

Sequential Rhodium-, Silver- and Gold-Catalyzed Synthesis of Fused Dihydrofurans

Supporting information

Hengbin Wang¹, Justin R. Denton² and Huw M. L. Davies^{1*}

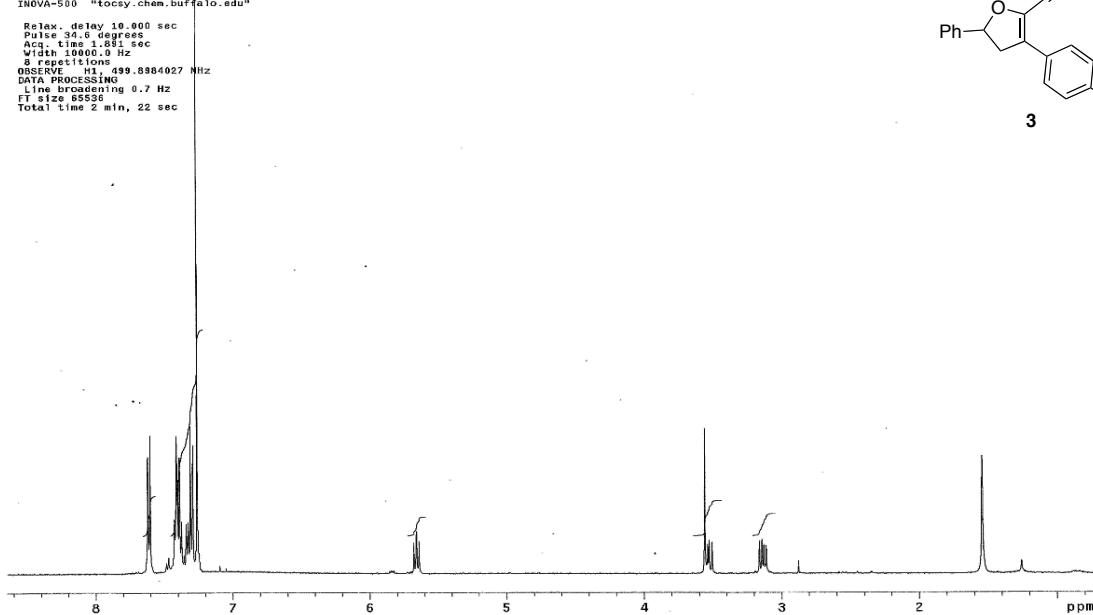
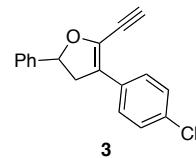
¹Department of Chemistry, Emory University, 1515 Dickey Drive, Atlanta, GA 30322, USA and ²Department of Chemistry, University at Buffalo, The State University of New York, Buffalo, NY 14260-3000 USA.

hmdavie@emory.edu

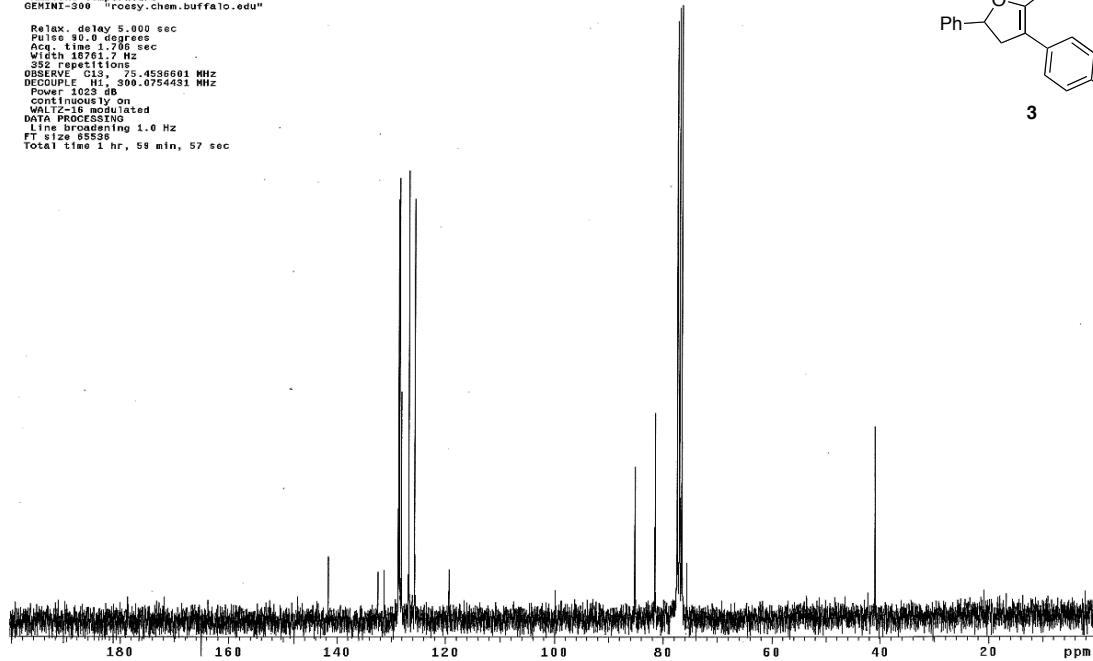
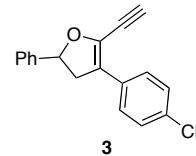
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Experimental Procedures	SI-2-43
Spectral data	SI-44-93

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 Solvent: CDCl₃
 Ambient temperature
 INOVA-500 "tocsy.chem.buffalo.edu"
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 Pulse 30.0 degrees
 Acq. time 1.681 sec
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 8 repetitions
 OBSERVE: H1 499.8884027 MHz
 DATA PROCESSING:
 Line broadening 0.7 Hz
 FT size 65536
 Total time 2 min, 22 sec

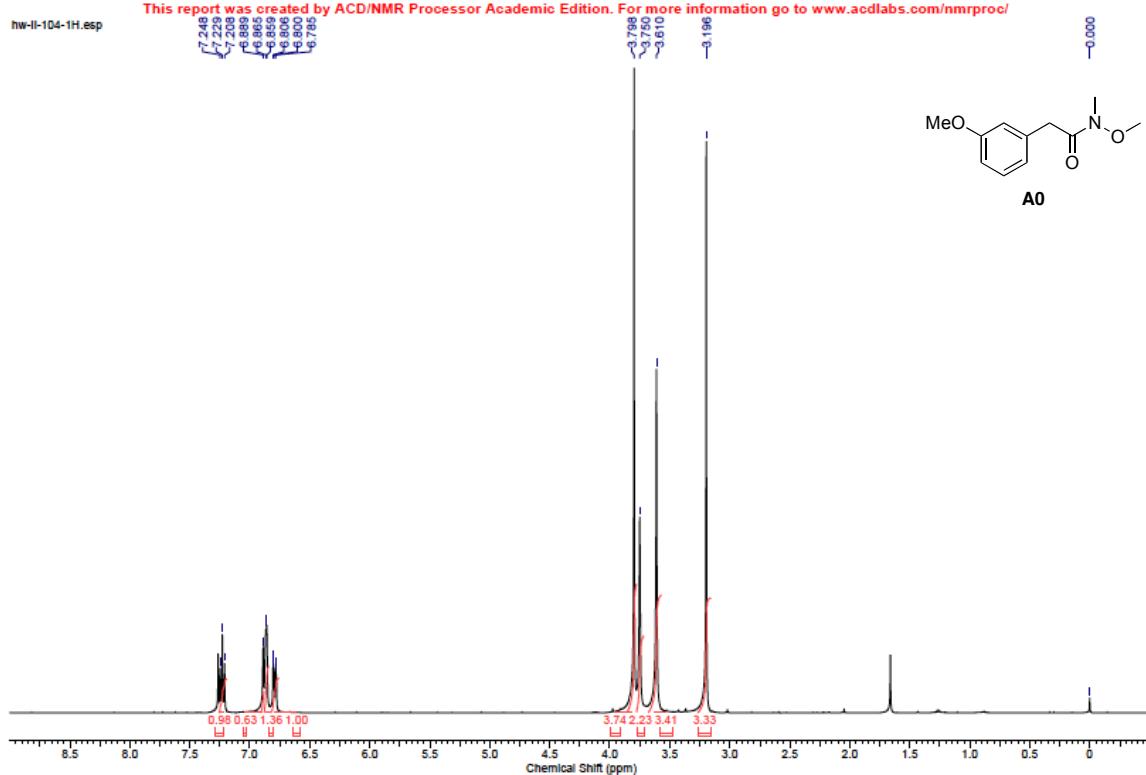


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 DECOUPLE: H1 300.0754431 MHz
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 continuously on
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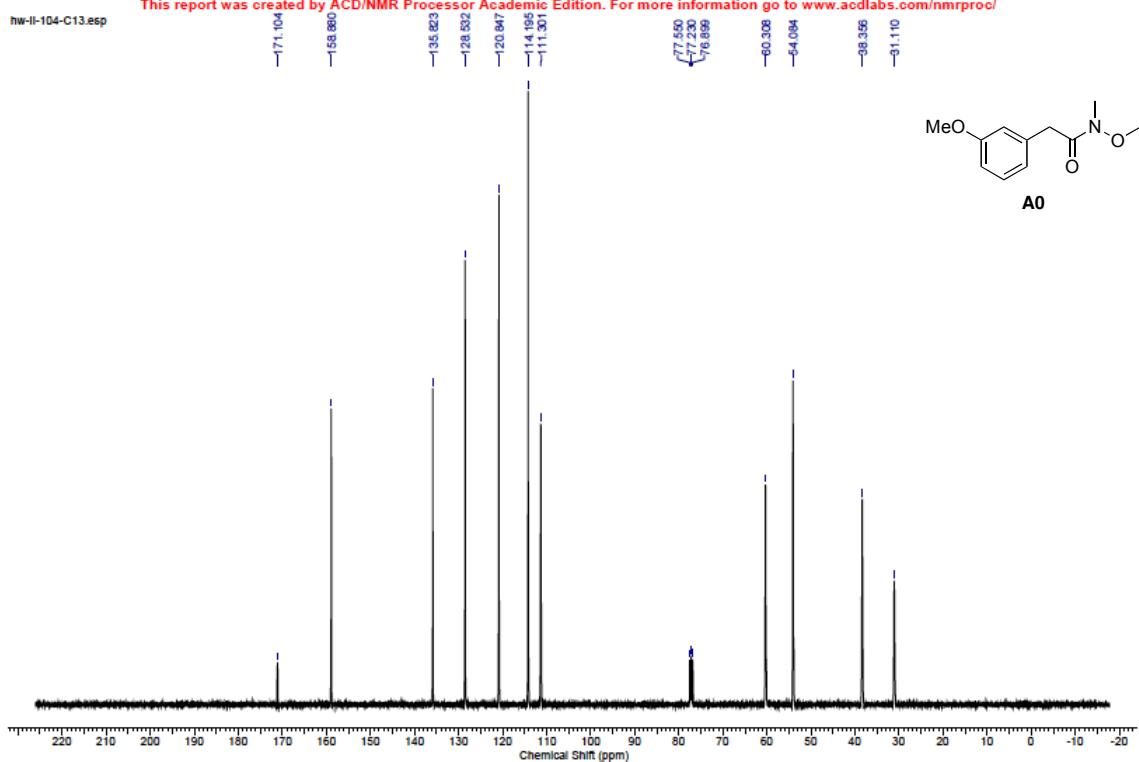


¹H and ¹³C spectra of compound 3

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/
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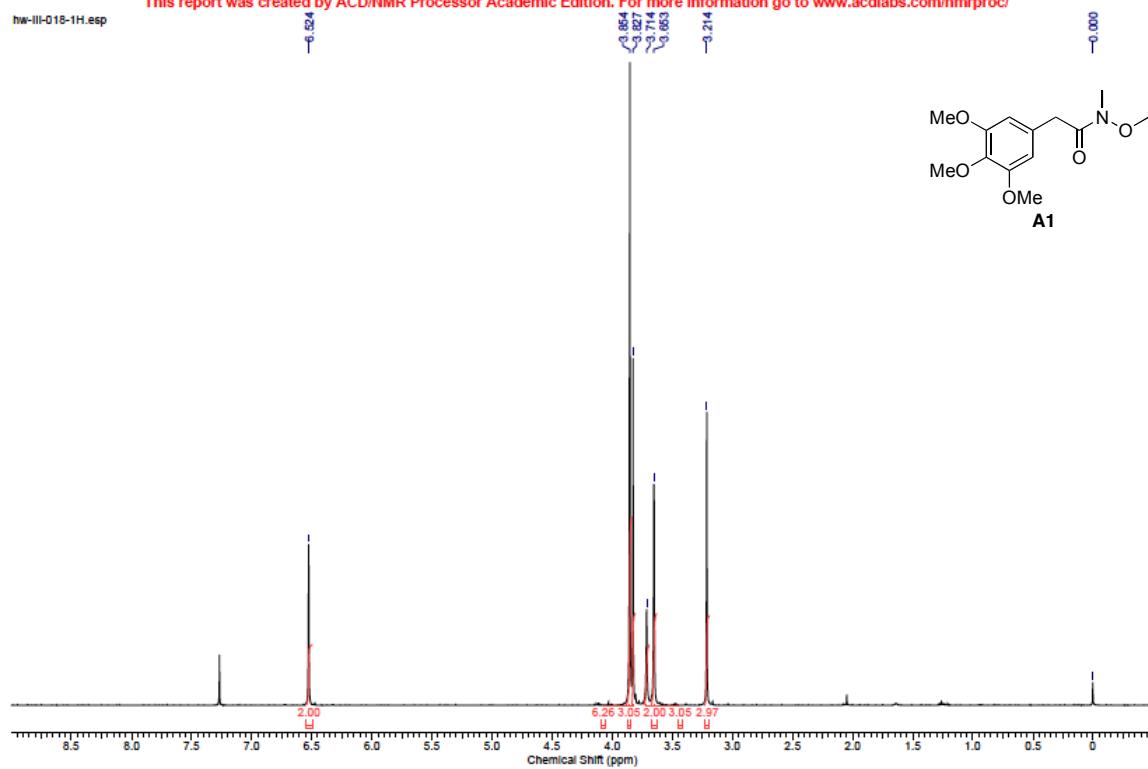


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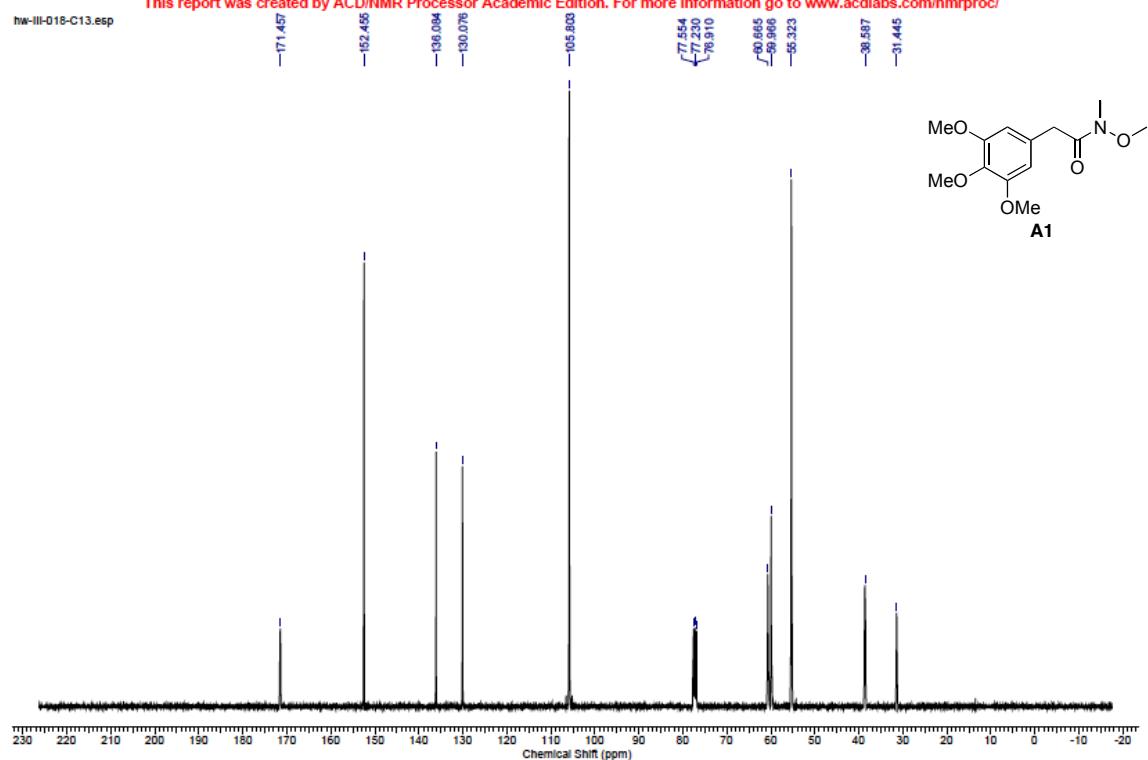


¹H and ¹³C spectra of compound A0

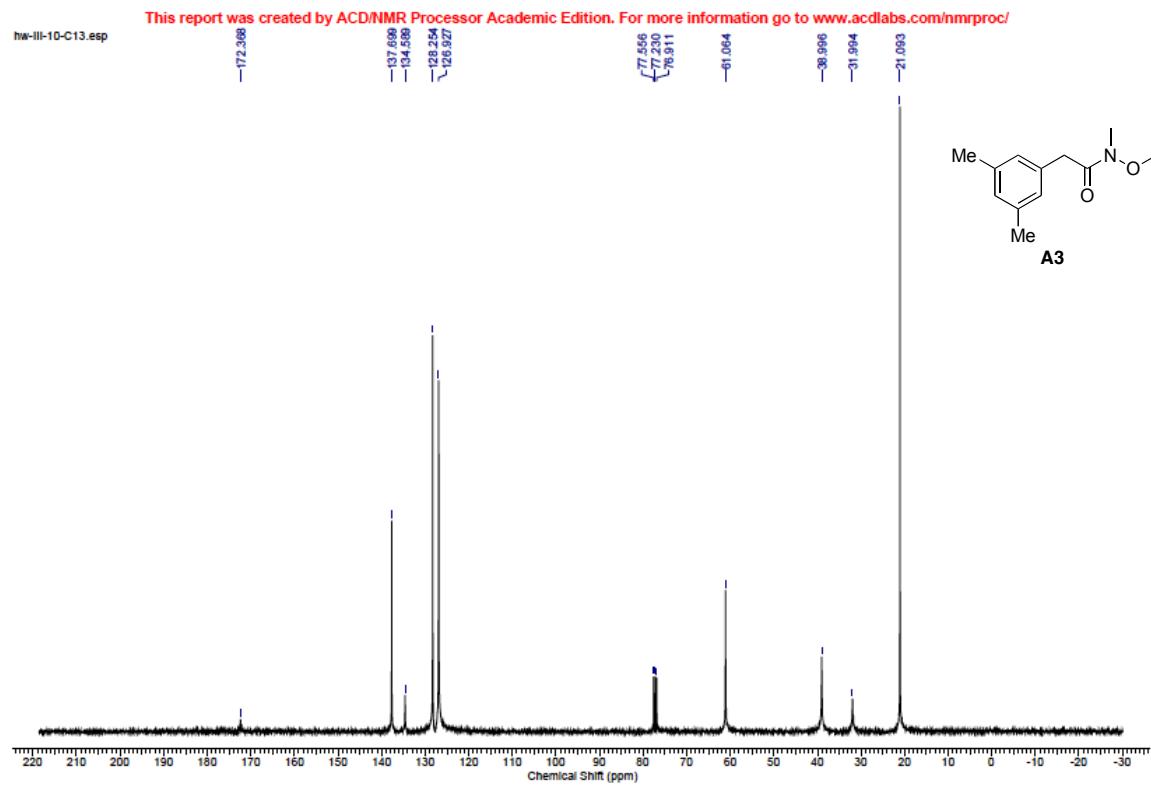
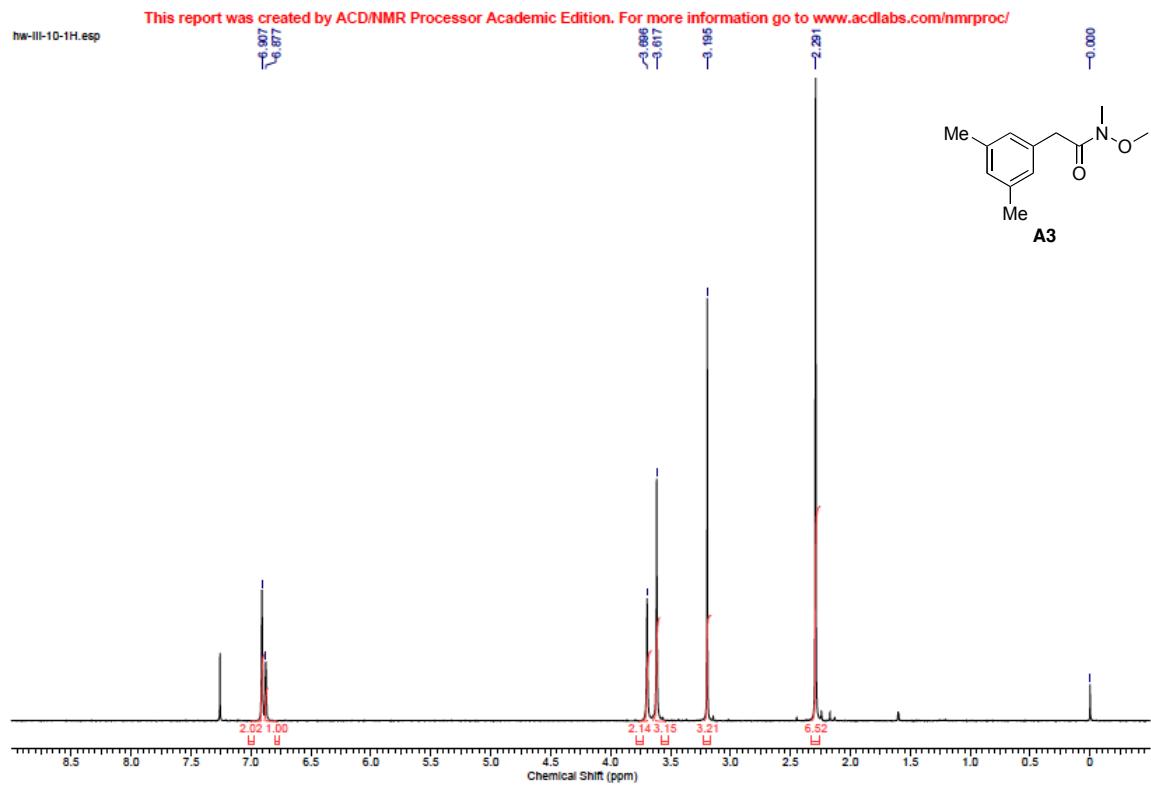
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This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/
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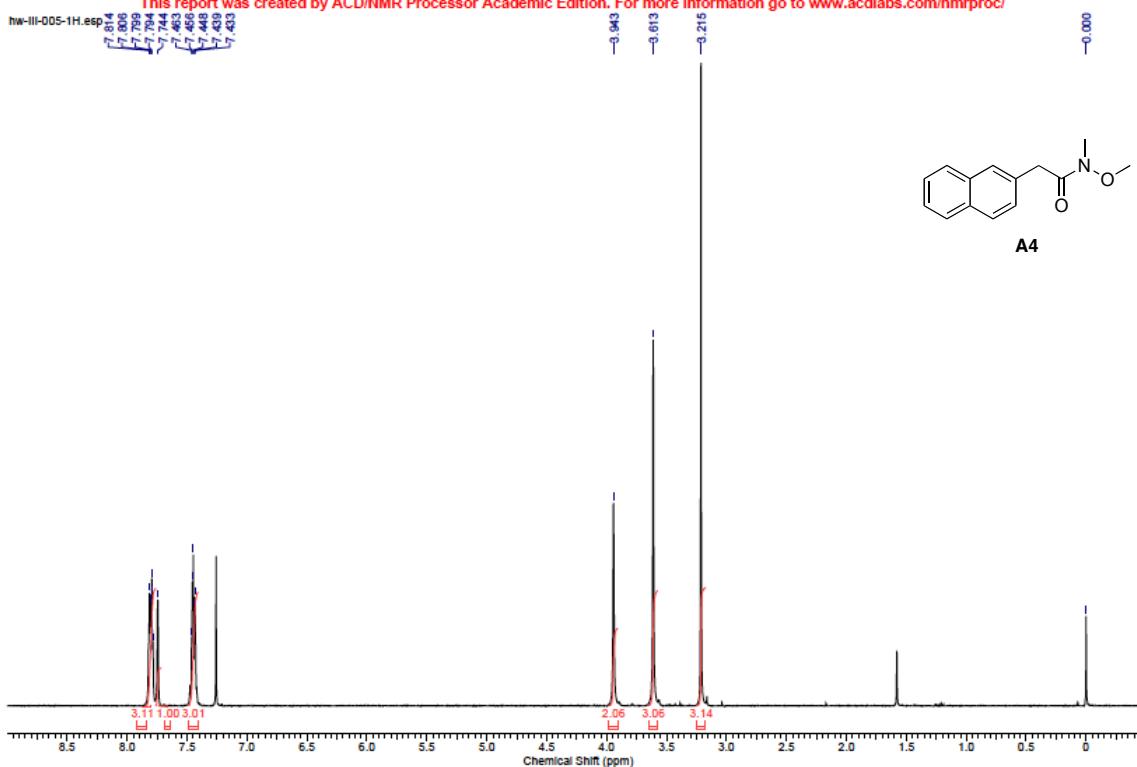


¹H and ¹³C spectra of compound A1



¹H and ¹³C spectra of compound A3

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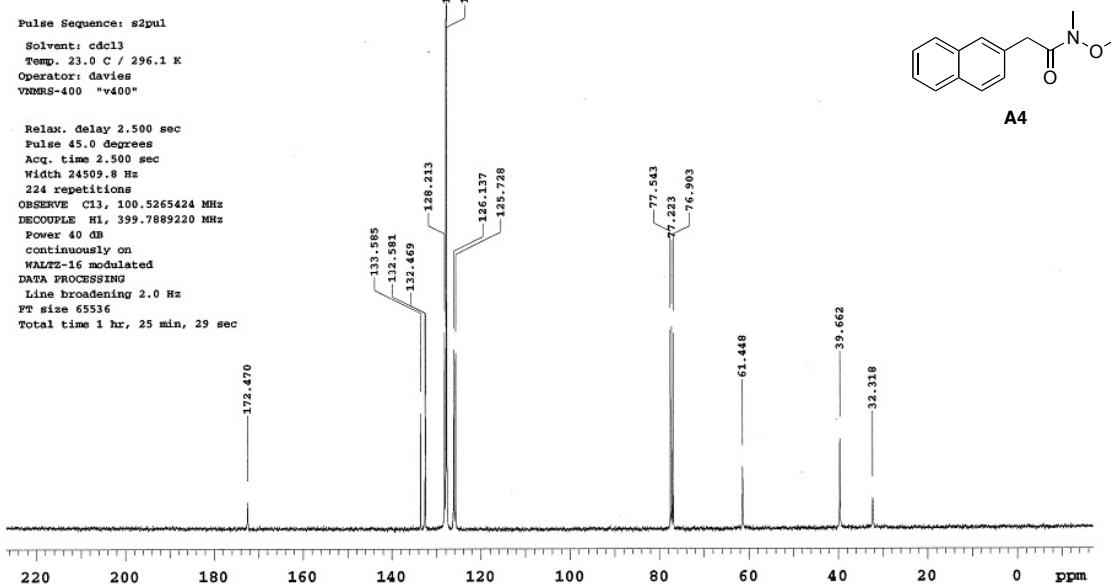
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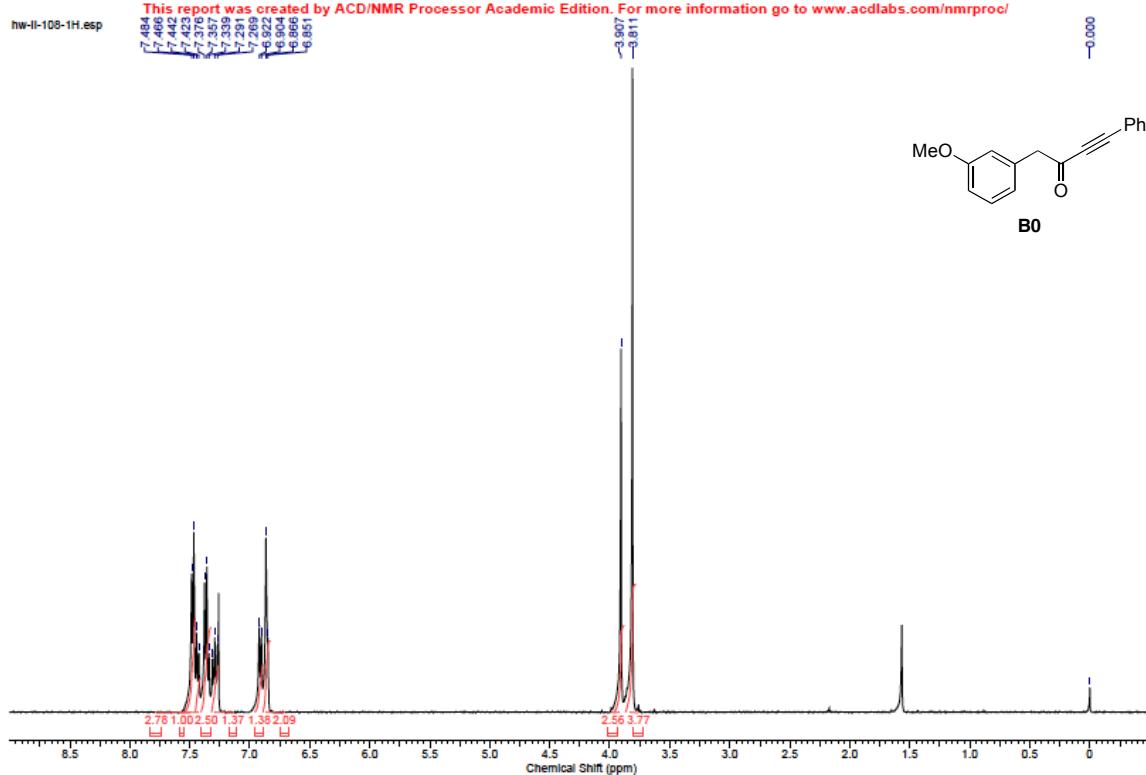
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Temp: 23.0 C / 296.1 K
Operator: davies
VNMRs-400 "v400"

Relax. delay 2.500 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 24509.8 Hz
224 repetitions
OBSERVE C13, 100.5265424 MHz
DECOUPLE H1, 399.7889220 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 25 min, 29 sec

