Synthesis of α-Indolylacrylates as potential anti-cancer agents by Brønsted acid ionic liquid catalyst and butyl acetate solvent†

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1. Synthesis of ionic liquid 1a[1]

Brønsted acid IL **1a** was synthesized through the following three steps: (**i**) synthesis of **2a**: in 100 ml of around bottomed flask equipped with mechanical stirring, equal amount of divinyl sulfone **2a** (5.0 g, 42.3 mmol) was mixed with 1-(3-aminopropanyl)imidazole (5.2 g, 42.3 mmol) in 75 ml of methanol; the mixture was stirred at 60 °C for 24 h; then, volatile methanol was removed by a rotary evaporator; a yellow-pale oil was obtained, which is **2a**, in nearly quantitative yield. (**ii**) synthesis of **3a** via quaternization: **2a** (10.0 g, 41.1 mmol), 1,3-propanesulfonate (10.0 g, 82.3 mmol), and acetonitrile (75 ml) were mixed in a 250 ml of around bottomed flask equipped with mechanical stirring; the mixture was stirred at 80 °C for 24 h; a yellow solid was generated; then the solvent was decanted out; the yellow solid was filtrated, and washed with acetone (5.0 ml × 3); **3a** can be obtained as white solid; then, the white solid was dried at 60 °C under vacuum (20 mmHg) for 4 h; and (**iii**) acidification: **3a** (10.0 g, 20.5 mmol) was mixed with triflic acid (3.1 g, 20.5 mmol) in 25 mL of around bottomed flask; to facilitate the reaction, a small amount of water (0.25 ml) was also added in this step. Then, the mixture was stirred at 100 °C for 24 h. The generated ionic liquid was washed with ethyl acetate (5.0 ml × 3) and diethyl ether (5.0 ml × 3); then, water and volatile solvents were removed under reduced pressure; finally, **1a** was obtained as a yellow-pale viscous liquid. Through all these three-step reactions, the ionic liquid **1a** can be synthesized in 88% yield.

2. Synthesis of Forbes's ionic liquid 1b[2]

1-Methylimidazole (8.2 g, 0.1 mol) is stirred under solvent-free conditions with 1,3-propanesultone (12.2 g, 0.1 mol) at 50 oC for 6 hours. After reaction, the obtained viscous liquid was washed with ethyl acetate (10 ml × 3) and then dried under vacuum (10 mmHg) for 2 hours. With this procedure, 3-(1-methyl-3-imidazolio)propanesulfonate was obtained in 97 % of yield (mp. = 215-216 oC). In a 100 ml of round bottomed flask equipped with mechanical stirring, 3-(1-methyl-3-imidazolio)propanesulfonate (10.2 g, 50 mmol) was mixed with trifloromethanesulfonic acid (7.5 g, 50 mmol). The mixture was then heated at 80 ˚C for 6 hours under stirring. Then, the generated viscous liquid washed with ethyl acetate (10 ml × 3). After 6 hours of drying at 60 °C under vacuum (20 mmHg), Forbes's IL **1b** was obtained as a brown viscous liquid (16.8 g, 95 %).

3. Synthesis of ionic liquid 1c[3]

In 250 ml of round bottomed flask equipped with mechanical stirring, divinyl sulfone **2c** (11.8 g, 0.10 mol) was mixed with n-butylamine (7.3 g, 0.10 mol) in methanol (150 ml). The mixture was then stirred at 60 °C for 5 hours. Methanol was removed by a rotary evaporator under vacuum, and **3c** was obtained as a color-less oil quantitatively. The obtained **3c** was then mixed with 1,3-propane sultone (13.4 g, 0.11 mol) in acetonitrile (150 ml). The solution was refluxed for 5 hours. Then, the generated white solid was filtrated, and washed with acetone (10 ml × 3). After 6 hours of drying at 60 °C under vacuum (20 mmHg), **4c** was obtained as a white powder (23.1 g, 74 %). In a 100 ml of round bottomed flask, **4c** (15.6 g, 50 mmol) was mixed with trifluoromethanesulfonic acid (7.5 g, 50 mmol). Then, the mixture was stirred at 80 °C for 8 hours. In order to facilitate the stirring, water (1.0 ml) was added into the system. The formed liquid was washed with ethyl acetate (15 ml × 3) and diethyl ether (15 ml × 3). After that, it was dried at 80 °C under vacuum (10 mmHg) for 4 hours. Finally, IL **1c** was obtained as a viscous brown oil (22.2g, 96 %).

4. Figure S1: X-ray Single Crystal Data for 5a

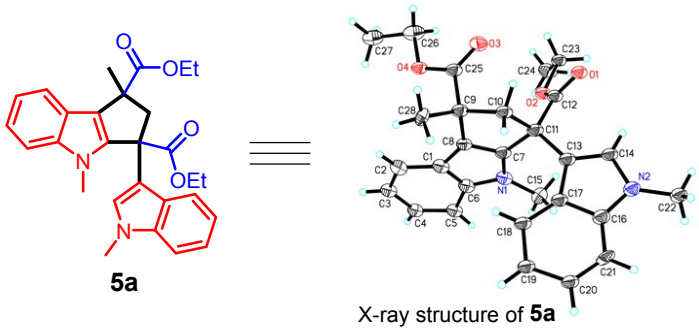


Table 1. Crystal data and structure refinement for 5a.

Identification code	exp_1776
Empirical formula	$C_{28}H_{30}N_2O_4$
Formula weight	458.54
Temperature/K	100.01(10)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	16.179(2)
b/Å	8.8178(15)
c/Å	17.233(2)
α/°	90
β/°	103.819(13)
γ/°	90
Volume/Å^3	2387.3(6)
Z	2
ρ_{calc}g/cm^3	0.638
μ/mm^{-1}	0.043
F(000)	488.0
Crystal size/mm^3	0.13 × 0.12 × 0.11
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.868 to 49.98

Index ranges	$-15 \le h \le 19, -8 \le k \le 10, -20 \le l \le 20$
Reflections collected	11382
Independent reflections	7256 [R_{int} = 0.0473, R_{sigma} = 0.0860]
Data/restraints/parameters	7256/109/174
Goodness-of-fit on F^2	1.070
Final R indexes [I>=2σ (I)]	R_1 = 0.0732, wR_2 = 0.1990
Final R indexes [all data]	R_1 = 0.0787, wR_2 = 0.2059
Largest diff. peak/hole / e $Å^{-3}$	0.51/-0.47
Flack parameter	-0.2(9)

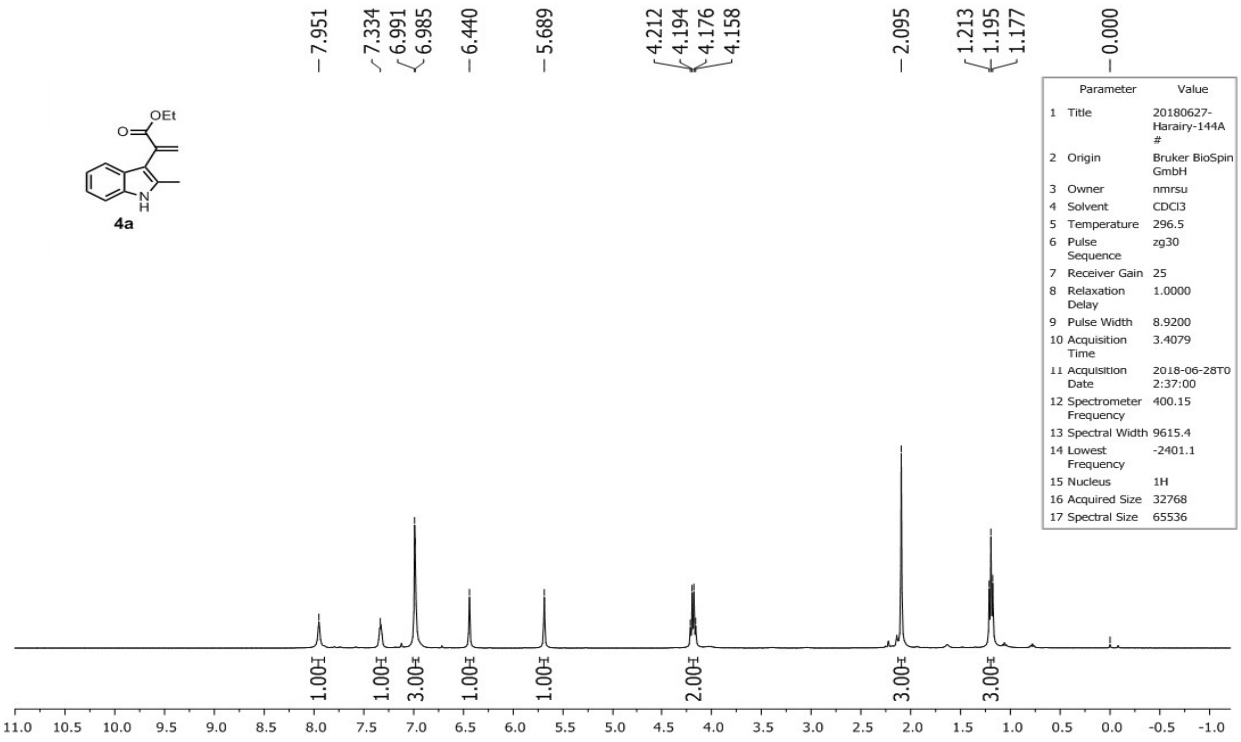
Crystal structure determination of 5a

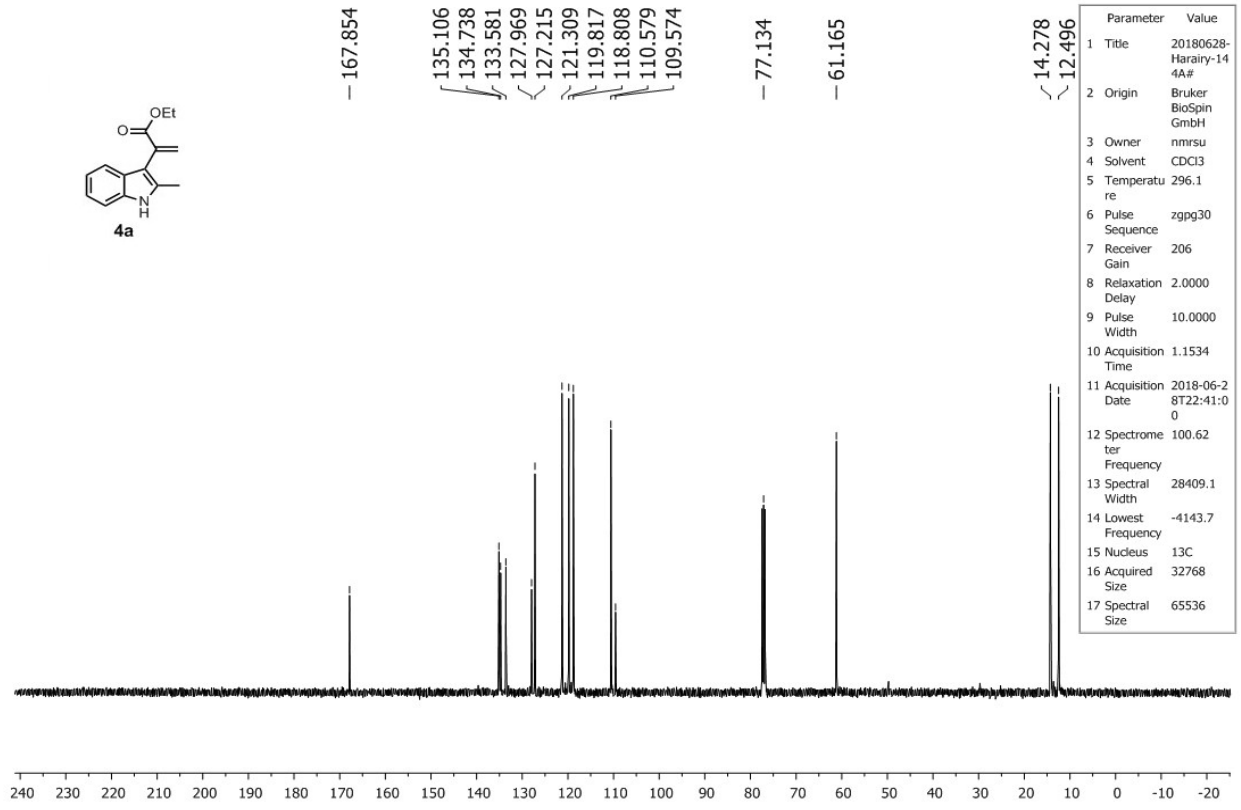
Crystal Data for $C_{28}H_{30}N_2O_4$ (*M* =458.54 g/mol): monoclinic, space group $P2_1$ (no. 4), *a* = 16.179(2) Å, *b* = 8.8178(15) Å, *c* = 17.233(2) Å, *β* = 103.819(13)°, *V* = 2387.3(6) $Å^3$, *Z* = 2, *T* = 100.01(10) K, μ(MoKα) = 0.043 mm^{-1}, *Dcalc* = 0.638 g/cm^3, 11382 reflections measured (4.868° ≤ 2Θ ≤ 49.98°), 7256 unique (R_{int} = 0.0473, R_{sigma} = 0.0860) which were used in all calculations. The final R_1 was 0.0732 (I > 2σ(I)) and wR_2 was 0.2059 (all data).

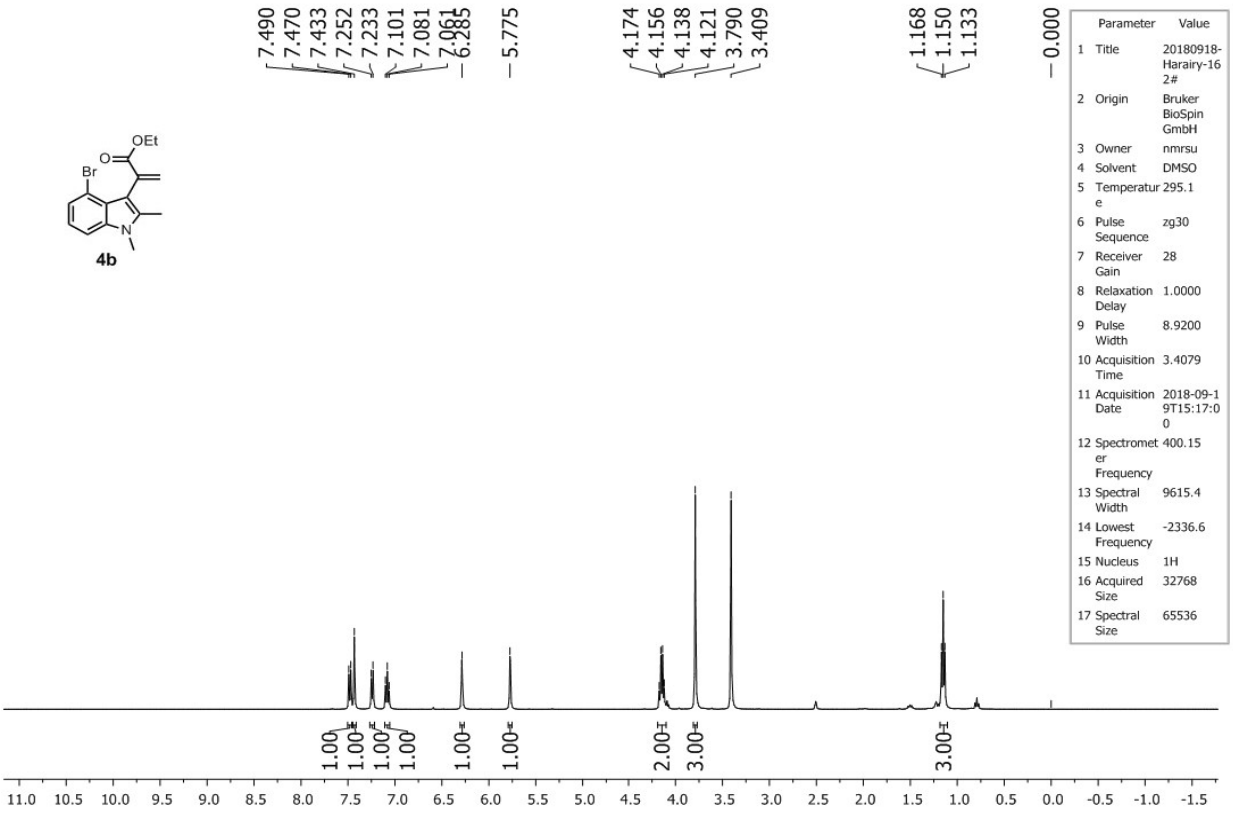
5. References

1. (a) A. El-Harairy, B. Lai, L. Vaccaro, M. Li, Y. Gu, *Advanced Synthesis & Catalysis*. 2019, **361**, 3342-3350; b) A. El-Harairy, M. Yue, W. Fan, F. Popowycz, Y. Queneau, M. Li, Y. Gu, *ChemCatChem*, 2019, **11**, 4403-4410.
2. Y. Gu, F. Shi, Y. Deng, *J. Mol. Catal. A: Chem*. 2004, **212**, 71–75.
3. A. Taheri, X. Pan, C. Liu, Y. Gu, *ChemSusChem* 2014, **7**, 2094–2100.

