Electronic Supplementary Material (ESI) for Chemical Communications

C- and *N*-Metalated Nitriles: NMR and X-ray Analyses

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(Cyano(phenyl)methyl)magnesium chloride (9): A THF solution (1 mL) of *i*PrMgCl (2.0 M, 2 mmol, 1.15 equiv.) was added dropwise (~5 min) to a 0 °C solution (1.5 mL) of phenylacetonitrile (203 mg, 1.7 mmol). After 15 min, the cooling bath was removed and the reaction allowed to warm to room temperature. After 1 h, 750 *u*L of this solution was transferred by syringe to a dry NMR tube capped with a septum and used for unlocked ^{13}C and HETCOR NMR analyses:^{13}C NMR: δ 31.3 (d), 113.3(d), 118.2 (d), 127.5 (d), 147.6 (s).

(Cyano(2-methoxyphenyl)methyl) ^{6}lithium (71-^{6}Li) in d^{8} THF: Neat Bu^{6}Li (12 M, 28 *u*L) was added via syringe to a -75 °C, d^{8}-THF solution (700 *u*L) of 2-(2-methoxyphenyl)acetonitrile (47 mg, 0.31 mmol) in a dry NMR tube fitted with a valve cap. The deprotonation was monitored by ^{1}H NMR with spectra taken after each injection of between 5*u*L and 10 *u*L of BuLi. A temperature of -75 °C was maintained in the spectrometer and the time when the sample was outside of the instrument for adding incremental BuLi injections was minimized. ^{1}H NMR (500 MHz, THF-d^{8}) δ 2.81 (s, 1H), 3.58 (s, 3H), 6.00 (t, *J* = 7.3 Hz, 1H), 6.33 (d, *J* = 7.6 Hz, 1H), 6.41 (t, *J* = 7.3 Hz, 1H), 6.65 (d, *J* = 7.5 Hz, 1H); ^{6}Li NMR (74 MHz, THF-d^{8}) δ 0.68; ^{13}C NMR (126 MHz, THF-d^{8}) δ 25.82 (d), 55.55 (q), 110.51 (d), 112.21 (d), 118.98 (d), 121.67 (d), 139.58 (s), 146.01 (s), 151.50 (s).

(Cyano(2-methoxyphenyl)methyl) ^{6}lithium (71-^{6}Li) in d^{10} Et$_2$O: Neat Bu^{6}Li (12 M, 27 *u*L) was added via syringe to a -75 °C, d^{8}-THF solution (700 *u*L) of 2-(2-methoxyphenyl)acetonitrile (45 mg, 0.31 mmol) in a dry NMR tube fitted with a valve cap. The deprotonation was monitored by ^{1}H NMR with spectra taken after each injection of between 5*u*L and 10 *u*L of BuLi. A temperature of -75 °C was maintained in the spectrometer and the time when the sample was

outside of the instrument for adding incremental BuLi injections was minimized. ^{1}H NMR (500 MHz, Et_2O-d^{10}) δ 3.34 (s, 1H), 3.50 (s, 2H), 3.81 (s, 3H), 6.43 (s, 1H), 6.60 (s, 1H), 6.69 (s, 1H), 6.98 (s, 1H); ^{6}Li NMR (74 MHz, Et_2O -d^{10}) δ 1.42; ^{13}C NMR (126 MHz, Et_2O -d^{10}) δ 25.87, 55.09, 109.76, 115.22, 120.12, 121.17, 121.23, 136.45, 151.87, 155.26.

(Cyano(2-methoxyphenyl)methyl)magnesium chloride (71-Mg): A THF solution (6 mL) of *i*PrMgCl (2.0 M, 12 mmol, 1.14 equiv.) was added dropwise (~5 min) to a 0 °C, solution (5 mL) of 2-(2-methoxyphenyl)acetonitrile (1.54 g, 10.5 mmol). After 15 min, the cooling bath was removed and the reaction allowed to warm to room temperature. After 1 h, gentle heating was applied to afford a clear, dark-yellow solution. 750 *u*L of this solution was transferred by syringe to a dry NMR tube capped with a septum. The solvent was removed under reduced pressure at room temperature, and then the NMR tube was charged with 700 *u*L of dry d^8 THF for NMR analysis. The remaining solution was concentrated by removing approximately 1/3 of the solvent under vacuum. After leaving the solution for 5 days undisturbed at -20 °C crystals were obtained for crystallographic analysis. ^{1}H NMR (400 MHz, THF) δ 2.98 (s, 1H), 3.59 (s, 3H), 6.11 (t, J = 7.2 Hz, 1H), 6.39 (d, J = 8.3 Hz, 1H), 6.43 (t, J = 7.3 Hz, 1H), 6.69 (dd, J = 7.7, 1.4 Hz, 1H); ^{13}C NMR (101 MHz, THF) δ 25.85, 55.62, 110.87, 114.07, 119.62, 121.62, 138.15, 147.56, 152.03.

(Cyano(2-methoxyphenyl)methyl) copper (71-Cu): A hexanes solution (1.2 mL) of BuLi (2.5 M, 2.89 mmol, 1.14 equiv.) was added dropwise (~5 min) to a -78 °C, THF solution (2.0 mL) of 2-(2-methoxyphenyl)acetonitrile (372 mg, 2.53 mmol). After 15 min, the solution was transferred by syringe to a 25 mL Schlenk flask containing a -78 °C, THF slurry (3 mL) of

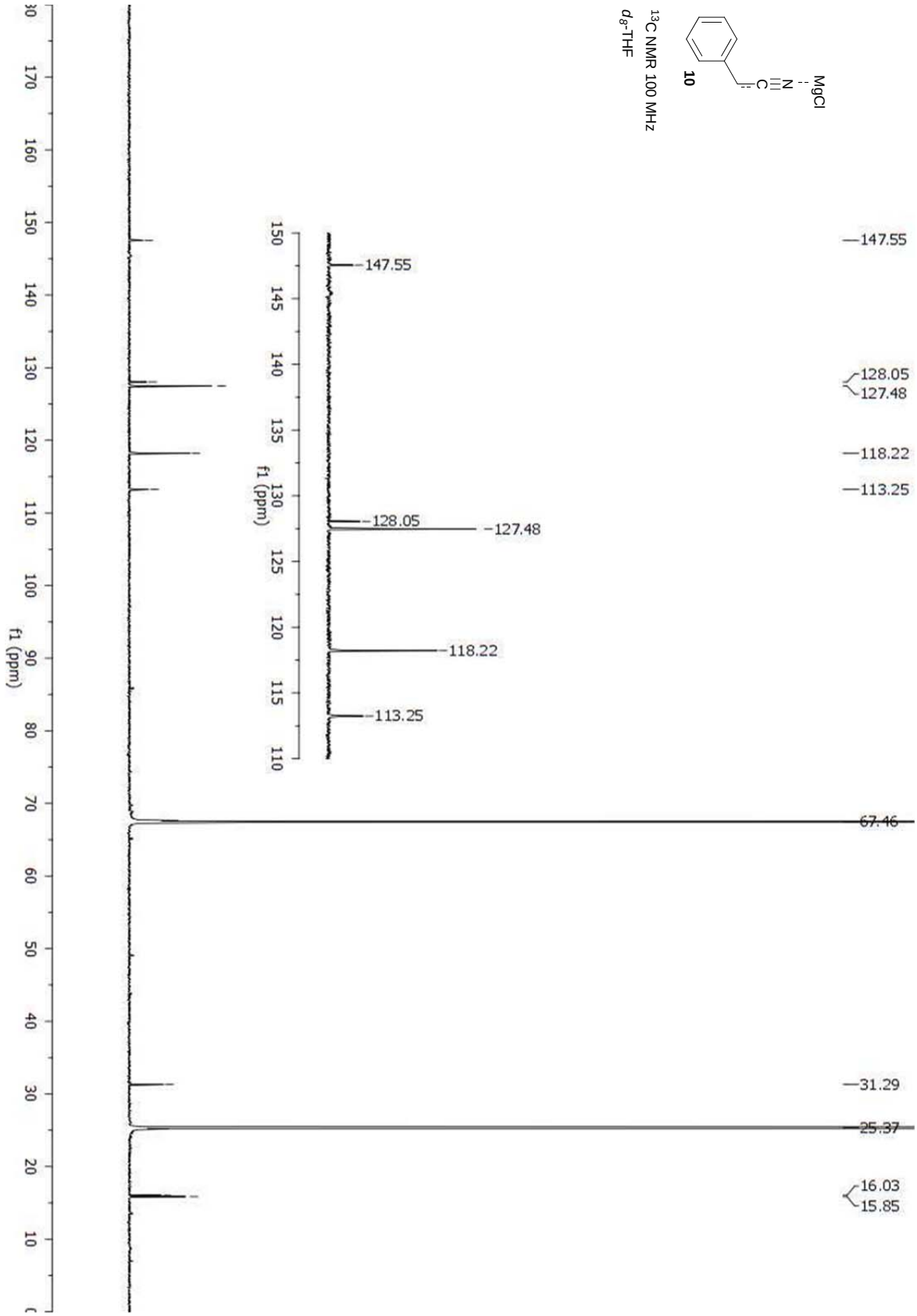
copper (I) iodide (580 mg, 3.05 mmol, 1.20 equiv.). 750 *u*L of this solution was transferred by syringe to a dry NMR tube capped with a septum and used for unlocked ^{13}C NMR analysis. ^{13}C NMR (101 MHz, THF) δ 20.04, 54.74 (q), 109.49 (d), 120.12 (d), 120.99 (d), 123.41 (d), 130.42 (s), 131.18 (s), 153.72 (s).

(Cyanocyclohexyl)lithium (43a): A flame-dried, round bottom flask with a magnetic stirbar, THF (3 mL) and *N*,*N*-diisopropyl amine (330 *u*L, 238 mg, 2.4 mmol) was cooled to -78 °C and then BuLi (2.5M in Hexanes, 900 *u*L, 2.3 mmol) was added. After 30 min neat cyclohexanecarbonitrile (250 *u*L, 230 mg 2.1 mmol) was added. After 30 minutes the cold bath is removed and after 15 min 750 *u*L of the solution is transferred to a dry NMR tube sealed with a rubber septum. ^{13}C NMR (126 MHz, THF) δ 163.50 (CN).

(1-Cyanocyclohexyl)magnesium bromide (72): A dry NMR tube fitted with a valved, septum sealed cap was charged with a THF solution (0.55M , 700 *u*L) of MesMgBr. Neat 1-bromocyclohexanecarbonitrile (up to 75 *u*L) was added at -35 °C until the ^{13}C NMR resonances of MesMgBr completely disappear. Alternatively the magnesiated nitrile **9c** was generated by the dropwise addition of 1-bromocyclohexanecarbonitrile (244 mg, 1.3 mmol) to a 0 °C, THF solution of MesMgBr (0.53 M, 1.5 mmol). After 30 min a portion of the solution was transferred to an NMR tube for analysis. Inducing crystallizations from this solution by removing the solvent and cooling (-80 °C, 5 days) yields only [1,1'-bi(cyclohexane)]-1,1'-dicarbonitrile. ^{13}C NMR (126 MHz, THF) δ 30.34, 31.16, 35.23, 45.92, 123.69, 126.58 (CN).

(1-Cyanocyclohexyl)(methyl)copper (45a): Solutions in THF were prepared by following a previously reported procedure[1] in which 1-bromocyclohexanecarbonitrile (262.8 mg, 1.40 mmol) was treated with Me_2CuLi.[2] For NMR analyses, after the exchange reaction is allowed to proceed for 1.5 h, the solution is vacuum filtered in a glovebox and 700 *u*L is transferred to a dry,

