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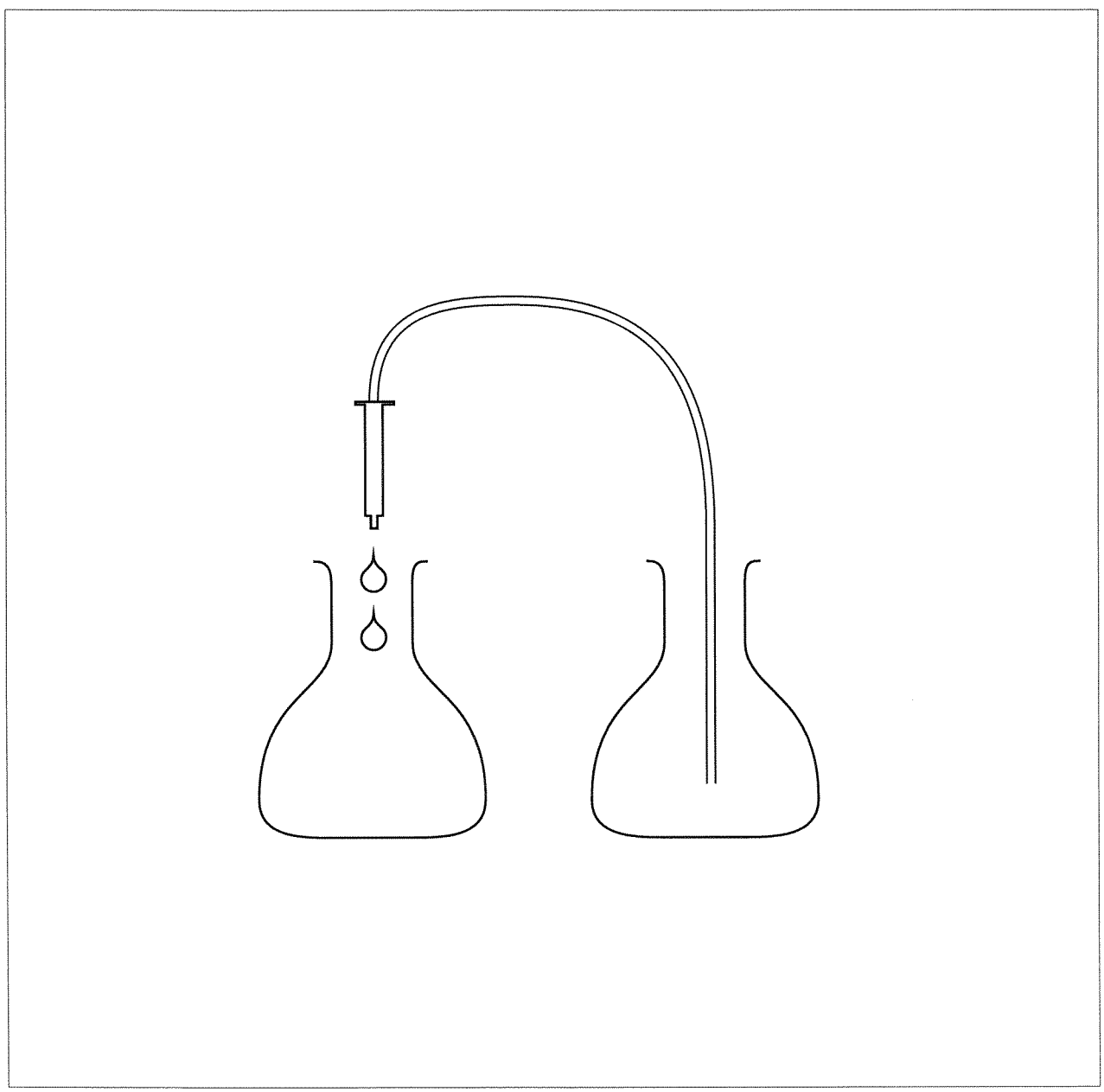
Development of an analytical method for the analysis of methylene-bis-thiocyanate at low level concentrations in water



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REPORT

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Abstract

An analytical method for the low level analysis of methylene-bis-thiocyanate (MBT) in waste water was developed. Prior to the instrumental analysis by HPLC, enrichment of MBT is achieved by solid phase extraction. Among several solid phase materials, best results were obtained using LiChrolut® EN (non-ionogenic, highly porous polystyrene-divinylbenzene polymer) which is known to retain polar substances. With the method developed, MBT may be determined at levels down to 1 µg/l water.

4 keywords, Norwegian	4 keywords, English
1. Analysemetodikk	1. Analytical method
2. Lave nivå av metylen-bis-tiocyanat	2. Low levels of methylene-bis-thoicyanate
3. Fast fase ekstraksjon	3. Solid phase extraction
4. HPLC analyse	4. HPLC analysis

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Development of an analytical method for the analysis of methylene-bis-thiocyanate at low level concentrations in waste water.

Preface

The aim of this study was to develop an analytical method for low level concentrations of methylene-bis-thiocyanate (MBT) in waste water from a paper mill. MBT is a micorbiocide and disinfectant which can be used as a wood preservative. MBT is classified as toxic to the environment with a no effect concentration value at 0.4 µg/l water. In December 96 Albright & Wilson made contact to look at the possibility to develop an analytical method were MBT could be detected down to 0.4 µg/l. The previous method used by Albright & Wilson was suitable for concentrations down to 1 mg/l.
The project was financially supported by Albright & Wilson.

Oslo, 100497

Torgunn Sætre

Contents

Summary

An analytical method for methylene-bis-thiocyanate at low level concentrations in water was developed. The method chosen was a solid phase extraction on a LiChrolut® EN column. The analyte was eluted from the column using ethylacetate. The extract was evaporated to dryness before redissolving in water. The detection limit of this method was 1 µg/l when 1 l water was extracted. Several other solid phases were tried, but no other showed satisfactory retention properties.
The final extracts were analysed by HPLC using a reversed phase column (C-18), detection by UV at 200, 205, 210 and 220 nm. The eluent was 90 % distilled water, 10 % methanol (v/v) acidified with conc. phosphoric acid.
An experiment by storing solutions of MBT in polyethylene bottles was carried out to see if MBT absorbs or adsorbs onto the bottle surface. There were no indications that this happened.
An experiment was carried out to test the method on a real paper mill water sample. The waste water had to be filtered before extraction on the LiChrolut® EN column. There were no differences if the water was spiked with MBT before or after the filtration.
With this method applied on a water sample from Rauma Paper, Finland, it was possible to detect MBT down to a concentration of 0.4 µg/l, but the peak was to small to be quantitated, partly because of overlap with unknown components in the waste water. A solution with concentration 1 µg/l was quantitated with a recovery of 60 %.

