

Supporting Information File 2

for

A strategic approach to [6,6]-bicyclic lactones:

application towards the CD fragment of DH β E

Tue Heesgaard Jepsen[§], Emil Glibstrup[§], François Crestey, Anders A. Jensen and
Jesper Langgaard Kristensen*

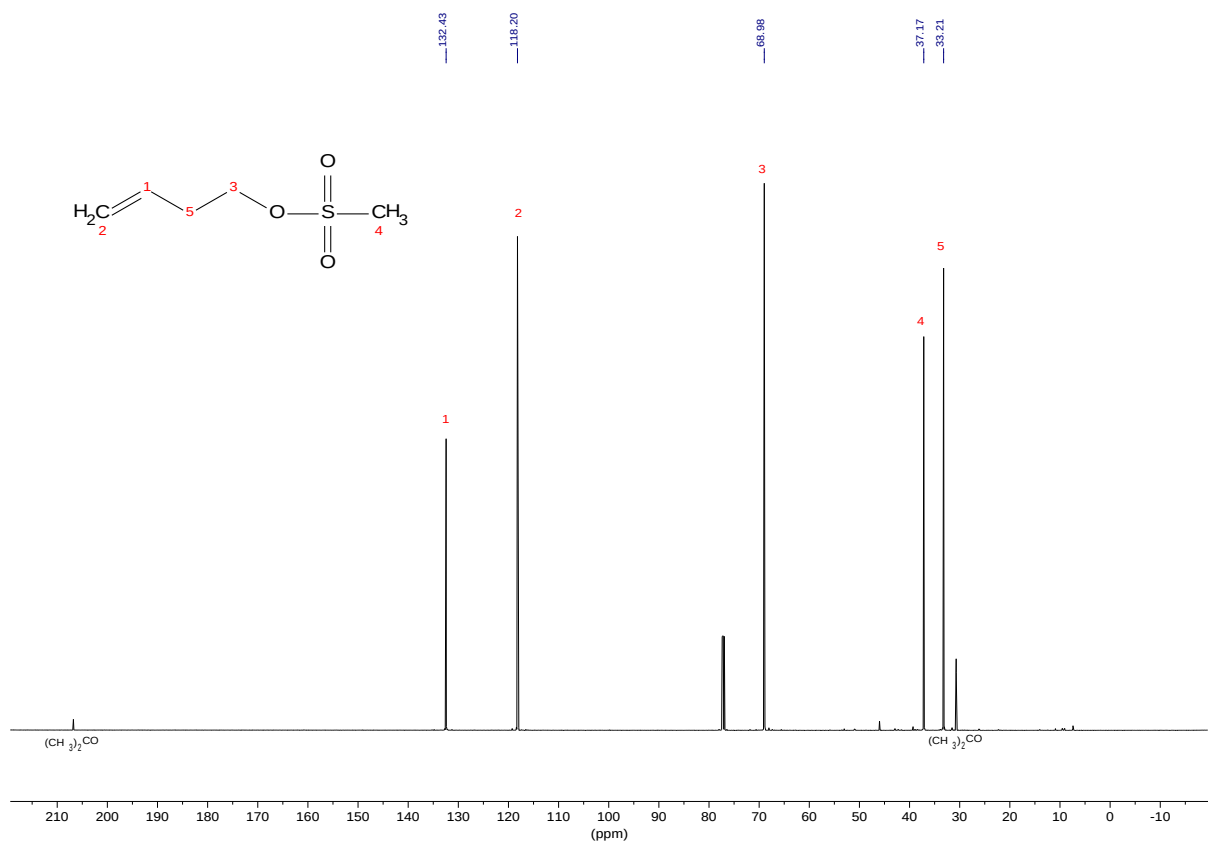
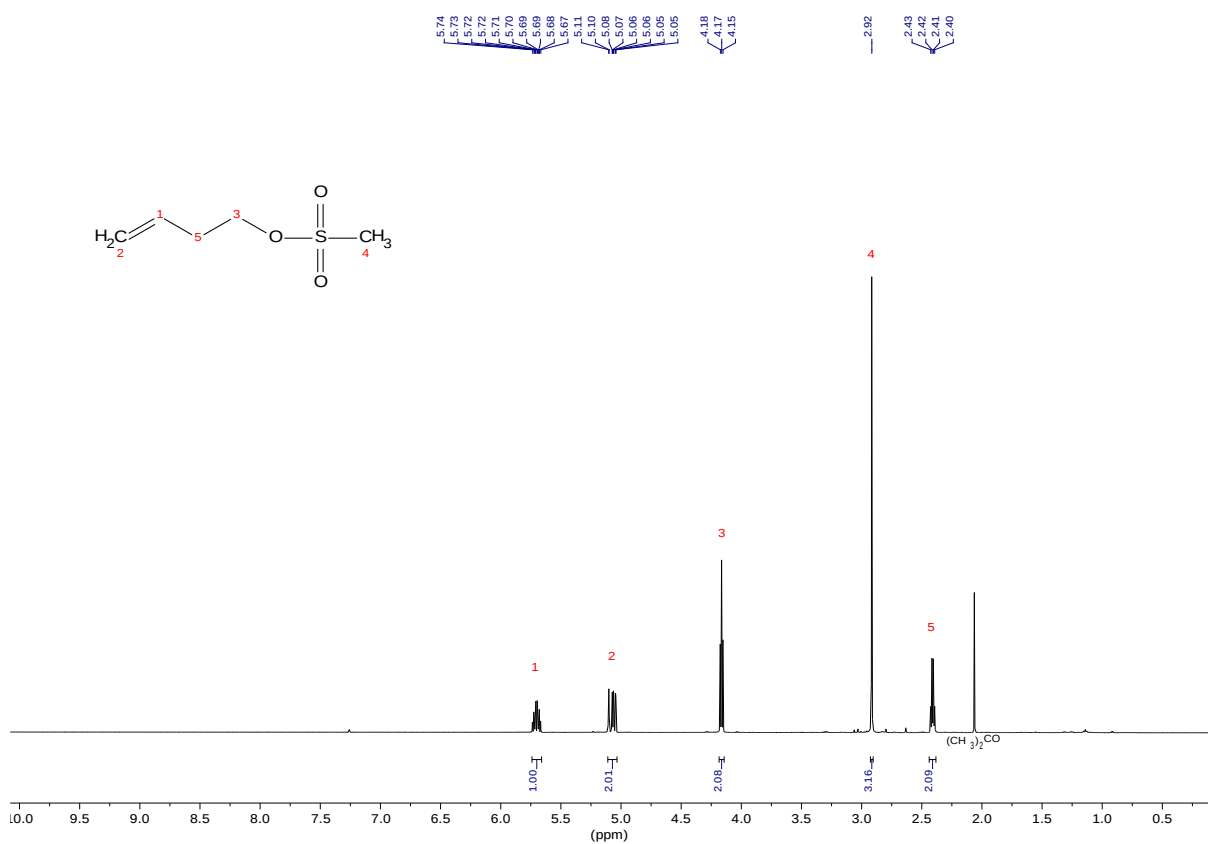
Address: Department of Drug Design and Pharmacology, Faculty of Health and Medical
Sciences, University of Copenhagen, Universitetsparken 2, 2100 Copenhagen, Denmark

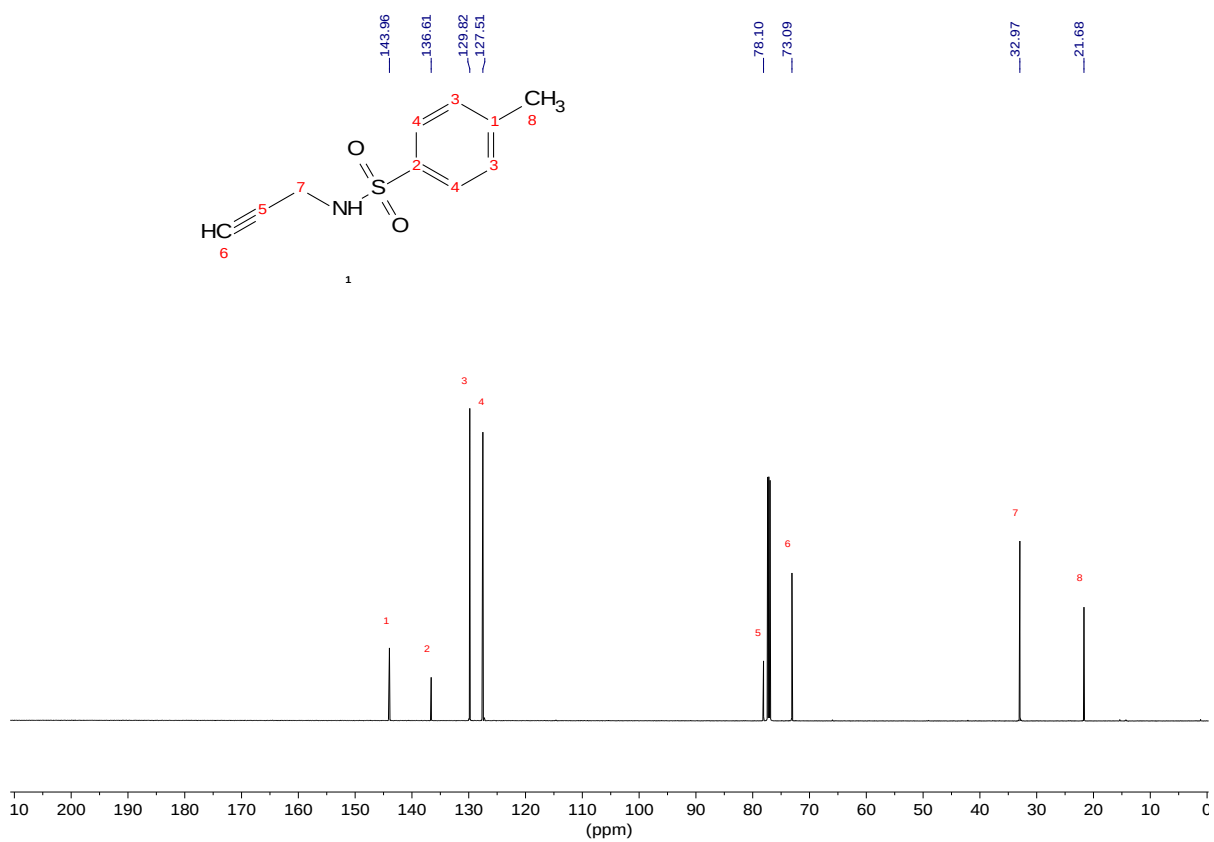
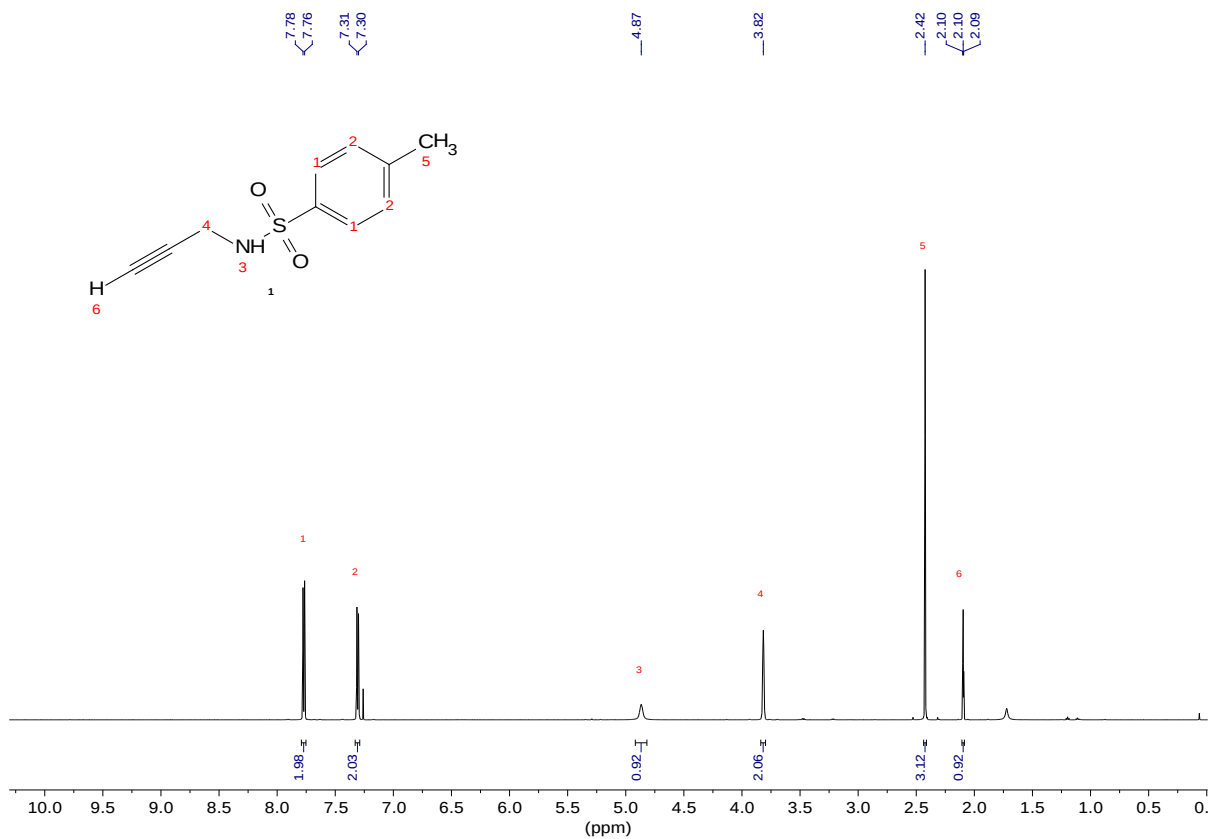
Email: Jesper Langgaard Kristensen - jesper.kristensen@sund.ku.dk

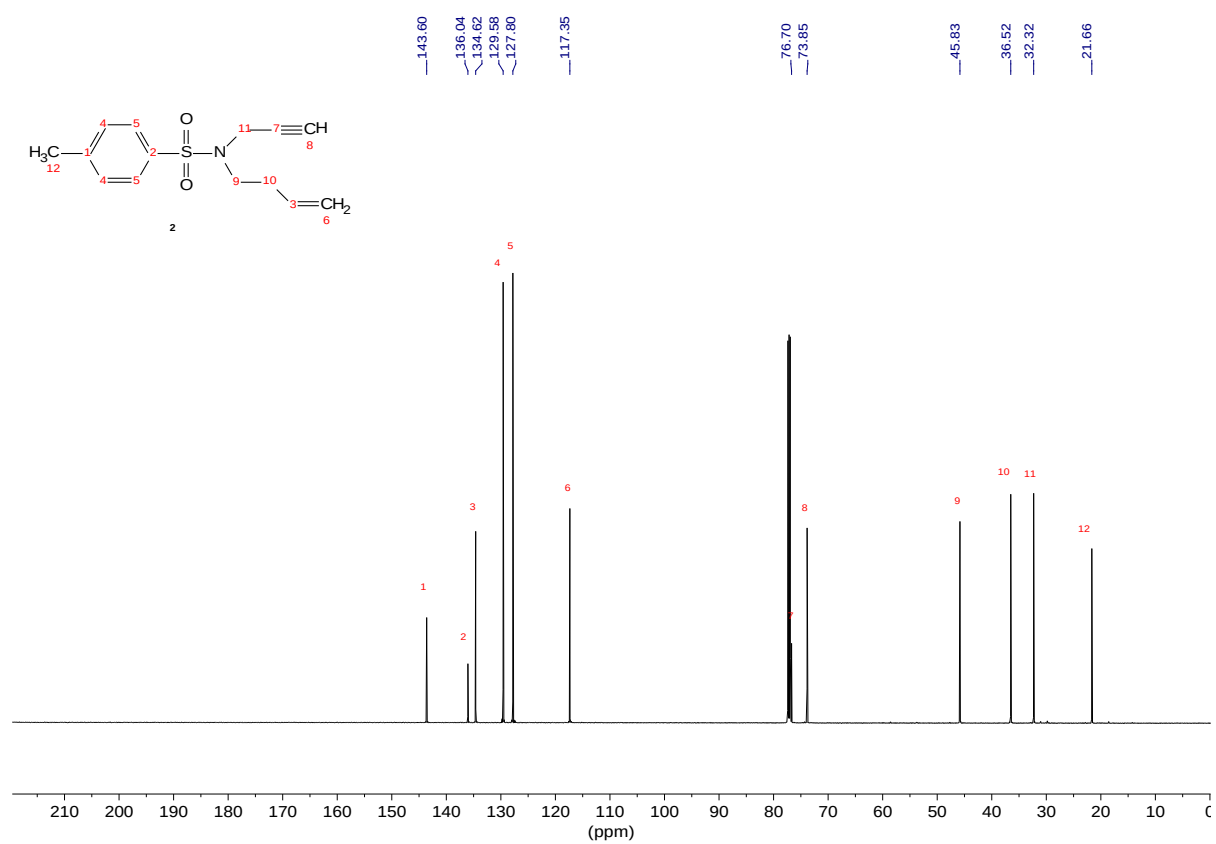
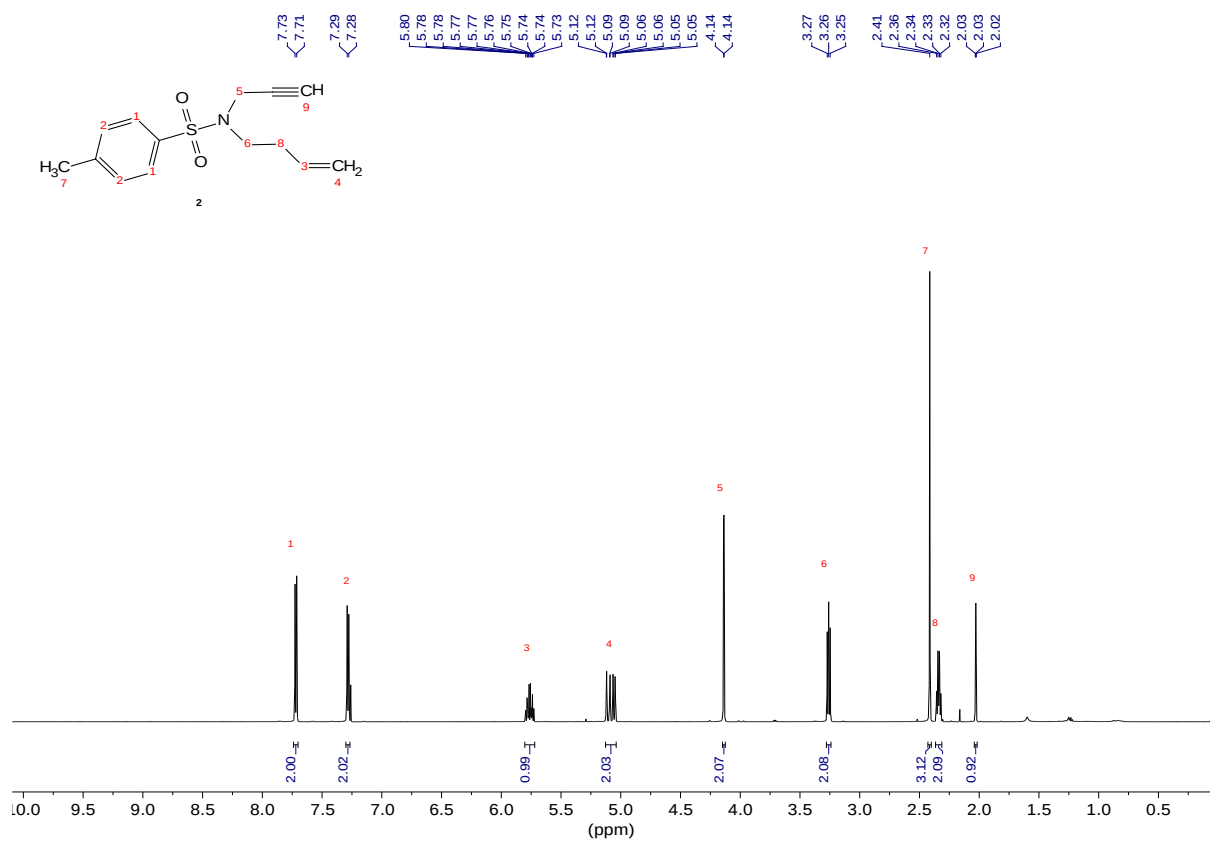
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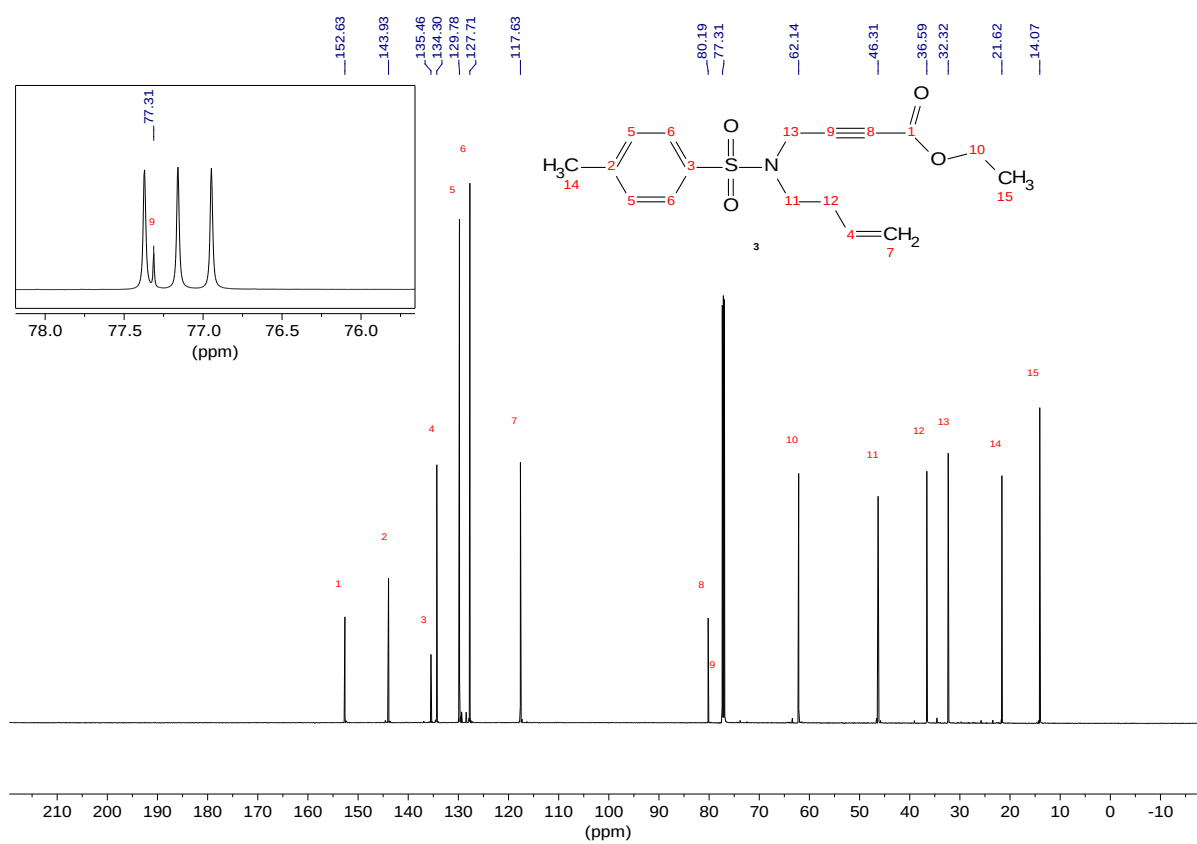
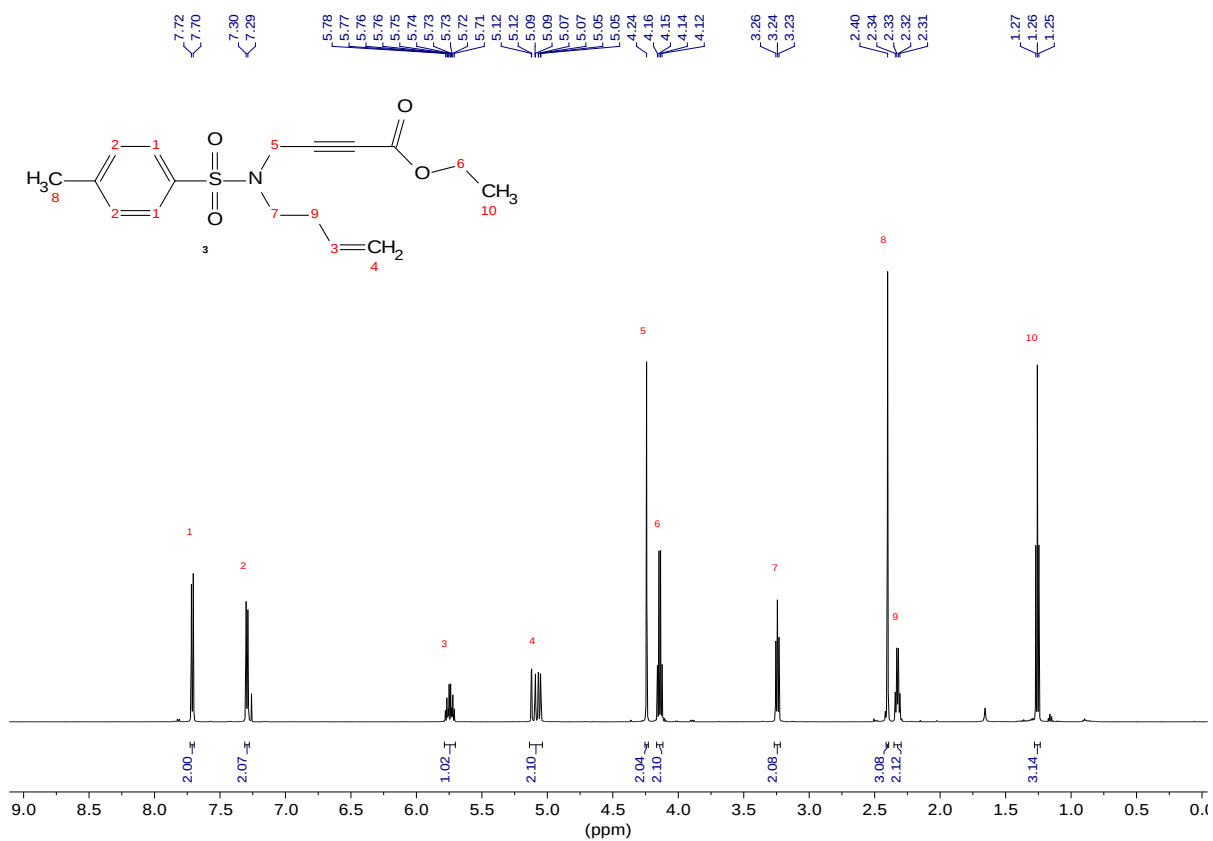
[§]Equally contributing authors

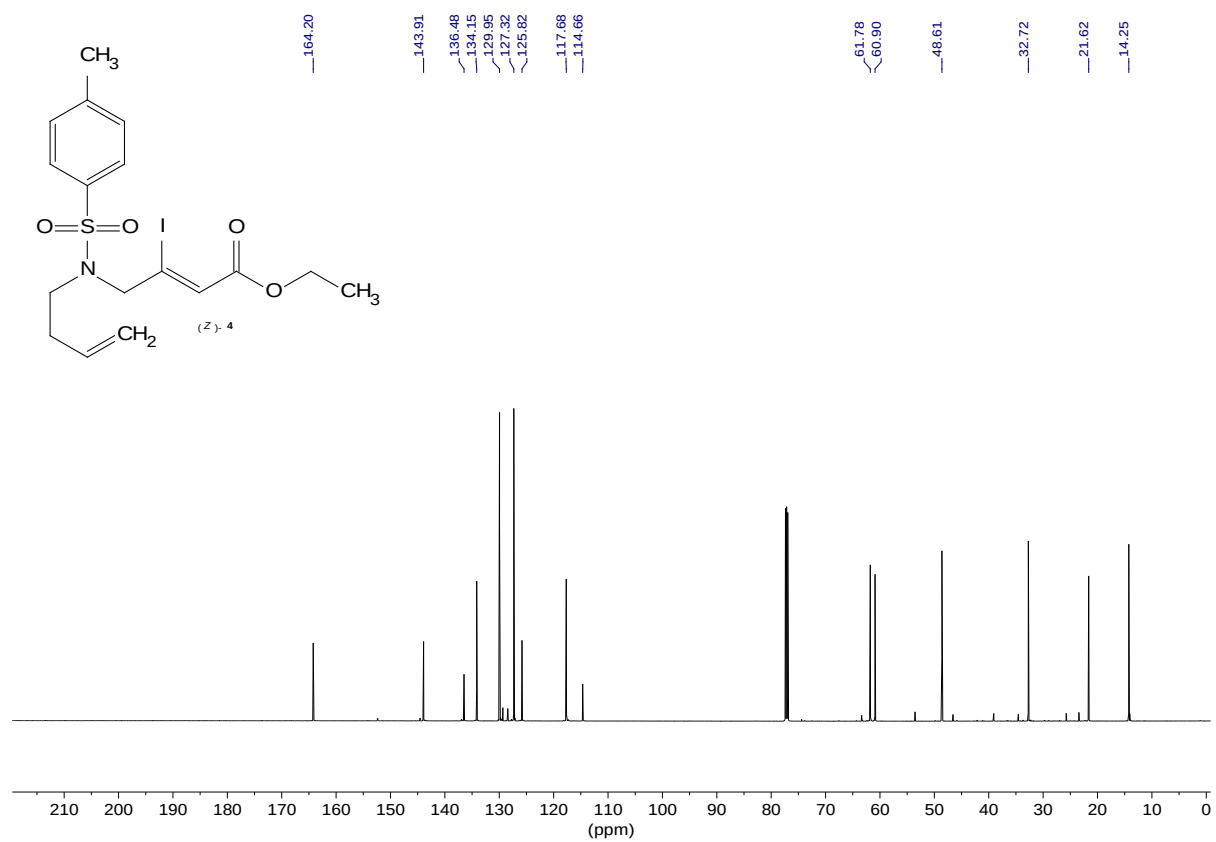
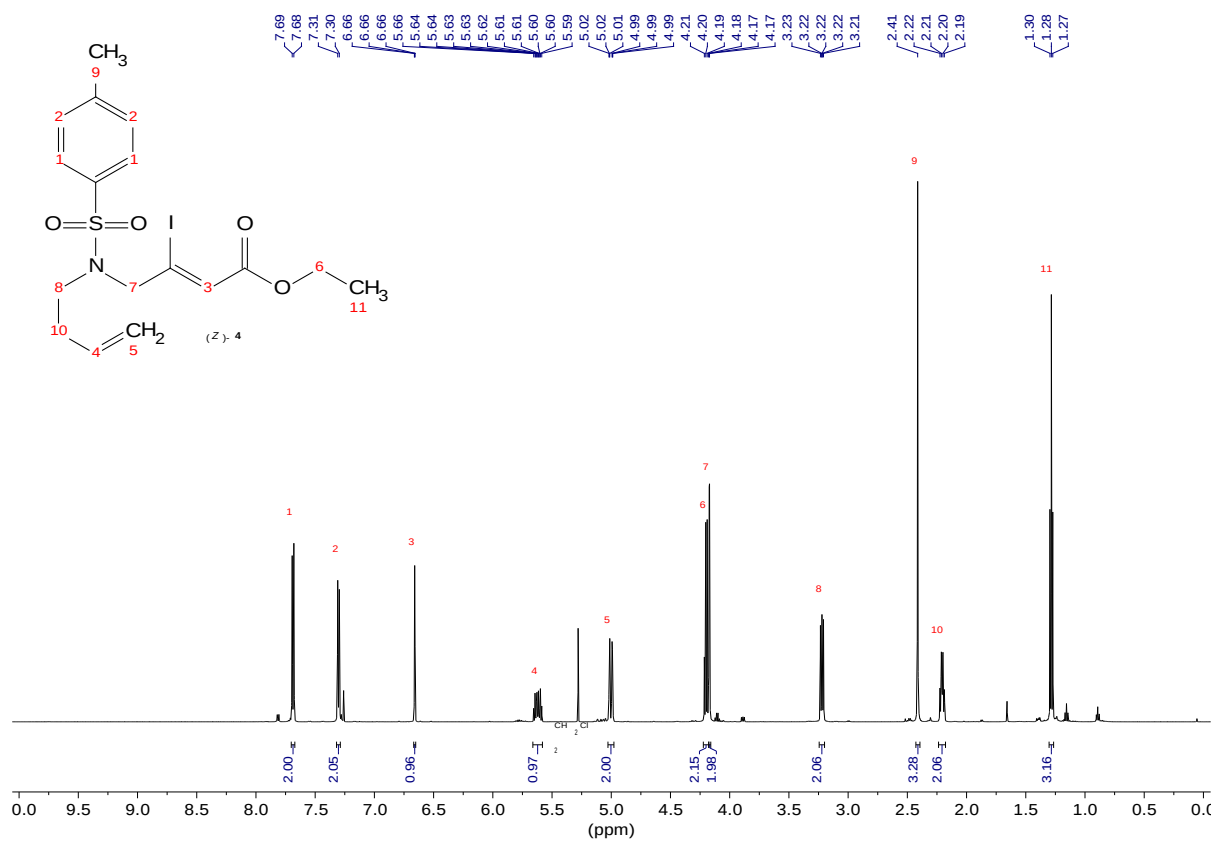
Copies of NMR spectra

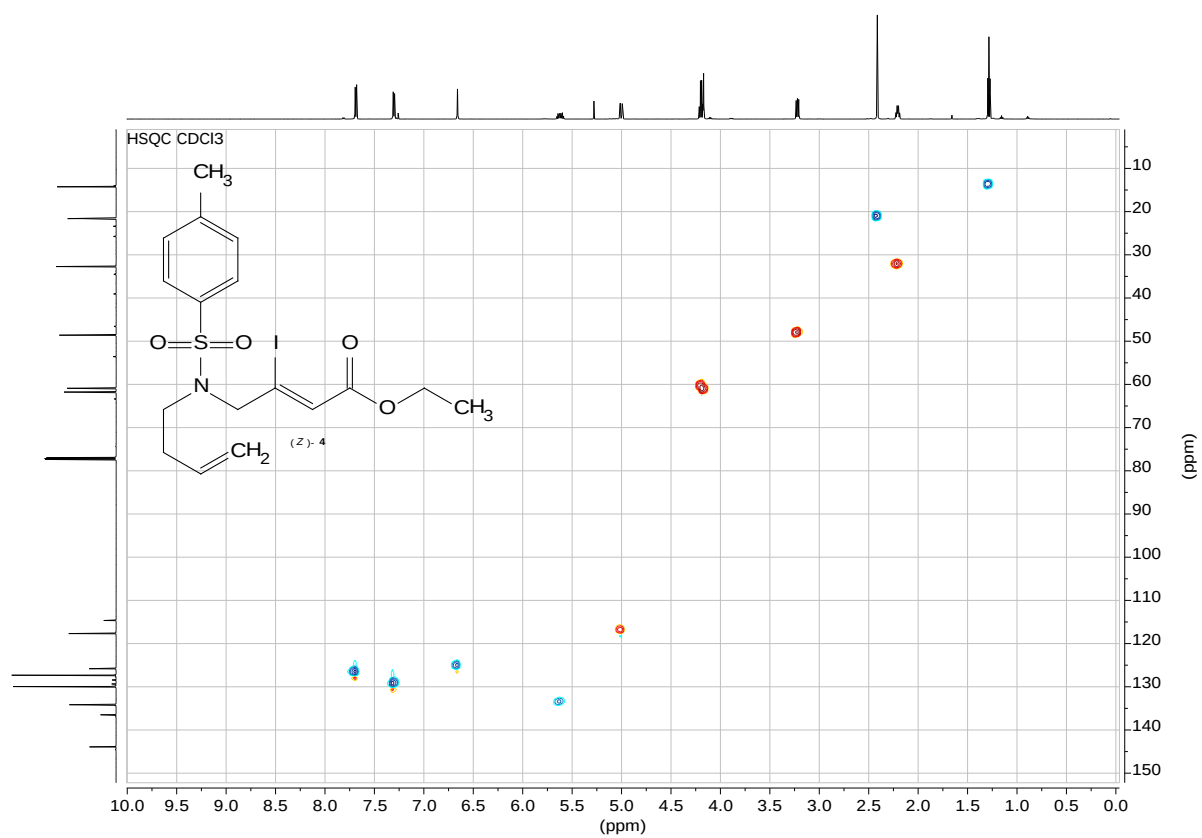
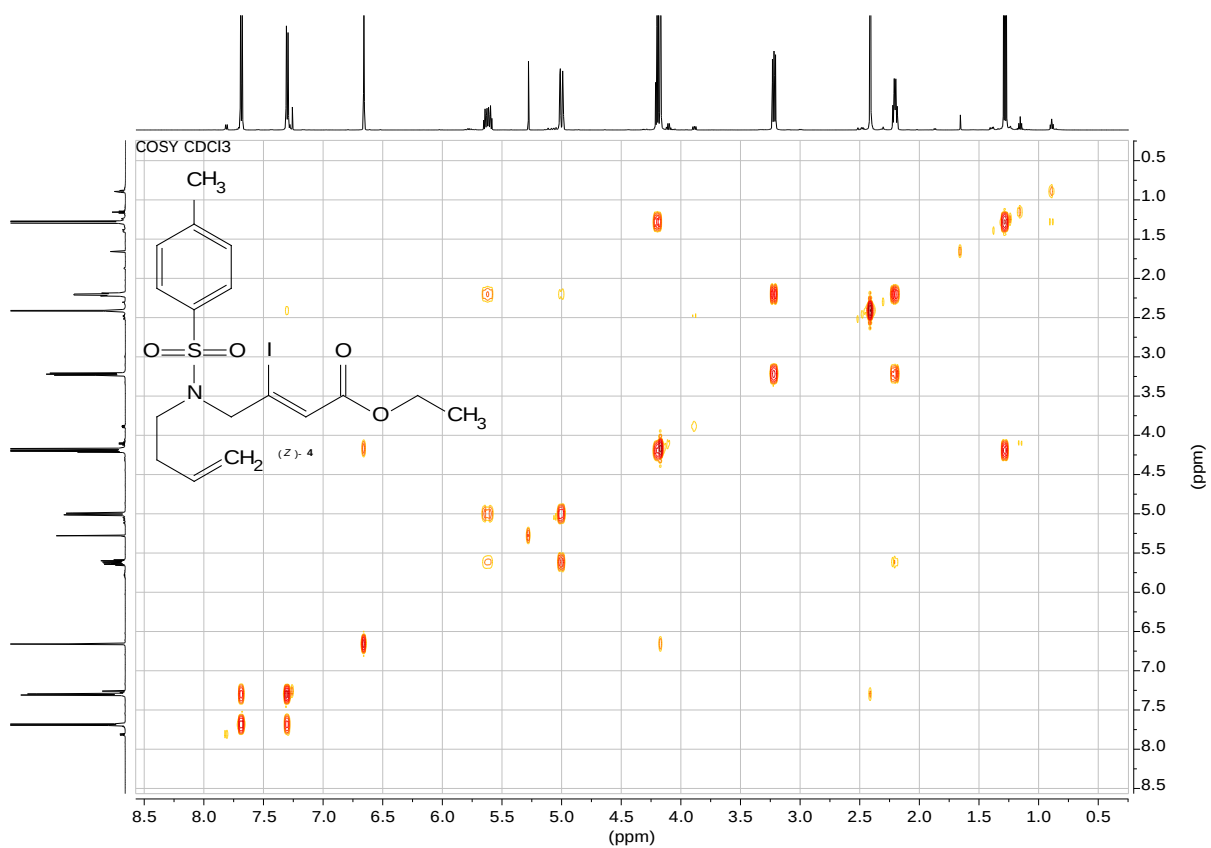


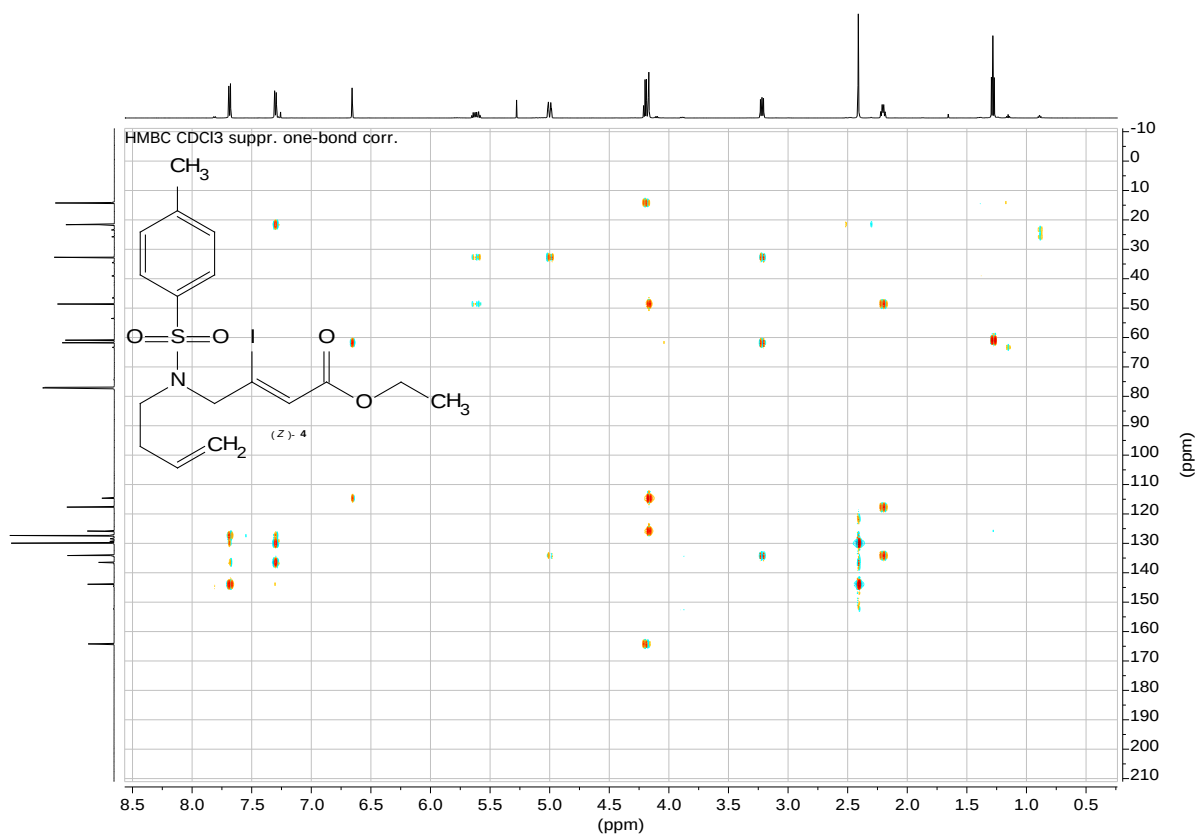


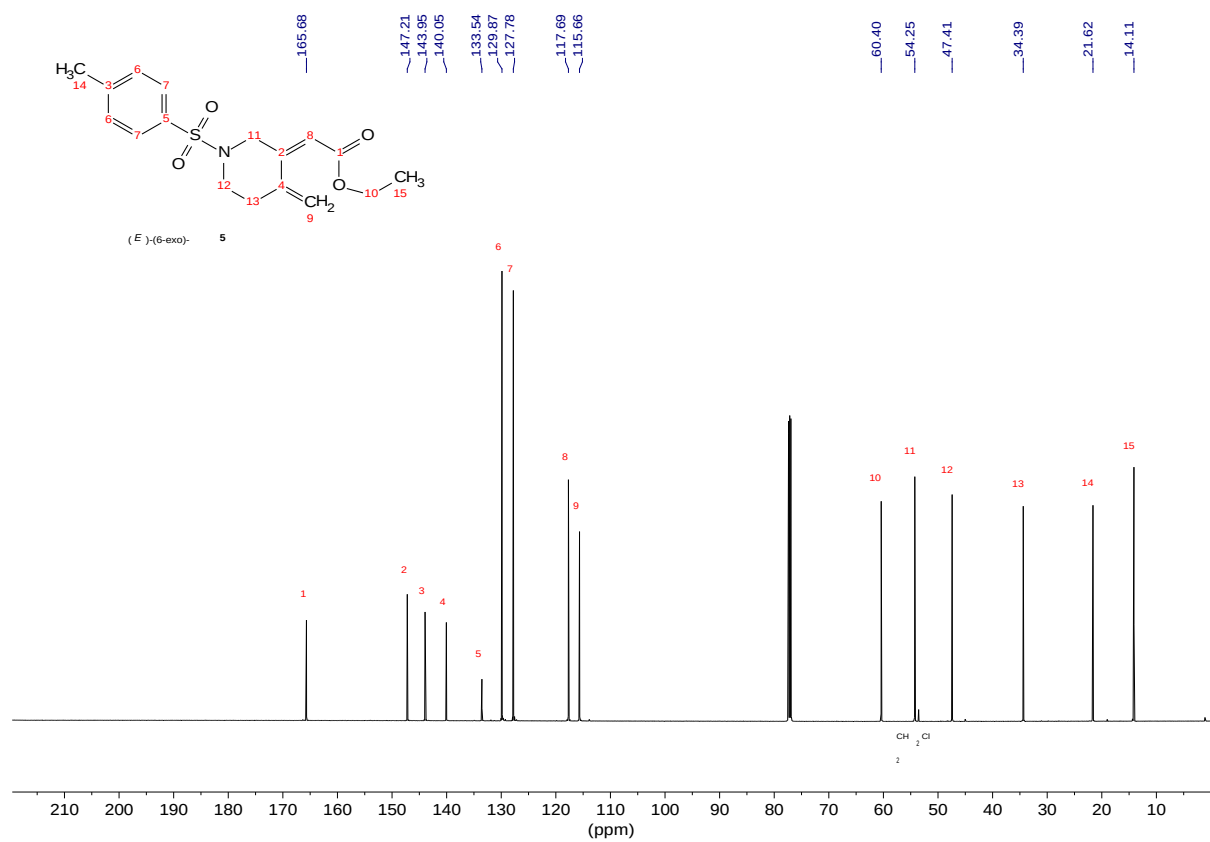
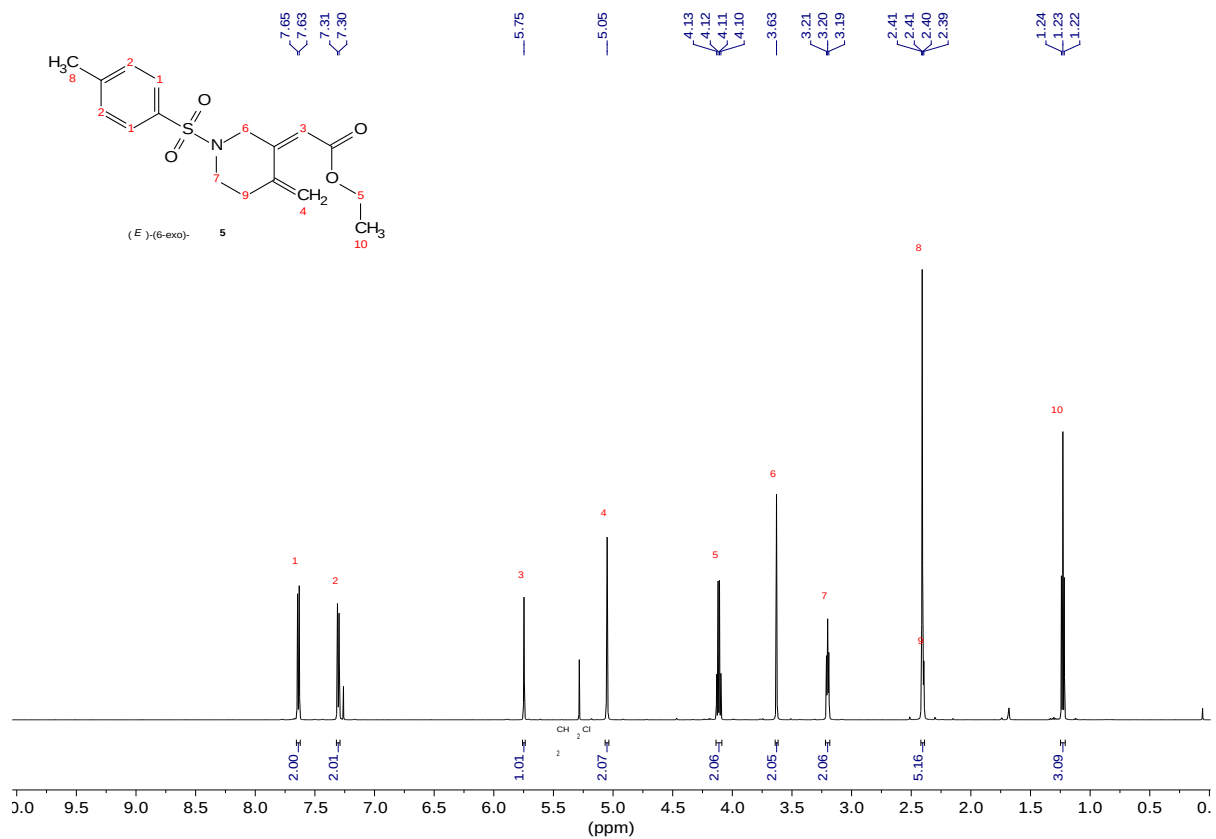


