Supporting Information for:

Synthesis of a BDPA-TEMPO Biradical

Eric L. Dane, Thorsten Maly, Galia T. Debelouchina, Robert G. Griffin, Timothy M. Swager

Department of Chemistry and Francis Bitter Magnet Lab

Massachusetts Institute of Technology

77 Massachusetts Ave., Cambridge MA 02139 USA

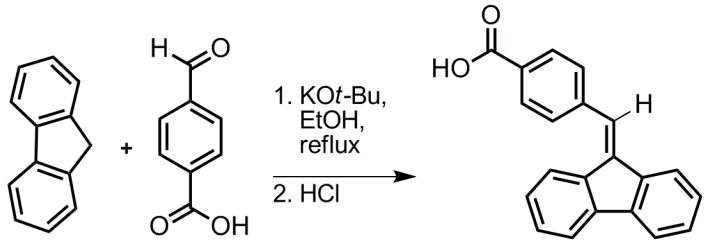
tswager@mit.edu

Table of contents **Page**

Materials. All chemicals, reagents, and solvents were used as received from commercial sources without further purification except dimethylacetamide,

dimethylformamide, and dimethylsufloxide, which were dried over oven-activated 4-Å molecular sieves.

Instrumentation. Proton nuclear magnetic resonance (^{1}H NMR) spectra and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on an Inova-500 (500 MHz) NMR spectrometer. Chemical shifts for protons are reported in parts per million downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent. Chemical shifts for carbon are reported in parts per million downfield from tetramethylsilane and are referenced to the carbon resonances of the solvent. Data are represented as follows: chemical shift, integration, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q= quartet, m = multiplet), and coupling constants in Hertz (Hz). The mass spectrometry data were obtained at the MIT mass spectrometry facility, using a Bruker Daltonics APEX II 3T FT-ICR-MS. Elemental analysis was obtained by Columbia Analytical Services, Tucson, AZ.

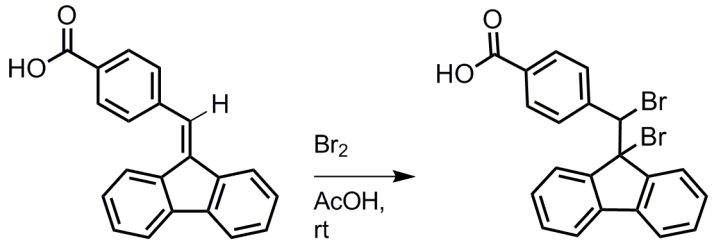


4

Compound 4: To an oven-dried 500-mL round-bottom flask containing a magnetic stir-bar were added 5.00 g (30.1 mmol, 1.00 equiv) of fluorene and 9.27 g (82.8 mmol, 2.75 equiv) of *t*-BuOK, followed by 300 mL of absolute ethanol. The flask was fitted with a water-cooled reflux condenser and heated to reflux with vigorous stirring, at which time 5.50 g (36.7 mmol, 1.20 equiv) of 4-carboxybenzaldehyde was added and the flask was allowed to reflux overnight. After being allowed to cool to room temperature, the reaction mixture was poured into a large flask containing excess 1M HCl and ice. This mixture was extracted with 2 portions of ethyl acetate (approximately 500 mL total), and the organic layer was subsequently washed three times with dilute sodium bicarbonate solution (pH 9) to remove terepthalic acid, which is an impurity in the 4-carboxybenzaldehyde starting material. Finally, the organic layer was washed with brine and dried over sodium sulfate. After removal of the ethyl acetate, the dark yellow material was twice refluxed in 30 mL of toluene, allowed to cool, and then filtered, to obtain 6.25 g of a bright yellow powder which contains a small amount of 4-methylbenzoic acid. This material can be purified by recrystallization from a refluxing mixture of tetrahydrofuran and acetic acid with significant losses of material (3.38 g of material yielded 1.87 g of pure material). Therefore, the impure material was carried through to the next step.

Compound 4:
^{1}H NMR (500 MHz, d_6-dimethylsulfoxide): δ 13.1 (1H, s), 8.07 (2H, d, J = 7.5 Hz), 7.99 (1H, d, J = 7.5 Hz), 7.96 (1H, s), 7.89 (1H, d, 7.5 Hz), 7.87 (1H, d, 7.5 Hz), 7.73 (2H, d, 8.0 Hz), 7.44 (2H, m), 7.37 (2H, t, J=7.0 Hz), 7.13 (1H, td (J= 7.0, 1.0 Hz).

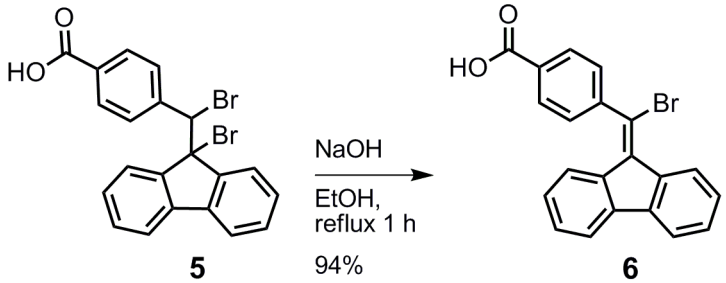
^{13}C NMR (125 MHz, d_6-dimethylsulfoxide): 167.0, 140.9, 140.8, 138.7, 138.5, 136.5, 135.4, 130.2, 129.6, 129.3, 129.1, 128.7, 127.3, 127.2, 127.0, 123.8, 121.0, 120.3, 119.9.
HRMS (ESI): 297.0924 [calc'd for M-H$^-$: 297.0921].
FT-IR: ν_{max}(KBr)/cm^{-1} 1674 s, 1601 s, 1419 m, 1290 s, 722 m.
MP: 250-255 °C (from tetrahydrofuran/acetic acid)



4 **5**

Compound 5: To an oven-dried 250-mL round-bottom flask equipped with a magnetic stir-bar were added 2.57 g of acid **4** (8.61 mmol, 1.00 equiv) and 65 mL of glacial acetic acid. The resulting suspension was heated to reflux at which time additional acetic acid was added drop-wise until a homogeneous solution was formed. The mixture was cooled to room temperature and a slight excess of bromine (0.47 mL, 1.45 g, 9.06 mmol, 1.10 equiv) was added slowly over 5 minutes. The suspension became a homogeneous red solution and was allowed to stir overnight at room temperature until a white precipitate formed. The resulting suspension was filtered and the filter cake was washed with hexanes to afford 2.85 g (6.22 mmol) of a white powder and a yield of 50% over two steps based on fluorene as the limiting reagent.

Compound 5:
^{1}H NMR (500 MHz, d_8-tetrahydrofuran): δ 10.86 (1H, bs), 8.21 (1H, d, J = 6.5 Hz), 7.69 (1H, d, J = 8.5 Hz), 7.60 (1H, 5.5 Hz), 7.56 (2H, d, J = 8.5), 7.44 (3H, m), 7.25 (2H, qd, J = 7.5 Hz, 1.5 Hz), 6.97 (2H, d, J = 8.0 Hz), 6.19 (1H, s).
^{13}C NMR (125 MHz, d_6-dimethylsulfoxide): 166.2, 144.0, 140.8, 138.6, 137.7, 130.1, 129.8, 129.4, 128.7, 127.8 (2), 127.7 (2), 126.0, 125.6, 120.2, 120.0, 65.8, 59.9.
HRMS (ESI): 454.9291 [calc'd for M-H$^-$: 454.9288].
FT-IR: ν_{max}(KBr)/cm^{-1} 1690 s, 1420 m, 1286 s, 743 m.
MP: 185-187 °C with decomposition (from acetic acid).

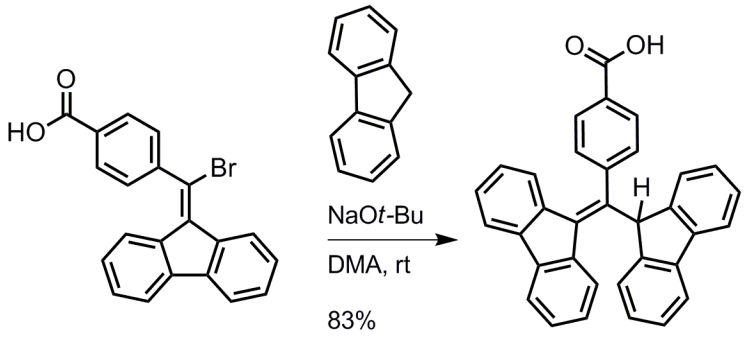


5 **6**

Compound 6: To an over-dried 250-mL round-bottom flask equipped with a stir-bar were added 1.01 g of dibromide **5** (2.21 mmol, 1.00 equiv) and 5.00 g (50 pellets) of

NaOH (125 mmol, 56.6 equiv). To the flask was added 125 mL of absolute ethanol and a water-cooled reflux condenser was attached. The reaction was refluxed for 60 minutes and then allowed to cool to room temperature. The solution was acidified with dilute hydrochloric acid and then extracted with 100 mL of ethyl acetate, which was subsequently washed with brine and dried over sodium sulfate. Removal of the solvent yielded 0.786 g (2.08 mmol, 94 % yield) of a yellow powder.

Compound 6:
^{1}H NMR (500 MHz, d_8-dimethylsulfoxide): δ 8.76 (1H, d, J = 7.5 Hz), 8.12 (2H, dd, J = 7.5, 1.5 Hz), 7.94 (1H, d, J = 7.5 Hz), 7.85 (1H, d, J = 7.5 Hz), 7.62 (2H, d, J = 7.5 Hz), 7.52 (1H, t, J = 7.0 Hz), 7.45 (1H, t, J= 7.0 Hz), 7.28 (1H, t, J = 7.0 Hz), 6.92 (1H, t, J = 7.0 Hz), 6.08 (1H, t, J = 7.5 Hz).
^{13}C NMR (125 MHz, d_6-dimethylsulfoxide): 166.6, 146.0, 140.6 139.3, 136.9, 136.8, 135.4, 131.5, 130.4, 129.8, 128.8, 128.5, 127.4, 127.2, 125.5(2), 123.0, 120.2, 120.0.
HRMS (EI): 376.0106 [calc'd 376.0093].
FT-IR: ν_{max}(KBr)/cm^{-1} 1691 s, 1604 w, 1445 m, 728 m.

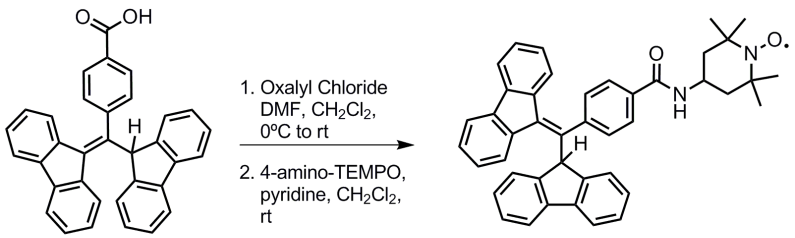


6 **7**

Compound 7: To an over-dried 100-mL Schlenk flask equipped with a stir-bar and rubber septum were added 0.182 g (1.10 mmol, 1.50 equiv) of fluorene and 0.350 g (3.65 mmol, 5.00 equiv) of NaO*t*-Bu. The flask was evacuated under high vacuum and refilled with argon three times and 25 mL of dry dimethylacetamide (DMA) was added. The solution appeared dark red and was allowed to stir for 5 minutes. In a separate oven-dried 25-mL schlenk flask, 0.272 g (0.730 mmol, 1.00 equiv) of bromide **6** was added. The flask was evacuated under high vacuum and refilled with argon three times and 10 mL of dry DMA was added. After complete dissolution of bromide **6** in the DMA, the solution was slowly added to the 100-mL Schlenk flask by cannulation over a period of 10 minutes. The reaction immediately turned deep blue and was stirred for 1 hour at room temperature. Next, the flask was cooled in an ice bath and aqueous 1M HCl was added by pipette until the blue color disappeared. The solution was then further diluted with water and extracted into ethyl acetate, which was washed two times with neutral water and once with brine. The organic layer was dried over sodium sulfate and then removed to yield an orange solid. This orange solid was was purified by silica gel flash chromatography eluting with a gradient from pure DCM to 95 DCM : 5 MeOH. The solvent was removed and the solid was dissolved in approximately 1 mL of tetrahydrofuran(THF) and 2 mL of acetic acid with heating. This solution was allowed to cool to room temperature and the THF was allowed to evaporate, yielding, after filtration

and washing with hexanes, 0.280 g (0.605 mmol, 83 % yield) of acid **7** as an off-white powder**.**