Table 1. Final analytical method:

Extraction column:	LiChrolut® EN
Conditioning solvent:	1 column volume methanol, 1 column volume distilled water. The column was not allowed to dry out.
Flowrate:	Approx. 0.75 l/hour
Elution solvent:	Ethylacetate
Elution volume:	2 x 3 ml
Final extract volume	1.0 ml distilled water
Analytical column:	Brownlee™ Columns Spheri 5 RP-18, 5 micron, 220 x 4.6 mm
Flow:	1 ml/min
Mobile phase:	90 % distilled water/10 % methanol (v/v) acidified with 1 drop conc. H_3PO_4 pr. 100 ml eluent.
Detection:	UV λ = 200 nm, 205 nm, 210 m and 220 nm. 0.001 AUFS.
Injection volume:	200 µl

1. Introduction

The objective of this study was to develop an analytical method for methylene-bis-thiocyanate (MBT) at low level concentration in waste water.
The study was initiated on January 21. 1997, and was completed the day the final report was signed. The experimental work was done in the laboratories at the Norwegian Institute for Water Research in the period from January 21. to March 22. 1997. All original data generated from this study and the final report are stored at the above location.

2. Methods

2.1 HPLC instrumental method

The HPLC method which was used was received from Albright & Wilson. A RP-18 column from Applied Biosystems was used instead of the Spherisorb ODS column which was described in the method. After some preliminary work the mobile phase was modified to separate the MBT from some unknown interfering peaks occurring in the waste water. The final mobile phase composition was 90 % distilled water/10 % methanol (v/v) acidified with 1 drop conc. H_3PO_4 for each 100 ml eluent. Both λ = 200, 205, 210 nm and 220 nm were used to assure that the detected compound was MBT.

Table 2. Analytical conditions used during the method development.

Analytical instrumentation:	Waters HPLC 600 gradient pump, 490 programmable UV detector, 717 autosampler and Millennium software.
Column:	Brownlee™ Columns Spheri 5 RP-18, 5 micron, 220 x 4.6 mm
Flow:	1 ml/min
Mobile phases:	70 % distilled water/30 % methanol (v/v), acidified with 1 drop conc. H_3PO_4 pr. 100 ml eluent, 85 % distilled water/15 % methanol (v/v), acidified with 1 drop conc. H_3PO_4 pr. 100 ml eluent and 90 % distilled water/10 % methanol (v/v), acidified with 1 drop conc. H_3PO_4 pr. 100 ml eluent.
Wavelengths:	200, 205, 210 and 220 nm
Injection volume:	200 µl

2.2 Chemicals and equipment

All chemicals used were HPLC or analytical grade.
A 0.45 µm HV filter from Millipore was used to filter the mobile phase.
A GF/C filter from Wathman was used to filter the water sample. Pore size: 1.20 µm

2.3 Flow chart - method development

2.4 Extraction methods - small volume trials.

2.4.1 C18 solid phase extraction

The column used in the extraction was a 6 ml tube containing 500 mg sorbent from Varian. The column was conditioned with one column volume of methanol followed by one column volume of distilled water. 2.0 ml of a 1 mg/l solution of MBT in distilled water was passed though the column at a flow rate of approximately 2 ml/min.
The column was then eluted with 2 x 2 ml methanol.
The extraction procedure was repeated, but using acetonitrile, acetone and dimethylformamide as eluent to remove the MBT from the column. The eluates were adjusted to 1.0 ml. Both the water passed through the column and the solvent extracts were analysed by HPLC.

2.4.2 Cyanopropyl solid phase extraction

The column used in the extraction was a 2.8 ml tube containing 500 mg sorbent from Varian. The column was conditioned with one column volume of methanol followed by one column volume of distilled water. 2.0 ml of a 1 mg/l solution of MBT in distilled water was passed through the column at a flow rate of approximately 2 ml/min.
The column was then eluted with 2 x 2 ml methanol.
The extraction procedure was repeated, but using acetonitrile and the original HPLC mobile phase (70 % distilled water, 30 % methanol acidified with 1 drop of conc. H_3PO_4 per 100 ml eluent) as eluent to remove the MBT from the column. The eluates were adjusted to 1.0 ml. Both the water passed through the column and the solvent extracts were analysed by HPLC.

2.4.3 LiChrolut® EN solid phase extraction

The column used in the extraction was a 3 ml tube containing 200 mg sorbent from Merck. The column was conditioned with one column volume of methanol followed by one column volume of distilled water. 2.0 ml of a 1 mg/l solution of MBT in distilled water was passed though the column at a flow rate of approximately 2 ml/min.
The column was then eluted with 2 x 2 ml methanol.
The extraction procedure was repeated, but using acetonitrile, acetone and dimethylformamide as eluent to remove the MBT from the column. The volume of the eluate was adjusted to 1.0 ml. Both the water passed through the column and the solvent extracts were analysed by HPLC.
In another experiment ethylacetate was used as the elution solvent. The eluate was evaporated to dryness and redissolved in 1.0 ml distilled water before analysis by HPLC.

2.5 Extraction - 1 litre volume trials

2.5.1 C-18 column.

1 litre of distilled water spiked with MBT to a concentration of 1.0 µg/l was extracted on a C-18 column. The extraction was carried out over a period of 1.5 h. Methanol, 2 x 2 ml, was used to remove the MBT from the column. After evaporation to dryness and redissolving in 1.0 ml of distilled water, the extract was analysed by HPLC.

2.5.2 LiChrolut® EN column.

1 litre of distilled water spiked with MBT to a concentration of 1.0 µg/l was extracted on a LiChrolut® EN column. The extraction was carried out over a period of 1.5 hours. Ethylacetate, 3 ml,

was used to remove the MBT from the column. After evaporation to dryness and redissolving in 1.0 ml distilled water, the water extract was analysed. The same extraction column was eluted once more with 3 ml ethylacetate, and the eluate was treated as above.
The extraction was repeated using 2 x 3 ml ethylacetate as the elution solvent. The extract was evaporated to dryness, and redissolved in 1.0 ml of distilled water before analysis by HPLC.

2.5.3 Liquid liquid extraction

The solubility of MBT was tested in ethylacetate, diethyleter and dichloromethane. The MBT showed to be freely soluble in ethylacetate and in dichloromethane, but ethylacetate is too soluble in water to be an effective liquid liquid extraction solvent. A 1.0 µg/l solution of MBT in distilled water was extracted with dichloromethane, 2 x 50 ml. The extraction process was carried out over a 30 min period each time. The two dichloromethane extracts were evaporated to dryness, and then redissolved in 1.0 ml distilled water before analysis by HPLC.
The extraction was repeated, but using 50 g sodium chloride to salt out the MBT from the water phase. The dichloromethane extracts were treated as in the previous experiment.

