

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

Supporting information

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

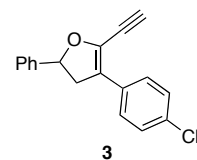
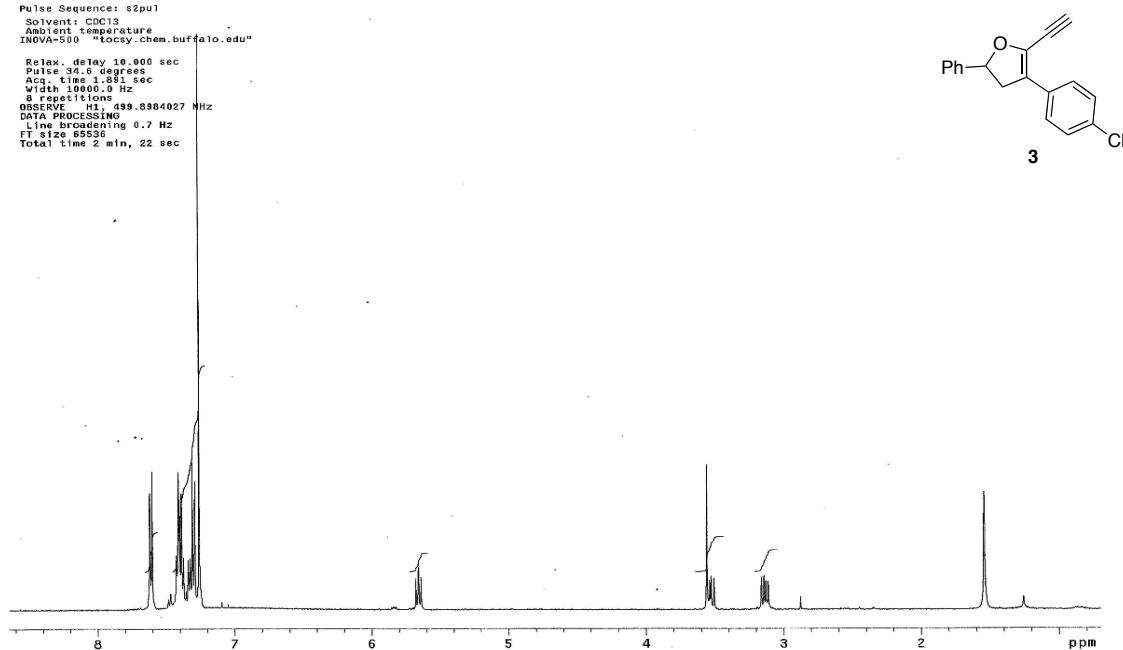
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hmdavie@emory.edu

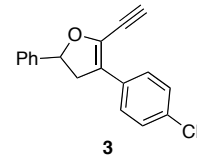
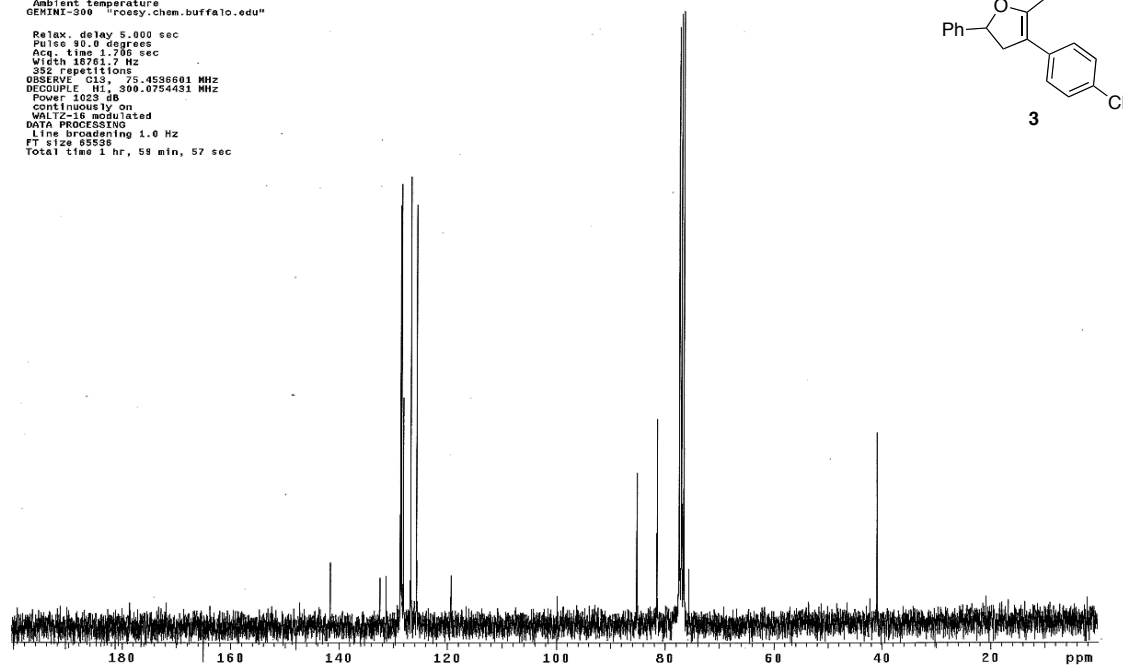
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Experimental Procedures	SI-2-43
Spectral data	SI-44-93

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Solvent: CDCl3
Ambient temperature
INOVA-500 "tocsy.chem.buffalo.edu"
Relax. delay 10.000 sec
Pulse 90.0 degrees
Acq. time 1.081 sec
Width 10000.0 Hz
8 repetitions
OBSERVE F1: 499.8384027 MHz
DATA PROCESSING
Line broadening 0.7 Hz
FT size 85536
Total time 2 min, 22 sec



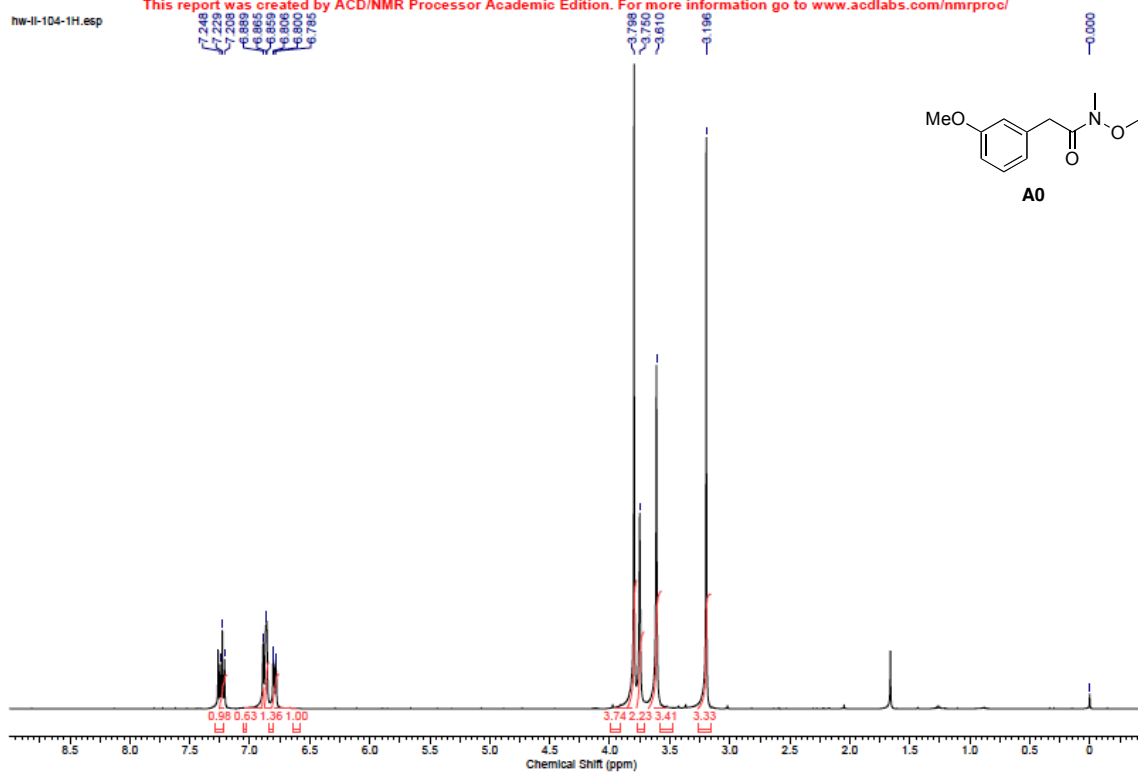
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Width 10761.7 Hz
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OBSERVE G13: 75.4536601 MHz
DECOUPLE H1: 300.0754431 MHz
Power 1023 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
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Total time 1 hr, 59 min, 57 sec



^1H and ^{13}C spectra of compound 3

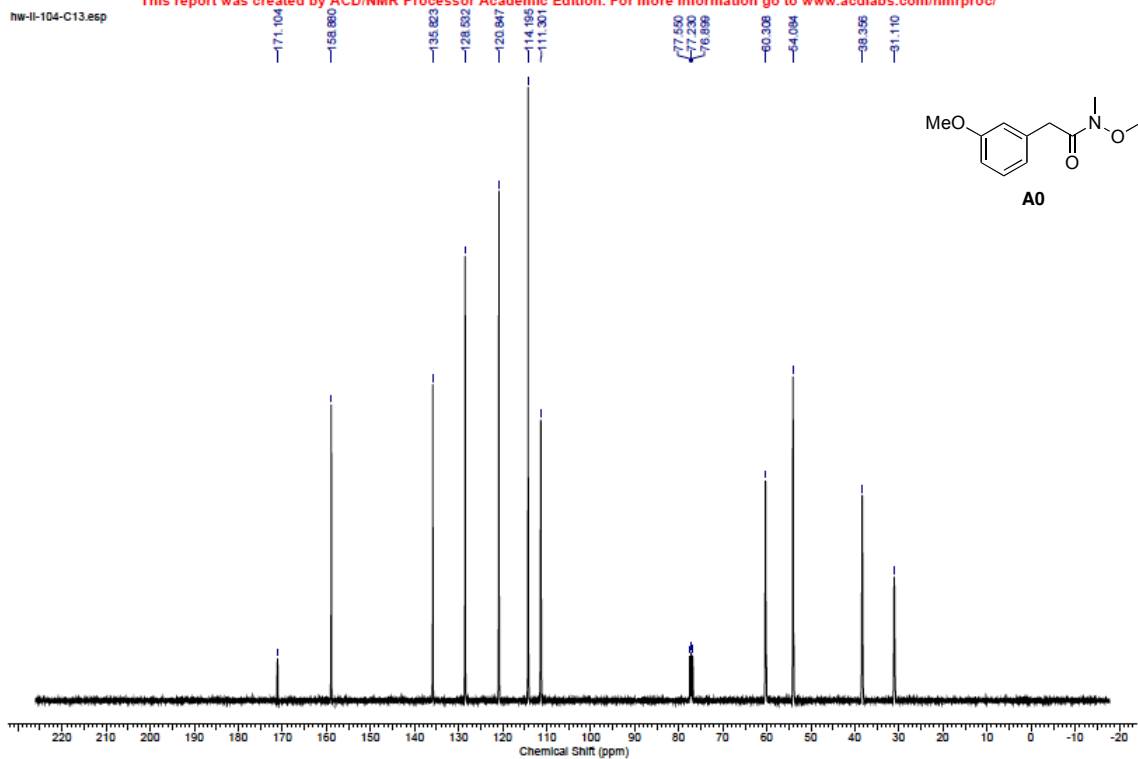
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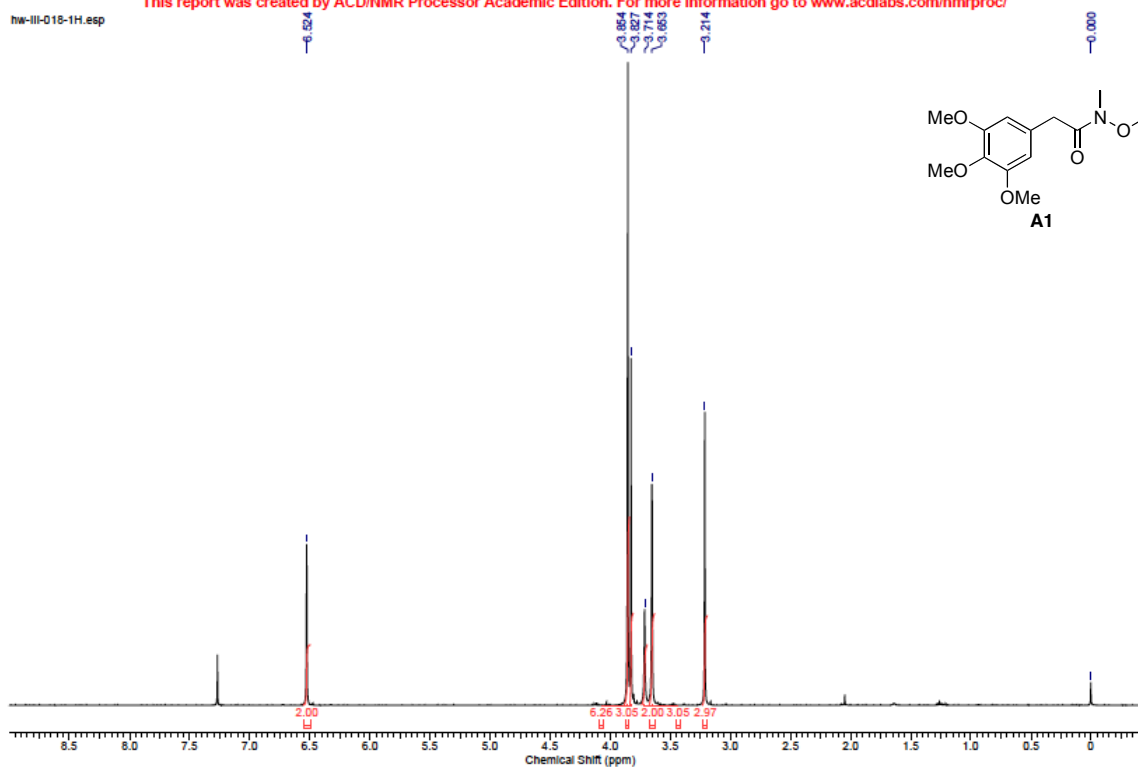
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¹H and ¹³C spectra of compound A0

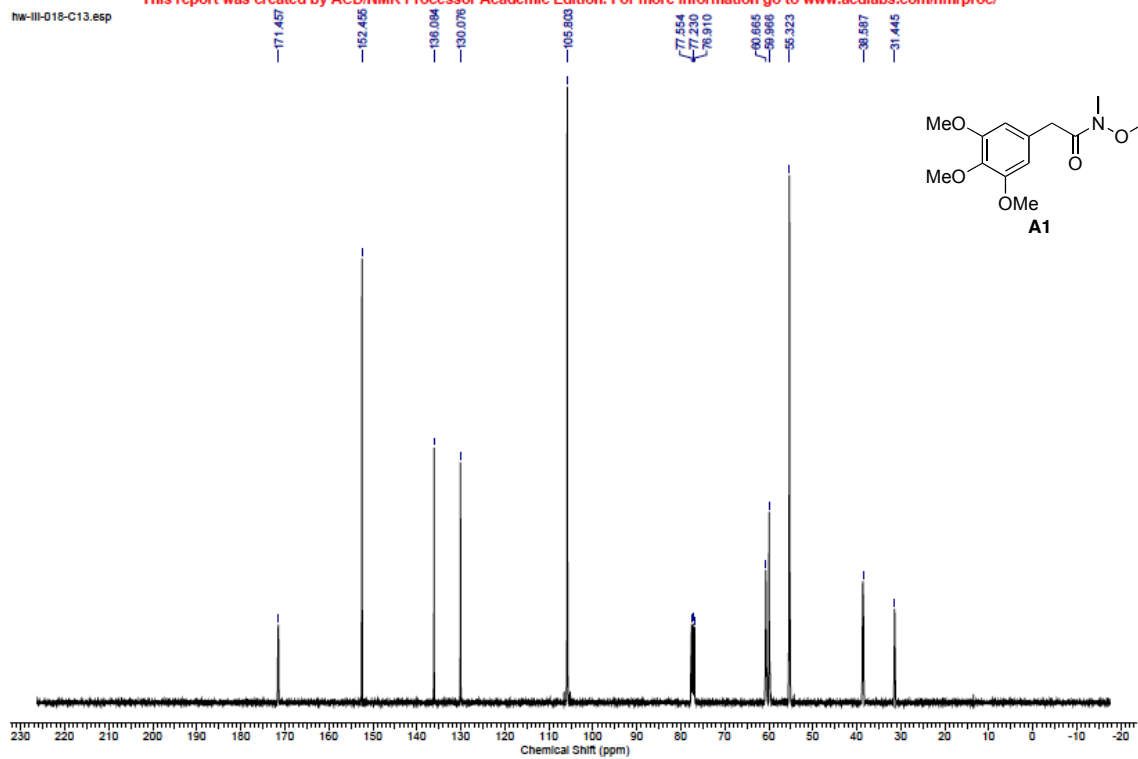
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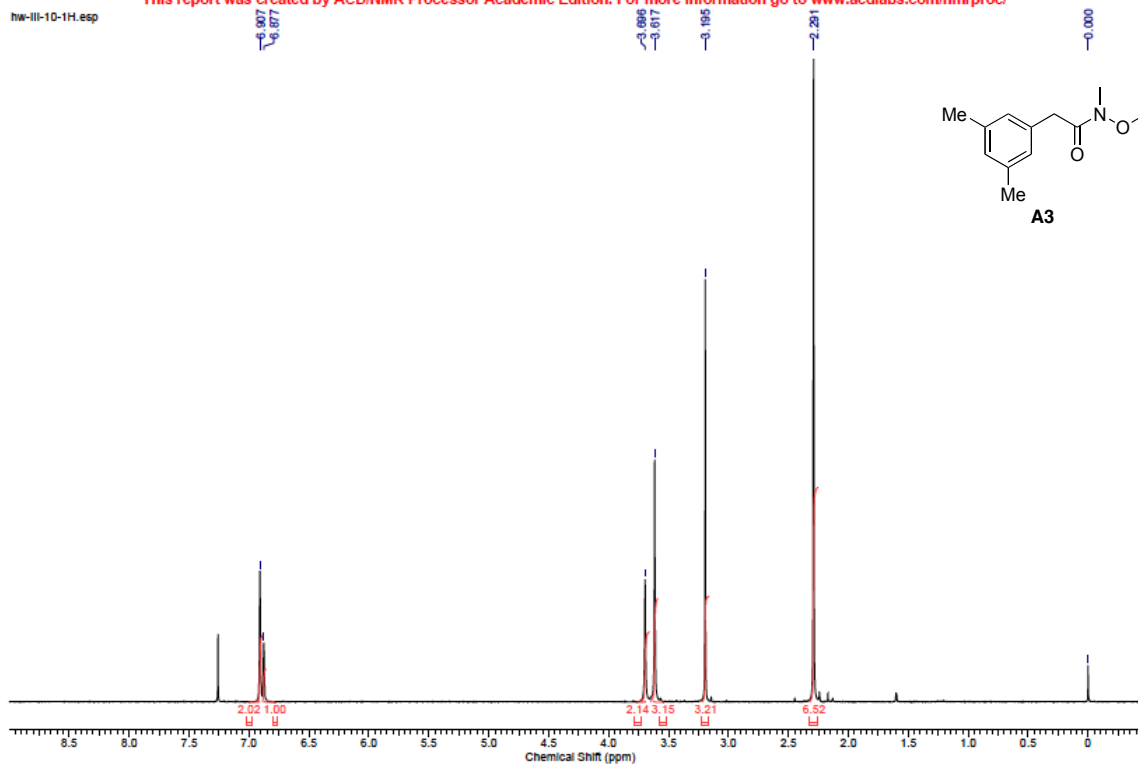
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¹H and ¹³C spectra of compound A1

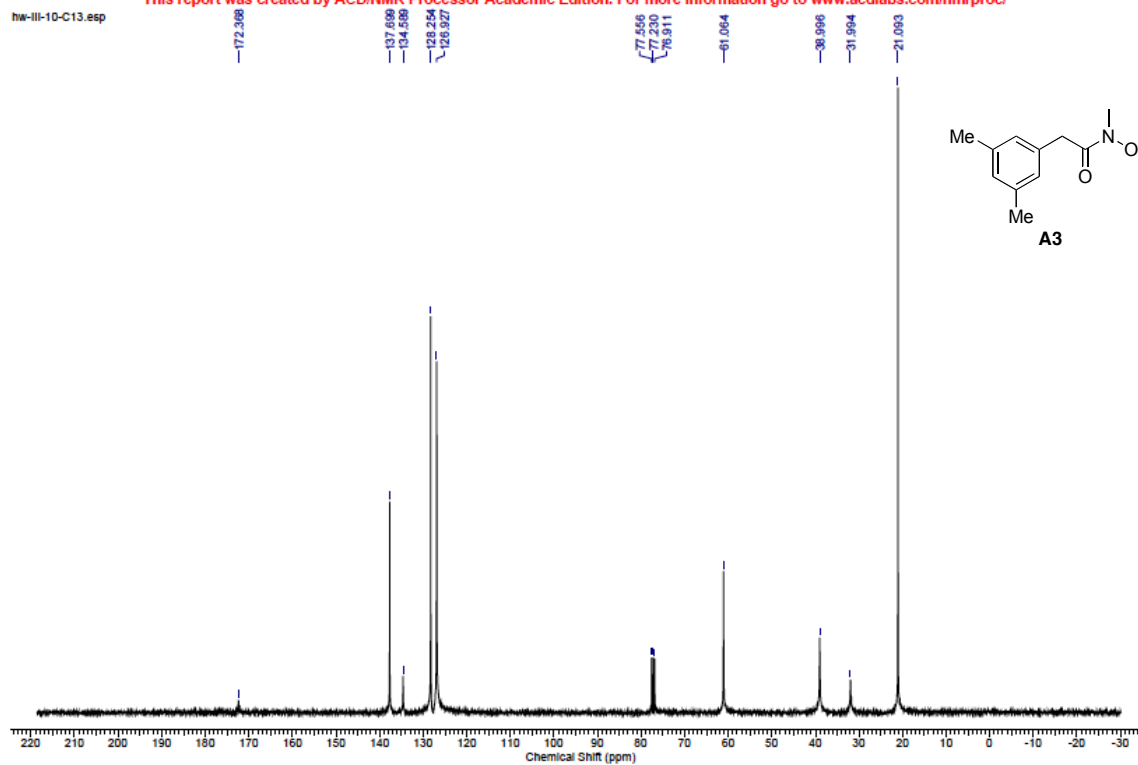
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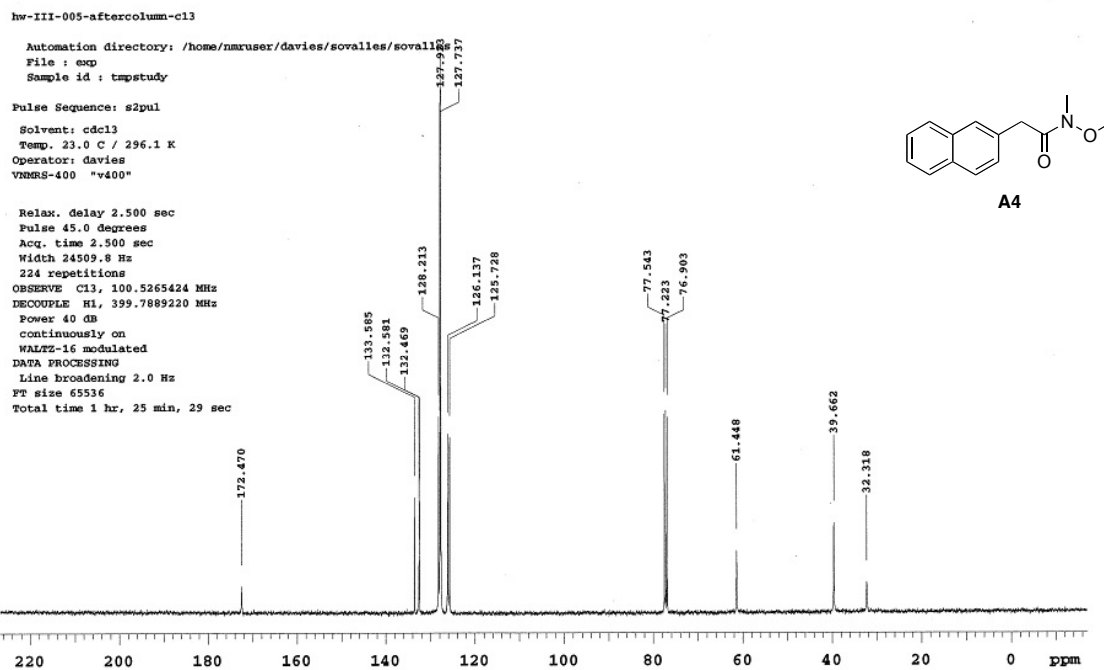
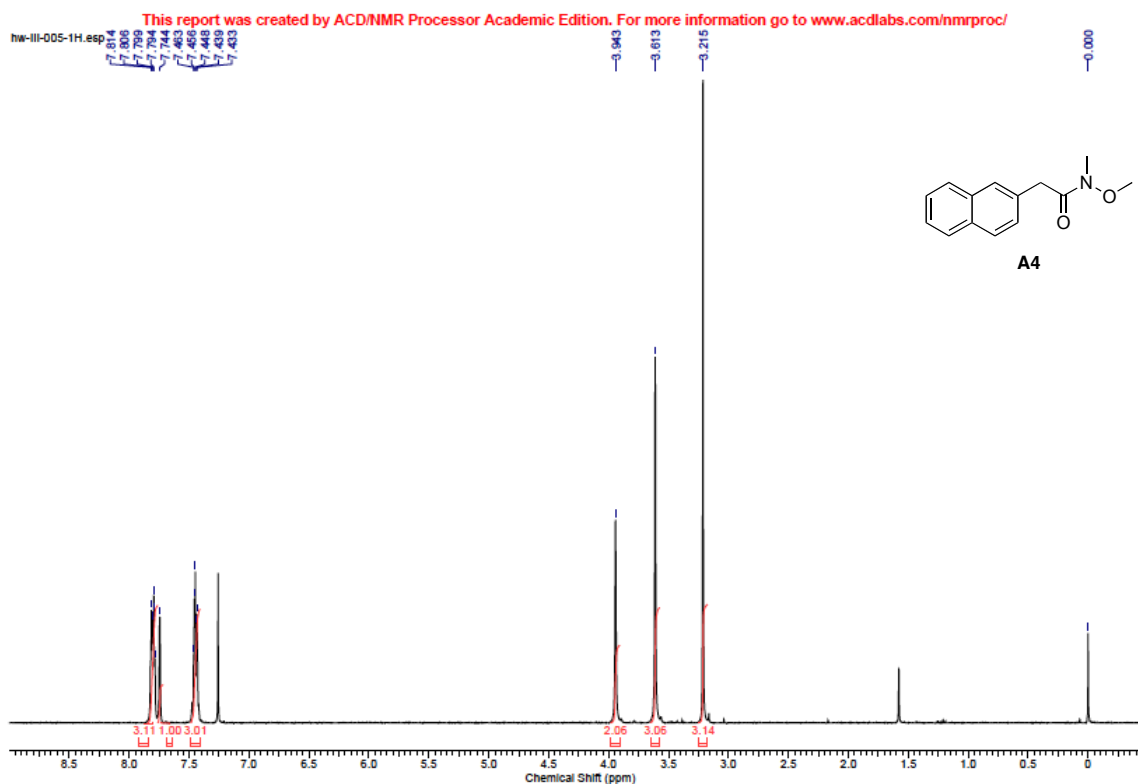


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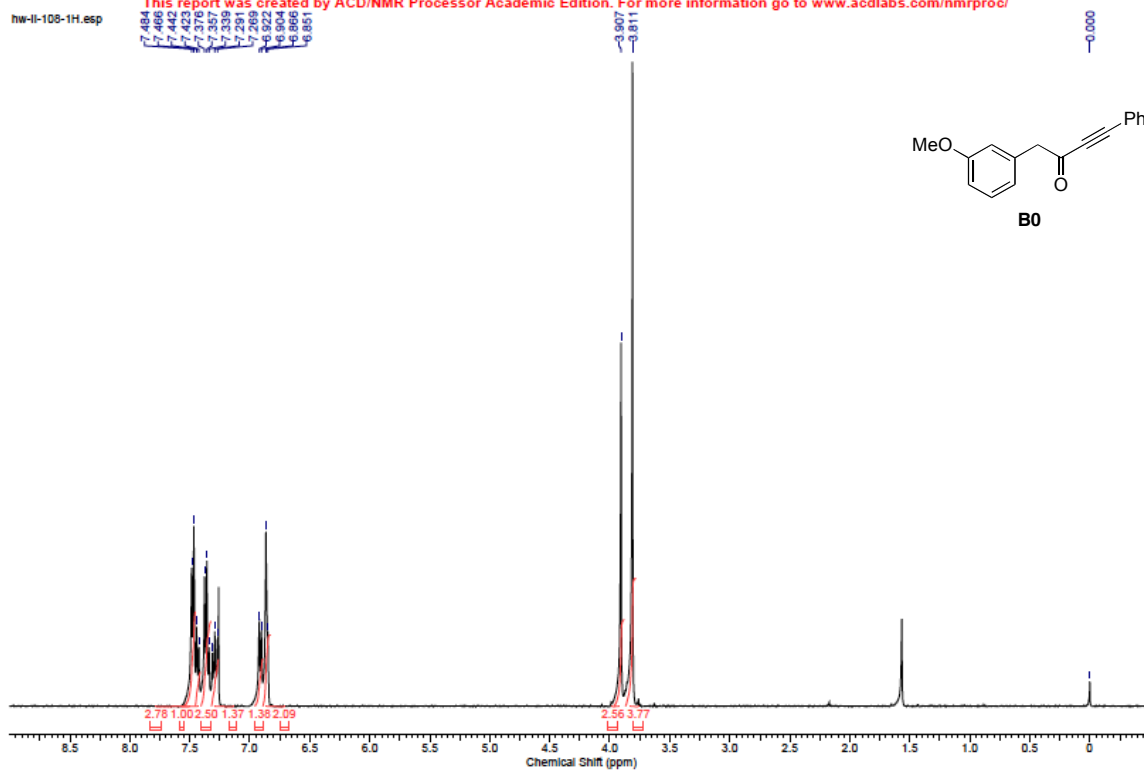
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^1H and ^{13}C spectra of compound A4