6. NMR Spectra



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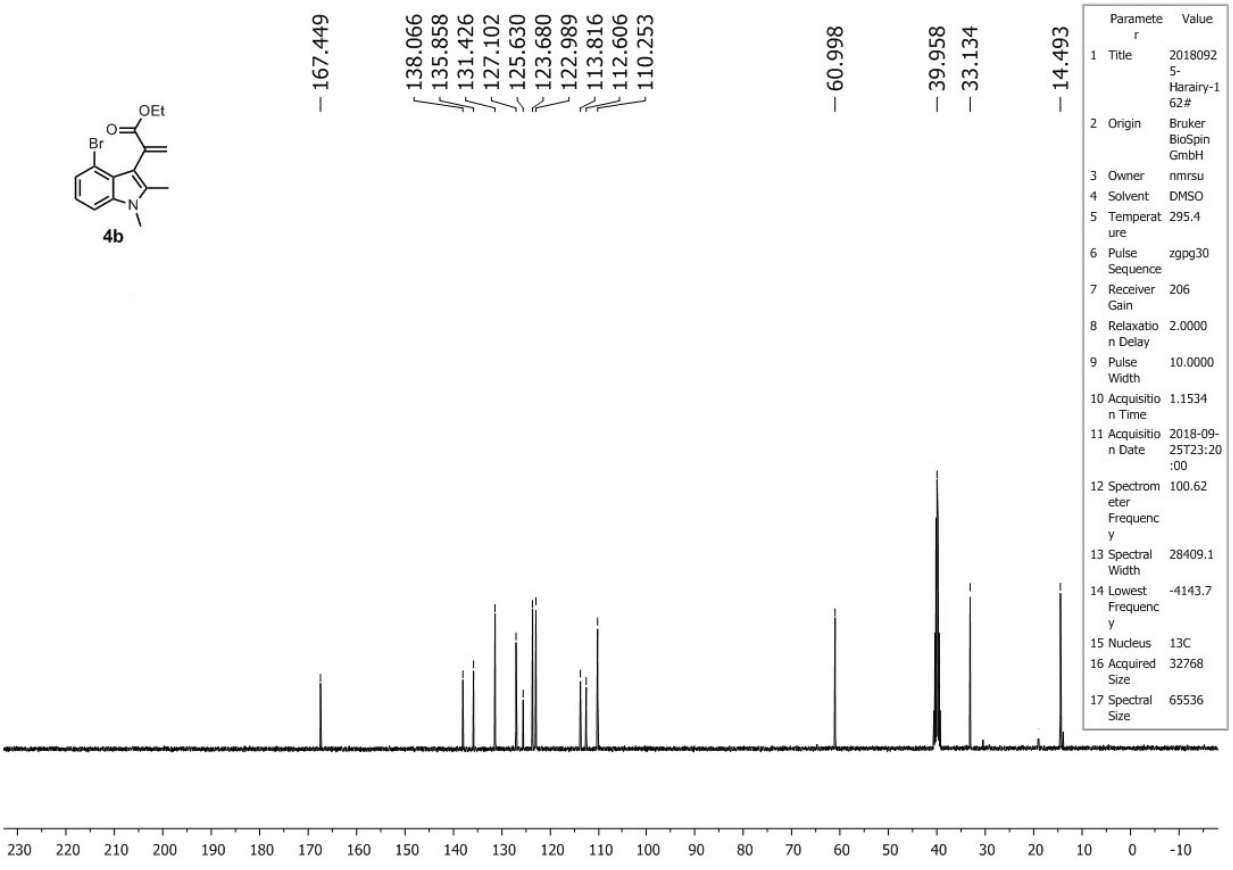
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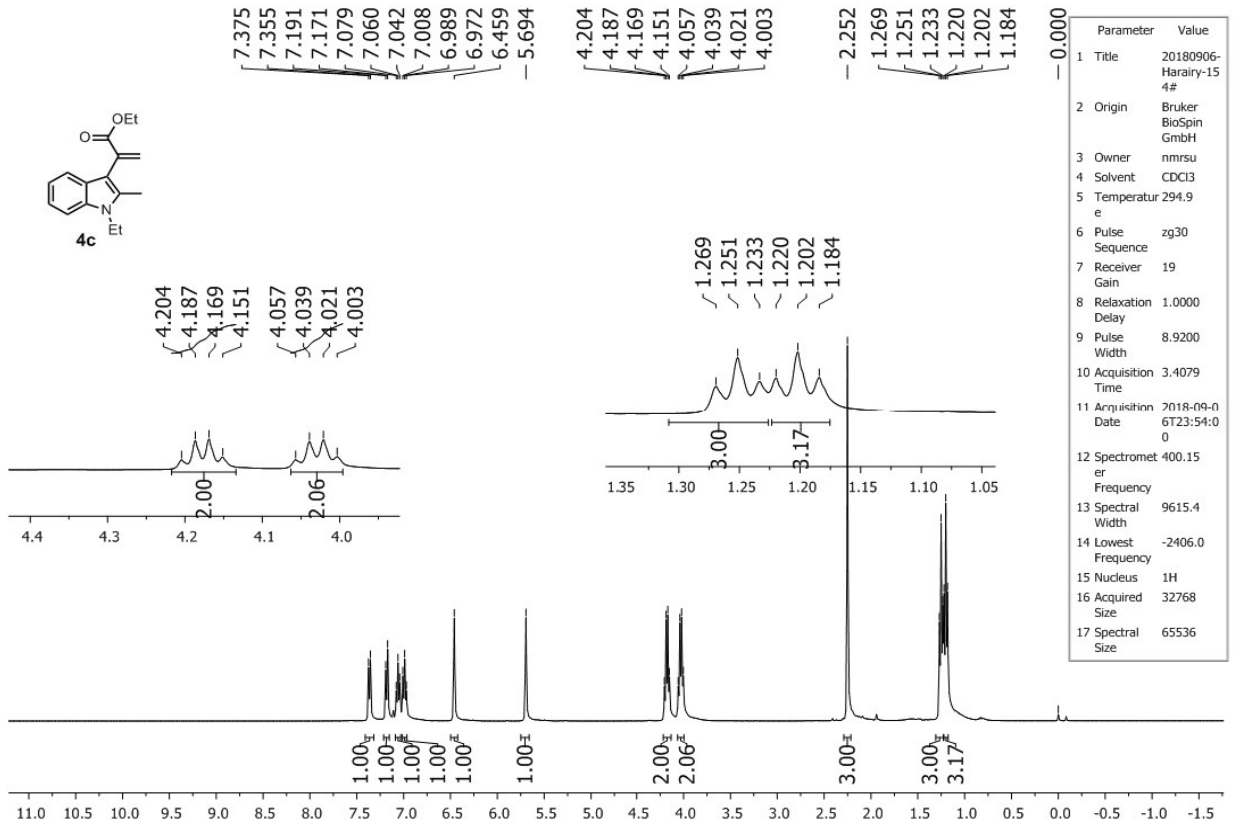
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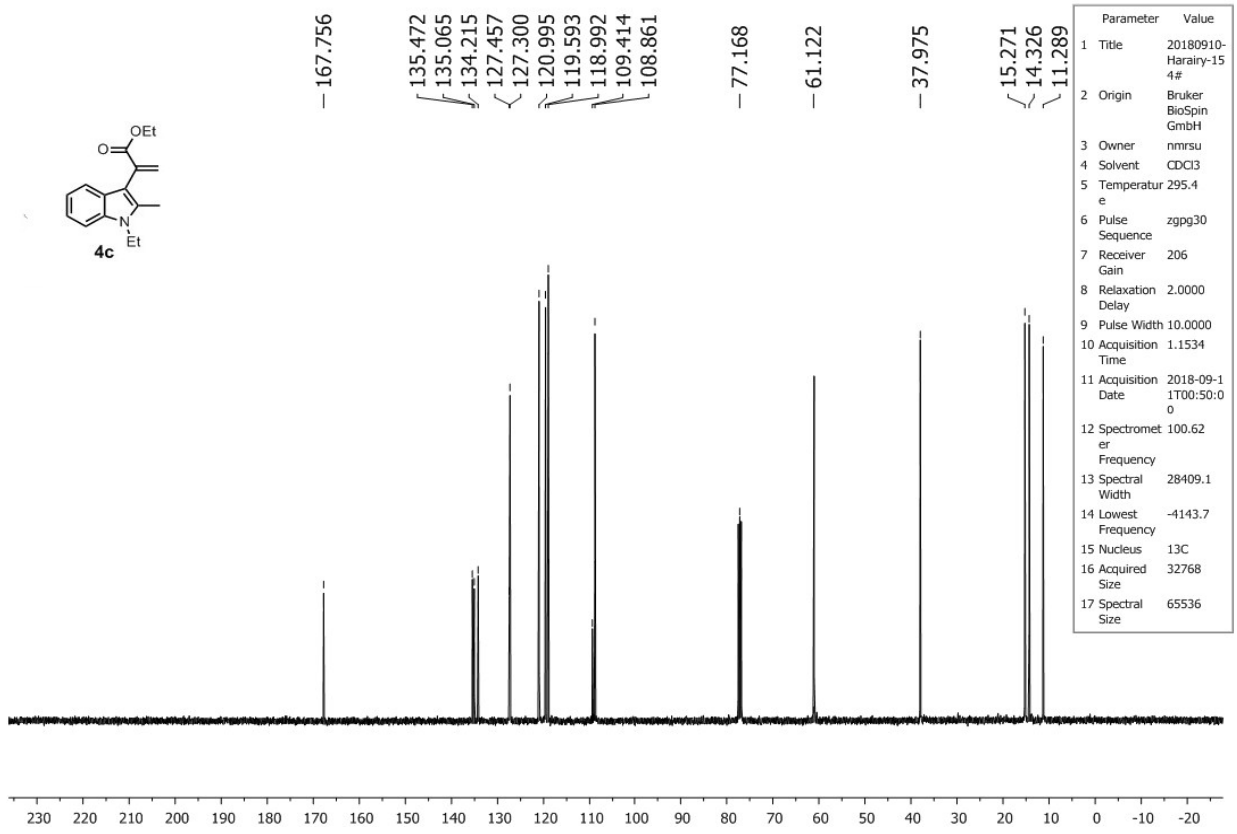
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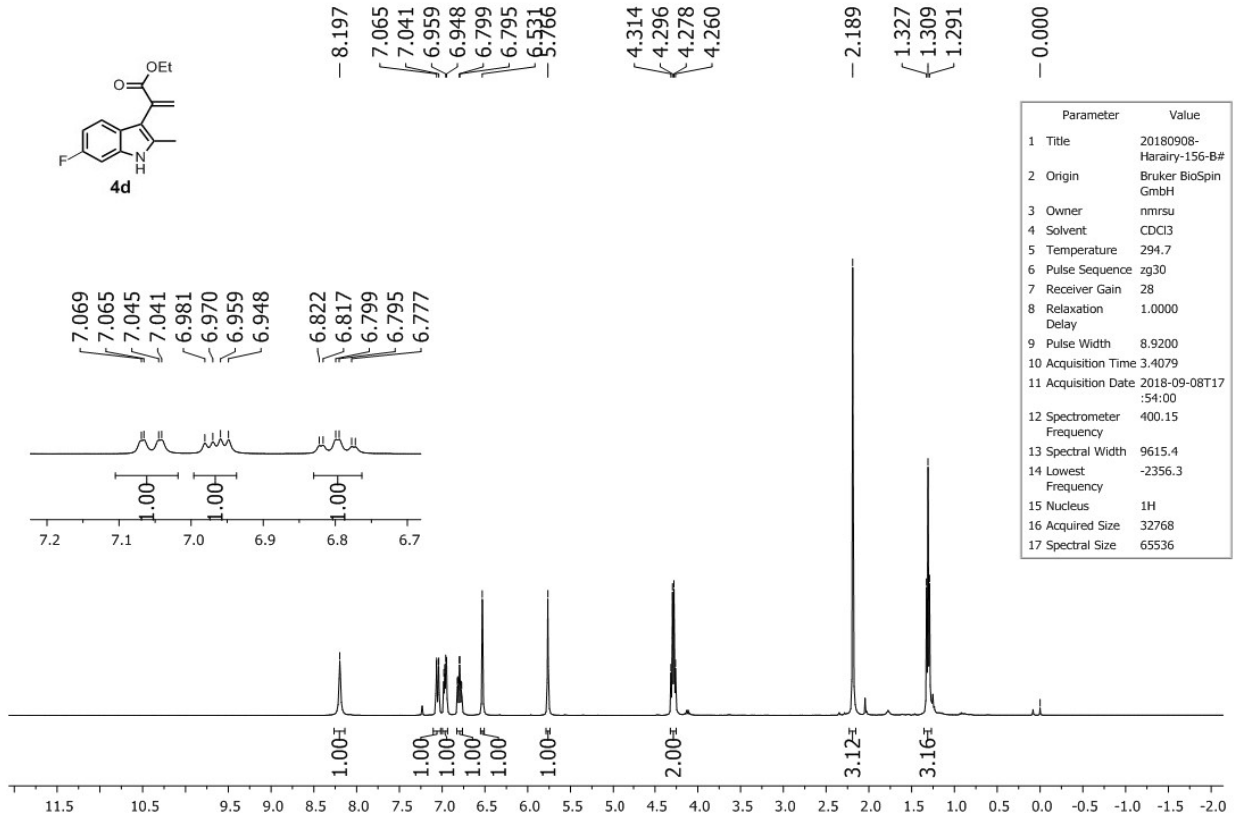
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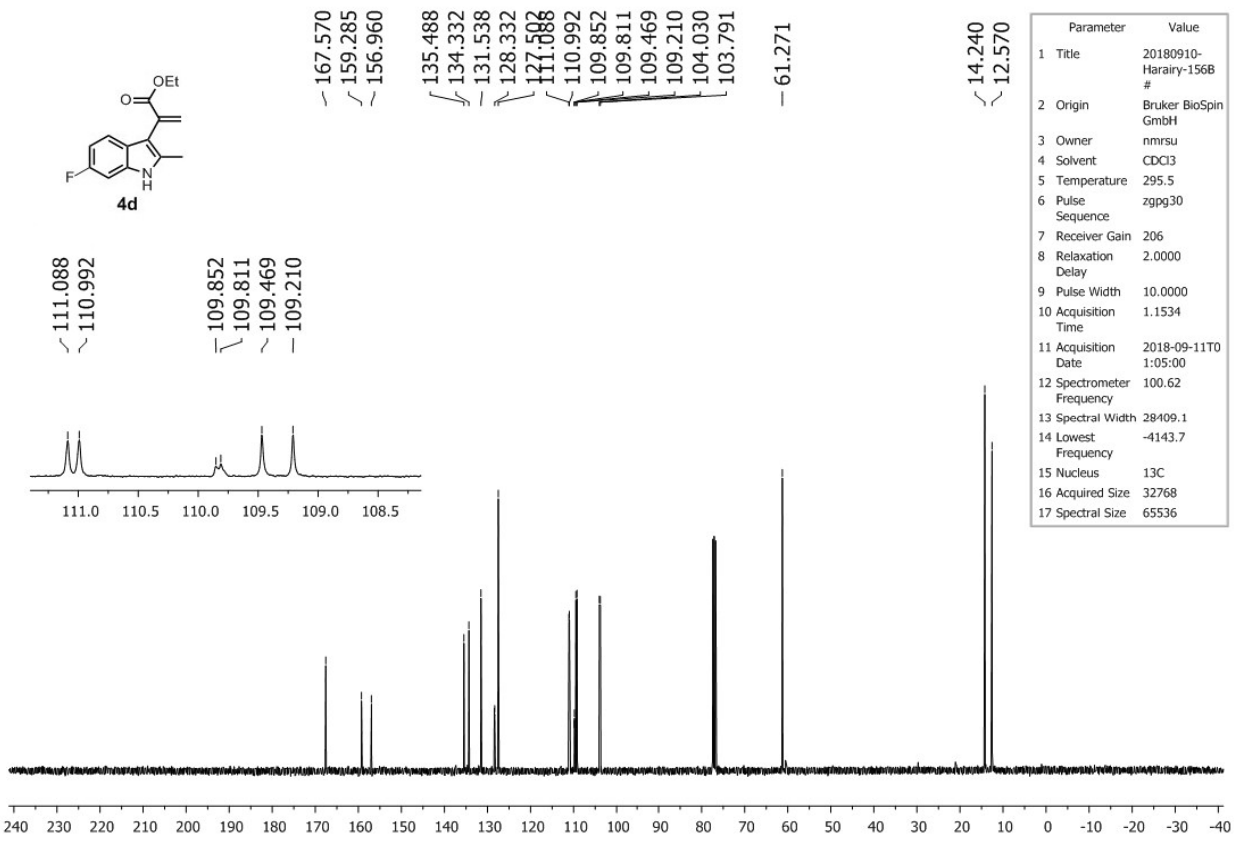
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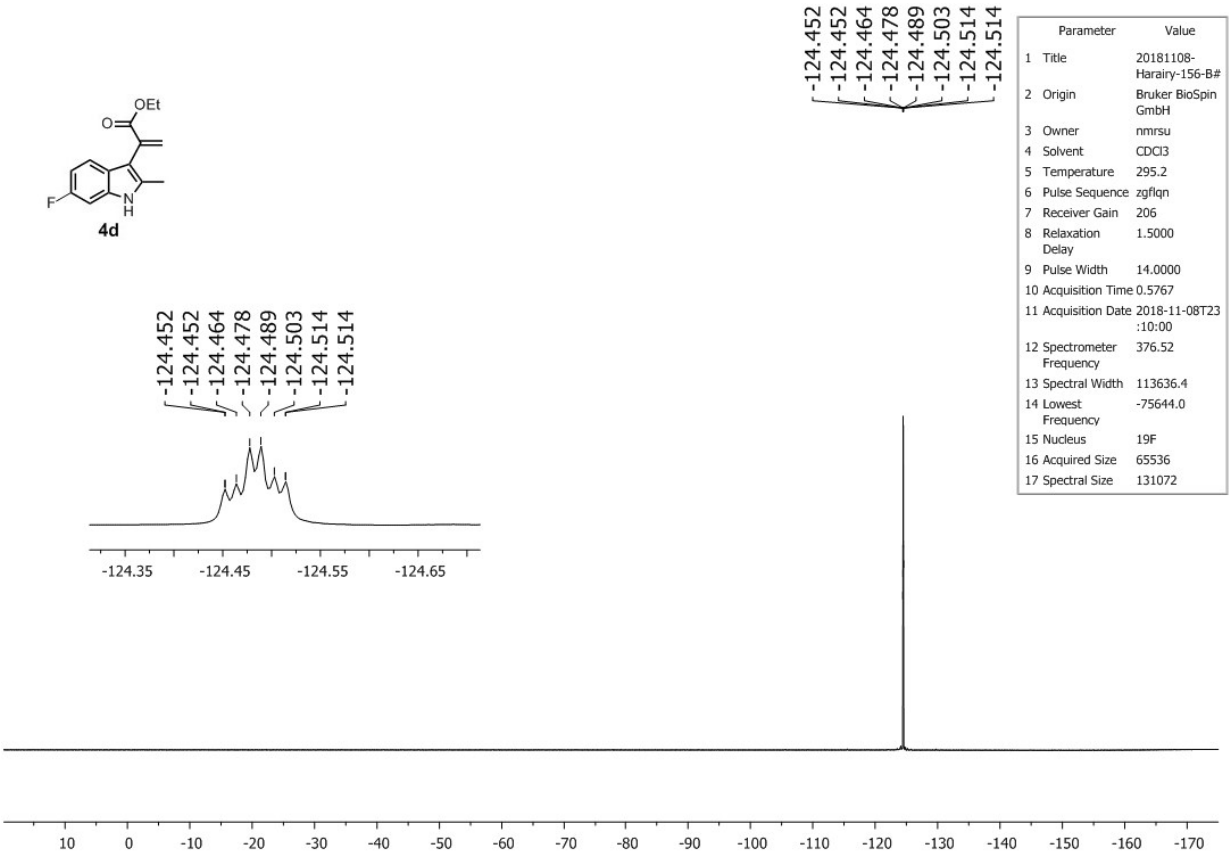

