Copper-catalyzed C-3 benzylation of quinoxalin-2(1*H*)-ones with benzylsulfonyl hydrazides

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Supporting information

Table of contents

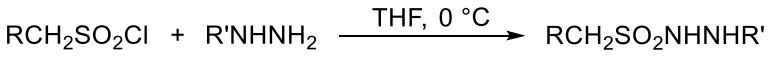
1. General information

^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on a Bruker AC-400 FT spectrometer (400 MHz, 100 MHz, 376 MHz) or on a Bruker AC-500 FT spectrometer (500 MHz, 125 MHz, 471 MHz). The chemical shifts of ^{1}H NMR and ^{13}C NMR spectra were referenced internally with tetramethylsilane (δ H 0.00), $CDCl_3$ (δ C 77.2), and $(CD_3)_2SO$ (δ H 2.50, δ C 39.5). The chemical shifts of ^{19}F NMR spectra were referenced to external $CFCl_3$ (δ F 0.00). Chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz, respectively. The following abbreviations are used in reporting NMR data: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. High resolution mass spectra (HRMS) were recorded on a LC-TOF spectrometer (Micromass). Electrospray ionization (ESI) mass spectrometry data were acquired using a Thermo LTQ Orbitrap XL instrument equipped with an ESI source and controlled by Xcalibur software. Melting points are uncorrected.

Benzylsulfonyl hydrazides **1**[1] and quinoxalin-2(1*H*)-ones **2**[2] were prepared according to literature procedures. The rest of chemicals were purchased from the Sinopharm Chemical Reagent Co., Meryer, Acros, Alfa Aesar, Adamas, and TCI, Energy Chemical, Leyan, Bidepharm, and used as received.

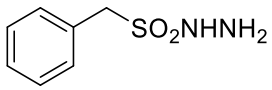
Abbreviations: Boc = *tert*-butoxycarbonyl, Cbz = benzyloxycarbonyl, DCE = 1,2-dichloroethane, DCP = dicumyl peroxide, DMF = *N*,*N*-dimethylformamide, DMSO = dimethyl sulfoxide, DTBP = di-*tert*-butyl peroxide, TBHP = *tert*-butyl hydroperoxide, TBPB = *tert*-butyl peroxybenzoate, TEMPO = 2,2,6,6-tetramethyl-1-piperidinyloxy, THF = tetrahydrofuran.

2. Preparation of sulfonyl hydrazides[1c]



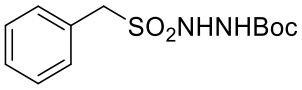
2

Hydrazine monohydrate (626 mg, 12.5 mmol) (or a monosubstituted hydrazine, 5.0 mmol) was added dropwise to a solution of the sulfonyl chloride[1a,b] (5.0 mmol) in tetrahydrofuran (30 mL) under nitrogen at 0 °C. During the addition the reaction solution became cloudy and a white precipitate of hydrazine hydrochloride was deposited. The mixture was stirred at 0 °C for 30 min, added ethyl acetate (50 mL), and washed with saturated brine (3 × 30 mL). The organic layer was dried over anhydrous sodium sulfate, filtered, and added slowly to stirred hexane (30 mL) over 5 min. After being stirred for 10 min, the mixture was filtered, and the collected solid was dried in vacuum.



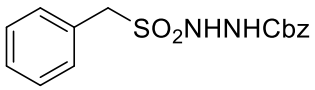
2a

Phenylmethanesulfonohydrazide (**2a**)[3] was obtained (680 mg, 73% yield) as a white solid. m.p. 125-127 °C. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.91 (br, 1H), 7.38-7.33 (m, 5H), 4.46 (br, 2H), 4.37 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 130.9, 130.1, 128.3, 127.9, 53.1. HRMS (ESI) calcd for $C_7H_{11}N_2O_2S^+$ $(M + H)^+$ 187.0536, found 187.0535.



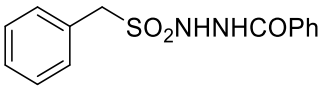
2ab

tert-Butyl 2-(benzylsulfonyl)hydrazine-1-carboxylate (**2ab**) was obtained (923 mg, 64% yield) as a white solid. m.p. 118-120 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 9.28 (br, 1H), 9.22 (br, 1H), 7.45 (d, J = 7.5 Hz, 2H), 7.39-7.33 (m, 3H), 4.33 (s, 2H), 1.44 (s, 9H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 155.6, 131.0, 129.6, 128.3, 128.1, 79.9, 57.3, 28.1. HRMS (ESI) calcd for $C_{12}H_{19}N_2O_4S^+$ $(M + H)^+$ 287.1060, found 287.1058.



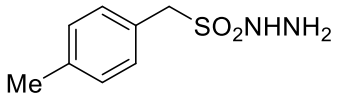
2ac

Benzyl 2-(benzylsulfonyl)hydrazine-1-carboxylate (**2ac**) was obtained (830 mg, 52% yield) as a white solid. m.p. 116-118 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 9.74 (br, 1H), 9.43 (br, 1H), 7.46-7.37 (m, 10H), 5.16 (s, 2H), 4.38 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 156.5, 136.4, 131.1, 129.5, 128.5, 128.3, 128.2, 128.0, 66.4, 57.3. HRMS (ESI) calcd for $C_{15}H_{17}N_2O_4S^+$ $(M + H)^+$ 321.0904, found 321.0903.



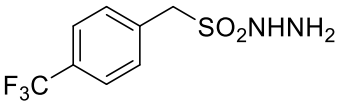
2ad

N'-Benzoyl-1-phenylmethanesulfonohydrazide (**2ad**) was obtained (564 mg, 39% yield) as a white solid. m.p. 173-175 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.75 (br, 1H), 9.68 (br, 1H), 7.93 (d, J = 7.5 Hz, 2H), 7.61 (t, J = 7.0 Hz, 1H), 7.54-7.49 (m, 4H), 7.38-7.35 (m, 3H), 4.45 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 166.4, 132.2, 132.0, 131.1, 129.5, 128.5, 128.3, 128.1, 127.7, 58.3. HRMS (ESI) calcd for $C_{14}H_{15}N_2O_3S^+$ $(M + H)^+$ 291.0798, found 291.0797.



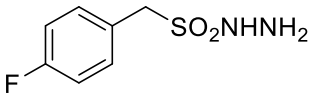
2b

p-Tolylmethanesulfonohydrazide (**2b**) was obtained (625 mg, 62% yield) as a white solid. m.p. 143-145 ℃. ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.85 (br, 1H), 7.27 (d, J = 7.8 Hz, 2H), 7.18 (d, J = 7.8 Hz, 2H), 4.43 (br, 2H), 4.32 (s, 2H), 2.31 (s, 3H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 137.2, 130.7, 128.9, 127.0, 52.8, 20.8. HRMS (ESI) calcd for $C_8H_{13}N_2O_2S^+$ $(M + H)^+$ 201.0692, found 201.0689.



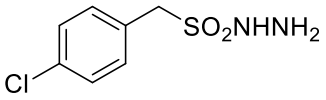
2c

(4-(Trifluoromethyl)phenyl)methanesulfonohydrazide (**2c**)[4] was obtained (831 mg, 65% yield) as a white solid. m.p. 118-120 ℃. ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.99 (br, 1H), 7.74 (d, J = 8.0 Hz, 2H), 7.61 (d, J = 8.0 Hz, 2H), 4.51 (br, 2H; s, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 135.0, 131.7, 128.6 (q, J = 31.6 Hz), 125.1 (q, J = 3.7 Hz), 124.3 (q, J = 270.5 Hz), 52.7. ^{19}F NMR (376 MHz, DMSO-d_6) δ -56.26. HRMS (ESI) calcd for $C_8H_{10}N_2O_2F_3S^+$ $(M + H)^+$ 255.0410, found 255.0409.



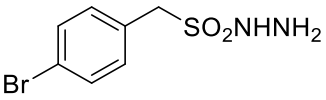
2d

(4-Fluorophenyl)methanesulfonohydrazide (**2d**) was obtained (840 mg, 82% yield) as a white solid. m.p. 133-136 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.89 (br, 1H), 7.42 (t, J = 6.8 Hz, 2H), 7.20 (t, J = 8.3 Hz, 2H), 4.46 (br, 2H), 4.38 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 162.0 (d, J = 242.6 Hz), 132.9 (d, J = 8.3 Hz), 126.5 (d, J = 2.9 Hz), 115.2 (d, J = 21.4 Hz), 52.1. ^{19}F NMR (471 MHz, DMSO-d_6) δ -114.44. HRMS (ESI) calcd for $C_7H_{10}FN_2O_2S^+$ $(M + H)^+$ 205.0442, found 205.0443.



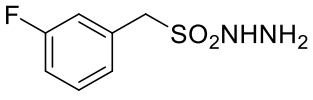
2e

(4-Chlorophenyl)methanesulfonohydrazide (**2e**)[5] was obtained (782 mg, 71% yield) as a white solid. m.p. 127-130 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.93 (br, 1H), 7.44 (d, J = 8.5 Hz, 2H), 7.40 (d, J = 8.5 Hz, 2H), 4.48 (br, 2H), 4.39 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 132.8, 132.7, 129.3, 128.3, 52.2. HRMS (ESI) calcd for $C_7H_{10}ClN_2O_2S^+$ $(M + H)^+$ 221.0146, found 221.0144.



2f

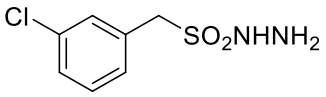
(4-Bromophenyl)methanesulfonohydrazide (**2f**)[5] was obtained (1.15 g, 87% yield) as a white solid. m.p. 136-139 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.93 (br, 1H), 7.57 (d, J = 8.0 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 4.47 (br, 2H), 4.38 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 133.0, 131.3, 129.7, 121.4, 52.3. HRMS (ESI) calcd for $C_7H_{10}BrN_2O_2S^+$ $(M + H)^+$ 264.9641, found 264.9639.



2g

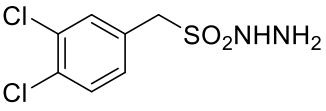
(3-Fluorophenyl)methanesulfonohydrazide (**2g**) was obtained (693 mg, 68% yield) as a white solid. m.p. 108-111 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.96 (br, 1H), 7.44-7.39 (m, 1H), 7.24-7.16 (m, 3H), 4.49 (br, 2H), 4.42 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 161.9 (J = 241.6 Hz), 132.8 (J = 8.1 Hz), 130.2 (J = 8.3 Hz), 127.1 (J = 2.8 Hz), 117.5 (J = 21.8 Hz), 114.8 (J = 20.8 Hz), 52.4. ^{19}F

NMR (471 MHz, DMSO-d_6) δ -113.62. HRMS (ESI) calcd for $C_7H_{10}FN_2O_2S^+$ $(M + H)^+$ 205.0442, found 205.0440.



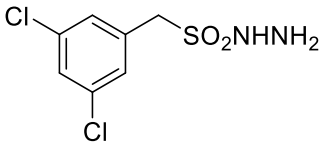
2h

(3-Chlorophenyl)methanesulfonohydrazide (**2h**) was obtained (790 mg, 72% yield) as a white solid. m.p. 117-120 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.99 (br, 1H), 7.46 (s, 1H), 7.41-7.36 (m, 3H), 4.50 (br, 2H), 4.42 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 132.9, 132.7, 130.5, 130.2, 129.7, 127.9, 52.3. HRMS (ESI) calcd for $C_7H_{10}ClN_2O_2S^+$ $(M + H)^+$ 221.0146, found 221.0143.



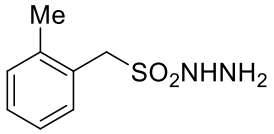
2i

(3,4-Dichlorophenyl)methanesulfonohydrazide (**2i**) was obtained (631 mg, 49% yield) as a white solid. m.p. 88-90 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.98 (br, 1H), 7.65-7.63 (m, 2H), 7.39 (dd, J = 8.5, 2.0 Hz, 1H), 4.49 (br, 2H), 4.43(s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 132.6, 131.4, 131.2, 130.8, 130.4, 51.6. HRMS (ESI) calcd for $C_7H_9Cl_2N_2O_2S^+$ $(M + H)^+$ 254.9756, found 254.9752.



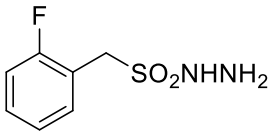
2j

(3,5-Dichlorophenyl)methanesulfonohydrazide (**2j**) was obtained (955 mg, 75% yield) as a white solid. m.p. 117-119 ℃. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.05 (br, 1H), 7.57 (t, J = 1.6 Hz, 1H), 7.46 (d, J = 1.6 Hz, 2H), 4.52 (br, 2H), 4.45 (s, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 134.4, 133.8, 129.5, 127.6, 51.7. HRMS (ESI) calcd for $C_7H_9Cl_2N_2O_2S^+$ $(M + H)^+$ 254.9756, found 254.9754.



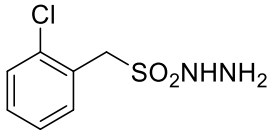
2k

o-Tolylmethanesulfonohydrazide (**2k**) was obtained (780 mg, 78% yield) as a white solid. m.p. 91-94 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.97 (br, 1H), 7.30 (d, J = 7.0 Hz, 1H), 7.26-7.18 (m, 3H), 4.50 (br, 2H), 4.41 (s, 2H), 2.38 (s, 3H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 138.2, 131.9, 130.3, 128.4, 128.1, 125.9, 50.9, 19.3. HRMS (ESI) calcd for $C_8H_{13}N_2O_2S^+$ $(M + H)^+$ 201.0692, found 201.0691.



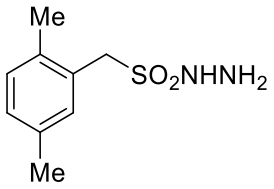
2l

(2-Fluorophenyl)methanesulfonohydrazide (**2l**)[4] was obtained (824 mg, 81% yield) as a white solid. m.p. 90-93 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.08 (br, 1H), 7.48 (t, J = 7.3 Hz, 1H), 7.41 (dd, J = 13.5, 6.0 Hz, 1H), 7.25-7.22 (m, 2H), 4.50 (br, 2H), 4.45 (s, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 161.1 (J = 246.0 Hz), 133.2 (J = 3.1 Hz), 130.4 (J = 8.3 Hz), 124.5 (J = 3.4 Hz), 117.4 (J = 15.0 Hz), 115.4 (J = 21.5 Hz), 46.7. ^{19}F NMR (471 MHz, DMSO-d_6) δ -116.74. HRMS (ESI) calcd for $C_7H_{10}FN_2O_2S^+$ $(M + H)^+$ 205.0442, found 205.0441.



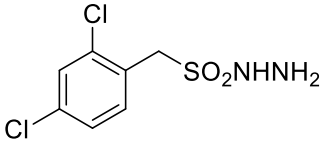
2m

(2-Chlorophenyl)methanesulfonohydrazide (**2m**) was obtained (860 mg, 78% yield) as a white solid. m.p. 92-94 ℃. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.06 (br, 1H), 7.53-7.48 (m, 2H), 7.40-7.34 (m, 2H), 4.54 (s, 2H), 4.50 (br, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 134.5, 133.2, 129.9, 129.5, 128.2, 127.2, 50.3. HRMS (ESI) calcd for $C_7H_{10}ClN_2O_2S^+$ $(M + H)^+$ 221.0144, found 221.0143.



2n

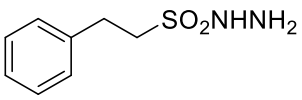
(2,5-Dimethylphenyl)methanesulfonohydrazide (**2n**) was obtained (716 mg, 67% yield) as a white solid. m.p. 101-103 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.95 (br, 1H), 7.09 (d, J = 7.8 Hz, 2H), 7.04 (d, J = 7.8 Hz, 1H), 4.48 (br, 2H), 4.35 (s, 2H), 2.32 (s, 3H), 2.27 (s, 3H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 135.0, 134.7, 132.3, 130.1, 128.7, 128.1, 50.9, 20.5, 18.9. HRMS (ESI) calcd for $C_9H_{15}N_2O_2S^+$ $(M + H)^+$ 215.0849, found 215.0848.



2o

(2,4-Dichlorophenyl)methanesulfonohydrazide (**2o)** was obtained (852 mg, 67% yield) as a white solid. m.p. 110-112 ℃. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.12 (br, 1H), 7.63 (s, 1H), 7.53 (d, J = 8.3 Hz, 1H), 7.44 (d, J = 8.3 Hz, 1H), 4.54 (s, 2H), 4.52 (br, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 135.4,

134.4, 133.7, 128.9, 127.5, 127.4, 49.9. HRMS (ESI) calcd for $C_7H_9Cl_2N_2O_2S^+$ $(M + H)^+$ 254.9756, found 254.9752.

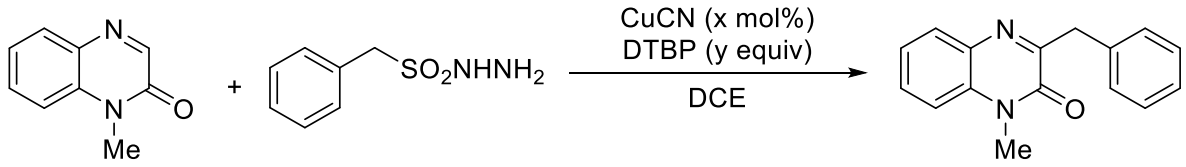


2p

2-Phenylethane-1-sulfonohydrazide (**2p**)[6] was obtained (531 mg, 53% yield) as a white solid. m.p. 48-50 °C. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.96 (br, 1H), 7.33-7.29 (m, 4H), 7.24-7.21 (m, 1H), 4.44 (br, 2H), 3.36-3.33 (m, 2H), 2.96-2.93 (m, 2H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 138.7, 128.5, 126.5, 48.2, 29.0. HRMS (ESI) calcd for $C_8H_{13}N_2O_2S^+$ $(M + H)^+$ 201.0692, found 201.0690.

3. Screening of the reaction conditions

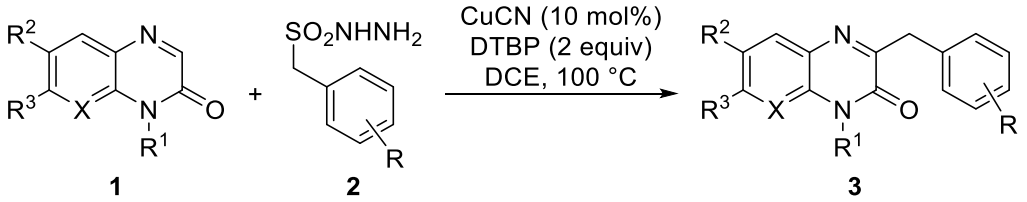
The following reactions were performed according to the general procedure as shown below. Summarized below are some of the results.



1a (0.20 mmol) **2a** (z mmol) **3a**

Entry	x	y	z	Temperature (°C)	Time (h)	Yield (%) of **3a**
1	5	2	0.30	100	10	65
2	10	2	0.30	100	10	76
3	20	2	0.30	100	10	55
4	10	3	0.30	100	10	44
5	10	4	0.30	100	10	42
6	10	5	0.30	100	10	18
7	10	2	0.24	100	10	68
8	10	2	0.40	100	10	62
9	10	2	0.60	100	10	72
10	10	2	0.30	70	10	28
11	10	2	0.30	25	10	0
12	10	2	0.30	100	3	58
13	10	2	0.30	100	6	68
14	10	2	0.30	100	16	67
15	10	2	0.30	100	24	74

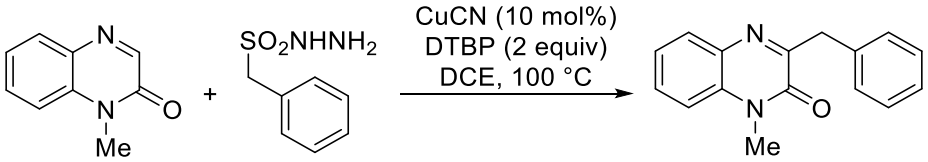
4. General procedure for the C-3 benzylation of quinoxalin-2(1*H*)-ones



1 **2** **3**

To a 10 mL Schlenk tube equipped with a magnetic stir bar was charged with quinoxalin-2(1*H*)-one **1** (0.20 mmol), benzylsulfonyl hydrazide **2** (0.30 mmol), and CuCN (1.79 mg, 0.020 mmol, 10 mol%). The tube was sealed with a septum, evacuated, and backfilled with nitrogen three times. 1,2-Dichloroethane (1.0 mL) and di-*tert*-butyl peroxide (58.5 mg, 73.5 μL, 0.40 mmol) were added successively via syringe with gentle stirring. The mixture was heated at 100 °C for 10 h, and then cooled to room temperature. The excess solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography (petroleum/ethyl acetate = 10:1~5:1 v/v) to give compound **3**.

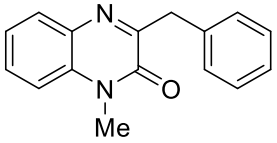
5. A gram-scale reaction



1a **2a** **3a**

To a 100 mL Schlenk tube equipped with a magnetic stir bar was charged with quinoxalin-2(1*H*)-one **1a** (1.60 g, 10.0 mmol), benzylsulfonyl hydrazide **2a** (2.79 g, 15.0 mmol), and CuCN (89.5 mg, 1.00 mmol, 10 mol%). The tube was sealed with a septum, evacuated, and backfilled with nitrogen three times. 1,2-Dichloroethane (10 mL) and di-*tert*-butyl peroxide (2.92 g, 3.67 mL, 20.0 mmol) were added successively via syringe with gentle stirring. The mixture was heated at 100 °C for 10 h, and then cooled to room temperature. The excess solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography (petroleum/ethyl acetate = 10:1~5:1 v/v) to give compound **3a** (1.88 g, 75% yield).

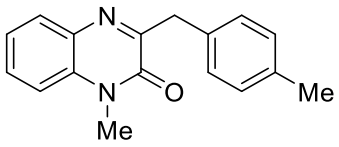
6. Analytical data for the products



3a

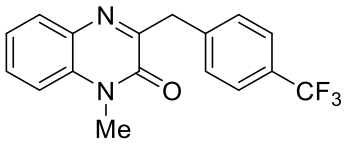
3-Benzyl-1-methylquinoxalin-2(1*H*)-one (**3a**)[7,8] was obtained (38.0 mg, 76% yield) as a white solid. m.p. 89-90 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, J = 8.0, 1.6 Hz, 1H), 7.53-7.46 (m, 3H),

7.34-7.19 (m, 5H), 4.26 (s, 2H), 3.65 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 154.9, 137.2, 133.5, 132.9, 130.1, 130.0, 129.7, 128.5, 126.7, 123.7, 113.7, 40.9, 29.2. HRMS (ESI) calcd for $C_{16}H_{15}N_2O^+$ $(M + H)^+$ 251.1179, found 251.1175.



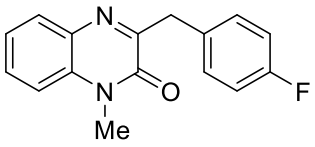
3b

1-Methyl-3-(4-methylbenzyl)quinoxalin-2(1*H*)-one (**3b**)[7,8] was obtained (39.8 mg, 75% yield) as a white solid. m.p. 117-118 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, J = 8.0, 1.2 Hz, 1H), 7.53-7.48 (m, 1H), 7.37-7.30 (m, 3H), 7.25 (d, J = 7.2 Hz, 1H), 7.10 (d, J = 8.0 Hz, 2H), 4.22 (s, 2H), 3.64 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 154.9, 136.2, 134.1, 133.5, 132.9, 130.1, 129.9, 129.5, 129.2, 123.7, 113.6, 40.5, 29.2, 21.2. HRMS (ESI) calcd for $C_{17}H_{17}N_2O^+$ $(M + H)^+$ 265.1335, found 265.1338.



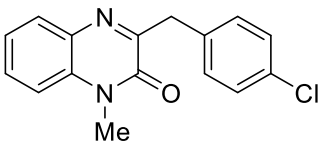
3c

1-Methyl-3-(4-(trifluoromethyl)benzyl)quinoxalin-2(1*H*)-one (**3c**) was obtained (42.4 mg, 67% yield) as a white solid. m.p. 125-126 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, J = 8.0, 1.6 Hz, 1H), 7.59-7.53 (m, 5H), 7.36-7.33 (m, 1H), 7.30-7.26 (m, 1H), 4.31 (s, 2H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 154.8, 141.3, 133.5, 132.8, 130.3, 130.2, 130.0, 129.0 (q, J = 32.1 Hz), 125.4 (q, J = 3.8 Hz), 124.4 (q, J = 270.4 Hz), 123.9, 113.8, 40.7, 29.3. HRMS (ESI) calcd for $C_{17}H_{14}F_3N_2O^+$ $(M + H)^+$ 319.1053, found 319.1055.



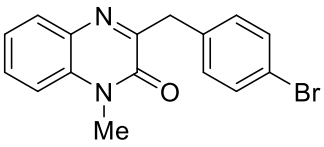
3d

3-(4-Fluorobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3d**)[7,8] was obtained (43.3 mg, 81% yield) as a white solid. m.p. 109-110 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, J = 8.0, 1.2 Hz, 1H), 7.51 (ddd, J = 8.8, 7.6, 1.6 Hz, 1H), 7.42 (dd, J = 8.8, 5.6 Hz, 2H), 7.35-7.31 (m, 1H), 7.27-7.24 (m, 1H), 6.96 (t, J = 8.8, 2H), 4.22 (s, 2H), 3.65 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.8 (d, J = 242.9 Hz), 159.0, 154.7, 133.3, 132.7 (132.71, 132.69), 131.1 (d, J = 7.9 Hz), 130.0 (d, J = 7.6 Hz), 123.7, 115.2 (d, J = 21.1 Hz), 113.6, 40.0, 29.1. HRMS (ESI) calcd for $C_{16}H_{14}FN_2O^+$ $(M + H)^+$ 269.1085, found 269.1086.



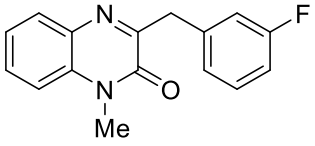
3e

3-(4-Chlorobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3e**)[8] was obtained (34.4 mg, 60% yield) as a white solid. m.p. 129-130 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 7.6 Hz, 1H), 7.52 (t, J = 7.8 Hz, 1H), 7.39 (d, J = 8.0 Hz, 2H),7.33 (t, J = 7.6 Hz, 1H), 7.25 (t, J = 7.6 Hz, 3H), 4.22 (s, 2H), 3.65 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.9, 154.8, 135.6, 133.4, 132.8, 132.6, 131.0, 130.2, 130.1, 128.6, 123.8, 113.7, 40.2, 29.2. HRMS (ESI) calcd for $C_{16}H_{14}ClN_2O^+$ $(M + H)^+$ 285.0789, found 285.0790.



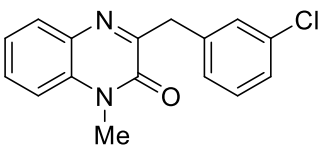
3f

3-(4-Bromobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3f**)[7] was obtained (54.6 mg, 83% yield) as a white solid. m.p. 123-124 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, J = 8.0, 1.6 Hz, 1H), 7.53 (ddd, J = 8.6, 7.4, 1.5 Hz, 1H), 7.42-7.38 (m, 2H), 7.36-7.31 (m, 3H), 7.28-7.26 (m, 1H), 4.20 (s, 2H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 154.7, 136.1, 133.4, 132.8, 131.5, 131.4, 130.2, 130.1, 123.8, 120.7, 113.7, 40.3, 29.2. HRMS (ESI) calcd for $C_{16}H_{14}BrN_2O^+$ $(M + H)^+$ 329.0284, found 329.0285.



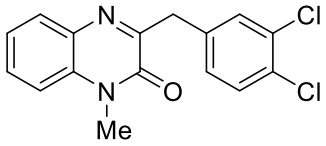
3g

3-(3-Fluorobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3g**) was obtained (35.3 mg, 66% yield) as a white solid. m.p. 84-85 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, J = 8.0, 1.2 Hz, 1H), 7.55-7.51 (m, 1H), 7.36-7.32 (m, 1H), 7.28-7.23 (m, 3H), 7.16 (d, J = 10.4 Hz, 1H), 6.92-6.86 (m, 1H), 4.25 (s, 2H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9 (d, J = 243.8 Hz), 158.7, 154.8, 139.6 (d, J = 7.6 Hz), 133.4, 132.8, 130.1 (d, J = 9.8 Hz), 129.9, 129.8, 125.3 (d, J = 2.8 Hz), 123.8, 116.5 (d, J = 21.4 Hz), 113.7, 113.5, 40.5, 29.3. HRMS (ESI) calcd for $C_{16}H_{14}FN_2O^+$ $(M + H)^+$ 269.1085, found 269.1084.



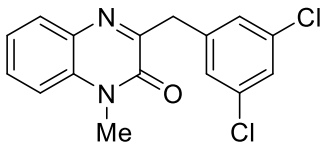
3h

3-(3-Chlorobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3h**) was obtained (44.5 mg, 78% yield) as a white solid. m.p. 103-104 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (dd, J = 8.0, 1.2 Hz, 1H), 7.38-7.33 (m, 2H), 7.24-7.15 (m, 2H), 7.11-7.04 (m, 3H), 4.10 (s, 2H), 3.50 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 154.6, 139.1, 134.1, 133.3, 132.7, 130.1, 130.0, 129.6, 129.5, 127.9, 126.8, 123.7, 113.6, 40.3, 29.2. HRMS (ESI) calcd for $C_{16}H_{14}ClN_2O^+$ $(M + H)^+$ 285.0789, found 285.0791.



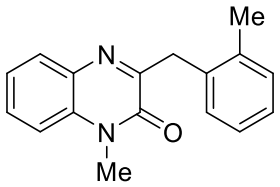
3i

3-(3,4-Dichlorobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3i**) was obtained (42.0 mg, 66% yield) as a white solid. m.p. 162-163 ℃. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (dd, J = 8.0, 1.0 Hz, 1H), 7.55-7.52 (m, 2H), 7.36-7.26 (m, 4H), 4.19 (s, 2H), 3.66 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.2, 154.6, 137.3, 133.4, 132.7, 132.3, 131.4, 130.8, 130.3, 130.1, 129.2, 123.9, 113.8, 39.9, 29.3. HRMS (ESI) calcd for $C_{16}H_{13}Cl_2N_2O^+$ $(M + H)^+$ 319.0399, found 319.0404.



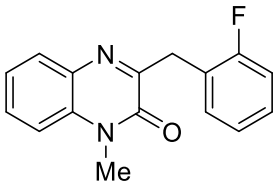
3j

3-(3,5-Dichlorobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3j**) was obtained (50.6 mg, 79% yield) as a white solid. m.p. 142-143 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (dd, J = 8.0, 1.2 Hz, 1H), 7.54-7.50 (m, 1H), 7.35-7.31 (m, 3H), 7.27-7.24 (m, 1H), 7.18 (t, J = 2.0 Hz, 1H), 4.18 (s, 2H), 3.65 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 154.6, 140.4, 134.7, 133.4, 132.7, 130.4, 130.1, 128.0, 126.9, 123.8, 113.7, 40.1, 29.3. HRMS (ESI) calcd for $C_{16}H_{13}Cl_2N_2O^+$ $(M + H)^+$ 319.0399, found 319.0391.



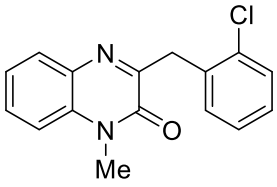
3k

1-Methyl-3-(2-methylbenzyl)quinoxalin-2(1*H*)-one (**3k**)[7] was obtained (28.8 mg, 54% yield) as a white solid. m.p. 94-95 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (dd, J = 7.8, 1.4 Hz, 1H), 7.53-7.48 (m, 1H), 7.35-7.32 (m, 1H), 7.31-7.25 (m, 2H), 7.19-7.12 (m, 3H), 4.28 (s, 2H), 3.67 (s, 3H), 2.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.2, 154.8, 137.5, 135.6, 133.3, 132.8, 130.4, 130.2, 130.1, 129.9, 126.8, 125.9, 123.6, 113.6, 38.0, 29.2, 20.1. HRMS (ESI) calcd for $C_{17}H_{17}N_2O^+$ $(M + H)^+$ 265.1335, found 265.1337.



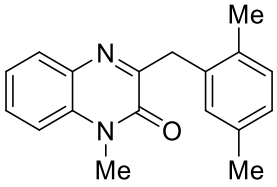
3l

3-(2-Fluorobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3l**) was obtained (46.1mg, 86% yield) as a white solid. m.p. 135-136 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (dd, J = 8.0, 1.6 Hz, 1H), 7.51 (ddd, J = 8.6, 7.4, 1.5 Hz, 1H), 7.36 (td, J = 7.8, 1.8 Hz, 1H), 7.33-7.19 (m, 3H), 7.09-7.03 (m, 2H), 4.32 (s, 2H), 3.68 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.5 (d, J = 245.1 Hz), 158.2, 154.8, 133.4, 132.8, 131.7 (d, J = 4.4 Hz), 130.2, 130.1, 128.5 (d, J = 8.0 Hz), 124.3 (d, J = 15.6 Hz), 124.0 (d, J = 3.6 Hz), 123.7, 115.5 (d, J = 21.8 Hz), 113.7, 33.9, 29.2. HRMS (ESI) calcd for $C_{16}H_{14}FN_2O^+$ $(M + H)^+$ 269.1085, found 269.1084.



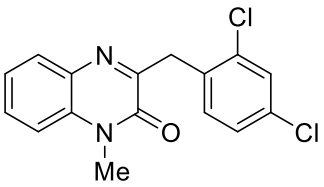
3m

3-(2-Chlorobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3m**)[7] was obtained (47.4 mg, 83% yield) as a white solid. m.p. 141-142 ℃. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, J = 7.5 Hz, 1H), 7.50 (t, J = 7.5 Hz, 1H), 7.39-7.25 (m, 4H), 7.19-7.18 (m, 2H), 4.41 (s, 2H), 3.68 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.2, 154.7, 135.3, 134.8, 133.2, 132.7, 131.4, 130.2, 130.0, 129.5, 128.0, 126.7, 123.6, 113.6, 38.1, 29.2. HRMS (ESI) calcd for $C_{16}H_{14}ClN_2O^+$ $(M + H)^+$ 285.0789, found 285.0782.



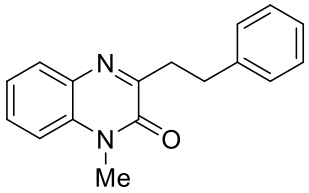
3n

3-(2,5-Dimethylbenzyl)-1-methylquinoxalin-2(1*H*)-one (**3n**) was obtained (22.7 mg, 41% yield) as a white solid. m.p. 110-111 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (dd, J = 7.8, 1.4 Hz, 1H), 7.51-7.46 (m, 1H), 7.31-7.27 (m, 1H), 7.25-7.22 (m, 1H), 7.13 (s, 1H), 7.06 (d, J = 7.6 Hz, 1H), 6.94 (d, J = 7.6 Hz, 1H), 4.23 (s, 2H), 3.65 (s, 3H), 2.41 (s, 3H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 154.8, 135.3, 135.2, 134.3, 133.3, 132.8, 130.8, 130.3, 130.1, 129.8, 127.5, 123.5, 113.6, 37.9, 29.2, 21.1, 19.7. HRMS (ESI) calcd for $C_{18}H_{19}N_2O^+$ $(M + H)^+$ 279.1492, found 279.1484.



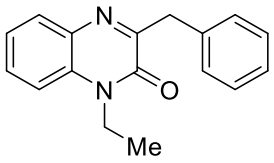
3o

3-(2,4-Dichlorobenzyl)-1-methylquinoxalin-2(1*H*)-one (**3o**) was obtained (55.8 mg, 87% yield) as a white solid. m.p. 135-136 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (dd, J = 8.0, 1.6 Hz, 1H), 7.53-7.48 (m, 1H), 7.38 (d, J = 2.4 Hz, 1H), 7.31-7.26 (m, 3H), 7.16 (dd, J = 8.4, 2.0 Hz, 1H), 4.36 (s, 2H), 3.68 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.5, 154.6, 135.5, 133.9, 133.2, 133.0, 132.6, 132.3, 130.1, 129.3, 126.9, 123.7, 113.6, 37.6, 29.2. HRMS (ESI) calcd for $C_{16}H_{13}Cl_2N_2O^+$ $(M + H)^+$ 319.0399, found 319.0391.



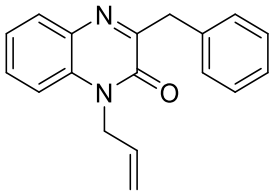
3p

1-Methyl-3-phenethylquinoxalin-2(1*H*)-one (**3p**)[8] was obtained (10.4 mg, 20% yield) as a white solid. m.p. 87-88 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 7.2 Hz, 1H), 7.54-7.50 (m, 1H), 7.35-7.25 (m, 6H), 7.19 (t, J = 7.2 Hz, 1H), 3.69 (s, 3H), 3.29-3.25 (m, 2H), 3.15-3.11 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 154.9, 141.7, 133.2, 132.8, 129.8, 128.7, 128.4, 126.0, 123.7, 113.7, 36.1, 32.6, 29.1. HRMS (ESI) calcd for $C_{17}H_{17}N_2O^+$ $(M + H)^+$ 265.1336, found 265.1338.



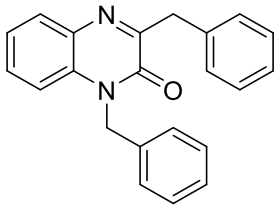
3q

3-Benzyl-1-ethylquinoxalin-2(1*H*)-one (**3q**)[9] was obtained (42.2 mg, 80% yield) as a white solid. m.p. 79-80 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, J = 7.8, 1.4 Hz, 1H), 7.48-7.44 (m, 3H), 7.30-7.17 (m, 5H), 4.26 (s, 2H), 4.23 (q, J = 7.2 Hz, 2H), 1.31 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 154.2, 137.2, 133.1, 132.2, 130.2, 129.9, 129.6, 128.4, 126.6, 123.4, 113.4, 40.6, 37.4, 12.4. HRMS (ESI) calcd for $C_{17}H_{17}N_2O^+$ $(M + H)^+$ 265.1335, found 265.1332.



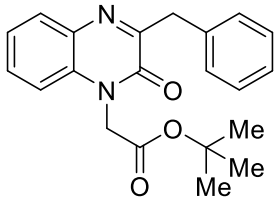
3r

1-Allyl-3-benzylquinoxalin-2(1*H*)-one (**3r**)[8] was obtained (42.1 mg, 76% yield) as a white solid. m.p. 75-76 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, J = 8.0, 1.2 Hz, 1H), 7.48-7.46 (m, 3H), 7.32-7.18 (m, 5H), 5.94-5.84 (m, 1H), 5.25-5.21 (m, 1H), 5.15-5.10 (m, 1H), 4.85 (dt, J = 5.2, 1.6 Hz, 2H), 4.28 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 154.4, 137.2, 133.0, 132.6, 130.7, 130.1, 129.9, 129.6, 128.5, 126.7, 123.7, 118.2, 114.2, 44.7, 40.8. HRMS (ESI) calcd for $C_{18}H_{17}N_2O^+$ $(M + H)^+$ 277.1335, found 277.1333.



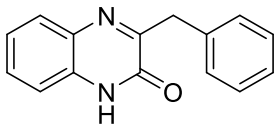
3s

1,3-Dibenzylquinoxalin-2(1*H*)-one (**3s**)[7,8] was obtained (45.5 mg, 70% yield) as a white solid. m.p. 128-129 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (dd, J = 7.8, 1.4 Hz, 1H), 7.50-7.48 (m, 2H), 7.35-7.15 (m, 11H), 5.40 (s, 2H), 4.32 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 154.9, 137.2, 135.3, 133.1, 132.7, 130.1, 129.9, 129.6, 129.0, 128.5, 127.7, 126.9, 126.7, 123.7, 114.4, 46.0, 40.8. HRMS (ESI) calcd for $C_{22}H_{19}N_2O^+$ $(M + H)^+$ 327.1492, found 327.1488.



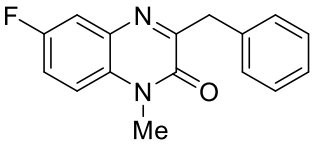
3t

tert-Butyl 2-(3-benzyl-2-oxoquinoxalin-1(2*H*)-yl)acetate (**3t**) was obtained (45.0 mg, 64% yield) as a white solid. m.p. 85-86 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.83 (m, 1H), 7.44 (d, J = 7.2 Hz, 3H), 7.44 (q, J = 7.2 Hz, 3H), 7.18 (t, J = 7.4 Hz, 1H), 7.00 (d, J = 8.4 Hz, 1H), 4.86 (s, 2H), 4.26 (s, 2H), 1.41 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1, 159.1, 154.3, 136.9, 132.8, 132.5, 130.2, 130.0, 129.5, 128.4, 126.6, 123.8, 113.1, 83.1, 44.3, 40.7, 27.9. HRMS (ESI) calcd for $C_{21}H_{23}N_2O_3^+$ $(M + H)^+$ 351.1703, found 351.1701.



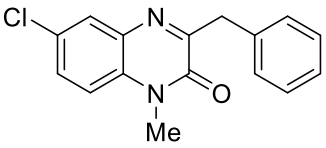
3u

3-Benzylquinoxalin-2(1*H*)-one (**3u**)[8,10] was obtained (27.0 mg, 57% yield) as a white solid. m.p. 199-200 ℃. ^{1}H NMR (500 MHz, $CDCl_3$) δ 12.47 (br, 1H), 7.84 (d, J = 8.0 Hz, 1H), 7.48 (d, J = 6.8 Hz, 3H), 7.34-7.30 (m, 1H), 7.29-7.25 (m, 3H), 7.20 (t, J = 7.5 Hz, 1H), 4.30 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.8, 156.7, 137.1, 133.0, 131.3, 130.1, 129.7, 129.1, 128.5, 126.8, 124.3, 115.8, 40.1. HRMS (ESI) calcd for $C_{15}H_{13}N_2O^+$ $(M + H)^+$ 237.1022, found 237.1018.



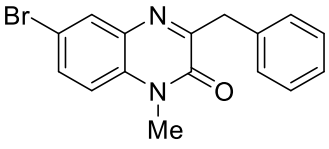
3v

3-Benzyl-6-fluoro-1-methylquinoxalin-2(1*H*)-one (**3v**)[8] was obtained (39.0 mg, 73% yield) as a white solid. m.p. 132-133 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, J = 8.8, 6.0 Hz, 1H), 7.44 (d, J = 7.2 Hz, 2H), 7.30-7.25 (m, 2H), 7.20 (t, J = 7.4 Hz, 1H), 7.02 (td, J = 8.8, 2.4 Hz, 1H), 6.92 (dd, J = 10.0, 2.8 Hz, 1H), 4.22 (s, 2H), 3.59 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2 (d, J = 248.6 Hz), 158.2 (d, J = 3.4 Hz), 154.6, 137.0, 134.8 (d, J = 11.5 Hz), 131.9 (d, J = 10.4 Hz), 129.6, 129.6 (d, J = 2.2 Hz), 128.5, 126.7, 111.4 (d, J = 23.2 Hz), 100.6 (d, J = 27.6 Hz), 40.7, 29.4. HRMS (ESI) calcd for $C_{16}H_{14}FN_2O^+$ $(M + H)^+$ 269.1085, found 237.1081.



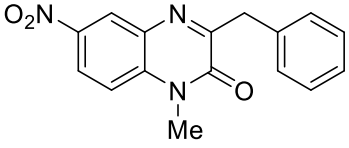
3w

3-Benzyl-6-chloro-1-methylquinoxalin-2(1*H*)-one (**3w**)[7,8] was obtained (42.9 mg, 75% yield) as a white solid. m.p. 168-169 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, J = 8.4 Hz, 1H), 7.43 (d, J = 7.6 Hz, 2H), 7.27 (t, J = 7.6 Hz, 2H), 7.24-7.16 (m, 3H), 4.21 (s, 2H), 3.56 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 154.4, 136.8, 135.7, 134.1, 131.2, 131.0, 129.6, 128.5, 126.7, 123.9, 113.6, 40.7, 29.2. HRMS (ESI) calcd for $C_{16}H_{14}ClN_2O^+$ $(M + H)^+$ 285.0789, found 285.0789.



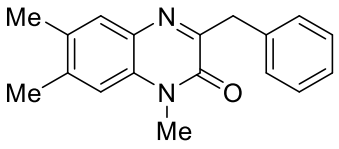
3x

3-Benzyl-6-bromo-1-methylquinoxalin-2(1*H*)-one (**3x**)[7] was obtained (35.5 mg, 54% yield) as a white solid. m.p. 172-173 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, J = 8.0 Hz, 1H), 7.45-7.41 (m, 4H), 7.29 (t, J = 7.6 Hz, 2H), 7.21 (t, J = 7.4 Hz, 1H), 4.23 (s, 2H), 3.61 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 154.5, 136.8, 134.5, 131.7, 131.3, 129.7, 128.6, 126.9, 126.8, 124.0, 116.7, 40.9, 29.4. HRMS (ESI) calcd for $C_{16}H_{14}BrN_2O^+$ $(M + H)^+$ 329.0284, found 329.0282.



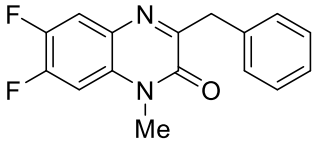
3y

3-Benzyl-1-methyl-6-nitroquinoxalin-2(1*H*)-one (**3y**) was obtained (19.0 mg, 32% yield) as a white solid. m.p. 132-133 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 (d, J = 2.8 Hz, 1H), 8.35 (dd, J = 9.2, 2.8 Hz, 1H), 7.45 (d, J = 7.2 Hz, 2H), 7.36 (d, J = 9.2 Hz, 1H), 7.32-7.21 (m, 3H), 4.27 (s, 2H), 3.70 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0, 154.4, 143.4, 138.0, 136.1, 131.9, 129.7, 128.7, 127.0, 125.8, 124.5, 114.3, 40.8, 29.8. HRMS (ESI) calcd for $C_{16}H_{14}N_3O_3^+$ $(M + H)^+$ 296.1030, found 296.1027.



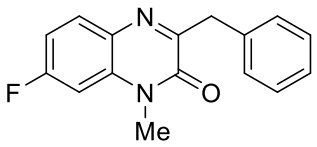
3z

3-Benzyl-1,6,7-trimethylquinoxalin-2(1*H*)-one (**3z**)[7,8] was obtained (36.6 mg, 66% yield) as a white solid. m.p. 172-173 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.45 (d, J = 7.2 Hz, 2H), 7.26 (t, J = 7.6 Hz, 2H), 7.17 (t, J = 7.2 Hz, 1H), 6.95 (s, 1H), 4.22 (s, 2H), 3.57 (s, 3H), 2.35 (s, 3H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 154.7, 139.6, 137.4, 132.4, 131.3, 131.1, 130.0, 129.5, 128.3, 126.5, 114.1, 40.7, 29.0, 20.5, 19.2. HRMS (ESI) calcd for $C_{18}H_{19}N_2O^+$ $(M + H)^+$ 279.1492, found 279.1487.



3aa

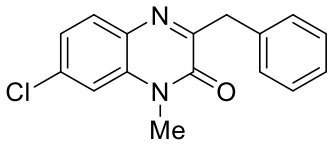
3-Benzyl-6,7-difluoro-1-methylquinoxalin-2(1*H*)-one (**3aa**) was obtained (46.1 mg, 81% yield) as a white solid. m.p. 94-95 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (dd, J = 10.2, 8.2 Hz, 1H), 7.41 (d, J = 7.2 Hz, 2H), 7.25 (t, J = 7.6 Hz, 2H), 7.17 (t, J = 7.4 Hz, 1H), 7.01 (dd, J = 11.3, 7.0 Hz, 1H), 4.20 (s, 2H), 3.55 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8 (d, J = 3.5 Hz), 154.2, 151.1 (dd, J = 251.2, 14.3 Hz), 146.5 (dd, J = 245.2, 13.9 Hz), 136.6, 130.5 (dd, J = 8.9, 1.7 Hz), 129.5, 128.9 (dd, J = 9.2, 2.9 Hz), 128.4, 126.7, 117.5 (dd, J = 17.9, 2.0 Hz), 102.2 (*d*, J = 22.9 Hz), 40.6, 29.6. HRMS (ESI) calcd for $C_{16}H_{13}F_2N_2O^+$ $(M + H)^+$ 287.0990, found 287.0985.



3ab

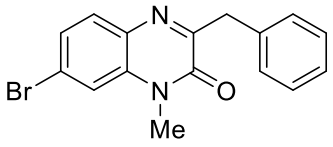
3-Benzyl-6-fluoro-1-methylquinoxalin-2(1*H*)-one (**3ab**)[7] was obtained (40.5 mg, 75% yield) as a white solid. m.p. 117-118 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (dd, J = 8.8, 2.8 Hz, 1H), 7.44 (d, J = 7.2 Hz, 2H), 7.29-7.23 (m, 2H), 7.22-7.13 (m, 3H), 4.24 (s, 2H), 3.60 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.8, 158.6 (d, J = 242.0 Hz), 154.3, 136.8, 133.3 (d, J = 11.2 Hz), 130.0 (d, J = 2.0 Hz),

129.6, 128.5, 126.7, 117.5 (d, J = 23.8 Hz), 115.4 (d, J = 22.3 Hz), 114.7 (d, J = 8.7 Hz), 40.8, 29.4. HRMS (ESI) calcd for $C_{16}H_{14}FN_2O$ $(M + H)^+$ 269.1085, found 269.1081.



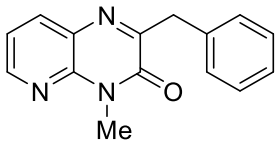
3ac

3-Benzyl-7-chloro-1-methylquinoxalin-2(1*H*)-one (**3ac**)[7] was obtained (37.7 mg, 66% yield) as a white solid. m.p. 97-98 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, J = 2.4 Hz, 1H), 7.45-7.43 (m, 2H), 7.40 (dd, J = 8.8, 2.4 Hz, 1H), 7.29-7.24 (m, 2H), 7.21-7.17 (m, 1H), 7.10 (d, J = 8.8 Hz, 1H), 4.23 (s, 2H), 3.58 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.7, 154.3, 136.7, 133.2, 132.0, 129.8, 129.6, 129.3, 128.8, 128.5, 126.8, 114.7, 40.7, 29.3. HRMS (ESI) calcd for $C_{16}H_{14}ClN_2O^+$ $(M + H)^+$ 285.0789, found 285.0785.



3ad

3-Benzyl-7-bromo-1-methylquinoxalin-2(1*H*)-one (**3ad**)[7] was obtained (43.8 mg, 67% yield) as a white solid. m.p. 115-116 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 2.0 Hz, 1H), 7.52 (dd, J = 8.8, 2.4 Hz, 1H), 7.43 (d, J = 7.2 Hz, 2H), 7.29-7.24 (m, 2H), 7.19 (t, J = 7.2 Hz, 1H), 7.03 (d, J = 8.8 Hz, 1H), 4.23 (s, 2H), 3.57 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.6, 154.3, 136.7, 133.5, 132.5, 132.4, 132.3, 129.6, 128.5, 126.7, 116.1, 115.0, 40.7, 29.3. HRMS (ESI) calcd for $C_{16}H_{14}BrN_2O^+$ $(M + H)^+$ 329.0284, found 329.0279.

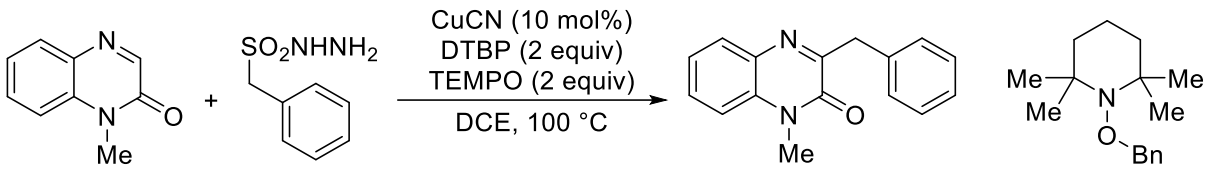


3ae

2-Benzyl-4-methylpyrido[2,3-*b*]pyrazin-3(4*H*)-one (**3ae**) was obtained (26.2 mg, 52% yield) as a white solid. m.p. 108-109 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, J = 3.2 Hz, 1H), 8.12 (d, J = 8.0 Hz, 1H), 7.46 (d, J = 7.2 Hz, 2H), 7.31-7.26 (m, 3H), 7.21 (t, J = 7.2 Hz, 1H), 4.27 (s, 2H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.7, 156.0, 149.1, 144.2, 137.2, 136.6, 129.7, 128.6, 128.2, 126.8, 119.6, 40.7, 27.9. HRMS (ESI) calcd for $C_{15}H_{14}N_3O^+$ $(M + H)^+$ 252.1131, found 252.1132.

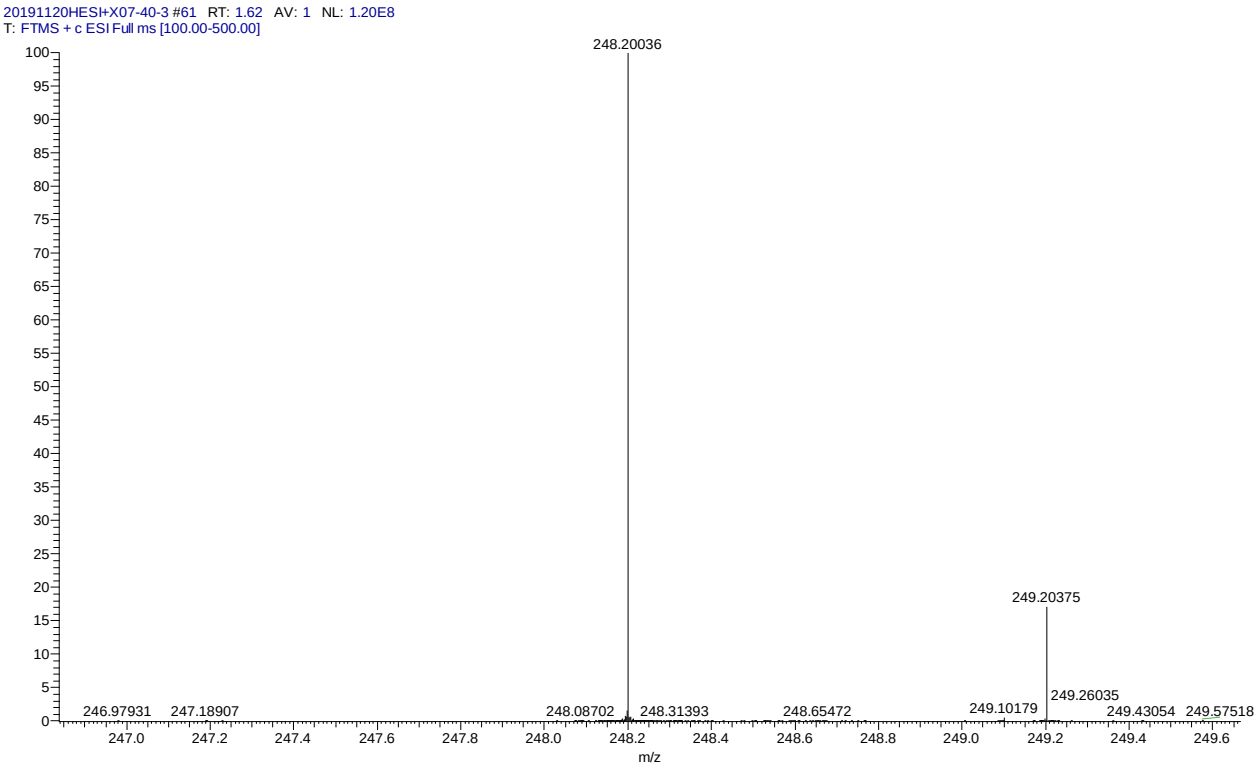
7. Radical capture experiments

(1) The reaction with 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO)



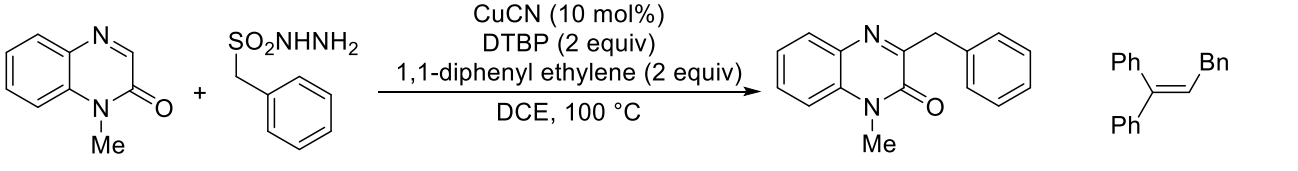
1a **2a** **3a** (trace) **4a** (detected by ESI-MS)

To a 10 mL Schlenk tube equipped with a magnetic stir bar was charged with quinoxalin-2(1*H*)-one **1a** (32.0 mg, 0.20 mmol), benzylsulfonyl hydrazide **2a** (55.8 mg, 0.30 mmol), CuCN (1.79 mg, 0.020 mmol, 10 mol%), and TEMPO (62.6 mg, 0.40 mmol). The tube was sealed with a septum, evacuated, and backfilled with nitrogen three times. 1,2-Dichloroethane (1.0 mL) and di-*tert*-butyl peroxide (58.5 mg, 73.5 μL, 0.40 mmol) were added successively via syringe with gentle stirring. The mixture was heated at 100 °C for 10 h, cooled to room temperature, and subjected to ESI-MS (positive mode) analysis. Copied below is the ESI-MS spectrum we obtained.



TEMPO-Bn (**4a**): HRMS (ESI) calcd for $C_{16}H_{26}NO^+$ $(M + H)^+$ 248.2009, found 248.2004.

