Cu-Catalyzed Carbamoylation versus Amination of Quinoline *N*-Oxide with Formamides

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Supporting Information

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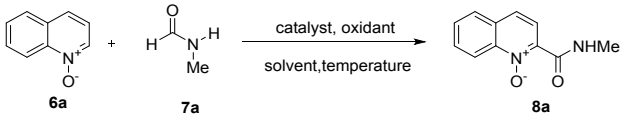
1. General Information

Reactions were monitored by TLC on silica gel plates (GF254), and the analytical thin-layer chromatography (TLC) was performed on precoated, glass-backed silica gel plates. Commercially available materials were used as purchased. ^{1}H and ^{13}C NMR spectra were recorded on Bruker AV400 (400 MHz) spectrometers. Chemical shifts were recorded in parts per million (ppm, δ) relative to tetramethylsilane (δ 0.00) or chloroform (δ = 7.26, singlet). NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), dd (doublet of doublets), m (multiplets), etc. All first-order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br). HRMS were recorded on Waters Xevo G2 Q-TOF instrument.

2. Optimization of the Reaction Conditions

2.1 Optimization of the Carbamoylation Reaction Conditions

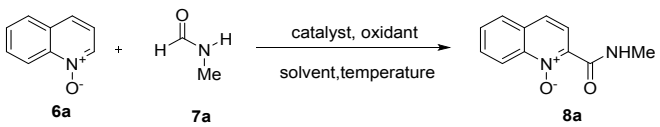
Table S1 Screening of Copper Salts [a]



Entry	Catalyst	Oxidant	Solvent	Temperature	Yield [b] (%)
1	Cu_2O	TBHP	DMSO	rt	32
2	CuI	TBHP	DMSO	rt	7
3	CuCl	TBHP	DMSO	rt	29
4	$Cu(MeCN)_4BF_4$	TBHP	DMSO	rt	63
5	CuOTf	TBHP	DMSO	rt	58
6	CuBr	TBHP	DMSO	rt	85
7	CuCN	TBHP	DMSO	rt	69
8	CuOAc	TBHP	DMSO	rt	33

[a] Reaction conditions: **6a** (0.2 mmol), **7a** (10 equiv), TBHP (5.0-6.0 M in decane) (2 equiv), Cu catalyst (10 mol%), DMSO (1 mL), room temperature; [b] Isolated yield.

Table S2 Screening of Oxidants [a]

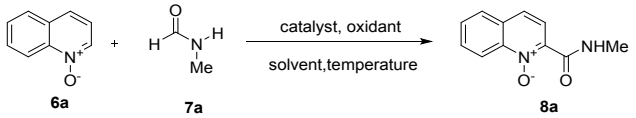


Entry	Catalyst	Oxidant	Solvent	Temperature	Yield [b] (%)
1	CuBr	TBHP	DMSO	rt	85
2	CuBr	DTBP	DMSO	rt	71
3	CuBr	BPO	DMSO	rt	62

4	CuBr	$K_2S_2O_8$	DMSO	rt	trace
5	CuBr	Ag_2CO_3	DMSO	rt	trace
6	CuBr	$PhI(OAc)_2$	DMSO	rt	trace
7	CuBr	H_2O_2	DMSO	rt	39
8	CuBr	O_2	DMSO	rt	trace
9	CuBr	air	DMSO	rt	trace

[a] Reaction conditions: **6a** (0.2 mmol), **7a** (10 equiv), oxidant (2 equiv), CuBr (10 mol%), DMSO (1 mL), room temperature; [b] Isolated yield.

Table S3 Screening of Solvents [a]



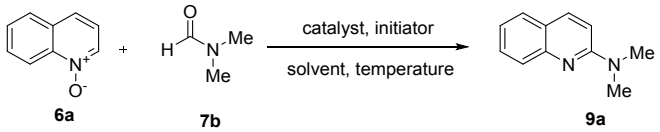
6a **7a** **8a**

Entry	Catalyst	Oxidant	Solvent	Temperature	Yield [b] (%)
1	CuBr	TBHP	DMSO	rt	85
2	CuBr	TBHP	EtOAc	rt	57
3	CuBr	TBHP	DCE	rt	trace
4	CuBr	TBHP	MeCN	rt	trace
5	CuBr	TBHP	toluene	rt	50
6	CuBr	TBHP	EtOH	rt	trace

[a] Reaction conditions: **6a** (0.2 mmol), **7a** (10 equiv), TBHP (5.0-6.0 M in decane) (2 equiv), CuBr (10 mol%), solvent (1 mL), room temperature; [b] Isolated yield.

2.2 Optimization of the Amination Reaction Conditions

Table S4 Screening of Copper Salts [a]



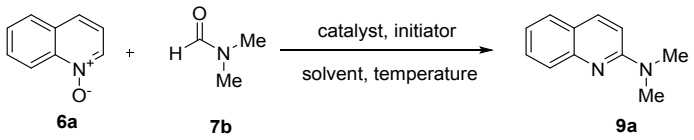
6a **7b** **9a**

Entry	Catalyst	Initiator	Solvent	Temperature	Yield [b] (%)
1	CuBr	TBHP	DMSO	rt	11
2	CuI	TBHP	DMSO	rt	50
3	CuCl	TBHP	DMSO	rt	trace
4	$Cu(MeCN)_4BF_4$	TBHP	DMSO	rt	trace
5	CuOTf	TBHP	DMSO	rt	trace
6	Cu_2O	TBHP	DMSO	rt	trace

7	CuCN	TBHP	DMSO	rt	trace
8	CuOAc	TBHP	DMSO	rt	trace

[a] Reaction conditions: **6a** (0.2 mmol), **7b** (10 equiv), TBHP (5.0-6.0 M in decane) (0.05 equiv), Cu catalyst (10 mol%), DMSO (1 mL), room temperature; [b] Isolated yield.

Table S5 Screening of Initiators [a]

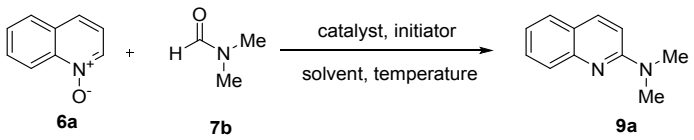


6a **7b** **9a**

Entry	Catalyst	Initiator	Solvent	Temperature	Yield [b] (%)
1	CuI	TBHP	DMSO	rt	50
2	CuI	DTBP	DMSO	rt	37
3	CuI	$K_2S_2O_8$	DMSO	rt	trace
4	CuI	Ag_2CO_3	DMSO	rt	trace
5	CuI	$PhI(OAc)_2$	DMSO	rt	trace
6	CuI	H_2O_2	DMSO	rt	12
7	CuI	O_2	DMSO	rt	trace
8	CuI	air	DMSO	rt	trace

[a] Reaction conditions: **6a** (0.2 mmol), **7b** (10 equiv), initiator (0.05 equiv), CuI (10 mol%), DMSO (1 mL), room temperature; [b] Isolated yield.

Table S6 Screening of Solvents [a]

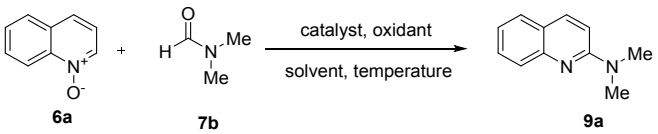


6a **7b** **9a**

Entry	Catalyst	Initiator	Solvent	Temperature	Yield [b] (%)
1	CuI	TBHP	DMSO	rt	50
2	CuI	TBHP	EtOAc	rt	trace
3	CuI	TBHP	CH_2Cl_2	rt	67
4	CuI	TBHP	MeCN	rt	trace
6	CuI	TBHP	toluene	rt	trace
7	CuI	TBHP	EtOH	rt	trace

[a] Reaction conditions: **6a** (0.2 mmol), **7b** (10 equiv), TBHP (5.0-6.0 M in decane) (0.05 equiv), CuI (10 mol%), solvent (1 mL), room temperature; [b] Isolated yield.

Table S7 Screening of Temperatures [a]



6a **7b** **9a**

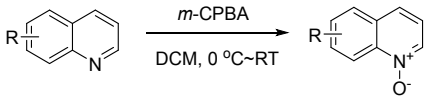
Entry	Catalyst	Initiator	Solvent	Temperature	Yield [b] (%)
1	CuI	TBHP	CH_2Cl_2	rt	67
2	CuI	TBHP	CH_2Cl_2	40 °C	73

[a] Reaction conditions: **6a** (0.2 mmol), **7b** (10 equiv), TBHP (5.0-6.0 M in decane) (0.05 equiv), CuI (10 mol%), CH_2Cl_2 (1 mL); [b] Isolated yield.

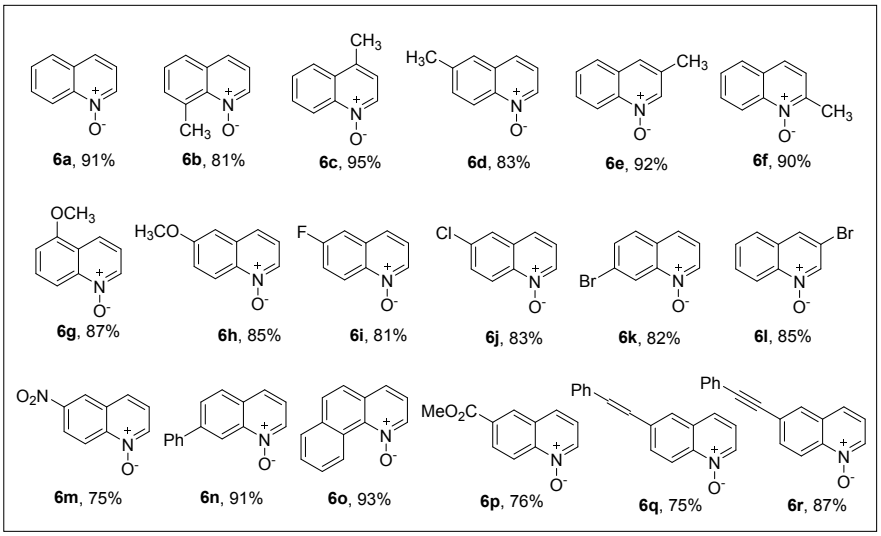
3. General Procedure and Characterization of Products

3.1 Synthesis of substrates quinoline *N*-oxide derivatives **6**

The substrates quinoline *N*-oxide derivatives **6** were synthesized according to the corresponding literatures with minor modifications. [1-4]

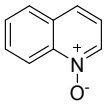


12 **6**



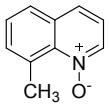
Under vigorous magnetic stirring, 3-chloroperbenzoic acid (*m*-CPBA) (345 mg, 2 mmol) in CH_2Cl_2 (10 mL) was dropped into solution of quinoline derivatives **12** (2 mmol) in CH_2Cl_2 (10 mL) cooled to 0 °C. After the completion of this course, the reaction mixture was allowed up to room

temperature and stirred overnight. An aqueous solution of saturated $NaHCO_3$ was added to the mixture to neutralize residual *m*-CPBA. The resulting mixture was extracted with CH_2Cl_2 (3 × 10 mL). The organic phase was combined and washed with saturated NaCl solution (3 × 5 mL). The organic layer was dried over anhydrous Na_2SO_4, filtered and evaporated under reduced pressure to give crude products, which were purified by column chromatography (silica gel 300–400 mesh, EA: MeOH (8:1) as eluent). The products were identified by ^1H NMR and ^{13}C NMR spectra and compared to the previous literatures.



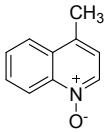
6a

The compound is known [5] and was prepared according to general procedure 3.1 (yellow oil, 0.26 g, 91% yield): ^1H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.75 (d, 1H, J = 8 Hz, ArH), 8.55 (d, 1H, J = 8 Hz, ArH), 7.89-7.87 (m, 1H, ArH), 7.79-7.75 (m, 2H, ArH), 7.67-7.64 (m, 1H, ArH), 7.33-7.29 (m, 1H, ArH).



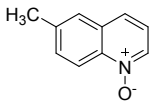
6b

The compound is known [5] and was prepared according to general procedure 3.1 (yellow solid, mp. 54 °C, 0.26 g, 81% yield): ^1H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.43 (d, 1H, J = 8 Hz, ArH), 7.87-7.82 (m, 2H, ArH), 7.53-7.45 (m, 2H, ArH), 7.39-7.36 (m, 1H, ArH), 3.07 (s, 3H, CH_3).



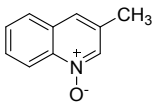
6c

The compound is known [5] and was prepared according to general procedure 3.1 (yellow solid, mp. 75 °C, 0.30 g, 95% yield): ^1H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.59 (d, 1H, J = 8 Hz, ArH), 8.48 (d, 1H, J = 8 Hz, ArH), 8.12 (d, 1H, J = 4 Hz, ArH), 7.84-7.77 (m, 2H, ArH), 7.33 (d, 1H, J = 4 Hz, ArH), 2.63 (s, 3H, CH_3).



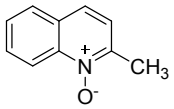
6d

The compound is known [5] and was prepared according to general procedure 3.1 (yellow solid, mp. 65 °C, 0.26 g, 83% yield): ^1H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.51 (d, 1H, J = 8 Hz, ArH), 8.43 (d, 1H, J = 8 Hz, ArH), 7.84 (s, 1H, ArH), 7.83 (d, 1H, J = 8 Hz, ArH), 7.65 (d, 1H, J = 8 Hz, ArH), 7.45-7.41 (m, 1H, ArH), 3.33 (s, 3H, CH_3).



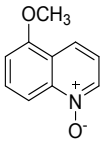
6e

The compound is known [6] and was prepared according to general procedure 3.1 (yellow solid, mp. 52 °C, 0.29 g, 92% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.55 (s, 1H, ArH), 8.50 (d, 1H, J = 8 Hz, ArH), 7.97 (d, 1H, J = 8 Hz, ArH), 7.77-7.67 (m, 3H, ArH), 2.41 (s, 3H, CH_3).



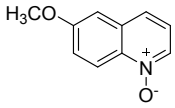
6f

The compound is known [6] and was prepared according to general procedure 3.1 (yellow solid, mp. 70 °C, 0.29 g, 90% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.56 (d, 1H, J = 8 Hz, ArH), 8.05 (d, 1H, J = 8 Hz, ArH), 7.86-7.78 (m, 2H, ArH), 7.70-7.66 (m, 1H, ArH), 7.59-7.57 (m, 1H, ArH), 2.58 (s, 3H, CH_3).



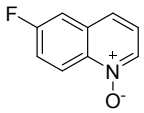
6g

The compound is known [7] and was prepared according to general procedure 3.1 (yellow solid, mp. 101 °C, 0.30 g, 87% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.58 (d, 1H, J = 4 Hz, ArH), 8.08 (d, 1H, J = 8 Hz, ArH), 8.02 (d, 1H, J = 8 Hz, ArH), 7.75-7.73 (m, 1H, ArH), 7.44-7.40 (m, 1H, ArH), 7.21 (d, 1H, J = 4 Hz, ArH), 4.02 (s, 3H, OCH_3).



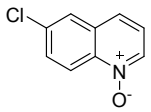
6h

The compound is known [5] and was prepared according to general procedure 3.1 (yellow solid, mp. 89 °C, 0.30 g, 85% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.45-8.41 (m, 2H, ArH), 7.82 (d, 1H, J = 8 Hz, ArH), 7.50 (d, 1H, J = 4 Hz, ArH), 7.44-7.40 (m, 2H, ArH), 3.92 (s, 3H, OCH_3).



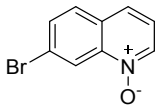
6i

The compound is known [5] and was prepared according to general procedure 3.1 (yellow solid, mp. 109 °C, 0.26 g, 81% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.80-8.76 (m, 1H, ArH), 8.49 (d, 1H, J = 4 Hz, ArH), 7.68 (d, 1H, J = 8 Hz, ArH), 7.54-7.49 (m, 2H, ArH), 7.35-7.28 (m, 1H, ArH).



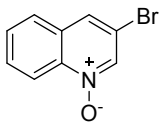
6j

The compound is known [5] and was prepared according to general procedure 3.1 (yellow solid, mp. 123 °C, 0.30 g, 83% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.61 (d, 1H, J = 4 Hz, ArH), 8.53 (d, 1H, J = 8 Hz, ArH), 8.26 (s, 1H, ArH), 7.92-7.89 (m, 1H, ArH), 7.84 (d, 1H, J = 4 Hz, ArH), 7.55-7.52 (m, 1H, ArH).



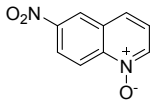
6k

The compound is known [8] and was prepared according to general procedure 3.1 (yellow solid, mp. 142 °C, 0.37 g, 82% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.69 (s, 1H, ArH), 8.63 (d, 1H, J = 4 Hz, ArH), 8.08 (d, 1H, J = 8 Hz, ArH), 7.97 (d, 1H, J = 8 Hz, ArH), 7.92 (d, 1H, J = 4 Hz, ArH), 7.55-7.51 (m, 1H, ArH).



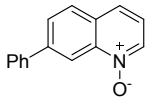
6l

The compound is known [9] and was prepared according to general procedure 3.1 (yellow solid, mp. 106 °C, 0.38 g, 85% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.66 (s, 1H, ArH), 8.48 (d, 1H, J = 8 Hz, ArH), 8.29 (s, 1H, ArH), 8.05 (d, 1H, J = 8 Hz, ArH), 7.87-7.76 (m, 2H, ArH).



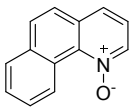
6m

The compound is known [3] and was prepared according to general procedure 3.1 (yellow solid, mp. 228 °C, 0.29 g, 75% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 9.16 (d, 1H, J = 4 Hz, ArH), 8.78-8.70 (m, 2H, ArH), 8.50 (d, 1H, J = 4 Hz, ArH), 8.24 (d, 1H, J = 8 Hz, ArH), 7.69-7.65 (m, 1H, ArH).



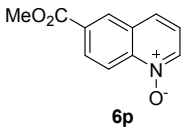
6n

The compound is known [7] and was prepared according to general procedure 3.1 (yellow solid, mp. 121 °C, 0.40 g, 91% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 9.00 (s, 1H, ArH), 8.57 (d, 1H, J = 4 Hz, ArH), 7.93 (s, 2H, ArH), 7.80-7.75 (m, 3H, ArH), 7.53-7.49 (m, 2H, ArH), 7.45-7.41 (m, 1H, ArH), 7.31-7.27 (m, 1H, ArH).



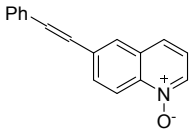
6o

The compound is known [5] and was prepared according to general procedure 3.1 (yellow solid, mp. 95 °C, 0.36 g, 93% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 10.87-10.85 (m, 1H, ArH), 8.68 (d, 1H, J = 8 Hz, ArH), 7.96-7.93 (m, 1H, ArH), 7.92-7.88 (m, 1H, ArH), 7.82-7.76 (m, 3H, ArH), 7.67 (d, 1H, J = 8 Hz, ArH), 7.44-7.40 (m, 1H, ArH).



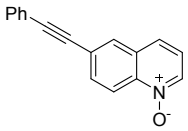
6p

The compound is known [6] and was prepared according to general procedure 3.1 (yellow solid, mp. 139 °C, 0.31 g, 76% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.78 (s, 1H, ArH), 8.71-8.70 (m, 1H, ArH), 8.64-8.61 (m, 1H, ArH), 8.26-8.24 (m, 1H, ArH), 8.16-8.14 (m, 1H, ArH), 7.60-7.56 (m, 1H, ArH), 3.95 (s, 3H, OCH_3).



6q

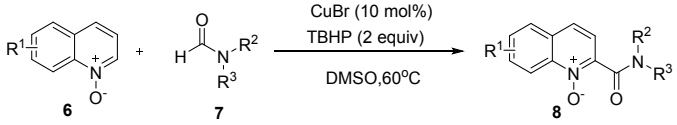
The compound is known [10] and was prepared according to general procedure 3.1 (yellow solid, mp. 138 °C, 0.37 g, 75% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.74 (d, 1H, J = 12 Hz, ArH), 8.48 (d, 1H, J = 8 Hz, ArH), 8.01-7.99 (m, 1H, ArH), 7.85 (s, 1H, ArH), 7.73 (d, 1H, J = 12 Hz, ArH), 7.58-7.56 (m, 2H, ArH), 7.42-7.39 (m, 2H, ArH), 7.34-7.25 (m, 4H, ArH+2CH).



6r

The compound is known [11] and was prepared according to general procedure 3.1 (yellow solid, mp. 128 °C, 0.43 g, 87% yield): ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 8.63 (d, 1H, J = 4 Hz, ArH), 8.56 (d, 1H, J = 8 Hz, ArH), 8.36 (s, 1H, ArH), 7.97-7.92 (m, 2H, ArH), 7.65-7.64 (m, 2H, ArH), 7.55-7.49 (m, 4H, ArH).

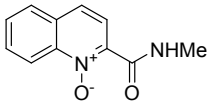
3.2 General procedure for the copper-catalyzed carbamoylation of quinoline *N*-oxide



6 **7** **8**

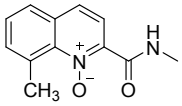
To a reaction tube were added quinoline *N*–oxides **6** (0.2 mmol) and formamide **7** (10 equiv), CuBr (10 mol %), TBHP (5.0–6.0 M in decane) (2 equiv) and DMSO (1 mL). Then the

tube was charged with air, and was stirred at 60 °C until complete consumption of starting material as monitored by TLC and GC–MS analysis. After the reaction was finished, the reaction mixture was washed with brine. The aqueous phase was re–extracted with ethyl acetate. The combined organic extracts were dried over Na_2SO_4, concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography to afford the desired quinoline–*N*–oxide–2–carboxamides **8**.



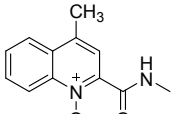
8a

The compound is known[12] and was prepared according to general procedure 3.2 (yellow oil, 36 mg, 91% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.45 (s, 1H, NH), 8.79 (d, 1H, *J* = 8 Hz, ArH), 8.44 (d, 1H, *J* = 12 Hz, ArH), 7.91 (d, 1H, *J* = 8 Hz, ArH), 7.87–7.81 (m, 2H, ArH), 7.74–7.70 (m, 1H, ArH), 3.12 (d, 3H, *J* = 4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.2, 131.1, 130.7, 129.8, 128.2, 126.5, 122.7, 120.2, 26.3.



8b

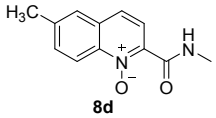
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8b** (yellow oil, 36 mg, 85% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.37 (s, 1H, NH), 8.35 (d, 1H, *J* = 8 Hz, ArH), 7.74 (d, 1H, *J* = 12 Hz, ArH), 7.69–7.67(m, 1H, ArH), 7.53–7.48 (m, 2H, ArH), 3.17 (s, 3H, CH_3), 3.09 (d, 3H, *J* = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.6, 141.7, 138.4, 134.3, 132.6, 129.2, 126.9, 126.7, 122.6, 26.2, 25.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{13}N_2O_2$ $[M + H]^+$ 217.0977, found 217.0994.



8c

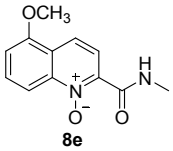
The compound is known[12] and was prepared according to general procedure 3.2 (yellow oil, 39 mg, 90% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.60 (s, 1H, NH), 8.43 (d, 1H, *J* = 8 Hz, ArH), 8.26 (s, 1H, ArH), 8.01 (d, 1H, *J* = 8 Hz, ArH), 7.85–7.81 (m, 1H, ArH), 7.77–7.73 (m, 1H, ArH), 3.11 (d, 3H, *J* = 4 Hz, CH_3), 2.72 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)

161.3, 141.3, 136.6, 135.2, 130.7, 130.3, 129.5, 124.8, 122.8, 120.6, 26.3, 18.4.



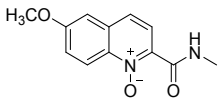
8d

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8d** (yellow oil, 39 mg, 92% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.50 (s, 1H, NH), 8.65 (d, 1H, J = 8 Hz, ArH), 8.38 (d, 1H, J = 8 Hz, ArH), 7.75 (d, 1H, J = 8 Hz, ArH), 7.65–7.63 (m, 2H, ArH), 3.10 (d, 3H, J = 8 Hz, CH_3), 2.56 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.3, 140.3, 136.8, 133.3, 130.9, 127.1, 126.0, 122.7, 119.9, 26.3, 21.5; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{13}N_2O_2$ $[M + H]^+$ 217.0977, found 217.0959.



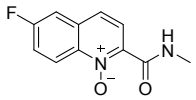
8e

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8e** (yellow solid, mp. 171 °C, 39 mg, 85% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.50 (s, 1H, NH), 8.38 (d, 1H, J = 8 Hz, ArH), 8.32 (d, 1H, J = 12 Hz, ArH), 8.24 (d, 1H, J = 8 Hz, ArH), 7.74–7.70 (m, 1H, ArH), 7.02 (d, 1H, J = 8 Hz, ArH), 4.04 (s, 3H, OCH_3), 3.11 (d, 3H, J = 4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 164.6, 161.2, 155.7, 142.6, 137.7, 131.4, 123.4, 121.5, 111.6, 107.5, 56.2, 26.3; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{13}N_2O_3$ $[M + H]^+$ 233.0926, found 233.0927.



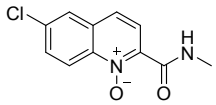
8f

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8f** (yellow solid, mp. 152 °C, 39 mg, 85% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.30 (s, 1H, NH), 8.58 (d, 1H, J = 8 Hz, ArH), 8.29 (d, 1H, J = 8 Hz, ArH), 7.64 (d, 1H, J = 8 Hz, ArH), 7.34 (d, 1H, J = 8 Hz, ArH), 7.03 (s, 1H, ArH), 3.88 (s, 3H, OCH_3), 3.02 (d, 3H, J = 4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.3, 159.3, 136.2, 134.8, 131.4, 124.5, 122.5, 122.3, 120.8, 104.8, 54.8, 25.3; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{13}N_2O_3$ $[M + H]^+$ 233.0926, found 233.0927.



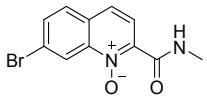
8g

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8g** (yellow oil, 36 mg, 83% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.25 (s, 1H, NH), 8.81–8.78 (m, 1H, ArH), 8.44 (d, 1H, *J* = 8 Hz, ArH), 7.78 (d, 1H, *J* = 12 Hz, ArH), 7.58–7.51 (m, 2H, ArH), 3.11 (d, 3H, *J* = 4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 163.8, 161.3, 160.9, 138.8, 137.1, 132.1, 125.6, 124.0, 123.5, 121.1, 120.8, 111.9, 26.3; HRMS (ESI–Q–TOF) exact mass calcd for $C_{11}H_{10}FN_2O_2$ $[M + H]^+$ 221.0726, found 221.0708.



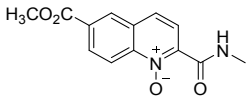
8h

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8h** (yellow oil, 41 mg, 88% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.30 (s, 1H, NH), 8.70 (d, 1H, *J* = 8 Hz, ArH), 8.44 (d, 1H, *J* = 8 Hz, ArH), 7.87 (s, 1H, ArH), 7.75–7.71 (m, 2H, ArH), 3.11 (d, 3H, *J* = 4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.8, 140.3, 137.6, 136.3, 131.9, 131.4, 126.9, 125.3, 124.0, 122.1, 26.4; HRMS (ESI–Q–TOF) exact mass calcd for $C_{11}H_{10}ClN_2O_2$ $[M + H]^+$ 237.0431, found 237.0421.



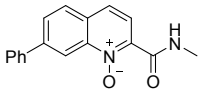
8i

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8i** (yellow solid, mp. 148 °C, 46 mg, 83% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.30 (s, 1H, NH), 8.96 (s, 1H, ArH), 8.43 (d, 1H, *J* = 8 Hz, ArH), 7.81–7.75 (m, 3H, ArH), 3.11 (d, 3H, *J* = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.7, 142.2, 138.0, 133.4, 129.4, 129.3, 126.0, 125.8, 123.1, 123.0, 26.4; HRMS (ESI–Q–TOF) exact mass calcd for $C_{11}H_{10}BrN_2O_2$ $[M + H]^+$ 280.9926, found 280.9924.



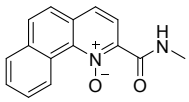
8j

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8j** (yellow solid, mp. 175 °C, 41 mg, 80% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.33 (s, 1H, NH), 8.82 (d, 1H, J = 8 Hz, ArH), 8.62 (s, 1H, ArH), 8.49 (d, 1H, J = 8 Hz, ArH), 8.38 (d, 1H, J = 8 Hz, ArH), 7.94 (d, 1H, J = 8 Hz, ArH), 4.03 (s, 3H, OCH_3), 3.12 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 165.5, 160.8, 143.5, 138.8, 131.3, 130.8, 130.6, 130.2, 127.0, 123.6, 120.8, 52.8, 26.4; HRMS (ESI–Q–TOF) exact mass calcd for $C_{13}H_{13}N_2O_4$ $[M + H]^+$ 261.0875, found 261.0878.



8k

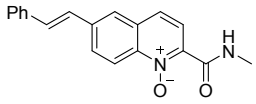
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8k** (yellow solid, mp. 178 °C, 49 mg, 88% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.47 (s, 1H, NH), 9.01 (s, 1H, ArH), 8.42 (d, 1H, J = 8 Hz, ArH), 7.99–7.94 (m, 2H, ArH), 7.85 (d, 1H, J = 8 Hz, ArH), 7.79–7.77 (m, 2H, ArH), 7.54–7.51 (m, 2H, ArH), 7.47–7.43 (m, 1H, ArH), 3.12 (d, 3H, J = 4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.2, 144.2, 142.2, 139.2, 129.8, 129.3, 129.2, 128.7, 127.6, 126.2, 122.5, 117.6, 26.3; HRMS (ESI–Q–TOF) exact mass calcd for $C_{17}H_{15}N_2O_2$ $[M + H]^+$ 279.1133, found 279.1115.



8l

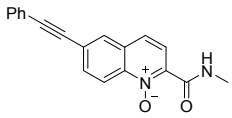
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8l** (yellow oil, 40 mg, 80% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.49 (s, 1H, NH), 10.66–10.64 (m, 1H, ArH), 8.63 (d, 1H, J = 8 Hz, ArH), 8.00–7.97 (m, 1H, ArH), 7.95 (d, 1H, J = 8 Hz, ArH), 7.89 (d, 1H, J = 8 Hz, ArH), 7.83–7.79 (m, 2H, ArH), 7.69 (d, 1H, J = 8 Hz, ArH), 3.15 (d, 3H, J = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.7, 134.7, 132.2, 132.0, 129.3, 128.7, 128.5, 128.2, 126.3, 126.2, 124.9, 124.2, 26.4; HRMS (ESI–Q–TOF) exact mass calcd for $C_{15}H_{13}N_2O_2$ [M +

H]$^+$ 253.0977, found 253.0962.



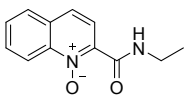
8m

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8m** (yellow solid, mp. 175 °C, 49 mg, 81% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.42 (s, 1H, NH), 8.73 (d, 1H, *J* = 8 Hz, ArH), 8.41 (d, 1H, *J* = 8 Hz, ArH), 8.04–8.02 (m, 1H, ArH), 7.85–7.80 (m, 2H, ArH), 7.58–7.56 (m, 2H, ArH), 7.43–7.39 (m, 2H, ArH), 7.35–7.25 (m, 3H, ArH+2CH), 3.11 (d, 3H, *J* = 4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.2, 141.0, 139.0, 136.4, 132.6, 131.3, 129.0, 128.9, 128.7, 126.9, 126.4, 125.6, 123.2, 120.5, 26.3; HRMS (ESI–Q–TOF) exact mass calcd for $C_{19}H_{17}N_2O_2$ [M + H]$^+$ 305.1290, found 305.1291.



8n

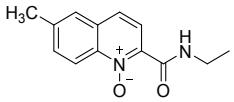
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8n** (yellow solid, mp. 185 °C, 46 mg, 77% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.34 (s, 1H, NH), 8.73 (d, 1H, *J* = 12 Hz, ArH), 8.42 (d, 1H, *J* = 8 Hz, ArH), 8.03 (s, 1H, ArH), 7.88 (d, 1H, *J* = 8 Hz, ArH), 7.78 (d, 1H, *J* = 8 Hz, ArH), 7.58–7.56 (m, 2H, ArH), 7.41–7.38 (m, 3H, ArH), 3.10 (d, 3H, *J* = 4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.0, 140.9, 137.7, 133.8, 131.8, 130.9, 130.6, 129.1, 128.5, 125.9, 125.4, 123.5, 122.3, 120.4, 93.1, 87.7, 26.3; HRMS (ESI–Q–TOF) exact mass calcd for $C_{19}H_{15}N_2O_2$ [M + H]$^+$ 303.1133, found 303.1128.



8o

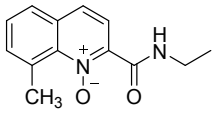
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8o** (yellow oil, 35 mg, 81% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.49 (s, 1H, NH), 8.78 (d, 1H, *J* = 8 Hz, ArH), 8.43 (d, 1H, *J* = 8 Hz, ArH), 7.90 (d, 1H, *J* = 8 Hz, ArH), 7.86–7.81 (m, 2H, ArH), 7.74–7.70 (m, 1H, ArH), 3.63–3.56 (m, 2H, CH_2), 1.34 (t, 3H, *J* = 8 Hz, CH_3); ^{13}C NMR (100 MHz,

$CDCl_3$) δ (ppm) 160.3, 141.8, 137.6, 131.1, 130.7, 129.7, 128.2, 126.5, 122.7, 120.1, 34.7, 14.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{13}N_2O_2$ $[M + H]^+$ 217.0977, found 217.0977.



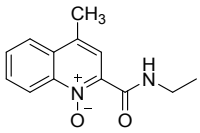
8p

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8p** (yellow oil, 36 mg, 79% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.40 (s, 1H, NH), 8.58 (d, 1H, J = 8 Hz, ArH), 8.31 (d, 1H, J = 8 Hz, ArH), 7.69 (d, 1H, J = 8 Hz, ArH), 7.58–7.56 (m, 2H, ArH), 3.55–3.48 (m, 2H, CH_2), 2.49 (s, 3H, CH_3), 1.25 (t, 3H, J = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.4, 140.3, 137.0, 133.4, 130.9, 127.2, 126.1, 122.8, 119.8, 34.6, 21.5, 14.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{13}H_{15}N_2O_2$ $[M + H]^+$ 231.1134, found 231.1143.



8q

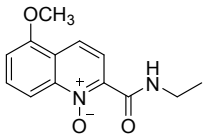
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8q** (yellow oil, 36 mg, 80% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.40 (s, 1H, NH), 8.34 (d, 1H, J = 8 Hz, ArH), 7.72 (d, 1H, J = 8 Hz, ArH), 7.68–7.65 (m, 1H, ArH), 7.51–7.47 (m, 2H, ArH), 3.61–3.54 (m, 2H, CH_2), 3.16 (s, 3H, CH_3), 1.32 (t, 3H, J = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.8, 141.8, 138.5, 134.3, 132.6, 129.1, 126.9, 122.7, 34.6, 25.7, 14.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{13}H_{15}N_2O_2$ $[M + H]^+$ 231.1134, found 231.1143.



8r

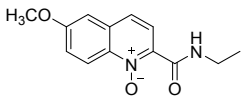
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8r** (yellow solid, mp. 88 °C, 39 mg, 85% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.70 (s, 1H, NH), 8.85–8.83 (m, 1H, ArH), 8.27 (s, 1H, ArH), 8.02 (d, 1H, J = 8 Hz, ArH), 7.86–7.82 (m, 1H, ArH), 7.77–7.73 (m, 1H, ArH), 3.63–3.56 (m, 2H, CH_2), 2.73 (s, 3H, CH_3), 1.33 (t, 3H, J = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.4, 141.3, 135.3, 130.8, 130.3, 129.5, 124.9, 122.8, 120.6, 34.7,

18.4, 14.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{13}H_{15}N_2O_2$ $[M + H]^+$ 231.1134, found 231.1121.



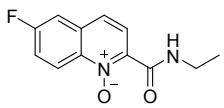
8s

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8s** (yellow solid, mp. 155 °C, 39 mg, 80% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.50 (s, 1H, NH), 8.38 (d, 1H, J = 8 Hz, ArH), 8.32 (d, 1H, J = 12 Hz, ArH), 8.24 (d, 1H, J = 8 Hz, ArH), 7.74–7.70 (m, 1H, ArH), 7.02 (d, 1H, J = 8 Hz, ArH), 4.04 (s, 3H, OCH_3), 3.62–3.55 (m, 2H, CH_2), 1.33 (t, 3H, J = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.4, 155.7, 142.7, 137.9, 131.4, 123.5, 121.6, 121.5, 111.6, 107.5, 56.2, 34.7, 14.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{13}H_{14}N_2O_3$ $[M + H]^+$ 247.1083, found 247.1100.



8t

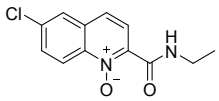
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8t** (yellow oil, 36 mg, 75% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.40 (s, 1H, NH), 8.68 (d, 1H, J = 12 Hz, ArH), 8.38 (d, 1H, J = 8 Hz, ArH), 7.74 (d, 1H, J = 12 Hz, ArH), 7.45 (d, 1H, J = 4 Hz, ArH), 7.12 (s, 1H, ArH), 3.97 (s, 3H, OCH_3), 3.61–3.55 (m, 2H, CH_2), 1.32 (t, 3H, J = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.4, 137.2, 135.9, 132.4, 125.5, 123.5, 123.4, 121.8, 105.9, 55.9, 34.6, 14.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{13}H_{15}N_2O_3$ $[M + H]^+$ 247.1083, found 247.1080.



8u

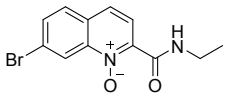
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8u** (yellow oil, 33 mg, 71% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.30 (s, 1H, NH), 8.82–8.79 (m, 1H, ArH), 8.45 (d, 1H, J = 8 Hz, ArH), 7.78 (d, 1H, J = 8 Hz, ArH), 7.59–7.51 (m, 2H, ArH), 3.62–3.55 (m,

2H, CH_2), 1.33 (t, 3H, J = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 163.8, 161.2, 160.0, 138.8, 137.2, 132.1, 125.6, 124.1, 123.4, 121.1, 120.8, 111.9, 34.7, 14.6; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{12}FN_2O_2$ $[M + H]^+$ 235.0833, found 235.0831.



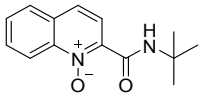
8v

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8v** (yellow solid, mp. 139 °C, 37 mg, 75% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.30 (s, 1H, NH), 8.72 (d, 1H, J = 12 Hz, ArH), 8.45 (d, 1H, J = 8 Hz, ArH), 7.89 (s, 1H, ArH), 7.76–7.73 (m, 2H, ArH), 3.62–3.55 (m, 2H, CH_2), 1.34 (t, 3H, J = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.2, 140.3, 137.8, 136.3, 131.9, 131.5, 128.8, 127.8, 127.5, 126.9, 125.3, 124.2, 122.1, 43.9, 29.3; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{12}ClN_2O_2$ $[M + H]^+$ 251.0587, found 251.0591.



8w

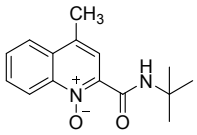
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8w** (yellow solid, mp. 125 °C, 46 mg, 77% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.30 (s, 1H, NH), 8.96 (s, 1H, ArH), 8.43 (d, 1H, J = 8 Hz, ArH), 7.82–7.75 (m, 3H, ArH), 3.62–3.56 (m, 2H, CH_2), 1.33 (t, 3H, J = 8 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.8, 142.1, 138.1, 133.4, 129.4, 129.3, 126.0, 125.8, 123.1, 123.0, 34.7, 14.6; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{12}BrN_2O_2$ $[M + H]^+$ 295.0082, found 295.0066.



8x

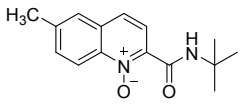
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8x** (yellow solid, mp. 115 °C, 34 mg, 73% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.60 (s, 1H, NH), 8.78 (d, 1H, J = 8 Hz, ArH), 8.42 (d, 1H, J = 8 Hz, ArH), 7.91–7.81 (m, 3H, ArH), 7.73–7.69 (m, 1H, ArH), 1.55 (s, 9H, $3CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.0, 141.7, 138.5, 131.1, 130.6, 129.6, 128.2, 126.6, 122.5, 120.0, 51.5, 28.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{14}H_{17}N_2O_2$

$[M + H]^+$ 245.1290, found 245.1270.



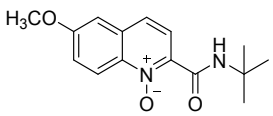
8y

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8y** (yellow solid, mp. 126 °C, 37 mg, 71% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.80 (s, 1H, NH), 8.43 (d, 1H, J = 8 Hz, ArH), 8.26 (s, 1H, ArH), 8.01 (d, 1H, J = 8 Hz, ArH), 7.86–7.81 (m, 1H, ArH), 7.76–7.72 (m, 1H, ArH), 2.71 (s, 3H, CH_3), 1.54 (s, 9H, $3CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.1, 141.2, 137.7, 135.4, 130.8, 130.3, 129.4, 124.8, 122.7, 120.6, 51.5, 28.7, 18.4; HRMS (ESI–Q–TOF) exact mass calcd for $C_{15}H_{19}N_2O_2$ $[M + H]^+$ 259.1447, found 259.1466.



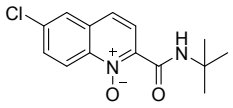
8z

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8z** (yellow solid, mp. 115 °C, 37 mg, 72% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.50 (s, 1H, NH), 8.56 (d, 1H, J = 8 Hz, ArH), 8.29 (d, 1H, J = 8 Hz, ArH), 7.68 (d, 1H, J = 8 Hz, ArH), 7.56–7.54 (m, 2H, ArH), 2.48 (s, 3H, CH_3), 1.46 (s, 9H, $3CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)159.1, 140.2, 137.8, 133.3, 130.8, 127.1, 126.2, 122.5, 119.8, 51.4, 28.7, 21.4; HRMS (ESI–Q–TOF) exact mass calcd for $C_{15}H_{18}N_2O_2$ $[M + H]^+$ 259.1447, found 259.1462.



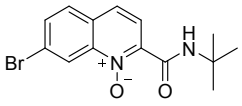
8aa

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8aa** (yellow oil, 43 mg, 79% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.55 (s, 1H, NH), 8.67 (d, 1H, J = 8 Hz, ArH), 8.37 (d, 1H, J = 4 Hz, ArH), 7.74 (d, 1H, J = 4 Hz, ArH), 7.43 (d, 1H, J = 8 Hz, ArH), 7.12 (s, 1H, ArH), 3.97 (s, 3H, OCH_3), 1.53 (s, 9H, $3CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.2, 159.2, 137.2, 136.8, 132.4, 125.7, 123.5, 123.1, 121.7, 105.9, 55.8, 51.4, 50.2, 29.6, 28.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{15}H_{19}N_2O_3$ $[M + H]^+$ 275.1396, found 275.1379.



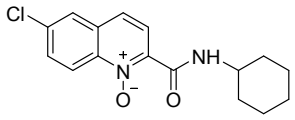
8ab

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8ab** (yellow solid, mp. 118 °C, 38 mg, 69% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.40 (s, 1H, NH), 8.72 (d, 1H, J = 8 Hz, ArH), 8.44 (d, 1H, J = 8 Hz, ArH), 7.88 (s, 1H, ArH), 7.76–7.73 (m, 2H, ArH), 1.54 (s, 9H, $3CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 158.6, 140.2, 138.6, 136.1, 131.9, 131.4, 126.9, 125.3, 123.9, 122.0, 51.6, 28.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{14}H_{16}ClN_2O_2$ [M + H]$^+$ 279.0900, found 279.0920.



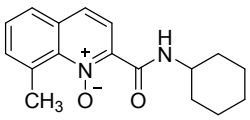
8ac

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8ac** (yellow solid, mp. 127 °C, 42 mg, 65% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.50 (s, 1H, NH), 8.99 (s, 1H, ArH), 8.43 (d, 1H, J = 12 Hz, ArH), 7.82–7.75 (m, 3H, ArH), 1.54 (s, 9H, $3CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 158.6, 142.1, 139.0, 133.3, 129.4, 129.3, 126.1, 125.9, 123.0, 122.9, 51.6, 28.7; HRMS (ESI–Q–TOF) exact mass calcd for $C_{14}H_{16}BrN_2O_2$ [M + H]$^+$ 323.0395, found 323.0371.



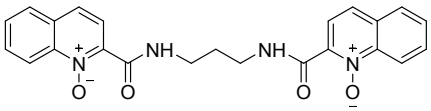
8ad

The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8ad** (yellow solid, mp. 128 °C, 41 mg, 68% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.36 (d, 1H, J = 8 Hz, NH), 8.73 (d, 1H, J = 8 Hz, ArH), 8.46 (d, 1H, J = 8 Hz, ArH), 7.90 (s, 1H, ArH), 7.77–7.74 (m, 2H, ArH), 4.10–4.07 (m, 1H, CH), 2.05–2.03 (m, 2H, CH_2), 1.80–1.75 (m, 2H, CH_2), 1.65–1.62 (m, 1H, CH), 1.49–1.34 (m, 5H, CH+2CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.0, 140.3, 137.9, 136.2, 131.9, 131.4, 126.9, 125.3, 124.2, 122.0, 48.6, 32.7, 25.7, 24.6; HRMS (ESI–Q–TOF) exact mass calcd for $C_{16}H_{18}ClN_2O_2$ [M + H]$^+$ 305.1057, found 305.1050.



8ae

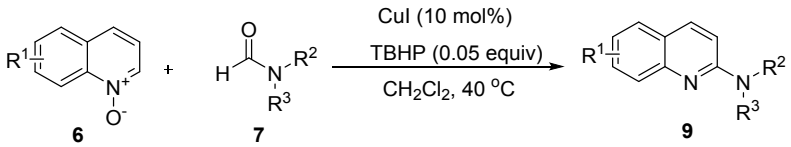
The compound was prepared according to general procedure 3.2 and purified by flash column chromatography (2:1 petroleum ether: ethyl acetate) to afford **8ae** (yellow oil, 41 mg, 73% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.43 (d, 1H, J = 4 Hz, NH), 8.33 (d, 1H, J = 12 Hz, ArH), 7.69 (d, 1H, J = 12 Hz, ArH), 7.65–7.63 (m, 1H, ArH), 7.49–7.44 (m, 2H, ArH), 4.11–4.04 (m, 1H, CH), 3.16 (s, 3H, CH_3), 2.07–2.04 (m, 2H, CH_2), 1.80–1.77 (m, 2H, CH_2), 1.66–1.63 (m, 1H, CH), 1.49–1.42 (m, 4H, $2CH_2$), 1.34–1.32 (m, 1H, CH); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.8, 141.8, 138.7, 134.3, 132.5, 129.1, 126.9, 126.5, 122.7, 48.6, 32.8, 25.7, 24.8; HRMS (ESI–Q–TOF) exact mass calcd for $C_{17}H_{21}N_2O_2$ $[M + H]^+$ 285.1603, found 285.1590.