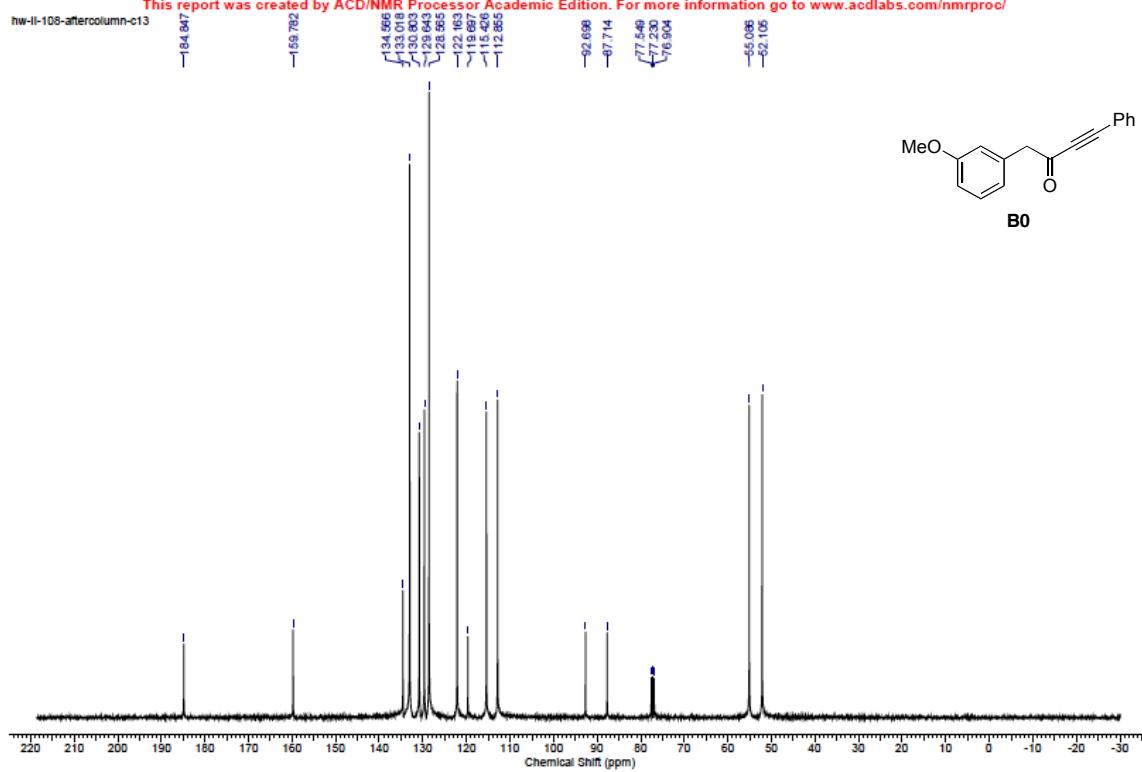


¹H and ¹³C spectra of compound A4

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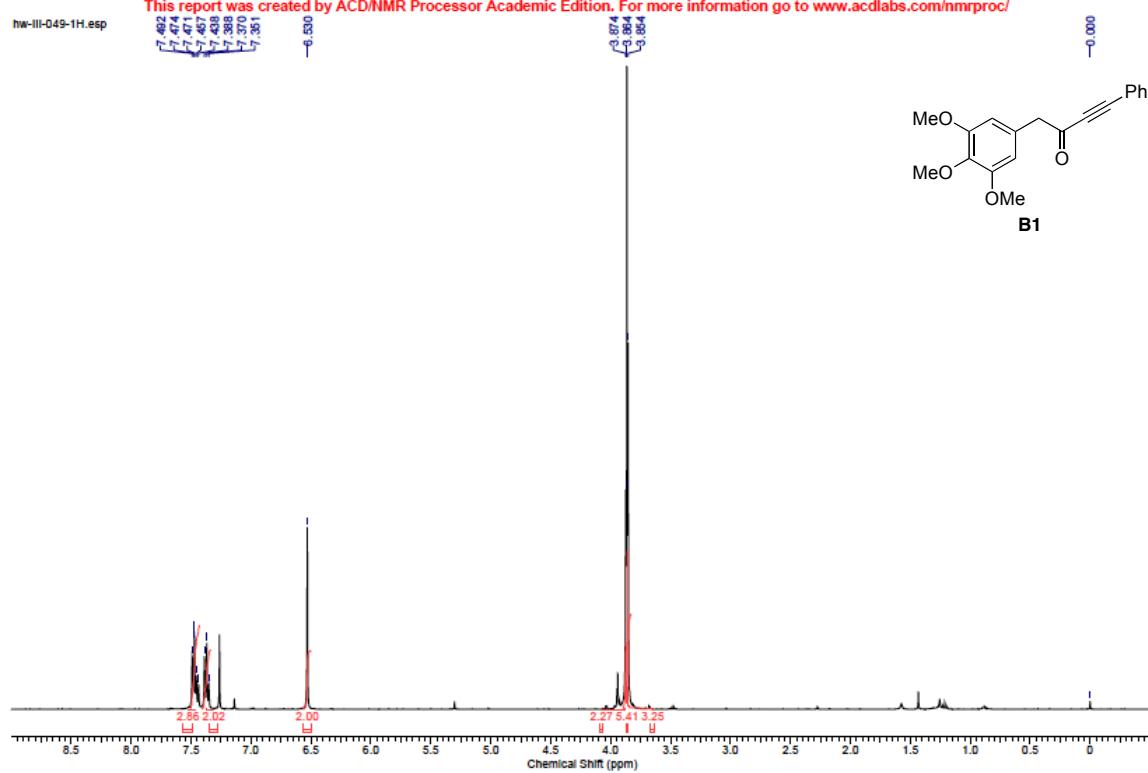


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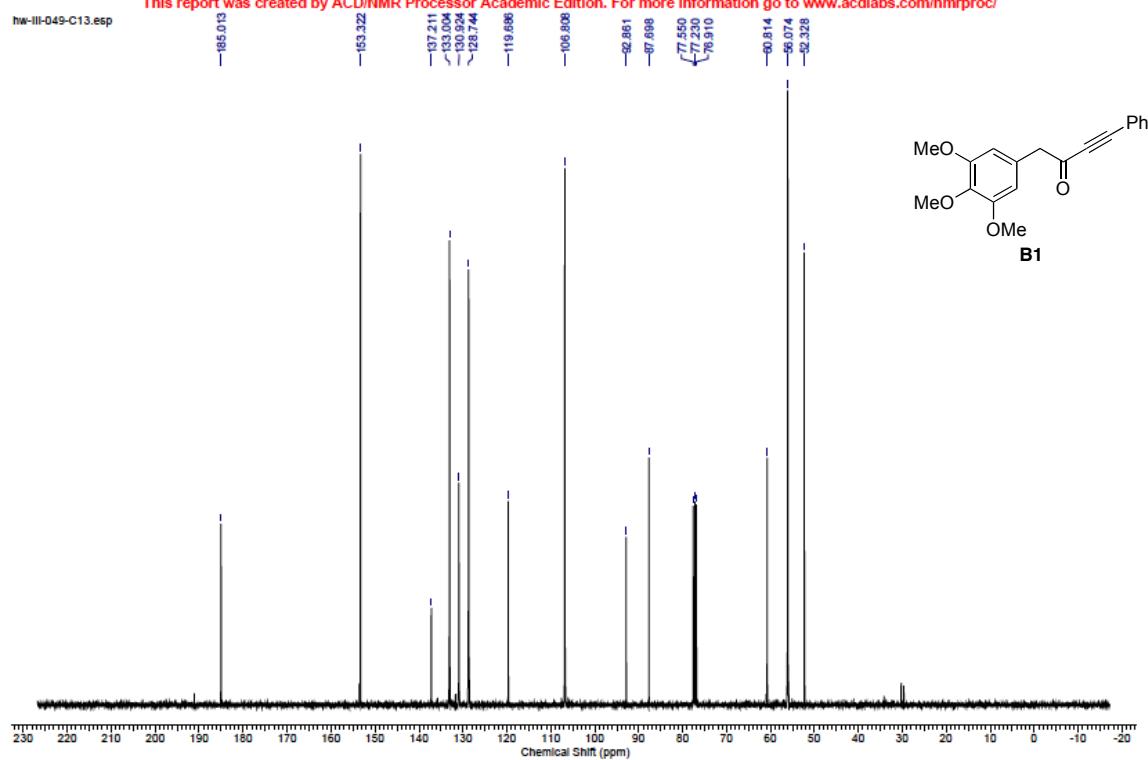


¹H and ¹³C spectra of compound B0

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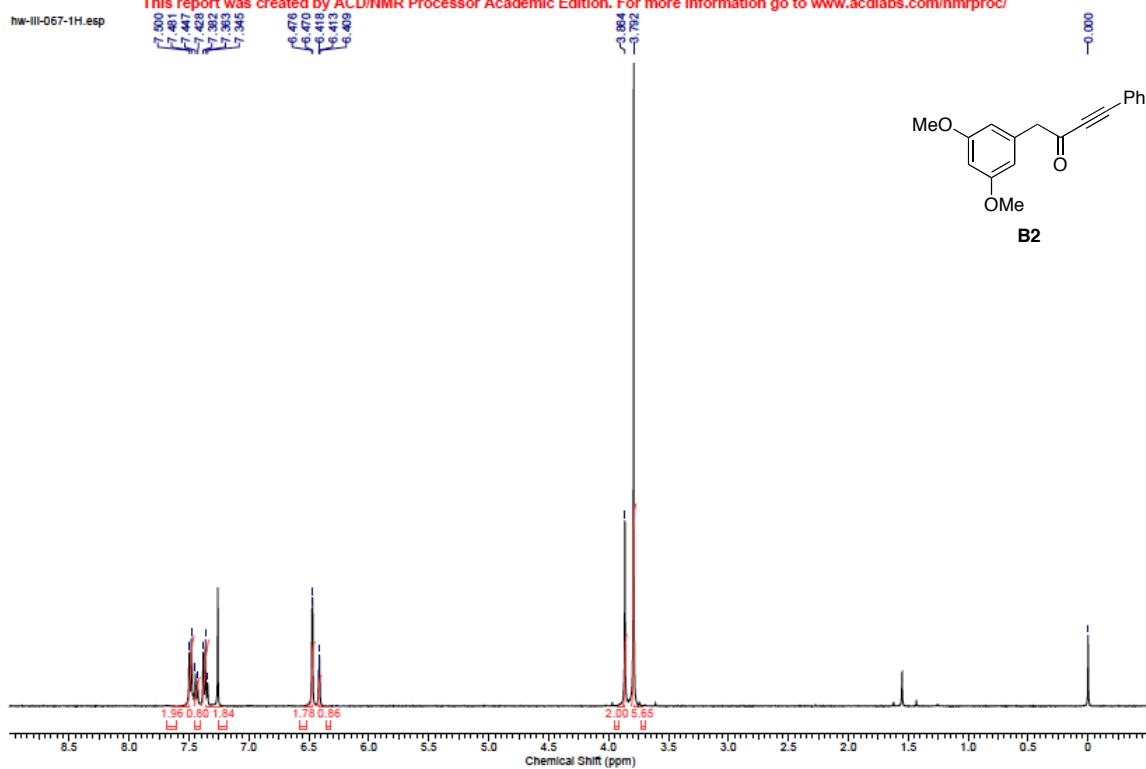


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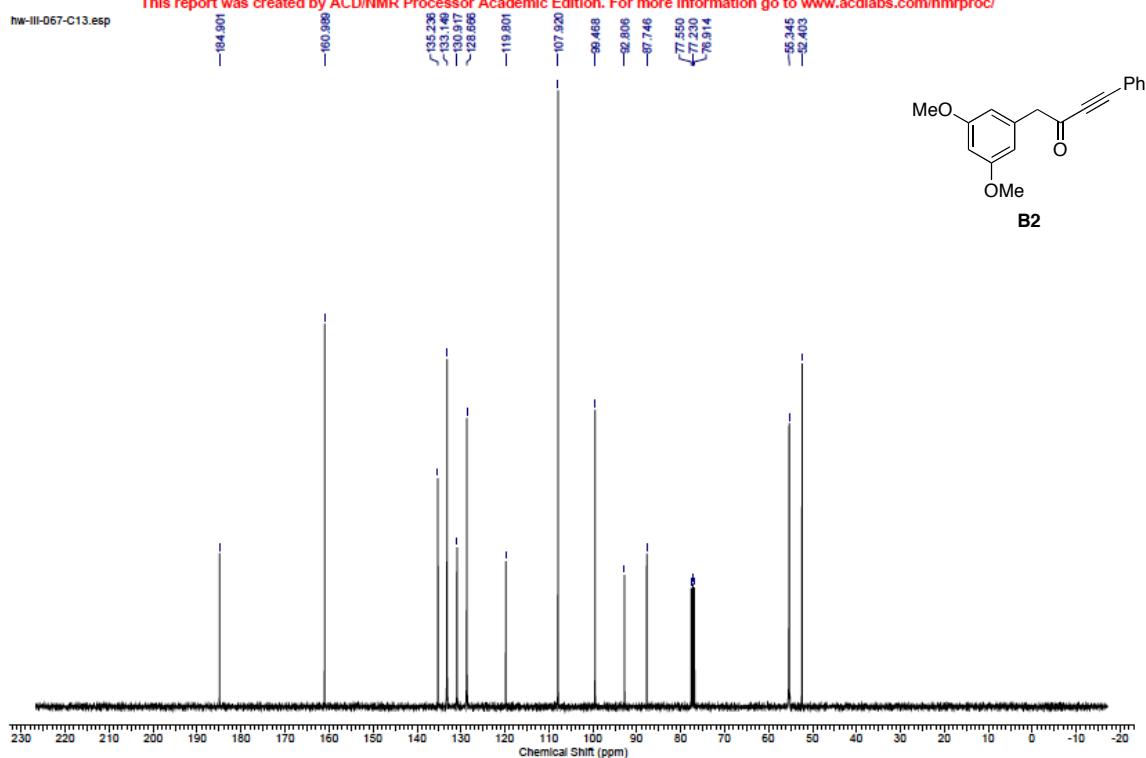


¹H and ¹³C spectra of compound B1

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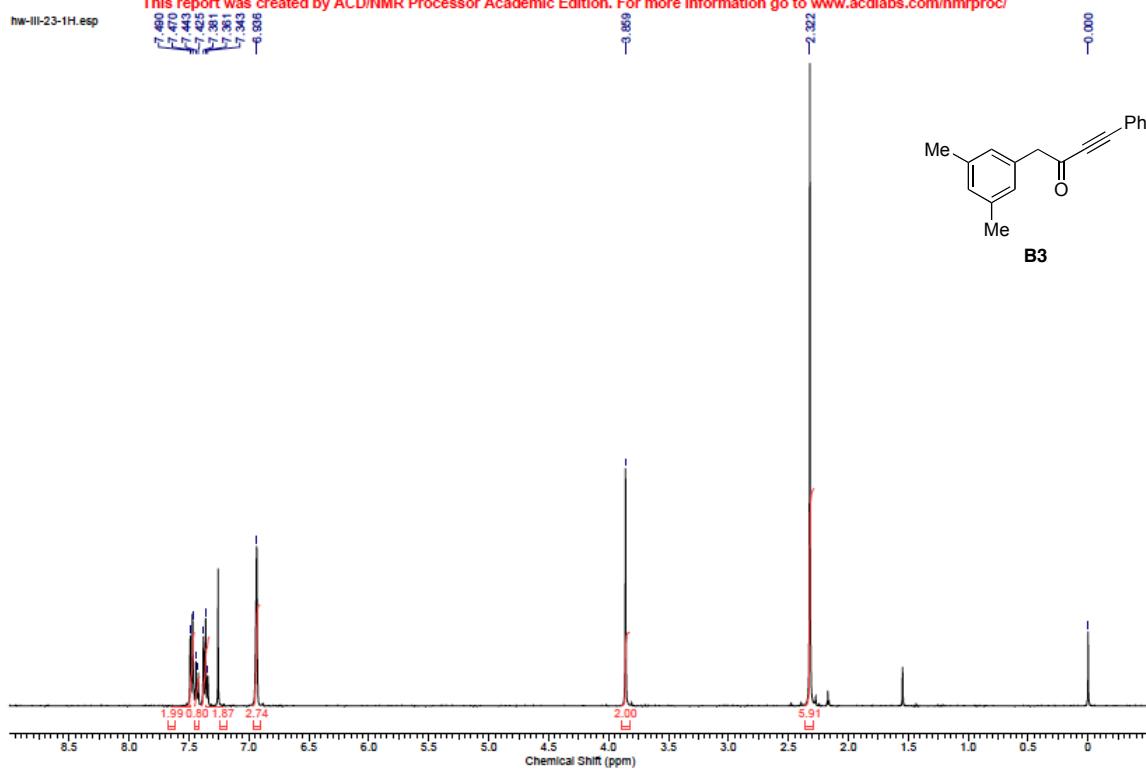


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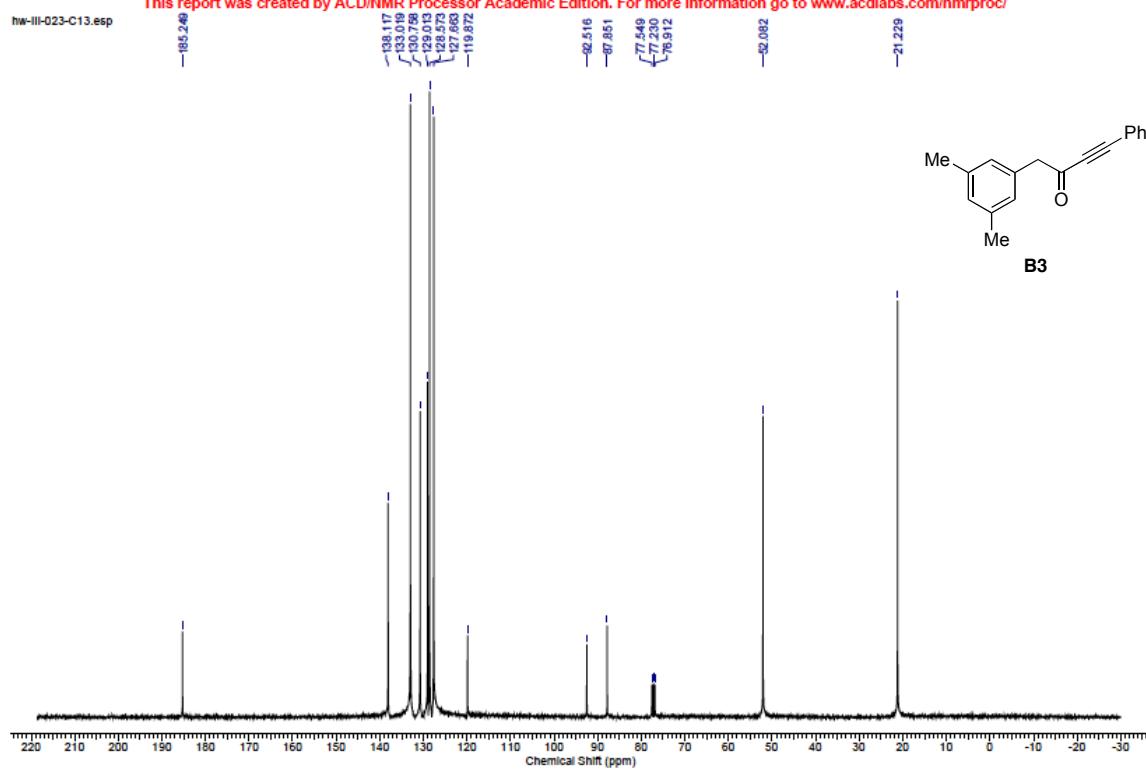


¹H and ¹³C spectra of compound B2

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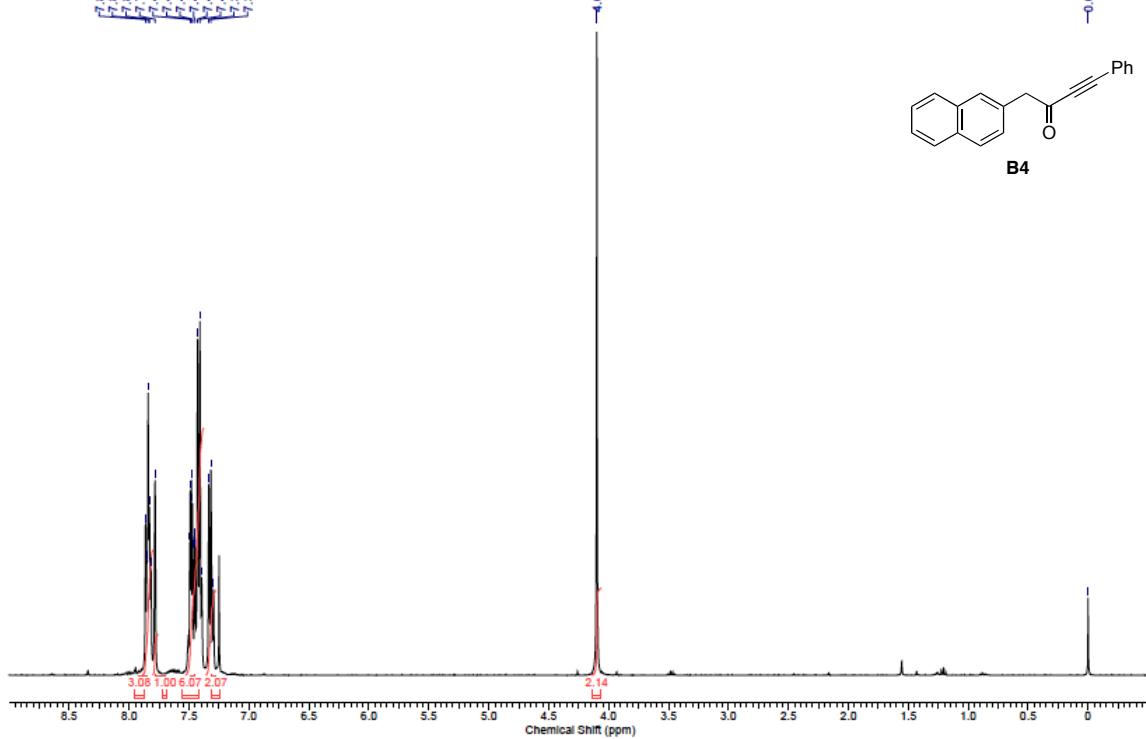


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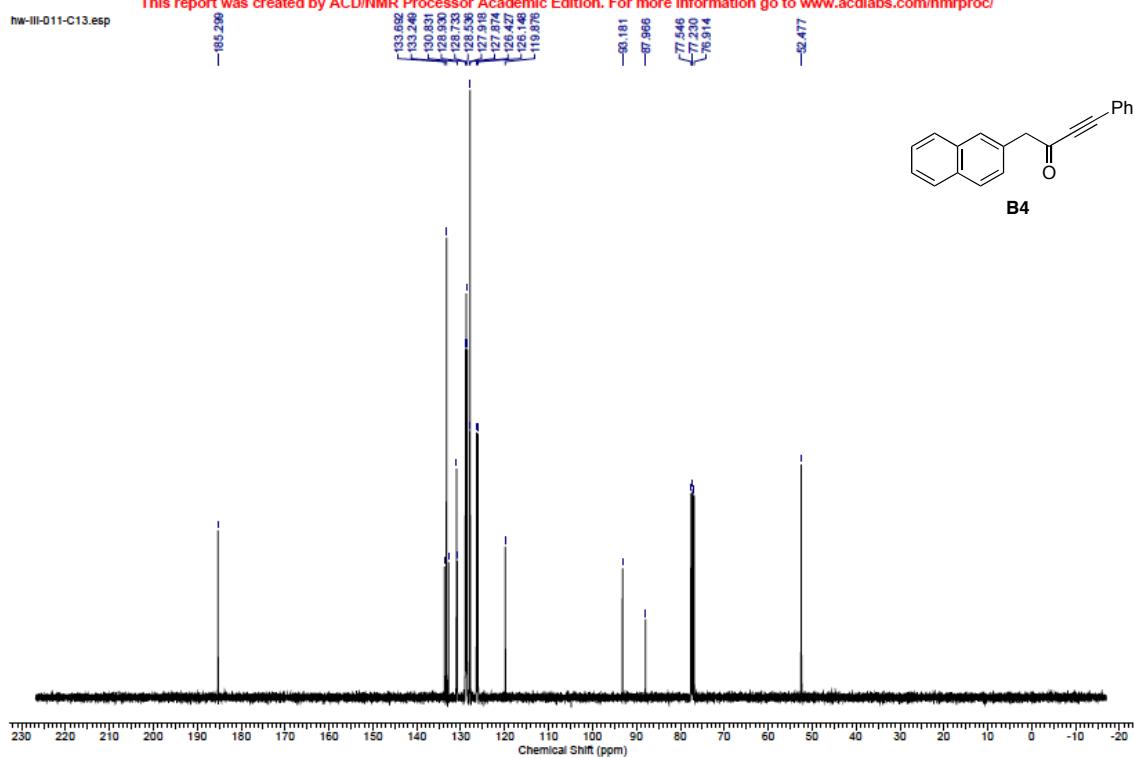


¹H and ¹³C spectra of compound B3

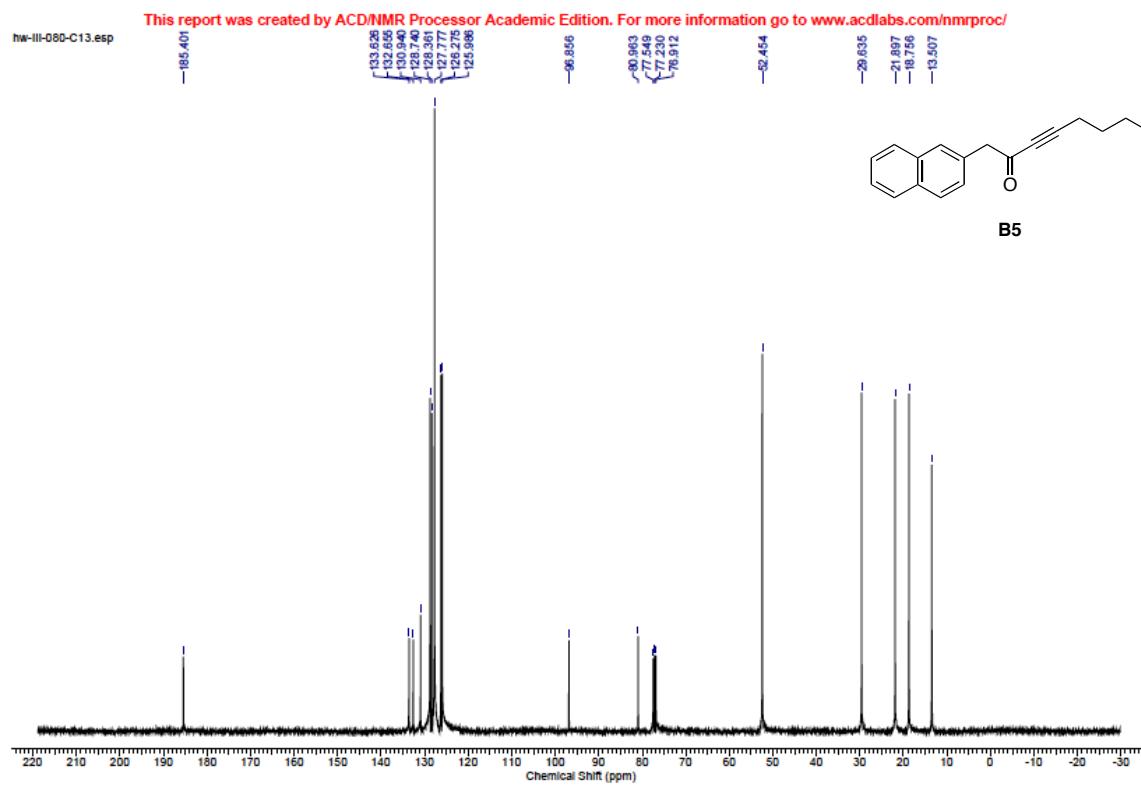
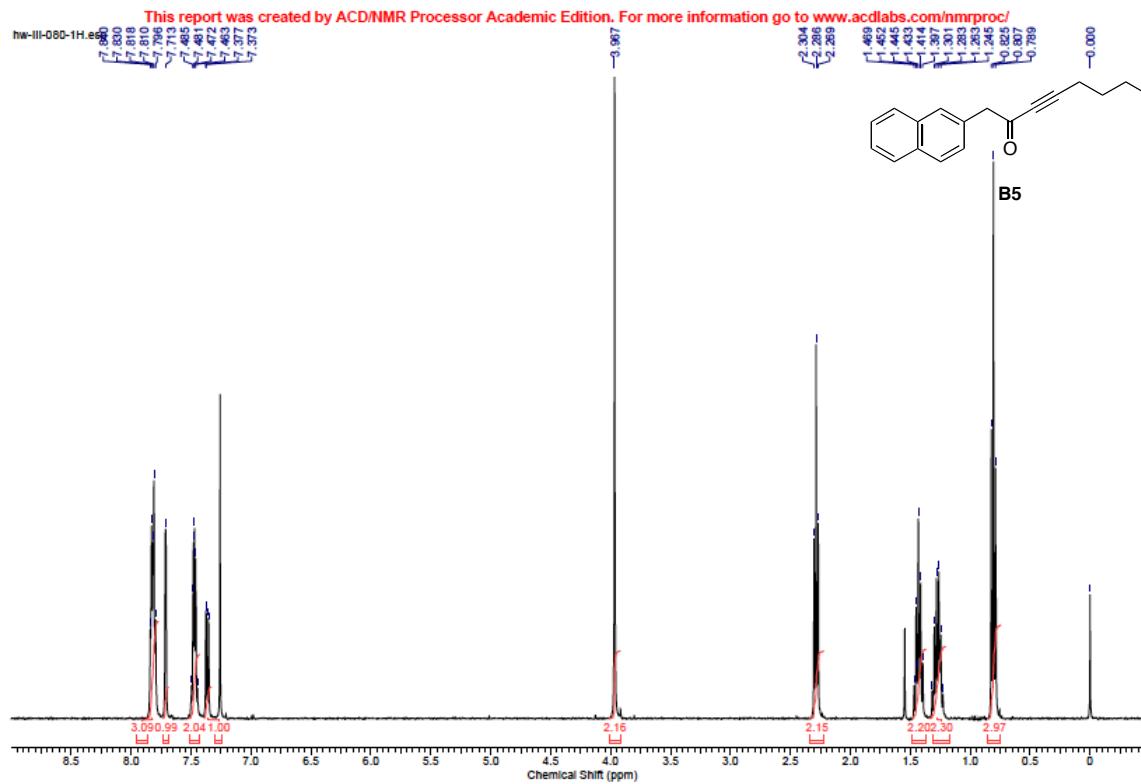
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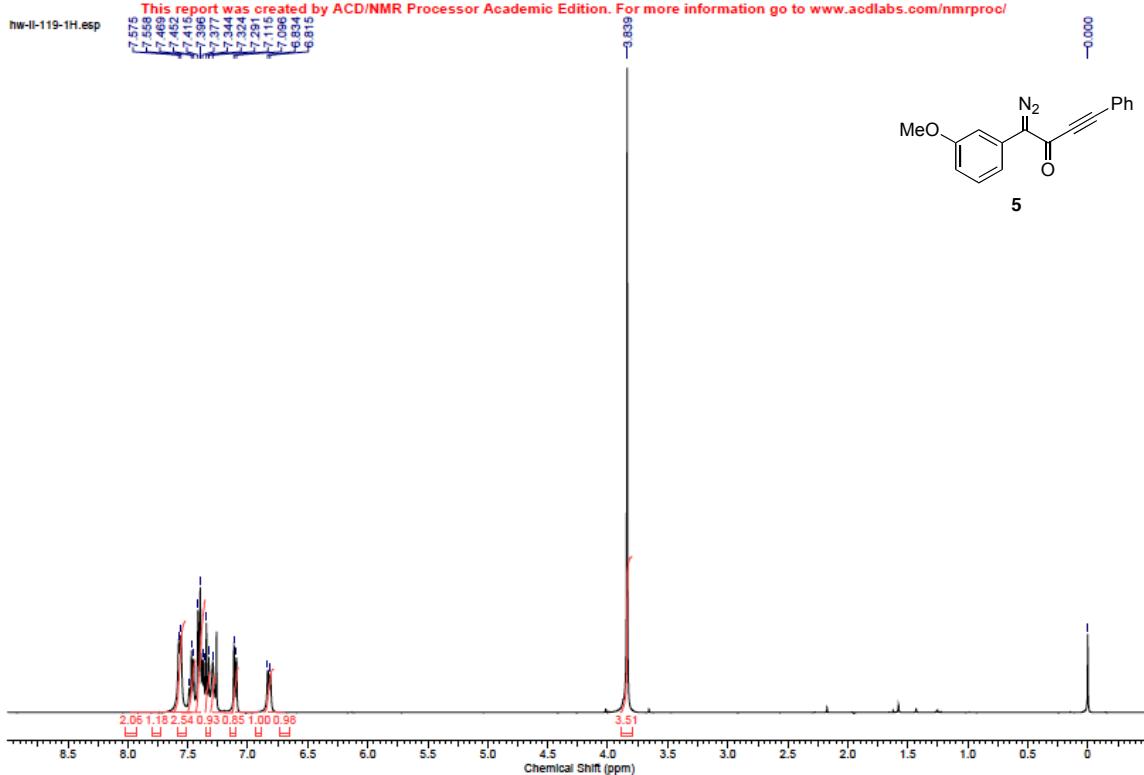