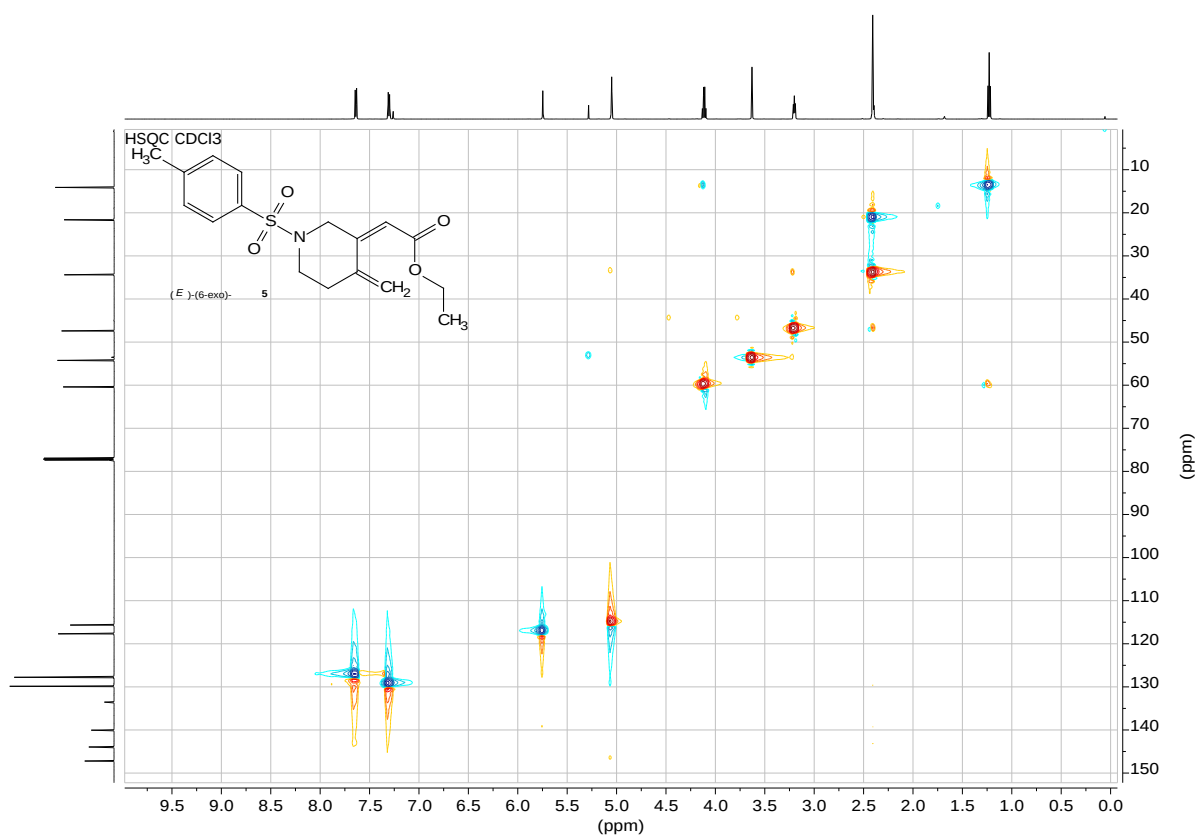
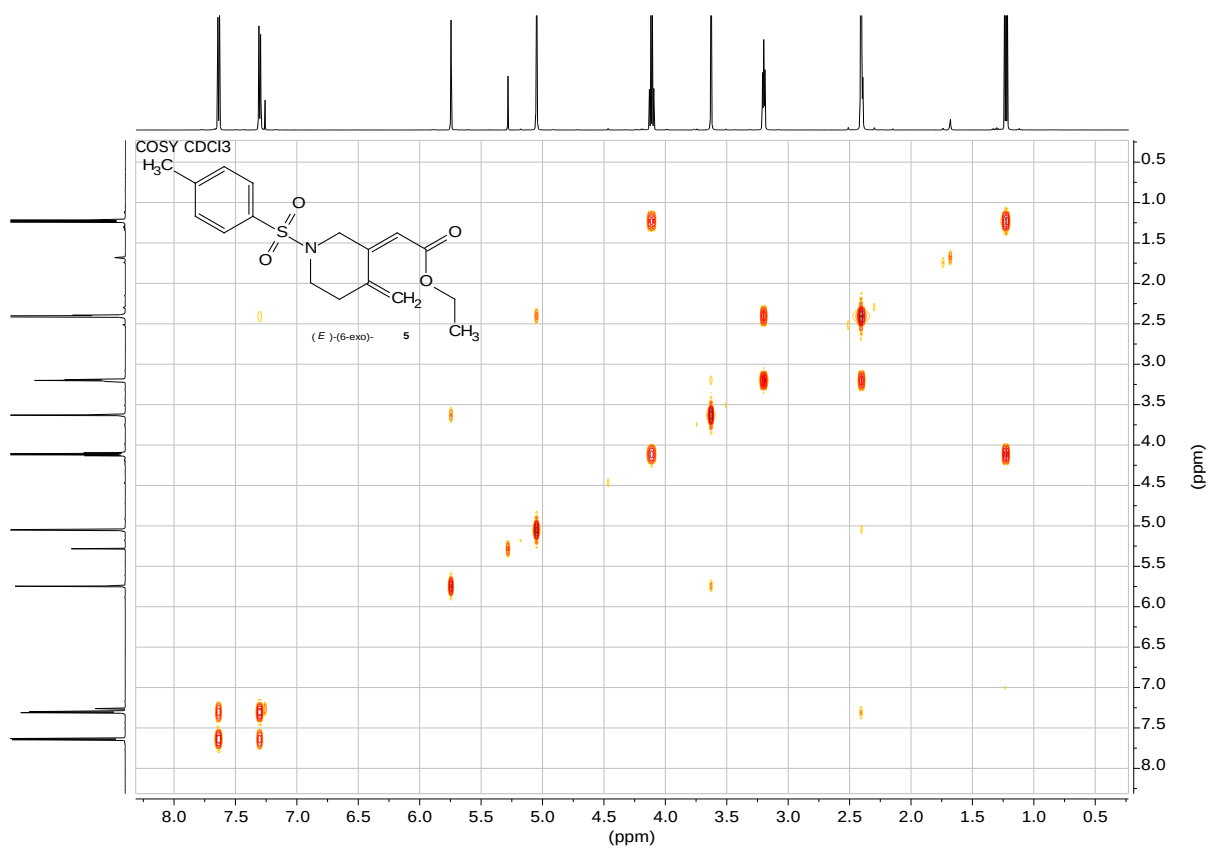


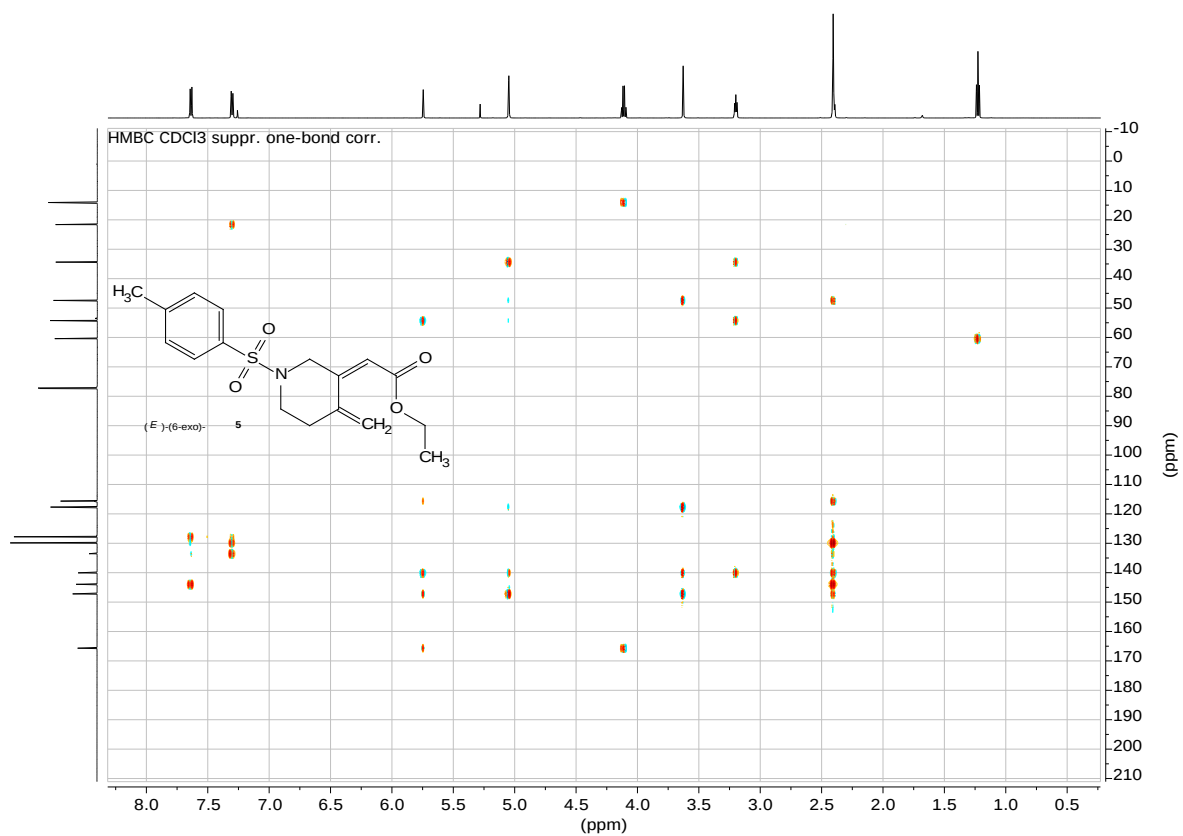


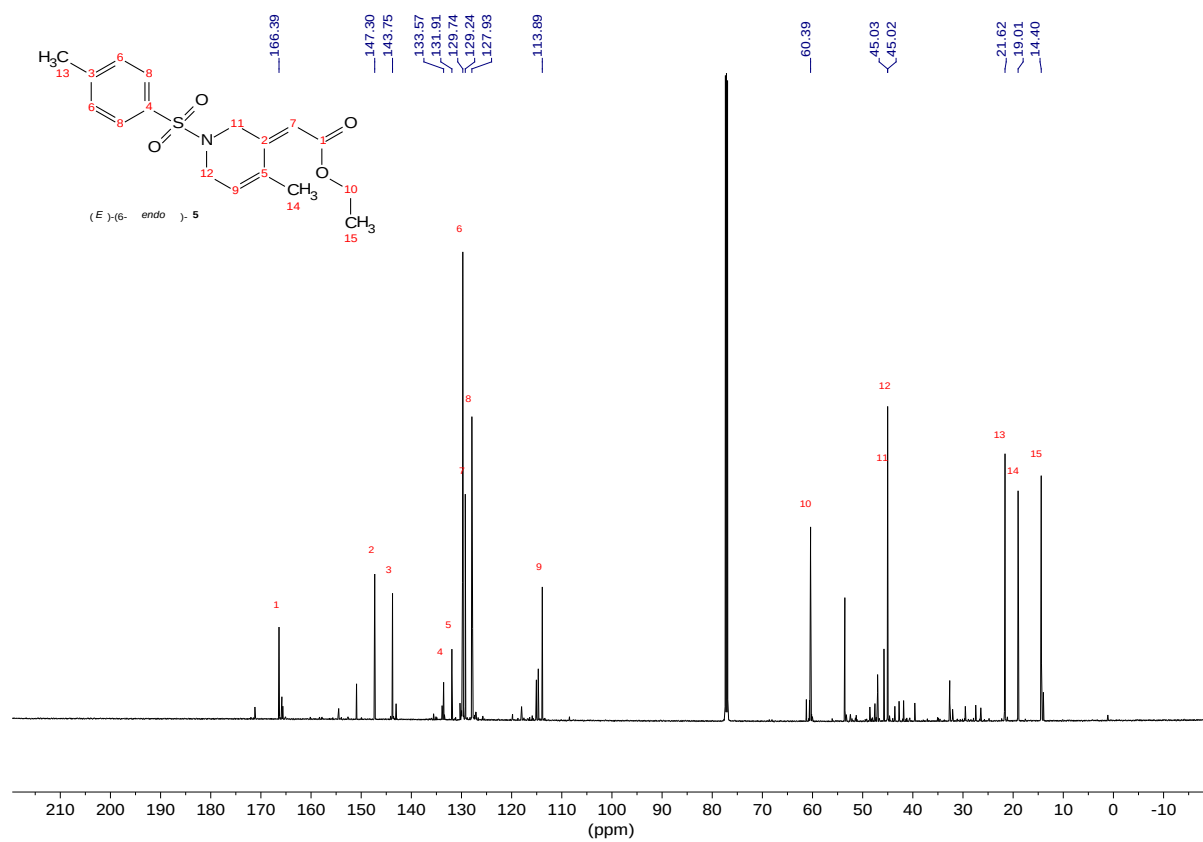
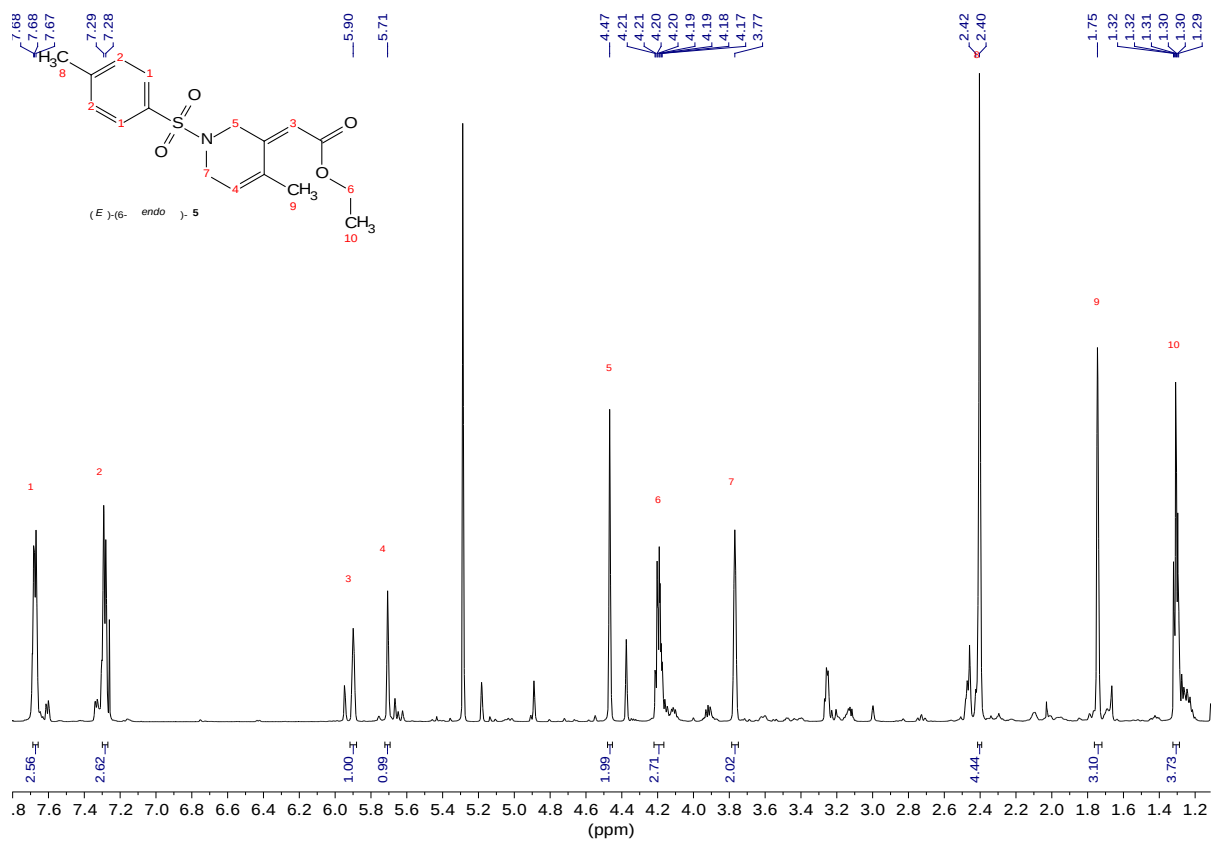


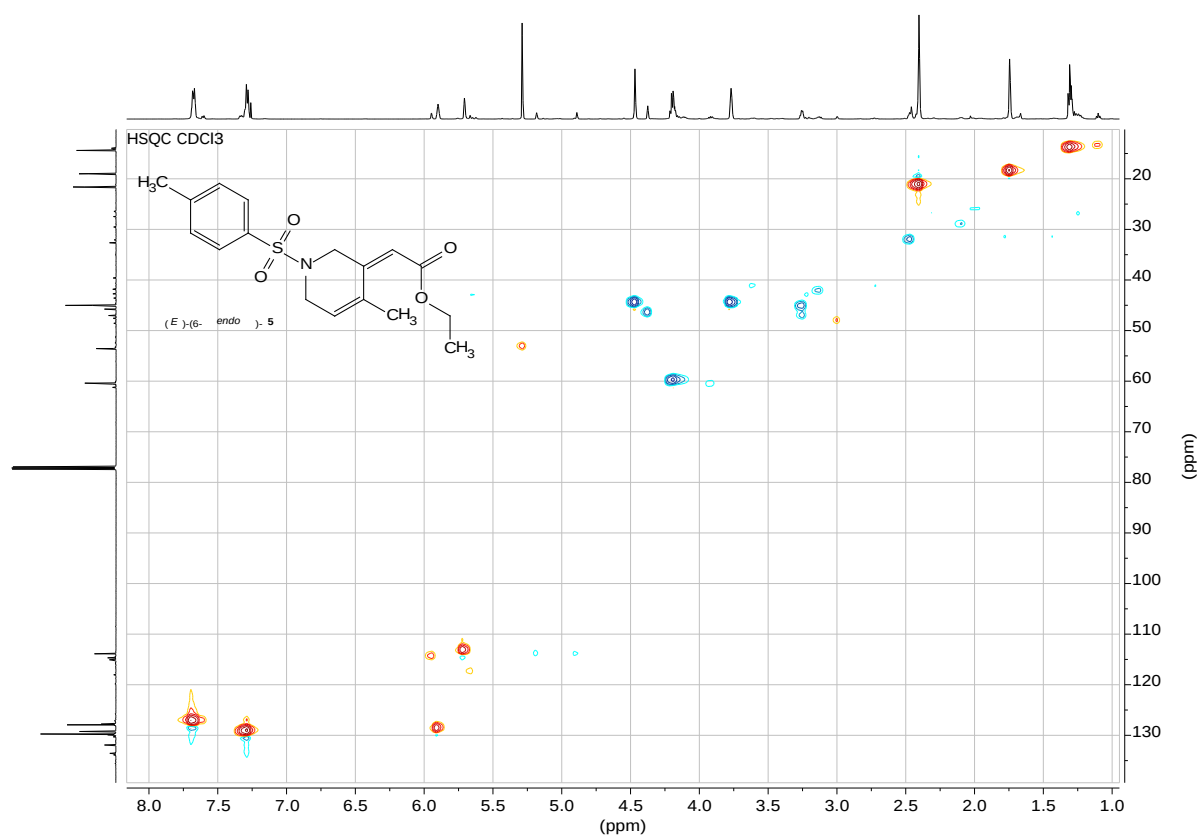
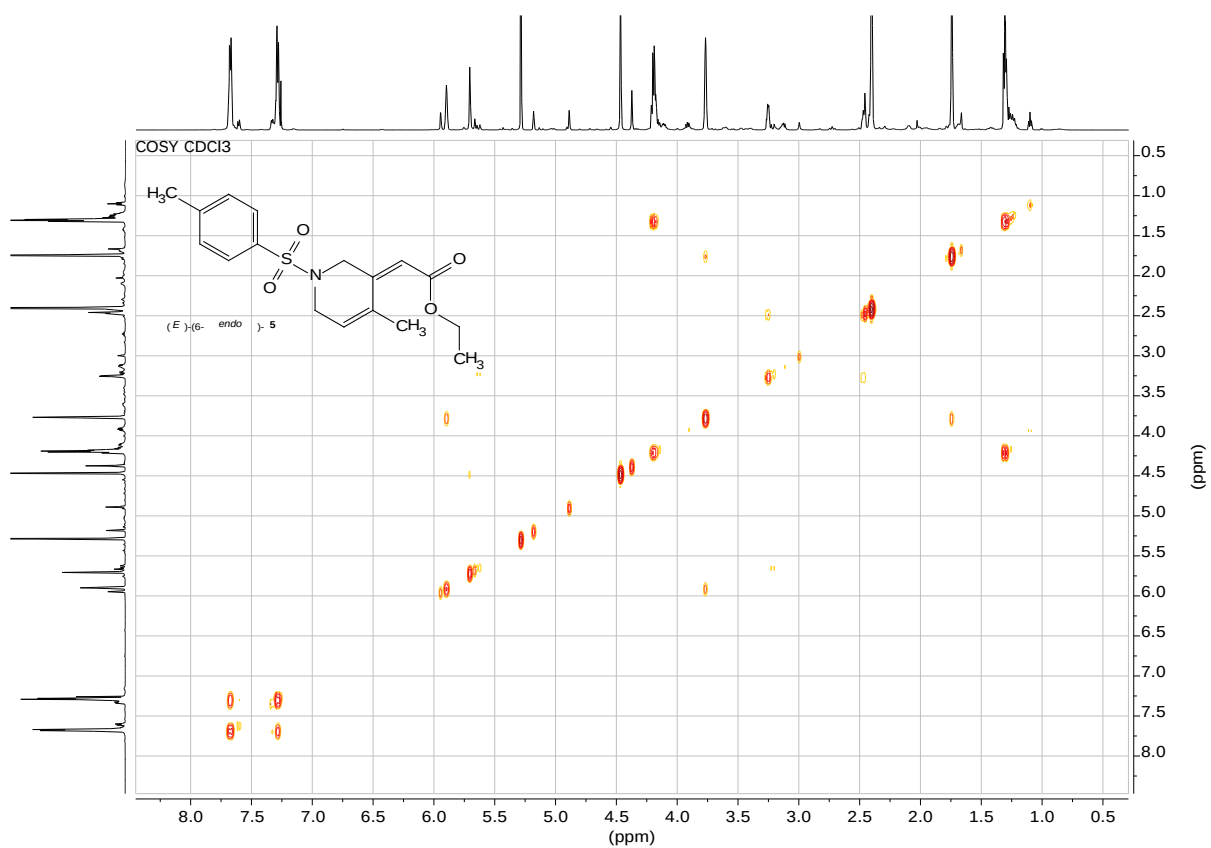


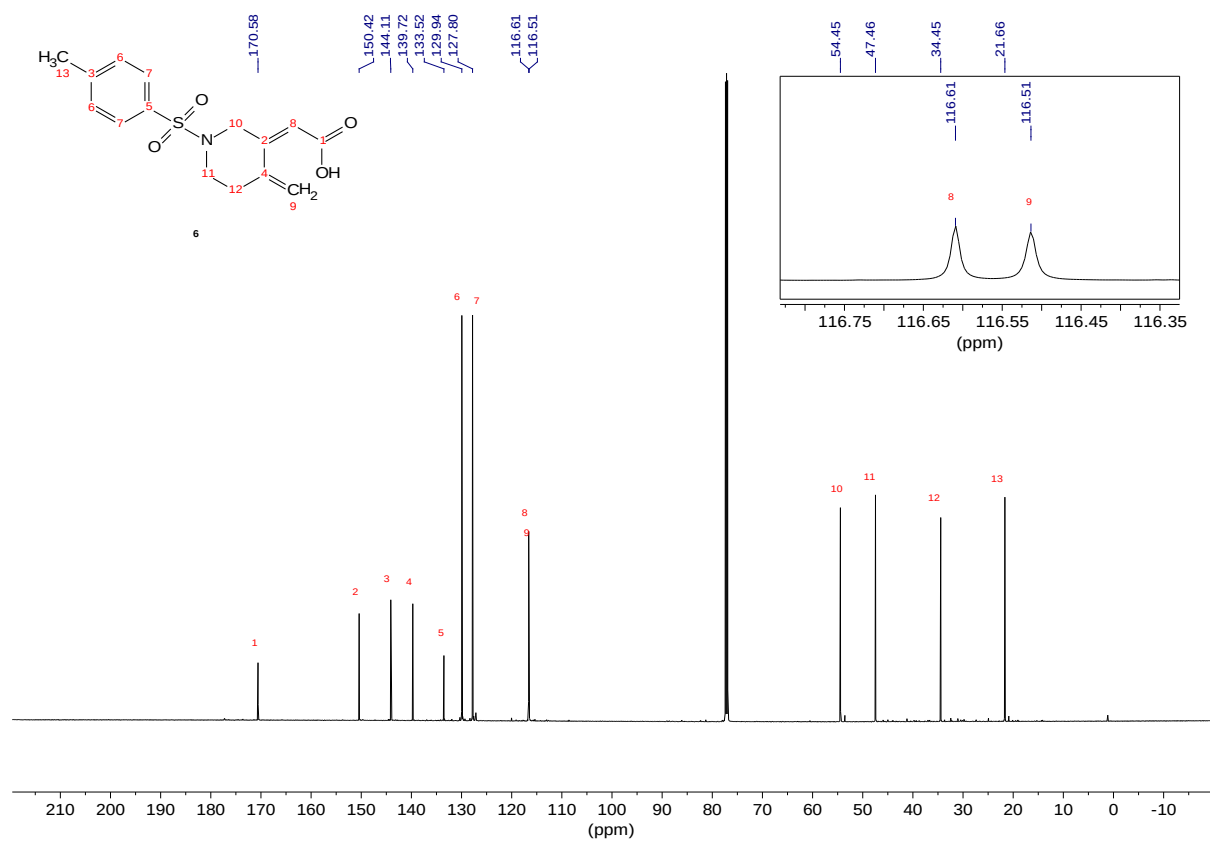
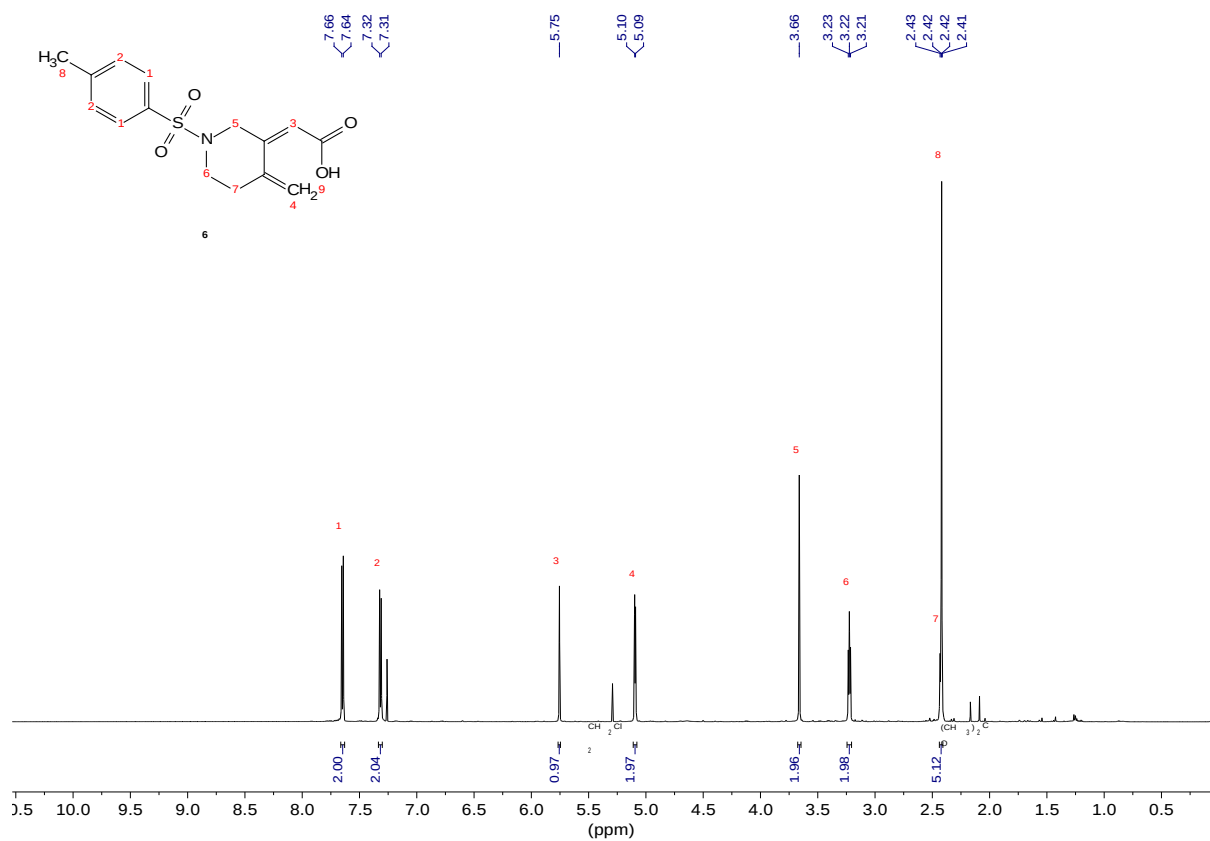


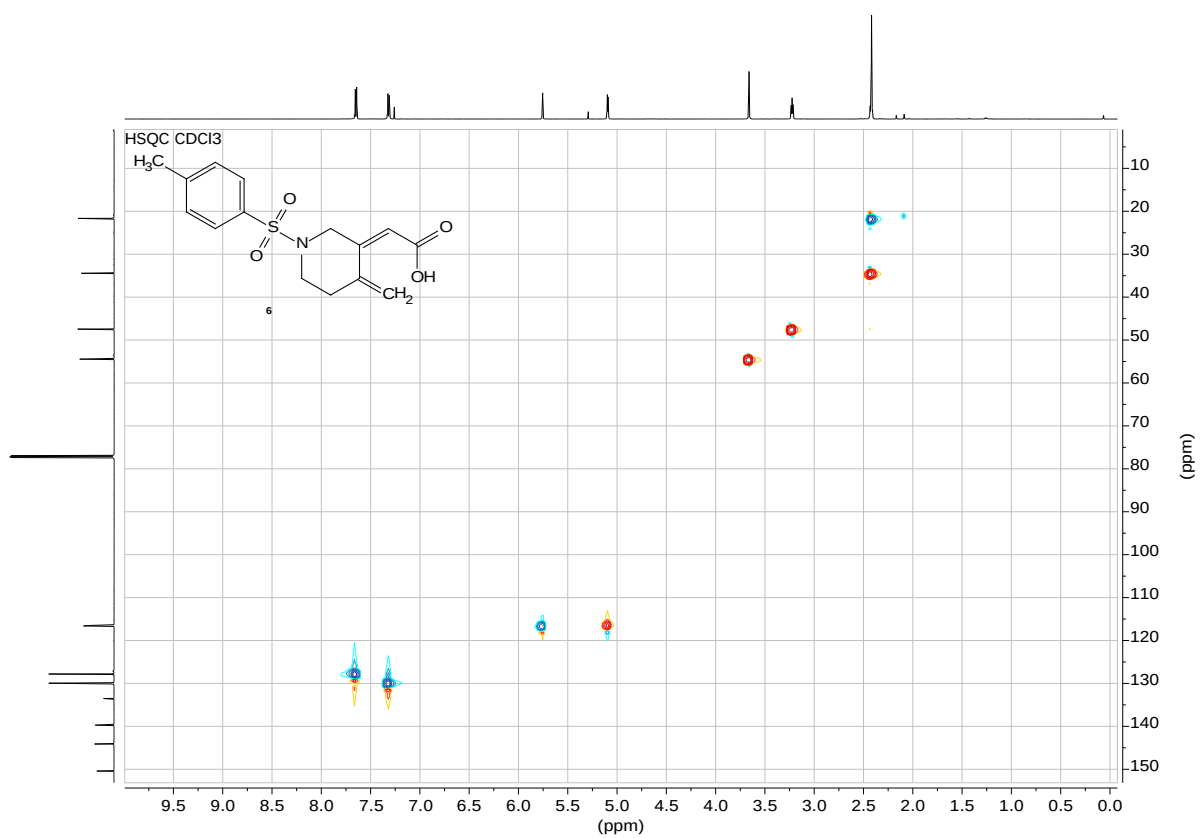
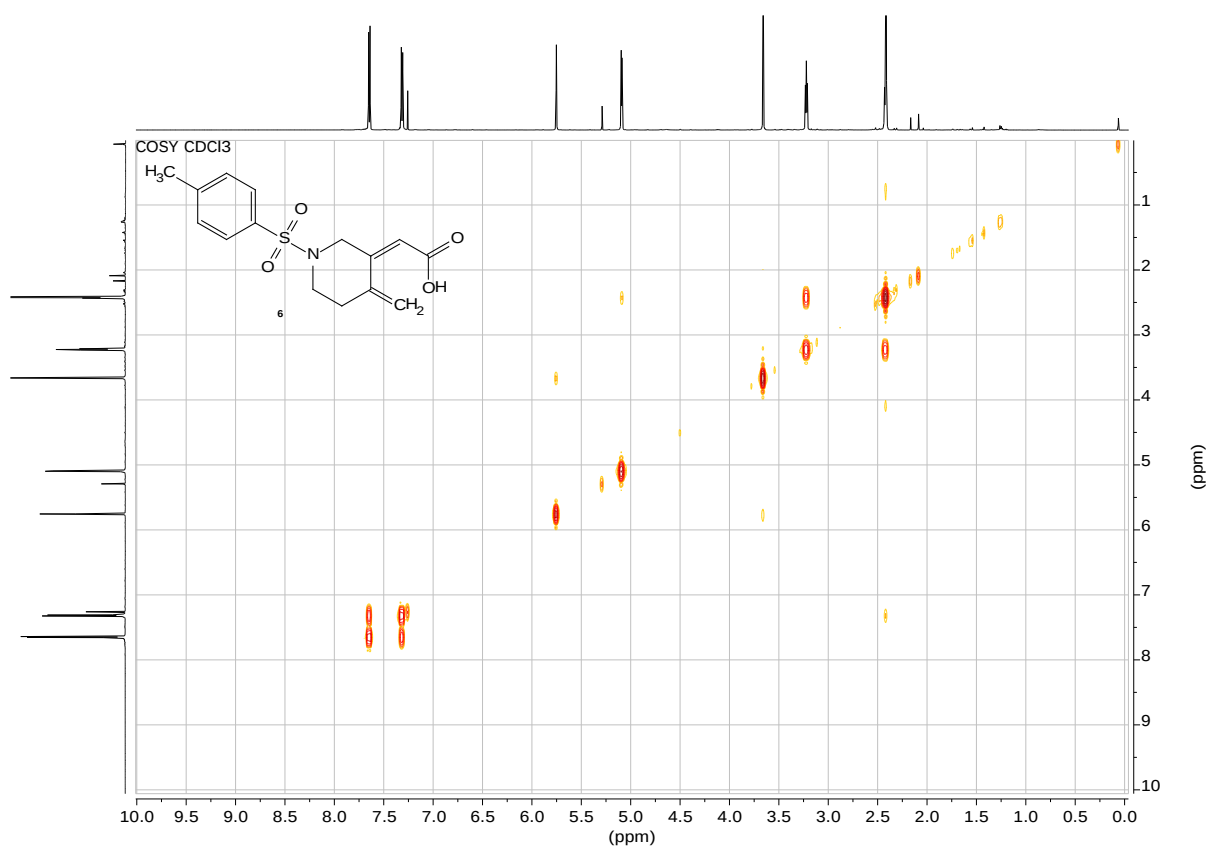


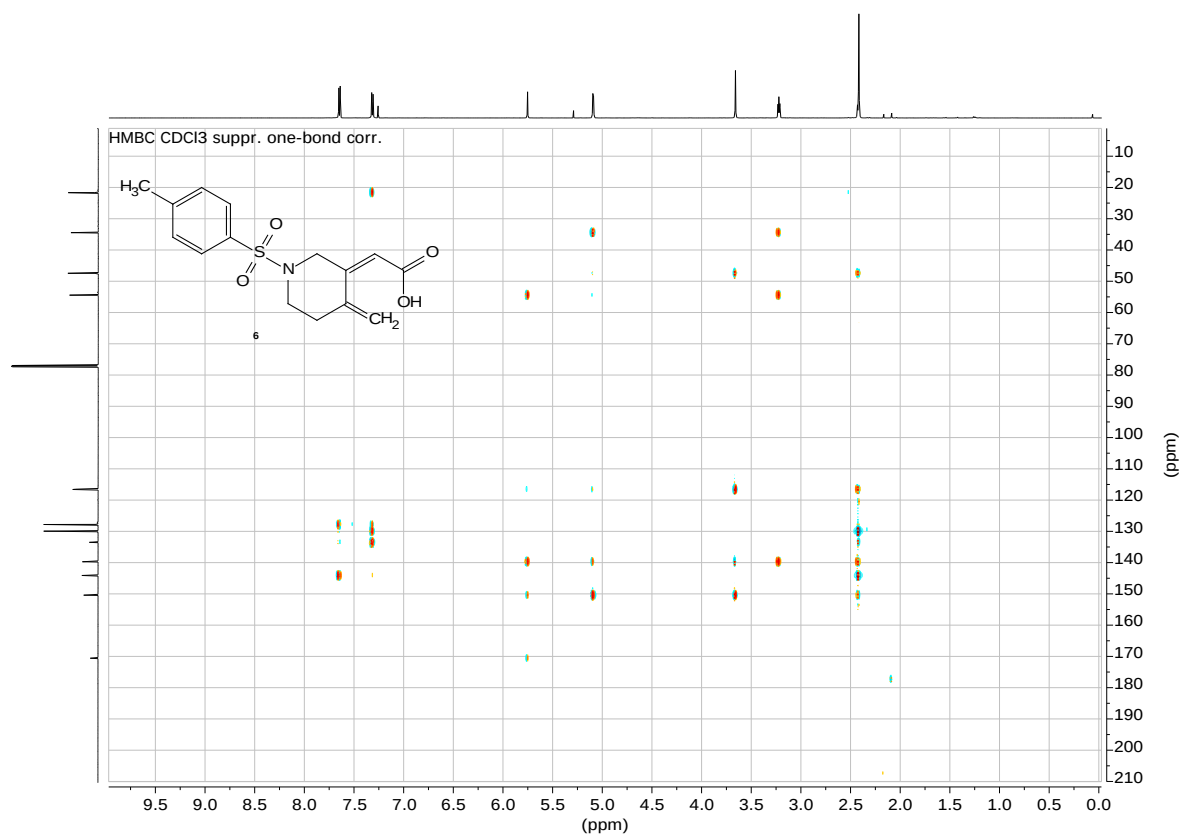


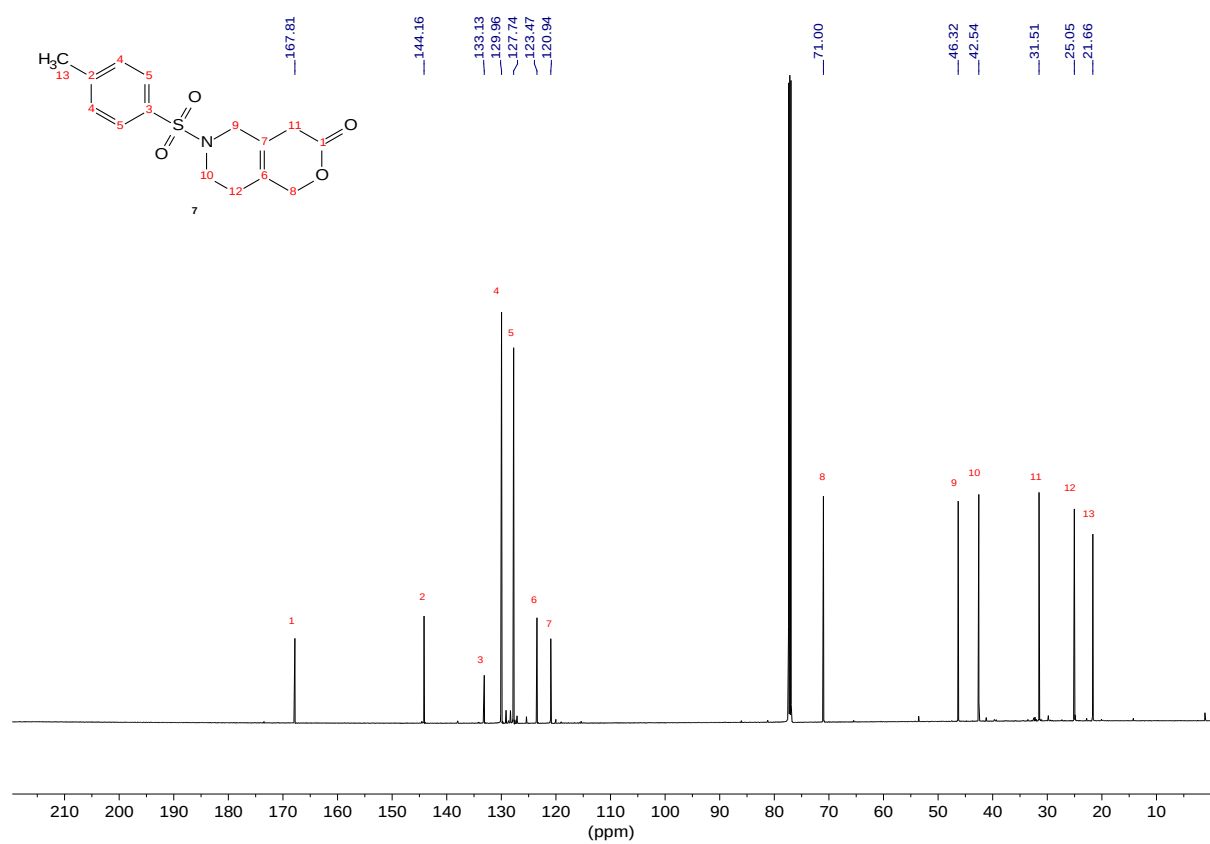
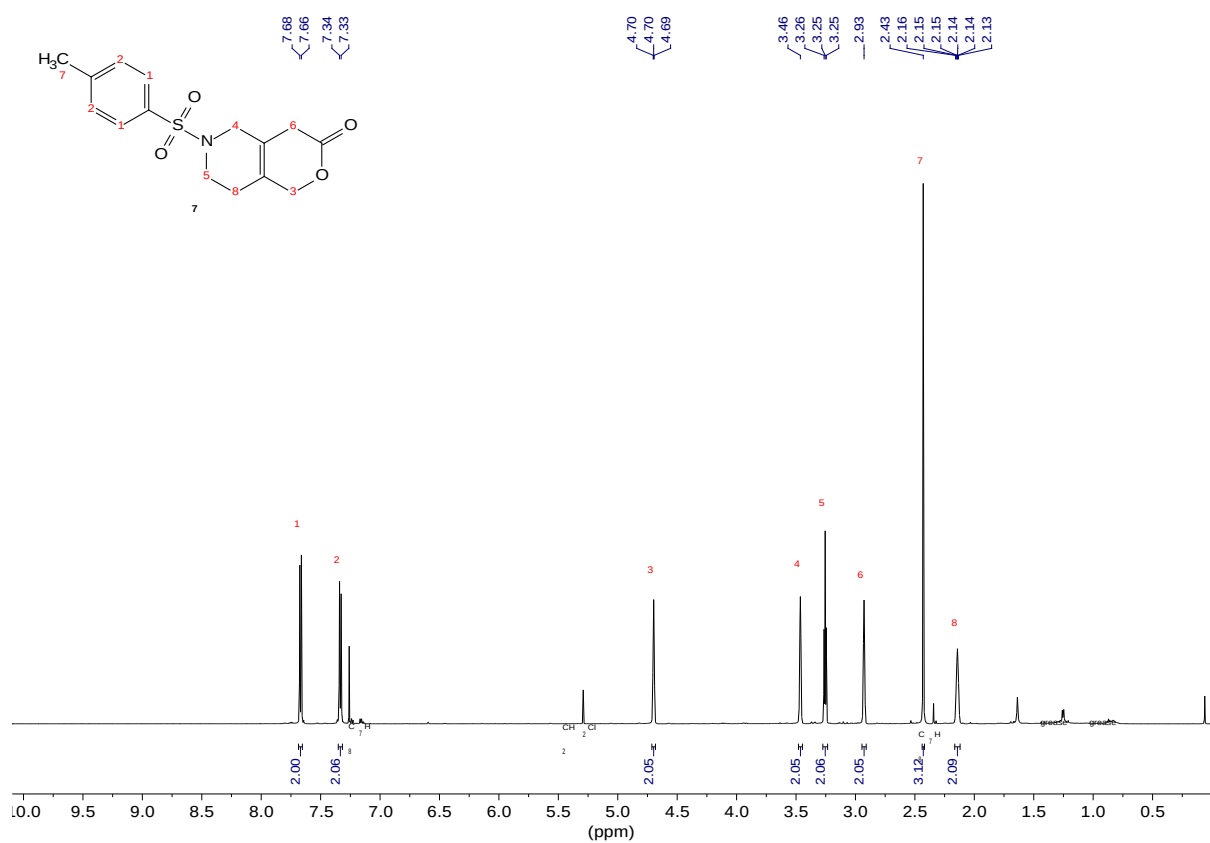


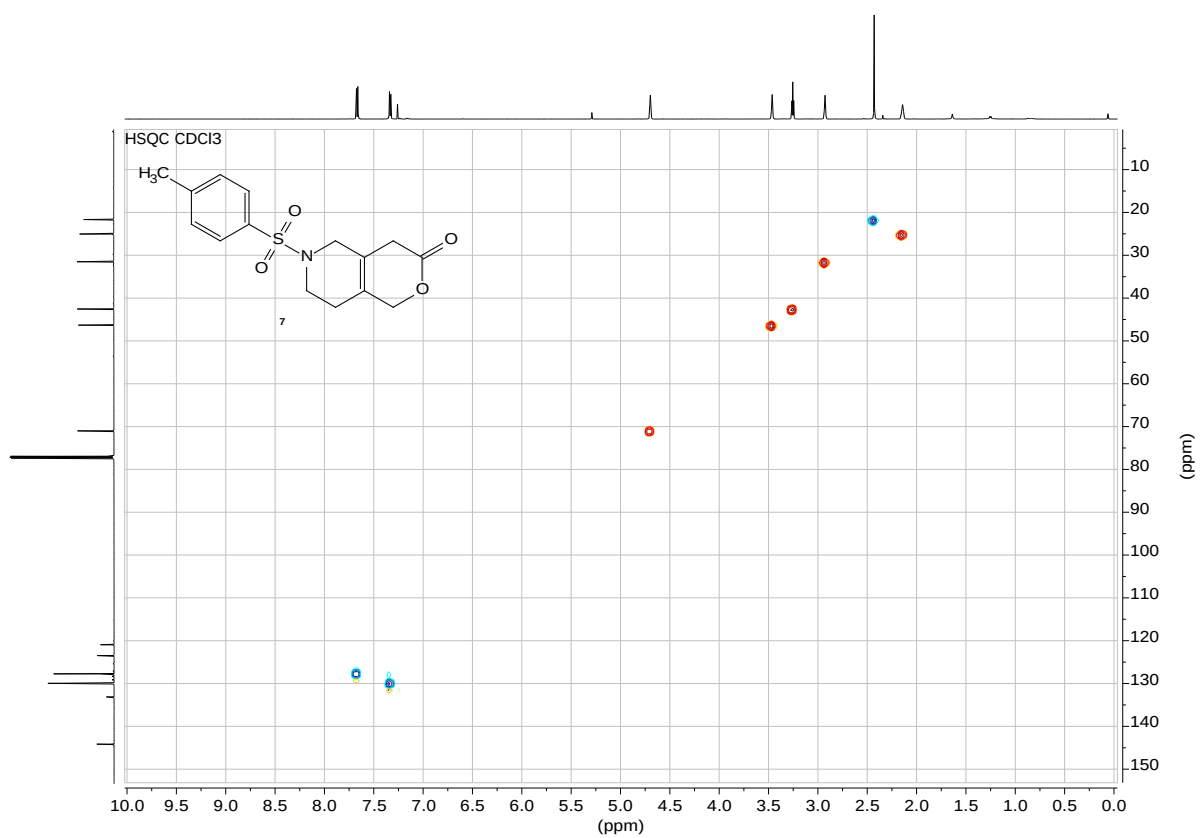
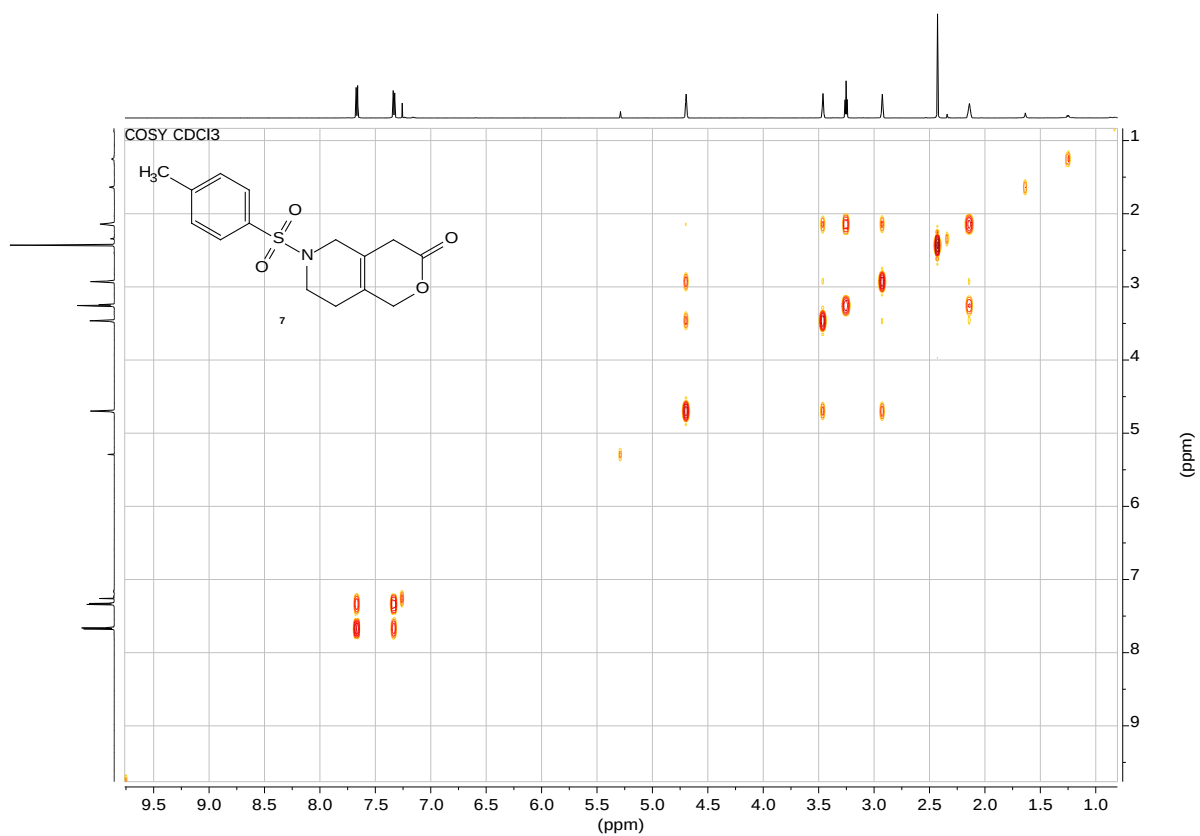


