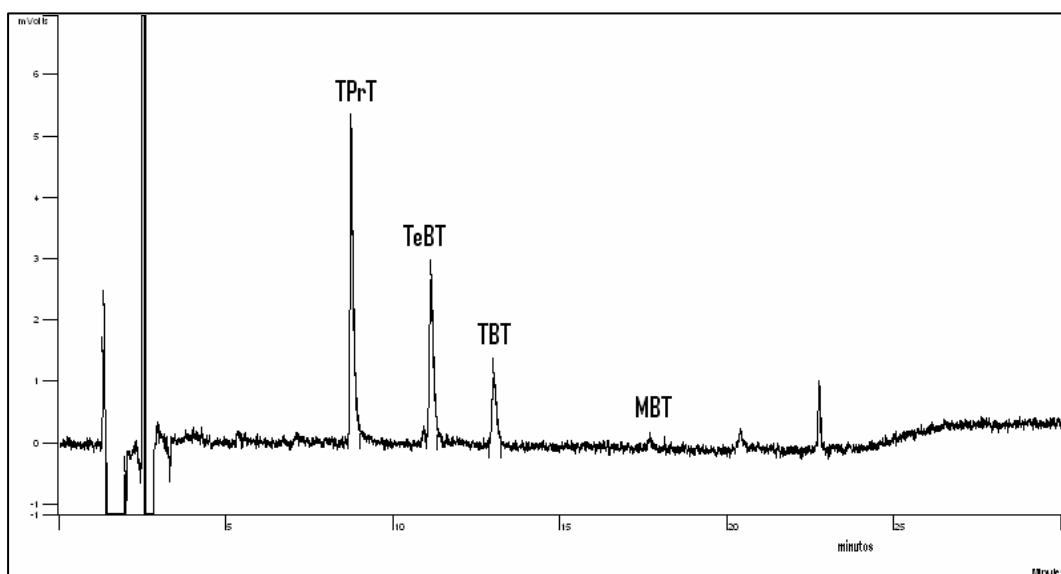
Ponto 14 - Fontes

**Figura A.14.** Cromatograma do ponto 14.

Ponto 15 – Ilha dos Porcos

**Figura A.15.** Cromatograma do ponto 15.

Ponto 16 – Frades

**Figura A.16.** Cromatograma do ponto 16.

Ponto 17 – Canal de Salvador

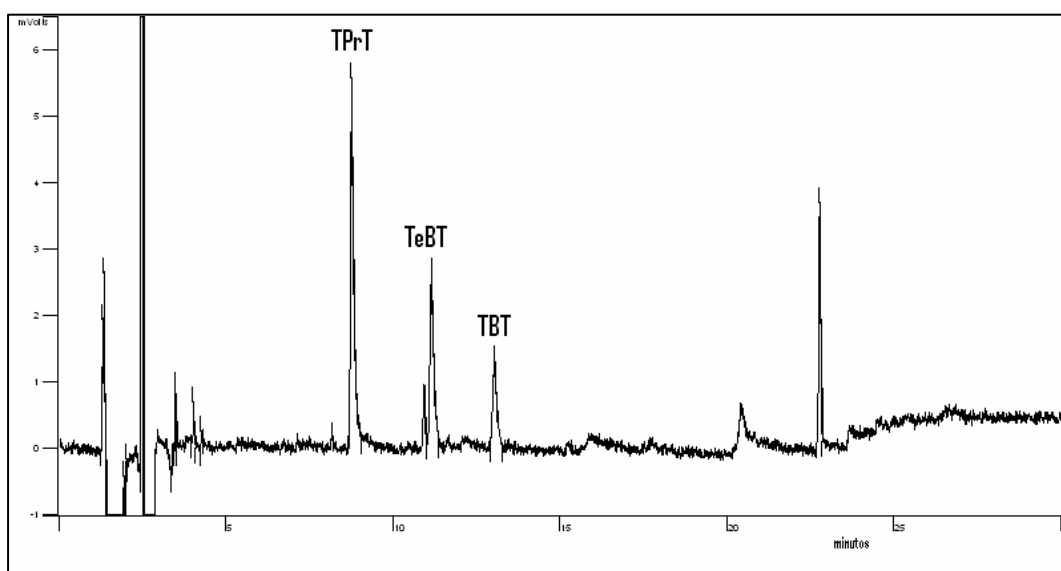
**Figura A.17.** Cromatograma do ponto 17.

Tabela A.3. Equipamentos usados em cada etapa do procedimento de determinação dos compostos orgânicos de estanho.

