Supporting Information

The Journal of Organic Chemistry

Synthesis of Substituted 2,3-Benzodiazepines

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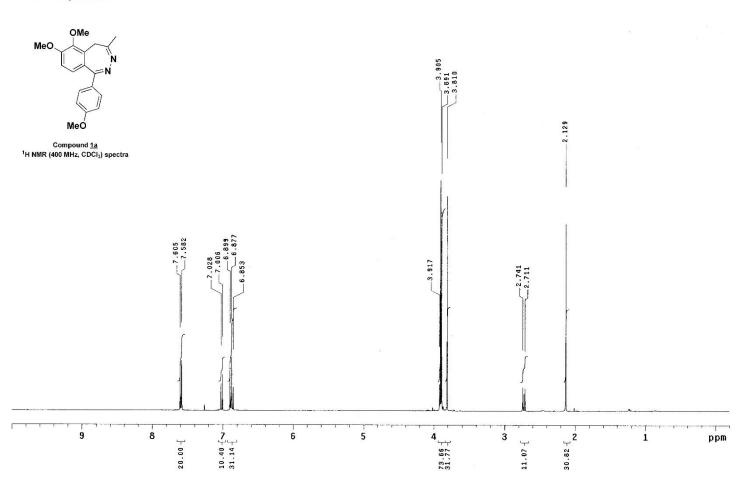
Email: mychang@kmu.edu.tw

I.	Scanned Photocopies of ¹ H and ¹³ C NMR Spectra for Compounds 1a~1aa	S-2~S-109
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Total Pages of Supporting Information : (243).

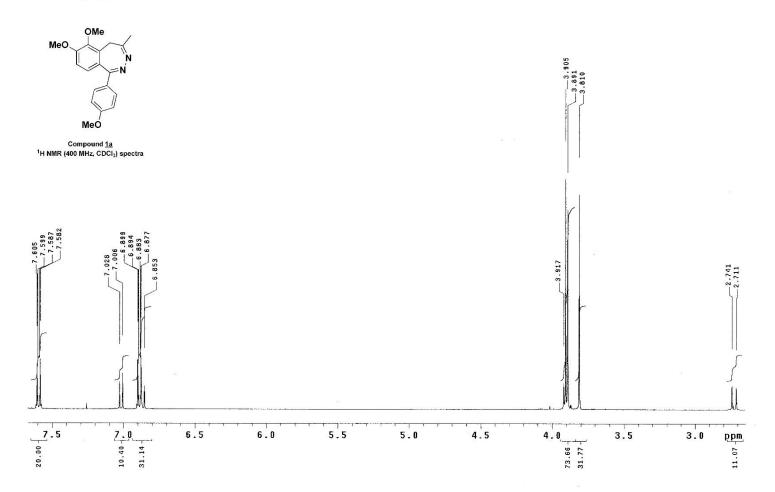
YR1040701

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Jul 2 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions

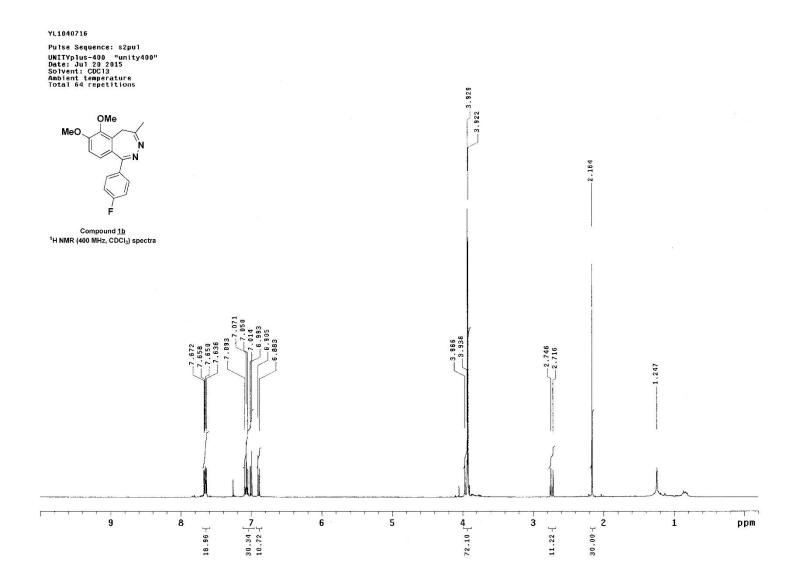


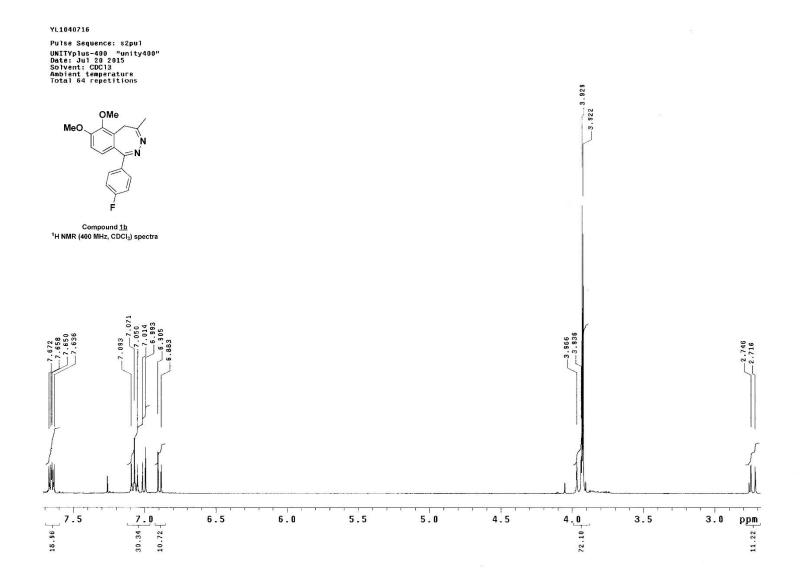
YR1040701

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Jul 2 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions



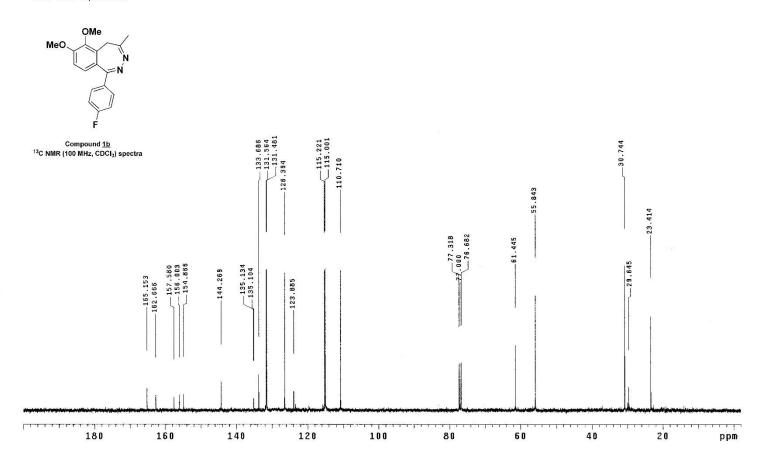
YR1040701 Pulse Sequence: s2pul UNITYPIUS-400 "unity400" Date: Jul 2 2015 Solvent: CDCl3 Ambient temperature Total 1632 repetitions OMe MeO. MeO Compound 1a 130.935 ¹³C NMR (100 MHz, CDCI₃) spectra 113.394 445 55.714 126.455 -30.577 110. 23.308 61.316 160.908 ____157.7 124.044 144.041 Farrit trate 140 120 100 80 60 180 160 40 20 ppm





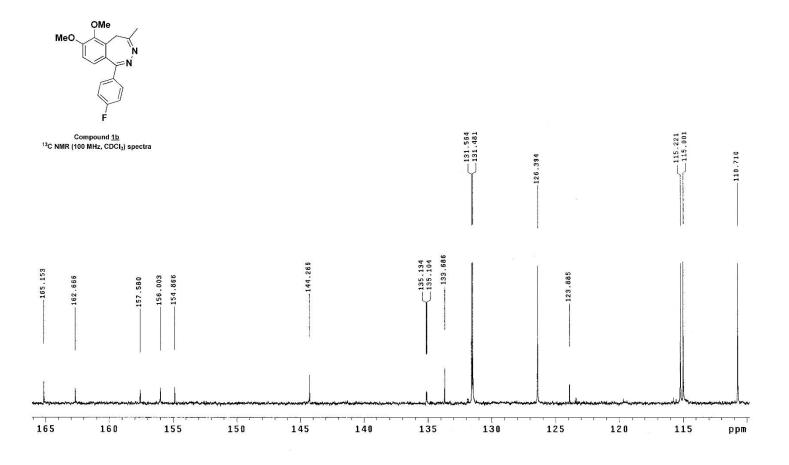
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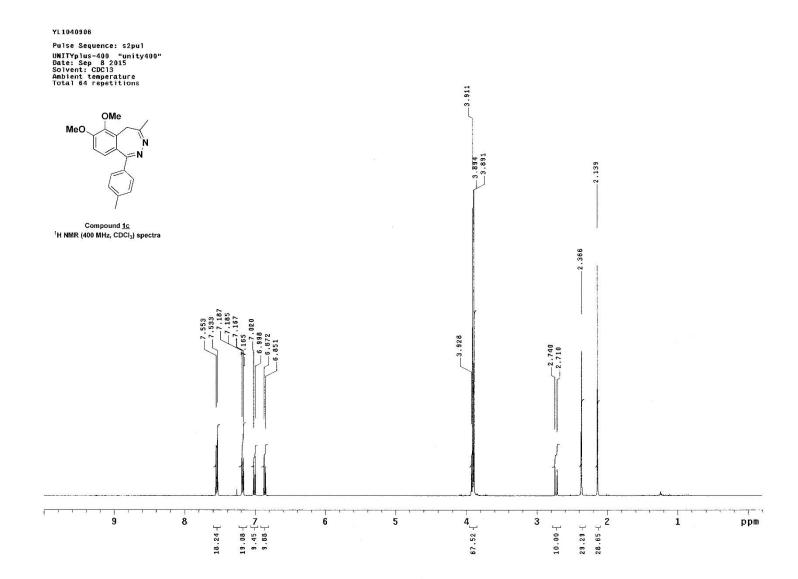
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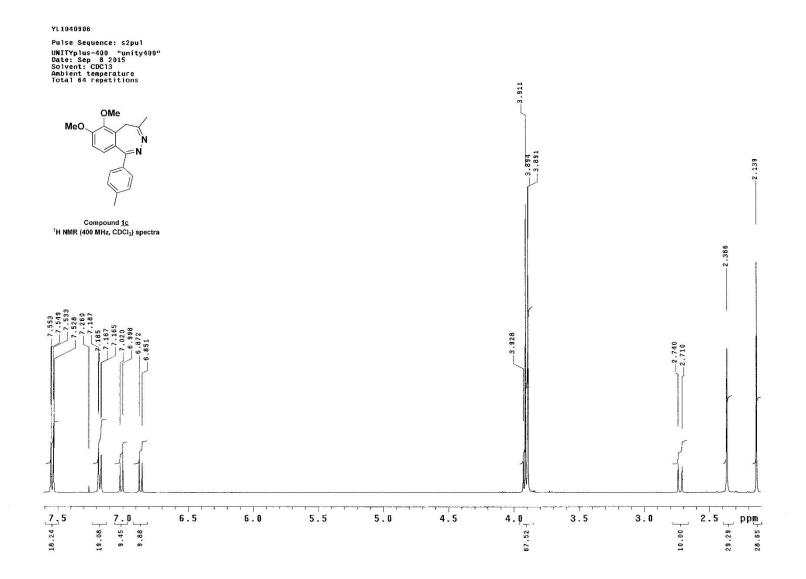


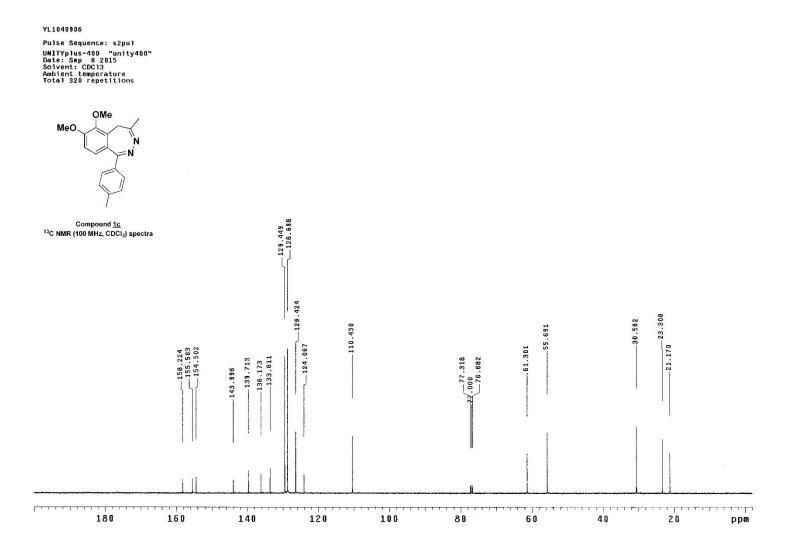
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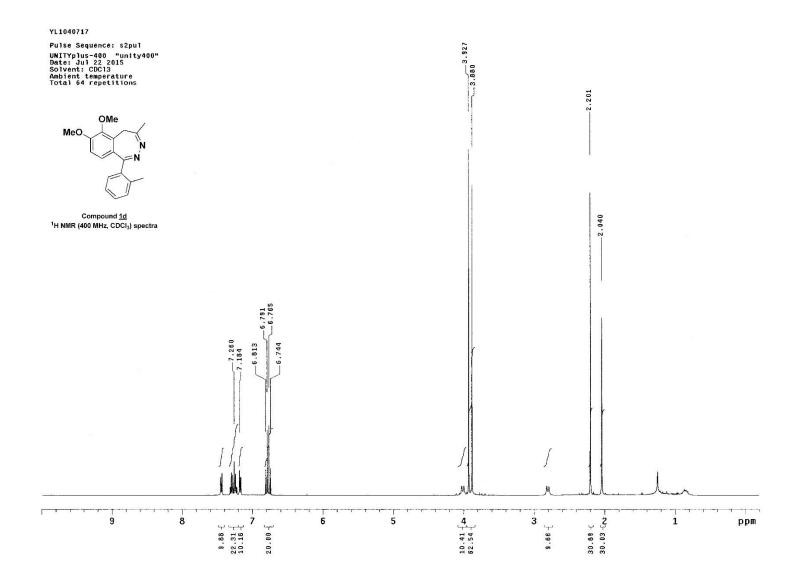
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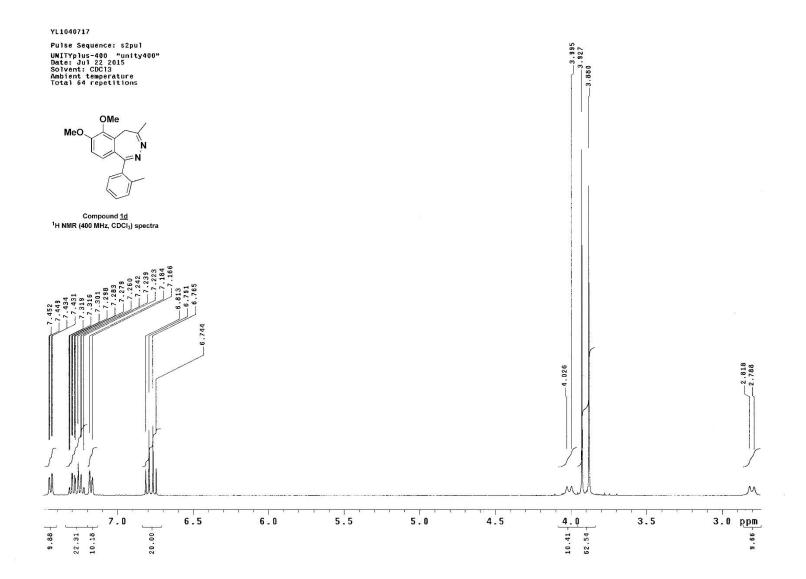








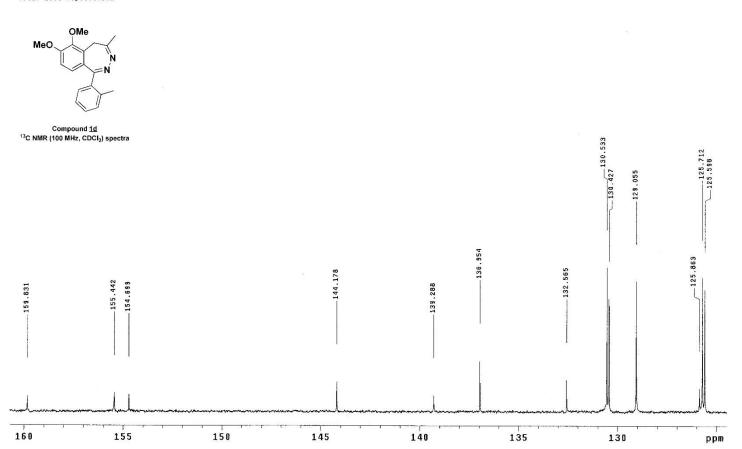


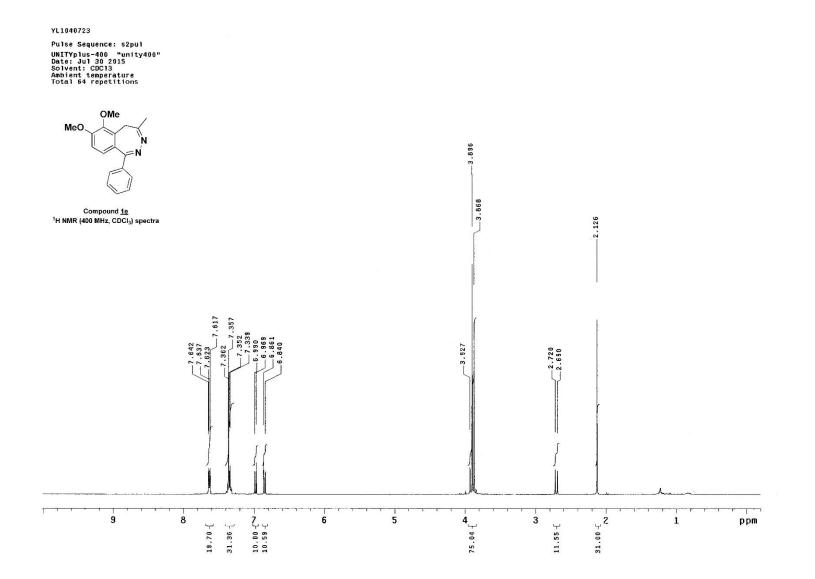


YL1040717 Pulse Sequence: s2pul UNITY plus-400 "unity400" Date: Jul 22 2015 Solvent: CDC13 Ambient temperature Total 2080 repetitions OMe MeO. Compound <u>1d</u> ¹³C NMR (100 MHz, CDCI₃) spectra 30,600 110.930 55.782 23.611 61.483 20.162 77.318 .000 76.689 -155.442-154.699 29.630 159.831 1 11 T Т 11 11 1 11 180 160 140 120 100 80 60 40 28 ppm

YL1040717

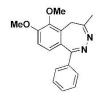
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Jul 22 2015 Solvent: CDCl3 Ambient temperature Total 2080 repetitions



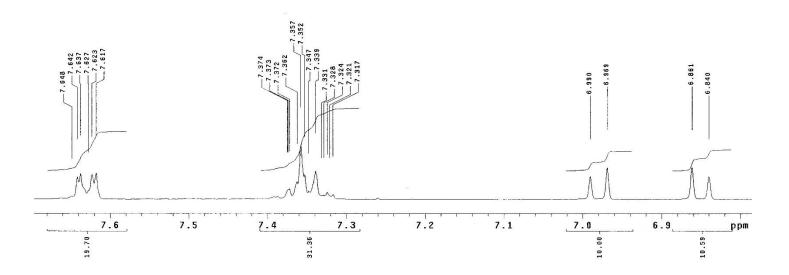


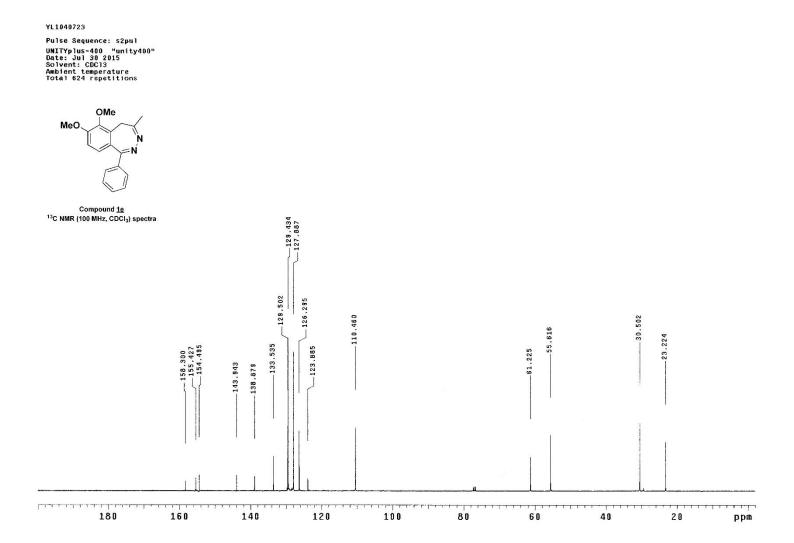
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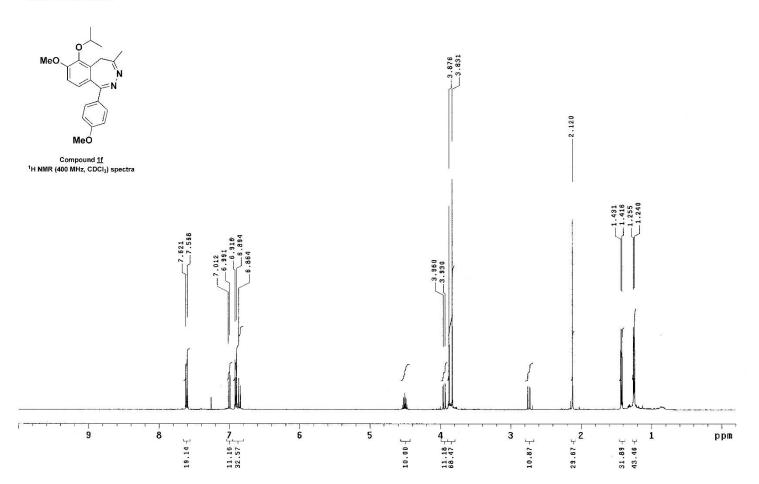
Compound <u>1e</u> ¹H NMR (400 MHz, CDCI₃) spectra





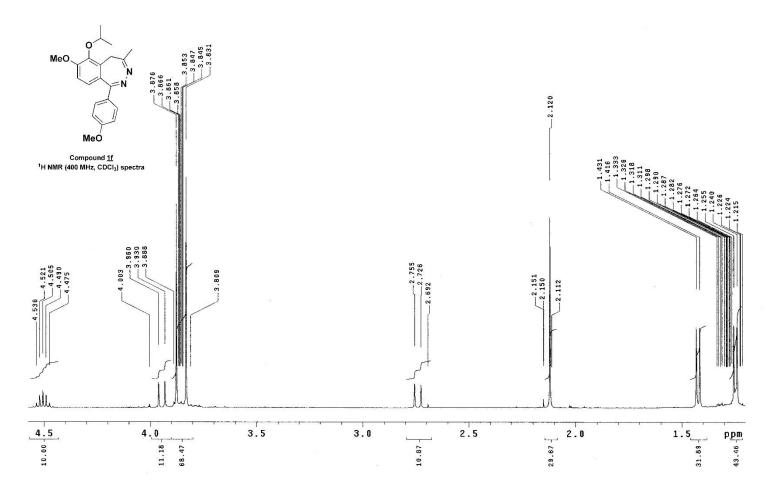
YL1040729

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 5 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



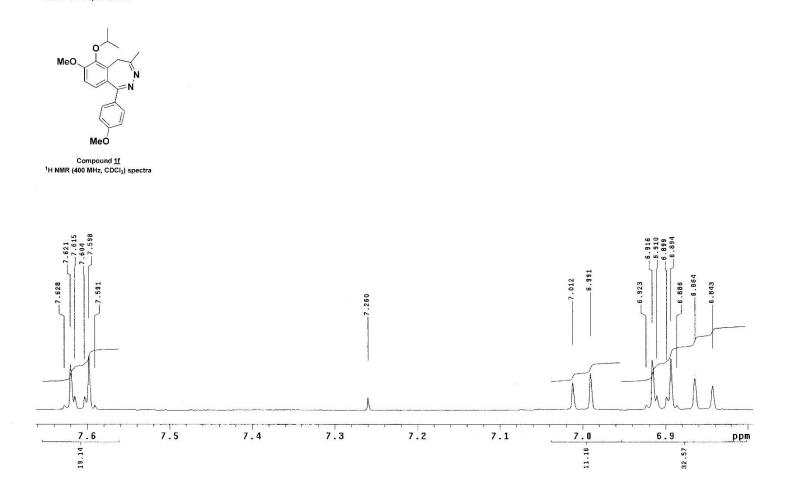
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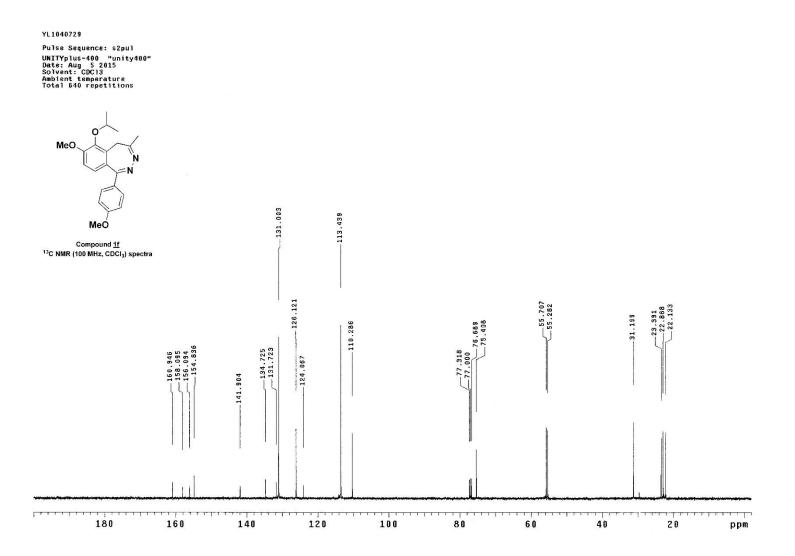
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 5 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions



YL1040729

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 5 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



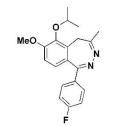


YL1040821 Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 24 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions 3.955 2.126 MeO. Compound <u>1q</u> ¹H NMR (400 MHz, CDCl₃) spectra 118 -1.433 -3.985 2.730 4.517 1 30.09 -{ S 19.52 **5** 9.80 **5** 10.00 **5** 5 9 8 6 30.18 44 3 ppm 1 30.38 £ 38.07 £ 9.39 -{ 9.92 -{ 18.47 -{

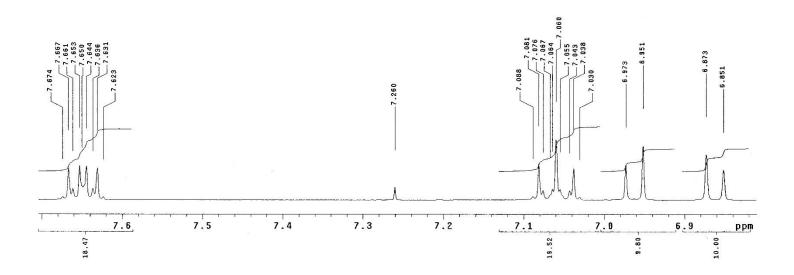
YL1040821 Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 24 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions 2.126 3.879 MeO. Compound <u>1q</u> ¹H NMR (400 MHz, CDCl₃) spectra -1.256 -3.985 2.700 2.023 ıllı 4.5 68.6 - **0** 3.5 2.5 2.0 38.07 dd 3.0 1.5___ 9.92 30.09 { 30.38 -

YL1040821

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 24 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

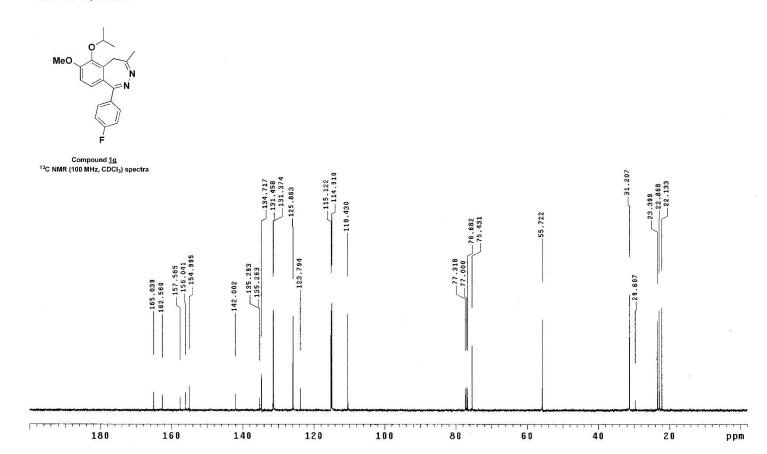


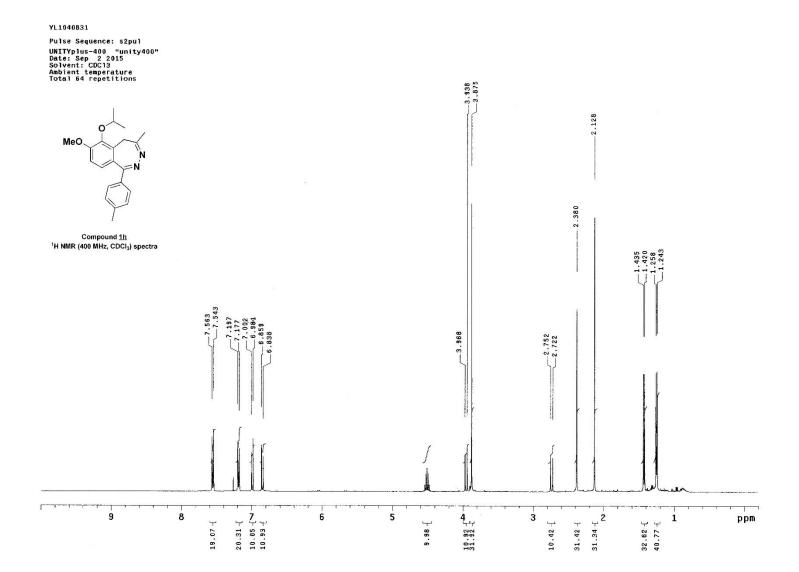
Compound <u>1g</u> ¹H NMR (400 MHz, CDCl₃) spectra

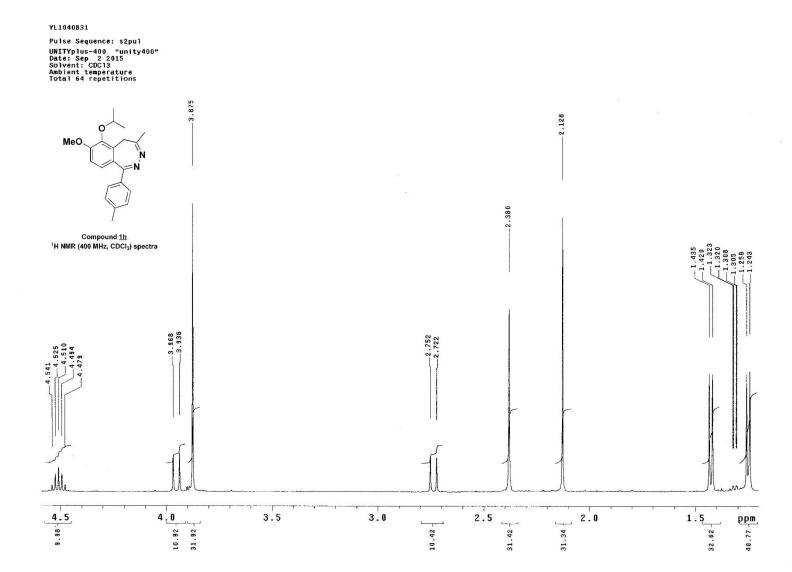


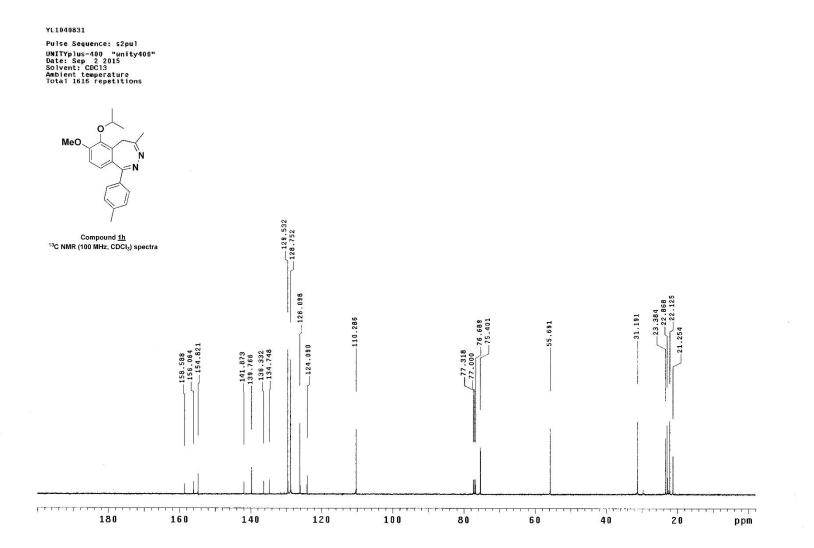
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Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 24 2015 Solvent: CDCl3 Ambient temperature Total 720 repetitions

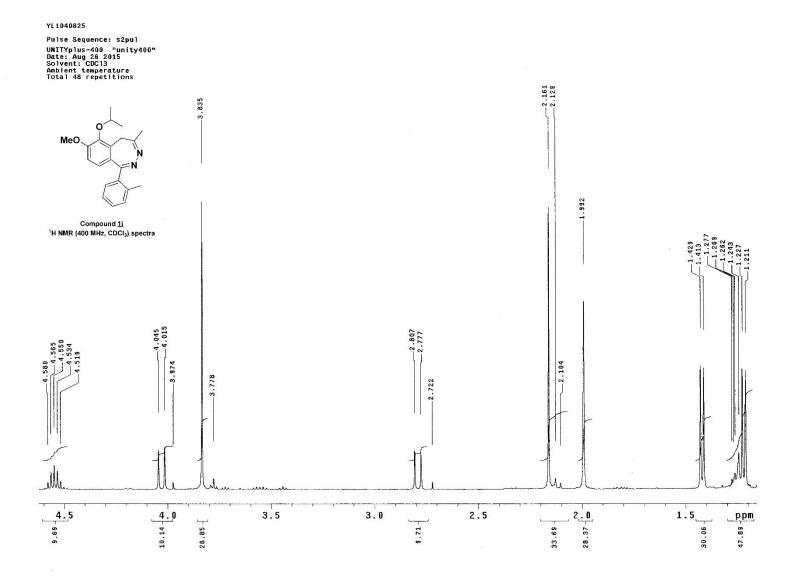








YL1040825 Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDC13 Ambient temperature Total 48 repetitions 835 2.161 ŝ MeO. -1.992 Compound 1i ¹H NMR (400 MHz, CDCl₃) spectra 6.757 4.045 2.807 6.779 6.69.9 7.280-7.160 4.550 N 5 33.69 { 28.37 £N -9 5 10.14 {.**h**. 28.85 -€ 8 6 3 ppm 7 1 9.09 { 22.00 { 10.12 { 18.97 -{ } 69 € 9.71 -{ 30.06 € 47.89 {



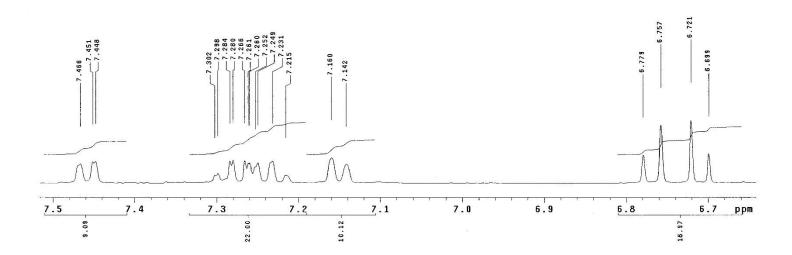
S-31

YL1040825

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDC13 Ambient temperature Total 48 repetitions

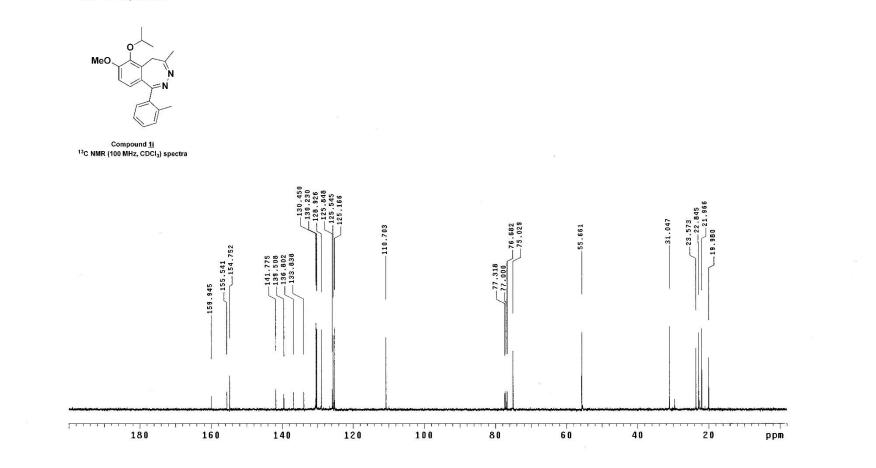
MeO.

