Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

A dual biomimetic process for the selective aerobic oxidative coupling of primary amines using pyrogallol as a precatalyst. Isolation of the [5+2] cycloaddition redox intermediates

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I. Materials and methods

All reagents were purchased from commercial suppliers, Acros Organics, Alfa Aeser Sigma Aldrich or TCI europe, and used without further purification. Column chromatography was performed on silica gel 60 particle size 40-63 micron from Sigma Aldrich using the solvent system as stated. Mass spectra were recorded on a Q-ToF1 spectrometer, equipped with the negative electrospray mode (ES-). ^{1}H NMR and 1D proton decoupled ^{13}C NMR spectra were recorded on a Bruker Avance I spectrometer operating at 300 MHz and 75 MHz, respectively. Chemical shifts δ are given in ppm relative to TMS and coupling constants *J*, in Hertz. The measurements were carried out using the standard pulse-sequences. The carbon type (methyl, methylene, methine, or quaternary) was determined by DEPT experiments. 2D experiments (HSQC and HMBC) were performed on a 400 MHz Bruker Avance III spectrometer or on a 600 MHz Bruker Avance I spectrometer using standard pulse sequences.

All cross-coupled imines are known: imine **24a**,[1] imine **24b**,[2] imine **24c**,[2] imine **24d**,[2] imine **24e**,[3] imine **24f**,[3] imine **24g**,[3] imine **24h**,[3] imine **24i**,[4] imine **24j**,[2] imine **24k**,[3] imine **24l**,[3,5] imine **24m,**[2] imine **24n**,[6] imine **24o**.[7]

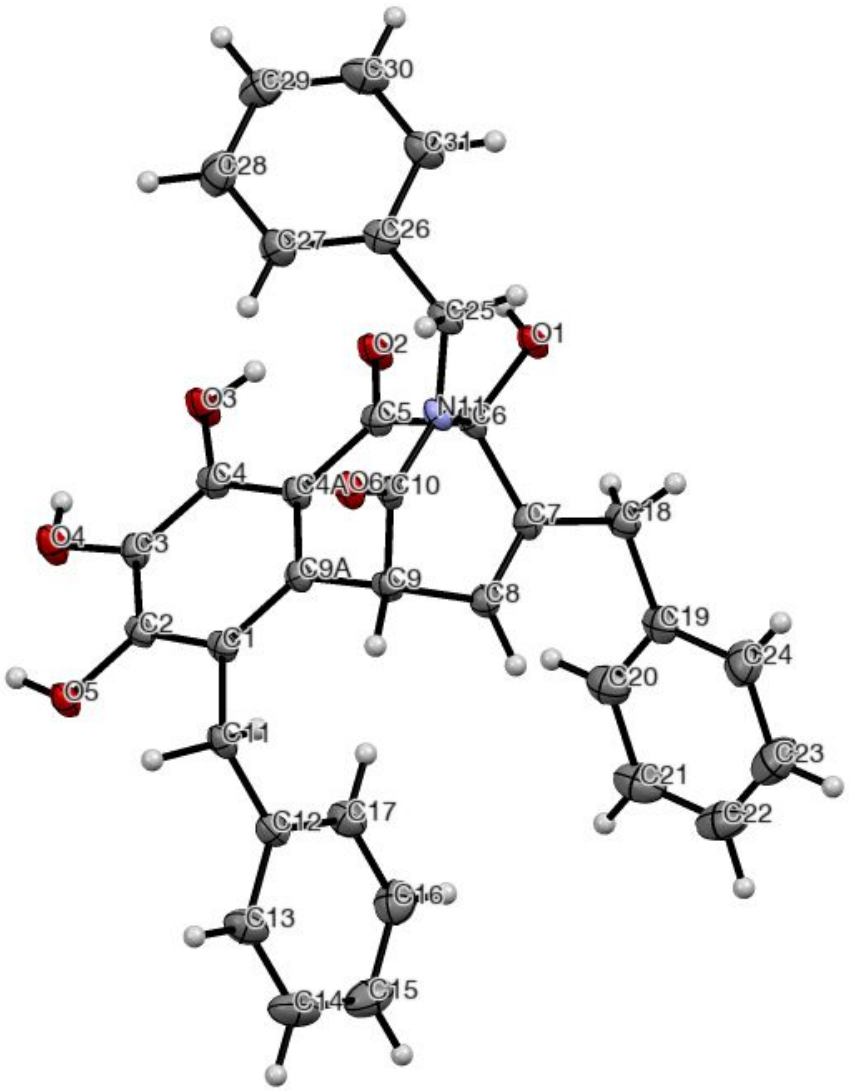
All 1,2-substituted benzimidazoles are known: benzimidazole **25a**,[8] benzimidazole **25b**,[9] benzimidazole **25c**,[9] benzimidazole **25d**,[8] benzimidazole **25e**,[8] benzimidazole **25f**,[9] benzimidazole **25g**,[3] benzimidazole **25h**,[9] benzimidazole **25i**,[9] benzimidazole **25j**,[9] benzimidazole **25k**,[9] benzimidazole **25l**,[9] benzimidazole **25m**,[9] benzimidazole **25n**,[9] benzimidazole **25o**.[9]

II. Crystallographic studies

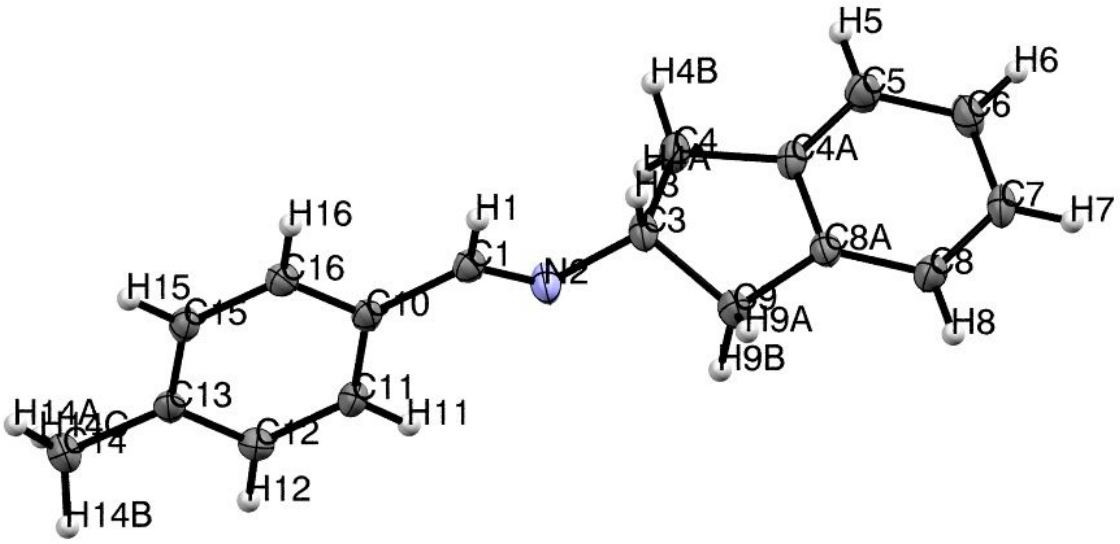
Data were collected by using a VENTURE PHOTON100 CMOS Bruker diffractometer (Cu-Kα radiation) controlled by APEX3 software package.[10] Data integration and global cell refinement were performed with the program SAINT.[11] Data were corrected for absorption by the multiscan semiempirical method implemented in SADABS.[12] The structure was solved by direct methods using SHELXS-97.[13]

Refinement, based on F^2, was carried out by full matrix least squares with SHELXL-2008 software.[14] All non hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms were located on a difference Fourier map and placed in their geometrically generated positions and allowed to ride on their parent atoms with an isotropic thermal parameter 20 % higher to that of the atom of attachment.

The crystal data collection and refinement parameters are given in Table S1. CCDC 1965257, CCDC 1981805, CCDC 1981802 contain the supplementary crystallographic data of compounds **19red**, **24j** and **25d,** respectively for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/Community/Requestastructure.



19red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C6
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C8
H15
C15
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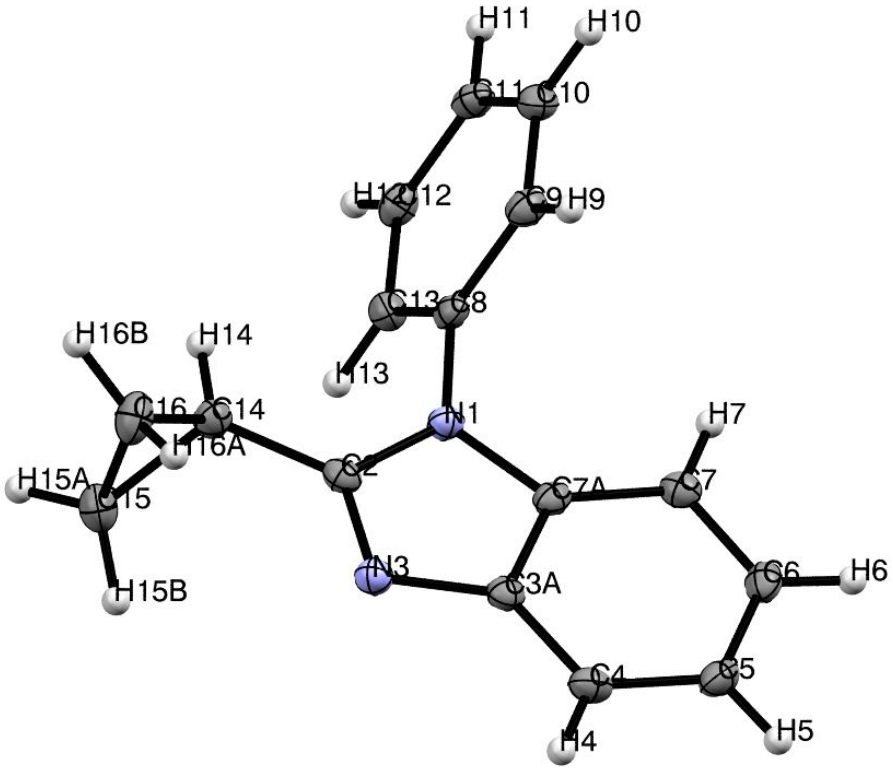
H14B

24j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C9
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H16A
C2
H15A
C7A
C7
C15
N3
C3A
C6
H6
H15B
C4
C5
H5
H4

25d

Table S1 - Crystal data and structure refinements for **19red**, **24j** and **25d**

Compounds	**19d**	**24j**	**25d**
Formula	$C_{33}H_{27}NO_6$	$C_{17}H_{17}N$	$C_{16}H_{14}N_2$
CCDC	1965257	1981805	1981802
Fw	533.55	235.31	234.29
T(K)	100(2)	100(2)	100(2)
Radiation type ; wavelength (Å)	Cu $K\alpha$; 1.54178	Cu $K\alpha$; 1.54178	Cu $K\alpha$; 1.54178
crystal system	triclinic	triclinic	triclinic
space group	P -1	P -1	P -1
unit cell dimension			
a (Å)	12.6327(6)	5.6742(2)	10.3880(4)
b (Å)	15.2434(7)	8.7921(4)	10.8621(4)
c (Å)	15.9800(7)	13.5713(5)	11.9356(4)
α (°)	102.027(2)	103.259(1)	103.368(1)
β (°)	105.432(2)	95.099(1)	111.316(1)
γ (°)	112.595(2)	95.624(1)	90.267(1)
Cell volume *V* (Å^3)	2566.4(2)	651.48(4)	1214.77(8)
Z	4	2	4
d(calc) (Mg/m^3)	1.381	1.200	1.281
abs coeff μ (mm^{-1})	0.776	0.526	0.591
crystal size (mm^3)	0.27 × 0.22× 0.05	0.23 × 0.19× 0.04	0.21 × 0.16× 0.10
crystal color	yellow	colorless	colorless
F_{000}	1120	252	496
θ range [deg]	3.061 - 66.744	3.368 - 63.997	4.106 – 59.042
index ranges	-15<h<15	-6<h<6	-11<h<11
	-18<k<18	-10<k<10	-12<k<12
	-19<l<19	-15<l<15	-13<l<13
no. of reflns collected	77250	21496	29618
no. of unique reflns	9064	2166	3491
R(int)	0.0269	0.0223	0.0147
GOF on F^2	1.011	1.072	1.099
Reflns obs. [I>2σ(I)]	7943	1995	3328
Parameters	743	166	325
R1/wR2[a,b],[I>2σ(I)]	0.0375/0.0998	0.0439/0.1096	0.0314/0.0776
R1/wR2[a,b], all data	0.0427/0.1045	0.0464/0.1122	0.0327/0.0784

largest diff peak and hole [e.Å^{-3}]	0.237 and -0.245	0.357 and -0.351	0.121 and -0.243

[a]R1 = Σ||*Fo*| – |*Fc*||/Σ|*Fo*|. [b] wR2 = {Σ[w($F_O^2 - F_c^2$)^2]/ Σ [w(F_O^2)2]} ; where w = $q/\sigma^2(F_O^2) + (qp)^2 + bp$. GOF = S = {Σ[w($F_O^2 - F_c^2$)^2]/ (n – p)$^{1/2}$}.

III. References

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9 K. M. H Nguyen and M. Largeron, *Eur. J.* Org. Chem. 2016, **2016**, 1025.

10 *APEX3. Data Collection Software*, Bruker AXS Inc.: Madison, Wisconsin, USA, 2007.

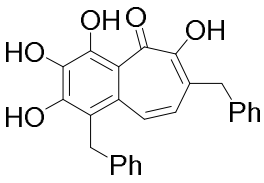
11 Bruker *SAINT*, Bruker AXS Inc: Madison, Wisconsin, USA, 2007.

12 *SADABS.2008/1*, Bruker AXS Inc.: Madison, Wisconsin, USA, 2007.

13 Sheldrick, G. M. *SHELXS-97, Program for crystal structure solution*, University of Gottingen, Germany, 1997.

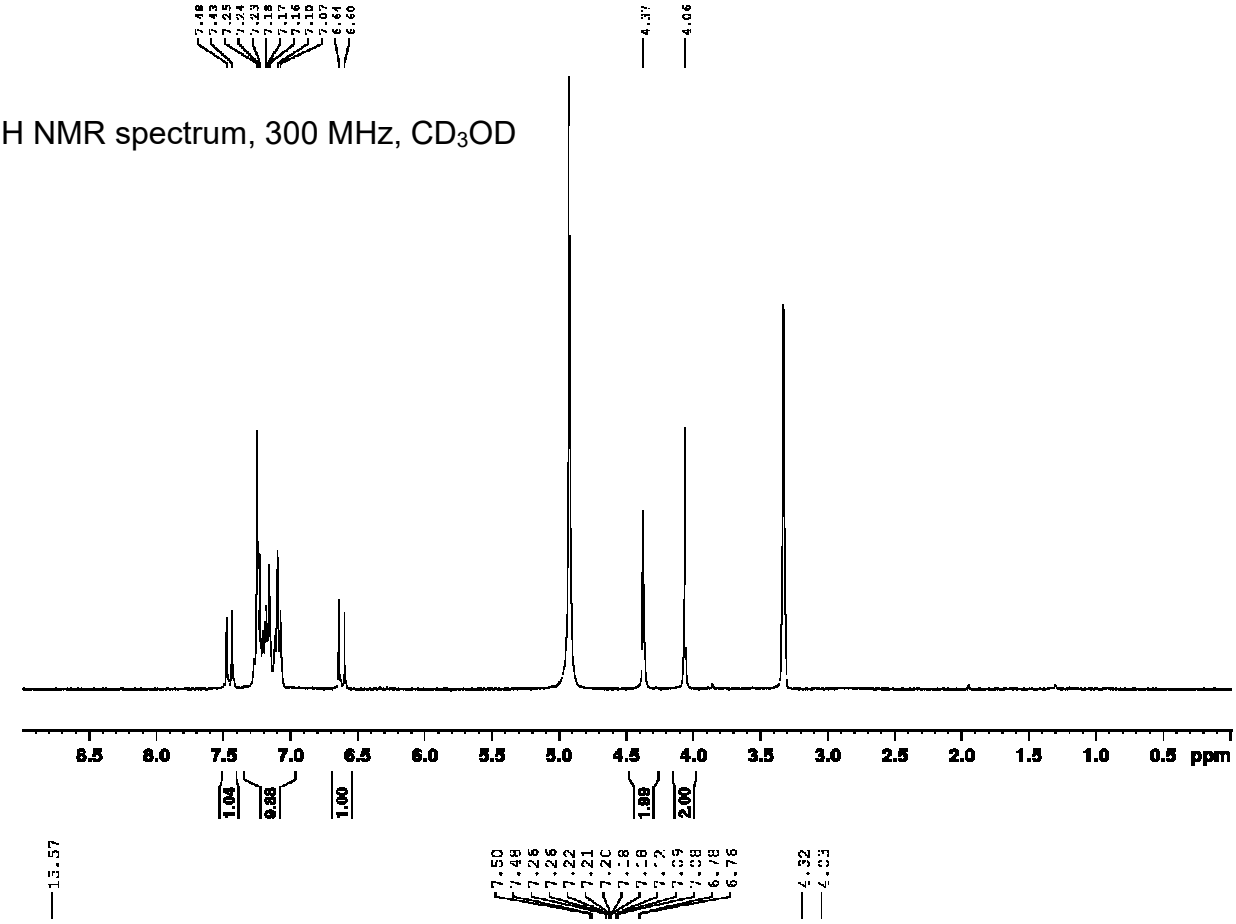
14 Sheldrick, G. M. *SHELXL-2008, Acta Crystallogr., Sect. A: Found. Crystallogr.* 2008, **64**, 112-122.

IV.High field ^{1}H NMR spectra and 1D proton-decoupled ^{13}C NMR spectra for all isolated products 18red, 19red, 24a-o and 25a-o. 2D NMR spectra (HMBC and HSQC) for compounds 18red and 19red. (see next page)



18red

^{1}H NMR spectrum, 300 MHz, CD_3OD



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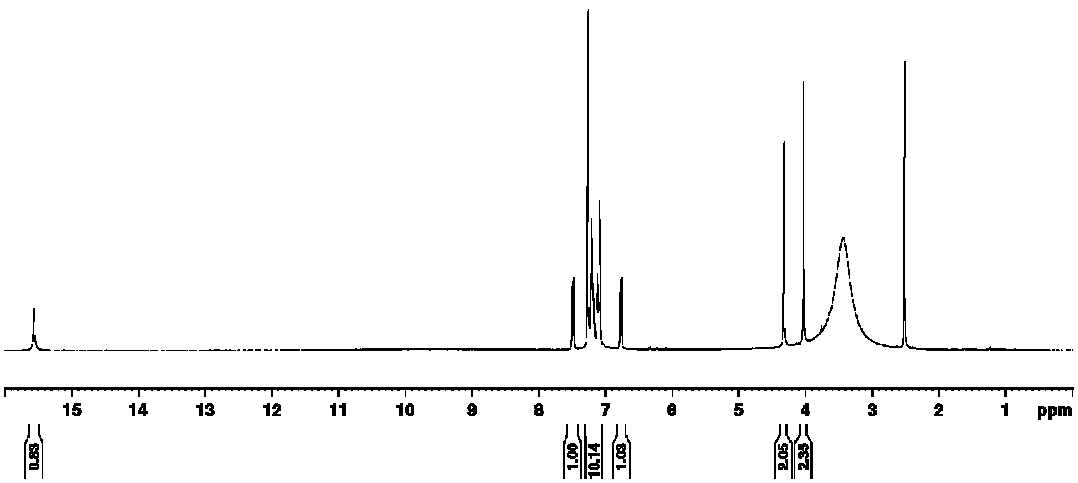
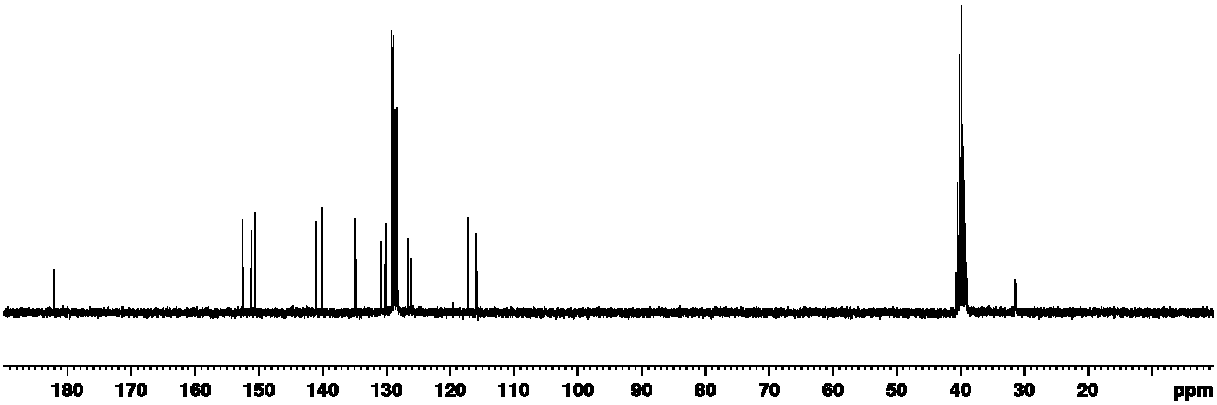


1D proton-decoupled ^{13}C NMR spectrum, 75 MHz, DMSO D_6



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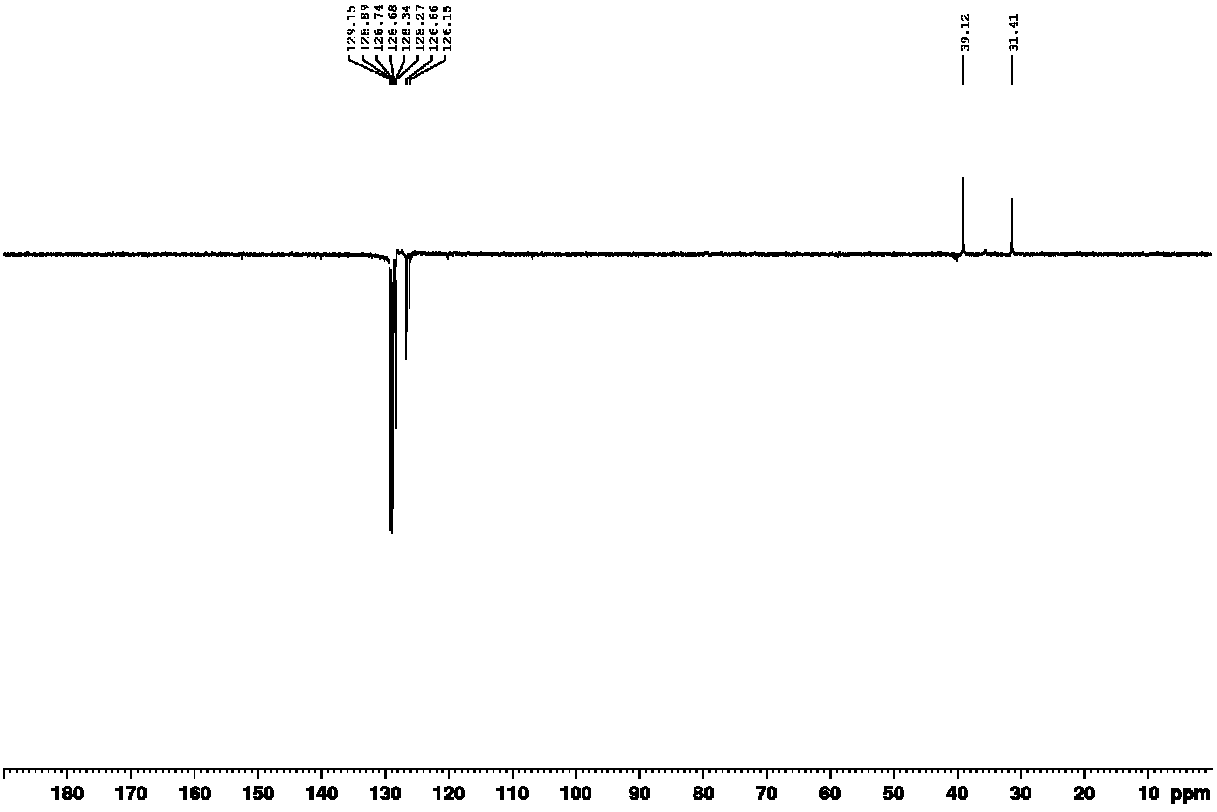

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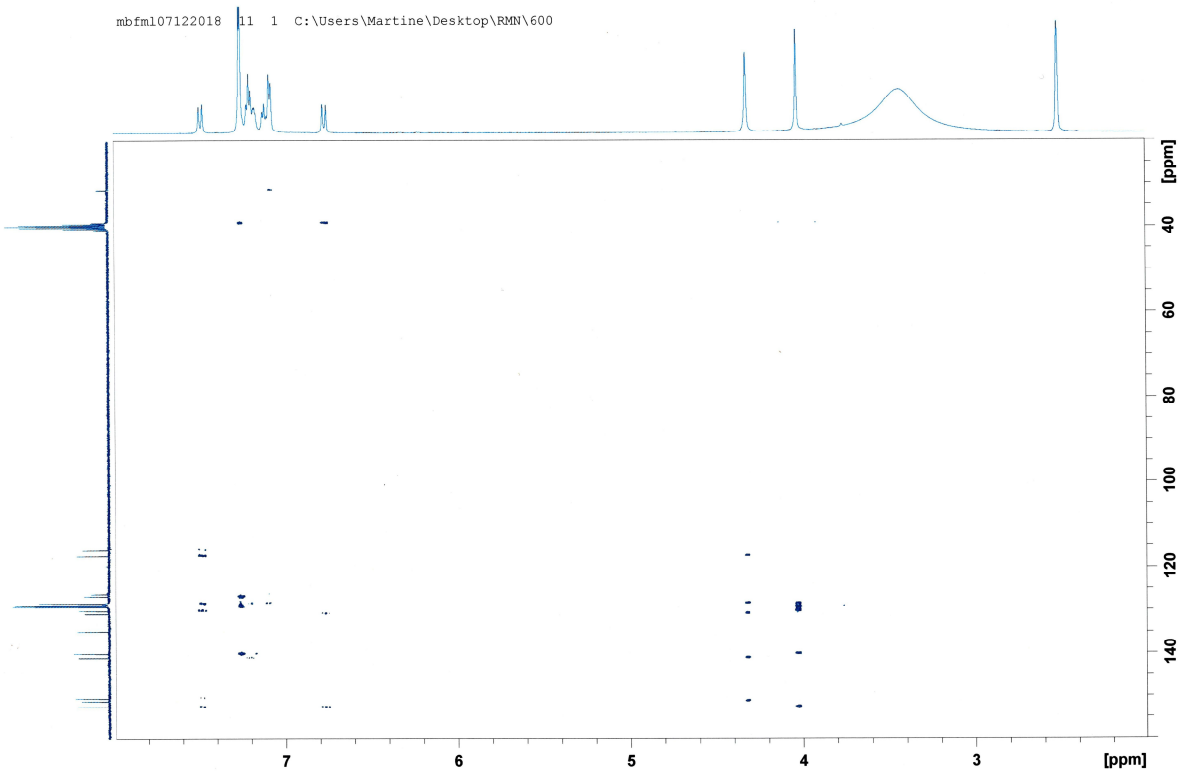

