

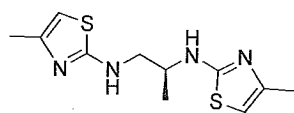
Synthesis of chiral polyaminothiazoles

Sergey Arutyunyan and Adel Nefzi*

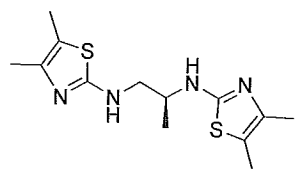
Torrey Pines Institute for Molecular Studies
11350 SW Village Parkway, Port Saint Lucie, FL 34987

Supporting Information

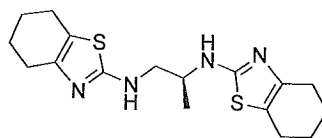
Structures of the dithiazoles



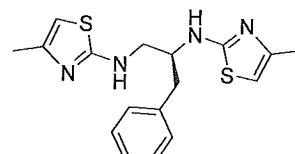
5a



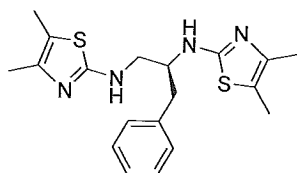
5b



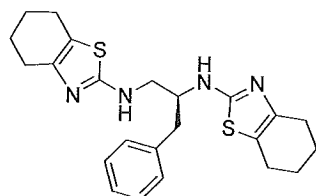
5c



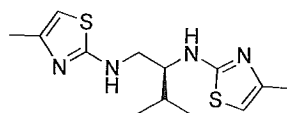
5d



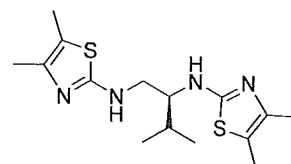
5e



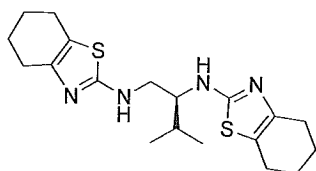
5f



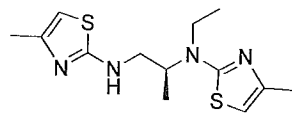
5g



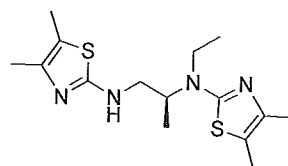
5h



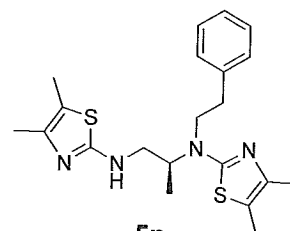
5m



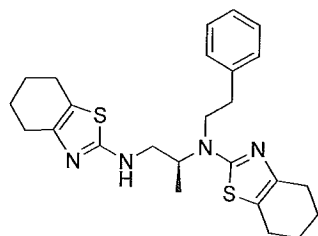
5n



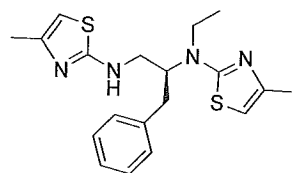
5o



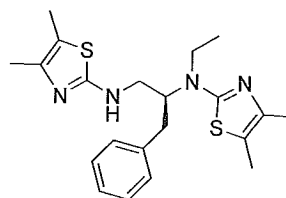
5p



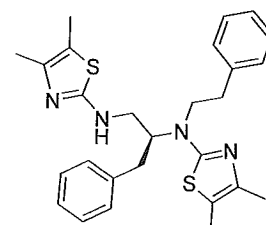
5i



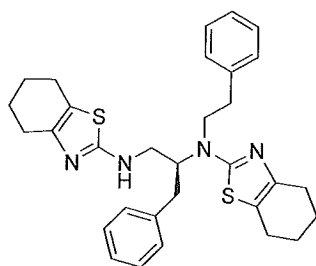
5j



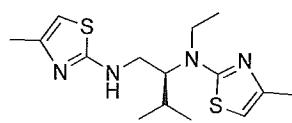
5k



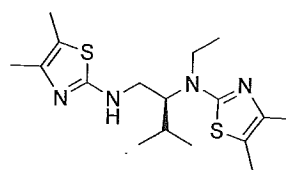
5l



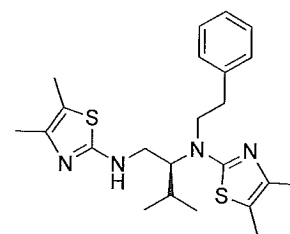
5q



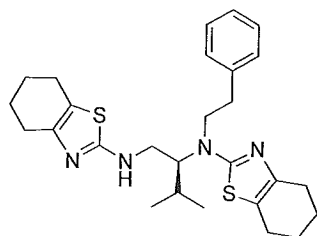
5r



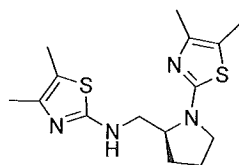
5s



5t

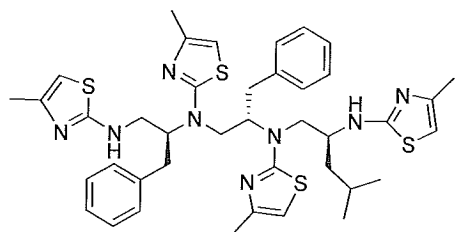


5u

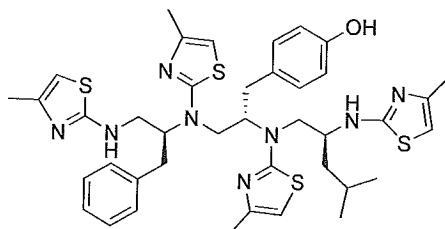


5v

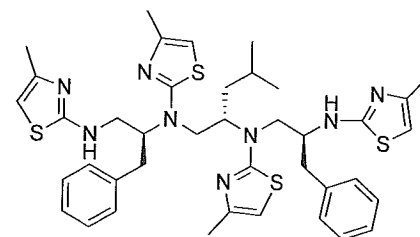
Structures of the tetrathiazoles



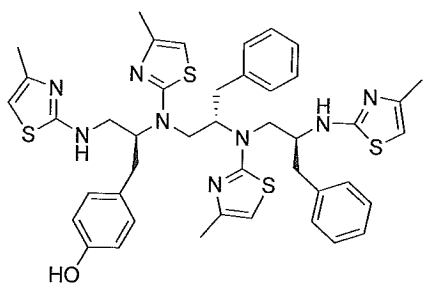
PT-1



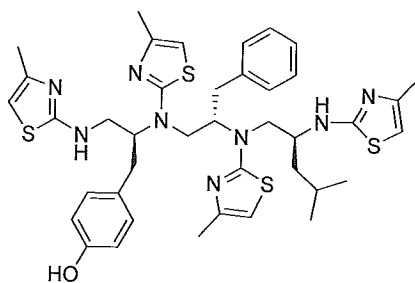
PT-2



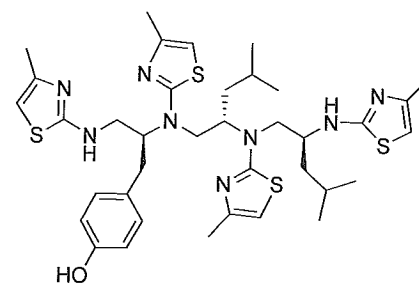
PT-3



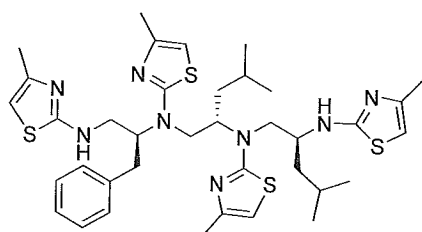
PT-4



PT-5



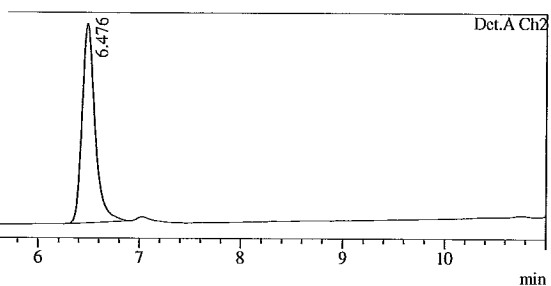
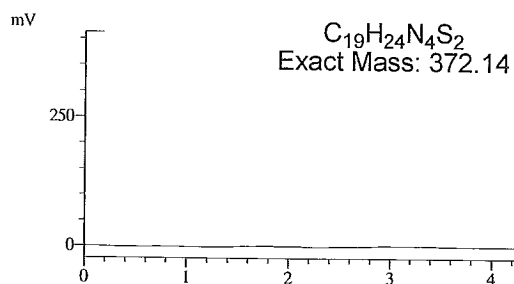
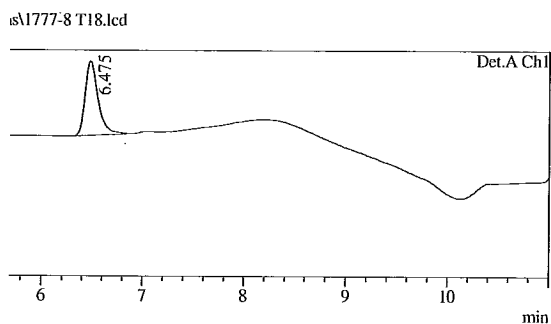
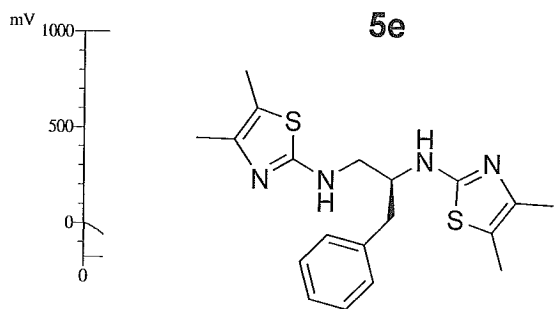
PT-6



PT-7

Date Acquired : 10/22/2009 4:11:48 PM
 Sample Name : 1777-8
 Sample ID : T18
 Vial # : 96
 Injection Volume : 5
 Data File : 1777-8 T18.lcd
 Method File : 6min 5.05 1Mn Clean(254) 214AQ1 1.lem