Compound <u>1i</u> ¹H NMR (400 MHz, CDCl₃) spectra



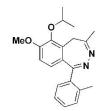
YL1040825

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDC13 Ambient temperature Total 400 repetitions

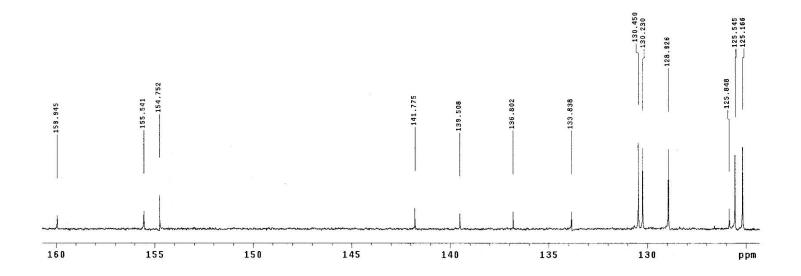


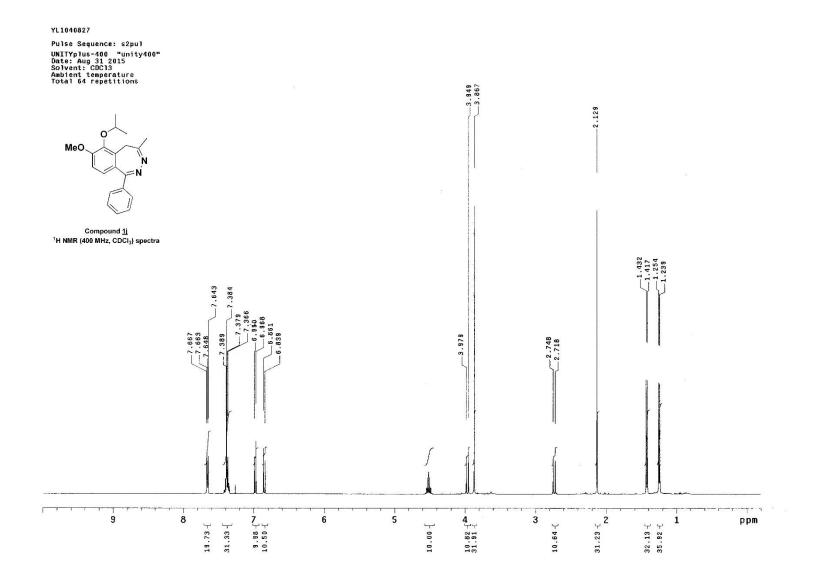
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Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDC13 Ambient temperature Total 400 repetitions

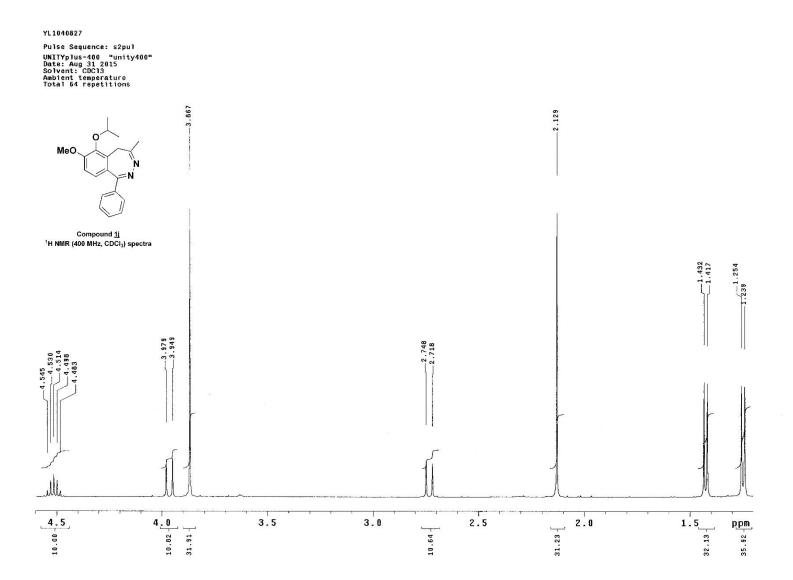


Compound <u>1i</u> ¹³C NMR (100 MHz, CDCl₃) spectra

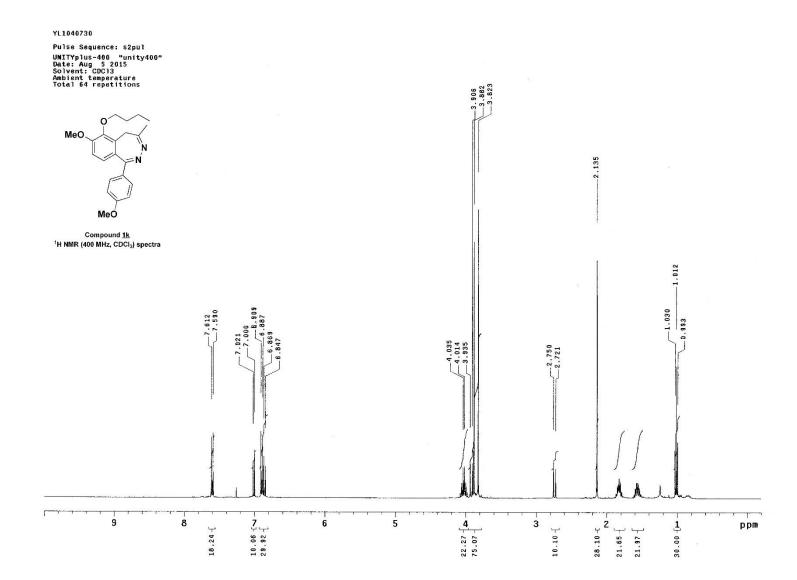


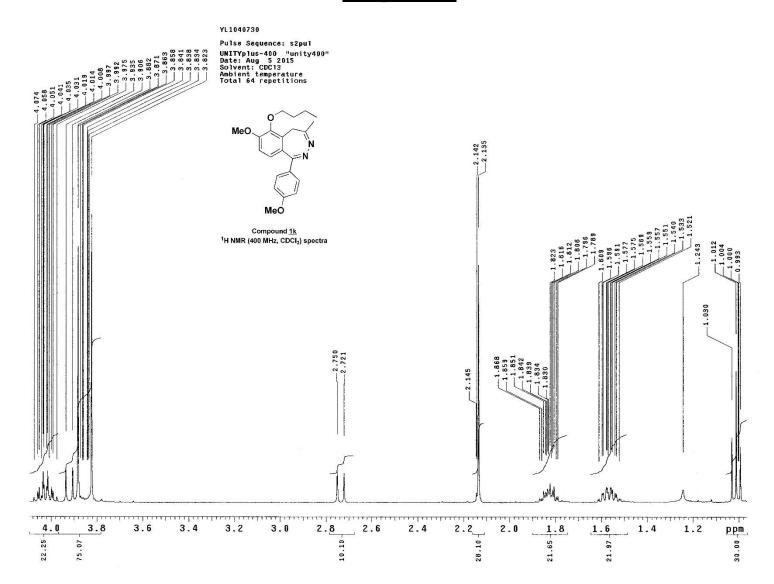


<u>Compound 1j</u>



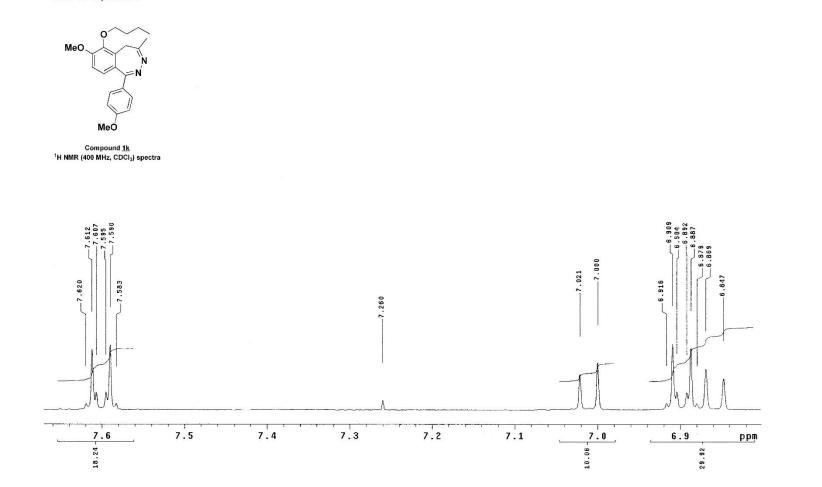
YL1040827 Pulse Sequence: s2pul UNITYPIUS-400 "unity400" Date: Aug 31 2015 Solvent: CDCl3 Ambient temperature Total 672 repetitions MeO _____129.540 ___127.971 Compound <u>1i</u> ¹³C NMR (100 MHz, CDCI₃) spectra 129.570 -23.361 -22.823 -22.072 125.992 31.146 110.301 76.682 55.654 123.946 158.634 -155.935 -154.821 77.318 77.000 134.725 -141.843 139.069 1 1 1 1 1 180 160 140 120 100 80 60 40 20 ppm

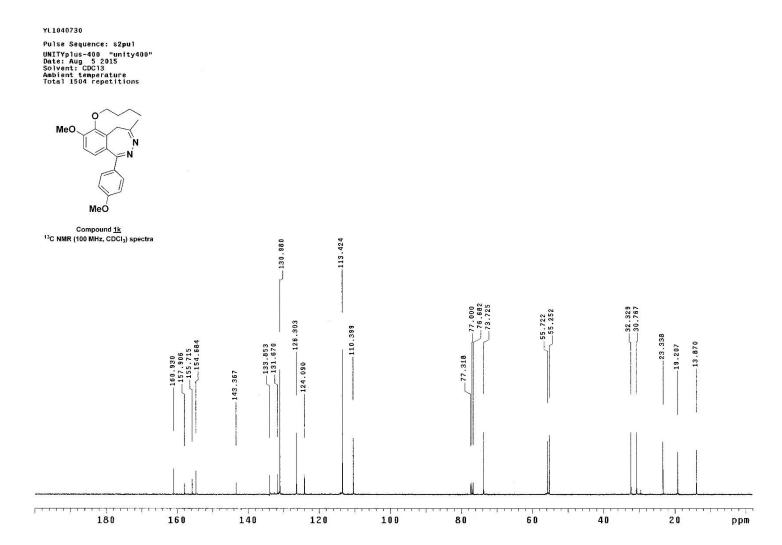


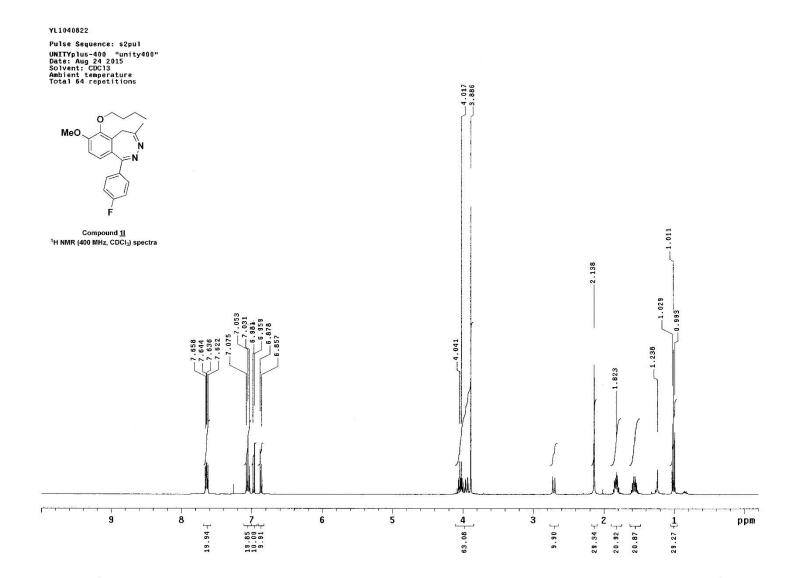


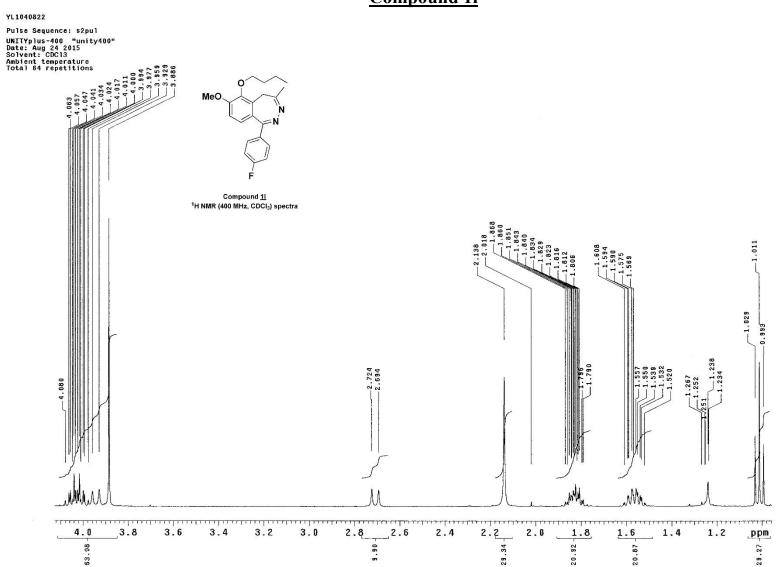
YL1040730

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 5 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions



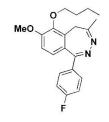




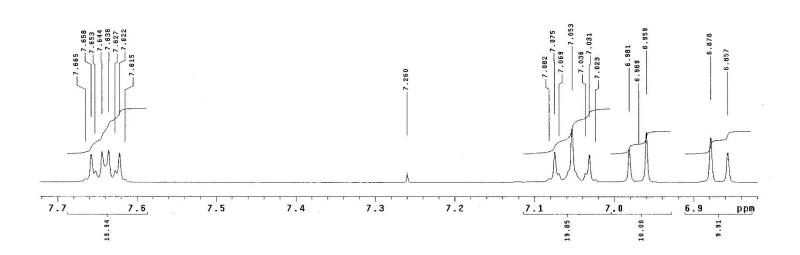


YL1040822

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 24 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



Compound <u>11</u> ¹H NMR (400 MHz, CDCI₃) spectra

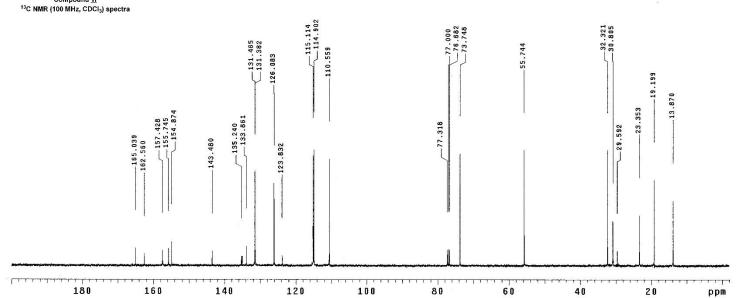


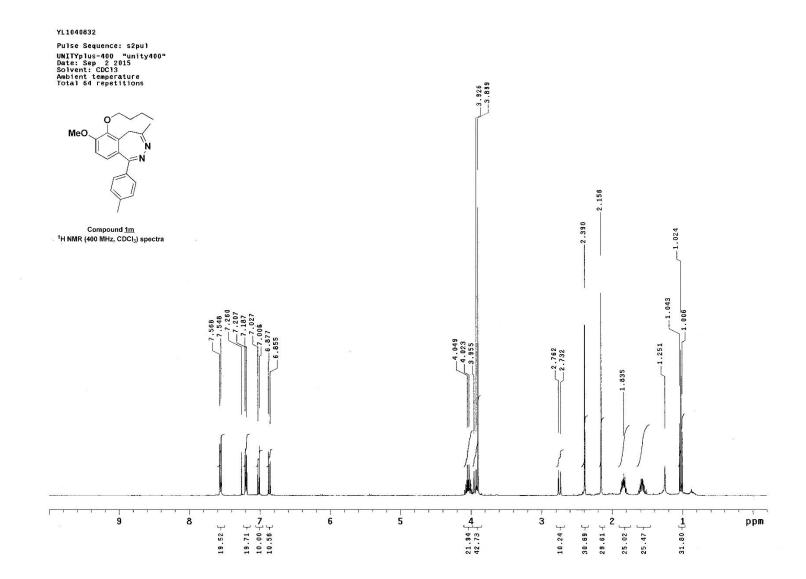
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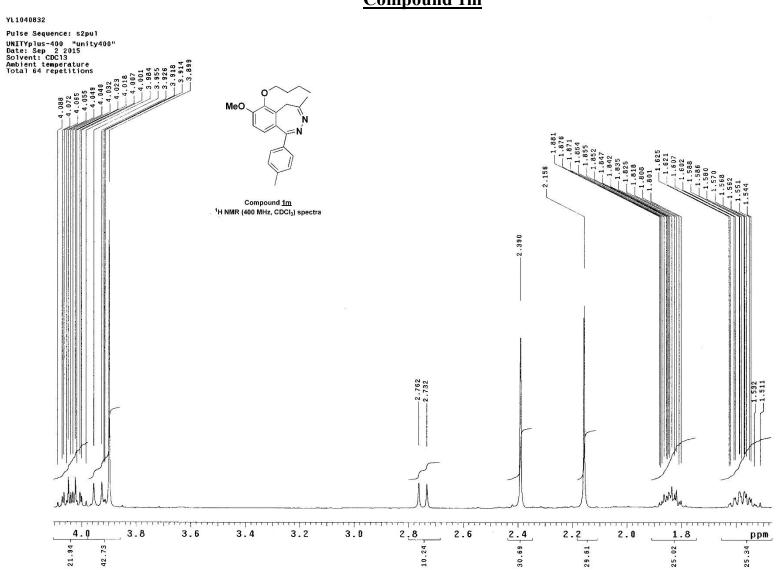
Pulse Sequence: s2pul UNITYPIUS-400 "unity400" Date: Aug 24 2015 Solvent: CDC13 Ambient temperature Total 704 repetitions



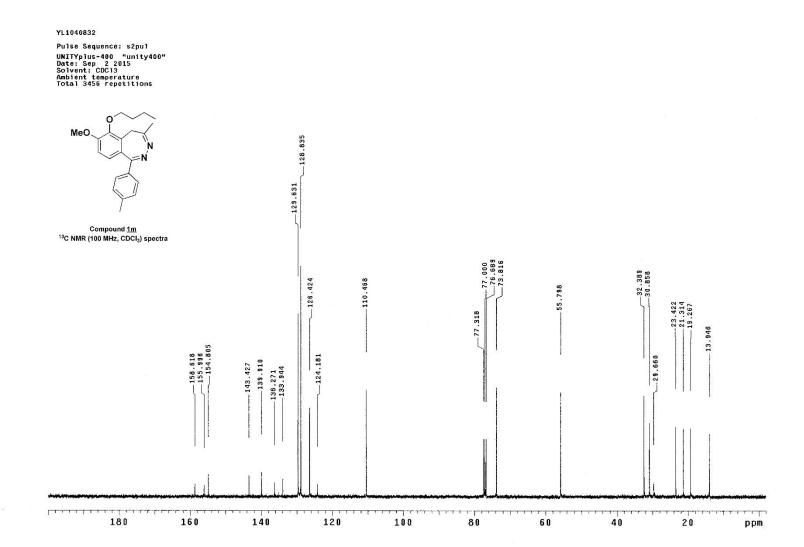
Compound <u>11</u> ¹³C NMR (100 MHz, CDCl₃) spectra







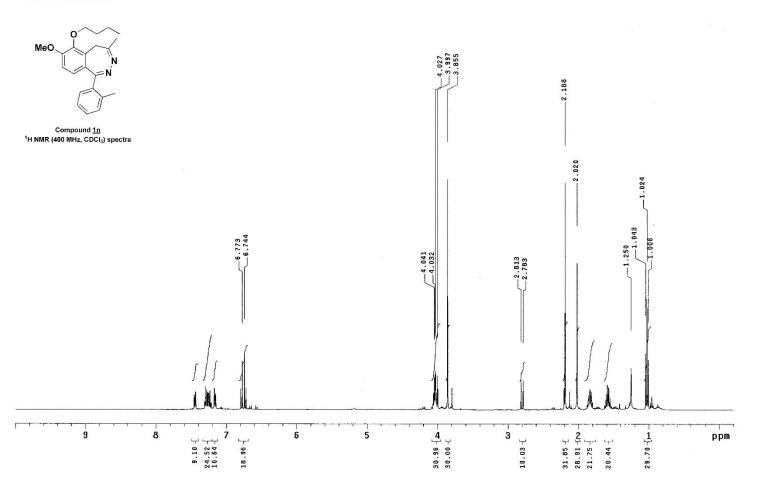
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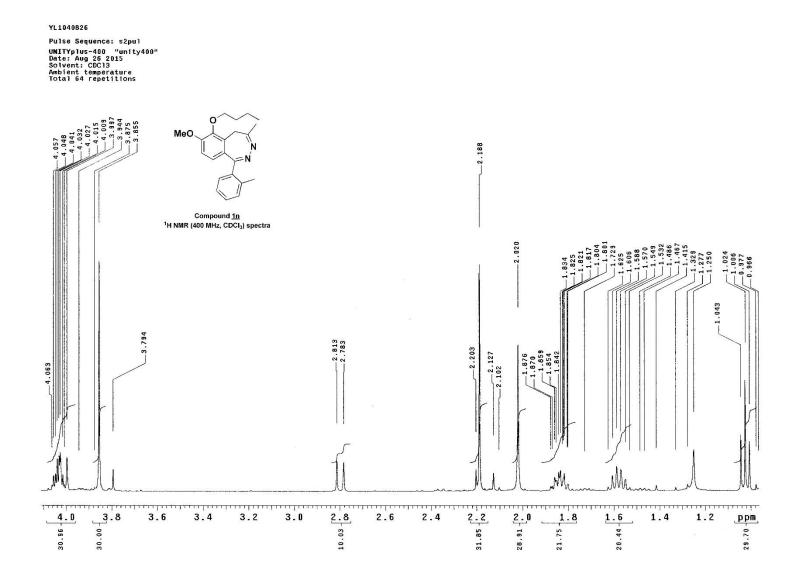


YL1040832 Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Sep 2 2015 Solvent: CDC13 Ambient temperature Total 3456 repetitions MeO 128.835 129.631 Compound <u>1m</u> ¹³C NMR (100 MHz, CDCI₃) spectra 126.424 139.910 154.805 143.427 133.944 136.271 158.618 155.996 124.181 155 150 145 140 135 130 ppm



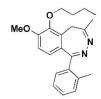
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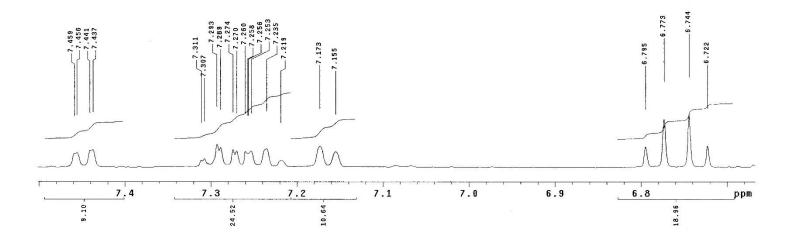


YL1040826

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



Compound <u>1n</u> ¹H NMR (400 MHz, CDCl₃) spectra

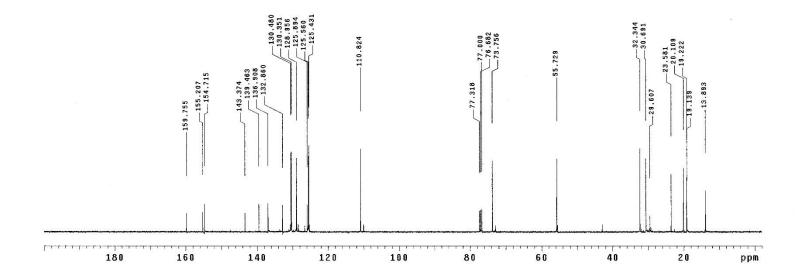


YL1040826

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDCl3 Ambient temperature Total 64000 repetitions

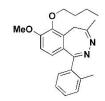
MeO

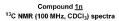
Compound <u>1n</u> ¹³C NMR (100 MHz, CDCl₃) spectra

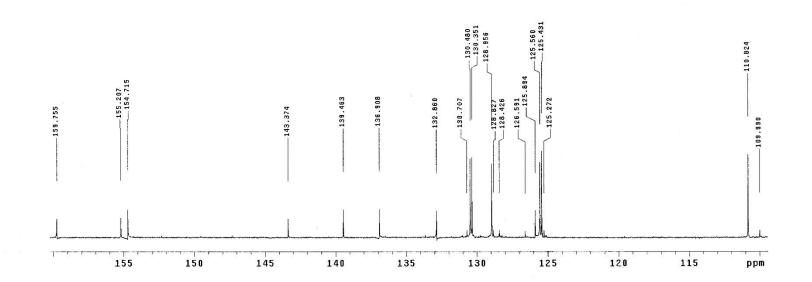


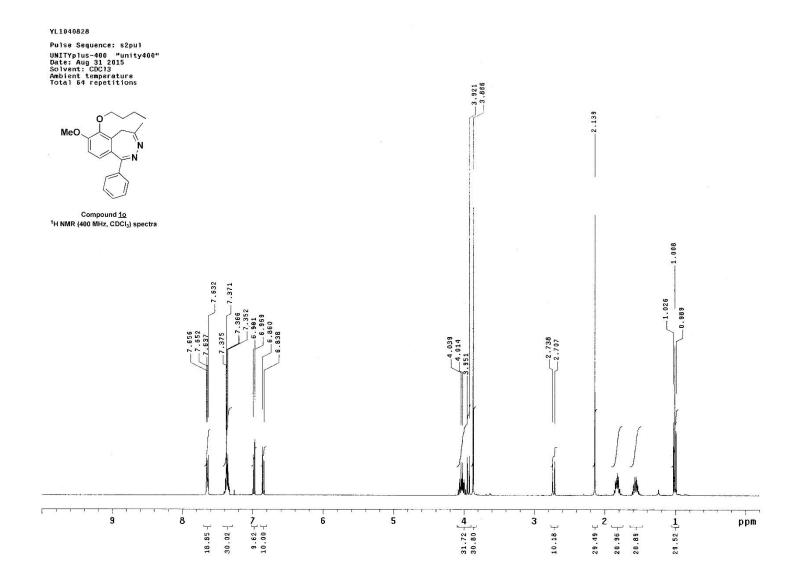
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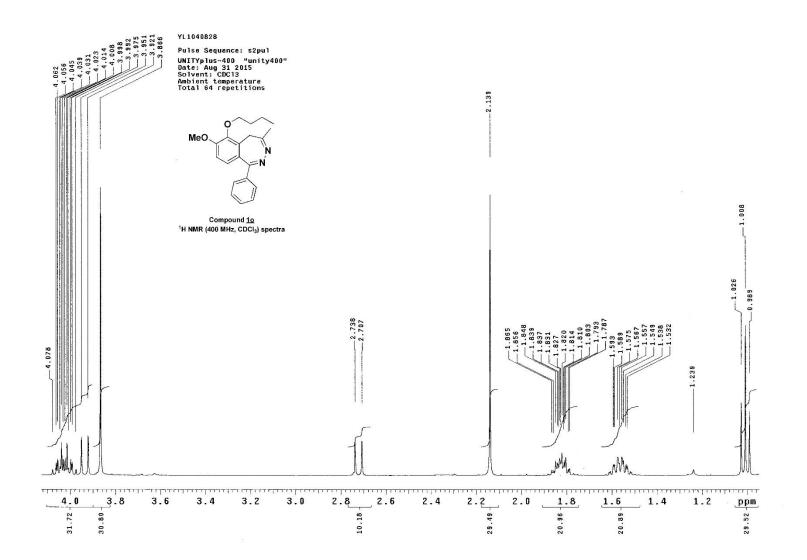
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDCl3 Ambient temperature Total 64000 repetitions









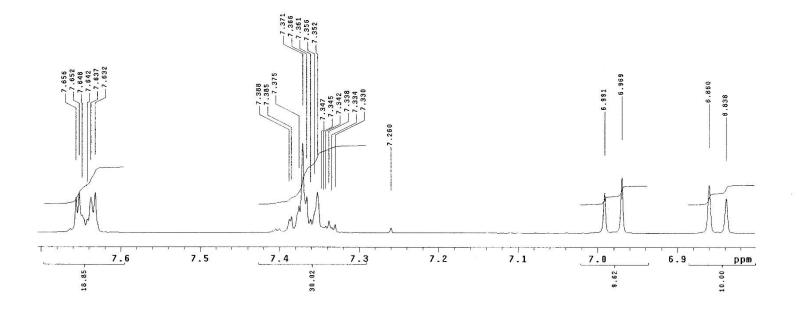


YL1040828

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 31 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

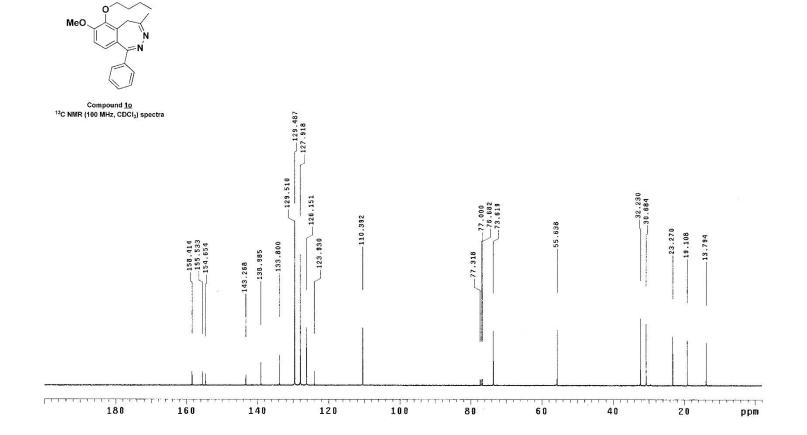
MeO.