septum-sealed NMR tube: ^{13}C NMR (126 MHz, 2.5:1 THF/Et_2O) δ 23.30, 24.72, 26.81, 36.49, 123.50. Solutions in ether were prepared by adding an ether solution of MeLi (5.1 mL, 1.6 mol.L^{-1}, 8.1 mmol) to a 0 °C, ethereal slurry (2 mL) of CuI (842 mg, 4.4 mol). After 50 min, an ether solution (5 mL) of 1-Bromo-cyclohexanecarbonitrile (693 mg, 3.7 mmol) was added an after 1.5 hour, 1.5 mL solution of this sample was removed by syringe to a sealed, N_2 protected NMR tube. ^{13}C NMR 15.1 (q, *J* = 126Hz, 1C), 23.6 (t, *J* = 127Hz, 1C), 25.1 (t, *J* = 128Hz, 1C), 27.2 (q, *J* = 130Hz, 1C), 34.9 (s, 1C), 37.2 (t, *J* = 129Hz, 1C), 65.7 (t, *J* = 141Hz, 1C), 125.1 (s, 1C). After 3 h at room temperature, the remaining solution was cooled to 0 °C and neat propargyl bromide (511 mg, 4.30 mmol). After 2 h saturated, aqueous NH_4Cl solution was added, the mixture was stirred vigorously with exposure to air for 30 min, and then the crude product was extracted with ethyl ether. The combined organic extracts were dried ($MgSO_4$) and purified by radial chromatography (1:100 to 1:50 EtOAc/hexanes) to afford 227 mg (47%) of 1-Propa-1,2-dienyl-cyclohexanecarbonitrile as an oil spectrally identical to material previously isolated.[1]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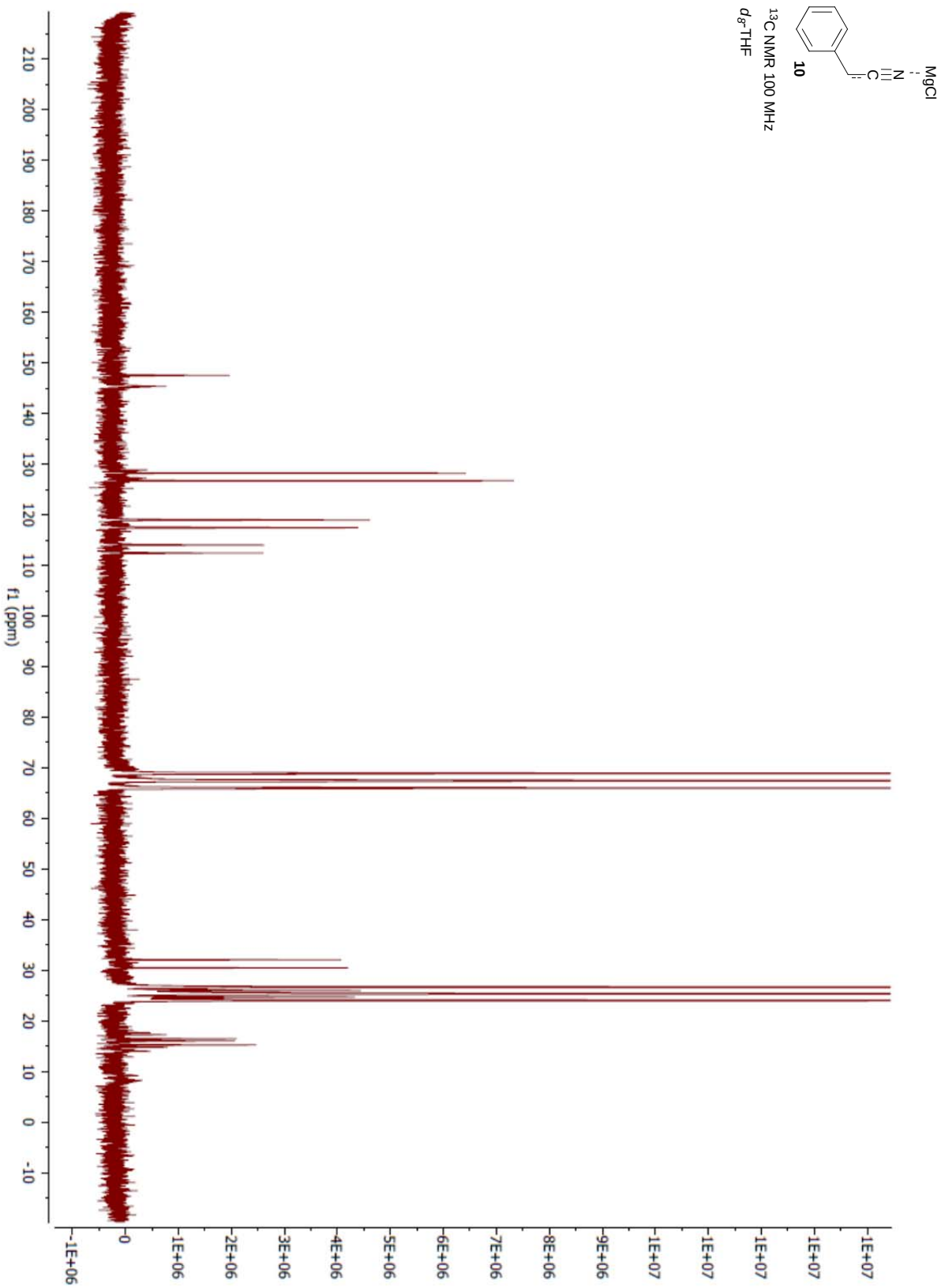
d_8-THF
10
MgCl
f1 (ppm)
147.55
128.05
127.48
118.22
113.25
67.46
31.29
25.37
16.03
15.85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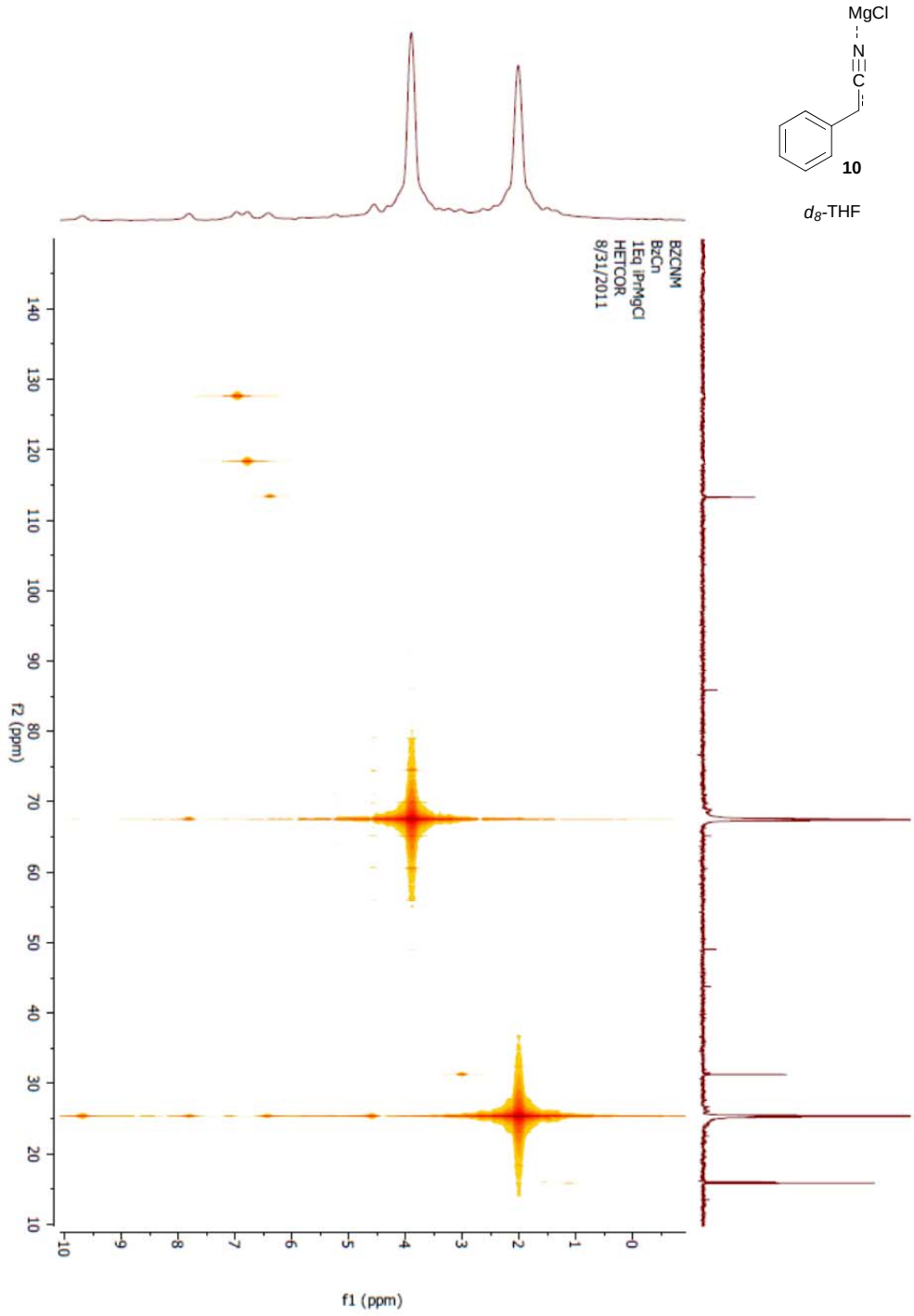
10
^{13}C NMR 100 MHz
d_8-THF
f1 (ppm)


MgCl
N
C
10
d_8-THF
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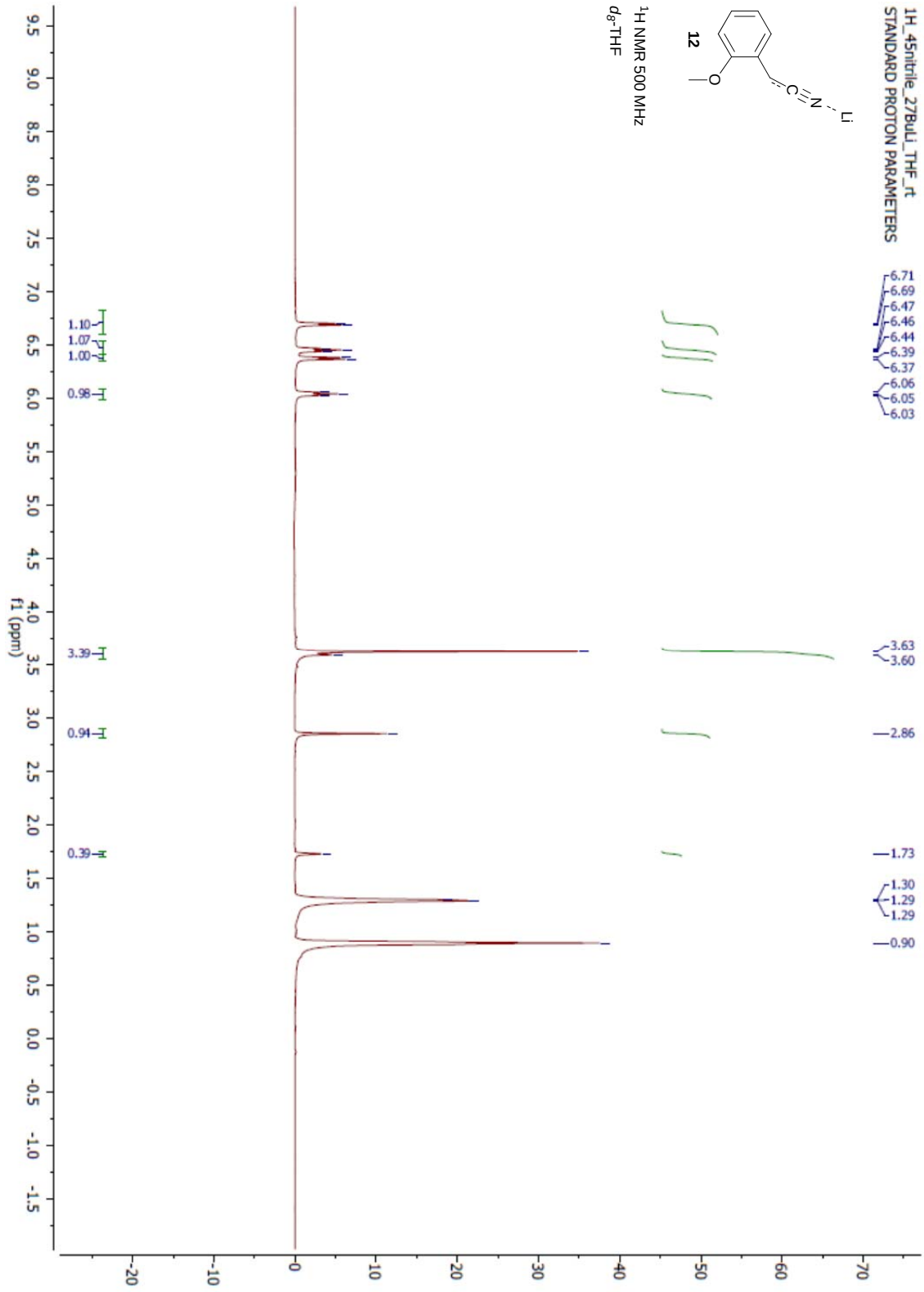
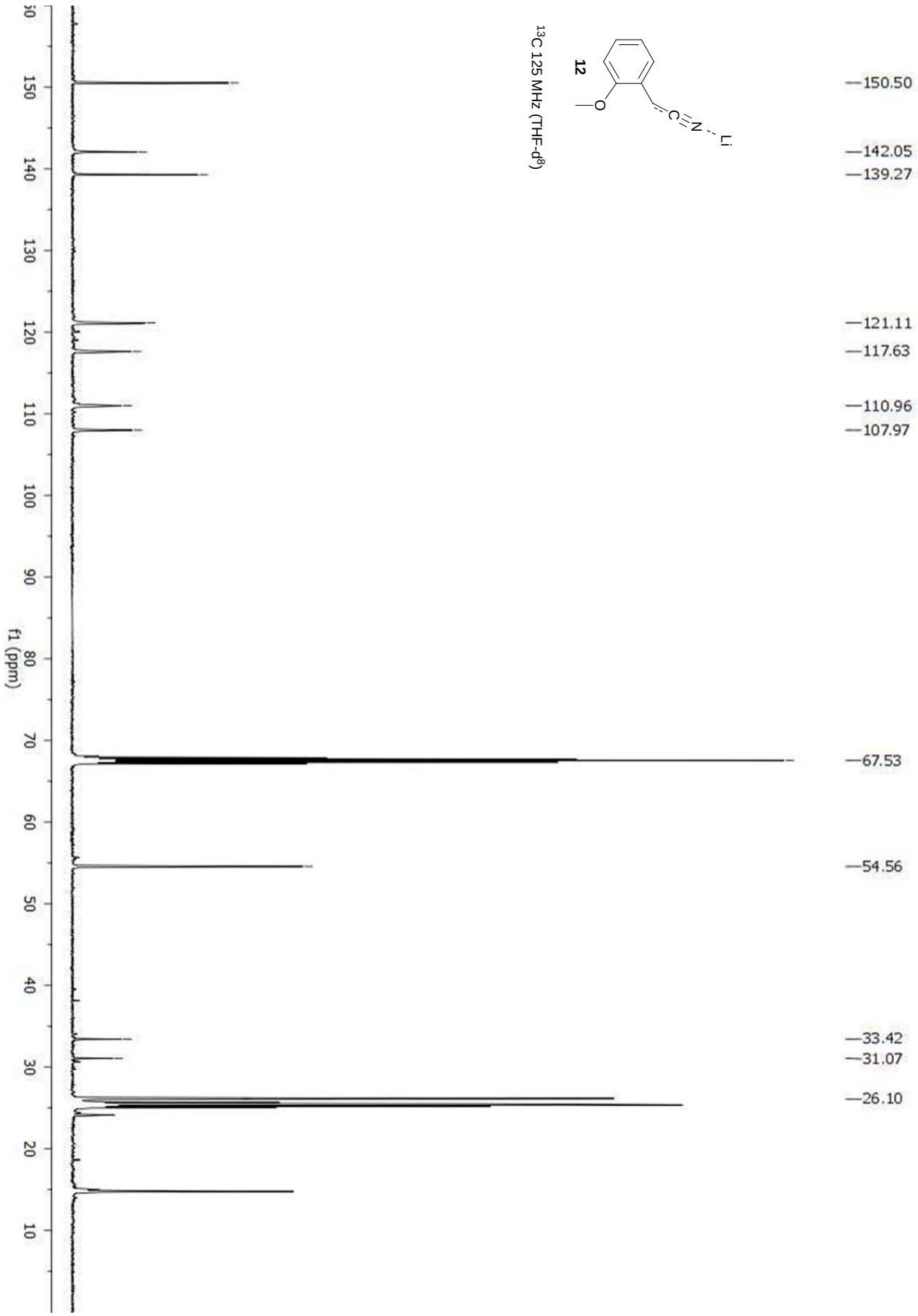
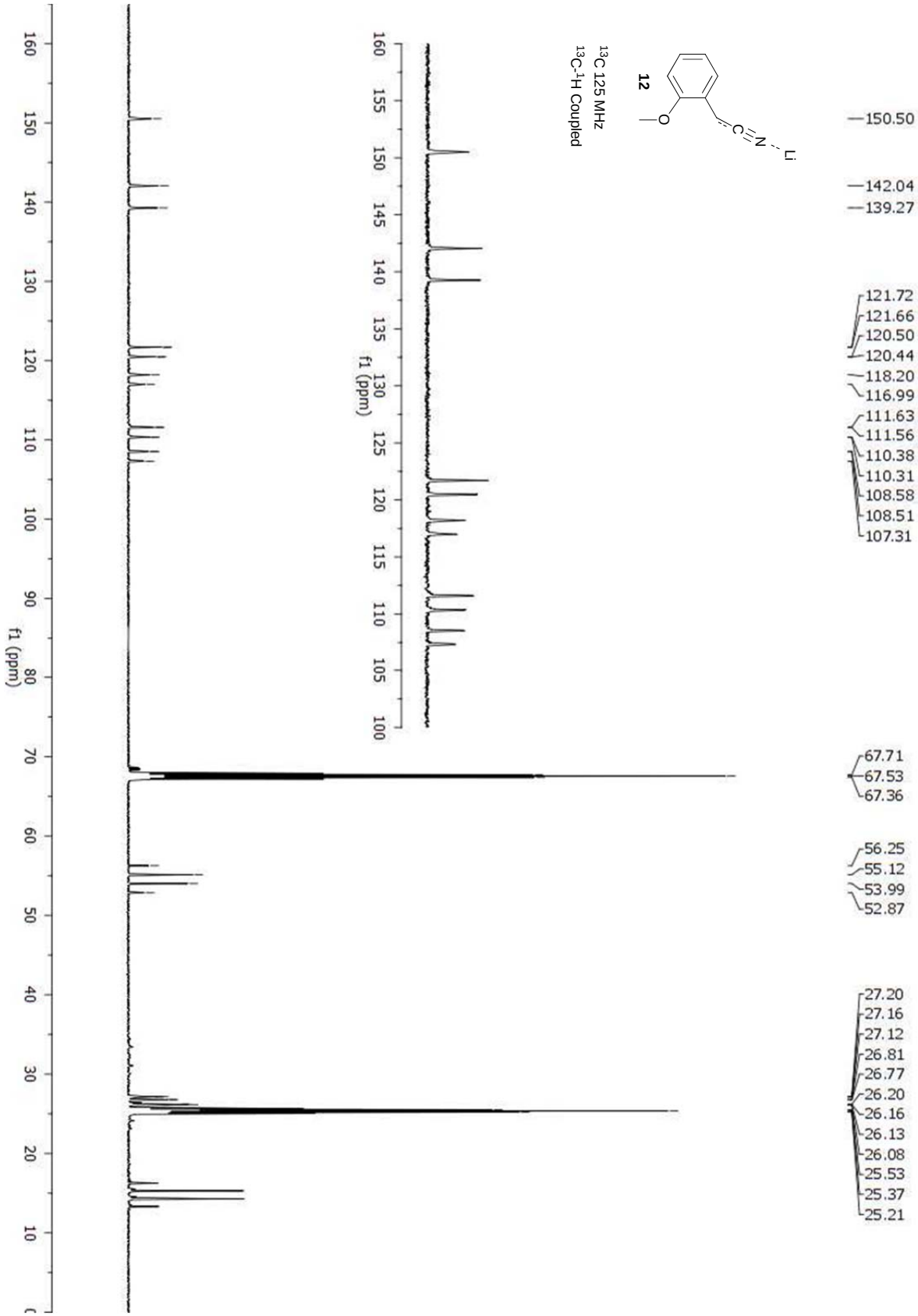
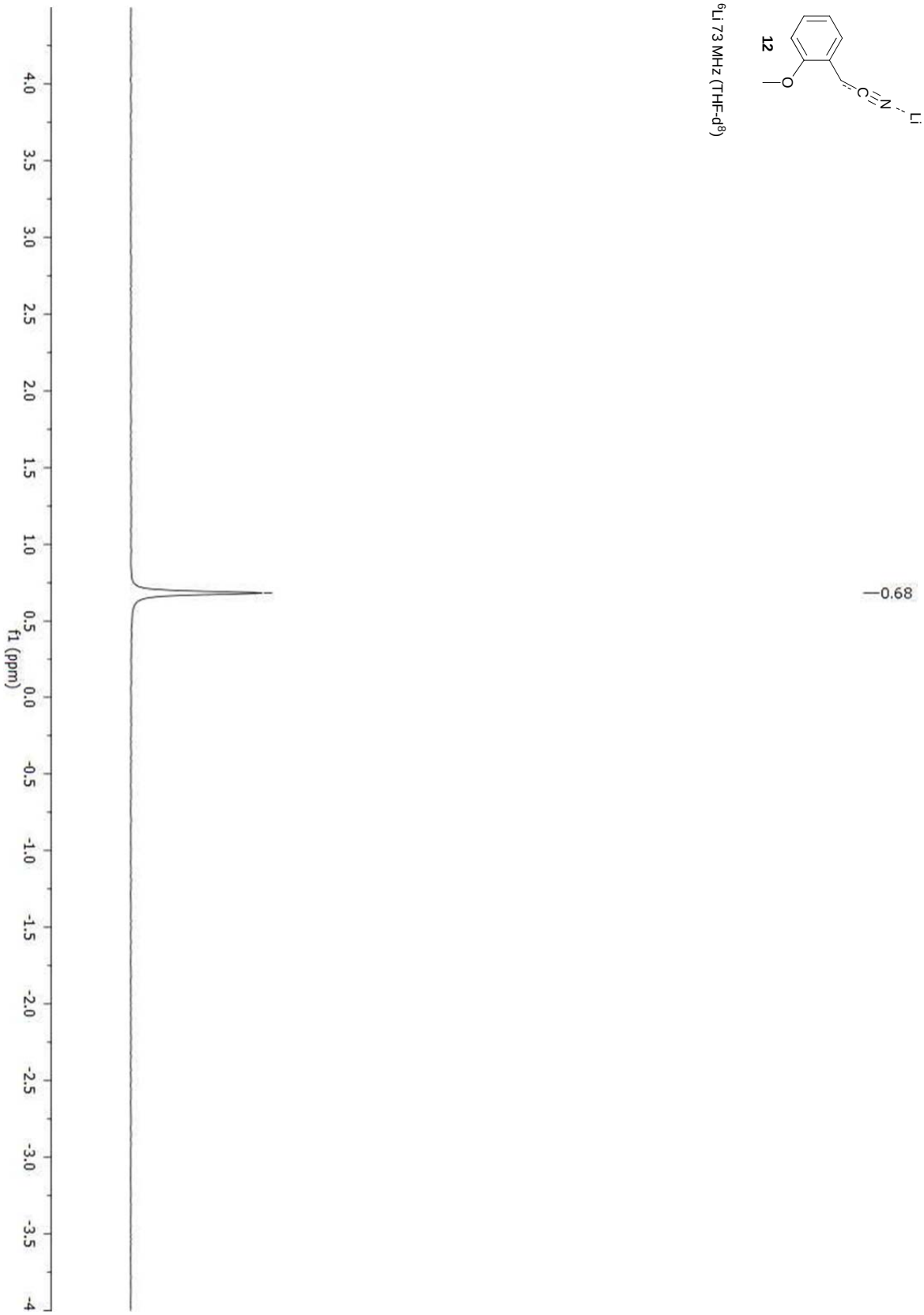
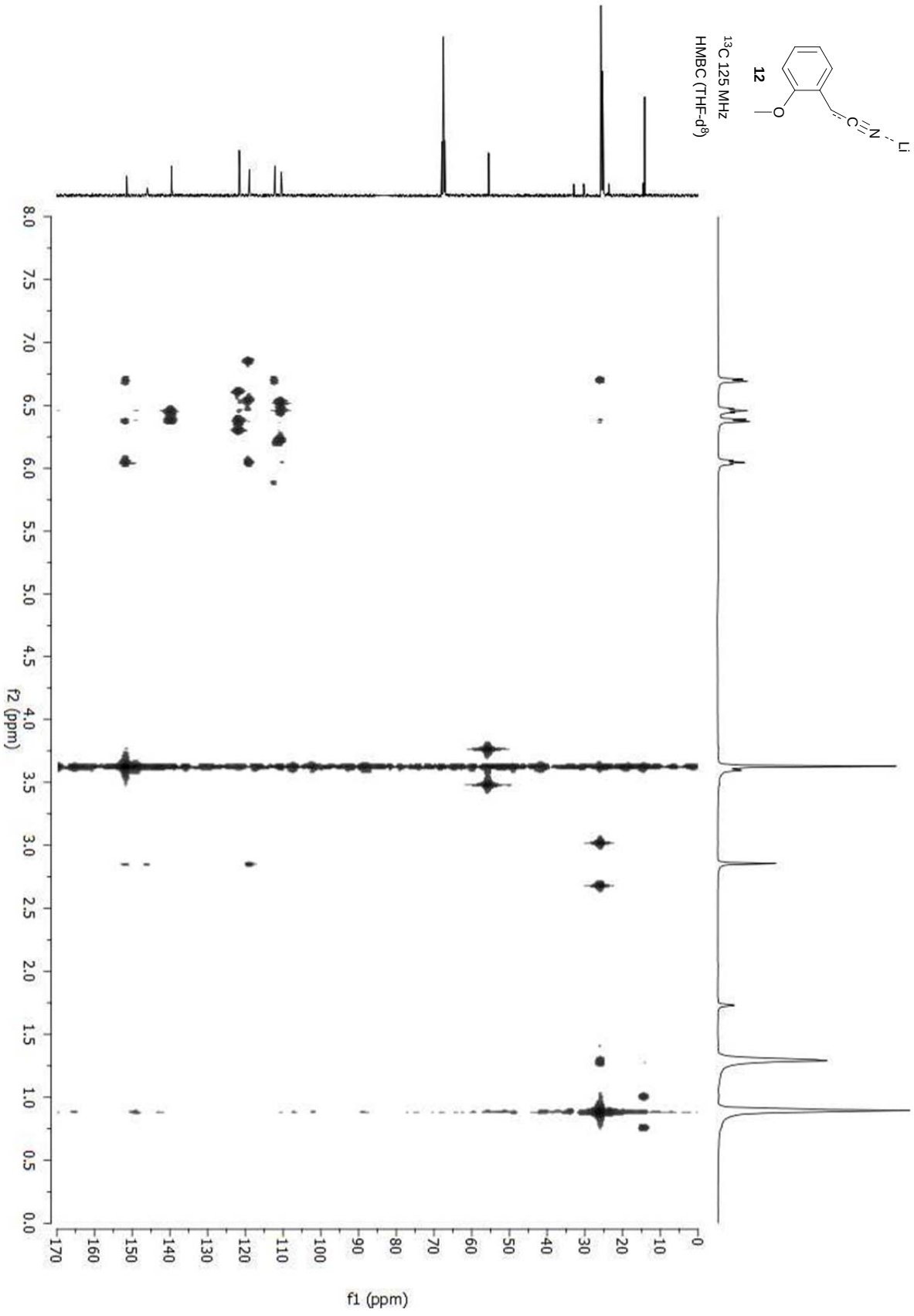
BzCn
1Eq iPrMgCl
HETCOR
8/31/2011
f2 (ppm)
f1 (ppm)