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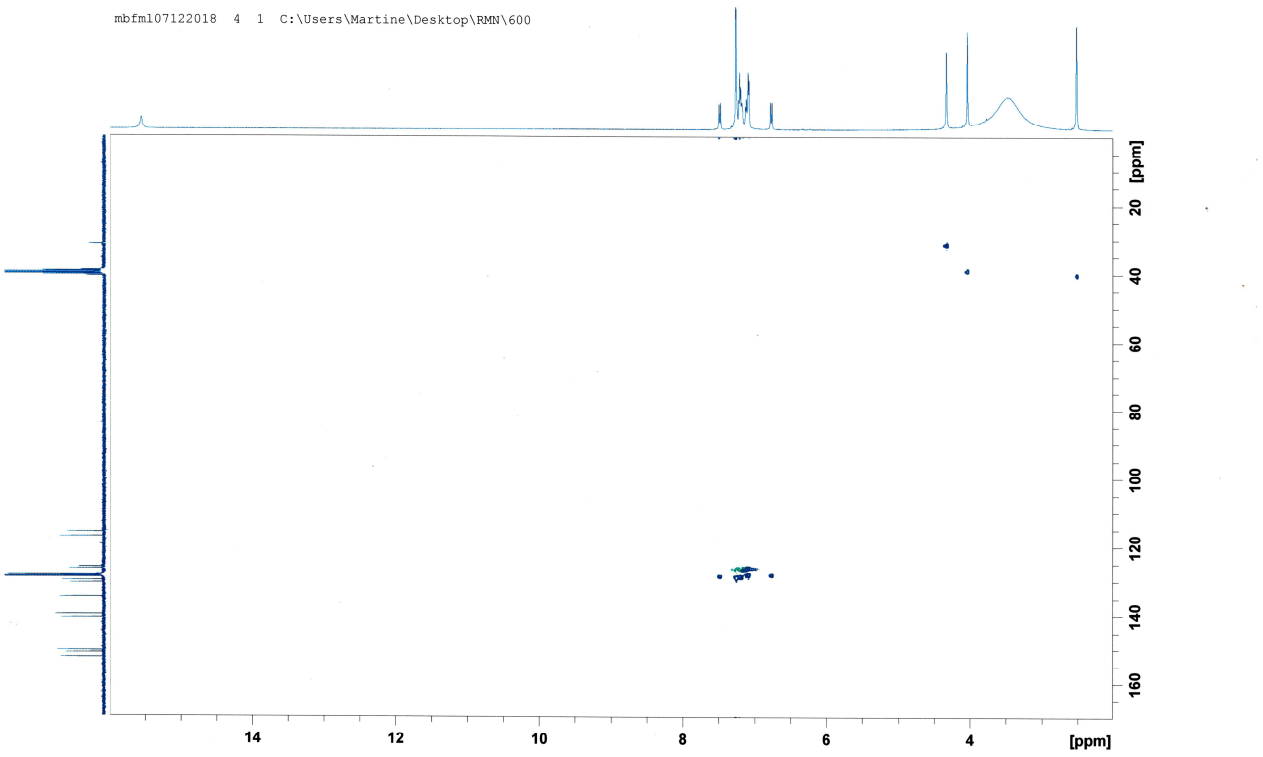
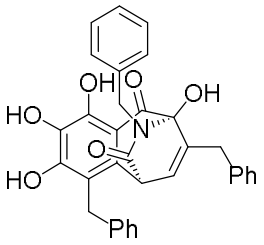
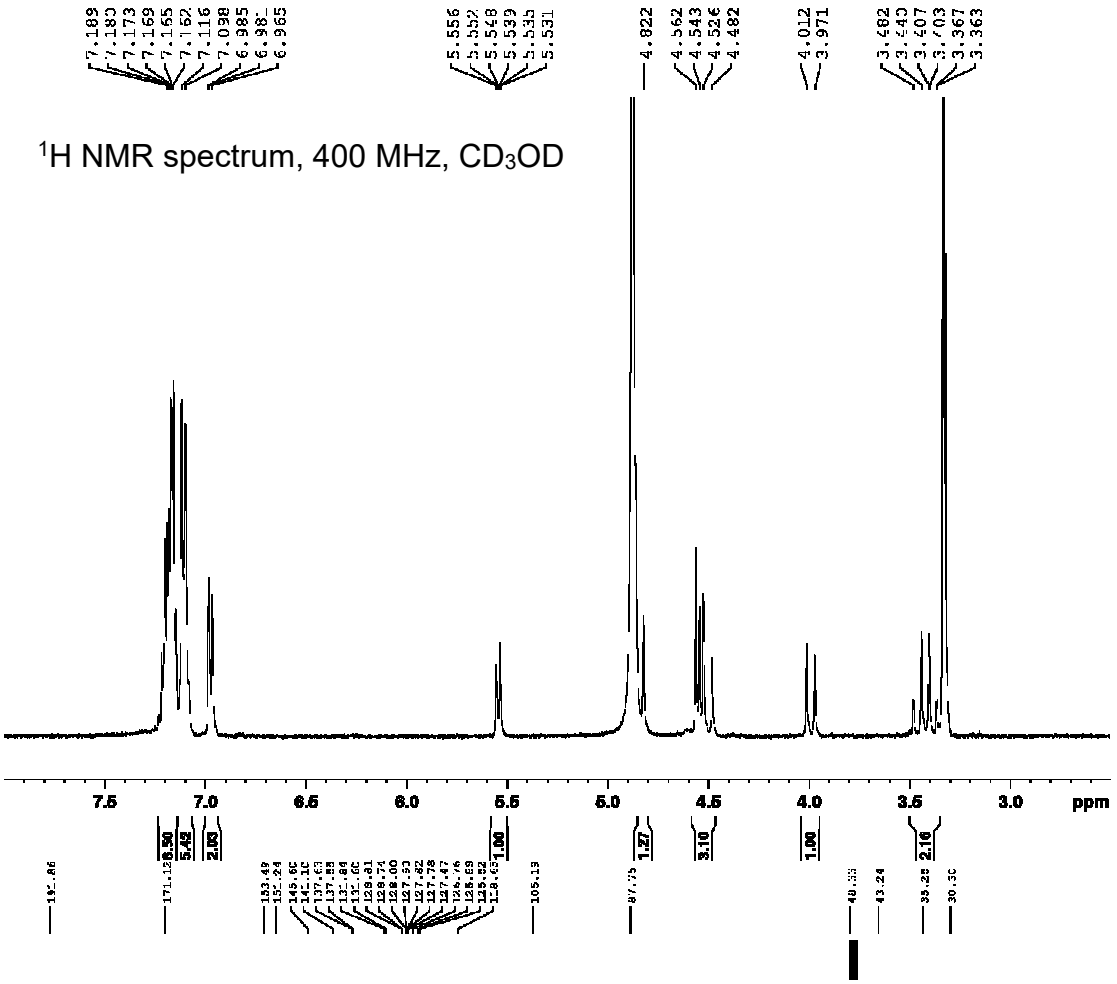
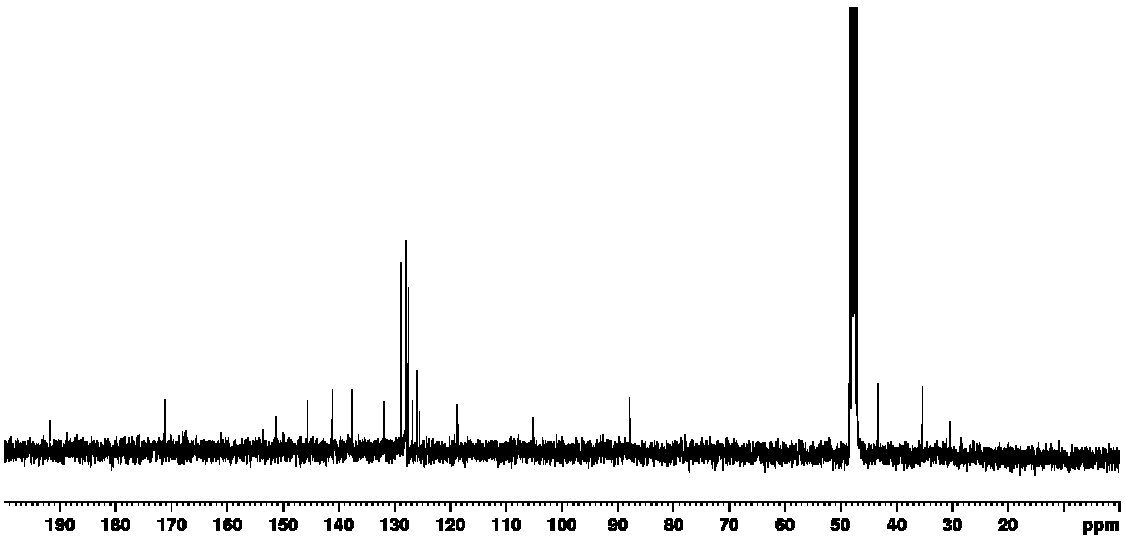




19red

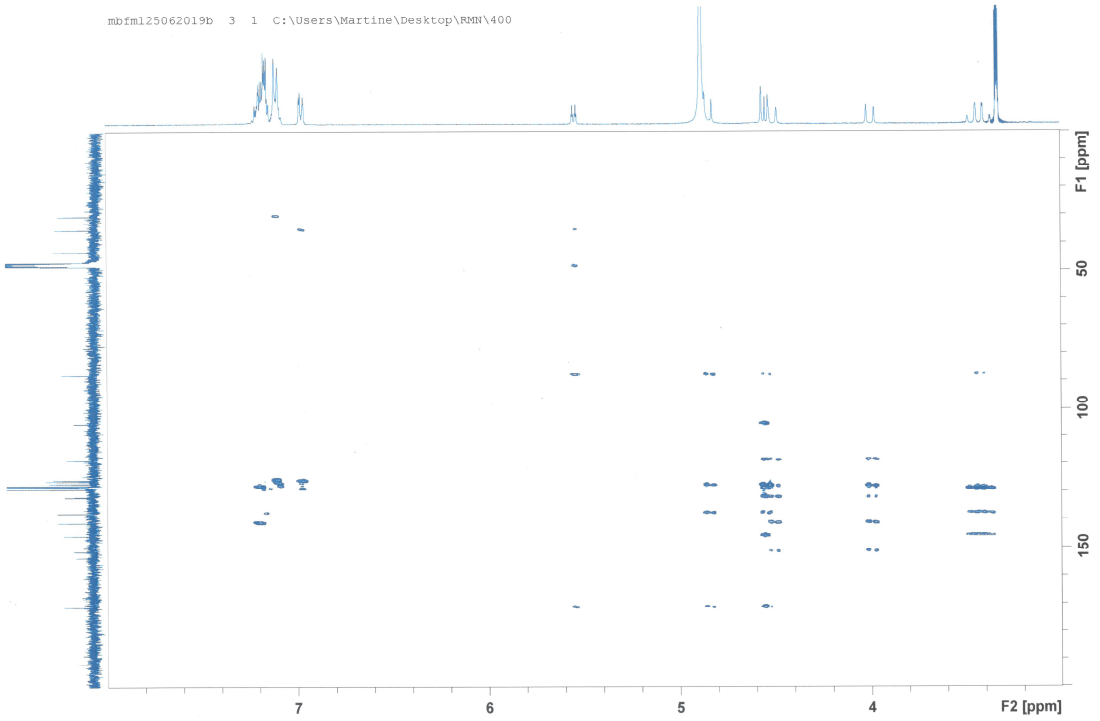


^{1}H NMR spectrum, 400 MHz, CD_3OD

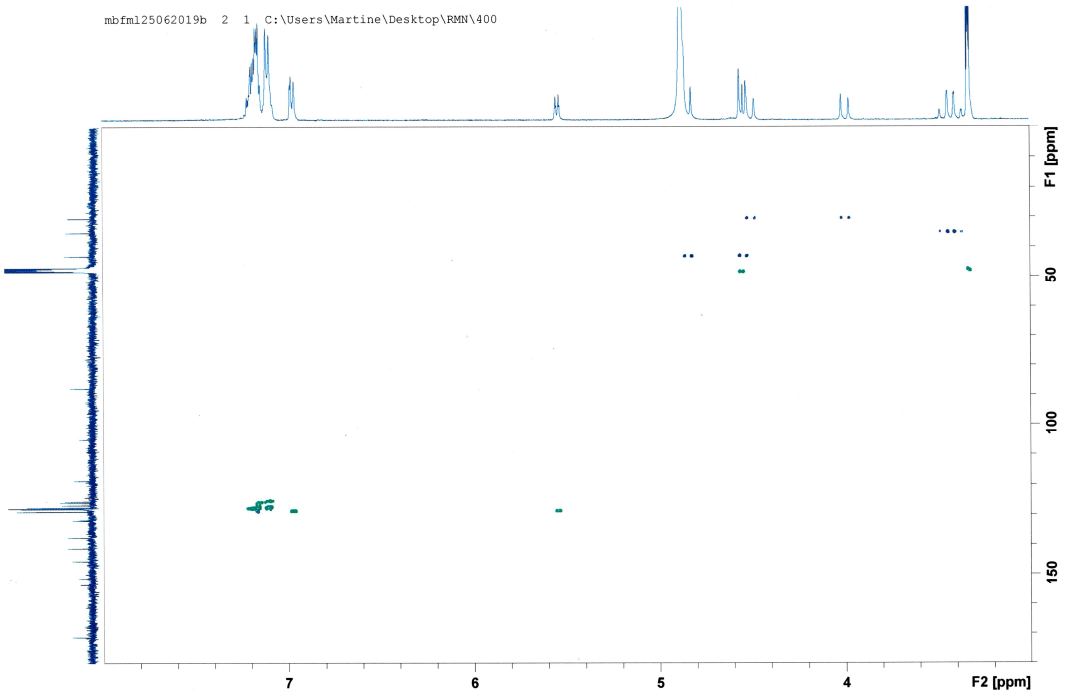


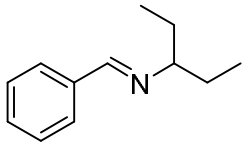
1D proton-decoupled ^{13}C NMR spectrum, 100 MHz, CD_3OD

HMBC Experiment (CD_3OD) for **19red**



HSQC Experiment (CD_3OD) for **19red**





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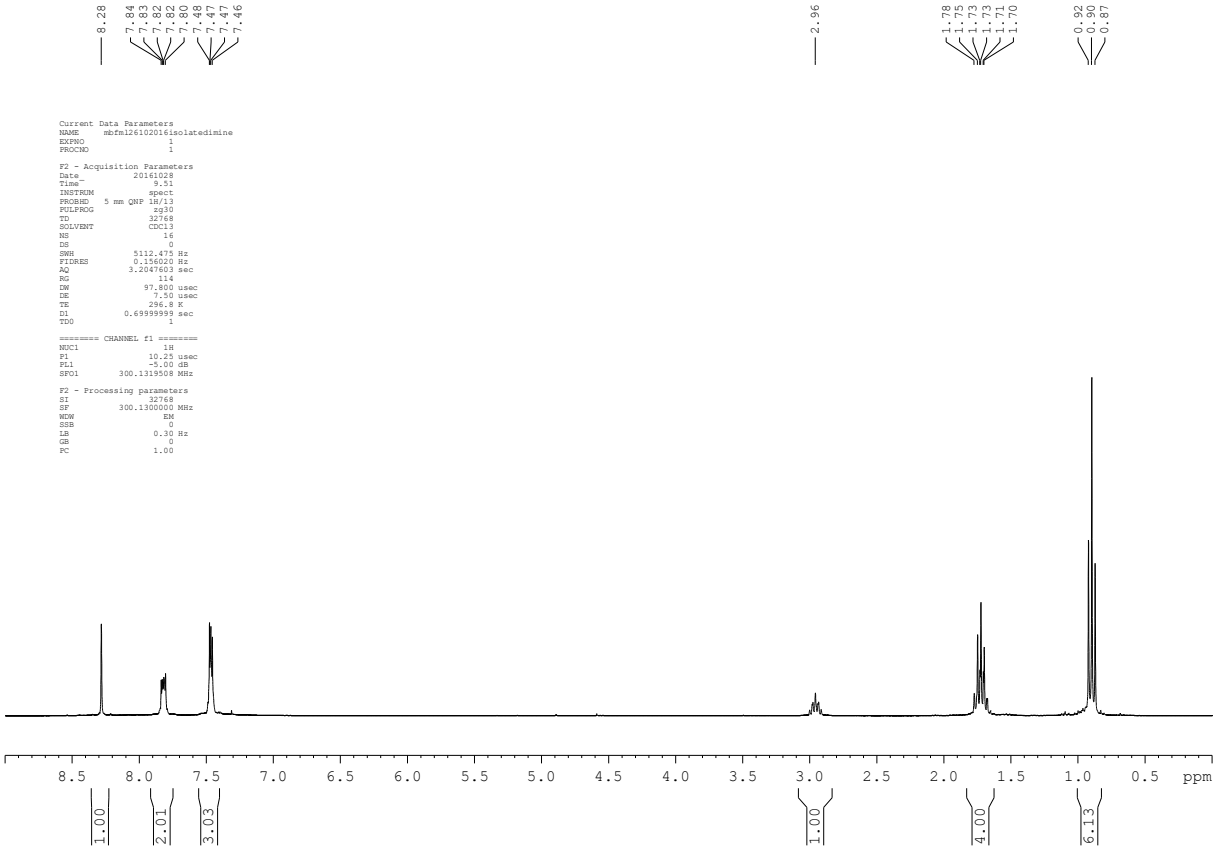
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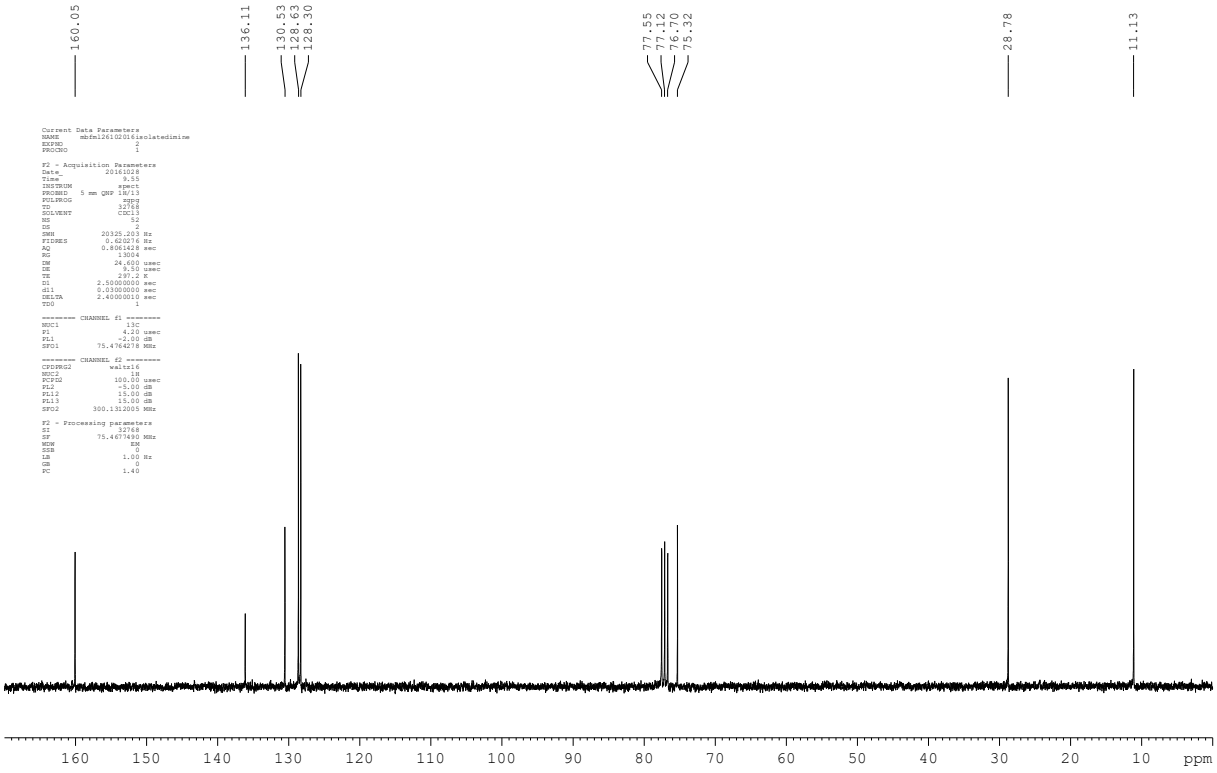
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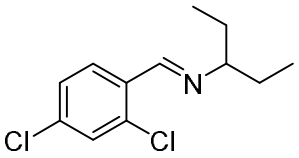
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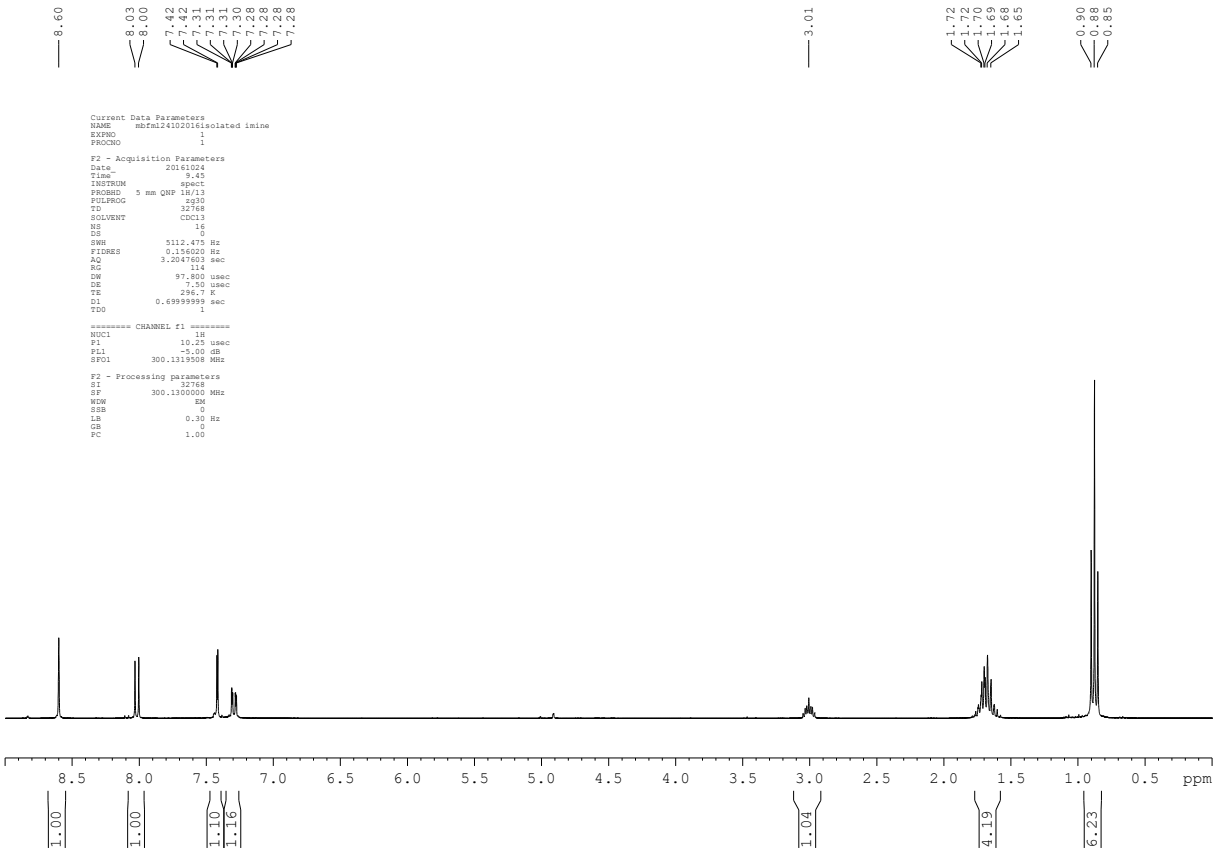
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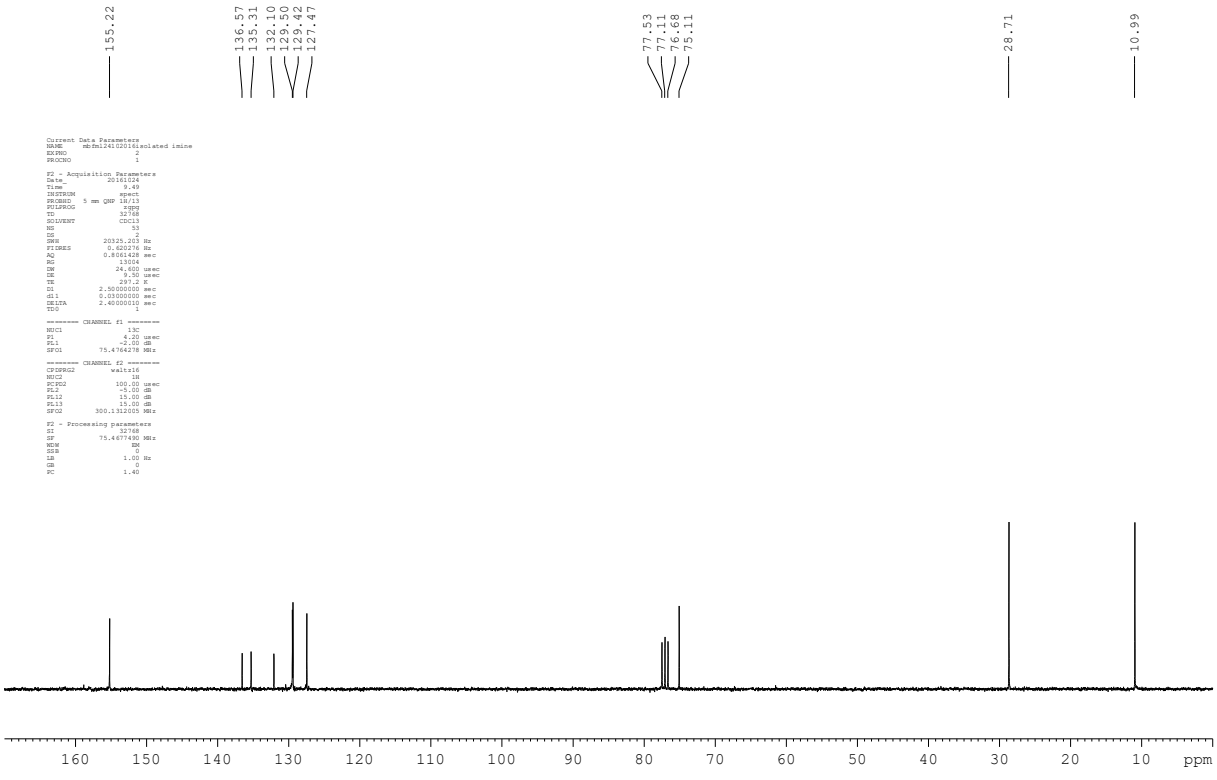
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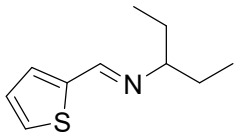
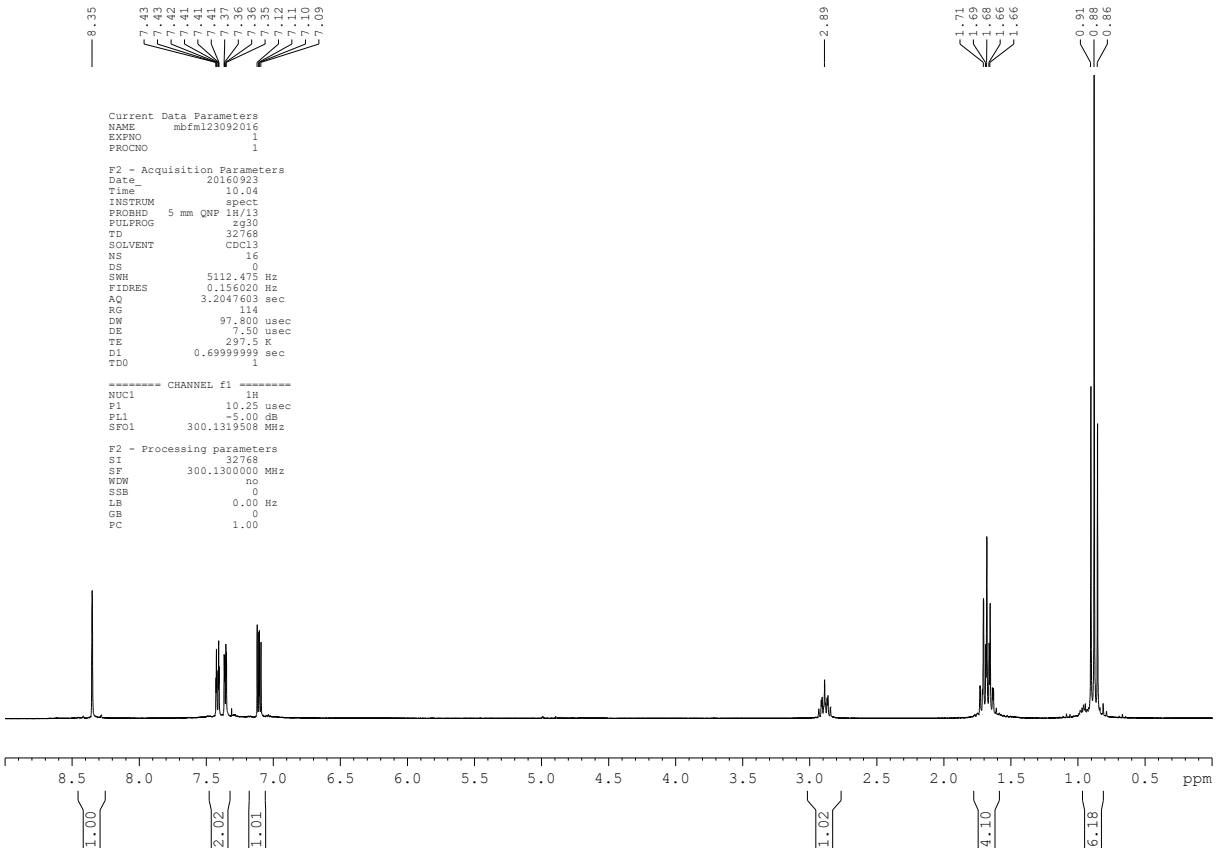