Sample Information



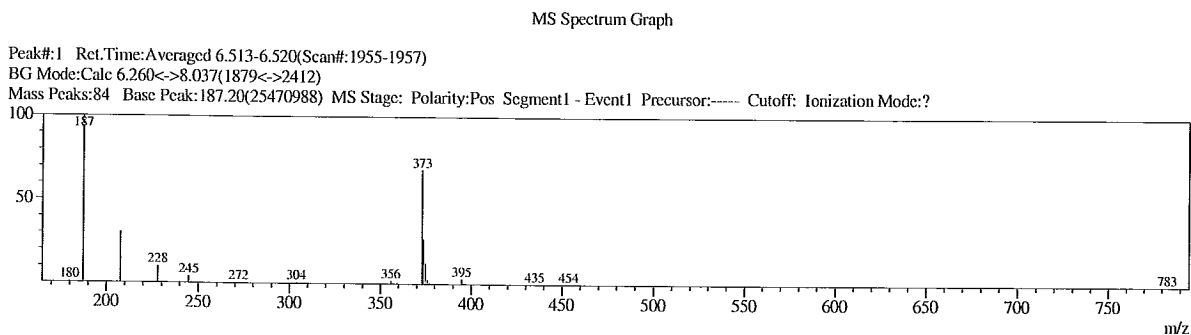
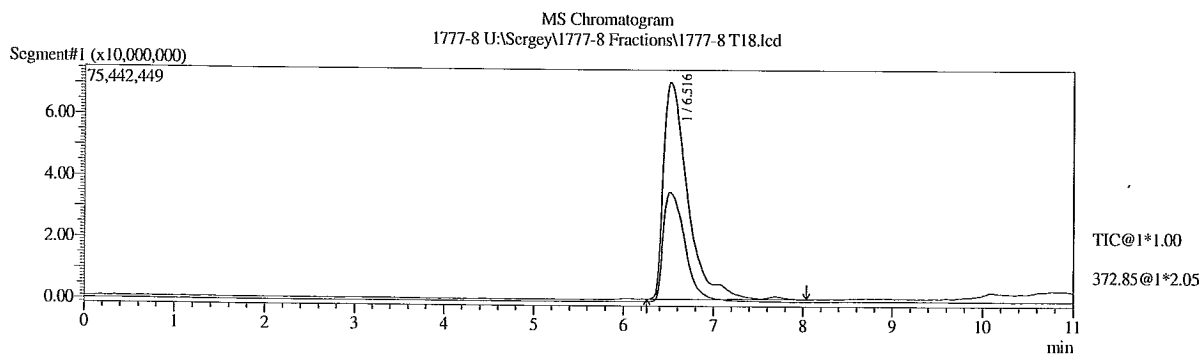
1 Det.A Ch1 / 214nm
 2 Det.A Ch2 / 254nm

PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.475	3490452	387072	100.000	100.000
Total		3490452	387072	100.000	100.000

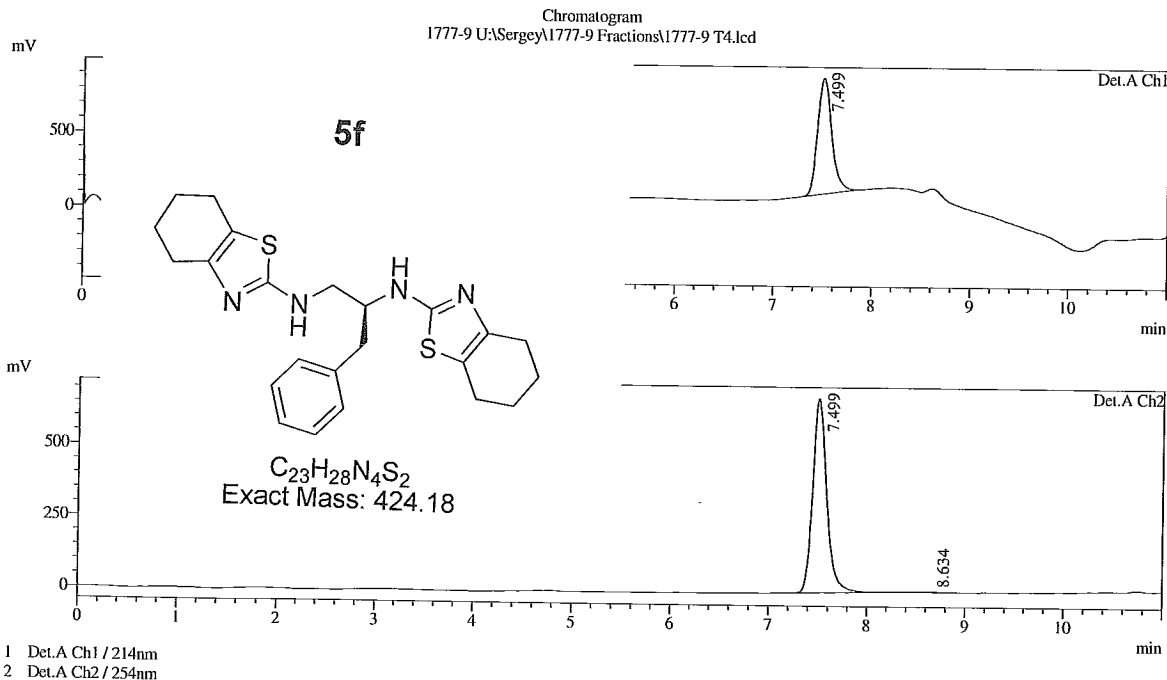
PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.476	3451526	383999	100.000	100.000
Total		3451526	383999	100.000	100.000



Date Acquired : 10/22/2009 4:54:34 PM
 Sample Name : 1777-9
 Sample ID : T4
 Vial # : 99
 Injection Volume : 5
 Data File : 1777-9 T4.lcd
 Method File : 6min 5-95, 1Min Clean(254)_(214) COL1.lcm

Sample Information

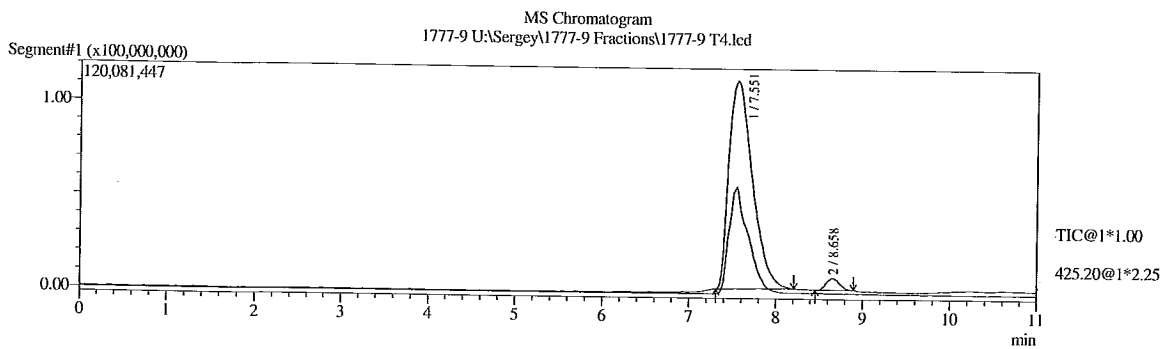


PeakTable
 Detector A Ch1 214nm

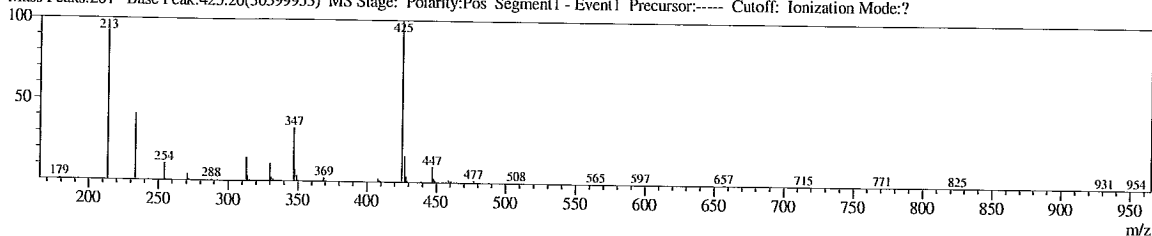
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.499	7750370	779023	100.000	100.000
Total		7750370	779023	100.000	100.000

PeakTable
 Detector A Ch2 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.499	6705539	677755	99.782	99.784
2	8.634	14621	1468	0.218	0.216
Total		6720160	679223	100.000	100.000

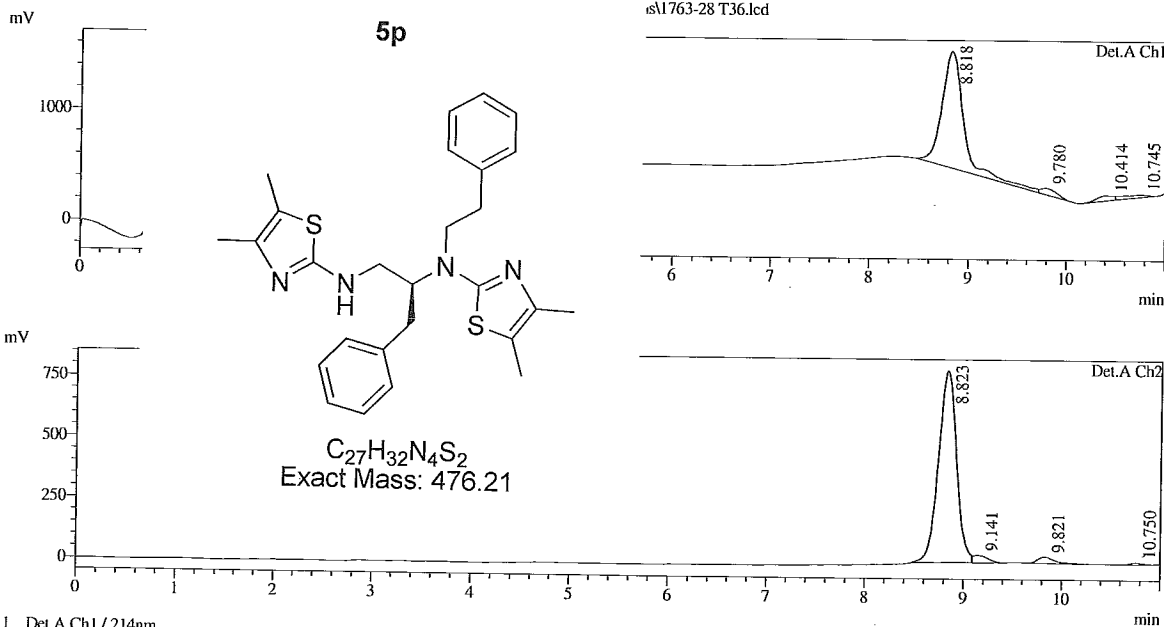


Peak#1 Ret. Time: Averaged 7.547-7.553(Scan#:2265-2267)
 BG Mode: Calc 7.313<->8.207(2195<->2463)
 Mass Peaks:201 Base Peak:425.20(30399953) MS Stage: Polarity:Pos Segment1 - Event1 Precursor:---- Cutoff: Ionization Mode:?