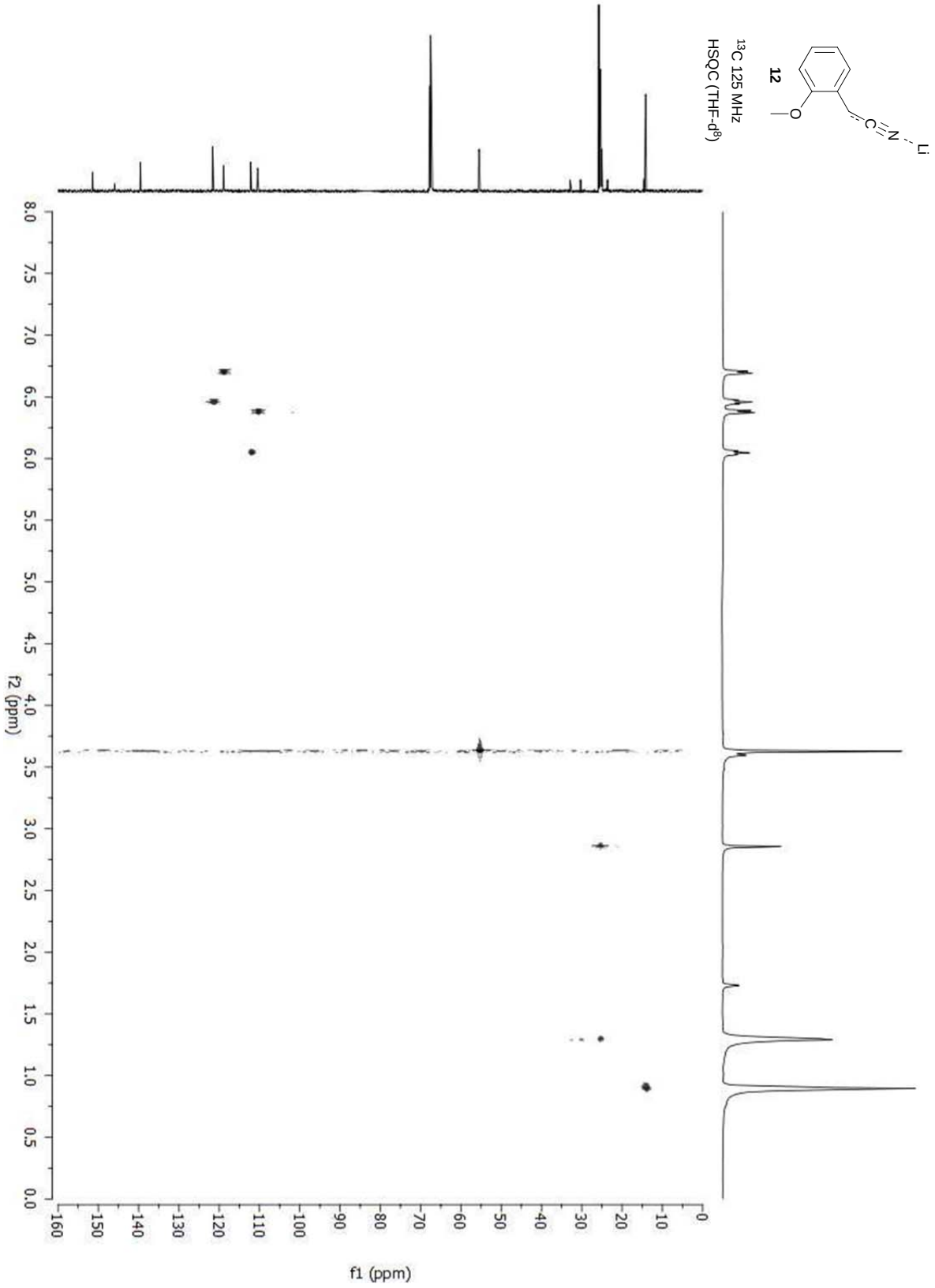

1H_45nitrile_27BuLi_THF_rt
STANDARD PROTON PARAMETERS
^{1}H NMR 500 MHz
d_8-THF
12
O
C
N
Li
6.71
6.69
6.47
6.46
6.44
6.39
6.37
6.06
6.05
6.03
3.63
3.60
2.86
1.73
1.30
1.29
1.29
0.90
1.10
1.07
1.00
0.98
3.39
0.94
0.39
f1 (ppm)

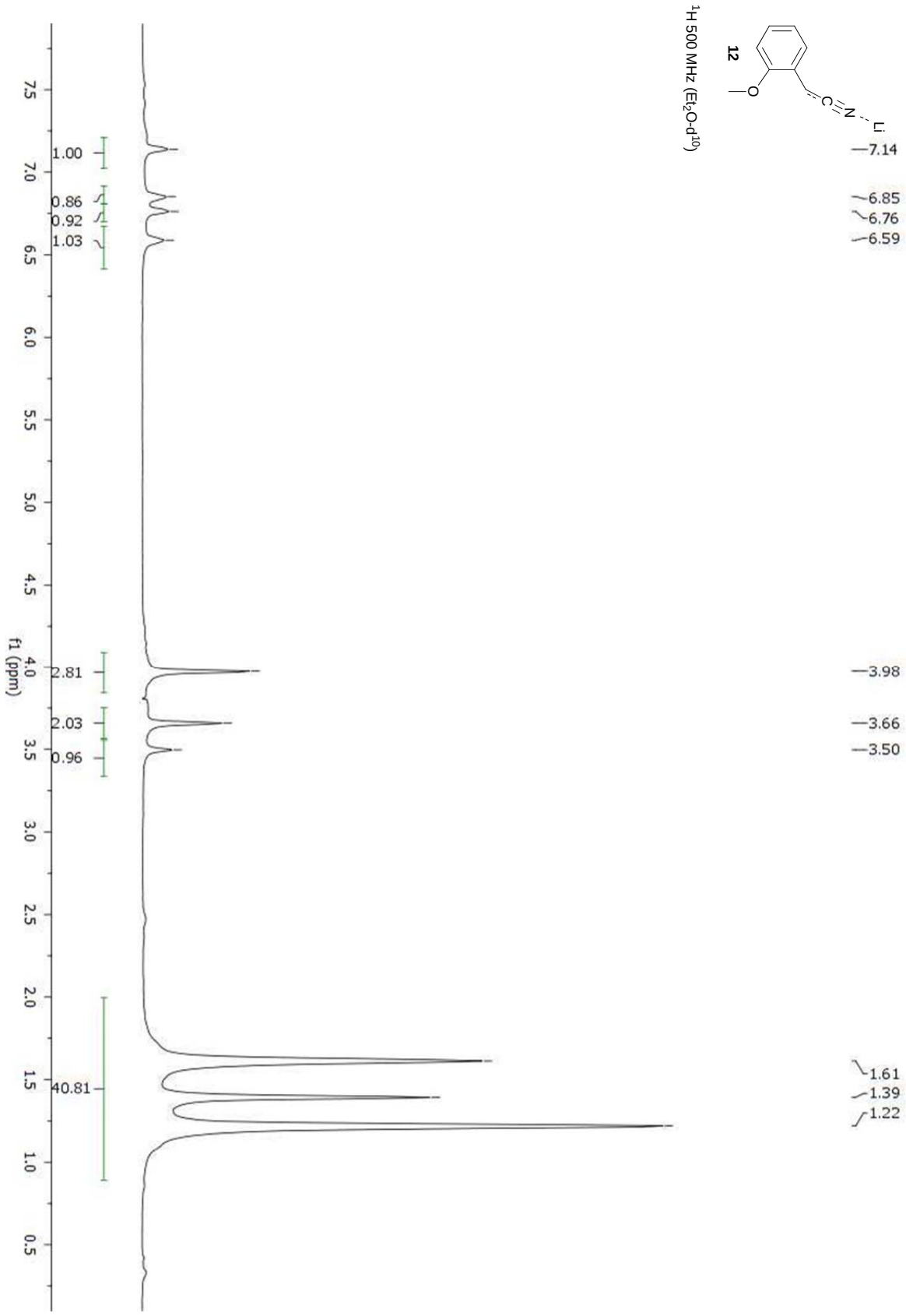

^{13}C 125 MHz (THF-d^8)
12
O
C
N
Li
150.50
142.05
139.27
121.11
117.63
110.96
107.97
67.53
54.56
33.42
31.07
26.10
f1 (ppm)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