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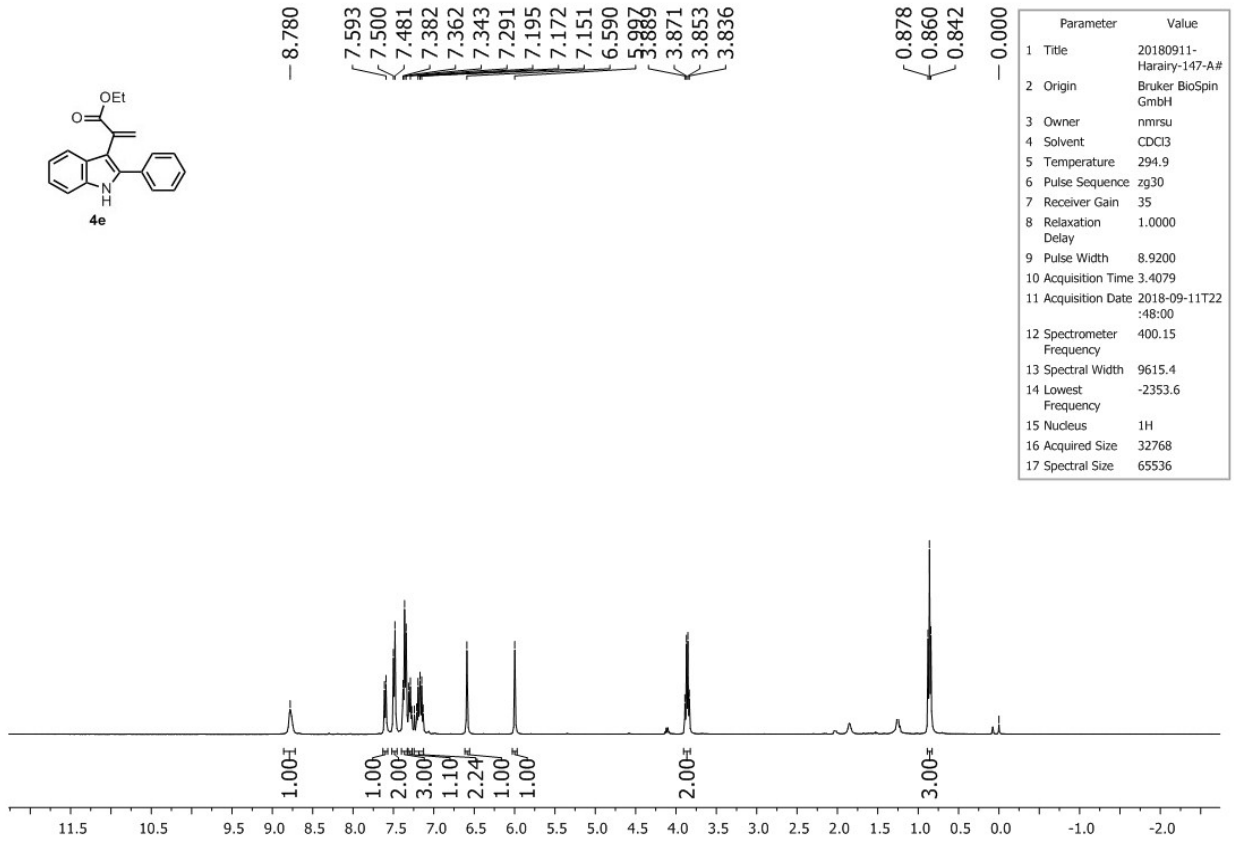

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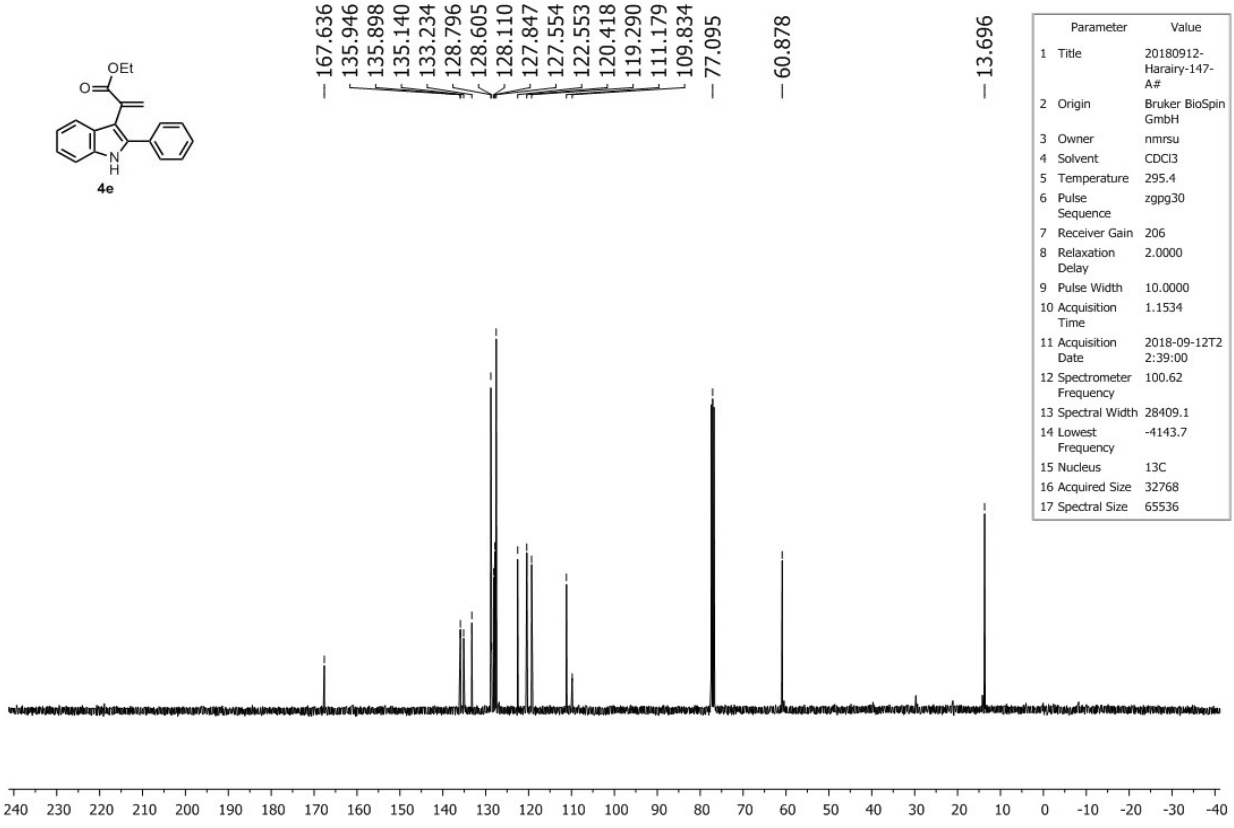

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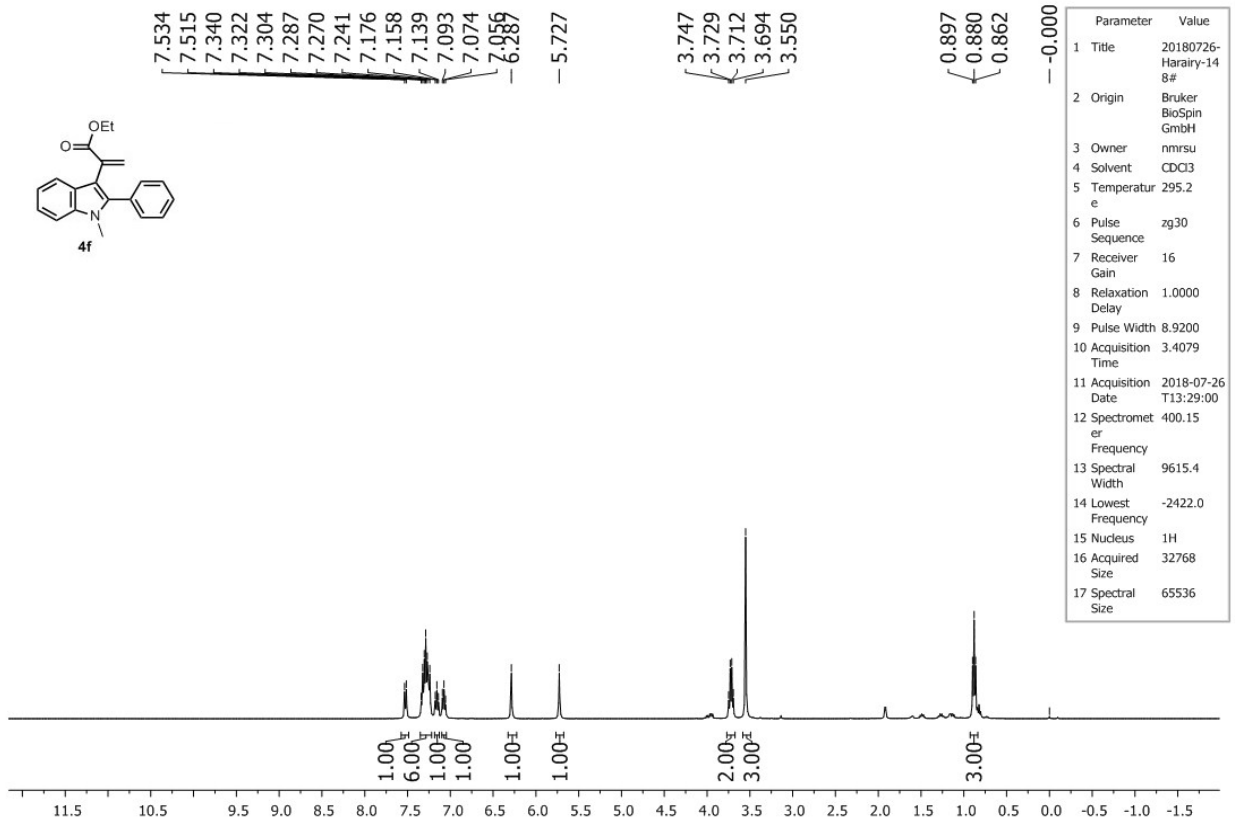
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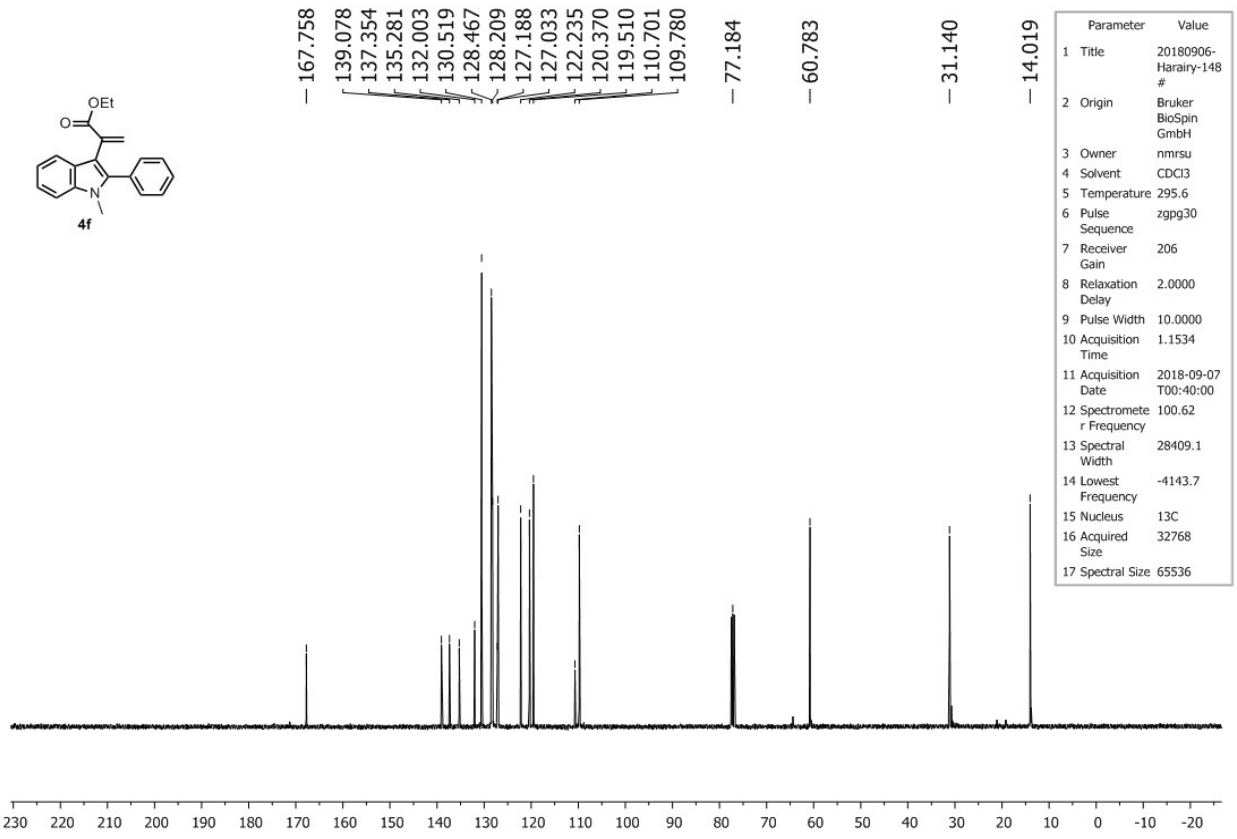
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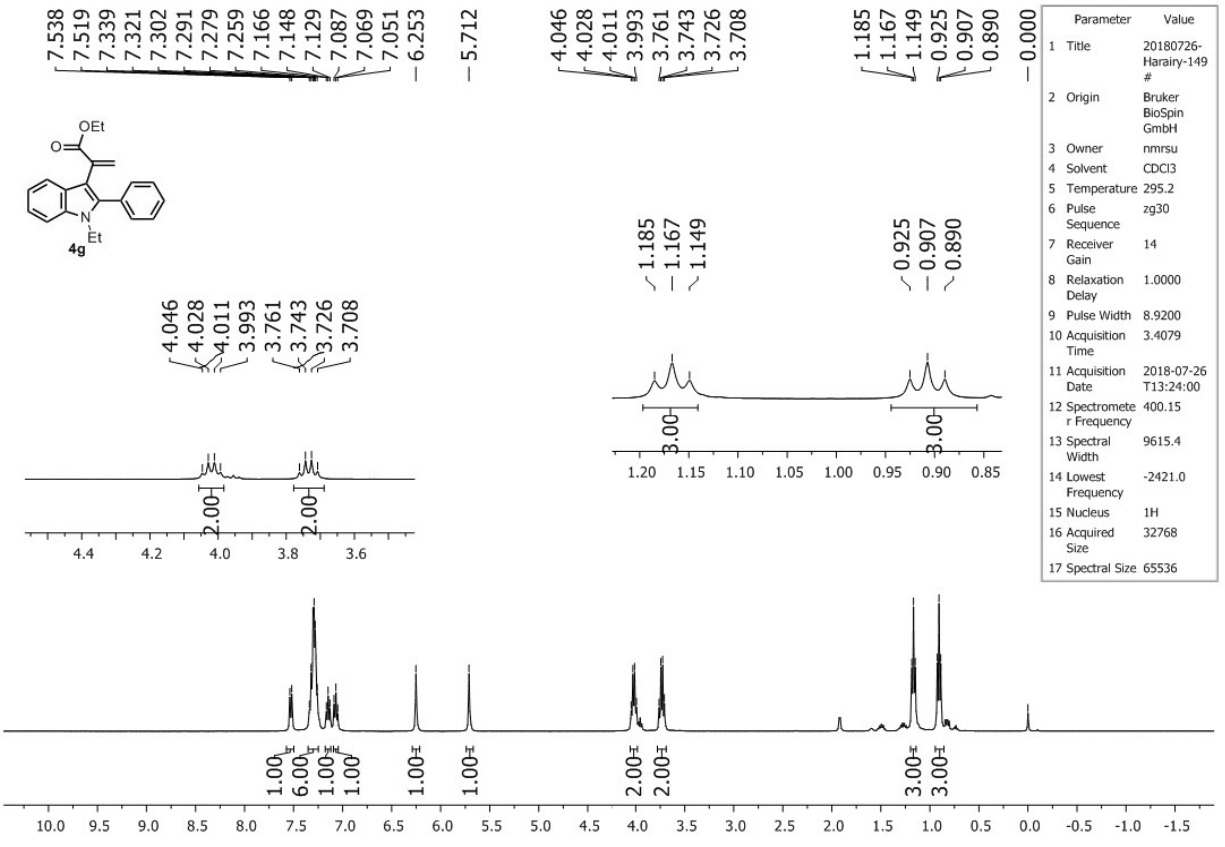
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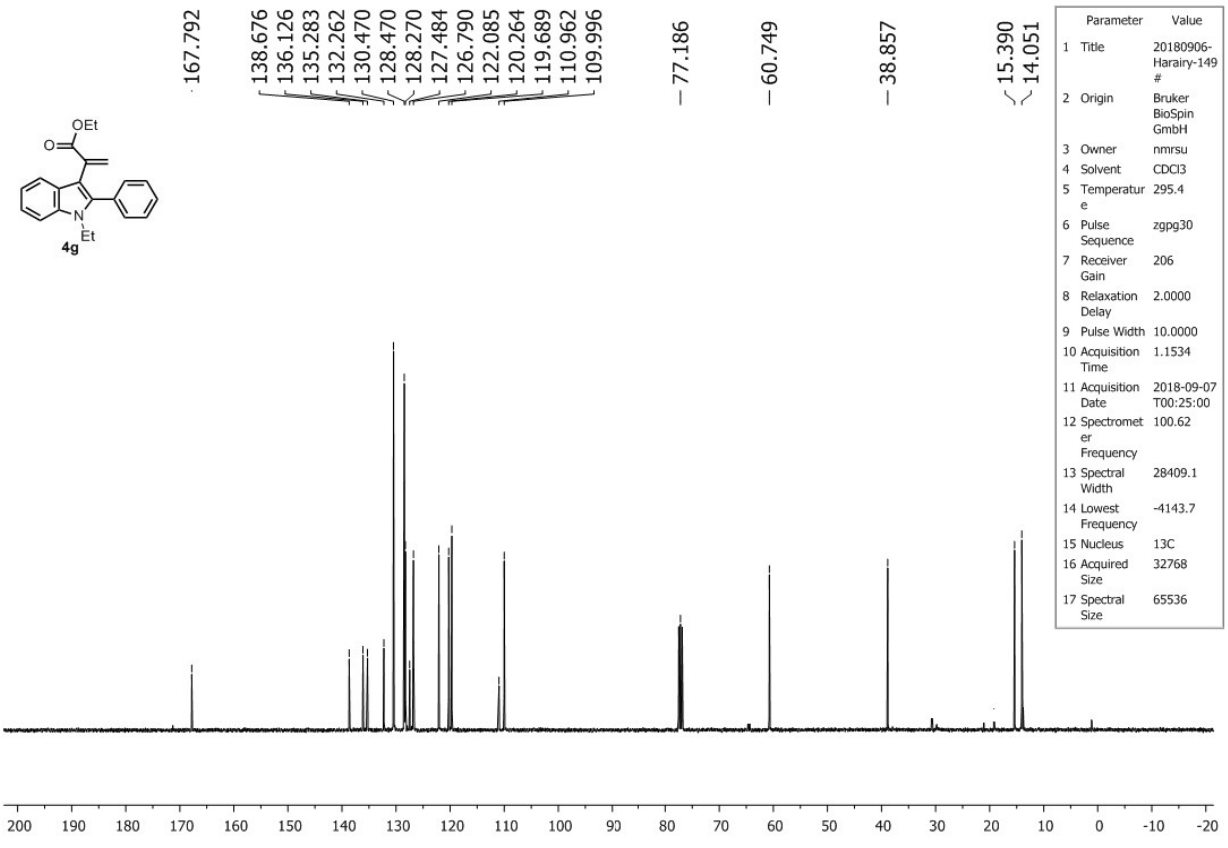
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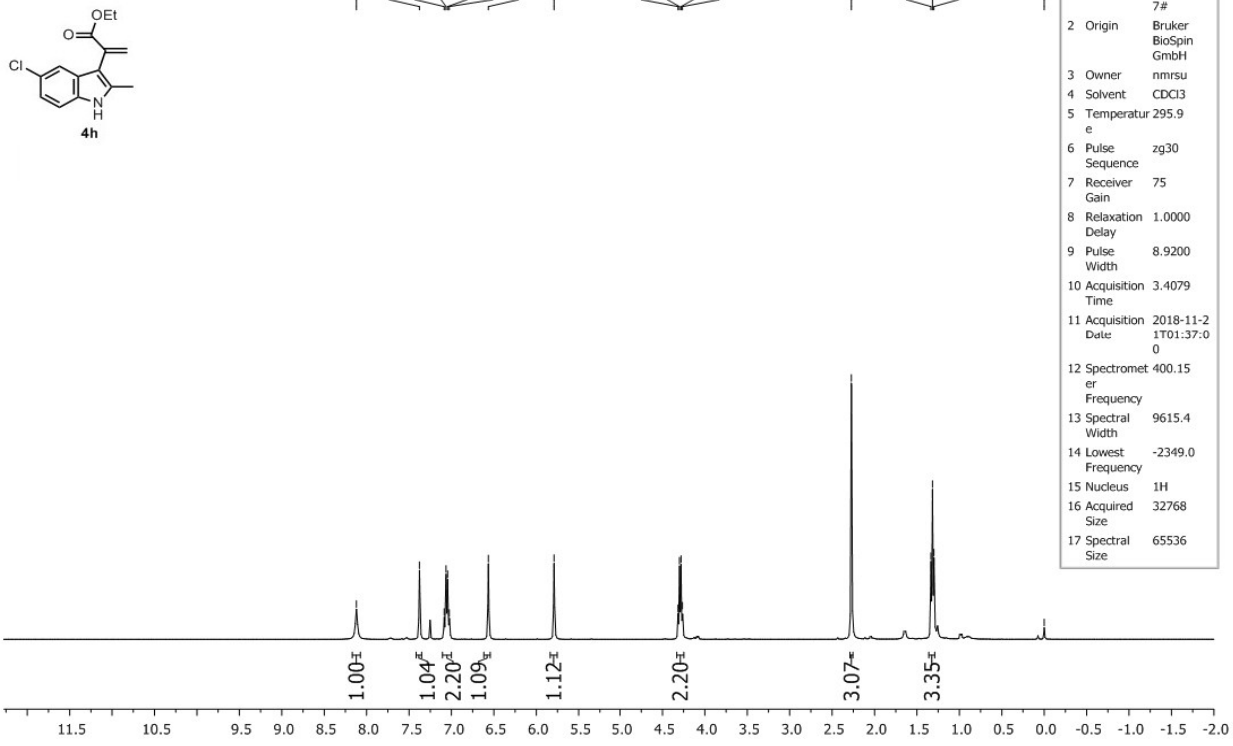
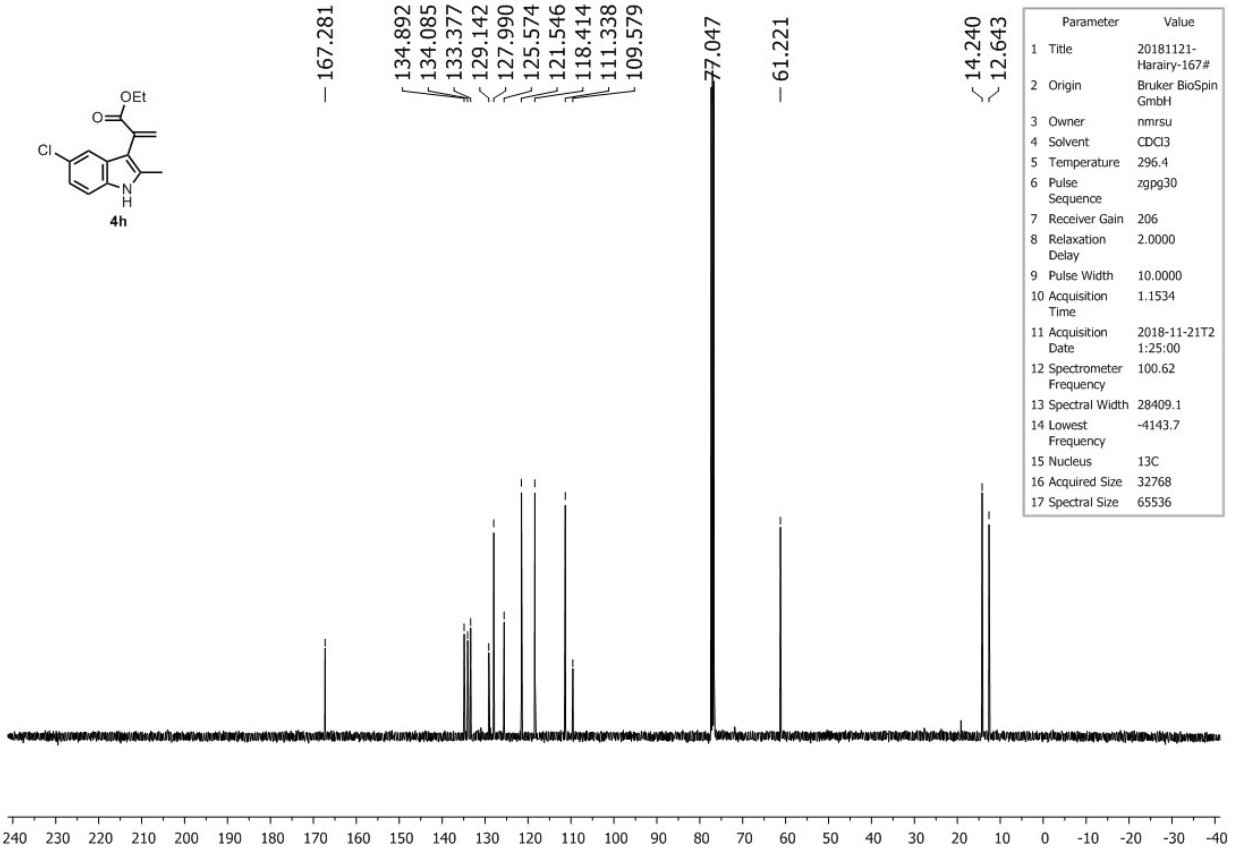
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-09-07 T00:25:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536