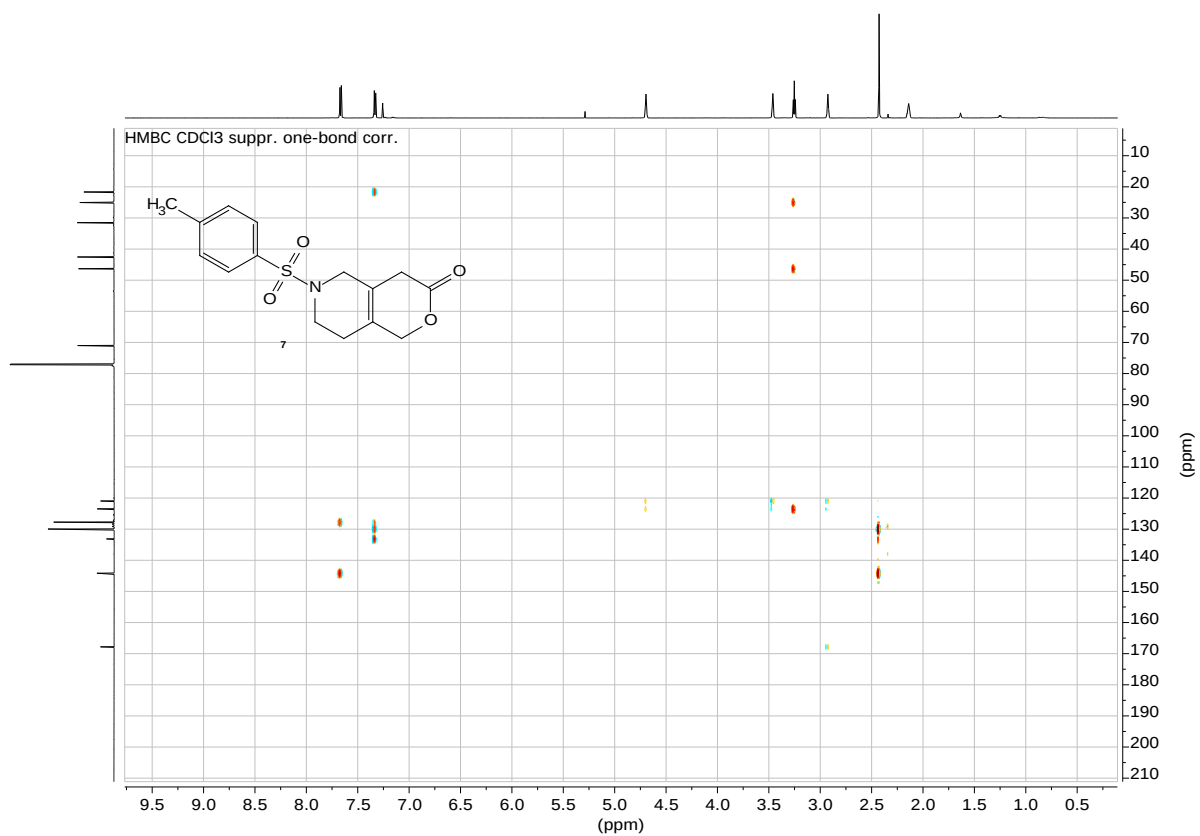


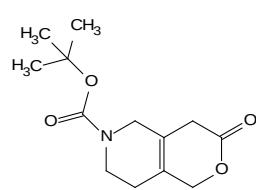




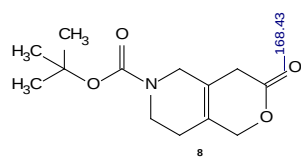
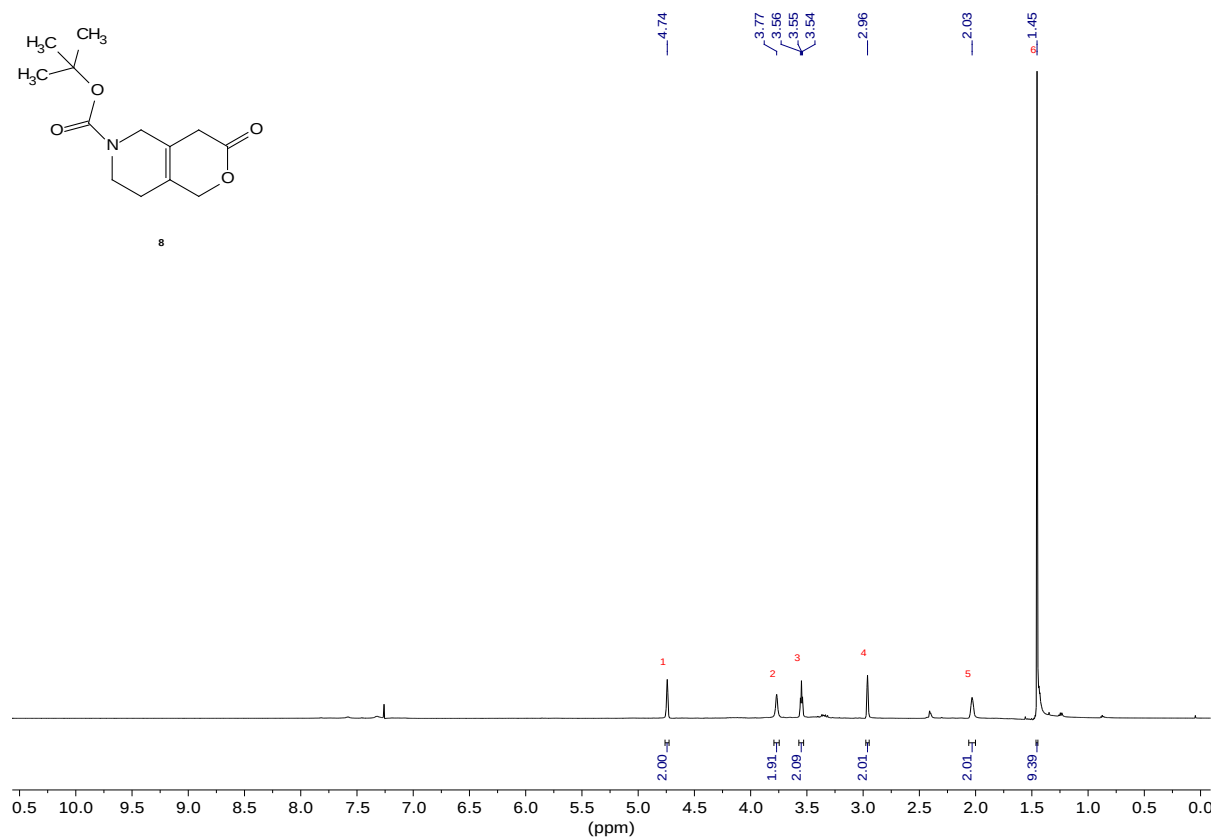




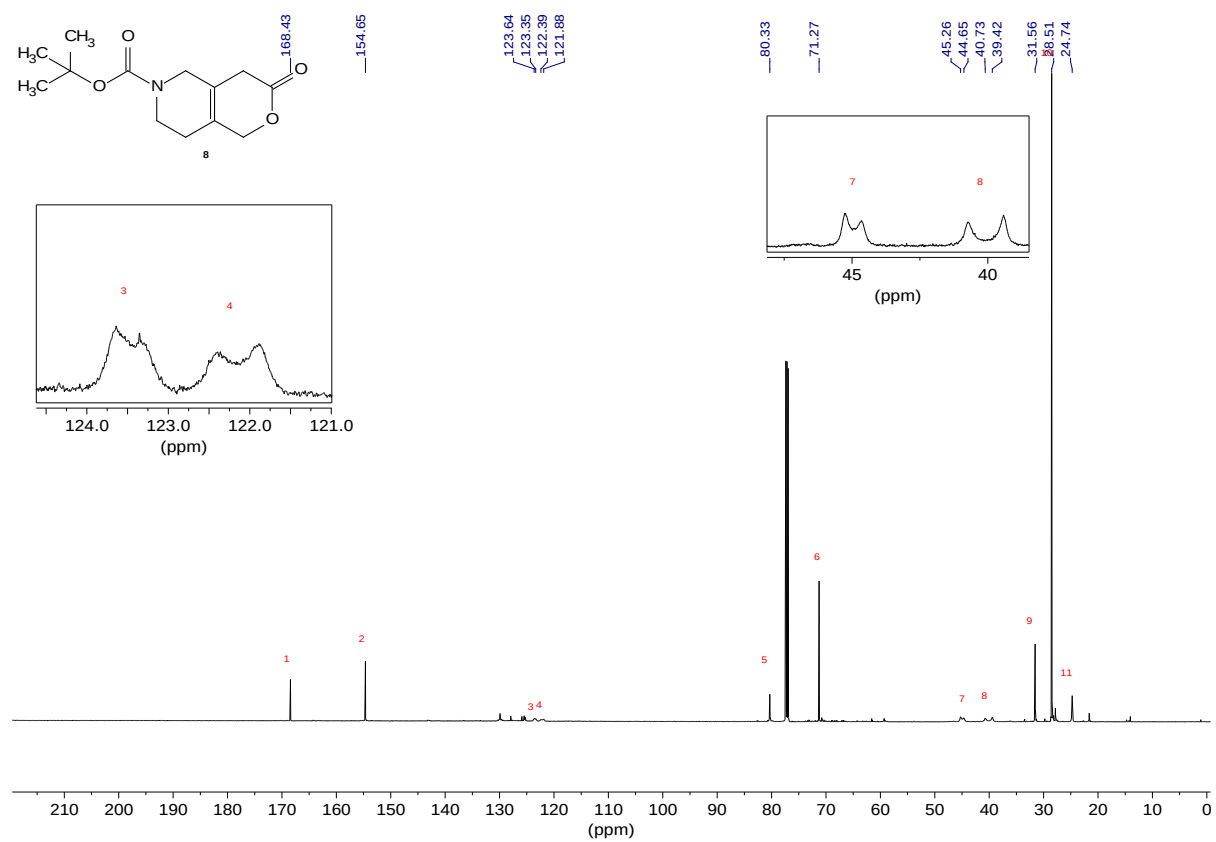


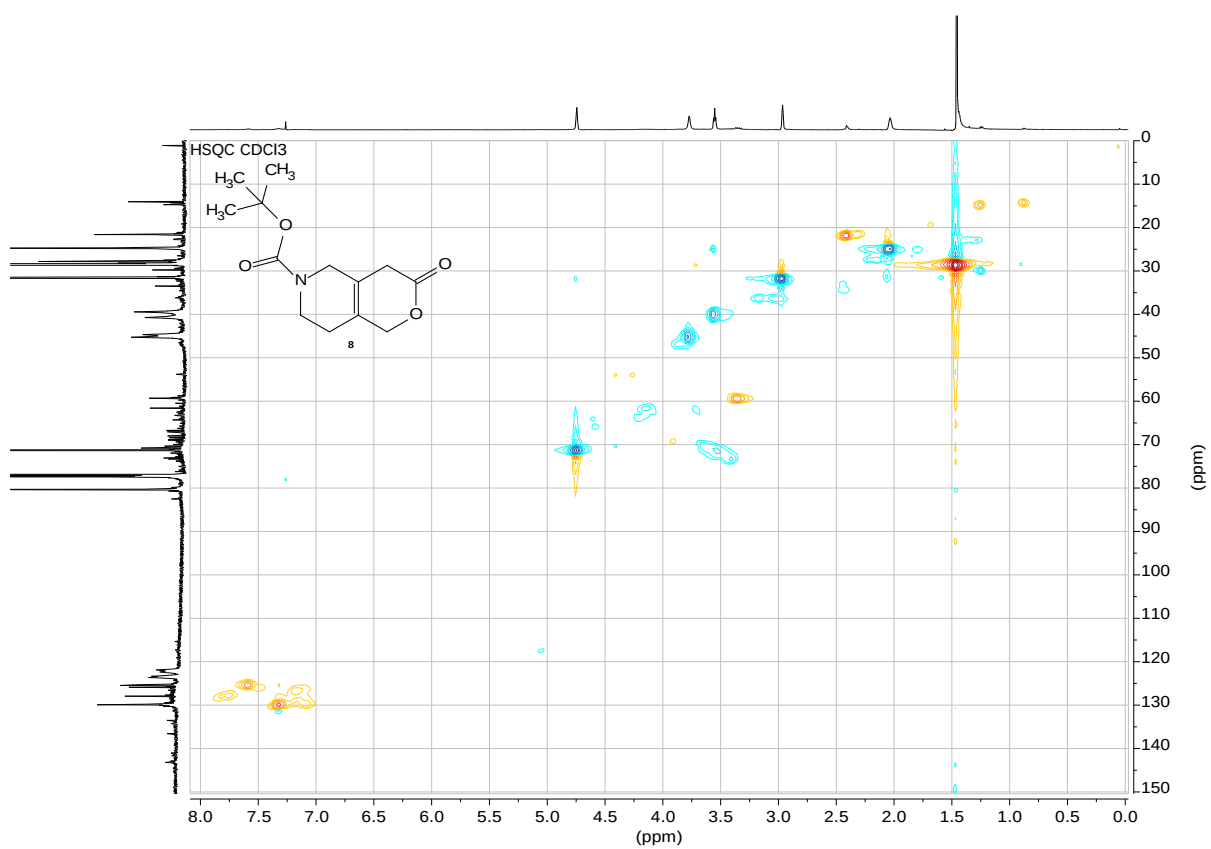
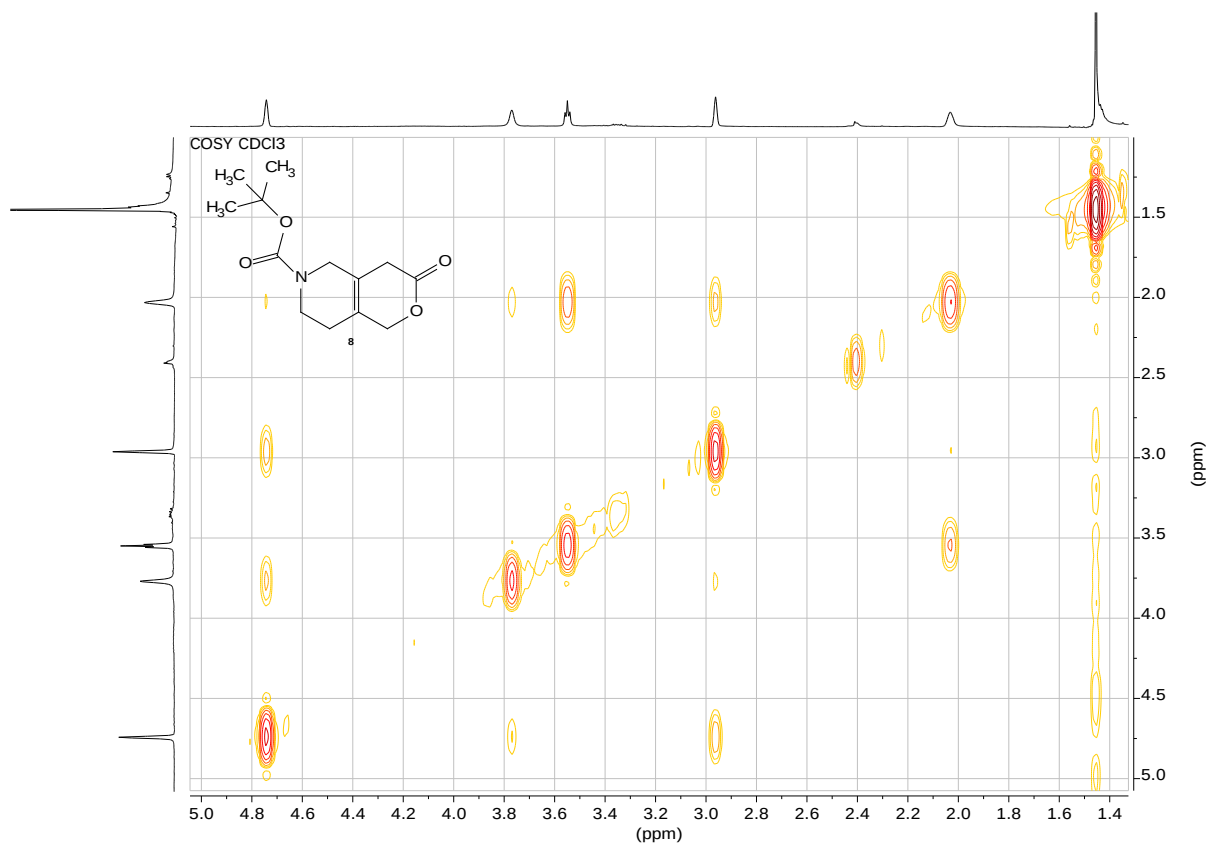


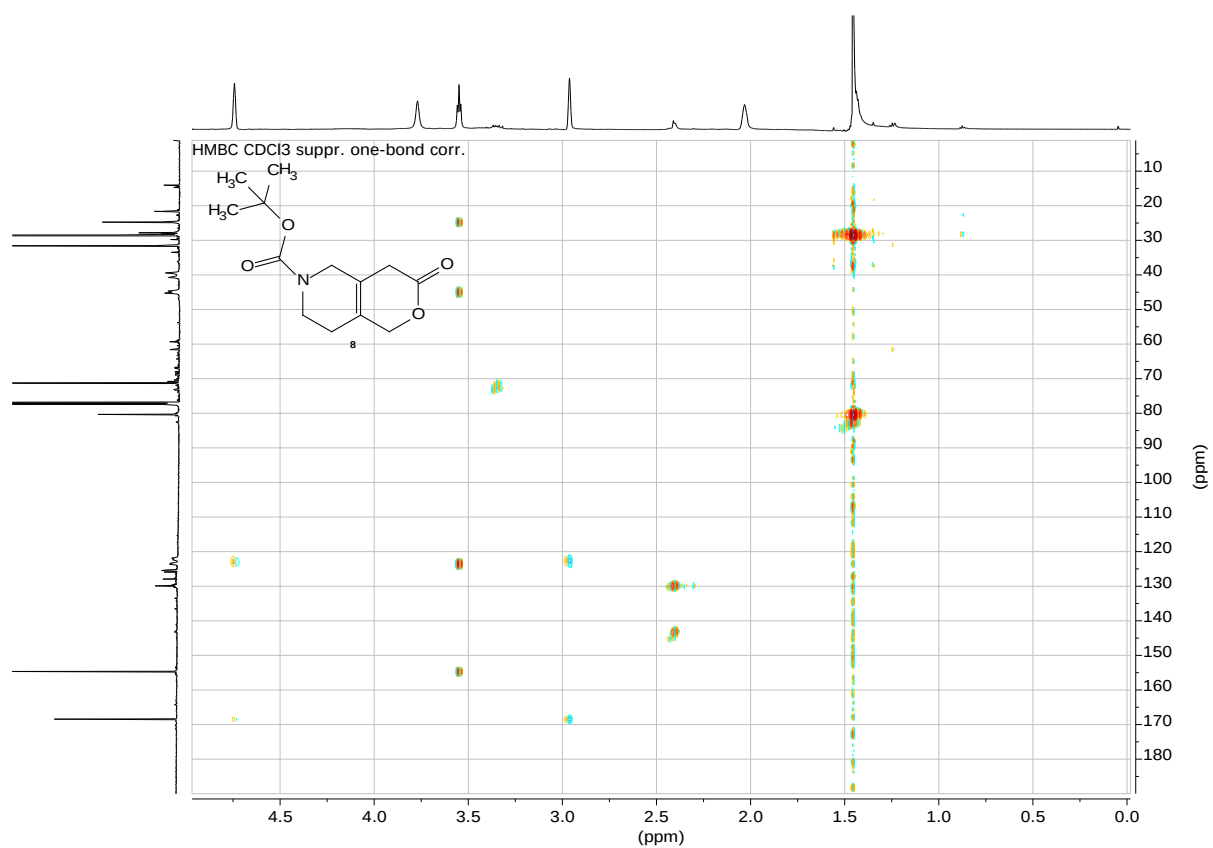
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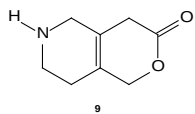
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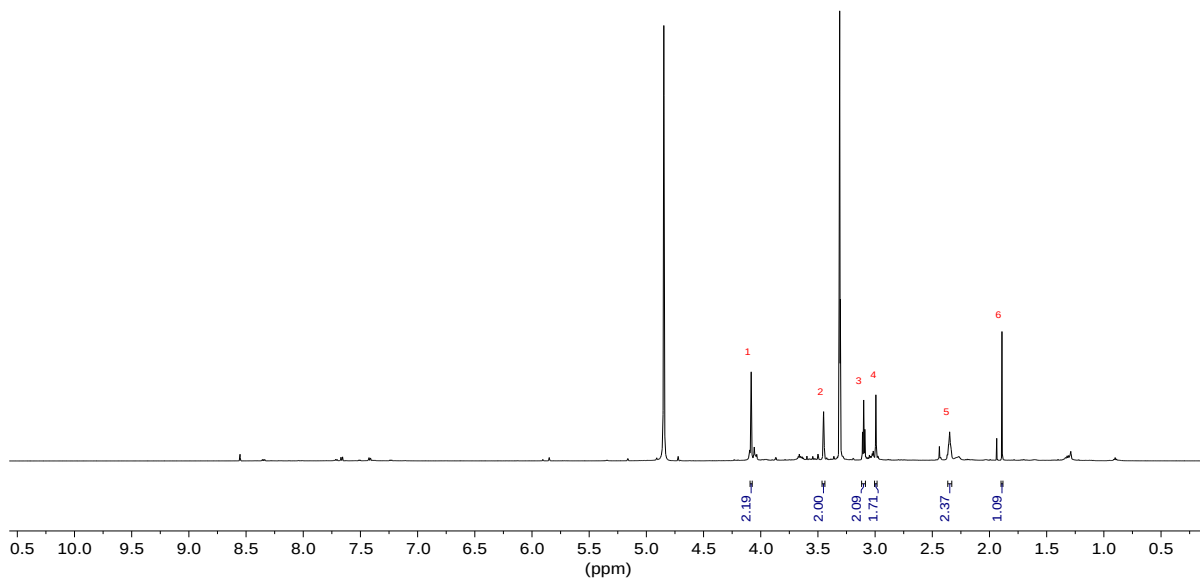




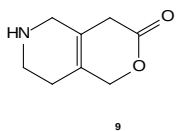
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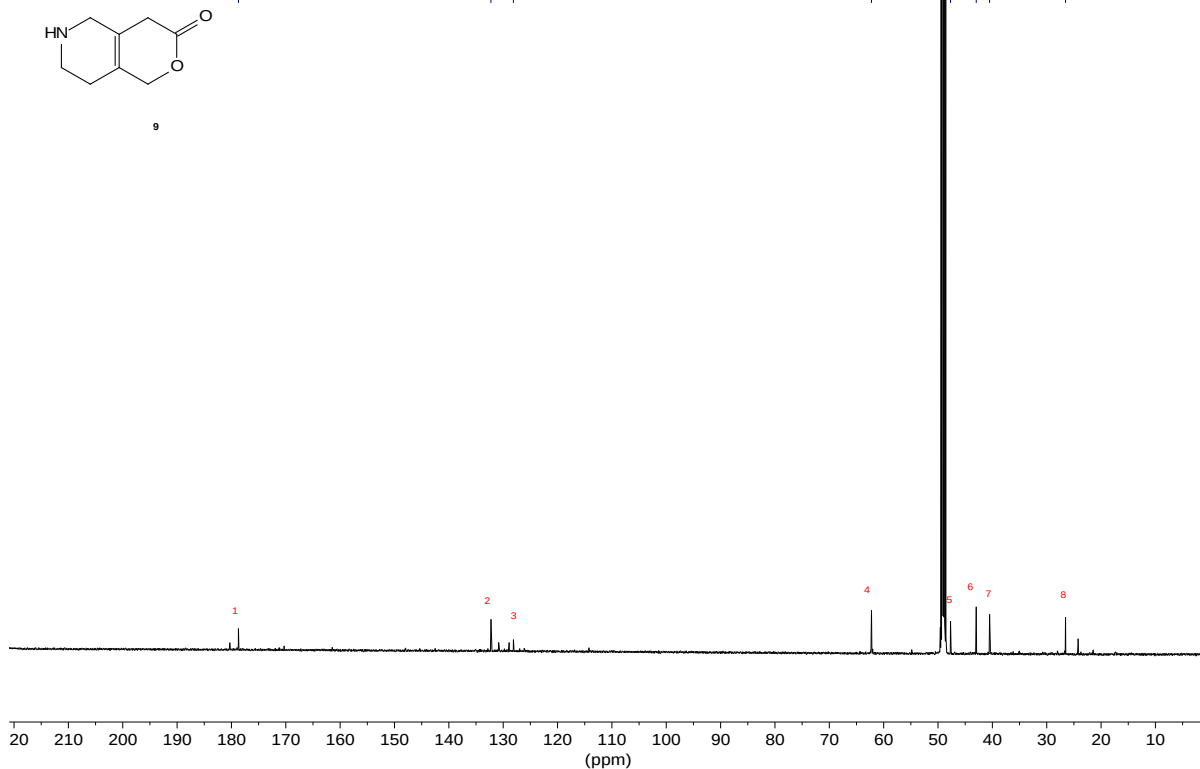
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2.35
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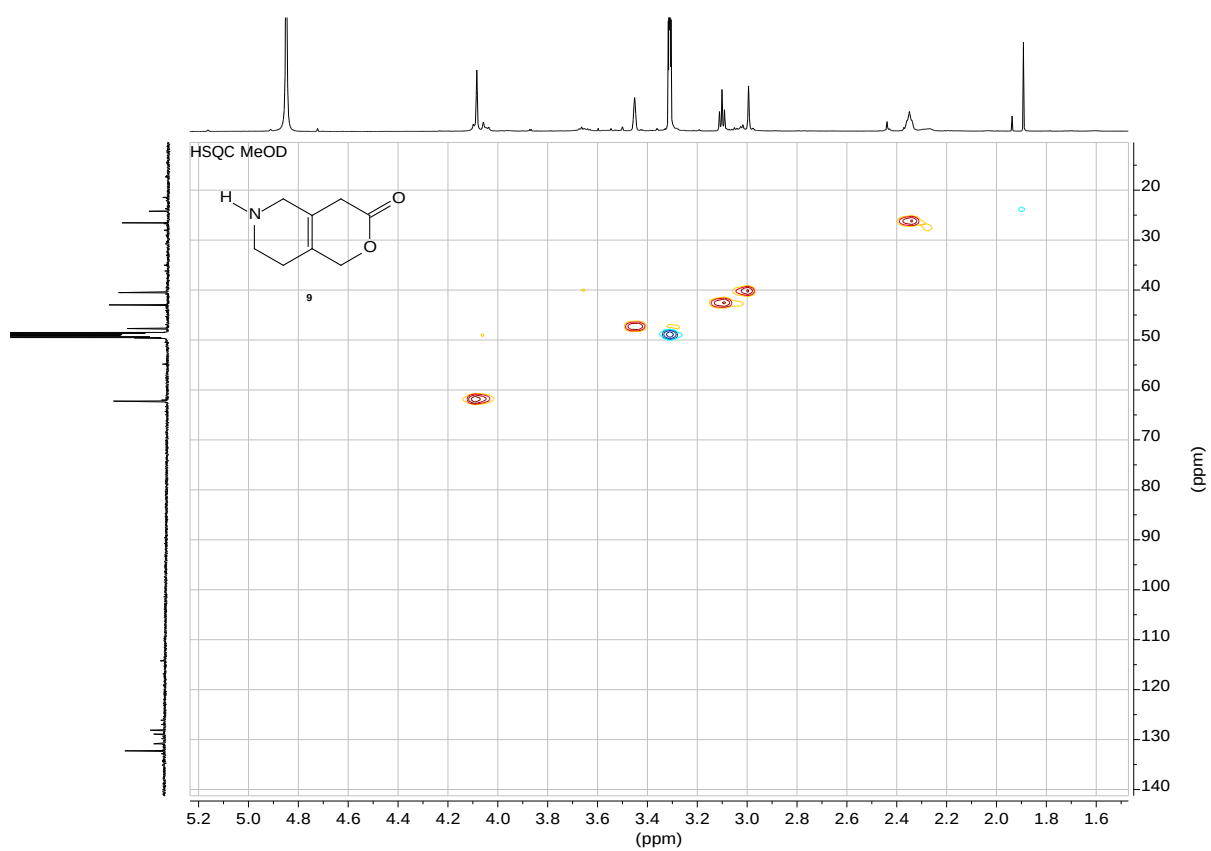
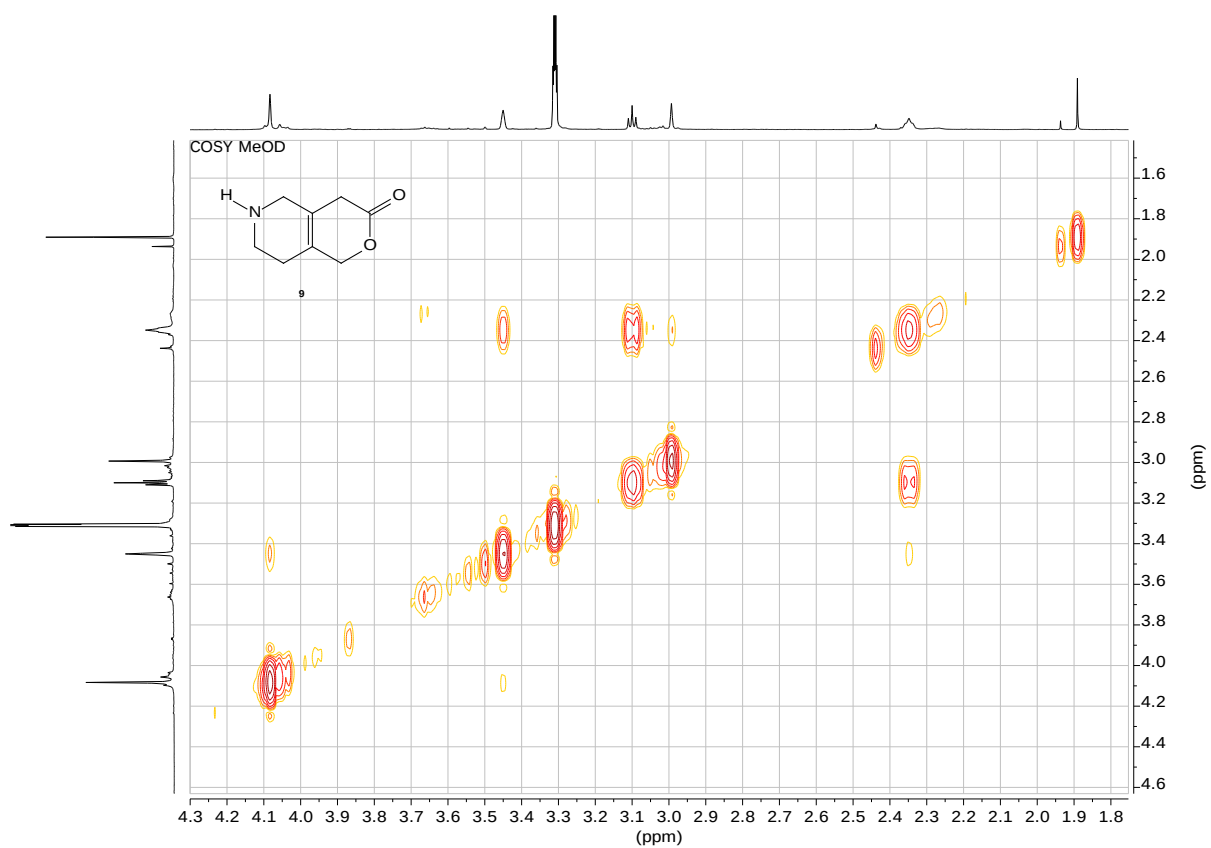


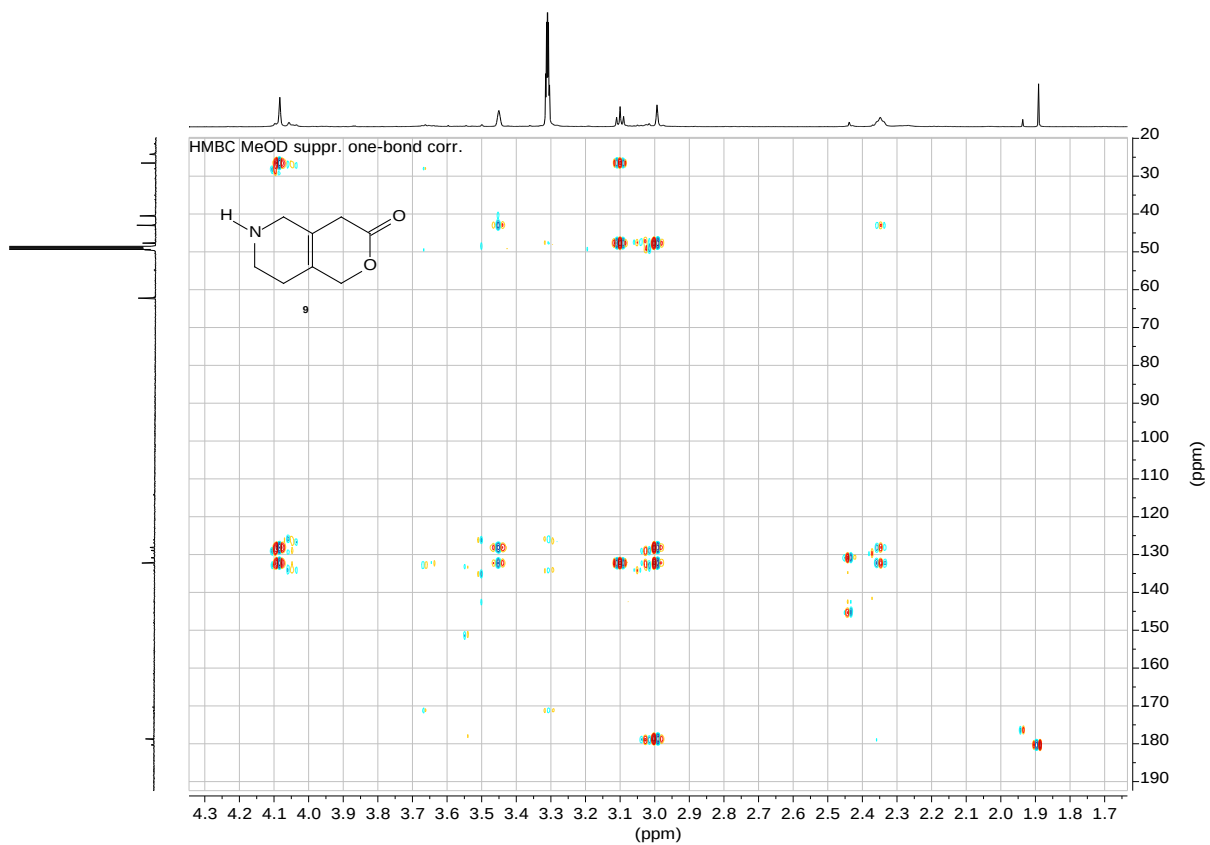
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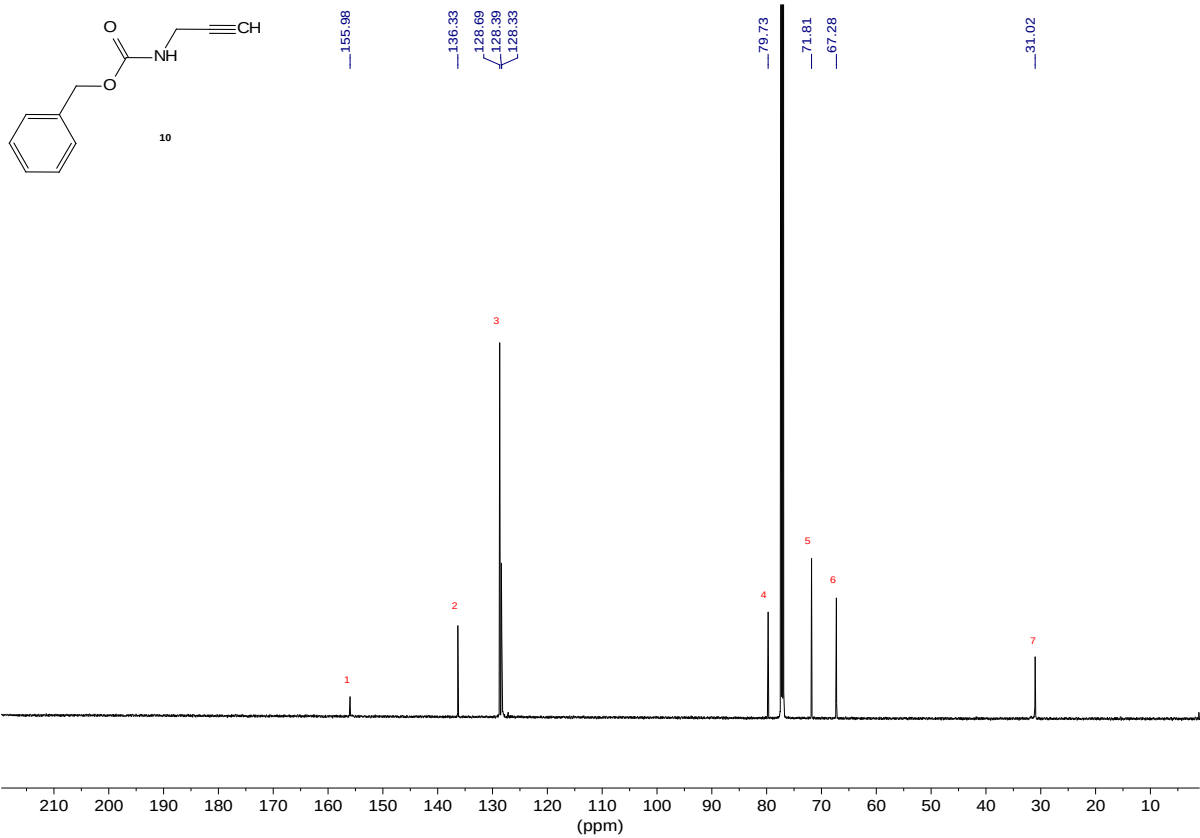
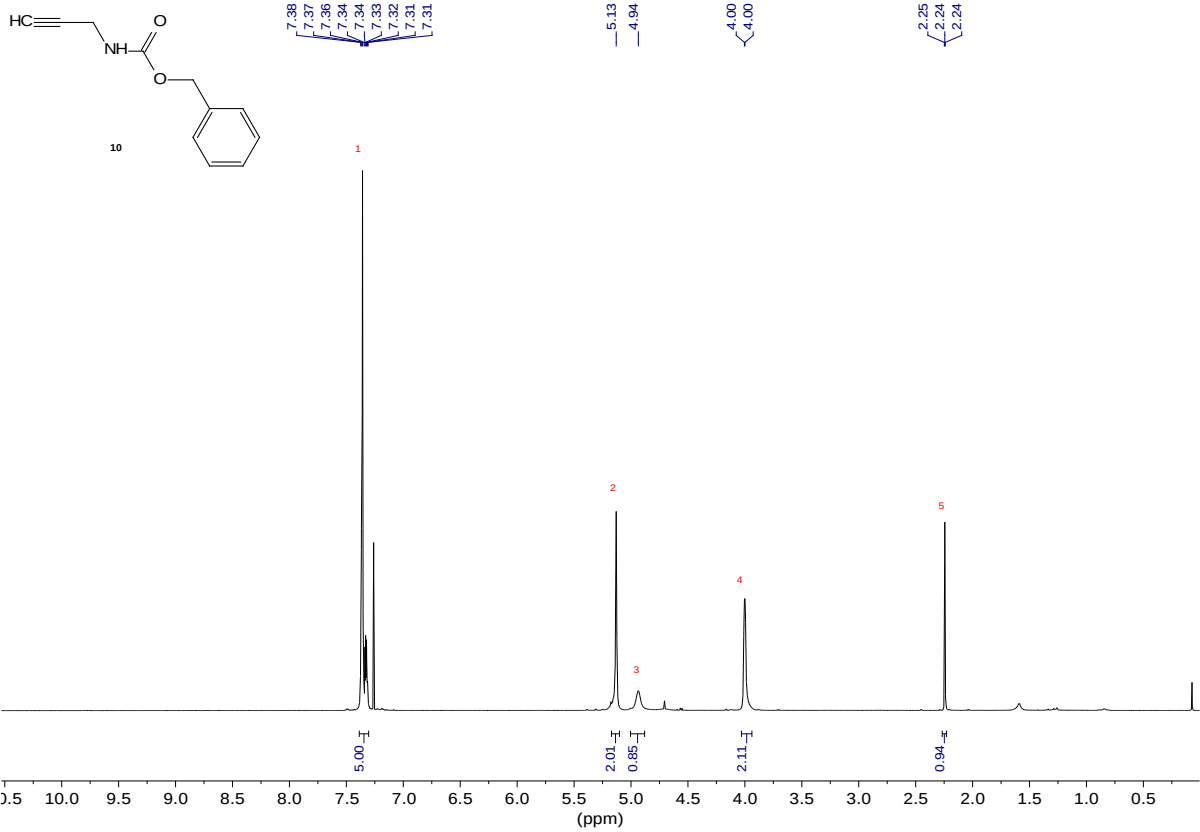


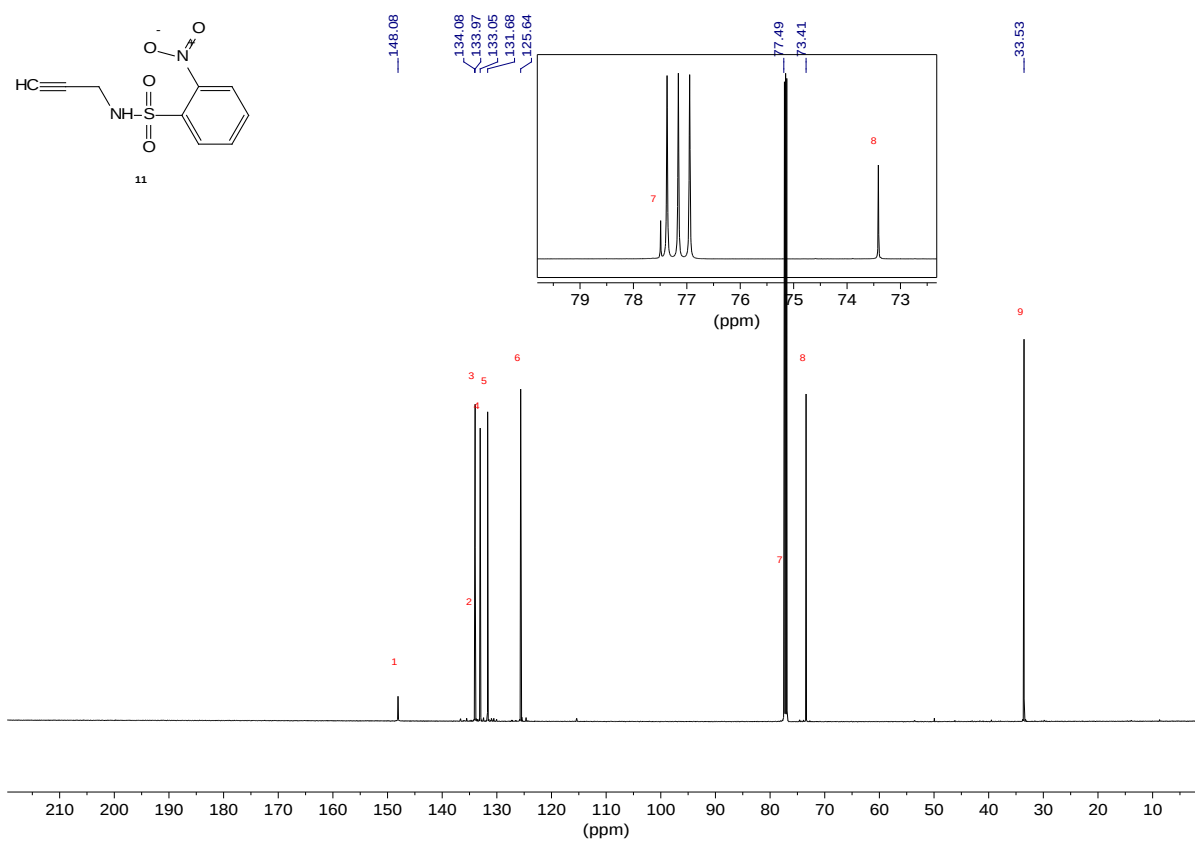
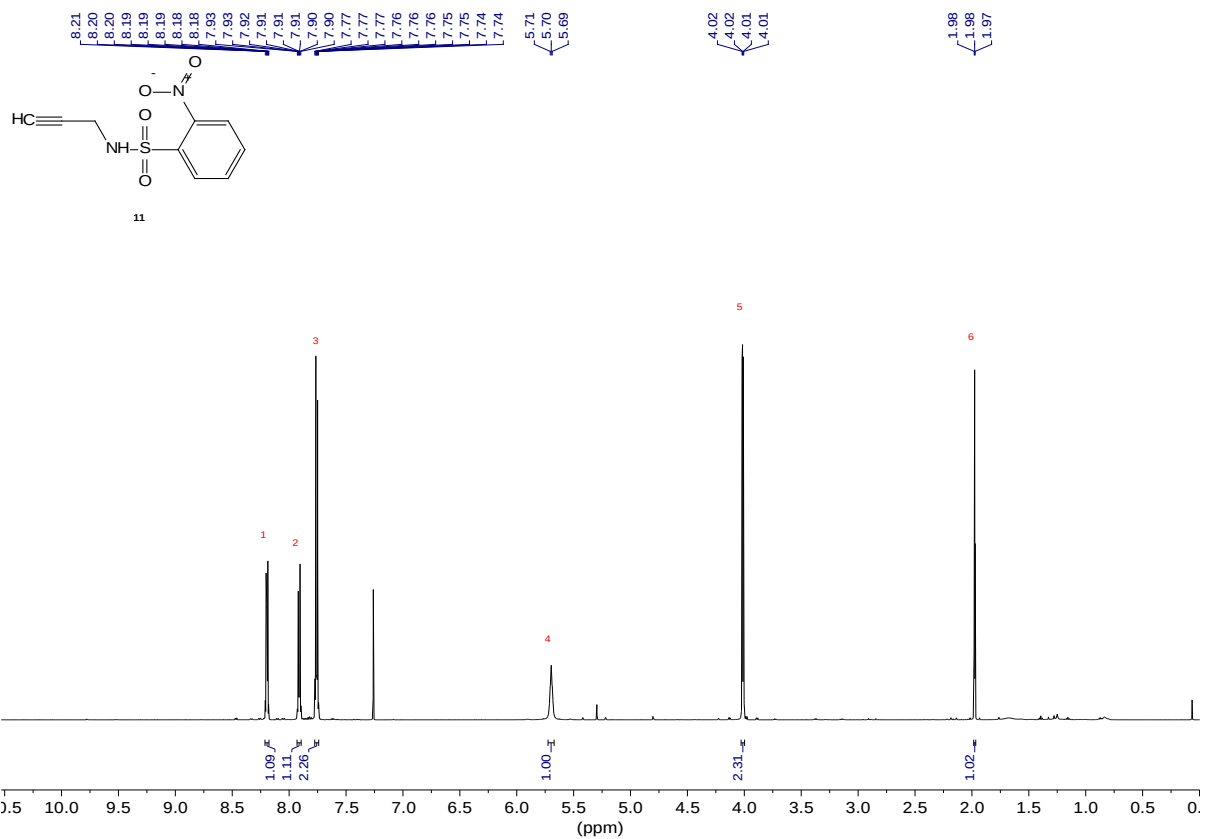
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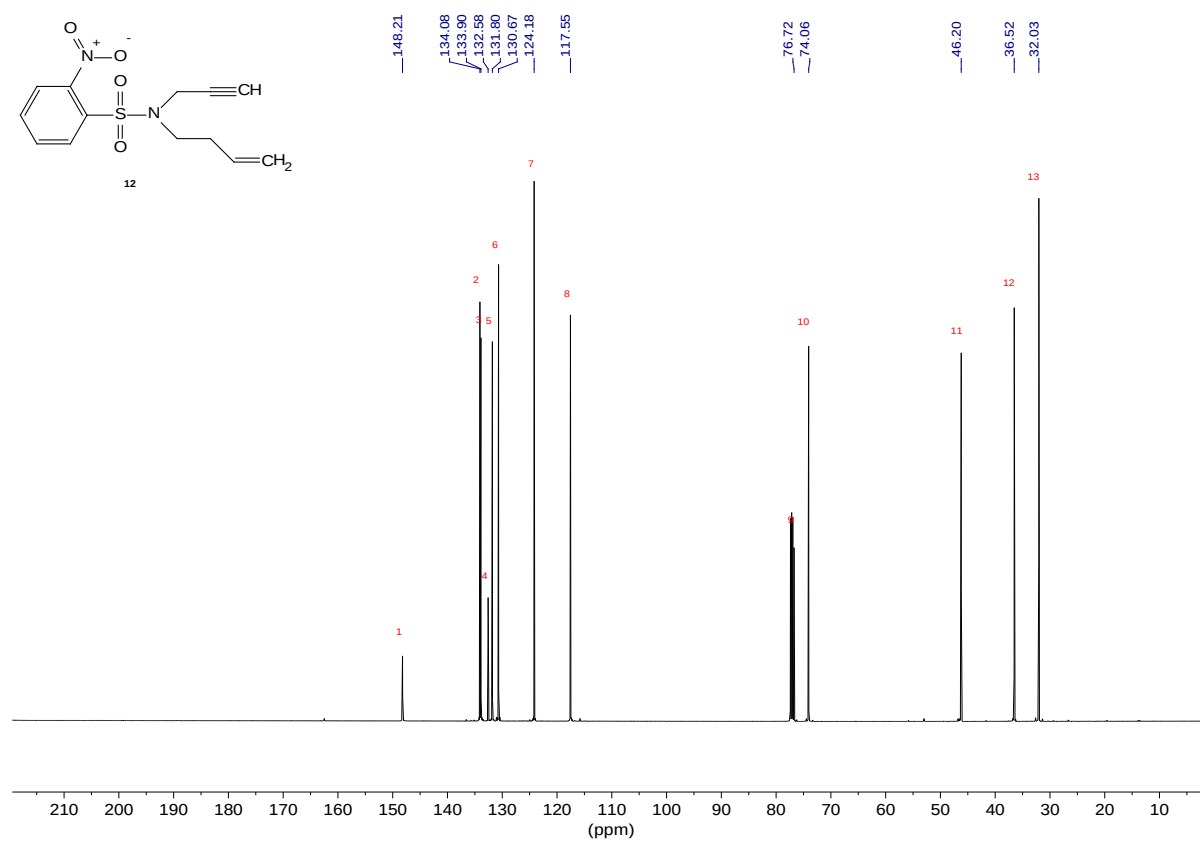
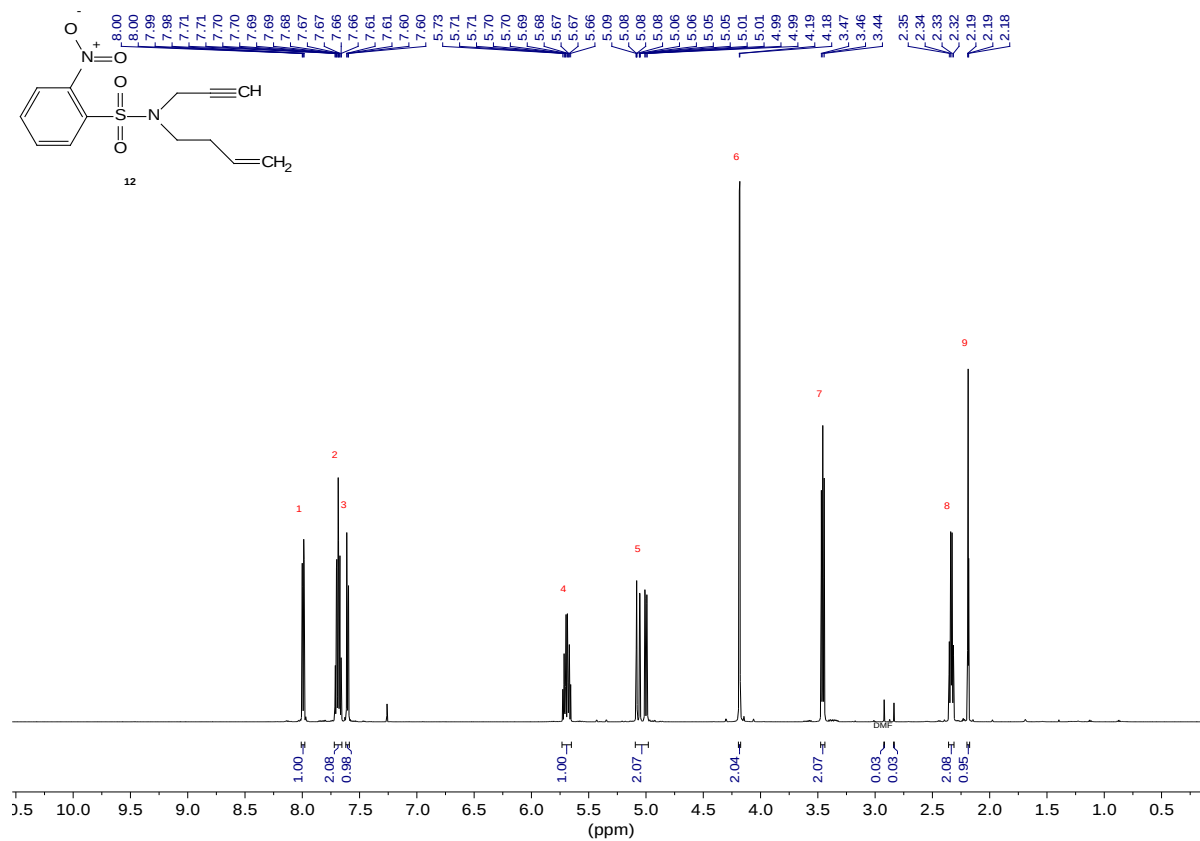


