-Supporting Information-

Synthesis of 3-substituted isoindolin-1-ones via a tandem desilylation-cross-coupling, hydroamidation sequence under aqueous phase-transfer conditions

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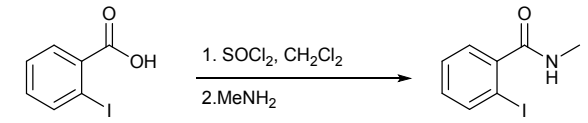
General

Unless otherwise mentioned, all the chemicals were purchased from commercial sources and used without further purification. Dry DMF was obtained by distillation over CaH_2 and the distillate was stored over molecular sieves in a Strauss flask under N_2 atmosphere. Water was degassed by several cycles (usually 3 cycles) of sonication- repressurization with N_2 under static vacuum. Flash column chromatography was performed to isolate products with suitable eluent as determined by TLC. ^{1}H, ^{13}C, and ^{19}F spectra were recorded on 400 MHz or 500 MHz Varian NMR spectrometers. ^{1}H NMR chemical shifts were determined relative to $CDCl_3$ as the internal standard at δ 7.26 ppm. ^{13}C NMR shifts were determined relative to $CDCl_3$ at δ 77.16 ppm. ^{19}F NMR chemical shifts were determined relative to $CFCl_3$ at δ 0.00 ppm. Mass spectra were recorded on a high resolution mass spectrometer, only for new compositional matter, the results are reported in the EI or in ESI mode.

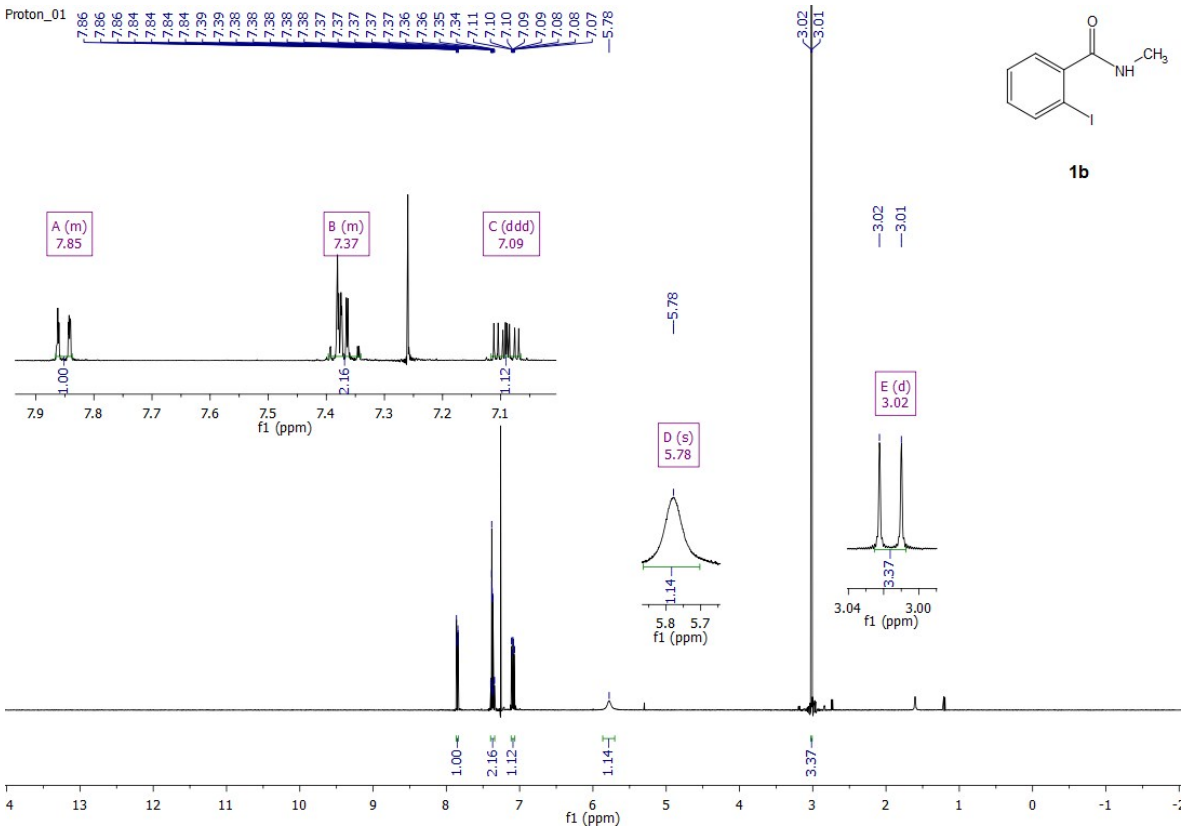
Preparation of benzamides substrates

2-iodo-N-methylbenzamide, 1b



[1b]

Benzoic acid (2.8 g, 11 mmol) were weighed into a 250 mL round bottom flask and 25 mL of dry dichloromethane (DCM) was added under nitrogen. To this suspension, $SOCl_2$ (1.57 g, 13.2 mmol) and a few drops of dimethylformamide (DMF) was added by syringe and stirred at room temperature overnight until the solution became complete clear which denoted the complete consumption of the benzoic. This solution was concentrated to half of its volume under reduced pressure and cooled down to 0 ^{o}C with an ice/water bath. To this solution, a pre-cooled, 40% aqueous solution of $MeNH_2$ (5 mL, 64 mmol) was added slowly (exothermic reaction) and the ensuing reaction was marked by the immediate formation of a white precipitate. Upon complete addition of $MeNH_2$, the mixture was allowed to warm up to room temperature and stirring was continued for 1 h. The solid was recovered by filtration, wash with cold DCM (15 mL) and cold water (15 mL). This solid was then dried under high vacuum overnight, affording an analytically pure **1b** as a white crystalline powder. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 – 7.83 (m, 1H), 7.39 – 7.36 (m, 2H), 7.09 (ddd, J = 8.0, 6.3, 2.9 Hz, 1H), 5.78 (s, 1H), 3.02 (d, J = 5.0 Hz, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.2, 142.4, 139.9, 131.1, 128.3, 128.2, 92.6, 26.8. These values are in agreement to those of the previously reported authentic compound.[8]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C (ddd)
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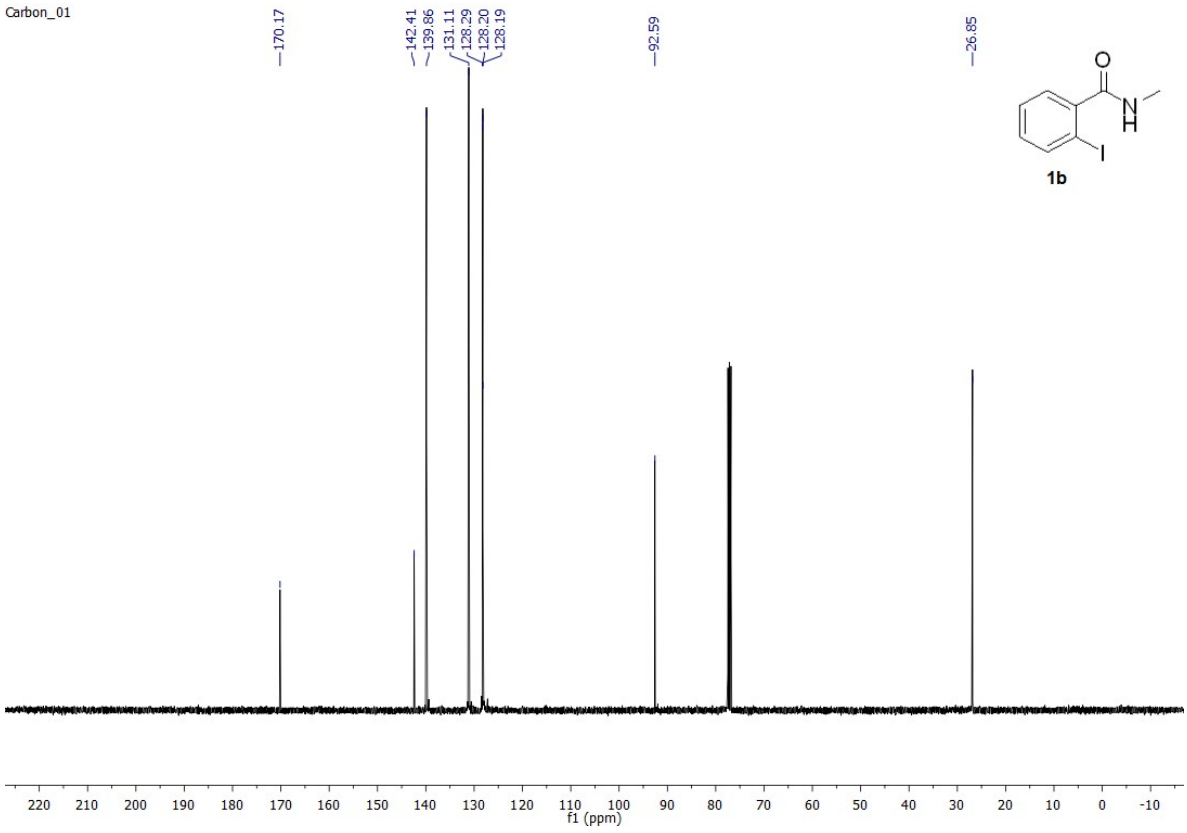
E (d)
3.02
1b
f1 (ppm)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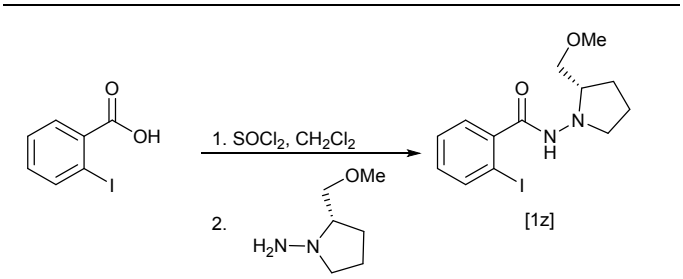
26.85
1b
f1 (ppm)

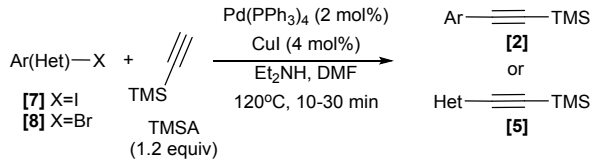
(S)-2-iodo-N-(2-(methoxymethyl)pyrrolidin-1-yl)benzamide, 1z



Benzoic acid (2 mmol, 496 mg) was weighed into a 50 mL round bottom flask and suspended in dry DCM (6 mL). The flask was closed with a septum under nitrogen. Subsequently, DMF (1 drop), $SOCl_2$ (1.2 eq, 2.4 mmol, 174 uL) were added via micro-syringe and the mixture was stirred at room temperature until the solution became clear (usually 2 h), which indicated the full consumption of benzoic acid. This solution of was then concentrated under reduced pressure to remove all volatiles to obtain crude 2-iodobenzoyl chloride. Subsequently, a fresh portion of dry DCM (6 mL) was added. In a separate vial, a solution of (S)-2-(methoxymethyl)pyrrolidin-1-amine (SAMP, 2 equiv, 4.4 mmol, 622 μL) in dry DCM (10 mL) was prepared and added dropwise to the solution of 2-iodobenzoyl chloride at 0°C. The mixture was allowed to warm up to room temperature and stirred for 30min.H2O (10 mL) was added and the product extracted with EtOAc (10 mL, 3 times). The organic layer was then concentrated and purified by flash column chromatography to obtain pure **1z** in 68% yield. Major rotamer **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 – 7.82 (m, 1H), 7.40 – 7.36 (m, 2H), 7.10 (ddd, *J* = 7.9, 5.7, 3.5 Hz, 1H), 6.68 (s, 1H), 3.64 (dd, *J* = 9.6, 5.2 Hz, 1H), 3.52 (dd, *J* = 9.6, 5.5 Hz, 1H), 3.46 (ddd, *J* = 8.9, 6.9, 4.1 Hz, 1H), 3.38 (d, *J* = 0.4 Hz, 3H), 3.23 (ddd, *J* = 13.2, 7.9, 5.3 Hz, 1H), 3.00 (q, *J* = 8.5, 8.5, 8.5 Hz, 1H), 2.12 – 2.01 (m, 1H), 1.96 – 1.86 (m, 2H), 1.77 – 1.64 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.1 , 141.2 , 139.9 , 131.3 , 128.5 , 128.2 , 92.9 , 75.3 , 64.4 , 59.4 , 55.3 , 26.7 , 21.5 . **HRMS (ESI)** Calcd. for $C_{13}H_{18}N_2O_2I$ (M+H)+ = 361.0413, Found = 361.0411.

Typical procedure for the preparation of trimethylsilylalkynes 2

Scheme 1. Preparation of silylalkynes **2** from (hetero)aryl halides

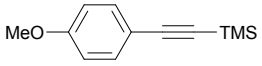


Trimethysilylalkynes **2** and some heterosilylalkynes **5**, were prepared from the corresponding (hetero)aryl iodides **7** or bromides **8** and trimethylsilylacetylene (TMSA) by a procedure adapted from a published report.[1] **2a**, **2e** and **5a-5e** are commercially available. A representative procedure is as follows:

The aryl iodide **7** or bromide **8** (3 mmol), $Pd(PPh_3)_4$ (69.3 mg, 0.06 mmol), CuI (22.9 mg, 0.12 mmol), trimethylsilylacetylene(0.51 mL, 3.6 mmol), diethylamine (4 mL) and dimethylformamide (1 mL) were mixed and stirred under nitrogen in a crimp-top vial at 120 °C until full consumption of starting material as determined by GC-MS analysis. Usually, aryl iodides **7**, required 5-10 min, while aryl bromides **8**, required from 15-30 min. Then, the reaction mixture was poured into 0.1 M aqueous HCl (5-10 mL) and extracted three times with diethyl ether (5-10 mL). The combined organic layers were washed with concentrated aqueous $NaHCO_3$ solution (5-10 mL) and water (5-10 mL) and then concentrated under reduced pressure. The residue was purified by flash chromatography using the appropriate eluent. The combined product fractions were concentrated on a rotatory evaporator.

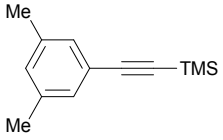
NMR Spectroscopic data of trimethylsilylalkynes 2.

((4-Methoxyphenyl)ethynyl)trimethylsilane, 2b



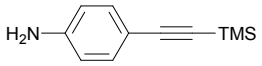
Obtained as yellow oil by the reaction of 4-Iodoanisole and TMSA in 86% isolated yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 – 7.38 (m, 2H), 6.84 – 6.79 (m, 2H), 3.80 (s, 3H), 0.24 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.9 , 133.6 , 115.4 , 113.9 , 105.3 , 92.6 , 55.4 , 0.2 . These values are in agreement to those of the previously reported authentic compound.[1]

((3,5-Dimethylphenyl)ethynyl)trimethylsilane, 2c



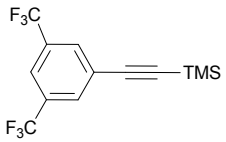
Obtained by the reaction of 1-iodo-3,5-dimethylbenzene and TMSA with 63% isolated yield as a yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.10 (s, 2H), 6.95 (s, 1H), 2.27 (s, 6H), 0.24 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 137.9 , 130.5 , 129.8 , 122.8 , 105.7 , 93.4 , 21.2 , 0.2 .

4-((Trimethylsilyl)ethynyl)aniline, 2d



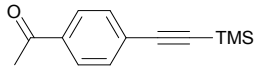
Obtained as a brown solid by the reaction of 4-iodoaniline and TMSA with 66% isolated yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29 – 7.25 (m, 2H), 6.60 – 6.54 (m, 2H), 3.79 (br. s, 2H, NH2), 0.22 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.9 , 133.5 , 114.7 , 112.6 , 106.1 , 91.5 , 0.3 . These values are in agreement to those of the previously reported authentic compound.[1]

((3,5-bis(trifluoromethyl)phenyl)ethynyl)trimethylsilane, 2f



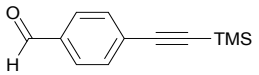
Obtained by the reaction of 1-bromo-3,5-bis(trifluoromethyl)benzene and TMSA with a 86% isolated yield as a colorless crystals after purification by sublimation as reported earlier. These values are in agreement to those of the previously reported authentic compound..[2] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 (s, 2H), 7.79 (s, 1H), 0.27 (d, J = 0.6 Hz, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 131.7 – 131.6 (m), 131.6 (q, J = 33.7, 33.7, 33.7 Hz), 125.3, 122.7 (q, J = 272.9, 272.8, 272.8 Hz), 121.6 (hept, J = 3.9, 3.9, 3.8, 3.8 Hz), 101.3, 98.6 , -0.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -63.7.

1-(4-((Trimethylsilyl)ethynyl)phenyl)ethanone, 2g



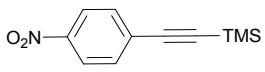
Obtained by the reaction of 1-(4-iodophenyl)ethanone and TMSA with a 83% isolated yield as a brown oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 – 7.86 (m, 2H), 7.56 – 7.51 (m, 2H), 2.60 (s, 3H), 0.26 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 197.5 , 136.5 , 132.2 , 128.3 , 128.1 , 104.1 , 98.3 , 26.8 , 0.0 . These values are in agreement to those of the previously reported authentic compound.[3]

4-((Trimethylsilyl)ethynyl)benzaldehyde, 2h



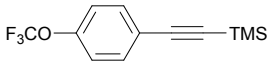
Obtained by the reaction of 4-iodobenzaldehyde and TMSA with a 53% isolated yield as a brown solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.00 (s, 1H), 7.94 – 7.72 (m, 2H), 7.70 – 7.51 (m, 2H), 0.27 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.6 , 135.7 , 132.6 , 129.6 , 129.5 , 104.0 , 99.2 , -0.1 . These values are in agreement to those of the previously reported authentic compound.[4]

Trimethyl((4-nitrophenyl)ethynyl)silane, 2i



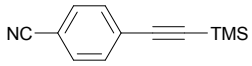
Obtained by the reaction of 1-iodo-4-nitrobenzene and TMSA with a 69% isolated yield as a orange solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.17 (m, 2H), 7.70 – 7.49 (m, 2H), 0.27 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 147.3 , 132.8 , 130.1 , 123.6 , 102.8 , 100.8 , -0.1 . These values are in agreement to those of the previously reported authentic compound.[5]

Trimethyl((4-(trifluoromethoxy)phenyl)ethynyl)silane, 2j



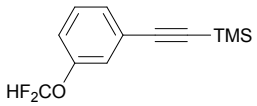
Obtained by the reaction of 1-iodo-4-(trifluoromethoxy)benzene and TMSA with a 70% isolated yield as a clear oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.50 – 7.46 (m, 2H), 7.16 – 7.12 (m, 2H), 0.25 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 149.1 (q, *J* = 1.8 Hz), 133.6 , 122.1 , 120.8 , 120.5 (q, *J* = 257.8 Hz), 103.6 , 95.4 , 0.0 **. ^{19}F NMR** (376 MHz, $CDCl_3$) δ -58.3 . **HRMS (EI)** Calcd for $C_{12}H_{13}OSiF_3$ (M^+) = 258.06879, found = 258.06883.

4-((Trimethylsilyl)ethynyl)benzonitrile, 2k



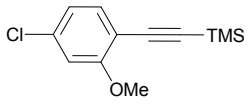
Obtained by the reaction of 4-bromobenzonitrile and TMSA with a 60% isolated yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.63 – 7.55 (m, 2H), 7.57 – 7.50 (m, 2H), 0.26 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 132.5 , 132.0 , 128.0 , 118.4 , 111.8 , 103.0 , 99.6 , -0.2 . These values are in agreement to those of the previously reported authentic compound..[1]

((3-(Difluoromethoxy)phenyl)ethynyl)trimethylsilane, 2l



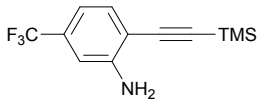
Obtained as a clear oil by the reaction of 1-(difluoromethoxy)-3-iodobenzene and TMSA with a 71% isolated yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 – 7.26 (m, 2H), 7.23 – 7.21 (m, 1H), 7.10 – 7.06 (m, 1H), 6.50 (t, *J* = 73.6 Hz, 1H), 0.25 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 151.0 , 129.8 , 129.2 , 125.0 , 122.9 , 120.2 , 115.9 (t, *J* = 260.3 Hz), 103.7 , 95.7 , 0.0. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -81.3 (d, *J* = 73.8 Hz). **HRMS (EI)** Calcd for $C_{12}H_{14}OSiF_2$ (M^+) = 240.07821, found = 240.07788.

((4-Chloro-2-methoxyphenyl)ethynyl)trimethylsilane, 2m



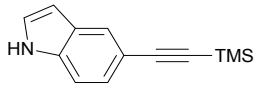
Obtained by the reaction of 4-chloro-1-iodo-2-methoxybenzene and TMSA with a 61% isolated yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 2.7 Hz, 1H), 7.22 (dd, *J* = 8.8, 2.7 Hz, 1H), 6.77 (d, *J* = 8.9 Hz, 1H), 3.86 (s, 3H), 0.26 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.1 , 133.7 , 129.8 , 125.2 , 114.0 , 111.9 , 100.1 , 99.8 , 56.3 , 0.1 . **HRMS (EI)** Calcd for $C_{12}H_{15}OClSi$ (M^+) = 238.05808, found = 238.05844

5-(Trifluoromethyl)-2-((trimethylsilyl)ethynyl)aniline, 2n

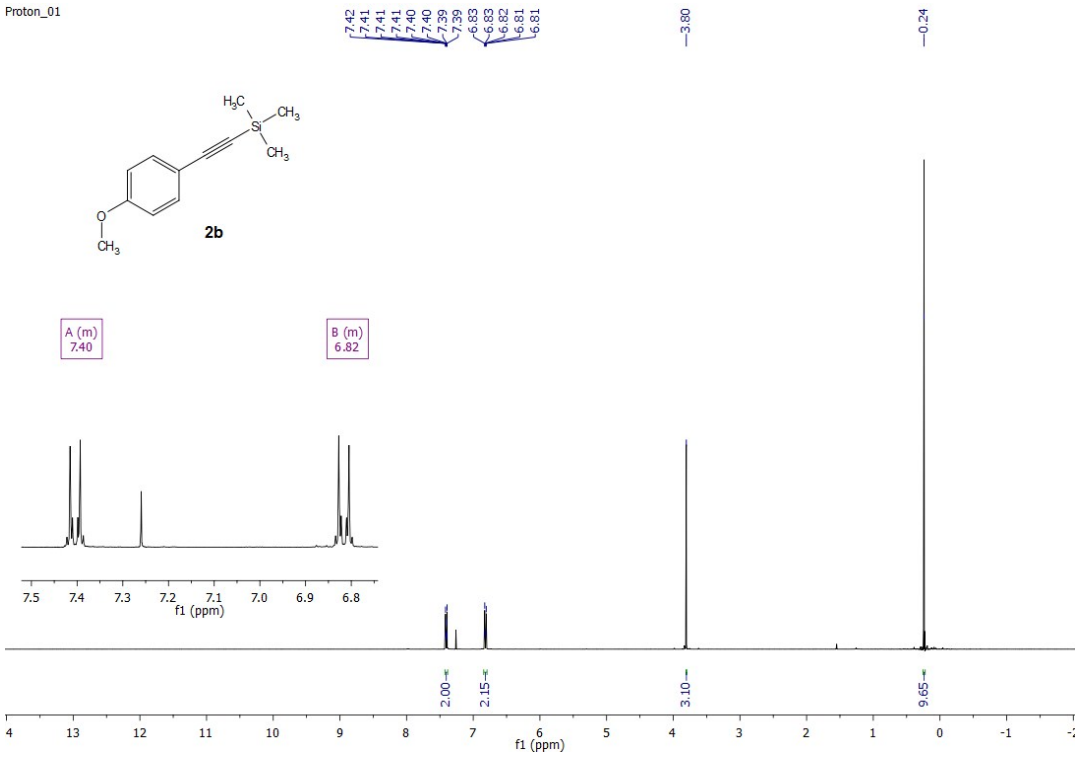


Obtained by the reaction of 2-iodo-5-(trifluoromethyl)aniline and TMSA with a 73% isolated yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 2.5 Hz, 1H), 7.32 (dd, *J* = 8.6, 2.3 Hz, 1H), 6.71 (d, *J* = 8.6 Hz, 1H), 4.53 (br. s, 2H), 0.27 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 150.7 , 129.8 (q, *J* = 3.7 Hz), 126.8 (q, *J* = 3.7 Hz), 124.4 (q, *J* = 270.7 Hz), 119.8 (q, *J* = 33.0 Hz), 113.7 , 107.5 , 101.3 , 100.2 , 0.2 . These values are in agreement to those of the previously reported authentic compound.[6]

5-((Trimethylsilyl)ethynyl)-1H-indole, 5f



Obtained by the reaction of 5-iodo-1H-indole and TMSA with a 61% isolated yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.17 (s, 1H), 7.81 (q, *J* = 1.0 Hz, 1H), 7.32 – 7.29 (m, 2H), 7.22 (dd, *J* = 3.3, 2.4 Hz, 1H), 6.52 (dd, *J* = 3.3, 2.0 Hz, 1H), 0.26 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.7 , 127.8 , 126.2 , 125.4 , 125.1 , 114.5 , 111.0 , 107.1 , 103.1 , 91.2 , 0.3 . These values are in agreement to those of the previously reported authentic compound.. [7]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6.81
3.80
0.24
2b
A (m)
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B (m)
6.82
7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8
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3.10
9.65
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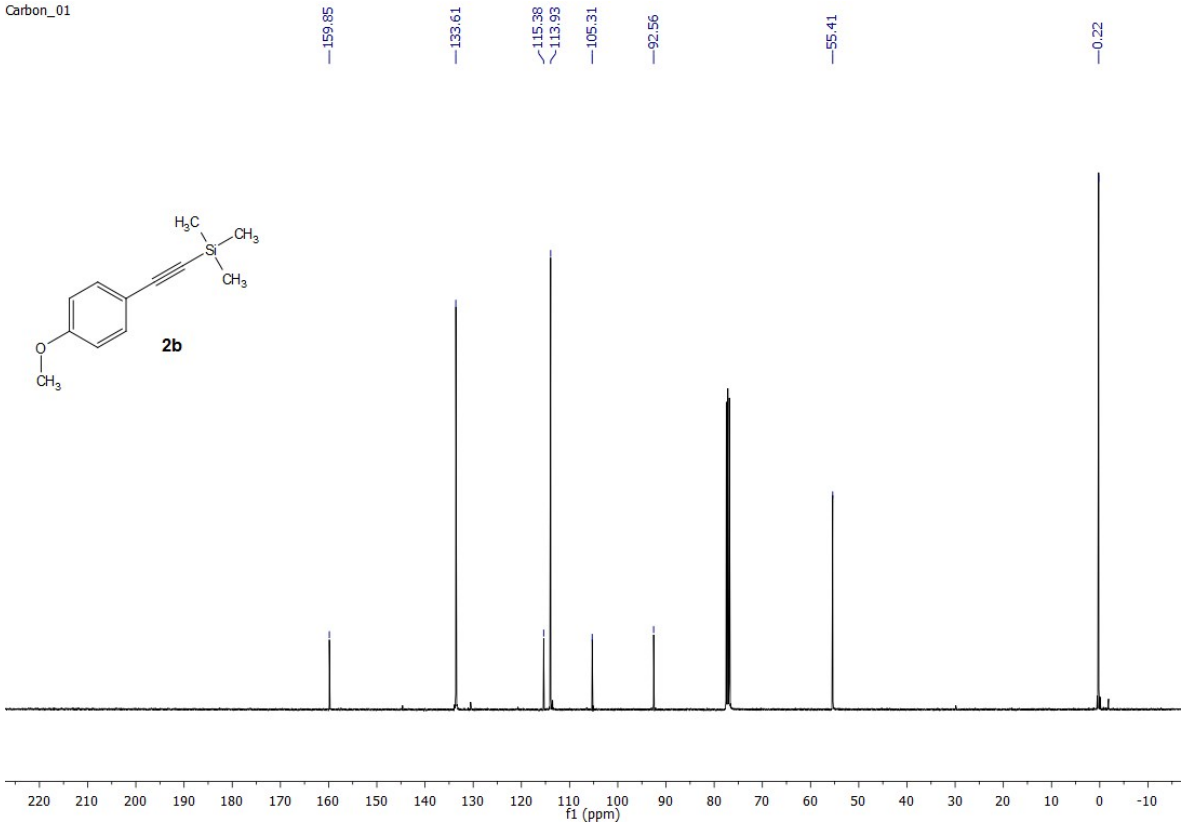
f1 (ppm)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2b
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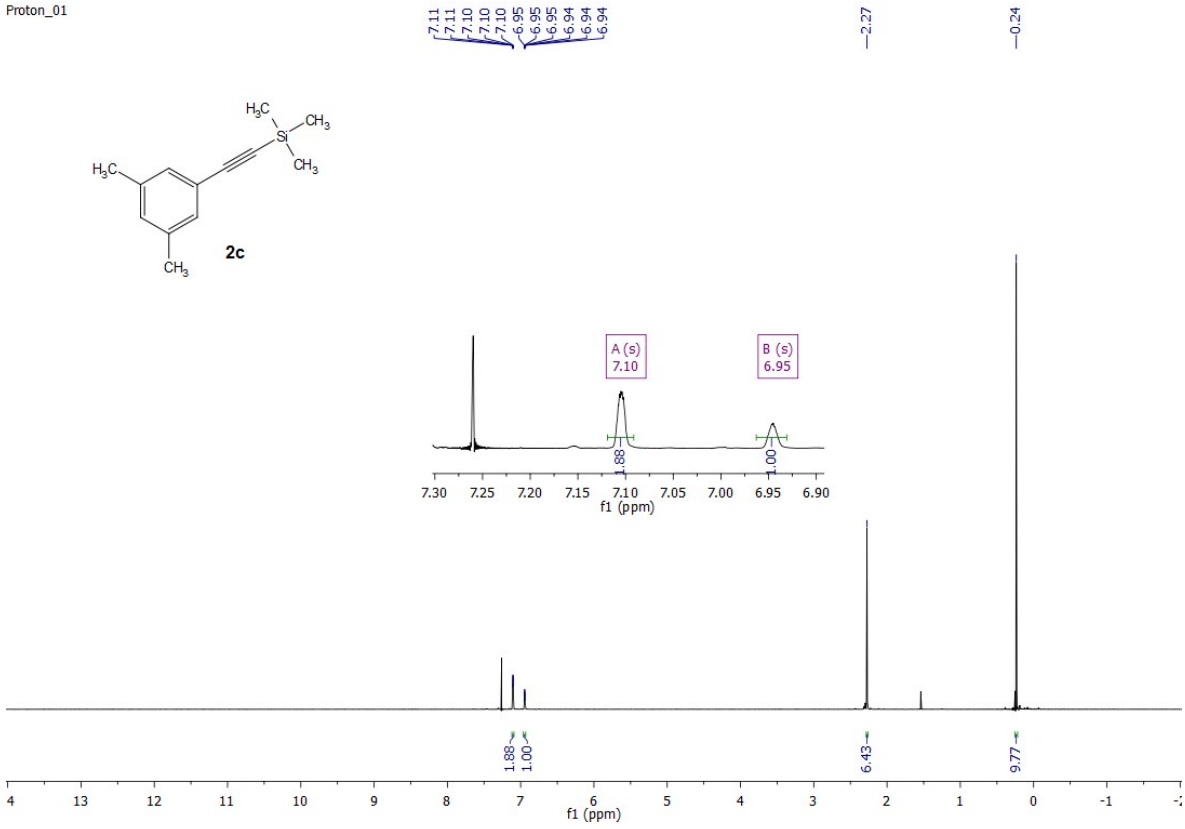
f1 (ppm)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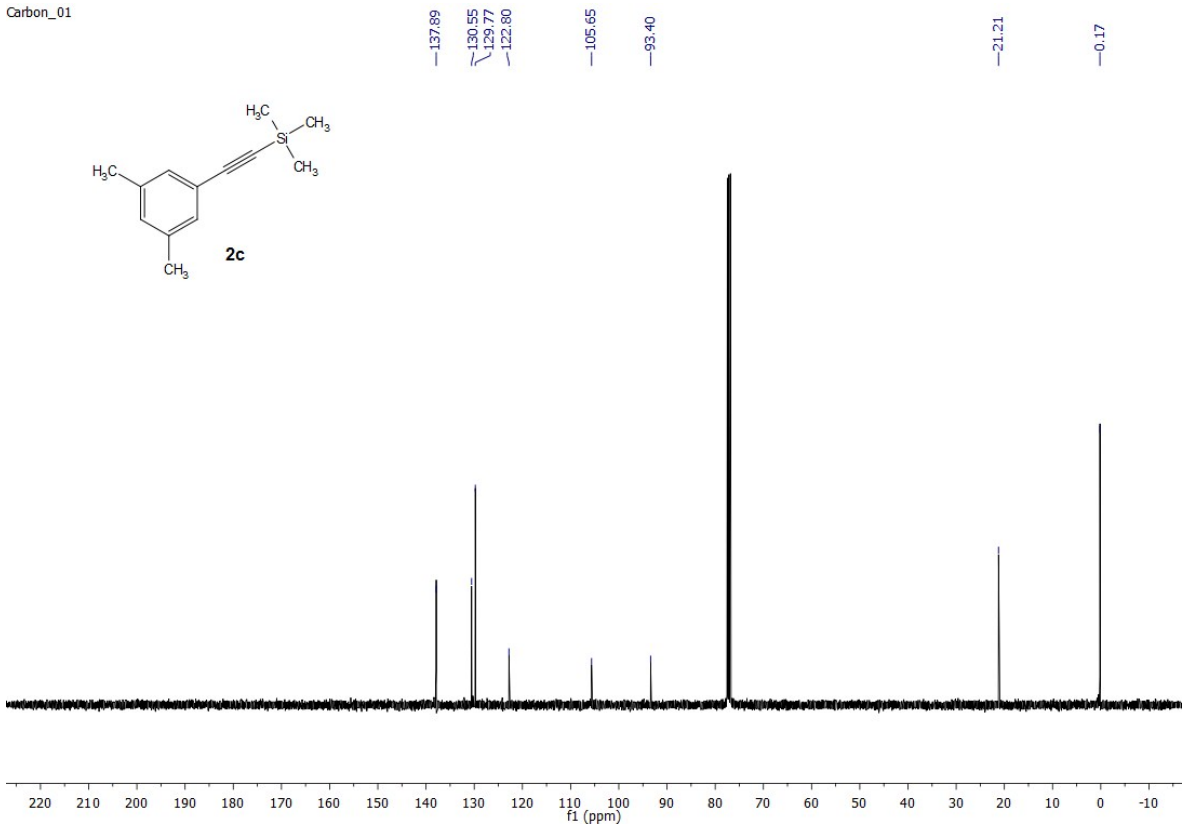
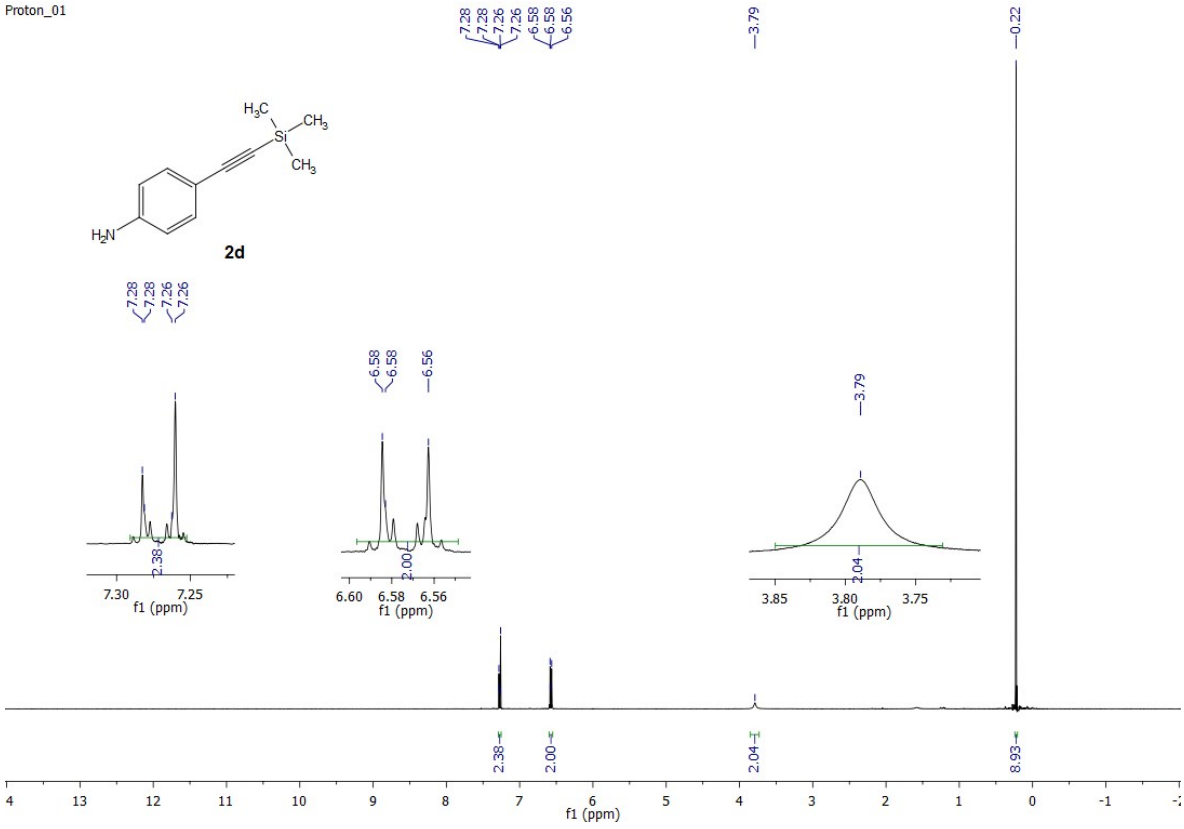
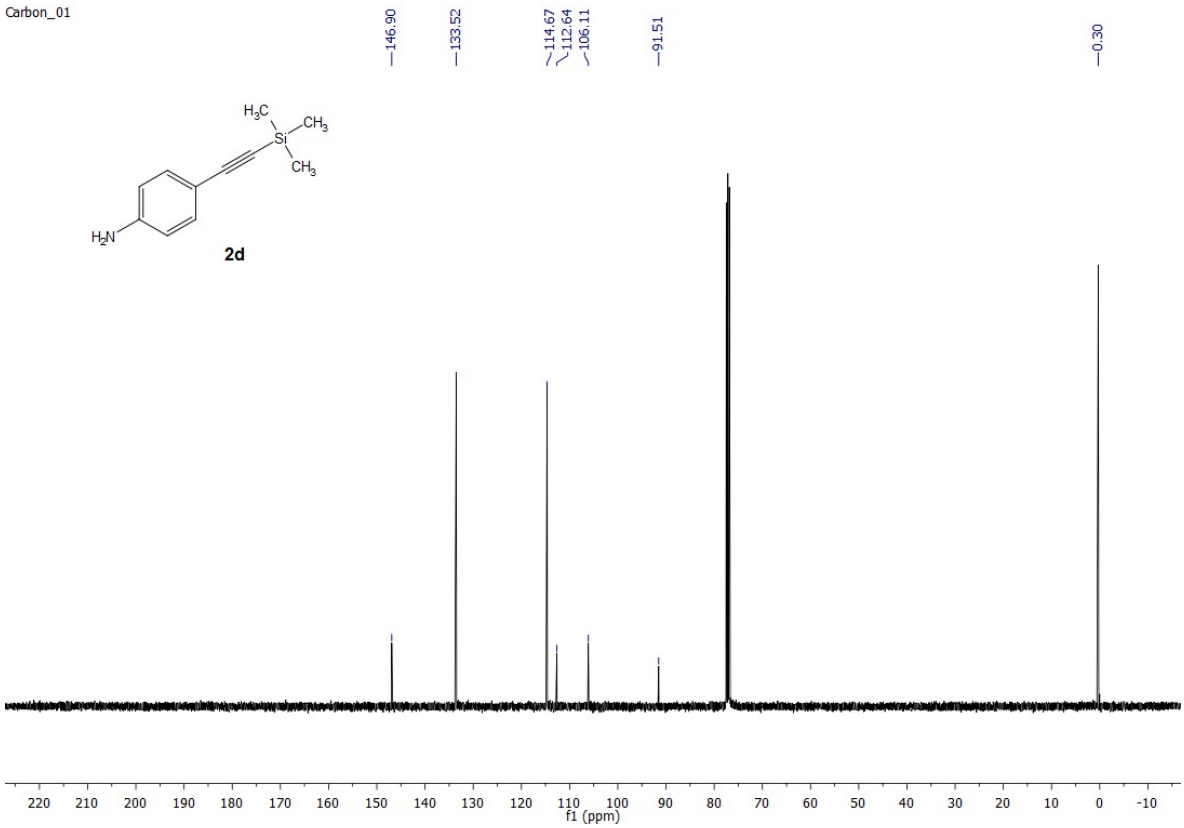
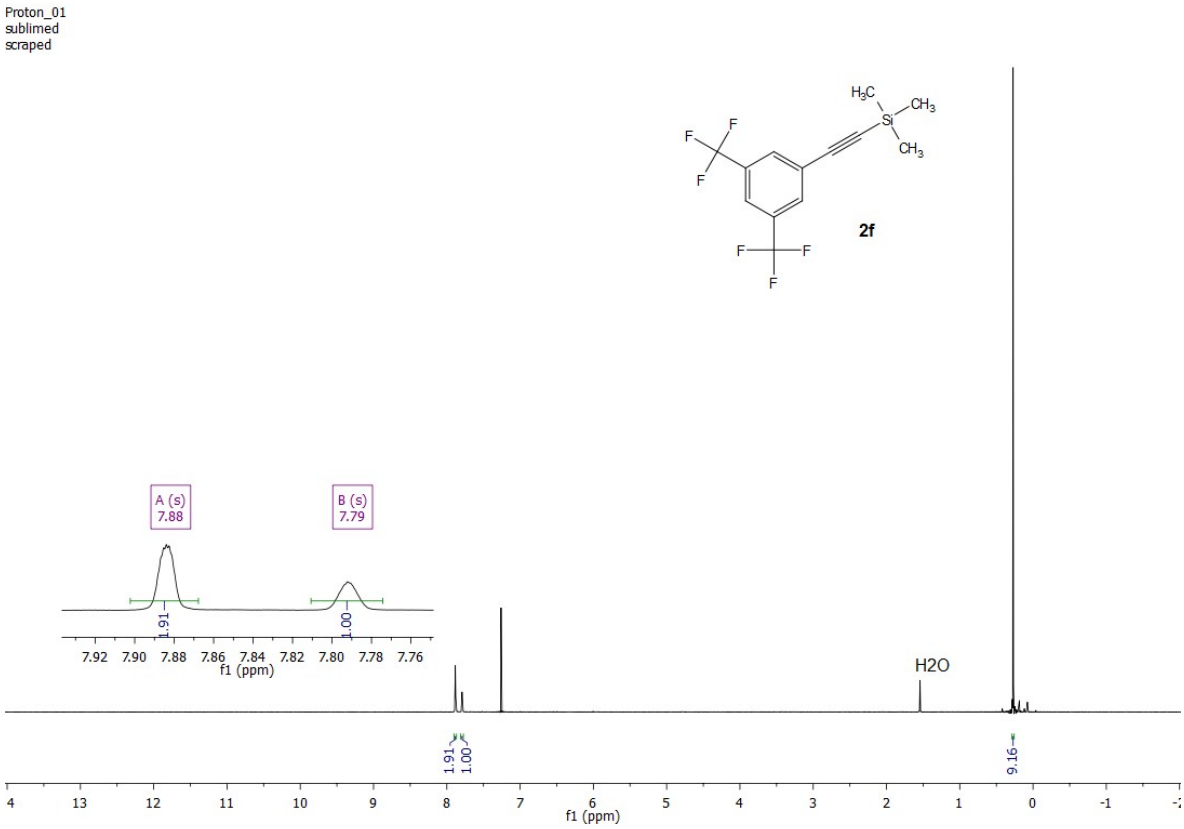
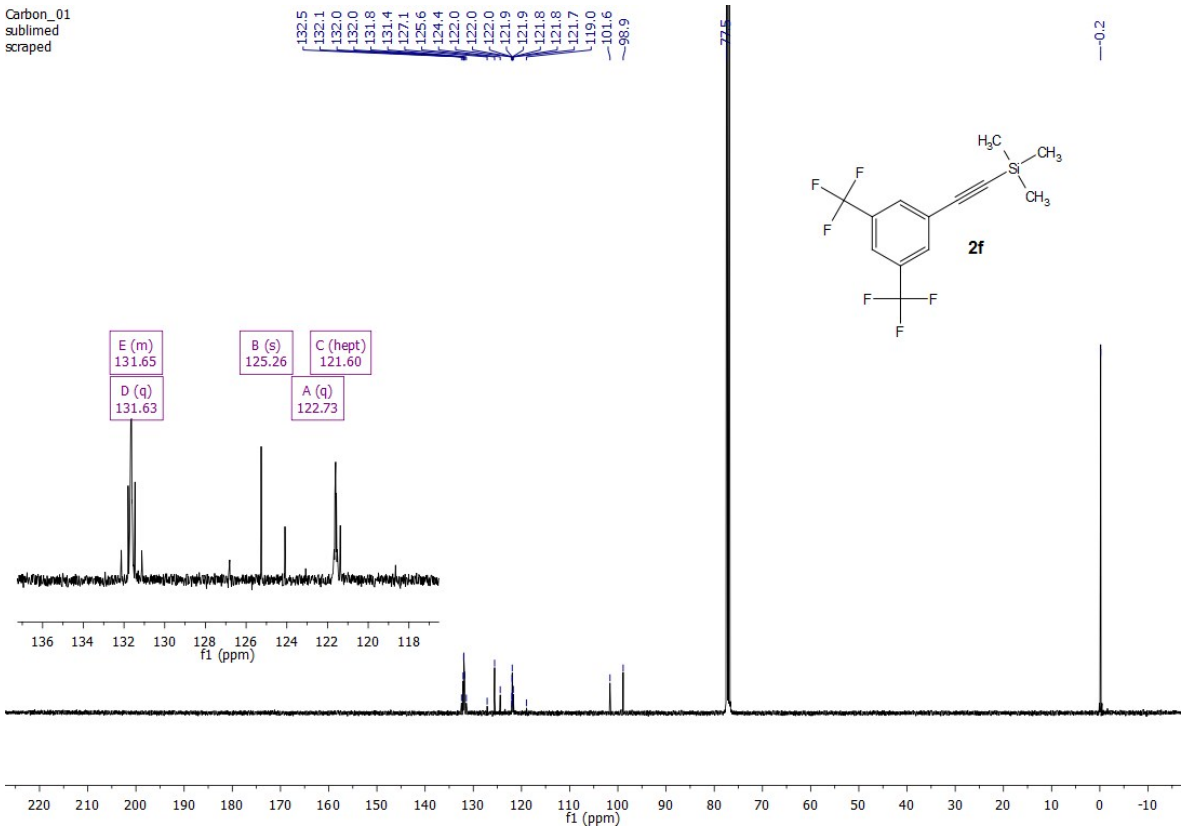
7.10
6.95
6.95
6.95
6.94
6.94
6.94
2.27
0.24
2c
A (s)
7.10
B (s)
6.95
1.88
1.00
7.30
7.25
7.20
7.15
7.10
7.05
7.00
6.95
6.90
f1 (ppm)
1.88
1.00
6.43
9.77
f1 (ppm)

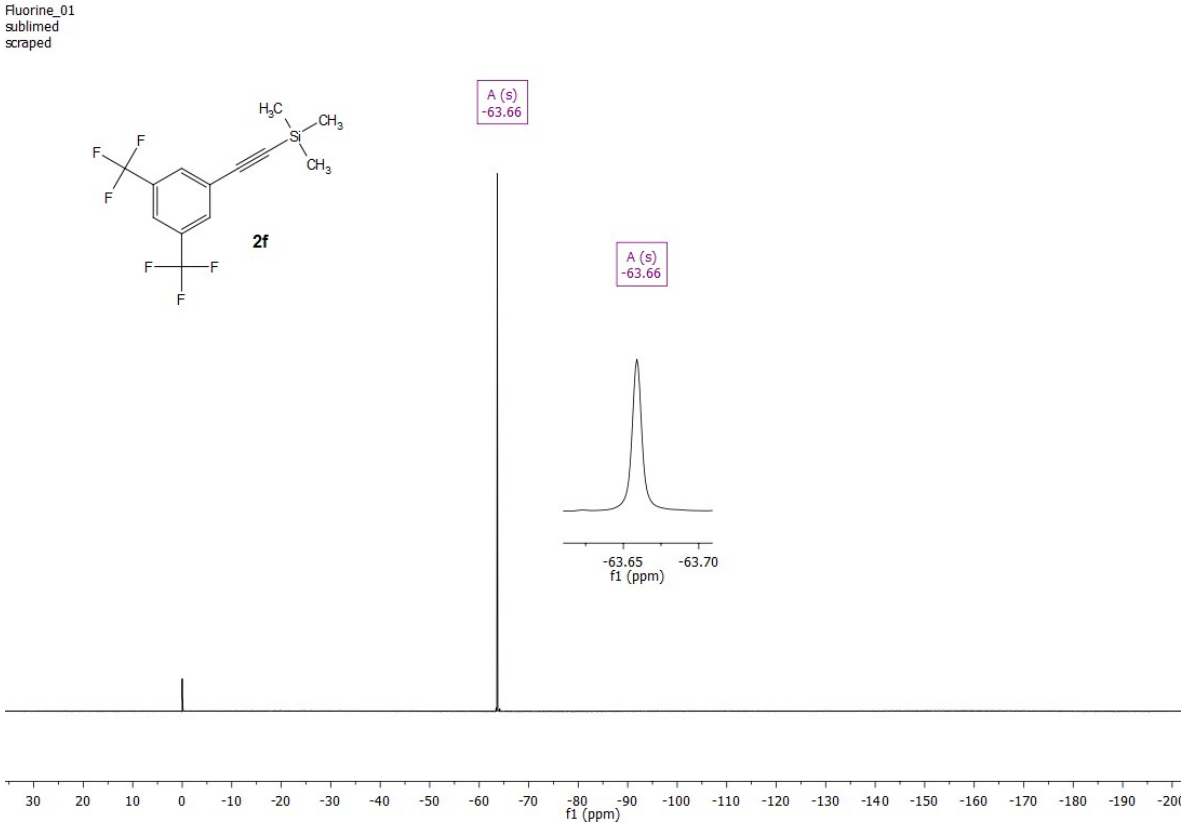

Carbon_01
137.89
130.55
129.77
122.80
105.65
93.40
21.21
0.17
2c
f1 (ppm)

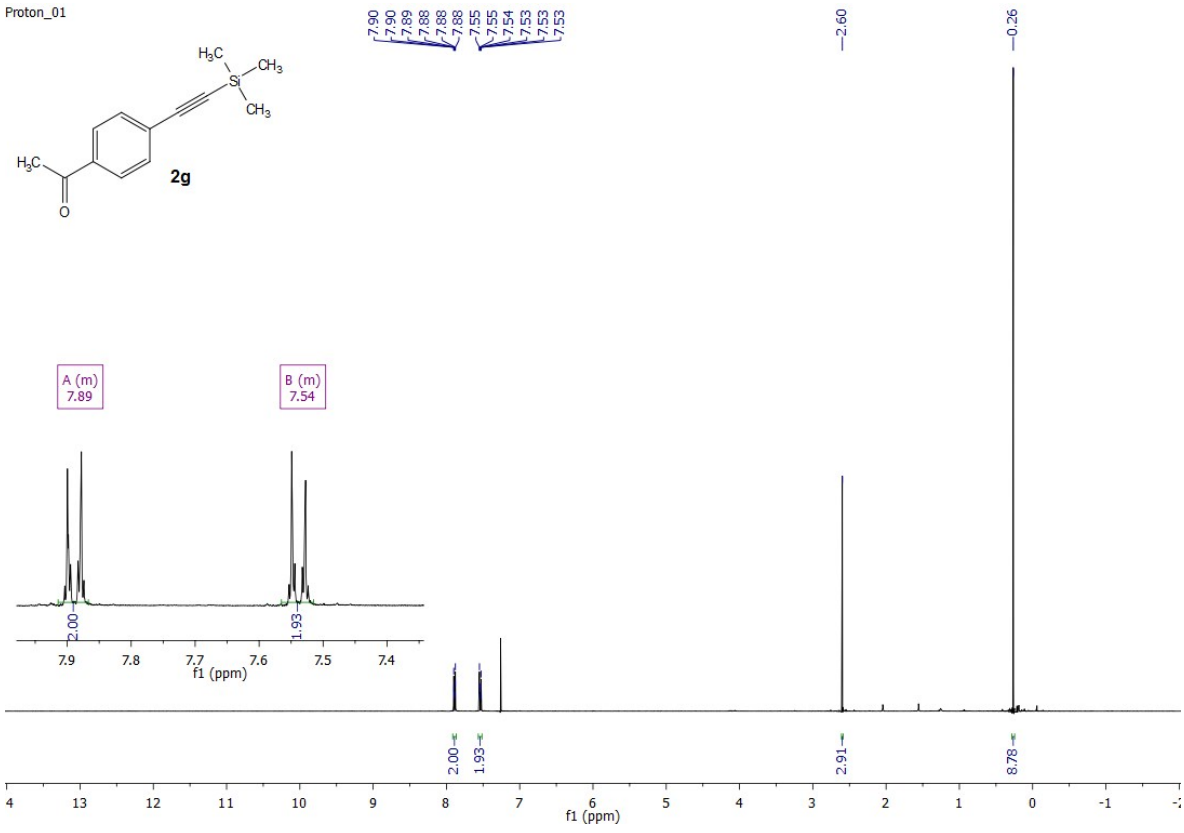

Proton_01
7.28
7.28
7.26
7.26
6.58
6.58
6.56
3.79
0.22
2d
2.38
2.00
2.04
8.93
f1 (ppm)

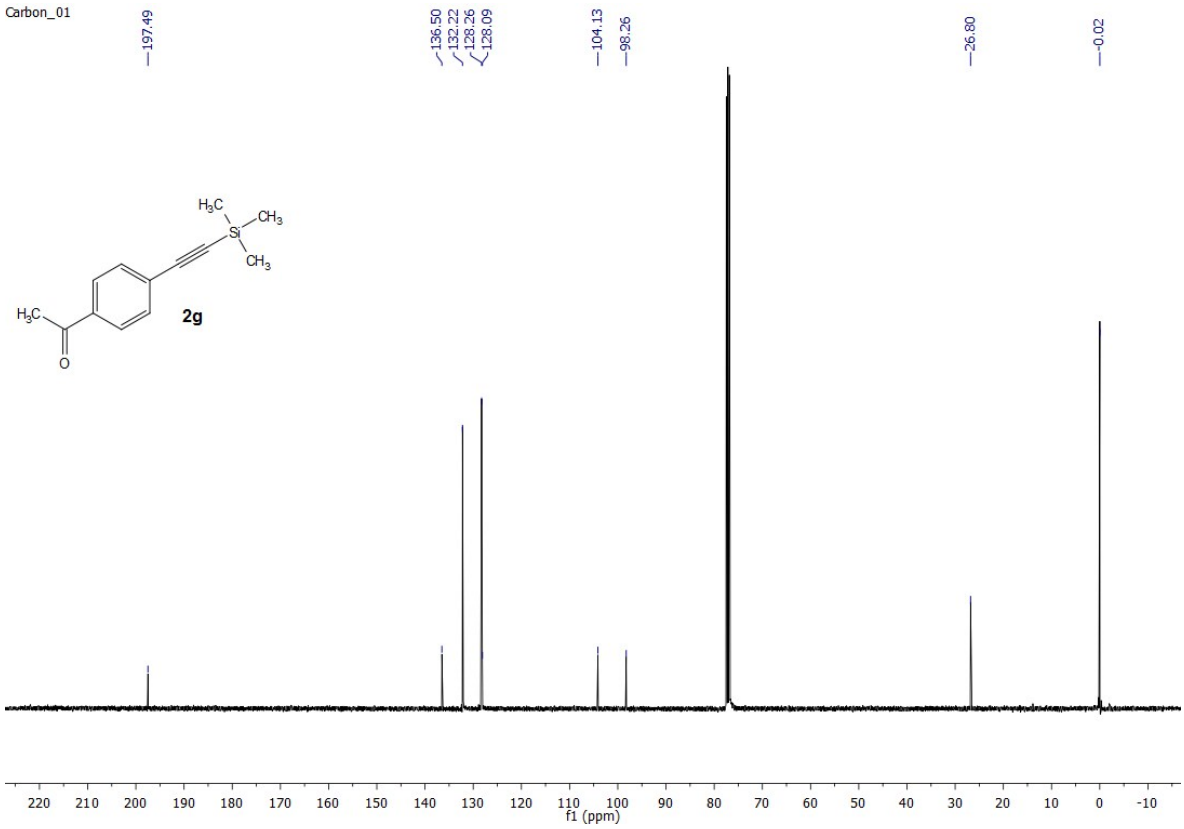

Carbon_01
146.90
133.52
114.67
112.64
106.11
91.51
0.30
2d
f1 (ppm)

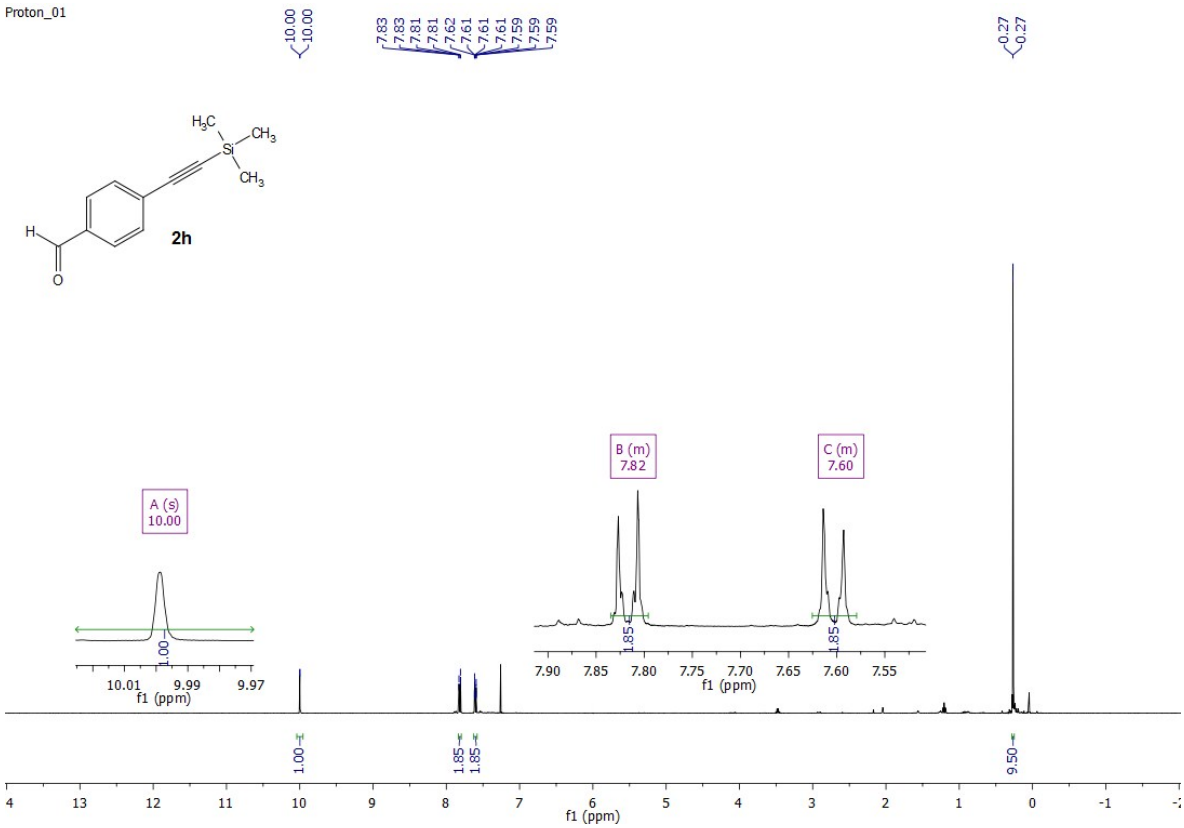

Proton_01
sublimed
scraped
2f
A (s)
7.88
B (s)
7.79
1.91
1.00
H2O
9.16
f1 (ppm)

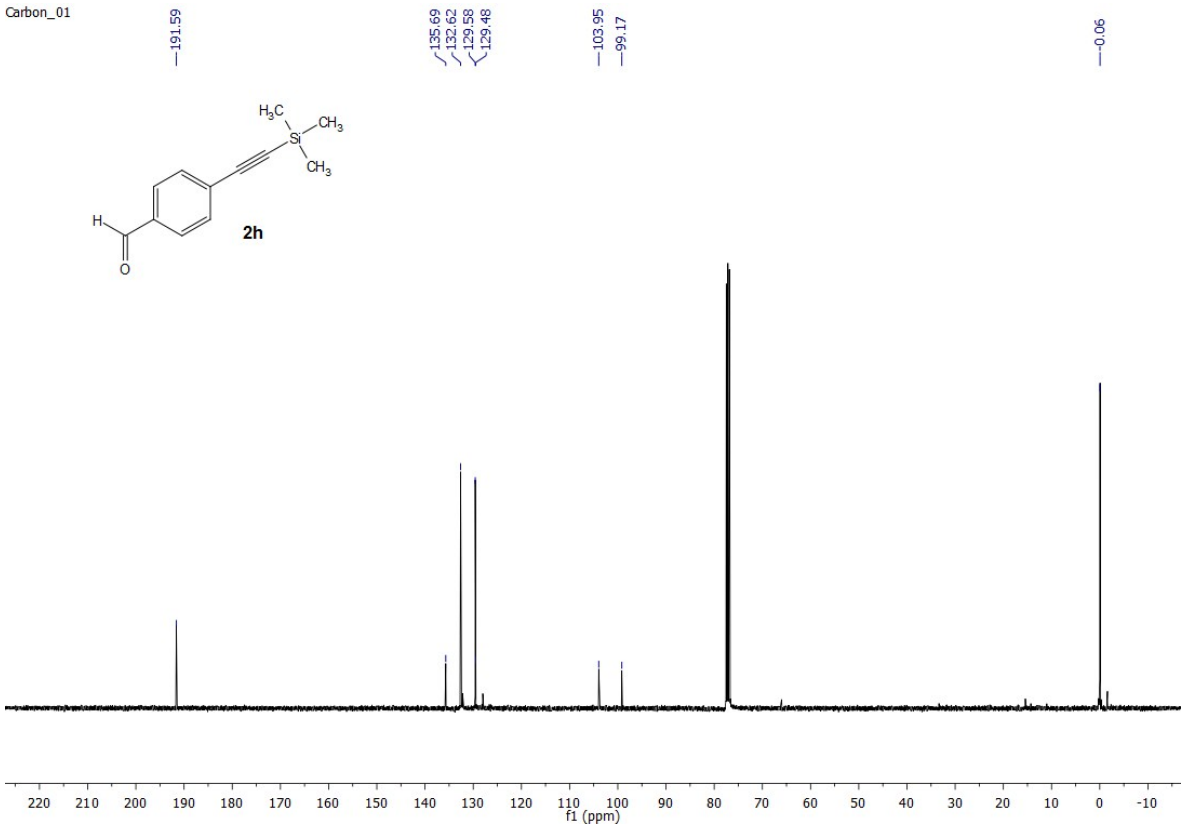

Carbon_01
sublimed
scraped
2f
E (m)
131.65
D (q)
131.63
B (s)
125.26
C (hept)
121.60
A (q)
122.73
f1 (ppm)

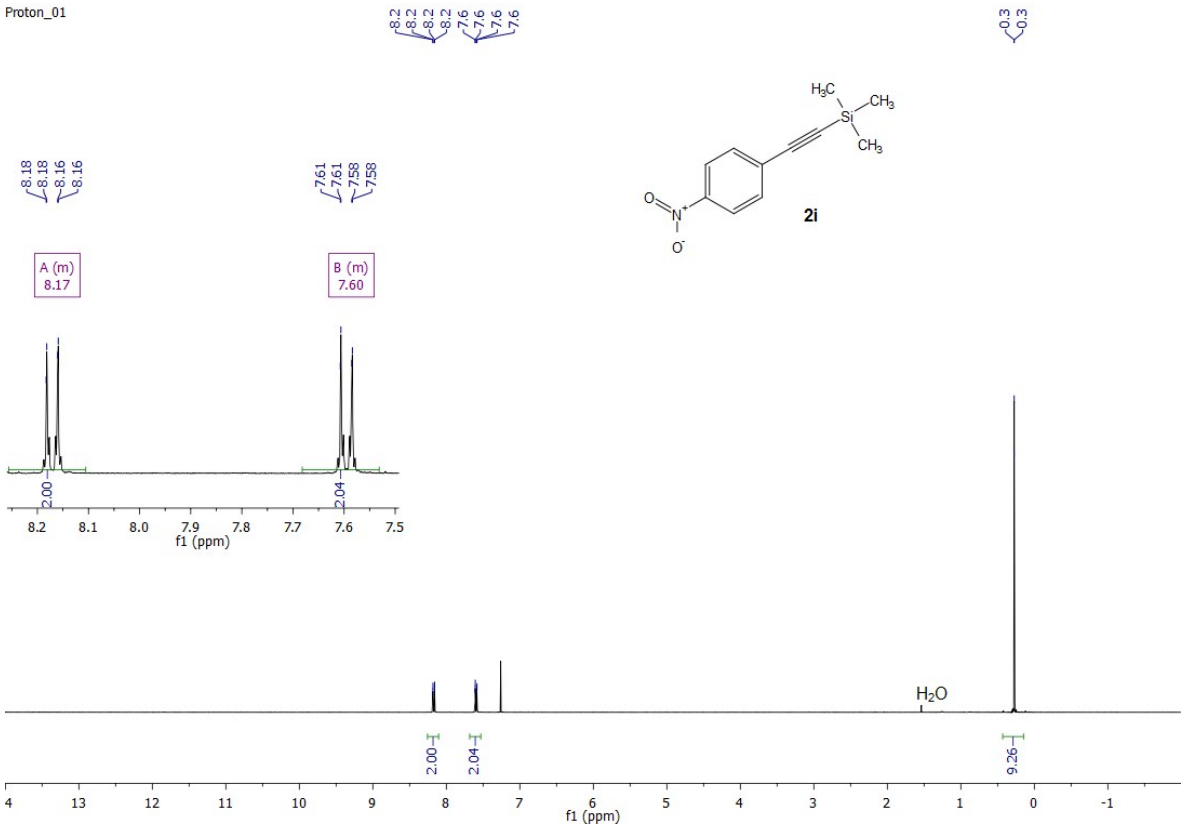

Fluorine_01
sublimed
scraped
A (s)
-63.66
2f
A (s)
-63.66
-63.65
-63.70
f1 (ppm)
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

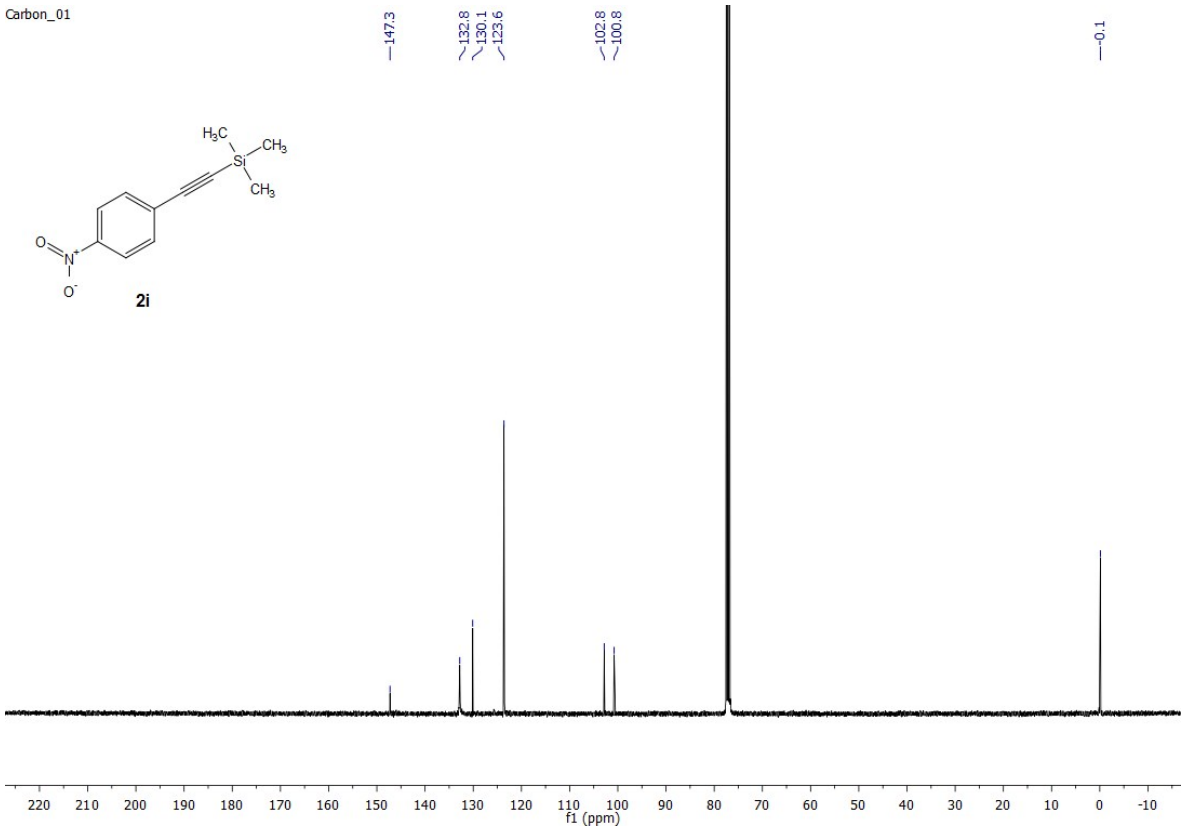

Proton_01
7.90
7.90
7.89
7.88
7.88
7.88
7.55
7.55
7.54
7.53
7.53
7.53
2.60
0.26
2g
A (m)
7.89
B (m)
7.54
2.00
1.93
7.9 7.8 7.7 7.6 7.5 7.4
f1 (ppm)
2.00
1.93
2.91
8.78
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)