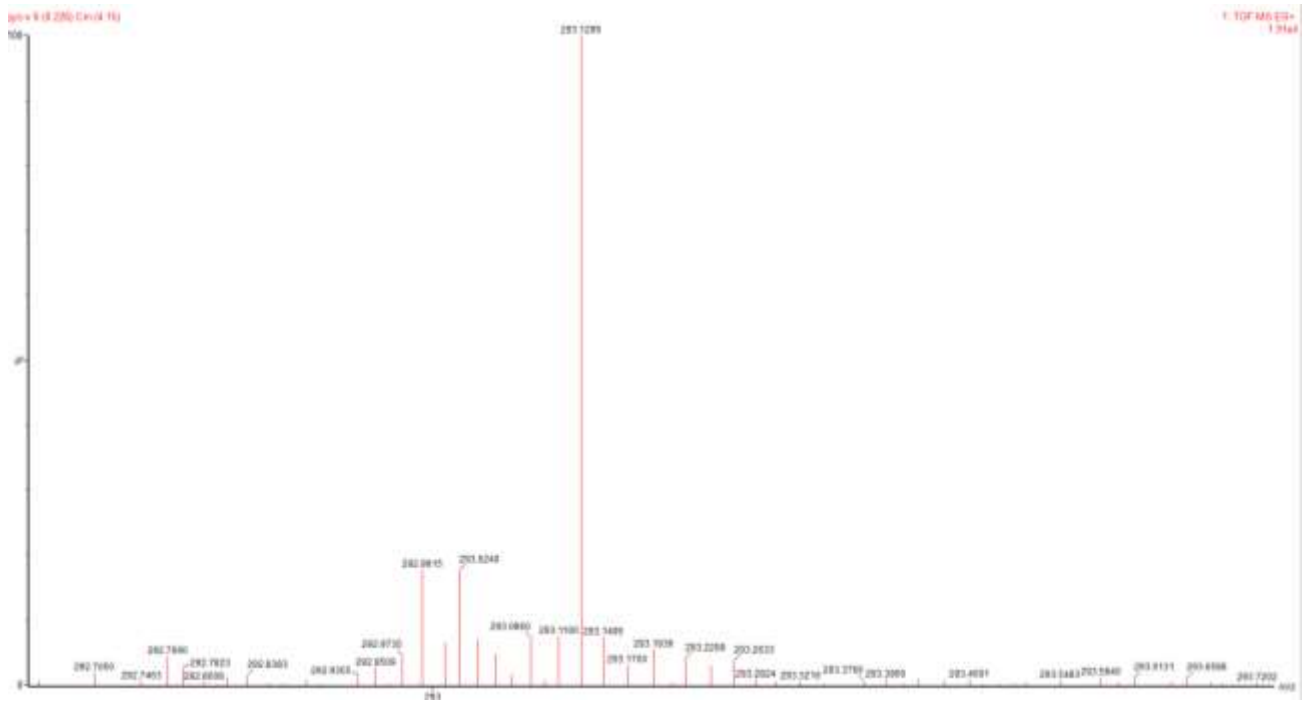
(2) The reaction with 1,1-diphenyl ethylene



1a **2a** **3a** (55% yield) **4b** (detected by ESI-MS)

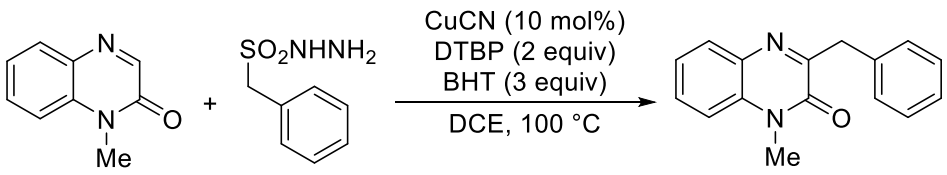
To a 10 mL Schlenk tube equipped with a magnetic stir bar was charged with quinoxalin-2(1*H*)-one **1a** (32.0 mg, 0.20 mmol), benzylsulfonyl hydrazide **2a** (55.8 mg, 0.30 mmol), CuCN (1.79 mg, 0.020 mmol, 10 mol%), and 1,1-diphenyl ethylene (108 mg, 106 μL, 0.60 mmol). The tube was sealed with a septum, evacuated, and backfilled with nitrogen three times. 1,2-Dichloroethane (1.0 mL) and di-*tert*-butyl peroxide (58.5 mg, 73.5 μL, 0.40 mmol) were added successively via syringe with gentle stirring. The mixture was heated at 100 °C for 10 h, and then cooled to room temperature. The residue was purified by silica gel column chromatography (petroleum/ethyl acetate = 10:1~5:1 v/v) to give compound **3a** (27.5 mg, 55% yield).

A very small portion of the above reaction mixture was subjected to ESI-MS (positive mode) analysis. Copied below is the ESI-MS spectrum we obtained.



Compound **4b:** HRMS (ESI) calcd for $C_{21}H_{18}Na^+$ $(M + Na)^+$ 2931307, found 293.1299.

(3) The reaction with 2,6-di-*tert*-butyl-4-methylphenol (BHT)



To a 10 mL Schlenk tube equipped with a magnetic stir bar was charged with quinoxalin-2(1*H*)-one **1a** (32.0 mg, 0.20 mmol), benzylsulfonyl hydrazide **2a** (55.8 mg, 0.30 mmol), CuCN (1.79 mg, 0.020 mmol, 10 mol%), and 2,6-di-*tert*-butyl-4-methylphenol (132 mg, 0.60 mmol). The tube was sealed with a septum, evacuated, and backfilled with nitrogen three times. 1,2-Dichloroethane (1.0 mL) and di-*tert*-butyl peroxide (58.5 mg, 73.5 μL, 0.40 mmol) were added successively via syringe with

gentle stirring. The mixture was heated at 100 °C for 10 h, and cooled to room temperature. The residue was purified by silica gel column chromatography (petroleum/ethyl acetate = 10:1~5:1 v/v) to give compound **3a** (30.1 mg, 60% yield).

8. References

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(3) Y. Yang, Y. Bao, Q. Guan, Q. Sun, Z. Zha and Z. Wang, *Green Chem.*, 2017, **19**, 112.

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(5) I. M. Tuchapskii and R. V. Vizgert, *J. Org. Chem., USSR*, 1975, **11**, 1901.

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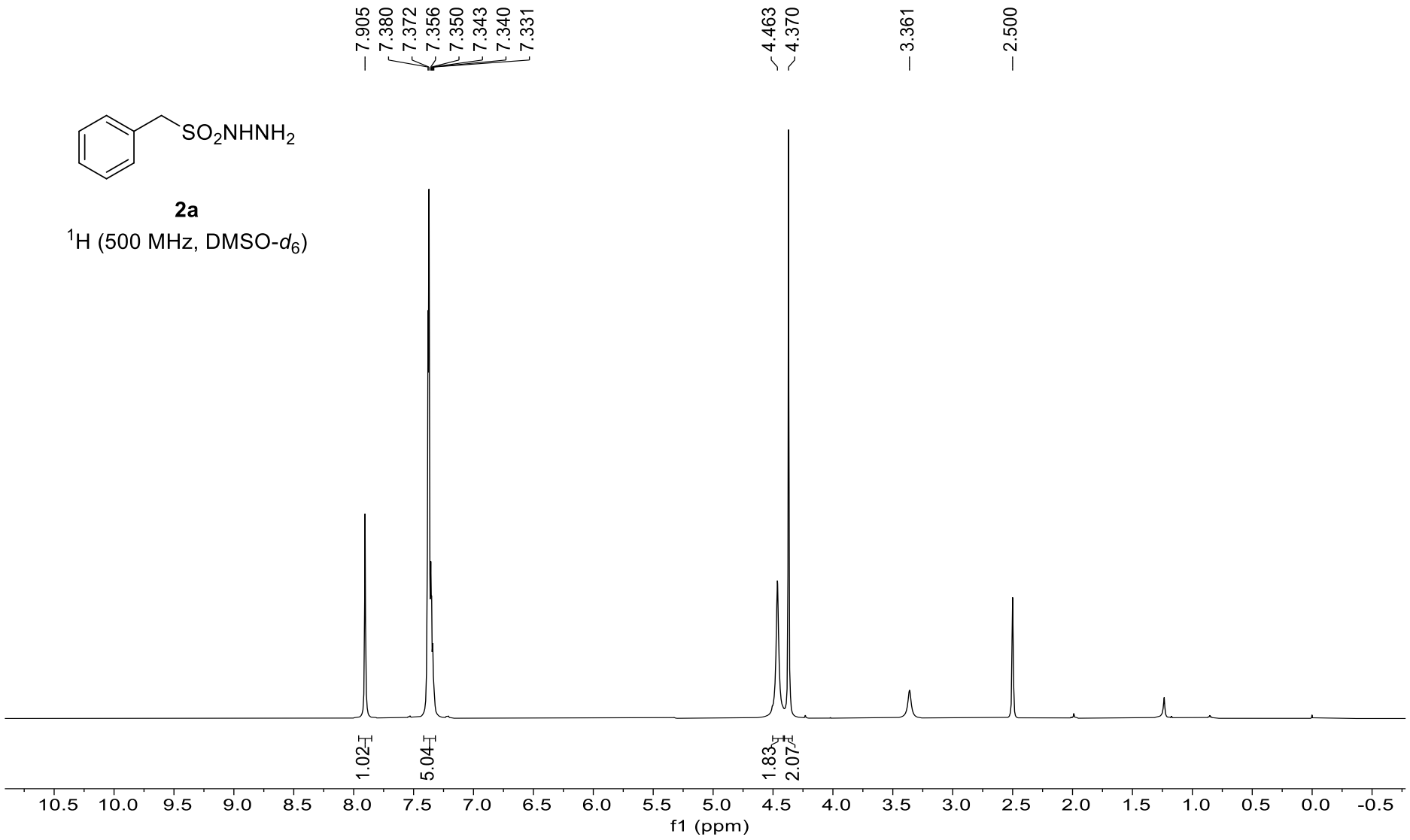
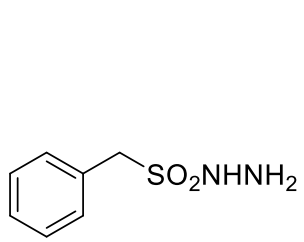
(9) B. Gerard, V. Eric, K. Micheline, C. Christine and E. Samer, WO 2009/109258 A1, 2009.

(10) Y. Gao, Z. Wu, L. Yu, Y. Wang and Y. Pan, *Angew. Chem. Int. Ed.*, 2020, **59**, 10859.

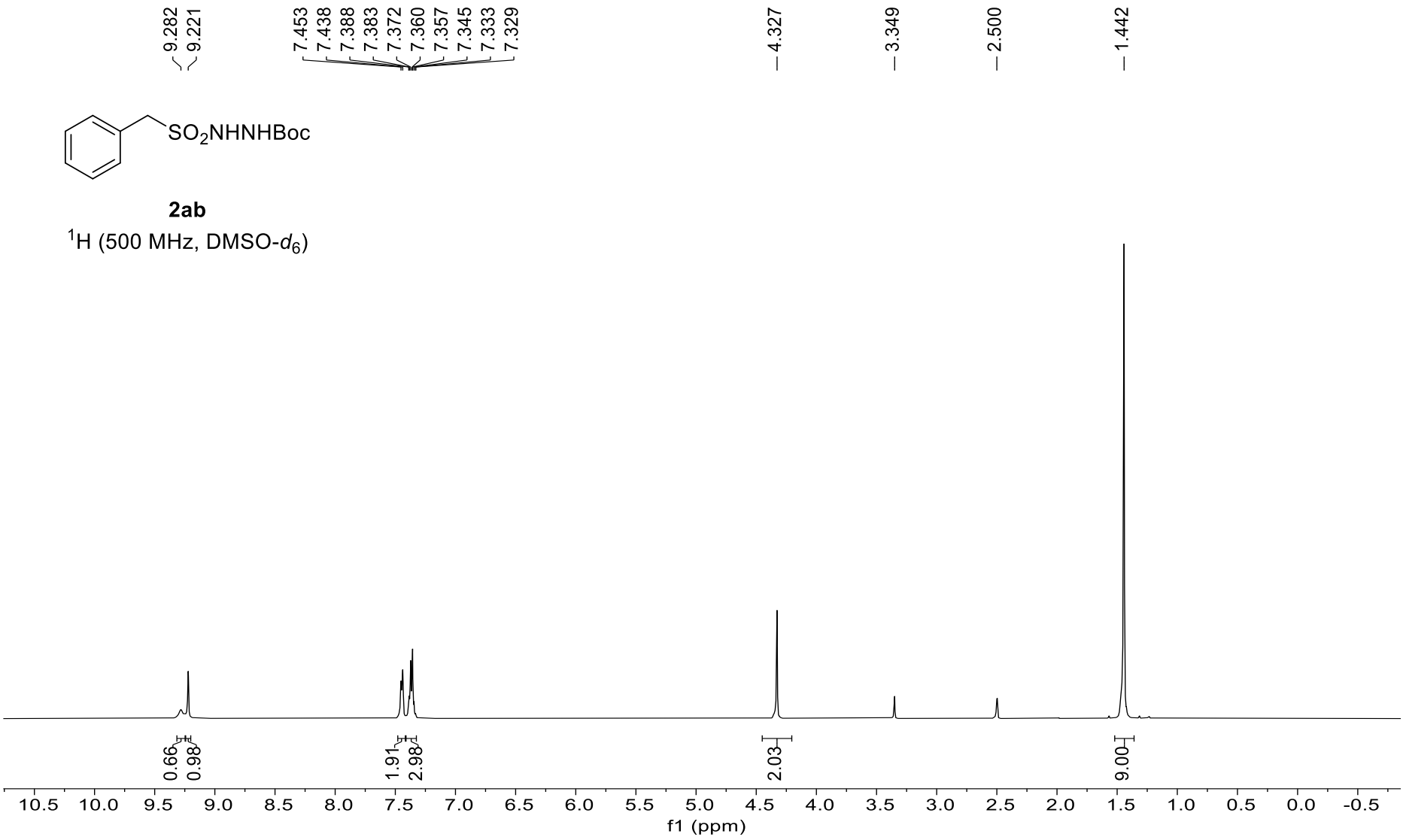
9. Copies of ^{1}H and ^{13}C NMR spectra

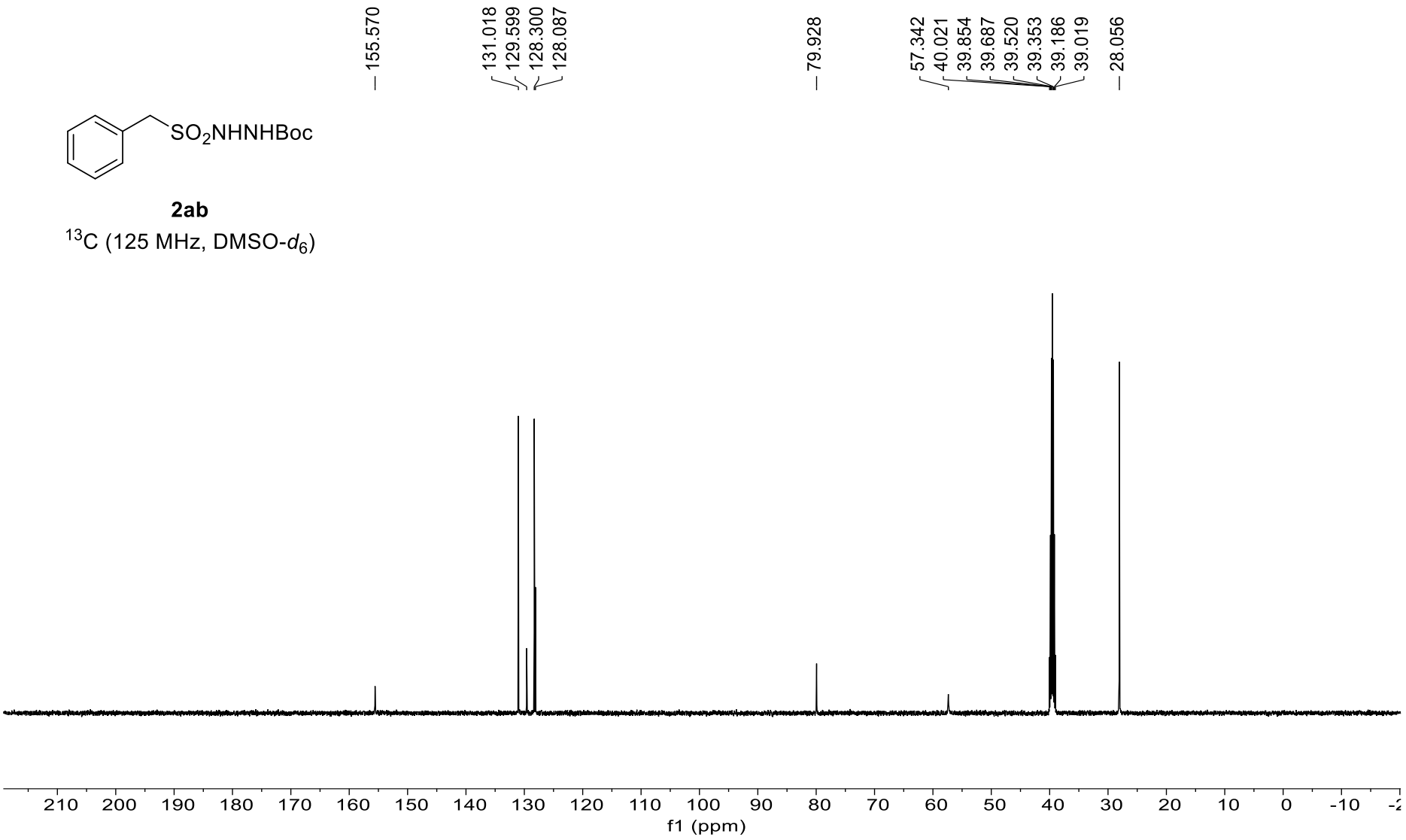

7.905
7.380
7.372
7.356
7.350
7.343
7.340
7.331
4.463
4.370
3.361
2.500
SO2NHNH2
2a
1H (500 MHz, DMSO-d6)
1.02
5.04
1.83
2.07
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

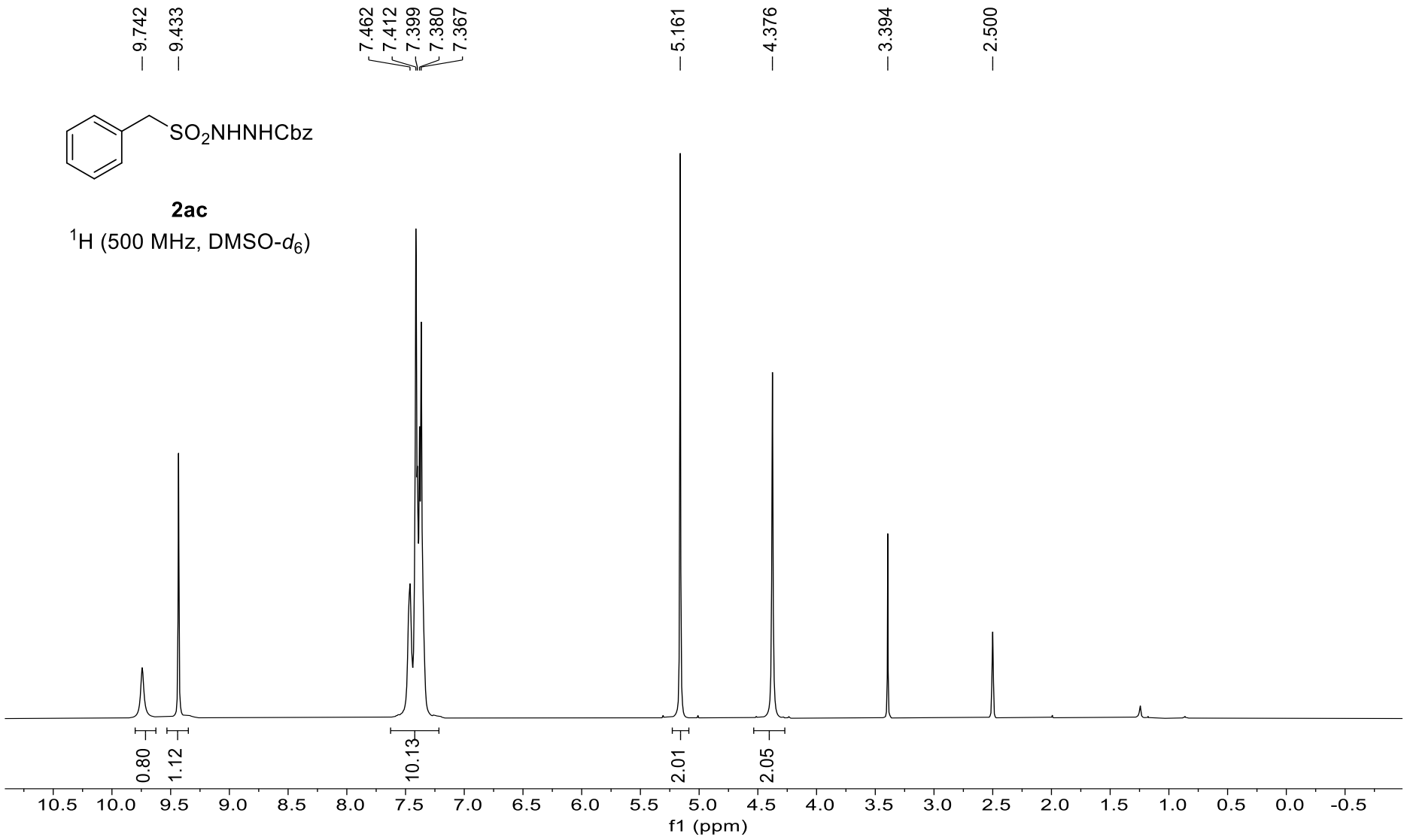


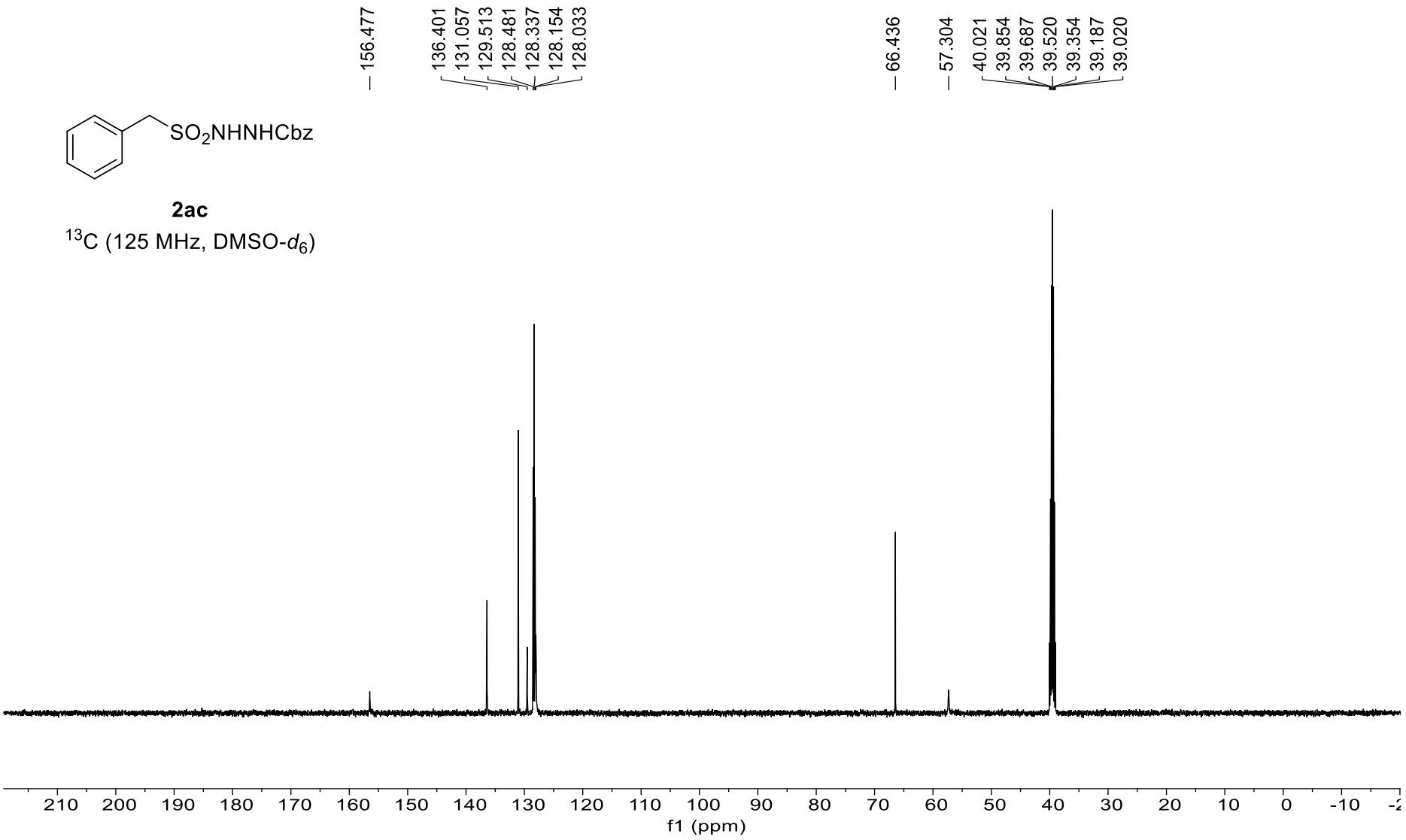
2a

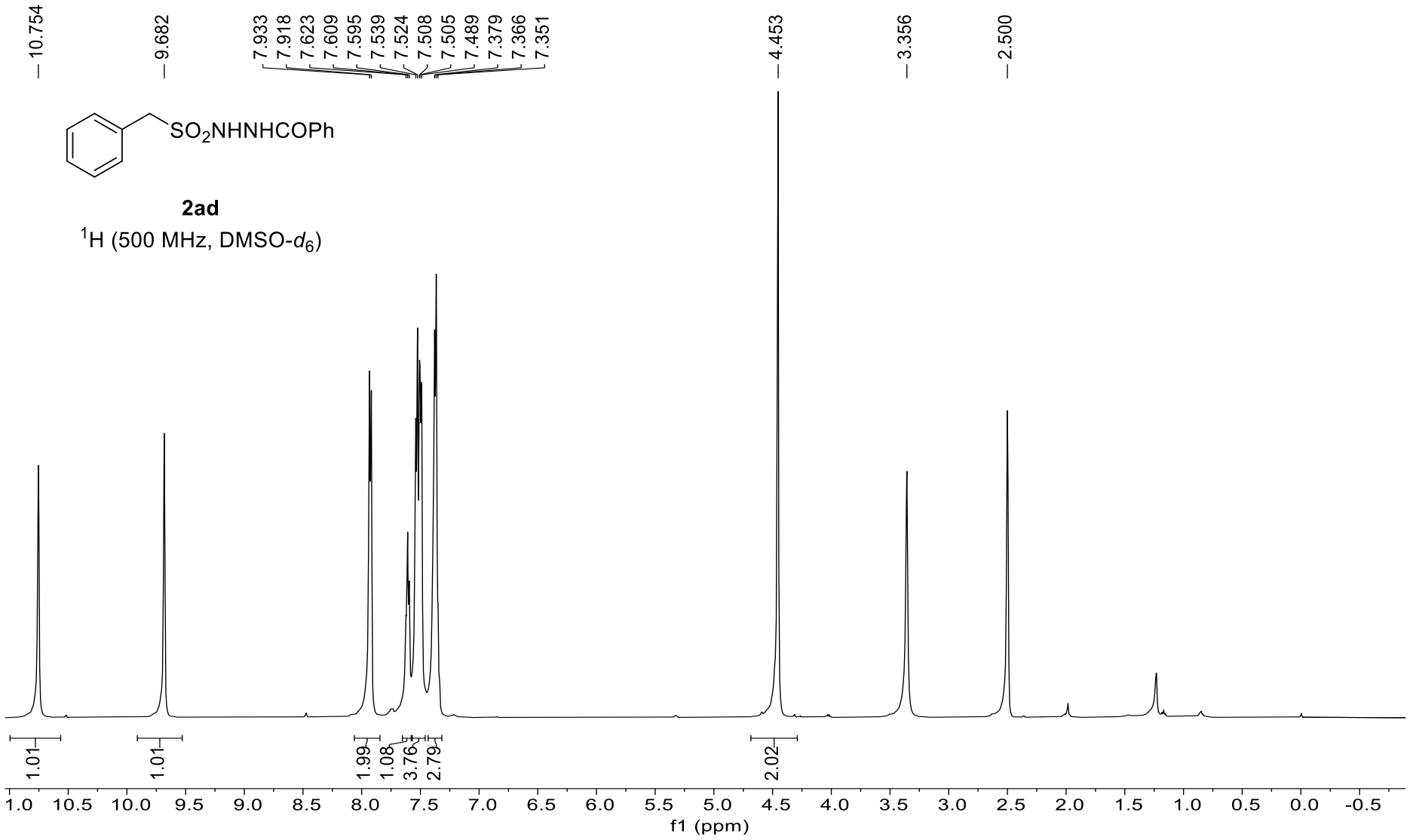
^{13}C (125 MHz, DMSO-d_6)

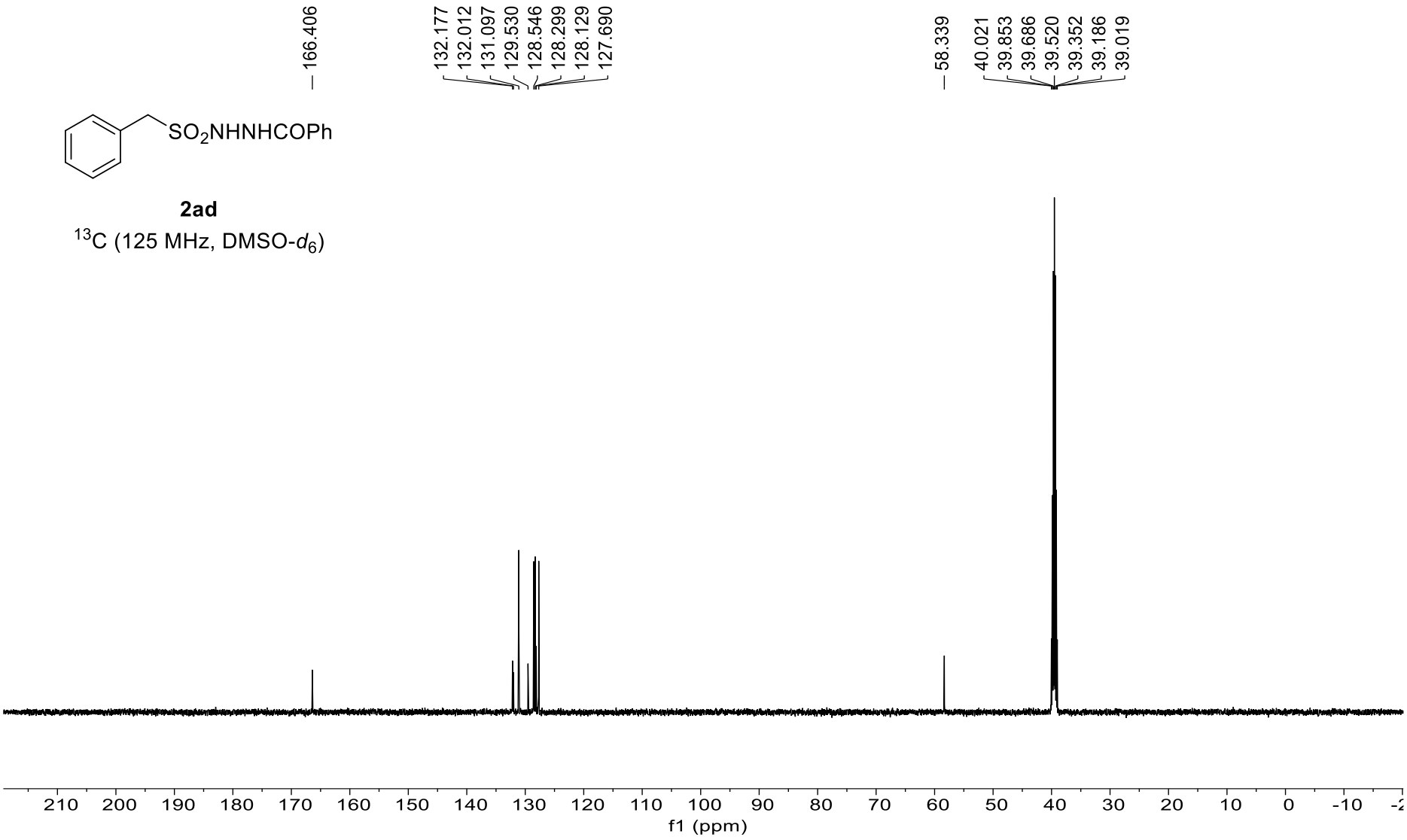

9.282
9.221
7.453
7.438
7.388
7.383
7.372
7.360
7.357
7.345
7.333
7.329
4.327
3.349
2.500
1.442
SO2NHNHBoc
2ab
1H (500 MHz, DMSO-d6)
0.66
0.98
1.91
2.98
2.03
9.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

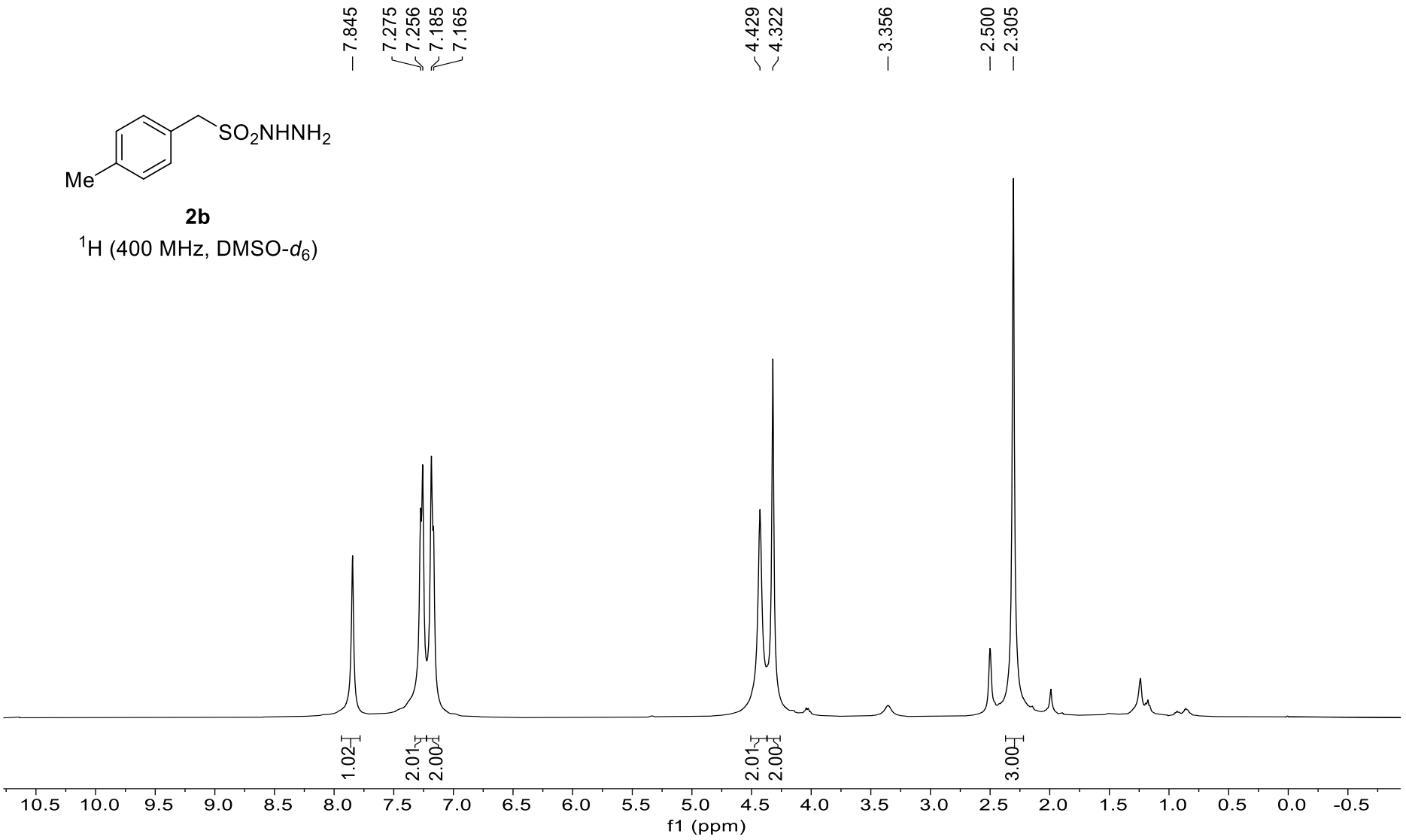

155.570
131.018
129.599
128.300
128.087
79.928
57.342
40.021
39.854
39.687
39.520
39.353
39.186
39.019
28.056
SO2NHNHBoc
2ab
13C (125 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

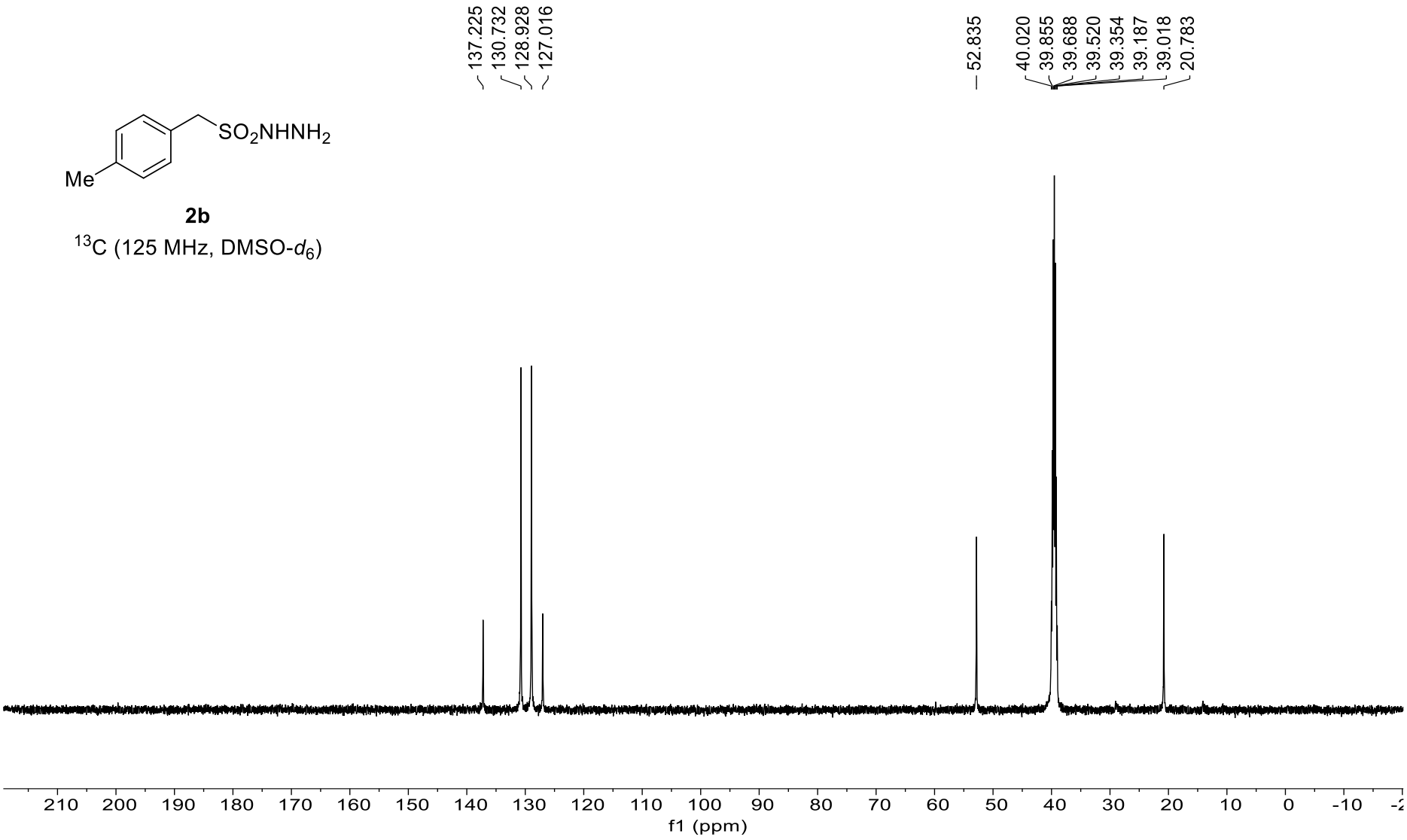

9.742
9.433
7.462
7.412
7.399
7.380
7.367
5.161
4.376
3.394
2.500
SO2NHNHCbz
2ac
1H (500 MHz, DMSO-d6)
0.80
1.12
10.13
2.01
2.05
f1 (ppm)

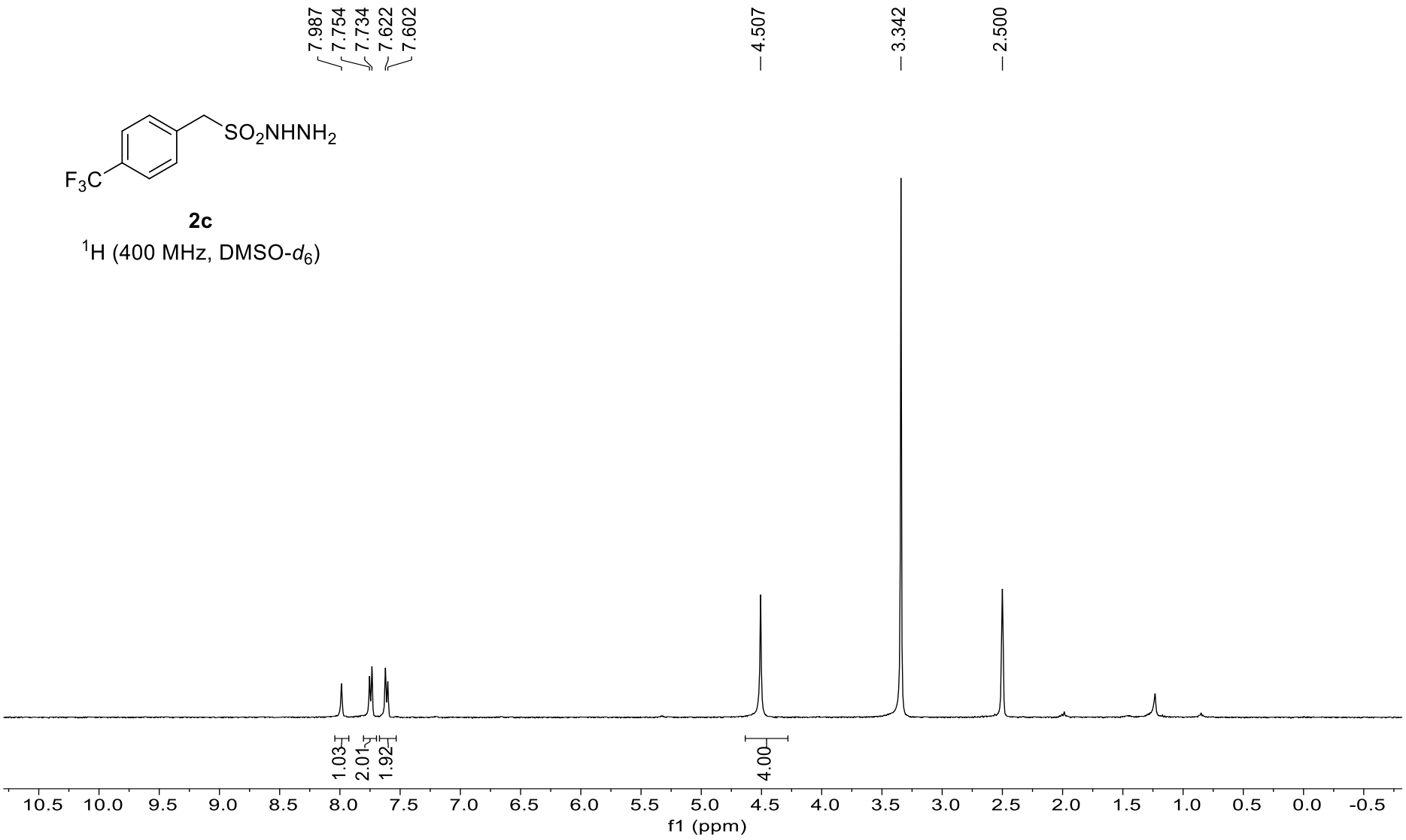

156.477
136.401
131.057
129.513
128.481
128.337
128.154
128.033
66.436
57.304
40.021
39.854
39.687
39.520
39.354
39.187
39.020
SO2NHNHCbz
2ac
13C (125 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

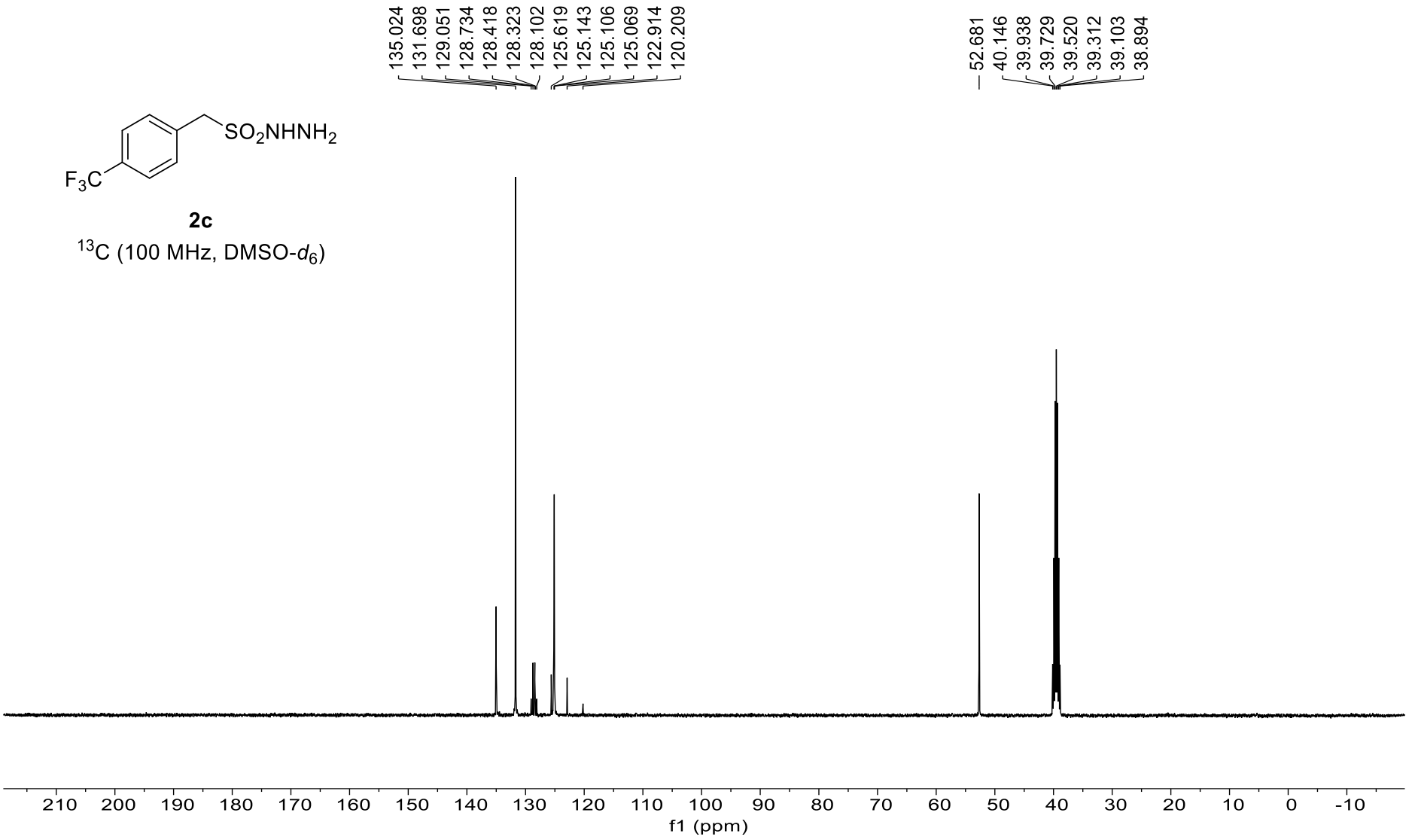

10.754
9.682
7.933
7.918
7.623
7.609
7.595
7.539
7.524
7.508
7.505
7.489
7.379
7.366
7.351
4.453
3.356
2.500
SO2NHNHCOPh
2ad
1H (500 MHz, DMSO-d6)
1.01
1.01
1.99
1.08
3.76
2.79
2.02
f1 (ppm)

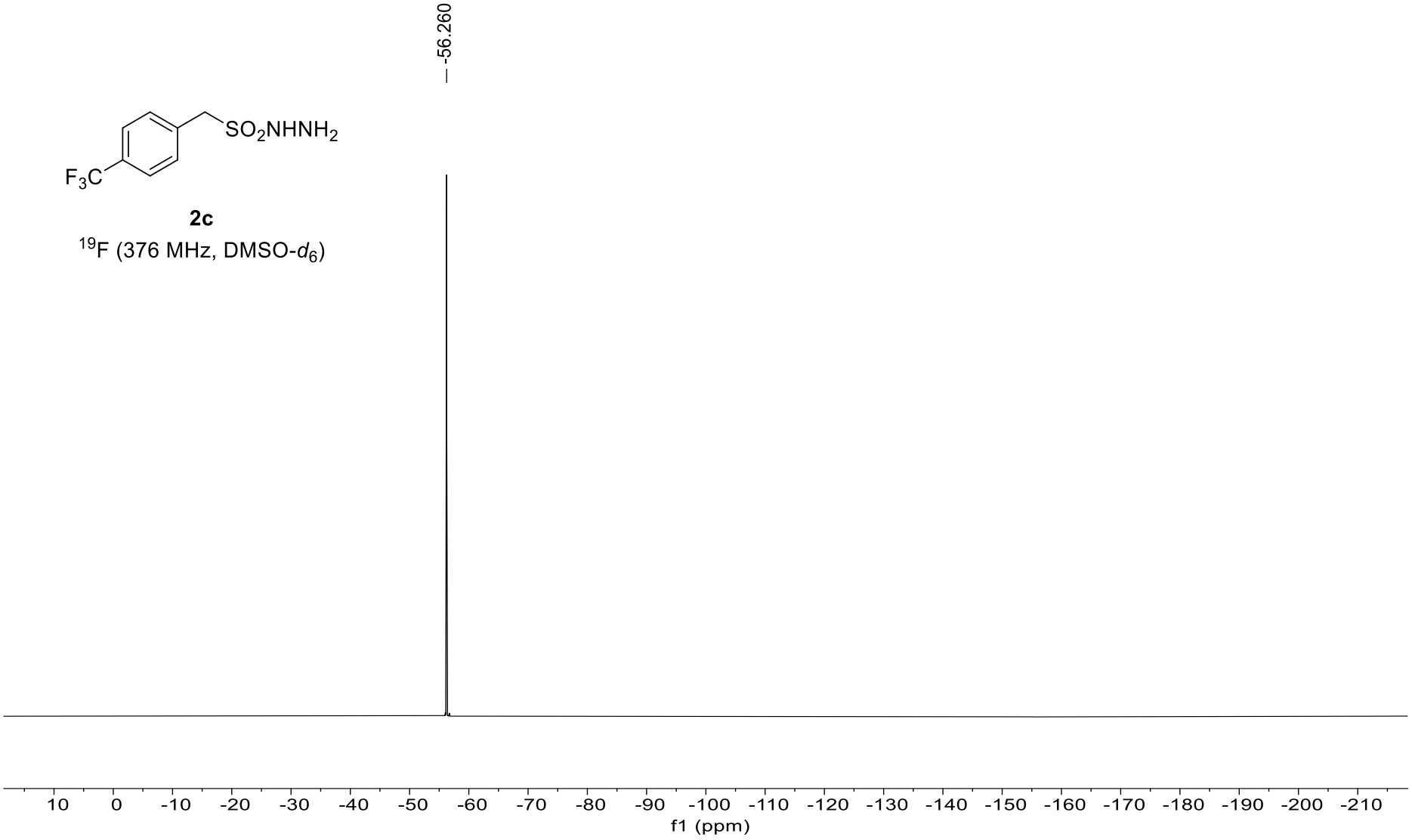

166.406
132.177
132.012
131.097
129.530
128.546
128.299
128.129
127.690
58.339
40.021
39.853
39.686
39.520
39.352
39.186
39.019
SO2NHNHCOPh
2ad
13C (125 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.845
7.275
7.256
7.185
7.165
4.429
4.322
3.356
2.500
2.305
SO2NHNH2
Me
2b
1H (400 MHz, DMSO-d6)
1.02
2.01
2.00
2.01
2.00
3.00
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
f1 (ppm)


137.225
130.732
128.928
127.016
52.835
40.020
39.855
39.688
39.520
39.354
39.187
39.018
20.783
SO2NHNH2
Me
2b
13C (125 MHz, DMSO-d6)
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)

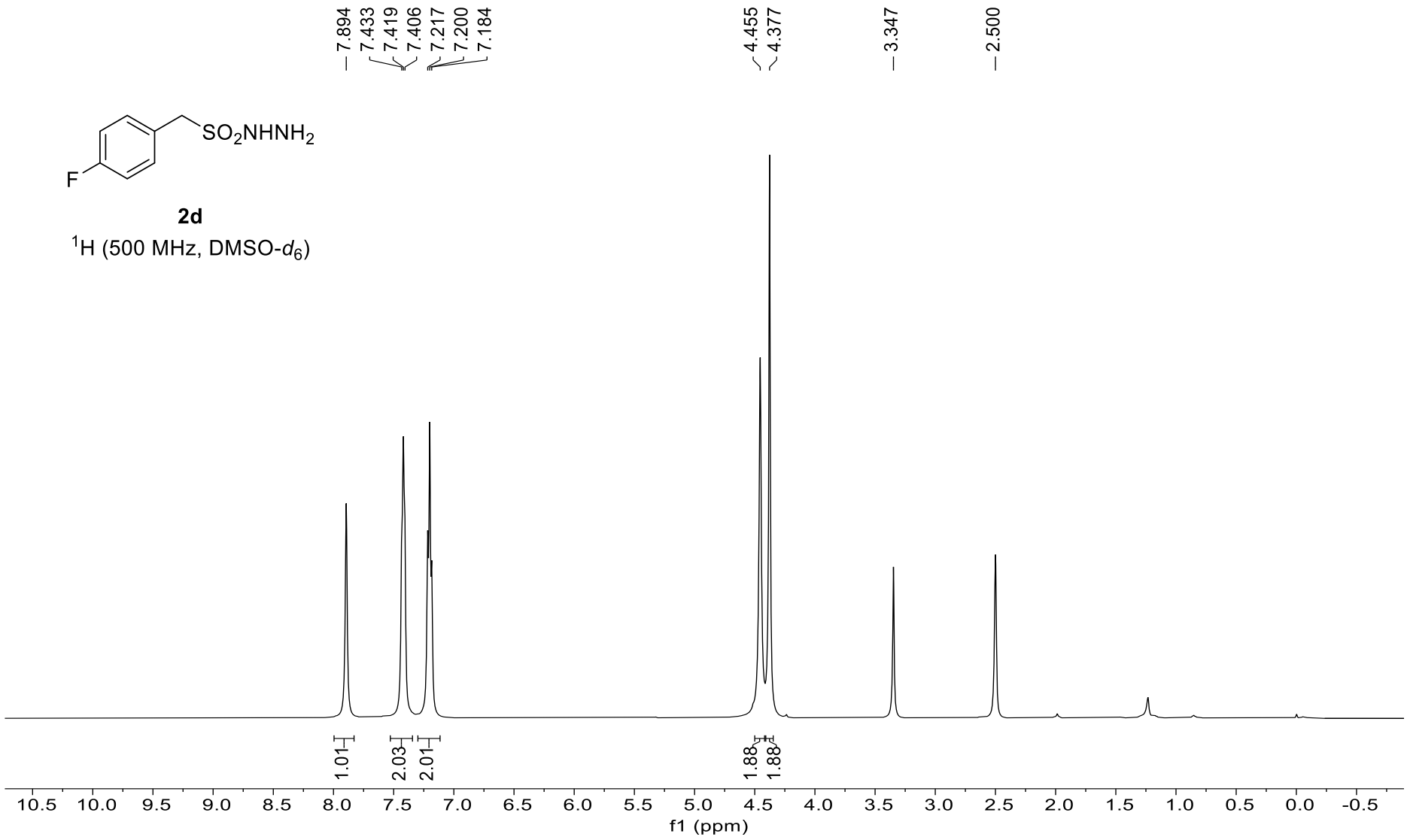

7.987
7.754
7.734
7.622
7.602
4.507
3.342
2.500
SO2NHNH2
F3C
2c
1H (400 MHz, DMSO-d6)
1.03
2.01
1.92
4.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