8af

The compound is known[13] and was prepared according to general procedure 3.2 (yellow solid, 56 mg, 68% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 11.75 (s, 2H, NH), 8.67 (d, 2H, J = 8 Hz, ArH), 8.44 (d, 2H, J = 8 Hz, ArH), 7.88 (d, 2H, J = 8 Hz, ArH), 7.83 (d, 2H, J = 8 Hz, ArH), 7.78–7.74 (m, 2H, ArH), 7.71–7.67 (m, 2H, ArH), 3.76–3.71 (m, 4H, $2CH_2$), 2.14–2.07 (m, 2H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.8, 141.8, 137.5, 131.0, 130.7, 129.7, 128.1, 126.3, 122.8, 120.2, 37.7, 29.3.

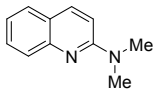
3.3 General procedure for the copper-catalyzed amination of quinoline *N*-oxide



6 **7** **9**

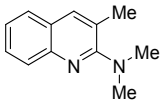
To a reaction tube were added quinoline *N*–oxides **6** (0.2 mmol) and formamide **7** (10 equiv), CuI (10 mol %), TBHP (5–6 M in decane) (0.05 equiv) and CH_2Cl_2 (1 mL). Then the tube was charged with air, and was stirred at 40 °C until complete consumption of starting material as monitored by TLC and GC–MS analysis. After the reaction was finished, the reaction mixture was washed with brine. The aqueous phase was re–extracted with ethyl acetate. The combined organic extracts were dried over Na_2SO_4, concentrated in vacuum, and

the resulting residue was purified by silica gel column chromatography to afford the desired quinoline–2–amines **9**.



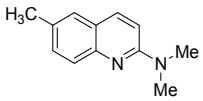
9a

The compound is known[14] and was prepared according to general procedure 3.3 (25 mg, 73% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.78 (d, 1H, J = 8 Hz, ArH), 7.63 (d, 1H, J = 8 Hz, ArH), 7.50 (d, 1H, J = 8 Hz, ArH), 7.46–7.42 (m, 1H, ArH), 7.12–7.09 (m, 1H, ArH), 6.81 (d, 1H, J = 8 Hz, ArH), 3.15 (s, 6H, $2CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.6, 137.2, 129.4, 127.2, 126.3, 122.4, 121.7, 109.1, 38.1.



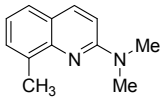
9b

The compound was prepared according to general procedure 3.3 and purified by flash column chromatography (5:1 petroleum ether: ethyl acetate) to afford **9b** (yellow oil, 26 mg, 69% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.81 (d, 1H, J = 8 Hz, ArH), 7.74 (s, 1H, ArH), 7.59 (d, 1H, J = 8 Hz, ArH), 7.54–7.50 (m, 1H, ArH), 7.30–7.26 (m, 1H, ArH), 3.00 (s, 6H, $2CH_3$), 2.45 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.9, 145.9, 138.1, 128.3, 127.1, 126.3, 125.3, 124.6, 123.5, 41.7, 19.9; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{15}N_2$ $[M + H]^+$ 187.1235, found 187.1217.



9c

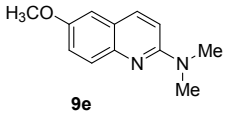
The compound is known[15] and was prepared according to general procedure 3.3 (25 mg, 68% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.79 (d, 1H, J = 8 Hz, ArH), 7.65 (d, 1H, J = 8 Hz, ArH), 7.37–7.35 (m, 2H, ArH), 6.87 (d, 1H, J = 12 Hz, ArH), 3.20 (s, 6H, $2CH_3$), 2.44 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.2, 136.8, 131.5, 131.1, 126.4, 126.0, 122.3, 109.1, 38.2, 21.1.



9d

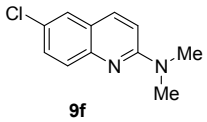
The compound was prepared according to general procedure 3.3 and purified by flash column

chromatography (5:1 petroleum ether: ethyl acetate) to afford **9d** (yellow oil, 28 mg, 74% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.83 (d, 1H, *J* = 8 Hz, ArH), 7.44–7.38 (m, 2H, ArH), 7.10–7.06 (m, 1H, ArH), 6.87 (d, 1H, *J* = 8 Hz, ArH), 3.21 (s, 6H, $2CH_3$), 2.65 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 156.8, 146.8, 137.5, 134.1, 129.5, 125.1, 122.0, 121.2, 108.5, 37.9, 17.8; HRMS (ESI–Q–TOF) exact mass calcd for $C_{12}H_{15}N_2$ $[M + H]^+$ 187.1235, found 187.1242.



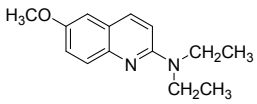
9e

The compound is known[15] and was prepared according to general procedure 3.3 (26 mg, 65% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.80 (d, 1H, *J* = 12 Hz, ArH), 7.66 (d, 1H, *J* = 12 Hz, ArH), 7.22-7.19 (m, 1H, ArH), 6.95 (d, 1H, *J* = 4 Hz, ArH), 6.88 (d, 1H, *J* = 8 Hz, ArH), 3.87 (s, 3H, OCH_3), 3.20 (s, 6H, $2CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 156.7, 154.5, 136.2, 127.7, 122.6, 121.0, 109.4, 106.2, 55.5, 38.2.



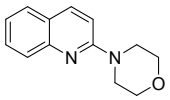
9f

The compound is known[15] and was prepared according to general procedure 3.3 (26 mg, 60% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.76 (d, 1H, *J* = 8 Hz, ArH), 7.62 (d, 1H, *J* = 8 Hz, ArH), 7.54 (s, 1H, ArH), 7.44 (d, 1H, *J* = 8 Hz, ArH), 6.90 (d, 1H, *J* = 12 Hz, ArH), 3.22 (s, 6H, $2CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.6, 136.2, 130.0, 127.8, 126.6, 126.0, 122.9, 109.9, 38.0.



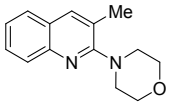
9g

The compound is known[16] and was prepared according to general procedure 3.3 (23 mg, 50% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.45 (d, 1H, *J* = 8 Hz, ArH), 7.59 (d, 1H, *J* = 8 Hz, ArH), 7.19–7.16 (m, 1H, ArH), 6.93 (d, 1H, *J* = 4 Hz, ArH), 6.81 (d, 1H, *J* = 8 Hz, ArH), 3.86 (s, 3H, OCH_3), 3.66–3.60 (m, 4H, $2CH_2$), 1.23 (t, 6H, *J* = 8 Hz, $2CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 154.4, 136.2, 127.7, 122.5, 120.7, 109.4, 106.3, 55.5, 42.4, 13.3.



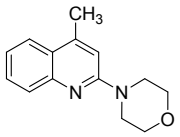
9h

The compound is known[17] and was prepared according to general procedure 3.3 (25 mg, 60% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.91 (d, 1H, J = 8 Hz, ArH), 7.72 (d, 1H, J = 8 Hz, ArH), 7.61 (d, 1H, J = 8 Hz, ArH), 7.57–7.53 (m, 1H, ArH), 7.27–7.25 (m, 1H, ArH), 6.95 (d, 1H, J = 8 Hz, ArH), 3.87–3.84 (m, 4H, $2CH_2$), 3.72–3.69 (m, 2H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.5, 147.7, 137.6, 129.6, 127.2, 126.8, 123.3, 122.7, 109.3, 66.9, 45.6.



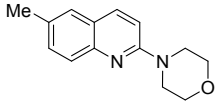
9i

The compound was prepared according to general procedure 3.3 and purified by flash column chromatography (5:1 petroleum ether: ethyl acetate) to afford **9i** (yellow oil, 26 mg, 60% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.88 (d, 1H, J = 8 Hz, ArH), 7.82 (s, 1H, ArH), 7.64 (d, 1H, J = 8 Hz, ArH), 7.58–7.54 (m, 1H, ArH), 7.37–7.33 (m, 1H, ArH), 3.92–3.89 (m, 4H, $2CH_2$), 3.35–3.33 (m, 4H, $2CH_2$), 2.44 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 156.1, 144.9, 136.1, 131.2, 130.7, 125.4, 125.3, 122.2, 108.3, 65.9, 44.8, 20.2; HRMS (ESI–Q–TOF) exact mass calcd for $C_{14}H_{17}N_2O$ $[M + H]^+$ 229.1341, found 229.1319.



9j

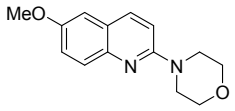
The compound is known[18] and was prepared according to general procedure 3.3 (30 mg, 67% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.77 (d, 1H, J = 8 Hz, ArH), 7.72 (d, 1H, J = 8 Hz, ArH), 7.56–7.52 (m, 1H, ArH), 7.28–7.24 (m, 1H, ArH), 6.80 (s, 1H, ArH), 3.85–3.83 (m, 4H, $2CH_2$), 3.70–3.68 (m, 4H, $2CH_2$), 2.60 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.4, 145.3, 129.4, 127.2, 123.5, 122.5, 109.6, 100.0, 66.9, 45.6, 19.3.



9k

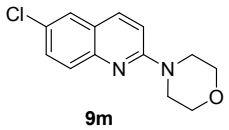
The compound was prepared according to general procedure 3.3 and purified by flash column chromatography (5:1 petroleum ether: ethyl acetate) to afford **9k** (yellow oil, 35 mg, 78%

yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.85 (d, 1H, *J* = 8 Hz, ArH), 7.65 (d, 1H, *J* = 8 Hz, ArH), 7.42–7.40 (m, 2H, ArH), 6.93 (d, 1H, *J* = 8 Hz, ArH), 3.87–3.85 (m, 4H, $2CH_2$), 3.70–3.68 (m, 4H, $2CH_2$), 2.47 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.2, 145.9, 137.1, 132.2, 131.7, 126.4, 123.3, 109.3, 66.9, 45.8, 21.2; HRMS (ESI–Q–TOF) exact mass calcd for $C_{14}H_{17}N_2O$ $[M + H]^+$229.1341, found 229.1319.



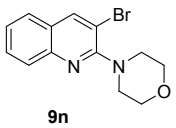
9l

The compound was prepared according to general procedure 3.3 and purified by flash column chromatography (5:1 petroleum ether: ethyl acetate) to afford **9l** (yellow oil, 33 mg, 68% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.85 (d, 1H, *J* = 8 Hz, ArH), 7.66 (d, 1H, *J* = 8 Hz, ArH), 7.25–7.22 (m, 1H, ArH), 6.97–6.93 (m, 2H, ArH), 3.88 (s, 3H, OCH_3), 3.87–3.84 (m, 4H, $2CH_2$), 3.66–3.63 (m, 4H, $2CH_2$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 156.6, 155.3, 143.3, 136.6, 128.2, 123.8, 121.3, 109.8, 106.0, 66.9, 55.5, 46.0; HRMS (ESI–Q–TOF) exact mass calcd for $C_{14}H_{17}N_2O_2$ $[M + H]^+$ 245.1290, found 245.1270.



9m

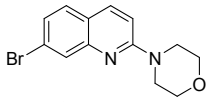
The compound was prepared according to general procedure 3.3 and purified by flash column chromatography (5:1 petroleum ether: ethyl acetate) to afford **9m** (yellow oil, 27 mg, 55% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.81 (d, 1H, *J* = 8 Hz, ArH), 7.63 (d, 1H, *J* = 8 Hz, ArH), 7.57 (s, 1H, ArH), 7.47 (d, 1H, *J* = 8 Hz, ArH), 6.96 (d, 1H, *J* = 8 Hz, ArH), 3.86–3.83 (m, 4H, $2CH_2$), 3.71–3.69 (m, 4H, $2CH_2$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.5, 146.2, 136.6, 130.2, 128.3, 127.7, 126.0, 123.8, 110.1, 66.8, 45.5; HRMS (ESI–Q–TOF) exact mass calcd for $C_{13}H_{14}ClN_2O$ $[M + H]^+$ 249.0795, found 249.0777.



9n

The compound was prepared according to general procedure 3.3 and purified by flash column chromatography (5:1 petroleum ether: ethyl acetate) to afford **9n** (yellow oil, 40 mg, 68% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.28 (s, 1H, ArH), 7.85 (d, 1H, *J* = 8 Hz, ArH),

7.65–7.61 (m, 2H, ArH), 7.42–7.38 (m, 1H, ArH), 3.93–3.91 (m, 4H, $2CH_2$), 3.50–3.48 (m, 4H, $2CH_2$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.3, 145.8, 141.6, 129.8, 127.7, 126.4, 125.1, 112.3, 66.9, 50.3; HRMS (ESI–Q–TOF) exact mass calcd for $C_{13}H_{14}BrN_2O$ $[M + H]^+$ 293.0290, found 293.0303.

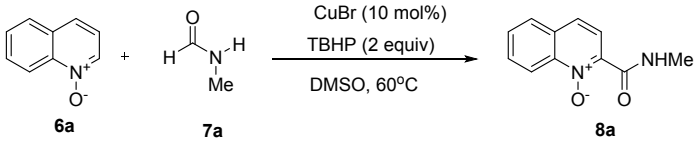


9o

The compound was prepared according to general procedure 3.3 and purified by flash column chromatography (5:1 petroleum ether: ethyl acetate) to afford **9o** (yellow oil, 33 mg, 57% yield): ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.90 (s, 1H, ArH), 7.86 (d, 1H, J = 8 Hz, ArH), 7.45 (d, 1H, J = 8 Hz, ArH), 7.32 (d, 1H, J = 8 Hz, ArH), 7.95 (d, 1H, J = 8 Hz, ArH), 3.86–3.83 (m, 4H, $2CH_2$), 3.73–3.70 (m, 4H, $2CH_2$); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.7, 148.5, 137.4, 129.0, 128.4, 125.9, 123.7, 121.8, 109.4, 66.8, 45.4; HRMS (ESI–Q–TOF) exact mass calcd for $C_{13}H_{14}BrN_2O$ $[M + H]^+$ 293.0290, found 293.0287.

4. Synthetic Utility of Methodology

4.1 Gram-scale reaction

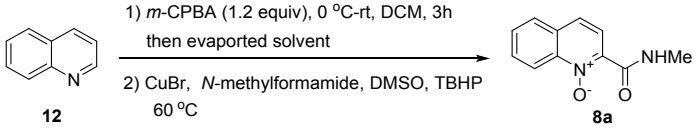


6a **7a** **8a**

Gram-scale reaction: 10 mmol scale, 85% (**8a**: 1.7 g)

Quinoline *N*-oxides **6a** (10 mmol, 1.45 g) was added in a 50 mL dried round-bottomed flask, followed by the addition of *N*-methylformylamide **7a** (10 equiv), CuBr (10 mol %), TBHP (5.0-6.0 M in decane) (2 equiv) and DMSO (20 mL). Then the mixture was stirred at 60 °C until complete consumption of starting material as monitored by TLC and GC-MS analysis. After the reaction was finished, the reaction mixture was washed with brine. The aqueous phase was re-extracted with ethyl acetate. The combined organic extracts were dried over Na_2SO_4, concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography to afford the desired quinoline-*N*-oxide-2-carboxamides **8a** (1.7 g, 85% yield).

4.2 Sequential one-pot process

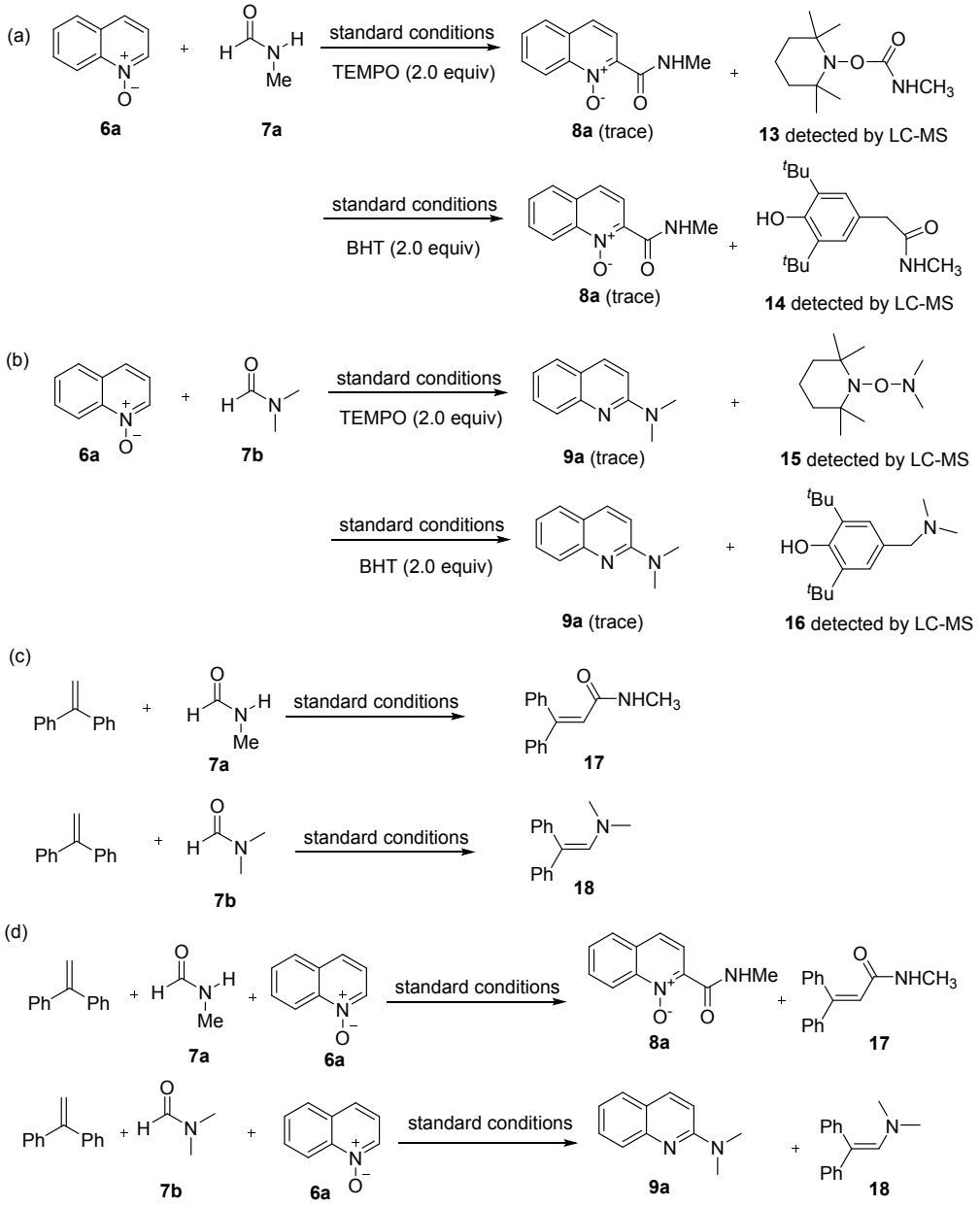


12 **8a**

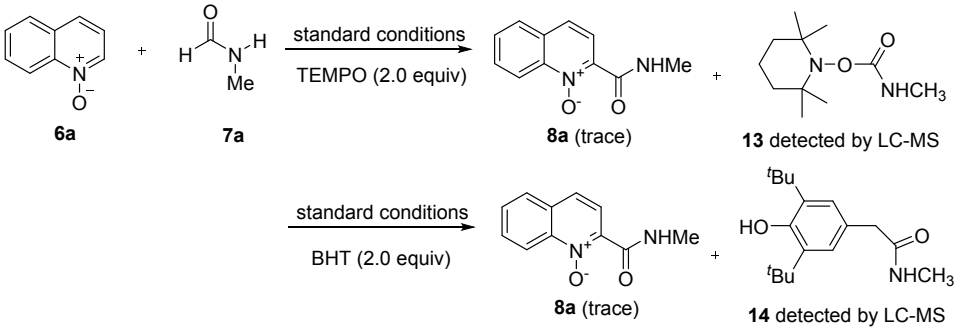
To a solution of quinoline **12** (10 mmol) in CH_2Cl_2 (40 mL) at 0 °C was added *m*-CPBA (12 mmol, 1.2 equiv). The mixture was stirred at 0 °C for 1 hour and then continued to stir at room temperature

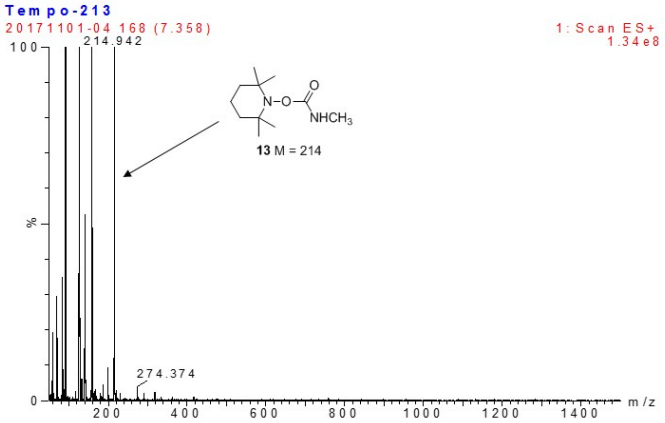
till the full conversion of quinoline as detected by TLC. Solvent was removed under reduced pressure. To the residue was added *N*-methylformylamide **7a** (10 equiv), CuBr (10 mol %), TBHP (5.0-6.0 M in decane) (2 equiv) and DMSO (20 mL). The mixture was stirred at 60 °C until complete consumption of starting material as monitored by TLC and GC-MS analysis. After the reaction was finished, the reaction mixture was washed with brine. The aqueous phase was re-extracted with ethyl acetate. The combined organic extracts were dried over Na_2SO_4, concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography to afford the desired quinoline-*N*-oxide-2-carboxamides **8a** (1.7g, 85% yield).

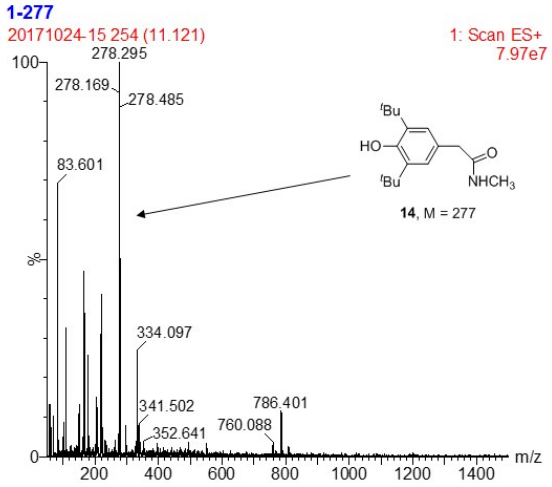
5. Mechanistic Study



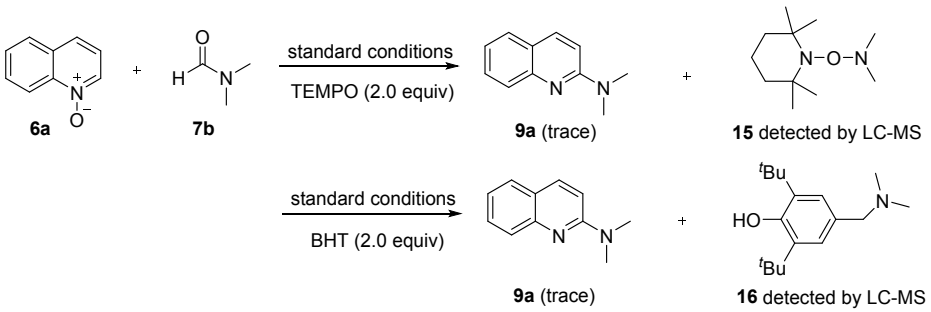
5.1 Addition of TEMPO and BHT to the Model Reaction of **6a** and **7a**

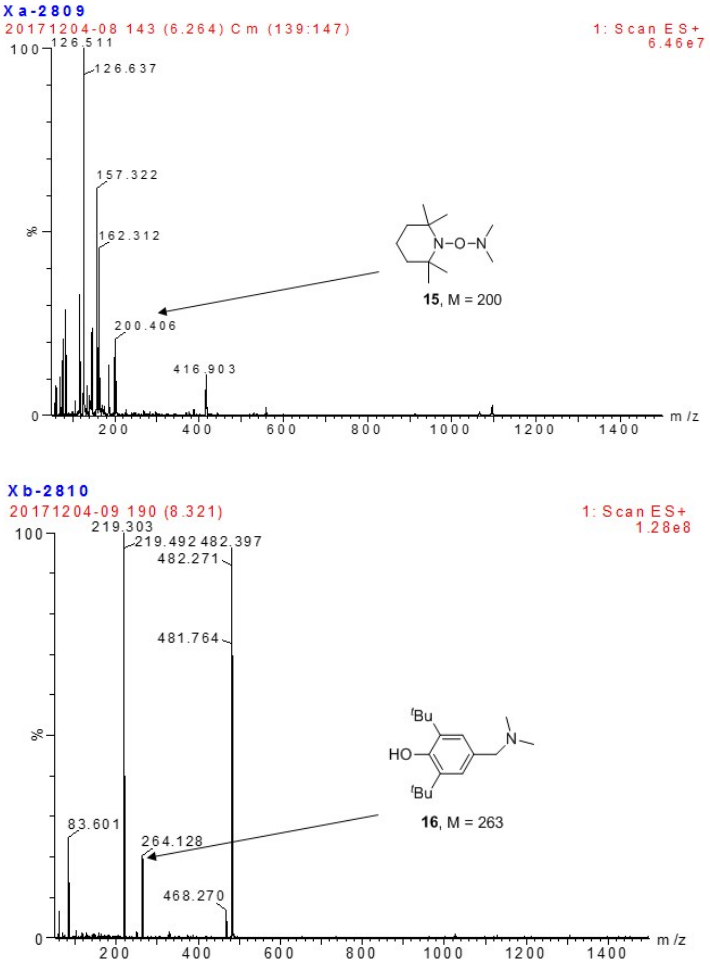




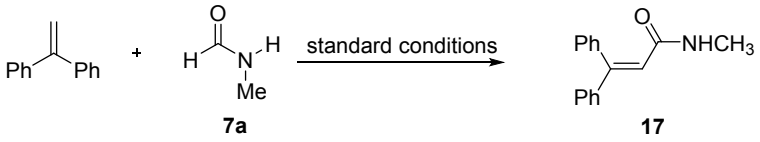


5.2 Addition of TEMPO and BHT to the Model Reaction of **6a** and **7b**

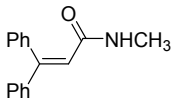




5.3 Reaction of *N*-methyl formamide **7a** with 1,1-diphenylethylene under standard conditions

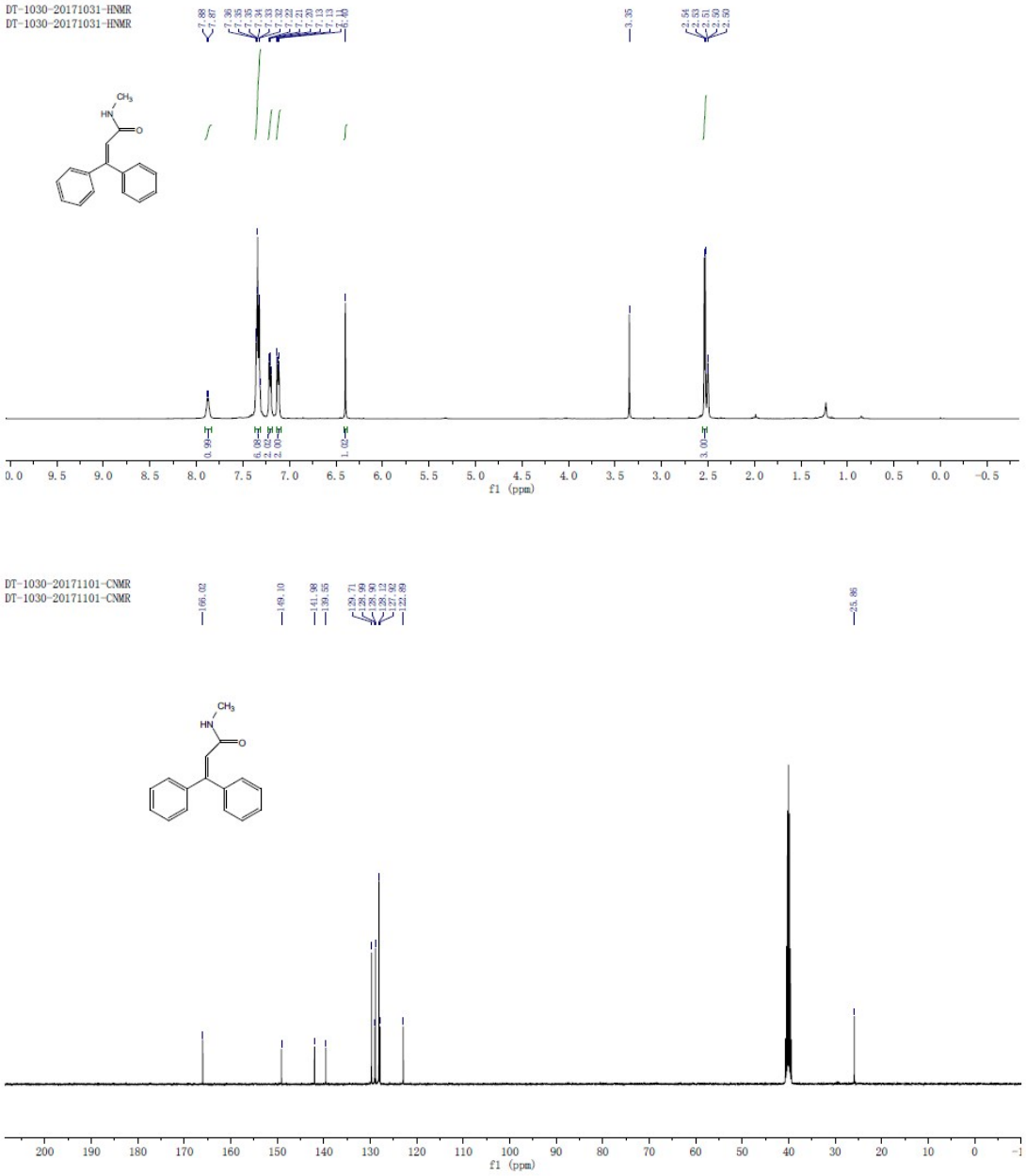


7a **17**

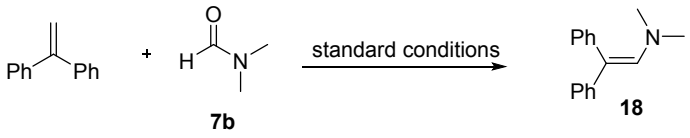


17

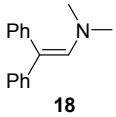
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.88 (d, 1H, *J* = 4 Hz, NH), 7.36-7.32 (m, 6H, ArH), 7.22-7.20 (m, 2H, ArH), 7.13-7.10 (m, 2H, ArH), 6.40 (s, 1H, CH), 2.54 (d, 3H, *J* = 4 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 166.0, 149.1, 142.0, 139.6, 129.7, 129.0, 128.9, 128.1, 127.9, 122.9, 25.9.



5.4 Reaction of DMF **7b** with 1,1-diphenylethylene under standard conditions

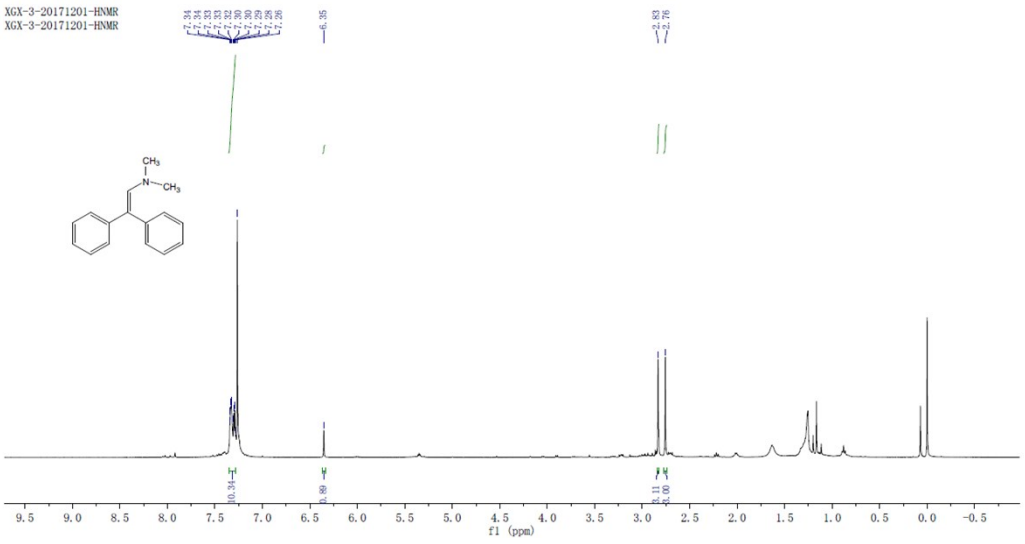


7b **18**

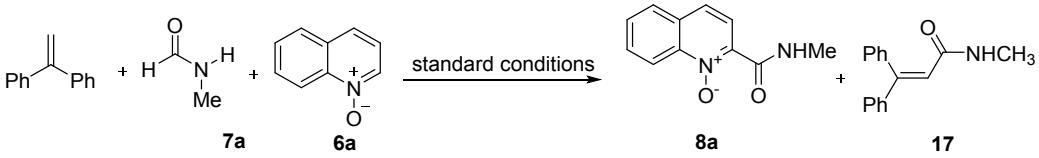


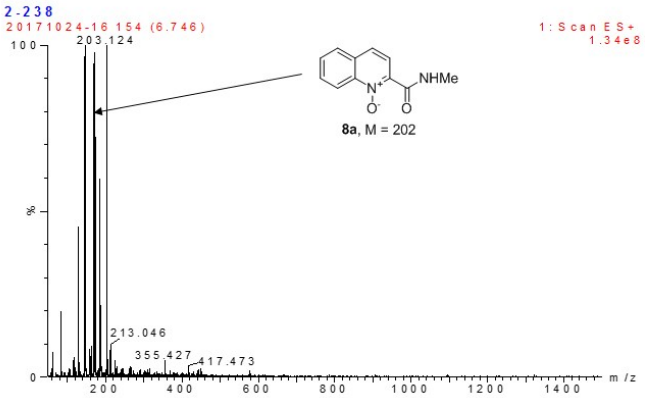
18

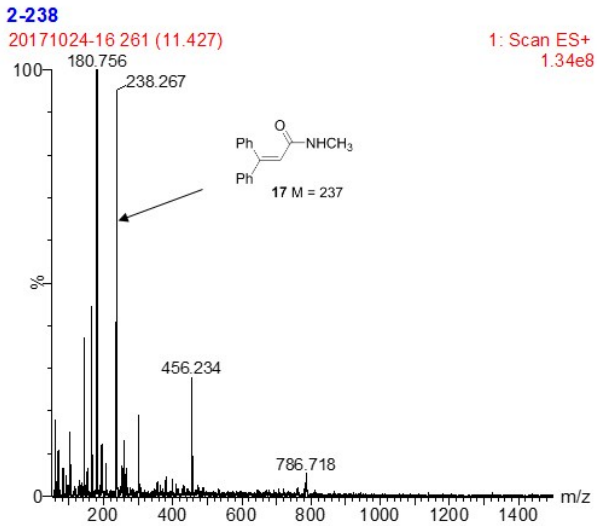
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.34-7.26 (m, 10H, ArH), 6.35 (s, 1H, CH), 2.83 (s, 3H, CH_3), 2.76 (s, 3H, CH_3).



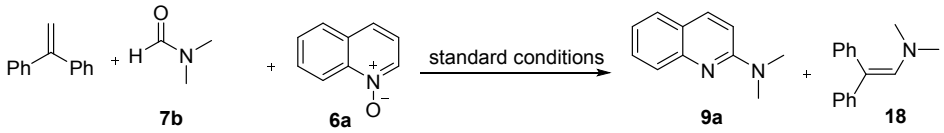
5.5 Reaction of quinoline-*N*-oxide **6a**, *N*-methyl formamide **7a** and 1,1-diphenylethylene under standard conditions

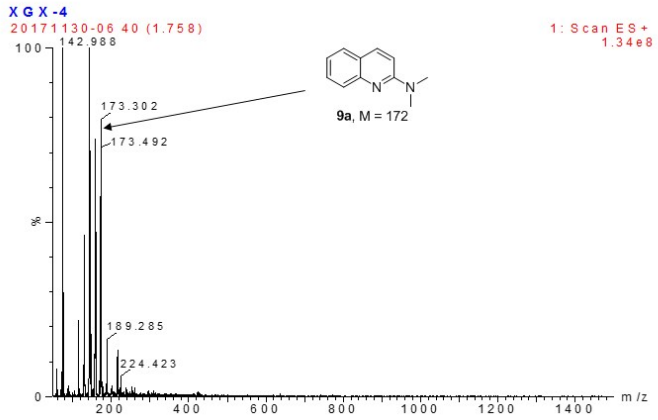


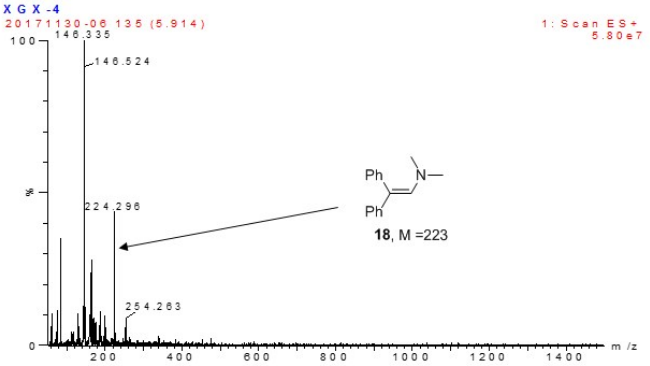




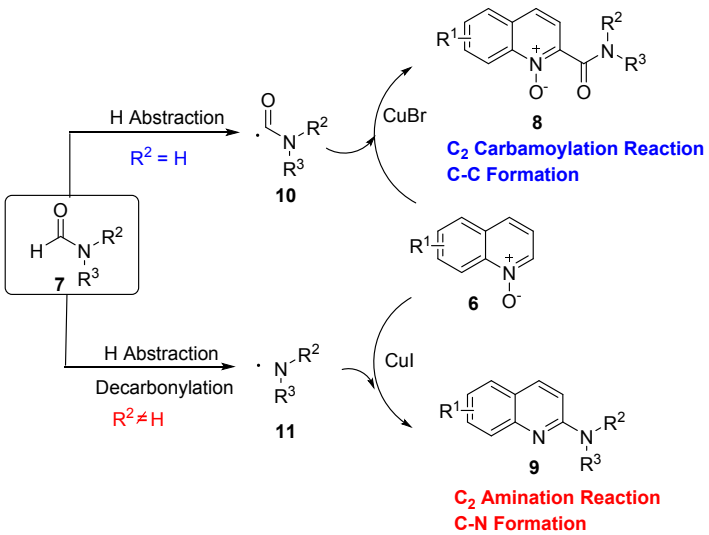
5.6 Reaction of quinoline-*N*-oxide **6a**, DMF **7b** and 1,1-diphenylethylene under standard conditions