Compound <u>1o</u> ¹H NMR (400 MHz, CDCl₃) spectra



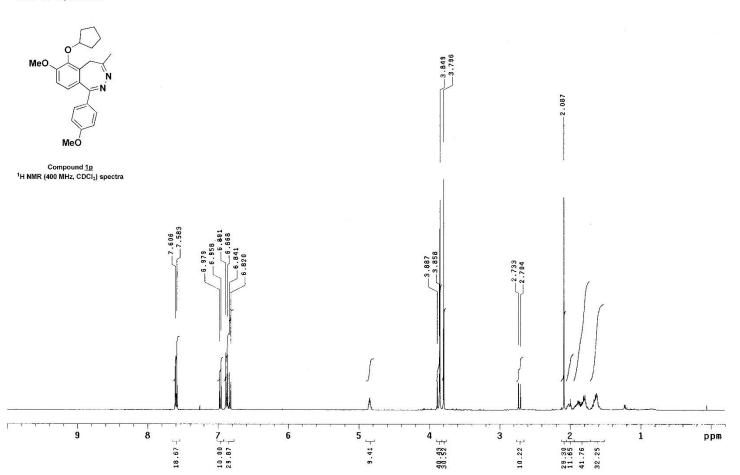
YL1040828

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 31 2015 Solvent: CDC13 Ambient temperature Total 336 repetitions



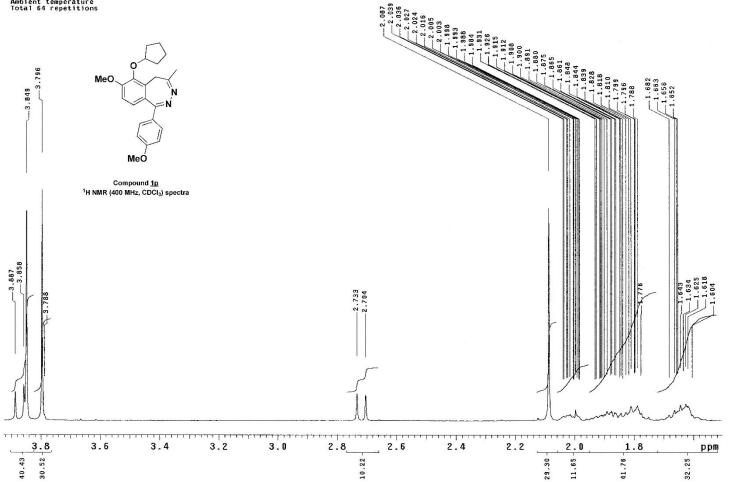
YL1040805

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 20 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



YL1040805

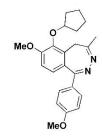
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 20 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



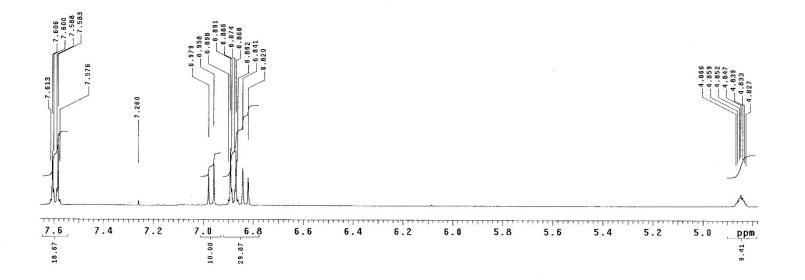
S-60

YL1040805

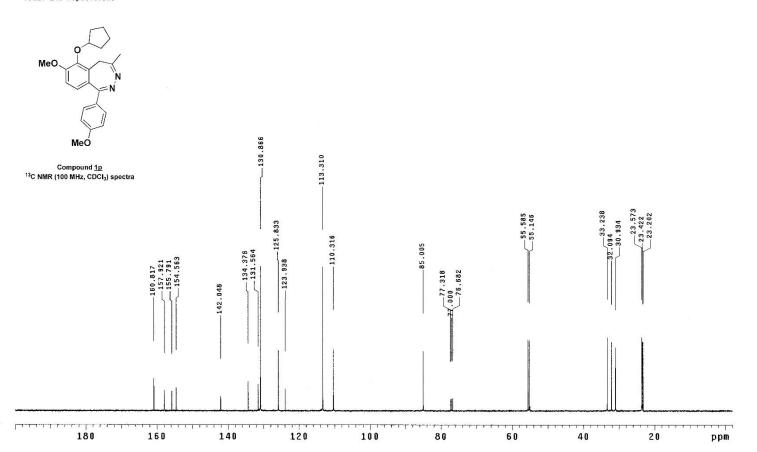
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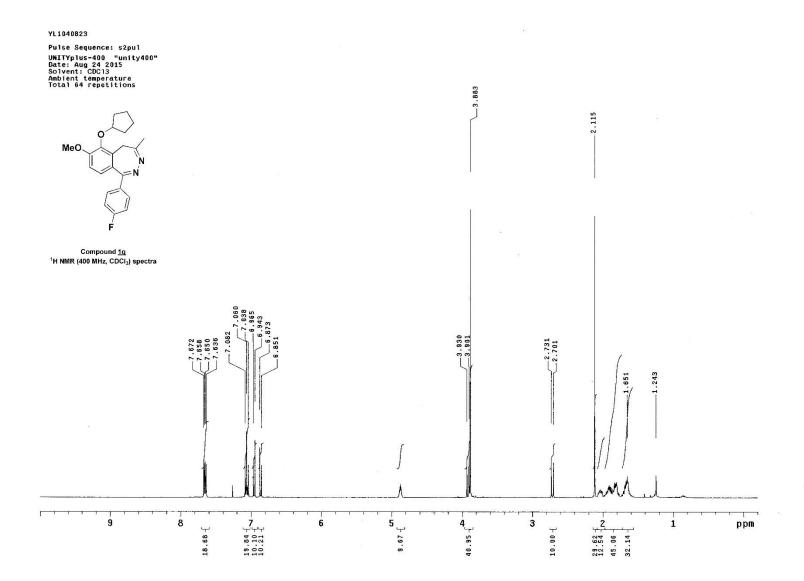


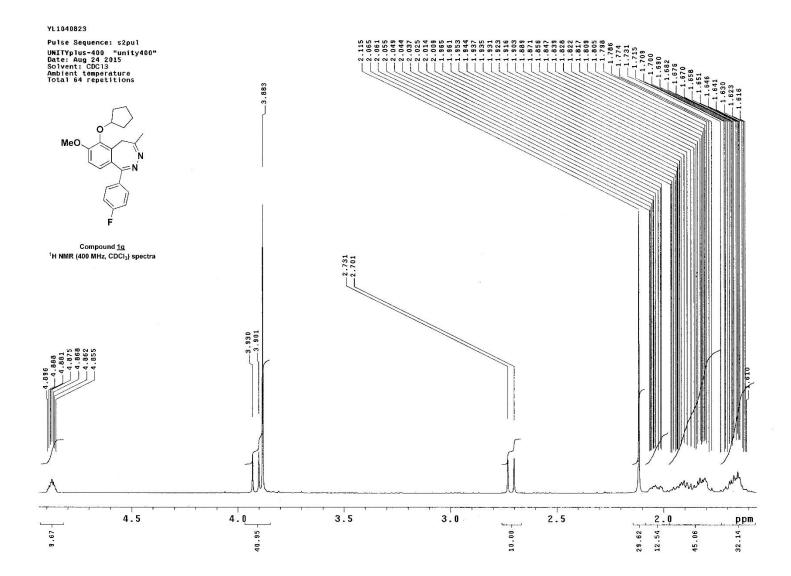
Compound <u>1p</u> ¹H NMR (400 MHz, CDCI₃) spectra



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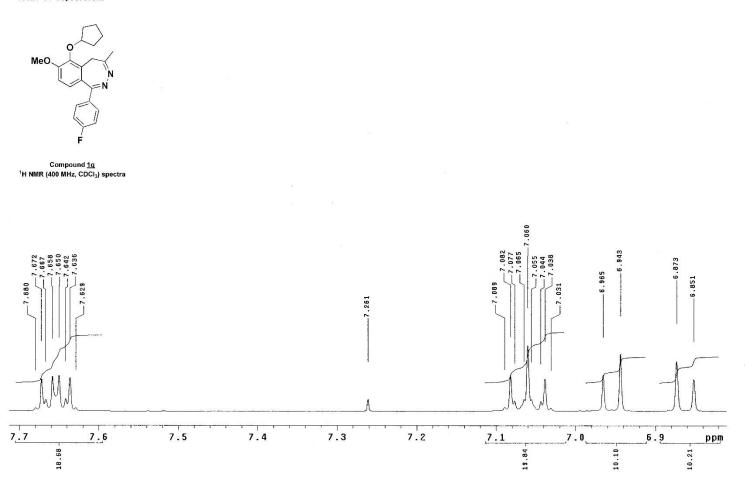






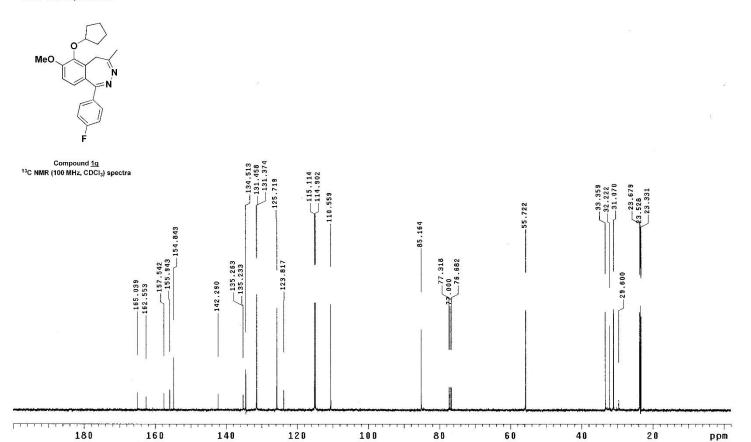
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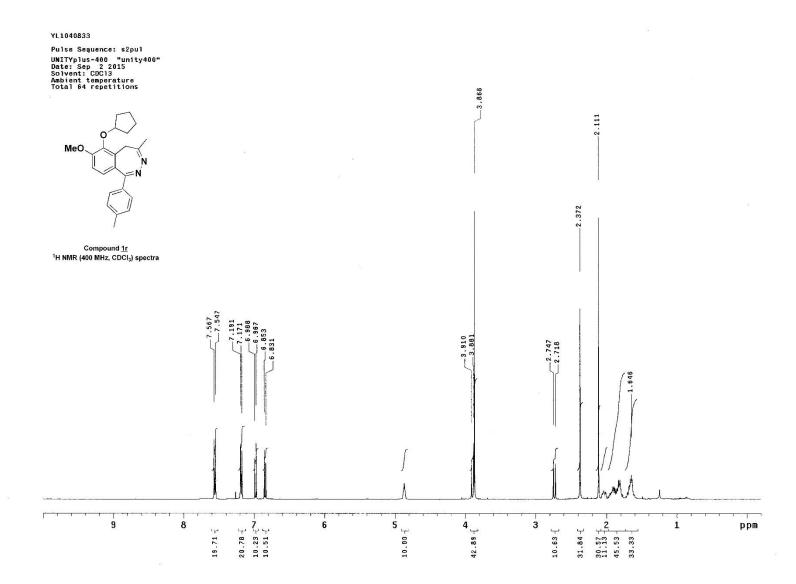
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 24 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

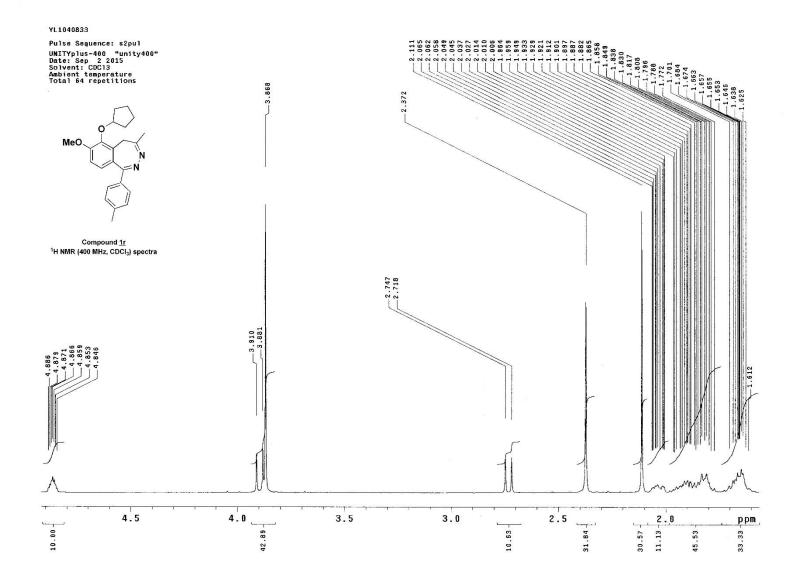


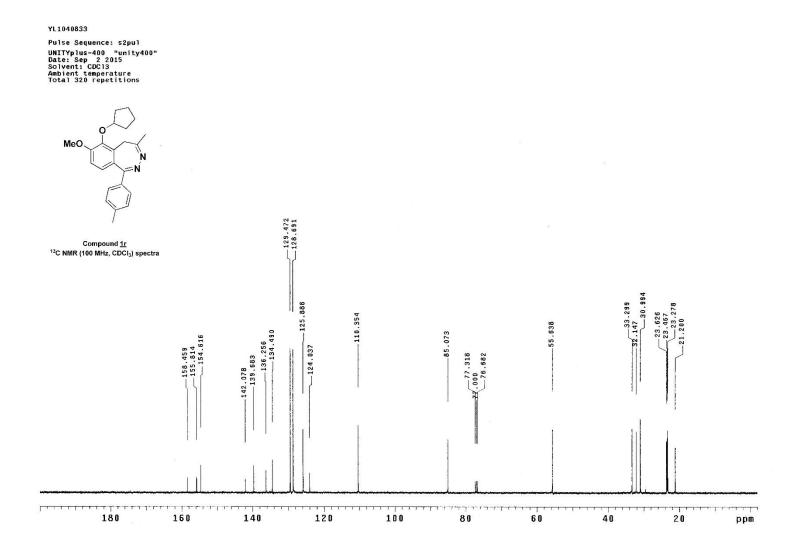
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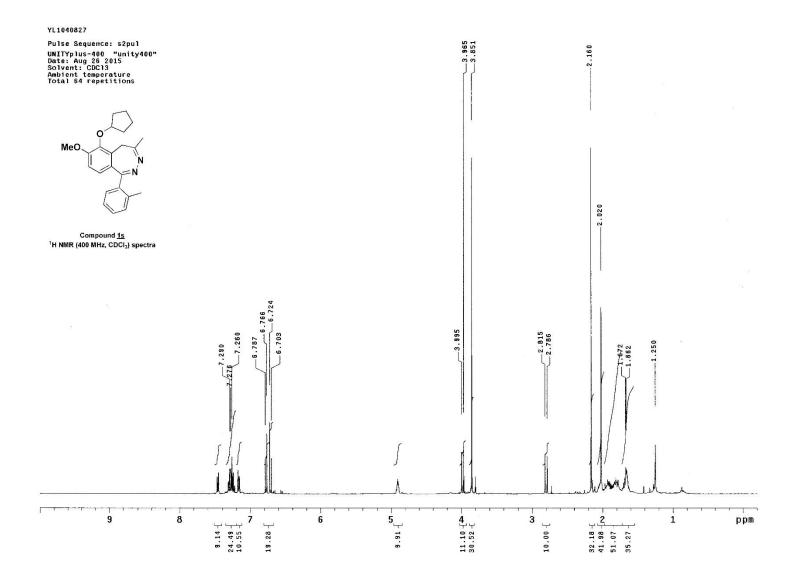
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 24 2015 Solvent: CDC13 Ambient temperature Total 640 repetitions

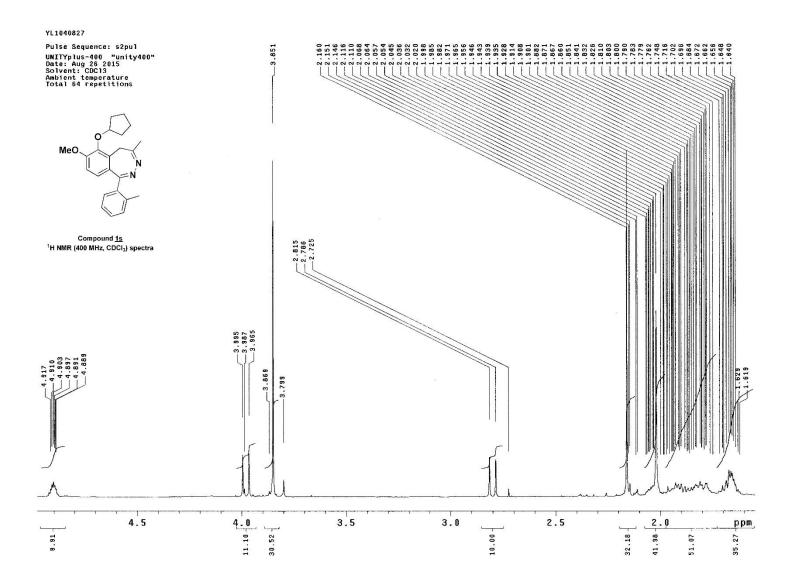






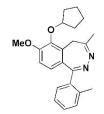




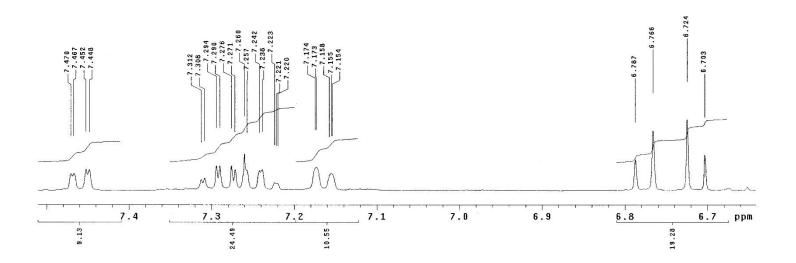


YL1040827

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



Compound <u>1s</u> ¹H NMR (400 MHz, CDCI₃) spectra

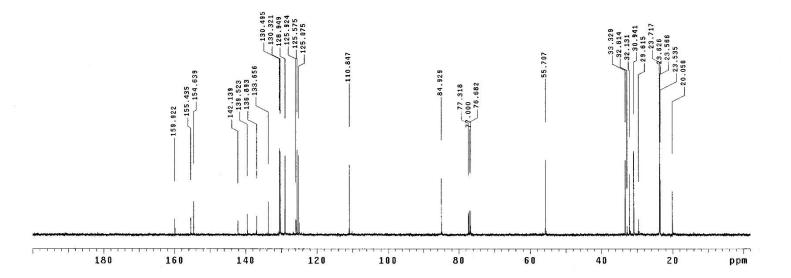


YL1040827

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDC13 Ambient temperature Total 944 repetitions

MeO.

Compound <u>1s</u> ¹³C NMR (100 MHz, CDCI₃) spectra

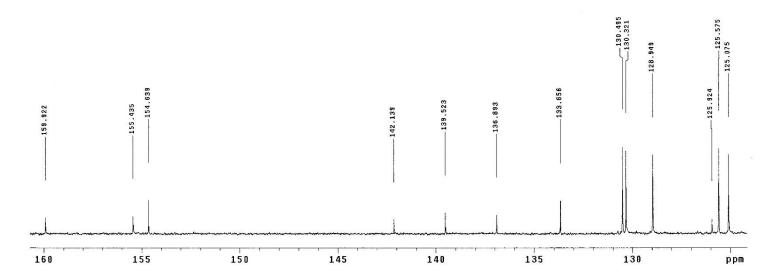


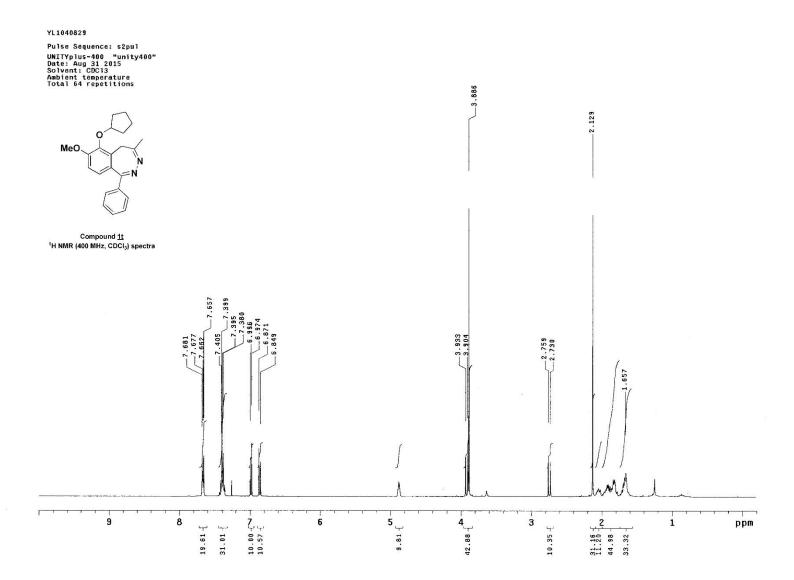
YL1040827

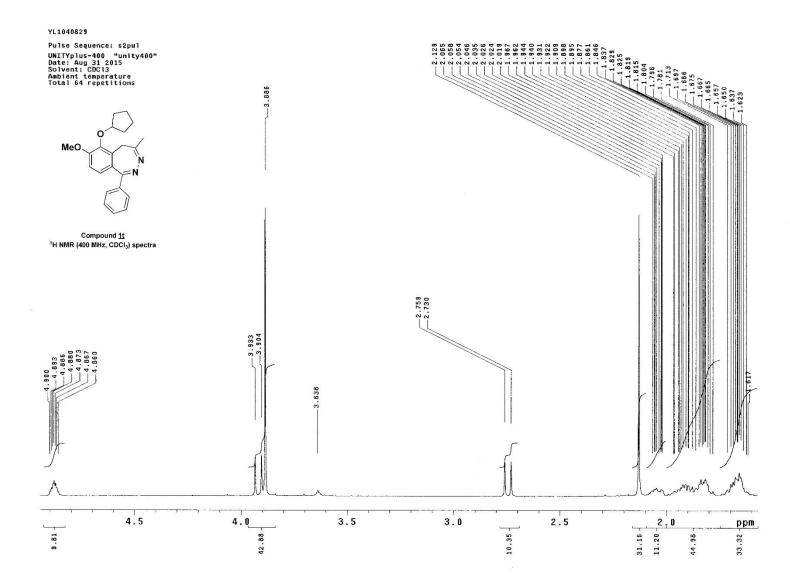
Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 26 2015 Solvent: CDC13 Ambient temperature Total 944 repetitions

MeO.

Compound <u>1s</u> ¹³C NMR (100 MHz, CDCI₃) spectra

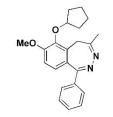




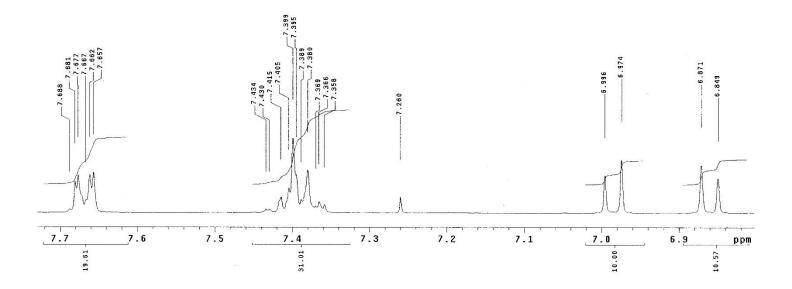


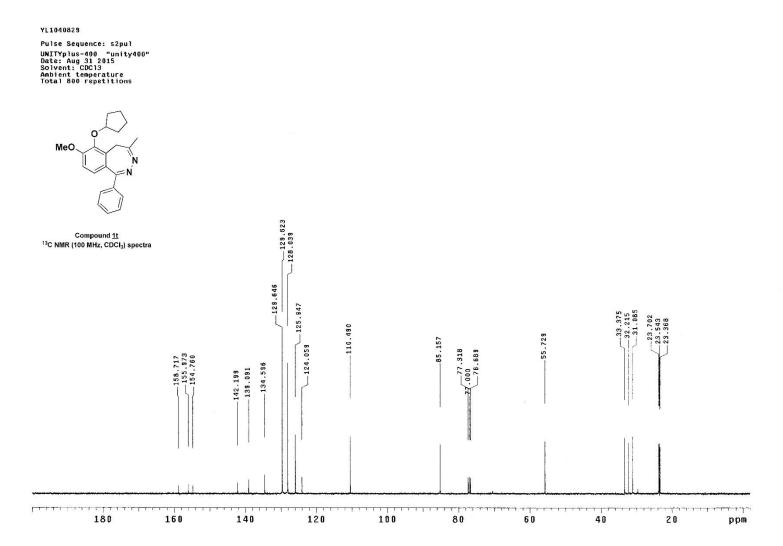
YL1040829

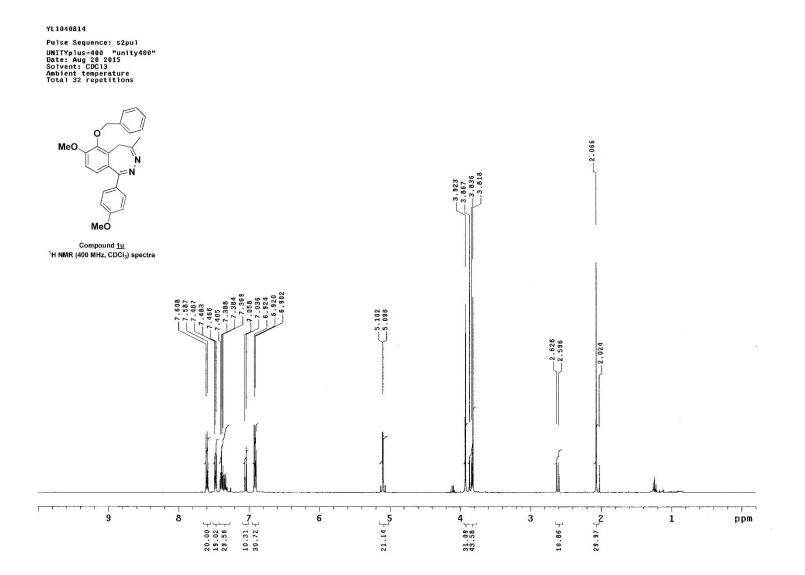
Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Aug 31 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



Compound <u>1t</u> ¹H NMR (400 MHz, CDCl₃) spectra

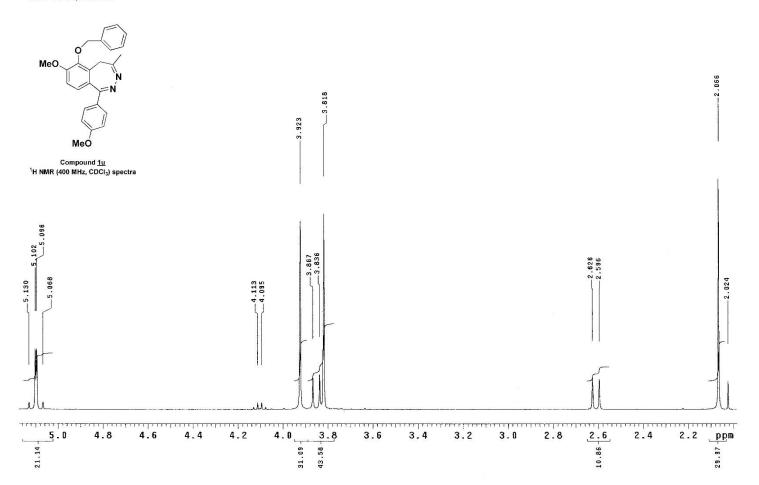






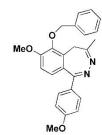
YL1040814

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Aug 20 2015 Solvent: CDCl3 Ambient temperature Total 32 repetitions

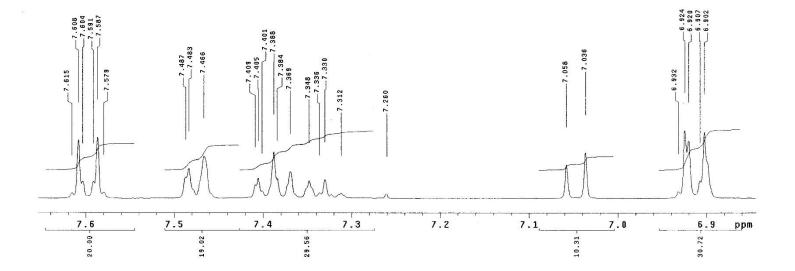


YL1040814

Pulse Sequence: s2pu) UMITYplus-400 "unity400" Date: Aug 20 2015 Solvent: CDC13 Ambient temperature Total 32 repetitions

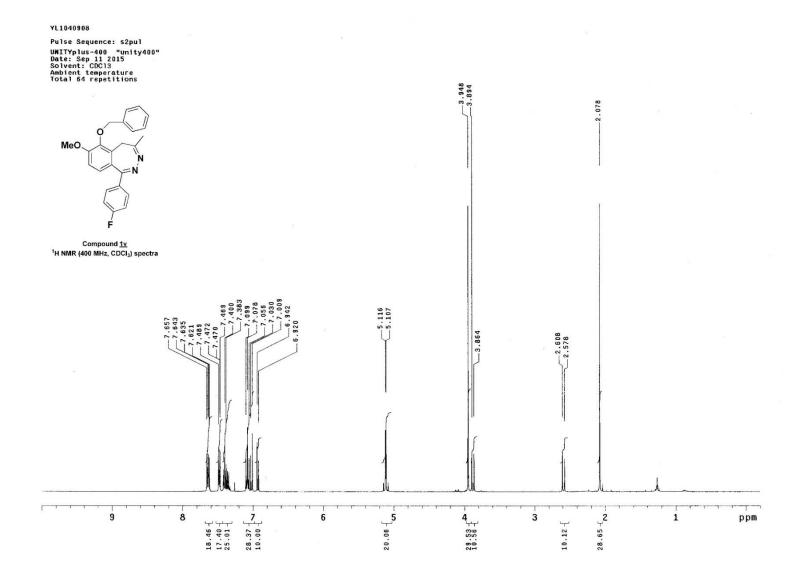


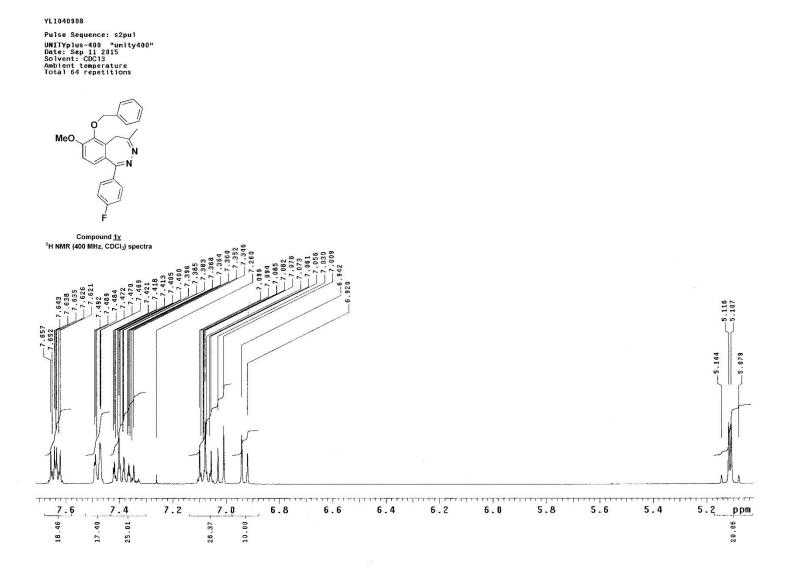
Compound <u>1u</u> ¹H NMR (400 MHz, CDCl₃) spectra

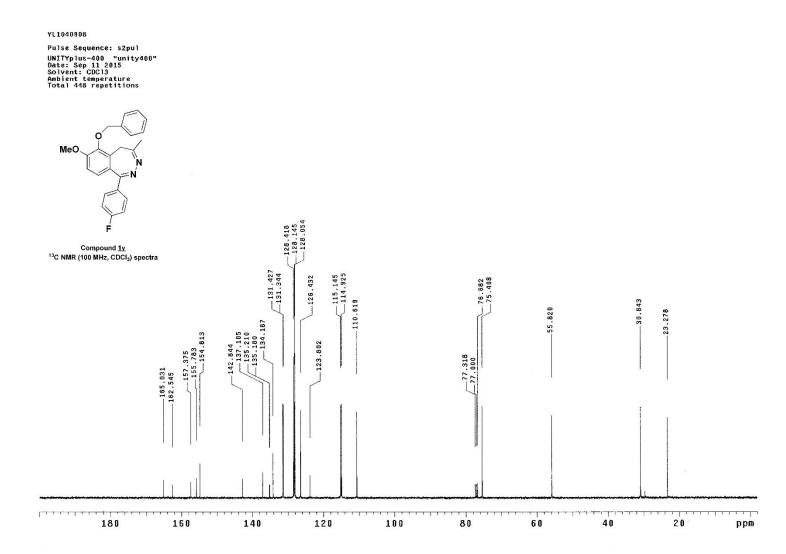


YL1040814 Pulse Sequence: s2pul UNITYPIUS-400 "unity400" Date: Aug 20 2015 Solvent: CDC13 Ambient temperature Total 208 repetitions MeO. MeÓ 113.348 130. Compound <u>1u</u> ¹³C NMR (100 MHz, CDCl₃) spectra 127.948 75.287 55.699 55.138 123.953 110.392 -23.164 -30.714 137.075 --134.058 --131.526 77.318 142.631 L 1111 -----1 1 1 1 180 160 140 100 80 60 40 20 120 ppm

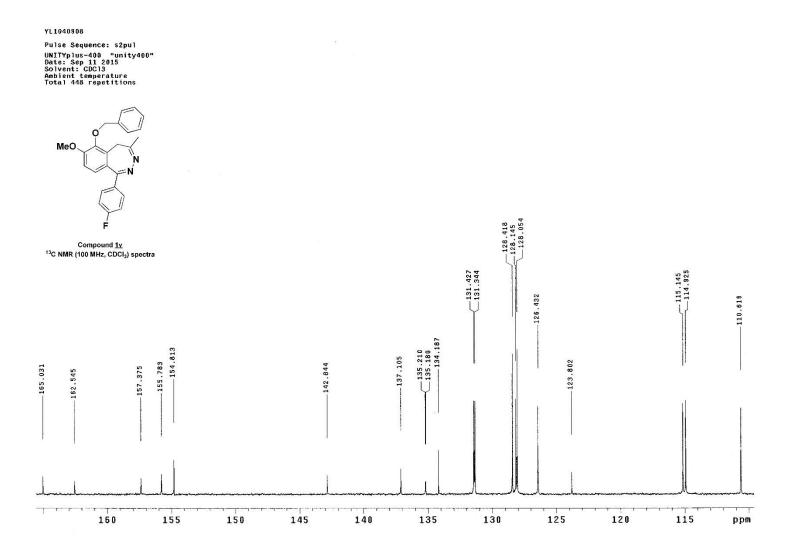
YL1040814 Pulse Sequence: s2pul UNITYPIUS-400 "unity400" Date: Aug 20 2015 Solvent: CDC13 Ambient temperature Total 208 repetitions MeO. MeO 113.348 128.304 128.001 .948 Compound <u>1u</u> ¹³C NMR (100 MHz, CDCl₃) spectra 130.851 127 126.546 110.392 160.839 134.058 131.526 -154.533 123.953 137.075 157.769 155,632 142.631 T T 160 155 150 125 120 115 145 140 135 130 ppm





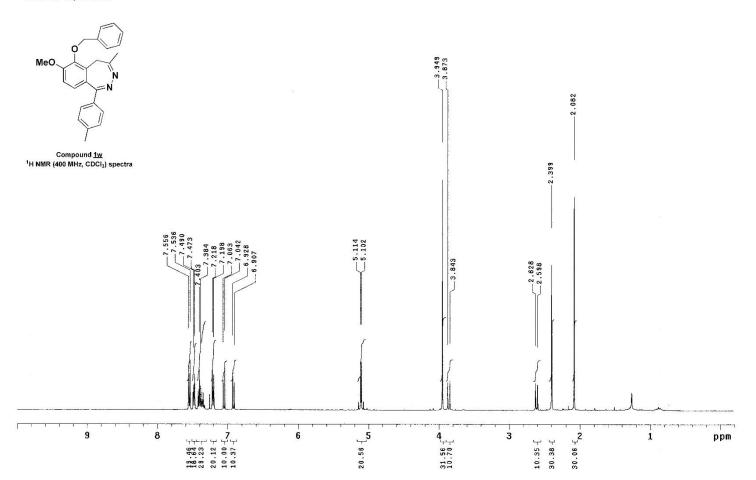


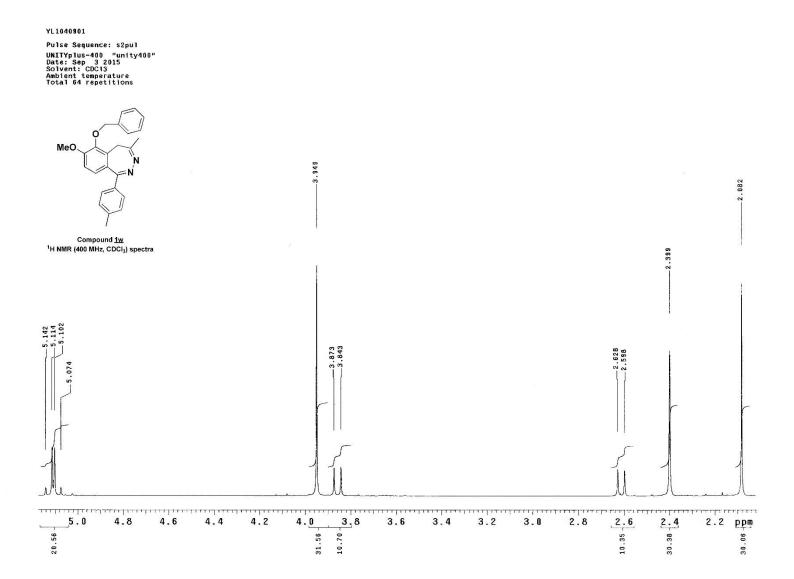
S-86



YL1040901

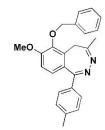
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 3 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



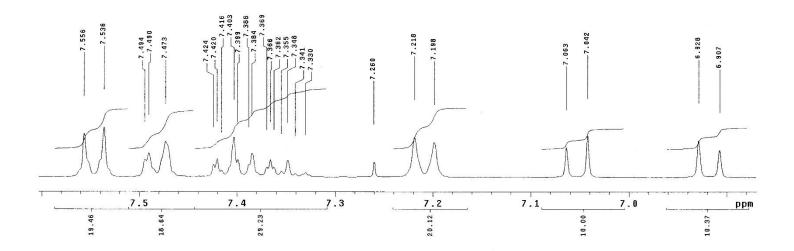


YL1040901

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 3 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions



Compound <u>1w</u> ¹H NMR (400 MHz, CDCl₃) spectra

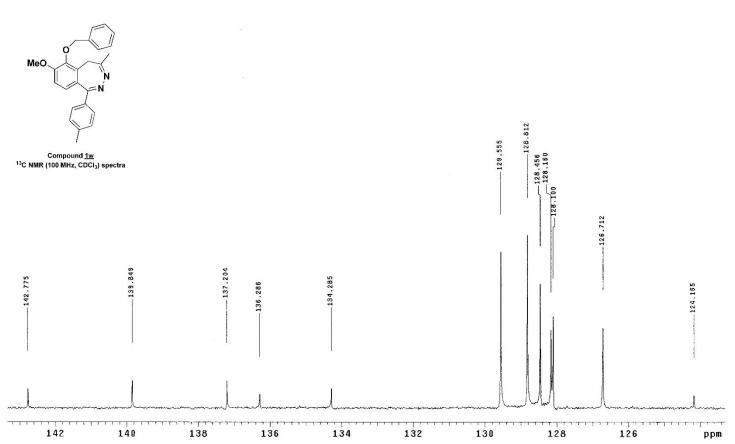


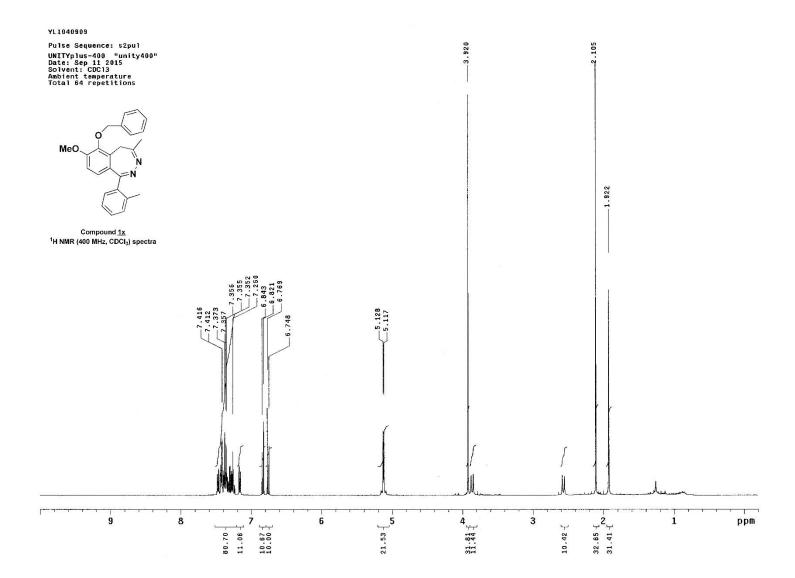
YL1040901 Pulse Sequence: s2pul UNITYplus-406 "unity400" Date: Sep 3 2015 Solvent: CDC13 Ambient temperature Total 1280 repetitions MeO. -128.812 129.555 Compound <u>1w</u> ¹³C NMR (100 MHz, CDCl₃) spectra 76.682 30.866 23.331 110.505 55.843 136.286 6 8 -77.311 124.165 158.421 -155.821 -154.677 29.630 F------1111 1111 111 80 180 160 140 60 120 100 40 20 ppm