2.5.4 Extraction of lower levels of MBT.

A 0.4 µg/l solution of MBT in distilled water was extracted on a LiChrolut® EN solid phase in the same manner as the 1.0 µg/l solution, using ethylacetate, 2 x 3 ml, as the elution solvent. The eluate was evaporated to dryness and redissolved in 1.0 ml distilled water before analysis by HPLC.

2.6 Storage in polyethylene bottles.

A 0.4 µg/l and a 1.0 µg/l solution were stored for one week in 1 litre polyethylene bottles at 4°C before sample workup and analysis. The work up procedure was extraction on a LiChrolut® EN extraction column, followed by elution with ethylacetate, 2 x 3 ml. The extracts were evaporated to dryness before redissolving in 1.0 ml distilled water. The extracts were analysed by HPLC.

2.7 Extraction of a real water sample.

2.7.1 Extraction of effluent water spiked with MBT.

Effluent water from a paper mill containing visible particles, which was known *not* to have been in contact with MBT, was used in the experiments. Experiments were conducted to verify if MBT absorbs or adsorbs to the particles in the water.

Two aliquots of the effluent water were filtered, pH adjusted to 5.5 with conc. phosphoric acid to avoid loss of MBT due to hydrolysis, and spiked with MBT to a level of 1 µg/l and 0.4 µg/l. The water aliquots were extracted on LiChrolut® EN columns as described above.

Two other aliquots of the effluent water were acidified to pH 5.5 with conc. phosphoric acid and spiked with MBT to a level of 1.0 µg/l and 0.4 µg/l. The samples were stored for two days at 4 °C prior to filtration. The filtrates were extracted in the same manner as described above.

The solvent eluates, were evaporated to dryness and redissolved in distilled water prior to analysis by HPLC.

2.7.2 Extraction of a "blank" effluent water.

One litre of effluent water was filtered and extracted in the same way as described above before analysis by HPLC.

3. Test Article, Methylene-bis-thiocyanate.

The test article, received in a glass bottle from Albright & Wilson, was stored at room temperature during the test period.

3.1 Description of the test article.

Methylene-bis-thiocyanate, lab code B263/1

Empirical formula:	$C_3H_2N_2S_2$
Molecular weight:	130
CAS. Number:	6317-18-6
Physical appearance:	Yellow granular solid
B.Nr.:	616C
Water solubility:	2.8 g/l

4. Results and Discussion

4.1 Solid phase extraction.

4.1.1 C-18 column.

Analysis of the water sample which had passed trough the column did not show any MBT. The MBT was retained on the column. During analysis of the various solvent extracts, MBT could not be detected in any of them, likely due to elution with the solvent as discussed in chapt. 4.2.

4.1.2 Cyanopropyl column.

The water sample which had passed through the column was analysed by HPLC, and contained detectable amounts of MBT. e.g. the MBT did not quantitatively retain on the column.

4.1.3 LiChrolut® EN column.

Analysis of the water sample which had passed trough the column did not show any MBT. The MBT was retained on the column. During analysis of the various solvent extracts, MBT could not be detected in any of them, likely due to elution with the solvent as discussed in chapt. 4.2.
However, analysing the ethylacetate extract which was evaporated to dryness and redissolved in 1.0 ml distilled water, showed some MBT. Using more ethylacetate e.g. 2 x 3 ml and evaporating the extract to dryness before redissolving in 1.0 ml distilled water gave MBT to a quantitative yield.

4.2 Solvents influence on the elution of the MBT on the HPLC system.

When no peaks were detected in any of the solvent extracts analysed by HPLC, the mobile phase was changed from the original composition to 85 % distilled water and 15 % methanol (v/v) and acidified in the same manner as the previous mobile phase. This did not solve the problem with the missing MBT peak at the expected retention time. The methanol and acetonitrile extracts from both the C18 and the LiChrolut® EN columns were evaporated to dryness, and redissolved in distilled water.

Analysing this water the MBT peak was detected in the methanol and acetonitrile extracts from the C18 column. There were no peaks detected in the eluates from the LiChrolut® EN columns.
The injection volume was as large as 200 µl. Injecting such a big volume pure solvent will disturb the chromatographic properties on the separation column, and the MBT will migrate in the solvent peak. To avoid this problem, the extraction solvent had to be removed by evaporation, followed by redissolving of the MBT in distilled water.

4.3 Extraction of 1 litre 1 µg/l MBT in distilled water

4.3.1 C-18 column

There was no MBT found in the solvent extract. The MBT was not retained on the column while the water volume was as large as 1 l.

4.3.2 LiChrolut® EN column

The solvent extract contained some MBT, but only to about 40 % yield. The elution from the same extraction tube was repeated once. The yield was about 30 %.
The total extraction was repeated using 2 x 3 ml ethylacetate to elute the MBT from the column. The extraction showed a 85 % yield.
The extraction procedure was repeated with lower content of MBT, e.g. 0.4 µg/l distilled water.
The extraction efficiency was measured to 82.5 % (Table 3, Appendix A)

4.3.3 Liquid liquid extraction.

The extracts were analysed by HPLC. The yield was approximately 30 %. Salting out the MBT had the opposite effect of what was expected. MBT was not detected in the extracts after addition of salt to water.

4.4 Samples stored in polyethylene bottles.

The extracts were analysed by HPLC. The yield was in the same order of magnitude that were shown for freshly prepared samples, e.g. 83 % for the 1 µg/l solution and 85 % for the 0.4 µg/l solution. There were no indications that the MBT adsorbs or absorbs to the polyethylene bottle. (Table 3, Appendix A)

4.5 Extraction of a real water sample.

4.5.1 Extraction of spiked effluent water.

The extracts were analysed by HPLC. There were observed some interferences with the chromatographic system applied. The mobile phase was changed to 90 % distilled water/10 % methanol and acidified with phosphoric acid, 1 drop pr 100 ml eluent. The interferences and the MBT could be separated with this eluent.
There were no differences in the recovery between the sample which was filtered prior to spiking and the sample which was spiked before filtering. The yield was 60 % in both cases for the 1 µg/l solution. (Table 3, Appendix A). The signal for the 0.4 µg/l solution was to low to be quantitated but it is possible to detect the compound.