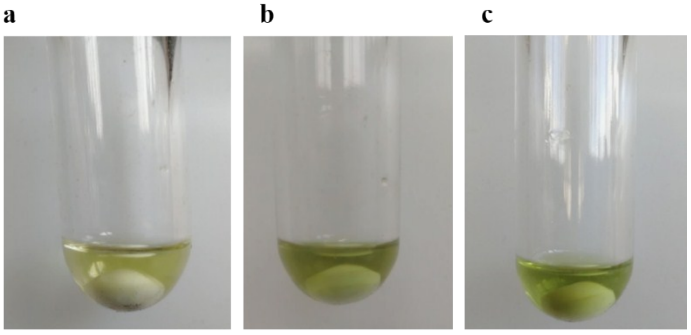


C_2 Selective Carbamoylation and Amination of Quinoline *N*-Oxide with Formamide

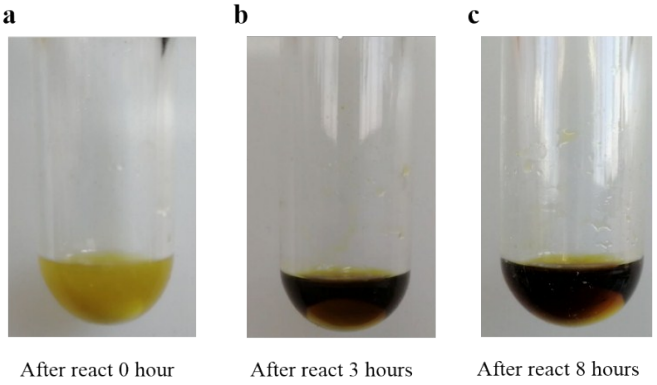


5.7 Reaction color changes for C_2 selective carbamoylation and amination of quinoline *N*-oxide with formamide

carbamoylation of quinoline *N*-oxide



amination of quinoline *N*-oxide



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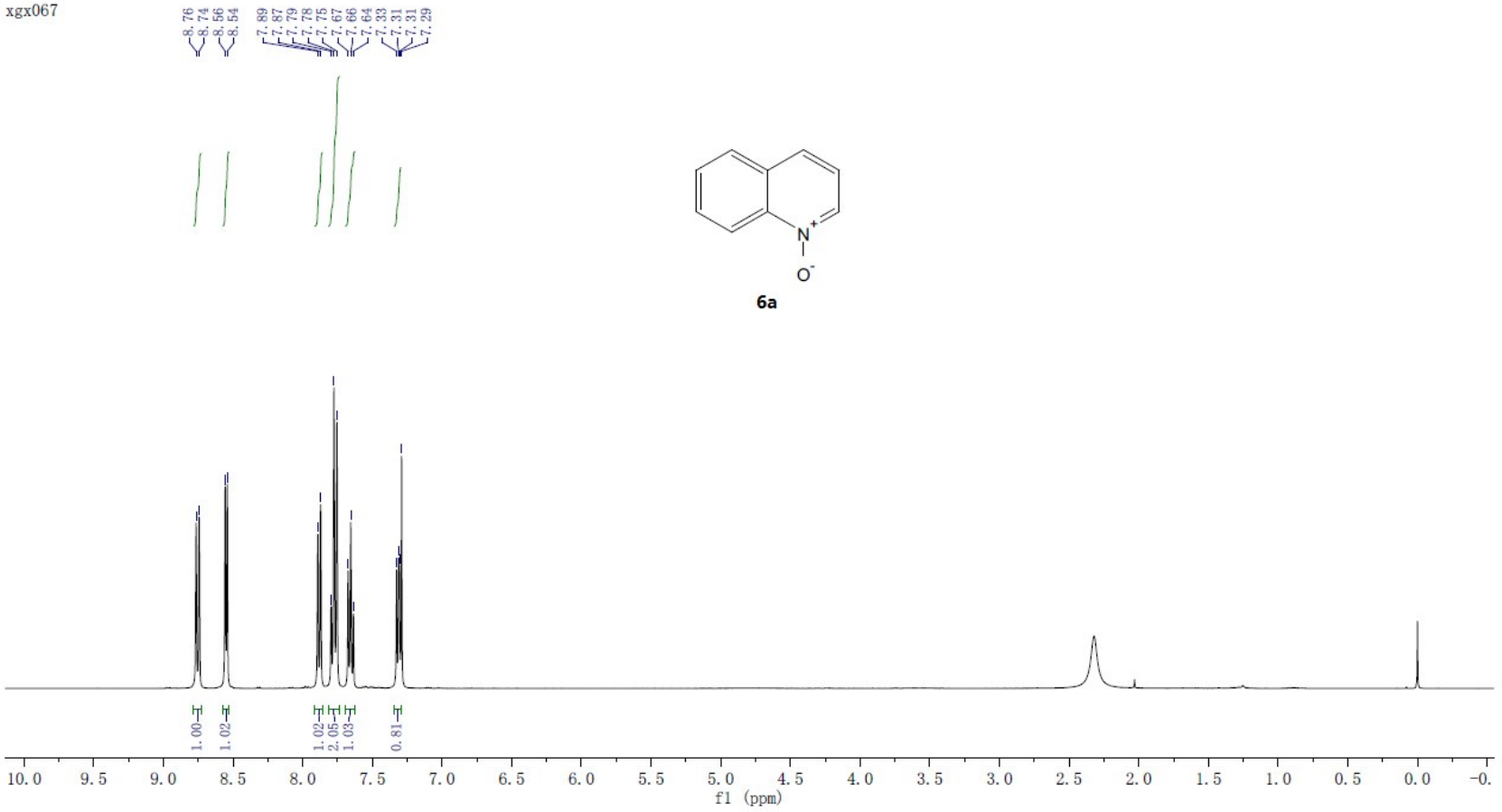
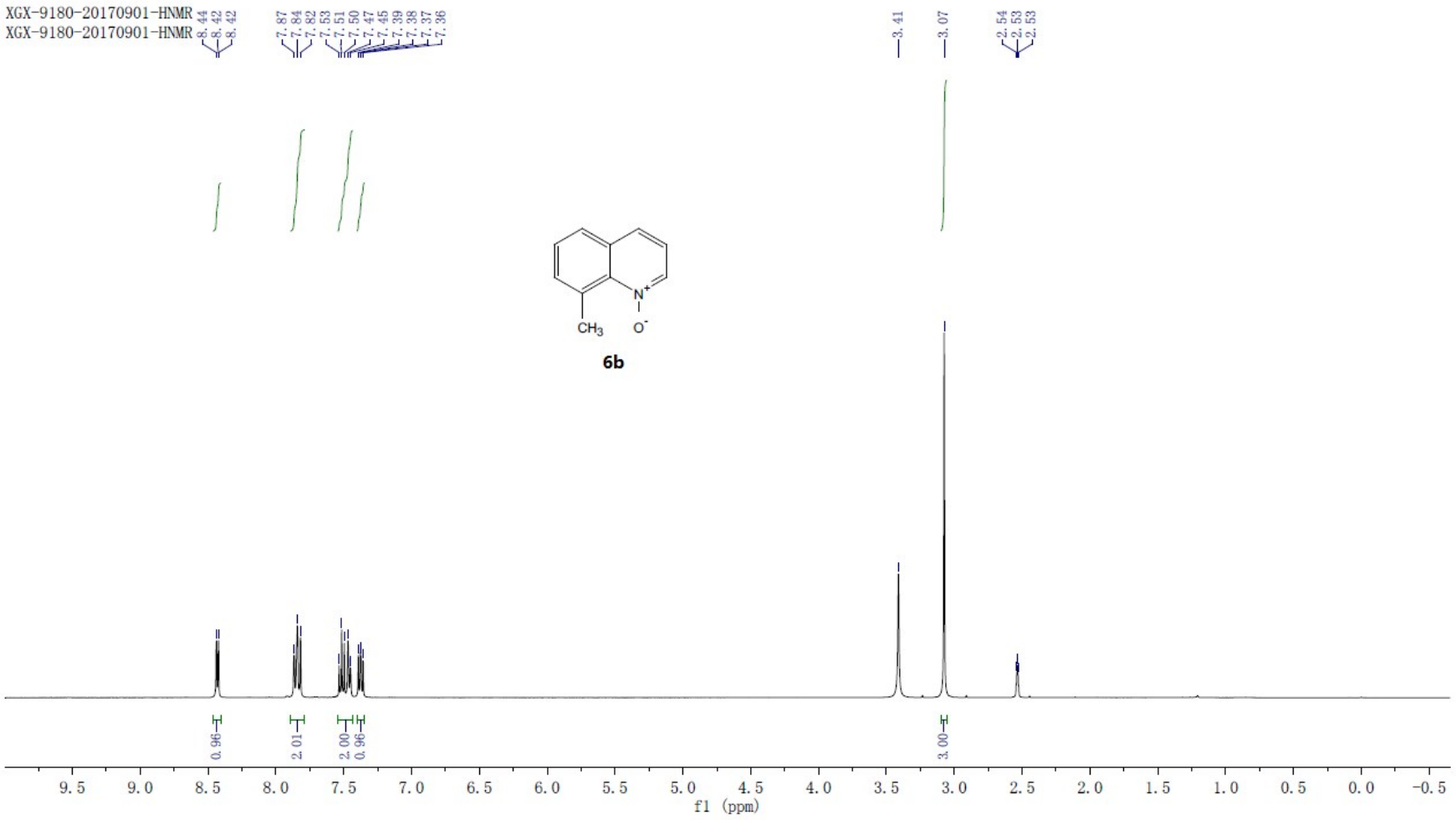
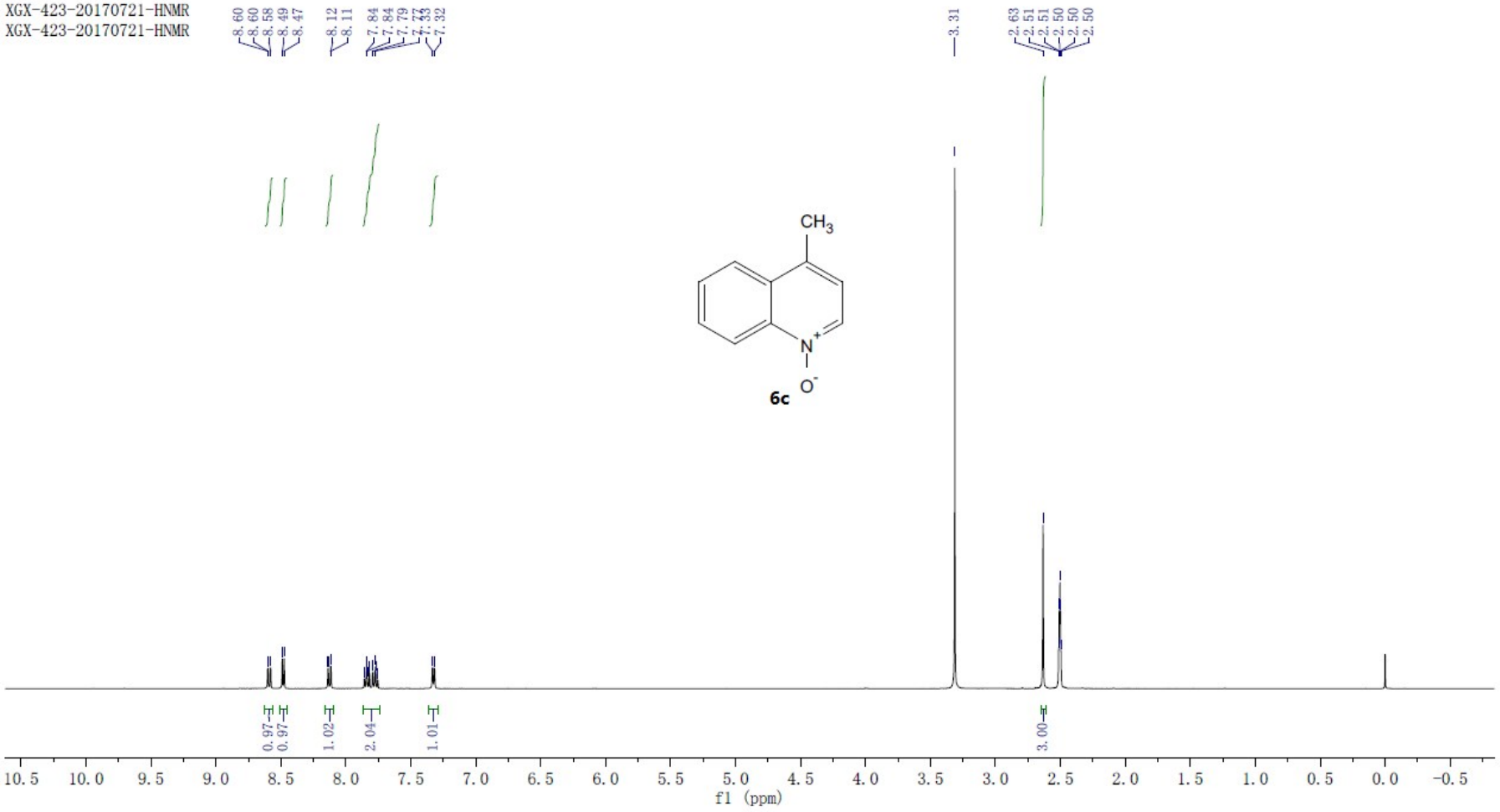
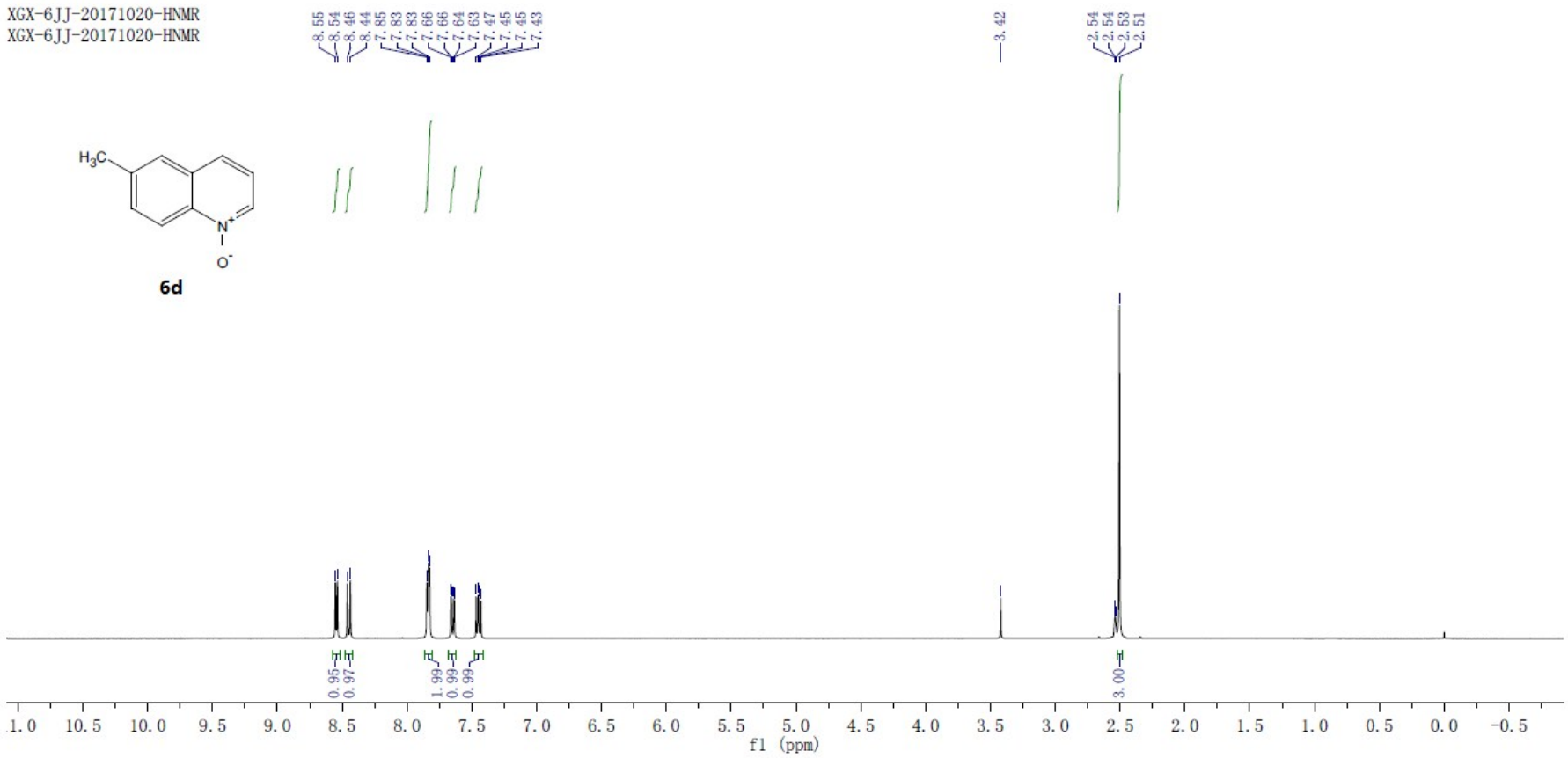
7. ^{1}H and ^{13}C NMR Spectra of Substrates and Products

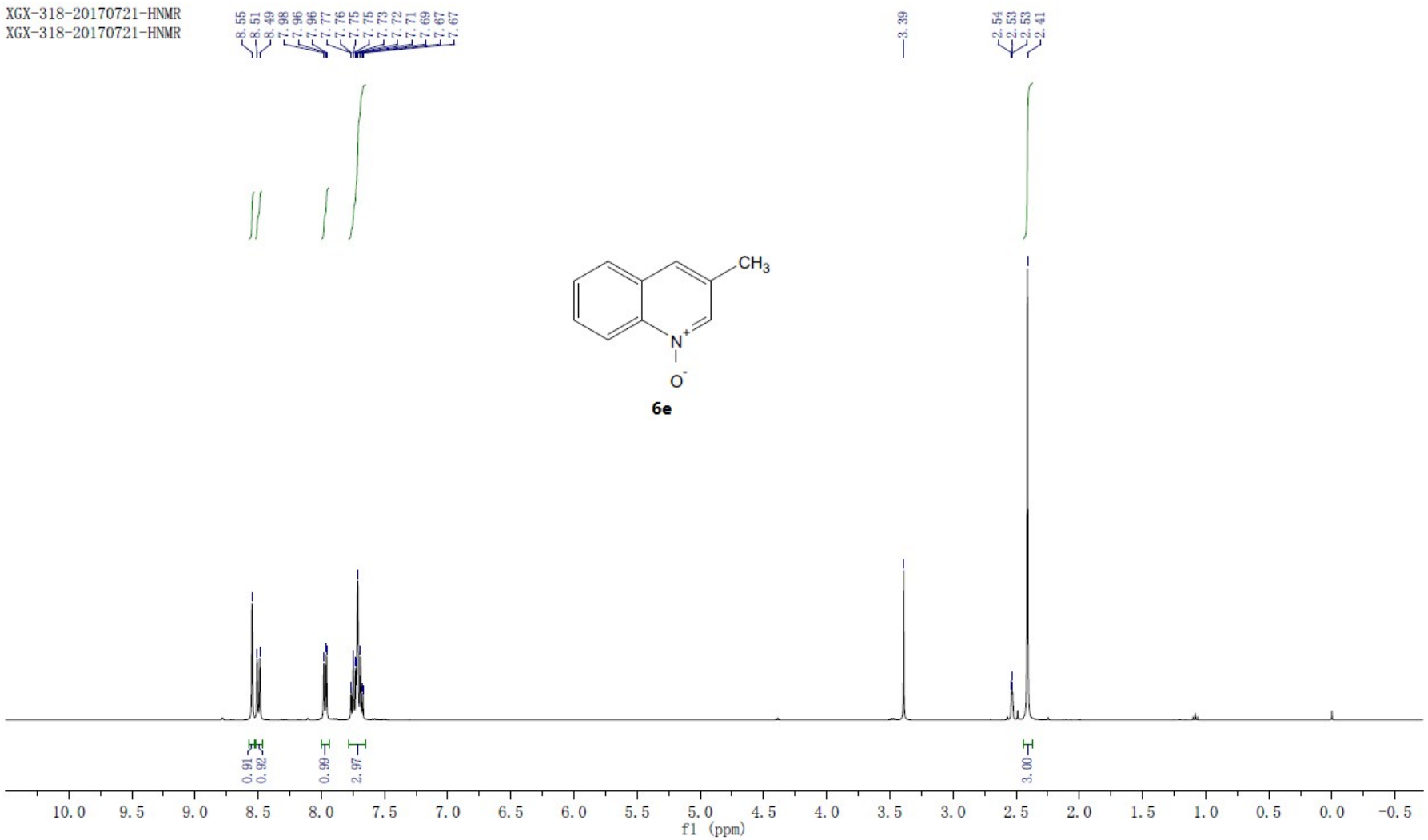
7.1 ^{1}H NMR Spectra of Substrates

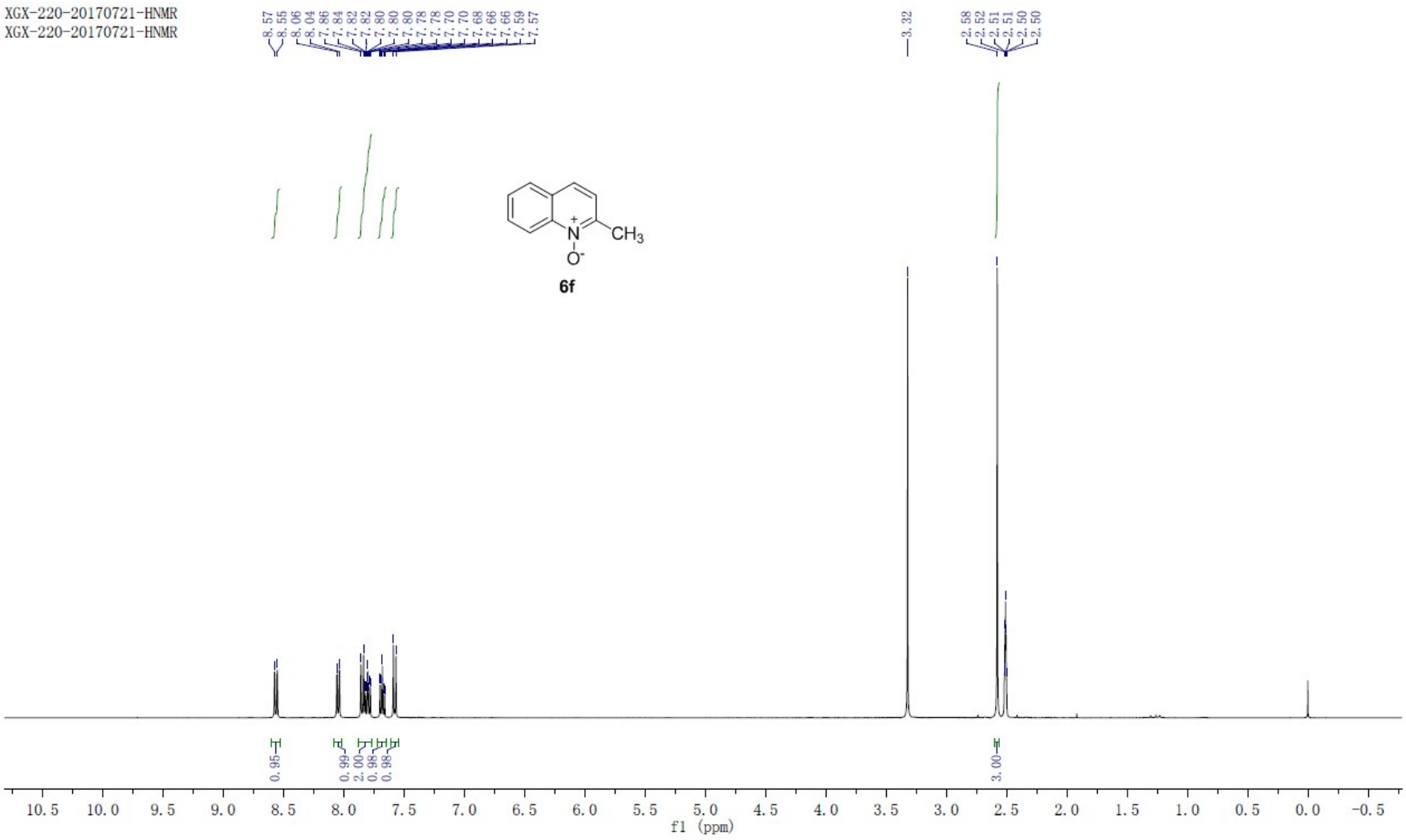


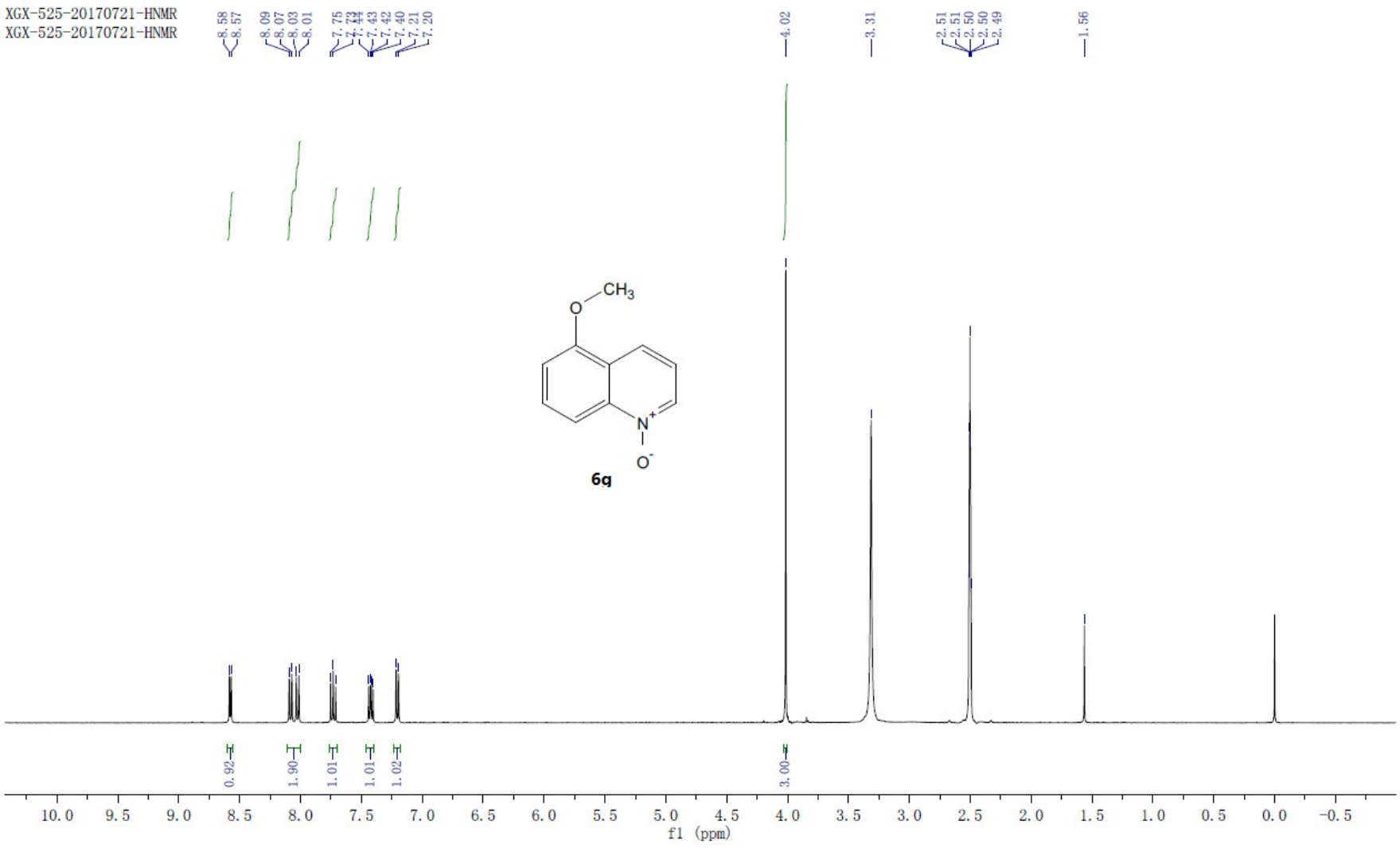

XGX-9180-20170901-HNMR
XGX-9180-20170901-HNMR
8.44
8.42
8.42
7.87
7.84
7.82
7.53
7.51
7.50
7.47
7.45
7.39
7.38
7.37
7.36
3.41
3.07
2.54
2.53
2.53
CH3
O-
N+
6b
0.96
2.01
2.00
0.96
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

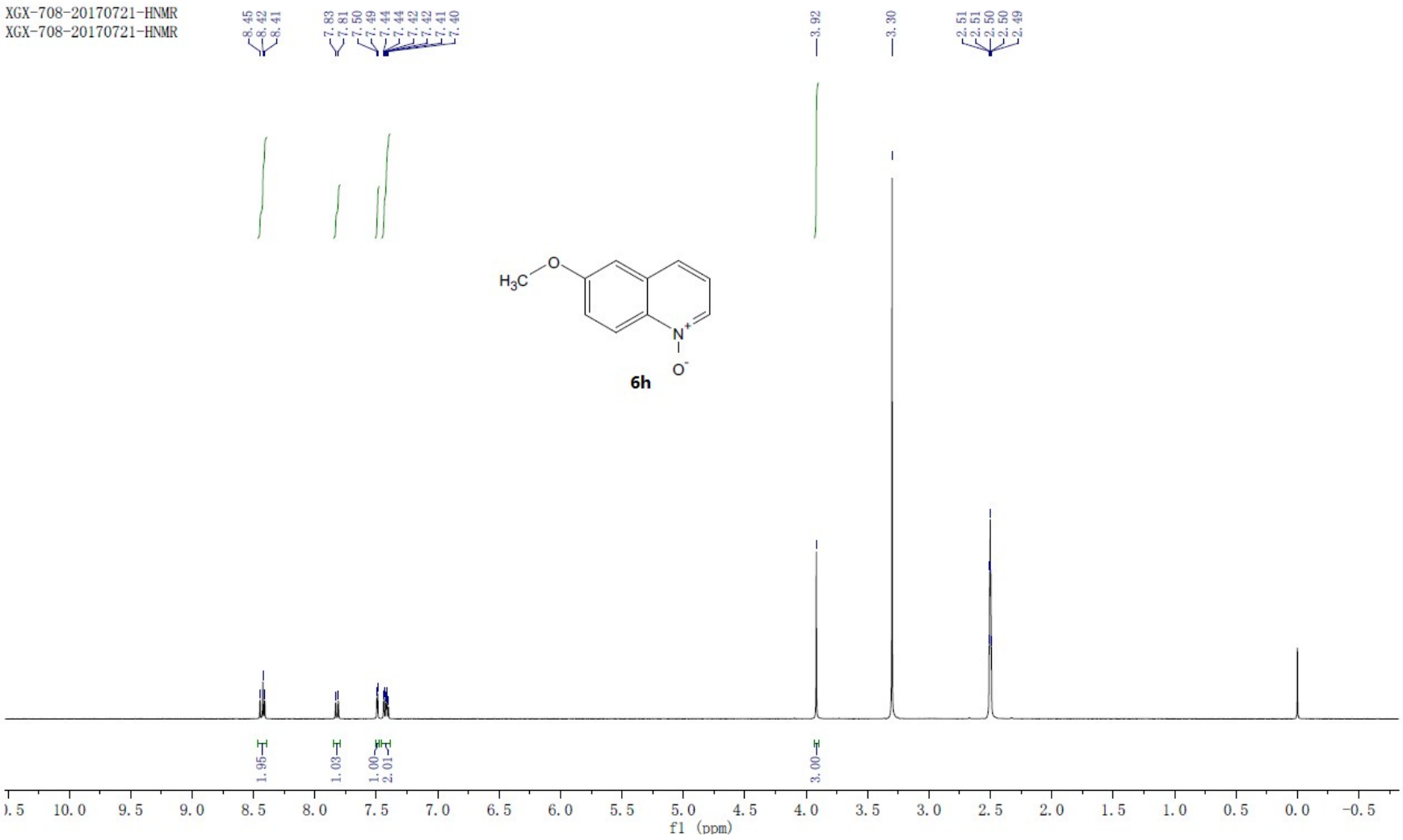

XGX-423-20170721-HNMR
XGX-423-20170721-HNMR
8.60
8.60
8.58
8.49
8.47
8.12
8.11
7.84
7.84
7.79
7.77
7.33
7.32
3.31
2.63
2.51
2.51
2.50
2.50
2.50
CH3
N+
O-
6c
0.97
0.97
1.02
2.04
1.01
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

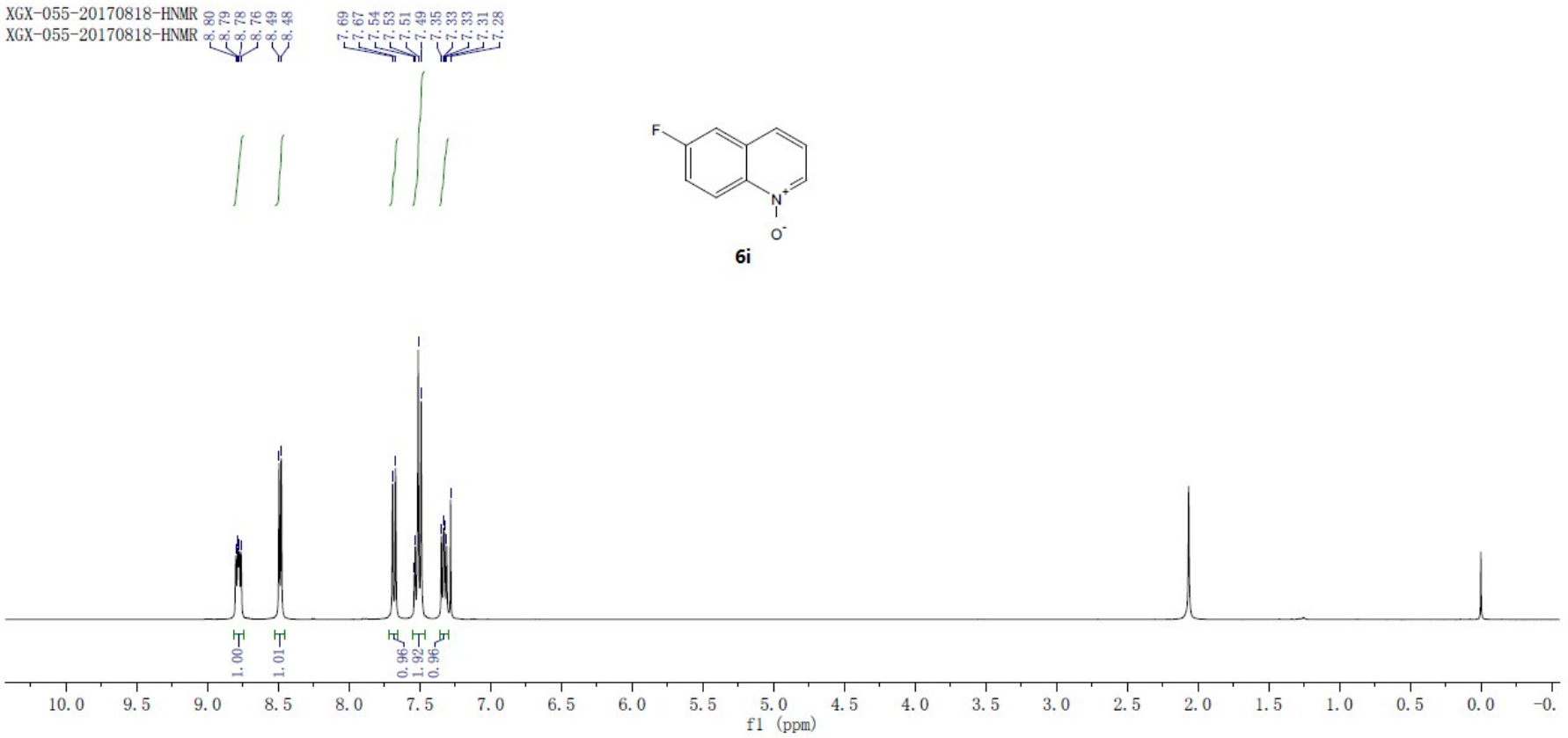

XGX-6JJ-20171020-HNMR
XGX-6JJ-20171020-HNMR
8.55
8.54
8.46
8.44
7.85
7.83
7.83
7.66
7.66
7.64
7.63
7.47
7.45
7.45
7.43
3.42
2.54
2.54
2.53
2.51
H3C
N+
O-
6d
0.95
0.97
1.99
0.99
0.99
3.00
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

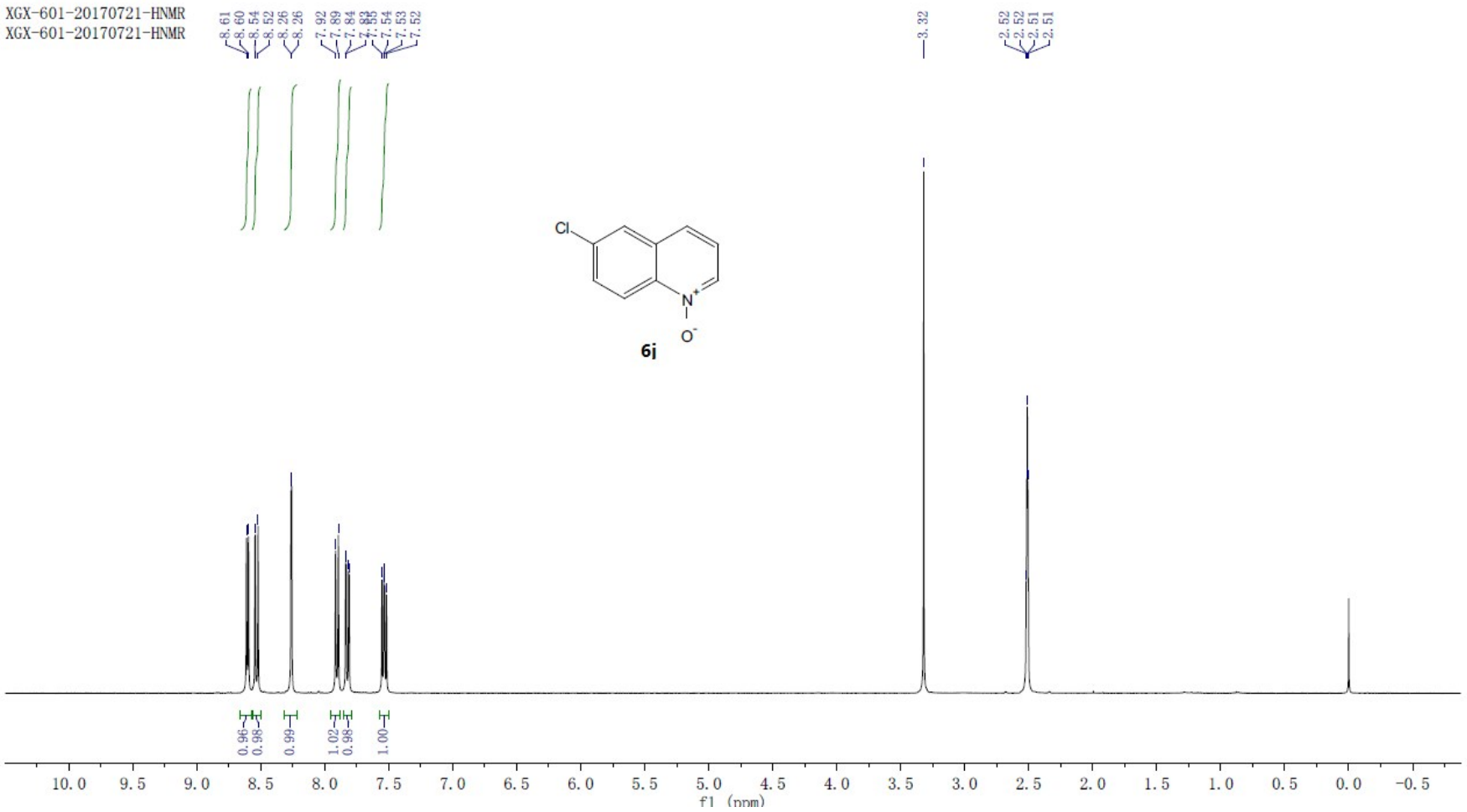

XGX-318-20170721-HNMR
XGX-318-20170721-HNMR
8.55
8.51
8.49
7.98
7.96
7.96
7.77
7.76
7.75
7.75
7.73
7.72
7.71
7.69
7.67
7.67
3.39
2.54
2.53
2.53
2.41
CH3
N+
O-
6e
0.91
0.92
0.99
2.97
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

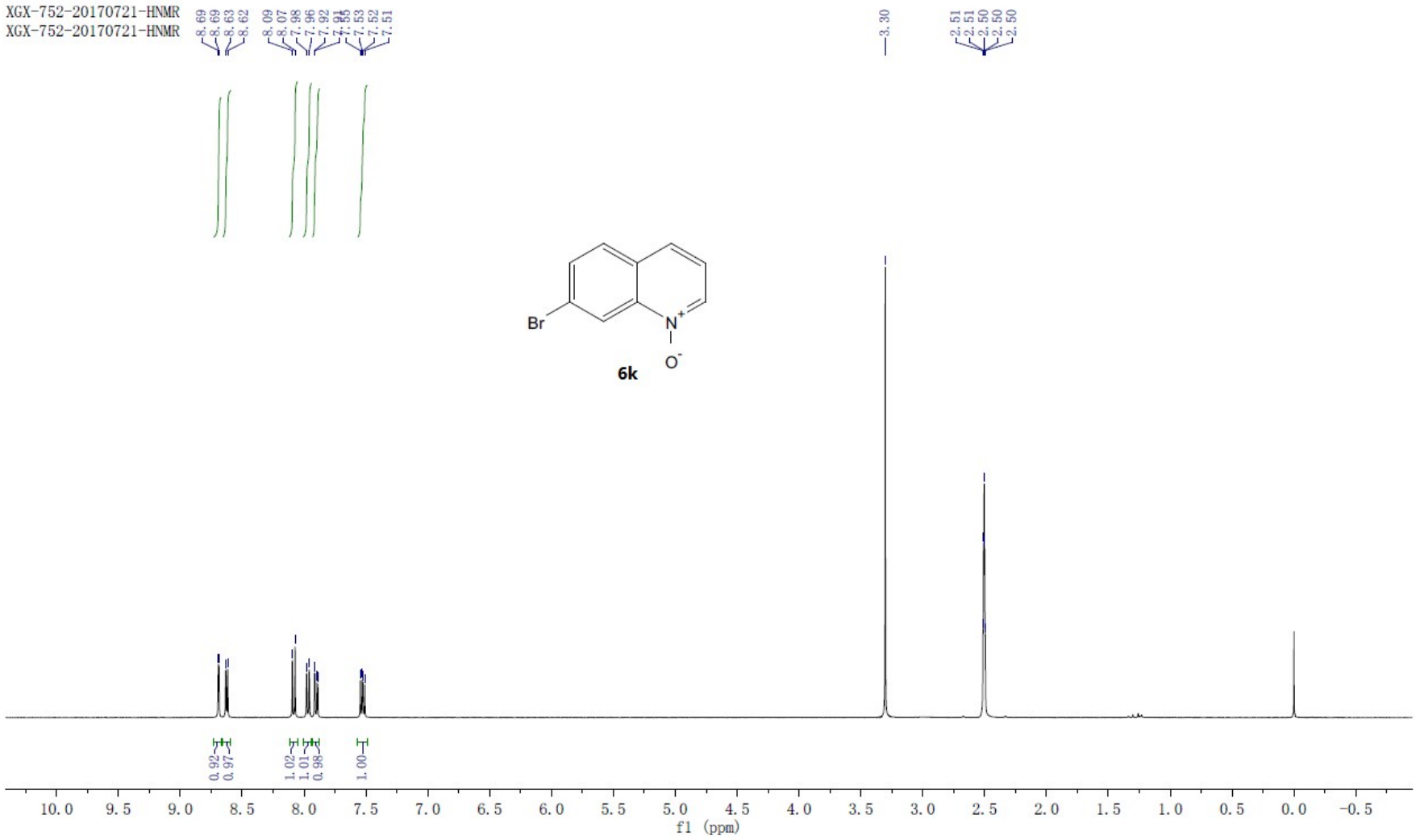

XGX-220-20170721-HNMR
XGX-220-20170721-HNMR
8.57
8.55
8.06
8.04
7.86
7.84
7.82
7.82
7.80
7.80
7.80
7.78
7.78
7.70
7.70
7.68
7.66
7.66
7.59
7.57
3.32
2.58
2.52
2.51
2.51
2.50
2.50
CH3
6f
0.95
0.99
2.00
0.98
0.98
3.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

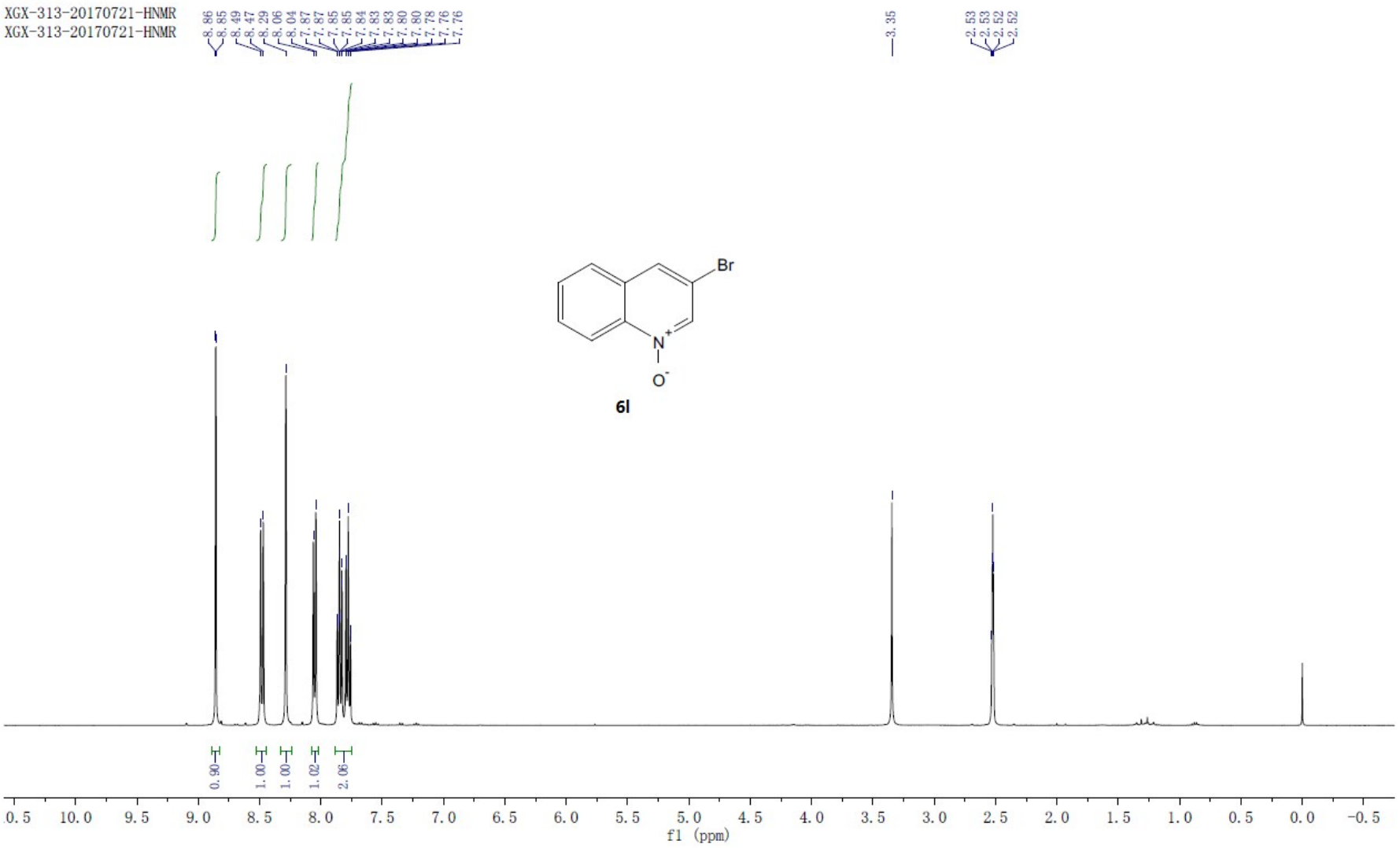

XGX-525-20170721-HNMR
XGX-525-20170721-HNMR
8.58
8.57
8.09
8.07
8.03
8.01
7.75
7.43
7.42
7.40
7.21
7.20
4.02
3.31
2.51
2.51
2.50
2.50
2.49
1.56
CH3
O
N+
O-
6q
0.92
1.90
1.01
1.01
1.02
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

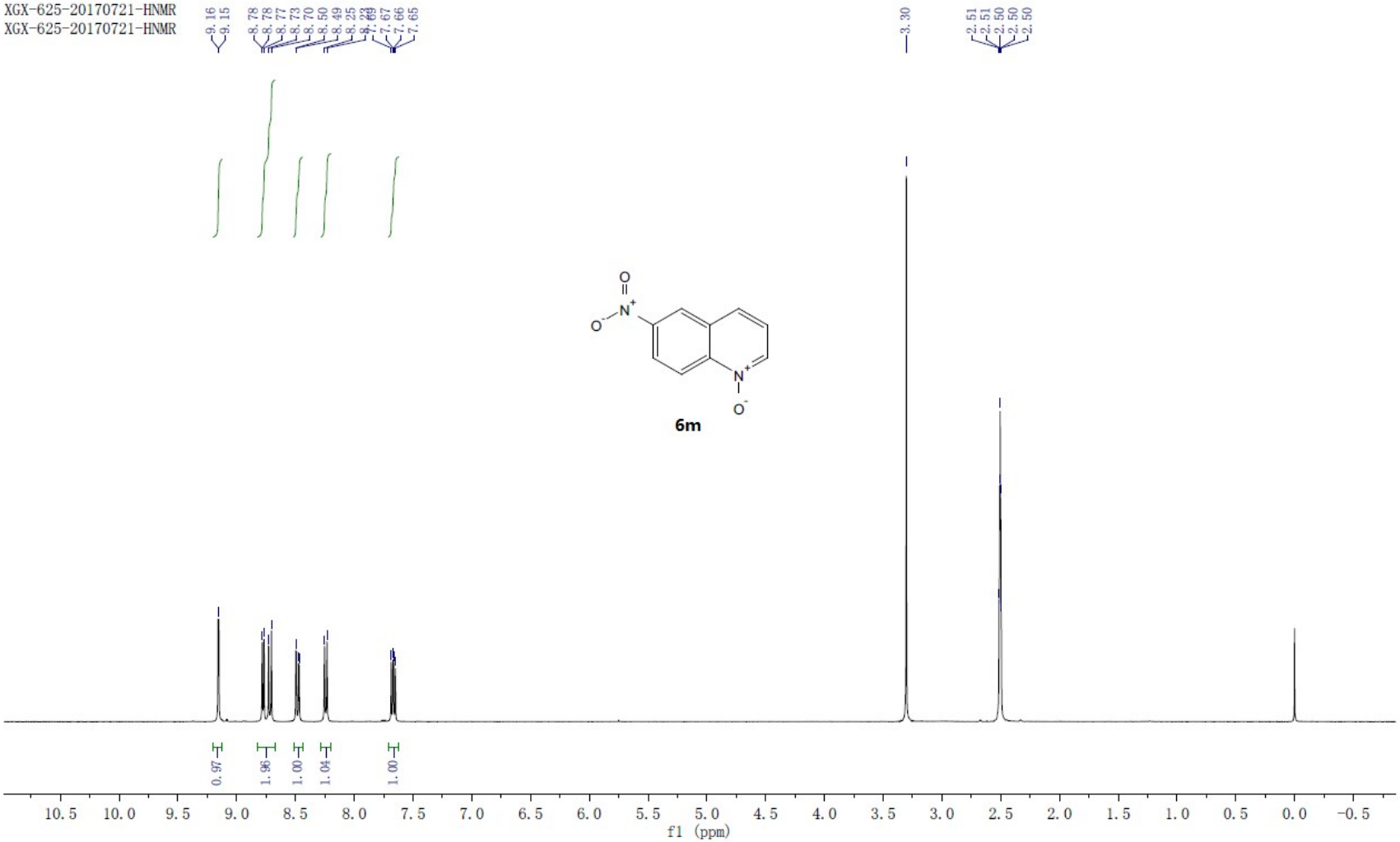

XGX-708-20170721-HNMR
XGX-708-20170721-HNMR
8.45
8.42
8.41
7.83
7.81
7.50
7.49
7.44
7.44
7.42
7.42
7.41
7.40
3.92
3.30
2.51
2.51
2.50
2.50
2.49
H3C
O
N+
O-
6h
1.95
1.03
1.00
2.01
3.00
f1 (ppm)

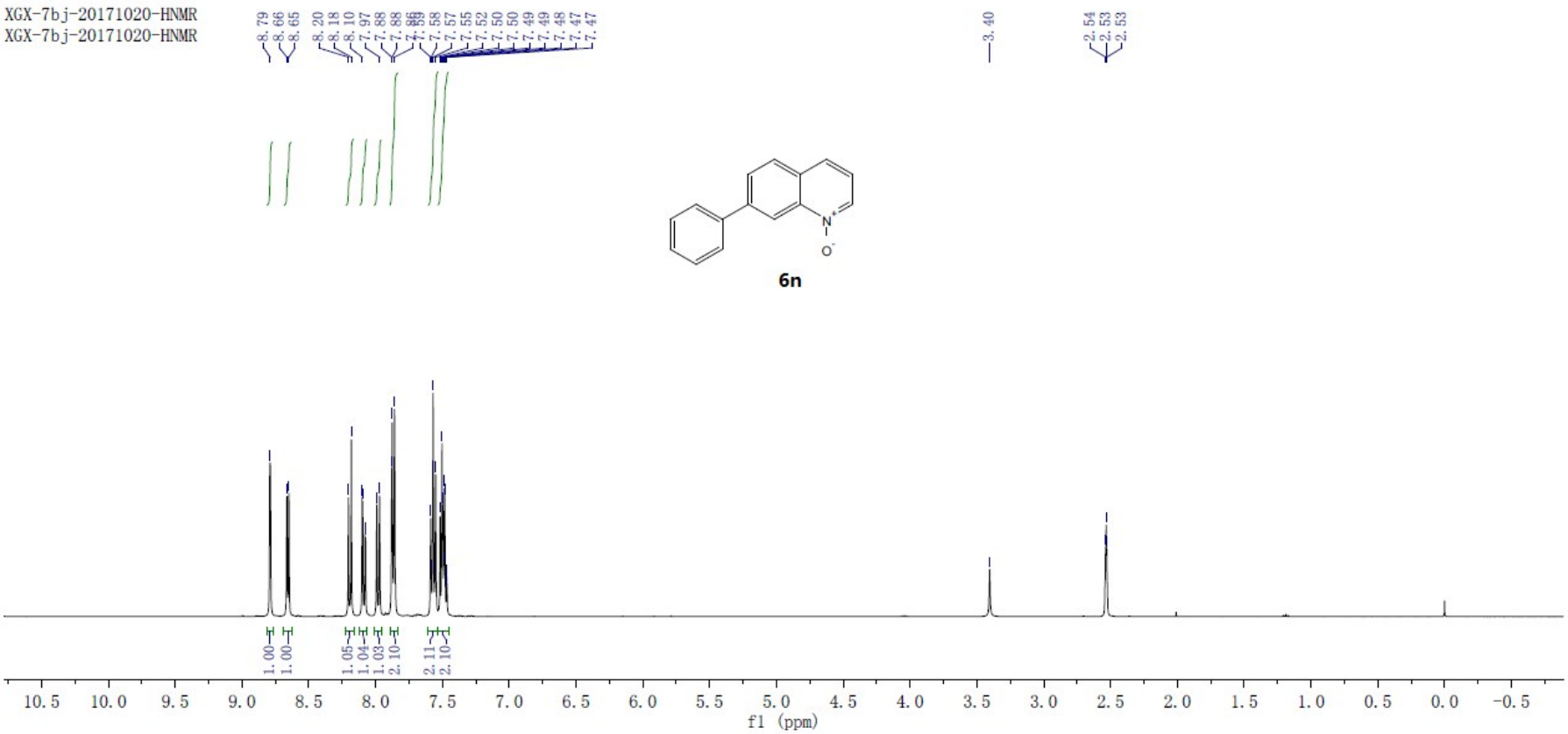

XGX-055-20170818-HNMR
XGX-055-20170818-HNMR
8.80
8.79
8.78
8.76
8.49
8.48
7.69
7.67
7.54
7.53
7.51
7.49
7.35
7.33
7.31
7.28
F
N+
O-
6i
1.00
1.01
0.96
1.92
0.96
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)