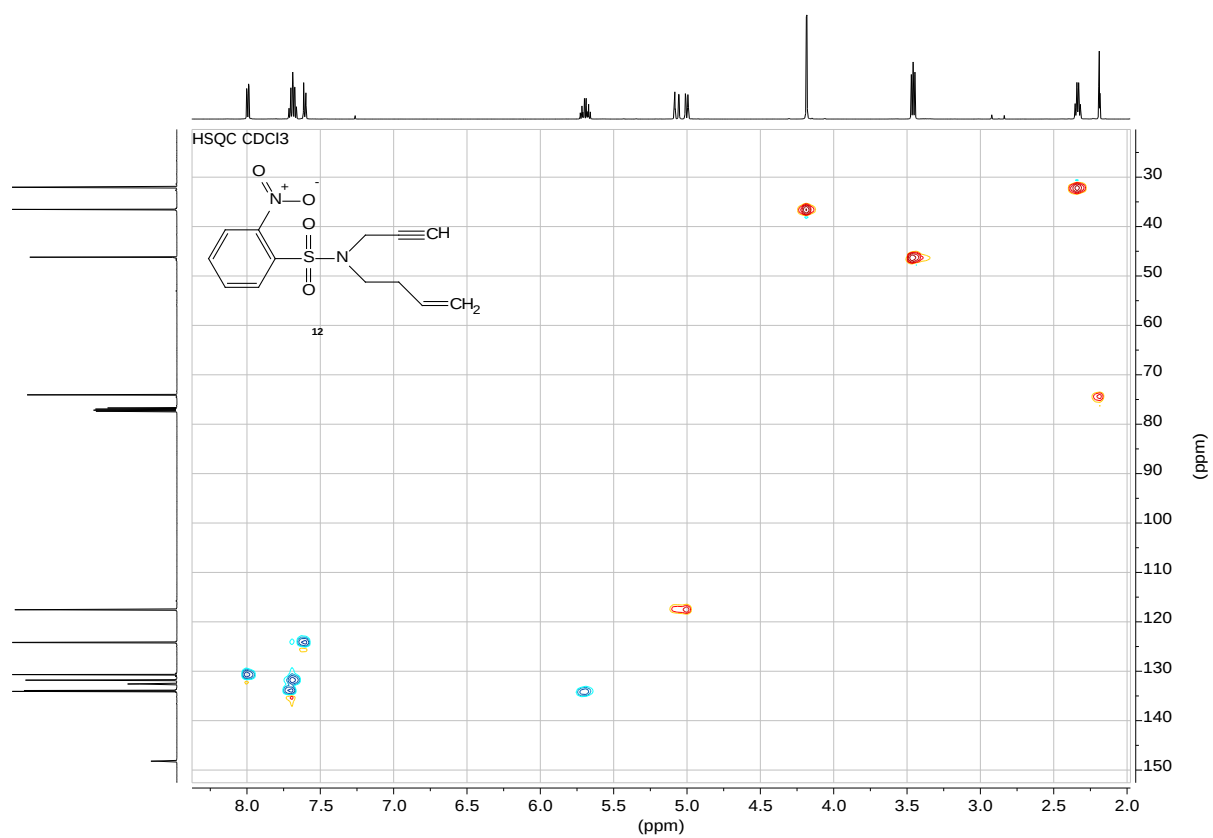
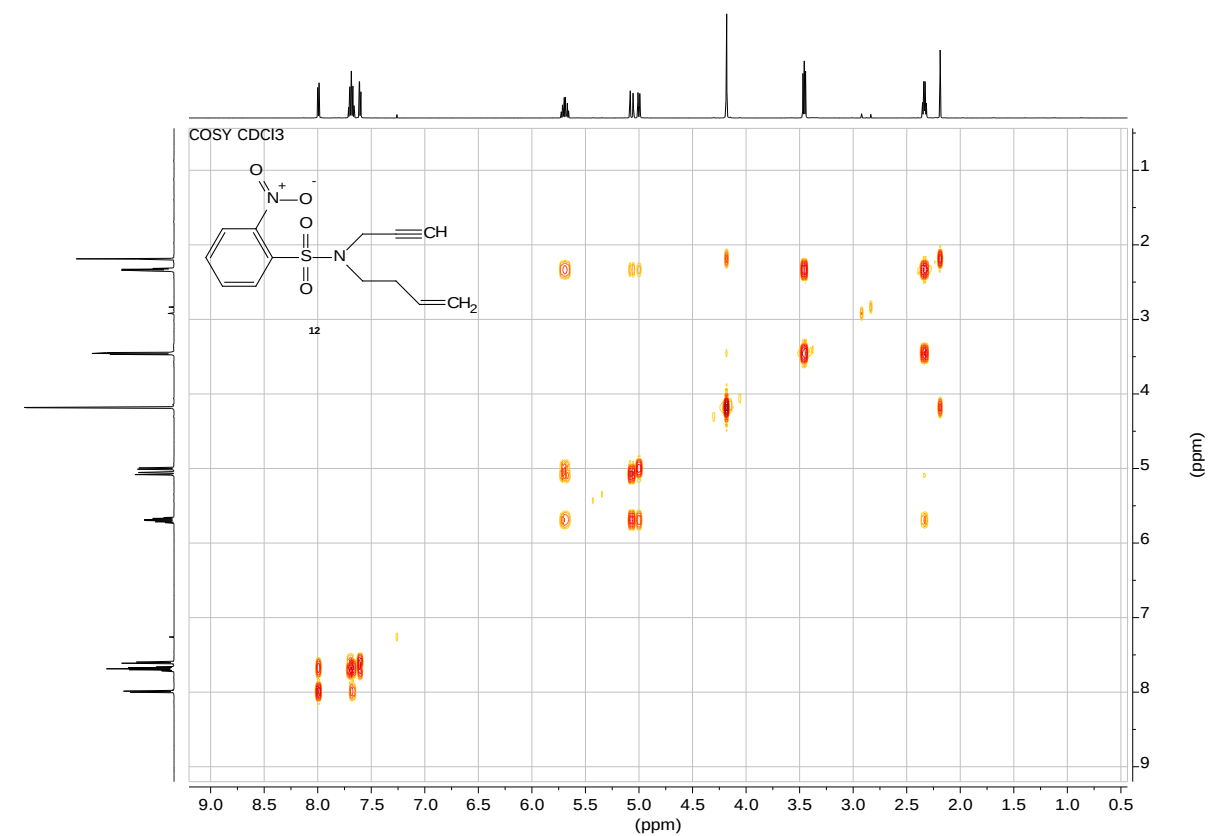


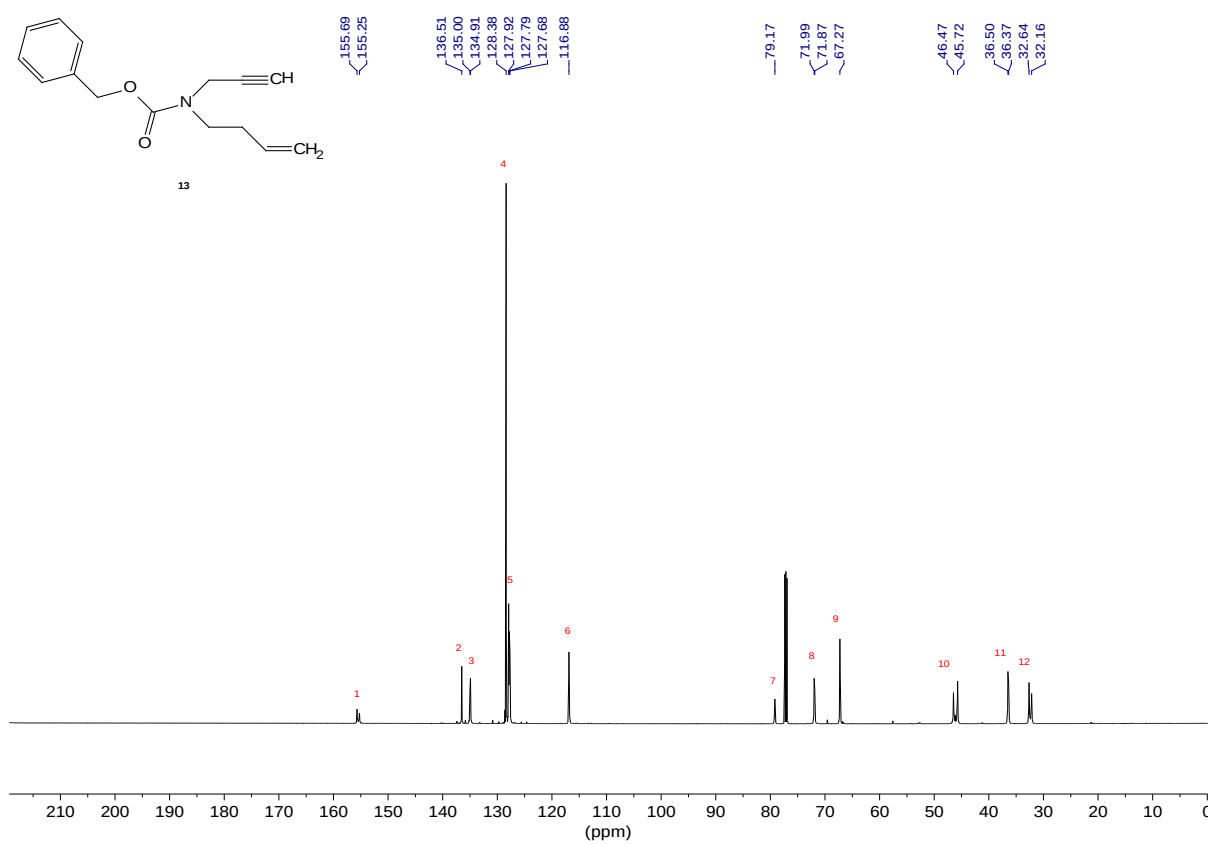
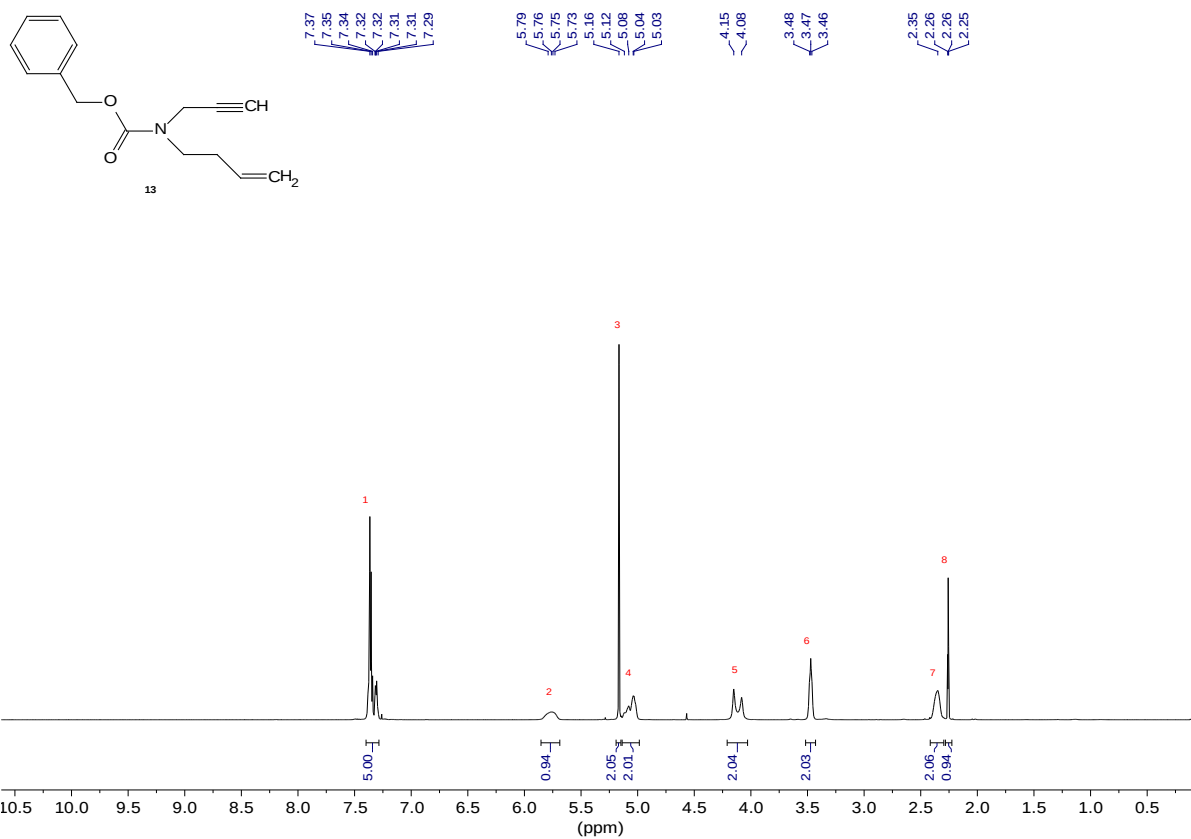


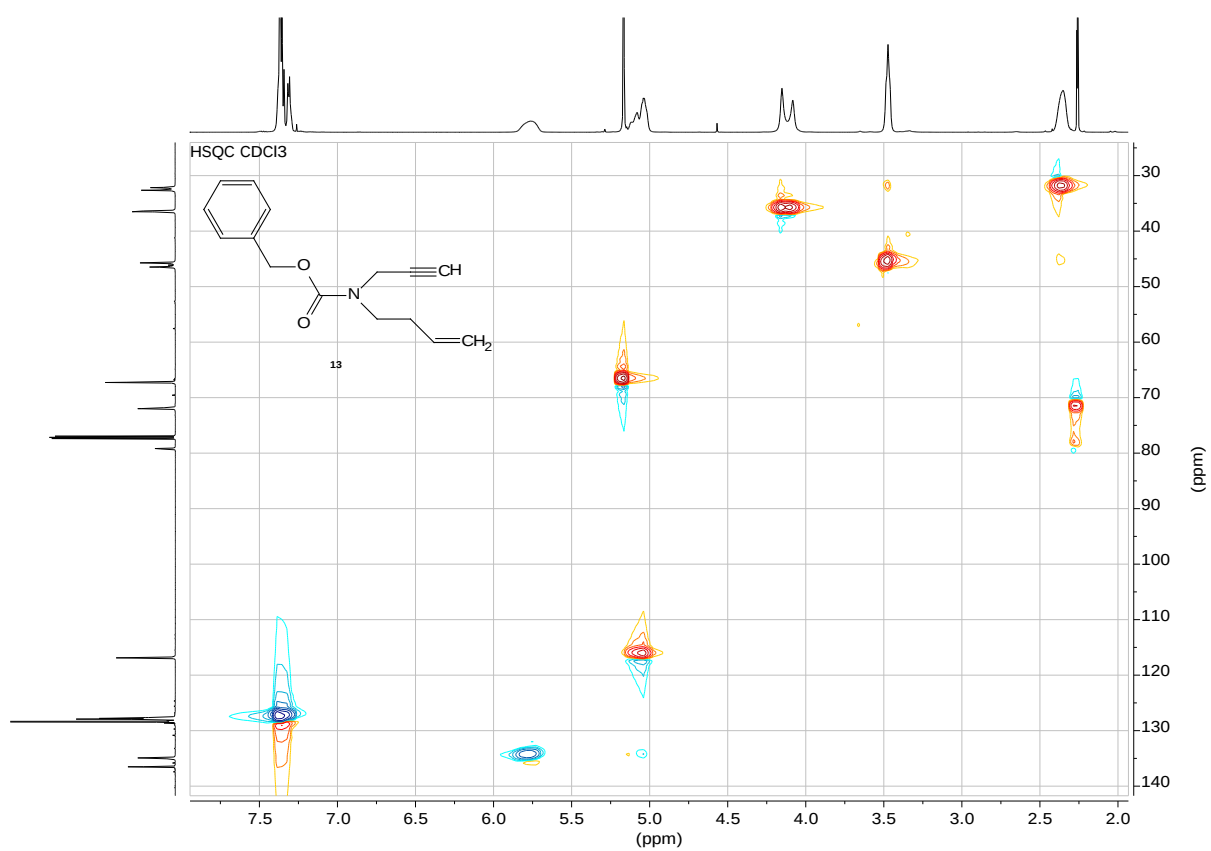
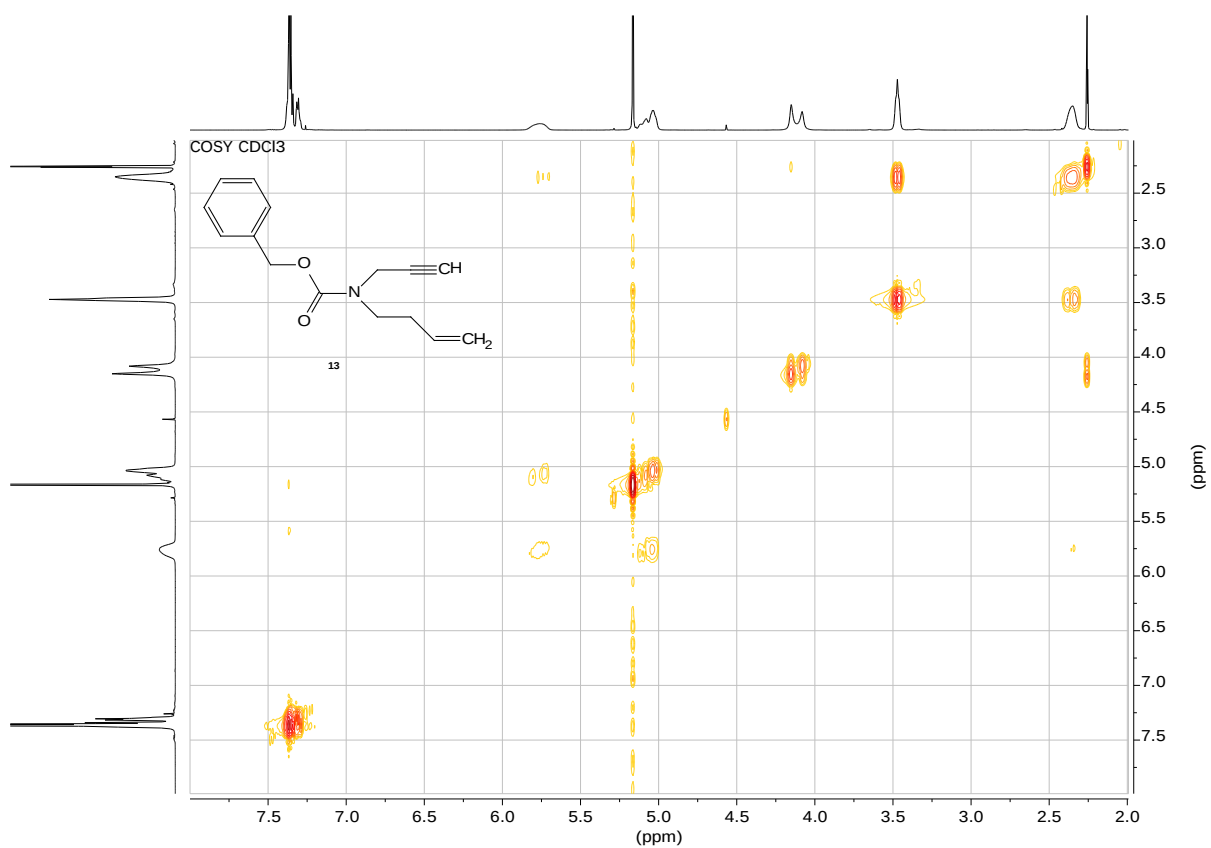


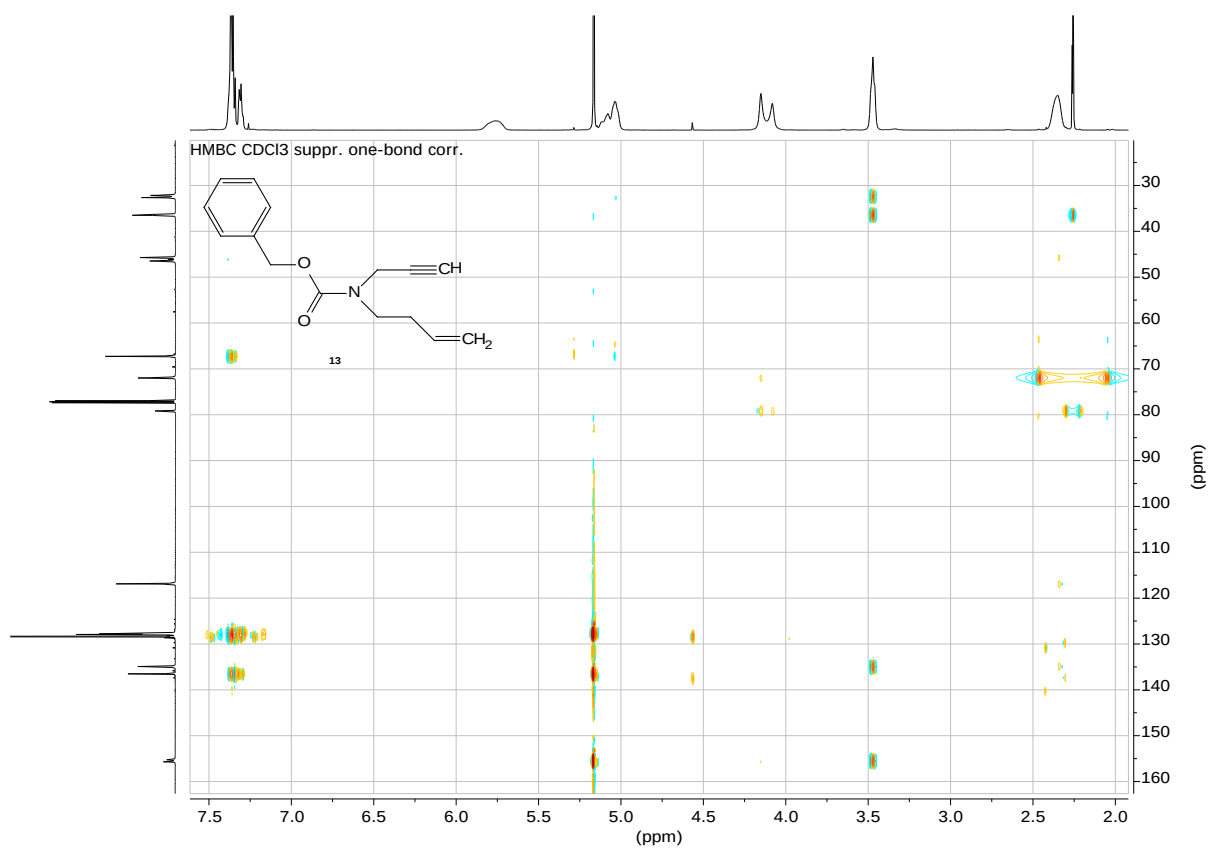


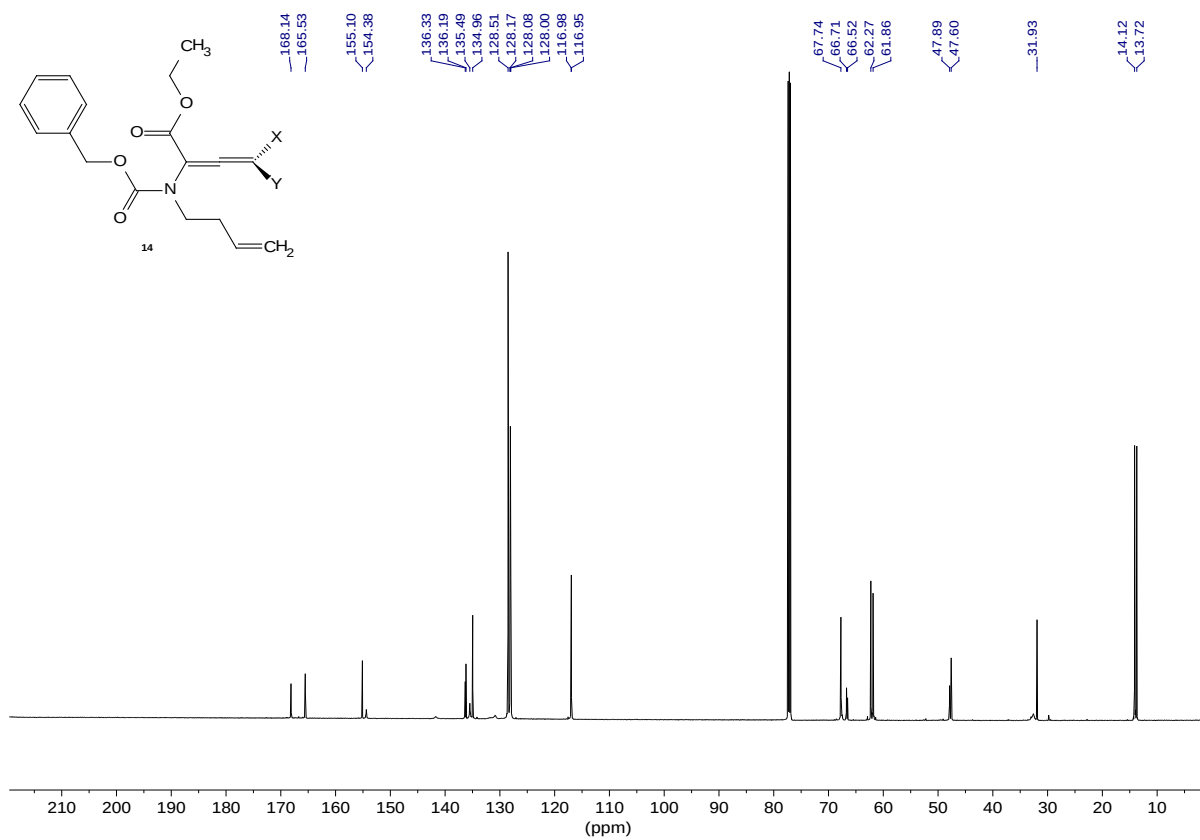
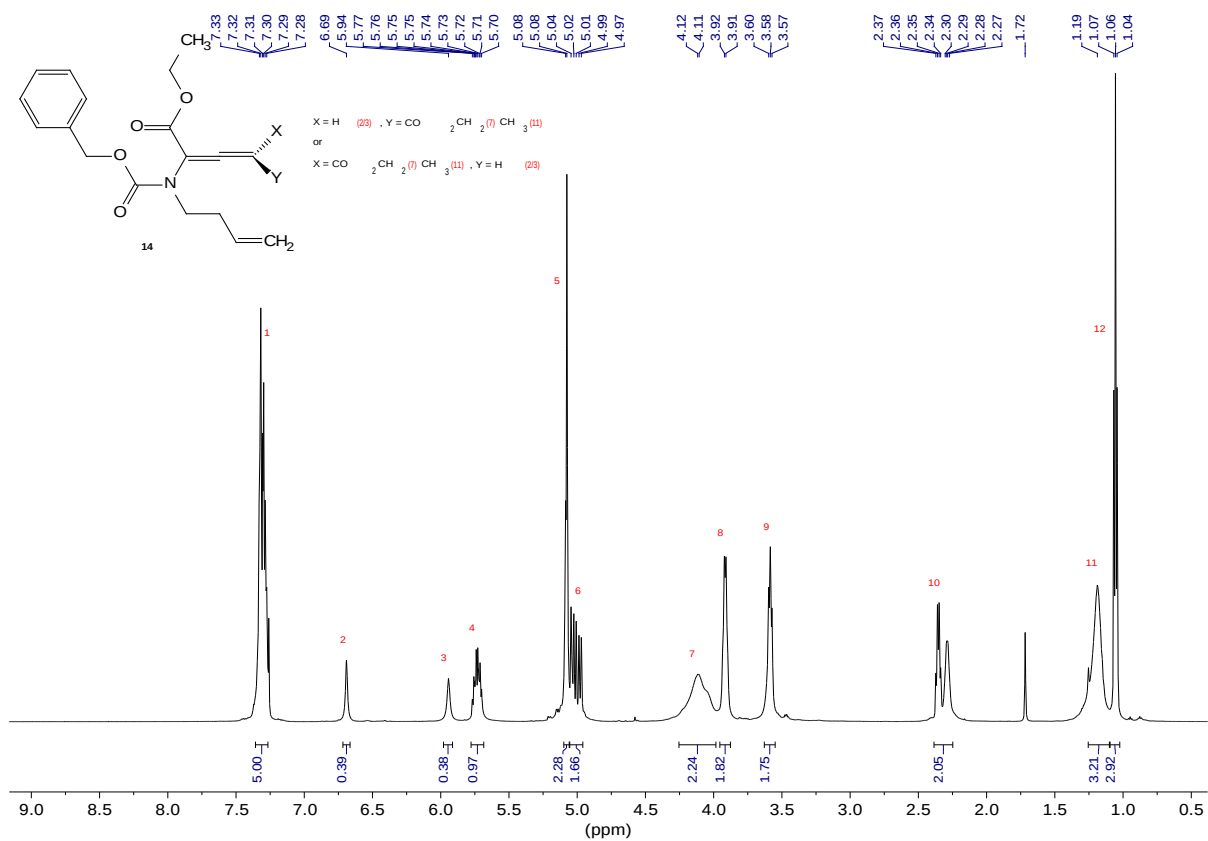


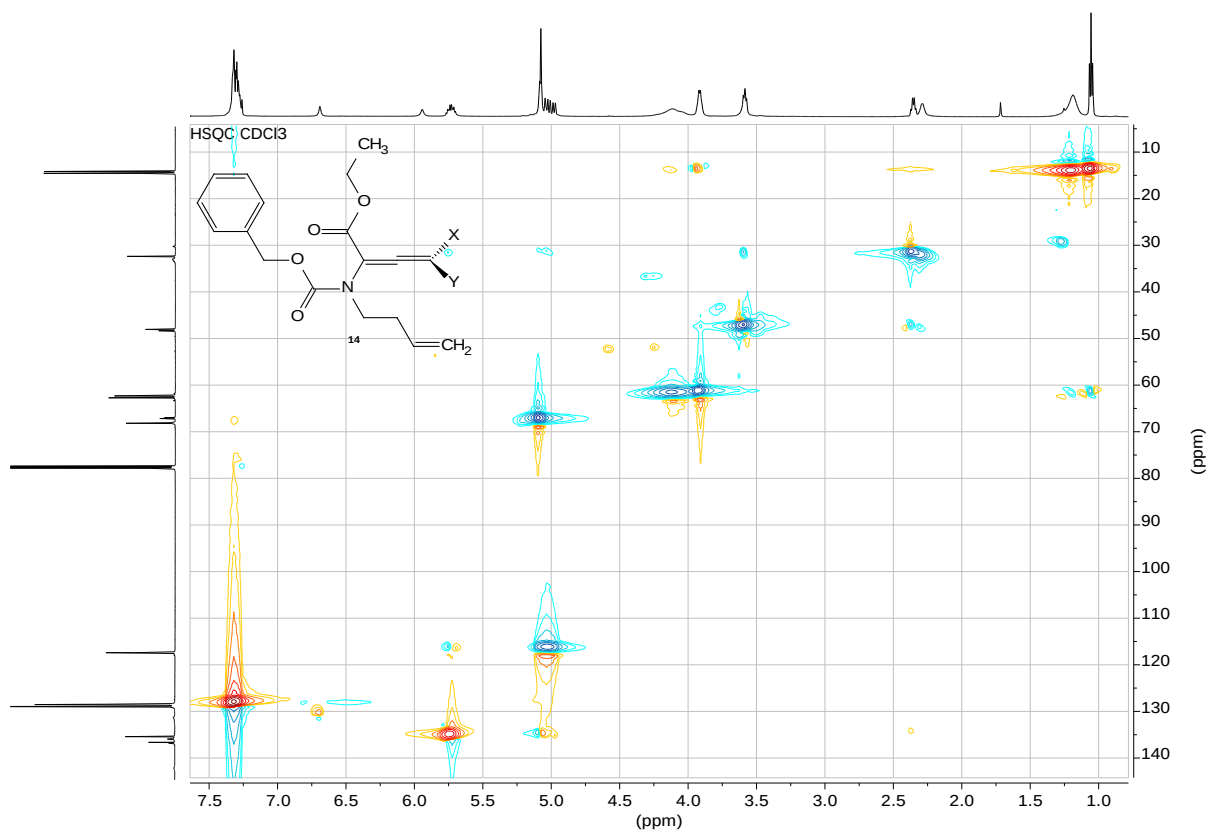
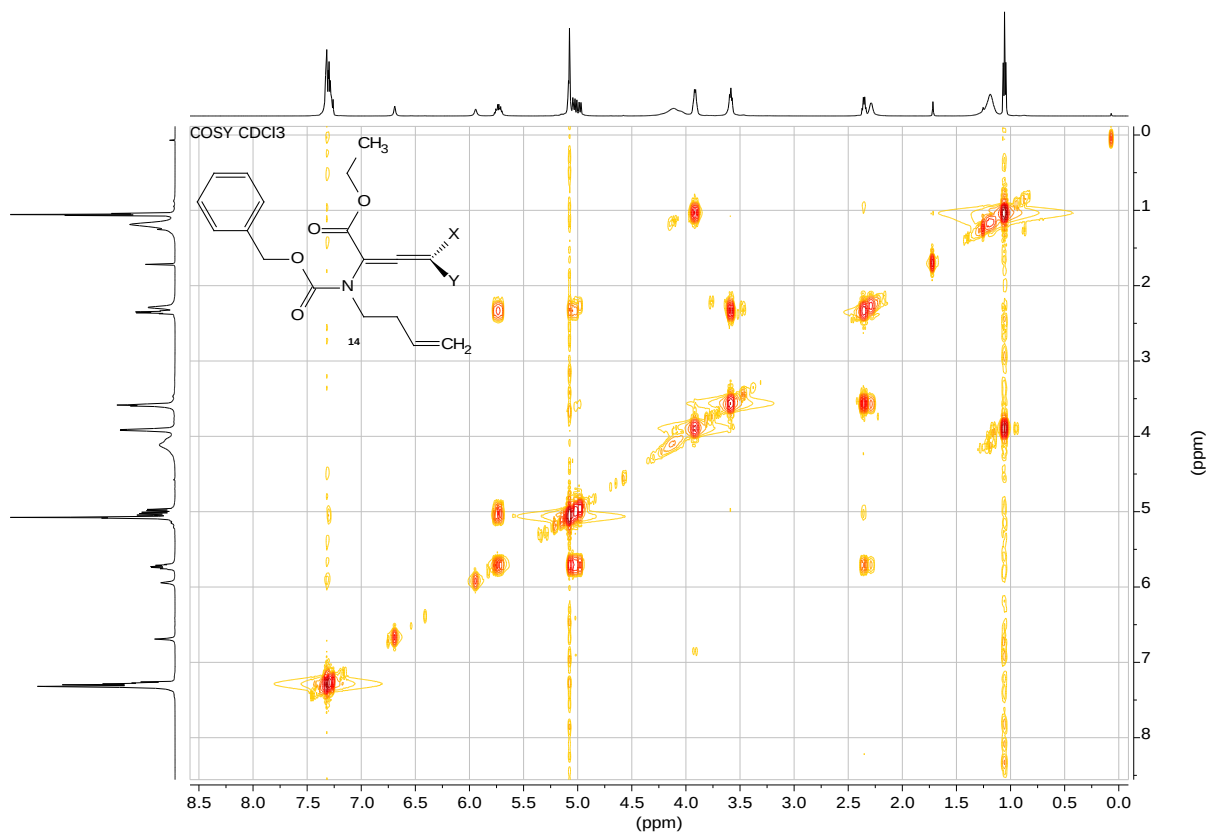


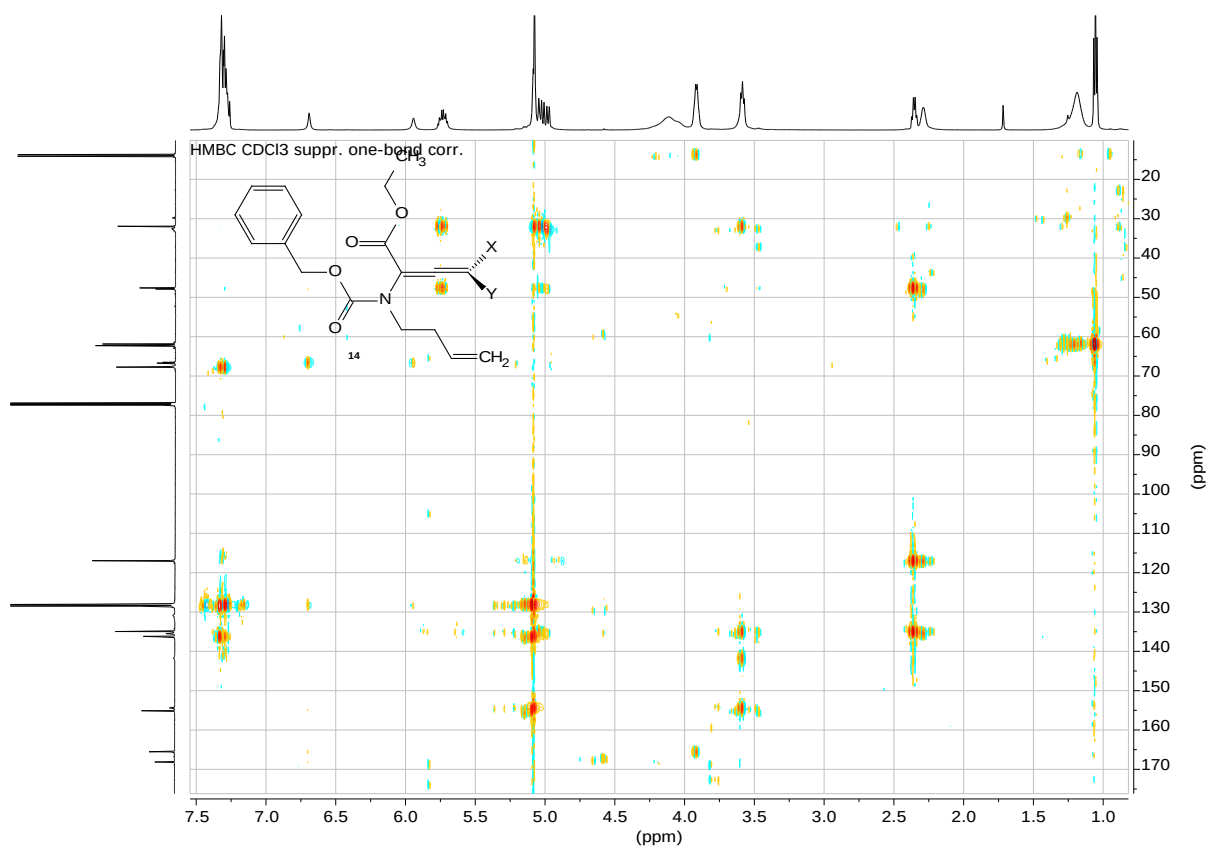


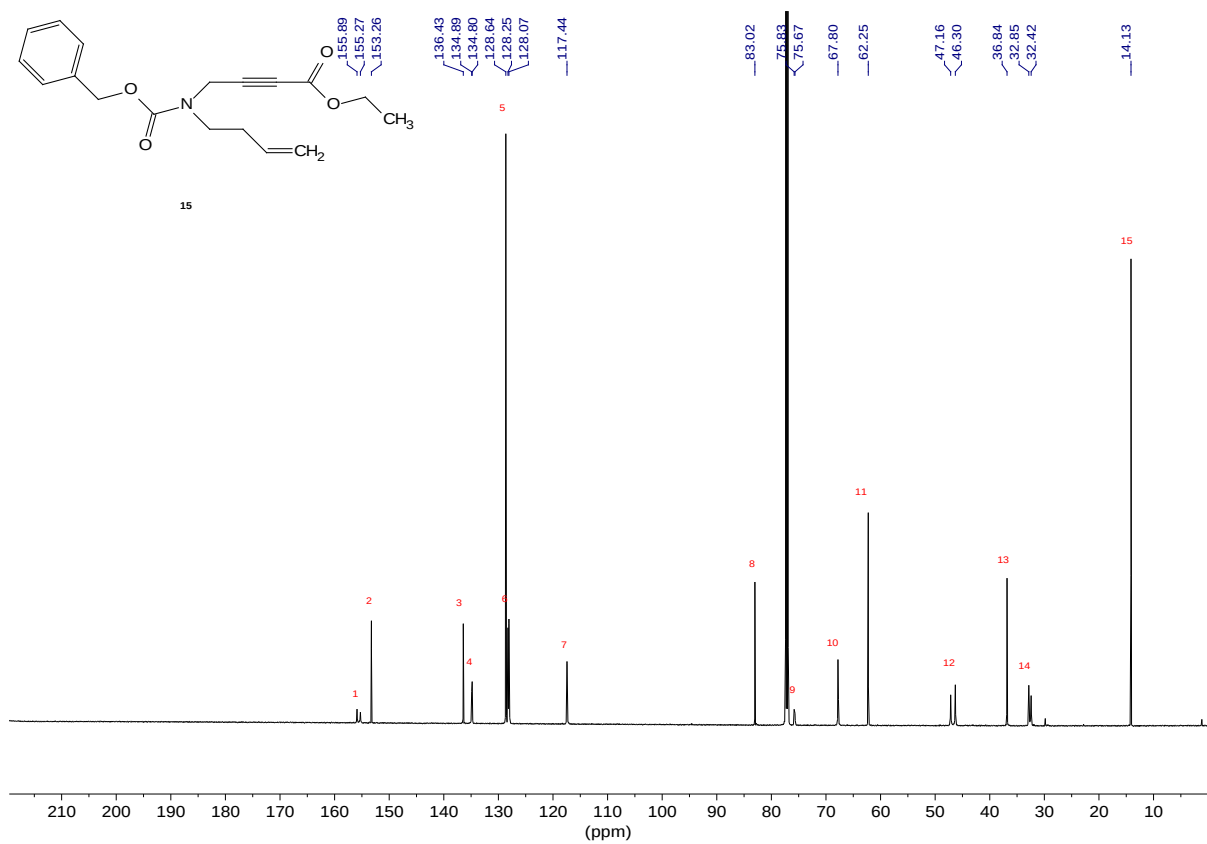
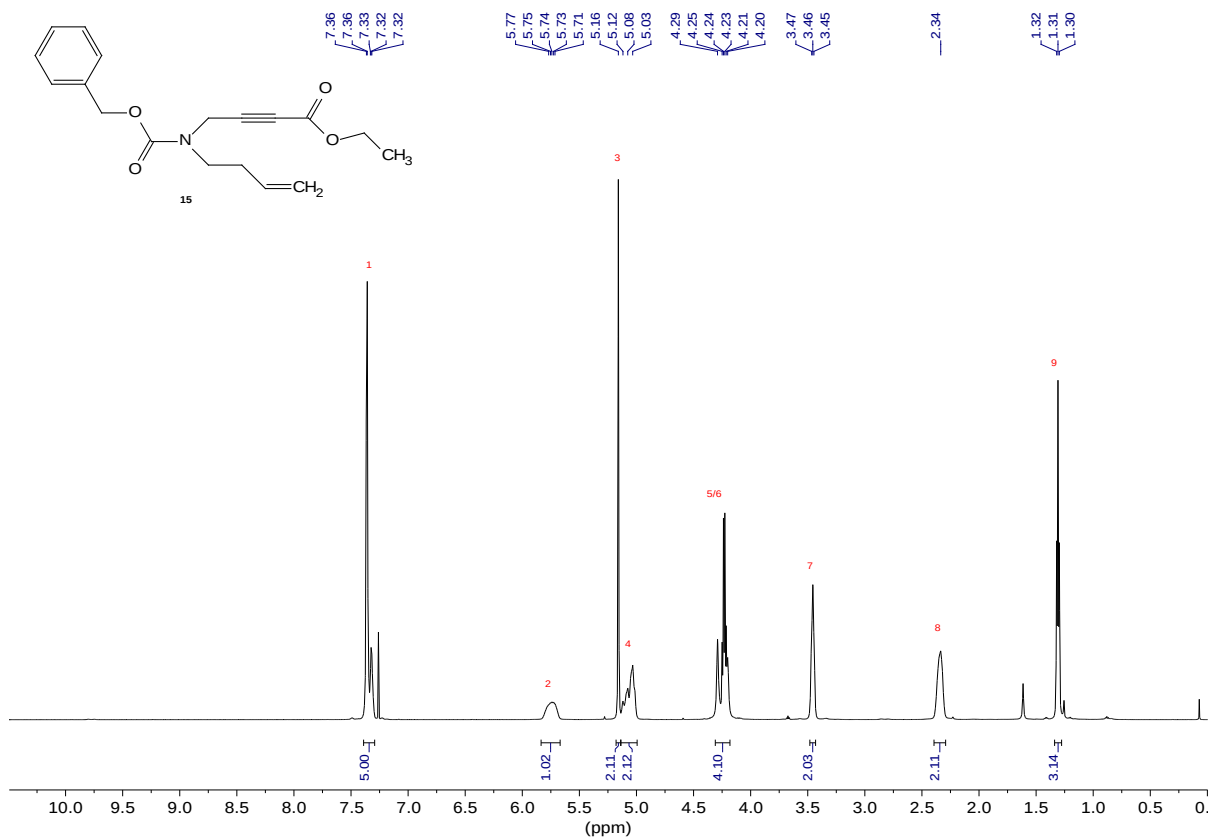


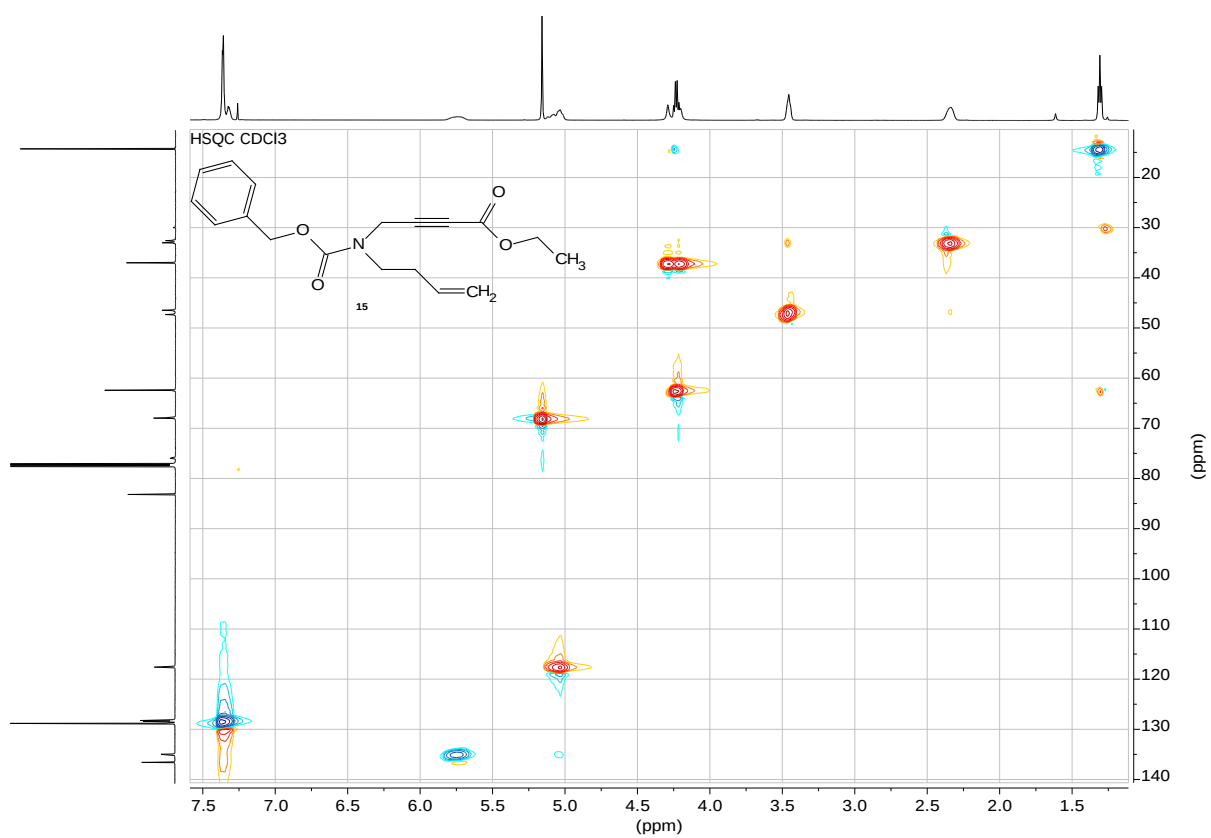
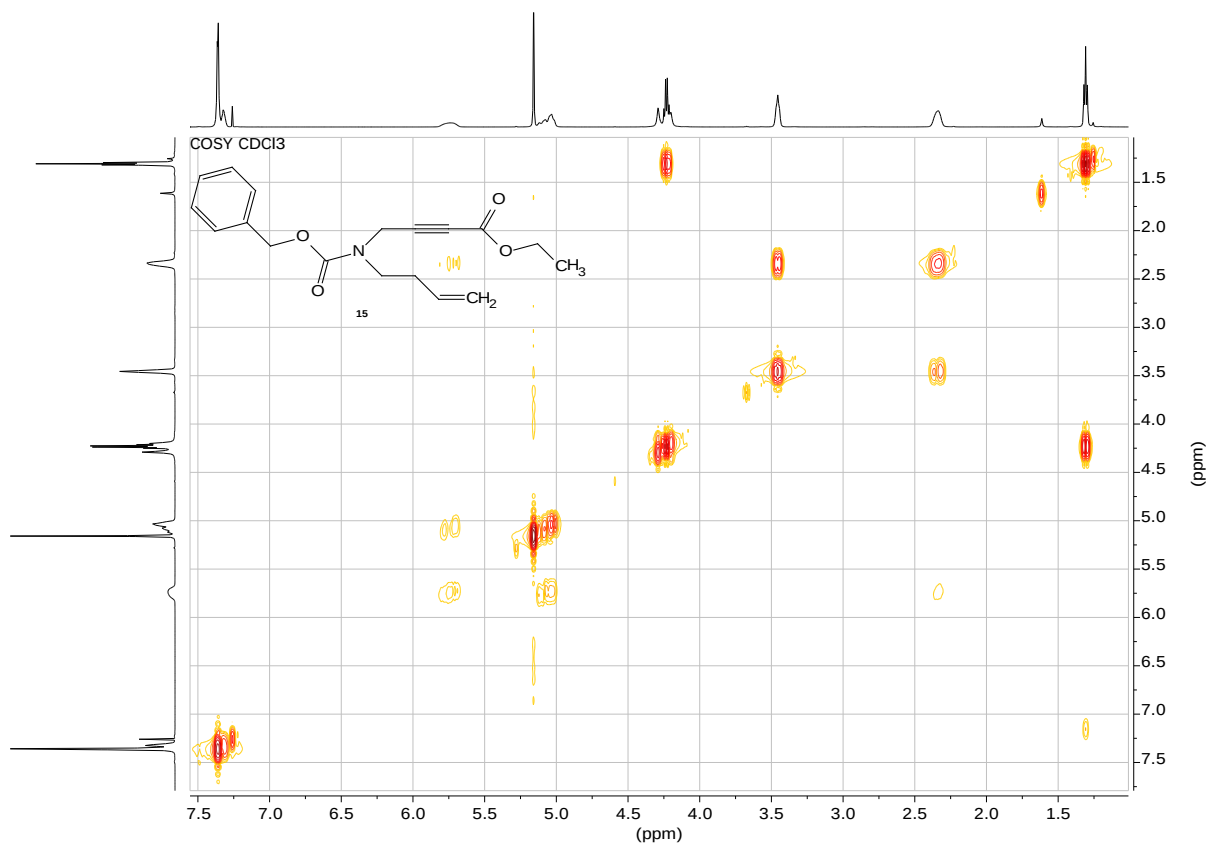