4.5.2 Extraction of a "blank" effluent water.

The extract was analysed by HPLC. At the retention time expected for MBT no response could be detected. Appendix B

5. Conclusion

As there was no MBT found in the extract from the C-18 column when 1 litre of distilled water was spiked with MBT, another solid phase extraction medium had to be tried. The LiChrolut® EN phase does retain polar compounds better than the C-18 phase. In the tests performed on small volumes there were no MBT in the water after extraction. The MBT was retained on the column. As the MBT was shown to be very soluble in ethylacetate, that solvent was tried as an extraction solvent from the solid phase. Analysing of ethylacetate extracts which had been evaporated to dryness and redissolved in distilled water showed MBT in the extracts.
Liquid liquid extraction using dichloromethane there was no enough MBT detected in the extract, only about 30 % yield.
Extraction of MBT in 1 litre distilled water samples was performed on the LiChrolut® EN column. The extraction efficiency was shown to be 85 %. This technique was tried on a lower level of MBT in distilled water, and showed to work out satisfactory.
The ratios between the UV absorptions at 200nm, 205 nm, 210 nm and 220 nm are given in table 5 Appendix A.
A test was run to see if samples can be stored in polyethylene bottles. There were no indications that the MBT adsorbs or absorbs to the bottle using distilled water in the experiment.
The method was shown to work well on real water samples as well, but with a lower recovery at about 60 %. For the waste water tested, it was possible to detect MBT in a 0.4 µg/l spiked sample, but the signal was to low to be quantified. The 1.0 µg/l solution was quantified. There were no differences in the results whether the water was spiked before or after filtration.
For other water samples there might be other interferences which can disturb the determination of low levels of MBT.

Table 3. Final analytical method:

Extraction column:	LiChrolut® EN
Conditioning solvent:	1 column volume methanol, 1 column volume distilled water. The column was not allowed to dry out.
Flowrate:	Approx. 0.75 l/hour
Elution solvent:	Ethylacetate
Elution volume:	2 x 3 ml
Final extract volume	1.0 ml distilled water
Analytical column:	Brownlee™ Columns Spheri 5 RP-18, 5 micron, 220 x 4.6 mm
Flow:	1 ml/min
Mobile phase:	90 % distilled water/10 % methanol (v/v) acidified with 1 dr. H_3PO_4 pr. 100 ml eluent.
Detection:	UV λ = 200 nm, 205 nm, 210 m and 220 nm. 0.001 AUFS.
Injection volume:	200 µl

Appendix A.

Table 4. Extraction efficiency.

Comments	Real concentration	Found	Deviation	Yield %
Distilled water spiked with MBT	1.0 µg/l	0.85	0.15	85.0
Distilled water spiked with MBT	0.4 µg/l	0.33	0.07	82.5
Distilled water spiked with MBT, stored on polyethylene bottles for 1 week	1.0 µg/l	0.83	0.17	83.0
Distilled water spiked with MBT, stored on polyethylene bottles for 1 week	0.4 µg/l	0.34	0.06	85.0
Real paper mill water sample, filtered and spiked with MBT	1.0 µg/l	0.61	0.39	61.0
Real paper mill water sample, filtered and spiked with MBT	0.4 µg/l			
Real paper mill water sample, spiked with MBT, stored two days and filtered.	1.0 µg/l	0.58	0.42	58.0
Real paper mill water sample, spiked with MBT, stored two days and filtered.	0.4 µg/l			

Table 5. Ratio between the response measured with different wavelengths.

Wavelengths	Ratio
$\frac{210nm}{220nm}$	3.4±0.1
$\frac{205nm}{220nm}$	4.8±0.4
$\frac{200nm}{220nm}$	6.2±0.3

Appendix B.

Chromatograms of MBT extracted from water samples.

Laboratory water spiked with 1.0 ppb MBT. Mobile phase 85 % water 15 % methanol, 1 drop phosphoric acid pr 100 ml eluent. $\lambda = 210$ nm.

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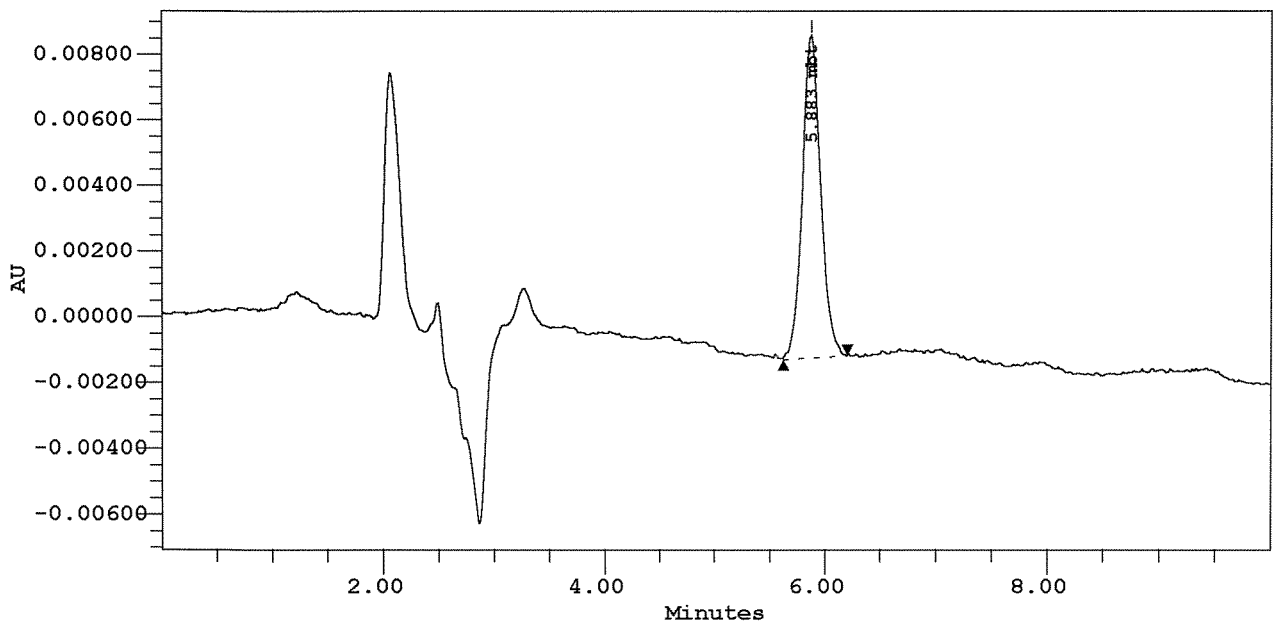
Report Method: mbt Version: 2.15
For Sample: 1 ppb Vial: 3 Injection: 1 Channel: 490 Ch2
Proc Chan: 490 Ch2 Processed: 17/02/97 10:29:06
Channel Descr:

Millennium Sample Information

Project Name:	MBT	Acquired By:	tos_niva
Sample Name:	1 ppb		
Vial:	3	Sample Type:	Unknown
Injection:	1	Volume:	200.00µl
Channel:	490 Ch2	Run Time:	10.0 min
Date Acquired:	17/02/97 10:11:10	Date Processed:	17/02/97 10:29:06
SampleWeight:	1.00000	Dilution:	1.00000
Acq Meth Set:	MBT		
Processing Method:	mbt85_15mfuv210nm		



Peak Results

#	Name	Ret Time (min)	Area (uV*sec)	Height (uV)	Amount	Int Type
1	mbt	5.883	112882	9814	0.829	BB

Laboratoy water spiked with 0.4 ppb MBT. Mobile phase 85 % water 15 % methanol, 1 drop phosphoric acid pr 100 ml eluent. $\lambda = 210$ nm.