XGX-601-20170721-HNMR
XGX-601-20170721-HNMR
8.61
8.60
8.54
8.52
8.26
8.26
7.92
7.89
7.84
7.83
7.55
7.54
7.53
7.52
3.32
2.52
2.52
2.51
2.51
Cl
N+
O-
6j
0.96
0.98
0.99
1.02
0.98
1.00
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)


XGX-752-20170721-HNMR
XGX-752-20170721-HNMR
8.69
8.69
8.63
8.62
8.09
8.07
7.98
7.96
7.92
7.53
7.52
7.51
3.30
2.51
2.51
2.50
2.50
2.50
Br
N+
O-
6k
0.92
0.97
1.02
1.01
0.98
1.00
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)


XGX-313-20170721-HNMR
XGX-313-20170721-HNMR
8.86
8.85
8.49
8.47
8.29
8.06
8.04
7.87
7.87
7.85
7.85
7.84
7.83
7.83
7.80
7.80
7.78
7.76
7.76
3.35
2.53
2.53
2.52
2.52
Br
N+
O-
6l
0.90
1.00
1.00
1.02
2.06
f1 (ppm)


XGX-625-20170721-HNMR
XGX-625-20170721-HNMR
9.16
9.15
8.78
8.78
8.77
8.73
8.70
8.50
8.49
8.25
7.67
7.66
7.65
3.30
2.51
2.51
2.50
2.50
2.50
6m
0.97
1.96
1.00
1.04
1.00
f1 (ppm)


XGX-7bj-20171020-HNMR
XGX-7bj-20171020-HNMR
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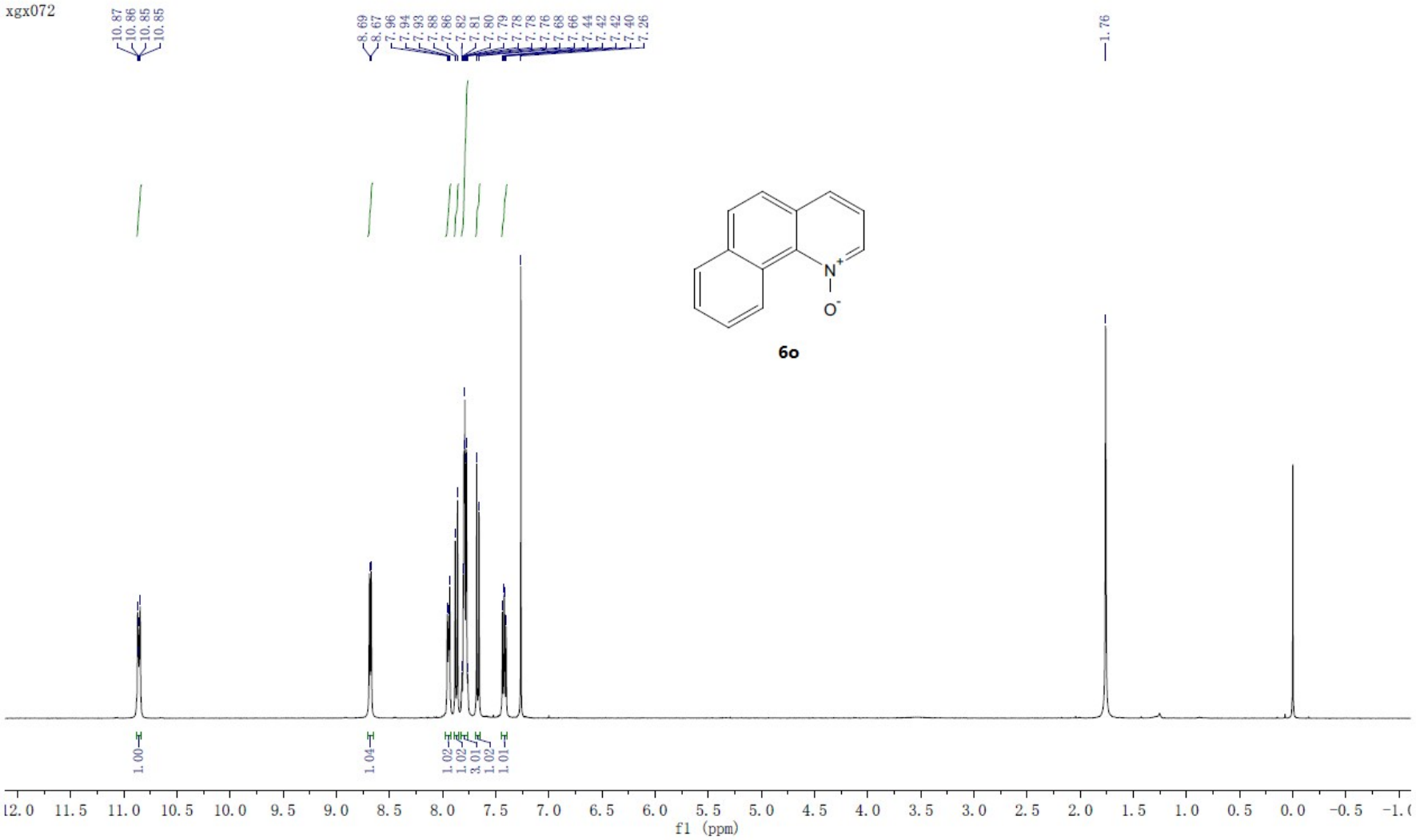
8.66
8.65
8.20
8.18
8.10
7.97
7.88
7.58
7.57
7.55
7.52
7.50
7.49
7.48
7.47
7.47
3.40
2.54
2.53
2.53
6n
1.00
1.00
1.05
1.04
1.03
2.10
2.11
2.10
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


xgx072
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10.85
10.85
8.69
8.67
7.96
7.94
7.93
7.88
7.86
7.82
7.81
7.80
7.79
7.78
7.78
7.76
7.66
7.44
7.42
7.42
7.40
7.26
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O-
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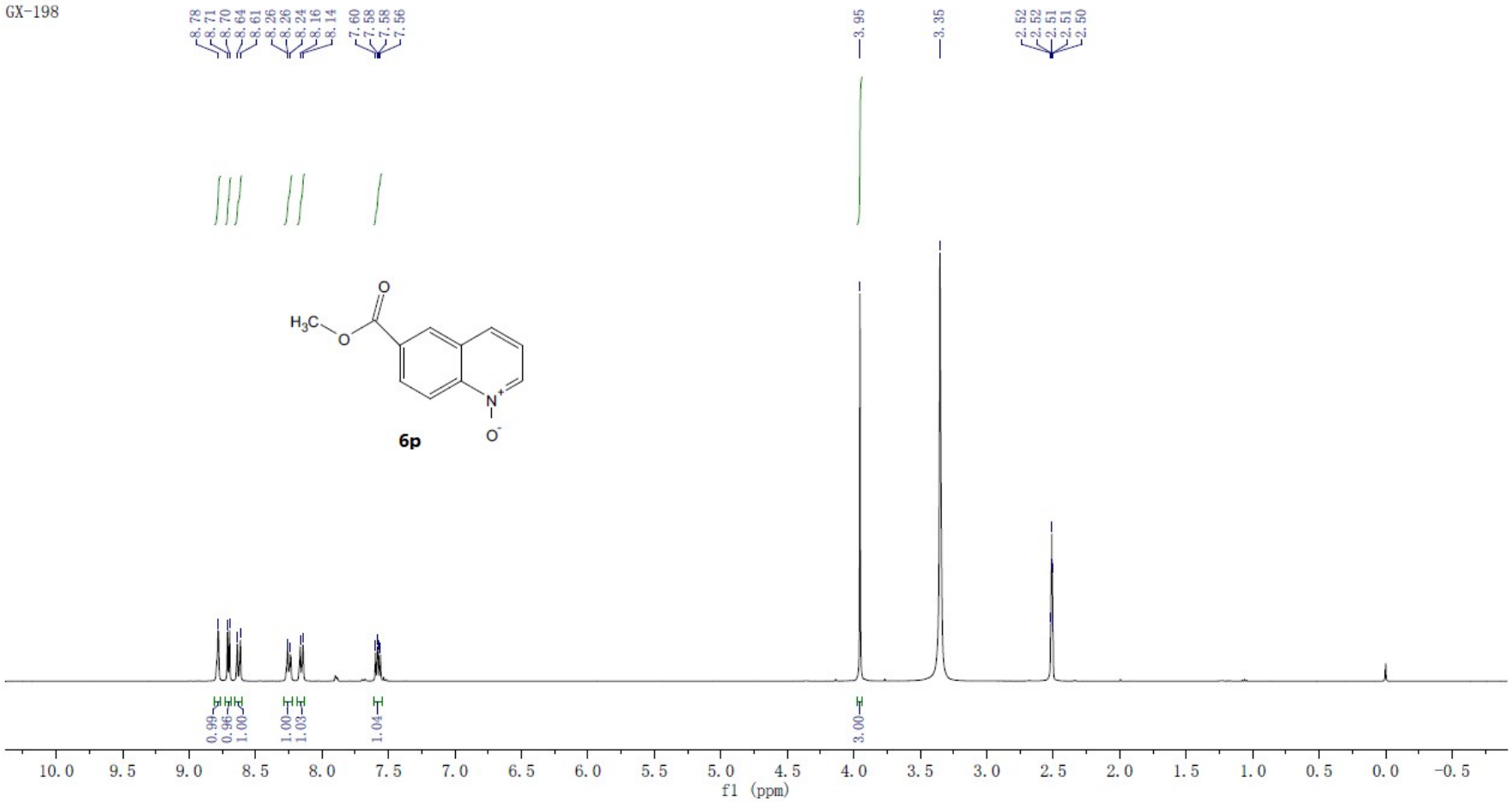
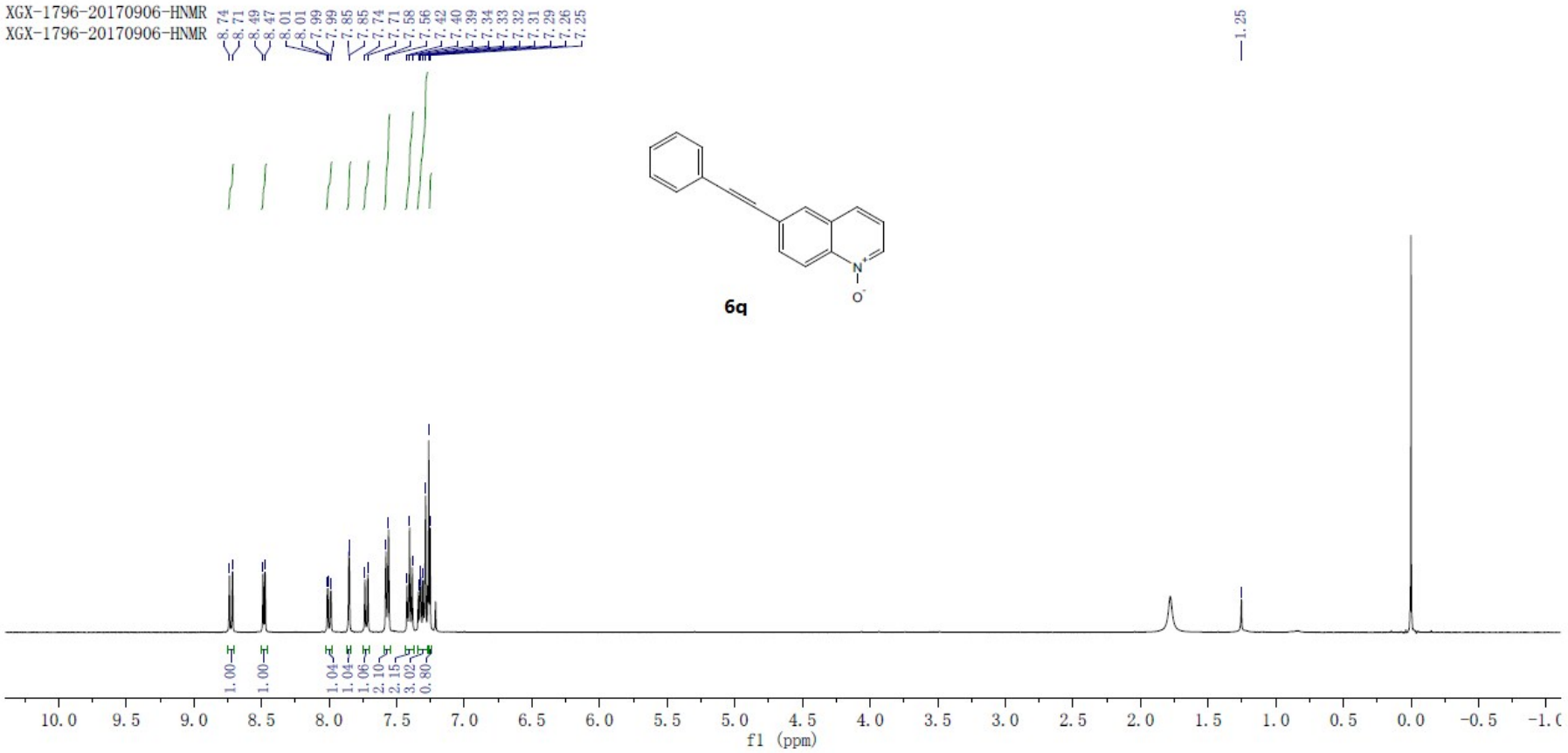
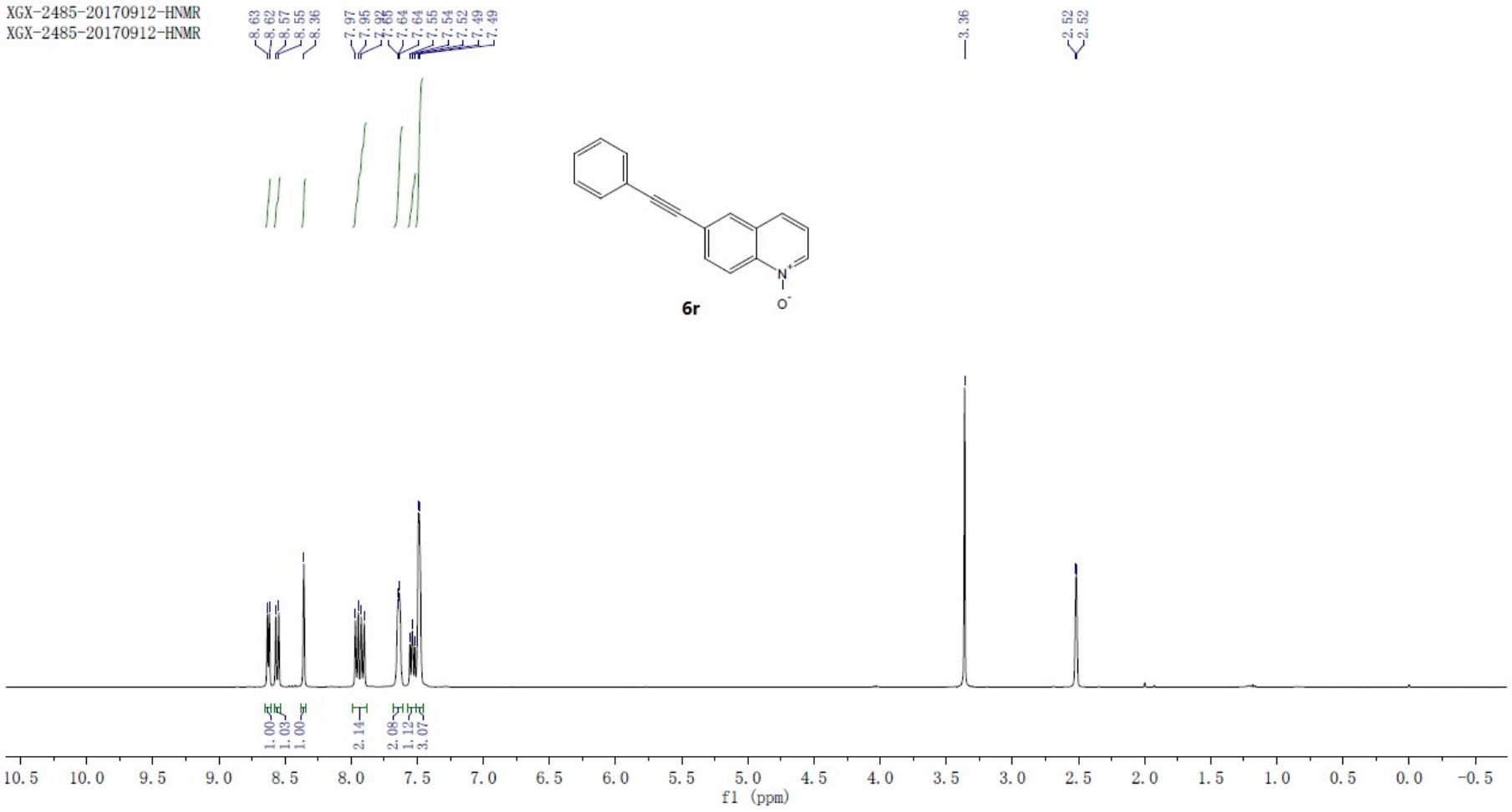
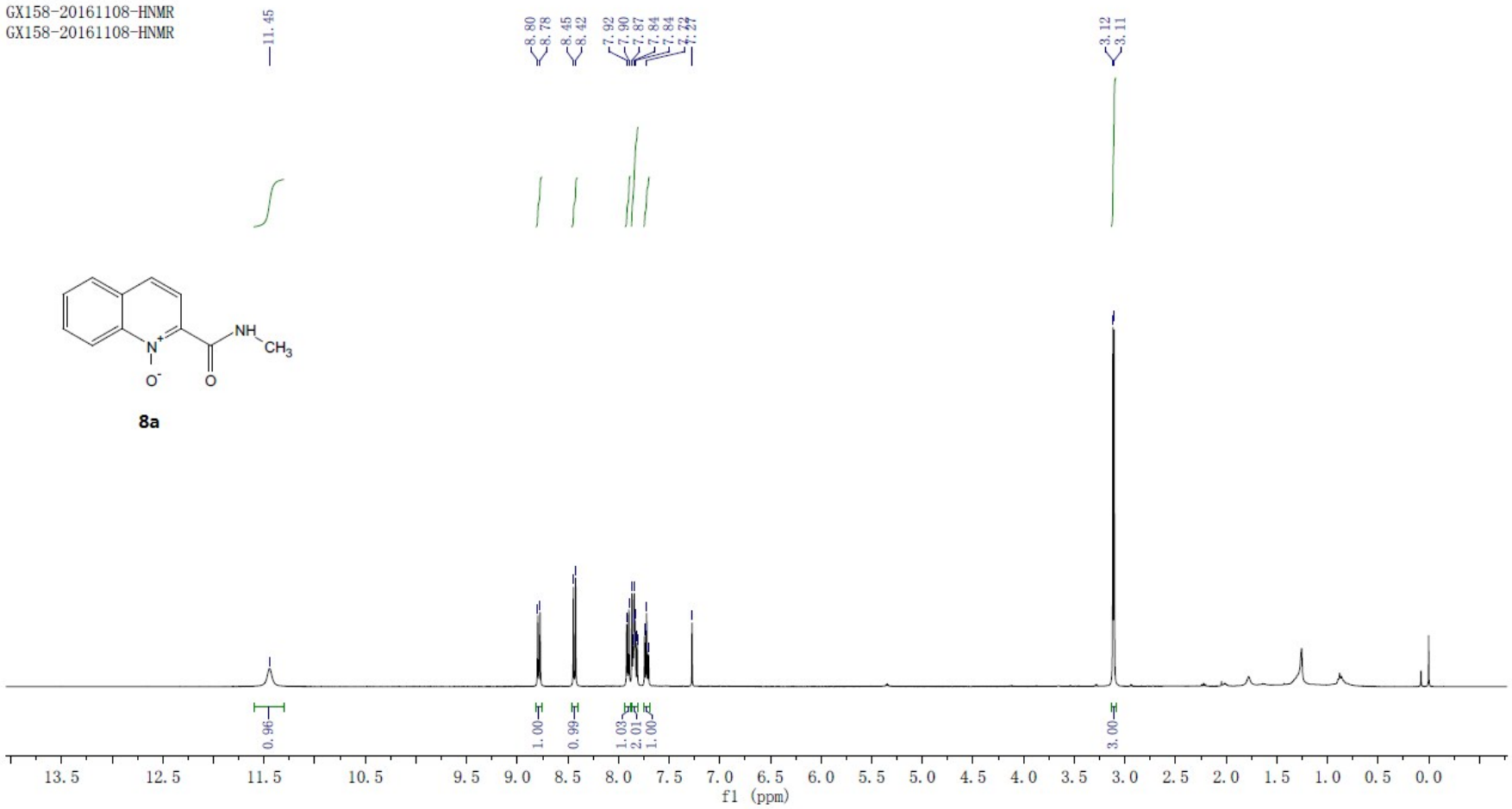
1.02
1.02
3.01
1.02
1.01
f1 (ppm)

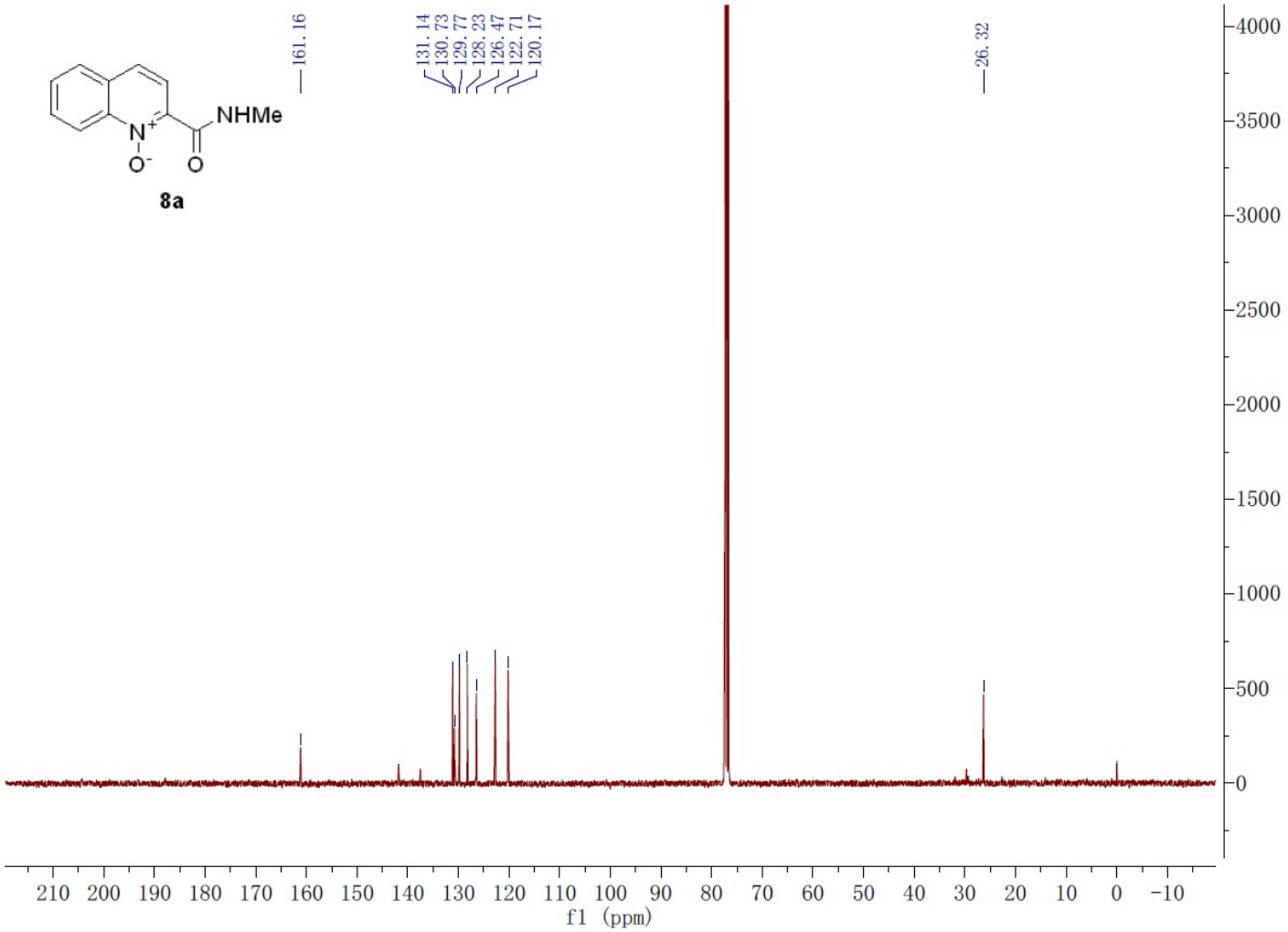

GX-198
8.78
8.71
8.70
8.64
8.61
8.26
8.26
8.24
8.16
8.14
7.60
7.58
7.58
7.56
3.95
3.35
2.52
2.52
2.51
2.51
2.50
O
H3C
O
N+
O-
6p
0.99
0.96
1.00
1.00
1.03
1.04
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

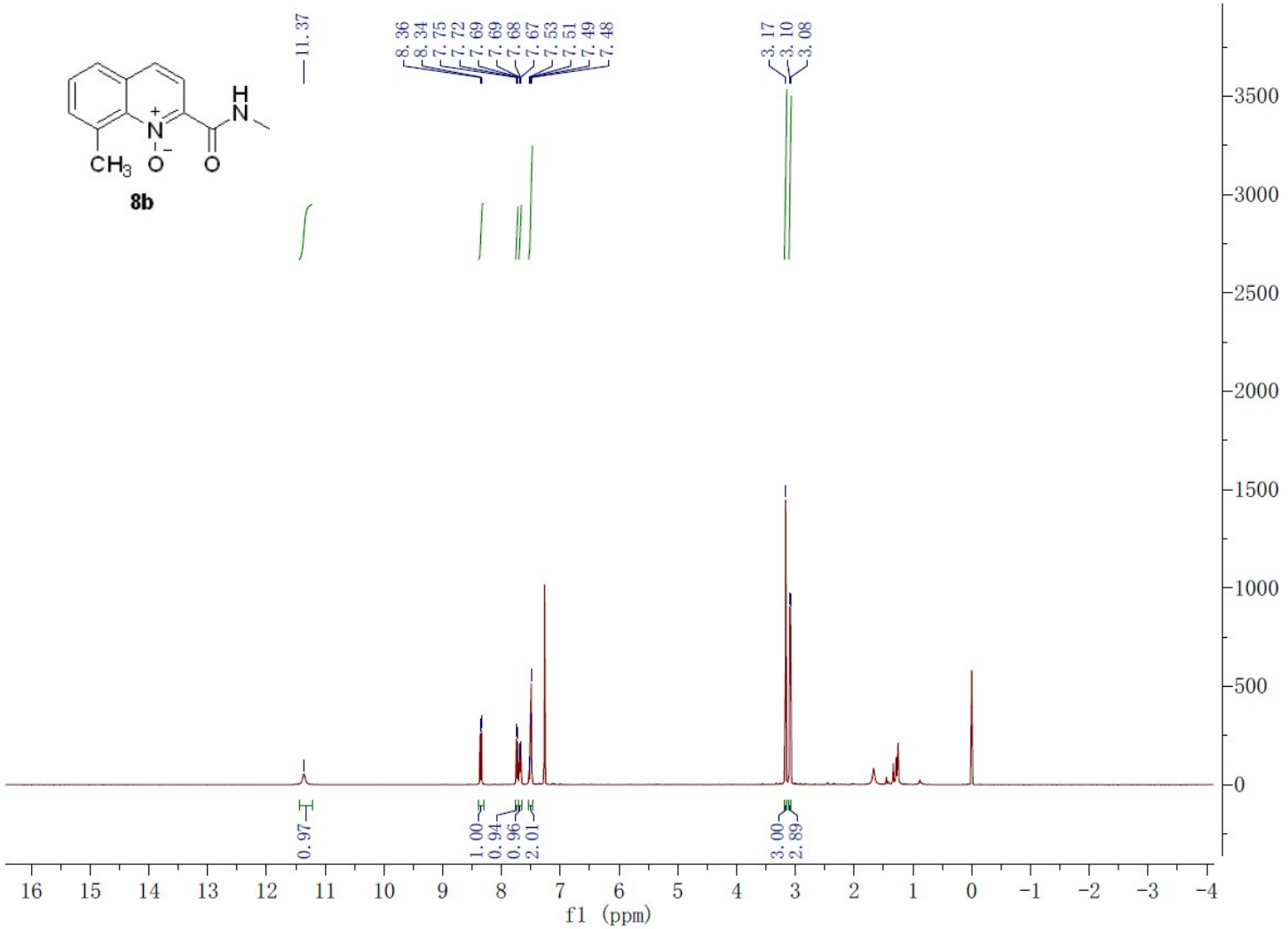

XGX-1796-20170906-HNMR
XGX-1796-20170906-HNMR
8.74
8.71
8.49
8.47
8.01
8.01
7.99
7.99
7.85
7.85
7.74
7.71
7.58
7.56
7.42
7.40
7.39
7.34
7.33
7.32
7.31
7.29
7.26
7.25
1.25
6q
1.00
1.00
1.04
1.04
1.06
2.10
2.15
3.02
0.80
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

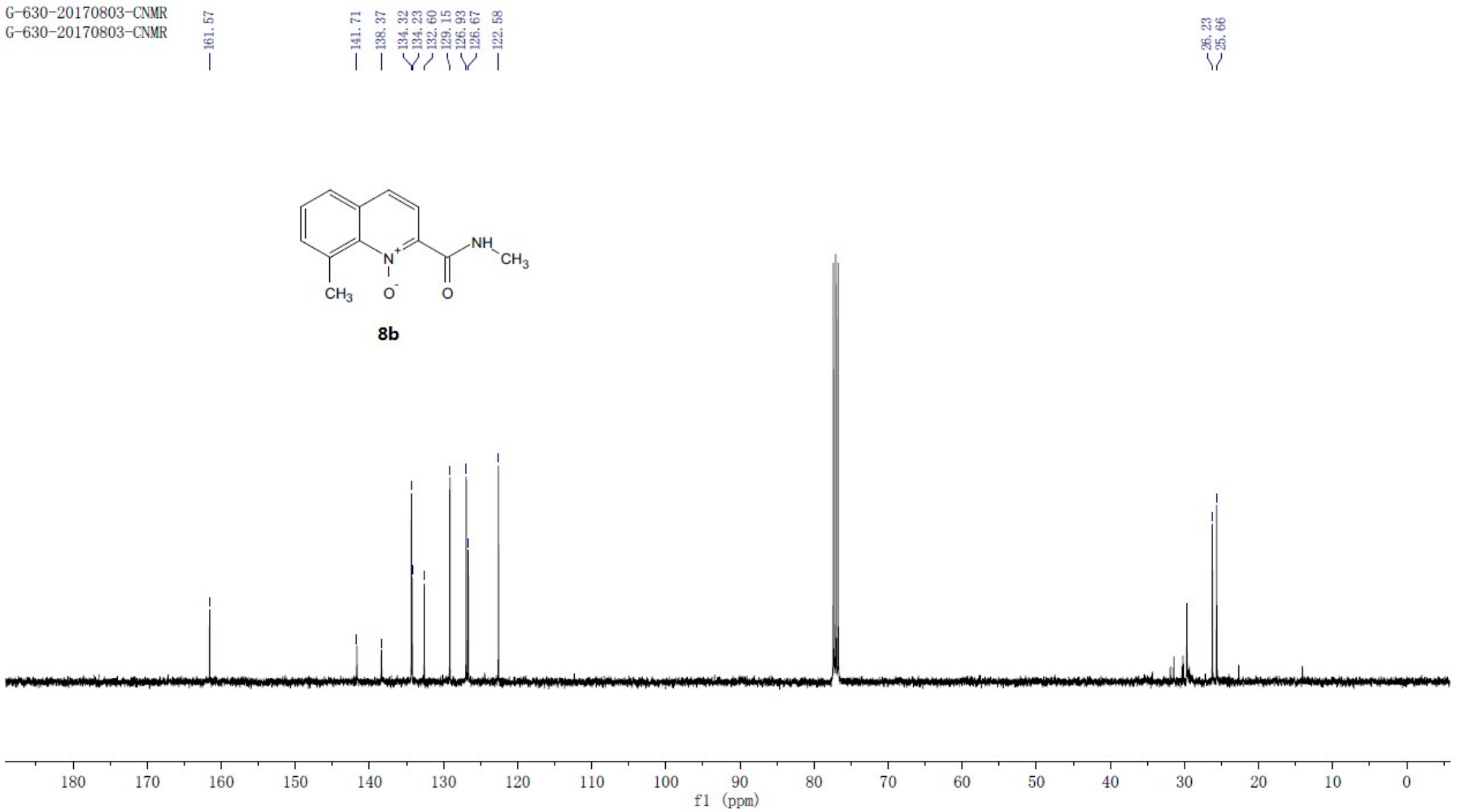


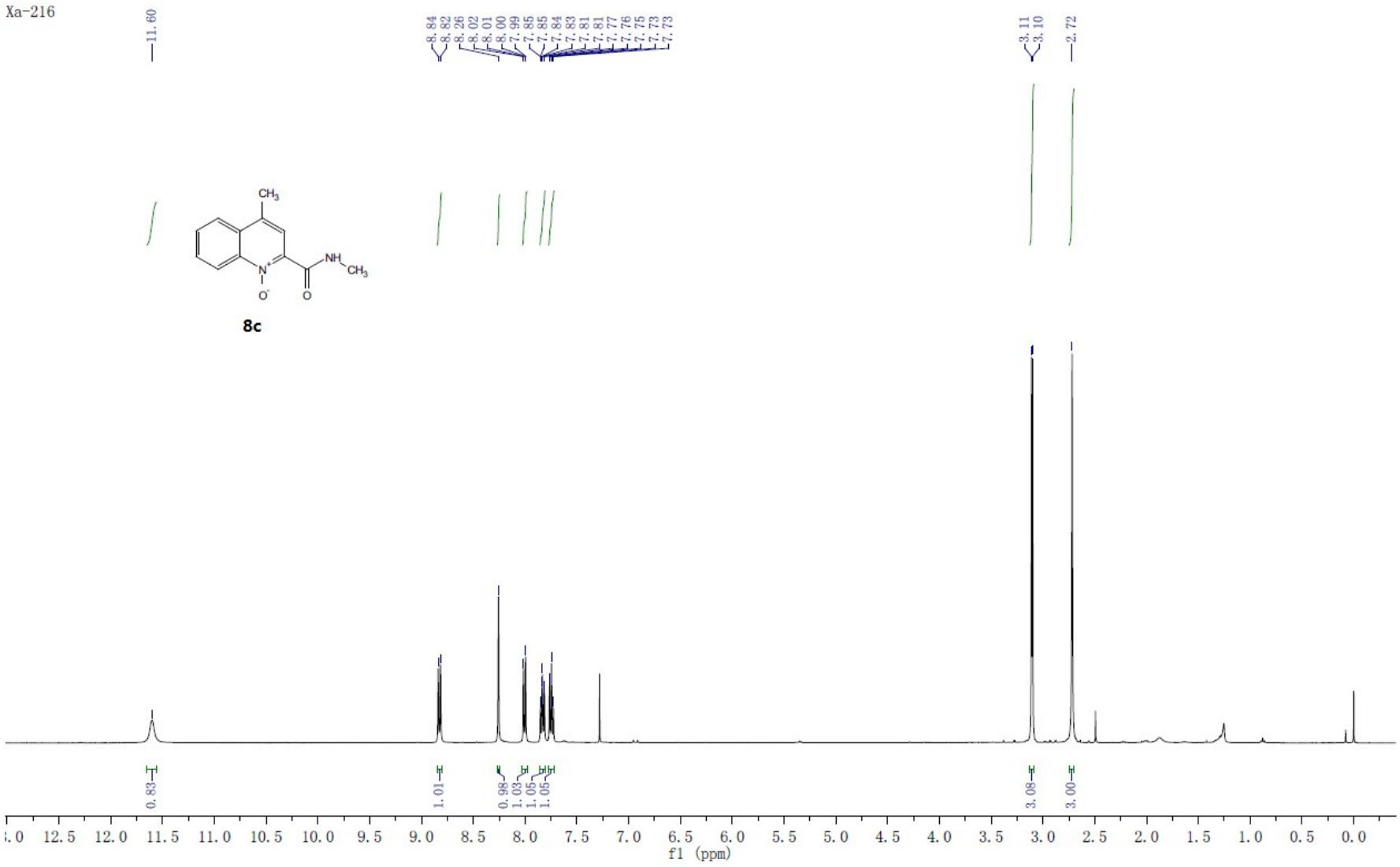
7.2 ^{1}H and ^{13}C NMR Spectra of Products

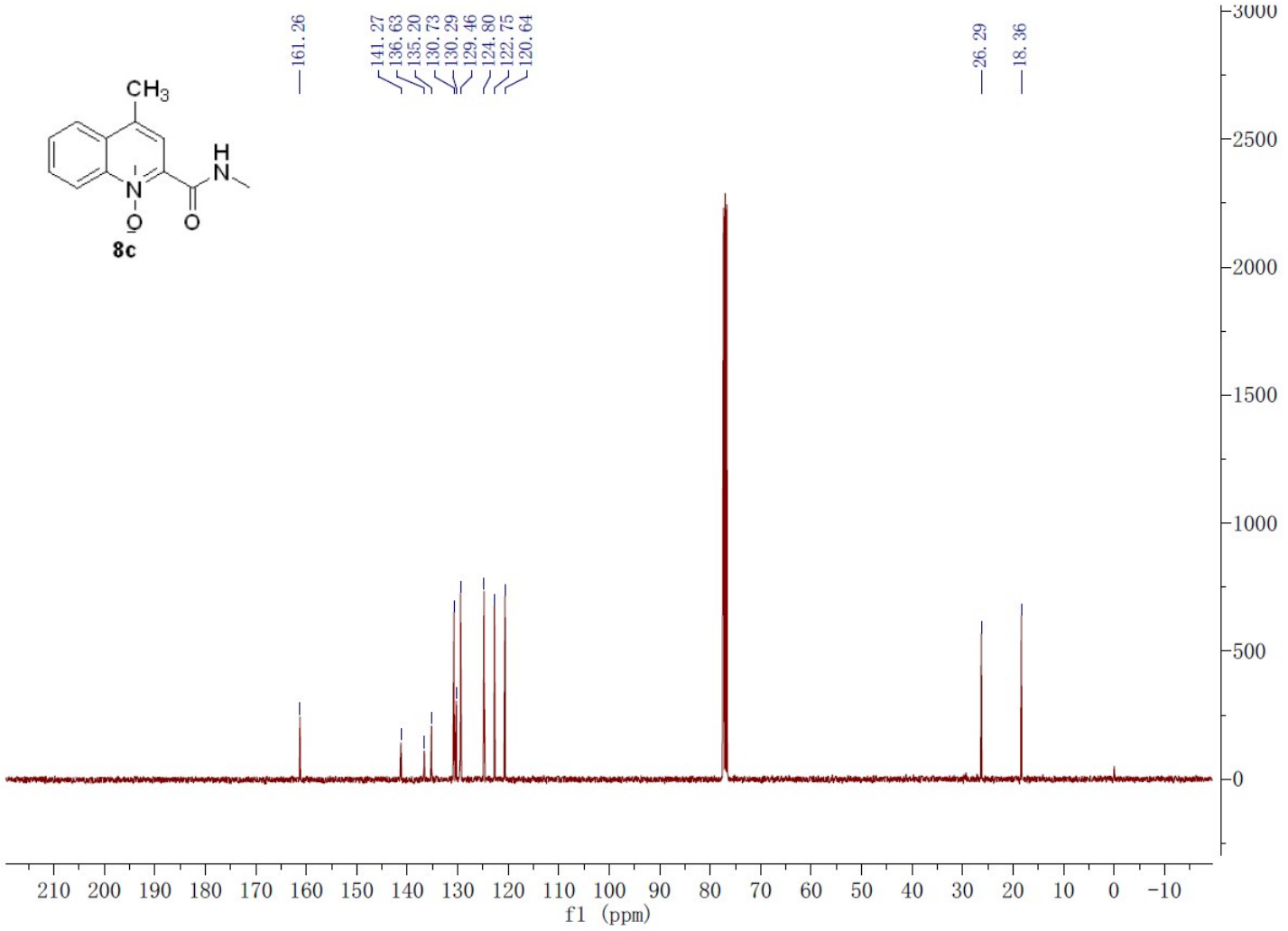

GX158-20161108-HNMR
GX158-20161108-HNMR
11.45
8.80
8.78
8.45
8.42
7.92
7.90
7.87
7.84
7.84
7.72
7.27
3.12
3.11
NH
CH3
N+
O-
O
8a
0.96
1.00
0.99
1.03
2.01
1.00
3.00
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

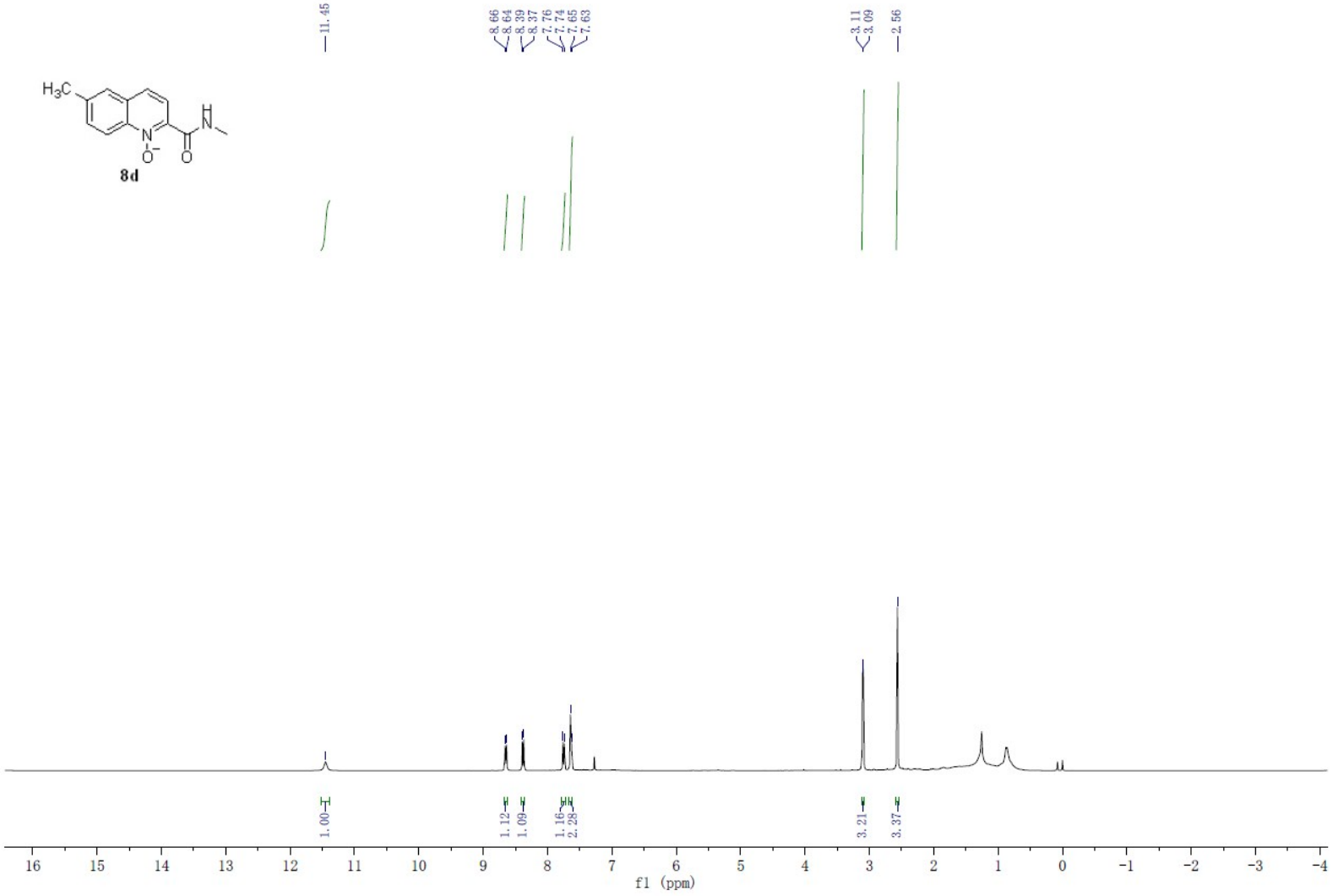

161.16
131.14
130.73
129.77
128.23
126.47
122.71
120.17
26.32
NHMe
N+
O-
O
8a
4000
3500
3000
2500
2000
1500
1000
500
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