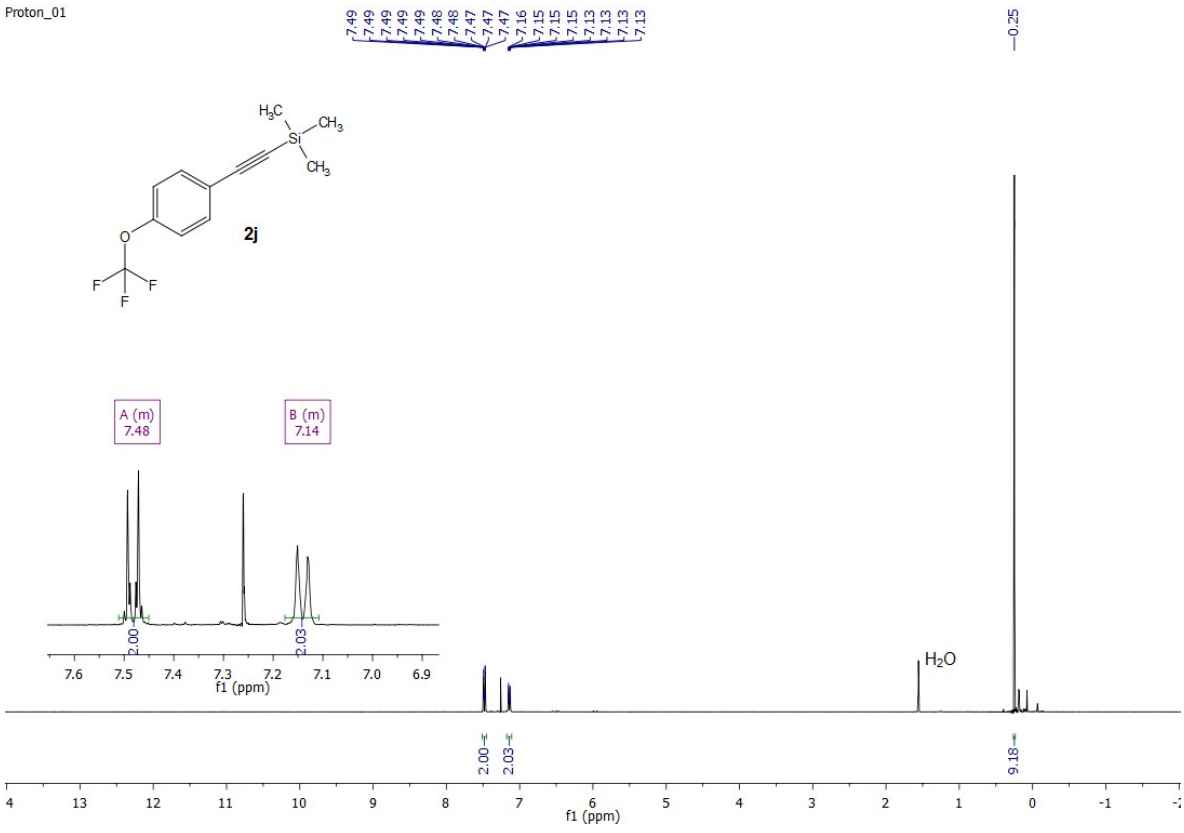

Carbon_01
197.49
136.50
132.22
128.26
128.09
104.13
98.26
26.80
-0.02
2g
f1 (ppm)

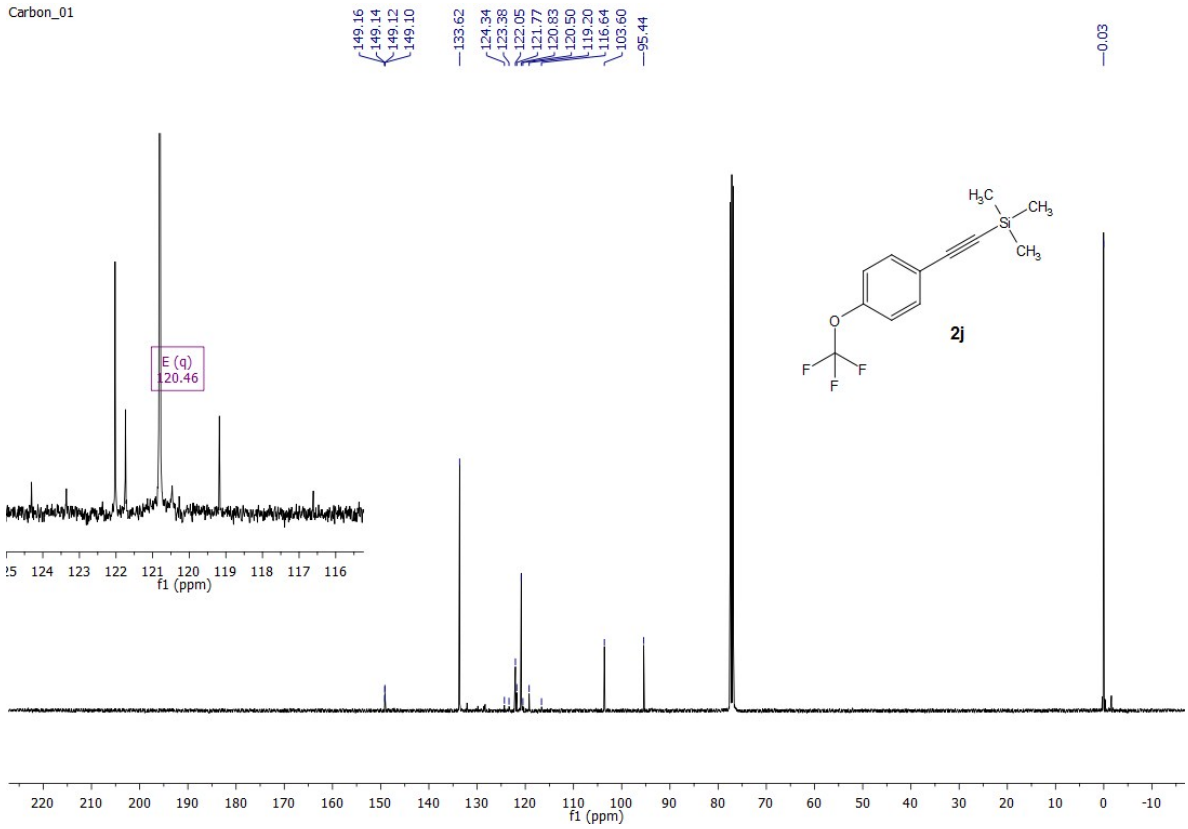

Proton_01
10.00
7.83
7.81
7.62
7.61
7.59
0.27
2h
A (s)
10.00
B (m)
7.82
C (m)
7.60
1.00
1.85
9.50
f1 (ppm)

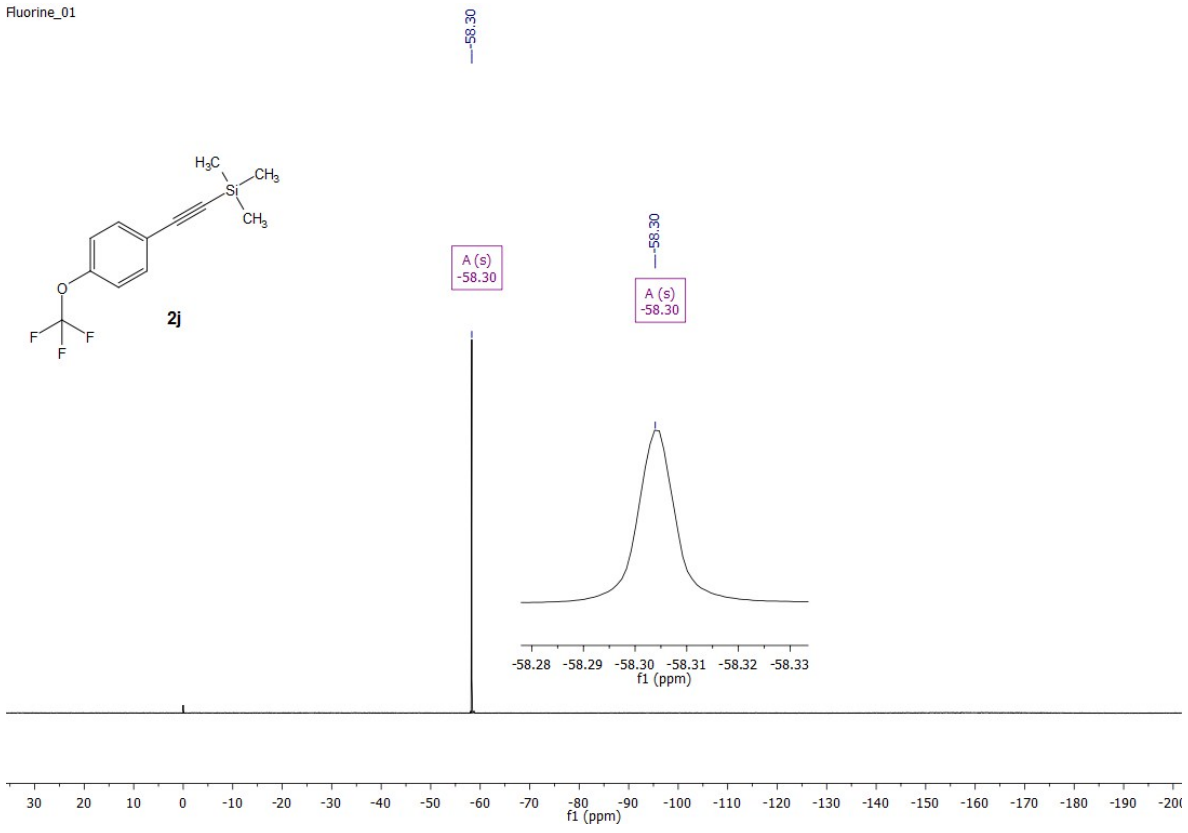

Carbon_01
191.59
135.69
132.62
129.58
129.48
103.95
99.17
0.06
2h
f1 (ppm)

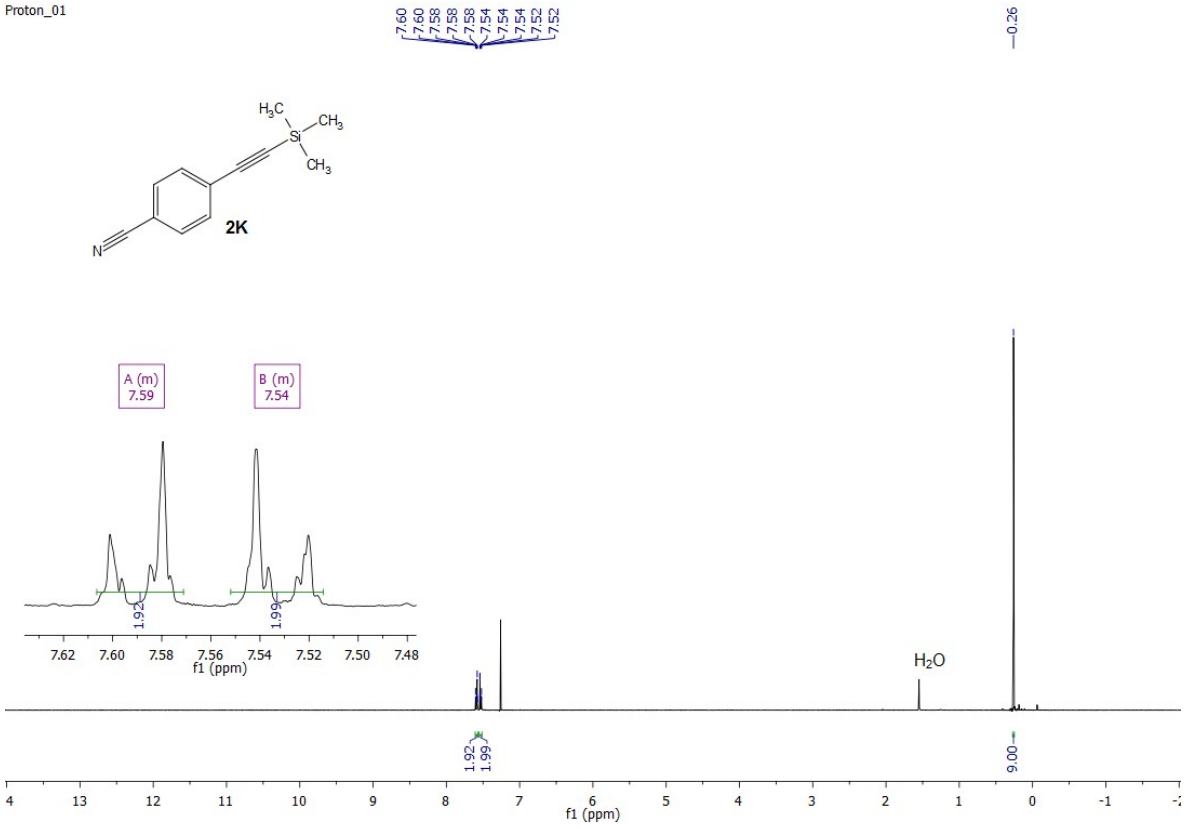

Proton_01
8.2
7.6
0.3
8.18
8.16
7.61
7.58
A (m)
8.17
B (m)
7.60
2.00
2.04
2i
H2O
9.26
f1 (ppm)

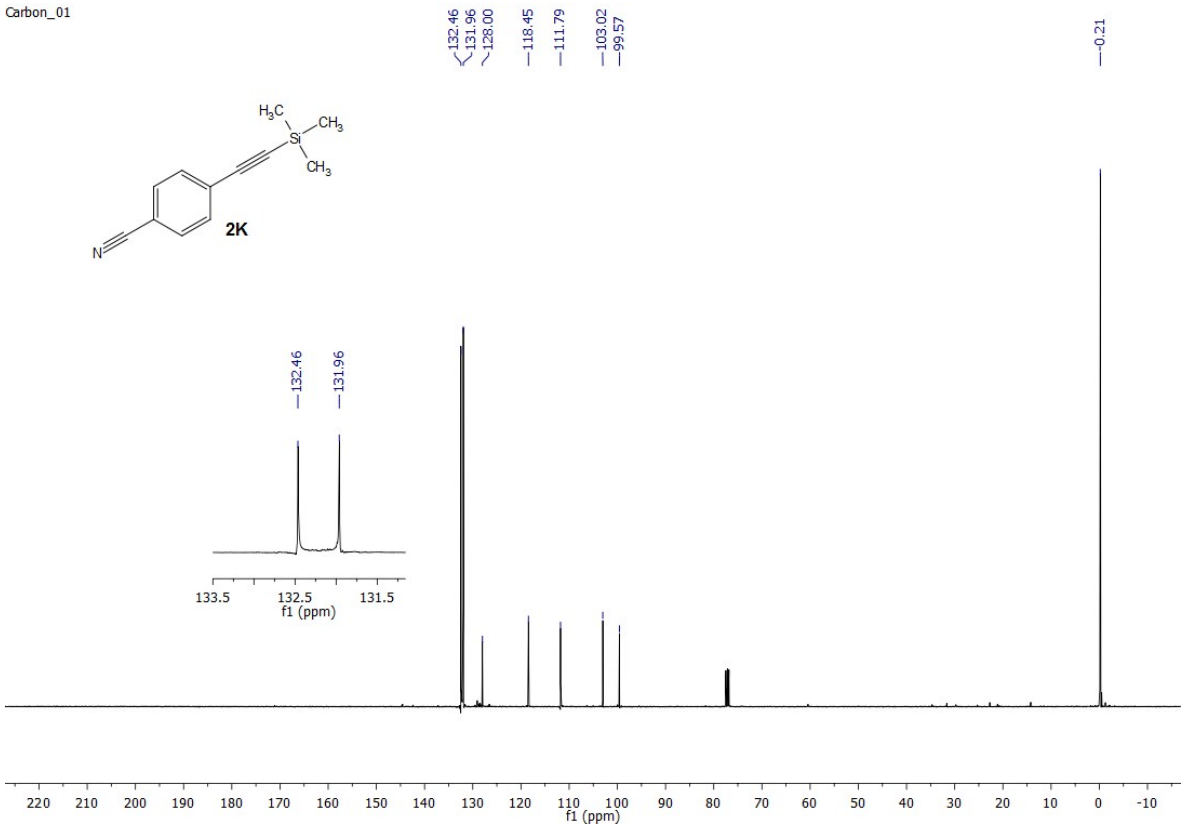

Carbon_01
147.3
132.8
130.1
123.6
102.8
100.8
0.1
2i
f1 (ppm)

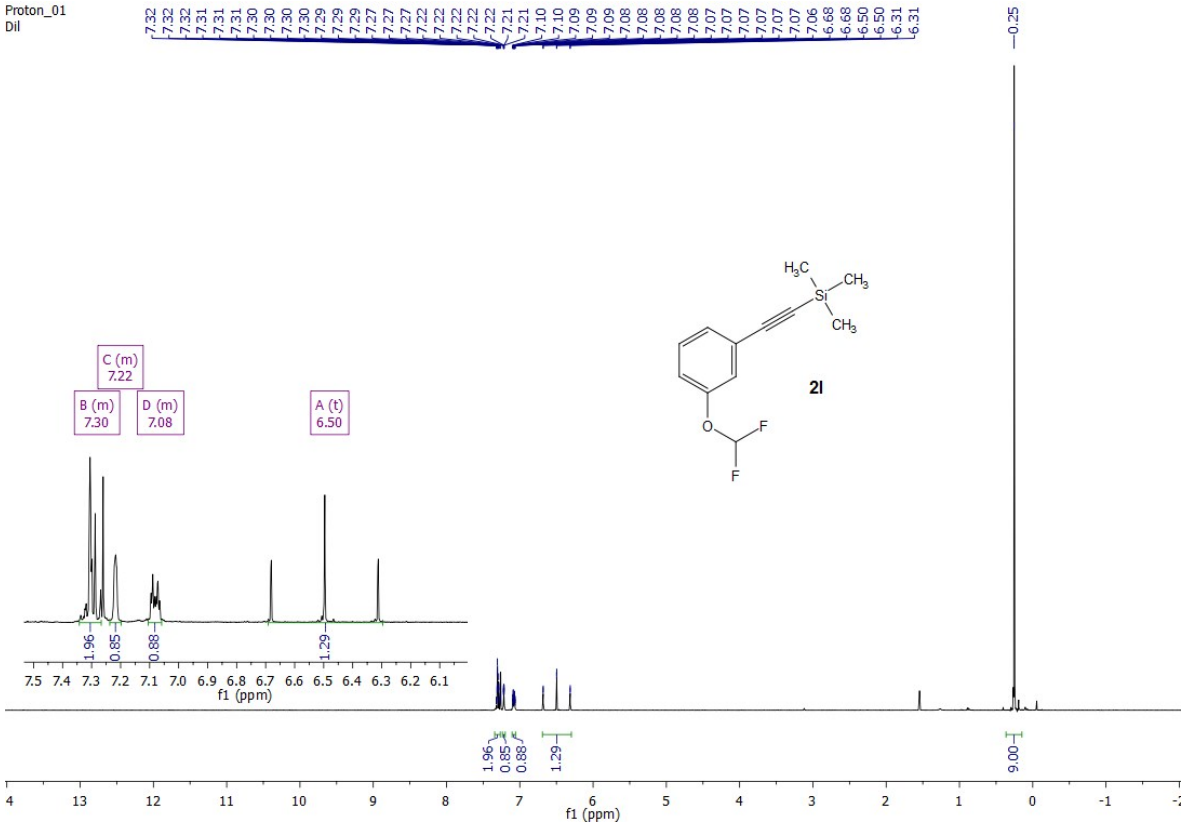

Proton_01
7.49
7.48
7.47
7.16
7.15
7.13
0.25
2j
A (m)
7.48
B (m)
7.14
2.00
2.03
H2O
9.18
f1 (ppm)

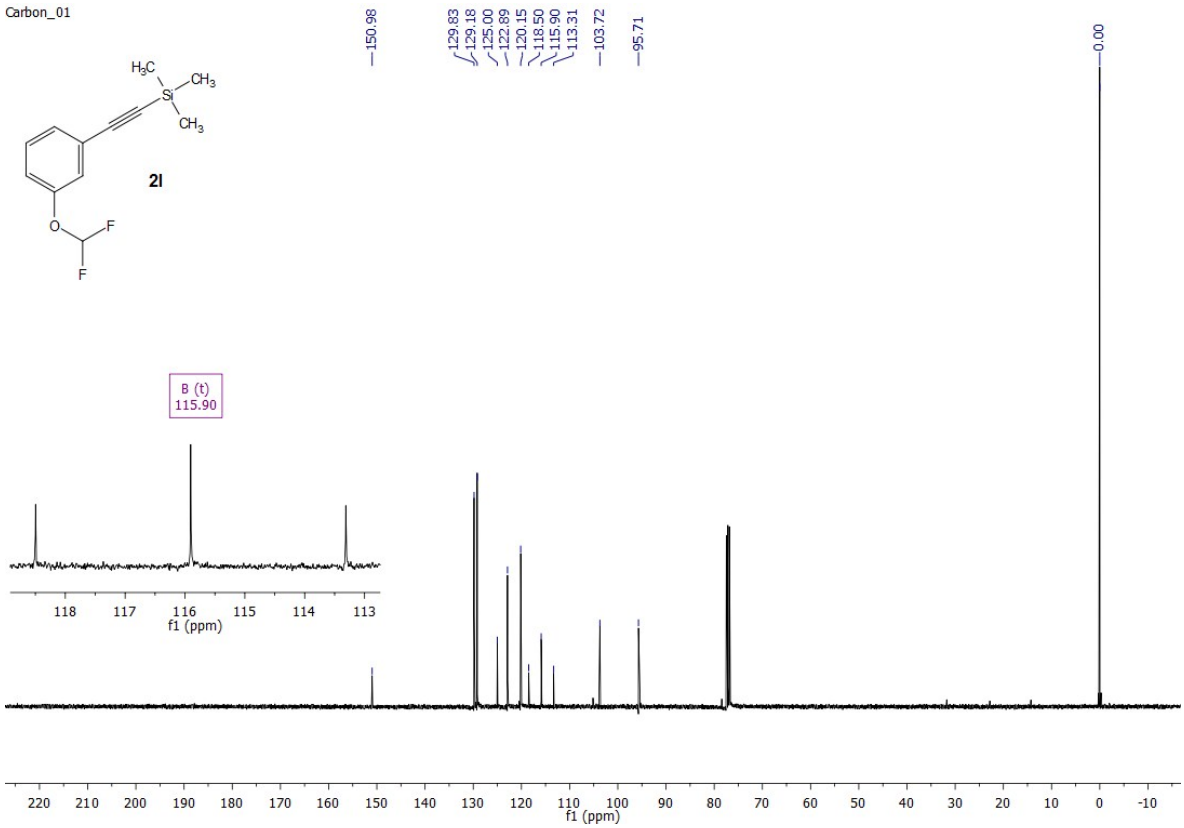

Carbon_01
149.16
149.14
149.12
149.10
133.62
124.34
123.38
122.05
121.77
120.83
120.50
119.20
116.64
103.60
95.44
0.03
E (q)
120.46
2j
f1 (ppm)

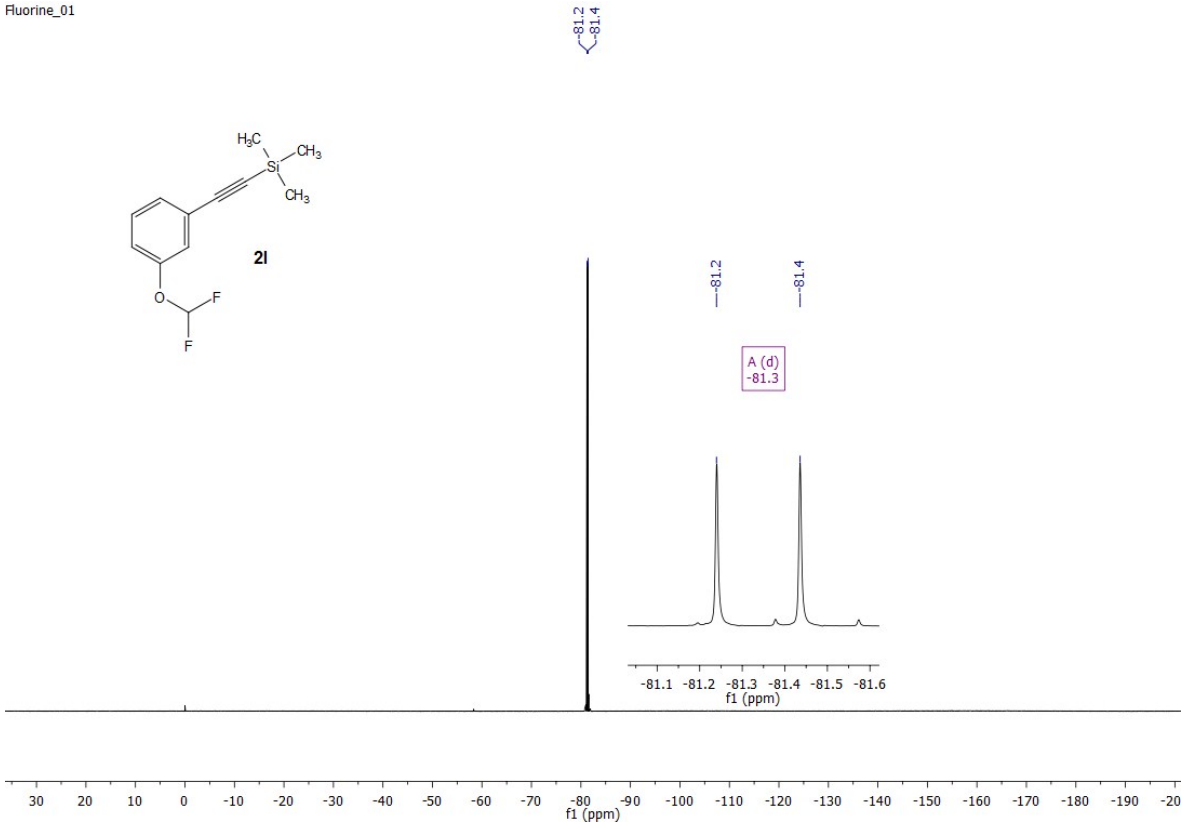

Fluorine_01
-58.30
A (s)
-58.30
2j
f1 (ppm)

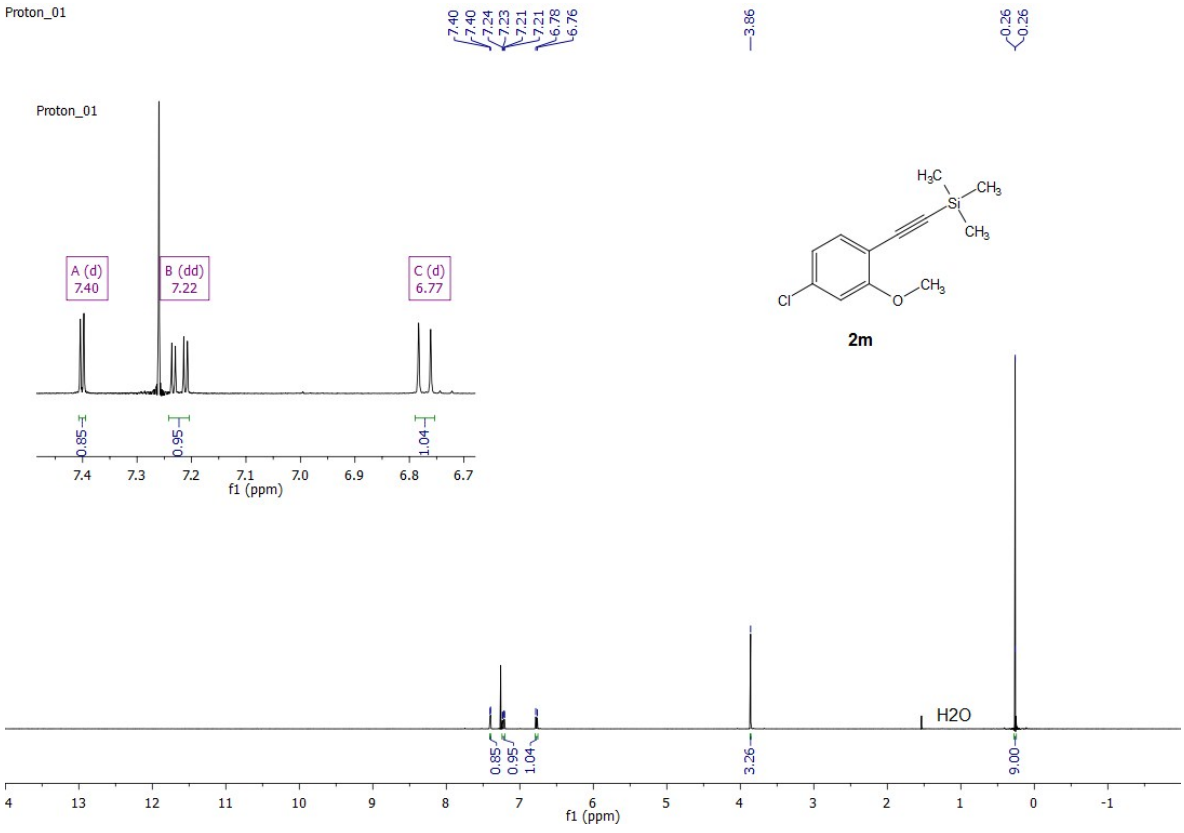

Proton_01
7.60
7.60
7.58
7.58
7.58
7.54
7.54
7.54
7.52
7.52
0.26
2K
A (m)
7.59
B (m)
7.54
1.92
1.99
7.62
7.60
7.58
7.56
7.54
7.52
7.50
7.48
f1 (ppm)
H2O
1.92
1.99
9.00
f1 (ppm)

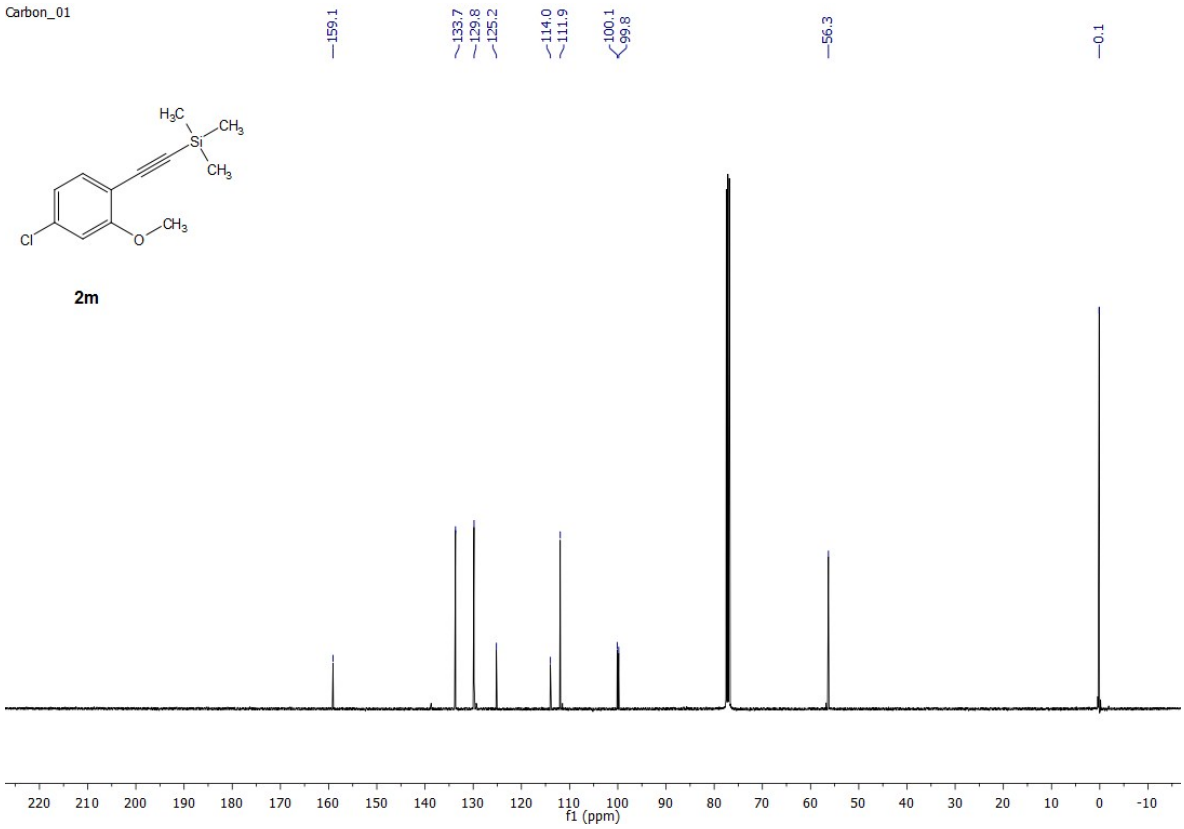

Carbon_01
132.46
131.96
128.00
118.45
111.79
103.02
99.57
0.21
2K
132.46
131.96
133.5
132.5
131.5
f1 (ppm)
f1 (ppm)

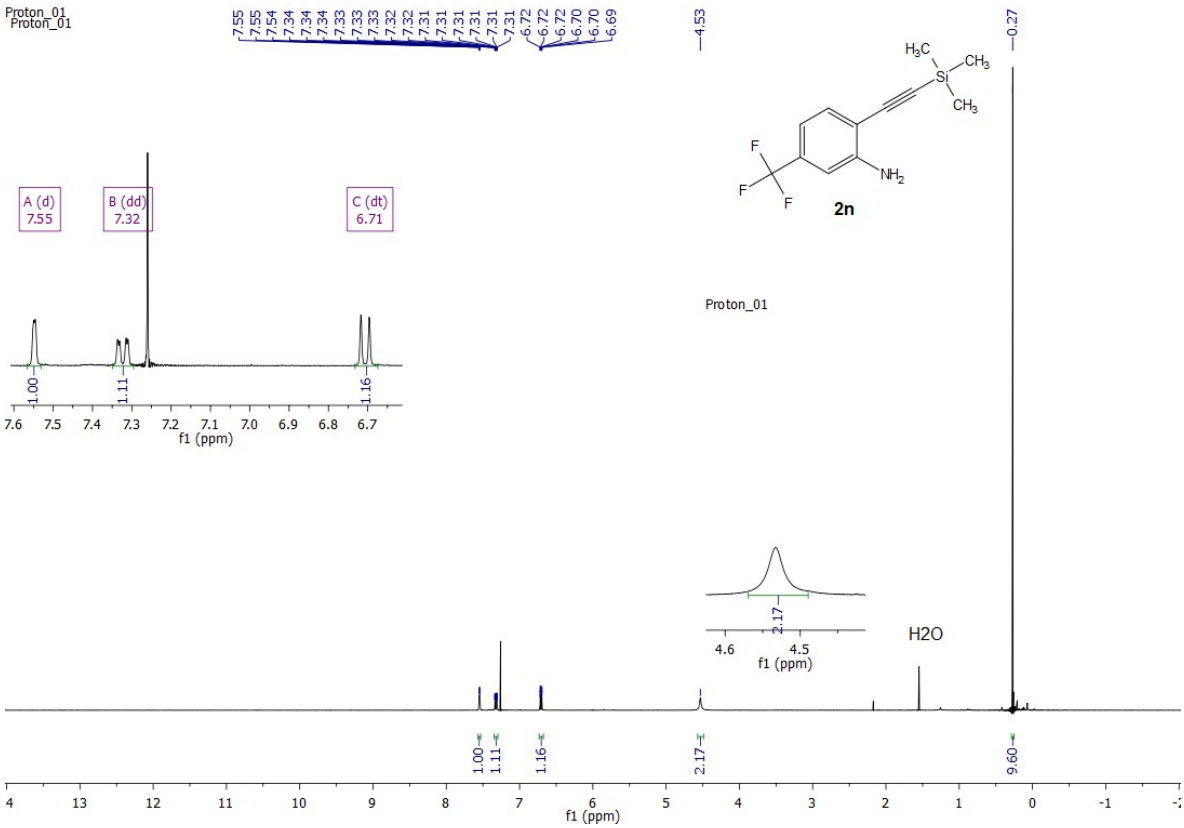

Proton_01
Dil
C (m)
7.22
B (m)
7.30
D (m)
7.08
A (t)
6.50
2l
f1 (ppm)

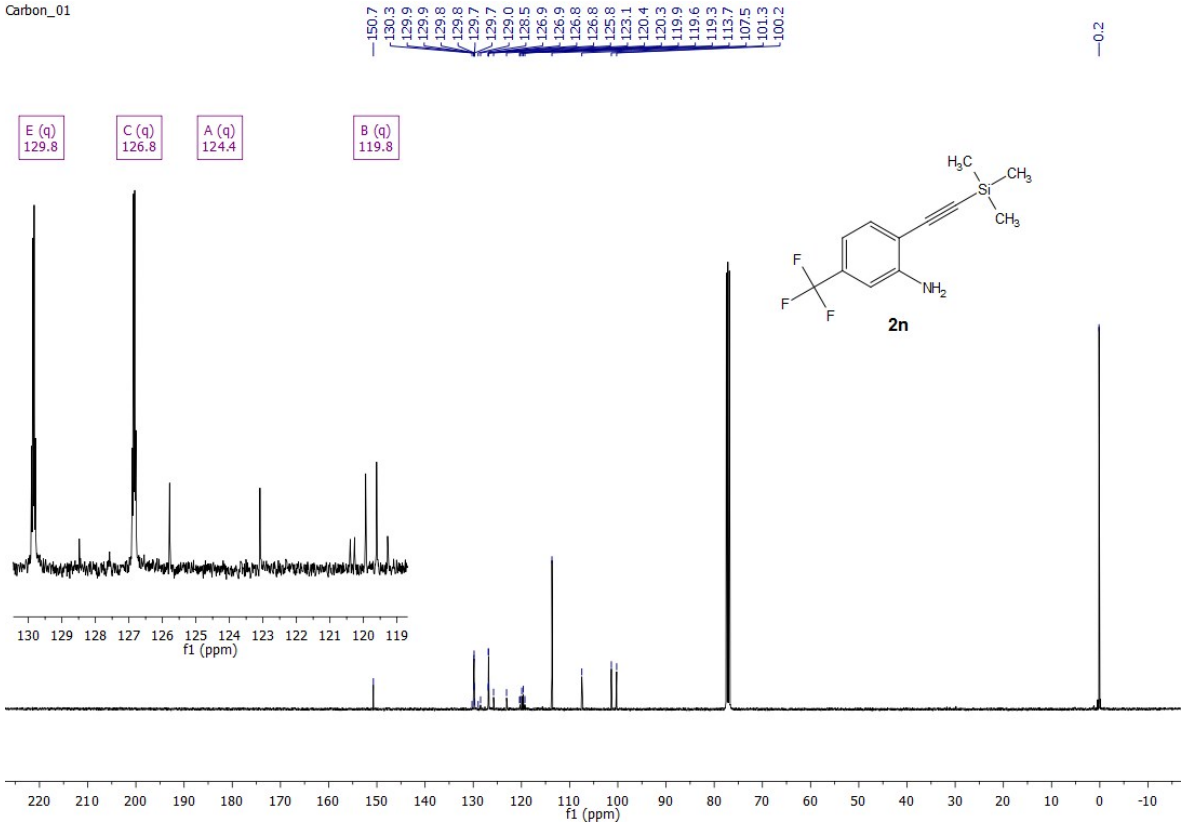

Carbon_01
B (t)
115.90
2l
f1 (ppm)

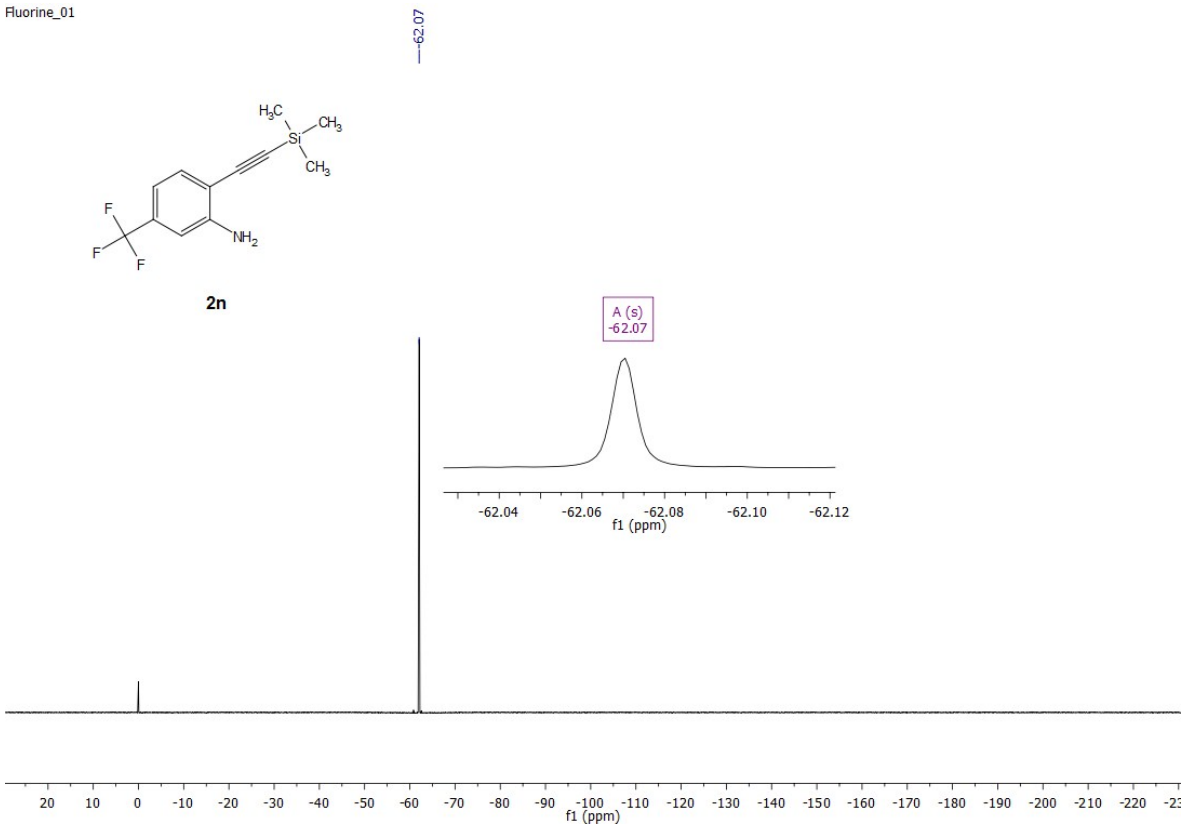

Fluorine_01
-81.2
-81.4
2l
A (d)
-81.3
f1 (ppm)

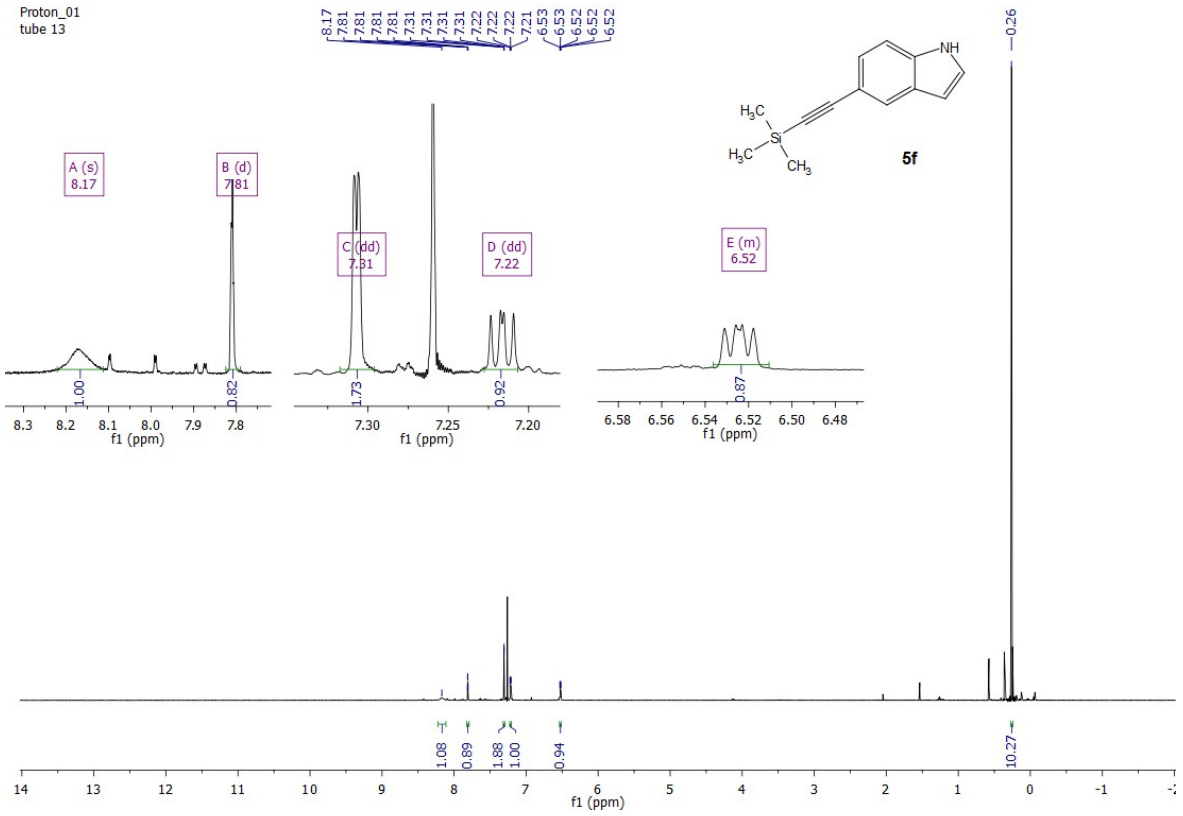

Proton_01
7.40
7.40
7.24
7.23
7.21
7.21
6.78
6.76
3.86
0.26
0.26
A (d)
7.40
B (dd)
7.22
C (d)
6.77
2m
H2O
f1 (ppm)

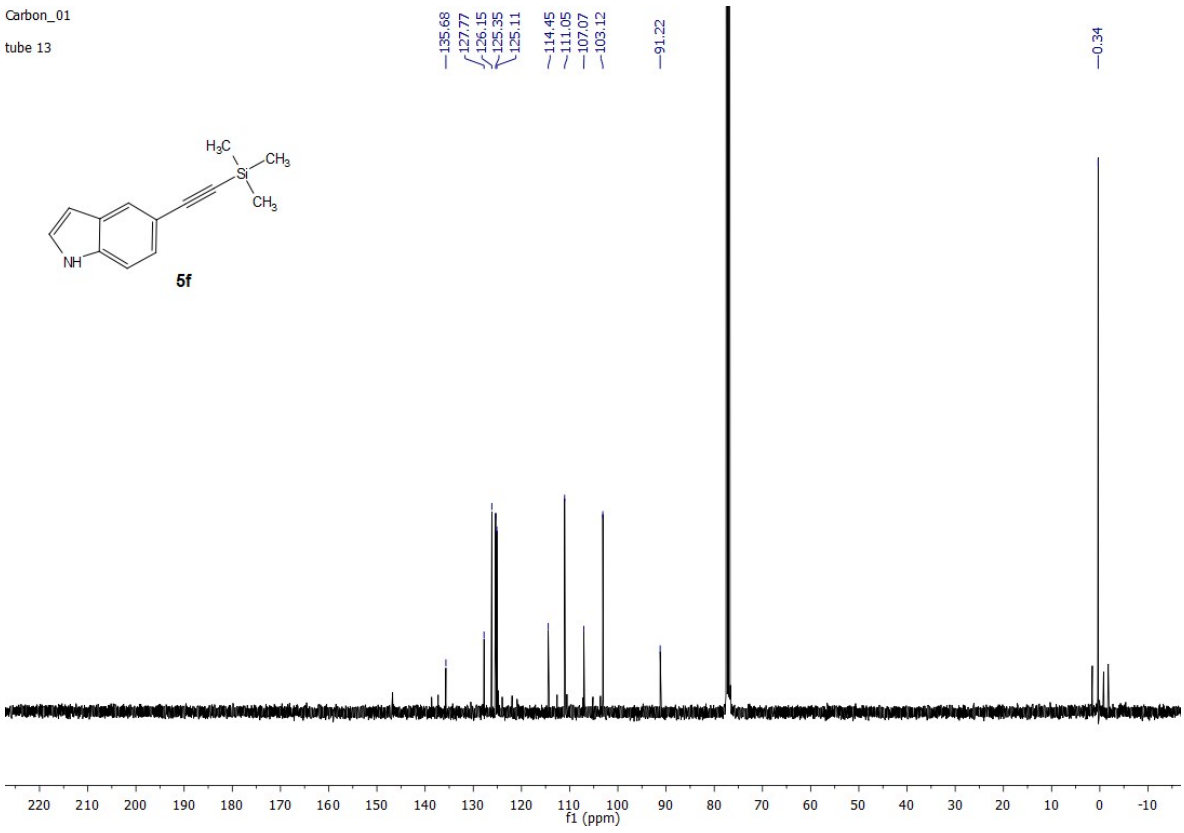

Carbon_01
159.1
133.7
129.8
125.2
114.0
111.9
100.1
99.8
56.3
0.1
2m
f1 (ppm)


Proton_01
Proton_01
7.55
7.54
7.34
7.33
7.32
7.31
6.72
6.70
6.69
4.53
0.27
2n
A (d)
7.55
B (dd)
7.32
C (dt)
6.71
1.00
1.11
1.16
f1 (ppm)
Proton_01
2.17
4.6
4.5
f1 (ppm)
H2O
9.60
f1 (ppm)


Carbon_01
150.7
130.3
129.9
129.9
129.8
129.8
129.7
129.7
129.0
128.5
126.9
126.9
126.8
126.8
125.8
123.1
120.4
120.3
119.9
119.6
119.3
113.7
107.5
101.3
100.2
0.2
E (q)
129.8
C (q)
126.8
A (q)
124.4
B (q)
119.8
130 129 128 127 126 125 124 123 122 121 120 119
f1 (ppm)
H3C
CH3
Si
CH3
F
F
F
NH2
2n
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

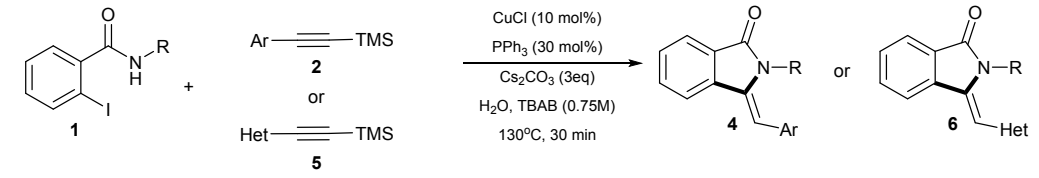

Fluorine_01
-62.07
H3C
CH3
Si
CH3
F
F
F
NH2
2n
A (s)
-62.07
-62.04 -62.06 -62.08 -62.10 -62.12
f1 (ppm)
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)


Proton_01
tube 13
A (s)
8.17
B (d)
7.81
C (dd)
7.31
D (dd)
7.22
E (m)
6.52
5f
f1 (ppm)


Carbon_01
tube 13
5f
f1 (ppm)

Typical procedure for the preparation of isoindolin-1-ones from silylalkynes (Tables 2 & 4)

Scheme 2. Triple tandem desilylation-cross-coupling-heterocyclization of alkynylsilanes **2** with iodobenzamides **1**



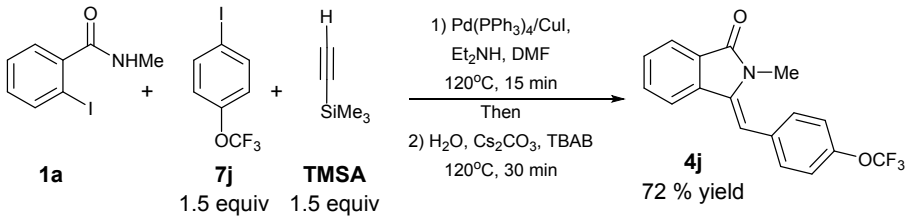
Inside an argon glovebox, the corresponding iodobenzamide **1** (0.5 mmol, 1 equiv), alkynylsilane **2** or heteroaryl alkynylsilane derivative **5** (0.75 mmol, 1.5 equiv), CuCl (5 mg, 0.05 mmol), PPh_3 (39 mg, 0.15 mmol), n-tetrabutylammonium bromide (242 mg, 0.75 mmol) and Cs_2CO_3 (488 mg, 1.50 mmol) were placed in a crimp-top vial and sealed. Liquid derivatives **2** or **5**, they were added using a syringe outside the glovebox. Then, 1 ml of water (degassed) was also added by syringe and the vial was place into a pre-heated oil bath at 130 ^oC for 30-40 min. Subsequently, the reaction mixture was allowed to cool down to room temperature, the vial was opened and the contents were poured into 10 ml of a 0.1 M solution of NH_4OH and extracted with EtOAc (3 times, 15 mL). The organic layers were combined, washed with brine (10 mL), water (5 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash chromatography using the appropriate eluent (usually Hex/EtOAc). The combined product fractions were concentrated on a rotatory evaporator.

Typical procedure for the preparation of isoindolin-1-ones 4 from terminal alkynes 3

The preparation of **4** from terminal alkynes **3** is analogous to the one described above, but using only 2 equiv of Cs_2CO_3 (325.8 mg, 1mmol).

Multicomponent procedure for the preparation of isoindolin-1-ones from activated aryl-iodides, TMSA and 1a

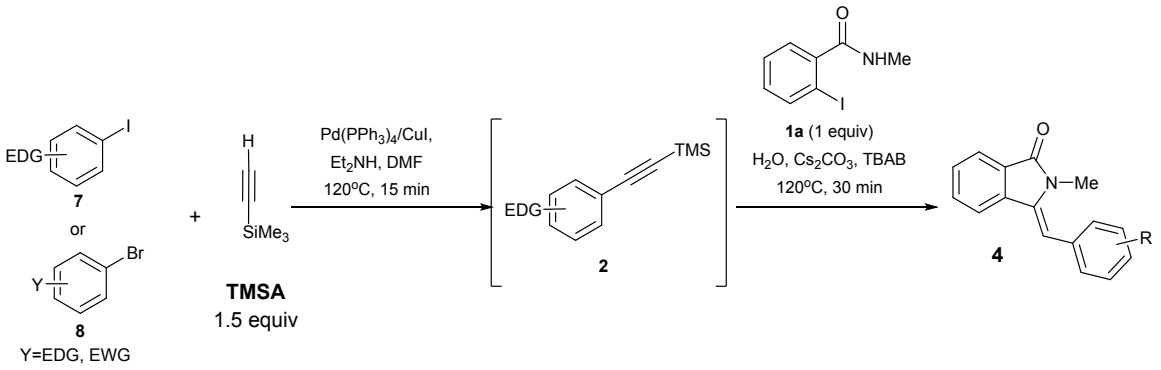
A representative procedure is shown below for the preparation of **4j** from **1a** and 4-(trifluoromethoxy)iodobenzene **7j.**



Inside an argon glovebox, **1a** (0.5 mmol, 1 equiv), $Pd(PPh_3)_4$ (0.015 mmol, 17.32 mg) and CuI (0.05 mmol, 9.5 mg) were placed in a crimp-top vial and sealed (Vial 1). Outside the glovebox, DMF (1 mL), Et_2NH (1

mL), **7j** (0.75 mmol, 216 mg) and TMSA (0.75 mmol, 73.6 mg) were added sequentially by syringe to Vial 1 and this mixture was heated at 120 °C for 15 min. After cooling to room temperature, 1 mL of a solution containing n-tetrabutylammonium bromide (242 mg, 0.75 mmol) and Cs_2CO_3 (488 mg, 1.50 mmol) was added, and this mixture was then heated at 120 °C for 30 min after which, the reaction mixture was then allowed to cool down to room temperature, the vial was opened and the contents were poured into 10ml of a 0.1 M solution of NH_4OH and extracted with EtOAc (3 times, 15 mL). The organic layers were combined, washed with brine (10 mL), water (5 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash chromatography using Hex/EtOAc to afford **4j** in 72% yield.

One-pot procedure for the preparation of isoindolin-1-one from unactivated aryl iodides or bromides, TMSA and 1a.

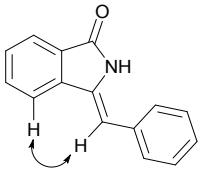


Inside an argon glovebox, $Pd(PPh_3)_4$ (0.015 mmol, 17.32 mg) and CuI (0.05 mmol, 9.5 mg) were placed in a crimp-top vial and sealed (Vial 1). In a second vial, **1a** (0.5 mmol, 1 equiv) n-tetrabutylammonium bromide (242 mg, 0.75 mmol) and Cs_2CO_3 (488 mg, 1.50 mmol) were weighed and sealed (Vial 2). Outside the glovebox, DMF (1 mL), Et_2NH(1 mL), aryl iodide **7** or aryl bromide **8** (0.75 mmol, 1.5 equiv) and TMSA (0.75 mmol, 73.6 mg) were added sequentially by syringe to Vial 1 and this mixture was heated at 120 °C for 15-30 minutes (monitored by GC-MS until complete formation of alkynylsilane **2**) and allowed to cool down to room temperature. Subsequently, H_2O (1 mL) was added to Vial 2 containing **1a**, Cs_2CO_3 and TBAB and this suspension was added to the vial containing alkynylsilane **2** (to ensure full transfer of all reagents, washing this vial with small amounts of DMF (1 mL) is recommended). This mixture was then heated at 120 °C for 30 min after which, the reaction mixture was allowed to cool down to room temperature, the vial was opened and the contents were poured into 10 ml of a 0.1 M solution of NH_4OH and extracted with EtOAc (3 times, 15 mL). The organic layers were combined, washed with brine (10 mL), water (5 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash chromatography using Hex/EtOAc to afford the corresponding products **4.**

NMR spectroscopic data of isoindolin-1-one products

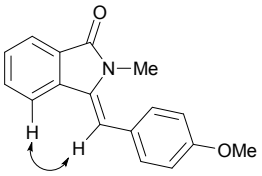
The configuration around the double bond of all products reported herein was confirmed by NOESY experiments (*vide infra*).

(*Z*)-3-Benzylideneisoindolin-1-one (4a)



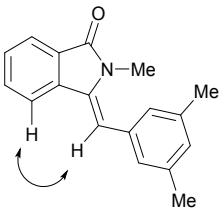
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.89 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.80 (dt, *J* = 7.8, 0.9 Hz, 1H), 7.69 – 7.62 (m, 1H), 7.53 (td, *J* = 7.5, 1.0 Hz, 1H), 7.44 (d, *J* = 4.4 Hz, 4H), 7.36 – 7.29 (m, 1H), 6.57 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.4 , 138.4 , 135.0 , 133.1 , 132.3 , 129.3 , 129.2 , 128.8 , 128.7 , 127.8 , 123.6 , 119.9 , 106.2 . The chemical shift corresponding to the N–H varies significantly with sample concentration. Data reported here corresponds to a concentration of 10 mg/mL. This data corresponds to the previously reported structure[8]

(*Z*)-3-(4-Methoxybenzylidene)-2-methylisoindolin-1-one (4b)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.73 (dt, *J* = 7.8, 0.9 Hz, 1H), 7.58 (td, *J* = 7.5, 1.2 Hz, 1H), 7.48 (td, *J* = 7.5, 0.9 Hz, 1H), 7.31 – 7.24 (m, 2H), 6.95 – 6.91 (m, 2H), 6.74 (s, 1H), 3.85 (s, 3H), 3.07 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.1 , 159.2 , 138.3 , 135.8 , 131.9 , 131.1 , 128.9 , 128.6 , 127.1 , 123.3 , 119.3 , 113.8 , 106.6 , 55.5 , 30.7 . **HRMS (ESI)** Calcd. for $C_{17}H_{16}NO_2$ $(M+H)^+$ = 266.1181, Found = 266.1181.

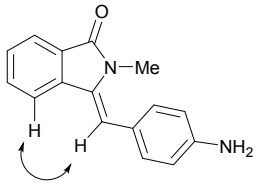
(*Z*)-3-(3,5-Dimethylbenzylidene)-2-methylisoindolin-1-one (4c)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dt, *J* = 7.5, 0.9 Hz, 1H), 7.71 (dt, *J* = 7.7, 0.9 Hz, 1H), 7.57 (td, *J* = 7.7, 1.1 Hz, 1H), 7.47 (td, *J* = 7.4, 0.9 Hz, 1H), 6.97 – 6.94 (m, 3H), 6.73 (s, 1H), 3.05 (s, 3H), 2.34 (s, 3H). **^{13}C**

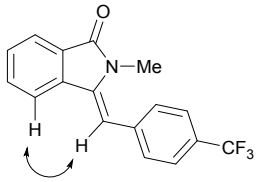
NMR (100 MHz, $CDCl_3$) δ 169.0 , 138.2 , 137.7 , 136.0 , 134.7 , 131.9 , 129.2 , 128.9 , 128.6 , 127.6 , 123.2 , 119.3 , 107.0 , 30.7 , 21.4 . **HRMS (ESI)** Calcd. for $C_{18}H_{18}NO$ $(M+H)^+$ = 264.1388, Found = 264.1389.

(*Z*)-3-(4-Aminobenzylidene)-2-methylisoindolin-1-one (4d)



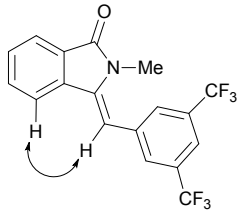
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dt, *J* = 7.5, 0.9 Hz, 1H), 7.72 (dt, *J* = 7.7, 0.9 Hz, 1H), 7.57 (td, *J* = 7.7, 1.2 Hz, 1H), 7.46 (td, *J* = 7.5, 0.9 Hz, 1H), 7.16 – 7.12 (m, 2H), 6.72 – 6.68 (m, 3H), 3.78 (br. s, 2H), 3.12 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.2 , 146.0 , 138.4 , 135.1 , 131.8 , 131.1 , 128.7 , 124.7 , 123.2 , 119.2 , 115.2 , 114.7 , 107.5 , 30.8 . **HRMS (ESI)** Calcd. for $C_{16}H_{15}N_2O$ $(M+H)^+$ = 251.1184, Found = 251.1186.

(*Z*)-2-Methyl-3-(4-(trifluoromethyl)benzylidene)isoindolin-1-one (4e)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 7.6, 1.0 Hz, 1H), 7.75 (dd, *J* = 7.7, 1.0 Hz, 1H), 7.68 – 7.59 (m, 3H), 7.52 (tt, *J* = 7.5, 7.5, 1.1 Hz, 1H), 7.47 (d, *J* = 8.0 Hz, 2H), 6.73 (s, 1H), 3.03 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0 , 138.8 , 137.9 , 137.6 , 132.9 (d, *J* = 218.2 Hz), 130.1 , 129.6 (q, *J* = 32.7 Hz), 129.6 , 128.6 , 125.2 (q, *J* = 3.8, 3.7, 3.7 Hz), 124.2 (q, *J* = 272.0 Hz), 123.5 , 119.5 , 30.8 . **^{19}F NMR** (282 MHz, $CDCl_3$) δ -63.1 . **HRMS (ESI)** Calcd. for $C_{17}H_{13}NOF_3$ $(M+H)^+$ = 304.0949, Found = 304.0948.

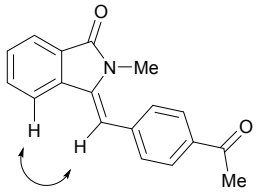
(*Z*)-3-(3,5-Bis(trifluoromethyl)benzylidene)-2-methylisoindolin-1-one (4f)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.83 (s, 1H), 7.81 (s, 2H), 7.74 (dt, *J* = 7.7, 0.8 Hz, 1H), 7.65 (td, *J* = 7.6, 1.2 Hz, 1H), 7.55 (td, *J* = 7.5, 7.4, 0.9 Hz, 1H), 6.69 (s, 1H), 3.01 (d, *J* = 0.6 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0 , 138.9 , 137.7 , 137.3 , 132.5 , 131.7 (q, *J* = 33.7, 33.3, 32.9 Hz),

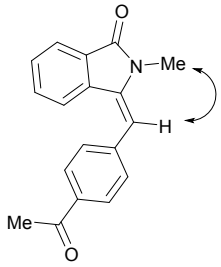
130.0, 129.9 – 129.6 (m), 128.5 , 123.7 , 123.3 (d, J = 272.8 Hz), 121.1 (d, J = 3.8 Hz), 119.6 , 102.1 , 31.0 .**HRMS (ESI)** Calcd. for $C_{18}H_{12}NOF_6$ $(M+H)^+$ = 372.0823, Found = 372.0818.

(*Z*)-3-(4-Acetylbenzylidene)-2-methylisoindolin-1-one (Z-4g)



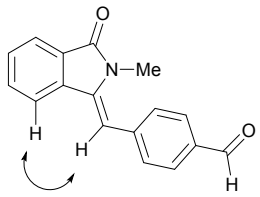
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.95 (m, 2H), 7.87 (dt, J = 7.5, 0.7 Hz, 1H), 7.75 (dt, J = 7.8, 1.2 Hz, 1H), 7.62 (td, J = 7.4, 1.2 Hz, 1H), 7.52 (td, J = 7.4, 1.4 Hz, 1H), 7.46 – 7.43 (m, 2H), 6.74 (s, 1H), 3.04 (s, 3H), 2.64 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 197.6 , 169.1 , 140.1 , 138.0 , 137.6 , 136.0 , 132.3 , 130.1 , 129.6 , 128.5 , 128.2 , 123.5 , 119.5 , 105.0 , 30.9 , 26.8 . **HRMS (ESI)** Calcd. for $C_{18}H_{16}NO_2$ $(M+H)^+$ = 278.1181, Found = 278.1183.

(*E*)-3-(4-Acetylbenzylidene)-2-methylisoindolin-1-one (E-4g)



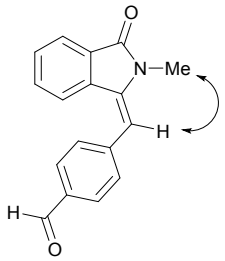
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 2H), 7.85 (dt, J = 7.5, 1.0, 1.0 Hz, 1H), 7.60 – 7.55 (m, 2H), 7.48 – 7.42 (m, 1H), 7.38 – 7.30 (m, 2H), 6.47 (s, 1H), 3.40 (s, 3H), 2.67 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 197.7 , 166.7 , 140.6 , 138.6 , 136.3 , 134.7 , 131.8 , 130.8 , 130.0 , 129.8 , 128.8 , 123.5 , 123.1 , 108.9 , 26.8 , 26.3 . **HRMS (ESI)** Calcd. for $C_{18}H_{16}NO_2$ $(M+H)^+$ = 278.1181, Found = 278.1183.