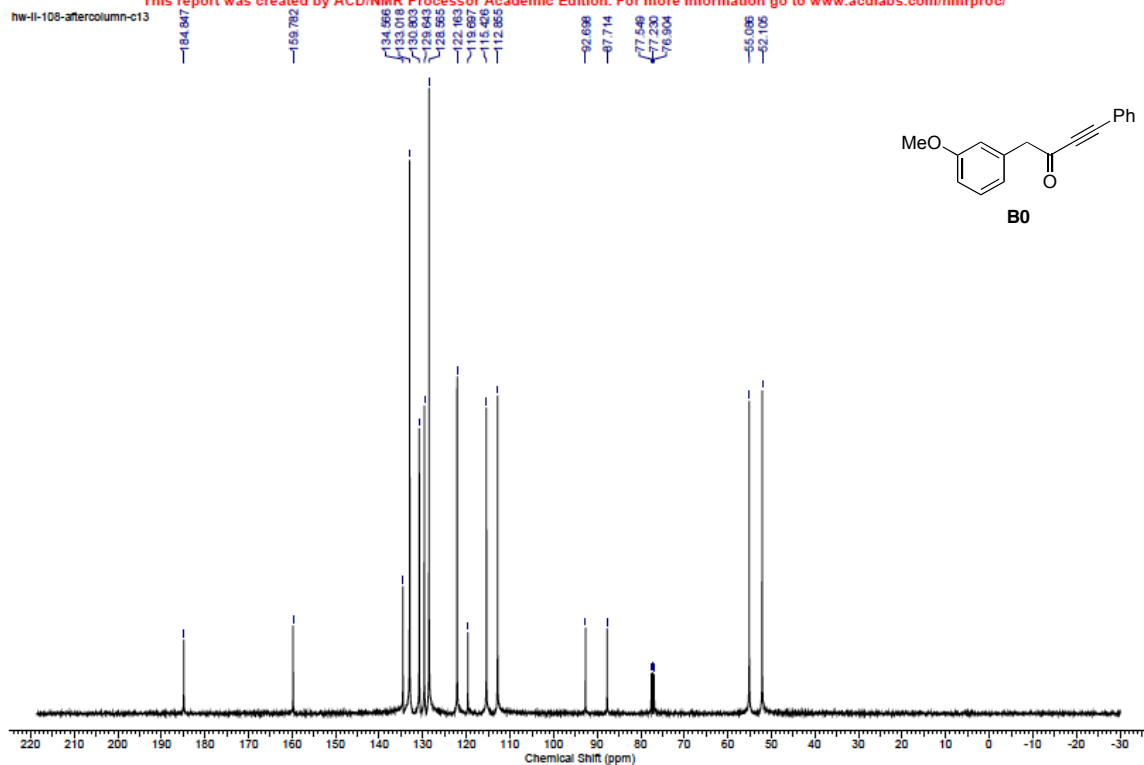
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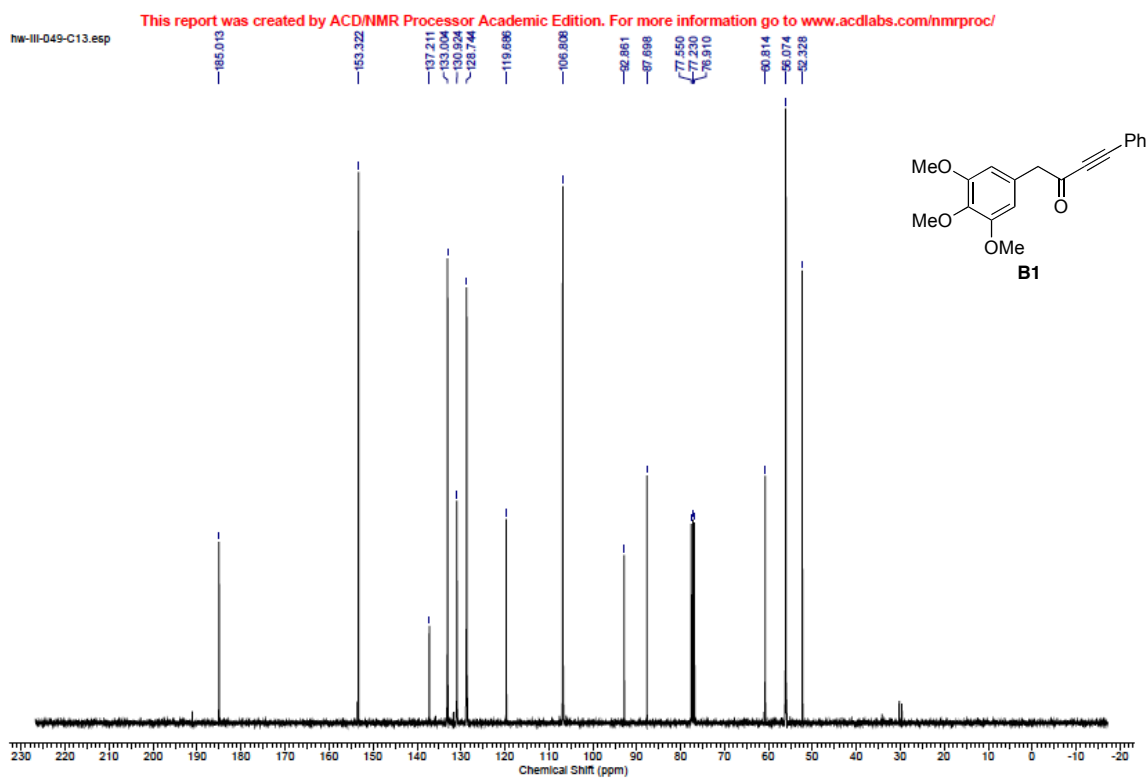
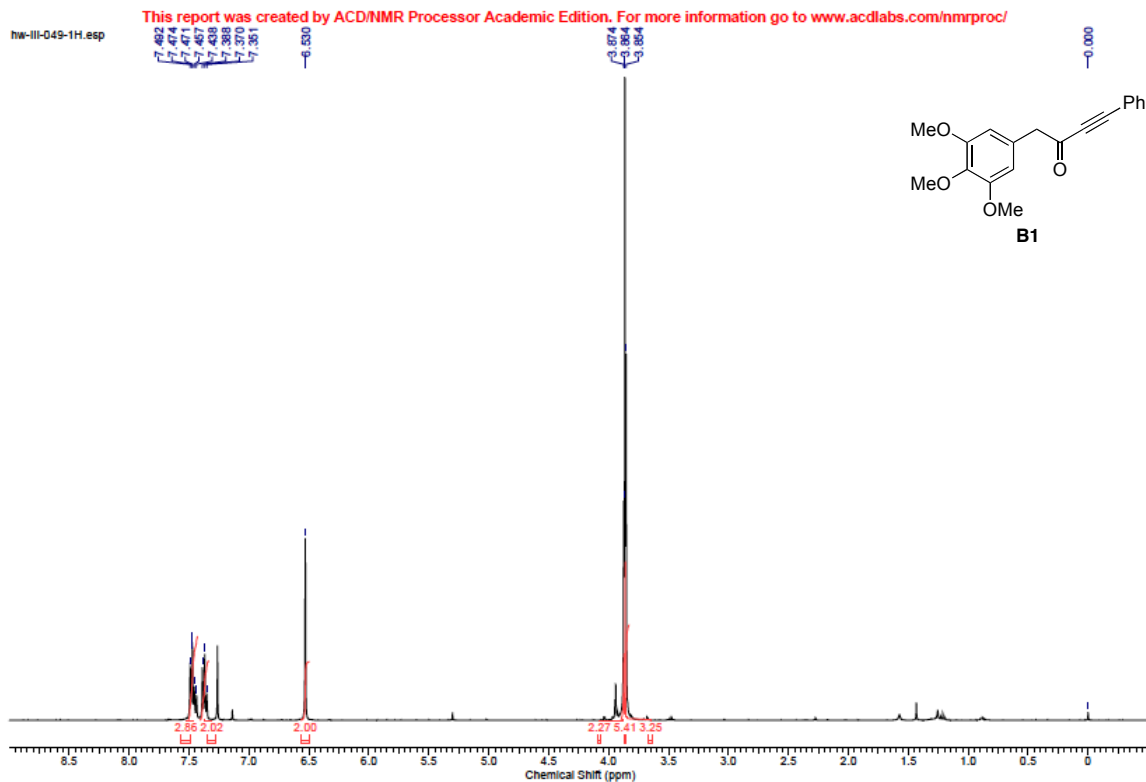


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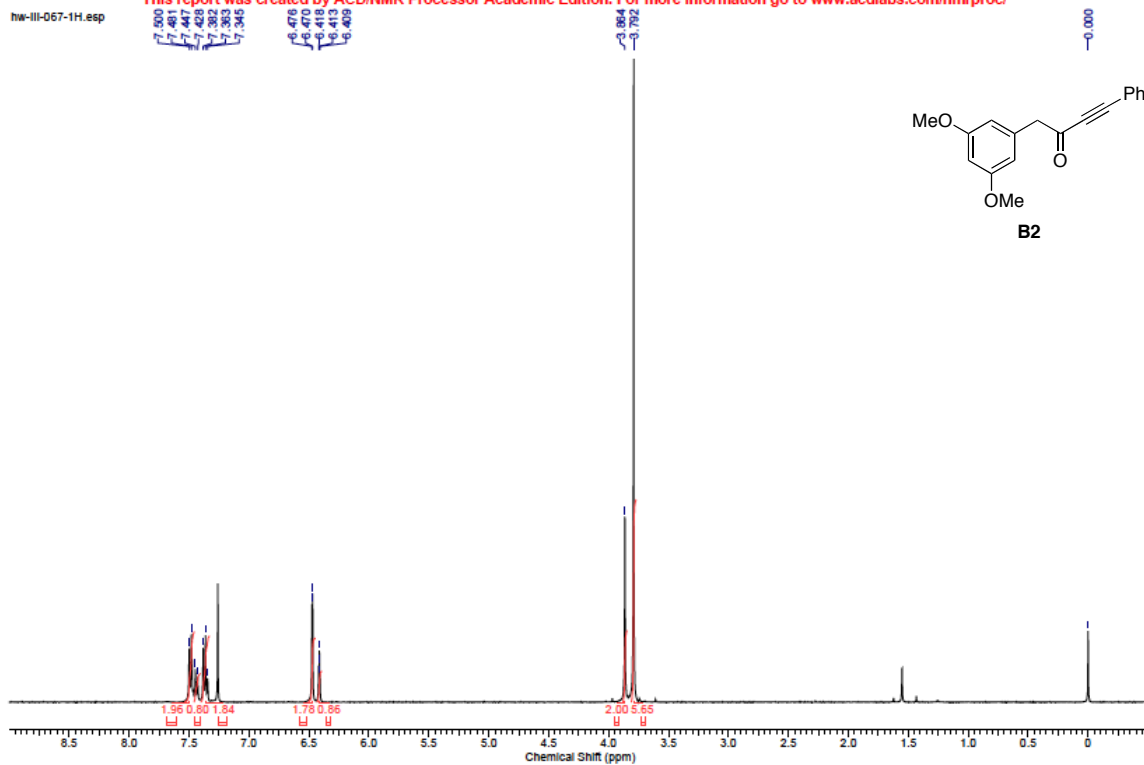
¹H and ¹³C spectra of compound B0



^1H and ^{13}C spectra of compound **B1**

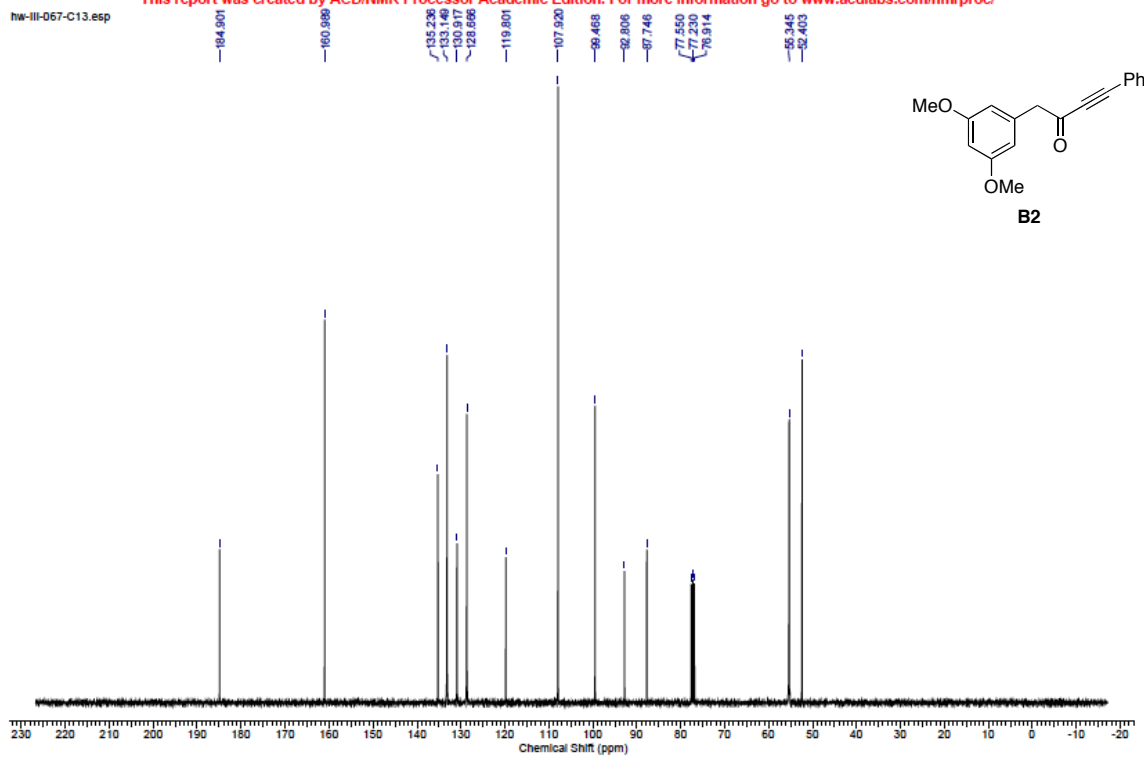
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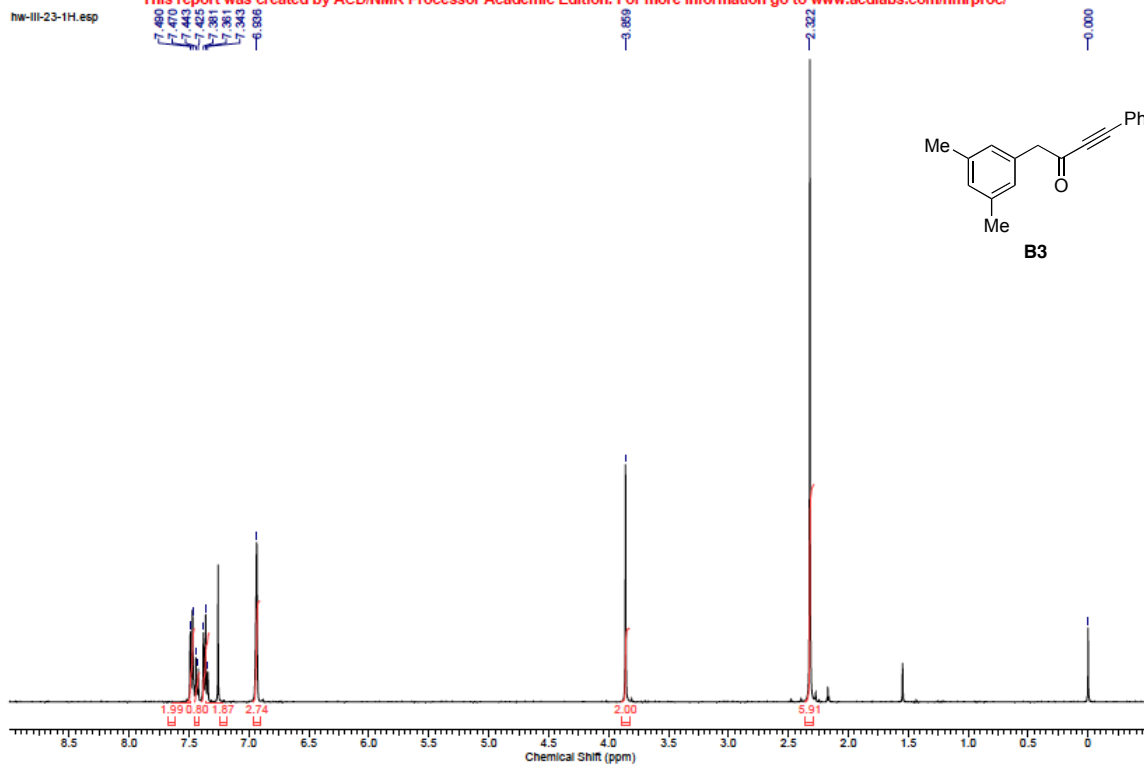
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^1H and ^{13}C spectra of compound **B2**

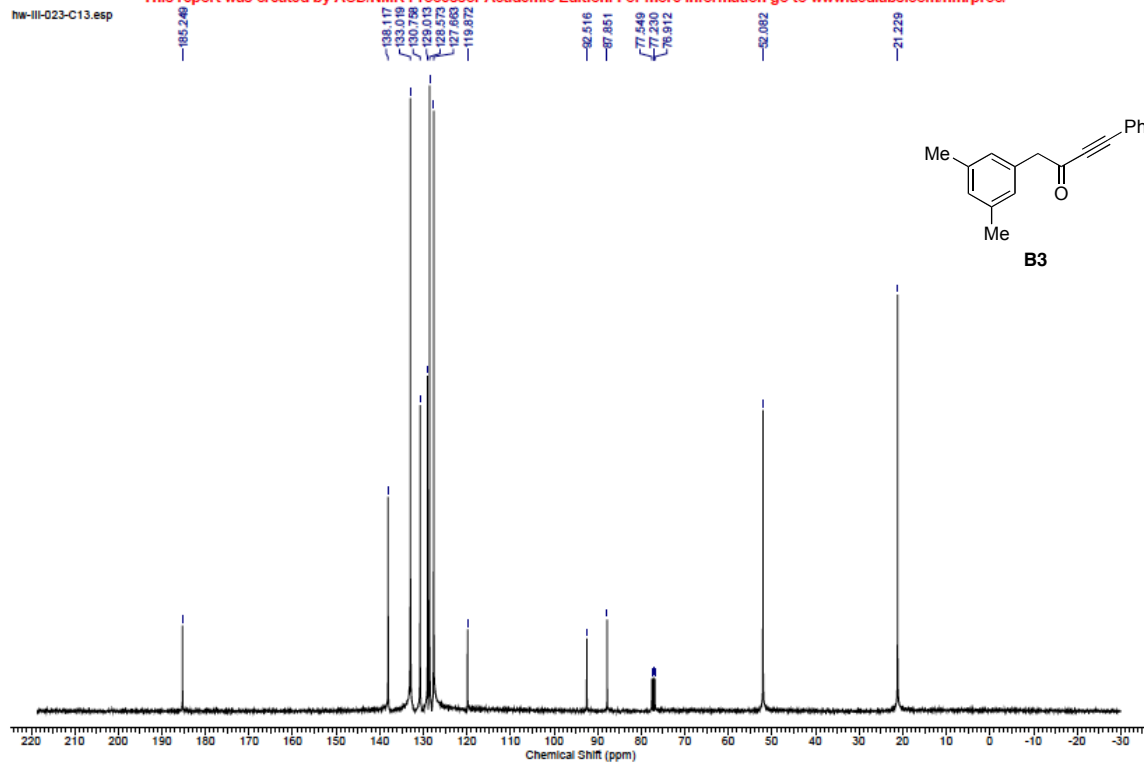
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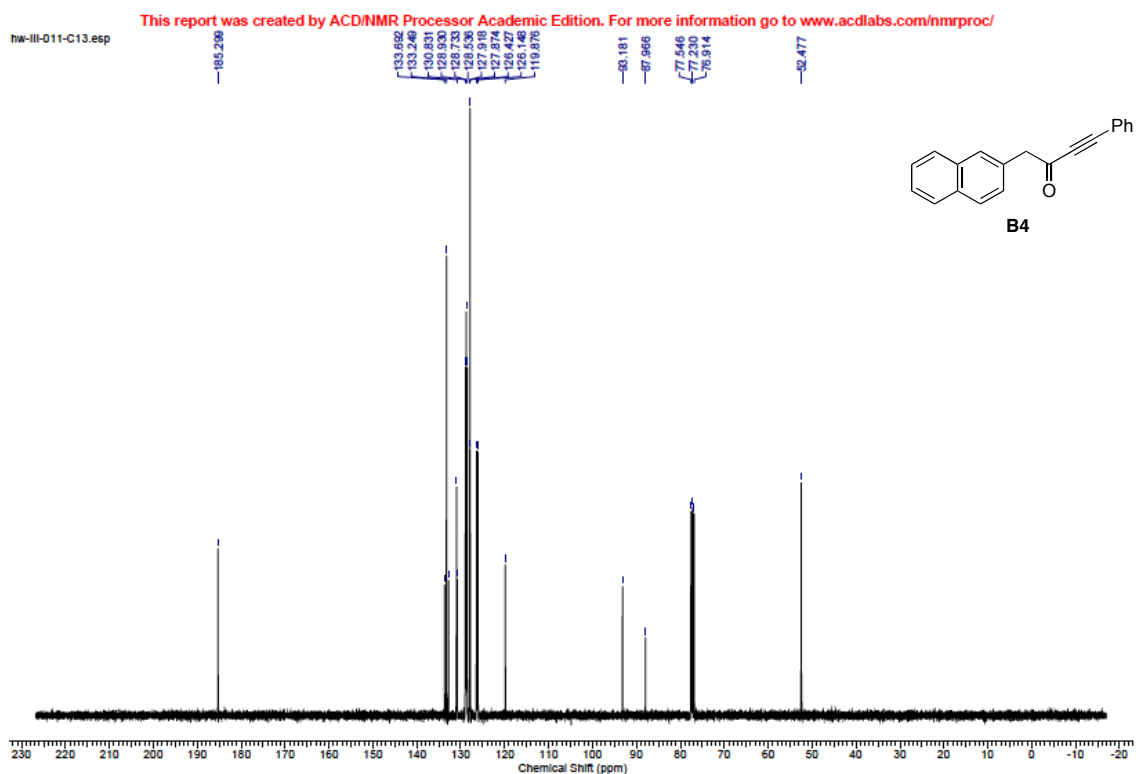
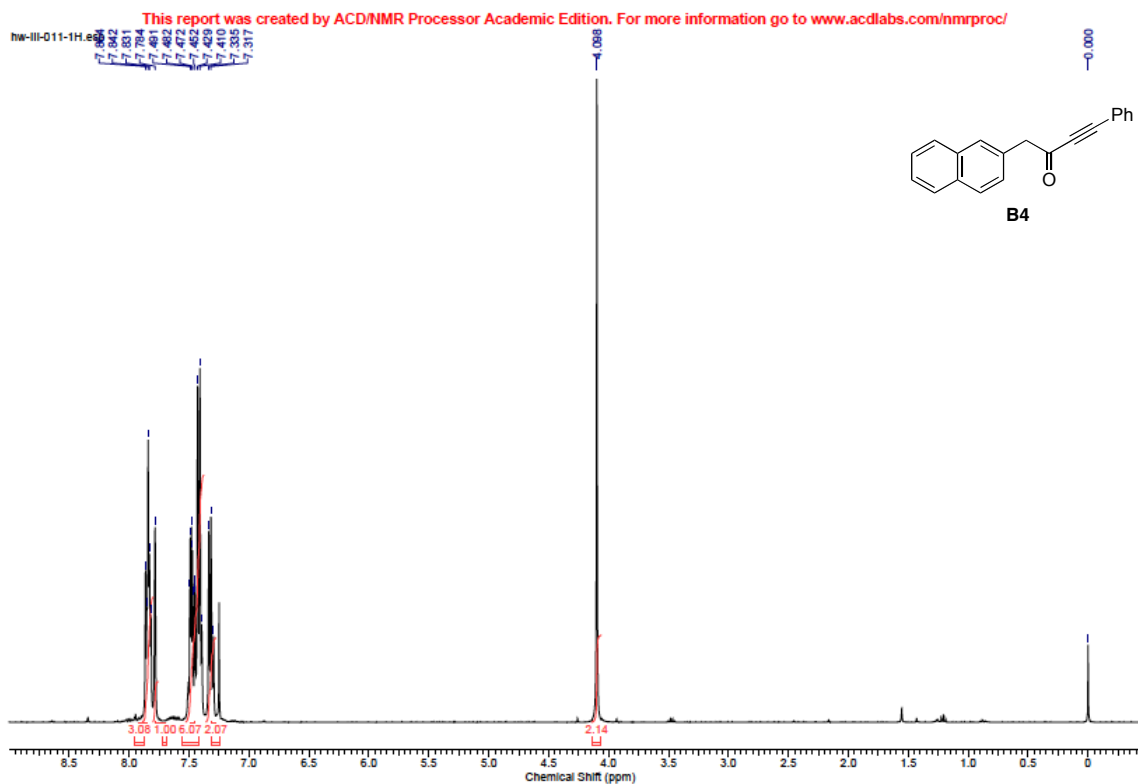


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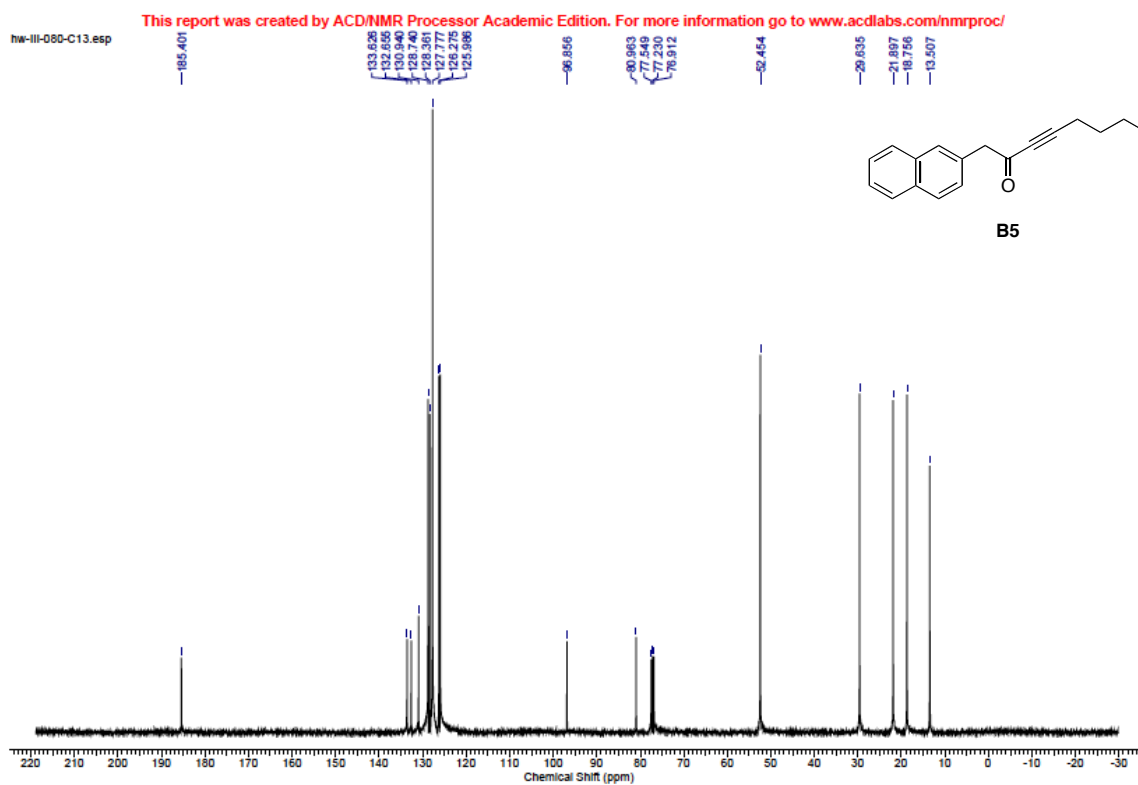
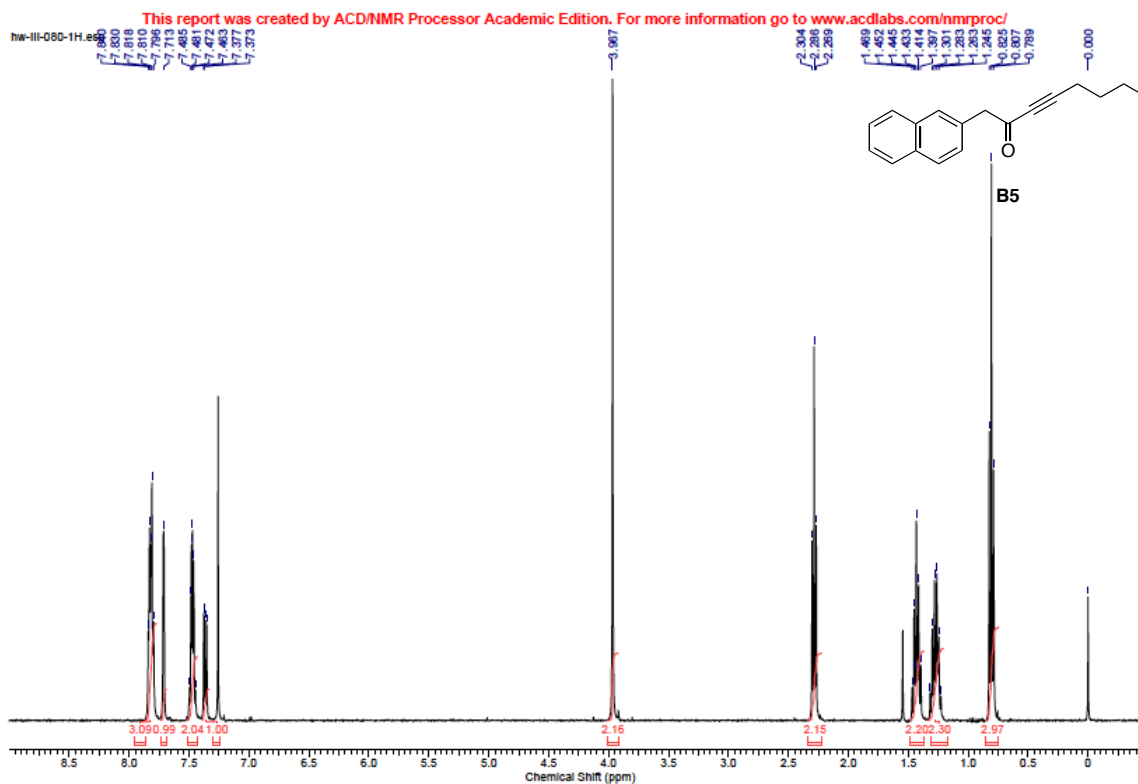
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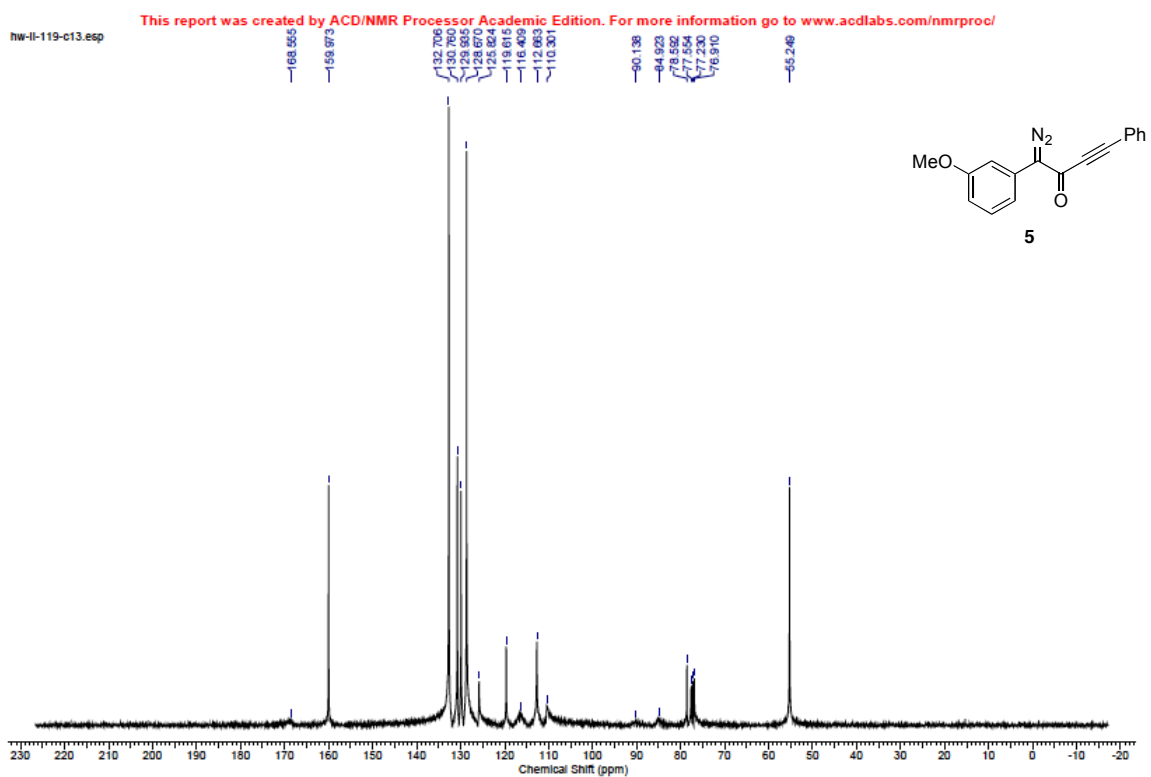
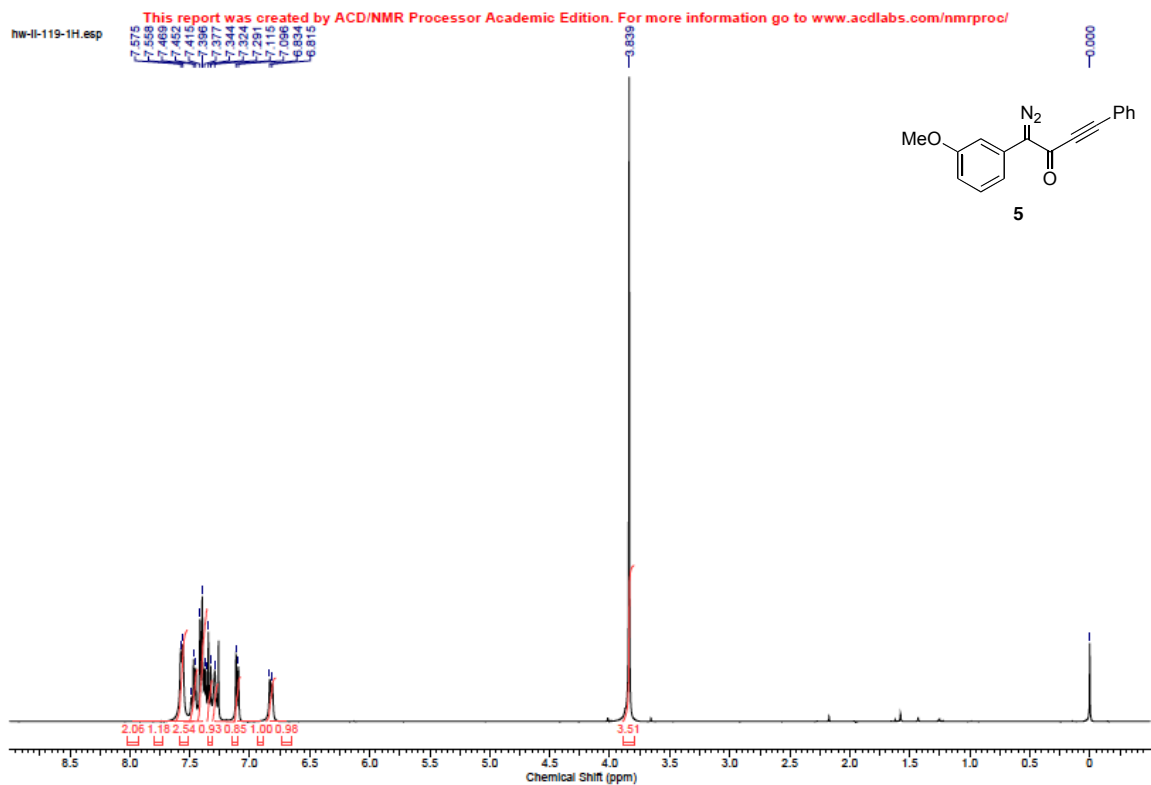
^1H and ^{13}C spectra of compound B3