Compound 7:
^{1}H NMR (500 MHz, d_8-tetrahydrofuran): δ 8.52 (1H, dd, J = 7.5, 1.0 Hz), 7.92 (1H, dq, J = 7.5, 0.50 Hz), 7.77 (1 H, dq, J = 7.5, 0.50 Hz), 7.68 (2H, dt, J = 6.5, 1.0 Hz), 7.63 (4H, m), 7.45 (1H, td, J = 7.5, 1.0 Hz), 7.36 (1H, td, J = 7.0, 1.0 Hz), 7.31 (2H, tt, J = 7.0, 1.0 Hz), 7.25 (2H, td, J = 7.5, 1.0 Hz), 7.18 (1H, td, J = 7.5, 1.0), 6.77 (2H, d, J = 7.5 Hz), 6.75 (1H, m), 6.56 (1H, s), 5.90 (1H, dt, J = 8.0, 1.0).
^{13}C NMR (125 MHz, d_8-tetrahydrofuran): 167.3, 145.1, 145.0, 144.3, 143.2, 142.5, 141.1, 140.2, 139.6, 137.2, 130.8, 130.1, 129.6, 129.3, 128.6, 128.5, 128.4, 127.9, 127.4, 127.0, 126.3, 125.2, 121.0, 120.8, 120.6, 120.1, 53.6.
HRMS (ESI): 461.1552 [calc'd for M-H$^-$: 461.1547].
FT-IR: ν_{max}(KBr)/cm^{-1} 1692 s, 1606 m, 1445 m, 1290 m, 730 m.
MP: >280 °C with decomposition (from tetrahydrofuran/acetic acid).



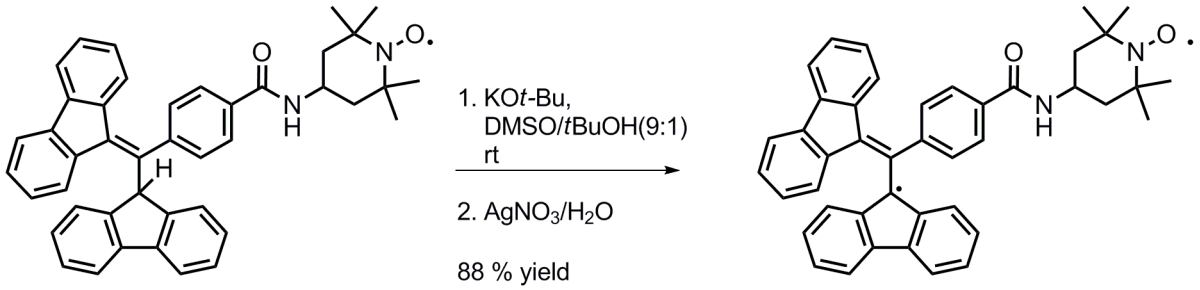
7 **8**

Compound 8: To an oven-dried 100-mL Schlenk flask equipped with a magnetic stir-bar and rubber septum was added 0.200 g (0.432 mmol, 1.00 equiv) of acid **7**. The flask was evacuated under high vacuum and refilled with argon three times and then 10 mL of dry dichloromethane (DCM) was added along with three drops of dry dimethylformamide (DMF). The reaction flask was cooled to 0 °C in an ice-bath and 0.080 mL (0.86 mmol, 2.0 equiv) of oxalyl chloride was added. The flask was slowly warmed to room temperature. After 1 hour at room temperature, the solvent was removed under high vacuum to obtain a yellow solid, which was then redissolved in 20 mL of dry DCM. In a separate flask, 0.149 g (0.864 mmol, 2.00 equiv) of 4-amino-TEMPO was added and the flask was evacuated and refilled with argon three times. To the flask was added 10 mL of dry DCM and 0.14 mL of dry pyridine (4 equiv) and the resulting solution was stirred in an ice-bath. The solution containing the acid chloride of **7** was slowly added to the amine containing flask, after which the flask was allowed to warm to room temperature overnight with stirring. The solution was washed three times with dilute acid and once with brine. It was dried over sodium sulfate and the solvent was removed. The material was purified by silica gel flash chromatography eluting with a gradient from pure DCM to 90 DCM : 10 ethyl acetate. After removing the solvent, 0.246 g (0.399 mmol, 92 % yield) of amide **8** was obtained as a pink powder. To characterize by NMR, 20 mgs of material was dissolved in 2 mL methanol and excess ascorbic acid was added to reduce the TEMPO radical to a hydroxylamine. After 10 minutes, the solvent was removed and the reduced compound was taken up in 2 mL of

chloroform. After removal of the chloroform, the resulting white solid was dissolved in 0.7 mL of deuterated acetone. The material still contained some ascorbic acid and it's oxidation products.

Compound 8:
HRMS (ESI): 616.3067 [calc'd for M+H: 616.3084].
Elemental Analysis: Theoretical: C: 83.87, H: 6.38, N: 4.55 Found: C: 82.99 H: 6.31 N: 4.34
FT-IR: ν_{max}(KBr)/cm^{-1} 1652 s, 1607 m, 1539 s, 1498 m, 1446 s, 1322 m, 1242 m, 732 s.

Compound 8H:
^{1}H NMR (500 MHz, d_6-acetone): δ 8.57 (1H, dd, J = 7.5, 1.0 Hz), 8.01 (1H, dd, J = 7.5, 1.0 Hz), 7.87 (1 H, dq, J = 7.5, 0.50 Hz), 7.76 (2H, dt, J = 7.0, 1.0 Hz), 7.69 (2H, d, J = 7.0 Hz), 7.56 (2H, d, J = 7.0 Hz), 7.52 (1H, t, J = 7.0 Hz), 7.45 (1H, t, J = 7.0 Hz), 7.35 (5H, m), 7.25 (1H, t, J = 7.0 Hz), 6.79 (1H, m), 6.78 (2H, d, J = 7.5 Hz), 6.54 (1H, s), 5.85 (1H, d, J = 8.0), 4.28 (1H, m), 1.82 (2H, dd, J = 12.0, 3.0 Hz), 1.50 (2H, t, J = 12.0 Hz), 1.18 (6H, s), 1.12 (6H, s).
^{13}C NMR (125 MHz, d_6-acetone): 166.1, 146.0, 145.8, 143.8, 143.5 (2), 143.0, 141.8, 140.4, 140.2, 137.8, 135.4, 130.3, 130.3, 129.6, 129.5 (2), 128.9, 128.5, 128.3, 127.8, 127.2, 127.0, 121.9, 121.8, 121.1, 53.3, 45.4, 41.9, 32.3, 20.3.
HRMS (ESI): 617.3142 [calc'd for M+H: 617.3163].