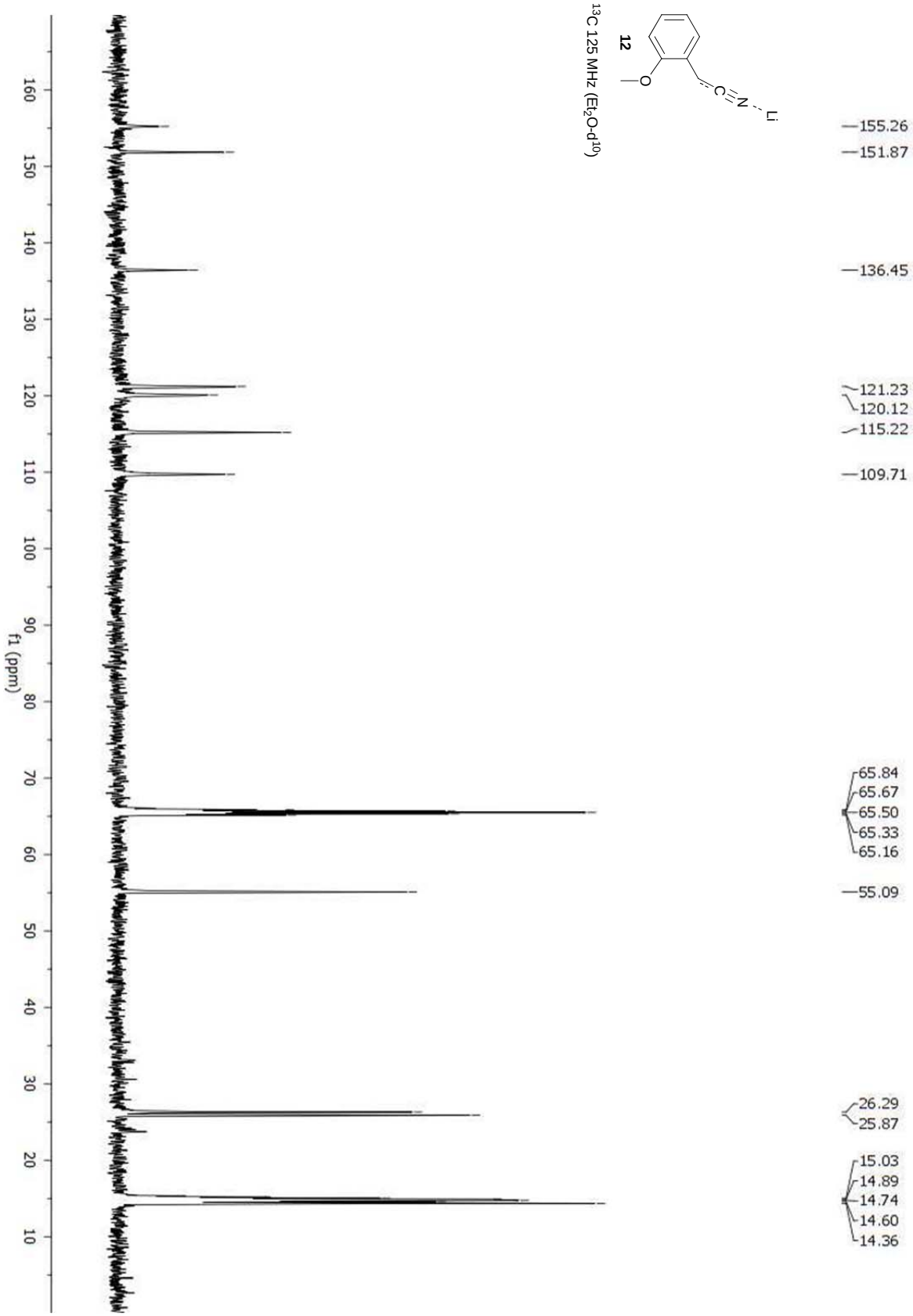

13C 125 MHz
13C-1H Coupled
12
O
C
N
Li
150.50
142.04
139.27
121.72
121.66
120.50
120.44
118.20
116.99
111.63
111.56
110.38
110.31
108.58
108.51
107.31
67.71
67.53
67.36
56.25
55.12
53.99
52.87
27.20
27.16
27.12
26.81
26.77
26.20
26.16
26.13
26.08
25.53
25.37
25.21
f1 (ppm)

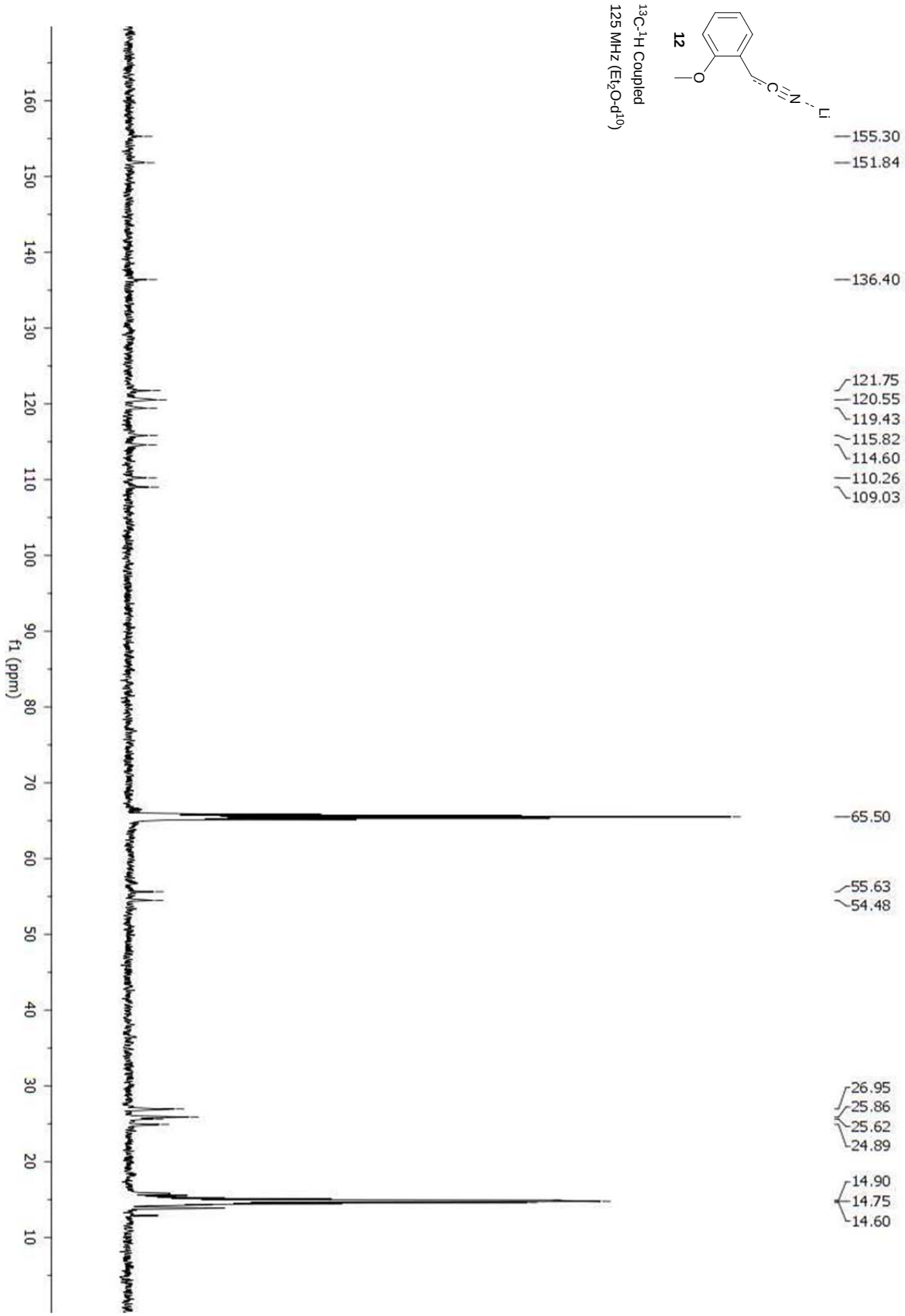

^{6}Li 73 MHz (THF-d^8)
12
O
C
N
Li
−0.68
f1 (ppm)
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-2.0
-2.5
-3.0
-3.5
-4

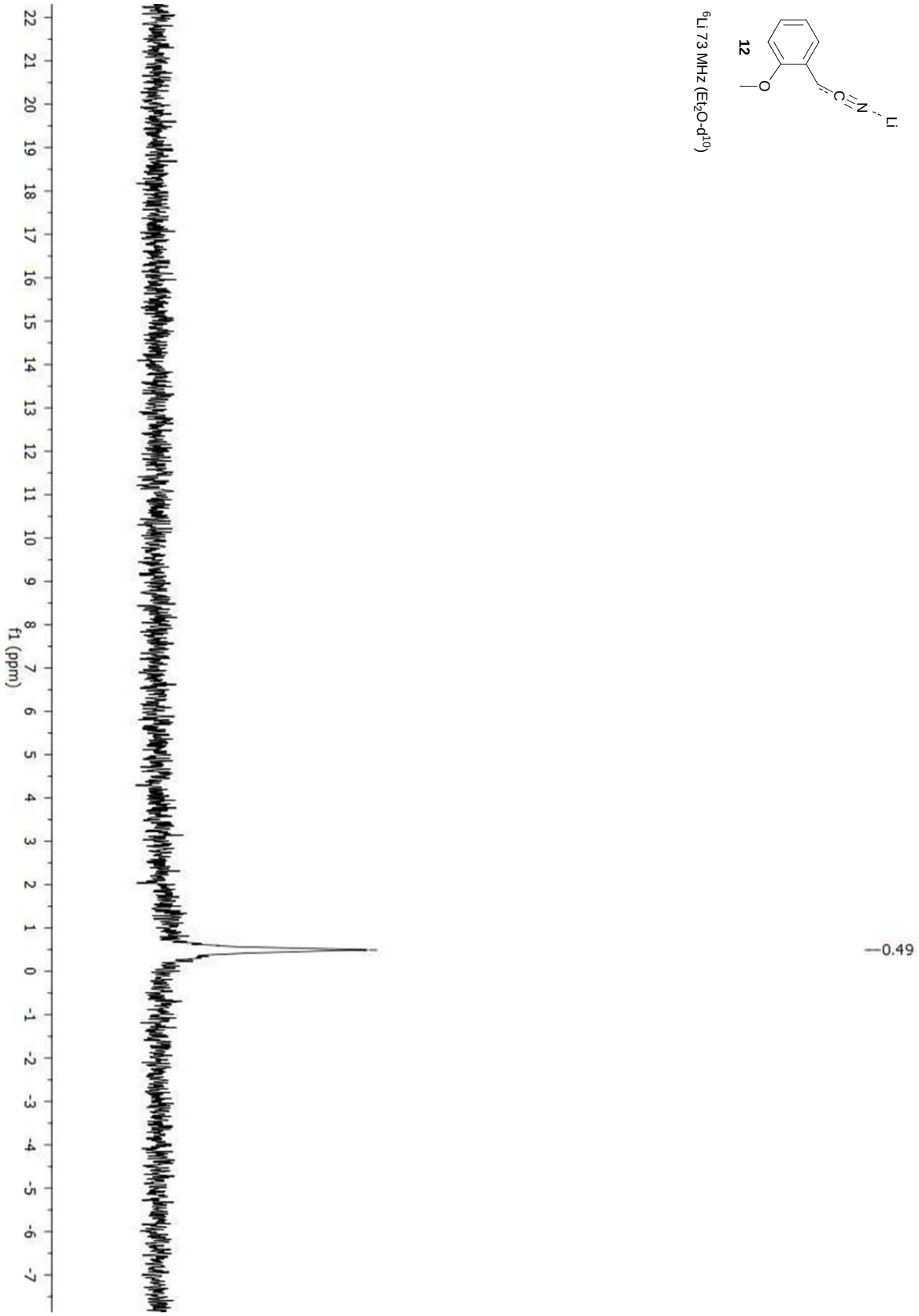

HMBC (THF-d8) 13C 125 MHz
12
O
C
N
Li
f2 (ppm)
f1 (ppm)

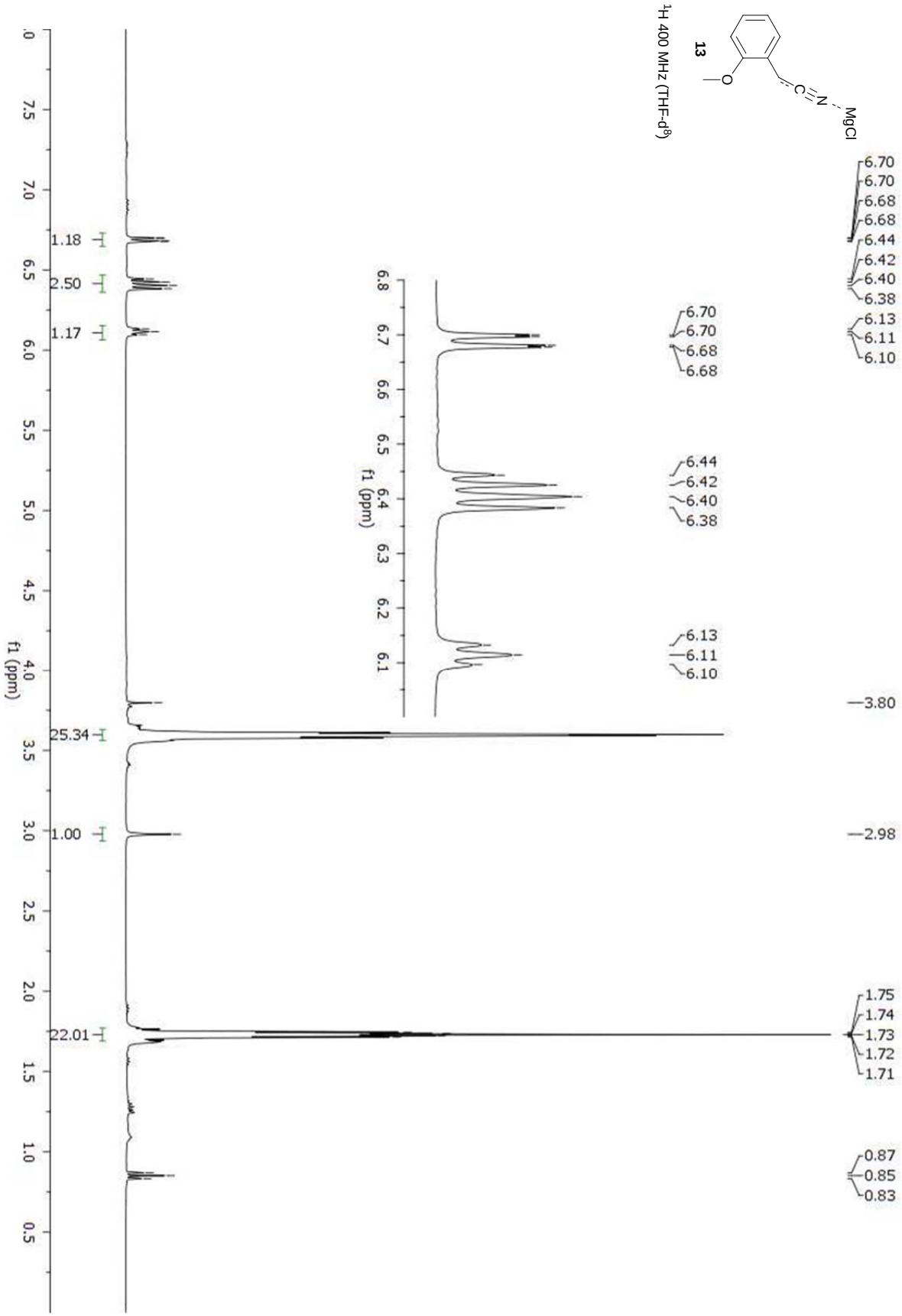

^{13}C 125 MHz
HSQC (THF-d^8)
12
O
C
N
Li
f2 (ppm)
f1 (ppm)