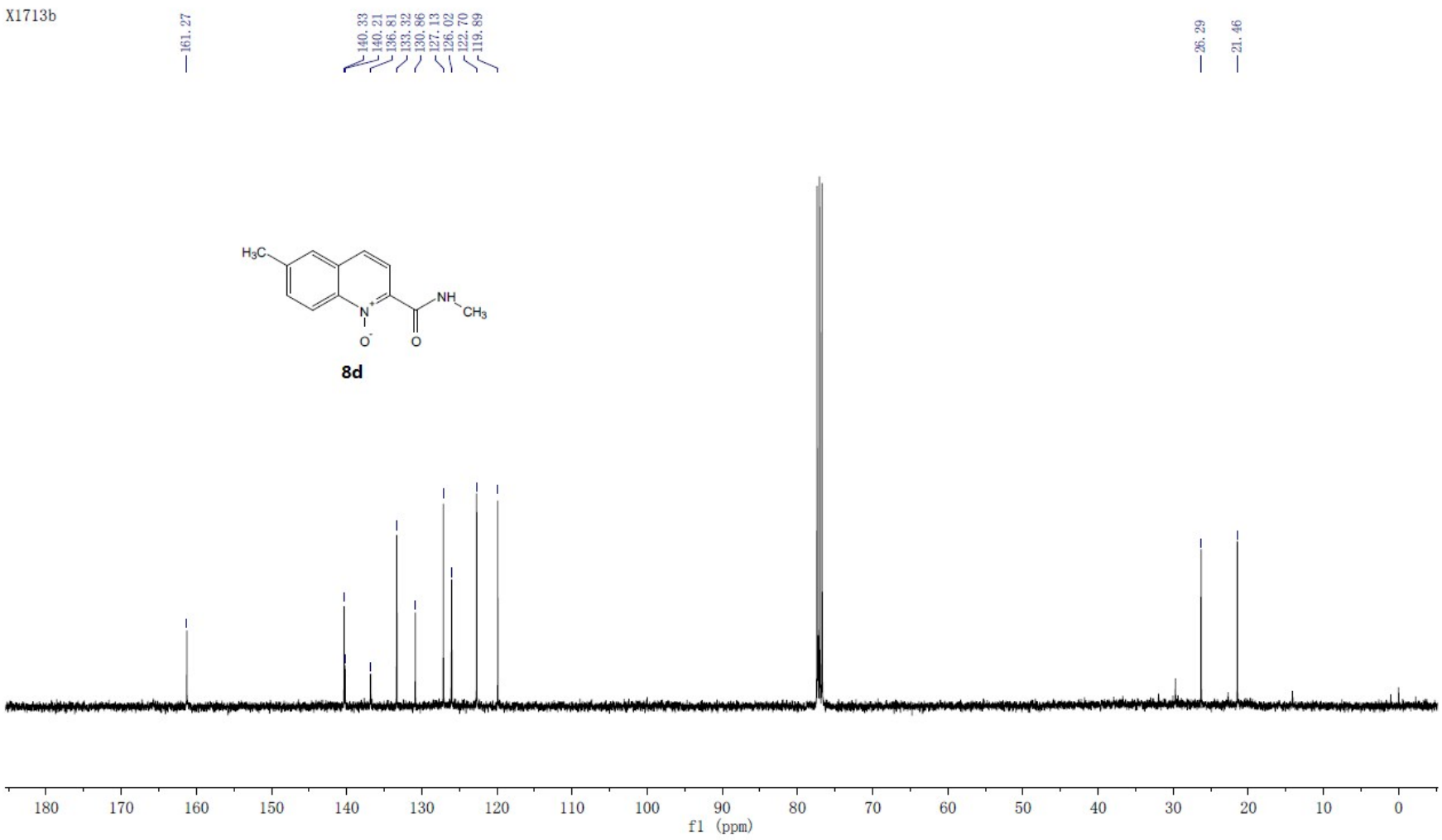

11.37
8.36
8.34
7.75
7.72
7.69
7.69
7.68
7.67
7.53
7.51
7.49
7.48
3.17
3.10
3.08
8b
0.97
1.00
0.94
0.96
2.01
3.00
2.89
f1 (ppm)

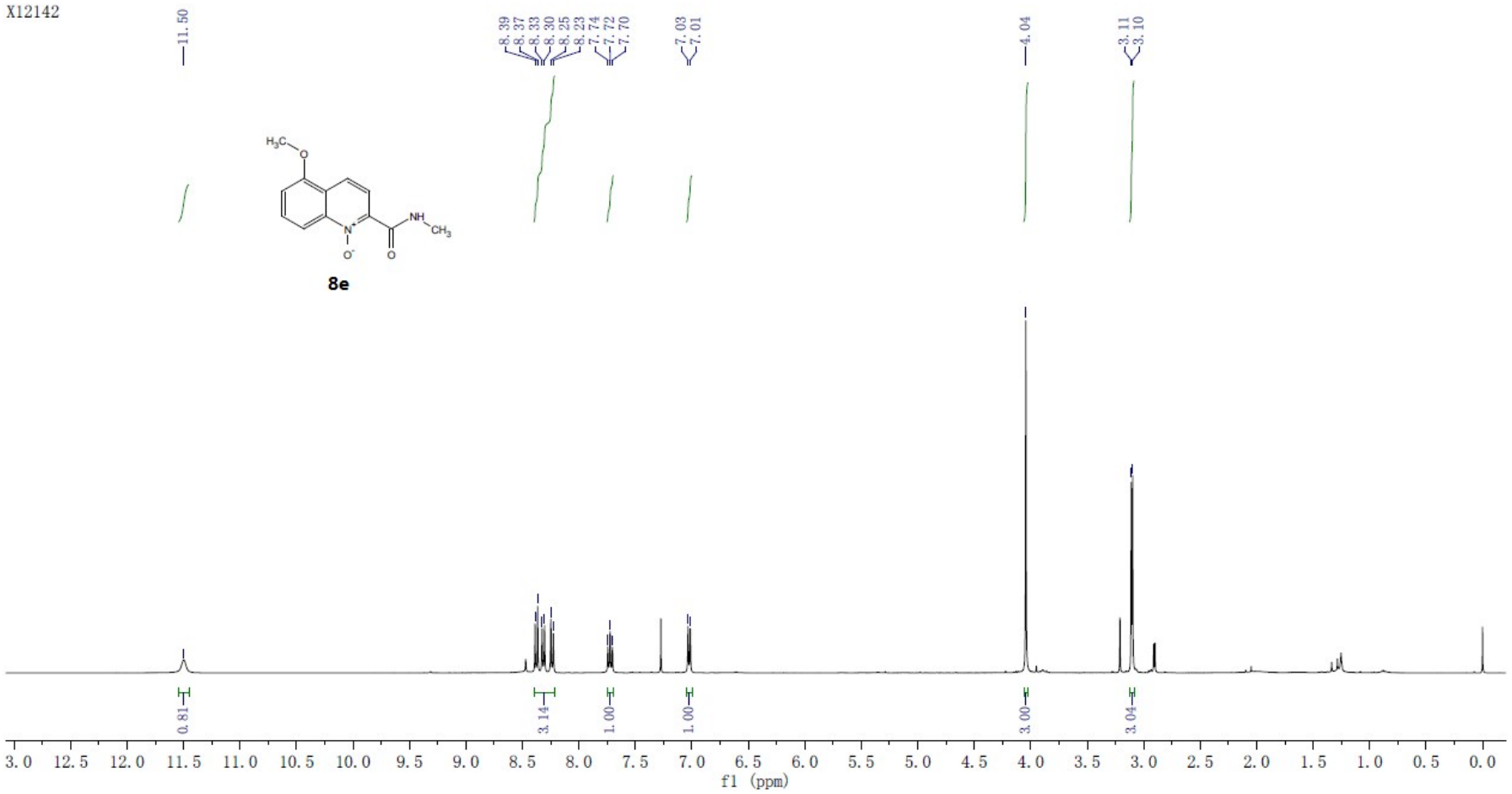

G-630-20170803-CNMR
G-630-20170803-CNMR
161.57
141.71
138.37
134.32
134.23
132.60
129.15
126.93
126.67
122.58
26.23
25.66
NH
CH3
N+
O-
CH3
O
8b
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

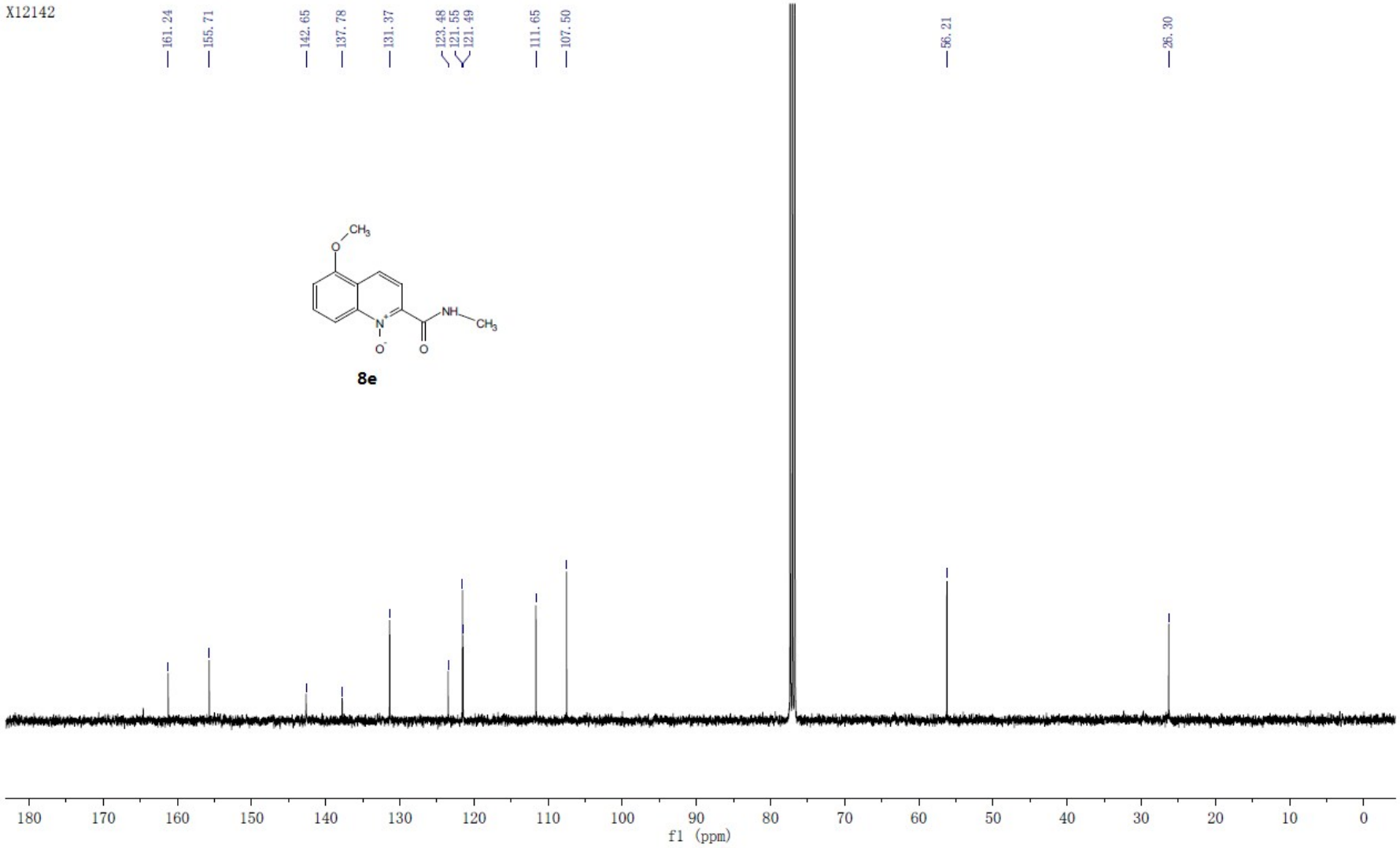

Xa-216
11.60
8.84
8.82
8.26
8.02
8.01
8.00
7.99
7.85
7.85
7.84
7.83
7.81
7.81
7.77
7.76
7.75
7.73
7.73
3.11
3.10
2.72
CH3
NH
CH3
N+
O-
O
8c
0.83
1.01
0.98
1.03
1.05
1.05
3.08
3.00
f1 (ppm)

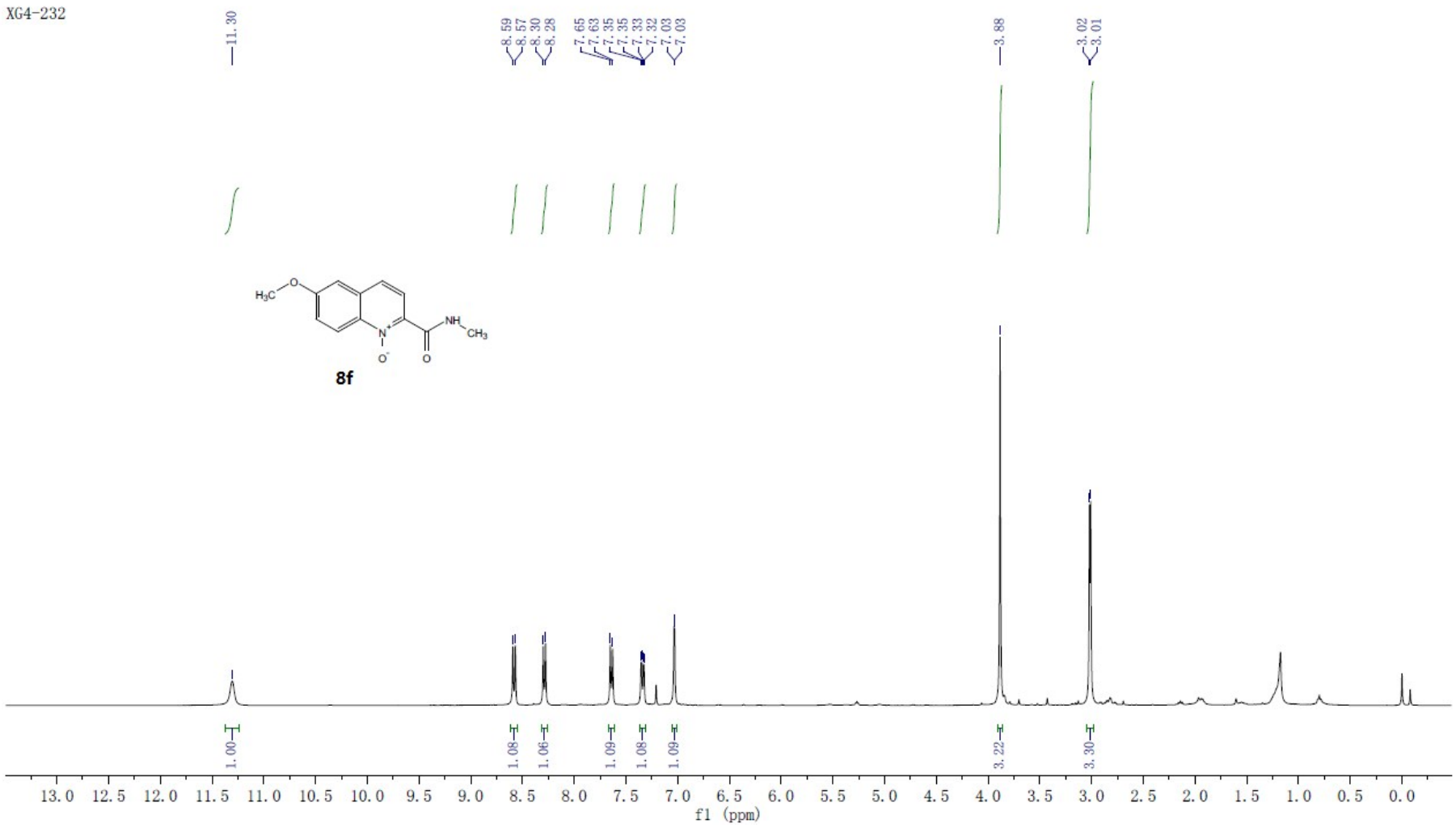

161.26
141.27
136.63
135.20
130.73
130.29
129.46
124.80
122.75
120.64
26.29
18.36
CH3
H
N
N
O
O
8c
3000
2500
2000
1500
1000
500
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

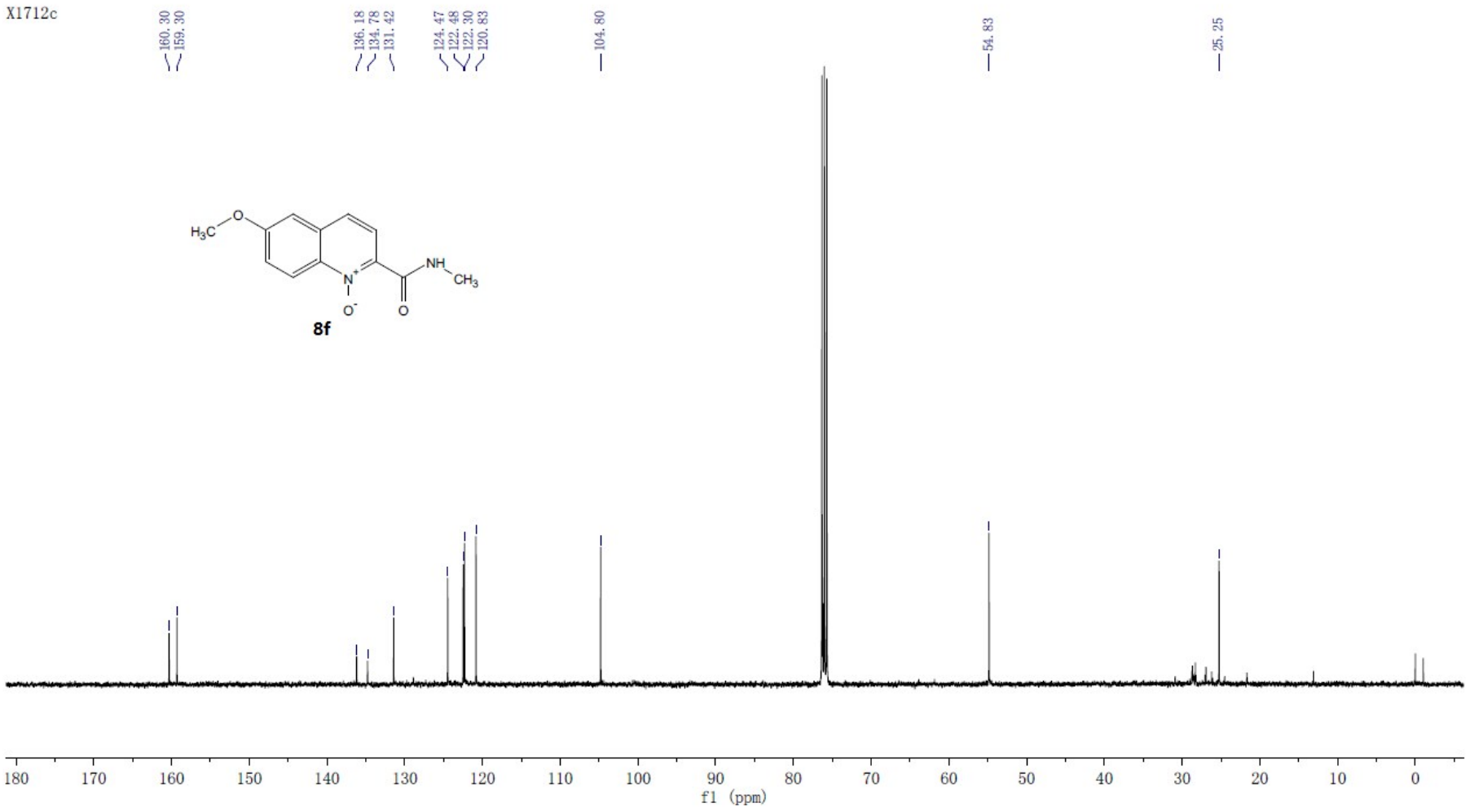

11.45
8.66
8.64
8.39
8.37
7.76
7.74
7.65
7.63
3.11
3.09
2.56
8d
1.00
1.12
1.09
1.16
2.28
3.21
3.37
f1 (ppm)

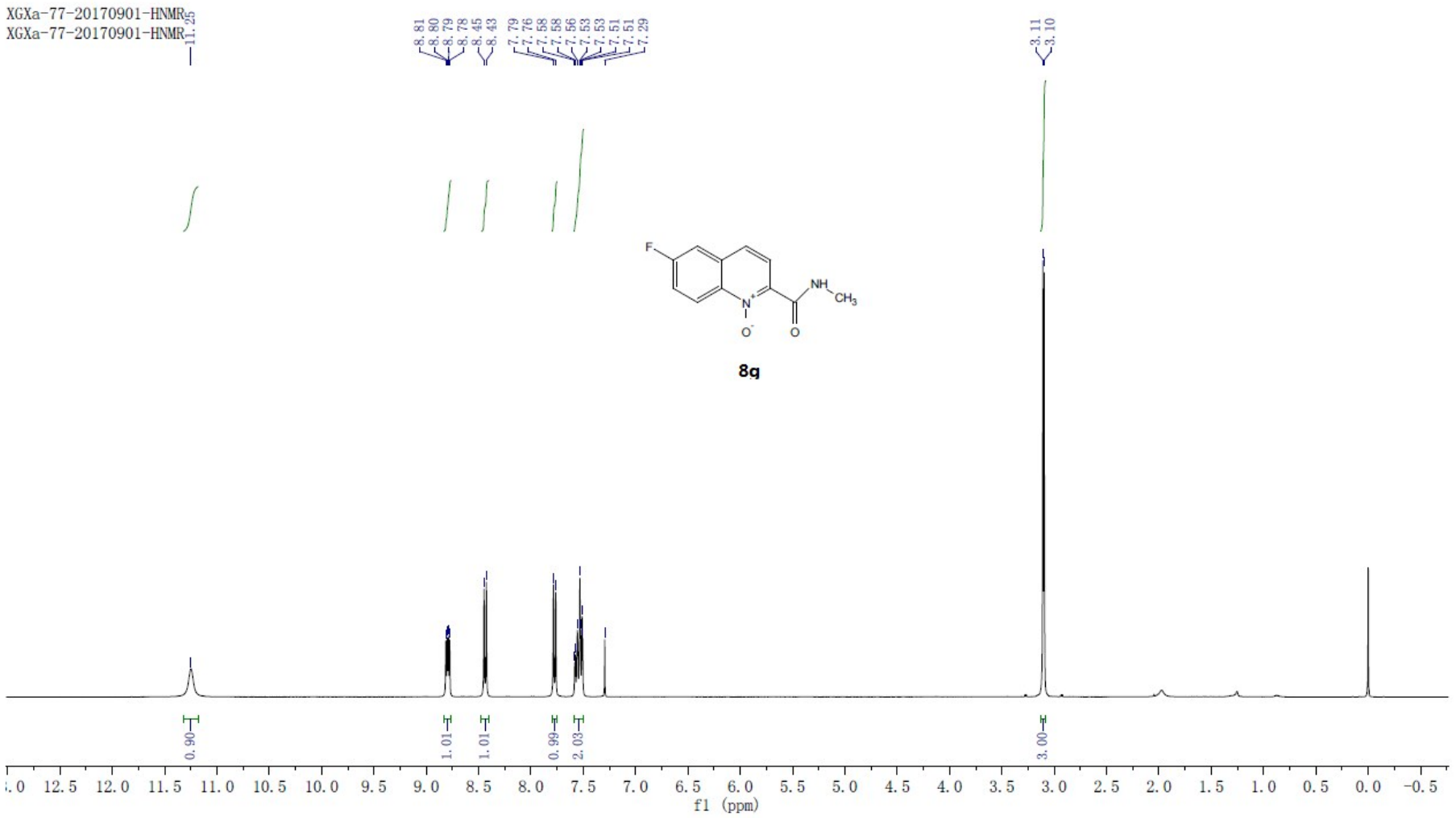

X1713b
161.27
140.33
140.21
136.81
133.32
130.86
127.13
126.02
122.70
119.89
26.29
21.46
H3C
N+
O-
NH
CH3
O
8d
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

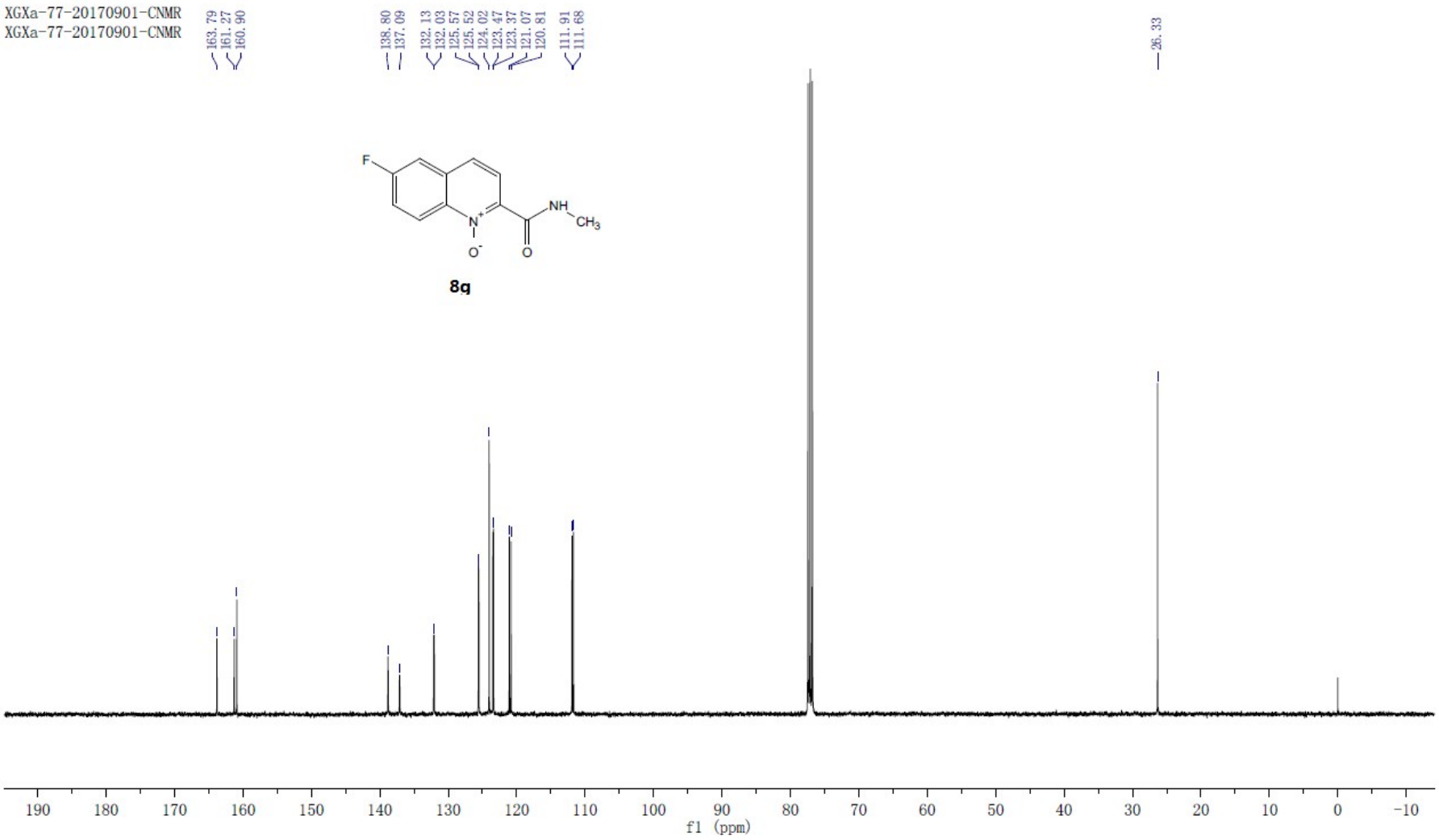

X12142
11.50
8.39
8.37
8.33
8.30
8.25
8.23
7.74
7.72
7.70
7.03
7.01
4.04
3.11
3.10
8e
0.81
3.14
1.00
1.00
3.00
3.04
f1 (ppm)

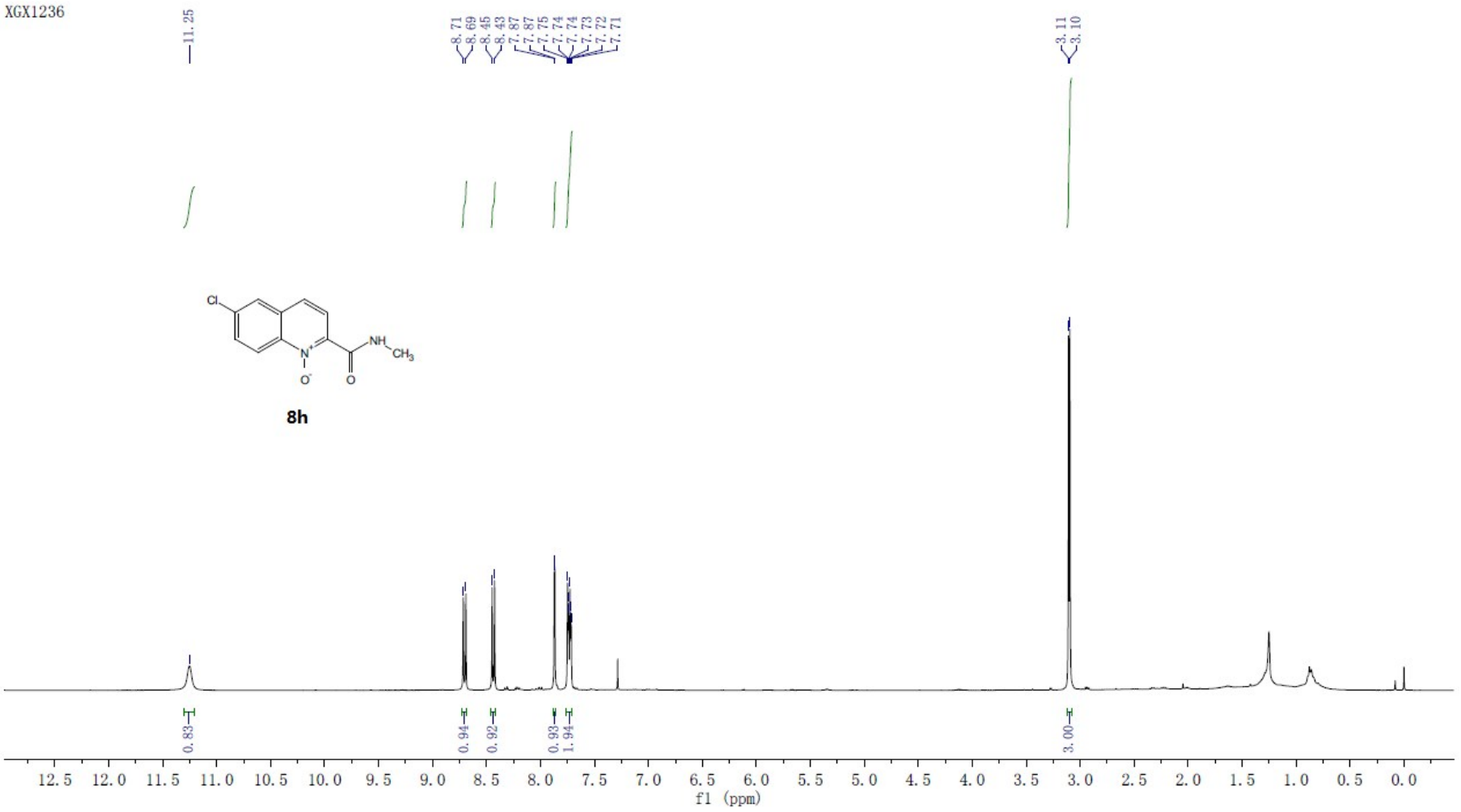

X12142
161.24
155.71
142.65
137.78
131.37
123.48
121.55
121.49
111.65
107.50
56.21
26.30
CH3
O
NH
CH3
N+
O-
O
8e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

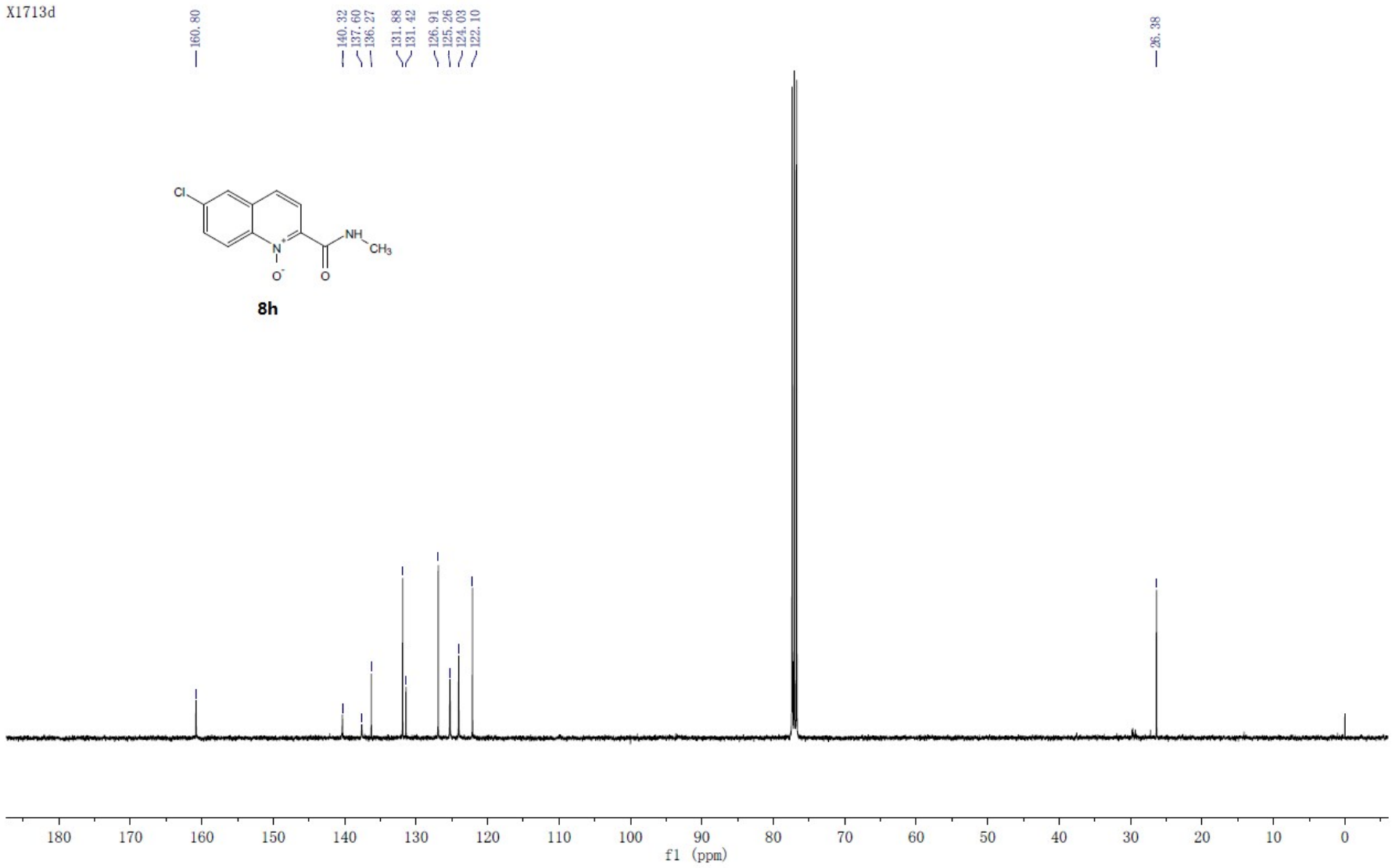

XG4-232
11.30
8.59
8.57
8.30
8.28
7.65
7.63
7.35
7.35
7.33
7.32
7.03
7.03
3.88
3.02
3.01
8f
1.00
1.08
1.06
1.09
1.08
1.09
3.22
3.30
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

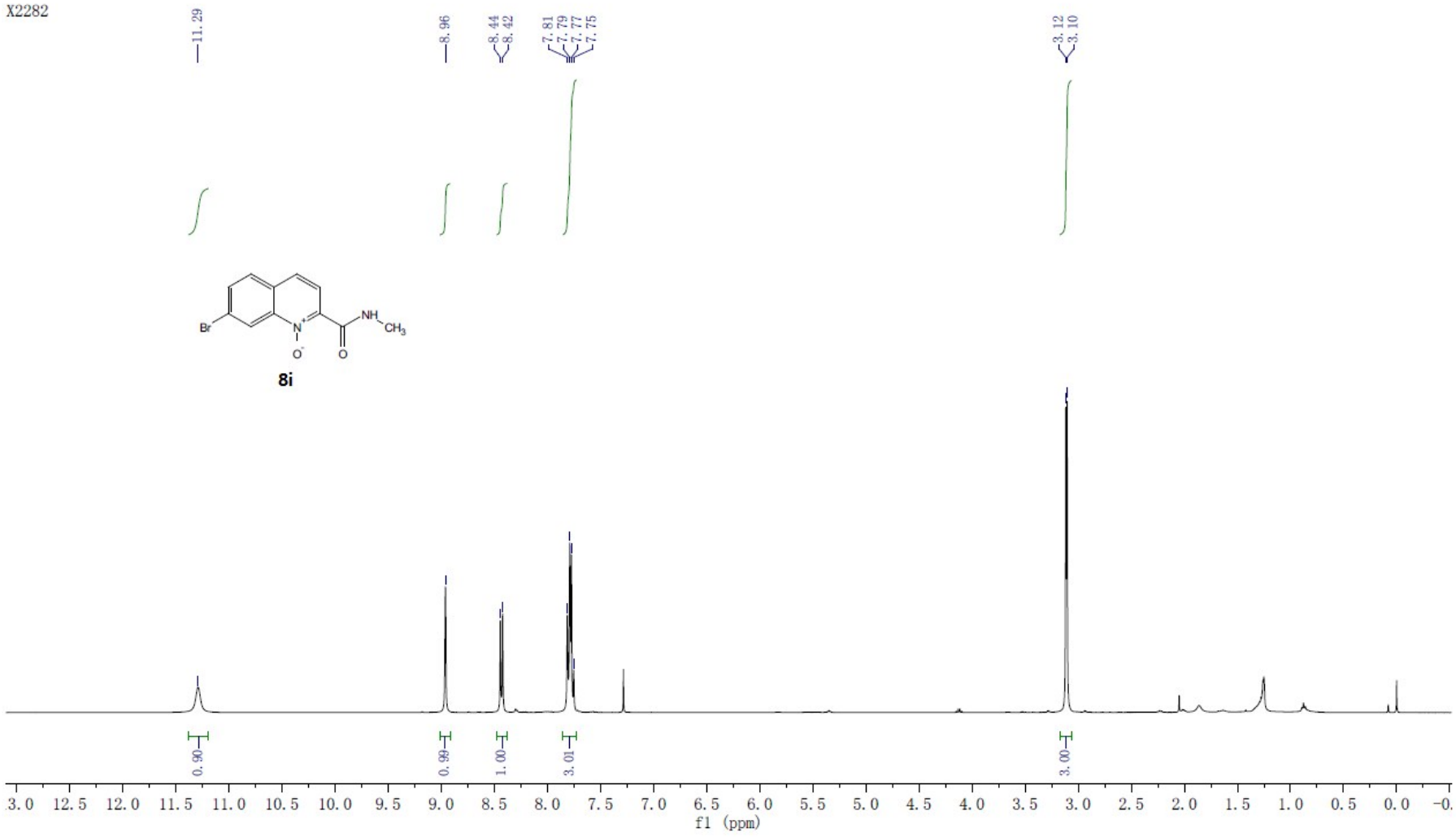

X1712c
160.30
159.30
136.18
134.78
131.42
124.47
122.48
122.30
120.83
104.80
54.83
25.25
8f
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

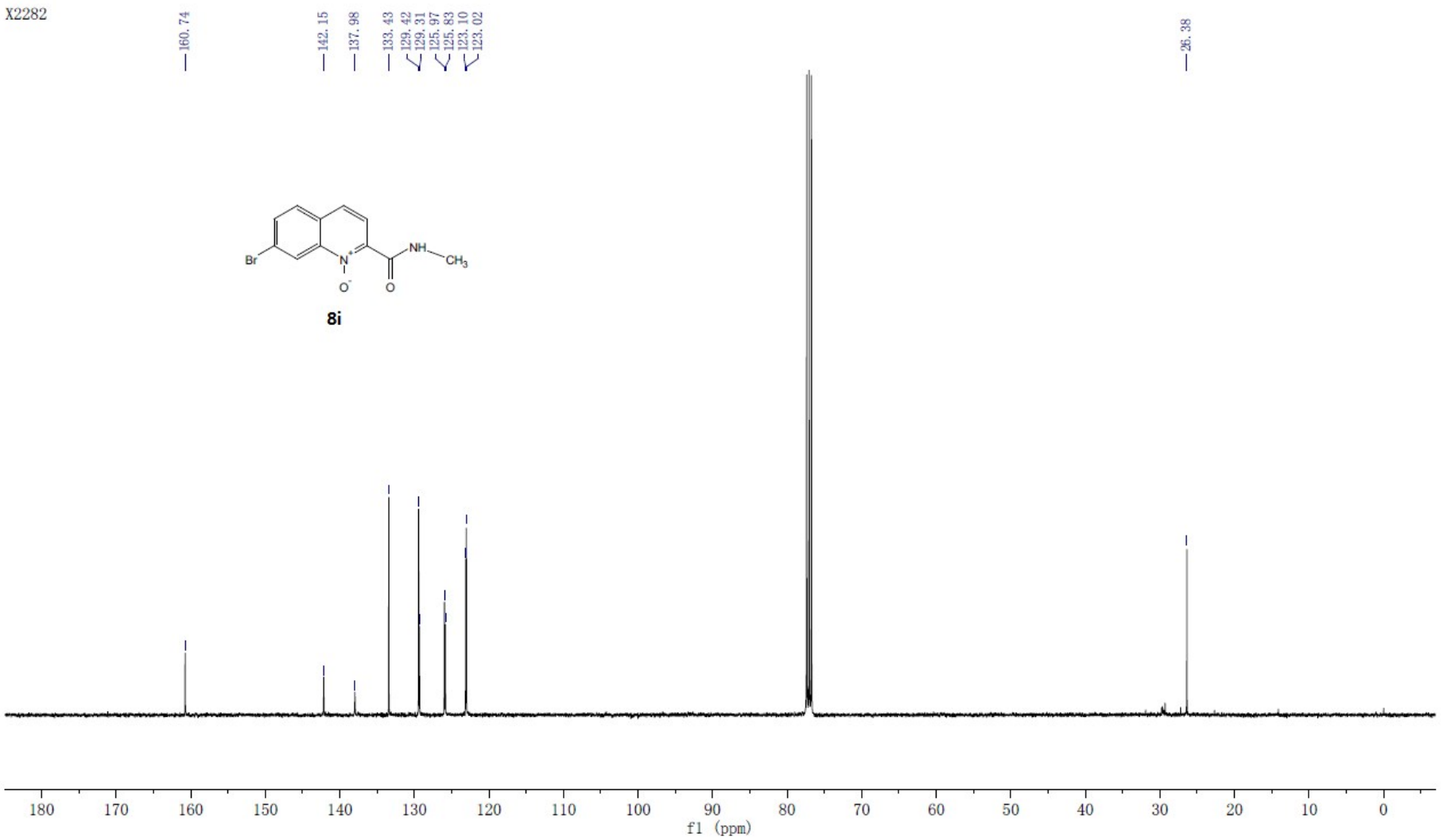

XGXa-77-20170901-HNMR
XGXa-77-20170901-HNMR
11.25
8.81
8.80
8.79
8.78
8.45
8.43
7.79
7.76
7.58
7.58
7.56
7.53
7.53
7.51
7.51
7.29
3.11
3.10
F
NH
CH3
N+
O-
O
8q
0.90
1.01
1.01
0.99
2.03
3.00
f1 (ppm)

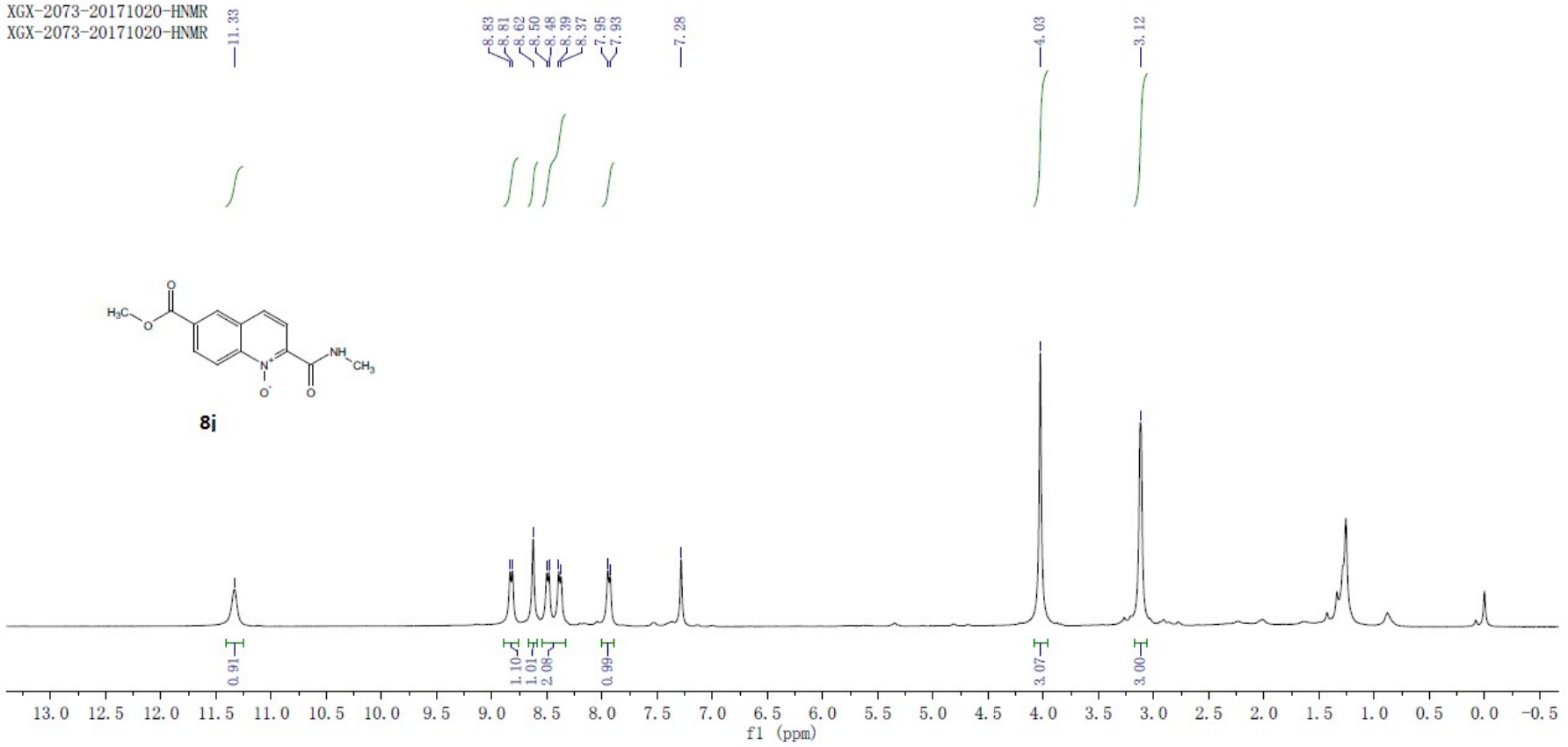

XGXa-77-20170901-CNMR
XGXa-77-20170901-CNMR
163.79
161.27
160.90
138.80
137.09
132.13
132.03
125.57
125.52
124.02
123.47
123.37
121.07
120.81
111.91
111.68
26.33
F
N+
O-
NH
CH3
O
8q
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