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Report Method: mbt Version: 2.15

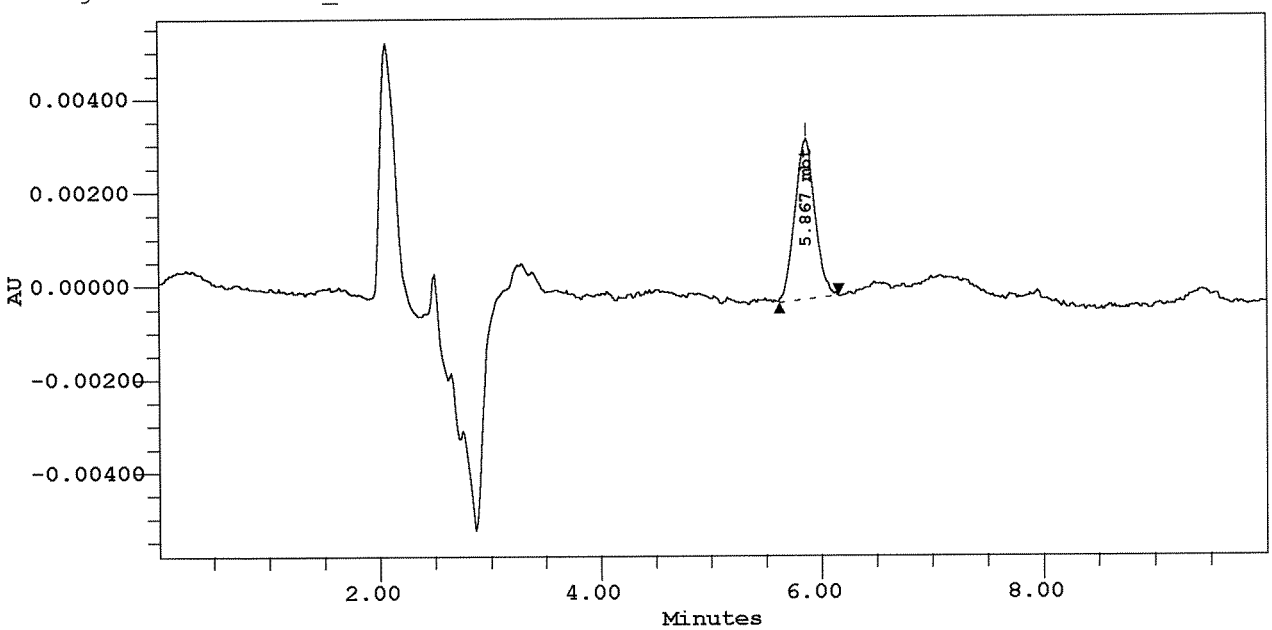
For Sample: 0.4 ppb Vial: 4 Injection: 1 Channel: 490 Ch2

Proc Chan: 490 Ch2 Processed: 17/02/97 10:36:08

Channel Descr:

Millennium Sample Information

Project Name:	MBT	Acquired By:	tos_niva
Sample Name:	0.4 ppb		
Vial:	4	Sample Type:	Unknown
Injection:	1	Volume:	200.00µl
Channel:	490 Ch2	Run Time:	10.0 min
Date Acquired:	17/02/97 10:24:41	Date Processed:	17/02/97 10:36:08
SampleWeight:	1.00000	Dilution:	1.00000
Acq Meth Set:	MBT		
Processing Method:	mbt85_15mfuv210nm		



Peak Results

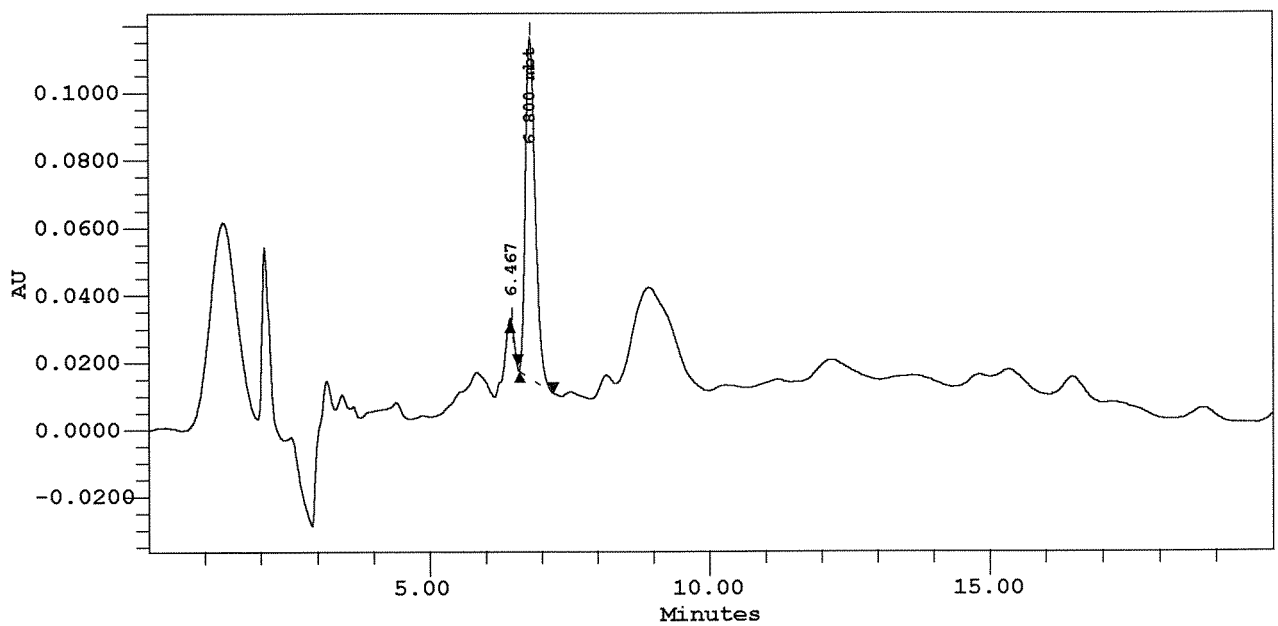
#	Name	Ret Time (min)	Area (uV*sec)	Height (uV)	Amount	Int Type
1	mbt	5.867	42288	3399	0.324	BB

Effluent water spiked with 10 ppb MBT. Mobile phase 90 % water 10 % methanol, 1 drop phosphoric acid pr 100 ml eluent. $\lambda = 210$ nm.