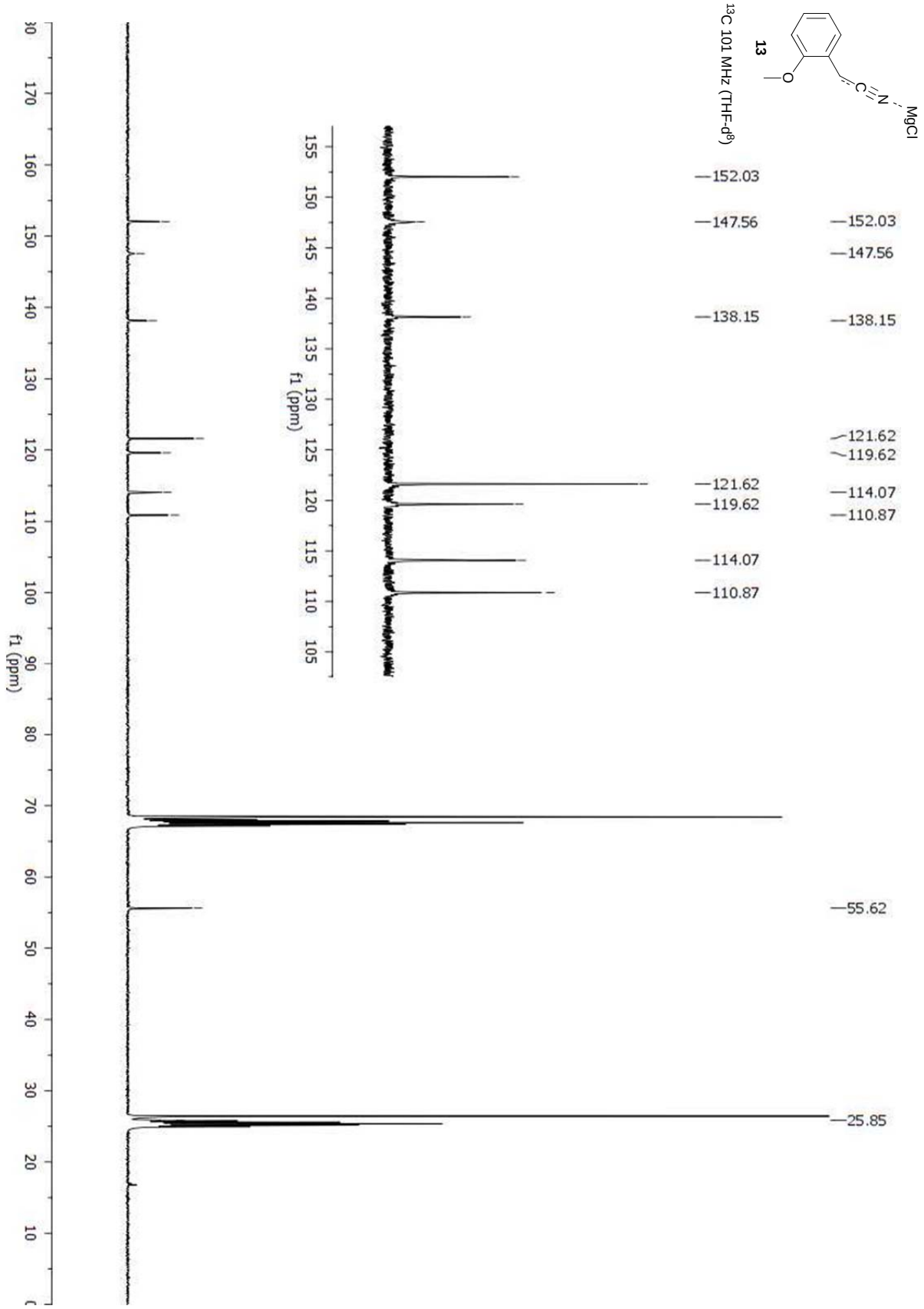

^{1}H 500 MHz (Et_2O-d^{10})
12
O
C
N
Li
7.14
6.85
6.76
6.59
3.98
3.66
3.50
1.61
1.39
1.22
1.00
0.86
0.92
1.03
2.81
2.03
0.96
40.81
f1 (ppm)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

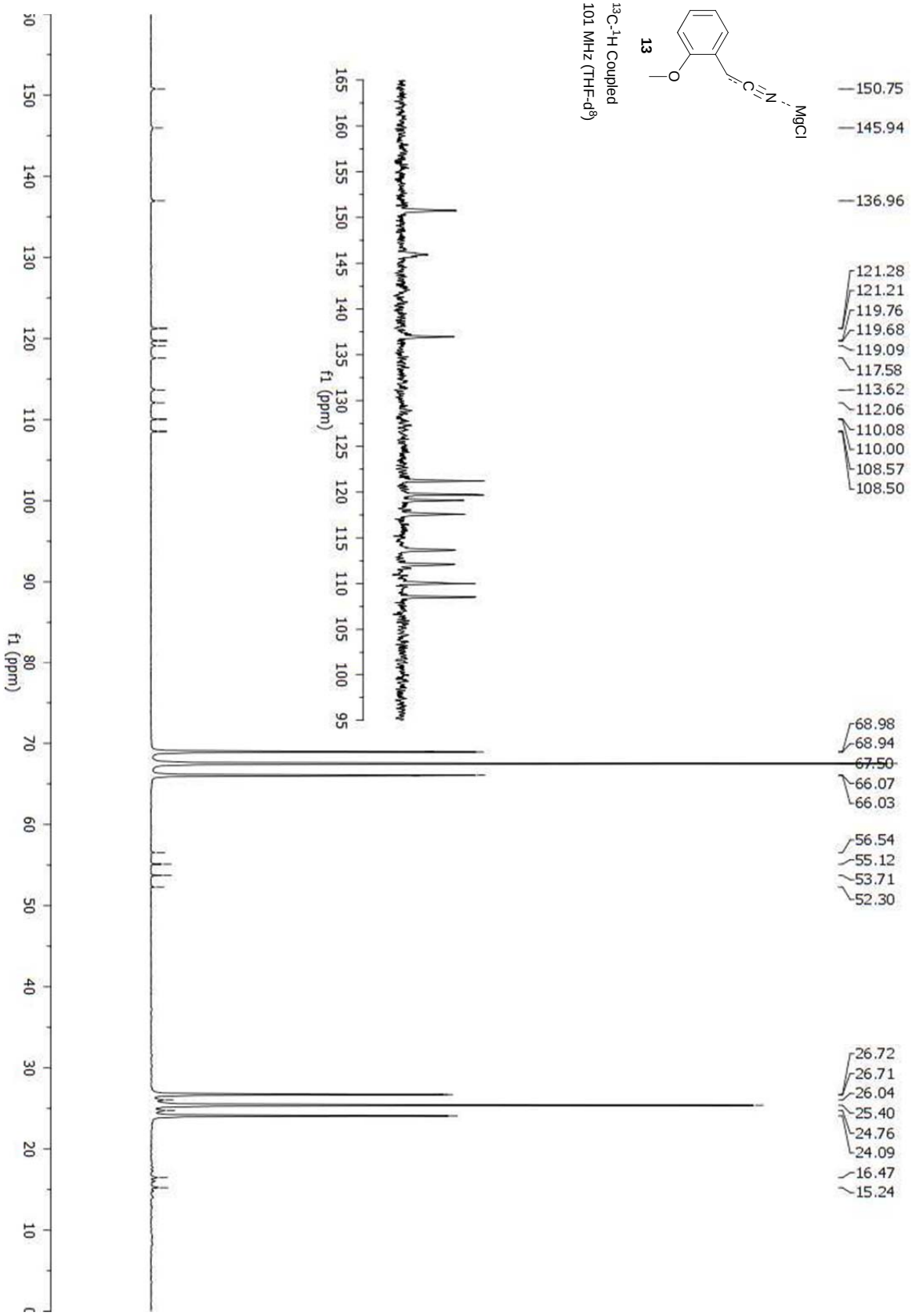

^{13}C 125 MHz (Et_2O-d^{10})
12
O
C
N
Li
155.26
151.87
136.45
121.23
120.12
115.22
109.71
65.84
65.67
65.50
65.33
65.16
55.09
26.29
25.87
15.03
14.89
14.74
14.60
14.36
f1 (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


^{13}C-^{1}H Coupled
125 MHz (Et_2O-d^{10})
12
O
C
N
Li
155.30
151.84
136.40
121.75
120.55
119.43
115.82
114.60
110.26
109.03
65.50
55.63
54.48
26.95
25.86
25.62
24.89
14.90
14.75
14.60
f1 (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


6Li 73 MHz (Et2O-d10)
12
O
C
N
Li
f1 (ppm)
22 21 20 19 18 17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5 -6 -7
0.49


13
MgCl
^{1}H 400 MHz (THF-d^8)
f1 (ppm)
1.18
2.50
1.17
25.34
1.00
22.01
6.70
6.70
6.68
6.68
6.44
6.42
6.40
6.38
6.13
6.11
6.10
3.80
2.98
1.75
1.74
1.73
1.72
1.71
0.87
0.85
0.83