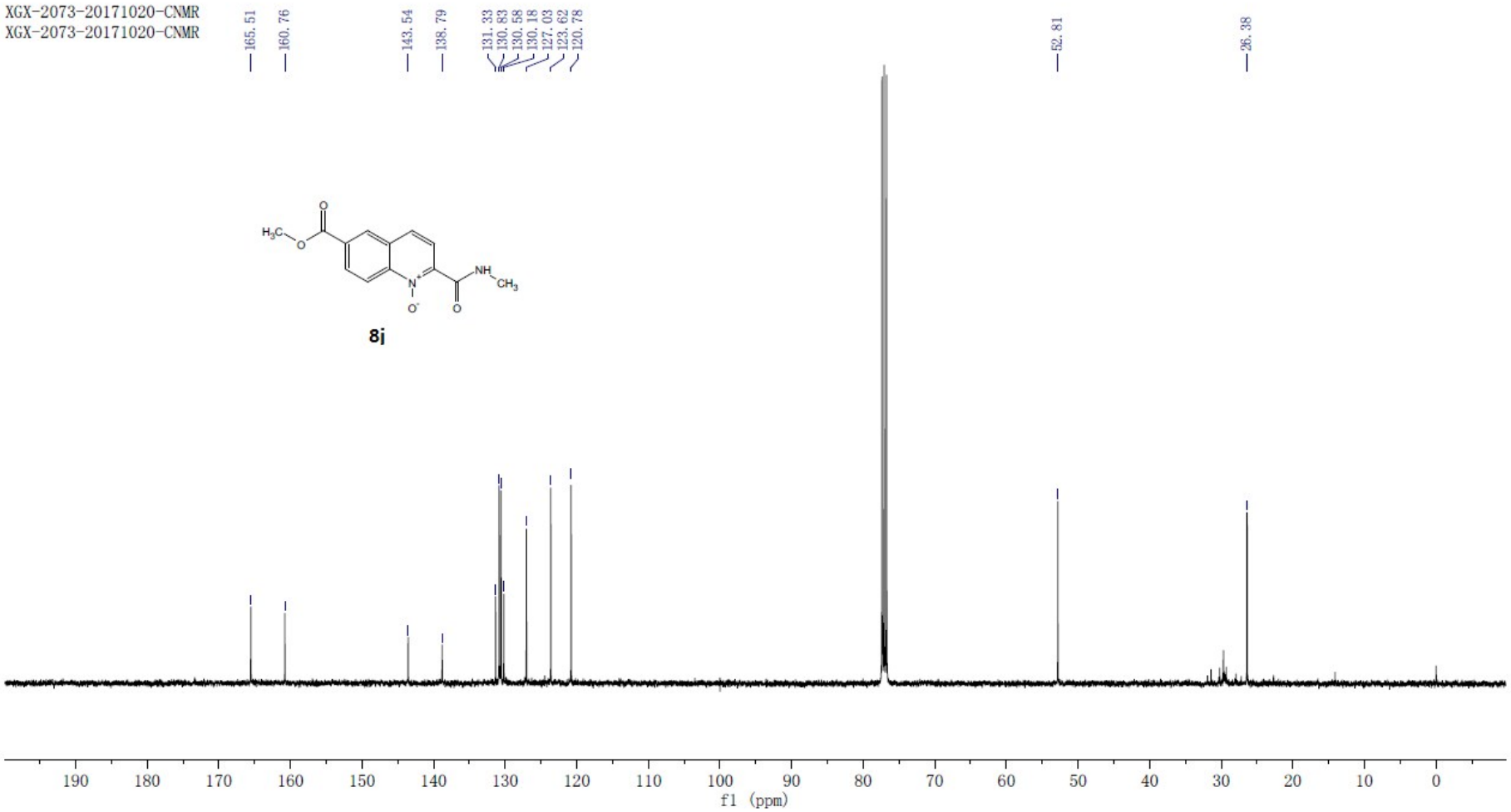

XGX1236
11.25
8.71
8.69
8.45
8.43
7.87
7.87
7.75
7.74
7.74
7.73
7.72
7.71
3.11
3.10
Cl
N+
O-
NH
CH3
O
8h
0.83
0.94
0.92
0.93
1.94
3.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

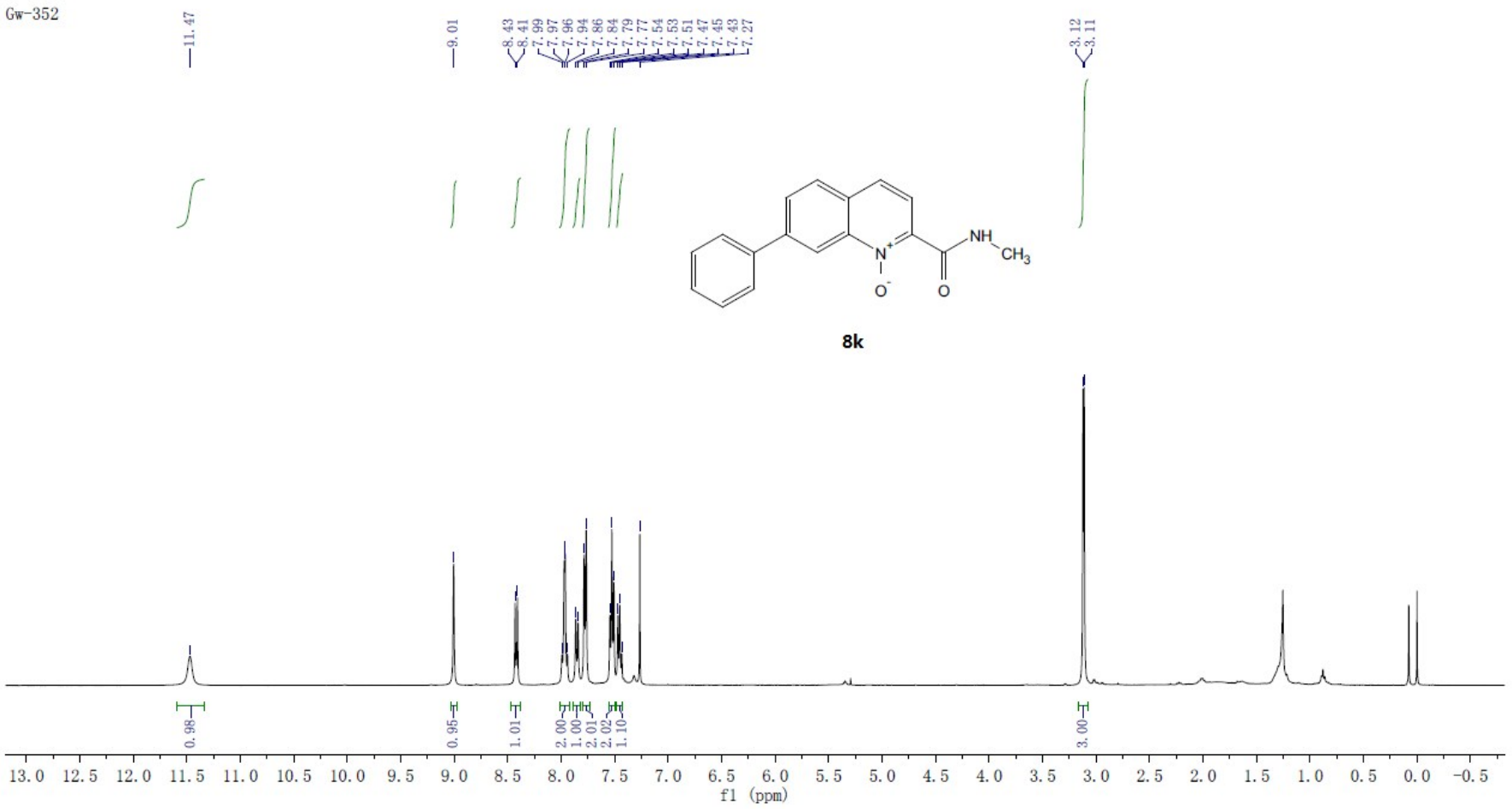

X1713d
160.80
140.32
137.60
136.27
131.88
131.42
126.91
125.26
124.03
122.10
26.38
Cl
N+
O-
NH
CH3
O
8h
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

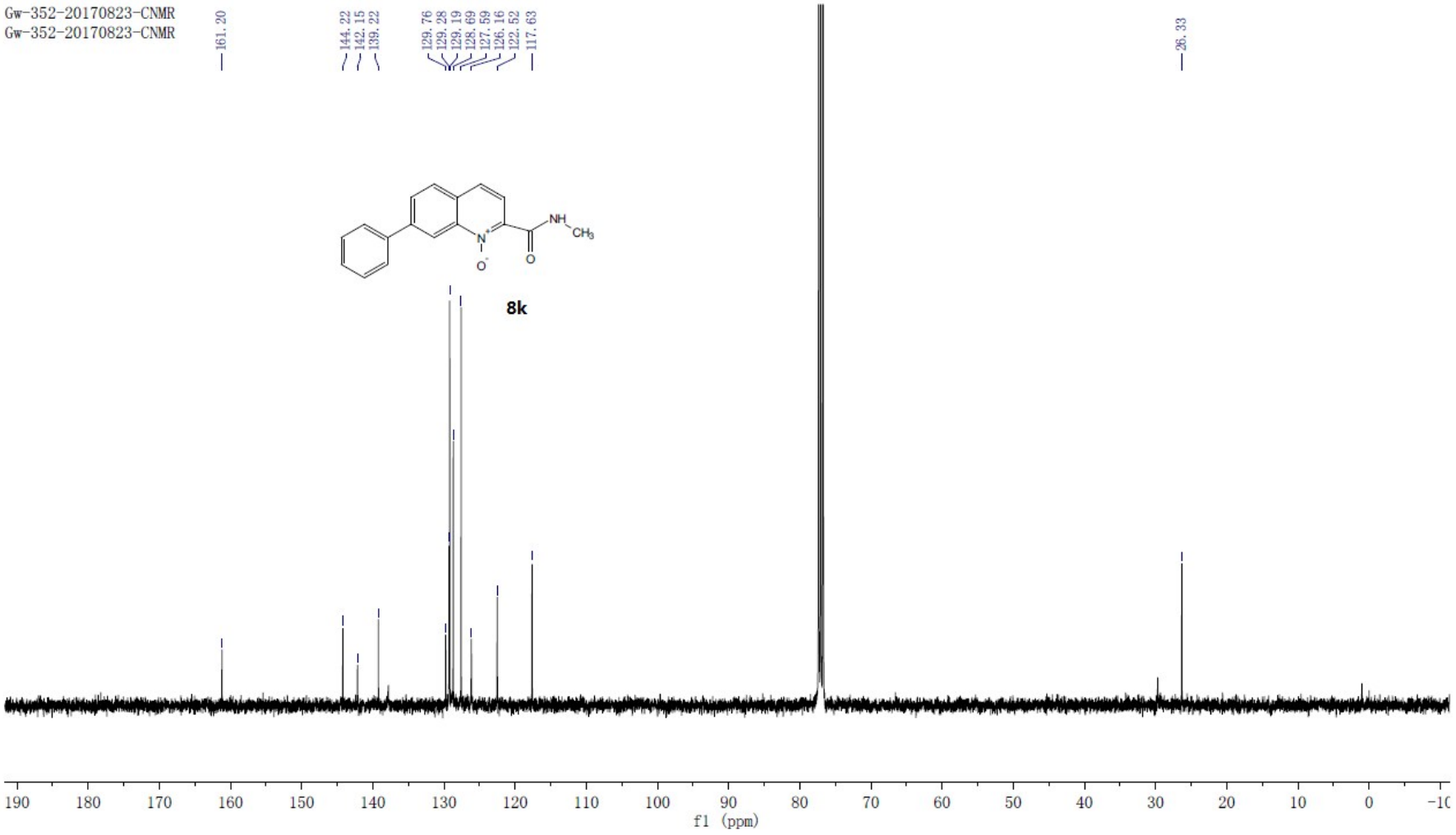

X2282
11.29
8.96
8.44
8.42
7.81
7.79
7.77
7.75
3.12
3.10
Br
N+
O-
NH
CH3
O
8i
0.90
0.99
1.00
3.01
3.00
f1 (ppm)

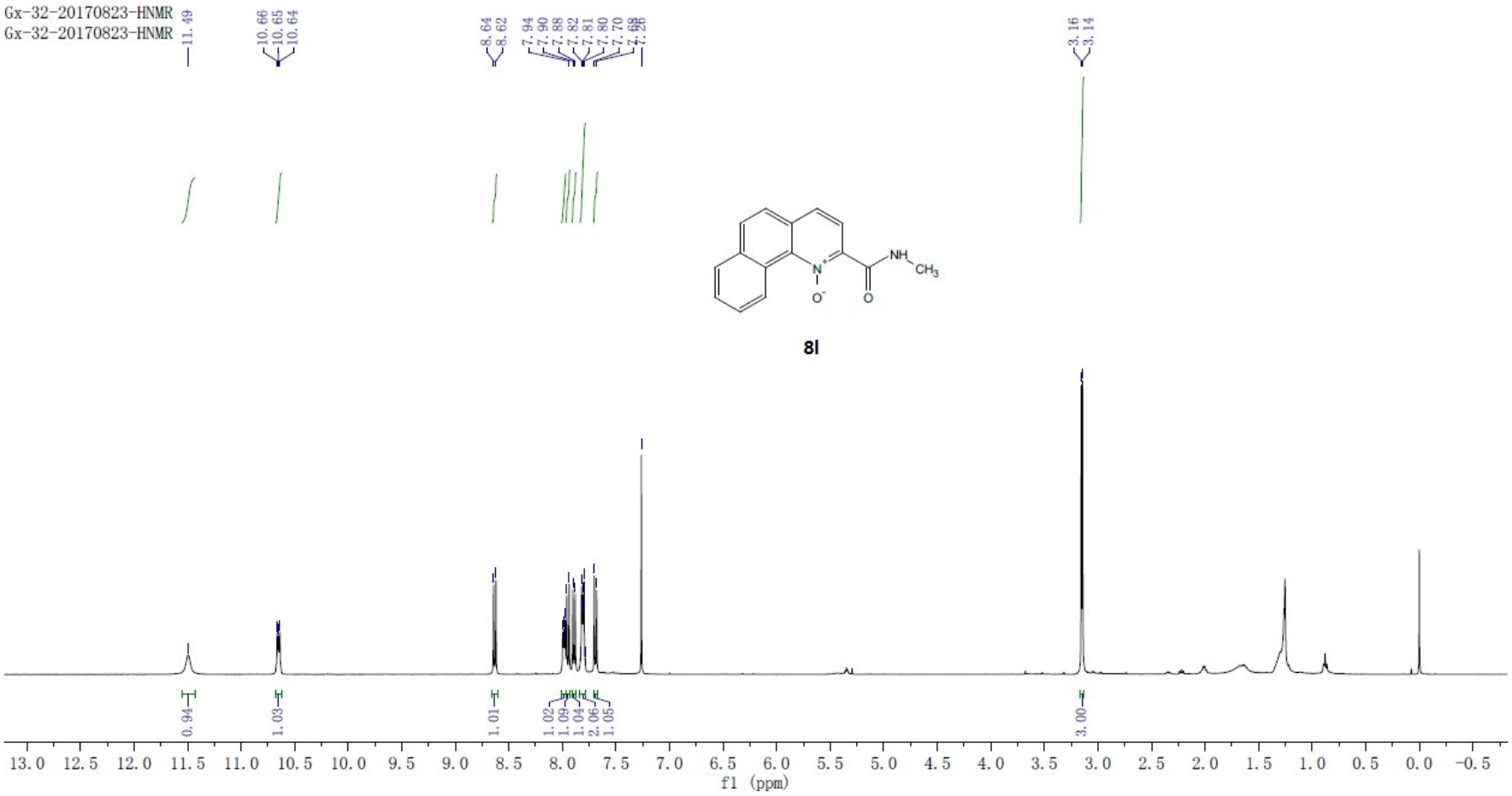

X2282
160.74
142.15
137.98
133.43
129.42
129.31
125.97
125.83
123.10
123.02
26.38
Br
N
O
NH
CH3
O
8i
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

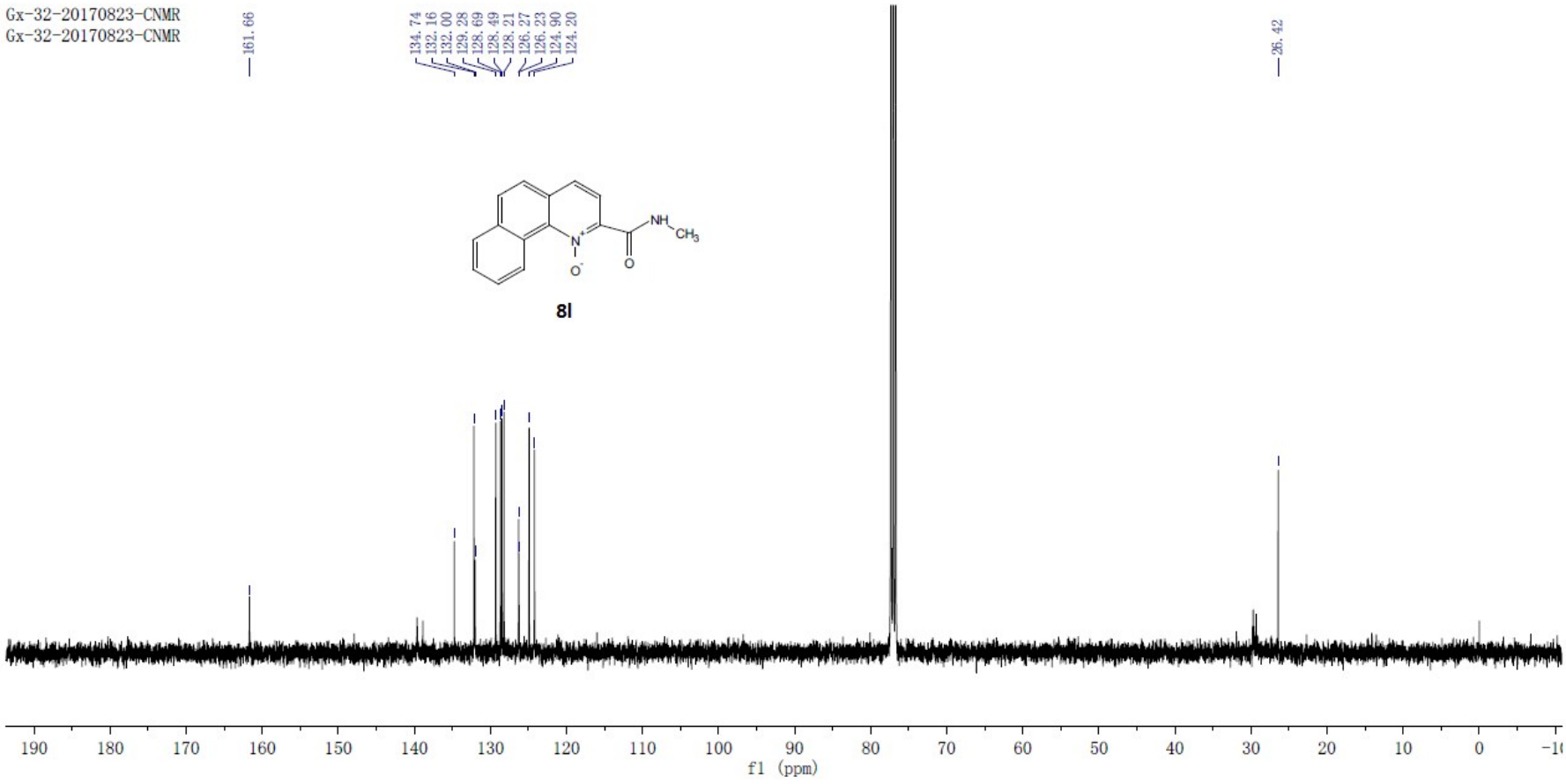

XGX-2073-20171020-HNMR
XGX-2073-20171020-HNMR
11.33
8.83
8.81
8.62
8.50
8.48
8.39
8.37
7.95
7.93
7.28
4.03
3.12
8j
0.91
1.10
1.01
2.08
0.99
3.07
3.00
f1 (ppm)

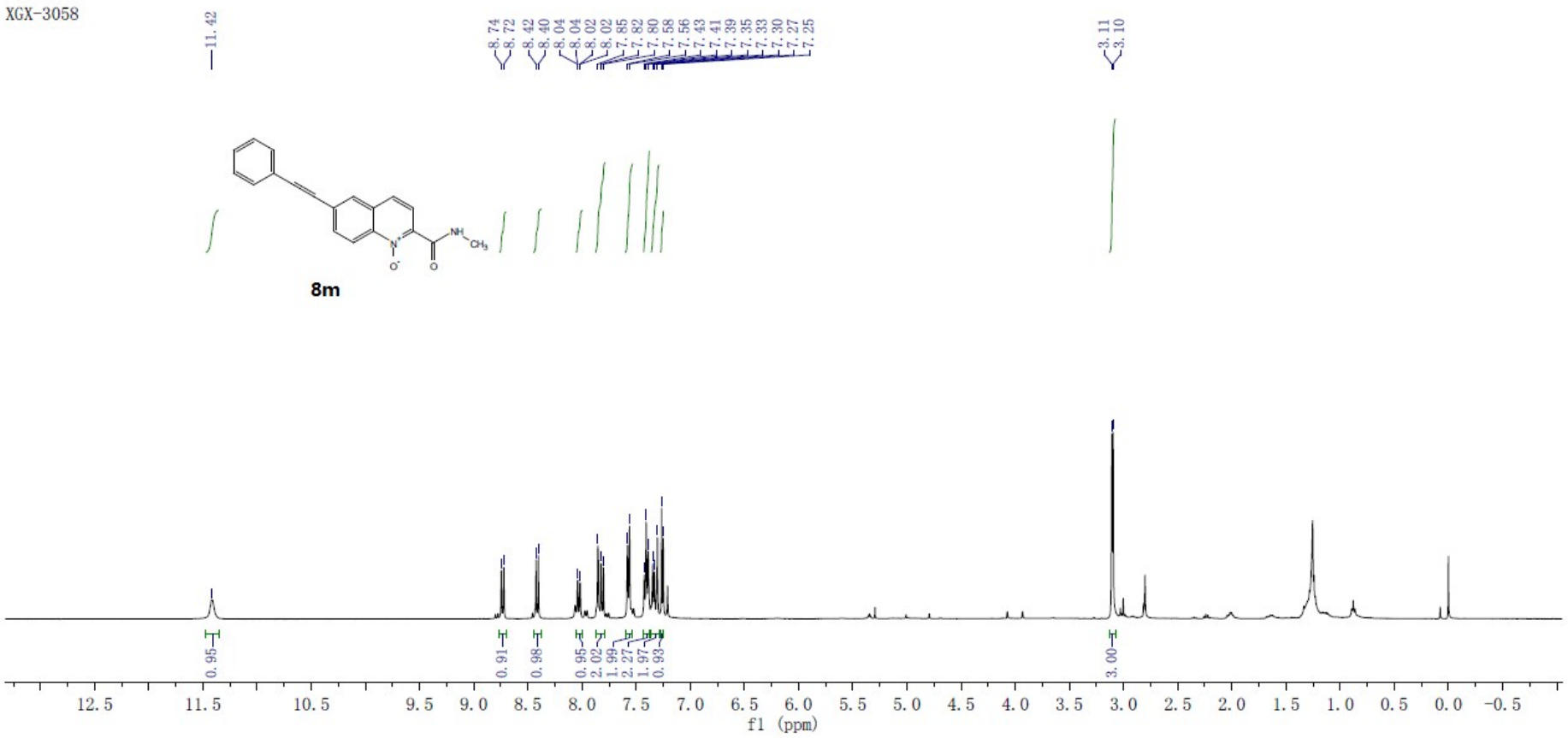

XGX-2073-20171020-CNMR
XGX-2073-20171020-CNMR
165.51
160.76
143.54
138.79
131.33
130.83
130.58
130.18
127.03
123.62
120.78
52.81
26.38
8j
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

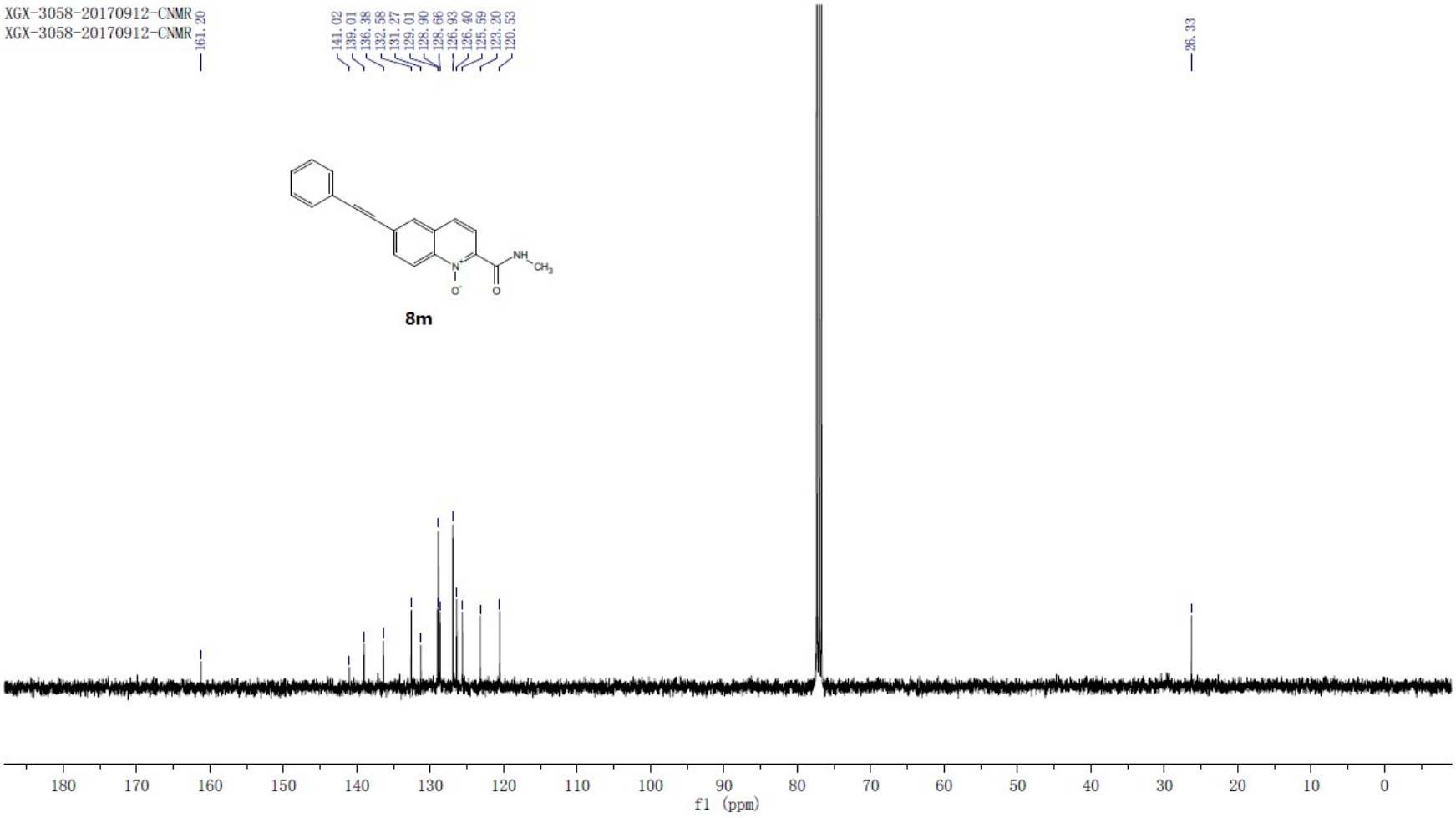

Gw-352
11.47
9.01
8.43
8.41
7.99
7.97
7.96
7.94
7.86
7.84
7.79
7.77
7.54
7.53
7.51
7.47
7.45
7.43
7.27
3.12
3.11
NH
CH3
N+
O-
O
8k
0.98
0.95
1.01
2.00
1.00
2.01
2.02
1.10
3.00
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

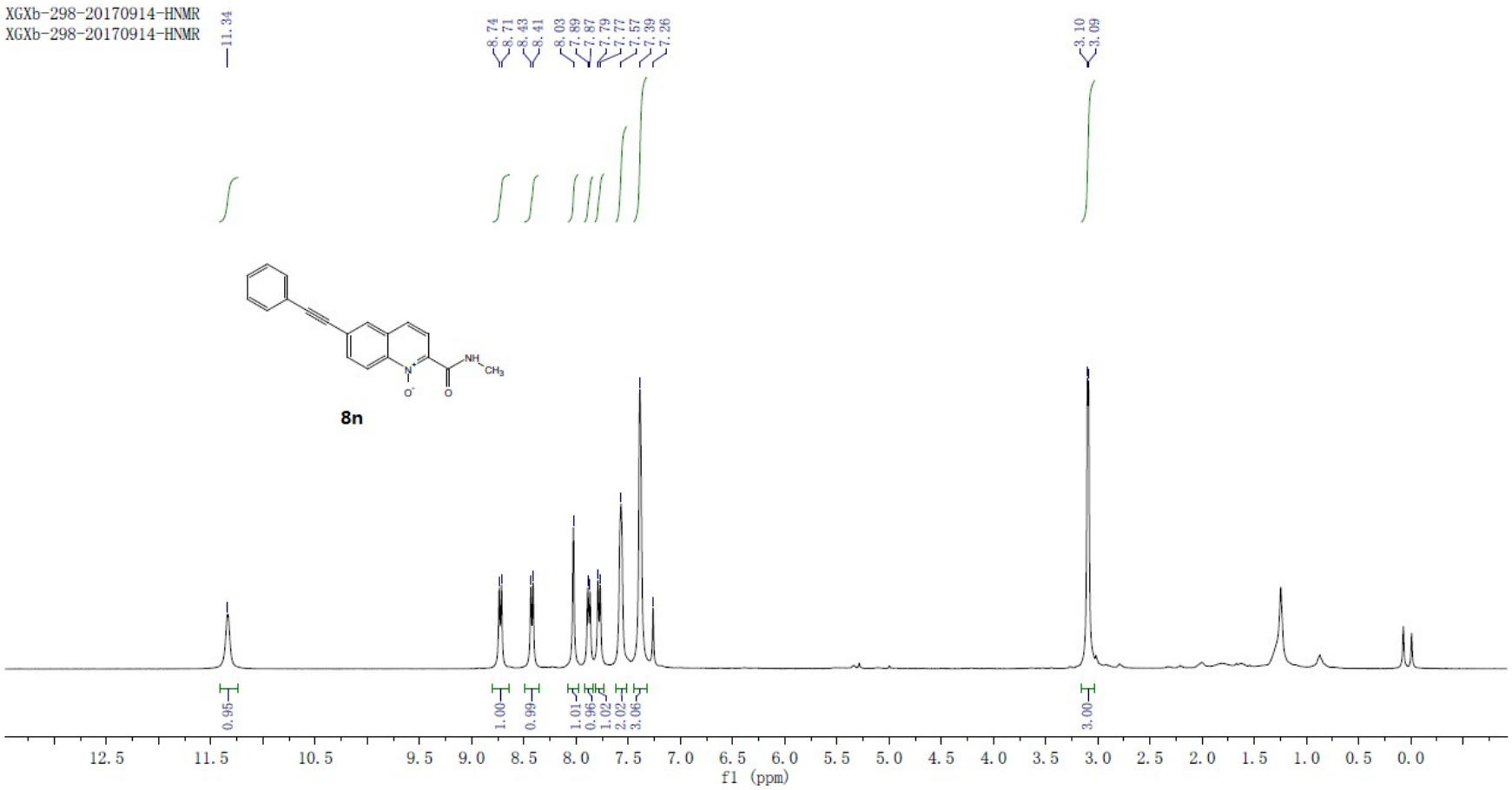

Gw-352-20170823-CNMR
Gw-352-20170823-CNMR
161.20
144.22
142.15
139.22
129.76
129.28
129.19
128.69
127.59
126.16
122.52
117.63
26.33
NH
CH3
N+
O-
O
8k
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

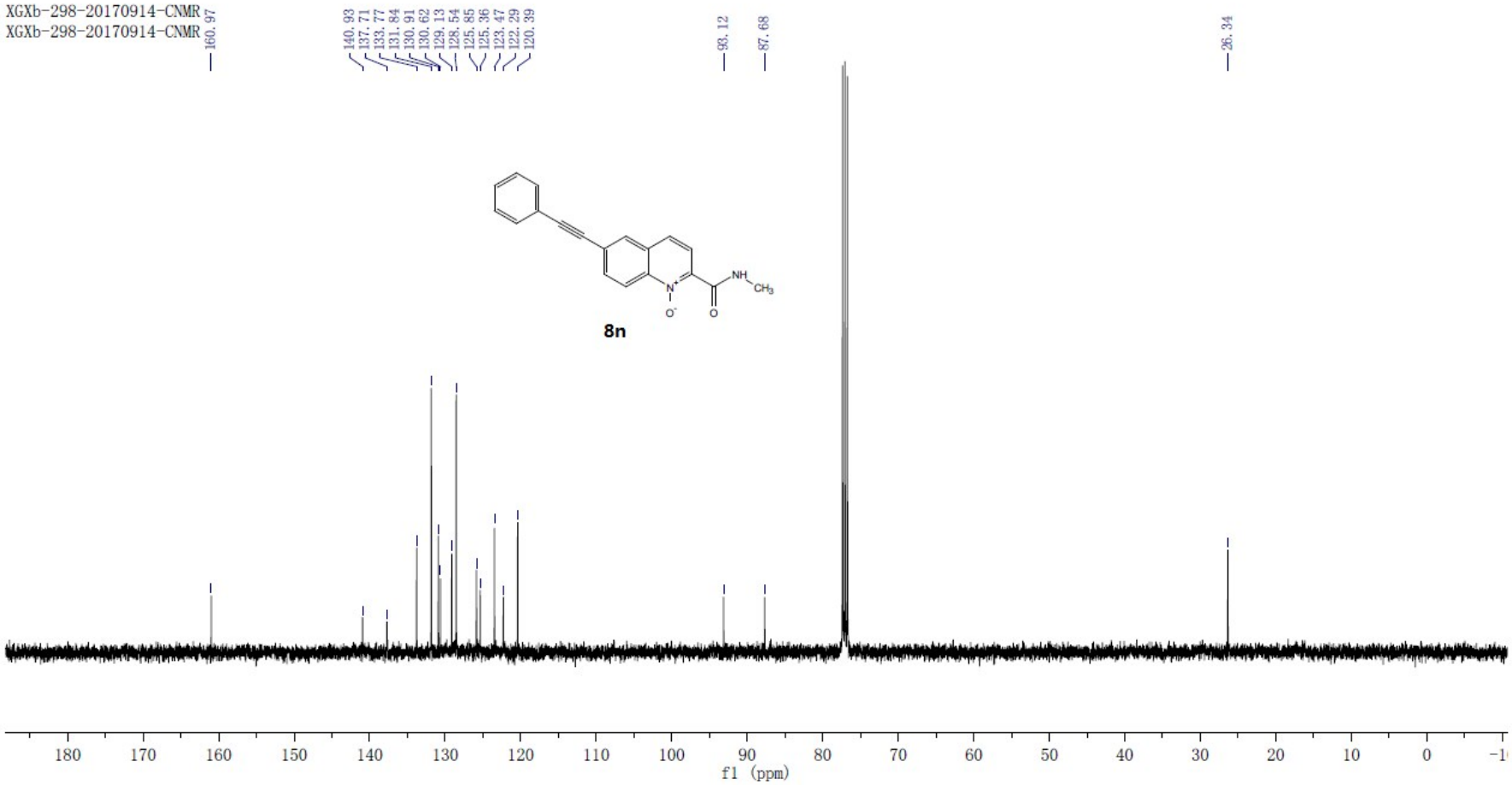

Gx-32-20170823-HNMR
Gx-32-20170823-HNMR
11.49
10.66
10.65
10.64
8.64
8.62
7.94
7.90
7.88
7.82
7.81
7.80
7.70
3.16
3.14
NH
CH3
N+
O-
O
8l
0.94
1.03
1.01
1.02
1.09
1.04
2.06
1.05
3.00
f1 (ppm)

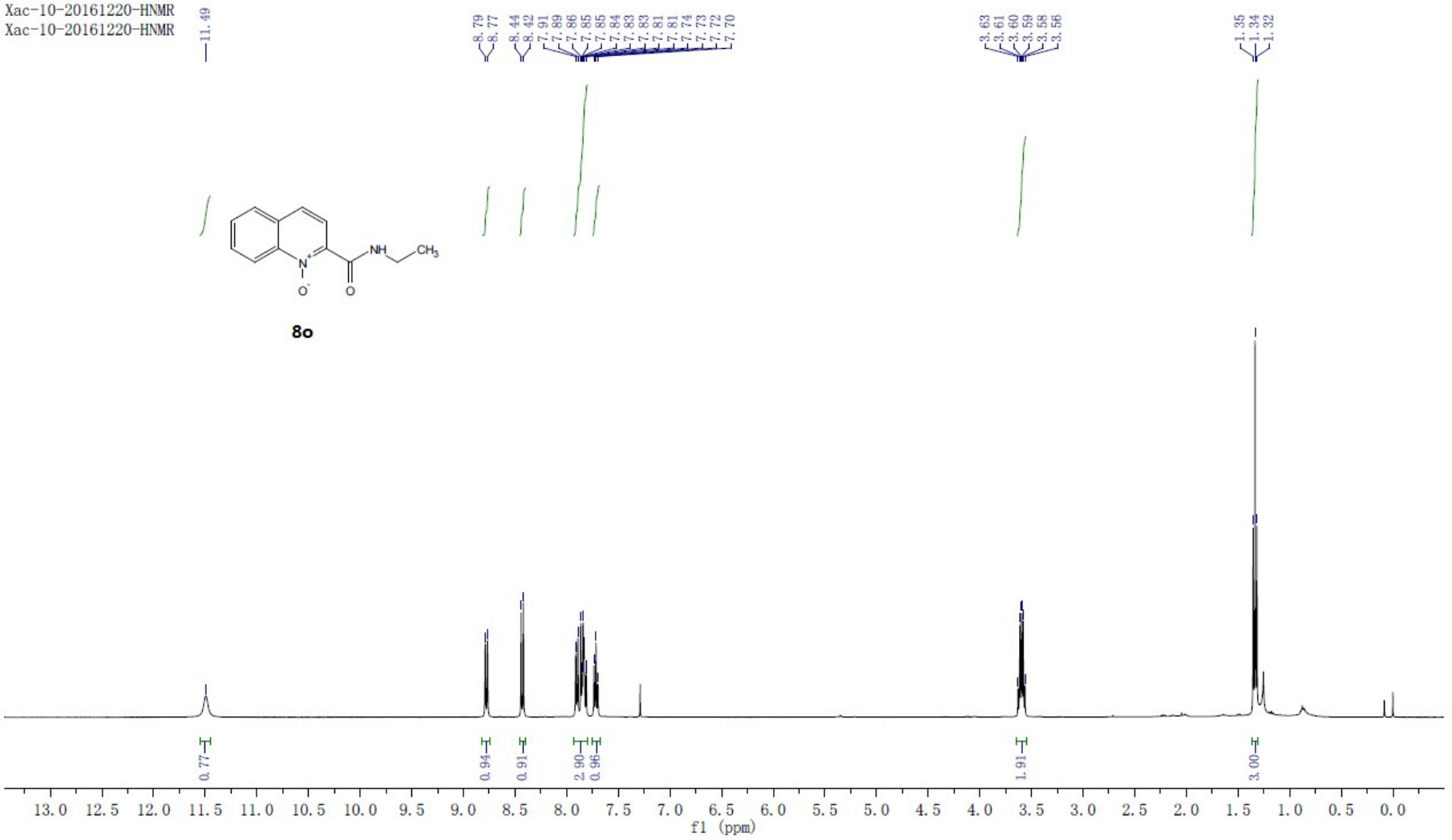

Gx-32-20170823-CNMR
Gx-32-20170823-CNMR
161.66
134.74
132.16
132.00
129.28
128.69
128.49
128.21
126.27
126.23
124.90
124.20
26.42
NH
CH3
N+
O-
O
8l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

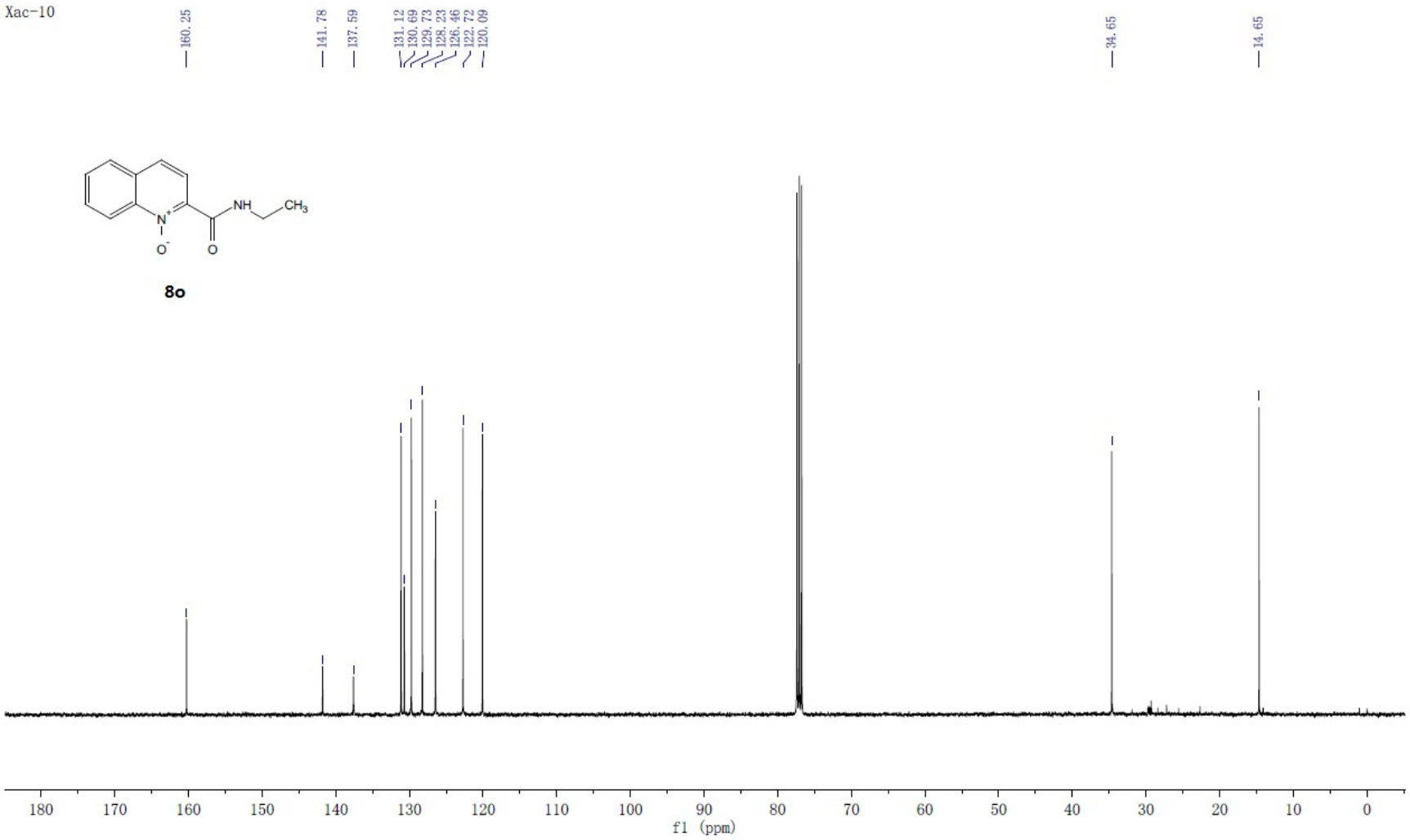

XGX-3058
11.42
8.74
8.72
8.42
8.40
8.04
8.04
8.02
8.02
7.85
7.82
7.80
7.58
7.56
7.43
7.41
7.39
7.35
7.33
7.30
7.27
7.25
3.11
3.10
8m
0.95
0.91
0.98
0.95
2.02
1.99
2.27
1.97
0.93
3.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

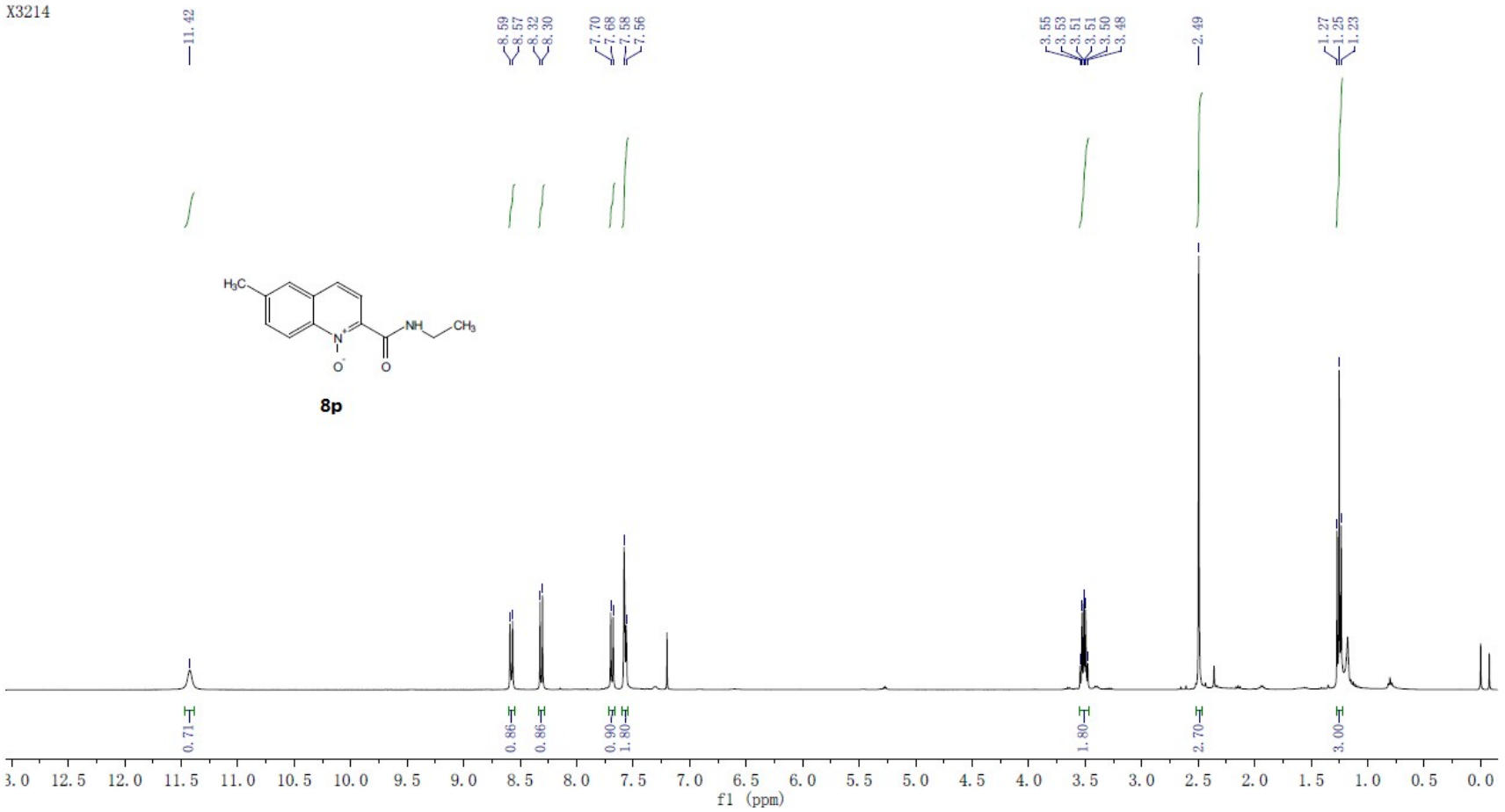

XGX-3058-20170912-CNMR
XGX-3058-20170912-CNMR
161.20
141.02
139.01
136.38
132.58
131.27
129.01
128.90
128.66
126.93
126.40
125.59
123.20
120.53
26.33
NH
CH3
N
O-
O
8m
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