***(Z)*-4-((2-Methyl-3-oxoisoindolin-1-ylidene)methyl)benzaldehyde (Z-4h)**



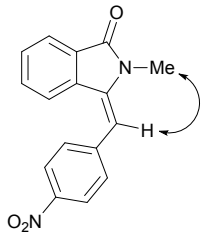
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.04 (s, 1H), 7.93 – 7.89 (m, 2H), 7.86 (dt, J = 7.5, 1.1 Hz, 1H), 7.75 (dt, J = 7.8, 0.9 Hz, 1H), 7.62 (td, J = 7.7, 1.2 Hz, 1H), 7.55 – 7.49 (m, 3H), 6.73 (s, 1H), 3.04 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.7 , 169.1 , 141.6 , 137.9 , 137.9 , 135.3 , 132.3 , 130.5 , 129.7 , 129.6 , 128.5 , 123.5 , 119.6 , 104.7 , 30.9 . **HRMS (ESI)** Calcd. for $C_{17}H_{14}NO_2$ $(M+H)^+$ = 264.1025, Found = 264.1023.

***(E)*-4-((2-Methyl-3-oxoisoindolin-1-ylidene)methyl)benzaldehyde (E-4h)**



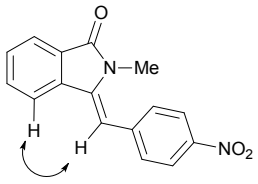
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.07 (s, 1H), 7.97 – 7.93 (m, 2H), 7.85 (dt, *J* = 7.5, 1.0, 1.0 Hz, 1H), 7.65 (dt, *J* = 8.1, 0.7, 0.7 Hz, 2H), 7.47 – 7.42 (m, 1H), 7.38 – 7.30 (m, 3H), 6.46 (s, 1H), 3.39 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.7 , 166.6 , 142.0 , 138.9 , 135.6 , 134.6 , 131.8 , 130.8 , 130.4 , 130.1 , 129.9 , 123.5, 123.0, 108.6 , 26.3 . **HRMS (ESI)** Calcd. for $C_{17}H_{14}NO_2$ $(M+H)^+$ = 264.1025, Found = 264.1023.

***(E)*-2-Methyl-3-(4-nitrobenzylidene)isoindolin-1-one (E-4i)**



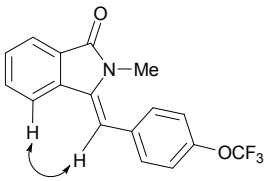
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (d, *J* = 8.8 Hz, 2H), 7.86 (dd, *J* = 7.6, 1.0 Hz, 1H), 7.65 (dd, *J* = 9.0, 0.9 Hz, 2H), 7.50 – 7.45 (m, 1H), 7.39 – 7.31 (m, 2H), 6.43 (s, 1H), 3.40 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.6 , 147.2 , 142.6 , 139.5 , 134.4 , 132.0 , 130.8 , 130.6 , 130.2 , 124.1 , 123.7 , 122.9 , 107.3 , 26.4. **HRMS (ESI)** Calcd. for $C_{16}H_{13}N_2O_3$ $(M+H)^+$ = 281.0926, Found = 281.0925.

***(Z)*-2-Methyl-3-(4-nitrobenzylidene)isoindolin-1-one (Z-4i)**



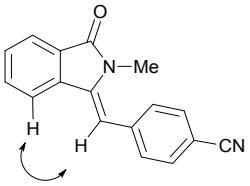
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (d, *J* = 8.7 Hz, 2H), 7.88 (dt, *J* = 7.5, 1.0, 1.0 Hz, 1H), 7.75 (dt, *J* = 7.7, 0.9, 0.9 Hz, 1H), 7.64 (td, *J* = 7.6, 7.5, 1.2 Hz, 1H), 7.55 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.53 – 7.49 (m, 2H), 6.71 (s, 1H), 3.04 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.9 , 146.7 , 142.0 , 138.5 , 137.6 , 132.3 , 130.5 , 129.8, 128.3 , 123.5 , 123.4 , 119.5 , 103.2 , 30.9 . **HRMS (ESI)** Calcd. for $C_{16}H_{13}N_2O_3$ $(M+H)^+$ = 281.0926, Found = 281.0925.

***(Z)*-2-Methyl-3-(4-(trifluoromethoxy)benzylidene)isoindolin-1-one (4j)**



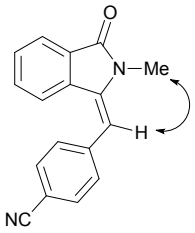
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dt, *J* = 7.6, 1.0 Hz, 1H), 7.73 (dt, *J* = 7.8, 0.9 Hz, 1H), 7.60 (td, *J* = 7.5, 1.2 Hz, 1H), 7.50 (td, *J* = 7.5, 1.0 Hz, 1H), 7.39 – 7.34 (m, 2H), 7.26 – 7.22 (m, 2H), 6.70 (s, 1H), 3.03 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0 , 148.5 , 137.9 , 137.0 , 133.6 , 132.1 , 131.2 , 129.4 , 128.5 , 123.4 , 120.7 , 120.6 (q, *J* = 257.5 Hz), 119.4 , 104.7 , 30.7 . **^{19}F NMR** (376 MHz, $CDCl_3$) δ -58.3. **HRMS (ESI)** Calcd. for $C_{17}H_{13}NO_2F_3$ $(M+H)^+$ = 320.0898, Found = 320.0897.

***(Z)*-4-((2-Methyl-3-oxoisoindolin-1-ylidene)methyl)benzonitrile (Z-4k)**



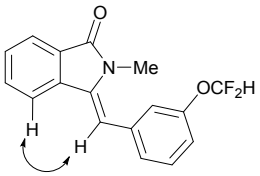
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (dt, *J* = 7.5, 1.1 Hz, 1H), 7.74 (dt, *J* = 7.8, 0.9 Hz, 1H), 7.70 – 7.66 (m, 2H), 7.62 (td, *J* = 7.4, 1.2 Hz, 1H), 7.53 (td, *J* = 7.4, 1.0 Hz, 1H), 7.48 – 7.43 (m, 2H), 6.68 (s, 1H), 3.02 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.1 , 140.1 , 138.2 , 137.8 , 132.4 , 132.0 , 130.5 , 129.8 , 128.5 , 123.6 , 119.6 , 118.8 , 111.1 , 103.8 , 31.0 . **HRMS (ESI)** Calcd. for $C_{17}H_{13}N_2O$ $(M+H)^+$ = 261.1028, Found = 261.1027.

***(E)*-4-((2-Methyl-3-oxoisoindolin-1-ylidene)methyl)benzonitrile (E-4k)**



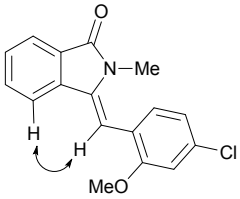
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.77 – 7.69 (m, 2H), 7.61 – 7.56 (m, 2H), 7.47 (td, *J* = 7.5, 1.1 Hz, 1H), 7.36 (td, *J* = 7.4, 1.2 Hz, 1H), 7.29 (dt, *J* = 7.9, 0.9 Hz, 1H), 6.41 (s, 1H), 3.38 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.6 , 140.5 , 139.2 , 134.4 , 132.6 , 131.9 , 130.8 , 130.5 , 130.1 , 123.7 , 122.8 , 118.8 , 111.4 , 107.8 , 26.3 . **HRMS (ESI)** Calcd. for $C_{17}H_{13}N_2O$ $(M+H)^+$ = 261.1028, Found = 261.1027.

***(Z)*-3-(3-(Difluoromethoxy)benzylidene)-2-methylisoindolin-1-one (4l)**



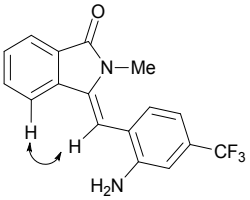
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dt, *J* = 7.8, 0.9 Hz, 1H), 7.73 (dt, *J* = 7.7, 0.8 Hz, 1H), 7.60 (td, *J* = 7.6, 1.2 Hz, 1H), 7.50 (td, *J* = 7.5, 0.9 Hz, 1H), 7.38 (t, *J* = 7.9, 7.9 Hz, 1H), 7.22 – 7.17 (m, 1H), 7.12 (s, 1H), 7.11 – 7.06 (m, 1H), 6.70 (s, 1H), 6.56 (t, *J* = 73.6 Hz, 1H), 3.04 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0 , 150.8 , 137.9 , 137.1 , 136.9 , 132.1 , 129.6 , 129.4 , 128.6 , 127.0 , 123.4 , 121.0 , 119.4 , 118.7 , 115.8 (t, *J* = 261.0 Hz), 104.9 , 30.7 . **^{19}F NMR** (376 MHz, $CDCl_3$) δ -81.4 (d, *J* = 73.6 Hz). **HRMS (ESI)** Calcd. for $C_{17}H_{14}NO_2F_2$ $(M+H)^+$ = 302.0993, Found = 302.0995.

***(Z)*-3-(4-Chloro-2-methoxybenzylidene)-2-methylisoindolin-1-one (4m)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dt, *J* = 7.7, 1.0 Hz, 1H), 7.76 (dt, *J* = 7.6, 0.9 Hz, 1H), 7.59 (td, *J* = 7.6, 1.1 Hz, 1H), 7.49 (td, *J* = 7.5, 0.9 Hz, 1H), 7.27 (dd, *J* = 8.8, 2.5 Hz, 1H), 7.21 (dd, *J* = 2.6, 0.9 Hz, 1H), 6.84 (d, *J* = 8.7 Hz, 1H), 6.58 (s, 1H), 3.85 (s, 3H), 3.06 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0 , 156.2 , 138.0 , 137.5 , 132.0 , 131.2 , 129.2 , 128.8 , 128.6 , 125.4 , 125.2 , 123.3 , 119.7 , 111.6 , 101.2 , 55.9 , 30.2 . **HRMS (ESI)** Calcd. for $C_{17}H_{15}NO_2Cl$ $(M+H)^+$ = 300.0791, Found = 300.0791.

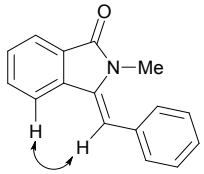
***(Z)*-3-(2-Amino-4-(trifluoromethyl)benzylidene)-2-methylisoindolin-1-one (4n)**



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.77 (dt, *J* = 7.7, 0.9 Hz, 1H), 7.62 (td, *J* = 7.6, 1.2 Hz, 1H), 7.53 (td, *J* = 7.5, 1.0 Hz, 1H), 7.42 – 7.37 (m, 2H), 6.77 (d, *J* = 8.3 Hz, 1H), 6.44 (s, 1H), 4.14 (s, 2H), 3.04 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 168.6 , 147.8 , 138.9 , 137.3 , 132.2 , 129.6 , 128.7 , 128.2 (q, *J* = 3.8 Hz), 126.2 (q, *J* = 3.8 Hz), 124.7 (q, *J* = 270.8 Hz), 123.4 , 119.9 (q, *J* = 33.1 Hz), 119.6 , 119.4 ,

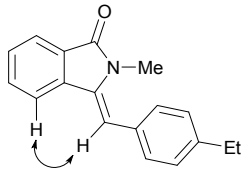
114.4 , 100.2 , 29.2 .**^{19}F NMR** (376 MHz, $CDCl_3$) δ -61.8 . **HRMS (ESI)** Calcd. for $C_{17}H_{14}N_2OF_3$ $(M+H)^+$ = 319.1058, Found = 319.1057.

***(Z)*-3-Benzylidene-2-methylisoindolin-1-one (4o)**



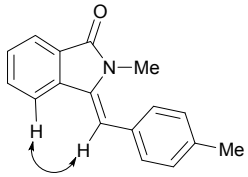
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.75 (d, *J* = 7.7 Hz, 1H), 7.60 (td, *J* = 7.5, 1.2 Hz, 1H), 7.49 (td, *J* = 7.5, 0.9 Hz, 1H), 7.42 – 7.37 (m, 2H), 7.37 – 7.30 (m, 3H), 6.79 (s, 1H), 3.04 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.1 , 138.1 , 136.3 , 134.9 , 132.0 , 129.8 , 129.1 , 128.6 , 128.2 , 127.6 , 123.3 , 119.4 , 106.7 , 30.7. This data corresponds to the previously reported structure [9]

***(Z)*-3-(4-Ethylbenzylidene)-2-methylisoindolin-1-one (4p)**



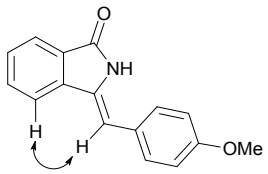
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (dt, *J* = 7.7, 0.9 Hz, 1H), 7.73 (dt, *J* = 7.7, 0.8 Hz, 1H), 7.58 (td, *J* = 7.6, 1.1 Hz, 1H), 7.48 (td, *J* = 7.5, 0.9 Hz, 1H), 7.29 – 7.19 (m, 4H), 6.76 (s, 1H), 3.06 (s, 3H), 2.69 (q, *J* = 7.6 Hz, 2H), 1.27 (t, *J* = 7.6 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.1 , 143.8 , 138.2 , 135.9 , 132.1 , 131.9 , 129.8 , 129.0 , 128.6 , 127.7 , 123.2 , 119.3 , 106.9 , 30.7 , 28.8 , 15.6 . **HRMS (ESI)** Calcd. for $C_{18}H_{18}NO$ $(M+H)^+$ = 264.1388 Found = 264.1398

***(Z)*-2-Methyl-3-(4-methylbenzylidene)isoindolin-1-one (4q)**



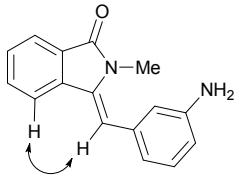
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (dt, *J* = 7.4, 1.0 Hz, 1H), 7.71 (dt, *J* = 7.6, 0.9 Hz, 1H), 7.57 (td, *J* = 7.7, 1.2 Hz, 1H), 7.46 (td, *J* = 7.4, 1.0 Hz, 1H), 7.25 – 7.14 (m, 4H), 6.74 (s, 1H), 3.05 (s, 3H), 2.38 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 168.9 , 138.1 , 137.3 , 135.8 , 133.9 , 133.7 , 131.8 , 129.6 , 128.8 , 128.4 , 123.1 , 119.2 , 106.7 , 30.6 , 21.3 . **HRMS (ESI)** Calcd. for $C_{17}H_{16}NO$ $(M+H)^+$ = 250.1232, Found= 250.1232.

***(Z)*-3-(4-Methoxybenzylidene)isoindolin-1-one (4r)**



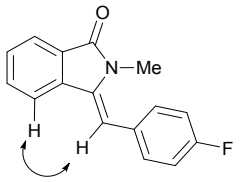
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.88 (dt, *J* = 7.6, 0.9 Hz, 1H), 7.77 (dt, *J* = 7.8, 0.8 Hz, 1H), 7.63 (td, *J* = 7.8, 1.1 Hz, 1H), 7.50 (td, *J* = 7.6, 1.0 Hz, 1H), 7.42 – 7.35 (m, 2H), 7.04 – 6.92 (m, 2H), 6.52 (s, 1H), 3.86 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0 , 159.3 , 138.4 , 132.3 , 131.8 , 130.8 , 129.9 , 129.0 , 127.6 , 123.7 , 119.8 , 114.9 , 106.0 , 55.6 . These values are in agreement to those of the previously reported authentic compound.[9]

***(Z)*-3-(3-Aminobenzylidene)-2-methylisoindolin-1-one (4s)**



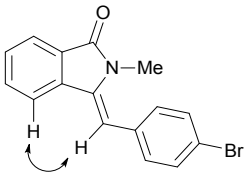
^{1}H NMR (400 MHz, DMSO-D_6) δ 8.03 (d, *J* = 7.8 Hz, 1H), 7.74 (dt, *J* = 7.5, 0.7 Hz, 1H), 7.68 (td, *J* = 7.7, 1.2 Hz, 1H), 7.54 (td, *J* = 7.8, 0.7 Hz, 1H), 7.05 (t, *J* = 7.7 Hz, 1H), 6.94 (s, 1H), 6.60 – 6.57 (m, 1H), 6.56 – 6.52 (m, 2H), 5.16 (s, 2H), 3.01 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 169.0 , 146.2 , 138.2 , 136.1 , 135.9 , 131.9 , 129.2 , 129.0 , 128.6 , 123.2 , 120.3 , 119.3 , 116.1 , 114.4 , 107.0 , 30.5 . **HRMS (ESI)** Calcd. for $C_{16}H_{15}N_2O$ $(M+H)^+$ = 251.1184, Found = 251.1186.

***(Z)*-3-(4-Fluorobenzylidene)-2-methylisoindolin-1-one (4t)**



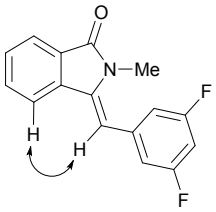
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dt, *J* = 7.6, 0.6 Hz, 1H), 7.72 (dt, *J* = 7.7, 0.8 Hz, 1H), 7.59 (td, *J* = 7.8, 0.6 Hz, 1H), 7.49 (td, *J* = 7.4, 0.8 Hz, 1H), 7.34 – 7.28 (m, 2H), 7.12 – 7.05 (m, 2H), 6.71 (s, 1H), 3.02 (s, 1H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -114.4 – -114.6 (m).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0 , 162.2 (d, *J* = 247.6 Hz), 138.0 , 136.6 , 132.1 , 131.4 (d, *J* = 8.0 Hz), 130.8 (d, *J* = 3.5 Hz), 129.2 , 128.6 , 123.3 , 119.3 , 115.3 (d, *J* = 21.6 Hz), 105.4 , 30.7 . **HRMS (ESI)** Calcd. for $C_{16}H_{13}NOF$ $(M+H)^+$ = 254.0981, Found = 254.0977.

***(Z)*-3-(4-Bromobenzylidene)-2-methylisoindolin-1-one (4u)**



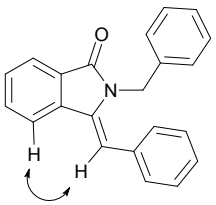
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 7.6, 1.1 Hz, 1H), 7.72 (dd, *J* = 7.7, 0.7 Hz, 1H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.54 – 7.47 (m, 3H), 7.22 (d, *J* = 8.6 Hz, 2H), 6.65 (s, 1H), 3.03 (s, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0 , 138.0 , 136.9 , 133.9 , 132.1 , 131.4 , 129.3 , 128.5 , 123.4 , 121.7 , 119.4 , 105.0 , 30.8 (*ipso* carbon not seen). **HRMS (ESI)** Calcd. for $C_{16}H_{13}NOBr$ $(M+H)^+$ = 314.0181, Found = 314.0176.

***(Z)*-3-(3,5-Difluorobenzylidene)-2-methylisoindolin-1-one (4v)**



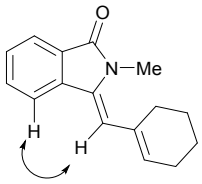
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (dt, *J* = 7.5, 0.9, 1H), 7.71 (dt, *J* = 7.7, 0.9 Hz, 1H), 7.61 (td, *J* = 7.5, 1.2 Hz, 1H), 7.52 (td, *J* = 7.4, 1.0 Hz, 1H), 6.91 – 6.83 (m, 2H), 6.78 (tt, *J* = 9.0, 2.3 Hz, 1H), 6.61 (s, 1H), 3.06 (s, 3H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -110.2 (ddd, *J* = 8.4, 6.4, 1.4 Hz). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0 , 162.7 (dd, *J* = 249.5, 13.2 Hz), 138.3 (t, *J* = 9.9 Hz), 137.8 , 137.7, 132.3 , 129.7 , 128.6 , 123.5 , 119.5 , 113.3 – 112.2 (m), 103.5 (t, *J* = 2.6 Hz), 103.1 (t, *J* = 25.4, Hz), 30.6. **HRMS (ESI)** Calcd. for $C_{16}H_{12}NOF_2$ $(M+H)^+$ = 272.0887, Found = 272.0889.

***(Z)*-2-Benzyl-3-benzylideneisoindolin-1-one (4w)**



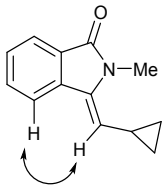
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.75 (dd, *J* = 7.7, 1.0 Hz, 1H), 7.63 (td, *J* = 7.7, 1.1 Hz, 1H), 7.53 (td, *J* = 7.4, 1.0 Hz, 1H), 7.29 – 7.22 (m, 3H), 7.10 – 7.02 (m, 5H), 6.72 (s, 1H), 6.53 (dd, *J* = 7.0, 1.3 Hz, 2H), 4.94 (s, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.2 , 138.6 , 136.9 , 134.7 , 134.5 , 132.2 , 129.8 , 129.2 , 128.2 , 128.1 , 128.0 , 127.5 , 126.8 , 126.5 , 123.7 , 119.6 , 107.7 , 45.0 . These values are in agreement to those of the previously reported authentic compound..[8]

***(Z)*-3-(Cyclohex-1-en-1-ylmethylene)-2-methylisoindolin-1-one (4x)**



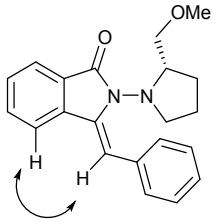
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (dt, *J* = 7.4, 0.9, 0.9 Hz, 1H), 7.61 (dt, *J* = 7.4, 0.9, 0.9 Hz, 1H), 7.52 (td, *J*= 7.6, 7.5, 1.1 Hz, 1H), 7.42 (td, *J* = 7.4, 7.4, 1.0 Hz, 1H), 6.03 (s, 1H), 5.73 – 5.68 (m, 1H), 3.32 (s, 3H), 2.22 – 2.16 (m, 2H), 2.16 – 2.12 (m, 2H), 1.76 – 1.68 (m, 2H), 1.68 – 1.60 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.8 , 138.3 , 134.7 , 132.1 , 131.7 , 129.0 , 128.6 , 128.5 , 123.1 , 119.1 , 110.1 , 30.1 , 29.8 , 25.6 , 22.7 , 22.1 . **HRMS (ESI)** Calcd. for $C_{16}H_{18}NO$ $(M+H)^+$ = 240.1388, Found = 240.1385.

***(Z)*-3-(Cyclopropylmethylene)-2-methylisoindolin-1-one (4y)**



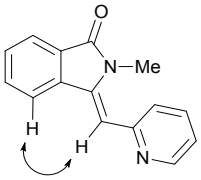
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.55 – 7.46 (m, 2H), 7.40 (td, *J* = 7.4, 1.5 Hz, 1H), 5.08 (d, *J* = 9.9 Hz, 1H), 3.63 (s, 3H), 2.12 (dtt, *J* = 9.7, 8.0, 4.7 Hz, 1H), 1.04 – 0.98 (m, 2H), 0.65 – 0.60 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.8 , 137.5 , 134.9 , 131.5 , 128.1 , 128.0 , 123.1 , 118.5 , 113.6 , 29.5 , 9.6 , 9.2 . **HRMS (ESI)** Calcd. for $C_{13}H_{14}NO$ $(M+H)^+$ = 200.1075, Found = 200.1078.

(S, *Z*)-3-Benzylidene-2-(2-(methoxymethyl)pyrrolidin-1-yl)isoindolin-1-one, 4z



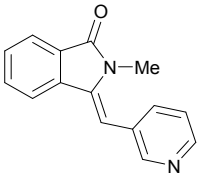
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.05 (dd, *J* = 7.7, 1.0 Hz, 1H), 7.75 – 7.67 (m, 2H), 7.56 (td, *J* = 7.5, 1.1 Hz, 1H), 7.49 – 7.44 (m, 2H), 7.34 (dd, *J* = 7.5, 1.6 Hz, 2H), 7.30 – 7.23 (m, 1H), 6.95 (s, 1H), 3.71 (p, *J* = 6.8 Hz, 1H), 3.59 (q, *J* = 8.0 Hz, 1H), 3.27 – 3.20 (m, 1H), 2.90 (s, 3H), 2.82 – 2.76 (m, 1H), 2.58 – 2.52 (m, 1H), 1.96 – 1.85 (m, 1H), 1.85 – 1.77 (m, 1H), 1.52 – 1.40 (m, 1H), 1.15 – 1.04 (m, 1H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 166.2 , 136.6 , 135.4 , 134.3 , 132.9 , 130.3 , 129.5 , 127.4 , 127.2 , 127.1 , 122.6 , 120.6 , 108.3 , 75.2 , 60.0 , 58.4 , 51.4 , 27.7 , 22.2 . **HRMS (ESI)** Calcd. for $C_{21}H_{23}N_2O_2$ $(M+H)^+$ = 335.1760, Found = 335.1754.

***(Z)*-2-Methyl-3-(pyridin-2-ylmethylene)isoindolin-1-one (5a)**



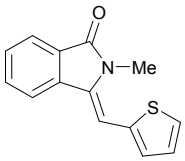
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, *J* = 4.8 Hz, 0H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.76 (d, *J* = 8.5 Hz, 1H), 7.71 (td, *J* = 7.7, 1.8 Hz, 1H), 7.60 (td, *J* = 7.7, 0.6 Hz, 1H), 7.51 (d, *J* = 7.4 Hz, 1H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.23 – 7.16 (m, 1H), 6.72 (s, 1H), 3.23 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.1 , 154.2 , 149.4 , 138.4 , 138.3 , 136.1 , 132.1 , 129.6 , 128.7 , 125.3 , 123.4 , 121.9 , 119.6 , 105.2 , 31.0 **HRMS (ESI)** Calcd. for $C_{15}H_{13}N_2O$ $(M+H)^+$ = 237.1028, Found = 237.1029.

***(Z)*-2-Methyl-3-(pyridin-3-ylmethylene)isoindolin-1-one (5b)**



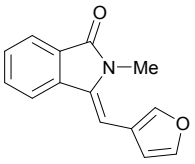
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (s, 1H), 8.57 (s, 1H), 7.86 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.75 (dt, *J* = 7.6, 0.8 Hz, 1H), 7.67 (d, *J* = 7.7 Hz, 1H), 7.62 (td, *J* = 7.7, 1.2 Hz, 1H), 7.52 (td, *J* = 7.5, 1.0 Hz, 1H), 7.34 (dd, *J* = 7.8, 4.7 Hz, 1H), 6.66 (s, 1H), 3.04 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.9 , 150.5 , 148.6 , 138.0 , 137.8 , 136.8 , 132.3 , 131.1 , 129.6 , 128.6 , 123.5 , 123.1 , 119.5 , 101.9 , 30.8 .**HRMS (ESI)** Calcd. for $C_{15}H_{13}N_2O$ $(M+H)^+$ = 237.1028, Found = 237.1028.

***(Z)*-2-Methyl-3-(thiophen-2-ylmethylene)isoindolin-1-one (5c)**



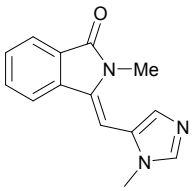
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.8 (dt, *J* = 7.5, 1.1 Hz, 1H), 7.7 (dt, *J* = 7.8, 0.9 Hz, 1H), 7.6 (td, *J* = 7.4, 1.1 Hz, 1H), 7.5 (td, *J* = 7.5, 1.0 Hz, 1H), 7.4 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.1 – 7.0 (m, 1H), 7.0 (dt, *J* = 3.5, 1.3 Hz, 1H), 6.7 (s, 1H), 3.2 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.8 , 137.8 , 137.3 , 136.4 , 132.1 , 129.3 , 128.9, 128.4 , 127.3 , 126.6 , 123.4 , 119.4 , 98.6 , 30.2 . **HRMS (ESI)** Calcd. for $C_{14}H_{12}NOS$ $(M+H)^+$ = 242.0640, Found = 242.0639.

***(Z)*-3-(Furan-3-ylmethylene)-2-methylisoindolin-1-one (5d)**



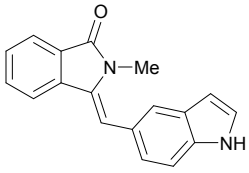
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (dt, *J* = 7.8, 1.2 Hz, 1H), 7.69 (dt, *J* = 7.8, 0.9 Hz, 1H), 7.57 (td, *J* = 7.8, 1.2 Hz, 1H), 7.50 – 7.44 (m, 3H), 6.46 (dd, *J* = 1.3, 0.7 Hz, 1H), 6.41 (s, 1H), 3.25 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.7 , 143.2 , 138.0 , 136.8 , 131.9 , 129.0 , 128.4 , 123.3 , 119.2 , 119.0 , 112.5 , 96.6 , 30.1. **HRMS (ESI)** Calcd. for $C_{14}H_{12}NO_2$ $(M+H)^+$ = 226.0868, Found = 226.0867.

***(Z)*-2-Methyl-3-((1-methyl-1H-imidazol-5-yl)methylene)isoindolin-1-one (5e)**

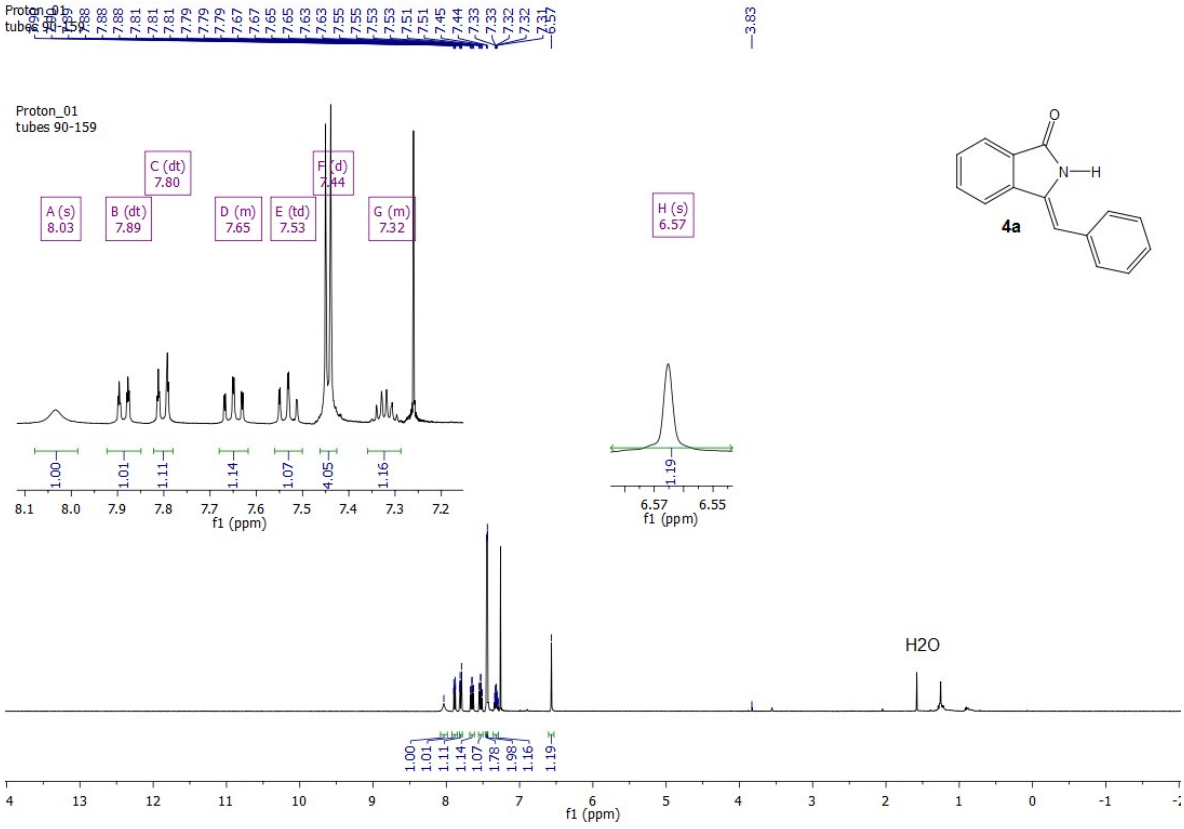


^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (dt, *J* = 7.3, 1.1, 1.1 Hz, 1H), 7.69 (dt, *J* = 7.7, 0.9, 0.9 Hz, 1H), 7.54 (td, *J* = 7.7, 7.5, 1.2 Hz, 1H), 7.50 (s, 1H), 7.44 (td, *J* = 7.5, 7.4, 1.0 Hz, 1H), 7.02 (s, 1H), 6.21 (s, 1H), 3.61 (s, 3H), 3.11 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.5 , 138.6 , 137.3 , 132.0 , 130.9 , 129.5 , 128.3 , 125.8 , 123.3 , 122.6 , 119.3 , 91.4 , 32.0 , 29.5. **HRMS (ESI)** Calcd. for $C_{14}H_{14}N_3O$ $(M+H)^+$ = 240.1137, Found = 240.1141.

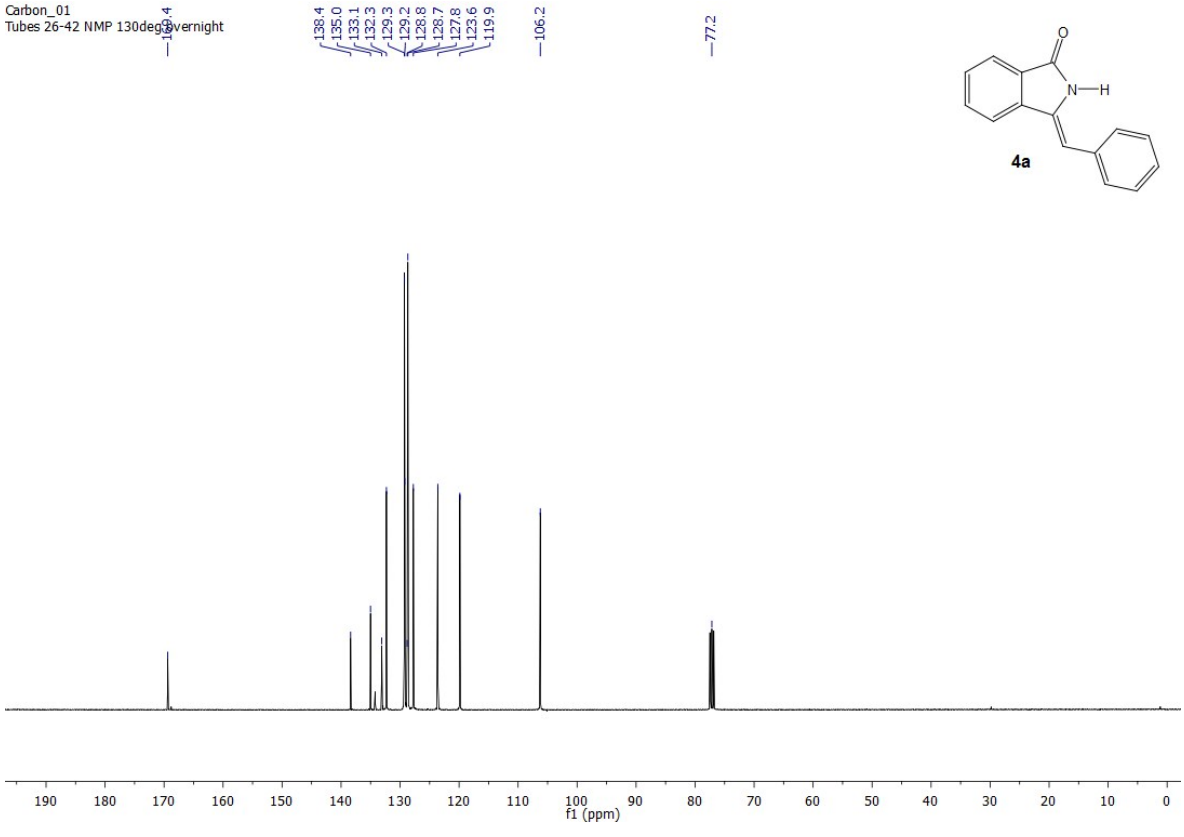
***(Z)*-3-((1H-Indol-5-yl)methylene)-2-methylisoindolin-1-one (5f)**



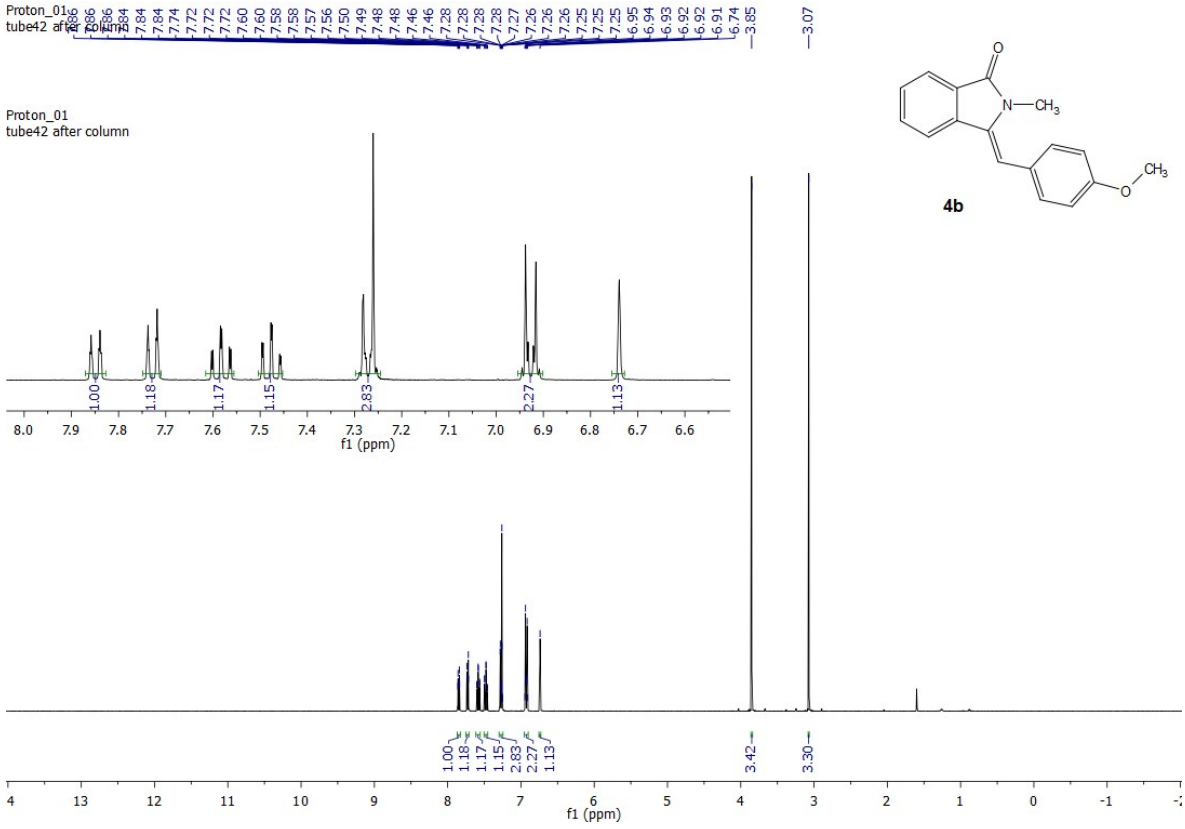
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (s, 1H), 7.86 (dd, *J* = 7.6, 0.9 Hz, 1H), 7.77 (dd, *J* = 7.8, 0.9 Hz, 1H), 7.62 – 7.56 (m, 2H), 7.47 (tt, *J* = 7.5, 7.5, 0.9, 0.9 Hz, 1H), 7.41 (dd, *J* = 8.3, 0.9 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.19 – 7.14 (m, 1H), 6.97 (d, *J* = 0.9 Hz, 1H), 6.57 (td, *J* = 2.1, 2.1, 1.0 Hz, 1H), 3.07 (d, *J* = 0.7 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.3 , 138.4 , 135.3 , 135.3 , 131.8 , 128.7 , 128.5 , 127.8 , 126.2 , 125.3 , 124.1 , 123.2 , 122.1 , 119.3 , 110.9 , 108.8 , 102.9 , 30.8 . **HRMS (ESI)** Calcd. for $C_{18}H_{15}N_2O$ $(M+H)^+$ = 275.1184, Found = 275.1186.

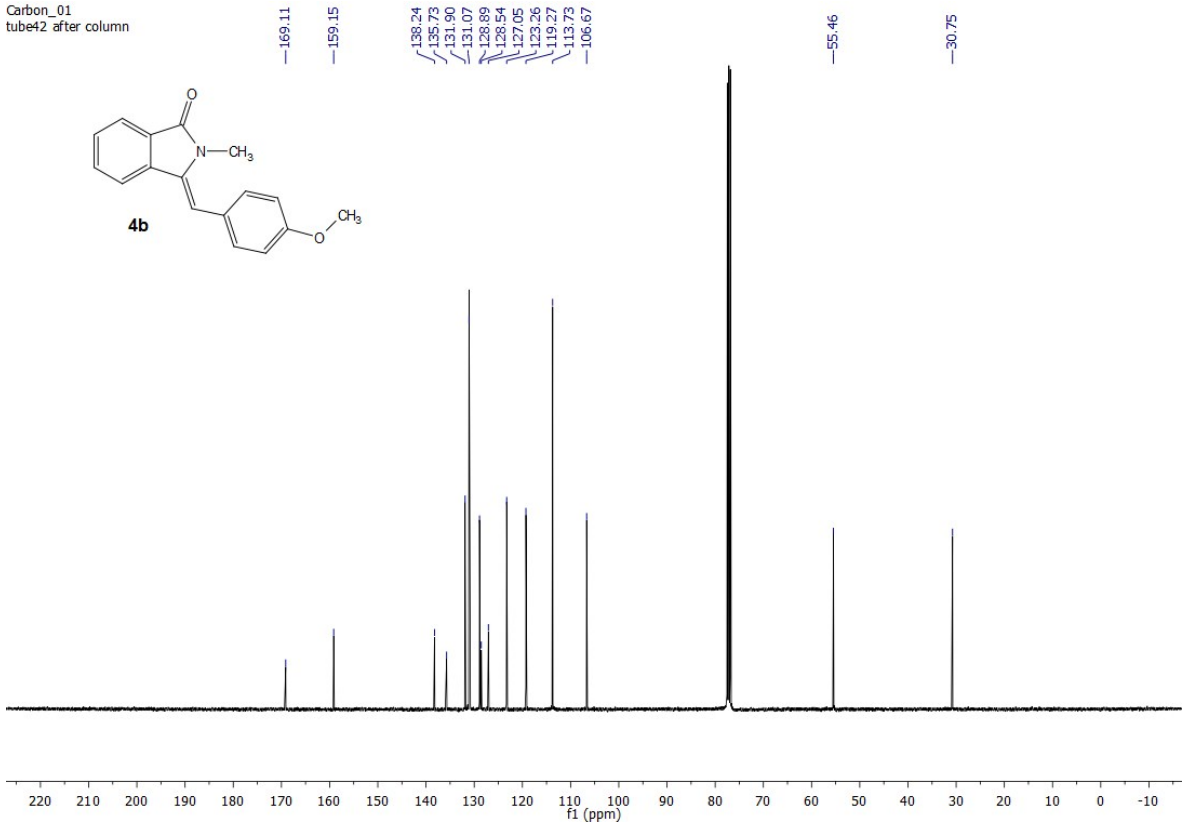

Proton_01
tubes 90-159
A (s) 8.03
B (dt) 7.89
C (dt) 7.80
D (m) 7.65
E (td) 7.53
F (d) 7.44
G (m) 7.32
H (s) 6.57
H2O
f1 (ppm)

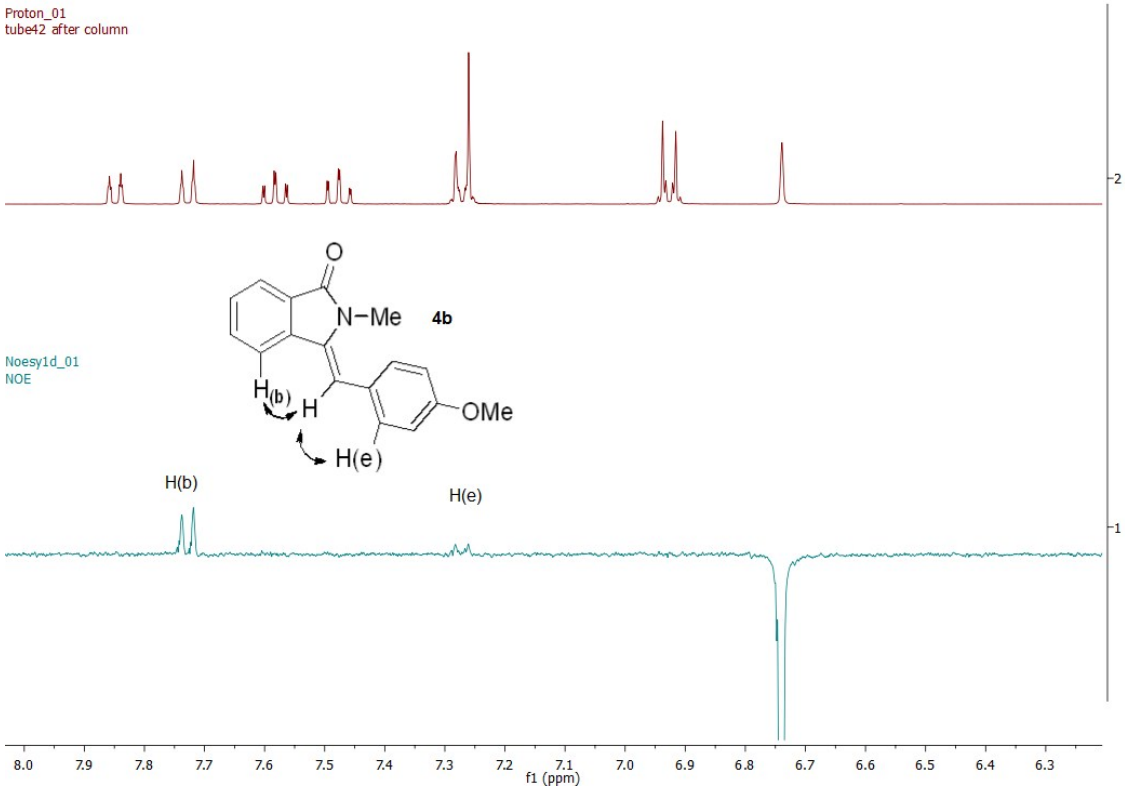
O
N—H
4a

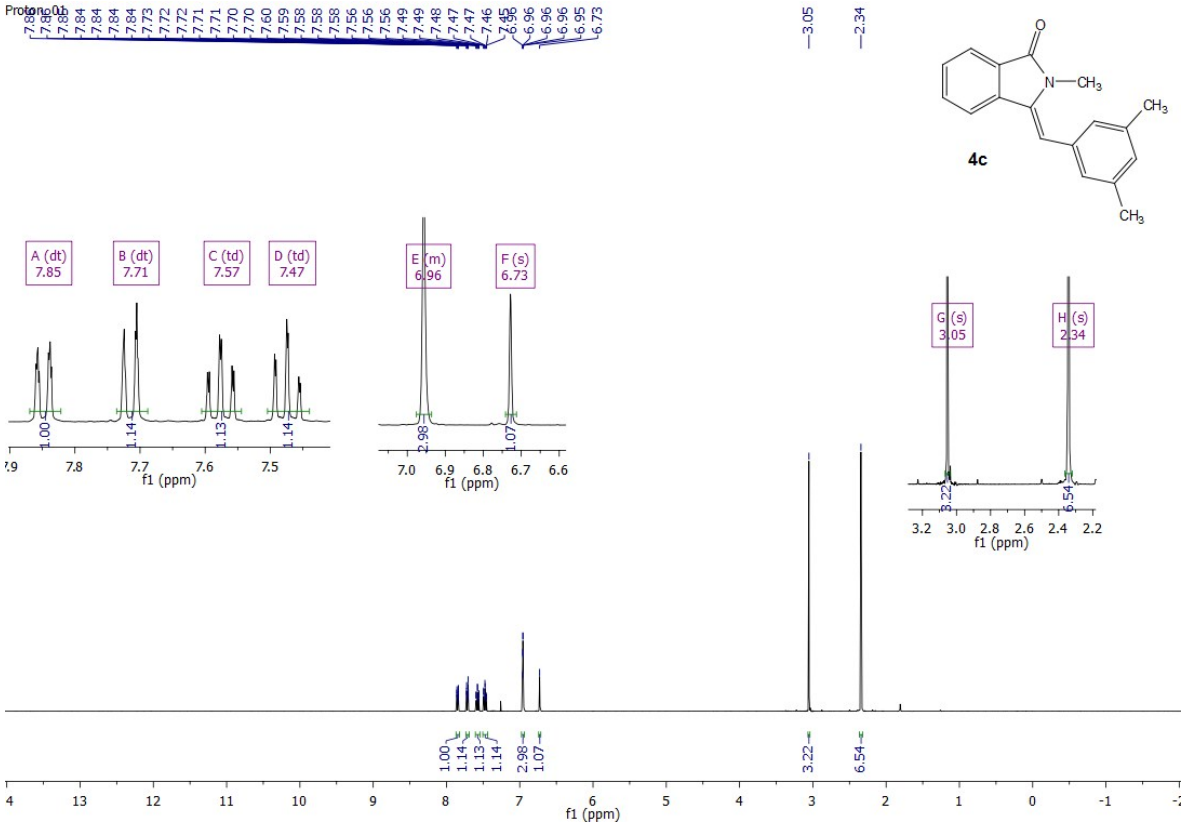

Carbon_01
Tubes 26-42 NMP 130deg overnight
f1 (ppm)

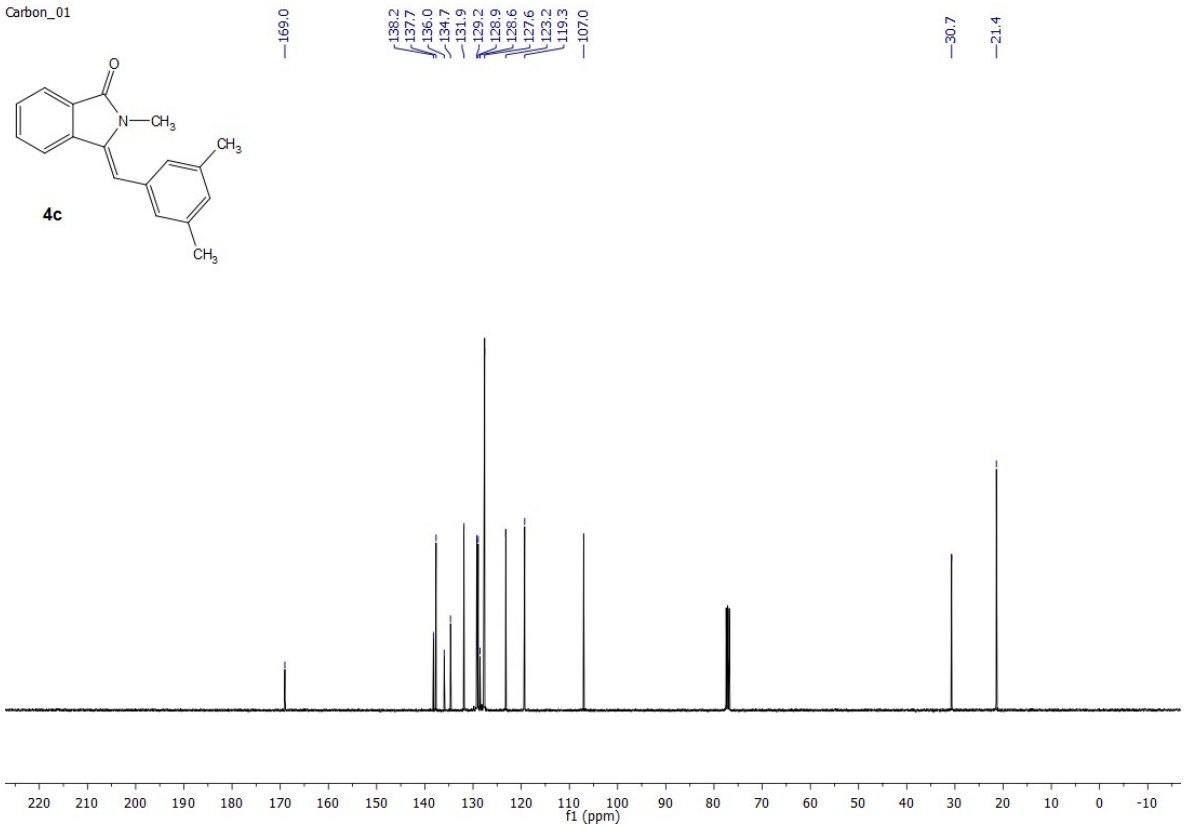
O
N—H
4a

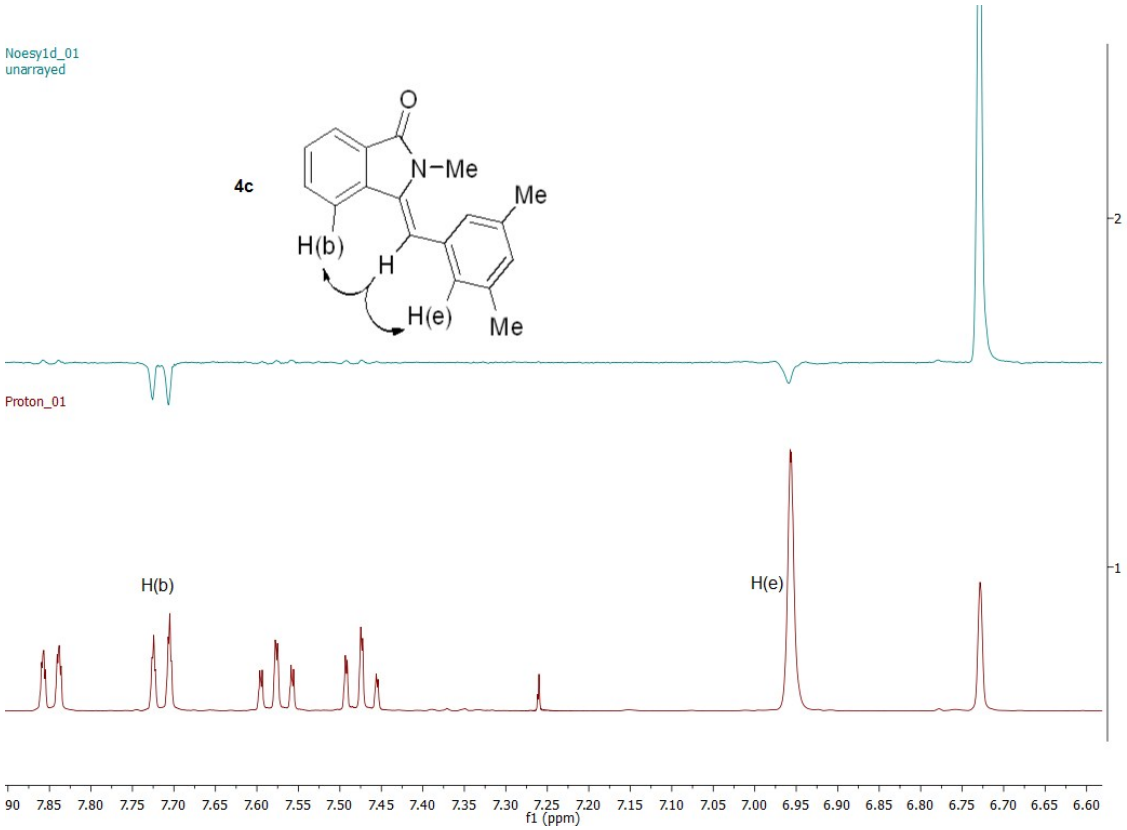

Proton_01
tube42 after column
7.86 7.86 7.86 7.84 7.84 7.84 7.74 7.72 7.72 7.72 7.60 7.60 7.58 7.58 7.57 7.56 7.50 7.49 7.48 7.48 7.46 7.46 7.28 7.28 7.28 7.28 7.27 7.26 7.26 7.26 7.25 7.25 7.25 6.95 6.94 6.93 6.92 6.92 6.91 6.74 3.85 3.07
1.00 1.18 1.17 1.15 2.83 2.27 1.13
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6
f1 (ppm)
O
N—CH3
CH3
O
4b
1.00 1.18 1.17 1.15 2.83 2.27 1.13 3.42 3.30
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)

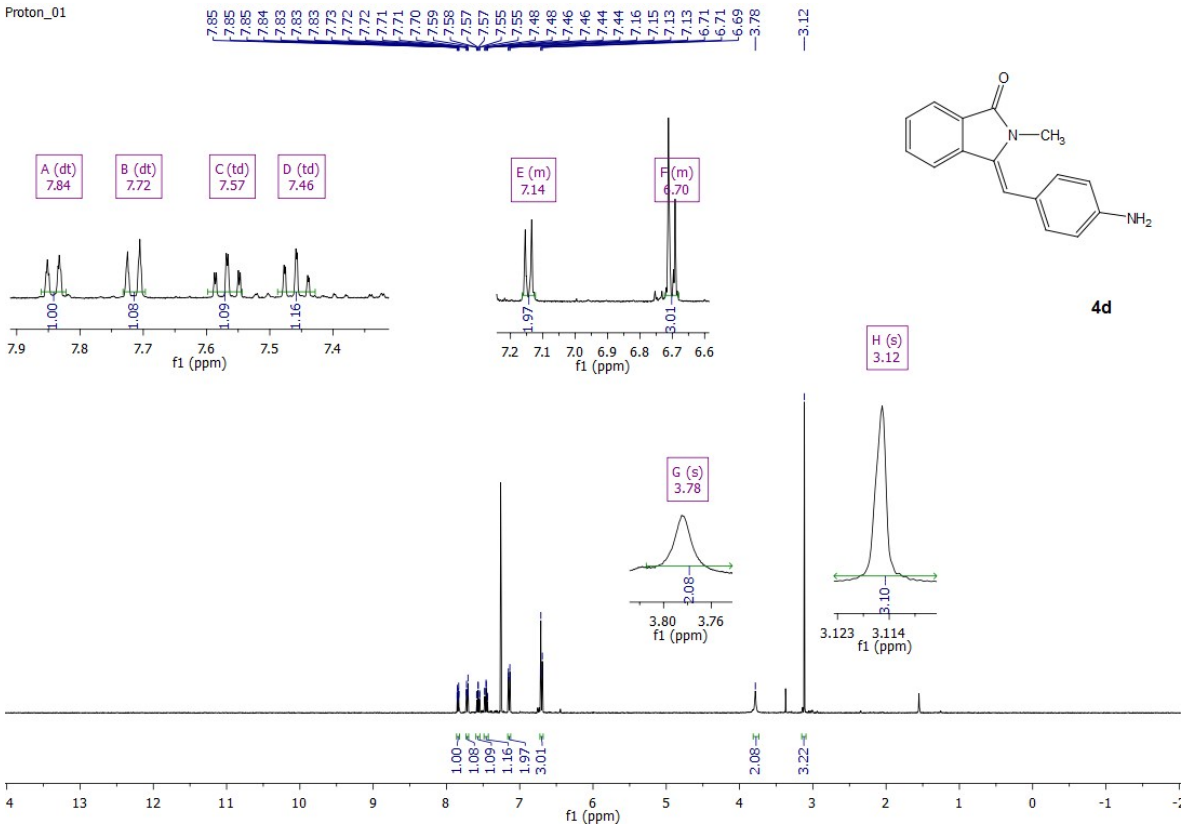

Carbon_01
tube42 after column
169.11 159.15 138.24 135.73 131.90 131.07 128.89 128.54 127.05 123.26 119.27 113.73 106.67 55.46 30.75
O
N—CH3
CH3
O
4b
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

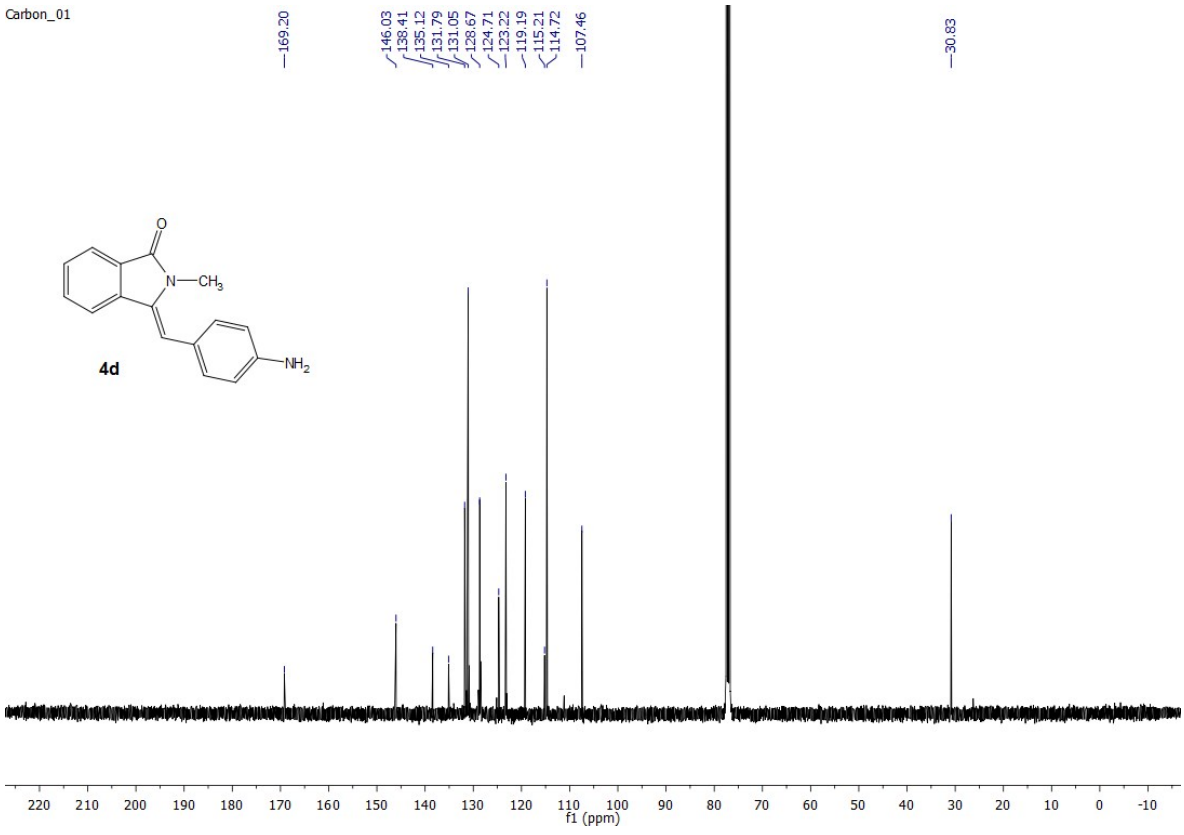

Proton_01
tube42 after column
Noesy1d_01
NOE
O
N–Me
4b
H(b)
H
OMe
H(e)
H(b)
H(e)
f1 (ppm)

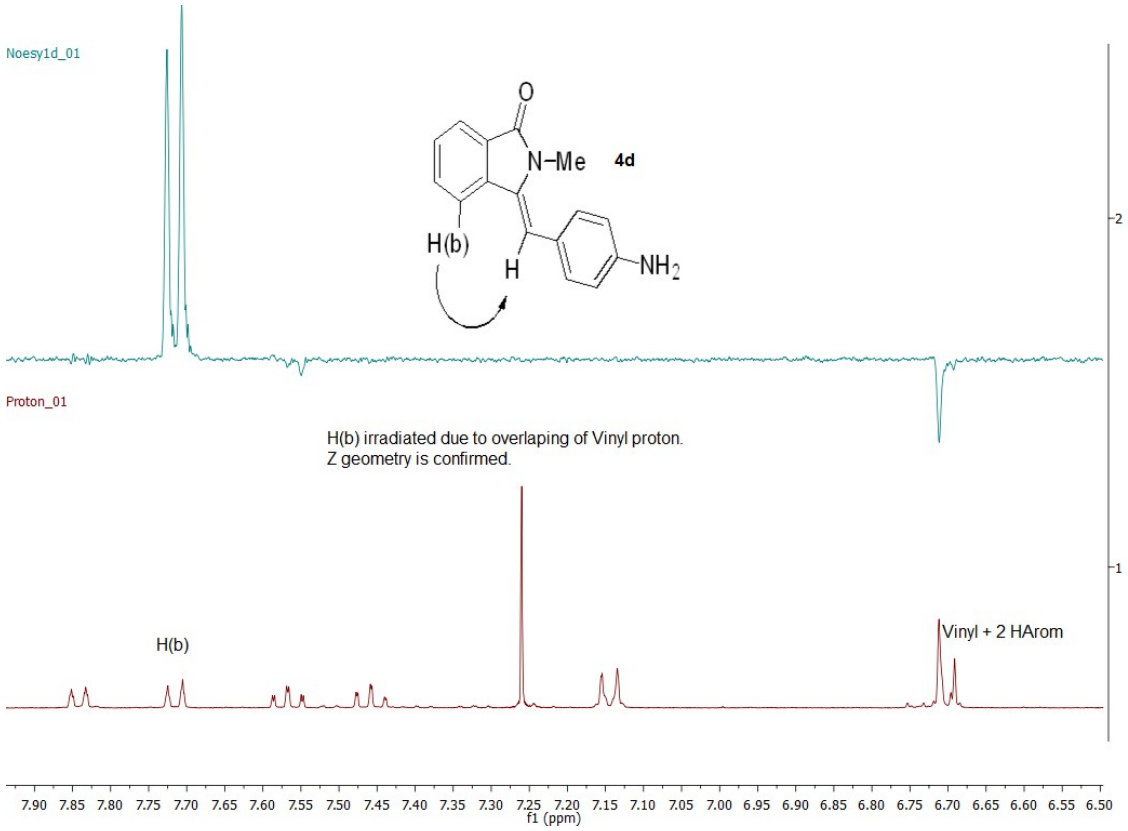

Proton_01
3.05
2.34
O
N—CH3
CH3
CH3
4c
A (dt) 7.85
B (dt) 7.71
C (td) 7.57
D (td) 7.47
E (m) 6.96
F (s) 6.73
G (s) 3.05
H (s) 2.34
1.00
1.14
1.13
1.14
2.98
1.07
3.22
6.54
f1 (ppm)

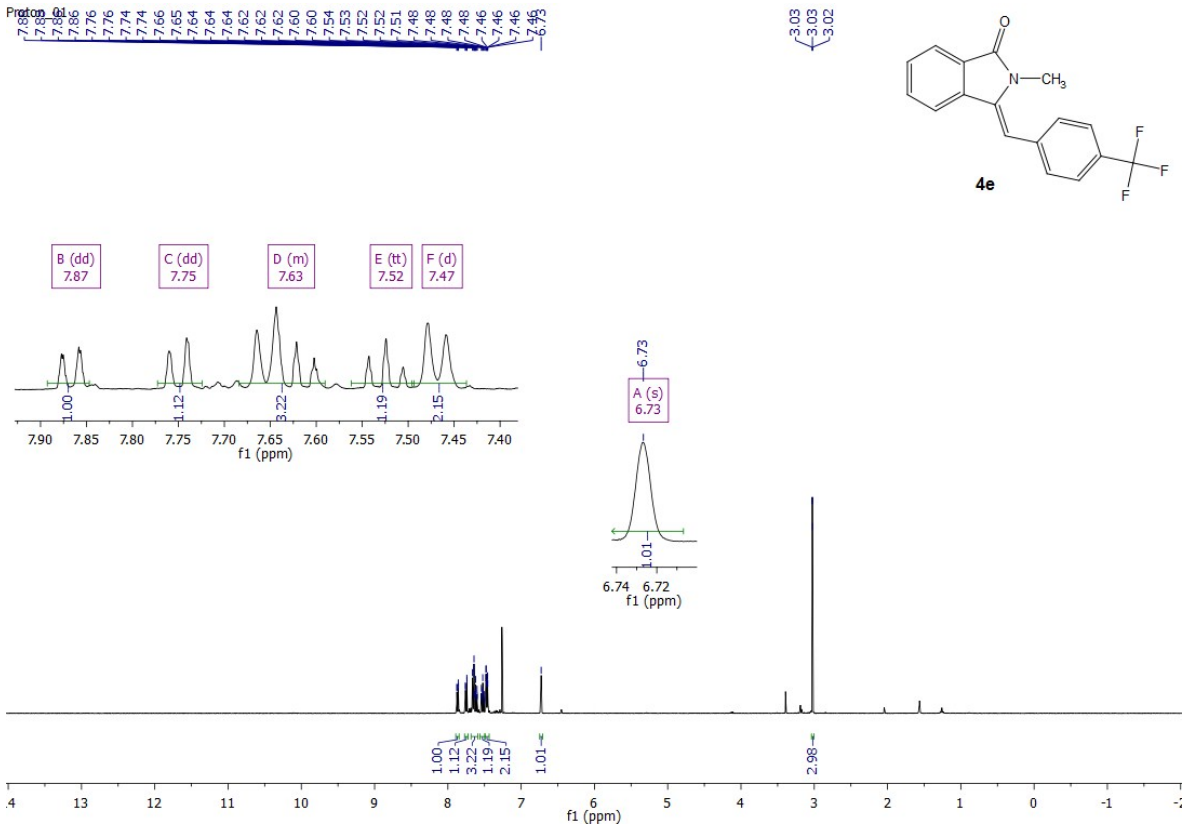

Carbon_01
169.0
138.2
137.7
136.0
134.7
131.9
129.2
128.9
128.6
127.6
123.2
119.3
107.0
30.7
21.4
O
N—CH3
CH3
CH3
4c
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Noesy1d_01
unarrayed
4c
O
N–Me
Me
H(b)
H
H(e)
Me
2
Proton_01
H(b)
H(e)
1
90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60
f1 (ppm)