Date Acquired : 10/22/2009 5:44:44 PM
 Sample Name : 1763-28
 Sample ID : T36
 Vial # : 103
 Injection Volume : 5
 Data File : 1763-28 T36.lcd
 Method File : 6min 5-95,1Min Clean(254)_(214) COL1.lcm

Sample Information



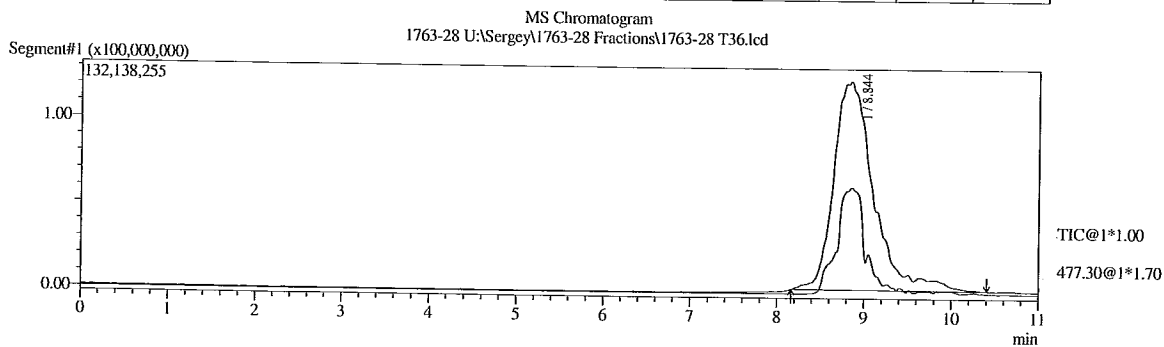
1 Det.A Ch1 / 214nm
 2 Det.A Ch2 / 254nm

PeakTable
 Detector A Ch1 214nm

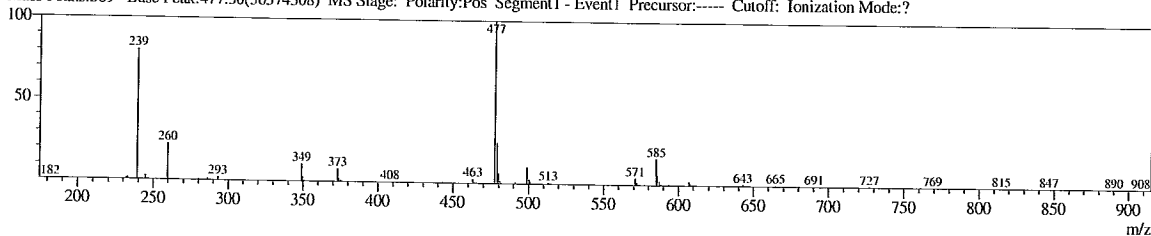
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.818	15947484	1045796	90.295	89.635
2	9.780	642757	54242	3.639	4.649
3	10.414	569634	44157	3.225	3.785
4	10.745	501613	22531	2.840	1.931
Total		17661489	1166727	100.000	100.000

PeakTable
 Detector A Ch2 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.823	10067473	787345	93.370	92.315
2	9.141	335236	32163	3.109	3.771
3	9.821	318461	25697	2.954	3.013
4	10.750	61205	7685	0.568	0.901
Total		10782374	852890	100.000	100.000



Peak#:1 Ret.Time: Averaged 8.840-8.847(Scan#:2653-2655)
 BG Mode: Calc 8.163<->10.403(2450<->3122)
 Mass Peaks: 309 Base Peak: 477.30(36574308) MS Stage: Polarity: Pos Segment1 - Event1 Precursor: ---- Cutoff: Ionization Mode: ?



MS Peak Table TIC

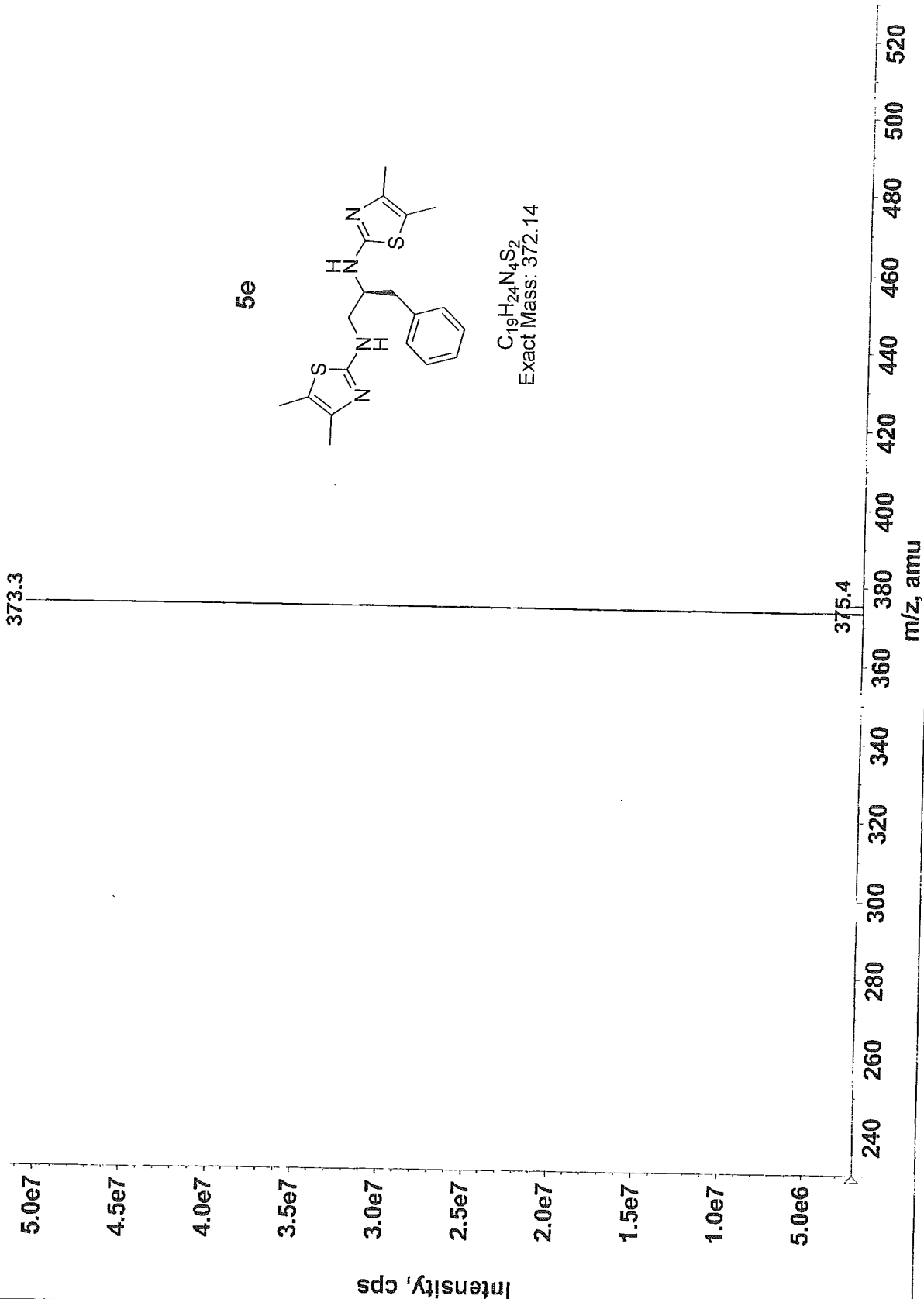
Peak#	Ret. Time	Peak Start	Peak End	Area	Area%	Height	Height%	A/H	Mark	Name	ID#	Event#
1	8.844	8.163	10.403	3864727545	100.00	122596051	100.00	31.52	S			1-1
Total				3864727545	100.00	122596051	100.00					

Printing Date: Tuesday, October 27, 2009
Polarity/Scan Type: Positive Q1 Scan

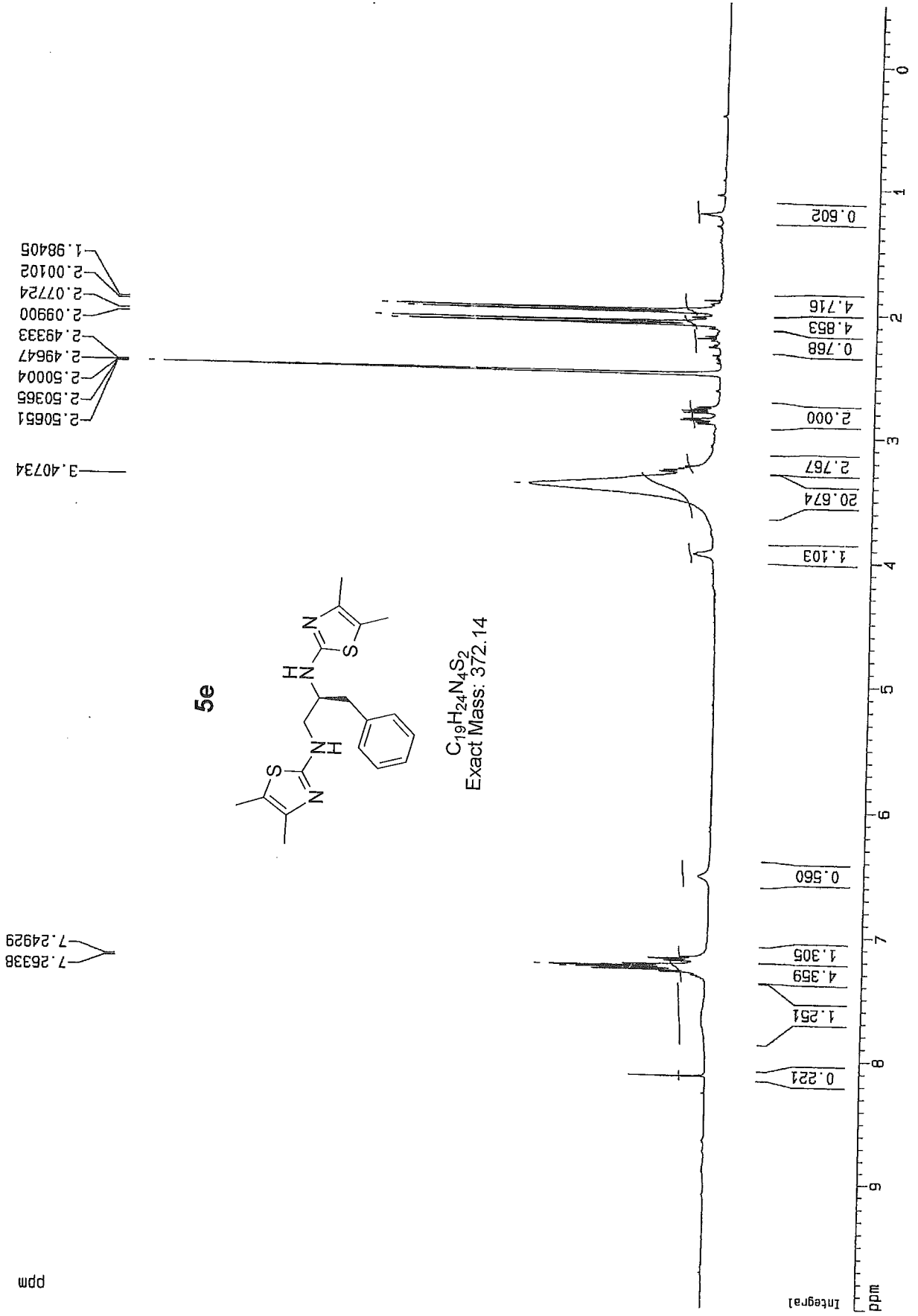
Sample Name: 1777-8 0905TPIM.995A

+Q1: 12 MCA scans from Sample 1 (1777-8 0905TPIM:995A) of TPIM0910....