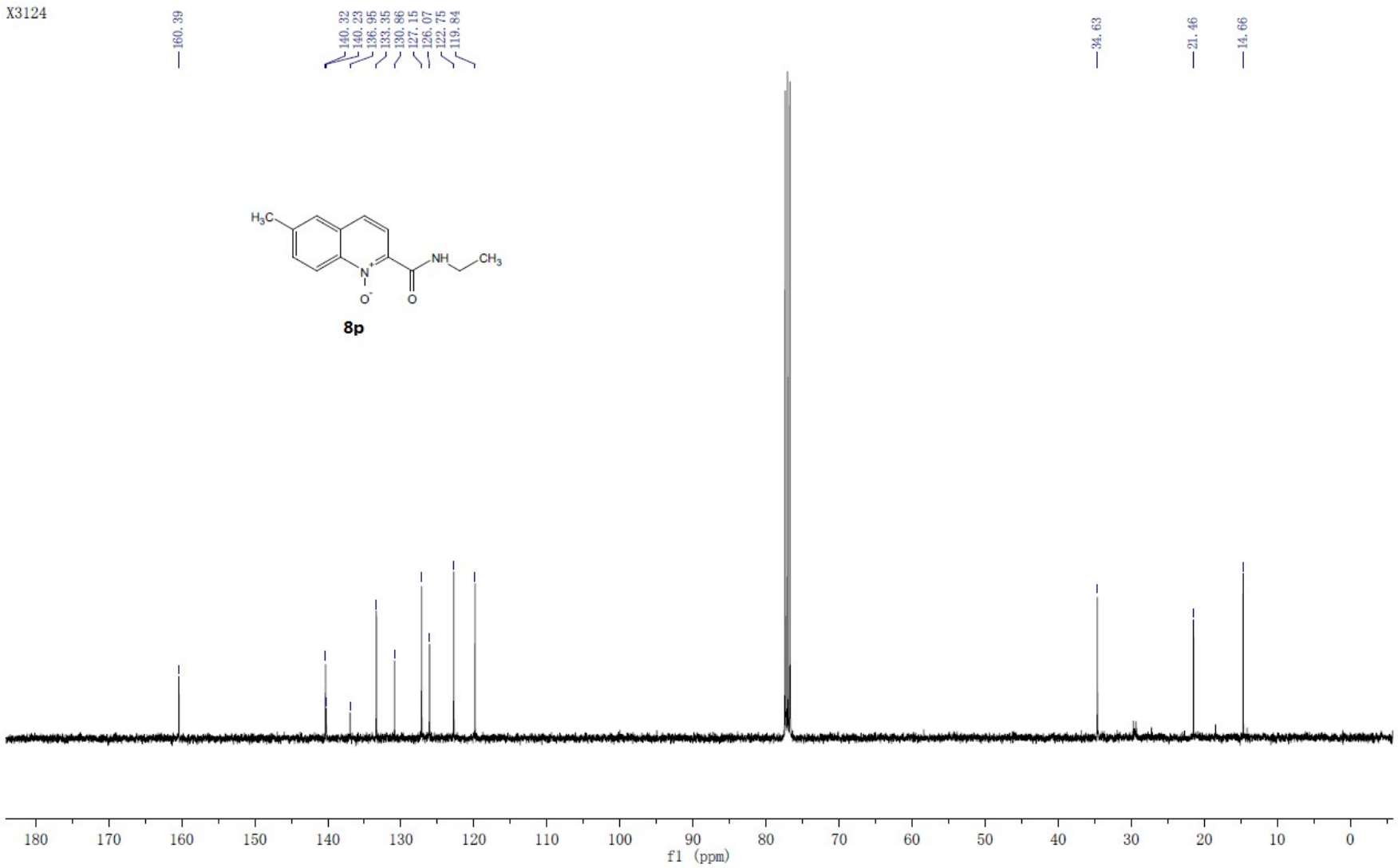

XGXb-298-20170914-HNMR
XGXb-298-20170914-HNMR
11.34
8.74
8.71
8.43
8.41
8.03
7.89
7.87
7.79
7.77
7.57
7.39
7.26
3.10
3.09
8n
0.95
1.00
0.99
1.01
0.96
1.02
2.02
3.06
3.00
f1 (ppm)

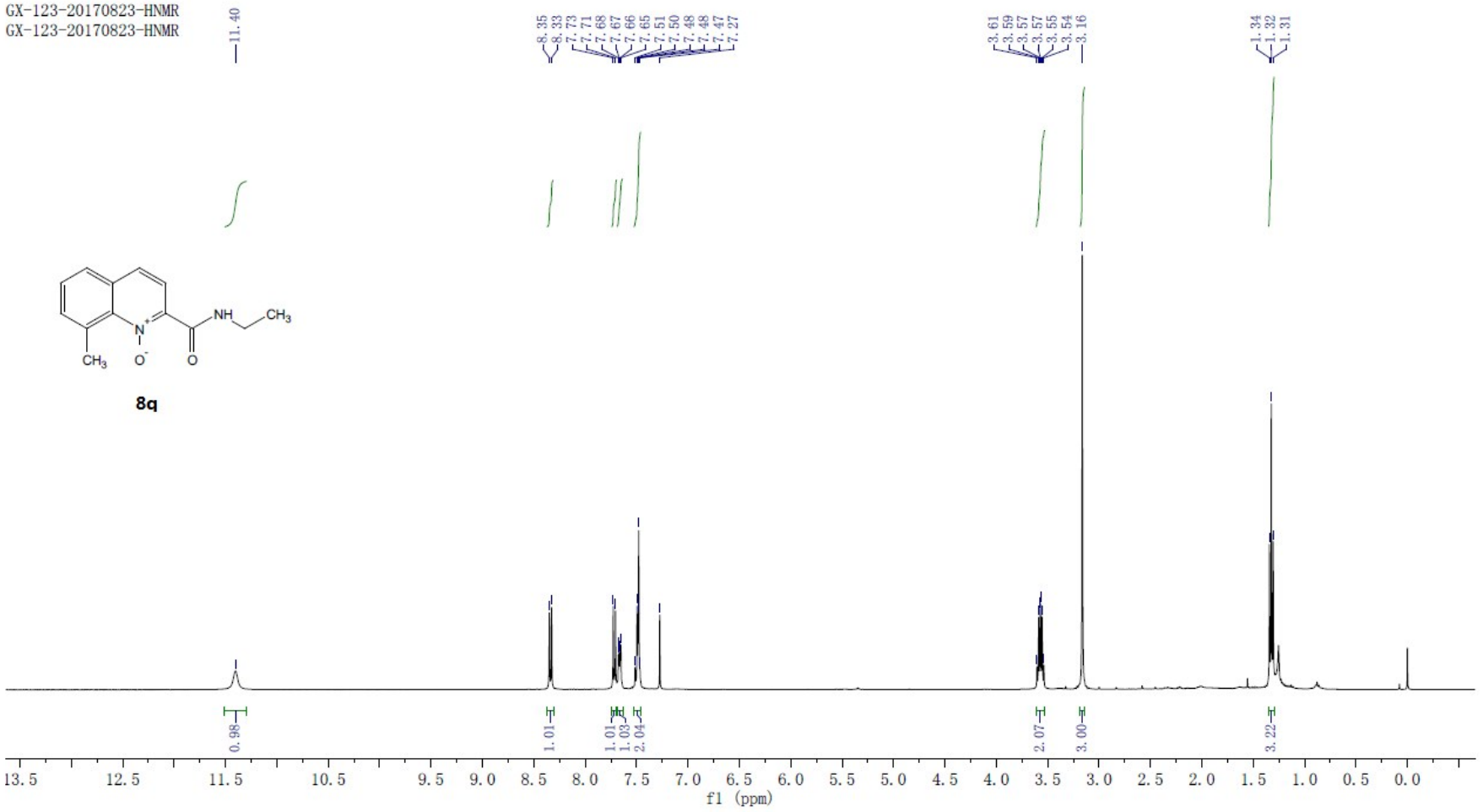

XGXb-298-20170914-CNMR
XGXb-298-20170914-CNMR
160.97
140.93
137.71
133.77
131.84
130.91
130.62
129.13
128.54
125.85
125.36
123.47
122.29
120.39
93.12
87.68
26.34
NH
CH3
N+
O-
O
8n
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

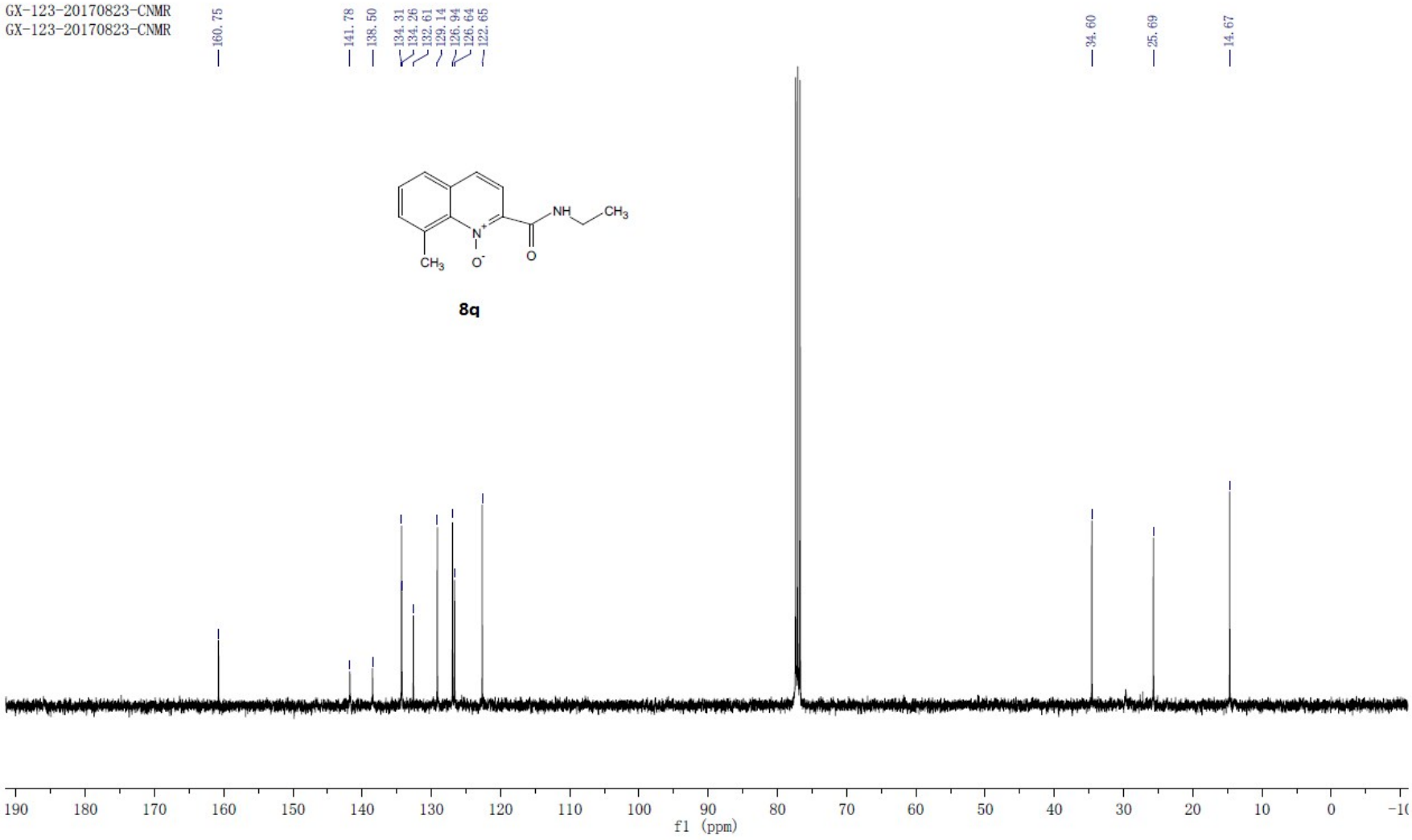

Xac-10-20161220-HNMR
Xac-10-20161220-HNMR
11.49
8.79
8.77
8.44
8.42
7.91
7.89
7.86
7.85
7.85
7.84
7.83
7.83
7.81
7.81
7.74
7.73
7.72
7.70
3.63
3.61
3.60
3.59
3.58
3.56
1.35
1.34
1.32
NH
CH3
N+
O-
O
8o
0.77
0.94
0.91
2.90
0.96
1.91
3.00
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

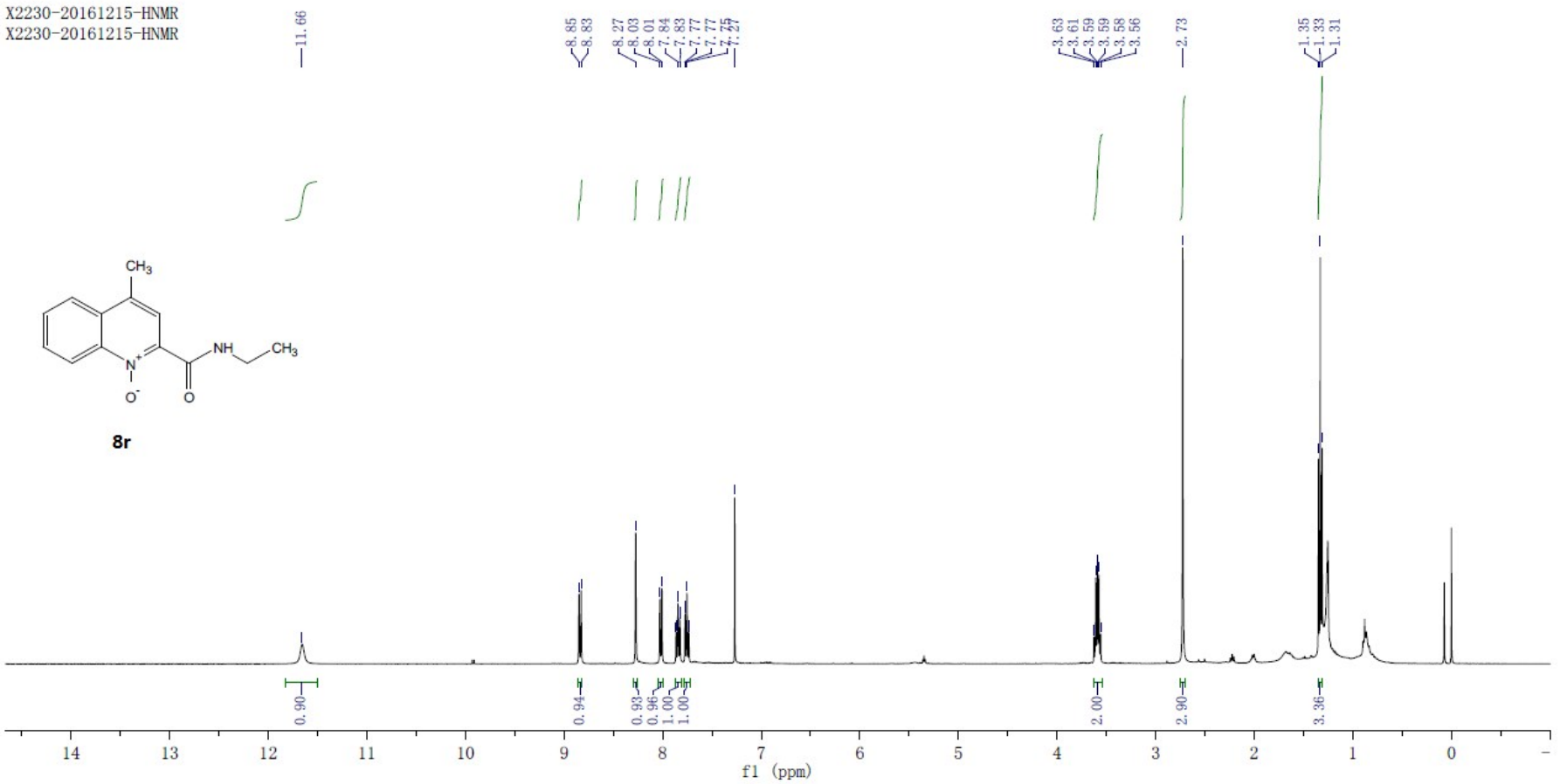

Xac-10
160.25
141.78
137.59
131.12
130.69
129.73
128.23
126.46
122.72
120.09
34.65
14.65
NH
CH3
N+
O-
O
8o
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

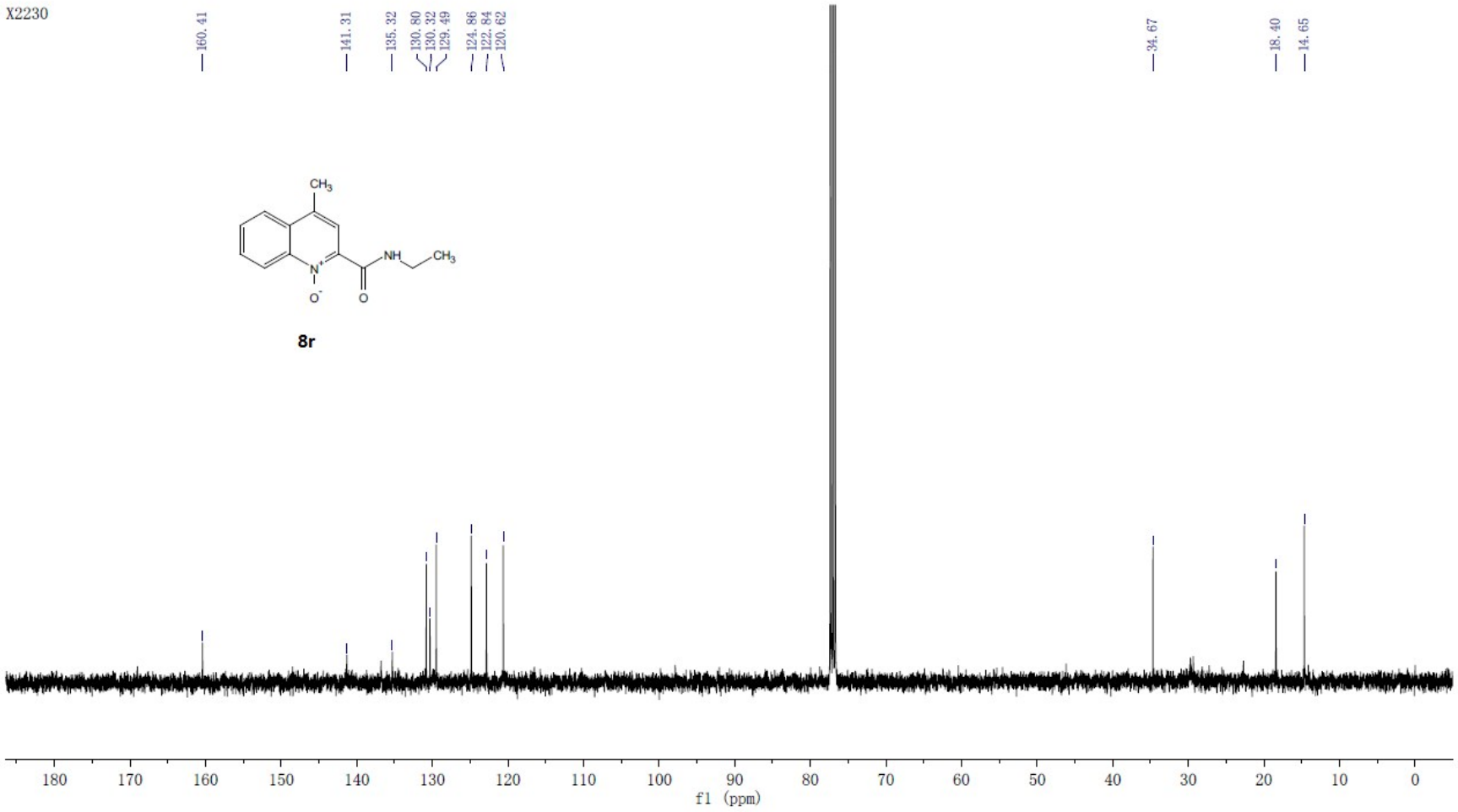

X3214
11.42
8.59
8.57
8.32
8.30
7.70
7.68
7.58
7.56
3.55
3.53
3.51
3.51
3.50
3.48
2.49
1.27
1.25
1.23
H3C
N+
O-
NH
CH3
O
8p
0.71
0.86
0.86
0.90
1.80
1.80
2.70
3.00
f1 (ppm)

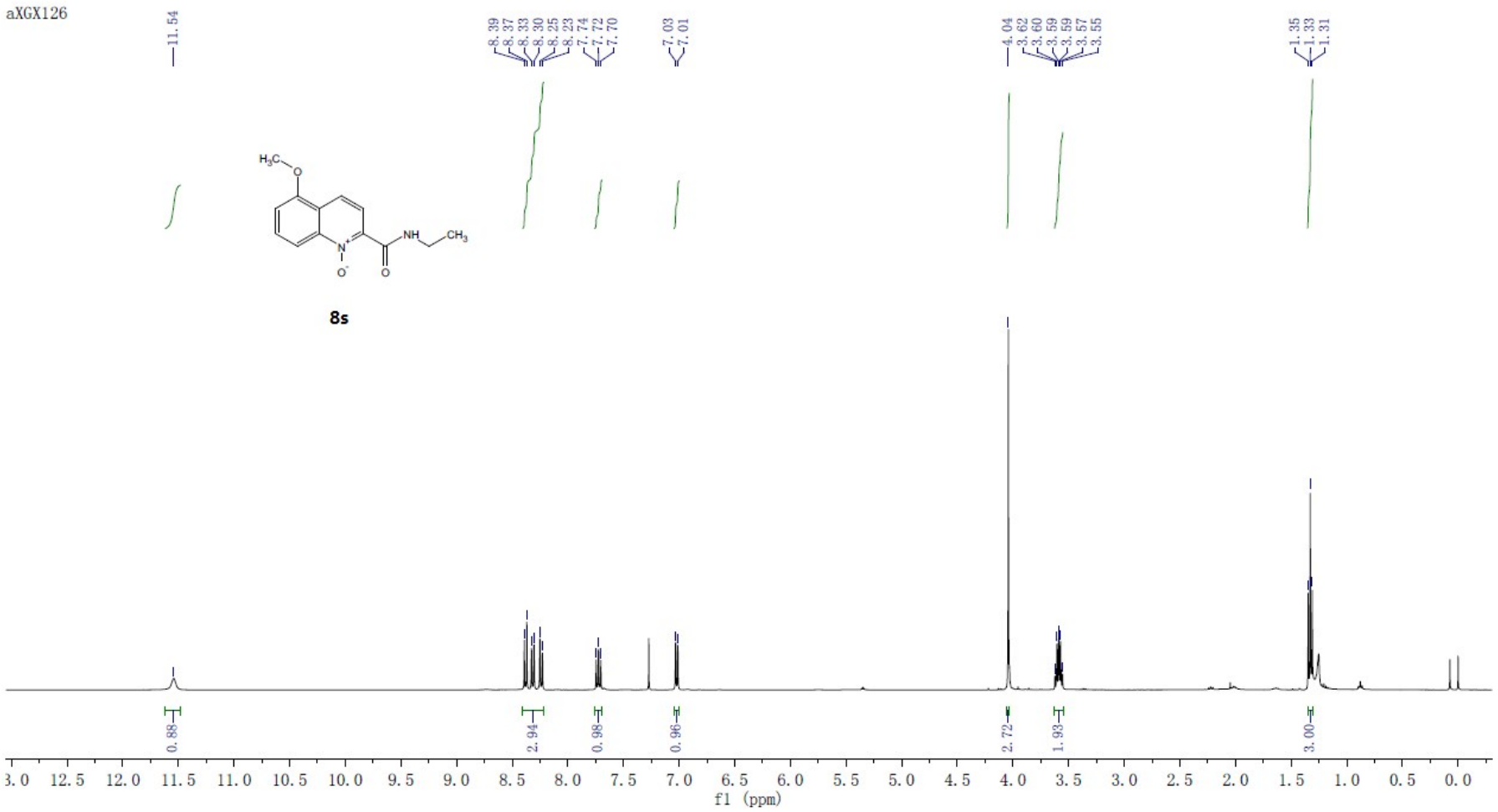

X3124
160.39
140.32
140.23
136.95
133.35
130.86
127.15
126.07
122.75
119.84
34.63
21.46
14.66
H3C
N+
O-
NH
CH3
O
8p
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

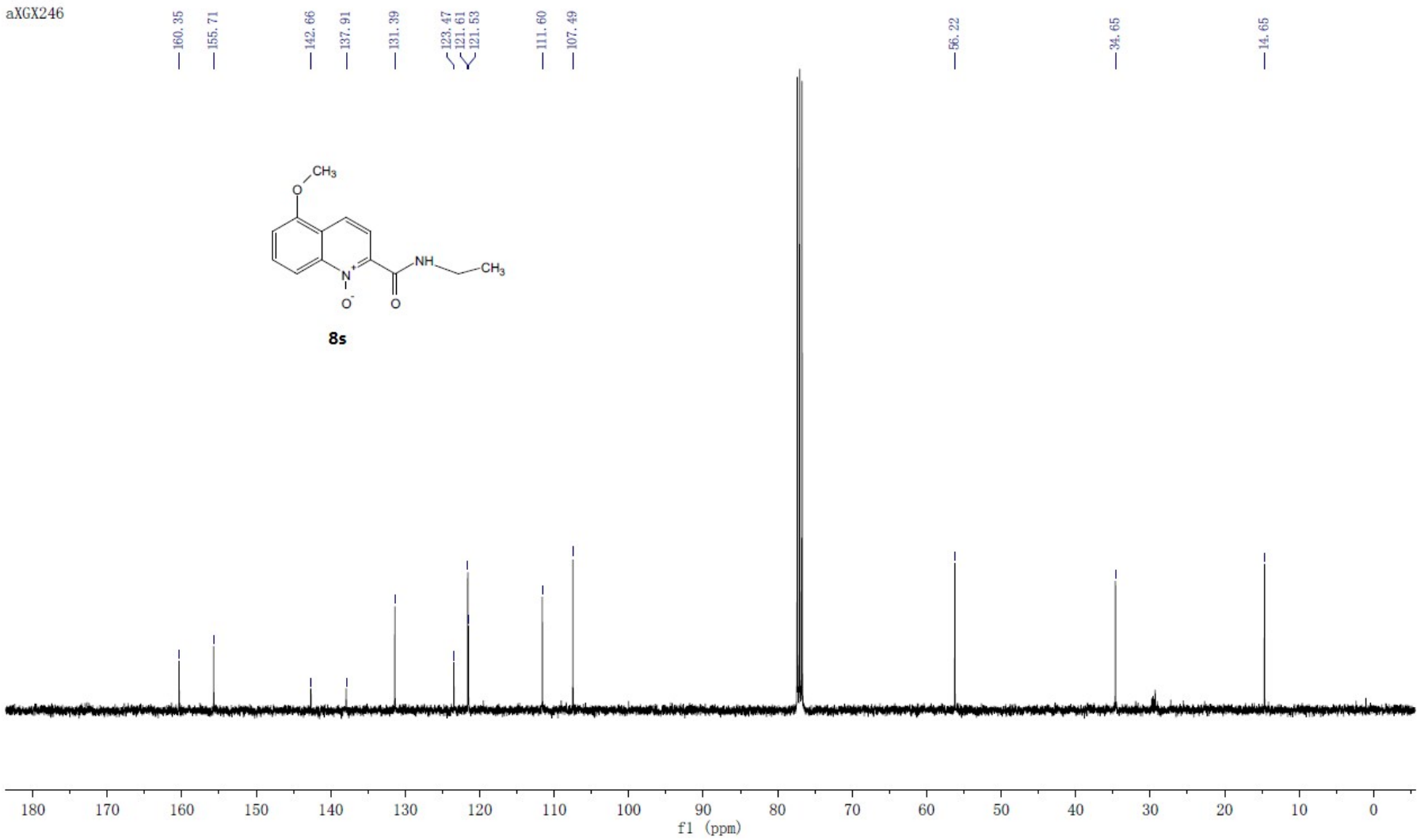

GX-123-20170823-HNMR
GX-123-20170823-HNMR
11.40
8.35
8.33
7.73
7.71
7.68
7.67
7.66
7.65
7.51
7.50
7.48
7.48
7.47
7.27
3.61
3.59
3.57
3.57
3.55
3.54
3.16
1.34
1.32
1.31
NH
CH3
N+
O-
CH3
O
8q
0.98
1.01
1.01
1.03
2.04
2.07
3.00
3.22
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

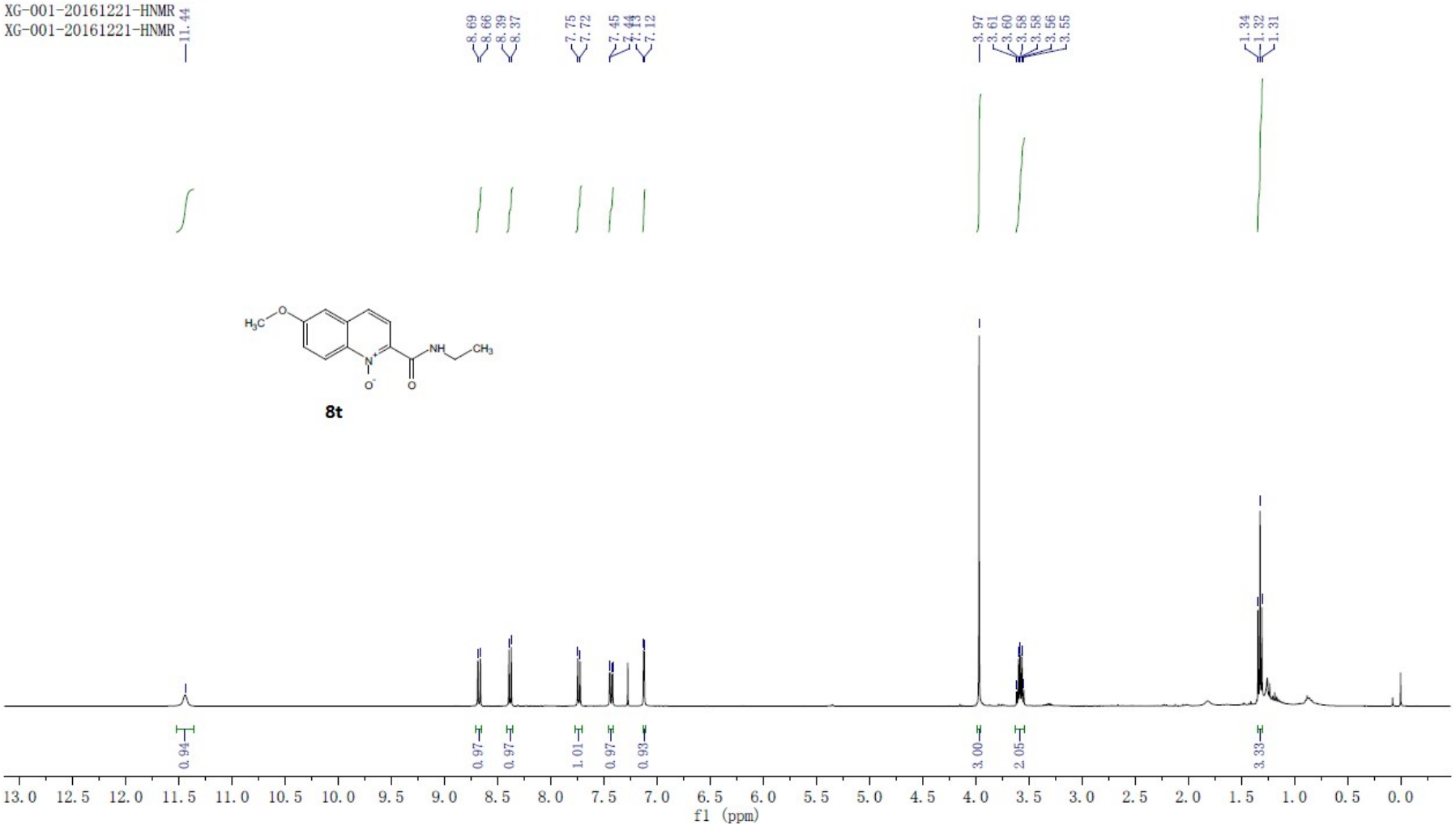

GX-123-20170823-CNMR
GX-123-20170823-CNMR
160.75
141.78
138.50
134.31
134.26
132.61
129.14
126.94
126.64
122.65
34.60
25.69
14.67
NH
CH3
N+
O-
CH3
O
8q
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

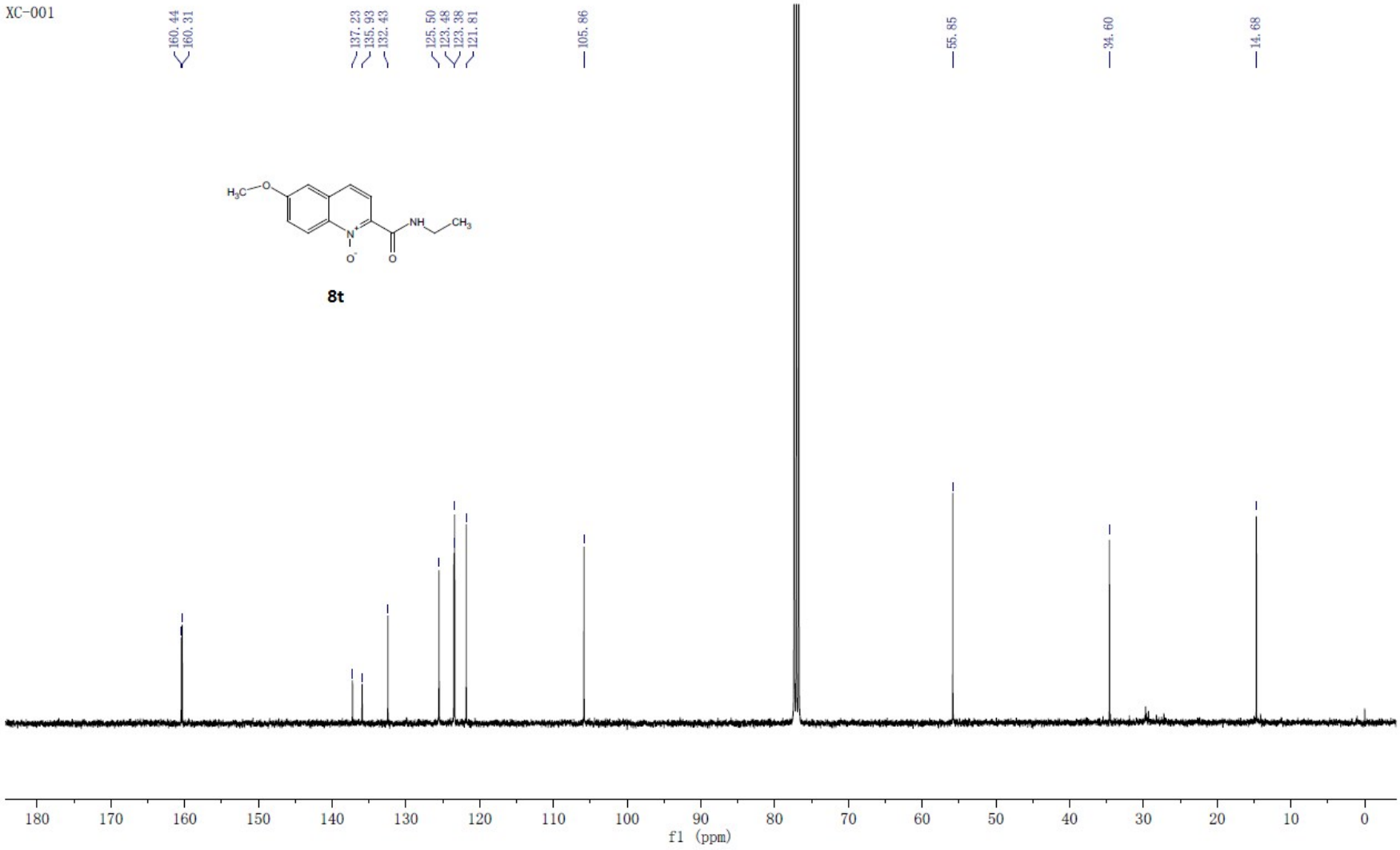

X2230-20161215-HNMR
X2230-20161215-HNMR
11.66
8.85
8.83
8.27
8.03
8.01
7.84
7.83
7.77
7.77
7.75
7.27
3.63
3.61
3.59
3.59
3.58
3.56
2.73
1.35
1.33
1.31
CH3
NH
CH3
N+
O-
O
8r
0.90
0.94
0.93
0.96
1.00
1.00
2.00
2.90
3.36
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

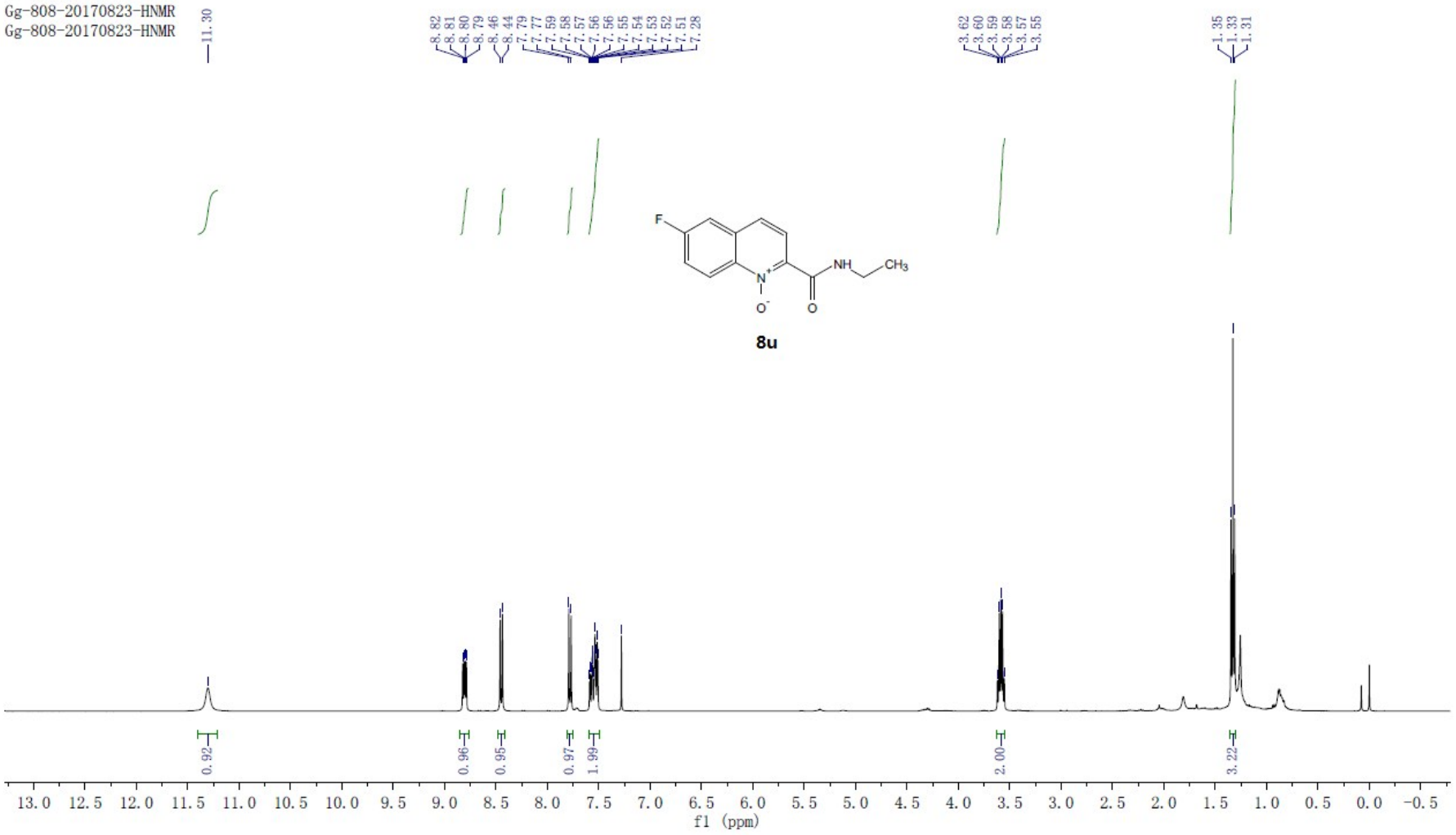

X2230
160.41
141.31
135.32
130.80
130.32
129.49
124.86
122.84
120.62
34.67
18.40
14.65
CH3
NH
CH3
N+
O-
O
8r
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

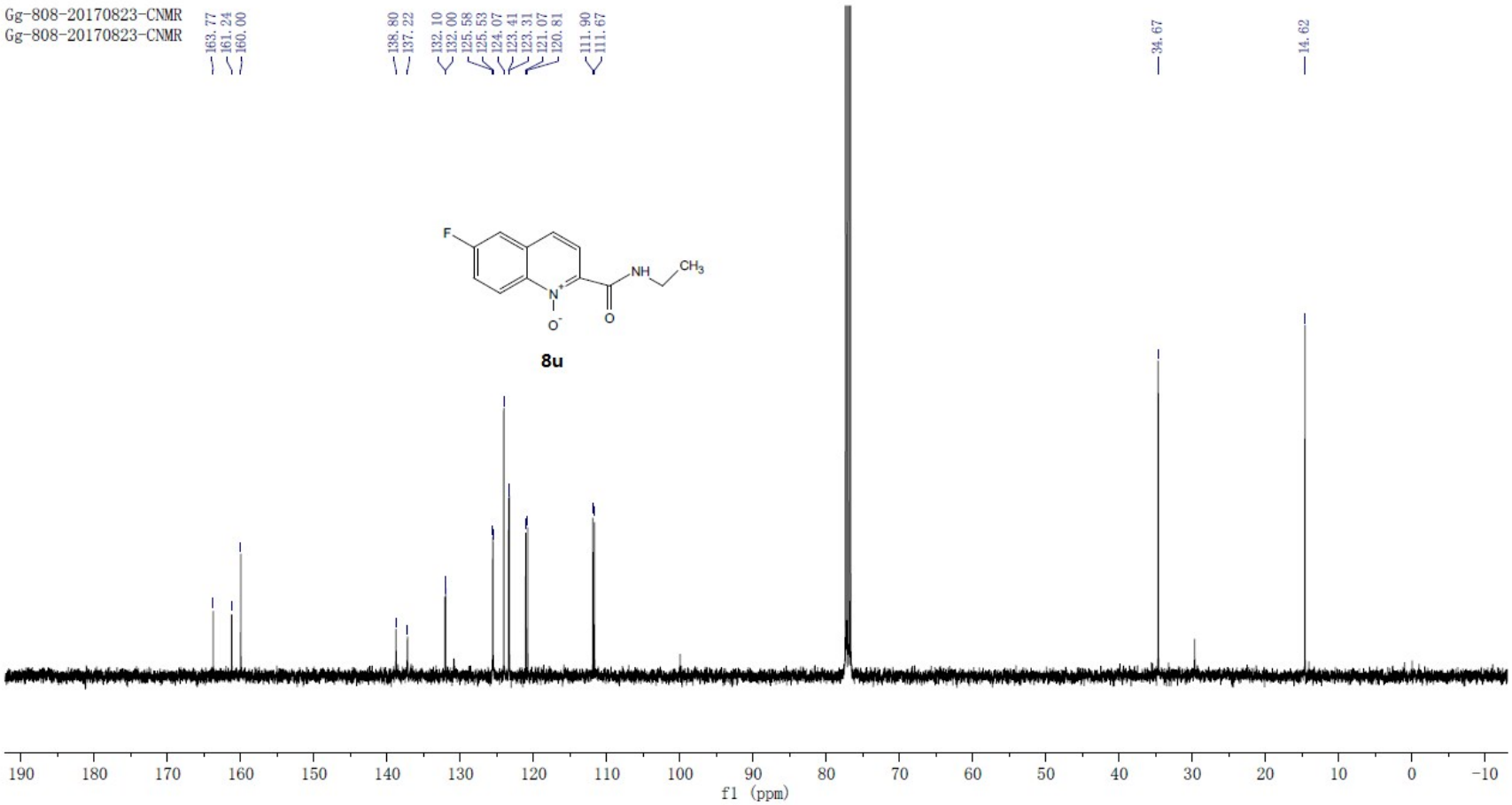

aXGX126
11.54
8.39
8.37
8.33
8.30
8.25
8.23
7.74
7.72
7.70
7.03
7.01
4.04
3.62
3.60
3.59
3.59
3.57
3.55
1.35
1.33
1.31
8s
0.88
2.94
0.98
0.96
2.72
1.93
3.00
f1 (ppm)