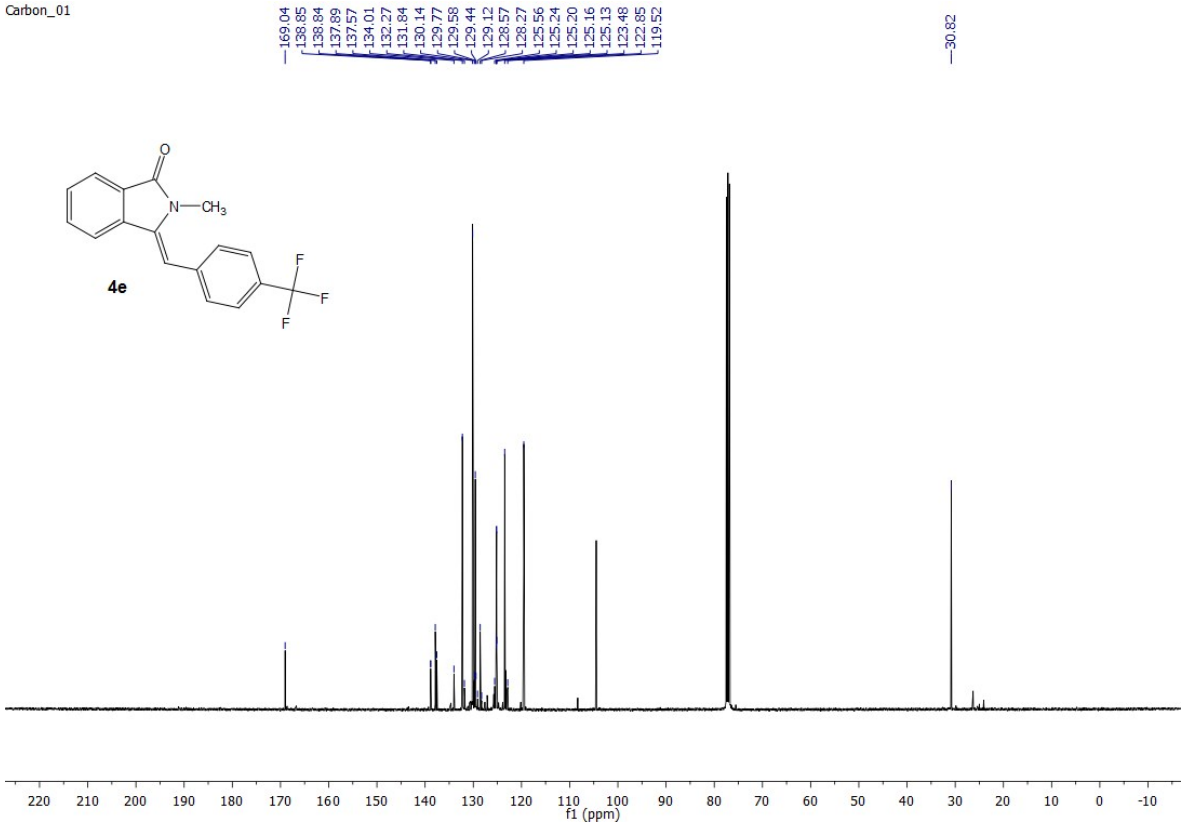

Proton_01
A (dt) 7.84
B (dt) 7.72
C (td) 7.57
D (td) 7.46
E (m) 7.14
F (m) 6.70
G (s) 3.78
H (s) 3.12
4d
f1 (ppm)


Carbon_01
169.20
146.03
138.41
135.12
131.79
131.05
128.67
124.71
123.22
119.19
115.21
114.72
107.46
30.83
4d
f1 (ppm)

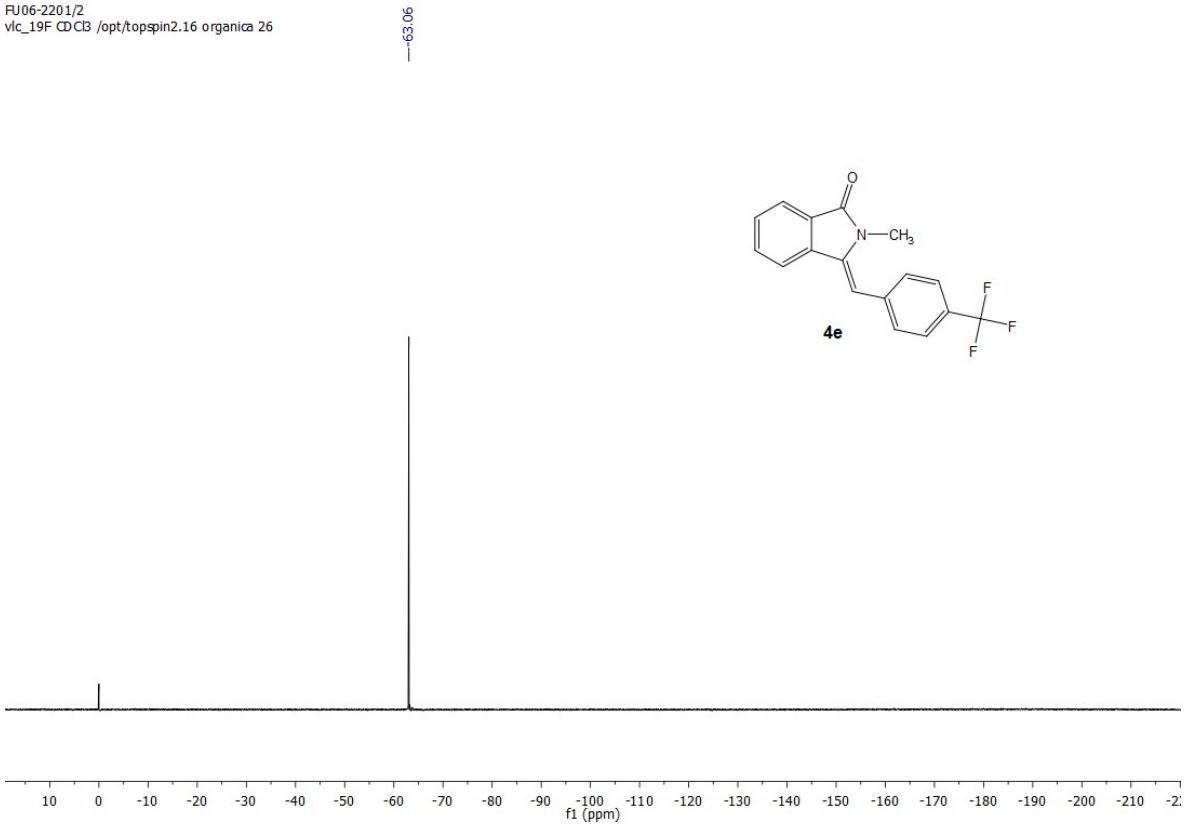

Noesy1d_01
O
N–Me
4d
H(b)
H
NH2
2
Proton_01
H(b) irradiated due to overlaping of Vinyl proton.
Z geometry is confirmed.
1
H(b)
Vinyl + 2 HArom
7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60 6.55 6.50
f1 (ppm)

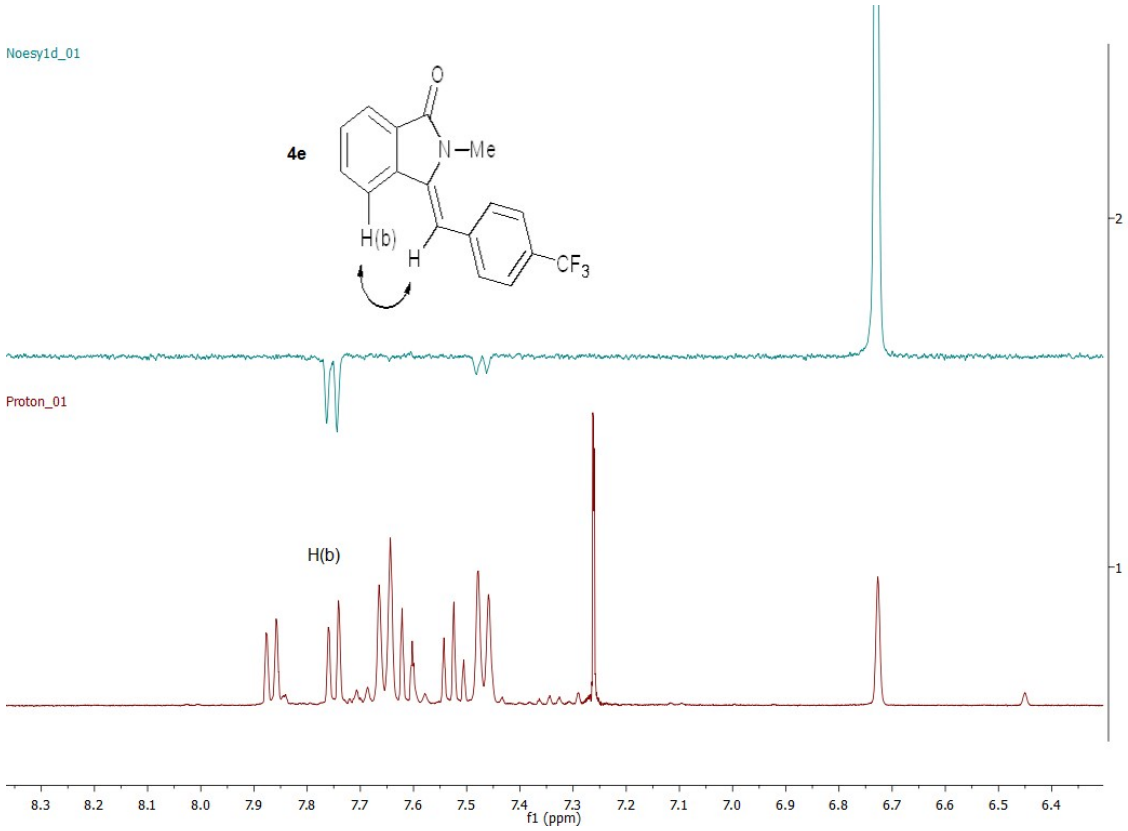

Proton_01
3.03
3.03
3.02
O
N—CH3
F
F
F
4e
B (dd)
7.87
C (dd)
7.75
D (m)
7.63
E (tt)
7.52
F (d)
7.47
1.00
1.12
3.22
1.19
2.15
7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40
f1 (ppm)
6.73
A (s)
6.73
1.01
6.74 6.72
f1 (ppm)
1.00
1.12
3.22
1.19
2.15
1.01
2.98
f1 (ppm)

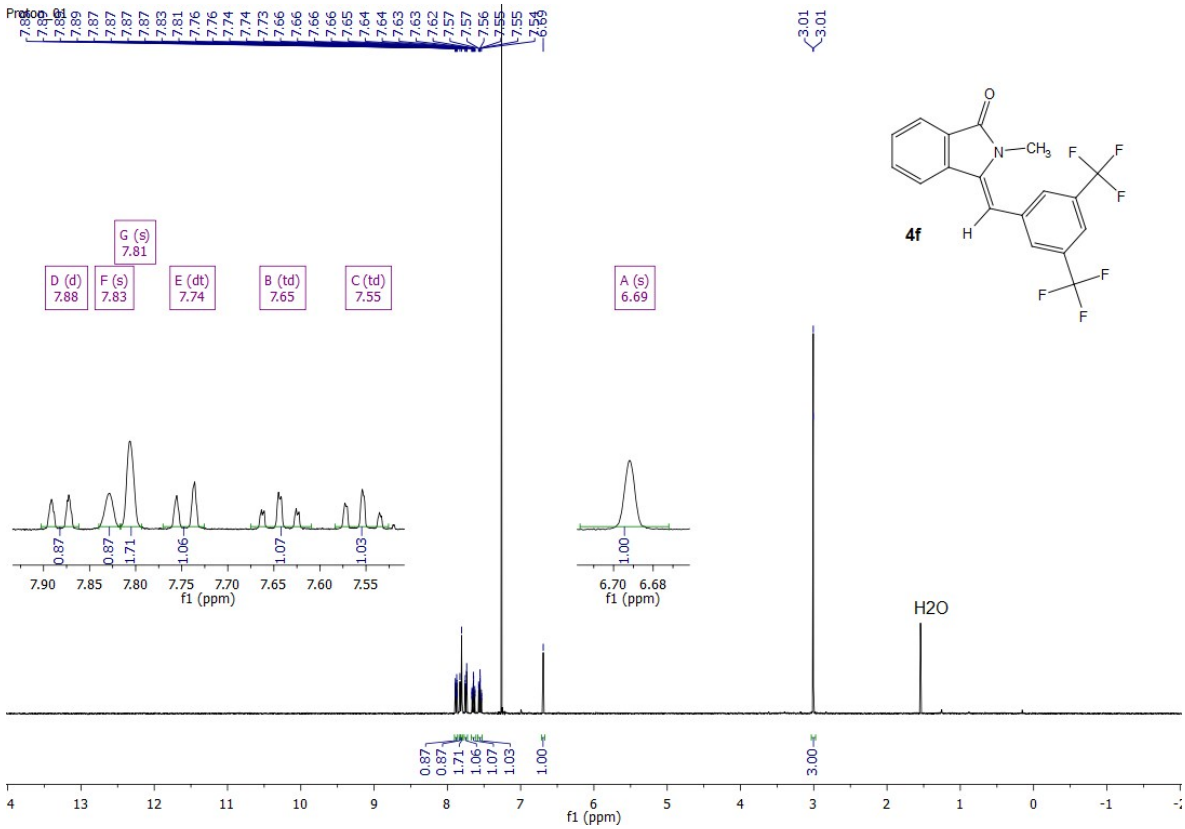
Carbon_01

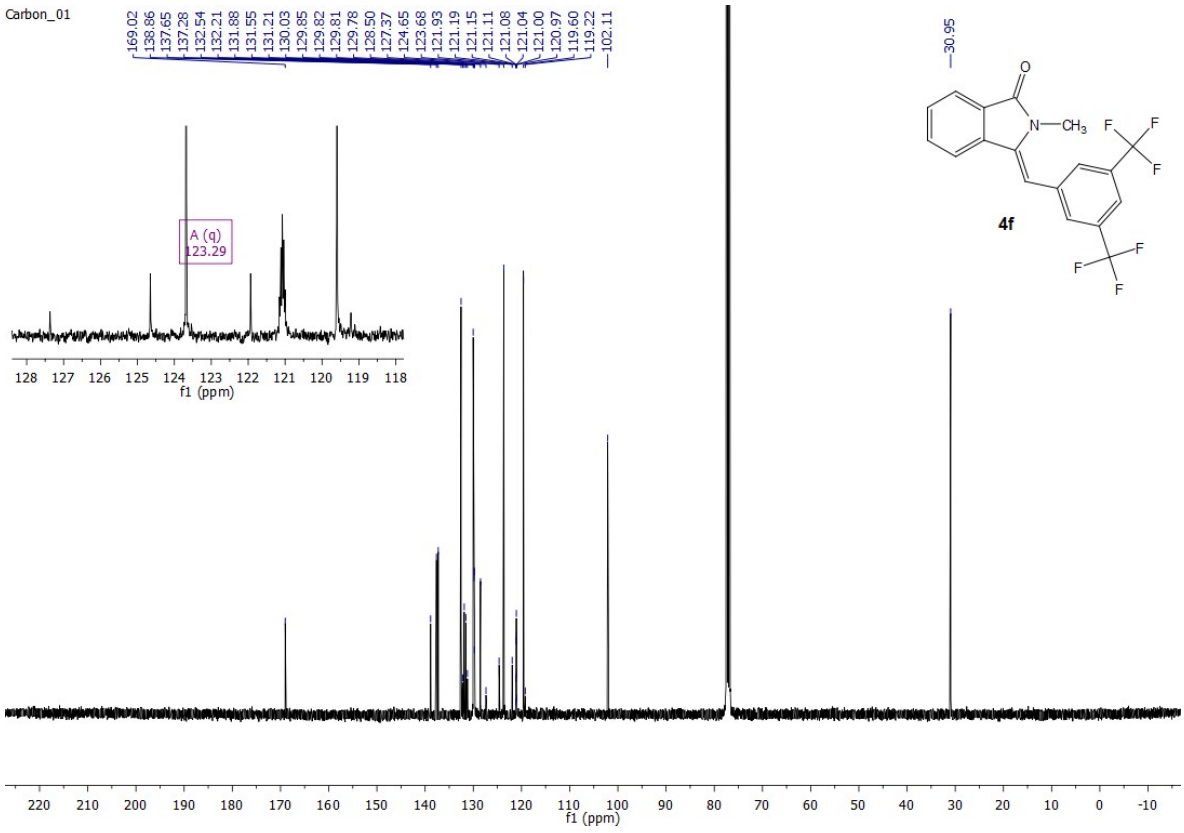


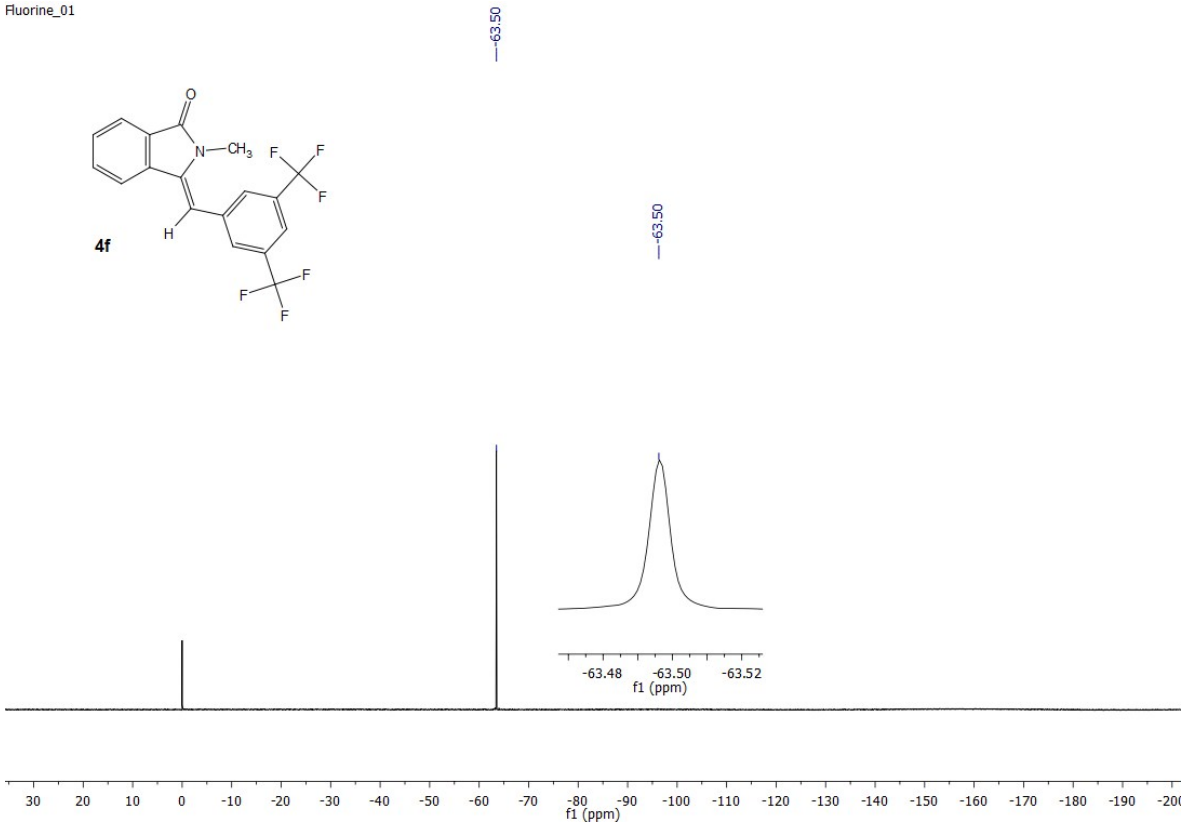
FU06-2201/2
vlc_19F CDCl3 /opt/topspin2.16 organica 26

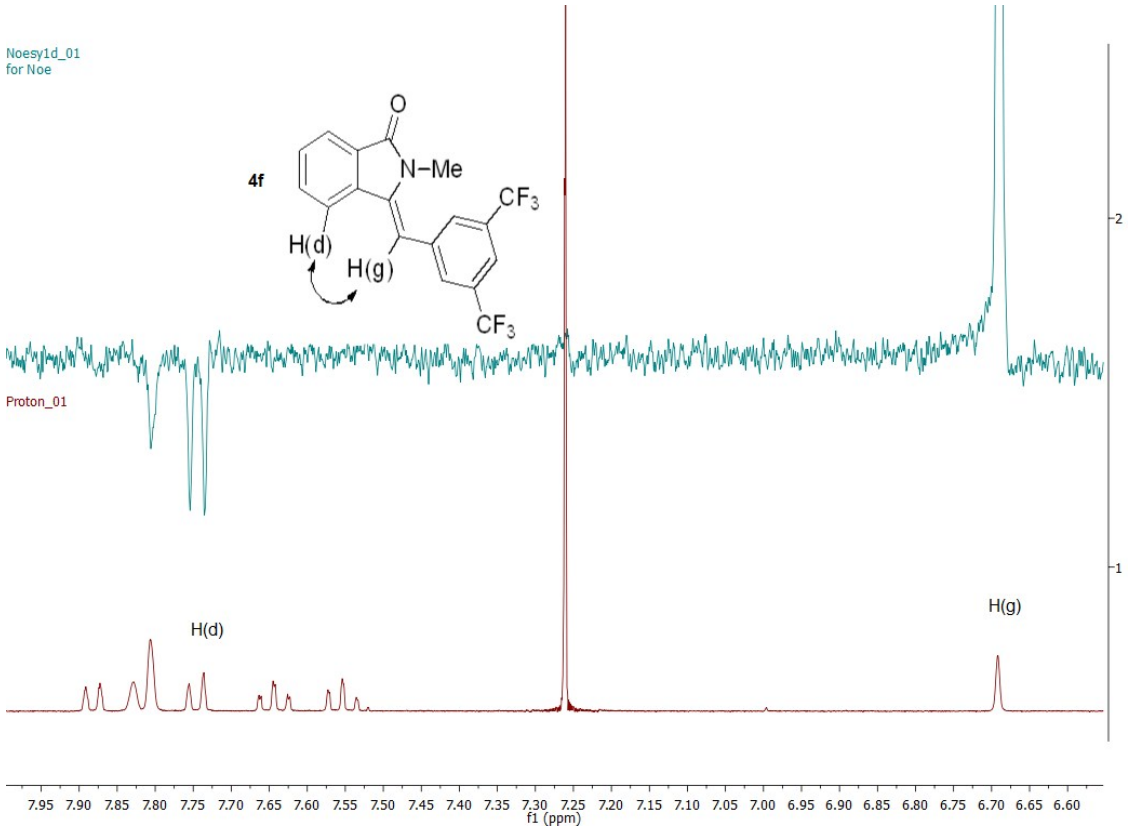


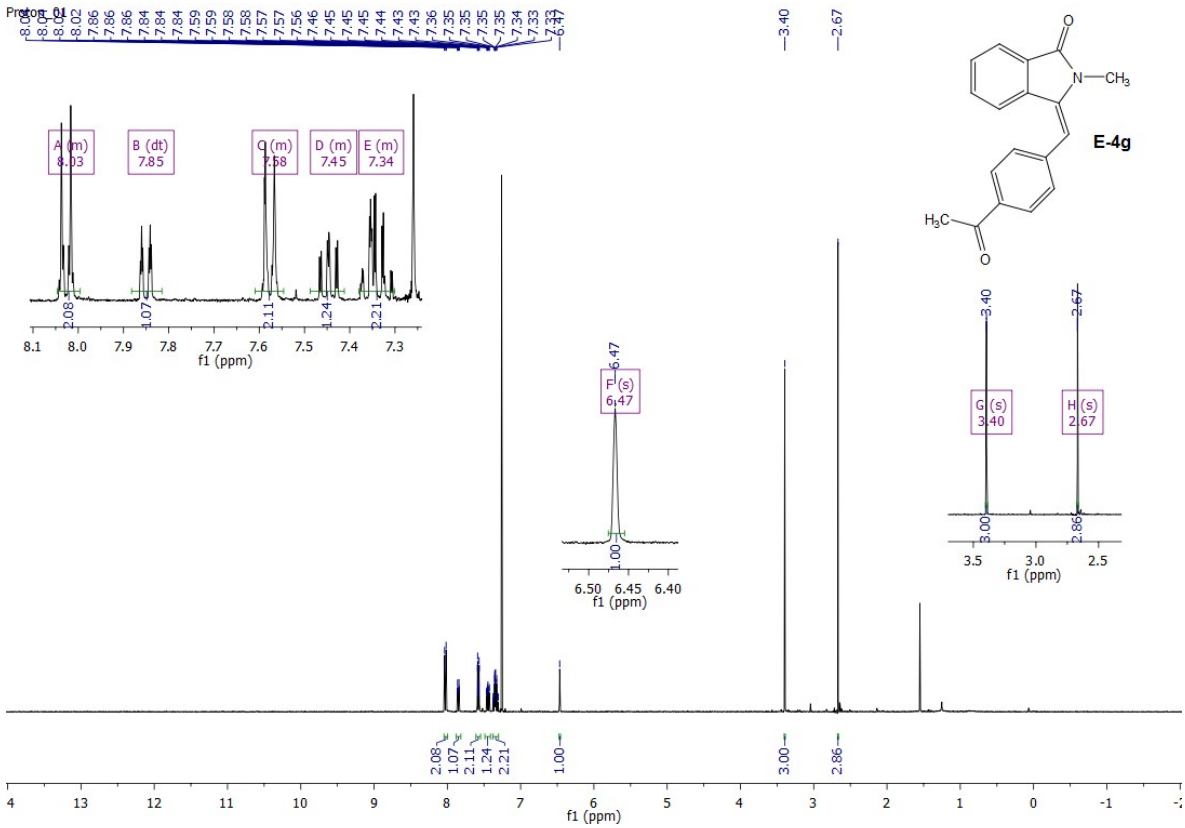

Noesy1d_01
4e
O
N–Me
H(b)
H
CF3
Proton_01
H(b)
2
1
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4
f1 (ppm)

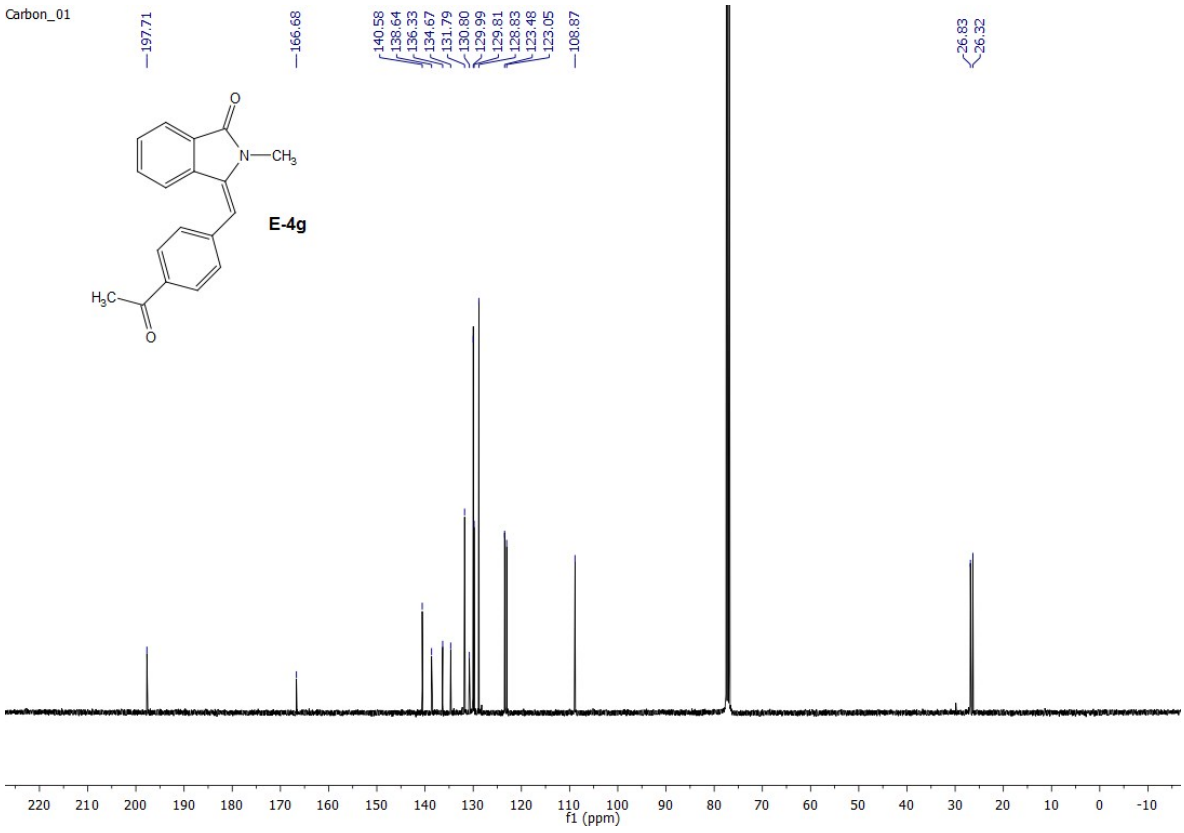

Proton_01
3.01
3.01
4f
O
N–CH3
F
F
F
H
F
F
F
G (s)
7.81
D (d)
7.88
F (s)
7.83
E (dt)
7.74
B (td)
7.65
C (td)
7.55
A (s)
6.69
0.87
0.87
1.71
1.06
1.07
1.03
1.00
3.00
H2O
7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55
f1 (ppm)
6.70 6.68
f1 (ppm)
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)

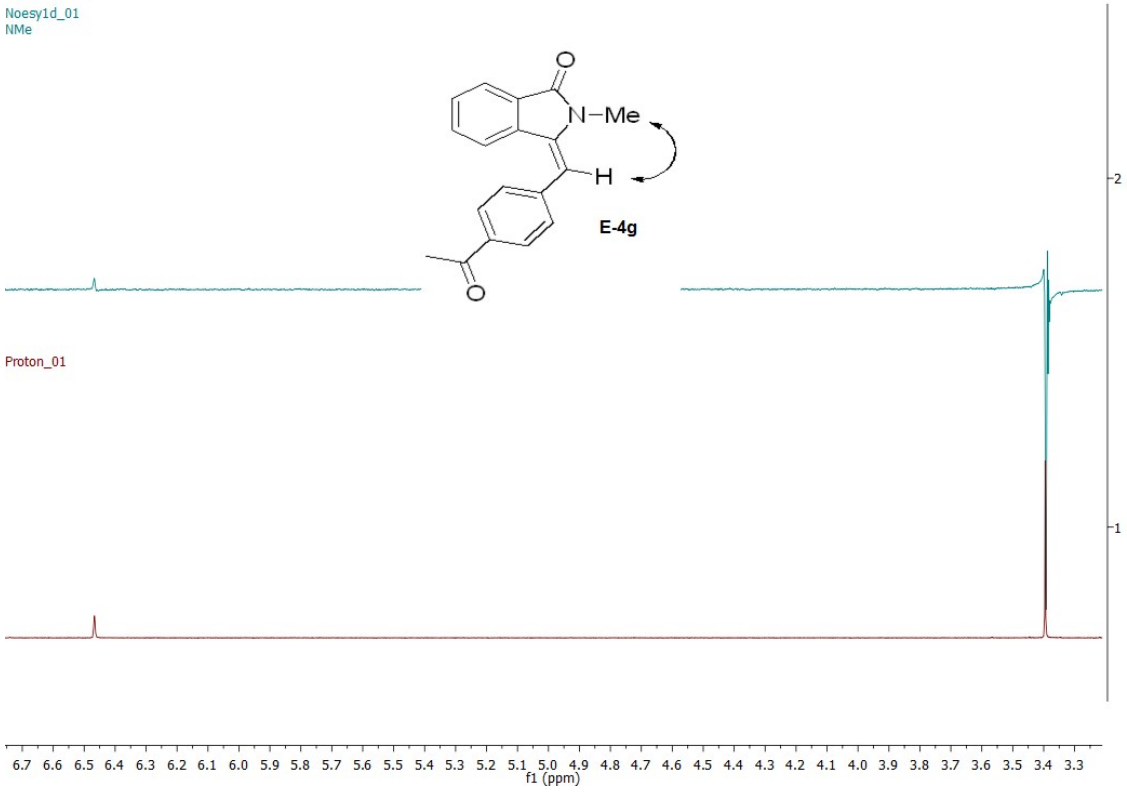

Carbon_01
169.02
138.86
137.65
137.28
132.54
132.21
131.88
131.55
131.21
130.03
129.85
129.82
129.81
129.78
128.50
127.37
124.65
123.68
121.93
121.19
121.15
121.11
121.08
121.04
121.00
120.97
119.60
119.22
102.11
30.95
A (q)
123.29
f1 (ppm)
4f
f1 (ppm)

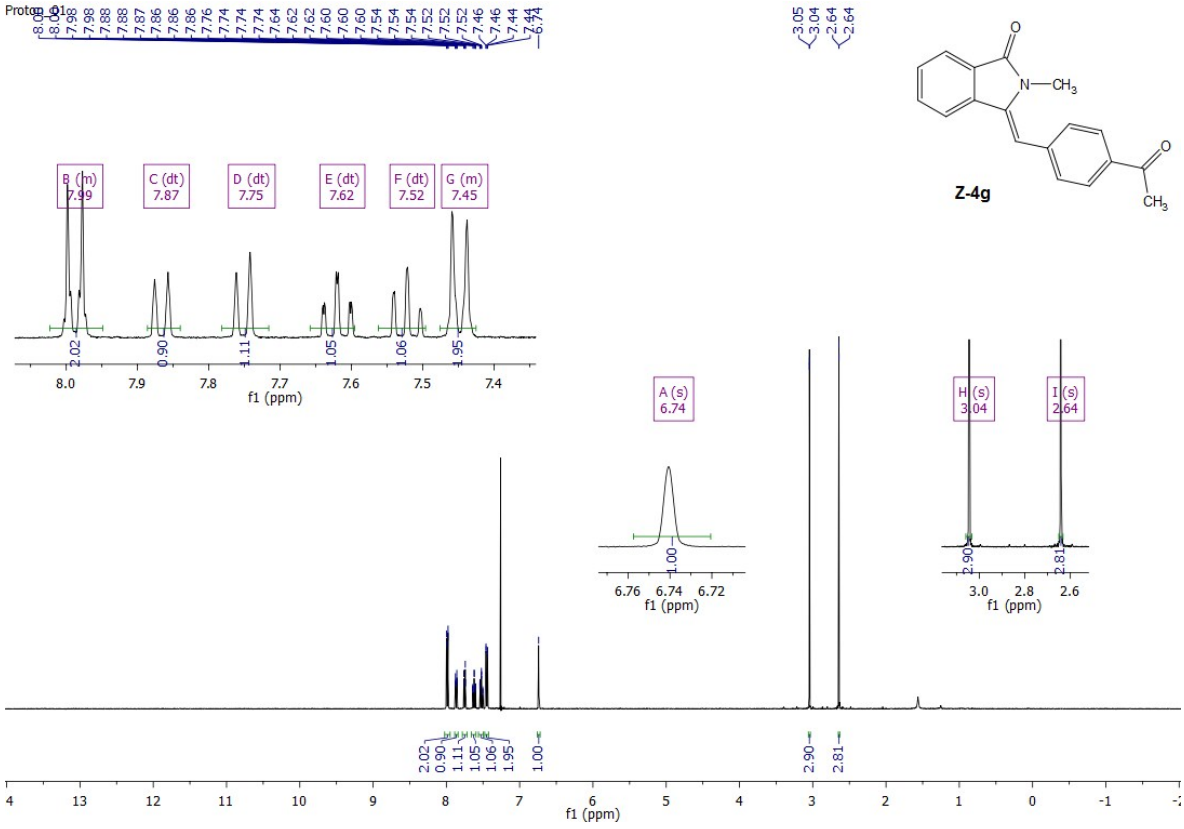

Fluorine_01
-63.50
-63.50
4f
-63.48
-63.50
-63.52
f1 (ppm)
f1 (ppm)


Noesy1d_01
for Noe
4f
O
N–Me
CF3
H(d)
H(g)
CF3
Proton_01
H(d)
H(g)
2
1
7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60
f1 (ppm)

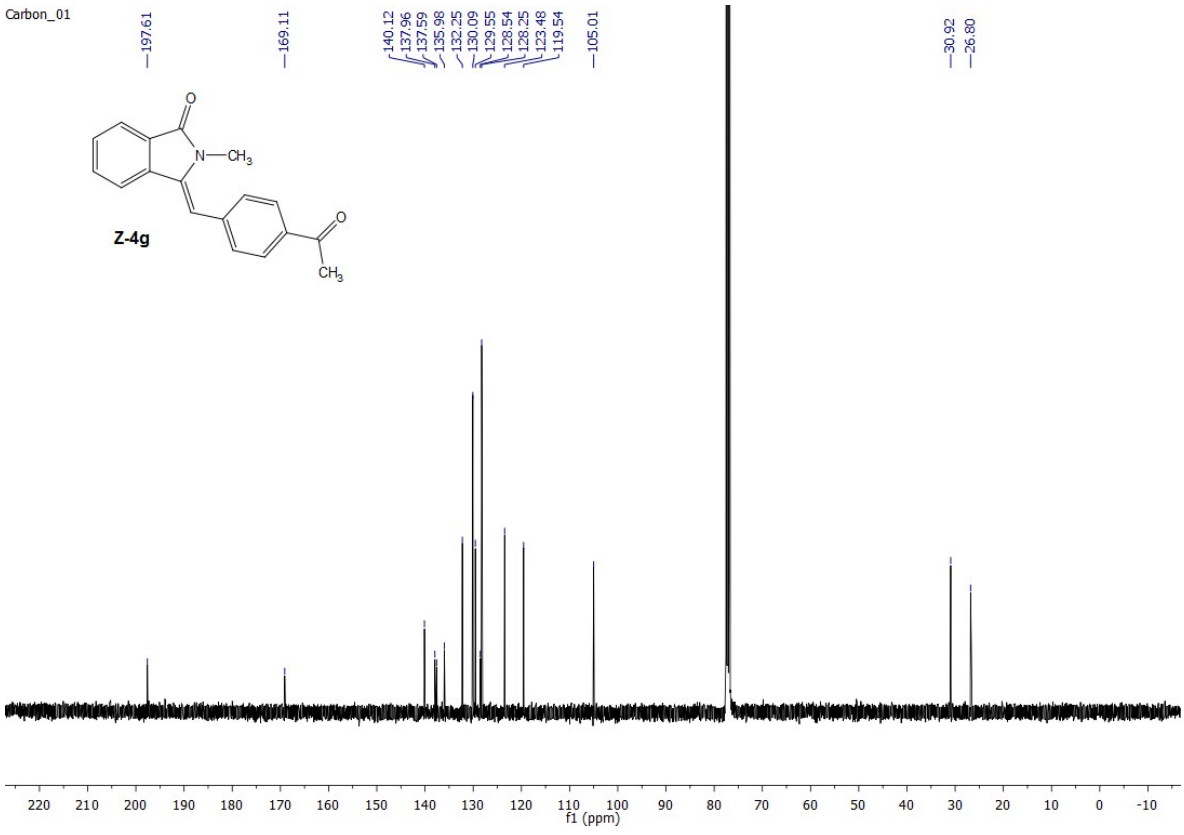

Proton_01
8.03 8.01 8.02 7.86 7.86 7.84 7.84 7.84 7.59 7.59 7.58 7.58 7.57 7.57 7.56 7.46 7.45 7.45 7.44 7.43 7.43 7.36 7.35 7.35 7.35 7.34 7.33 7.33 6.47
3.40
2.67
A (m) 8.03
B (dt) 7.85
C (m) 7.58
D (m) 7.45
E (m) 7.34
2.08
1.07
2.11
1.24
2.21
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3
f1 (ppm)
F (s) 6.47
1.00
6.50 6.45 6.40
f1 (ppm)
N–CH3
E-4g
H3C
O
G (s) 3.40
H (s) 2.67
3.00
2.86
3.5 3.0 2.5
f1 (ppm)
2.08
1.07
2.11
1.24
2.21
1.00
3.00
2.86
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


Carbon_01
197.71
166.68
140.58
138.64
136.33
134.67
131.79
130.80
129.99
129.81
128.83
123.48
123.05
108.87
26.83
26.32
N—CH3
E-4g
H3C
O
f1 (ppm)

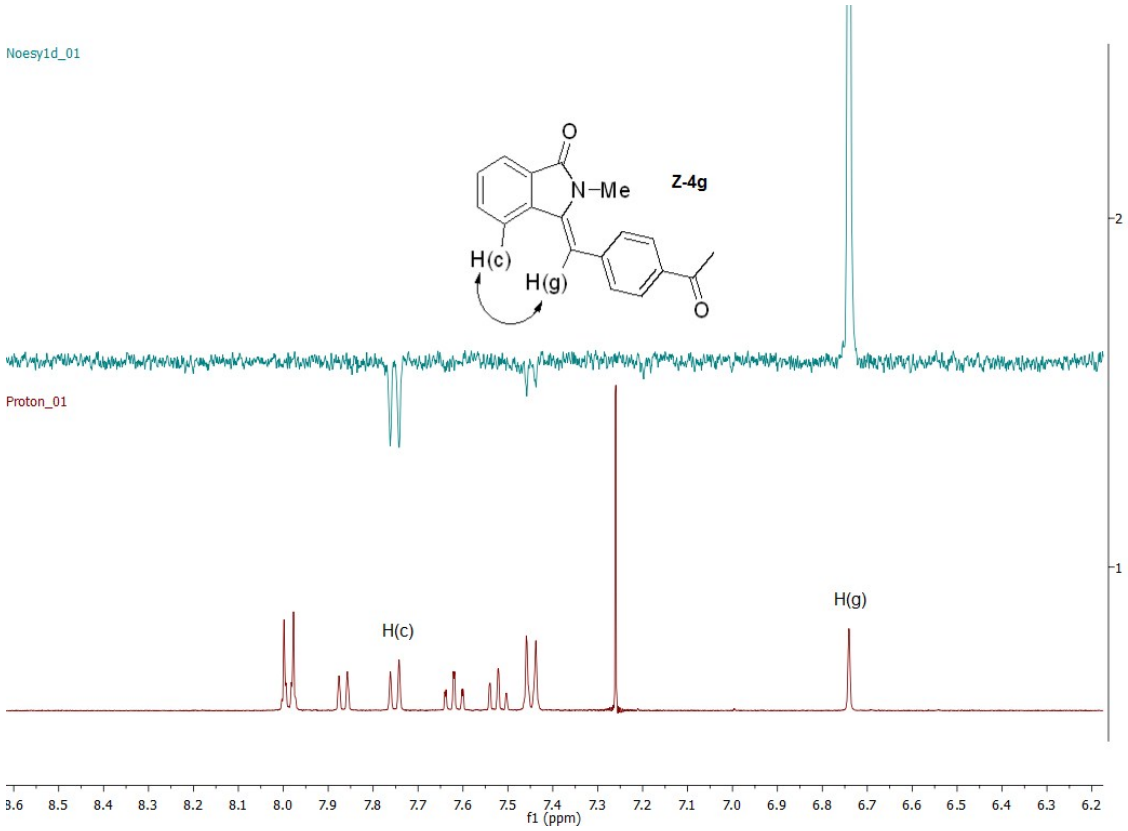

Noesy1d_01
NMe
N–Me
H
E-4g
Proton_01
f1 (ppm)


Proton_01
B (m) 7.99
C (dt) 7.87
D (dt) 7.75
E (dt) 7.62
F (dt) 7.52
G (m) 7.45
A (s) 6.74
H (s) 3.04
I (s) 2.64
f1 (ppm)

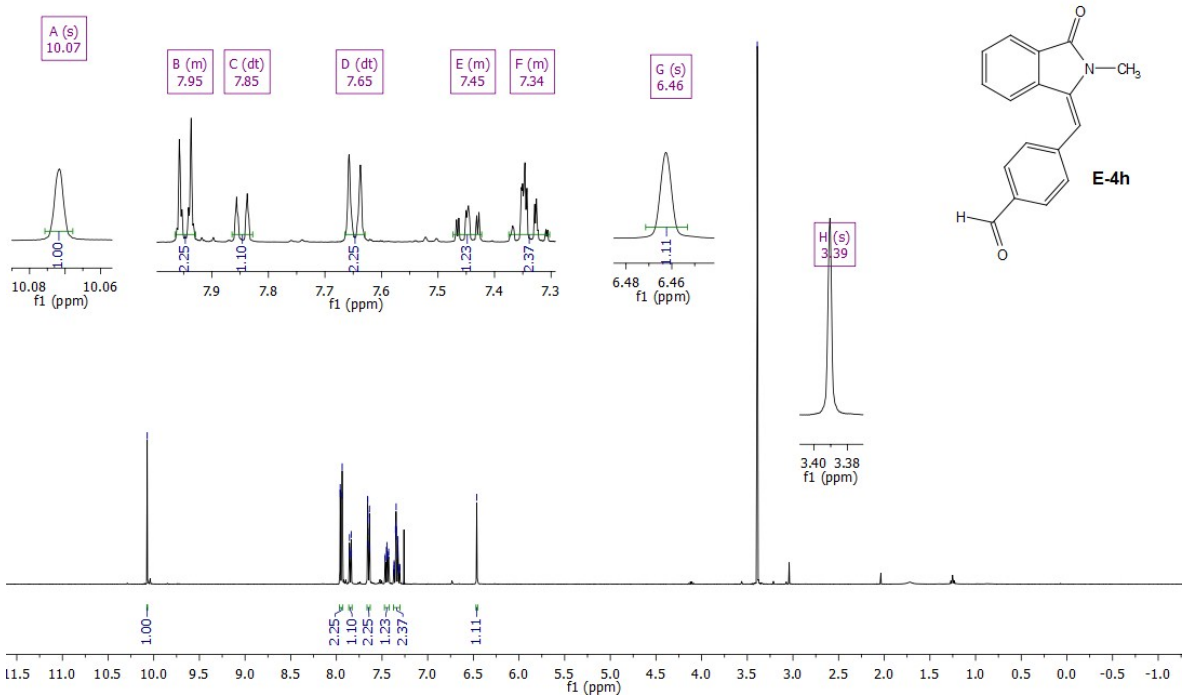
Z-4g

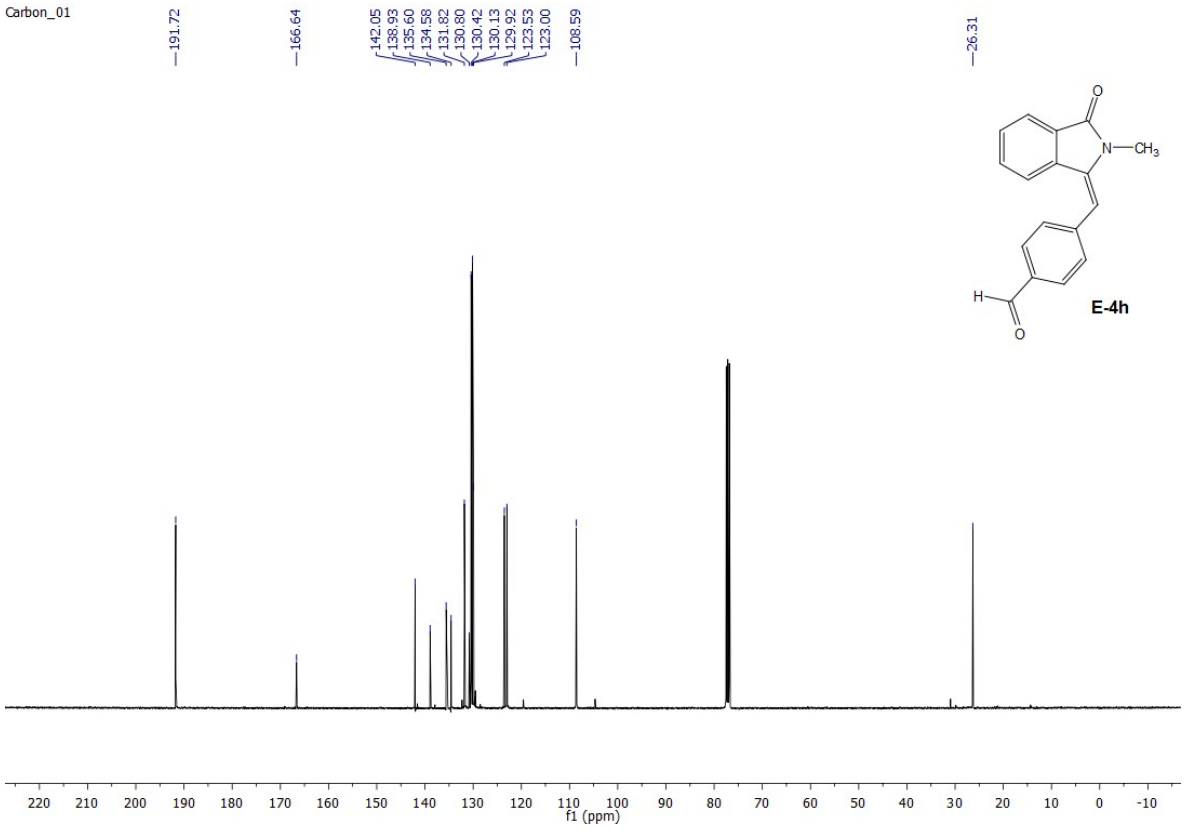

Carbon_01
f1 (ppm)

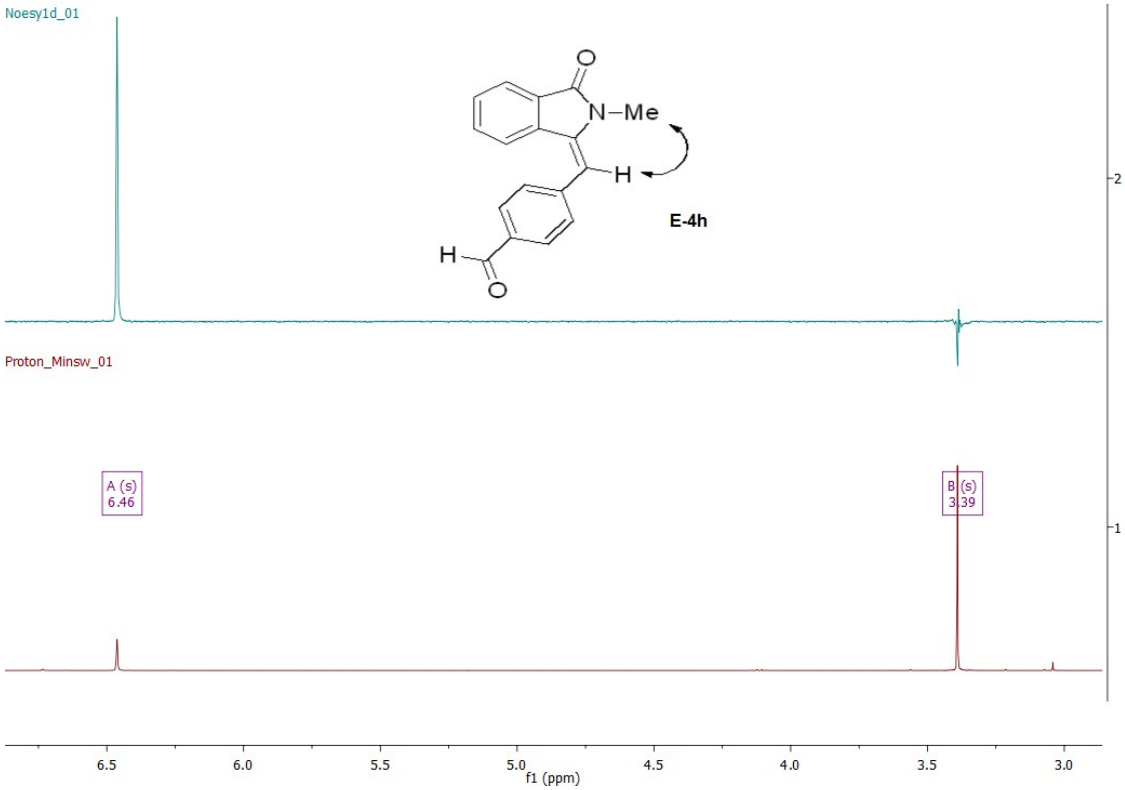
Z-4g

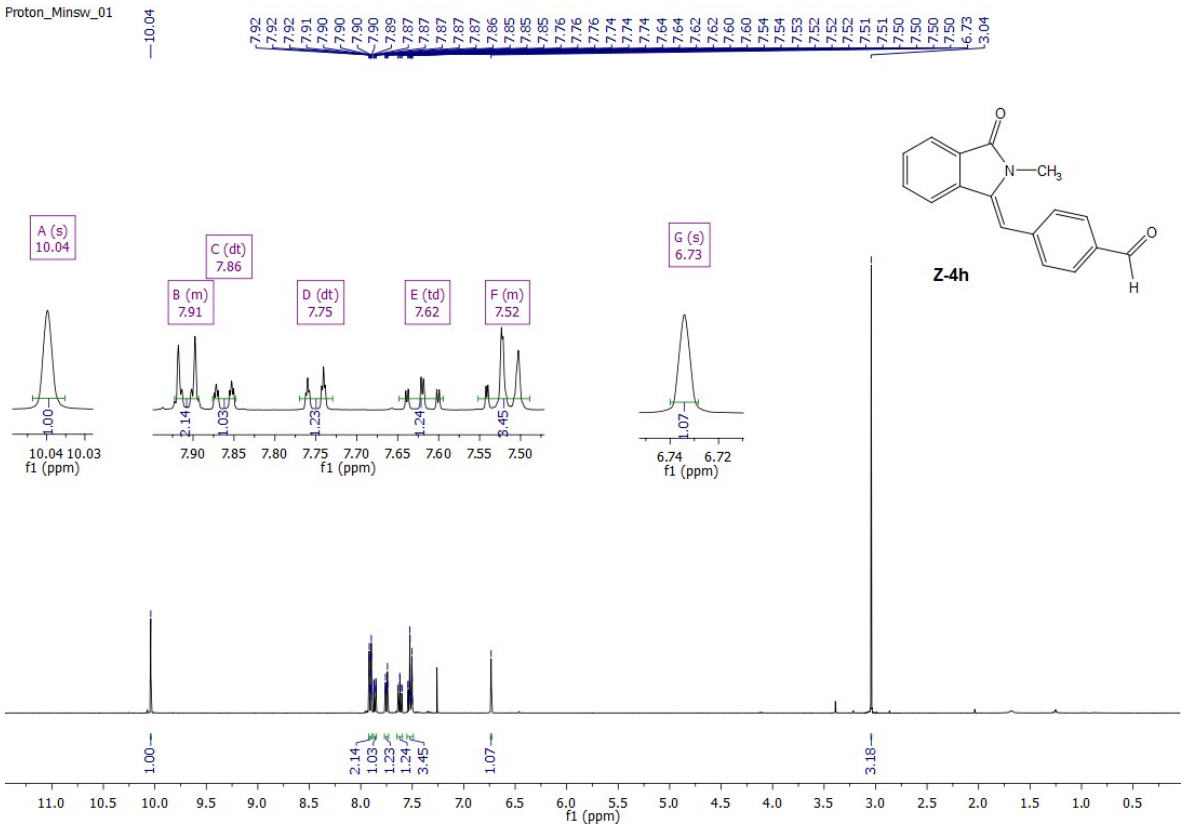

Noesy1d_01
O
N–Me
Z-4g
H(c)
H(g)
O
Proton_01
H(c)
H(g)
2
1
8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2
f1 (ppm)

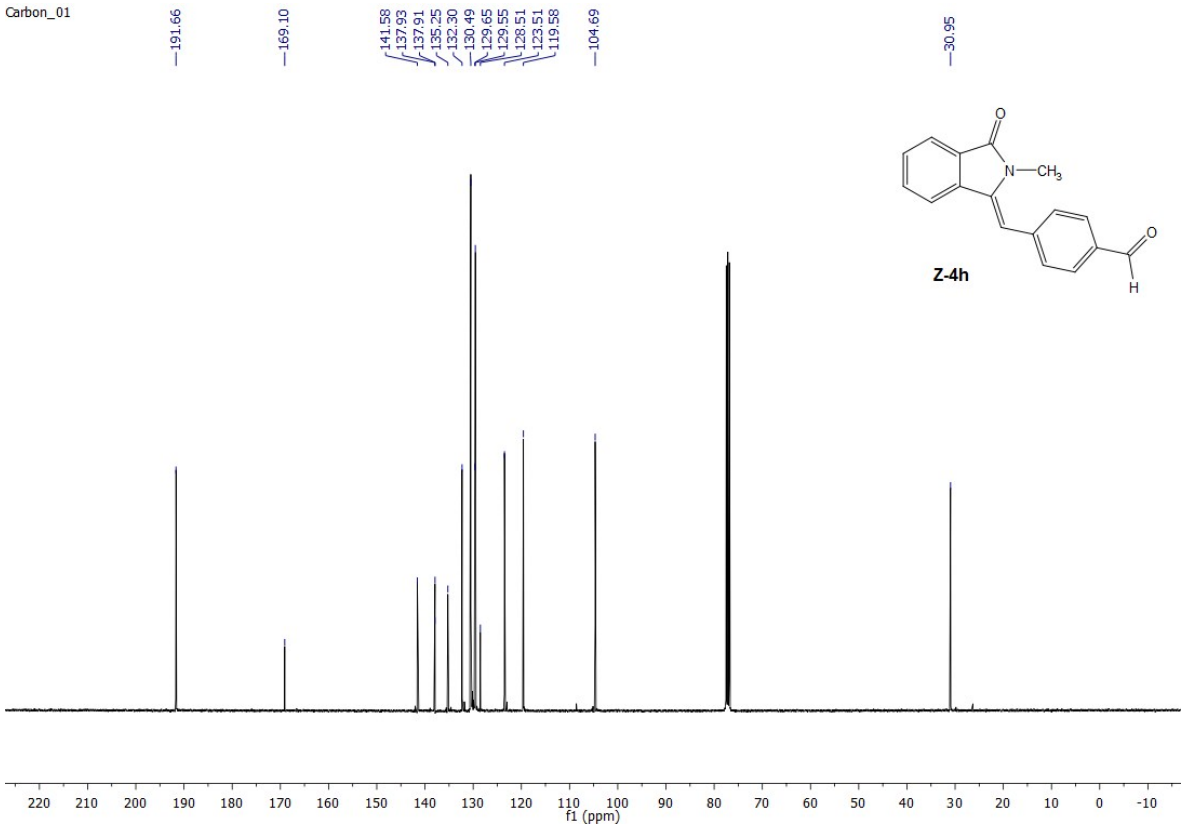

Proton_Minsw_01
10.07
7.96 7.96 7.94 7.94 7.86 7.86 7.85 7.84 7.84 7.83 7.66 7.66 7.66 7.66 7.65 7.64 7.64 7.64 7.63 7.47 7.46 7.45 7.45 7.45 7.44 7.44 7.43 7.43 7.37 7.37 7.37 7.37 7.35 7.35 7.35 7.35 7.34 7.34 7.33 7.33 7.33 7.33 7.32 7.31 7.31 7.31 7.31 6.46 3.39

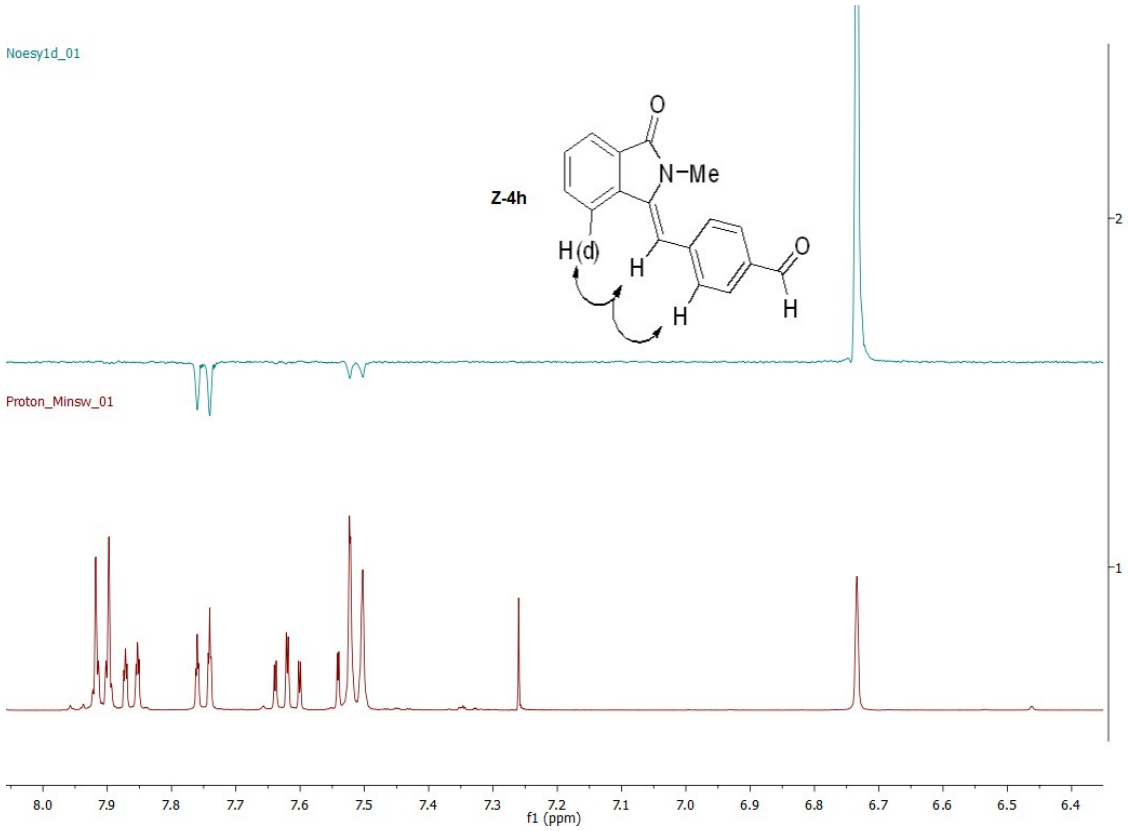

A (s)
10.07
B (m)
7.95
C (dt)
7.85
D (dt)
7.65
E (m)
7.45
F (m)
7.34
G (s)
6.46
H (s)
3.39
1.00
2.25
1.10
2.25
1.23
2.37
1.11
10.08 10.06
f1 (ppm)
7.9 7.8 7.7 7.6 7.5 7.4 7.3
f1 (ppm)
6.48 6.46
f1 (ppm)
3.40 3.38
f1 (ppm)
O
N–CH3
E-4h
H
O
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

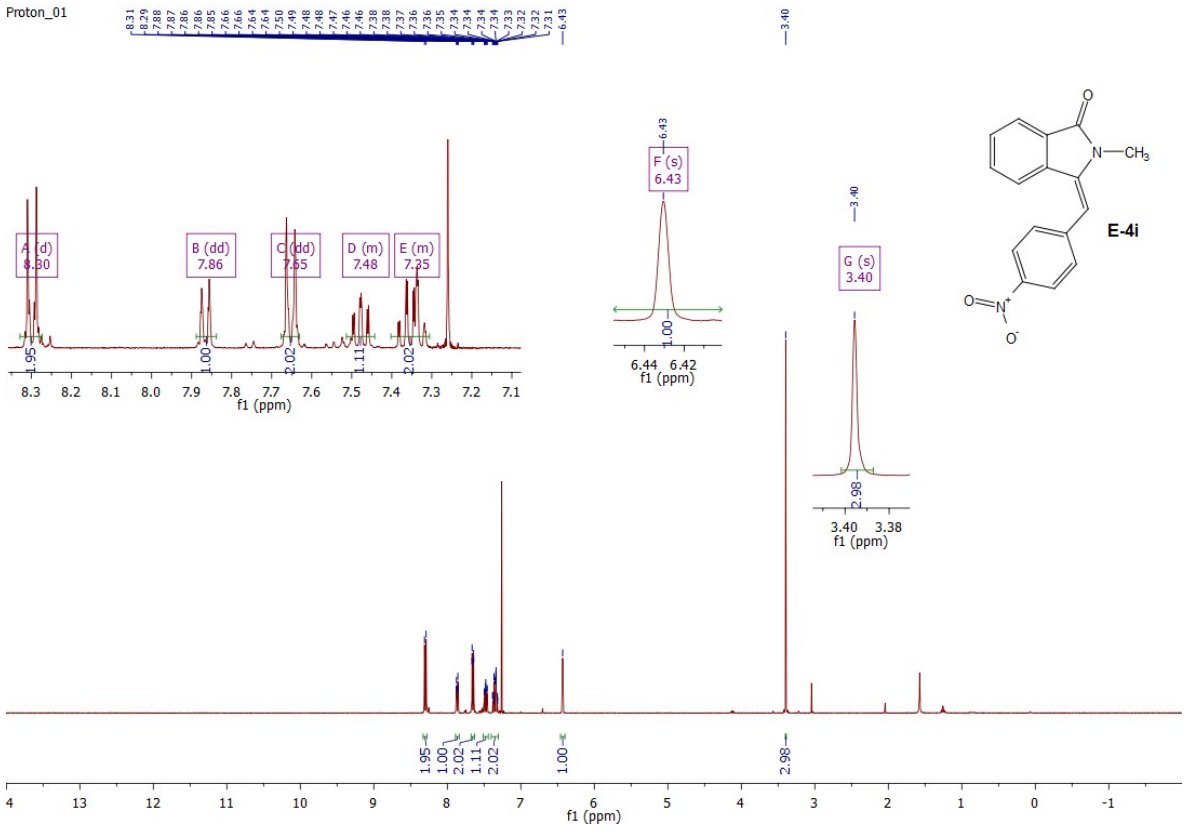

Carbon_01
191.72
166.64
142.05
138.93
135.60
134.58
131.82
130.80
130.42
130.13
129.92
123.53
123.00
108.59
26.31
N—CH3
O
H
O
E-4h
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

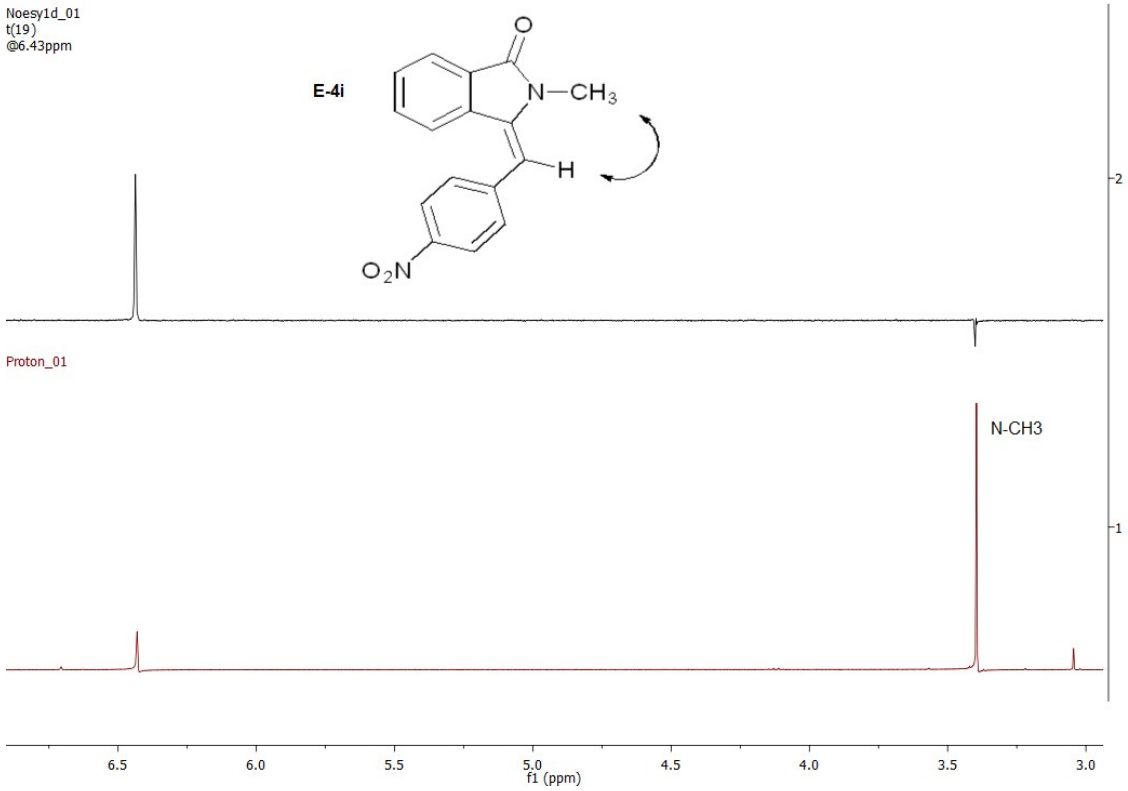

Noesy1d_01
N–Me
H
E-4h
H
O
2
Proton_Minsw_01
A (s)
6.46
B (s)
3.39
1
6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0
f1 (ppm)