Millennium Results Report March 26, 1997 Page: 1 of 1
Report Method: mbt Version: 2.15
For Sample: 10 PPB Vial: 3 Injection: 1 Channel: 490 Ch2
Proc Chan: 490 Ch2 Processed: 21/03/97 05:41:24
Channel Descr:

Millennium Sample Information

Project Name:	MBT	Acquired By:	tos_niva
Sample Name:	10 PPB		
Vial:	3	Sample Type:	Unknown
Injection:	1	Volume:	200.00µl
Channel:	490 Ch2	Run Time:	20.0 min
Date Acquired:	21/03/97 03:50:10	Date Processed:	21/03/97 05:41:24
SampleWeight:	1.00000	Dilution:	1.00000
Acq Meth Set:	MBT		
Processing Method:	mbt140397_210nm		



Peak Results

#	Name	Ret Time (min)	Area (uV*sec)	Height (uV)	Amount	Int Type
1	mbt	6.800	1229184	101136	7.355	BB

Effluent water spiked with 1.0 ppb MBT. Mobile phase 90 % water 10 % methanol, 1 drop phosphoric acid pr 100 ml eluent. $\lambda = 210$ nm.

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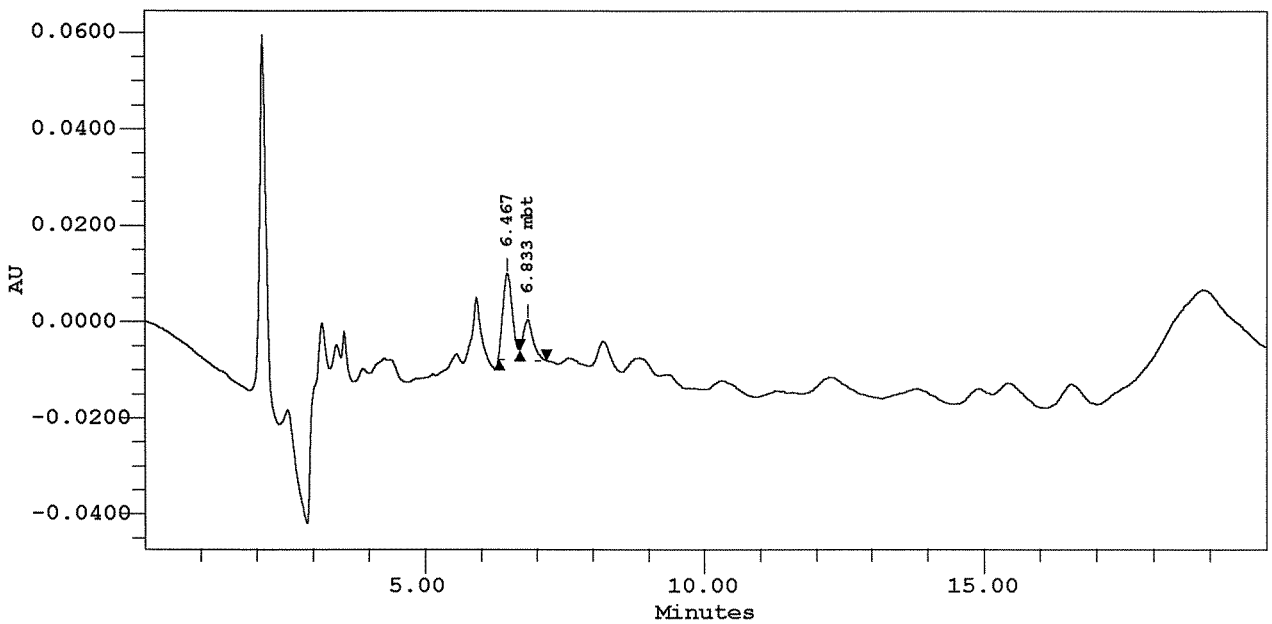
Report Method: mbt Version: 2.15
For Sample: 1 PPB Vial: 1 Injection: 1 Channel: 490 Ch2
Proc Chan: 490 Ch2 Processed: 26/03/97 03:22:11
Channel Descr:

Millennium Sample Information

Project Name:	MBT	Acquired By:	tos_niva
Sample Name:	1 PPB		
Vial:	1	Sample Type:	Unknown
Injection:	1	Volume:	200.00µl
Channel:	490 Ch2	Run Time:	20.0 min
Date Acquired:	21/03/97 03:03:11	Date Processed:	26/03/97 03:22:11
SampleWeight:	1.00000	Dilution:	1.00000
Acq Meth Set:	MBT		
Processing Method:	mbt140397_210nm		



Peak Results

#	Name	Ret Time (min)	Area (uV*sec)	Height (uV)	Amount	Int Type
1	mbt	6.833	105451	8314	0.592	VM

Effluent water spiked with 0.4 ppb MBT. Mobile phase 90 % water 10 % methanol, 1 drop phosphoric acid pr 100 ml eluent. $\lambda = 210$ nm.

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Report Method: mbt Version: 2.15

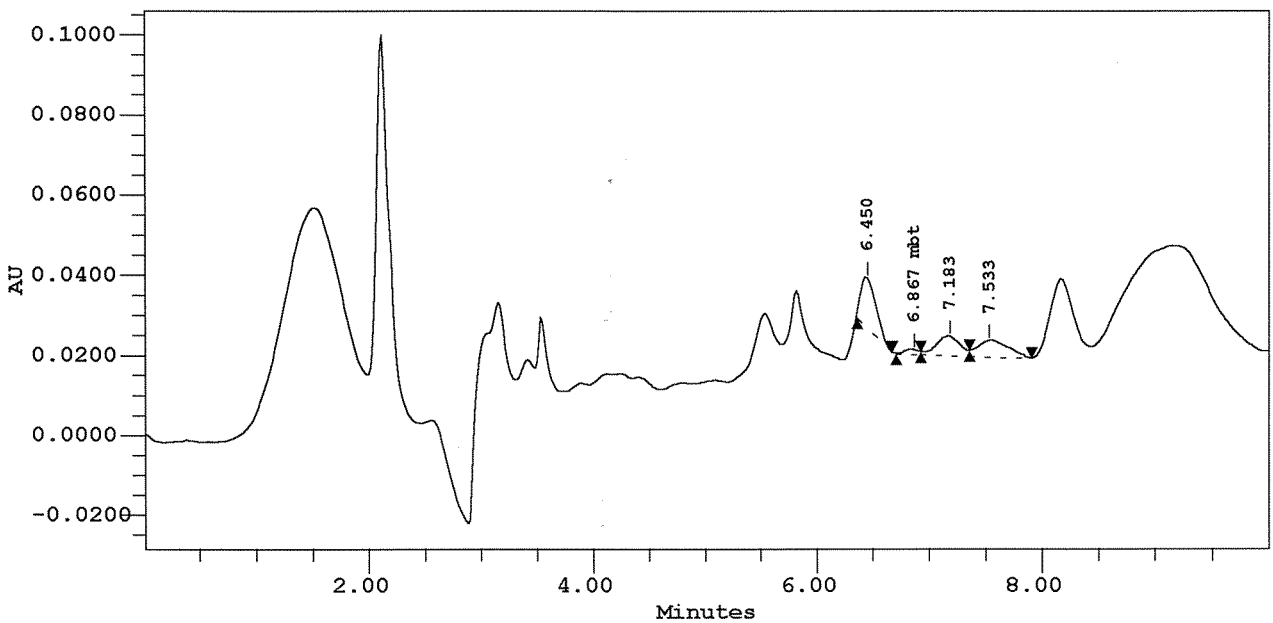
For Sample: 0.4ppb Vial: 5 Injection: 1 Channel: 490 Ch2