^1H and ^{13}C spectra of compound **B4**



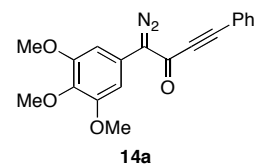
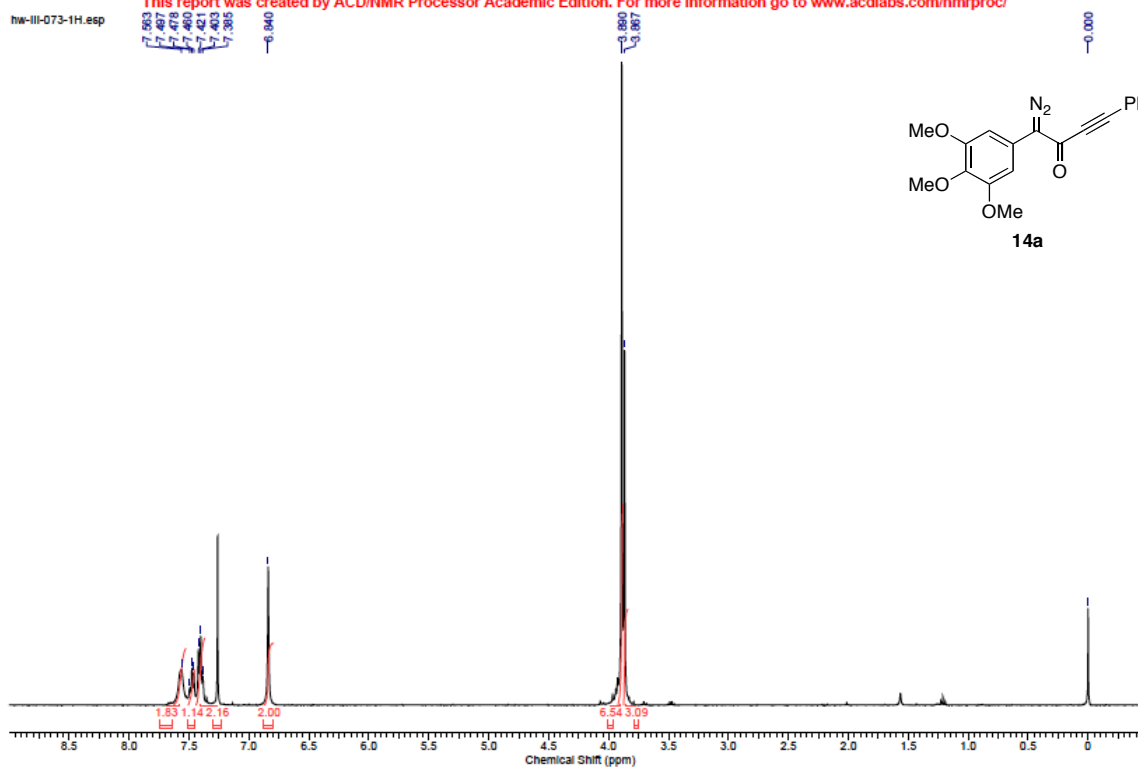
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^1H and ^{13}C spectra of compound **5**

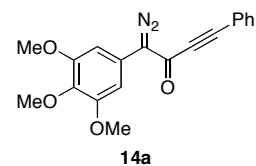
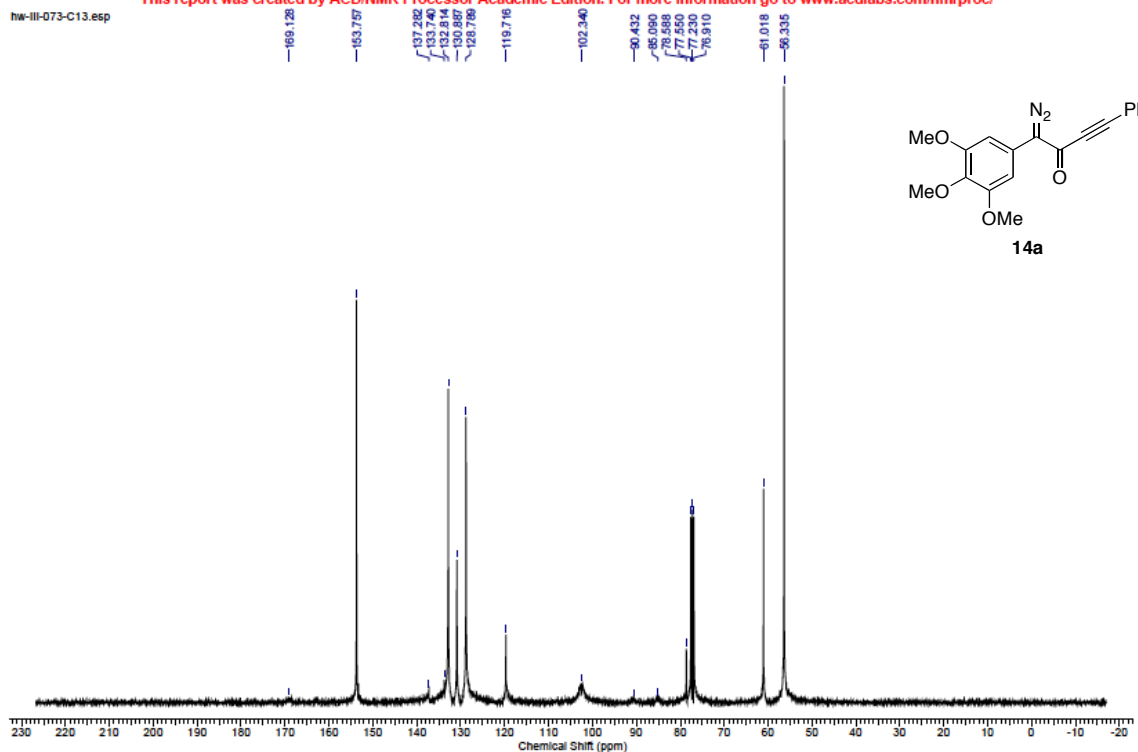
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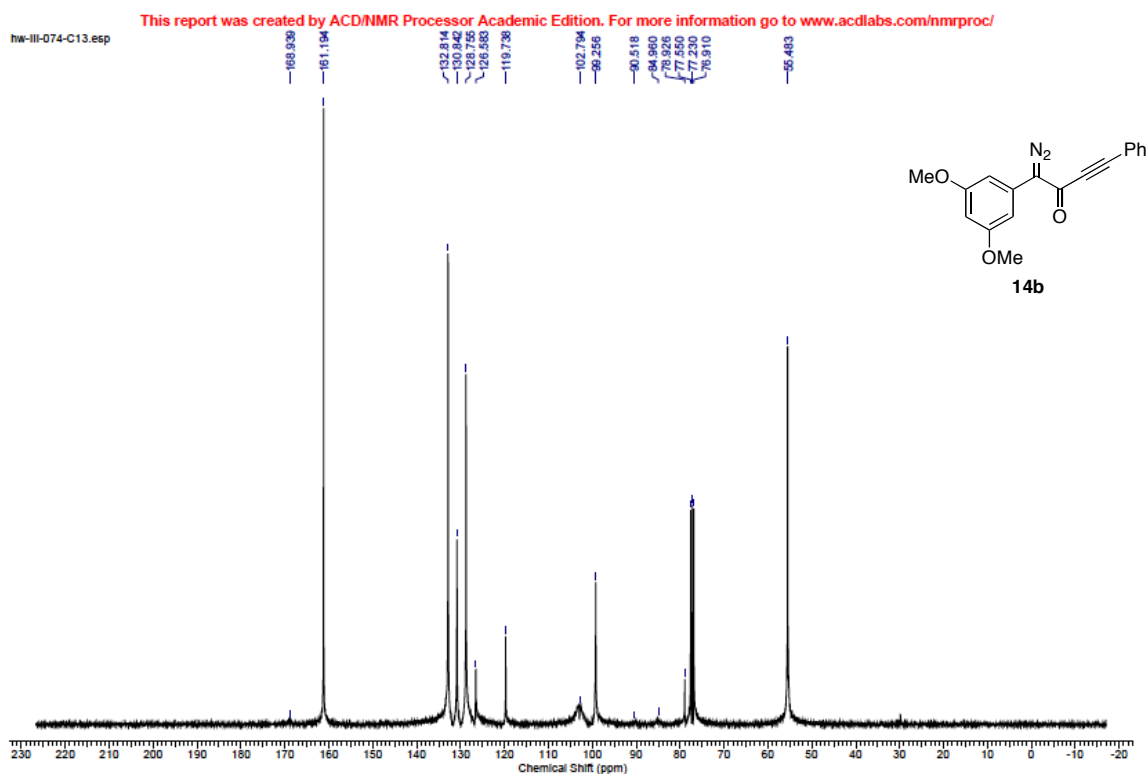
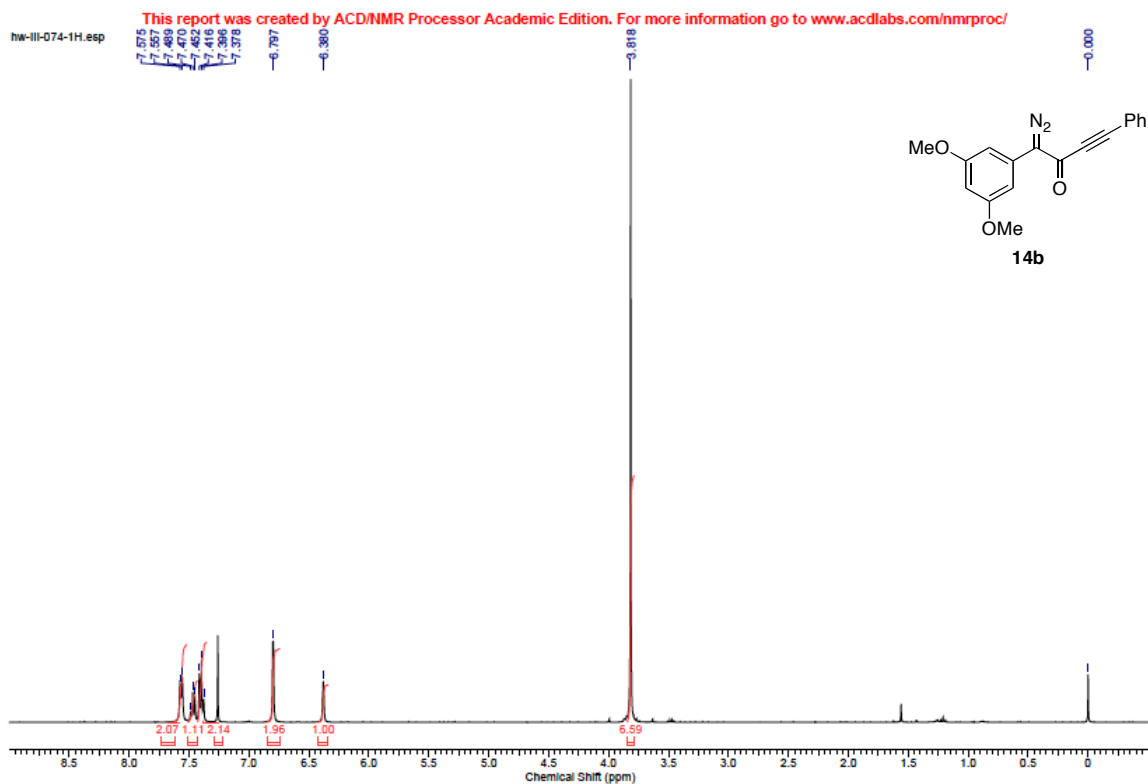


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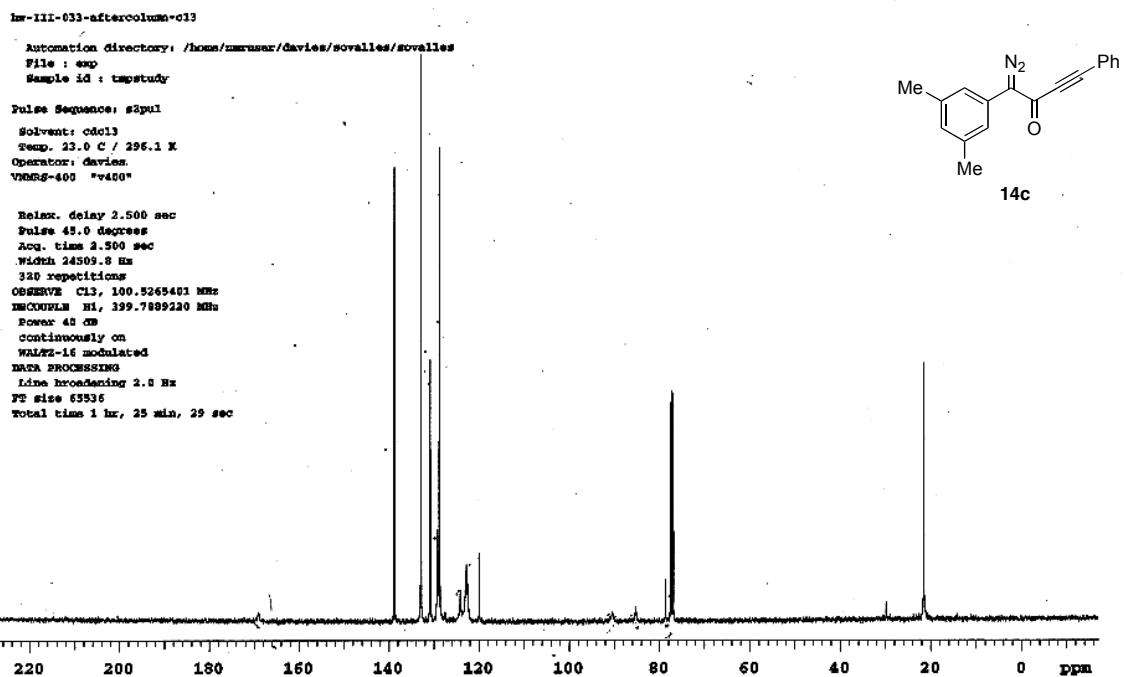
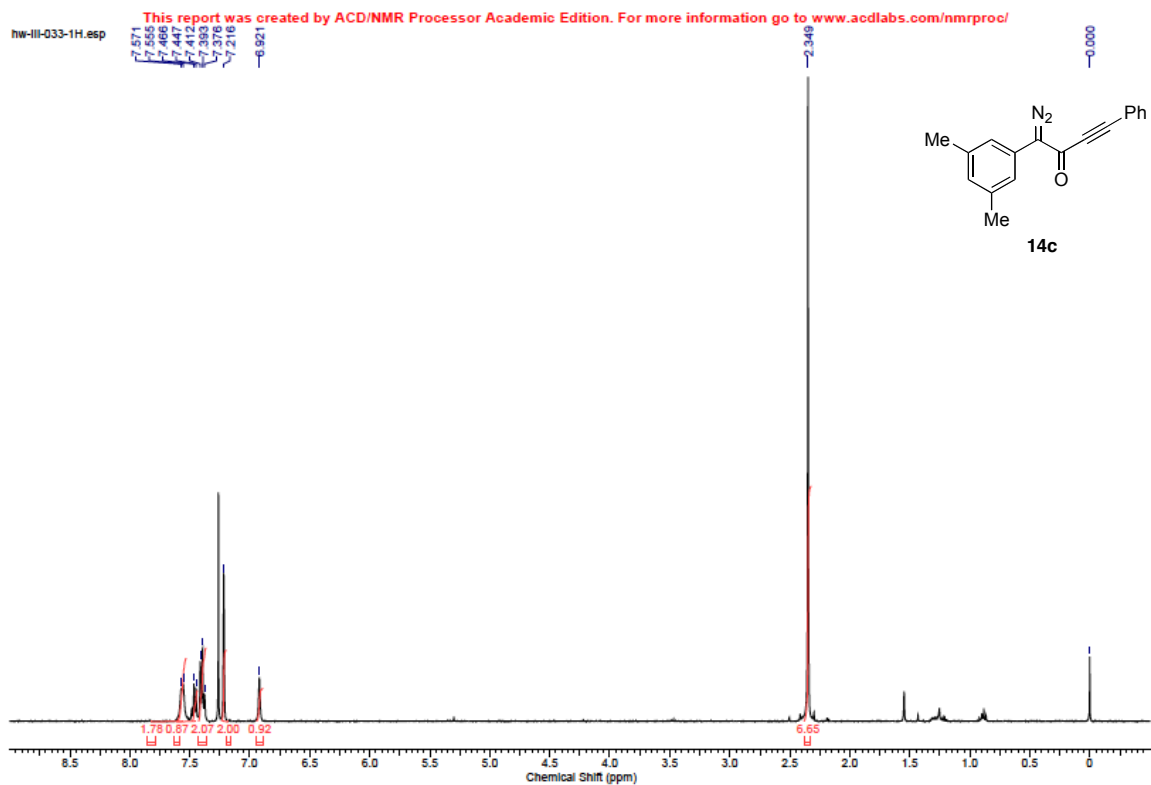
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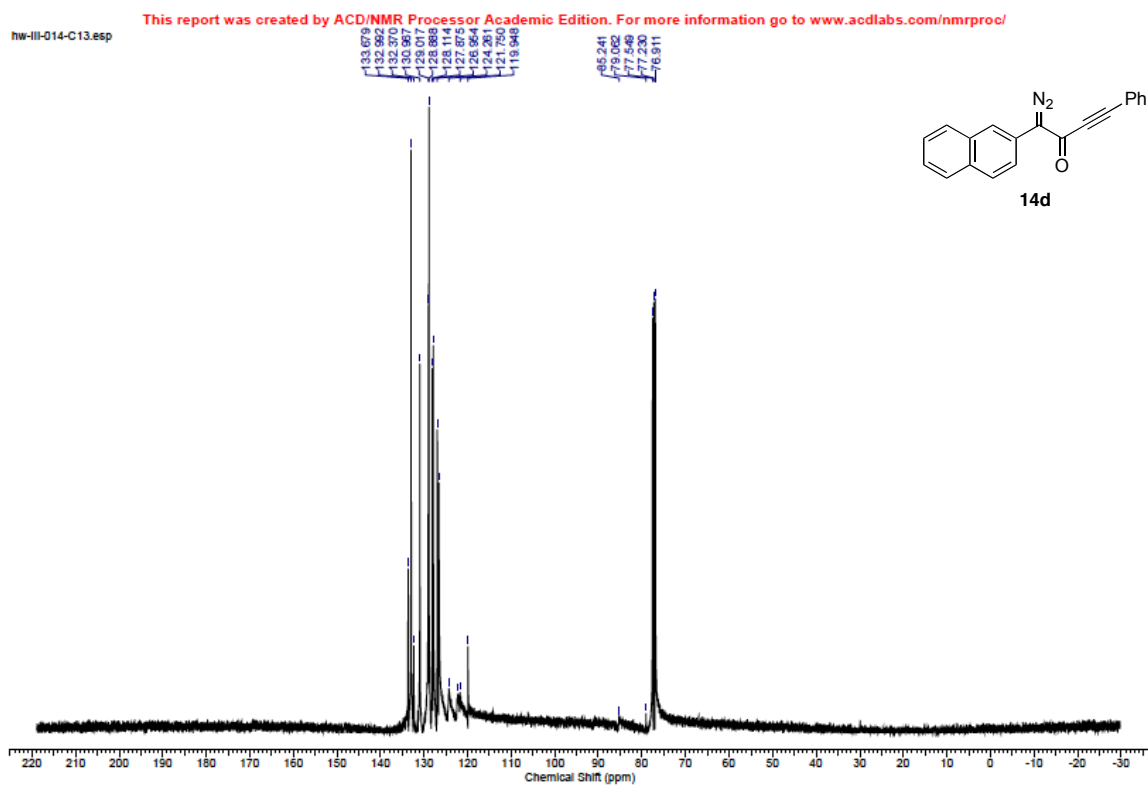
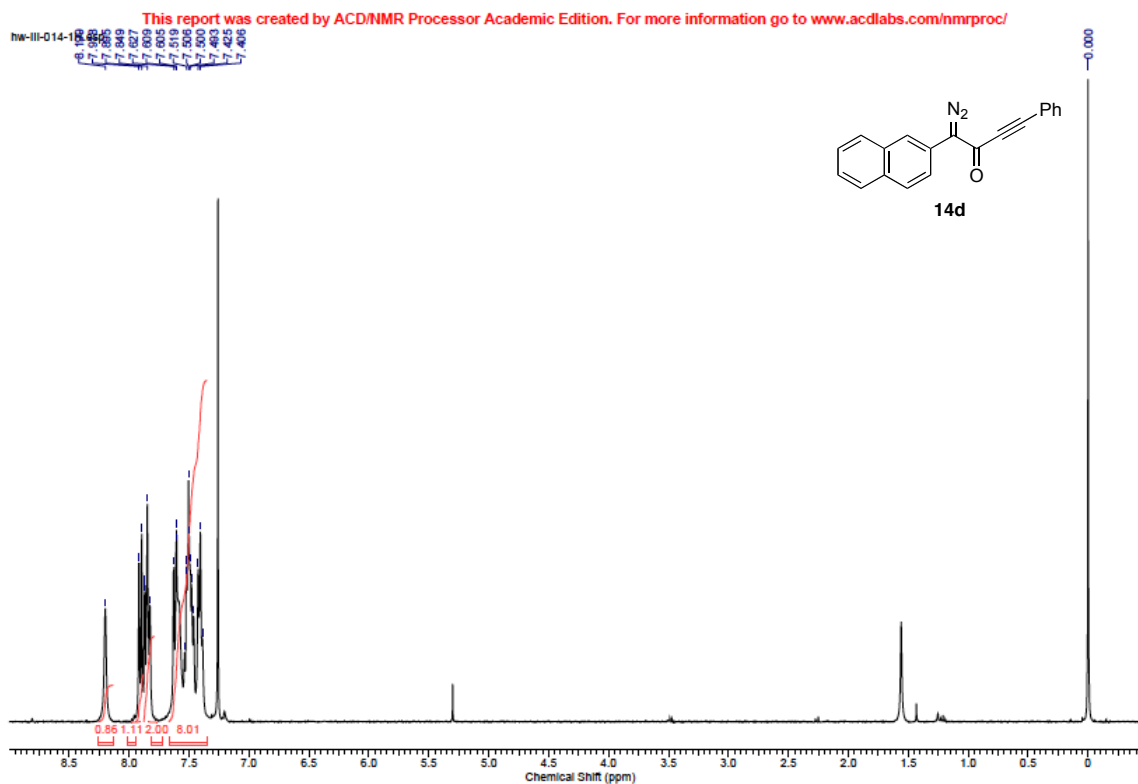
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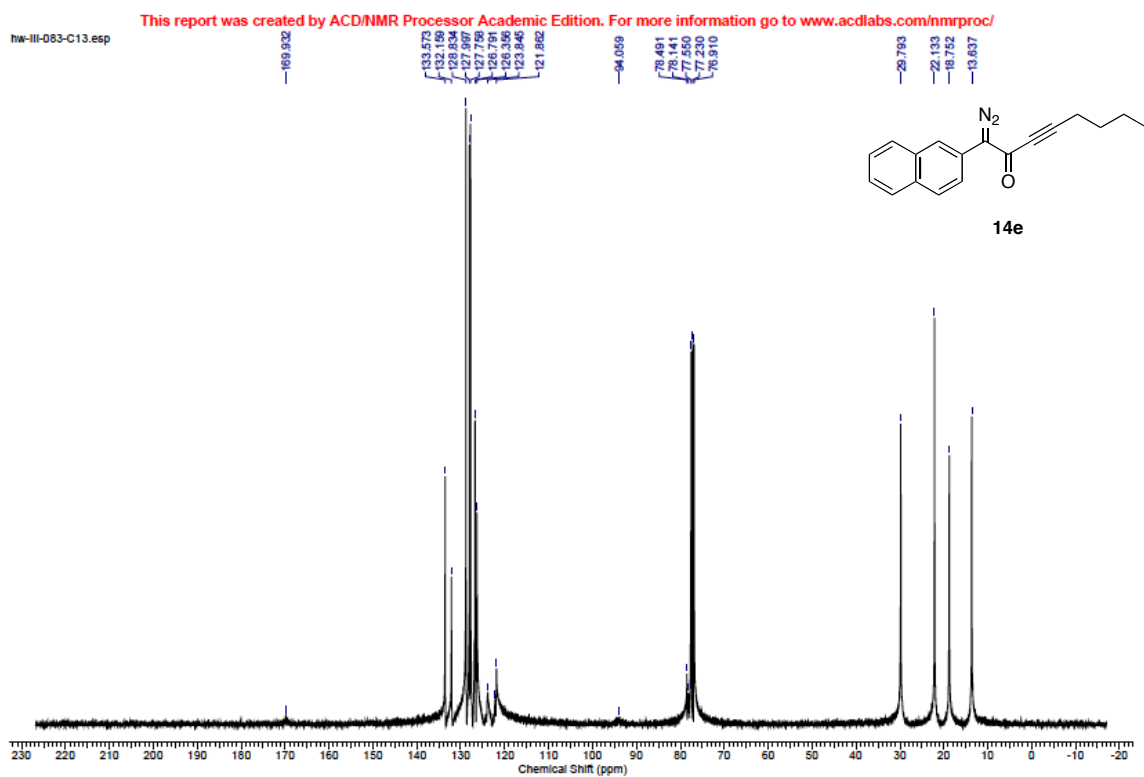
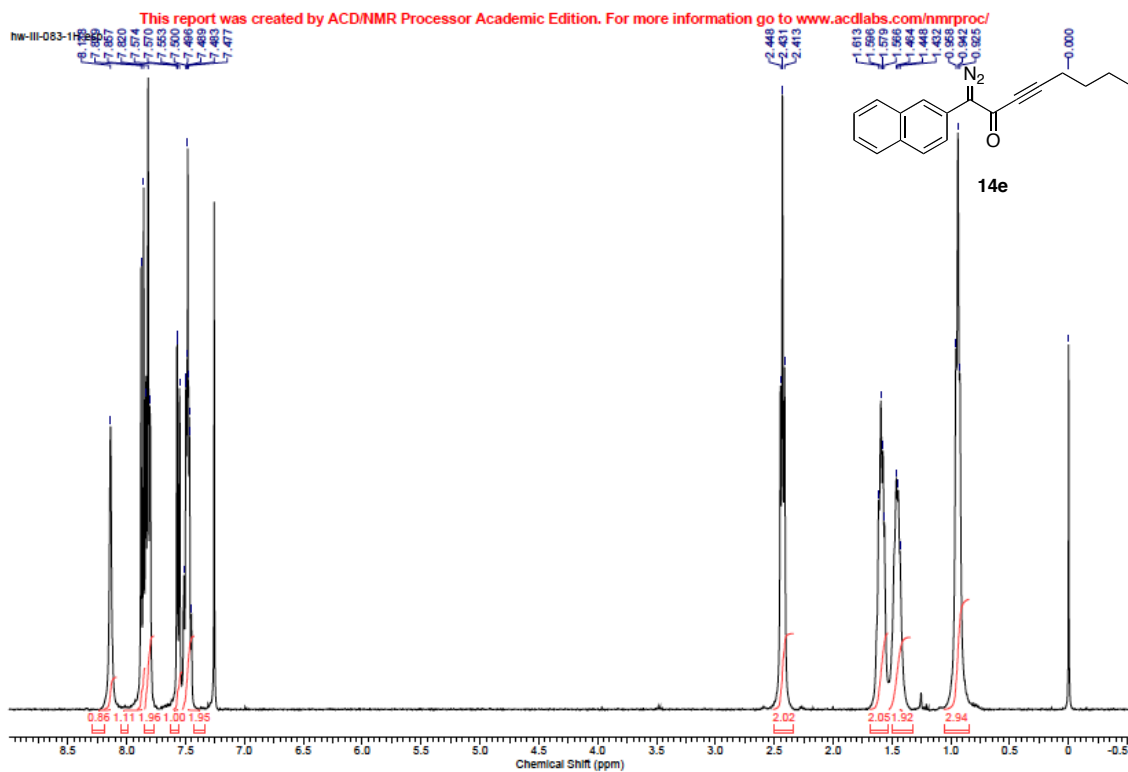
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^1H and ^{13}C spectra of compound **14c**



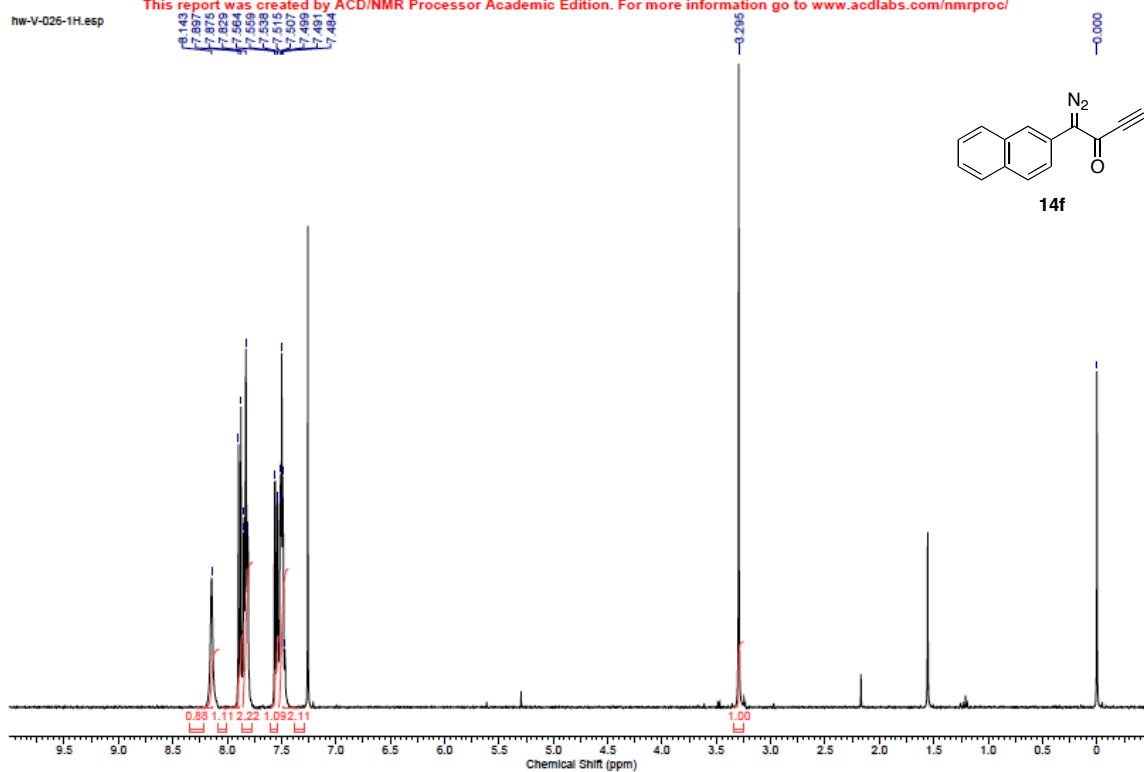
^1H and ^{13}C spectra of compound **14d**