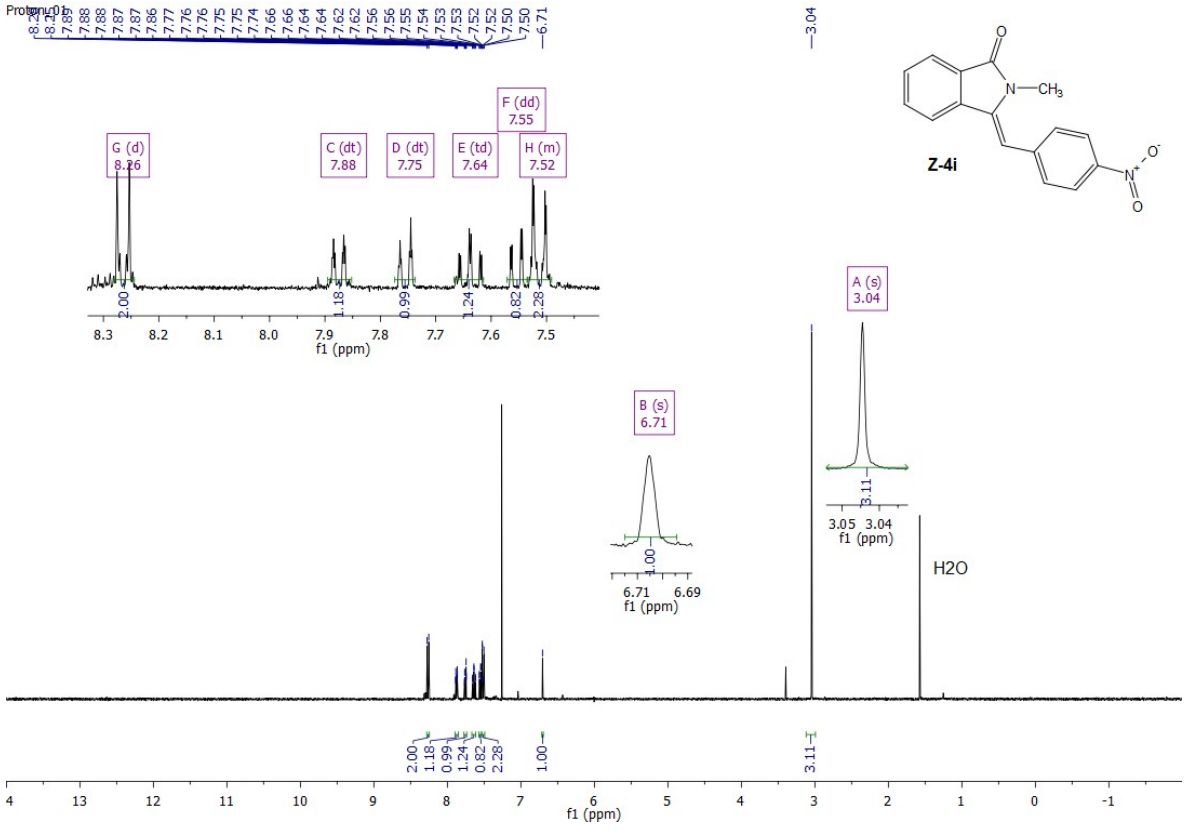

Proton_Minsw_01
A (s) 10.04
B (m) 7.91
C (dt) 7.86
D (dt) 7.75
E (td) 7.62
F (m) 7.52
G (s) 6.73
Z-4h
f1 (ppm)

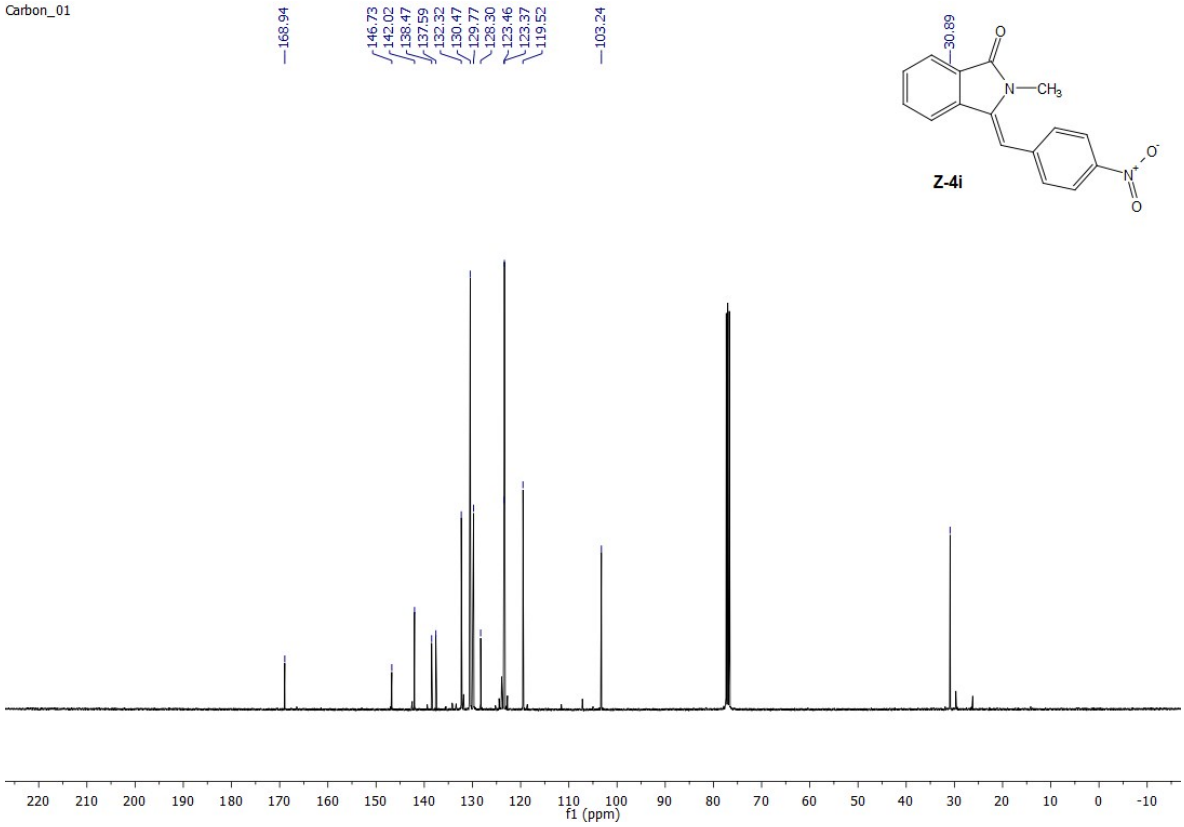

Carbon_01
Z-4h
f1 (ppm)

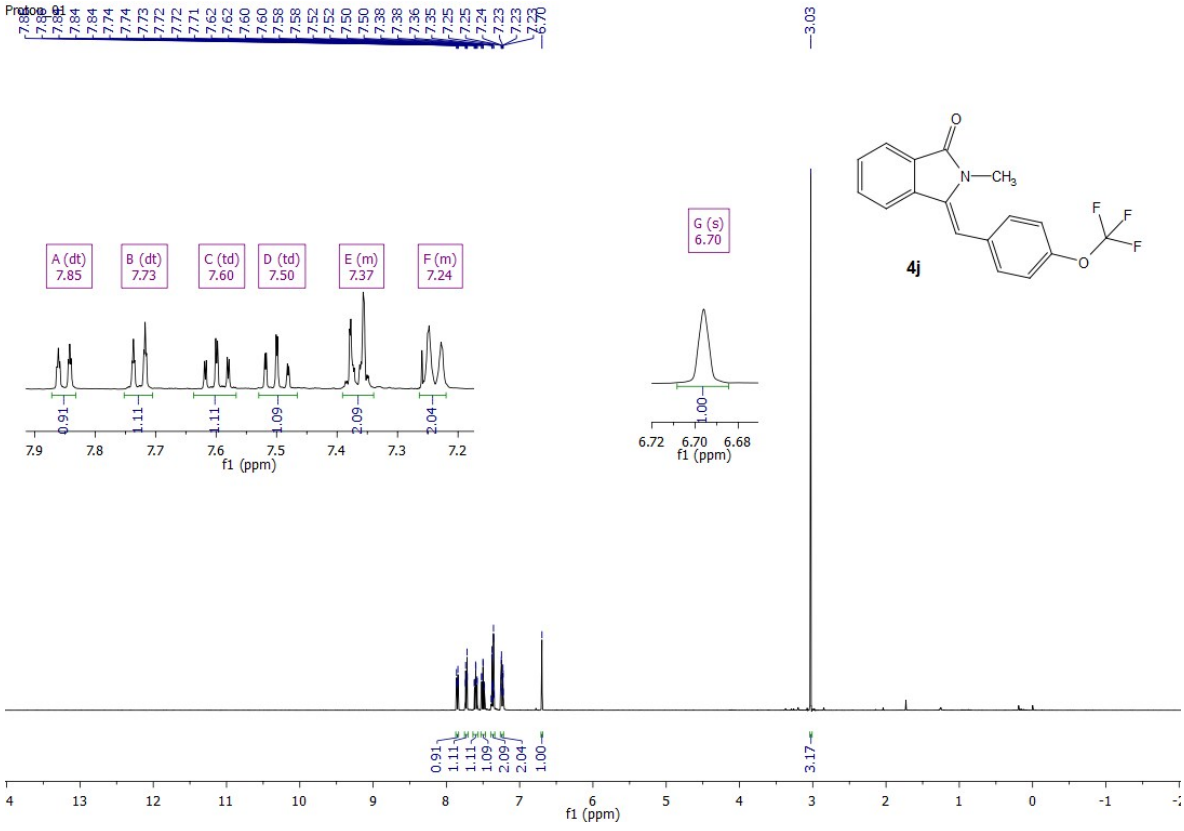

Noesy1d_01
Z-4h
O
N−Me
H(d)
H
H
O
H
Proton_Minsw_01
2
1
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4
f1 (ppm)

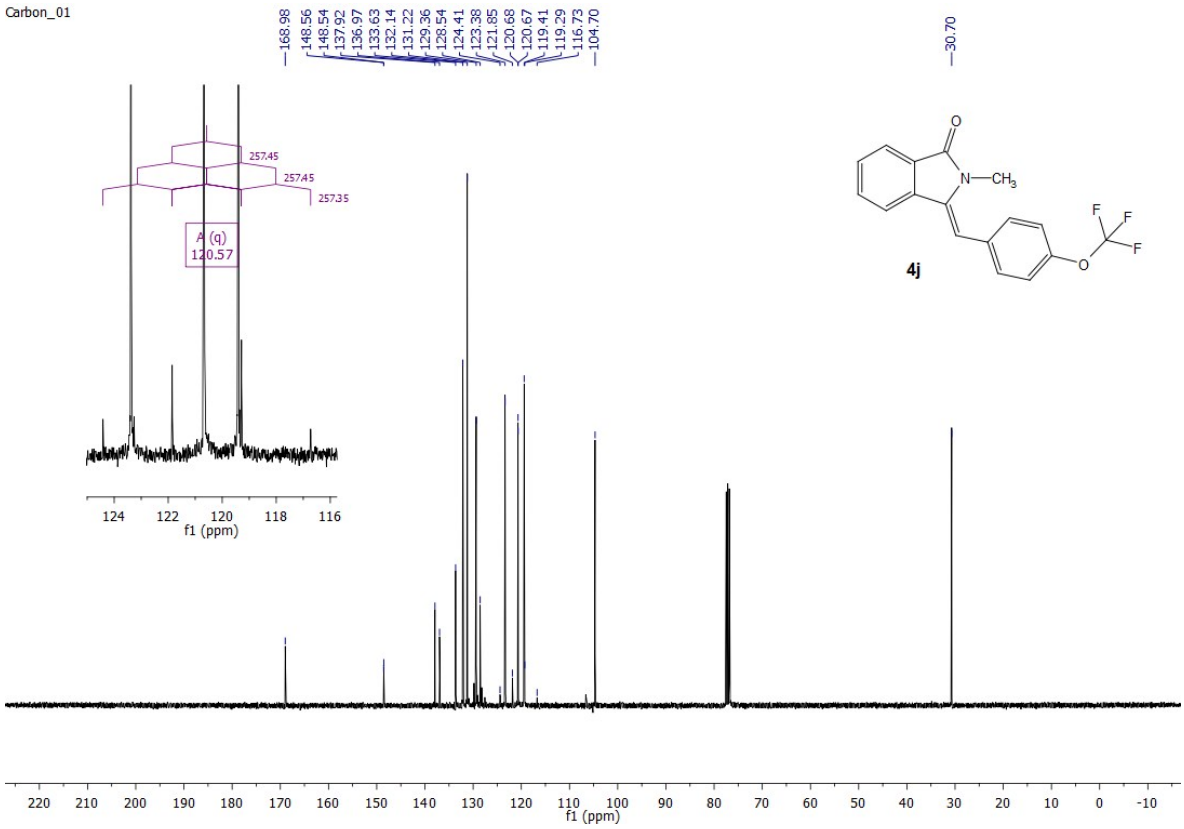

Proton_01
A (d) 8.30
B (dd) 7.86
C (dd) 7.65
D (m) 7.48
E (m) 7.35
F (s) 6.43
G (s) 3.40
1.95
1.00
2.02
1.11
2.02
1.00
2.98
f1 (ppm)
O
N—CH3
E-4i
O
N+
O-

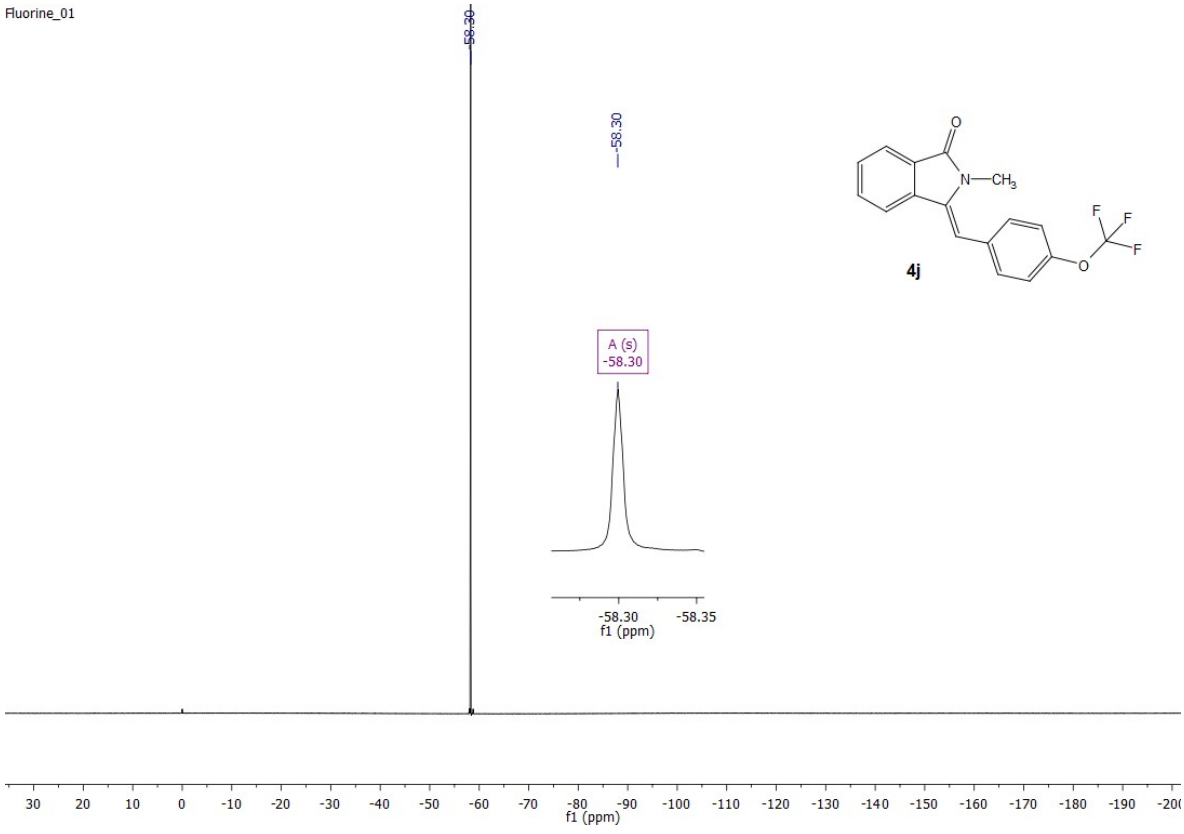

Noesy1d_01
t(19)
@6.43ppm
E-4i
O
N—CH3
H
O2N
2
Proton_01
N-CH3
1
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
f1 (ppm)

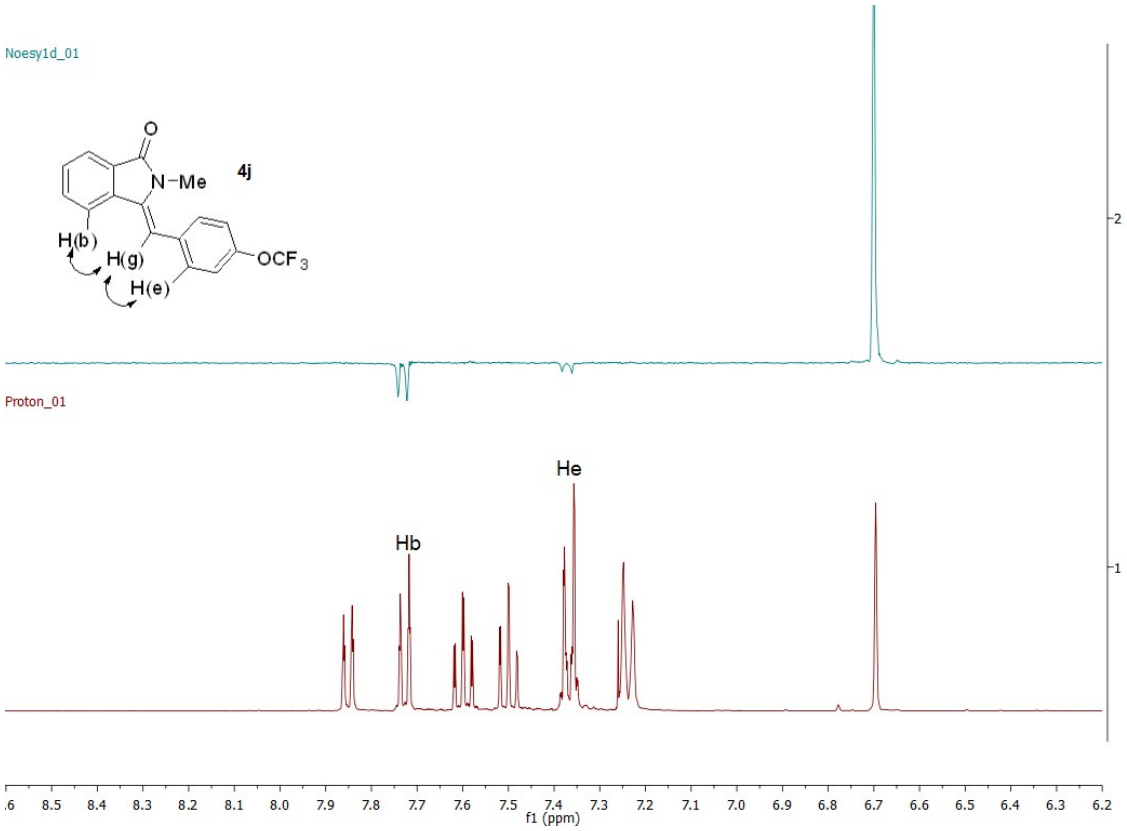

Proton_01
3.04
6.71
F (dd)
7.55
G (d)
8.26
C (dt)
7.88
D (dt)
7.75
E (td)
7.64
H (m)
7.52
Z-4i
A (s)
3.04
B (s)
6.71
H2O
2.00
1.18
0.99
1.24
0.82
2.28
1.00
3.11
f1 (ppm)

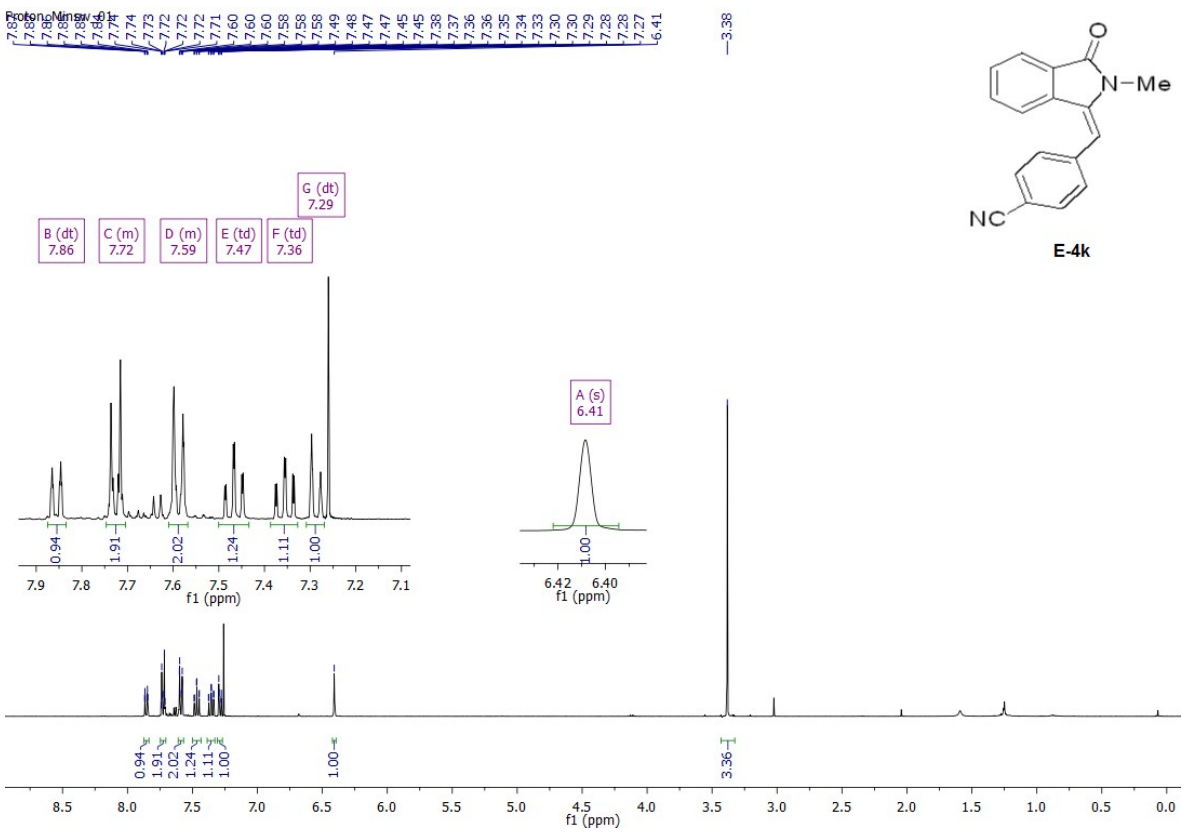

Carbon_01
168.94
146.73
142.02
138.47
137.59
132.32
130.47
129.77
128.30
123.46
123.37
119.52
103.24
30.89
Z-4i
f1 (ppm)

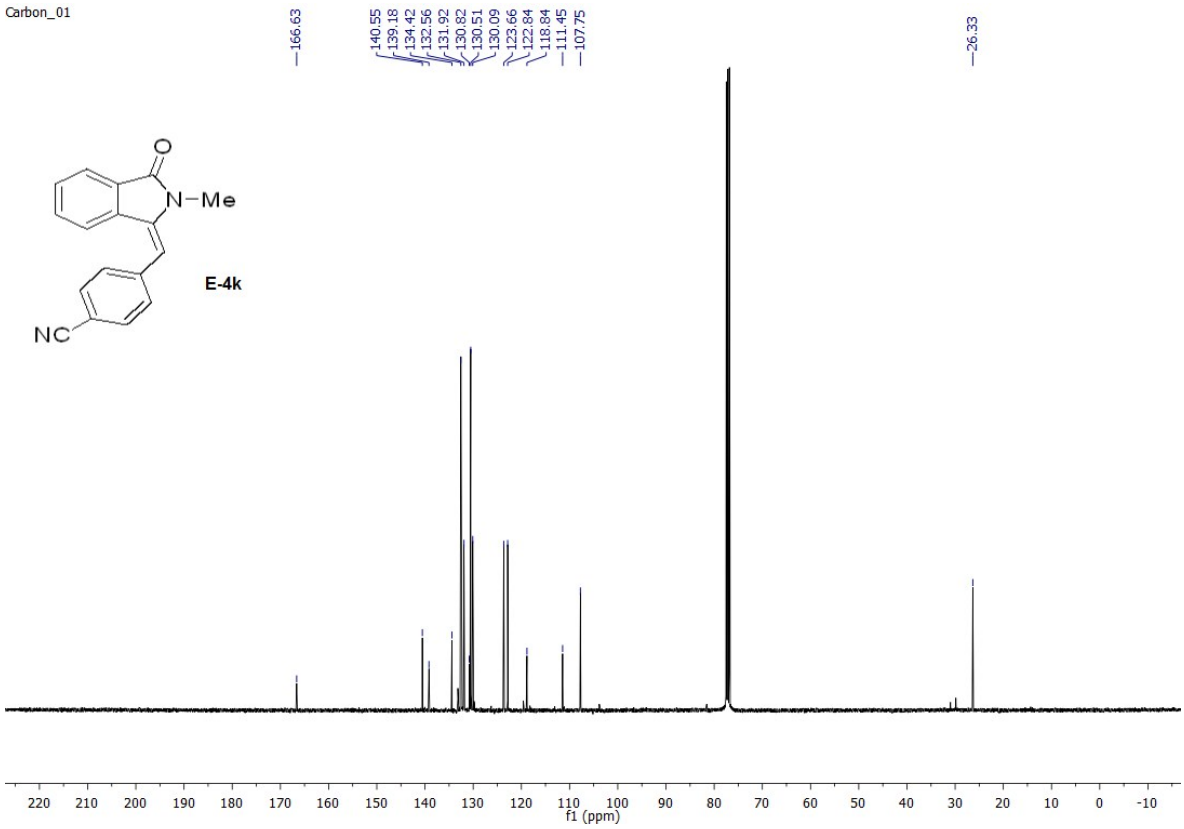

Proton_01
7.85
7.84
7.74
7.73
7.72
7.71
7.62
7.60
7.58
7.52
7.50
7.38
7.36
7.35
7.25
7.24
7.23
6.70
3.03
A (dt) 7.85
B (dt) 7.73
C (td) 7.60
D (td) 7.50
E (m) 7.37
F (m) 7.24
G (s) 6.70
4j
f1 (ppm)

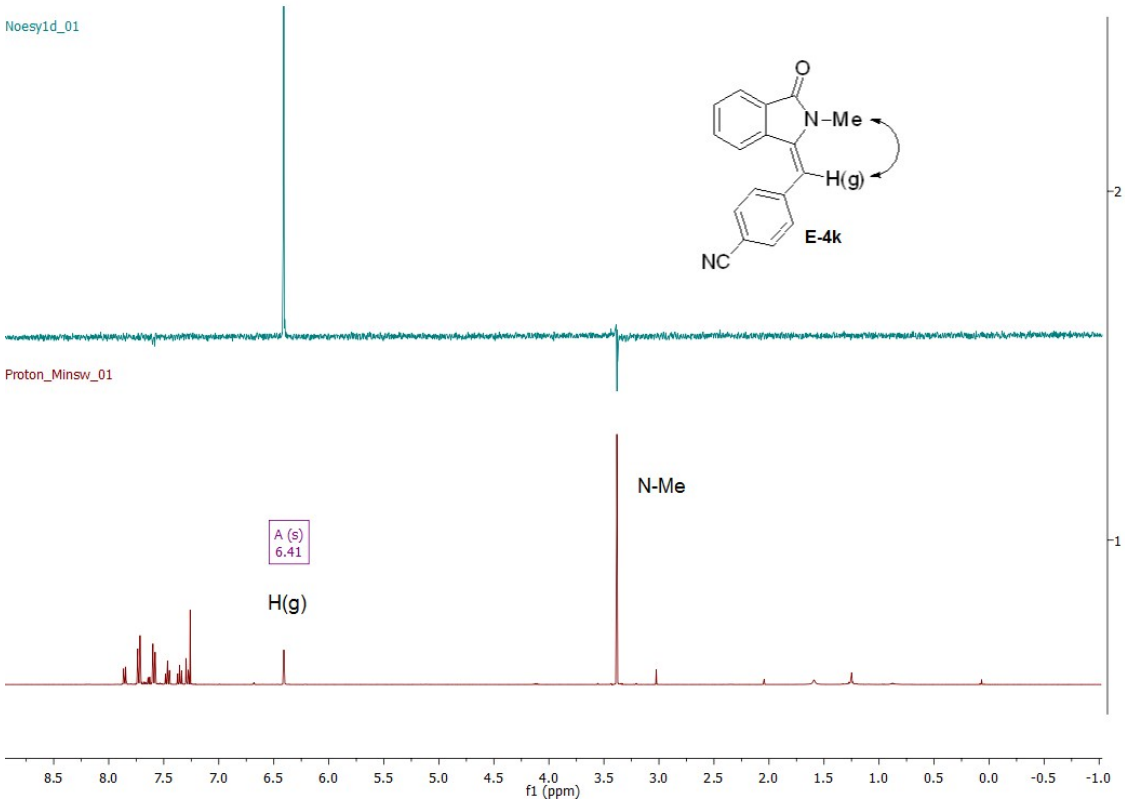

Carbon_01
168.98
148.56
148.54
137.92
136.97
133.63
132.14
131.22
129.36
128.54
124.41
123.38
121.85
120.68
120.67
119.41
119.29
116.73
104.70
30.70
257.45
257.45
257.35
A (q)
120.57
124 122 120 118 116
f1 (ppm)
O
N—CH3
F F F
O
4j
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

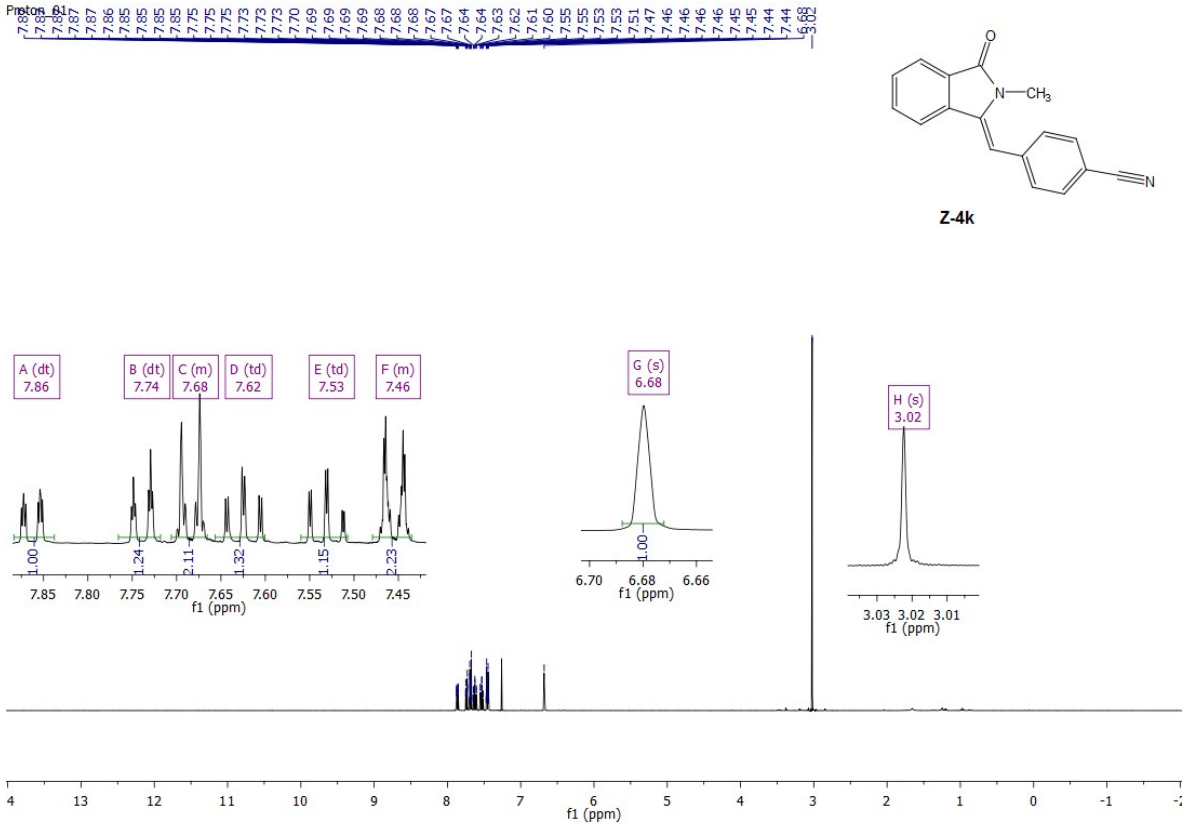

Fluorine_01
58.30
58.30
A (s)
-58.30
-58.30 -58.35
f1 (ppm)
O
N—CH3
F F F
O
4j
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)


Noesy1d_01
4j
N–Me
H(b)
H(g)
H(e)
OCF3
Proton_01
He
Hb
f1 (ppm)

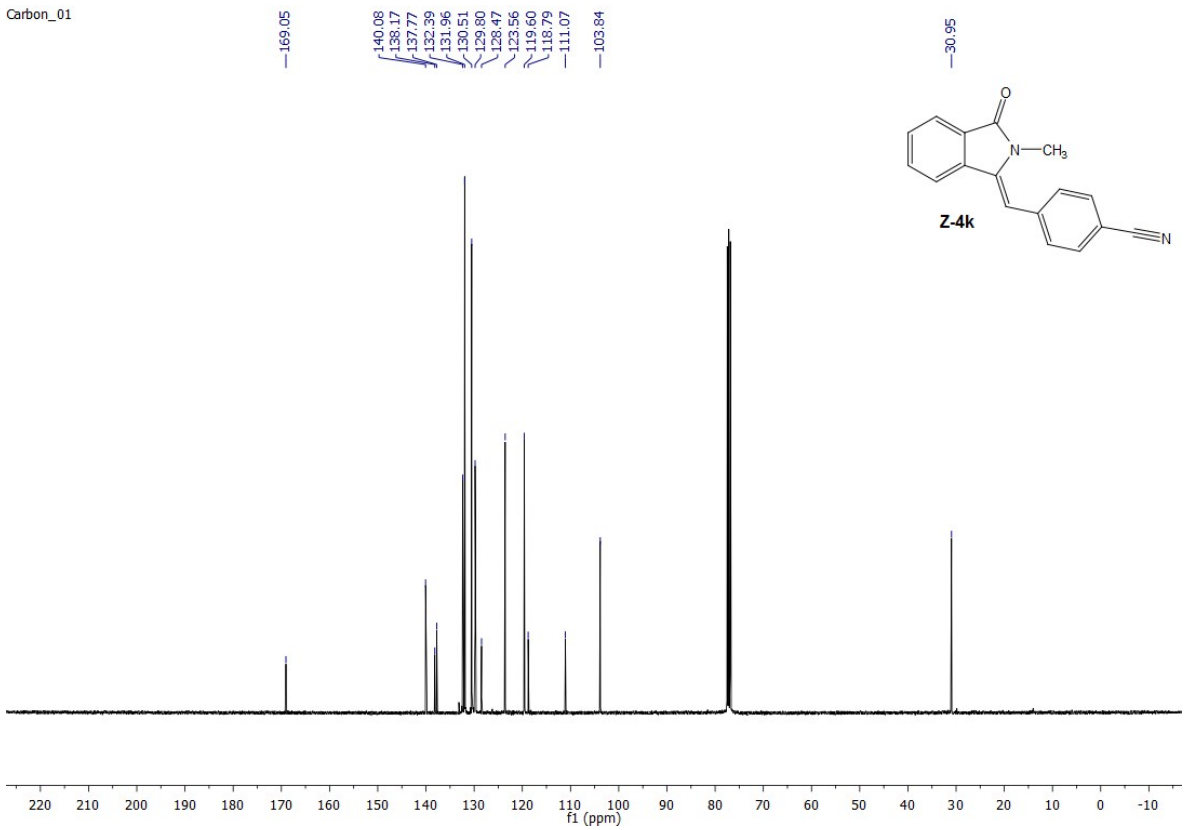

Proton_Nmsu_01
N–Me
NC
E-4k
B (dt) 7.86
C (m) 7.72
D (m) 7.59
E (td) 7.47
F (td) 7.36
G (dt) 7.29
A (s) 6.41
f1 (ppm)


Carbon_01
166.63
140.55
139.18
134.42
132.56
131.92
130.82
130.51
130.09
123.66
122.84
118.84
111.45
107.75
26.33
N–Me
E-4k
NC
f1 (ppm)

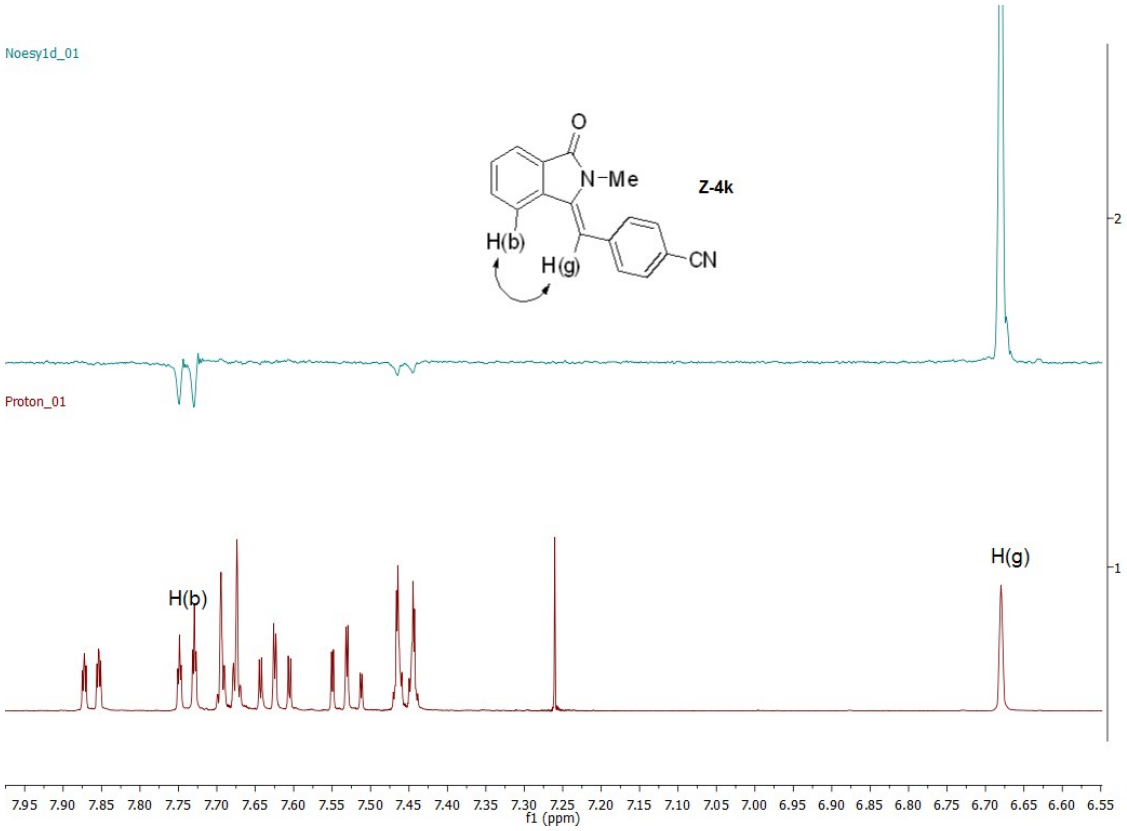

Noesy1d_01
N–Me
H(g)
E-4k
NC
Proton_Minsw_01
N-Me
A (s)
6.41
H(g)
f1 (ppm)

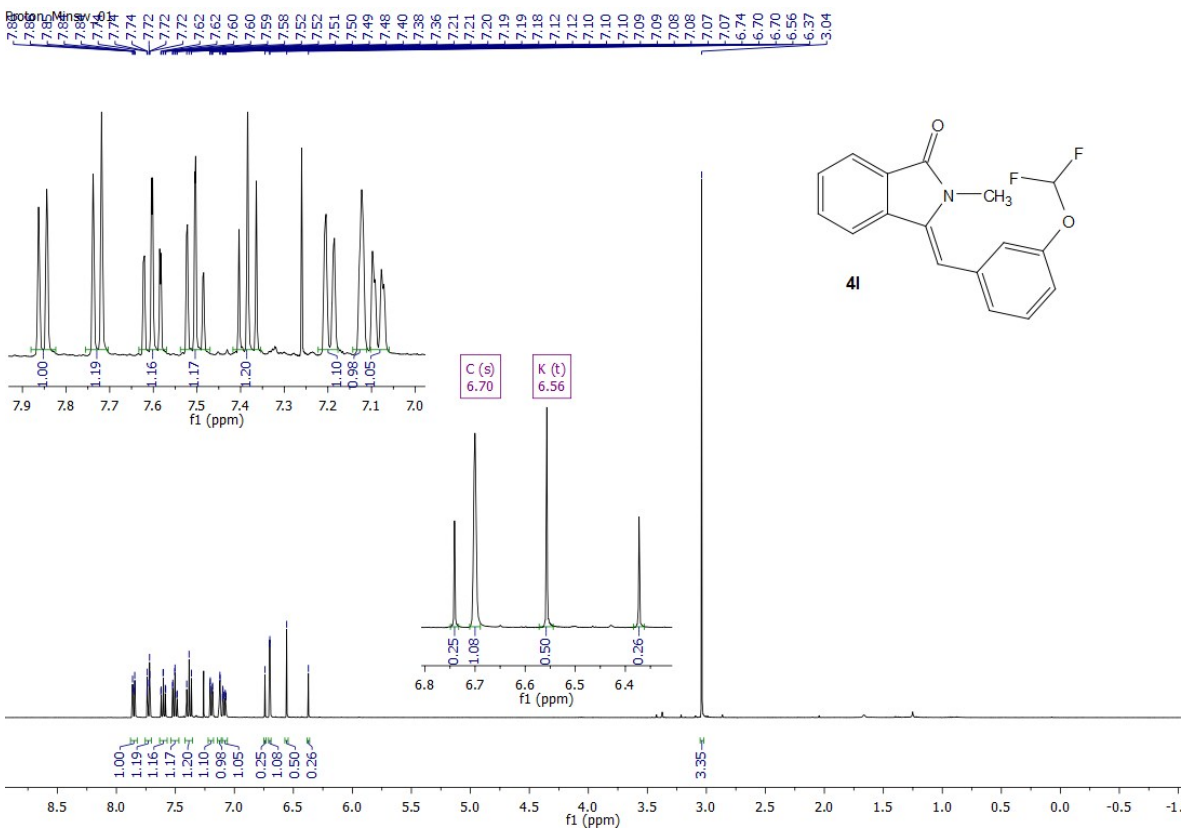

Proton_01
A (dt) 7.86
B (dt) 7.74
C (m) 7.68
D (td) 7.62
E (td) 7.53
F (m) 7.46
G (s) 6.68
H (s) 3.02
f1 (ppm)

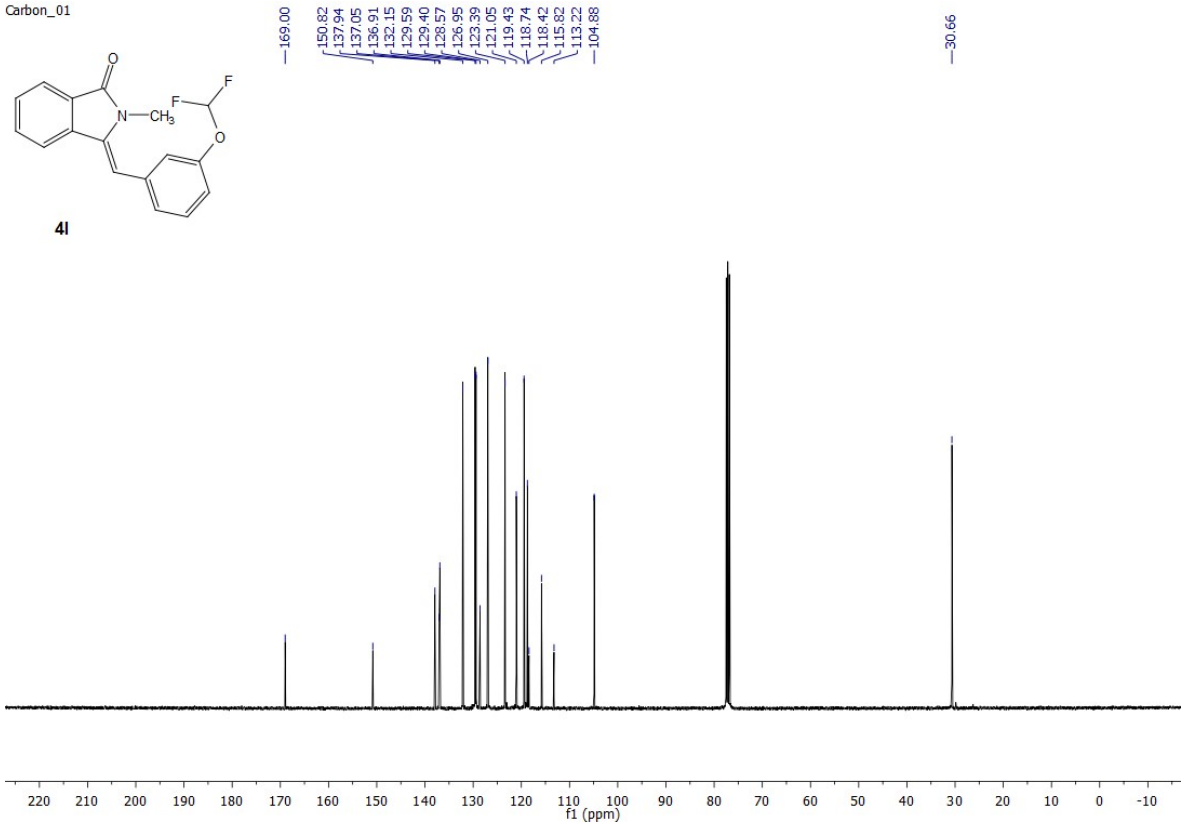
Z-4k

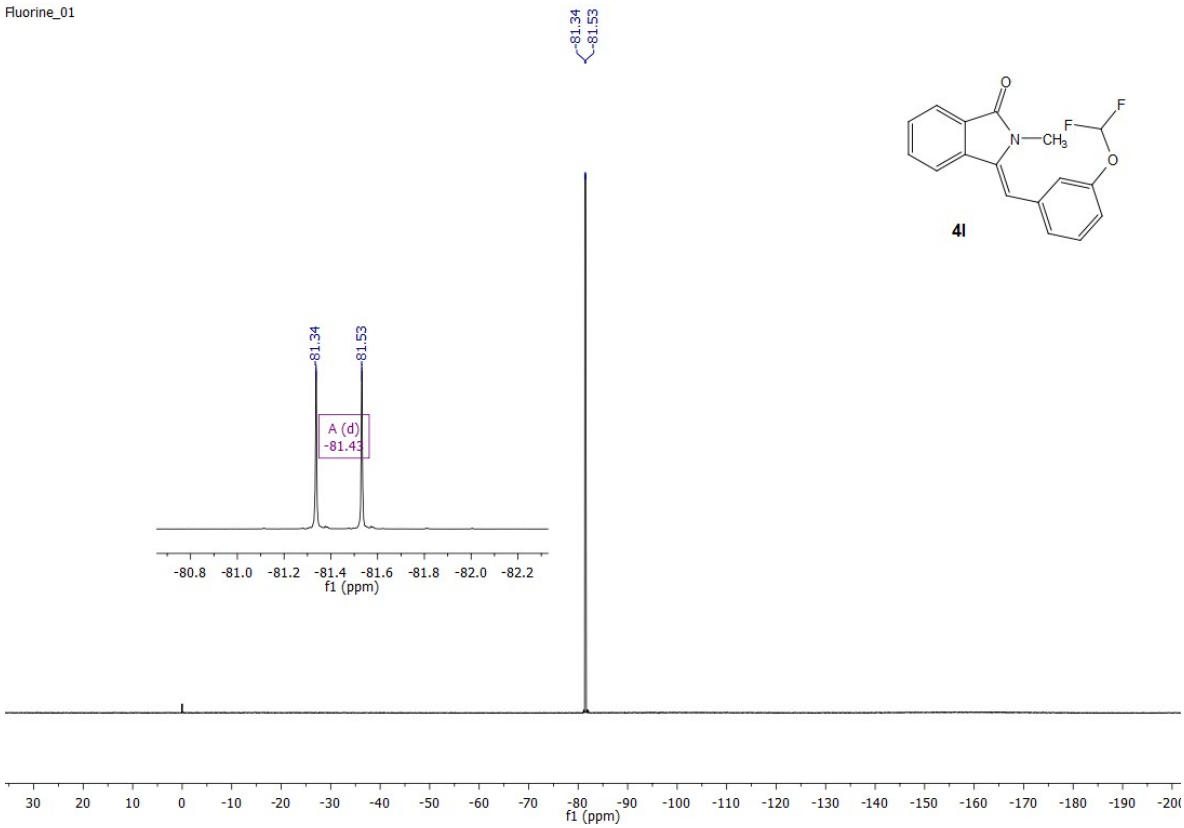

Carbon_01
169.05
140.08
138.17
137.77
132.39
131.96
130.51
129.80
128.47
123.56
119.60
118.79
111.07
103.84
30.95
f1 (ppm)

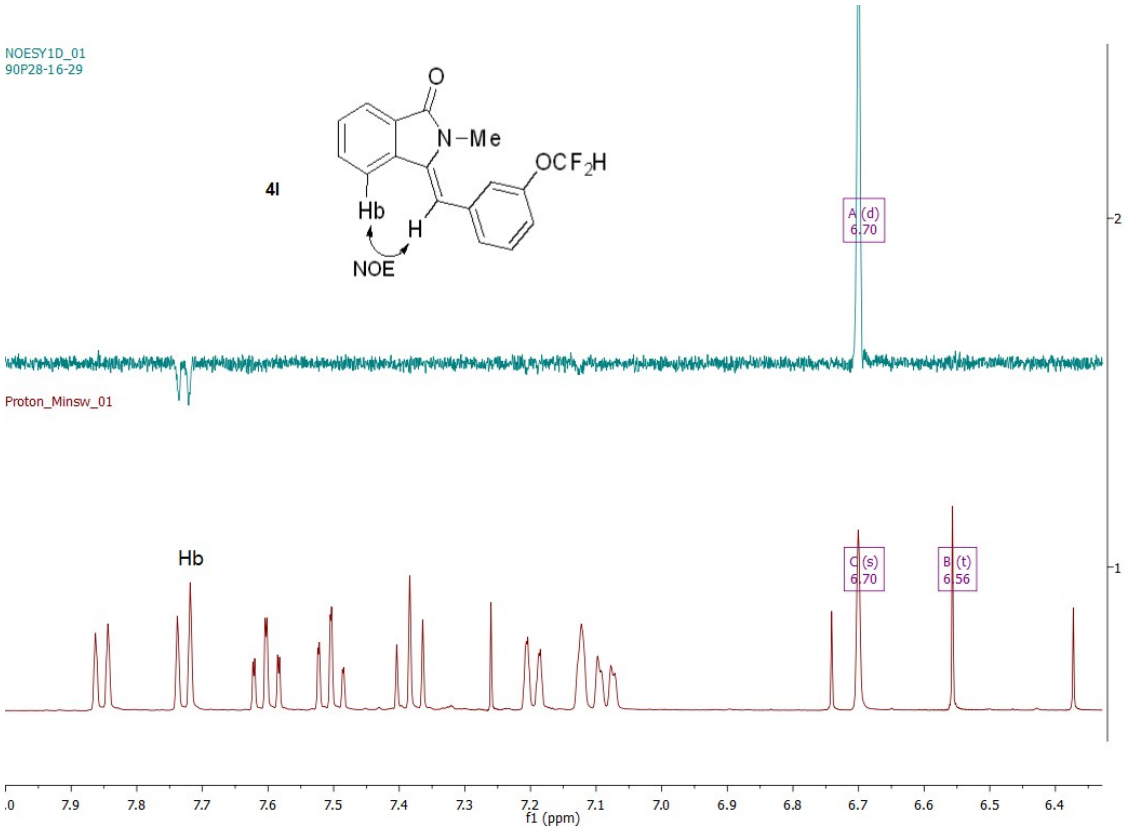
Z-4k

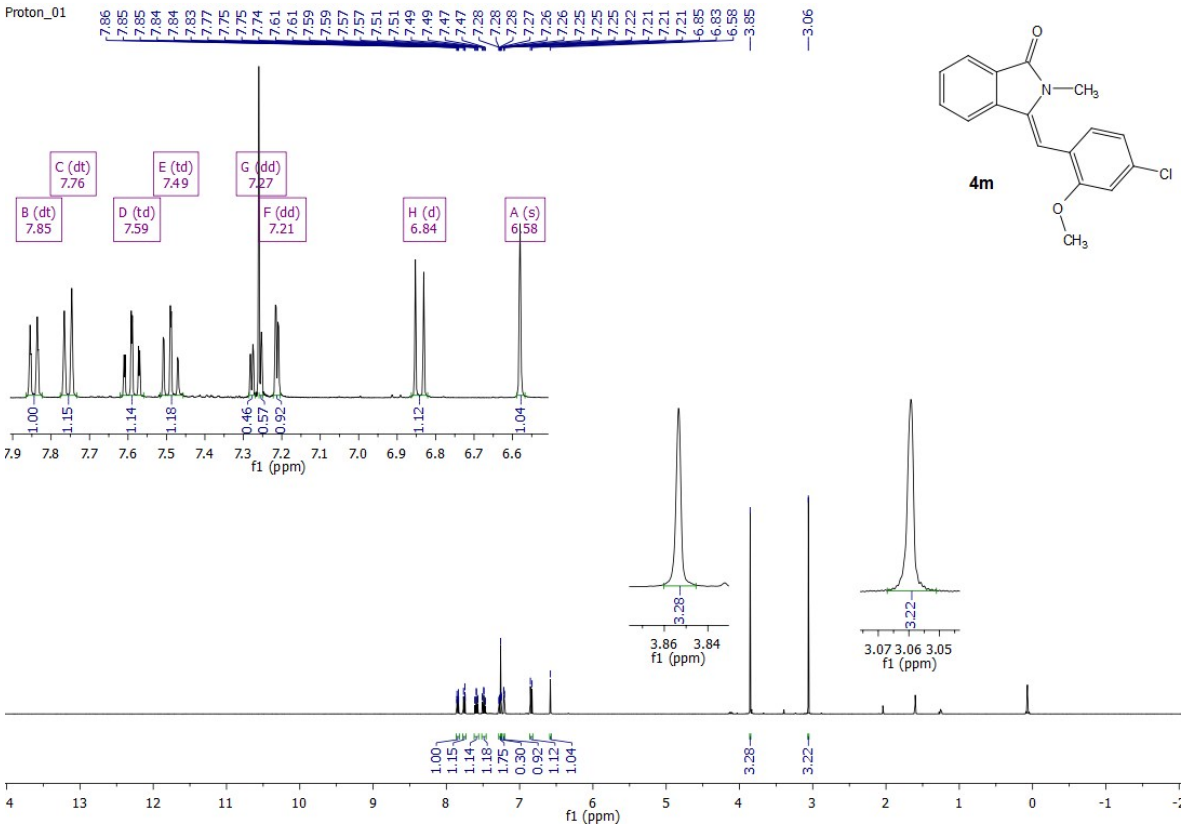

Noesy1d_01
O
N–Me
Z-4k
H(b)
H(g)
CN
Proton_01
H(b)
H(g)
2
1
7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60 6.55
f1 (ppm)

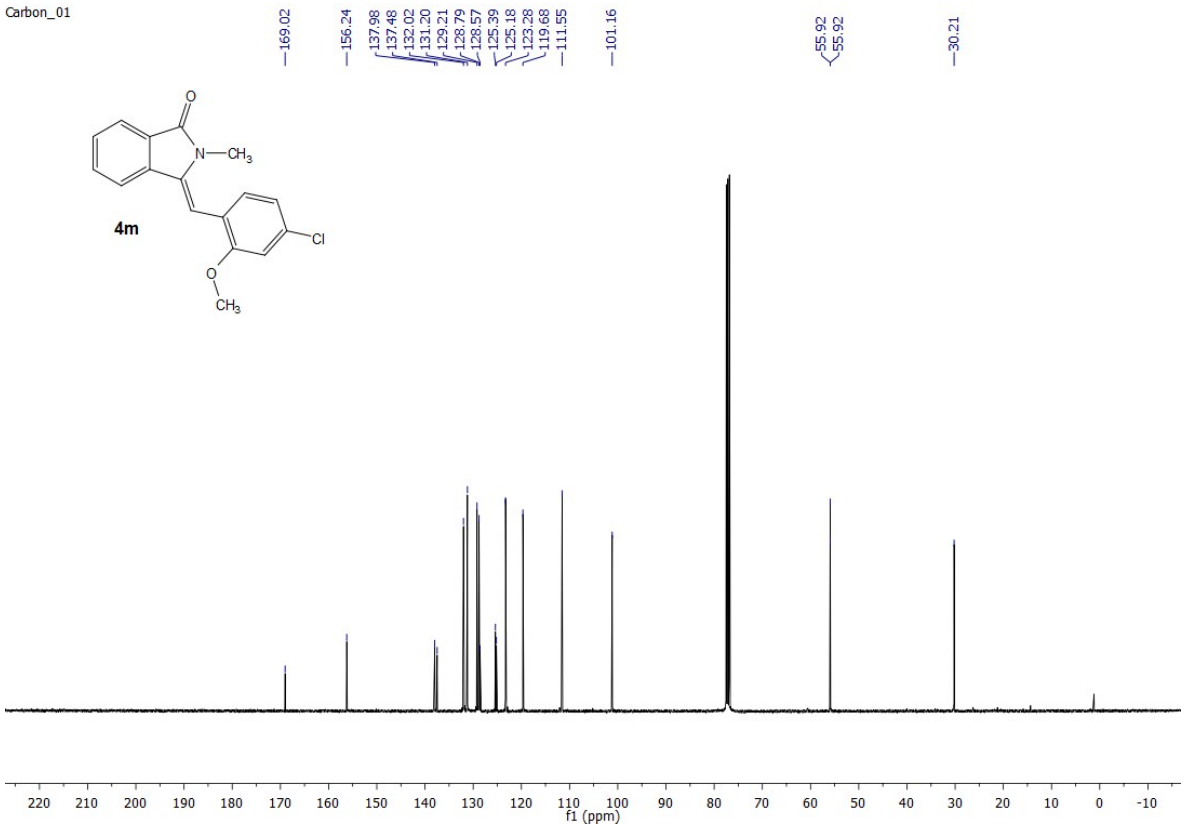

Proton_Minsw_01
7.88 7.86 7.85 7.84 7.74 7.74 7.74 7.72 7.72 7.72 7.62 7.62 7.60 7.60 7.59 7.58 7.52 7.52 7.51 7.50 7.49 7.48 7.40 7.38 7.36 7.21 7.21 7.20 7.19 7.19 7.18 7.12 7.12 7.10 7.10 7.10 7.09 7.09 7.08 7.08 7.07 7.07 6.74 6.70 6.70 6.56 6.37 3.04
O
N
CH3
F
F
O
4l
1.00 1.19 1.16 1.17 1.20 1.10 0.98 1.05
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
C (s)
6.70
K (t)
6.56
0.25 1.08 0.50 0.26
6.8 6.7 6.6 6.5 6.4
f1 (ppm)
1.00 1.19 1.16 1.17 1.20 1.10 0.98 1.05 0.25 1.08 0.50 0.26
3.35
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

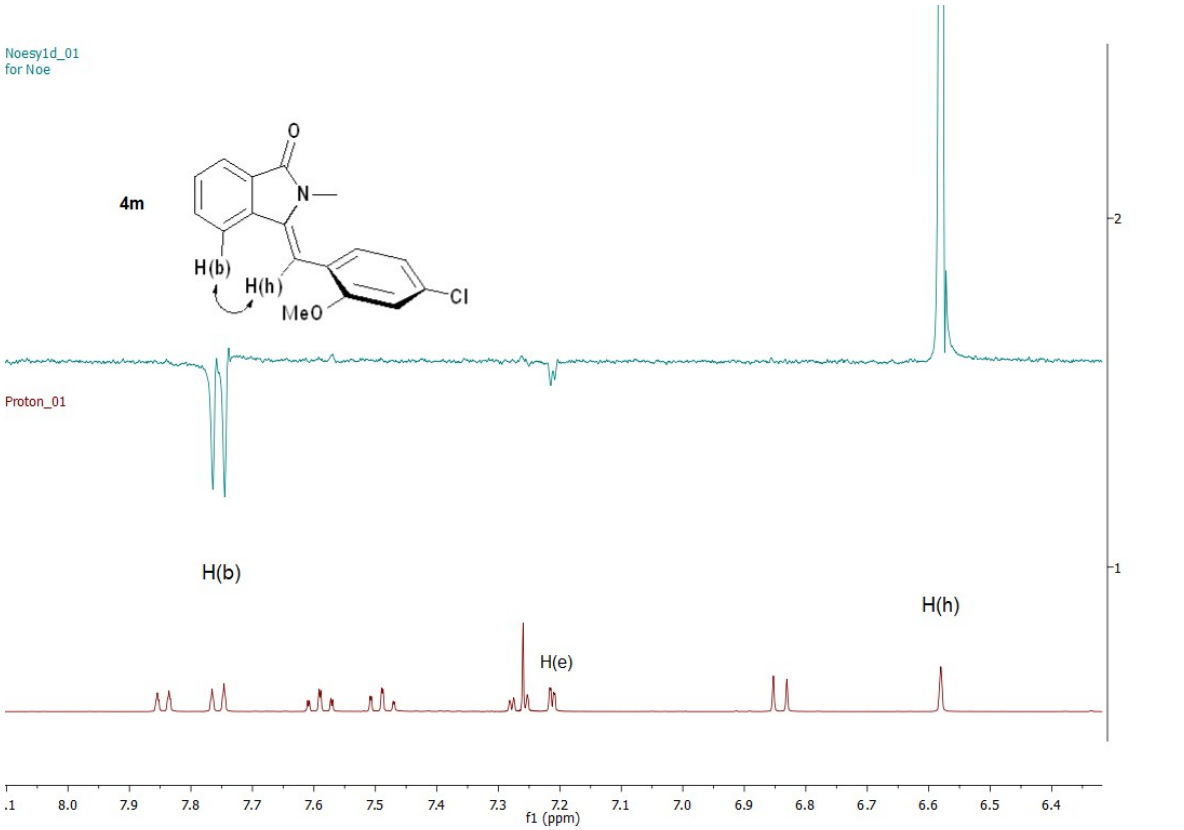

Carbon_01
169.00
150.82
137.94
137.05
136.91
132.15
129.59
129.40
128.57
126.95
123.39
121.05
119.43
118.74
118.42
115.82
113.22
104.88
30.66
O
N
CH3
F
F
O
4l
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

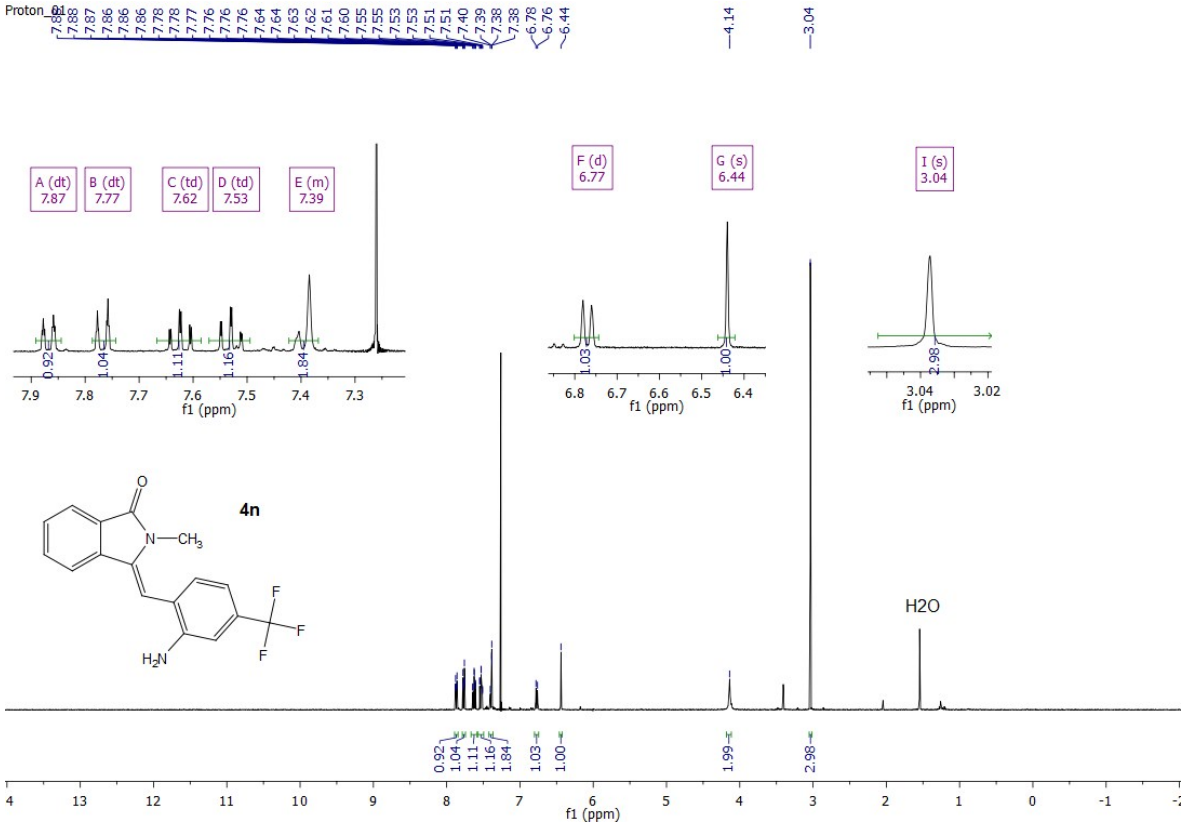

Fluorine_01
81.34
81.53
O
N
CH3
F
F
O
4l
81.34
81.53
A (d)
-81.43
-80.8 -81.0 -81.2 -81.4 -81.6 -81.8 -82.0 -82.2
f1 (ppm)
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

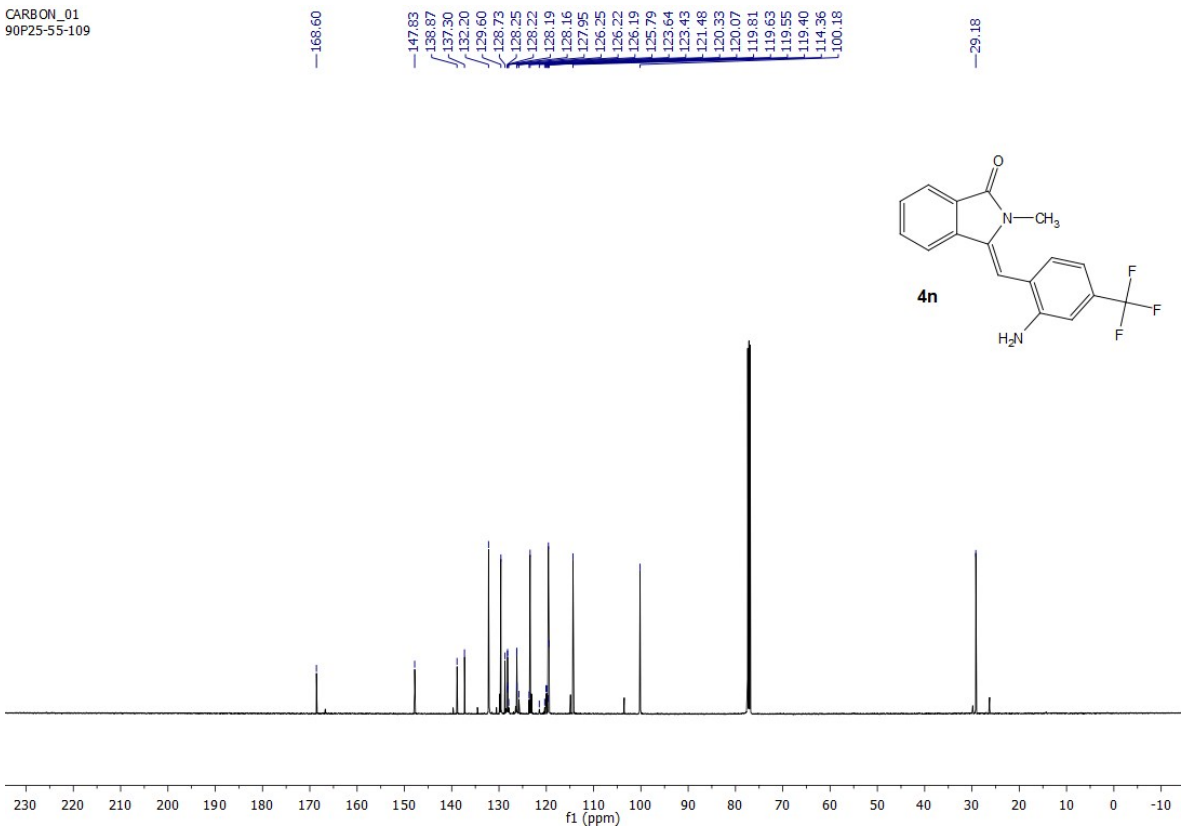

NOESY1D_01
90P28-16-29
4l
O
N–Me
OCF2H
Hb
H
NOE
A (d)
6.70
Proton_Minsw_01
Hb
C (s)
6.70
B (t)
6.56
f1 (ppm)