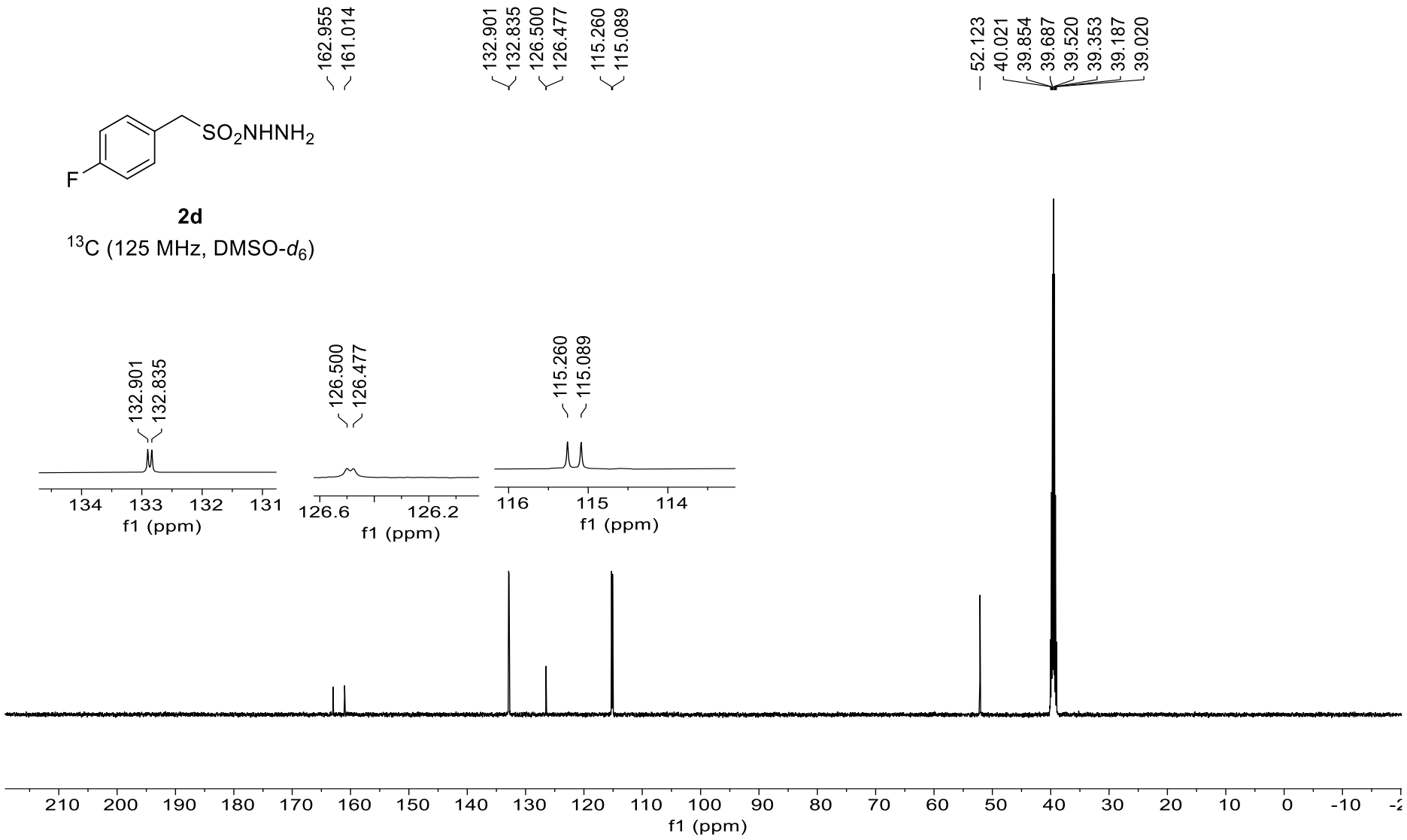

135.024
131.698
129.051
128.734
128.418
128.323
128.102
125.619
125.143
125.106
125.069
122.914
120.209
52.681
40.146
39.938
39.729
39.520
39.312
39.103
38.894
SO2NHNH2
F3C
2c
13C (100 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

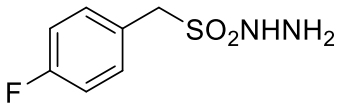


2c

^{19}F (376 MHz, DMSO-d_6)

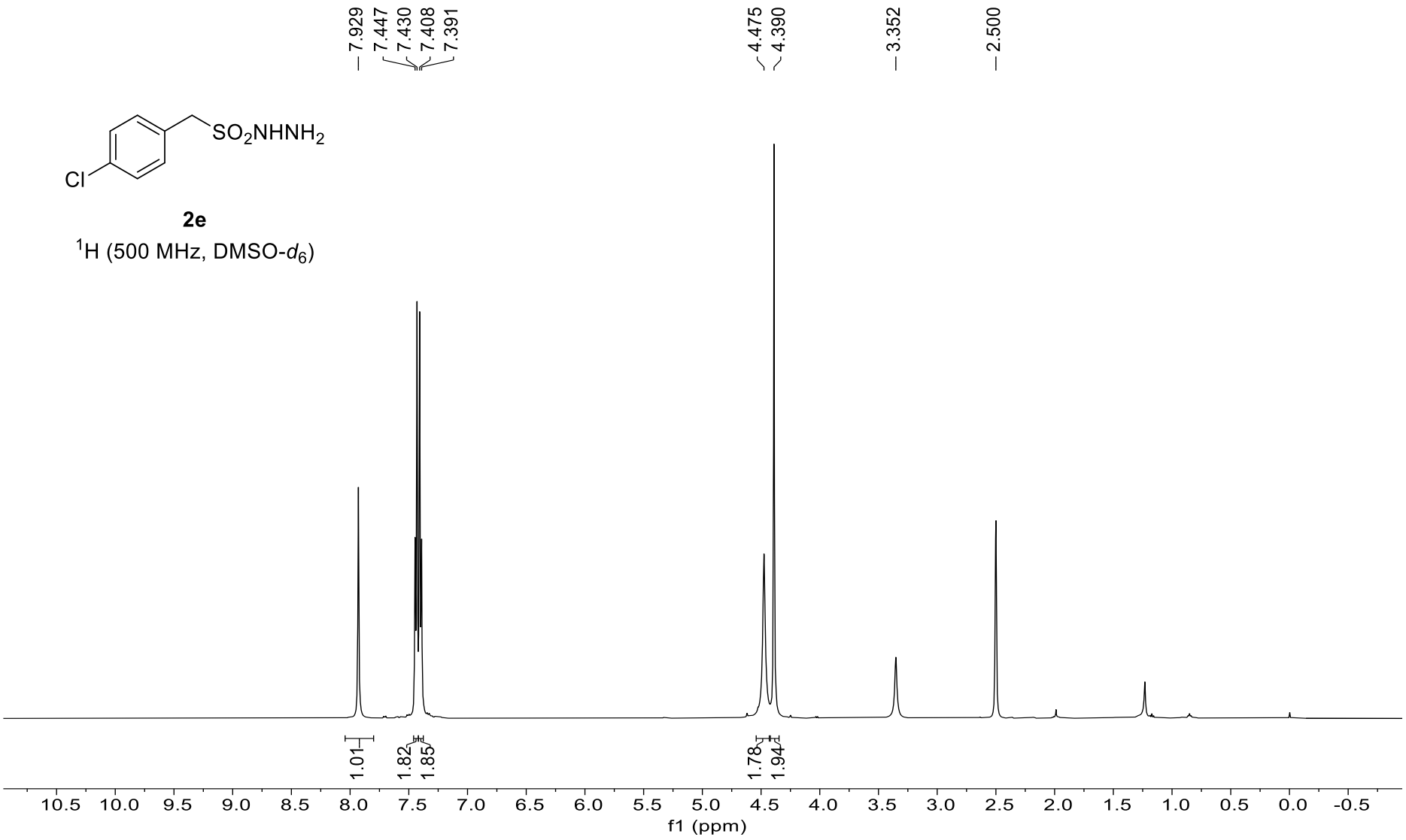

7.894
7.433
7.419
7.406
7.217
7.200
7.184
4.455
4.377
3.347
2.500
SO2NHNH2
F
2d
1H (500 MHz, DMSO-d6)
1.01
2.03
2.01
1.88
1.88
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

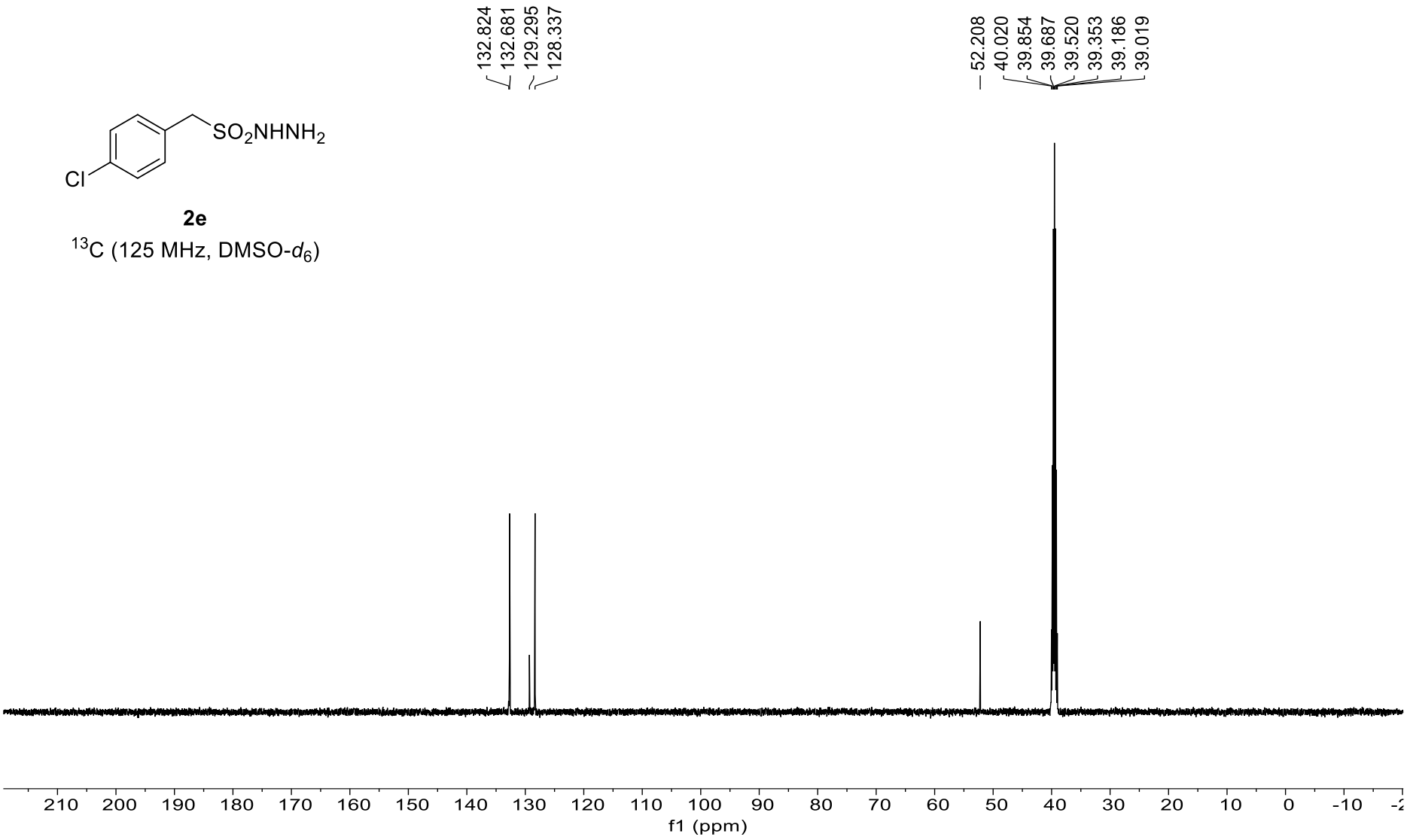

162.955
161.014
132.901
132.835
126.500
126.477
115.260
115.089
52.123
40.021
39.854
39.687
39.520
39.353
39.187
39.020
SO2NHNH2
F
2d
13C (125 MHz, DMSO-d6)
f1 (ppm)

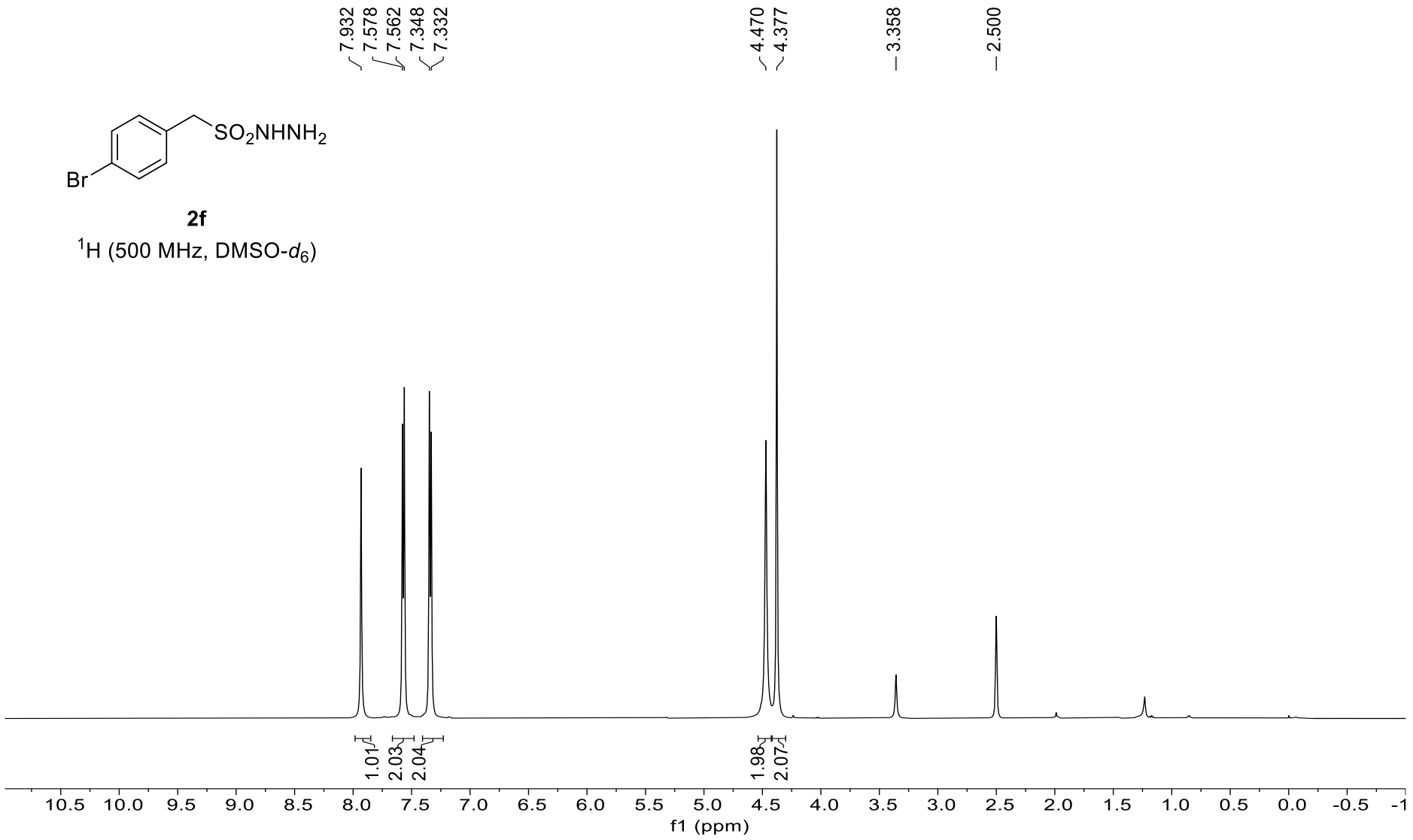


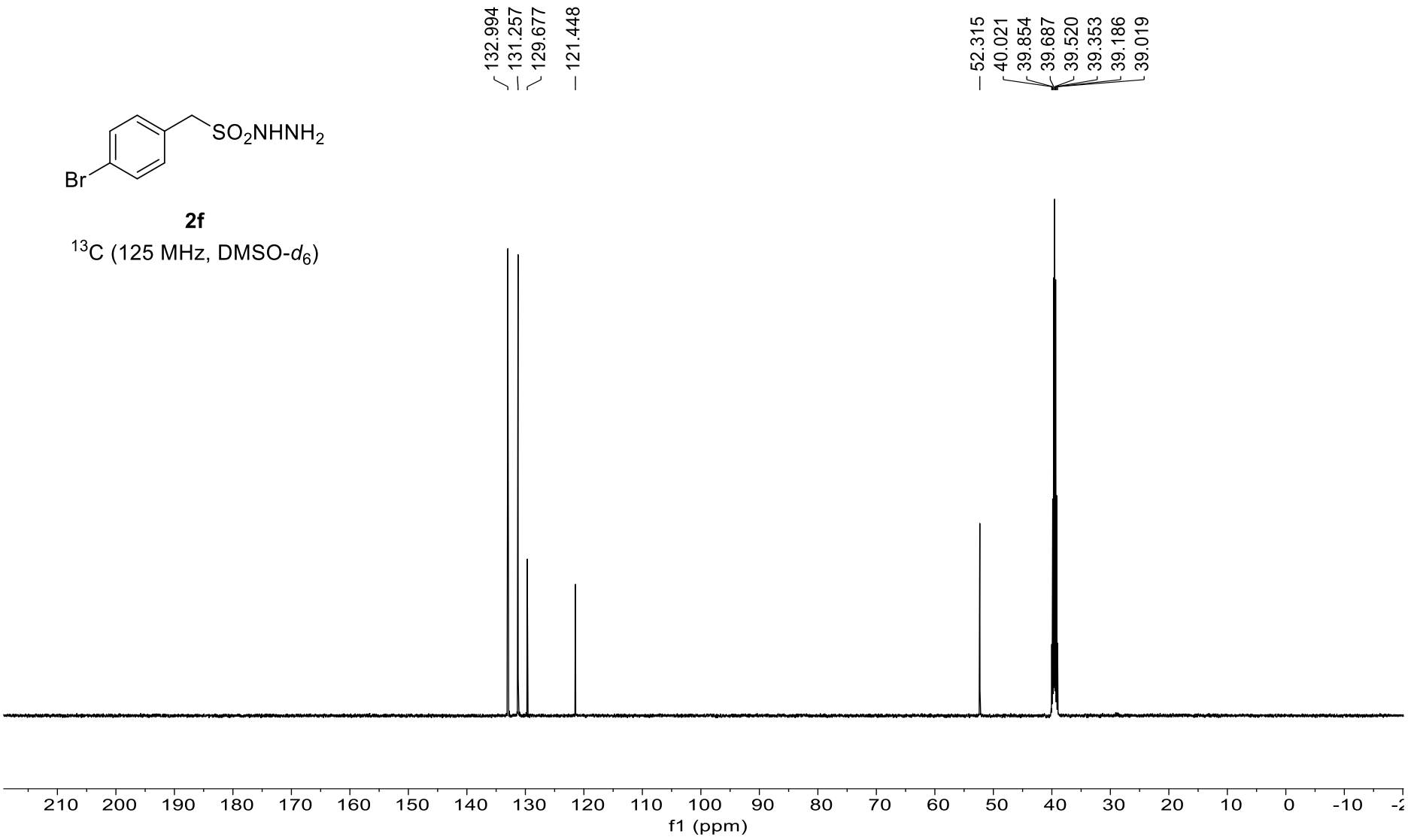
2d

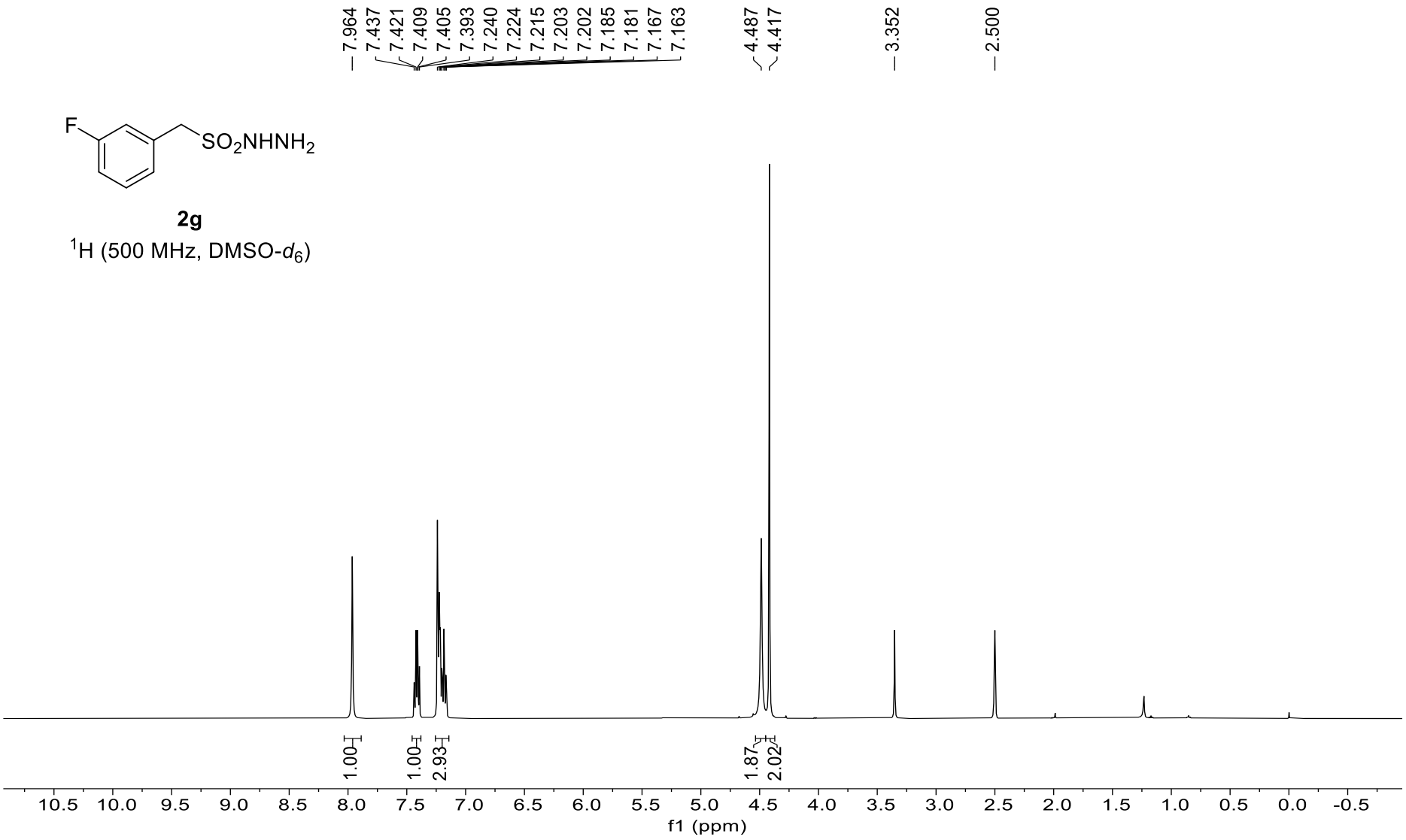
^{19}F (471 MHz, DMSO-d_6)

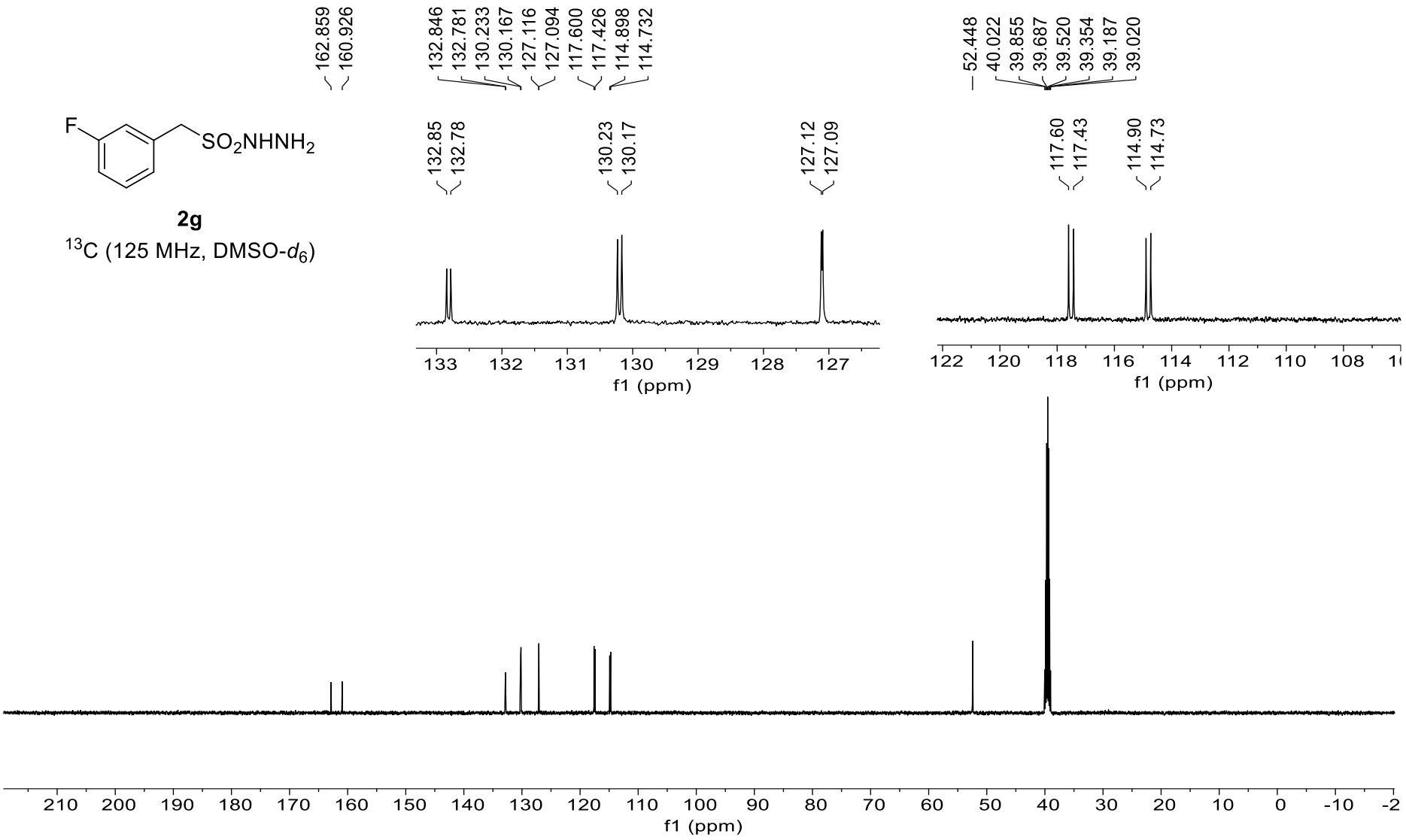

7.929
7.447
7.430
7.408
7.391
4.475
4.390
3.352
2.500
SO2NHNH2
Cl
2e
1H (500 MHz, DMSO-d6)
1.01
1.82
1.85
1.78
1.94
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

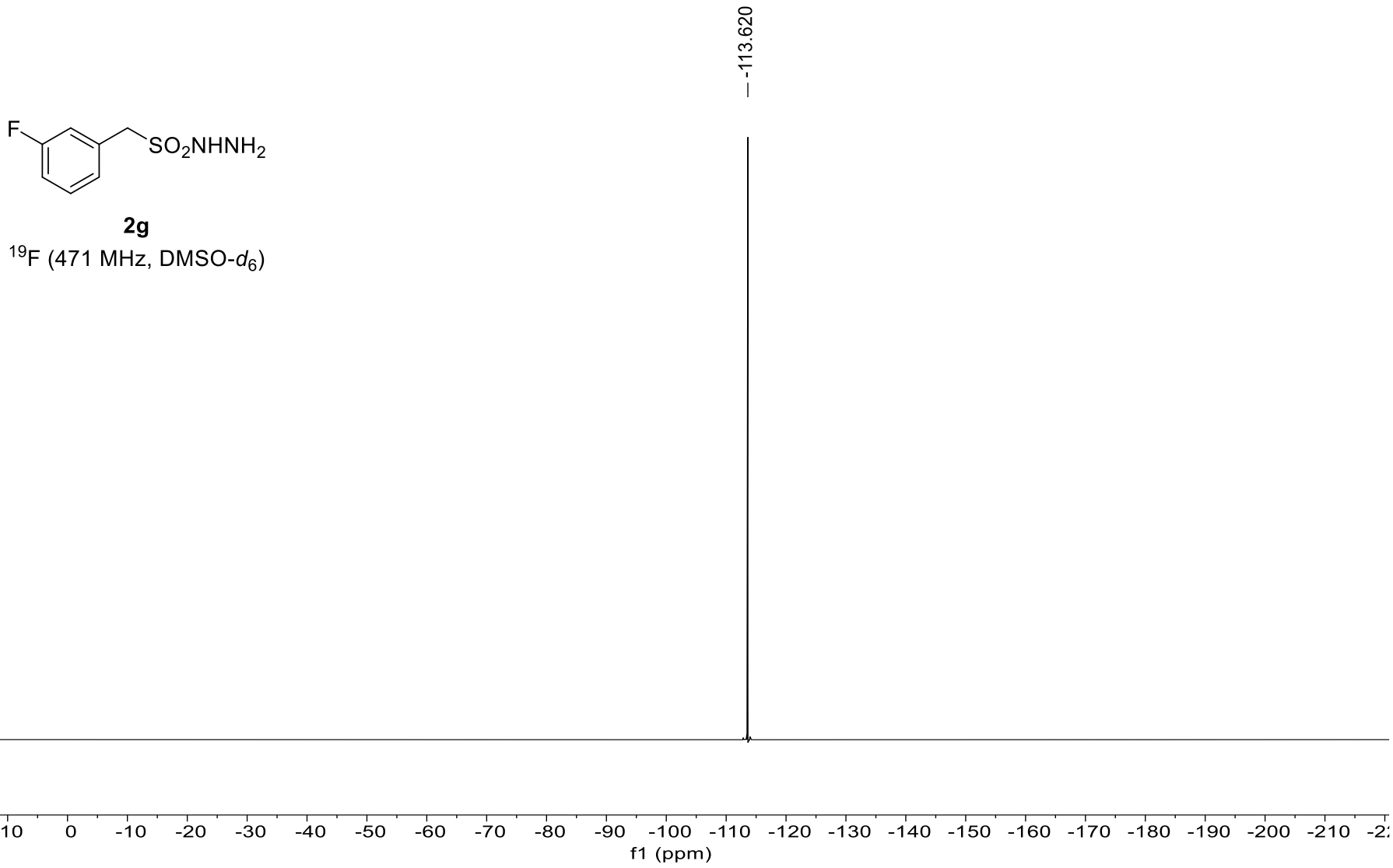

132.824
132.681
129.295
128.337
52.208
40.020
39.854
39.687
39.520
39.353
39.186
39.019
SO2NHNH2
Cl
2e
13C (125 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.932
7.578
7.562
7.348
7.332
4.470
4.377
3.358
2.500
SO2NHNH2
Br
2f
1H (500 MHz, DMSO-d6)
1.01
2.03
2.04
1.98
2.07
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
f1 (ppm)


132.994
131.257
129.677
121.448
52.315
40.021
39.854
39.687
39.520
39.353
39.186
39.019
SO2NHNH2
Br
2f
13C (125 MHz, DMSO-d6)
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)

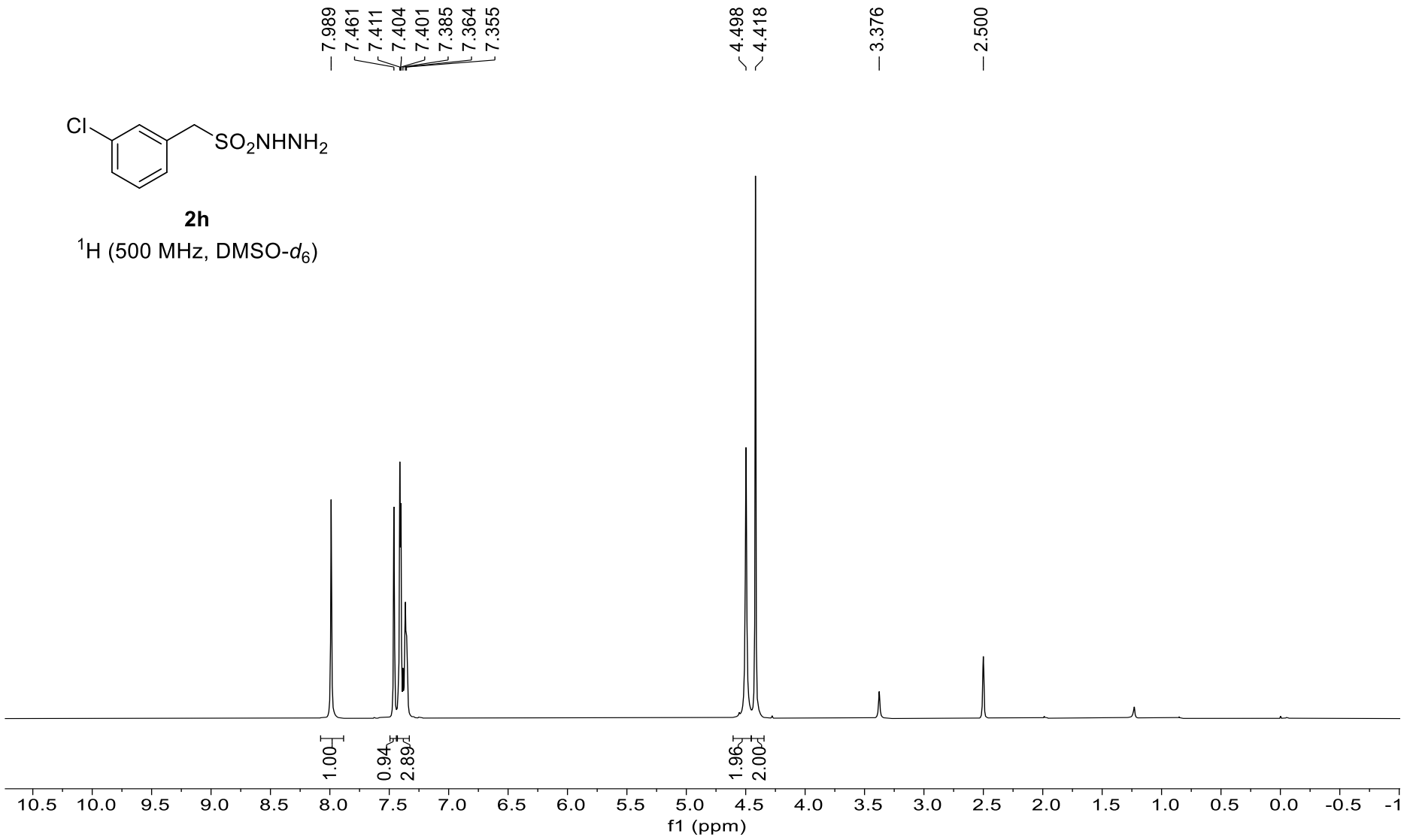

7.964
7.437
7.421
7.409
7.405
7.393
7.240
7.224
7.215
7.203
7.202
7.185
7.181
7.167
7.163
4.487
4.417
3.352
2.500
F
SO2NHNH2
2g
1H (500 MHz, DMSO-d6)
1.00
1.00
2.93
1.87
2.02
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

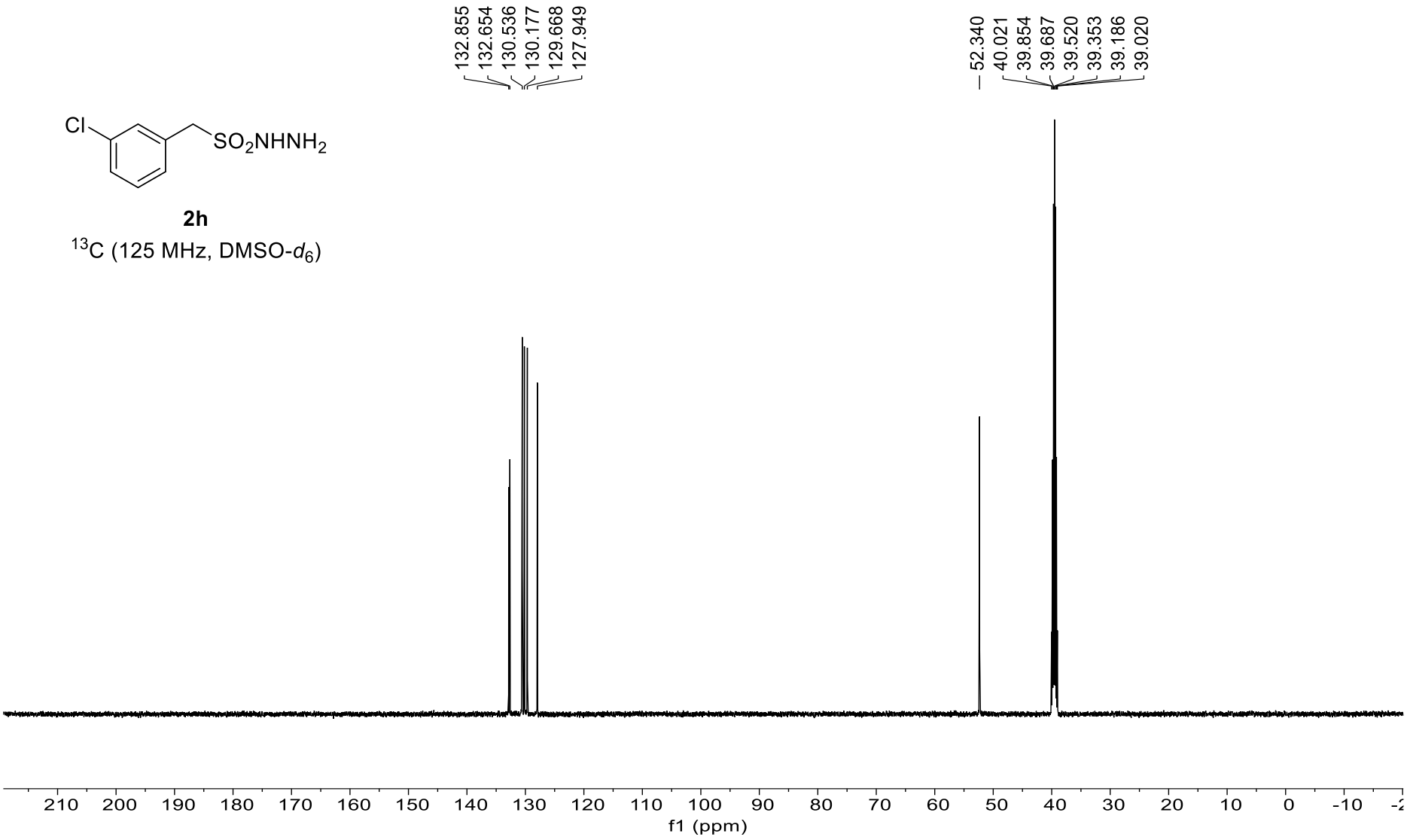

F
SO2NHNH2
2g
13C (125 MHz, DMSO-d6)
162.859
160.926
132.846
132.781
130.233
130.167
127.116
127.094
117.600
117.426
114.898
114.732
52.448
40.022
39.855
39.687
39.520
39.354
39.187
39.020
132.85
132.78
130.23
130.17
127.12
127.09
117.60
117.43
114.90
114.73
f1 (ppm)
f1 (ppm)
f1 (ppm)

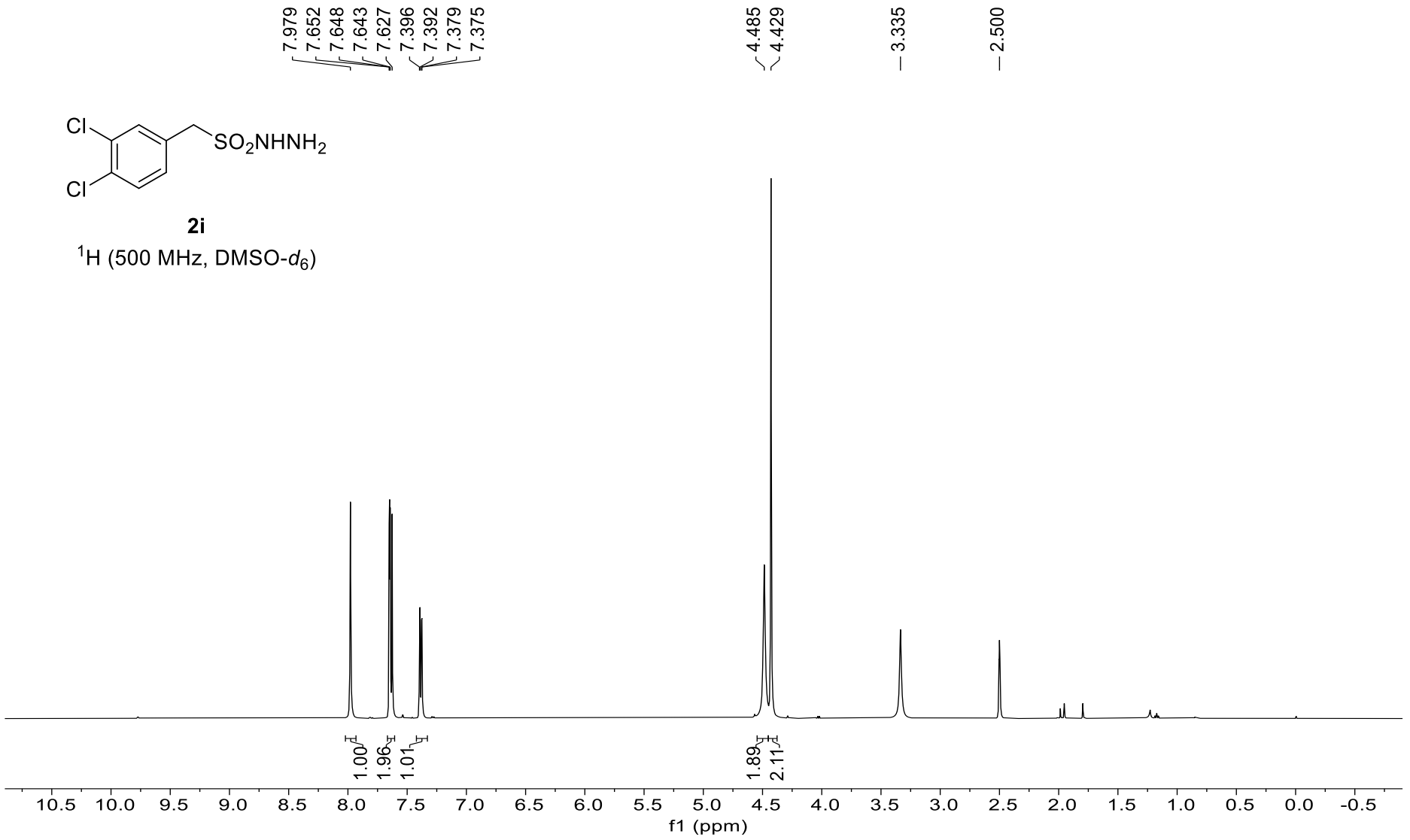


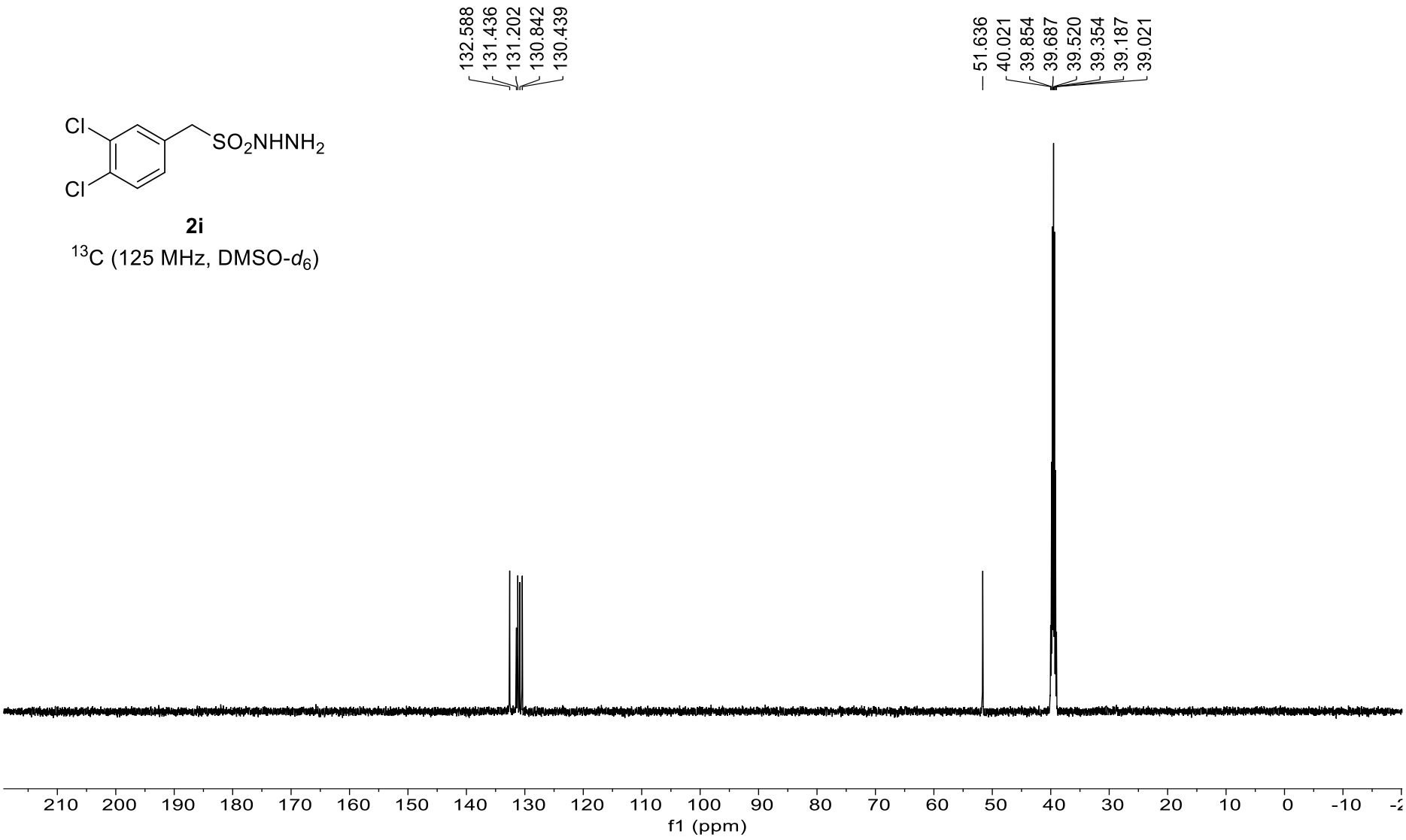
2g

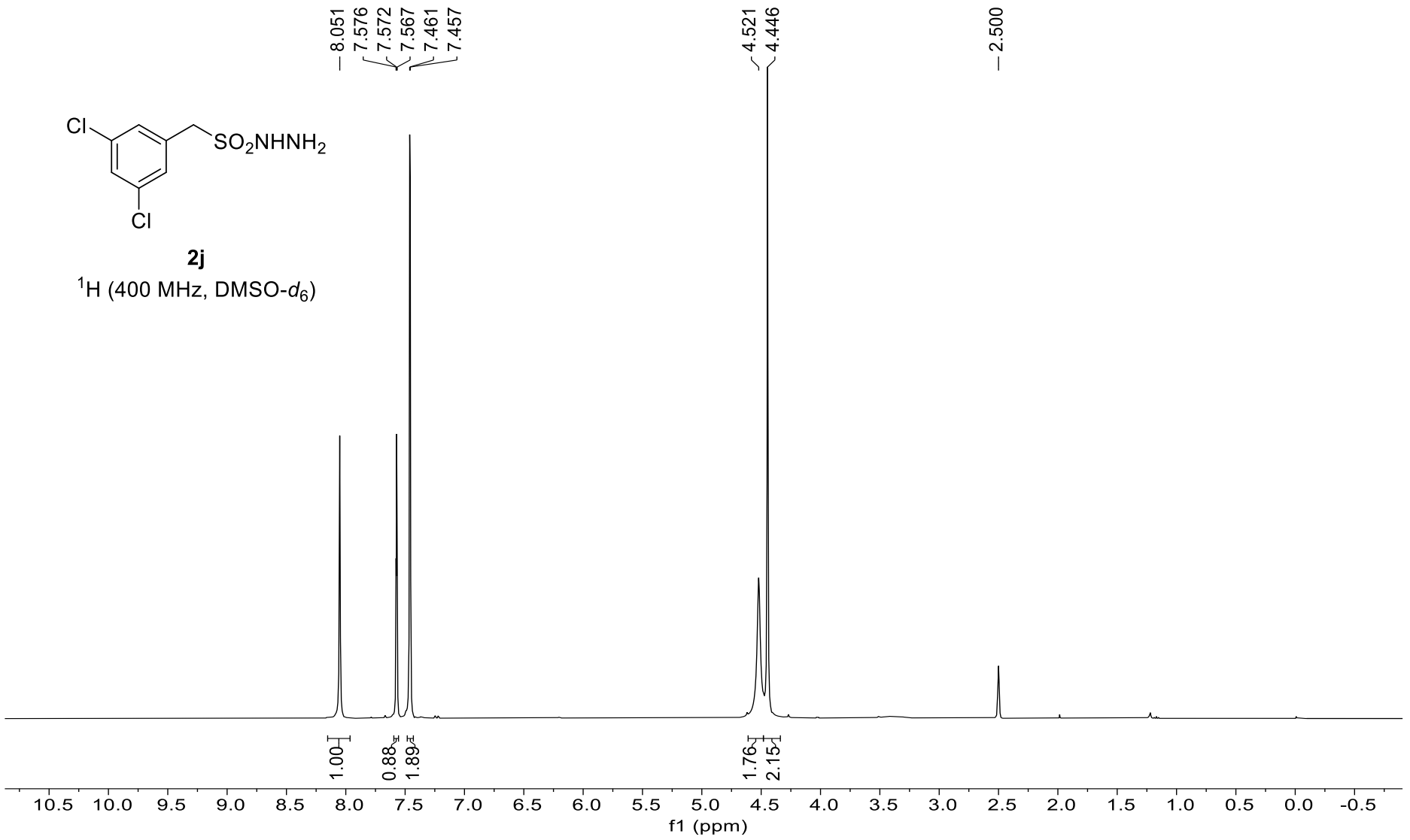
^{19}F (471 MHz, DMSO-d_6)

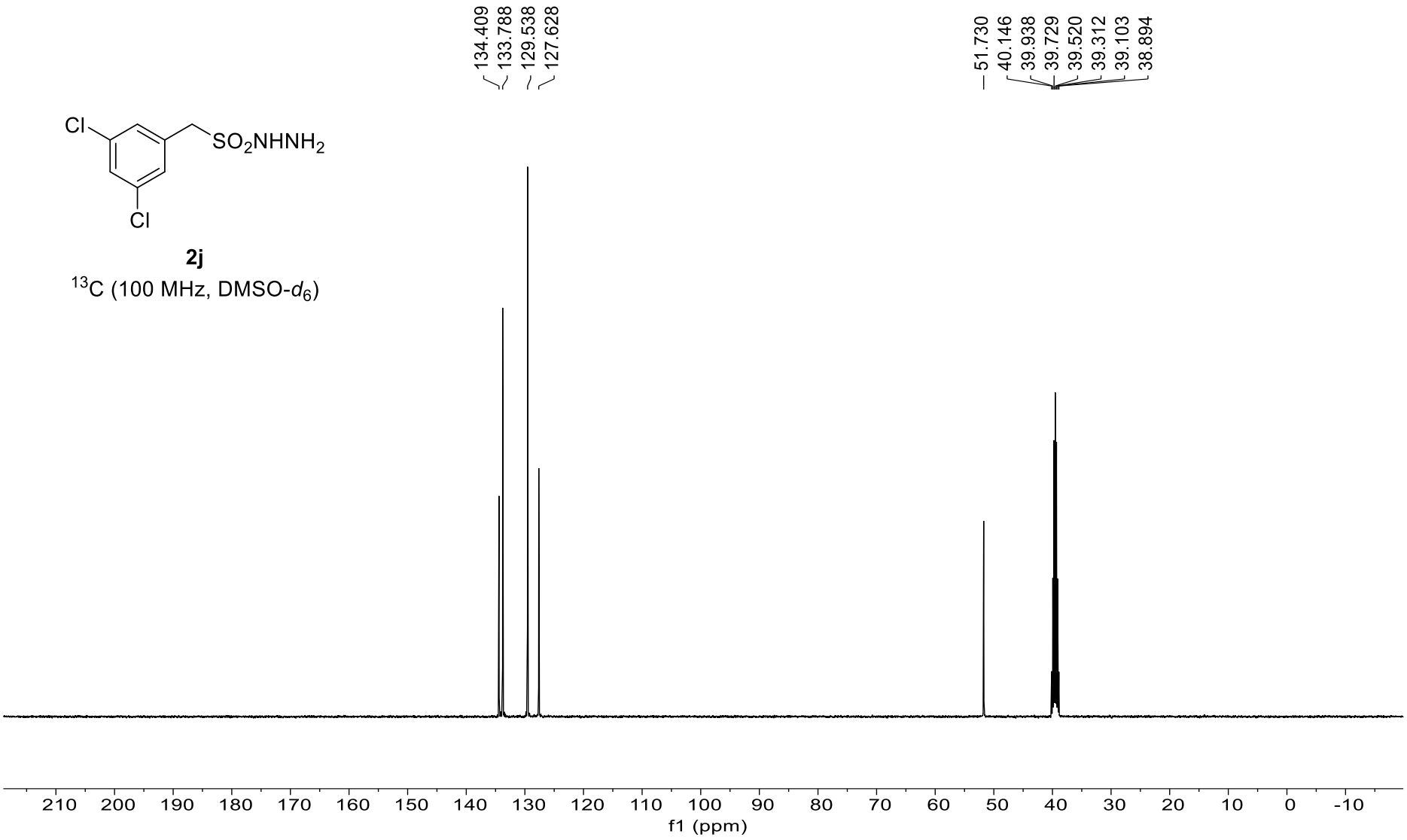

7.989
7.461
7.411
7.404
7.401
7.385
7.364
7.355
4.498
4.418
3.376
2.500
Cl
SO2NHNH2
2h
1H (500 MHz, DMSO-d6)
1.00
0.94
2.89
1.96
2.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

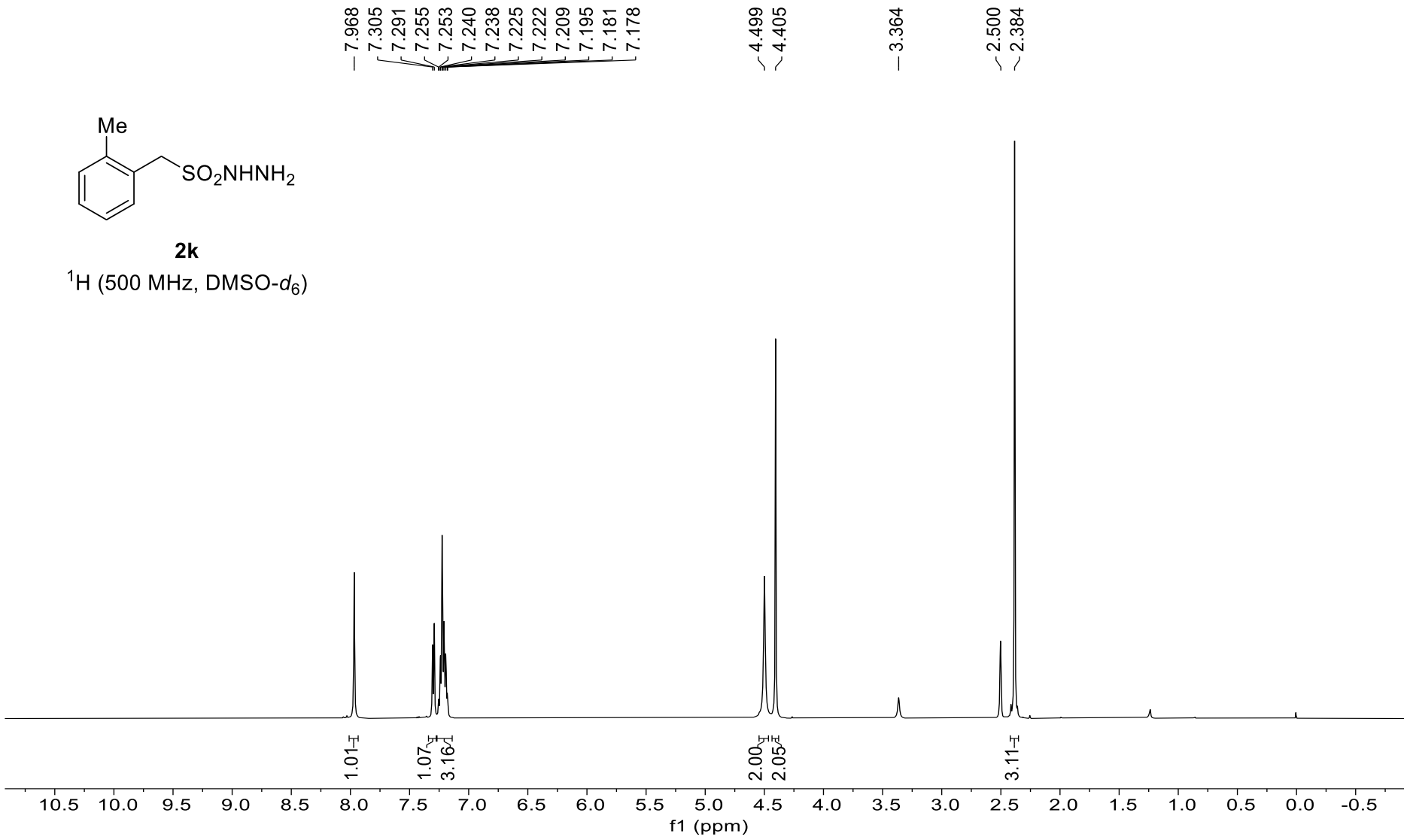

132.855
132.654
130.536
130.177
129.668
127.949
52.340
40.021
39.854
39.687
39.520
39.353
39.186
39.020
Cl
SO2NHNH2
2h
13C (125 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