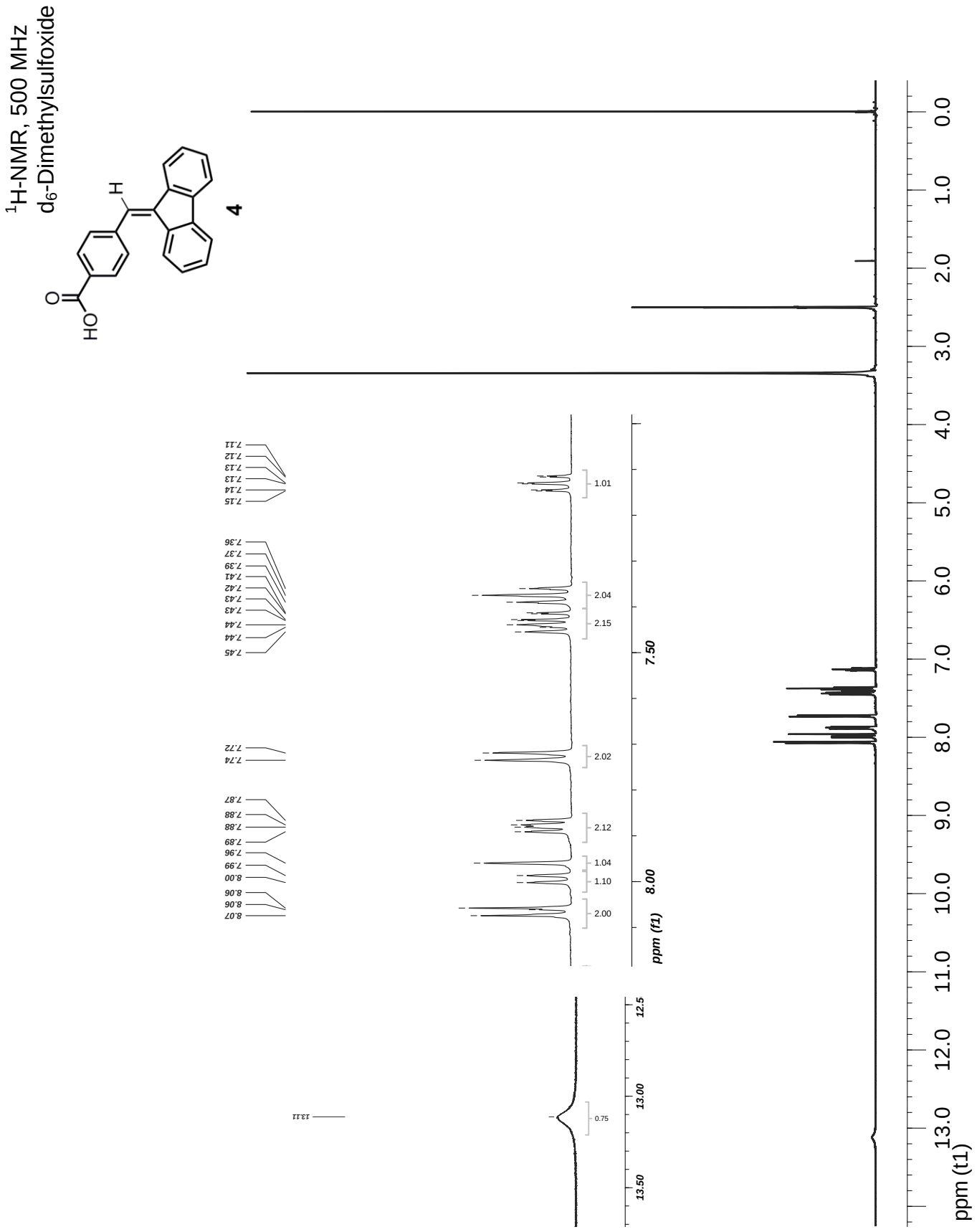


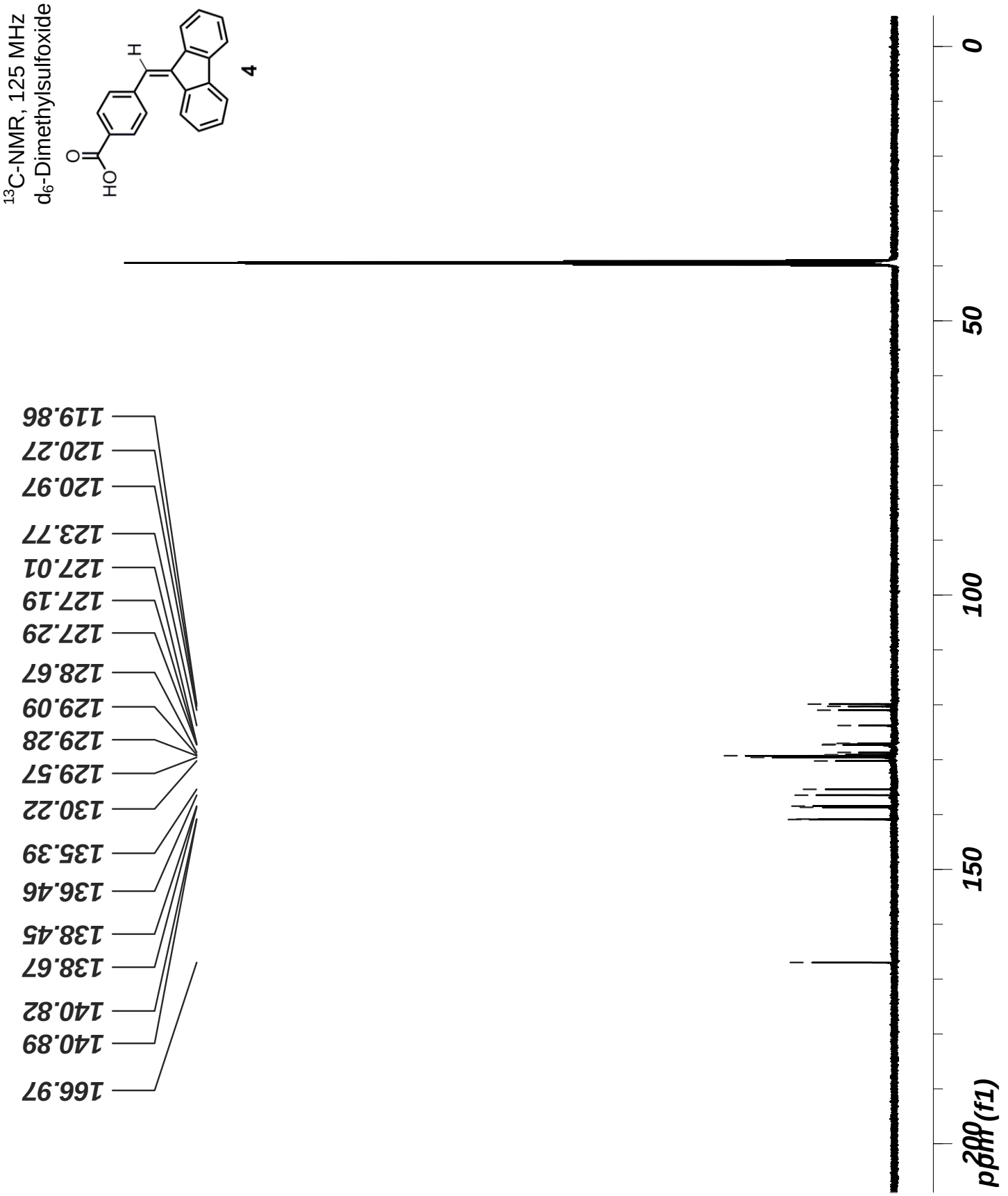
8 **9**
BDPA-TEMPO biradical

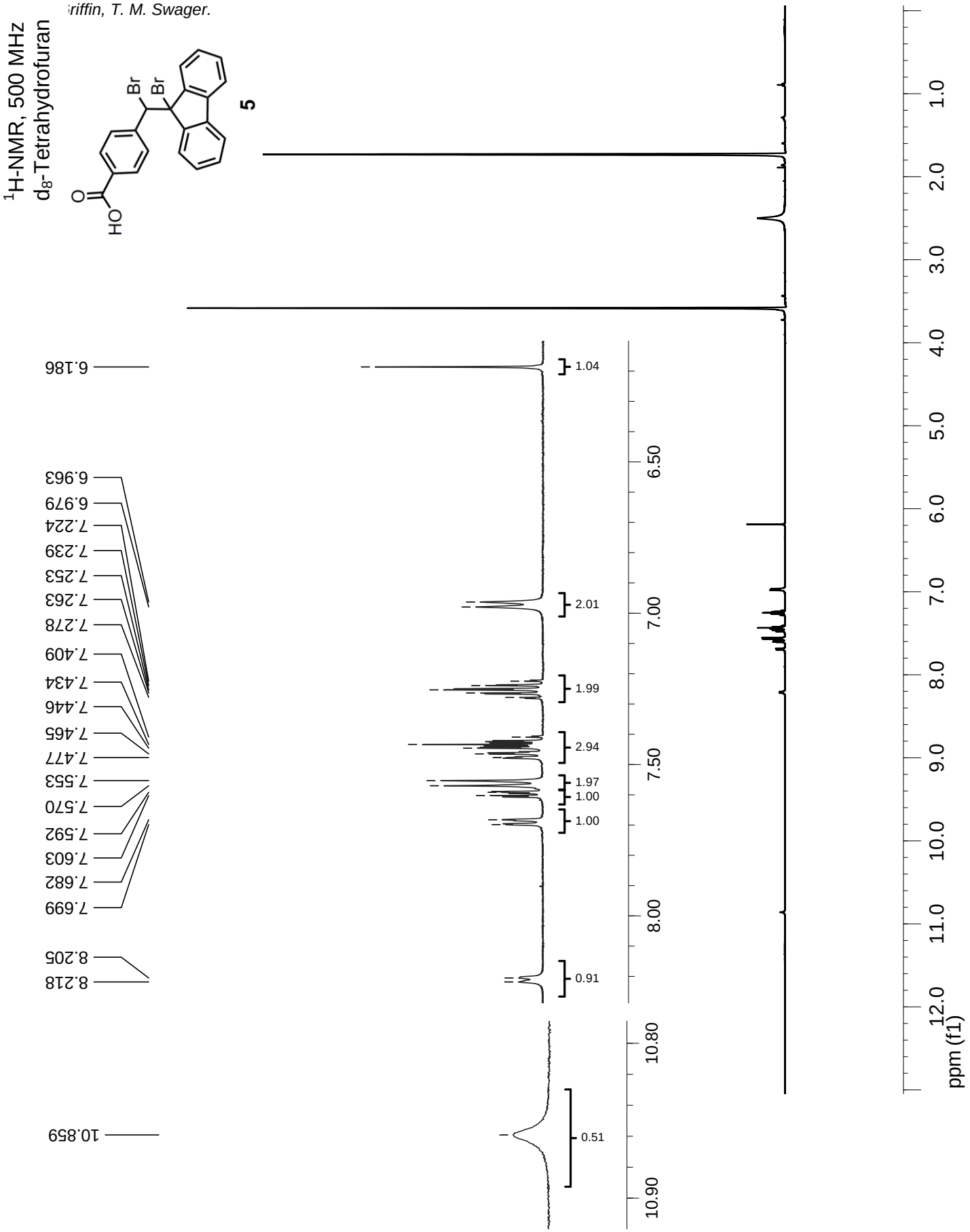
BDPA-TEMPO Biradical (Compound 9): To an over-dried Schlenk flask equipped with a magnetic stir-bar, 0.0800 g (0.130 mmol, 1.00 equiv) of **8** and 0.0440 g (0.390 mmol, 3.00 equiv) of potassium *t*-butoxide were added and the flask was evacuated and refilled with argon three times. To the flask was added 80 mL of a dry 9 to 1 solution of dimethylsulfoxide and *tert*-butyl alcohol. The flask was vigorously stirred for 20 minutes and then 0.0660 g (0.390 mmol, 3.00 equiv) silver nitrate was dissolved in 1.5 mL of water and added to the reaction. After stirring for 1 minute, additional water was added and the solution was extracted with 50 mL of diethyl ether four times for a total volume of 200 mL. The ether was washed once with both water and brine, and then dried over sodium sulfate. The ether solution was filtered through a small plug of silica on a fritted funnel to ensure that any remaining silver particles were removed. Removal of the

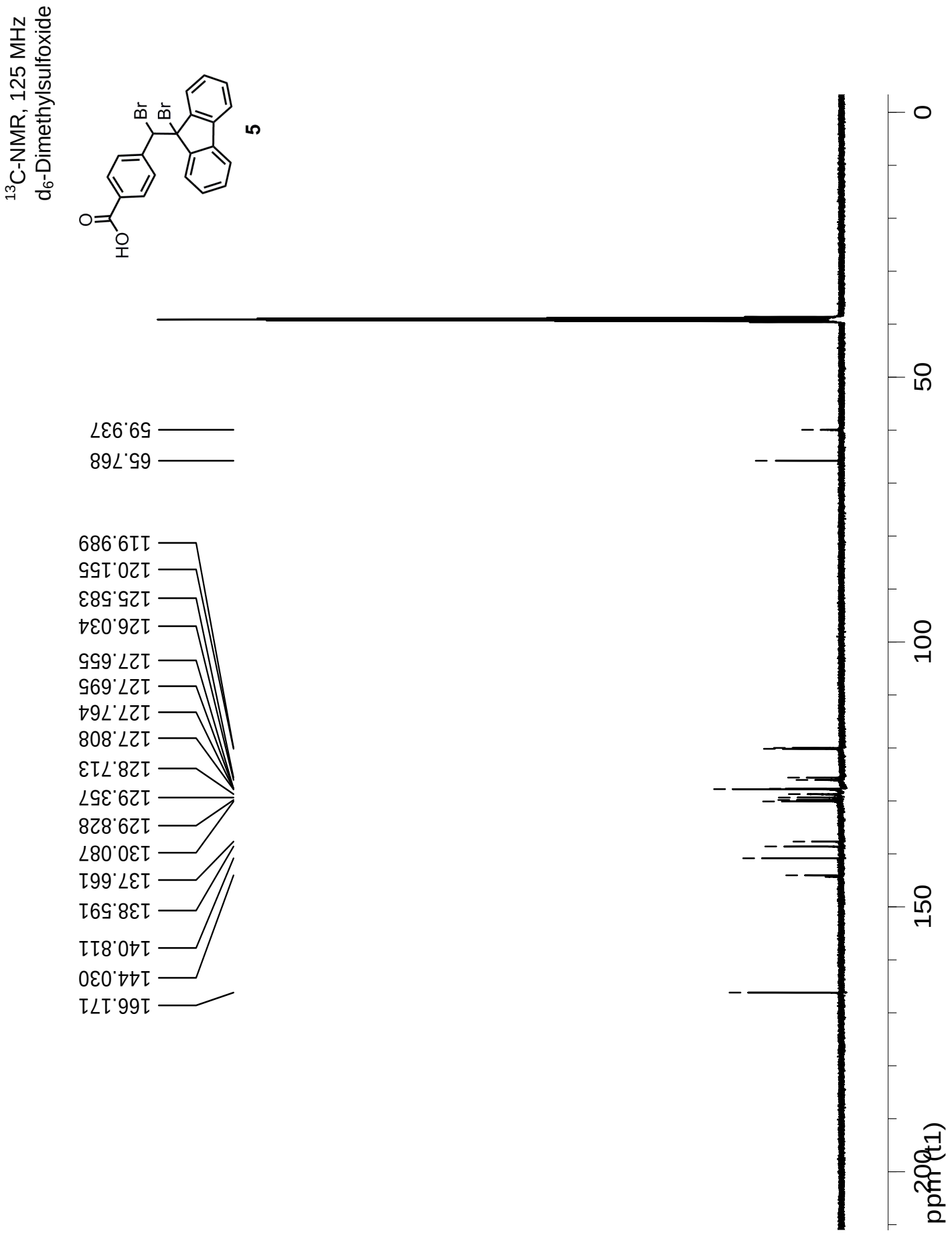
ether yielded 0.0760 g (0.124 mmol, 95 % mass recovery) of a dark brown material with a slight metallic luster. Based on EPR measurements, we estimate the purity of material to be at least 90 %, with the major impurity being the starting material, **8**. Therefore, taking into account mass recovery and purity, we estimate the yield of the reaction to be at least 85 %.