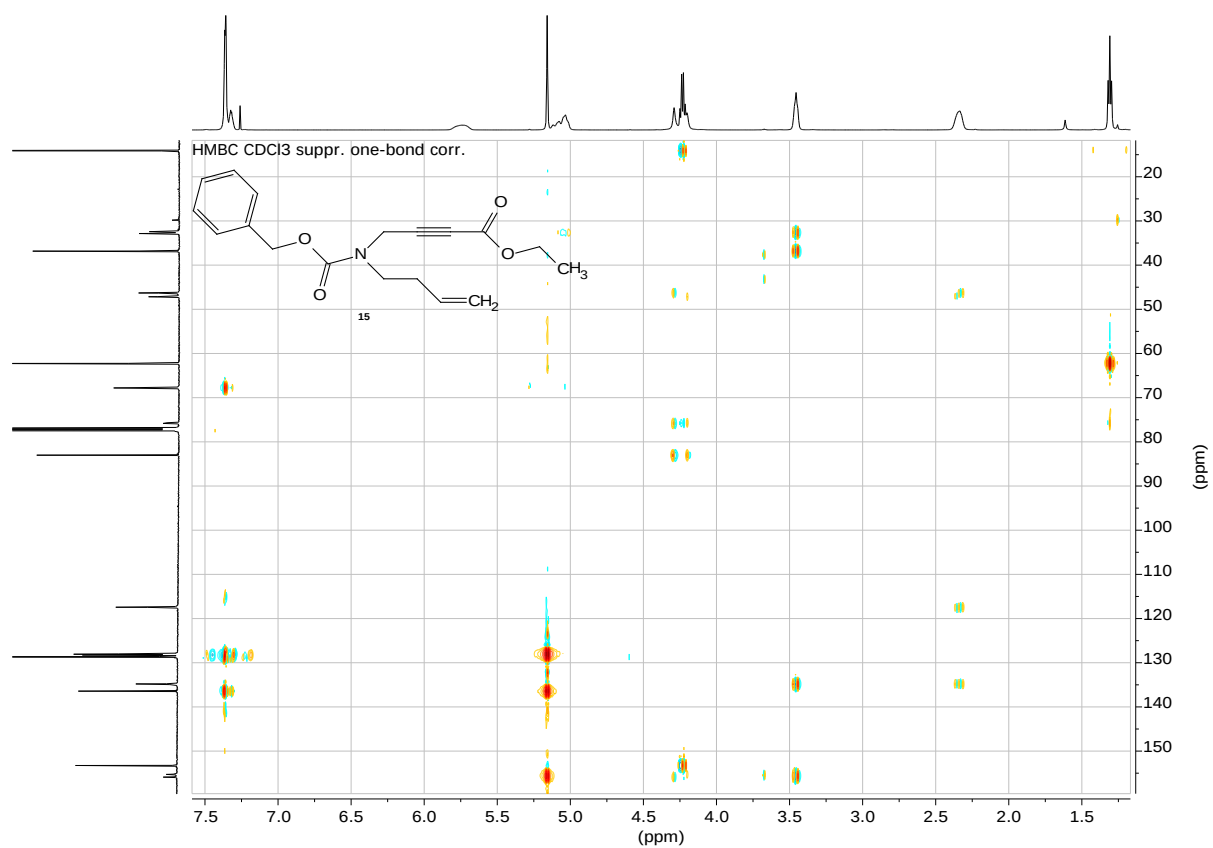


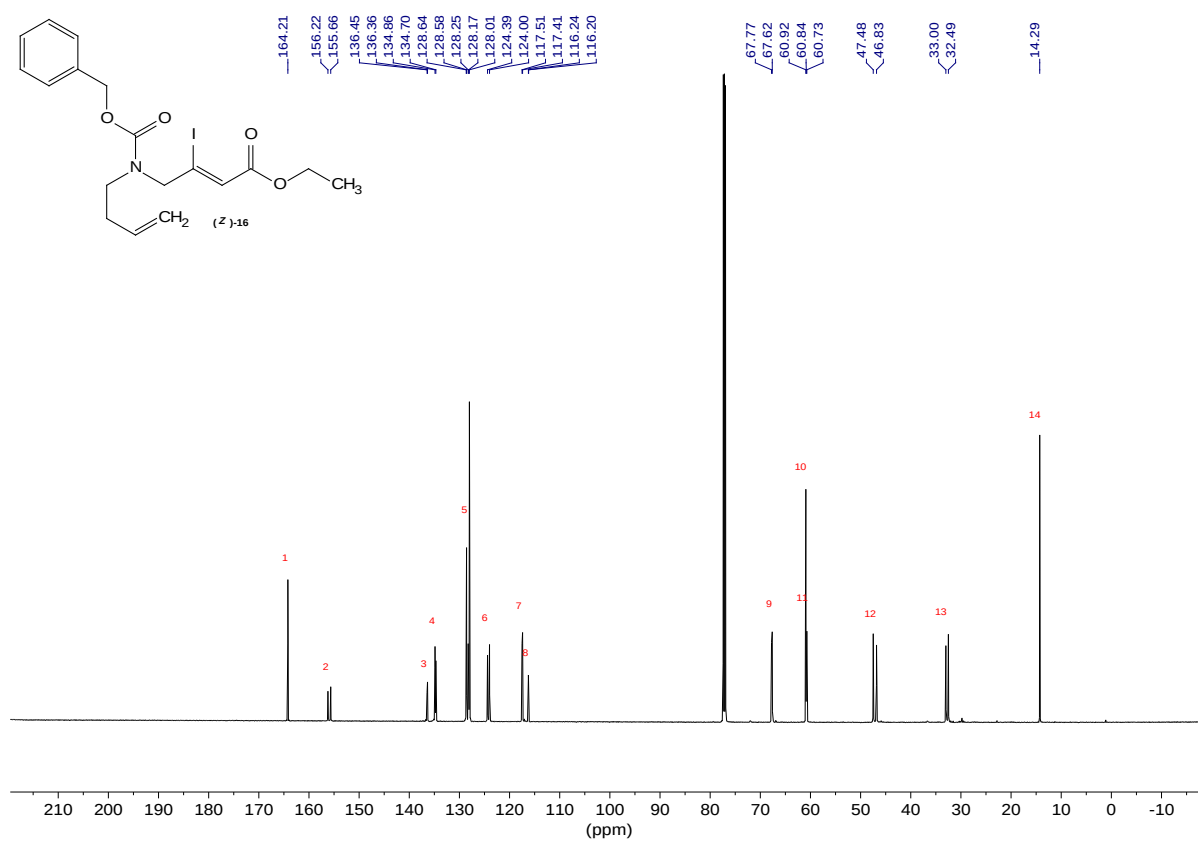
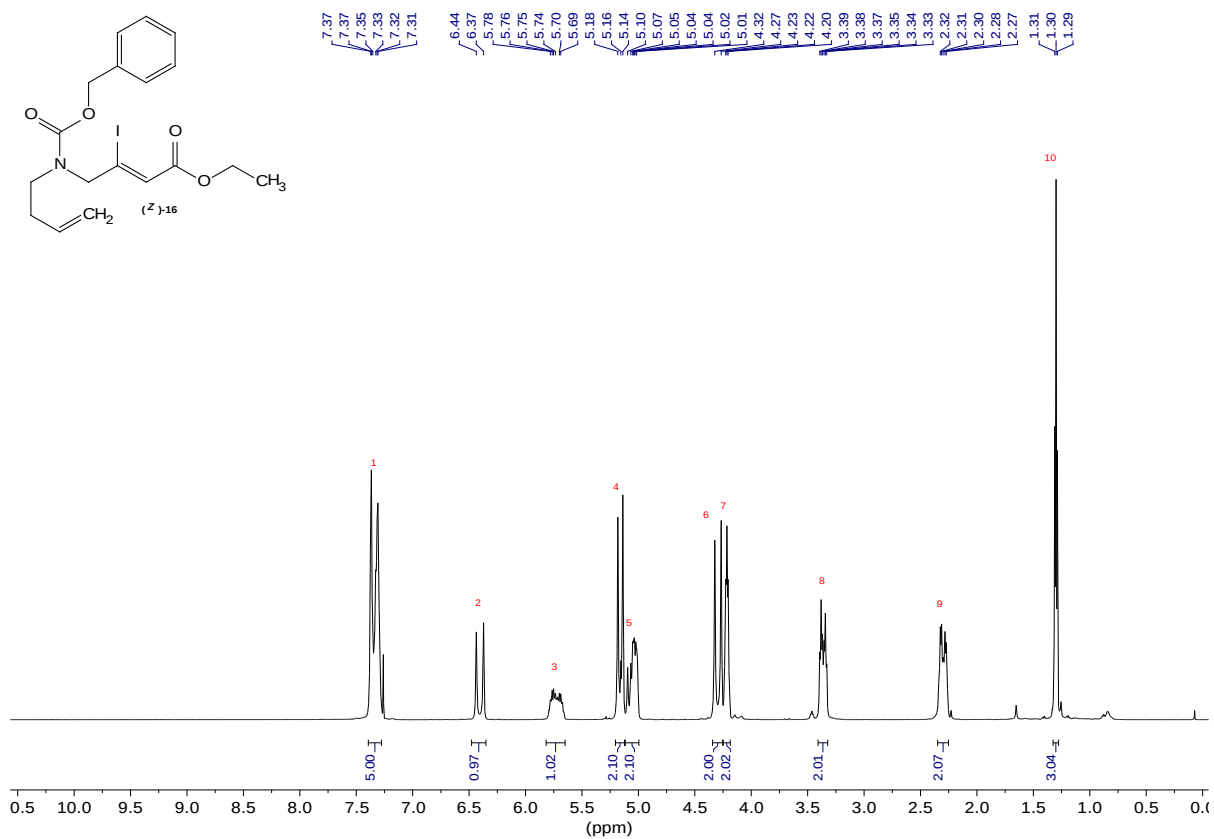


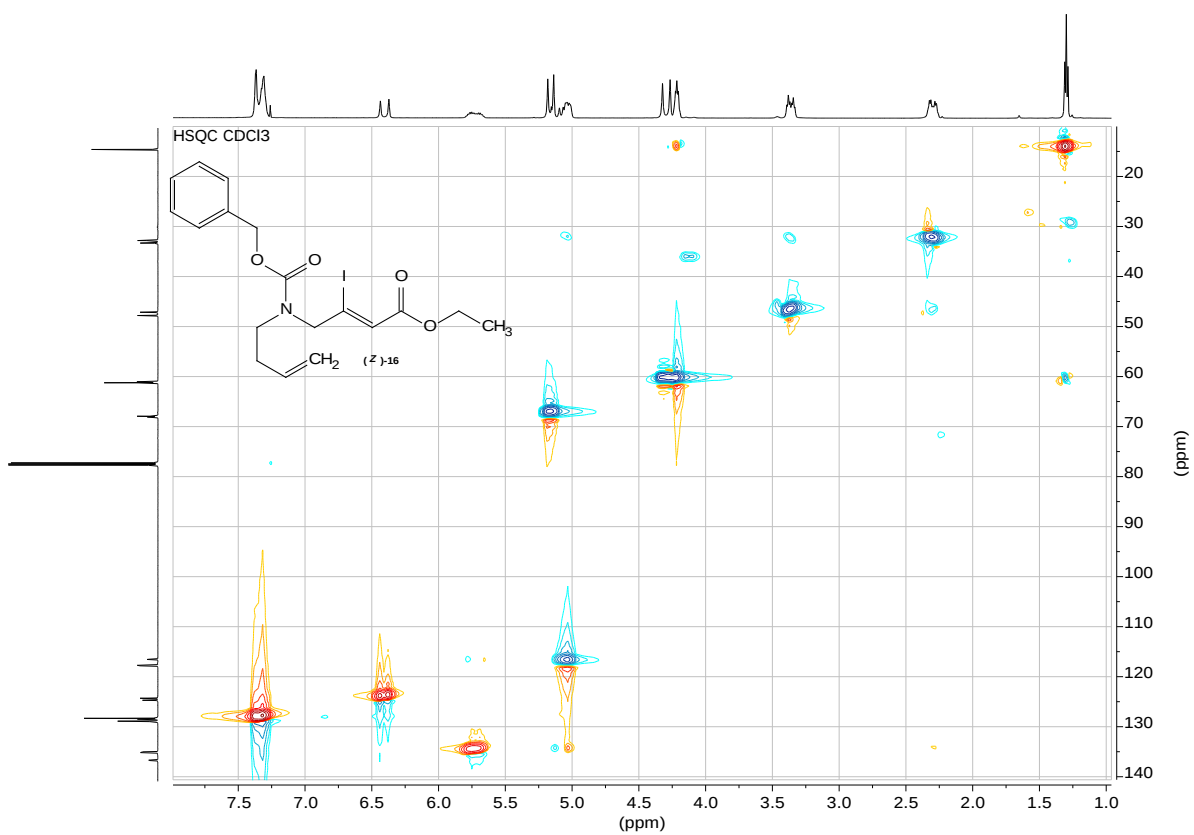
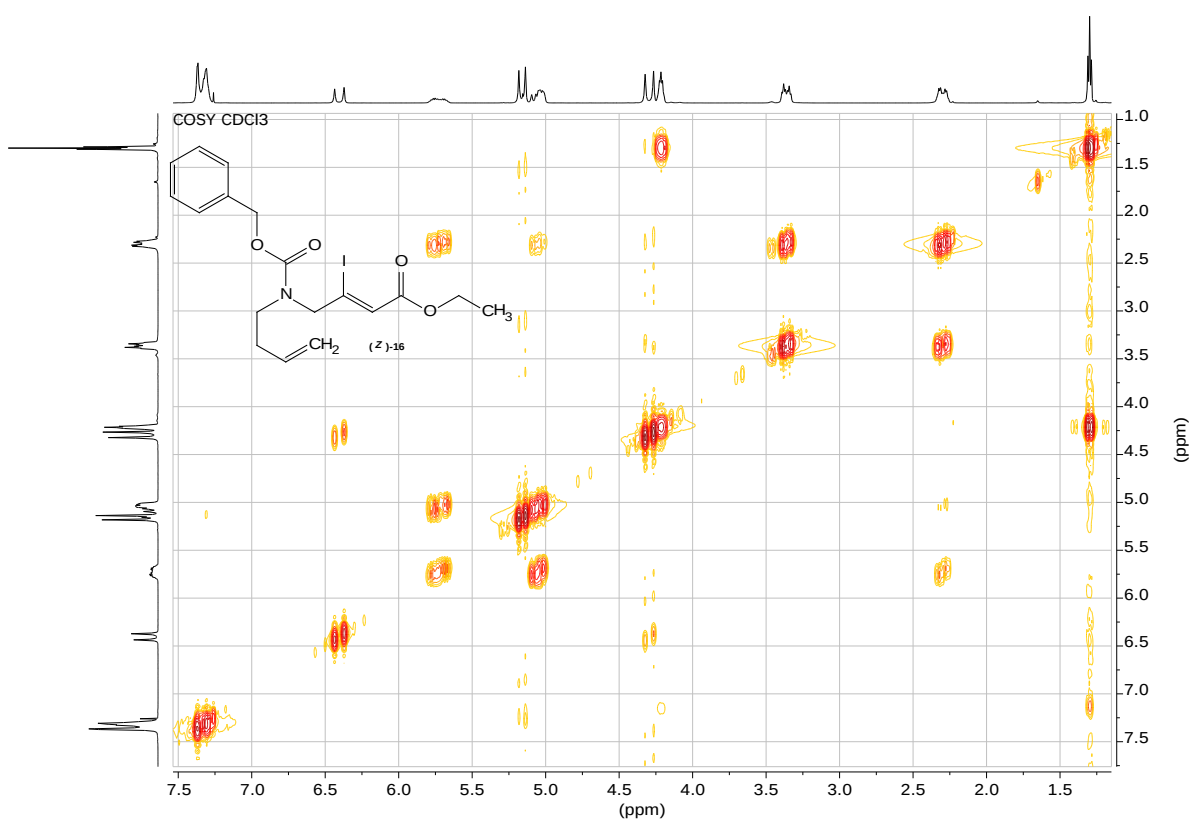


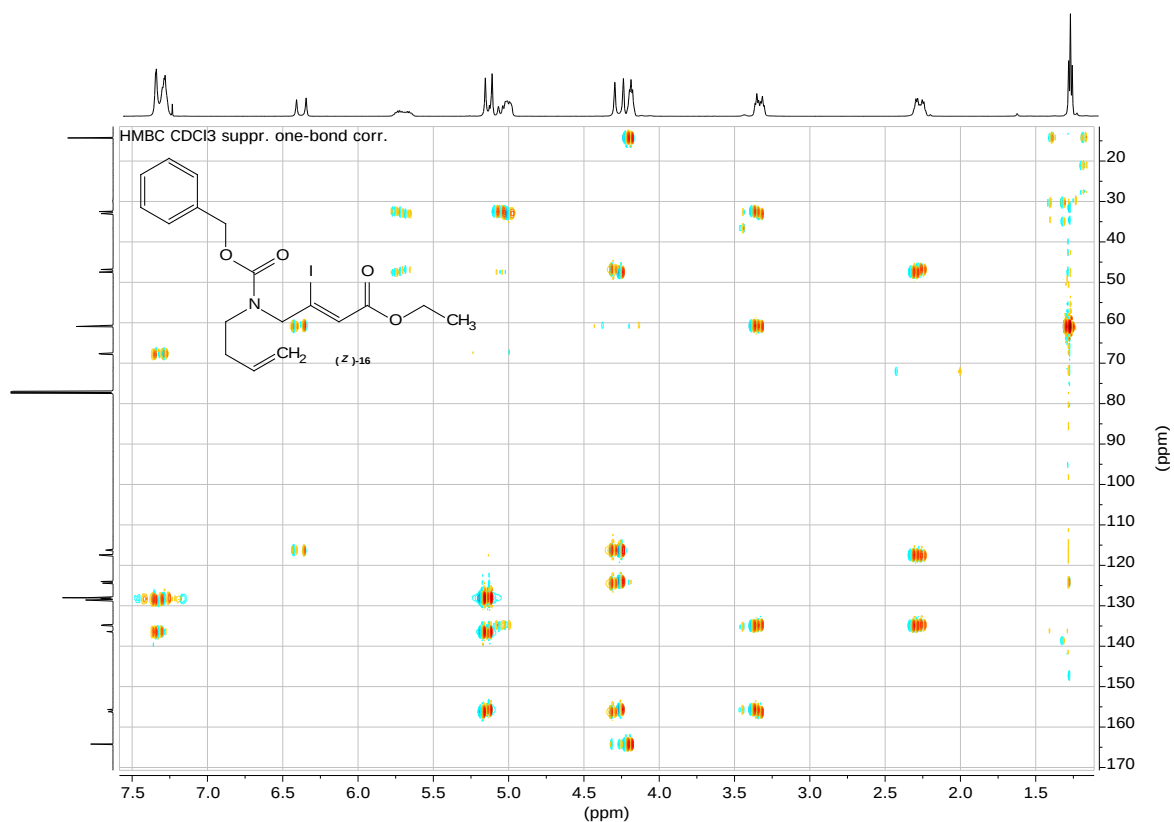




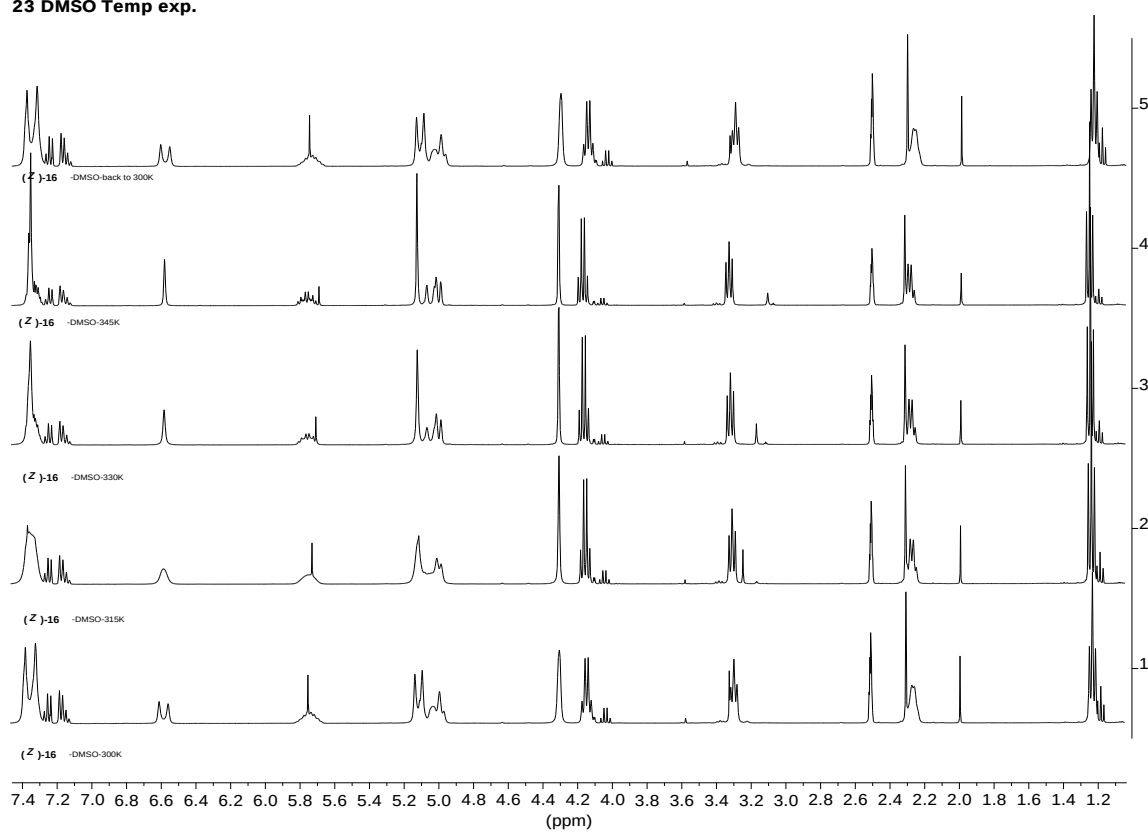


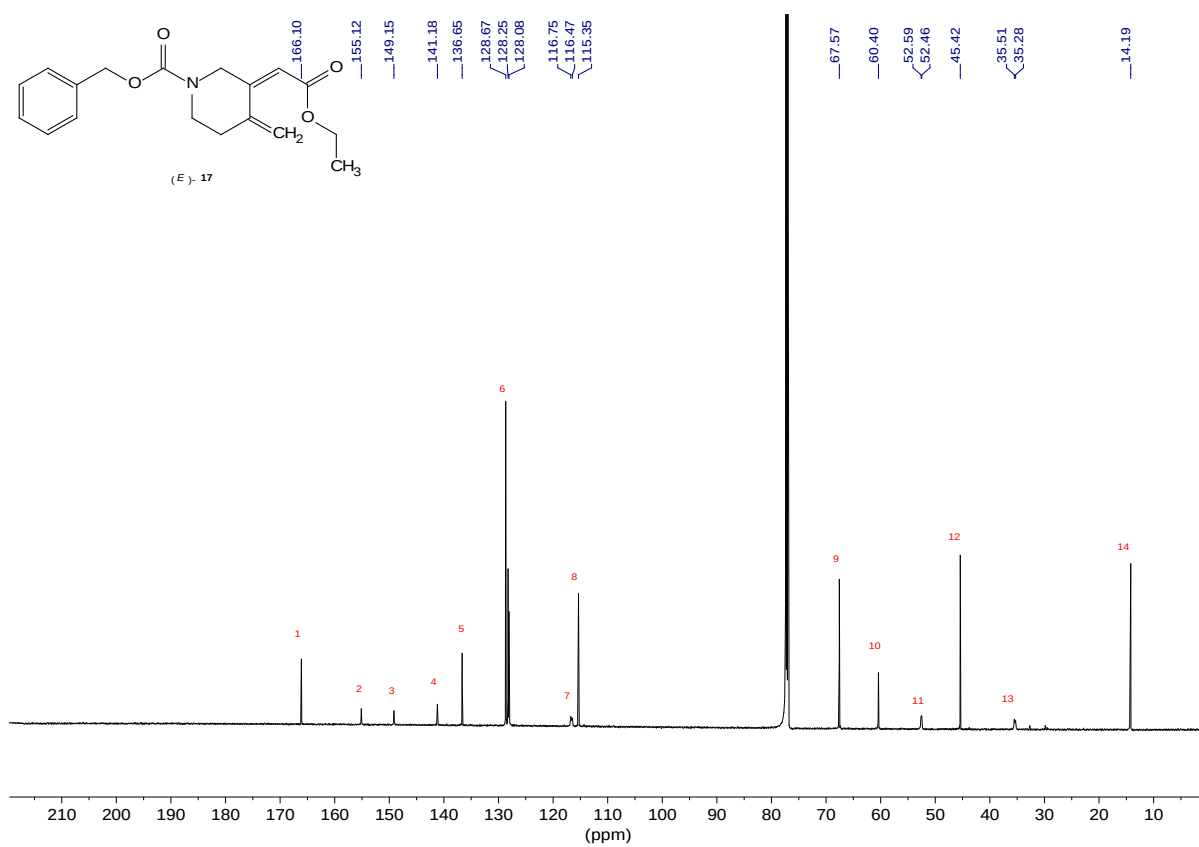
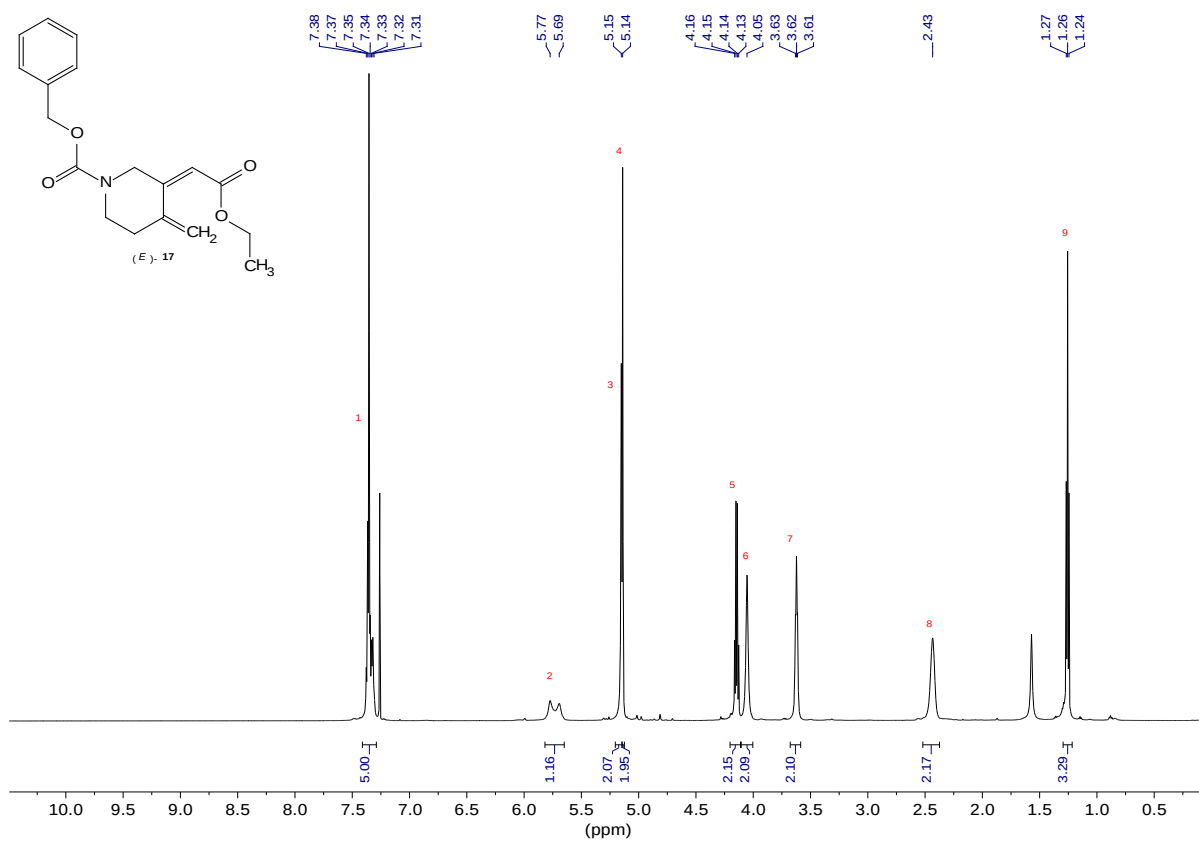


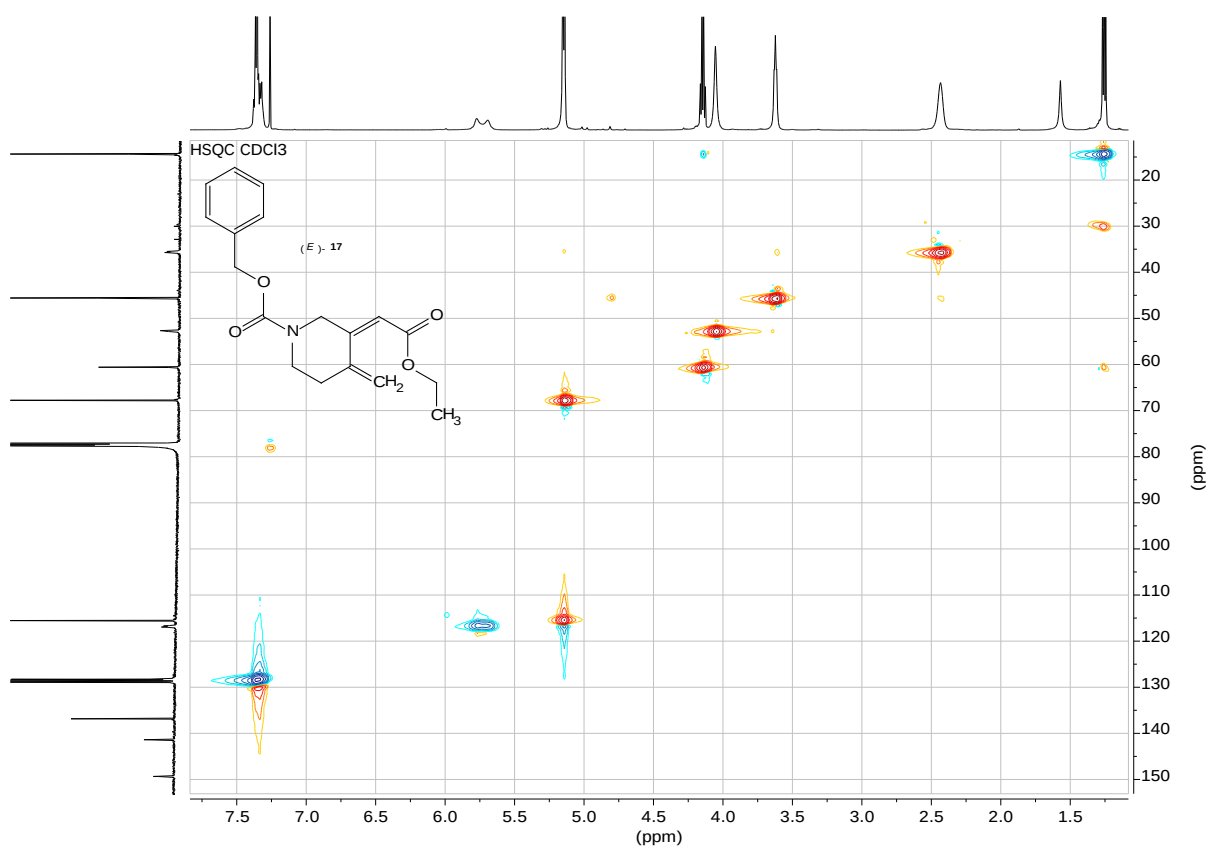
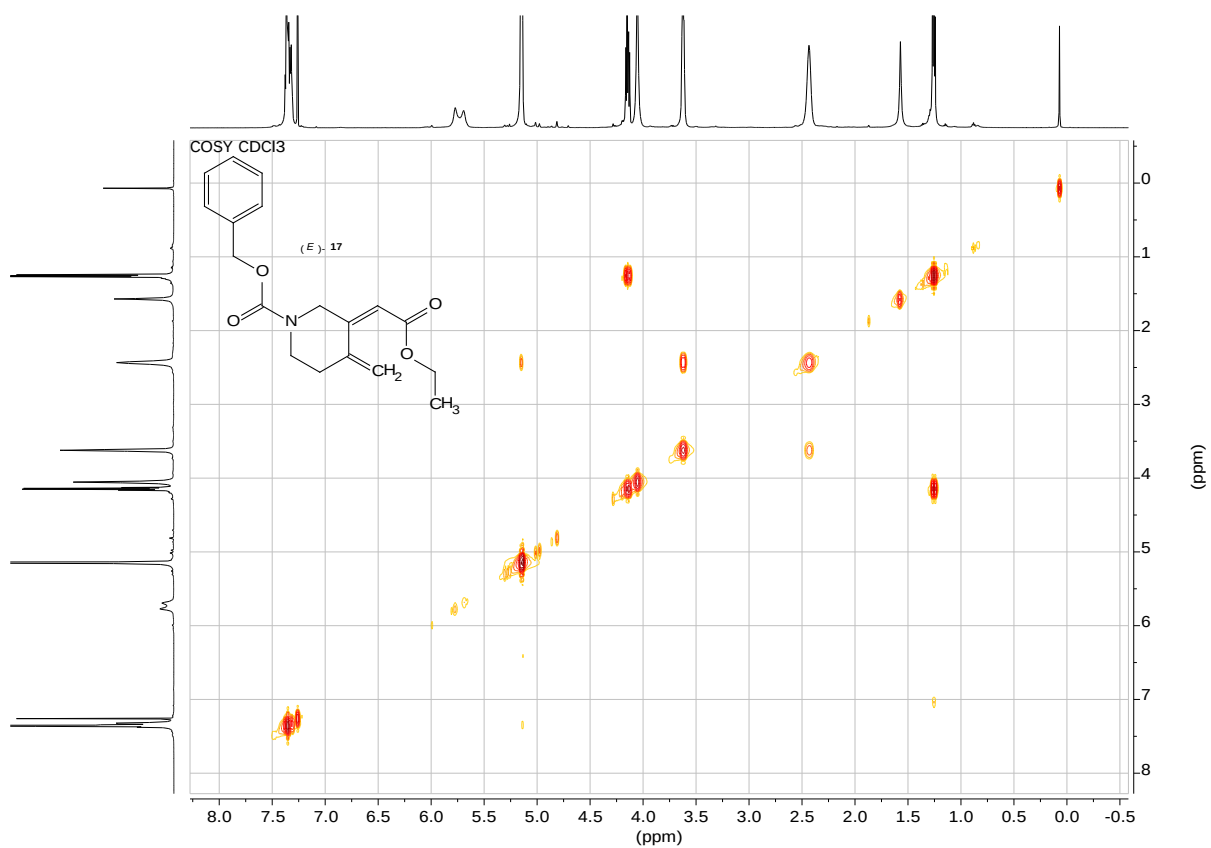


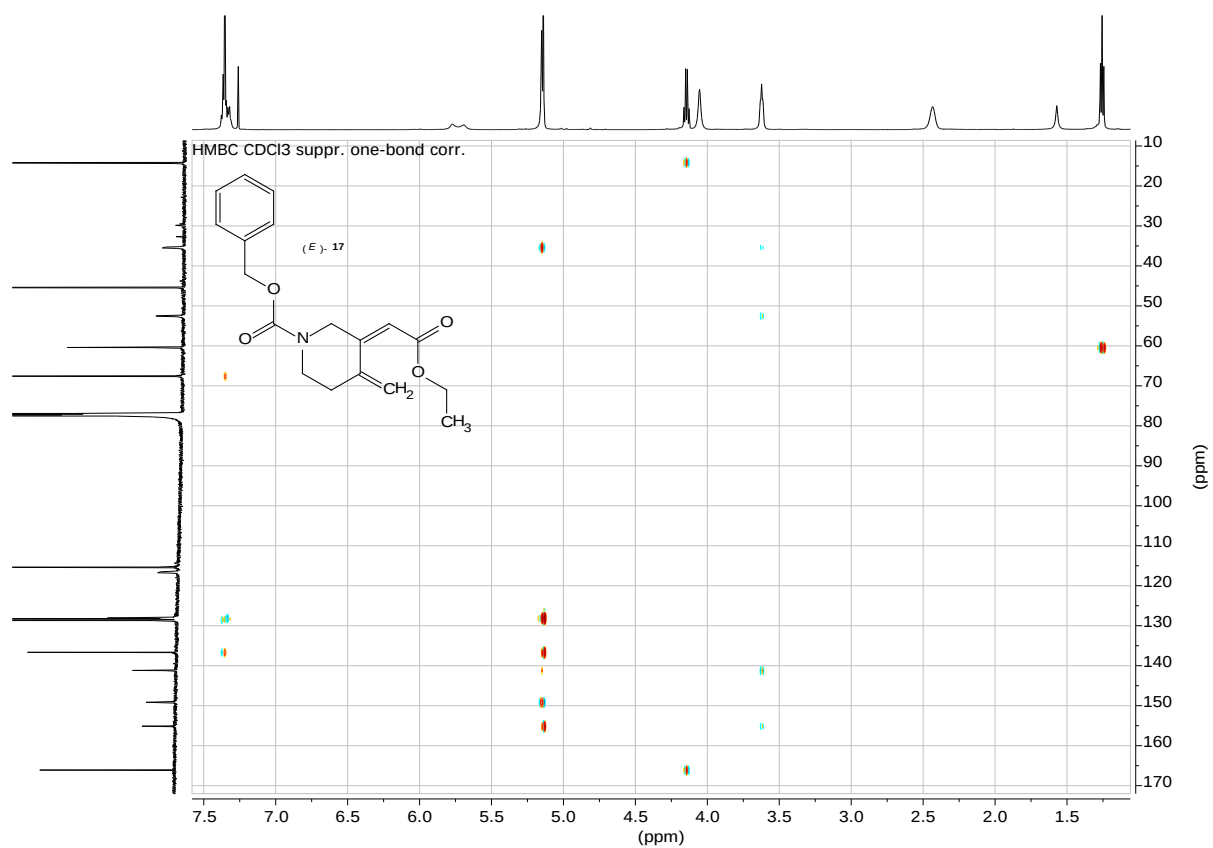


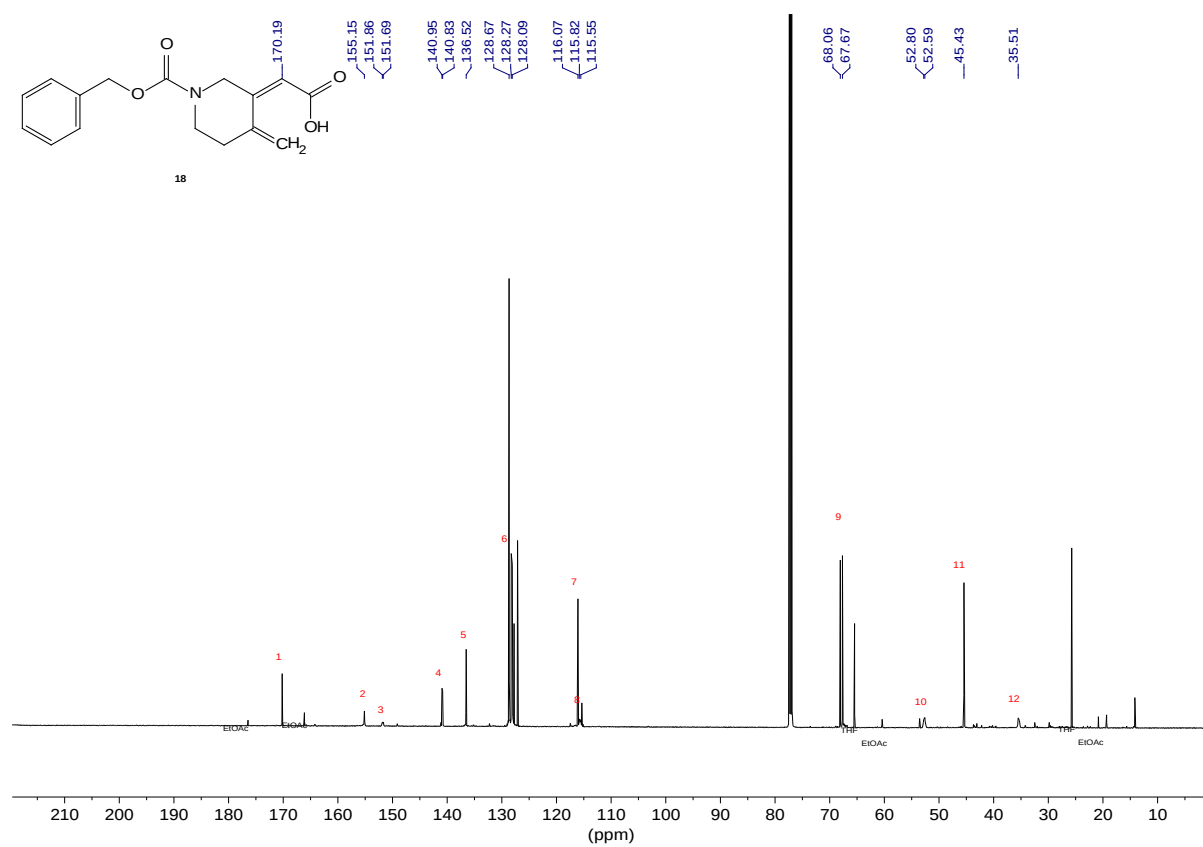
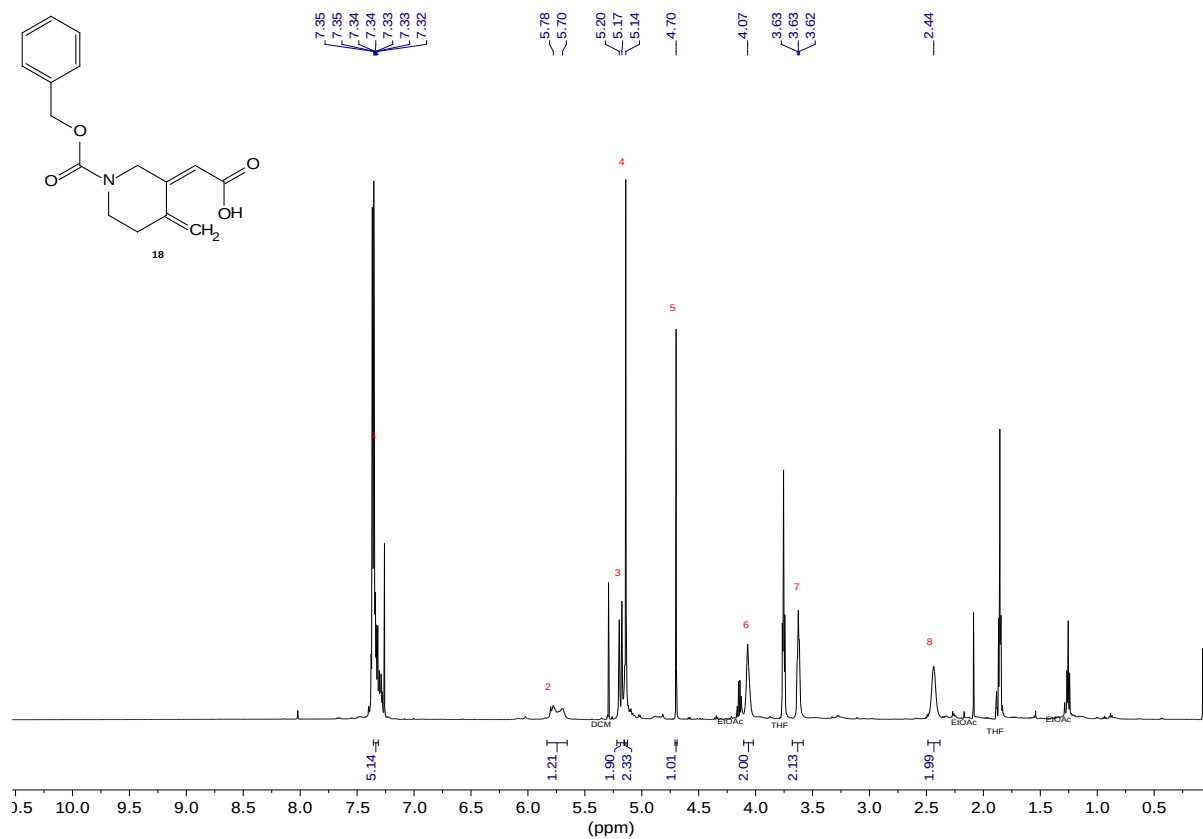
23 DMSO Temp exp.