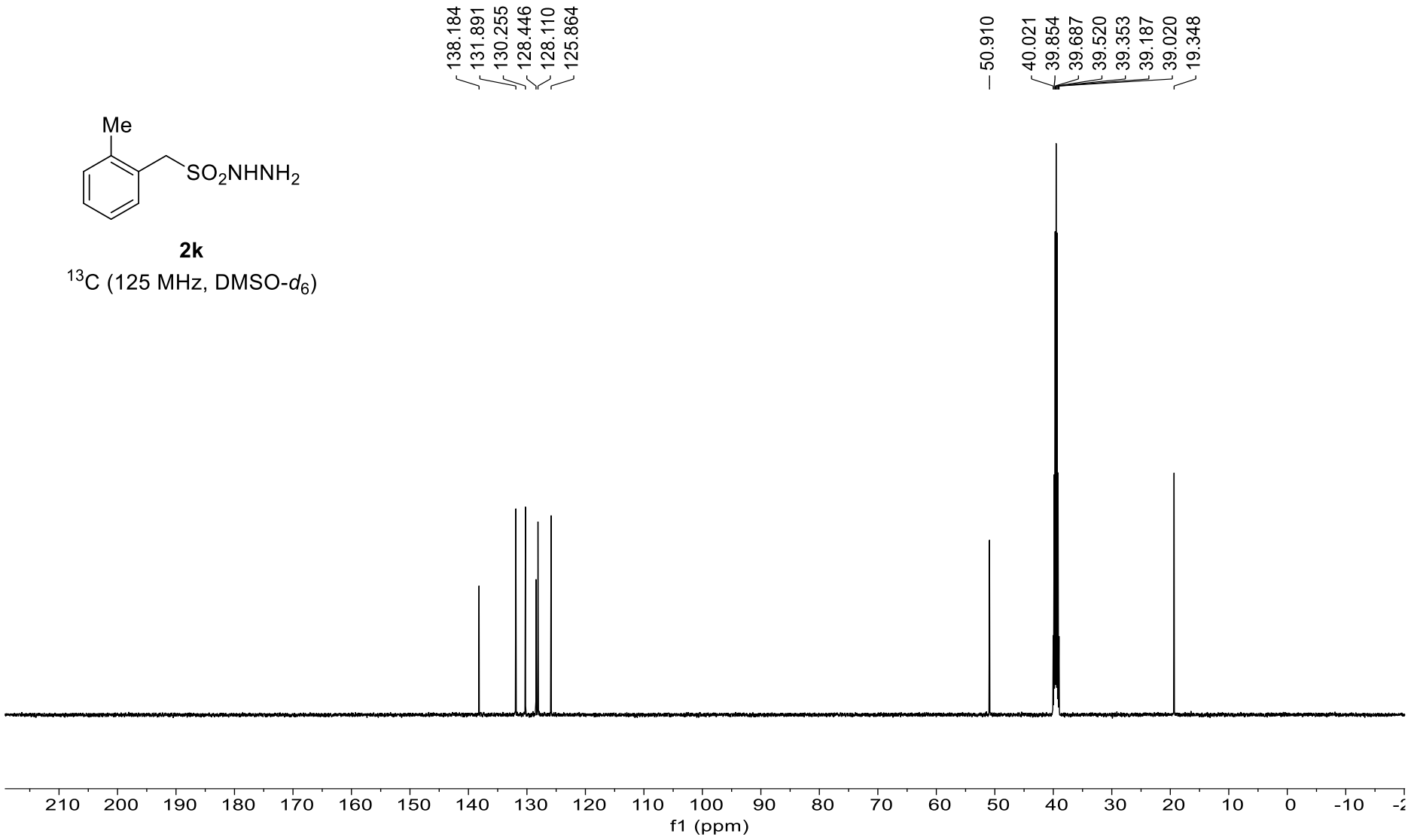

7.979
7.652
7.648
7.643
7.627
7.396
7.392
7.379
7.375
4.485
4.429
3.335
2.500
Cl
Cl
SO2NHNH2
2i
1H (500 MHz, DMSO-d6)
1.00
1.96
1.01
1.89
2.11
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

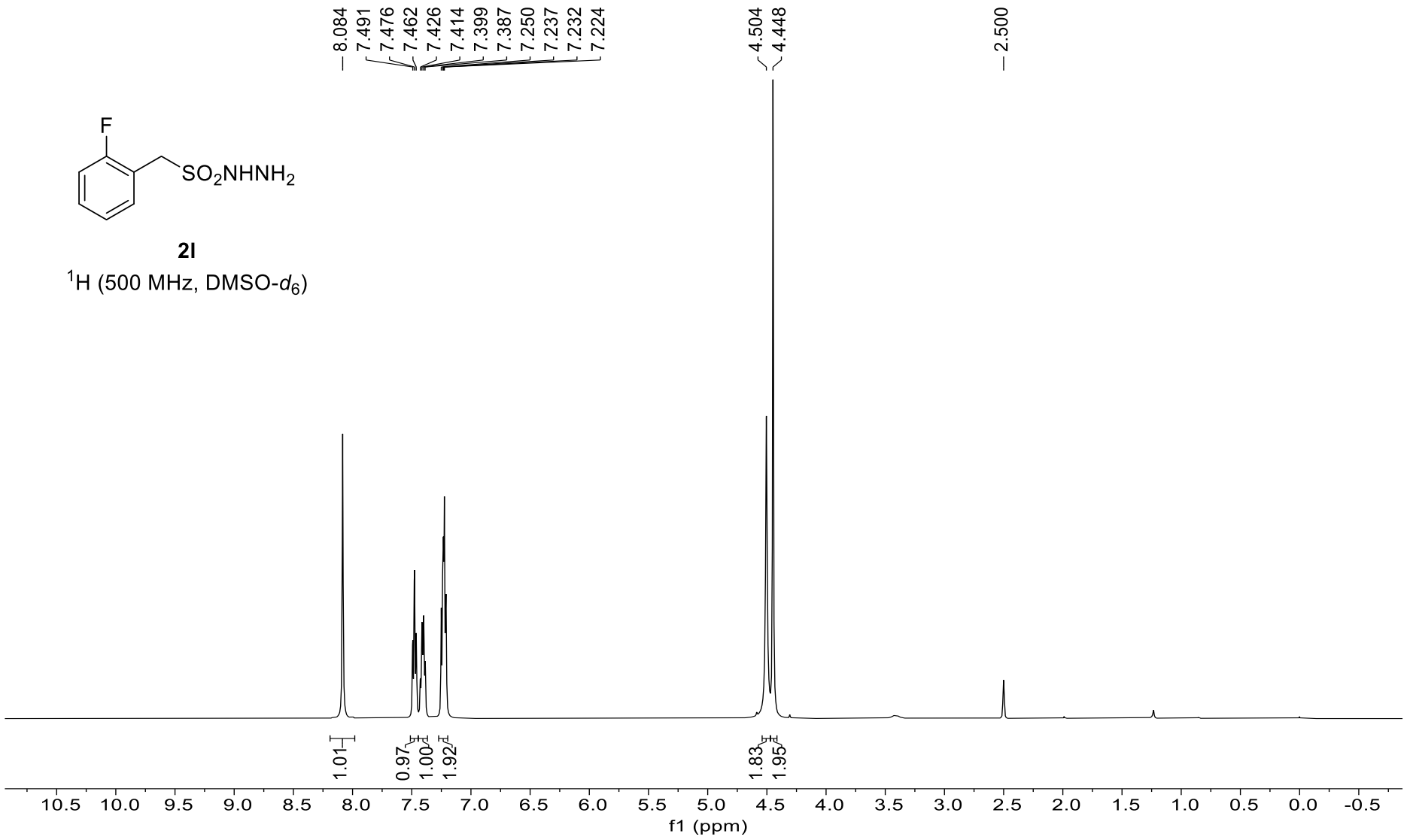

132.588
131.436
131.202
130.842
130.439
51.636
40.021
39.854
39.687
39.520
39.354
39.187
39.021
Cl
Cl
SO2NHNH2
2i
13C (125 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

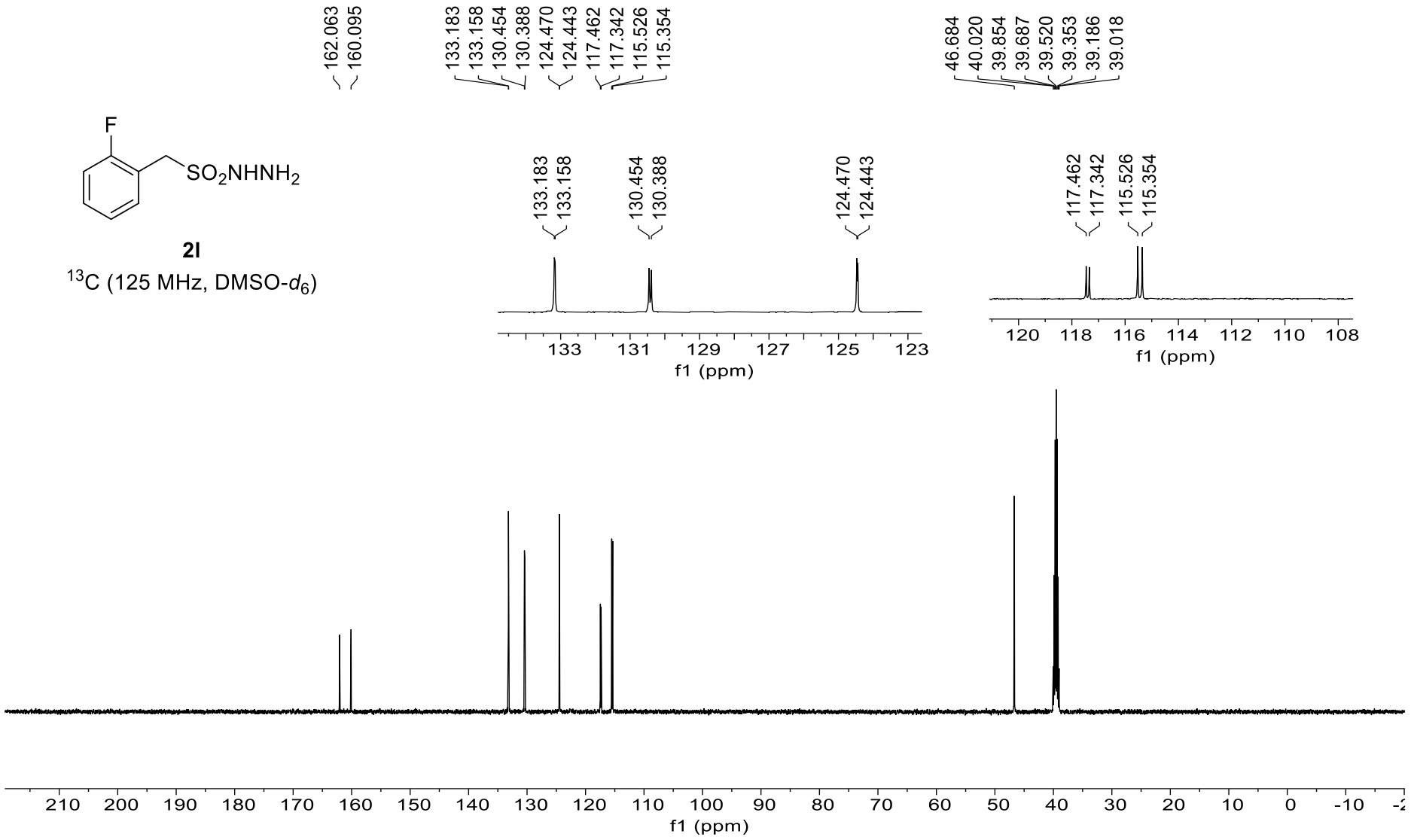

8.051
7.576
7.572
7.567
7.461
7.457
4.521
4.446
2.500
Cl
SO2NHNH2
Cl
2j
1H (400 MHz, DMSO-d6)
1.00
0.88
1.89
1.76
2.15
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

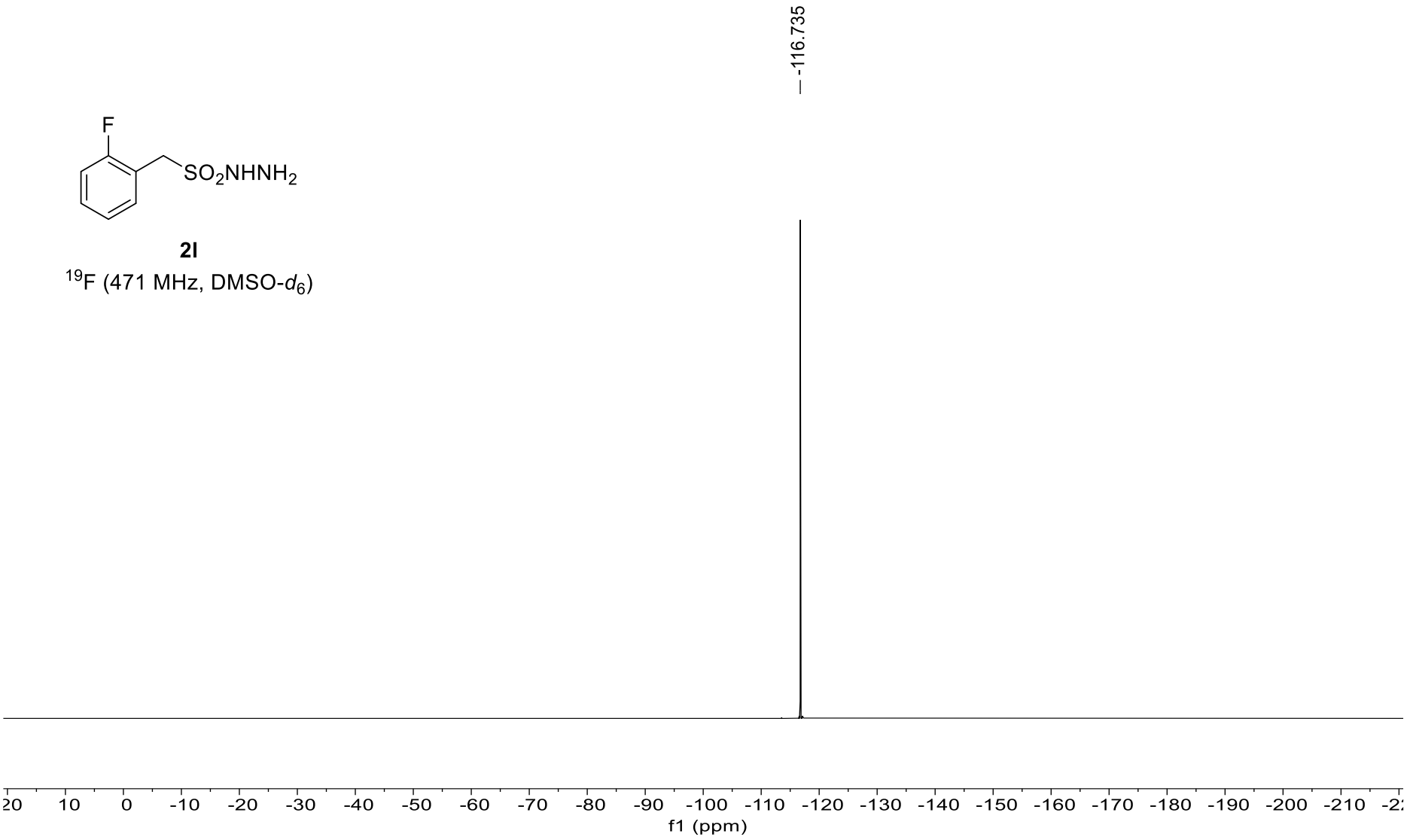

134.409
133.788
129.538
127.628
51.730
40.146
39.938
39.729
39.520
39.312
39.103
38.894
Cl
SO2NHNH2
Cl
2j
13C (100 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.968
7.305
7.291
7.255
7.253
7.240
7.238
7.225
7.222
7.209
7.195
7.181
7.178
4.499
4.405
3.364
2.500
2.384
Me
SO2NHNH2
2k
1H (500 MHz, DMSO-d6)
1.01
1.07
3.16
2.00
2.05
3.11
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
f1 (ppm)


138.184
131.891
130.255
128.446
128.110
125.864
50.910
40.021
39.854
39.687
39.520
39.353
39.187
39.020
19.348
Me
SO2NHNH2
2k
13C (125 MHz, DMSO-d6)
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)

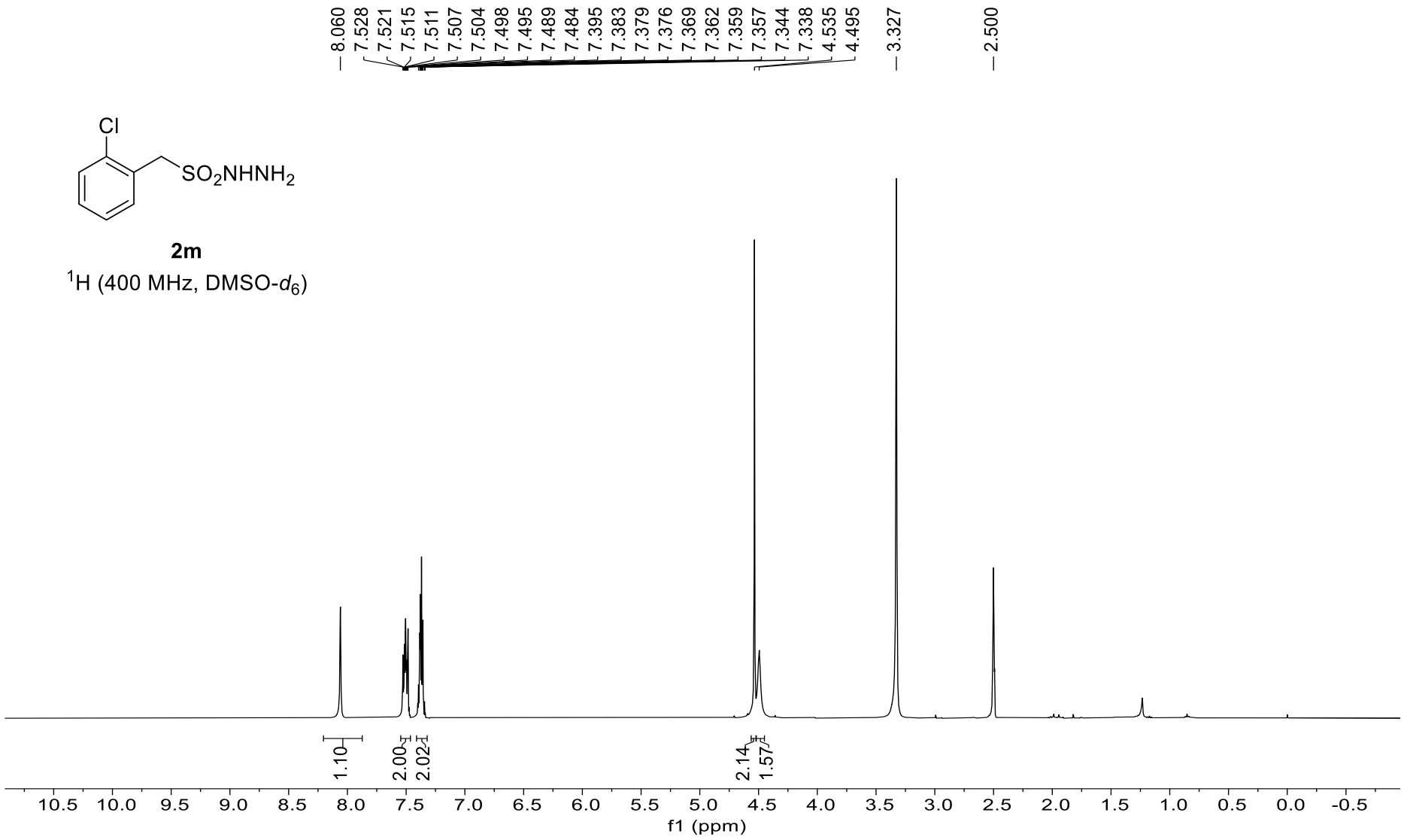

8.084
7.491
7.476
7.462
7.426
7.414
7.399
7.387
7.250
7.237
7.232
7.224
4.504
4.448
2.500
F
SO2NHNH2
2l
1H (500 MHz, DMSO-d6)
1.01
0.97
1.00
1.92
1.83
1.95
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

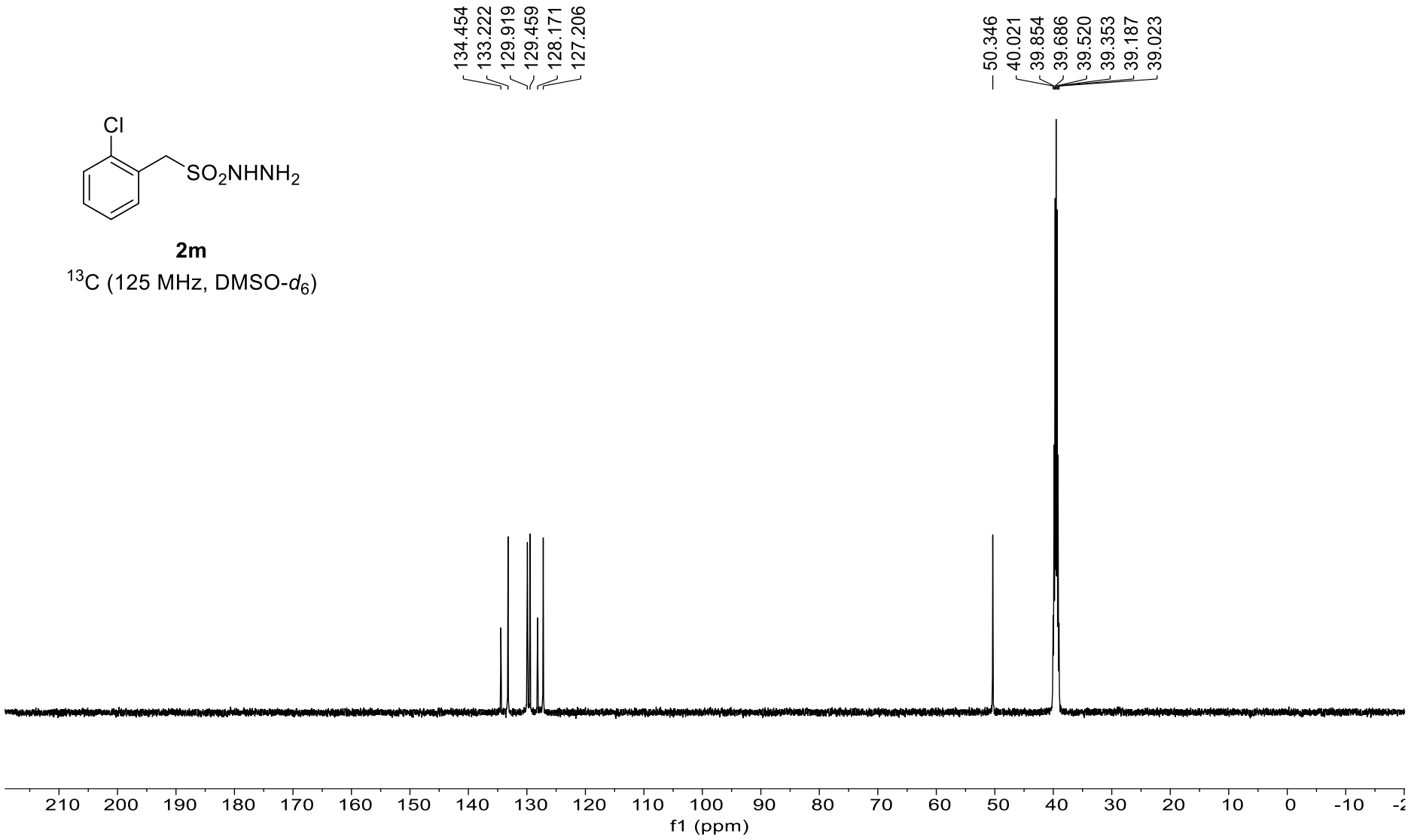

162.063
160.095
133.183
133.158
130.454
130.388
124.470
124.443
117.462
117.342
115.526
115.354
46.684
40.020
39.854
39.687
39.520
39.353
39.186
39.018
F
SO2NHNH2
2l
13C (125 MHz, DMSO-d6)
f1 (ppm)
f1 (ppm)
f1 (ppm)

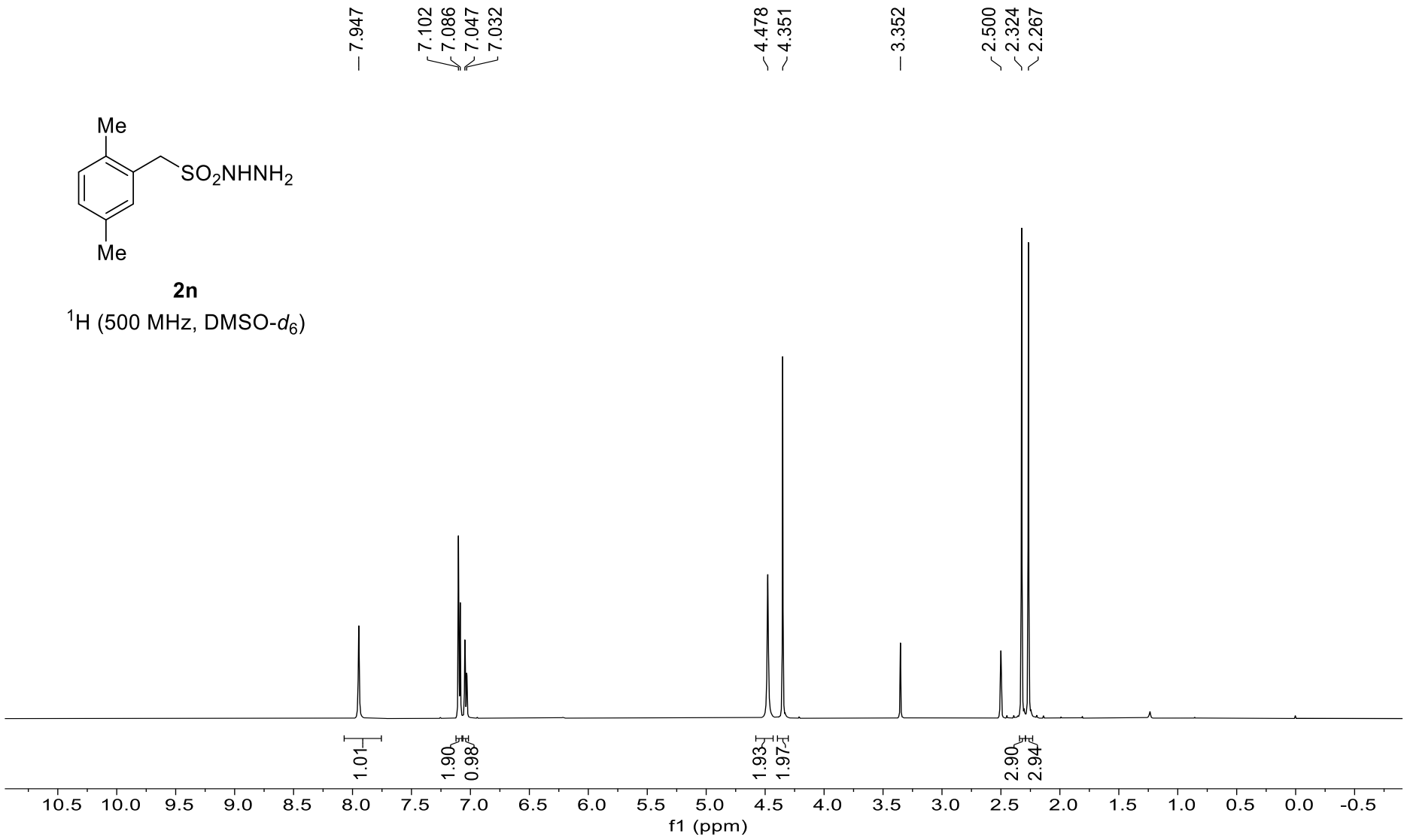


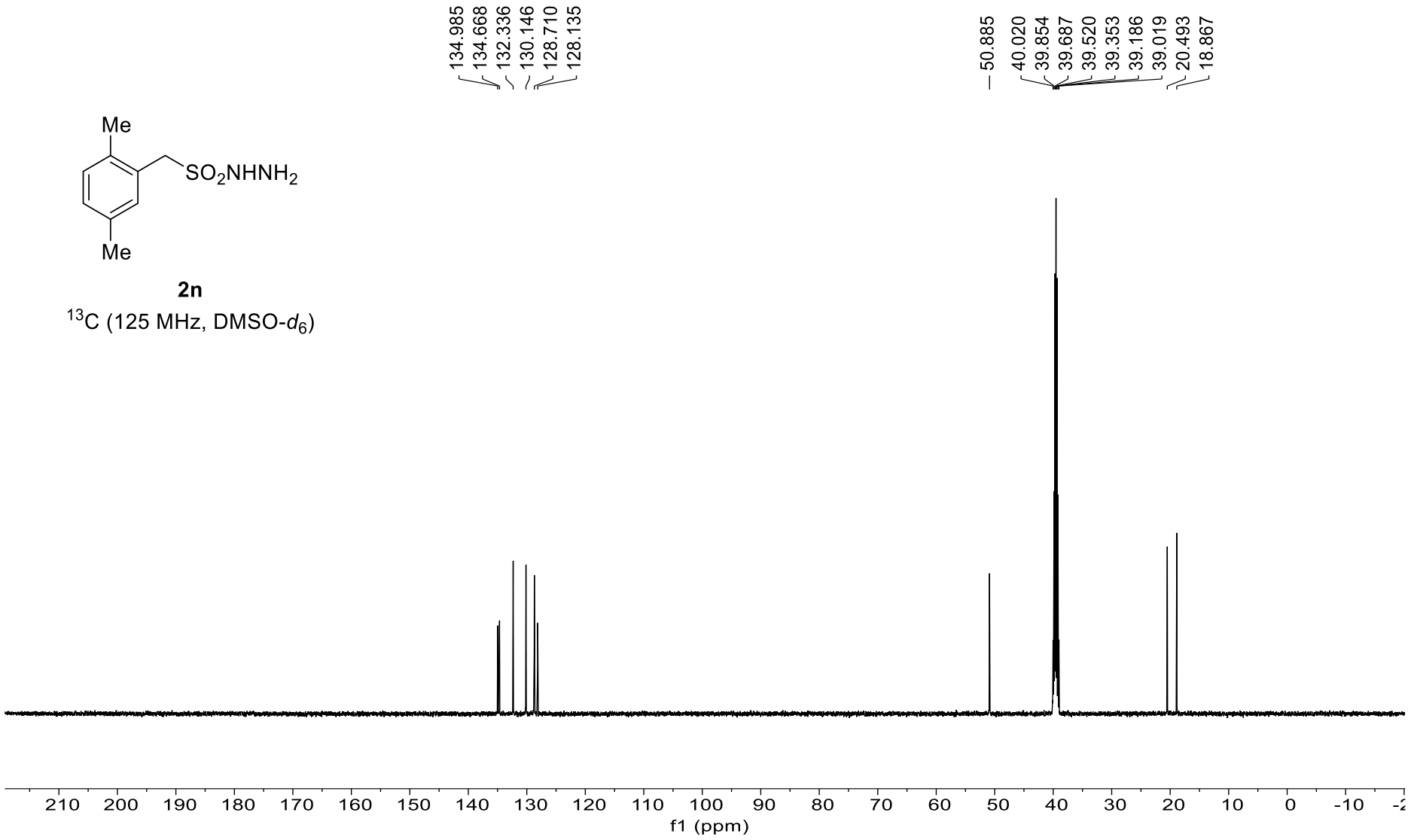
2l

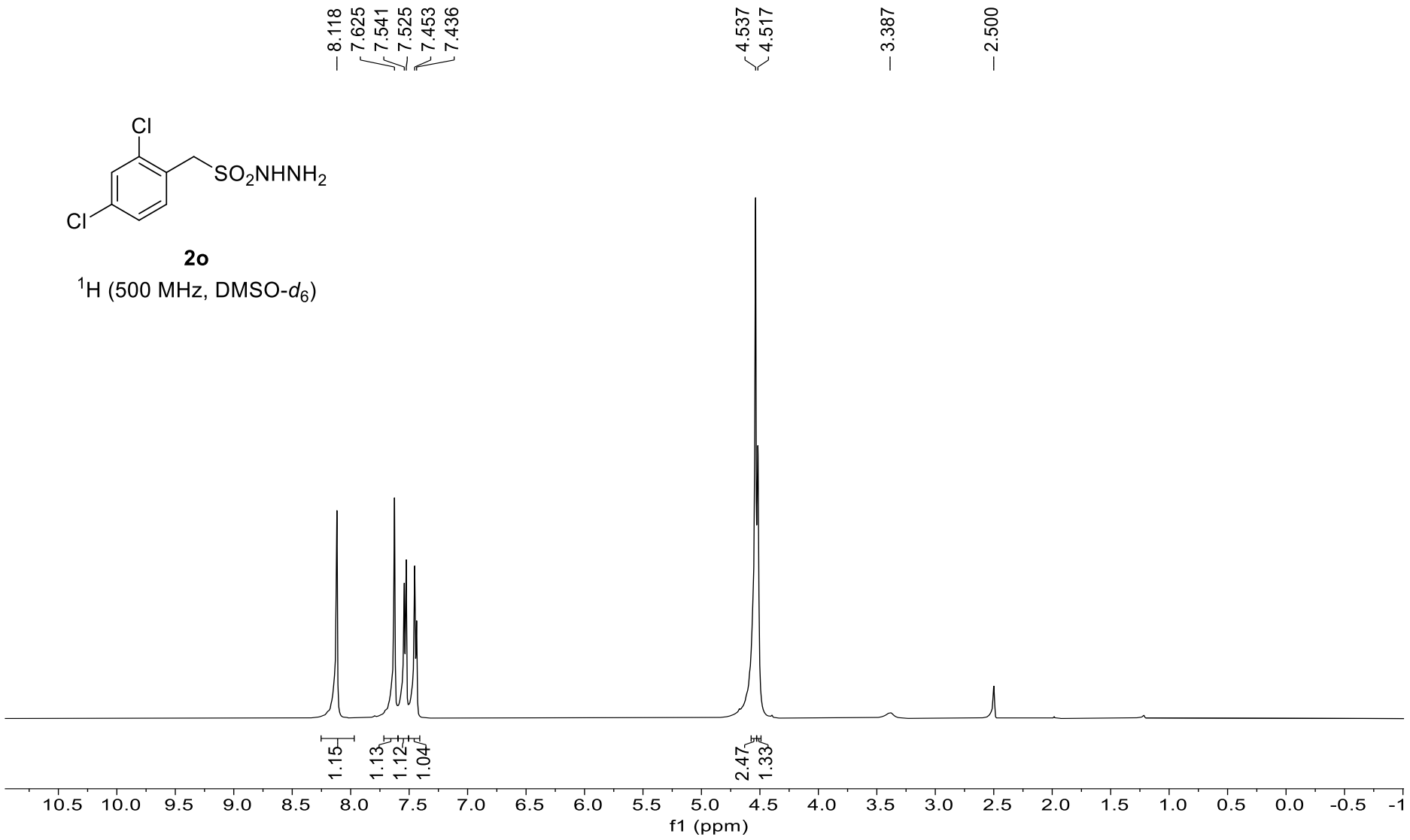
^{19}F (471 MHz, DMSO-d_6)

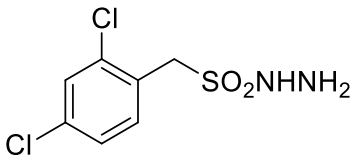

8.060
7.528
7.521
7.515
7.511
7.507
7.504
7.498
7.495
7.489
7.484
7.395
7.383
7.379
7.376
7.369
7.362
7.359
7.357
7.344
7.338
4.535
4.495
3.327
2.500
Cl
SO2NHNH2
2m
1H (400 MHz, DMSO-d6)
1.10
2.00
2.02
2.14
1.57
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


134.454
133.222
129.919
129.459
128.171
127.206
50.346
40.021
39.854
39.686
39.520
39.353
39.187
39.023
Cl
SO2NHNH2
2m
13C (125 MHz, DMSO-d6)
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)


7.947
7.102
7.086
7.047
7.032
4.478
4.351
3.352
2.500
2.324
2.267
Me
SO2NHNH2
Me
2n
1H (500 MHz, DMSO-d6)
1.01
1.90
0.98
1.93
1.97
2.90
2.94
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
f1 (ppm)

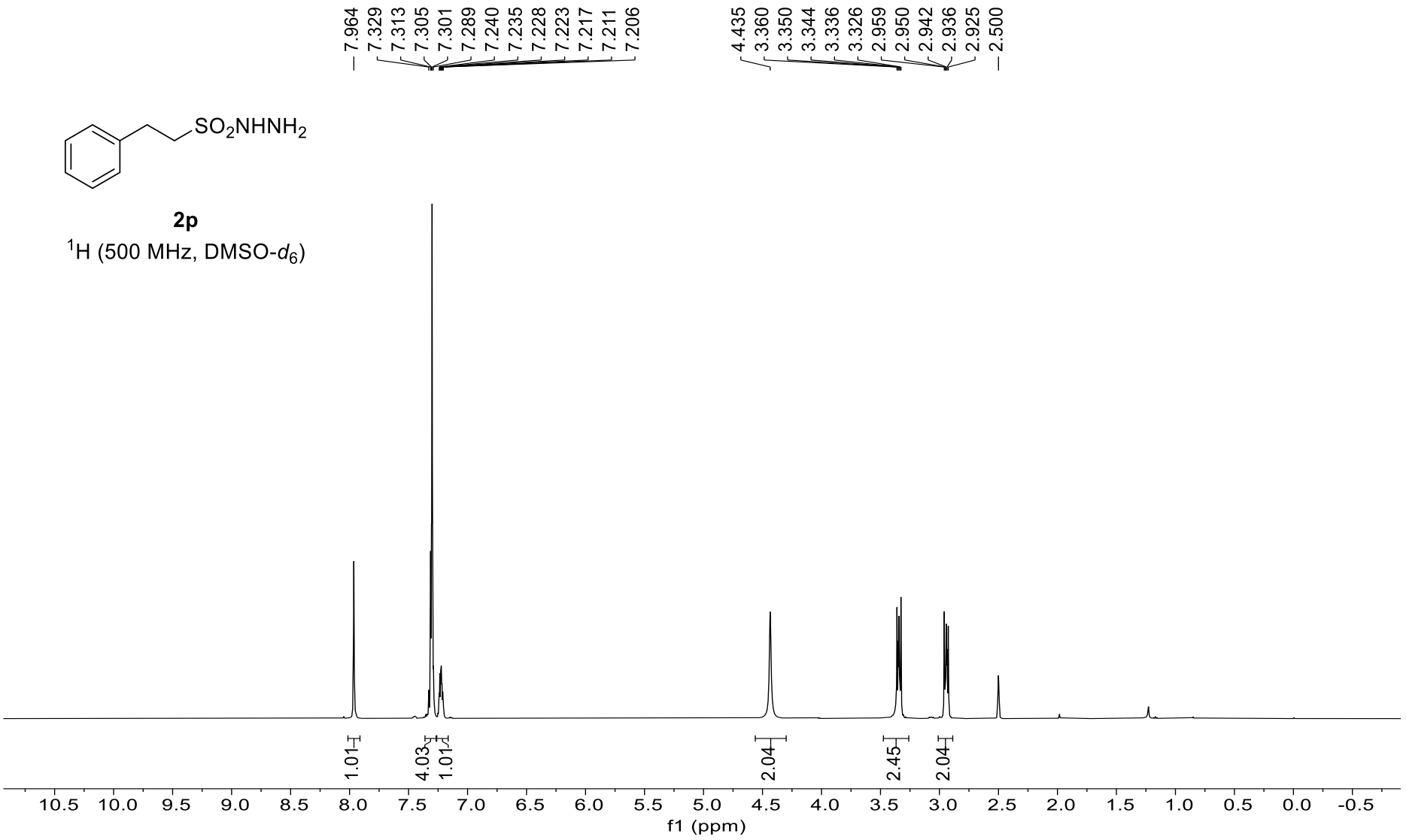

134.985
134.668
132.336
130.146
128.710
128.135
50.885
40.020
39.854
39.687
39.520
39.353
39.186
39.019
20.493
18.867
Me
SO2NHNH2
Me
2n
13C (125 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

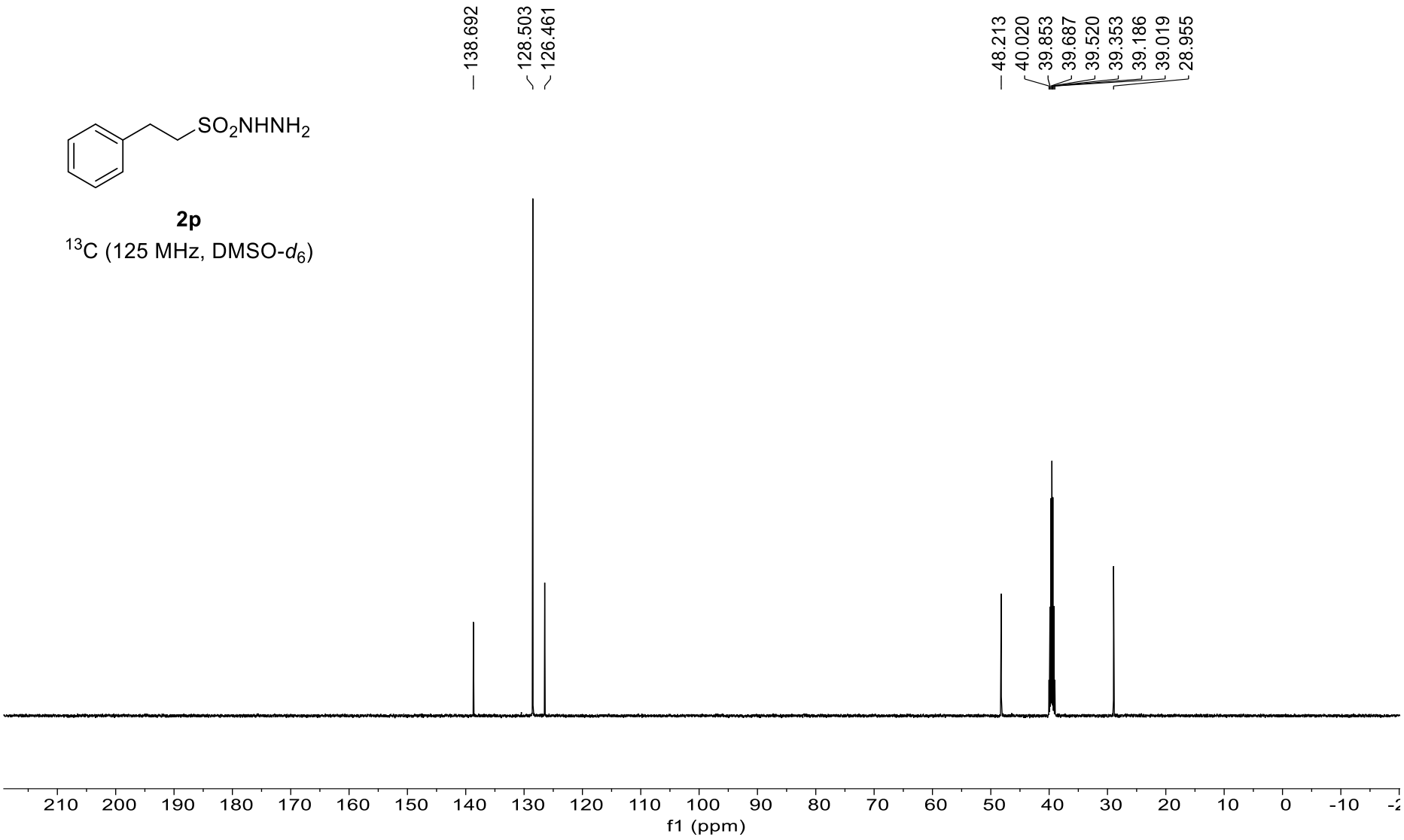

8.118
7.625
7.541
7.525
7.453
7.436
4.537
4.517
3.387
2.500
Cl
Cl
SO2NHNH2
2o
1H (500 MHz, DMSO-d6)
1.15
1.13
1.12
1.04
2.47
1.33
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

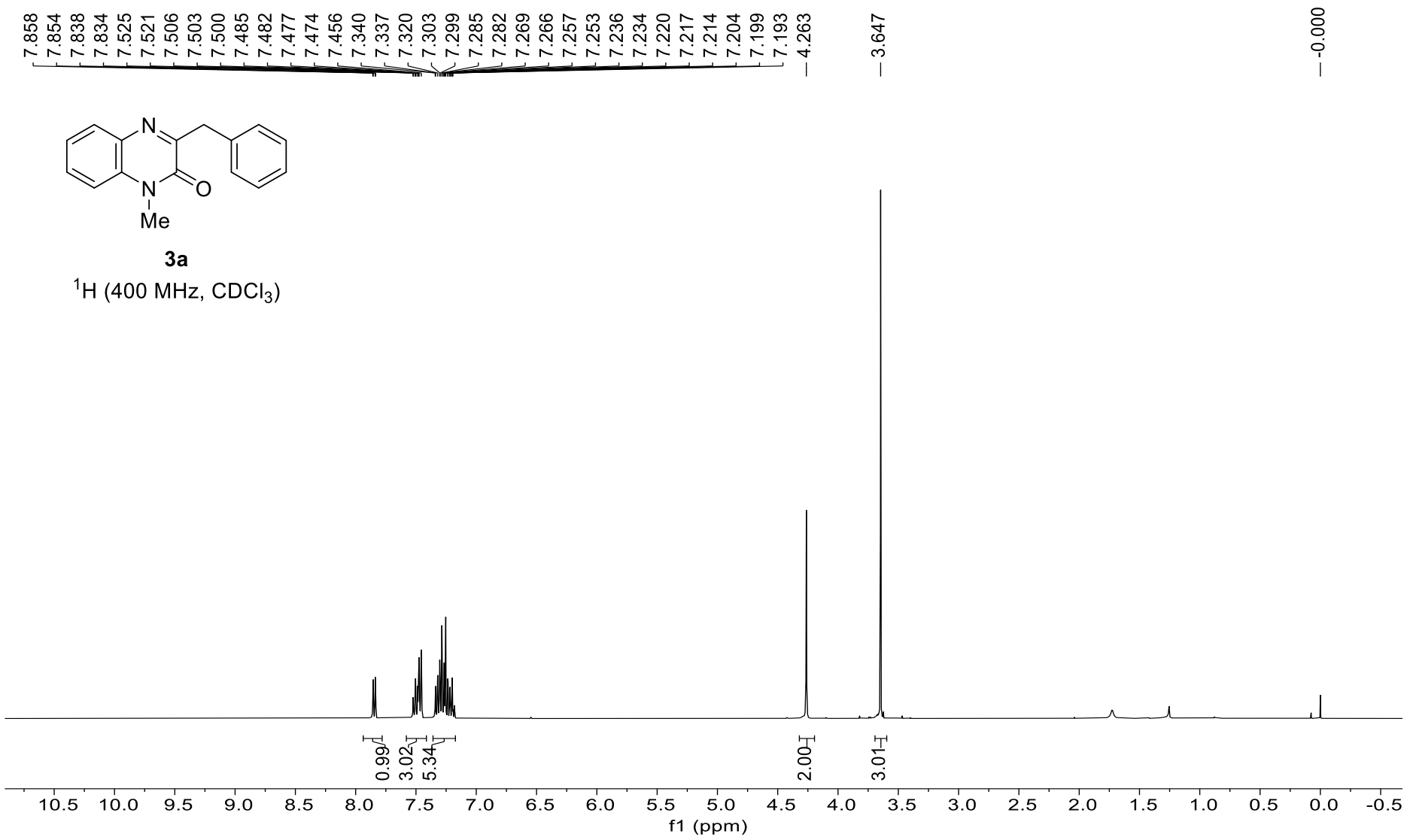


2o

^{13}C (125 MHz, DMSO-d_6)

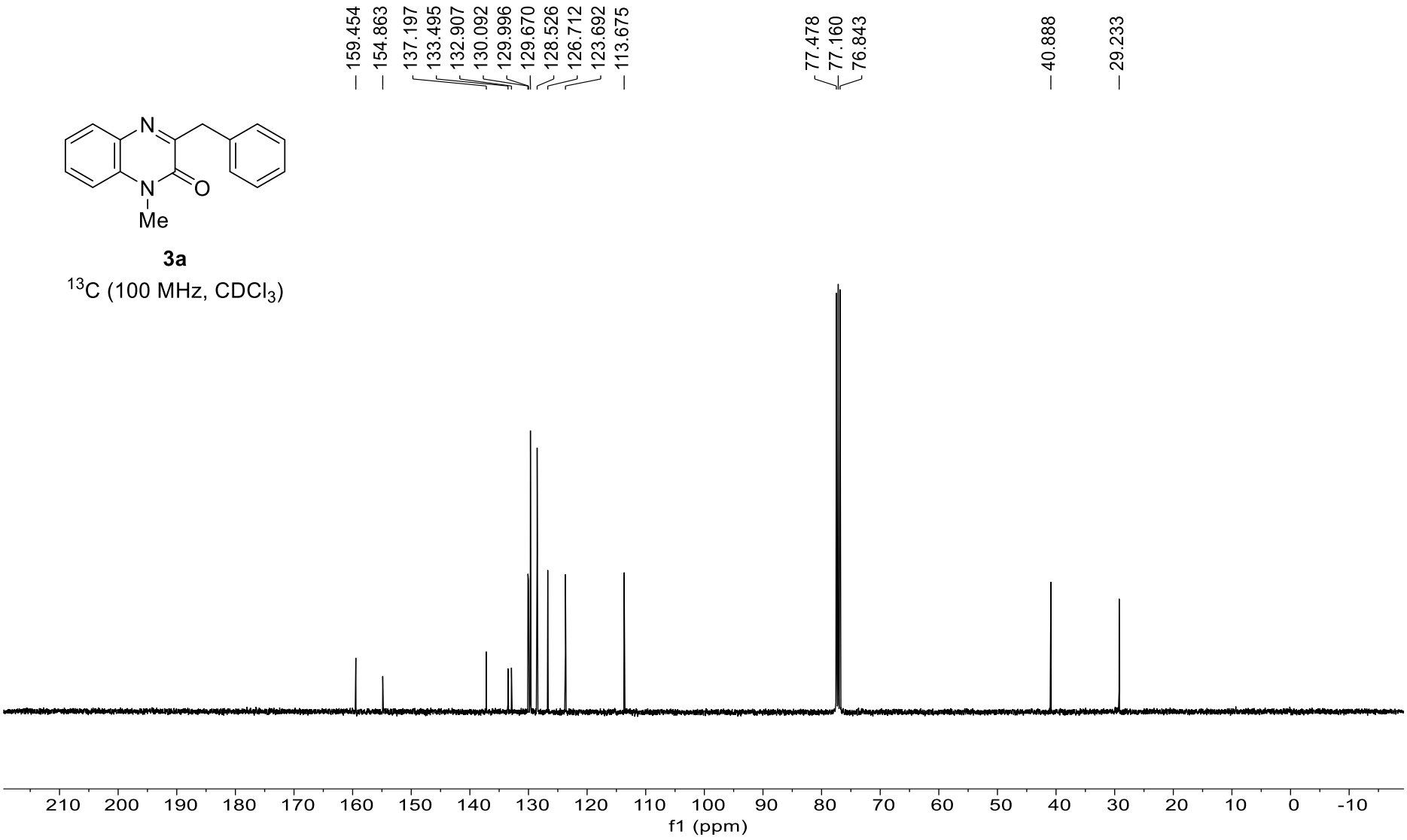

7.964
7.329
7.313
7.305
7.301
7.289
7.240
7.235
7.228
7.223
7.217
7.211
7.206
4.435
3.360
3.350
3.344
3.336
3.326
2.959
2.950
2.942
2.936
2.925
2.500
SO2NHNH2
2p
1H (500 MHz, DMSO-d6)
1.01
4.03
1.01
2.04
2.45
2.04
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

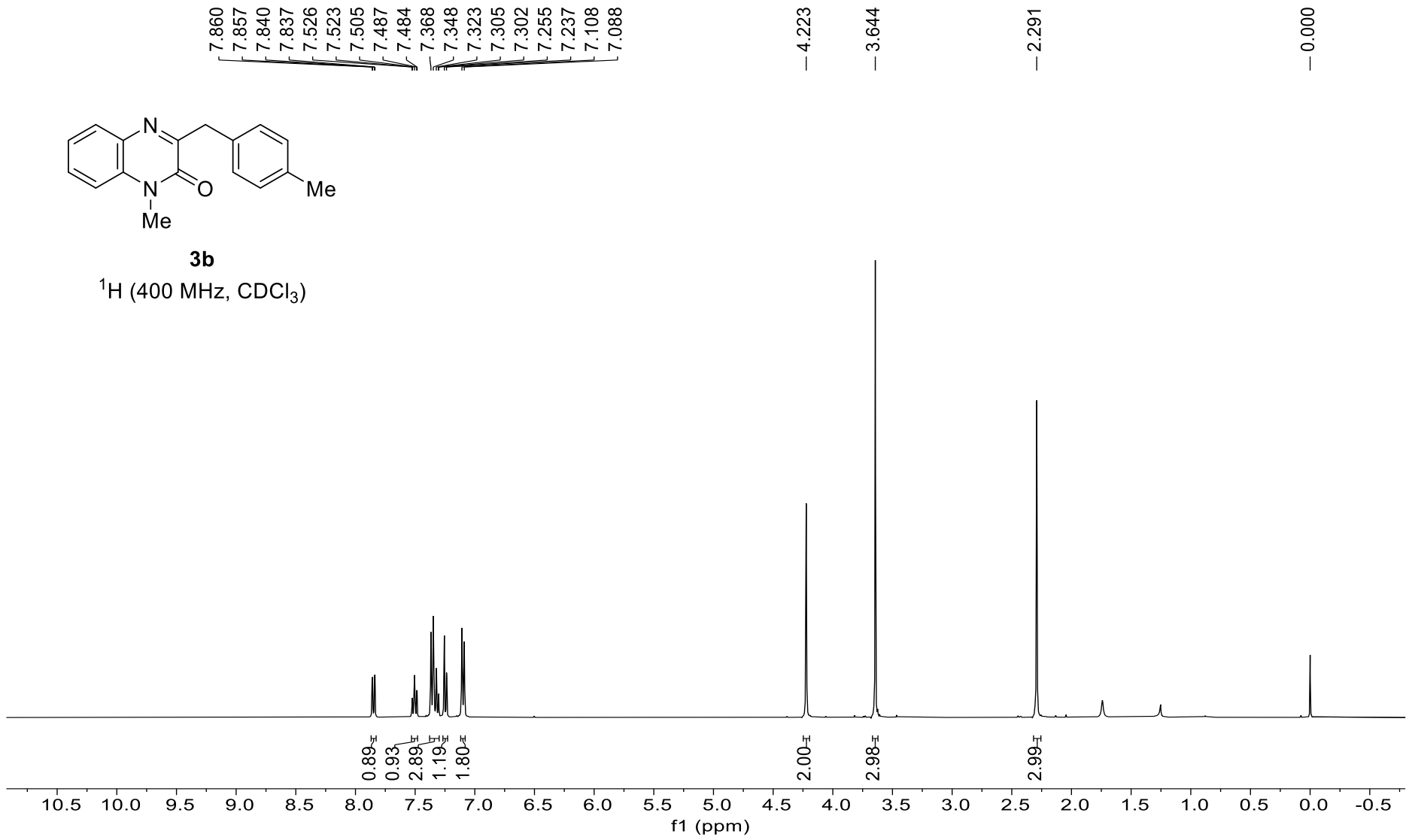

138.692
128.503
126.461
48.213
40.020
39.853
39.687
39.520
39.353
39.186
39.019
28.955
SO2NHNH2
2p
13C (125 MHz, DMSO-d6)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