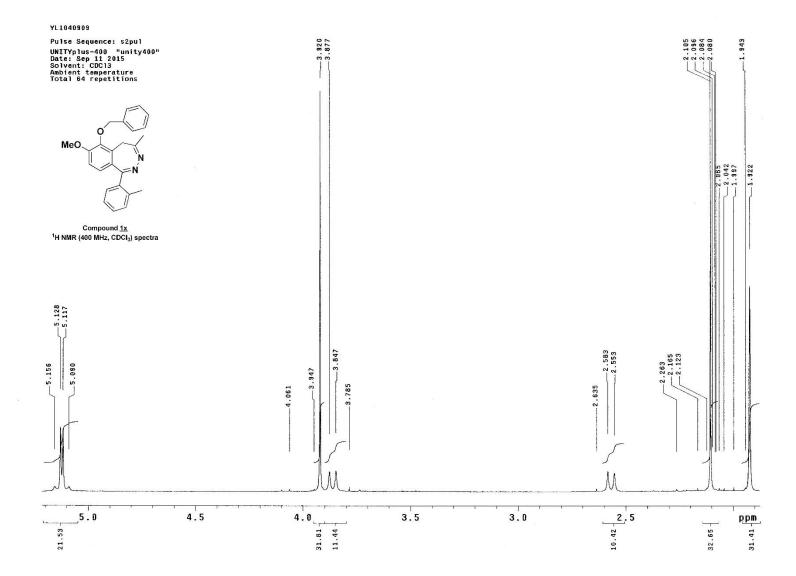


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Pulse Sequence: s2pul UNITYplus-406 "unity400" Date: Sep 3 2015 Solvent: CDC13 Ambient temperature Total 1280 repetitions



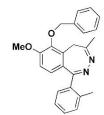




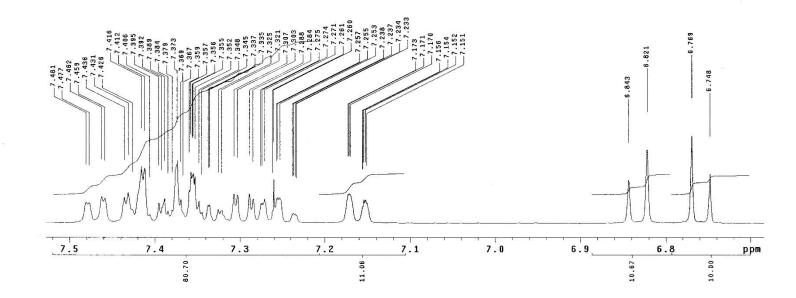
S-94

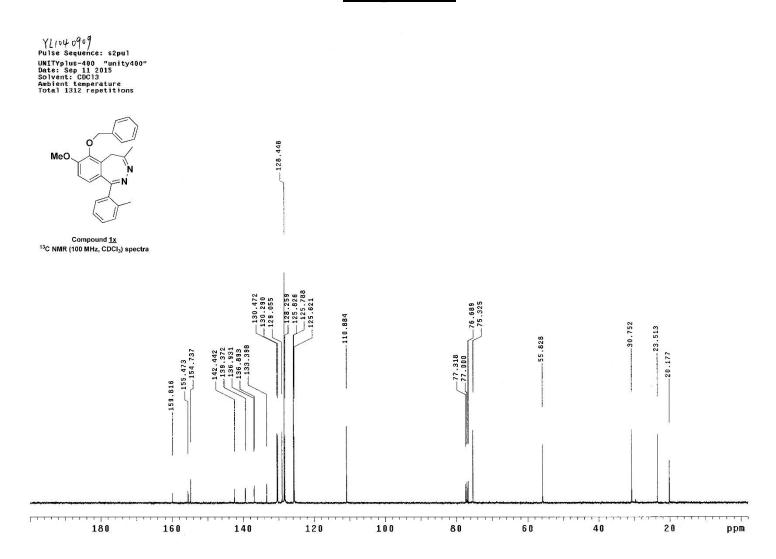
YL1040909

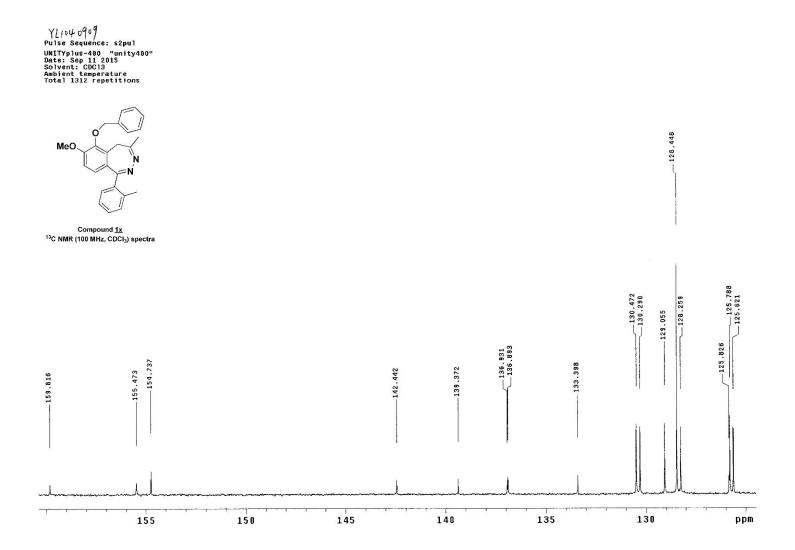
Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Sep 11 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

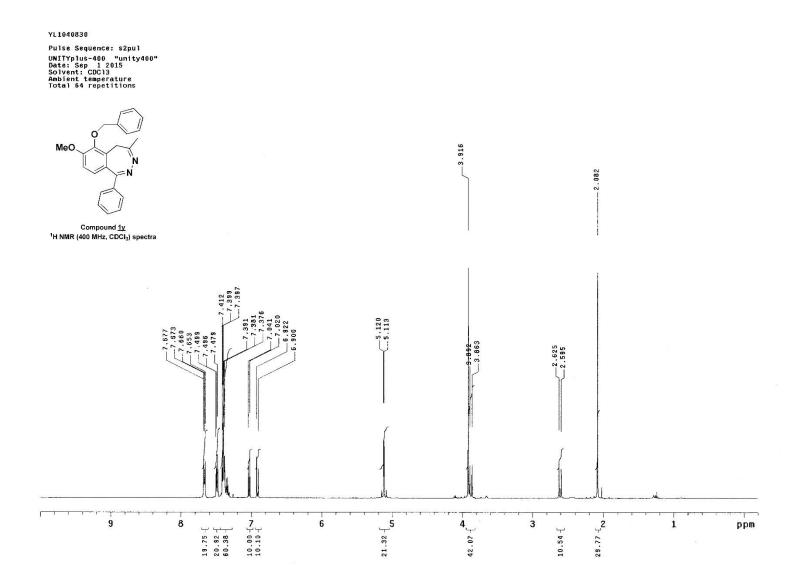


Compound <u>1x</u> ¹H NMR (400 MHz, CDCI₃) spectra



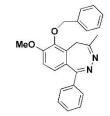




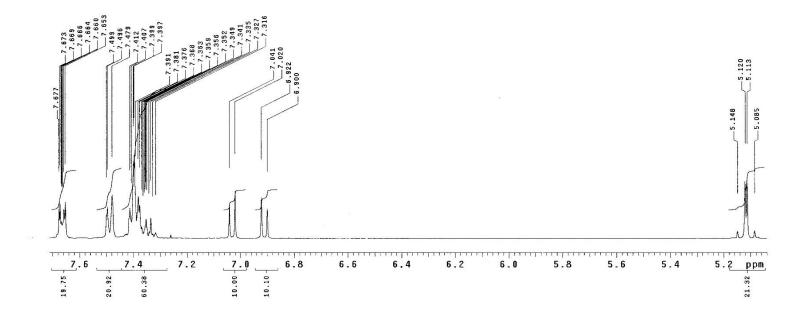


YL1040830

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Sep 1 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

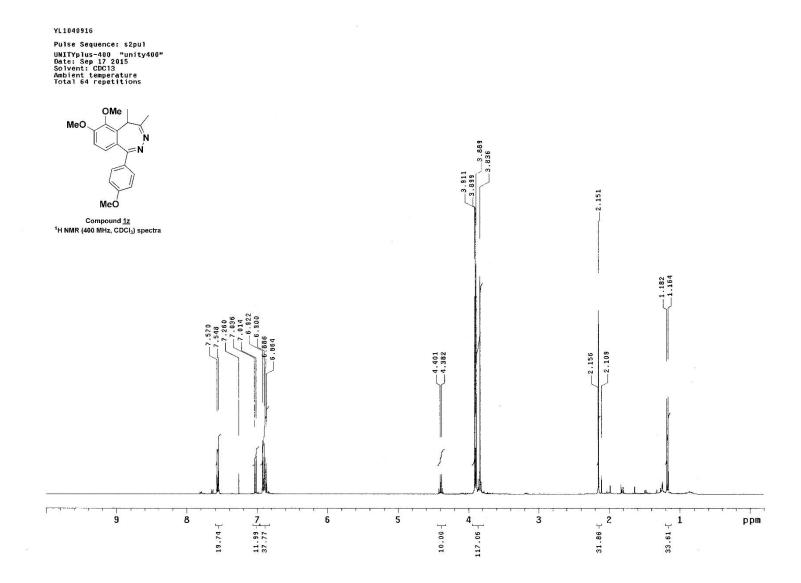


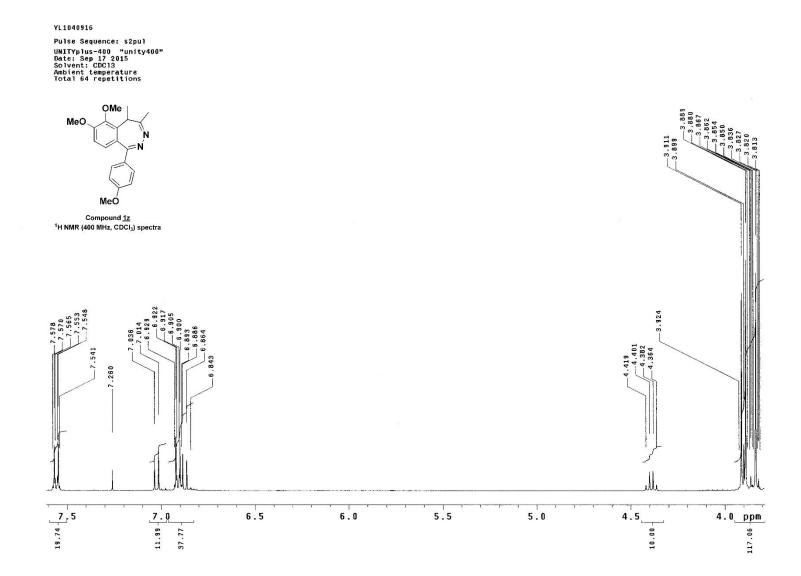
Compound <u>1v</u> ¹H NMR (400 MHz, CDCI₃) spectra



YL1040830 Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 1 2015 Solvent: CDC13 Ambient temperature Total 704 repetitions MeO. Compound <u>1y</u> ¹³C NMR (100 MHz, CDCI₃) spectra -129.441 .386 -75.196 126 110.407 55.616 30.638 23.103 123.809 -158.201-155.359 -154.487 _____ 1,11 1111 TTT 11 1 1 1 180 160 140 120 100 80 60 40 20 ppm

YL1040830 Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 1 2015 Solvent: CDC13 Ambient temperature Total 704 repetitions MeO. Compound <u>1y</u> ¹³C NMR (100 MHz, CDCI₃) spectra -129.441 110.407 126.386 134.043 138.894 155.359 123.809 158.201 136.969 154.487 142.548 150 155 145 140 115 135 130 125 120 ppm



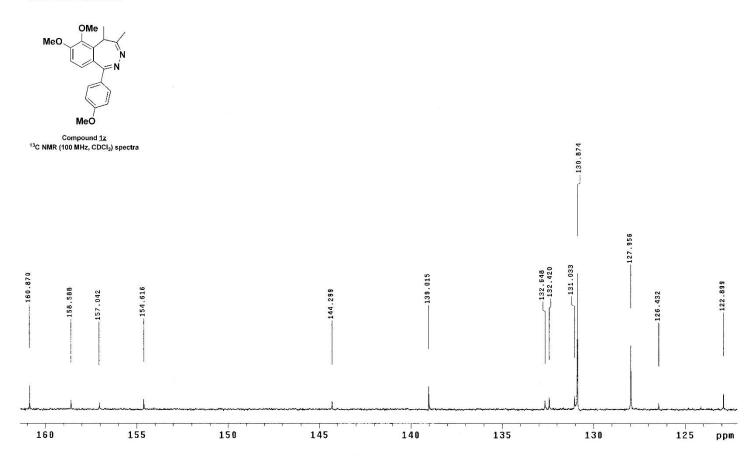


S-103

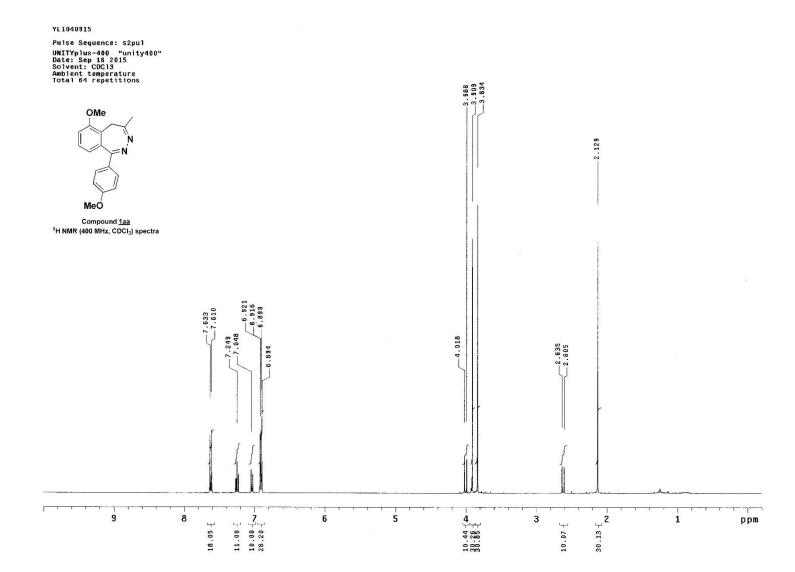
YL1040916 Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 17 2015 Solvent: CDC13 Ambient temperature Total 1680 repetitions OMe MeO MeÓ Compound <u>1z</u> ¹³C NMR (100 MHz, CDCI₃) spectra 132.420 -131.033 -130.874 113.492 -127.956 -55.805 551 35.937 25.165 110. 77.318 .000 76.682 158 588 126.432 -132.648 61.680 830 157.042 154.616 -139.015 144.299 -13.688 LILILIA 1 T T T T T T T 1.1 11 11 180 160 140 120 100 80 60 40 20 ppm

YL1040916

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Sep 17 2015 Solvent: CDC13 Ambient temperature Total 1680 repetitions



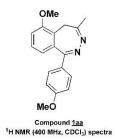
Compound 1aa

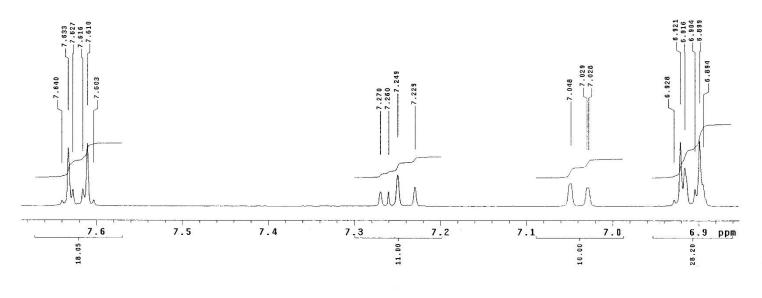


Compound 1aa

YL1040915

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 16 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions



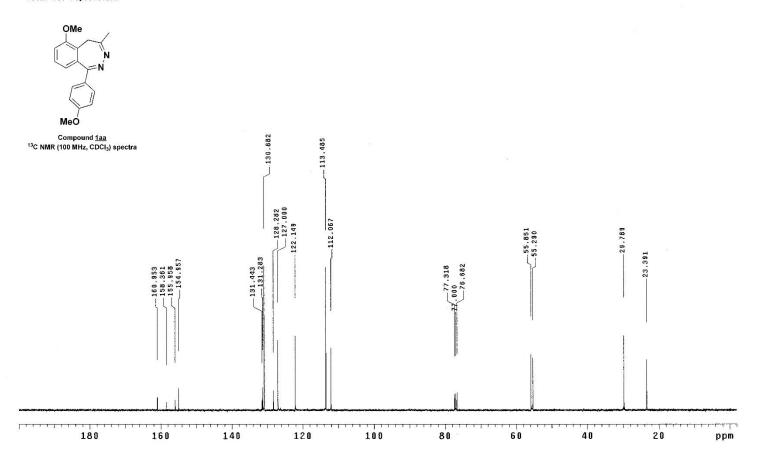


S-107

Compound 1aa

YL1040915

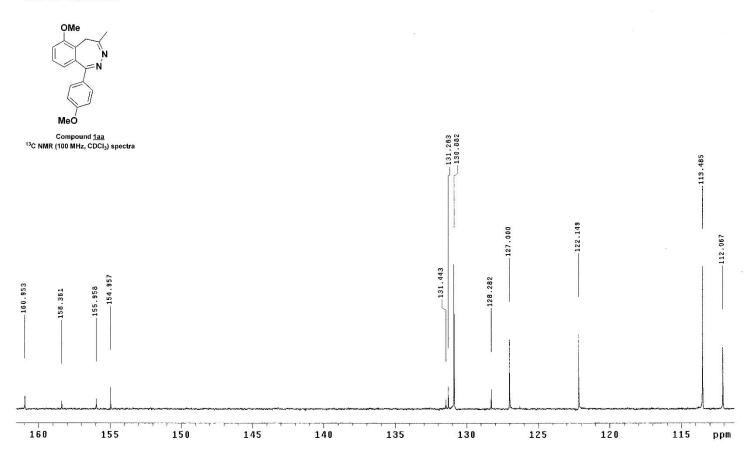
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 16 2015 Solvent: CDC13 Ambient temperature Total 736 repetitions

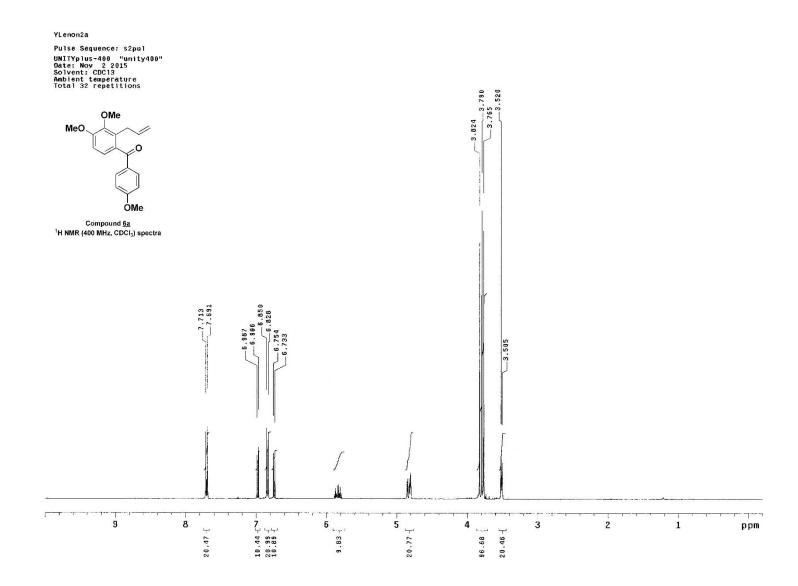


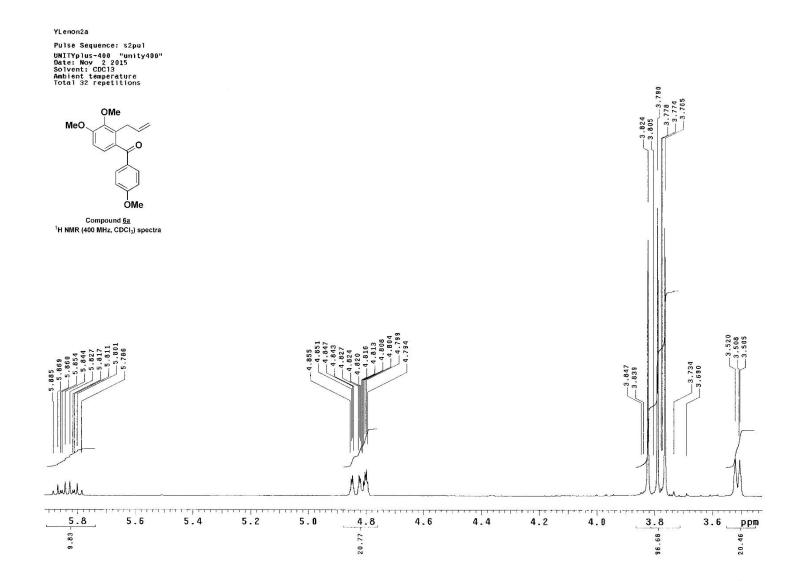
Compound 1aa

YL1040915

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 16 2015 Solvent: CDCl3 Ambient temperature Total 736 repetitions

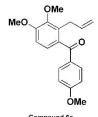




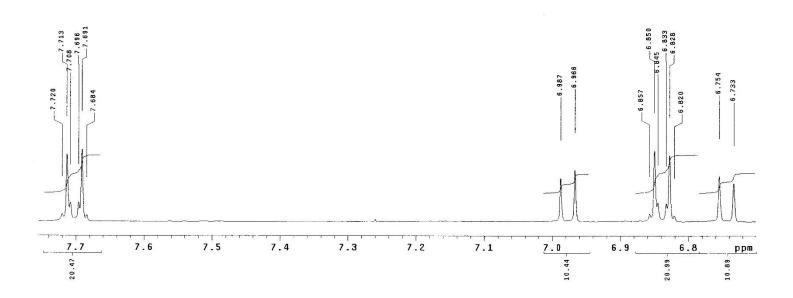


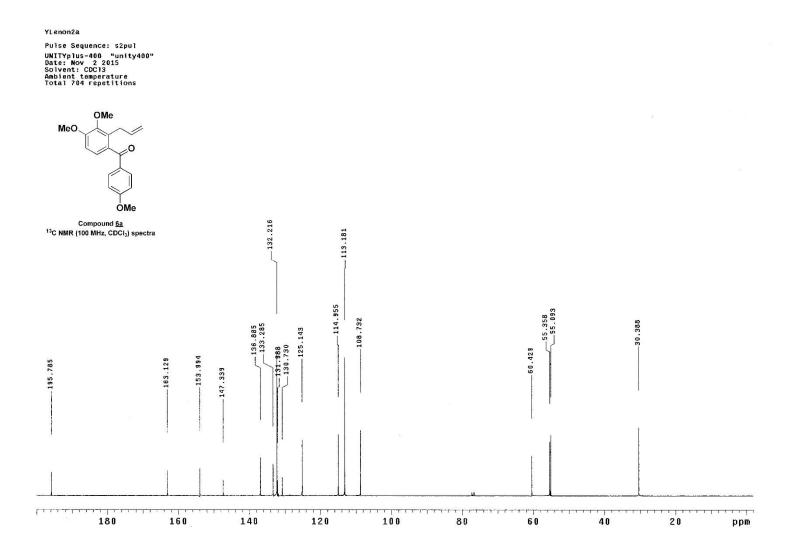
YLenon2a

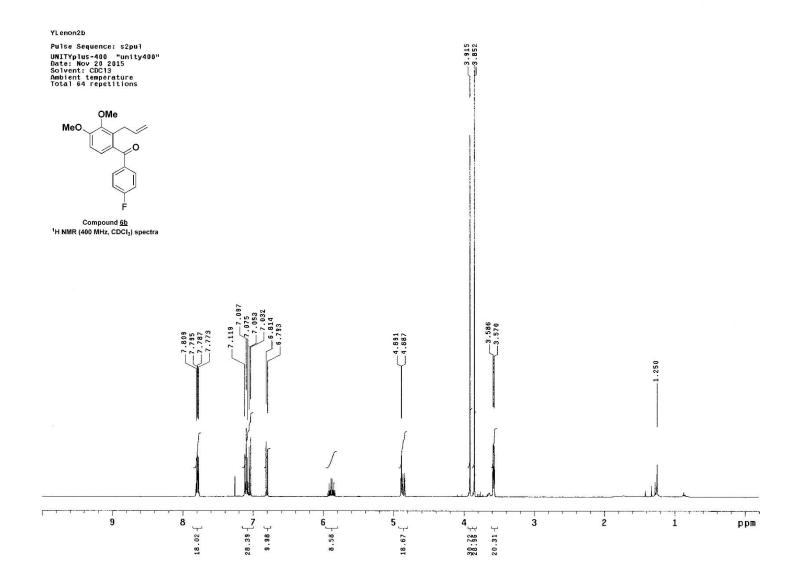
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDC13 Ambient temperature Total 32 repetitions

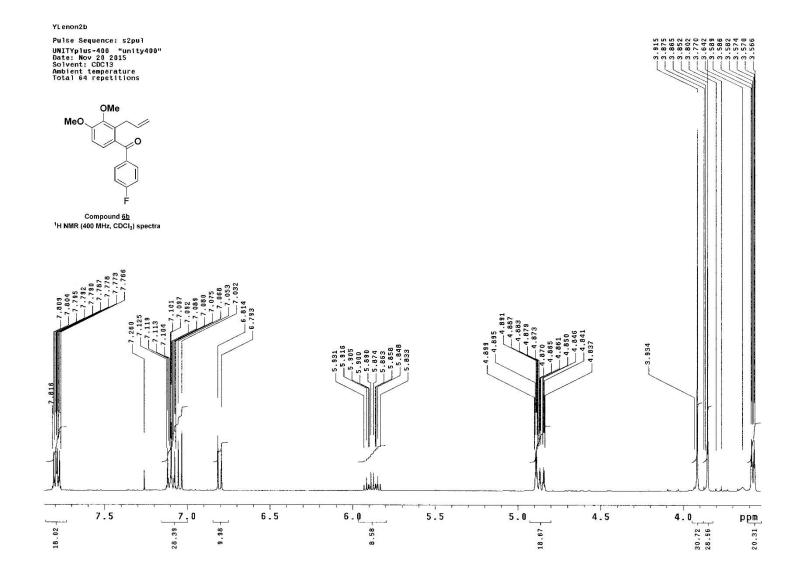


Compound <u>6a</u> ¹H NMR (400 MHz, CDCl₃) spectra

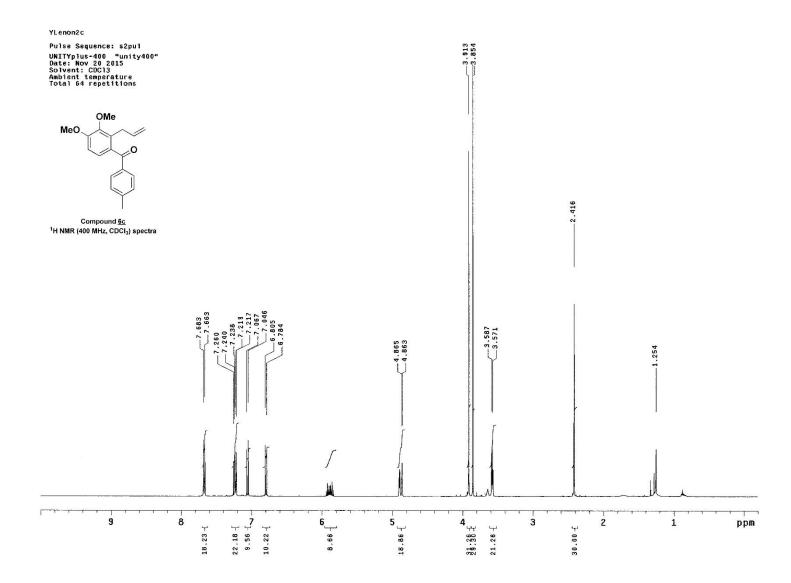


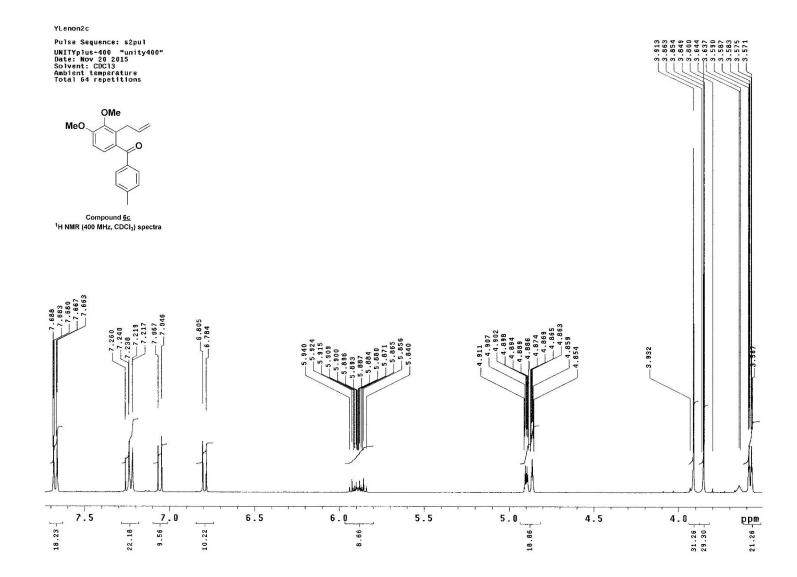




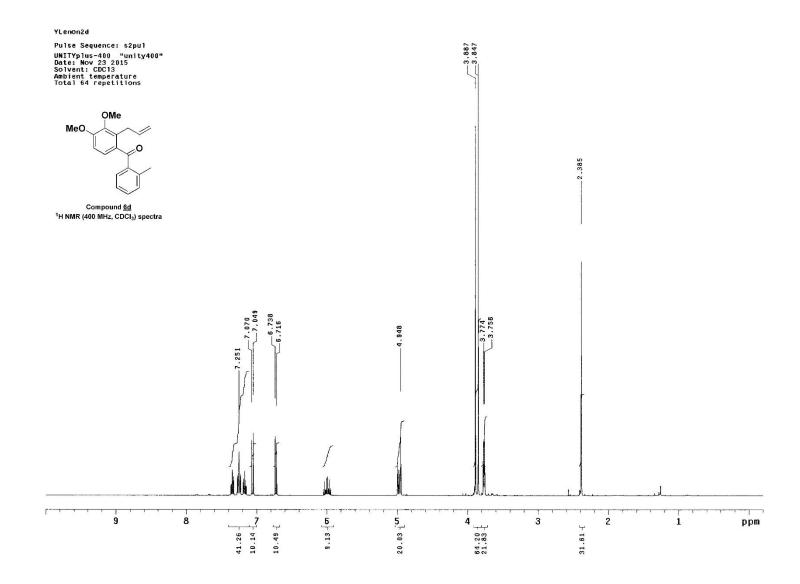


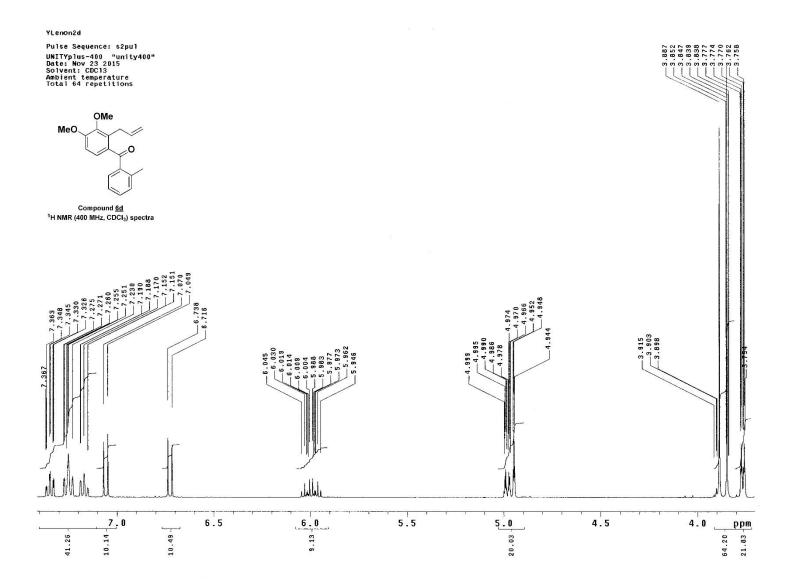
YLenon2b Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 20 2015 Solvent: CDC13 Ambient temperature Total 1360 repetitions OMe MeO Compound <u>6b</u> ¹³C NMR (100 MHz, CDCl₃) spectra 30.547 -115.221 115.380 108.936 55.714 967 77.318 .689 154.730 60.854 -29.653 000 147.756 195.876 -166.813 164.281 180 11111 1 1 1 1 1 4 · 4 · 1 · 4 · 1 · 1 · 4 · 3 <u>....</u> 160 140 20 120 100 80 60 ppm 40





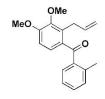
YLenon2c Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 20 2015 Solvent: CDC13 Ambient temperature Total 1040 repetitions OMe MeO. Compound <u>6c</u> ¹³C NMR (100 MHz, CDCI₃) spectra 012 163 397 30.615 125.894 108.868 -55.699 115.296 -137.166 135.779 __77.318 .000 __76.682 143.571 21.625 60.831 -29.660 -154.480 197.172 -147.650 **____** بط في في ال 111 111 1.1.1 The second 1.1.1 11 1 1 TT 180 160 140 120 100 80 60 40 20 ppm



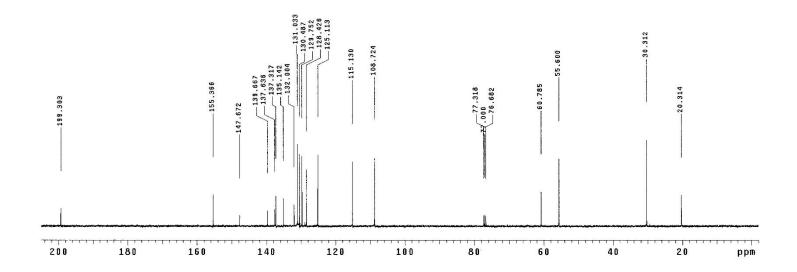


YLenon2d

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 23 2015 Solvent: CDCl3 Ambient temperature Total 224 repetitions

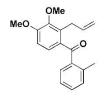


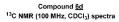
Compound <u>6d</u> ¹³C NMR (100 MHz, CDCl₃) spectra