8.121
7.377
7.085
7.064
7.046
7.025
6.566
5.791
4.316
4.299
4.281
4.264
2.273
1.335
1.317
1.300
0.000
1.00
1.04
2.20
1.09
1.12
2.20
3.07
3.35
4h
Parameter Value
1 Title 20181120-Harairy-167#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 295.9
6 Pulse Sequence zg30
7 Receiver Gain 75
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-11-21T01:37:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2349.0
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536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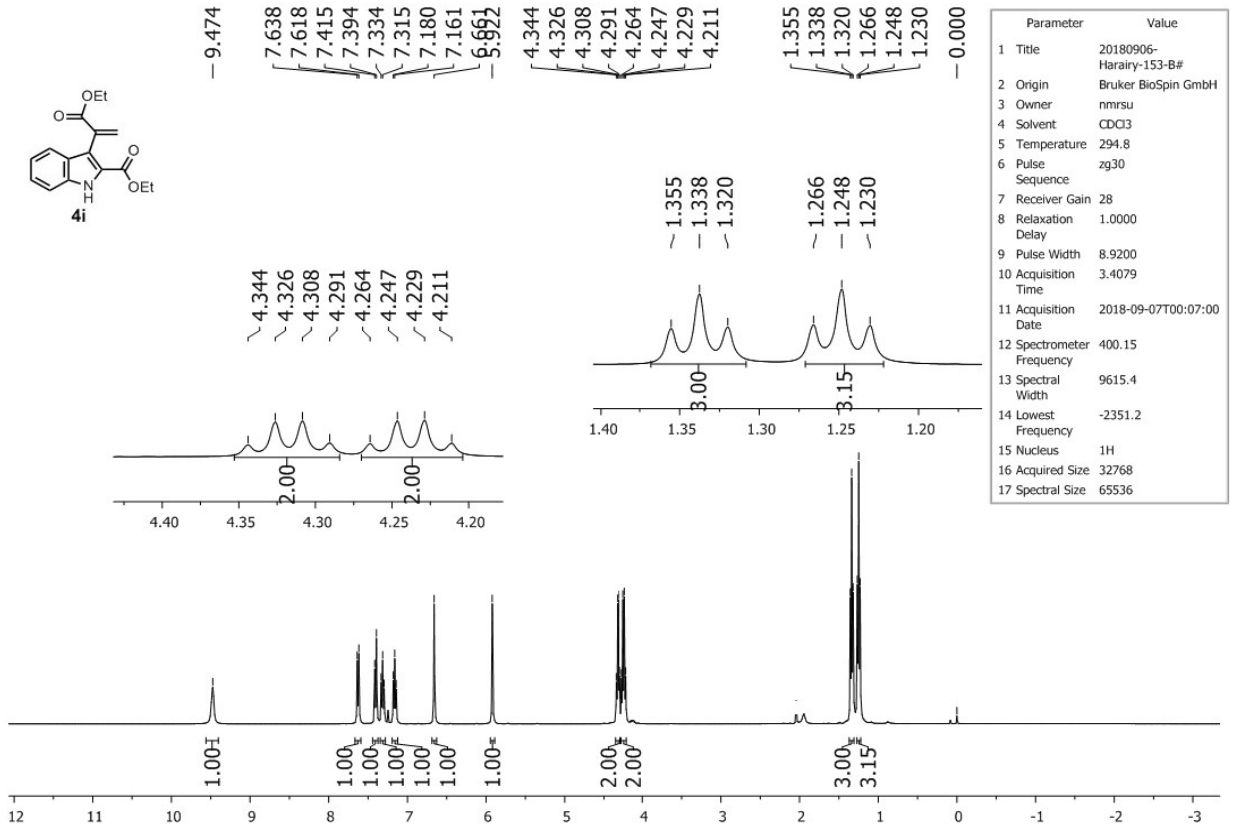
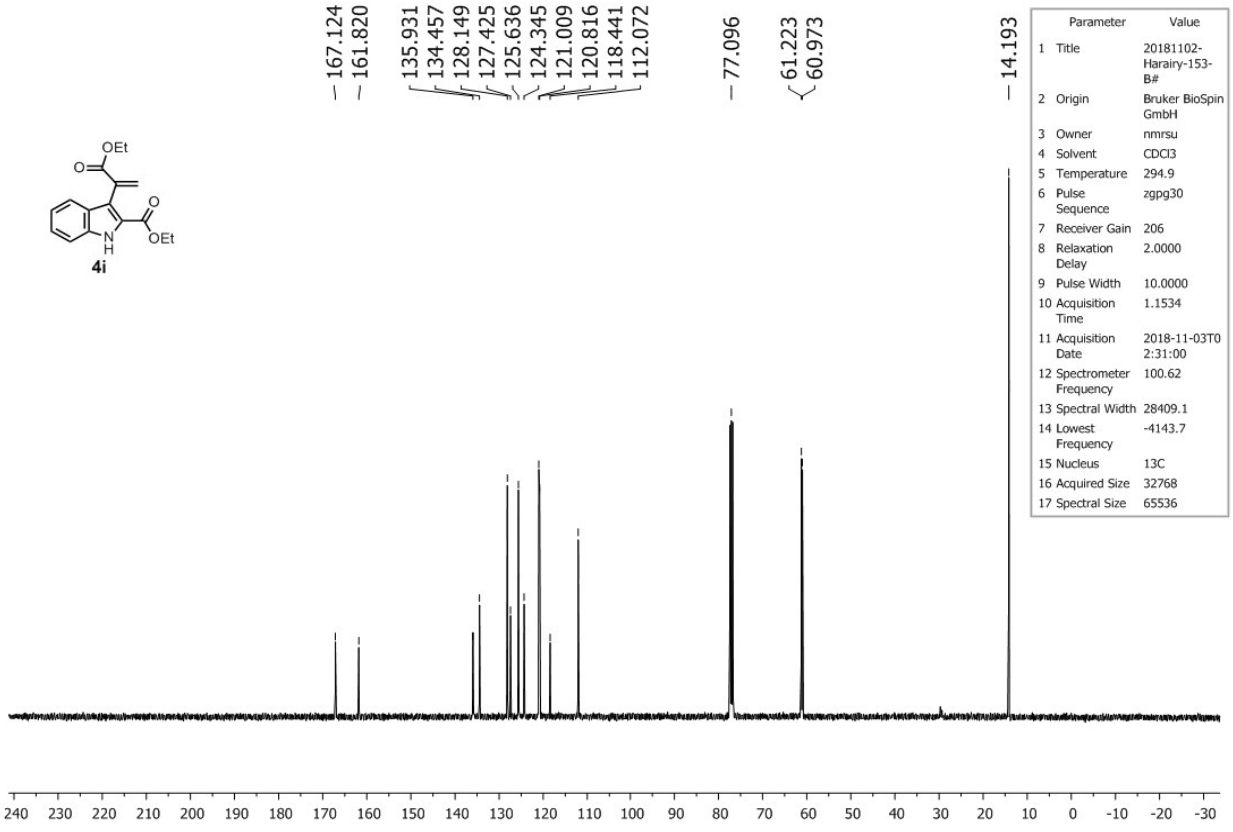
134.892
134.085
133.377
129.142
127.990
125.574
121.546
118.414
111.338
109.579
77.047
61.221
14.240
12.643
4h
Parameter Value
1 Title 20181121-Harairy-167#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 296.4
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-11-21T21:25:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536


9.474
7.638
7.618
7.415
7.394
7.334
7.315
7.180
7.161
6.661
5.922
4.344
4.326
4.308
4.291
4.264
4.247
4.229
4.211
1.355
1.338
1.320
1.266
1.248
1.230
0.000
4i
Parameter Value
1 Title 20180906-Harairy-153-B#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.8
6 Pulse Sequence zg30
7 Receiver Gain 28
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-09-07T00:07:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2351.2
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536
2.00
2.00
3.00
3.15
1.00
1.00
1.00
1.00
1.00
1.00
2.00
2.00
3.00
3.15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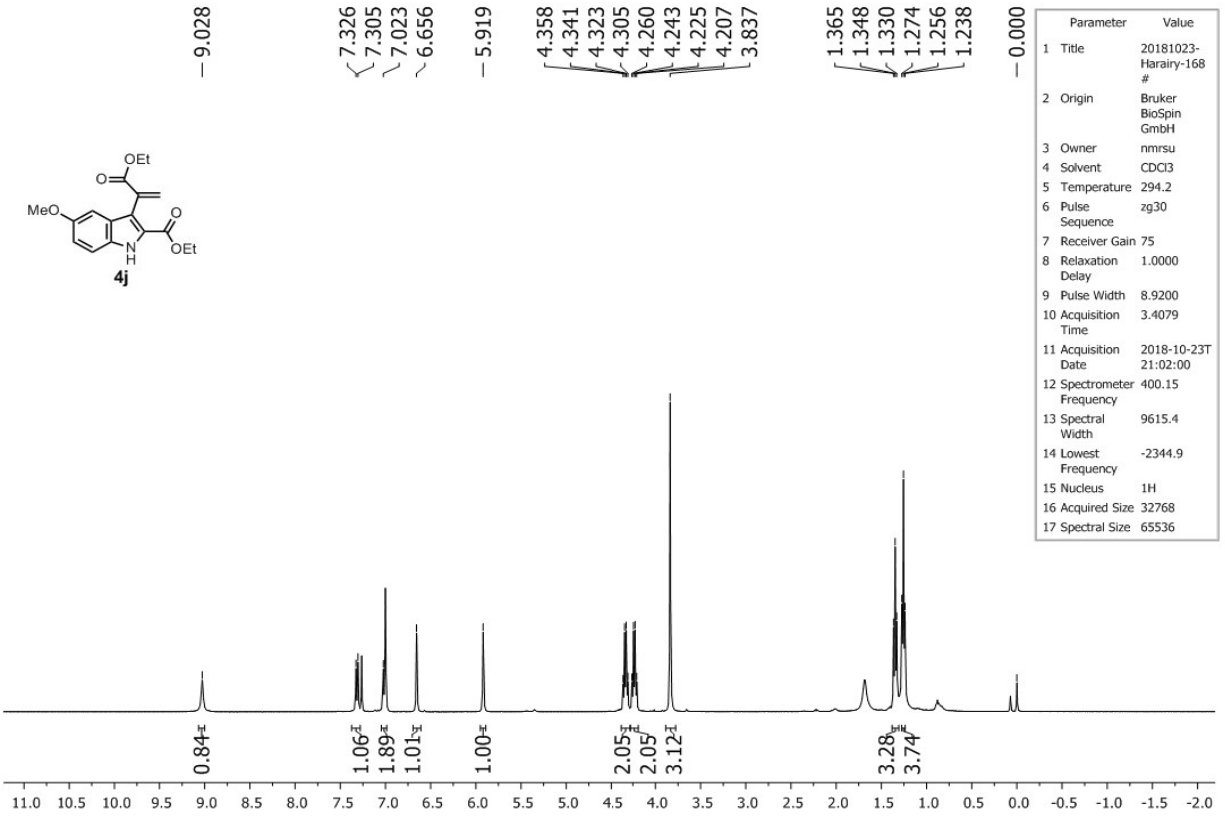
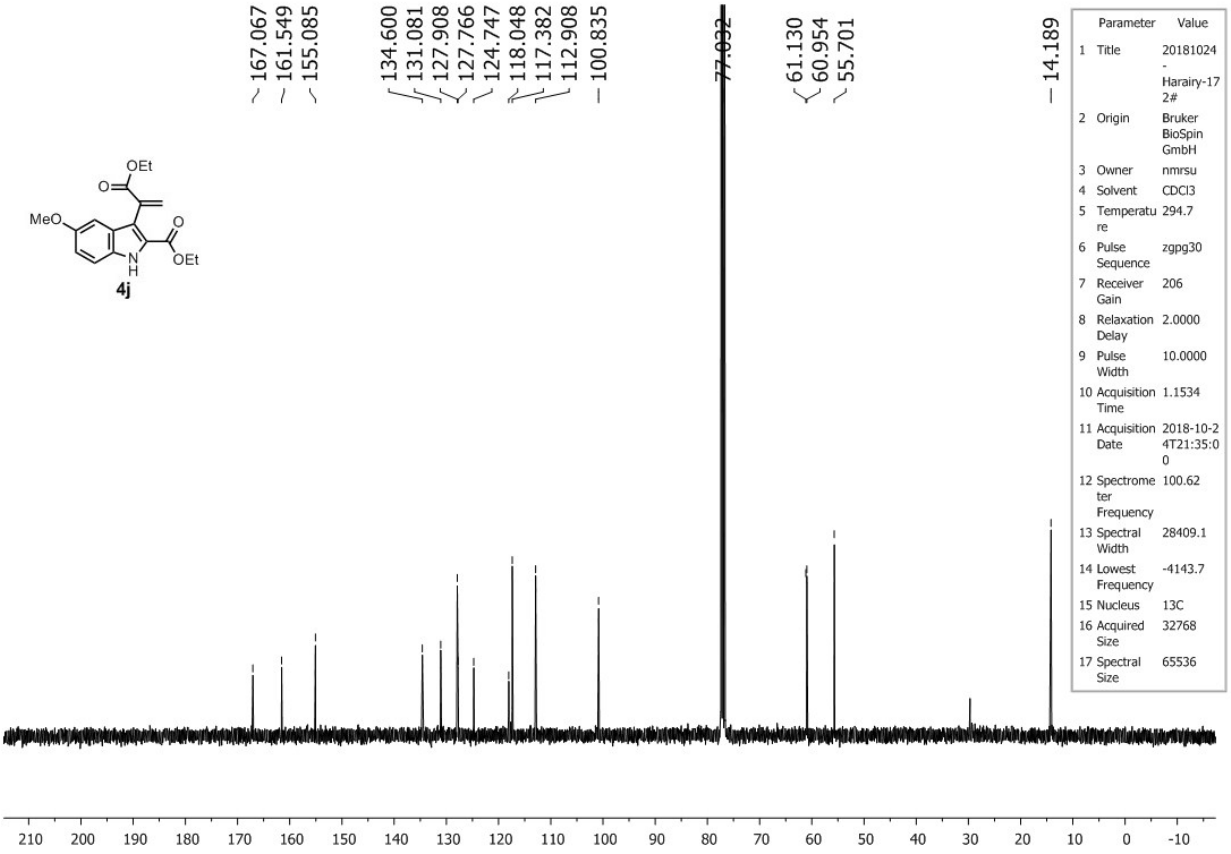
135.931
134.457
128.149
127.425
125.636
124.345
121.009
120.816
118.441
112.072
77.096
61.223
60.973
14.193
4i
Parameter Value
1 Title 20181102-Harairy-153-B#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.9
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-11-03T02:31:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536