¹H and ¹³C spectra of compound B4

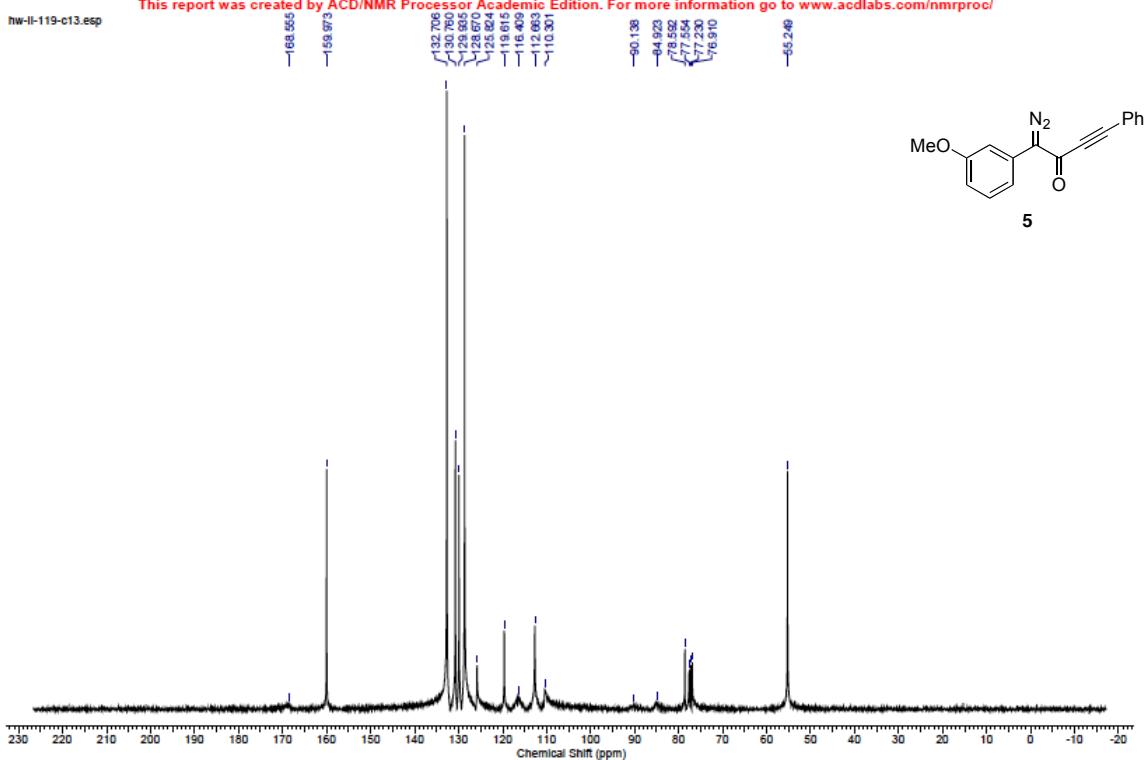


¹H and ¹³C spectra of compound **B5**

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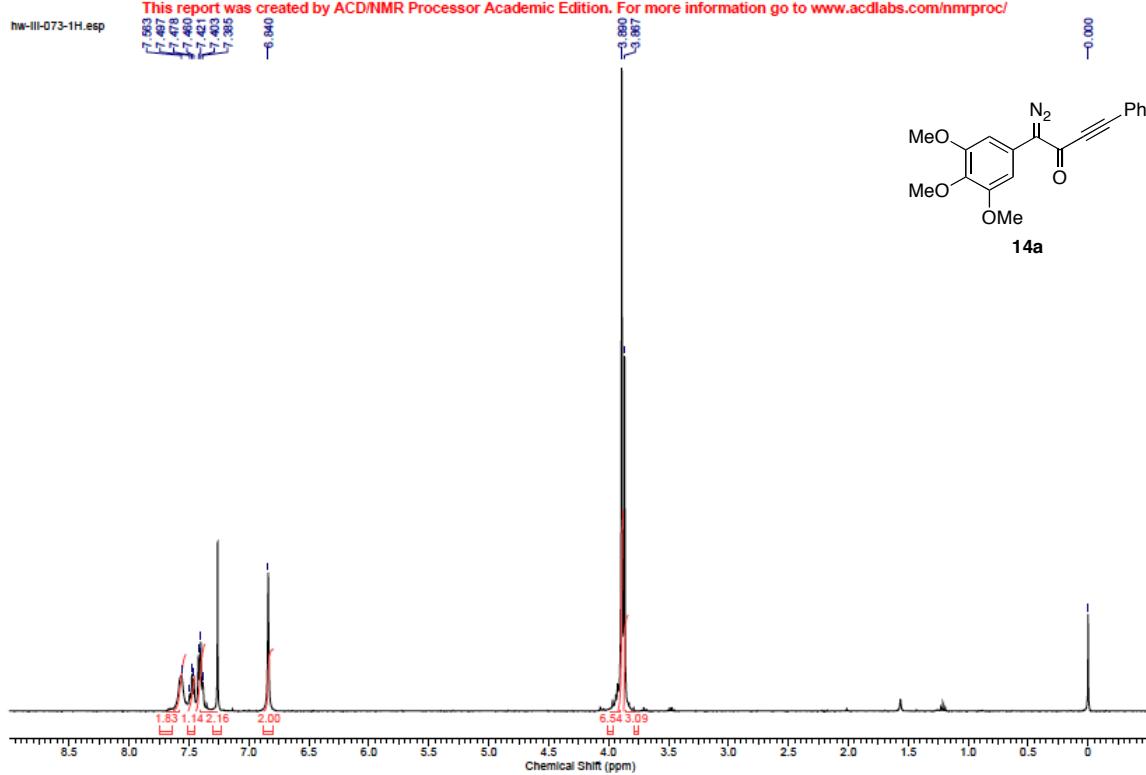


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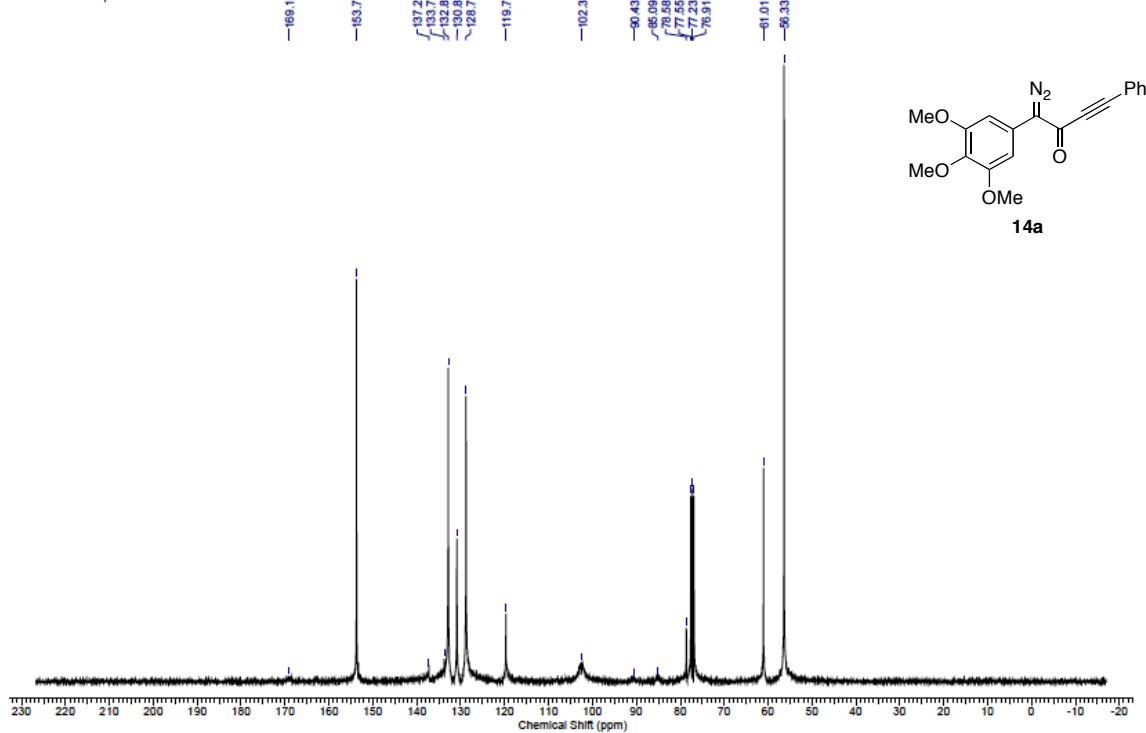


¹H and ¹³C spectra of compound **5**

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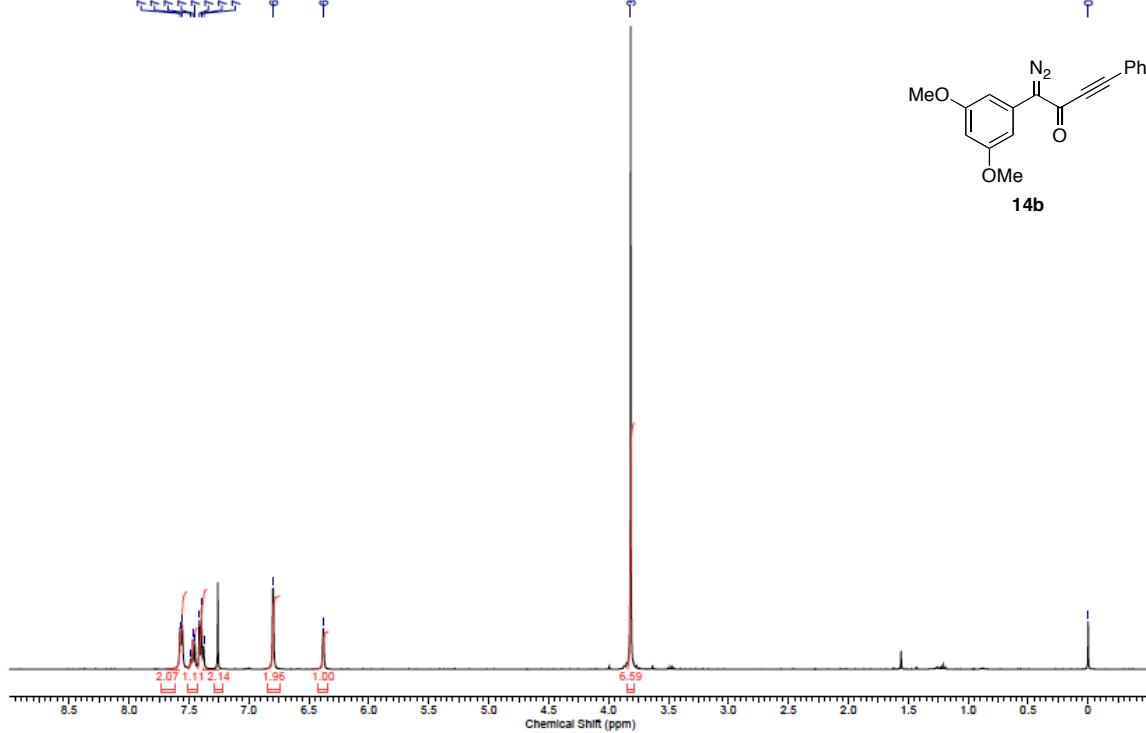


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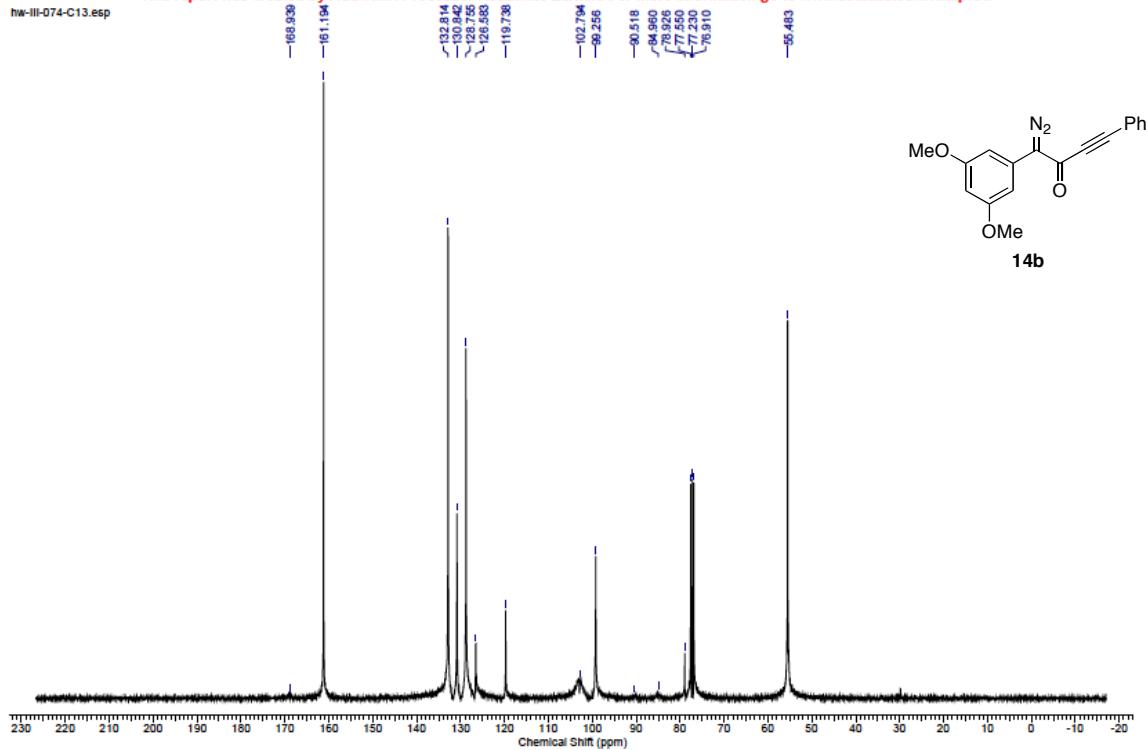


¹H and ¹³C spectra of compound 14a

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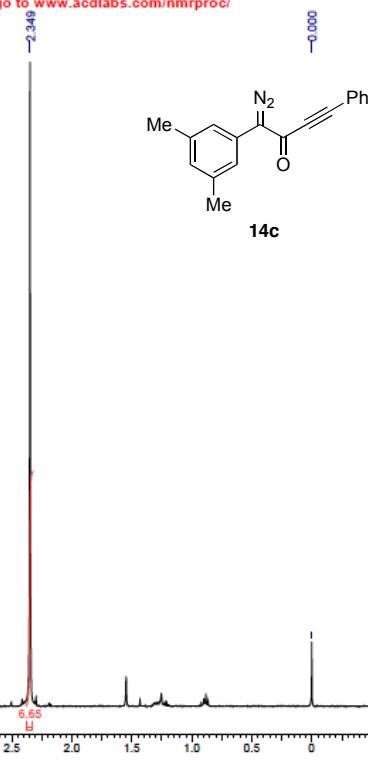


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¹H and ¹³C spectra of compound 14b

hw-III-033-1H.esp This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/
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7.555
7.466
7.447
7.412
7.393
7.376
7.216
-6.921



hw-III-033-aftercolumn-013

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File : exp
Sample id : tmptestudy

Pulse Sequence: s2pul

Solvent: cdc13

Temp: 23.0 C / 295.1 K

Operator: davies

VNMRS-400 "v400"

Relax. delay 2.500 sec

Pulse 45.0 degrees

Acu. time 2.500 sec

Width 24509.8 Hz

320 repetitions

OBSERVE C13, 100.5265401 MHz

INCOHERENT HI, 399.7889220 MHz

Power 45 dB

continuously on

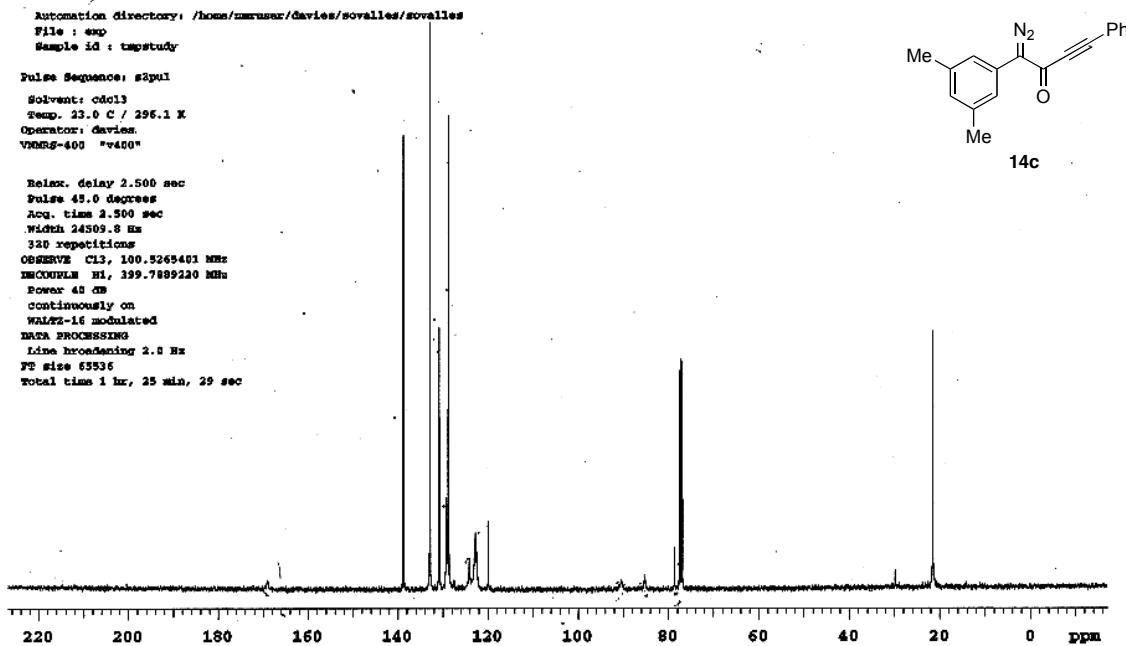
WALZ-16 modulated

DATA PROCESSING

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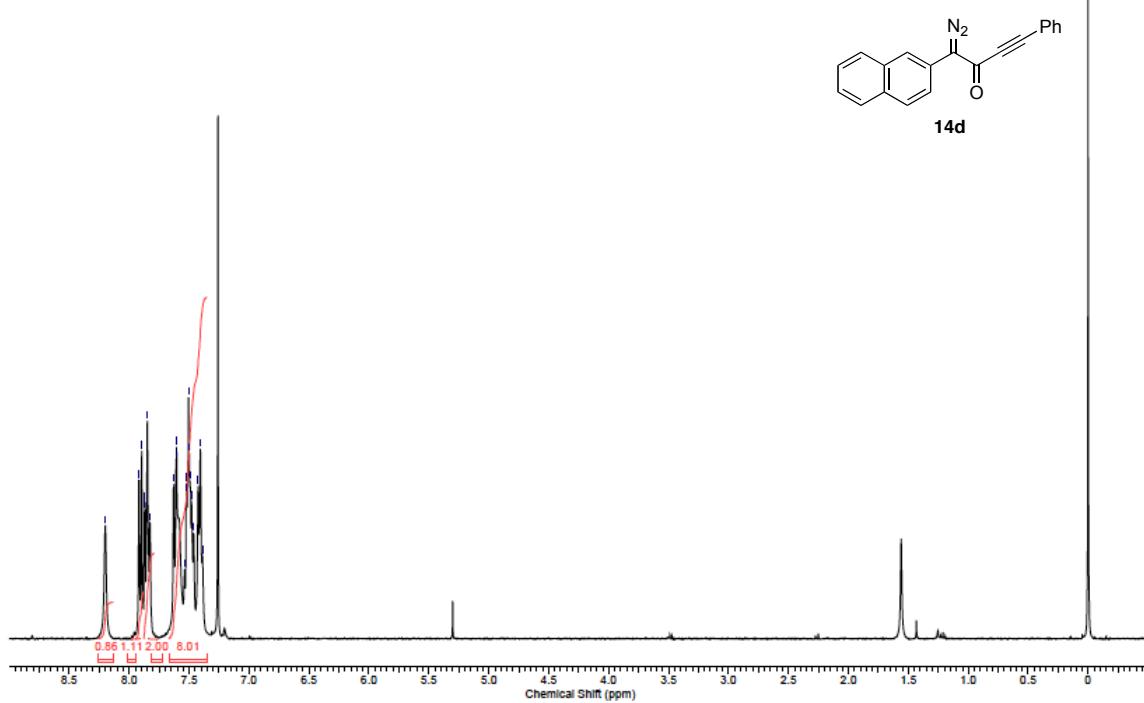
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Total time 1 hr, 25 min, 29 sec

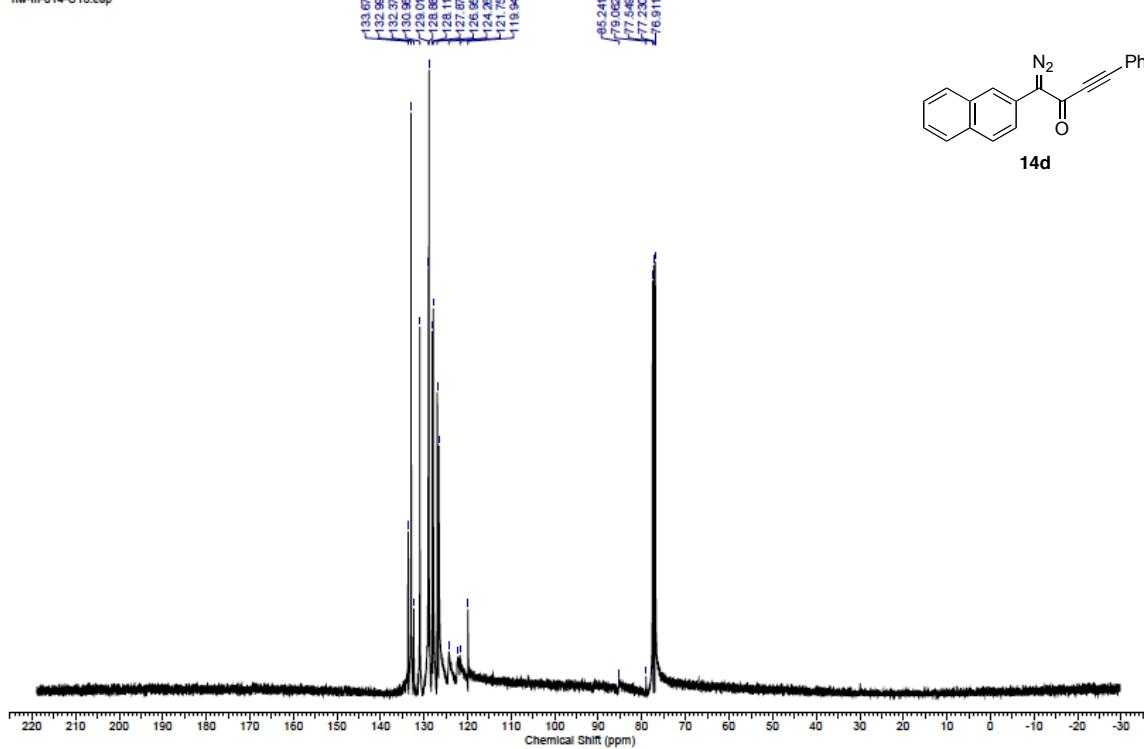


¹H and ¹³C spectra of compound 14c

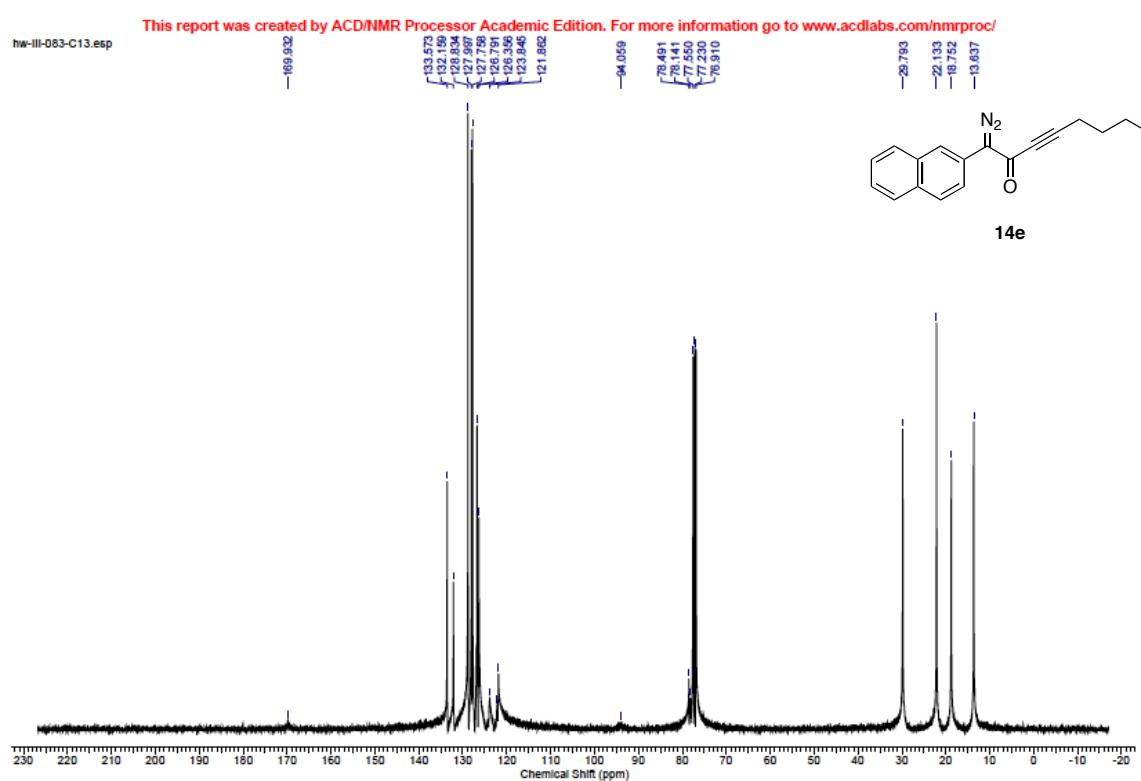
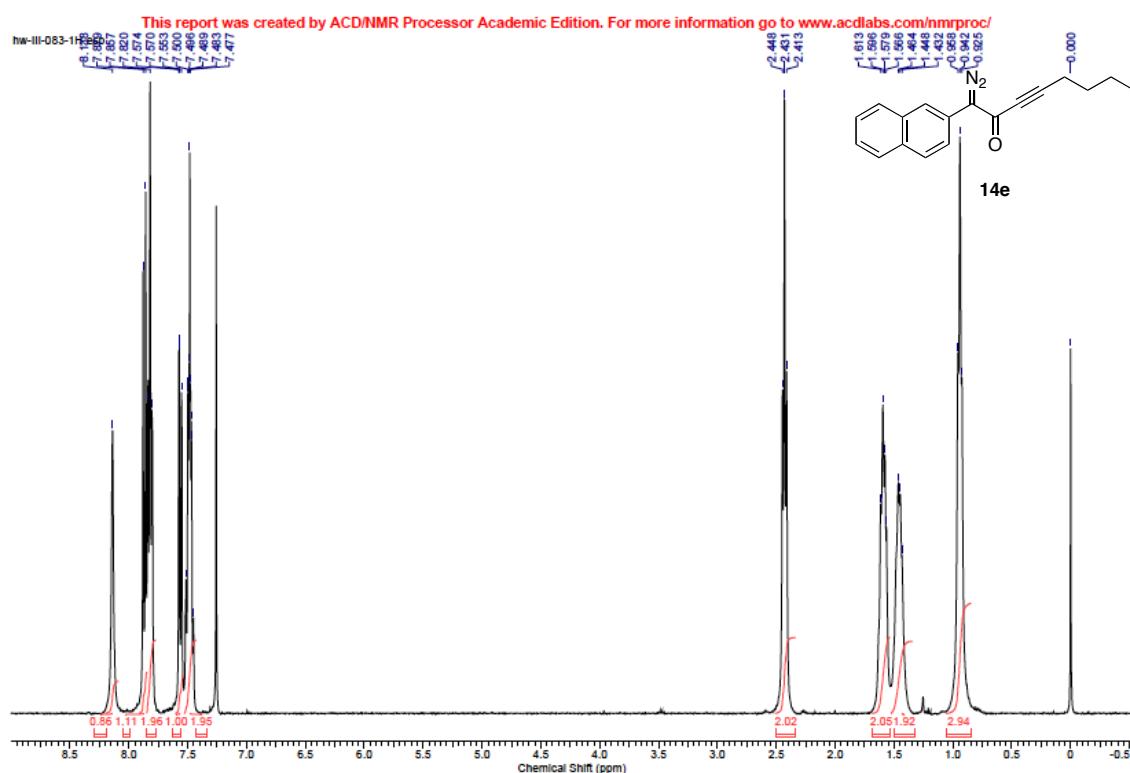
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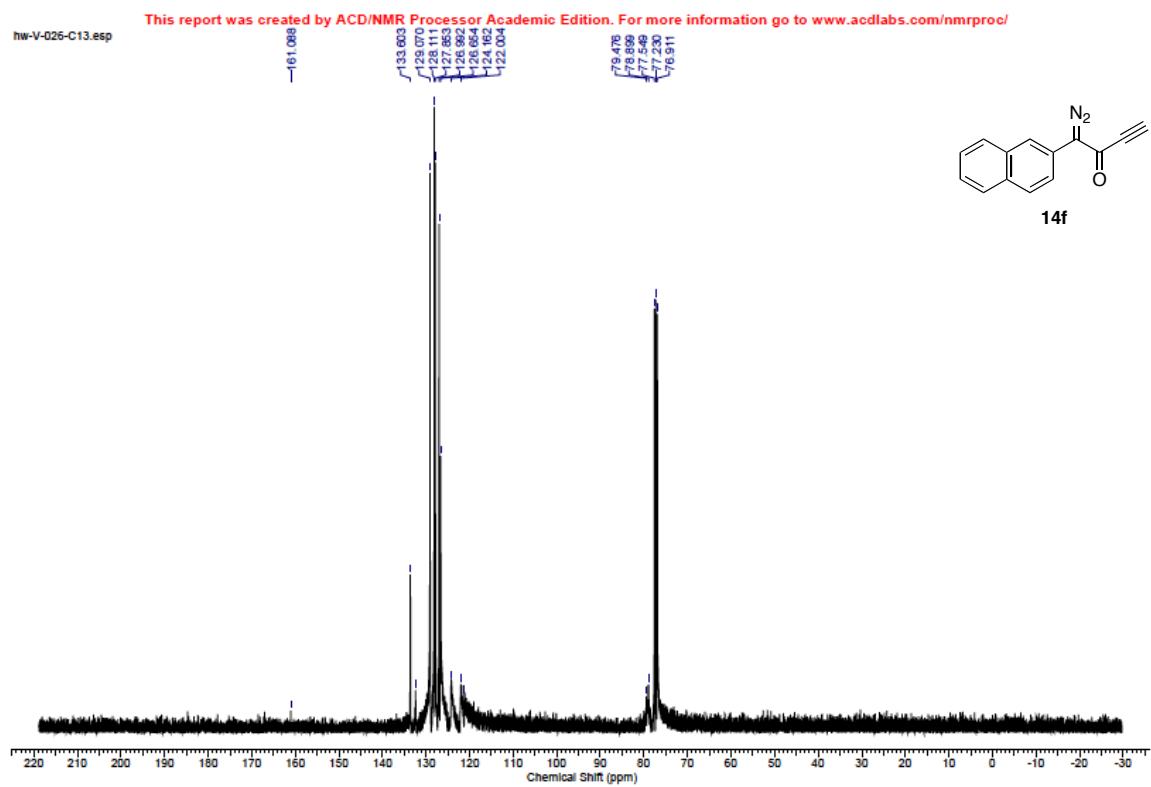
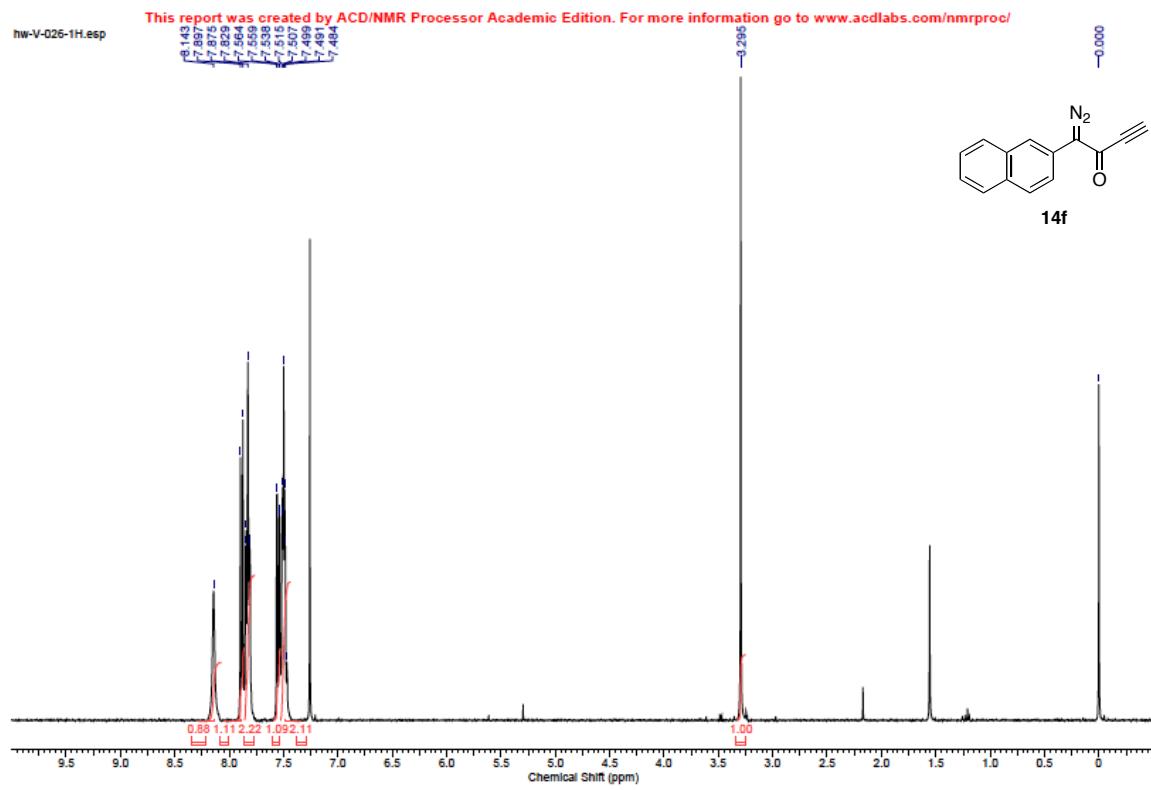
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¹H and ¹³C spectra of compound 14d

