

Supporting Information for:

Identification of Neuropeptide S Antagonists: Structure-Activity Relationship Studies, X-Ray Crystallography, and In vivo Evaluation

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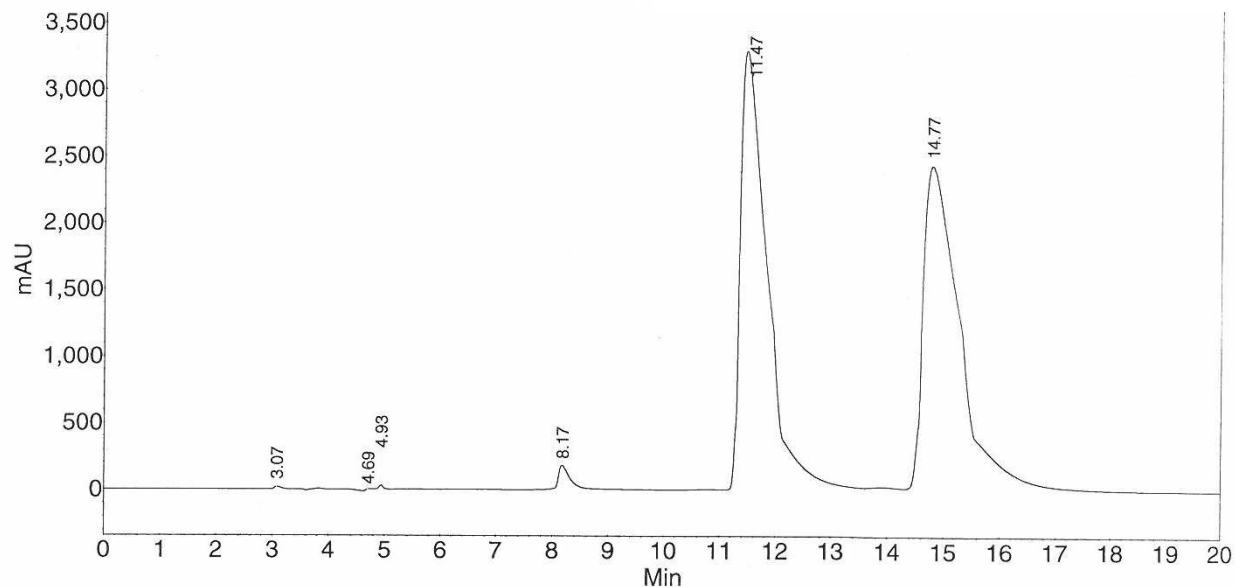
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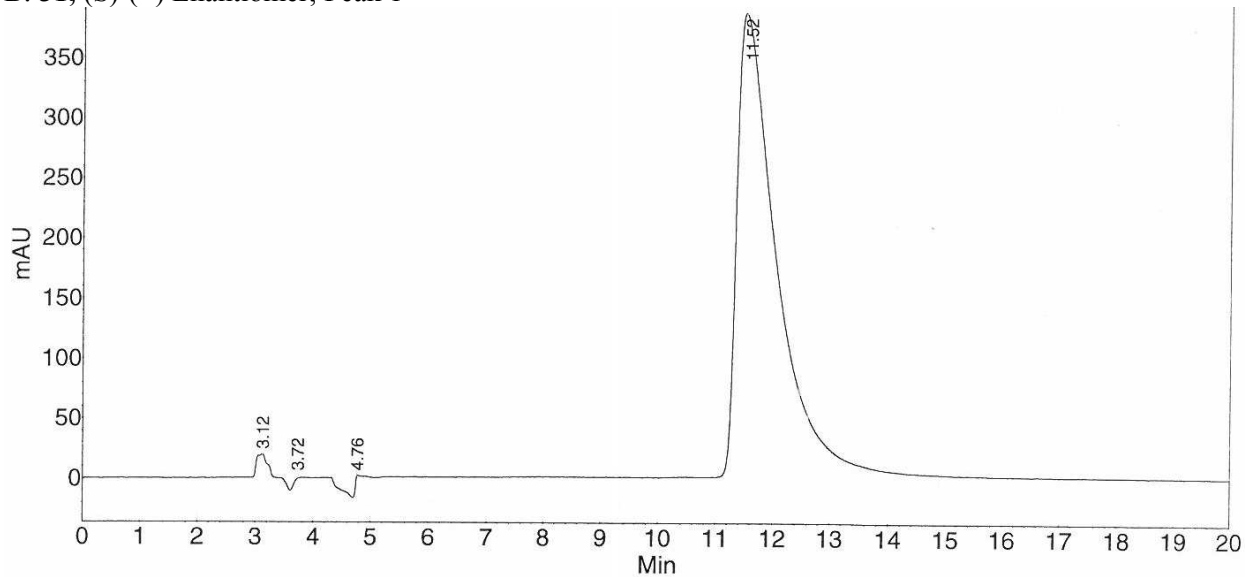
A: SHA-68 (1) Racemate

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	3.07	0.05	16.4	1.8	0.052
2	UNKNOWN	4.69	0.04	11.6	1.3	0.038
3	UNKNOWN	4.93	0.11	34.5	3.6	0.106
4	UNKNOWN	8.17	1.00	170.8	34.3	1.002
5	UNKNOWN	11.47	50.14	3273.7	1717.4	50.140
6	UNKNOWN	14.77	48.66	2398.1	1666.8	48.662
Total			100.00	5905.2	3425.3	100.000



B: 31, (S)-(-) Enantiomer; Peak 1



C: **32**; (R)-(+ Enantiomer; Peak 2

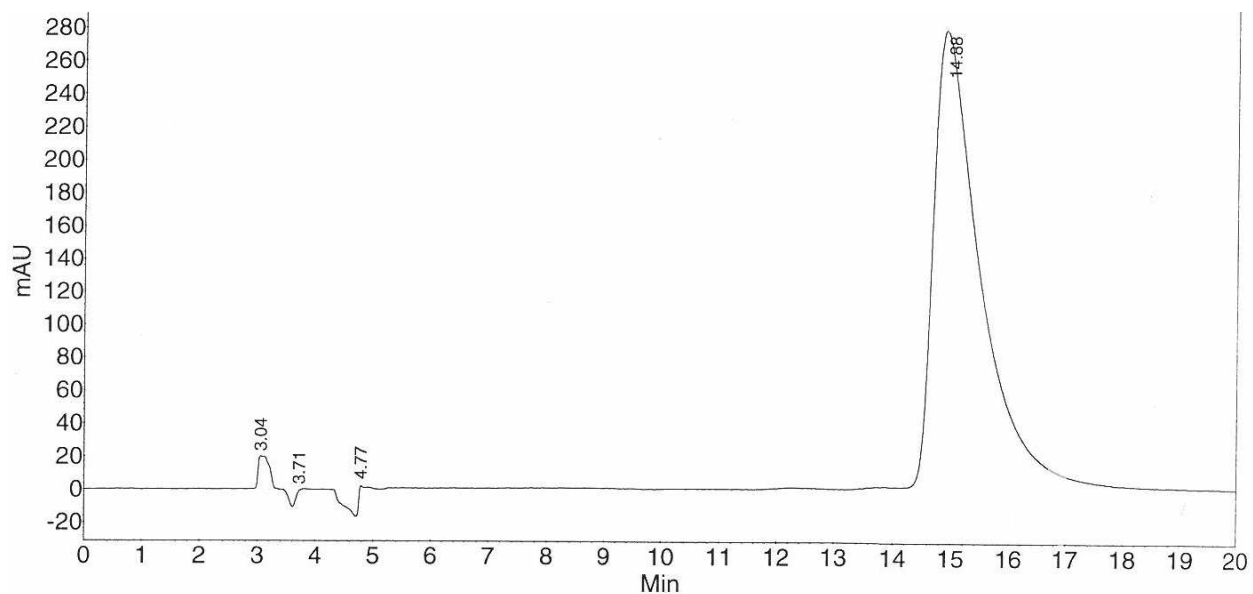
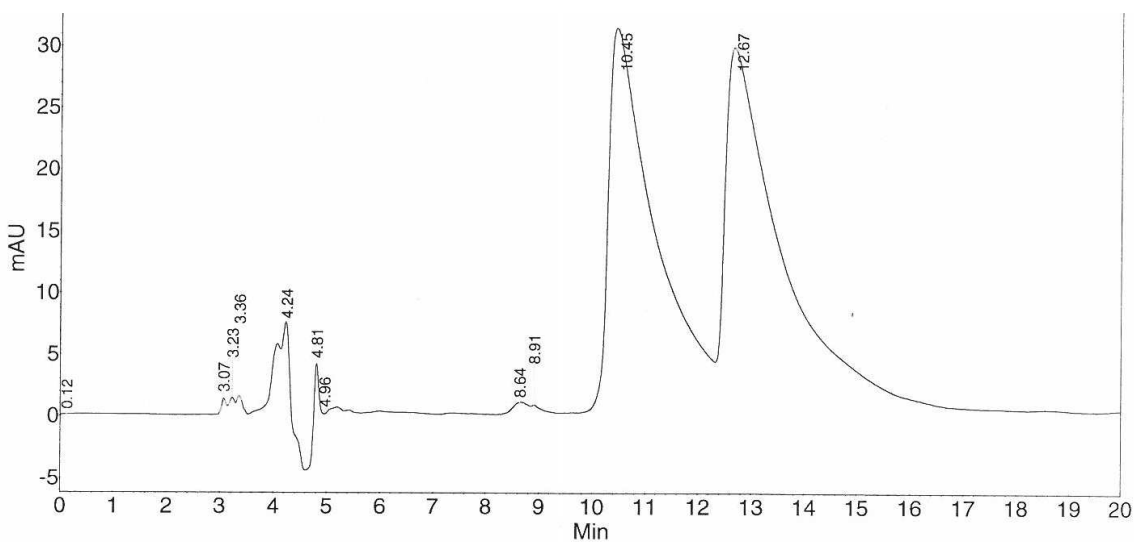


Figure 5: HPLC Chromatograms of SHA-68 (**1**) (A), **31** (S)-(-) Enantiomer; Peak 1 (B), and **32** (R)-(+ Enantiomer; Peak 2 (C) on chiral column (NP; Chiralpak IA, IPA/hexanes 1/9) recorded at 204 nm)

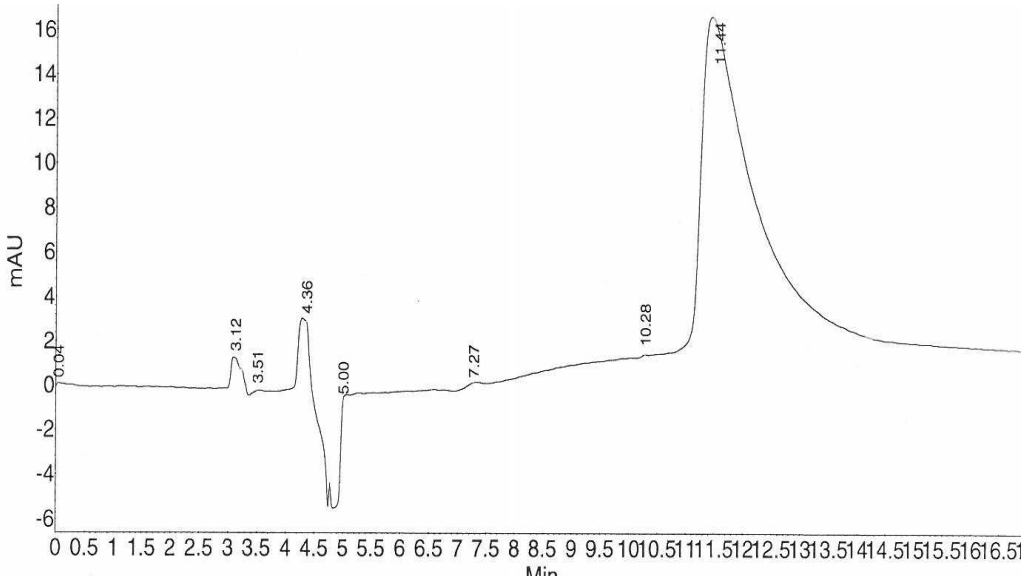
A: RTI-118 (2) Racemate

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	0.12	0.00	0.0	0.0	0.004
2	UNKNOWN	3.07	0.20	1.3	0.1	0.202
3	UNKNOWN	3.23	0.25	1.4	0.2	0.248
4	UNKNOWN	3.36	0.30	1.5	0.2	0.298
5	UNKNOWN	4.24	0.90	5.1	0.6	0.899
6	UNKNOWN	4.81	1.56	7.5	1.0	1.557
7	UNKNOWN	4.96	0.85	2.4	0.6	0.846
8	UNKNOWN	8.64	0.35	0.8	0.2	0.350
9	UNKNOWN	8.91	0.09	0.4	0.1	0.087
10	UNKNOWN	10.45	46.67	30.9	30.5	46.667
11	UNKNOWN	12.67	48.84	28.7	31.9	48.843
Total			100.00	80.0	65.4	100.000



B: 33, (S)(-) Enantiomer; Peak 1



C: **34**; (R)-(+ Enantiomer; Peak 2

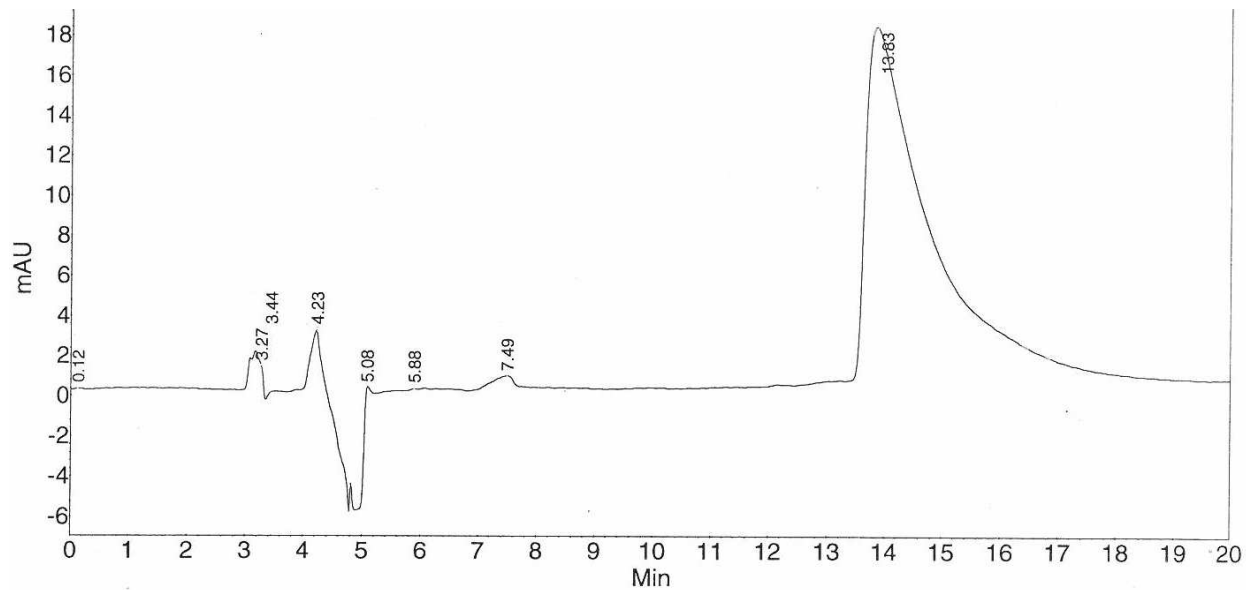
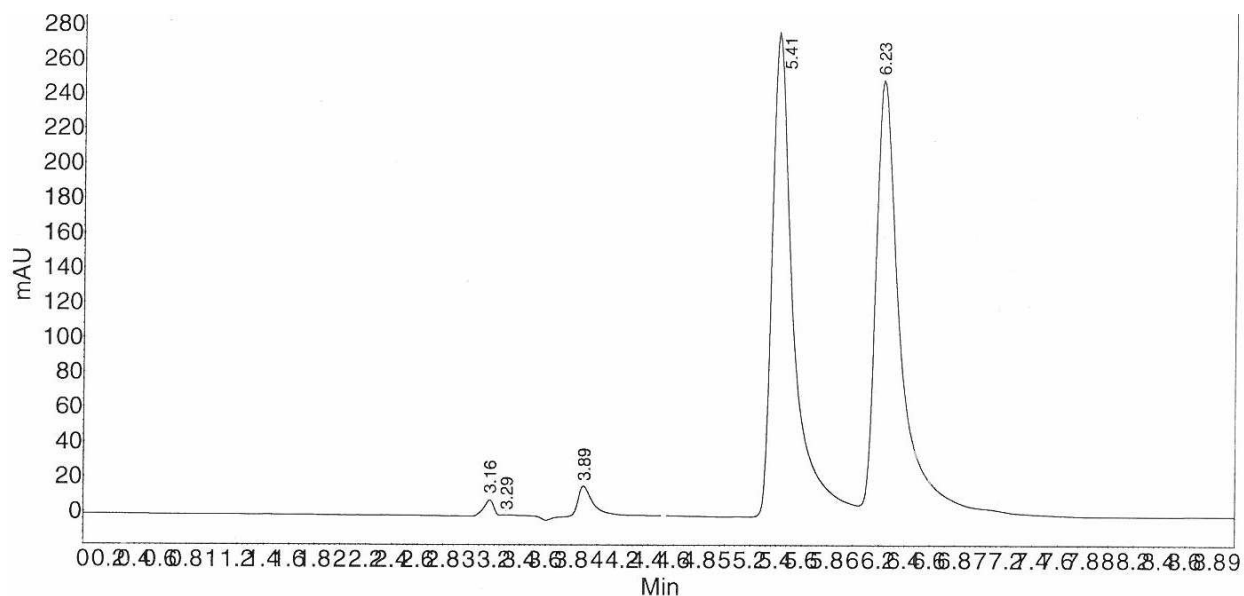


Figure 6: HPLC Chromatograms of RTI-118 (**2**) (A), **33** (S)-(-) Enantiomer; Peak 1 (B), and **34** (R)-(+ Enantiomer; Peak 2 (C) on chiral column (NP; Chiralpak IA, hexanes/IPA/DEA 9/1/0.1) recorded at 258 nm)