Proc Chan: 490 Ch2 Processed: 21/03/97 07:30:33

Channel Descr:

Millennium Sample Information

Project Name:	MBT	Acquired By:	tos_niva
Sample Name:	0.4ppb		
Vial:	5	Sample Type:	Unknown
Injection:	1	Volume:	180.00µl
Channel:	490 Ch2	Run Time:	10.0 min
Date Acquired:	21/03/97 07:16:00	Date Processed:	21/03/97 07:30:33
SampleWeight:	1.00000	Dilution:	1.00000
Acq Meth Set:	MBT		
Processing Method:	mbt140397_210nm		



Peak Results

#	Name	Ret Time (min)	Area (uV*sec)	Height (uV)	Amount	Int Type
1	mbt	6.867	9405	1205	0.014	BV

"Blank" efflunet water. Mobile phase 90 % water 10 % methanol, 1 drop phosphoric acid pr 100 ml eluent. $\lambda = 210$ nm.

Millennium Results Report		March 26, 1997			Page: 1 of 1
Report Method:	mbt		Version:	2.15	
For Sample:	BL PR	Vial: 2	Injection:	1	Channel: 490 Ch2
Proc Chan:	490 Ch2		Processed:	21/03/97 07:47:09	
Channel Descr:					

Millennium Sample Information

Project Name:	MBT	Acquired By:	tos_niva
Sample Name:	BL PR		
Vial:	2	Sample Type:	Unknown
Injection:	1	Volume:	200.00µl
Channel:	490 Ch2	Run Time:	20.0 min
Date Acquired:	21/03/97 05:00:04	Date Processed:	21/03/97 07:47:09
SampleWeight:	1.00000	Dilution:	1.00000
Acq Meth Set:	MBT		
Processing Method:	mbt140397_210nm		