Max: 5.1e7 cps



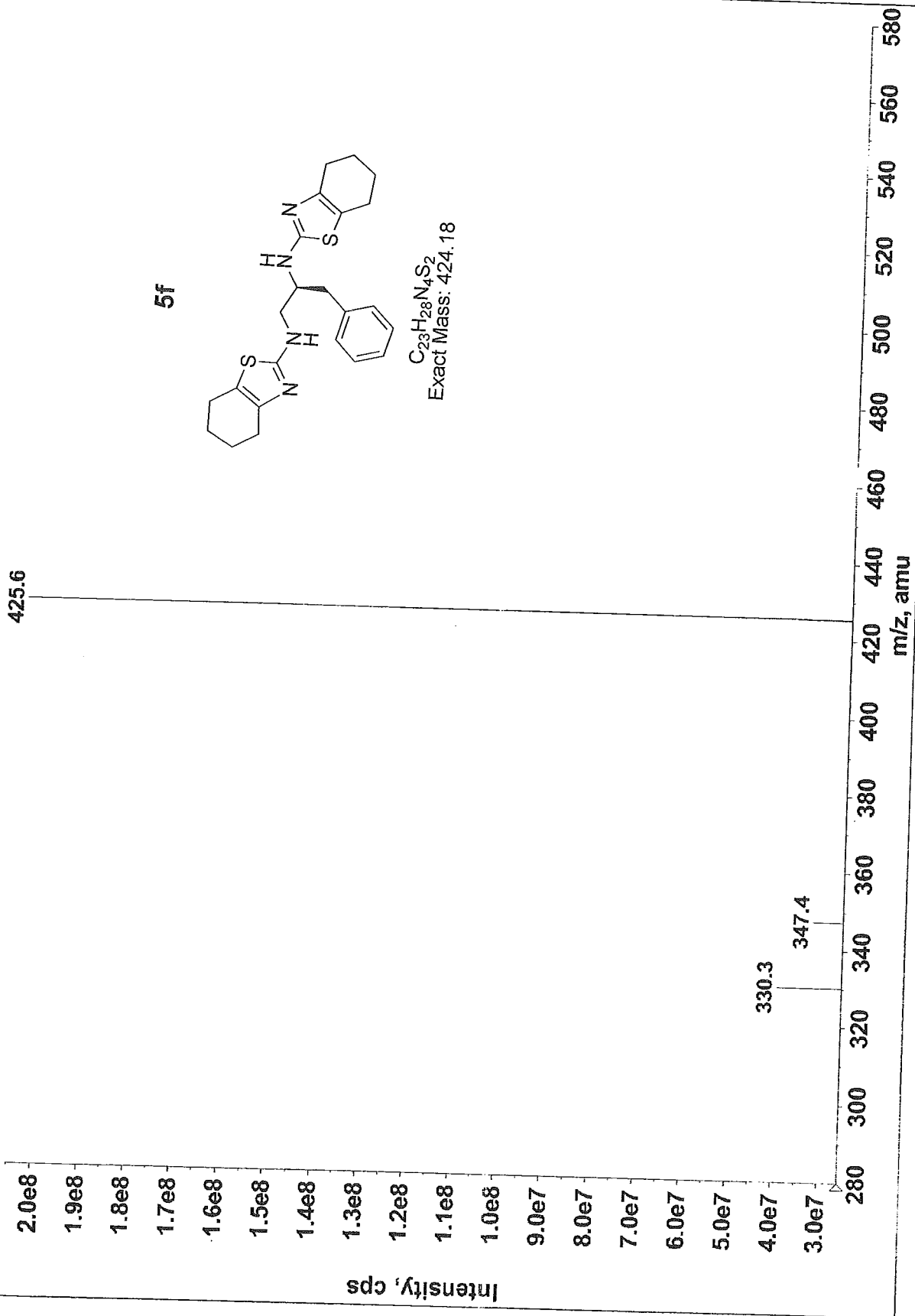
0905TPIM.995A, 1777-8, DMSO, H-1, NUMEGA 10-27-09



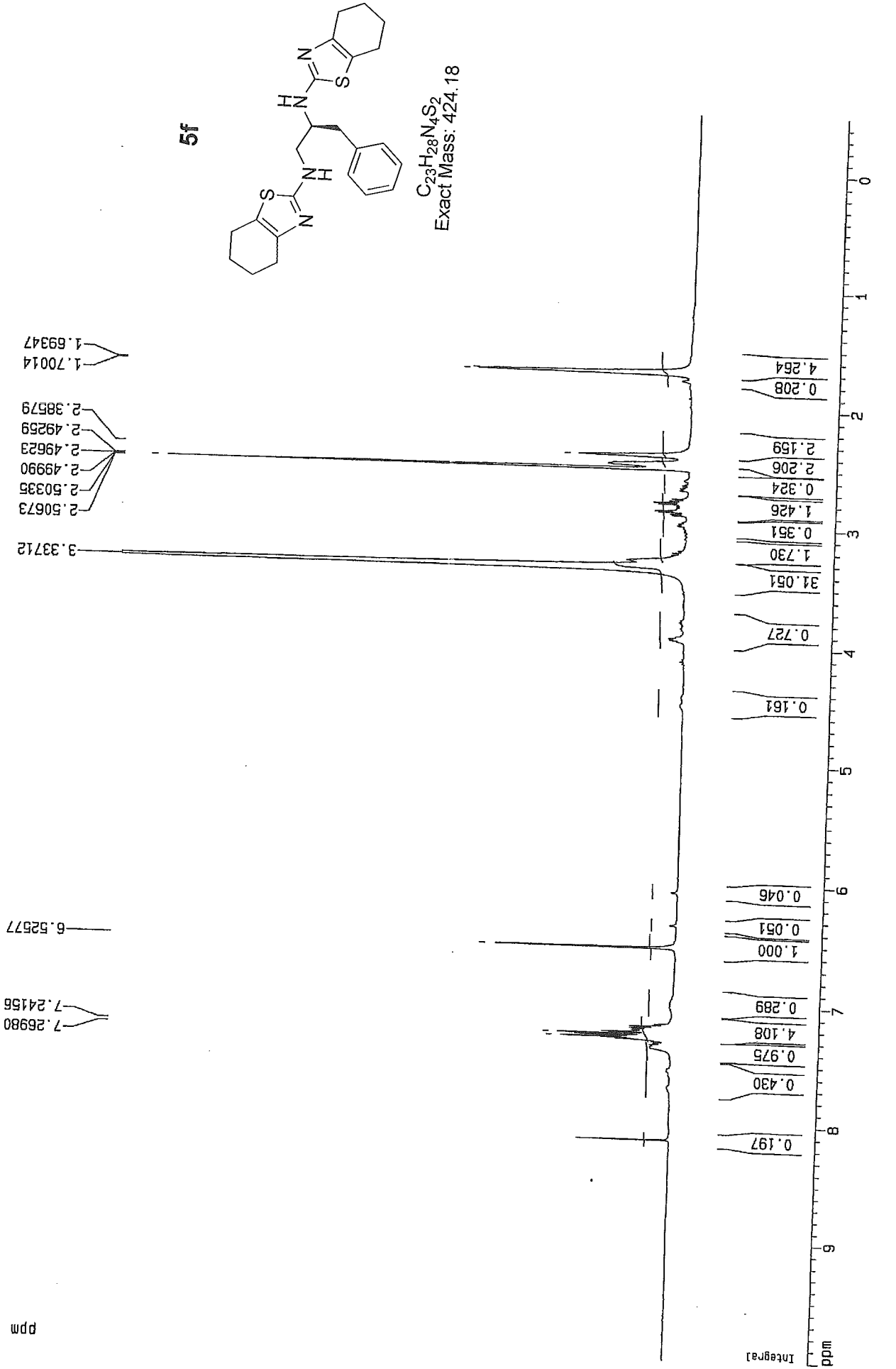
Printing Date: Tuesday, October 27, 2009
Polarity/Scan Type: Positive Q1 Scan

Sample Name: 1777-9 0905TPIM.955B

+Q1 12 MCA scans from Sample 3 (1777-9 0905TPIM.955B) of TPIM0910.... Max 2.0e8 cps.