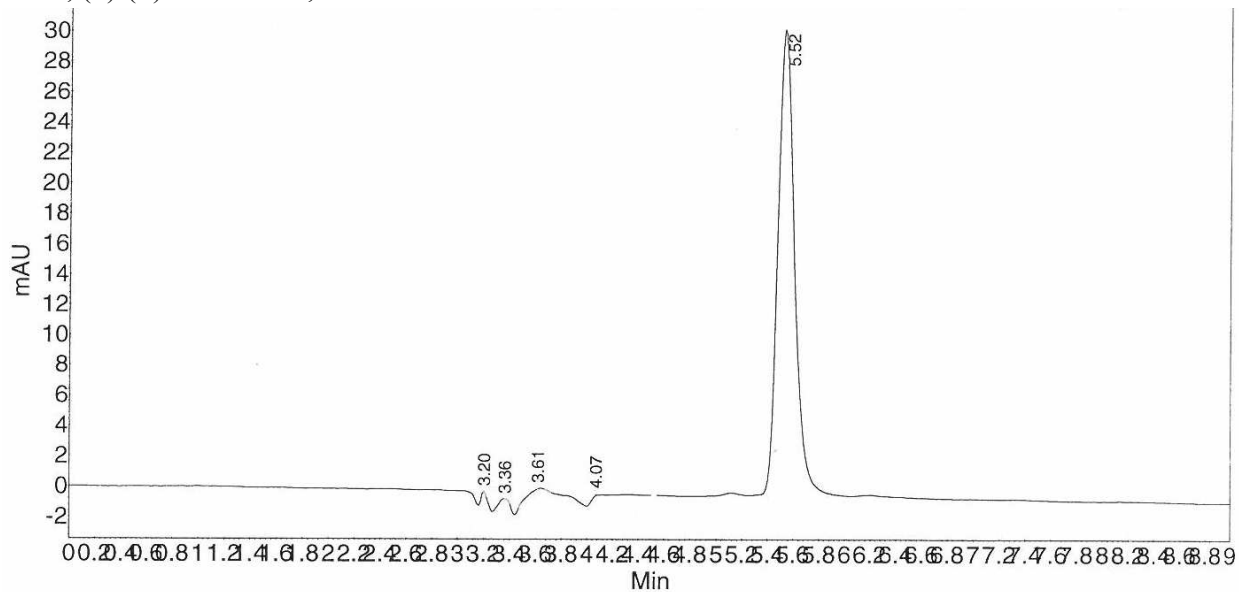
A: 37, Racemate

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	3.16	0.75	9.3	0.8	0.753
2	UNKNOWN	3.29	0.05	0.5	0.1	0.047
3	UNKNOWN	3.89	2.19	17.7	2.4	2.194
4	UNKNOWN	5.41	48.52	277.9	53.1	48.521
5	UNKNOWN	6.23	48.48	248.1	53.1	48.485
Total			100.00	553.5	109.5	100.000



B: 38, (S)-(-) Enantiomer; Peak 1



C: **39**; (R)-(+ Enantiomer; Peak 2

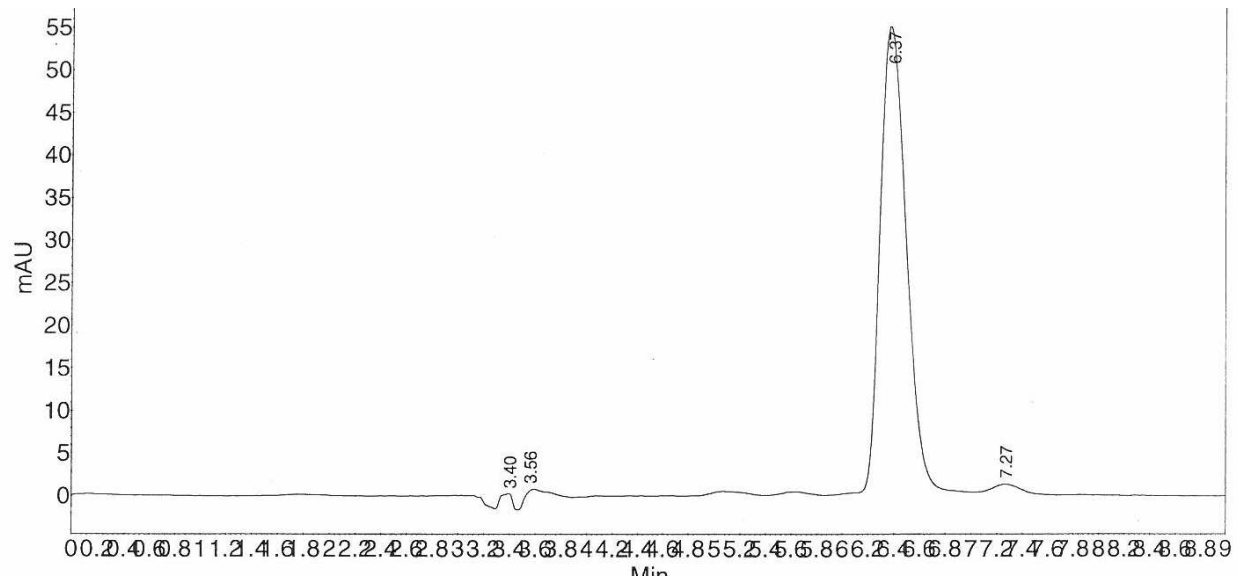


Figure 7: HPLC Chromatograms of **37** (A), **38** (S)-(-) Enantiomer; Peak 1 (B), and **39** (R)-(+ Enantiomer; Peak 2 (C) on chiral column (NP; Chiralpak IA, MTBE/ ethanol/DEA 2/8/0.1) recorded at 258 nm)

Table S1 ESI MS data of NPS antagonists 1 to 30

Compound	Molecular Mass	
	Found	Calculated
1	468.17 [M+H] ^{+a}	468.48
2	449.25 [M+H] ^{+a}	449.58
6	450.18 [M+Na] ^{+a}	450.50
7	429.2 [M+H] ^{+b}	429.50
8	429.0 [M+H] ^{+b} ,	429.50
9	429.3 [M+H] ^{+b} ;	429.50
10	443.4 [M+H] ^{+b} ,	443.53
11	443.5 [M+H] ^{+b} ,	443.53
12	443.5 [M+H] ^{+b} ,	443.53
13	565.3 [M+Na] ^{+b} .	565.24
14	443.5 [M+H] ^{+b}	443.21
15	458.3 [M+H] ^{+b}	457.55
16	388.16 [M+Na] ^{+b}	388.16

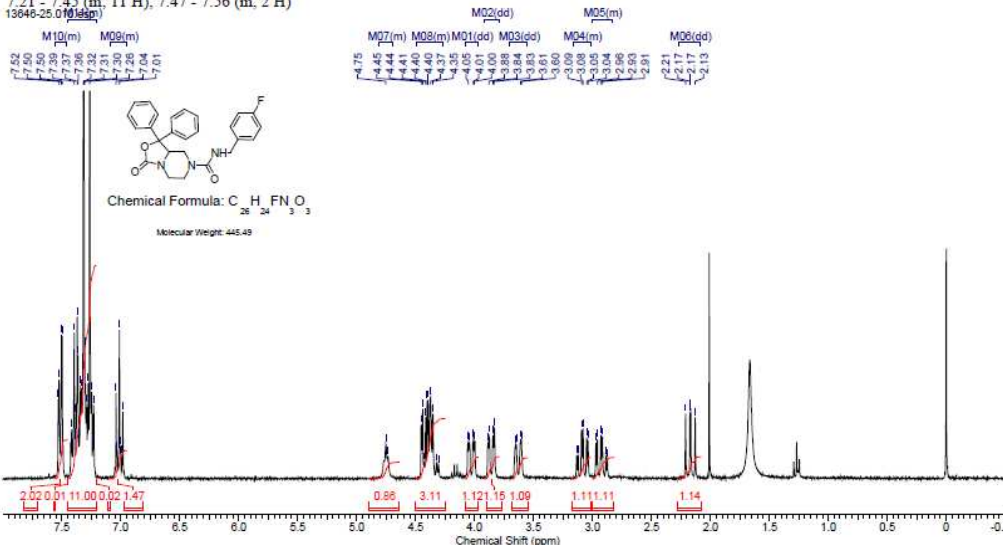
17	402.18 [M+Na] ^{+a} .	402.18
18	402.18 [M+Na] ^{+a} .	402.18
19	416.19 [M+Na] ^{+a} .	416.20
20	430.21 [M+Na] ^{+a}	430.21
21	444.22 [M+Na] ^{+a}	421.24
22	395.21 [M+H] ^{+a}	395.21
23	409.22 [M+H] ^{+a}	409.23
24	445.22 [M+Na] ^{+a}	445.22
25	434.0 [M+H] ^{+b}	434.56
26	435.2 [M+H] ^{+b}	435.55
27	507.1 [M+H] ^{+b}	507.61
28	407.6 [M+H] ^{+b}	407.50
29	507.2 [M+H] ^{+b}	507.61
30	407.4 [M+H] ^{+b}	407.50

Found values determined by a) ESI HRMS; b) ESI MS

¹H NMR spectra: SHA-68, 1

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Spectrum Offset (Hz)	1841.7688	Sweep Width (Hz)	6172.46	Temperature (degree C)	27.000

¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 2.17 (dd, $J=13.19, 11.30$ Hz, 1 H), 2.83 - 3.00 (m, 1 H), 3.01 - 3.16 (m, 1 H), 3.63 (dd, $J=13.00, 1.70$ Hz, 1 H), 3.86 (dd, $J=13.00, 2.45$ Hz, 1 H), 4.03 (dd, $J=13.19, 2.26$ Hz, 1 H), 4.26 - 4.49 (m, 3 H), 4.69 - 4.81 (m, 1 H), 6.95 - 7.09 (m, 1 H), 7.21 - 7.45 (m, 11 H), 7.47 - 7.56 (m, 2 H)

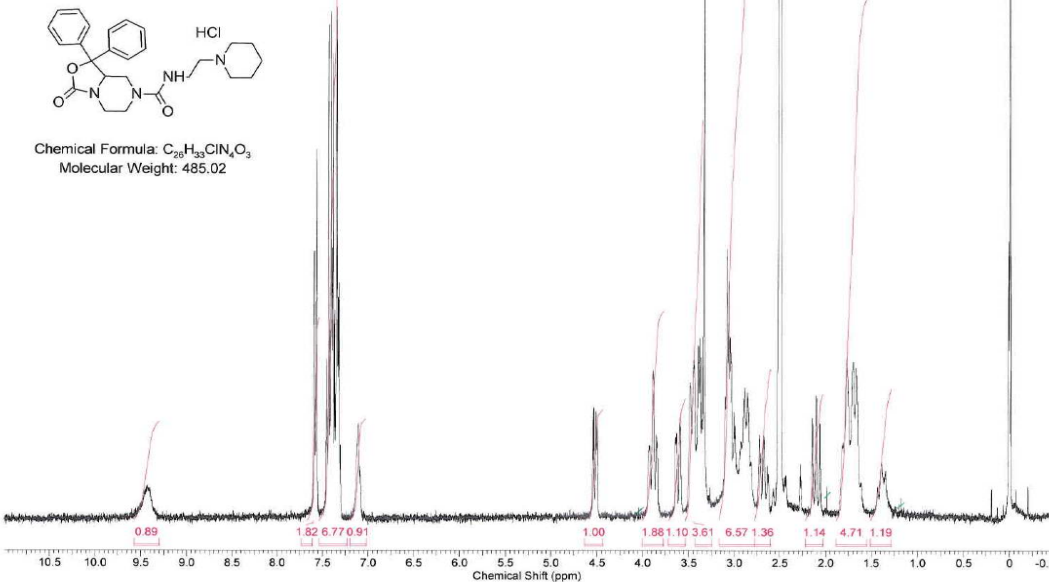


RTI-118, 2

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Compound 6

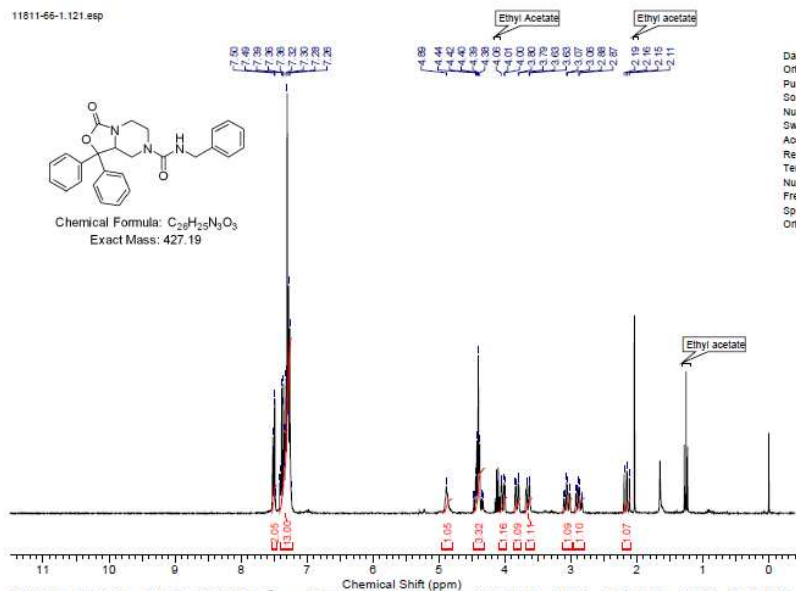
11811-66-1

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Date Stamp: 21 Nov 2007 20:18:08
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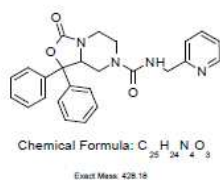


^1H NMR (300 MHz, *CHLOROFORM-d*) δ ppm 2.10 - 2.22 (1 H, m) 2.81 - 2.95 (1 H, m) 3.00 - 3.12 (1 H, m) 3.59 - 3.70 (1 H, m) 3.82 (1 H, dd, $J=13.0, 3.2$ Hz) 4.03 (1 H, dt, $J=13.3, 1.8$ Hz) 4.31 - 4.49 (3 H, m) 4.89 (1 H, br. s.) 7.24 - 7.42 (13 H, m) 7.47 - 7.54 (2 H, m)

Compound 7

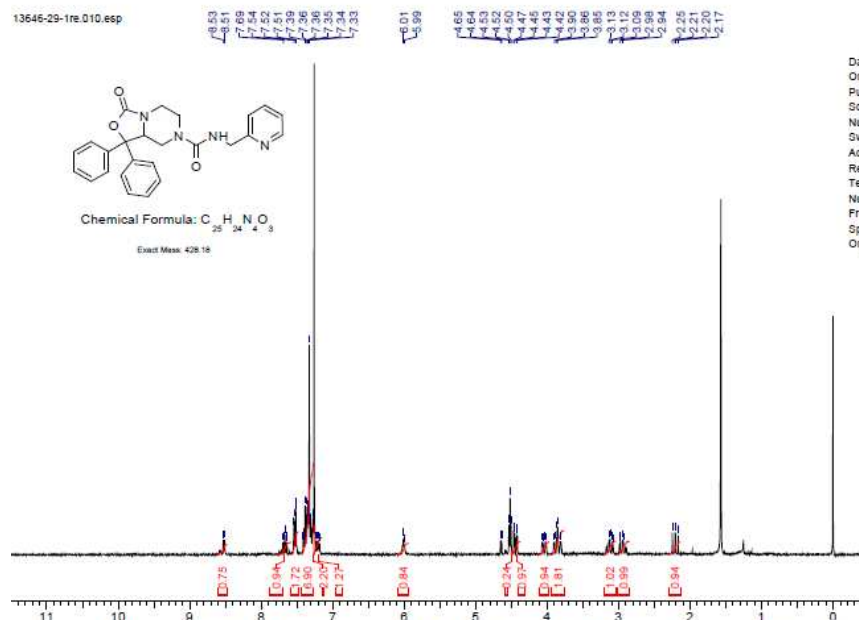
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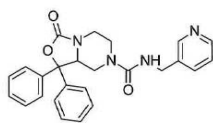


^1H NMR (300 MHz, *CHLOROFORM-d*) δ ppm 2.21 (1 H, dt, $J=13.3, 1.3$ Hz) 2.96 (1 H, dd, $J=13.6, 3.8$ Hz) 3.10 (1 H, dd, $J=13.2, 3.8$ Hz) 3.78 - 3.93 (2 H, m) 4.04 (1 H, dd, $J=13.4, 2.4$ Hz) 4.44 (1 H, dd, $J=11.3, 3.8$ Hz) 4.48 - 4.68 (2 H, m) 5.95 - 6.09 (1 H, m) 7.21 (1 H, dd, $J=7.2, 5.3$ Hz) 7.27 - 7.44 (9 H, m) 7.49 - 7.60 (2 H, m) 7.67 (1 H, td, $J=7.6, 1.7$ Hz) 8.52 (1 H, d, $J=4.9$ Hz)

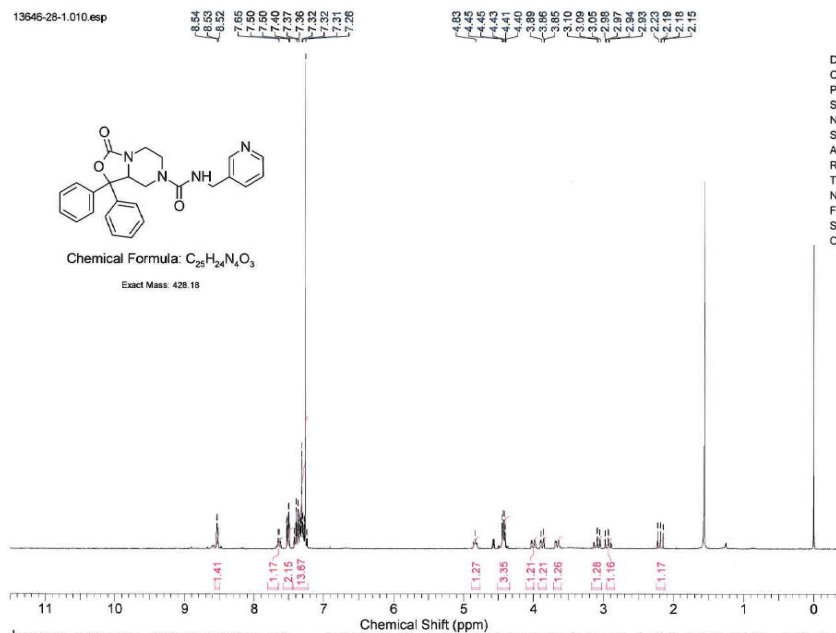
Compound 8

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13646-28-1.010.esp



Exact Mass: 428.18



^1H NMR (300 MHz, *CHLOROFORM-d*) δ ppm 2.19 (1 H, dd, $J=13.6, 11.3$ Hz) 2.95 (1 H, dd, $J=13.0, 3.2$ Hz) 3.03 - 3.15 (1 H, m) 3.61 - 3.70 (1 H, m) 3.82 - 3.93 (1 H, m) 3.95 - 4.05 (1 H, m) 4.34 - 4.51 (2 H, m) 4.83 (1 H, br. s.) 7.22 - 7.43 (10 H, m) 7.48 - 7.55 (2 H, m) 7.64 (1 H, d, $J=7.2$ Hz) 8.51 - 8.57 (1 H, m)