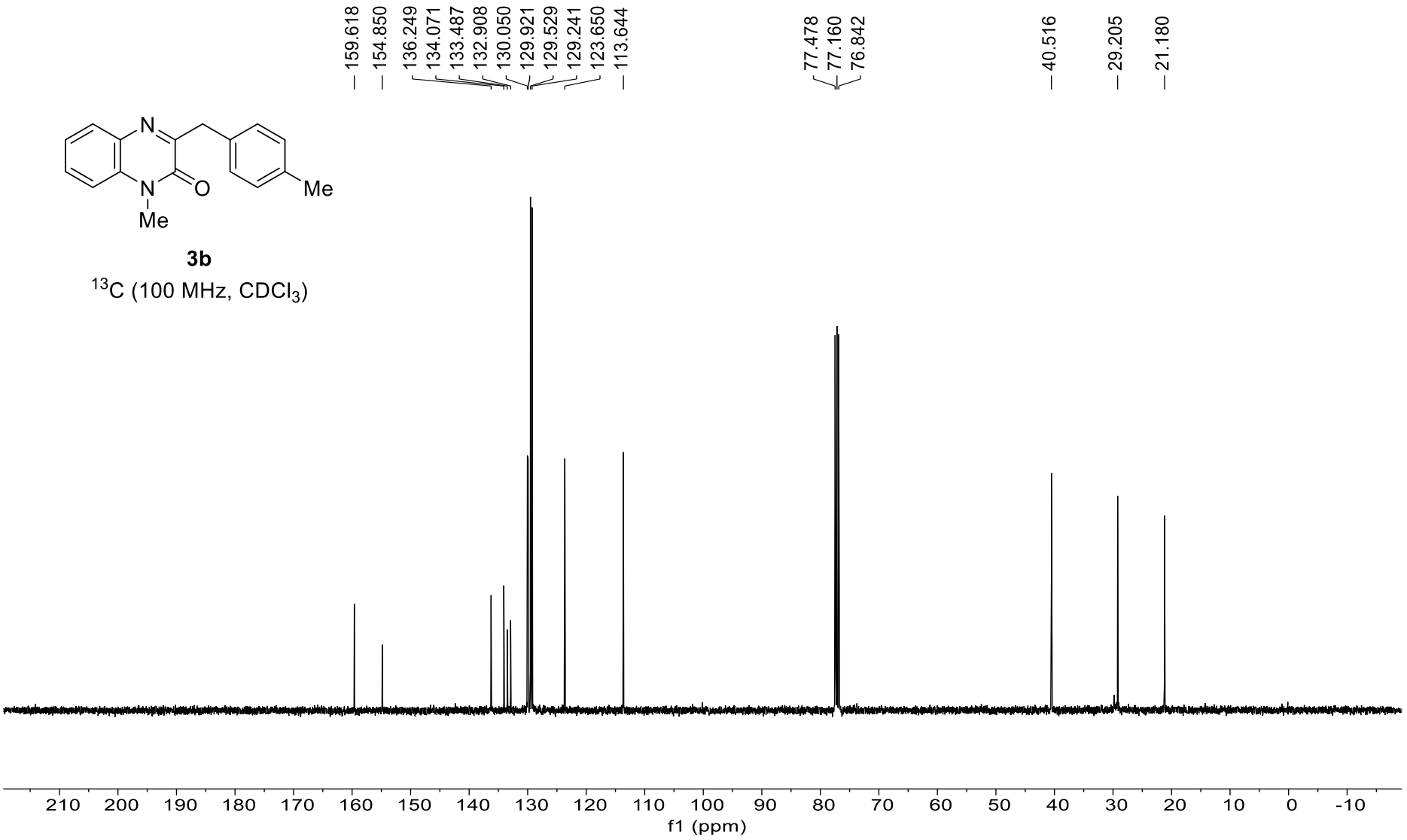


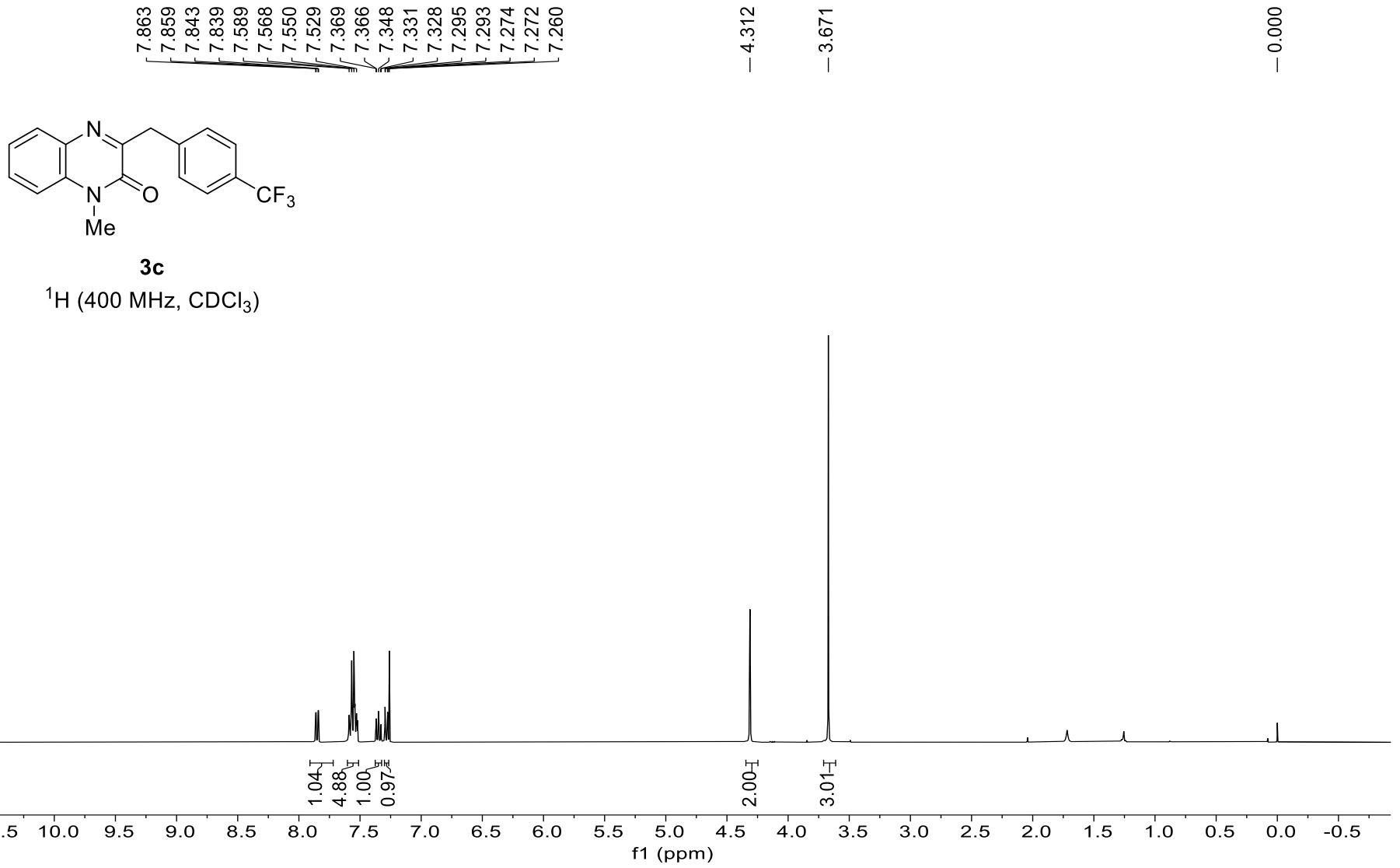
3a

^{1}H (400 MHz, $CDCl_3$)


159.454
154.863
137.197
133.495
132.907
130.092
129.996
129.670
128.526
126.712
123.692
113.675
77.478
77.160
76.843
40.888
29.233
N
N
O
Me
3a
13C (100 MHz, CDCl3)
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)

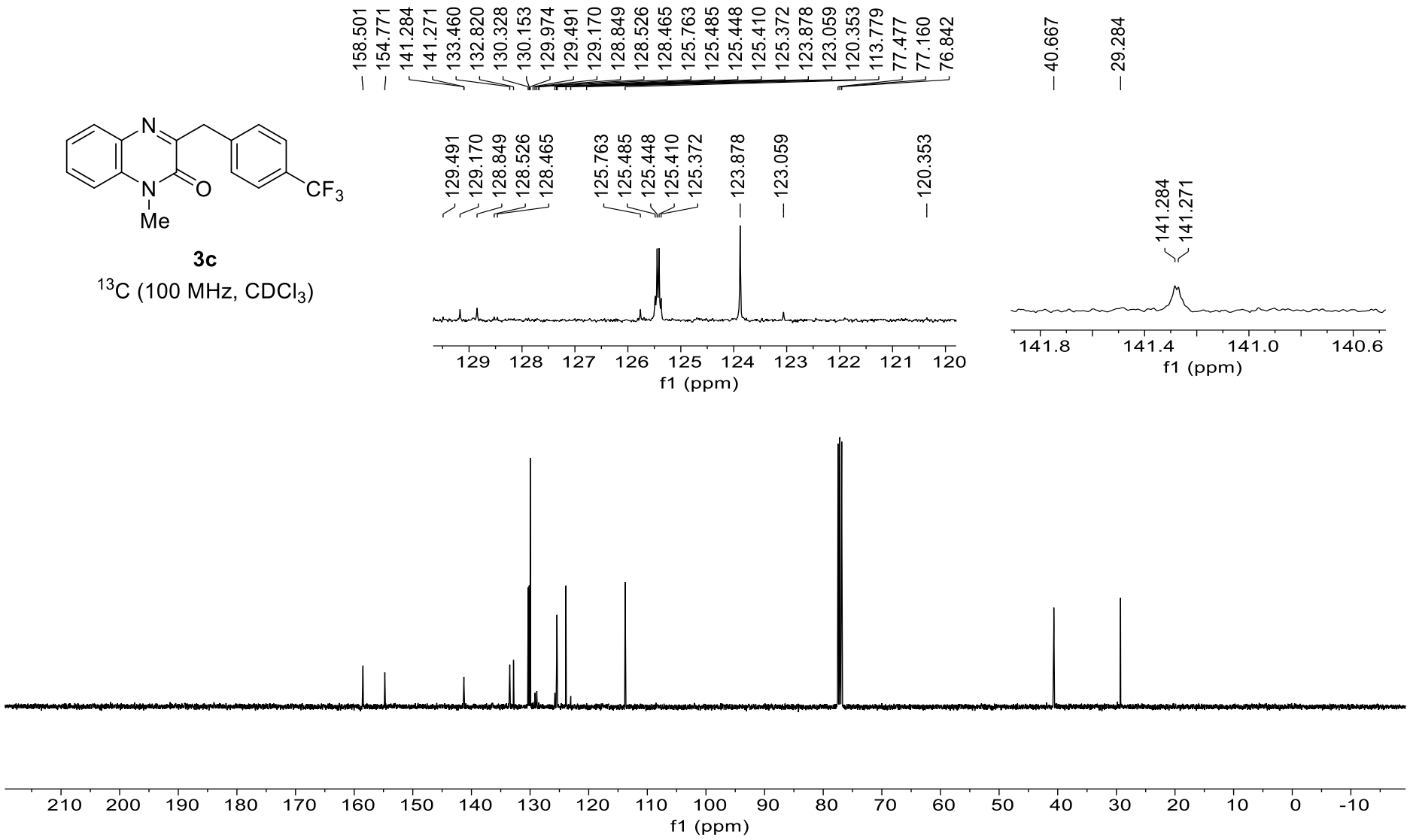

7.860
7.857
7.840
7.837
7.526
7.523
7.505
7.487
7.484
7.368
7.348
7.323
7.305
7.302
7.255
7.237
7.108
7.088
4.223
3.644
2.291
0.000
N
N
O
Me
Me
3b
1H (400 MHz, CDCl3)
0.89
0.93
2.89
1.19
1.80
2.00
2.98
2.99
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

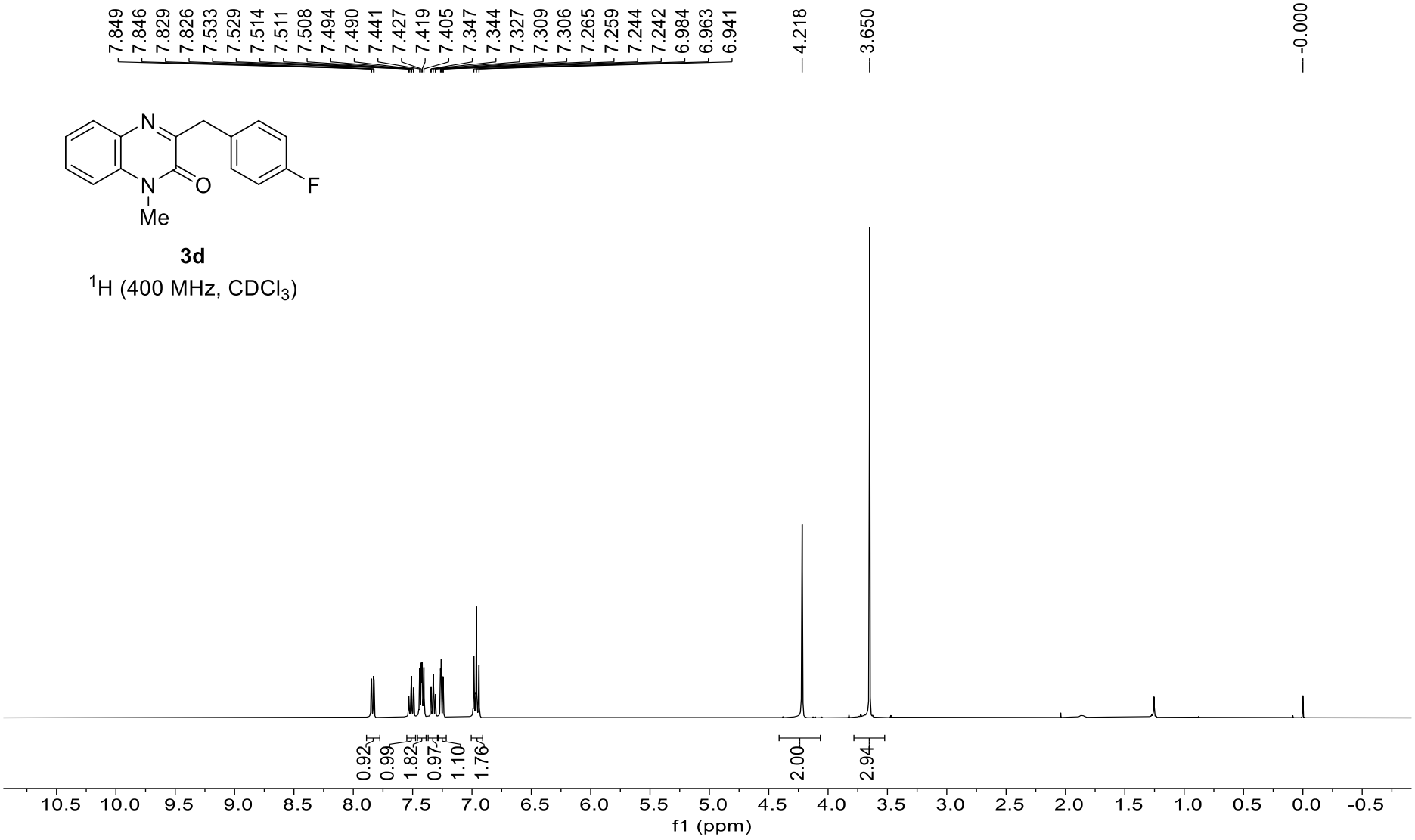

159.618
154.850
136.249
134.071
133.487
132.908
130.050
129.921
129.529
129.241
123.650
113.644
77.478
77.160
76.842
40.516
29.205
21.180
N
N
O
Me
Me
3b
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

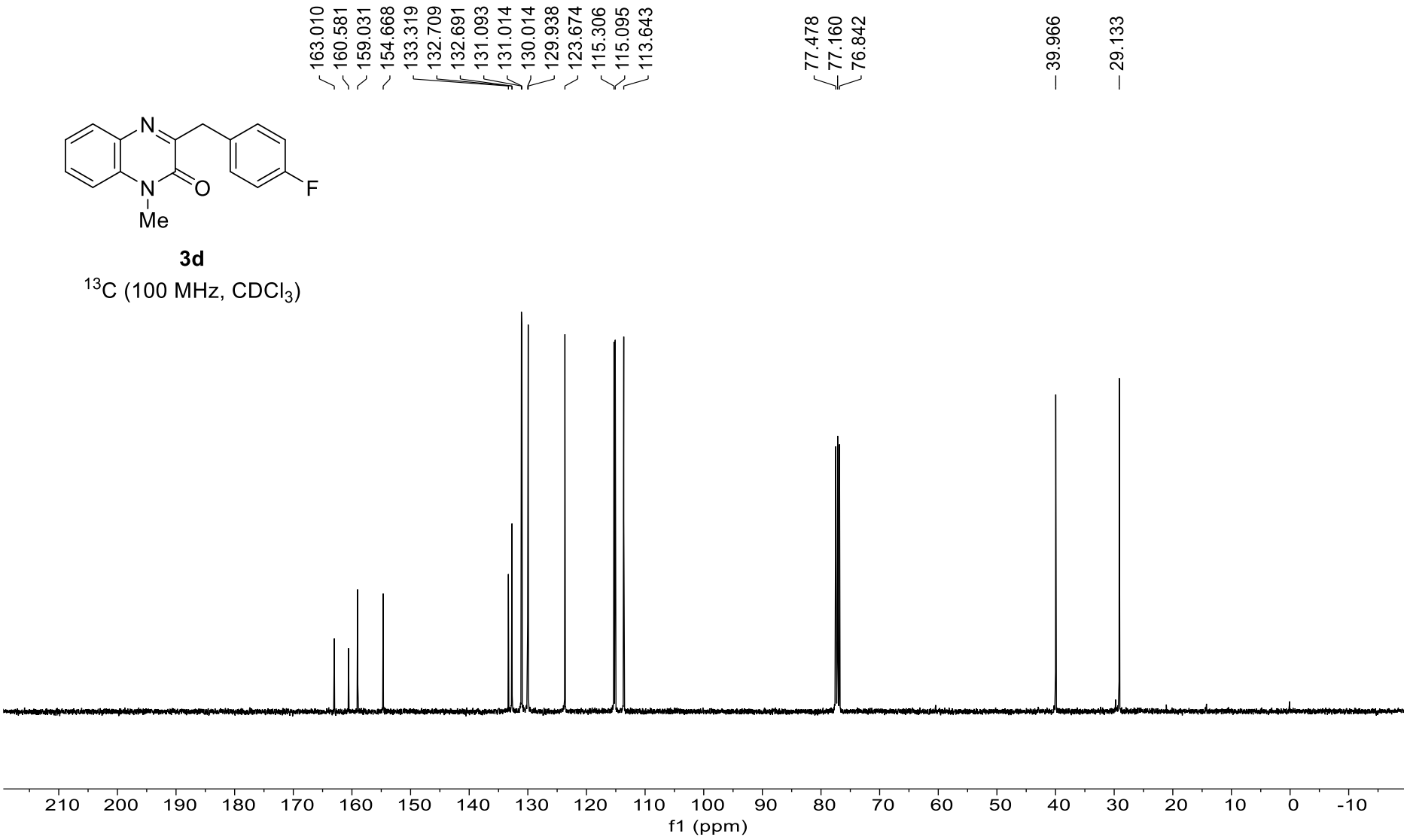


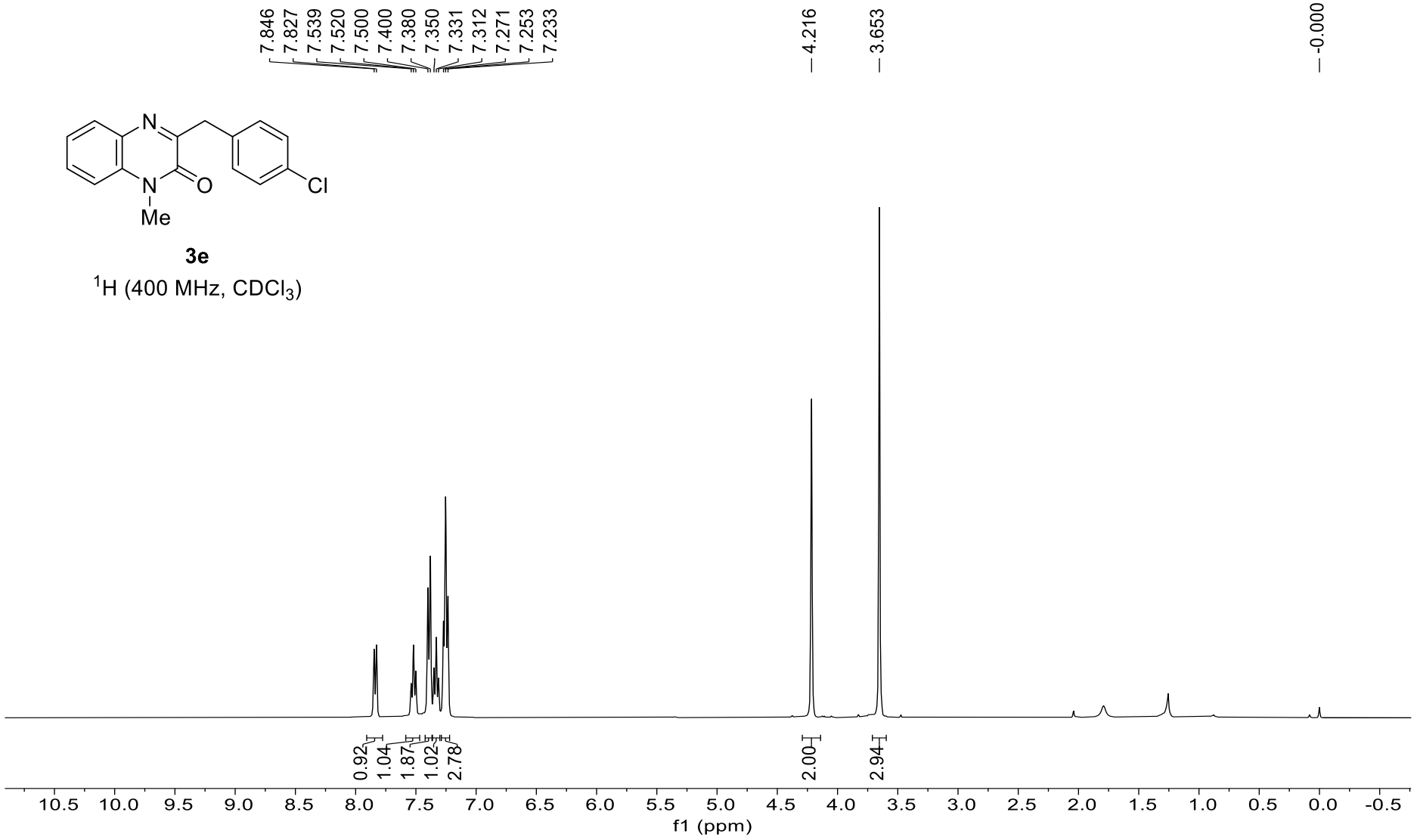
3c

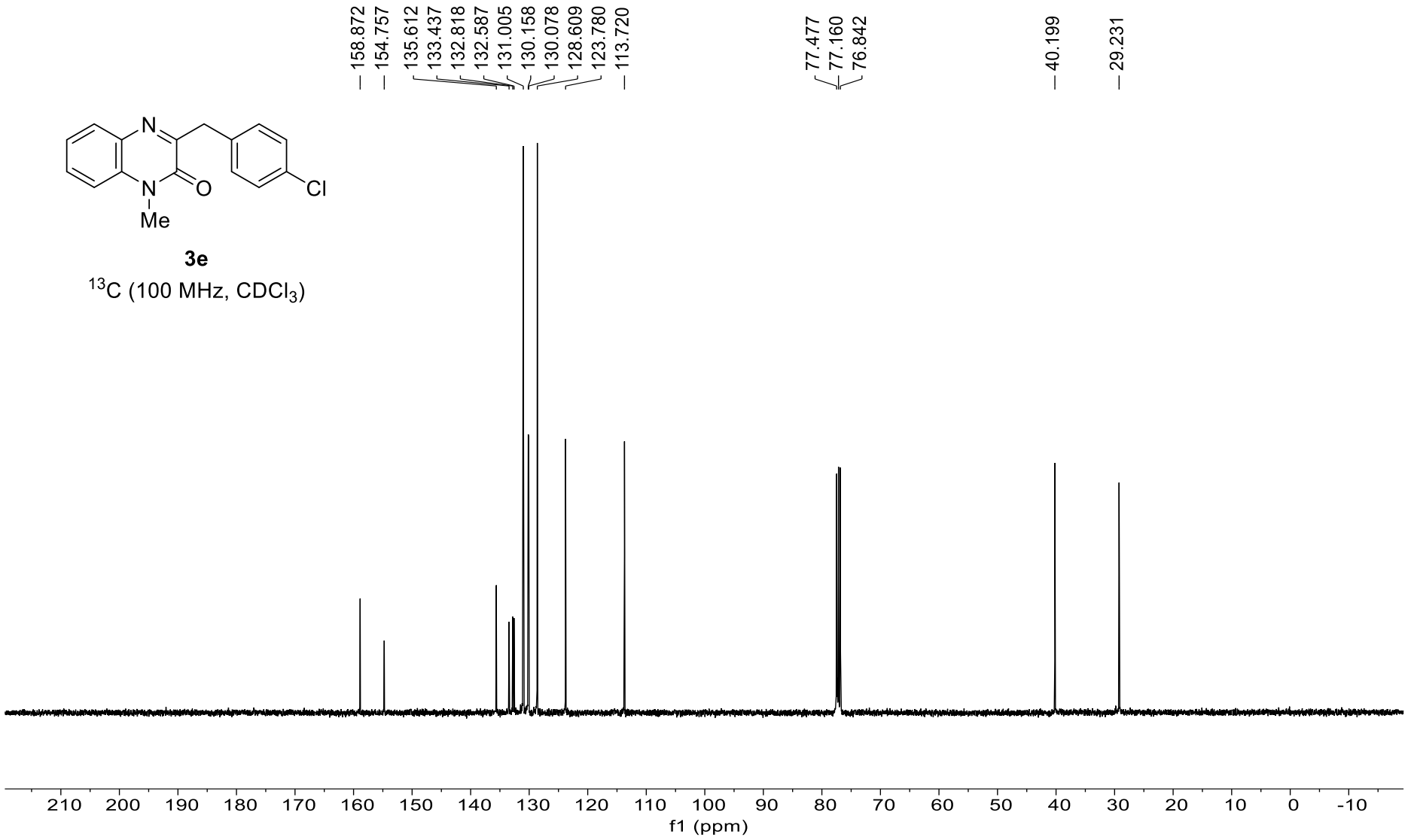
^{1}H (400 MHz, $CDCl_3$)

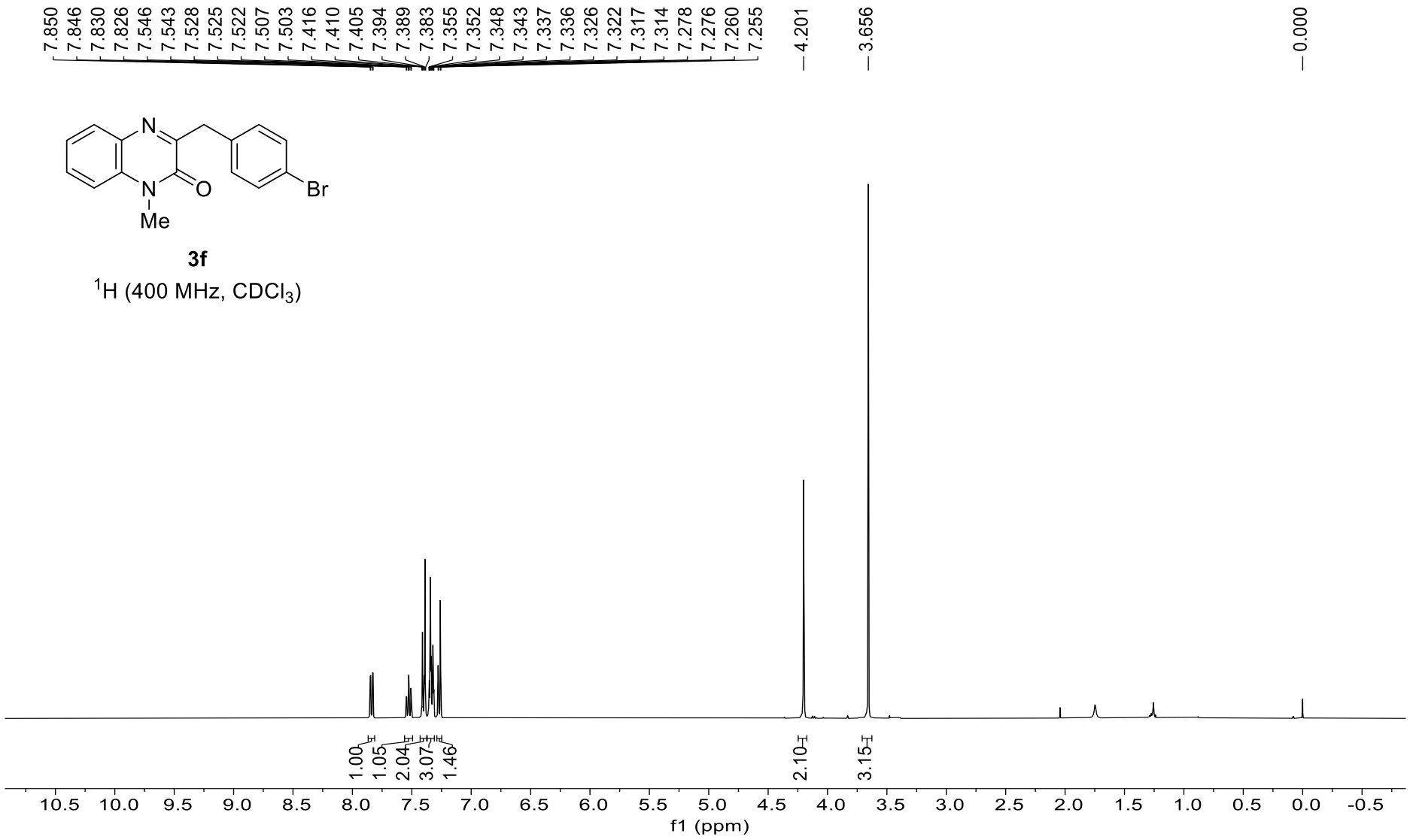

158.501
154.771
141.284
141.271
133.460
132.820
130.328
130.153
129.974
129.491
129.170
128.849
128.526
128.465
125.763
125.485
125.448
125.410
125.372
123.878
123.059
120.353
113.779
77.477
77.160
76.842
40.667
29.284
N
N
O
Me
CF3
3c
13C (100 MHz, CDCl3)
f1 (ppm)
f1 (ppm)
f1 (ppm)


7.849
7.846
7.829
7.826
7.533
7.529
7.514
7.511
7.508
7.494
7.490
7.441
7.427
7.419
7.405
7.347
7.344
7.327
7.309
7.306
7.265
7.259
7.244
7.242
6.984
6.963
6.941
4.218
3.650
-0.000
N
N
O
F
Me
3d
1H (400 MHz, CDCl3)
0.92
0.99
1.82
0.97
1.10
1.76
2.00
2.94
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
f1 (ppm)


163.010
160.581
159.031
154.668
133.319
132.709
132.691
131.093
131.014
130.014
129.938
123.674
115.306
115.095
113.643
77.478
77.160
76.842
39.966
29.133
N
N
O
F
Me
3d
13C (100 MHz, CDCl3)
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)

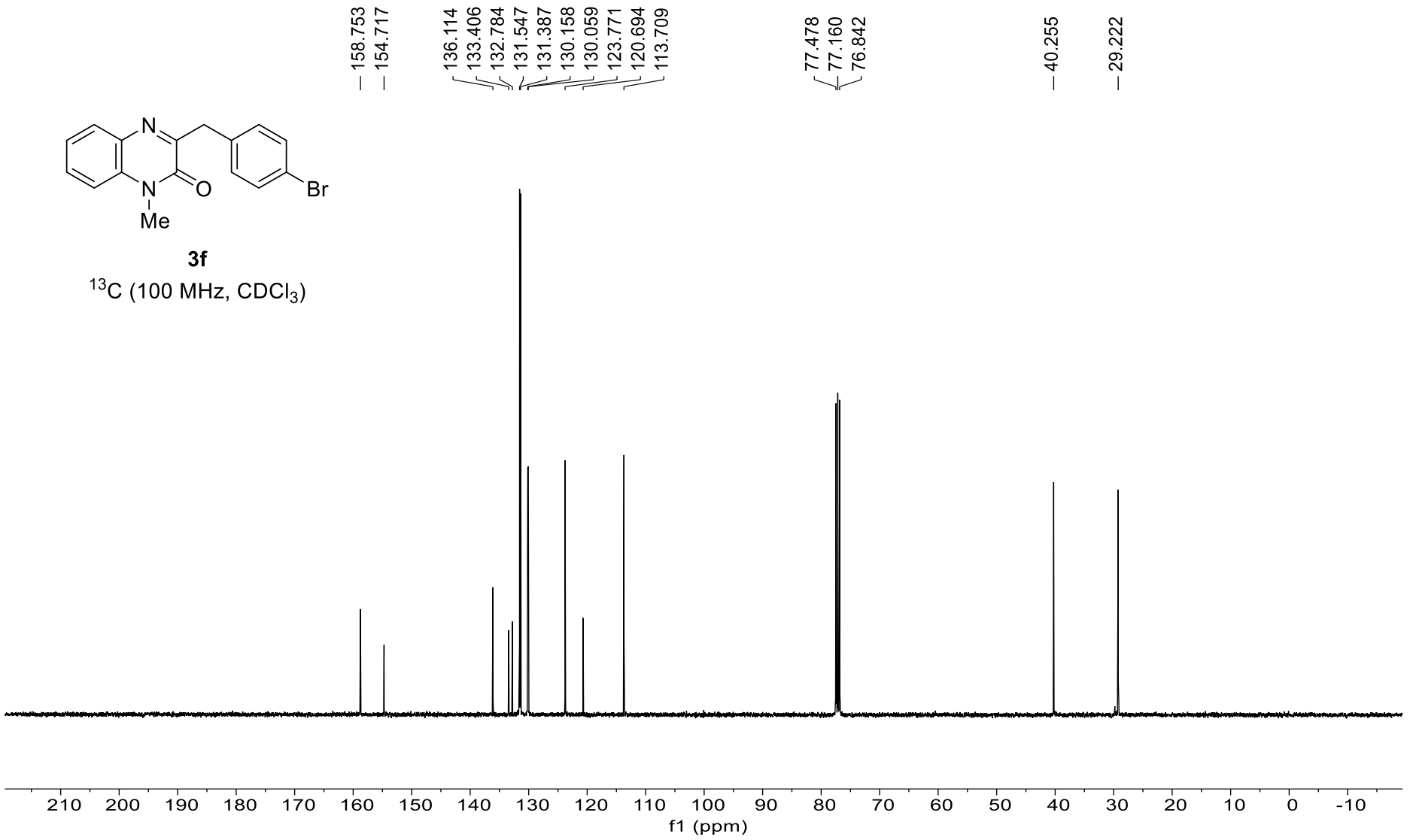

7.846
7.827
7.539
7.520
7.500
7.400
7.380
7.350
7.331
7.312
7.271
7.253
7.233
4.216
3.653
-0.000
N
N
O
Cl
Me
3e
1H (400 MHz, CDCl3)
0.92
1.04
1.87
1.02
2.78
2.00
2.94
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

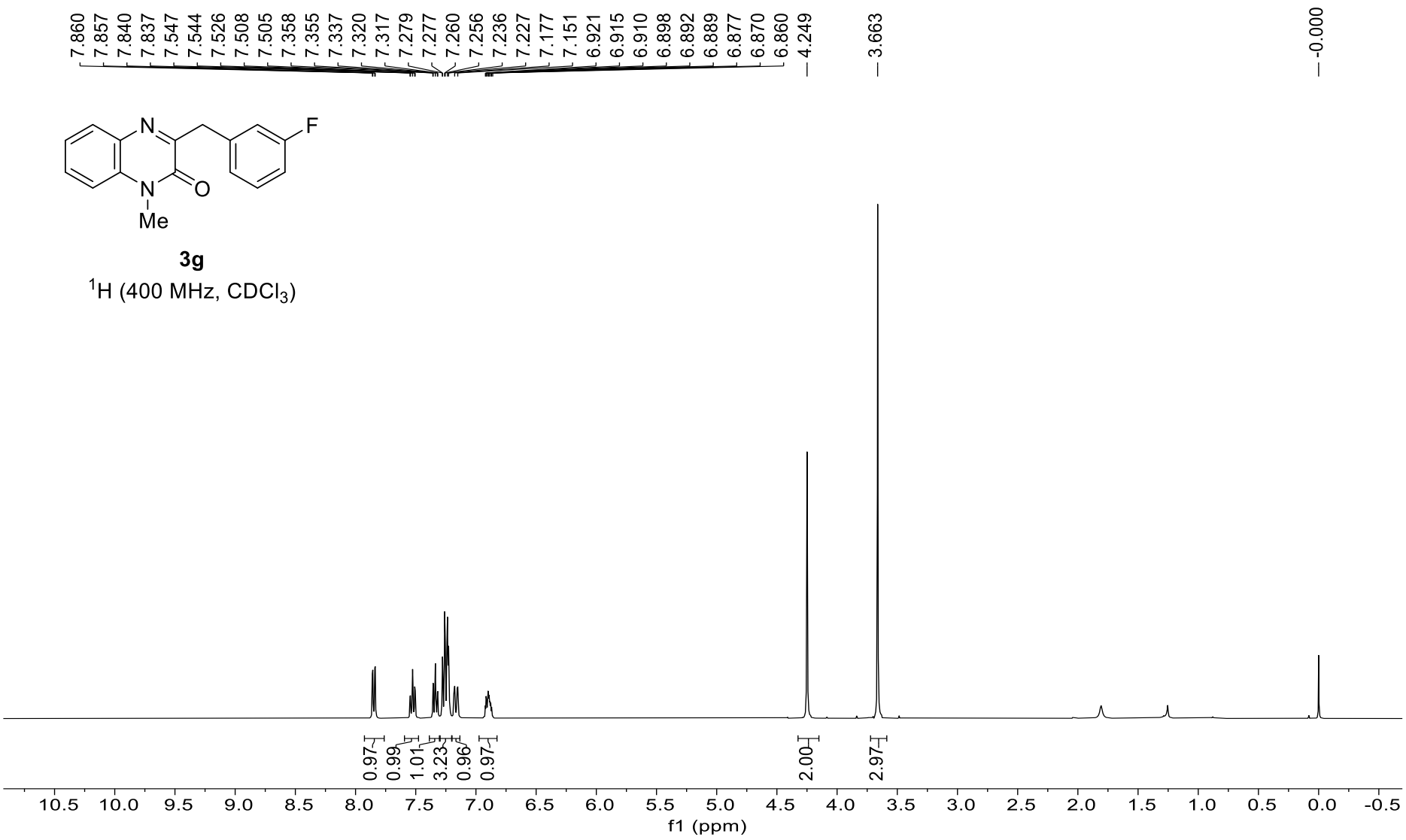

158.872
154.757
135.612
133.437
132.818
132.587
131.005
130.158
130.078
128.609
123.780
113.720
77.477
77.160
76.842
40.199
29.231
N
N
O
Me
Cl
3e
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

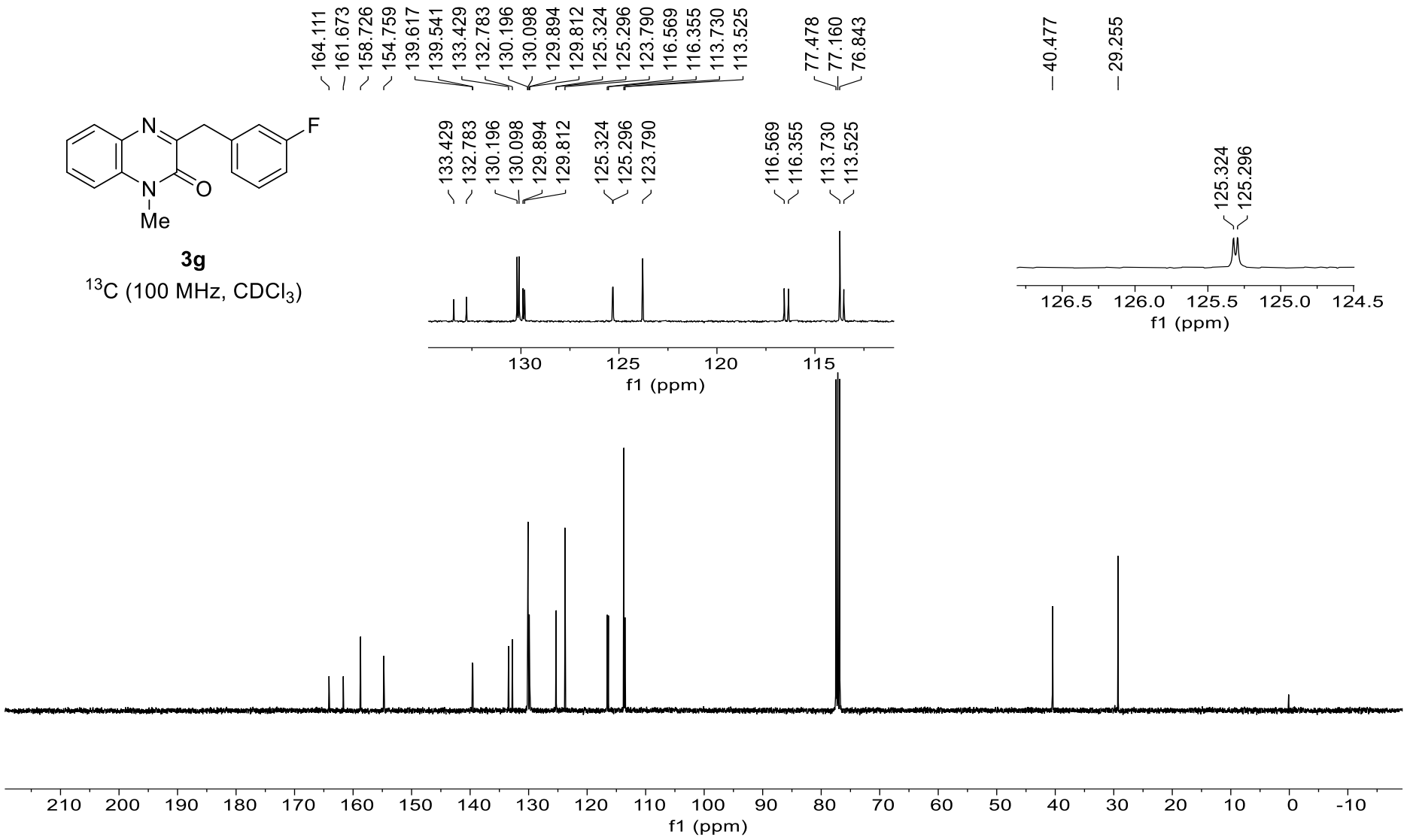


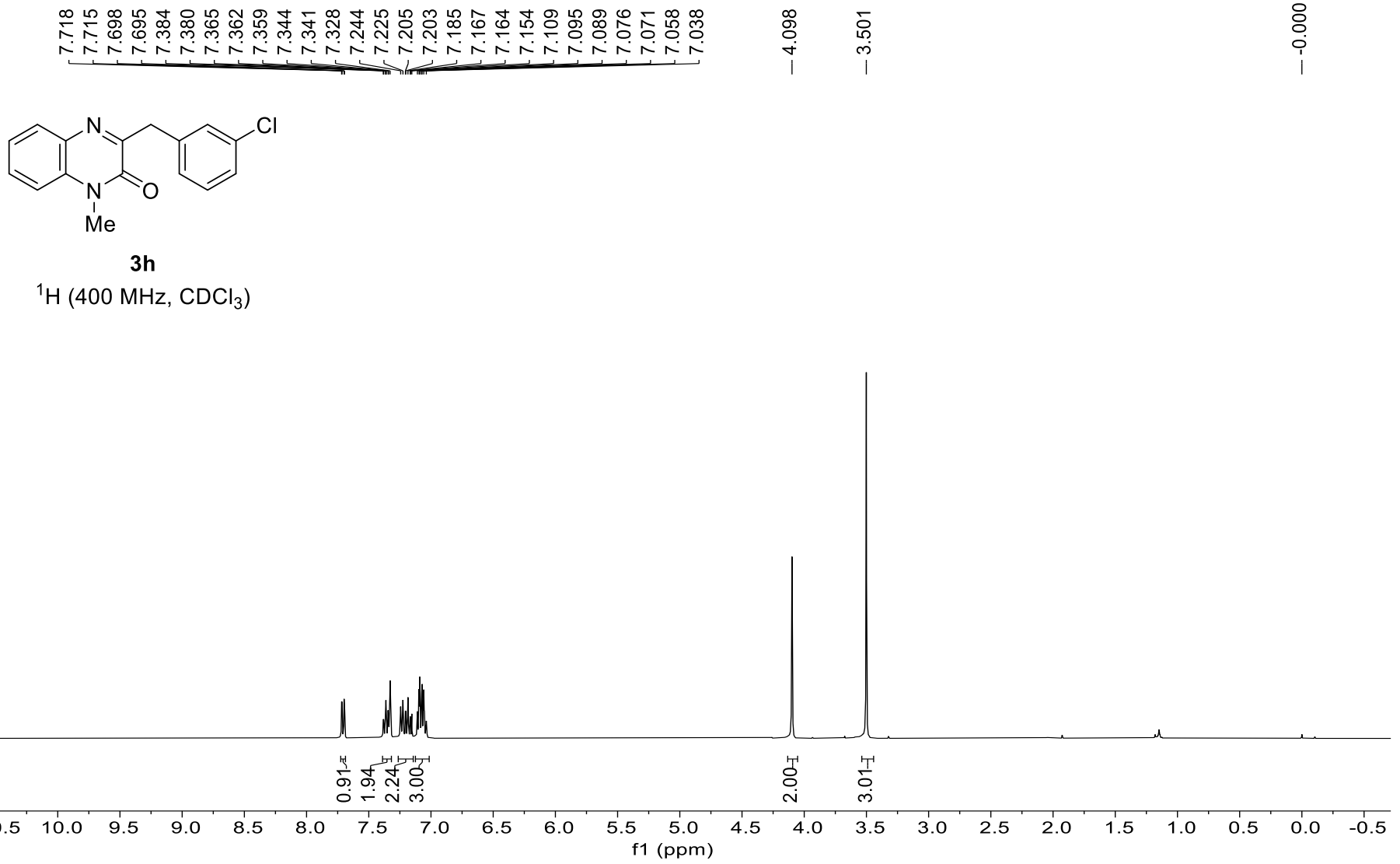
3f

^{1}H (400 MHz, $CDCl_3$)


158.753
154.717
136.114
133.406
132.784
131.547
131.387
130.158
130.059
123.771
120.694
113.709
77.478
77.160
76.842
40.255
29.222
N
N
O
Me
Br
3f
13C (100 MHz, CDCl3)
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)

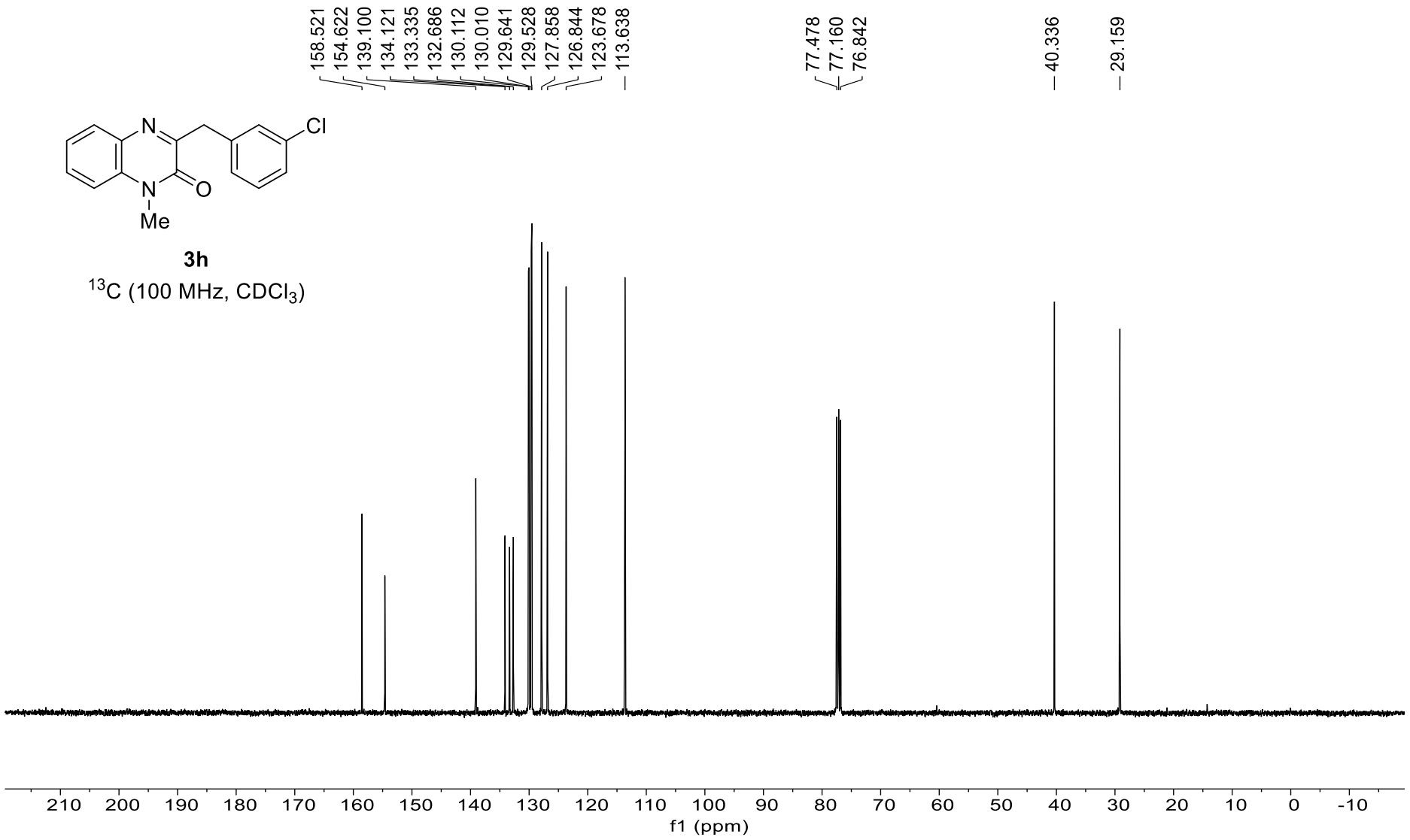

7.860
7.857
7.840
7.837
7.547
7.544
7.526
7.508
7.505
7.358
7.355
7.337
7.320
7.317
7.279
7.277
7.260
7.256
7.236
7.227
7.177
7.151
6.921
6.915
6.910
6.898
6.892
6.889
6.877
6.870
6.860
4.249
3.663
-0.000
N
F
N
O
Me
3g
1H (400 MHz, CDCl3)
0.97
0.99
1.01
3.23
0.96
0.97
2.00
2.97
10.5 10.0 9.5 9.0 8.5 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
f1 (ppm)

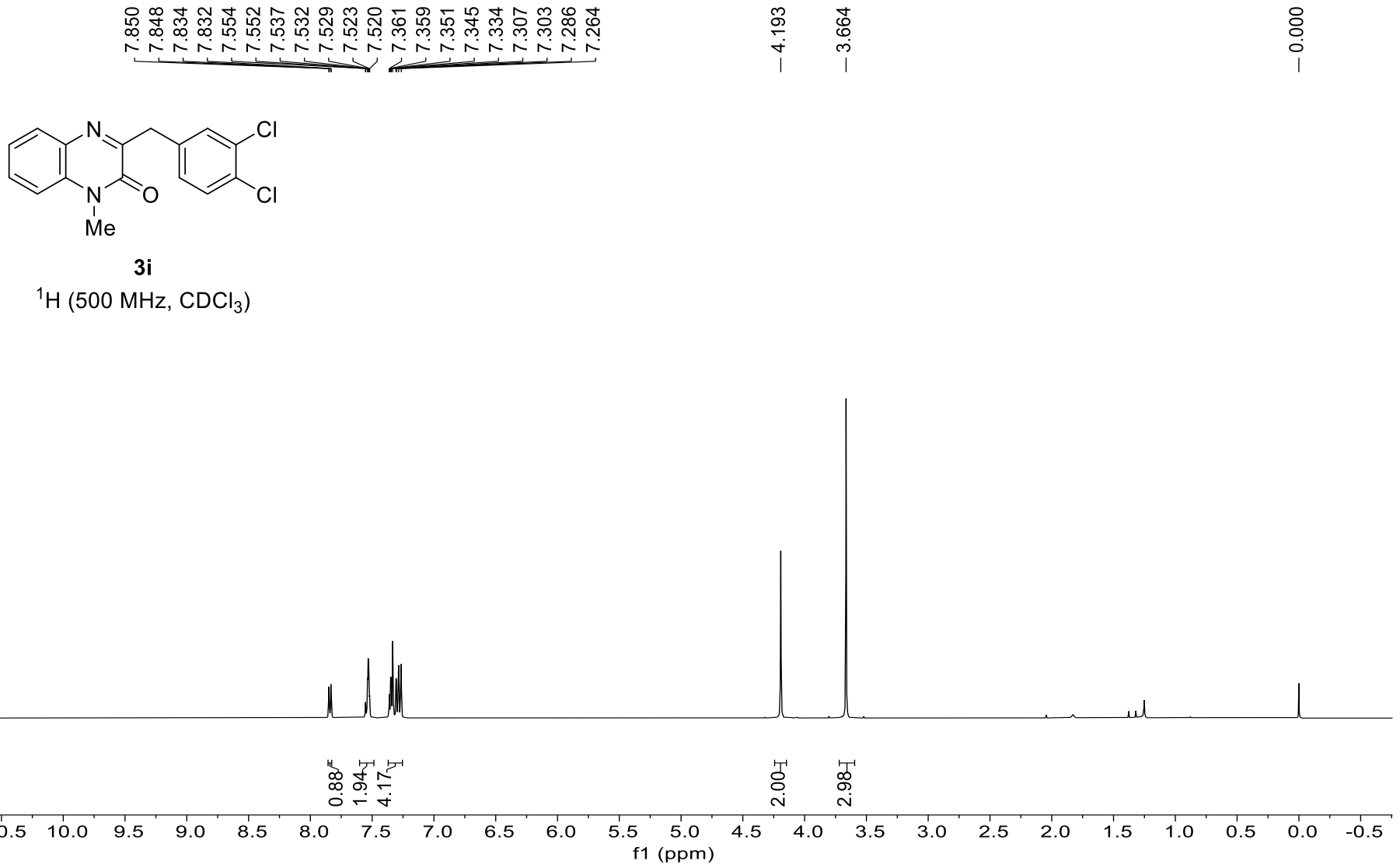

164.111
161.673
158.726
154.759
139.617
139.541
133.429
132.783
130.196
130.098
129.894
129.812
125.324
125.296
123.790
116.569
116.355
113.730
113.525
77.478
77.160
76.843
40.477
29.255
N
F
N
O
Me
3g
13C (100 MHz, CDCl3)
f1 (ppm)
f1 (ppm)
f1 (ppm)



3h

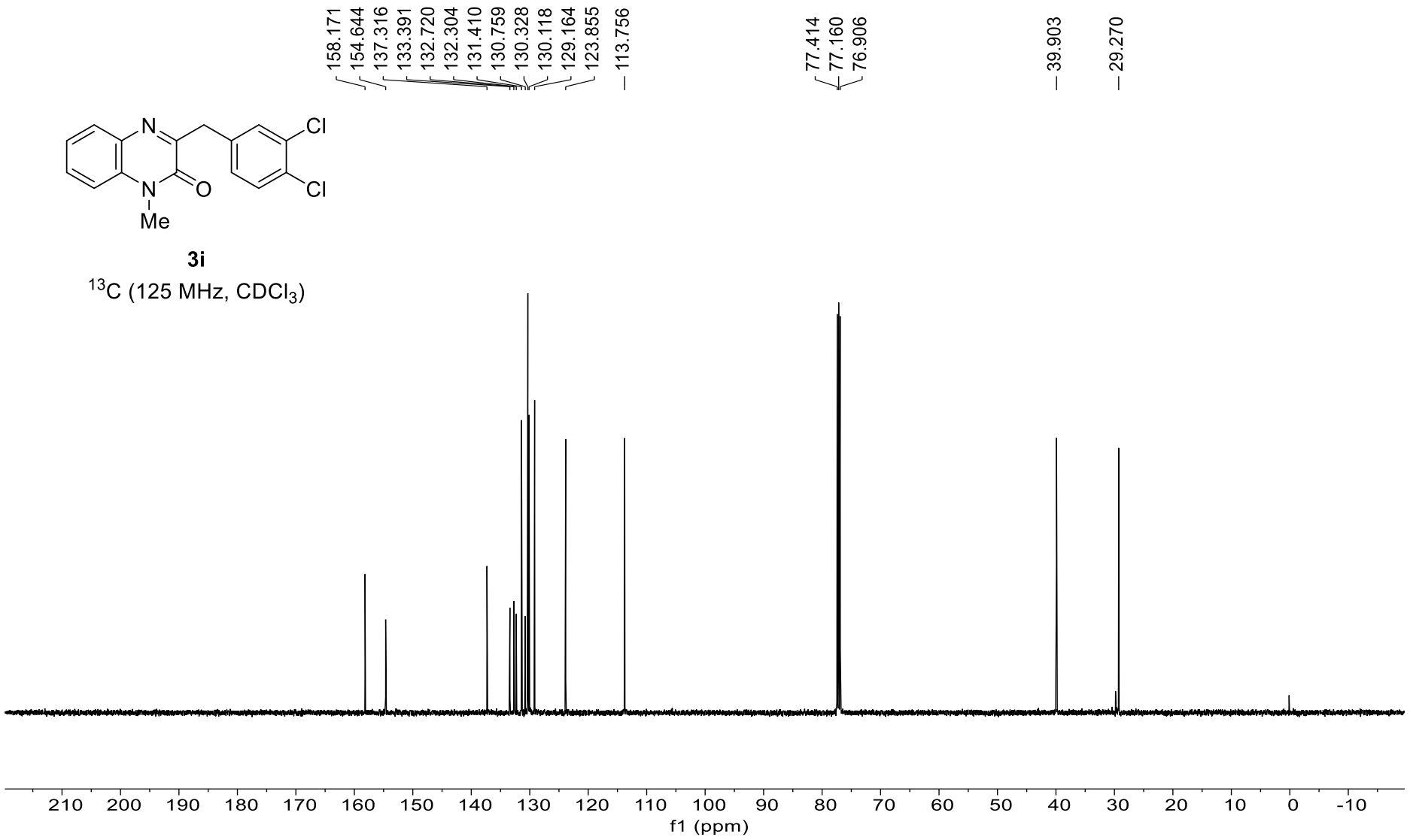
^{1}H (400 MHz, $CDCl_3$)