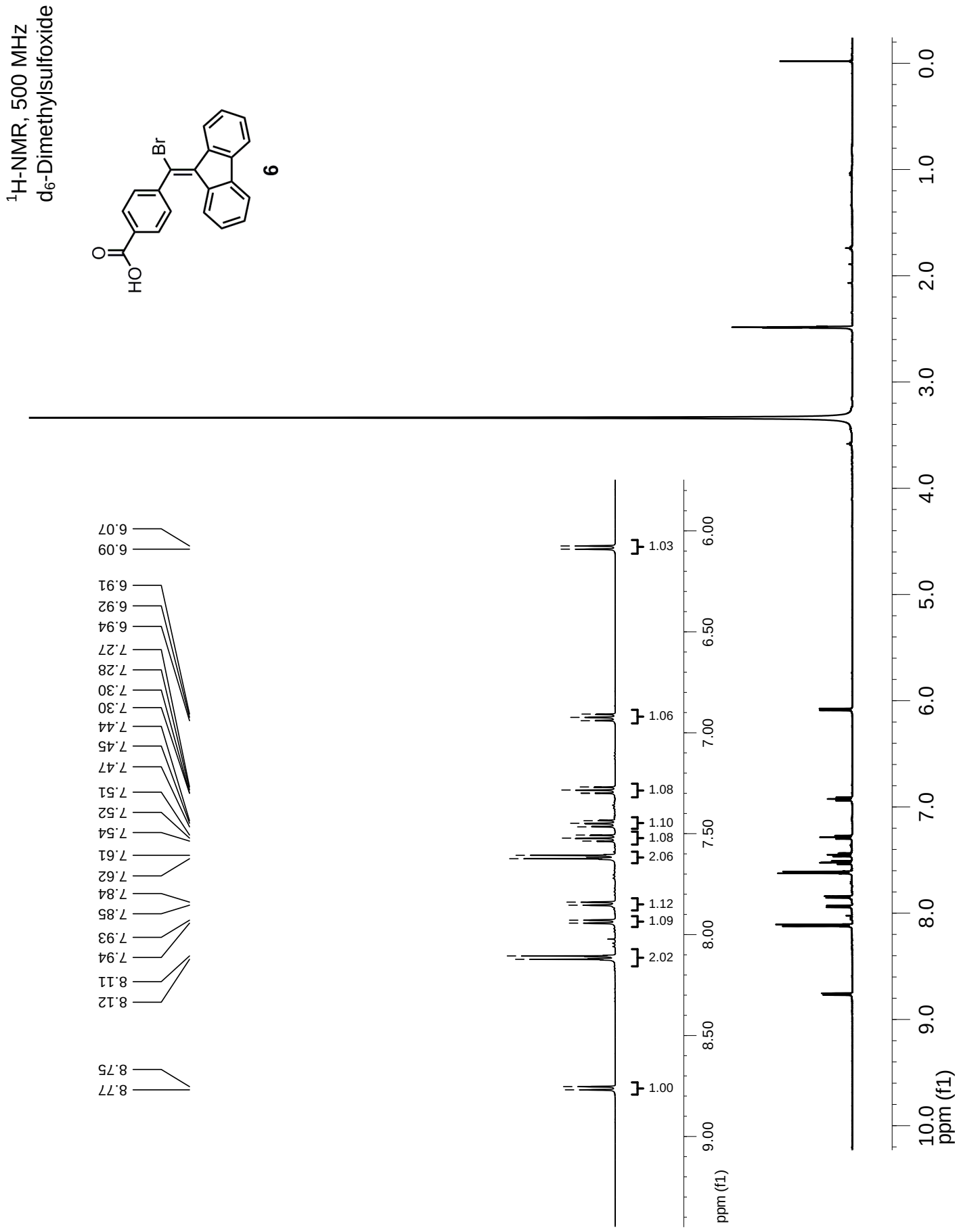
FT-IR: ν_{max}(KBr)/cm^{-1} 1646 s, 1607 m, 1539 s, 1497 m, 1444 s, 1324 m, 1242 m, 731 s.
HRMS (ESI): 614.2938 [calc'd for M-: 614.2939].

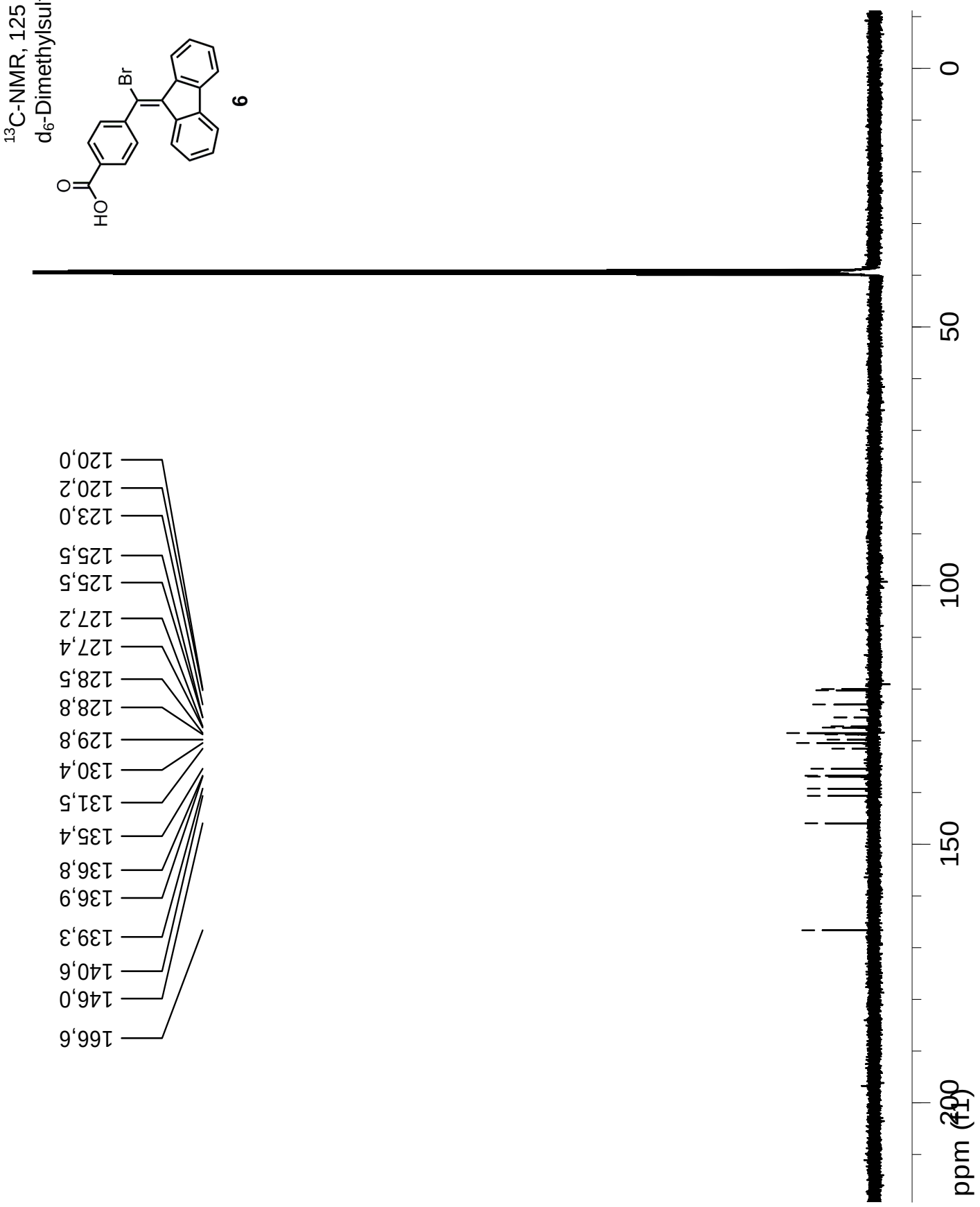

^{1}H-NMR, 500 MHz
d_6-Dimethylsulfoxide
4
HO
O
H
7.11
7.12
7.13
7.13
7.14
7.15
7.36
7.37
7.39
7.41
7.42
7.43
7.43
7.44
7.44
7.45
7.72
7.74
7.87
7.88
7.88
7.89
7.96
7.99
8.00
8.06
8.06
8.07
13.11
1.01
2.04
2.15
2.02
2.12
1.04
1.10
2.00
0.75
7.50
8.00
12.5
13.00
13.50
ppm (f1)
ppm (t1)

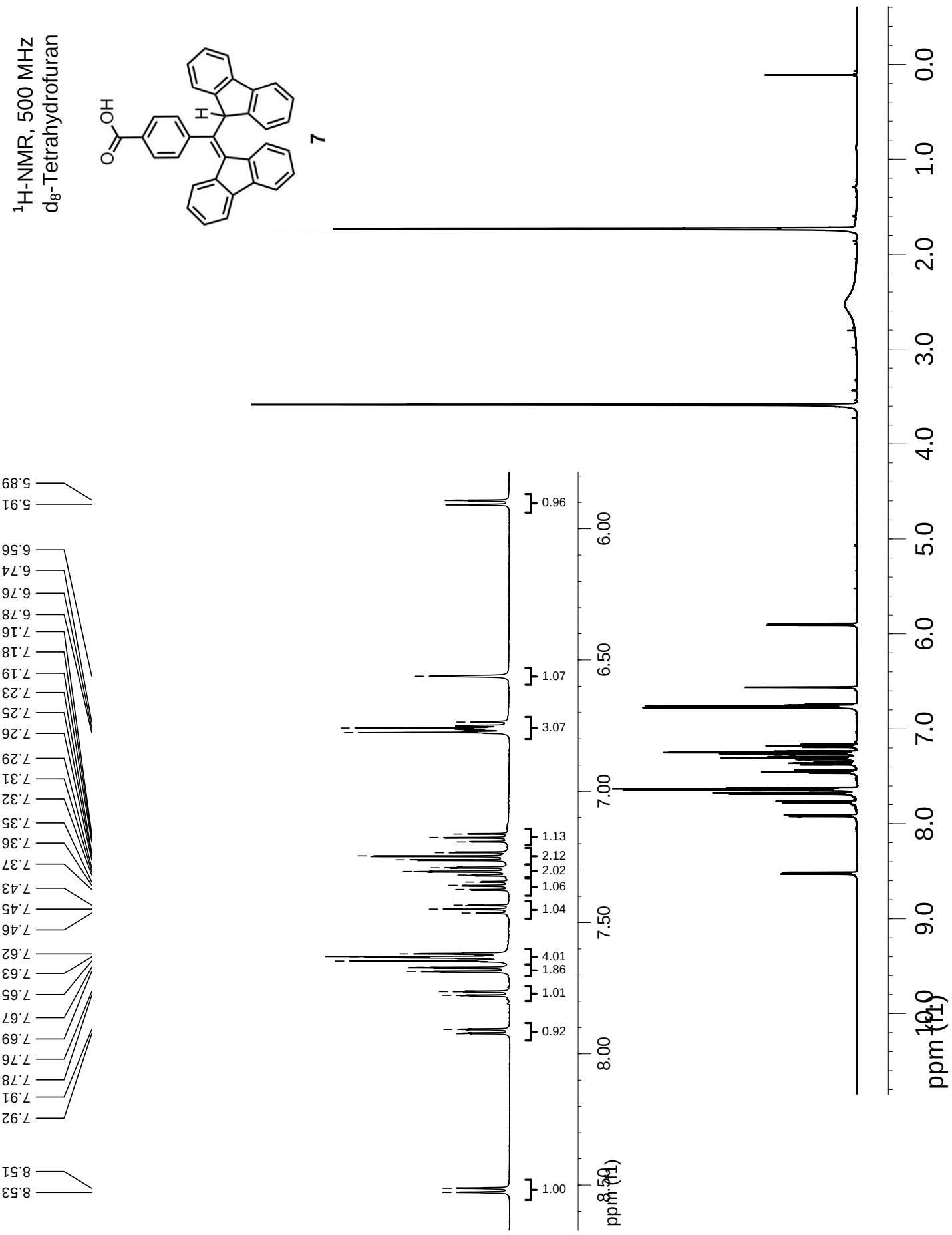

^{13}C-NMR, 125 MHz
d_6-Dimethylsulfoxide
4
166.97
140.89
140.82
138.67
138.45
136.46
135.39
130.22
129.57
129.28
129.09
128.67
127.29
127.19
127.01
123.77
120.97
120.27
119.86
0
50
100
150
200
ppm (f1)