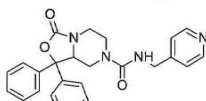
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Date Stamp 11 Mar 2013 15:24:4E
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Nucleus ^1H
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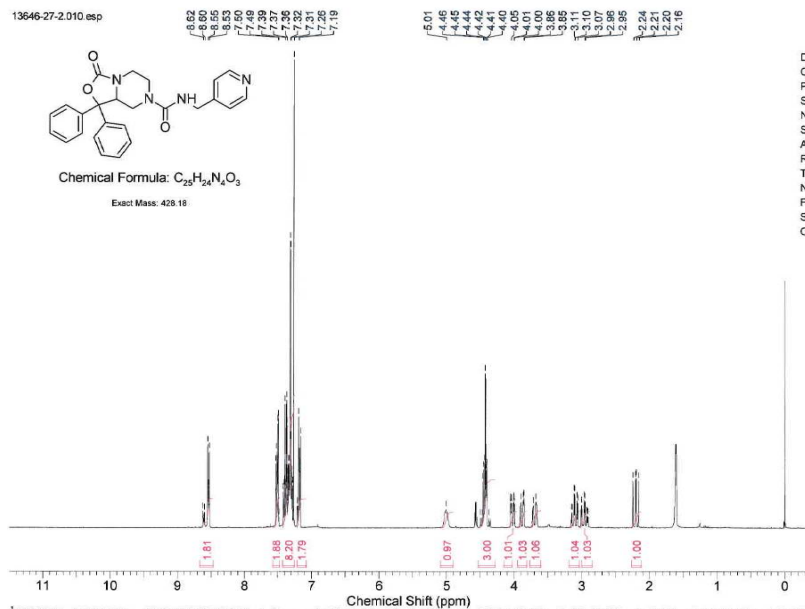
Compound 9

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Exact Mass: 428.18



^1H NMR (300 MHz, *CHLOROFORM-d*) δ ppm 2.20 (1 H, dd, $J=13.6, 11.3$ Hz) 2.89 - 3.02 (1 H, m) 3.03 - 3.18 (1 H, m) 3.62 - 3.75 (1 H, m) 3.82 - 3.94 (1 H, m) 4.02 (1 H, ddd, $J=13.4, 3.6, 1.5$ Hz) 4.31 - 4.51 (3 H, m) 5.01 (1 H, br. s.) 7.13 - 7.23 (2 H, m) 7.24 - 7.44 (8 H, m) 7.47 - 7.58 (2 H, m) 8.49 - 8.64 (2 H, m) 8.50 - 8.51 (1 H, m)

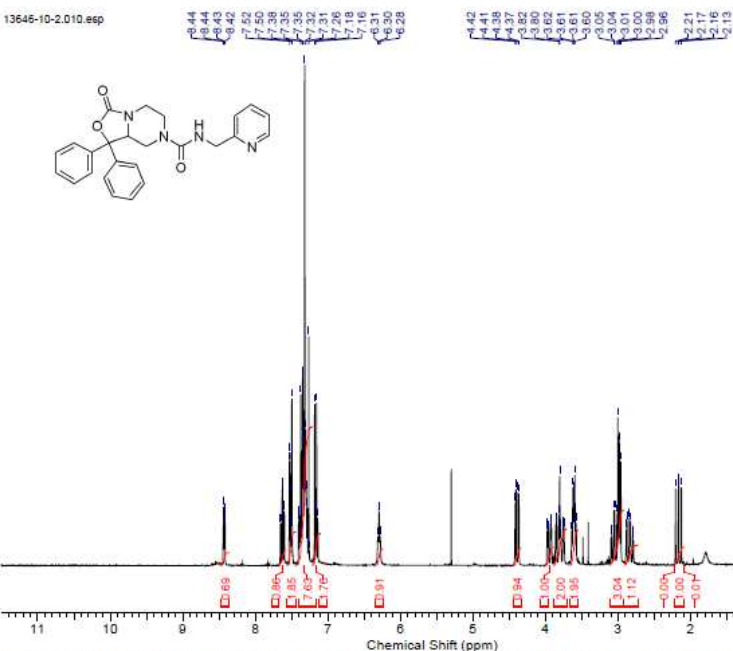
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Date Stamp 11 Mar 2013 14:52:4E
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Temperature (degree C) 27.000
Nucleus ^1H
Frequency (MHz) 300.13
Spectrum Offset (Hz) 1841.8622
Original Points Count 16384

Compound 10

13646-10-2

13646-10-2.010.esp



¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 2.17 (1 H, dd, *J*=13.2, 11.3 Hz) 2.84 (1 H, td, *J*=12.8, 3.4 Hz) 2.93 - 3.11 (3 H, m) 3.55 - 3.66 (2 H, m) 3.72 - 3.87 (2 H, m) 3.89 - 4.00 (1 H, m) 4.39 (1 H, dd, *J*=11.1, 3.6 Hz) 6.30 (1 H, t, *J*=4.9 Hz) 7.12 - 7.21 (2 H, m) 7.23 - 7.42 (8 H, m) 7.46 - 7.55 (2 H, m) 7.63 (1 H, td, *J*=7.7, 1.9 Hz) 8.43 (1 H, dt, *J*=4.6, 1.5 Hz)

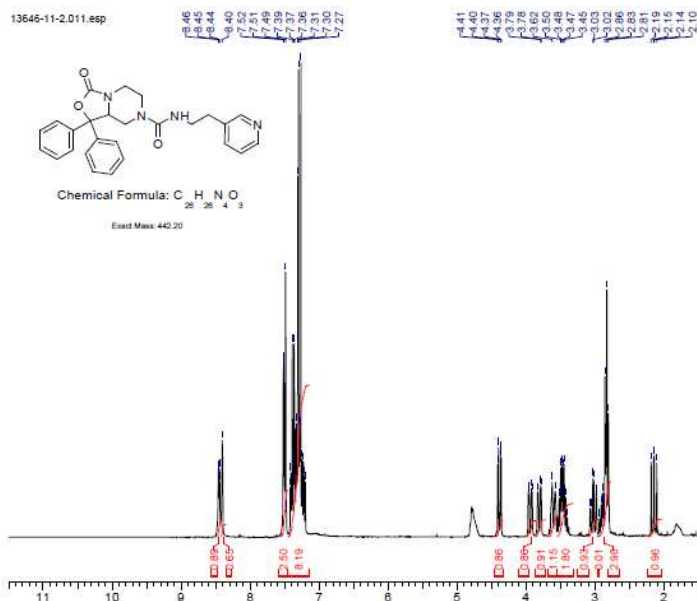
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Date Stamp 18 Feb 2013 16:20:00
Origin spect
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Temperature (degree C) 27.000
Nucleus 1H
Frequency (MHz) 300.13
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Original Points Count 16384

Compound 11

13646-11-2

13646-11-2.011.esp



¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 2.14 (1 H, dd, *J*=13.2, 11.3 Hz) 2.78 - 2.95 (3 H, m) 3.02 (1 H, td, *J*=12.7, 3.6 Hz) 3.34 - 3.53 (2 H, m) 3.60 (1 H, d, *J*=12.8 Hz) 3.81 (1 H, dd, *J*=12.8, 2.6 Hz) 3.94 (1 H, dd, *J*=13.2, 3.4 Hz) 4.39 (1 H, dd, *J*=11.3, 3.8 Hz) 7.17 - 7.44 (9 H, m) 7.46 - 7.56 (3 H, m) 8.35 - 8.50 (2 H, m)

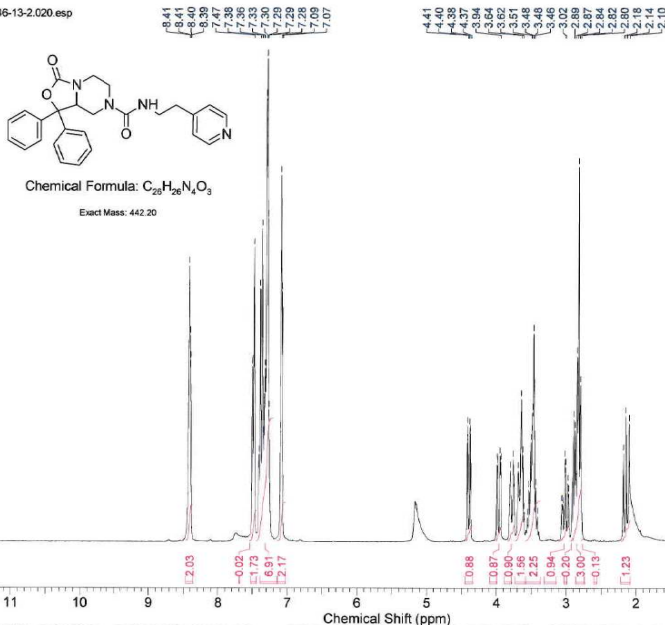
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Date Stamp 18 Feb 2013 16:54:08
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Temperature (degree C) 27.000
Nucleus 1H
Frequency (MHz) 300.13
Spectrum Offset (Hz) 1843.2217
Original Points Count 16384

Compound 12

13646-13-2

13646-13-2.020.esp



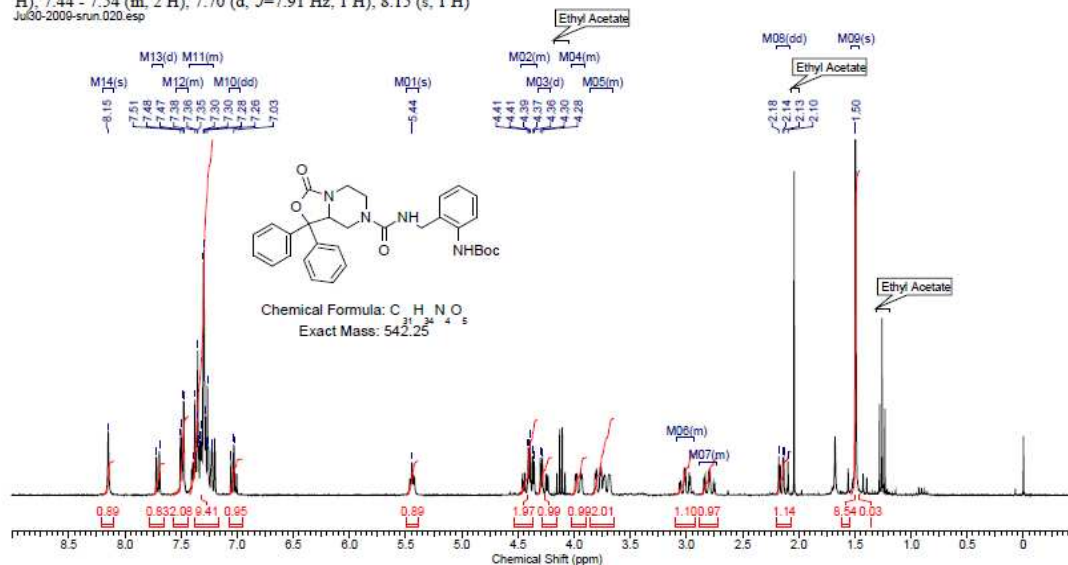
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 Original Points Count 16384

¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 2.08 - 2.22 (1 H, m) 2.78 - 2.91 (3 H, m) 3.01 (1 H, td, J=12.7, 3.2 Hz) 3.37 - 3.58 (2 H, m) 3.58 - 3.73 (2 H, m) 3.78 (1 H, d, J=13.2 Hz) 3.96 (1 H, dd, J=13.4, 2.8 Hz) 4.39 (1 H, dd, J=11.1, 3.6 Hz) 7.02 - 7.15 (2 H, m) 7.19 - 7.44 (7 H, m) 7.44 - 7.53 (2 H, m) 8.36 - 8.46 (2 H, m)

Compound 13

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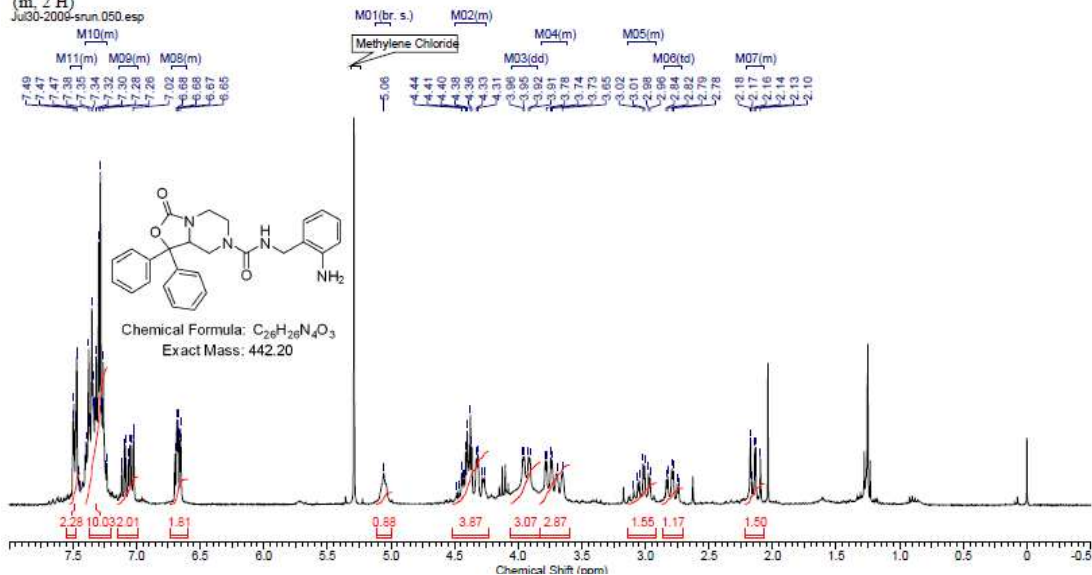
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 1.50 (s, 9 H), 2.14 (dd, J=13.37, 11.11 Hz, 1 H), 2.73 - 2.89 (m, 1 H), 2.93 - 3.09 (m, 1 H), 3.66 - 3.85 (m, 2 H), 3.91 - 4.02 (m, 1 H), 4.29 (d, J=5.65 Hz, 1 H), 4.34 - 4.48 (m, 2 H), 5.44 (s, 1 H), 7.04 (dd, J=7.54, 1.13 Hz, 1 H), 7.21 - 7.42 (m, 10 H), 7.44 - 7.54 (m, 2 H), 7.70 (d, J=7.91 Hz, 1 H), 8.15 (s, 1 H)



Compound 14

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Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46	Temperature (degree C)	27.000		

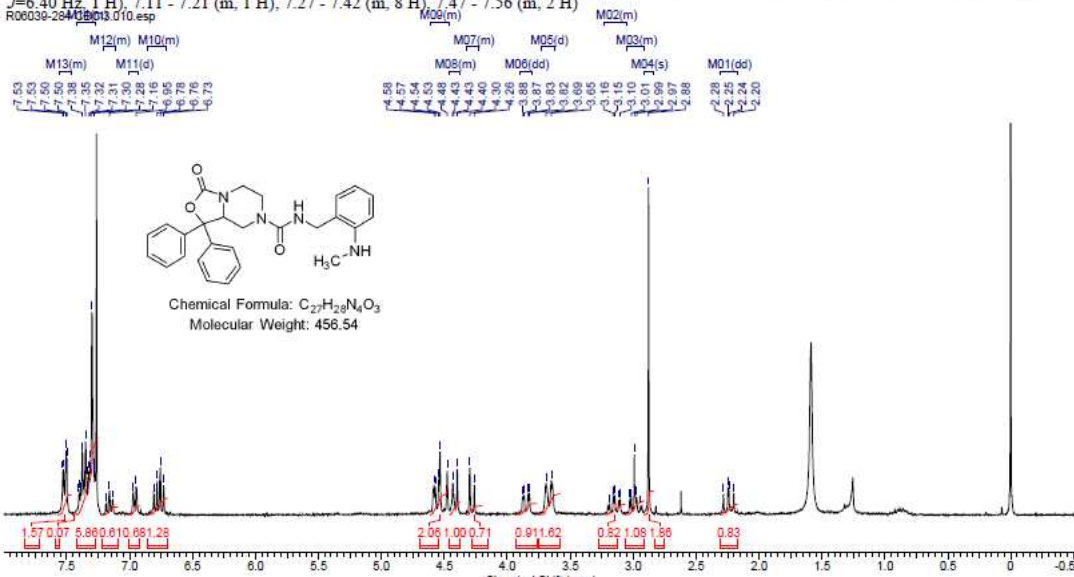
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 2.07 - 2.21 (m, 1 H), 2.79 (td, $J=12.81, 3.77$ Hz, 1 H), 2.92 - 3.13 (m, 1 H), 3.61 - 3.81 (m, 3 H), 3.93 (dd, $J=13.19, 3.01$ Hz, 3 H), 4.25 - 4.50 (m, 4 H), 5.06 (br. s., 1 H), 6.61 - 6.72 (m, 2 H), 6.99 - 7.14 (m, 2 H), 7.23 - 7.40 (m, 8 H), 7.44 - 7.52 (m, 2 H)



Compound 15

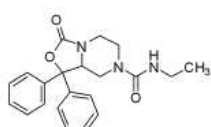
Acquisition Time (sec)	2.6542	Comment	R06039-284	Date	02 May 2013 14:31:28		
Date Stamp	02 May 2013 14:31:28			File Name	C:\Documents and Settings\chassler\My Documents\NMR\R06039-284 CDCI3\10\fid		
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	128	Origin	spect
Original Points Count	16384	Owner	nmrUSER	Points Count	16384	Pulse Sequence	zg
Receiver Gain	1024.00	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	1841.9103	Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46	Temperature (degree C)	27.000

¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 2.24 (dd, $J=13.56, 11.30$ Hz, 1 H), 2.88 (s, 3 H), 2.91 - 3.05 (m, 1 H), 3.05 - 3.23 (m, 1 H), 3.67 (d, $J=12.43$ Hz, 2 H), 3.85 (dd, $J=13.00, 2.83$ Hz, 1 H), 4.23 - 4.32 (m, 1 H), 4.38 - 4.46 (m, 1 H), 4.47 - 4.61 (m, 3 H), 6.71 - 6.86 (m, 2 H), 6.96 (d, $J=6.40$ Hz, 1 H), 7.11 - 7.21 (m, 1 H), 7.27 - 7.42 (m, 8 H), 7.47 - 7.56 (m, 2 H)