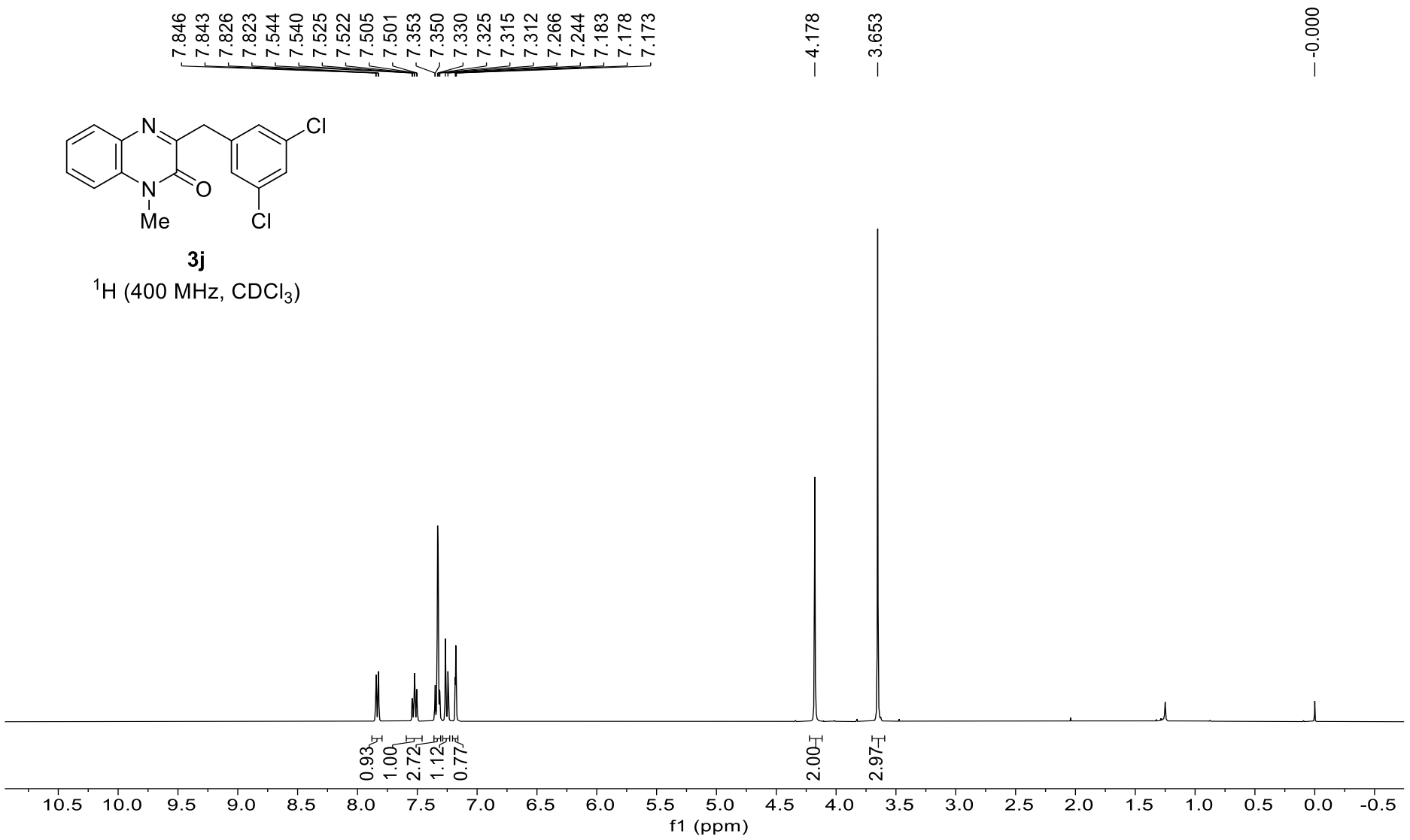

158.521
154.622
139.100
134.121
133.335
132.686
130.112
130.010
129.641
129.528
127.858
126.844
123.678
113.638
77.478
77.160
76.842
40.336
29.159
N
Cl
N
O
Me
3h
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

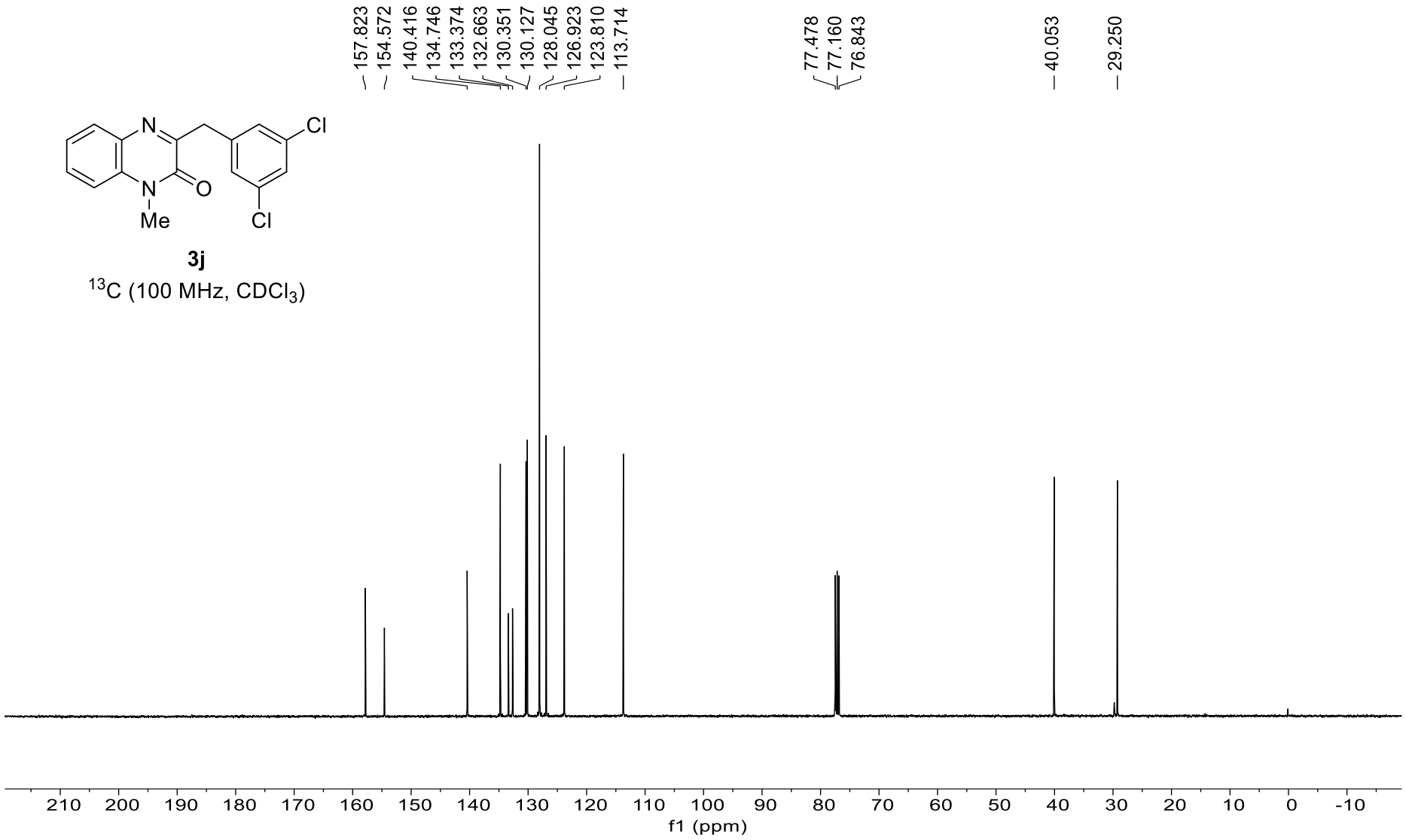


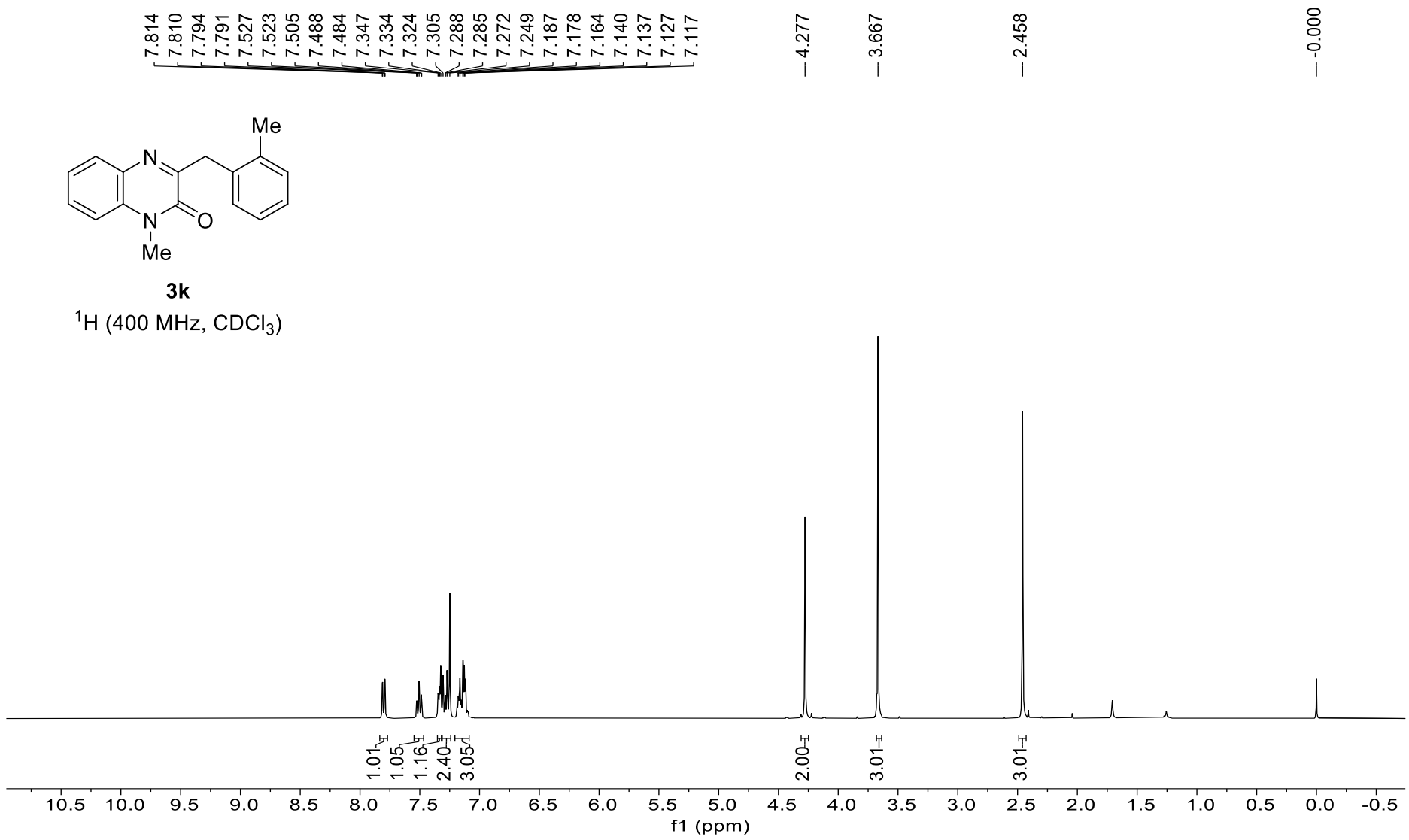
3i

^{1}H (500 MHz, $CDCl_3$)


158.171
154.644
137.316
133.391
132.720
132.304
131.410
130.759
130.328
130.118
129.164
123.855
113.756
77.414
77.160
76.906
39.903
29.270
N
N
O
Me
Cl
Cl
3i
13C (125 MHz, CDCl3)
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)

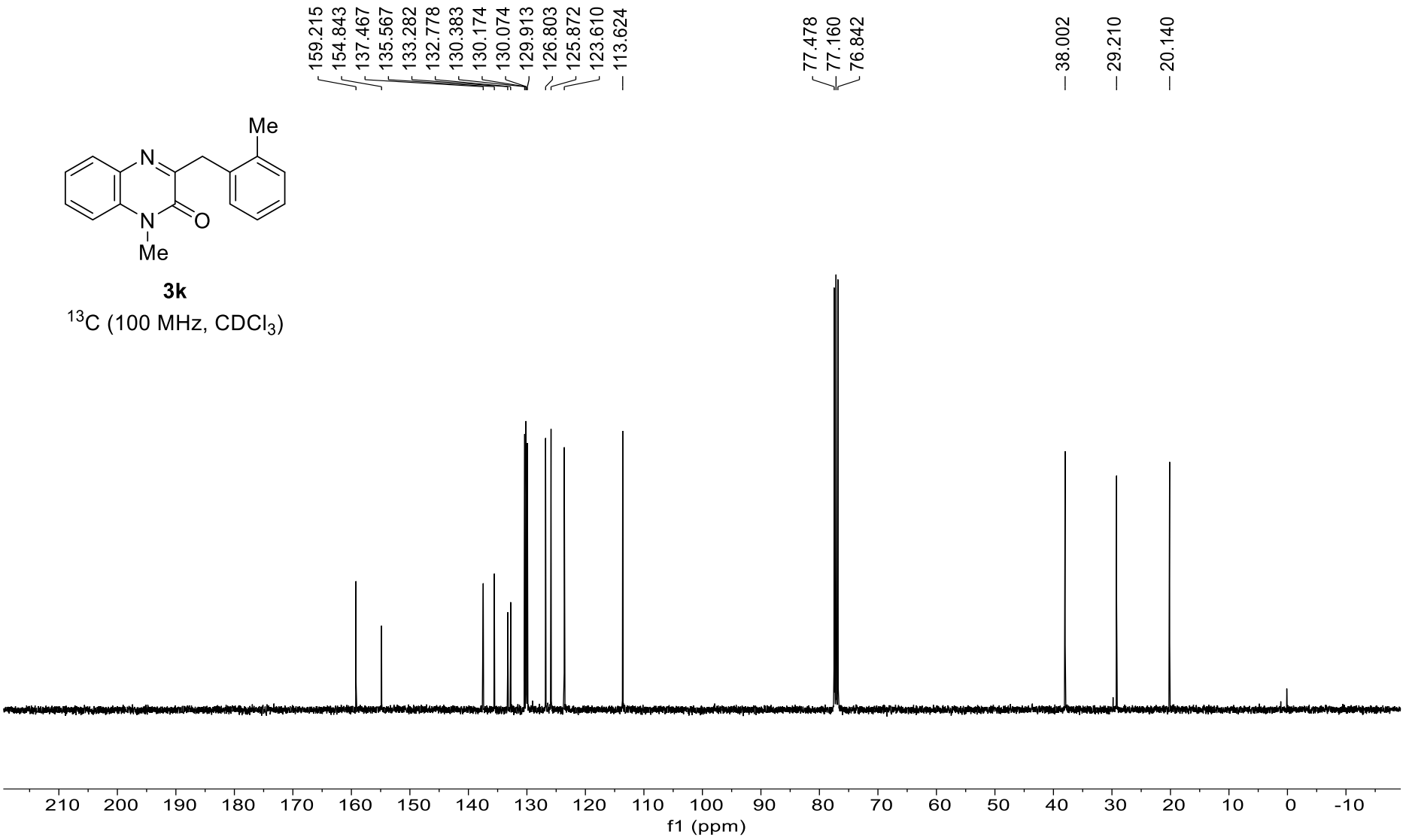

7.846
7.843
7.826
7.823
7.544
7.540
7.525
7.522
7.505
7.501
7.353
7.350
7.330
7.325
7.315
7.312
7.266
7.244
7.183
7.178
7.173
4.178
3.653
-0.000
N
Cl
N
O
Me
Cl
3j
1H (400 MHz, CDCl3)
0.93
1.00
2.72
1.12
0.77
2.00
2.97
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

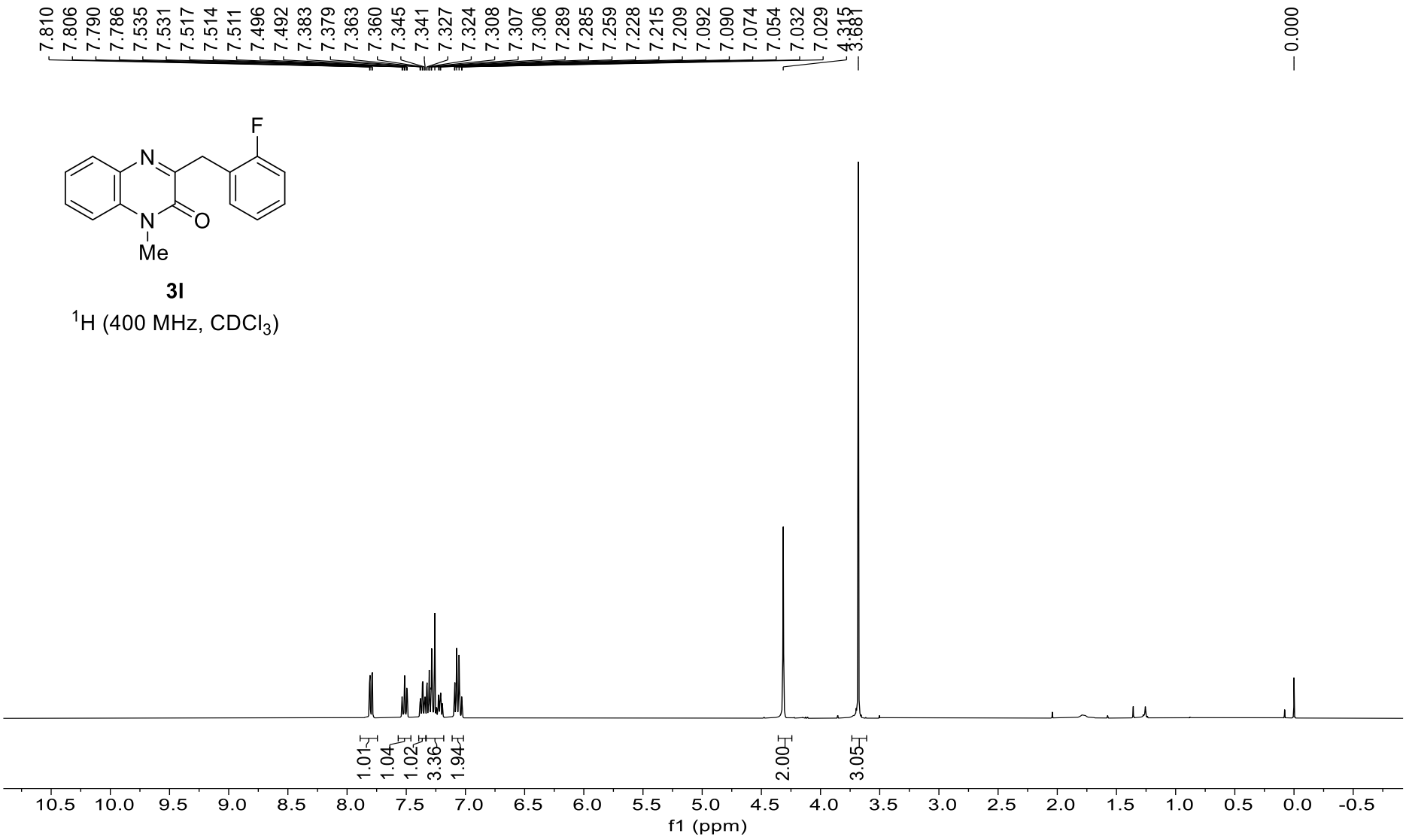

157.823
154.572
140.416
134.746
133.374
132.663
130.351
130.127
128.045
126.923
123.810
113.714
77.478
77.160
76.843
40.053
29.250
N
O
N
Me
Cl
Cl
3j
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



3k

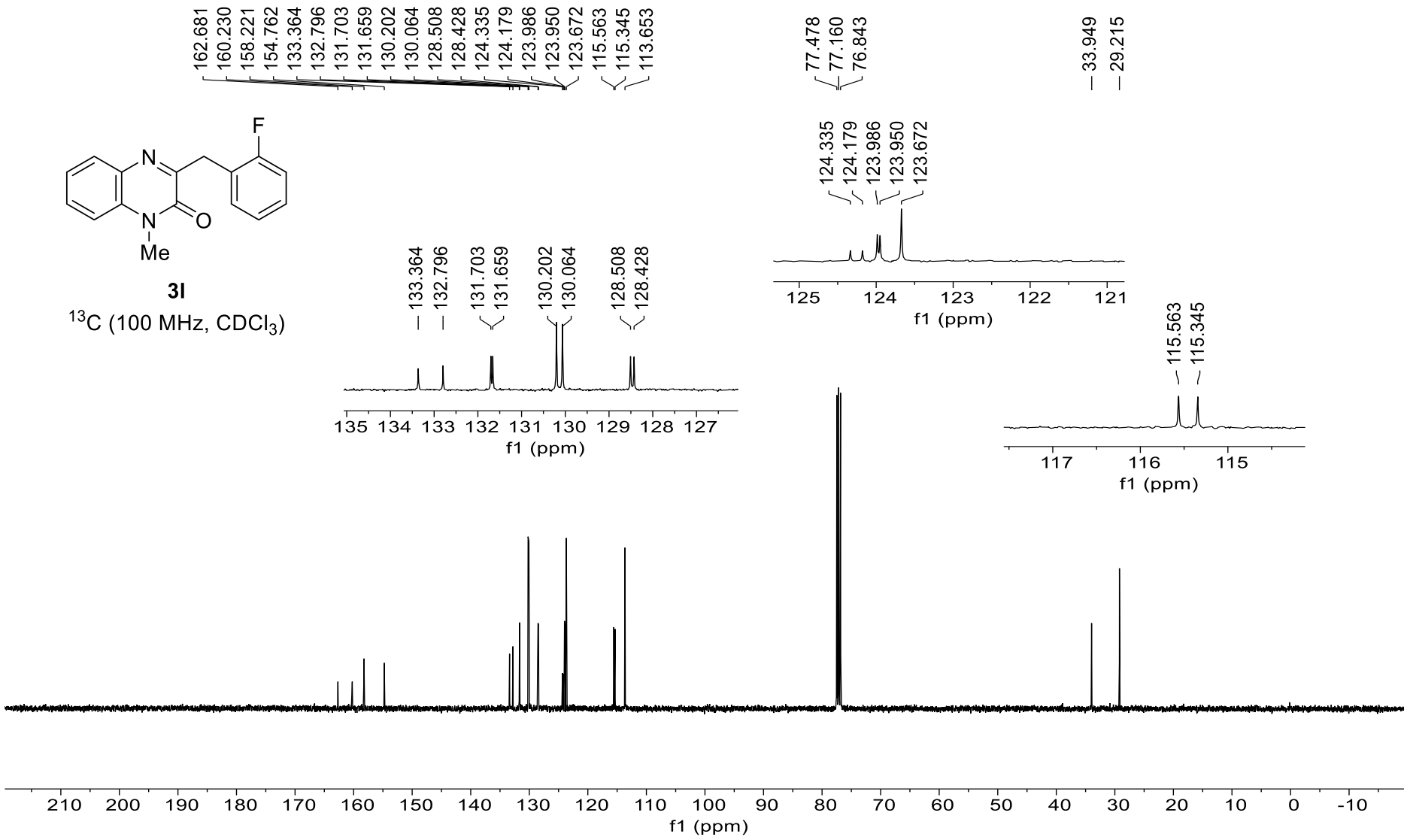
^{1}H (400 MHz, $CDCl_3$)

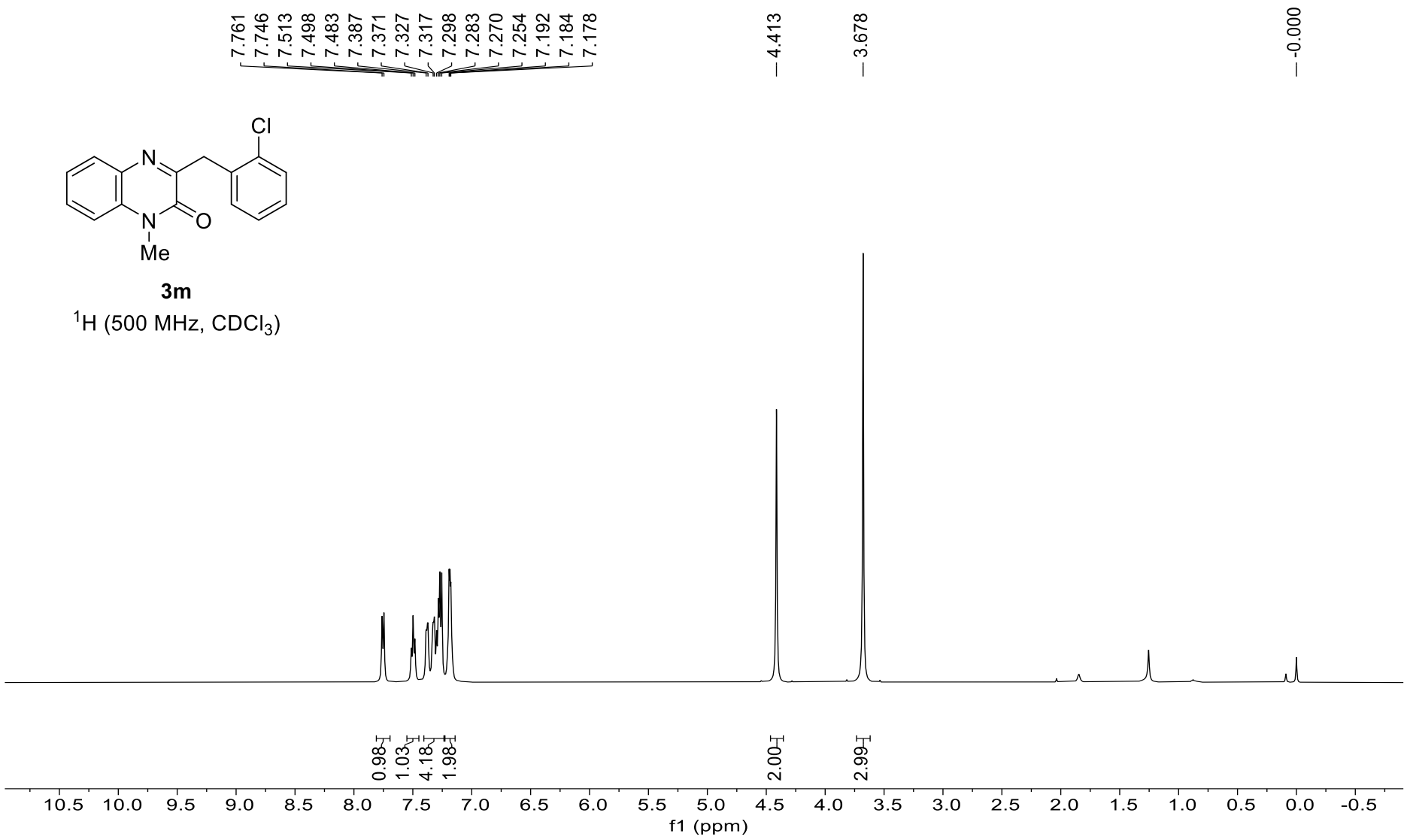

159.215
154.843
137.467
135.567
133.282
132.778
130.383
130.174
130.074
129.913
126.803
125.872
123.610
113.624
77.478
77.160
76.842
38.002
29.210
20.140
Me
N
N
O
Me
3k
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

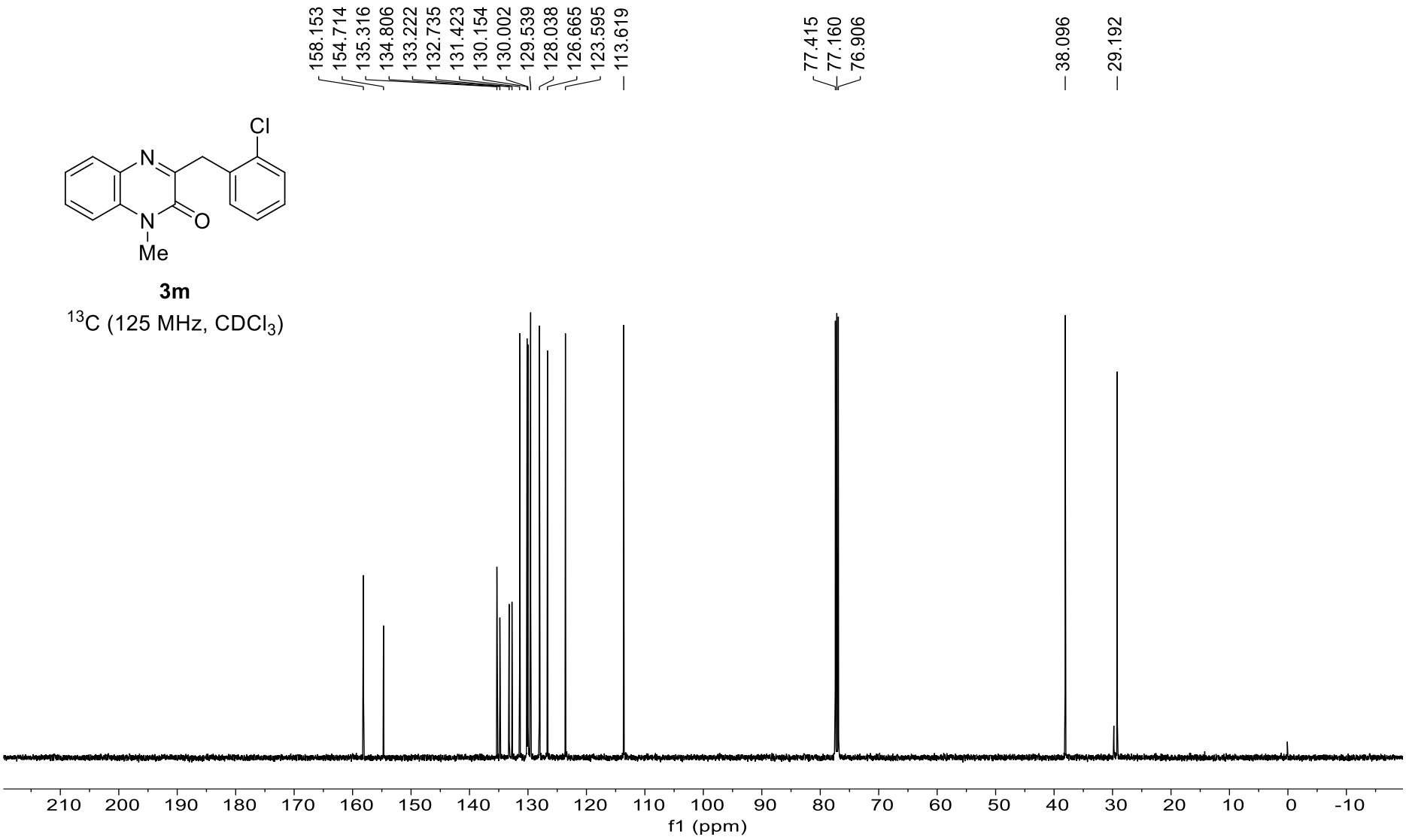


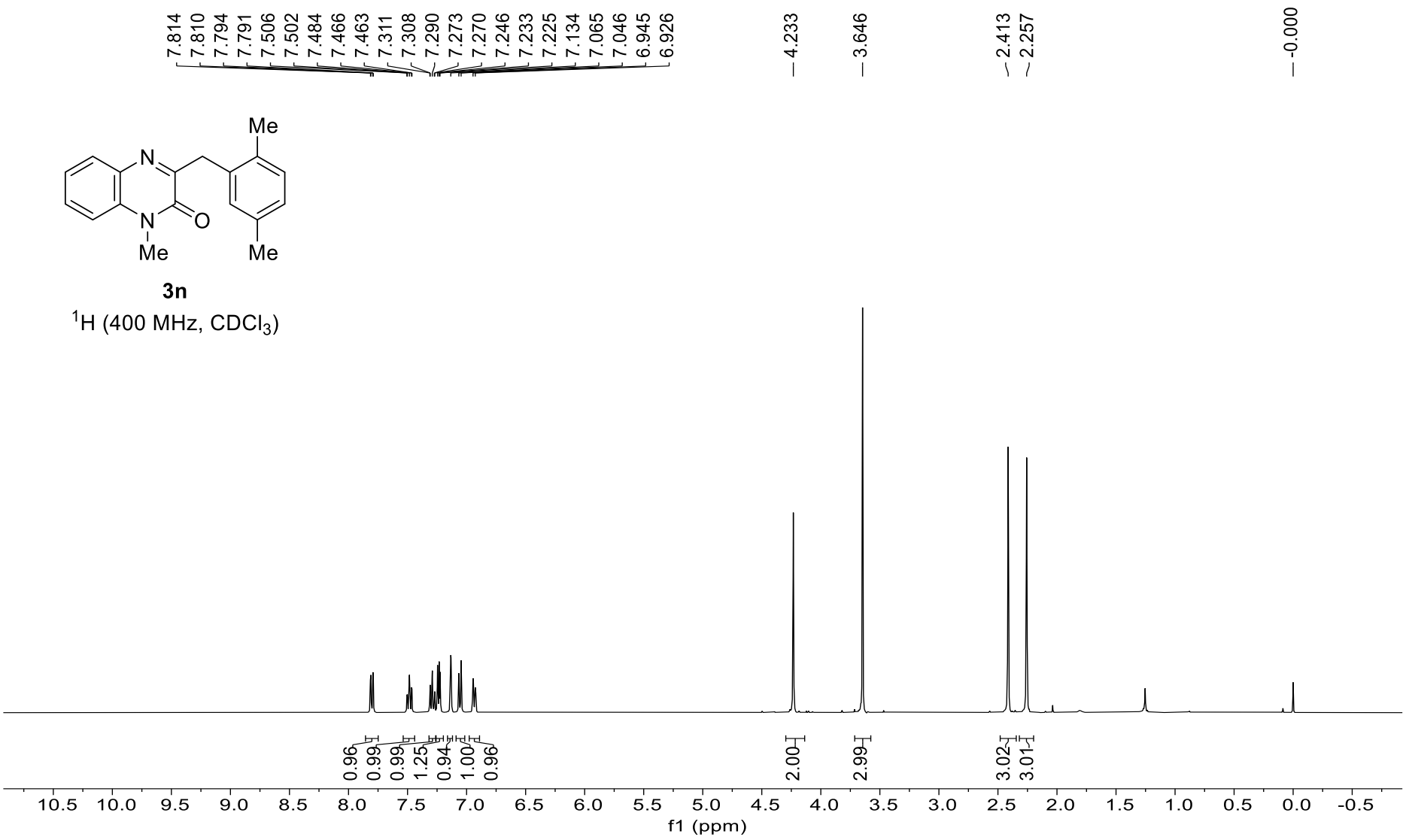
3l

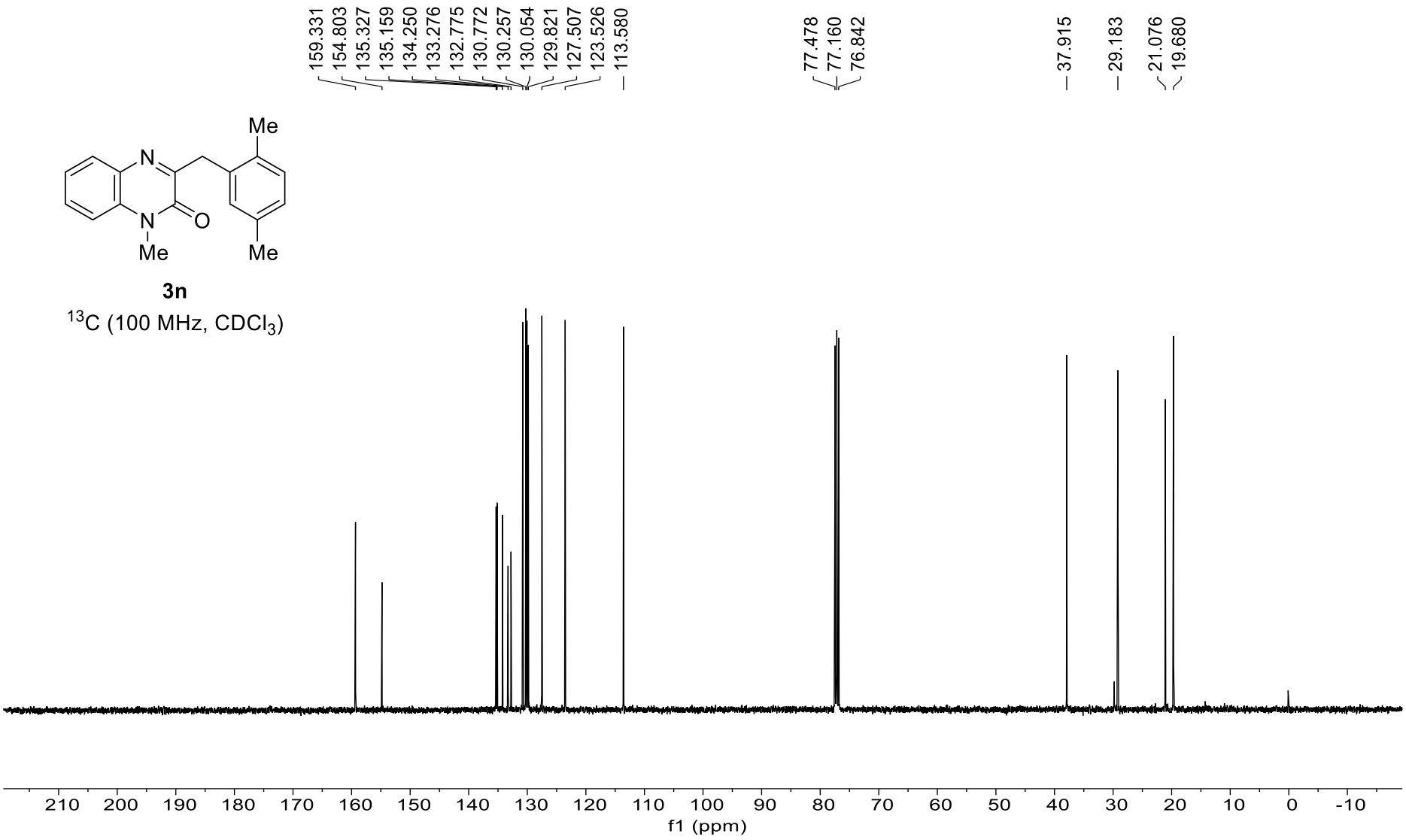
^{1}H (400 MHz, $CDCl_3$)

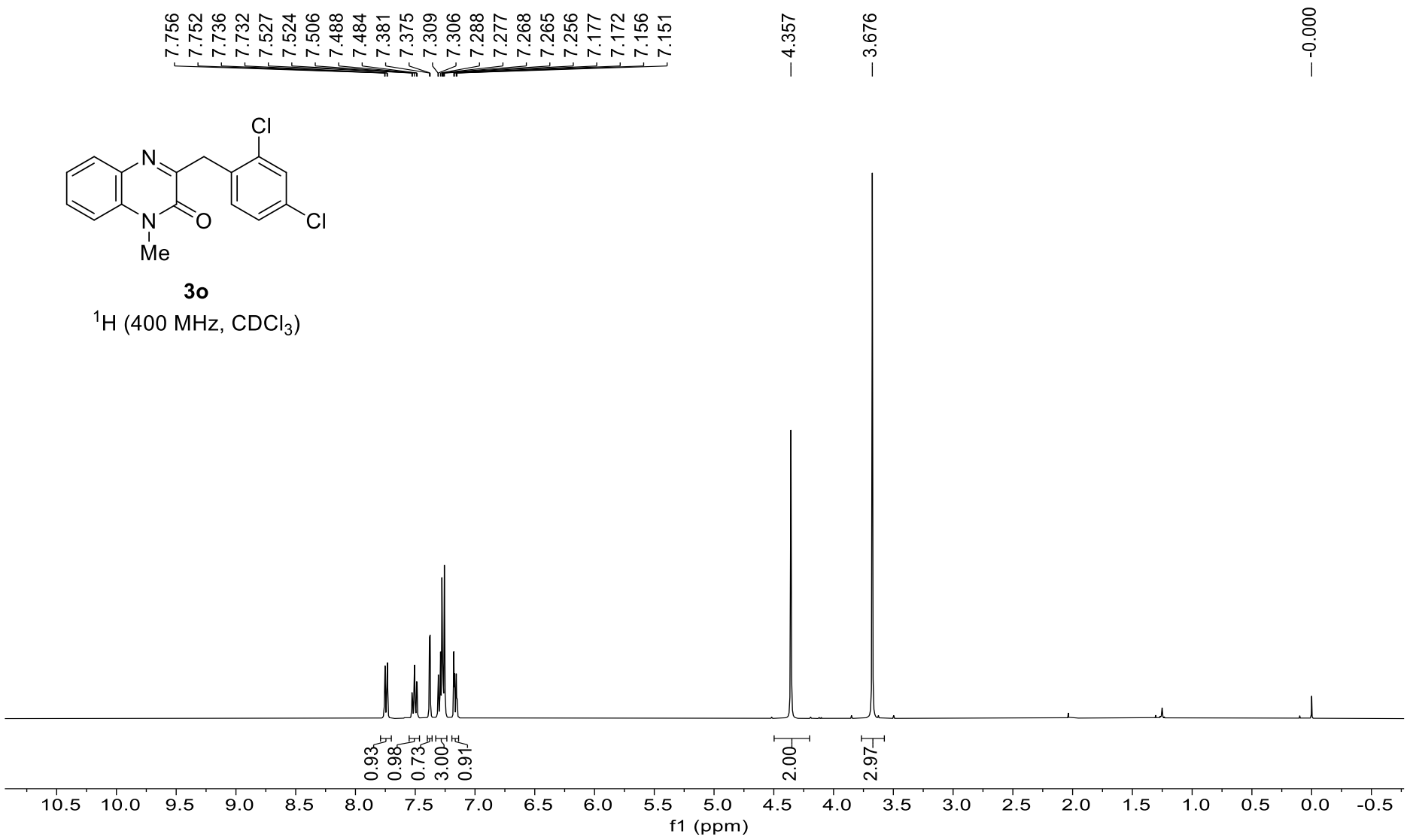

162.681
160.230
158.221
154.762
133.364
132.796
131.703
131.659
130.202
130.064
128.508
128.428
124.335
124.179
123.986
123.950
123.672
115.563
115.345
113.653
77.478
77.160
76.843
33.949
29.215
F
N
N
O
Me
3l
13C (100 MHz, CDCl3)
f1 (ppm)
f1 (ppm)
f1 (ppm)
f1 (ppm)

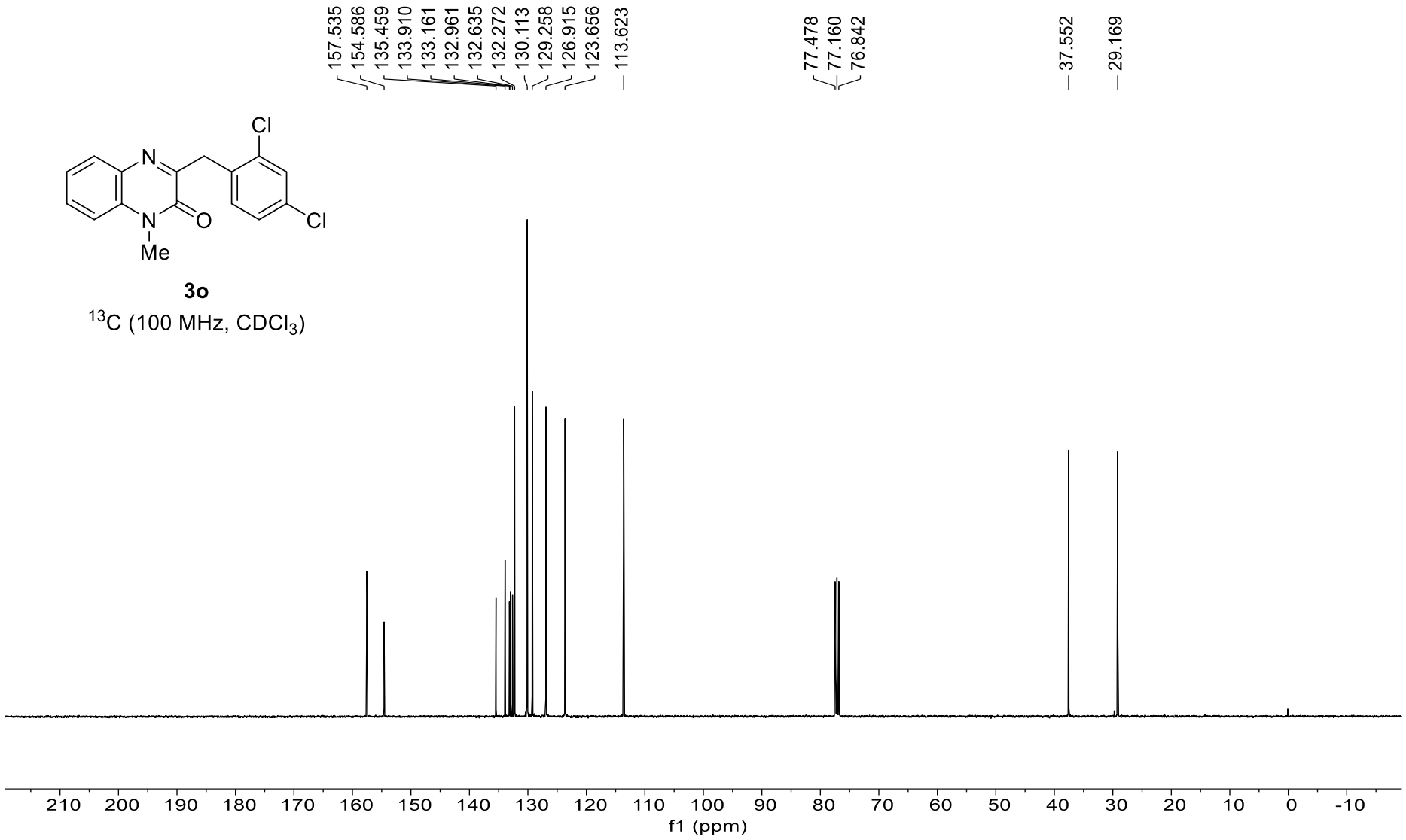

7.761
7.746
7.513
7.498
7.483
7.387
7.371
7.327
7.317
7.298
7.283
7.270
7.254
7.192
7.184
7.178
4.413
3.678
-0.000
Cl
N
N
O
Me
3m
1H (500 MHz, CDCl3)
0.98
1.03
4.18
1.98
2.00
2.99
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

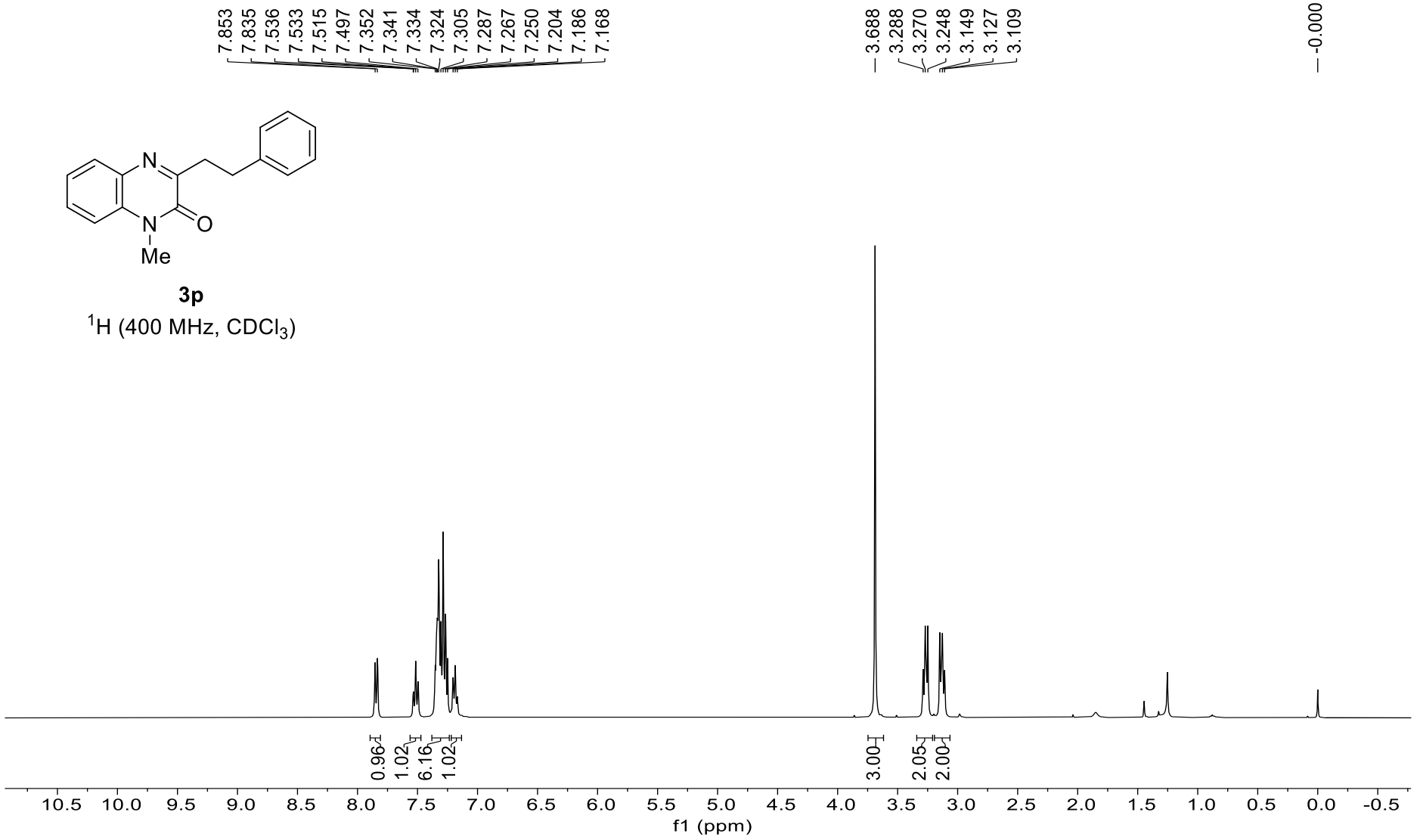

158.153
154.714
135.316
134.806
133.222
132.735
131.423
130.154
130.002
129.539
128.038
126.665
123.595
113.619
77.415
77.160
76.906
38.096
29.192
Cl
N
N
O
Me
3m
13C (125 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

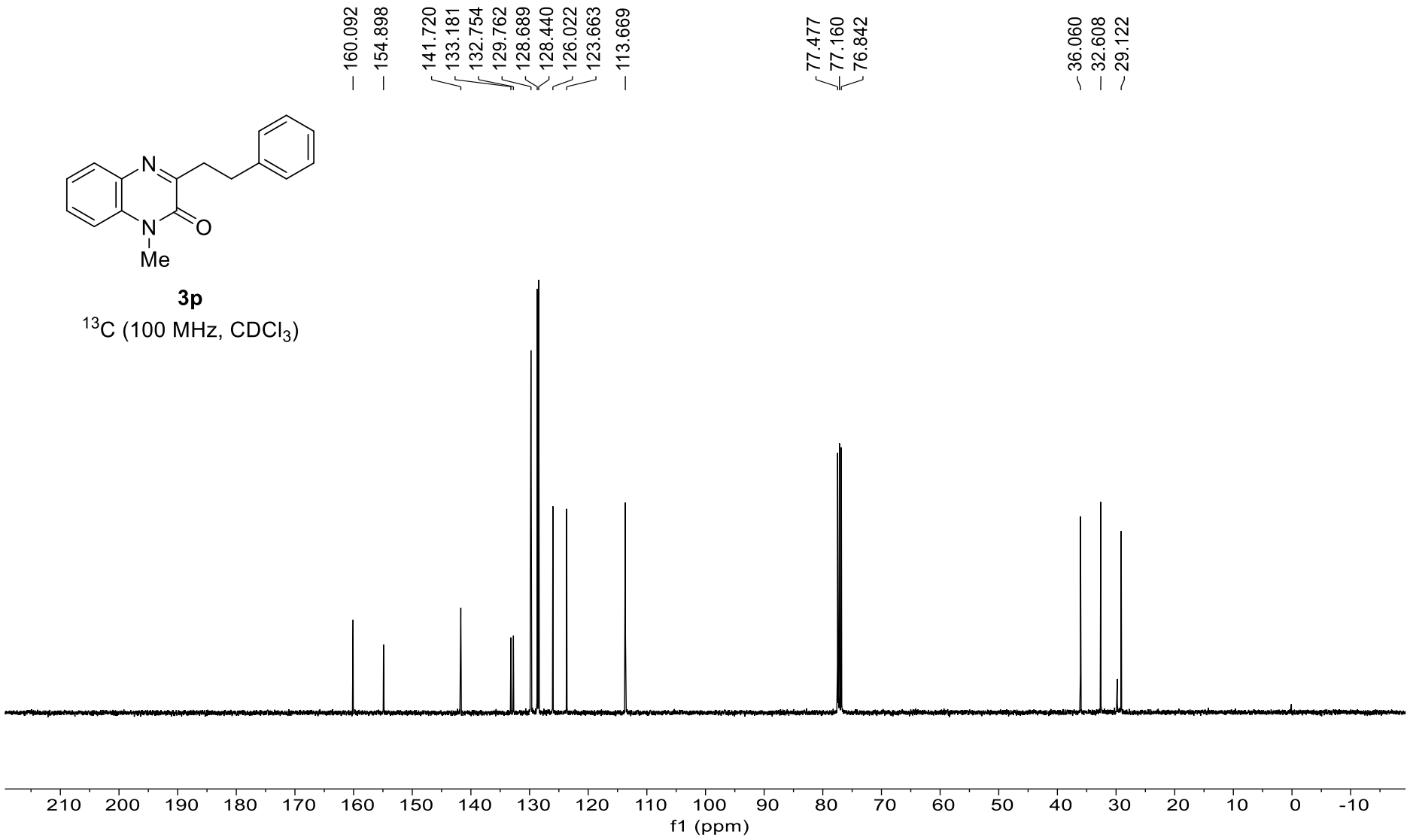

7.814
7.810
7.794
7.791
7.506
7.502
7.484
7.466
7.463
7.311
7.308
7.290
7.273
7.270
7.246
7.233
7.225
7.134
7.065
7.046
6.945
6.926
4.233
3.646
2.413
2.257
-0.000
Me
N
N
O
Me
Me
3n
1H (400 MHz, CDCl3)
0.96
0.99
0.99
1.25
0.94
1.00
0.96
2.00
2.99
3.02
3.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

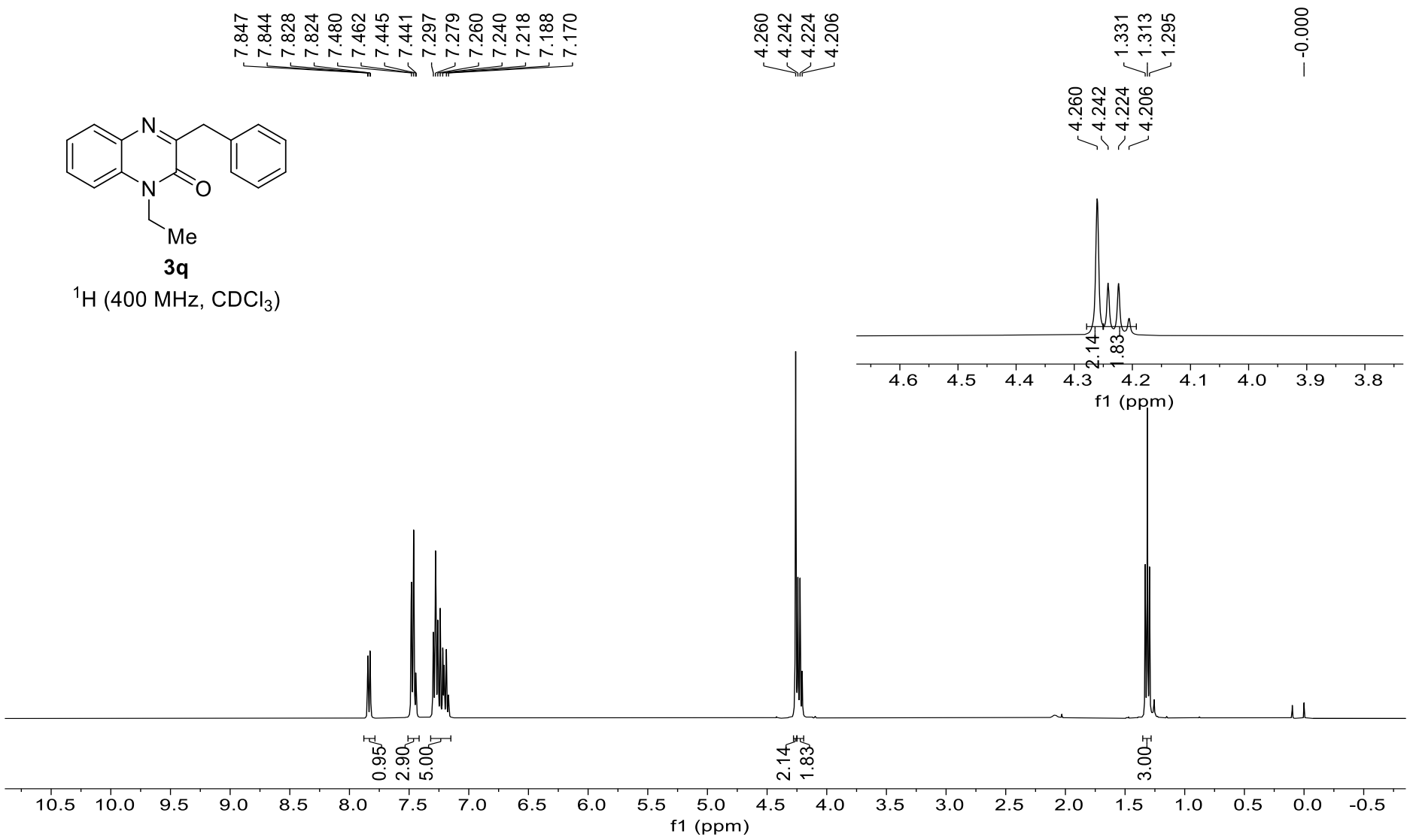

159.331
154.803
135.327
135.159
134.250
133.276
132.775
130.772
130.257
130.054
129.821
127.507
123.526
113.580
77.478
77.160
76.842
37.915
29.183
21.076
19.680
Me
N
N
O
Me
Me
3n
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