9.028
7.326
7.305
7.023
6.656
5.919
4.358
4.341
4.323
4.305
4.260
4.243
4.225
4.207
3.837
1.365
1.348
1.330
1.274
1.256
1.238
0.000
0.84
1.06
1.89
1.01
1.00
2.05
2.05
3.12
3.28
3.74
4j
Parameter Value
1 Title 20181023-Harairy-168#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.2
6 Pulse Sequence zg30
7 Receiver Gain 75
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-10-23T21:02:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2344.9
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536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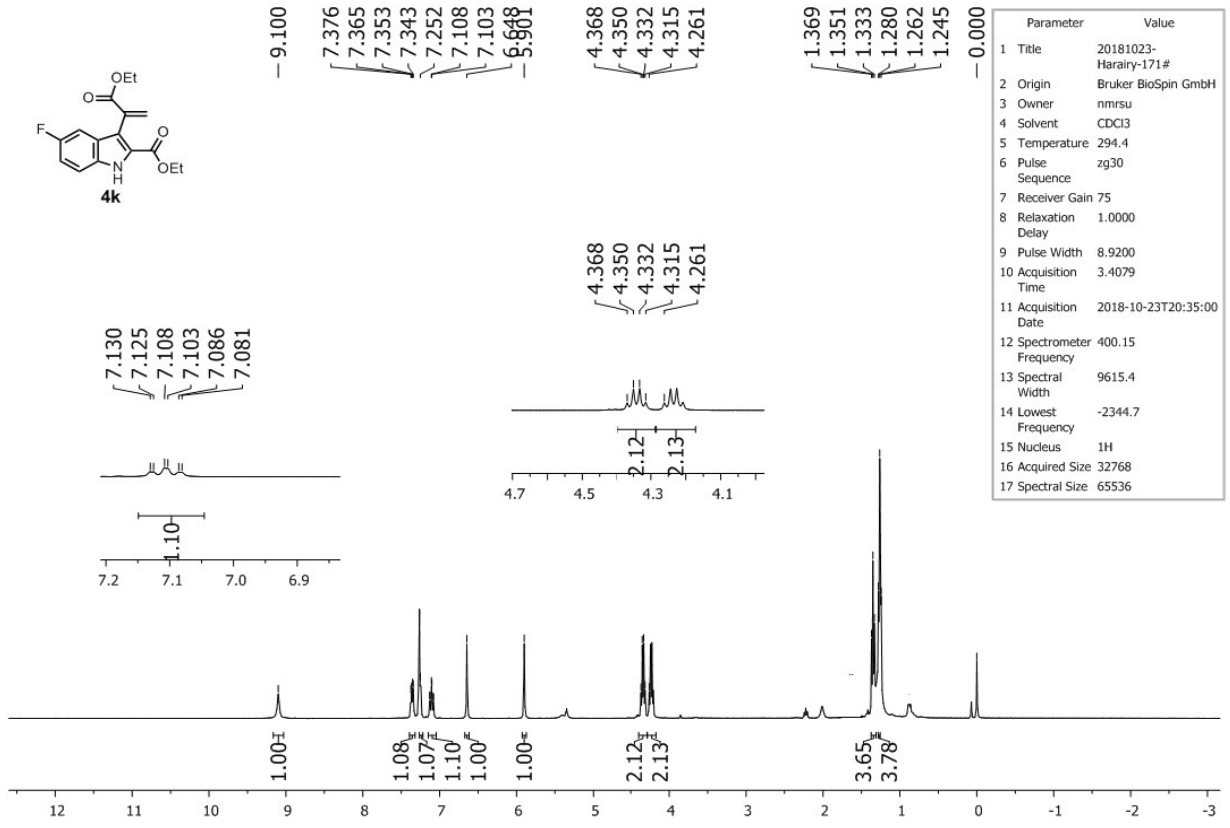
161.549
155.085
134.600
131.081
127.908
127.766
124.747
118.048
117.382
112.908
100.835
77.032
61.130
60.954
55.701
14.189
4j
Parameter Value
1 Title 20181024-Harairy-172#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.7
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-10-24T21:35:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536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7.376
7.365
7.353
7.343
7.252
7.108
7.103
6.648
5.901
4.368
4.350
4.332
4.315
4.261
1.369
1.351
1.333
1.280
1.262
1.245
0.000
4k
Parameter Value
1 Title 20181023-Harairy-171#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.4
6 Pulse Sequence zg30
7 Receiver Gain 75
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-10-23T20:35:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2344.7
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536
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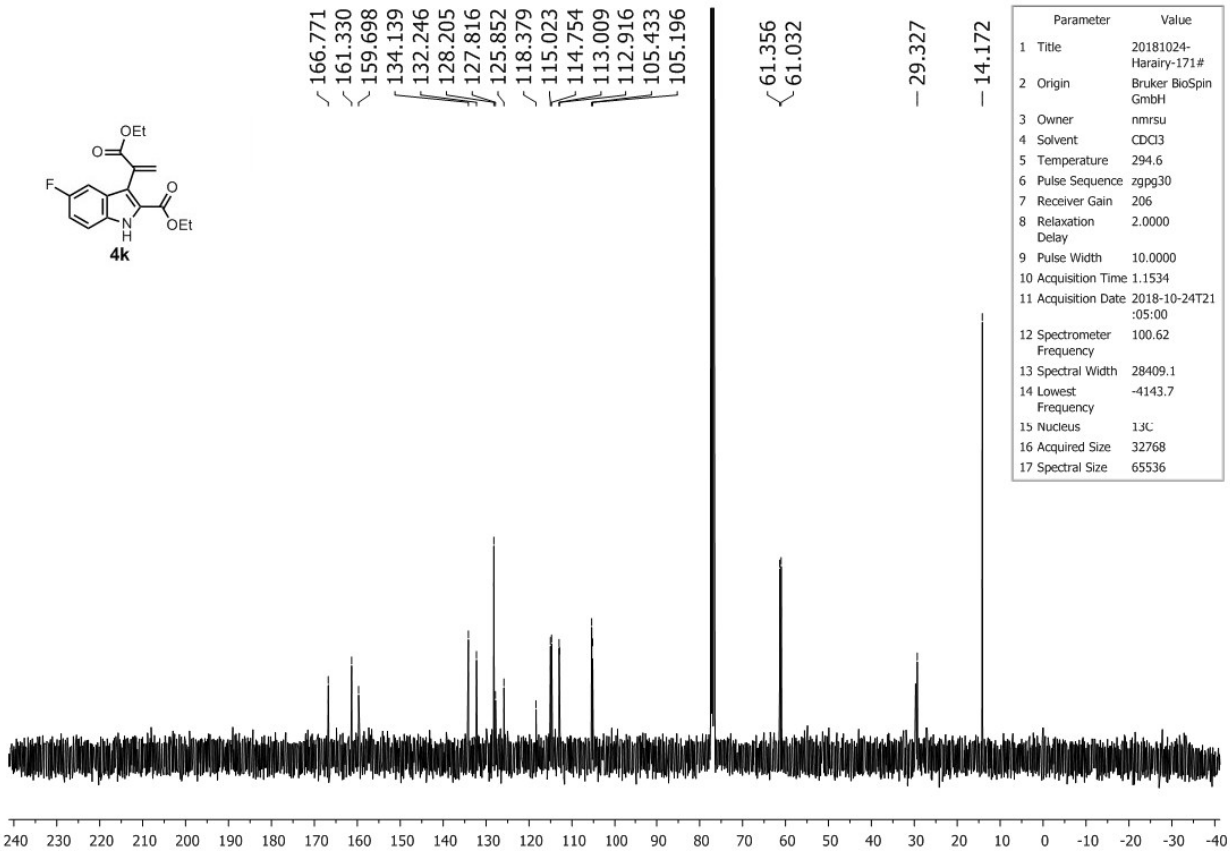
1.08
1.07
1.10
1.00
1.00
2.12
2.13
3.65
3.78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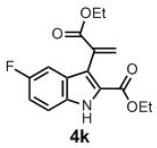
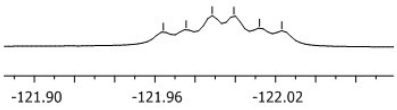
161.330
159.698
134.139
132.246
128.205
127.816
125.852
118.379
115.023
114.754
113.009
112.916
105.433
105.196
61.356
61.032
29.327
14.172
4k
Parameter Value
1 Title 20181024-Harairy-171#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.6
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-10-24T21:05:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536



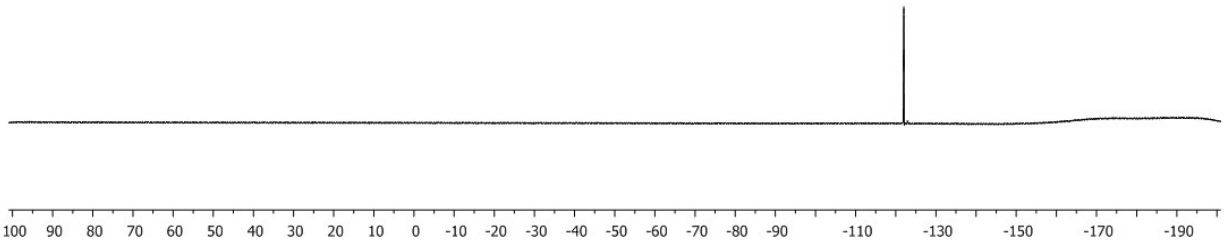
4k

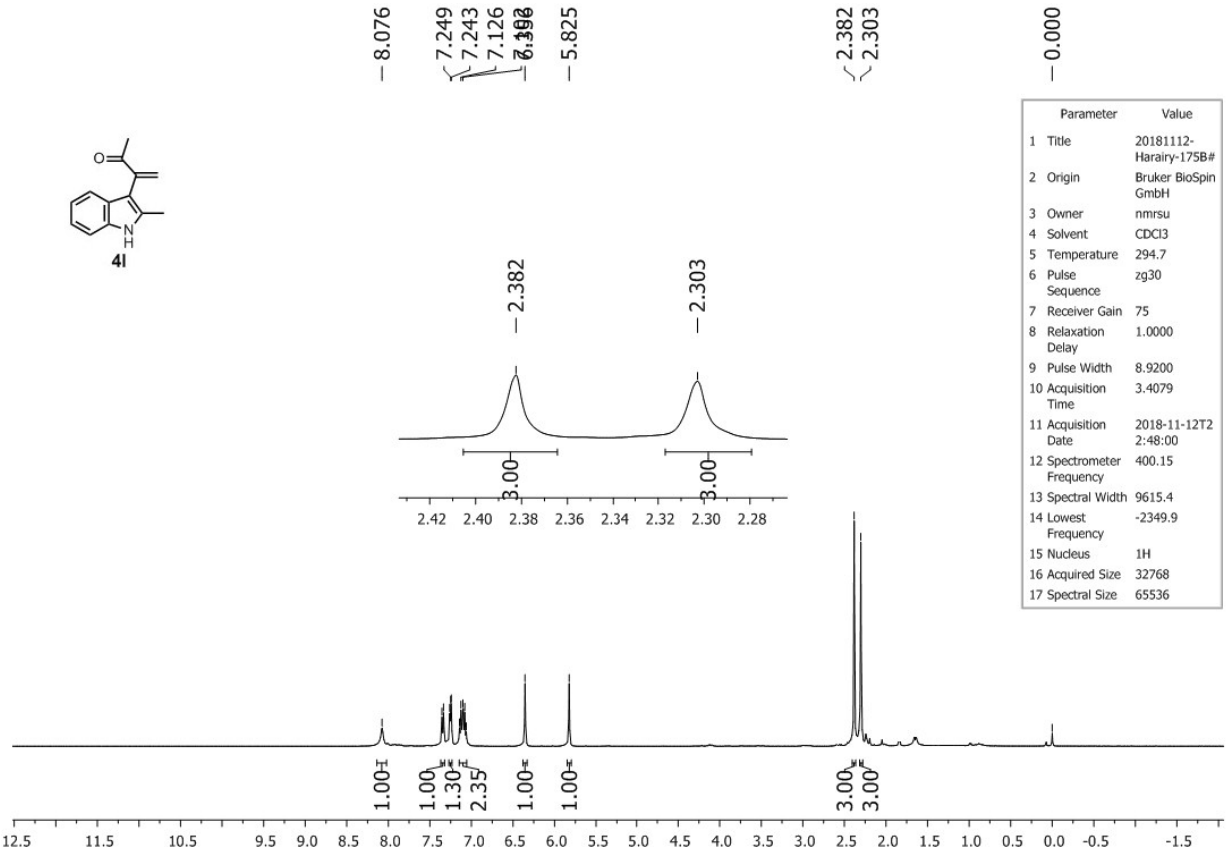
-121.964
-121.976
-121.989
-121.999
-122.012
-122.023

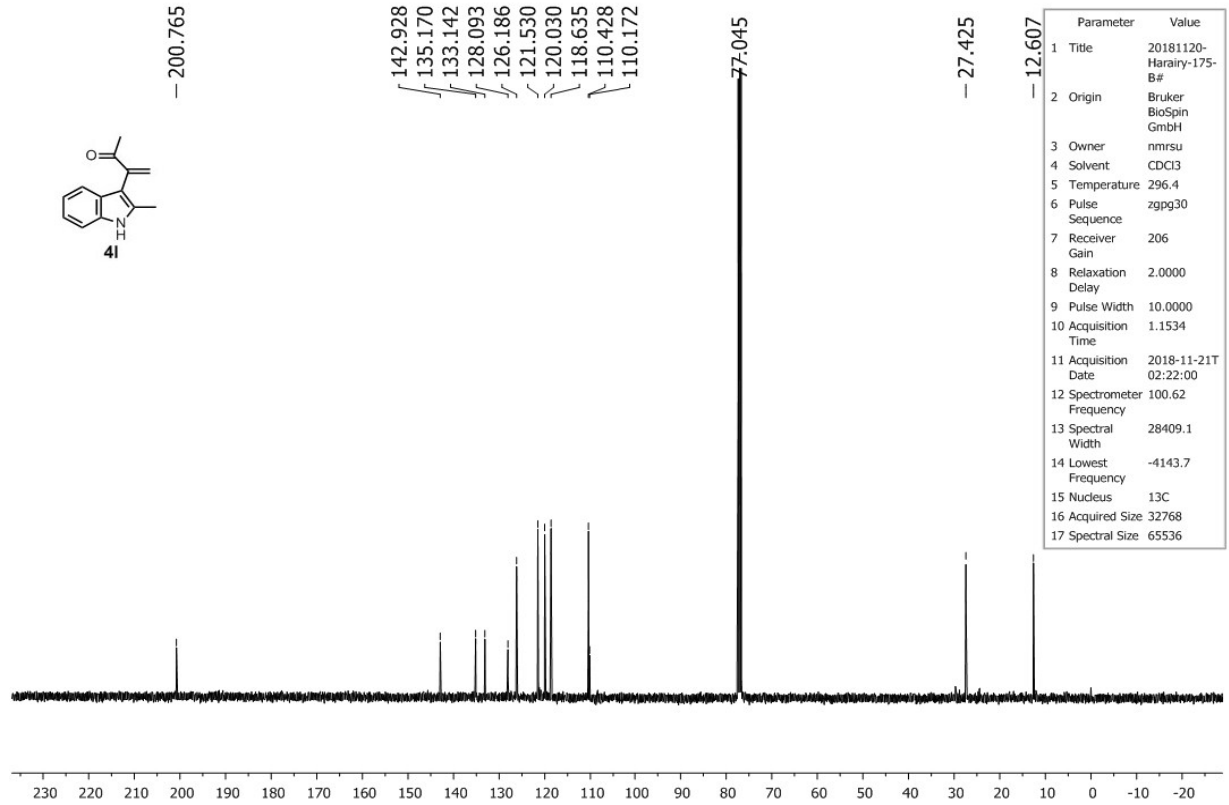


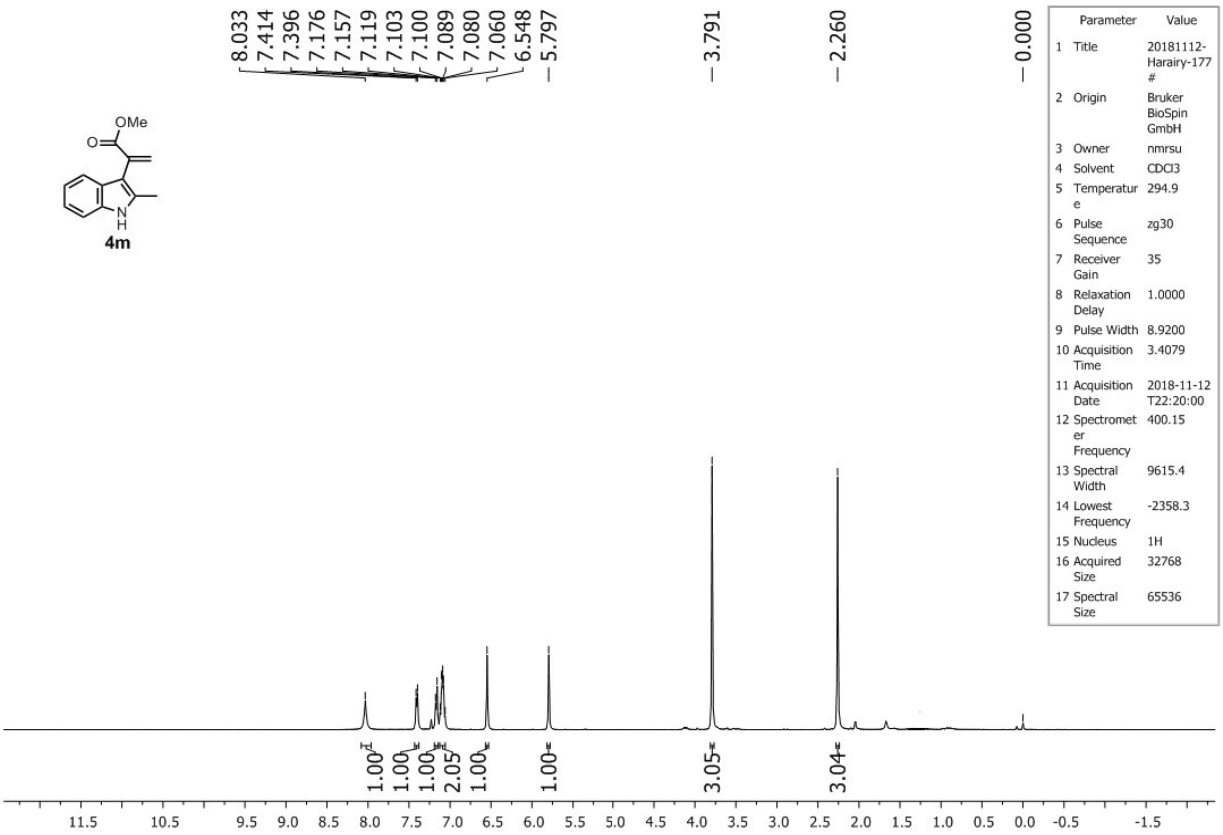
-121.964
-121.976
-121.989
-121.999
-122.012
-122.023