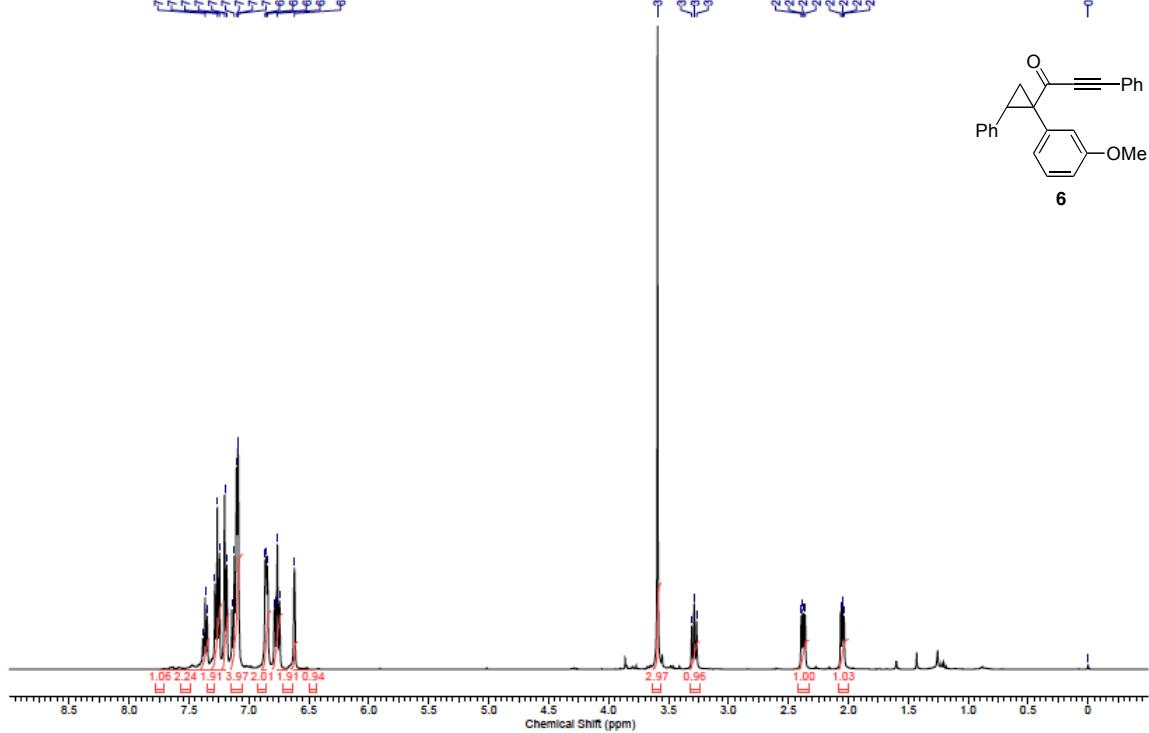


¹H and ¹³C spectra of compound **14e**

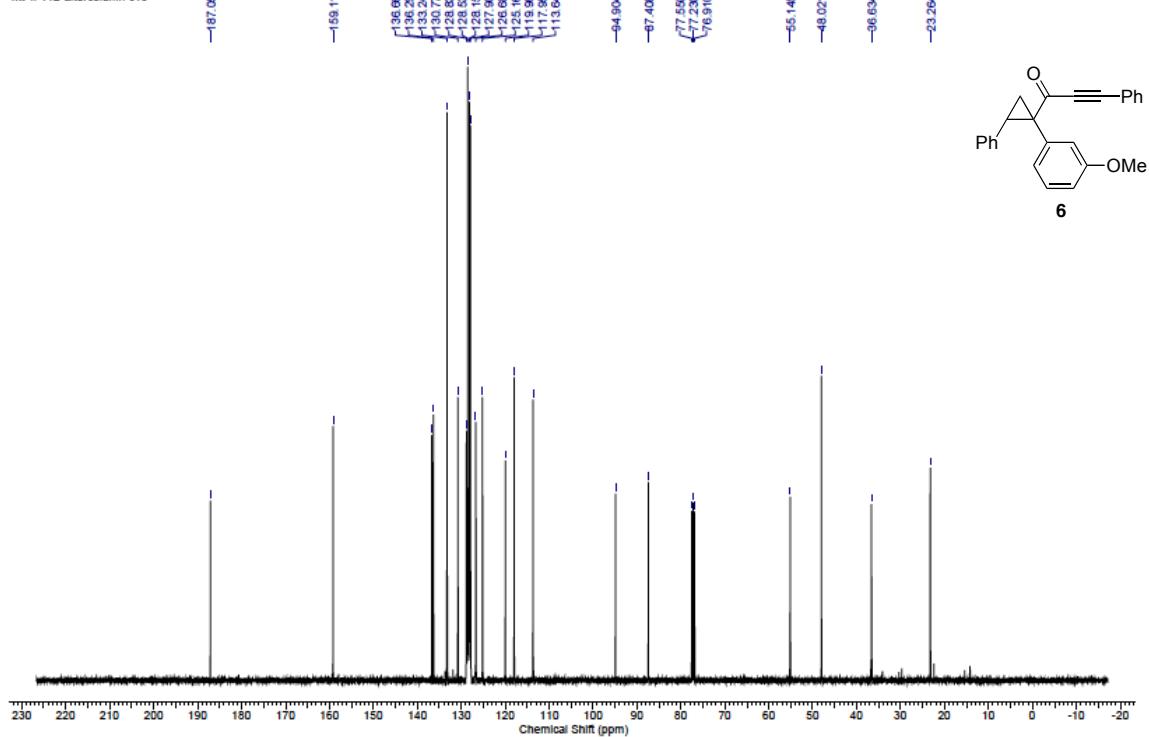


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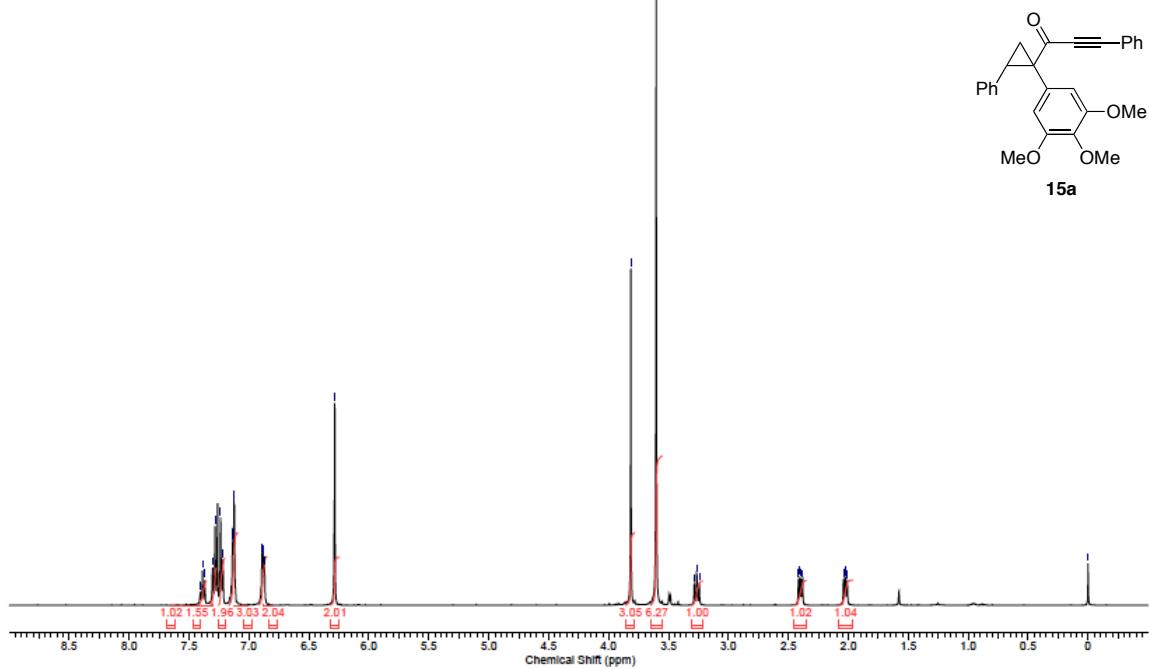


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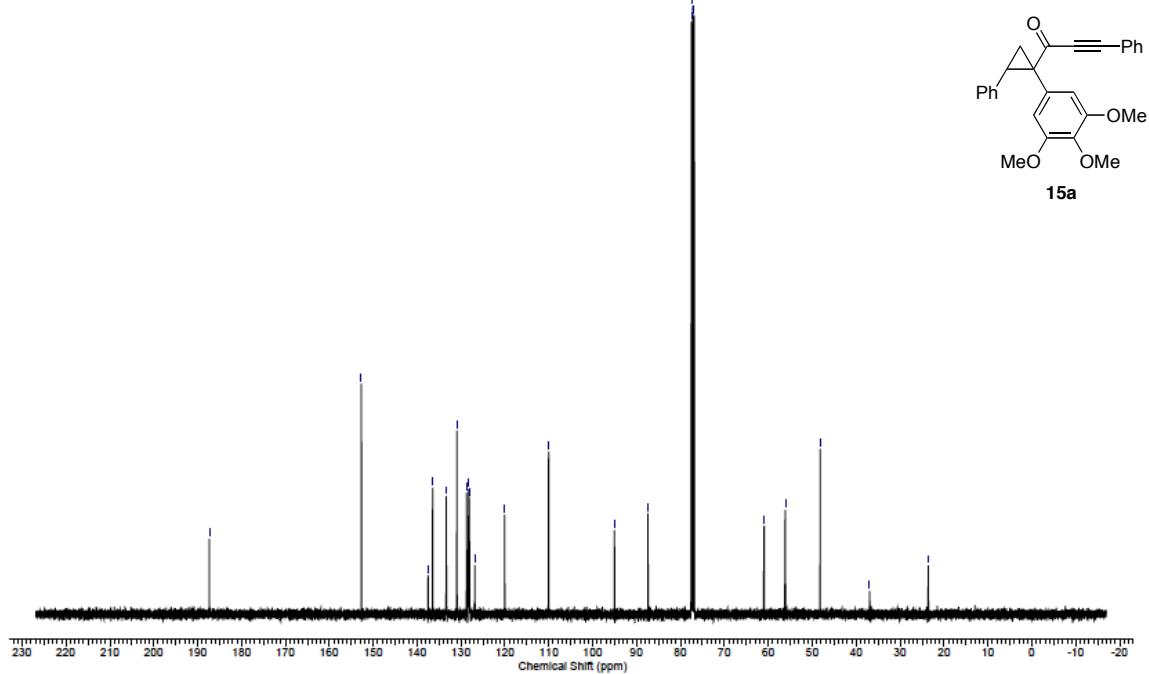


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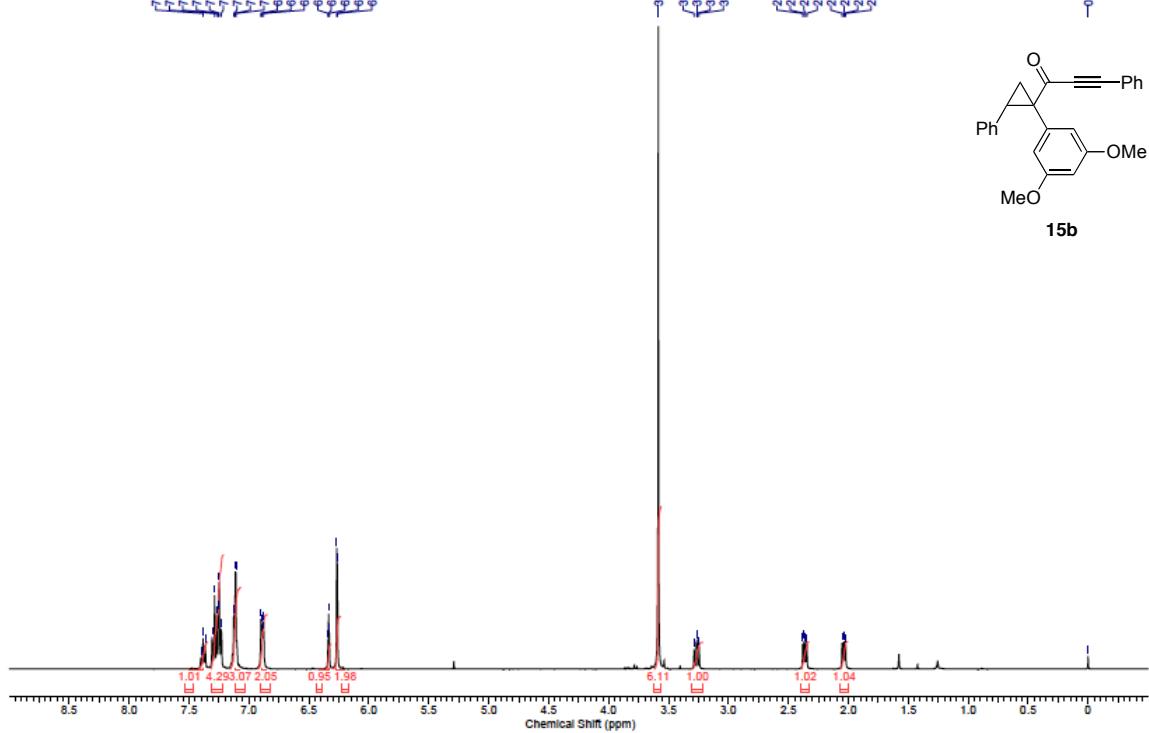


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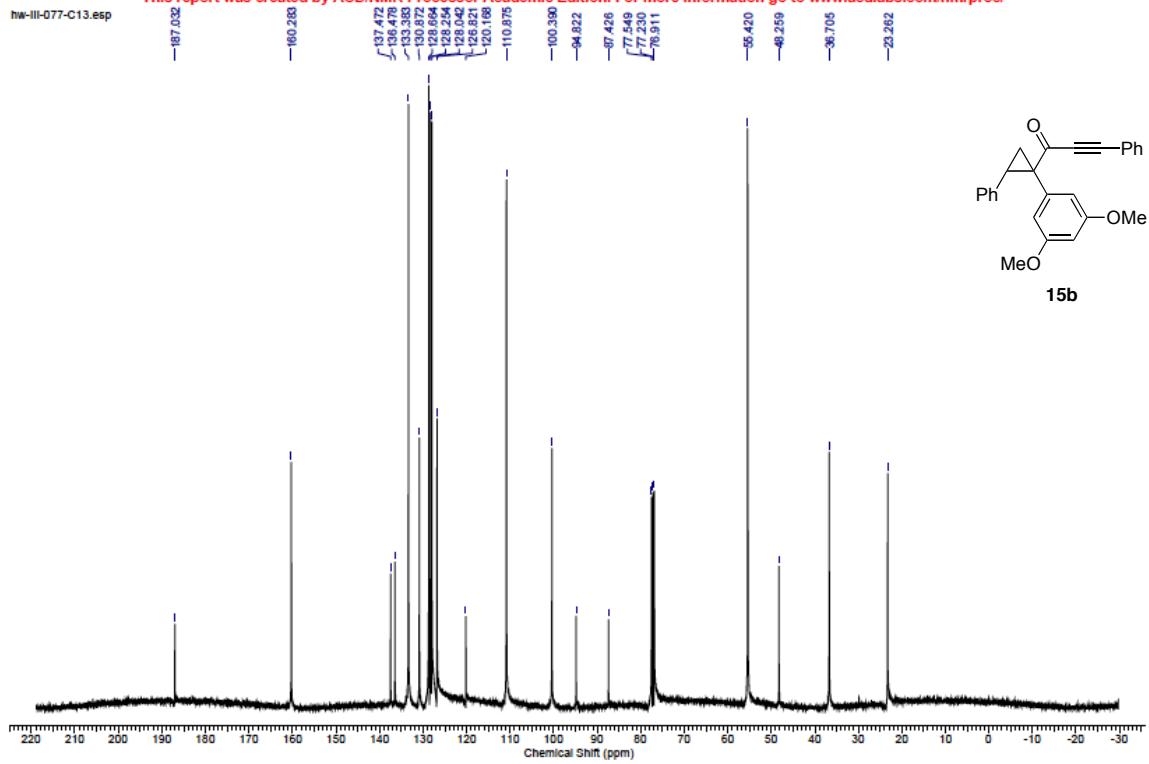


¹H and ¹³C spectra of compound 15a

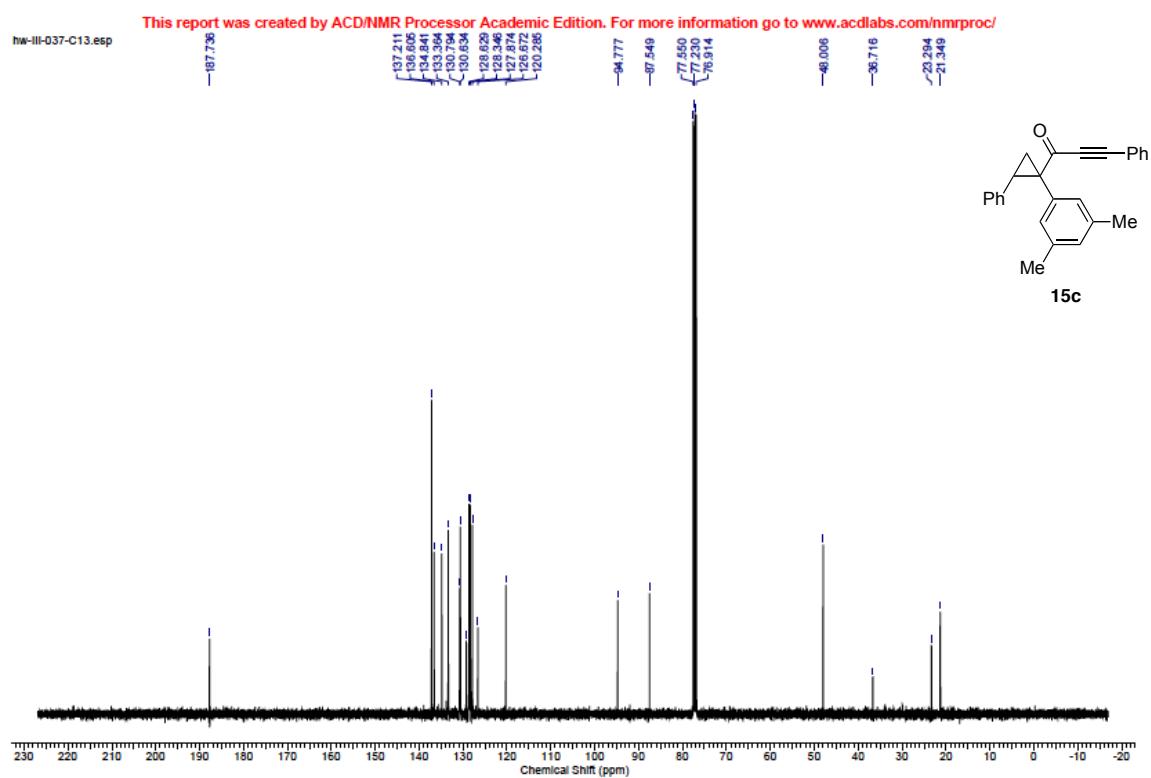
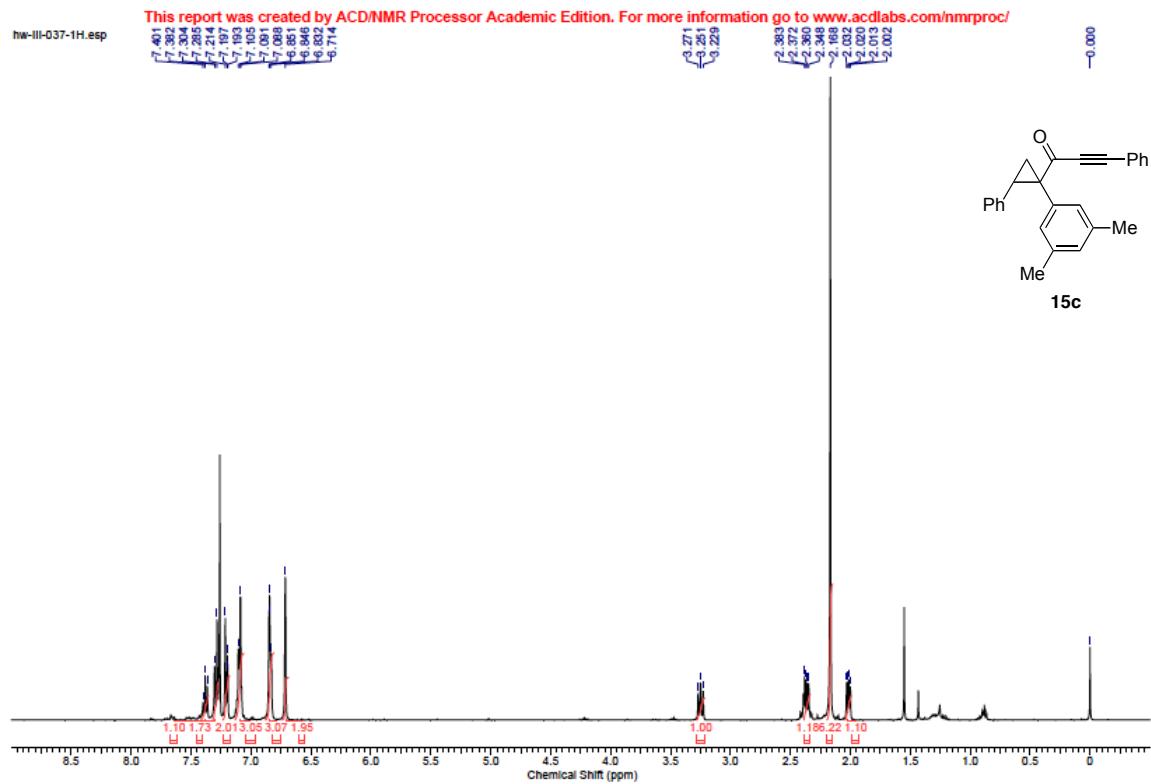
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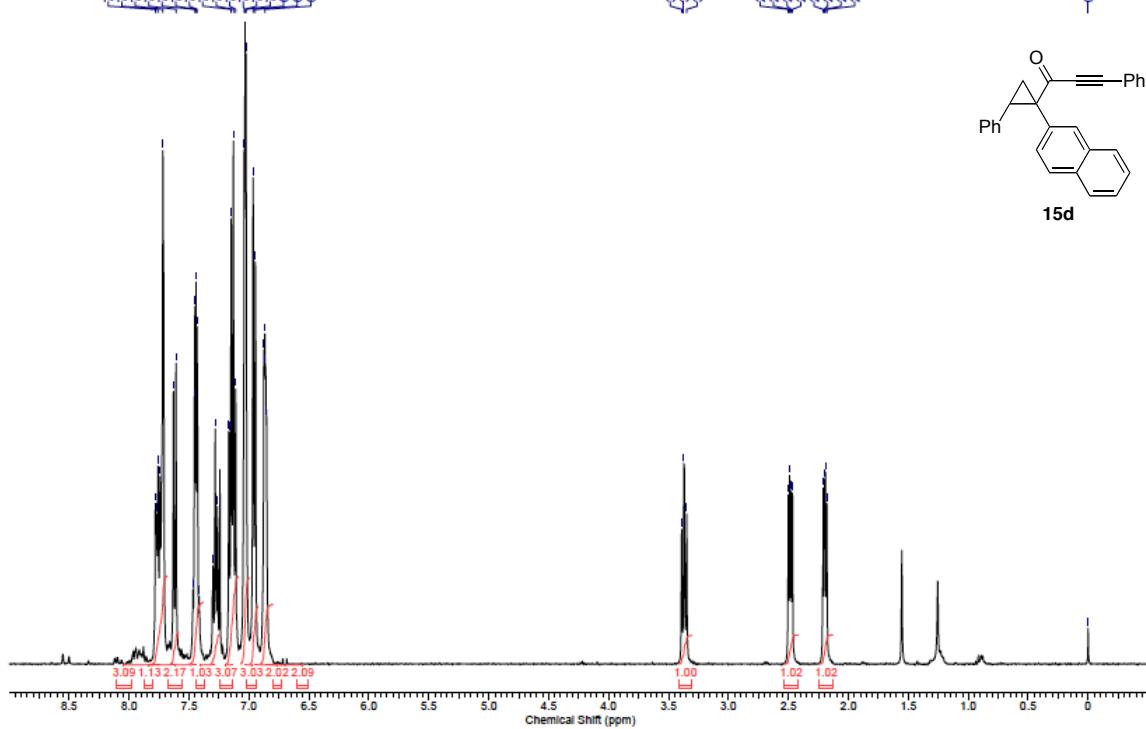