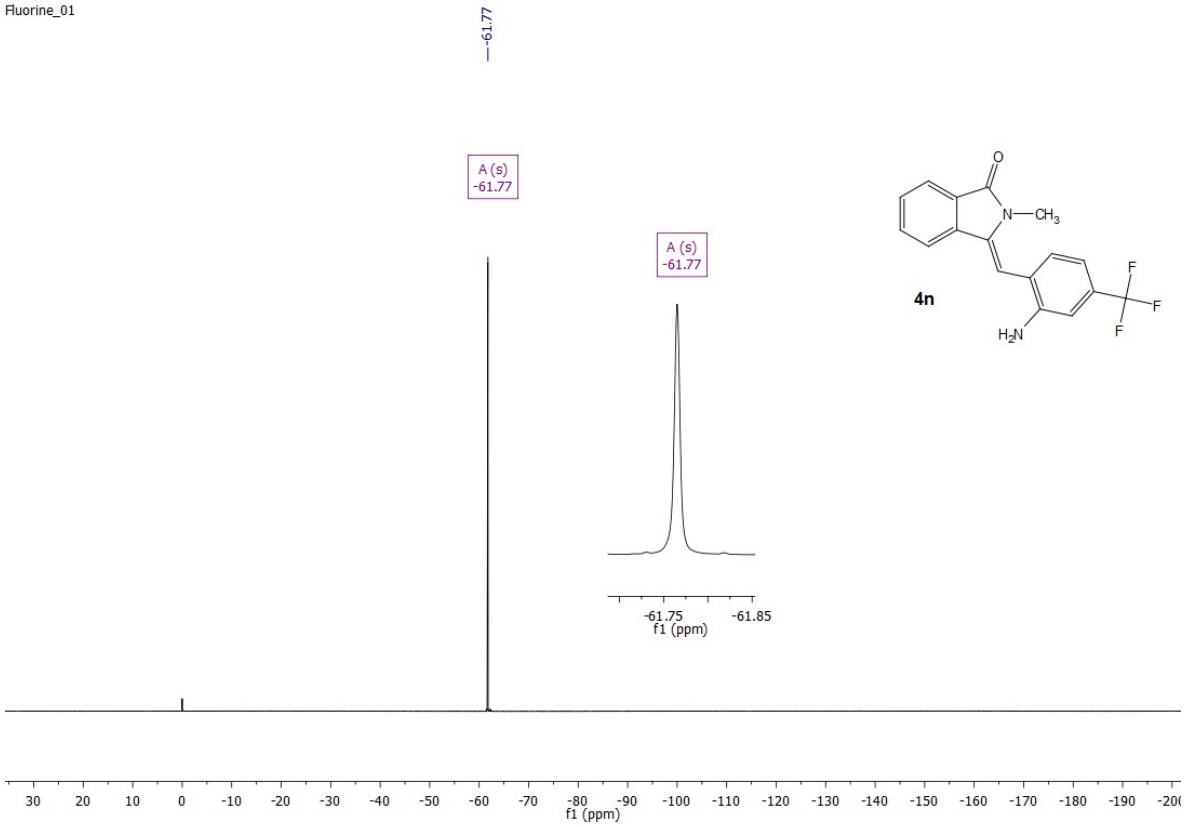

Proton_01
C (dt)
7.76
E (td)
7.49
G (dd)
7.27
B (dt)
7.85
D (td)
7.59
F (dd)
7.21
H (d)
6.84
A (s)
6.58
4m
O
N—CH3
Cl
O
CH3
3.28
3.22
f1 (ppm)

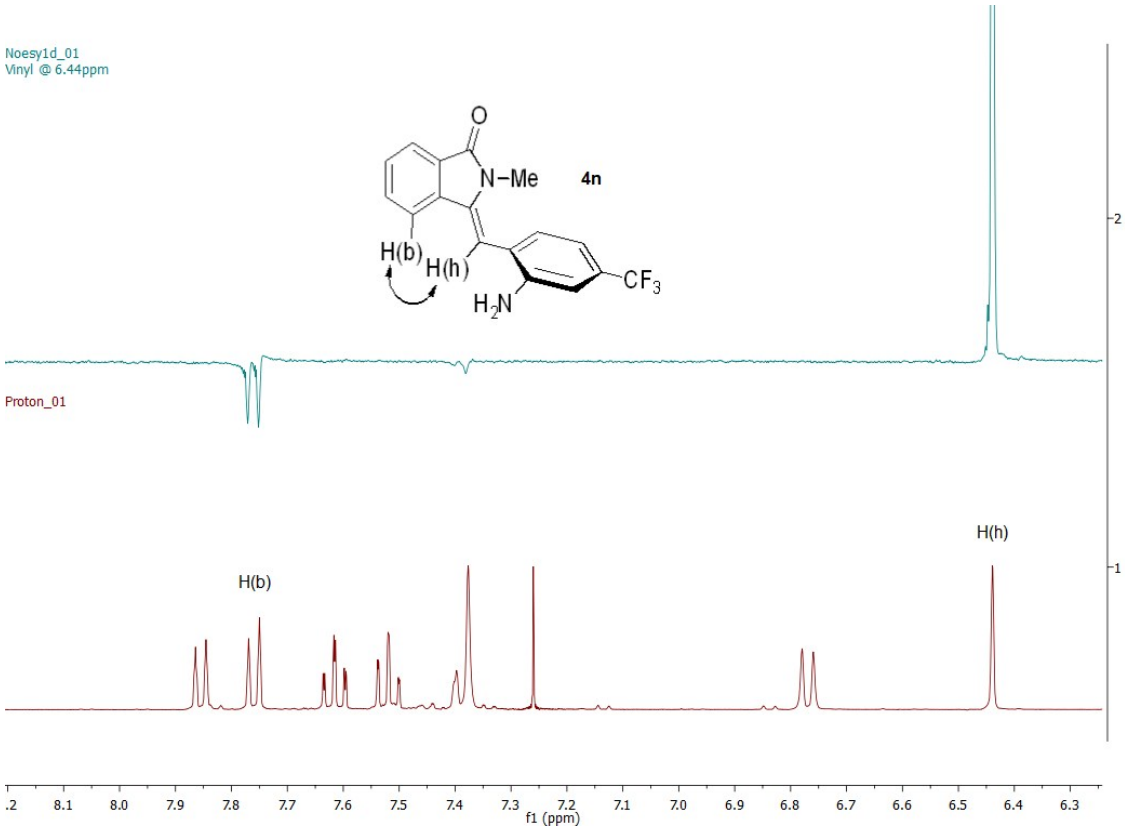

Carbon_01
169.02
156.24
137.98
137.48
132.02
131.20
129.21
128.79
128.57
125.39
125.18
123.28
119.68
111.55
101.16
55.92
55.92
30.21
4m
f1 (ppm)

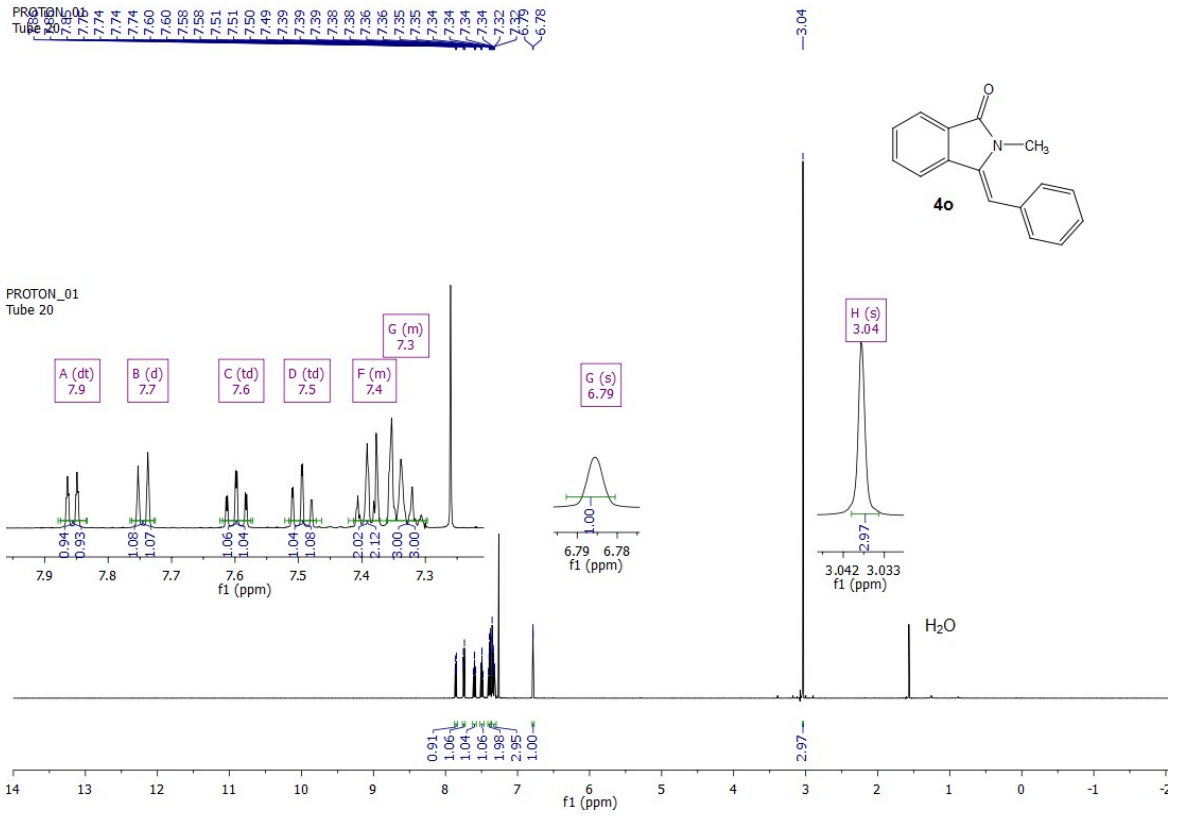

Noesy1d_01
for Noe
4m
H(b)
H(h)
MeO
Cl
Proton_01
H(b)
H(h)
H(e)
f1 (ppm)

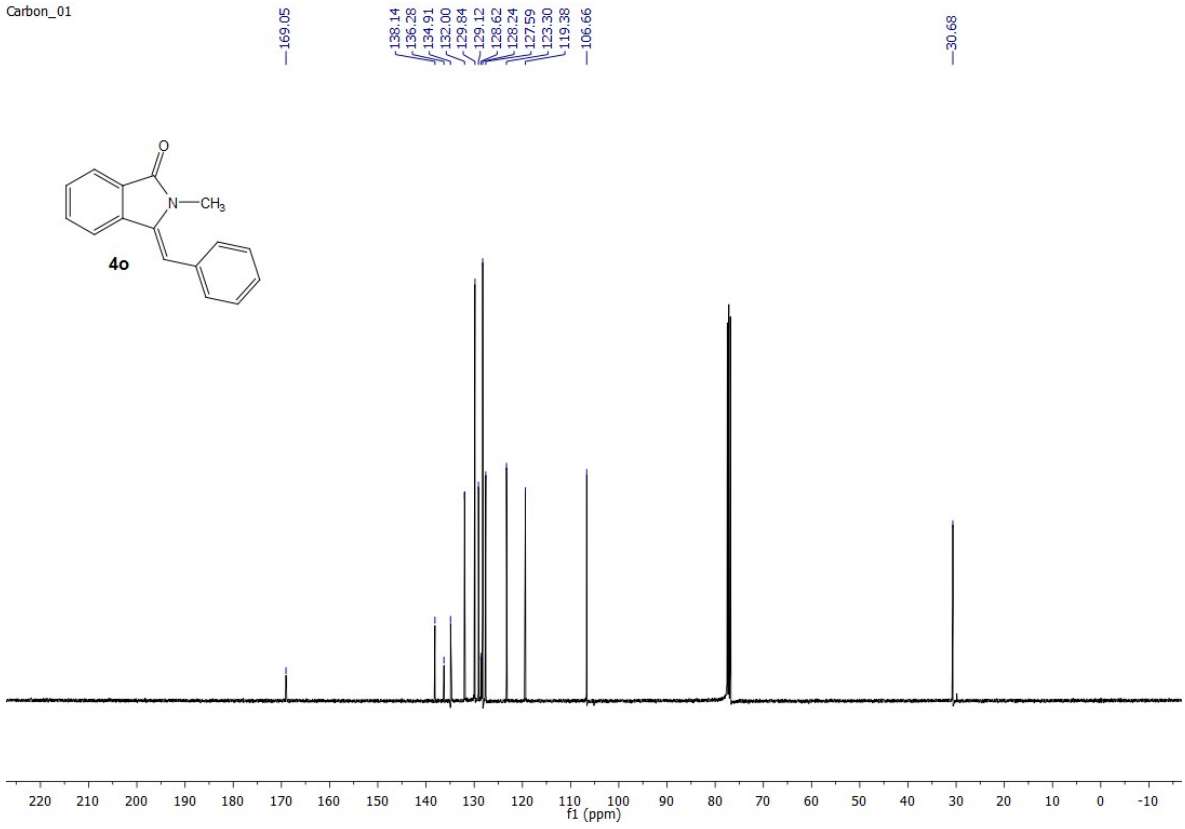

Proton_01
A (dt) 7.87
B (dt) 7.77
C (td) 7.62
D (td) 7.53
E (m) 7.39
F (d) 6.77
G (s) 6.44
I (s) 3.04
4n
H2O
f1 (ppm)

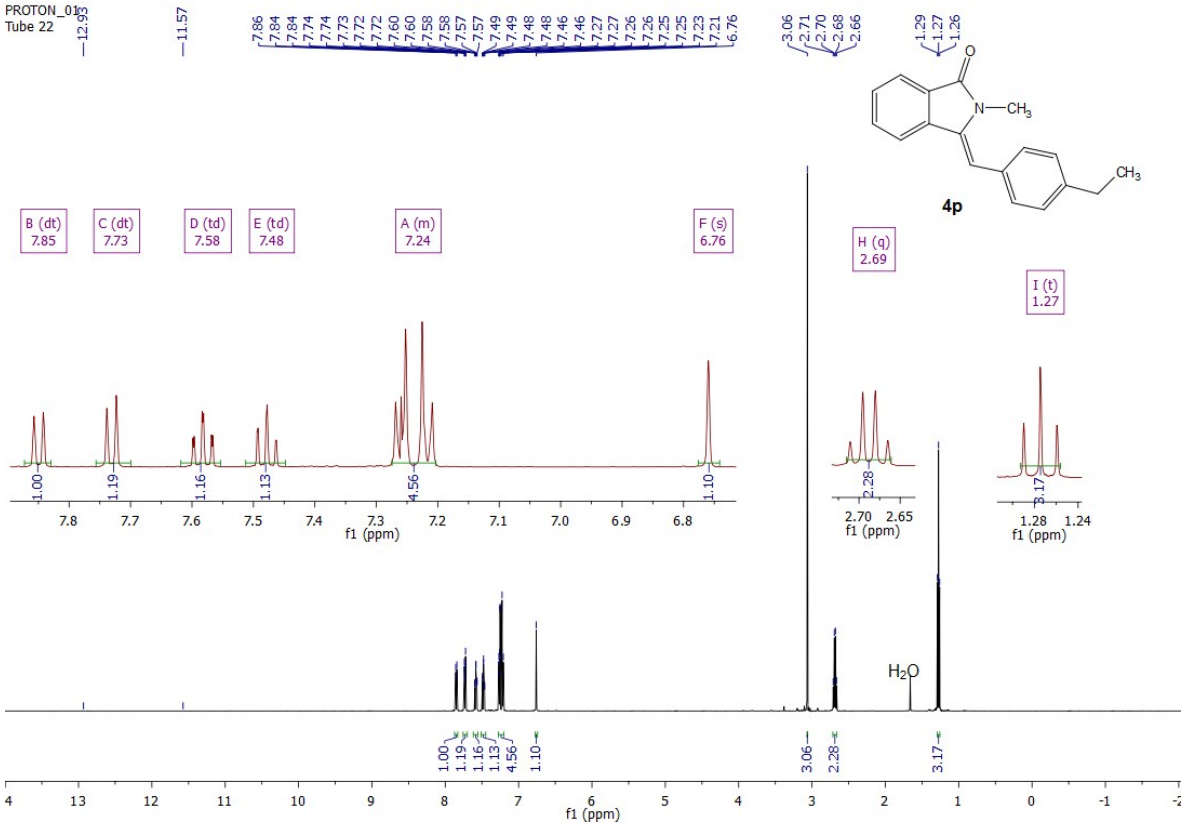

CARBON_01
90P25-55-109
4n
f1 (ppm)

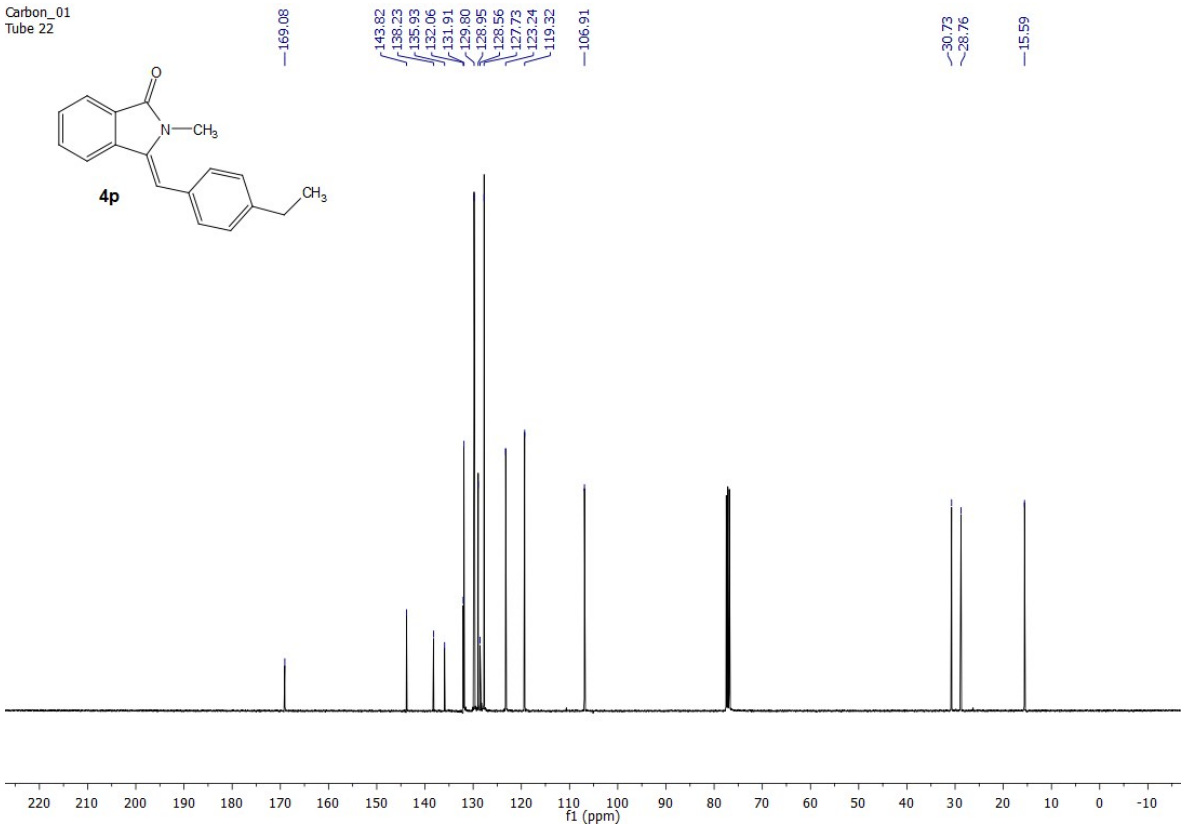

Fluorine_01
-61.77
A (s)
-61.77
A (s)
-61.77
-61.75
-61.85
f1 (ppm)
4n
f1 (ppm)

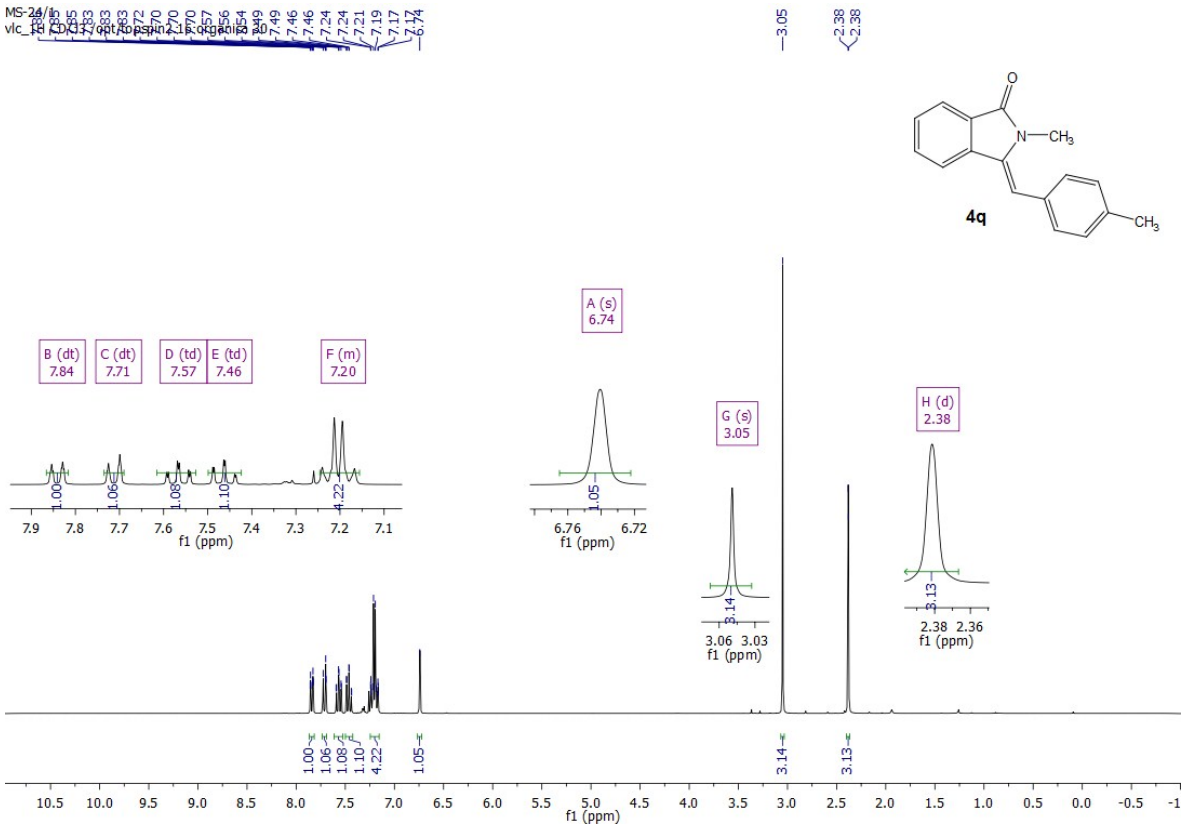

Noesy1d_01
Vinyl @ 6.44ppm
N–Me
4n
H(b)
H(h)
CF3
H2N
Proton_01
H(h)
H(b)
2
1
f1 (ppm)

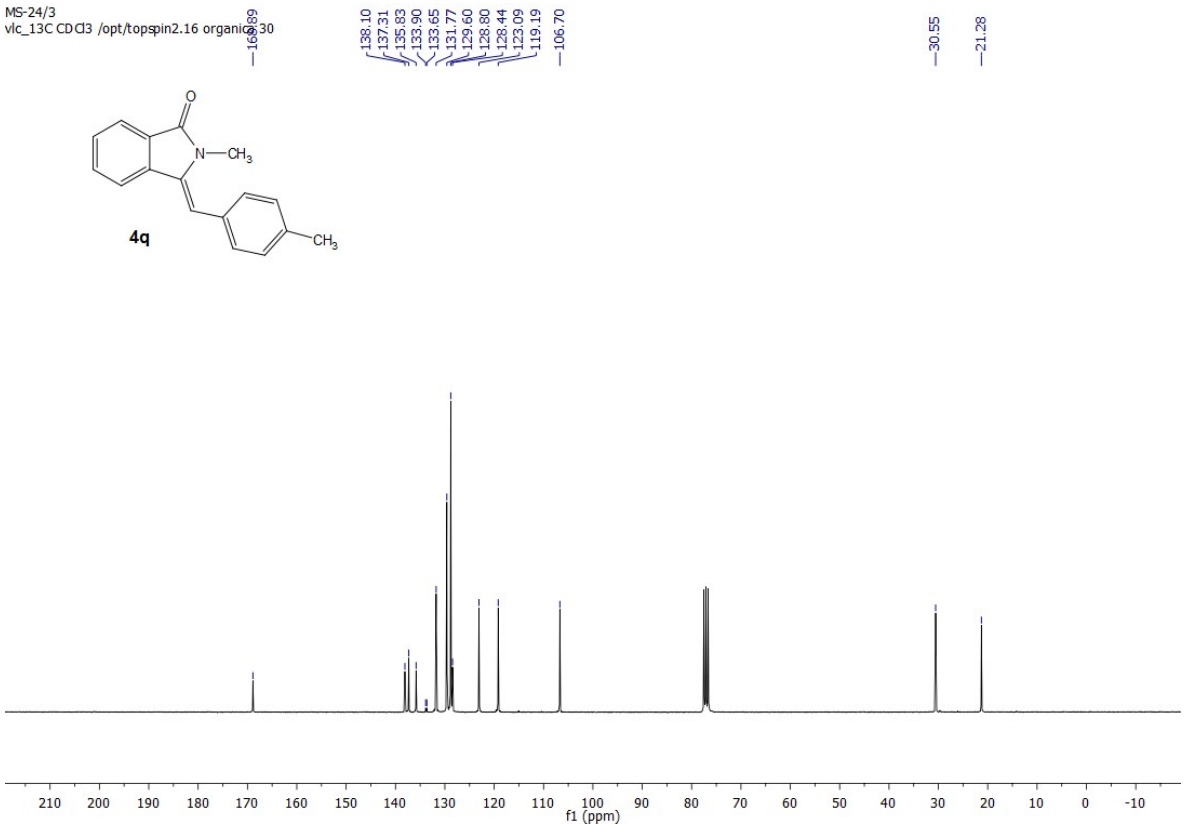

PROTON_01
Tube 20
4o
A (dt) 7.9
B (d) 7.7
C (td) 7.6
D (td) 7.5
F (m) 7.4
G (m) 7.3
G (s) 6.79
H (s) 3.04
H2O
f1 (ppm)

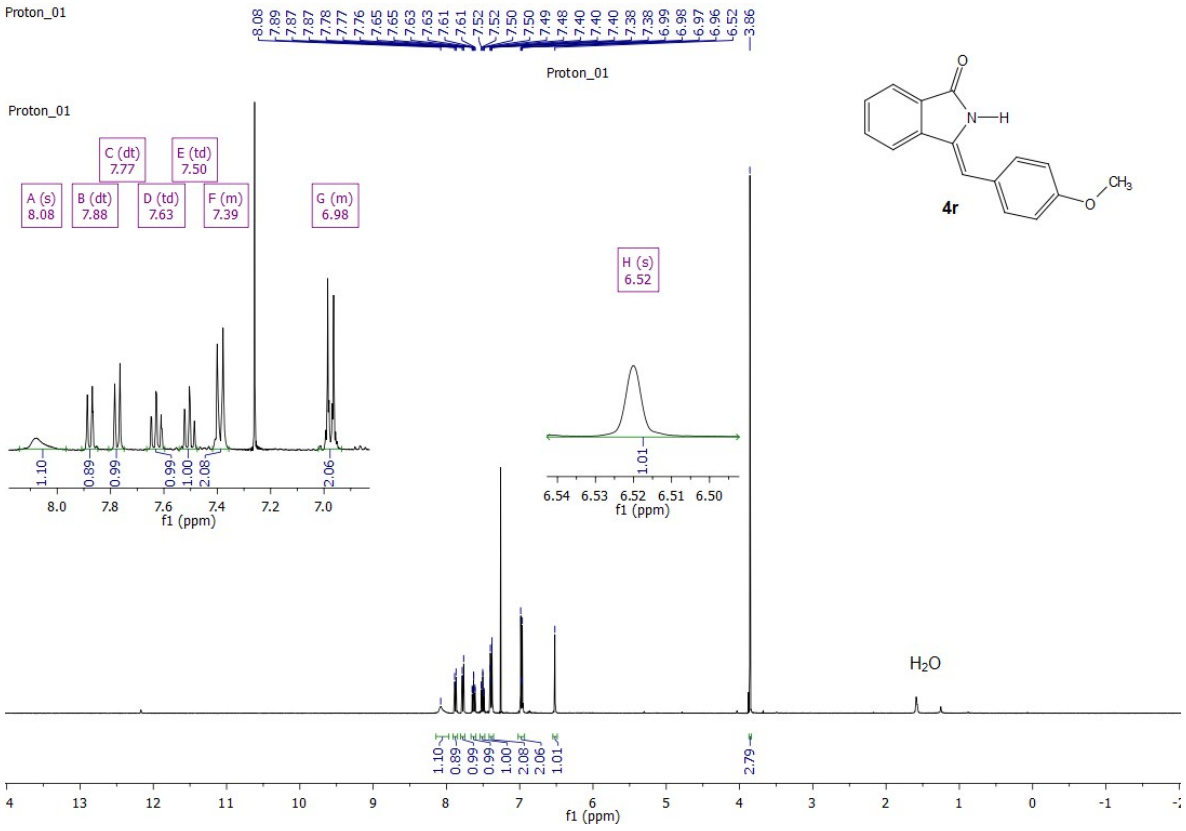

Carbon_01
4o
f1 (ppm)

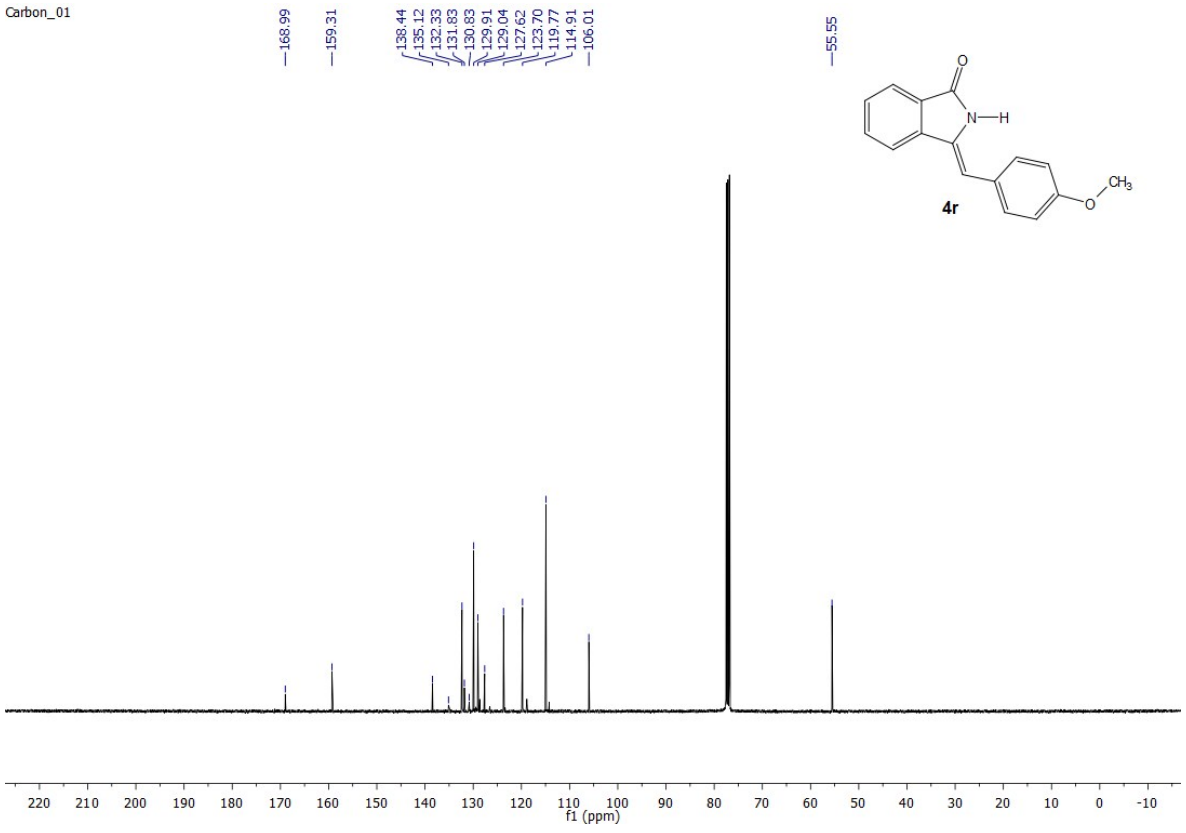

PROTON_01
Tube 22
4p
B (dt) 7.85
C (dt) 7.73
D (td) 7.58
E (td) 7.48
A (m) 7.24
F (s) 6.76
H (q) 2.69
I (t) 1.27
H2O
f1 (ppm)

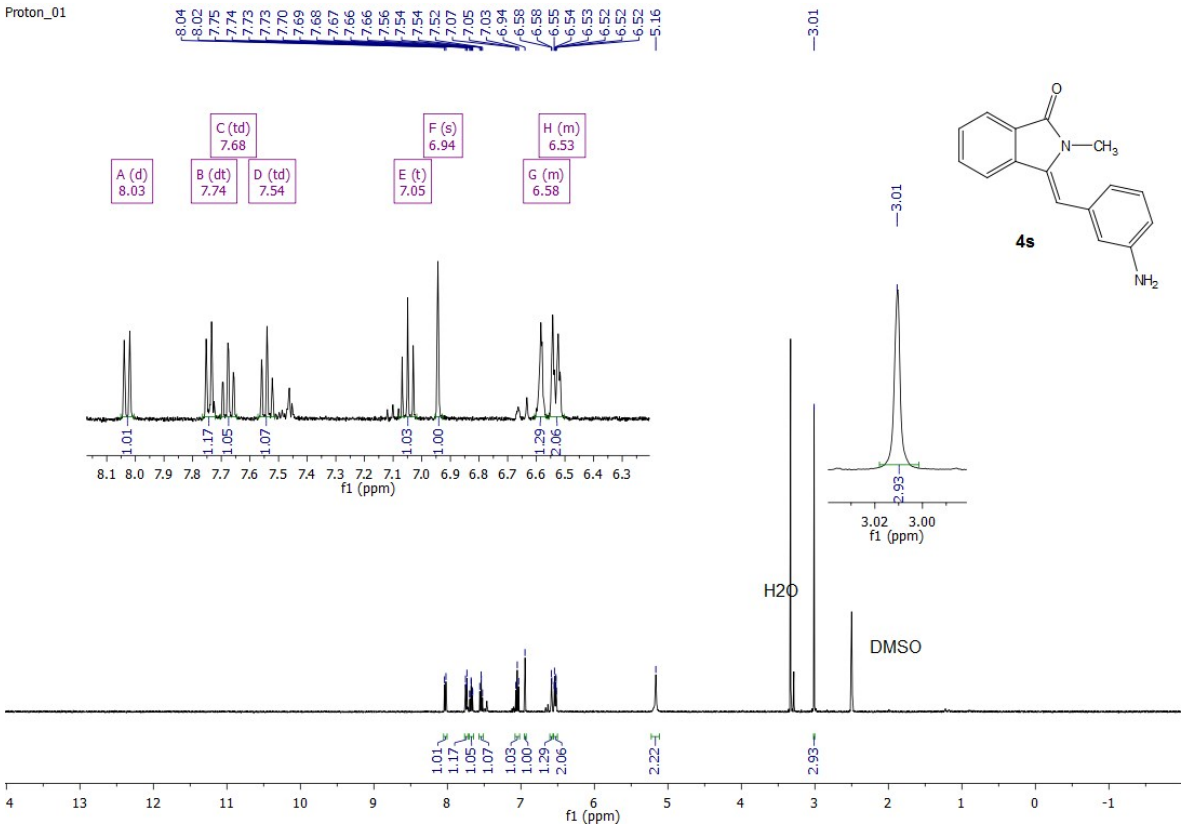

Carbon_01
Tube 22
4p
f1 (ppm)

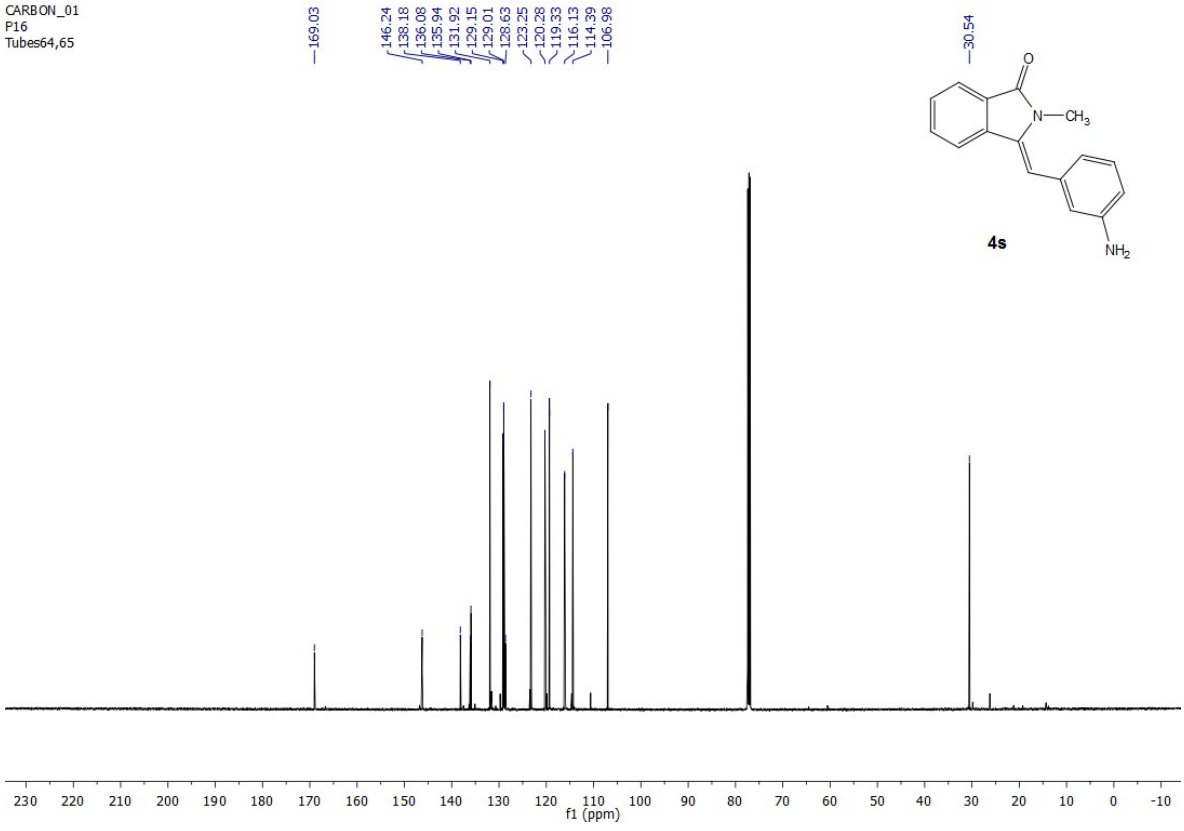

MS-24/1
3.05
2.38
2.38
4q
B (dt) 7.84
C (dt) 7.71
D (td) 7.57
E (td) 7.46
F (m) 7.20
A (s) 6.74
G (s) 3.05
H (d) 2.38
f1 (ppm)

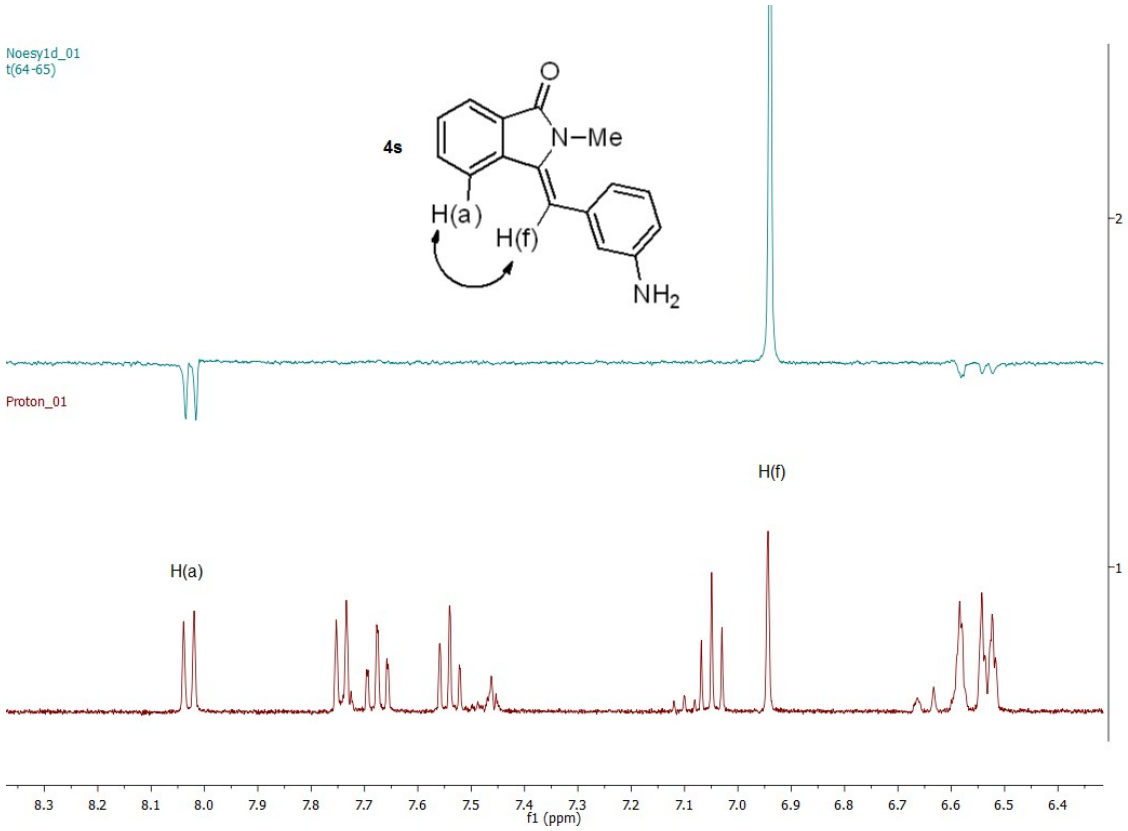

MS-24/3
vlc_13C CDCl3 /opt/topspin2.16 organic 30
168.89
138.10
137.31
135.83
133.90
133.65
131.77
129.60
128.80
128.44
123.09
119.19
106.70
30.55
21.28
4q
f1 (ppm)

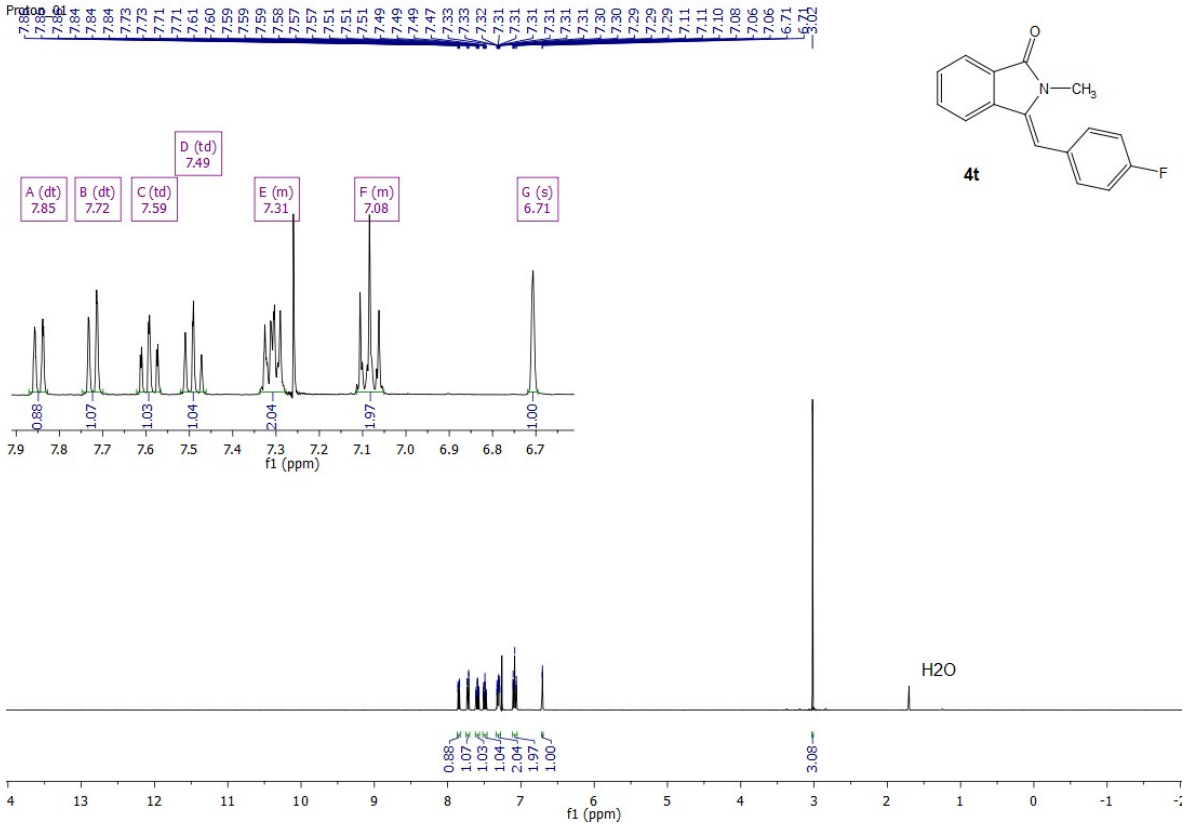

Proton_01
8.08
7.89
7.87
7.87
7.78
7.77
7.76
7.65
7.65
7.63
7.63
7.61
7.61
7.52
7.52
7.50
7.50
7.49
7.48
7.40
7.40
7.40
7.38
7.38
6.99
6.98
6.97
6.96
6.52
3.86
Proton_01
A (s) 8.08
B (dt) 7.88
C (dt) 7.77
D (td) 7.63
E (td) 7.50
F (m) 7.39
G (m) 6.98
H (s) 6.52
1.10
0.89
0.99
0.99
1.00
2.08
2.06
1.01
2.79
f1 (ppm)
H2O
4r

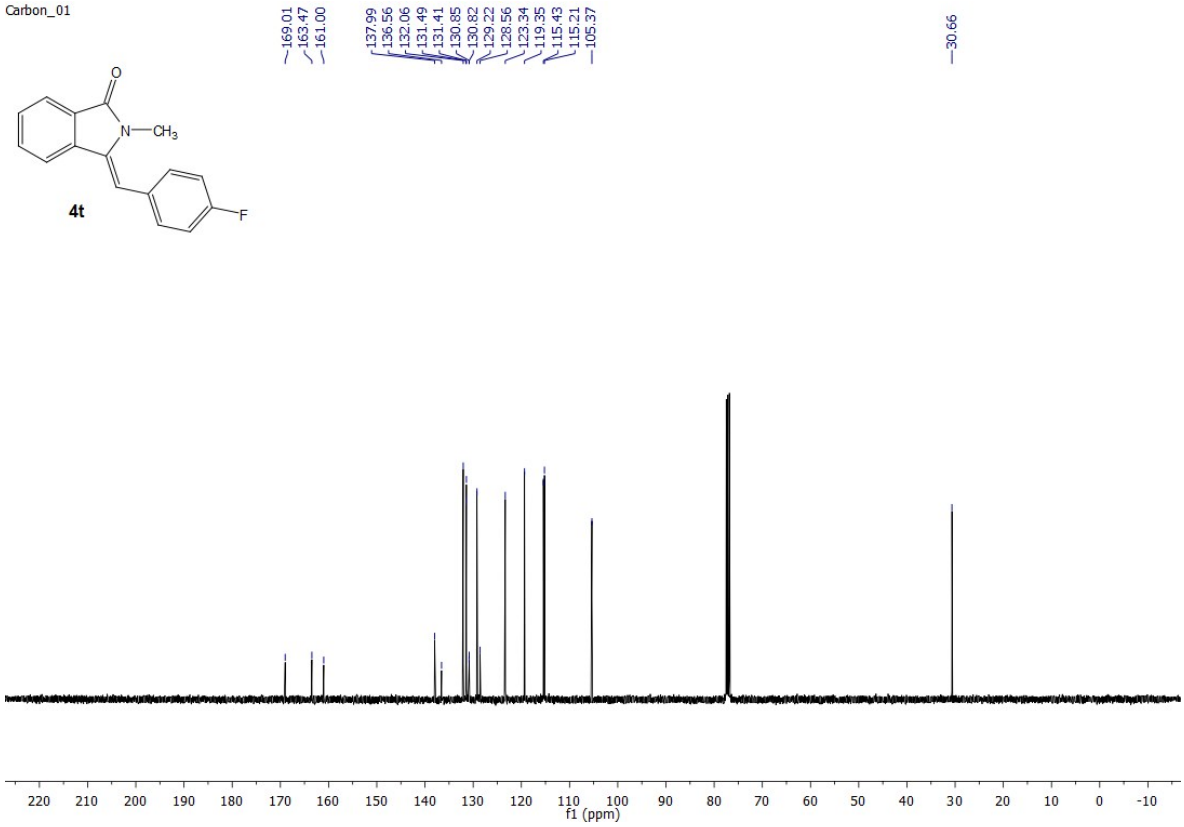

Carbon_01
168.99
159.31
138.44
135.12
132.33
131.83
130.83
129.91
129.04
127.62
123.70
119.77
114.91
106.01
55.55
4r
f1 (ppm)

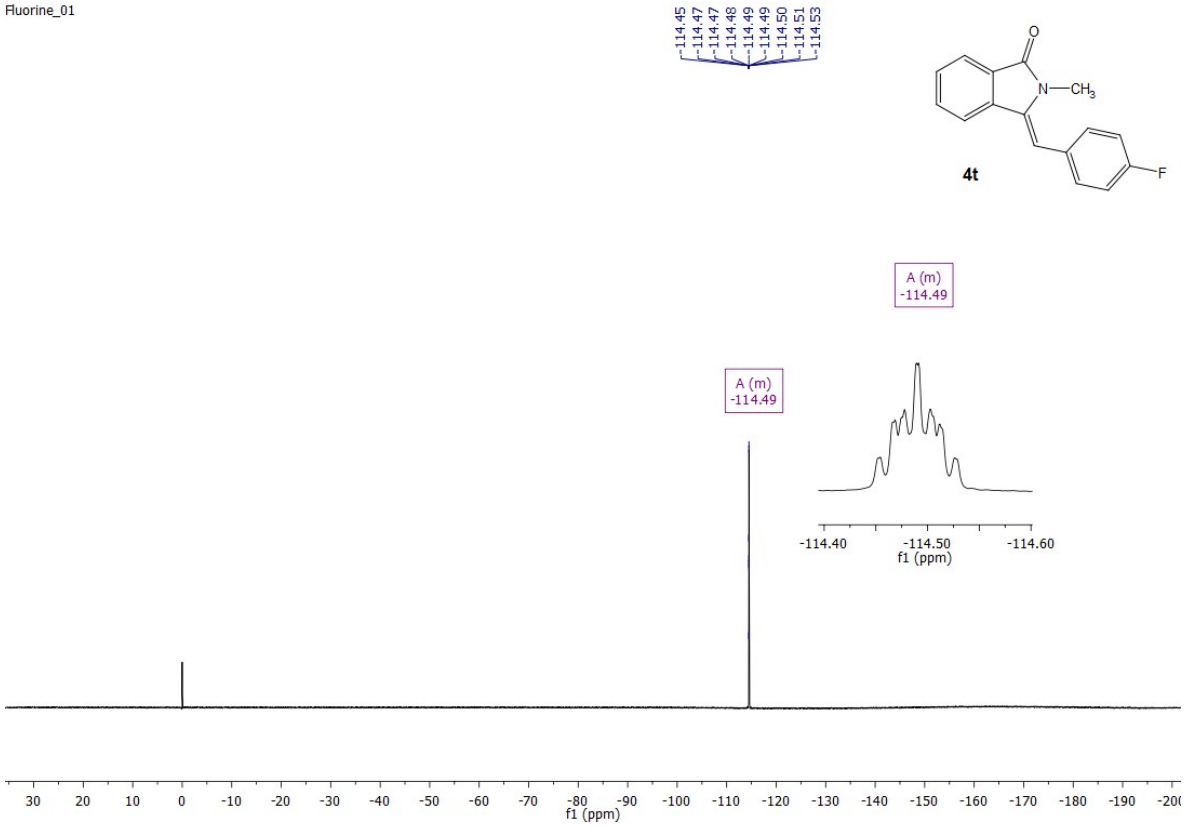

Proton_01
A (d) 8.03
B (dt) 7.74
C (td) 7.68
D (td) 7.54
E (t) 7.05
F (s) 6.94
G (m) 6.58
H (m) 6.53
4s
H2O
DMSO
f1 (ppm)

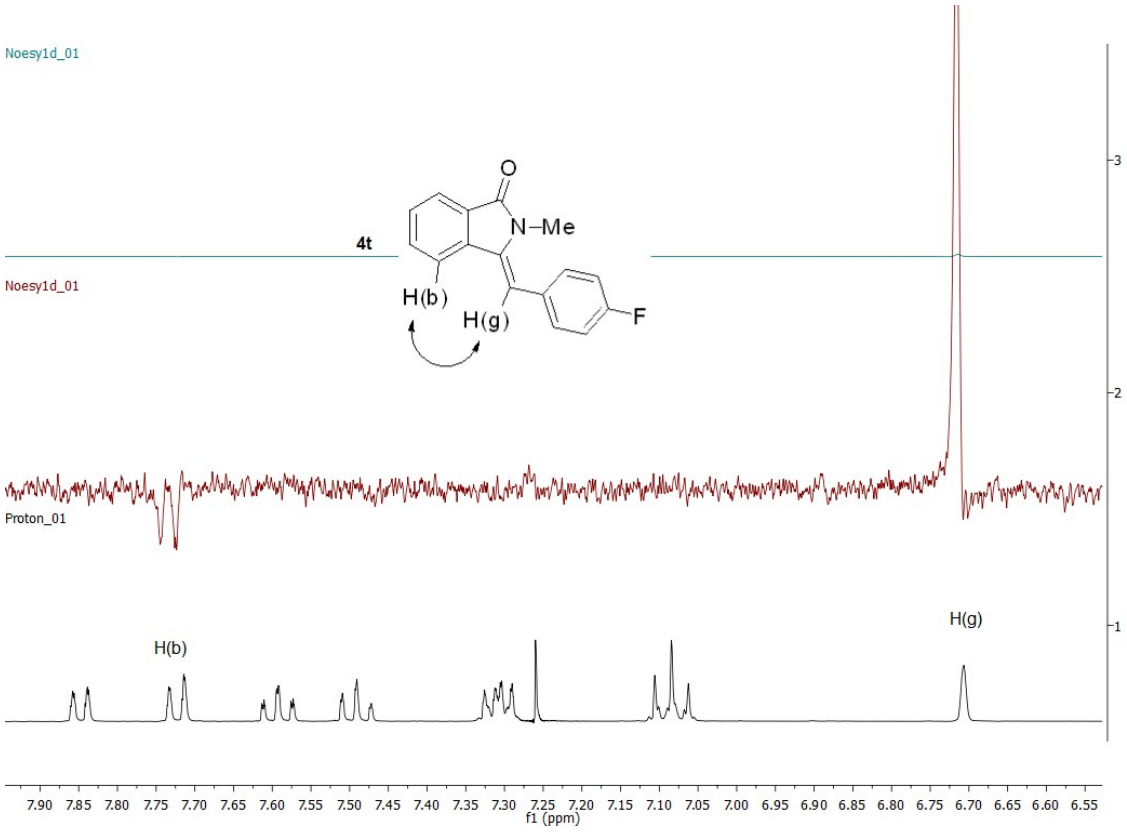

CARBON_01
P16
Tubes64,65
4s
f1 (ppm)

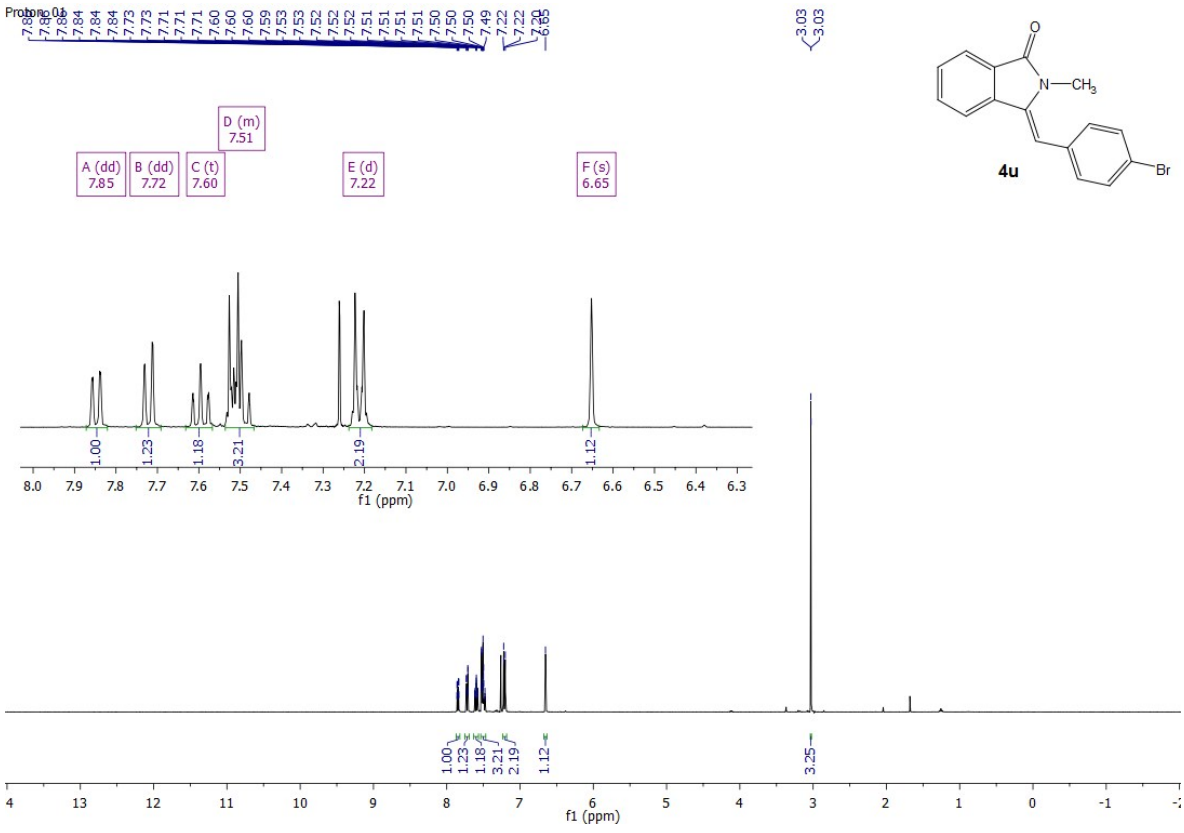

Noesy1d_01
t(64-65)
4s
O
N–Me
H(a)
H(f)
NH2
Proton_01
H(f)
H(a)
2
1
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4
f1 (ppm)

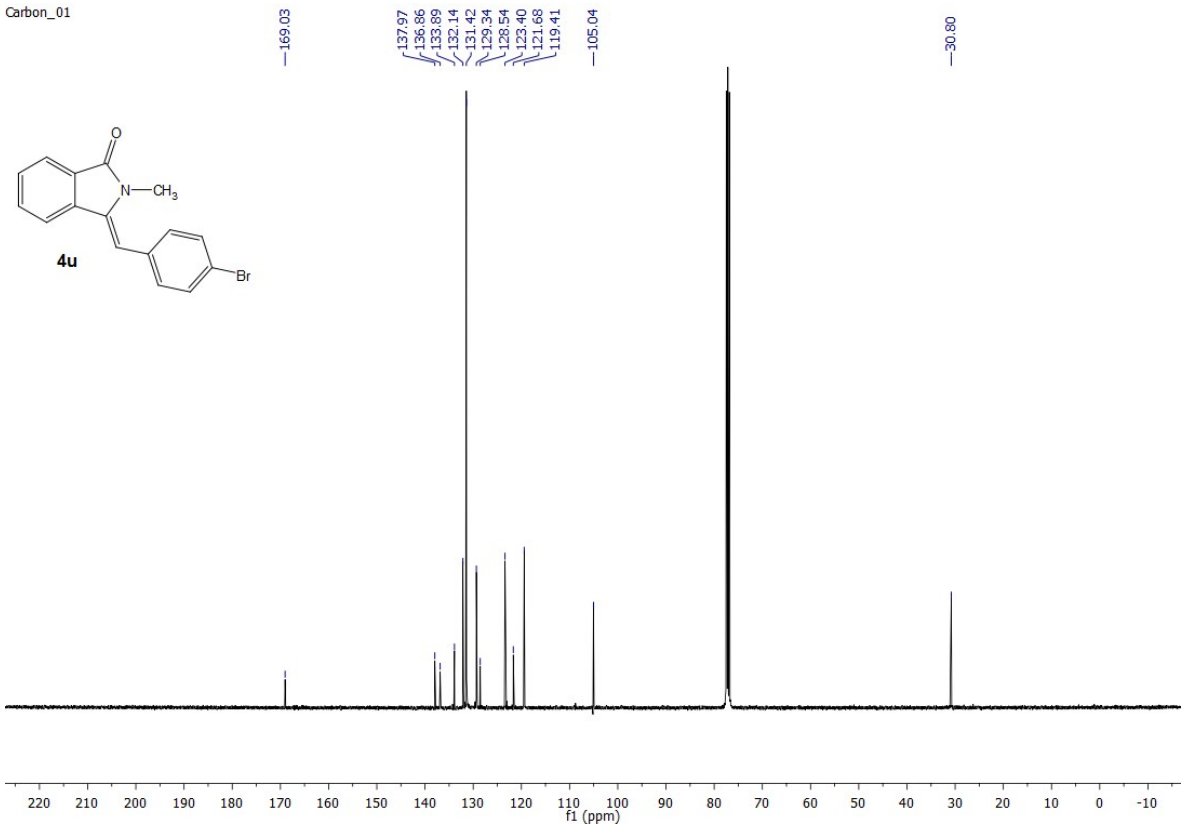

Proton_01
D (td)
7.49
A (dt)
7.85
B (dt)
7.72
C (td)
7.59
E (m)
7.31
F (m)
7.08
G (s)
6.71
0.88
1.07
1.03
1.04
2.04
1.97
1.00
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7
f1 (ppm)
O
N—CH3
F
4t
H2O
0.88
1.07
1.03
1.04
2.04
1.97
1.00
3.08
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)

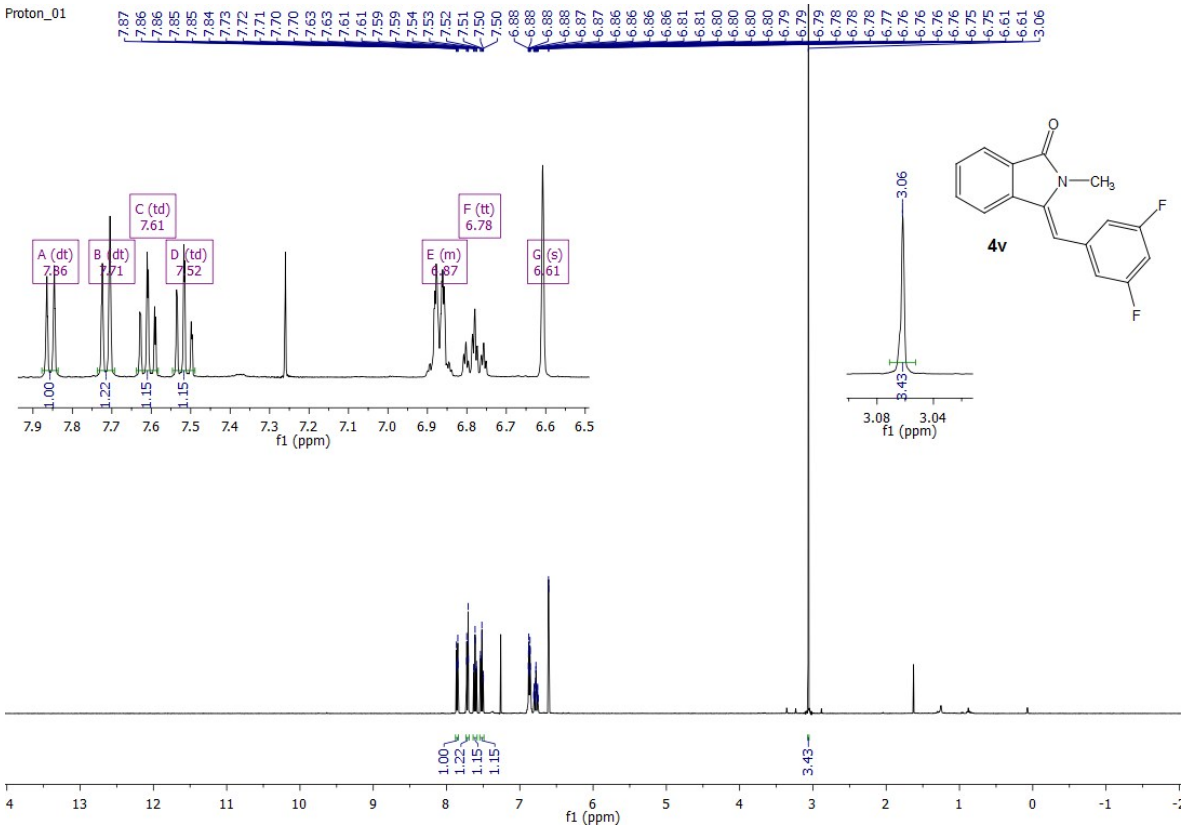

Carbon_01
169.01
163.47
161.00
137.99
136.56
132.06
131.49
131.41
130.85
130.82
129.22
128.56
123.34
119.35
115.43
115.21
105.37
30.66
O
N—CH3
4t
F
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