13
MgCl
^{13}C 101 MHz (THF-d^8)
152.03
147.56
138.15
121.62
119.62
114.07
110.87
55.62
25.85
f1 (ppm)


13
^{13}C-^{1}H Coupled
101 MHz (THF-d^8)
MgCl
150.75
145.94
136.96
121.28
121.21
119.76
119.68
119.09
117.58
113.62
112.06
110.08
110.00
108.57
108.50
68.98
68.94
67.50
66.07
66.03
56.54
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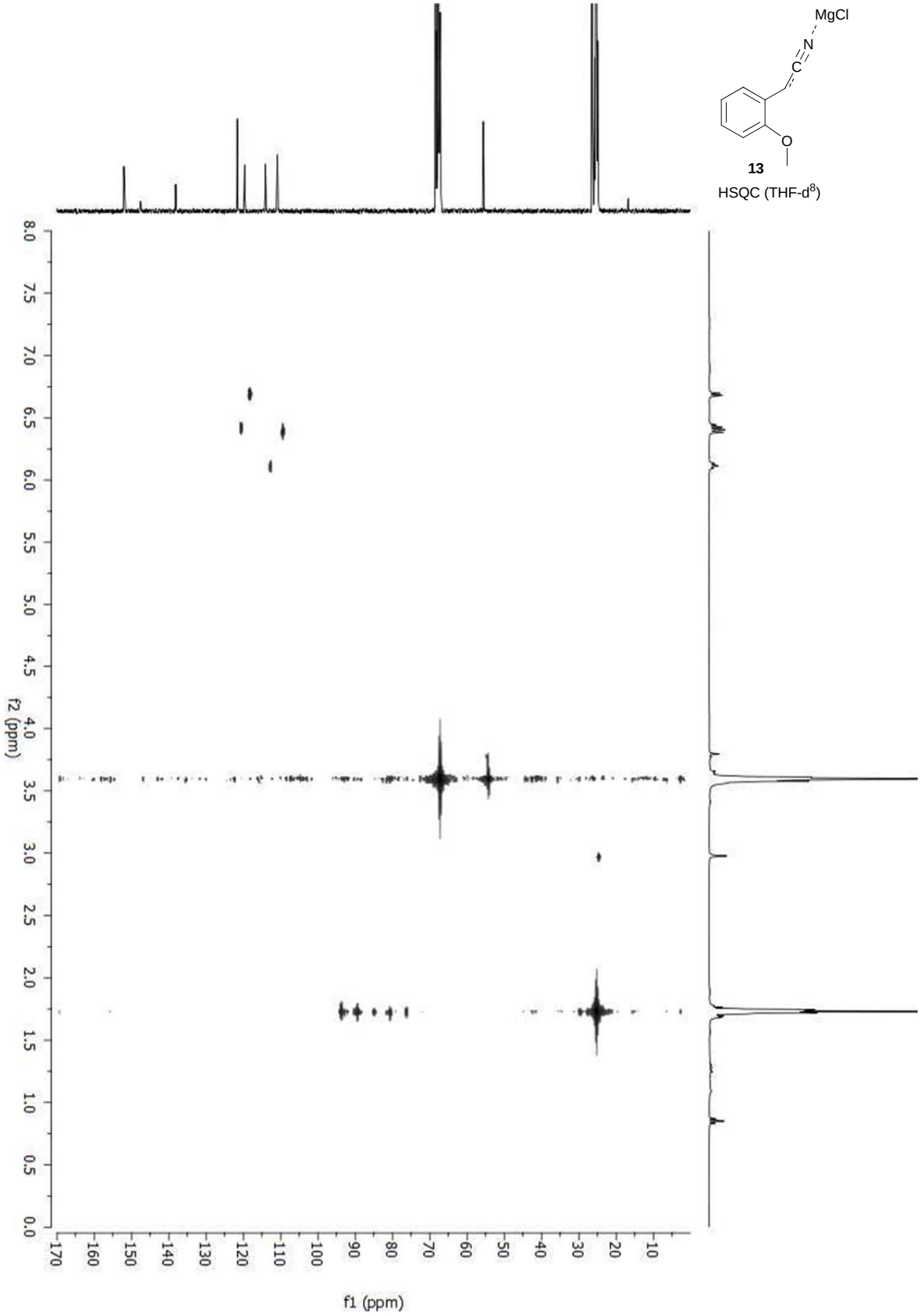
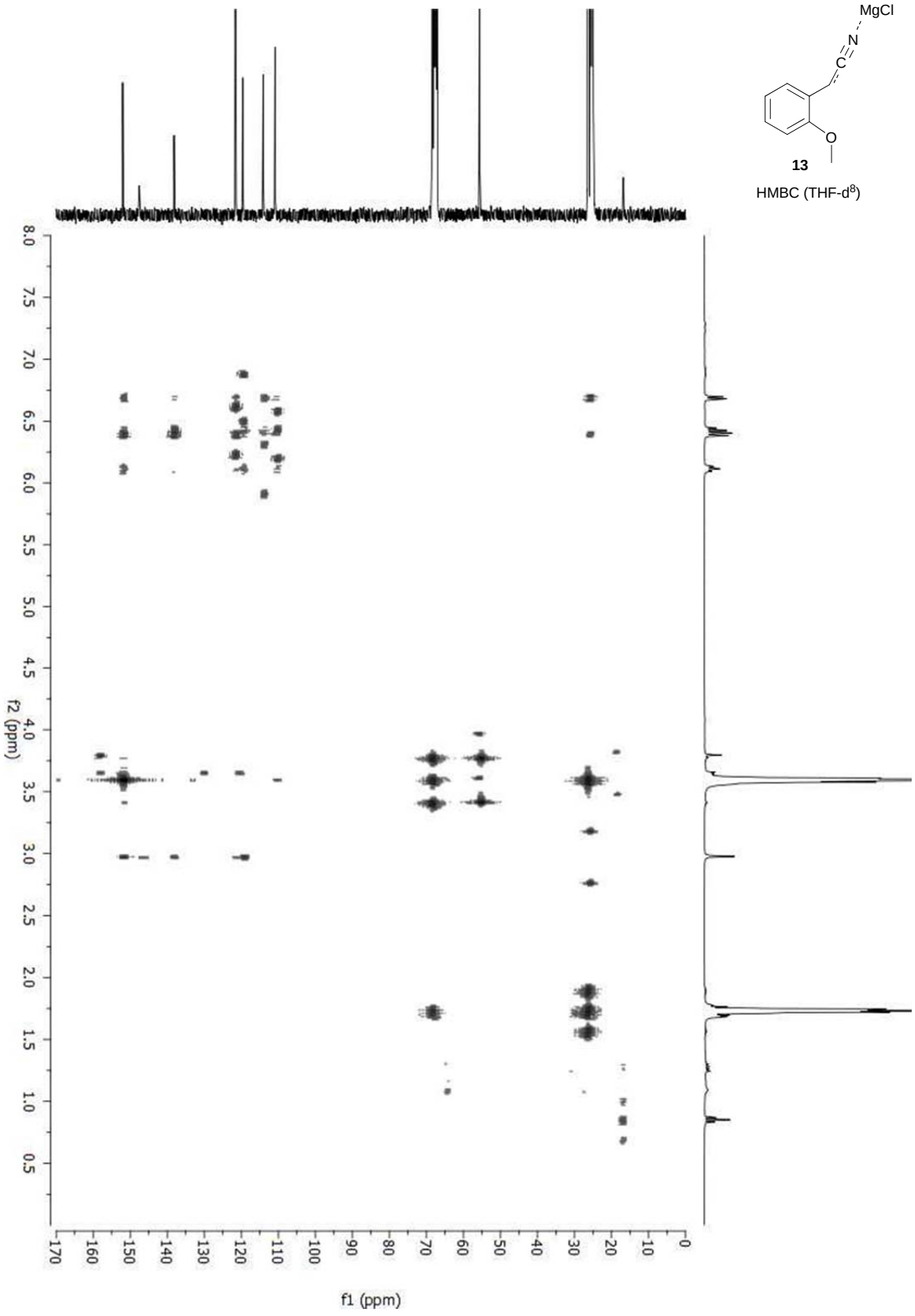
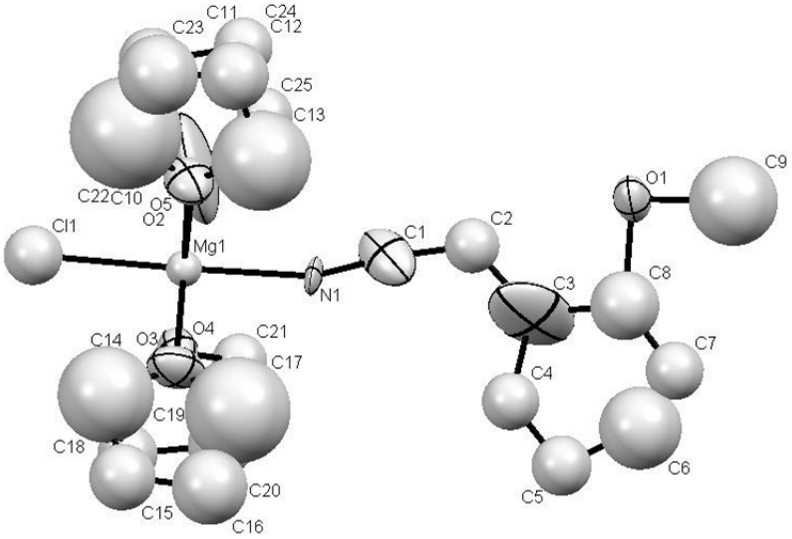
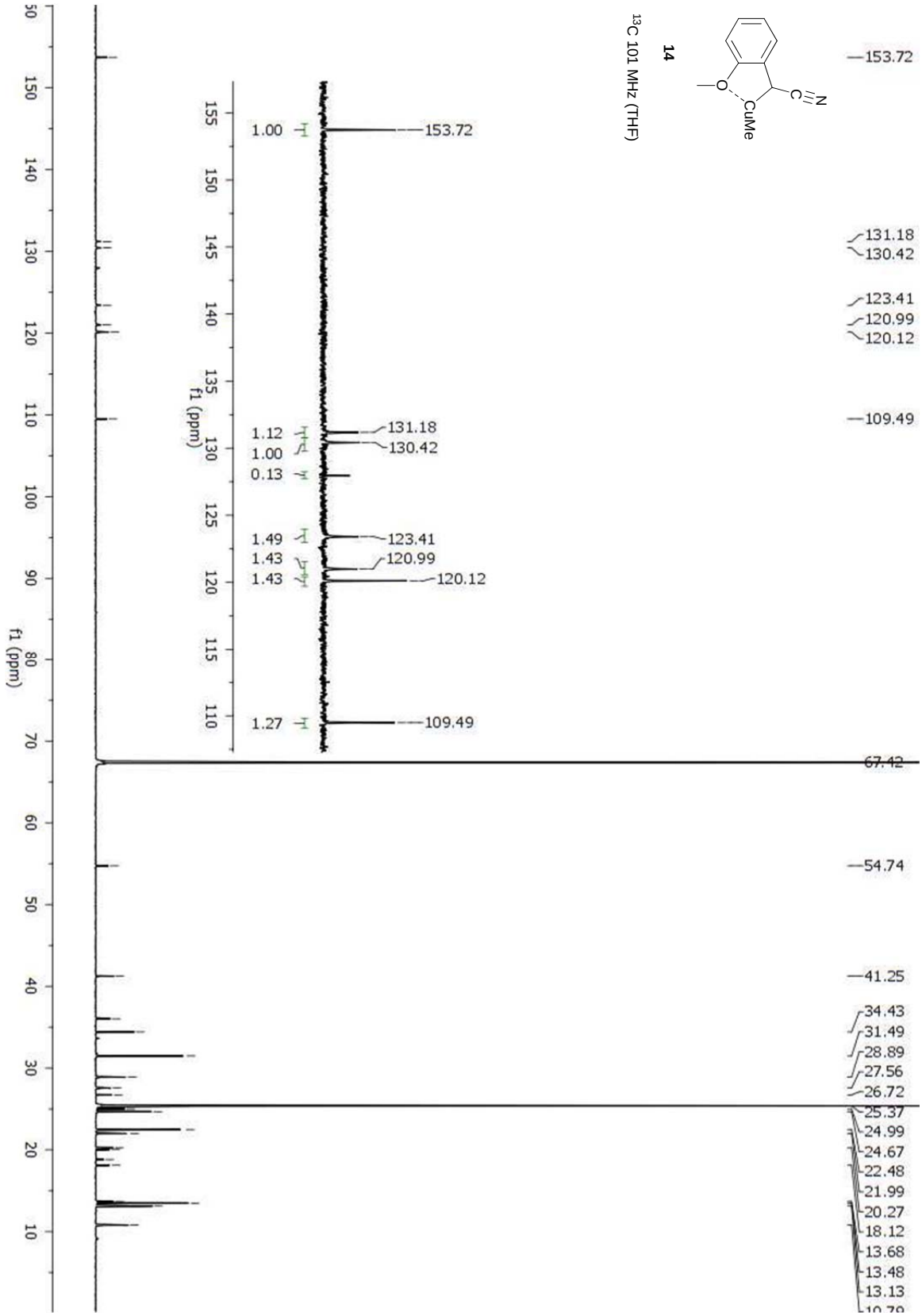
24.76
24.09
16.47
15.24
f1 (ppm)

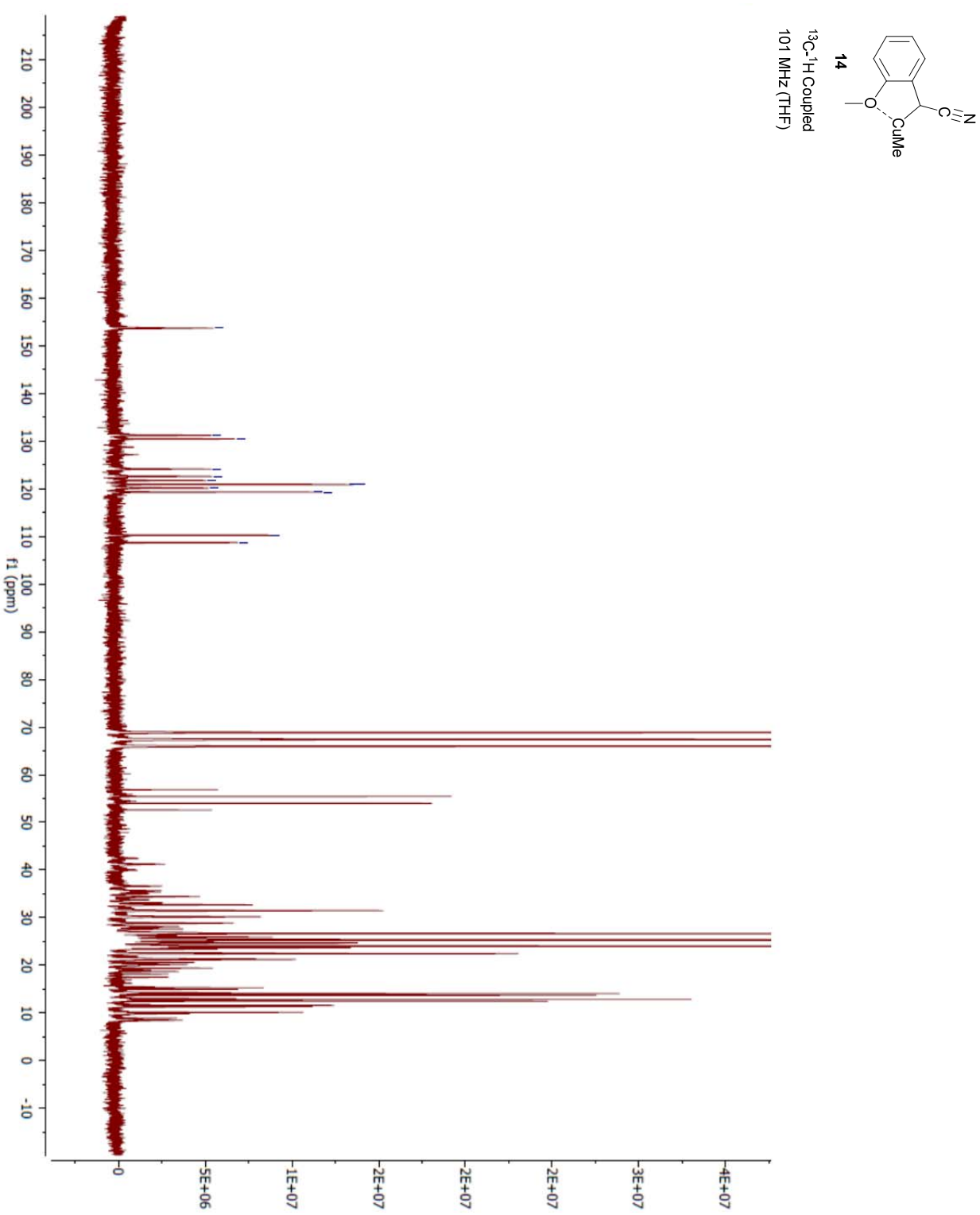

MgCl
N
C
O
13
HSQC (THF-d8)
f2 (ppm)
f1 (ppm)

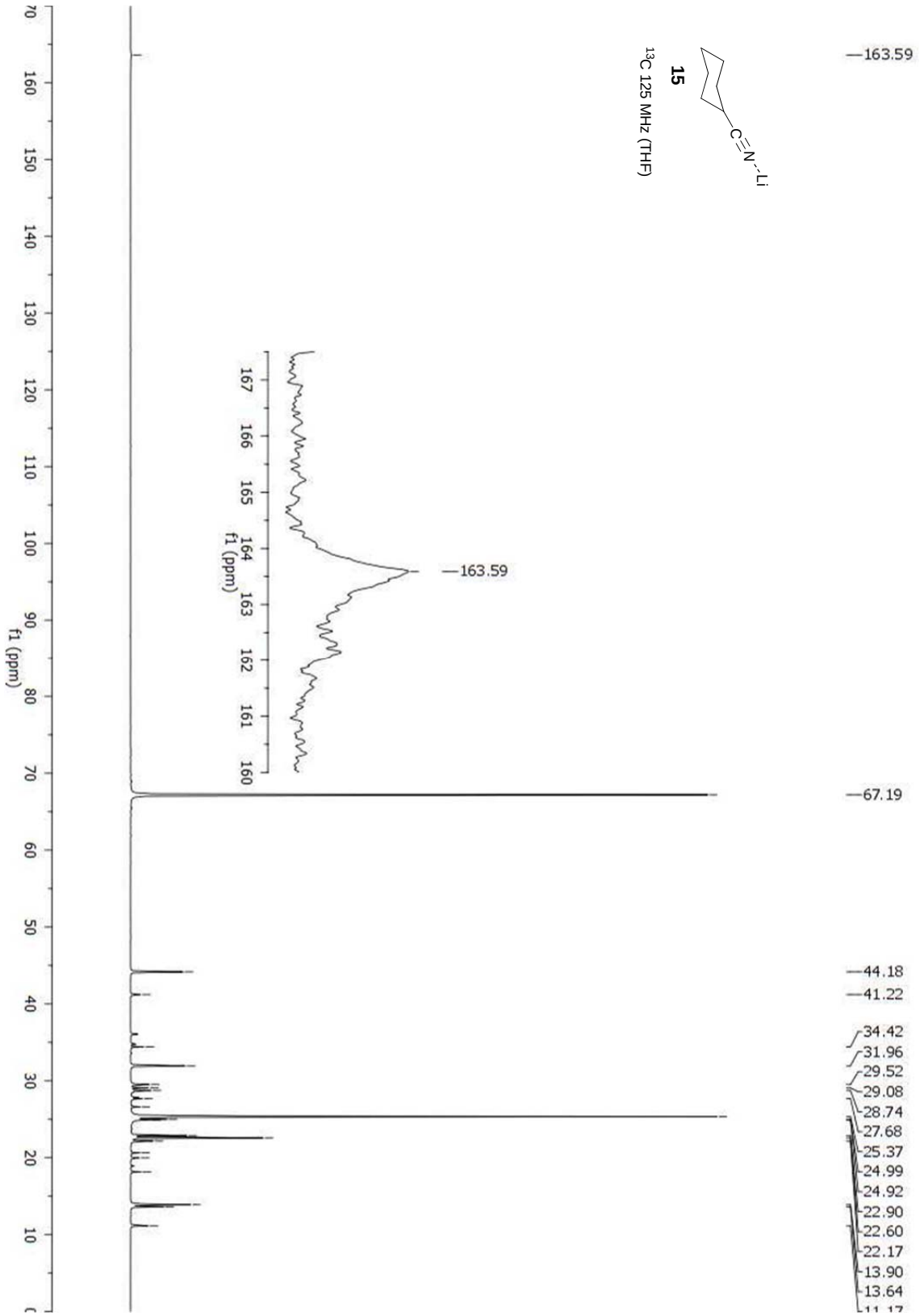

MgCl
N
C
O
13
HMBC (THF-d8)
f2 (ppm)
f1 (ppm)

ORTEP of 13


C11
C24
C23
C12
C25
C13
C22
C10
O5
O2
Cl1
Mg1
C1
C2
O1
C9
N1
C3
C8
C14
O3
O4
C21
C17
C7
C4
C19
C18
C6
C20
C5
C15
C16


^{13}C 101 MHz (THF)
14
CuMe
153.72
131.18
130.42
123.41
120.99
120.12
109.49
67.42
54.74
41.25
34.43
31.49
28.89
27.56
26.72
25.37
24.99
24.67
22.48
21.99
20.27
18.12
13.68
13.48
13.13
1.00
1.12
1.00
0.13
1.49
1.43
1.43
1.27
f1 (ppm)