¹H and ¹³C spectra of compound 15b

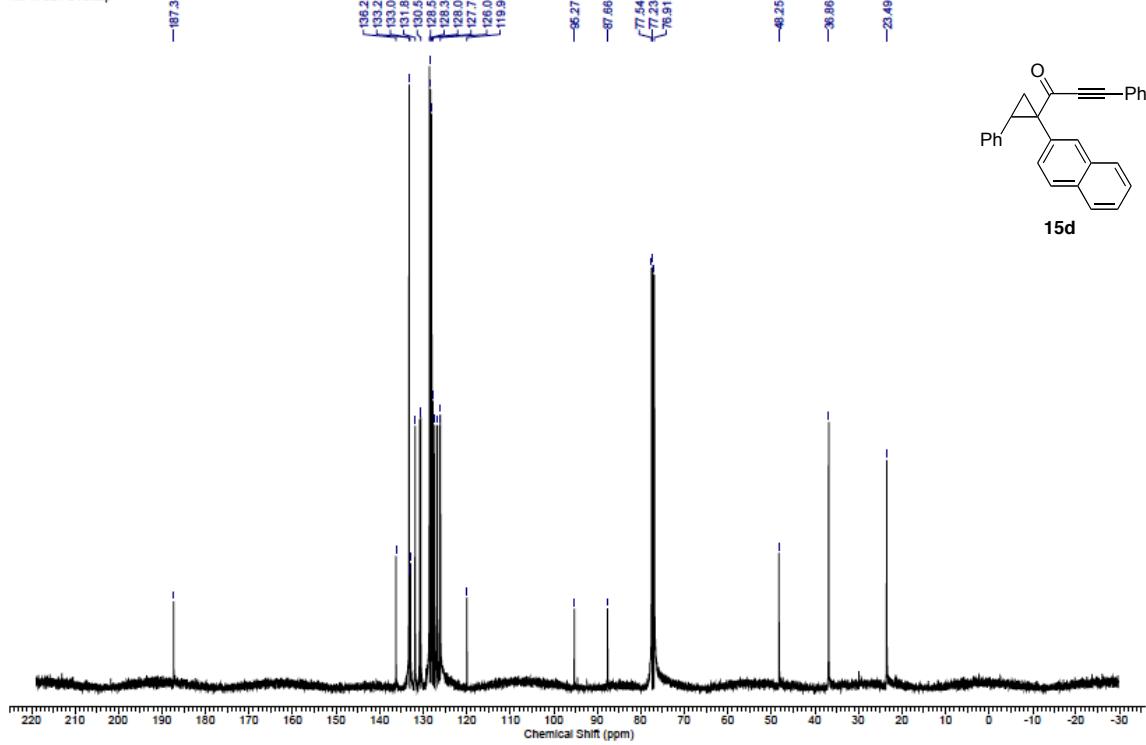


¹H and ¹³C spectra of compound 15c

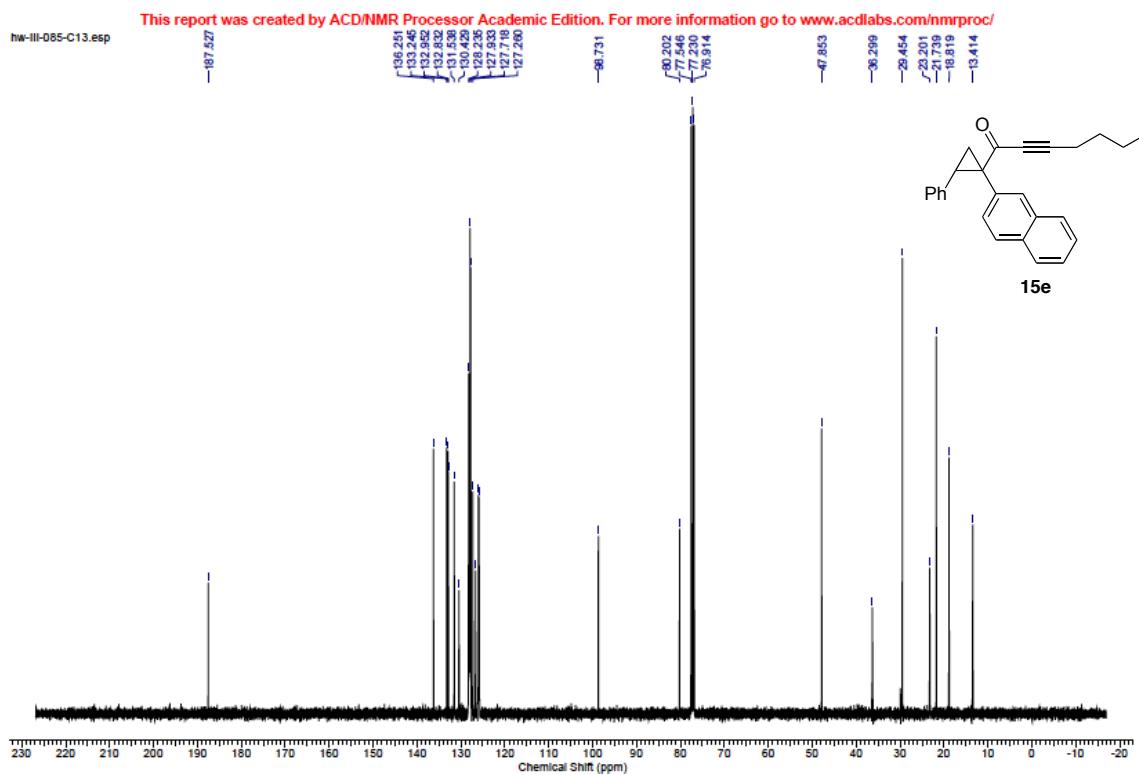
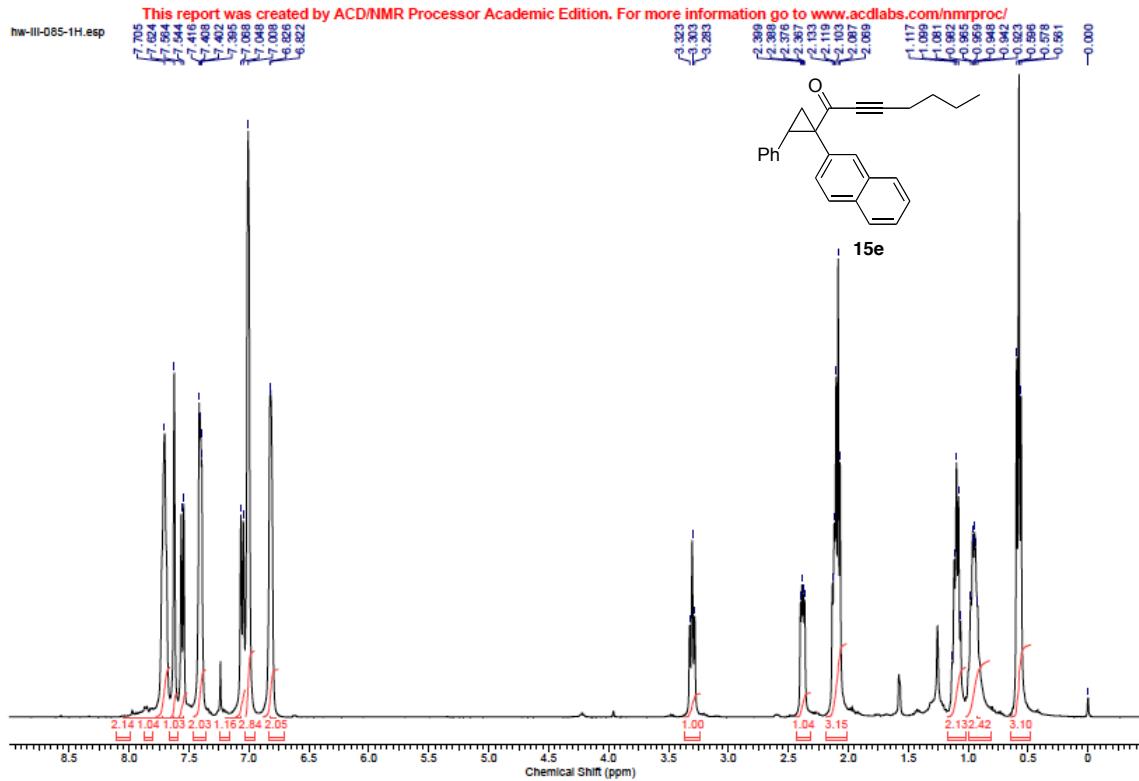
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¹H and ¹³C spectra of compound 15d

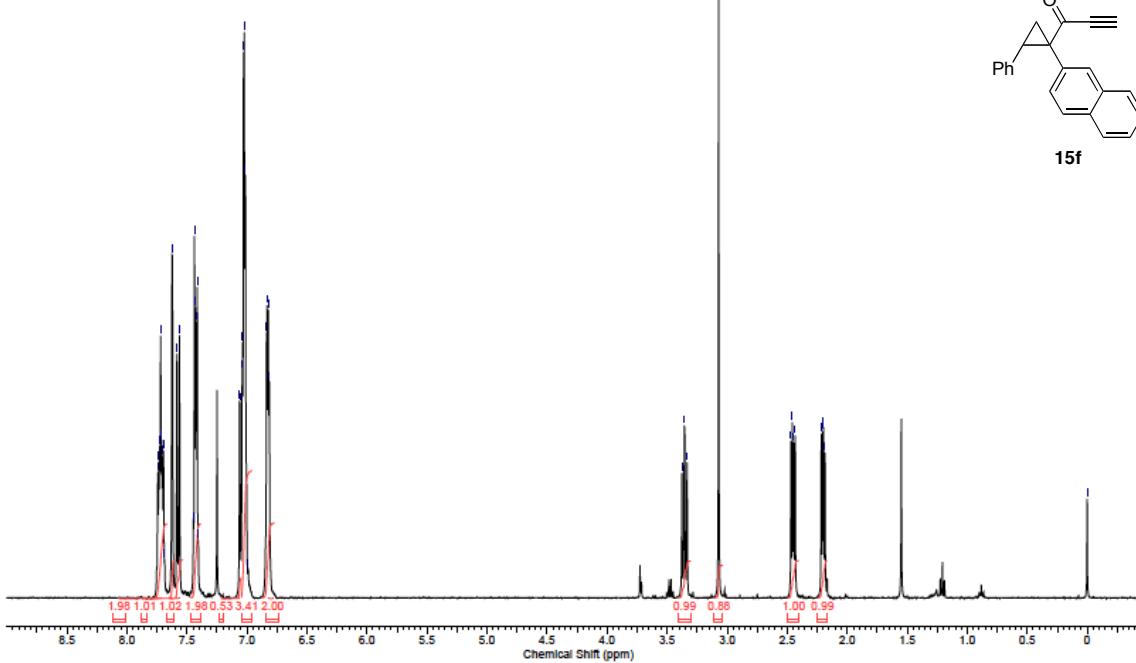
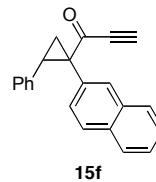


¹H and ¹³C spectra of compound 15e

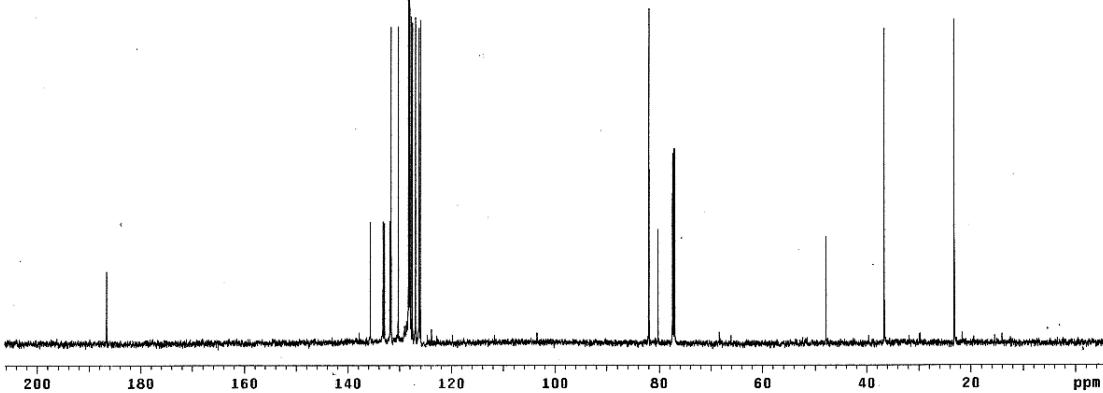
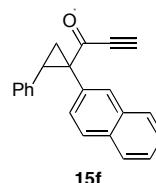
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-0.071 -0.056 -0.041 -0.035 -0.032 -0.027 -0.022 -0.017 -0.014

O ppm

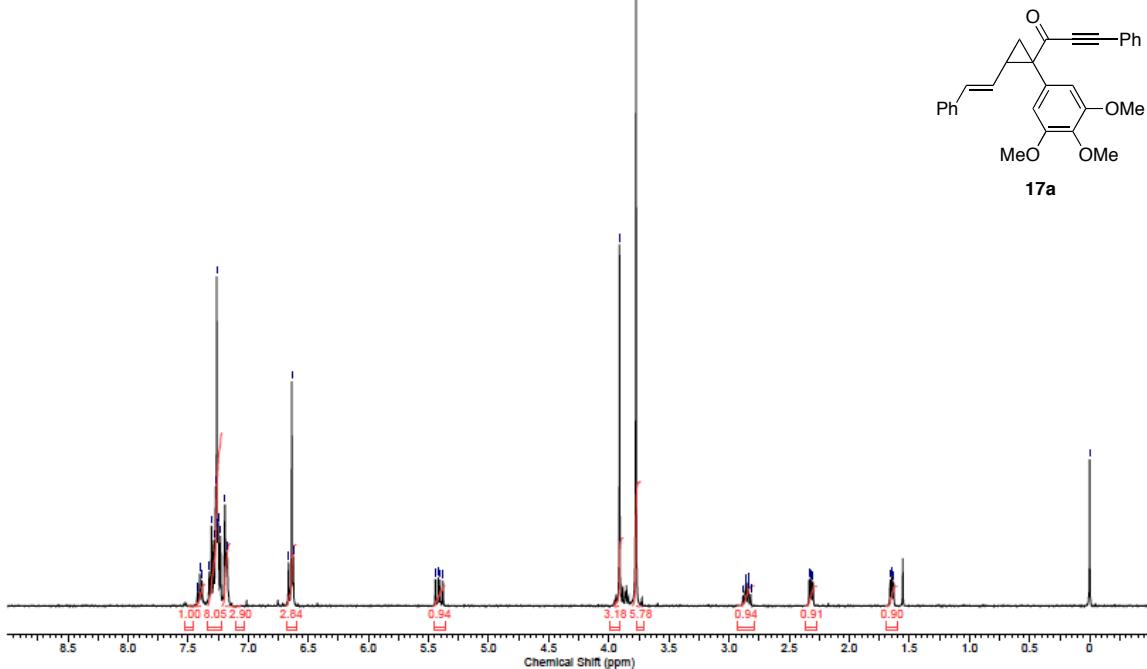


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Solvent: cdc13
Ambient temperature
User: 1-14-57
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PULSE SEQUENCE
Relaxation time: 2.000 sec,
Pulse 69.2 degrees
Acq. time 1.000 sec
Width 31948.9 Hz
576 scans
OBSERVE C13, 150.7688373 MHz
DECOUPLE H1, 599.5999983 MHz
Power 36
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WALT2-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 8 hr, 33 min, 51 sec
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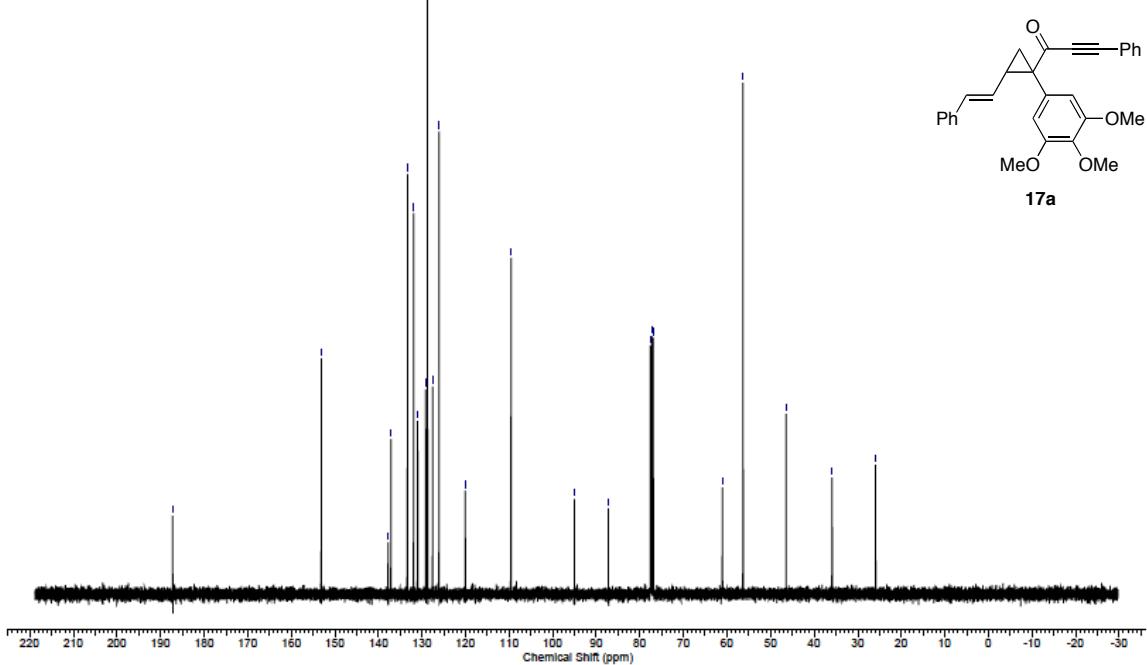


¹H and ¹³C spectra of compound 15f

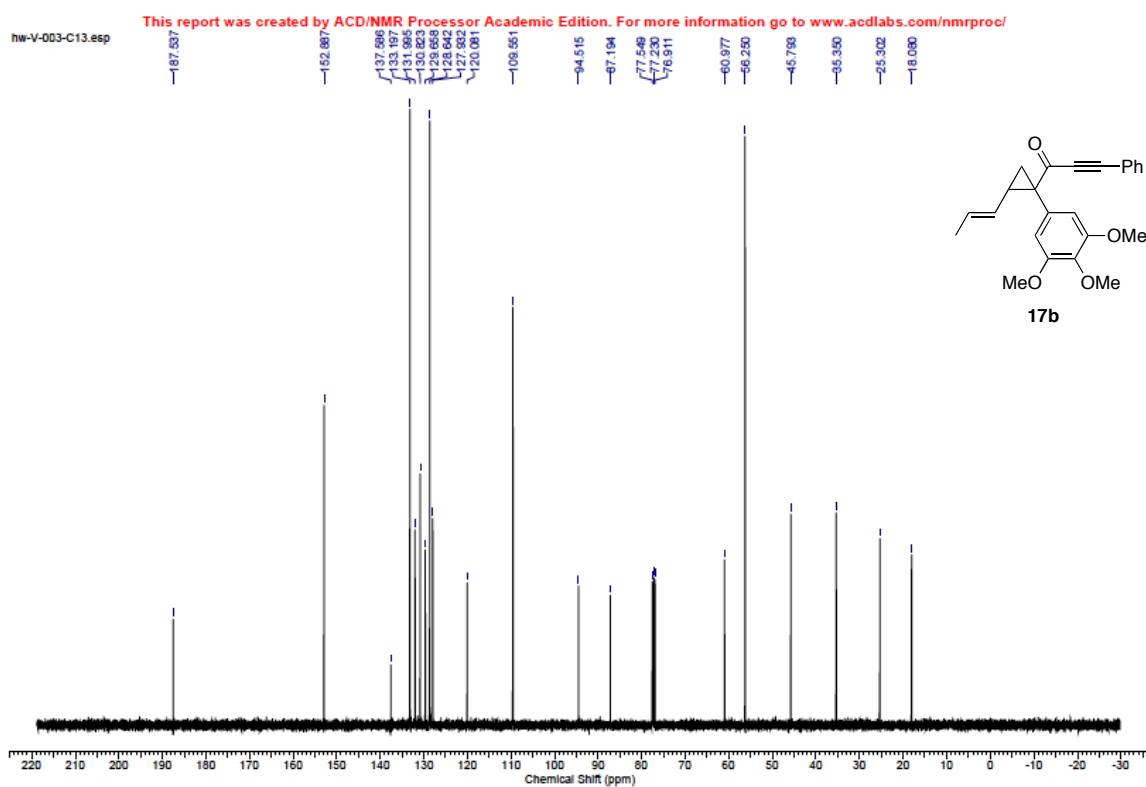
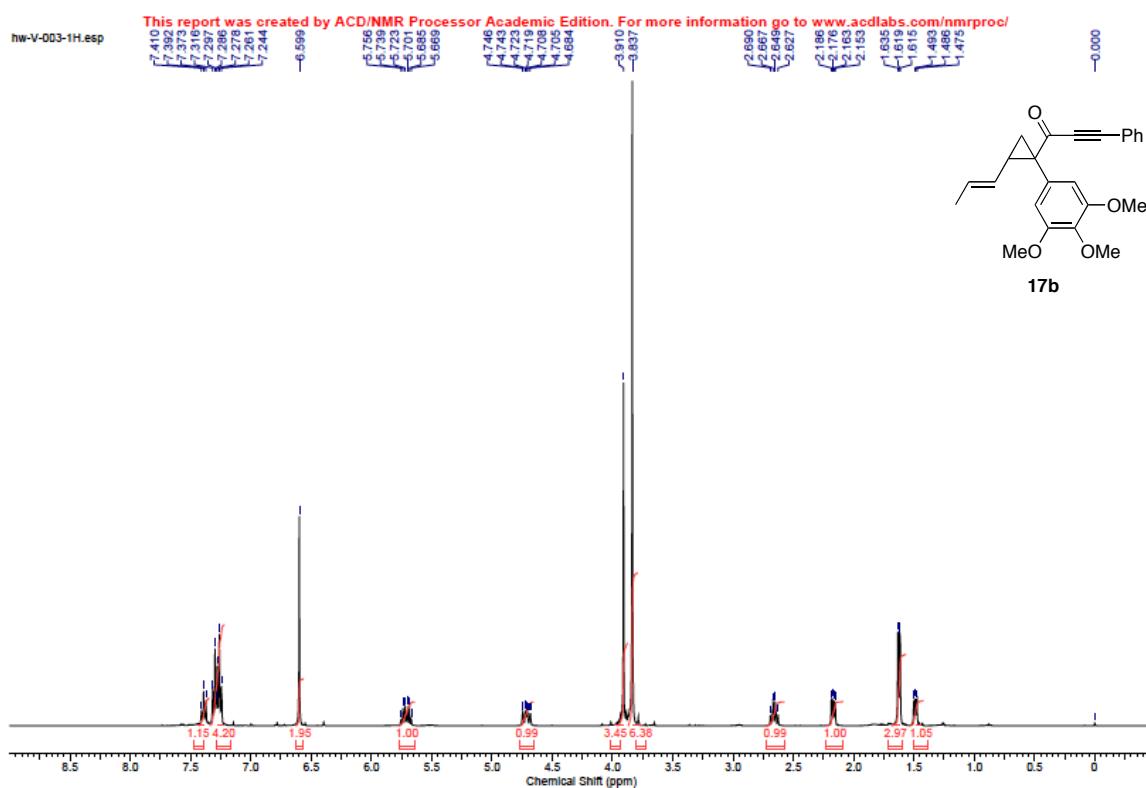
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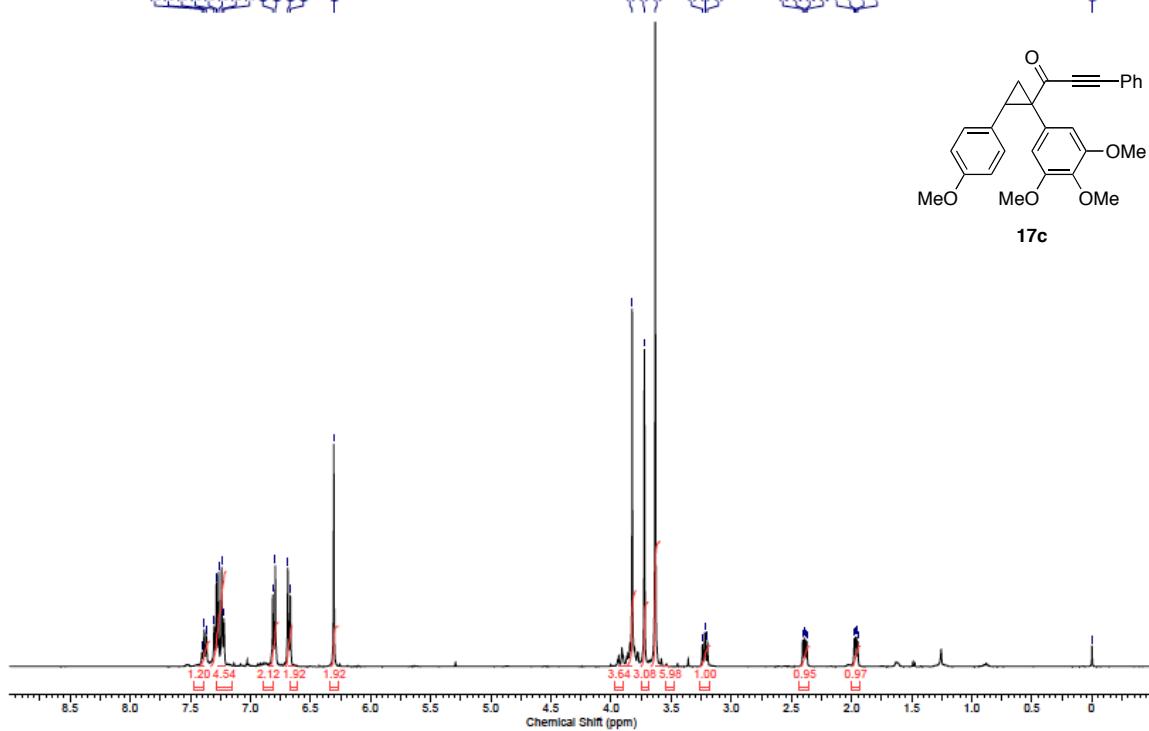


¹H and ¹³C spectra of compound 17a

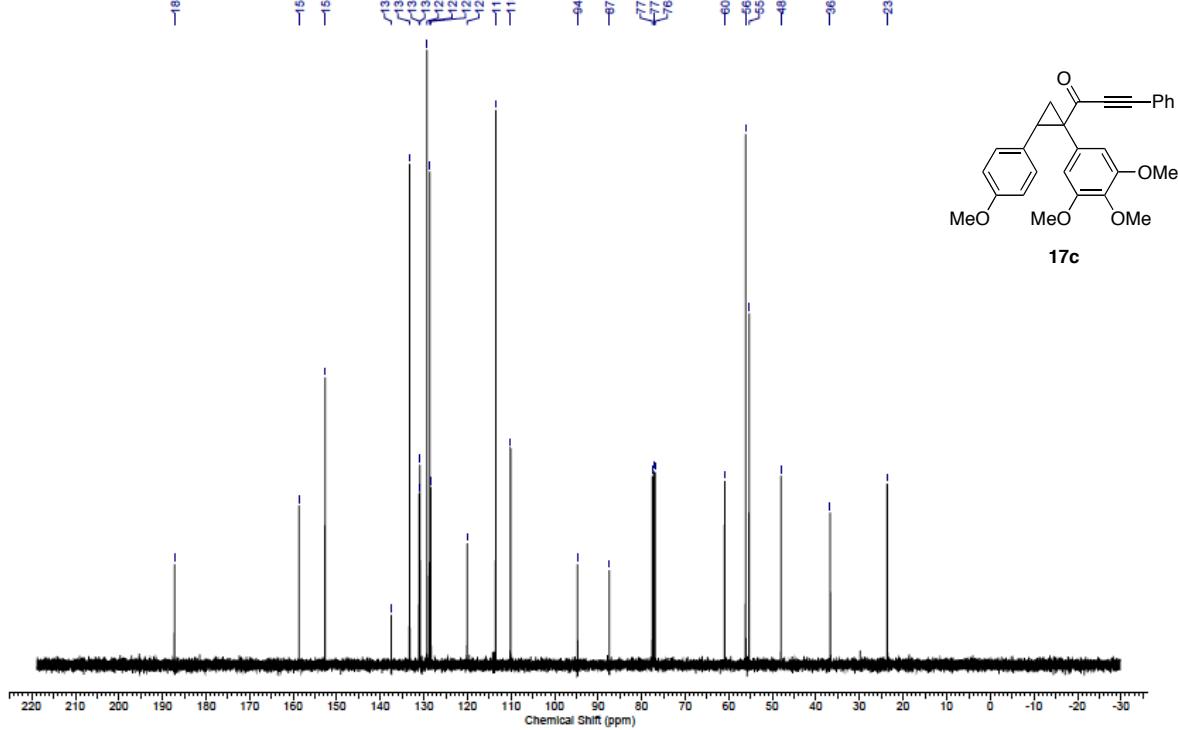


¹H and ¹³C spectra of compound **17b**

hw-IV-119-1H.esp
This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

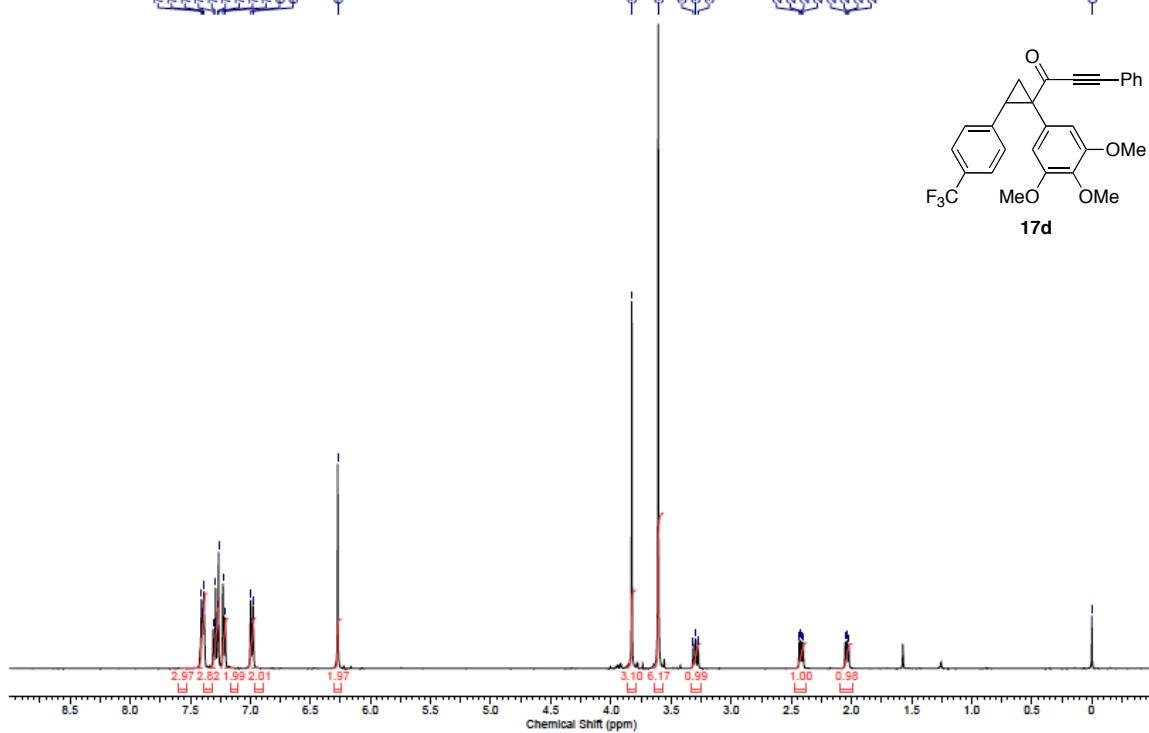


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This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

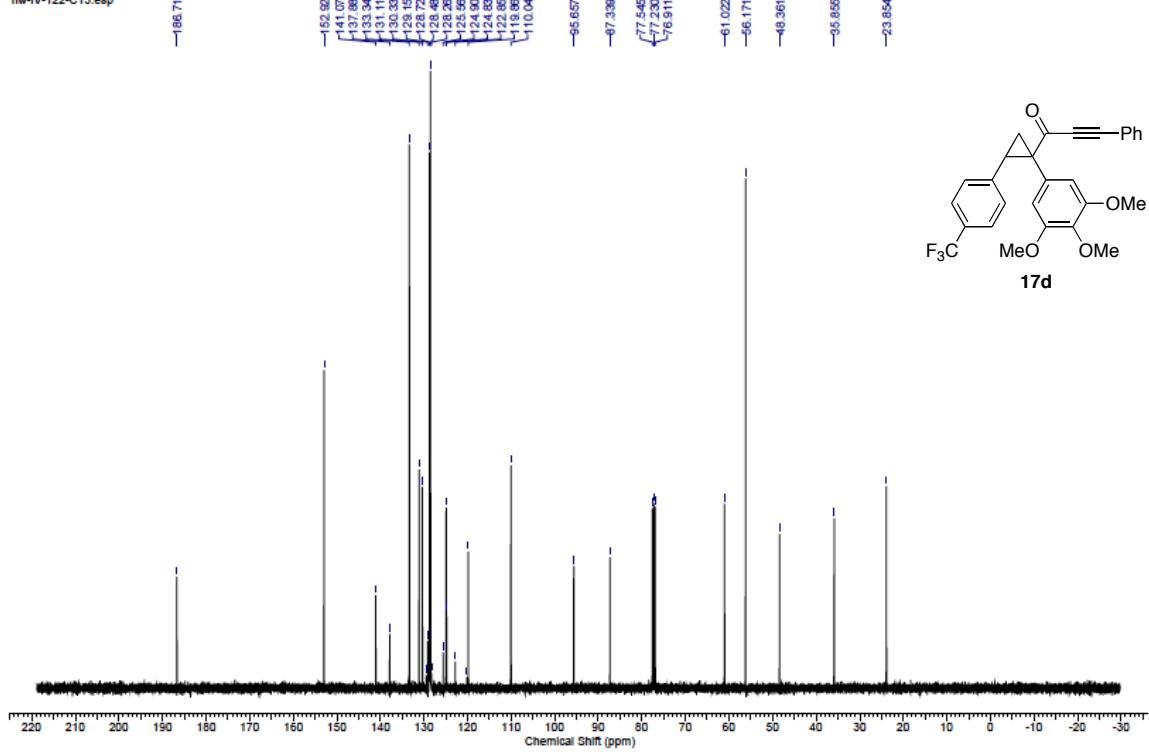


¹H and ¹³C spectra of compound 17c

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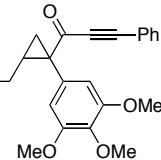
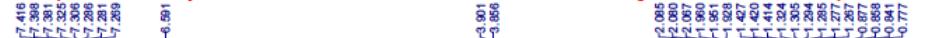
hw-IV-122-C13.esp This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/