Compound 16

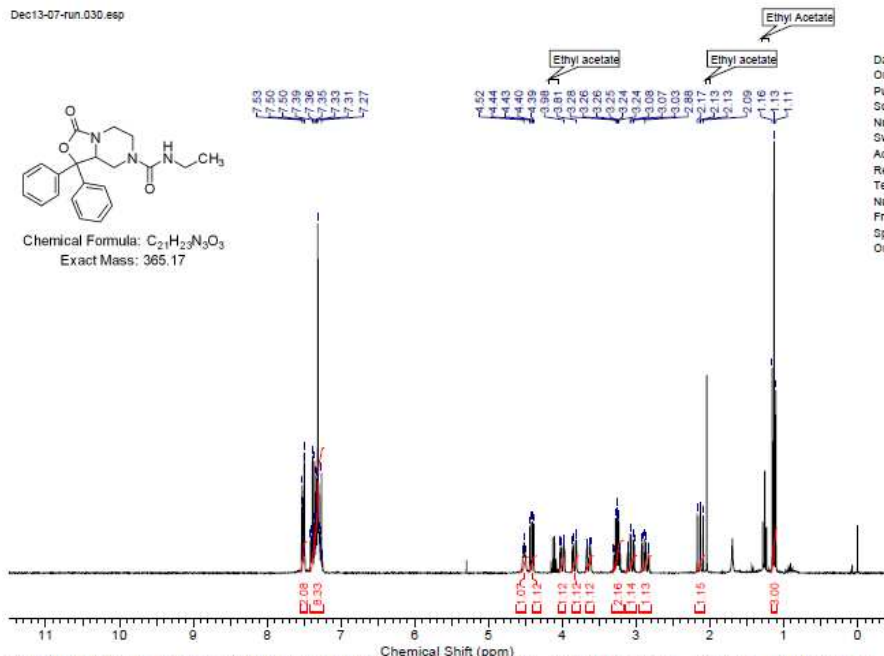
Dec13-07-run.030.esp



Chemical Formula: $C_{21}H_{23}N_3O_3$
Exact Mass: 365.17



Date Stamp 13 Dec 2007 19:46:16
Origin spect
Pulse Sequence zg
Solvent CHLOROFORM-d
Number of Transients 16
Sweep Width (Hz) 6172.46
Acquisition Time (sec) 2.6542
Receiver Gain 228.10
Temperature (degree C) 27.000
Nucleus 1H
Frequency (MHz) 300.13
Spectrum Offset (Hz) 1849.1913
Original Points Count 16384



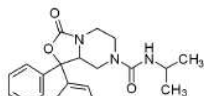
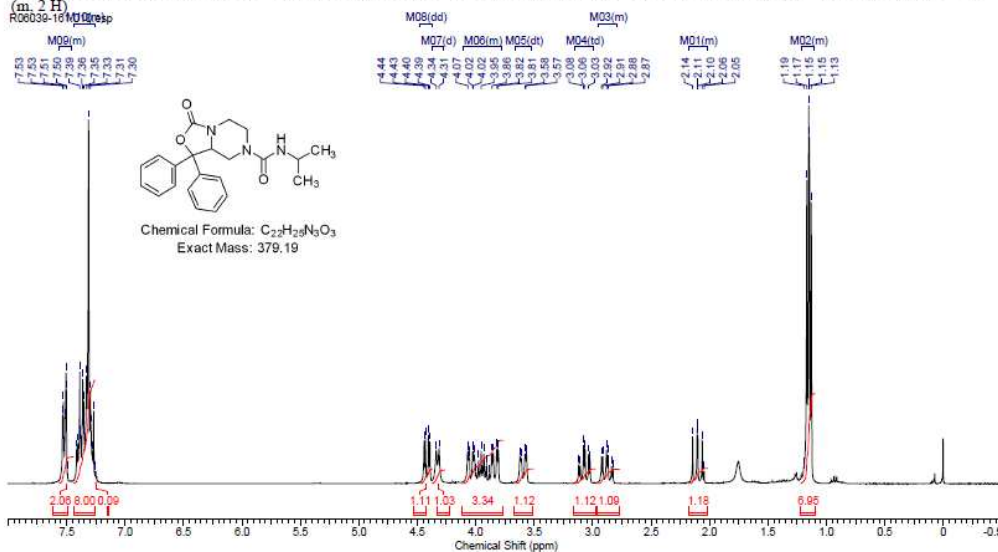
1H NMR (300 MHz, $CHLOROFORM-d$) δ ppm 1.13 (3 H, t, $J=7.2$ Hz) 2.07 - 2.20 (1 H, m) 2.81 - 2.96 (1 H, m) 3.07 (1 H, td, $J=12.7, 3.6$ Hz) 3.19 - 3.35 (2 H, m) 3.58 - 3.69 (1 H, m) 3.84 (1 H, dd, $J=13.0, 2.4$ Hz) 4.00 (1 H, ddd, $J=13.3, 3.5, 1.3$ Hz) 4.42 (1 H, dd, $J=11.3, 3.4$ Hz) 4.50 - 4.58 (1 H, m) 7.25 - 7.42 (8 H, m) 7.47 - 7.54 (2 H, m)

Compound 17

4/30/2013 5:01:06 PM
R06039-161

Acquisition Time (sec)	2.6542	Comment	R06039-161	Date	30 Apr 2013 16:03:12
Date Stamp	30 Apr 2013 16:03:12	File Name	C:\Documents and Settings\jchasser\My Documents\NMR\R06039-161\110\fid		
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	128
Original Points Count	16384	Owner	nmrUSER	Points Count	16384
Receiver Gain	203.20	SW(cyclical) (Hz)	6172.94	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1843.7799	Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46
				Temperature (degree C)	27.000

1H NMR (300 MHz, $CHLOROFORM-d$) δ ppm 1.10 - 1.21 (m, 6 H), 2.02 - 2.18 (m, 1 H), 2.79 - 2.95 (m, 1 H), 3.07 (td, $J=12.62, 3.39$ Hz, 1 H), 3.59 (dt, $J=13.19, 1.70$ Hz, 1 H), 3.78 - 4.11 (m, 3 H), 4.33 (d, $J=7.16$ Hz, 1 H), 4.42 (dd, $J=11.30, 3.77$ Hz, 1 H), 7.25 - 7.44 (m, 8 H), 7.46 - 7.56 (m, 2 H)



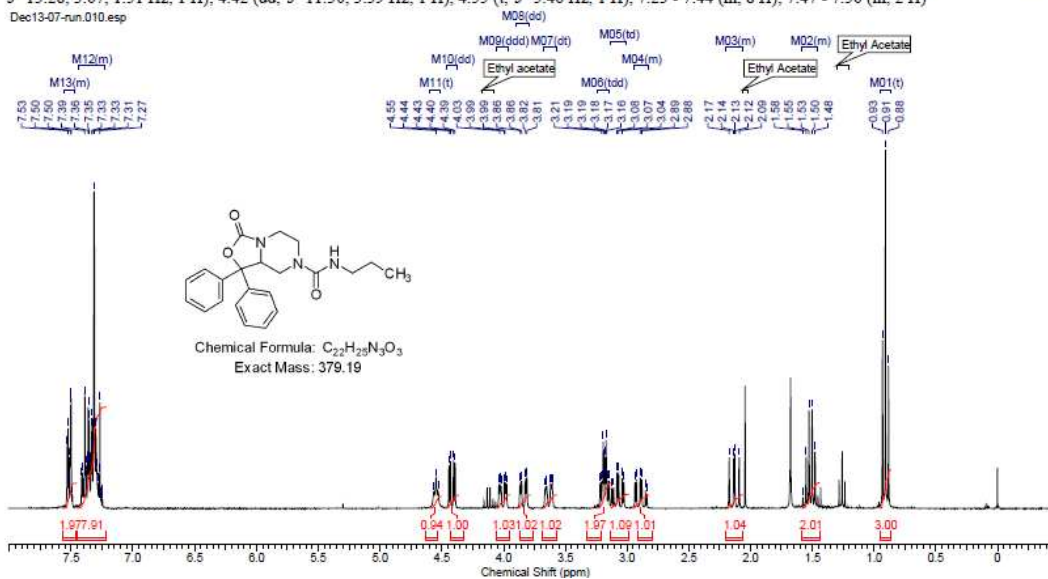
Chemical Formula: $C_{22}H_{25}N_3O_3$
Exact Mass: 379.19

Compound 18

Acquisition Time (sec)	2.6542	Date	13 Dec 2007 16:08:32	Date Stamp	13 Dec 2007 16:08:32
File Name	C:\Documents and Settings\chassier\My Documents\NMR\SPR NMR data raw files\SPR Archive data nmr\2005to2008\Dec13-07-run\10\fid				
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	16
Original Points Count	16384	Owner	nmr\USER	Points Count	16384
Receiver Gain	256.00	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46	Temperature (degree C)	27.000
				Pulse Sequence	zg
				Spectrum Offset (Hz)	1848.9683

¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 0.91 (t, $J=7.54$ Hz, 3 H), 1.46 - 1.59 (m, 2 H), 2.07 - 2.20 (m, 1 H), 2.83 - 2.95 (m, 1 H), 3.07 (td, $J=12.62, 3.39$ Hz, 1 H), 3.18 (tdd, $J=7.25, 7.25, 5.46, 1.88$ Hz, 2 H), 3.63 (dt, $J=13.19, 1.88$ Hz, 1 H), 3.84 (dd, $J=12.81, 2.64$ Hz, 1 H), 4.01 (ddd, $J=13.28, 3.67, 1.51$ Hz, 1 H), 4.42 (dd, $J=11.30, 3.39$ Hz, 1 H), 4.55 (t, $J=5.46$ Hz, 1 H), 7.23 - 7.44 (m, 8 H), 7.47 - 7.56 (m, 2 H)

Dec13-07-run.010.esp

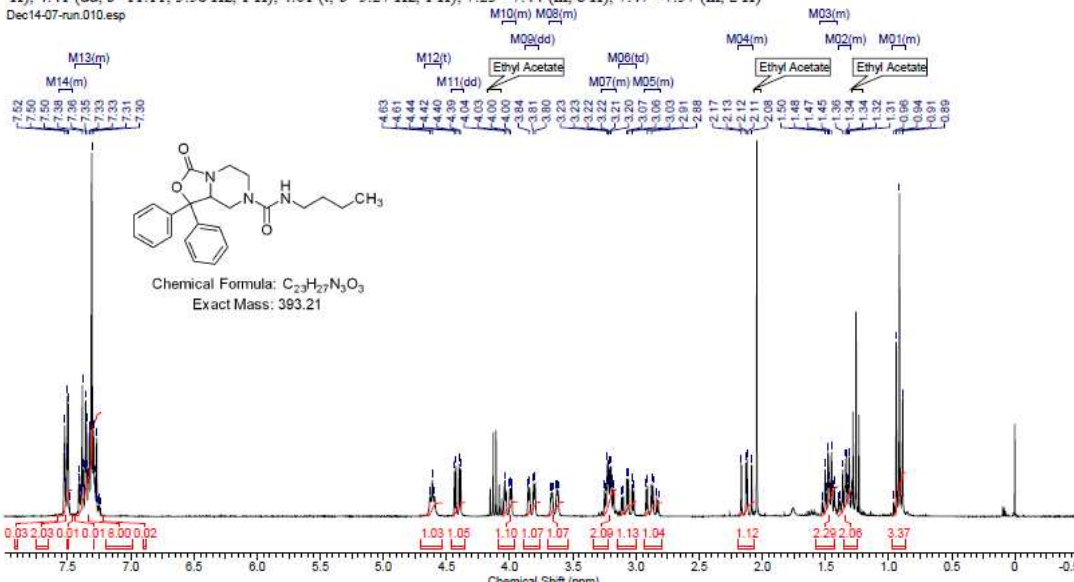


Compound 19

Acquisition Time (sec)	2.6542	Date	14 Dec 2007 17:44:32	Date Stamp	14 Dec 2007 17:44:32
File Name	C:\Documents and Settings\chassier\My Documents\NMR\SPR NMR data raw files\SPR Archive data nmr\2005to2008\Dec14-07-run\10\fid				
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	16
Original Points Count	16384	Owner	nmr\USER	Points Count	16384
Receiver Gain	128.00	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46	Temperature (degree C)	27.000
				Pulse Sequence	zg
				Spectrum Offset (Hz)	1850.2657

¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 0.87 - 0.97 (m, 3 H), 1.30 - 1.40 (m, 2 H), 1.41 - 1.54 (m, 2 H), 2.07 - 2.19 (m, 1 H), 2.81 - 2.94 (m, 1 H), 3.07 (td, $J=12.62, 3.39$ Hz, 1 H), 3.16 - 3.27 (m, 2 H), 3.59 - 3.69 (m, 1 H), 3.83 (dd, $J=13.00, 2.83$ Hz, 1 H), 3.95 - 4.05 (m, 1 H), 4.41 (dd, $J=11.11, 3.58$ Hz, 1 H), 4.61 (t, $J=5.27$ Hz, 1 H), 7.23 - 7.44 (m, 8 H), 7.47 - 7.57 (m, 2 H)

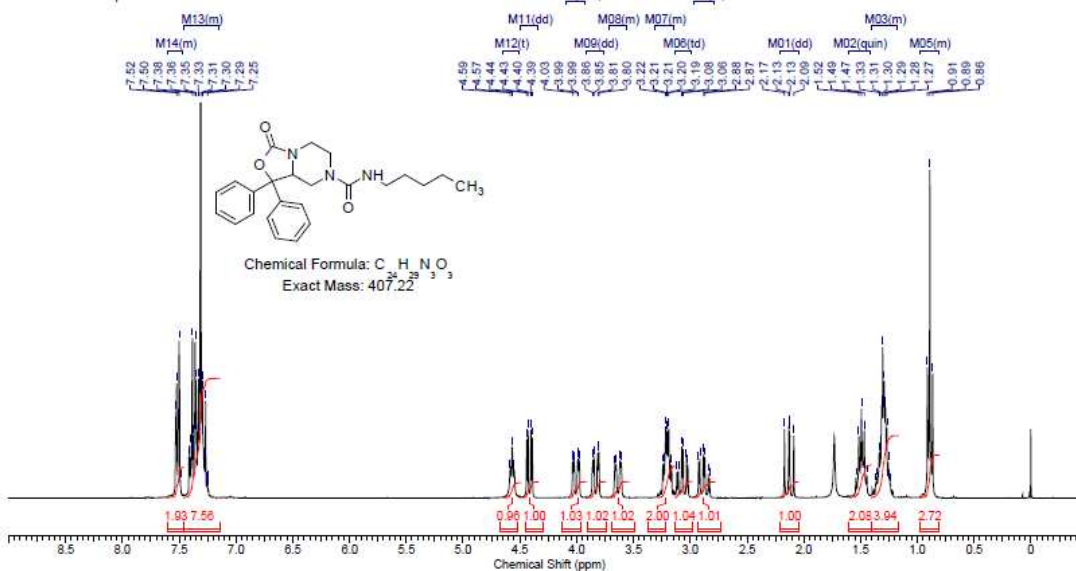
Dec14-07-run.010.esp



Compound 20

Acquisition Time (sec)	2.6542	Comment	R06039-180	Date	30 Apr 2013 17:07:12
Date Stamp	30 Apr 2013 17:07:12	File Name	C:\Documents and Settings\chasserl\My Documents\NMR\R06039-180\10fid		
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	128
Original Points Count	16384	Owner	nmrUSER	Points Count	16384
Receiver Gain	181.00	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1843.2789	Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46
				Temperature (degree C)	27.000

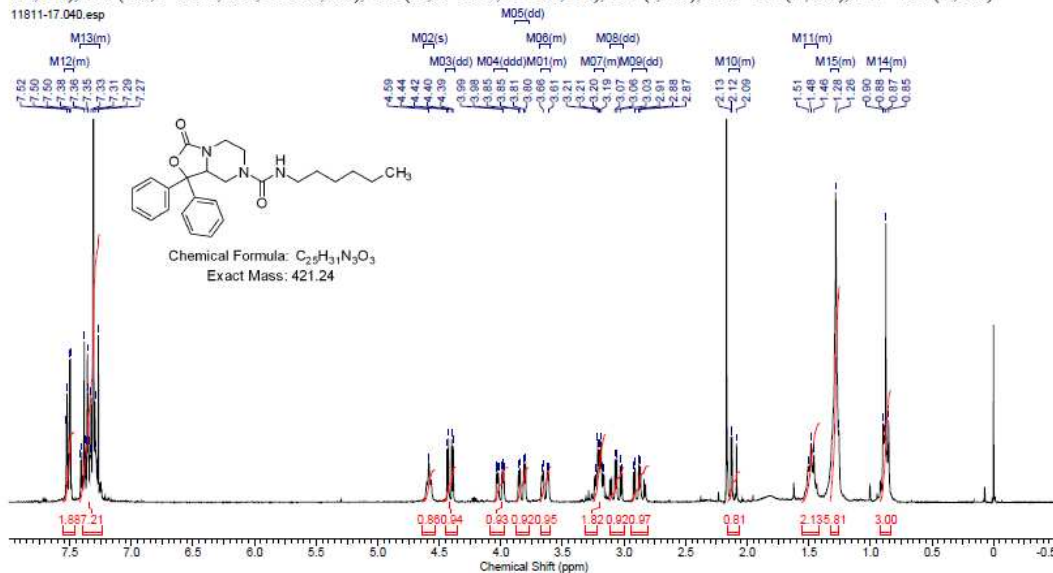
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 0.81 - 0.98 (m, 3 H), 1.18 - 1.41 (m, 4 H), 1.49 (quin, $J=7.25$ Hz, 2 H), 2.13 (dd, $J=13.19, 11.30$ Hz, 1 H), 2.79 - 2.97 (m, 1 H), 3.07 (td, $J=12.62, 3.77$ Hz, 1 H), 3.14 - 3.31 (m, 2 H), 3.56 - 3.72 (m, 1 H), 3.83 (dd, $J=13.19, 2.64$ Hz, 1 H), 4.01 (ddd, $J=13.37, 3.58, 1.13$ Hz, 1 H), 4.41 (dd, $J=11.30, 3.77$ Hz, 1 H), 4.57 (t, $J=5.46$ Hz, 1 H), 7.16 - 7.46 (m, 8 H), 7.47 - 7.61 (m, 2 H)



Compound 21

Acquisition Time (sec)	2.6542	Comment	11811-17	Date	05 Sep 2007 19:50:24
Date Stamp	05 Sep 2007 19:50:24	File Name	E:\Experimentals\Yzhang archive data 2008\nmr\11811-17\40\fd		
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	16
Original Points Count	16384	Owner	nmrUSER	Points Count	16384
Receiver Gain	161.30	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1849.5393	Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46
				Temperature (degree C)	27.000