^1H and ^{13}C spectra of compound 14e

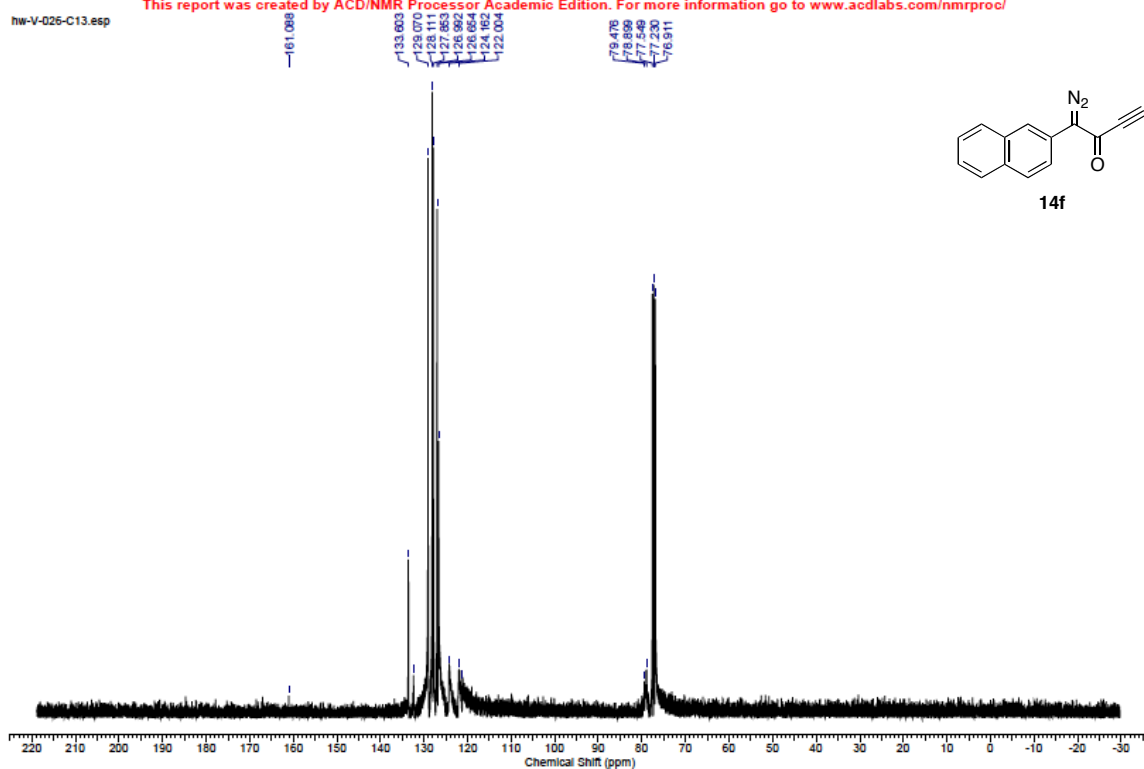
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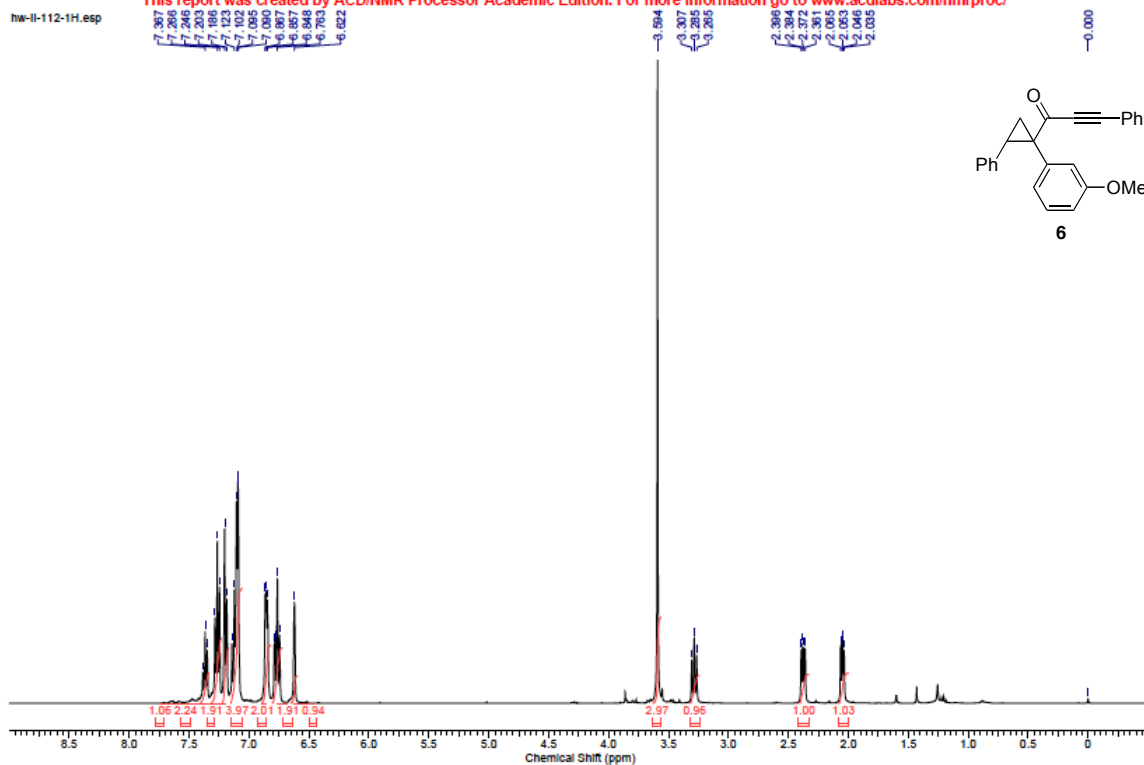
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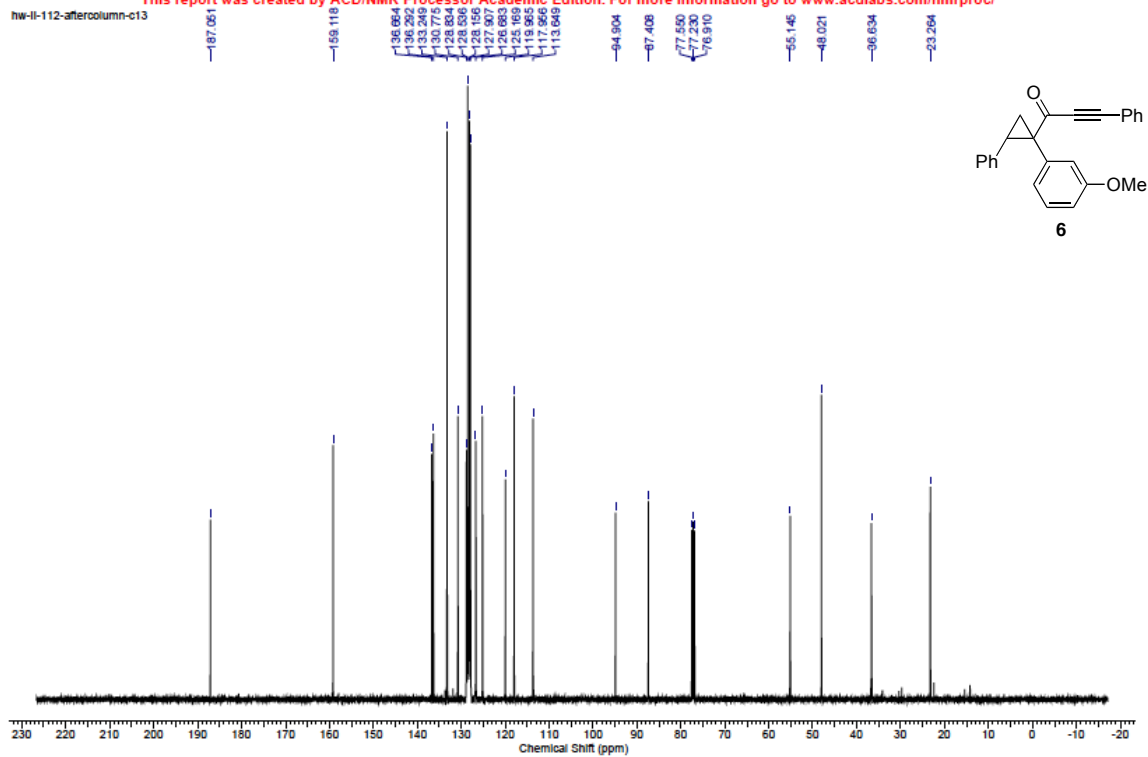
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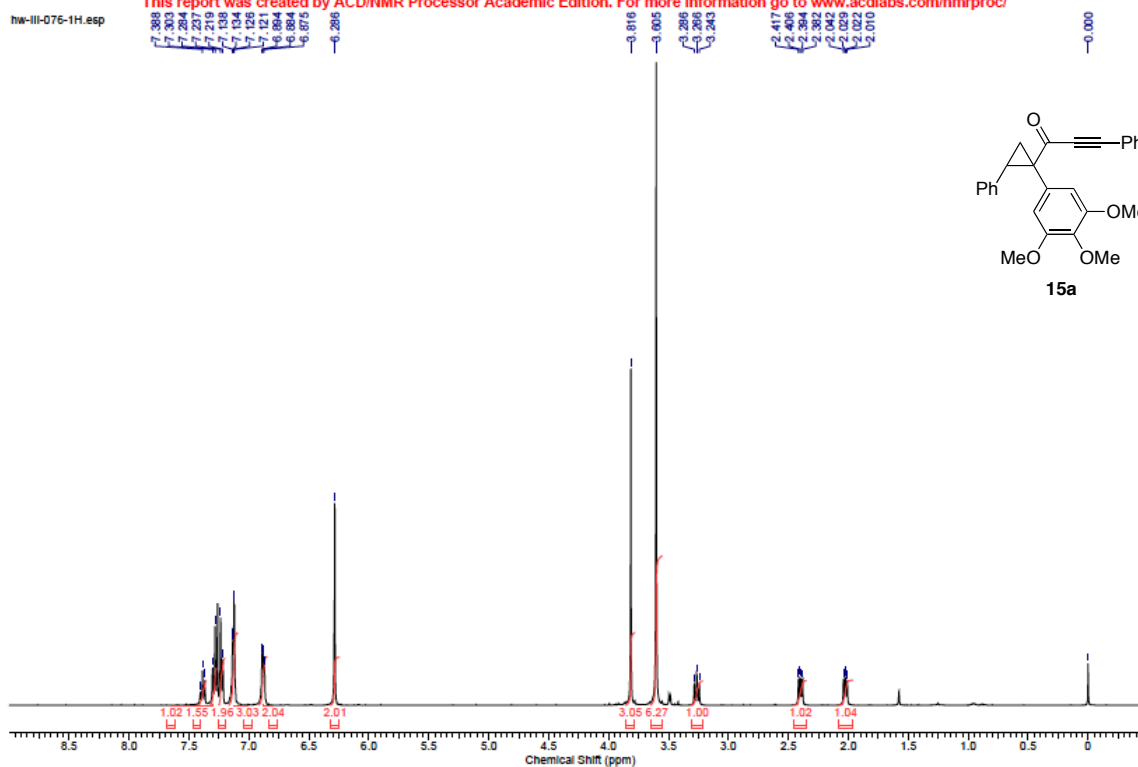
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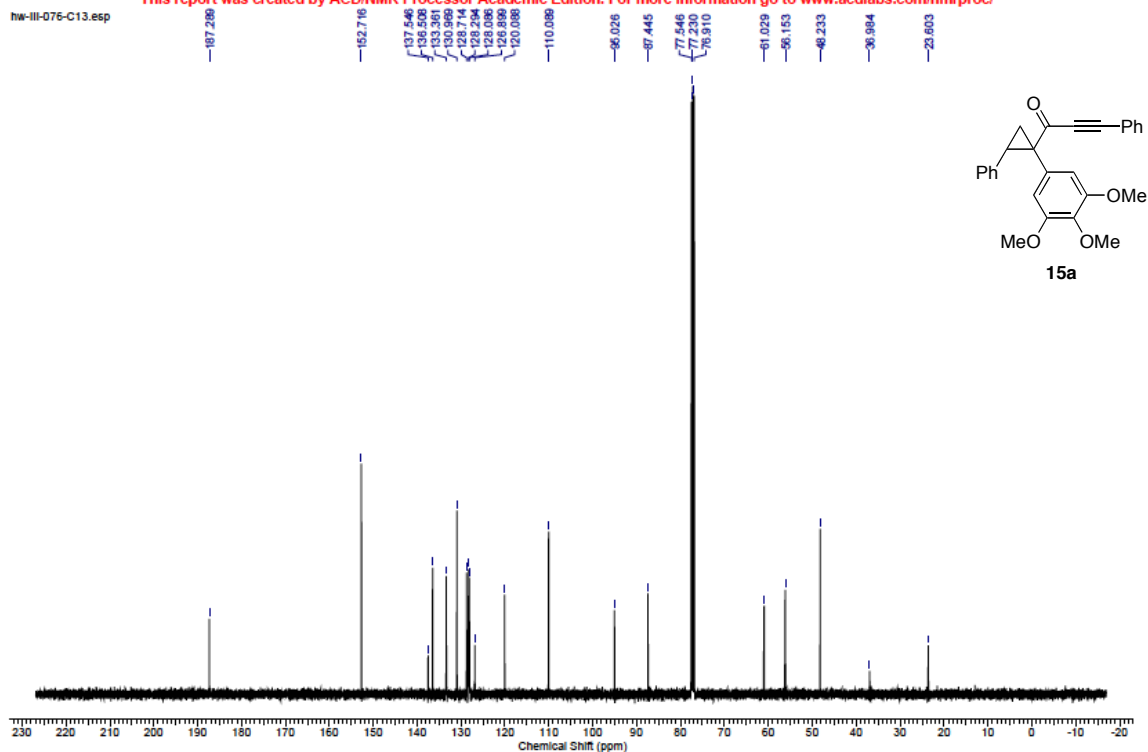
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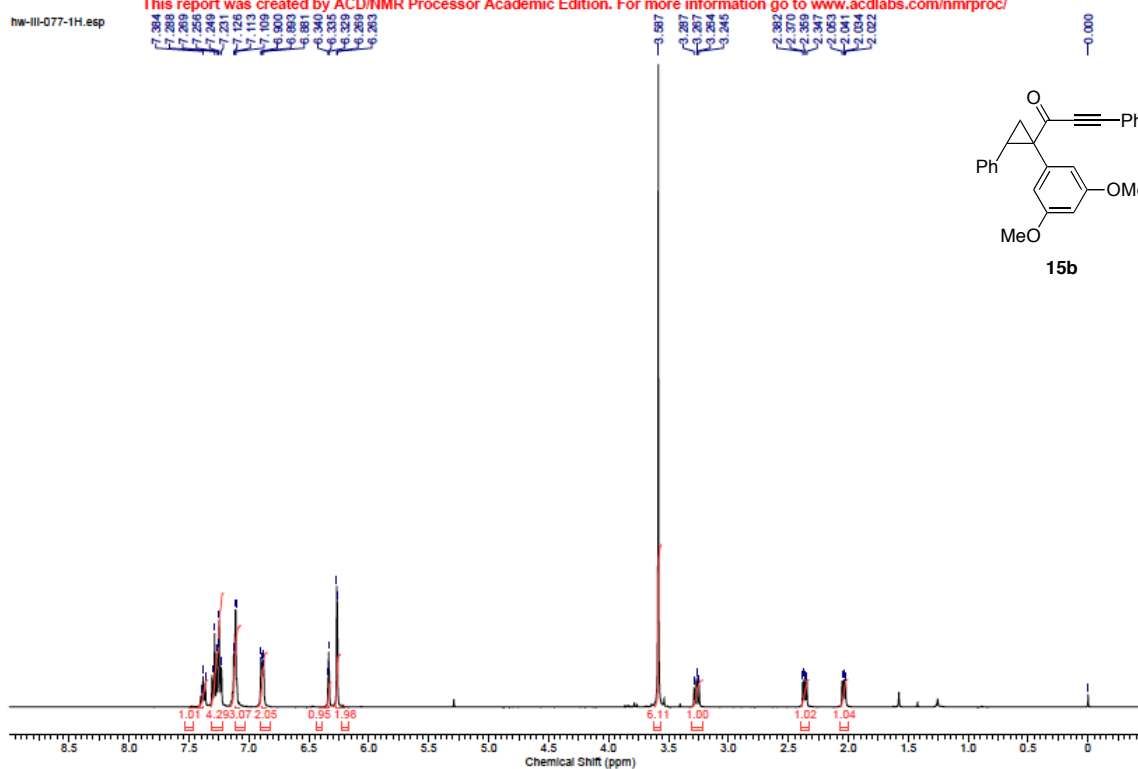
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¹H and ¹³C spectra of compound **15a**

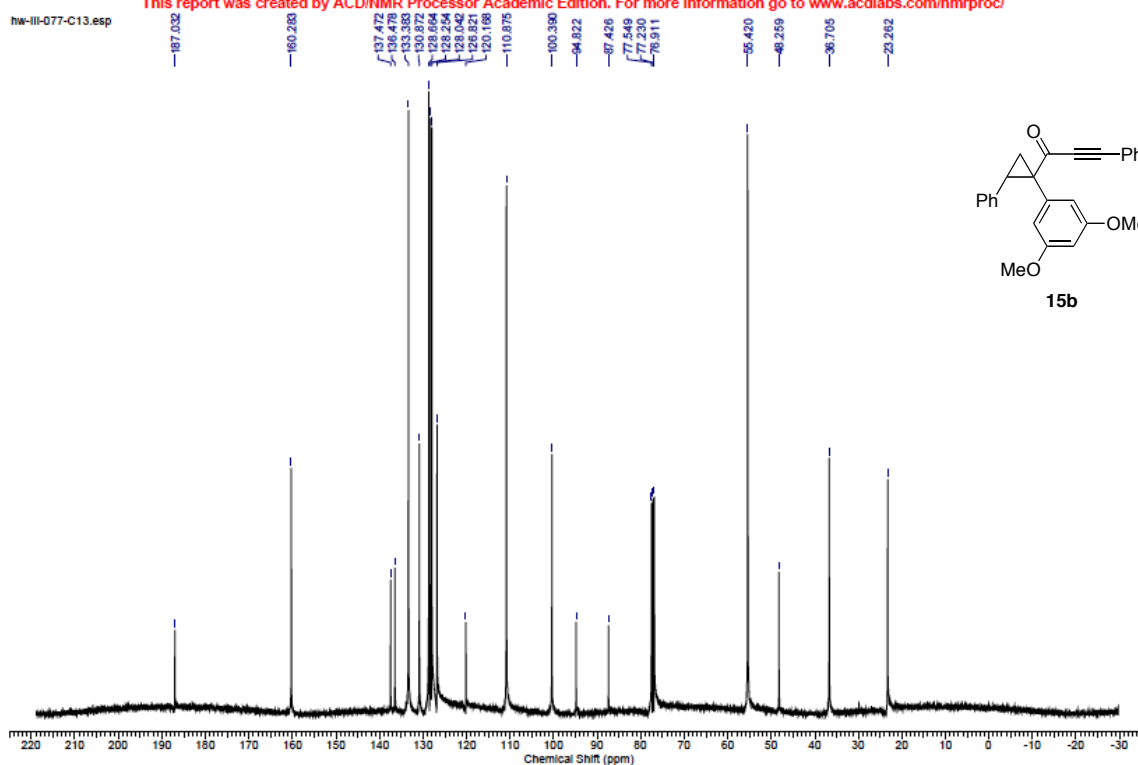
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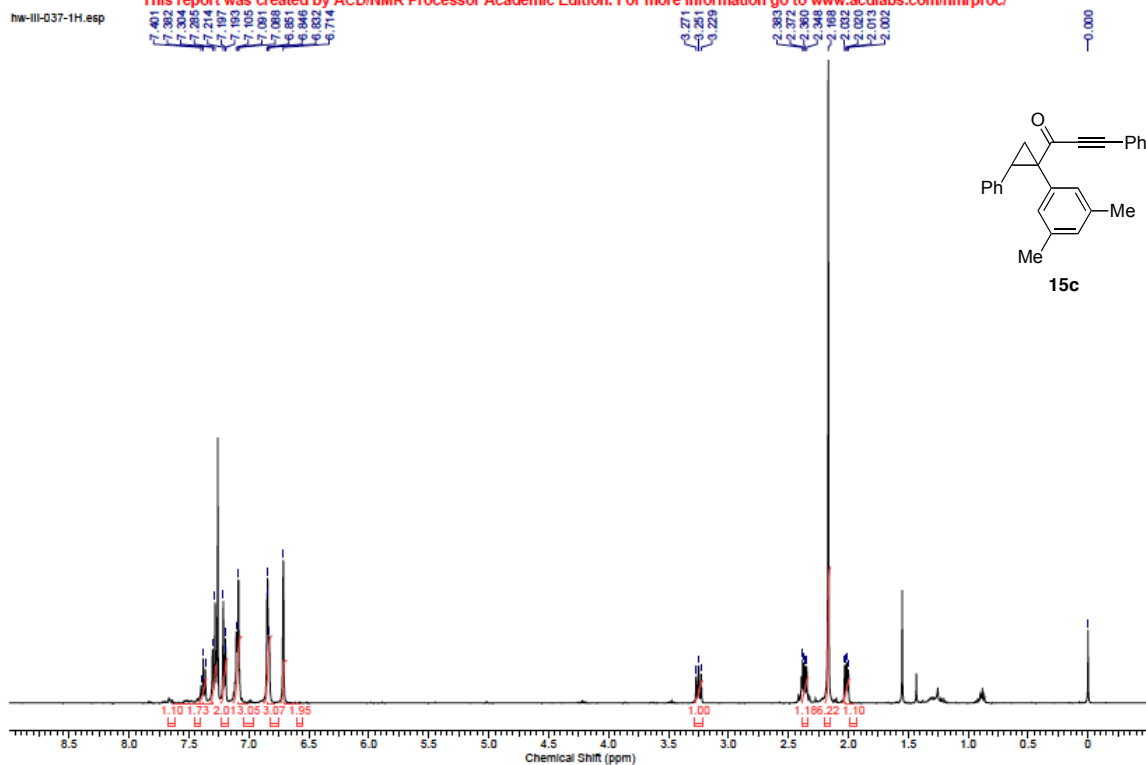
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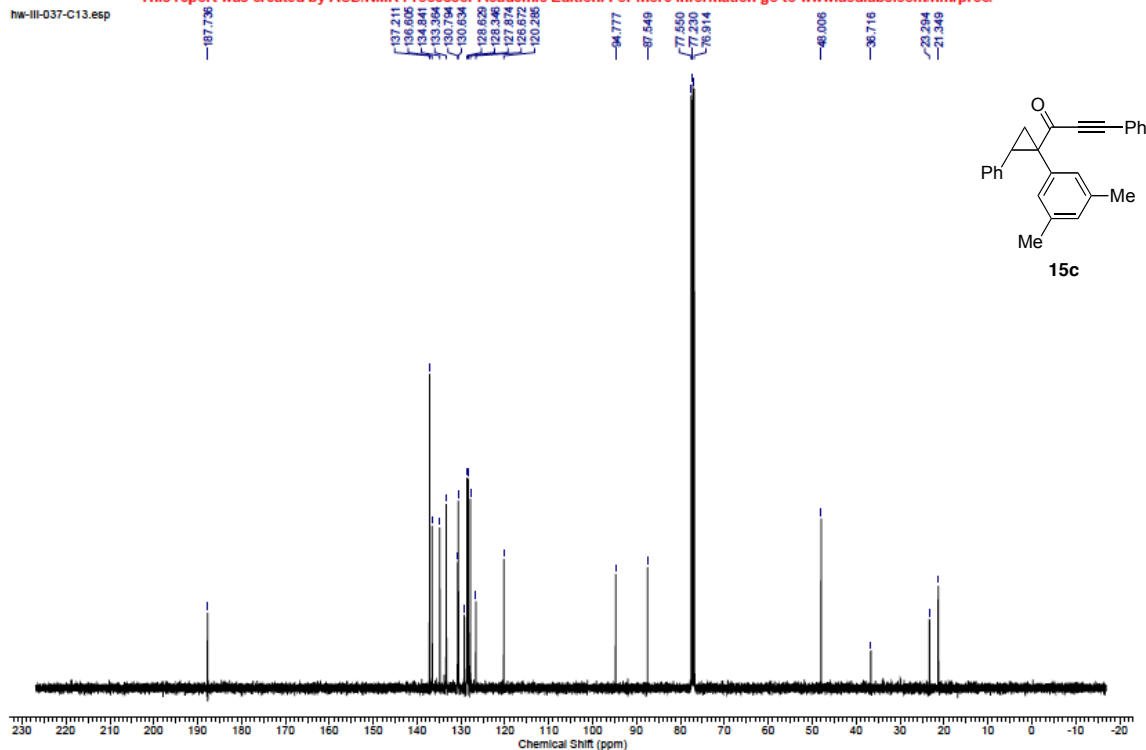
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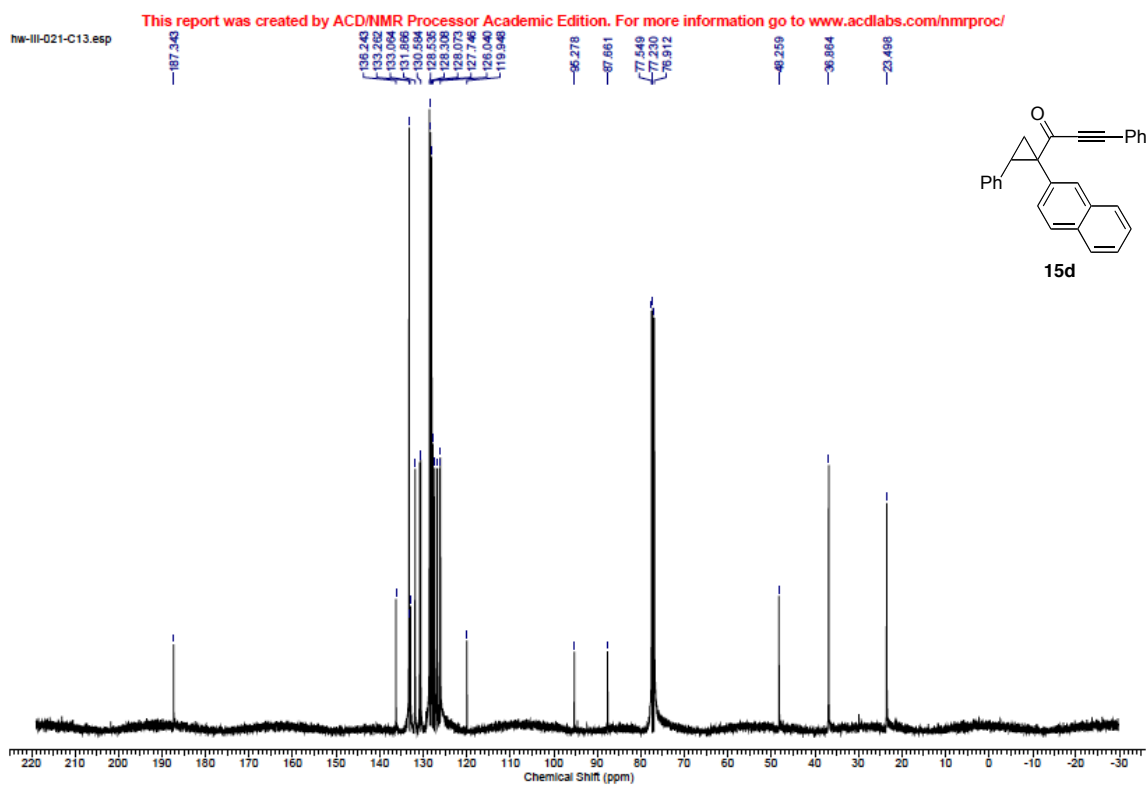
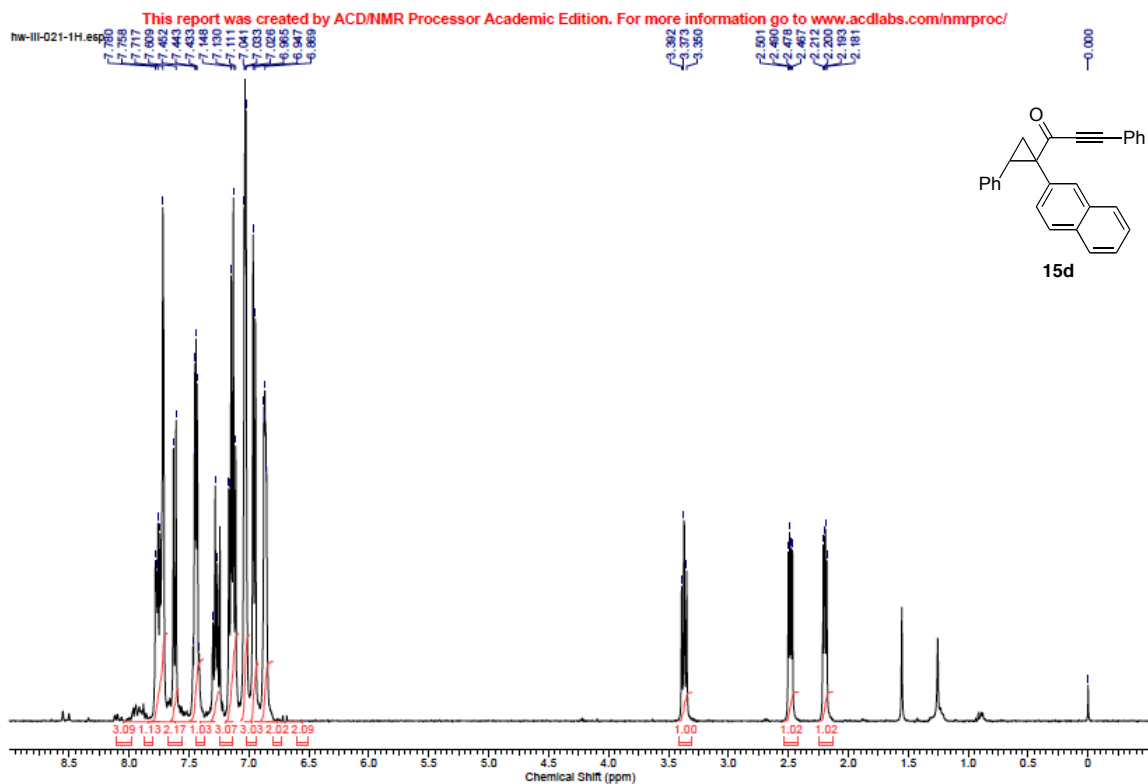


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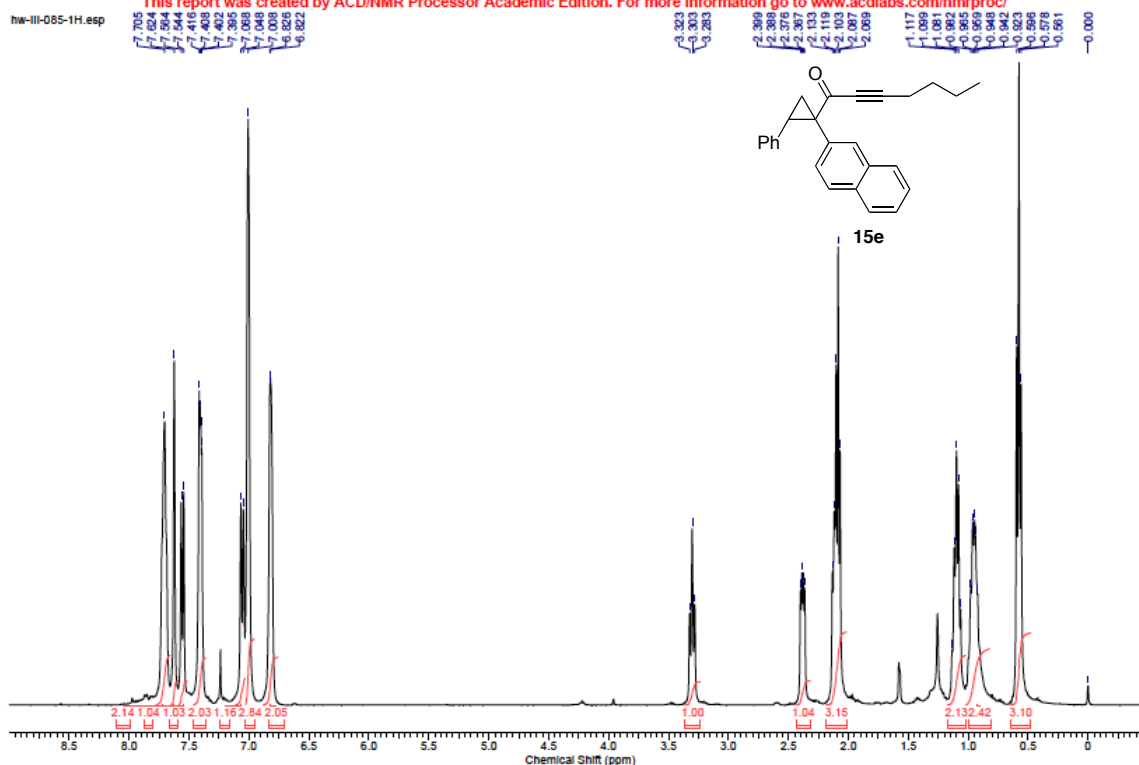


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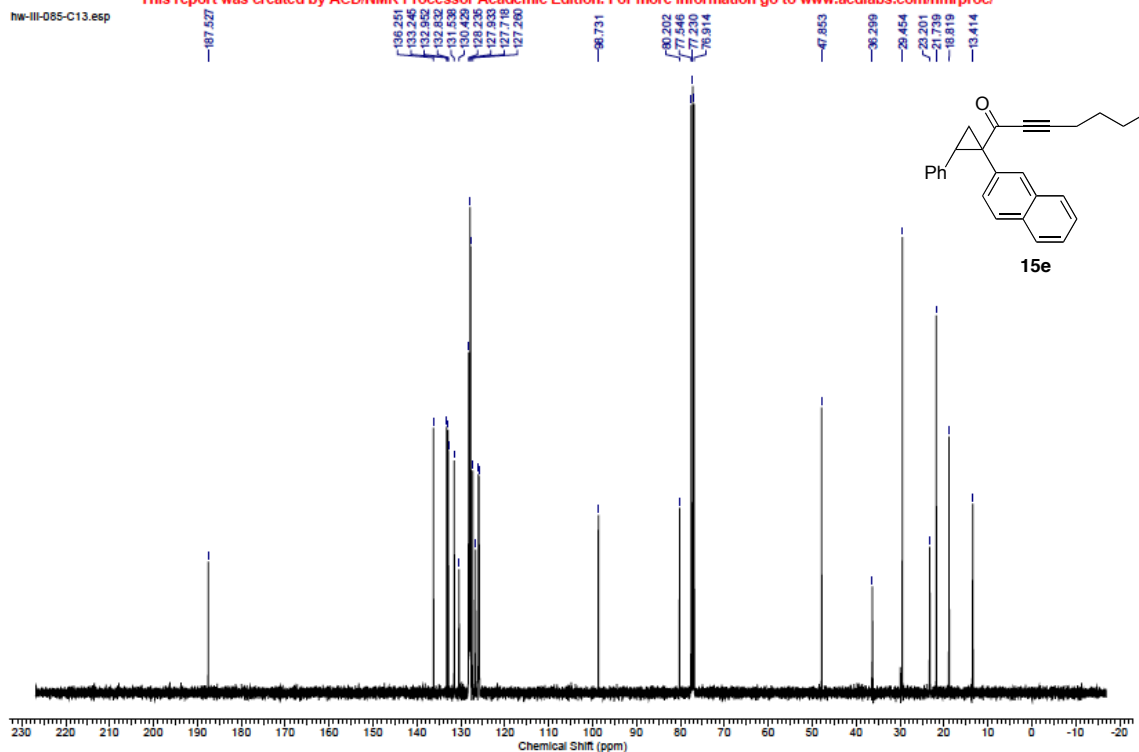


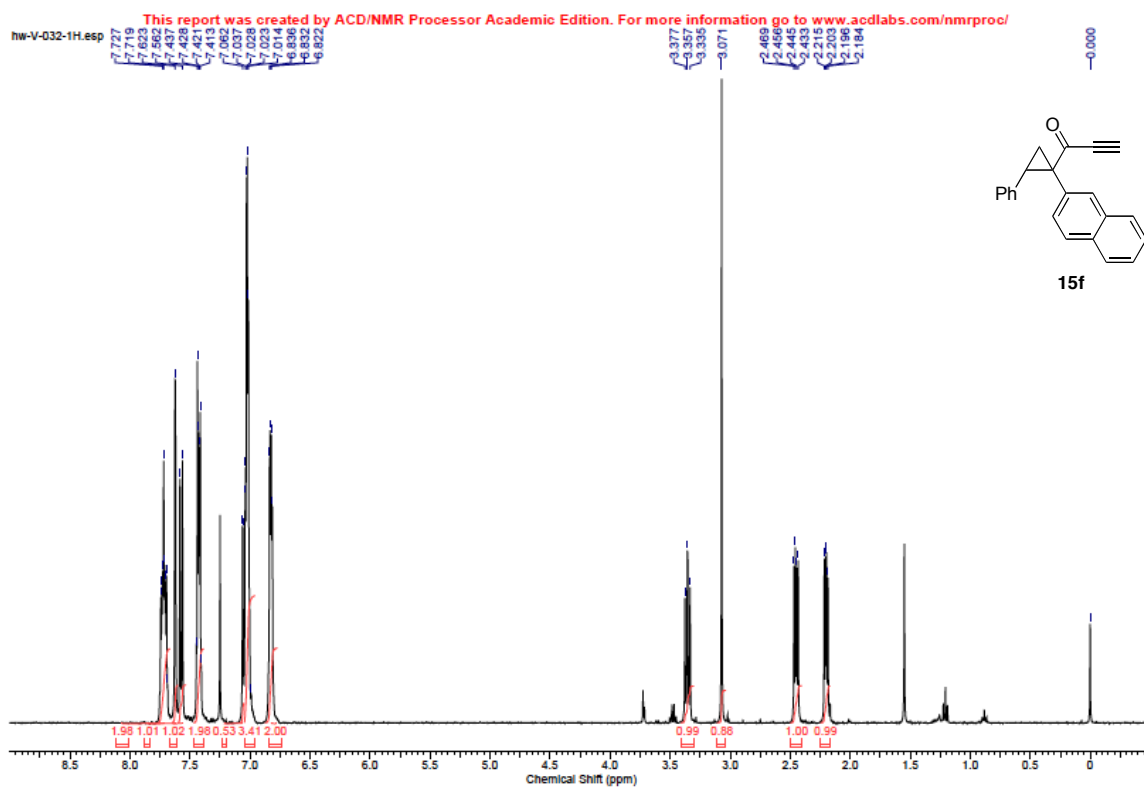
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¹H and ¹³C spectra of compound **15e**



hw-V-032-aftercolumn-c13

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

User: 1-14-87

UNITYplus-600 "up1600"