¹H and ¹³C spectra of compound 17d

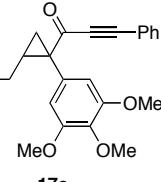
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This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/



hw-IV-105-C13.esp

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/



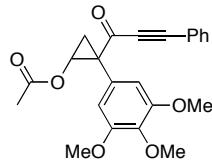
Chemical Shift (ppm)

^1H and ^{13}C spectra of compound 17e

hw-IV-109-1H.esp

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

4.44 4.45 3.95 3.96 3.97 3.99 3.21 2.91 2.92 2.93 2.94 2.25 2.26 0.99 0.98 0.97 0.96 0.95 0.94 0.93 0.92 0.91 0.90 0.89 0.88 0.87 0.86 0.85 0.84 0.83 0.82 0.81 0.80 0.79 0.78 0.77 0.76 0.75 0.74 0.73 0.72 0.71 0.70 0.69 0.68 0.67 0.66 0.65 0.64 0.63 0.62 0.61 0.60 0.59 0.58 0.57 0.56 0.55 0.54 0.53 0.52 0.51 0.50 0.49 0.48 0.47 0.46 0.45 0.44 0.43 0.42 0.41 0.40 0.39 0.38 0.37 0.36 0.35 0.34 0.33 0.32 0.31 0.30 0.29 0.28 0.27 0.26 0.25 0.24 0.23 0.22 0.21 0.20 0.19 0.18 0.17 0.16 0.15 0.14 0.13 0.12 0.11 0.10 0.09 0.08 0.07 0.06 0.05 0.04 0.03 0.02 0.01 0.00



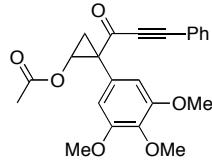
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

Chemical Shift (ppm)

hw-IV-109-C13.esp

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

-185.699 -171.181 -153.194 -137.923 -133.446 -131.077 -129.006 -128.887 -119.781 -108.804 -96.725 -87.206 -77.549 -77.230 -76.911 -60.973 -60.587 -60.250 -44.473 -22.143 -20.886

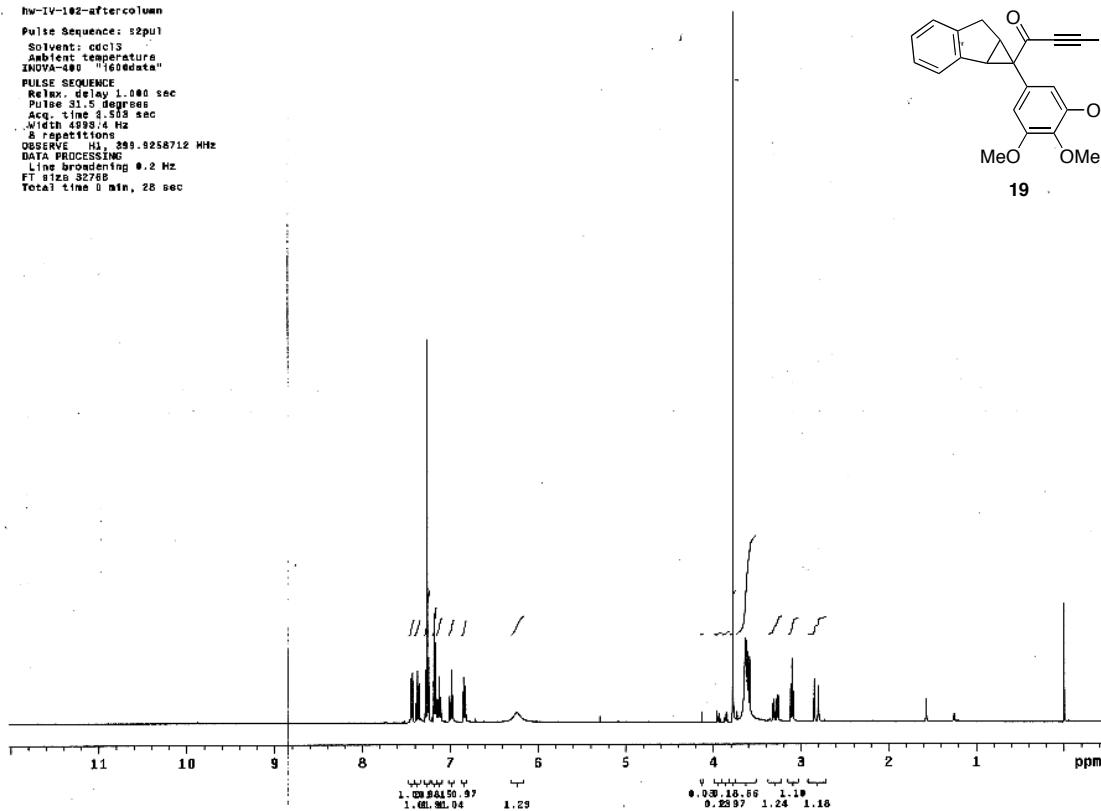
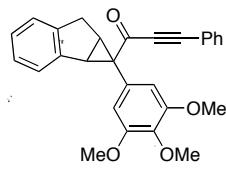


220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30

Chemical Shift (ppm)

¹H and ¹³C spectra of compound 17f

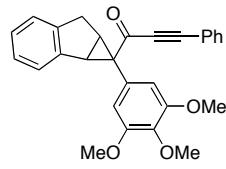
hw-IV-102-aftercolumn
 Pulse Sequence: 52pul
 Solvent: CDCl₃
 Ambient Temperature
 INOVA-400 "1600data"
 PULSE SEQUENCE
 Relax. delay 1.00 sec
 Pulse 31.5 degrees
 Acq. time 4.0 sec
 Width 4334.4 Hz
 8 repetitions
 OBSERVE: H1 299.9258712 MHz
 DATA ACQUISITION
 Line broadening 0.2 Hz
 FT size 32768
 Total time 0 min, 28 sec



This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

hw-IV-102-C13.esp

187.086
 152.696
 143.742
 141.720
 137.246
 133.322
 133.872
 128.686
 128.550
 126.919
 126.393
 124.932
 124.884
 120.107
 110.416
 -95.046
 -87.760
 -77.230
 -77.549
 -70.911
 -69.905
 -66.169
 -49.639
 -44.905
 -35.510
 -34.034

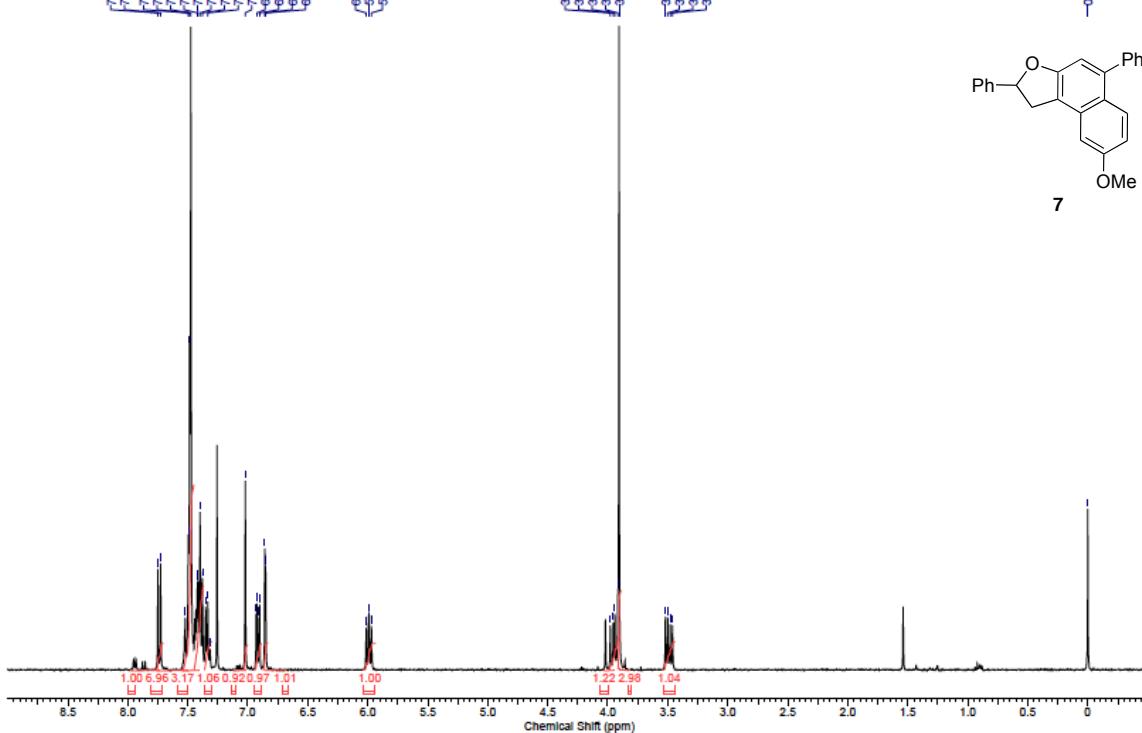


220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30

Chemical Shift (ppm)

¹H and ¹³C spectra of compound 19

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/
hw-II-129-B-1H.esp



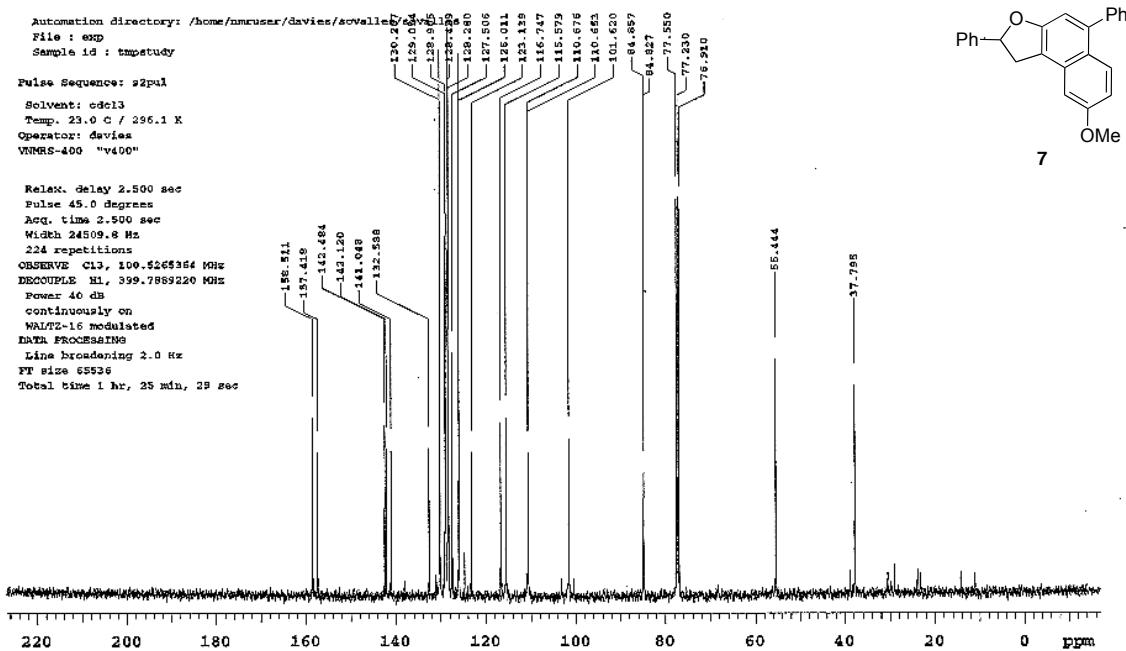
hw-129-aftercolumn-B-c13

Automation directory: /home/nmruser/davies/acvalley
File : exp
Sample id : tmsstudy

Pulse Sequence: s2pul

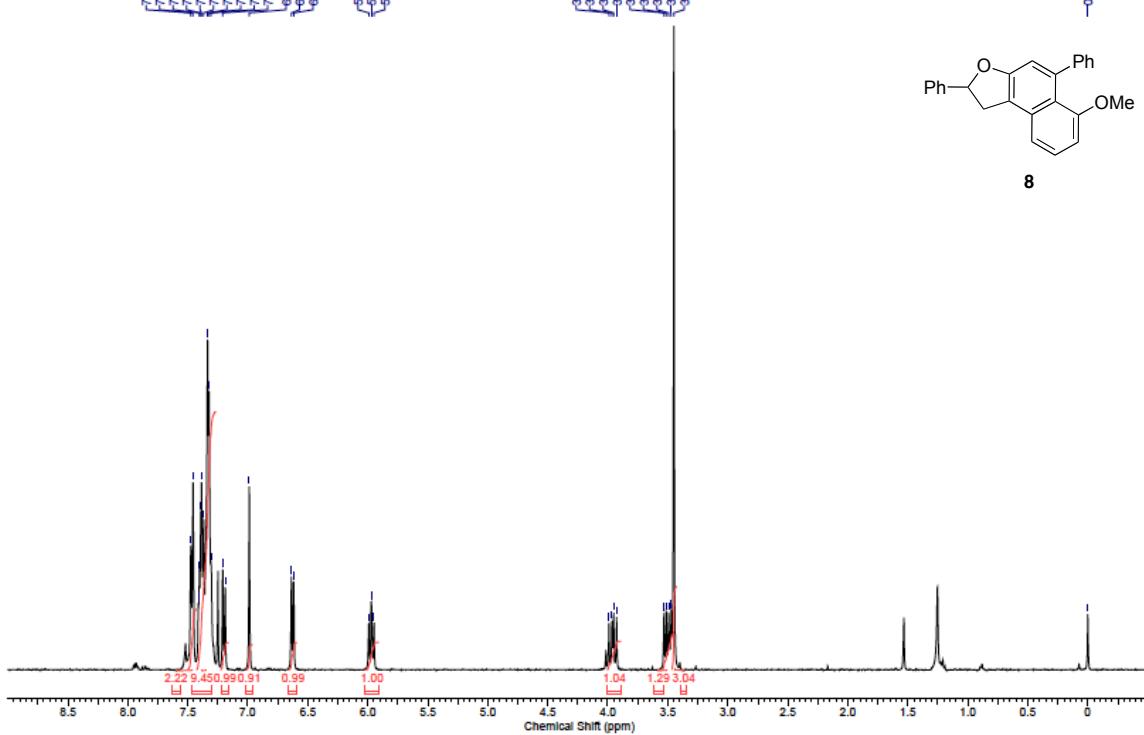
Solvent: cdc13
Temp. 23.0 C / 296.1 K
Operator: davies
VNMRs-400 "v400"

Relax. delay 2.500 sec
Pulse 45.0 degrees
Aq. time 2.500 sec
Width 24509.8 Hz
224 repetitions
OBSERVE CL3, 100.5265364 MHz
DECOUPLE HL, 399.7869220 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 25 min, 29 sec

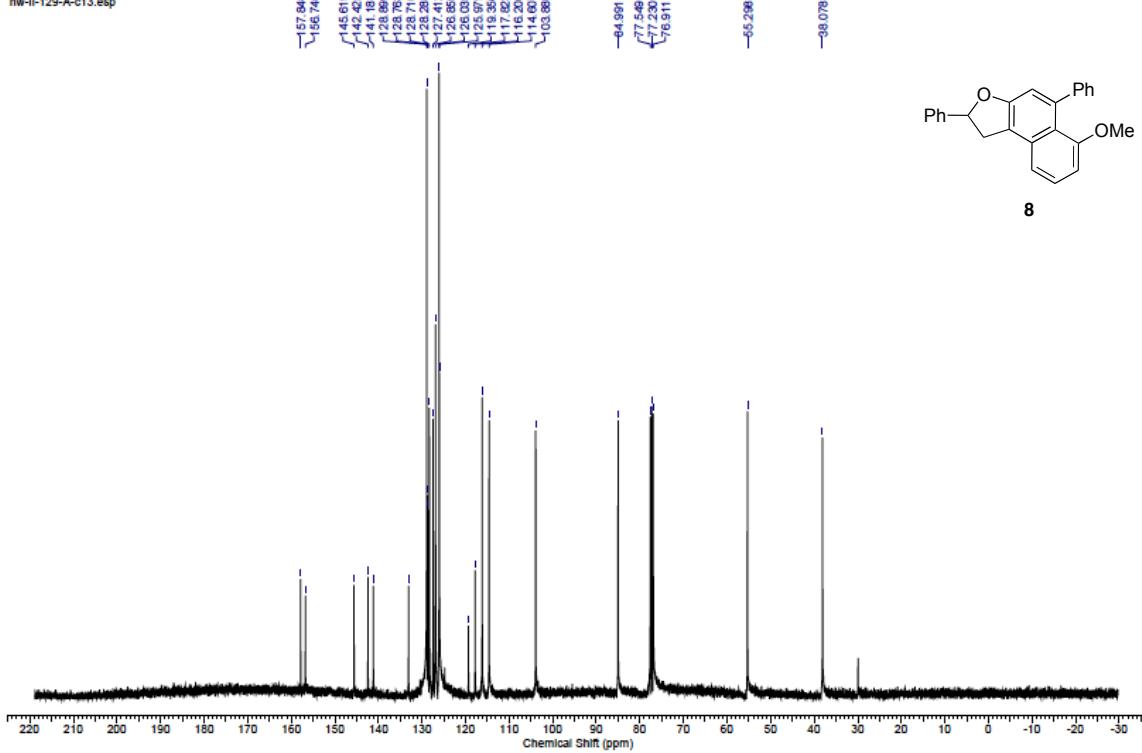


¹H and ¹³C spectra of compound 7

hw-II-129-A-1H.esp This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

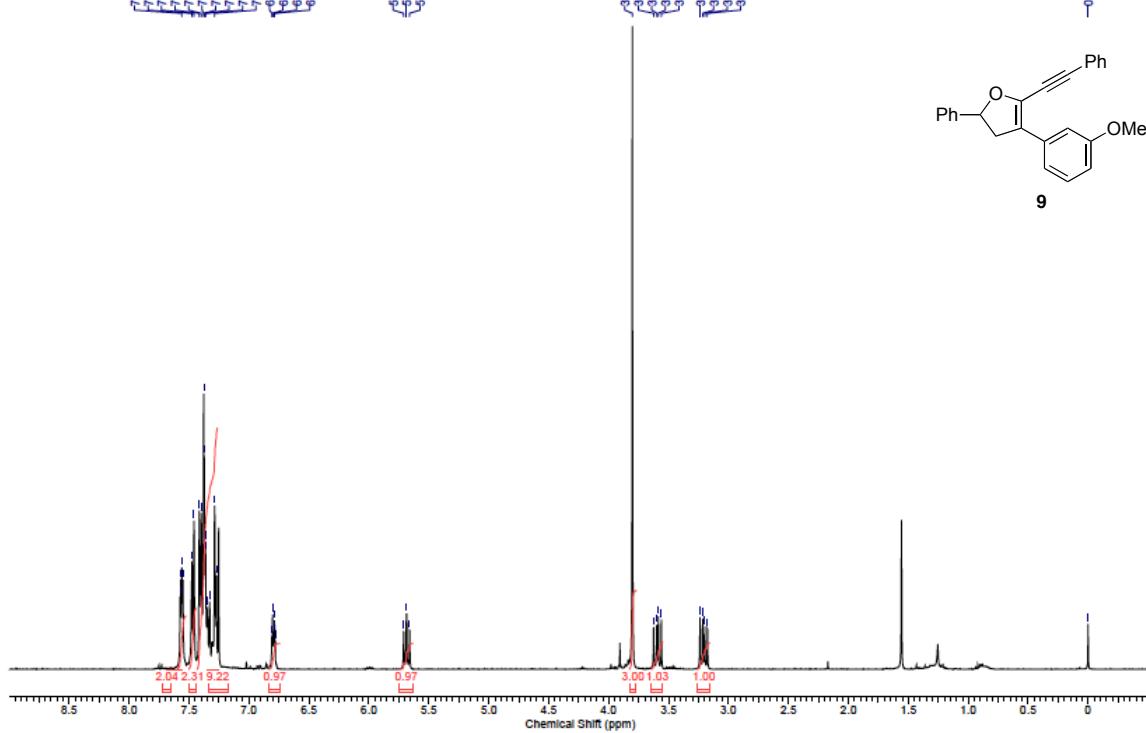


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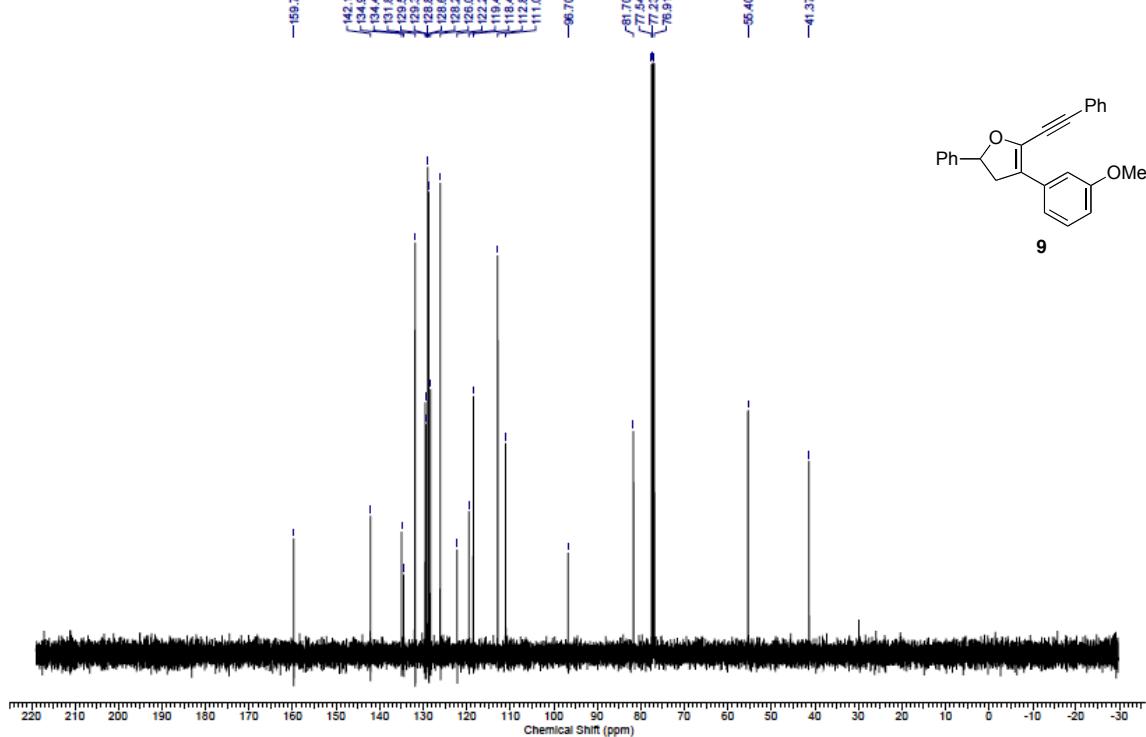


¹H and ¹³C spectra of compound 8

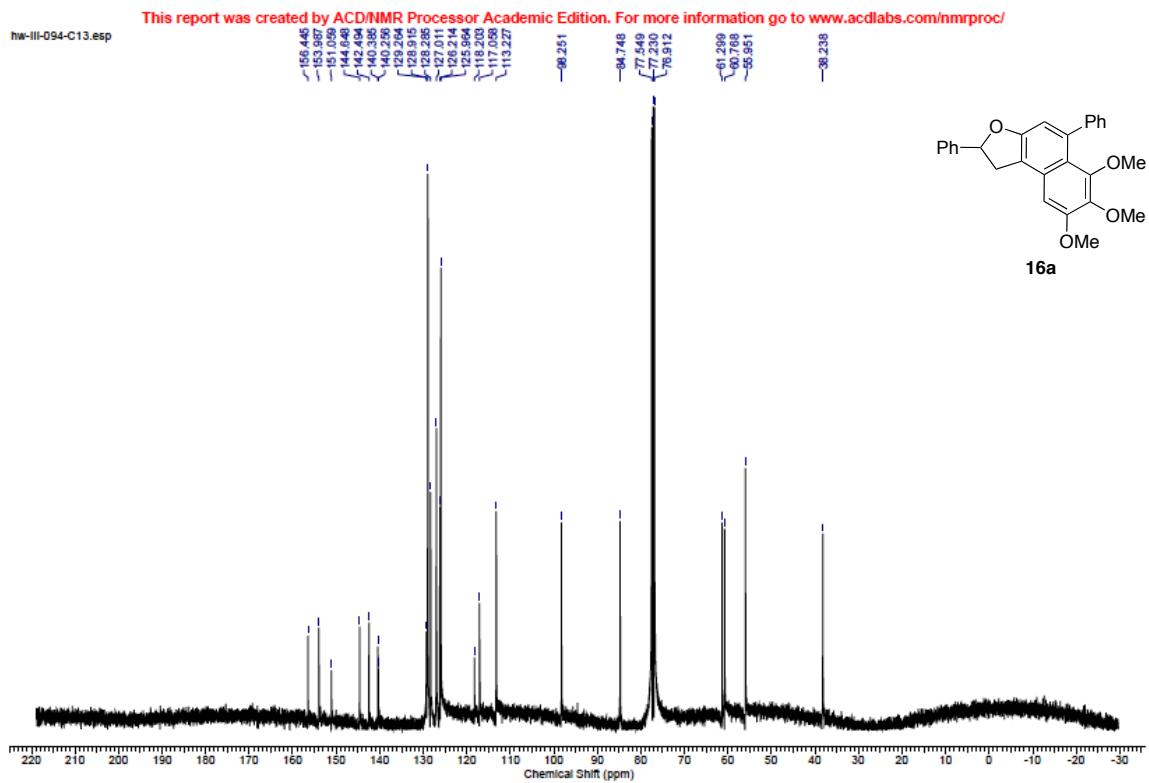
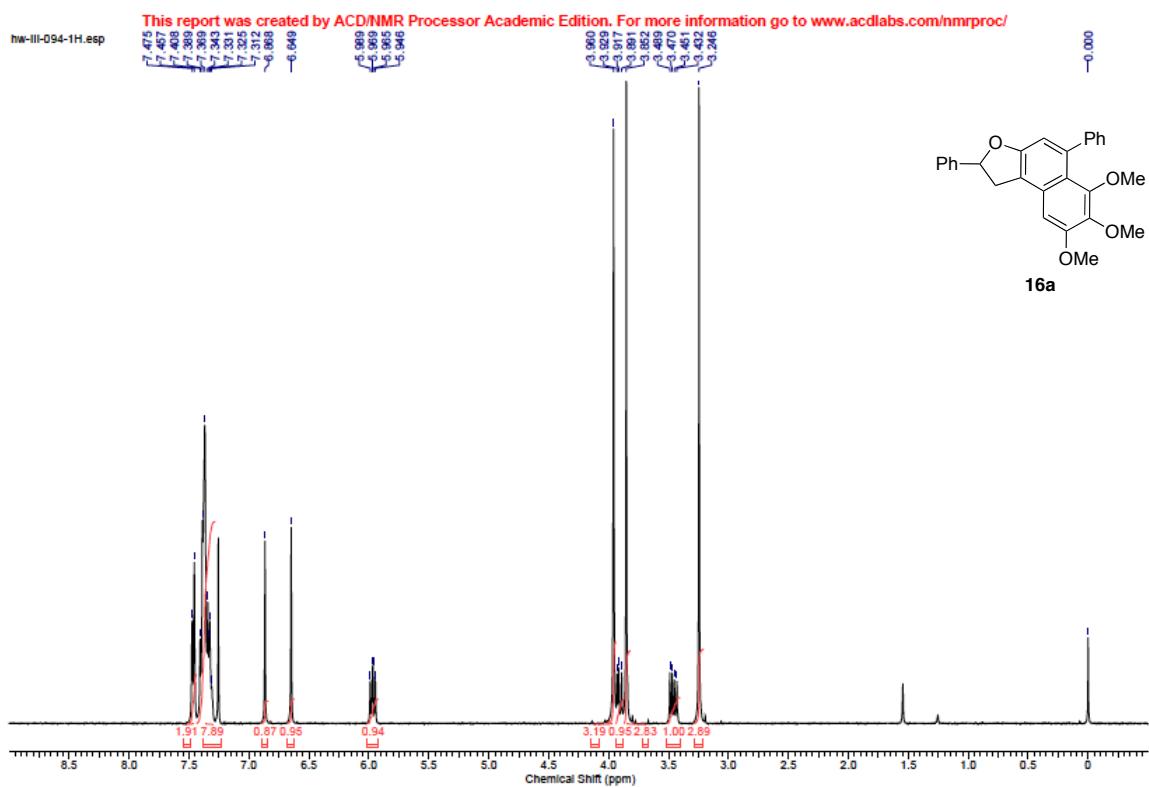
hw-V-043-1H.esp This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/