	Parameter	Value
1	Title	20181108-Harairy-171#
2	Origin	Bruker BioSpin GmbH
3	Owner	nmrsu
4	Solvent	CDCl3
5	Temperature	295.3
6	Pulse Sequence	zgflqn
7	Receiver Gain	206
8	Relaxation Delay	1.5000
9	Pulse Width	14.0000
10	Acquisition Time	0.5767
11	Acquisition Date	2018-11-08T23:05:00
12	Spectrometer Frequency	376.52
13	Spectral Width	113636.4
14	Lowest Frequency	-75644.0
15	Nucleus	19F
16	Acquired Size	65536
17	Spectral Size	131072




8.076
7.249
7.243
7.126
5.825
2.382
2.303
0.000
4l
2.382
2.303
3.00
3.00
2.42 2.40 2.38 2.36 2.34 2.32 2.30 2.28
Parameter Value
1 Title 20181112-Harairy-175B#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.7
6 Pulse Sequence zg30
7 Receiver Gain 75
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-11-12T12:48:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2349.9
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536
1.00 1.00 1.30 2.35 1.00 1.00 3.00 3.00
12.5 11.5 10.5 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 -1.5


200.765
142.928
135.170
133.142
128.093
126.186
121.530
120.030
118.635
110.428
110.172
77.045
27.425
12.607
4l
Parameter Value
1 Title 20181120-Harairy-175-B#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 296.4
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-11-21T02:22:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20


8.033
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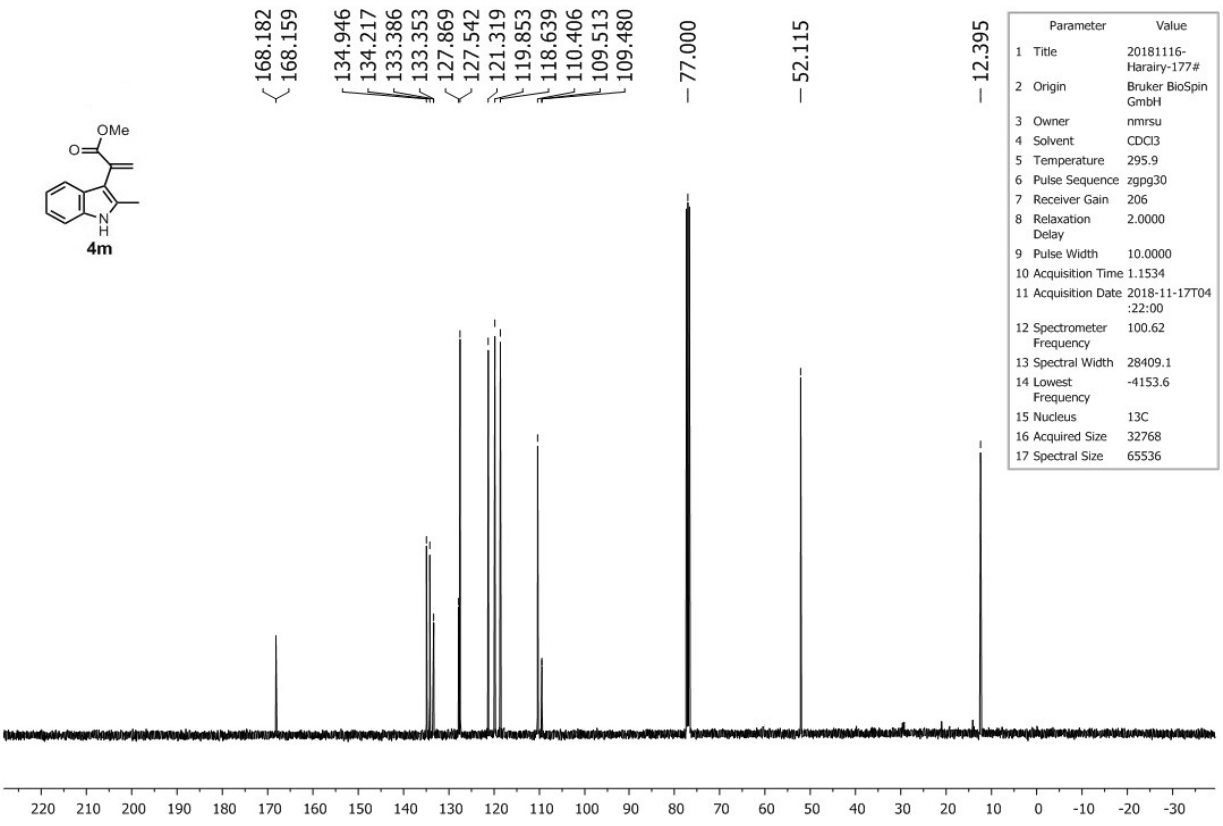
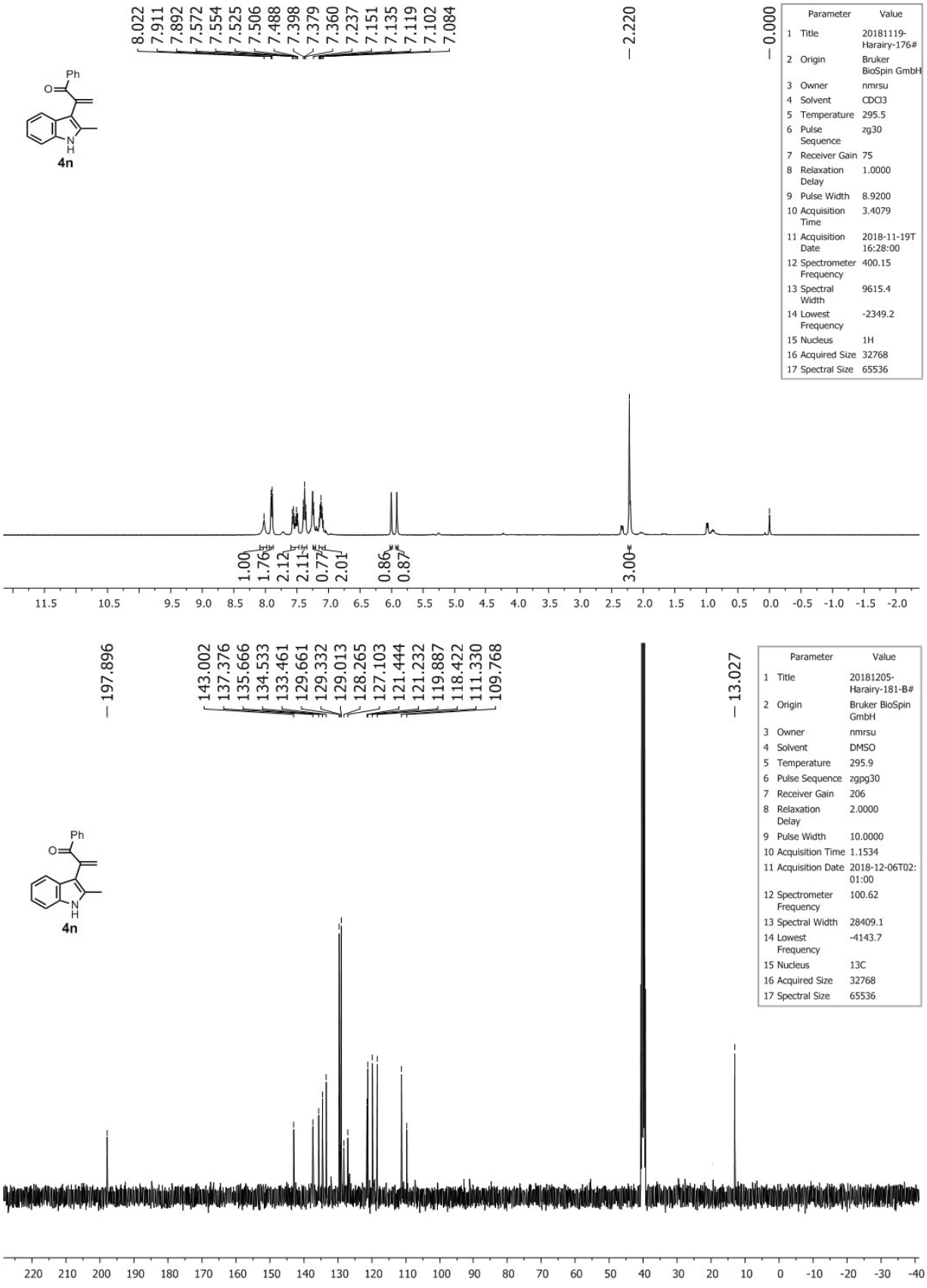
7.396
7.176
7.157
7.119
7.103
7.100
7.089
7.080
7.060
6.548
5.797
3.791
2.260
0.000
OMe
O
N
H
4m
1.00
1.00
1.00
2.05
1.00
1.00
3.05
3.04
Parameter Value
1 Title 20181112-Harairy-177#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.9
6 Pulse Sequence zg30
7 Receiver Gain 35
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-11-12T22:20:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2358.3
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536