24c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FIDRES 0.156020 Hz
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RG 114
DW 97.800 usec
DE 7.50 usec
TE 297.5 K
D1 0.69999999 sec
TD0 1
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NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
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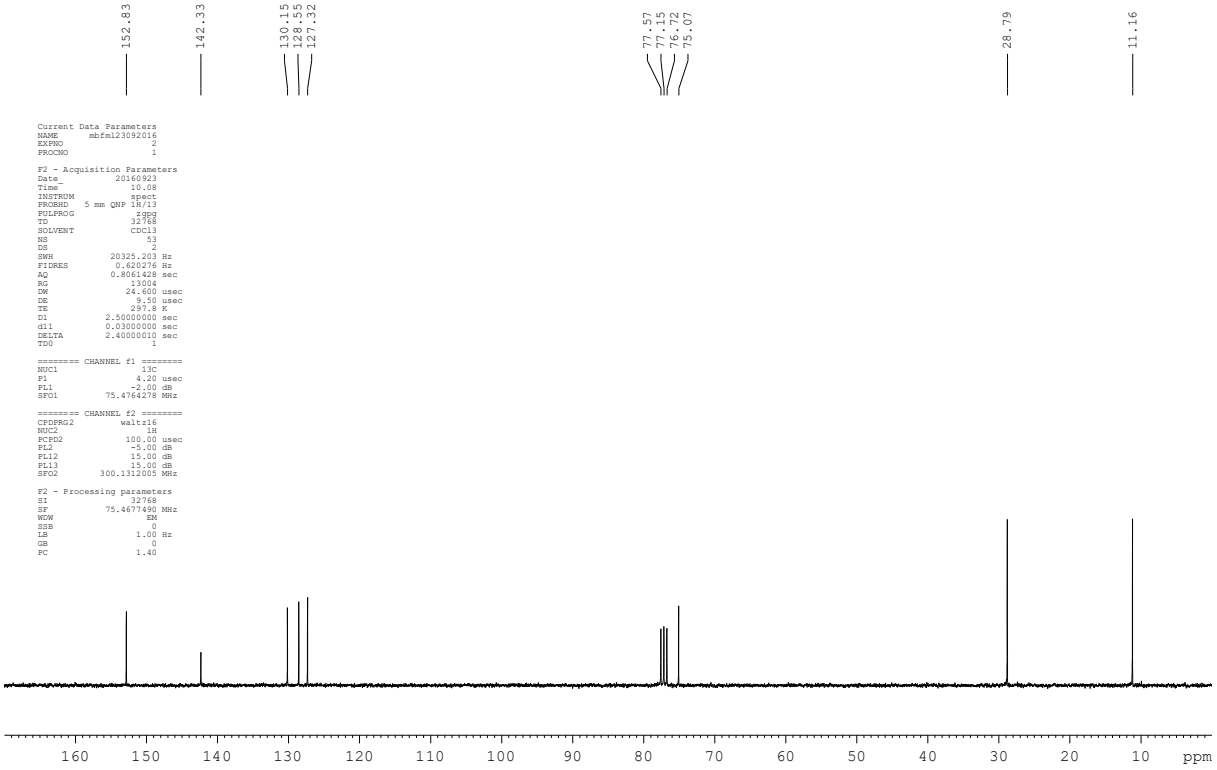
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EXPNO 2
PROCNO 1
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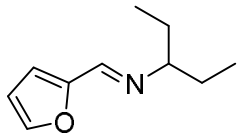
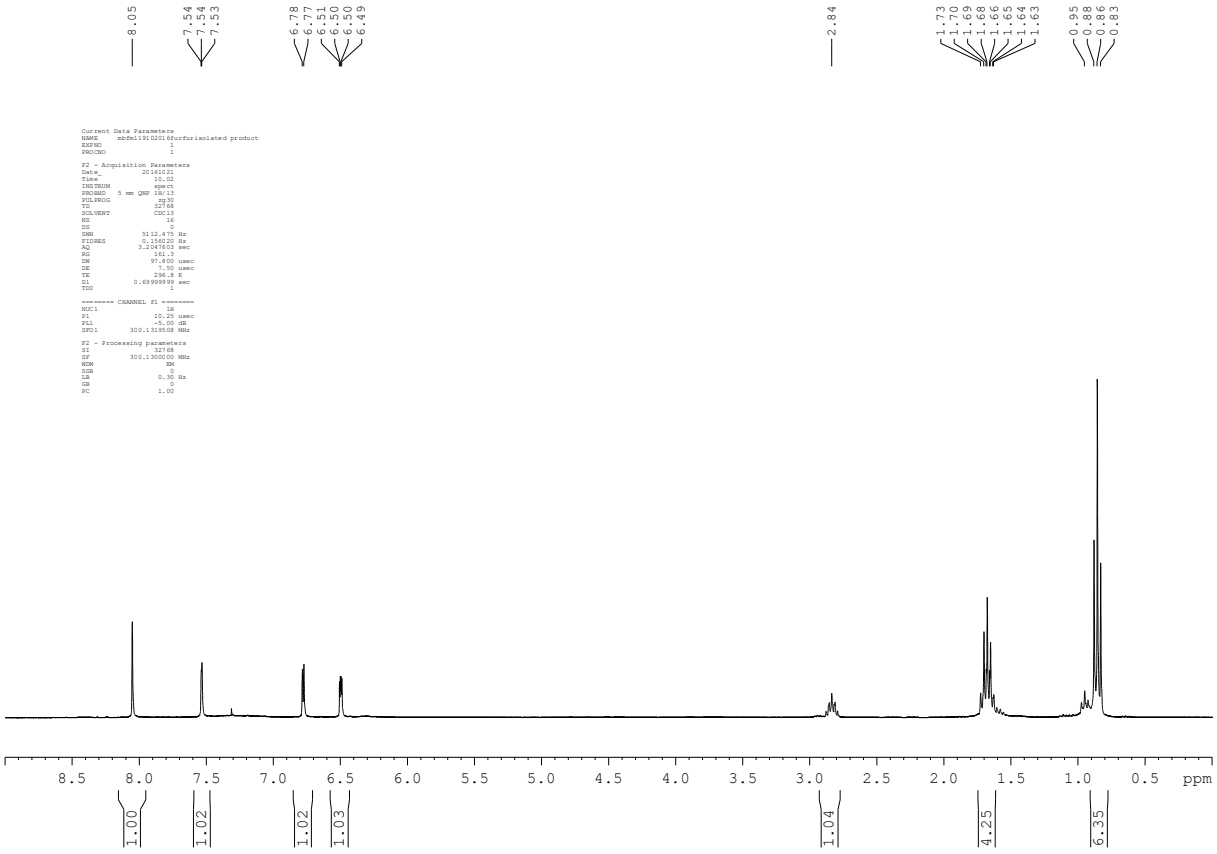
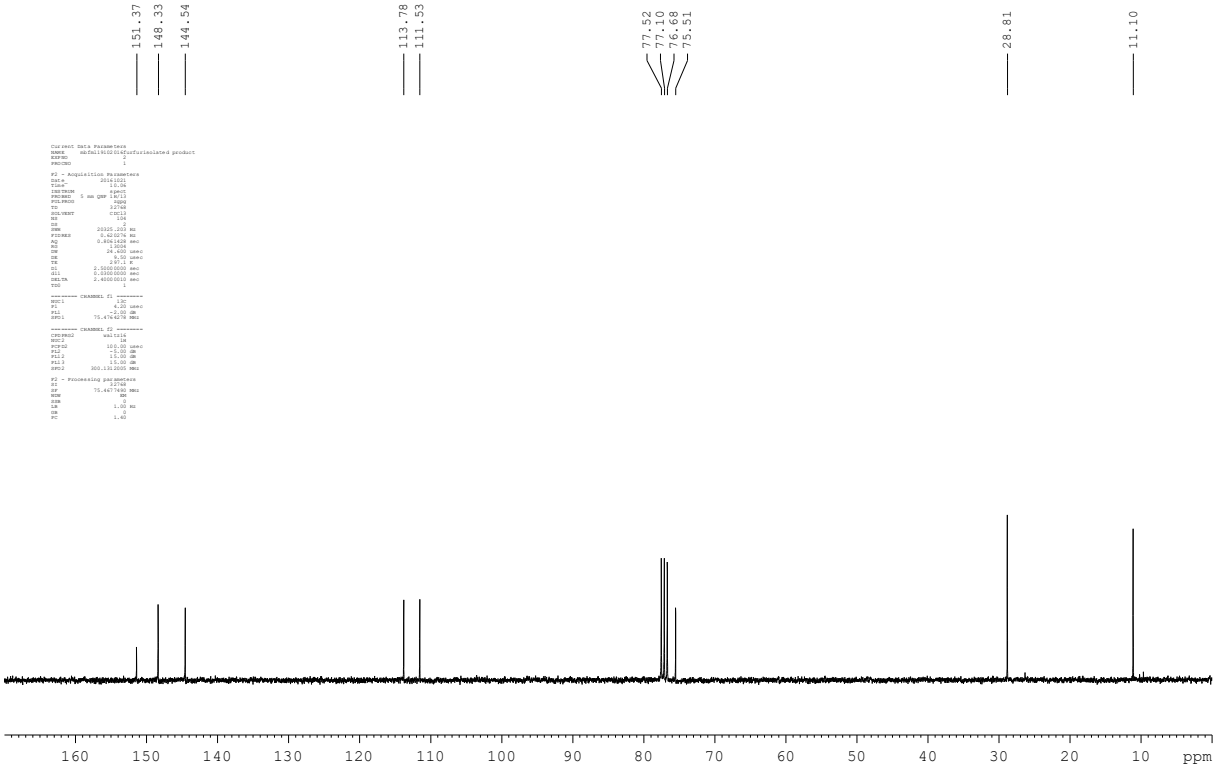
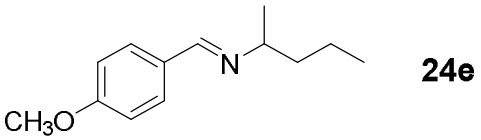
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 53
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 297.8 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



24d

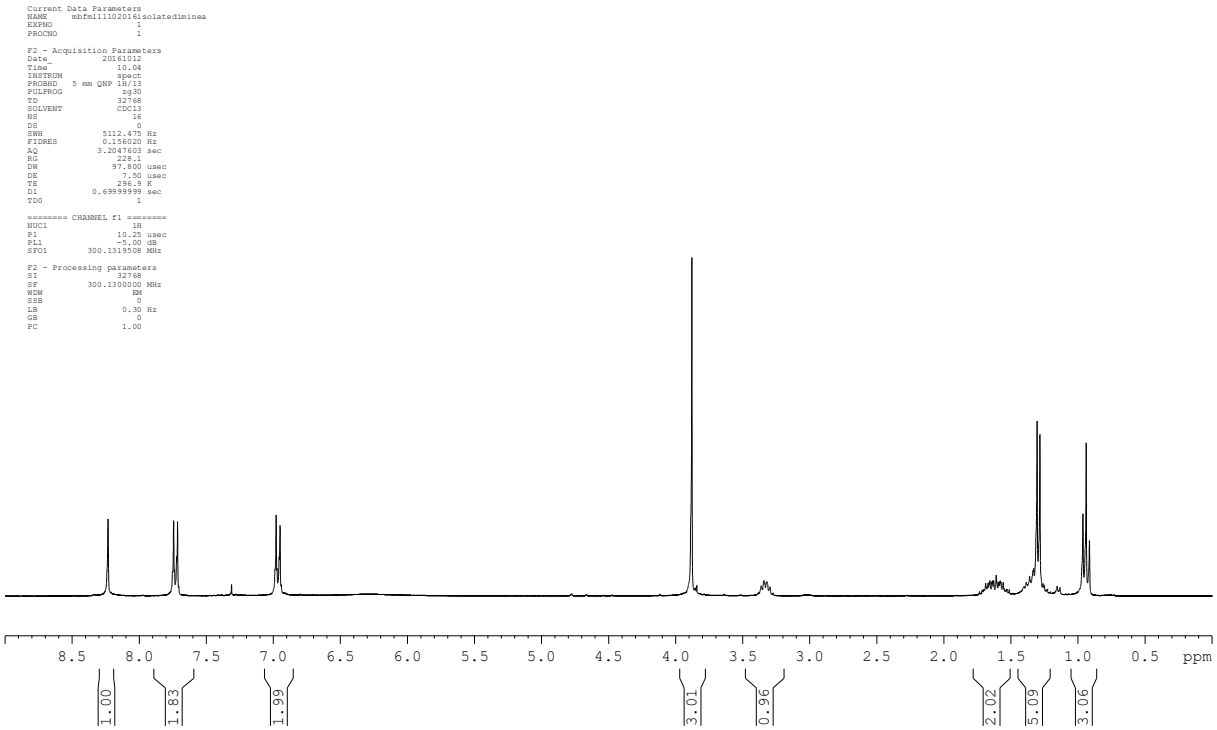

8.05
7.54
7.54
7.53
6.78
6.77
6.51
6.50
6.50
6.49
2.84
1.73
1.70
1.69
1.68
1.66
1.65
1.64
1.63
0.95
0.88
0.86
0.83
1.00
1.02
1.02
1.03
1.04
4.25
6.35
ppm

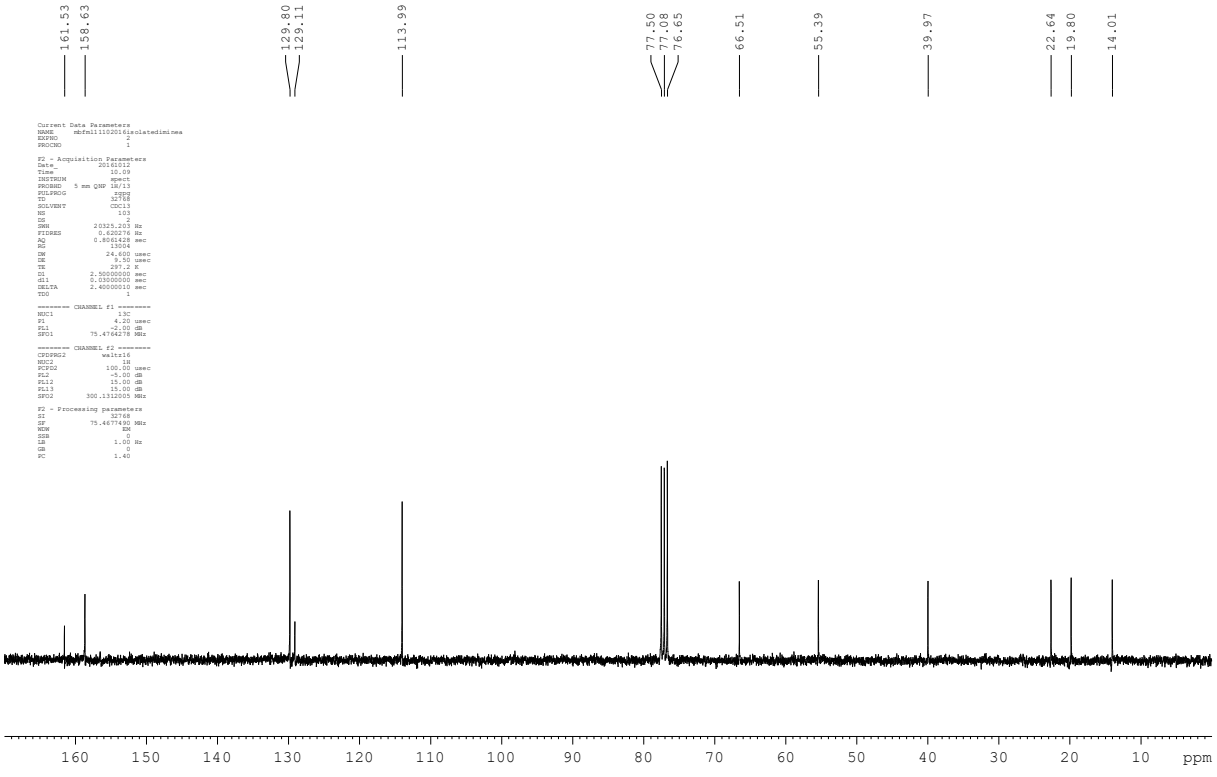

151.37
148.33
144.54
113.78
111.53
77.52
77.10
76.68
75.51
28.81
11.10
ppm

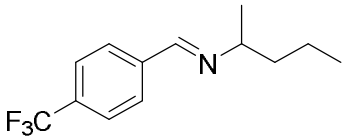

N
CH3O

24e

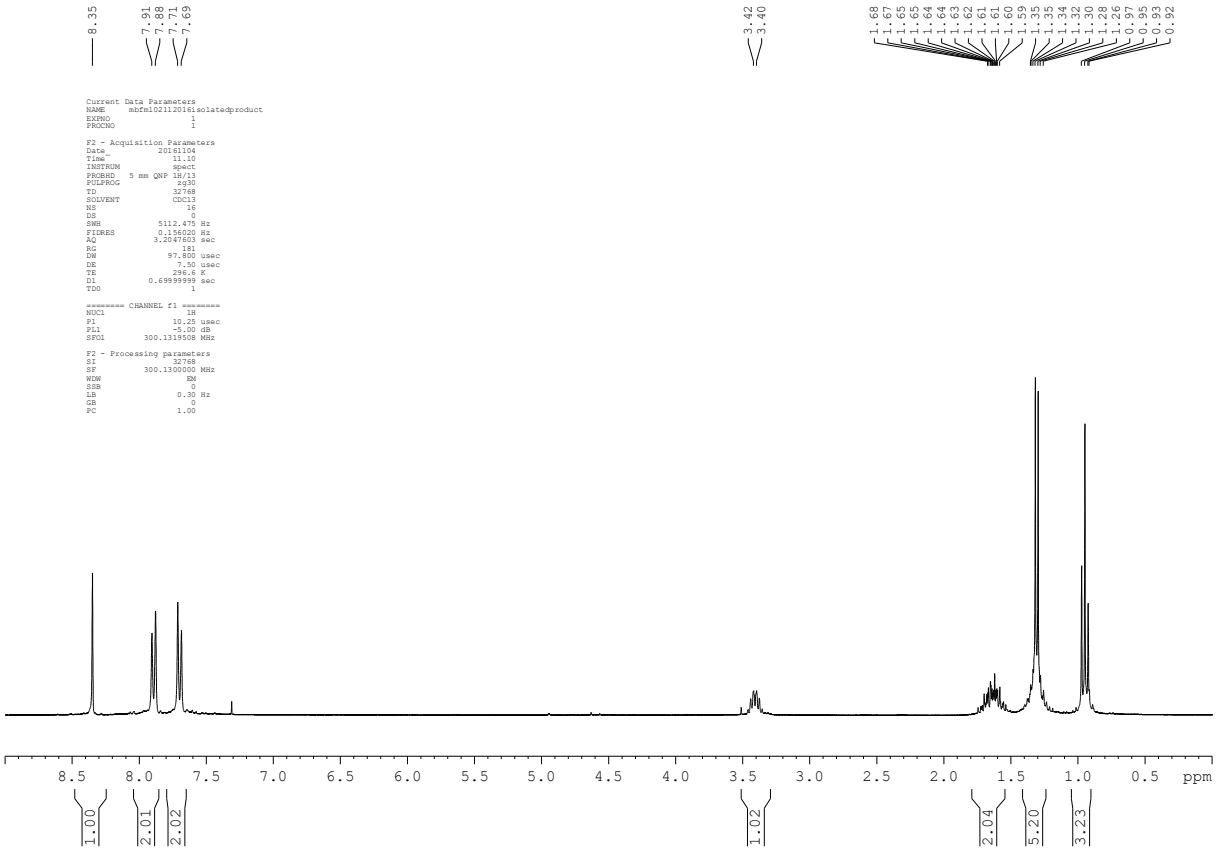

8.23
7.75
7.74
7.74
7.72
7.71
6.99
6.98
6.97
6.96
6.95
3.88
3.34
1.66
1.64
1.63
1.61
1.61
1.59
1.58
1.36
1.36
1.34
1.33
1.31
1.28
0.96
0.94
0.92

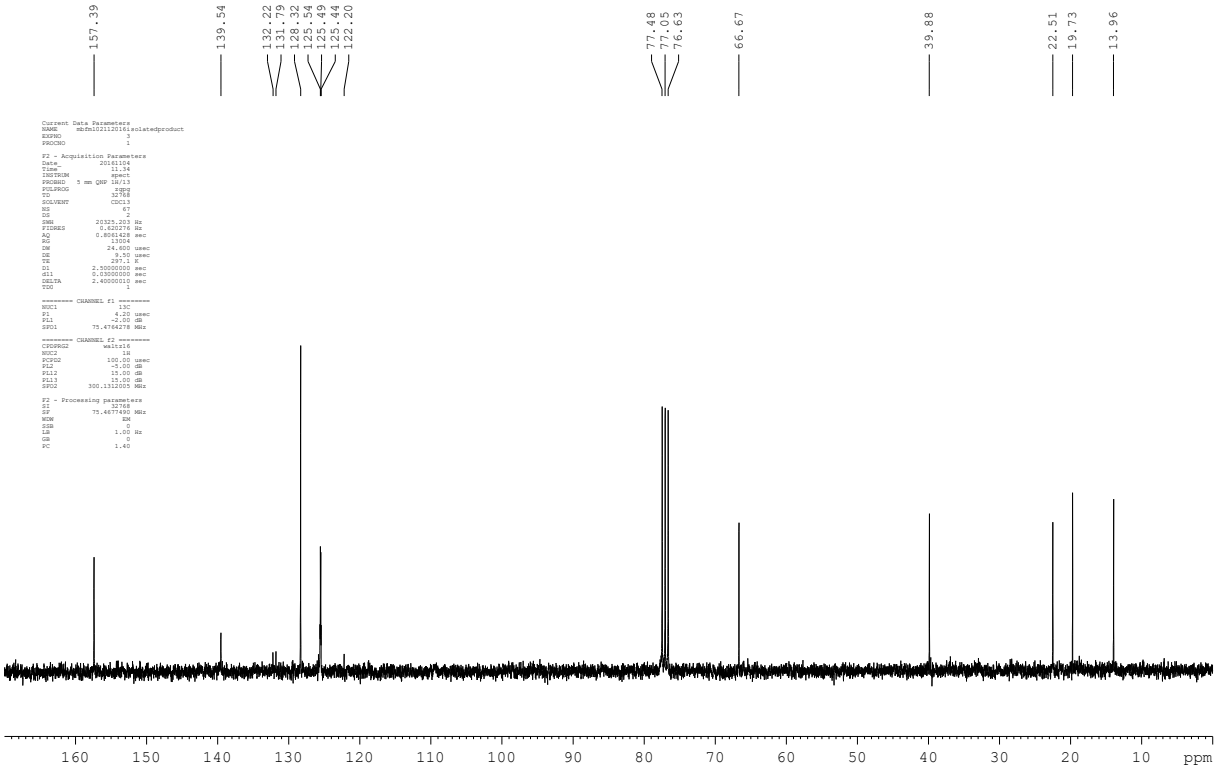

Current Data Parameters
NAME mbfml111020161isolatedimine
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161012
Time 10.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 228.1
DW 97.800 usec
DE 7.50 usec
TE 296.9 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.00
1.83
1.99
3.01
0.96
2.02
5.09
3.06

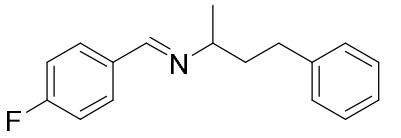

161.53
158.63
129.80
129.11
113.99
77.50
77.08
76.65
66.51
55.39
39.97
22.64
19.80
14.01
Current Data Parameters
NAME mbfml111020161isolatedimine
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161012
Time 10.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 103
DS 0
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061426 sec
RG 2206
DW 24.600 usec
DE 7.50 usec
TE 297.2 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======= CHANNEL f1 =======
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