14
^{13}C-^{1}H Coupled
101 MHz (THF)
CuMe
N
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
0
5E+06
1E+07
2E+07
2E+07
2E+07
3E+07
4E+07


15
13C 125 MHz (THF)
C≡N:-Li
163.59
67.19
44.18
41.22
34.42
31.96
29.52
29.08
28.74
27.68
25.37
24.99
24.92
22.90
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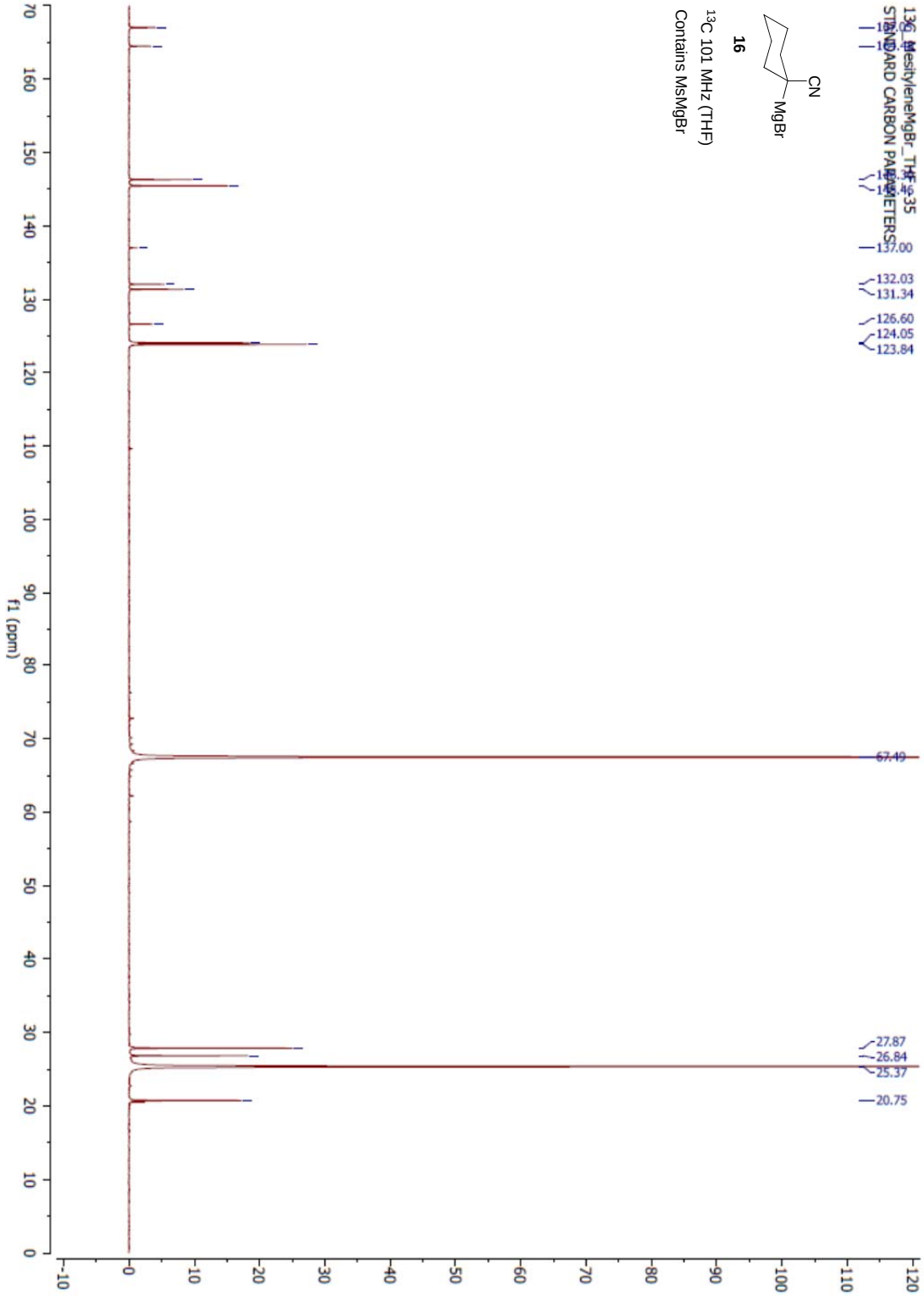
13.64
f1 (ppm)


136 MesityleneMgBr_THF 35
STANDARD CARBON PARAMETERS
137.00
132.03
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124.05
123.84
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25.37
20.75
16
CN
MgBr
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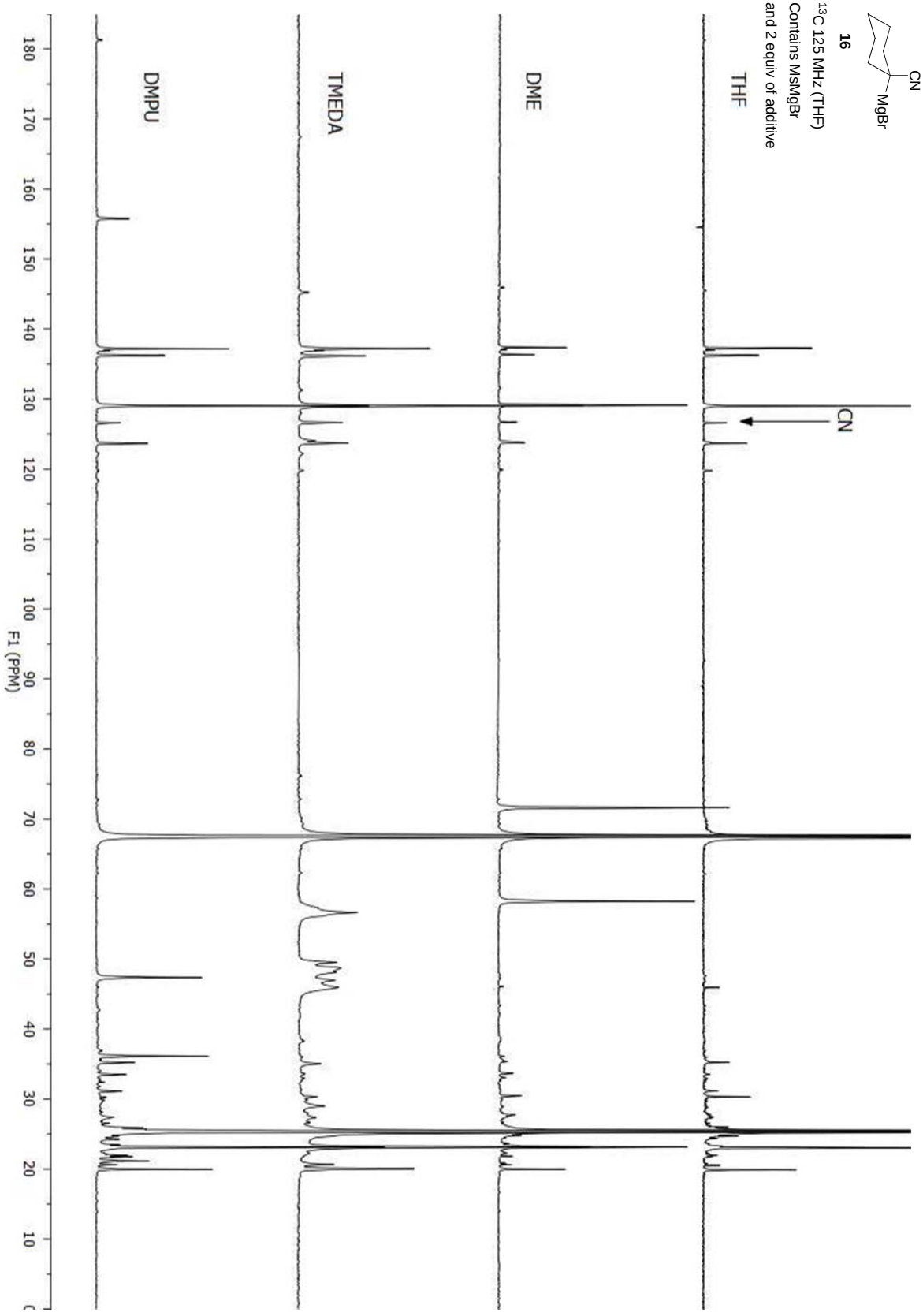
Contains MsMgBr
f1 (ppm)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MgBr
16
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Contains MsMgBr
and 2 equiv of additive
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TMEDA
DMPU
CN
F1 (PPM)
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20
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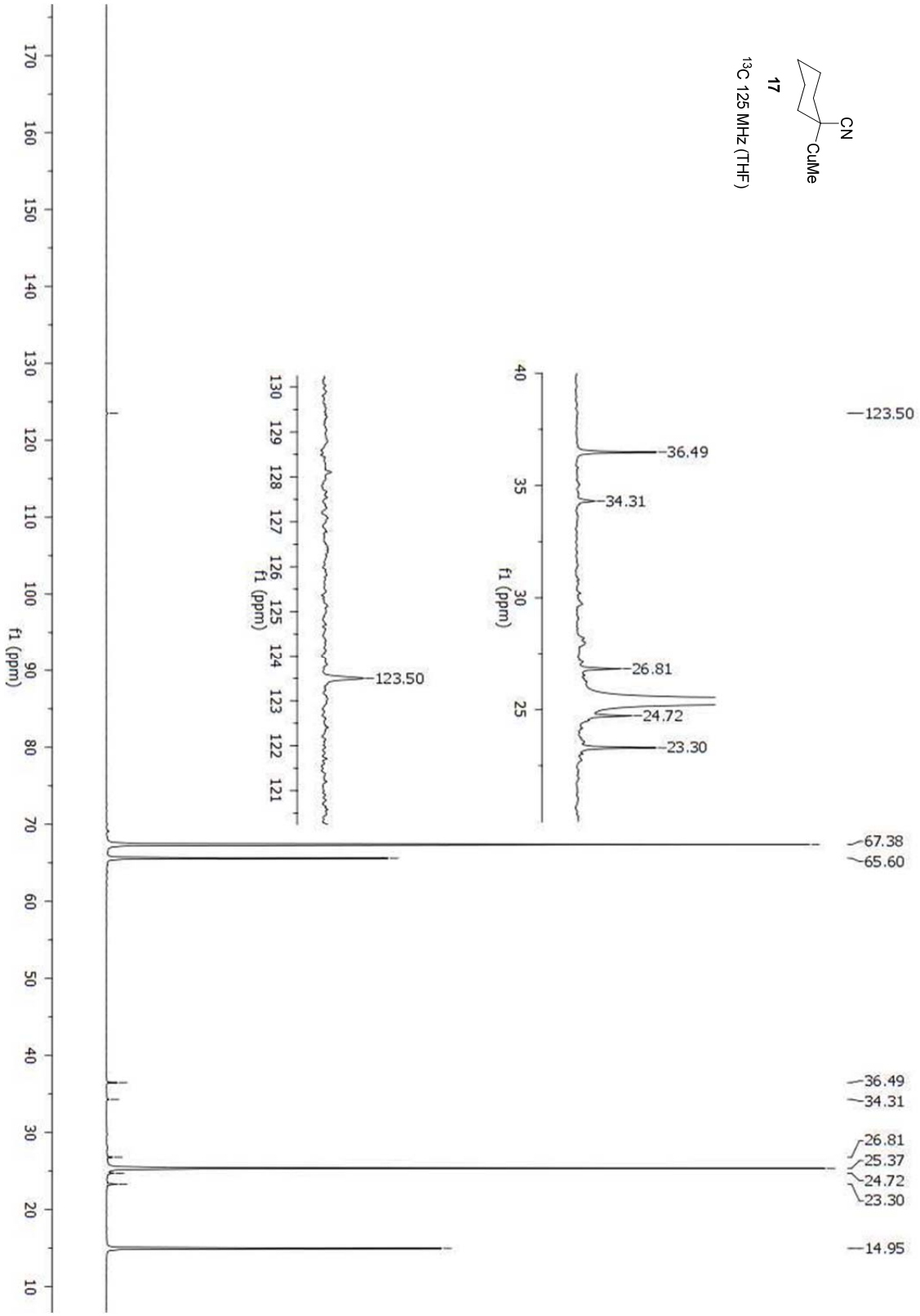
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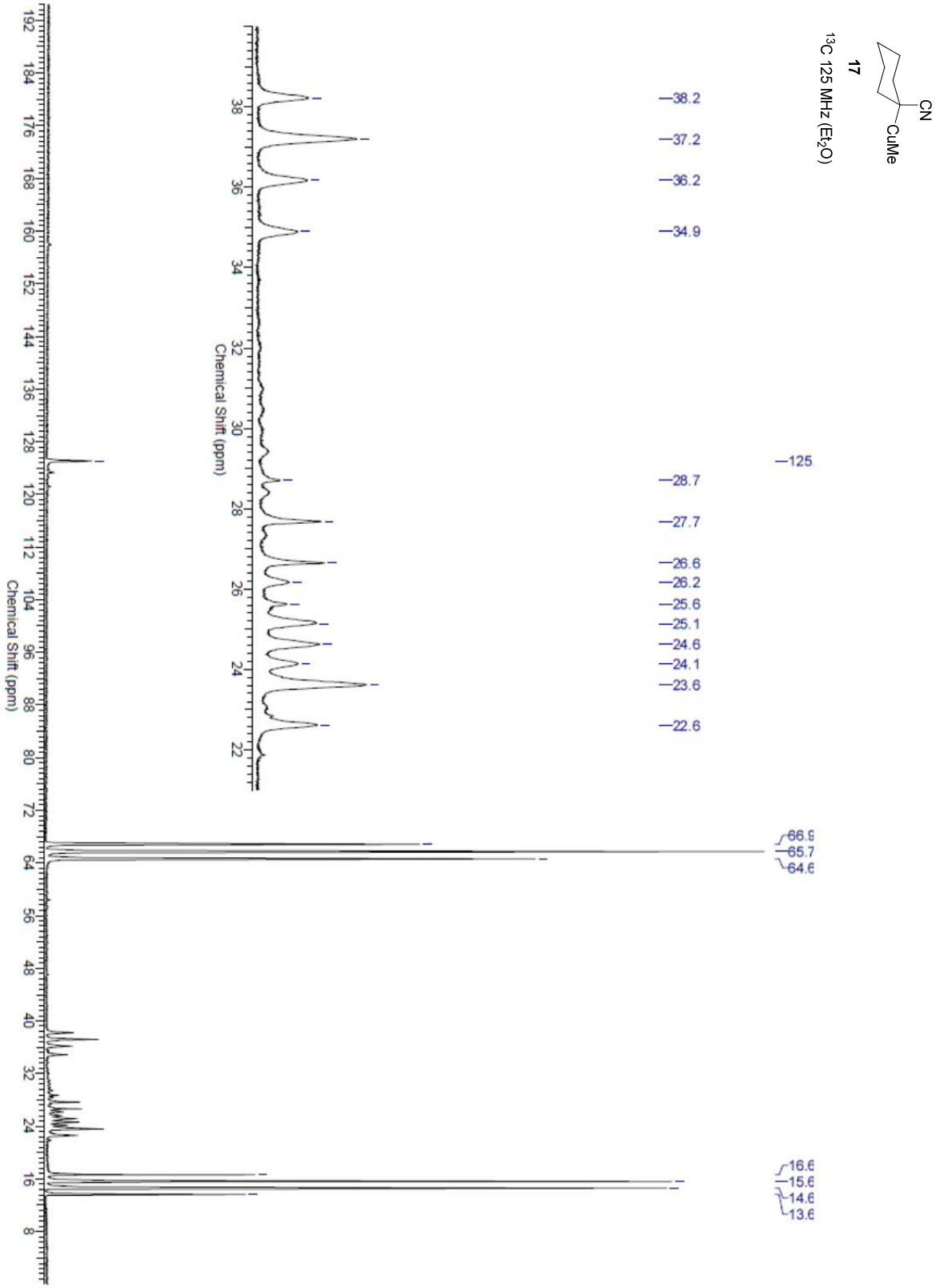
14.95
f1 (ppm)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CN
CuMe
Chemical Shift (ppm)
Chemical Shift (ppm)
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36.2
34.9
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27.7
26.6
26.2
25.6
25.1
24.6
24.1
23.6
22.6
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CIF Data cyano(2-methoxyphenyl)methanide magnesium chloride

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 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
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Cl5 Cl 0.3829(3) -0.0727(2) 0.5747(2) 0.0826(7) Uani 1 1 d . . .