168.182
168.159
134.946
134.217
133.386
133.353
127.869
127.542
121.319
119.853
118.639
110.406
109.513
109.480
77.000
52.115
12.395
OMe
O
N
H
4m
Parameter Value
1 Title 20181116-Harairy-177#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 295.9
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-11-17T04:22:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4153.6
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536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7.892
7.572
7.554
7.525
7.506
7.488
7.398
7.379
7.360
7.237
7.151
7.135
7.119
7.102
7.084
2.220
0.000
Ph
O
N
H
4n
Parameter Value
1 Title 20181119-Harairy-176#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 295.5
6 Pulse Sequence zg30
7 Receiver Gain 75
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-11-19T 16:28:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2349.2
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536
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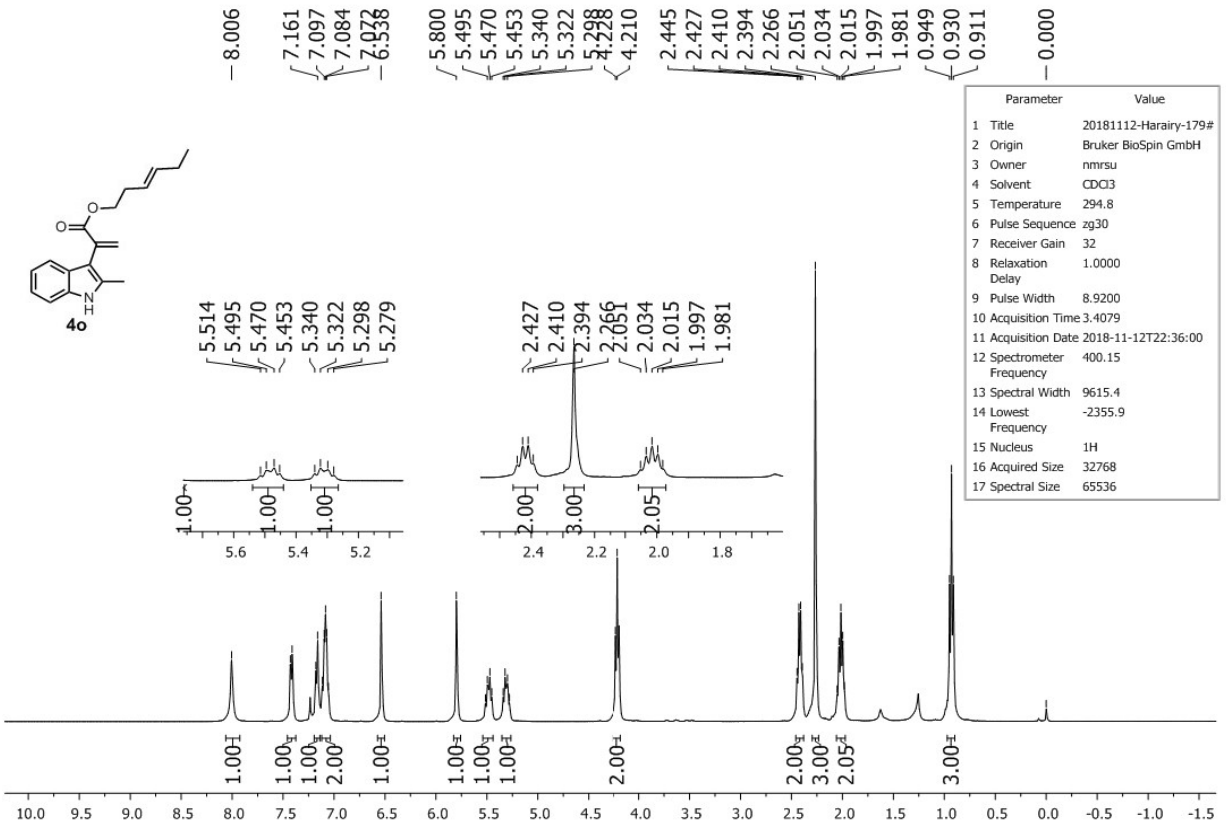
1.76
2.12
2.11
0.77
2.01
0.86
0.87
3.00
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
197.896
143.002
137.376
135.666
134.533
133.461
129.661
129.332
129.013
128.265
127.103
121.444
121.232
119.887
118.422
111.330
109.768
13.027
Ph
O
N
H
4n
Parameter Value
1 Title 20181205-Harairy-181-B#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent DMSO
5 Temperature 295.9
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-12-06T02: 01:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40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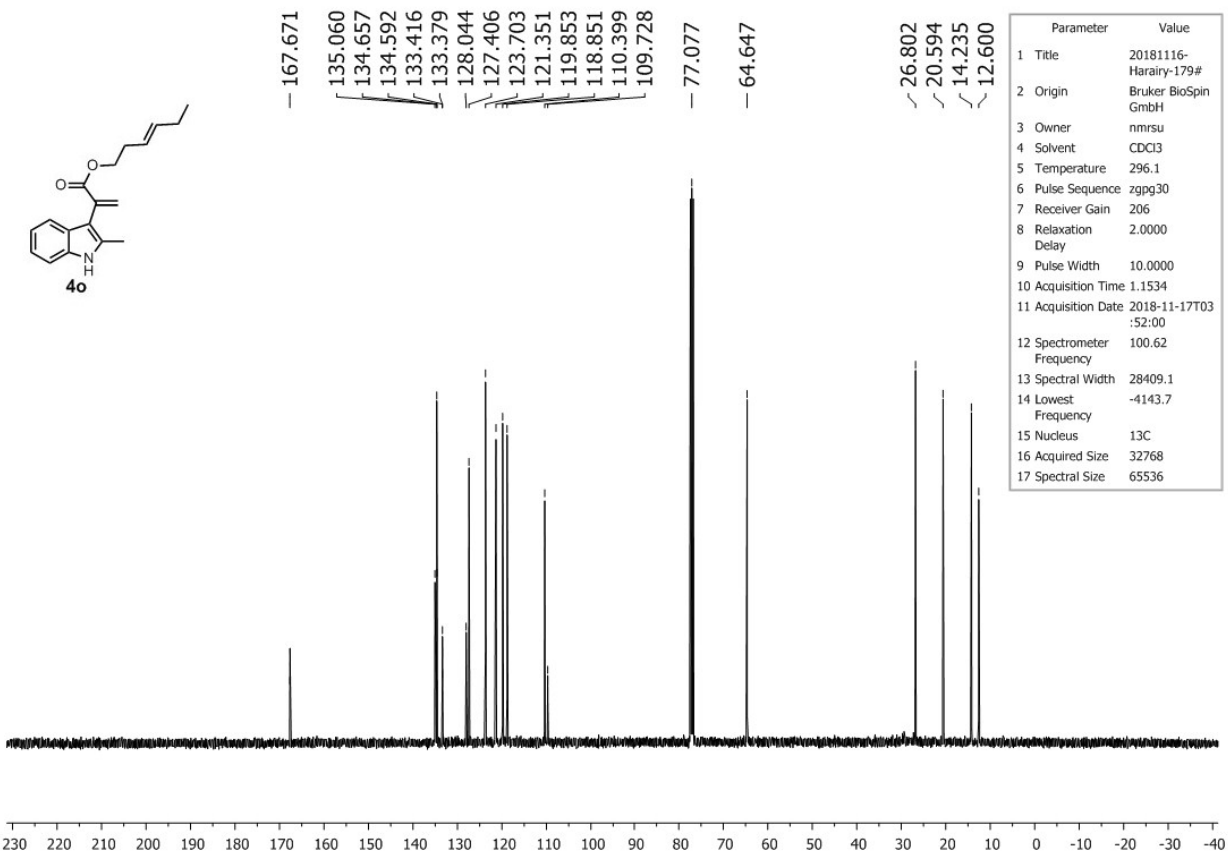
7.084
6.538
5.800
5.495
5.470
5.453
5.340
5.322
4.228
4.210
2.445
2.427
2.410
2.394
2.266
2.051
2.034
2.015
1.997
1.981
0.949
0.930
0.911
0.000
4o
Parameter Value
1 Title 20181112-Harairy-179#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.8
6 Pulse Sequence zg30
7 Receiver Gain 32
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-11-12T22:36:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2355.9
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536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133.379
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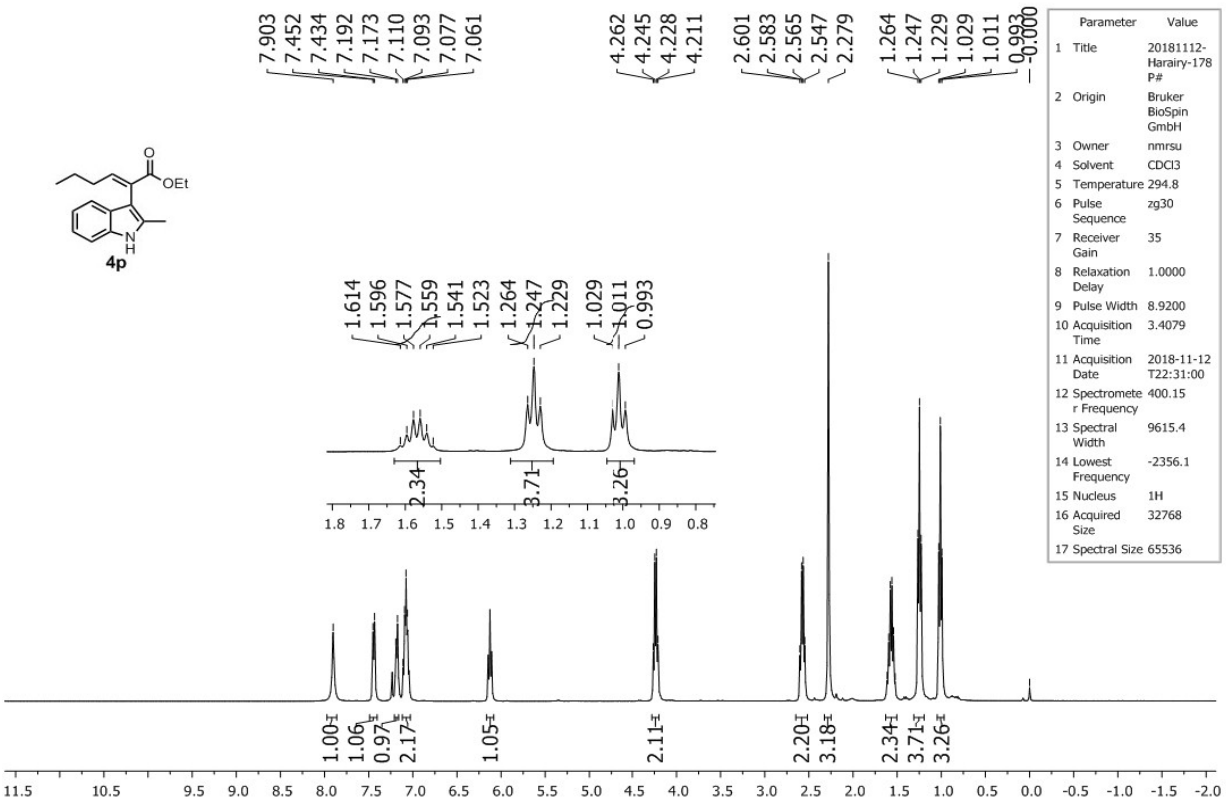
127.406
123.703
121.351
119.853
118.851
110.399
109.728
77.077
64.647
26.802
20.594
14.235
12.600
4o
Parameter Value
1 Title 20181116-Harairy-179#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 296.1
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-11-17T03:52:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536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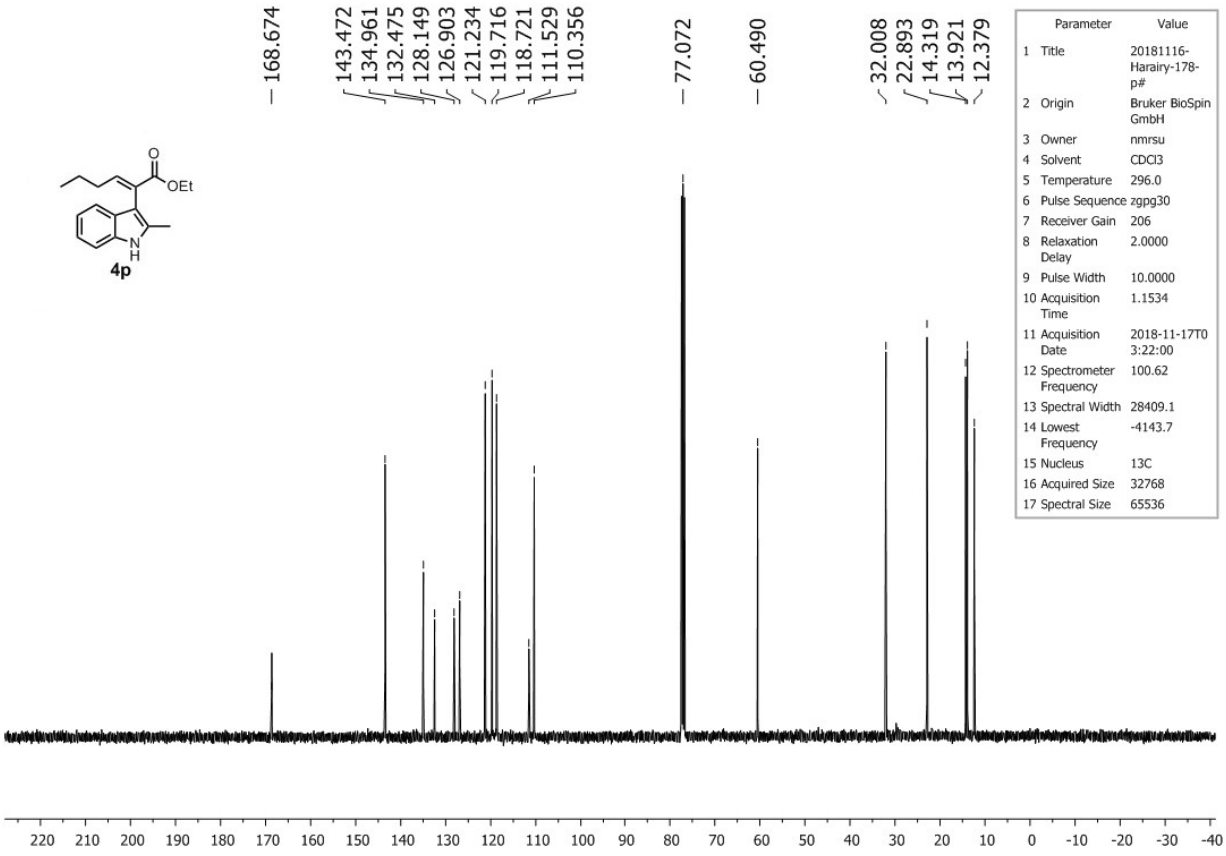
7.434
7.192
7.173
7.110
7.093
7.077
7.061
4.262
4.245
4.228
4.211
2.601
2.583
2.565
2.547
2.279
1.264
1.247
1.229
1.029
1.011
0.993
0.000
OEt
N
H
4p
1.614
1.596
1.577
1.559
1.541
1.523
1.264
1.247
1.229
1.029
1.011
0.993
2.34
3.71
3.26
1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8
1.00
1.06
0.97
2.17
1.05
2.11
2.20
3.18
2.34
3.71
3.26
11.5 10.5 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 -1.5 -2.0
Parameter Value
1 Title 20181112-Harairy-178 P#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.8
6 Pulse Sequence zg30
7 Receiver Gain 35
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-11-12 T22:31:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2356.1
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536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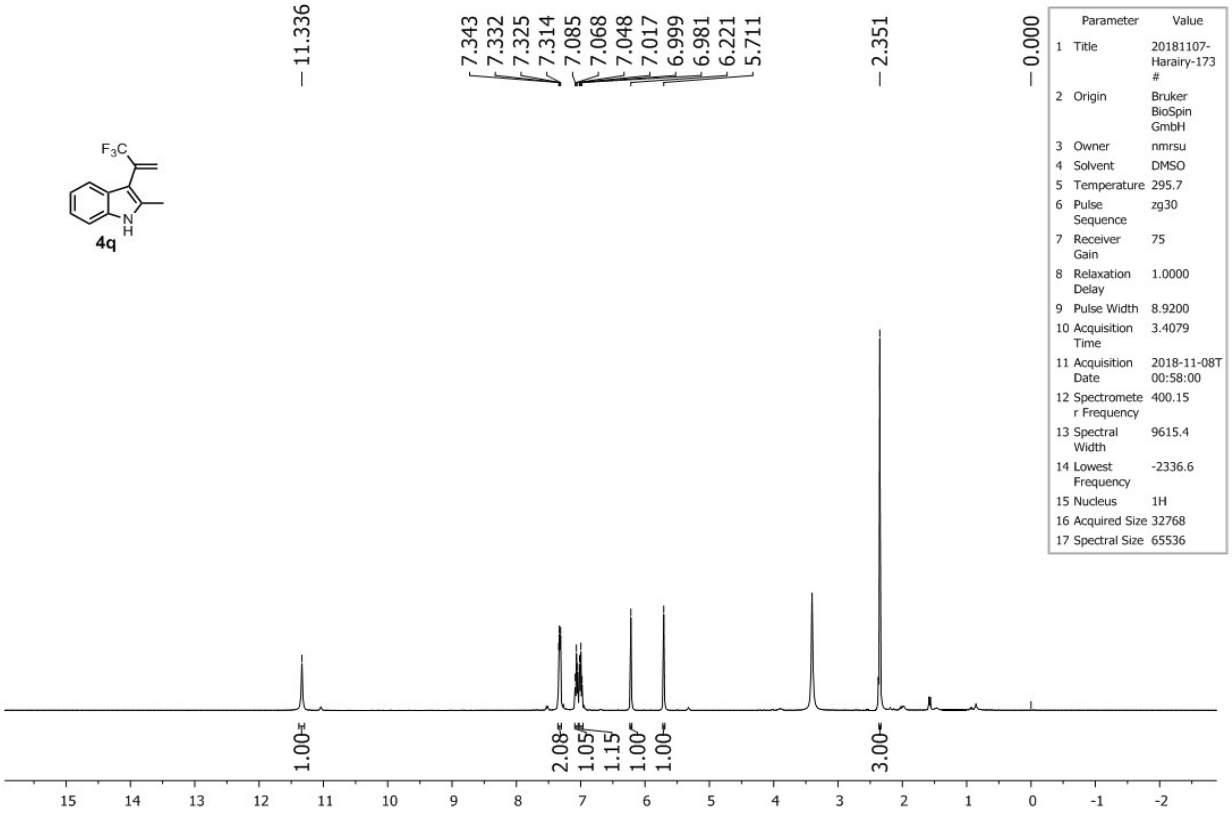
132.475
128.149
126.903
121.234
119.716
118.721
111.529
110.356
77.072
60.490
32.008
22.893
14.319
13.921
12.379
OEt
N
H
4p
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
Parameter Value
1 Title 20181116-Harairy-178-p#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 296.0
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-11-17T03:22:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536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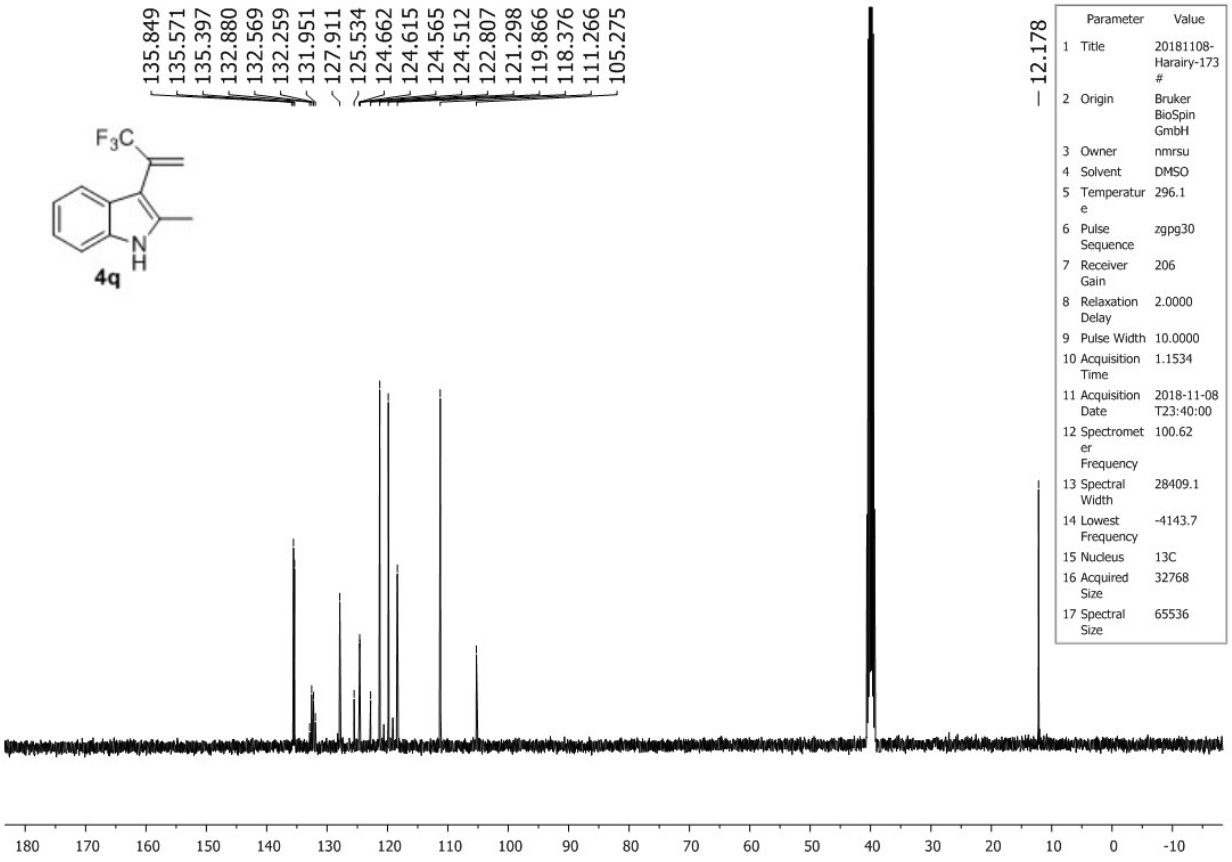
7.343
7.332
7.325
7.314
7.085
7.068
7.048
7.017
6.999
6.981
6.221
5.711
2.351
0.000
F3C
N
H
4q
1.00
2.08
1.05
1.15
1.00
1.00
3.00
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
Parameter Value
1 Title 20181107-Harairy-173 #
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent DMSO
5 Temperature 295.7
6 Pulse Sequence zg30
7 Receiver Gain 75
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-11-08T 00:58:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2336.6
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536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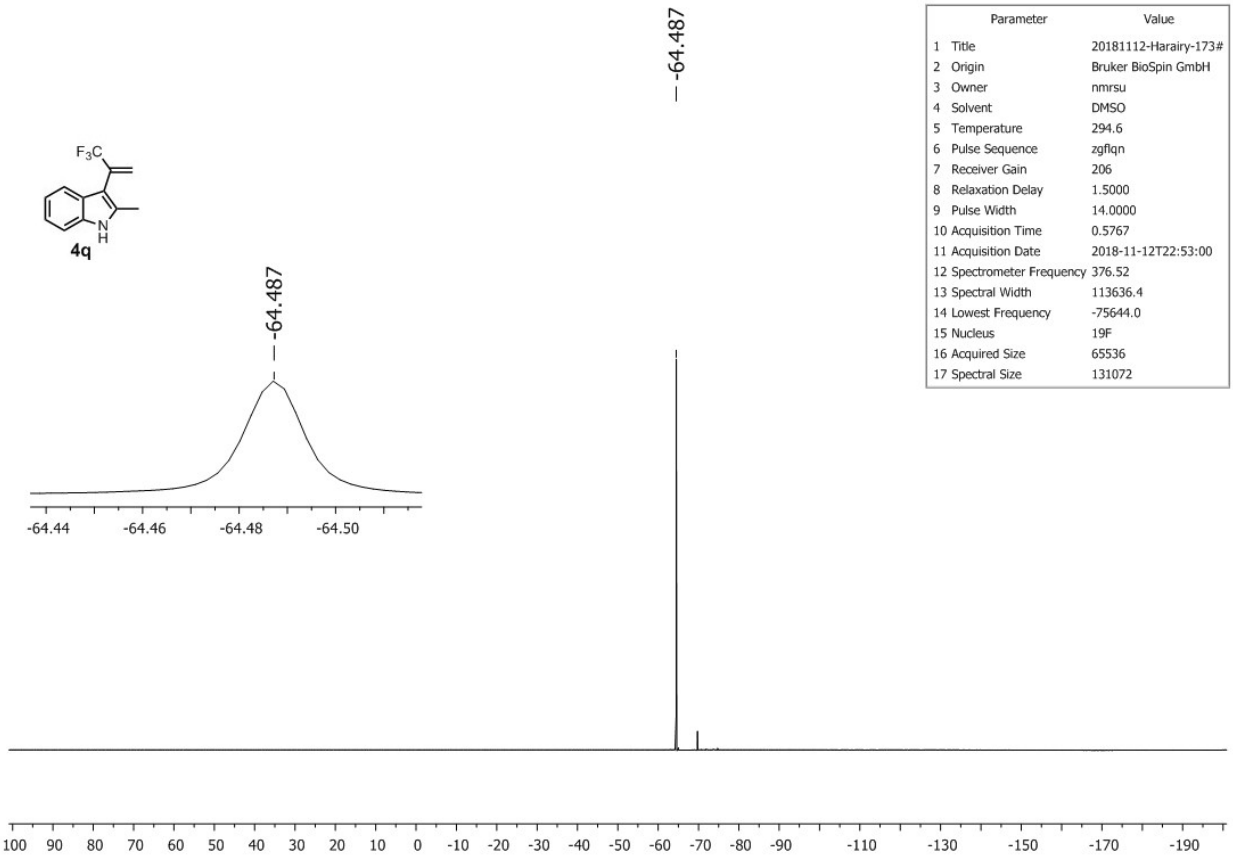
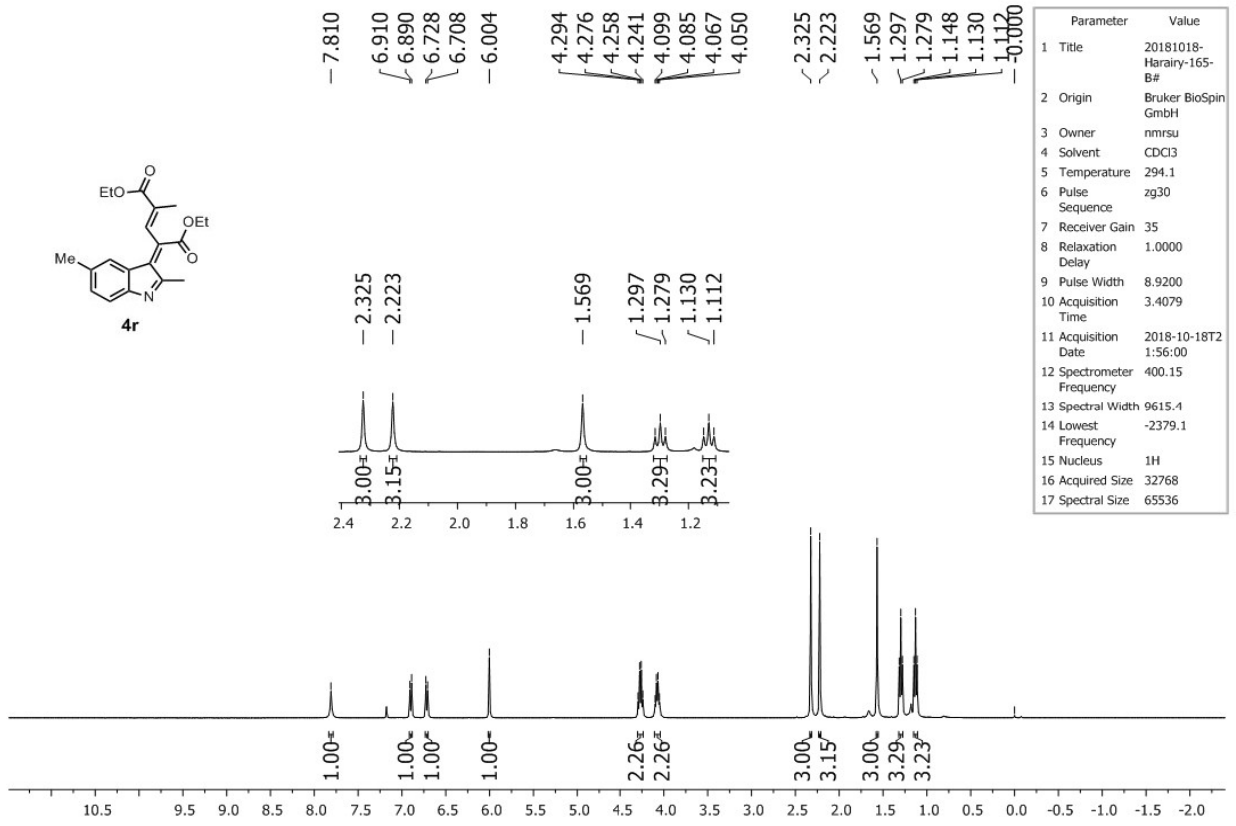
135.571
135.397
132.880
132.569
132.259
131.951
127.911
125.534
124.662
124.615
124.565
124.512
122.807
121.298
119.866
118.376
111.266
105.275
12.178
F3C
N
H
4q
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
Parameter Value
1 Title 20181108-Harairy-173 #
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent DMSO
5 Temperature 296.1
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-11-08 T23:40:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536