0905TPIM.995B, 1777-9, DMSO, H-1, NUMEGA 10-27-09

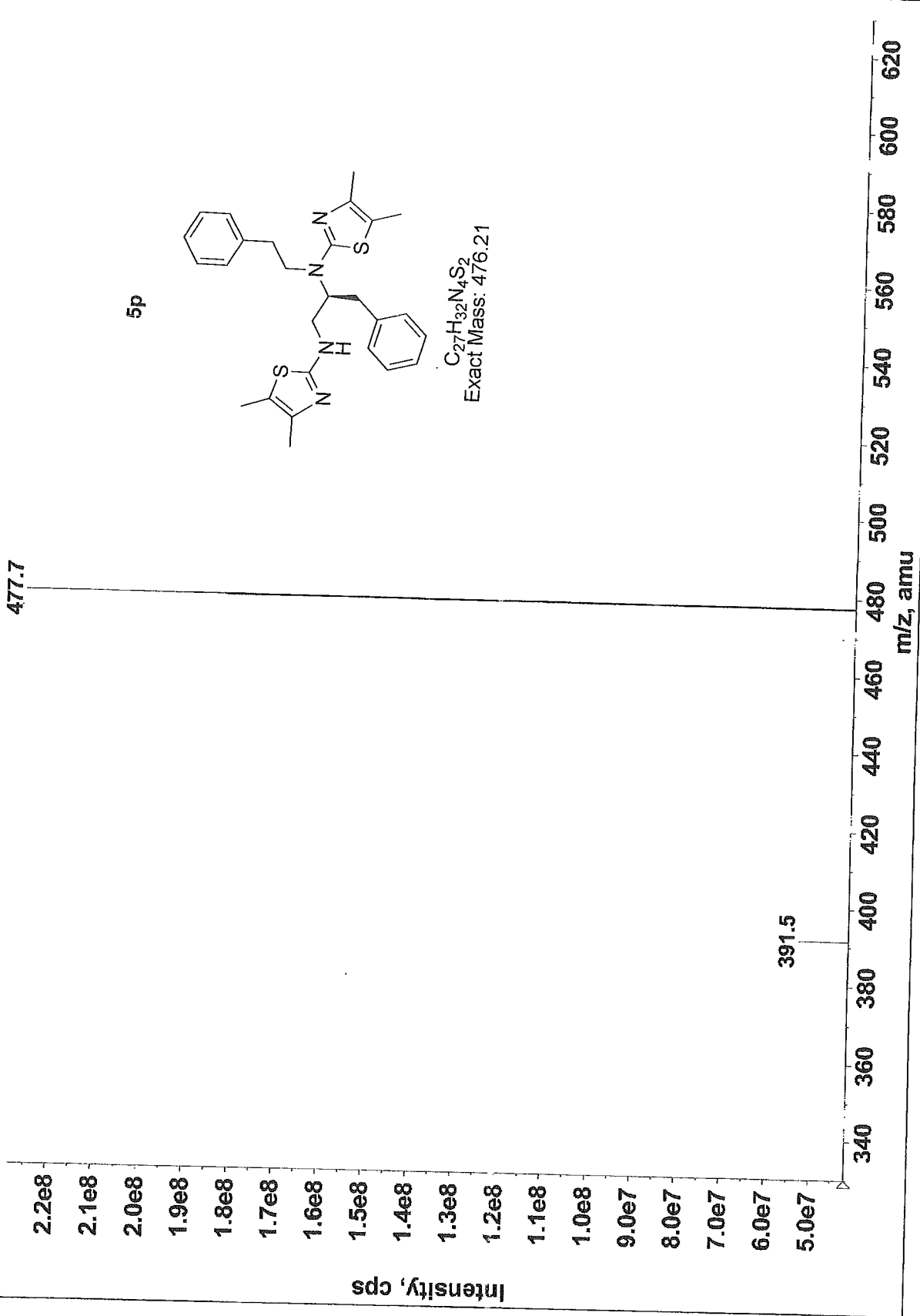


RECEIVED 10/30/2009 19:27

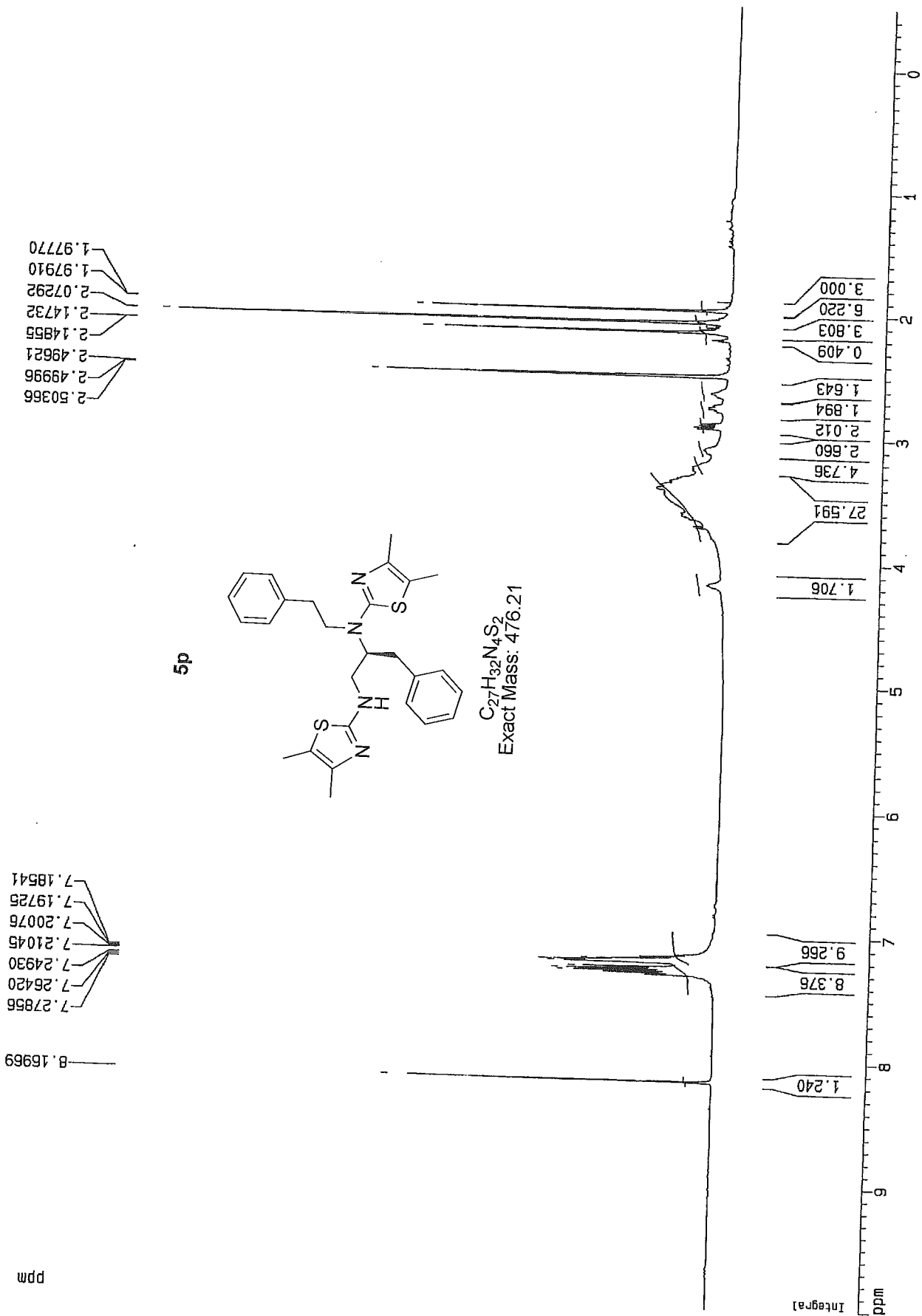
Printing Date: Tuesday, October 27, 2009
Polarity/Scan Type: Positive Q1 Scan

Sample Name: 1763-28

+Q1: 12 MCA scans from Sample 5 (1763-28: 0905TPIM995C) of TPIM0910... Max: 2.3e8 cps



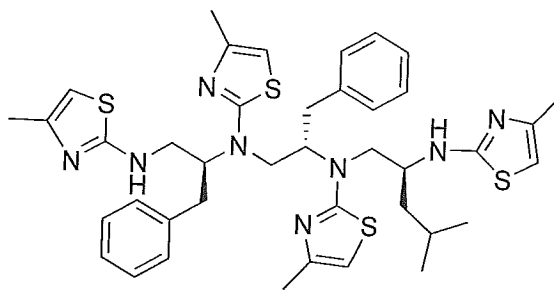
0905TPIM.995C, 1763-28, DMSO, H-1, NUMEGA 10-27-09



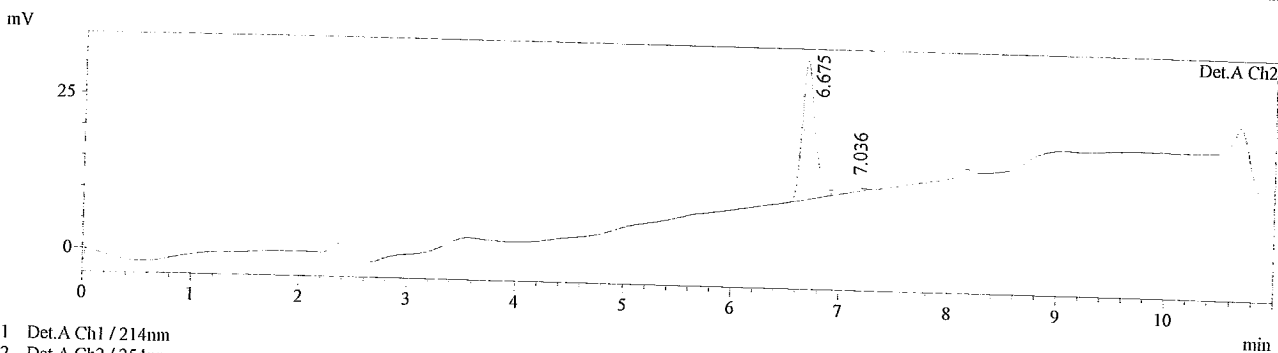
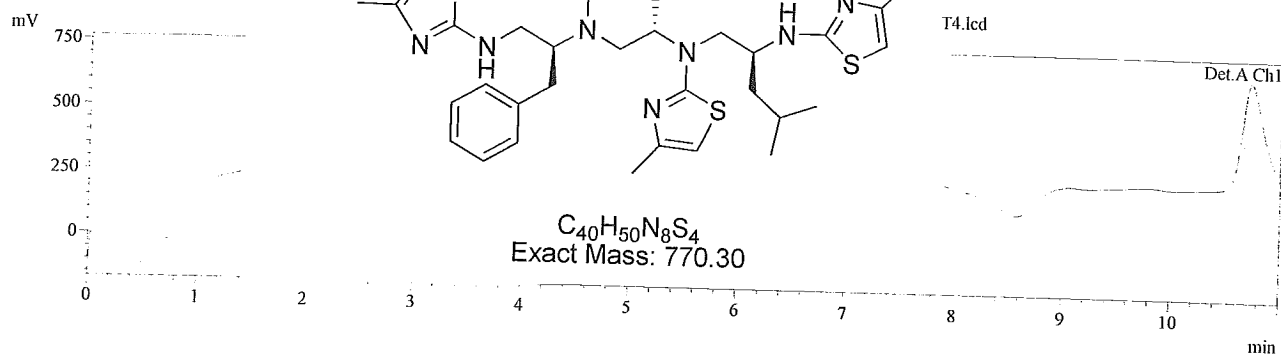
RECEIVED 10/30/2009 19:27

Date Acquired : 12/3/2
 Sample Name : PT-1 F
 Sample ID : T4
 Vial # : 13
 Injection Volume : 5
 Data File : PT-1 F
 Method File : 6min 5

PT-1



$C_{40}H_{50}N_8S_4$
 Exact Mass: 770.30



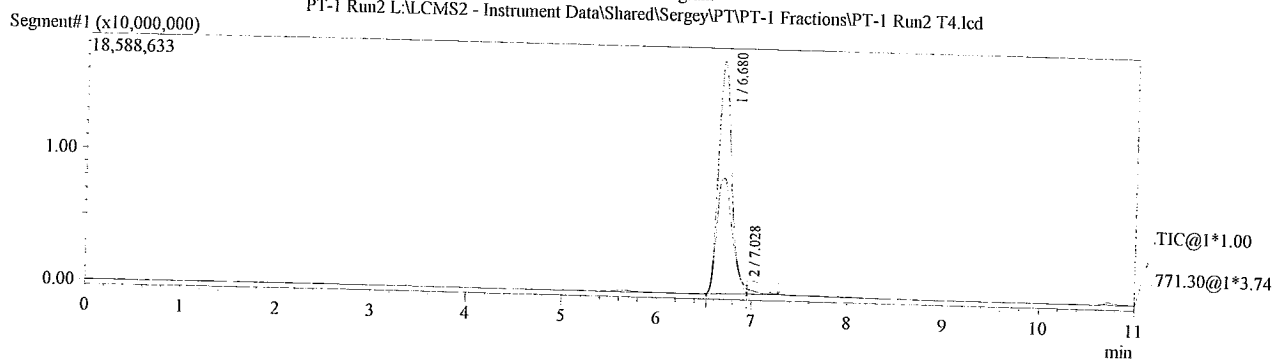
1 Det.A Ch1 / 214nm
 2 Det.A Ch2 / 254nm

PeakTable

Detector A Ch2 254nm					
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.675	187937	22166	89.187	91.122
2	7.036	22785	2160	10.813	8.878
Total		210722	24326	100.000	100.000

MS Chromatogram

PT-1 Run2 LALCMS2 - Instrument Data\Shared\Sergey\PT\PT-1 Fractions\PT-1 Run2 T4.lcd

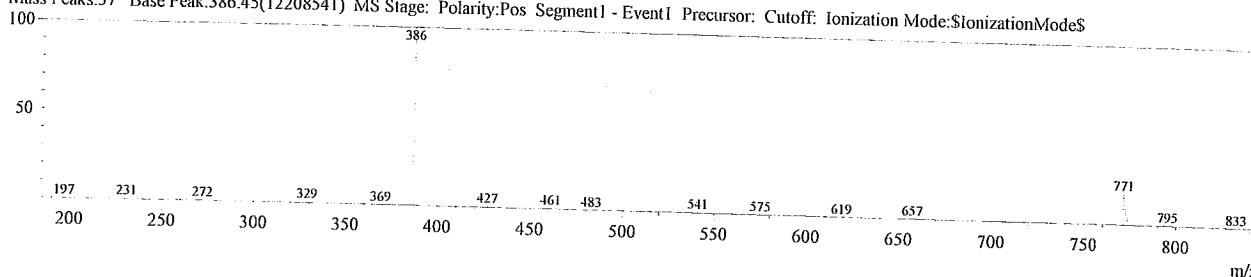


MS Spectrum Graph

Peak#:1 Ret.Time:Averaged 6.677-6.683(Scan#:2004-2006)

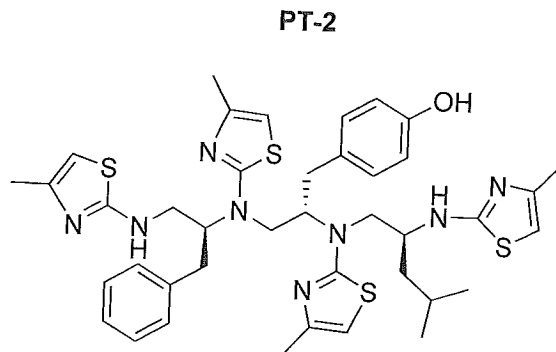
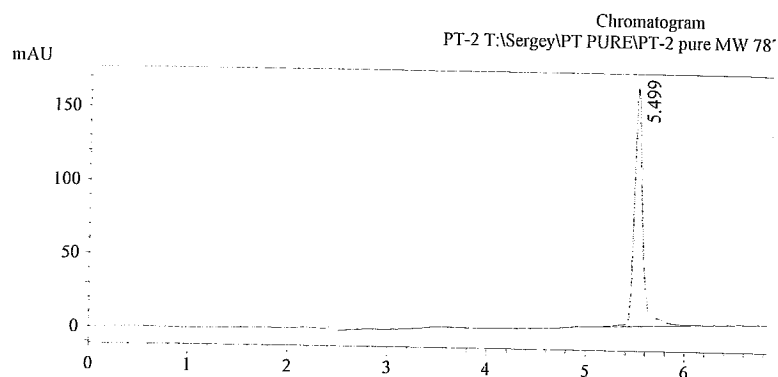
BG Mode:Calc 6.527<->6.950(1959<->2086)