PULSE SEQUENCE

Relax. delay 2.000 sec

Pulse 69.2 degrees

Acq. time 1.000 sec

Width 31948.9 Hz

576 Repetitions

OBSERVE C13, 150.7688373 MHz

DECOUPLE H1, 509.5999983 MHz

Power 36 dB

continuously on

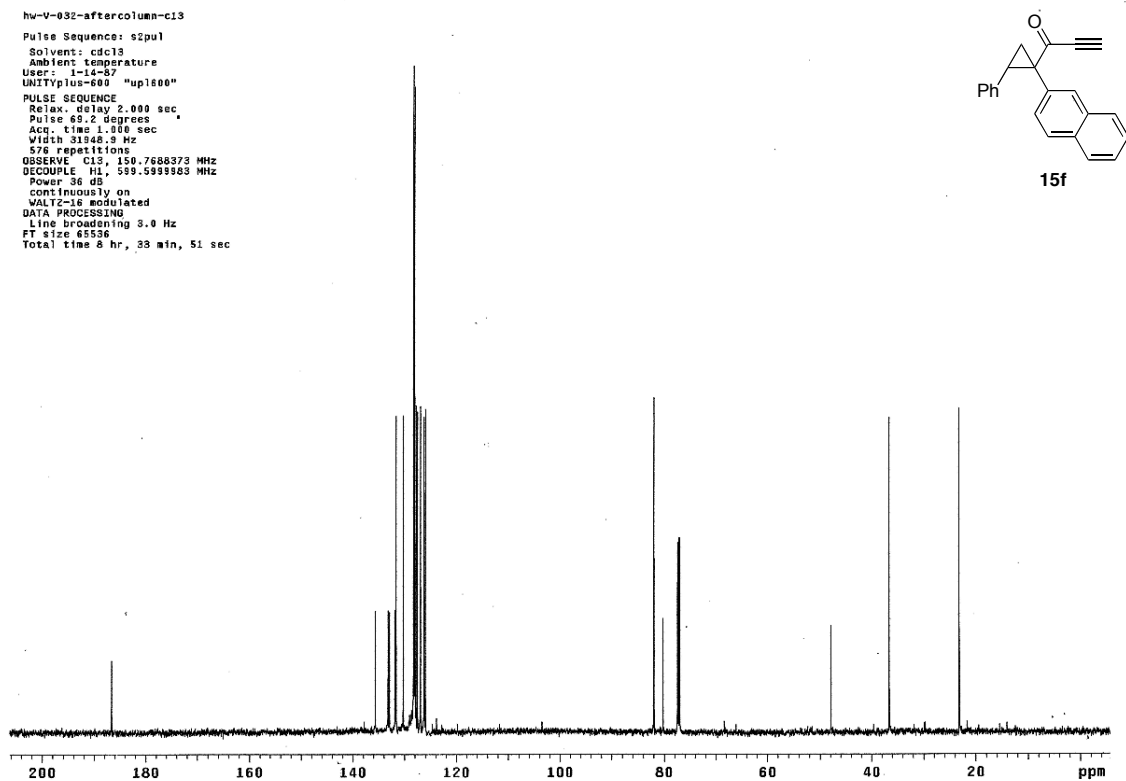
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DATA PROCESSING

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FT size 65536

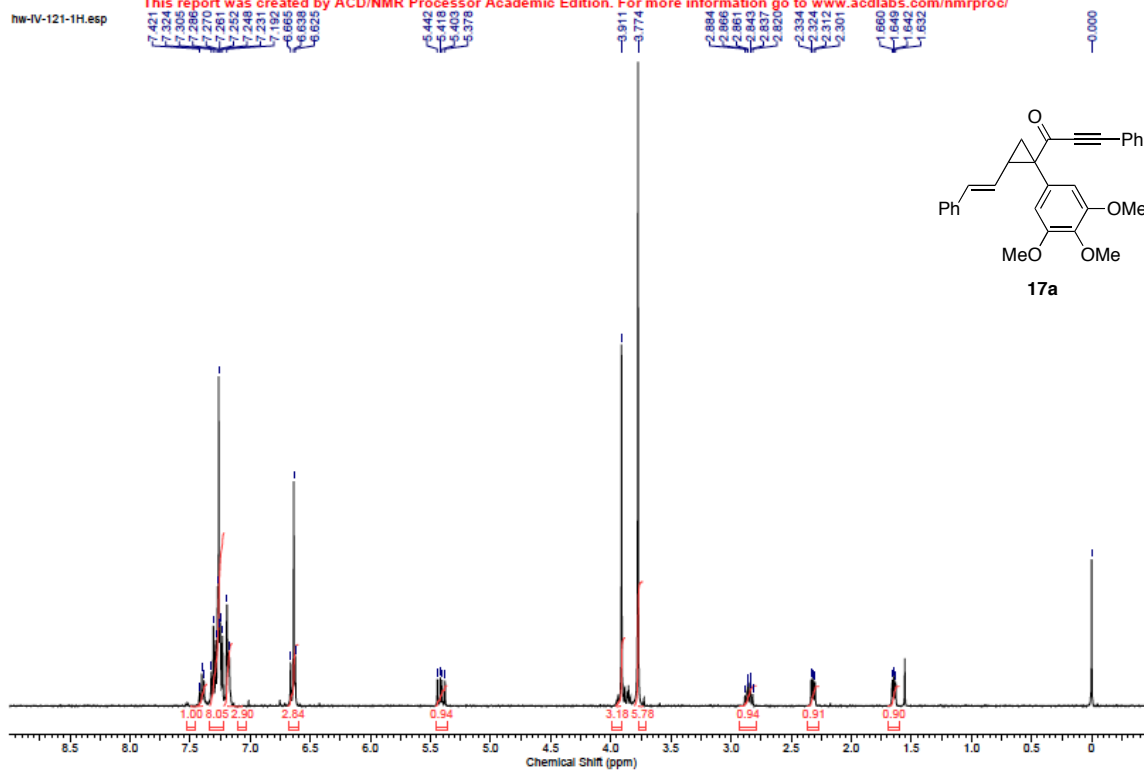
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^1H and ^{13}C spectra of compound **15f**

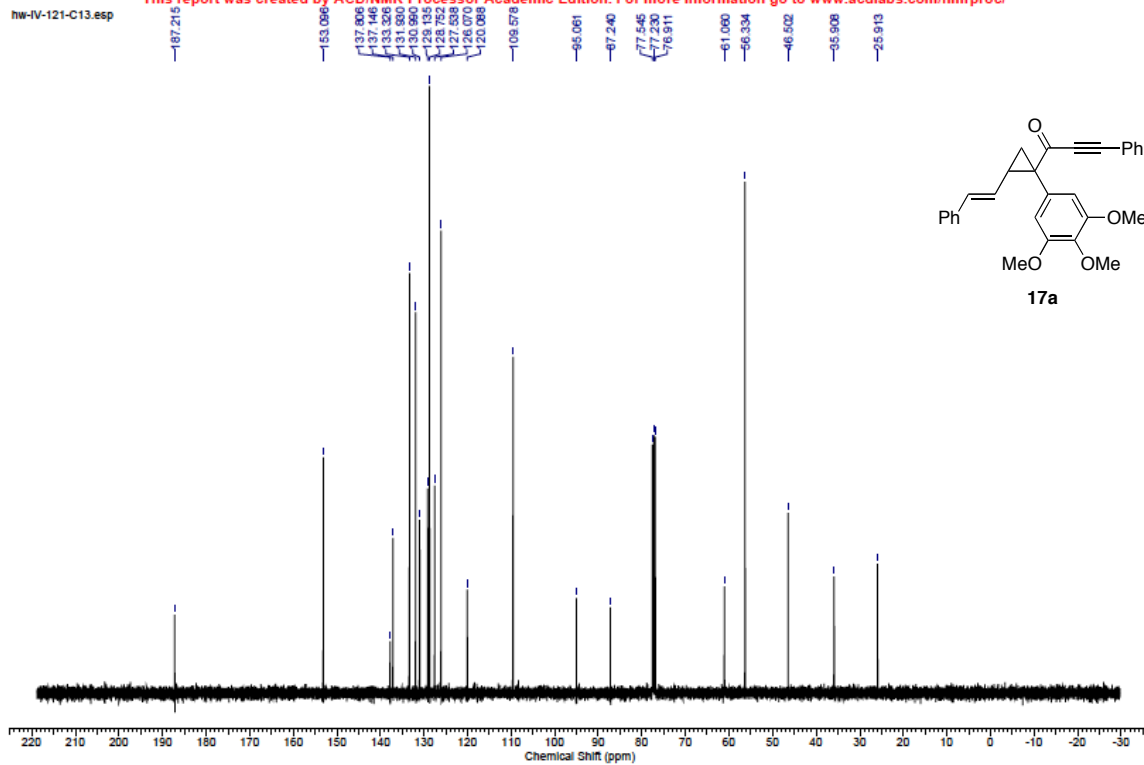
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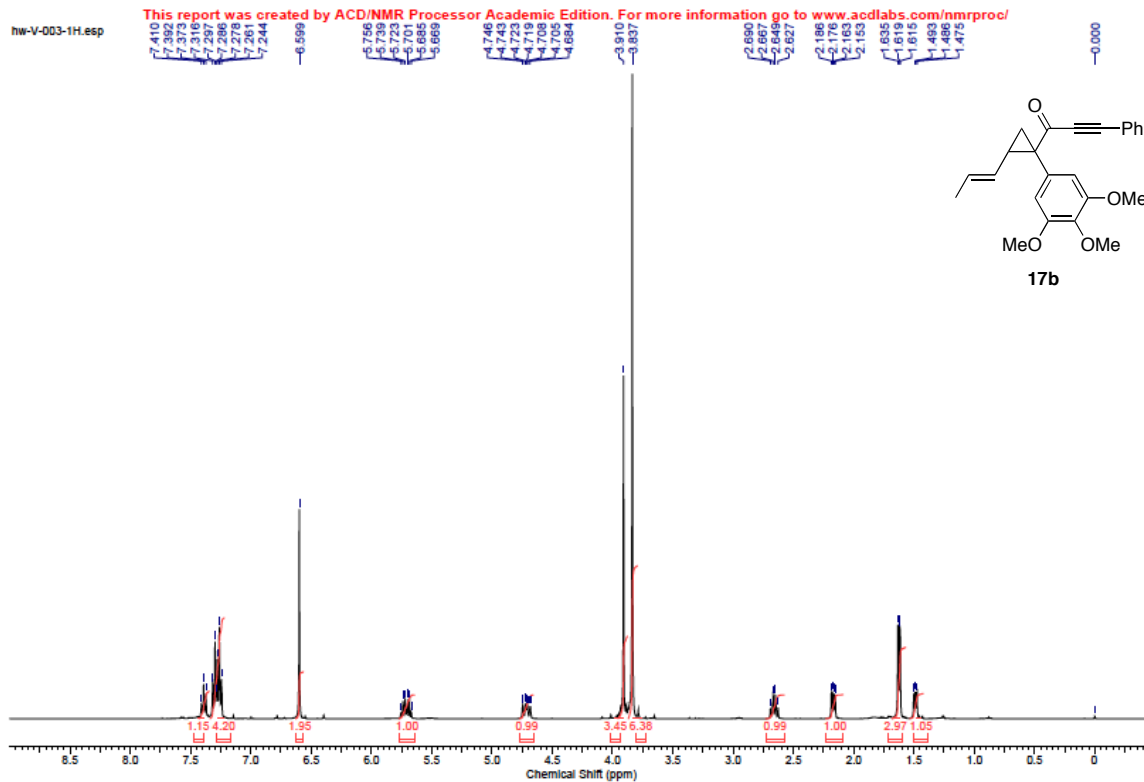
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¹H and ¹³C spectra of compound 17a

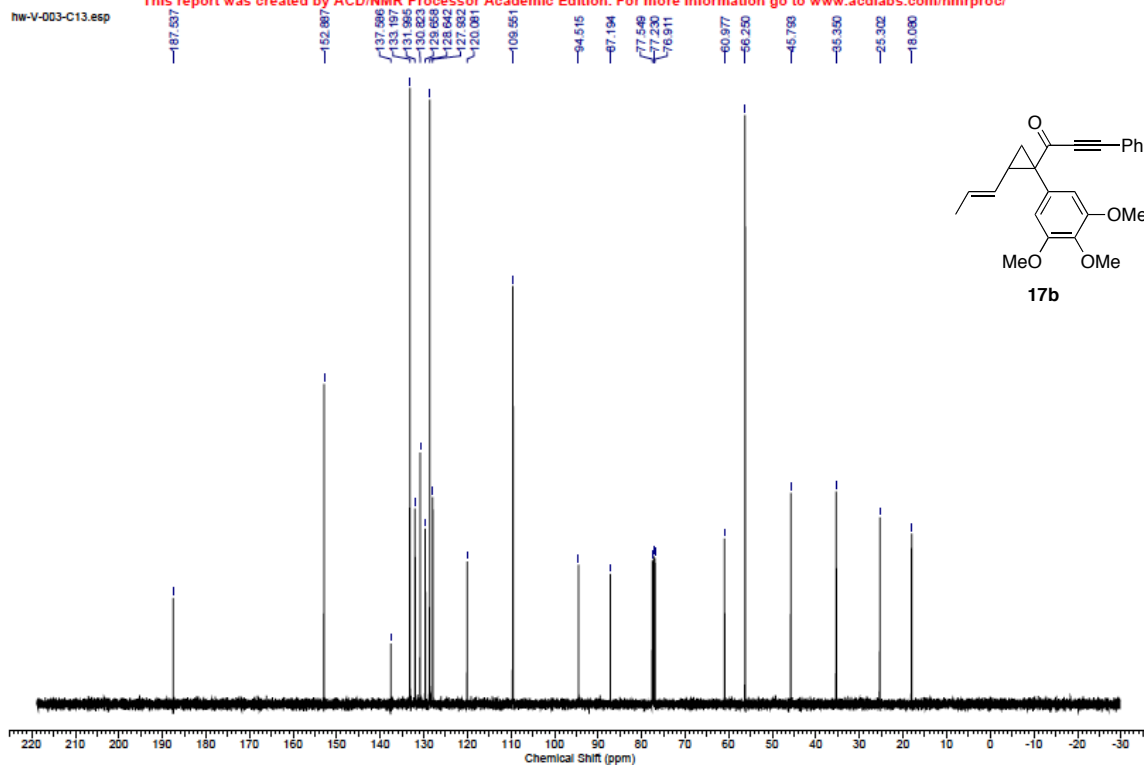
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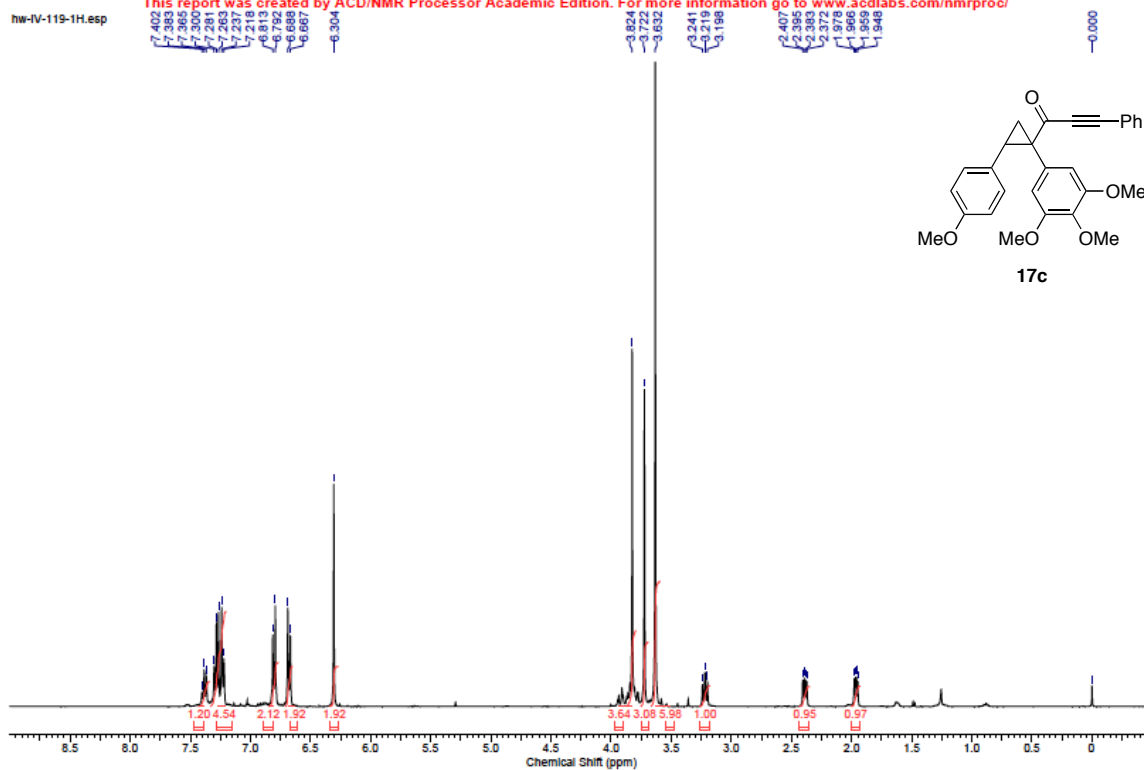
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¹H and ¹³C spectra of compound 17b

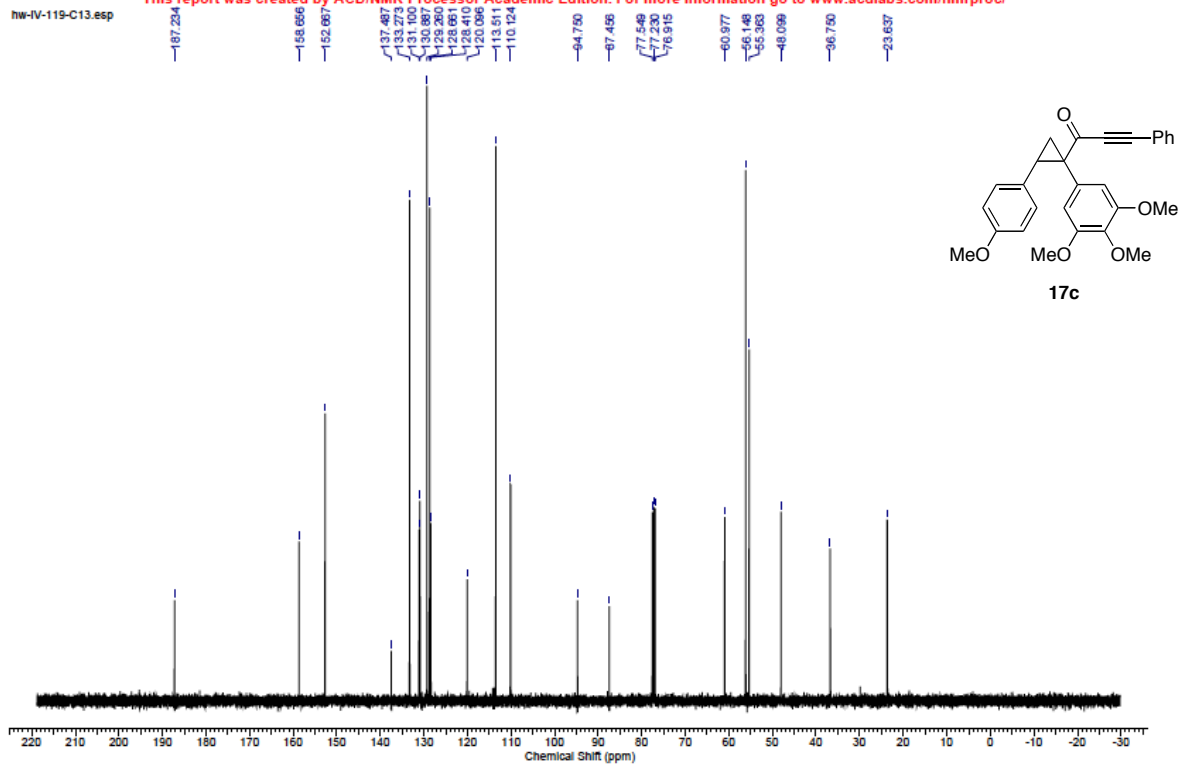
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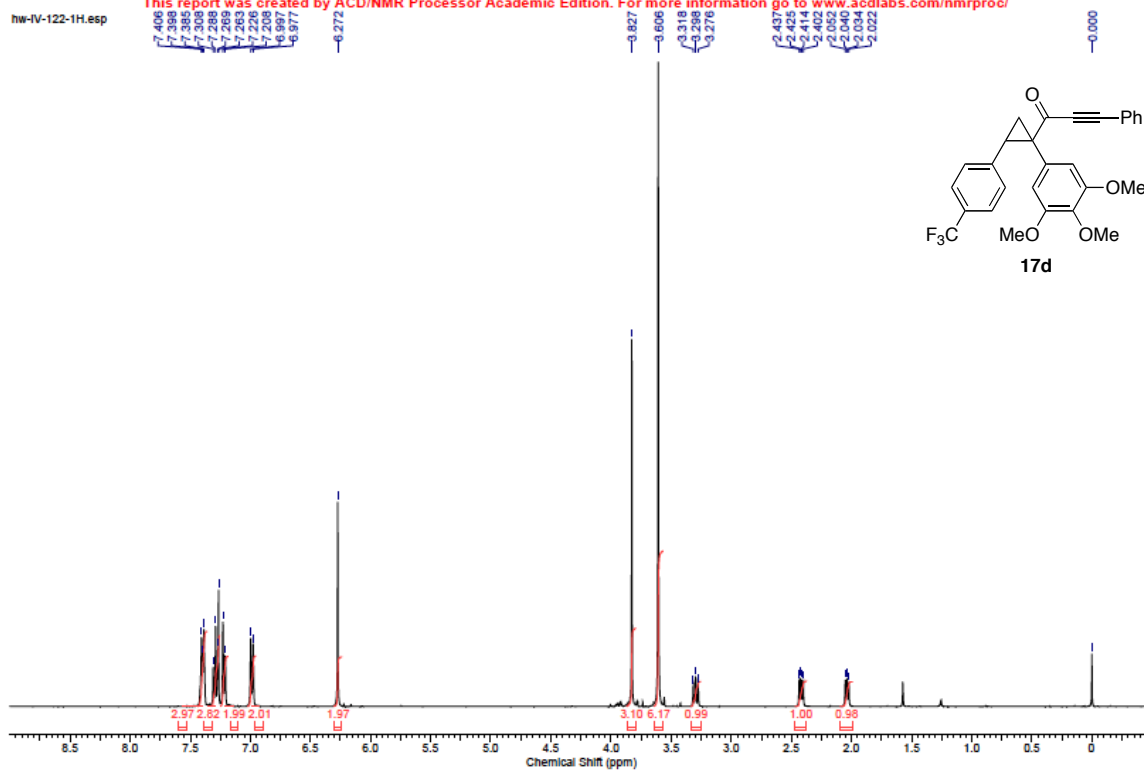
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¹H and ¹³C spectra of compound 17c

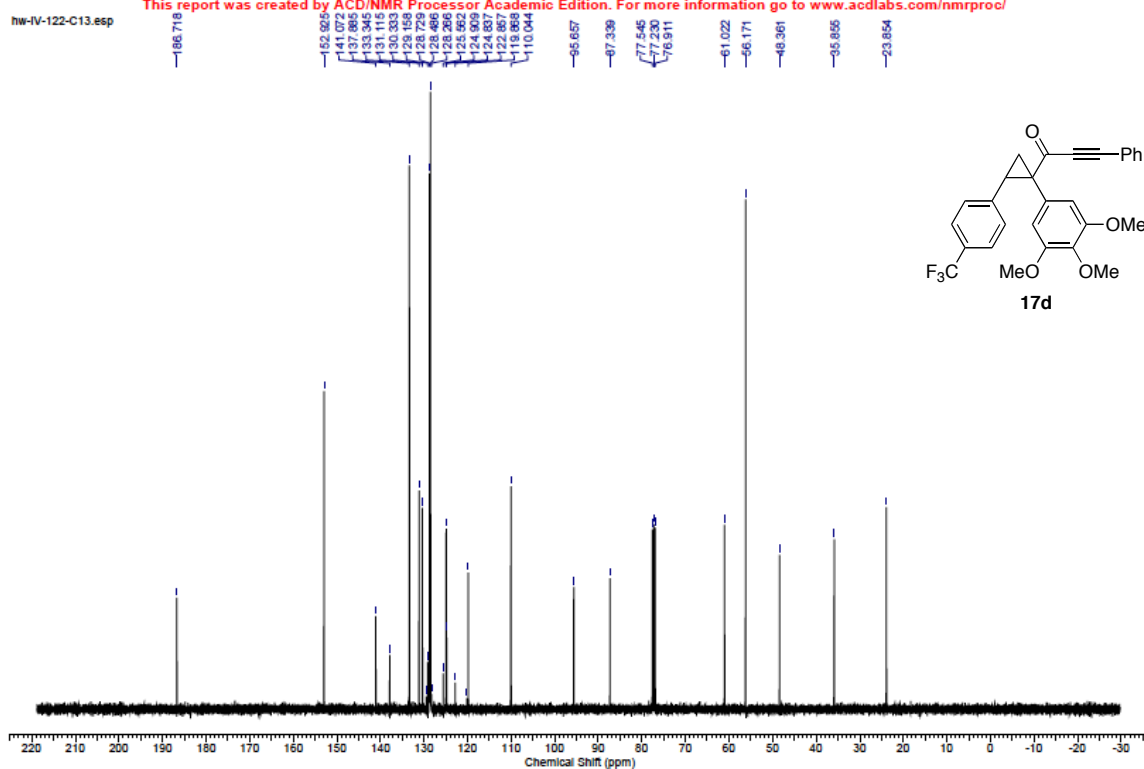
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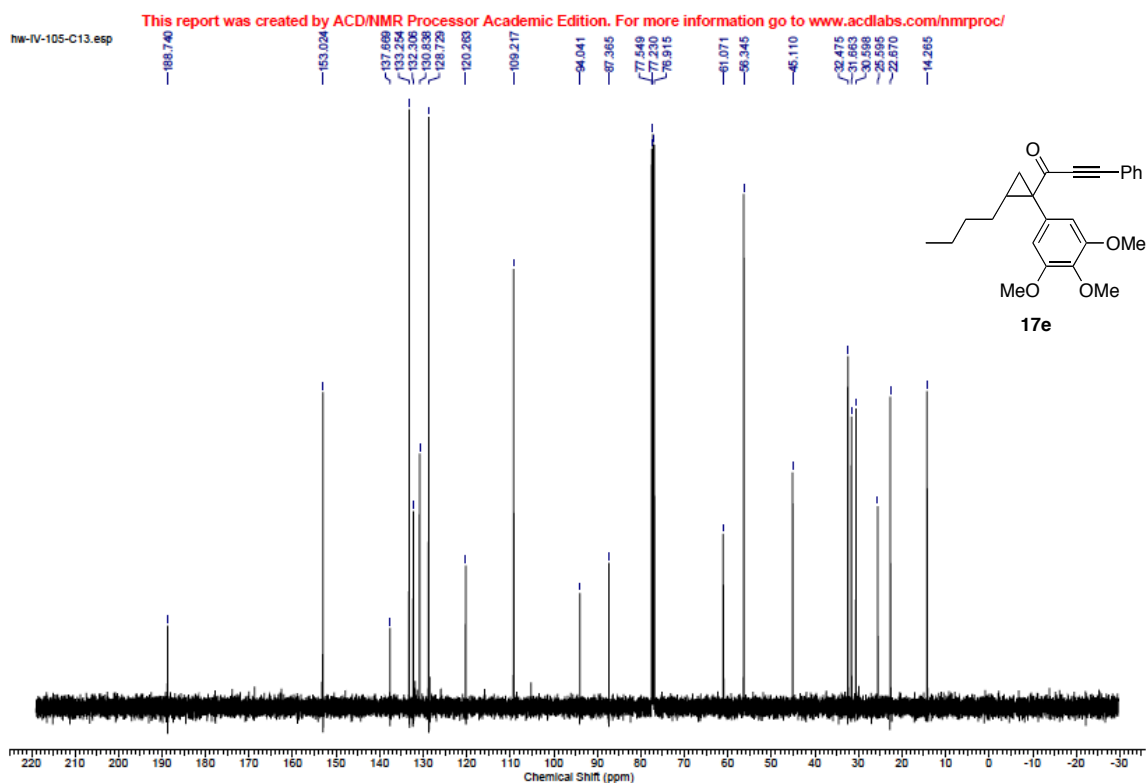
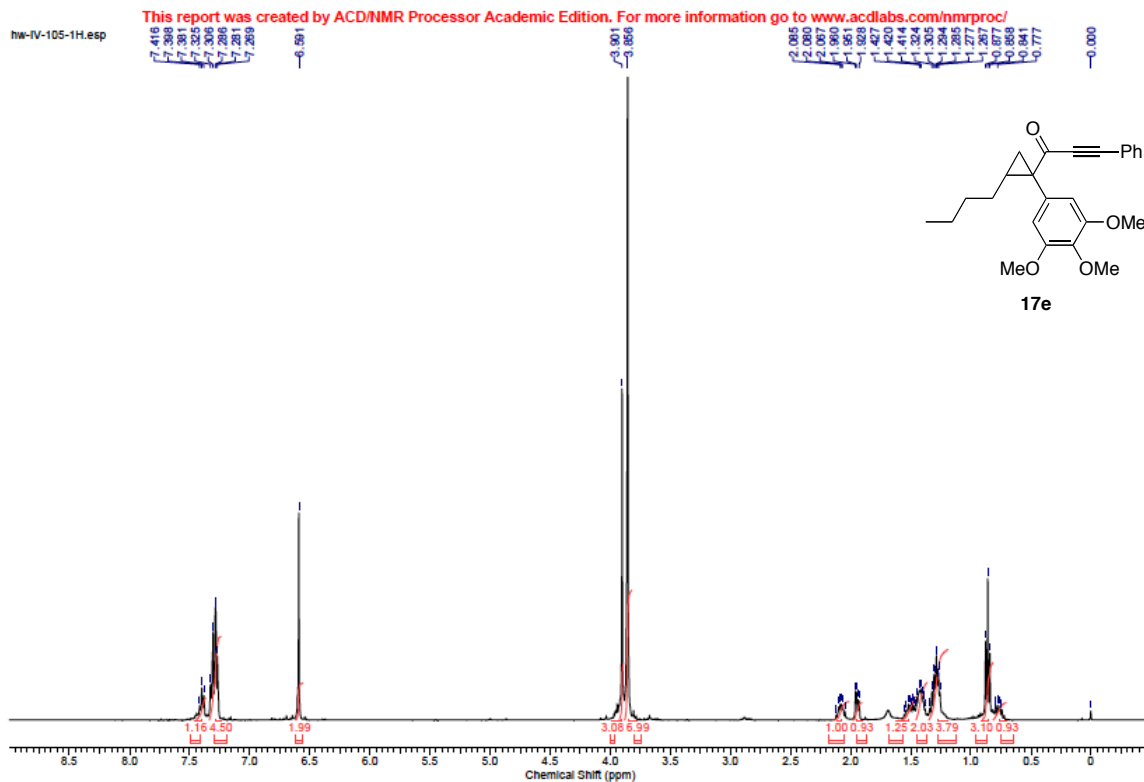


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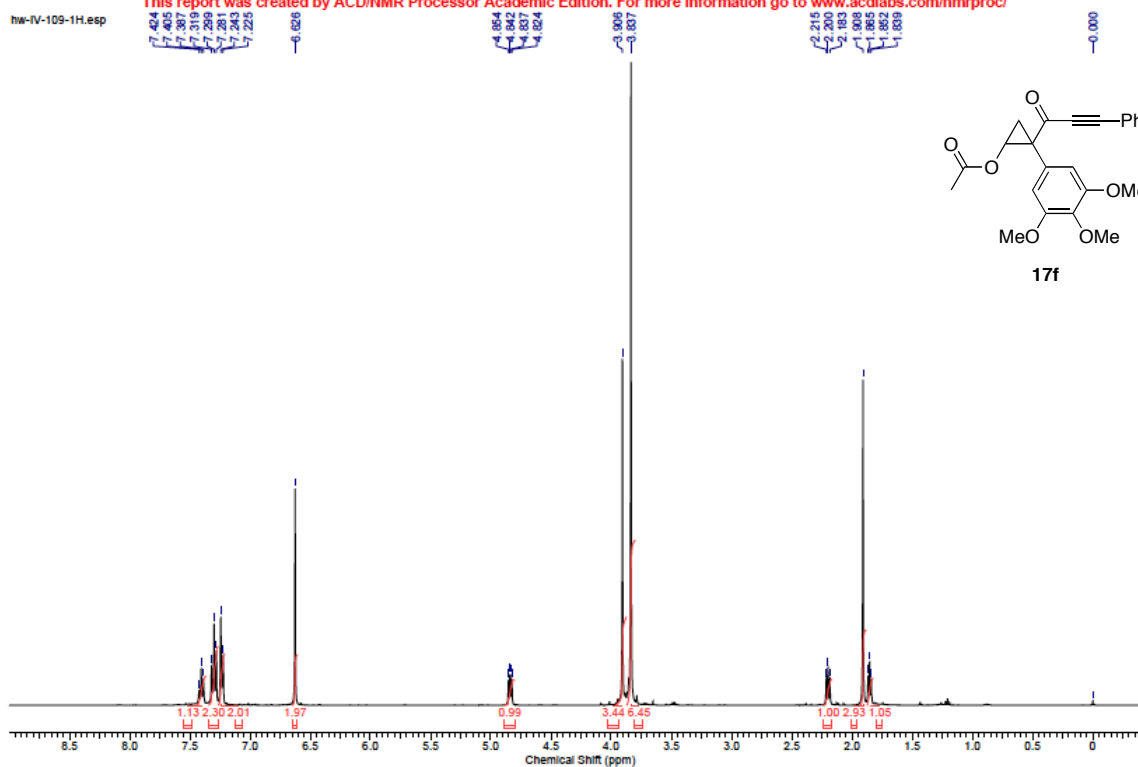
¹H and ¹³C spectra of compound 17d