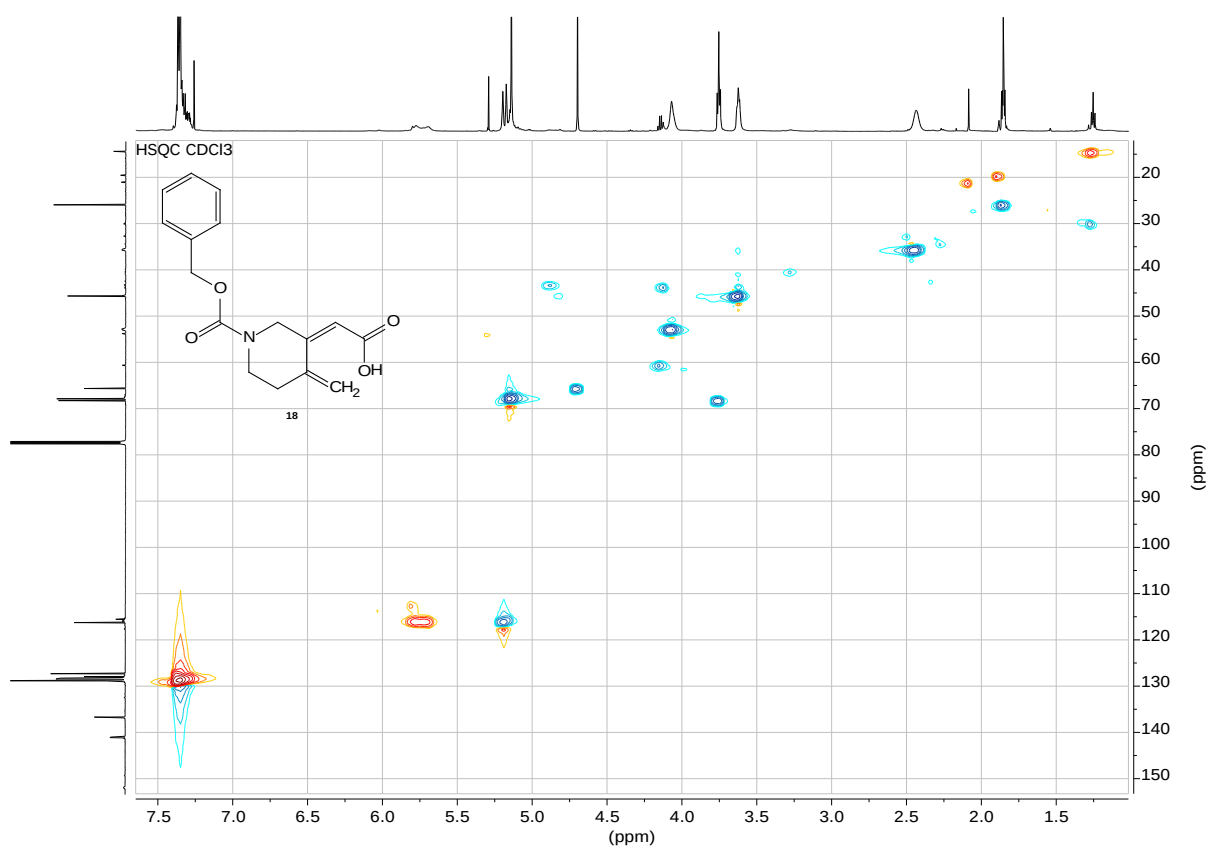
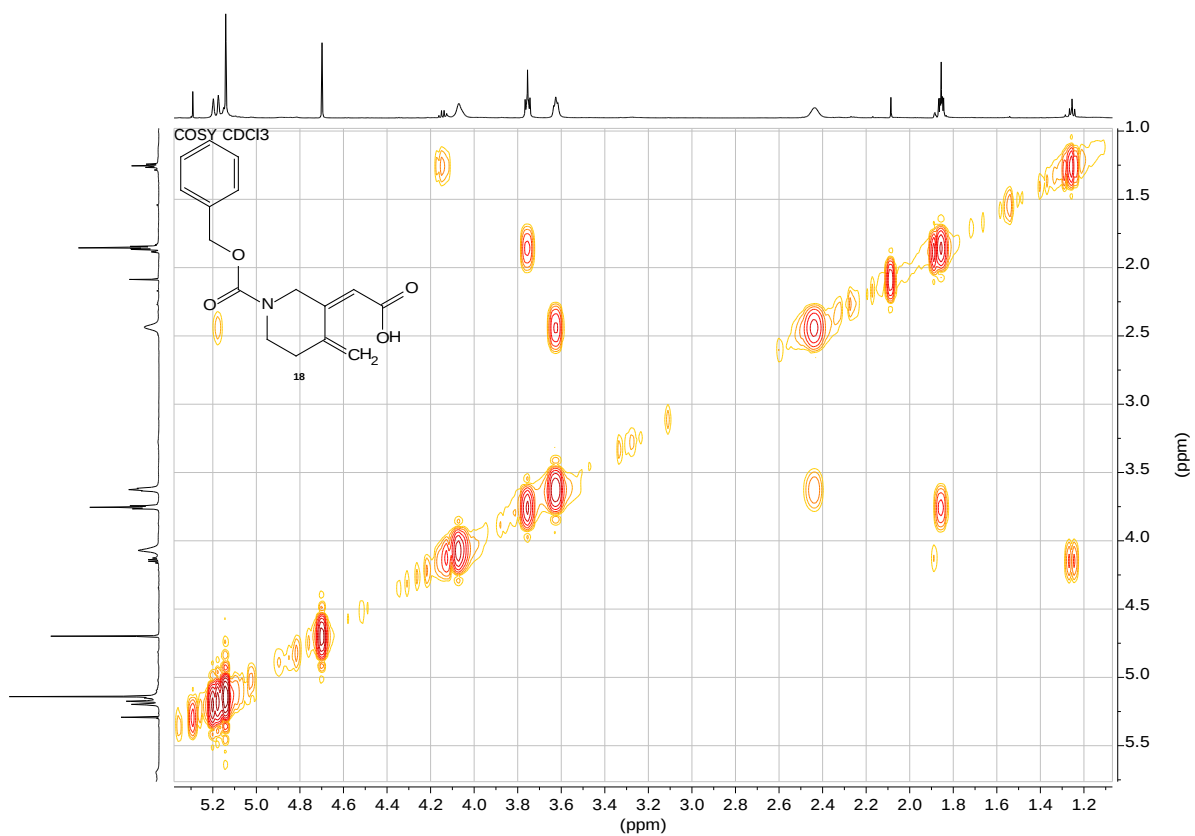


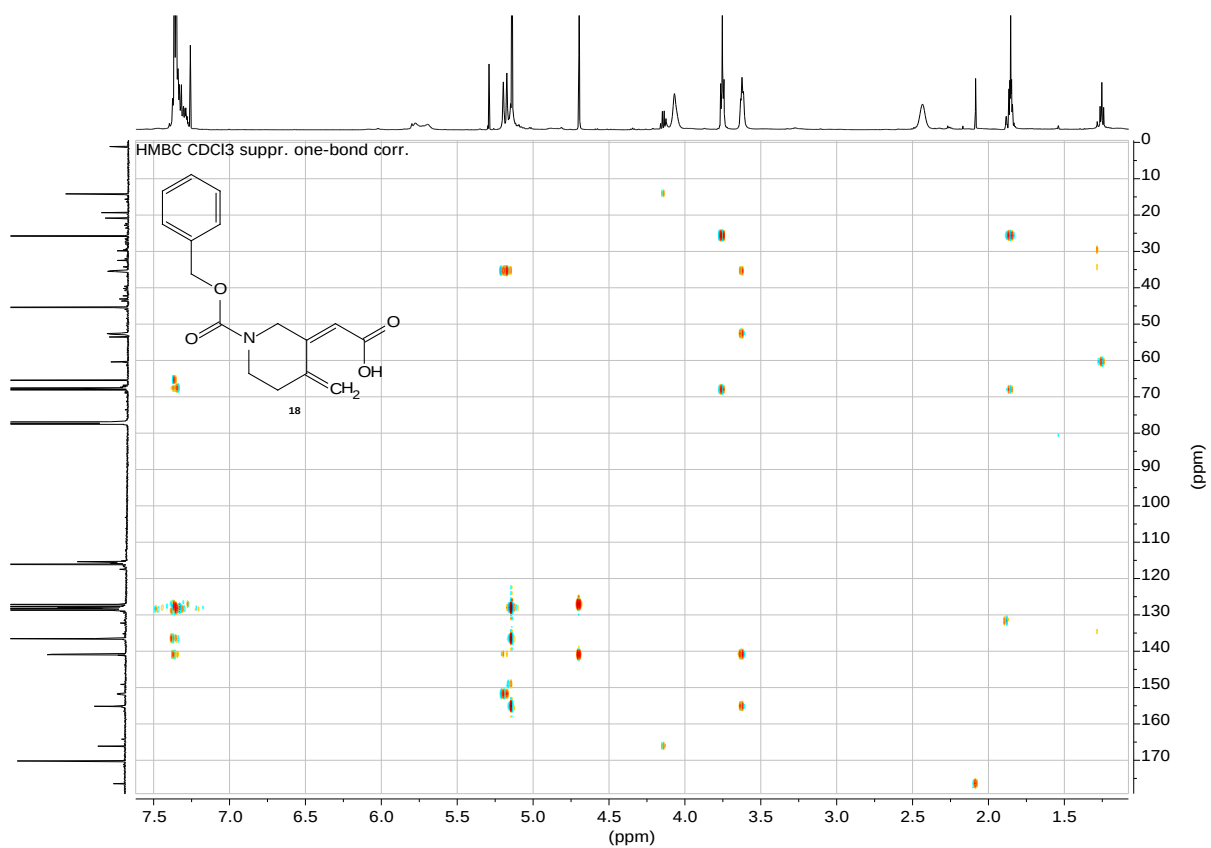


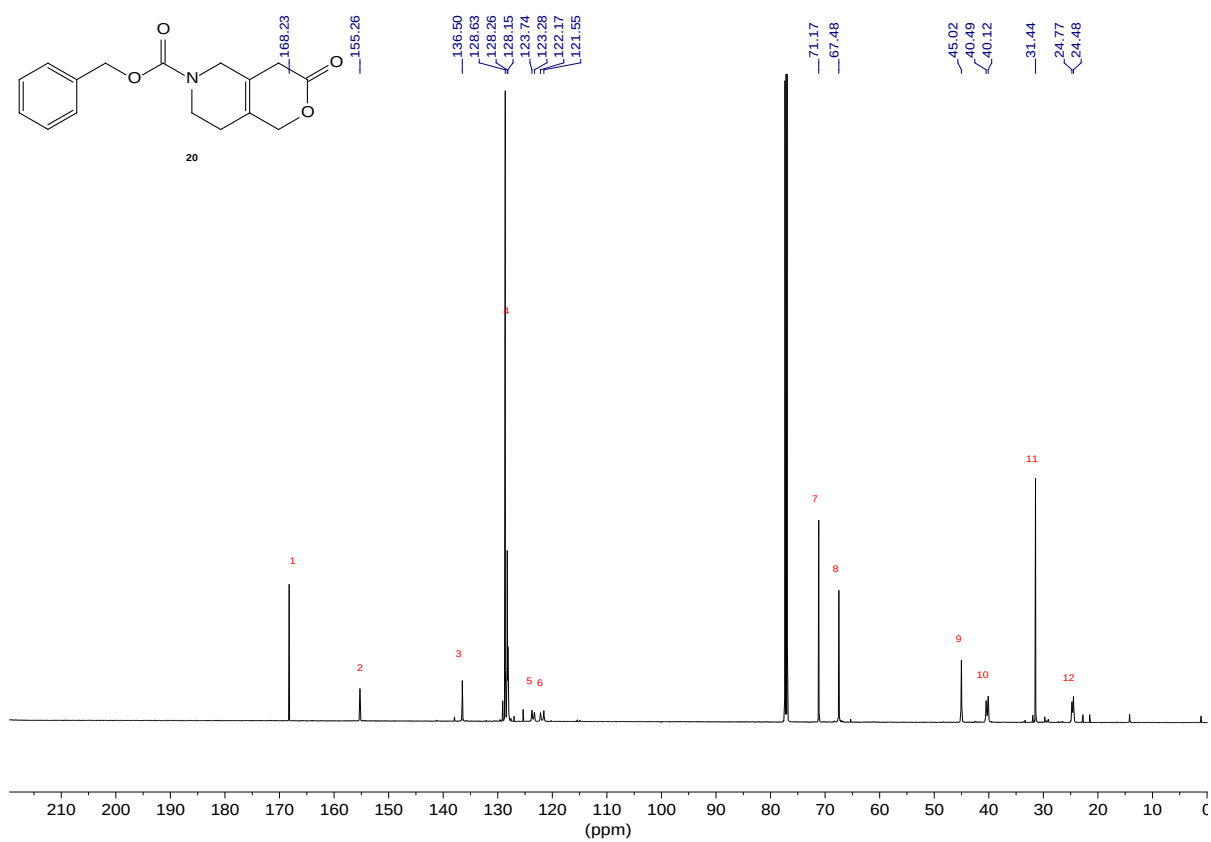
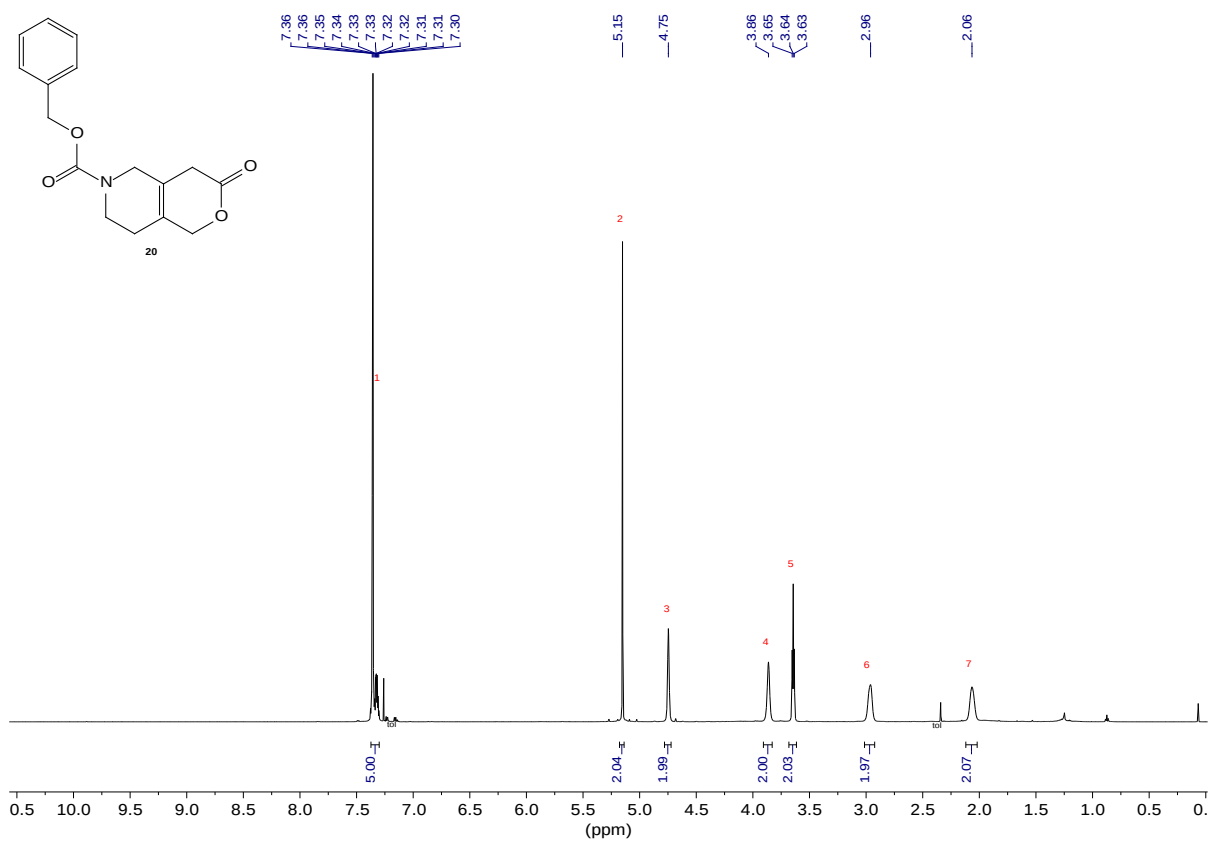


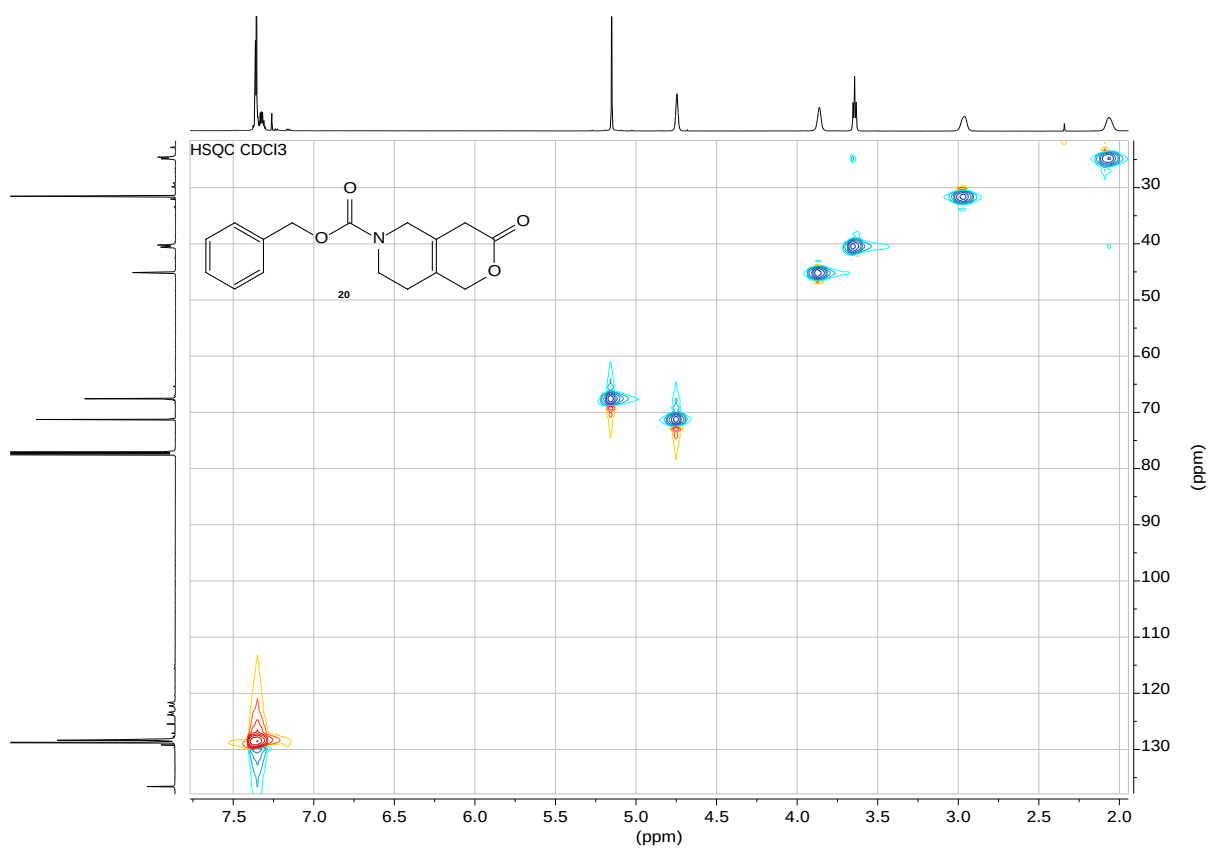
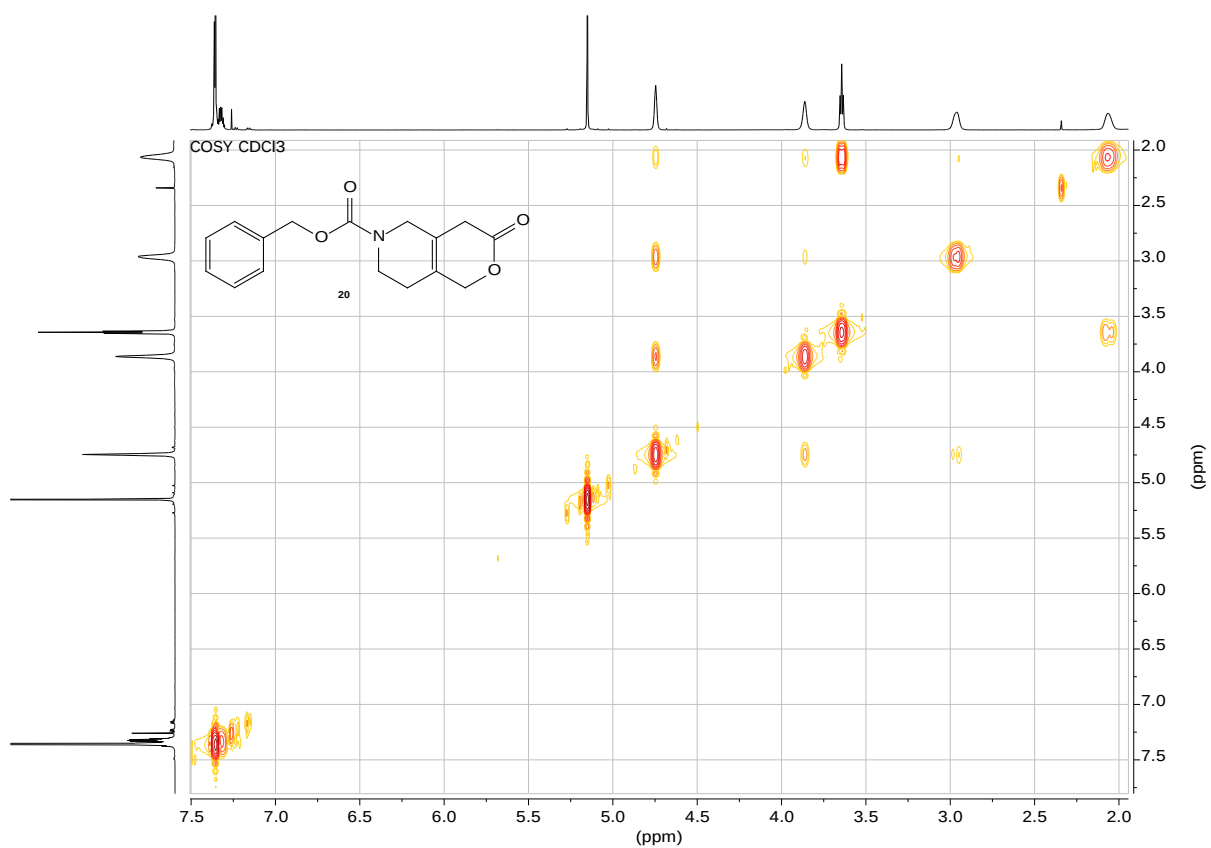


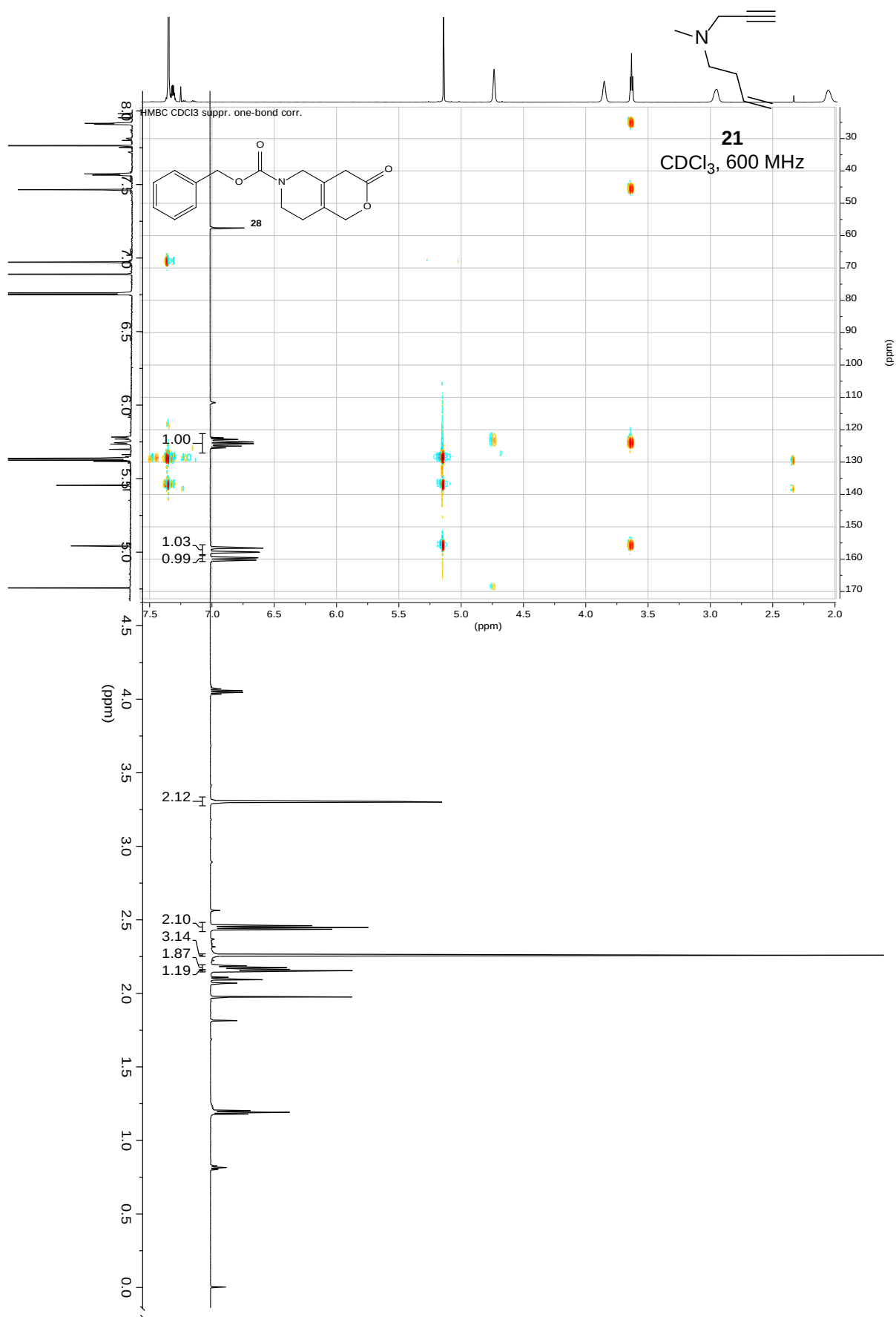


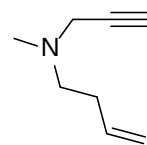




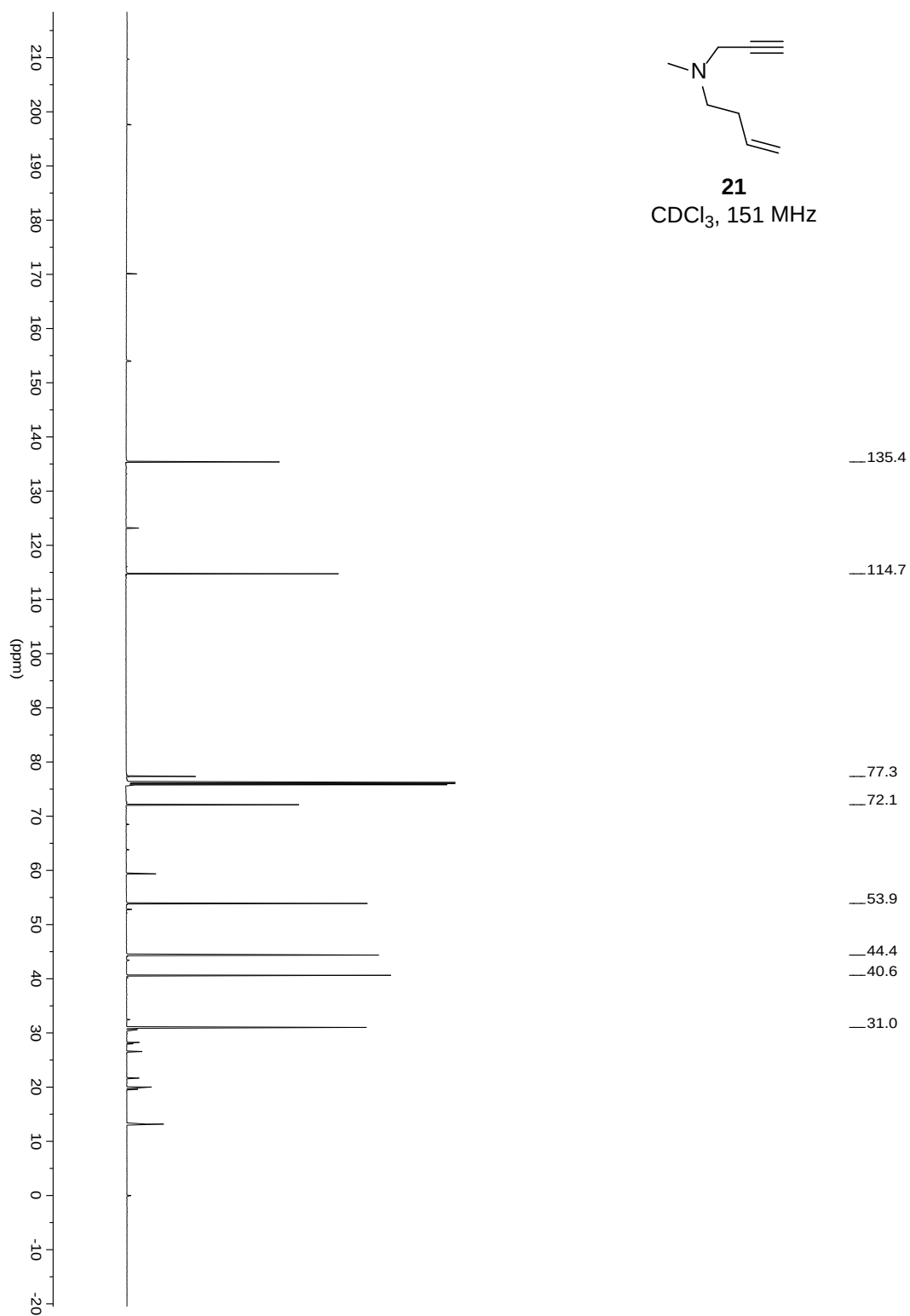


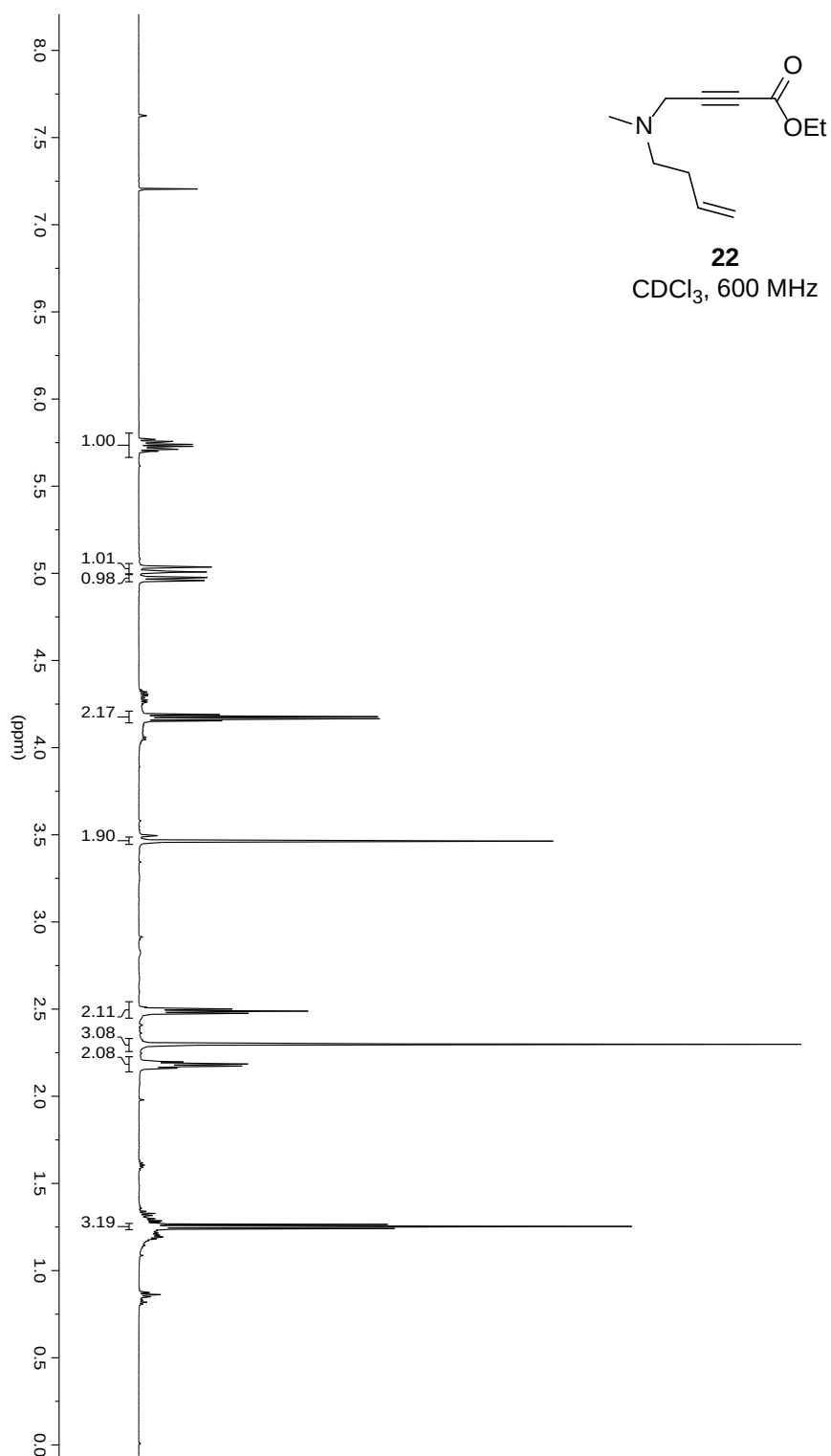


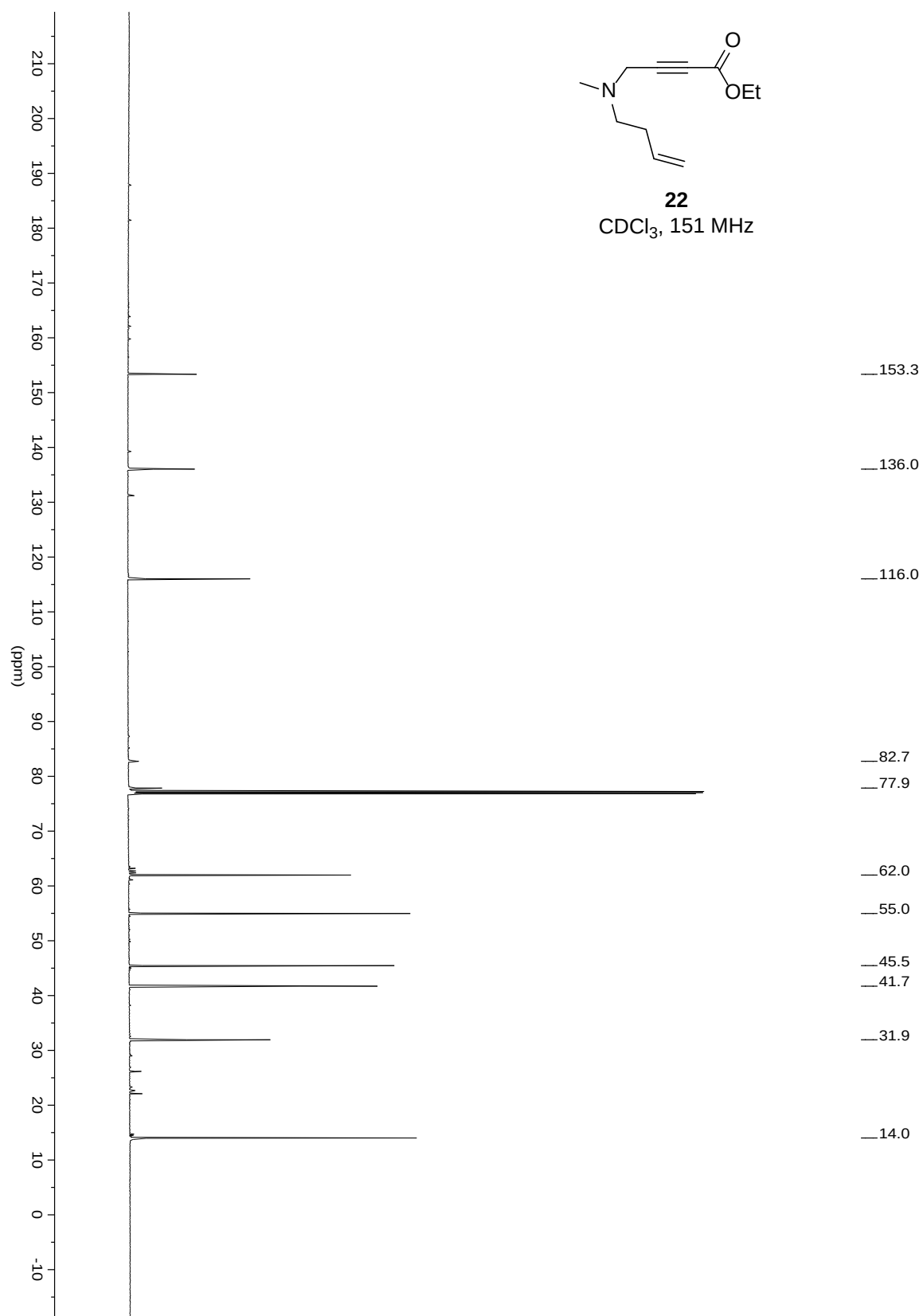


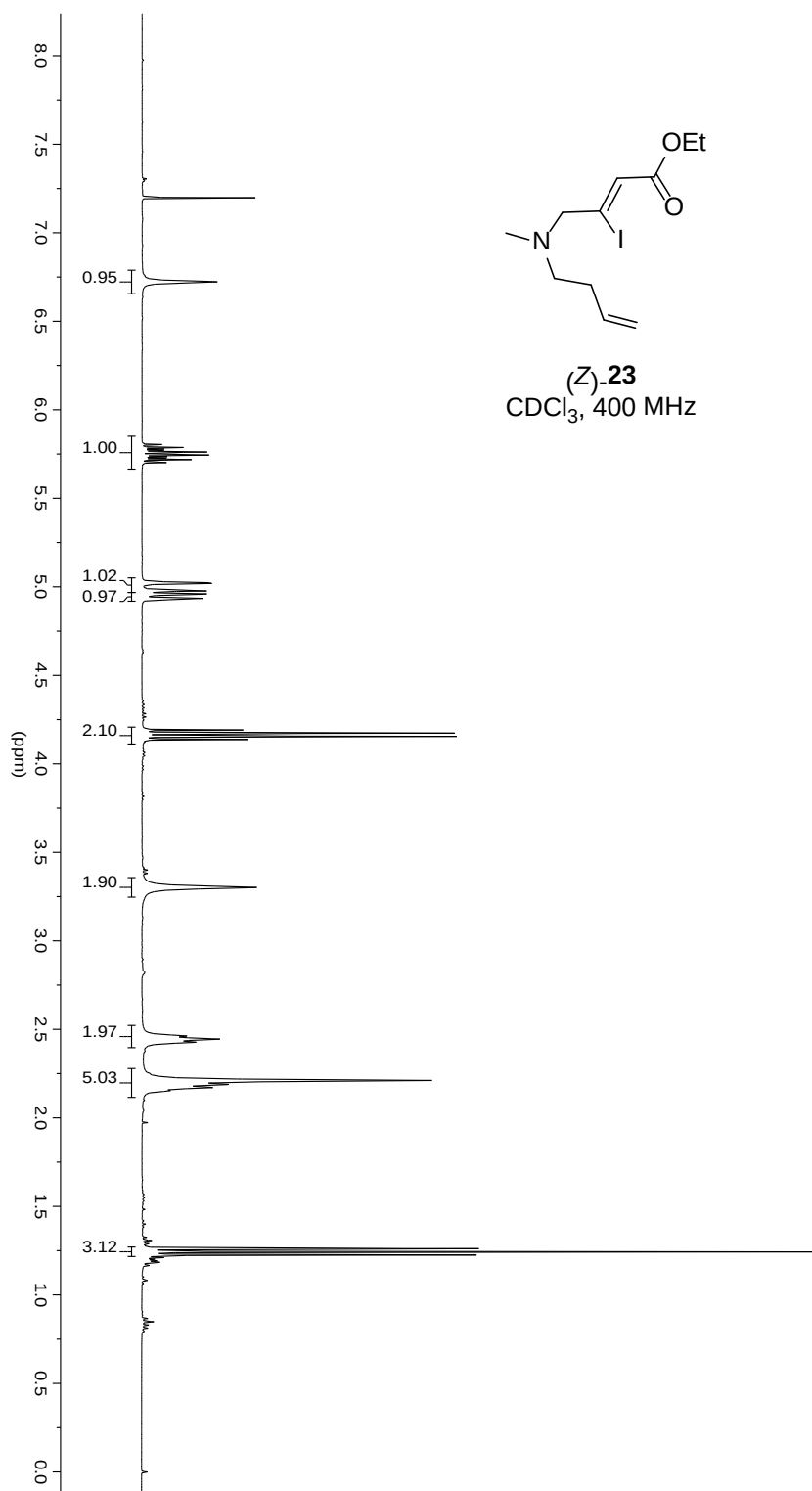


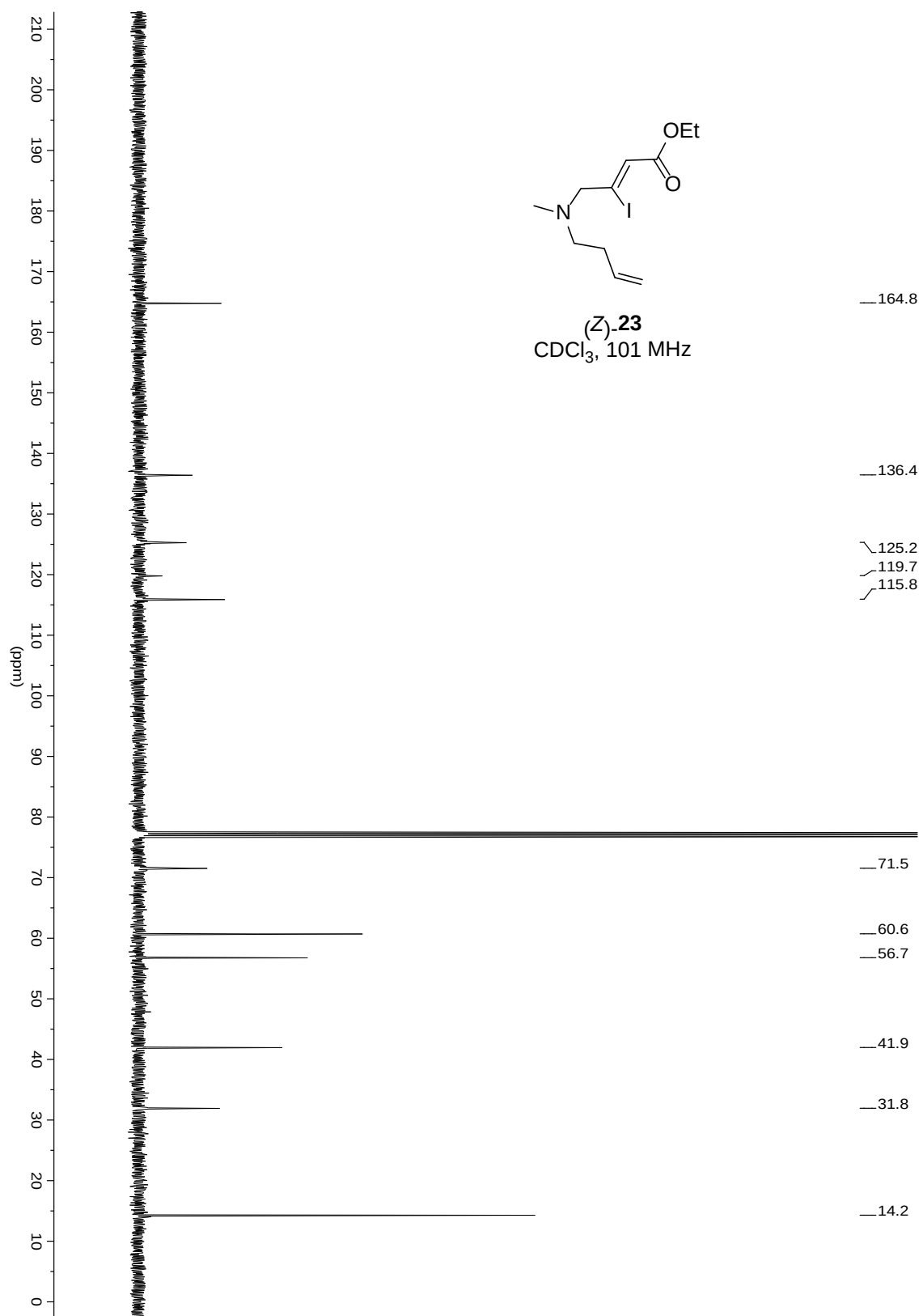
21
CDCl₃, 151 MHz

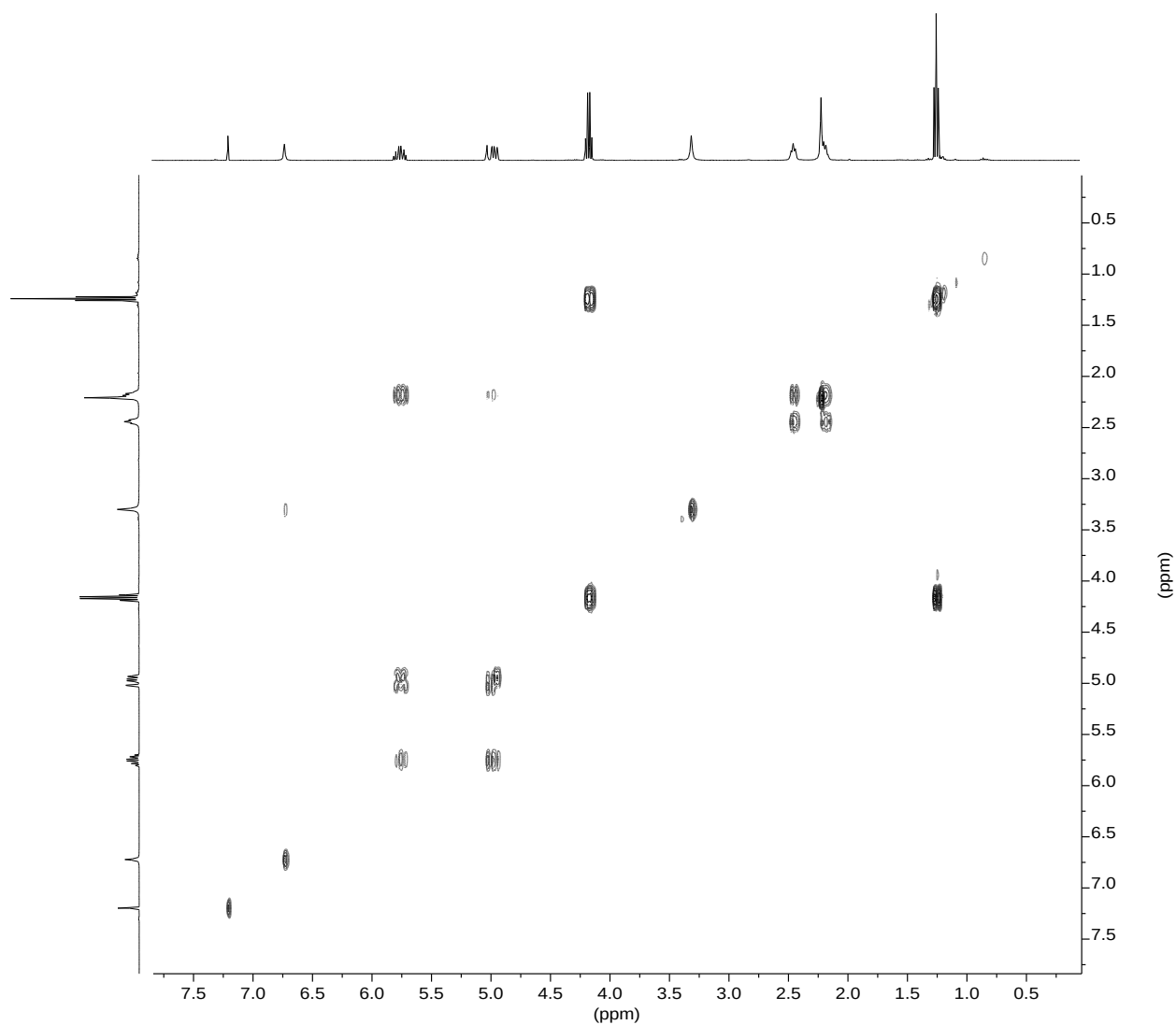
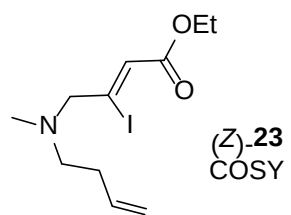


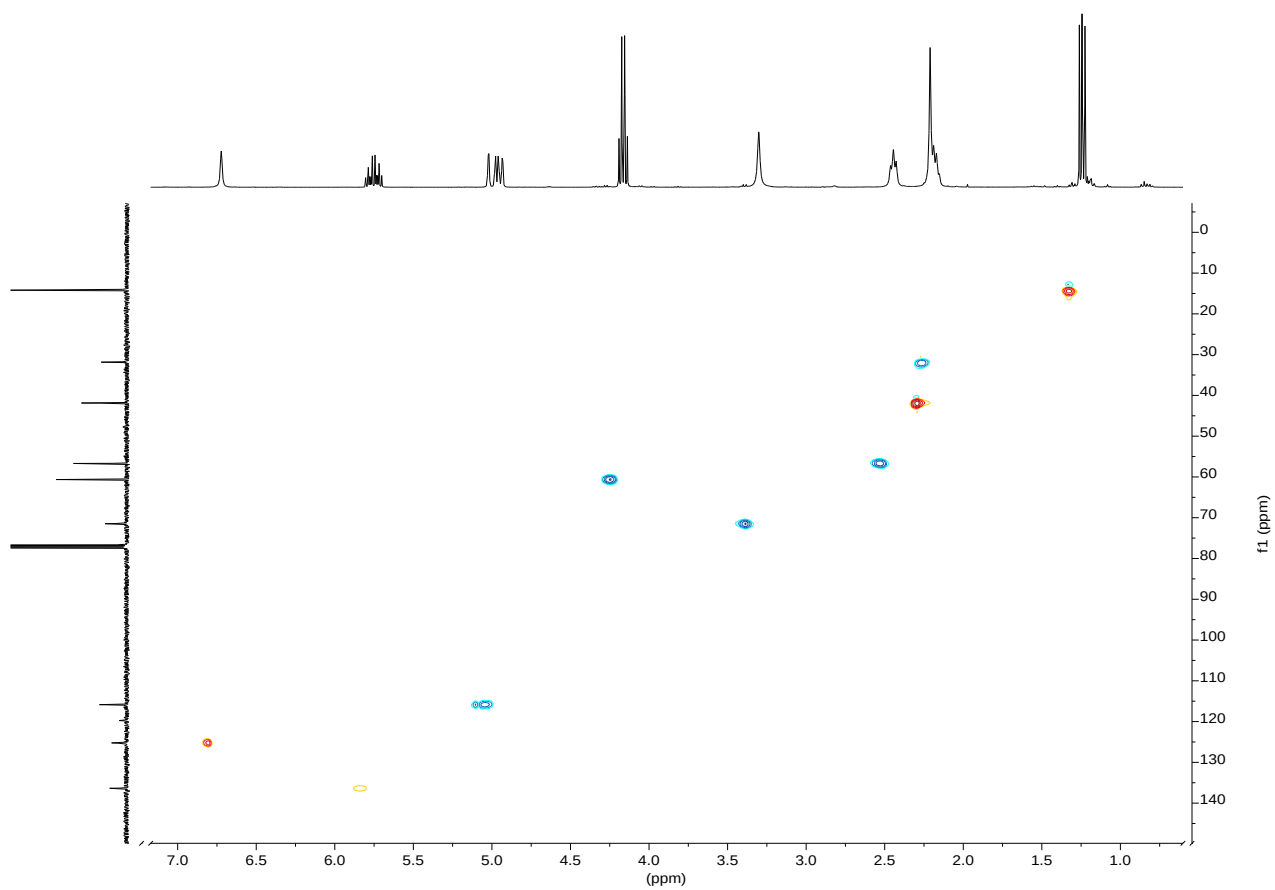
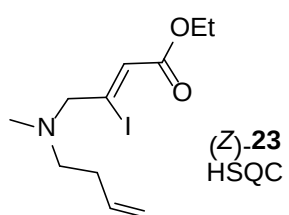