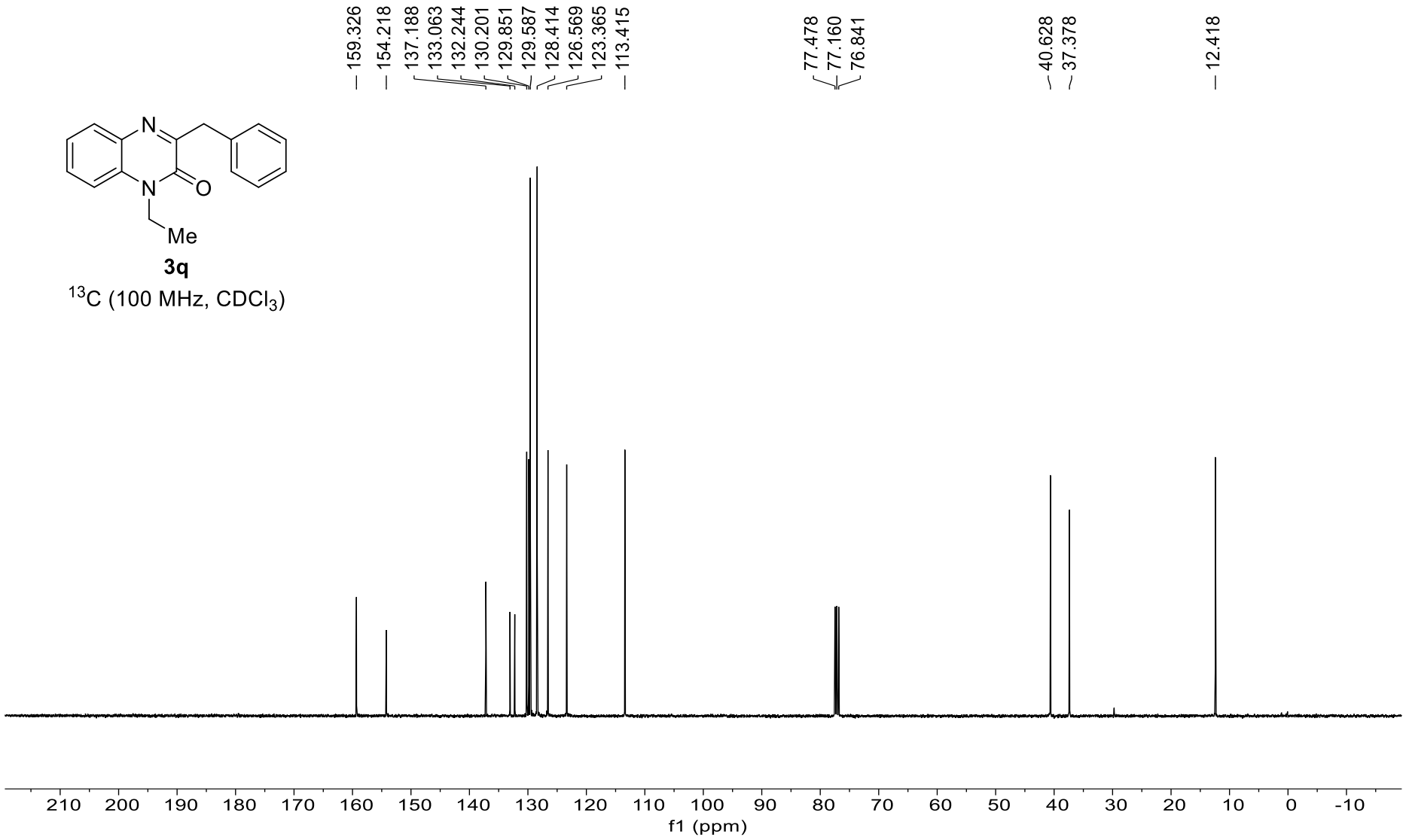

7.756
7.752
7.736
7.732
7.527
7.524
7.506
7.488
7.484
7.381
7.375
7.309
7.306
7.288
7.277
7.268
7.265
7.256
7.177
7.172
7.156
7.151
4.357
3.676
-0.000
Cl
N
N
O
Me
Cl
3o
1H (400 MHz, CDCl3)
0.93
0.98
0.73
3.00
0.91
2.00
2.97
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

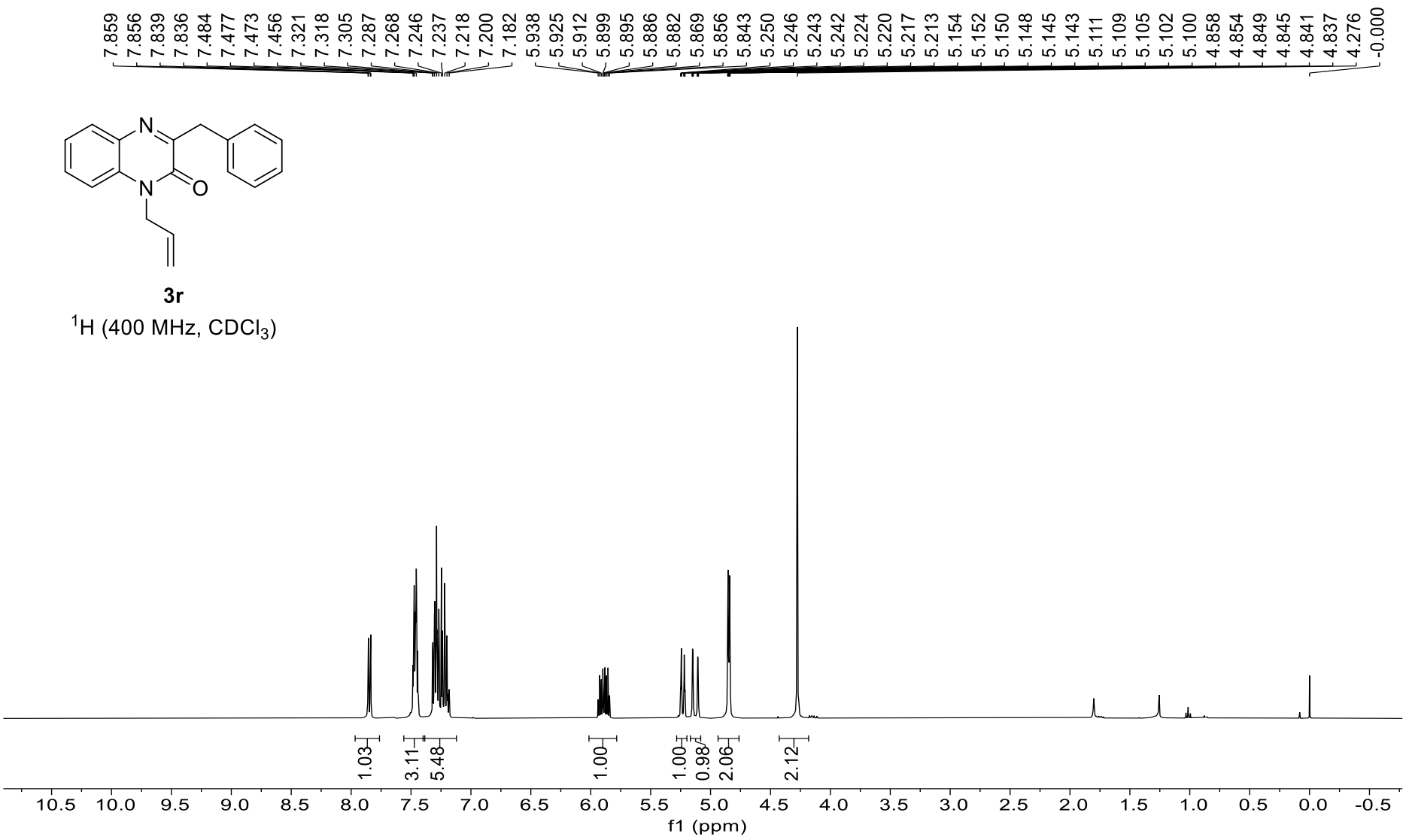

157.535
154.586
135.459
133.910
133.161
132.961
132.635
132.272
130.113
129.258
126.915
123.656
113.623
77.478
77.160
76.842
37.552
29.169
Cl
N
N
O
Me
Cl
3o
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

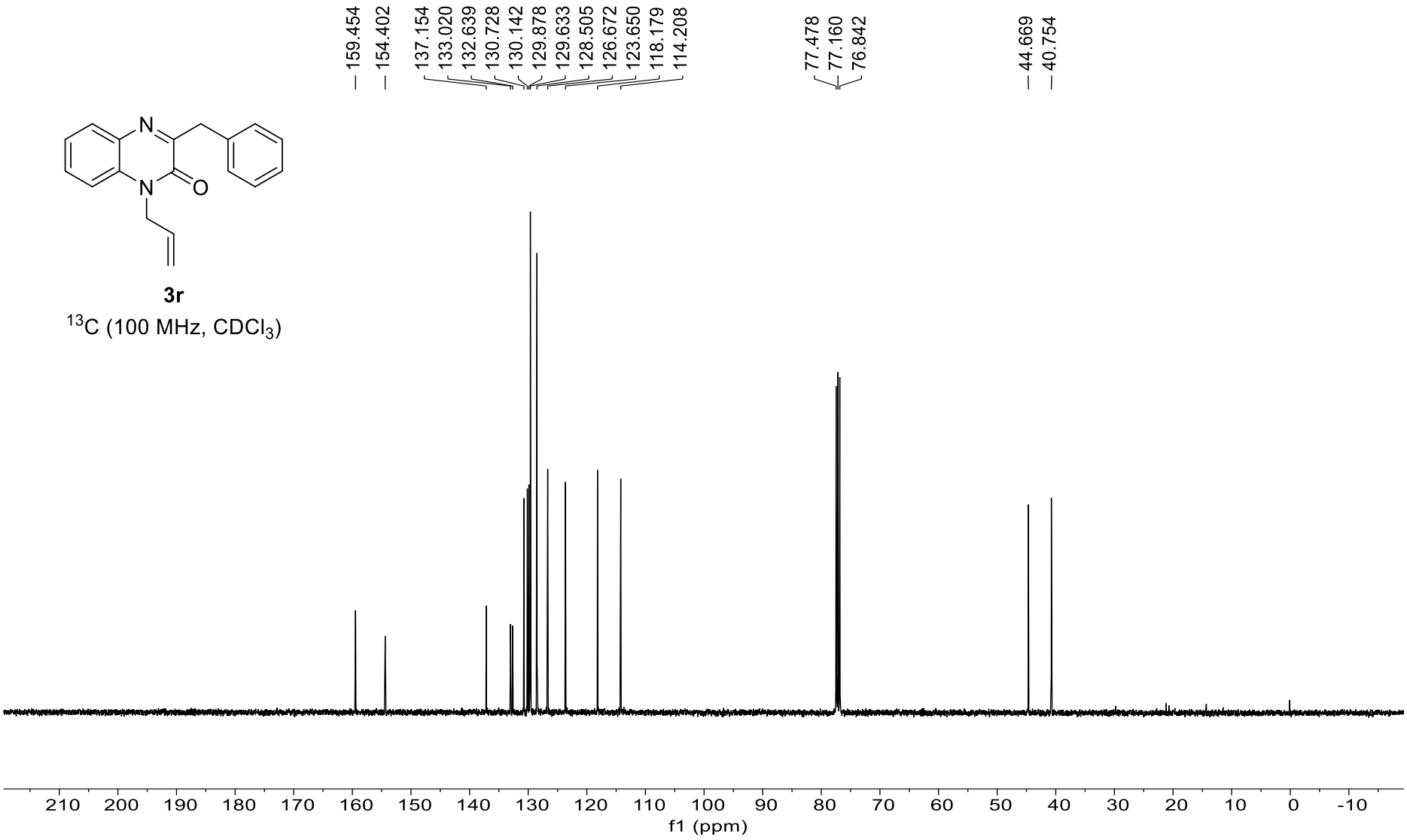

7.853
7.835
7.536
7.533
7.515
7.497
7.352
7.341
7.334
7.324
7.305
7.287
7.267
7.250
7.204
7.186
7.168
3.688
3.288
3.270
3.248
3.149
3.127
3.109
-0.000
N
N
O
Me
3p
1H (400 MHz, CDCl3)
0.96
1.02
6.16
1.02
3.00
2.05
2.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

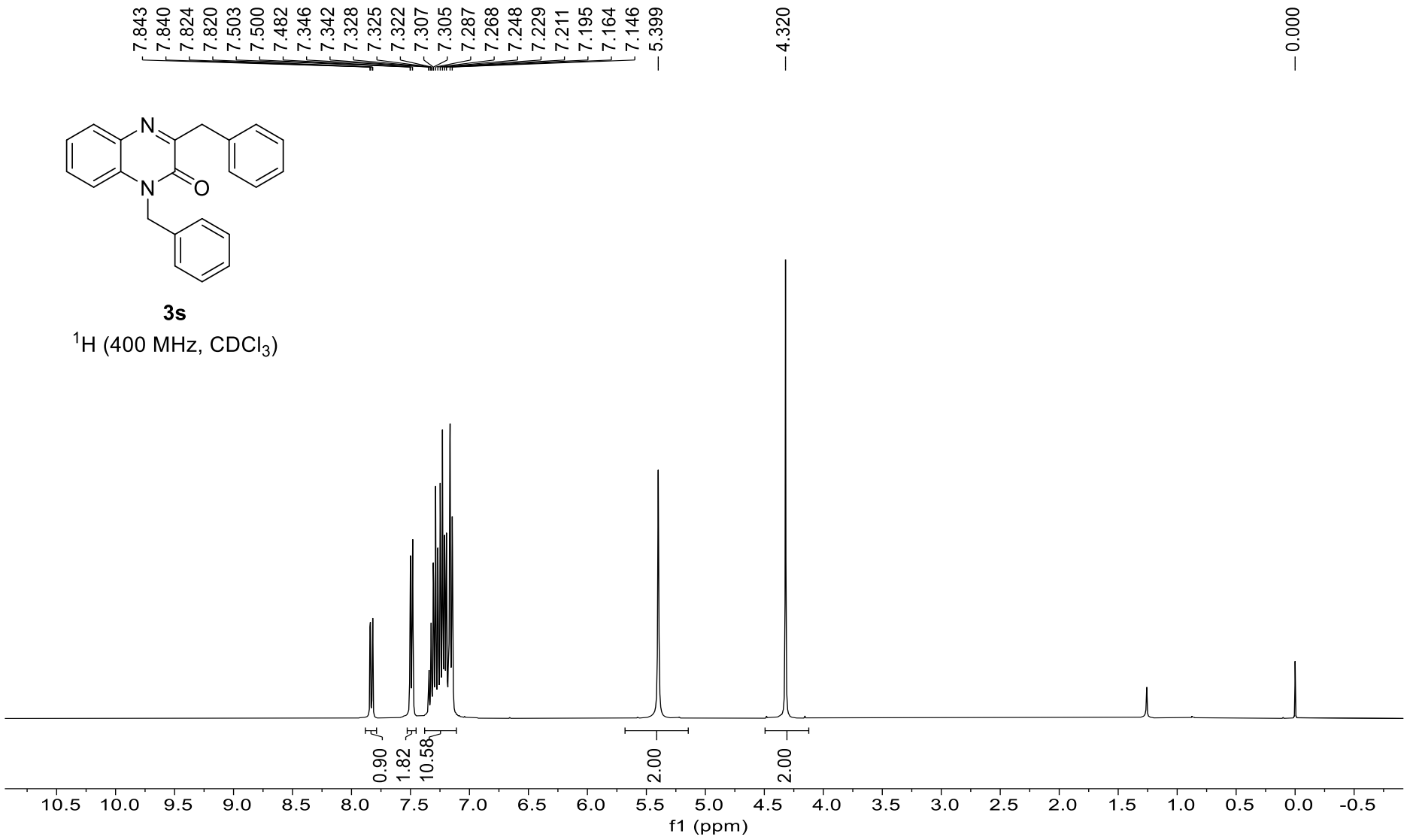

160.092
154.898
141.720
133.181
132.754
129.762
128.689
128.440
126.022
123.663
113.669
77.477
77.160
76.842
36.060
32.608
29.122
N
N
O
Me
3p
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.847
7.844
7.828
7.824
7.480
7.462
7.445
7.441
7.297
7.279
7.260
7.240
7.218
7.188
7.170
4.260
4.242
4.224
4.206
1.331
1.313
1.295
-0.000
N
N
O
Me
3q
1H (400 MHz, CDCl3)
2.14
1.83
f1 (ppm)
0.95
2.90
5.00
2.14
1.83
3.00
f1 (ppm)


159.326
154.218
137.188
133.063
132.244
130.201
129.851
129.587
128.414
126.569
123.365
113.415
77.478
77.160
76.841
40.628
37.378
12.418
N
N
O
Me
3q
13C (100 MHz, CDCl3)
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)

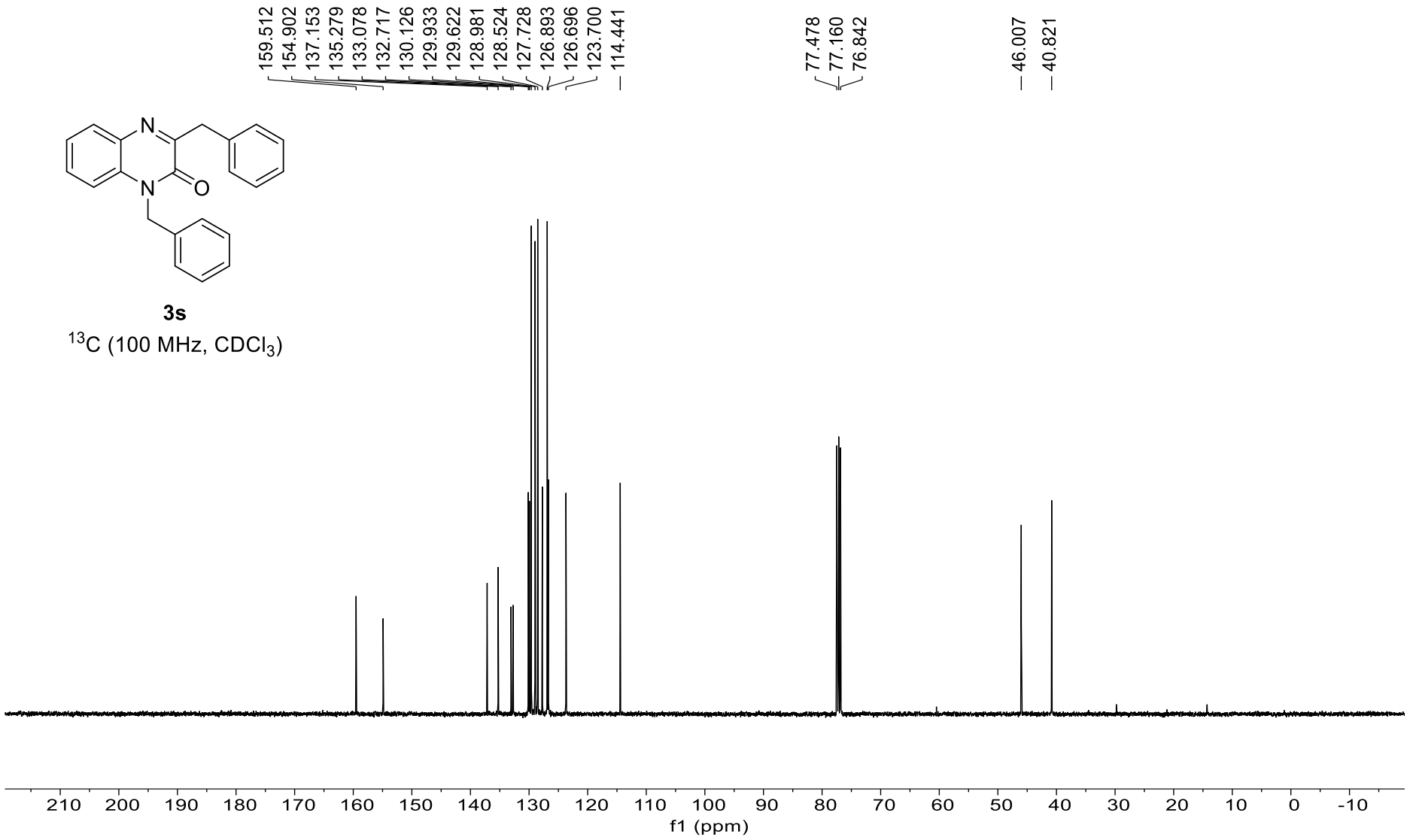

7.859
7.856
7.839
7.836
7.484
7.477
7.473
7.456
7.321
7.318
7.305
7.287
7.268
7.246
7.237
7.218
7.200
7.182
5.938
5.925
5.912
5.899
5.895
5.886
5.882
5.869
5.856
5.843
5.250
5.246
5.243
5.242
5.224
5.220
5.217
5.213
5.154
5.152
5.150
5.148
5.145
5.143
5.111
5.109
5.105
5.102
5.100
4.858
4.854
4.849
4.845
4.841
4.837
4.276
-0.000
N
N
O
3r
1H (400 MHz, CDCl3)
1.03
3.11
5.48
1.00
1.00
0.98
2.06
2.12
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
f1 (ppm)

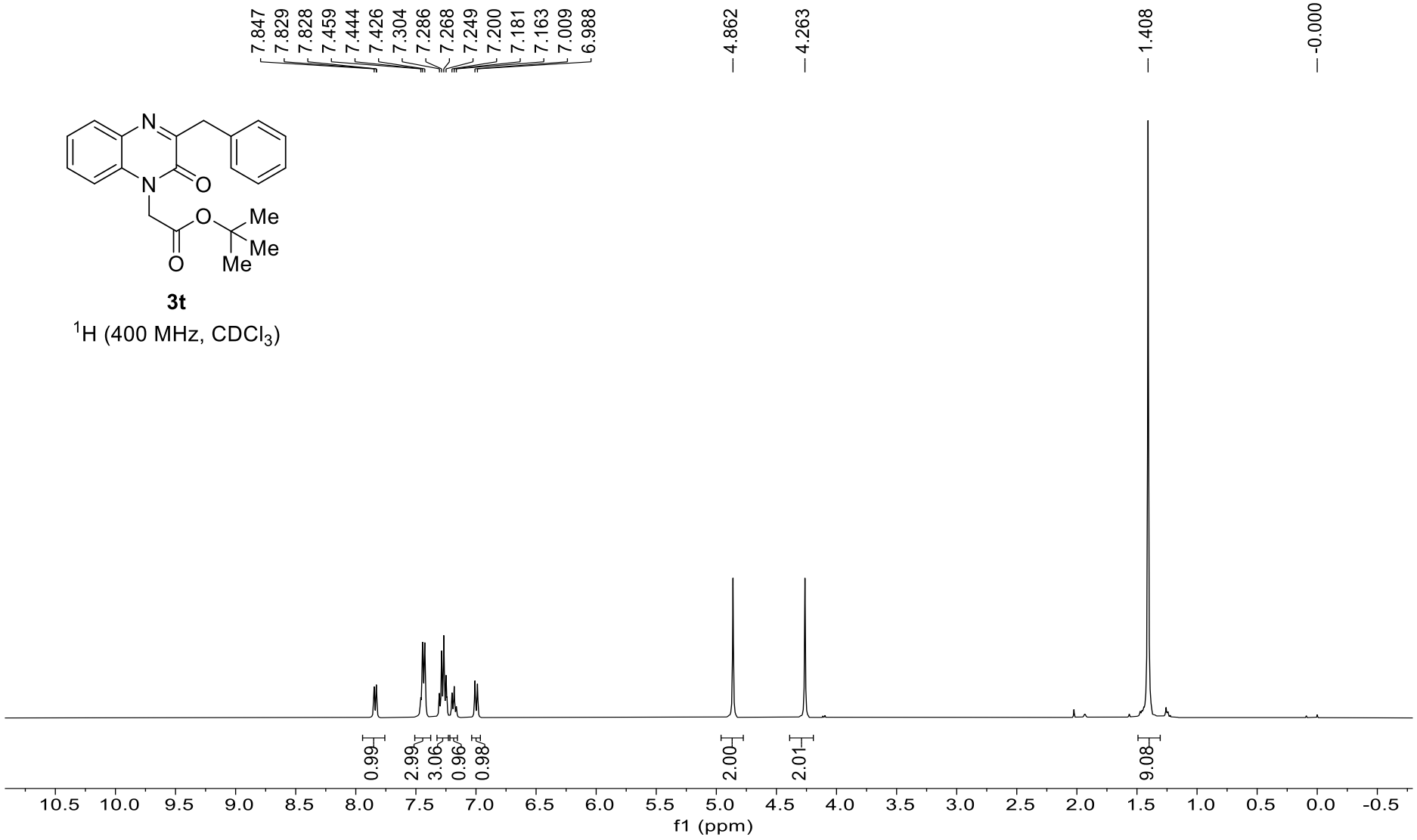

159.454
154.402
137.154
133.020
132.639
130.728
130.142
129.878
129.633
128.505
126.672
123.650
118.179
114.208
77.478
77.160
76.842
44.669
40.754
N
N
O
3r
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

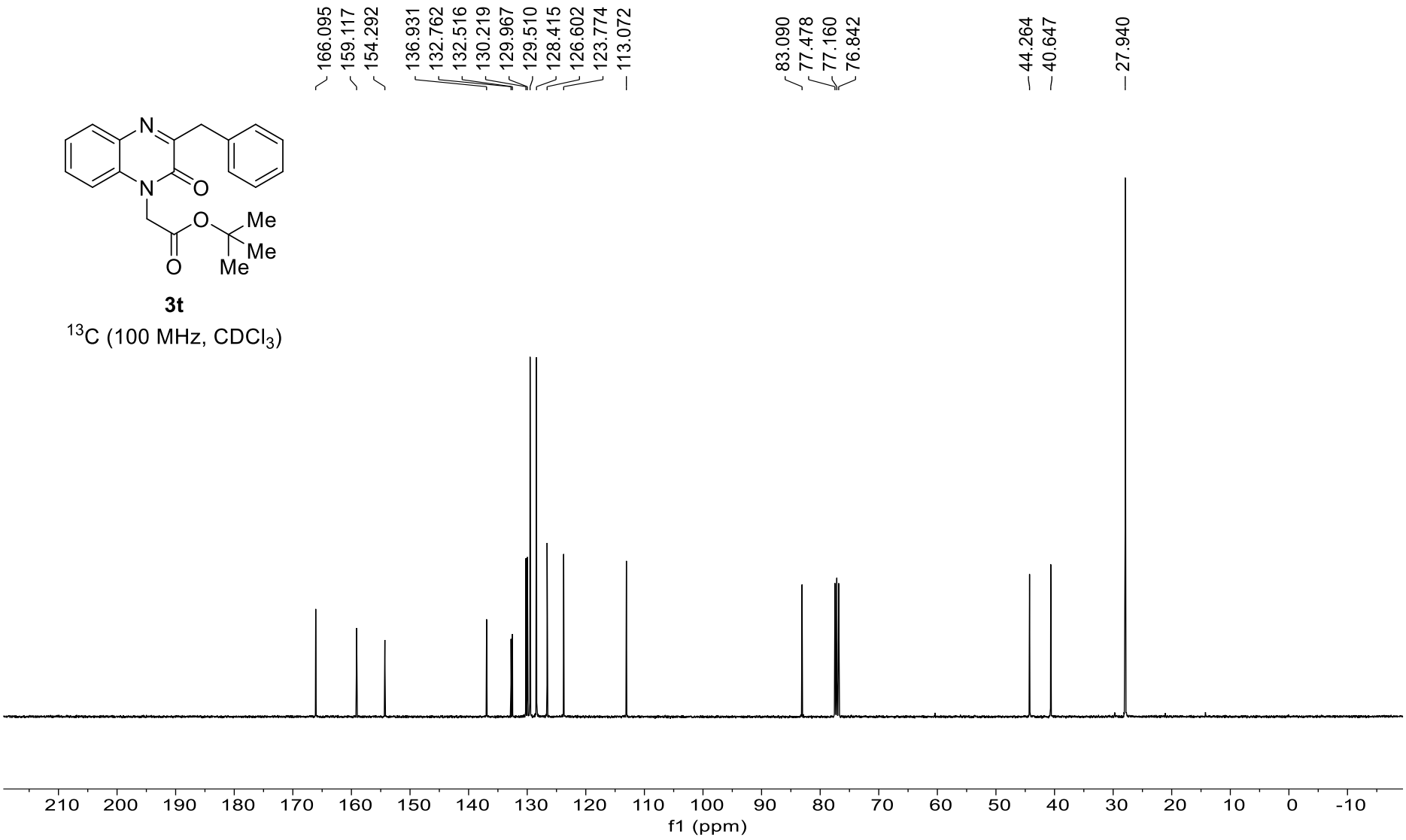


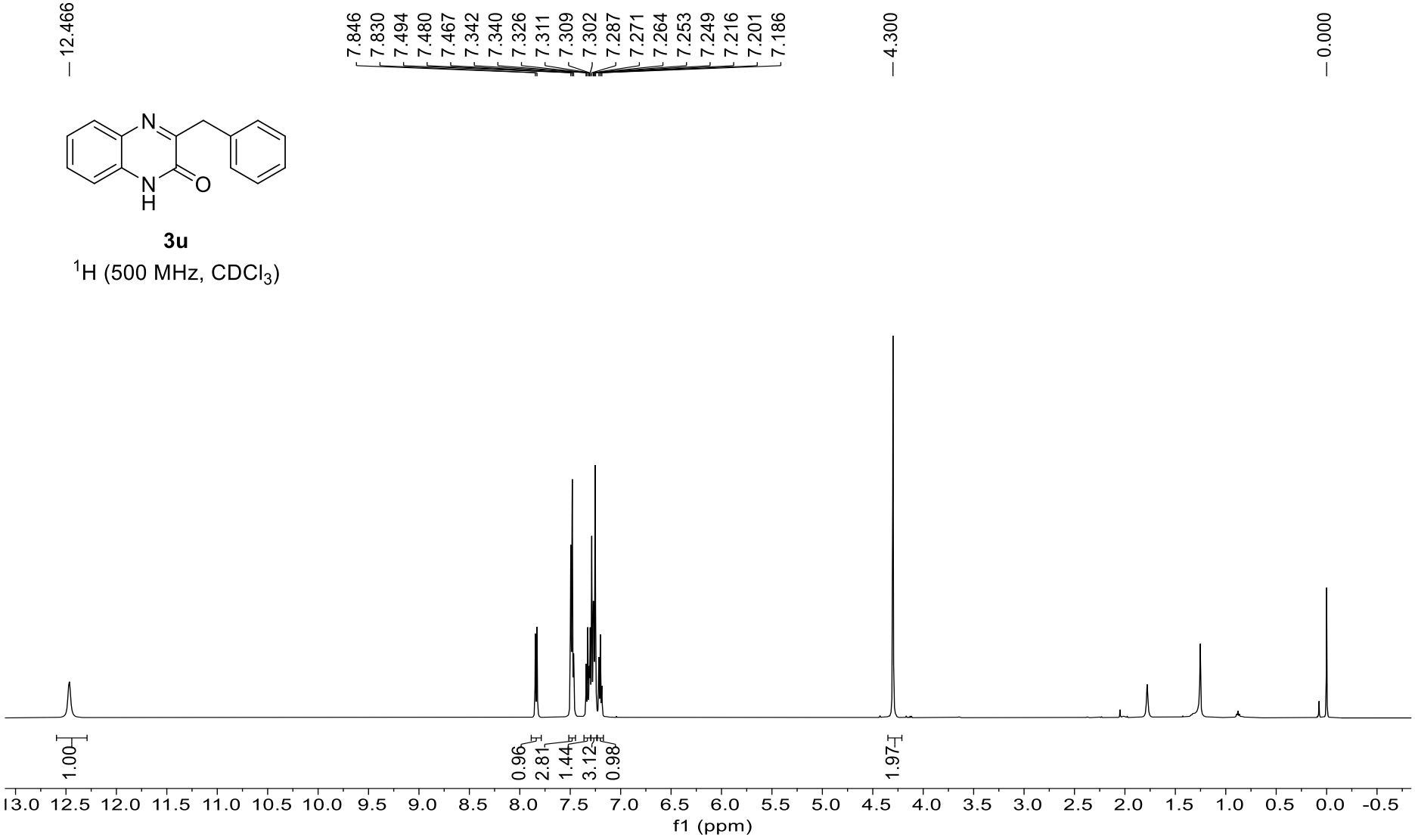
3s

^{1}H (400 MHz, $CDCl_3$)


159.512
154.902
137.153
135.279
133.078
132.717
130.126
129.933
129.622
128.981
128.524
127.728
126.893
126.696
123.700
114.441
77.478
77.160
76.842
46.007
40.821
N
N
O
3s
13C (100 MHz, CDCl3)
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)

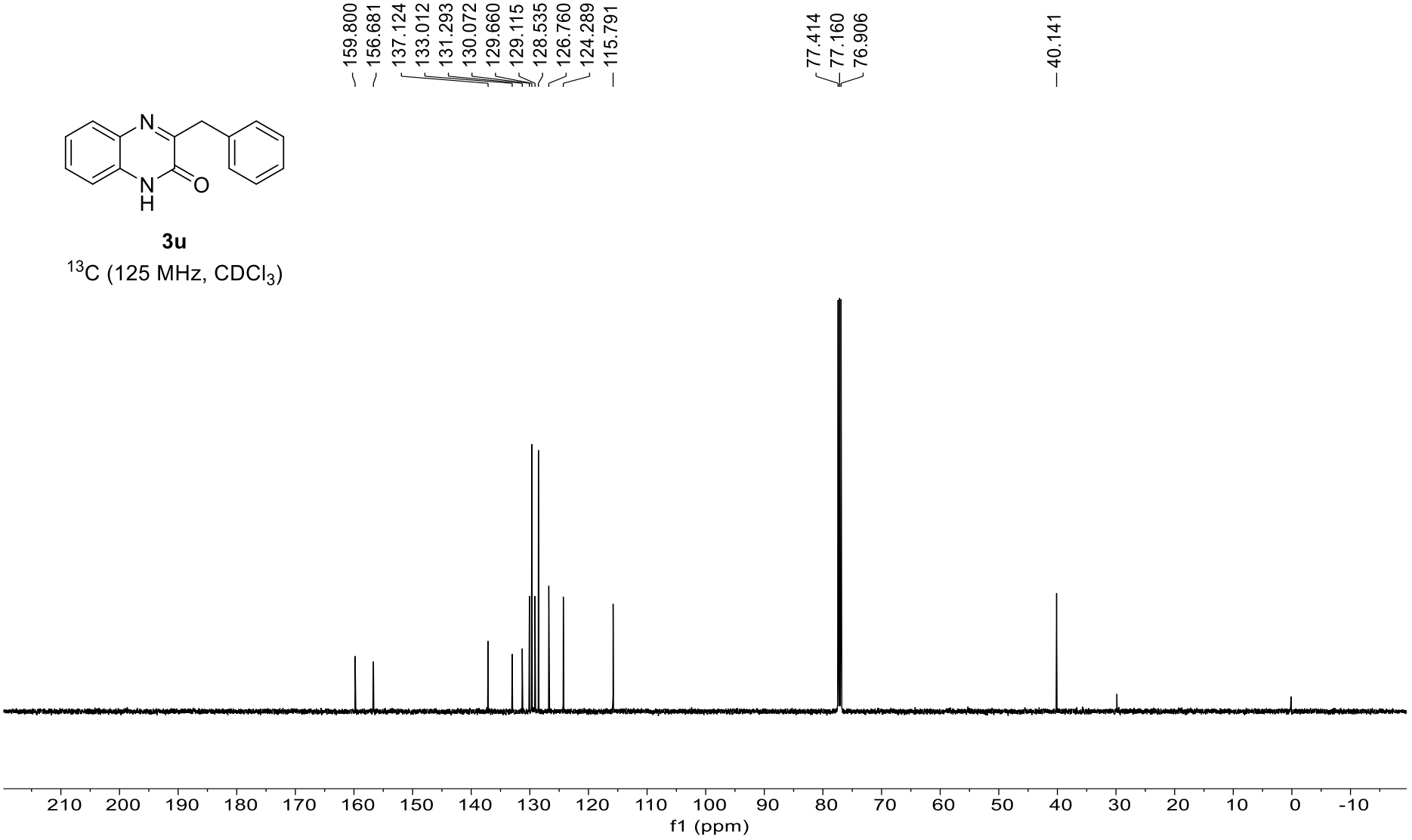

7.847
7.829
7.828
7.459
7.444
7.426
7.304
7.286
7.268
7.249
7.200
7.181
7.163
7.009
6.988
4.862
4.263
1.408
-0.000
N
N
O
O
O
Me
Me
Me
3t
1H (400 MHz, CDCl3)
0.99
2.99
3.06
0.96
0.98
2.00
2.01
9.08
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


166.095
159.117
154.292
136.931
132.762
132.516
130.219
129.967
129.510
128.415
126.602
123.774
113.072
83.090
77.478
77.160
76.842
44.264
40.647
27.940
N
N
O
O
O
Me
Me
Me
3t
13C (100 MHz, CDCl3)
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)



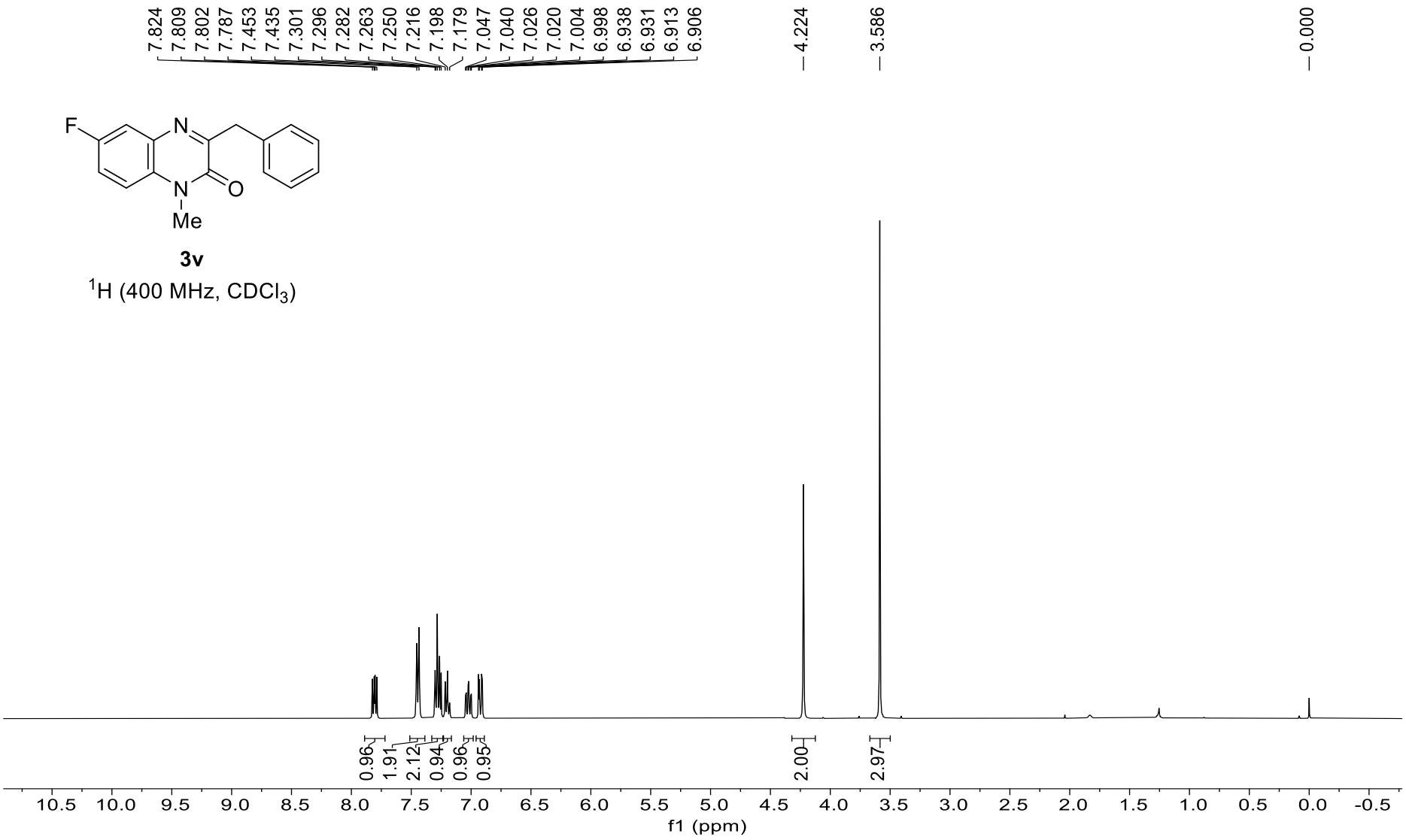
3u

^{1}H (500 MHz, $CDCl_3$)



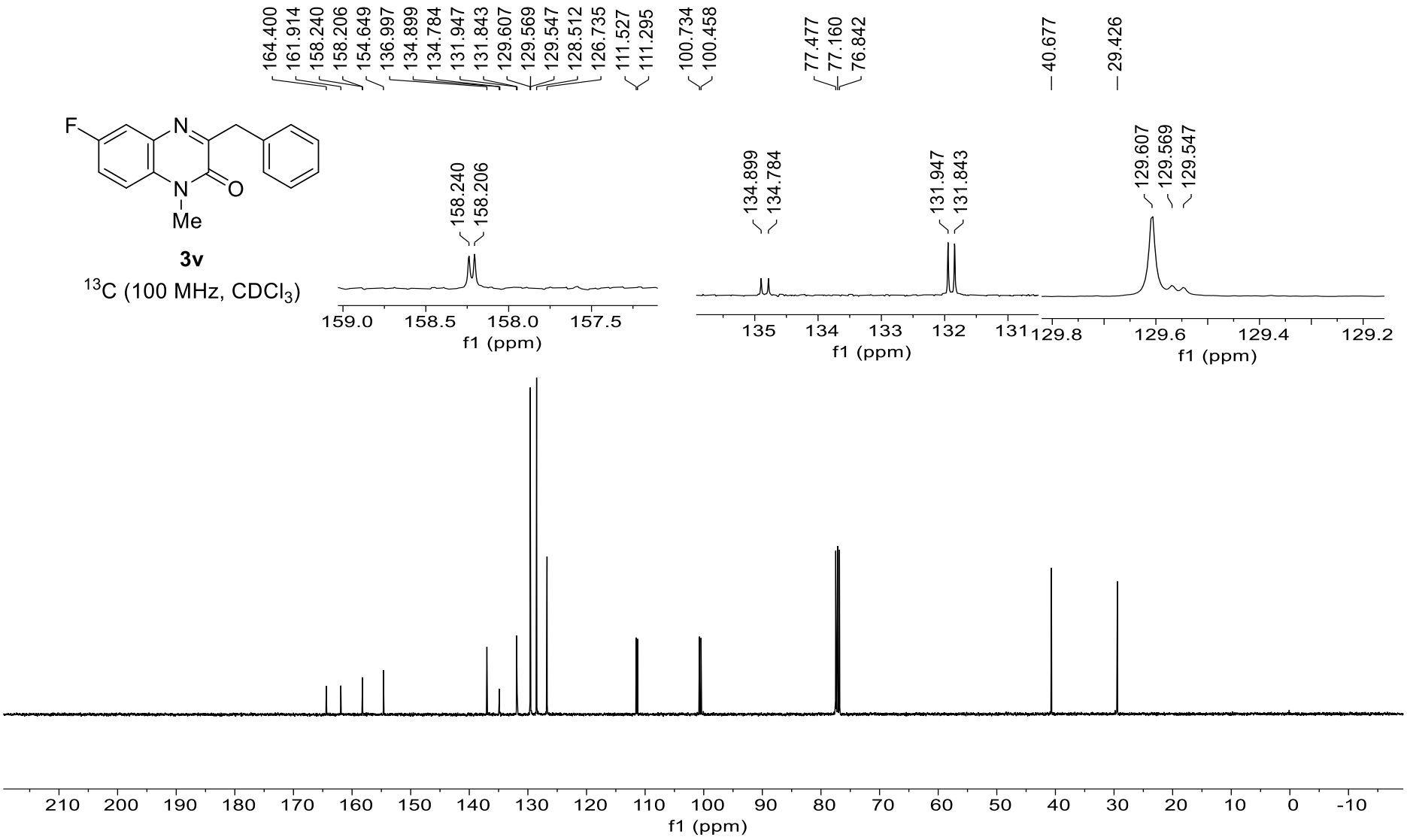
3u

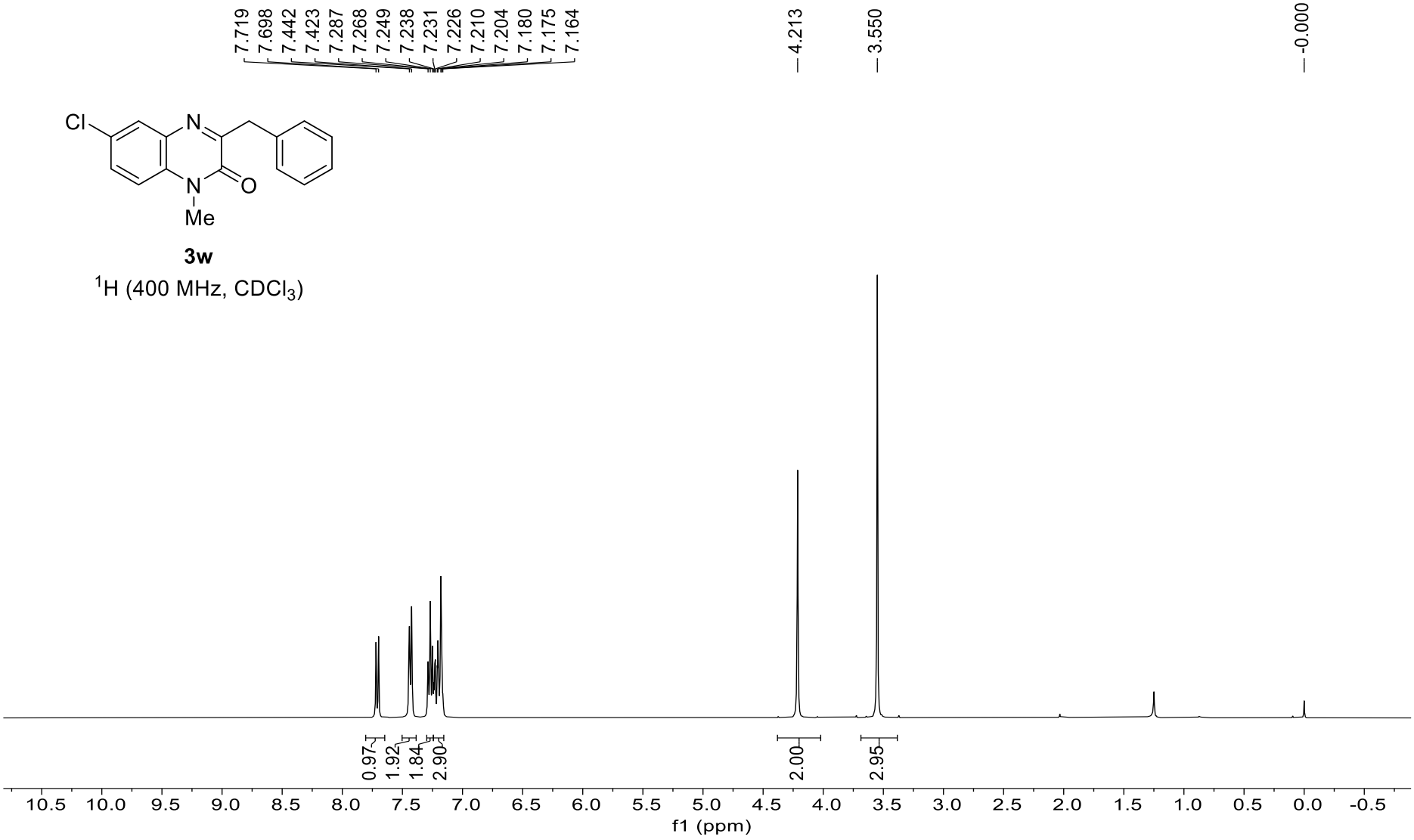
^{13}C (125 MHz, $CDCl_3$)

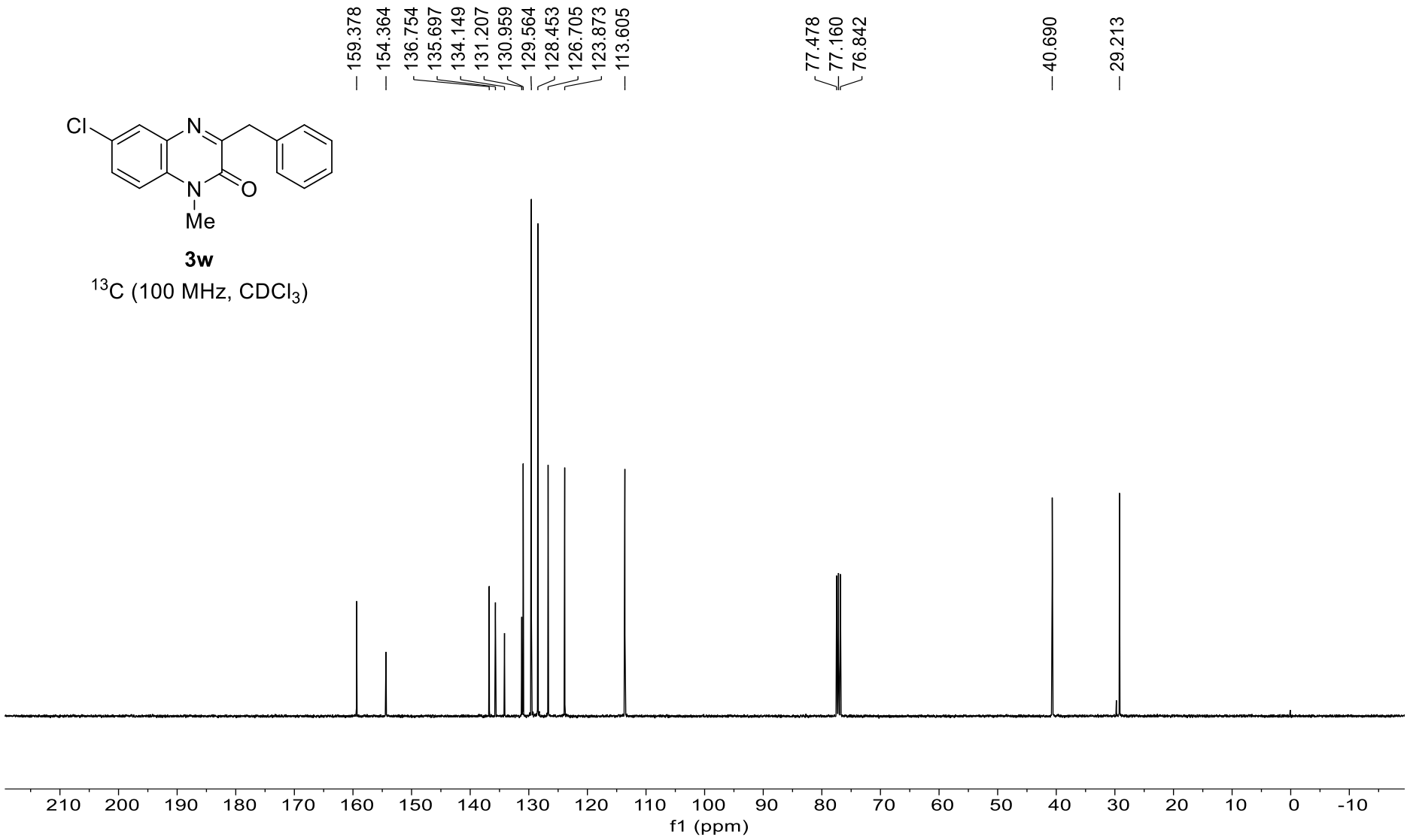


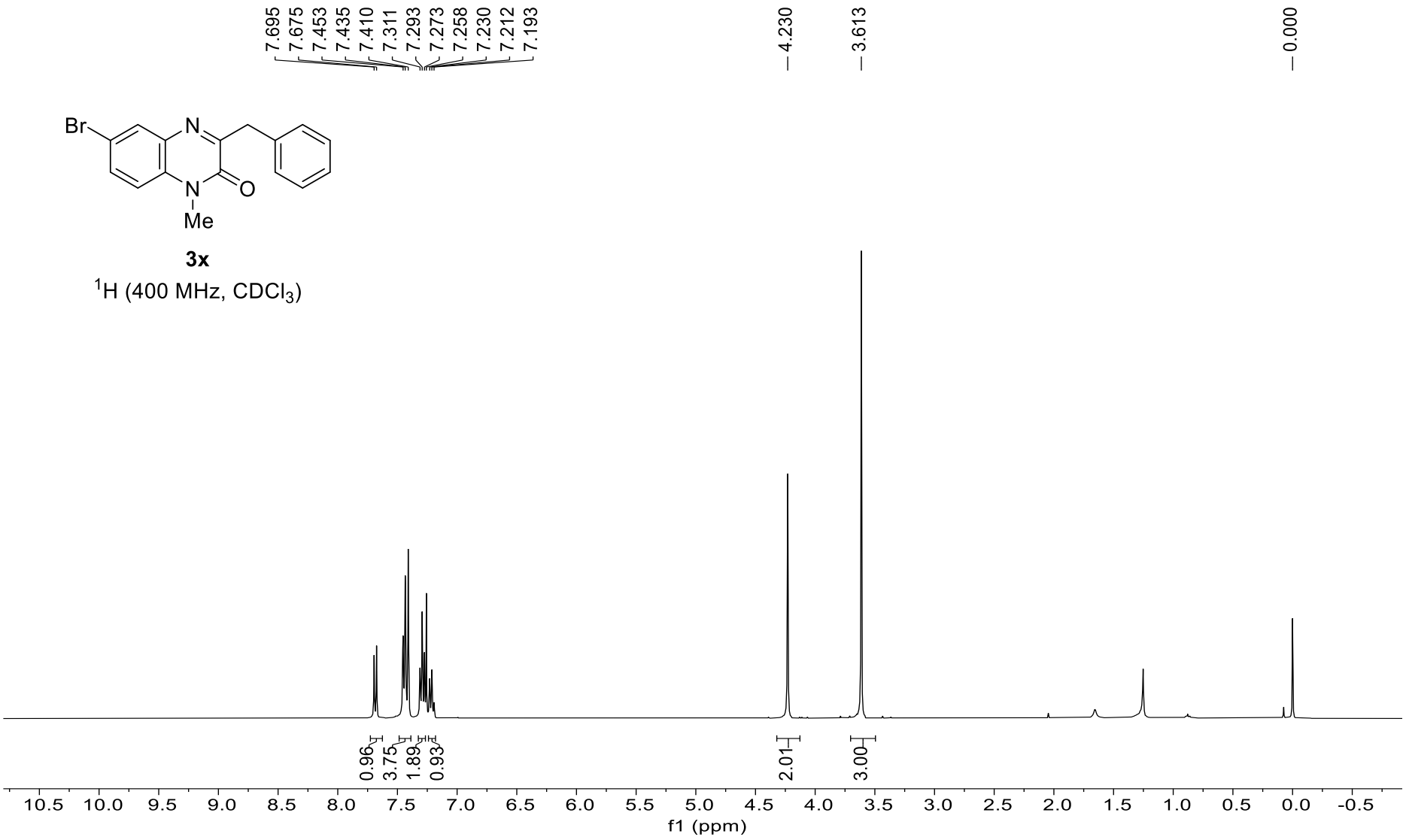
3v

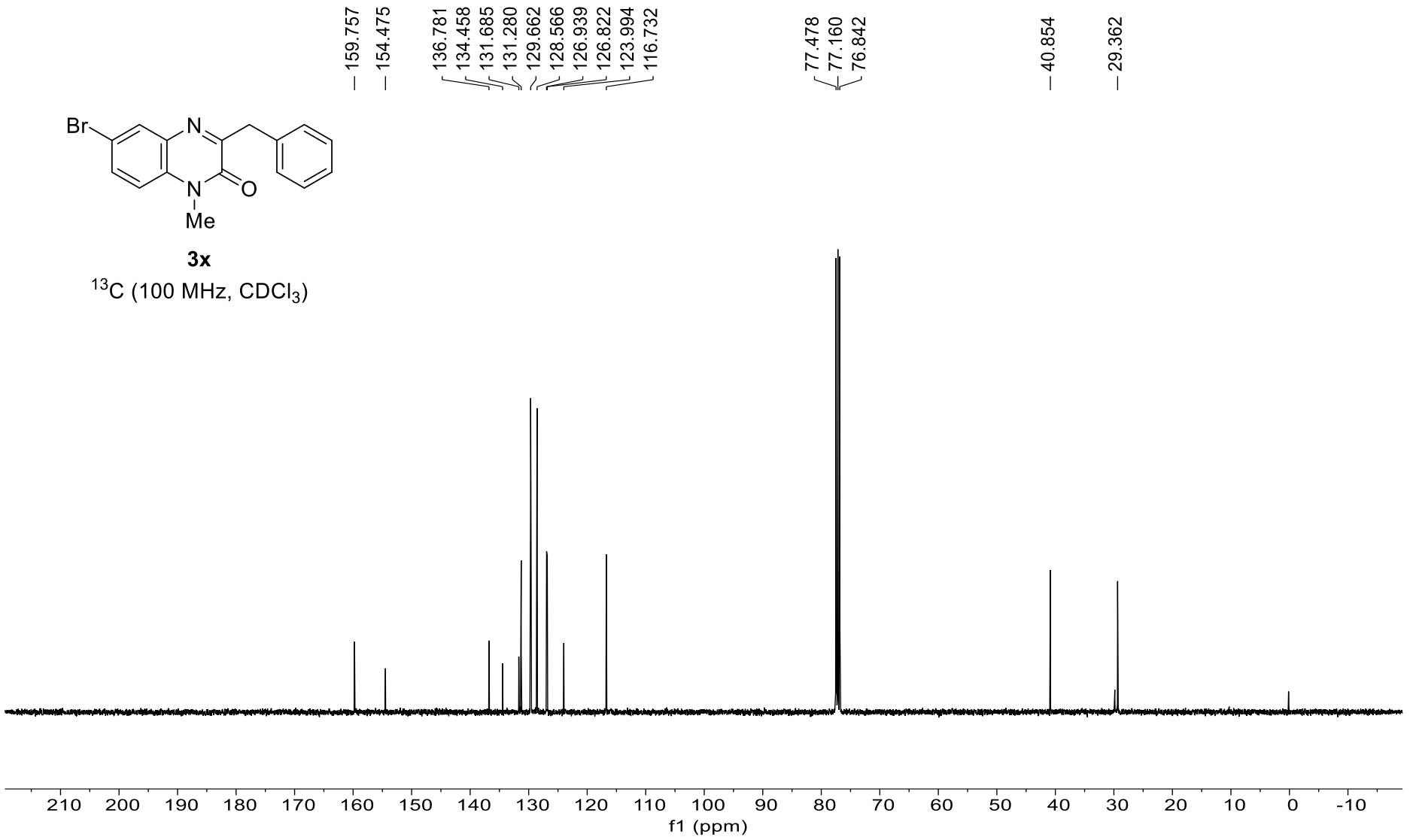
^{1}H (400 MHz, $CDCl_3$)