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

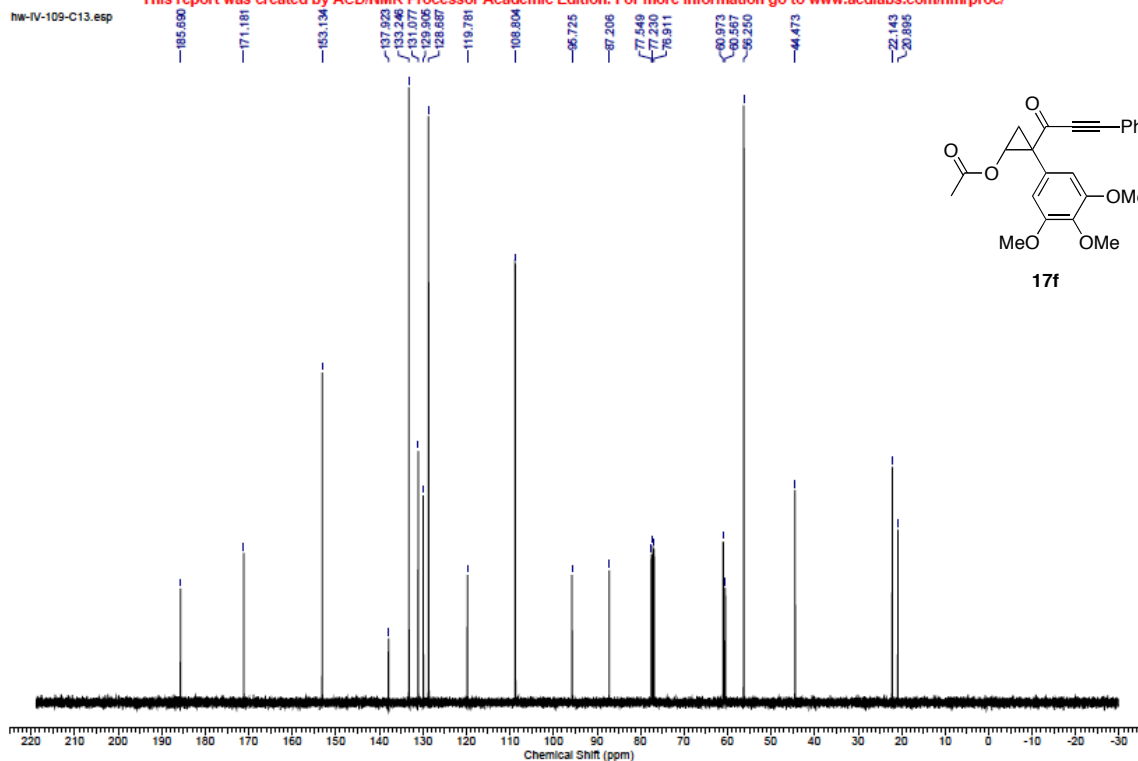
hw-IV-109-1H.esp

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hw-IV-109-C13.esp

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



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

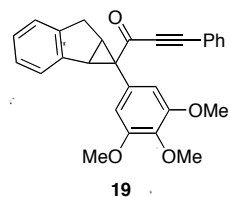
hw-IV-102-after:column
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
INOVA-400 "1600data"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 31.5 degrees
Acq. time 4.555 sec
Width 4599.4 Hz
8 repetitions
OBSERVE H₁, 299.9258712 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 8 min, 28 sec

COc1cc(C(=O)C#CC23C4C=CC=CC=C4C23)c(OC)c(O)c1

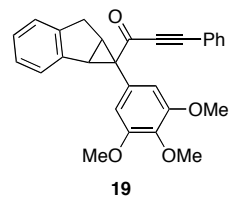
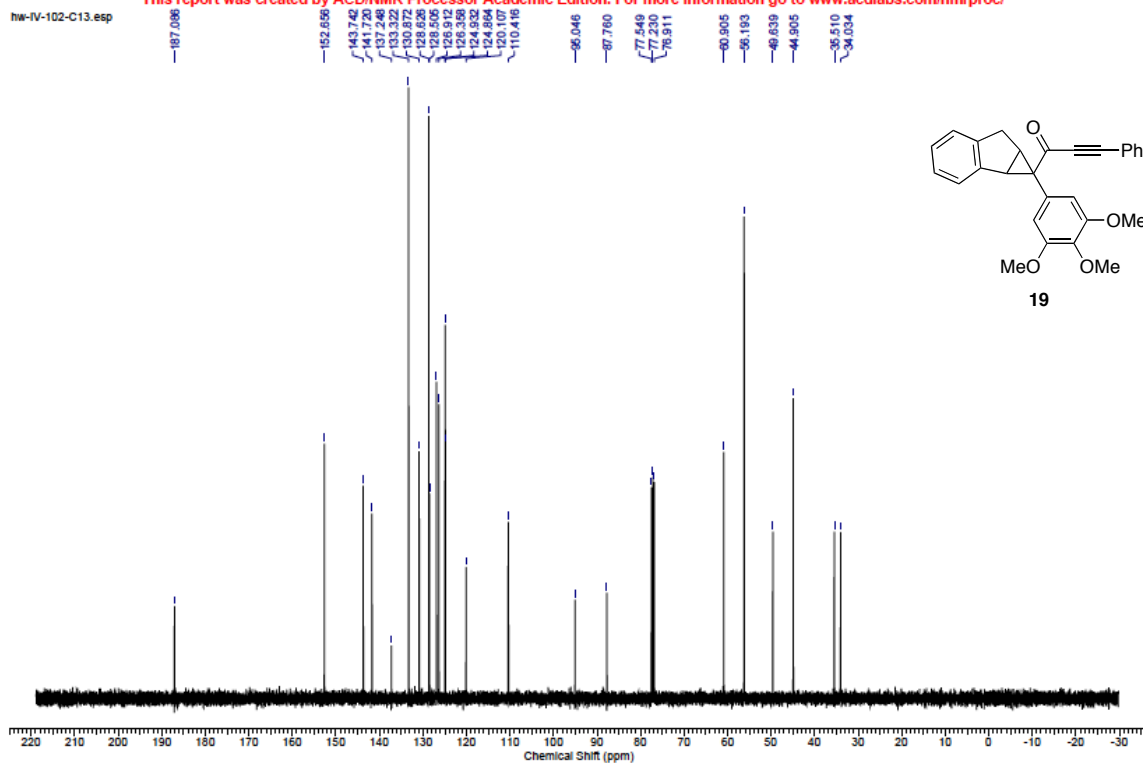
19

11 10 9 8 7 6 5 4 3 2 1 ppm

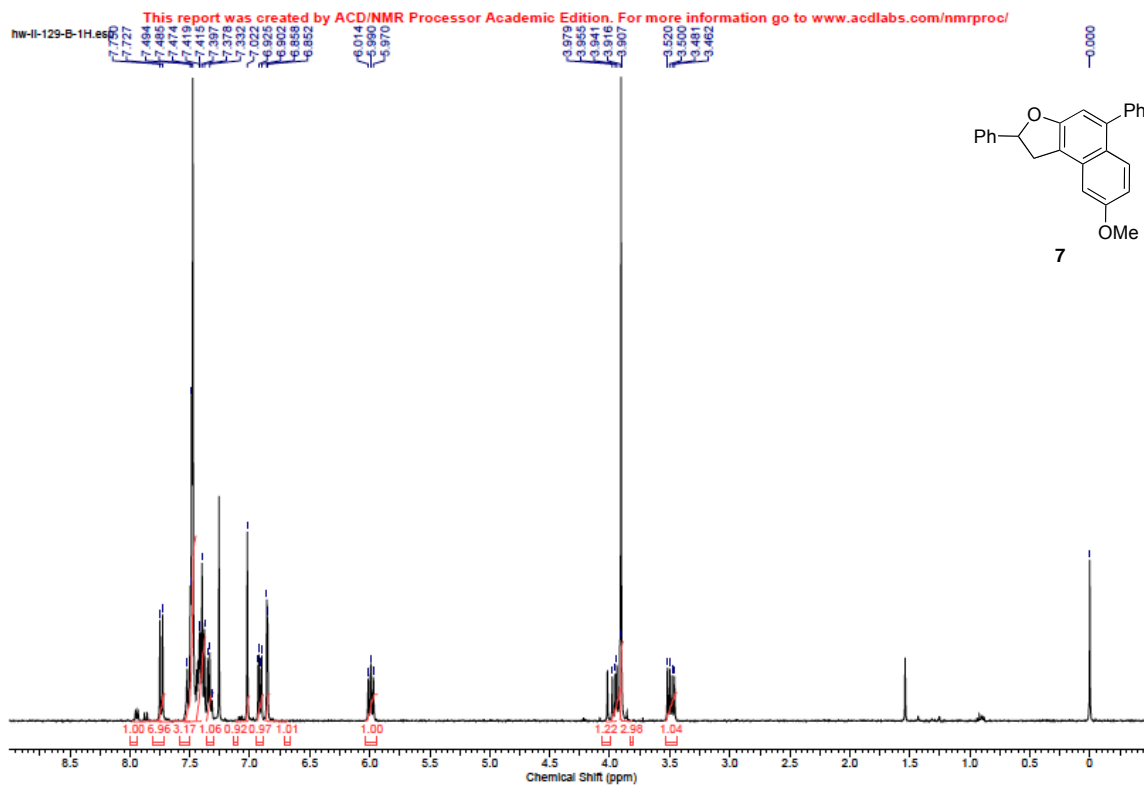
1.00 0.94 1.04 1.29 0.23 0.97 1.06 1.19 1.18



hw-IV-102-C13.esp



SI-76



hw-129-aftercolumn-B-013

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

Pulse Sequence: s2pul

Solvent: cdcl3

Temp: 23.0 C / 296.1 K

Operator: davies

VNMR-400 "v400"

Relax. delay 2.500 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 24509.8 Hz

224 repetitions

OBSERVE C13, 100.5265364 MHz

DECOUPLE H1, 399.7869220 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

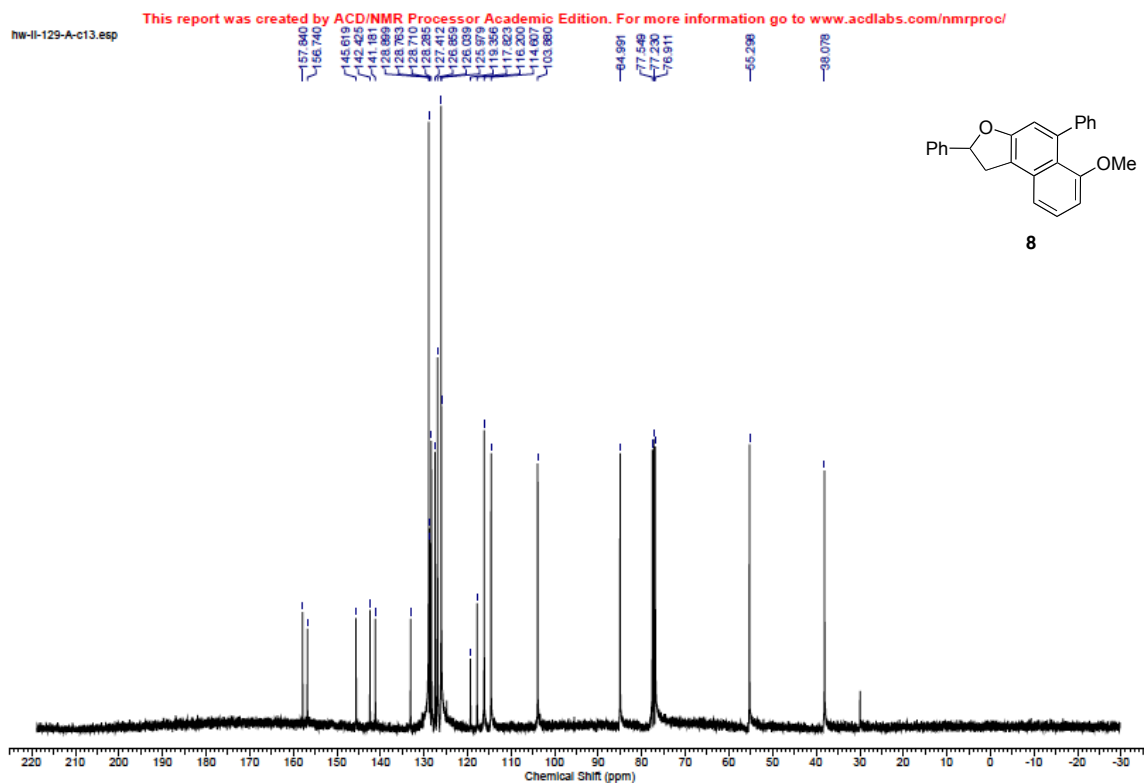
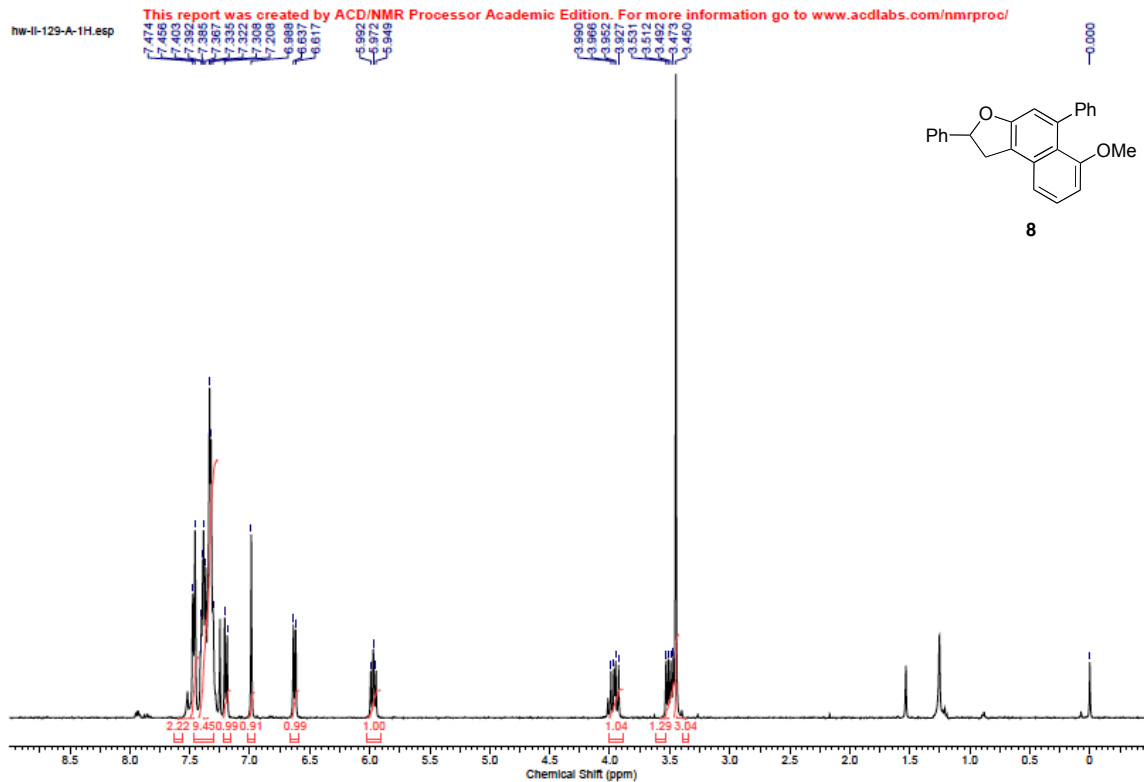
DATA PROCESSING

Line broadening 2.0 Hz

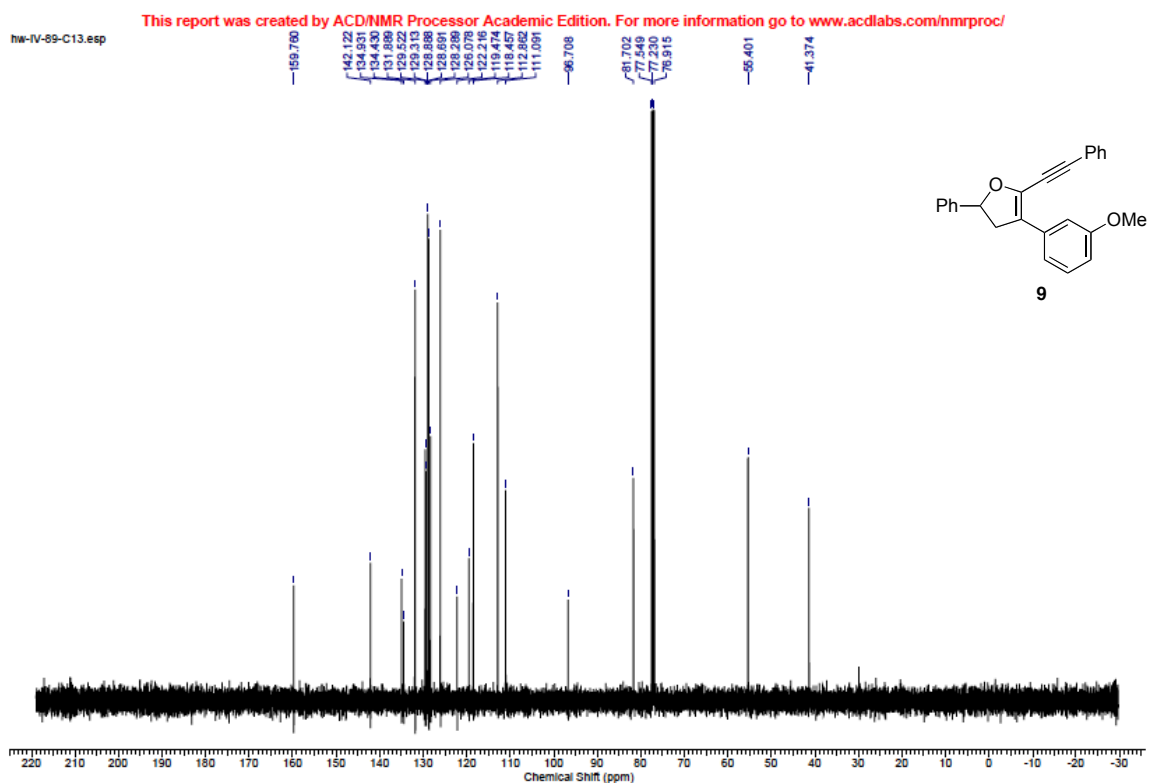
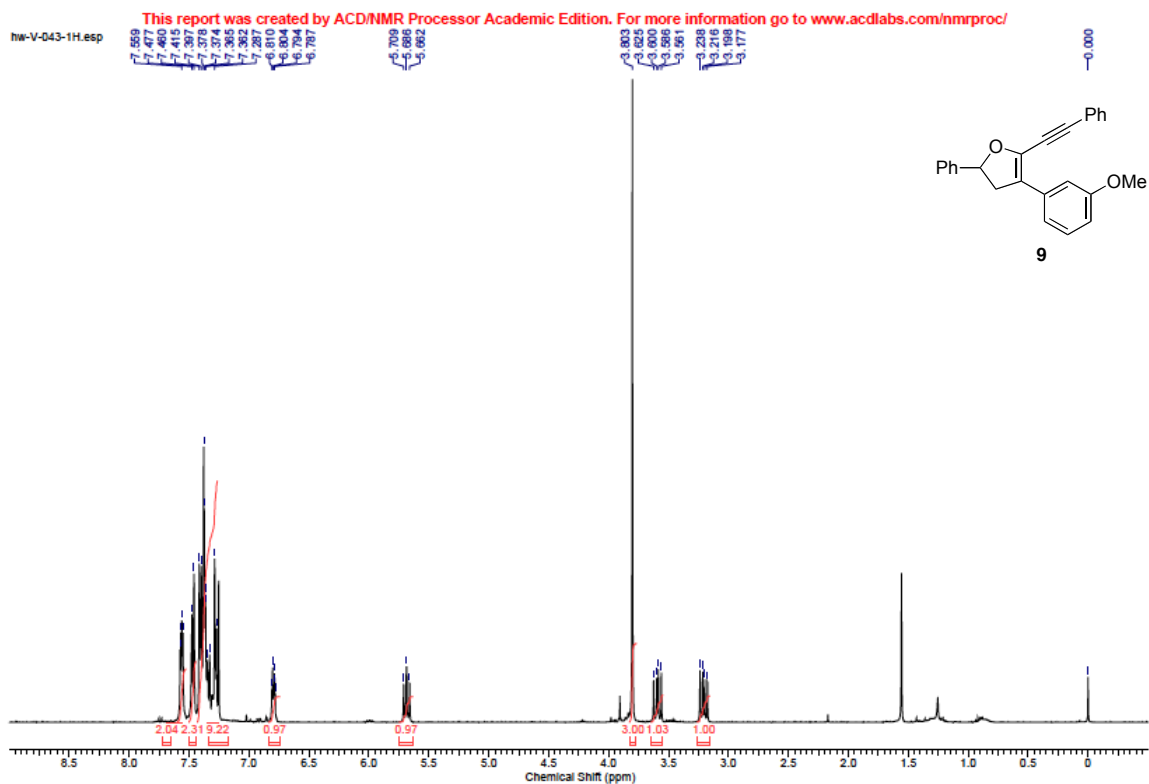
FT size 65536

Total time 1 hr, 25 min, 29 sec

^1H and ^{13}C spectra of compound 7



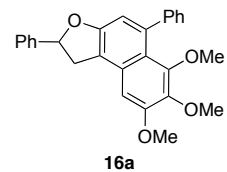
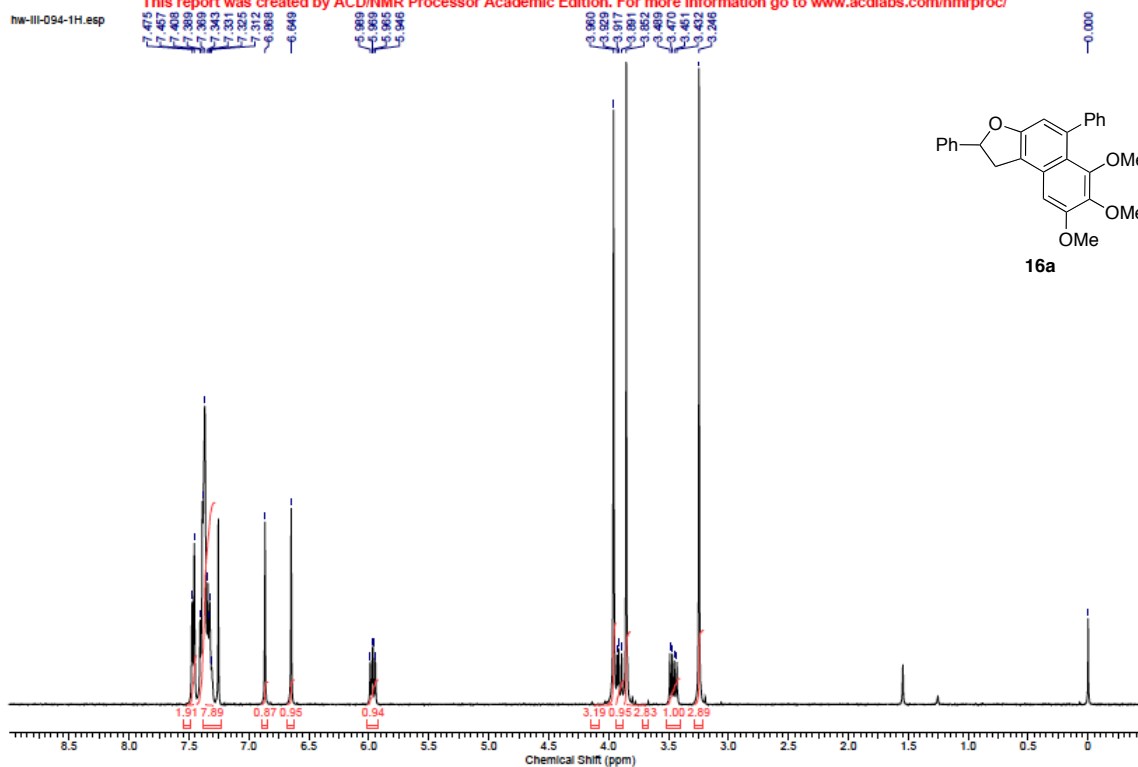
^1H and ^{13}C spectra of compound **8**



^1H and ^{13}C spectra of compound 9

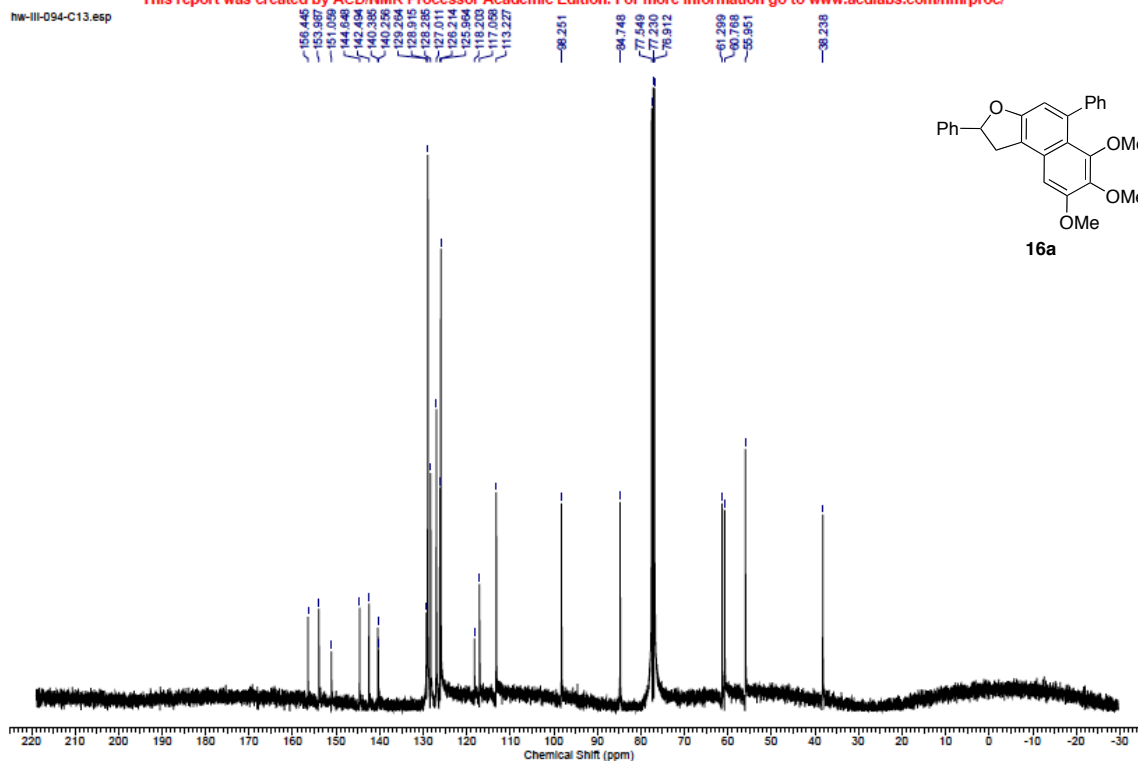
hw-III-094-1H.esp

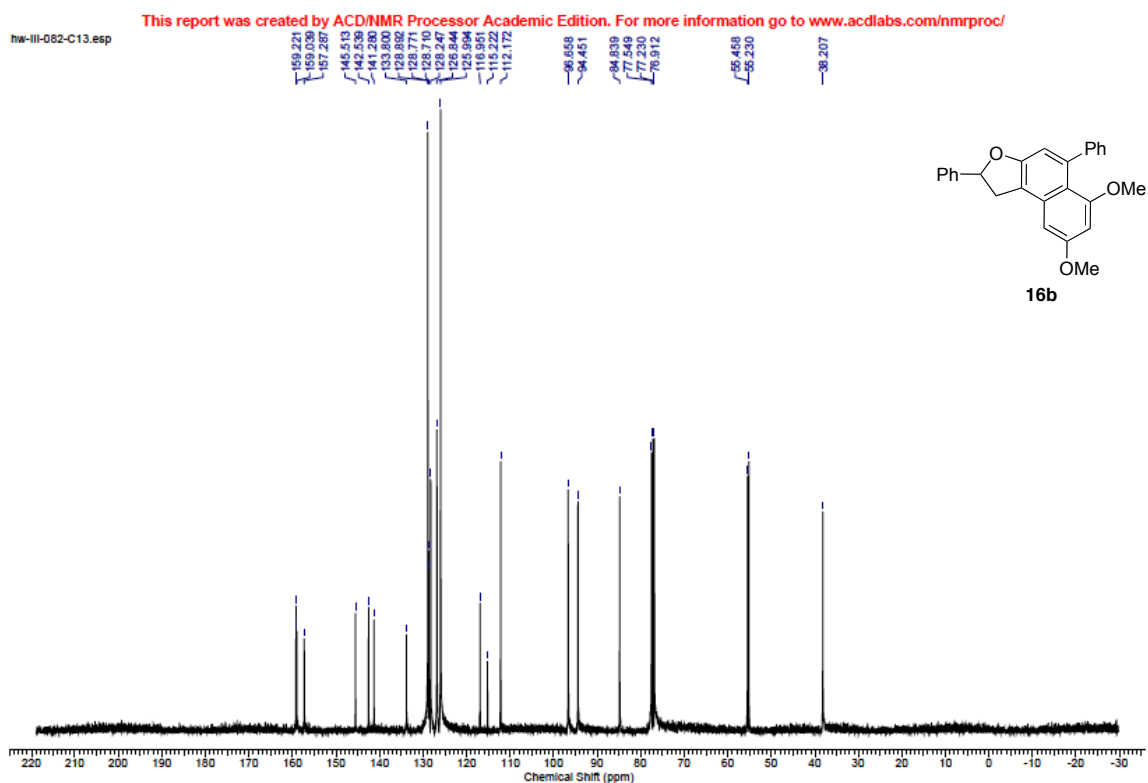
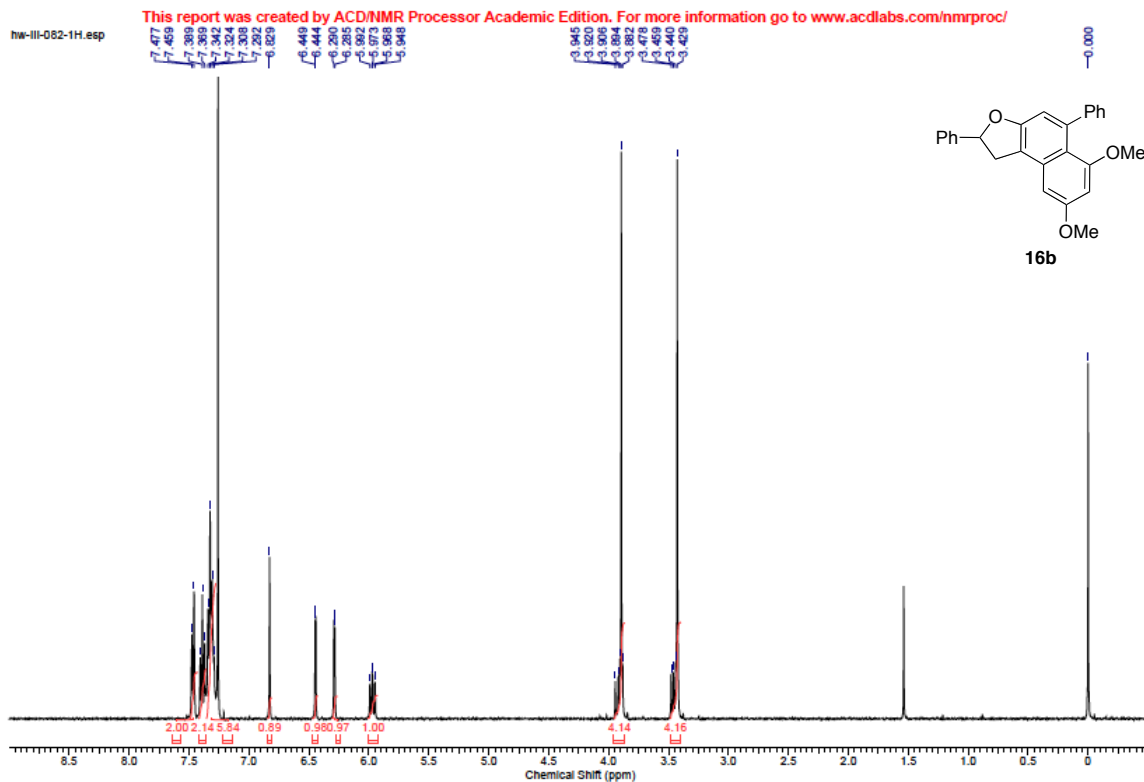
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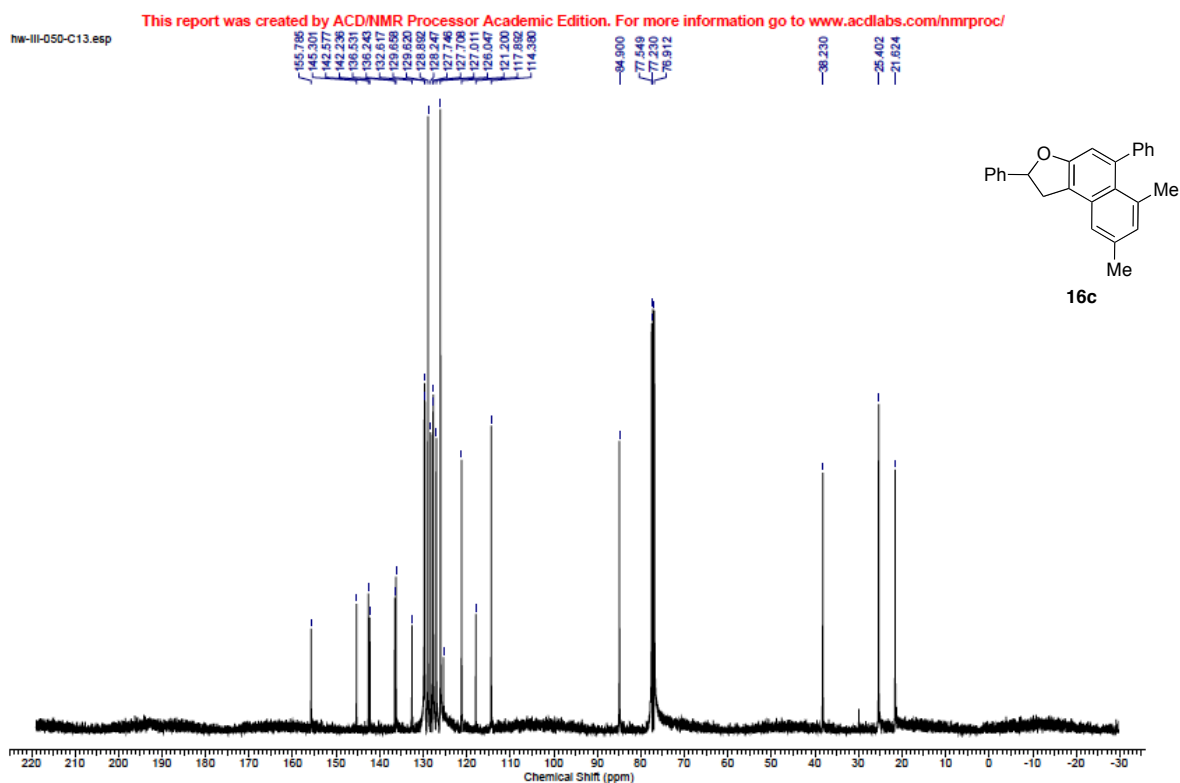
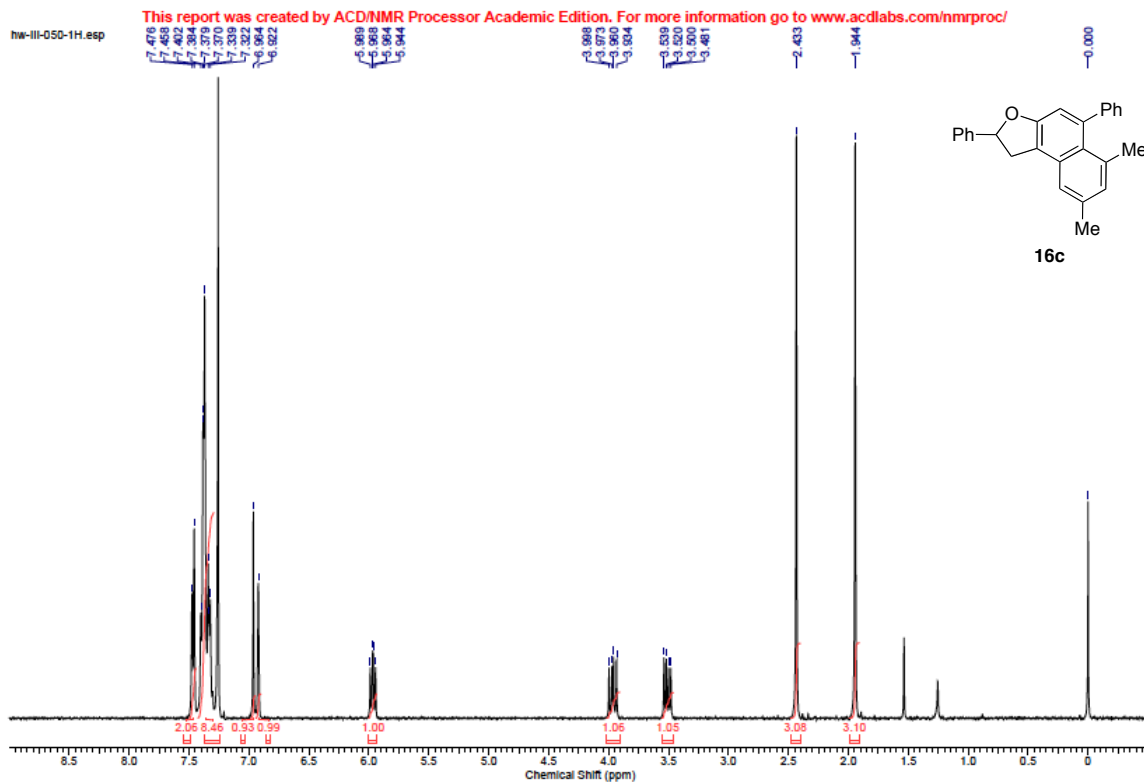
hw-III-094-C13.esp