Mg2 Mg 0.7169(2) 0.59097(18) 0.08672(15) 0.0310(4) Uani 1 1 d . . .

O2 O 0.0726(6) 0.0171(5) 0.4622(4) 0.0633(15) Uani 1 1 d . . .

O1 O 0.3489(6) 0.1744(5) 0.4644(4) 0.0586(14) Uani 1 1 d . . .

C2 C 0.5679(15) 0.2129(17) 0.3619(11) 0.136(7) Uani 1 1 d . . .

C3 C 0.469(2) 0.3013(18) 0.3551(15) 0.203(12) Uani 1 1 d . . .

C1 C 0.445(3) 0.1171(17) 0.381(2) 0.262(16) Uani 1 1 d . . .

C5 C -0.077(2) 0.0679(16) 0.428(2) 0.255(17) Uani 1 1 d . . .

C4 C 0.104(2) -0.0969(15) 0.4189(18) 0.223(12) Uani 1 1 d . . .

C6 C -0.041(2) -0.1181(18) 0.3589(14) 0.153(8) Uani 1 1 d . . .

N1 N 0.8798(5) 0.7588(6) 0.0854(5) 0.0241(14) Uani 1 1 d . . .

Cl3 Cl 0.5478(4) 0.4286(3) 0.0883(2) 0.0949(9) Uani 1 1 d . . .

O4 O 0.5770(5) 0.6670(4) -0.0282(3) 0.0445(11) Uani 1 1 d . . .

O6 O 0.8573(5) 0.5182(4) -0.0257(4) 0.0504(12) Uani 1 1 d . . .

C7 C 0.351(2) 0.2850(19) 0.448(2) 0.30(2) Uani 1 1 d . . .

C8 C 1.0258(9) 0.5374(8) -0.0405(8) 0.073(3) Uani 1 1 d . . .

C9 C 1.0654(12) 0.4678(11) -0.1297(9) 0.096(4) Uani 1 1 d . . .

C10 C 0.9428(12) 0.3730(10) -0.1382(9) 0.097(4) Uani 1 1 d . . .

C13 C 0.3532(11) 0.7141(9) -0.1297(7) 0.082(3) Uani 1 1 d . . .

C12 C 0.4827(12) 0.8006(12) -0.1405(8) 0.108(5) Uani 1 1 d . . .

C14 C 0.4147(9) 0.6346(9) -0.0473(10) 0.097(4) Uani 1 1 d . . .

C15 C 0.6269(10) 0.7335(8) -0.1070(6) 0.070(2) Uani 1 1 d . . .

C16 C -0.1488(18) -0.0260(19) 0.3614(9) 0.135(6) Uani 1 1 d . . .

C17 C 0.8034(10) 0.4382(10) -0.1054(7) 0.084(3) Uani 1 1 d . . .

O7 O 0.8565(6) 0.5196(5) 0.2025(4) 0.0632(15) Uani 1 1 d . . .

O8 O 0.0741(5) 0.0173(4) 0.6905(4) 0.0504(12) Uani 1 1 d . . .

C18 C 0.1263(10) -0.0628(10) 0.7671(7) 0.084(3) Uani 1 1 d . . .

C19 C -0.0916(8) 0.0394(8) 0.7077(8) 0.079(3) Uani 1 1 d . . .

C20 C -0.1398(13) -0.0327(11) 0.7944(9) 0.094(4) Uani 1 1 d . . .

C21 C -0.0134(11) -0.1205(11) 0.8047(9) 0.093(4) Uani 1 1 d . . .

C28 C 1.006(3) 0.5586(19) 0.234(2) 0.34(2) Uani 1 1 d . . .

C27 C 0.827(2) 0.4125(15) 0.252(2) 0.222(13) Uani 1 1 d . . .

C29 C 1.0716(19) 0.467(2) 0.2996(10) 0.147(8) Uani 1 1 d . . .

C30 C 0.966(3) 0.3828(18) 0.3082(13) 0.151(8) Uani 1 1 d . . .

O9 O 0.3520(5) 0.1654(4) 0.6920(3) 0.0455(11) Uani 1 1 d . . .

C32 C 1.0884(13) 0.9859(8) 0.0778(8) 0.082(3) Uani 1 1 d . . .

C33 C 1.1120(12) 0.8714(9) 0.0815(11) 0.094(3) Uani 1 1 d . . .

C31 C 0.9375(17) 1.0570(13) 0.0795(12) 0.108(4) Uani 1 1 d . . .

O10 O 0.5808(6) 0.6729(5) 0.1972(4) 0.0610(15) Uani 1 1 d . . .

C34 C 0.582(2) 0.7891(17) 0.2114(19) 0.263(17) Uani 1 1 d . . .

C36 C 0.939(2) 0.7731(11) 0.0829(8) 0.100(5) Uani 1 1 d . . .

C38 C 0.9023(16) 1.1514(13) 0.0767(12) 0.115(5) Uani 1 1 d . . .

O11 O 1.3686(12) 0.9884(13) 0.0735(8) 0.161(6) Uani 1 1 d . . .

C40 C 1.0556(19) 1.200(2) 0.080(2) 0.236(14) Uani 1 1 d . . .

C42 C 1.1850(13) 1.1843(9) 0.0832(8) 0.077(3) Uani 1 1 d . . .

C41 C 1.2253(12) 1.0794(13) 0.0761(8) 0.084(3) Uani 1 1 d . . .

C47 C 0.0235(14) 0.6554(8) 0.5806(12) 0.103(4) Uani 1 1 d . . .

C51 C 0.5759(9) 0.2175(9) 0.7873(7) 0.078(3) Uani 1 1 d . . .

C50 C 0.4443(12) 0.3001(11) 0.8067(9) 0.102(4) Uani 1 1 d . . .

C48 C 0.5173(11) 0.1355(10) 0.7117(12) 0.102(4) Uani 1 1 d . . .

C49 C 0.3014(11) 0.2372(9) 0.7664(7) 0.077(3) Uani 1 1 d . . .

C54 C 0.452(2) 0.7933(18) 0.3076(10) 0.145(7) Uani 1 1 d . . .

C53 C 0.3518(17) 0.7134(15) 0.2990(12) 0.123(5) Uani 1 1 d . . .

C57 C -0.2857(16) 0.585(2) 0.5886(7) 0.127(6) Uani 1 1 d . . .

C55 C 0.0008(13) 0.5429(11) 0.5859(9) 0.089(3) Uani 1 1 d . . .

C58 C -0.2531(11) 0.6788(9) 0.5835(7) 0.067(2) Uani 1 1 d . . .

C59 C -0.1165(13) 0.7152(17) 0.5798(11) 0.149(8) Uani 1 1 d . . .

C56 C -0.1490(11) 0.4895(7) 0.5829(5) 0.060(2) Uani 1 1 d . . .

C60 C -0.1785(11) 0.3696(7) 0.5814(9) 0.075(2) Uani 1 1 d . . .

O12 O -0.413(2) 0.5008(13) 0.5898(8) 0.174(6) Uani 1 1 d . . .

C61 C 0.475(3) 0.609(2) 0.268(2) 0.259(16) Uani 1 1 d . . .

C64 C 1.4960(15) 1.101(3) 0.0736(15) 0.232(15) Uani 1 1 d . . .

N2 N 0.0505(5) 0.2542(4) 0.5760(3) 0.0235(10) Uani 1 1 d . . .

C68 C -0.5751(18) 0.576(3) 0.5852(13) 0.28(2) Uani 1 1 d . . .

C69 C -0.0342(15) 0.2879(8) 0.5808(6) 0.076(3) Uani 1 1 d . . .

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Cl5 0.0721(15) 0.0716(15) 0.1040(17) -0.0052(13) 0.0080(12) 0.0074(12)

Mg2 0.0308(8) 0.0249(8) 0.0374(9) 0.0012(6) 0.0000(6) -0.0010(6)

O2 0.049(3) 0.061(3) 0.080(3) -0.033(3) -0.012(3) 0.001(3)

O1 0.055(3) 0.074(4) 0.046(2) 0.007(2) 0.007(2) -0.013(3)

C2 0.075(8) 0.216(19) 0.117(10) 0.051(11) 0.056(7) -0.019(10)

C3 0.150(13) 0.23(2) 0.228(19) 0.197(18) 0.122(14) 0.086(14)

C1 0.31(3) 0.141(14) 0.34(3) -0.074(16) 0.27(3) -0.132(16)

C5 0.187(16) 0.181(17) 0.40(3) -0.23(2) -0.23(2) 0.111(14)

C4 0.204(18) 0.148(14) 0.32(3) -0.192(18) -0.085(19) 0.004(14)

C6 0.109(11) 0.199(18) 0.151(13) -0.133(14) 0.009(10) -0.032(11)

N1 0.0035(15) 0.031(3) 0.038(3) -0.012(2) -0.0029(15) -0.014(2)

Cl3 0.0913(19) 0.0811(18) 0.112(2) 0.0033(15) 0.0067(15) -0.0129(15)

O4 0.043(2) 0.051(3) 0.040(2) 0.0085(19) -0.0112(17) -0.007(2)

O6 0.040(2) 0.044(3) 0.067(3) -0.019(2) 0.007(2) -0.009(2)

C7 0.216(19) 0.24(2) 0.45(4) 0.31(3) 0.24(2) 0.160(17)

C8 0.039(4) 0.073(6) 0.107(6) -0.050(5) 0.021(4) -0.016(4)

C9 0.069(6) 0.122(9) 0.098(7) -0.083(7) 0.029(5) -0.025(6)

C10 0.070(6) 0.098(8) 0.124(8) -0.075(7) 0.023(6) -0.028(6)

C13 0.072(5) 0.093(7) 0.082(5) 0.057(5) -0.039(4) -0.034(5)

C12 0.074(6) 0.146(11) 0.104(8) 0.076(8) -0.041(5) -0.057(7)

C14 0.038(4) 0.087(7) 0.167(10) 0.073(7) -0.031(5) -0.023(4)

C15 0.065(5) 0.077(6) 0.068(5) 0.038(4) 0.003(4) 0.000(4)

C16 0.105(10) 0.224(19) 0.076(7) -0.033(9) -0.043(7) -0.046(11)

C17 0.057(5) 0.130(9) 0.066(5) -0.057(6) -0.011(4) 0.005(5)

O7 0.044(3) 0.069(4) 0.076(3) 0.022(3) -0.007(2) 0.001(3)

O8 0.034(2) 0.056(3) 0.061(3) 0.032(2) 0.0133(19) 0.021(2)

C18 0.053(4) 0.129(9) 0.071(5) 0.056(6) -0.004(4) 0.018(5)

C19 0.032(3) 0.083(6) 0.121(7) 0.060(5) 0.032(4) 0.018(3)

C20 0.085(7) 0.099(8) 0.097(7) 0.064(6) 0.030(5) 0.000(6)

C21 0.055(5) 0.109(8) 0.114(8) 0.069(7) 0.003(5) 0.009(5)

C28 0.32(3) 0.20(2) 0.49(4) 0.18(3) -0.37(3) -0.10(2)

C27 0.154(14) 0.135(13) 0.38(3) 0.201(19) -0.080(18) -0.047(12)

C29 0.114(11) 0.25(2) 0.080(7) 0.040(11) -0.024(7) 0.096(13)

C30 0.156(15) 0.178(18) 0.118(11) 0.101(12) 0.008(11) 0.049(13)

O9 0.036(2) 0.062(3) 0.039(2) -0.008(2) -0.0051(17) 0.000(2)

C32 0.101(7) 0.061(6) 0.083(6) -0.007(4) 0.010(5) -0.048(5)

C33 0.055(5) 0.066(6) 0.160(10) 0.012(6) -0.011(6) -0.013(4)

C31 0.102(9) 0.093(9) 0.130(10) 0.032(8) -0.008(8) 0.018(8)

O10 0.045(3) 0.090(4) 0.048(3) -0.027(3) 0.005(2) 0.006(3)

C34 0.191(16) 0.208(18) 0.39(3) -0.26(2) 0.22(2) -0.137(15)

C36 0.179(13) 0.084(9) 0.037(4) -0.005(5) -0.010(7) 0.114(9)

C38 0.092(8) 0.099(10) 0.153(12) 0.034(9) -0.032(8) -0.037(8)

O11 0.100(7) 0.278(16) 0.106(6) -0.047(8) 0.015(5) -0.091(9)

C40 0.094(11) 0.30(3) 0.31(3) 0.09(2) -0.055(14) -0.153(17)

C42 0.068(6) 0.076(7) 0.086(6) 0.010(5) 0.002(5) -0.014(5)

C41 0.058(5) 0.137(11) 0.058(5) 0.002(5) 0.013(4) 0.042(6)

C47 0.088(8) 0.032(5) 0.189(13) -0.002(6) 0.015(8) 0.018(4)

C51 0.050(4) 0.106(7) 0.080(5) -0.055(5) -0.031(4) 0.020(5)

C50 0.068(6) 0.119(9) 0.118(8) -0.082(7) -0.041(6) 0.044(6)

C48 0.045(5) 0.079(7) 0.182(13) -0.021(8) -0.027(6) 0.003(5)

C49 0.069(5) 0.082(6) 0.081(6) -0.045(5) 0.010(4) -0.003(5)

C54 0.133(12) 0.220(18) 0.083(8) -0.088(10) 0.042(8) 0.038(12)

C53 0.098(9) 0.132(13) 0.138(11) -0.030(9) 0.080(9) 0.003(9)

C57 0.086(7) 0.260(19) 0.036(4) -0.008(7) 0.007(4) -0.093(10)

C55 0.079(7) 0.093(8) 0.096(7) 0.002(6) -0.011(5) -0.017(6)

C58 0.051(5) 0.074(6) 0.075(5) -0.006(4) 0.009(4) 0.010(4)

C59 0.056(6) 0.26(2) 0.128(10) -0.011(12) -0.001(6) 0.109(10)

C56 0.077(5) 0.058(5) 0.046(4) 0.014(3) 0.006(3) 0.029(4)

C60 0.068(5) 0.044(4) 0.113(7) -0.001(4) -0.001(5) 0.013(4)

O12 0.245(17) 0.189(12) 0.089(6) -0.012(7) -0.001(8) 0.018(12)

C61 0.24(2) 0.193(19) 0.35(3) -0.006(19) 0.26(3) 0.020(16)

C64 0.054(7) 0.48(4) 0.165(15) 0.07(2) -0.014(8) -0.112(15)

N2 0.0079(16) 0.030(2) 0.033(2) 0.0043(17) -0.0029(15) 0.0167(16)

C68 0.077(10) 0.68(6) 0.098(11) 0.02(2) 0.014(8) 0.18(2)

C69 0.112(9) 0.060(6) 0.055(4) -0.003(4) 0.003(5) -0.059(6)

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 are estimated using the full covariance matrix.  The cell esds are taken
 into account individually in the estimation of esds in distances, angles
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 used when they are defined by crystal symmetry.  An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.
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C13 C12 C15 102.3(9) . . ?

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C6 C16 C5 107.7(10) . . ?

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