Mass Peaks:57 Base Peak:386.45(12208541) MS Stage: Polarity:Pos Segment1 - Event1 Precursor: Cutoff: Ionization Mode:\$ionizationMode\$



Date Acquired : 12/4/2009 12:40:31 PM
 Sample Name : PT-2
 Sample ID : Pure MW 787 pooled002
 Injection Volume : 5
 Data File : PT-2 pure MW 787 pooled.lcd
 Method File : 6 min 5-95 D2only, 2Min Clean_05NOV09.1cm

Sample Information

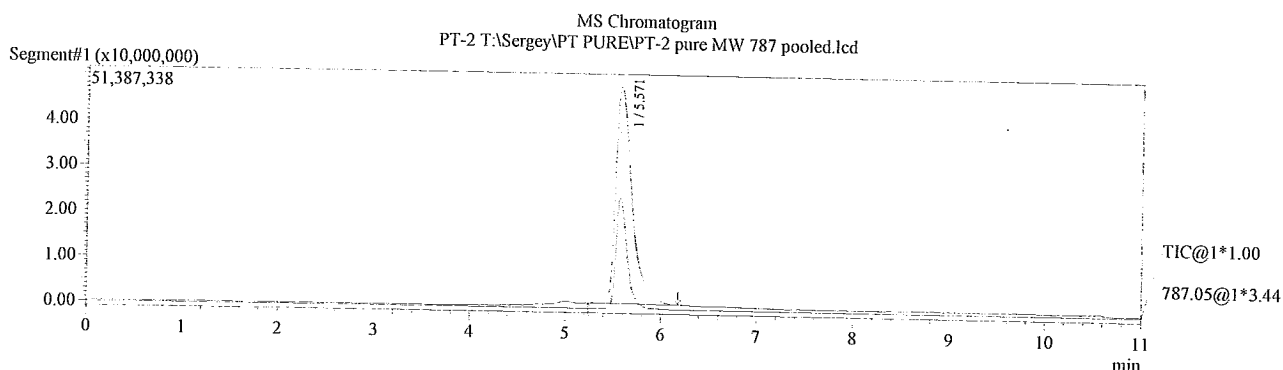


$C_{40}H_{50}N_8OS_4$
 Exact Mass: 786.30

1 PDA Multi 1 / 254nm 4nm

PDA Ch1 254nm 4nm PeakTable

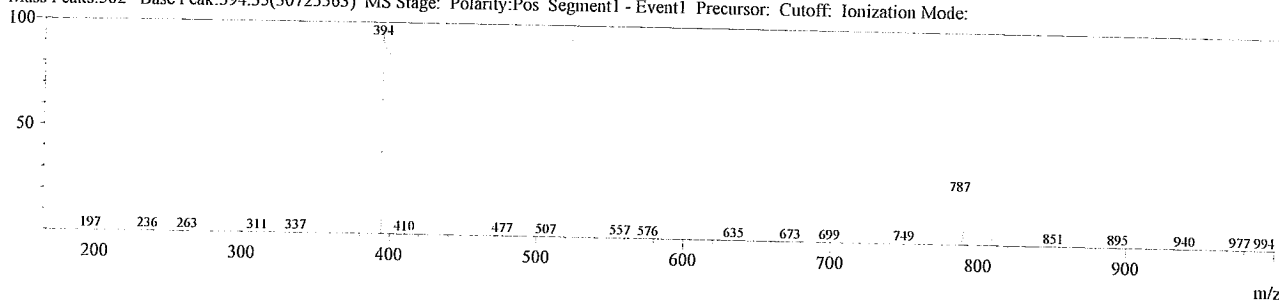
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.499	937440	161306	100.000	100.000
Total		937440	161306	100.000	100.000



MS Spectrum Graph

Peak#:1 Ret.Time:Averaged 5.567-5.573(Scan#:1671-1673)
 BG Mode:Calc 5.240<->6.180(1573<->1855)

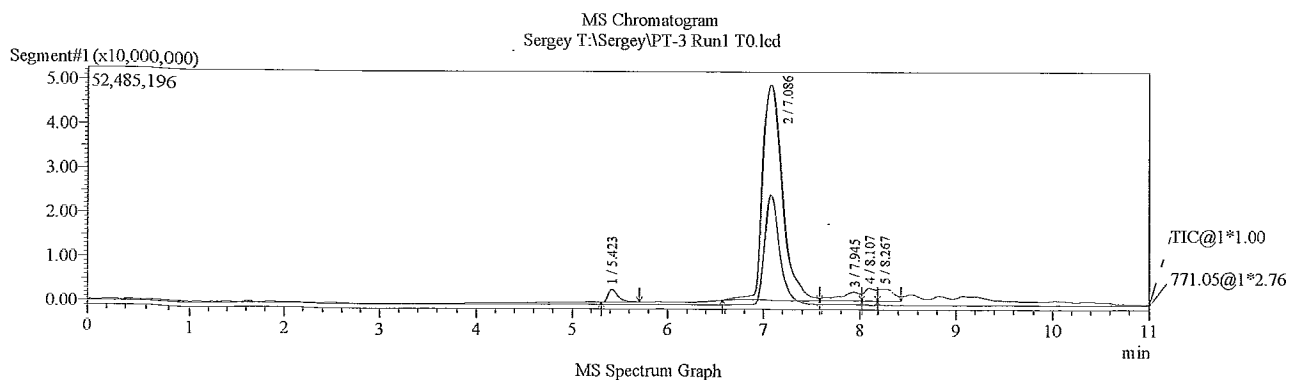
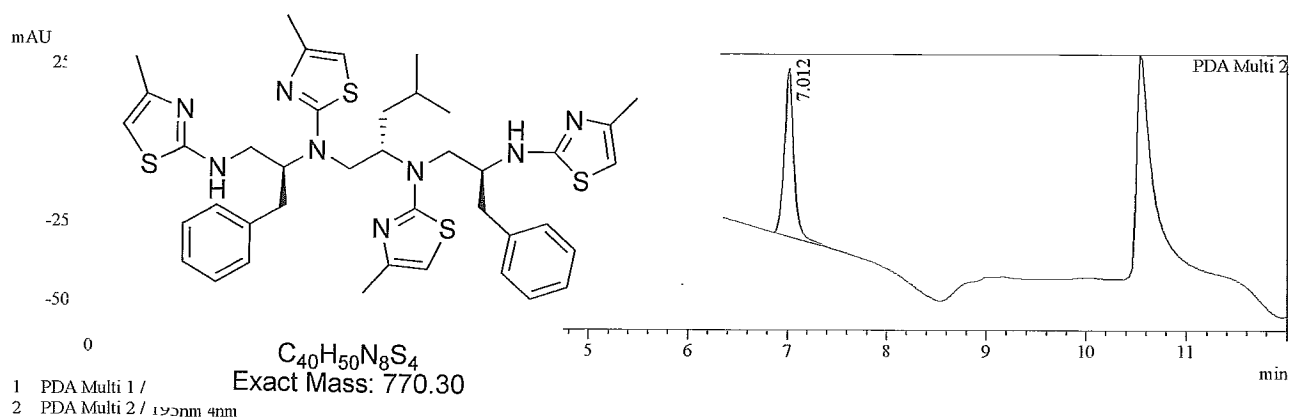
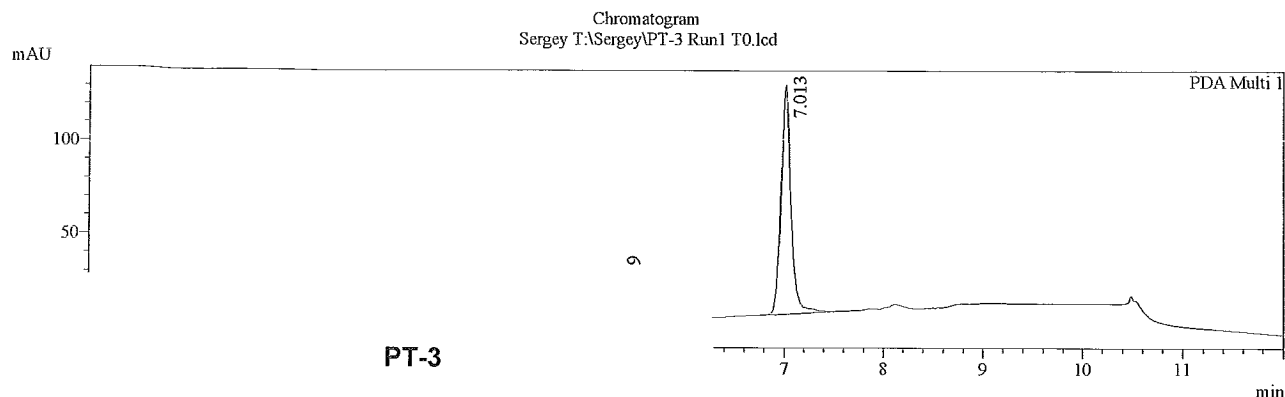
Mass Peaks:502 Base Peak:394.35(30725563) MS Stage: Polarity:Pos Segment1 - Event1 Precursor: Cutoff: Ionization Mode:



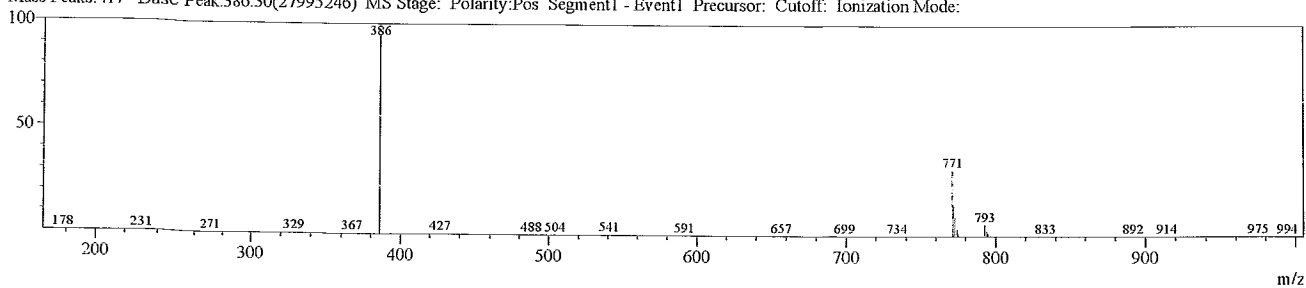
MS Peak Table TIC

Peak#	Ret.Time	Peak Start	Peak End	Area	Area%	Height	Height%	A/H	Mark	Name	ID#	Event#
1	5.571	5.240	6.180	566144061	100.00	47507950	100.00	11.91				
Total				566144061	100.00	47507950	100.00					1-1