N
F3C

24f


8.35
7.91
7.88
7.71
7.69
3.42
3.40
1.68
1.67
1.65
1.65
1.64
1.64
1.63
1.62
1.61
1.61
1.60
1.59
1.35
1.35
1.34
1.32
1.30
1.28
1.26
0.97
0.95
0.93
0.92
Current Data Parameters
NAME mbfm102112016isolatedproduct
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161104
Time 11.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 181
DW 97.800 usec
DE 7.50 usec
TE 296.6 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.00
2.01
2.02
1.02
2.04
5.20
3.23

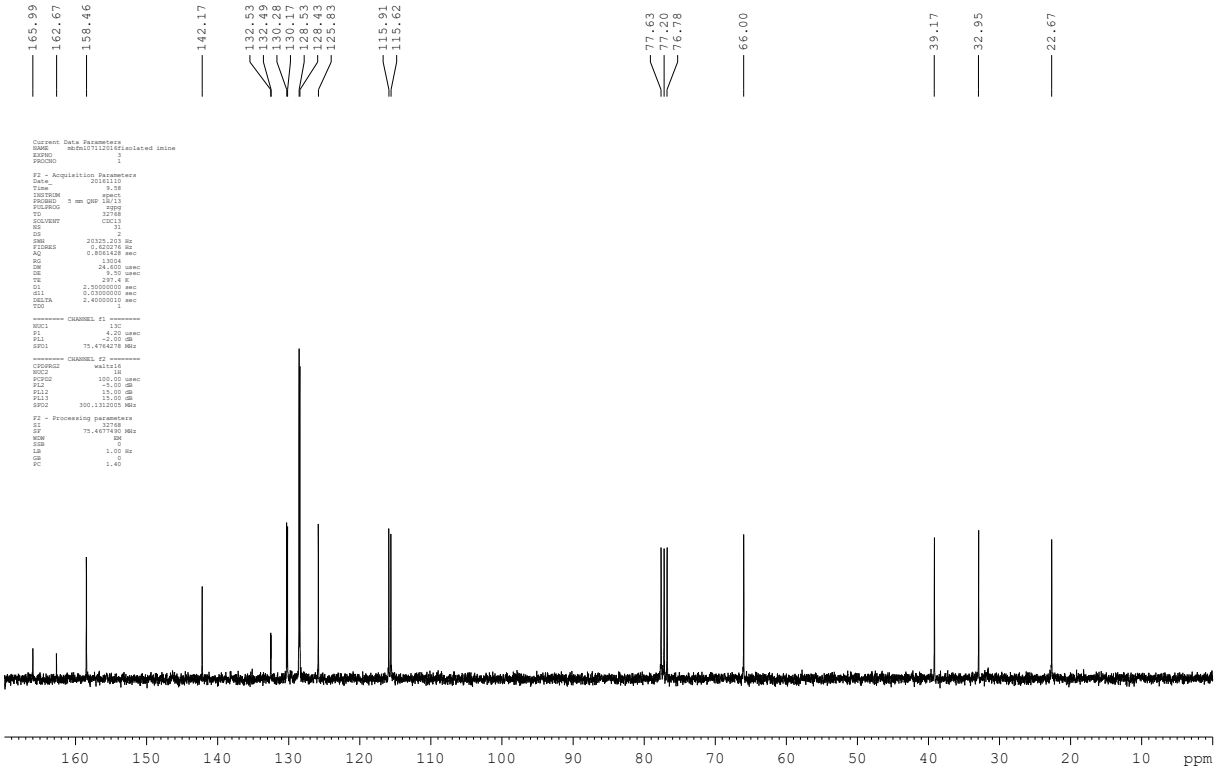

157.39
139.54
132.22
131.79
128.32
125.54
125.49
125.44
122.20
77.48
77.05
76.63
66.67
39.88
22.51
19.73
13.96
Current Data Parameters
NAME mbfm102112016isolatedproduct
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161104
Time 11.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 67
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 sec
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 297.1 K
D1 2.50000000 sec
D11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

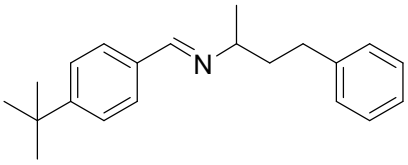

N
F

24g


8.30 7.87 7.86 7.85 7.84 7.82 7.39 7.39 7.39 7.37 7.37 7.36 7.34 7.29 7.28 7.27 7.25 7.21 7.18 7.15
3.45 3.45 3.44 3.43 3.43 2.74 2.72 2.71 2.69 2.67 2.65 2.64 2.62 2.13 2.11 2.10 2.08 2.07 2.07 2.05 2.04 2.03 2.03 2.02 2.02 1.40 1.38

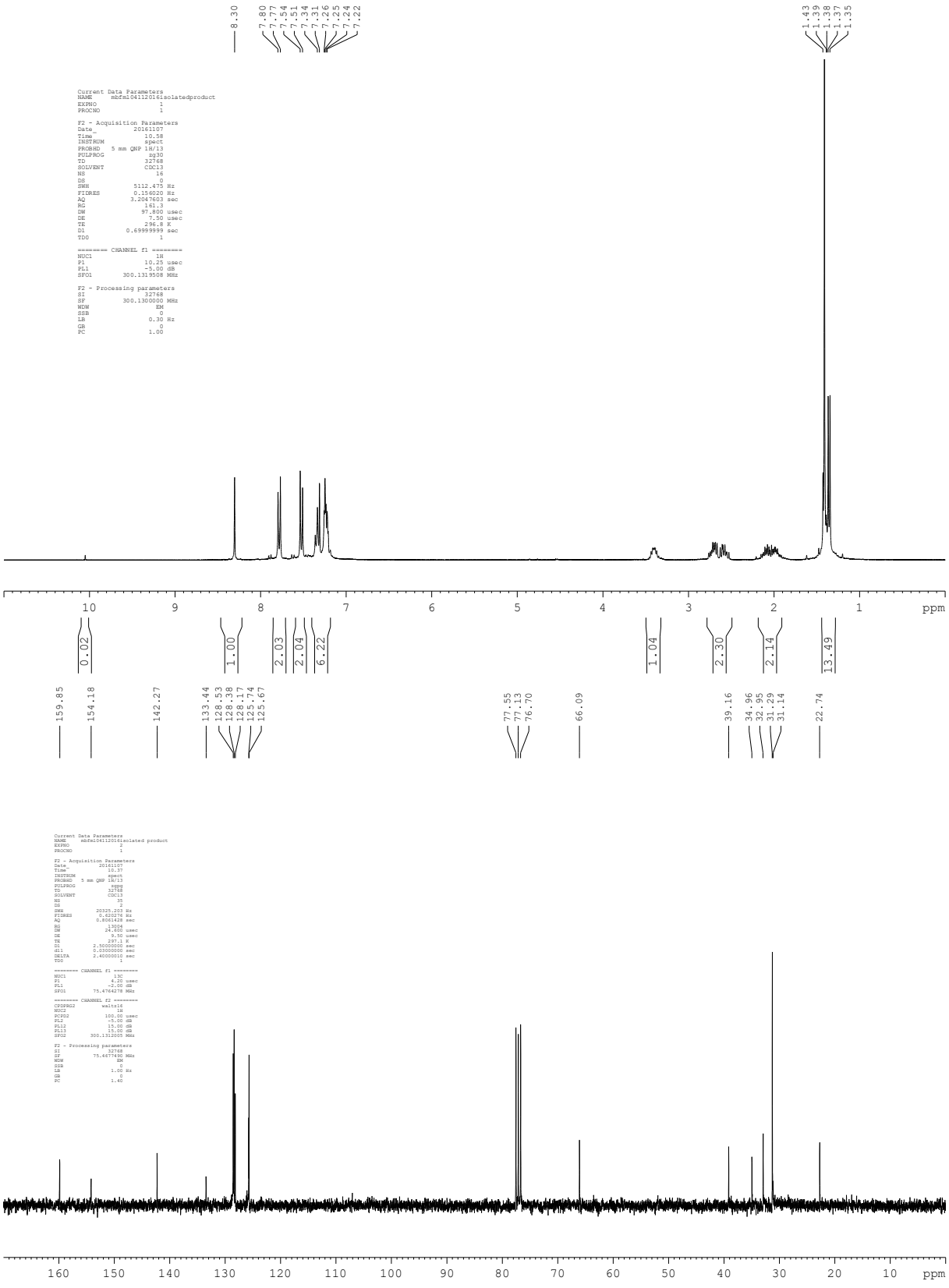

Current Data Parameters
NAME mbfm107112016fisolated imine
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161110
Time 9.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 71.8
DW 97.800 usec
DE 7.50 usec
TE 297.1 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.00 2.07 8.50 1.08 2.43 2.31 3.16

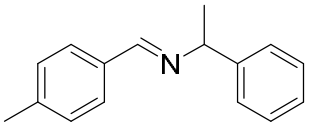

165.99 162.67 158.46 142.17 132.53 132.49 130.29 130.17 128.53 128.43 125.83 115.91 115.62 77.63 77.20 76.78 66.00 39.17 32.95 22.67
Current Data Parameters
NAME mbfm107112016fisolated imine
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161110
Time 9.58
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 91
DS 0
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8058496 sec
RG 2.0000
DW 24.600 usec
DE 9.50 usec
TE 297.4 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



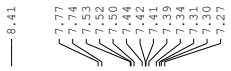
24h

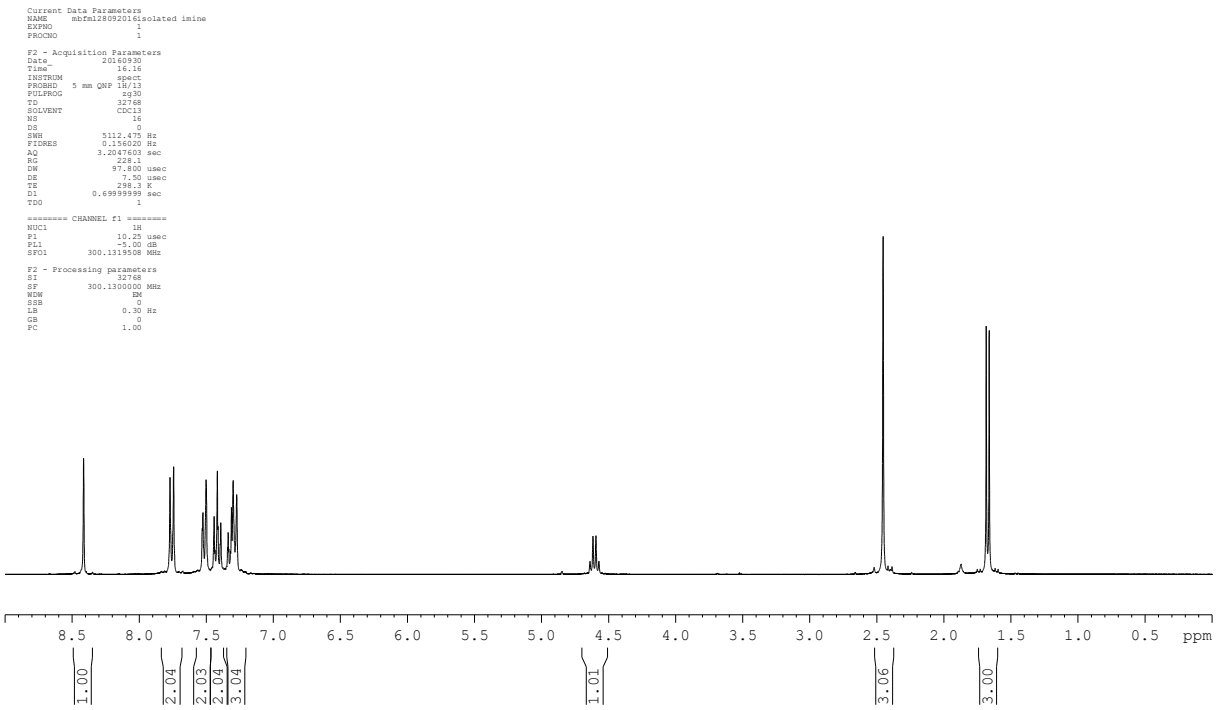
(contained trace of aldehyde formed during column chromatography)

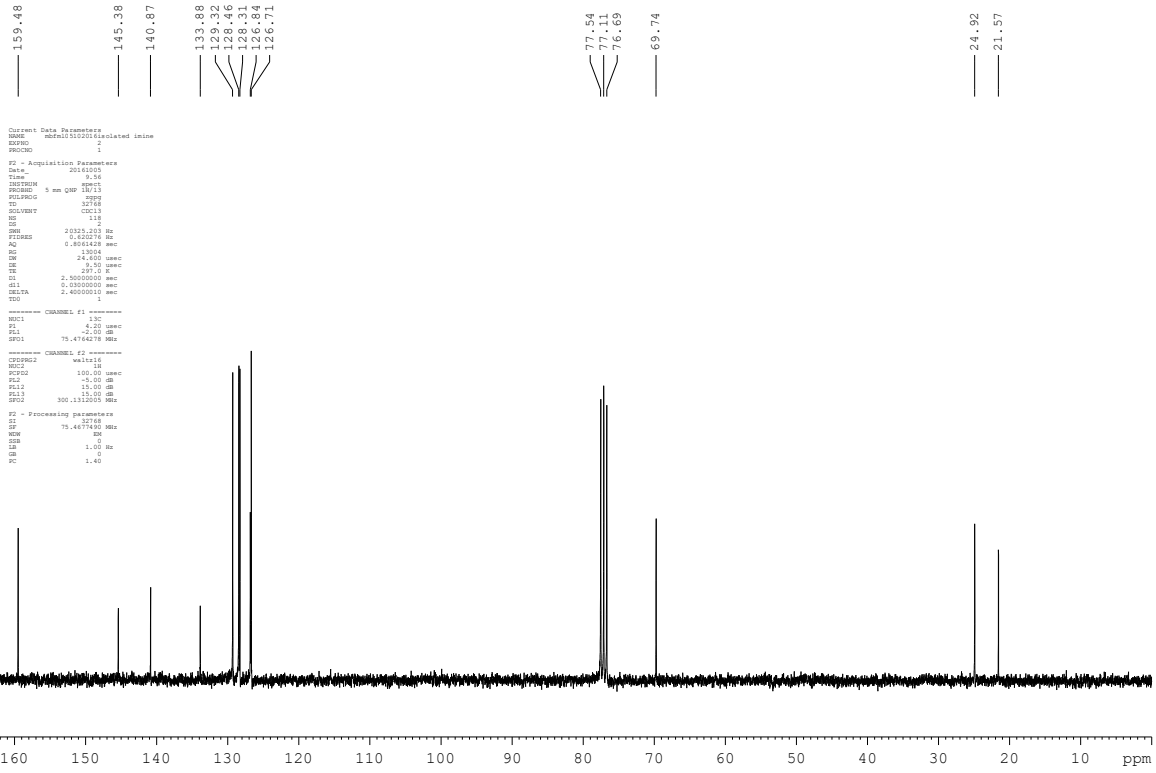


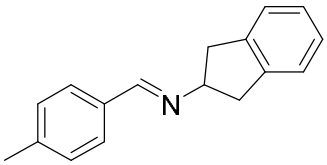


24i


8.41
7.77
7.74
7.53
7.52
7.50
7.44
7.42
7.41
7.39
7.34
7.31
7.30
7.27
4.62
4.59
2.45
1.68
1.66


Current Data Parameters
NAME mbfml28092016isolated imine
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date 20160930
Time 16.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 228.1
DW 97.800 usec
DE 7.50 usec
TE 298.3 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.00
2.04
2.03
2.04
3.04
1.01
3.06
3.00


159.48
145.38
140.87
133.88
129.32
128.46
128.31
126.84
126.71
77.54
77.11
76.69
69.74
24.92
21.57
Current Data Parameters
NAME mbfml15102016isolated imine
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161005
Time 9.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 118
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 297.0 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312905 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


N

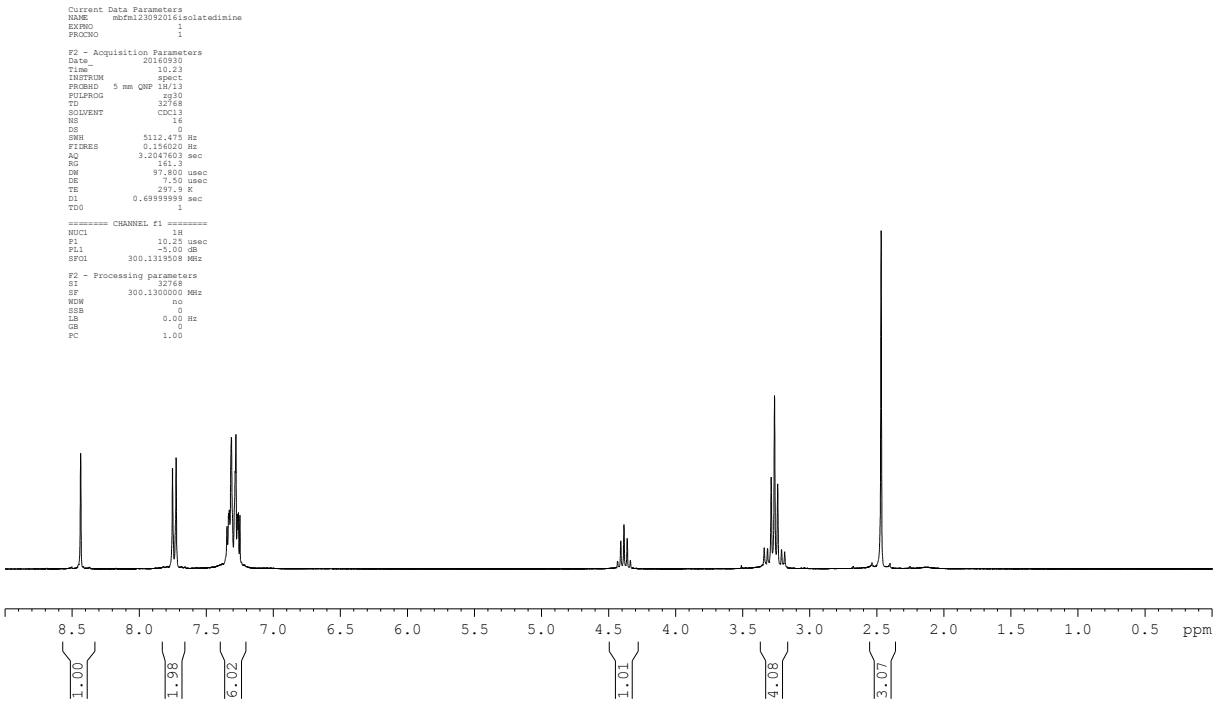
24j

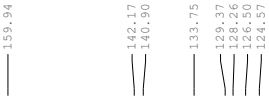

8.44
7.75
7.72
7.34
7.33
7.31
7.28
7.27
7.26
7.25

4.38


3.29
3.26
3.24

2.47


Current Data Parameters
NAME mbfm123092016isolatedimine
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160930
Time 10.23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 161.3
DW 97.800 usec
DE 7.50 usec
TE 297.9 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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 ppm
1.00
1.98
6.02
1.01
4.08
3.07

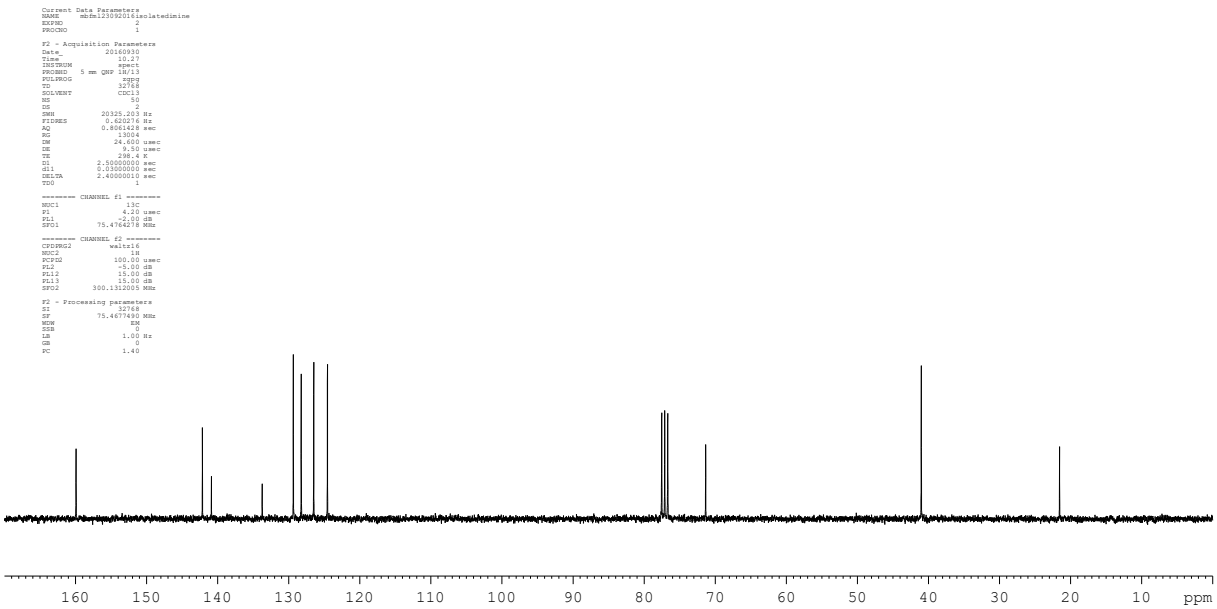

159.94
142.17
140.90
133.75
129.37
128.26
126.50
124.57

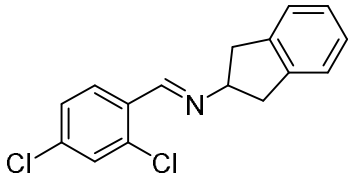

77.57
77.15
76.72

71.38

41.04

21.59


Current Data Parameters
NAME mbfm123092016isolatedimine
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160930
Time 10.27
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 50
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 298.4 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



24k


```
Current Data Parameters
NAME      mbfml25102016isolatedimine
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20161027
Time_              16.29
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           5112.475 Hz
FIDRES        0.156020 Hz
AQ            3.2047603 sec
RG                456.1
DW               97.800 usec
DE                7.50 usec
TE                296.8 K
D1           0.69999999 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.25 usec
PL1               -5.00 dB
SFO1        300.1319508 MHz

F2 - Processing parameters
SI                32768
SF          300.1300000 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00
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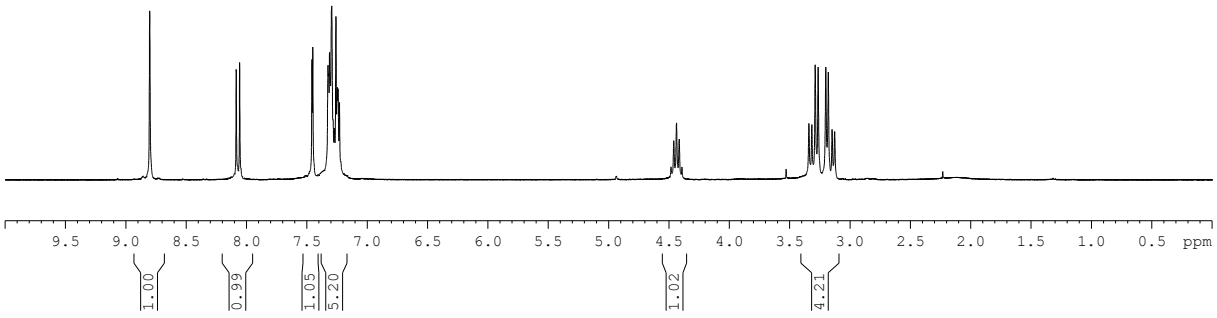


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Current Data Parameters
NAME      mbfml25102016isolatedimine
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20161027
Time              16.44
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg
TD                32768
SOLVENT           CDCl3
NS                  247
DS                    2
SWH           20325.203 Hz
FIDRES        0.620276 Hz
AQ            0.8061428 sec
RG                13004
DW               24.600 usec
DE                9.50 usec
TE                297.4 K
D1           2.50000000 sec
d11          0.03000000 sec
DELTA        2.40000010 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                4.20 usec
PL1               -2.00 dB
SFO1         75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2               -5.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                 1.40
```
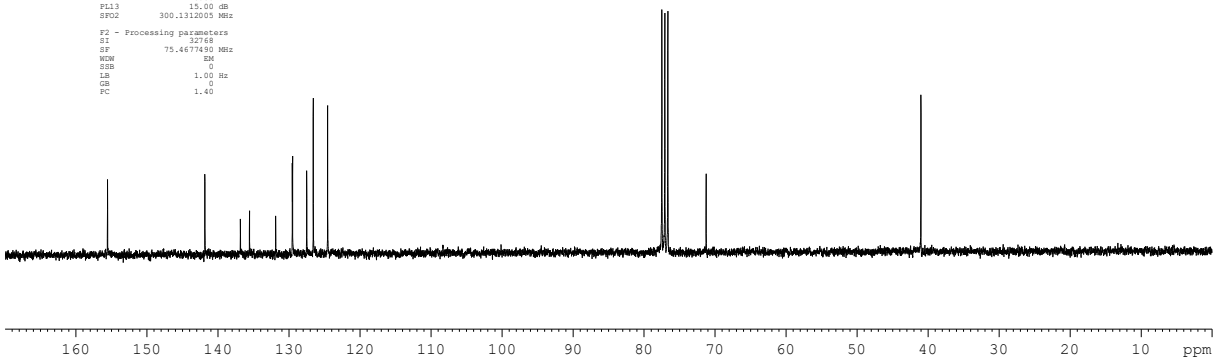
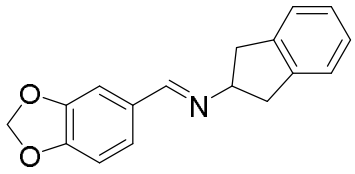
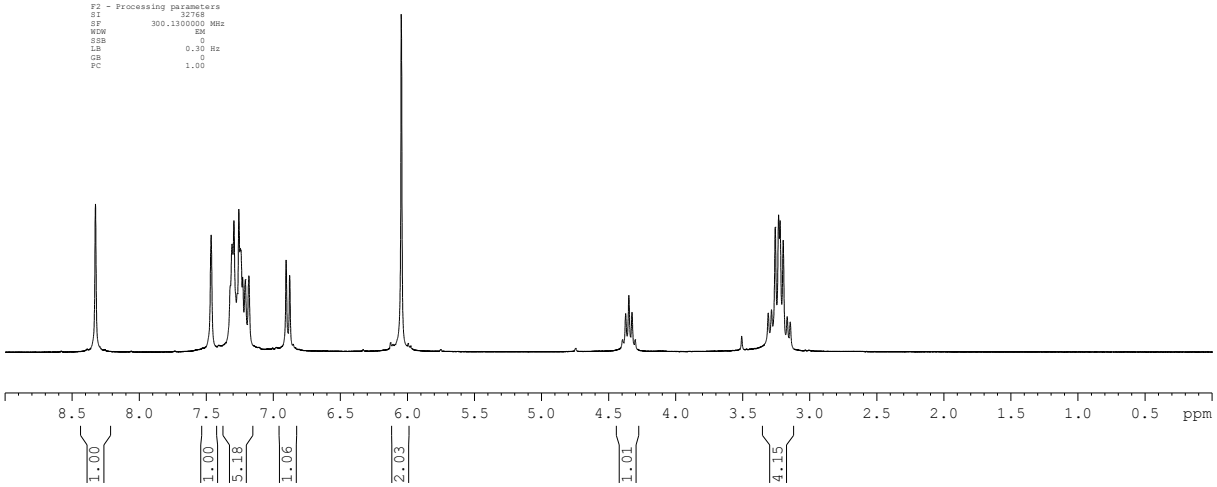