hw-IV-89-C13.esp This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

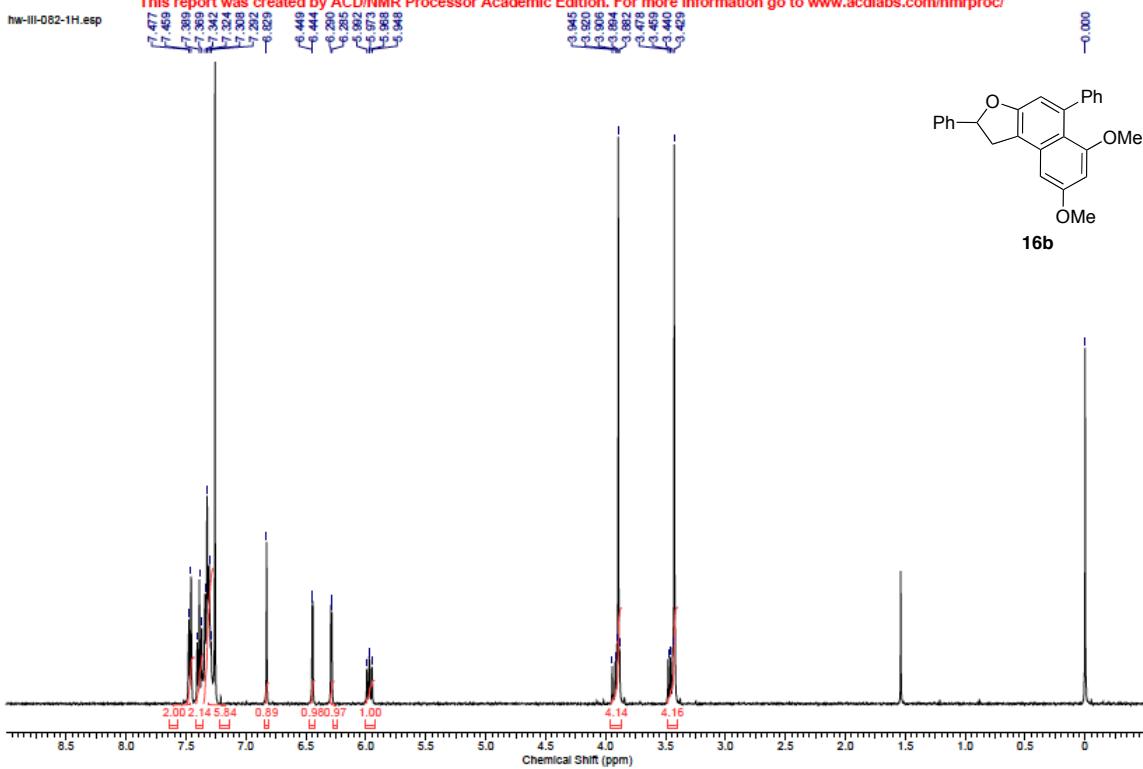


¹H and ¹³C spectra of compound 9

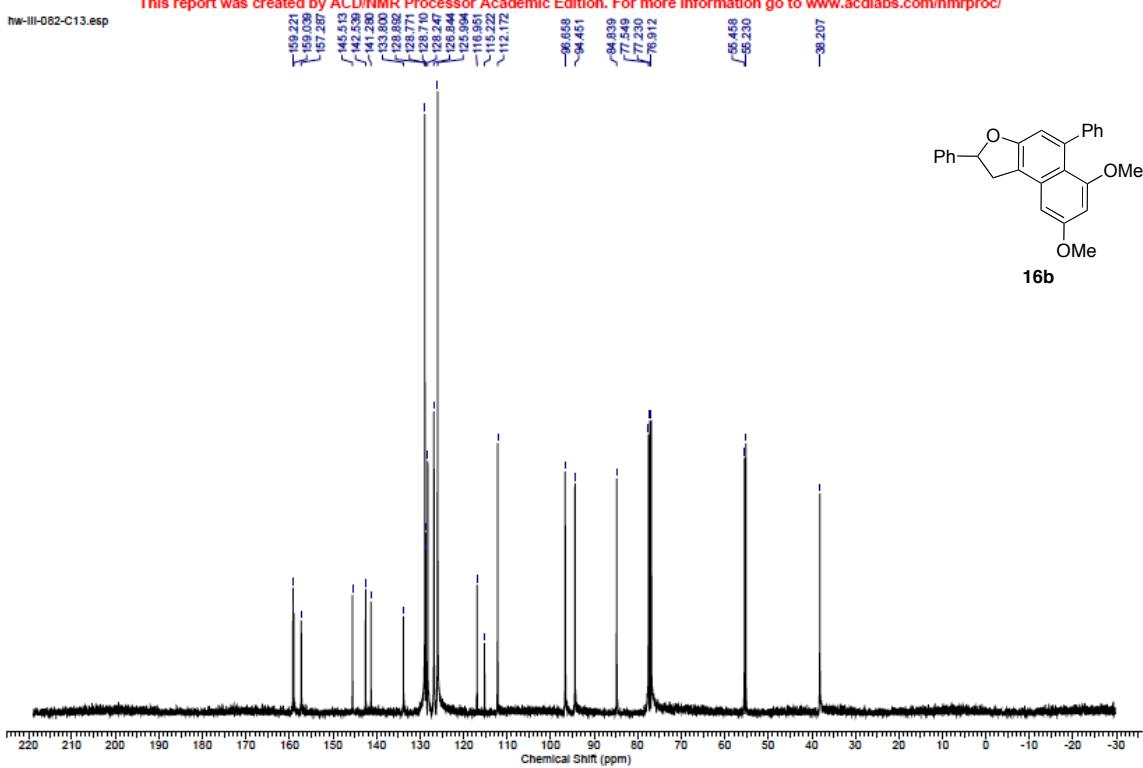


¹H and ¹³C spectra of compound 16a

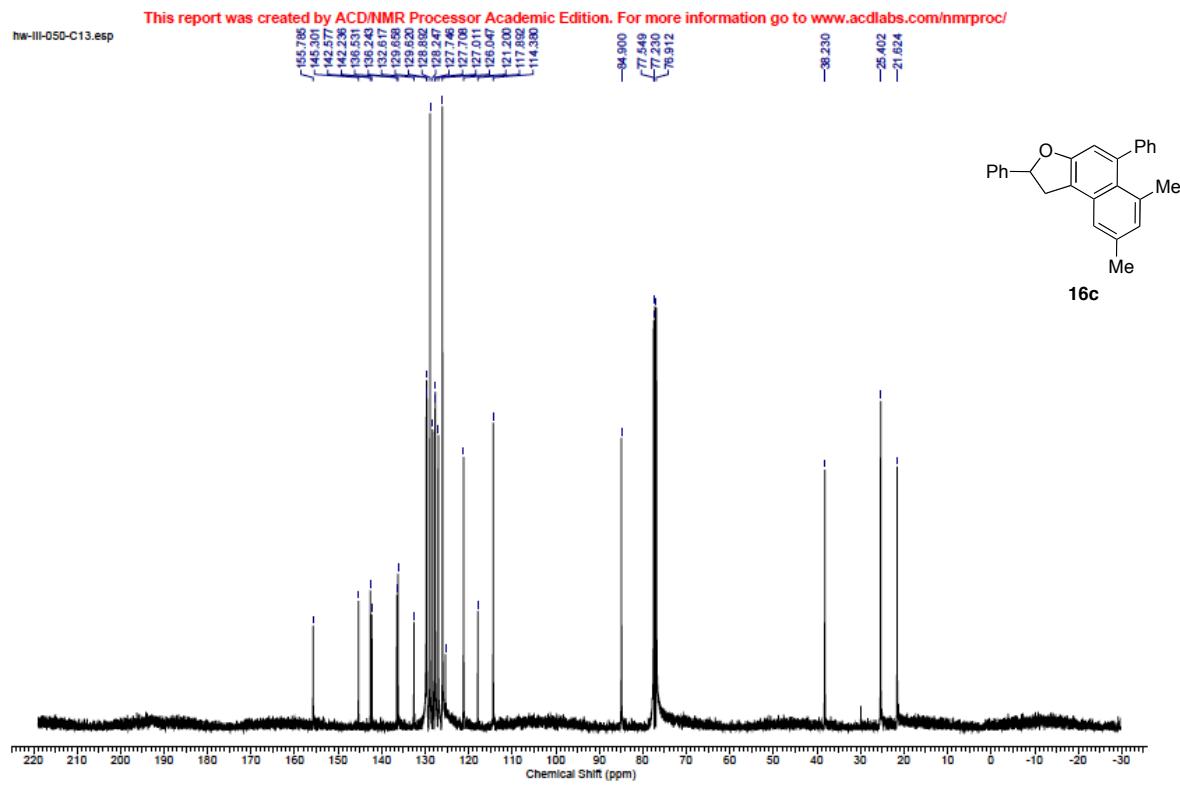
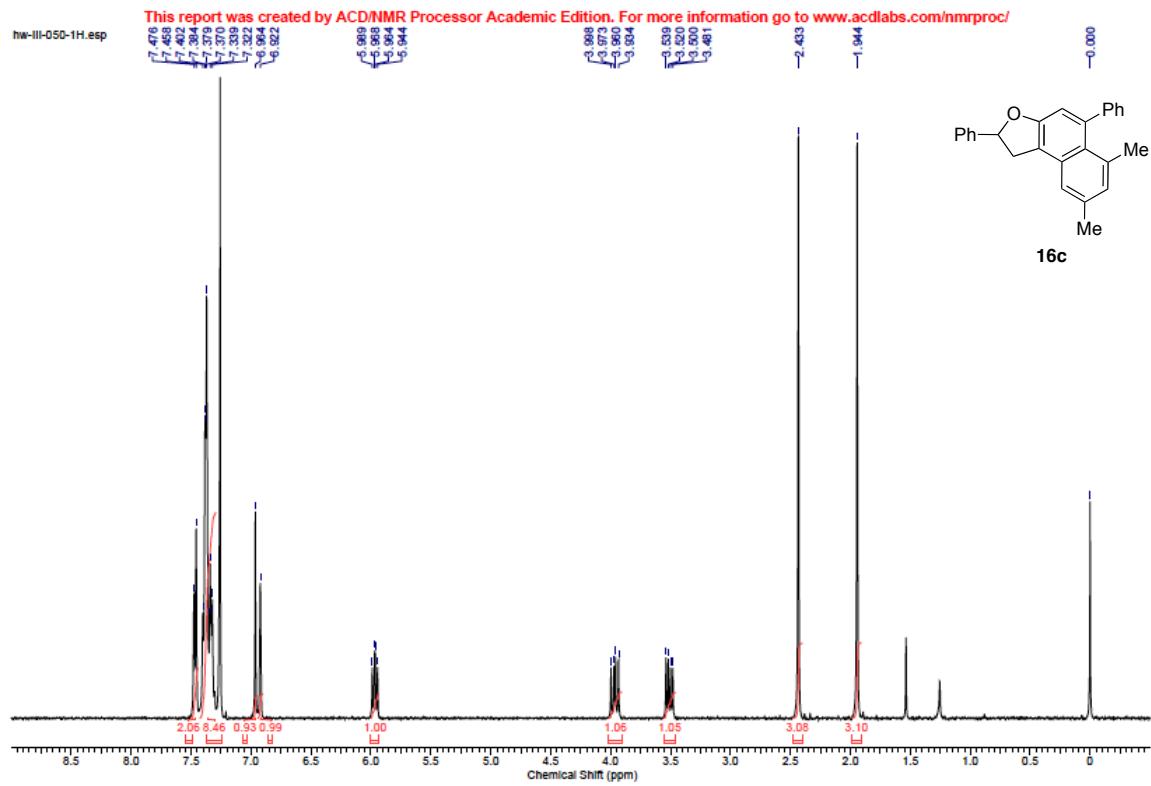
This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/



This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

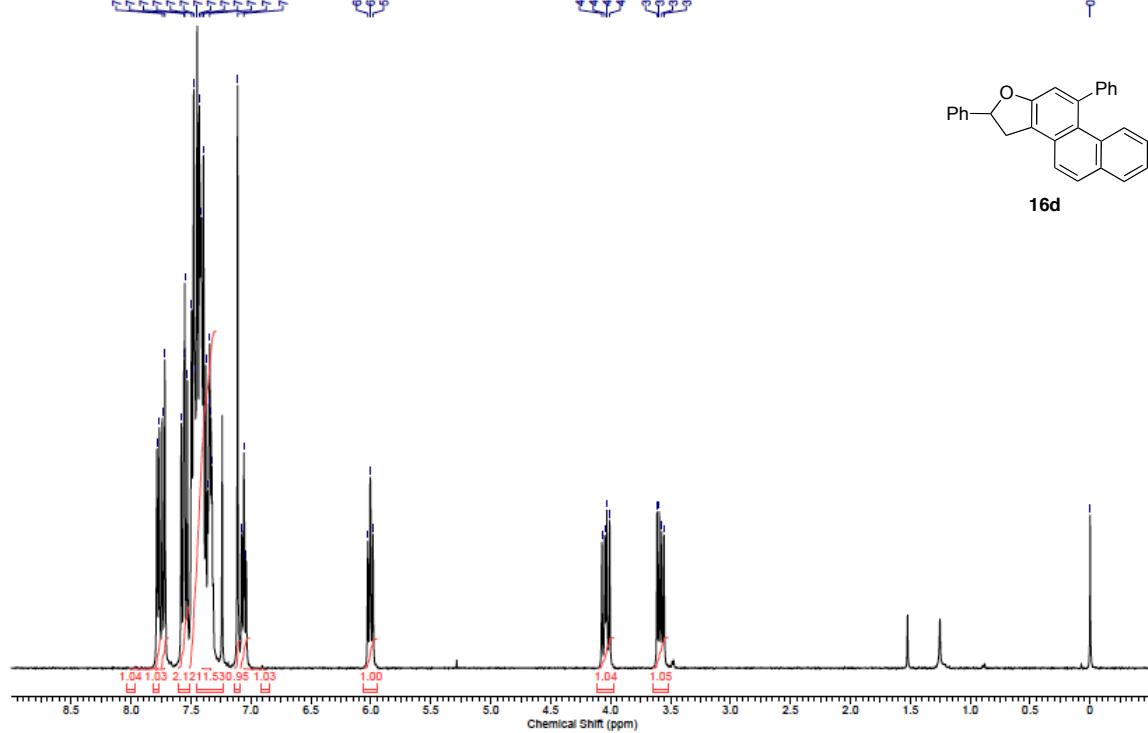


¹H and ¹³C spectra of compound **16b**

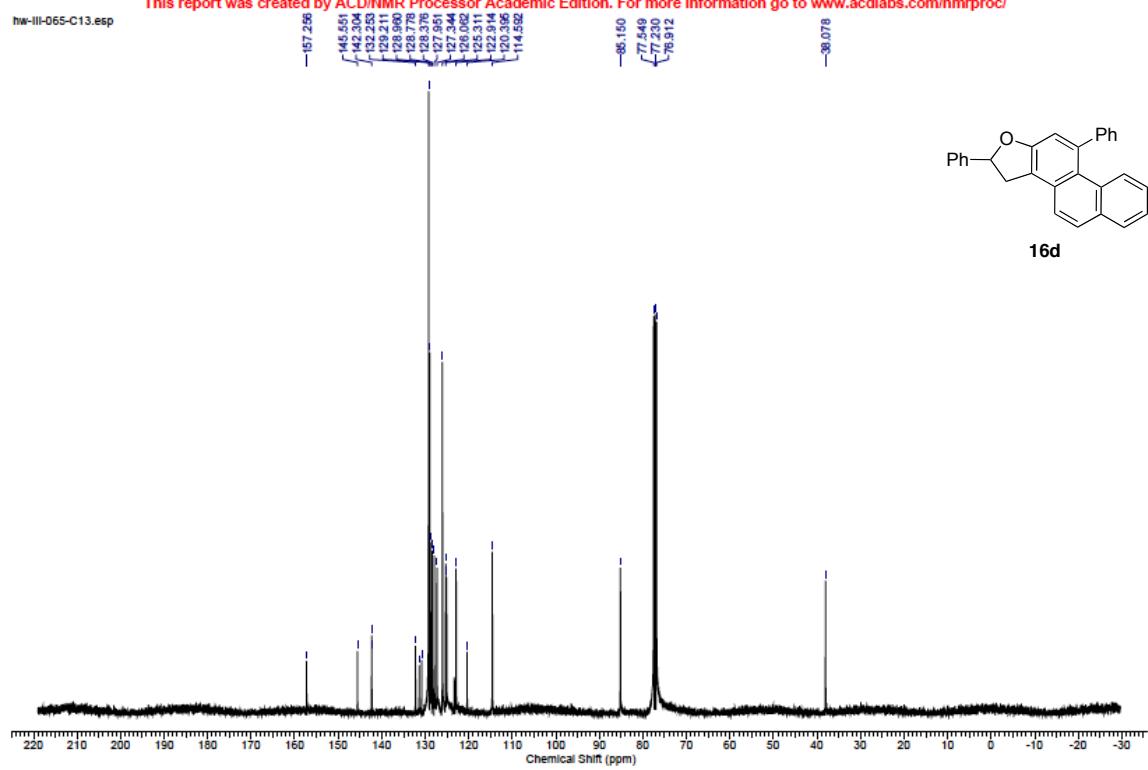


¹H and ¹³C spectra of compound 16c

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/
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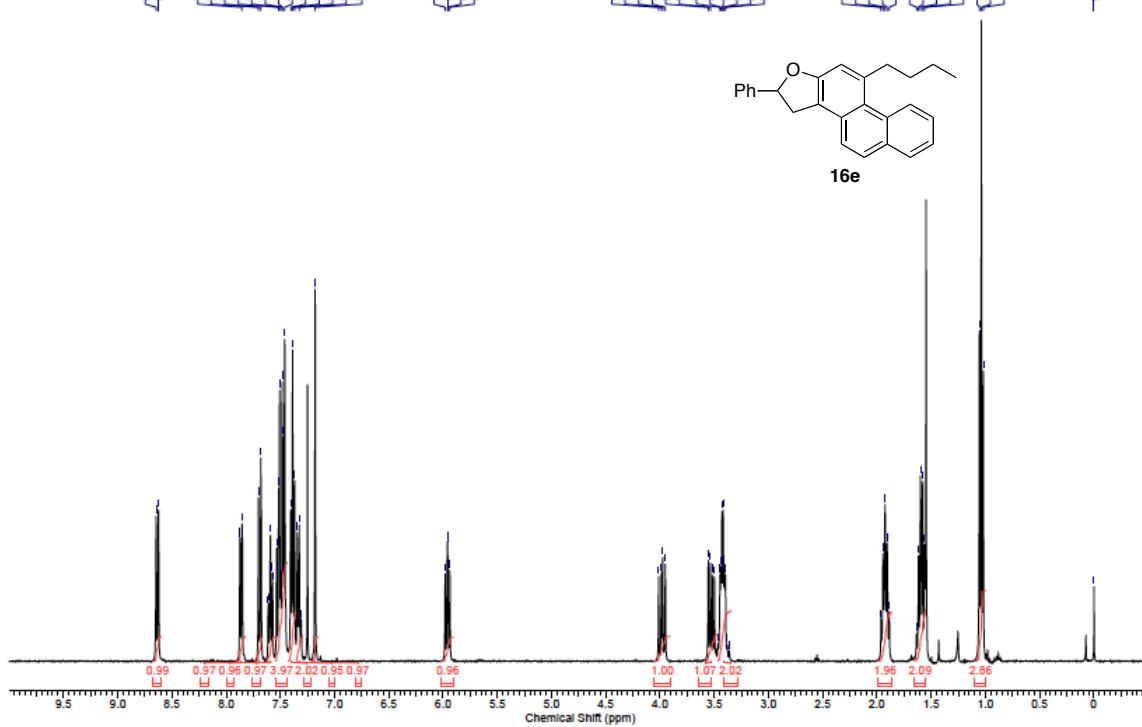


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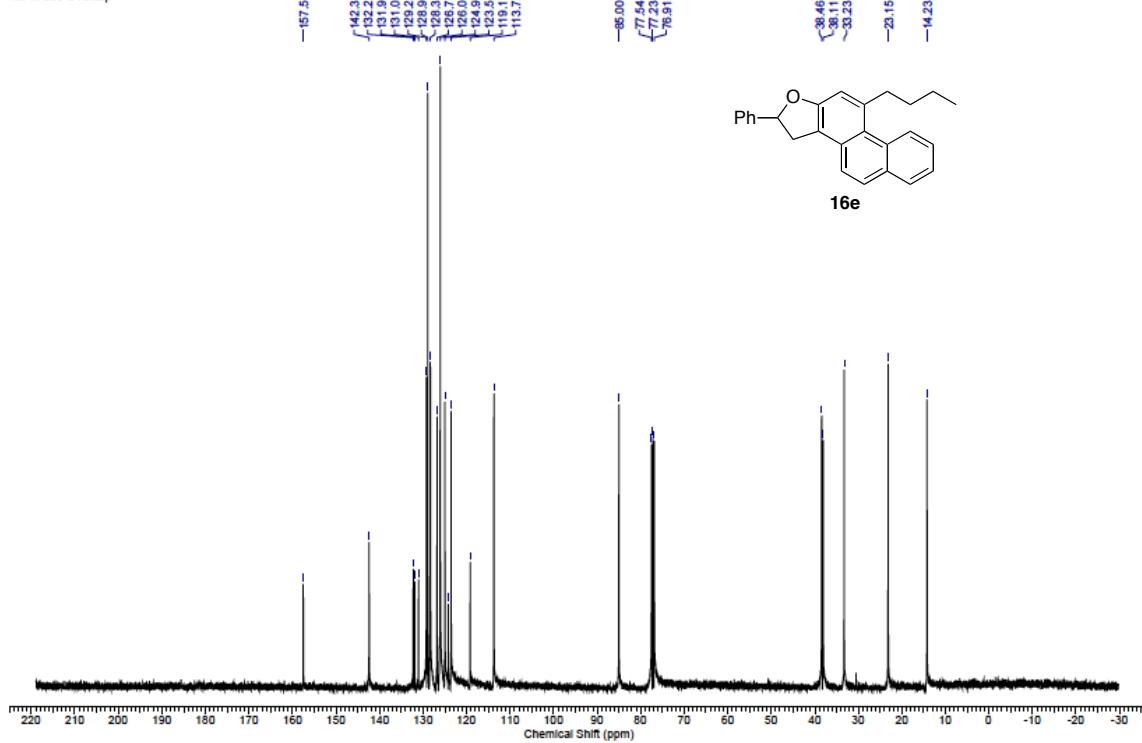


¹H and ¹³C spectra of compound 16d

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/
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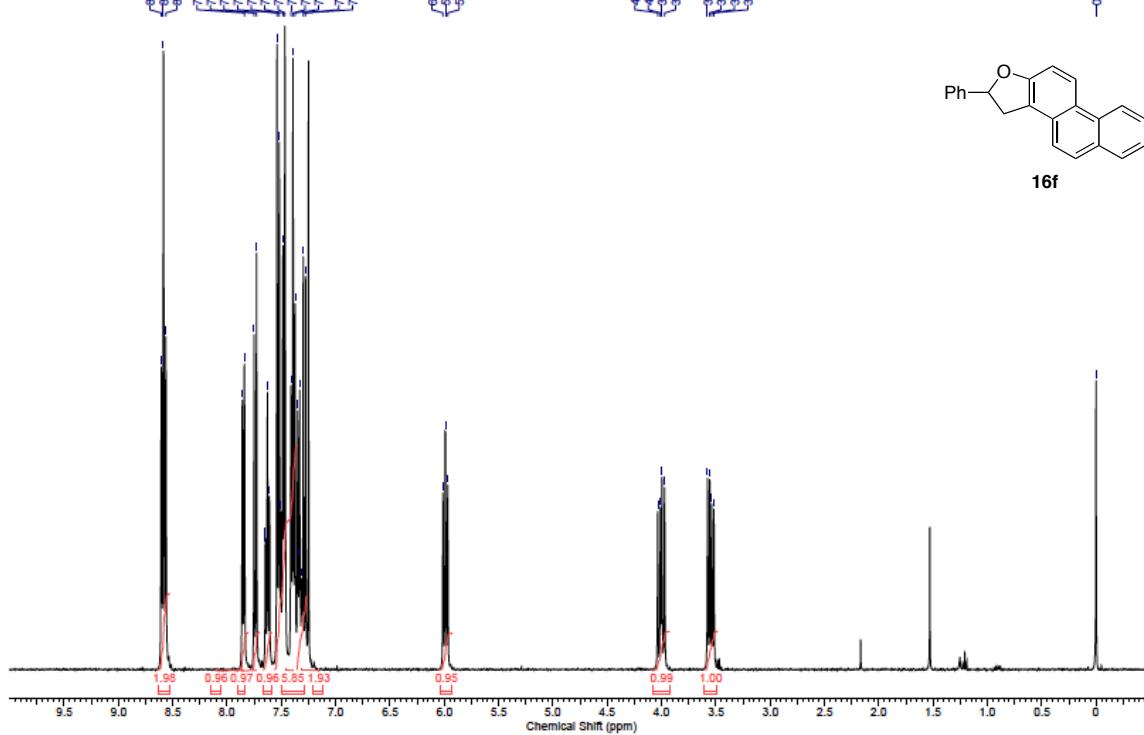


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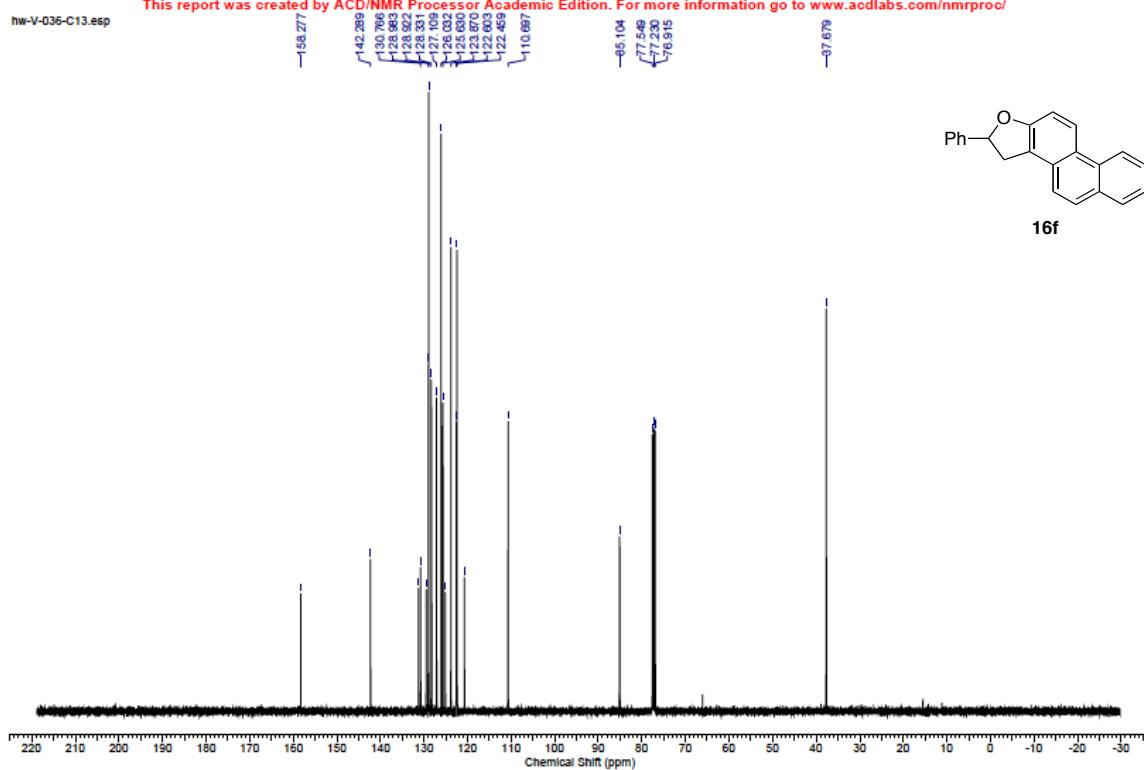


¹H and ¹³C spectra of compound 16e

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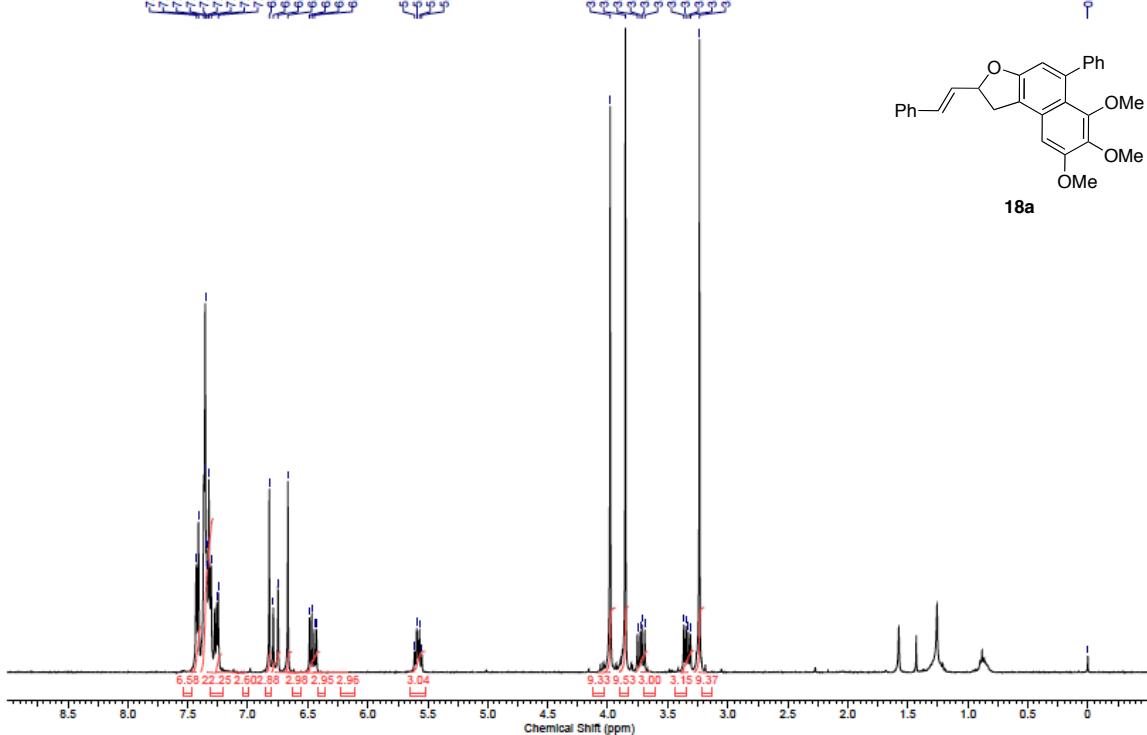


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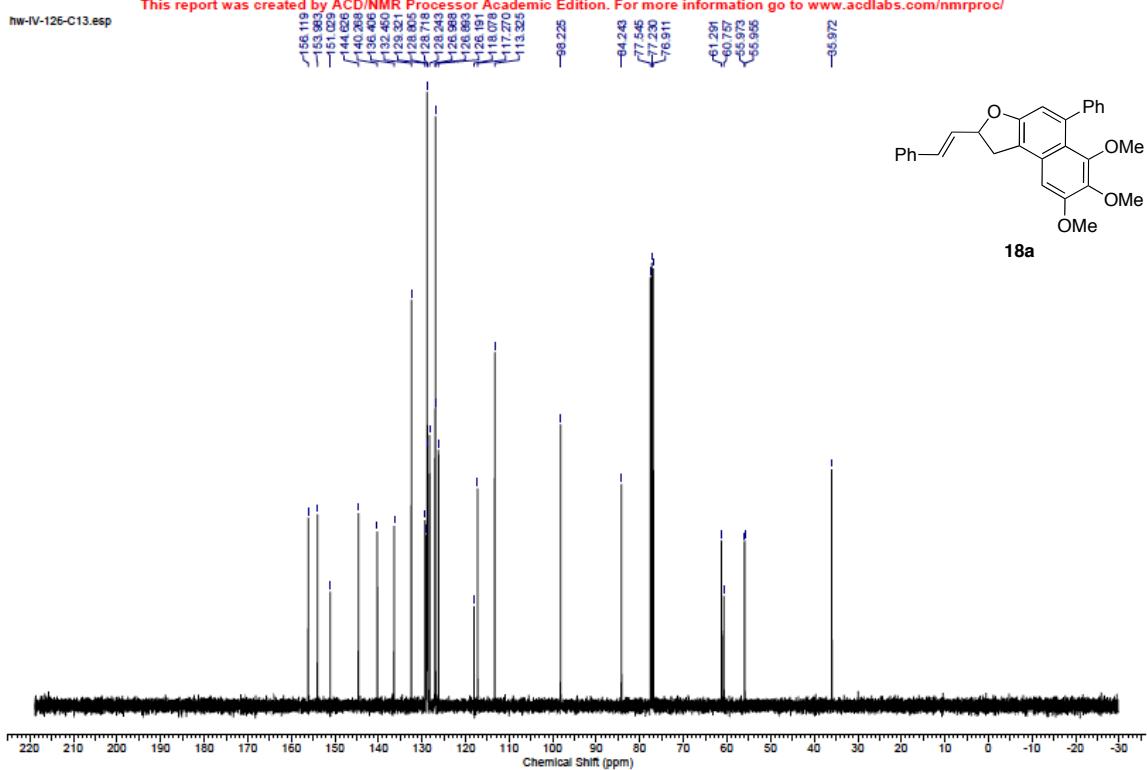


¹H and ¹³C spectra of compound 16f

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This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