Sample Information
 Date Acquired : 12/2/2009 5:54:51 PM
 Sample Name : Sergey
 Sample ID : PT-3Run1 T0
 Injection Volume : 5
 Data File : PT-3 Run1 T0.lcd
 Method File : 6 min 5-95 D2only, 2Min Clean_05NOV09.lcm

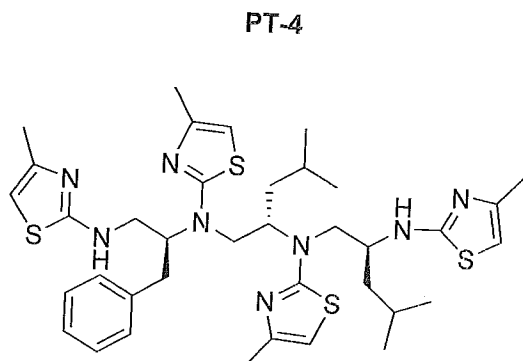


Peak#: 2 Ret. Time: Averaged 7.083-7.090(Scan#:2126-2128)
 BG Mode: Calc 6.573<->7.583(1973<->2276)
 Mass Peaks: 417 Base Peak: 386.30(27995246) MS Stage: Polarity: Pos Segment1 - Event1 Precursor: Cutoff: Ionization Mode:

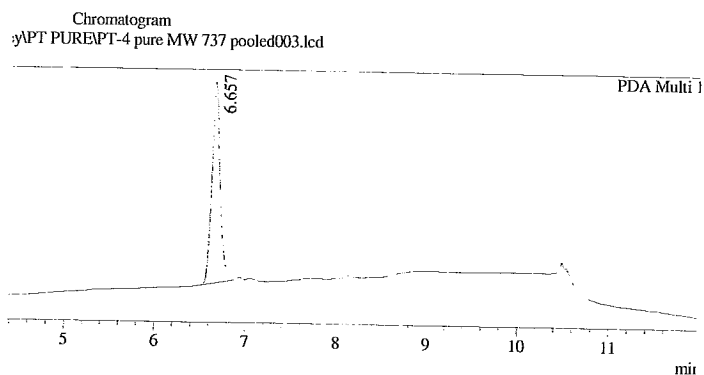


Date Acquired : 12/4/2009 12:53:04 PM
 Sample Name : PT-4
 Sample ID : Pure MW 737 pooled003
 Injection Volume : 5
 Data File : PT-4 pure MW 737 pooled003.lcd
 Method File : 6 min 5-95 D2only, 2Min Clean_05NOV09.lcm

Sample Information

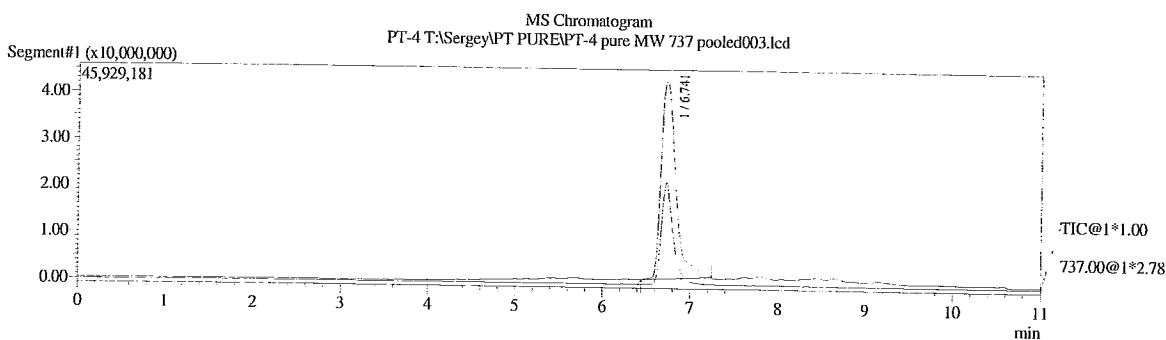


$C_{37}H_{52}N_8S_4$
 Exact Mass: 736.32



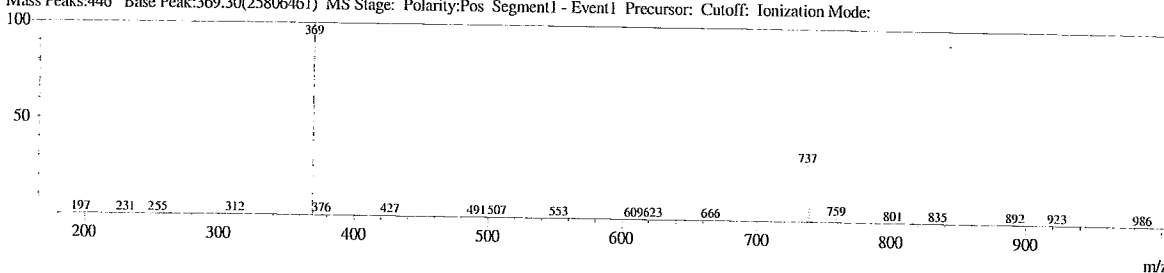
PeakTable

Area	Height	Area %	Height %
504738	79873	100.000	100.000
504738	79873	100.000	100.000



MS Spectrum Graph

Peak#:1 Ret.Time:Averaged 6.737-6.743(Scan#:2022-2024)
 BG Mode:Calc 6.440<->7.240(1933<->2173)
 Mass Peaks:446 Base Peak:369.30(25806461) MS Stage: Polarity:Pos Segment1 - Event1 Precursor: Cutoff: Ionization Mode:



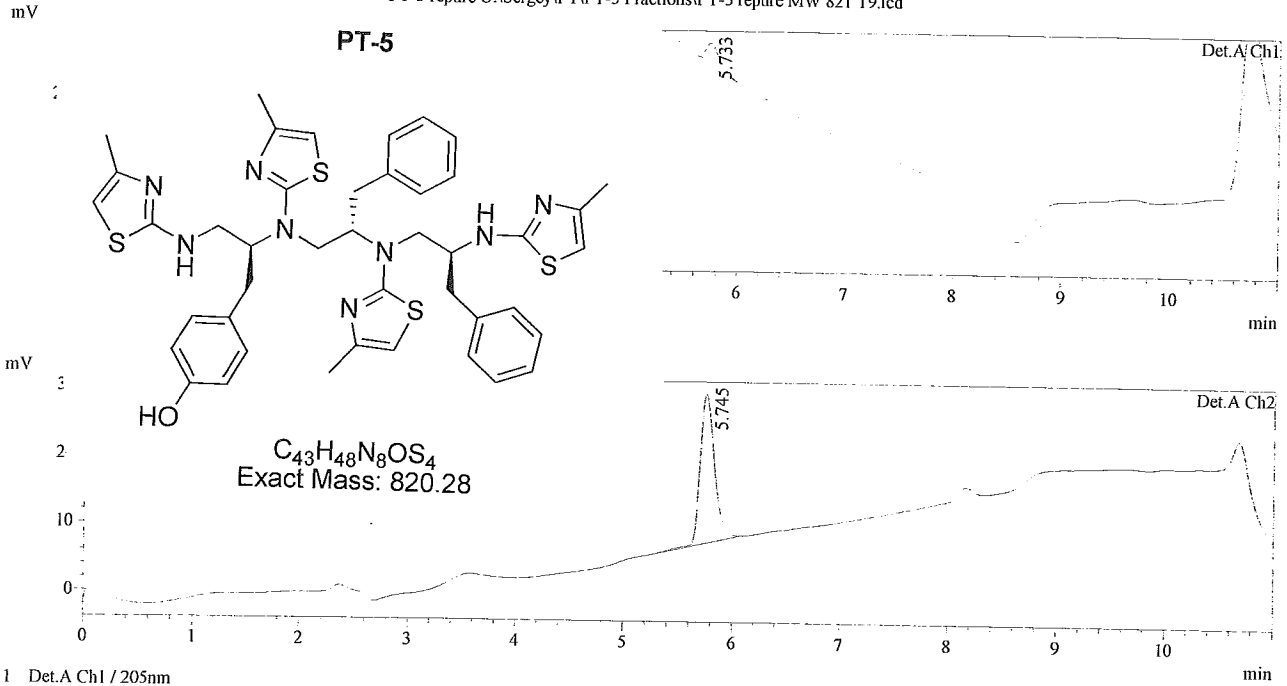
MS Peak Table TIC

Peak#	Ret.Time	Peak Start	Peak End	Area	Area%	Height	Height%	A/H	Mark	Name	ID#	Event#
1	6.741	6.440	7.240	494329917	100.00	40725889	100.00	12.13				
Total				494329917	100.00	40725889	100.00					1-1

Date Acquired : 12/15/2009 2:13:54 PM
 Sample Name : PT-5 repure
 Sample ID : MW 821 T9
 Vial # : 94
 Injection Volume : 5
 Data File : PT-5 repure MW 821 T9.lcd
 Method File : 6min 5-95,1Min Clean(254)_(205) COL1.lcm

Sample Information

Chromatogram
 PT-5 repure U:\Sergey\PT\PT-5 Fractions\PT-5 repure MW 821 T9.lcd



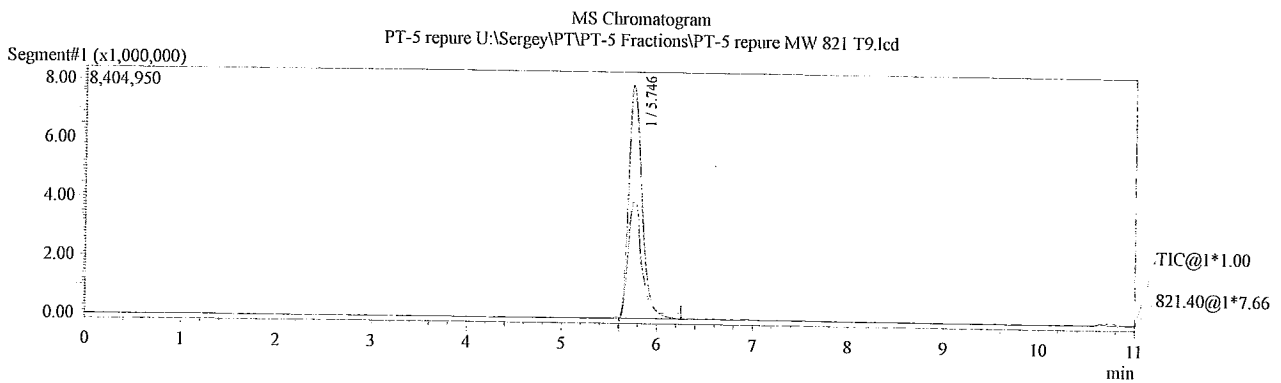
1 Det.A Ch1 / 205nm
 2 Det.A Ch2 / 254nm

PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.733	366314	43688	100.000	100.000
Total		366314	43688	100.000	100.000

PeakTable

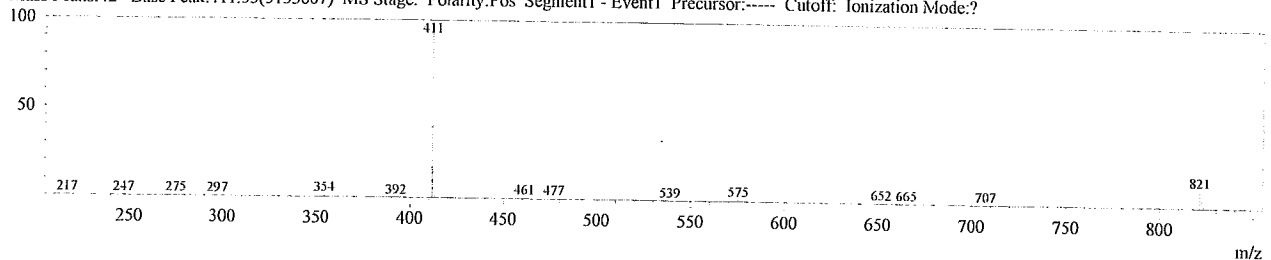
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.745	198366	21872	100.000	100.000
Total		198366	21872	100.000	100.000



MS Spectrum Graph

Peak#:1 Ret.Time:Averaged 5.743-5.750(Scan#:1724-1726)
 BG Mode:Calc 5.607<->6.253(1683<->1877)

Mass Peaks:42 Base Peak:411.55(5153607) MS Stage: Polarity:Pos Segment1 - Event1 Precursor:---- Cutoff: Ionization Mode:?