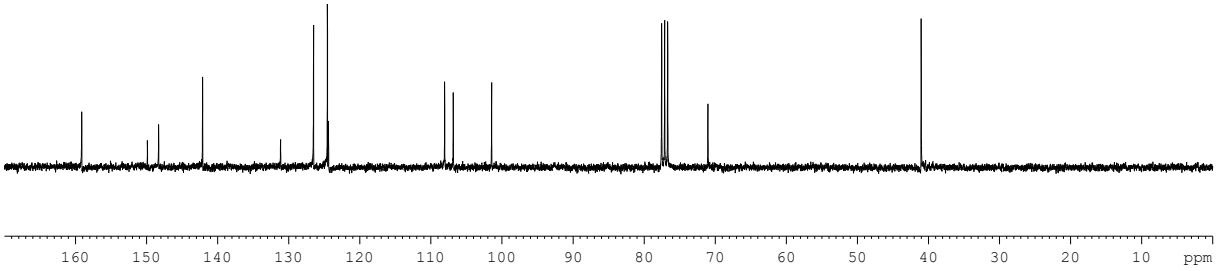


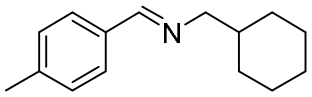
24l


```
Current Data Parameters
NAME      mbfml16112016alsolatedimine
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161116
Time               16.06
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH             5112.475 Hz
FIDRES          0.156020 Hz
AQ              3.2047603 sec
RG                228.1
DW                97.800 usec
DE                 7.50 usec
TE                296.8 K
D1             0.69999999 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.25 usec
PL1               -5.00 dB
SFO1         300.1319508 MHz

F2 - Processing parameters
SI                32768
SF           300.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

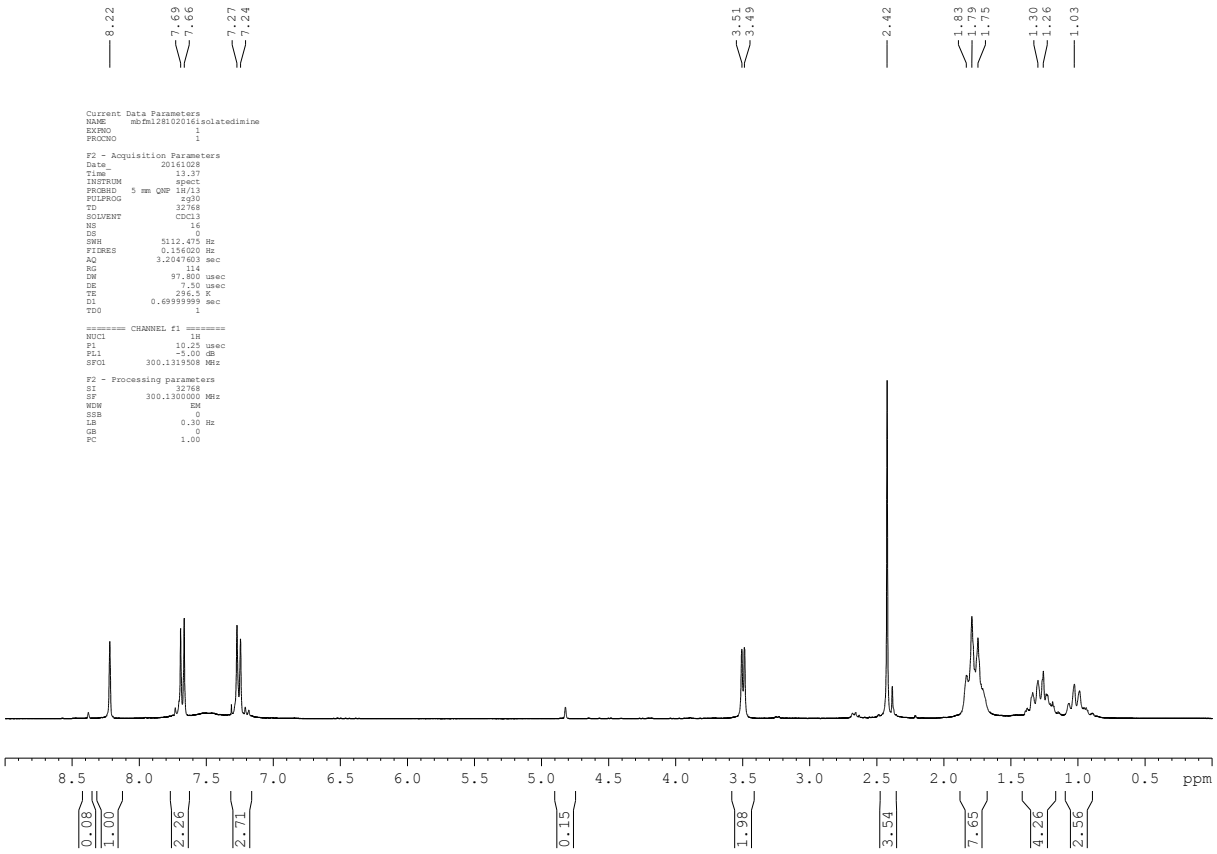


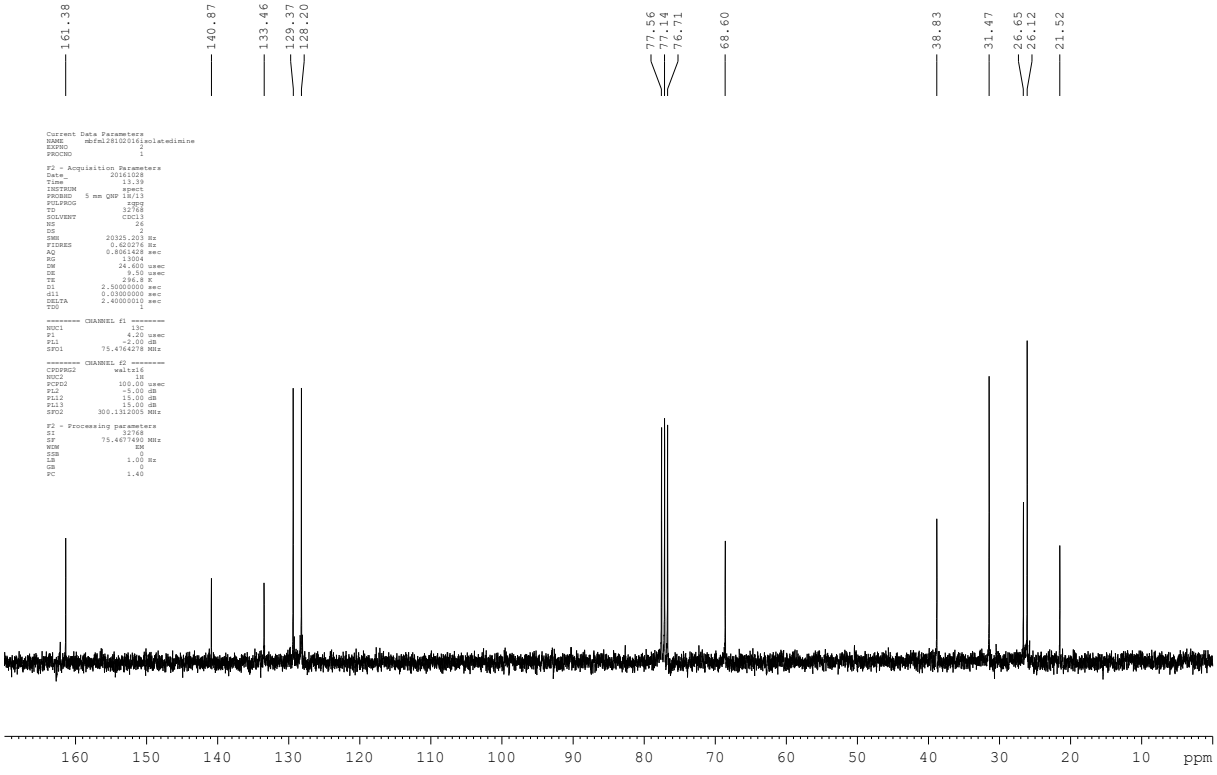

```
Current Data Parameters
NAME      mbfml16112016alsolatedimine
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20161116
Time               16.22
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg
TD                32768
SOLVENT           CDCl3
NS                  110
DS                    2
SWH            20325.203 Hz
FIDRES          0.620276 Hz
AQ              0.8061428 sec
RG                13004
DW                24.600 usec
DE                 6.50 usec
TE                297.4 K
D1             2.50000000 sec
d11            0.03000000 sec
DELTA          2.40000010 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 4.20 usec
PL1               -2.00 dB
SFO1          75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2               -5.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                32768
SF            75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

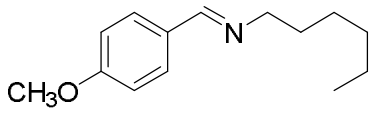




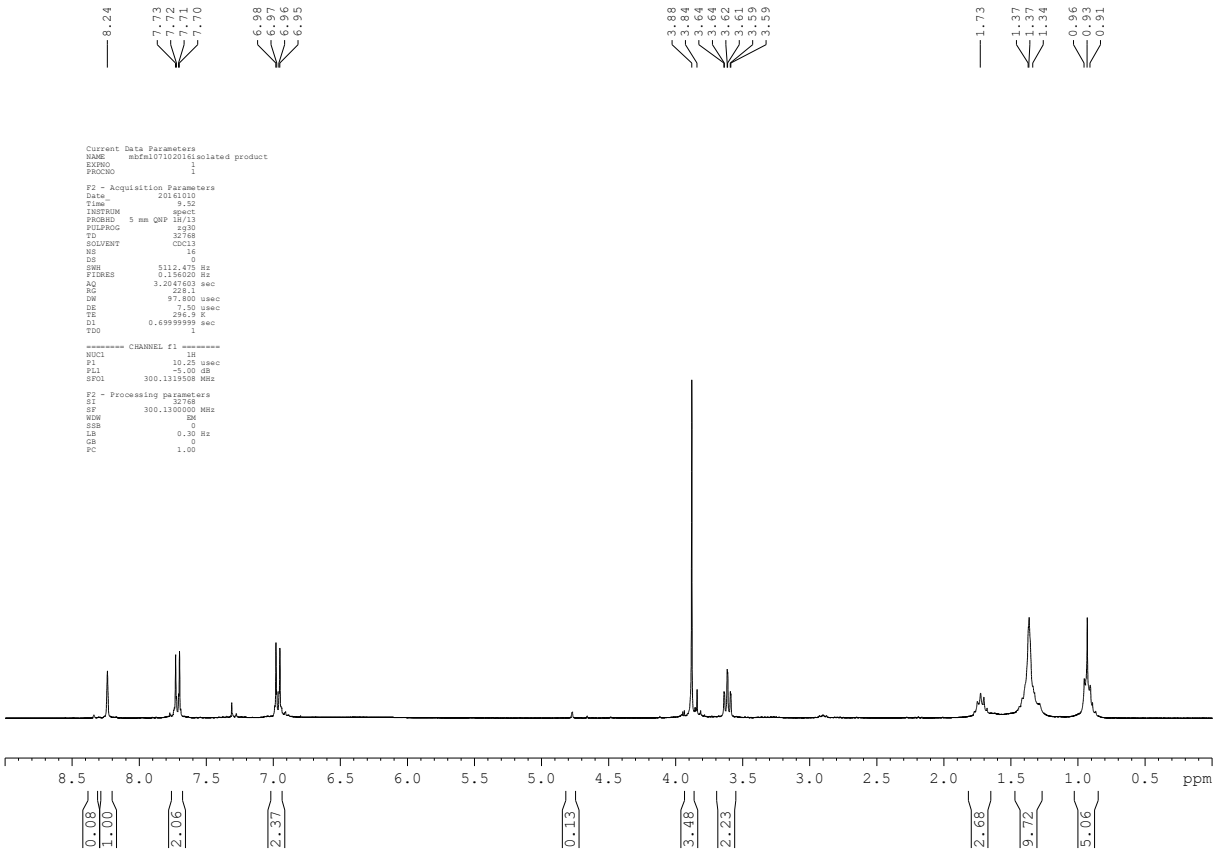
24m (contained 7% of homocoupled imine)

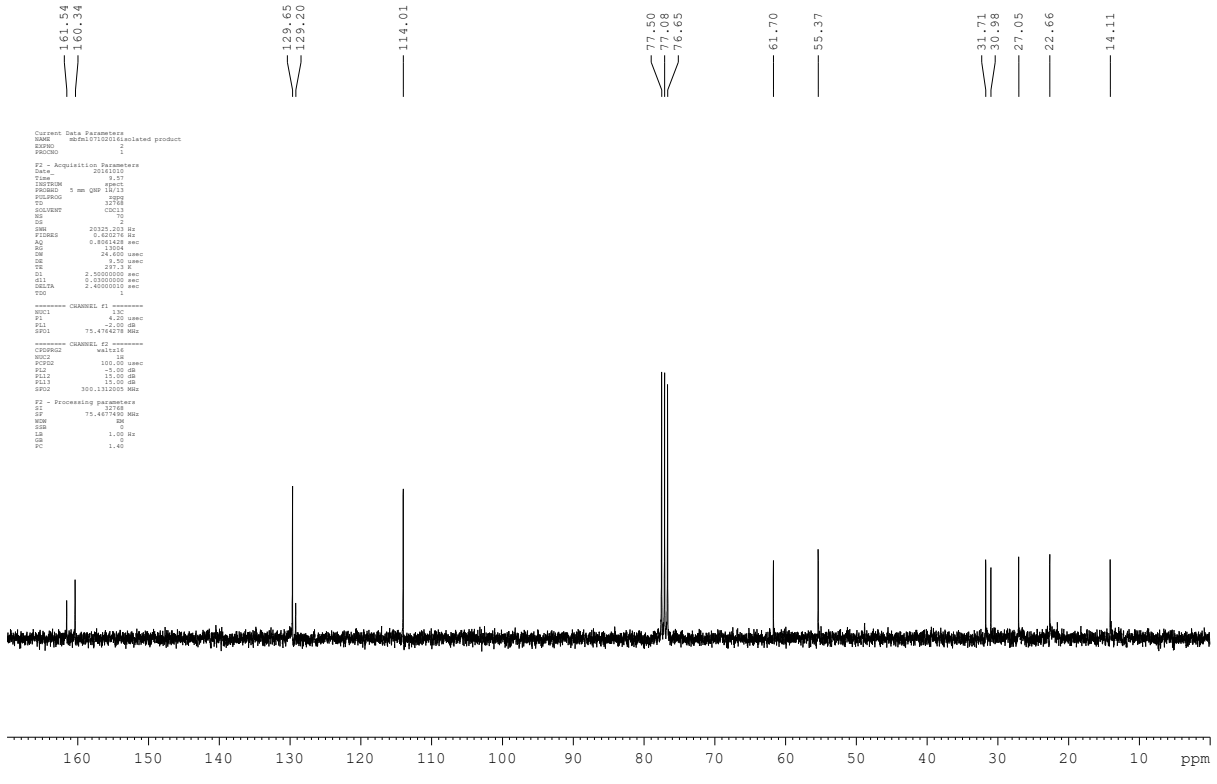


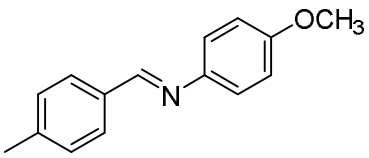




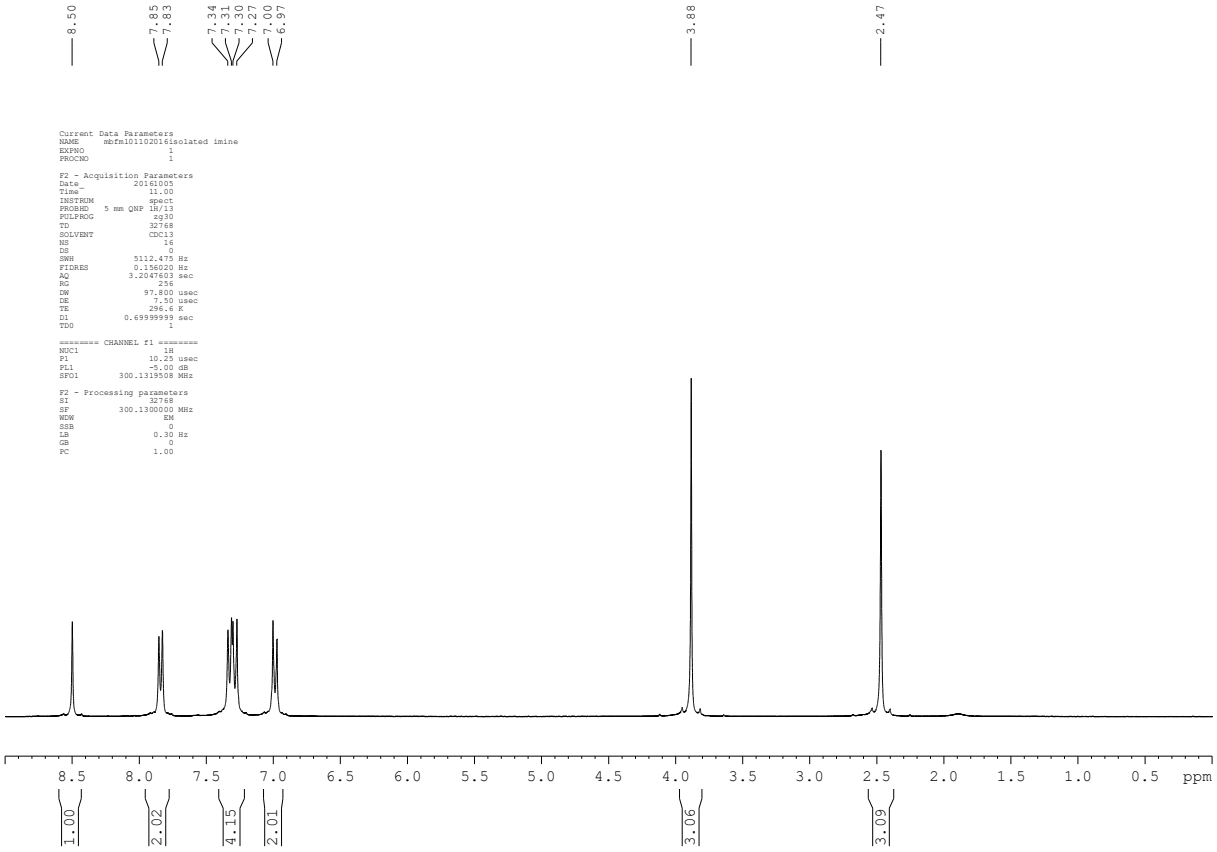
24n (contained 7% of homocoupled imine)

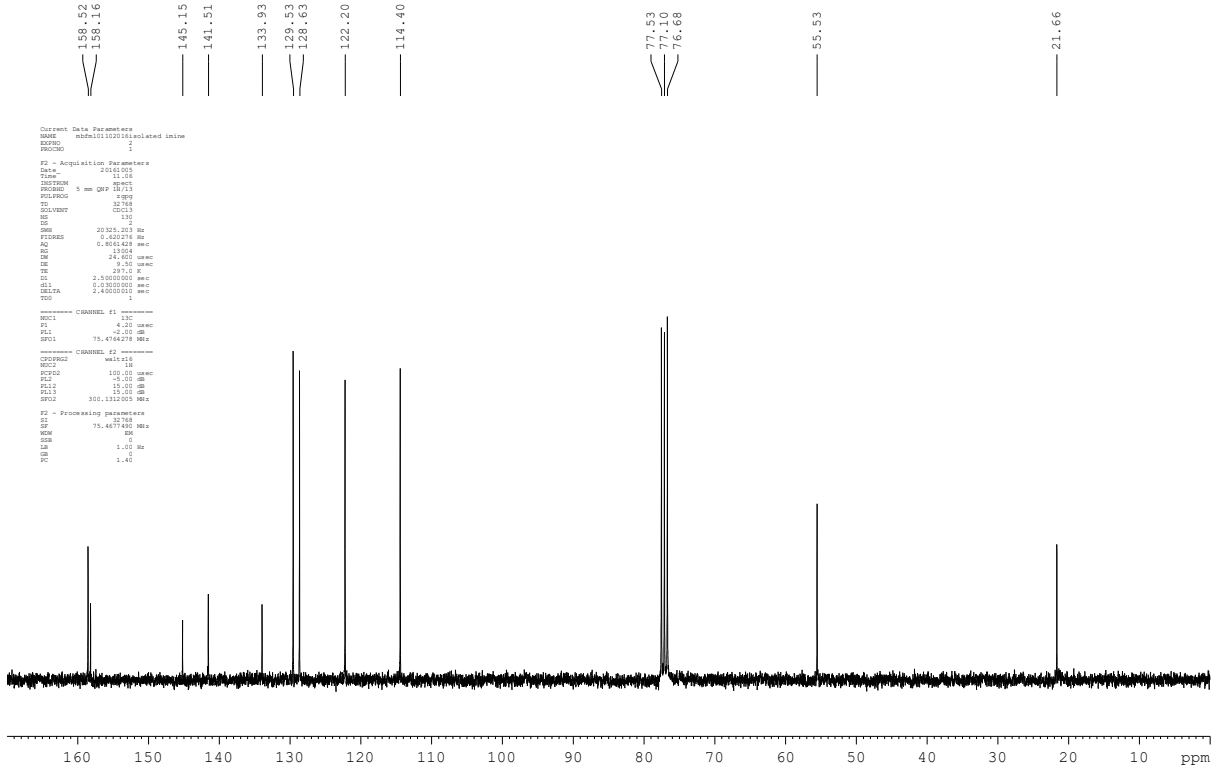


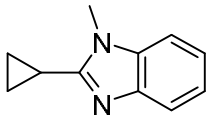



OCH3
N

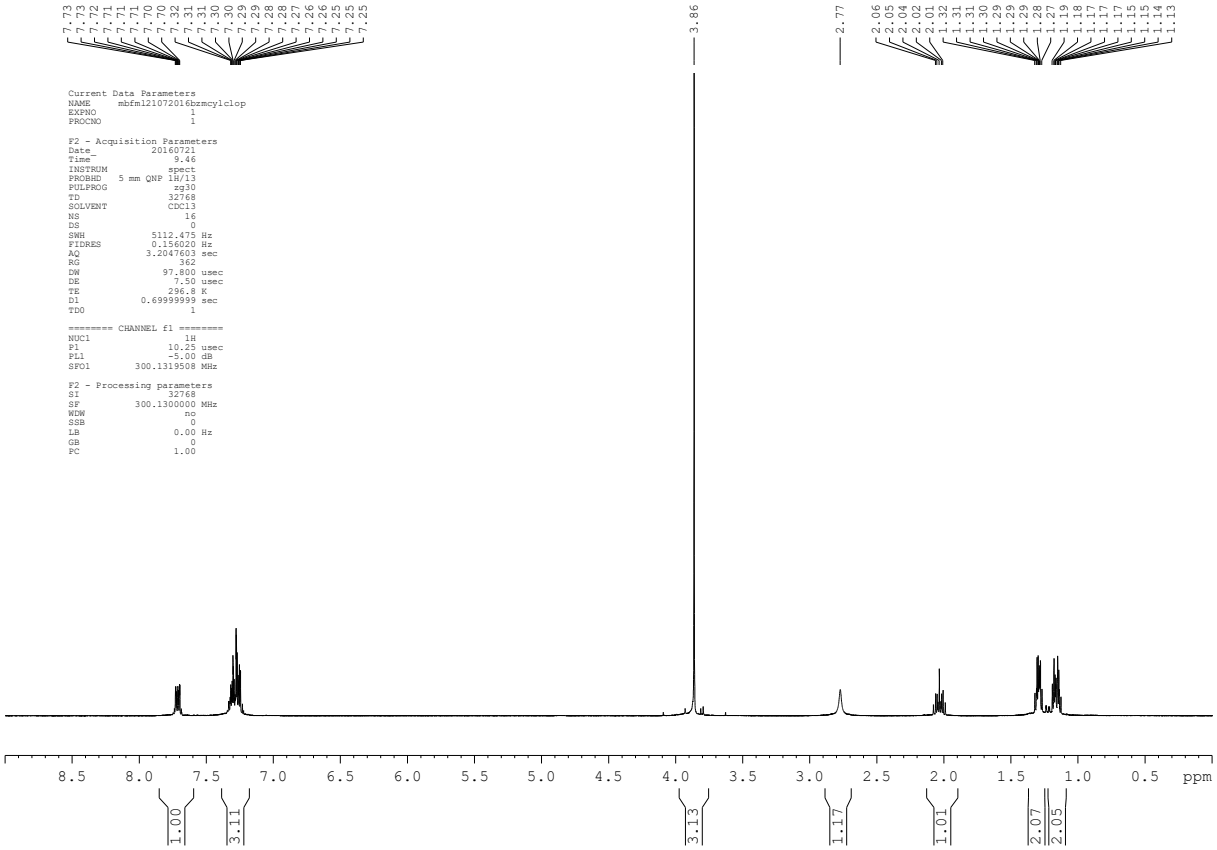
24o

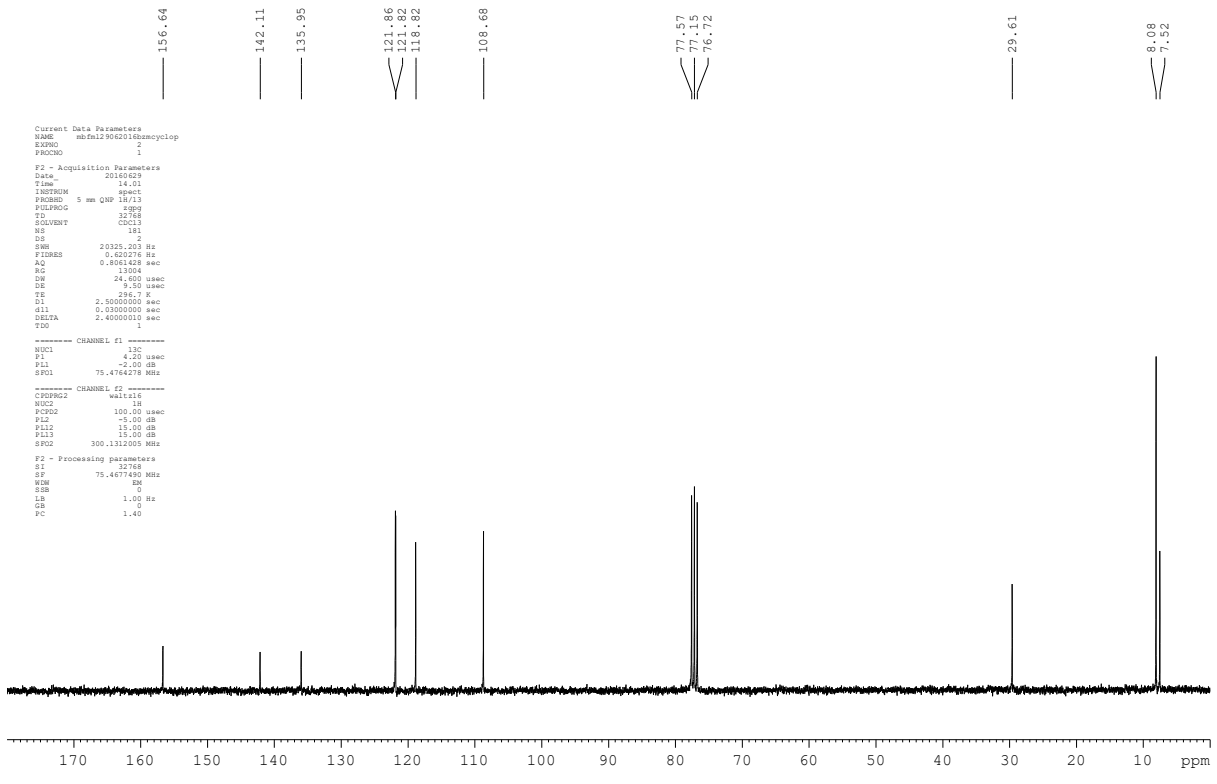

8.50
7.85
7.83
7.34
7.31
7.30
7.27
7.00
6.97
3.88
2.47
Current Data Parameters
NAME mbfm101102016isolated imine
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161005
Time 11.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 256
DW 97.800 usec
DE 7.50 usec
TE 296.6 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
ppm
1.00
2.02
4.15
2.01
3.06
3.09