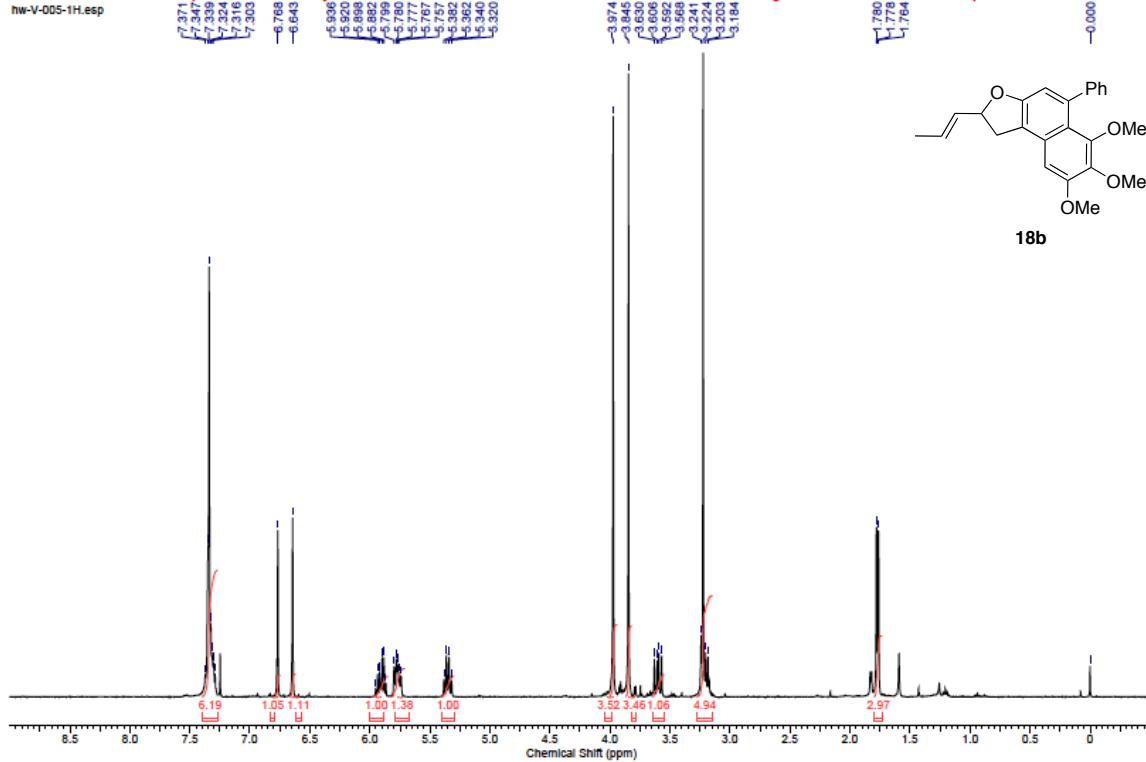


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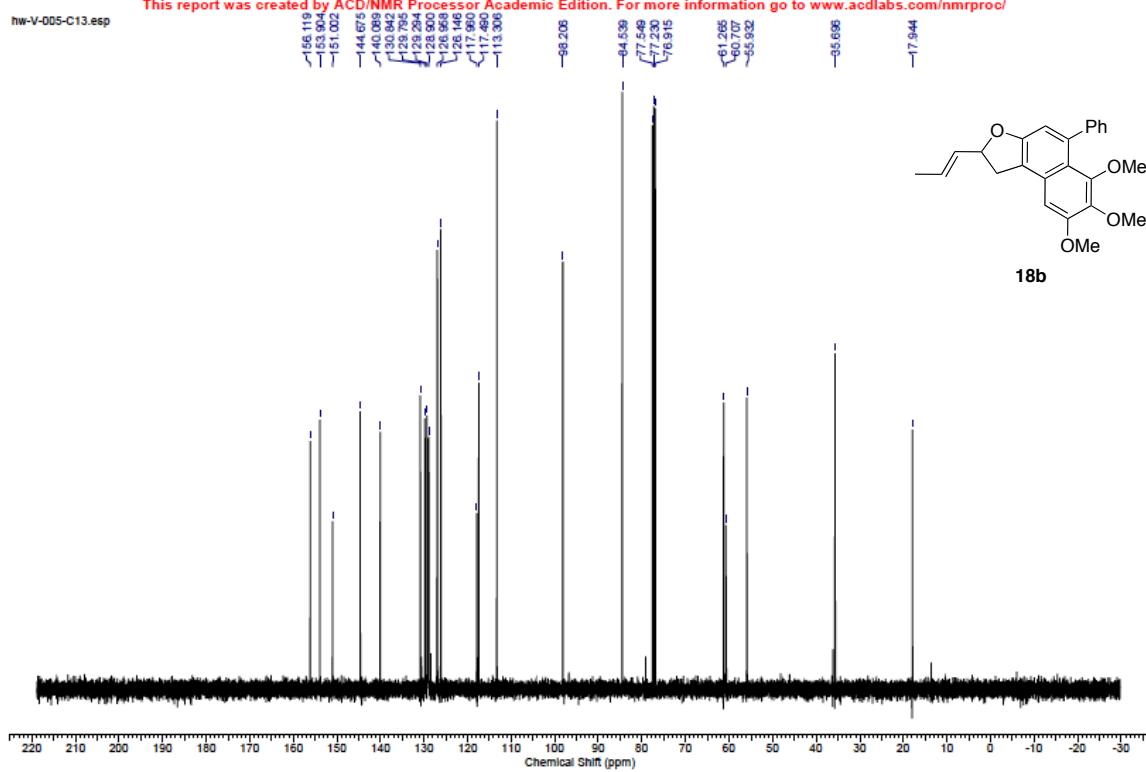


¹H and ¹³C spectra of compound 18a

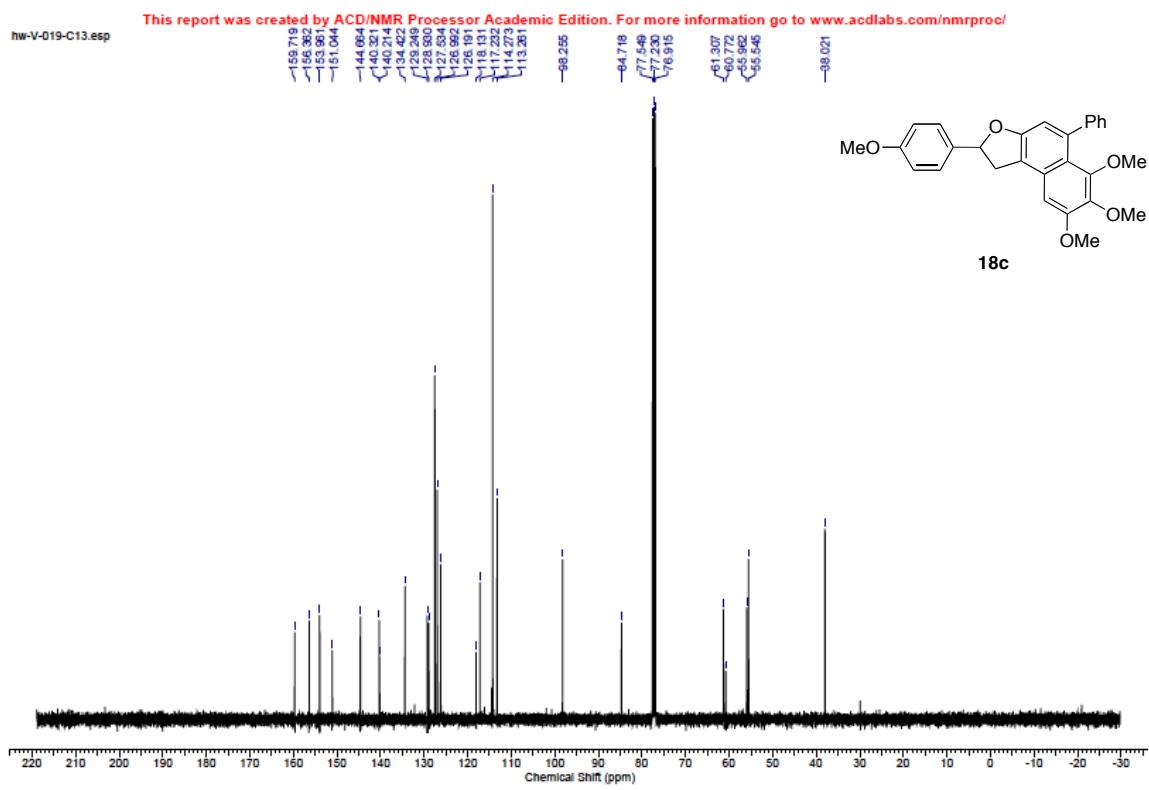
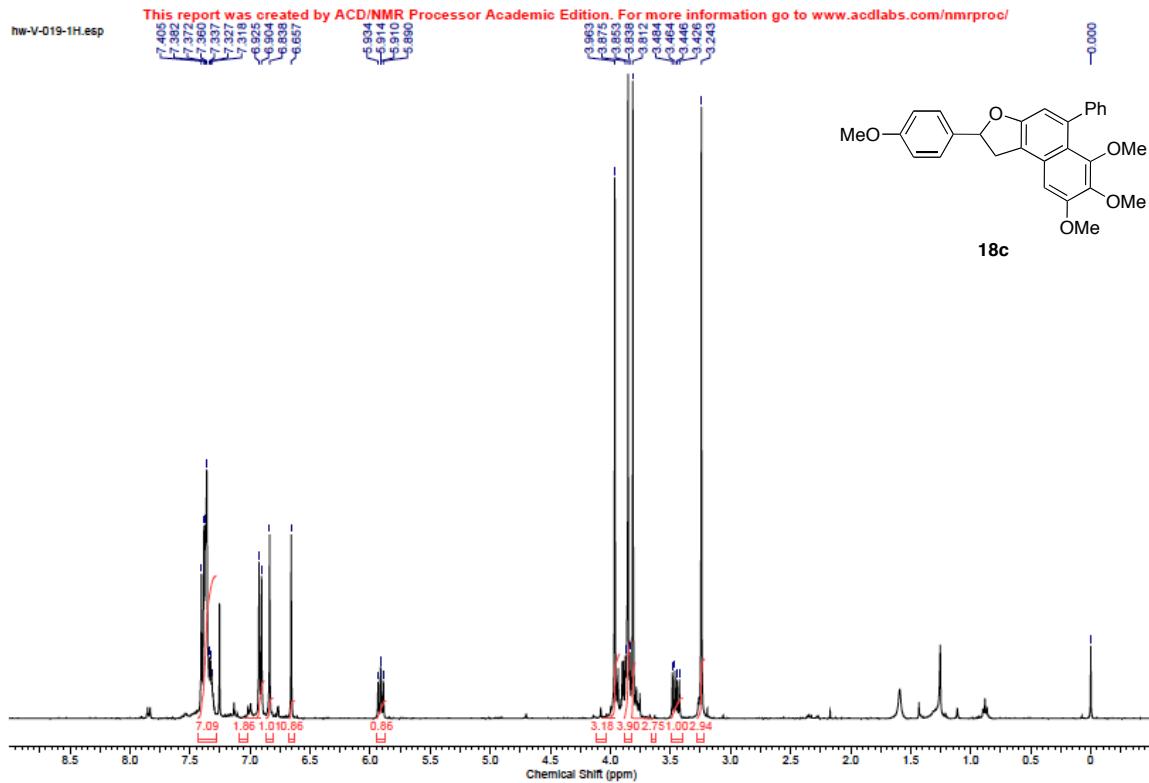
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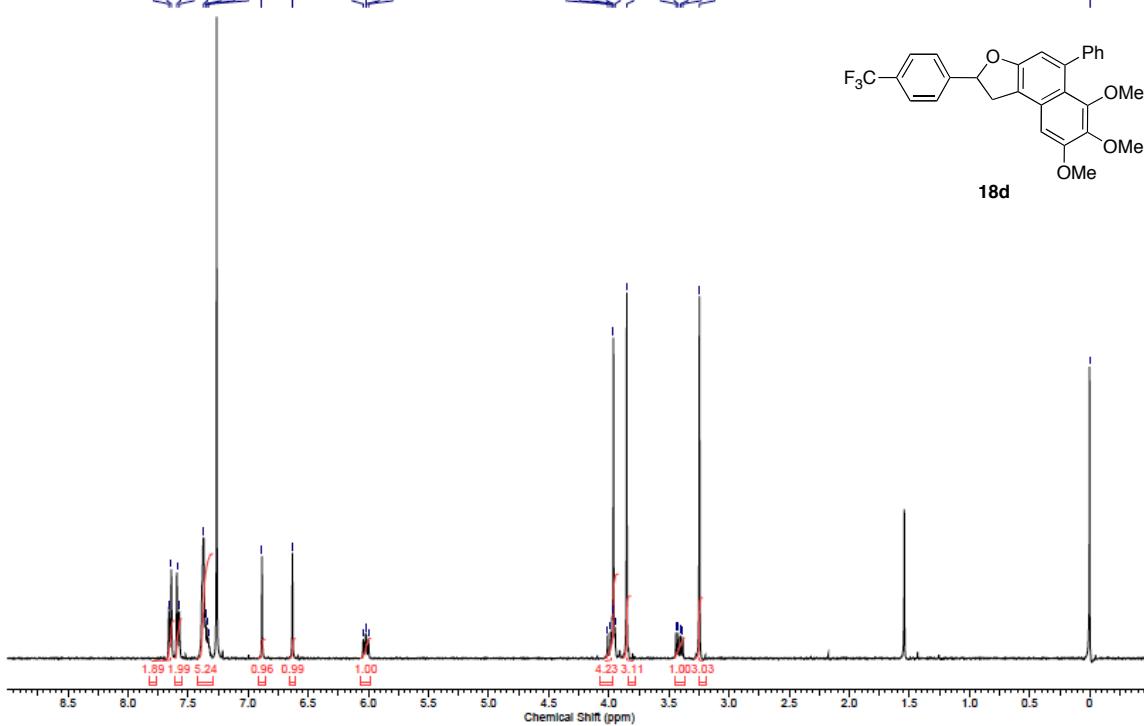


¹H and ¹³C spectra of compound 18b

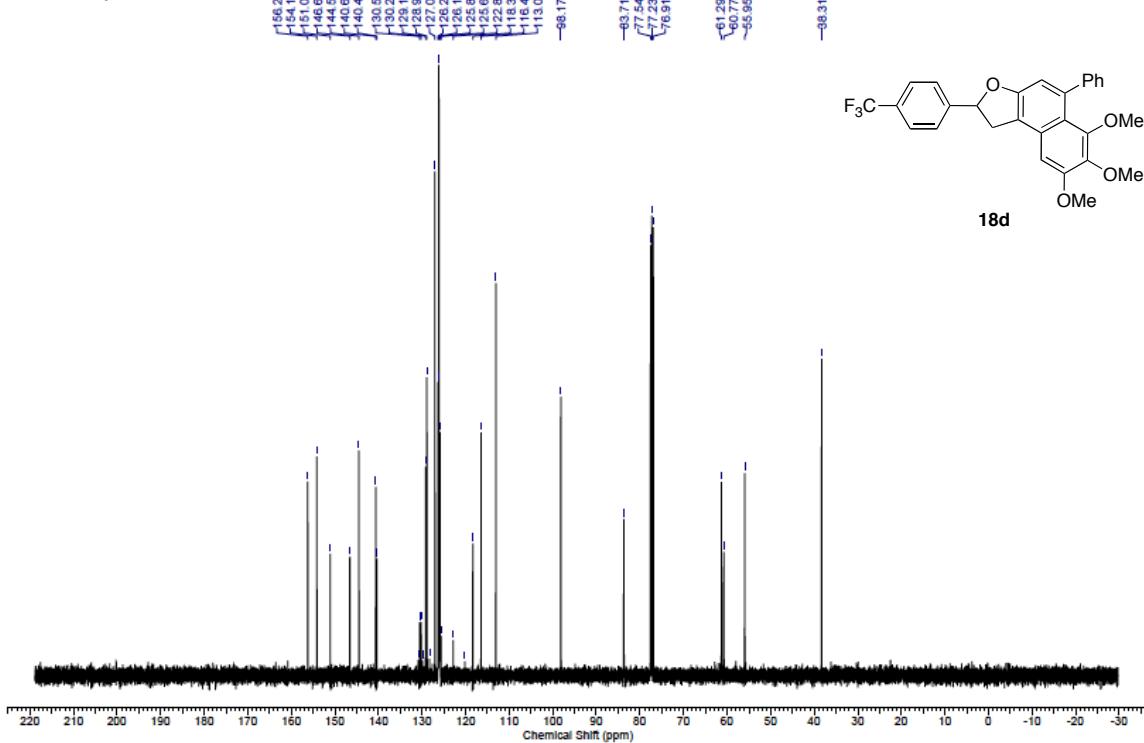


¹H and ¹³C spectra of compound 18c

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/
hw-IV-125-1H.esp



This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/
hw-IV-125-C13.esp



¹H and ¹³C spectra of compound 18d

hw-IV-117-1H.esp

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

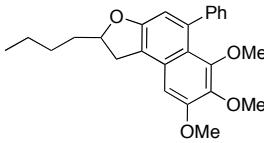
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0.651

7.016
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4.960
4.941

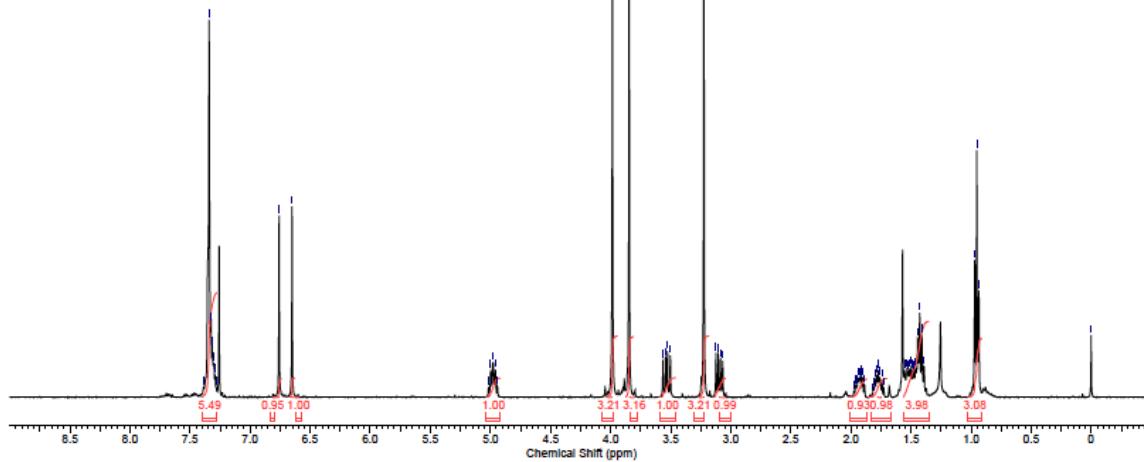
-0.985
-0.846
-0.563
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-0.525
-0.502
-0.225
-0.105
-0.087

1.982
1.919
1.737
1.714
1.534
1.514
1.488
1.466
1.446
1.411
1.382
0.992
0.935

-0.000



18e

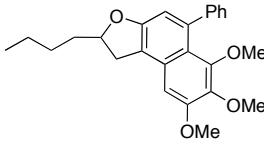


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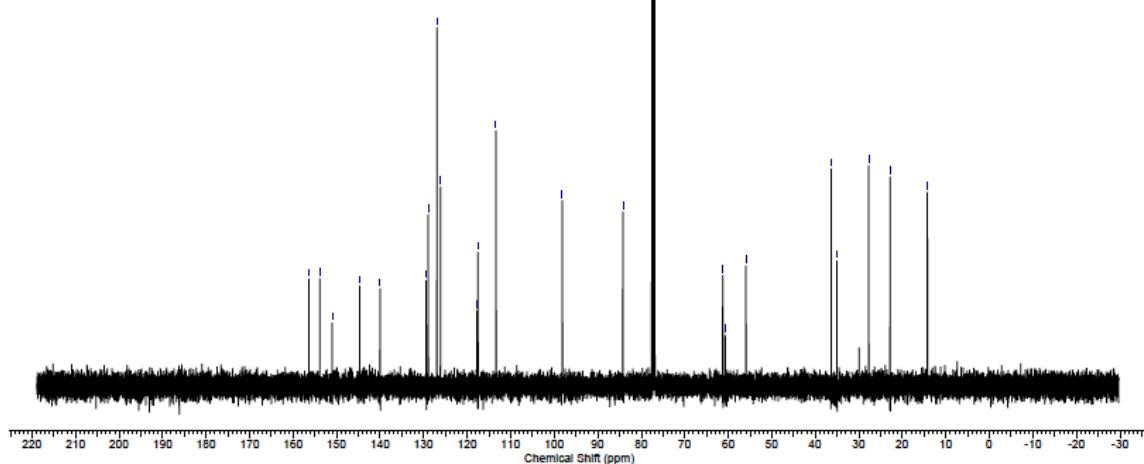
This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

156.267
153.677
151.67
144.746
140.017
129.427
128.991
126.127
117.816
117.839
113.401

68.167
64.240
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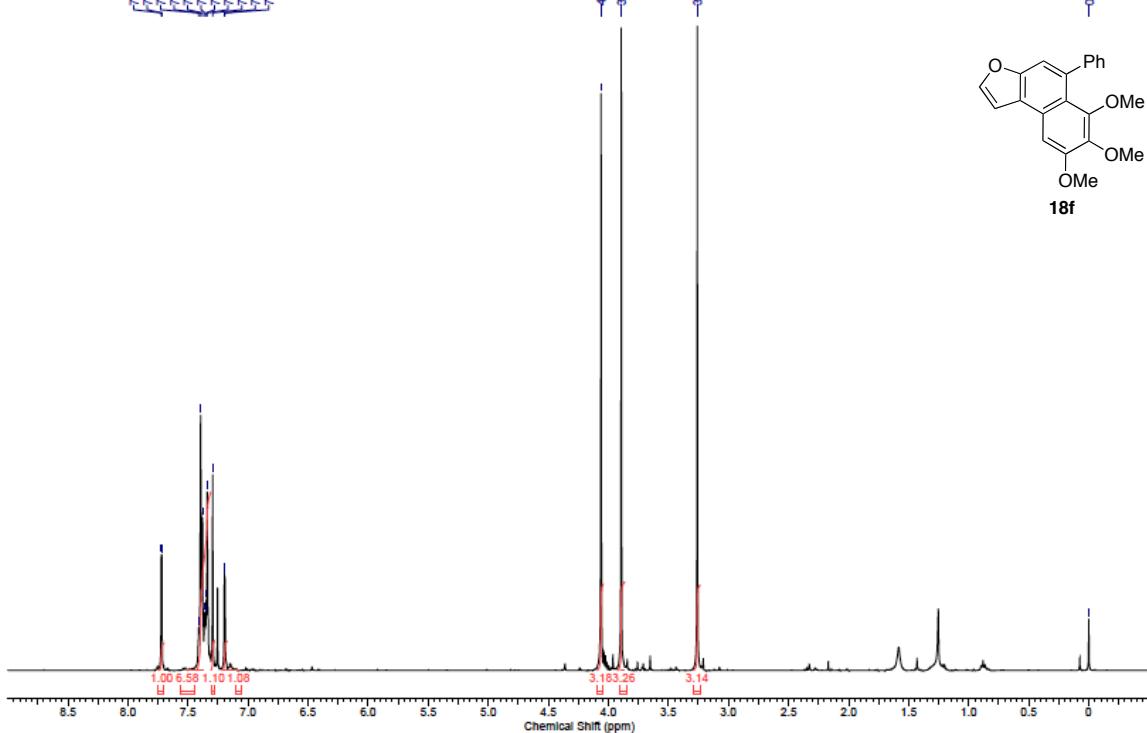


18e

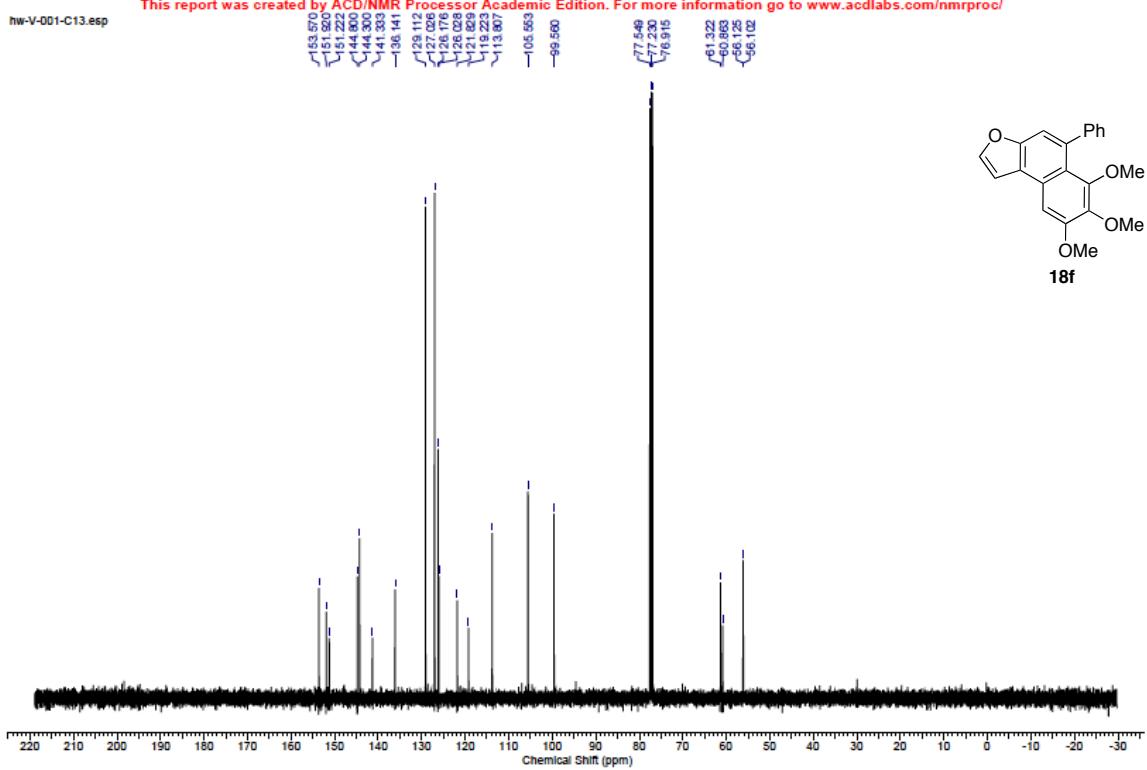


¹H and ¹³C spectra of compound 18e

hw-V-001-1H.esp This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

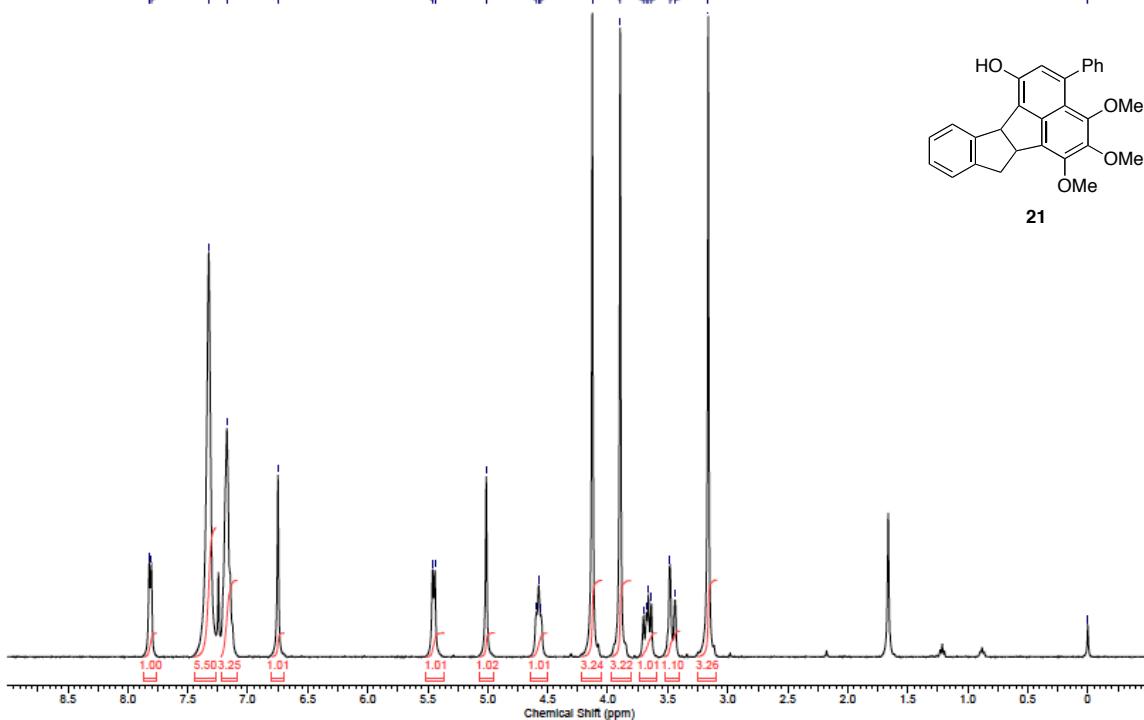
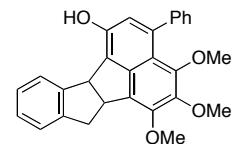


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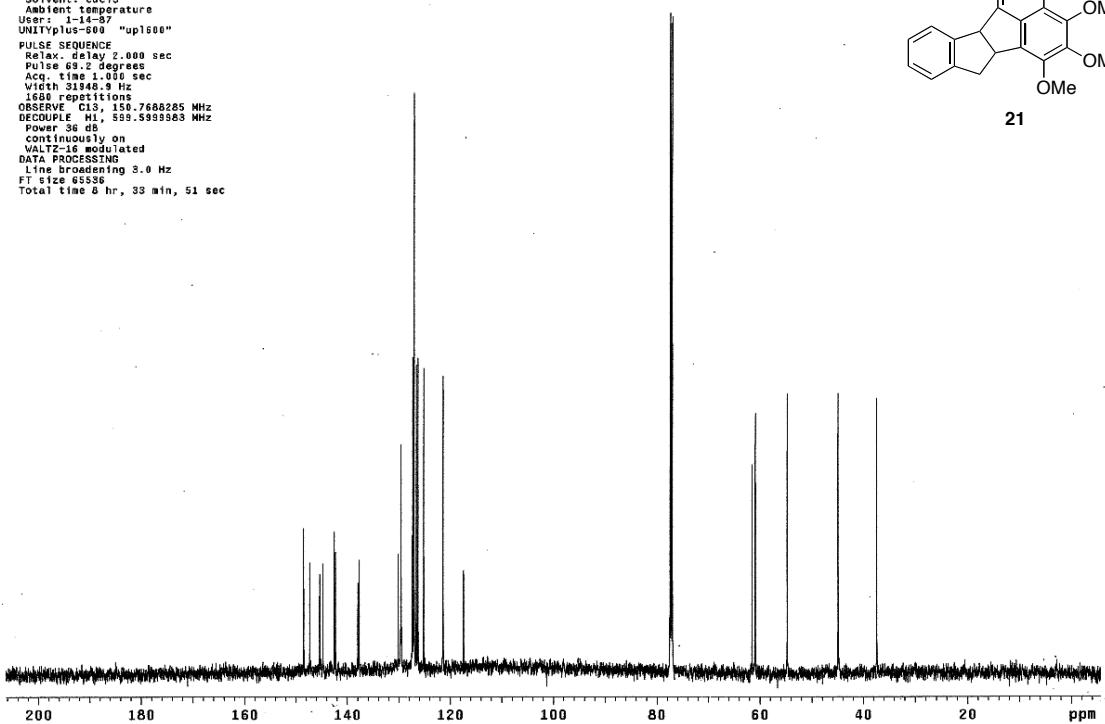
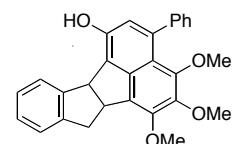


¹H and ¹³C spectra of compound 18f

hw-V-022-B-1H.esp
This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/



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Solvent: cdcl3
Atmospheric temperature
User: 1-14-87
UNITplus-600 "up1606"
PULSE SEQUENCE
Relax: delay 2.000 sec
Pulse 60 degrees
Acq time 1.000 sec
Width 31948.9 Hz
1681 repetition
OBSERVE: 131.55, 76.768285 MHz
DECOUPLE: 1H, 595.599983 MHz
Power 36 dB
Continuum 0.1V on
WALTZ58 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 8 hr, 33 min, 51 sec
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¹H and ¹³C spectra of compound **21**