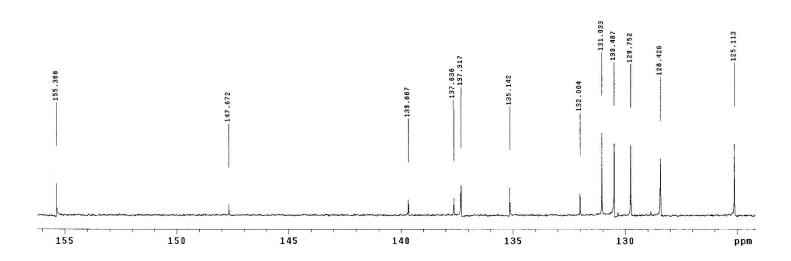


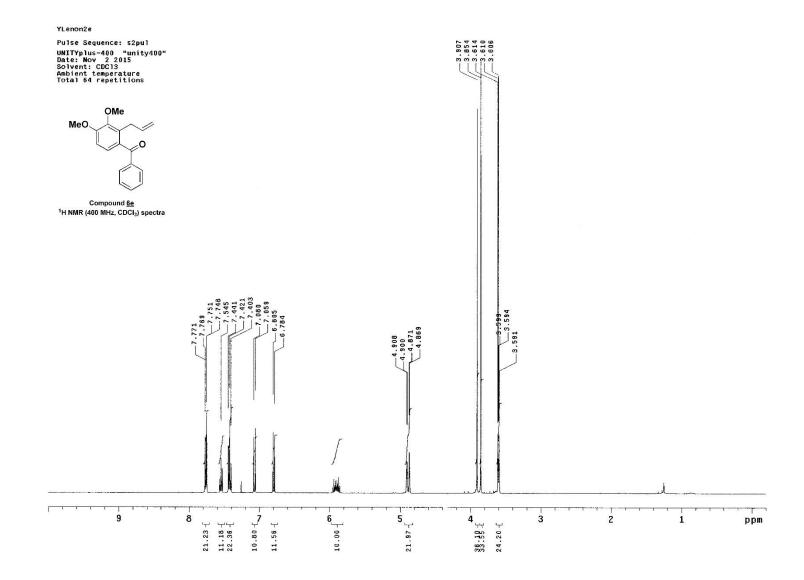
YL enon2d

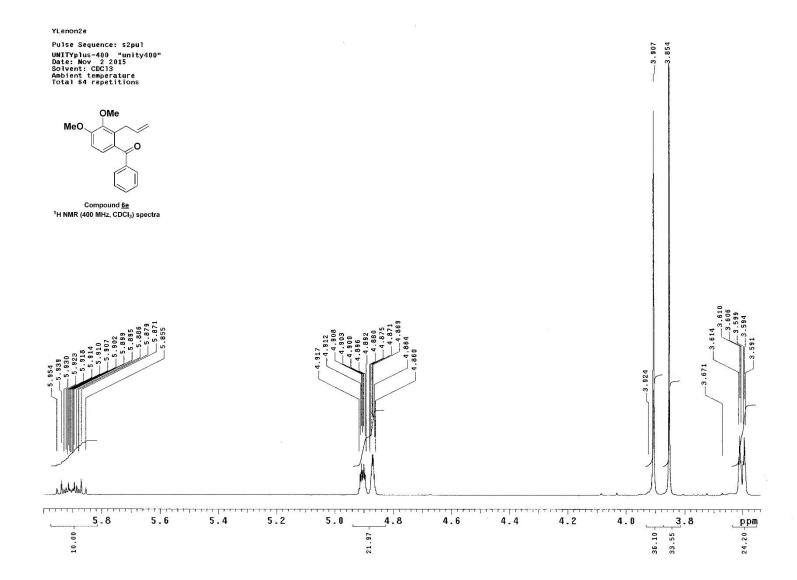
Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Nov 23 2015 Solvent: CDC13 Ambient temperature Total 224 repetitions





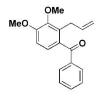




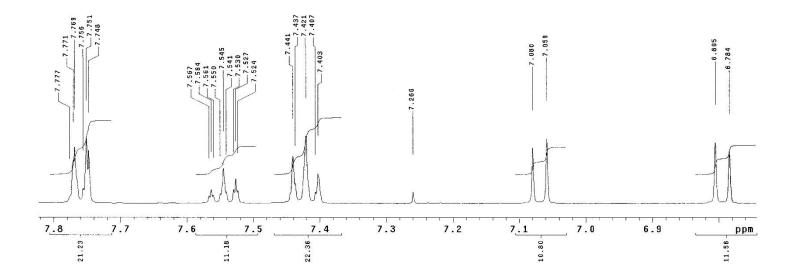


YLenon2e

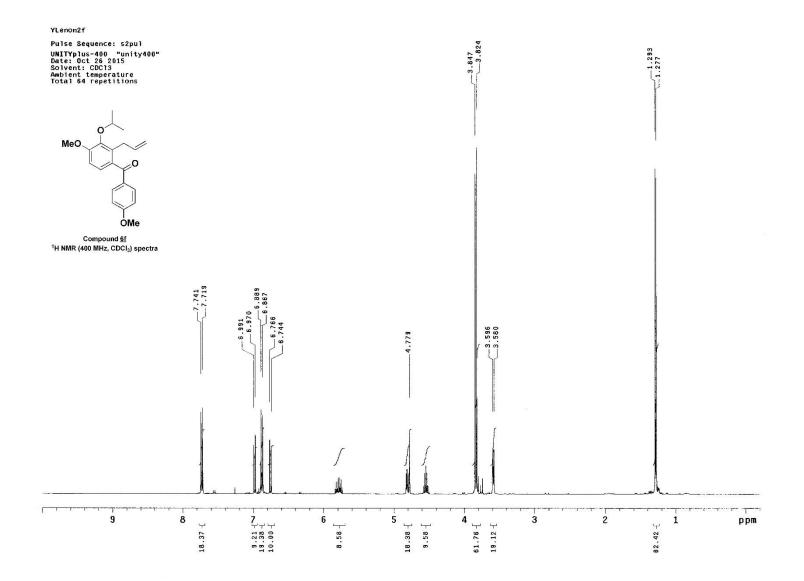
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

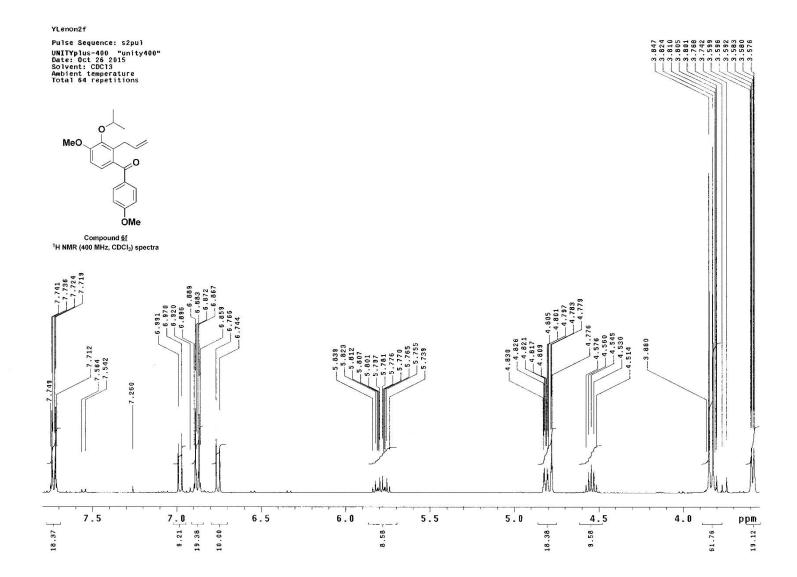


Compound <u>6e</u> ¹H NMR (400 MHz, CDCI₃) spectra

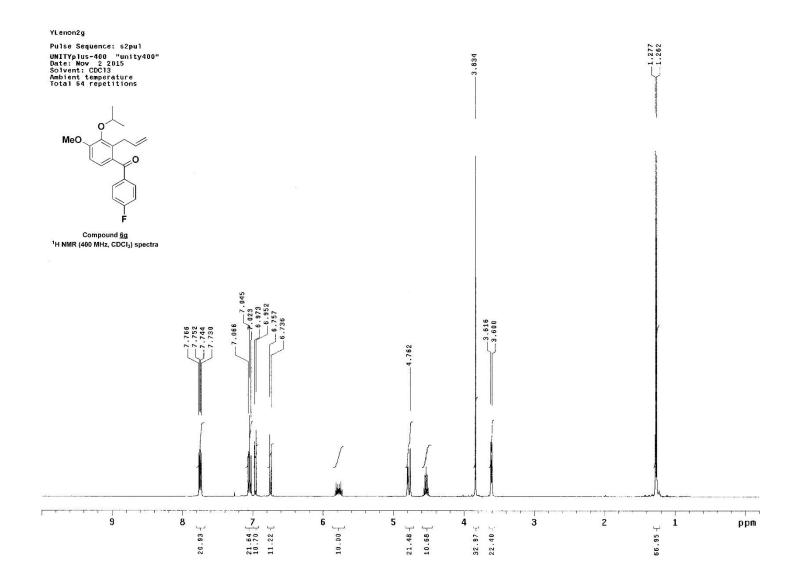


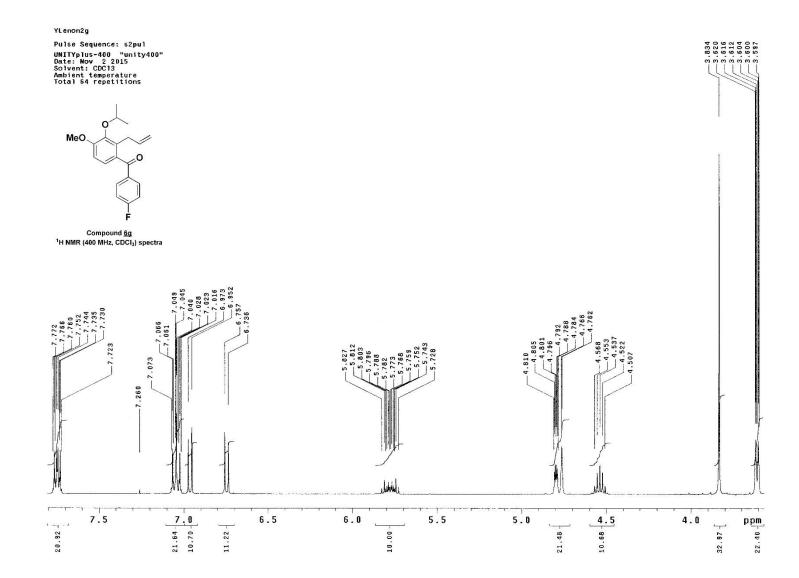
YLenon2e Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDC13 Ambient temperature Total 2208 repetitions OMe MeO. Compound <u>6e</u> ¹³C NMR (100 MHz, CDCl₃) spectra 131.731 -130.139 128.145 126.235 132.633 30.524 108.815 115.266 .55,654 137.120 137.120 134.209 60.793 154.646 77.318 682 197.370 138. 147.650 فجؤجف بلحاف فيط بناء سفيف الربار عرجاني بمقتلك 111 - - -1 180 140 100 80 40 160 120 60 20 ppm

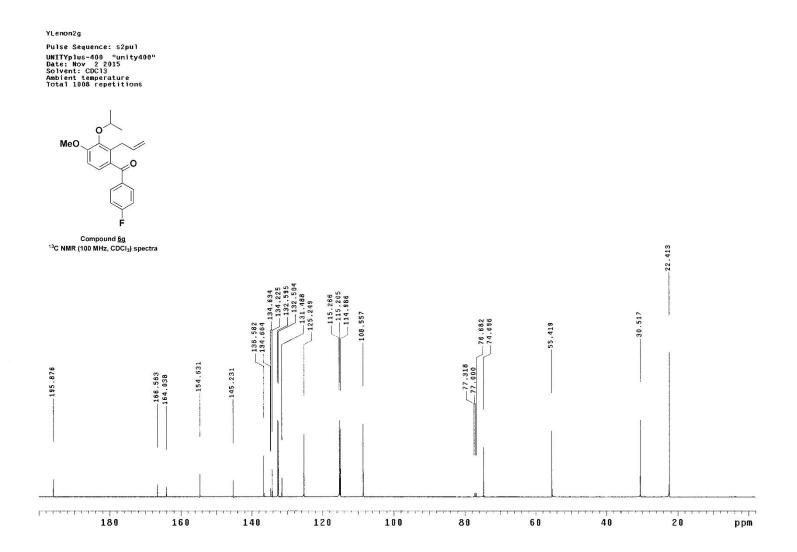




YLenon2f Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Oct 26 2015 Solvent: CDC13 Ambient temperature Total 3568 repetitions MeO ÓМе 113.303 Compound <u>6f</u> ¹³C NMR (100 MHz, CDCI₃) spectra -132.383 22.482 115.243 55.472 55.290 626 30.752 124.787 136.643 - 133.838131.071 108. 74.680 145.133 163.220 154.237 196.346 Liliii. 111 1 1 1 1 -4-4-1-4-4-4-4-4 **** 180 160 140 20 120 100 80 60 40 ppm

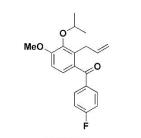




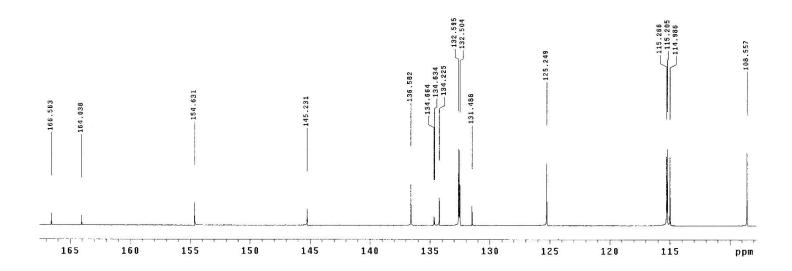


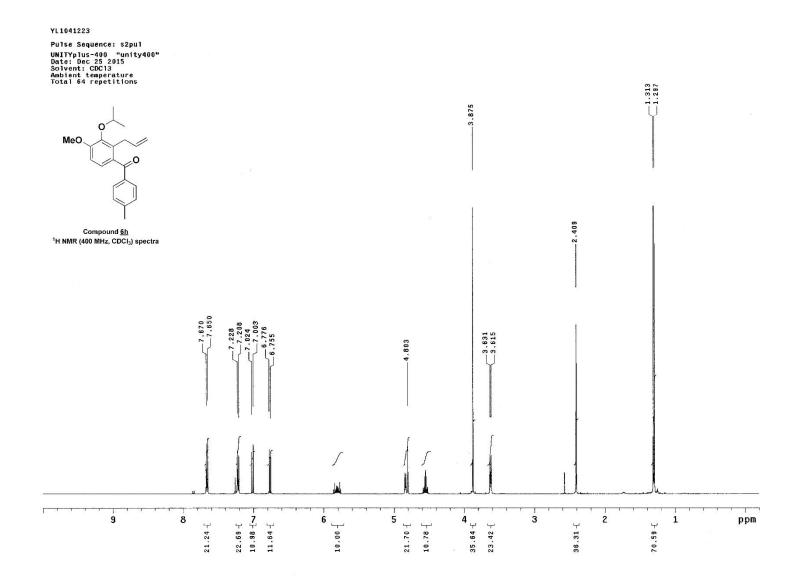
YLenon2g

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDCl3 Ambient temperature Total 1008 repetitions



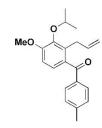
Compound <u>6q</u> ¹³C NMR (100 MHz, CDCI₃) spectra



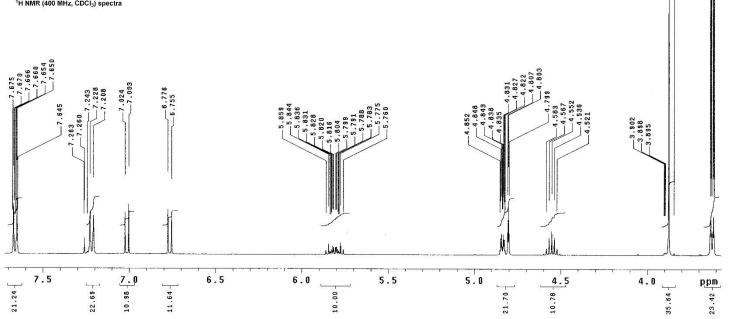


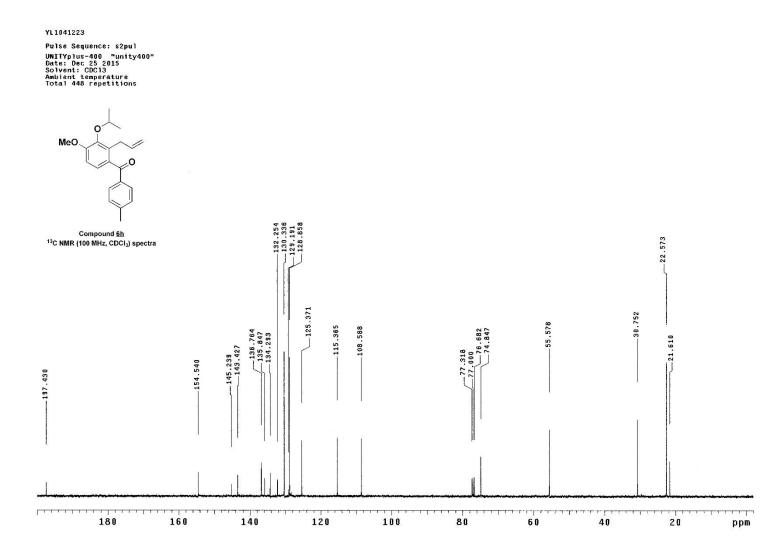
YL1041223

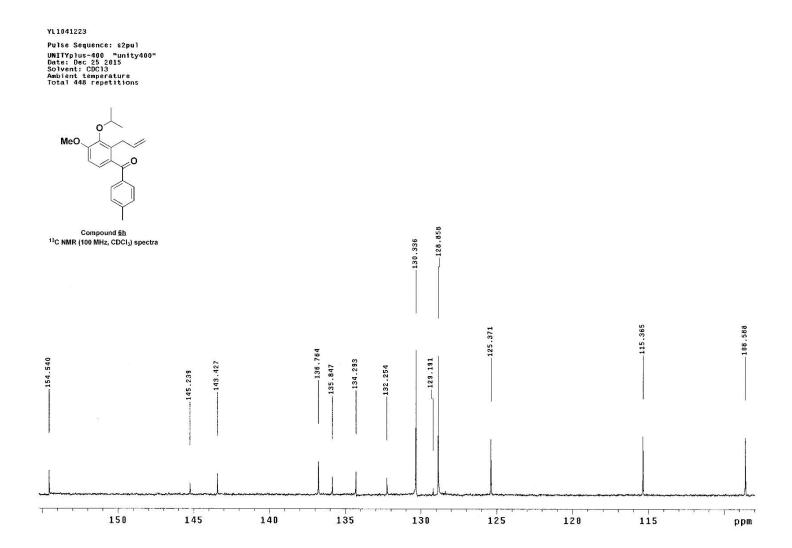
Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Dec 25 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

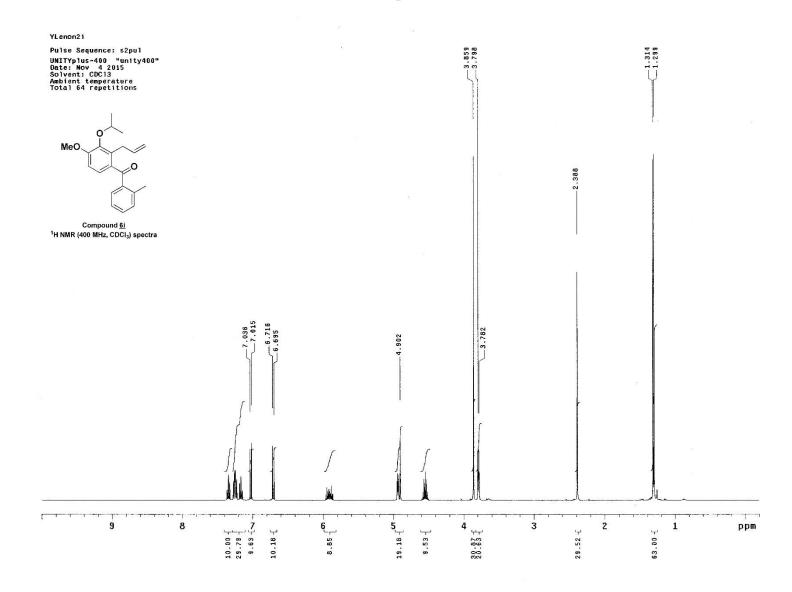


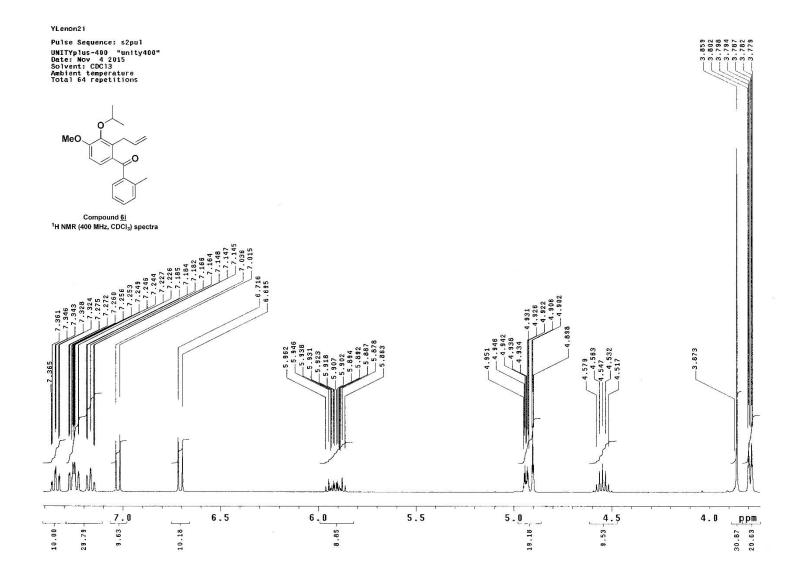
Compound <u>6h</u> ¹H NMR (400 MHz, CDCI₃) spectra





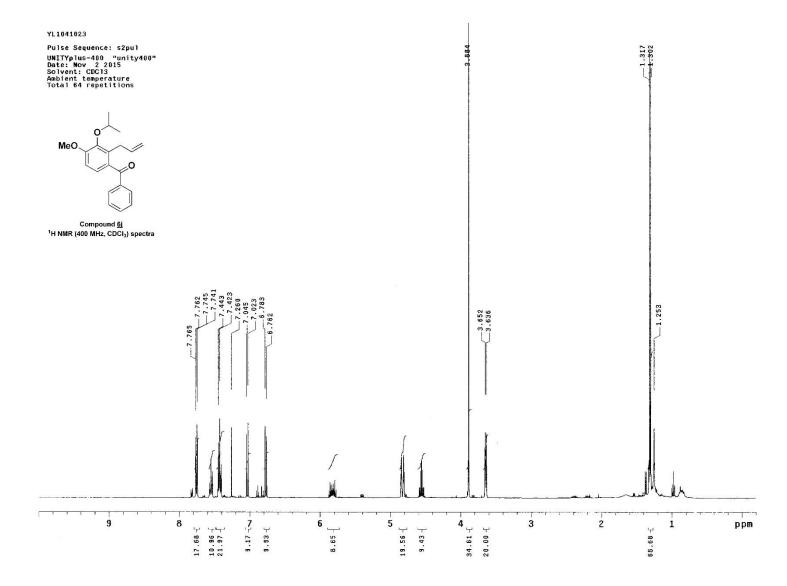




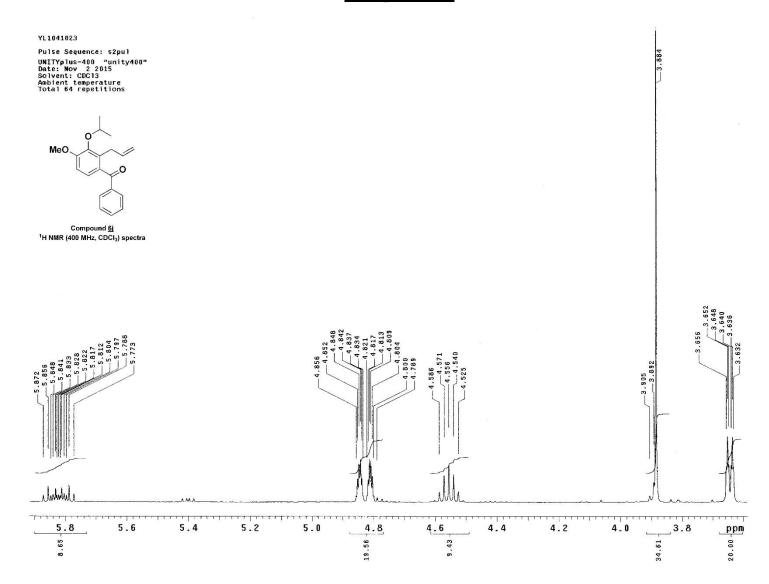


YL enon21 Pulse Sequence: s2pul UNITYPlus-400 "unity400" Date: Nov 4 2015 Solvent: CDC13 Ambient temperature Total 1696 repetitions MeO. Compound <u>6i</u> ¹³C NMR (100 MHz, CDCI₃) spectra 22.519 30.524 108.512 -76.682 -74.832 115.220 139.690 137.810 137.022 137.022 135.453 55.540 374 100 155.442 77.318 145.322 199.545 200 TTT TITI 1 120 100 20 180 160 140 80 60 40 ppm

<u>Compound 6j</u>



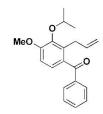
<u>Compound 6j</u>



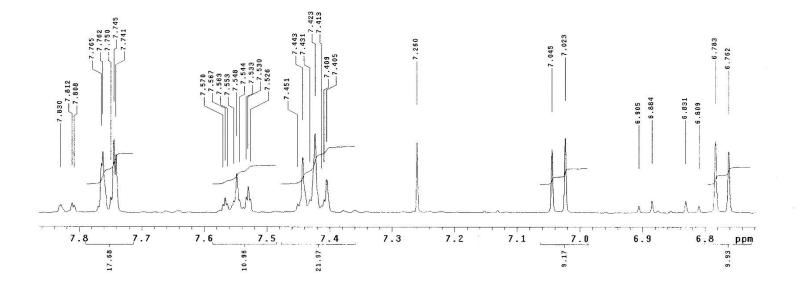
<u>Compound 6j</u>

YL1041023

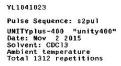
Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions

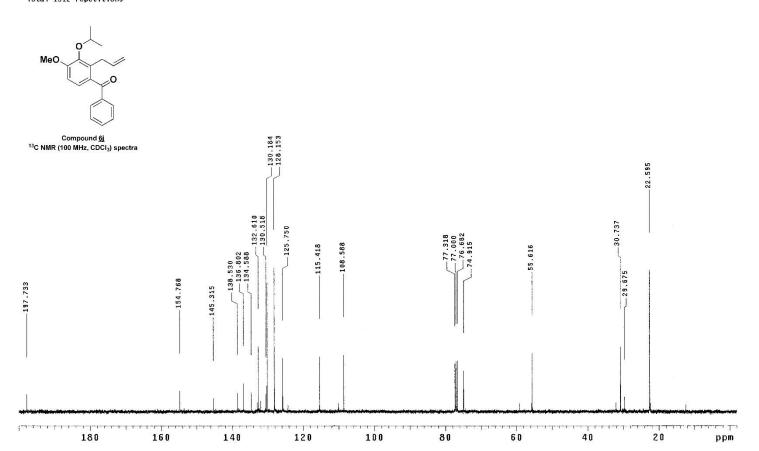


Compound <u>6i</u> ¹H NMR (400 MHz, CDCI₃) spectra



<u>Compound 6j</u>

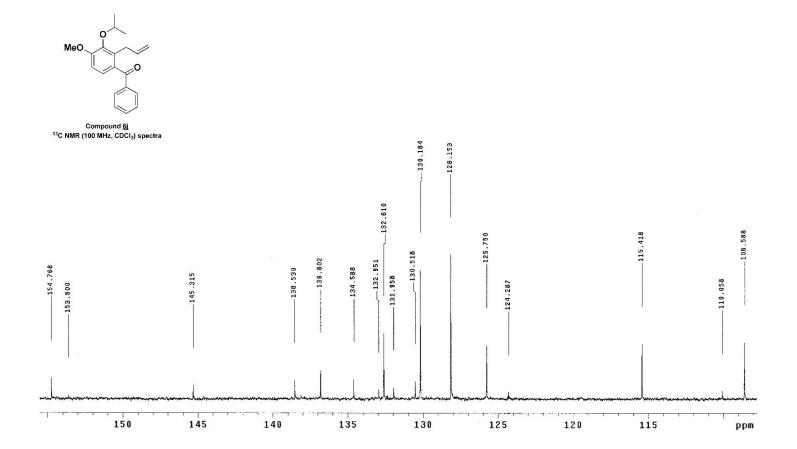


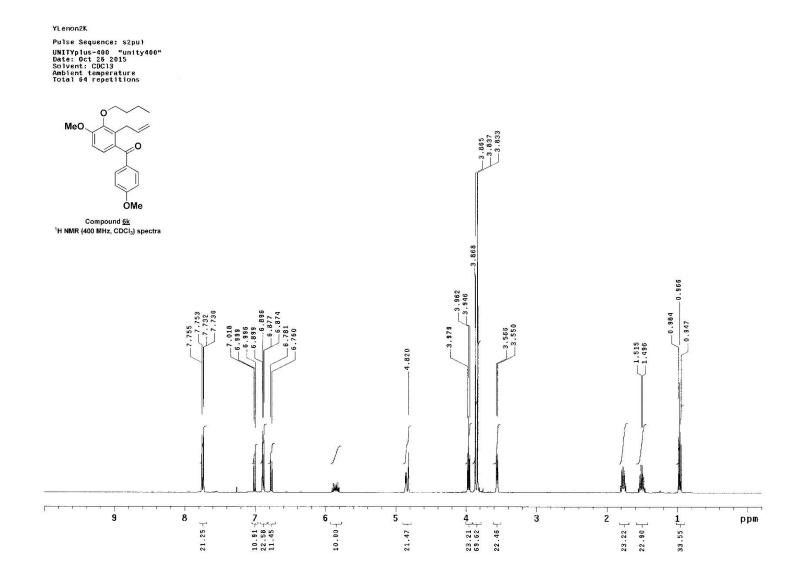


<u>Compound 6j</u>

YL1041023

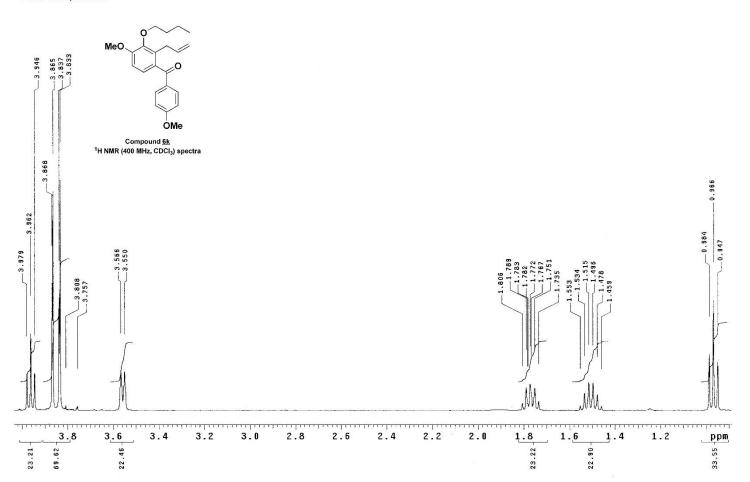
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDC13 Ambient temperature Total 1312 repetitions





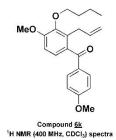
YLenon2K

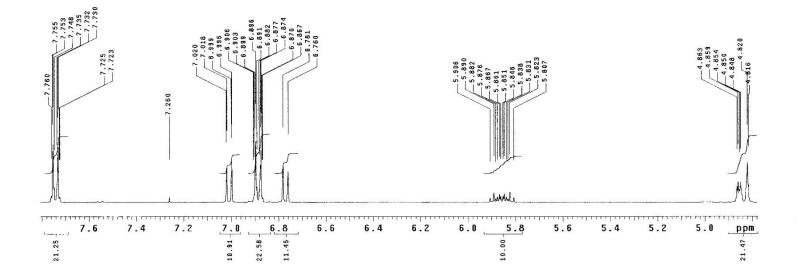
Pulse Sequence: s2pu) UNITVplus-400 "unity400" Date: Oct 26 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions

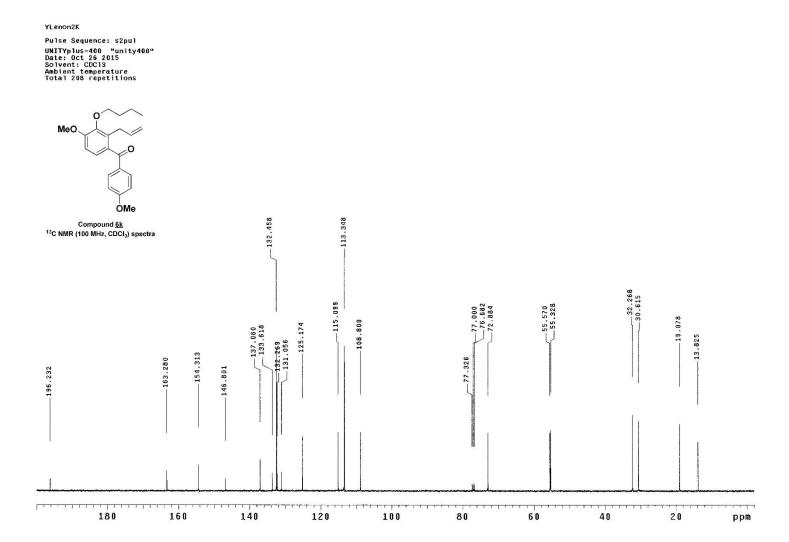


YLenon2K

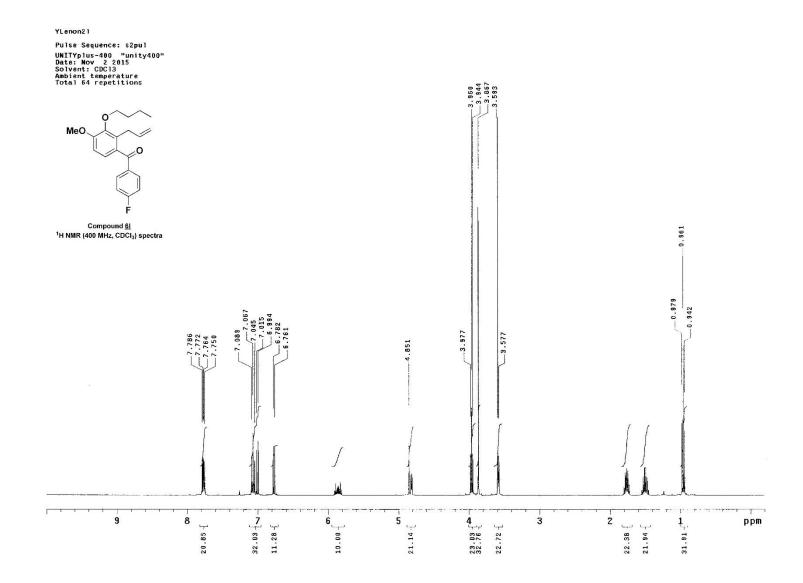
Pulse Sequence: s2pu) UNITYplus-400 "unity400" Date: oct 26 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions