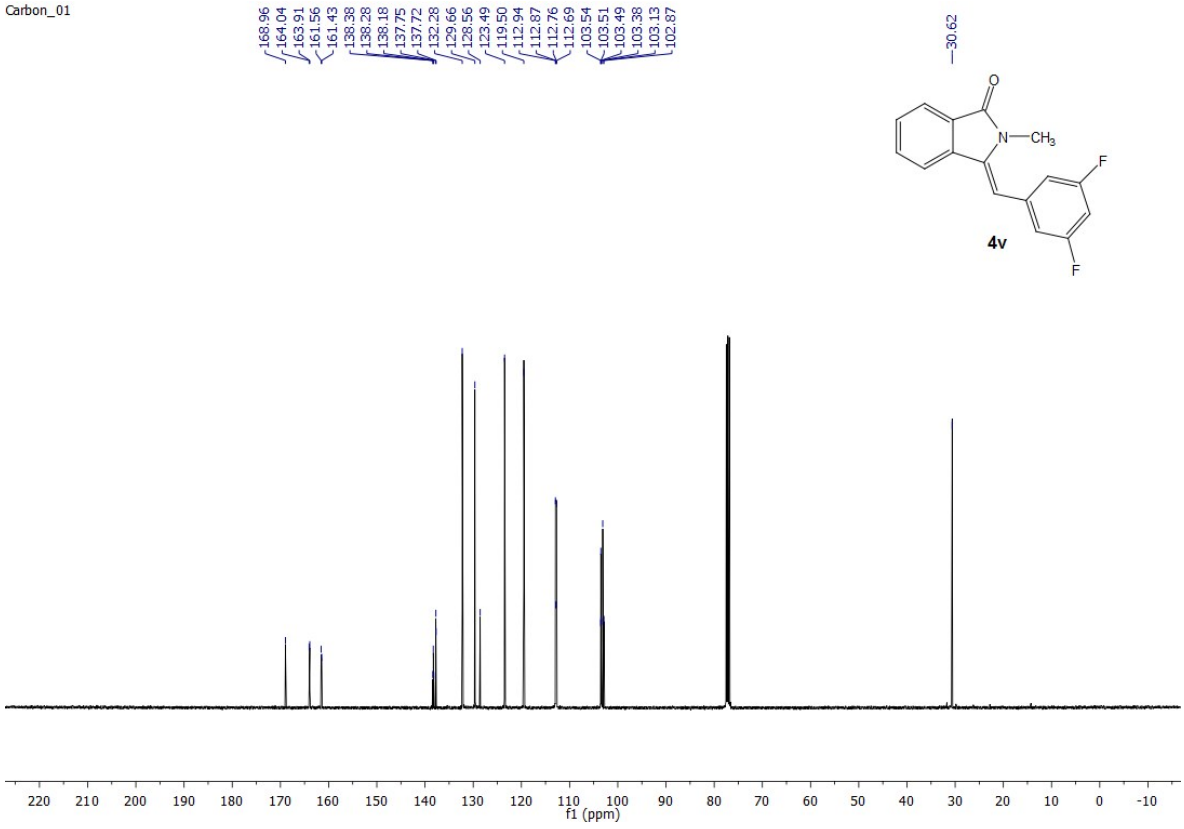

Fluorine_01
-114.45
-114.47
-114.47
-114.48
-114.49
-114.49
-114.50
-114.51
-114.53
O
N—CH3
4t
F
A (m)
-114.49
A (m)
-114.49
-114.40
-114.50
-114.60
f1 (ppm)
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

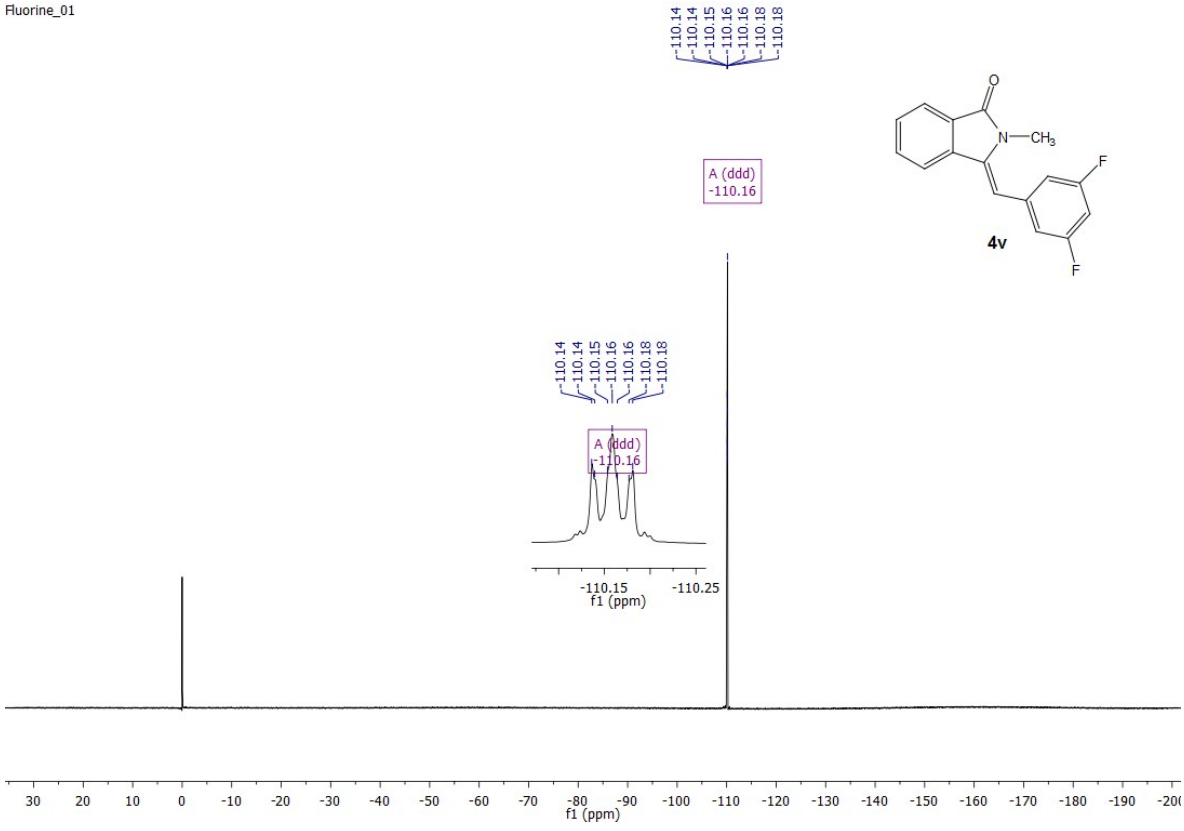

Noesy1d_01
4t
O
N–Me
H(b)
H(g)
F
Noesy1d_01
Proton_01
H(b)
H(g)
f1 (ppm)

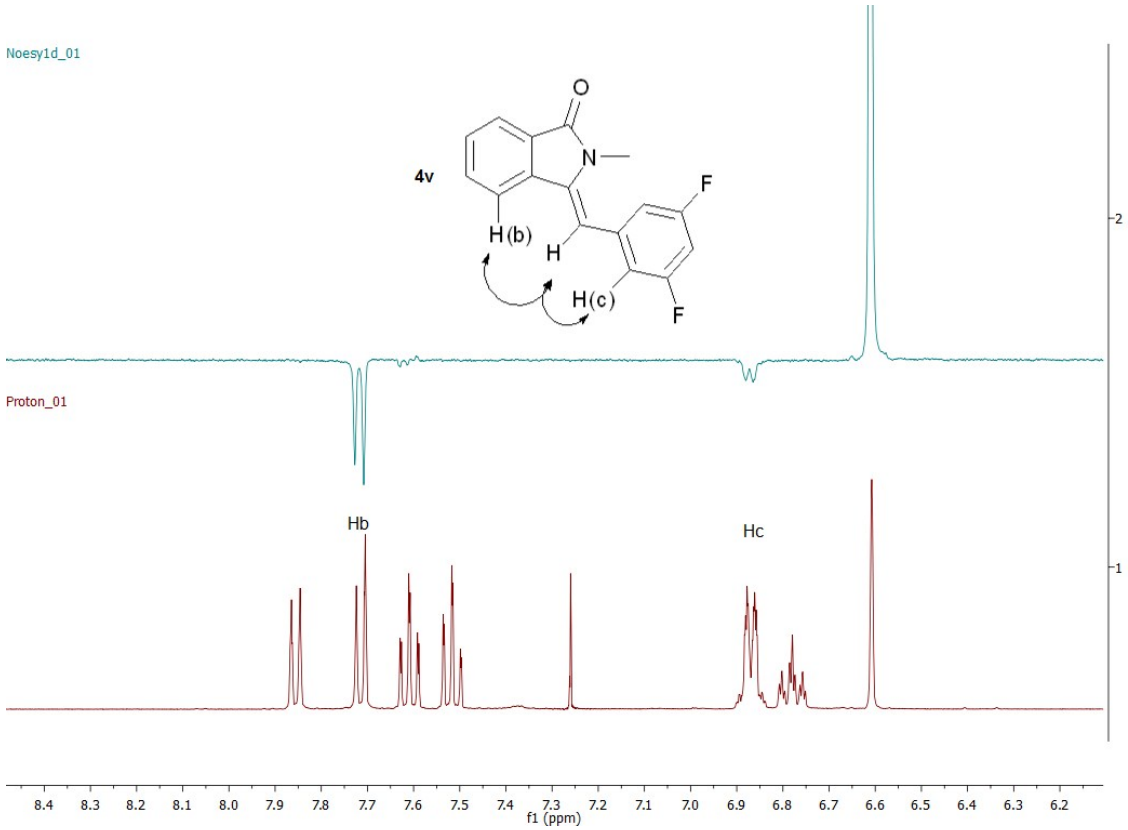

D (m)
7.51
A (dd)
7.85
B (dd)
7.72
C (t)
7.60
E (d)
7.22
F (s)
6.65
4u
f1 (ppm)


Carbon_01
169.03
137.97
136.86
133.89
132.14
131.42
129.34
128.54
123.40
121.68
119.41
105.04
30.80
O
N—CH3
Br
4u
f1 (ppm)

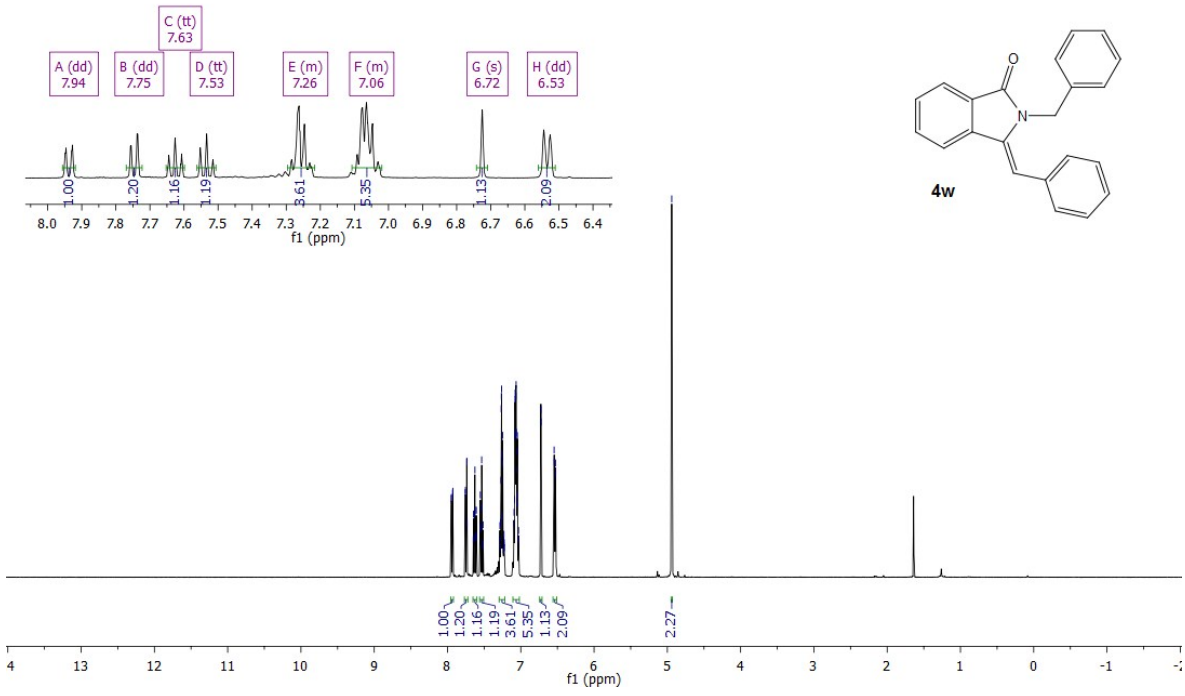

Proton_01
A (dt) 7.86
B (dt) 7.71
C (td) 7.61
D (td) 7.52
E (m) 6.87
F (tt) 6.78
G (s) 6.61
3.06
O
N—CH3
F
F
4v
1.00
1.22
1.15
1.15
3.43
f1 (ppm)

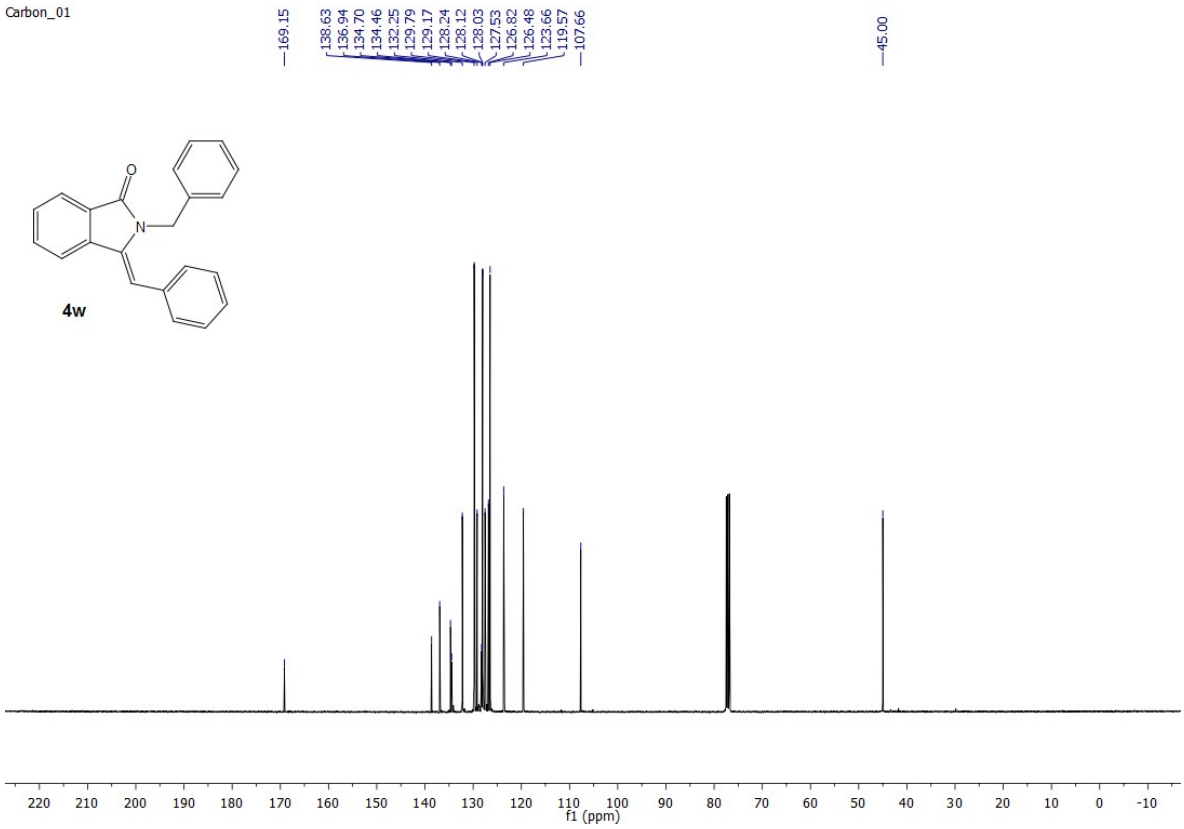

Carbon_01
168.96
164.04
163.91
161.56
161.43
138.38
138.28
138.18
137.75
137.72
132.28
129.66
128.56
123.49
119.50
112.94
112.87
112.76
112.69
103.54
103.51
103.49
103.38
103.13
102.87
30.62
O
N
CH3
F
F
4v
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

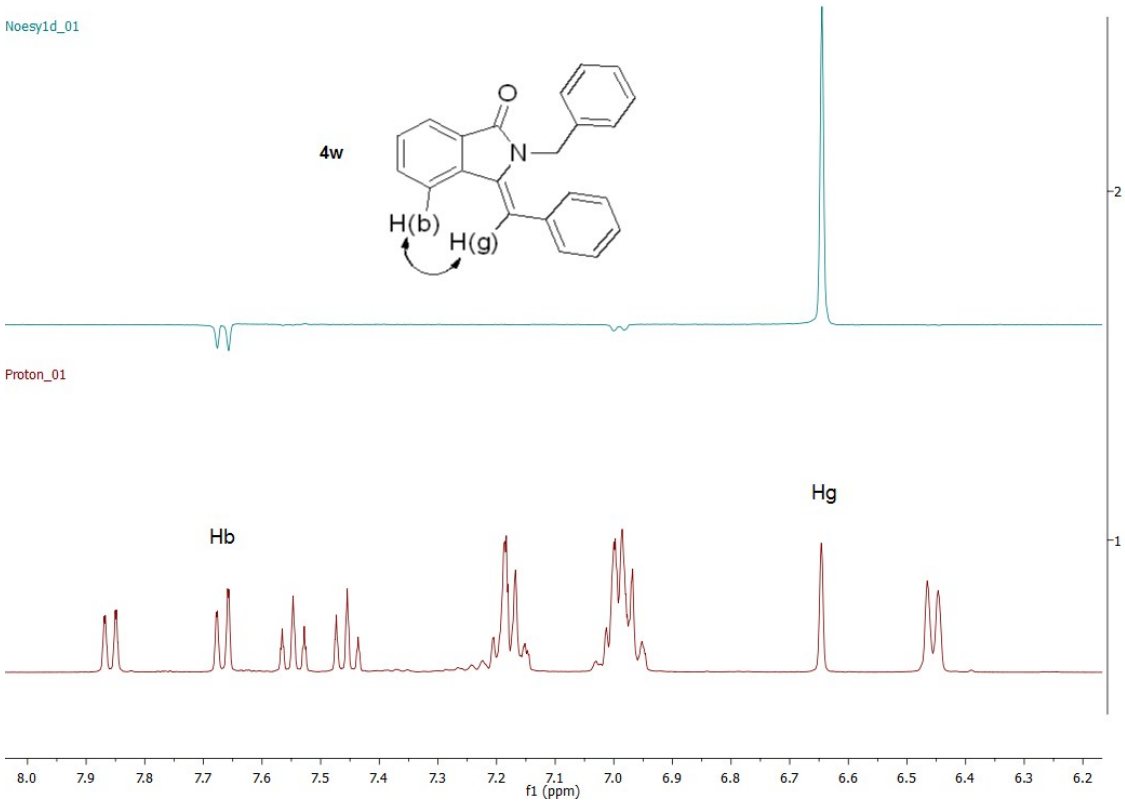

Fluorine_01
-110.14
-110.14
-110.15
-110.16
-110.16
-110.18
-110.18
A (ddd)
-110.16
O
N
CH3
F
F
4v
-110.15
-110.25
f1 (ppm)
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190
f1 (ppm)

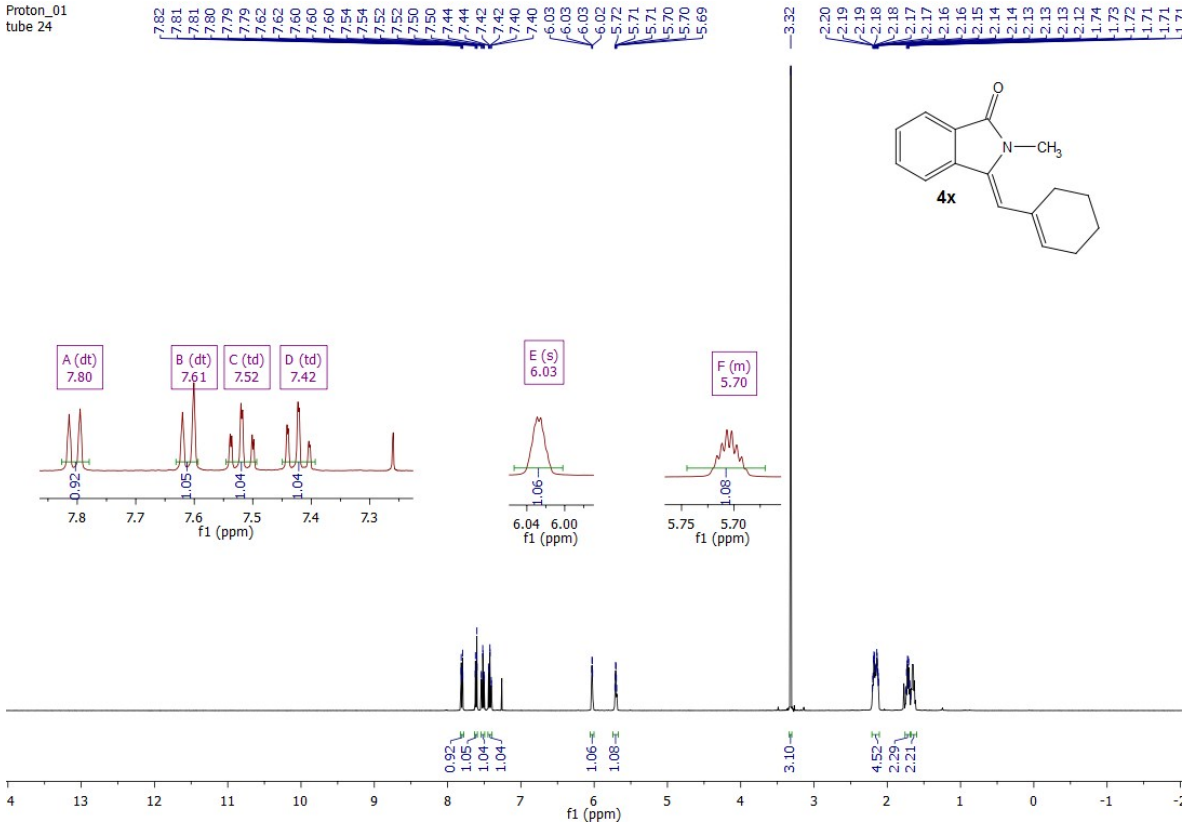

Noesy1d_01
4v
O
N
F
H(b)
H
H(c)
F
Proton_01
Hb
Hc
2
1
8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2
f1 (ppm)

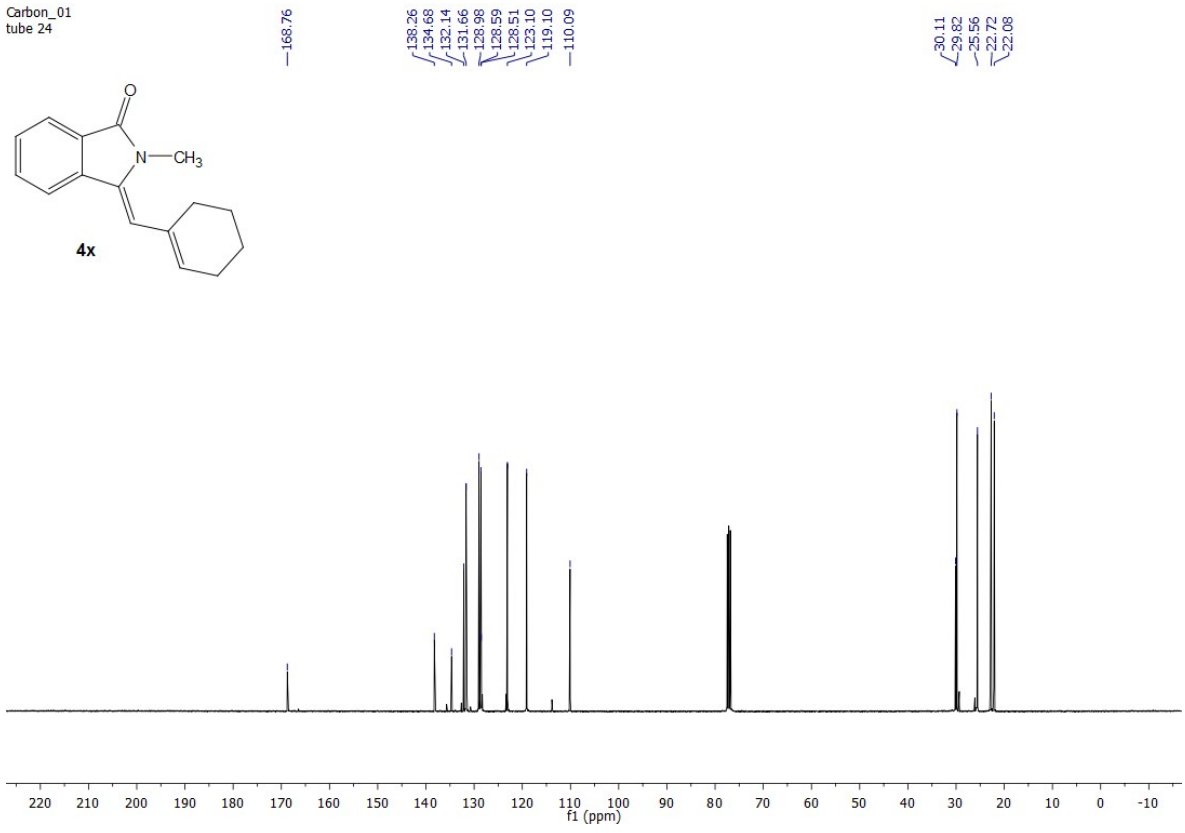

Proton_01

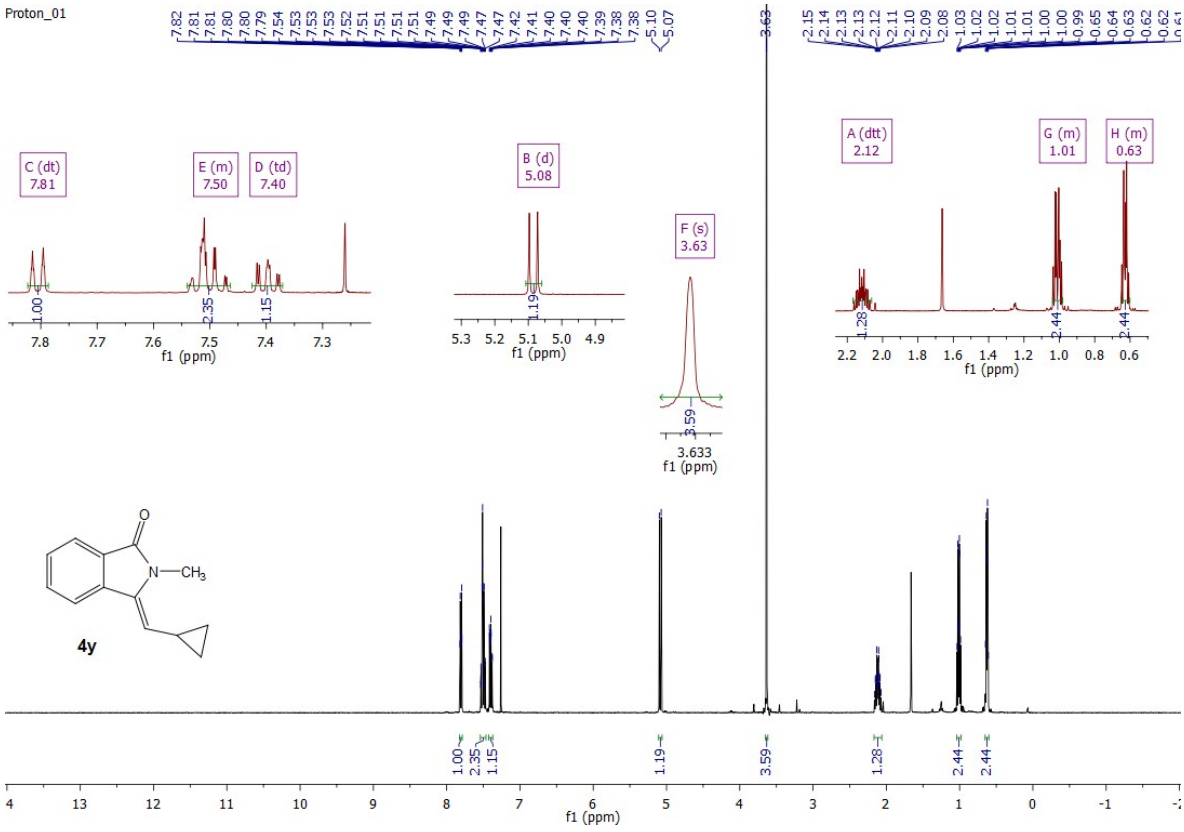

C (tt)
7.63
A (dd)
7.94
B (dd)
7.75
D (tt)
7.53
E (m)
7.26
F (m)
7.06
G (s)
6.72
H (dd)
6.53
1.00
1.20
1.16
1.19
3.61
5.35
1.13
2.09
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4
f1 (ppm)
O
N
4w
1.00
1.20
1.16
1.19
3.61
5.35
1.13
2.09
2.27
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)

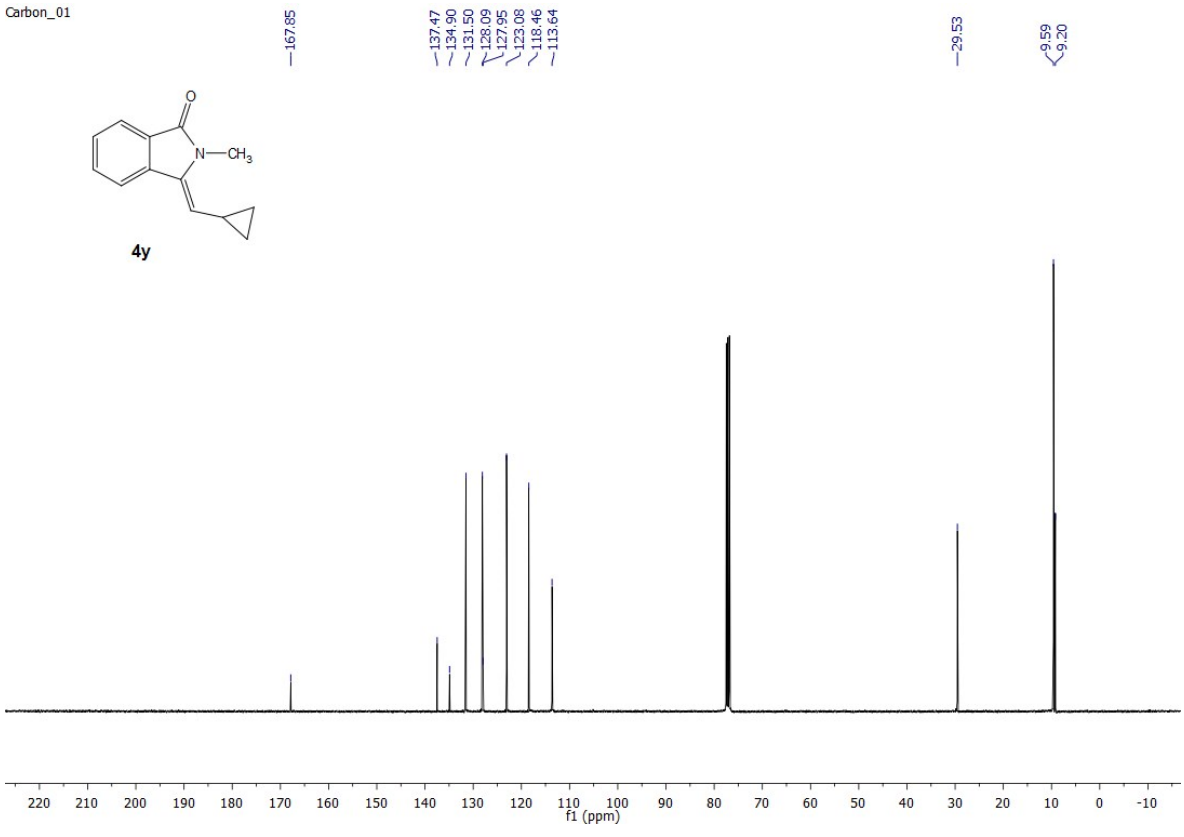

Carbon_01
169.15
138.63
136.94
134.70
134.46
132.25
129.79
129.17
128.24
128.12
128.03
127.53
126.82
126.48
123.66
119.57
107.66
45.00
4w
f1 (ppm)

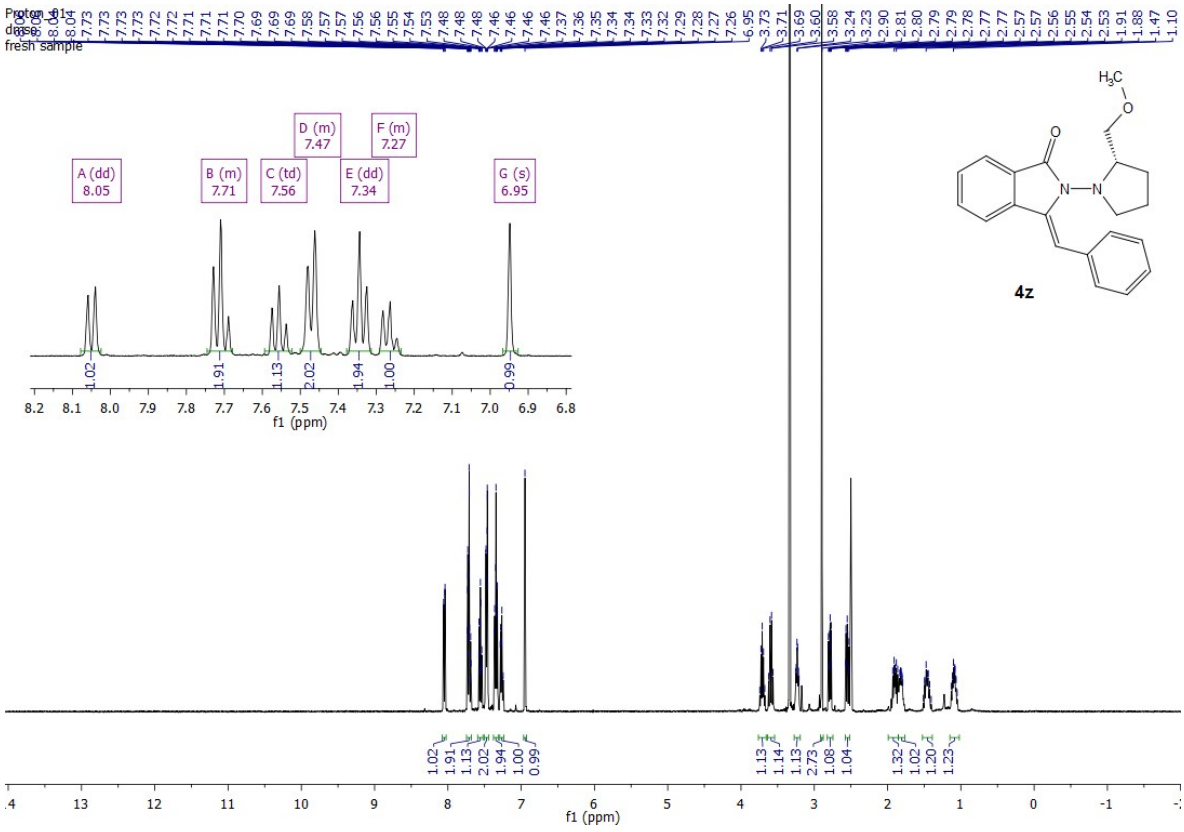

Noesy1d_01
4w
H(b)
H(g)
Proton_01
Hb
Hg
f1 (ppm)

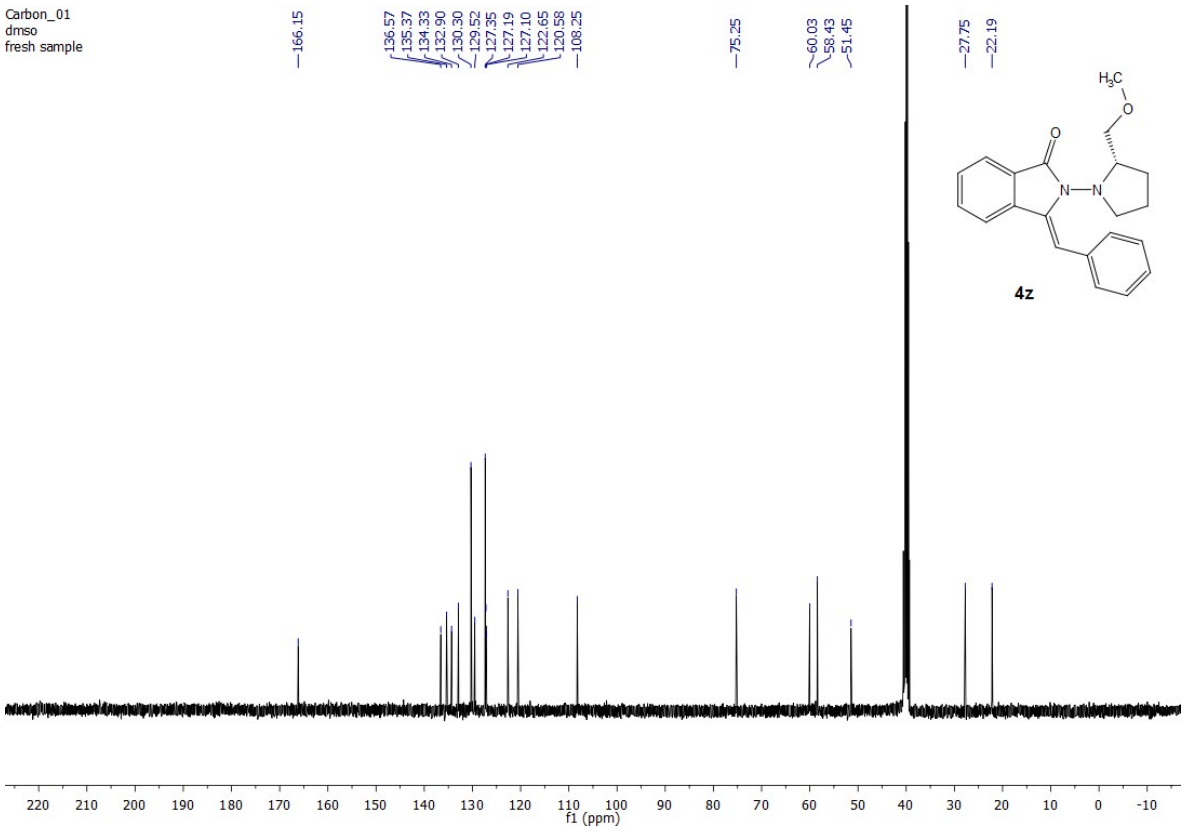

Proton_01
tube 24
4x
A (dt) 7.80
B (dt) 7.61
C (td) 7.52
D (td) 7.42
E (s) 6.03
F (m) 5.70
f1 (ppm)

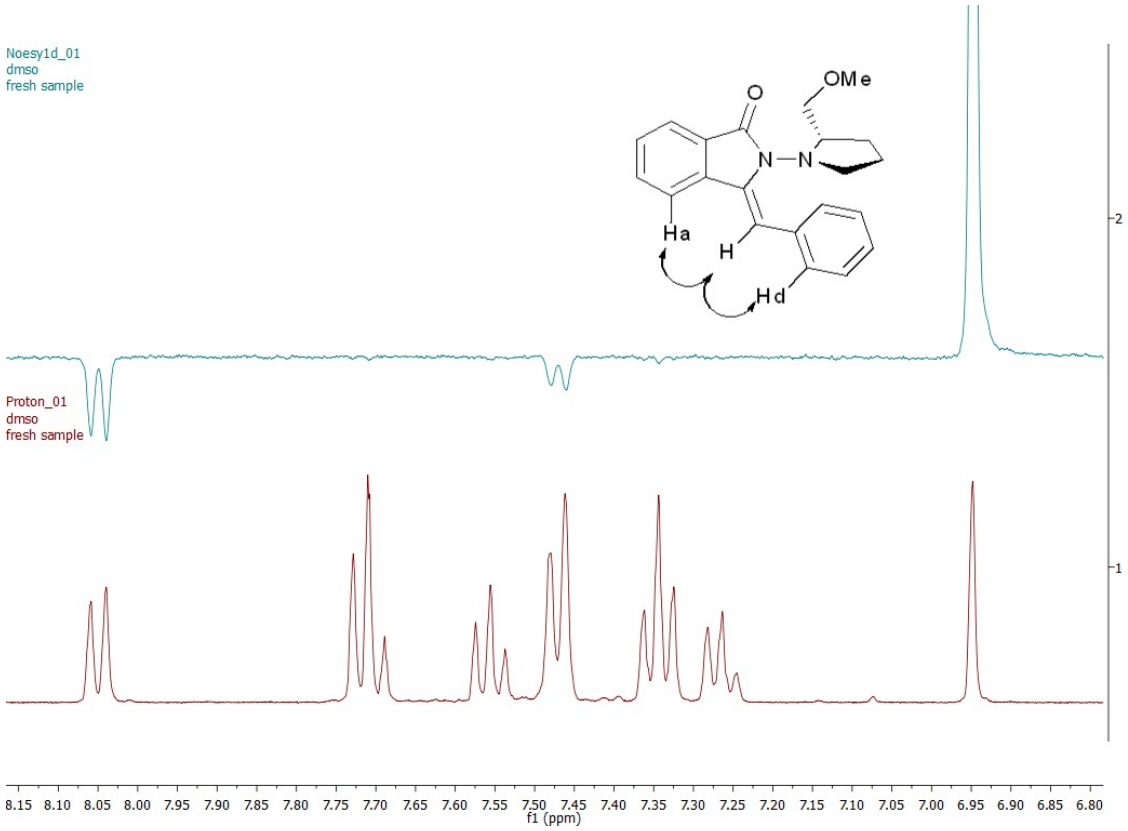

Carbon_01
tube 24
4x
168.76
138.26
134.68
132.14
131.66
128.98
128.59
128.51
123.10
119.10
110.09
30.11
29.82
25.56
22.72
22.08
f1 (ppm)

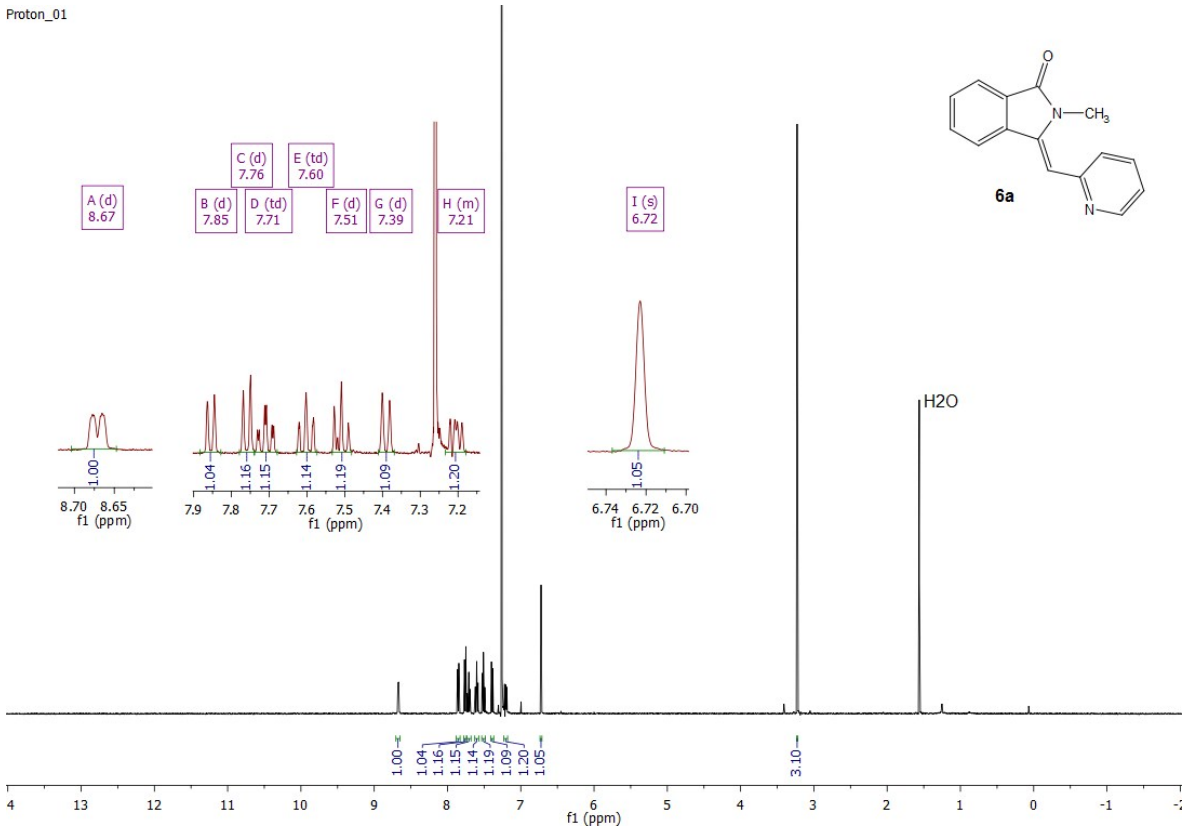

Proton_01
C (dt) 7.81
E (m) 7.50
D (td) 7.40
B (d) 5.08
F (s) 3.63
A (dtt) 2.12
G (m) 1.01
H (m) 0.63
f1 (ppm)
4y

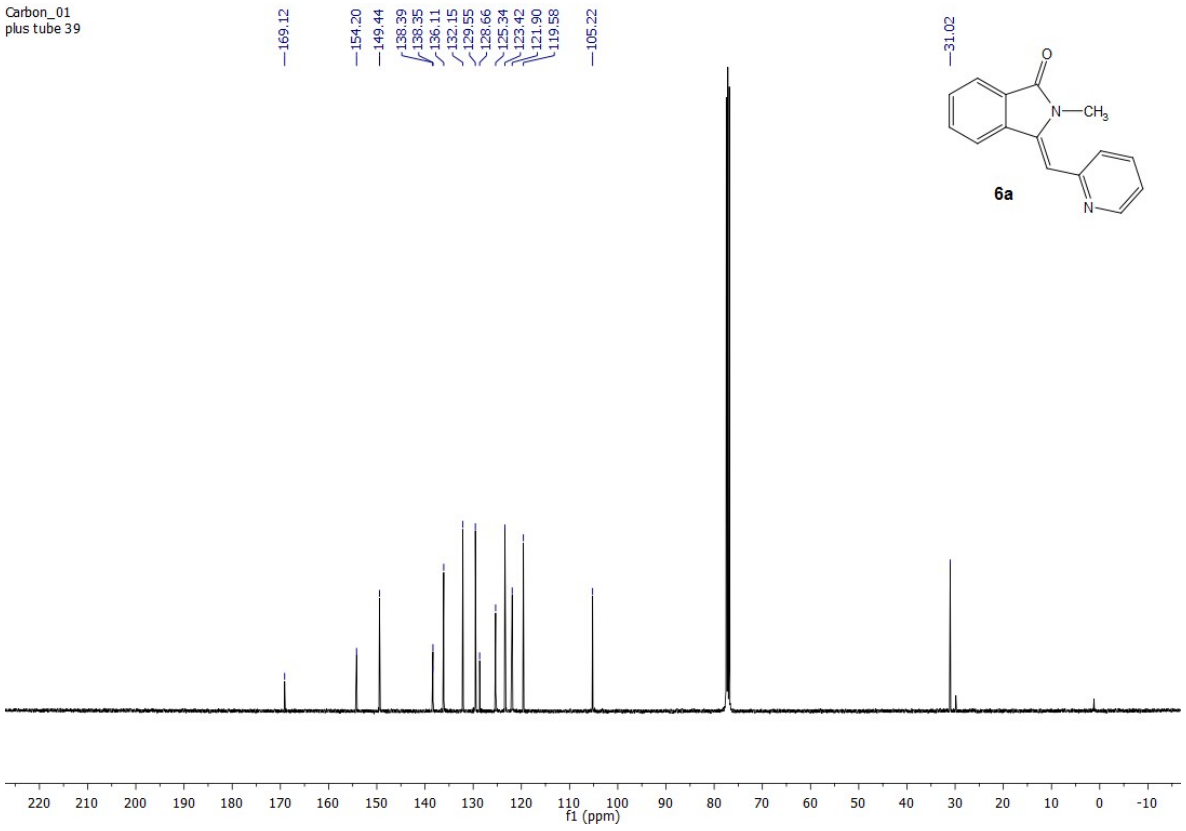

Carbon_01
167.85
137.47
134.90
131.50
128.09
127.95
123.08
118.46
113.64
29.53
9.59
9.20
f1 (ppm)
4y

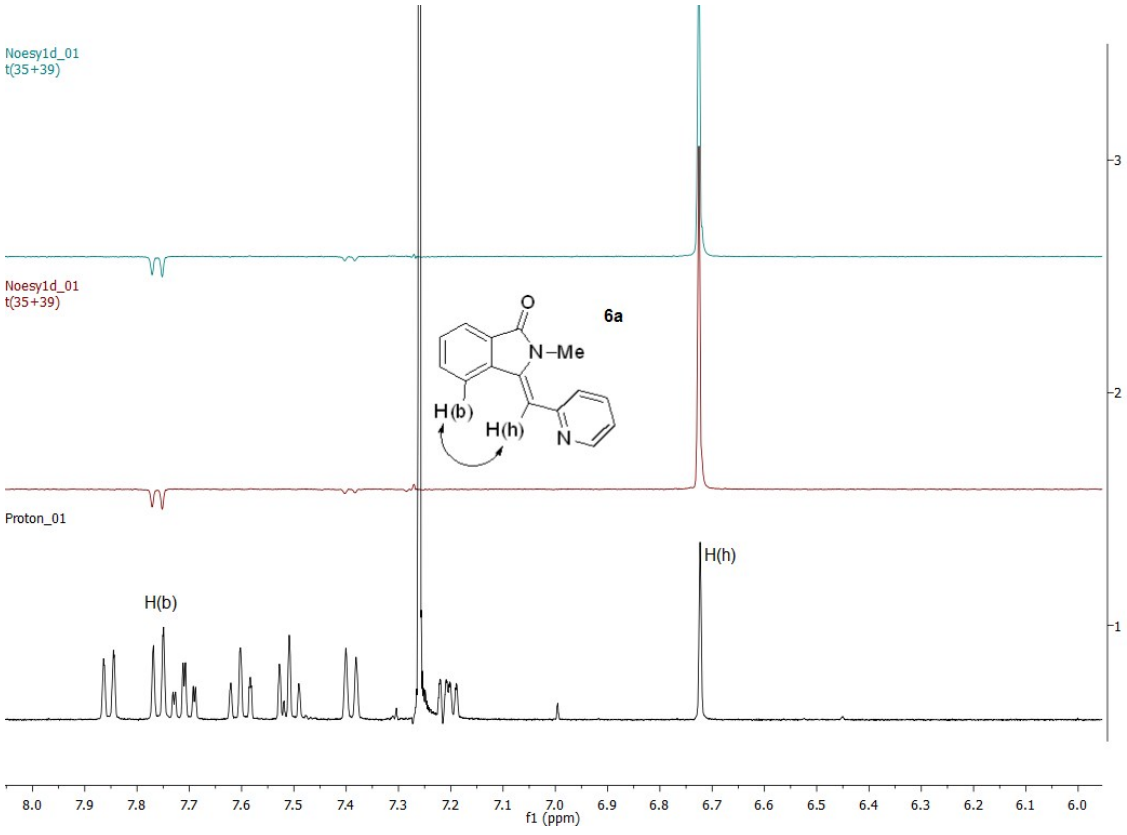

Proton_01
dmso
fresh sample
A (dd)
8.05
B (m)
7.71
C (td)
7.56
D (m)
7.47
E (dd)
7.34
F (m)
7.27
G (s)
6.95
f1 (ppm)
4z

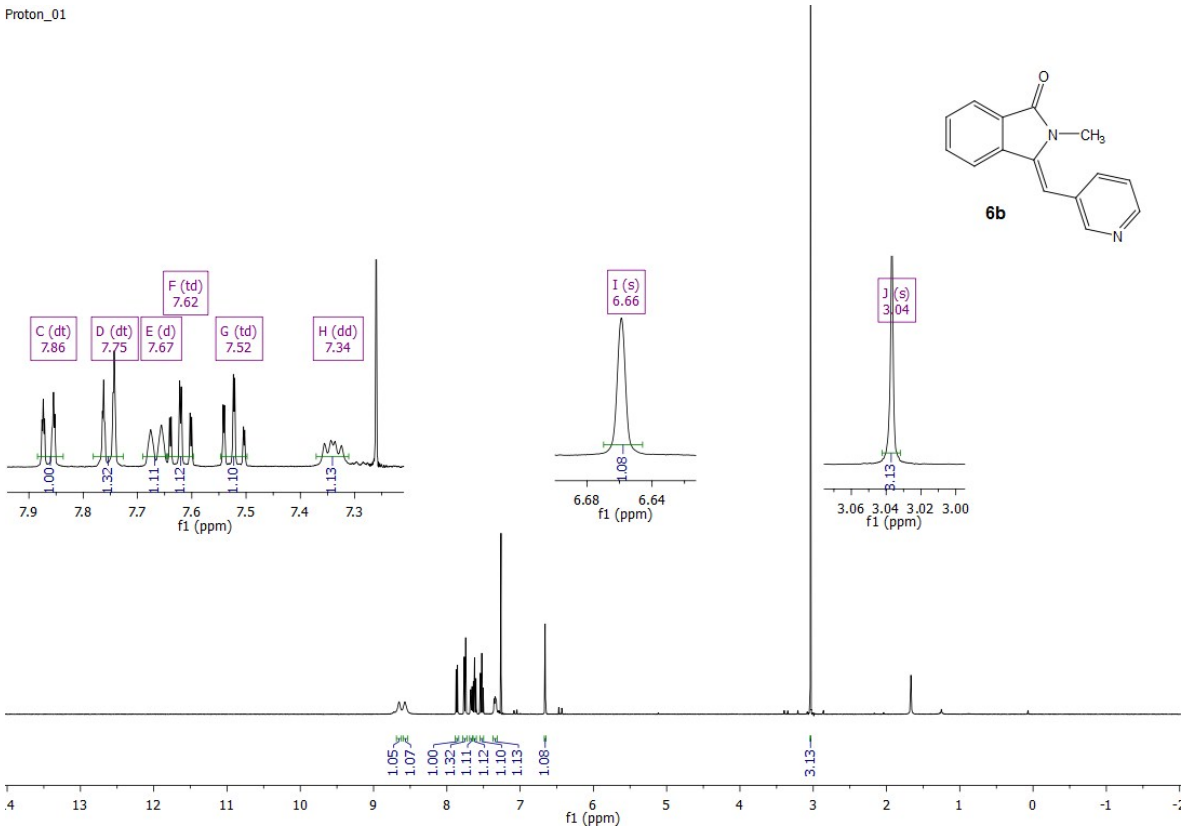

Carbon_01
dmso
fresh sample
166.15
136.57
135.37
134.33
132.90
130.30
129.52
127.35
127.19
127.10
122.65
120.58
108.25
75.25
60.03
58.43
51.45
27.75
22.19
f1 (ppm)
4z

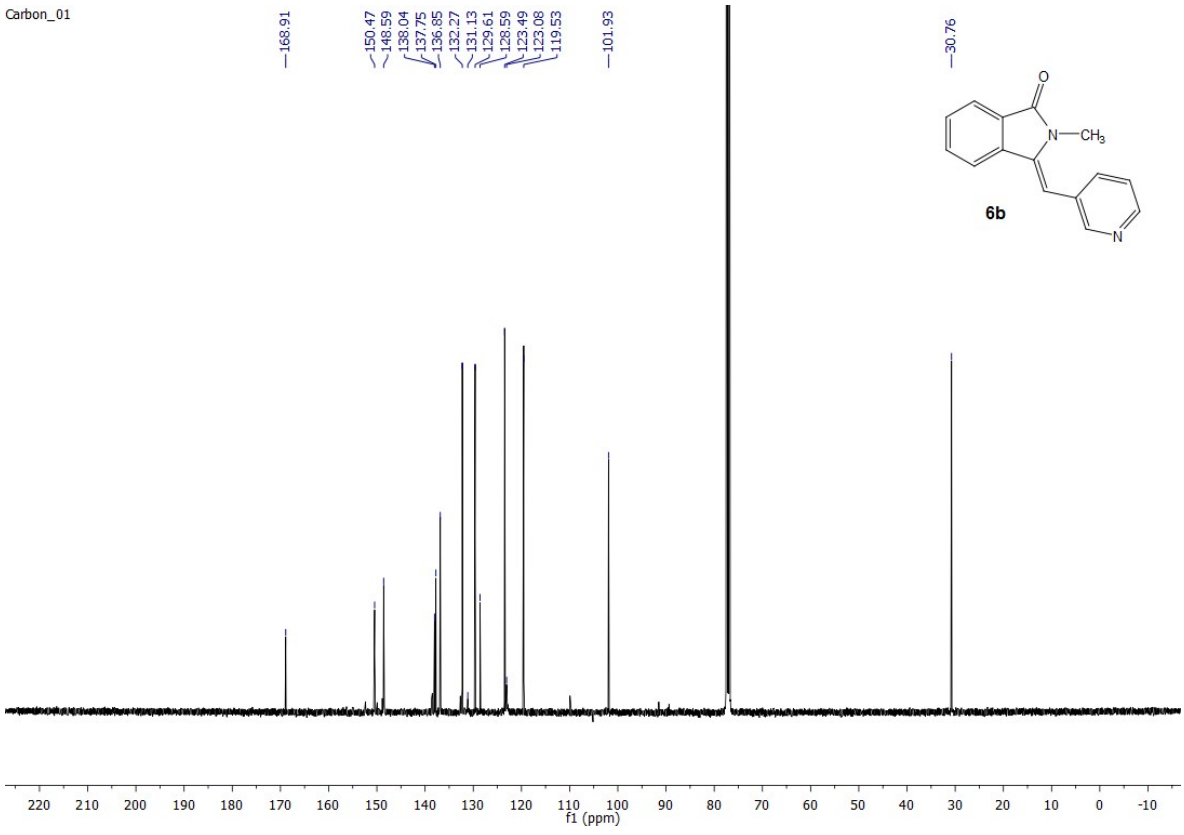

Noesy1d_01
dmso
fresh sample
O
OMe
N—N
Ha
H
Hd
2
Proton_01
dmso
fresh sample
1
8.15 8.10 8.05 8.00 7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80
f1 (ppm)

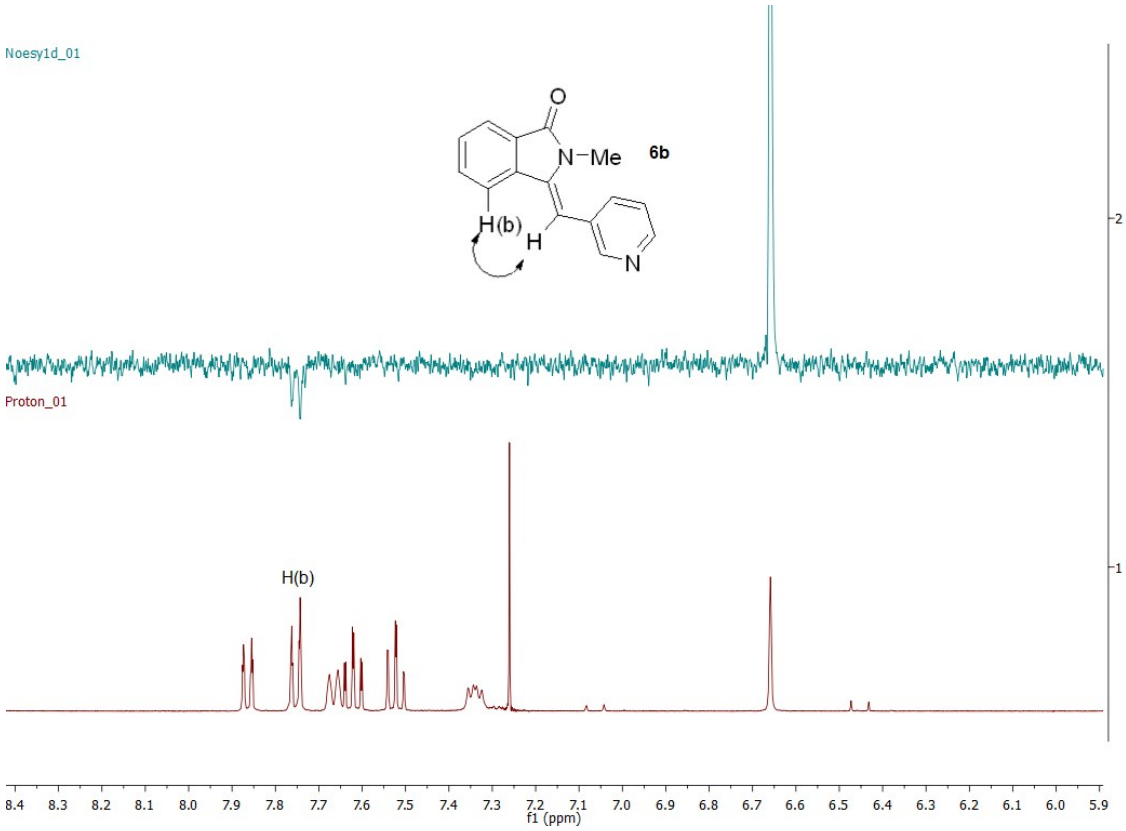

Proton_01
A (d) 8.67
B (d) 7.85
C (d) 7.76
D (td) 7.71
E (td) 7.60
F (d) 7.51
G (d) 7.39
H (m) 7.21
I (s) 6.72
O
N—CH3
N
6a
H2O
1.00
1.04
1.16
1.15
1.14
1.19
1.09
1.20
1.05
3.10
f1 (ppm)

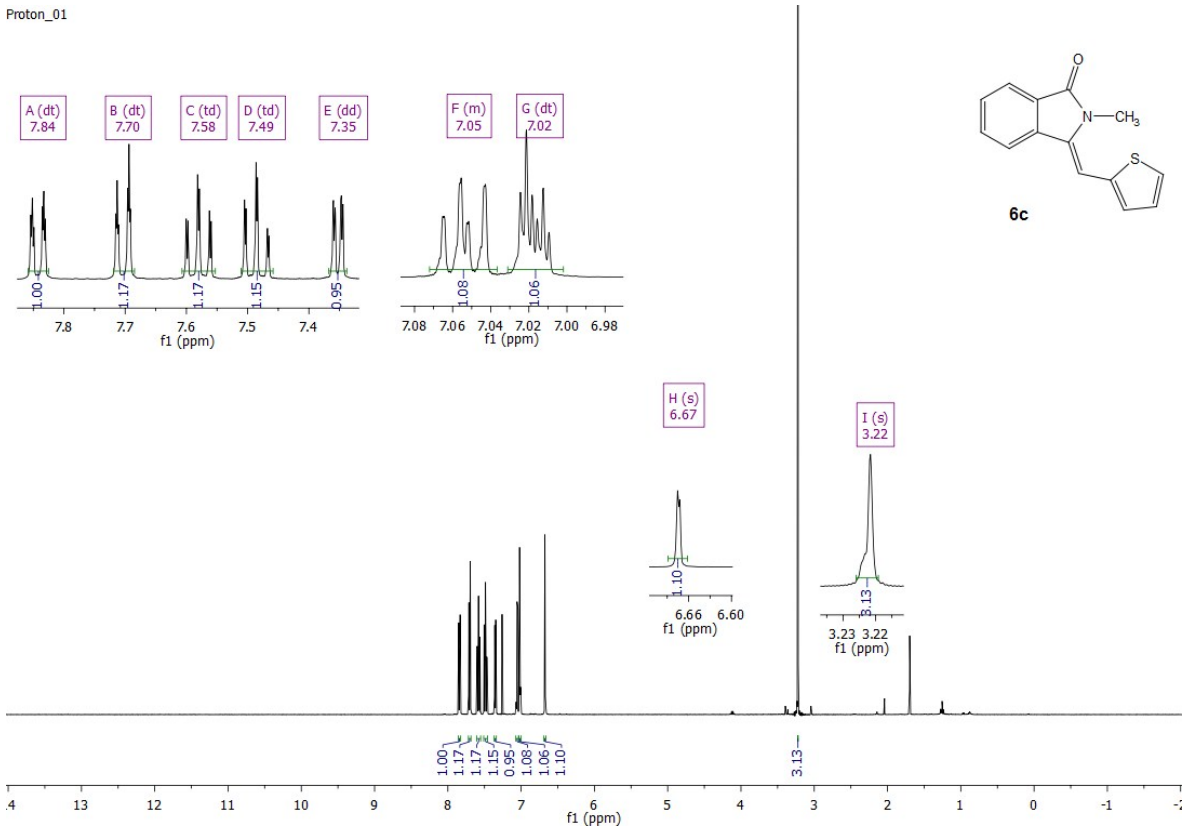

Carbon_01
plus tube 39
169.12
154.20
149.44
138.39
138.35
136.11
132.15
129.55
128.66
125.34
123.42
121.90
119.58
105.22
31.02
6a
f1 (ppm)

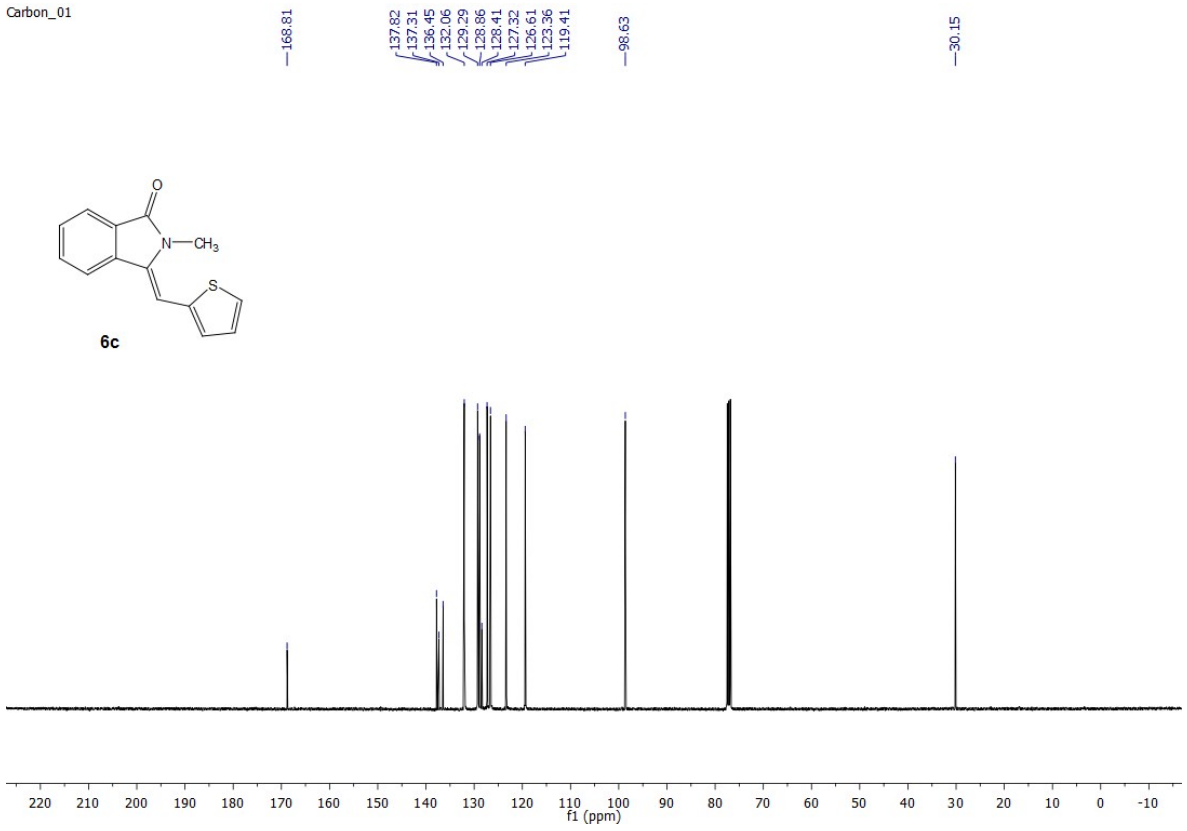

Noesy1d_01
t(35+39)
Noesy1d_01
t(35+39)
Proton_01
6a
N–Me
H(b)
H(h)
H(b)
H(h)
f1 (ppm)