<i>Etapa</i>	<i>Equipamento</i>	<i>Especificações</i>
Extração	Balança	Mettler AE 200 e Mettler Toledo AT 261
	Vortex	Fisherbrand WhirliMixer 75 W, 50-60 Hz
	Ultrassom	Branson 5210R-MT 50-60 Hz, 1,5 Ampere
	Centrífuga	IEC Centra MP4R
Pós-extração: remoção do ácido acético	Evaporador rotativo	Yamato RE47 com Bomba de vácuo Marconi MA057
Derivação	Evaporador rotativo	Yamato RE47 com Bomba de vácuo Marconi MA057
Separação e identificação	Cromatógrafos em fase gasosa	Modelo CP-3800 – Varian
	Coluna capilar	DB-17 (30 m x 0,25 mm D.I. x 0,25 µm de espessura de filme) - J&W
	Detector PFPD	Composição da fase estacionária: 50% fenilmetilpolisiloxano Varian
	Softwares	Star Chromatography Workstation versão 5 - Varian

D.I. = diâmetro interno

Tabela A.4. Vidraria usada em cada etapa do procedimento de determinação dos compostos orgânicos de estanho.

<i>Etapa</i>	<i>Vidraria</i>	<i>Especificações</i>
Extração	Tubo de ensaio Pipeta volumétrica Pipeta graduada Pipetas Pasteur	Tampa com septo de teflon 10 mL 5 mL
Pós-extração: remoção do ácido acético	Funil de separação Pipeta volumétrica Becher Funil Balão Pipetas Pasteur	125 mL 10 mL 100 mL 50 mL com fundo redondo
Derivação	Tubo de ensaio Seringa Proveta Funil Balão Pipetas Pasteur	Tampa com septo de teflon 5 mL 10 mL 50 mL com fundo redondo
“Cleanup”	Coluna Proveta Frascos âmbar Pipetas Pasteur	0,5 cm de diâmetro interno e 10 cm de comprimento com torneira de teflon 25 mL 20 mL
Eliminação de enxofre	Espátula pequena Becher	50 mL

Tabela A.5. Reagentes, solventes e padrões utilizados em cada etapa do procedimento de determinação dos compostos orgânicos de estanho.

<i>Etapa</i>	<i>Reagente</i>	<i>Fórmula</i>	<i>Especificações</i>	<i>Fabricante</i>
Extração	Cloreto de tripropilestanho	$(C_3H_7)_3SnCl$	*	*
	Tolueno	$C_6H_5CH_3$	ChromAR [®] HPLC ou UltimAR [®]	Mallinckrodt Aldrich
	Ácido acético	CH_3COOH	99,7% ou Suprapur [®]	Merck
Pós-extração: remoção do ácido acético	Amônio pirrolidino ditiocarbamato (APDC)	$C_5H_8NS_2.NH_4$	97%	Fluka
	Lã de vidro		descontaminado em mufla (450°C)	Merck
	Sulfato de sódio anidro	Na_2SO_4	descontaminado em mufla (450°C)	Merck
Derivação	Brometo de pentil-magnésio	$CH_3(CH_2)_4HgBr$	Solução 2,0 mol L ⁻¹ em dietil-éter	Aldrich
	Água Milli-Q	H_2O	R.E. < 18MO cm ^a	Milli-Q TM Water System ZD20 (Millipore)
	Ácido clorídrico	HCl	37% fumegante	Merck
	Lã de vidro	*	*	*
	Sulfato de sódio anidro	*	*	*
“Cleanup”	Lã de vidro	*	*	*
	Óxido de alumínio 90 neutro ativo	Al_2O_3	70-230 mesh, descontaminado em mufla (450°C) 2% desativado	Merck
	Sulfato de sódio anidro	*	*	*
	Hexano	$CH_3(CH_2)_4CH_3$	95% n-hexano HPLC/CG UltimAR9 [®] OmniSolv [®]	Merck
Eliminação de enxofre	Nitrato de Prata Sílica Gel	$AgNO_3$ *	Gr para análise para coluna cromatográfica 60 (0,0063 – 0,200 mm)	Merck *

^a R.E. = resistência específica.

Tabela A.6. Compostos orgânicos de estanho padrões.

<i>Padrão</i>	<i>Abreviação</i>	<i>Fórmula</i>	<i>Pureza</i>	<i>Fabricante</i>	<i>CAS</i>
Tetrabutilestanho	TeBT	$(C_4H_9)_4Sn$	93%	Aldrich	[1461 – 25-2]
Cloreto de tributilestanho	TBTCl	$(C_4H_9)_3SnCl$	96%	Aldrich	[1461-22-9]
Dicloreto de dibutilestanho	DBTC ₂	$(C_4H_9)_2SnCl_2$	96%	Aldrich	[683-18-1]
Tricloreto de butilestanho	MBTC ₃	$(C_4H_9)SnCl_3$	95%	Aldrich	[1118-46-3]
Cloreto de tripropilestanho	TPrTCl	$(C_3H_7)_3SnCl$	> 98%	Aldrich	[2279-76-7]