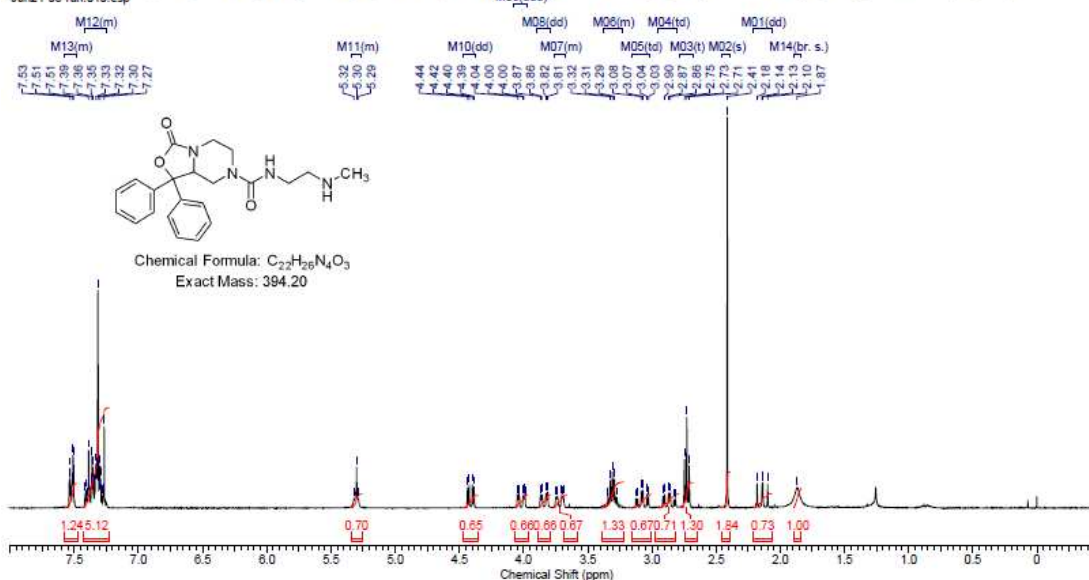
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 0.84 - 0.92 (m, 3 H), 1.25 - 1.33 (m, 6 H), 1.43 - 1.53 (m, 2 H), 2.06 - 2.15 (m, 1 H), 2.89 (dd, $J=13.00, 3.58$ Hz, 1 H), 3.05 (dd, $J=12.62, 3.58$ Hz, 1 H), 3.16 - 3.24 (m, 2 H), 3.60 - 3.69 (m, 1 H), 3.60 - 3.68 (m, 1 H), 3.83 (dd, $J=13.00, 2.83$ Hz, 1 H), 4.01 (ddd, $J=13.38, 3.58, 1.13$ Hz, 1 H), 4.41 (dd, $J=11.30, 3.77$ Hz, 1 H), 4.59 (s, 1 H), 7.26 - 7.42 (m, 8 H), 7.47 - 7.55 (m, 2 H)



Compound 22

Acquisition Time (sec)	2.6542	Date	24 Jun 2008 18:42:08	Date Stamp	24 Jun 2008 18:42:08
File Name	C:\Documents and Settings\hassler\My Documents\NMR\SPR NMR data raw files\SPR Archive data nmr\2008\2008\Jun24-08-run\10\fid				
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	16
Original Points Count	16384	Owner	nmrUSER	Points Count	16384
Receiver Gain	362.00	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46	Temperature (degree C)	27.000
				Spectrum Offset (Hz)	1848.8284

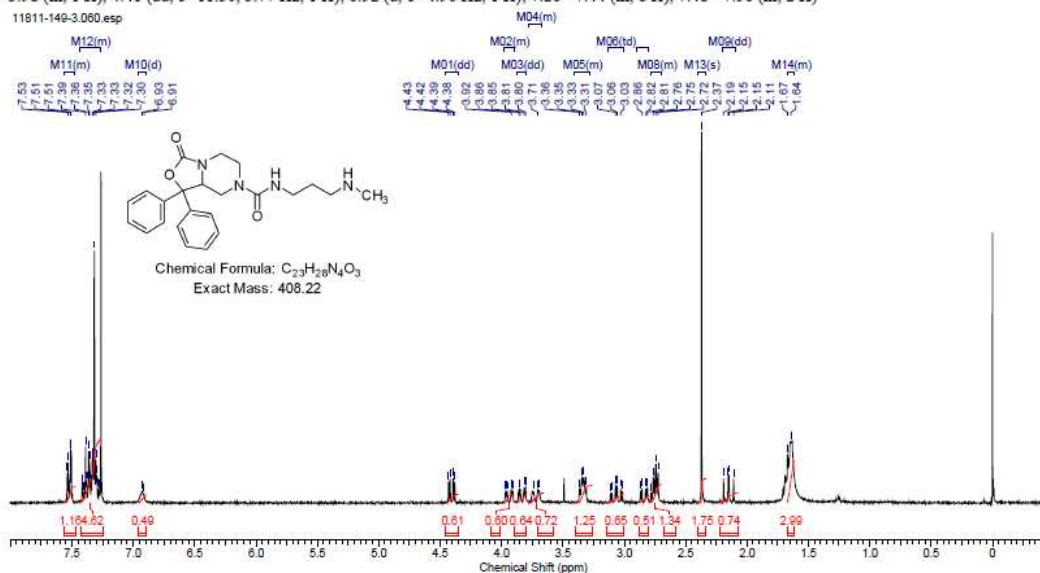
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 1.87 (br. s., 1 H), 2.14 (dd, $J=13.19, 11.30$ Hz, 1 H), 2.41 (s, 3 H), 2.73 (t, $J=5.65$ Hz, 2 H), 2.86 (td, $J=12.81, 3.77$ Hz, 1 H), 3.08 (td, $J=12.72, 3.58$ Hz, 1 H), 3.23 - 3.38 (m, 2 H), 3.67 - 3.75 (m, 1 H), 3.84 (dd, $J=13.19, 2.64$ Hz, 1 H), 4.02 (ddd, $J=13.37, 3.58, 1.13$ Hz, 1 H), 4.41 (dd, $J=11.30, 3.77$ Hz, 1 H), 5.26 - 5.34 (m, 1 H), 7.25 - 7.42 (m, 8 H), 7.48 - 7.58 (m, 2 H)



Compound 23

Acquisition Time (sec)	2.6542	Comment	11811-149-3	Date	27 Mar 2008 22:49:38
Date Stamp	27 Mar 2008 22:49:38				
File Name	E:\Experimentals\Yzhang archive data 2008\nmr\11811-149-3\60\fid				
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	16
Original Points Count	16384	Owner	nmrUSER	Points Count	16384
Receiver Gain	812.70	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46	Temperature (degree C)	27.000
				Spectrum Offset (Hz)	1847.8197

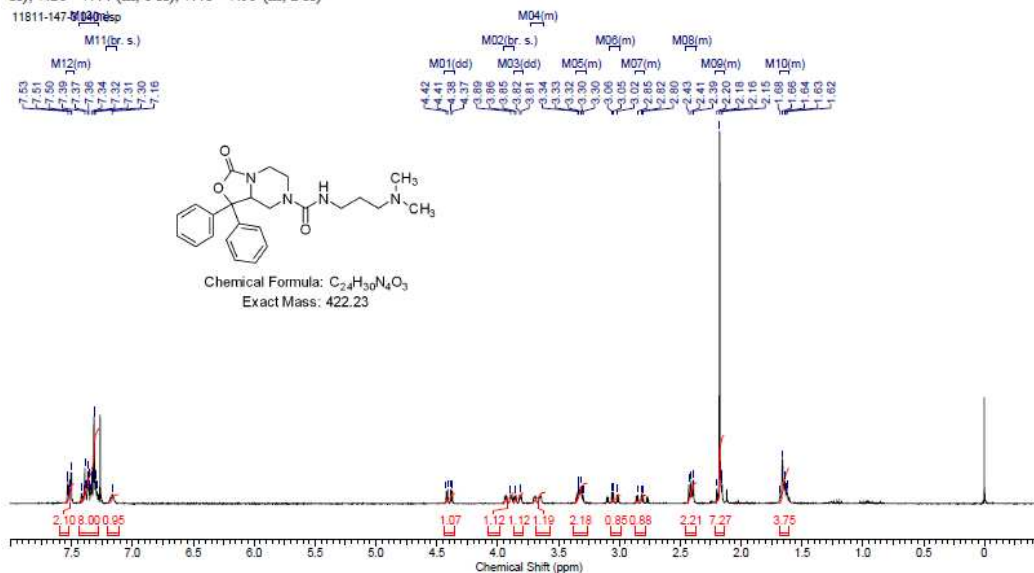
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 1.62 - 1.67 (m, 3 H), 2.15 (dd, $J=13.37, 11.11$ Hz, 1 H), 2.37 (s, 3 H), 2.70 - 2.78 (m, 2 H), 2.84 (dd, $J=13.19, 3.39$ Hz, 1 H), 3.07 (td, $J=12.62, 3.77$ Hz, 1 H), 3.28 - 3.41 (m, 2 H), 3.67 - 3.77 (m, 1 H), 3.83 (dd, $J=13.37, 2.83$ Hz, 1 H), 3.90 - 3.98 (m, 1 H), 4.41 (dd, $J=11.30, 3.77$ Hz, 1 H), 6.92 (d, $J=4.90$ Hz, 1 H), 7.26 - 7.44 (m, 8 H), 7.48 - 7.56 (m, 2 H)



Compound 24

Acquisition Time (sec)	2.6542	Comment	11811-147-3	Date	25 Mar 2008 16:25:36
Date Stamp	25 Mar 2008 16:25:36			File Name	E:\Experimentals\Yzhang archive data 2008\nmr\11811-147-340\fid
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	16
Original Points Count	16384	Owner	nmr\USER	Points Count	16384
Receiver Gain	512.00	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1848.3538	Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46
				Temperature (degree C)	27.000

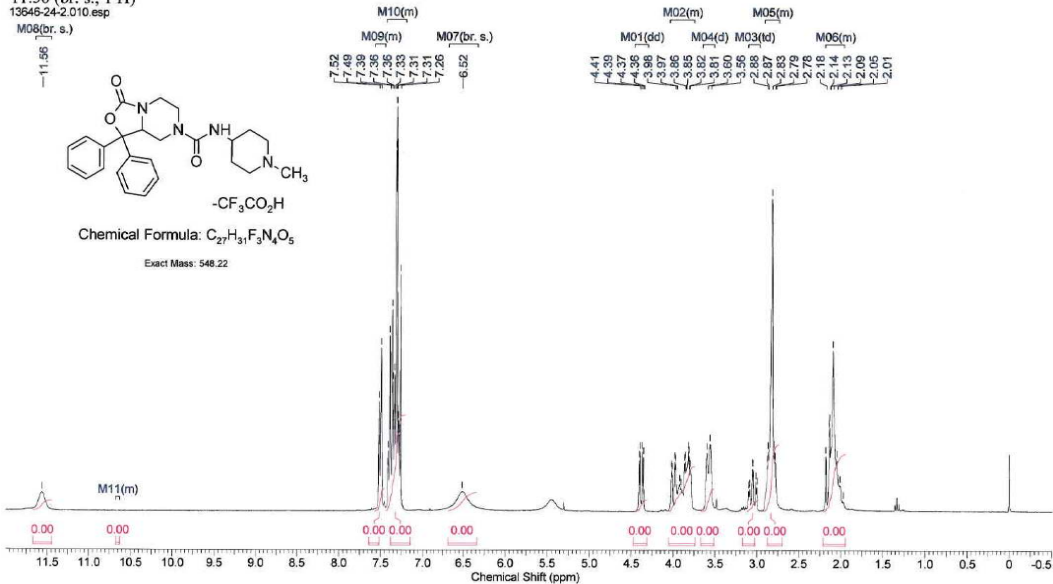
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 1.61 - 1.68 (m, 3 H), 2.14 - 2.21 (m, 6 H), 2.37 - 2.45 (m, 2 H), 2.79 - 2.87 (m, 1 H), 3.00 - 3.08 (m, 1 H), 3.28 - 3.36 (m, 2 H), 3.62 - 3.73 (m, 1 H), 3.83 (dd, $J=12.81, 2.64$ Hz, 1 H), 3.89 (br. s., 1 H), 4.40 (dd, $J=11.11, 3.58$ Hz, 1 H), 7.16 (br. s., 1 H), 7.28 - 7.44 (m, 8 H), 7.48 - 7.55 (m, 2 H)



Compound 25

Acquisition Time (sec)	2.6542	Comment	13646-24-2	Date	27 Mar 2013 13:42:24
Date Stamp	27 Mar 2013 13:42:24			File Name	C:\Documents and Settings\chasser\My Documents\NMR\13646-24-2\10\fid
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	128
Original Points Count	16384	Owner	nmr\USER	Points Count	16384
Receiver Gain	512.00	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1842.1953	Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46
				Temperature (degree C)	27.000

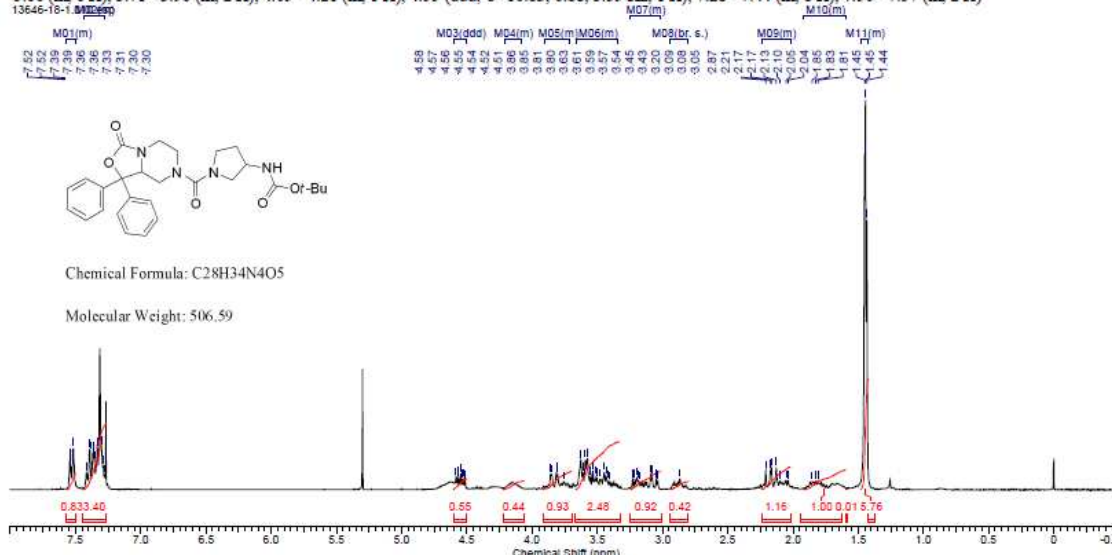
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 1.97 - 2.20 (m, 5 H), 2.75 - 2.92 (m, 6 H), 3.05 (td, $J=12.62, 3.39$ Hz, 1 H), 3.58 (d, $J=12.06$ Hz, 2 H), 3.76 - 4.05 (m, 4 H), 4.38 (dd, $J=11.11, 3.58$ Hz, 1 H), 6.52 (br. s., 1 H), 7.20 - 7.44 (m, 8 H), 7.46 - 7.58 (m, 2 H), 10.63 - 10.68 (m, 0 H), 11.56 (br. s., 1 H)



Compound 26

Acquisition Time (sec)	2.6542	Comment	13646-17-1	Date	20 Feb 2013 14:24:48
Date Stamp	20 Feb 2013 14:24:48	File Name	C:\Documents and Settings\chassier\My Documents\NMR\13646-17-1\1010.fid		
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	64
Origin	nmrUSER	Points Count	16384	Pulse Sequence	zg
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	1843.3958	Receiver Gain	256.00
Temperature (degree C)	27.000	Spectrum Type	STANDARD	SW (Hz)	6172.54
				Sweep Width (Hz)	6172.46

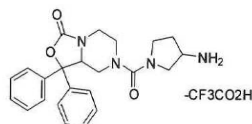
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 1.42 - 1.48 (m, 9 H), 1.60 - 1.92 (m, 1 H), 2.02 - 2.24 (m, 2 H), 2.87 (br. s., 1 H), 3.02 - 3.25 (m, 2 H), 3.34 - 3.66 (m, 6 H), 3.71 - 3.90 (m, 2 H), 4.09 - 4.21 (m, 1 H), 4.55 (ddd, J=10.83, 6.88, 3.39 Hz, 1 H), 7.28 - 7.44 (m, 8 H), 7.50 - 7.57 (m, 2 H)



Compound 27

13646-21-1

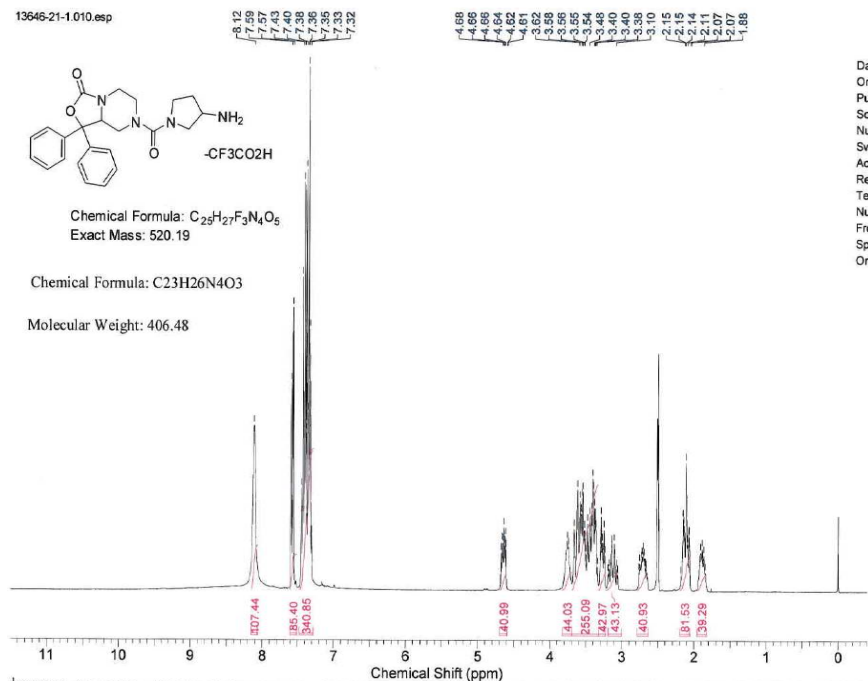
13646-21-1010.esp



Chemical Formula: C₂₅H₂₇F₃N₄O₅
Exact Mass: 520.19

Chemical Formula: C₂₃H₂₆N₄O₃

Molecular Weight: 406.48



¹H NMR (300 MHz, DMSO-d₆) δ ppm 1.82 - 1.95 (1 H, m), 2.06 - 2.20 (2 H, m), 2.65 - 2.78 (1 H, m), 3.05 - 3.21 (1 H, m), 3.27 (1 H, dt, J=11.6, 4.0 Hz), 3.33 - 3.70 (6 H, m), 3.75 (1 H, d, J=4.5 Hz), 4.64 (1 H, ddd, J=10.8, 6.3, 3.6 Hz), 7.29 - 7.48 (8 H, m), 7.58 (2 H, d, J=7.5 Hz), 8.12 (3 H, br. s.)