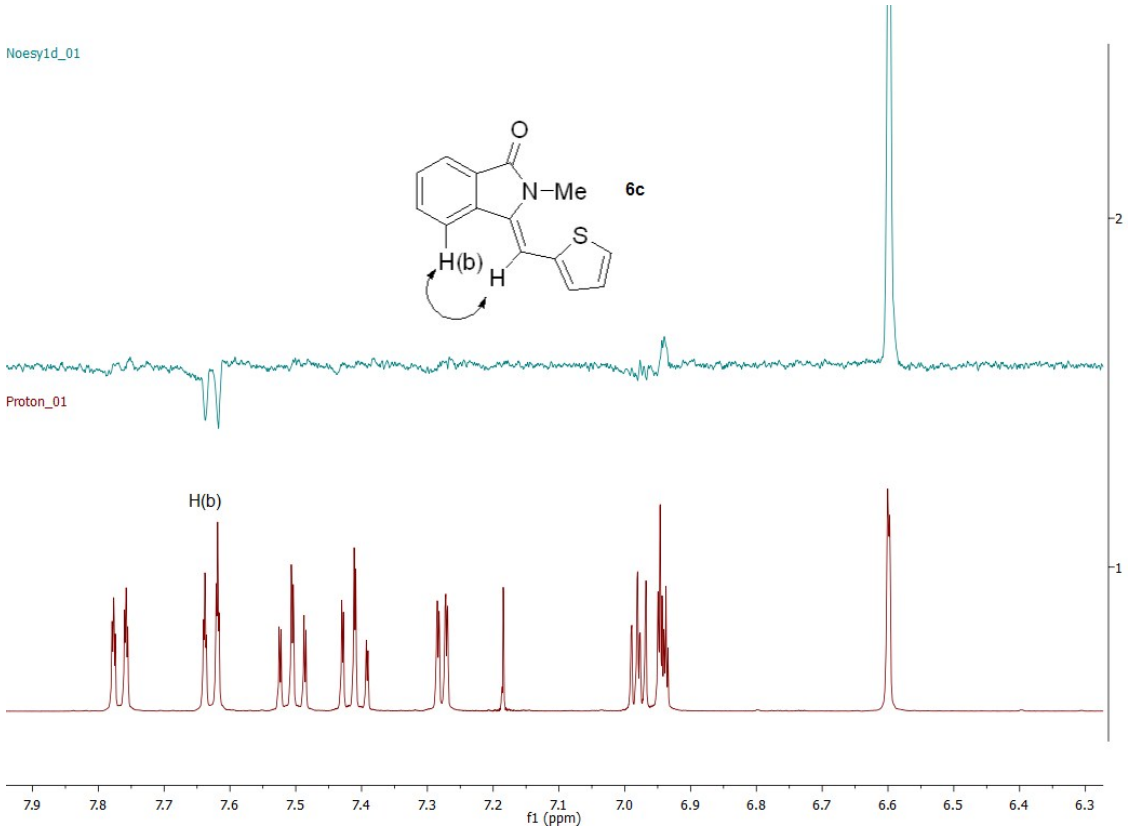

Proton_01
C (dt) 7.86
D (dt) 7.75
E (d) 7.67
F (td) 7.62
G (td) 7.52
H (dd) 7.34
I (s) 6.66
J (s) 3.04
1.00
1.32
1.11
1.12
1.10
1.13
1.08
3.13
f1 (ppm)
O
N
CH3
6b

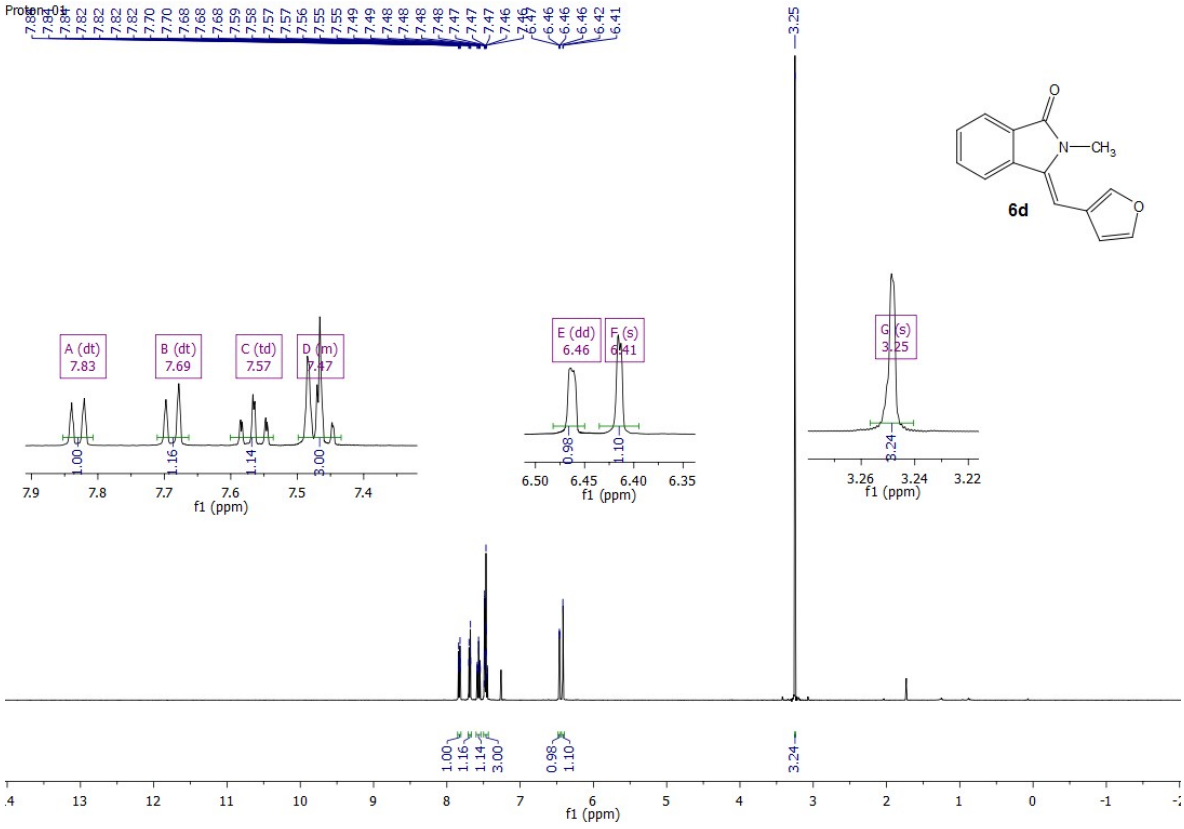

Carbon_01
168.91
150.47
148.59
138.04
137.75
136.85
132.27
131.13
129.61
128.59
123.49
123.08
119.53
101.93
30.76
f1 (ppm)
O
N
CH3
6b

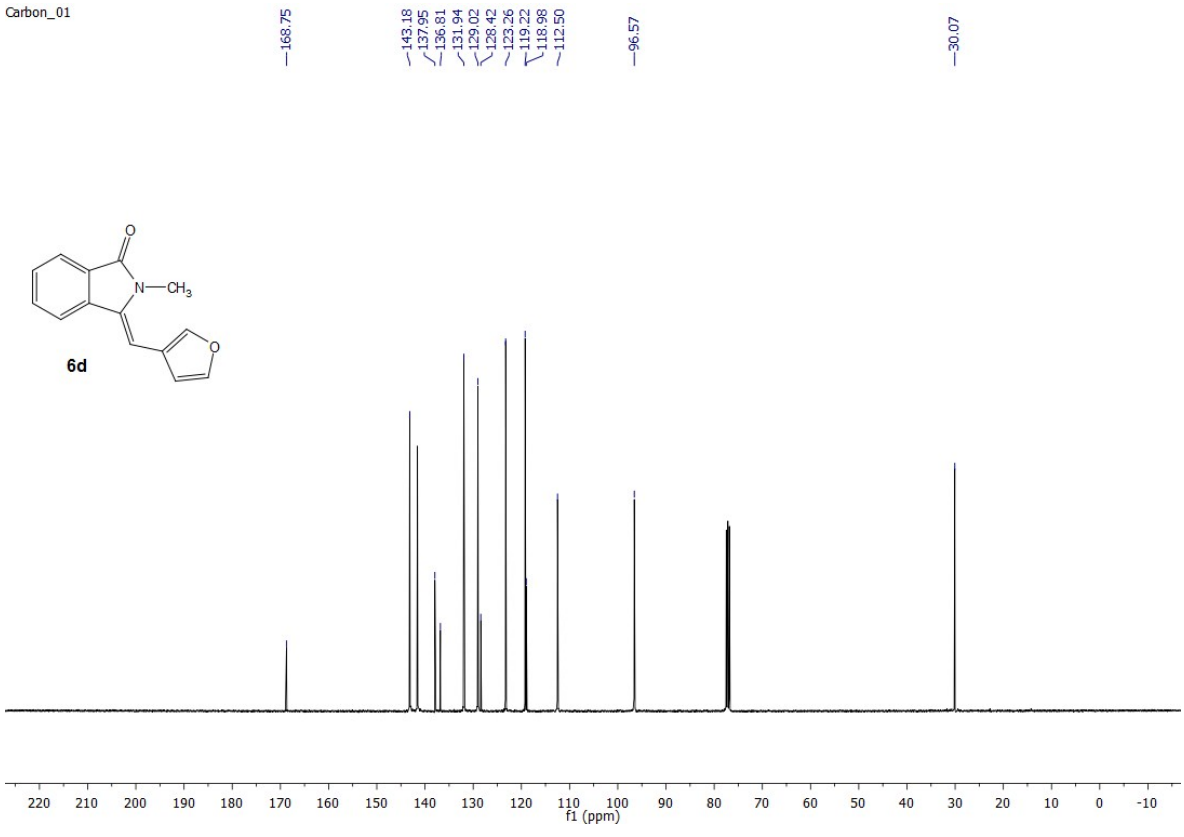

Noesy1d_01
O
N−Me
6b
H(b)
H
N
Proton_01
H(b)
2
1
8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 6.0 5.9
f1 (ppm)

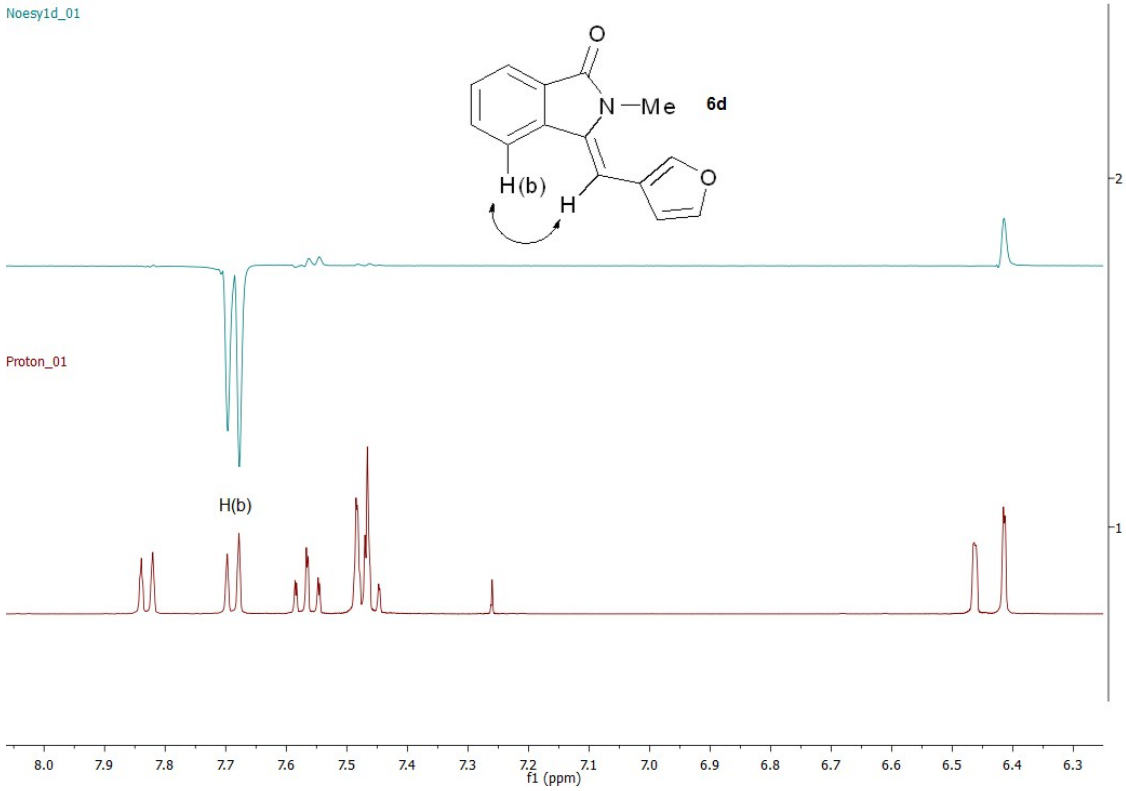

Proton_01
A (dt) 7.84
B (dt) 7.70
C (td) 7.58
D (td) 7.49
E (dd) 7.35
F (m) 7.05
G (dt) 7.02
1.00
1.17
1.17
1.15
0.95
1.08
1.06
7.8 7.7 7.6 7.5 7.4
7.08 7.06 7.04 7.02 7.00 6.98
f1 (ppm)
O
N−CH3
S
6c
H (s) 6.67
I (s) 3.22
1.10
3.13
6.66 6.60
3.23 3.22
f1 (ppm)
1.00 1.17 1.17 1.15 0.95 1.08 1.06 1.10
3.13
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)

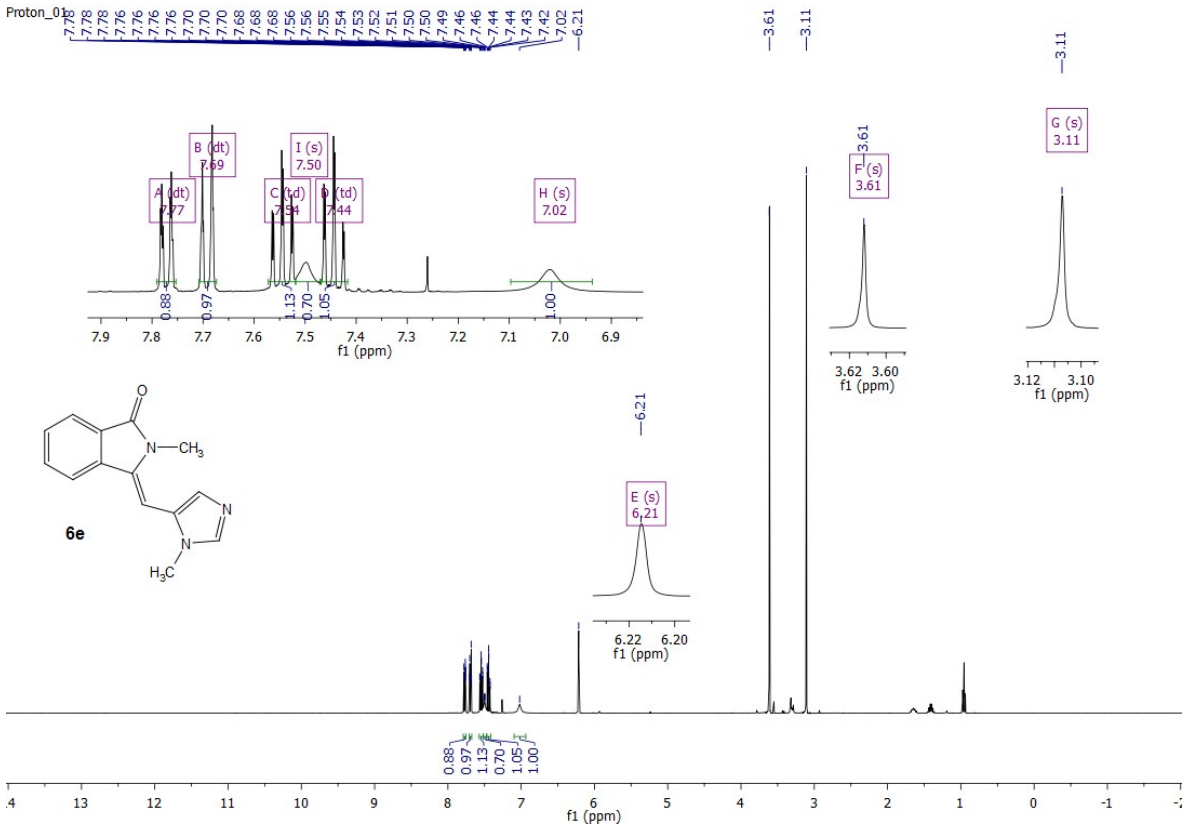

Carbon_01
168.81
137.82
137.31
136.45
132.06
129.29
128.86
128.41
127.32
126.61
123.36
119.41
98.63
30.15
O
N
CH3
S
6c
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

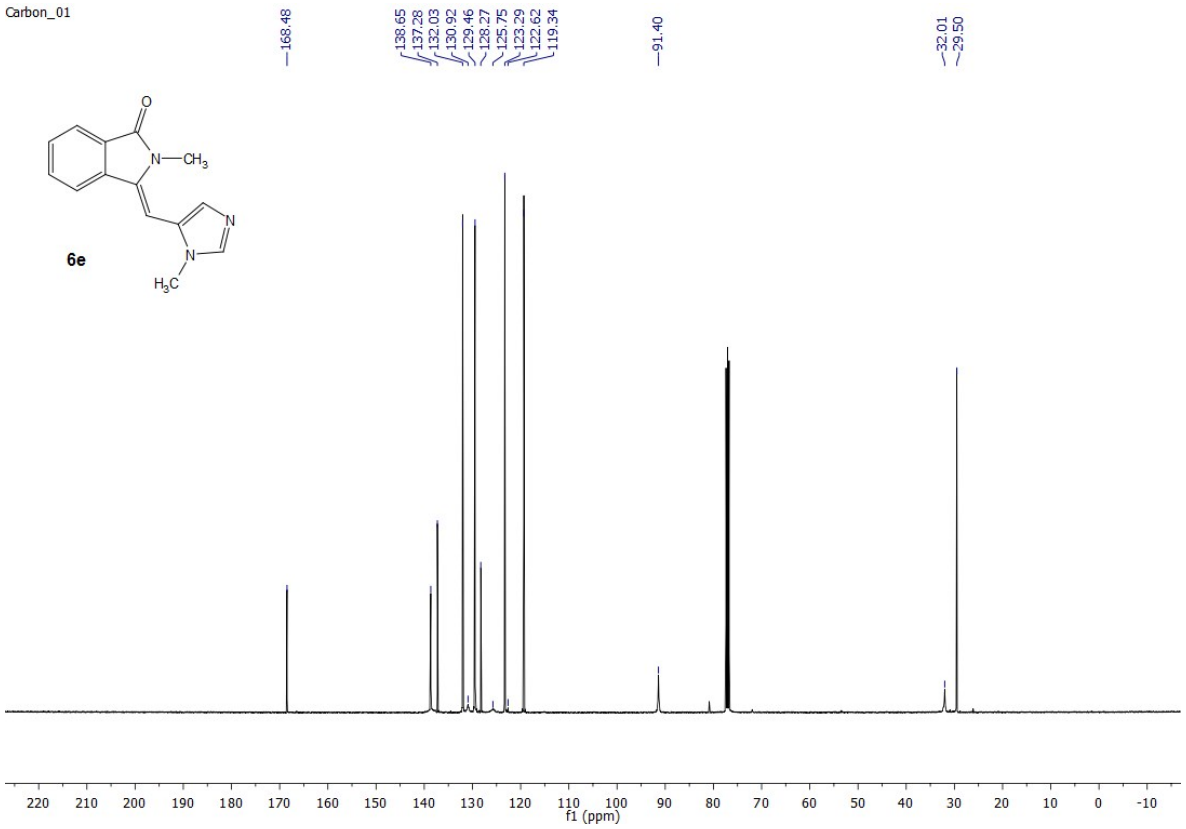

Noesy1d_01
O
N–Me
6c
S
H(b)
H
2
Proton_01
H(b)
1
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3
f1 (ppm)

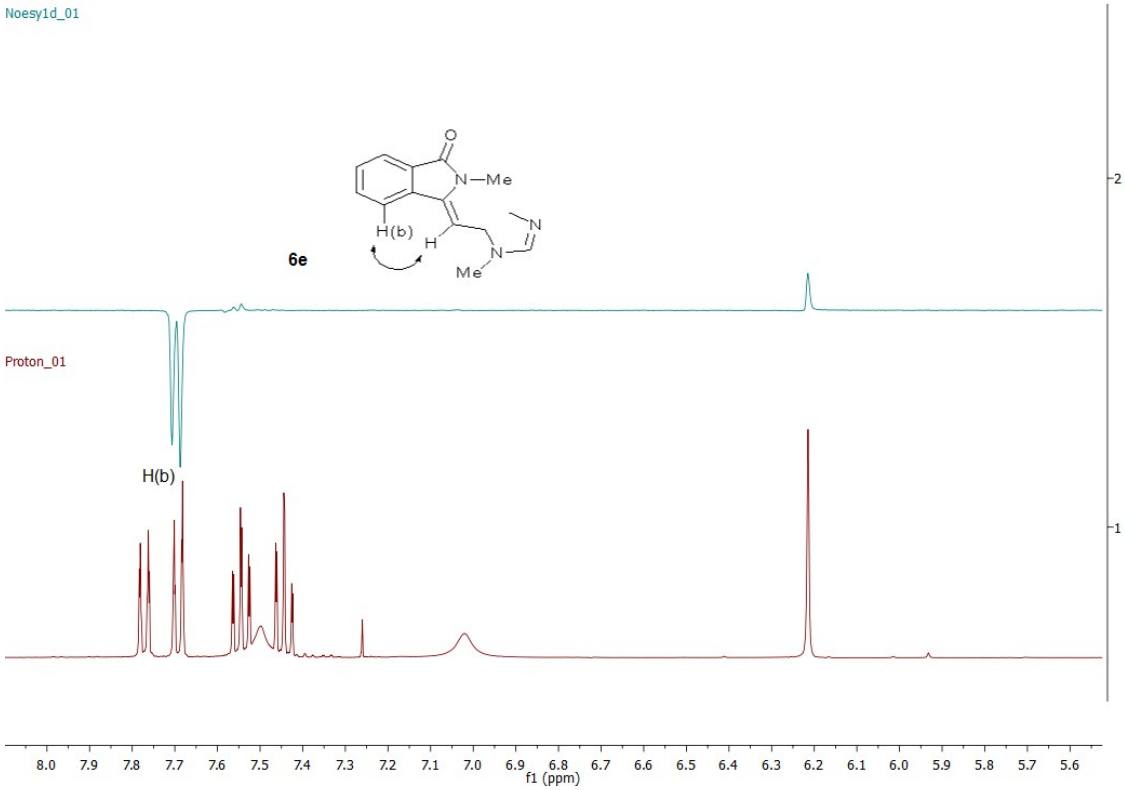

Proton_01
3.25
6d
A (dt) 7.83
B (dt) 7.69
C (td) 7.57
D (m) 7.47
E (dd) 6.46
F (s) 6.41
G (s) 3.25
f1 (ppm)

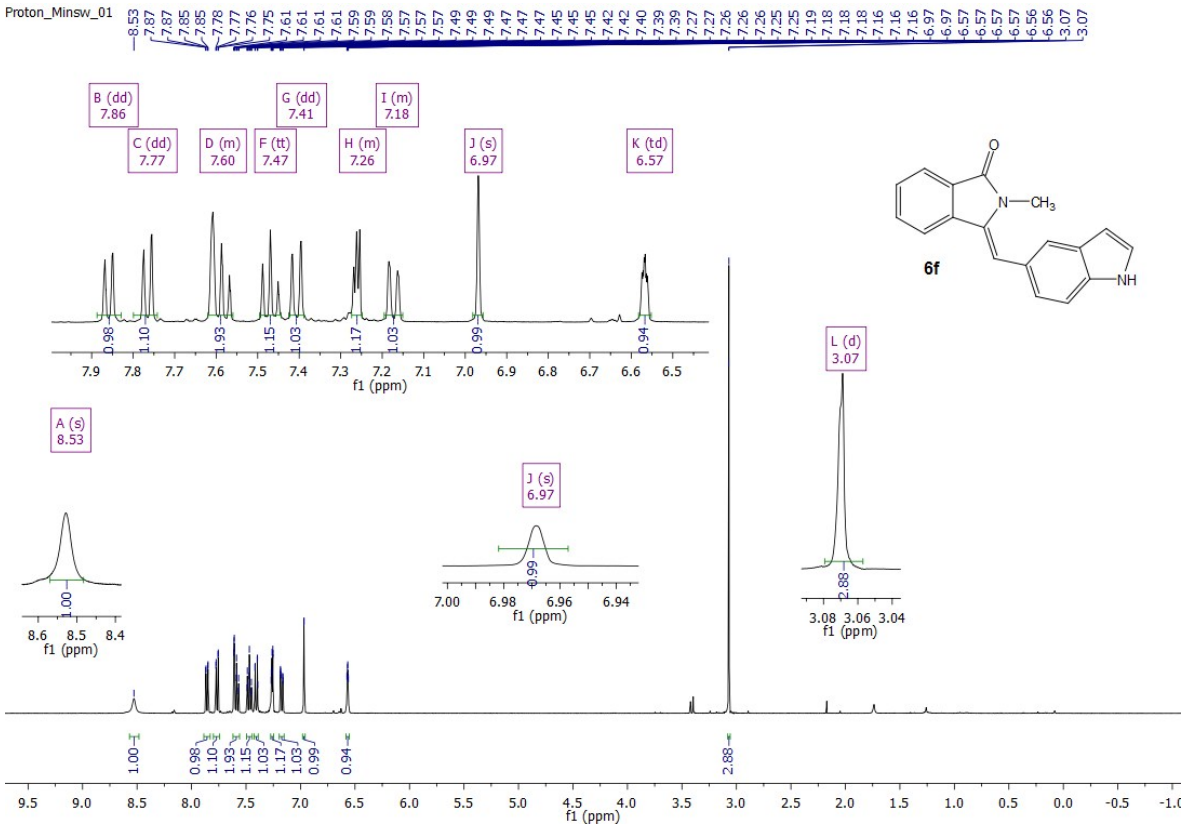

Carbon_01
168.75
143.18
137.95
136.81
131.94
129.02
128.42
123.26
119.22
118.98
112.50
96.57
30.07
6d
f1 (ppm)

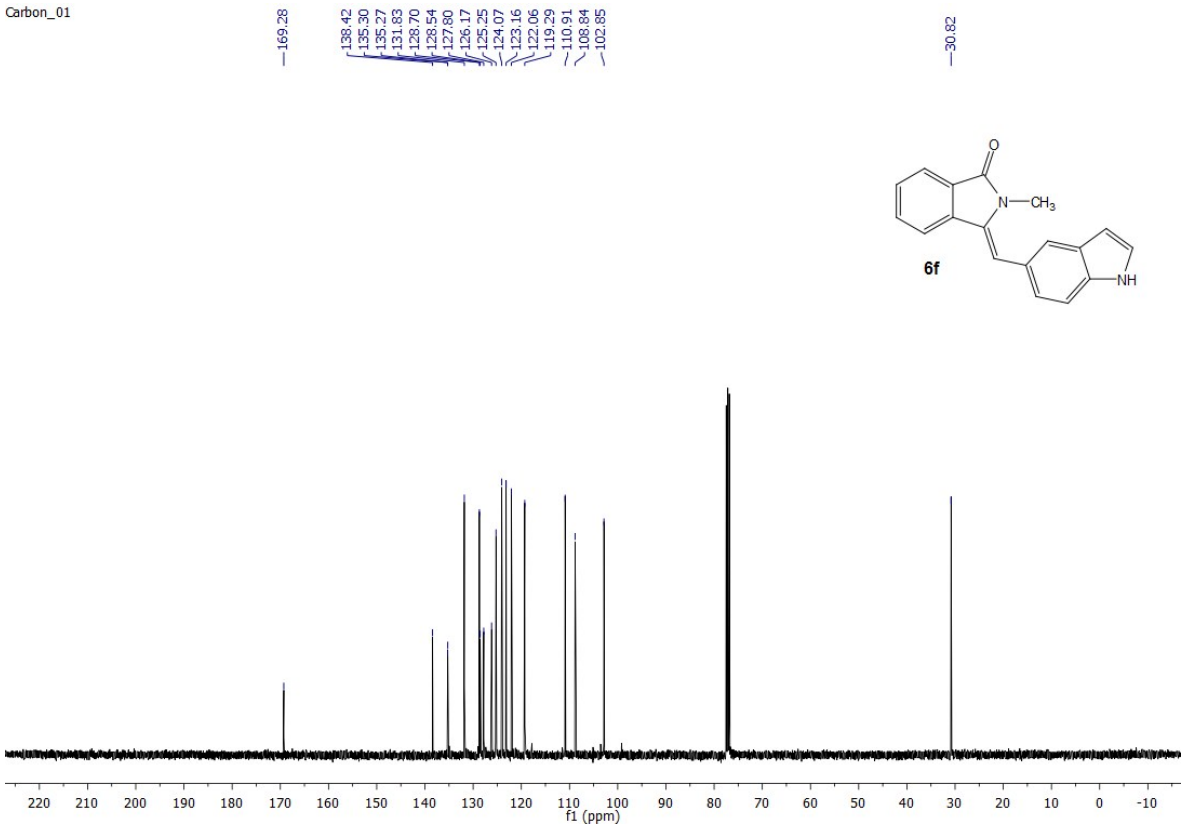

Noesy1d_01
O
N—Me
6d
H(b)
H
O
Proton_01
H(b)
2
1
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3
f1 (ppm)

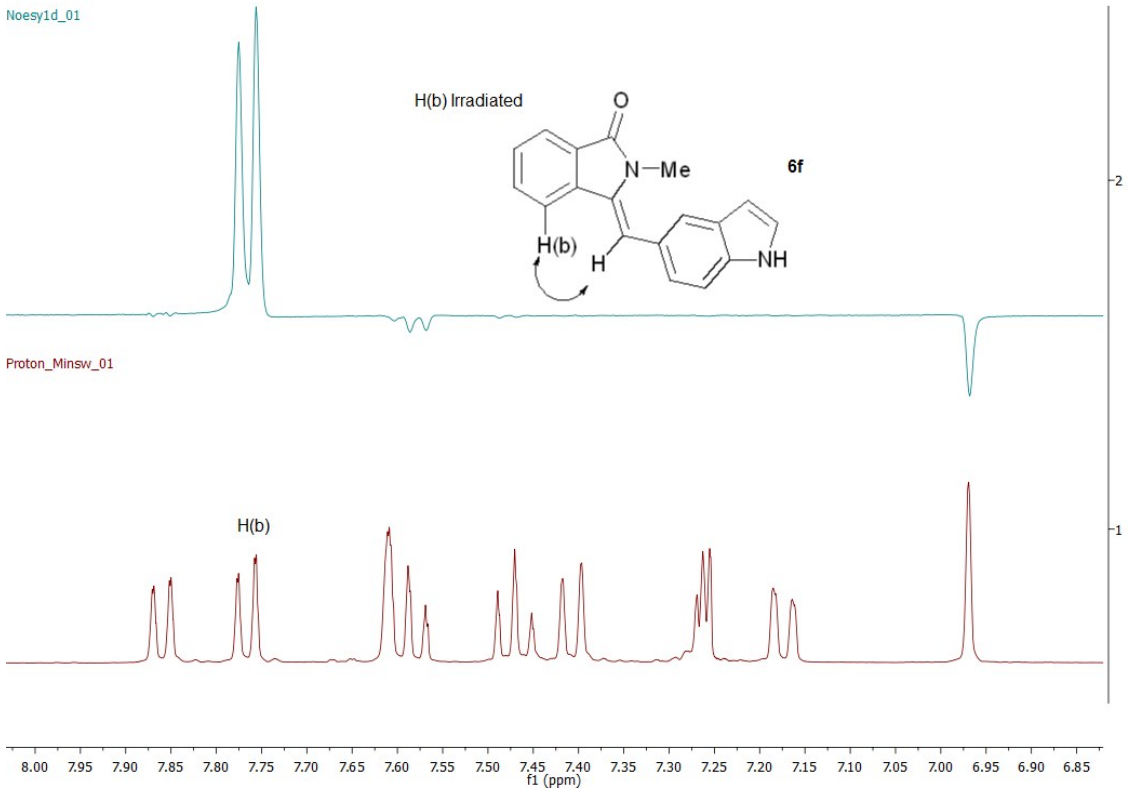

Proton_01
A (dt) 7.77
B (dt) 7.69
C (td) 7.54
D (td) 7.44
I (s) 7.50
H (s) 7.02
0.88 0.97 1.13 0.70 1.05 1.00
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
f1 (ppm)
3.61
3.11
F (s) 3.61
G (s) 3.11
3.62 3.60
3.12 3.10
f1 (ppm)
6.21
E (s) 6.21
6.22 6.20
f1 (ppm)
O
N—CH3
N
N
H3C
6e
0.88 0.97 1.13 0.70 1.05 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


Carbon_01
168.48
138.65
137.28
132.03
130.92
129.46
128.27
125.75
123.29
122.62
119.34
91.40
32.01
29.50
6e
f1 (ppm)


Noesy1d_01
Proton_01
H(b)
6e
f1 (ppm)


Proton_Minsw_01
B (dd) 7.86
C (dd) 7.77
D (m) 7.60
F (tt) 7.47
G (dd) 7.41
H (m) 7.26
I (m) 7.18
J (s) 6.97
K (td) 6.57
L (d) 3.07
A (s) 8.53
6f
f1 (ppm)


Carbon_01
169.28
138.42
135.30
135.27
131.83
128.70
128.54
127.80
126.17
125.25
124.07
123.16
122.06
119.29
110.91
108.84
102.85
30.82
6f
f1 (ppm)


Noesy1d_01
H(b) Irradiated
O
N—Me
6f
H(b)
H
NH
Proton_Minsw_01
H(b)
2
1
8.00 7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85
f1 (ppm)

Crystallographic data for product 4a, (Z)-3-benzylideneisoindolin-1-one.

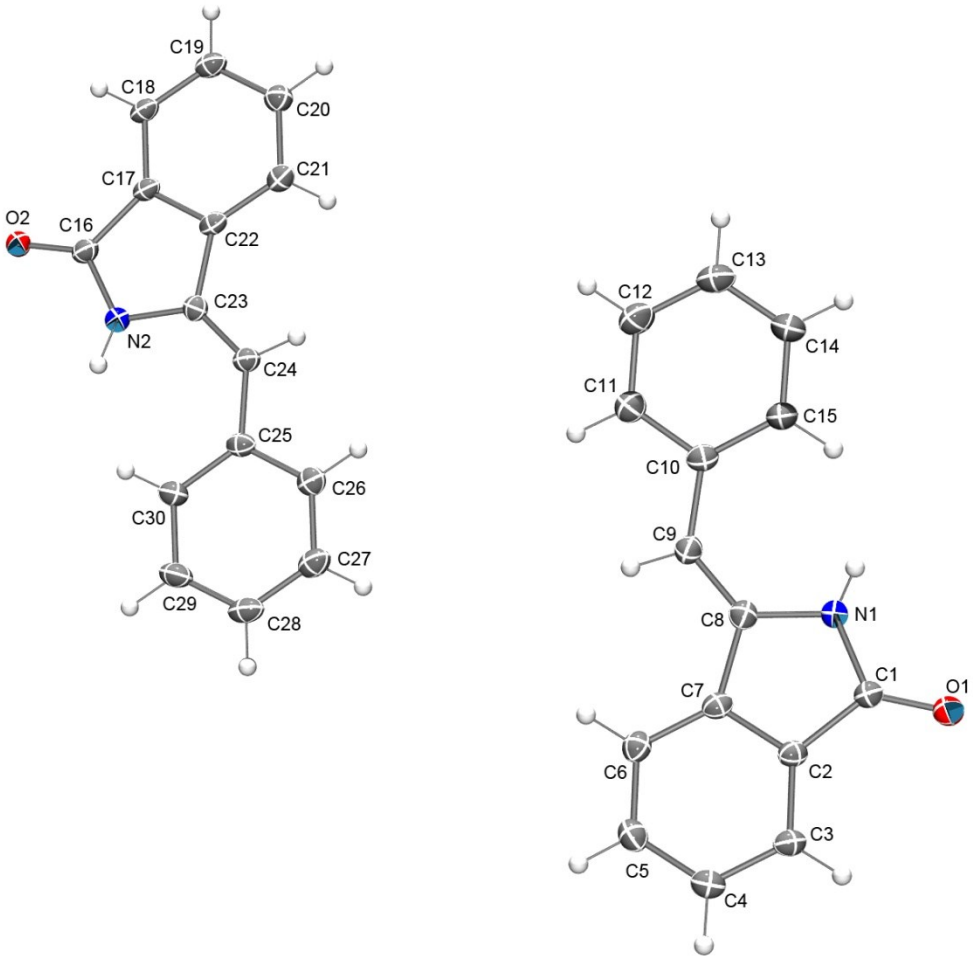


Table 1. Sample and crystal data for 4a

Identification code	4a-Z
Chemical formula	$C_{15}H_{11}NO$
Formula weight	221.25
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal size	0.236 x 0.324 x 0.353 mm
Crystal habit	clear pale yellow prism

Crystal system	monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 11.5374(6) Å	α = 90°
	b = 16.5896(9) Å	β = 90.3460(10)°
	c = 11.5369(6) Å	γ = 90°
Volume	2208.1(2) $Å^3$	
Z	8	
Density (calculated)	1.331 g/cm^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	928	

Table 2. Data collection and structure refinement for 4a.

Diffractometer	Bruker APEX DUO
Radiation source	fine-focus tube, MoKα
Theta range for data collection	1.76 to 30.51°
Index ranges	-16<=h<=16, -23<=k<=23, -16<=l<=16
Reflections collected	51671
Independent reflections	6712 [R(int) = 0.0432]
Coverage of independent reflections	99.6%
Absorption correction	multi-scan
Max. and min. transmission	0.9800 and 0.9710
Structure solution technique	direct methods
Structure solution program	SHELXTL XT 2013/1 (Sheldrick, 2013)
Refinement method	Full-matrix least-squares on F^2
Refinement program	SHELXTL XL 2013/2 (Bruker AXS, 2013)

Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	6712 / 0 / 315	
Goodness-of-fit on F^2	1.070	
Δ/σ_{max}	0.001	
Final R indices	5025 data; $I>2\sigma(I)$	R1 = 0.0650, wR2 = 0.1717
	all data	R1 = 0.0870, wR2 = 0.1849
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0938P)^2+1.3034P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.796 and -0.320 eÅ^{-3}	
R.M.S. deviation from mean	0.069 eÅ^{-3}	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for 4a.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.88474(14)	0.03315(10)	0.76100(14)	0.0204(3)
C2	0.83963(13)	0.98987(9)	0.65777(14)	0.0194(3)
C3	0.72964(14)	0.99050(10)	0.60777(15)	0.0222(3)
C4	0.70942(15)	0.94064(11)	0.51264(15)	0.0244(3)
C5	0.79837(16)	0.89201(11)	0.46920(15)	0.0254(3)
C6	0.90846(15)	0.89226(10)	0.51867(15)	0.0232(3)
C7	0.92893(13)	0.94173(10)	0.61409(14)	0.0196(3)
C8	0.03378(14)	0.95578(10)	0.68570(14)	0.0207(3)
C9	0.13864(14)	0.92334(10)	0.66606(15)	0.0219(3)
C10	0.25143(14)	0.93746(10)	0.72013(15)	0.0220(3)

	x/a	y/b	z/c	U(eq)
C11	0.34652(15)	0.89679(11)	0.67402(16)	0.0273(4)
C12	0.45822(16)	0.91025(12)	0.71531(18)	0.0325(4)
C13	0.47741(16)	0.96412(13)	0.80508(18)	0.0322(4)
C14	0.38448(17)	0.00349(14)	0.8532(2)	0.0394(5)
C15	0.27311(16)	0.99073(14)	0.81148(19)	0.0367(5)
C16	0.05473(14)	0.12382(10)	0.04285(14)	0.0218(3)
C17	0.11323(13)	0.17170(10)	0.13427(14)	0.0197(3)
C18	0.22807(13)	0.16976(11)	0.17222(14)	0.0222(3)
C19	0.26046(14)	0.22190(11)	0.26023(15)	0.0246(3)
C20	0.17931(14)	0.27442(11)	0.30915(15)	0.0241(3)
C21	0.06441(14)	0.27621(10)	0.27088(15)	0.0226(3)
C22	0.03186(13)	0.22410(10)	0.18206(14)	0.0193(3)
C23	0.91991(14)	0.21131(10)	0.12167(14)	0.0193(3)
C24	0.82259(14)	0.25211(10)	0.14481(14)	0.0206(3)
C25	0.70591(13)	0.24796(10)	0.09714(14)	0.0202(3)
C26	0.62347(15)	0.29840(11)	0.14978(15)	0.0241(3)
C27	0.50902(15)	0.29981(12)	0.11291(16)	0.0279(4)
C28	0.47363(15)	0.25042(12)	0.02187(16)	0.0287(4)
C29	0.55358(15)	0.20025(12)	0.96850(16)	0.0289(4)
C30	0.66853(15)	0.19845(11)	0.00540(15)	0.0256(3)
N1	0.99933(12)	0.01102(9)	0.77170(12)	0.0210(3)
N2	0.94072(12)	0.14949(9)	0.04020(12)	0.0218(3)
O1	0.83325(10)	0.08035(8)	0.82670(11)	0.0254(3)
O2	0.09711(10)	0.07168(8)	0.97957(10)	0.0242(3)

Table 4. Bond lengths (Å) for 4a.

C1-O1	1.243(2)	C1-N1	1.377(2)
C1-C2	1.482(2)	C2-C3	1.391(2)
C2-C7	1.400(2)	C3-C4	1.393(2)
C3-H3	0.95	C4-C5	1.401(2)
C4-H4	0.95	C5-C6	1.389(2)
C5-H5	0.95	C6-C7	1.392(2)
C6-H6	0.95	C7-C8	1.479(2)
C8-C9	1.345(2)	C8-N1	1.410(2)
C9-C10	1.459(2)	C9-H9	0.95
C10-C11	1.396(2)	C10-C15	1.397(3)
C11-C12	1.389(3)	C11-H11	0.95
C12-C13	1.385(3)	C12-H12	0.95
C13-C14	1.375(3)	C13-H13	0.95
C14-C15	1.386(3)	C14-H14	0.95
C15-H15	0.95	C16-O2	1.235(2)
C16-N2	1.383(2)	C16-C17	1.480(2)
C17-C18	1.393(2)	C17-C22	1.395(2)
C18-C19	1.384(3)	C18-H18	0.95
C19-C20	1.400(2)	C19-H19	0.95
C20-C21	1.395(2)	C20-H20	0.95
C21-C22	1.391(2)	C21-H21	0.95
C22-C23	1.479(2)	C23-C24	1.339(2)
C23-N2	1.413(2)	C24-C25	1.453(2)
C24-H24	0.95	C25-C30	1.405(2)
C25-C26	1.407(2)	C26-C27	1.385(2)
C26-H26	0.95	C27-C28	1.391(3)

C27-H27	0.95	C28-C29	1.389(3)
C28-H28	0.95	C29-C30	1.391(2)
C29-H29	0.95	C30-H30	0.95
N1-H1N	0.819(18)	N2-H2N	0.83(2)

Table 5. Bond angles (°) for 4a.

O1-C1-N1	125.11(15)	O1-C1-C2	128.92(15)
N1-C1-C2	105.97(13)	C3-C2-C7	121.78(15)
C3-C2-C1	130.14(15)	C7-C2-C1	108.06(14)
C2-C3-C4	118.06(15)	C2-C3-H3	121.0
C4-C3-H3	121.0	C3-C4-C5	120.29(16)
C3-C4-H4	119.9	C5-C4-H4	119.9
C6-C5-C4	121.43(16)	C6-C5-H5	119.3
C4-C5-H5	119.3	C5-C6-C7	118.48(15)
C5-C6-H6	120.8	C7-C6-H6	120.8
C6-C7-C2	119.95(15)	C6-C7-C8	131.98(15)
C2-C7-C8	108.07(14)	C9-C8-N1	129.54(16)
C9-C8-C7	125.20(15)	N1-C8-C7	105.21(13)
C8-C9-C10	131.69(16)	C8-C9-H9	114.2
C10-C9-H9	114.2	C11-C10-C15	117.11(16)
C11-C10-C9	117.43(16)	C15-C10-C9	125.41(15)
C12-C11-C10	121.41(17)	C12-C11-H11	119.3
C10-C11-H11	119.3	C13-C12-C11	120.26(17)
C13-C12-H12	119.9	C11-C12-H12	119.9
C14-C13-C12	119.13(17)	C14-C13-H13	120.4
C12-C13-H13	120.4	C13-C14-C15	120.74(19)

C13-C14-H14	119.6	C15-C14-H14	119.6
C14-C15-C10	121.33(18)	C14-C15-H15	119.3
C10-C15-H15	119.3	O2-C16-N2	125.64(16)
O2-C16-C17	128.08(15)	N2-C16-C17	106.27(14)
C18-C17-C22	122.07(15)	C18-C17-C16	129.82(15)
C22-C17-C16	108.11(13)	C19-C18-C17	117.93(15)
C19-C18-H18	121.0	C17-C18-H18	121.0
C18-C19-C20	120.43(15)	C18-C19-H19	119.8
C20-C19-H19	119.8	C21-C20-C19	121.47(16)
C21-C20-H20	119.3	C19-C20-H20	119.3
C22-C21-C20	118.16(15)	C22-C21-H21	120.9
C20-C21-H21	120.9	C21-C22-C17	119.94(14)
C21-C22-C23	131.87(14)	C17-C22-C23	108.19(14)
C24-C23-N2	130.21(15)	C24-C23-C22	124.35(15)
N2-C23-C22	105.43(13)	C23-C24-C25	132.58(16)
C23-C24-H24	113.7	C25-C24-H24	113.7
C30-C25-C26	117.86(15)	C30-C25-C24	126.32(15)
C26-C25-C24	115.82(15)	C27-C26-C25	121.54(16)
C27-C26-H26	119.2	C25-C26-H26	119.2
C26-C27-C28	119.79(17)	C26-C27-H27	120.1
C28-C27-H27	120.1	C29-C28-C27	119.66(16)
C29-C28-H28	120.2	C27-C28-H28	120.2
C28-C29-C30	120.78(17)	C28-C29-H29	119.6
C30-C29-H29	119.6	C29-C30-C25	120.38(17)
C29-C30-H30	119.8	C25-C30-H30	119.8
C1-N1-C8	112.64(14)	C1-N1-H1N	135.0(12)

C8-N1-H1N	111.5(12)	C16-N2-C23	111.98(14)
C16-N2-H2N	131.3(14)	C23-N2-H2N	116.7(14)

Table 6. Anisotropic atomic displacement parameters (Å^2) for 4a.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0178(7)	0.0218(7)	0.0215(7)	0.0008(6)	-0.0001(5)	0.0005(6)
C2	0.0169(7)	0.0171(7)	0.0241(8)	0.0006(6)	-0.0004(5)	-0.0005(5)
C3	0.0172(7)	0.0217(8)	0.0276(8)	0.0014(6)	-0.0019(6)	0.0002(6)
C4	0.0222(8)	0.0245(8)	0.0265(8)	0.0017(6)	-0.0034(6)	-0.0021(6)
C5	0.0287(8)	0.0246(8)	0.0227(8)	-0.0026(6)	0.0002(6)	-0.0036(7)
C6	0.0229(8)	0.0230(8)	0.0237(8)	-0.0001(6)	0.0042(6)	-0.0007(6)
C7	0.0169(7)	0.0180(7)	0.0239(8)	0.0028(6)	0.0017(5)	-0.0016(5)
C8	0.0219(7)	0.0184(7)	0.0220(8)	0.0002(6)	0.0018(6)	-0.0010(6)
C9	0.0204(7)	0.0215(7)	0.0239(8)	-0.0029(6)	0.0021(6)	-0.0005(6)
C10	0.0186(7)	0.0222(8)	0.0253(8)	0.0025(6)	0.0002(6)	-0.0016(6)
C11	0.0252(8)	0.0271(8)	0.0295(9)	-0.0024(7)	-0.0006(7)	0.0023(7)
C12	0.0236(8)	0.0341(10)	0.0397(11)	-0.0015(8)	-0.0010(7)	0.0069(7)
C13	0.0205(8)	0.0388(10)	0.0372(10)	0.0013(8)	-0.0044(7)	0.0007(7)
C14	0.0233(9)	0.0508(13)	0.0440(12)	-0.0192(10)	-0.0039(8)	-0.0034(8)
C15	0.0187(8)	0.0470(12)	0.0445(12)	-0.0211(9)	-0.0009(7)	-0.0004(8)
C16	0.0168(7)	0.0274(8)	0.0213(8)	0.0010(6)	0.0005(5)	0.0016(6)
C17	0.0138(7)	0.0248(8)	0.0203(7)	0.0038(6)	0.0011(5)	0.0012(5)
C18	0.0147(7)	0.0286(8)	0.0234(8)	0.0043(6)	0.0016(6)	0.0034(6)
C19	0.0169(7)	0.0320(9)	0.0249(8)	0.0062(7)	-0.0009(6)	0.0024(6)
C20	0.0215(8)	0.0279(8)	0.0230(8)	0.0008(6)	-0.0010(6)	0.0006(6)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C21	0.0203(7)	0.0253(8)	0.0221(8)	0.0034(6)	0.0024(6)	0.0028(6)
C22	0.0147(6)	0.0236(7)	0.0197(7)	0.0057(6)	0.0019(5)	0.0011(5)
C23	0.0197(7)	0.0211(7)	0.0172(7)	0.0013(5)	0.0013(5)	-0.0014(6)
C24	0.0172(7)	0.0238(8)	0.0208(7)	0.0012(6)	-0.0007(5)	0.0000(6)
C25	0.0174(7)	0.0240(8)	0.0191(7)	0.0053(6)	-0.0023(5)	-0.0019(6)
C26	0.0240(8)	0.0275(8)	0.0209(8)	0.0032(6)	0.0003(6)	0.0008(6)
C27	0.0219(8)	0.0353(9)	0.0265(9)	0.0059(7)	0.0012(6)	0.0047(7)
C28	0.0183(7)	0.0409(10)	0.0270(9)	0.0088(7)	-0.0027(6)	-0.0018(7)
C29	0.0218(8)	0.0389(10)	0.0259(9)	0.0011(7)	-0.0060(6)	-0.0061(7)
C30	0.0202(8)	0.0316(9)	0.0250(8)	-0.0020(7)	-0.0034(6)	-0.0020(6)
N1	0.0192(6)	0.0226(7)	0.0213(7)	-0.0039(5)	0.0002(5)	0.0008(5)
N2	0.0183(6)	0.0269(7)	0.0202(7)	-0.0035(5)	-0.0001(5)	0.0008(5)
O1	0.0227(6)	0.0269(6)	0.0267(6)	-0.0035(5)	-0.0044(5)	0.0050(5)
O2	0.0186(5)	0.0317(7)	0.0222(6)	-0.0064(5)	-0.0016(4)	0.0051(5)

Table 7. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for 4a.

	x/a	y/b	z/c	U(eq)
H3	-0.3300	1.0240	0.6376	0.027
H4	-0.3650	0.9396	0.4771	0.029
H5	-0.2169	0.8581	0.4045	0.03
H6	-0.0316	0.8594	0.4881	0.028
H9	0.1394	0.8841	0.6061	0.026
H11	0.3346	0.8591	0.6131	0.033
H12	0.5217	0.8824	0.6818	0.039
H13	0.5538	0.9737	0.8331	0.039

	x/a	y/b	z/c	U(eq)
H14	0.3968	1.0399	0.9157	0.047
H15	0.2102	1.0188	0.8457	0.044
H18	1.2824	0.1338	0.1387	0.027
H19	1.3383	0.2221	0.2877	0.03
H20	1.2030	0.3096	0.3698	0.029
H21	1.0099	0.3120	0.3046	0.027
H24	0.8316	0.2915	0.2040	0.025
H26	0.6469	0.3323	0.2121	0.029
H27	0.4549	0.3344	0.1497	0.033
H28	0.3952	0.2510	-0.0037	0.034
H29	0.5295	0.1668	-0.0939	0.035
H30	0.7221	0.1636	-0.0316	0.031
H1N	0.0497(15)	1.0169(10)	0.8214(15)	0.004(4)
H2N	0.8852(18)	0.1362(12)	-0.0020(18)	0.017(5)

Crystallographic data for product (Z)-3-(cyclopropylmethylene)-2-methylisoindolin-1-one, 4y

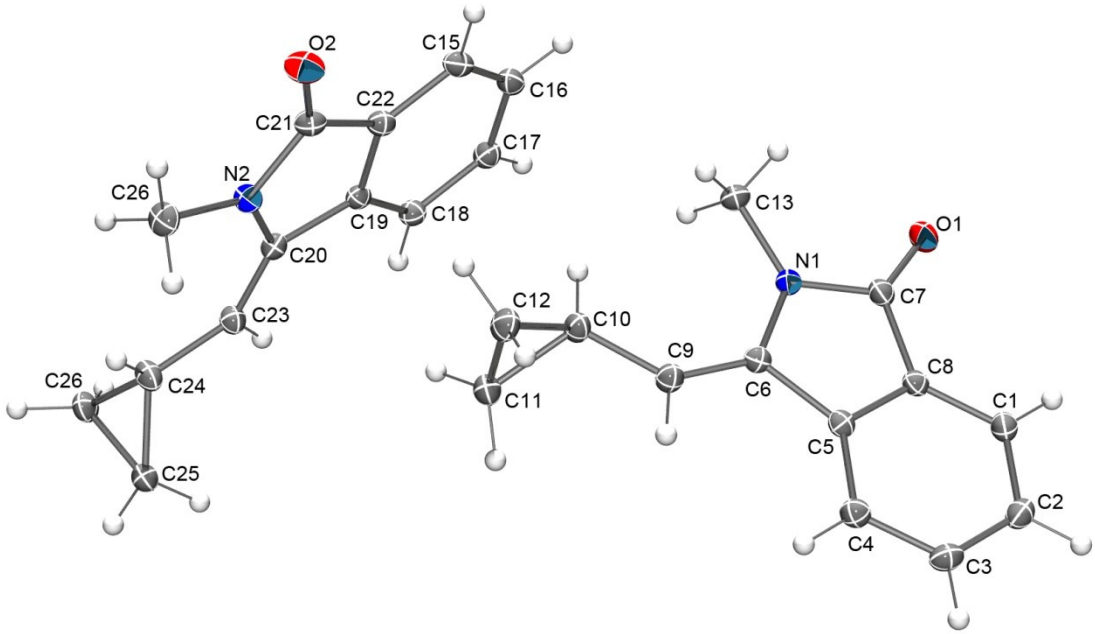


Table 1. Sample and crystal data for 4y.

Identification code	Socrat061114	
Chemical formula	$C_{13}H_{13}NO$	
Formula weight	199.24	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal size	0.240 x 0.248 x 0.602 mm	
Crystal habit	clear colourless prism	
Crystal system	triclinic	
Space group	P -1	
Unit cell dimensions	a = 7.6694(8) Å	α = 89.9200(19)°
	b = 8.4024(8) Å	β = 88.182(2)°
	c = 15.7611(16) Å	γ = 88.4170(19)°
Volume	1014.77(18) $Å^3$	

Z	4
Density (calculated)	1.304 g/cm^3
Absorption coefficient	0.083 mm^{-1}
F(000)	424

Table 2. Data collection and structure refinement for 4y.

Diffractometer	Bruker APEX DUO	
Radiation source	fine-focus tube, MoKα	
Theta range for data collection	1.29 to 30.62°	
Index ranges	-10<=h<=10, -11<=k<=11, -22<=l<=22	
Reflections collected	21818	
Independent reflections	6087 [R(int) = 0.0345]	
Coverage of independent reflections	97.6%	
Absorption correction	multi-scan	
Max. and min. transmission	0.9800 and 0.9520	
Structure solution technique	direct methods	
Structure solution program	SHELXTL XT 2013/6 (Sheldrick, 2013)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXTL XLMP 2014/1 (Bruker AXS, 2013)	
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	6087 / 0 / 273	
Goodness-of-fit on F^2	1.019	
Δ/σ_{max}	0.001	
Final R indices	4887 data; I>2σ(I)	R1 = 0.0428, wR2 = 0.1032

	all data	R1 = 0.0565, wR2 = 0.1121
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0537P)^2+0.2789P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.356 and -0.256 eÅ^{-3}	
R.M.S. deviation from mean	0.050 eÅ^{-3}	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for 4y.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.39120(14)	0.40019(14)	0.66433(7)	0.0181(2)
C2	0.41399(15)	0.23606(14)	0.66180(7)	0.0196(2)
C3	0.37768(15)	0.15047(13)	0.58848(7)	0.0198(2)
C4	0.31740(14)	0.22734(13)	0.51649(7)	0.0179(2)
C5	0.28950(13)	0.39223(13)	0.51952(6)	0.0146(2)
C6	0.22620(13)	0.50684(12)	0.45602(7)	0.0146(2)
C7	0.28549(14)	0.64630(13)	0.57829(7)	0.0166(2)
C8	0.32704(14)	0.47588(12)	0.59278(6)	0.0149(2)
C9	0.18682(14)	0.47003(13)	0.37621(7)	0.0170(2)
C10	0.14033(15)	0.57070(13)	0.30327(7)	0.0182(2)
C11	0.19740(15)	0.50802(14)	0.21602(7)	0.0206(2)
C12	0.00827(15)	0.50877(14)	0.24249(7)	0.0211(2)
C13	0.15909(15)	0.80688(13)	0.46264(7)	0.0190(2)
C15	0.91844(14)	0.00819(13)	0.23691(7)	0.0176(2)
C16	0.06193(15)	0.06048(13)	0.27973(7)	0.0186(2)

	x/a	y/b	z/c	U(eq)
C17	0.23177(15)	0.03223(13)	0.24659(7)	0.0180(2)
C18	0.26312(14)	0.95073(13)	0.17044(7)	0.0164(2)
C19	0.11880(13)	0.89895(12)	0.12741(6)	0.01416(19)
C20	0.10692(13)	0.81167(12)	0.04693(6)	0.0146(2)
C21	0.82557(14)	0.85831(13)	0.10248(7)	0.0169(2)
C22	0.95009(14)	0.92759(12)	0.16072(7)	0.0153(2)
C23	0.24791(14)	0.67446(14)	0.91622(7)	0.0187(2)
C24	0.40472(15)	0.56552(14)	0.89528(7)	0.0190(2)
C25	0.38275(15)	0.71842(14)	0.84808(7)	0.0202(2)
C26	0.84401(15)	0.71472(14)	0.96606(7)	0.0206(2)
C00F	0.24355(14)	0.76316(13)	0.99669(7)	0.0167(2)
N1	0.22585(12)	0.65708(11)	0.49646(6)	0.01541(18)
N2	0.92571(12)	0.79103(11)	0.03666(6)	0.01648(18)
O1	0.29704(12)	0.75881(10)	0.62716(5)	0.02401(19)
O2	0.66573(10)	0.85621(10)	0.10891(6)	0.02389(19)

Table 4. Bond lengths (Å) for 4y.

C1-C2	1.3858(16)	C1-C8	1.3908(14)
C1-H1	0.95	C2-C3	1.4014(16)
C2-H2	0.95	C3-C4	1.3904(15)
C3-H3	0.95	C4-C5	1.3967(15)
C4-H4	0.95	C5-C8	1.3946(14)
C5-C6	1.4720(14)	C6-C9	1.3416(15)
C6-N1	1.4148(13)	C7-O1	1.2271(13)
C7-N1	1.3841(13)	C7-C8	1.4774(15)
C9-C10	1.4733(15)	C9-H9	0.95

C10-C12	1.5187(15)	C10-C11	1.5204(15)
C10-H10	1.0	C11-C12	1.4964(17)
C11-H11A	0.99	C11-H11B	0.99
C12-H12A	0.99	C12-H12B	0.99
C13-N1	1.4528(14)	C13-H13A	0.98
C13-H13B	0.98	C13-H13C	0.98
C15-C16	1.3907(16)	C15-C22	1.3908(15)
C15-H15	0.95	C16-C17	1.4021(16)
C16-H16	0.95	C17-C18	1.3936(15)
C17-H17	0.95	C18-C19	1.3963(14)
C18-H18	0.95	C19-C22	1.3956(14)
C19-C20	1.4726(14)	C20-C00F	1.3476(15)
C20-N2	1.4196(13)	C21-O2	1.2275(13)
C21-N2	1.3830(14)	C21-C22	1.4769(15)
C23-C00F	1.4709(15)	C23-C24	1.5192(16)
C23-C25	1.5194(15)	C23-H23	1.0
C24-C25	1.4919(16)	C24-H24A	0.99
C24-H24B	0.99	C25-H25A	0.99
C25-H25B	0.99	C26-N2	1.4527(13)
C26-H26A	0.98	C26-H26B	0.98
C26-H26C	0.98	C00F-H00F	0.95

Table 5. Bond angles (°) for 4y.

C2-C1-C8	117.83(10)	C2-C1-H1	121.1
C8-C1-H1	121.1	C1-C2-C3	120.74(10)
C1-C2-H2	119.6	C3-C2-H2	119.6
C4-C3-C2	121.11(10)	C4-C3-H3	119.4

C2-C3-H3	119.4	C3-C4-C5	118.38(10)
C3-C4-H4	120.8	C5-C4-H4	120.8
C8-C5-C4	119.83(10)	C8-C5-C6	108.36(9)
C4-C5-C6	131.80(10)	C9-C6-N1	129.62(10)
C9-C6-C5	124.96(10)	N1-C6-C5	105.35(8)
O1-C7-N1	125.09(10)	O1-C7-C8	128.87(10)
N1-C7-C8	106.03(9)	C1-C8-C5	122.07(10)
C1-C8-C7	129.70(10)	C5-C8-C7	108.23(9)
C6-C9-C10	131.60(10)	C6-C9-H9	114.2
C10-C9-H9	114.2	C9-C10-C12	118.02(10)
C9-C10-C11	116.33(10)	C12-C10-C11	59.00(7)
C9-C10-H10	117.0	C12-C10-H10	117.0
C11-C10-H10	117.0	C12-C11-C10	60.45(7)
C12-C11-H11A	117.7	C10-C11-H11A	117.7
C12-C11-H11B	117.7	C10-C11-H11B	117.7
H11A-C11-H11B	114.8	C11-C12-C10	60.56(7)
C11-C12-H12A	117.7	C10-C12-H12A	117.7
C11-C12-H12B	117.7	C10-C12-H12B	117.7
H12A-C12-H12B	114.8	N1-C13-H13A	109.5
N1-C13-H13B	109.5	H13A-C13-H13B	109.5
N1-C13-H13C	109.5	H13A-C13-H13C	109.5
H13B-C13-H13C	109.5	C16-C15-C22	117.61(10)
C16-C15-H15	121.2	C22-C15-H15	121.2
C15-C16-C17	120.66(10)	C15-C16-H16	119.7
C17-C16-H16	119.7	C18-C17-C16	121.60(10)
C18-C17-H17	119.2	C16-C17-H17	119.2

C17-C18-C19	117.63(10)	C17-C18-H18	121.2
C19-C18-H18	121.2	C22-C19-C18	120.47(10)
C22-C19-C20	108.42(9)	C18-C19-C20	131.10(10)
C00F-C20-N2	129.50(10)	C00F-C20-C19	125.39(9)
N2-C20-C19	105.10(9)	O2-C21-N2	125.06(10)
O2-C21-C22	128.99(10)	N2-C21-C22	105.94(9)
C15-C22-C19	122.03(10)	C15-C22-C21	129.62(10)
C19-C22-C21	108.35(9)	C00F-C23-C24	118.57(9)
C00F-C23-C25	118.26(10)	C24-C23-C25	58.81(7)
C00F-C23-H23	116.3	C24-C23-H23	116.3
C25-C23-H23	116.3	C25-C24-C23	60.60(7)
C25-C24-H24A	117.7	C23-C24-H24A	117.7
C25-C24-H24B	117.7	C23-C24-H24B	117.7
H24A-C24-H24B	114.8	C24-C25-C23	60.59(7)
C24-C25-H25A	117.7	C23-C25-H25A	117.7
C24-C25-H25B	117.7	C23-C25-H25B	117.7
H25A-C25-H25B	114.8	N2-C26-H26A	109.5
N2-C26-H26B	109.5	H26A-C26-H26B	109.5
N2-C26-H26C	109.5	H26A-C26-H26C	109.5
H26B-C26-H26C	109.5	C20-C00F-C23	130.22(10)
C20-C00F-H00F	114.9	C23-C00F-H00F	114.9
C7-N1-C6	112.01(9)	C7-N1-C13	121.31(9)
C6-N1-C13	126.49(9)	C21-N2-C20	112.18(9)
C21-N2-C26	120.75(9)	C20-N2-C26	127.05(9)

Table 6. Anisotropic atomic displacement parameters ($Å^2$) for 4y.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0192(5)	0.0205(5)	0.0146(5)	0.0002(4)	-0.0001(4)	0.0005(4)
C2	0.0190(5)	0.0203(5)	0.0194(5)	0.0046(4)	-0.0009(4)	0.0011(4)
C3	0.0198(5)	0.0144(5)	0.0250(5)	0.0018(4)	0.0000(4)	0.0006(4)
C4	0.0197(5)	0.0150(5)	0.0190(5)	-0.0013(4)	-0.0006(4)	-0.0017(4)
C5	0.0131(5)	0.0159(5)	0.0147(4)	0.0000(4)	0.0015(3)	-0.0017(4)
C6	0.0144(5)	0.0134(5)	0.0161(5)	0.0001(4)	0.0008(4)	-0.0015(4)
C7	0.0192(5)	0.0165(5)	0.0141(5)	-0.0010(4)	0.0020(4)	-0.0001(4)
C8	0.0154(5)	0.0147(5)	0.0144(5)	-0.0004(4)	0.0020(4)	-0.0005(4)
C9	0.0195(5)	0.0146(5)	0.0171(5)	0.0006(4)	-0.0018(4)	-0.0014(4)
C10	0.0235(5)	0.0156(5)	0.0156(5)	-0.0007(4)	-0.0033(4)	-0.0009(4)
C11	0.0251(6)	0.0210(5)	0.0156(5)	-0.0002(4)	0.0002(4)	0.0013(4)
C12	0.0226(6)	0.0225(6)	0.0185(5)	-0.0001(4)	-0.0057(4)	-0.0002(4)
C13	0.0222(5)	0.0149(5)	0.0196(5)	0.0008(4)	-0.0007(4)	0.0031(4)
C15	0.0175(5)	0.0148(5)	0.0200(5)	0.0006(4)	0.0033(4)	0.0012(4)
C16	0.0241(5)	0.0149(5)	0.0166(5)	-0.0007(4)	0.0008(4)	0.0019(4)
C17	0.0201(5)	0.0157(5)	0.0182(5)	-0.0006(4)	-0.0040(4)	0.0001(4)
C18	0.0149(5)	0.0170(5)	0.0173(5)	0.0002(4)	-0.0007(4)	0.0005(4)
C19	0.0157(5)	0.0120(4)	0.0146(4)	0.0016(4)	-0.0001(4)	0.0003(4)
C20	0.0143(5)	0.0147(5)	0.0150(5)	0.0014(4)	-0.0016(4)	-0.0010(4)
C21	0.0146(5)	0.0152(5)	0.0208(5)	0.0005(4)	0.0005(4)	0.0001(4)
C22	0.0153(5)	0.0133(5)	0.0174(5)	0.0006(4)	0.0006(4)	-0.0001(4)
C23	0.0163(5)	0.0242(6)	0.0156(5)	-0.0042(4)	-0.0006(4)	-0.0010(4)
C24	0.0223(5)	0.0190(5)	0.0154(5)	-0.0010(4)	0.0007(4)	0.0017(4)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C25	0.0255(6)	0.0210(5)	0.0139(5)	0.0006(4)	0.0025(4)	0.0005(4)
C26	0.0173(5)	0.0242(6)	0.0208(5)	-0.0031(4)	-0.0051(4)	-0.0031(4)
C00F	0.0164(5)	0.0184(5)	0.0152(5)	-0.0008(4)	-0.0011(4)	-0.0011(4)
N1	0.0190(4)	0.0131(4)	0.0141(4)	-0.0001(3)	-0.0001(3)	0.0005(3)
N2	0.0139(4)	0.0185(4)	0.0171(4)	-0.0019(3)	-0.0015(3)	-0.0011(3)
O1	0.0376(5)	0.0173(4)	0.0170(4)	-0.0045(3)	-0.0007(3)	0.0020(3)
O2	0.0135(4)	0.0262(4)	0.0318(5)	-0.0041(4)	0.0011(3)	-0.0010(3)

Table 7. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for 4y.

	x/a	y/b	z/c	U(eq)
H1	0.4185	0.4591	0.7133	0.022
H2	0.4547	0.1810	0.7103	0.024
H3	0.3945	0.0380	0.5880	0.024
H4	0.2957	0.1691	0.4665	0.021
H9	0.1894	0.3591	0.3646	0.02
H10	0.1418	0.6886	0.3117	0.022
H11A	0.2349	0.5864	0.1726	0.025
H11B	0.2610	0.4039	0.2131	0.025
H12A	-0.0446	0.4051	0.2558	0.025
H12B	-0.0706	0.5876	0.2154	0.025
H13A	0.0446	0.7909	0.4386	0.028
H13B	0.2399	0.8449	0.4182	0.028
H13C	0.1477	0.8859	0.5083	0.028
H15	-0.1971	1.0269	0.2589	0.021
H16	0.0447	1.1159	0.3320	0.022

	x/a	y/b	z/c	U(eq)
H17	0.3278	1.0695	0.2768	0.022
H18	0.3786	0.9311	0.1486	0.02
H23	0.1330	0.6393	-0.1042	0.022
H24A	0.4982	0.5579	-0.0629	0.023
H24B	0.3841	0.4655	-0.1355	0.023
H25A	0.3486	0.7128	-0.2119	0.024
H25B	0.4626	0.8052	-0.1392	0.024
H26A	-0.2829	0.7310	-0.0285	0.031
H26B	-0.1270	0.6004	-0.0335	0.031
H26C	-0.1132	0.7613	-0.0874	0.031
H00F	0.3546	0.7906	0.0163	0.02

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