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

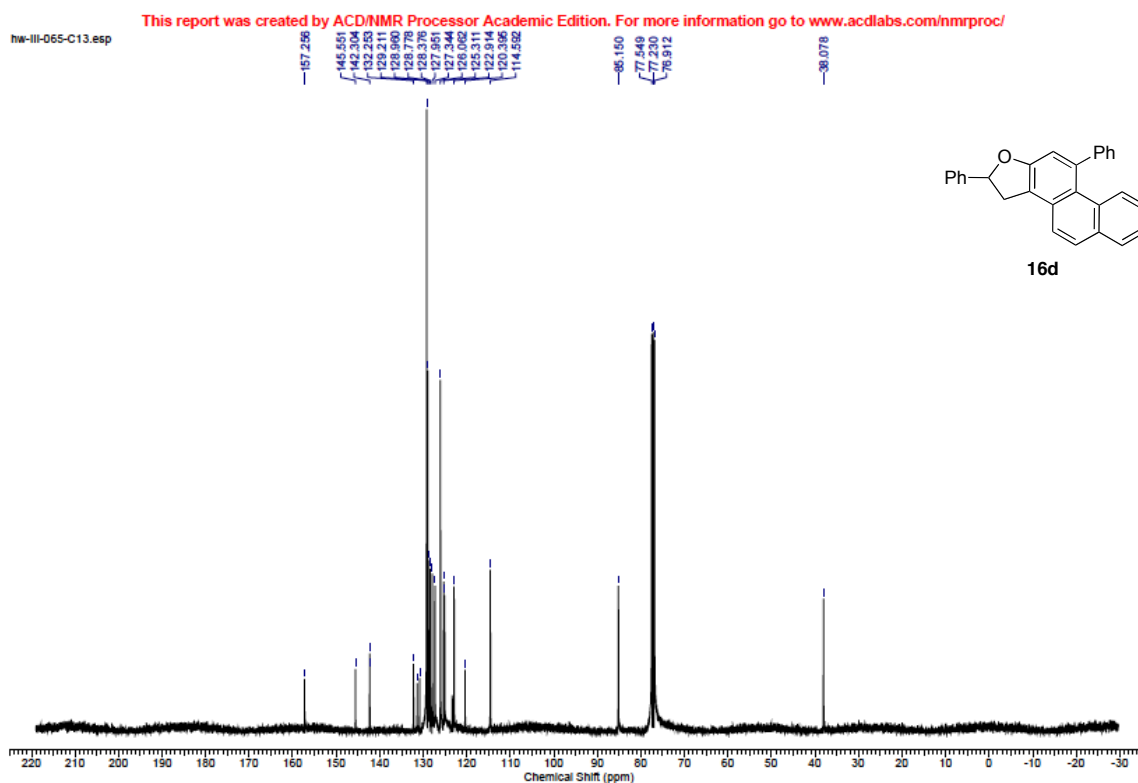
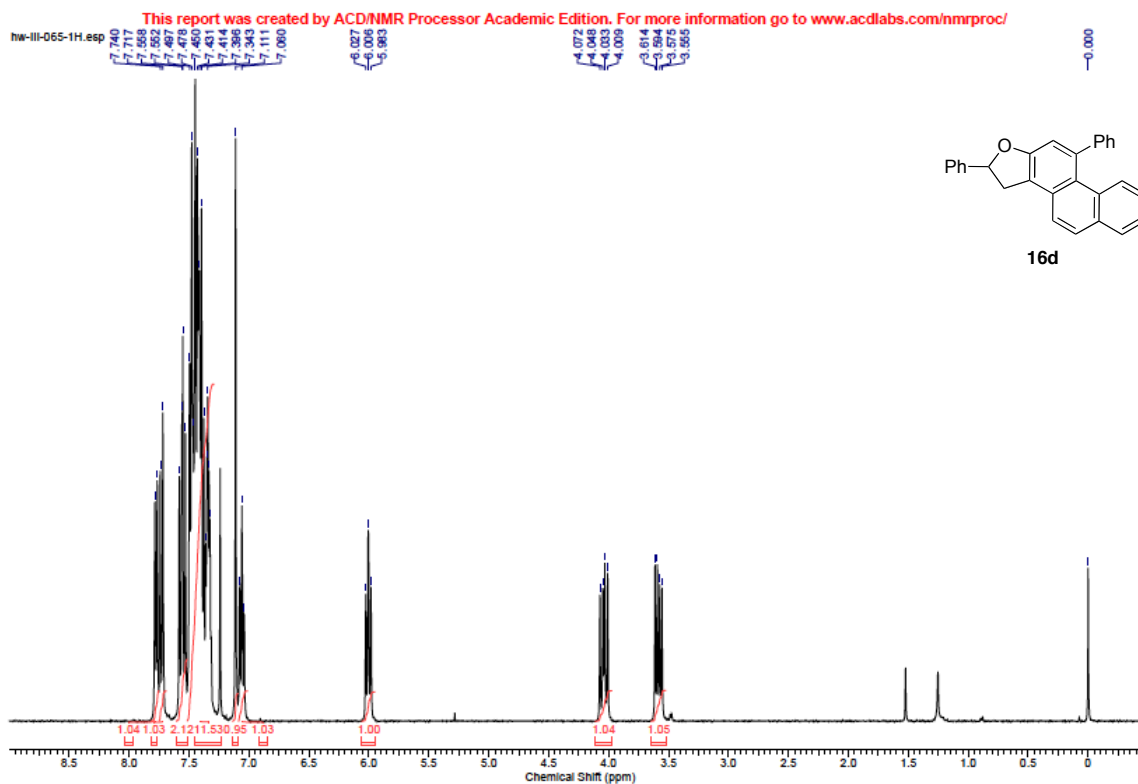




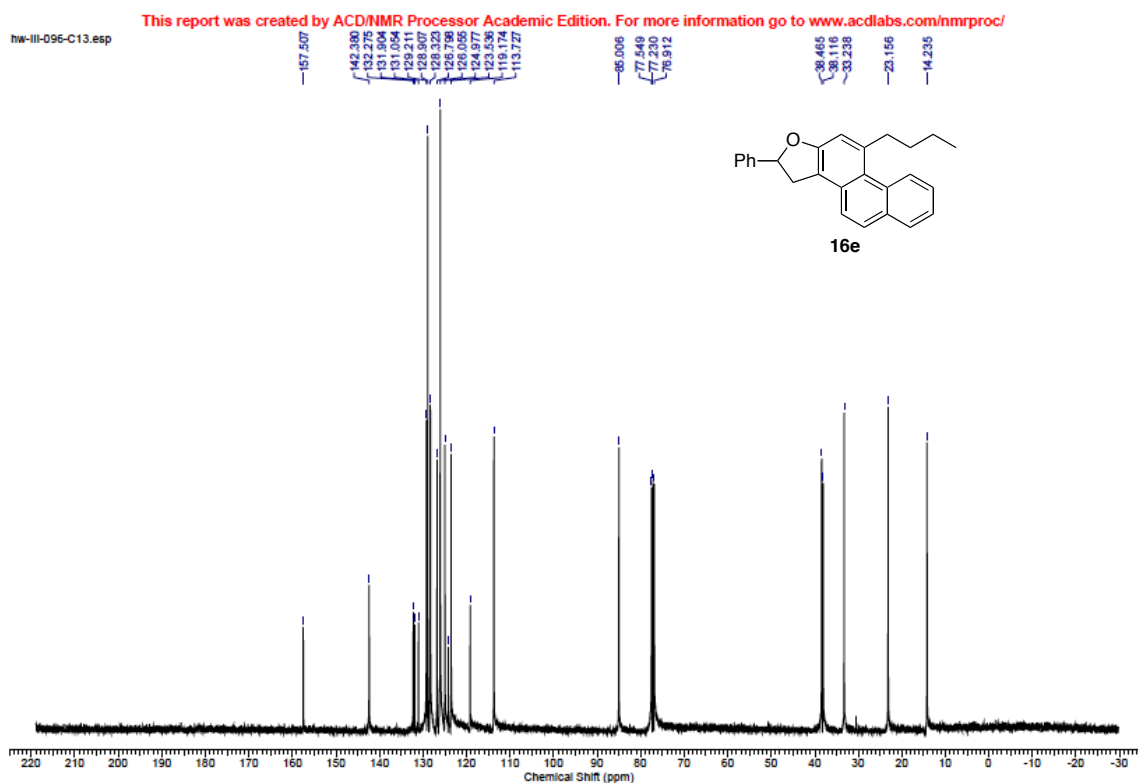
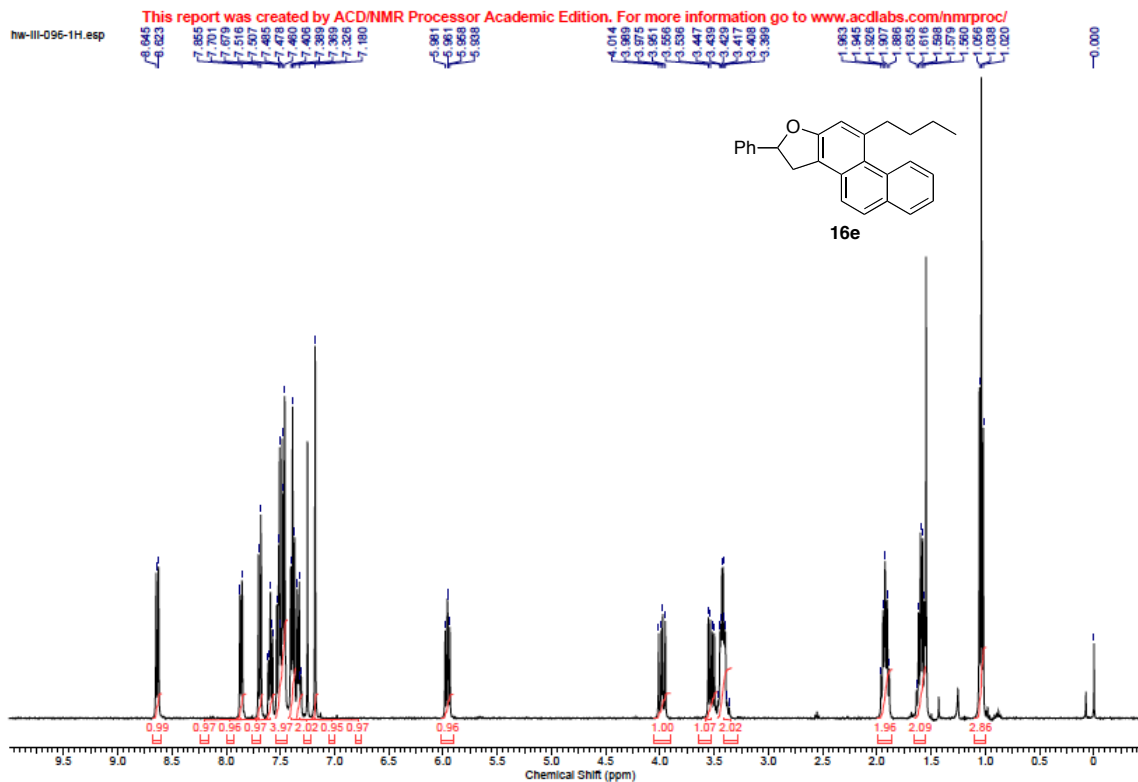
^1H and ^{13}C spectra of compound **16b**



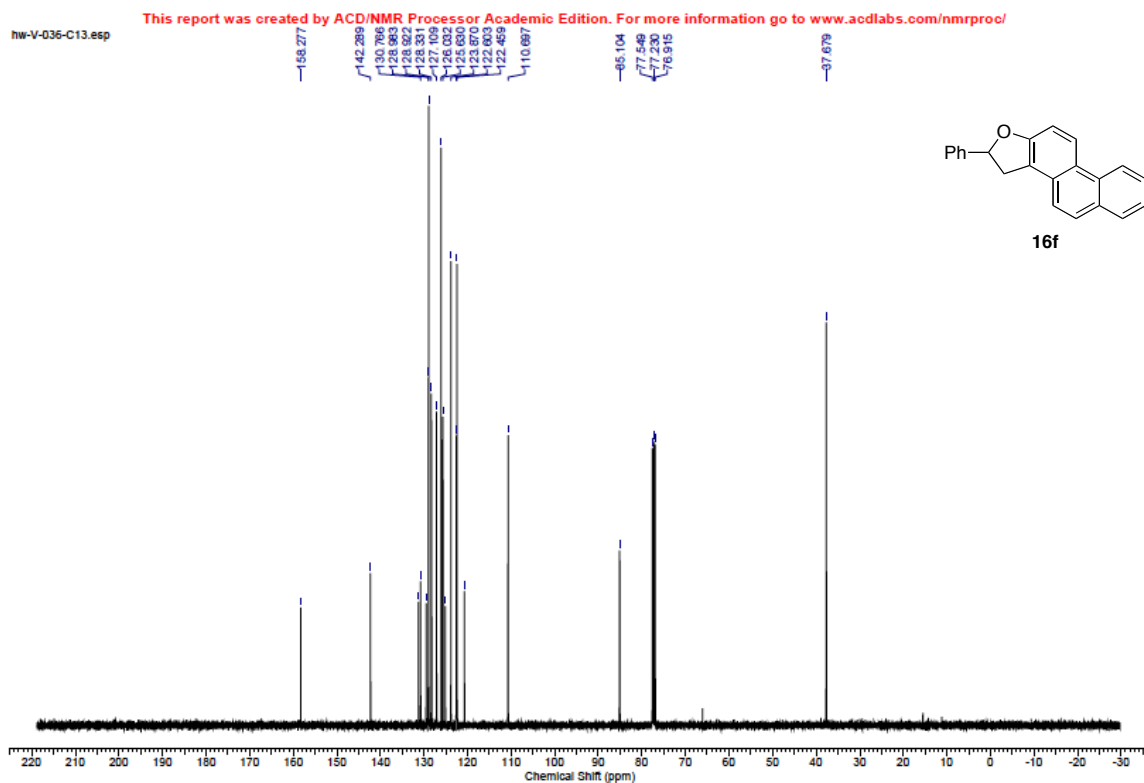
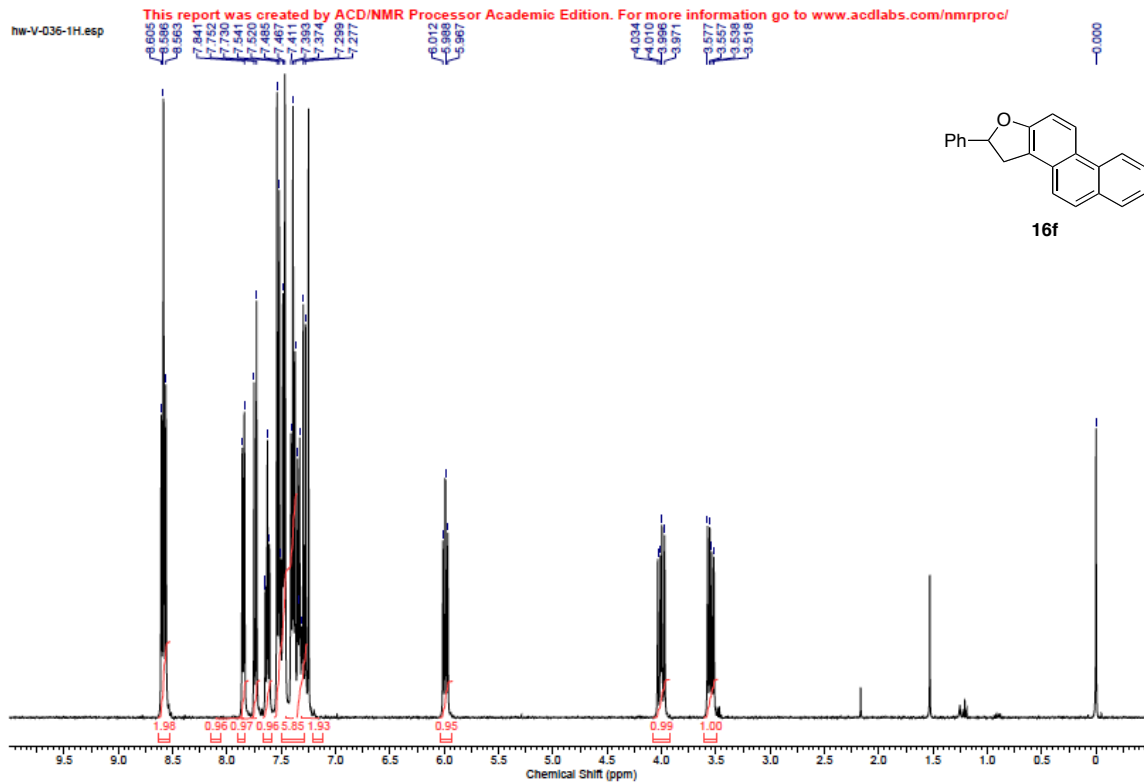
^1H and ^{13}C spectra of compound **16c**



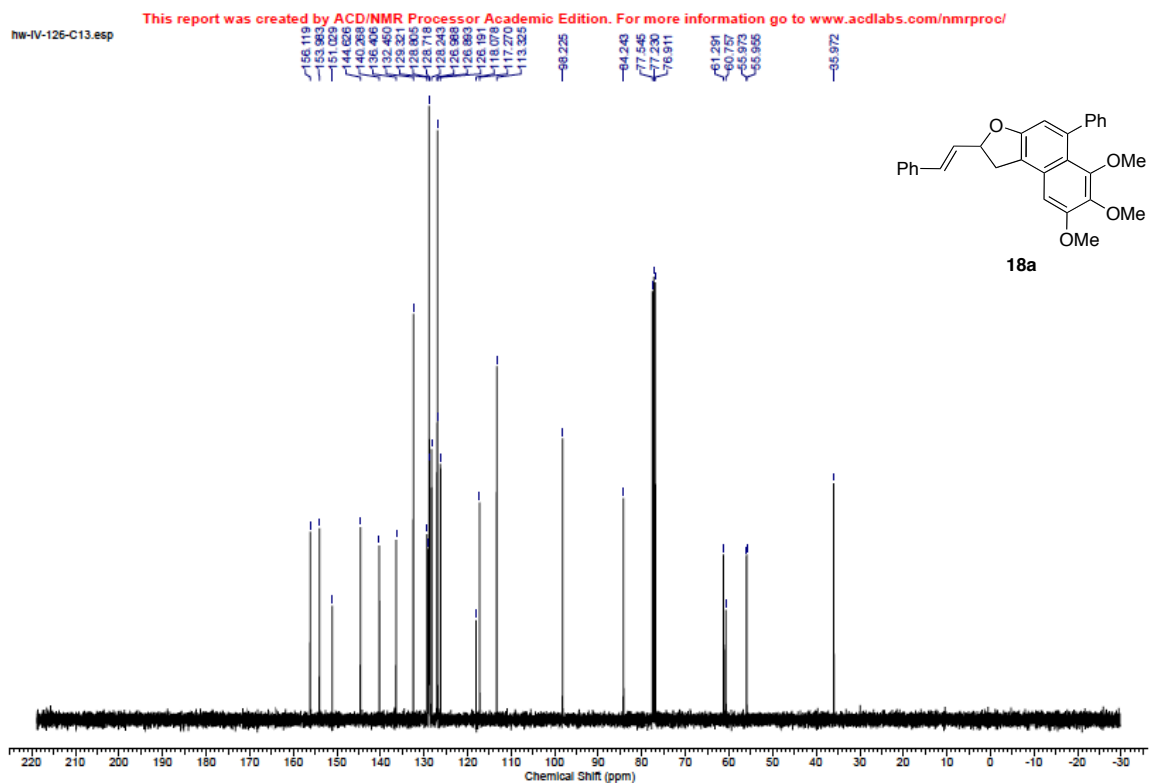
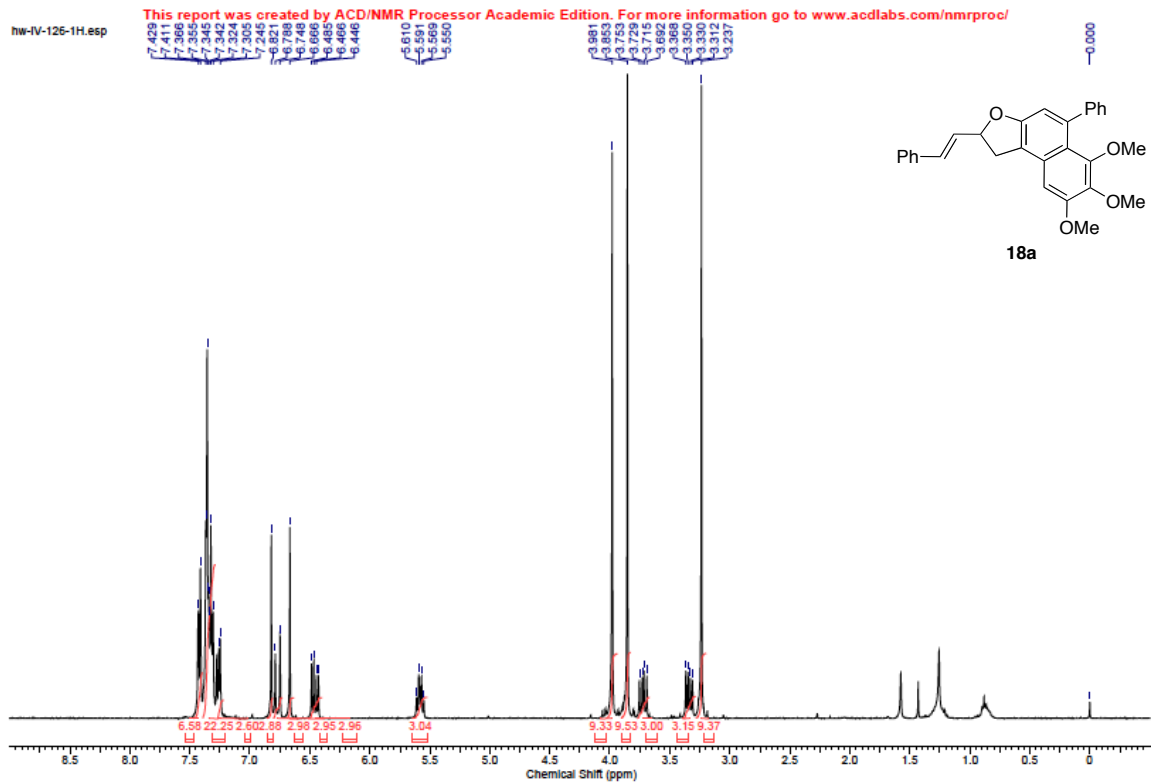
^1H and ^{13}C spectra of compound **16d**



^1H and ^{13}C spectra of compound **16e**



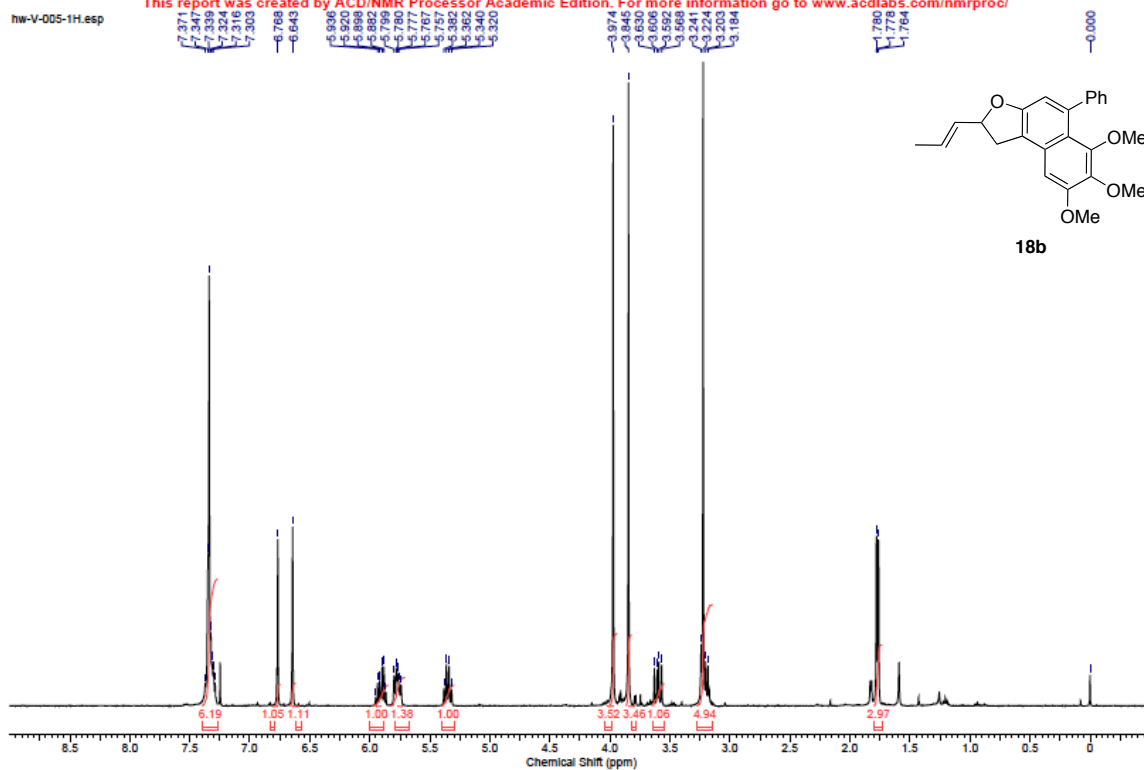
^1H and ^{13}C spectra of compound **16f**



^1H and ^{13}C spectra of compound **18a**

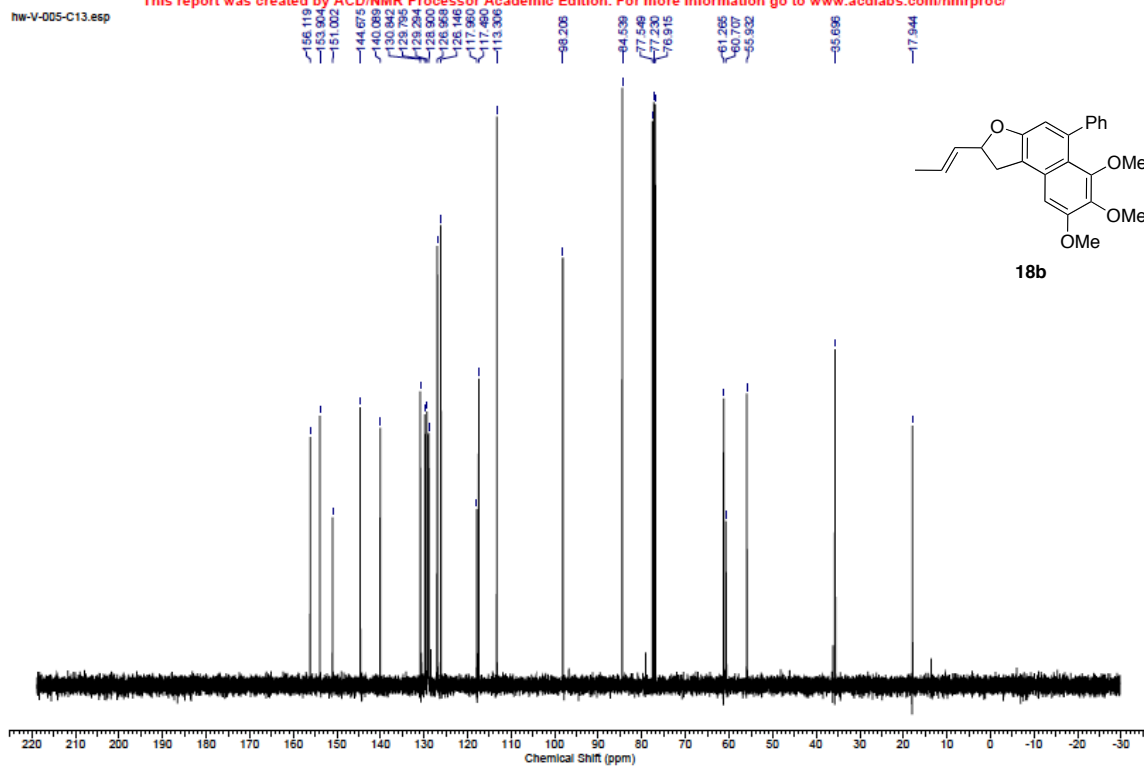
hw-V-005-1H.esp

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hw-V-005-C13.esp

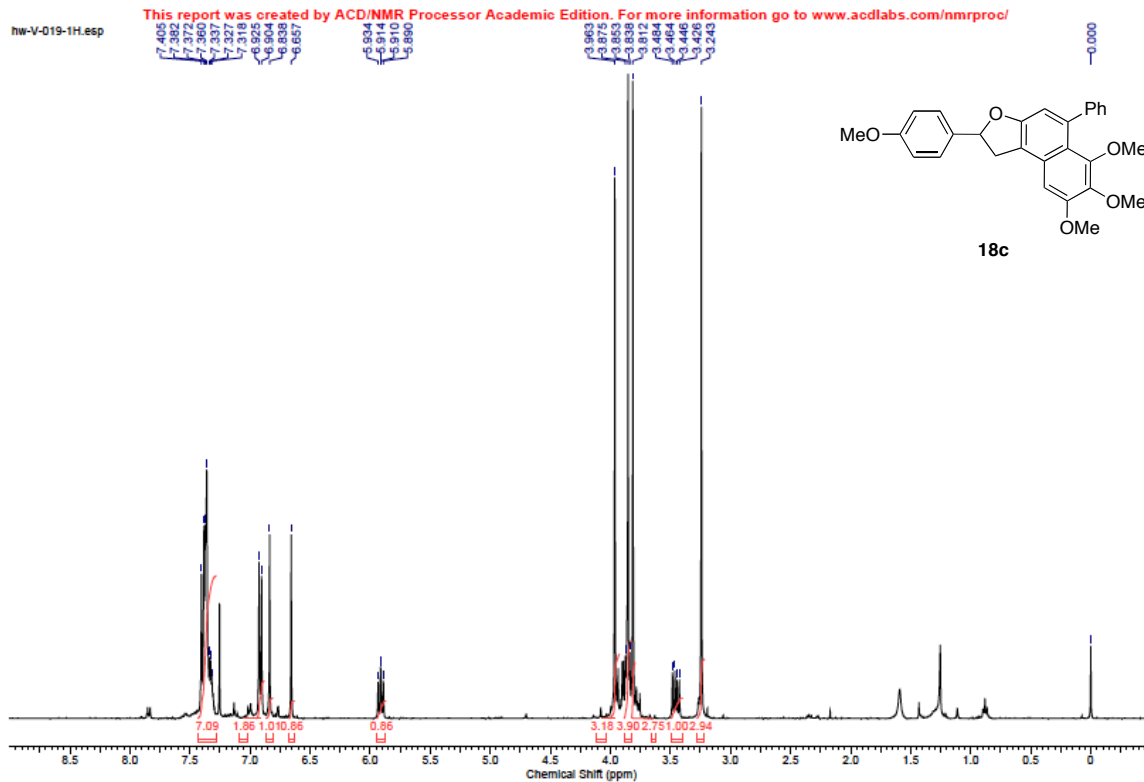
This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/