Parameter	Value
1 Title	20181112-Harairy-173#
2 Origin	Bruker BioSpin GmbH
3 Owner	nmrsu
4 Solvent	DMSO
5 Temperature	294.6
6 Pulse Sequence	zgflqn
7 Receiver Gain	206
8 Relaxation Delay	1.5000
9 Pulse Width	14.0000
10 Acquisition Time	0.5767
11 Acquisition Date	2018-11-12T22:53:00
12 Spectrometer Frequency	376.52
13 Spectral Width	113636.4
14 Lowest Frequency	-75644.0
15 Nucleus	19F
16 Acquired Size	65536
17 Spectral Size	131072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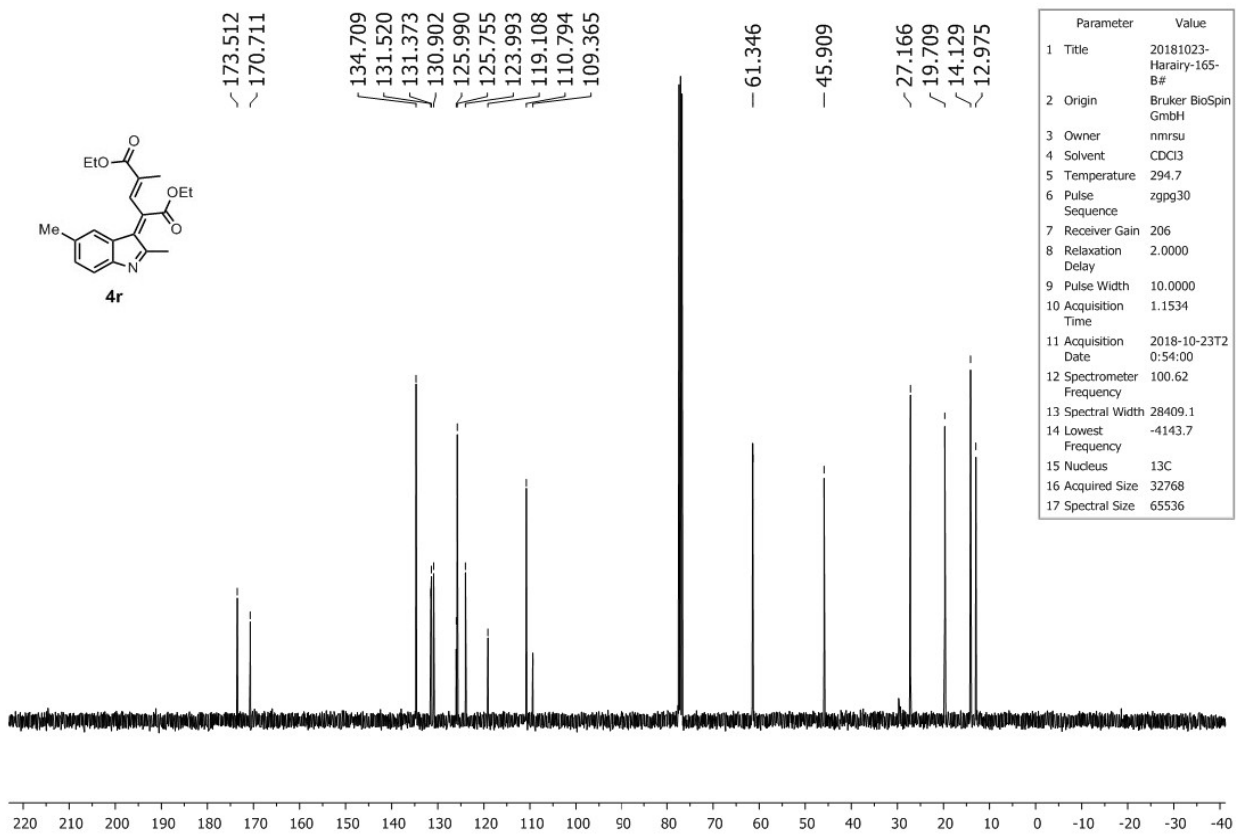
6.890
6.728
6.708
6.004
4.294
4.276
4.258
4.241
4.099
4.085
4.067
4.050
2.325
2.223
1.569
1.297
1.279
1.148
1.130
1.112
0.000
EtO
OEt
Me
4r
Parameter Value
1 Title 20181018-Harairy-165-B#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.1
6 Pulse Sequence zg30
7 Receiver Gain 35
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-10-18T21:56:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2379.1
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536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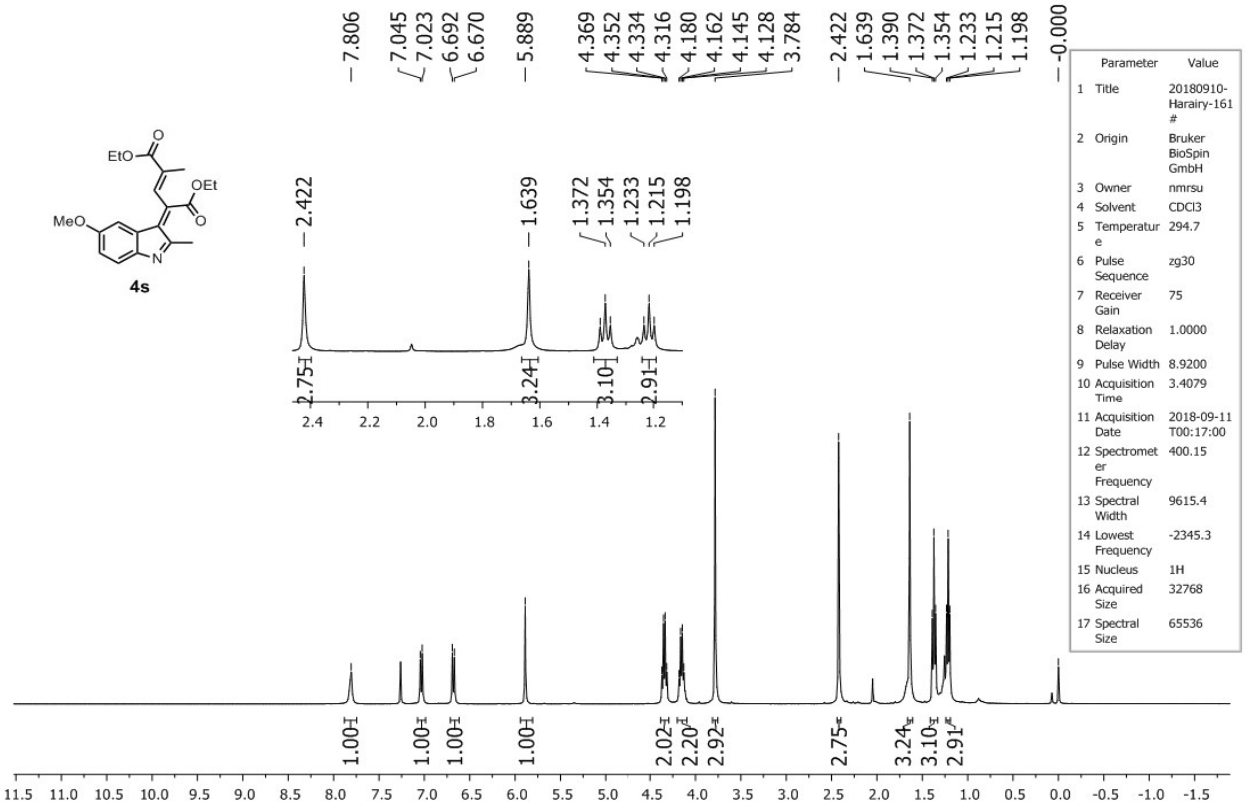
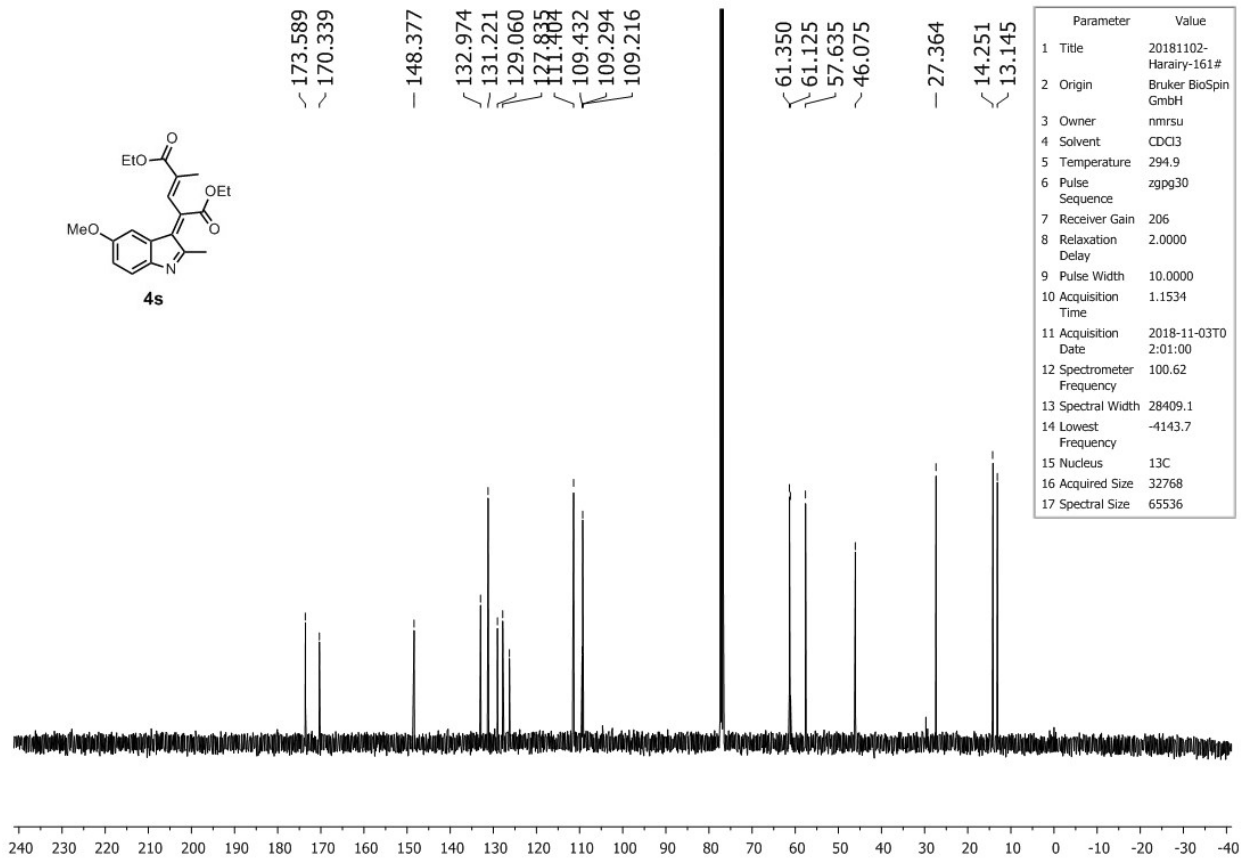
134.709
131.520
131.373
130.902
125.990
125.755
123.993
119.108
110.794
109.365
61.346
45.909
27.166
19.709
14.129
12.975
EtO
OEt
Me
4r
Parameter Value
1 Title 20181023-Harairy-165-B#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.7
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-10-23T20:54:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536


7.806
7.045
7.023
6.692
6.670
5.889
4.369
4.352
4.334
4.316
4.180
4.162
4.145
4.128
3.784
2.422
1.639
1.390
1.372
1.354
1.233
1.215
1.198
-0.000
EtO
OEt
MeO
4s
Parameter Value
1 Title 20180910-Harairy-161#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.7
6 Pulse Sequence zg30
7 Receiver Gain 75
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-09-11T00:17:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2345.3
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536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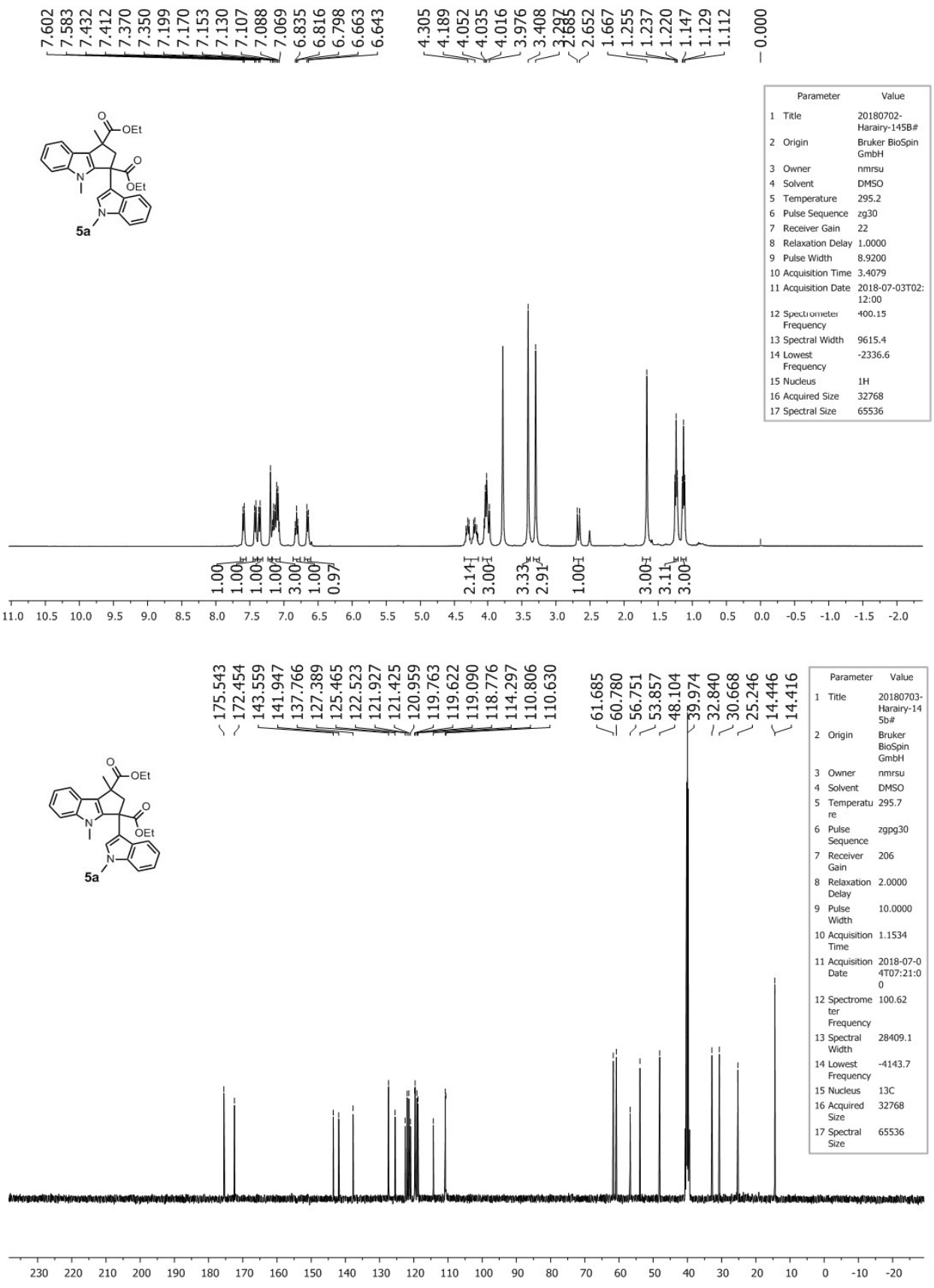
148.377
132.974
131.221
129.060
127.835
111.404
109.432
109.294
109.216
61.350
61.125
57.635
46.075
27.364
14.251
13.145
EtO
OEt
MeO
4s
Parameter Value
1 Title 20181102-Harairy-161#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent CDCl3
5 Temperature 294.9
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-11-03T02:01:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536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7.432
7.412
7.370
7.350
7.199
7.170
7.153
7.130
7.107
7.088
7.069
6.835
6.816
6.798
6.663
6.643
4.305
4.189
4.052
4.035
4.016
3.976
3.408
3.297
3.285
2.652
1.667
1.255
1.237
1.220
1.147
1.129
1.112
0.000
OEt
OEt
5a
Parameter Value
1 Title 20180702-Harairy-145B#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent DMSO
5 Temperature 295.2
6 Pulse Sequence zg30
7 Receiver Gain 22
8 Relaxation Delay 1.0000
9 Pulse Width 8.9200
10 Acquisition Time 3.4079
11 Acquisition Date 2018-07-03T02:12:00
12 Spectrometer Frequency 400.15
13 Spectral Width 9615.4
14 Lowest Frequency -2336.6
15 Nucleus 1H
16 Acquired Size 32768
17 Spectral Size 65536
1.00
1.00
1.00
1.00
3.00
1.00
0.97
2.14
3.00
3.33
2.91
1.00
3.00
3.11
3.00
175.543
172.454
143.559
141.947
137.766
127.389
125.465
122.523
121.927
121.425
120.959
119.763
119.622
119.090
118.776
114.297
110.806
110.630
61.685
60.780
56.751
53.857
48.104
39.974
32.840
30.668
25.246
14.446
14.416
OEt
OEt
5a
Parameter Value
1 Title 20180703-Harairy-145b#
2 Origin Bruker BioSpin GmbH
3 Owner nmrsu
4 Solvent DMSO
5 Temperature 295.7
6 Pulse Sequence zgpg30
7 Receiver Gain 206
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 1.1534
11 Acquisition Date 2018-07-04T07:21:00
12 Spectrometer Frequency 100.62
13 Spectral Width 28409.1
14 Lowest Frequency -4143.7
15 Nucleus 13C
16 Acquired Size 32768
17 Spectral Size 65536