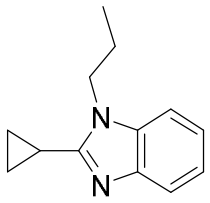

158.52
158.16
145.15
141.51
133.93
129.53
128.63
122.20
114.40
77.53
77.10
76.68
55.53
21.66
Current Data Parameters
NAME mbfm101102016isolated imine
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161005
Time 11.06
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 130
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 297.0 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.49000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312905 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



25a

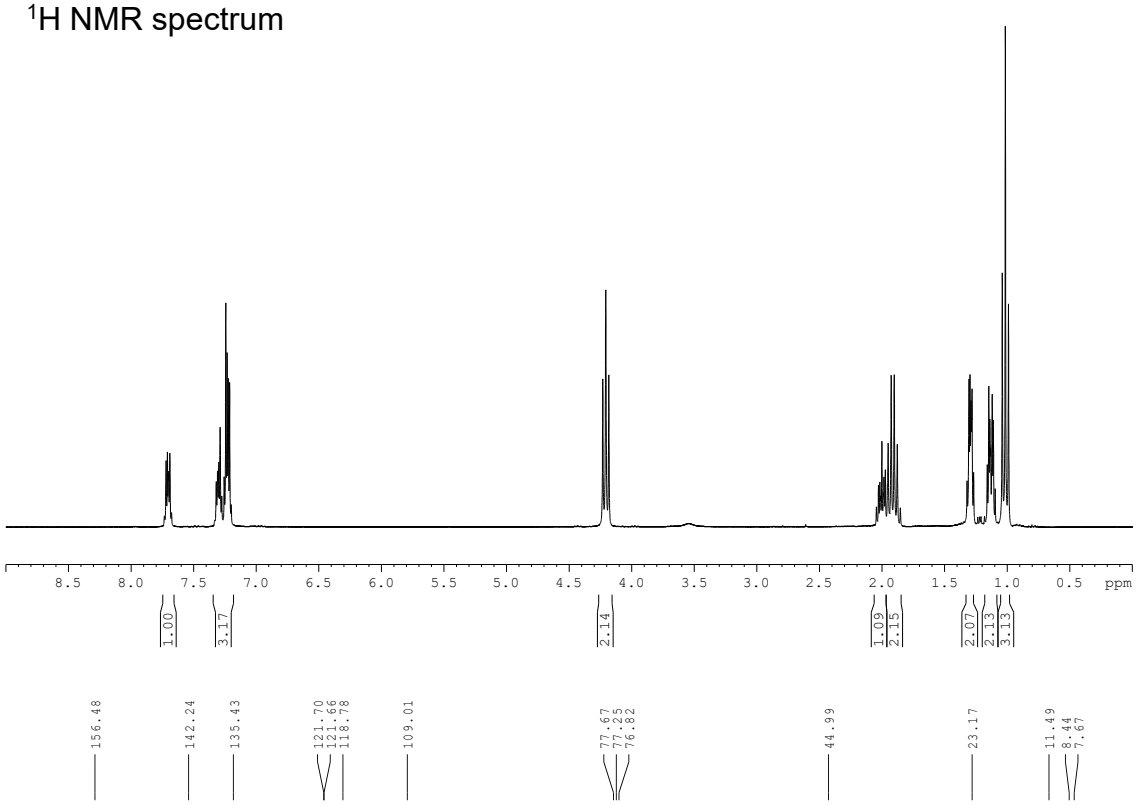

7.73 7.73 7.72 7.71 7.71 7.71 7.70 7.70 7.32 7.31 7.31 7.30 7.30 7.29 7.29 7.28 7.28 7.27 7.26 7.26 7.25 7.25 7.25
3.86
2.77
2.06 2.05 2.04 2.02 2.01 1.32 1.31 1.31 1.30 1.29 1.29 1.29 1.28 1.27 1.19 1.18 1.17 1.17 1.17 1.17 1.15 1.15 1.14 1.13
Current Data Parameters
NAME mbfm121072016bzmcylclop
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160721
Time 9.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 362
DW 97.800 usec
DE 7.50 usec
TE 296.8 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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 ppm
1.00 3.11 3.13 1.17 1.01 2.07 2.05


156.64
142.11
135.95
121.86
121.82
118.82
108.68
77.57
77.15
76.72
29.61
8.08
7.52
Current Data Parameters
NAME mbfm129062016bzmcyclop
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160629
Time 14.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 181
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 296.7 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677480 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