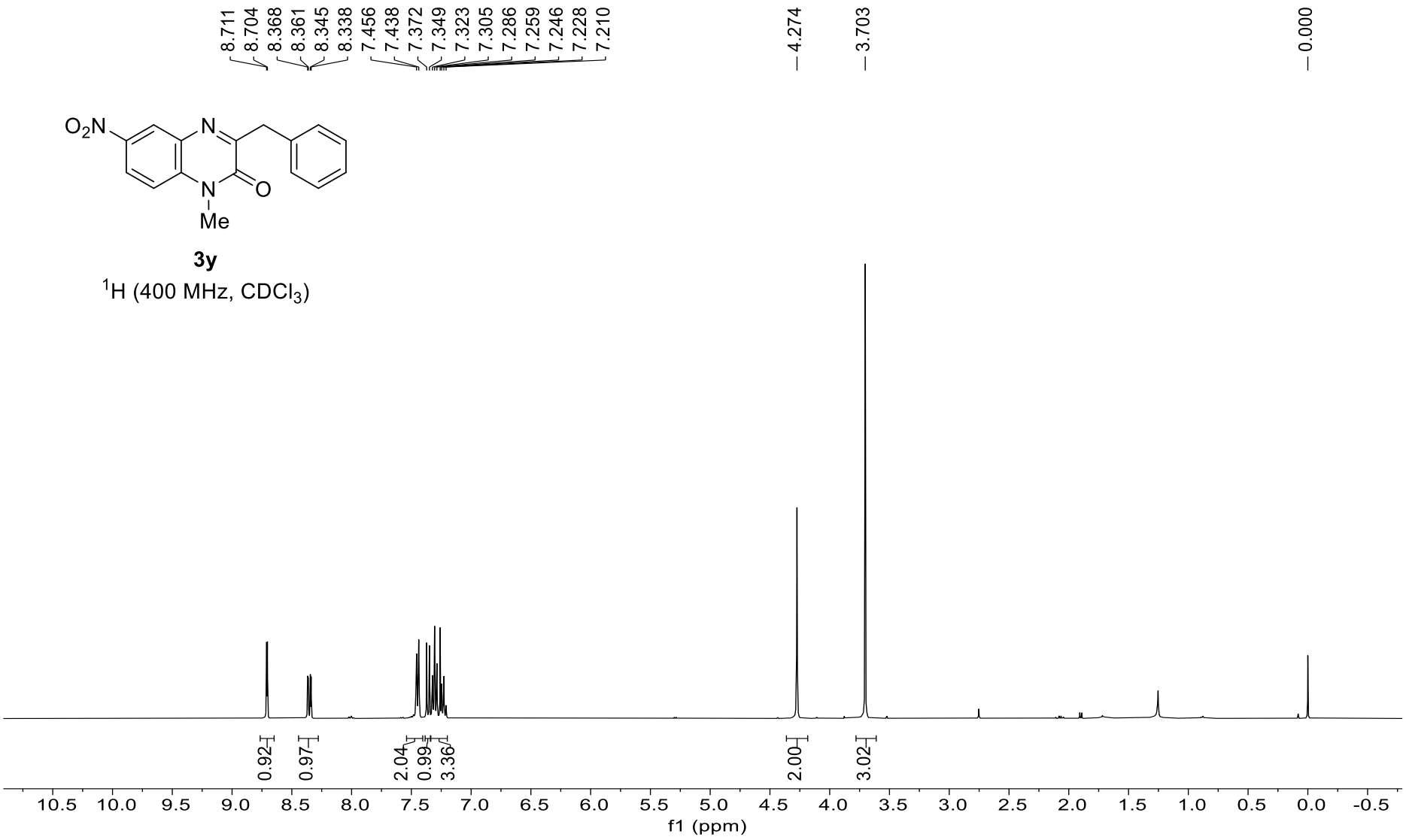

164.400
161.914
158.240
158.206
154.649
136.997
134.899
134.784
131.947
131.843
129.607
129.569
129.547
128.512
126.735
111.527
111.295
100.734
100.458
77.477
77.160
76.842
40.677
29.426
F
N
N
O
Me
3v
^{13}C (100 MHz, CDCl$_3$)
158.240
158.206
159.0
158.5
158.0
157.5
f1 (ppm)
134.899
134.784
131.947
131.843
135
134
133
132
131
f1 (ppm)
129.607
129.569
129.547
129.8
129.6
129.4
129.2
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

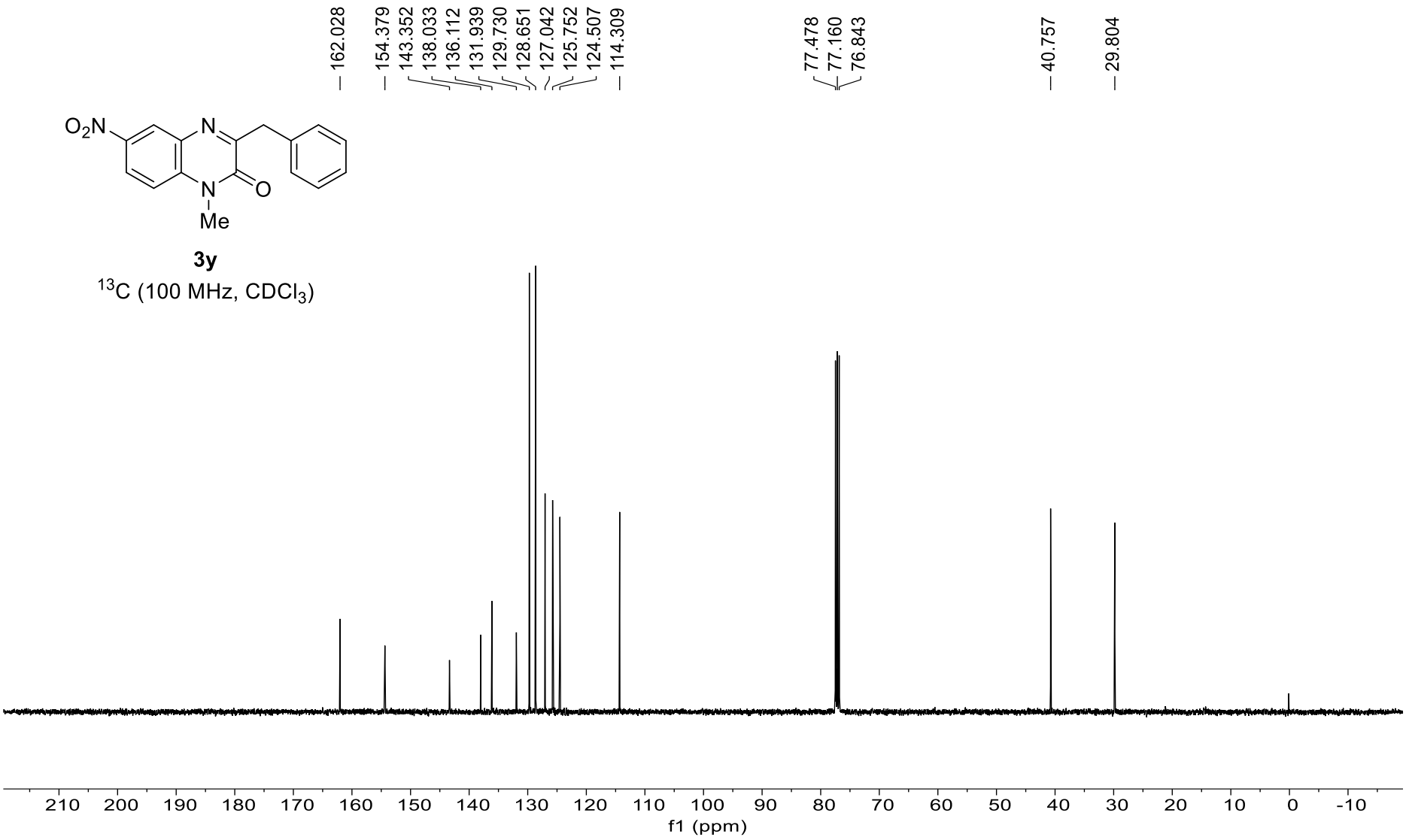

7.719
7.698
7.442
7.423
7.287
7.268
7.249
7.238
7.231
7.226
7.210
7.204
7.180
7.175
7.164
4.213
3.550
-0.000
Cl
N
N
O
Me
3w
1H (400 MHz, CDCl3)
0.97
1.92
1.84
2.90
2.00
2.95
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

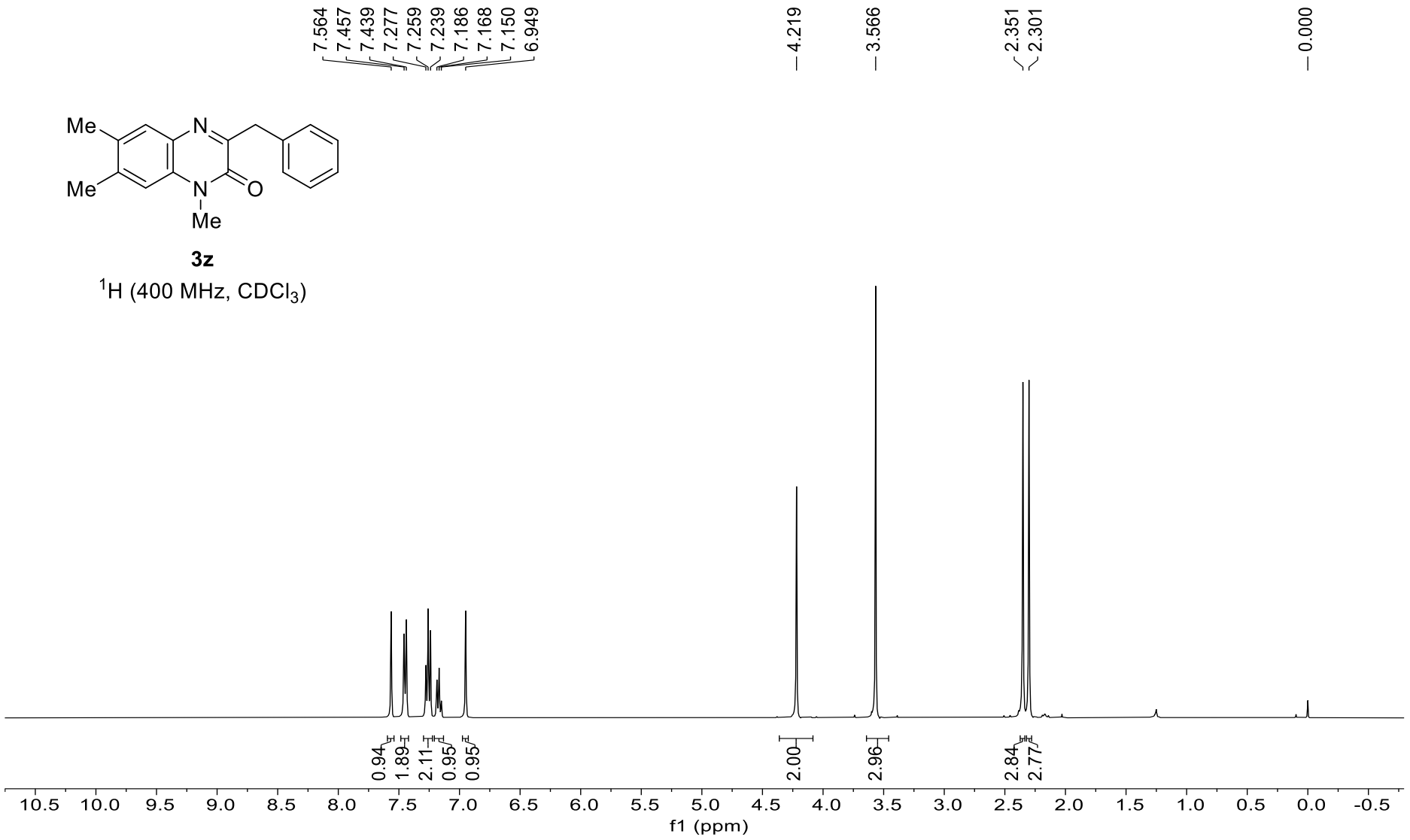

159.378
154.364
136.754
135.697
134.149
131.207
130.959
129.564
128.453
126.705
123.873
113.605
77.478
77.160
76.842
40.690
29.213
Cl
N
N
O
Me
3w
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

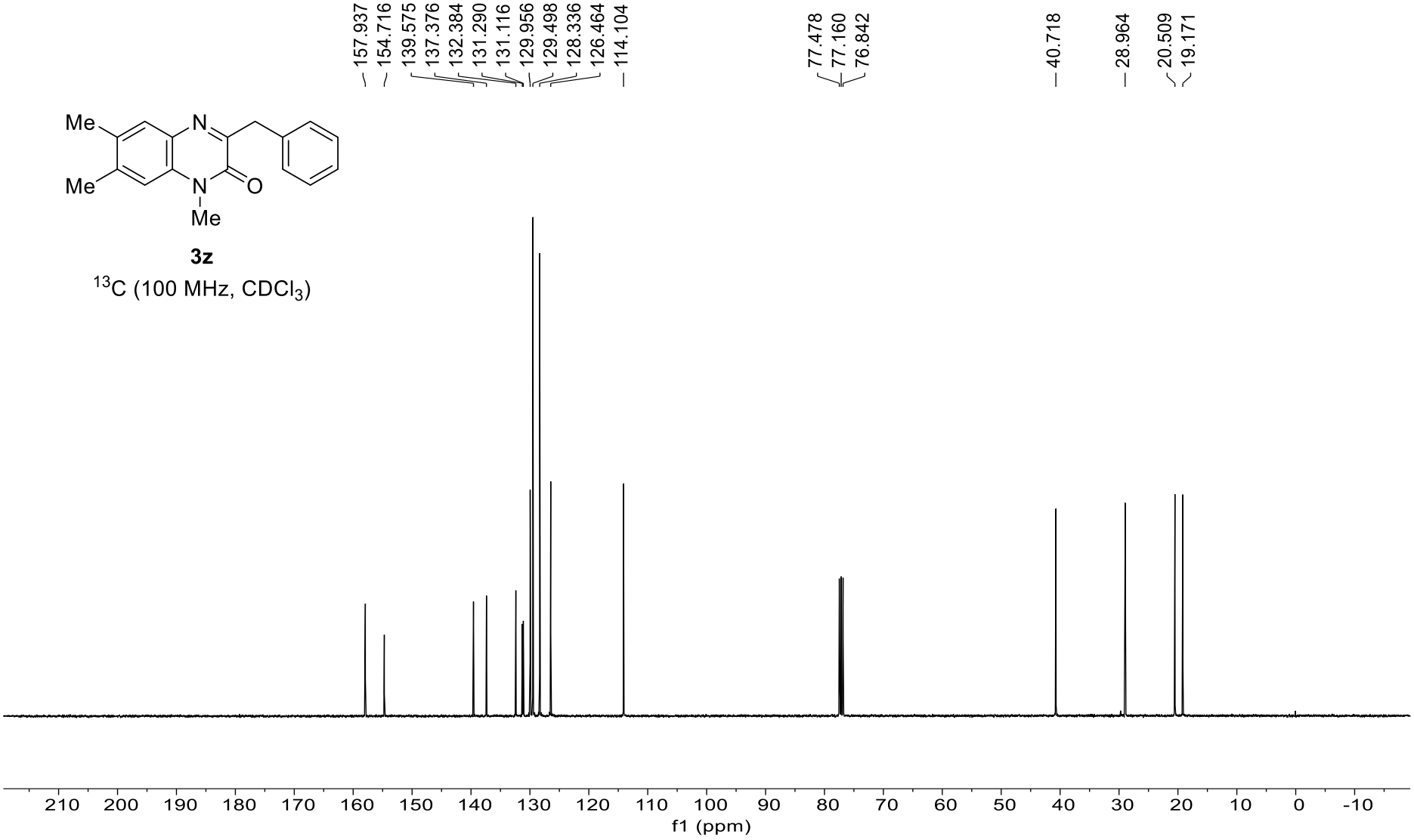

7.695
7.675
7.453
7.435
7.410
7.311
7.293
7.273
7.258
7.230
7.212
7.193
4.230
3.613
0.000
Br
N
N
O
Me
3x
1H (400 MHz, CDCl3)
0.96
3.75
1.89
0.93
2.01
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

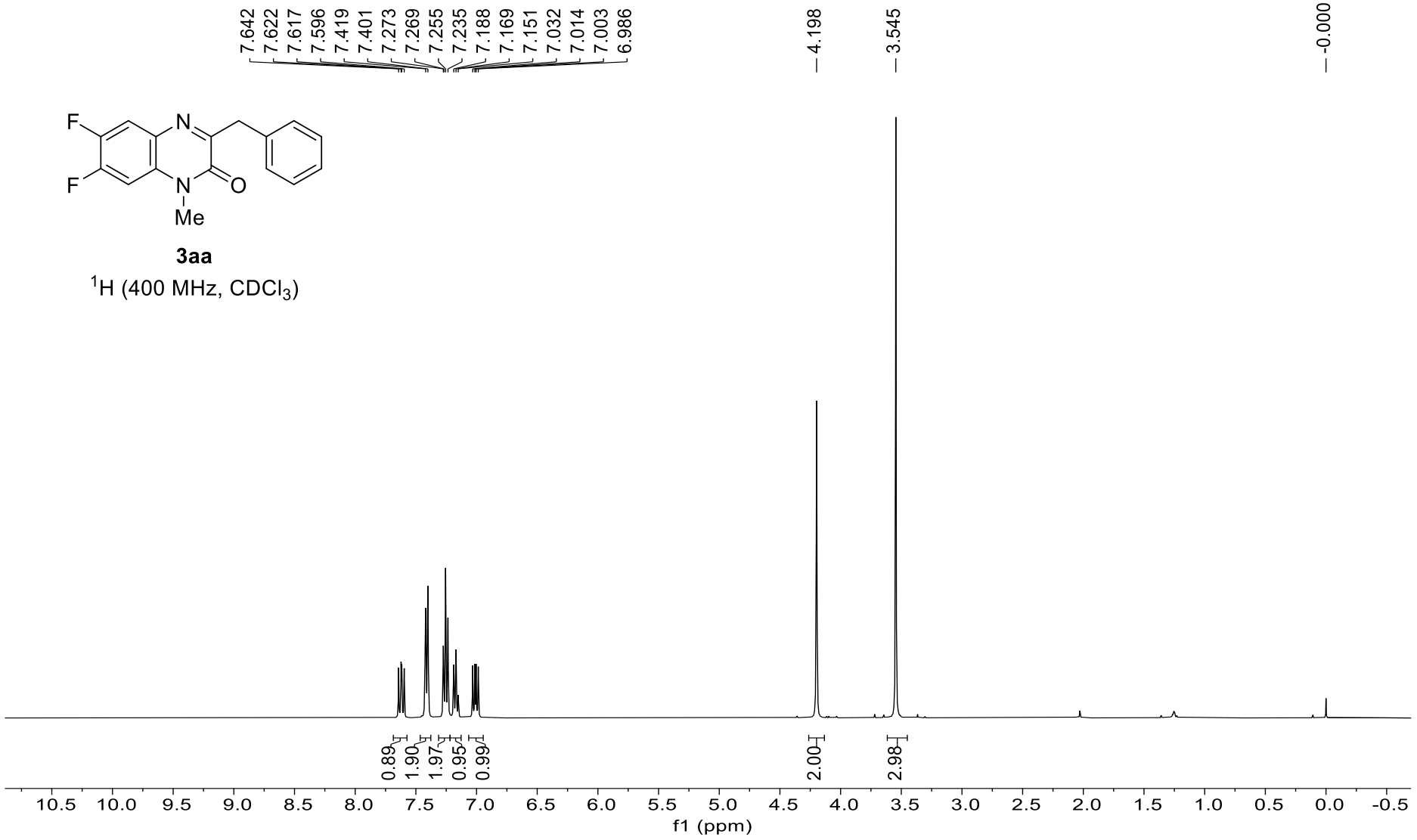

159.757
154.475
136.781
134.458
131.685
131.280
129.662
128.566
126.939
126.822
123.994
116.732
77.478
77.160
76.842
40.854
29.362
Br
N
N
O
Me
3x
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

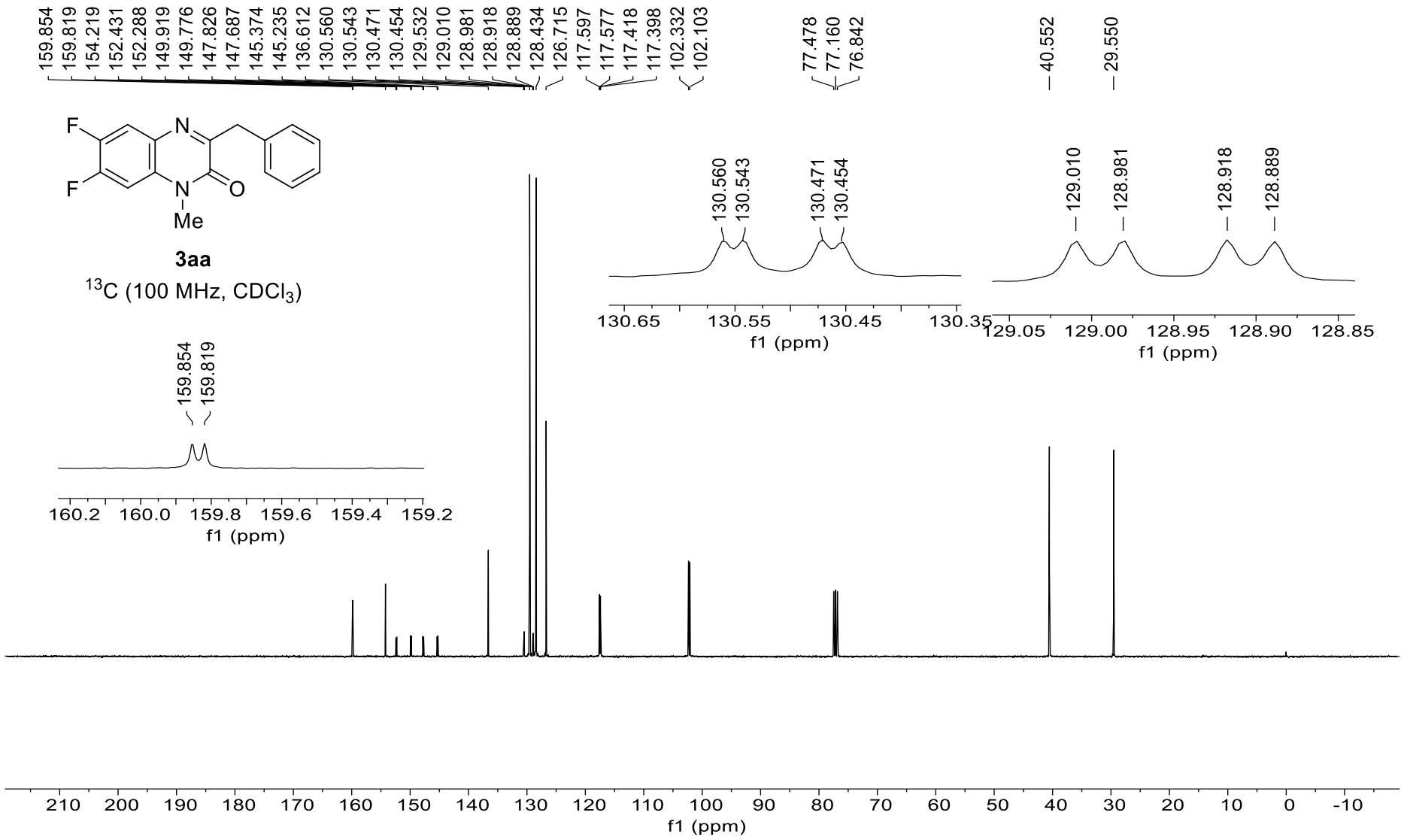

8.711
8.704
8.368
8.361
8.345
8.338
7.456
7.438
7.372
7.349
7.323
7.305
7.286
7.259
7.246
7.228
7.210
4.274
3.703
0.000
O2N
N
N
O
Me
3y
1H (400 MHz, CDCl3)
0.92
0.97
2.04
0.99
3.36
2.00
3.02
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

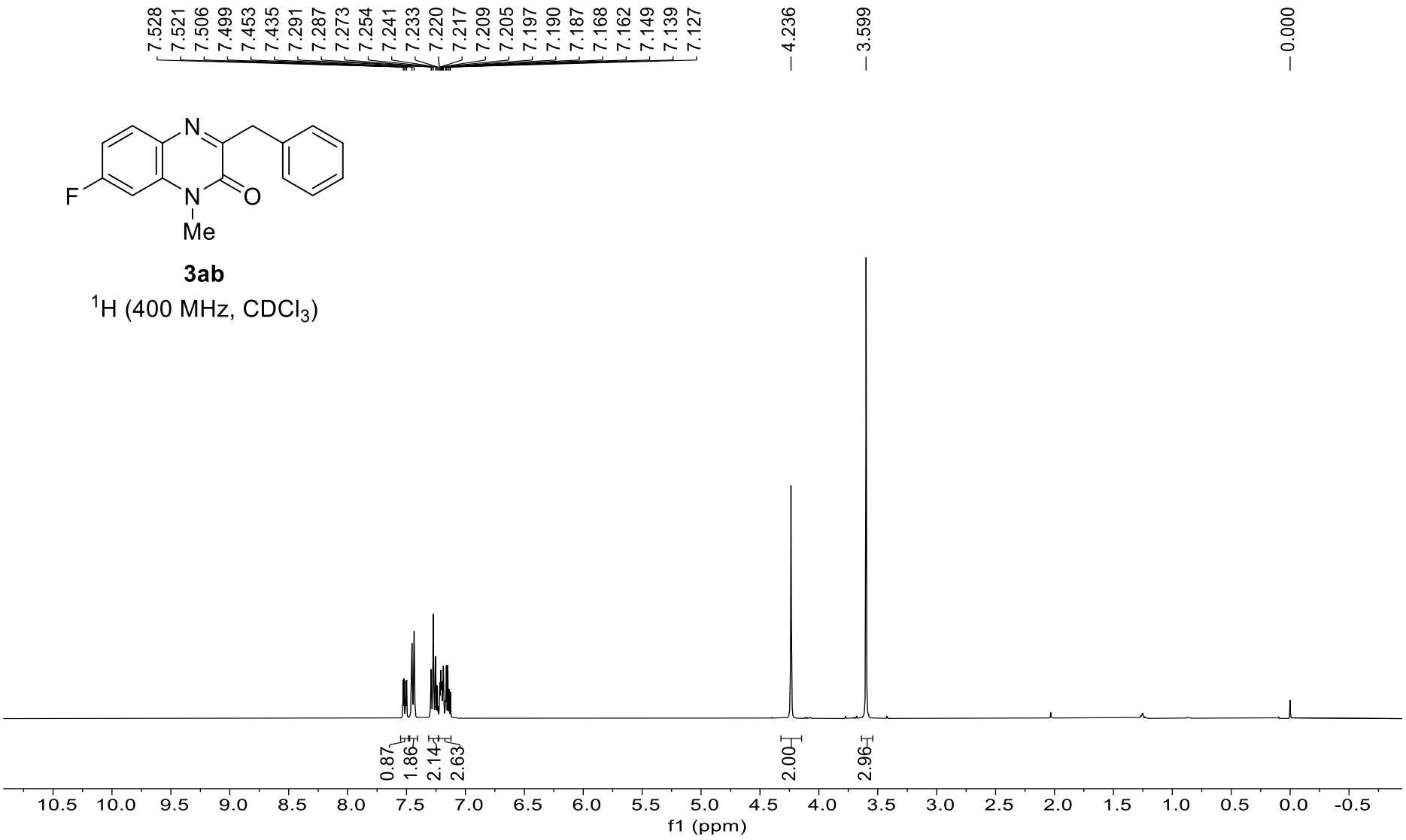

162.028
154.379
143.352
138.033
136.112
131.939
129.730
128.651
127.042
125.752
124.507
114.309
77.478
77.160
76.843
40.757
29.804
O2N
N
N
O
Me
3y
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

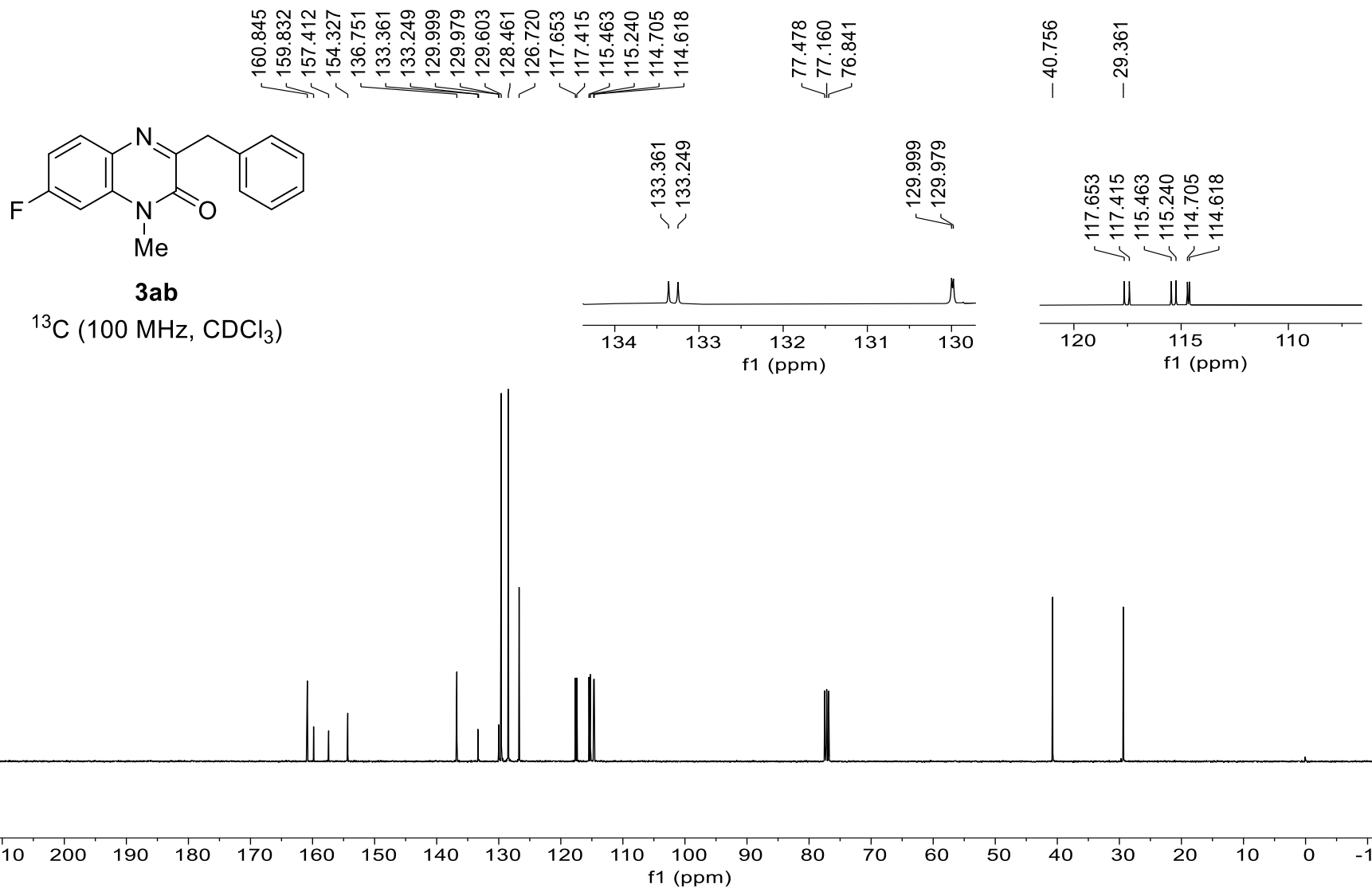

7.564
7.457
7.439
7.277
7.259
7.239
7.186
7.168
7.150
6.949
4.219
3.566
2.351
2.301
0.000
Me
Me
N
N
O
Me
3z
1H (400 MHz, CDCl3)
0.94
1.89
2.11
0.95
0.95
2.00
2.96
2.84
2.77
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

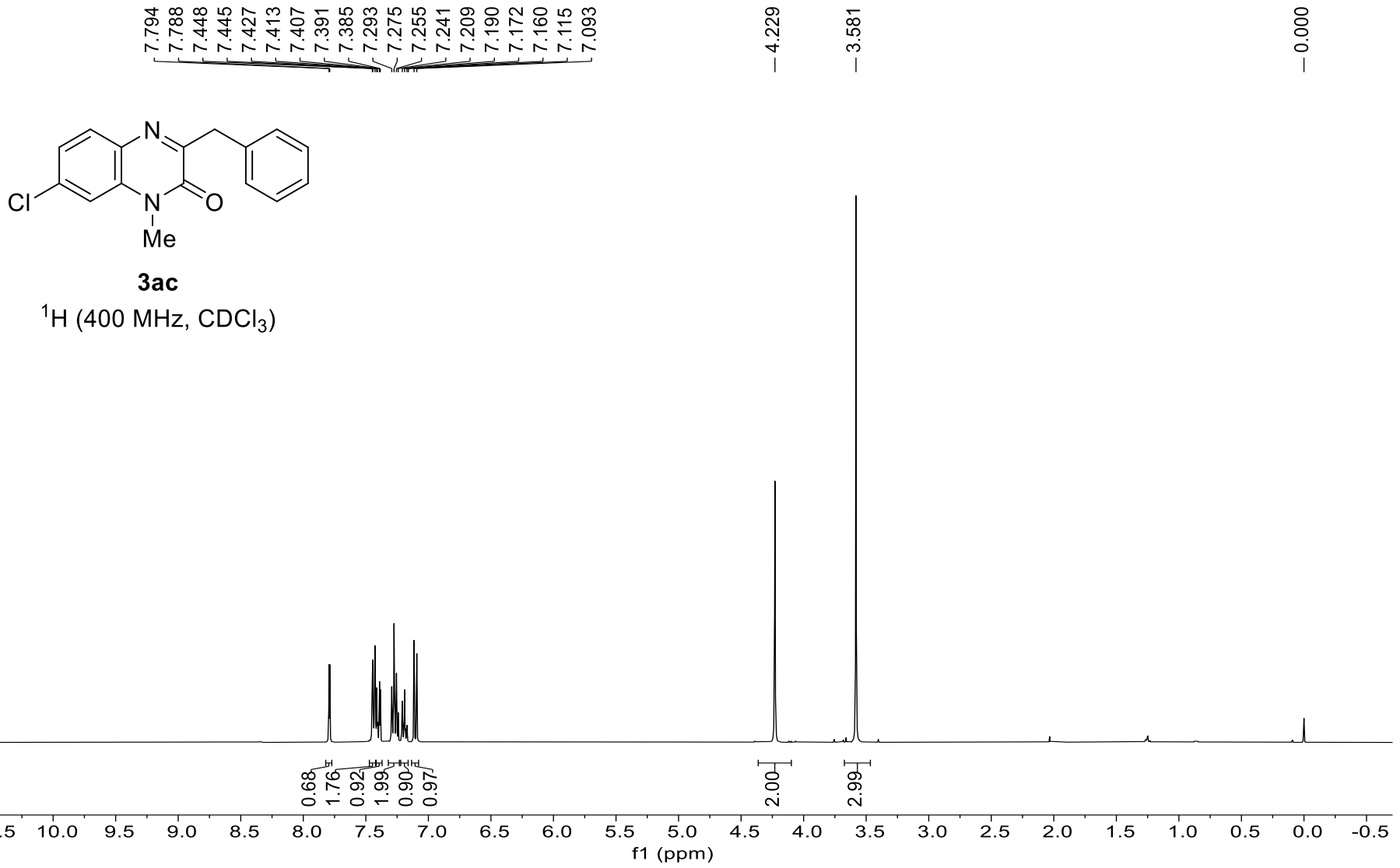

157.937
154.716
139.575
137.376
132.384
131.290
131.116
129.956
129.498
128.336
126.464
114.104
77.478
77.160
76.842
40.718
28.964
20.509
19.171
Me
Me
N
N
O
Me
3z
13C (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.642
7.622
7.617
7.596
7.419
7.401
7.273
7.269
7.255
7.235
7.188
7.169
7.151
7.032
7.014
7.003
6.986
4.198
3.545
-0.000
F
F
N
N
O
Me
3aa
1H (400 MHz, CDCl3)
0.89
1.90
1.97
0.95
0.99
2.00
2.98
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
f1 (ppm)


159.854
159.819
154.219
152.431
152.288
149.919
149.776
147.826
147.687
145.374
145.235
136.612
130.560
130.543
130.471
130.454
129.532
129.010
128.981
128.918
128.889
128.434
126.715
117.597
117.577
117.418
117.398
102.332
102.103
77.478
77.160
76.842
40.552
29.550
F
F
N
N
O
Me
3aa
13C (100 MHz, CDCl3)
130.560
130.543
130.471
130.454
130.65
130.55
130.45
130.35
f1 (ppm)
129.010
128.981
128.918
128.889
129.05
129.00
128.95
128.90
128.85
f1 (ppm)
159.854
159.819
160.2
160.0
159.8
159.6
159.4
159.2
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

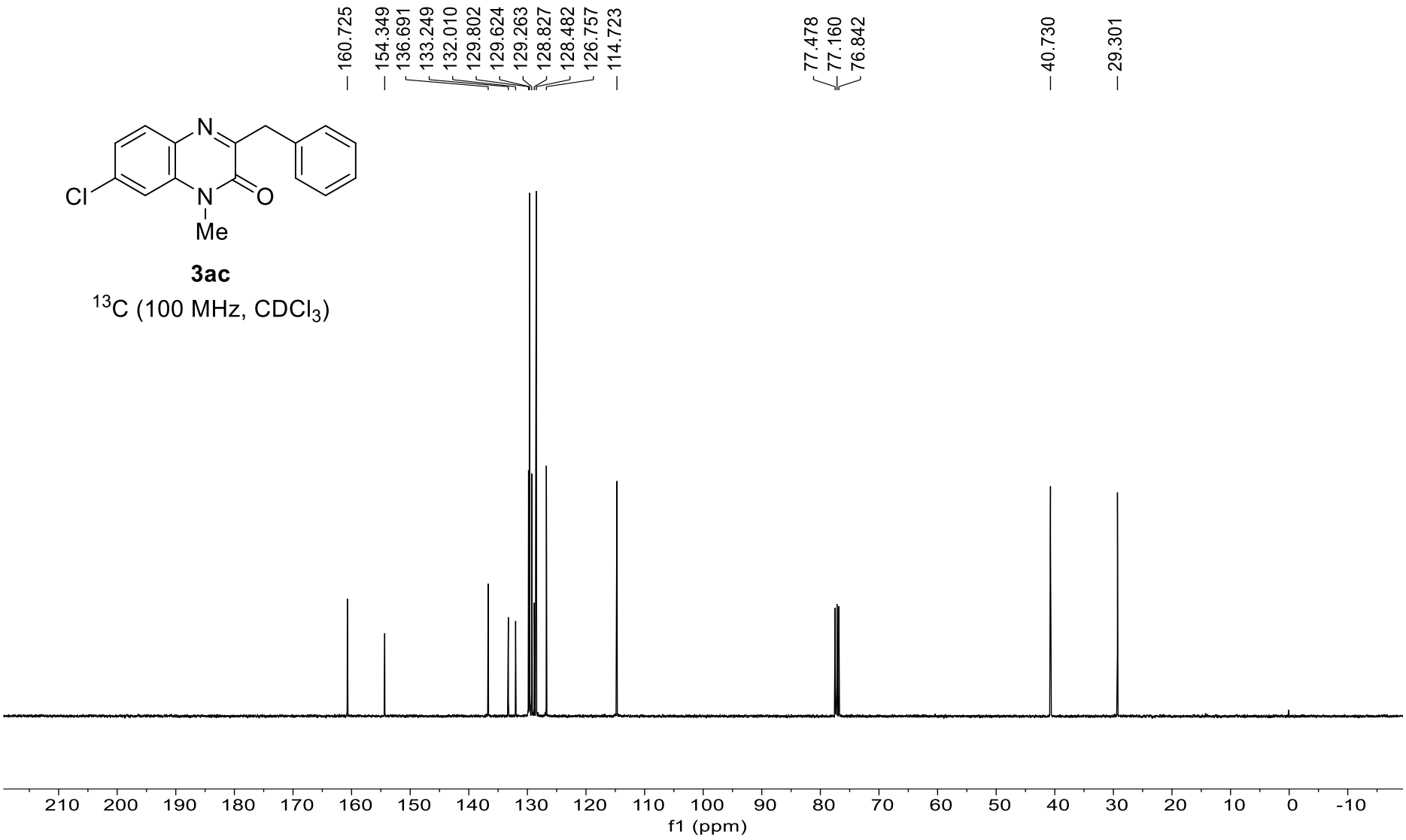

7.528
7.521
7.506
7.499
7.453
7.435
7.291
7.287
7.273
7.254
7.241
7.233
7.220
7.217
7.209
7.205
7.197
7.190
7.187
7.168
7.162
7.149
7.139
7.127
4.236
3.599
0.000
F
N
N
O
Me
3ab
1H (400 MHz, CDCl3)
0.87
1.86
2.14
2.63
2.00
2.96
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

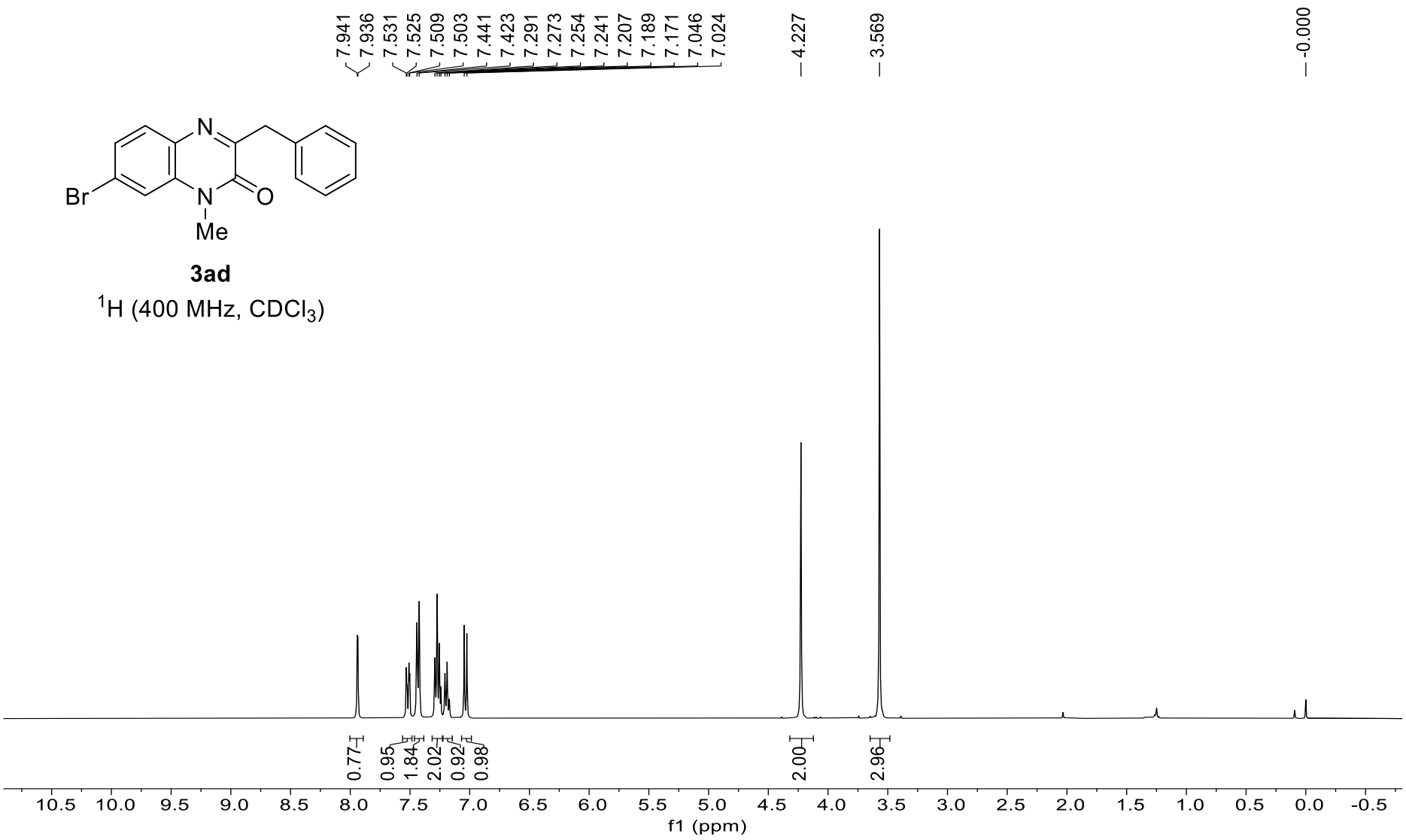

160.845
159.832
157.412
154.327
136.751
133.361
133.249
129.999
129.979
129.603
128.461
126.720
117.653
117.415
115.463
115.240
114.705
114.618
77.478
77.160
76.841
40.756
29.361
F
N
N
O
Me
3ab
13C (100 MHz, CDCl3)
f1 (ppm)
f1 (ppm)
f1 (ppm)

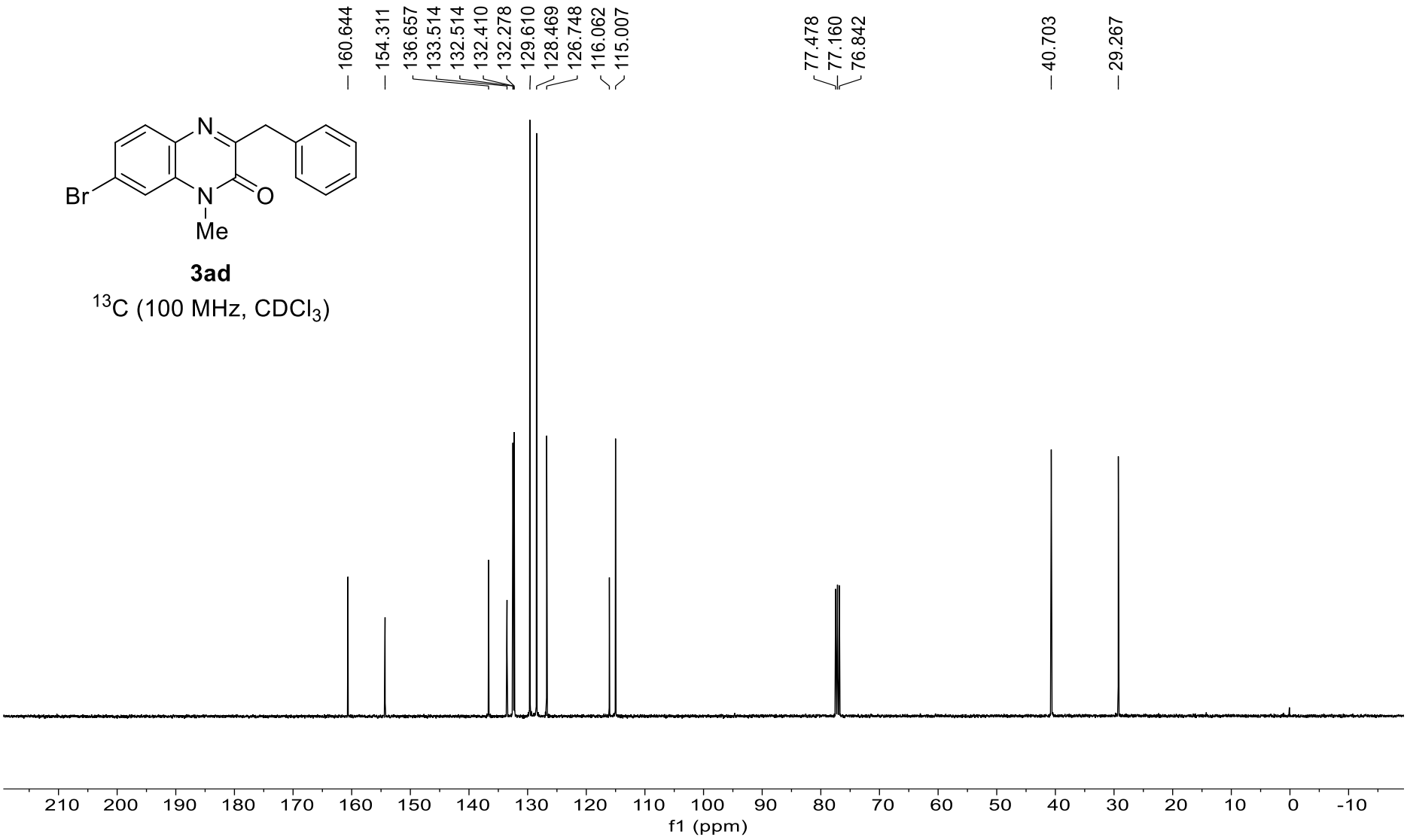


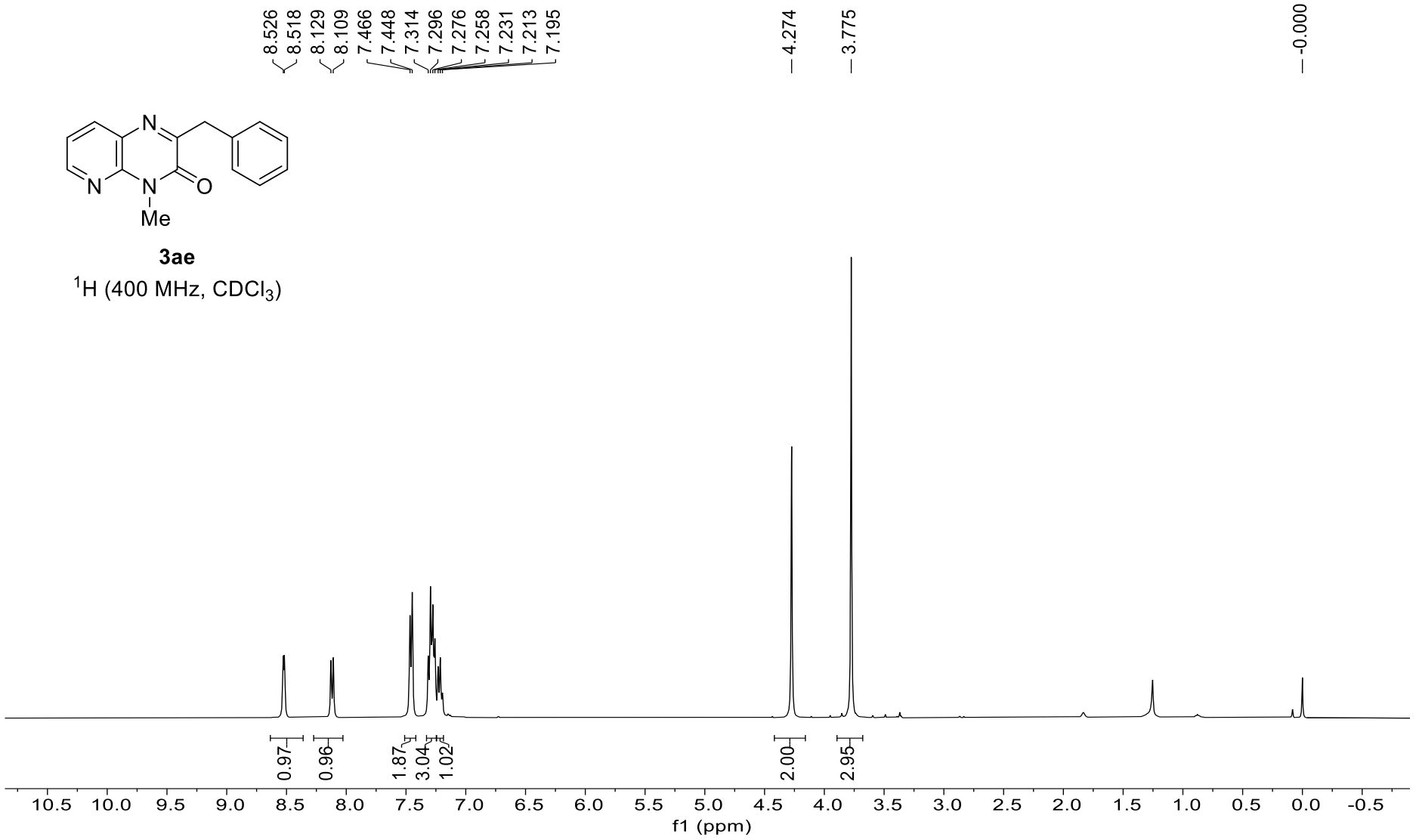
3ac

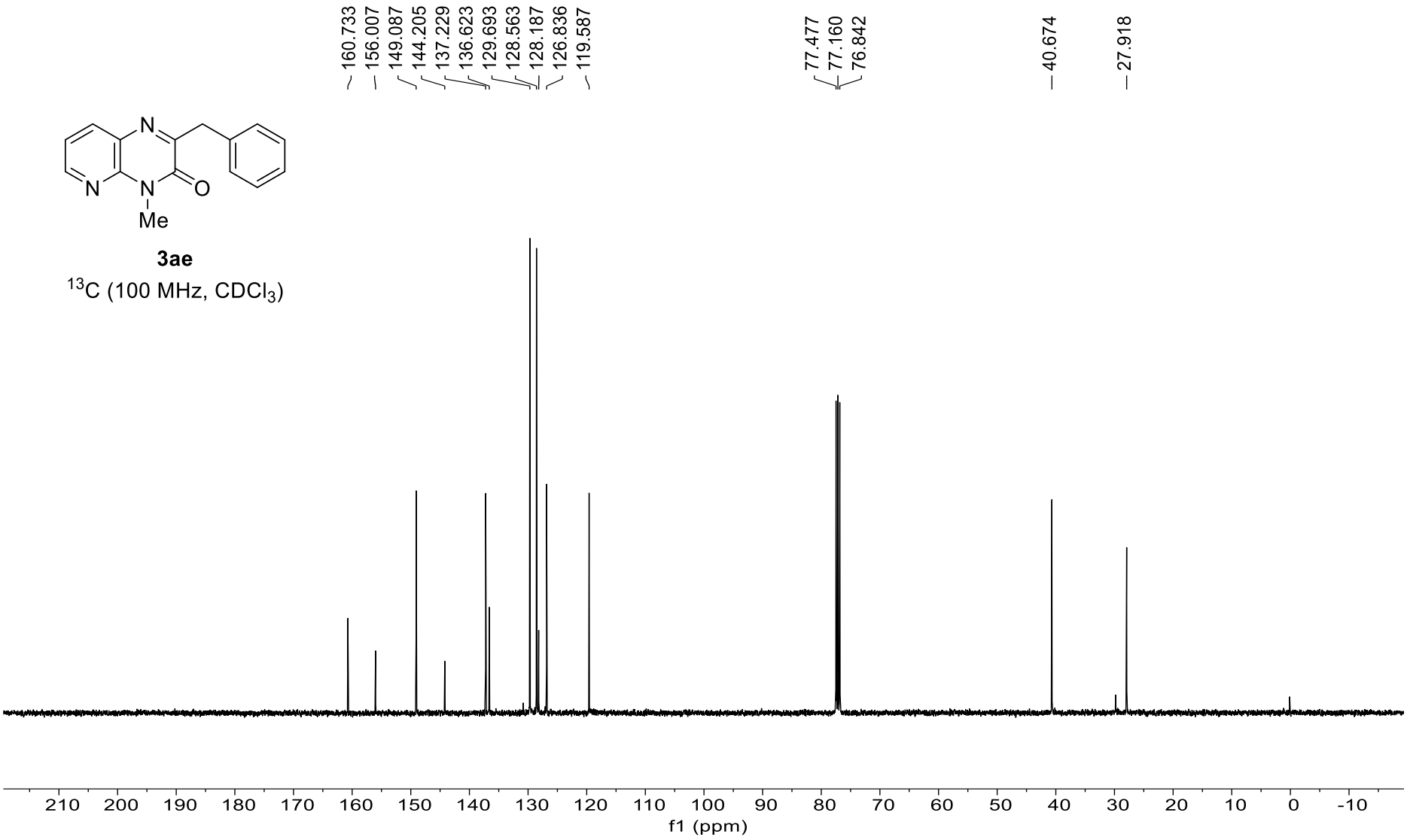
^{1}H (400 MHz, $CDCl_3$)


160.725
154.349
136.691
133.249
132.010
129.802
129.624
129.263
128.827
128.482
126.757
114.723
77.478
77.160
76.842
40.730
29.301
N
Cl
N
O
Me
3ac
13C (100 MHz, CDCl3)
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)


7.941
7.936
7.531
7.525
7.509
7.503
7.441
7.423
7.291
7.273
7.254
7.241
7.207
7.189
7.171
7.046
7.024
4.227
3.569
-0.000
N
Br
N
O
Me
3ad
1H (400 MHz, CDCl3)
0.77
0.95
1.84
2.02
0.92
0.98
2.00
2.96
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
f1 (ppm)


160.644
154.311
136.657
133.514
132.514
132.410
132.278
129.610
128.469
126.748
116.062
115.007
77.478
77.160
76.842
40.703
29.267
Br
N
N
O
Me
3ad
13C (100 MHz, CDCl3)
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)


8.526
8.518
8.129
8.109
7.466
7.448
7.314
7.296
7.276
7.258
7.231
7.213
7.195
4.274
3.775
-0.000
N
N
N
O
Me
3ae
1H (400 MHz, CDCl3)
0.97
0.96
1.87
3.04
1.02
2.00
2.95
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
f1 (ppm)


160.733
156.007
149.087
144.205
137.229
136.623
129.693
128.563
128.187
126.836
119.587
77.477
77.160
76.842
40.674
27.918
N
N
N
O
Me
3ae
13C (100 MHz, CDCl3)
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)