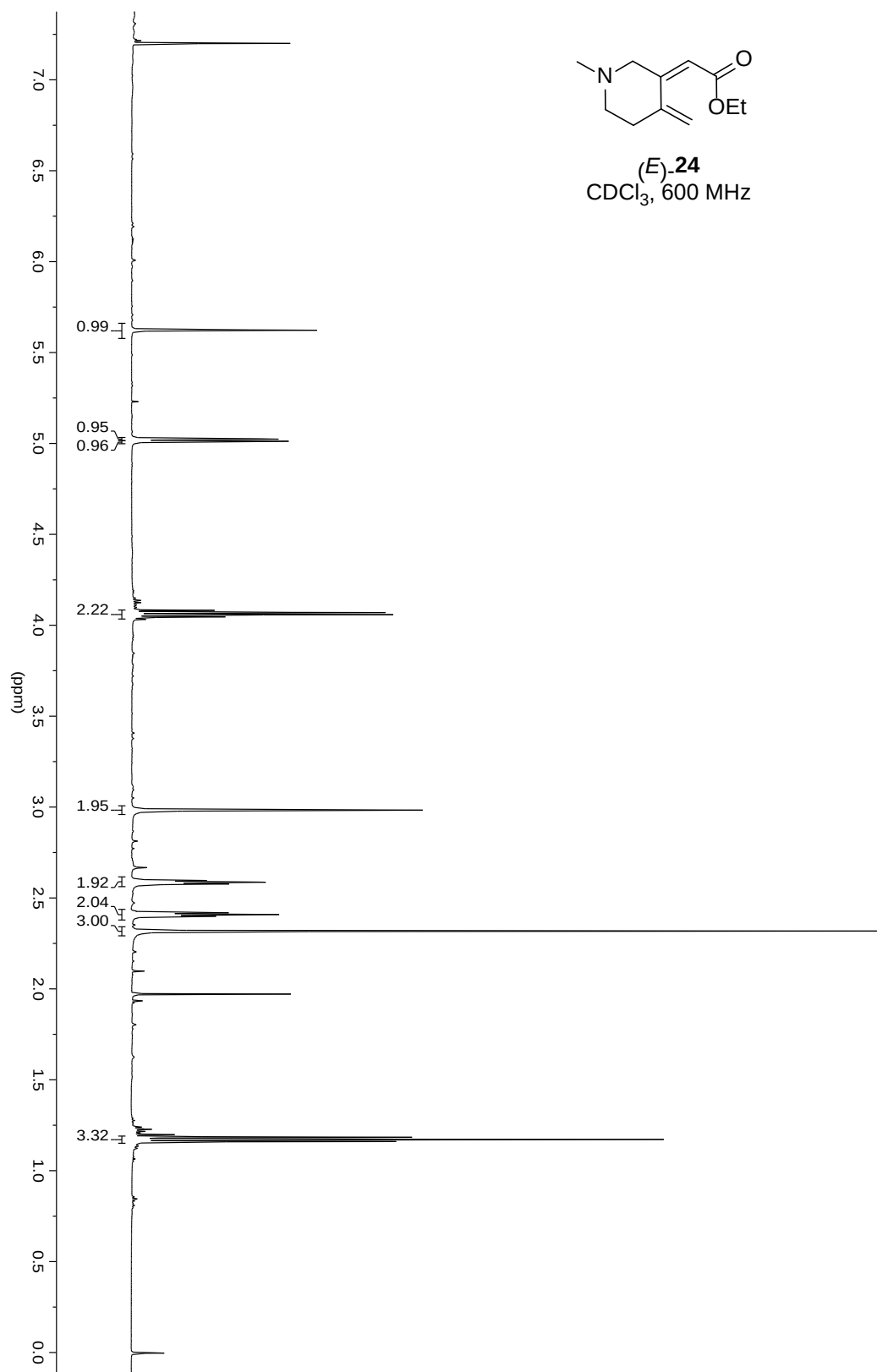


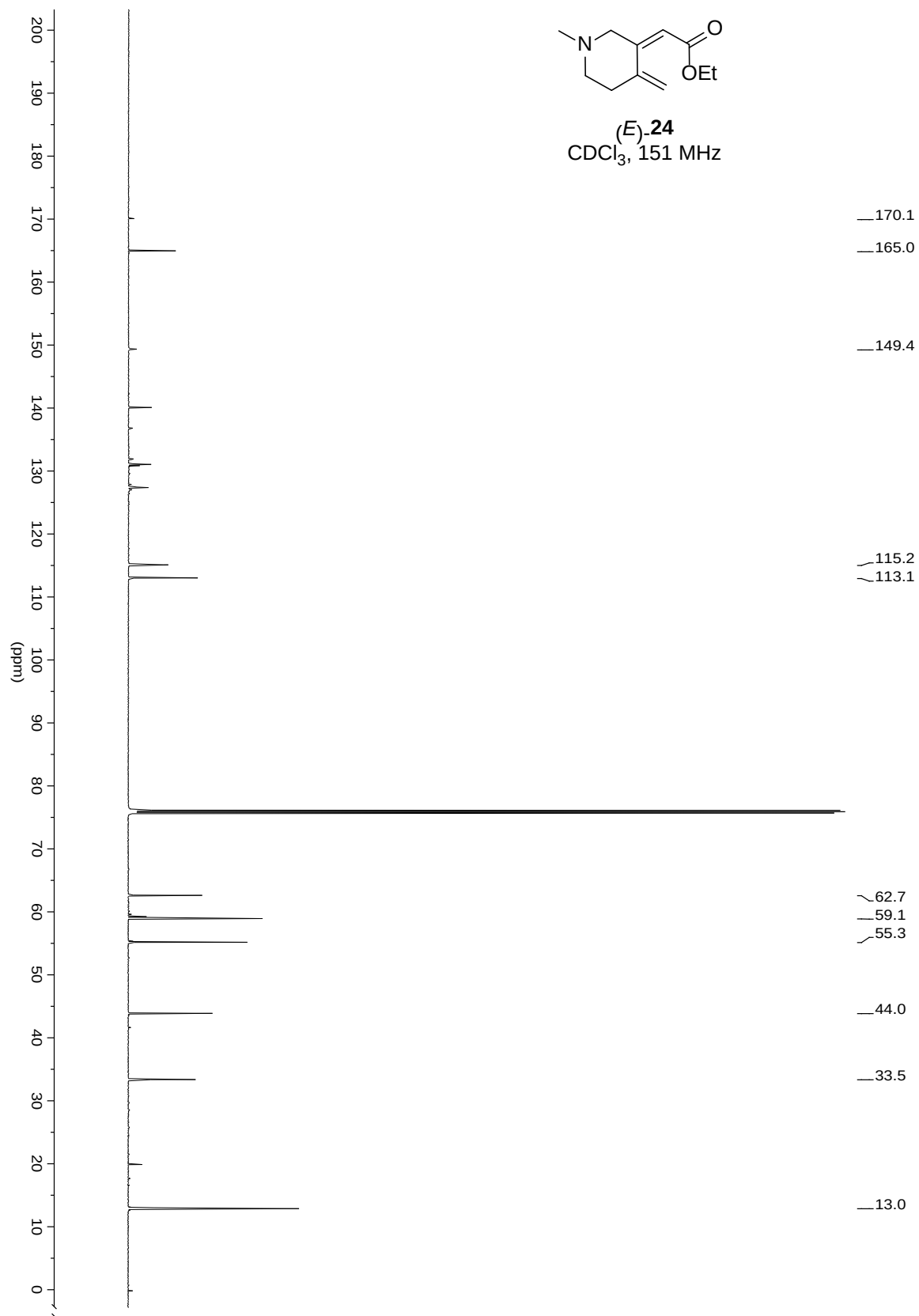


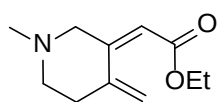




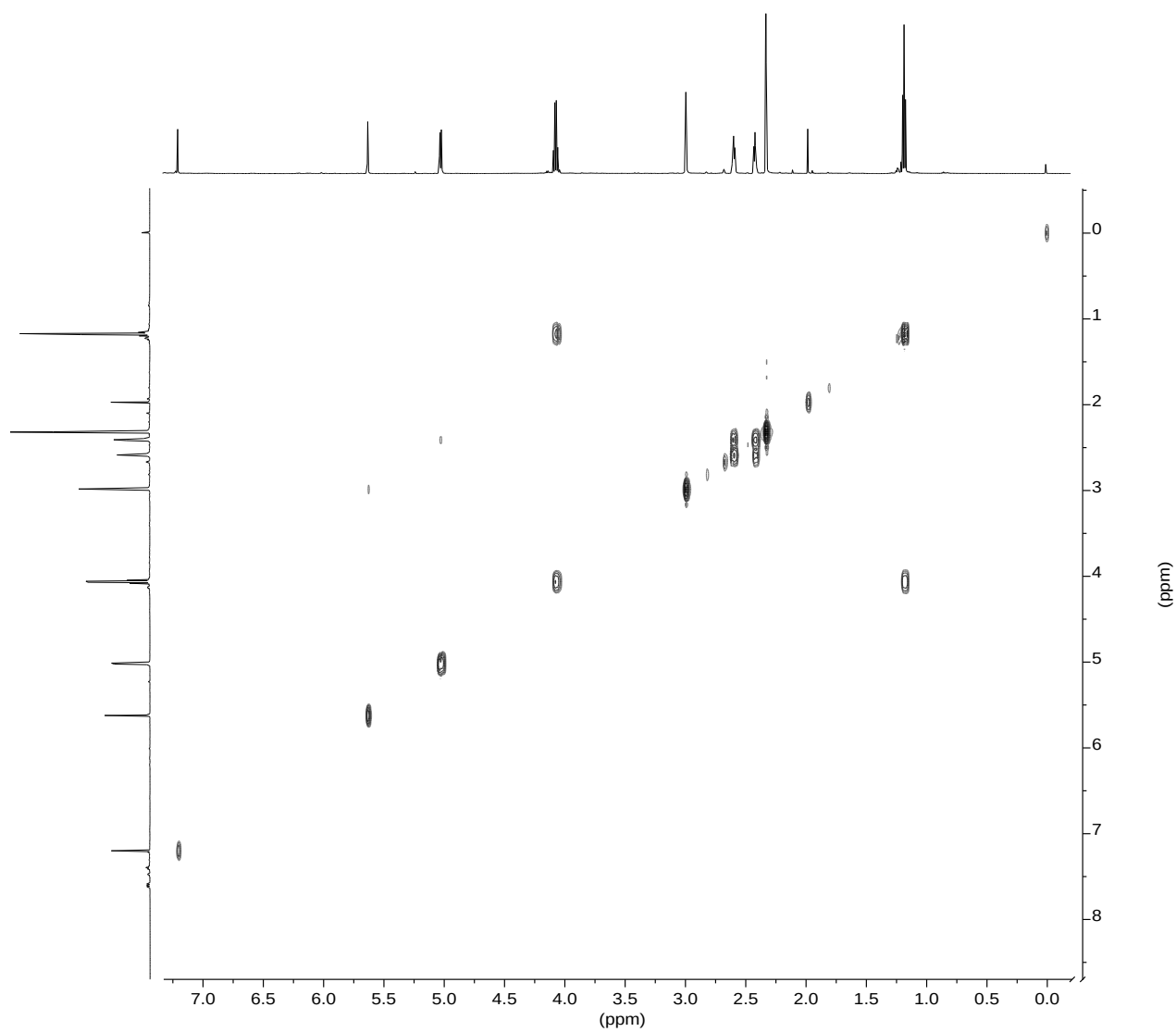


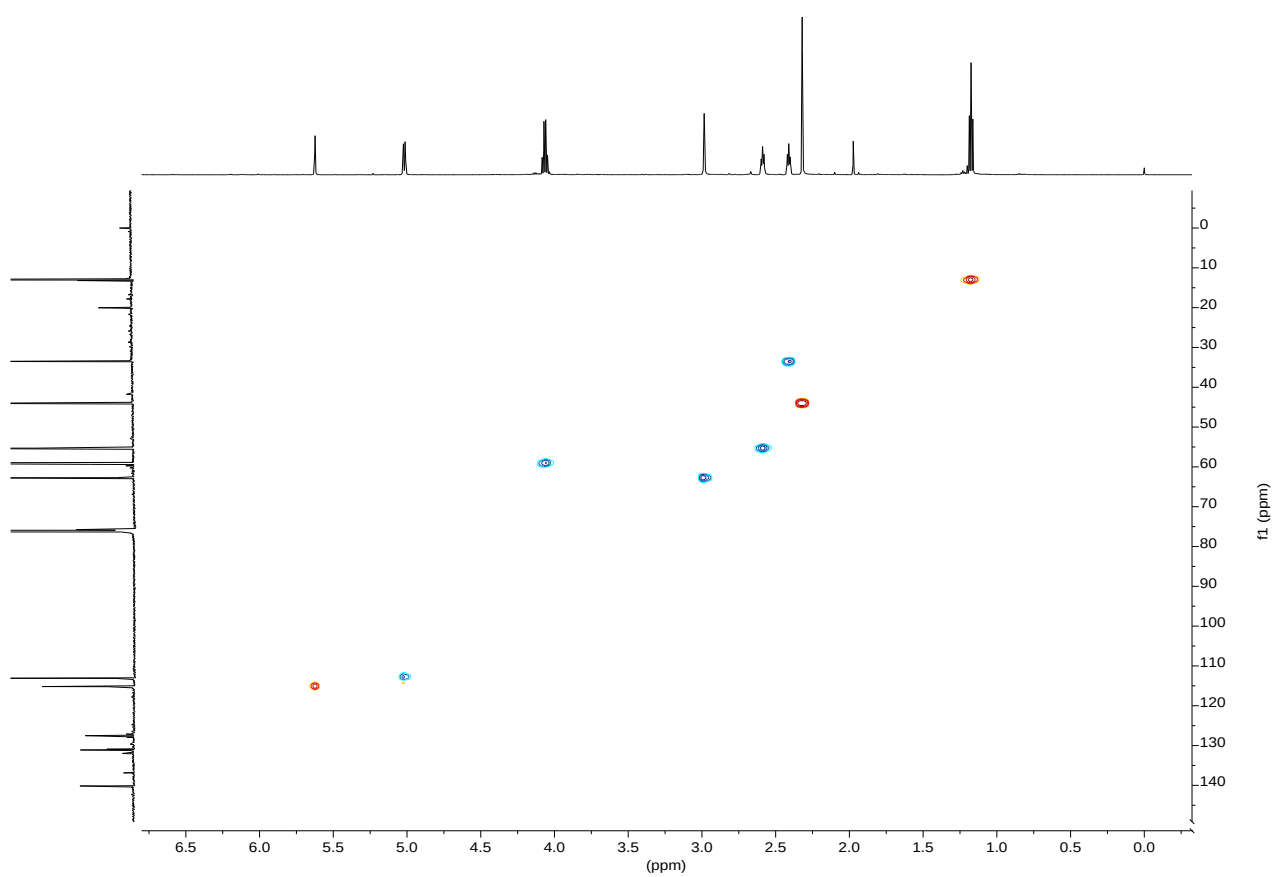
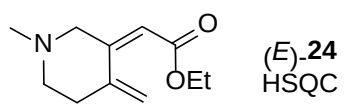


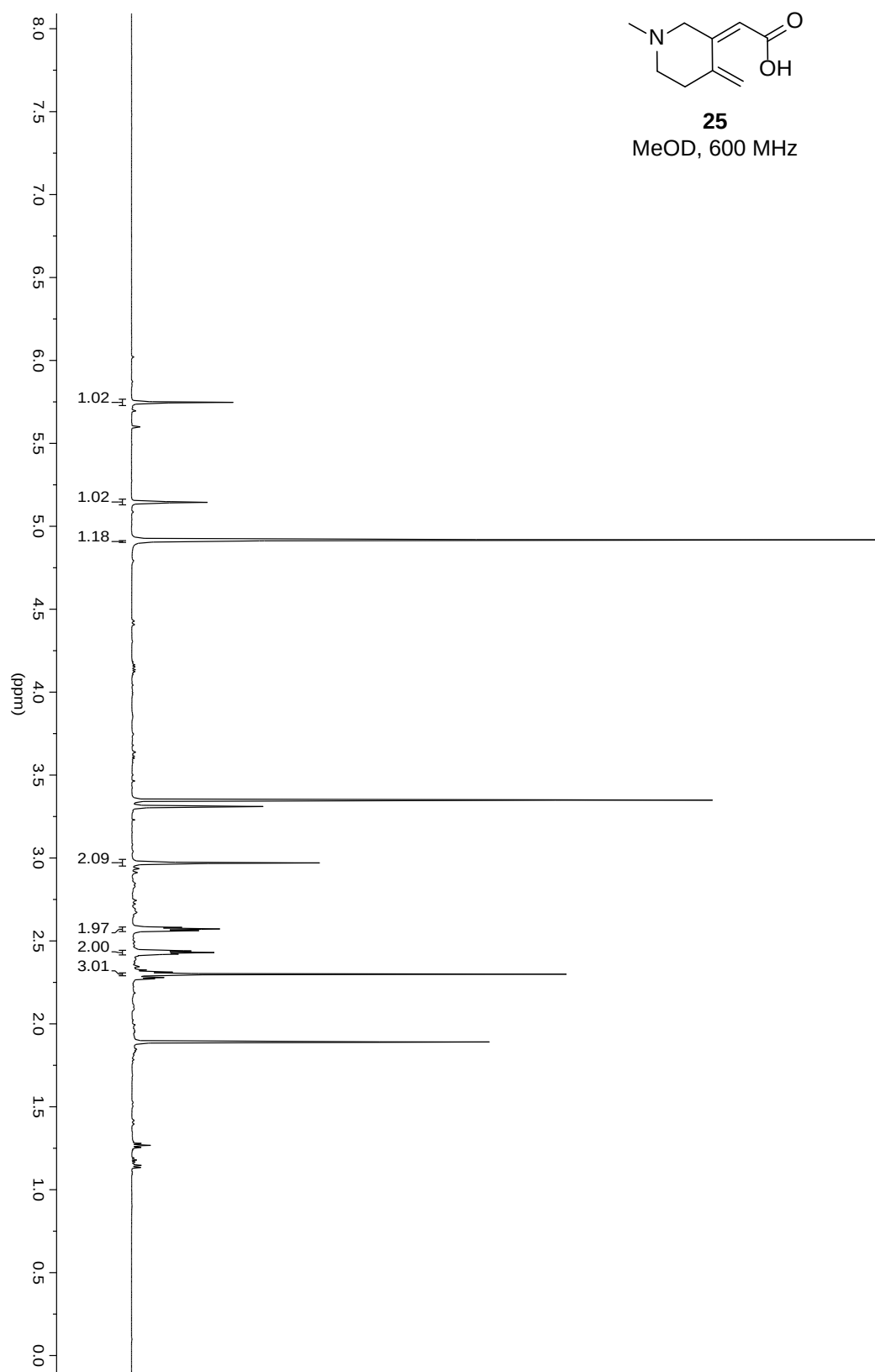


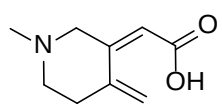


(E)-24
COSY

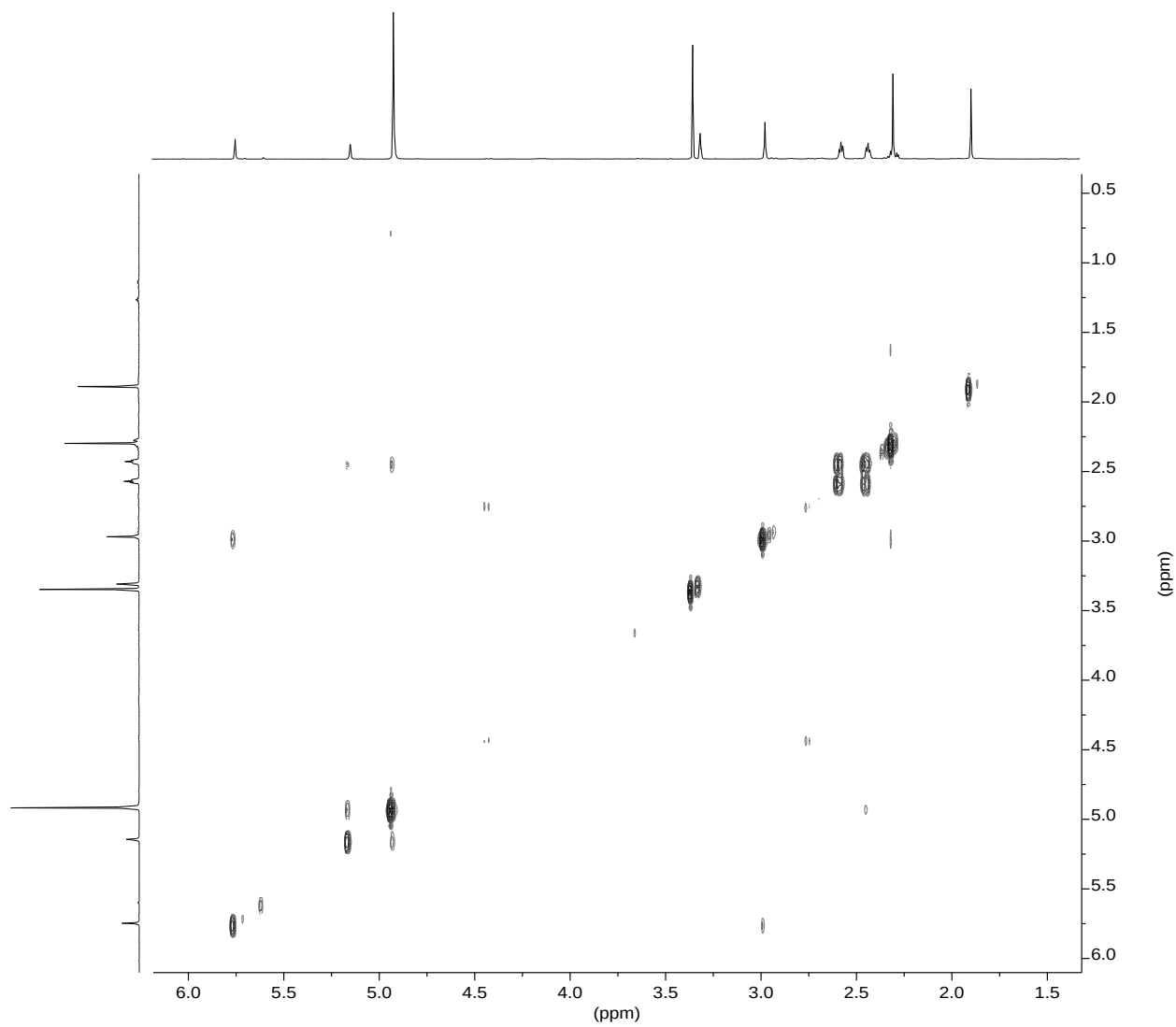


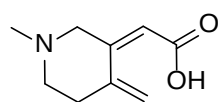




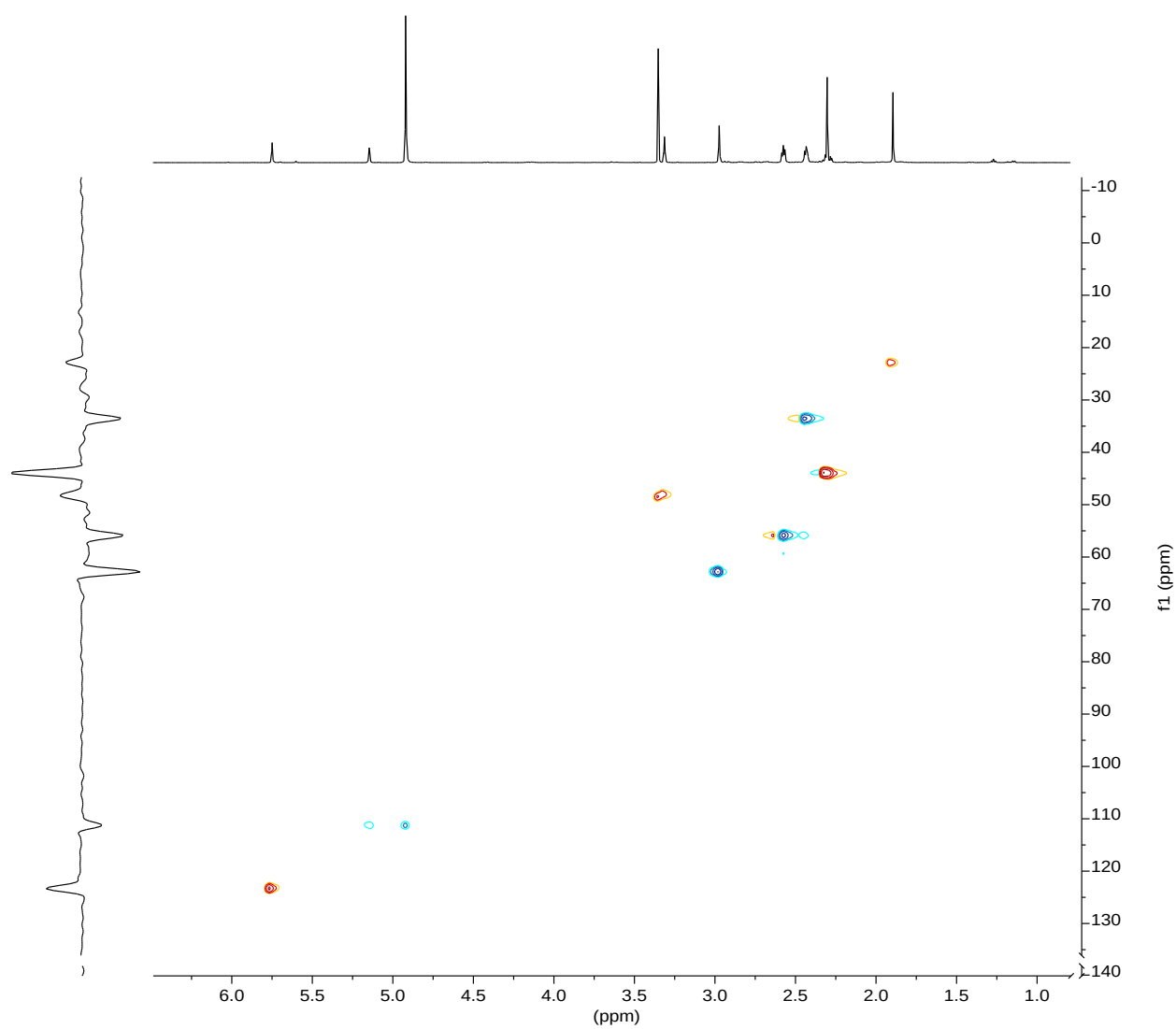


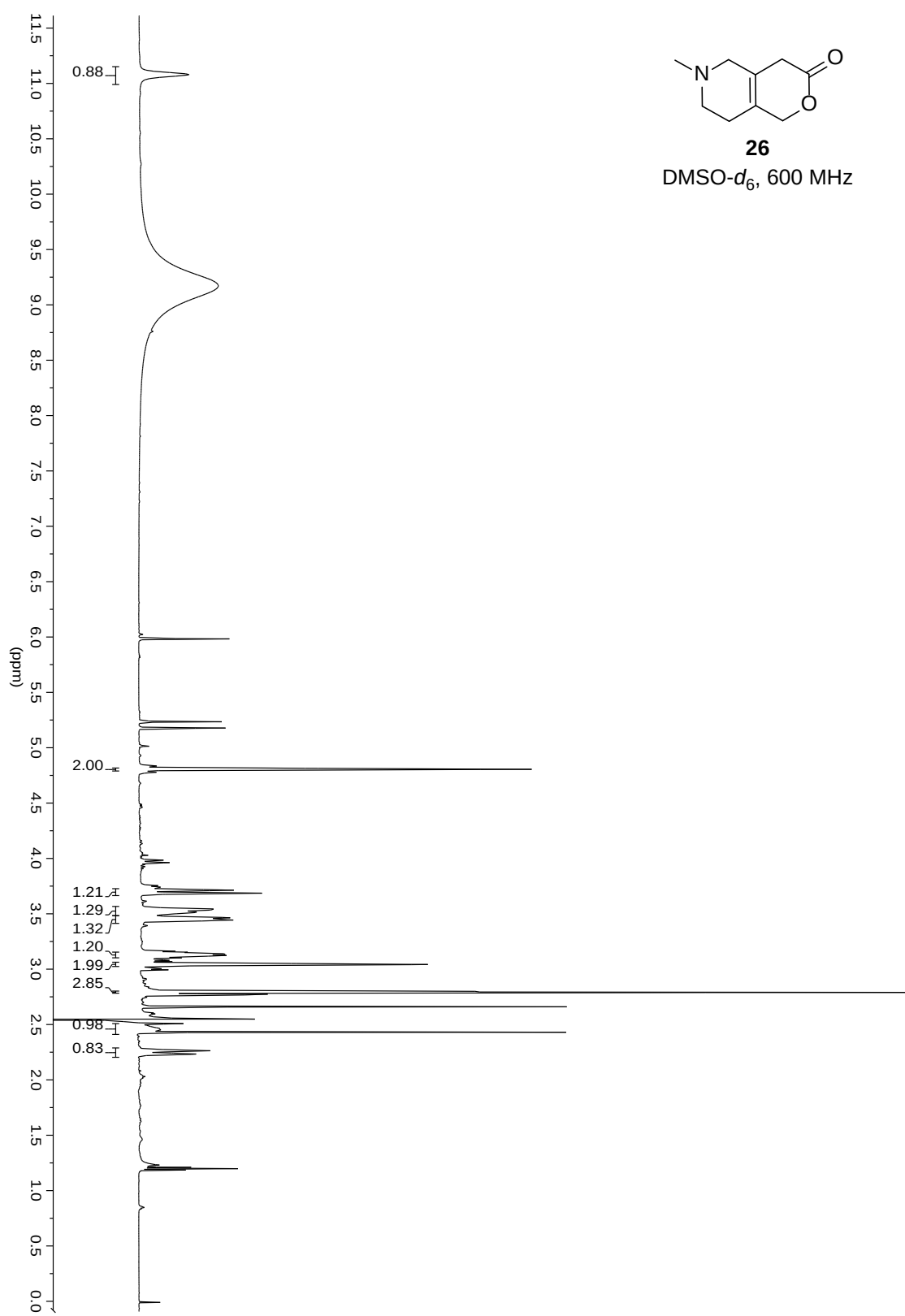
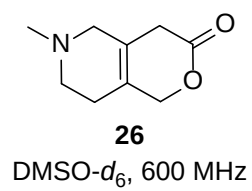
25
COSY

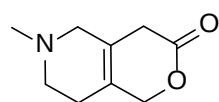




25
HSQC

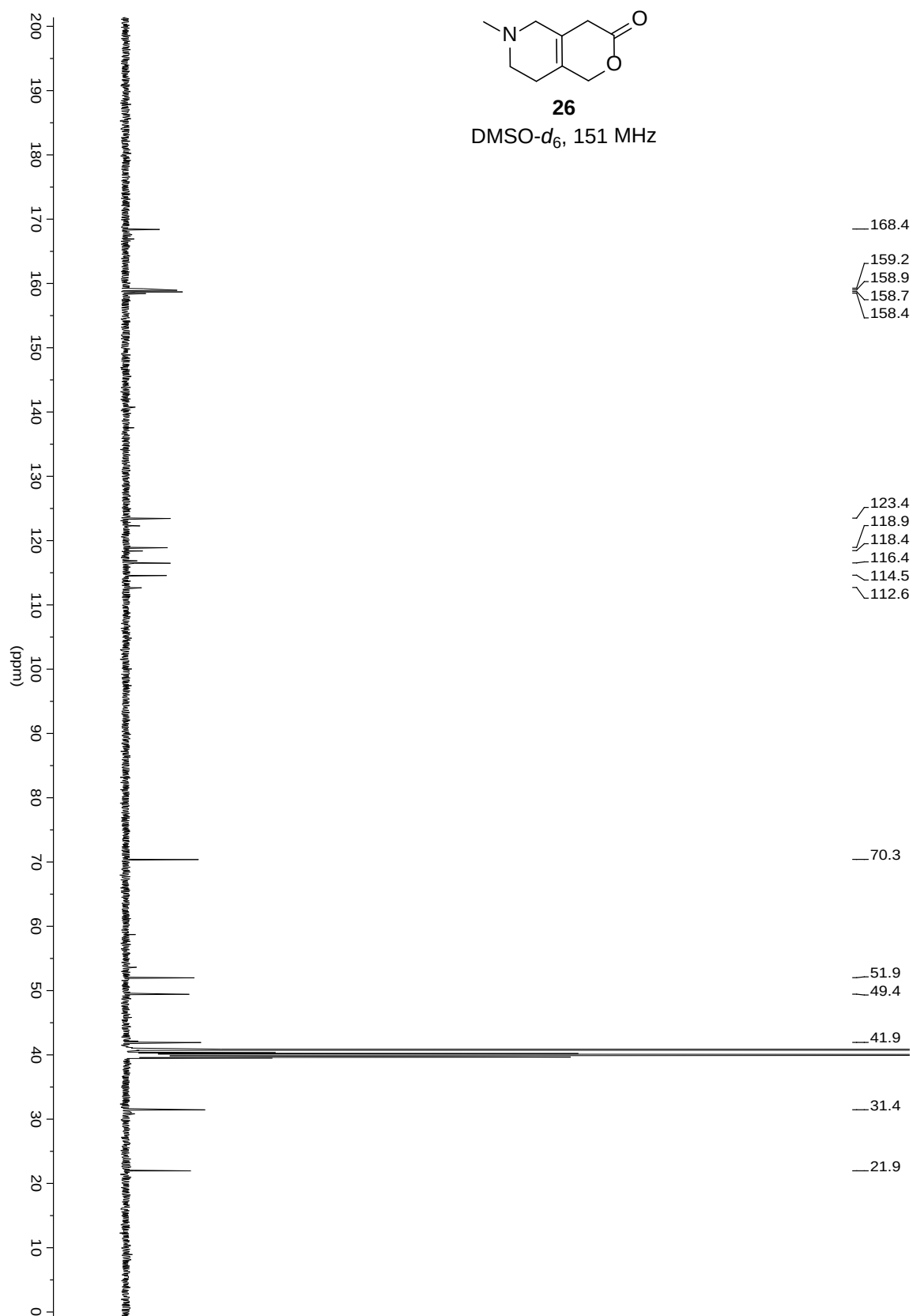


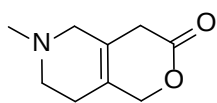




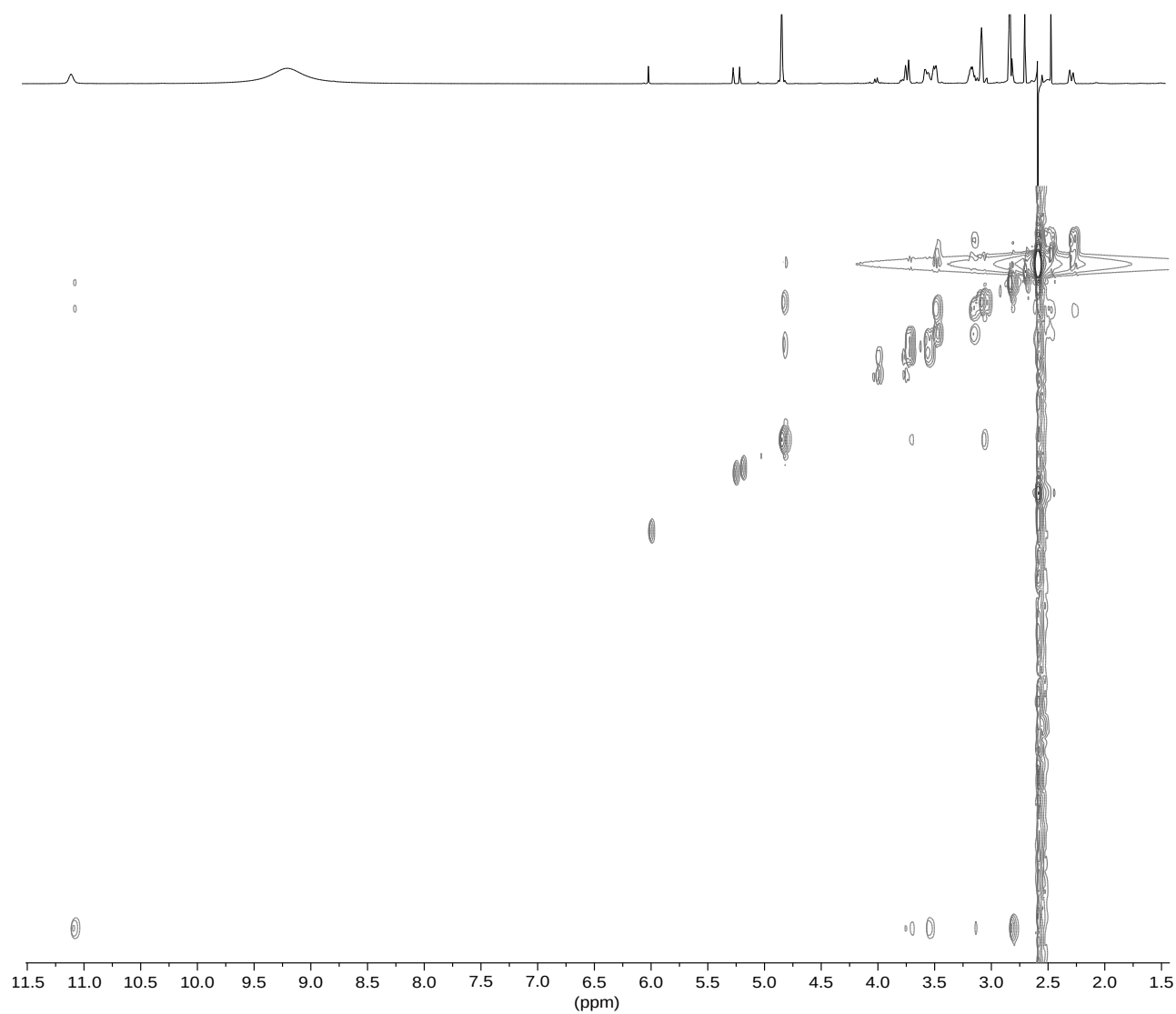
26

DMSO-*d*₆, 151 MHz





26
COSY



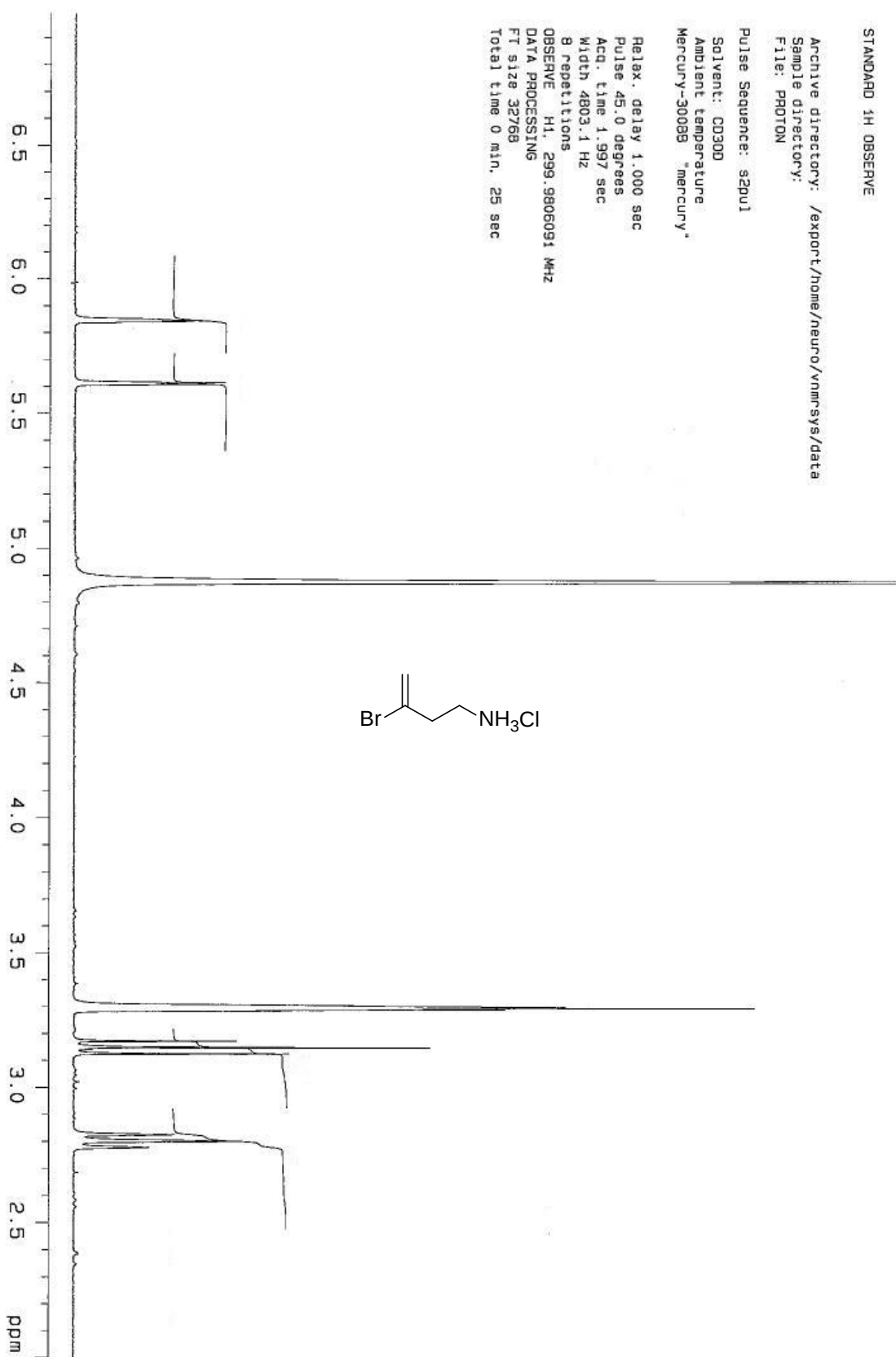
STANDARD 1H OBSERVE

Archive directory: /export/home/neuro/vnmr/ys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1

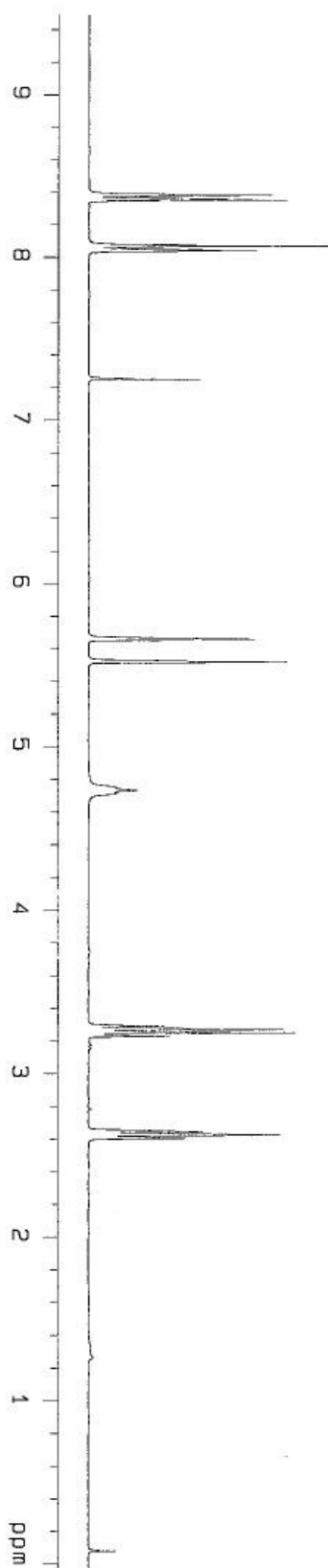
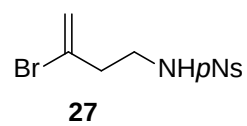
Solvent: CD3OD
Ambient temperature
Mercury-300088 "mercury"

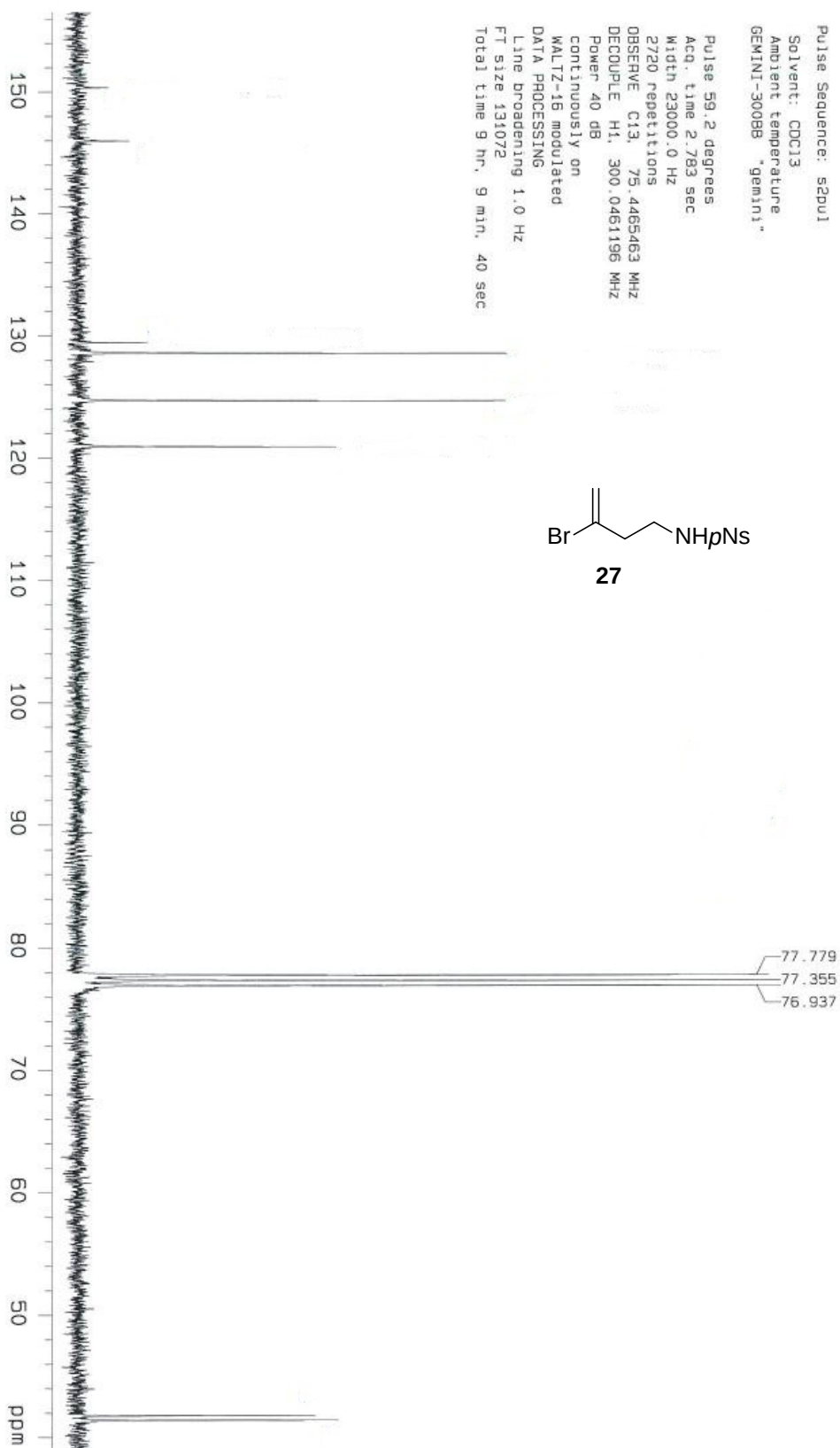
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.997 sec
Width 4803.1 Hz
8 repetitions
OBSERVE H1, 299.9806091 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: FCY_3
Mercury-300BB "mercury"

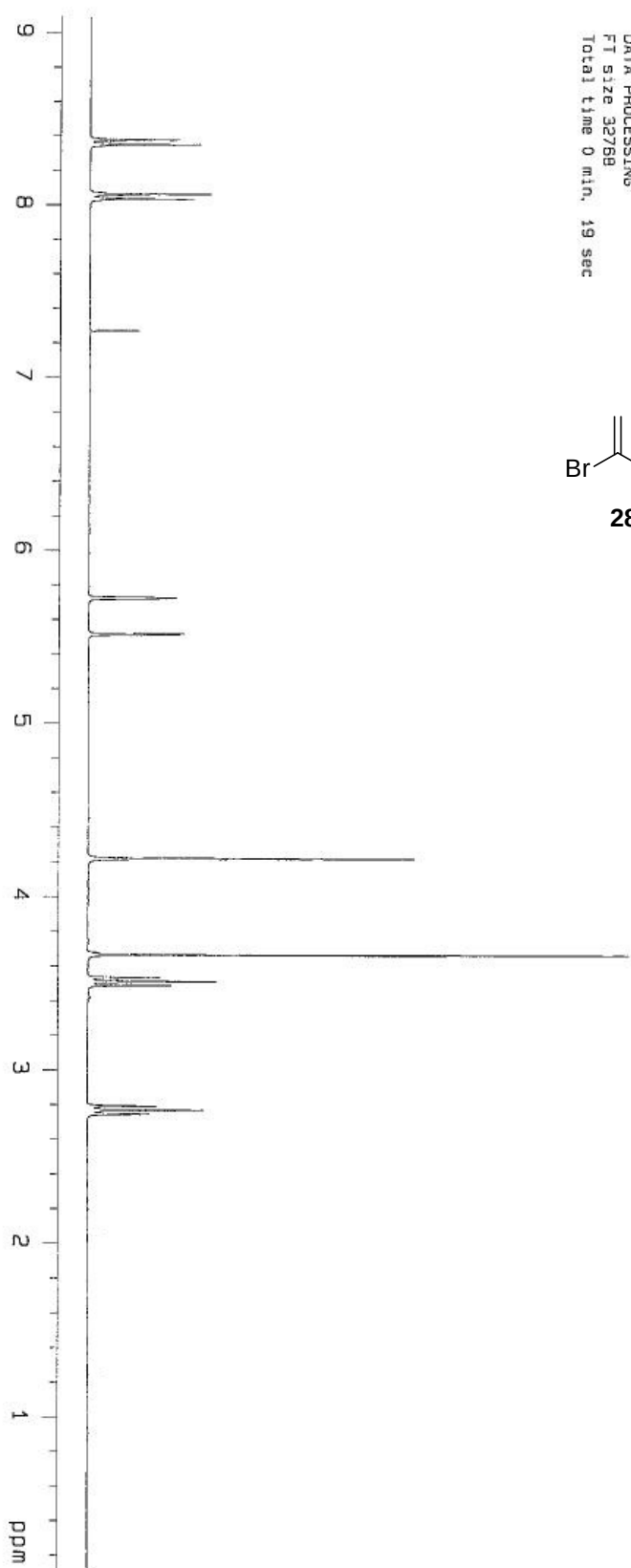
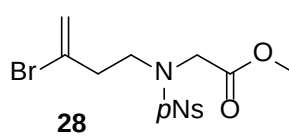
Relax. delay 1.000 sec
Pulse 71.6 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
6 repetitions
OBSERVE H1 299.9794235 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 19 sec

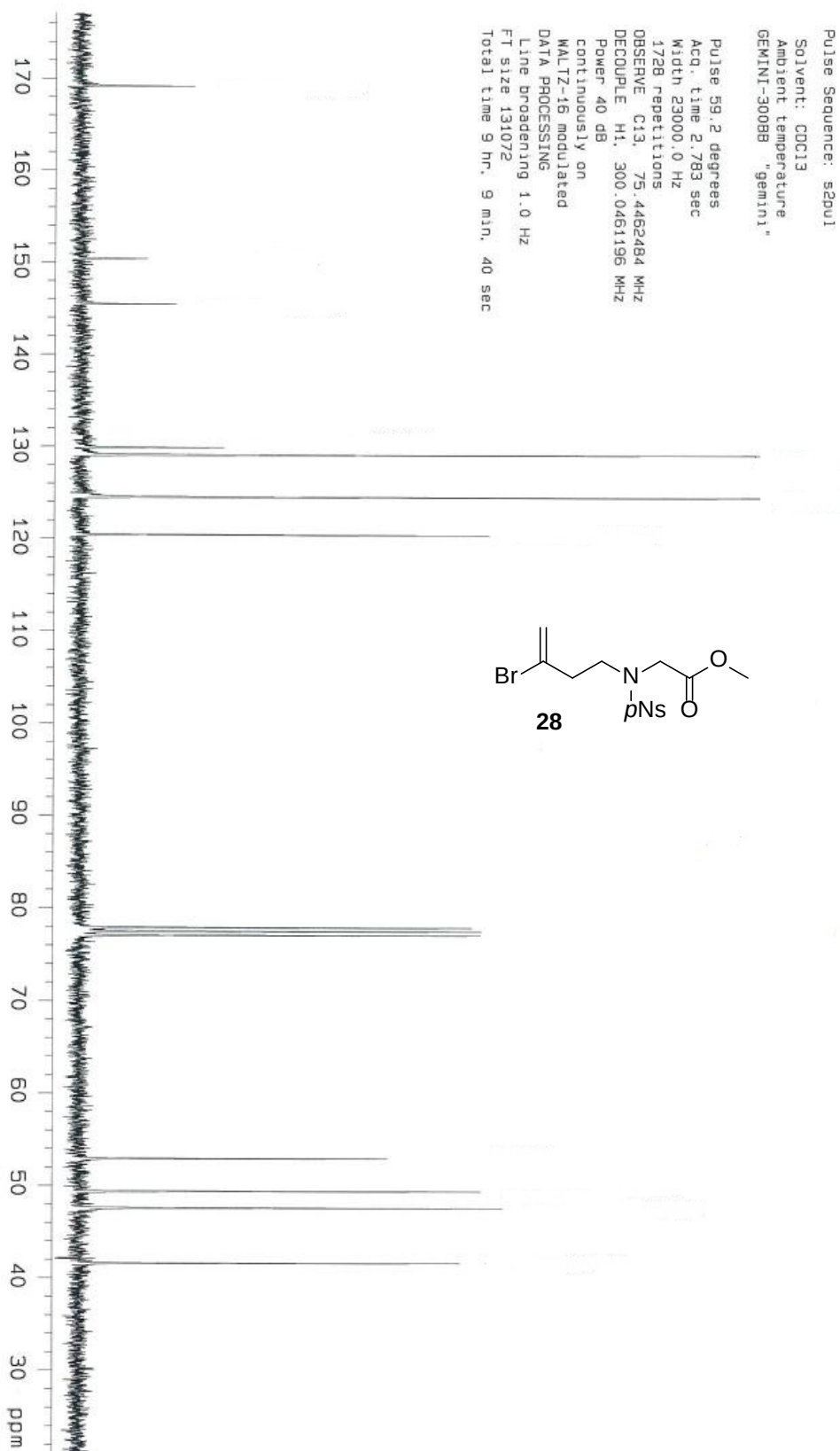




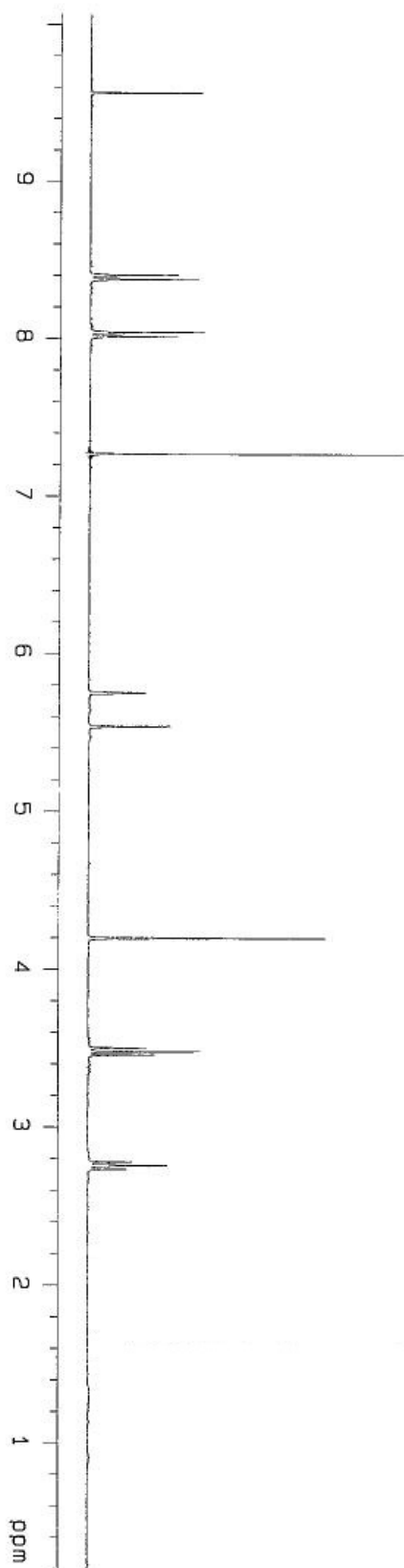
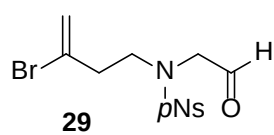
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: FCY_25
Mercury-300BBB "mercury"