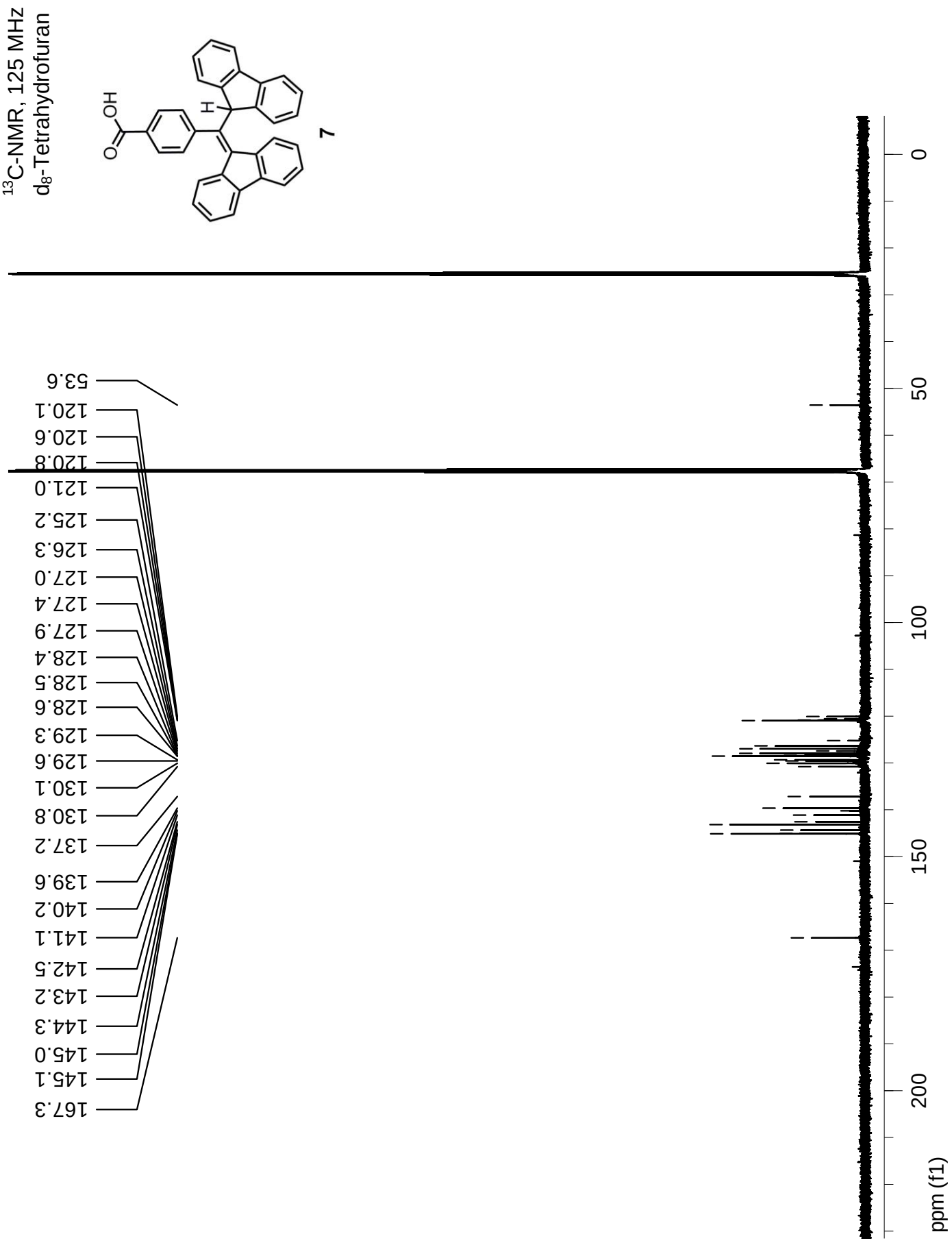

^{1}H-NMR, 500 MHz
d_8-Tetrahydrofuran
Br
Br
O
HO
5
6.186
6.963
6.979
7.224
7.239
7.253
7.263
7.278
7.409
7.434
7.446
7.465
7.477
7.553
7.570
7.592
7.603
7.682
7.699
8.205
8.218
10.859
1.04
2.01
1.99
2.94
1.97
1.00
1.00
0.91
0.51
6.50
7.00
7.50
8.00
10.80
10.90
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
12.0
ppm (f1)


^{13}C-NMR, 125 MHz
d_6-Dimethylsulfoxide
Br
Br
O
HO
5
59.937
65.768
119.989
120.155
125.583
126.034
127.655
127.695
127.764
127.808
128.713
129.357
129.828
130.087
137.661
138.591
140.811
144.030
166.171
0
50
100
150
200
ppm (t1)

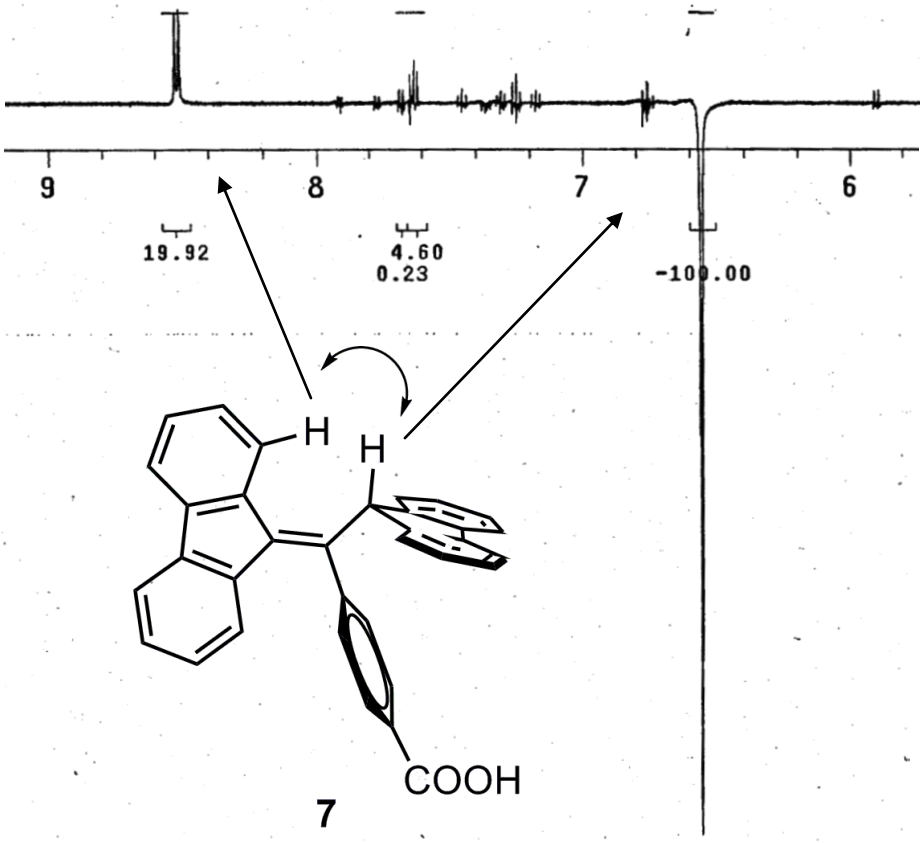

^{1}H-NMR, 500 MHz
d_6-Dimethylsulfoxide
Br
O
HO
6
8.77
8.75
8.12
8.11
7.94
7.93
7.85
7.84
7.62
7.61
7.54
7.52
7.51
7.47
7.45
7.44
7.30
7.30
7.28
7.27
6.94
6.92
6.91
6.09
6.07
1.00
2.02
1.09
1.12
2.06
1.08
1.10
1.08
1.06
1.03
9.00
8.50
8.00
7.50
7.00
6.50
6.00
ppm (f1)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

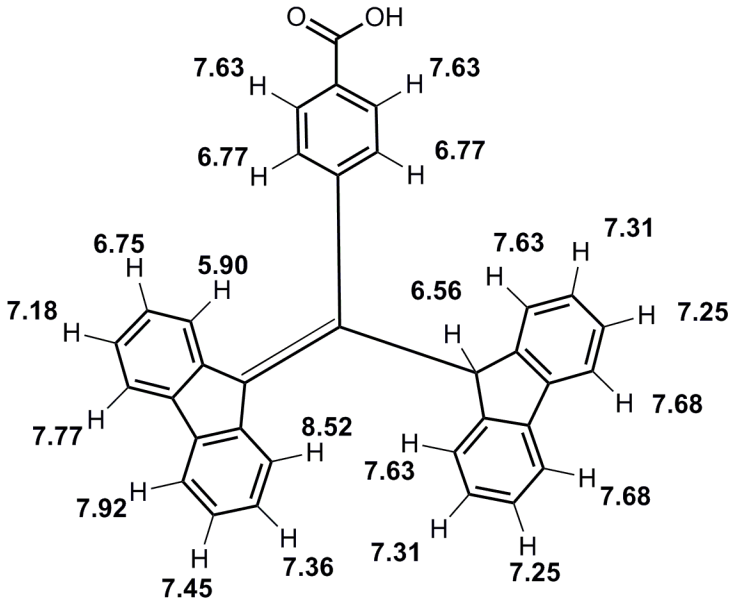

^{13}C-NMR, 125 MHz
d$_6$-Dimethylsulfoxide
Br
O
HO
6
120,0
120,2
123,0
125,5
125,5
127,2
127,4
128,5
128,8
129,8
130,4
131,5
135,4
136,8
136,9
139,3
140,6
146,0
166,6
0
50
100
150
200
ppm (f1)


^{1}H-NMR, 500 MHz
d_8-Tetrahydrofuran
7
ppm (t1)
0.96
1.07
3.07
1.13
2.12
2.02
1.06
1.04
4.01
1.86
1.01
0.92
1.00
5.89
5.91
6.56
6.74
6.76
6.78
7.16
7.18
7.19
7.23
7.25
7.26
7.29
7.31
7.32
7.35
7.36
7.37
7.43
7.45
7.46
7.62
7.63
7.65
7.67
7.69
7.76
7.78
7.91
7.92
8.51
8.53


^{13}C-NMR, 125 MHz
d_8-Tetrahydrofuran
7
53.6
120.1
120.6
120.8
121.0
125.2
126.3
127.0
127.4
127.9
128.4
128.5
128.6
129.3
129.6
130.1
130.8
137.2
139.6
140.2
141.1
142.5
143.2
144.3
145.0
145.1
167.3
0
50
100
150
200
ppm (f1)

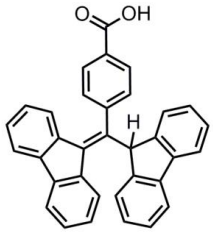
Measurement of NOE enhancement between the proton at 6.56 ppm and the proton at 8.52 ppm (in d_8-tetrahydrofuran, room temperature, 500 MHz).