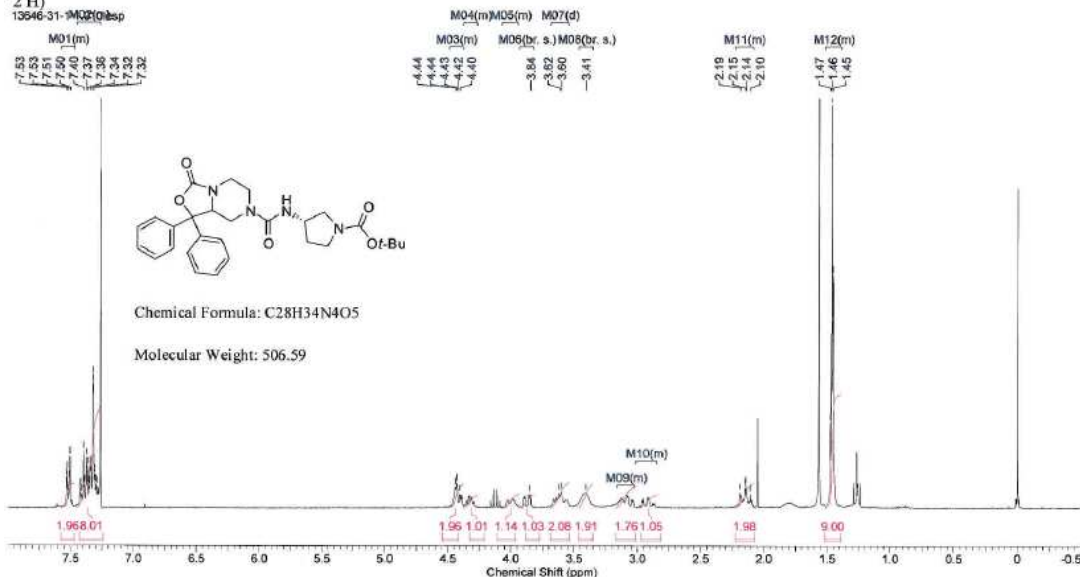
RTI
INTERNATIONAL
NMR Laboratory

Date Stamp 04 Apr 2013 13:21:04
Origin spect
Pulse Sequence zg
Solvent DMSO-d6
Number of Transients 128
Sweep Width (Hz) 6172.46
Acquisition Time (sec) 2.6542
Receiver Gain 256.00
Temperature (degree C) 27.000
Nucleus 1H
Frequency (MHz) 300.13
Spectrum Offset (Hz) 1853.6180
Original Points Count 16384

Compound 28

Acquisition Time (sec)	2.6542	Comment	14646-31-1-1	Date	13 Mar 2013 10:56:00
Date Stamp	13 Mar 2013 10:56:00			File Name	C:\Documents and Settings\chassier\My Documents\WNR\13646-31-1-1\10\fid
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	128
Original Points Count	16384	Owner	nmrUSER	Points Count	16384
Receiver Gain	1290.20	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1841.6641	Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46
				Temperature (degree C)	27.000

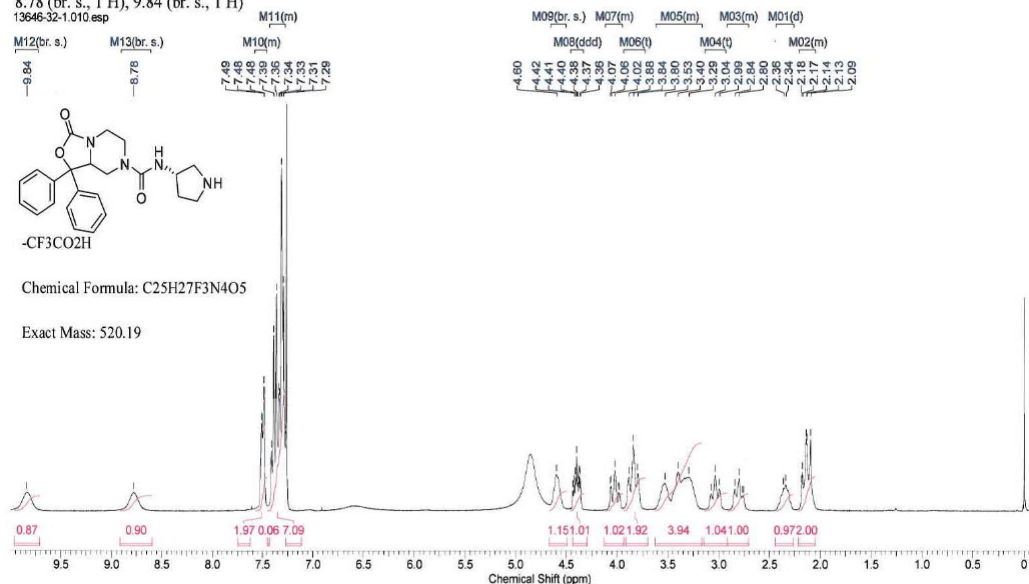
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 1.39 - 1.52 (m, 9 H), 2.08 - 2.23 (m, 2 H), 2.85 - 3.01 (m, 1 H), 3.02 - 3.16 (m, 2 H), 3.41 (br. s., 2 H), 3.61 (d, $J=5.65$ Hz, 2 H), 3.84 (br. s., 1 H), 3.94 - 4.07 (m, 1 H), 4.27 - 4.38 (m, 1 H), 4.38 - 4.49 (m, 2 H), 7.27 - 7.46 (m, 8 H), 7.47 - 7.57 (m, 2 H)



Compound 29

Acquisition Time (sec)	2.6542	Comment	13646-32-1	Date	18 Mar 2013 15:09:52
Date Stamp	18 Mar 2013 15:09:52			File Name	C:\Documents and Settings\chassier\My Documents\WNR\13646-32-1\10\fid
Frequency (MHz)	300.13	Nucleus	¹ H	Number of Transients	128
Original Points Count	16384	Owner	nmrUSER	Points Count	16384
Receiver Gain	1290.20	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1841.5530	Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46
				Temperature (degree C)	27.000

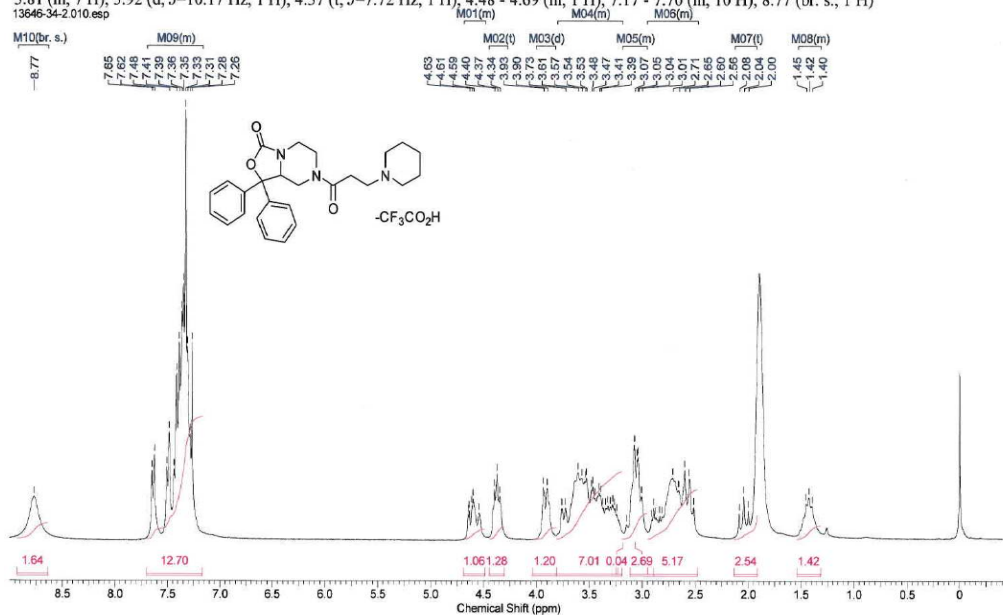
¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 2.05 - 2.21 (m, 2 H), 2.35 (d, $J=7.16$ Hz, 1 H), 2.71 - 2.92 (m, 1 H), 3.03 (t, $J=12.24$ Hz, 1 H), 3.17 - 3.6 4 H), 3.84 (t, $J=13.00$ Hz, 2 H), 3.95 - 4.12 (m, 1 H), 4.40 (ddd, $J=11.02, 7.44, 3.77$ Hz, 1 H), 4.60 (br. s., 1 H), 7.28 - 7.43 (m, 8 H), 7.45 - 7.58 (m, 2 H), 8.78 (br. s., 1 H), 9.84 (br. s., 1 H)



Compound 30

Acquisition Time (sec)	2.6542	Comment	13646-34-2	Date	02 Apr 2013 17:15:44
Date Stamp	02 Apr 2013 17:15:44	File Name	C:\Documents and Settings\chassier\My Documents\NMR\13646-34-2\10\fid		
Frequency (MHz)	300.13	Nucleus	1H	Number of Transients	128
Original Points Count	16384	Owner	nmrUSER	Points Count	16384
Receiver Gain	406.40	SW(cyclical) (Hz)	6172.84	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1842.1055	Spectrum Type	STANDARD	Sweep Width (Hz)	6172.46
				Temperature (degree C)	27.000

¹H NMR (300 MHz, CHLOROFORM-d) δ ppm 1.32 - 1.53 (m, 1 H), 2.04 (t, *J*=13.00 Hz, 2 H), 2.48 - 2.96 (m, 5 H), 2.96 - 3.19 (m, 3 H), 3.19 - 3.81 (m, 7 H), 3.92 (d, *J*=10.17 Hz, 1 H), 4.37 (t, *J*=7.72 Hz, 1 H), 4.48 - 4.69 (m, 1 H), 7.17 - 7.70 (m, 10 H), 8.77 (br. s., 1 H)



RP Analytical HPLC chromatograms:

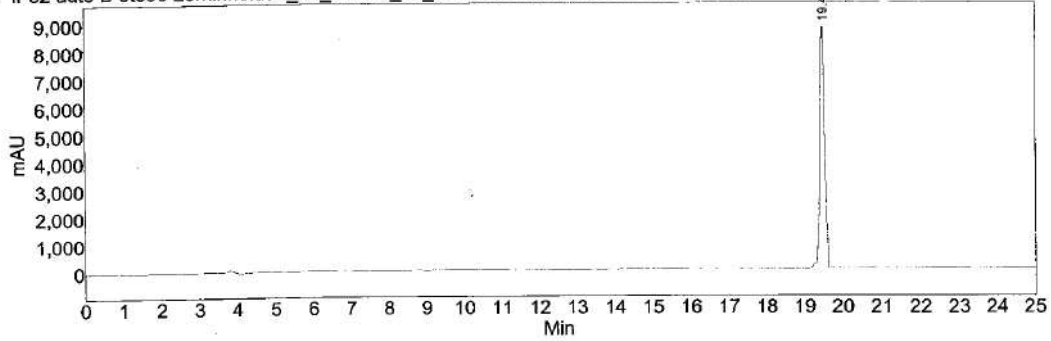
Compound 1 (SHA-68)

Chromatogram : 13646-25 fr 32 auto B 5to95 20minhold52_26_2013 6_31_12 PM

System : autosampler Prostar 410-210-335-ELSD
 Method : auto B 5 to 95 over 20 min
 User : Carla

Acquired : 2/26/2013 8:32:22 PM
 Processed : 2/27/2013 12:30:01 PM
 Printed : 9/25/2013 5:08:46 PM

13646-25 fr 32 auto B 5to95 20minhold52_26_2013 6_31_12 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time (Min)	Quantity [% Area]	Height (mAU)	Area (mAU.Min)	Area % [%]
1	UNKNOWN	19.47	100.00	8693.6	1280.3	100.000
Total			100.00	8693.6	1280.3	100.000

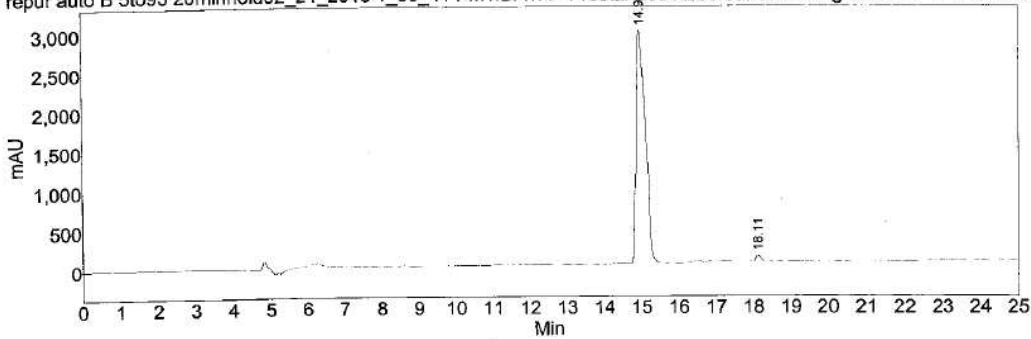
Compound 2 (RTI-118)

Chromatogram : 13646-8-1 repur auto B 5to95 20minhold52_21_2013 1_50_41 PI

System : autosampler Prostar 410-210-335-ELSD
 Method : auto B 5 to 95 over 20 min
 User : Carla

Acquired : 2/21/2013 1:51:51 PM
 Processed : 2/21/2013 2:30:21 PM
 Printed : 9/25/2013 1:27:14 PM

13646-8-1 repur auto B 5to95 20minhold52_21_2013 1_50_41 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL0605907



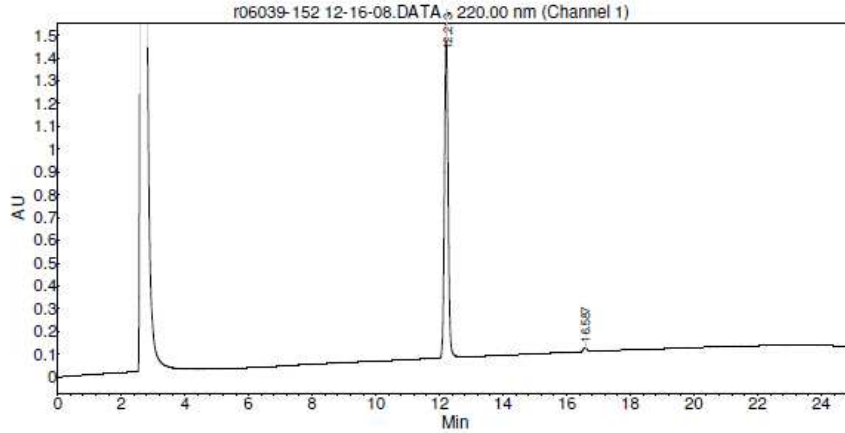
Index	Name	Time (Min)	Quantity [% Area]	Height (mAU)	Area (mAU.Min)	Area % [%]
1	UNKNOWN	14.95	99.24	2963.2	844.9	99.238
2	UNKNOWN	18.11	0.76	63.7	6.5	0.762
Total			100.00	3026.9	851.4	100.000

Compound 6

Chromatogram : r06039-152 12-16-08_channel2

System : NOT DEFINED
 Method : NOT DEFINED
 User : Tyler Graf

Acquired : 12/17/2008 3:14:30 AM
 Processed : 12/13/2013 3:19:38 PM
 Printed : 12/13/2013 3:19:48 PM



Peak results :

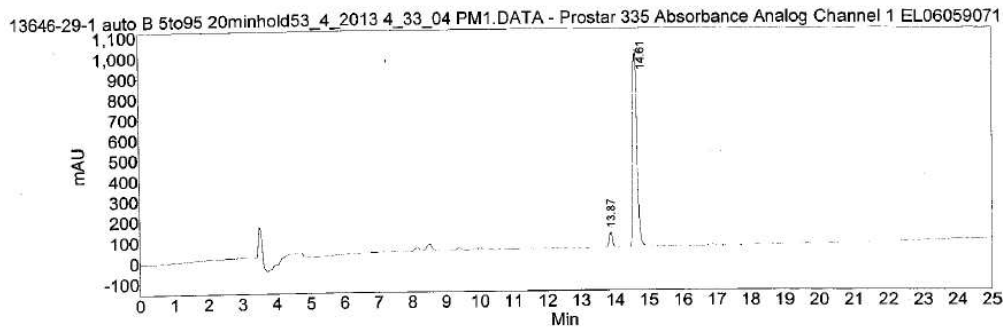
Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	12.213	98.71	0.13	98.657	1.4	11.3	98.712
2	UNKNOWN	16.587	1.29	0.12	1.343	0.0	0.1	1.288
Total			100.00		100.000	1.4	11.5	100.000

Compound 7

Chromatogram : 13646-29-1 auto B 5to95 20minhold53_4_2013 4_33_04 PM1_ch

System : autosampler Prostar 410-210-335-ELSD
 Method : auto B 5 to 95 over 20 min
 User : Carla

Acquired : 3/4/2013 4:34:14 PM
 Processed : 3/4/2013 5:42:41 PM
 Printed : 9/25/2013 5:11:56 PM



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	13.87	4.95	71.5	7.1	4.955
2	UNKNOWN	14.61	95.05	956.0	136.0	95.045
Total			100.00	1027.5	143.1	100.000

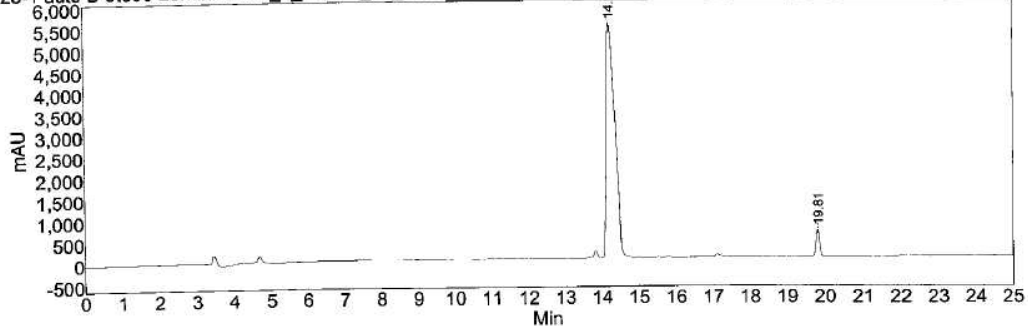
Compound 8

Chromatogram : 13646-28-1 auto B 5to95 20minhold53_1_2013 4_06_01 PM1_ch

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 3/1/2013 4:07:08 PM
Processed : 3/1/2013 4:53:57 PM
Printed : 12/27/2013 3:38:07 PM

13646-28-1 auto B 5to95 20minhold53_1_2013 4_06_01 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time (Min)	Quantity (% Area)	Height (mAU)	Area (mAU.Min)	Area % (%)
1	UNKNOWN	14.20	95.98	5462.3	1483.1	95.979
2	UNKNOWN	19.81	4.02	612.0	62.1	4.021
Total			100.00	6074.3	1545.2	100.000