Relax. delay 1.000 sec
Pulse 71.6 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
6 repetitions
OBSERVE H1, 299.9803953 MHz
DATA PROCESSING
F1 size 32768
Total time 0 min, 19 sec



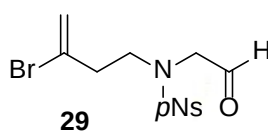


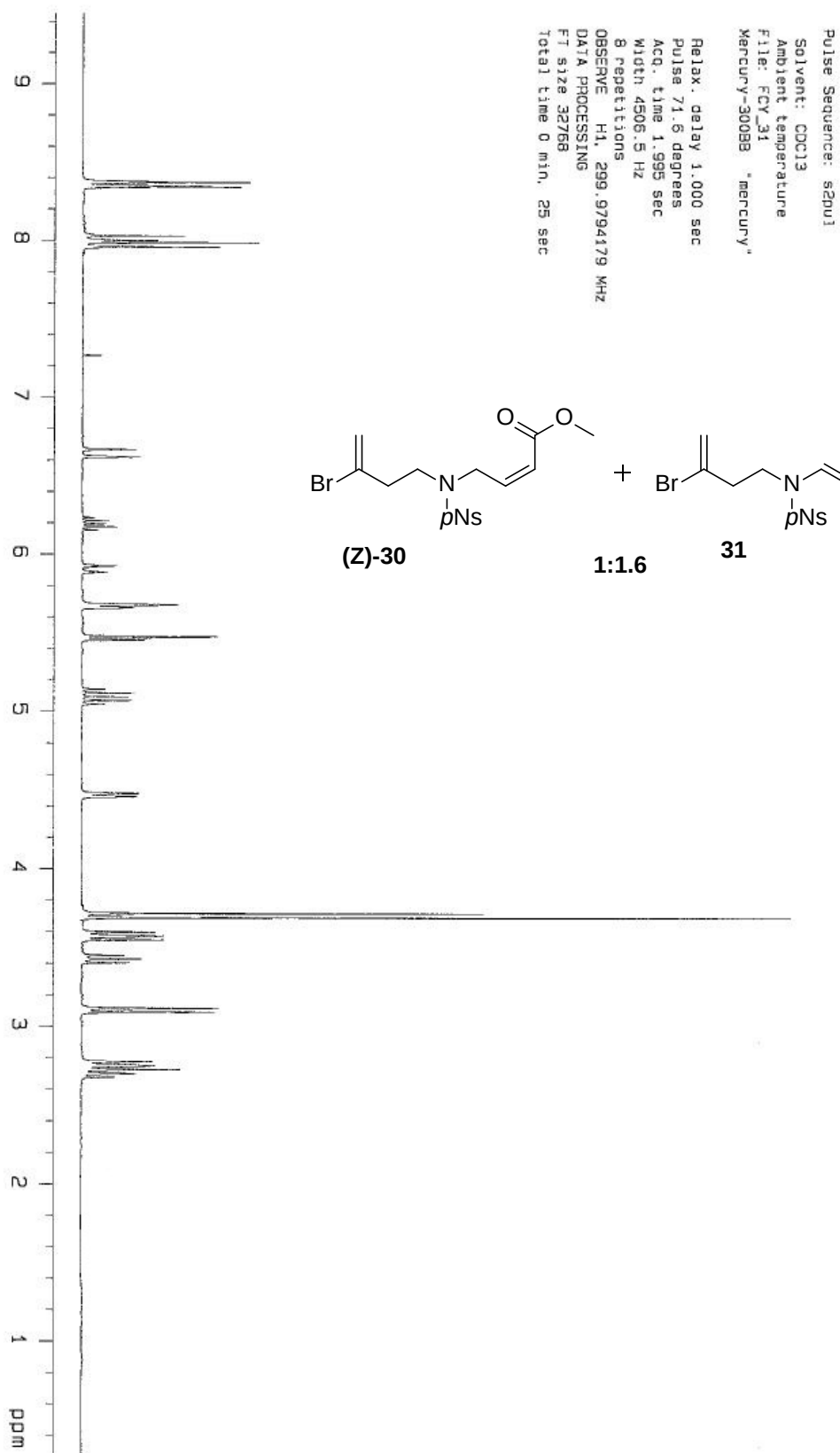
Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: FCY_33 "mercury"
 Mercury-300BB
 Relax. delay 1.000 sec
 Pulse 71.6 degrees
 Acq. time 1.995 sec
 Width 4506.5 Hz
 4 repetitions
 OBSERVE H1, 299.9794182 MHz
 DATA PROCESSING
 FT size 32768
 Total time 0 min, 13 sec



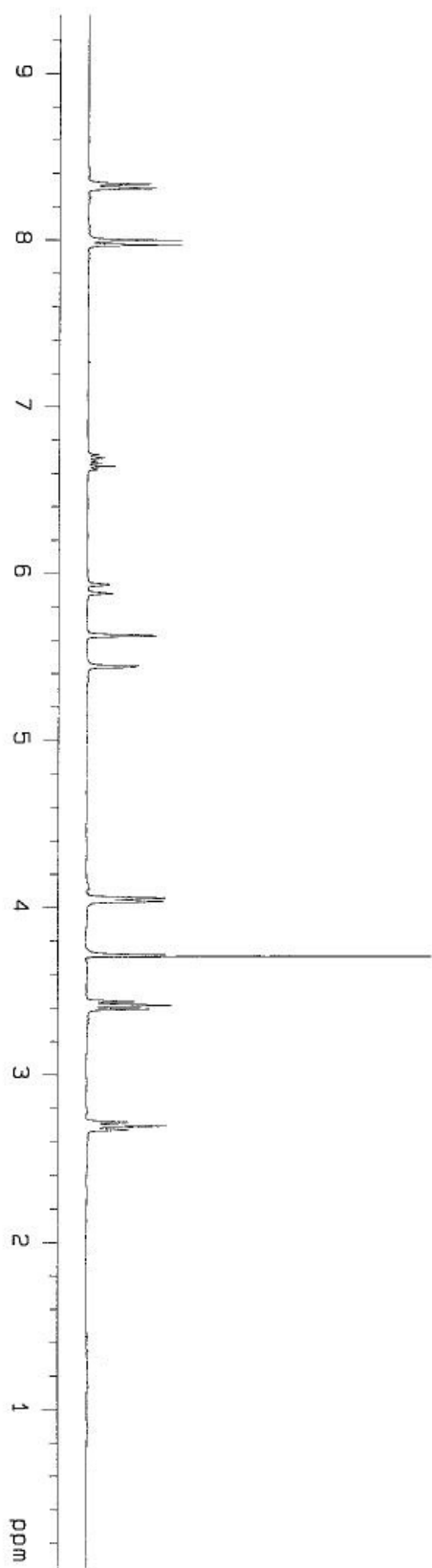
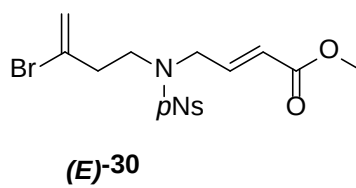
Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 GEMINI-3000B "gemin1"

Pulse 59.2 degrees
 Acq. time 2.783 sec
 Width 23000.0 Hz
 1536 repetitions
 OBSERVE C13, 75.462484 MHz
 DECOUPLE H1, 300.0461196 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 9 hr, 9 min, 40 sec



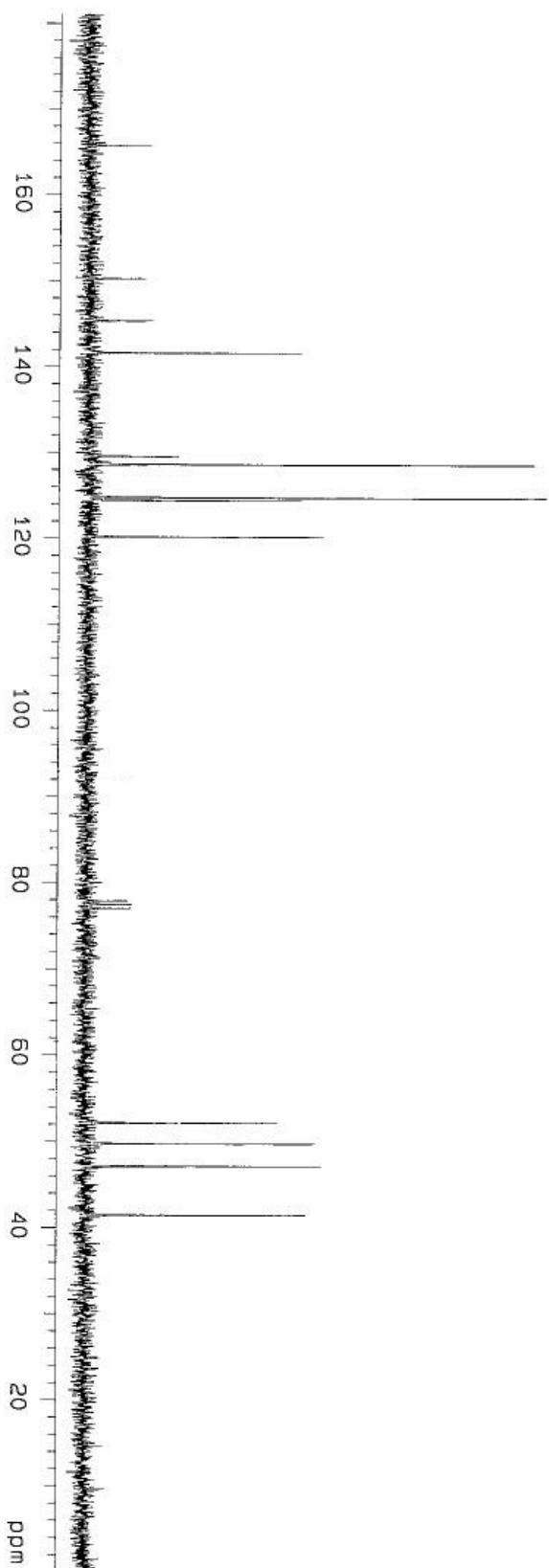
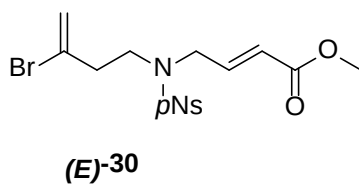


Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: FCY_53
 Mercury-300BB "mercury"
 Relax. delay 1.000 sec
 Pulse 71.6 degrees
 Acq. time 0.640 sec
 Width 100.0 kHz
 6 repetitions
 OBSERVE H1, 299.9806863 MHz
 DATA PROCESSING
 FT size 131072
 Total time 0 min, 17 sec

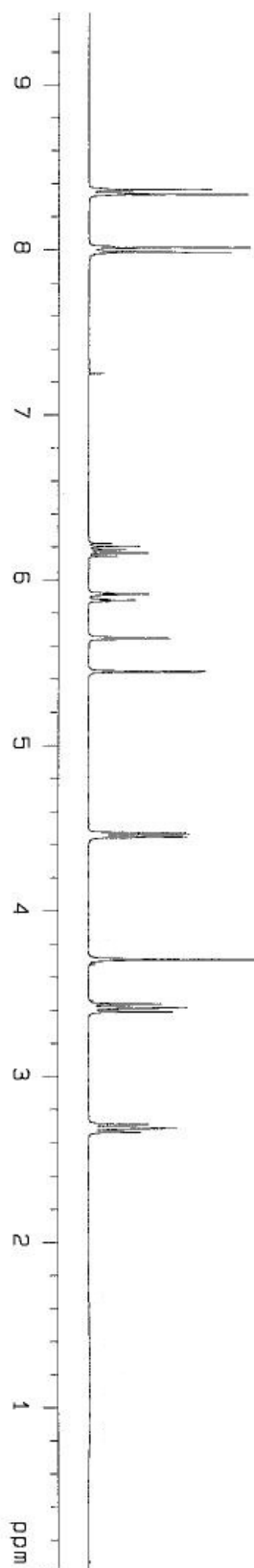
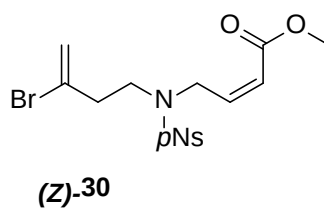


Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient temperature
 File: FGY_53_C13
 Mercury-300MR "mercury"

Pulse 55.7 degrees
 Acq. time 1.815 sec
 Width 18761.7 Hz
 192 repetitions
 OBSERVE C13, 75.4298680 MHz
 DECOUPLE H1, 299.9808873 MHz
 Power 35 dB
 continuously on
 MALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 5 hr, 52 min, 1 sec

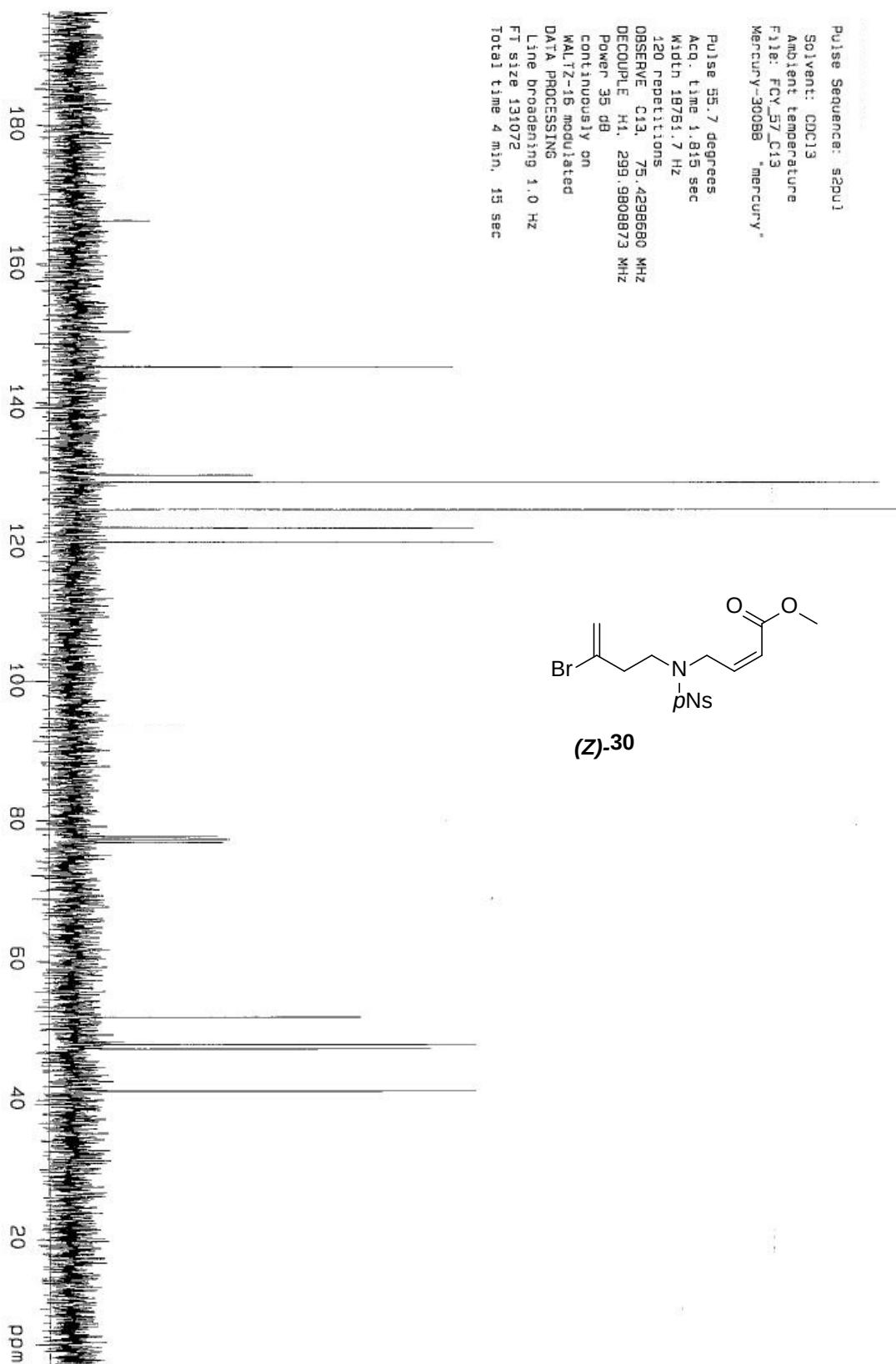


Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: FCY_57
 Mercury-300MB "mercury"
 Relax. delay 1.000 sec
 Pulse 71.6 degrees
 Acq. time 1.995 sec
 Width 4506.5 Hz
 8 repetitions
 OBSERVE H1, 299.9794235 MHz
 DATA PROCESSING
 FT size 32768
 Total time 0 min, 25 sec



Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
File: FCY_57_C13
Mercury-300BB "mercury"

Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18751.7 Hz
120 repetitions
OBSERVE C13, 75.4298680 MHz
DECOUPLE H1, 299.9808873 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 4 min, 15 sec



Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: F01_303

Mercury-300BB "mercury"

Relax. delay 1.000 sec

Pulse 71.6 degrees

Acq. time 1.985 sec

Width 4506.5 Hz

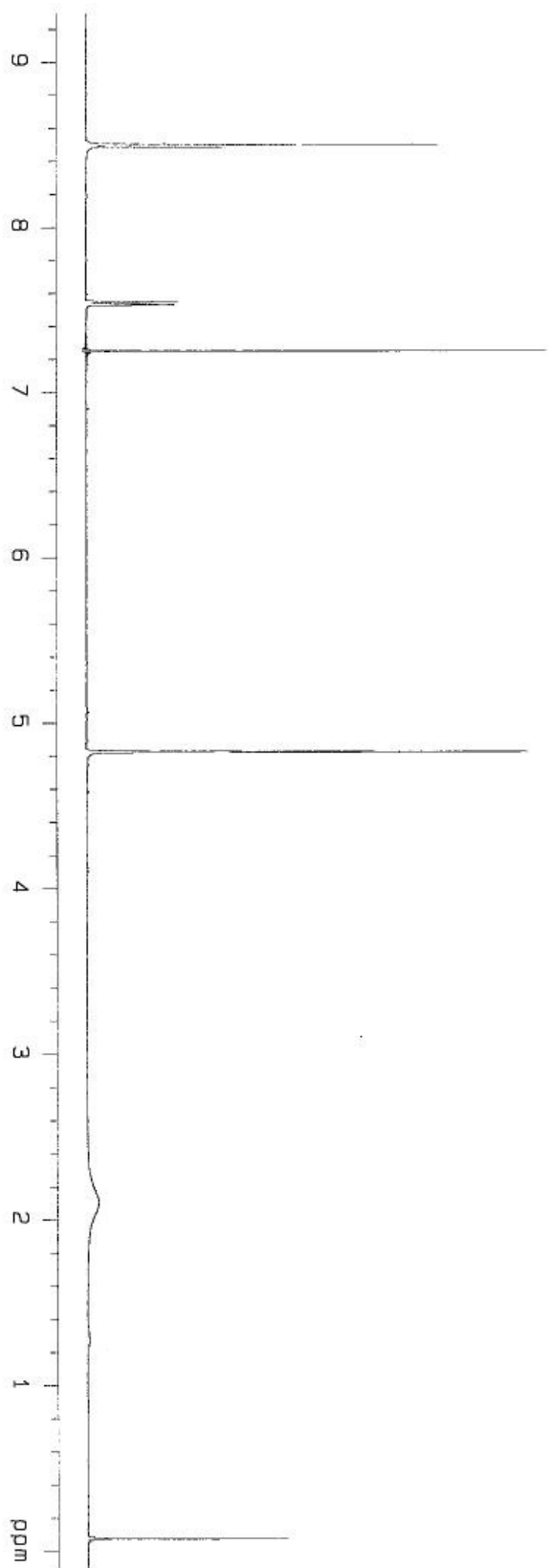
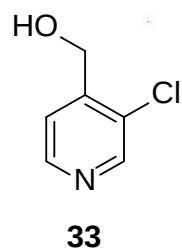
8 repetitions

OBSERVE H1 299.9794235 MHz

DATA PROCESSING

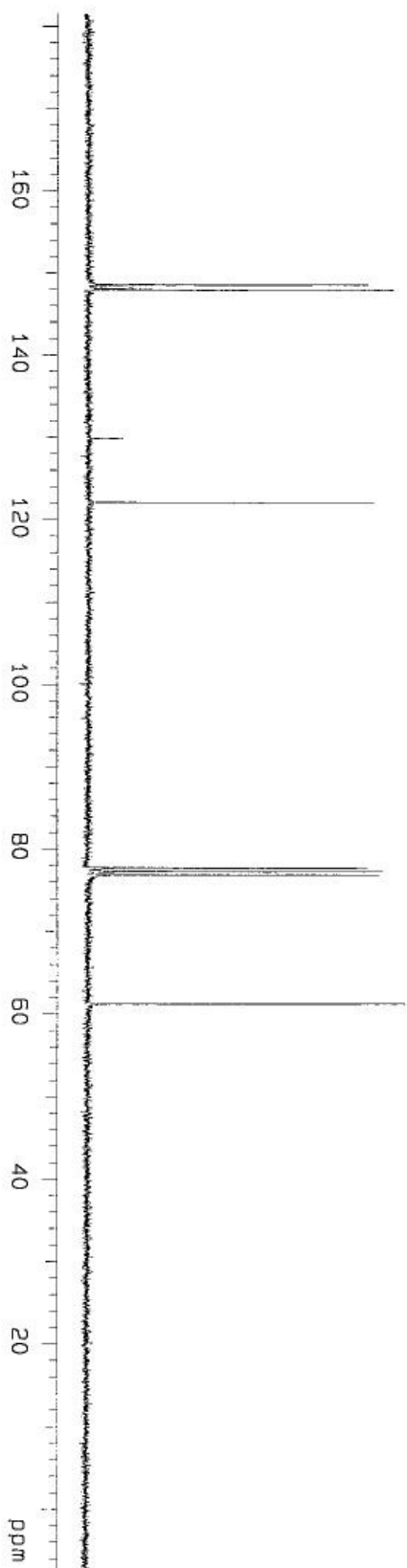
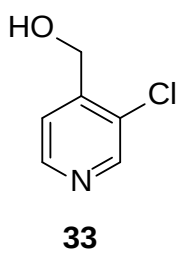
FT size 32768

Total time 0 min, 19 sec



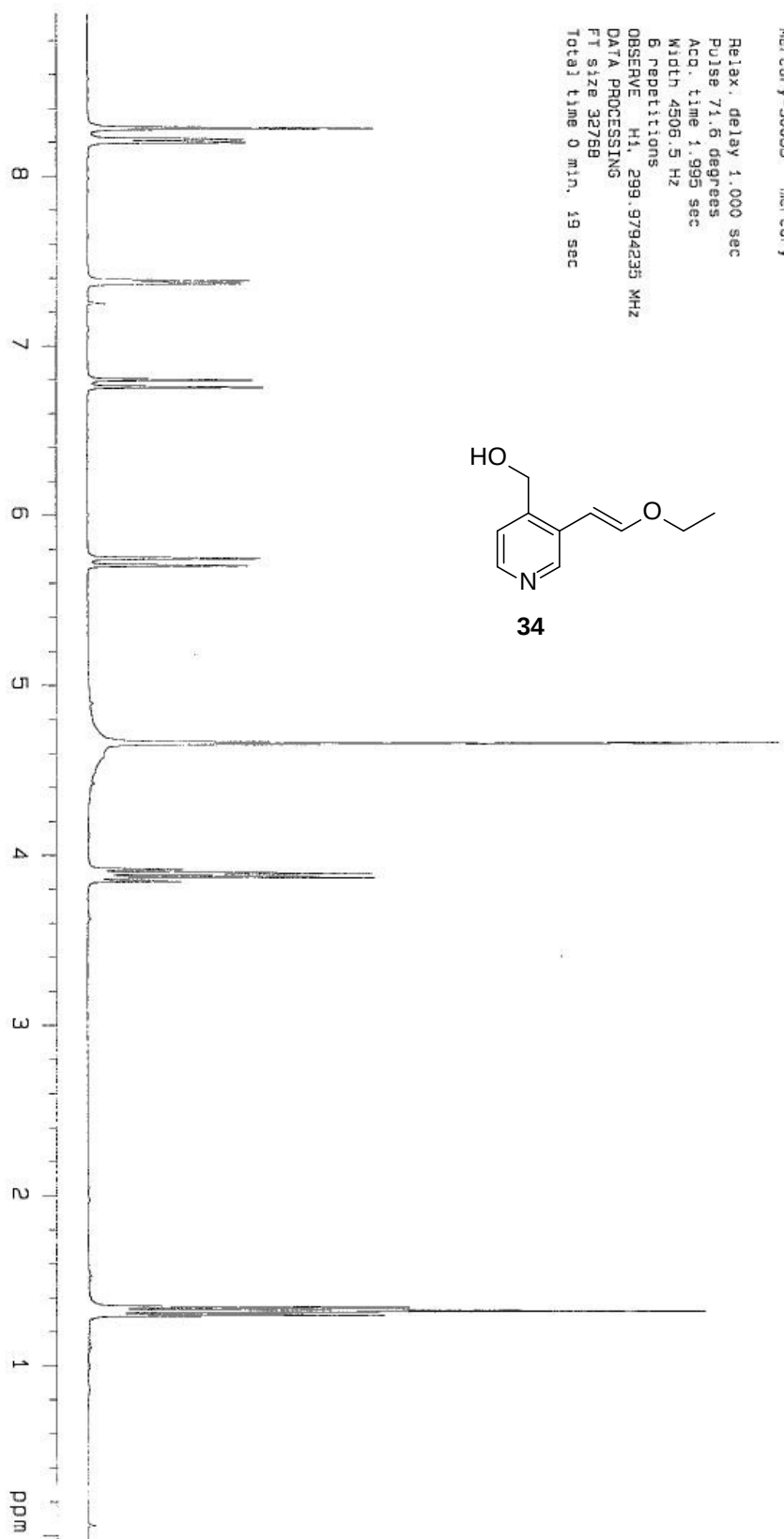
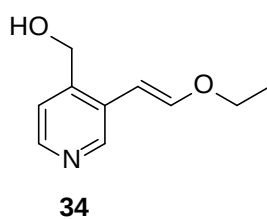
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: FCY_303_C13
Mercury-300NB "mercury"

Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
28496 repetitions
OBSERVE C13, 75.4298680 MHz
DECOUPLE H1, 299.9808873 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 23 hr, 28 min, 4 sec



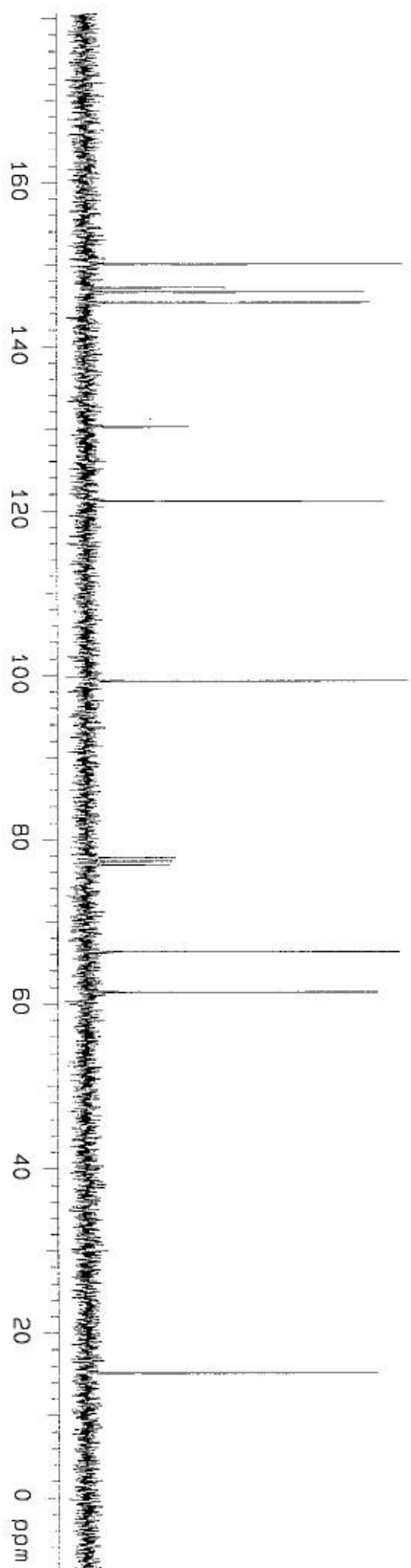
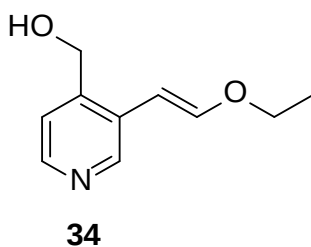
Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
File: F01_299
Mercury-3000B "mercury"

Relax. delay 1.000 sec
Pulse 71.6 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
6 repetitions
OBSERVE H1 299.9794235 MHz
DATA PROCESSING
F1 size 32768
Total time 0 min. 19 sec



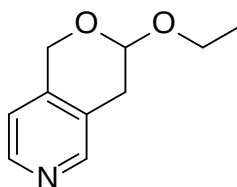
Pulse Sequence: s2puj
 Solvent: CDCl3
 Ambient temperature
 File: PCY_299_C13
 Mercury-30058 "mercury"

Pulse 55.7 degrees
 Acq. time 1.815 sec
 Width 18761.7 Hz
 144 repetitions
 OBSERVE C13, 75.4298680 MHz
 DECOUPLE H1, 299.9808873 MHz
 Power 35 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 11 hr, 44 min, 2 sec

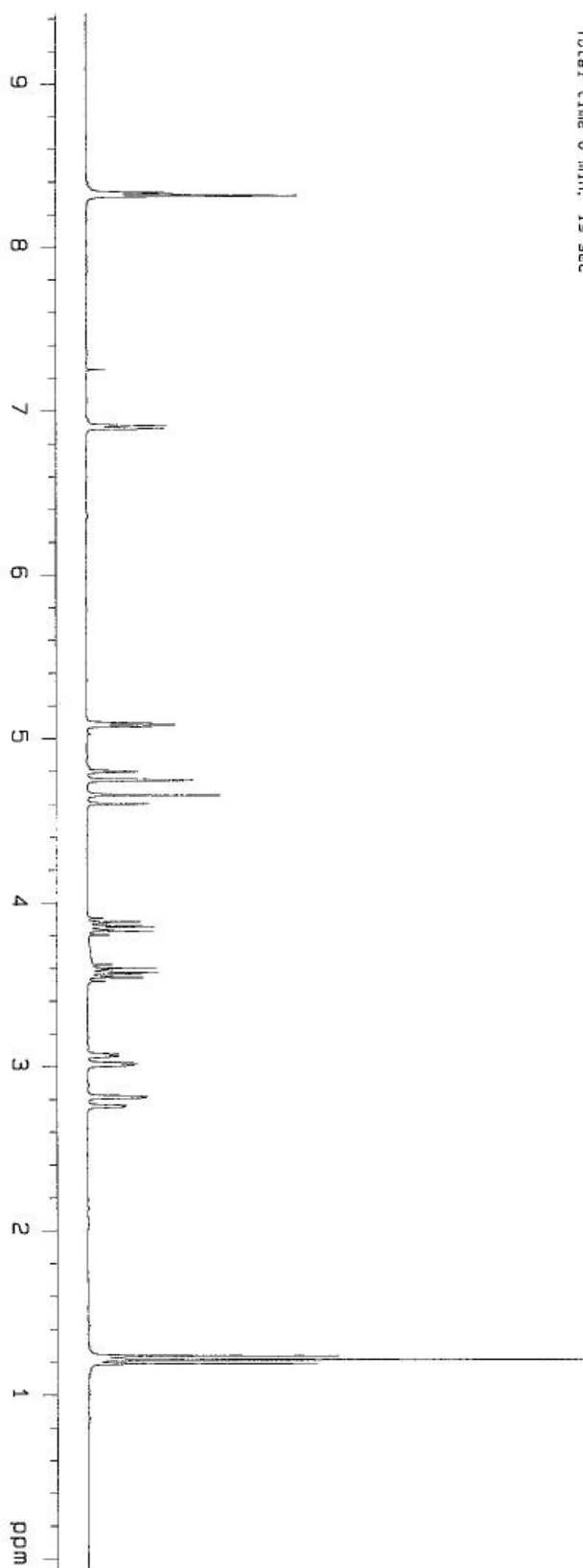


Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
File: FCY_308_Crude
Mercury-300088 "mercury"

Relax. delay 1.000 sec
Pulse 71.6 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
6 repetitions
OBSERVE H1, 299.9794235 MHz
DATA PROCESSING
F1 size 32768
Total time 0 min, 19 sec

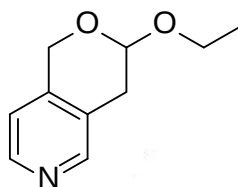


35

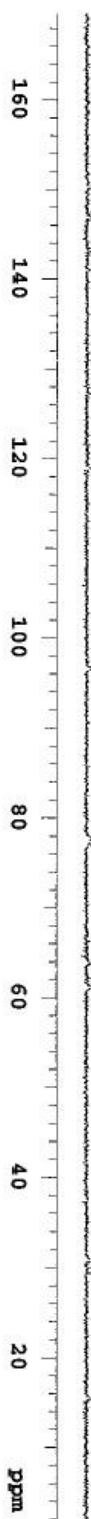


Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
GEMINT-300MB "gemint"

Pulse 59.2 degrees
Acq. time 2.783 sec
Width 23000.0 Hz
1056 repetitions
OBSERVE C13, 75.446103 MHz
DECOUPLE H1, 300.039342 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FM size 131072
Total time 18 hr, 19 min, 20 sec

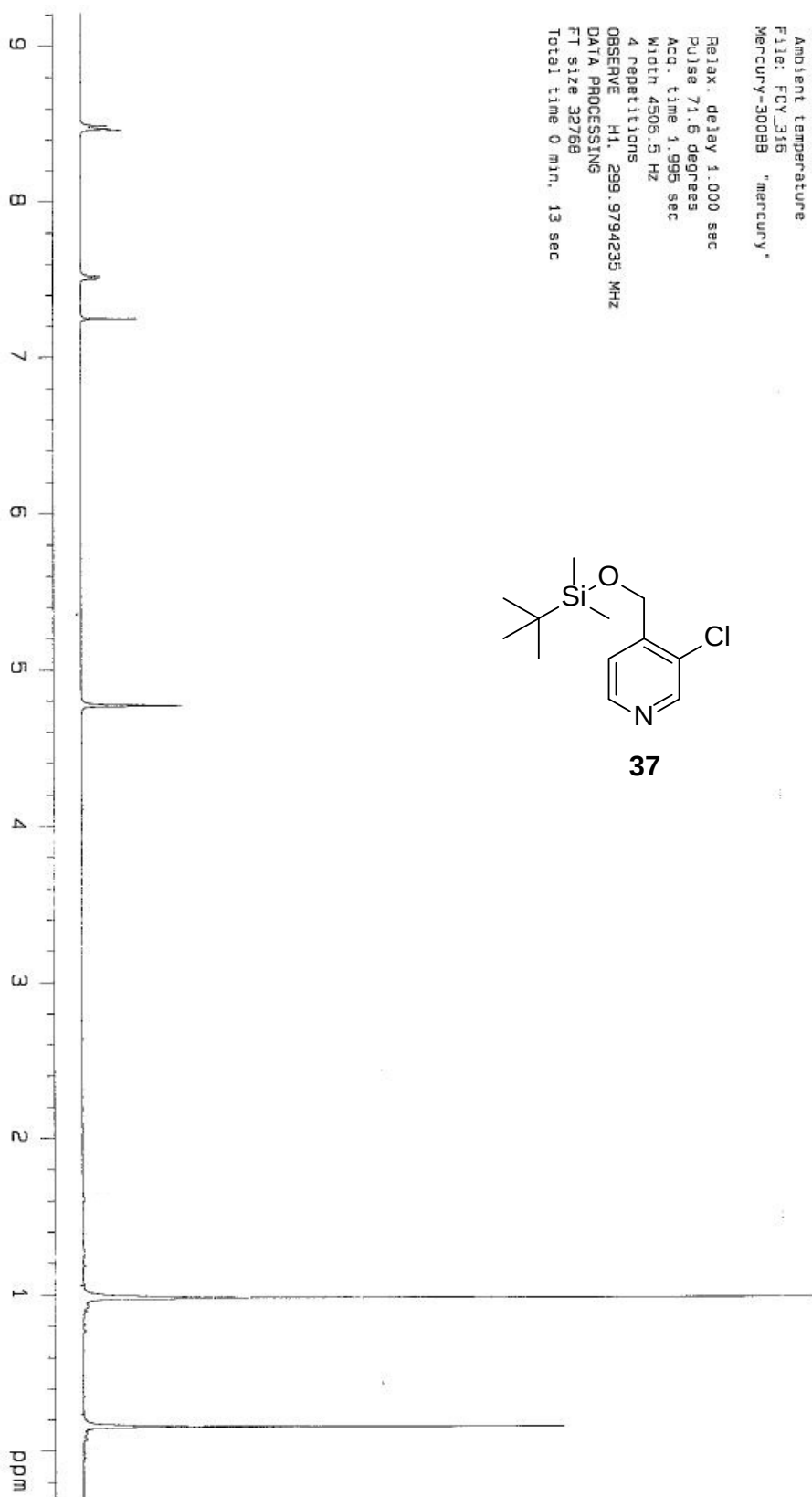
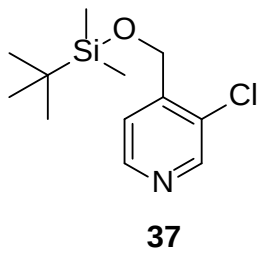


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Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
File: FCY_316
Mercury-300MR "mercury"

Relax. delay 1.000 sec
Pulse 71.6 degrees
Acq. time 1.995 sec
Width 4505.5 Hz
4 repetitions
OBSERVE H1, 299.9794235 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 13 sec



Pulse Sequence: zgpg30

Solvent: CDCl₃

Ambient temperature

GENIUM-300BB "gemini"

Pulse 59.2 degrees

Acq. time 2.783 sec

Width 23604.0 Hz

960 repetitions

OBSERVE: CH- 75.444103 MHz

DECOUPLE: H1, 300.0395342 MHz

Power 40 dB

continuously on

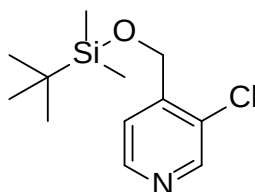
WALTZ-16 modulated

DATA PROCESSING

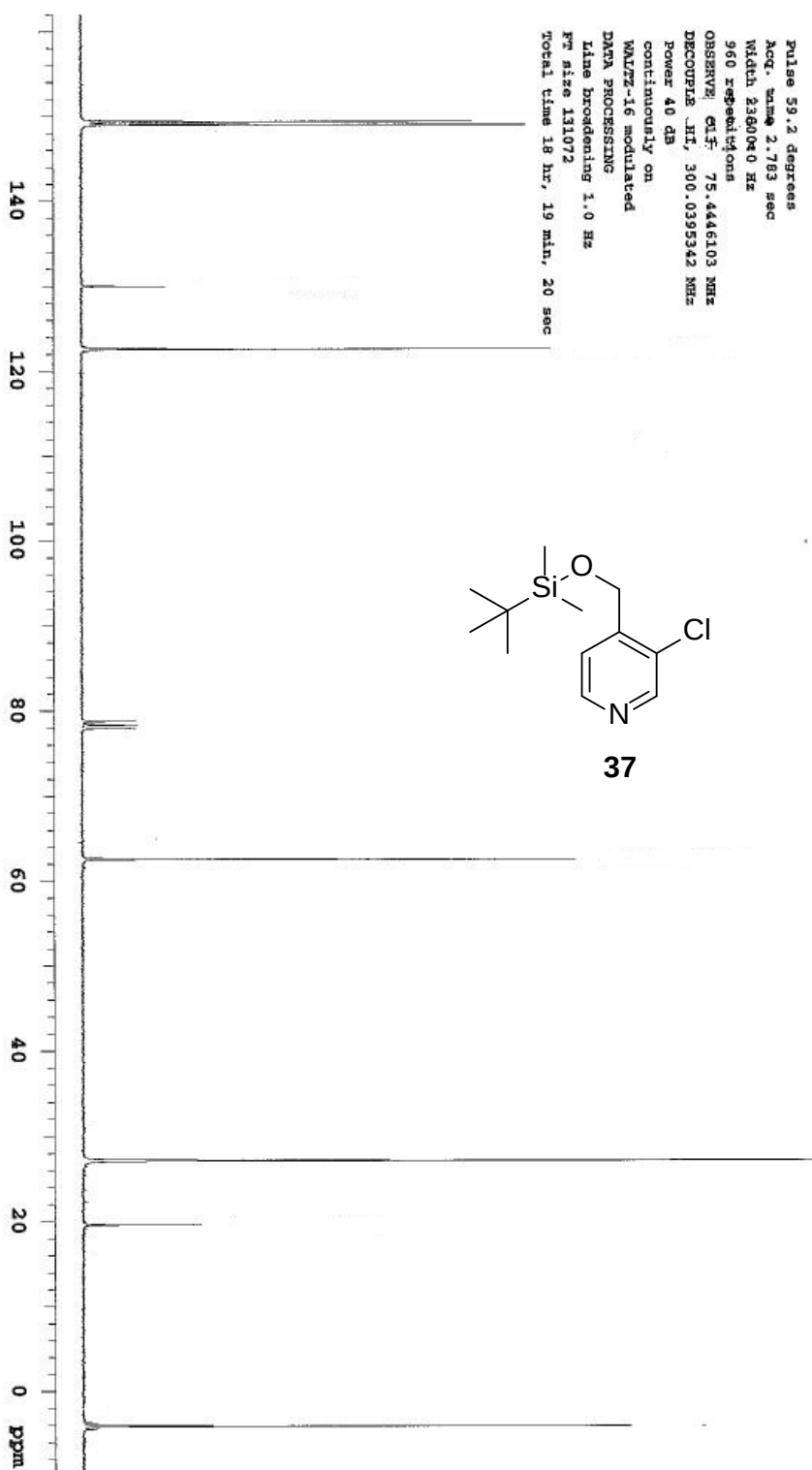
line broadening 1.0 Hz

FT size 131072

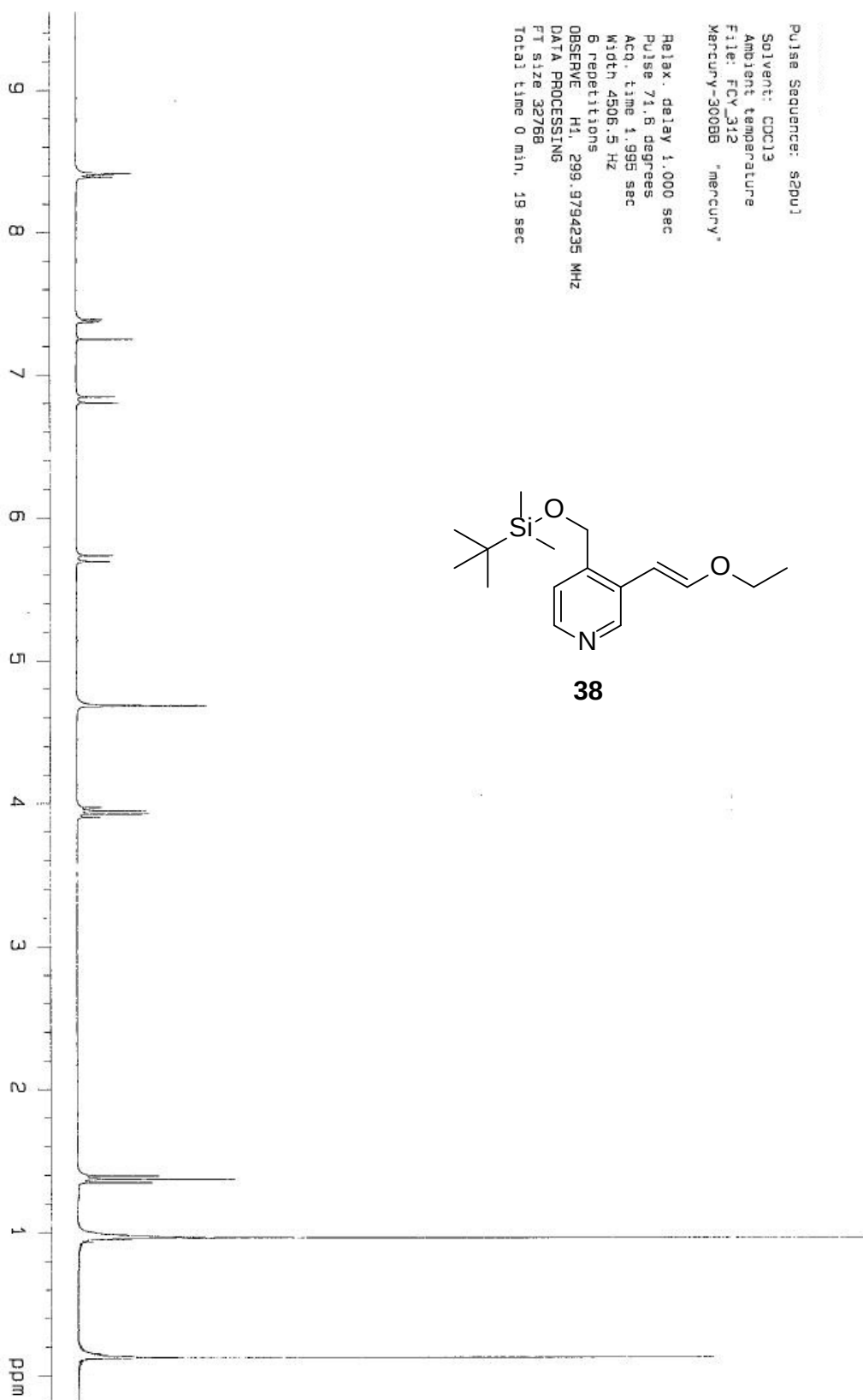
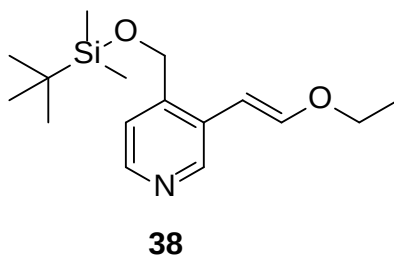
Total time 18 hr, 19 min, 20 sec



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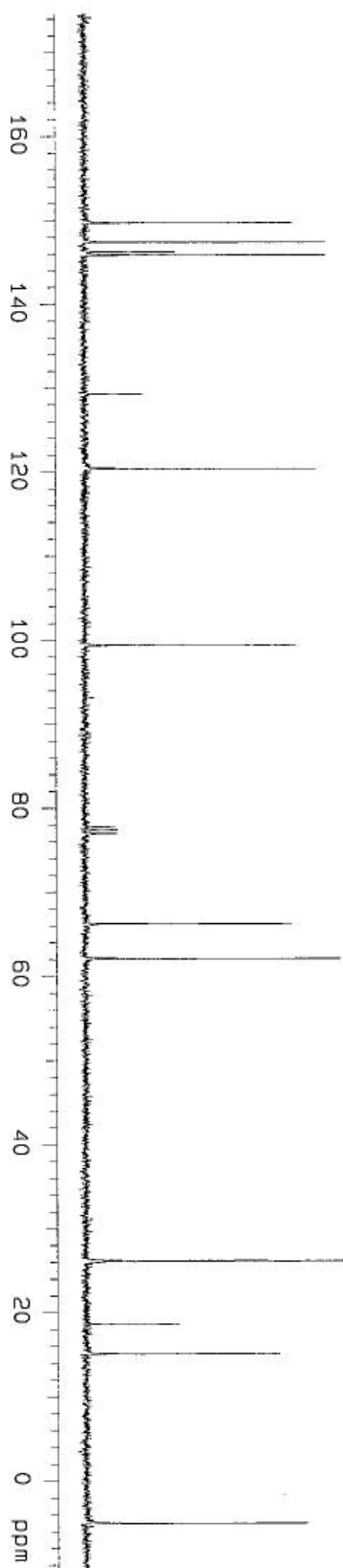
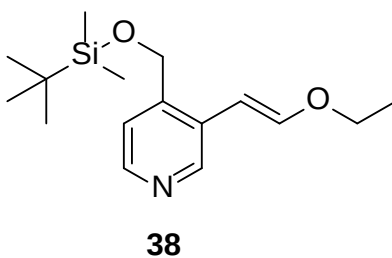


Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: FCY_312 "mercury"
 Mercury-300BBB
 Relax. delay 1.000 sec
 Pulse 71.6 degrees
 Acq. time 1.995 sec
 Width 4506.5 Hz
 6 repetitions
 OBSERVE H1: 299.9794235 MHz
 DATA PROCESSING
 FT size 32768
 Total time 0 min. 19 sec



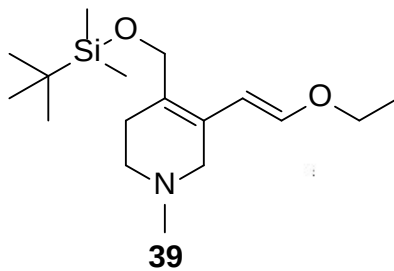
Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient temperature
 File: FCY_312_C13
 Mercury-300MB "mercury"

Pulse 55.7 degrees
 Acq. time 1.815 sec
 Width 18761.7 Hz
 400 repetitions
 OBSERVE C13: 75.4298680 MHz
 DECOUPLE H1: 299.9808873 MHz
 Power 35 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 117 hr, 20 min, 24 sec



Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: FCY_422
 Mercury-300BB "mercury"

Relax. delay 1.000 sec
 Pulse 71.6 degrees
 Acq. time 1.995 sec
 Width 4506.5 Hz
 6 repetitions
 OBSERVE H1, 299.9794235 MHz
 DATA PROCESSING
 FT size 32768
 Total time 0 min, 19 sec



Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: FCY_422_C13
 Mercury-300BB "mercury"

Pulse 55.7 degrees
 Acq. time 1.815 sec
 Width 18761.7 Hz
 28960 repetitions
 OBSERVE C13, 75.4296680 MHz
 DECOUPLE H1, 299.9808873 MHz
 Power 35 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 117 hr, 20 min, 24 sec