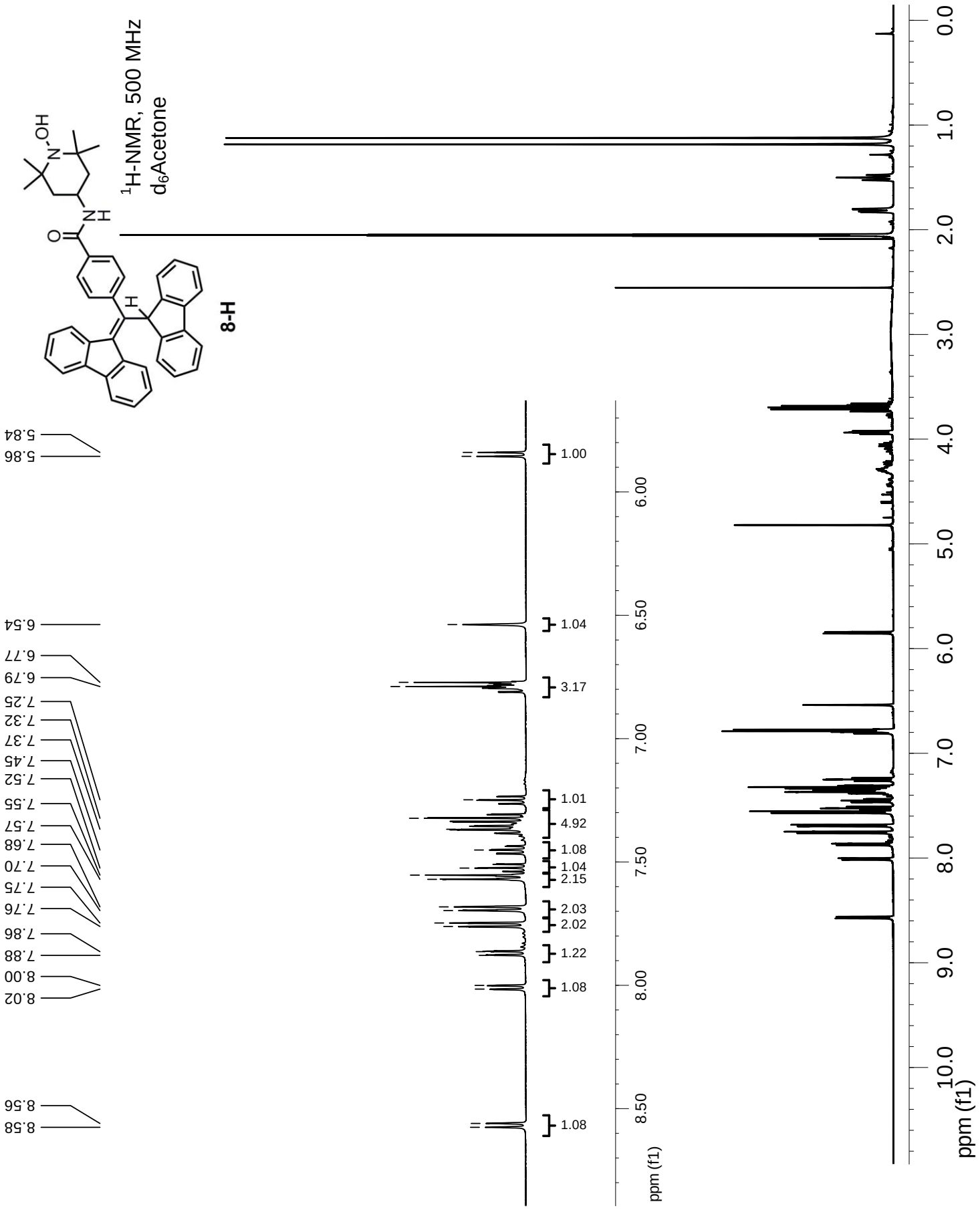


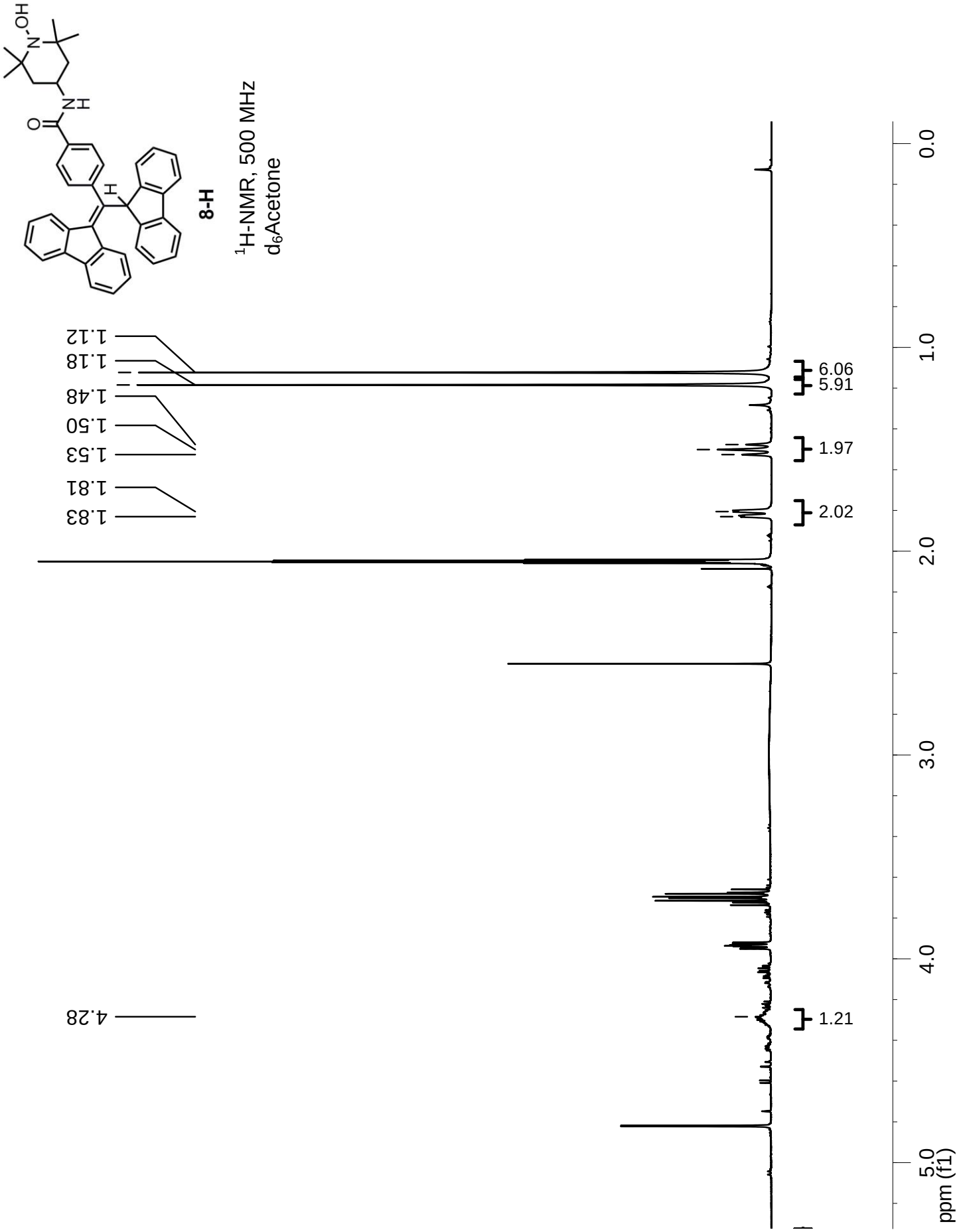
Peak assignment for compound **7** in ppm based on NOE-measurement and gCOSY (next page).

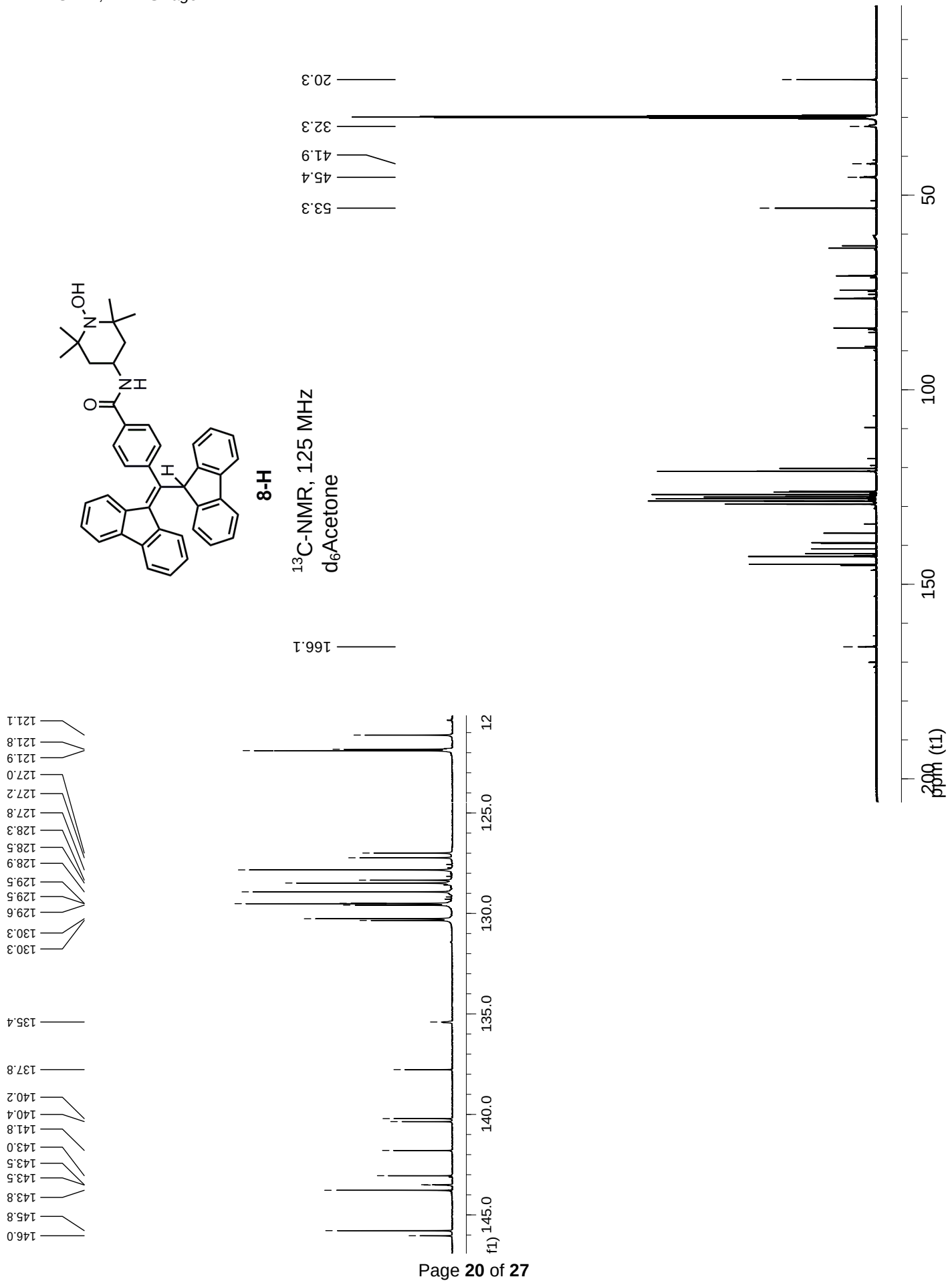
^{1}H-gCOSY-NMR, 500 MHz (see previous page for assignment of peaks).
d_8-Tetrahydrofuran



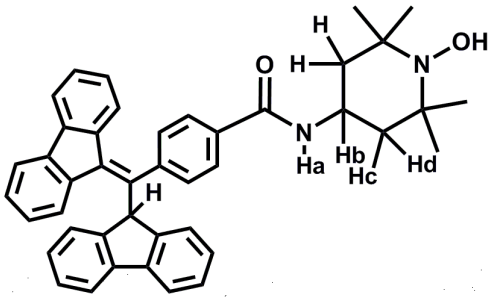
7

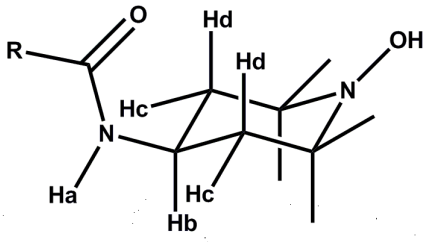

^{1}H-NMR, 500 MHz
d_6Acetone
8-H
5.84
5.86
6.54
6.77
6.79
7.25
7.32
7.37
7.45
7.52
7.55
7.57
7.68
7.70
7.75
7.76
7.86
7.88
8.00
8.02
8.56
8.58
1.00
1.04
3.17
1.01
4.92
1.08
1.04
2.15
2.03
2.02
1.22
1.08
1.08
ppm (f1)


8-H
^{1}H-NMR, 500 MHz
d_6Acetone
1.12
1.18
1.48
1.50
1.53
1.81
1.83
4.28
6.06
5.91
1.97
2.02
1.21
0.0
1.0
2.0
3.0
4.0
5.0
ppm (f1)