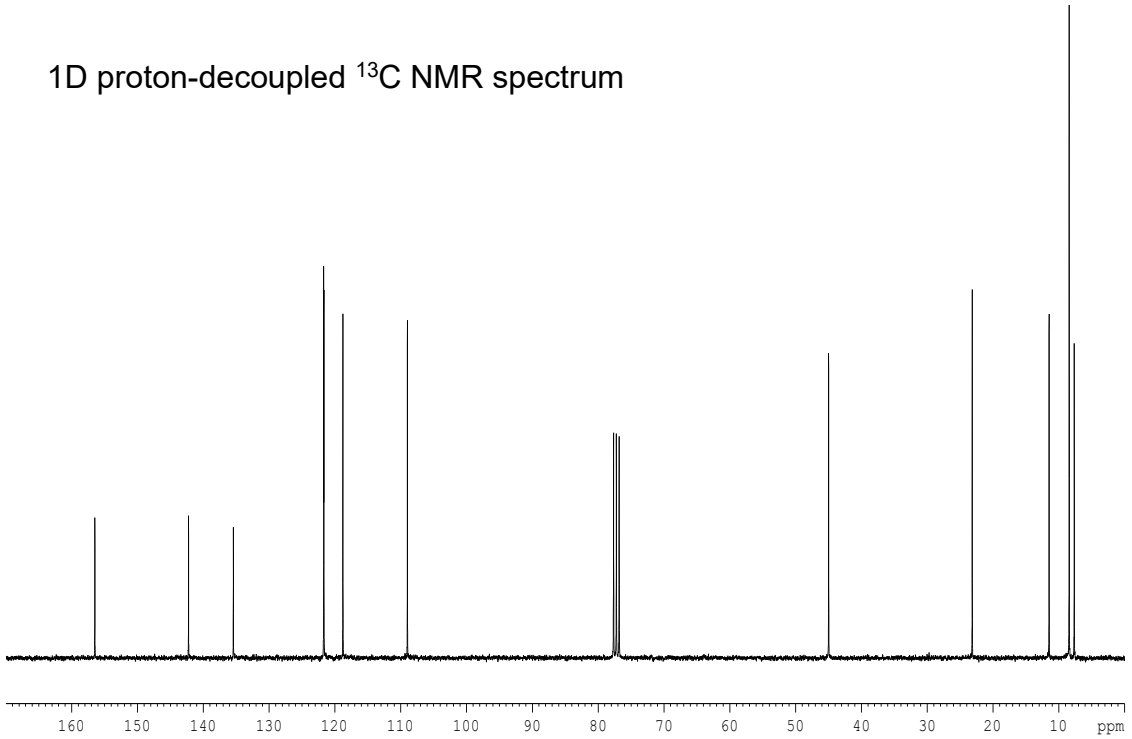


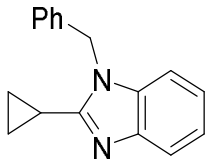
25b

^{1}H NMR spectrum



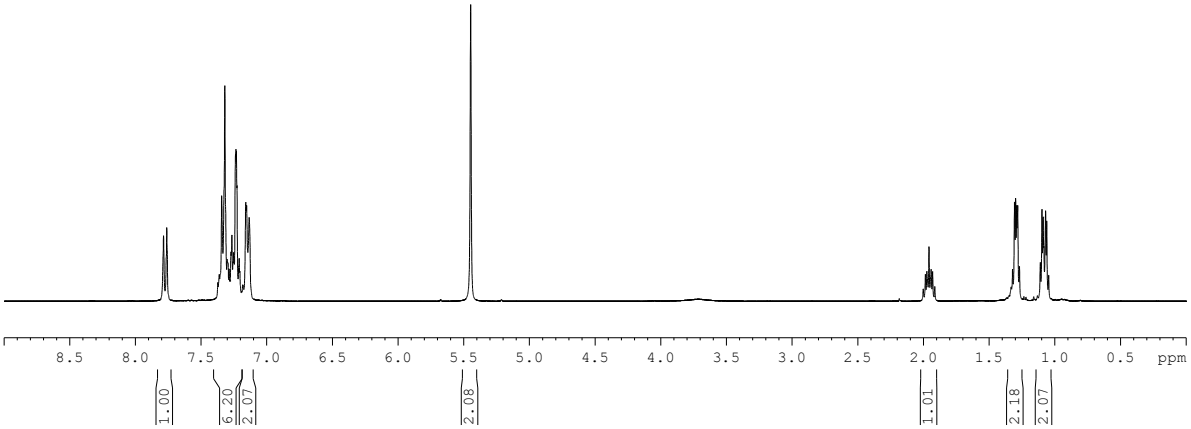
1D proton-decoupled ^{13}C NMR spectrum

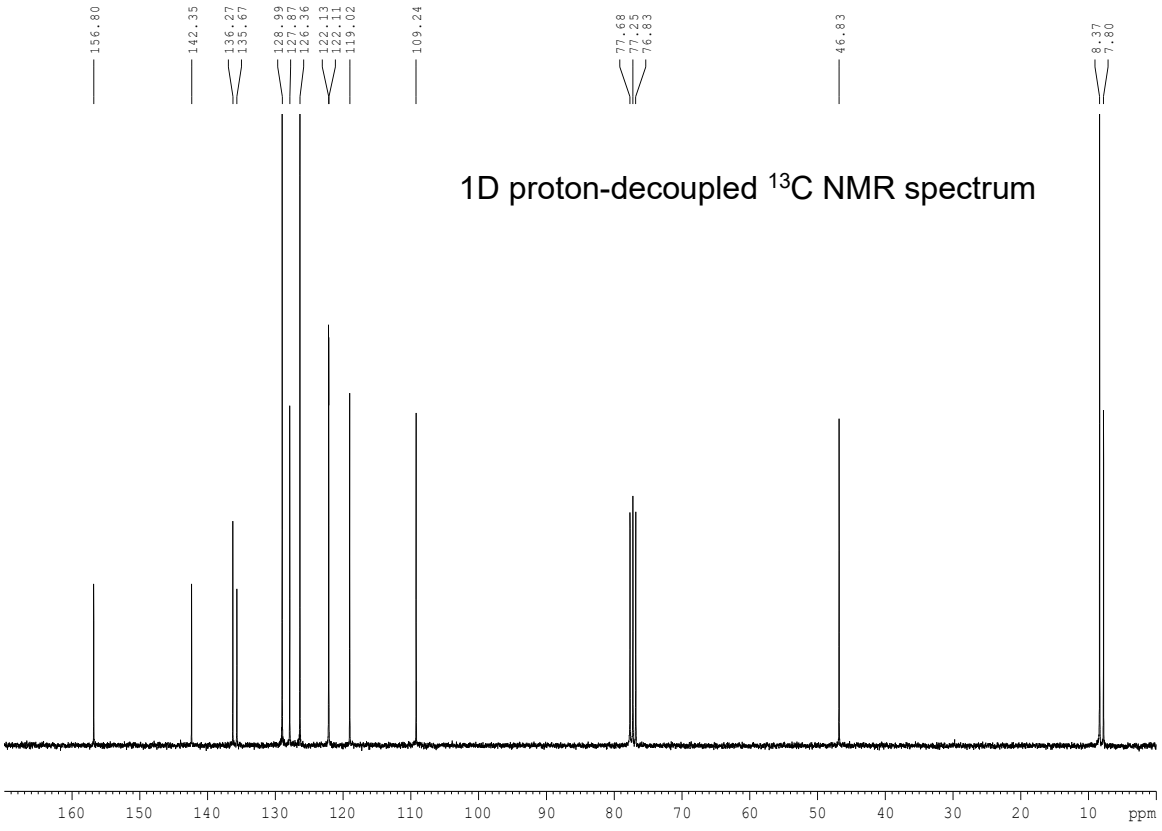




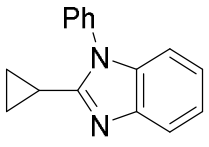
25c

^{1}H NMR spectrum

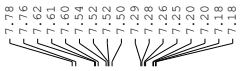


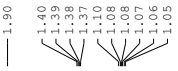


1D proton-decoupled ^{13}C NMR spectrum

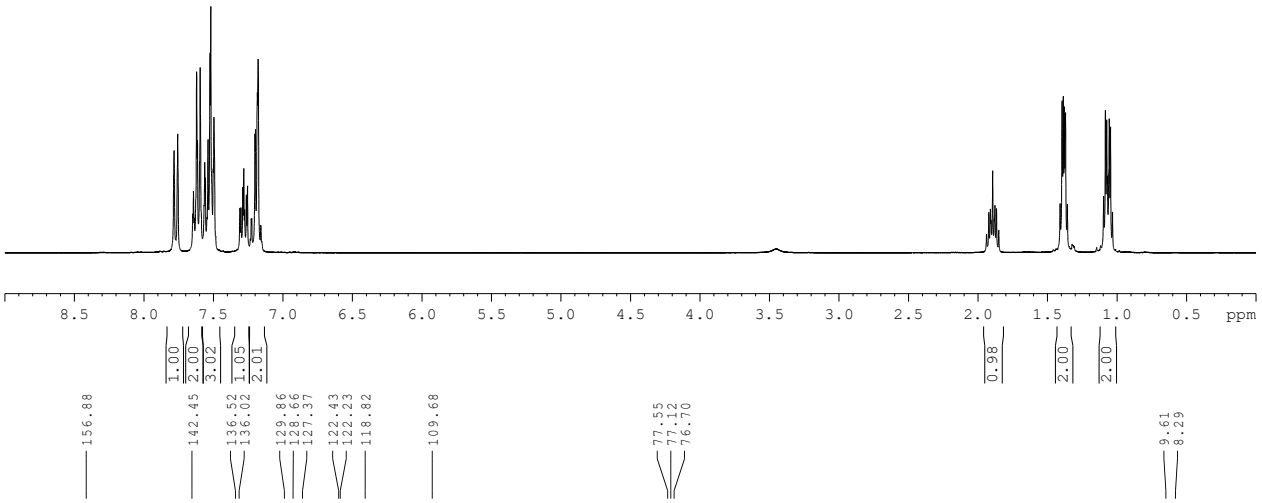


25d

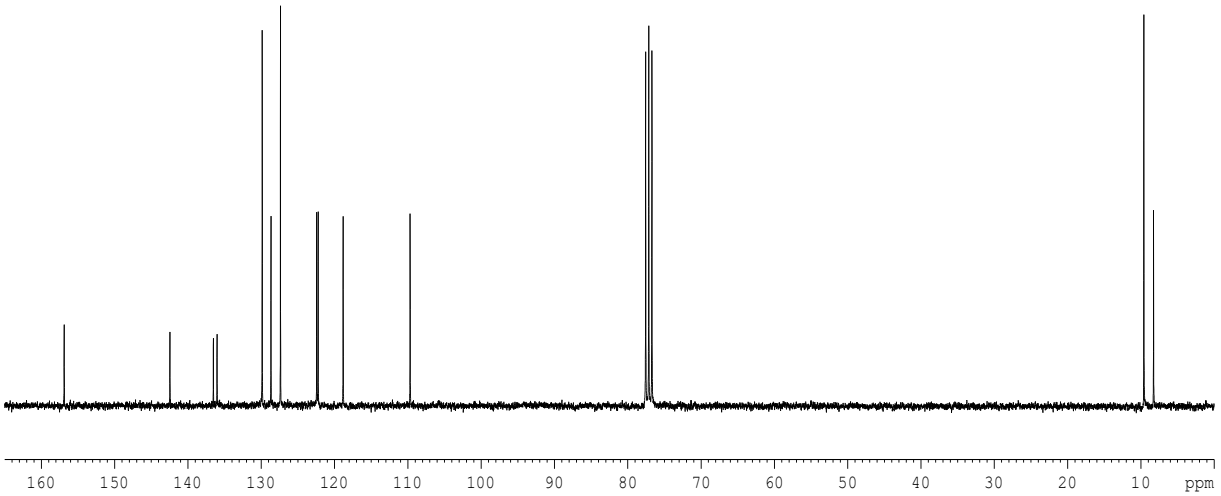


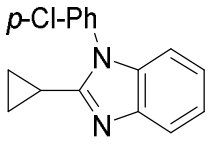


^{1}H NMR spectrum

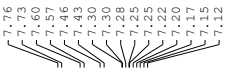


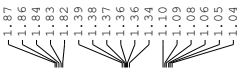
1D proton-decoupled ^{13}C NMR spectrum



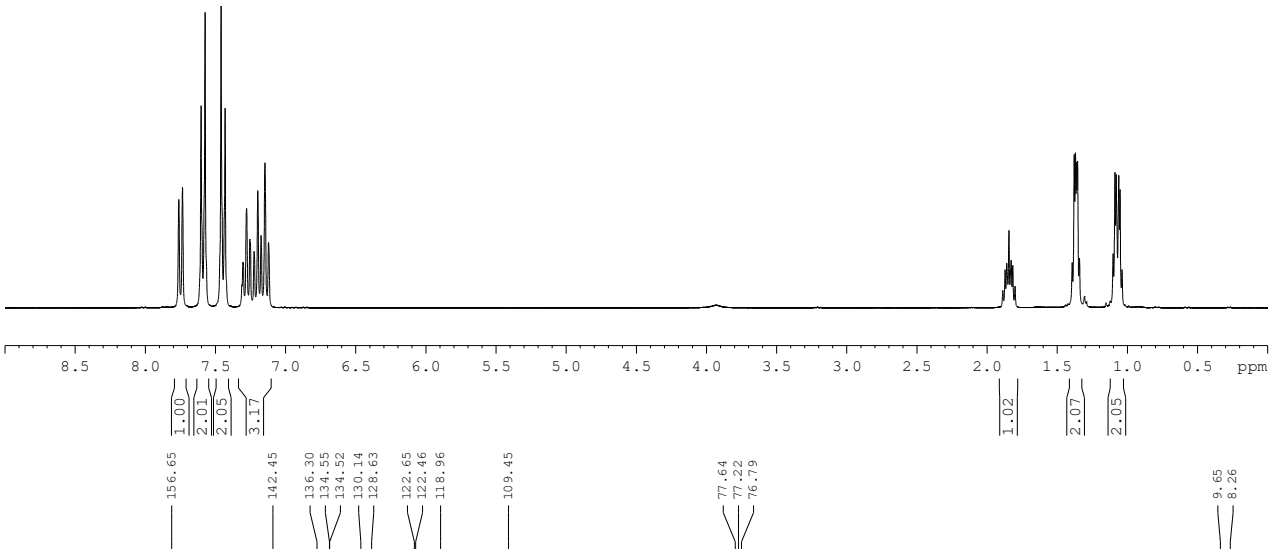


25e

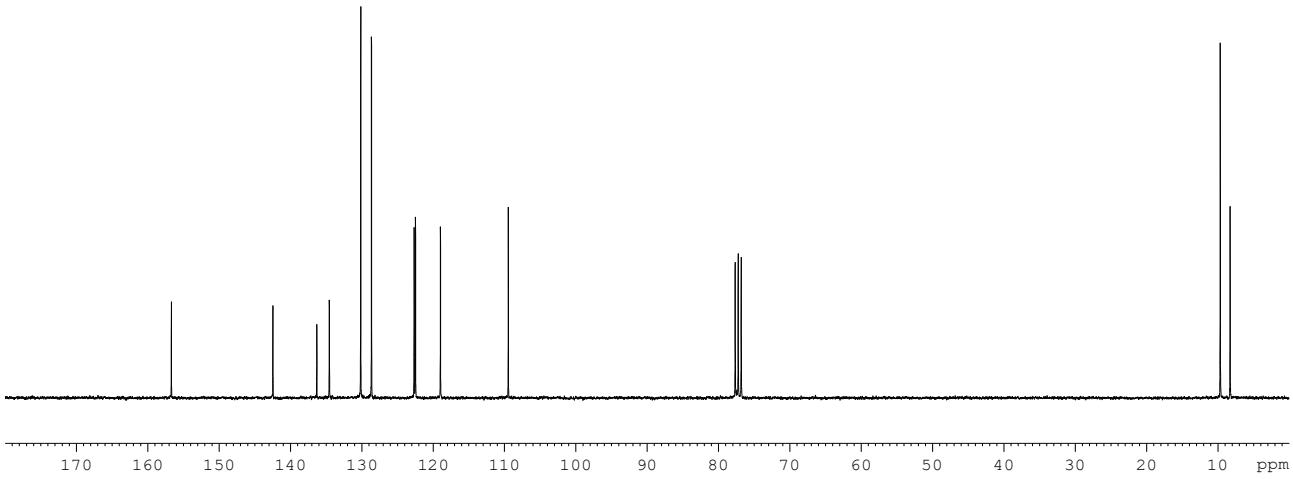


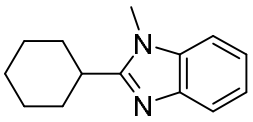


^{1}H NMR spectrum

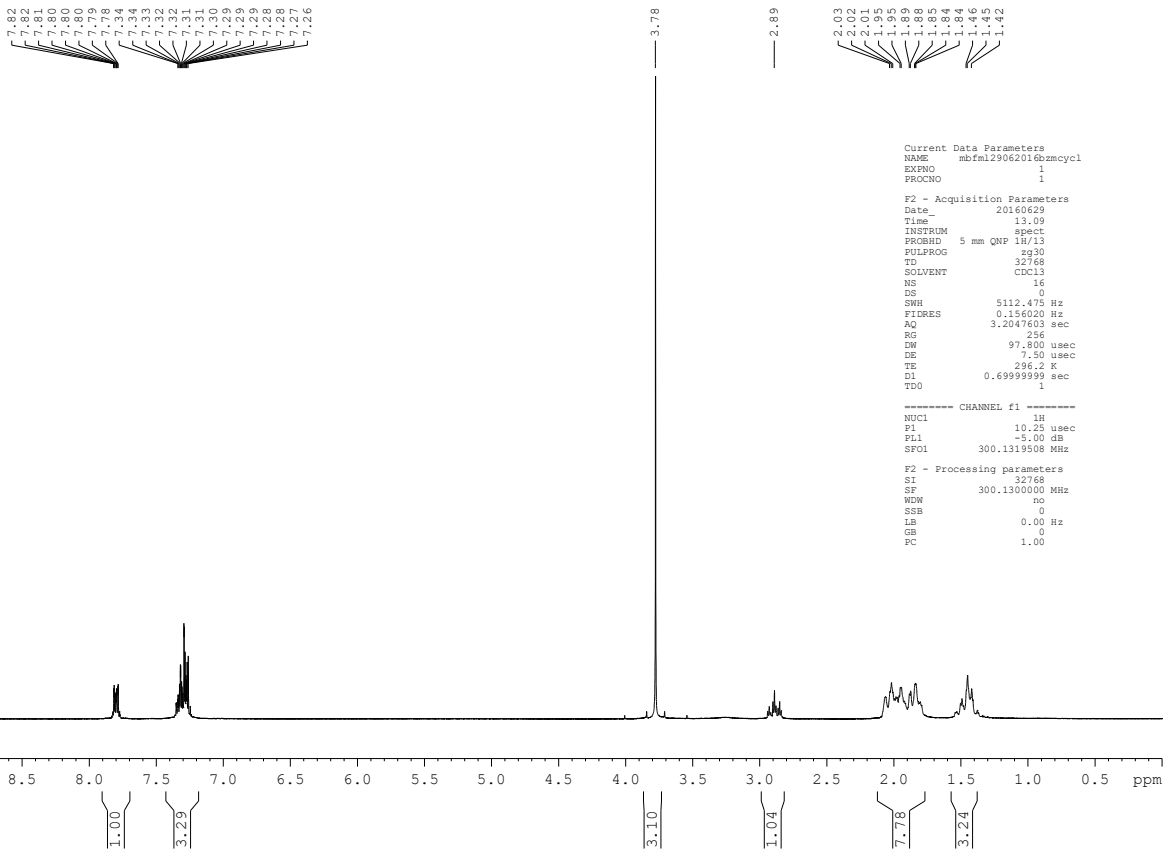


1D proton-decoupled ^{13}C NMR spectrum





25f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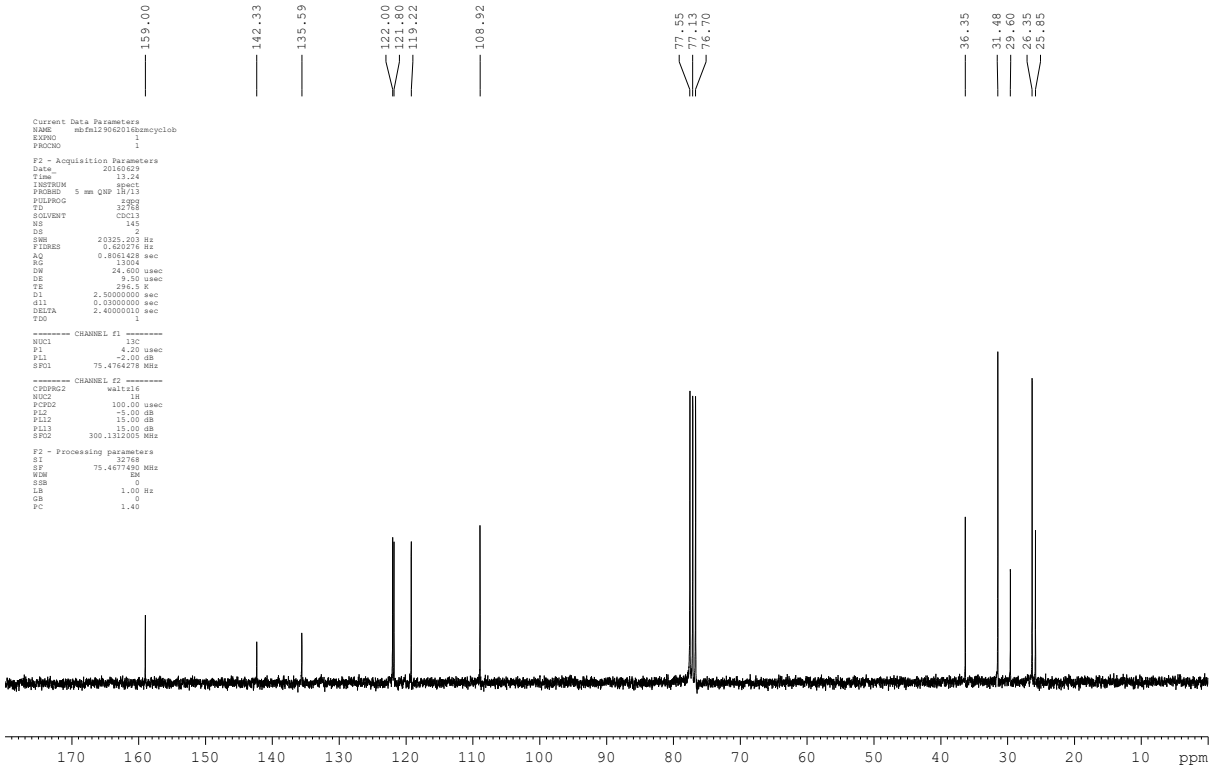
3.78
2.89
2.03 2.02 2.01 1.95 1.95 1.89 1.88 1.85 1.84 1.84 1.46 1.45 1.42
Current Data Parameters
NAME mbfml29062016bzmcycl
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160629
Time 13.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 256
DW 97.800 usec
DE 7.50 usec
TE 296.2 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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 ppm
1.00 3.29 3.10 1.04 7.78 3.24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135.59
122.00
121.80
119.22
108.92
77.55
77.13
76.70
36.35
31.48
29.60
26.35
25.85
Current Data Parameters
NAME mbfml29062016bzmcyclob
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160629
Time 13.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 145
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 296.5 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
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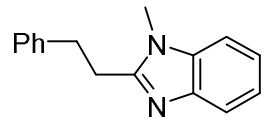
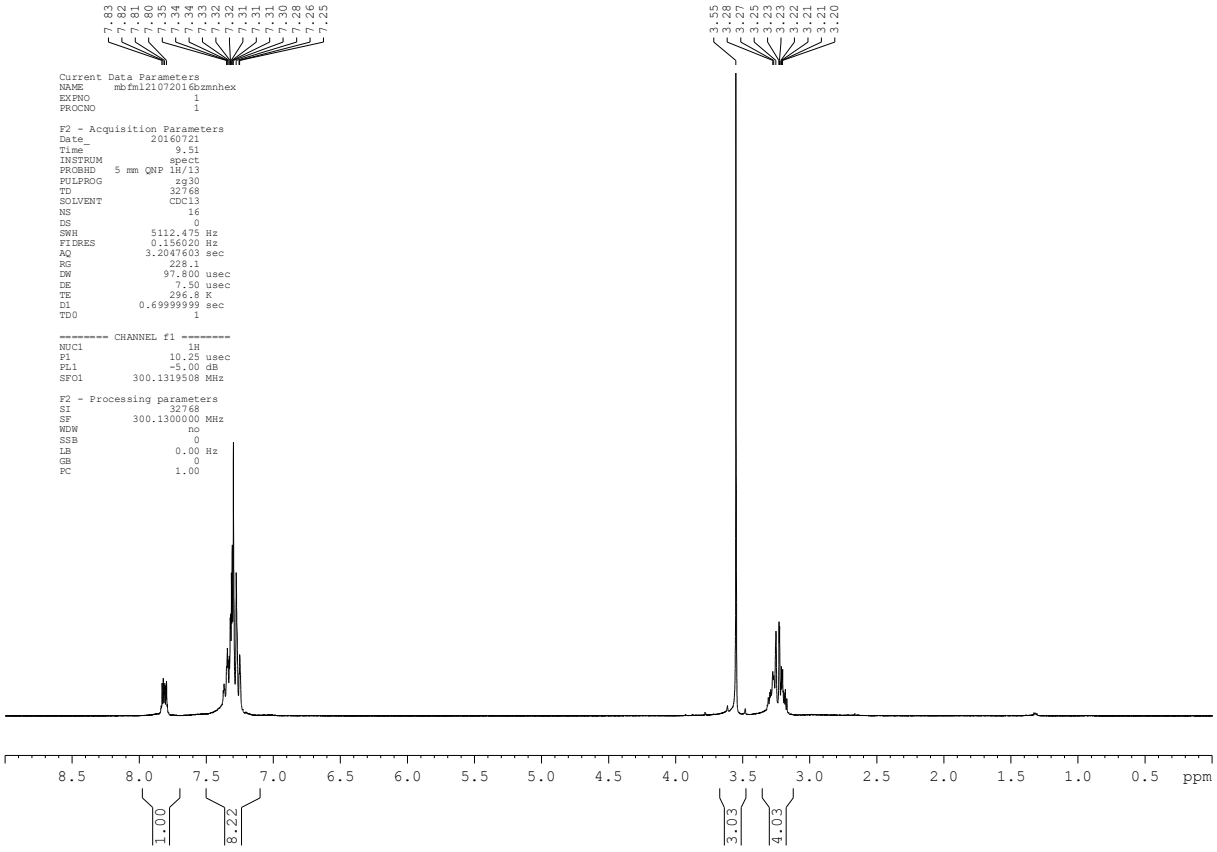
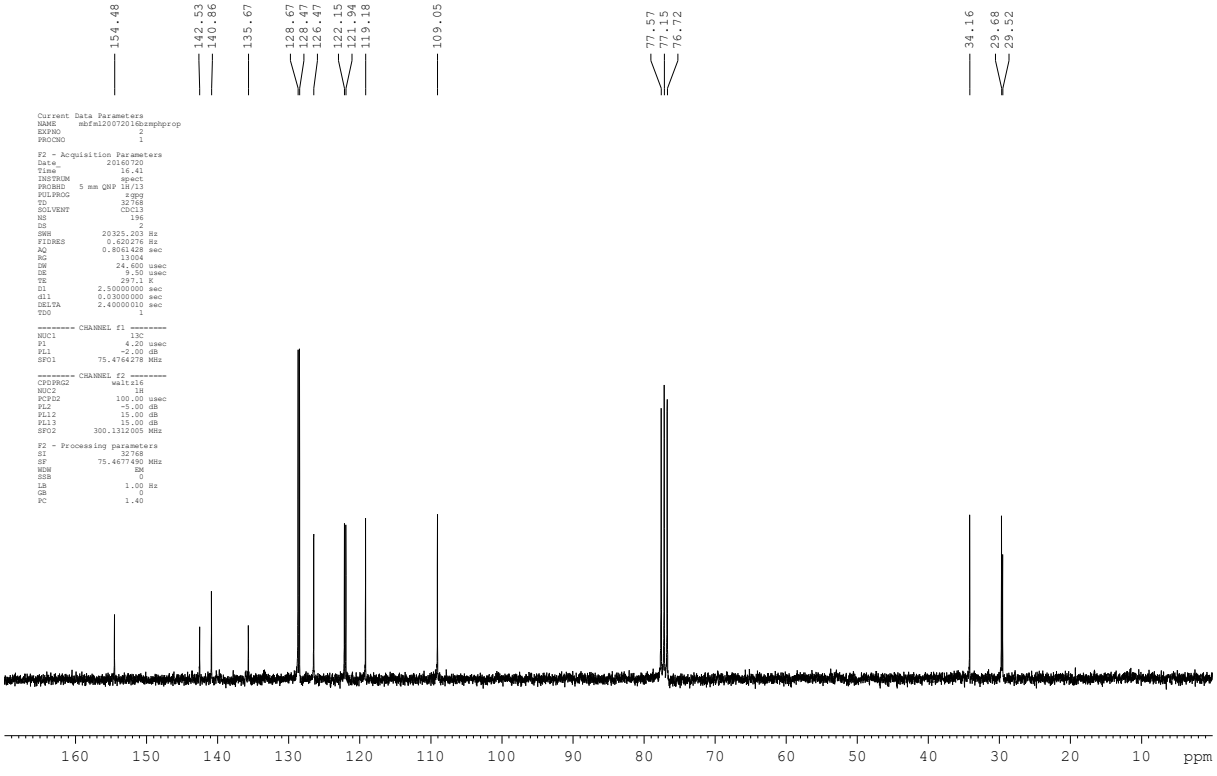
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Ph
N
N

25g


7.83
7.82
7.81
7.80
7.35
7.34
7.33
7.32
7.32
7.31
7.31
7.30
7.28
7.26
7.25
3.55
3.28
3.27
3.25
3.23
3.23
3.22
3.21
3.21
3.20
Current Data Parameters
NAME mbfm121072016bzmnhex
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160721
Time 9.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 228.1
DW 97.800 usec
DE 7.50 usec
TE 296.8 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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
ppm
1.00
8.22
3.03
4.03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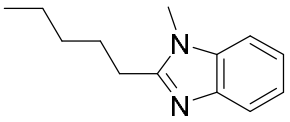
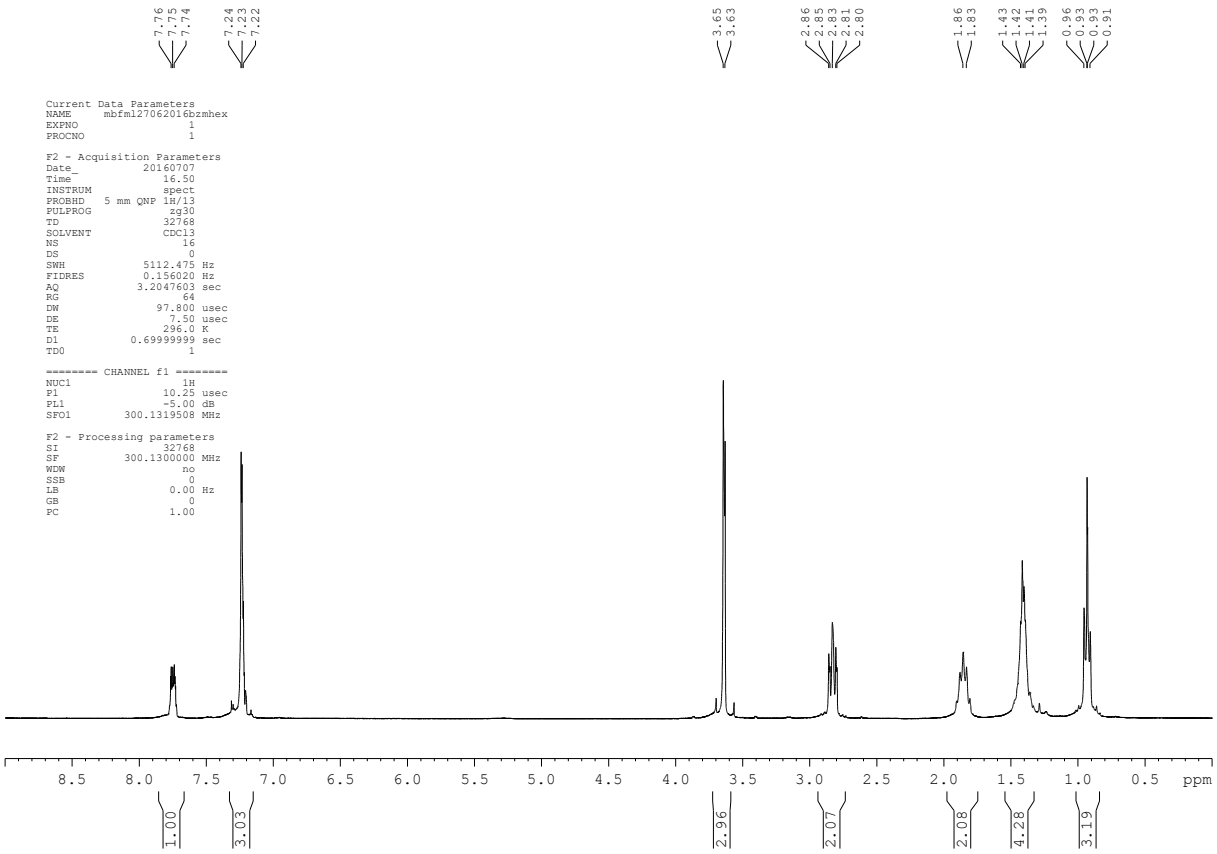
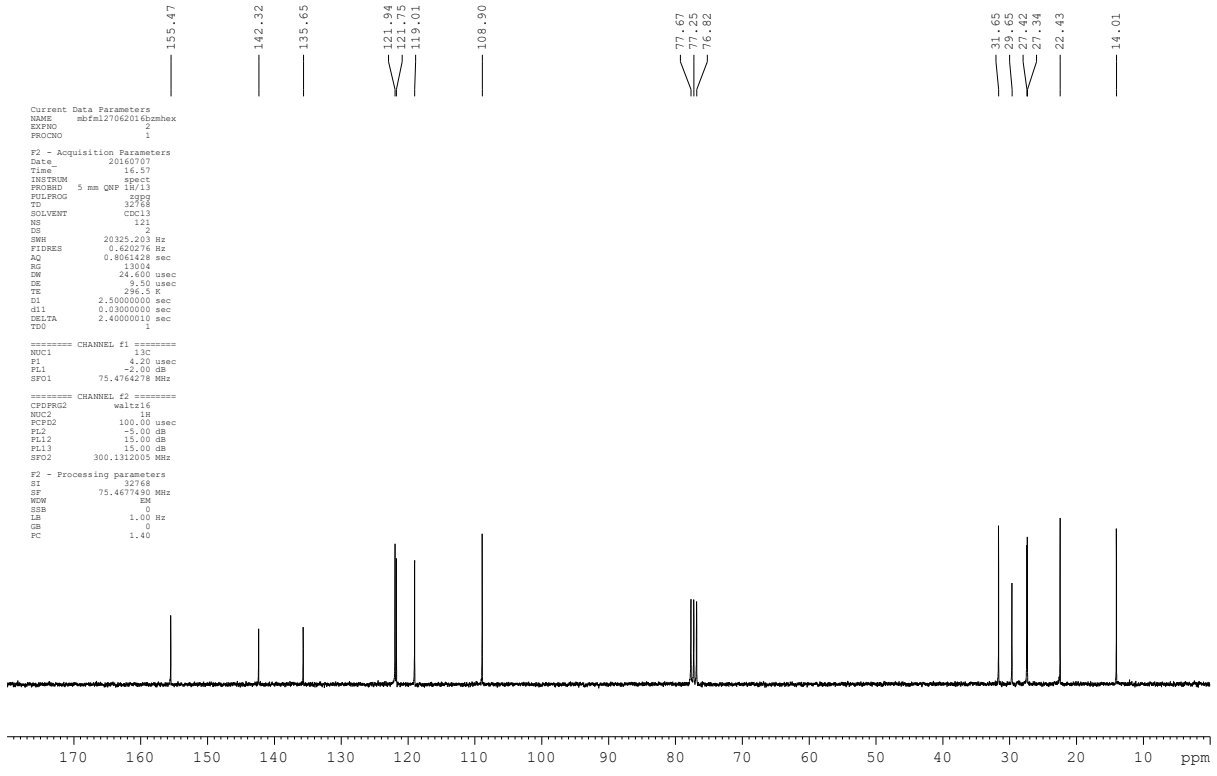
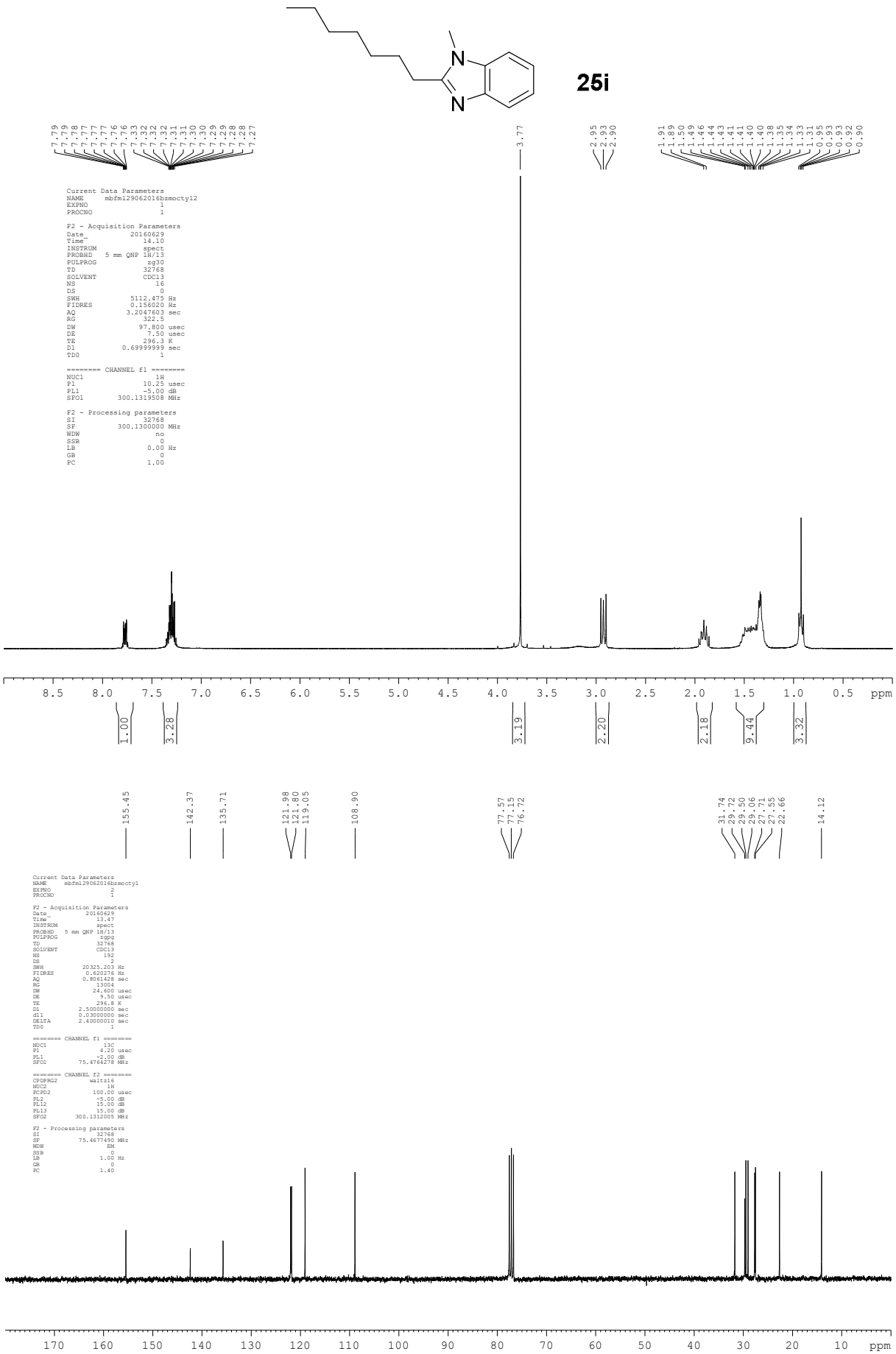
142.53
140.86
135.67
128.67
128.47
126.47
122.15
121.94
119.18
109.05
77.57
77.15
76.72
34.16
29.68
29.52
Current Data Parameters
NAME mbfm120072016bzmphprop
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160720
Time 16.41
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 196
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 297.1 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

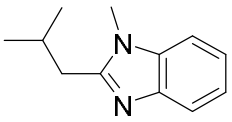


25h

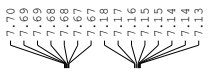

7.76
7.75
7.74
7.24
7.23
7.22
3.65
3.63
2.86
2.85
2.83
2.81
2.80
1.86
1.83
1.43
1.42
1.41
1.39
0.96
0.93
0.93
0.91
Current Data Parameters
NAME mbfml27062016bzmhex
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160707
Time 16.50
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 64
DW 97.800 usec
DE 7.50 usec
TE 296.0 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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 ppm
1.00
3.03
2.96
2.07
2.08
4.28
3.19


155.47
142.32
135.65
121.94
121.75
119.01
108.90
77.67
77.25
76.82
31.65
29.65
27.42
27.34
22.43
14.01
Current Data Parameters
NAME mbfml27062016bzmhex
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160707
Time 16.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 121
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 296.5 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


25i
7.79 7.78 7.77 7.77 7.76 7.33 7.32 7.32 7.31 7.31 7.30 7.29 7.28 7.28 7.27
3.77
2.95 2.93 2.90
1.91 1.89 1.50 1.49 1.46 1.44 1.43 1.41 1.41 1.40 1.40 1.38 1.35 1.34 1.33 1.31 0.95 0.93 0.93 0.92 0.90
Current Data Parameters
NAME mbfm129062016bzmoctyl2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160629
Time 14.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 322.5
DW 97.800 usec
DE 7.50 usec
TE 296.3 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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 ppm
1.00 3.28 3.19 2.20 2.18 9.44 3.32
155.45 142.37 135.71 121.98 121.80 119.05 108.90 77.57 77.15 76.72 31.74 29.72 29.50 29.06 27.71 27.55 22.66 14.12
Current Data Parameters
NAME mbfm129062016bzmoctyl
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160629
Time 13.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 192
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 296.8 K
D1 2.50000000 sec
D11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



25j

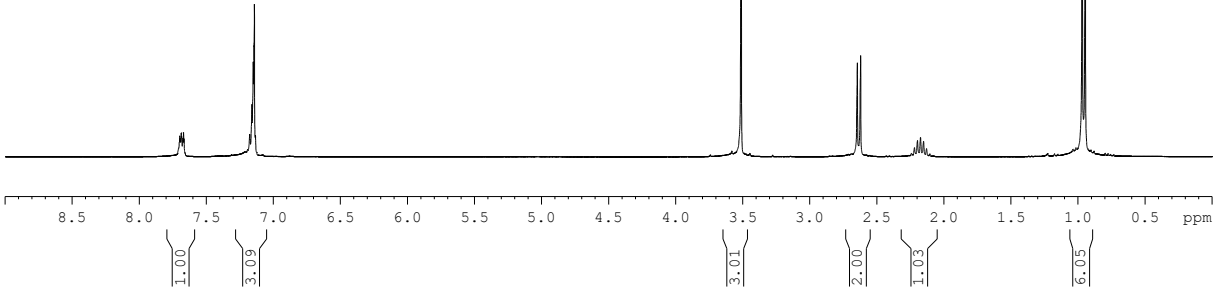



```
Current Data Parameters
NAME      mbfml28072016bzmisop
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20160708
Time                   9.51
INSTRUM               spect
PROBHD    5 mm QNP 1H/13
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                5112.475 Hz
FIDRES            0.156020 Hz
AQ                3.2047603 sec
RG                       32
DW                  97.800 usec
DE                   7.50 usec
TE                  296.3 K
D1              0.69999999 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.25 usec
PL1                   -5.00 dB
SFO1          300.1319508 MHz

F2 - Processing parameters
SI                    32768
SF            300.1300000 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00
```

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Current Data Parameters
NAME      mbfml28072016bzmisop
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20160708
Time                   9.56
INSTRUM               spect
PROBHD    5 mm QNP 1H/13
PULPROG              zgpg
TD                    32768
SOLVENT               CDCl3
NS                       82
DS                        2
SWH               20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                    13004
DW                  24.600 usec
DE                   9.50 usec
TE                  296.8 K
D1              2.50000000 sec
d11             0.03000000 sec
DELTA           2.40000010 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    13C
P1                    4.20 usec
PL1                  -2.00 dB
SFO1          75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2               100.00 usec
PL2                   -5.00 dB
PL12                  15.00 dB
PL13                  15.00 dB
SFO2          300.1312005 MHz

F2 - Processing parameters
SI                    32768
SF            75.4677490 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
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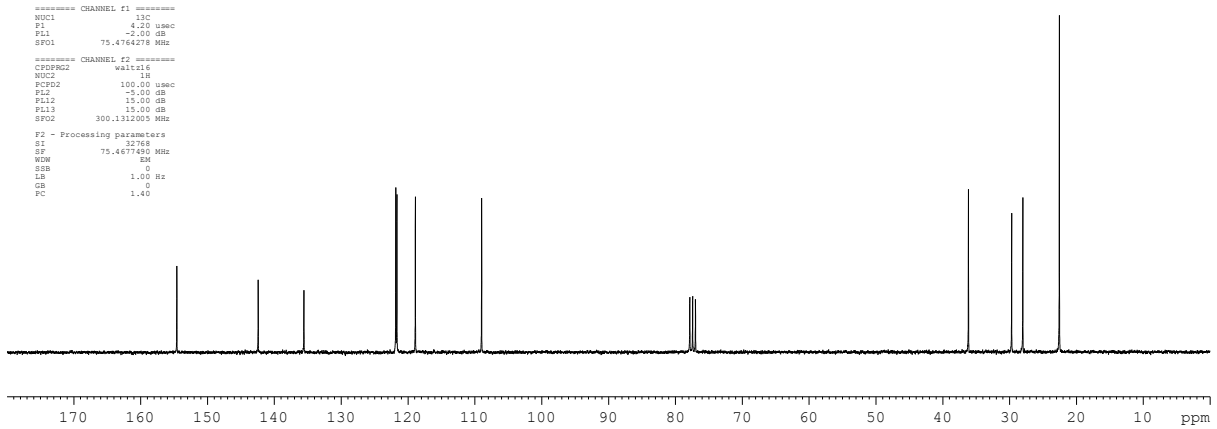
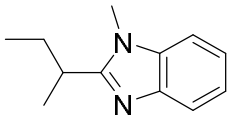
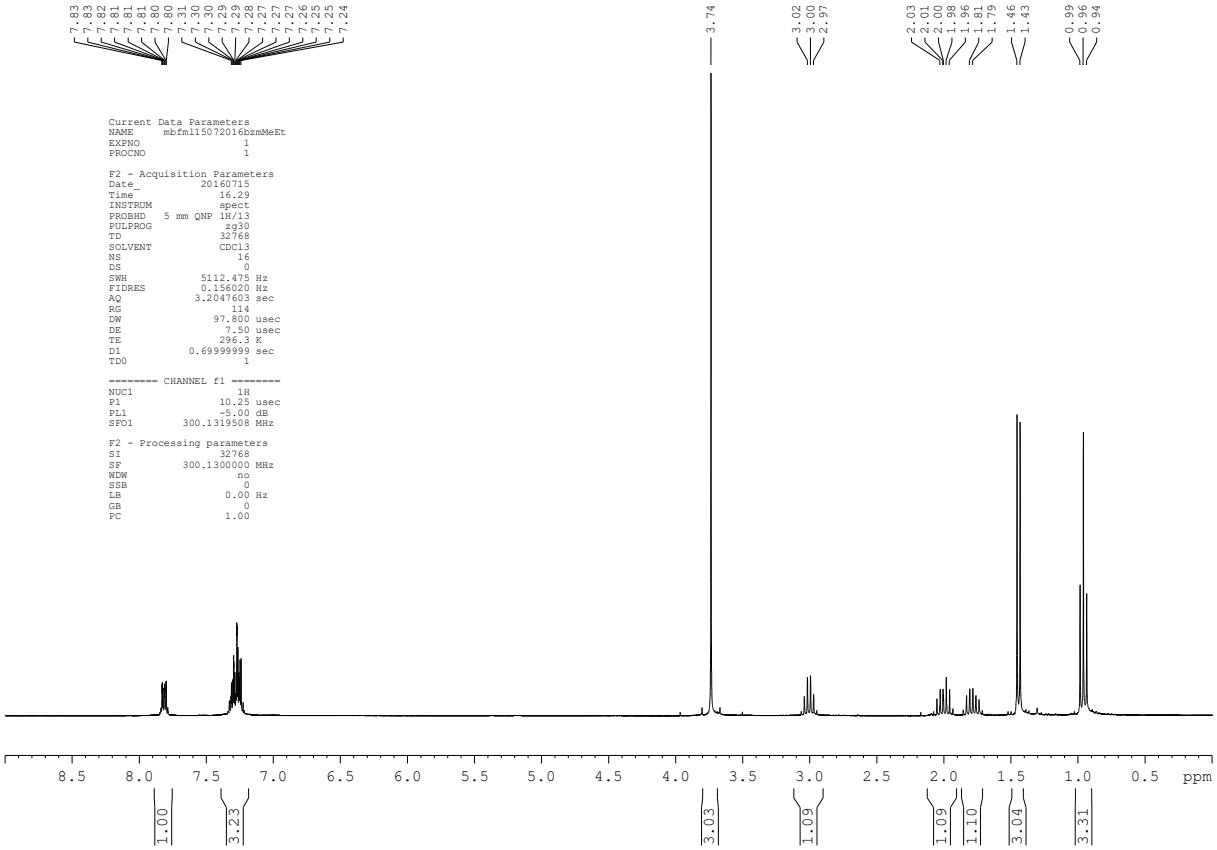