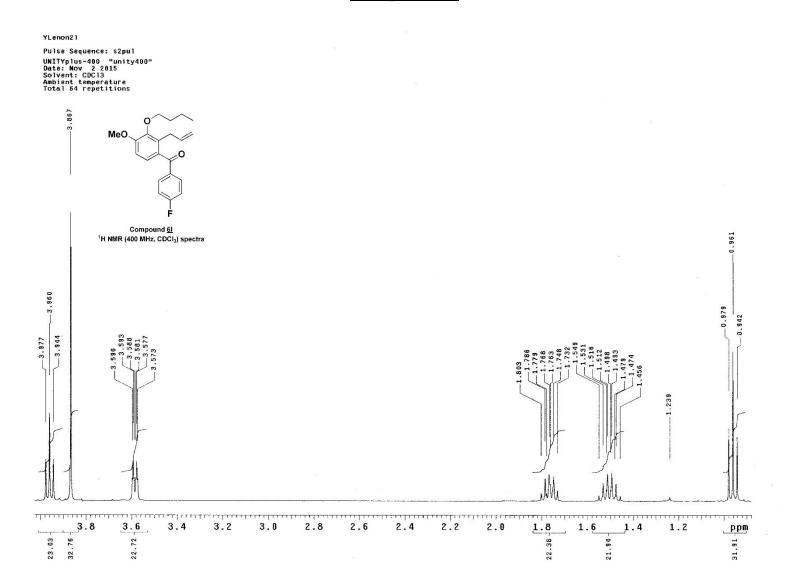






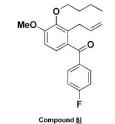
S-150



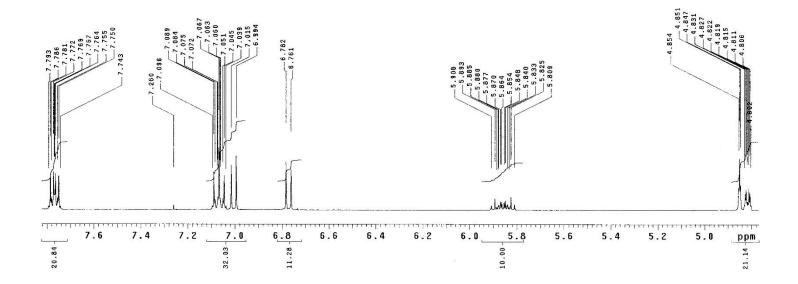


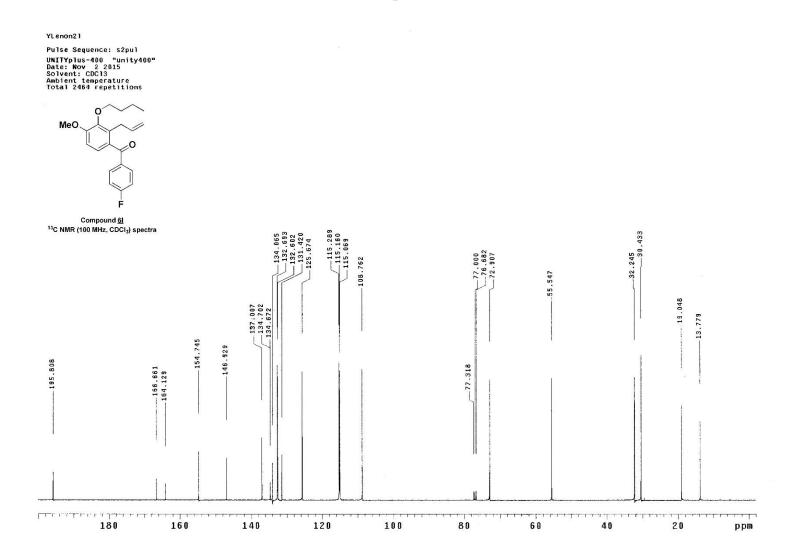
YLenon21

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions

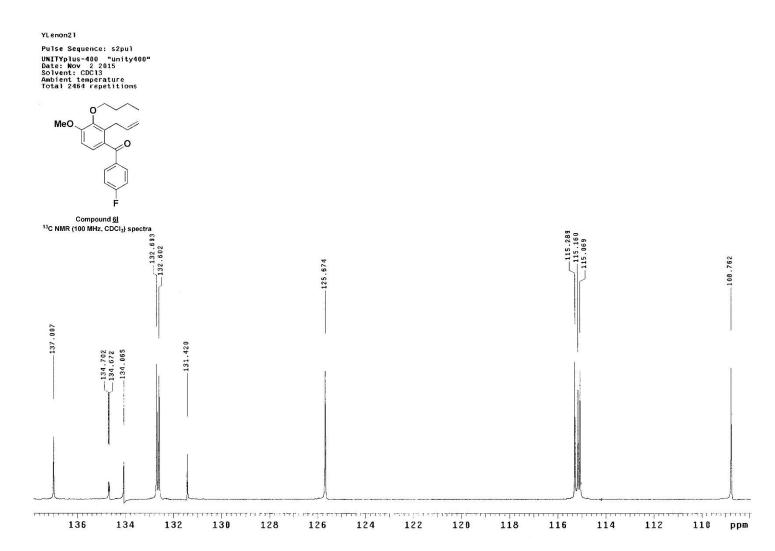


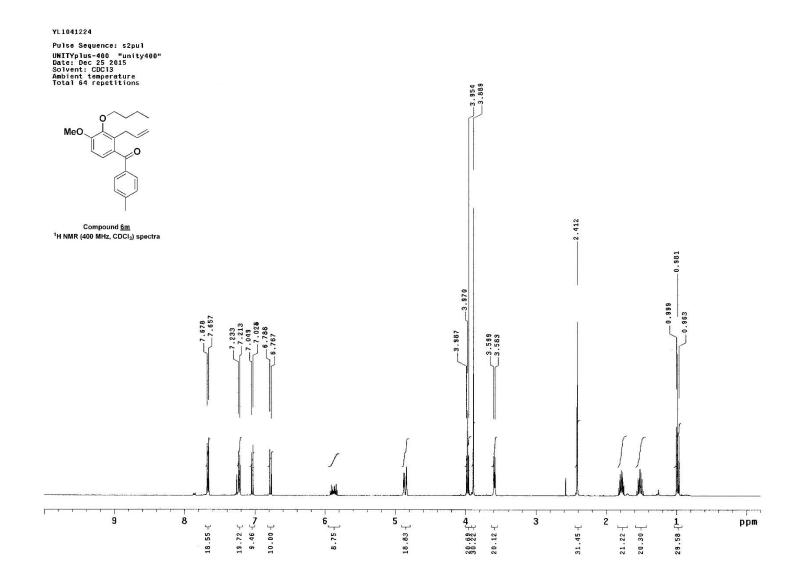
¹H NMR (400 MHz, CDCl₃) spectra

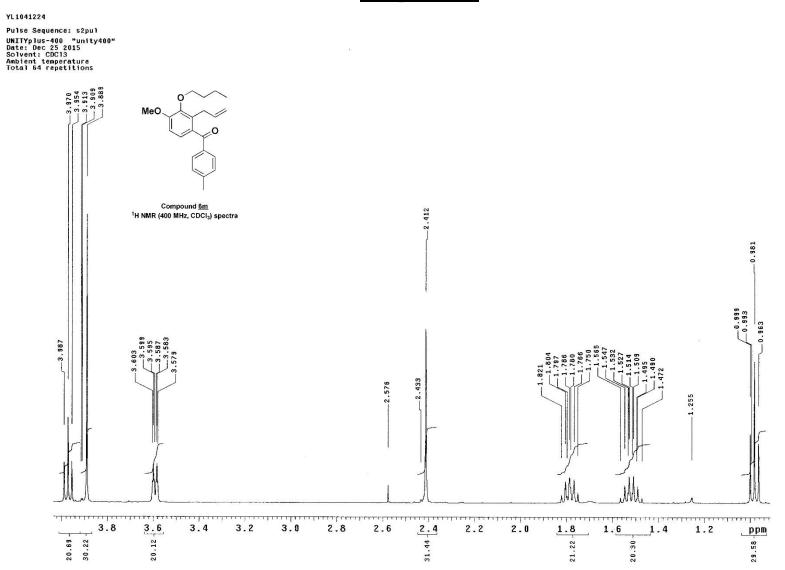




S-154



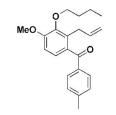




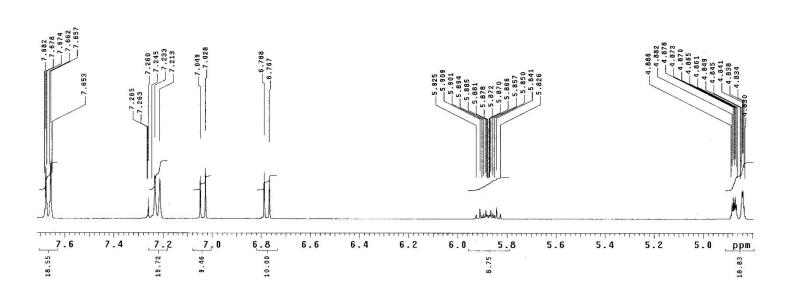
S-157

YL1041224

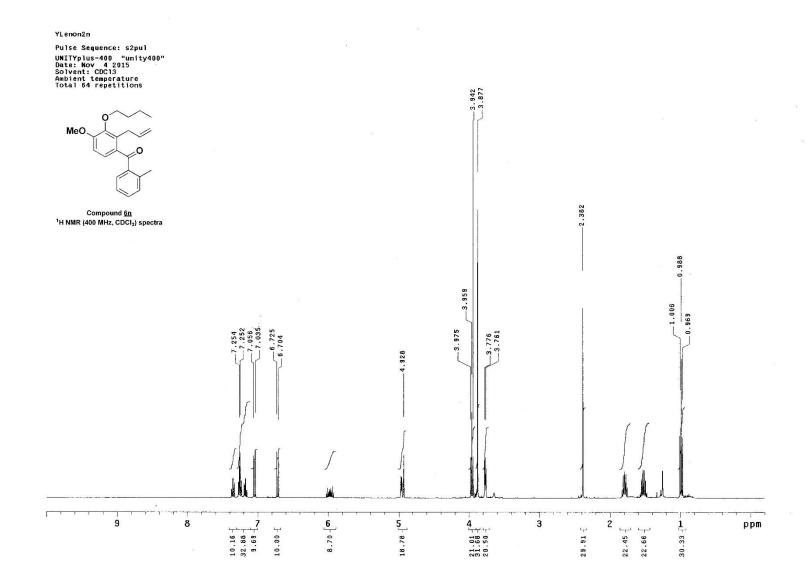
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Dec 25 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

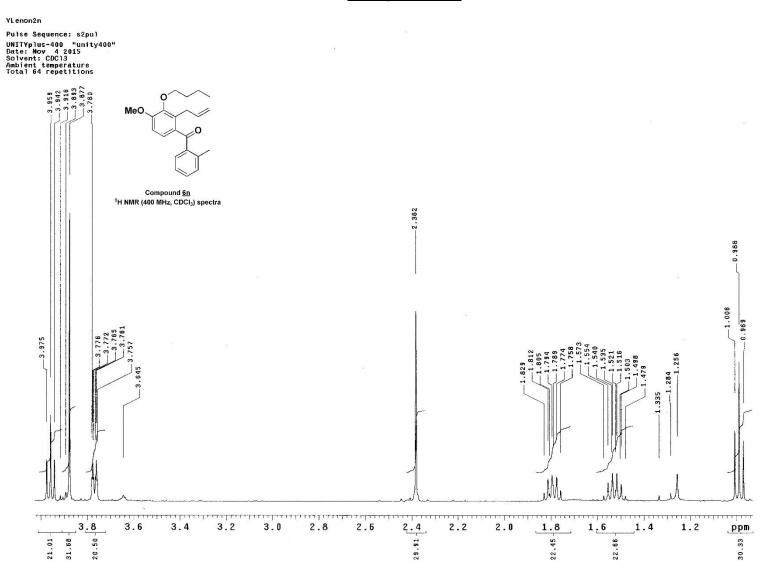


Compound <u>6m</u> ¹H NMR (400 MHz, CDCl₃) spectra



YL1041224 Pulse Sequence: s2pul UNITYPIUS-400 "unity400" Date: Dec 25 2015 Solvent: CDC13 Ambient temperature Total 1024 repetitions 0 MeO Compound 6m ¹³C NMR (100 MHz, CDCl₃) spectra 32.344 125.735 55.661 19.161 115.198 108.770 137.166 77.318 -146.899 _____143.495 154.593 13.908 -21.617 197.279 -1-1-1 180 160 140 120 100 80 60 40 20 ppm

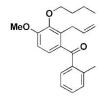




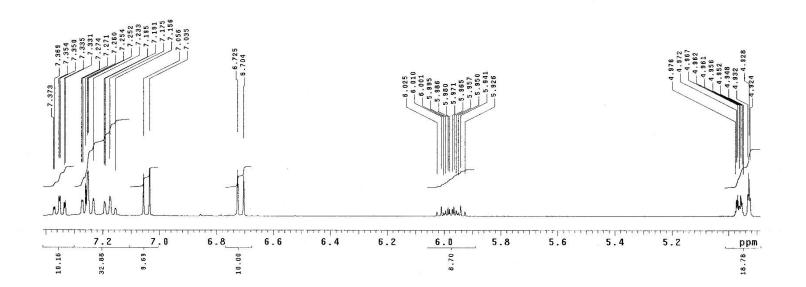
S-161

YL enon2n

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 4 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions

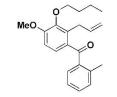


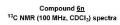
Compound <u>6n</u> ¹H NMR (400 MHz, CDCl₃) spectra

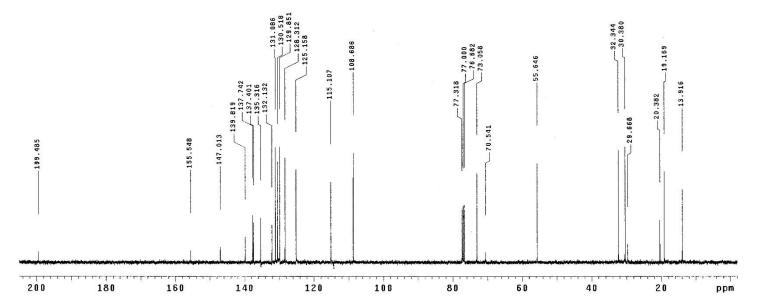


YLenon2n

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 4 2015 Solvent: CDC13 Ambient temperature Total 2144 repetitions

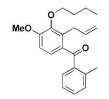


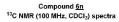


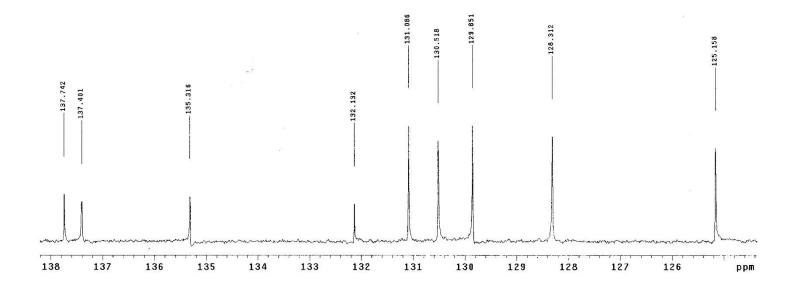


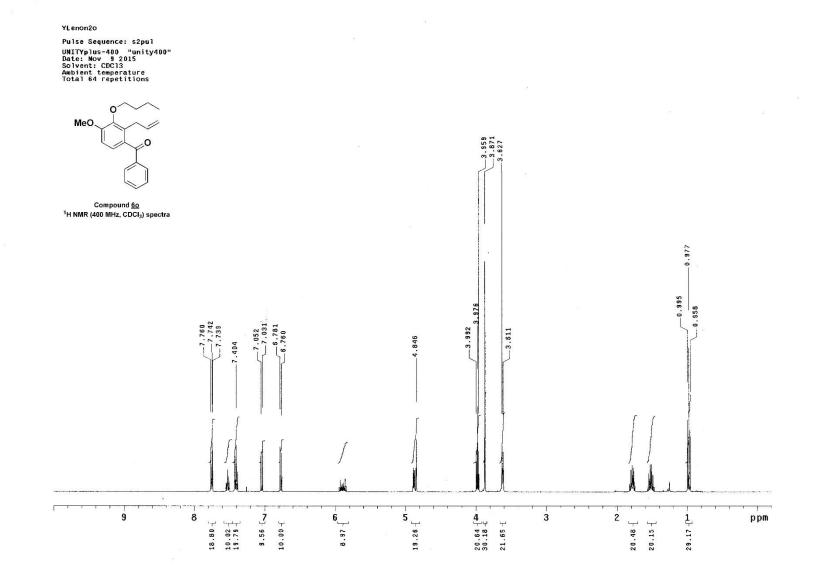
YLenon2n

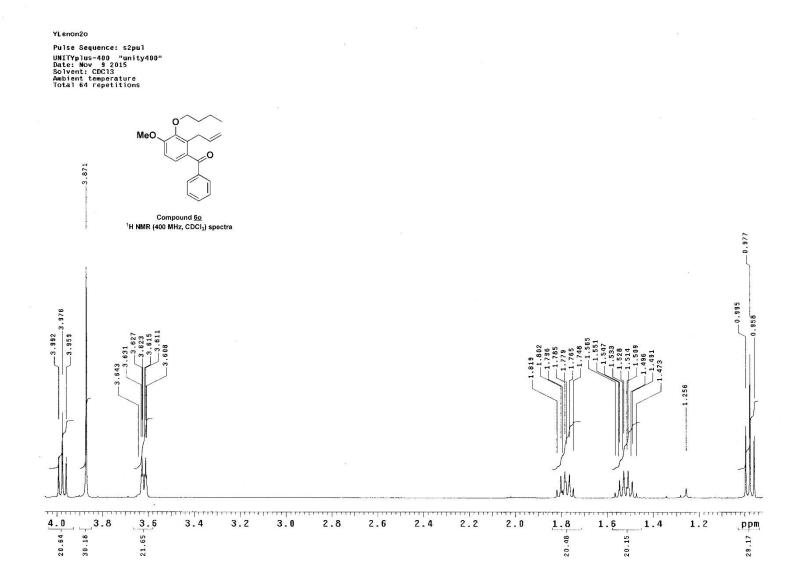
Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Nov 4 2015 Solvent: CDC13 Ambient temperature Total 2144 repetitions







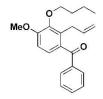




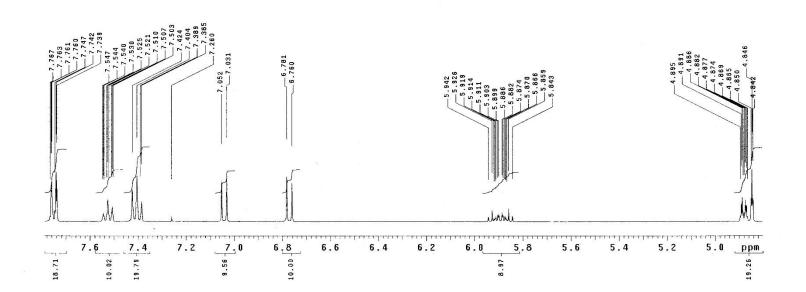
S-166

YL enon20

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 9 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

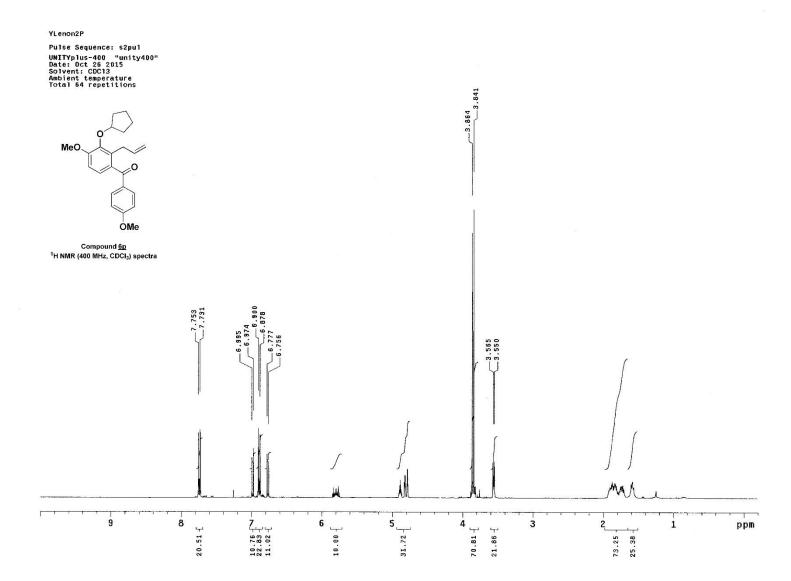


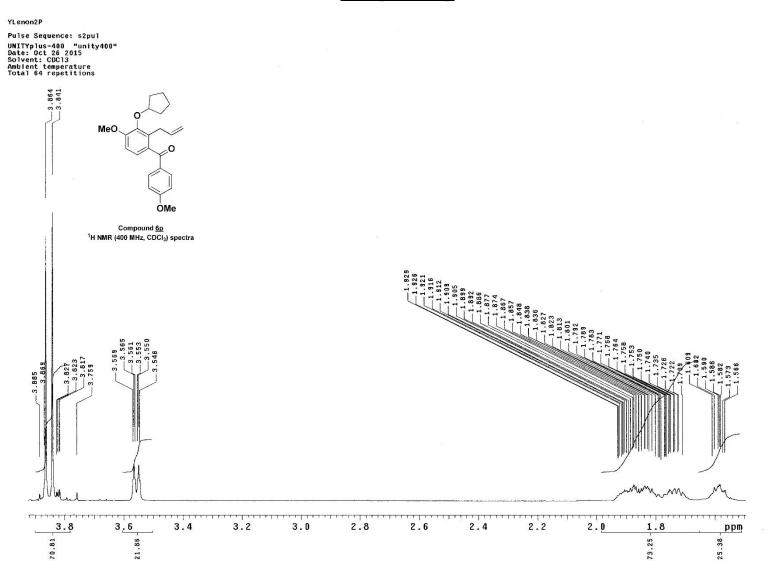
Compound <u>60</u> ¹H NMR (400 MHz, CDCI₃) spectra



S-167

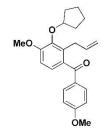
YLenon20 Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 9 2015 Solvent: CDCl3 Ambient temperature Total 128 repetitions 0 MeO. Compound <u>60</u> ¹³C NMR (100 MHz, CDCI₃) spectra 131.647 130.063 128.062 132.527 32.253 126.038 115.107 19,063 108.679 401 137.098 134.194 555 13.810 55 154.715 77.326 138. 146.854 197.385 180 ···· 1 11 1 - F- F-T-T-T-T-T-T-T de la de T 1 80 160 140 120 100 60 20 40 ppm

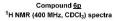


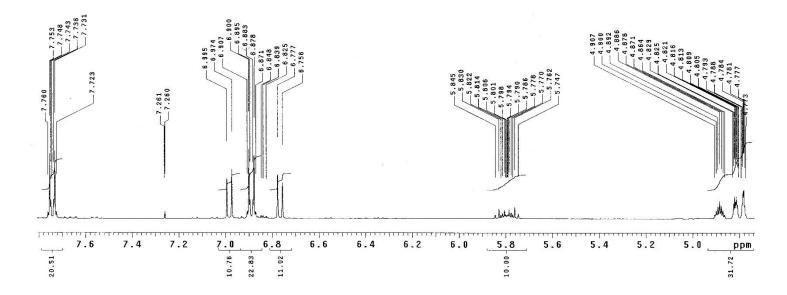


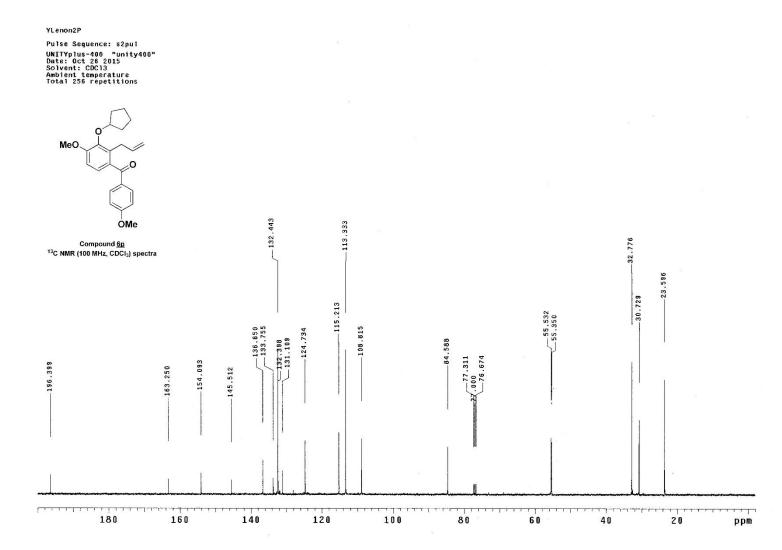
YL enon2P

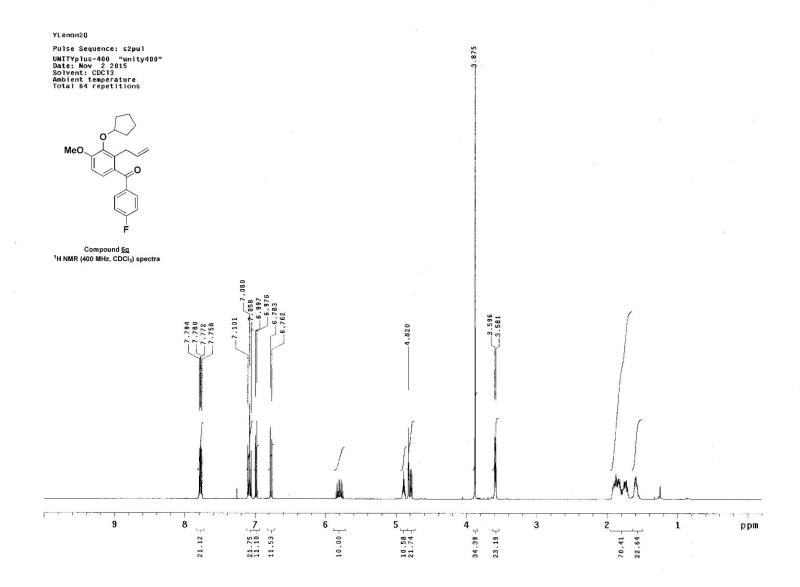
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Oct 26 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions





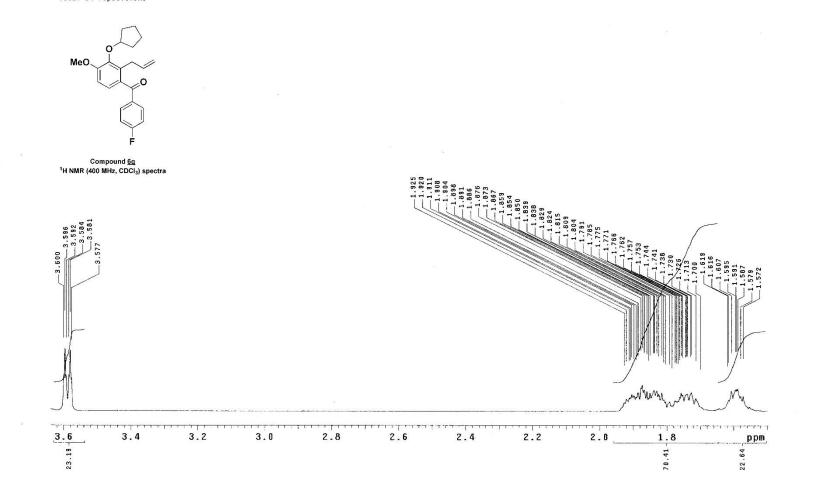






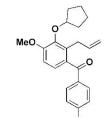
YL enon2Q

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions

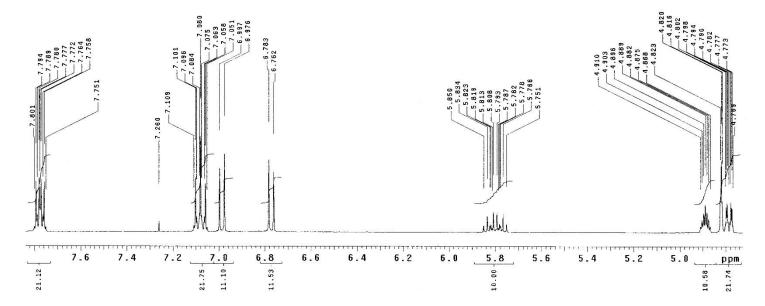


YL enon2Q

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDCl3 Ambient temperature Total 54 repetitions

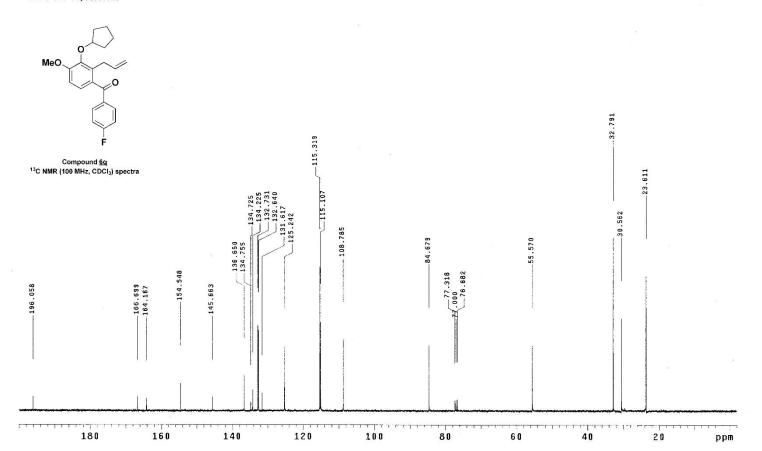


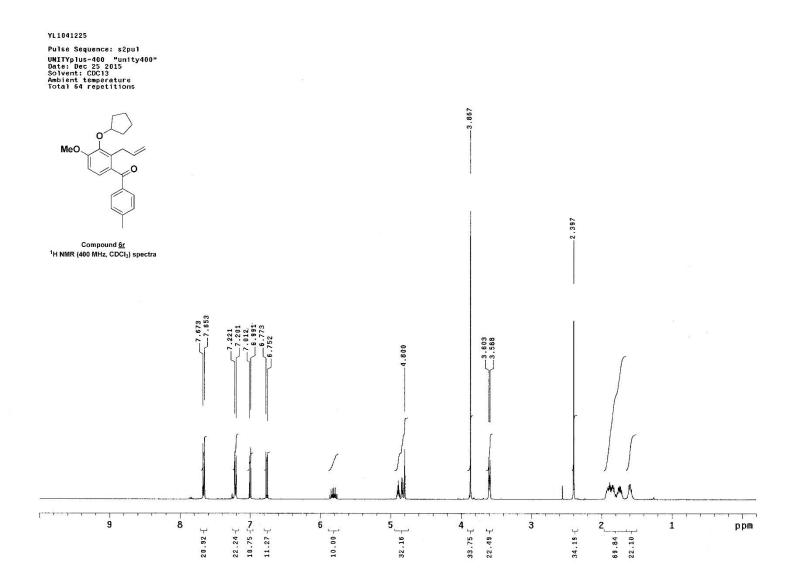
Compound <u>6q</u> ¹H NMR (400 MHz, CDCl₃) spectra

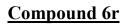


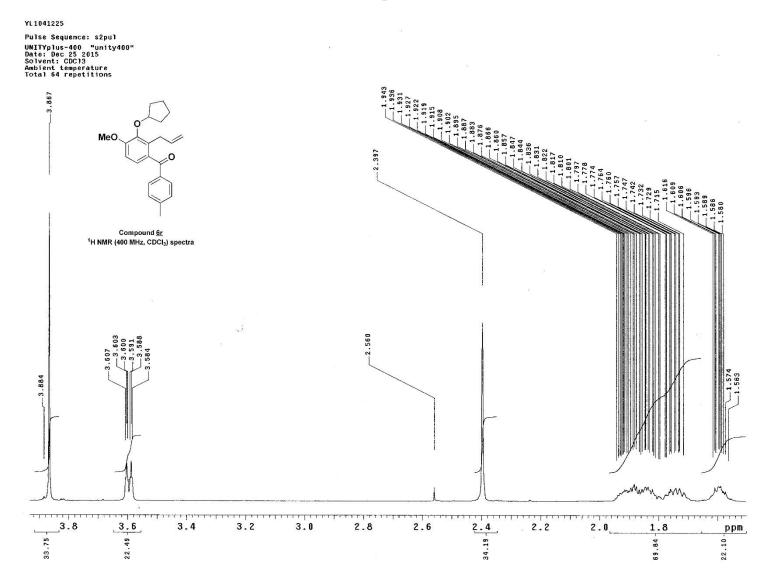
YLenon2Q

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDC13 Ambient temperature Total 160 repetitions



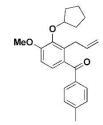




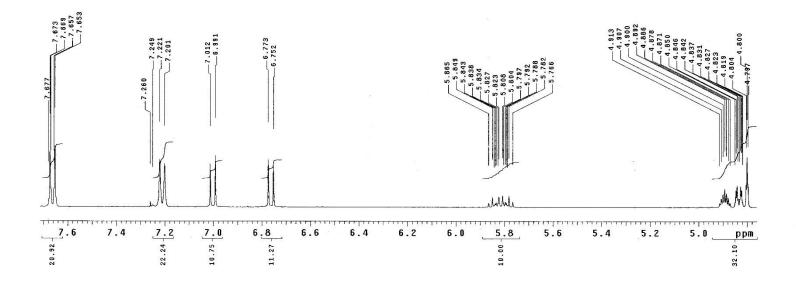


YL1041225

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Dec 25 2015 Solvent: CDC13 Ambient temporature Total 64 repetitions

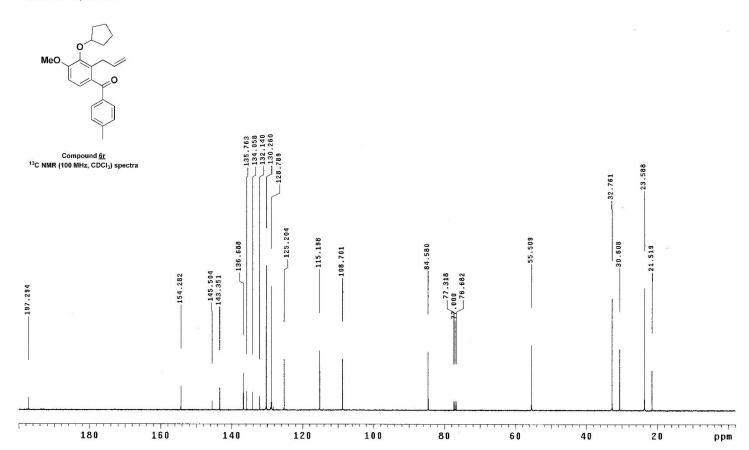


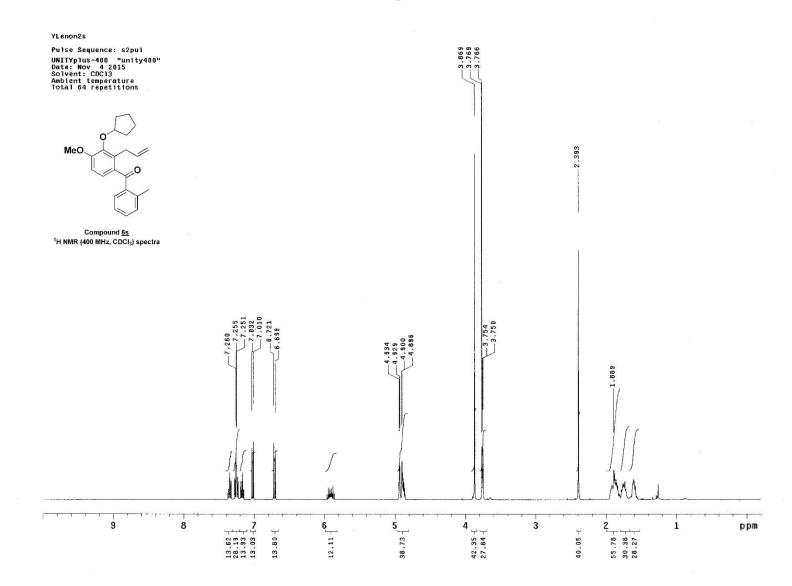
Compound <u>6r</u> ¹H NMR (400 MHz, CDCI₃) spectra

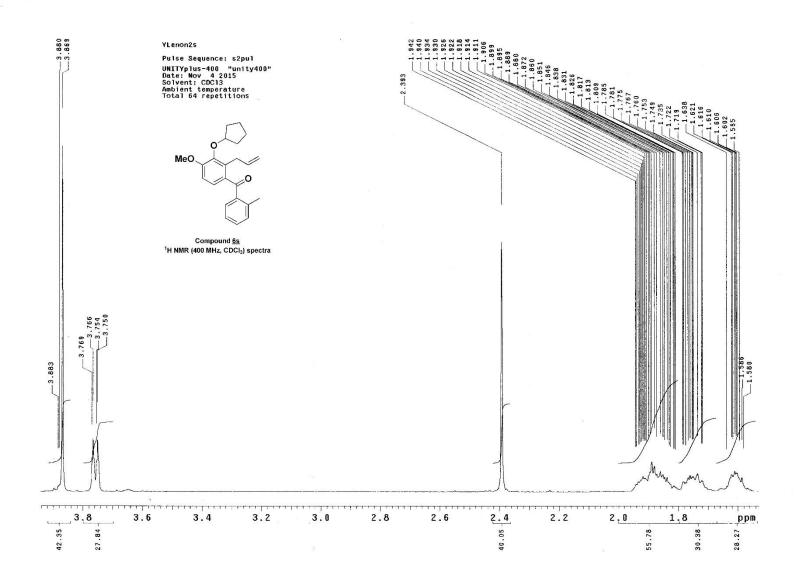


YL1041225

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Dec 25 2015 Solvent: CDC13 Ambient temperature Total 336 repetitions