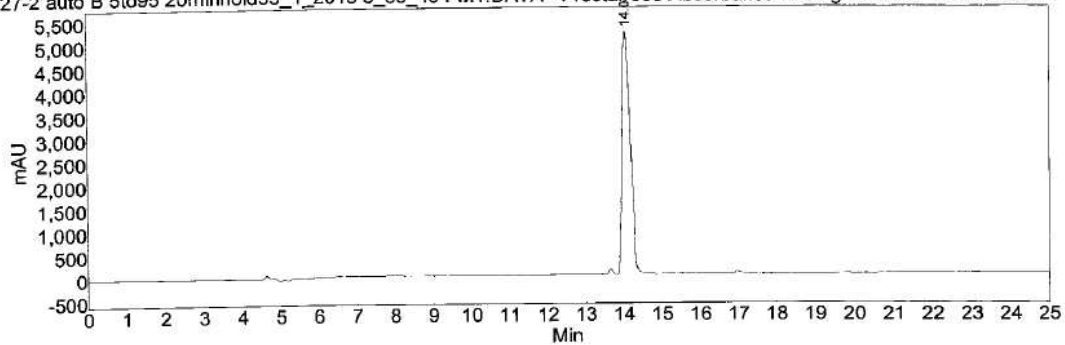
Compound 9

Chromatogram : 13646-27-2 auto B 5to95 20minhold53_1_2013 3_33_45 PM1_ch

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 3/1/2013 3:34:54 PM
Processed : 3/1/2013 4:54:42 PM
Printed : 12/27/2013 3:39:14 PM

13646-27-2 auto B 5to95 20minhold53_1_2013 3_33_45 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time (Min)	Quantity (% Area)	Height (mAU)	Area (mAU.Min)	Area % (%)
1	UNKNOWN	14.04	100.00	5182.4	1301.8	100.000
Total			100.00	5182.4	1301.8	100.000

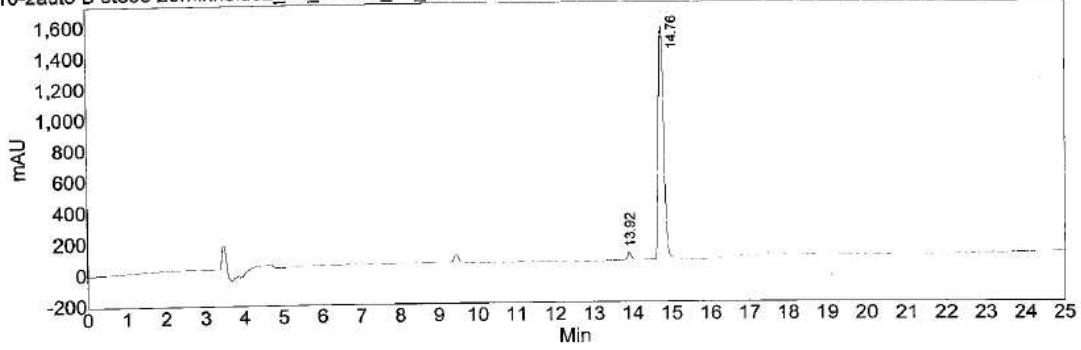
Compound 10

Chromatogram : 13545-10-2auto B 5to95 20minhold52_18_2013 7_32_47 PM1_ct

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 2/18/2013 7:33:58 PM
Processed : 2/19/2013 11:02:43 AM
Printed : 9/25/2013 5:15:45 PM

13545-10-2auto B 5to95 20minhold52_18_2013 7_32_47 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	13.92	2.13	53.5	5.1	2.131
2	UNKNOWN	14.76	97.87	1502.3	235.7	97.869
Total			100.00	1555.8	240.8	100.000

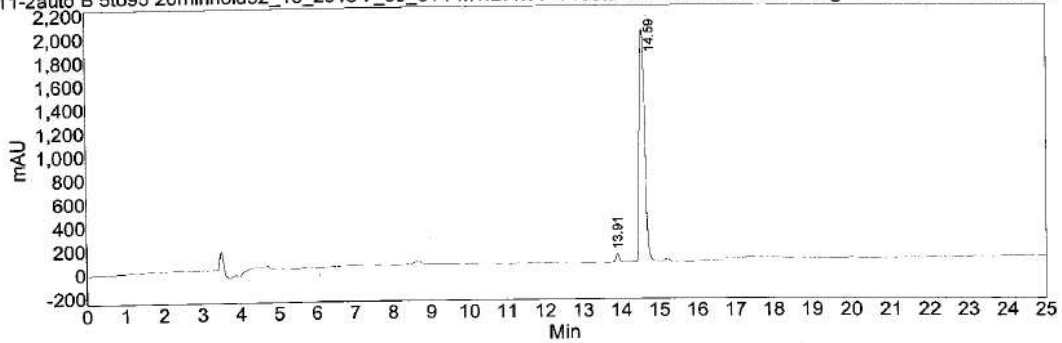
Compound 11

Chromatogram : 13646-11-2auto B 5to95 20minhold52_18_2013 7_00_31 PM1_ct

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 2/18/2013 7:01:40 PM
Processed : 2/19/2013 11:03:54 AM
Printed : 9/25/2013 5:16:54 PM

13646-11-2auto B 5to95 20minhold52_18_2013 7_00_31 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	13.91	2.08	70.4	6.7	2.077
2	UNKNOWN	14.59	97.92	1954.2	318.0	97.923
Total			100.00	2034.7	324.7	100.000

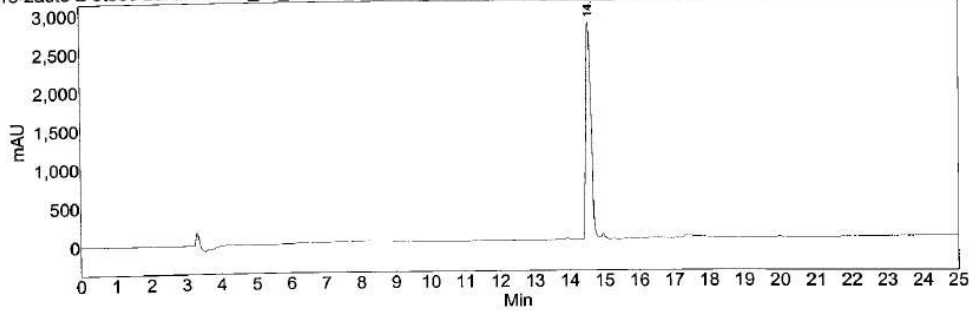
Compound 12

Chromatogram : 13646-13-2auto B 5to95 20minhold52_19_2013 1_52_01 PM1_ct

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 2/19/2013 1:53:09 PM
Processed : 2/19/2013 2:32:14 PM
Printed : 12/27/2013 3:40:35 PM

13646-13-2auto B 5to95 20minhold52_19_2013 1_52_01 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time (Min)	Quantity (% Area)	Height (mAU)	Area (mAU Min)	Area (%)
1	UNKNOWN	14.55	100.00	2806.6	488.4	100.000
Total			100.00	2806.6	488.4	100.000

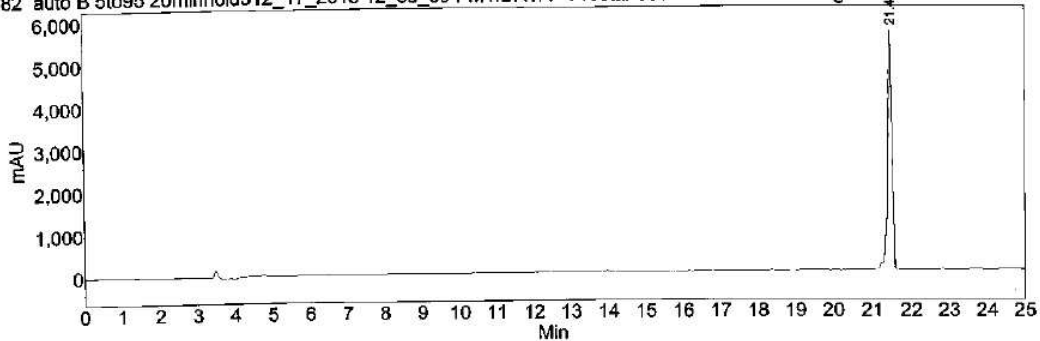
Compound 13

Chromatogram : R06039-282 auto B 5to95 20minhold512_17_2013 12_56_09 PM

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 12/17/2013 12:57:17 PM
Processed : 12/20/2013 3:07:35 PM
Printed : 12/23/2013 11:45:16 AM

R06039-282 auto B 5to95 20minhold512_17_2013 12_56_09 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time (Min)	Quantity (% Area)	Height (mAU)	Area (mAU Min)	Area (%)
1	UNKNOWN	21.48	100.00	5578.1	721.8	100.000
Total			100.00	5578.1	721.8	100.000

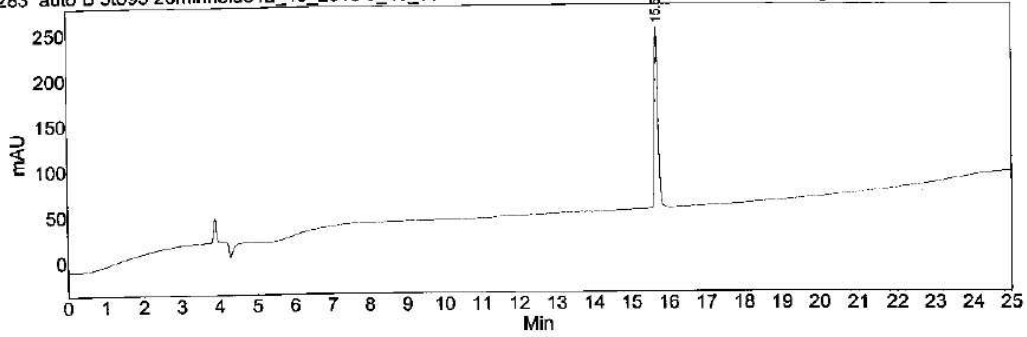
Compound 14

Chromatogram : R06039-283 auto B 5to95 20minhold512_19_2013 3_45_58 PM1

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 12/19/2013 3:47:06 PM
Processed : 12/19/2013 4:19:53 PM
Printed : 12/23/2013 11:44:48 AM

R06039-283 auto B 5to95 20minhold512_19_2013 3_45_58 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Time (min)	Quantity (%)	Height (mAU)	Area (mAU)	Area (%)
1	15.68	100.00	197.7	20.9	100.000
Total		100.00	197.7	20.9	100.000

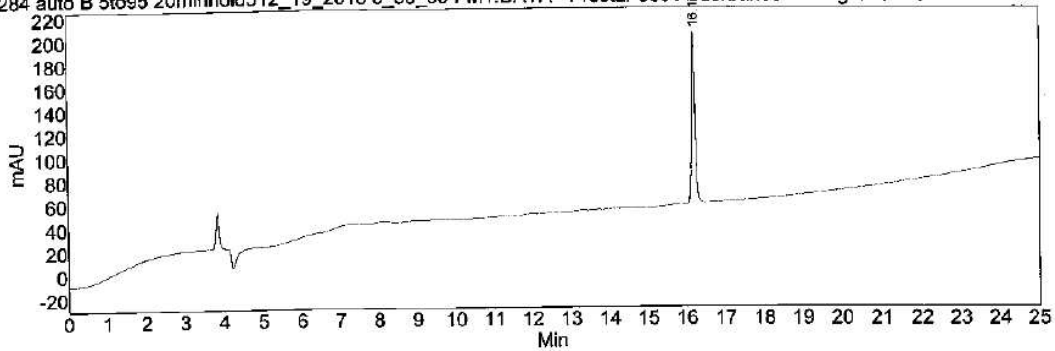
Compound 15

Chromatogram : R06039-284 auto B 5to95 20minhold512_19_2013 5_55_00 PM1

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 12/19/2013 5:56:09 PM
Processed : 12/20/2013 9:18:59 AM
Printed : 12/23/2013 11:44:23 AM

R06039-284 auto B 5to95 20minhold512_19_2013 5_55_00 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



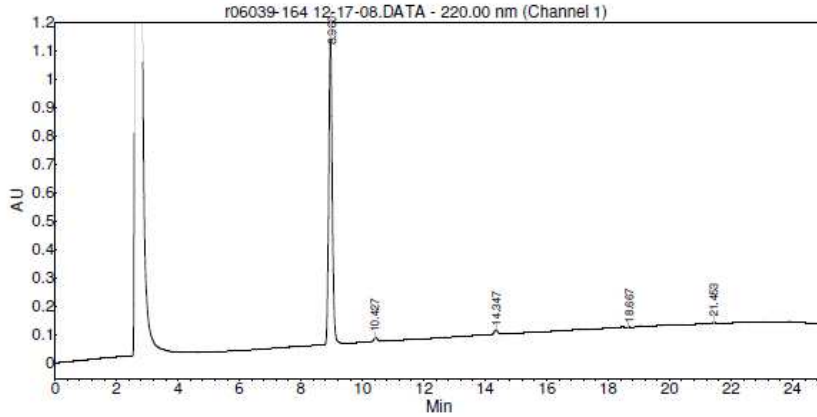
Index	Time (min)	Quantity (%)	Height (mAU)	Area (mAU)	Area (%)
1	16.17	100.00	146.0	15.4	100.000
Total		100.00	146.0	15.4	100.000

Compound 16

Chromatogram : r06039-164 12-17-08_channel2

System : NOT DEFINED
Method : NOT DEFINED
User : Tyler Graf

Acquired : 12/17/2008 5:49:11 AM
Processed : 12/13/2013 3:22:37 PM
Printed : 12/13/2013 3:22:44 PM



Peak results :

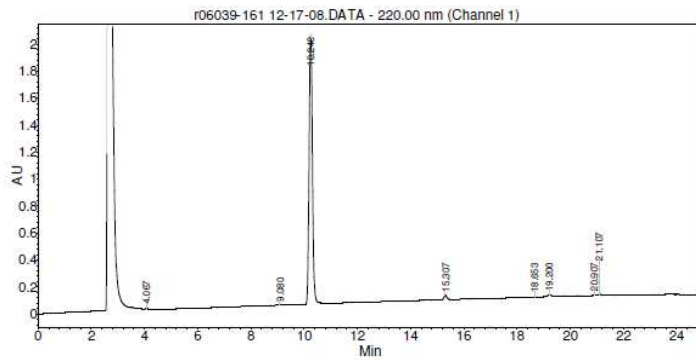
Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	8.960	96.98	0.12	96.679	1.1	8.6	96.984
2	UNKNOWN	10.427	1.29	0.12	1.332	0.0	0.1	1.286
3	UNKNOWN	14.347	1.36	0.12	1.413	0.0	0.1	1.365
4	UNKNOWN	18.667	0.09	0.07	0.183	0.0	0.0	0.086
5	UNKNOWN	21.453	0.28	0.09	0.394	0.0	0.0	0.280
Total			100.00		100.000	1.1	8.9	100.000

Compound 17

Chromatogram : r06039-161 12-17-08_channel2

System : NOT DEFINED
Method : NOT DEFINED
User : Tyler Graf

Acquired : 12/17/2008 5:18:16 AM
Processed : 12/13/2013 3:21:38 PM
Printed : 12/13/2013 3:21:54 PM



Peak results :

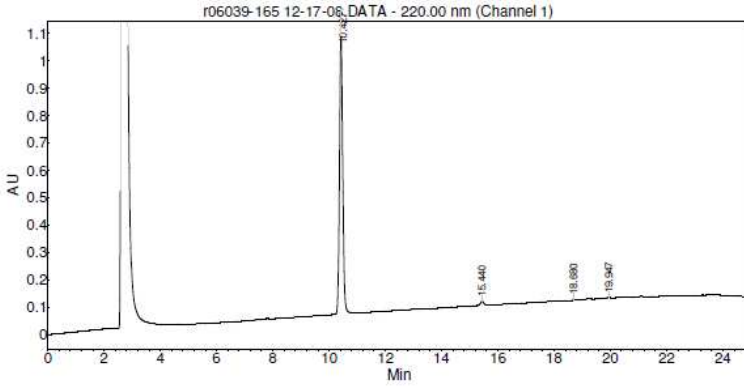
Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	4.067	0.10	0.07	0.175	0.0	0.0	0.099
2	UNKNOWN	9.080	0.15	0.11	0.198	0.0	0.0	0.145
3	UNKNOWN	10.240	96.93	0.13	96.612	2.0	16.9	96.933
4	UNKNOWN	15.307	1.48	0.12	1.639	0.0	0.3	1.478
5	UNKNOWN	18.653	0.04	0.08	0.095	0.0	0.0	0.042
6	UNKNOWN	19.200	0.72	0.18	0.870	0.0	0.1	0.717
7	UNKNOWN	20.907	0.12	0.10	0.175	0.0	0.0	0.123
8	UNKNOWN	21.107	0.46	0.10	0.535	0.0	0.1	0.461
Total			100.00		100.000	2.0	17.5	100.000

Compound 18

Chromatogram : r06039-165 12-17-08_channel2

System : NOT DEFINED
Method : NOT DEFINED
User : Tyler Graf

Acquired : 12/17/2008 6:20:05 AM
Processed : 12/13/2013 3:23:37 PM
Printed : 12/13/2013 3:30:13 PM



Peak results :

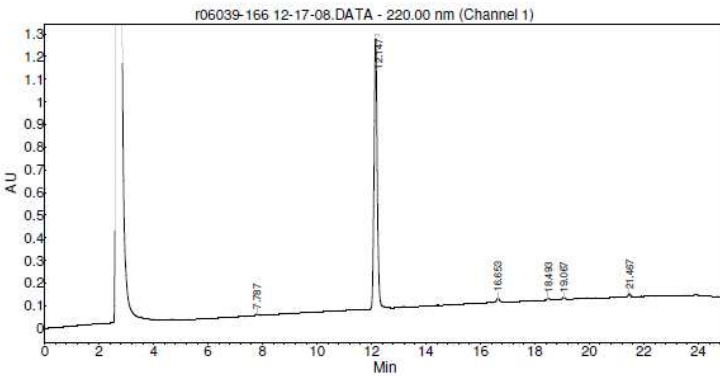
Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	10.427	98.32	0.12	98.063	1.0	8.1	98.320
2	UNKNOWN	15.440	1.39	0.12	1.438	0.0	0.1	1.392
3	UNKNOWN	18.680	0.09	0.07	0.200	0.0	0.0	0.090
4	UNKNOWN	19.947	0.20	0.09	0.299	0.0	0.0	0.198
Total			100.00		100.000	1.0	8.2	100.000

Compound 19

Chromatogram : r06039-166 12-17-08_channel2

System : NOT DEFINED
Method : NOT DEFINED
User : Tyler Graf

Acquired : 12/17/2008 6:50:59 AM
Processed : 12/13/2013 3:31:12 PM
Printed : 12/13/2013 3:31:22 PM



Peak results :