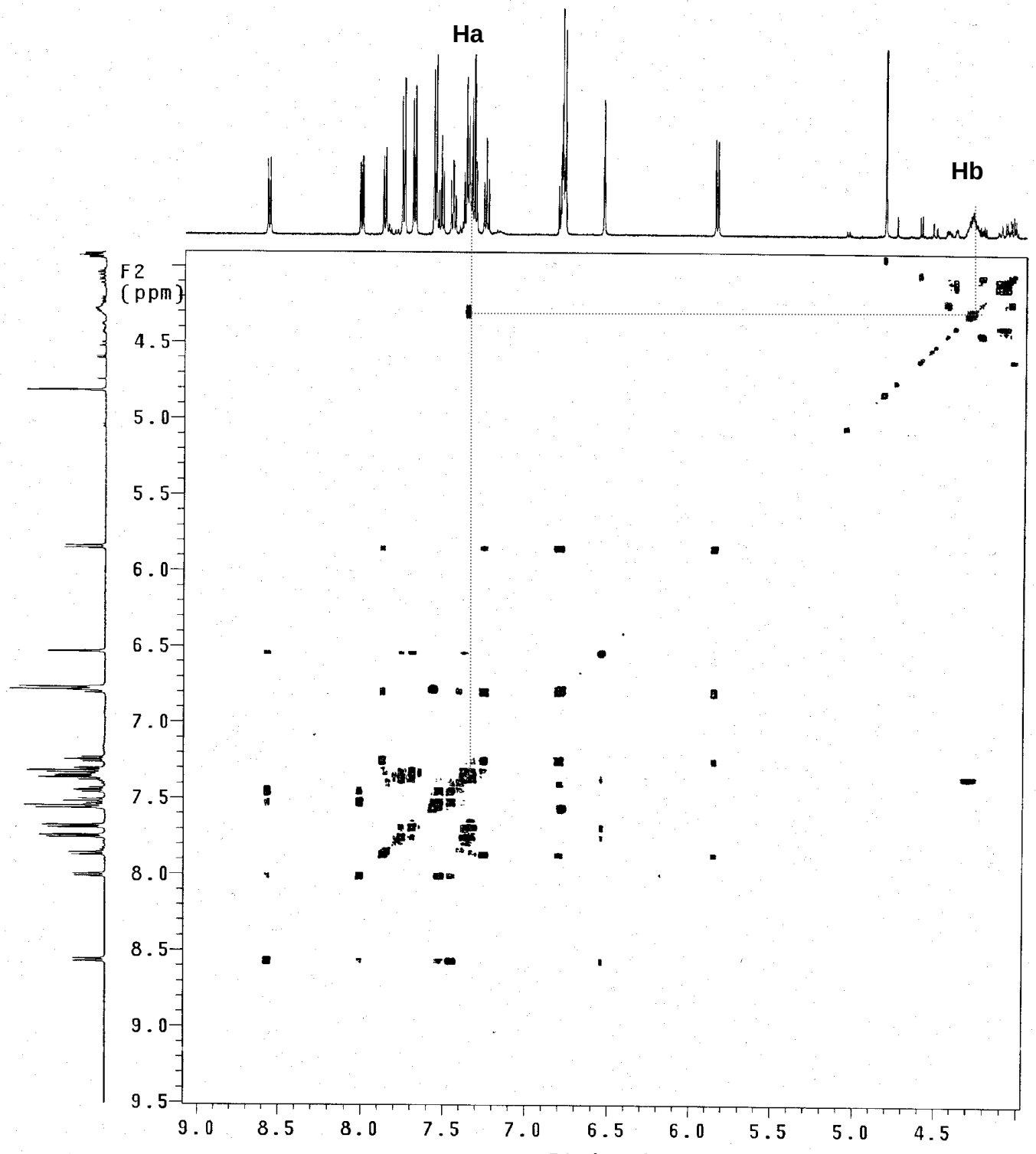

20.3
32.3
41.9
45.4
53.3
166.1
50
100
150
200
ppm (t1)
^{13}C-NMR, 125 MHz
d$_6$Acetone
8-H
121.1
121.8
121.9
127.0
127.2
127.8
128.3
128.5
128.9
129.5
129.5
129.6
130.3
130.3
135.4
137.8
140.2
140.4
141.8
143.0
143.5
143.5
143.8
145.8
146.0
125.0
130.0
135.0
140.0
145.0
f1)

gCOSY NMR, 500 MHz
d_6-Acetone

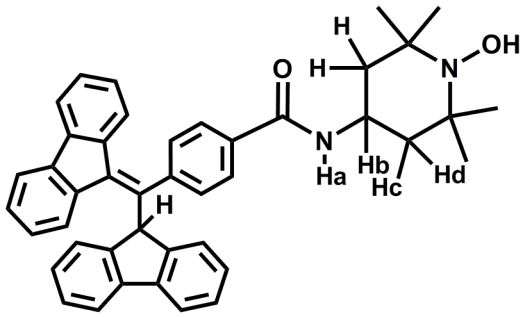




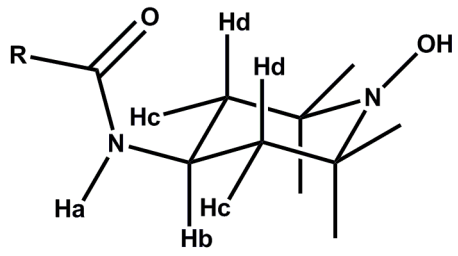
8



gCOSY NMR, 500 MHz
d_6Acetone



8



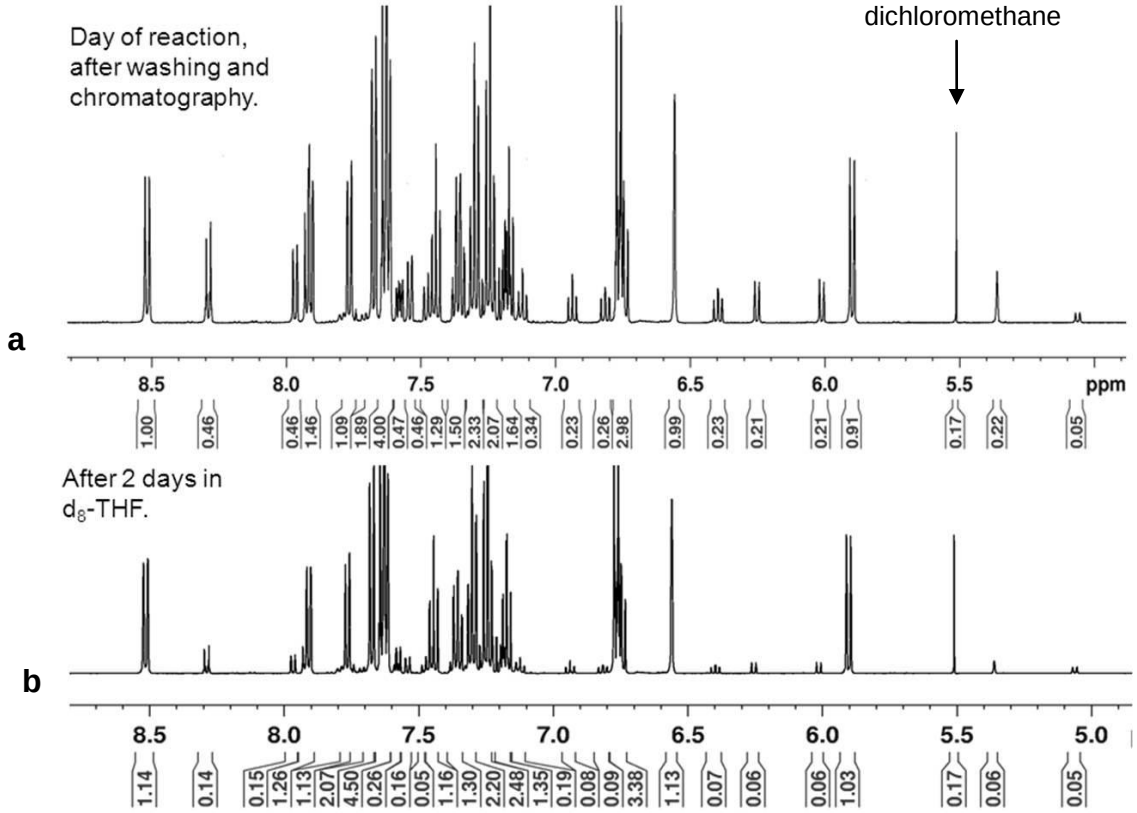
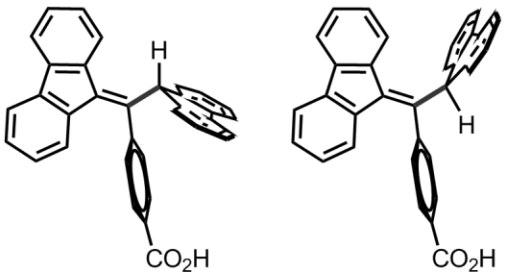


Figure 1. Spectrum **a** shows the aromatic portion of the proton NMR of compound **7** after chromatography but before recrystallization. Both rotamers are present in a ratio of 83:17 (**7a**:**7b**) based on integration of the signals. After being left in the NMR-tube, which was stored at or below room temperature, for two days, the proportion of **7a** increases with a concurrent decrease in **7b**, using the integration of both the DCM signal and an impurity present at slightly above 5.00 ppm as standards. In **b**, the rotamers are in a ratio of 95:5 (**7a**:**7b**). After a week under the same conditions, **7b** can no longer be detected.