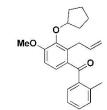




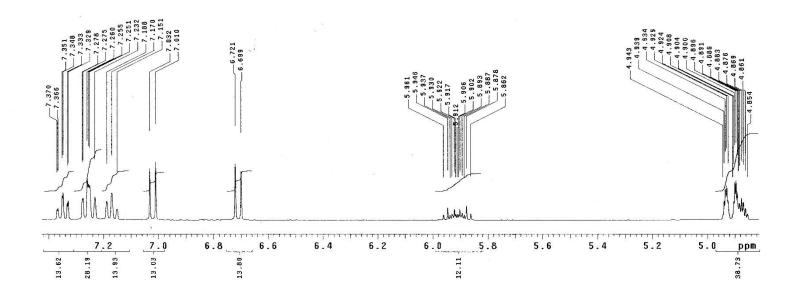


YLenon2s

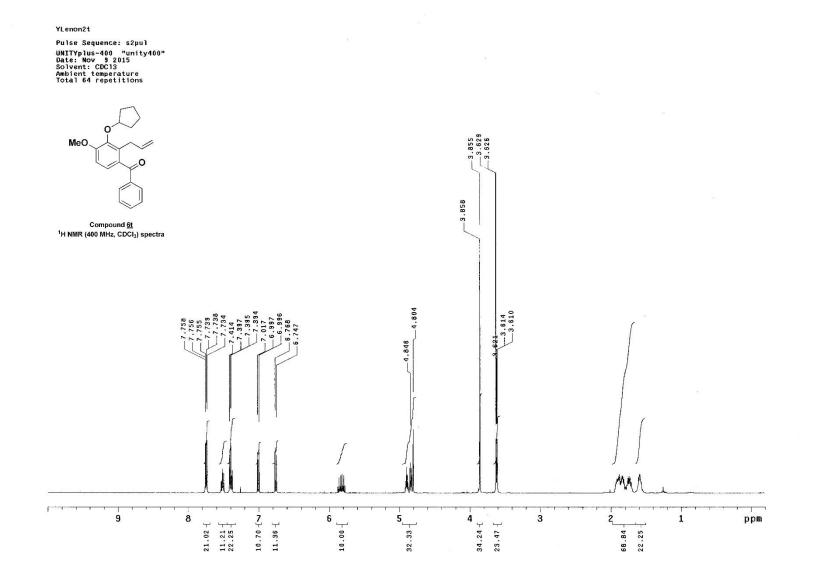
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 4 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



Compound <u>6s</u> ¹H NMR (400 MHz, CDCI₃) spectra

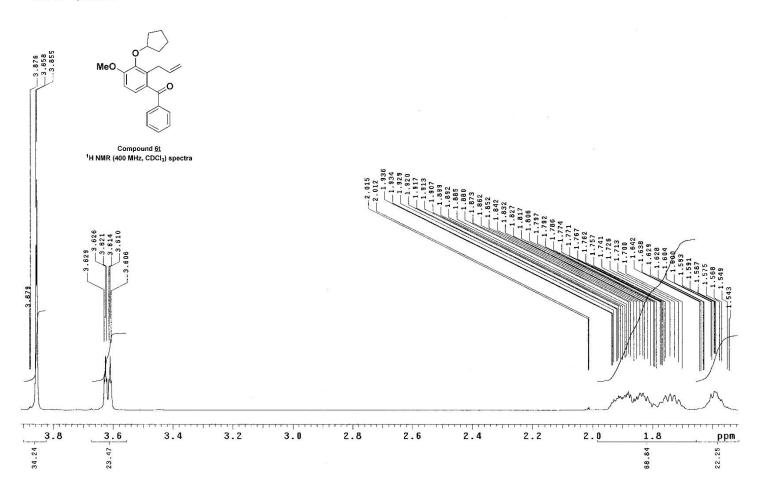


YLenon2s Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Nov 4 2015 Solvent: CDC13 Ambient temperature Total 928 repetitions MeO. Compound <u>6s</u> ¹³C NMR (100 MHz, CDCI₃) spectra .32.806 -23.649 30.441 108.663 115.167 .840 55.563 84.732 7137 __77.311 .000 __76.682 389 .155.268 199.553 .145.701 0 111 1 T T T T - 1 180 200 160 140 120 100 80 60 40 20 ppm



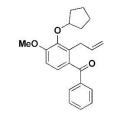
YLenon2t

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 9 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

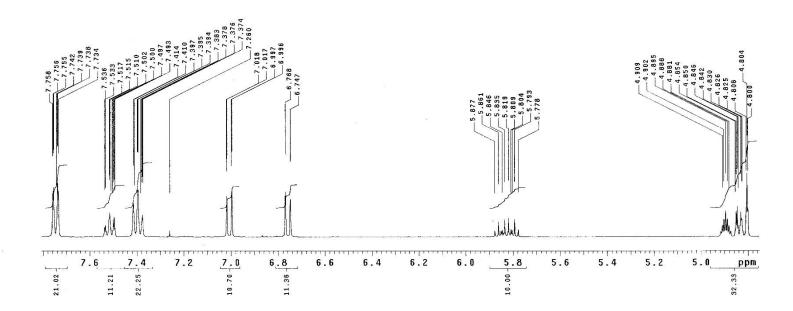


YLenon2t

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 9 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

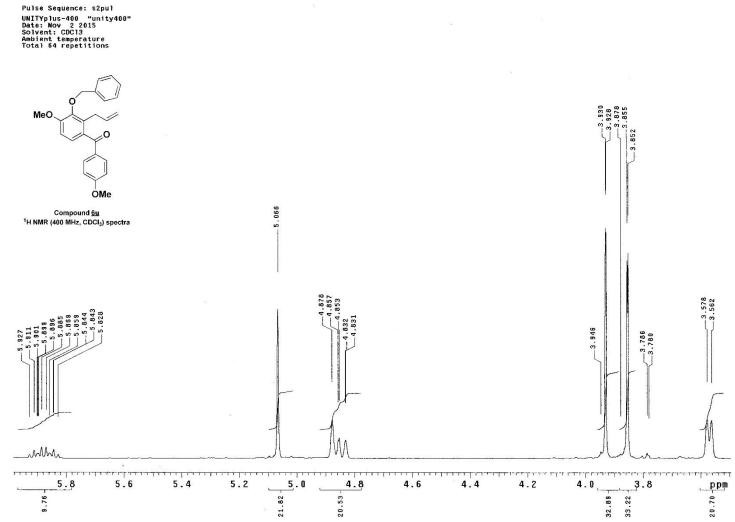


Compound <u>6t</u> ¹H NMR (400 MHz, CDCI₃) spectra



YLenon2t Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 9 2015 Solvent: CDC13 Ambient temperature Total 1632 repetitions MeO. -131.745 -130.002 -128.009 Compound 6t ¹³C NMR (100 MHz, CDCI₃) spectra 32.715 23.535 132.458 125.500 30.509 108.663 115.160 55.464 .379 -136.666 -134.232 84.543 -138. 145.527 154.426 197.468 to the state of th 11 180 160 140 100 80 60 40 20 120 ppm

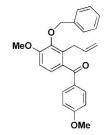
YLenon2u Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions -3.930 -928 MeO. OMe 5.066 Compound <u>6u</u> ¹H NMR (400 MHz, CDCI₃) spectra -578 -3.562 4.878 3 2 20.00 ل 13.91 ل 20.63 ل 10.63 ل 33.14 ل 9.76 f 9. 21.82 f. -20.53 f 33:22 1 9 8 3 ppm 1 20.70 -[



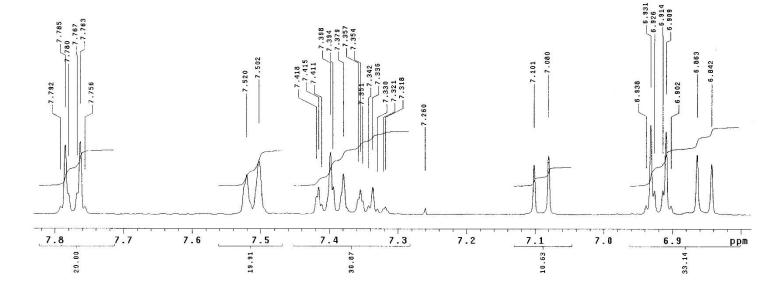
YLenon2u

YLenon2u

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

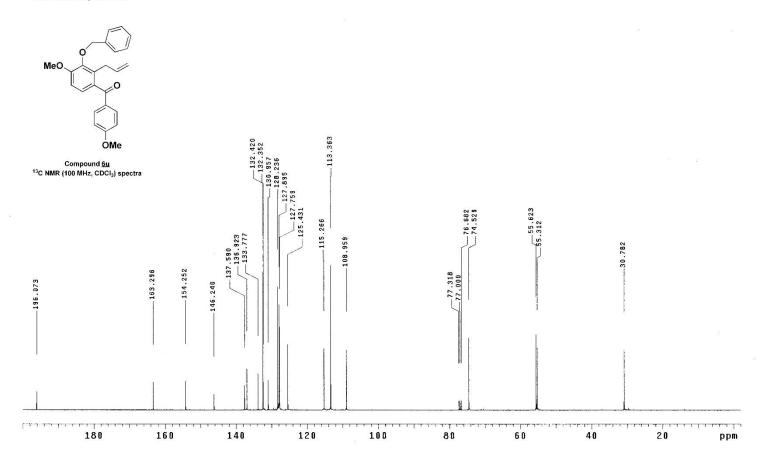


Compound <u>6u</u> ¹H NMR (400 MHz, CDCI₃) spectra



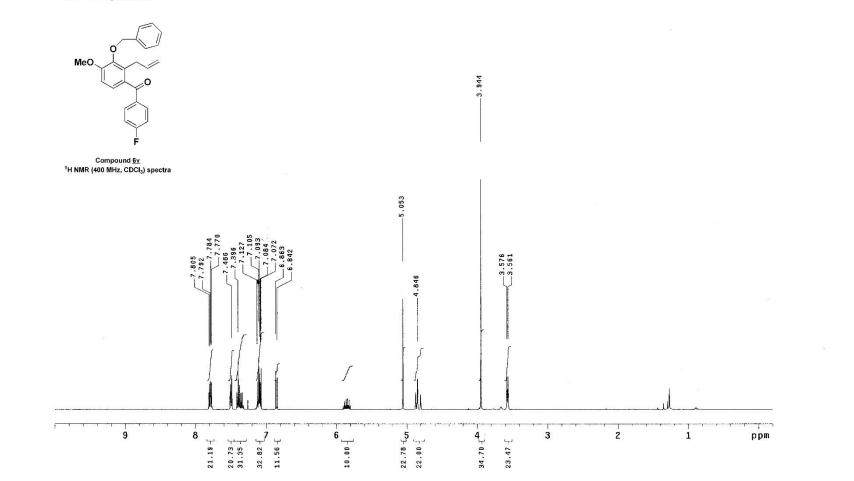
YL enon2u

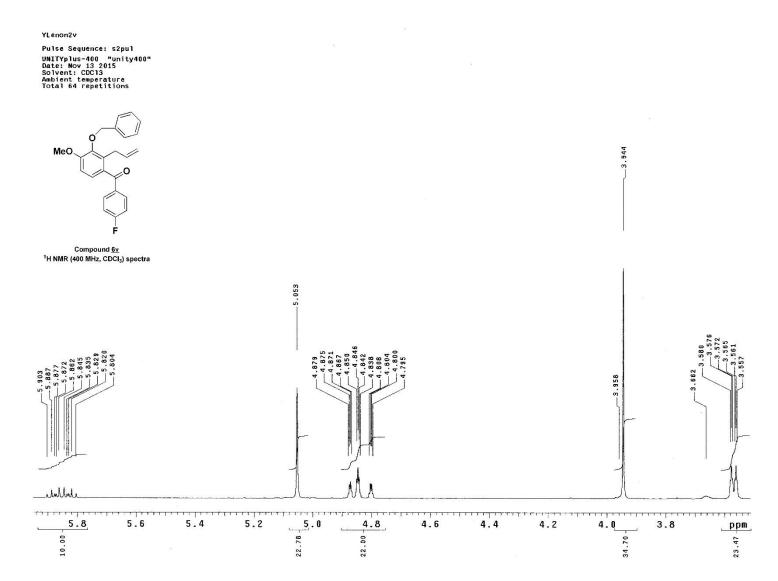
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDCl3 Ambient temperature Total 3200 repetitions



YLenon2v

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 13 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

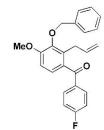




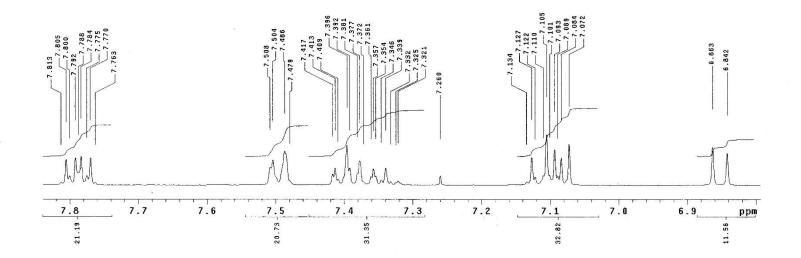
S-194

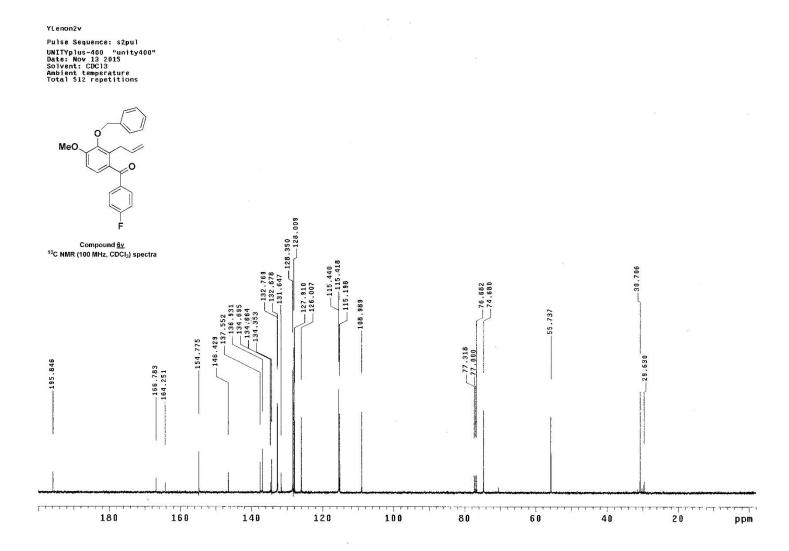
YLenon2v

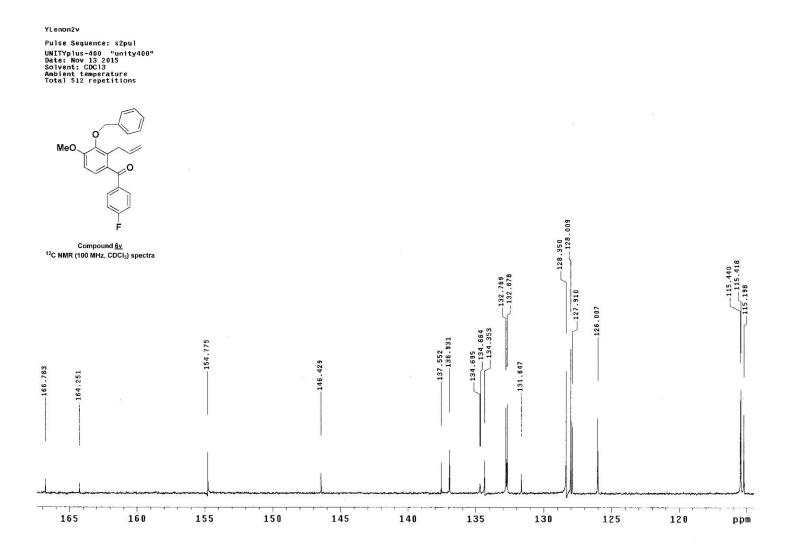
Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Nov 13 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



Compound <u>6v</u> ¹H NMR (400 MHz, CDCl₃) spectra

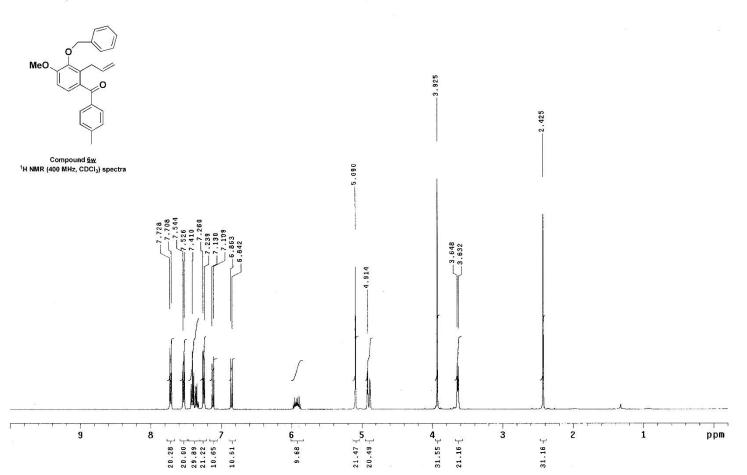






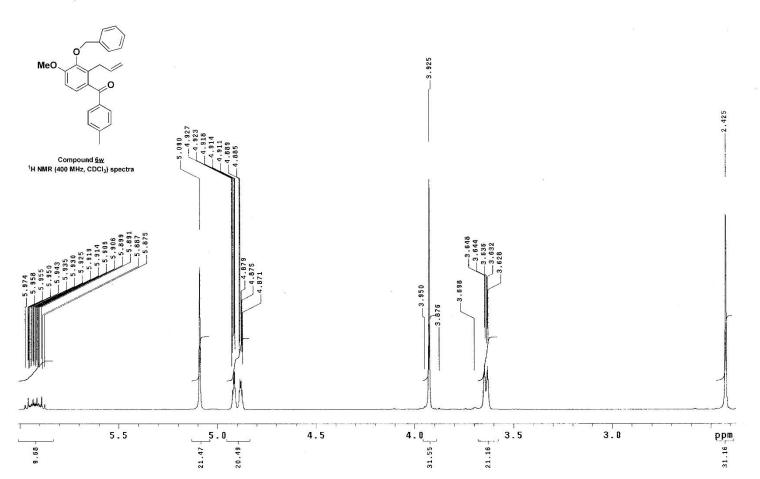
YLenon2w

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 10 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions



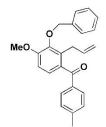
YL enon2w

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Nov 10 2015 Solvent: CDCl3 Ambient temperature Total 64 repetitions

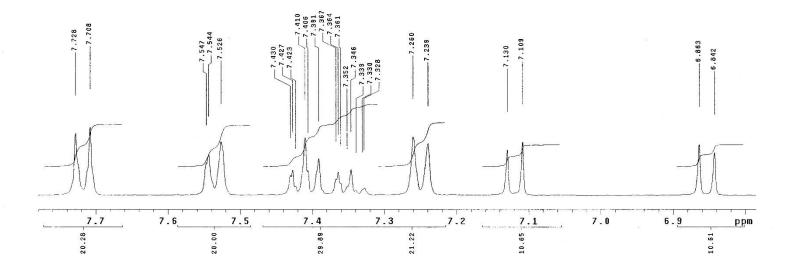


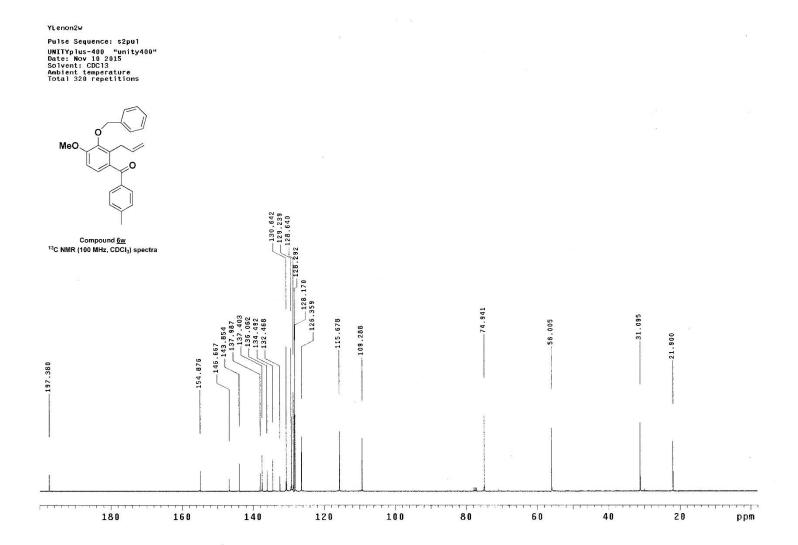
YLenon2w

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 10 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

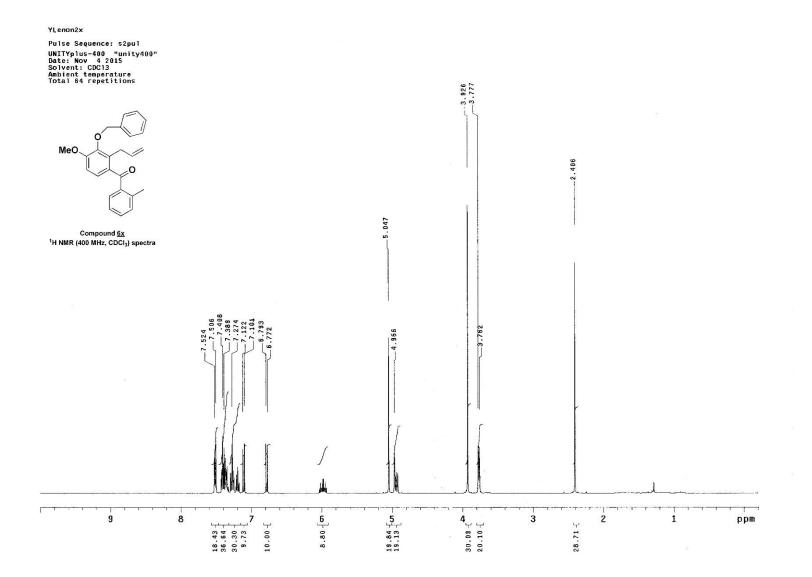


Compound <u>6w</u> ¹H NMR (400 MHz, CDCl₃) spectra

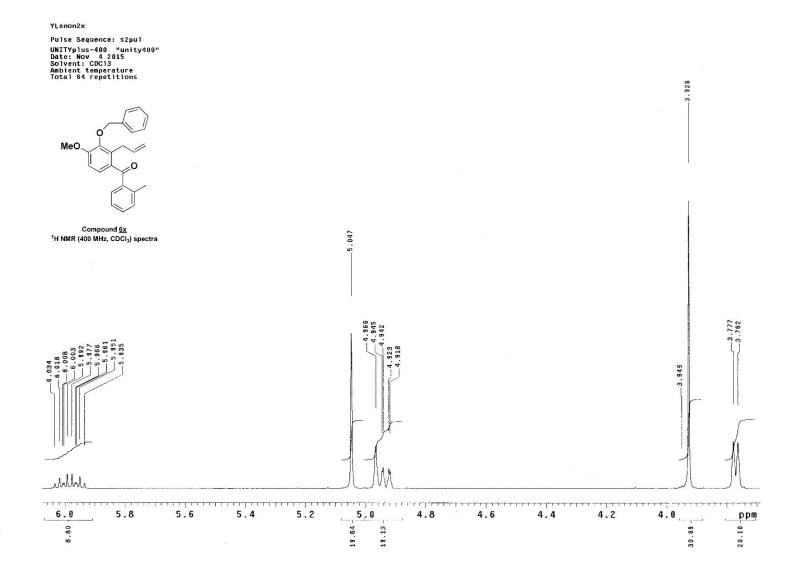




YL enon2w Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 10 2015 Solvent: CDC13 Ambient temperature Total 320 repetitions MeO. -130.642 Compound <u>6w</u> ¹³C NMR (100 MHz, CDCI₃) spectra 129.239 -128.640 -128.292 128.170 126.359 .403 134.492 143.854 136.062 154.876 137.987 137 468 146.667 132. 138 154 152 150 148 146 144 142 140 136 134 132 130 128 ppm



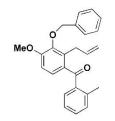
S-203



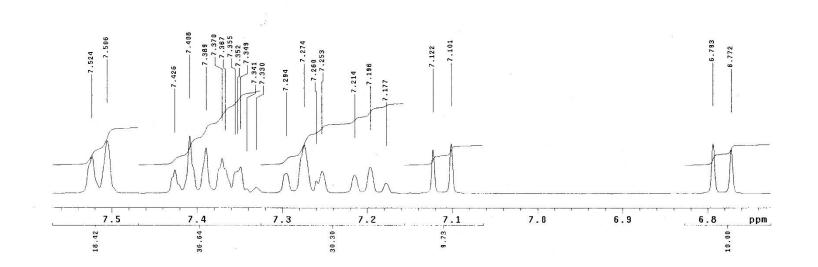
S-204

YLenon2x

Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Nov 4 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

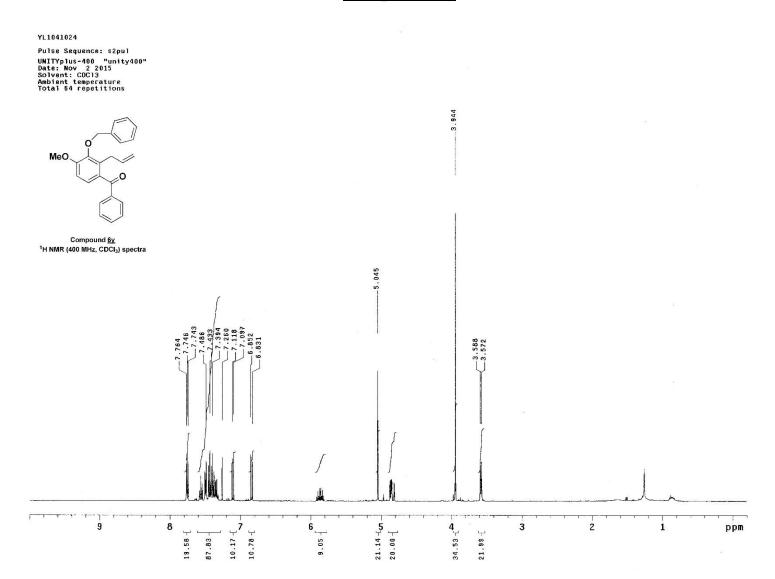


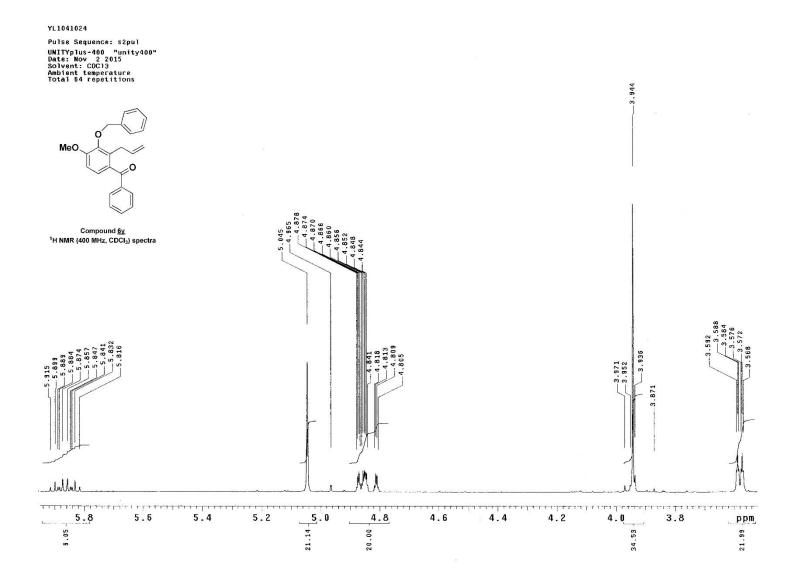
Compound <u>6x</u> ¹H NMR (400 MHz, CDCl₃) spectra



YLenon2x Pulse Sequence: s2pul UNITYPlus-400 "unity400" Date: Nov 4 2015 Solvent: CDC13 Ambient temperature Total 1728 repetitions MeO. Compound <u>6x</u> ¹³C NMR (100 MHz, CDCI₃) spectra 76.674 30.502 55.684 139.660 137.727 37.575 20.374 155.450 -77.311 199.340 146.452 111111111111111111 · - | - | - | - | - | - | - | - | r 1 1 1 1-1.1 180 100 20 200 160 140 120 80 60 40 ppm

YLenon2x Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 4 2015 Solvent: CDC13 Ambient temperature Total 1728 repetitions MeO. 127.948 Compound <u>6x</u> ¹³C NMR (100 MHz, CDCI₃) spectra 128.335 872 129.843 128.501 127 130.548 131.079 -137.257 139.660 .422 -137.727 137.575 208 132. 140 139 135 138 137 136 134 133 132 131 130 129 128 ppm

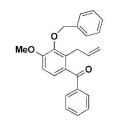




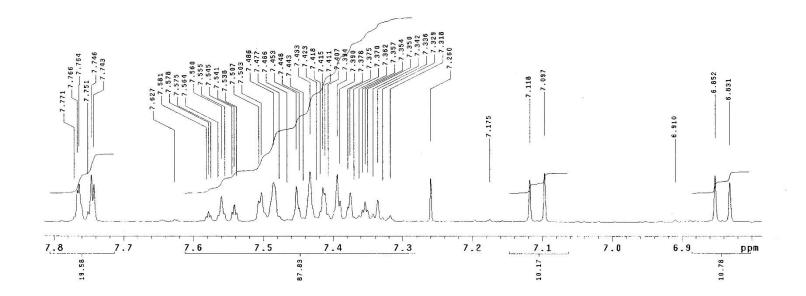
S-209

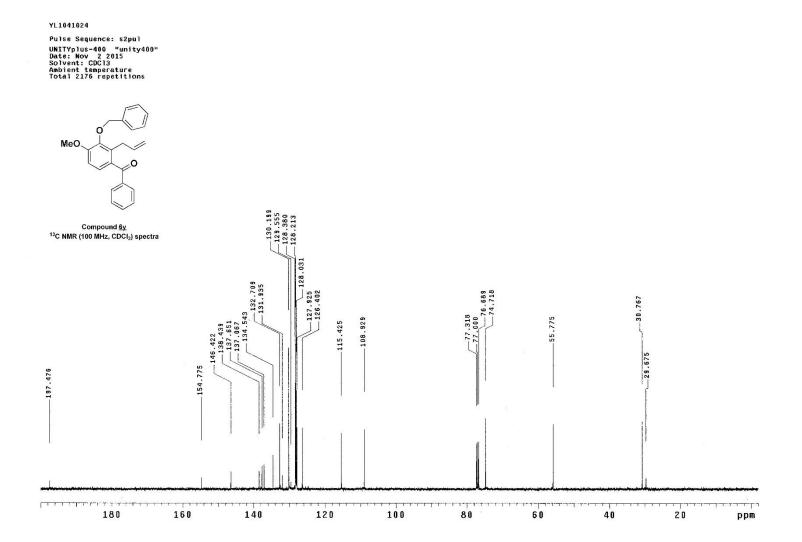
YL1041024

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Nov 2 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

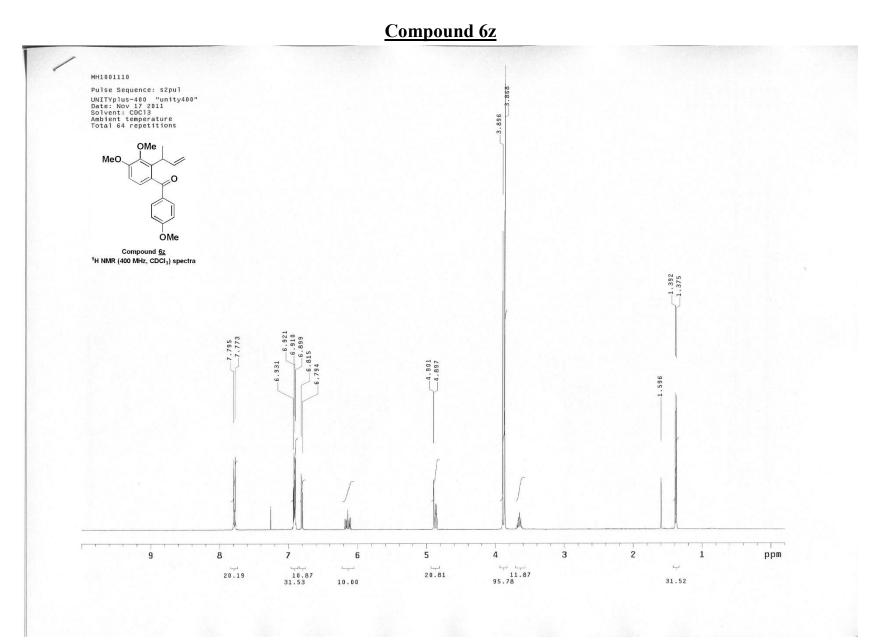


Compound <u>6v</u> ¹H NMR (400 MHz, CDCI₃) spectra

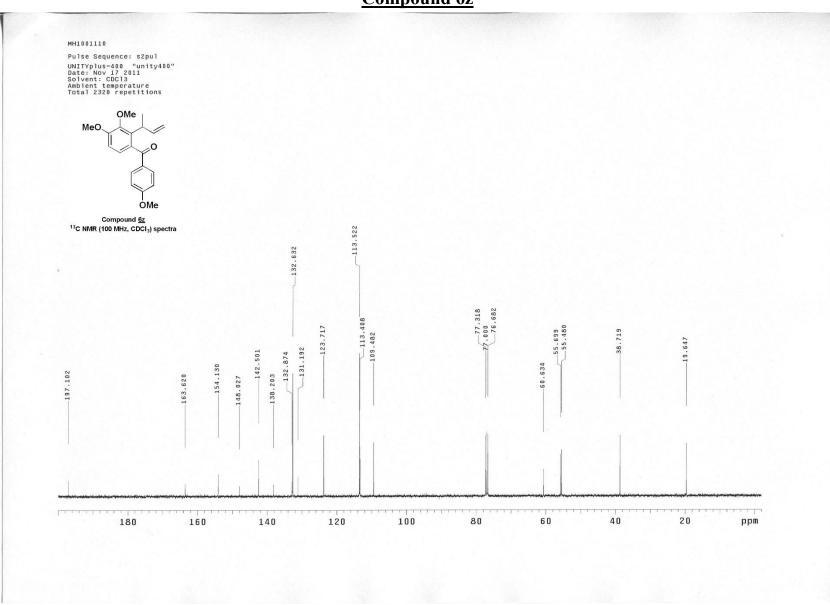




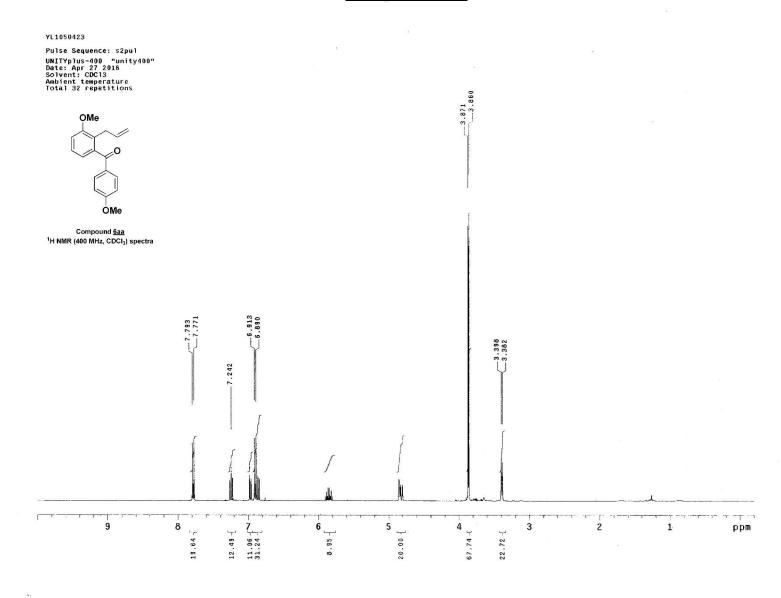
YL1041024 Pulse Sequence: s2pul UNITYPIUS-400 "unity400" Date: Nov 2 2015 Solvent: CDCl3 Ambient temperature Total 2176 repetitions MeO. Compound <u>6y</u> ¹³C NMR (100 MHz, CDCI₃) spectra _____128.213 -128.031 130.199 -128.380 127.925 132.709 126.402 134.543 137.067 137.651 138.439 131.935 129.555 march 1.1 138 135 137 136 134 133 132 131 130 129 128 127 ppm



S-213



Compound 6aa

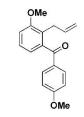


S-215

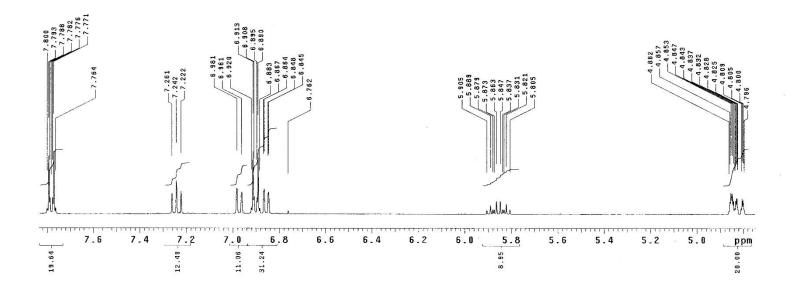
Compound 6aa

YL1050423

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Apr 27 2016 Solvent: CDC13 Ambient temperature Total 32 repetitions