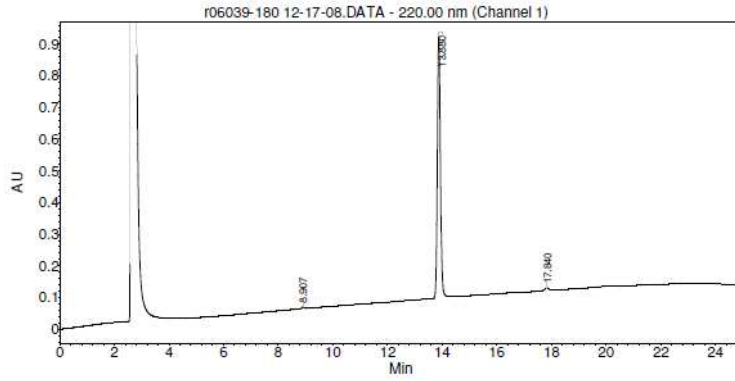
Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	7.787	0.37	0.11	0.450	0.0	0.0	0.367
2	UNKNOWN	12.147	98.34	0.12	96.649	1.2	9.7	98.337
3	UNKNOWN	16.653	1.33	0.12	1.397	0.0	0.1	1.331
4	UNKNOWN	18.483	0.52	0.10	0.654	0.0	0.1	0.518
5	UNKNOWN	19.067	0.66	0.10	0.802	0.0	0.1	0.660
6	UNKNOWN	21.467	0.79	0.09	1.048	0.0	0.1	0.786
Total			100.00		100.000	1.2	10.0	100.000

Compound 20

Chromatogram : r06039-180 12-17-08_channel2

System : NOT DEFINED
Method : NOT DEFINED
User : Tyler Graf

Acquired : 12/17/2008 9:25:35 AM
Processed : 12/13/2013 3:34:13 PM
Printed : 12/13/2013 3:40:41 PM



Peak results :

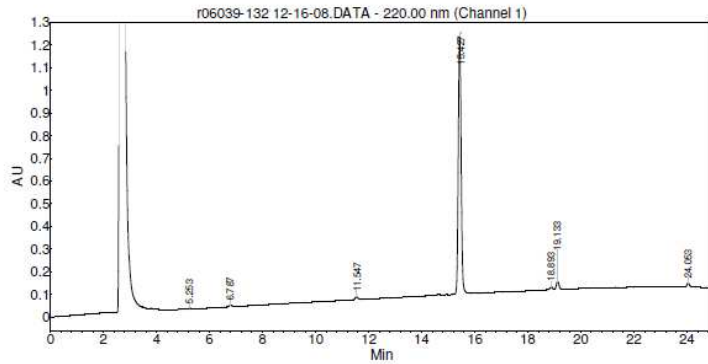
Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	8.907	0.46	0.10	0.569	0.0	0.0	0.458
2	UNKNOWN	13.880	98.54	0.12	98.549	0.8	6.4	98.542
3	UNKNOWN	17.840	1.00	0.14	0.883	0.0	0.1	1.000
Total			100.00		100.000	0.8	6.5	100.000

Compound 21

Chromatogram : r06039-132 12-16-08_channel2

System : NOT DEFINED
Method : NOT DEFINED
User : Tyler Graf

Acquired : 12/16/2008 11:06:56 PM
Processed : 12/13/2013 3:08:40 PM
Printed : 12/13/2013 3:12:57 PM



Peak results :

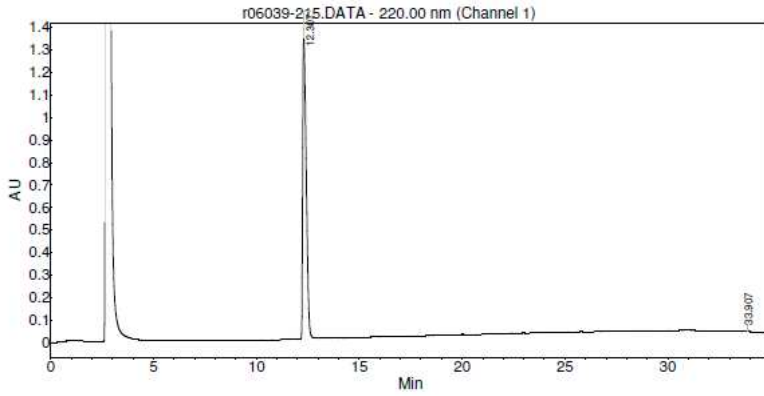
Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	5.253	0.19	0.09	0.270	0.0	0.0	0.189
2	UNKNOWN	6.787	0.58	0.13	0.566	0.0	0.1	0.581
3	UNKNOWN	11.547	1.02	0.12	1.012	0.0	0.1	1.018
4	UNKNOWN	15.427	92.95	0.11	92.805	1.1	8.4	92.961
5	UNKNOWN	18.893	1.28	0.12	1.085	0.0	0.1	1.280
6	UNKNOWN	18.133	2.92	0.11	2.959	0.0	0.1	2.918
7	UNKNOWN	24.053	1.05	0.09	1.303	0.0	0.1	1.054
Total			100.00		100.000	1.2	9.0	100.000

Compound 22

Chromatogram : r06039-215_channel2

System : NOT DEFINED
Method : NOT DEFINED
User : Tyler Graf

Acquired : 12/19/2008 5:53:02 AM
Processed : 12/13/2013 3:44:42 PM
Printed : 12/13/2013 3:44:48 PM



Peak results :

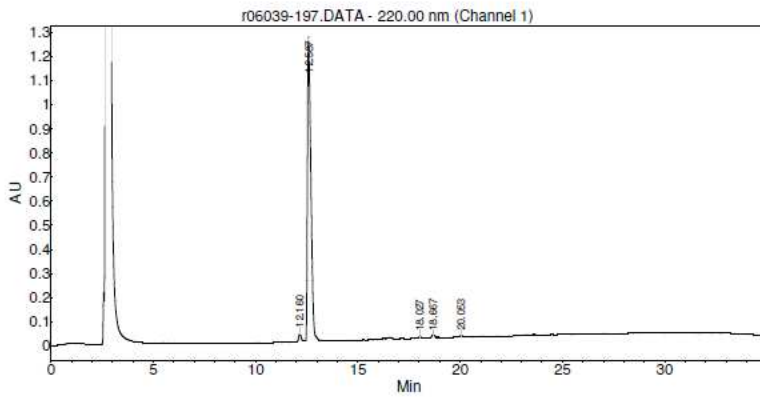
Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	12.307	99.82	0.19	99.641	1.3	15.8	99.816
2	UNKNOWN	33.907	0.18	0.09	0.359	0.0	0.0	0.184
Total			100.00		100.000	1.3	15.8	100.000

Compound 23

Chromatogram : r06039-197_channel2

System : NOT DEFINED
Method : NOT DEFINED
User : Tyler Graf

Acquired : 12/19/2008 3:50:13 AM
Processed : 12/13/2013 3:43:18 PM
Printed : 12/13/2013 3:43:25 PM



Peak results :

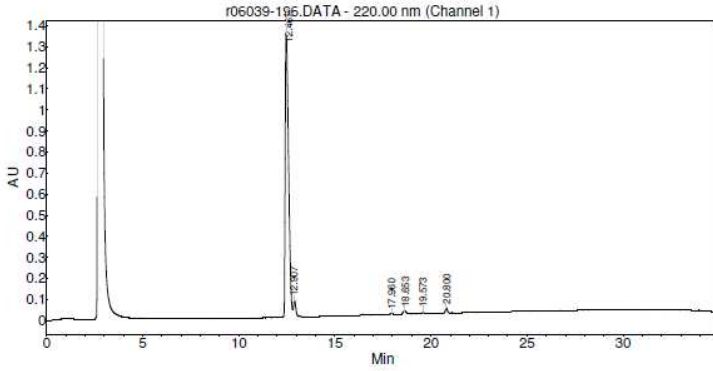
Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	12.160	1.96	0.13	2.514	0.0	0.3	1.956
2	UNKNOWN	12.587	96.67	0.19	95.678	1.2	14.8	96.672
3	UNKNOWN	18.027	0.36	0.13	0.514	0.0	0.1	0.361
4	UNKNOWN	18.667	0.77	0.17	0.925	0.0	0.1	0.766
5	UNKNOWN	20.053	0.25	0.13	0.369	0.0	0.0	0.245
Total			100.00		100.000	1.3	15.3	100.000

Compound 24

Chromatogram : r06039-196_channel2

System : NOT DEFINED
 Method : NOT DEFINED
 User : Tyler Graf

Acquired : 12/19/2008 3:09:19 AM
 Processed : 12/13/2013 3:42:20 PM
 Printed : 12/13/2013 3:42:34 PM



Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Width 50% [Min]	Height % [%]	Height [AU]	Area [AU.Sec]	Area % [%]
1	UNKNOWN	12.467	93.76	0.20	91.439	1.3	18.3	93.762
2	UNKNOWN	12.907	3.89	0.15	5.182	0.1	0.7	3.893
3	UNKNOWN	17.960	0.36	0.14	0.486	0.0	0.1	0.356
4	UNKNOWN	18.653	0.82	0.17	0.948	0.0	0.1	0.825
5	UNKNOWN	19.573	0.19	0.11	0.324	0.0	0.0	0.188
6	UNKNOWN	20.800	0.98	0.11	1.620	0.0	0.2	0.978
Total			100.00		100.000	1.5	17.4	100.000

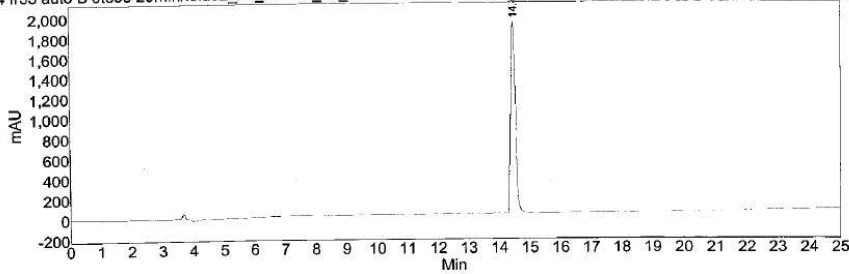
Compound 25

Chromatogram : 13646-24 fr55 auto B 5to95 20minhold52_26_2013 5_58_57 PM1

System : autosampler Prostar 410-210-335-ELSD
 Method : auto B 5 to 95 over 20 min
 User : Carla

Acquired : 2/26/2013 6:00:05 PM
 Processed : 2/27/2013 12:29:25 PM
 Printed : 9/25/2013 5:20:36 PM

13646-24 fr55 auto B 5to95 20minhold52_26_2013 5_58_57 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	14.47	100.00	1894.2	334.1	100.000
Total			100.00	1894.2	334.1	100.000

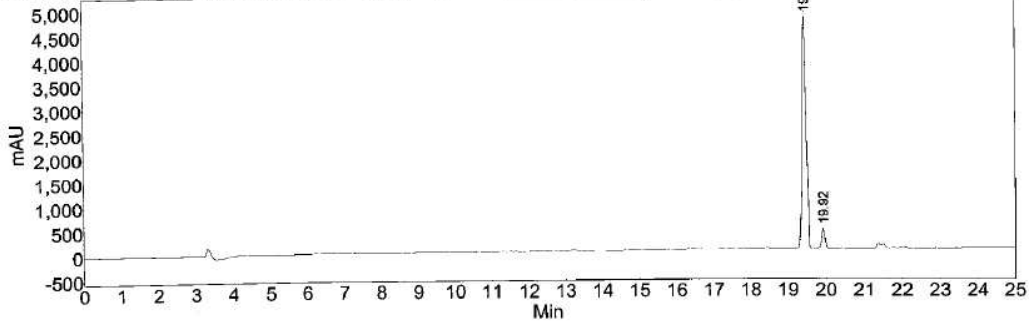
Compound 26

Chromatogram : 13646-11-1 auto B 5to95 20minhold52_20_2013 4_15_35 PM1_c

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 2/20/2013 4:16:43 PM
Processed : 12/27/2013 3:42:46 PM
Printed : 12/27/2013 3:43:00 PM

13646-11-1 auto B 5to95 20minhold52_20_2013 4_15_35 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	19.45	95.35	4731.5	606.5	95.346
2	UNKNOWN	19.92	4.65	354.5	29.6	4.654
Total			100.00	5085.9	636.1	100.000

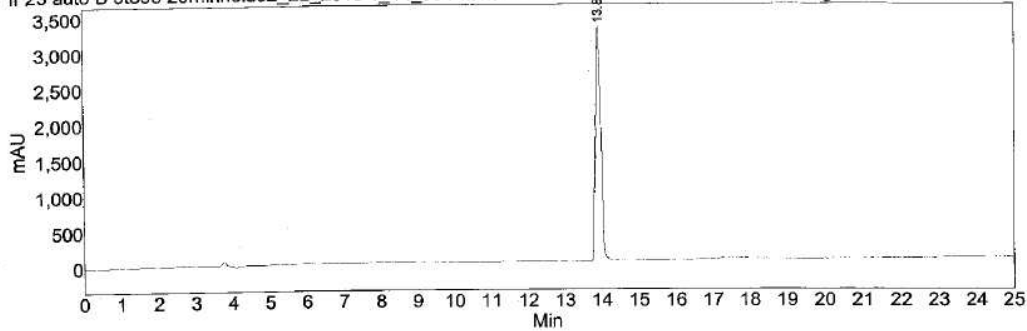
Compound 27

Chromatogram : 13646-21 fr 23 auto B 5to95 20minhold52_22_2013 1_44_35 PM'

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 2/22/2013 1:45:43 PM
Processed : 2/22/2013 2:27:55 PM
Printed : 9/25/2013 5:22:06 PM

13646-21 fr 23 auto B 5to95 20minhold52_22_2013 1_44_35 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	13.39	100.00	3258.7	541.9	100.000
Total			100.00	3258.7	541.9	100.000

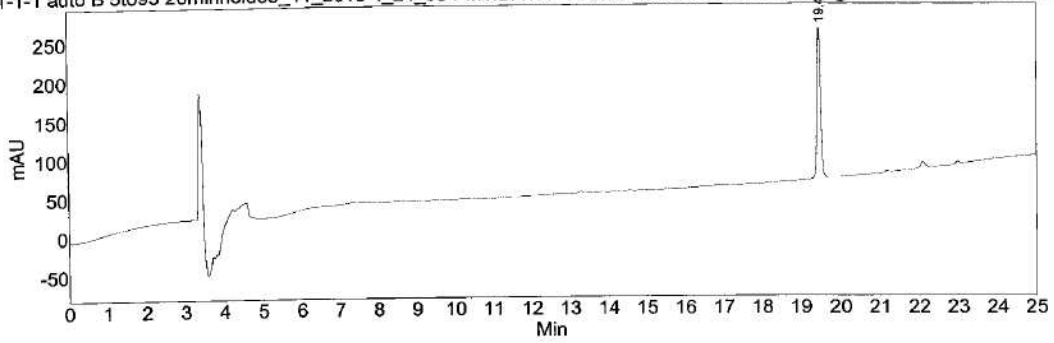
Compound 28

Chromatogram : 13646-31-1-1 auto B 5to95 20minhold53_11_2013 4_24_58 PM1_

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 3/11/2013 4:26:08 PM
Processed : 3/11/2013 5:03:36 PM
Printed : 12/27/2013 3:44:01 PM

13646-31-1-1 auto B 5to95 20minhold53_11_2013 4_24_58 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	19.48	100.00	193.2	20.5	100.000
Total			100.00	193.2	20.5	100.000

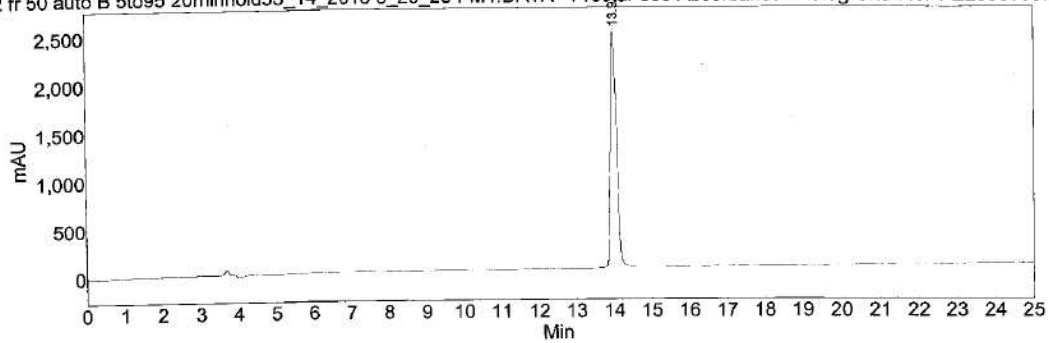
Compound 29

Chromatogram : 13646-32 fr 50 auto B 5to95 20minhold53_14_2013 6_29_28 PM1_

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 3/14/2013 6:30:38 PM
Processed : 3/15/2013 10:02:21 AM
Printed : 9/25/2013 5:23:52 PM

13646-32 fr 50 auto B 5to95 20minhold53_14_2013 6_29_28 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06059071



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	13.97	100.00	2442.8	467.3	100.000
Total			100.00	2442.8	467.3	100.000

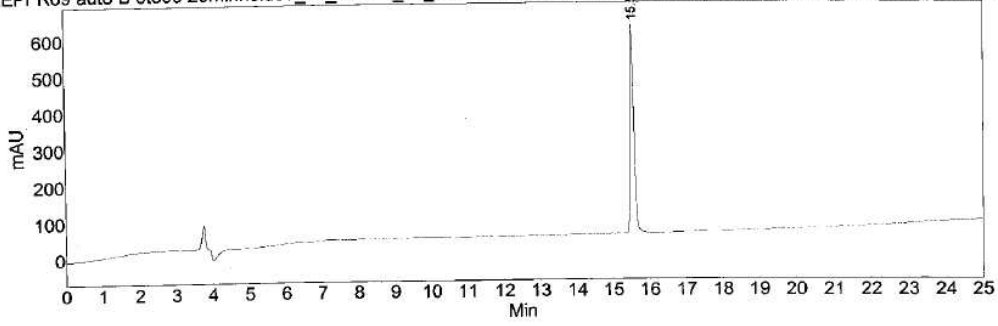
Compound 30

Chromatogram : 13646-34PREPFR69 auto B 5to95 20minhold53_28_2013 1_49_1

System : autosampler Prostar 410-210-335-ELSD
Method : auto B 5 to 95 over 20 min
User : Carla

Acquired : 3/28/2013 1:50:22 PM
Processed : 3/28/2013 3:43:32 PM
Printed : 9/25/2013 5:25:32 PM

346-34PREPFR69 auto B 5to95 20minhold53_28_2013 1_49_14 PM1.DATA - Prostar 335 Absorbance Analog Channel 1 EL060591



Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	15.52	100.00	569.6	70.1	100.000
Total			100.00	569.6	70.1	100.000