7a **7b**

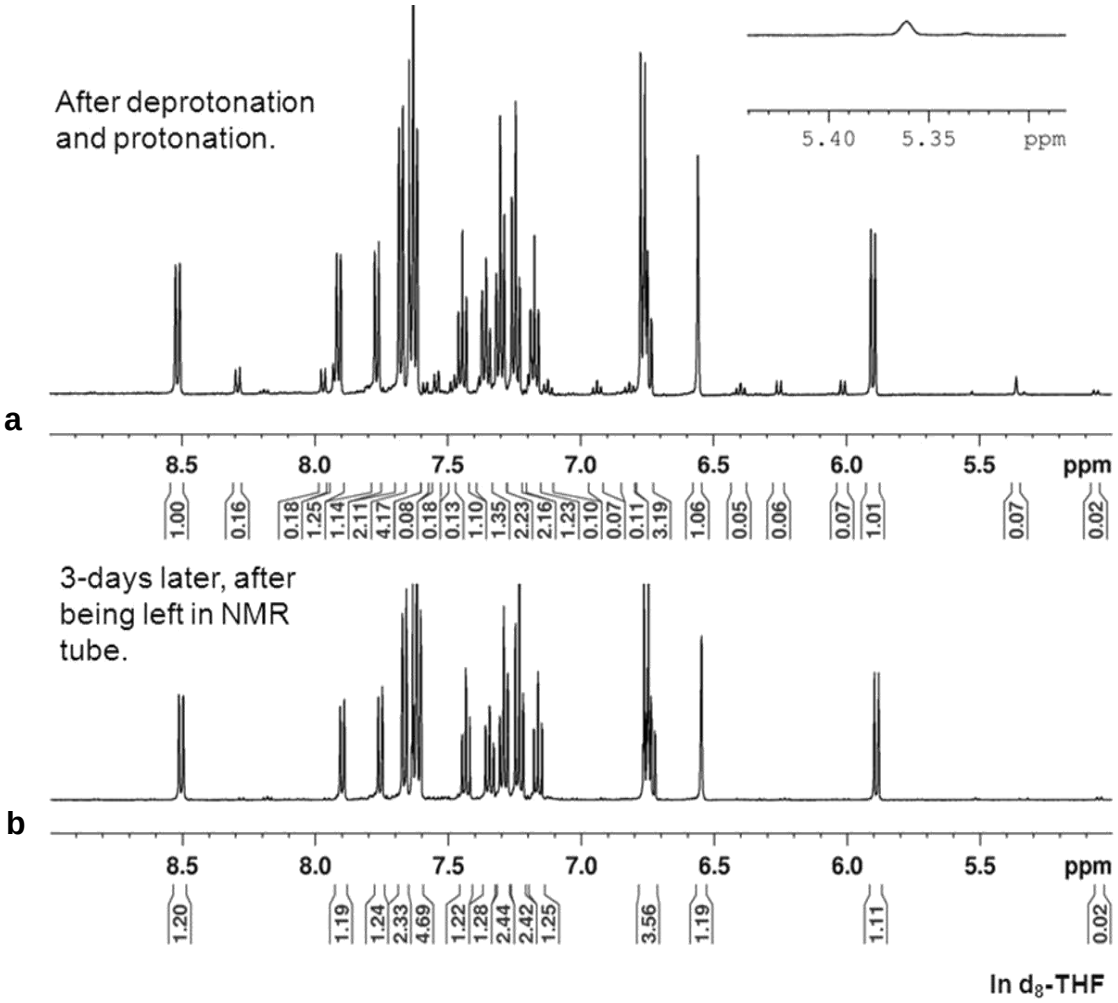
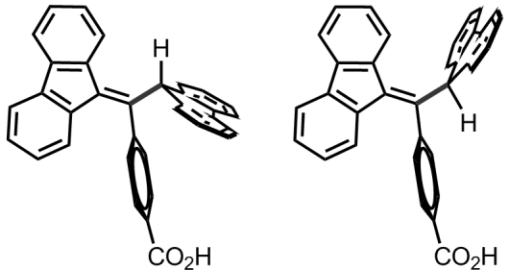
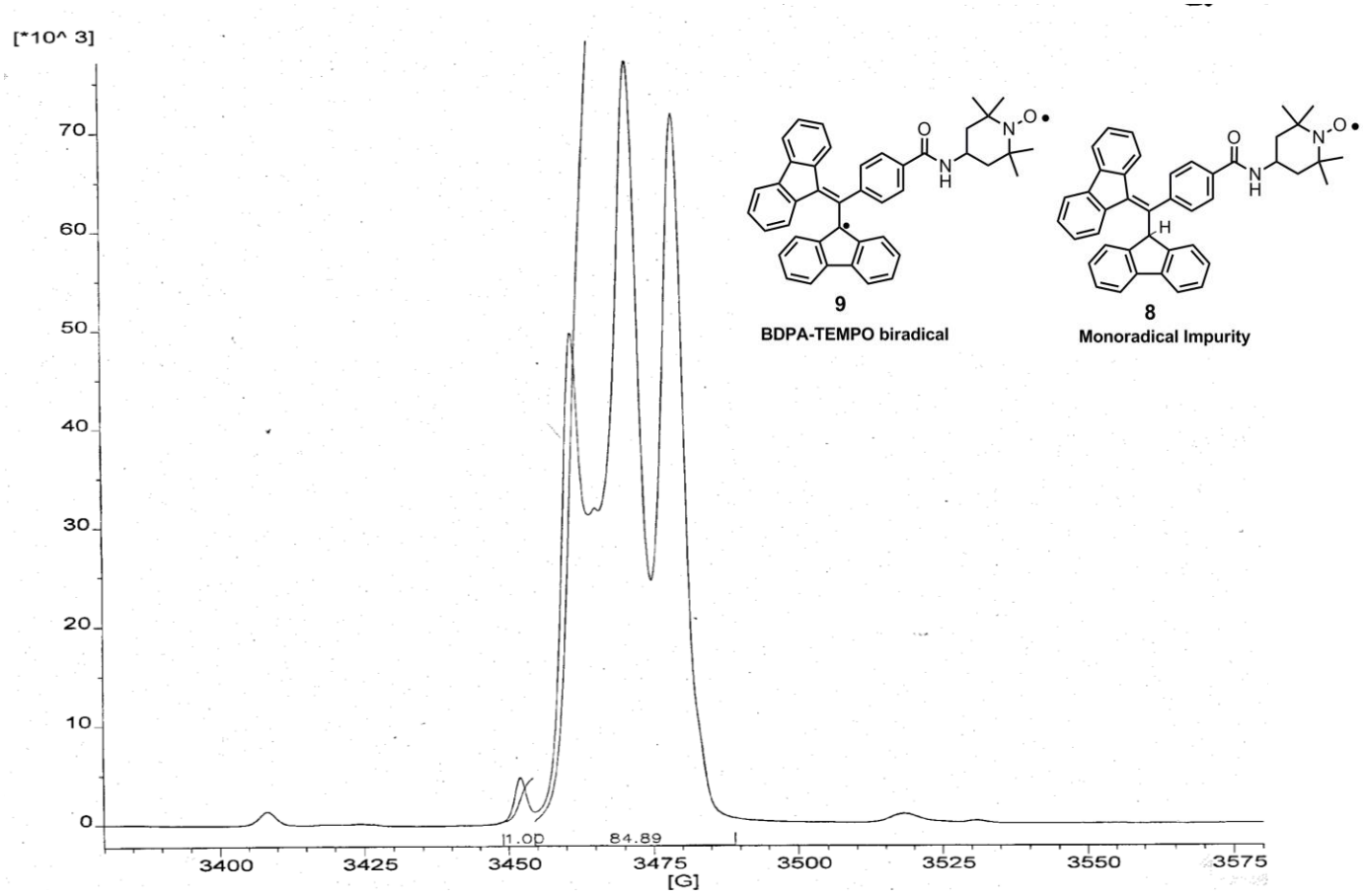


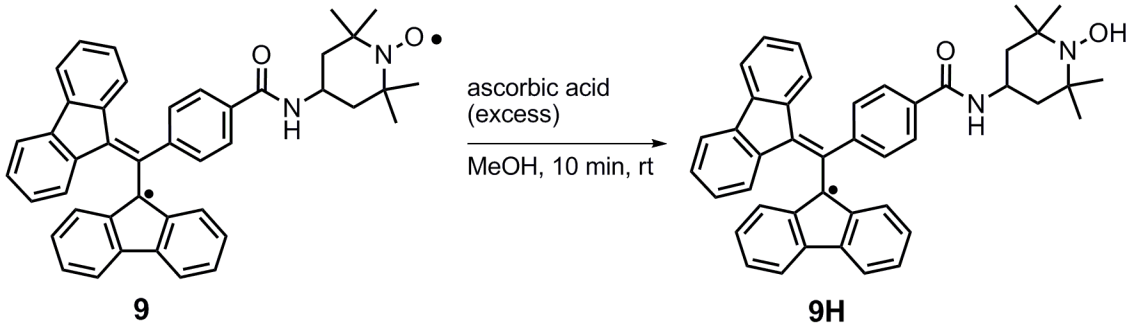
Figure 2. Spectrum **a** shows the aromatic portion of the proton NMR of compound **7** after the pure compound, displaying only the major rotamer in proton-NMR, was deprotonated in DMA with sodium *t*-butoxide and quenched with acid. As can be seen, the minor rotamer has reappeared. Spectrum **b** shows that after three days the minor rotamer is no longer visible.



7a **7b**



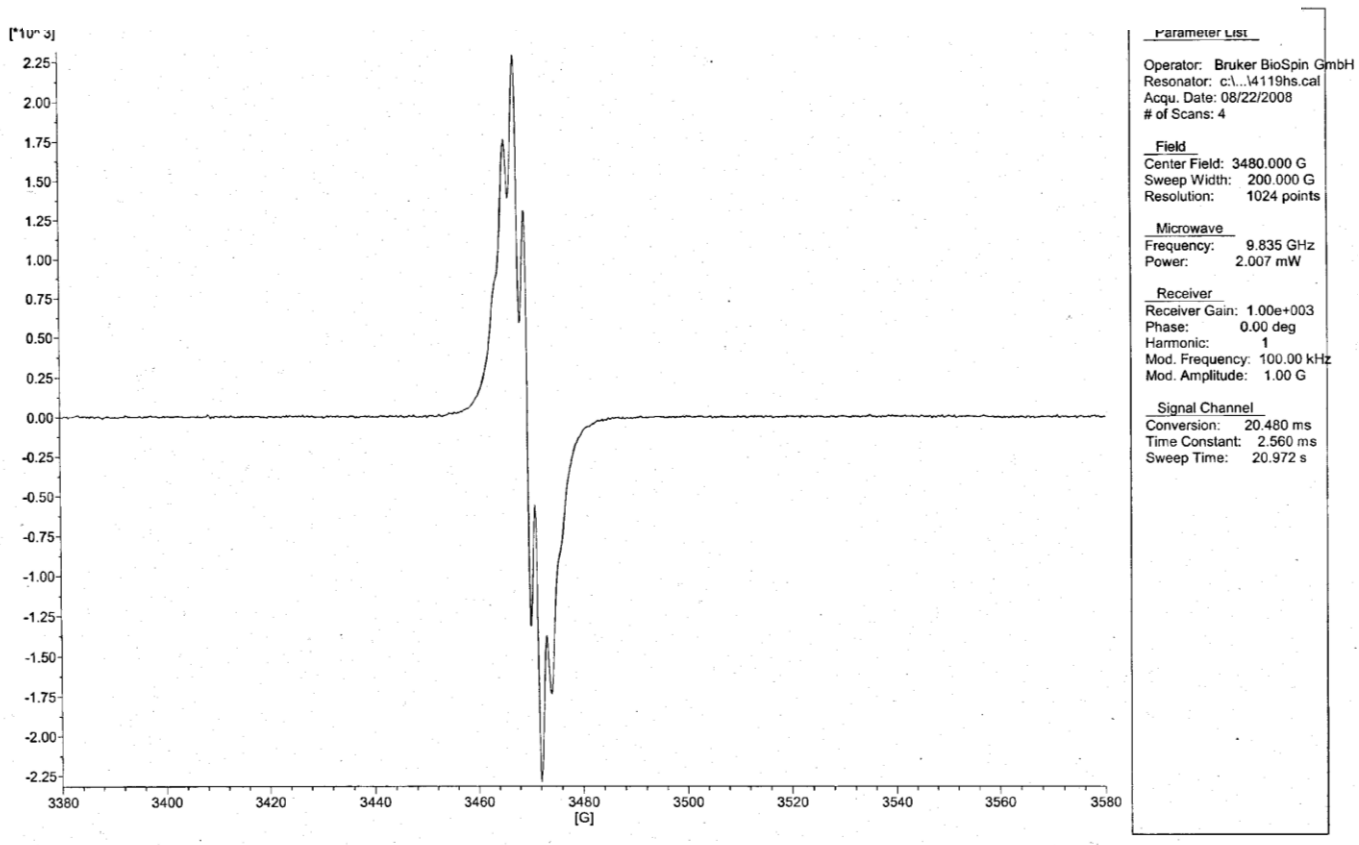
9 GHz EPR of **9** is shown above. The peak between 3450-3455 G, which is attributed to the monoradical impurity, is integrated and given the arbitrary value 1.00. The rest of the middle portion, which contains most of the biradical signal and two-thirds of the impurity signal, integrates to 84.89. The smaller integral represents one-third of the impurity's signal because it represents one of the three peaks of the triplet. Therefore, the portion of the larger intergral that is due to the biradical is 82.89 (84.89 – 2.00). Each biradical has two spins and therefore gives twice the signal compared to the monoradical impurity. Therefore, to compare on a molar basis the biradical's signal must be divided by 2, giving 41.45. The percentage of monoradical impurity is therefore (3.00/(3.00 + 41.45)) x 100 = 6.7 % and percentage of the biradical is (41.45/(3.00 + 41.45)) x 100 = 93.3 % based on integration of the EPR signal. Experimental parameters: rt, microwave power 2 mW, sweep width 20 mT, modulation amplitude 10 mT.

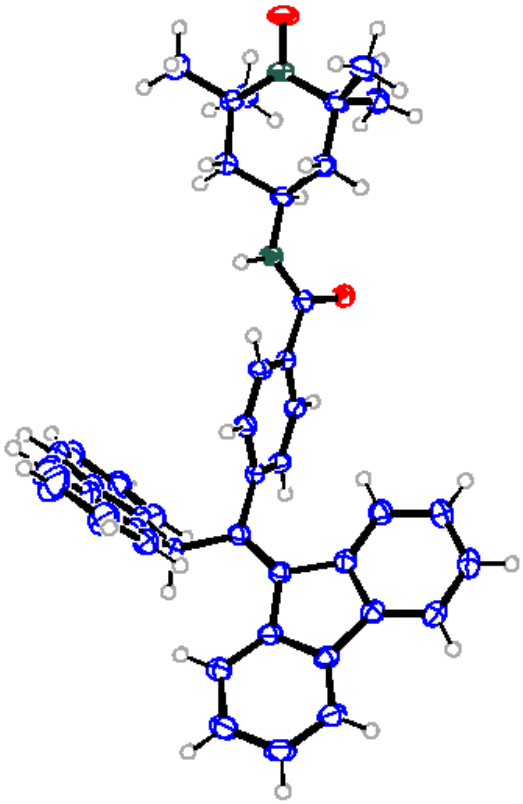


9 **9H**

BDPA-TEMPO biradical

Below is the 9 GHz solution EPR spectrum of **9H** , which is formed when the BDPA-TEMPO biradical is treated with excess ascorbic acid. Ascorbic acid reduces TEMPO, but does not reduce BDPA. The spectrum shown matches that of BDPA, a narrow linewidth with hyperfine coupling to protons on the fluorene rings.





X-ray ORTEP representation of **8** drawn with displacement ellipsoids to the 50% probability level.