25k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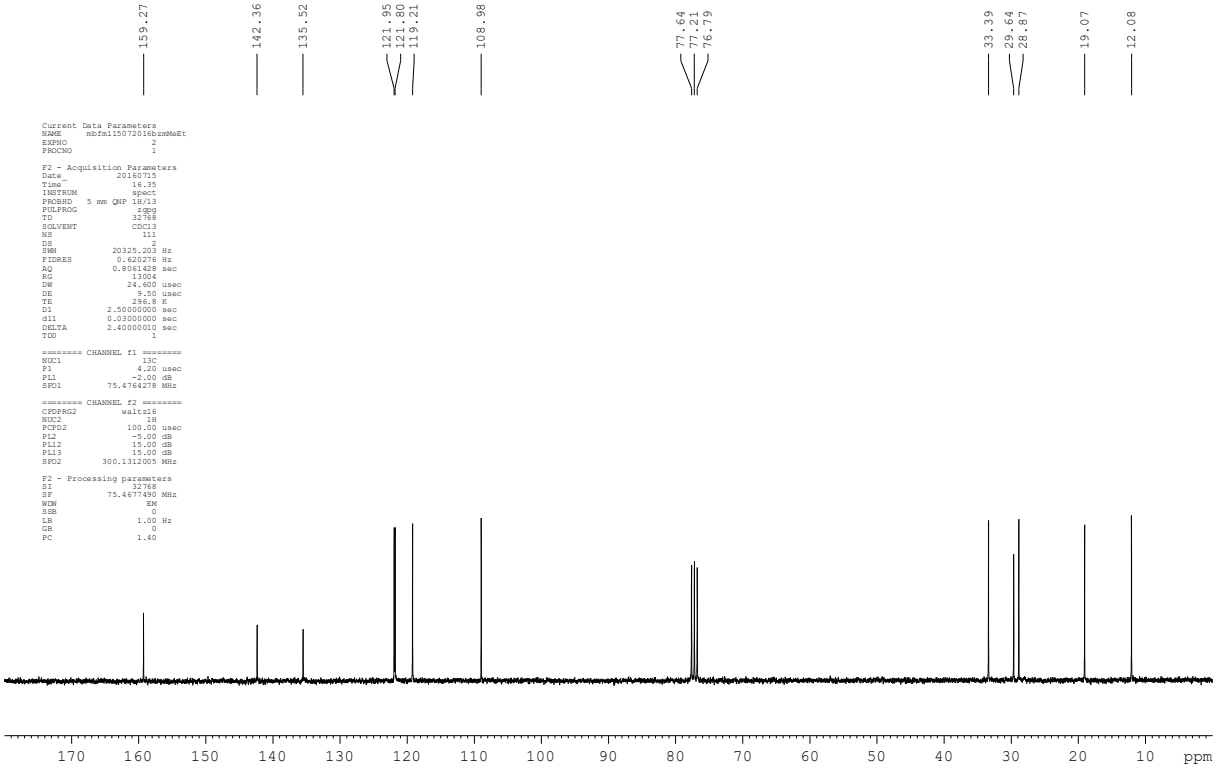
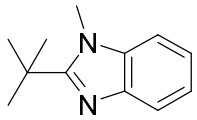
3.74
3.02 3.00 2.97
2.03 2.01 2.00 1.98 1.96 1.81 1.79
1.46 1.43
0.99 0.96 0.94
Current Data Parameters
NAME mbfml15072016bzmMeEt
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160715
Time 16.29
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5112.475 Hz
FIDRES 0.156020 Hz
AQ 3.2047603 sec
RG 114
DW 97.800 usec
DE 7.50 usec
TE 296.3 K
D1 0.69999999 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.25 usec
PL1 -5.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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 ppm
1.00
3.23
3.03
1.09
1.09
1.10
3.04
3.31


159.27
142.36
135.52
121.95 121.80 119.21
108.98
77.64 77.21 76.79
33.39
29.64
28.87
19.07
12.08
Current Data Parameters
NAME mbfml15072016bzmMeEt
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160715
Time 16.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 111
DS 2
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 13004
DW 24.600 usec
DE 9.50 usec
TE 296.8 K
D1 2.50000000 sec
d11 0.03000000 sec
DELTA 2.40000010 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 4.20 usec
PL1 -2.00 dB
SFO1 75.4764278 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -5.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



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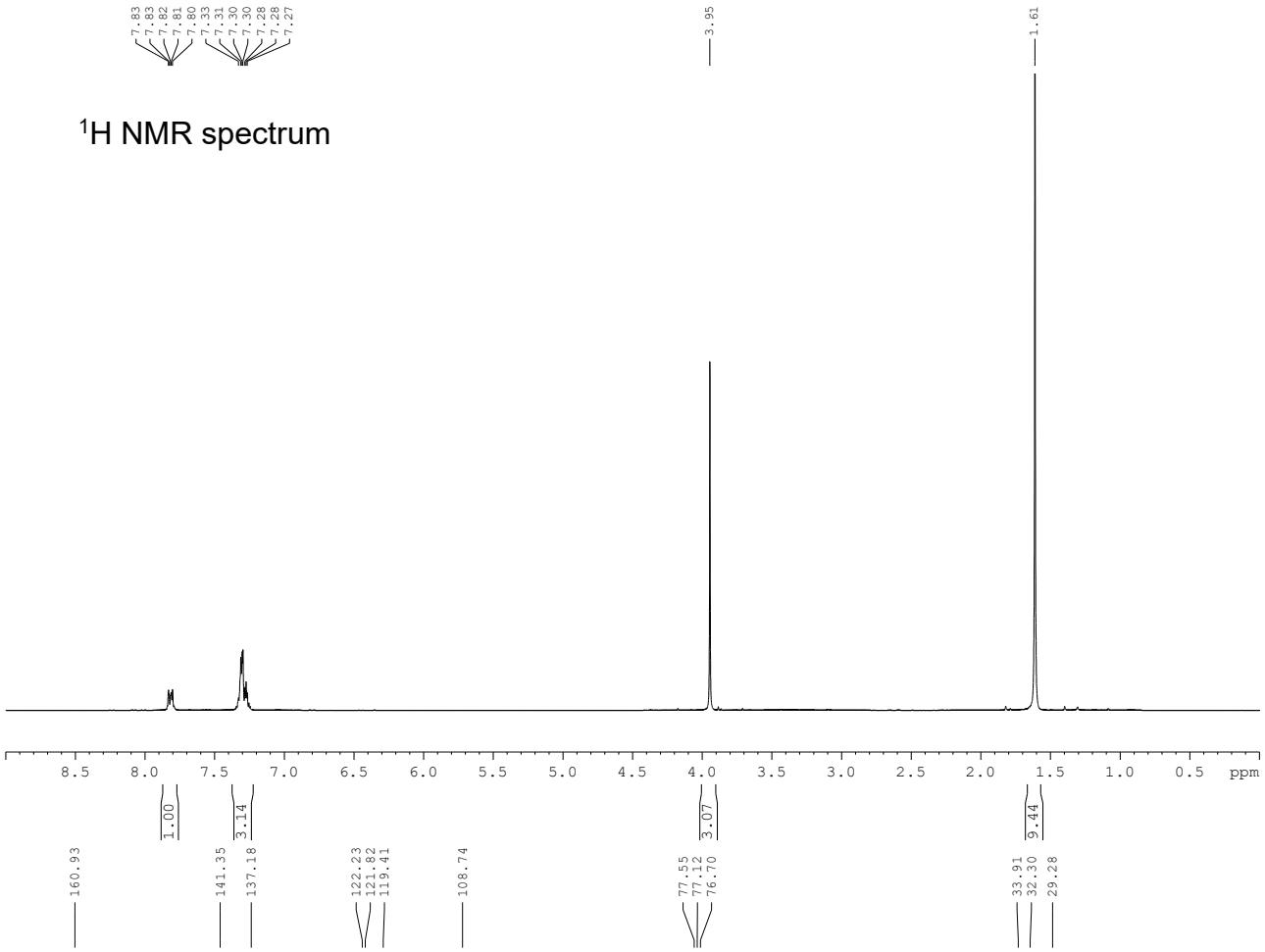
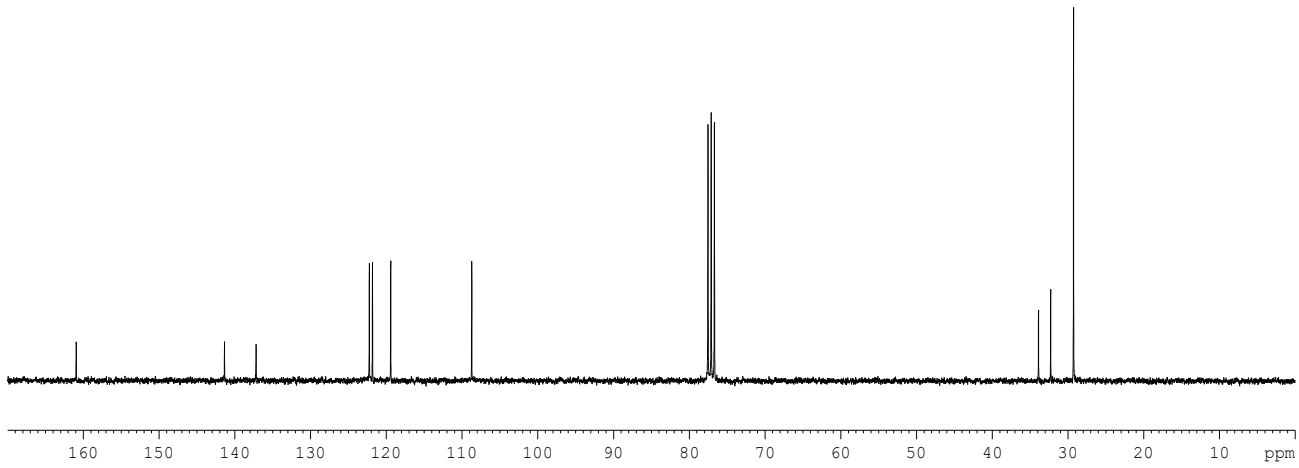
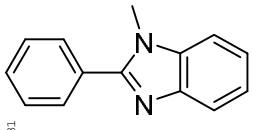


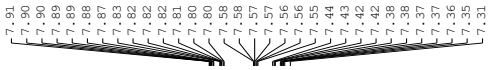
^{1}H NMR spectrum



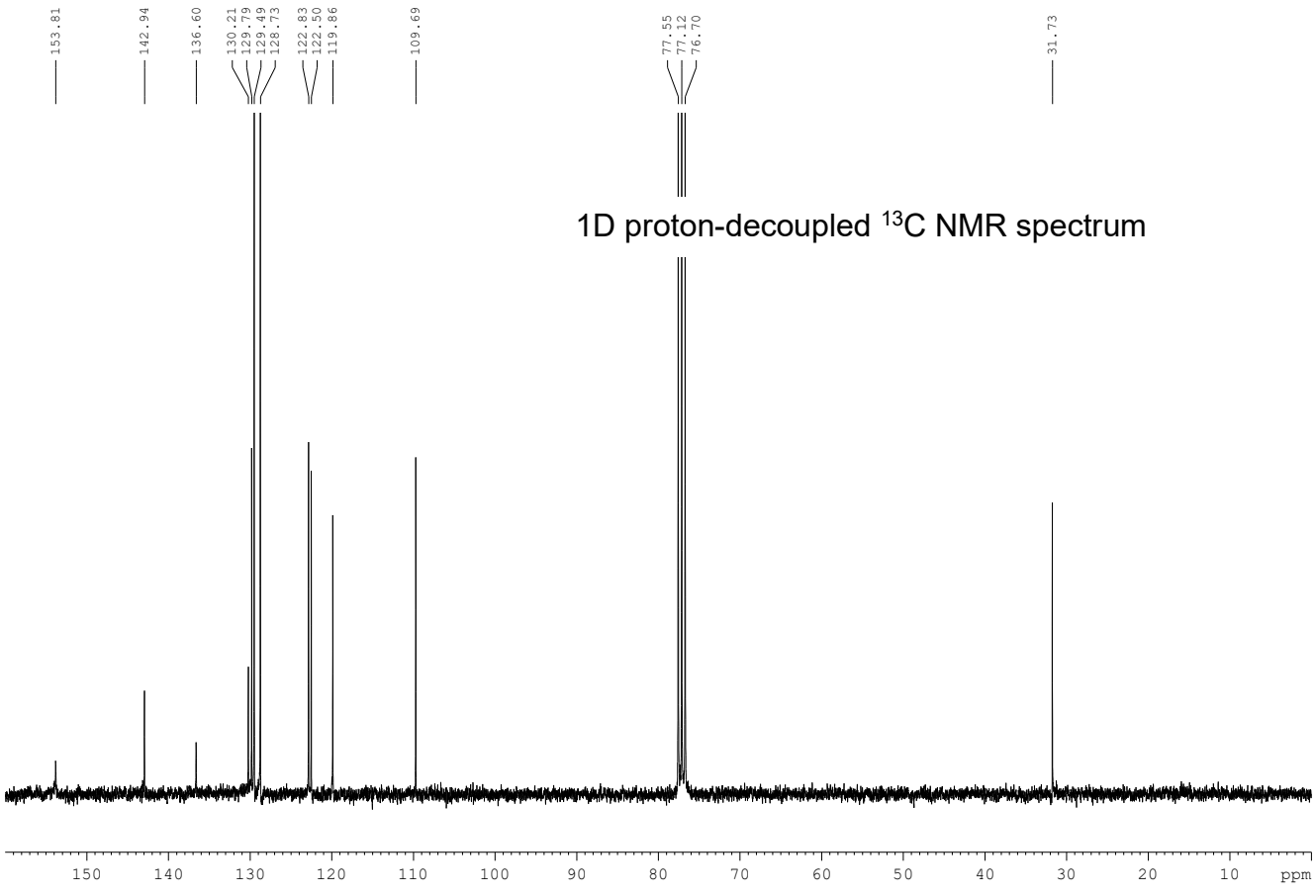
1D proton-decoupled ^{13}C NMR spectrum



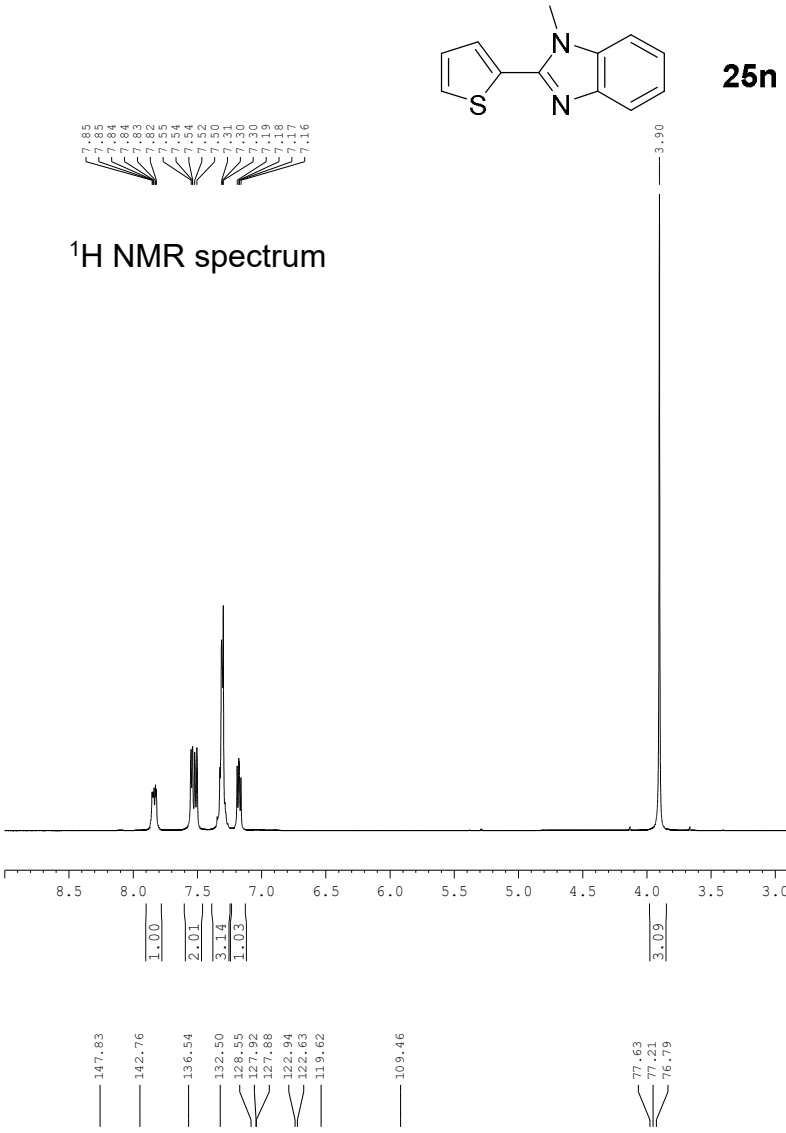
25m



^{1}H NMR spectrum

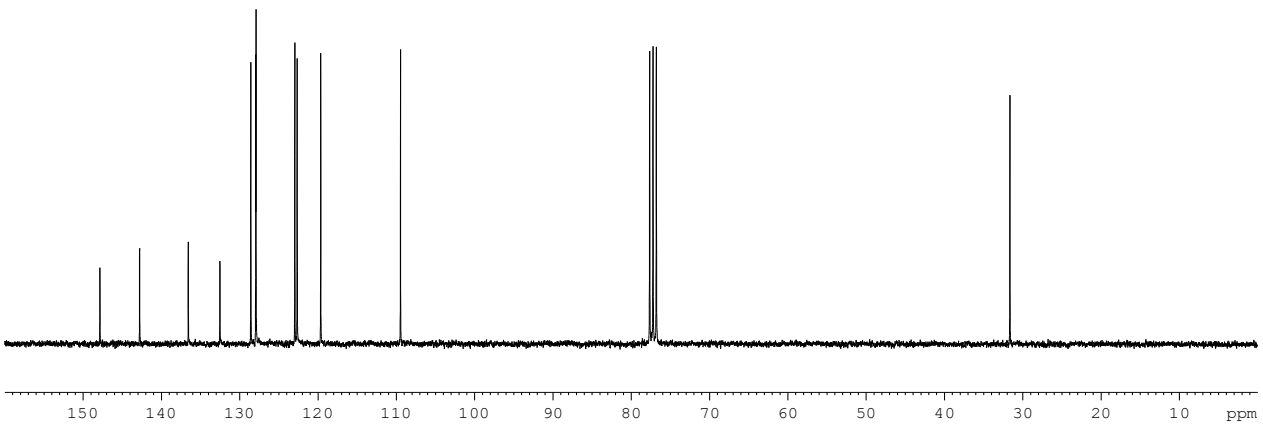


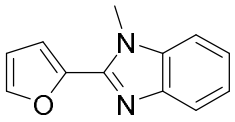
1D proton-decoupled ^{13}C NMR spectrum



^{1}H NMR spectrum

1D proton-decoupled ^{13}C NMR spectrum





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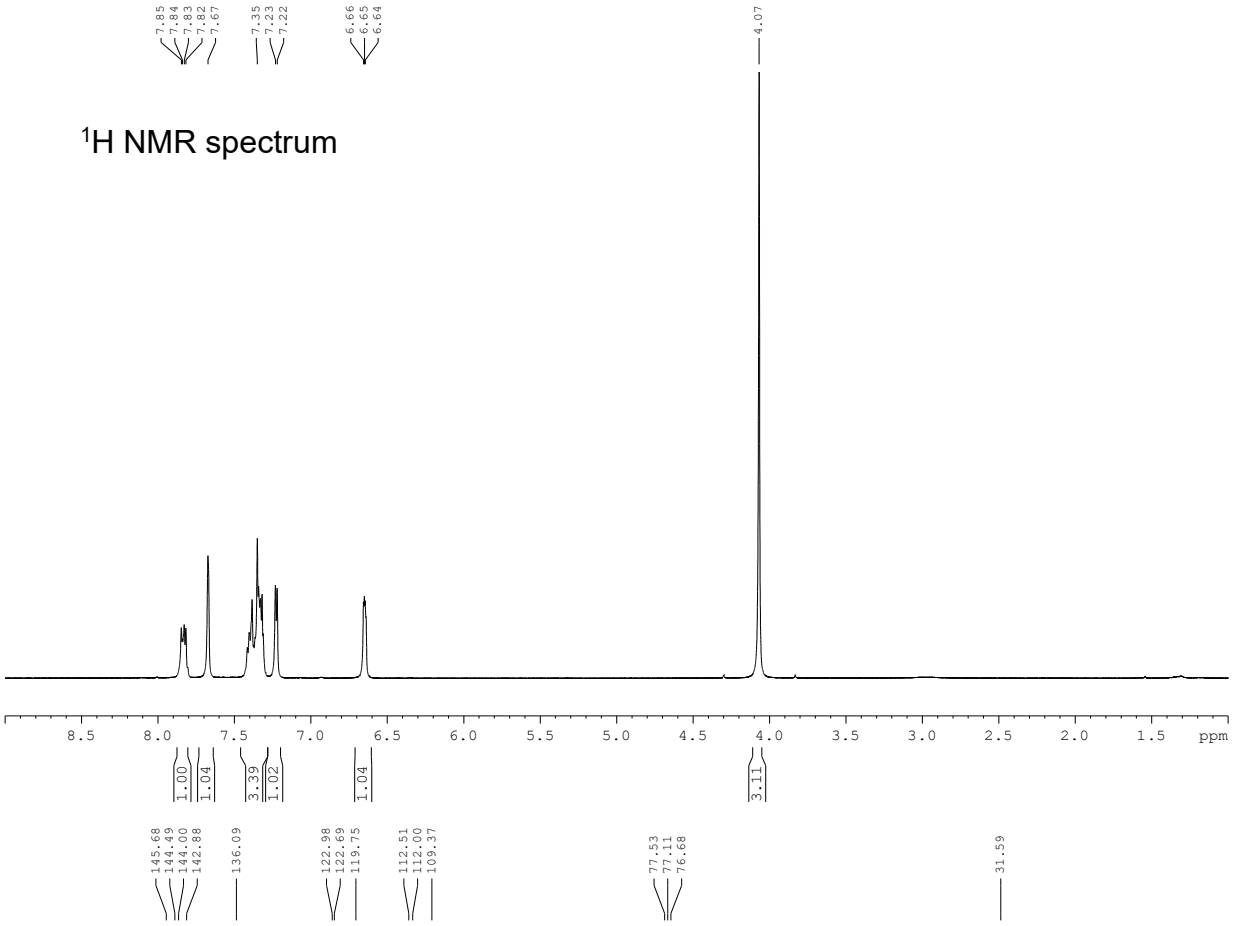
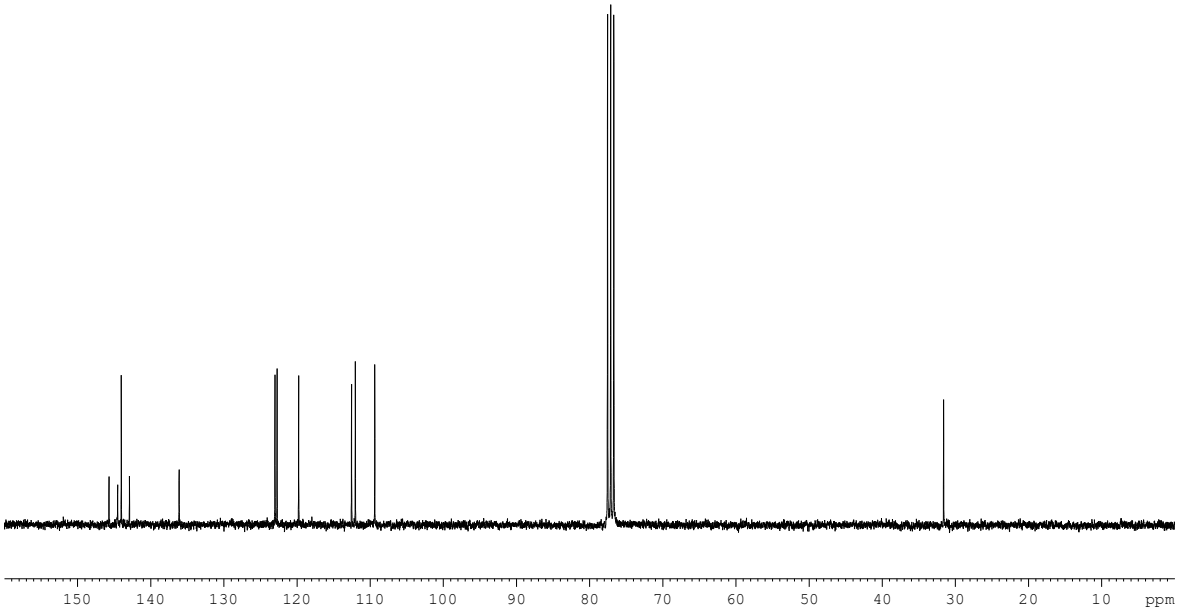


^{1}H NMR spectrum



1D proton-decoupled ^{13}C NMR spectrum