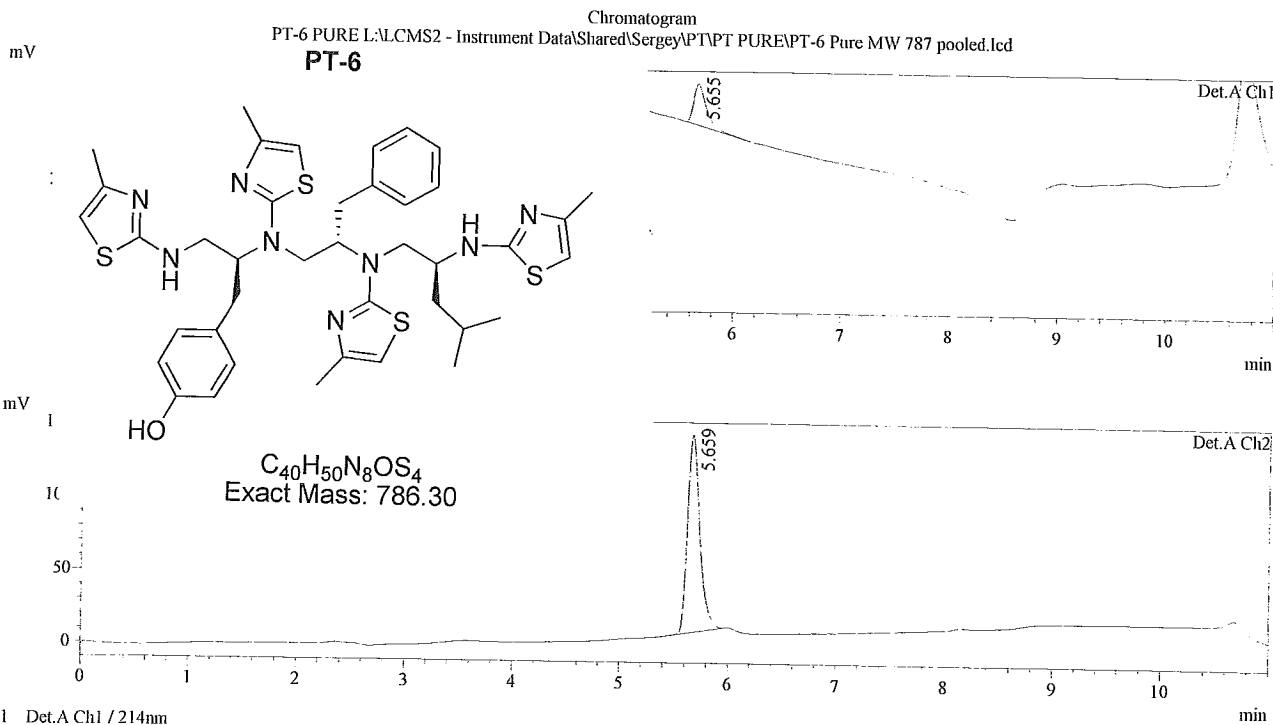


MS Peak Table TIC

Peak#	Ret.Time	Peak Start	Peak End	Area	Area%	Height	Height%	A/H	Mark	Name	ID#	Event#
1	5.746	5.607	6.253	76234559	100.00	7937463	100.00	9.60	SV			
Total				76234559	100.00	7937463	100.00					

Date Acquired : 12/4/2009 4:12:04 PM
 Sample Name : PT-6 PURE
 Sample ID : MW 787 pooled002
 Vial # : 12
 Injection Volume : 5
 Data File : PT-6 Pure MW 787 pooled.lcd
 Method File : 6min 5-95,1Min Clean(254)_ (214) COL1.lcm

Sample Information

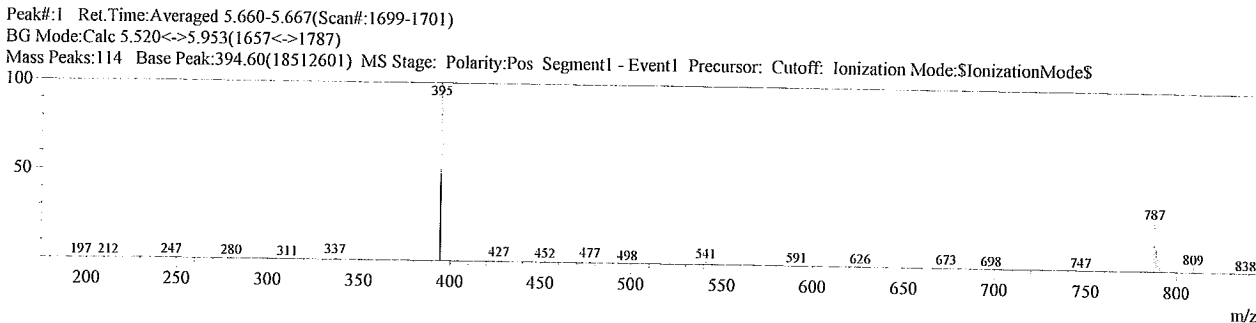
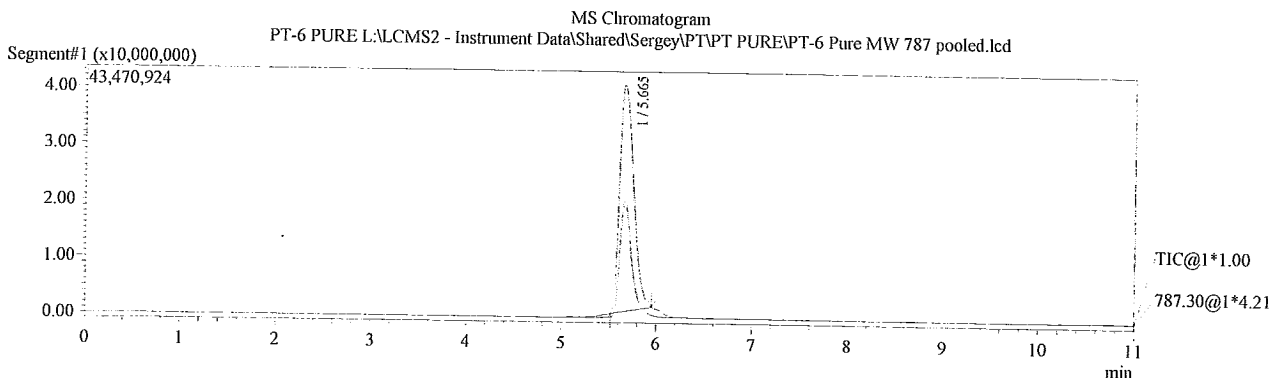


Detector A Ch1 214nm PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.655	1048245	130371	100.000	100.000
Total		1048245	130371	100.000	100.000

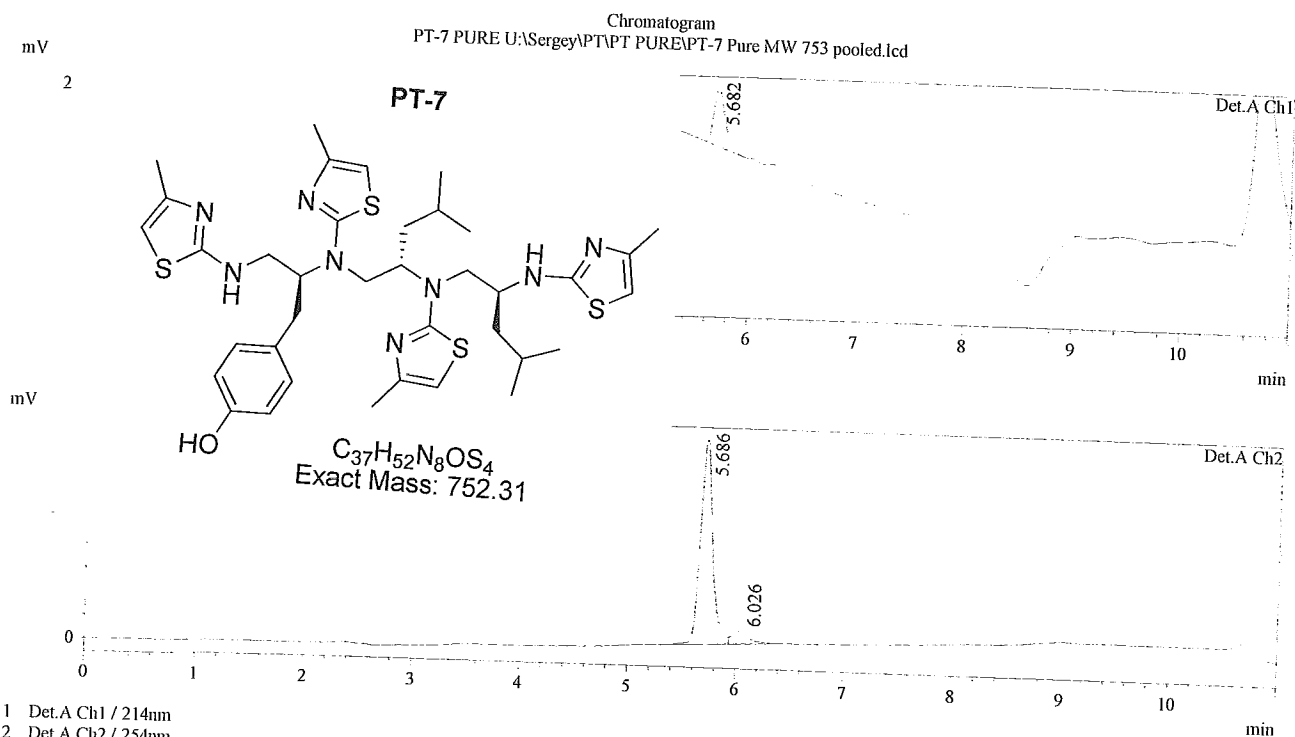
Detector A Ch2 254nm PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.659	1054009	134735	100.000	100.000
Total		1054009	134735	100.000	100.000



Date Acquired : 12/4/2009 3:12:05 PM
 Sample Name : PT-7 PURE
 Sample ID : MW 753 pooled
 Vial # : 10
 Injection Volume : 5
 Data File : PT-7 Pure MW 753 pooled.lcd
 Method File : 6min 5-95,1Min Clean(254)_(214) COL1.lcm

Sample Information



PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.682	917404	122940	100.000	100.000
Total		917404	122940	100.000	100.000

PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.686	1375537	172387	92.431	94.230
2	6.026	112633	10557	7.569	5.770
Total		1488170	182944	100.000	100.000