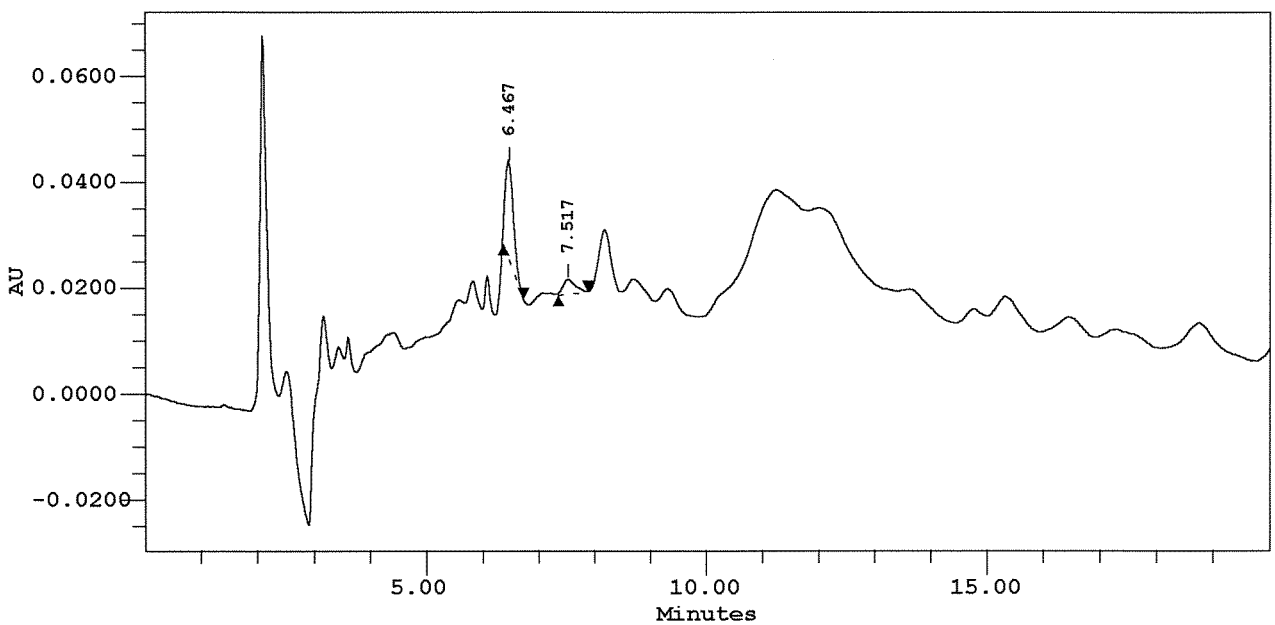
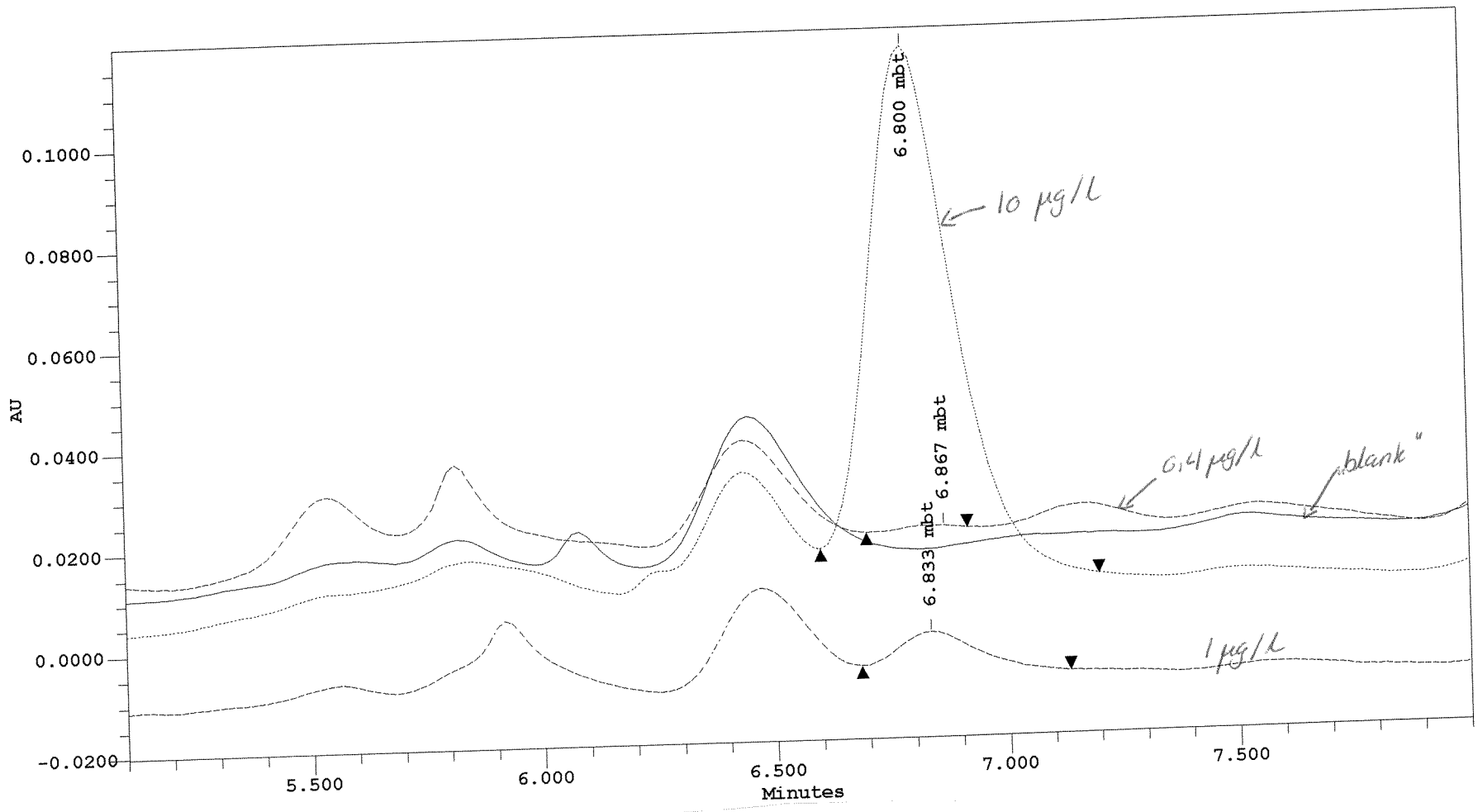
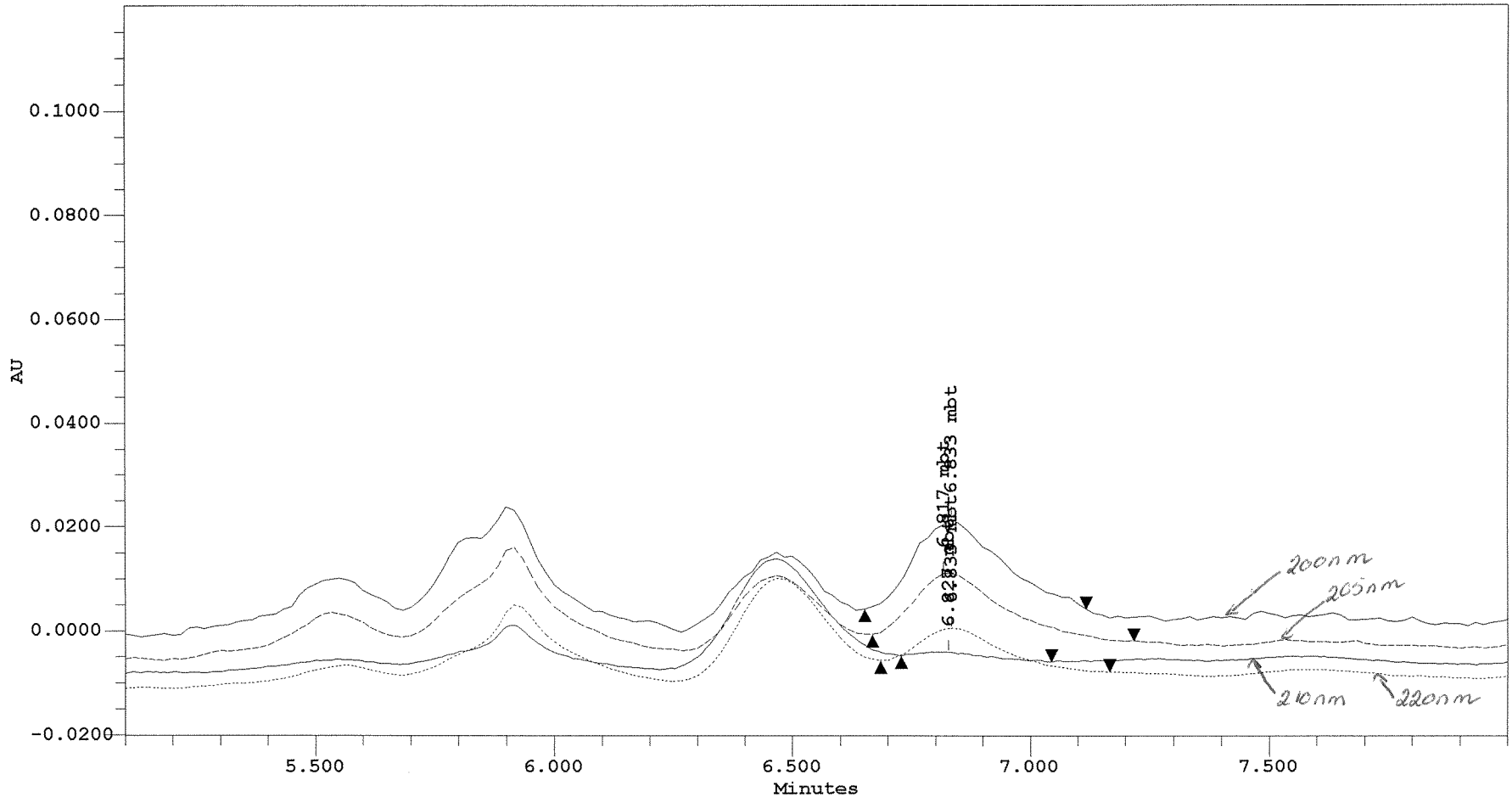


Table 'Peak Results' contains no data.



Chromatogram overlay. Effluent water spiked with 10 ppb, 1 ppb and 0.4 ppb MBT, and "blank" effluent water.



Chromatogram overlay. Effluent water spiked with 1.0 ppb MBT. Mobile phase 90 % water 10 % methanol, 1 drop phosphoric acid pr 100 ml eluent. 4 different wavelengths.

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