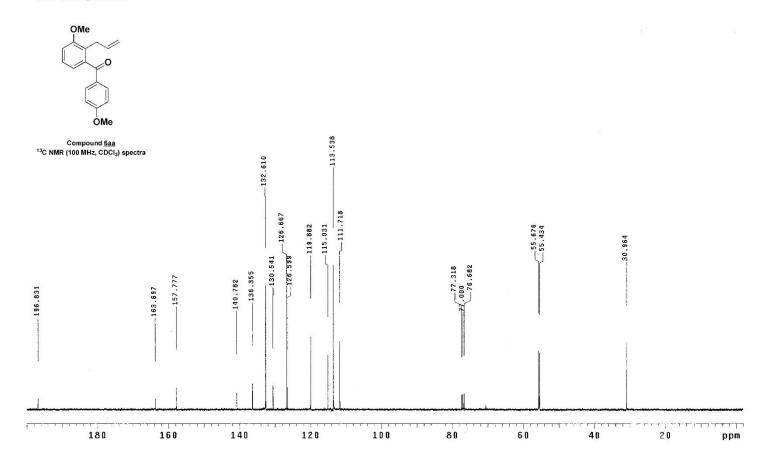
Compound <u>6aa</u> ¹H NMR (400 MHz, CDCI₃) spectra

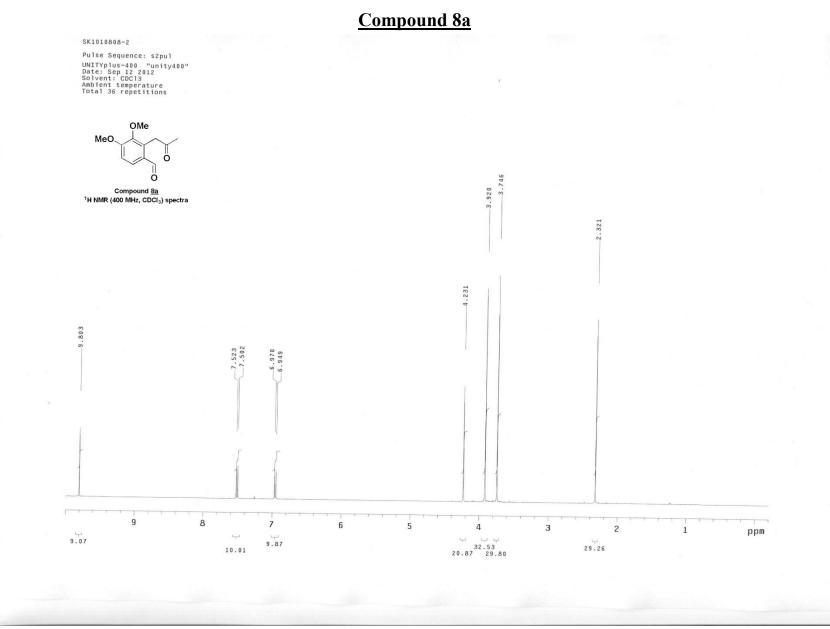


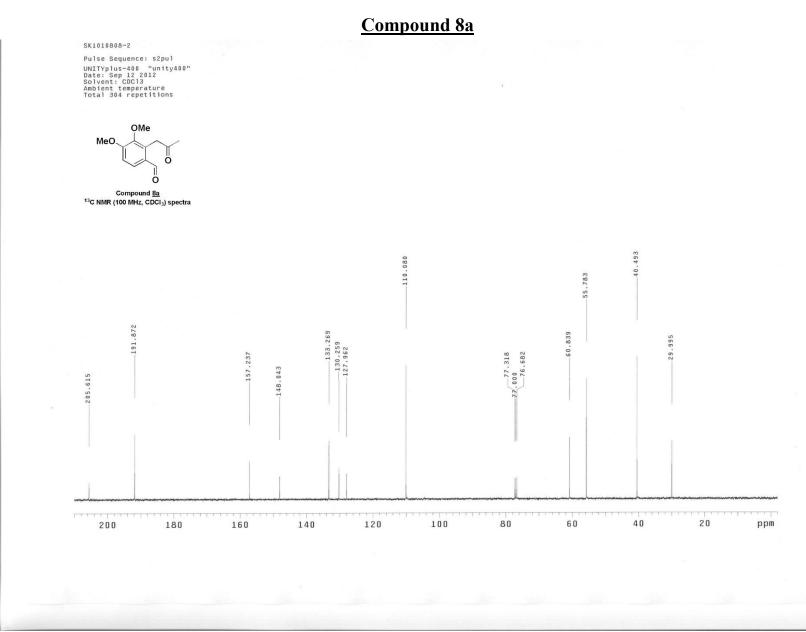
Compound 6aa

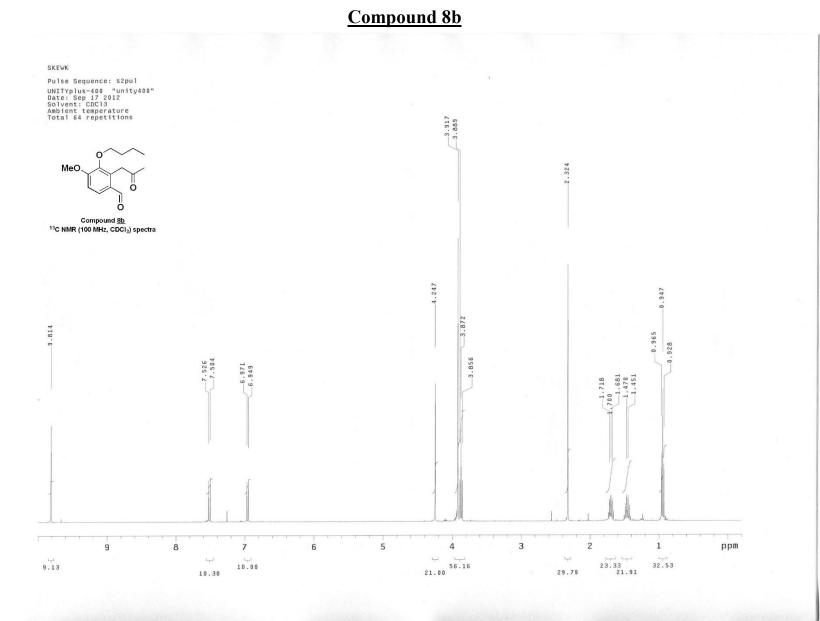
YL1050423

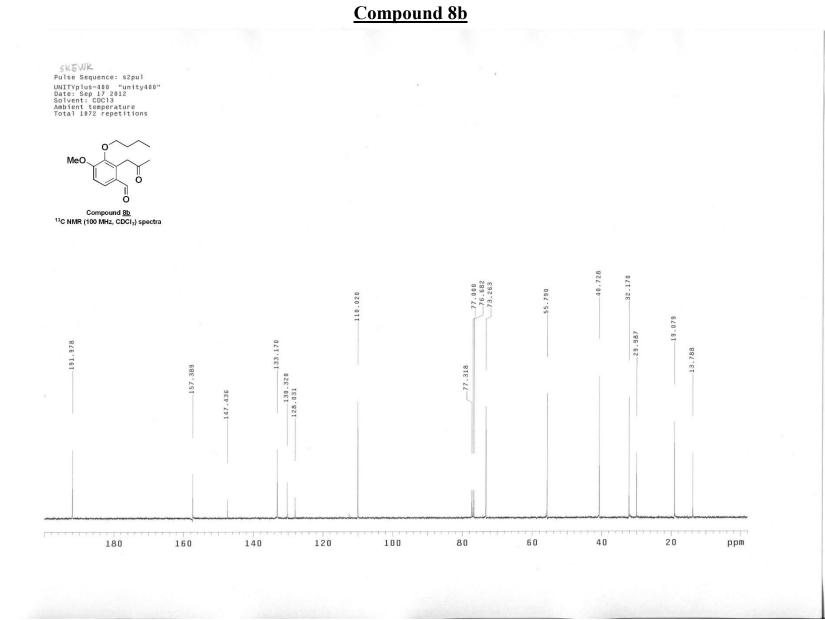
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Apr 27 2016 Solvent: CDC13 Ambient temperature Total 544 repetitions

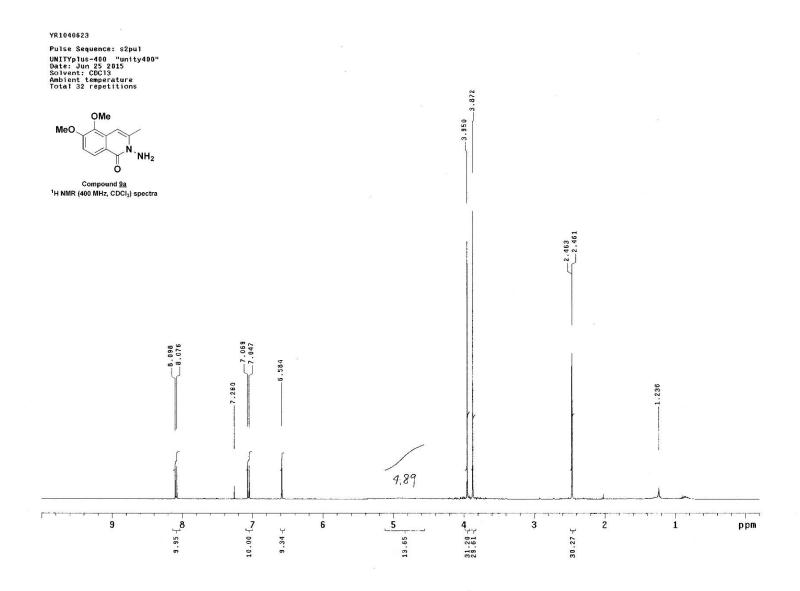






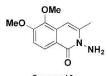




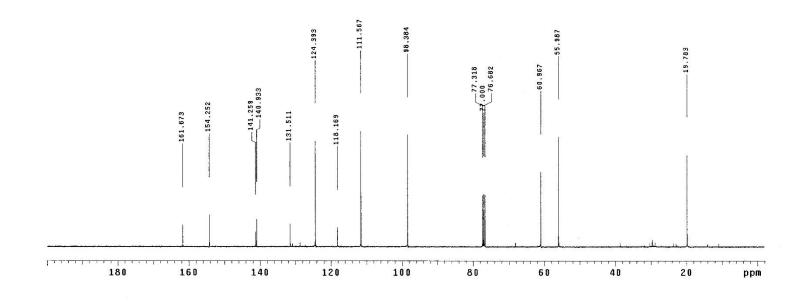


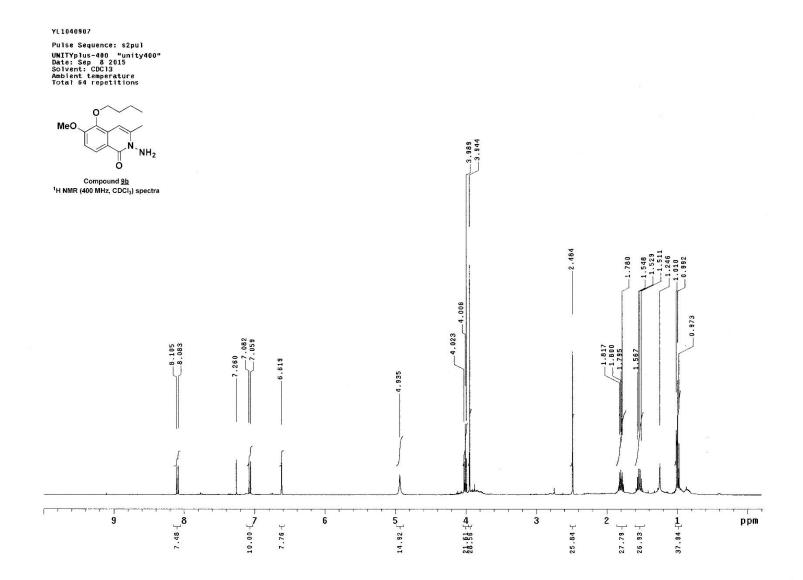
YR1040623

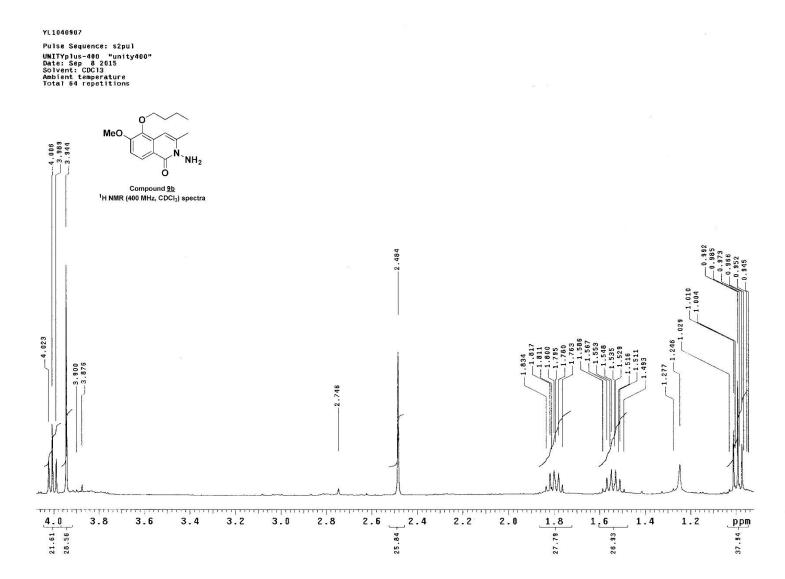
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Jun 25 2015 Solvent: CDCl3 Ambient temperature Total 6400 repetitions



Compound <u>9a</u> ¹³C NMR (100 MHz, CDCI₃) spectra





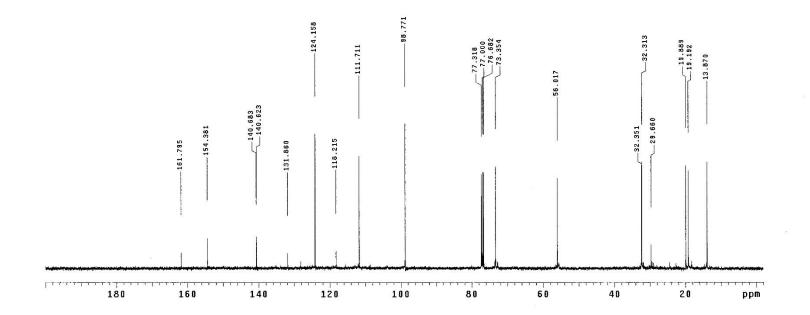


YL1040907

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 8 2015 Solvent: CDCl3 Ambient temperature Total 11728 repetitions

0 MeO NH2 Ö

Compound <u>9b</u> ¹³C NMR (100 MHz, CDCI₃) spectra

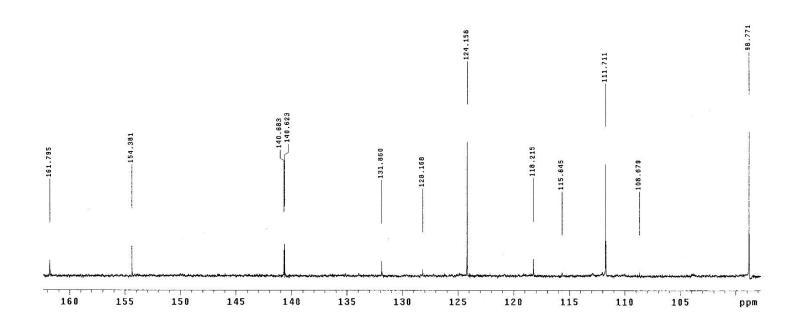


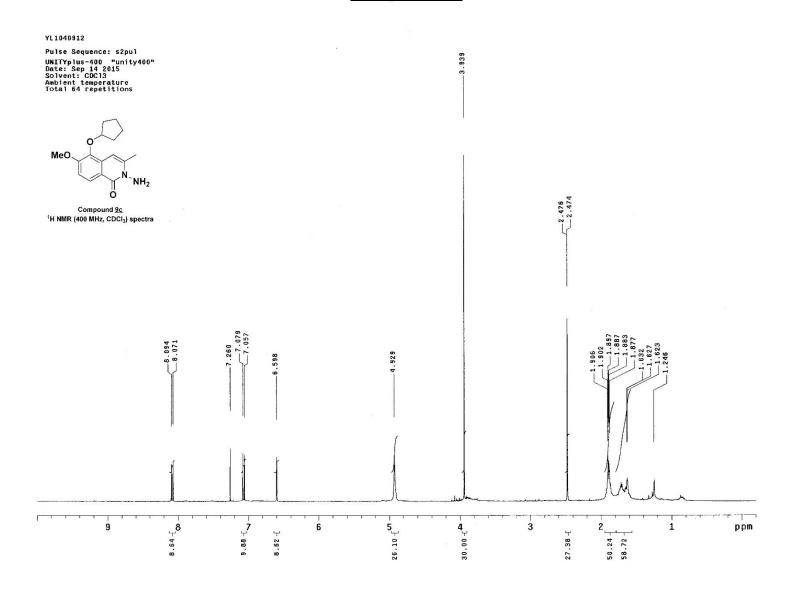
YL1040907

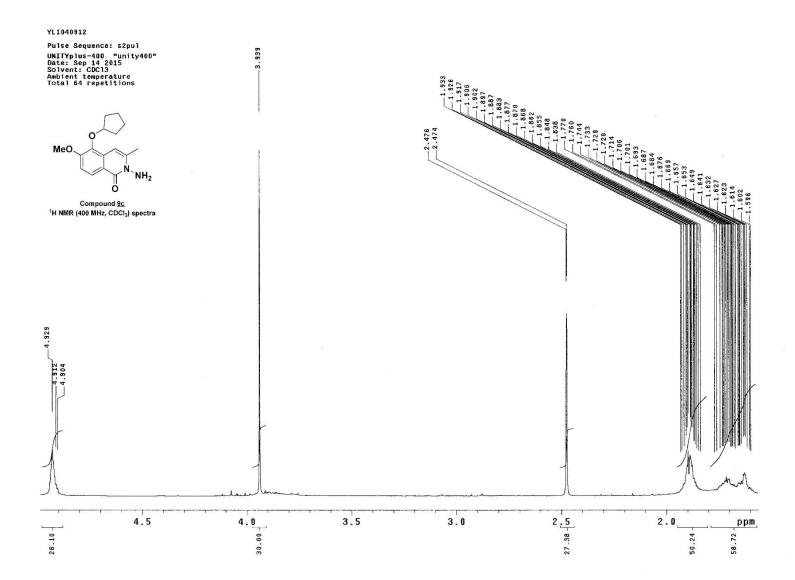
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Sep 8 2015 Solvent: CDCl3 Ambient temperature Total 11728 repetitions

0 MeO NH2 Ö

Compound <u>9b</u> ¹³C NMR (100 MHz, CDCI₃) spectra



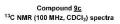


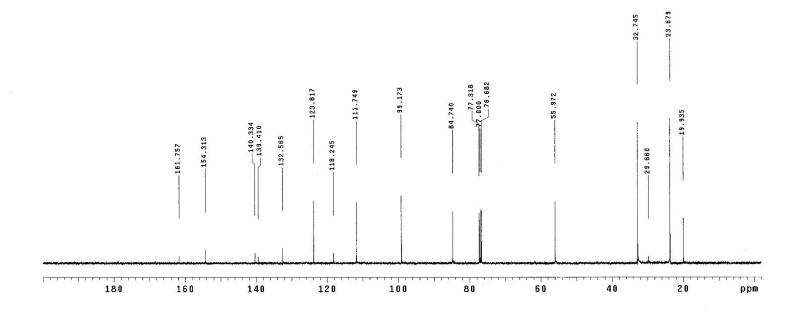


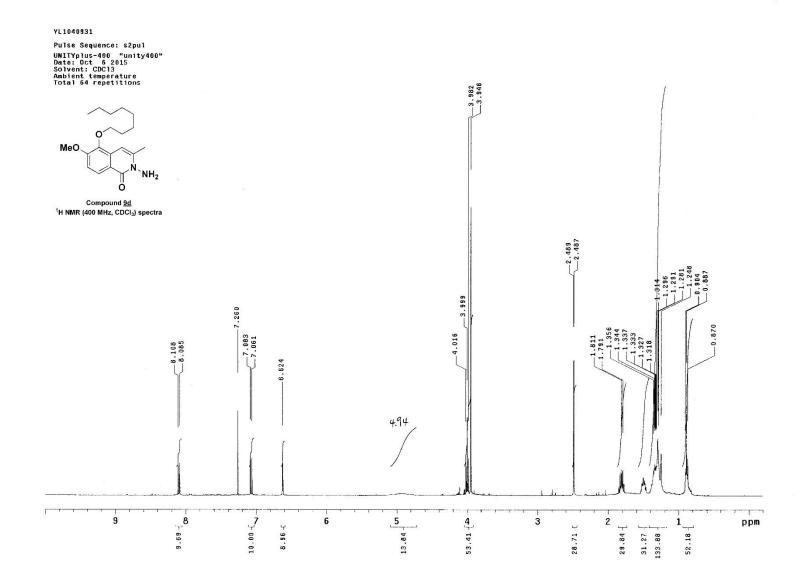
YL1040912

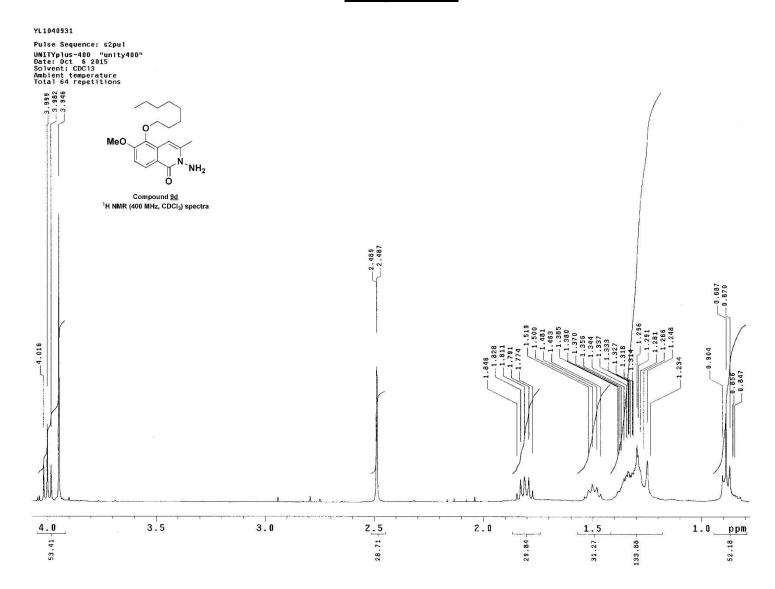
Pulse Sequence: s2pul UMITYplus-400 "unity400" Date: Sep 14 2015 Solvent: CDC13 Ambient temperature Total 4448 repetitions

MeO NH₂ 0







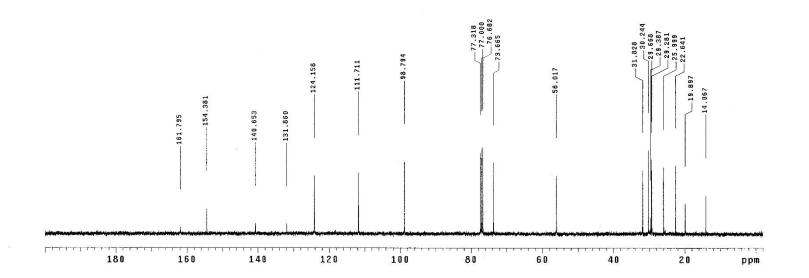


YL1040931

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Oct 6 2015 Solvent: CDC13 Ambient temperature Total 4272 repetitions

MeO NH_2 Ô

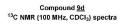


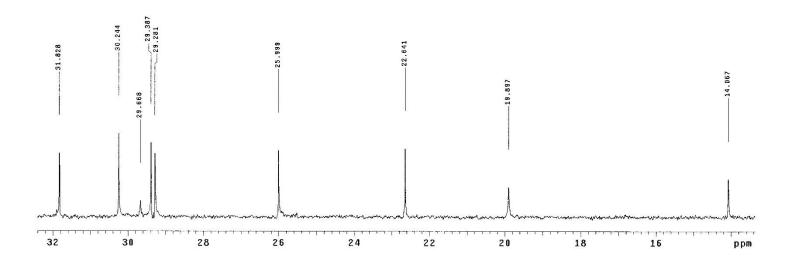


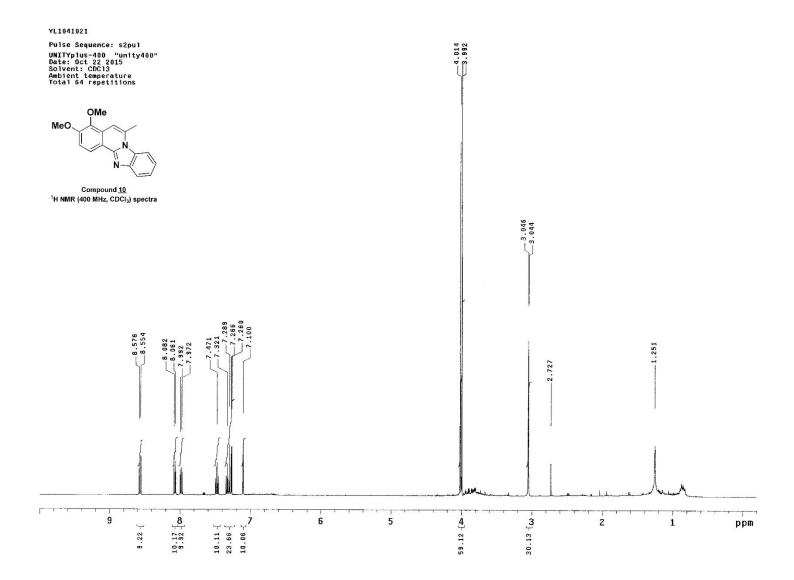
YL1040931

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Oct 6 2015 Solvent: CDCl3 Ambient temperature Total 4272 repetitions

MeO NH_2 Ô

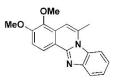




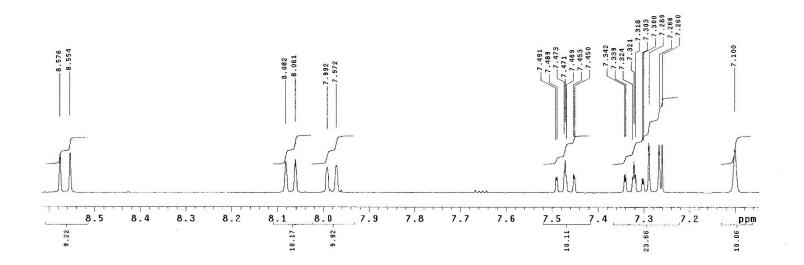


YL1041021

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Oct 22 2015 Solvent: CDC13 Ambient temperature Total 64 repetitions

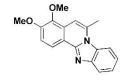


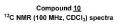
Compound <u>10</u> ¹H NMR (400 MHz, CDCl₃) spectra

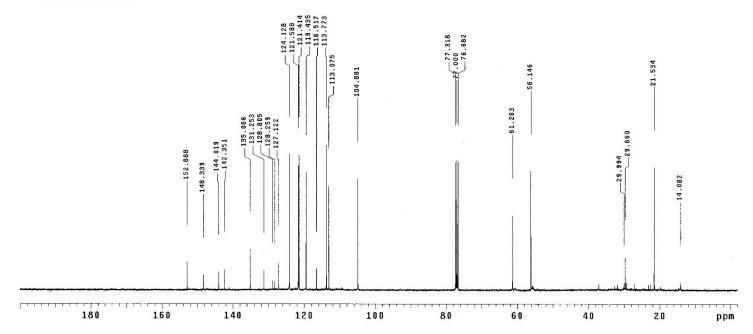


YL1041021

Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Oct 22 2015 Solvent: CDC13 Ambient temperature Total 12800 repetitions

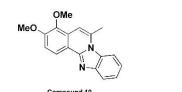




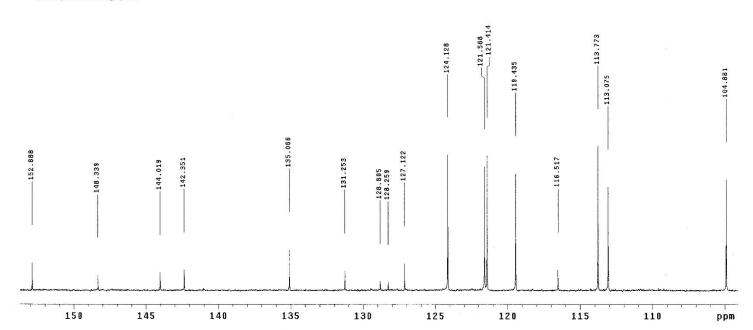


YL1041021

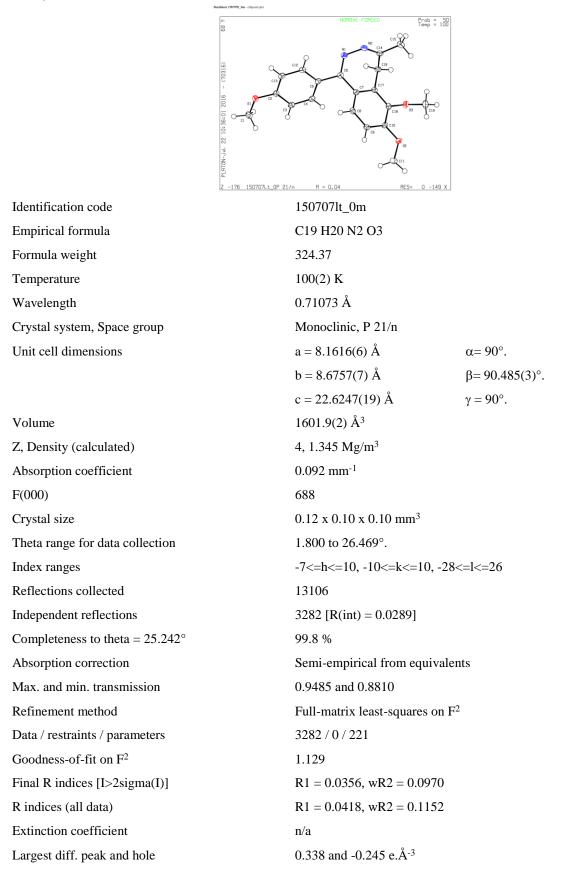
Pulse Sequence: s2pul UNITYplus-400 "unity400" Date: Oct 22 2015 Solvent: CDC13 Ambient temperature Total 12800 repetitions



Compound <u>10</u> ¹³C NMR (100 MHz, CDCI₃) spectra



Crystal data and structure refinement for **1a** (the thermal ellipsoid was drawn at the 50% probability level)



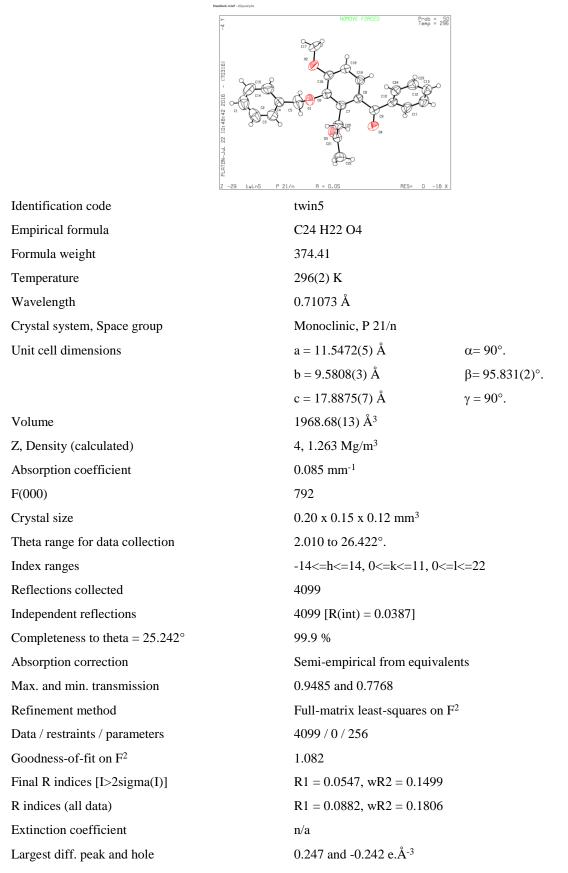
Crystal data and structure refinement for **1c** (the thermal ellipsoid was drawn at the 50% probability level)

level)	Datablack 150917_0m - ellipooid plot			
	λ ES (91002) - 910		Prob = 50 Temp = 296	
	9102 E1 46E 401 22 -98 150917 .0m P 21	/c R = 0.05 RES	²² ⊕ ^{c1} 5≈ 0 176 X	
Identification code		150917_0m		
Empirical formula		C19 H20 N2 O2		
Formula weight		308.37		
Temperature		296(2) K		
Wavelength		0.71073 Å		
Crystal system, Space group		Monoclinic, P 21/c		
Unit cell dimensions		a = 7.1877(5) Å		$\alpha = 90^{\circ}$.
		b = 25.1688(16) Å		$\beta = 110.284(4)^{\circ}.$
		c = 9.8090(6) Å		$\gamma = 90^{\circ}.$
Volume		1664.46(19) Å ³		
Z, Density (calculated)		4, 1.231 Mg/m ³		
Absorption coefficient		0.081 mm ⁻¹		
F(000)		656		
Crystal size		0.20 x 0.15 x 0.15 m	n ³	
Theta range for data collection		1.618 to 26.408°.		
Index ranges		-8<=h<=6, -31<=k<=	=30, -12<=	=l<=12
Reflections collected		13538		
Independent reflections		3399 [R(int) = 0.0362	2]	
Completeness to theta = 25.242°		99.9 %		
Absorption correction		Semi-empirical from equivalents		
Max. and min. transmission		0.9485 and 0.8499		
Refinement method		Full-matrix least-squares on F ²		
Data / restraints / parameters		3399 / 0 / 212		
Goodness-of-fit on F ²		1.030		
Final R indices [I>2sigma(I)]		R1 = 0.0481, wR2 =	0.1186	
R indices (all data)		R1 = 0.0649, wR2 =	0.1299	
Extinction coefficient		n/a		
Largest diff. peak and hole		0.166 and -0.167 e.Å	-3	

Crystal data and structure refinement for **1p** (the thermal ellipsoid was drawn at the 50% probability level)

level)	Daubleck me. (1999), file - ellipsed plot			
	≻ NOMOVE FORCED Prob = 5 ຫ	D		
	Z 39 mo_150908_P -1 R = 0.05 RES= 0 -14	¢		
Identification code	mo_150908_0m			
Empirical formula	C23 H26 N2 O3			
Formula weight	378.46			
Temperature	296(2) K			
Wavelength	0.71073 Å			
Crystal system, Space group	Triclinic, P -1			
Unit cell dimensions	a = 8.4384(6) Å	$\alpha = 70.841(2)^{\circ}.$		
	b = 11.0458(8) Å	$\beta = 77.317(2)^{\circ}.$		
	c = 12.1243(8) Å	$\gamma = 86.918(2)^{\circ}.$		
Volume	1041.25(13) Å ³			
Z, Density (calculated)	2, 1.207 Mg/m ³			
Absorption coefficient	0.080 mm^{-1}			
F(000)	404			
Crystal size	0.25 x 0.20 x 0.20 mm ³			
Theta range for data collection	1.820 to 26.514°.			
Index ranges	-10<=h<=10, -13<=k<=13,	-10<=h<=10, -13<=k<=13, -11<=l<=15		
Reflections collected	17072			
Independent reflections	4298 [R(int) = 0.0330]	4298 [R(int) = 0.0330]		
Completeness to theta = 25.242°	99.8 %	99.8 %		
Absorption correction	Semi-empirical from equiva	Semi-empirical from equivalents		
Max. and min. transmission	0.9485 and 0.8948	0.9485 and 0.8948		
Refinement method	Full-matrix least-squares on	Full-matrix least-squares on F ²		
Data / restraints / parameters	4298 / 180 / 302			
Goodness-of-fit on F ²	1.040			
Final R indices [I>2sigma(I)]	R1 = 0.0473, wR2 = 0.1277			
R indices (all data)	R1 = 0.0710, wR2 = 0.1461			
Extinction coefficient	n/a			
Largest diff. peak and hole	0.253 and -0.175 e.Å ⁻³			

Crystal data and structure refinement for 7y (the thermal ellipsoid was drawn at the 50% probability level)



Crystal data and structure refinement for **9a** (the thermal ellipsoid was drawn at the 50% probability level)