¹H and ¹³C spectra of compound 18b

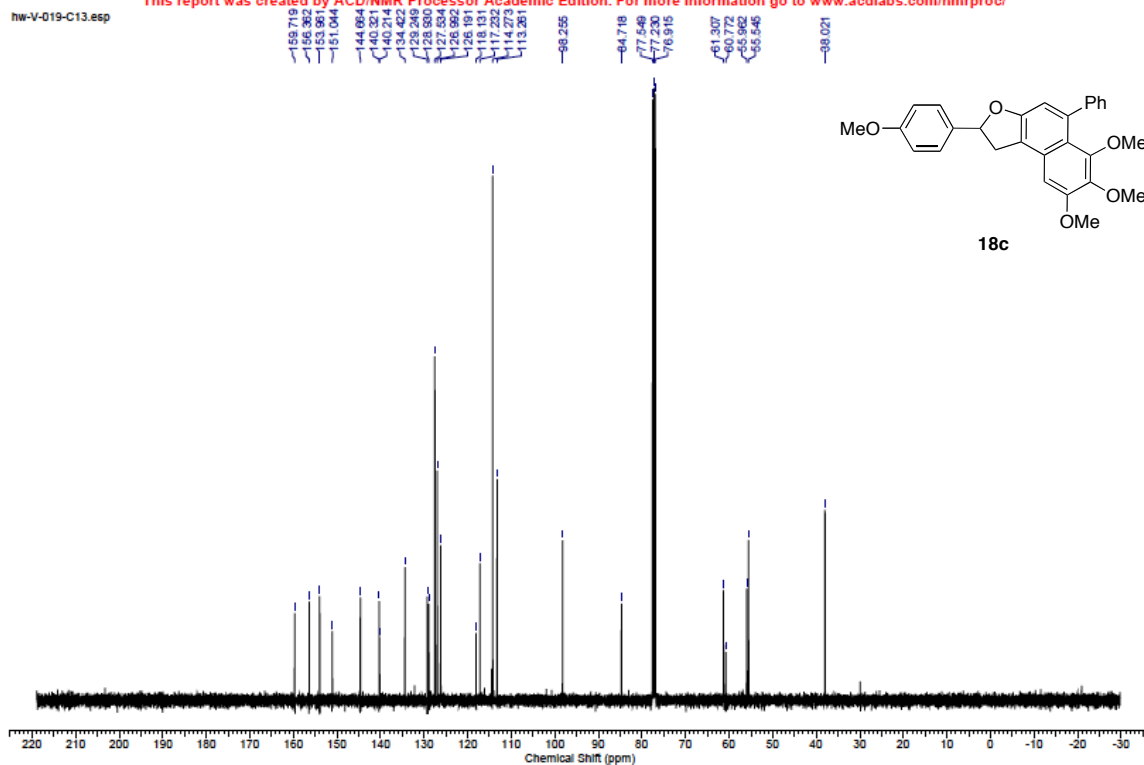
hw-V-019-1H.esp

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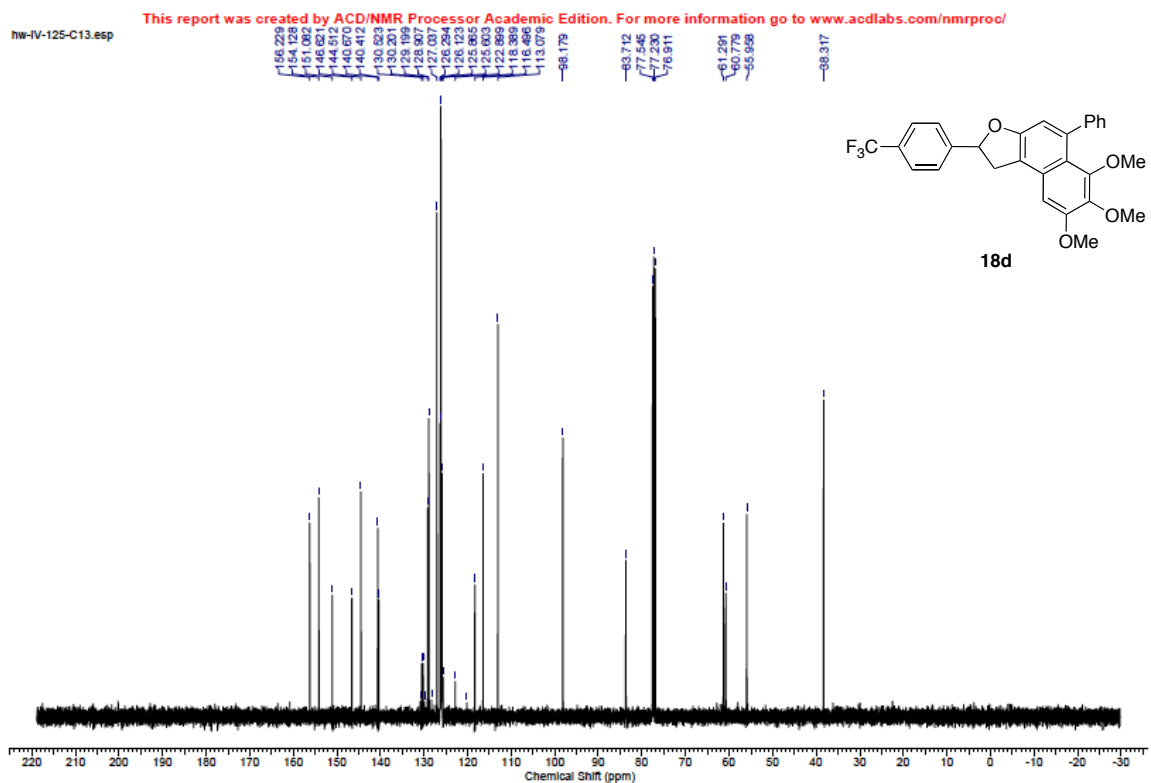
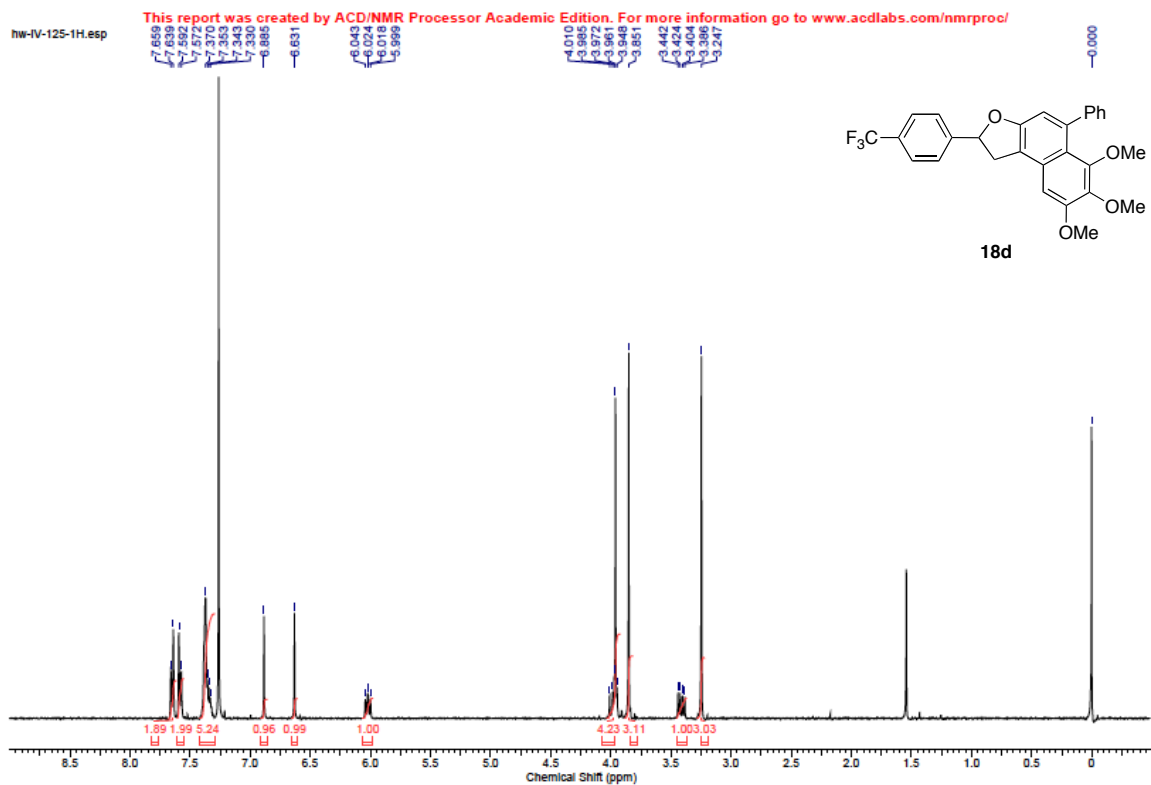


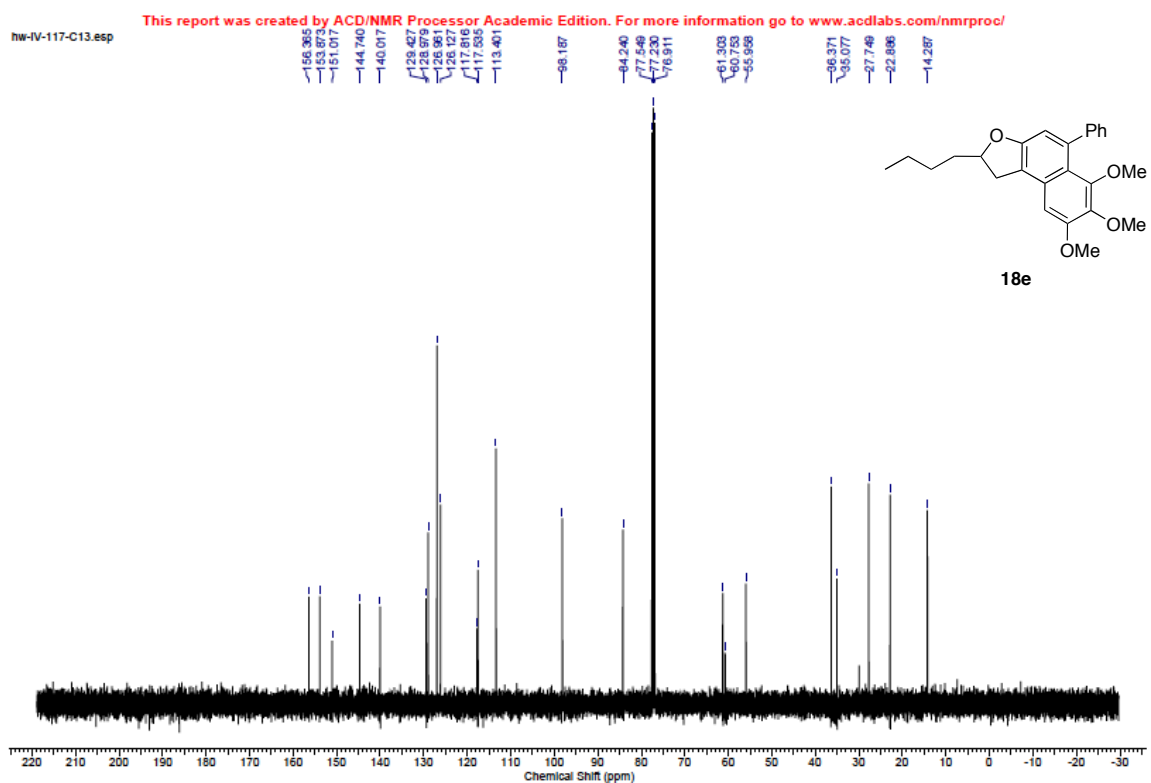
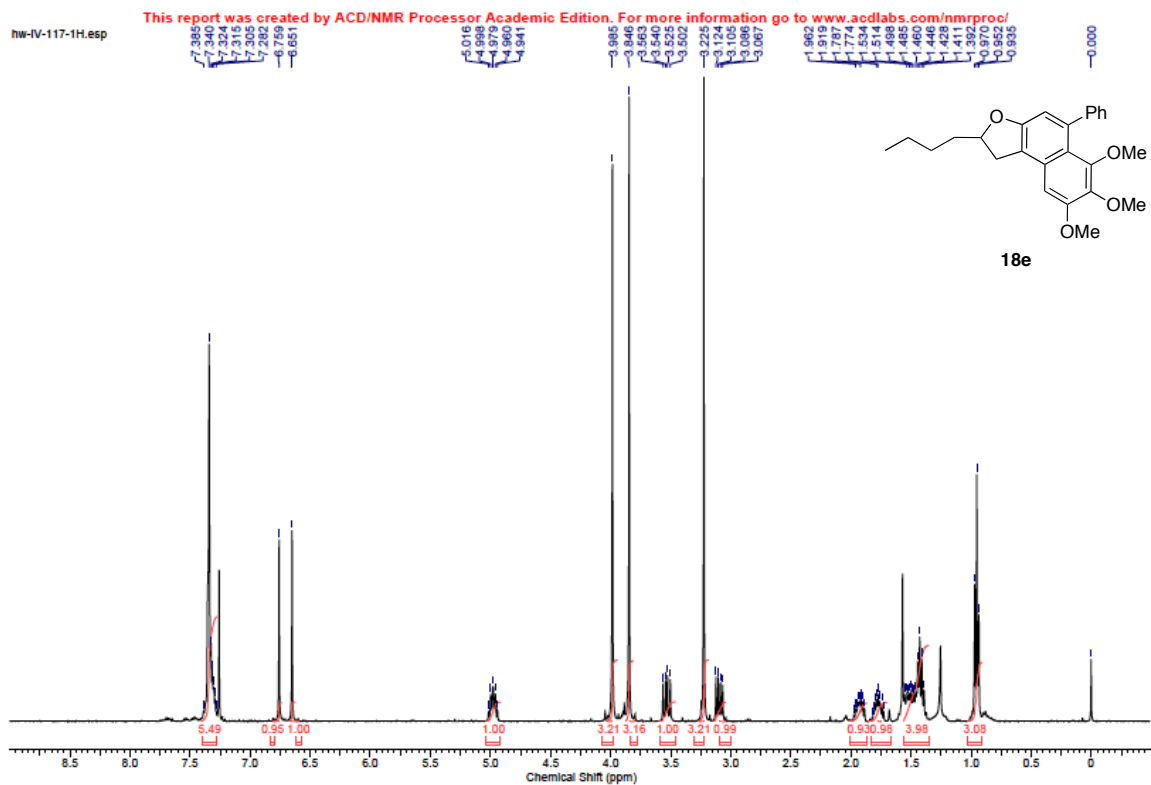
hw-V-019-C13.esp

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



^1H and ^{13}C spectra of compound **18c**

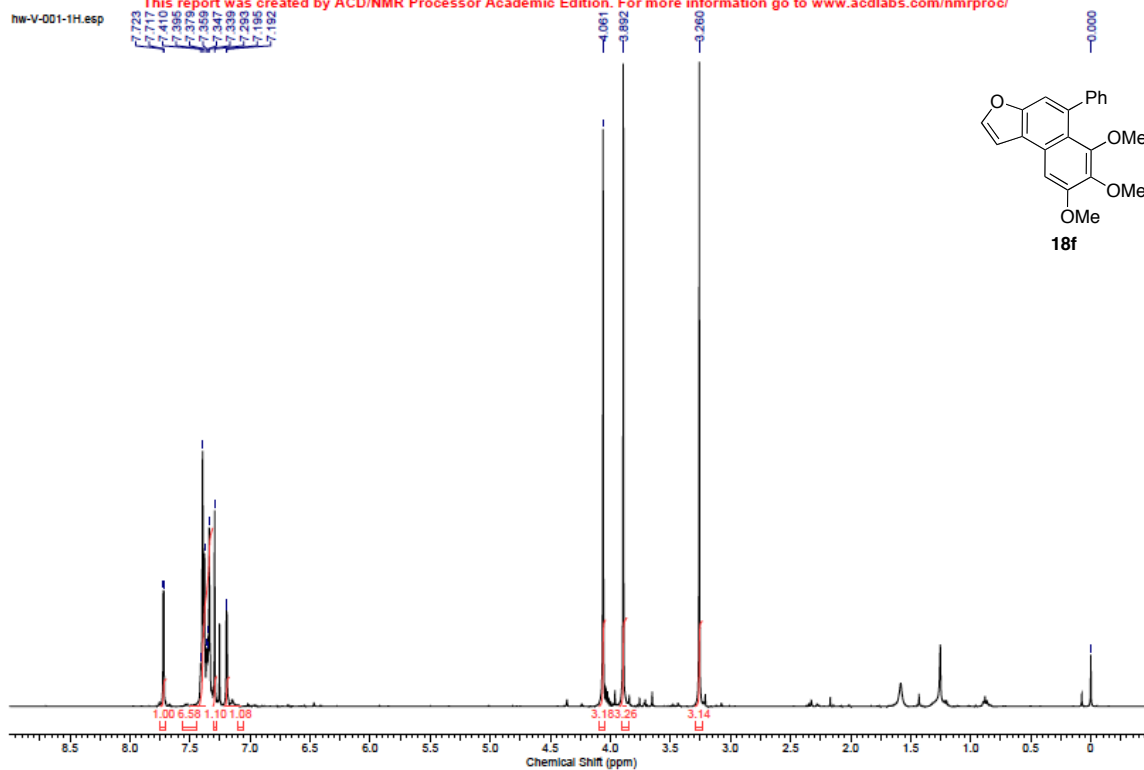
 ^1H and ^{13}C spectra of compound **18d**



^1H and ^{13}C spectra of compound **18e**

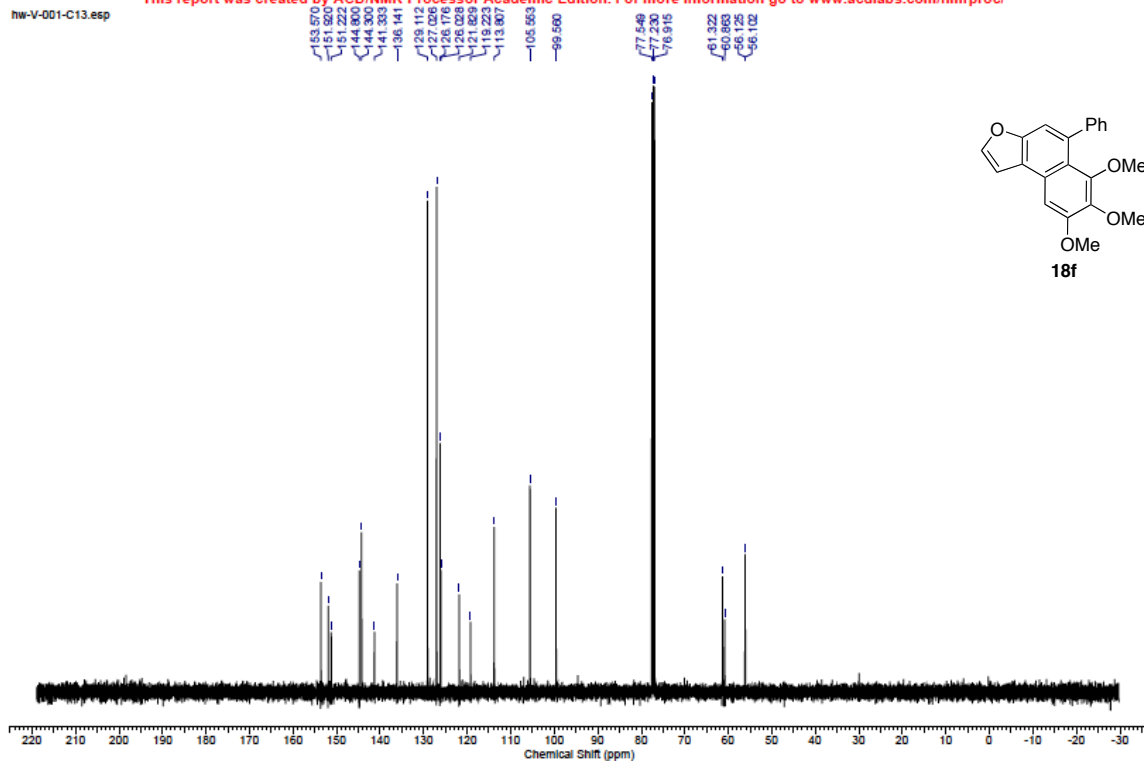
hw-V-001-1H.esp

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

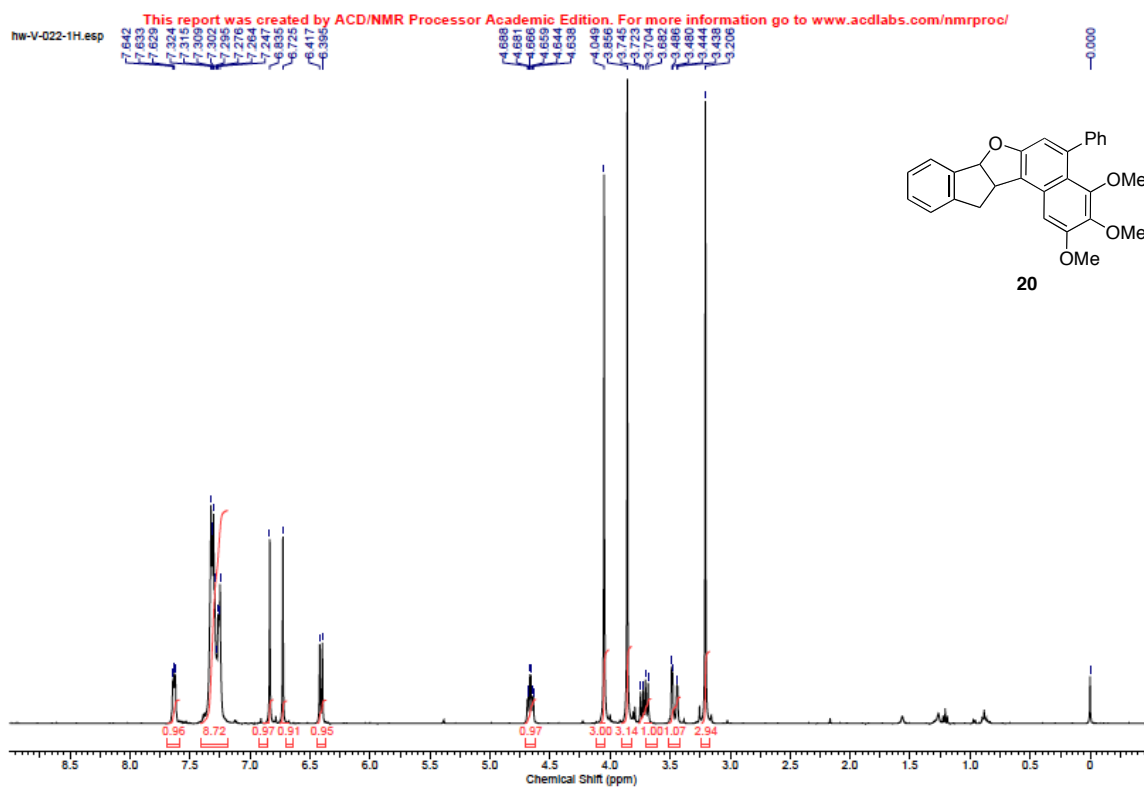


hw-V-001-C13.esp

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^1H and ^{13}C spectra of compound **18f**



hw-V-022-aftercolumn-A-c13

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

User: j-lc-87

UNITYplus-600 "up1600"

PULSE SEQUENCE

Relax. delay 2.000 sec

Pulse 69.2 degrees

Acq. time 1.000 sec

Width 31946.9 Hz

3488 repetitions

OBSERVE C13, 150.7606305 MHz

DECOUPLE H1, 599.5989303 MHz

Power 36 dB

continuously on

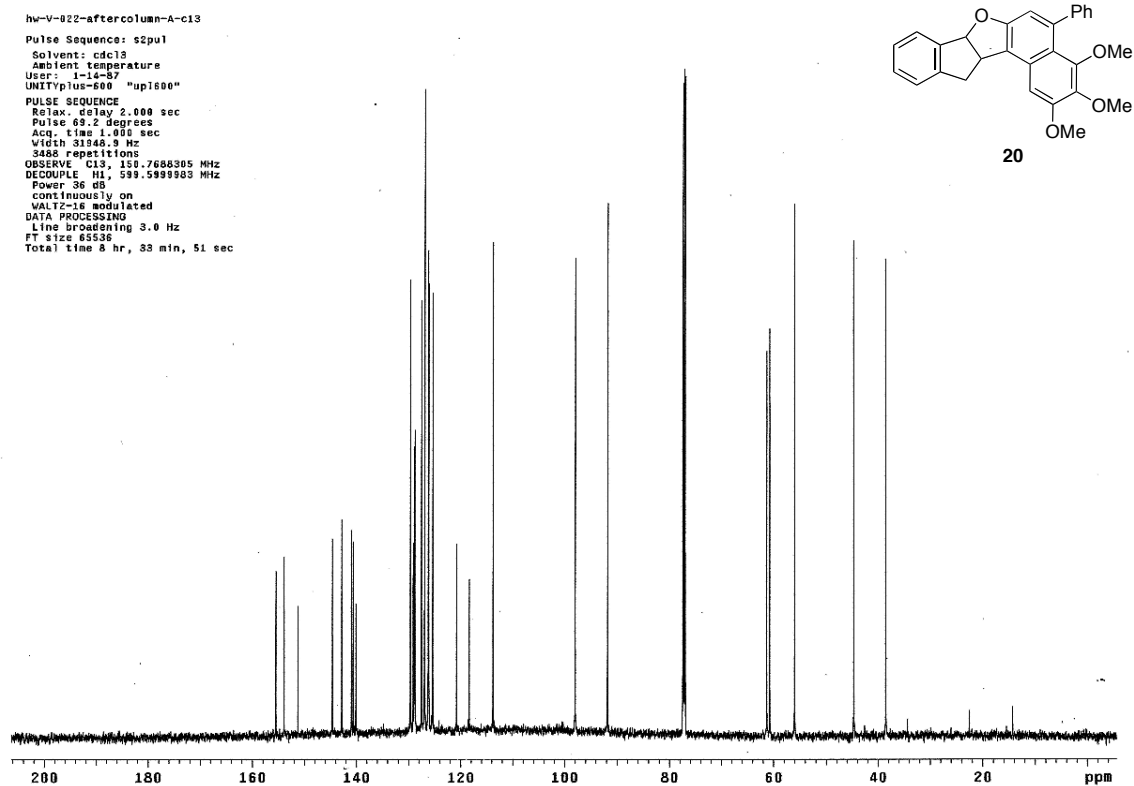
WALTZ-16 modulated

DATA PROCESSING

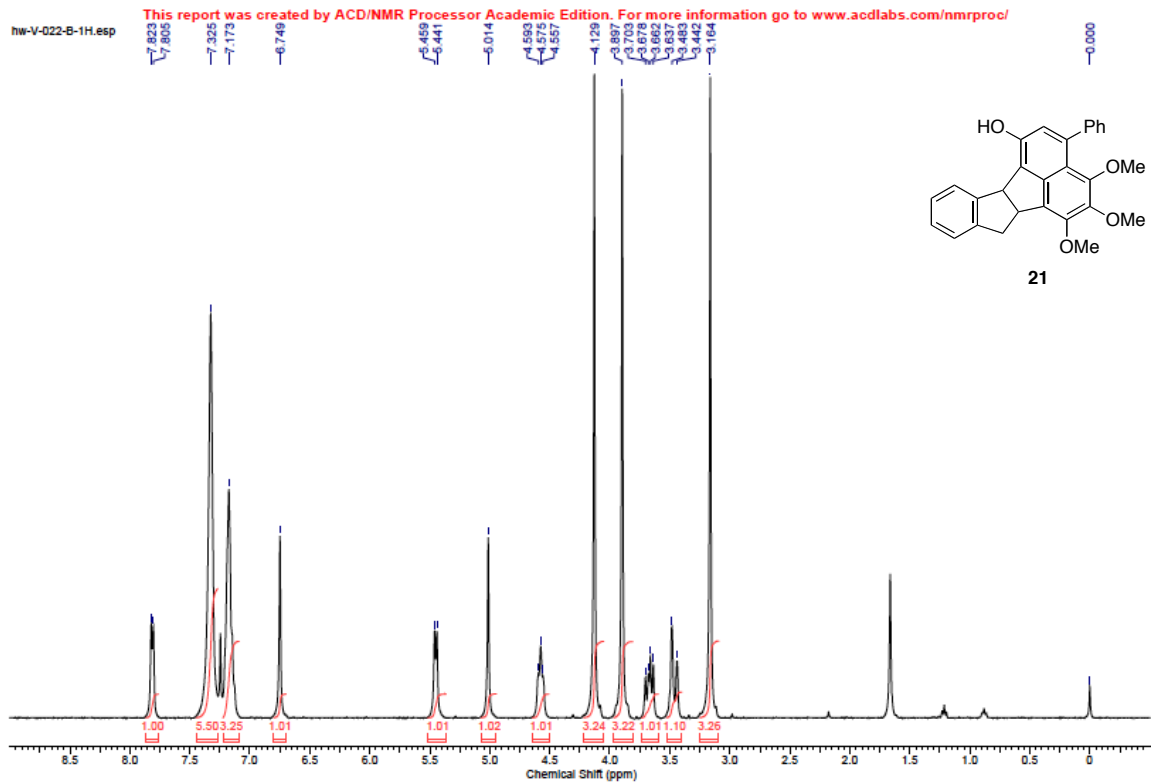
Line broadening 3.0 Hz

FT size 65536

Total time 8 hr, 33 min, 51 sec



^1H and ^{13}C spectra of compound 20



hw-V-022-aftercolumn-B-c13

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

User: 1-14-87

UNITYplus-500 "up1600"

PULSE SEQUENCE

Relax. delay 2.000 sec

Pulse 69.2 degrees

Acq. time 1.000 sec

Width 31948.9 Hz

1650 repetitions

OBSERVE C13, 150.7688285 MHz

DECOUPLE H1, 599.5999983 MHz

Power 38 dB

continuously on

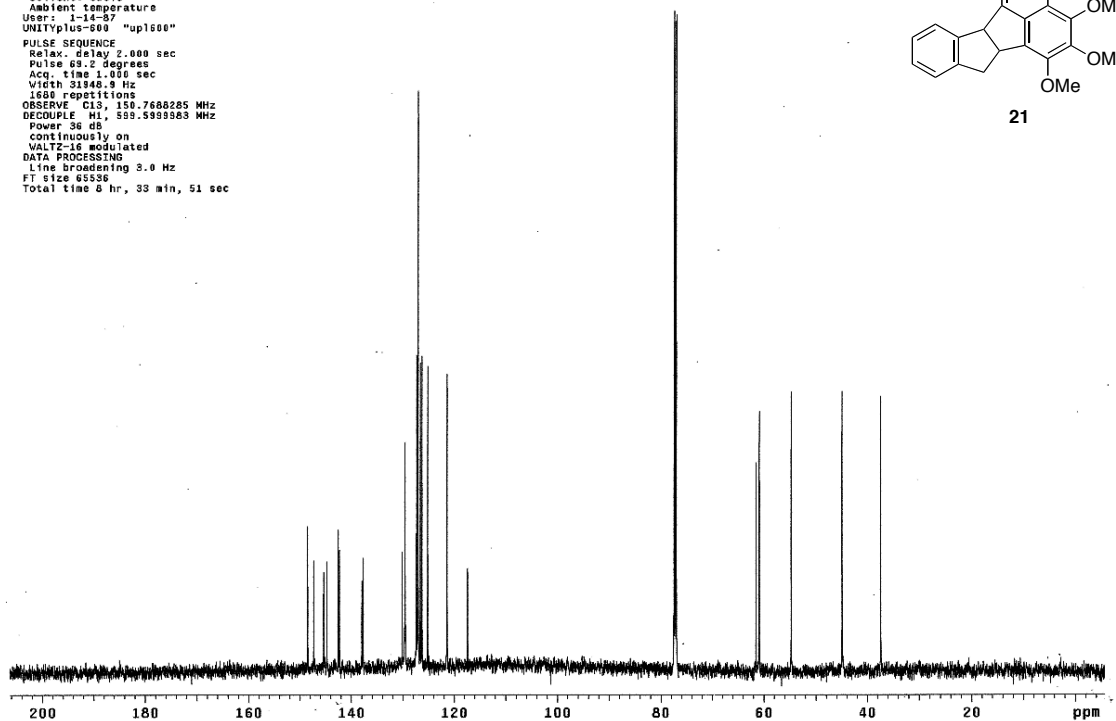
VALTZ-16 modulated

DATA PROCESSING

Line broadening 3.0 Hz

FT size 65536

Total time 8 hr, 33 min, 51 sec



^1H and ^{13}C spectra of compound 21