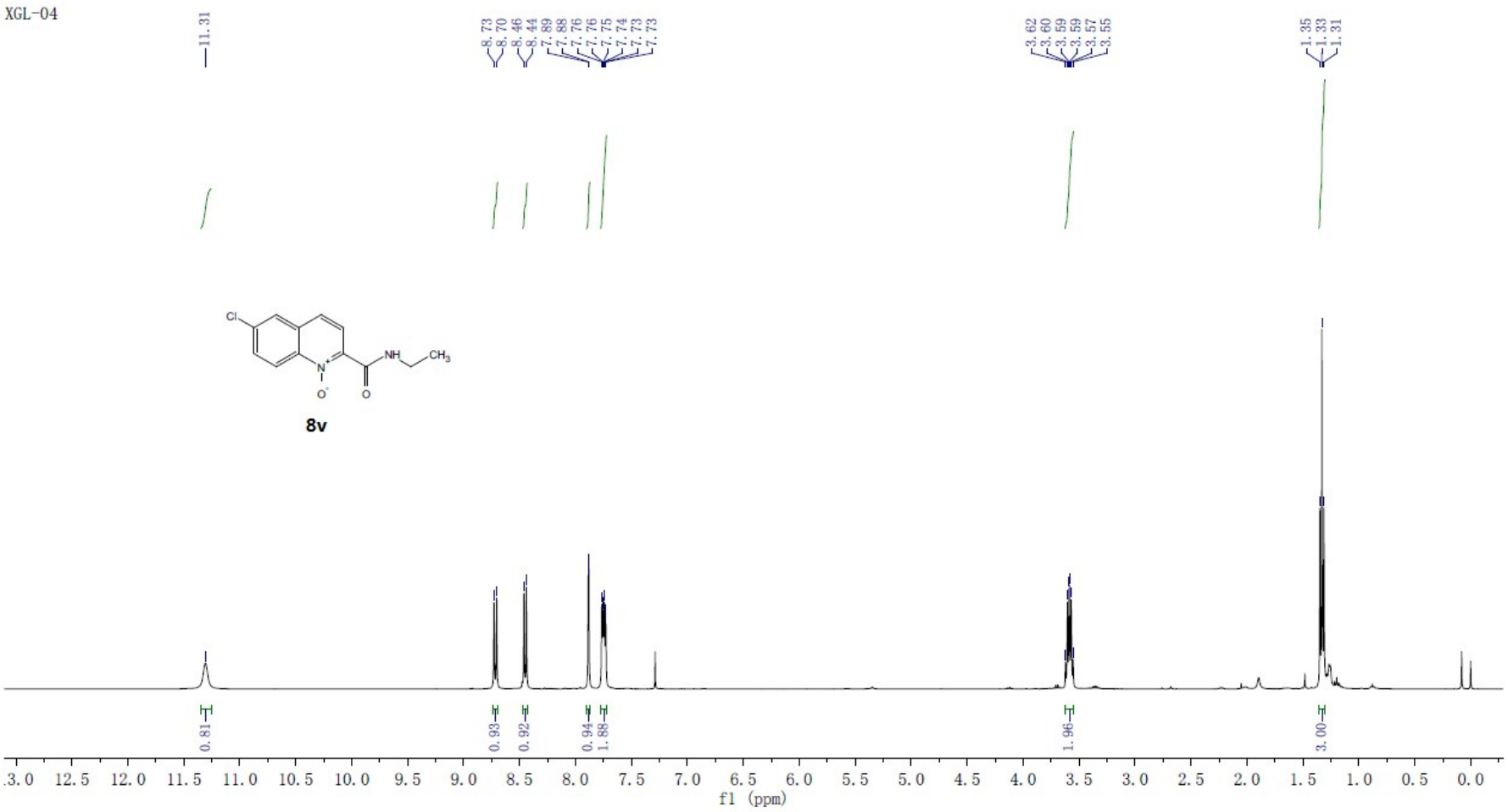

aXGX246
160.35
155.71
142.66
137.91
131.39
123.47
121.61
121.53
111.60
107.49
56.22
34.65
14.65
CH3
O
NH
CH3
N+
O-
O
8s
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


XG-001-20161221-HNMR
XG-001-20161221-HNMR
11.44
8.69
8.66
8.39
8.37
7.75
7.72
7.45
7.44
7.13
7.12
3.97
3.61
3.60
3.58
3.58
3.56
3.55
1.34
1.32
1.31
8t
0.94
0.97
0.97
1.01
0.97
0.93
3.00
2.05
3.33
f1 (ppm)

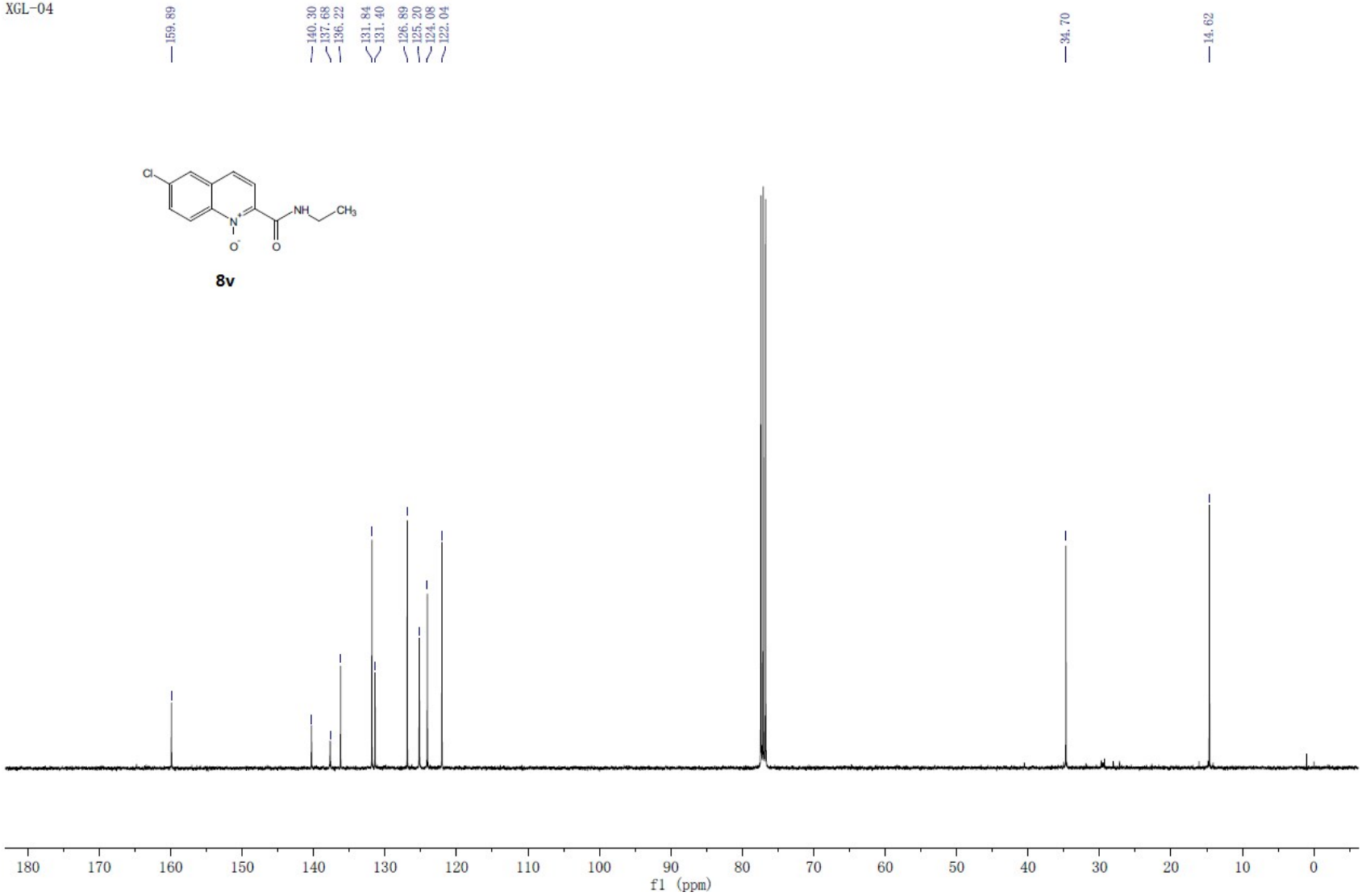

XC-001
160.44
160.31
137.23
135.93
132.43
125.50
123.48
123.38
121.81
105.86
55.85
34.60
14.68
8t
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Gg-808-20170823-HNMR
Gg-808-20170823-HNMR
11.30
8.82
8.81
8.80
8.79
8.46
8.44
7.79
7.77
7.59
7.58
7.57
7.56
7.56
7.55
7.54
7.53
7.52
7.51
7.28
3.62
3.60
3.59
3.58
3.57
3.55
1.35
1.33
1.31
F
N+
O-
NH
CH3
O
8u
0.92
0.96
0.95
0.97
1.99
2.00
3.22
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

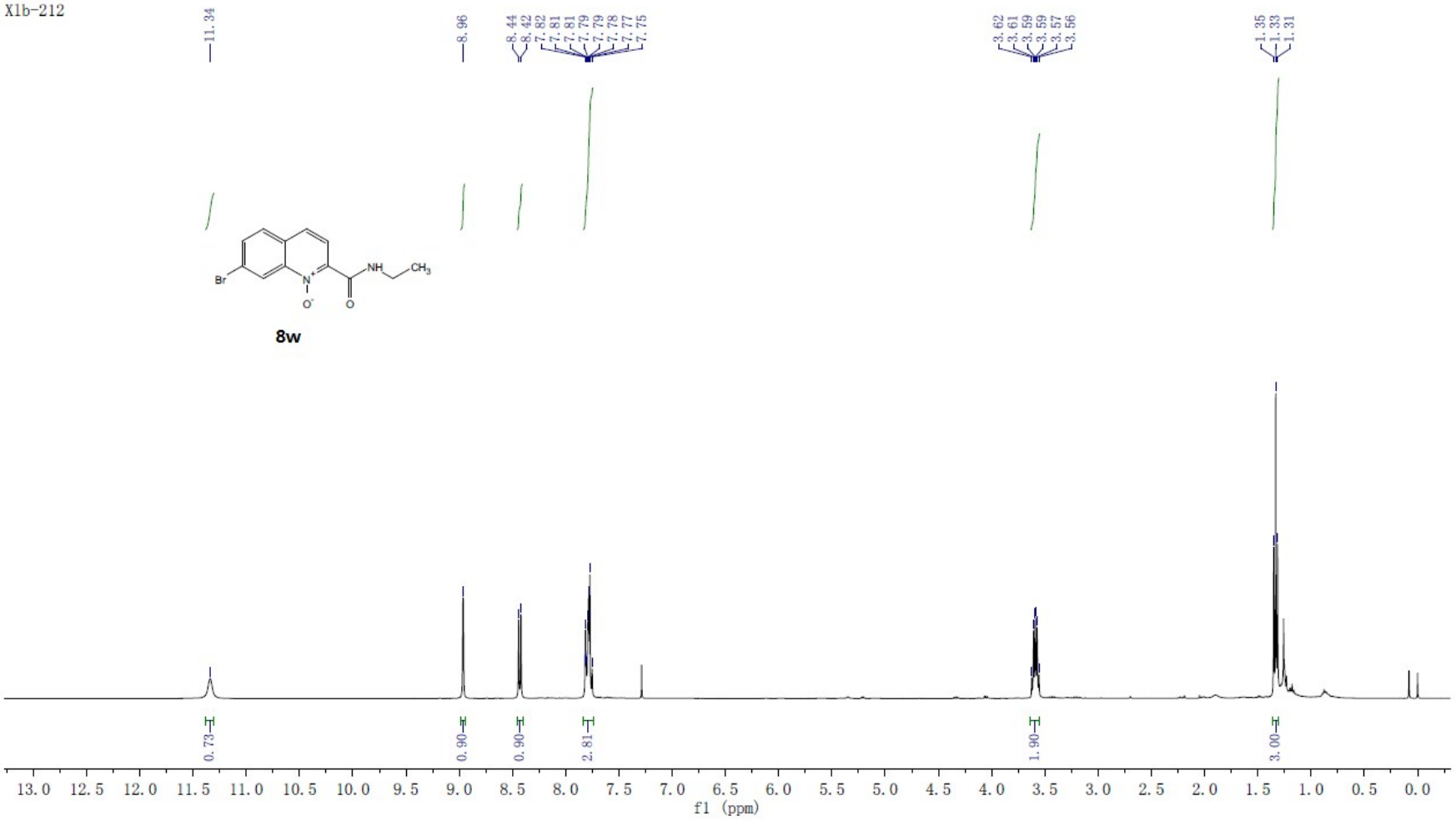

Gg-808-20170823-CNMR
Gg-808-20170823-CNMR
163.77
161.24
160.00
138.80
137.22
132.10
132.00
125.58
125.53
124.07
123.41
123.31
121.07
120.81
111.90
111.67
34.67
14.62
F
NH
CH3
N+
O-
O
8u
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

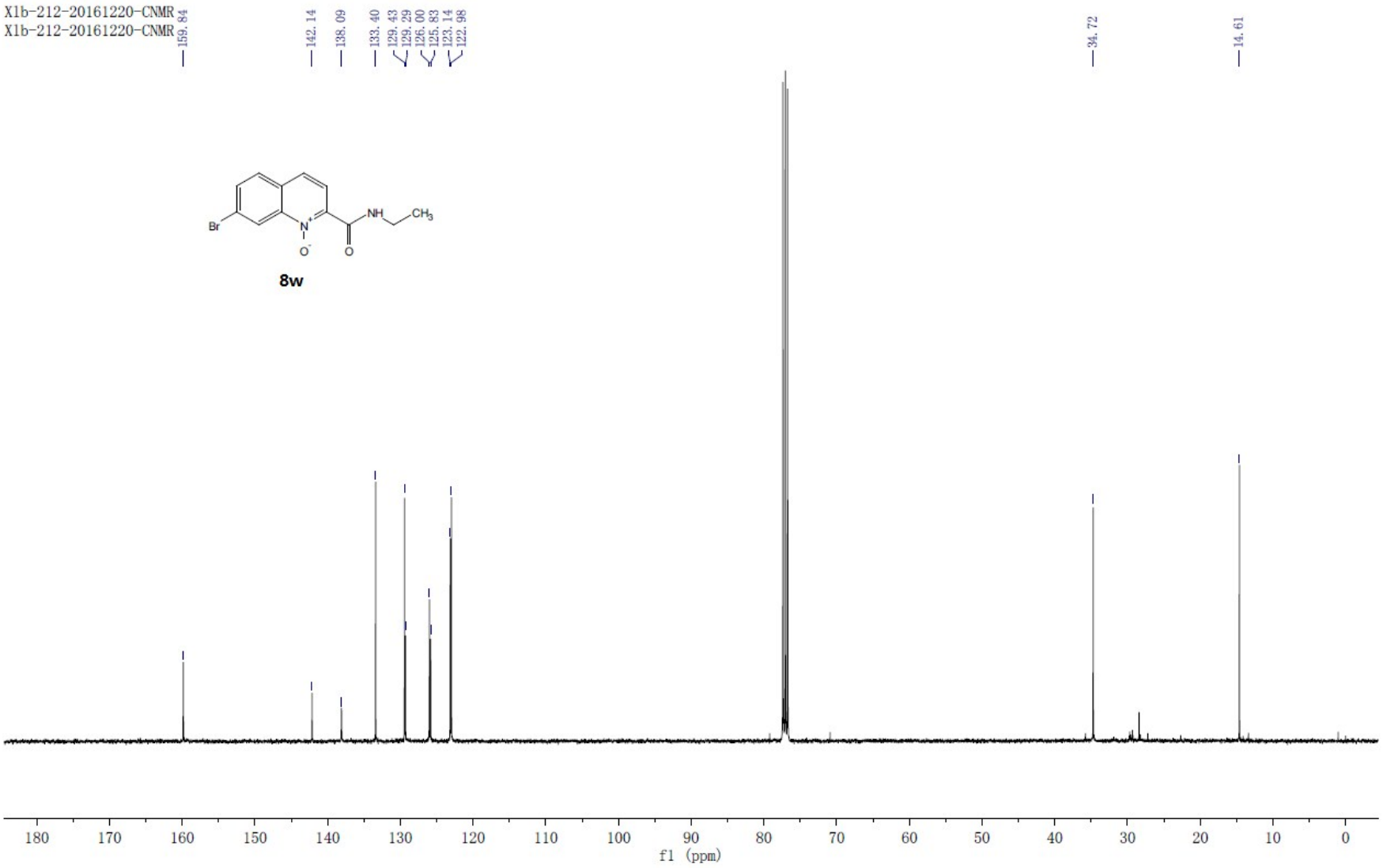

XGL-04
11.31
8.73
8.70
8.46
8.44
7.89
7.88
7.76
7.76
7.75
7.74
7.73
7.73
3.62
3.60
3.59
3.59
3.57
3.55
1.35
1.33
1.31
Cl
NH
CH3
O
O
8v
0.81
0.93
0.92
0.94
1.88
1.96
3.00
f1 (ppm)

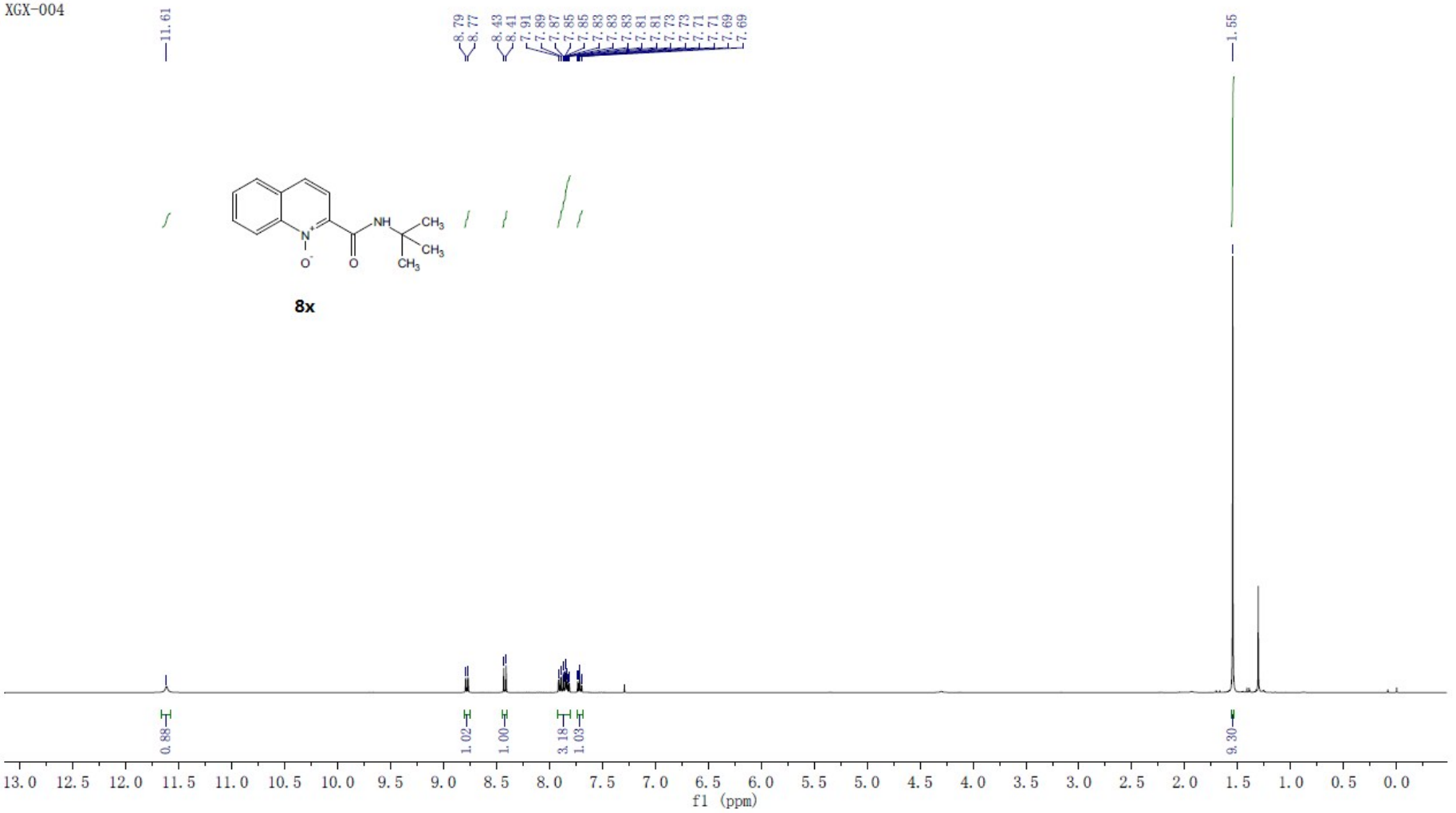
XGL-04

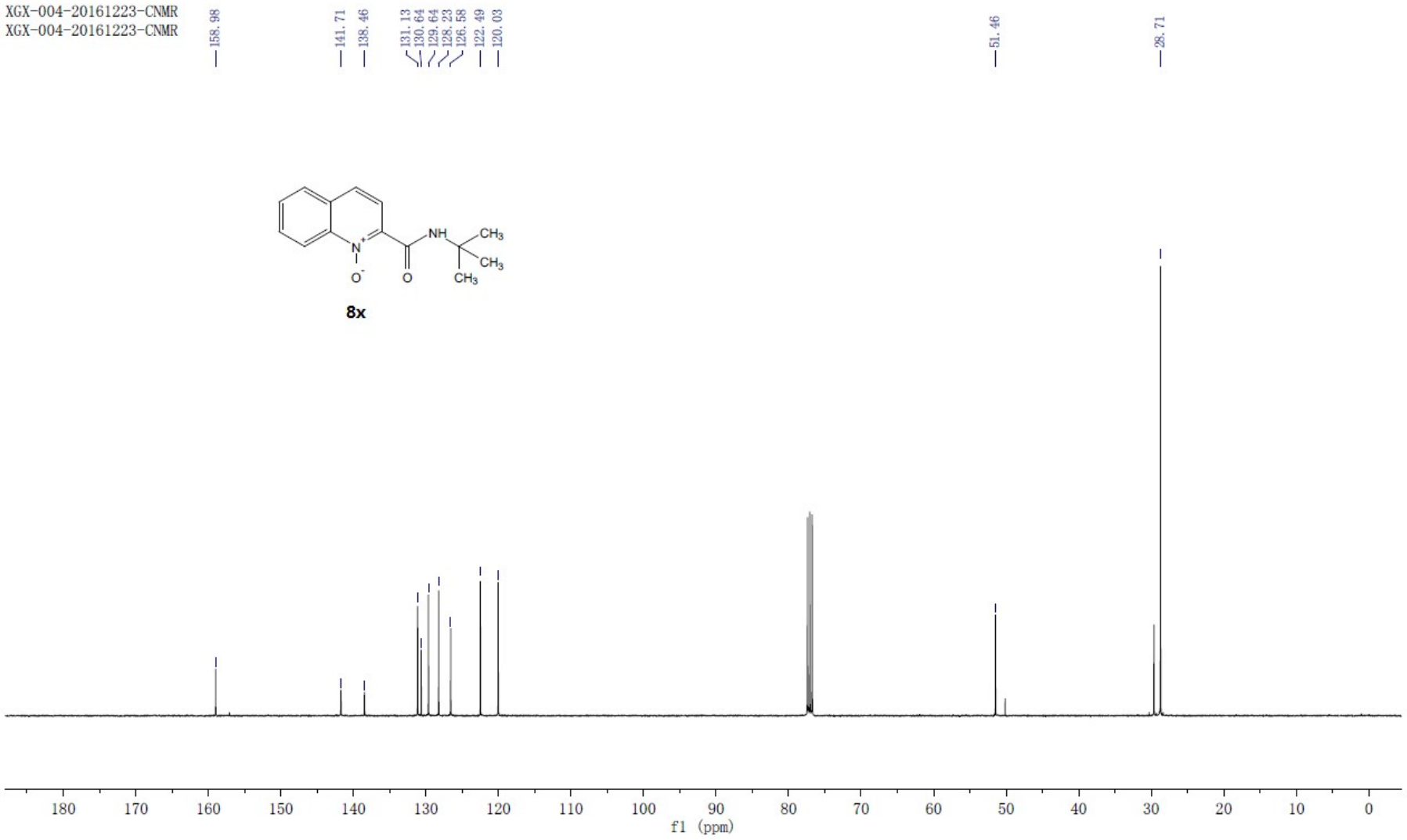

159.89
140.30
137.68
136.22
131.84
131.40
126.89
125.20
124.08
122.04
34.70
14.62
Cl
N+
O-
NH
CH3
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

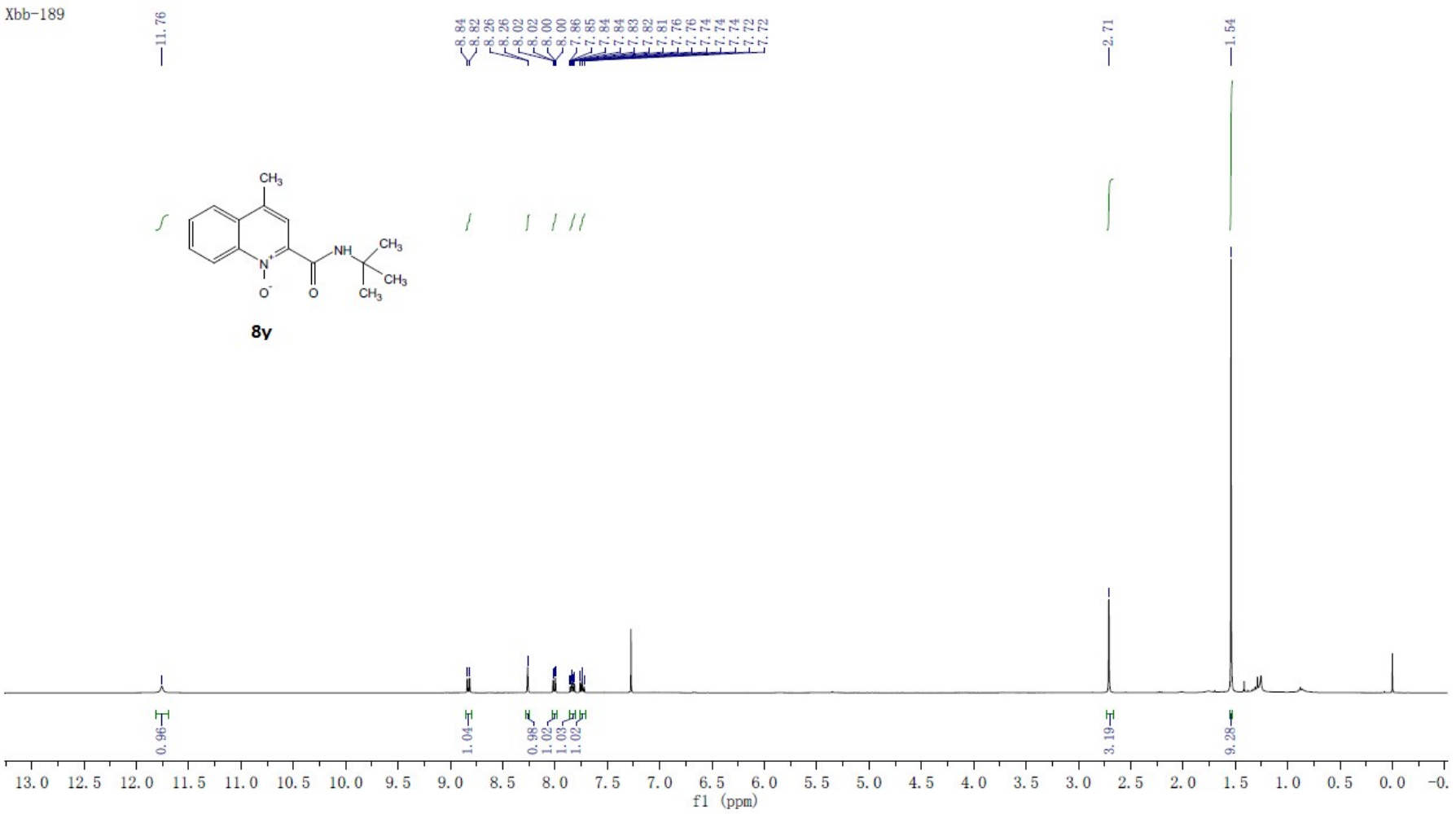
8v

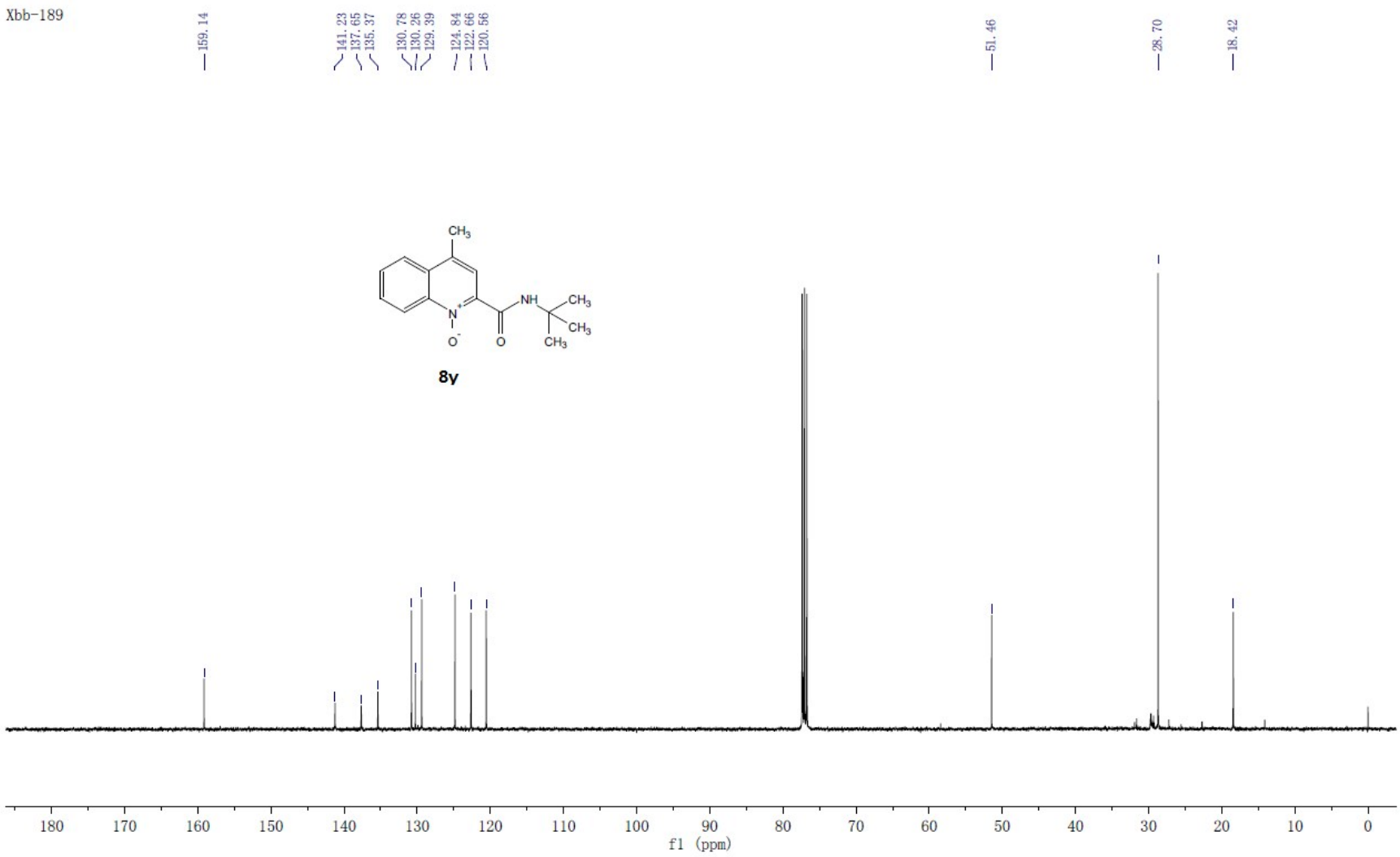

X1b-212
11.34
8.96
8.44
8.42
7.82
7.81
7.81
7.79
7.79
7.78
7.77
7.75
3.62
3.61
3.59
3.59
3.57
3.56
1.35
1.33
1.31
Br
N+
O-
NH
CH3
O
8w
0.73
0.90
0.90
2.81
1.90
3.00
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

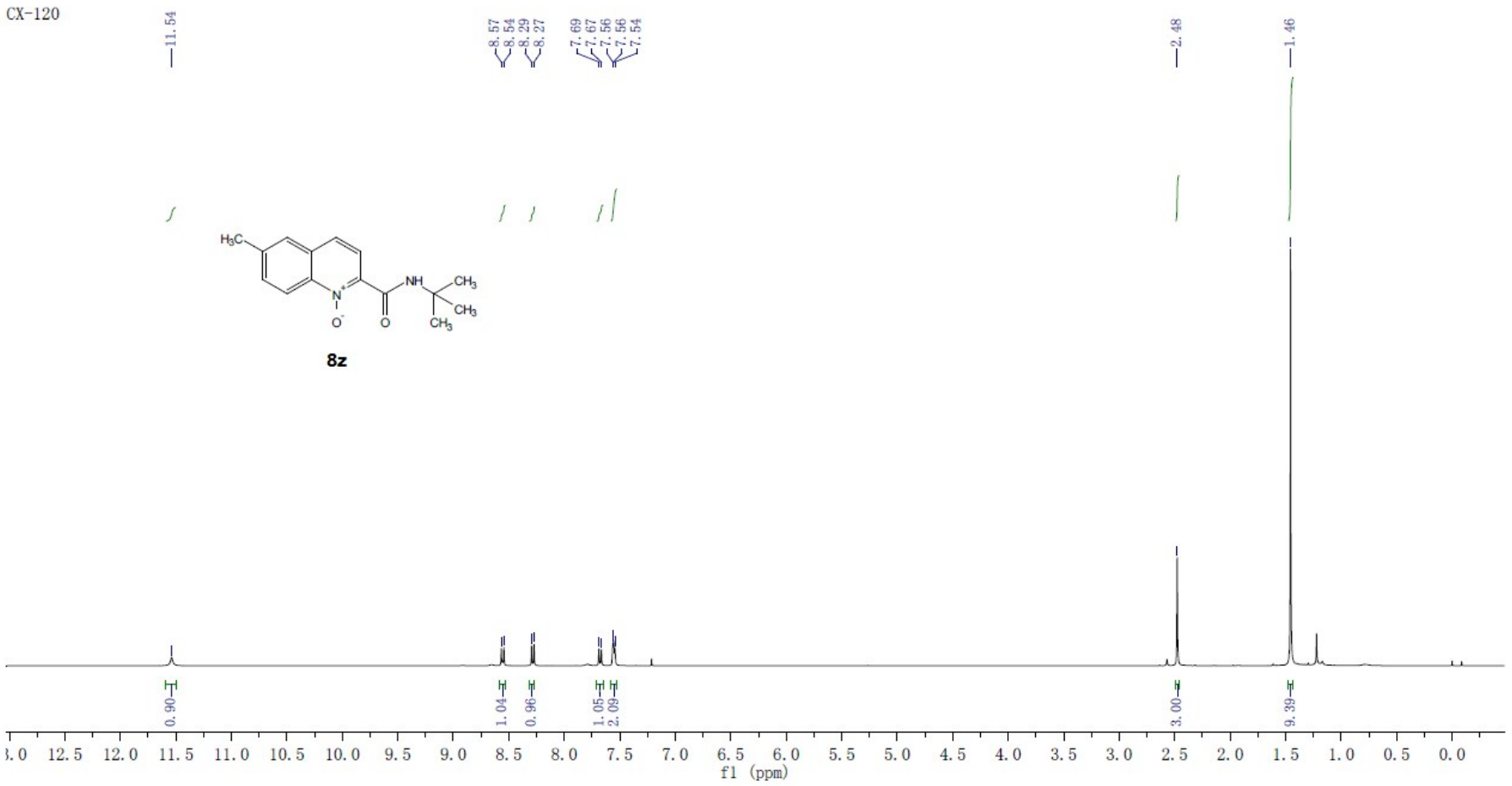

X1b-212-20161220-CNMR
X1b-212-20161220-CNMR
159.84
142.14
138.09
133.40
129.43
129.29
126.00
125.83
123.14
122.98
34.72
14.61
Br
N+
O-
NH
CH3
O
8w
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

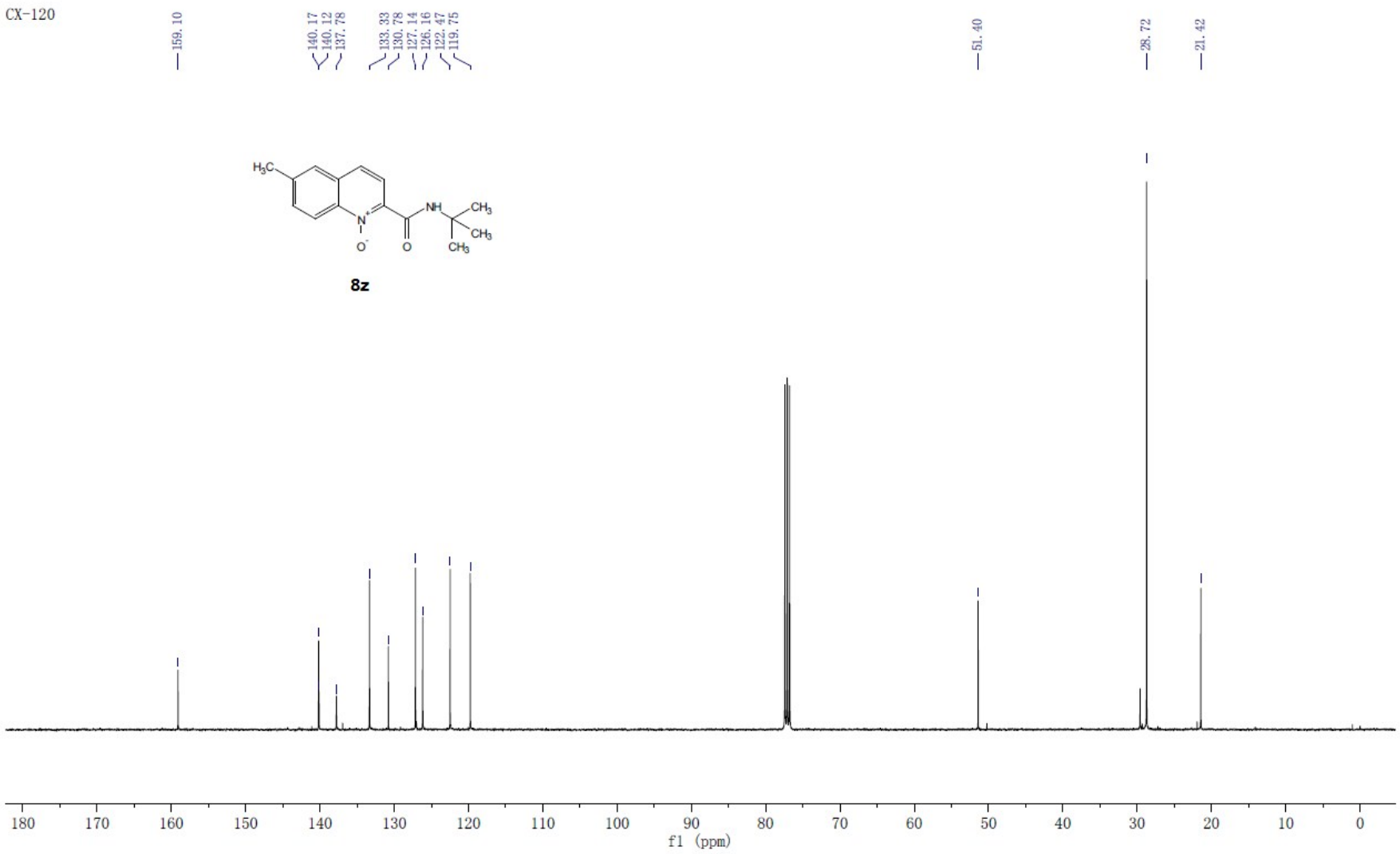

XGX-004
11.61
8.79
8.77
8.43
8.41
7.91
7.89
7.87
7.85
7.85
7.83
7.83
7.83
7.81
7.81
7.73
7.73
7.71
7.71
7.69
7.69
1.55
NH
CH3
CH3
CH3
N+
O-
O
8x
0.88
1.02
1.00
3.18
1.03
9.30
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

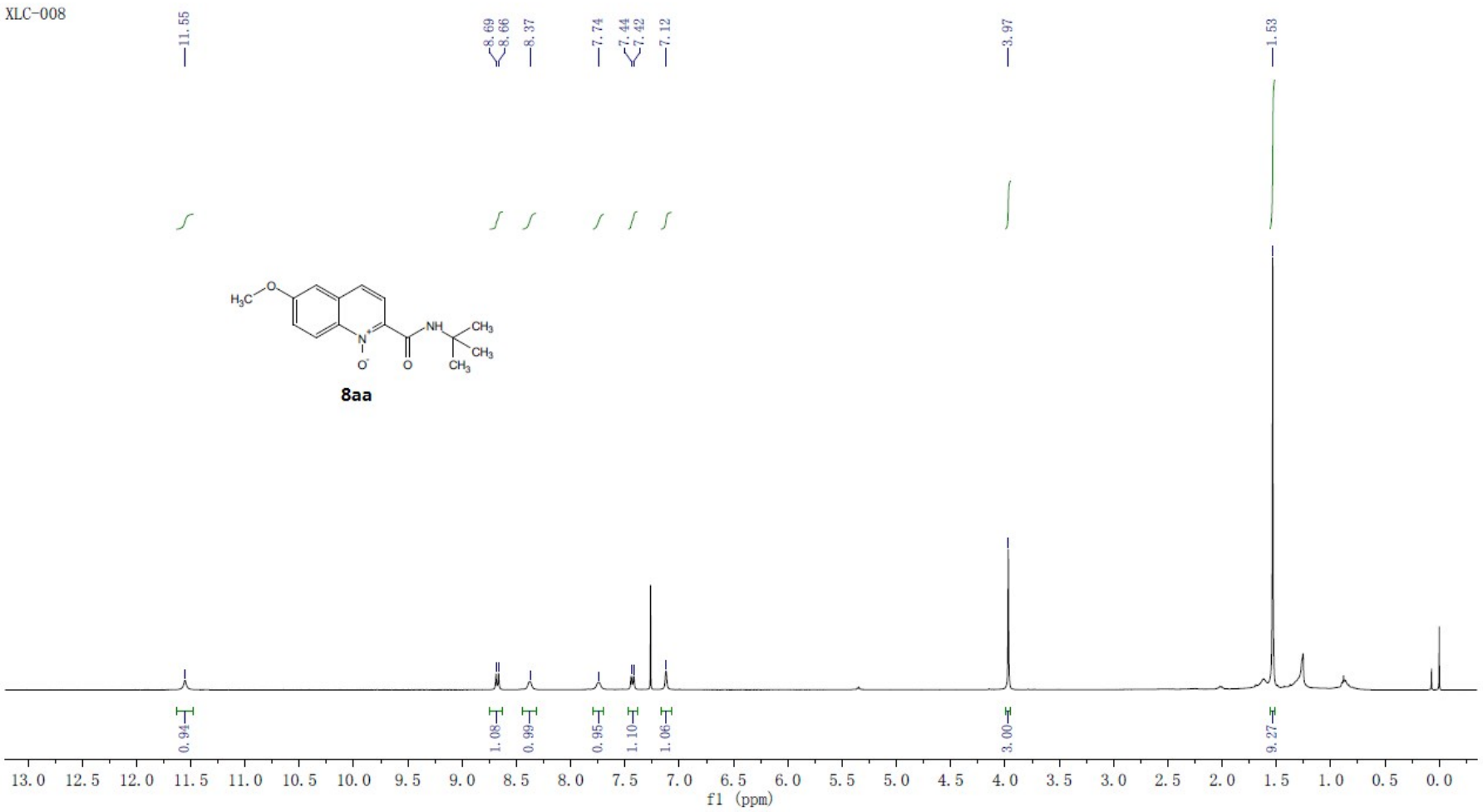

XGX-004-20161223-CNMR
XGX-004-20161223-CNMR
158.98
141.71
138.46
131.13
130.64
129.64
128.23
126.58
122.49
120.03
51.46
28.71
NH
CH3
CH3
CH3
N+
O-
O
8x
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Xbb-189
11.76
8.84
8.82
8.26
8.02
8.02
8.00
8.00
7.86
7.85
7.84
7.84
7.83
7.82
7.81
7.76
7.76
7.74
7.74
7.74
7.72
7.72
2.71
1.54
CH3
NH
CH3
CH3
CH3
N+
O-
O
8y
0.96
1.04
0.98
1.02
1.03
1.02
3.19
9.28
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

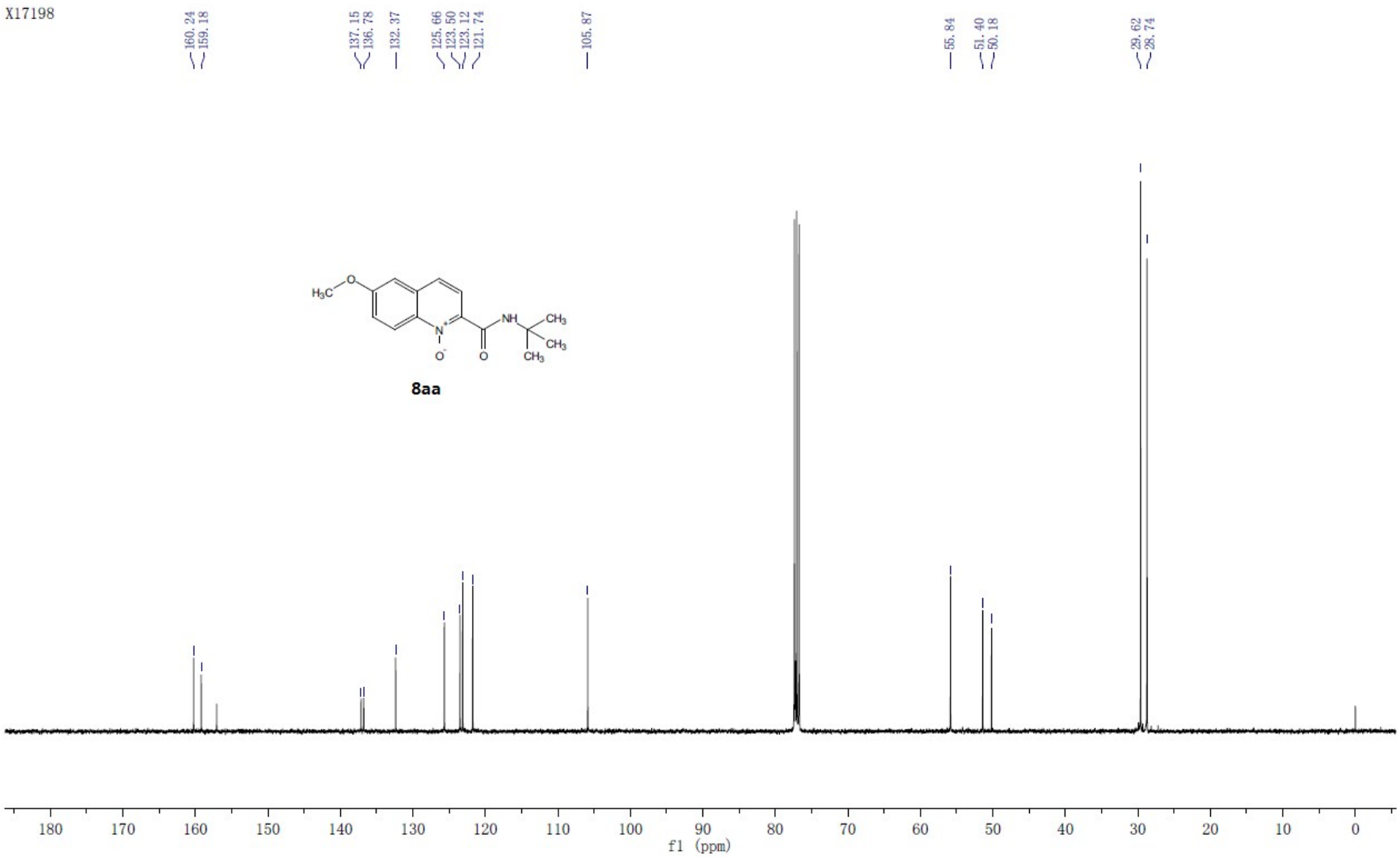

Xbb-189
159.14
141.23
137.65
135.37
130.78
130.26
129.39
124.84
122.66
120.56
51.46
28.70
18.42
CH3
N+
O-
NH
O
CH3
CH3
CH3
8y
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

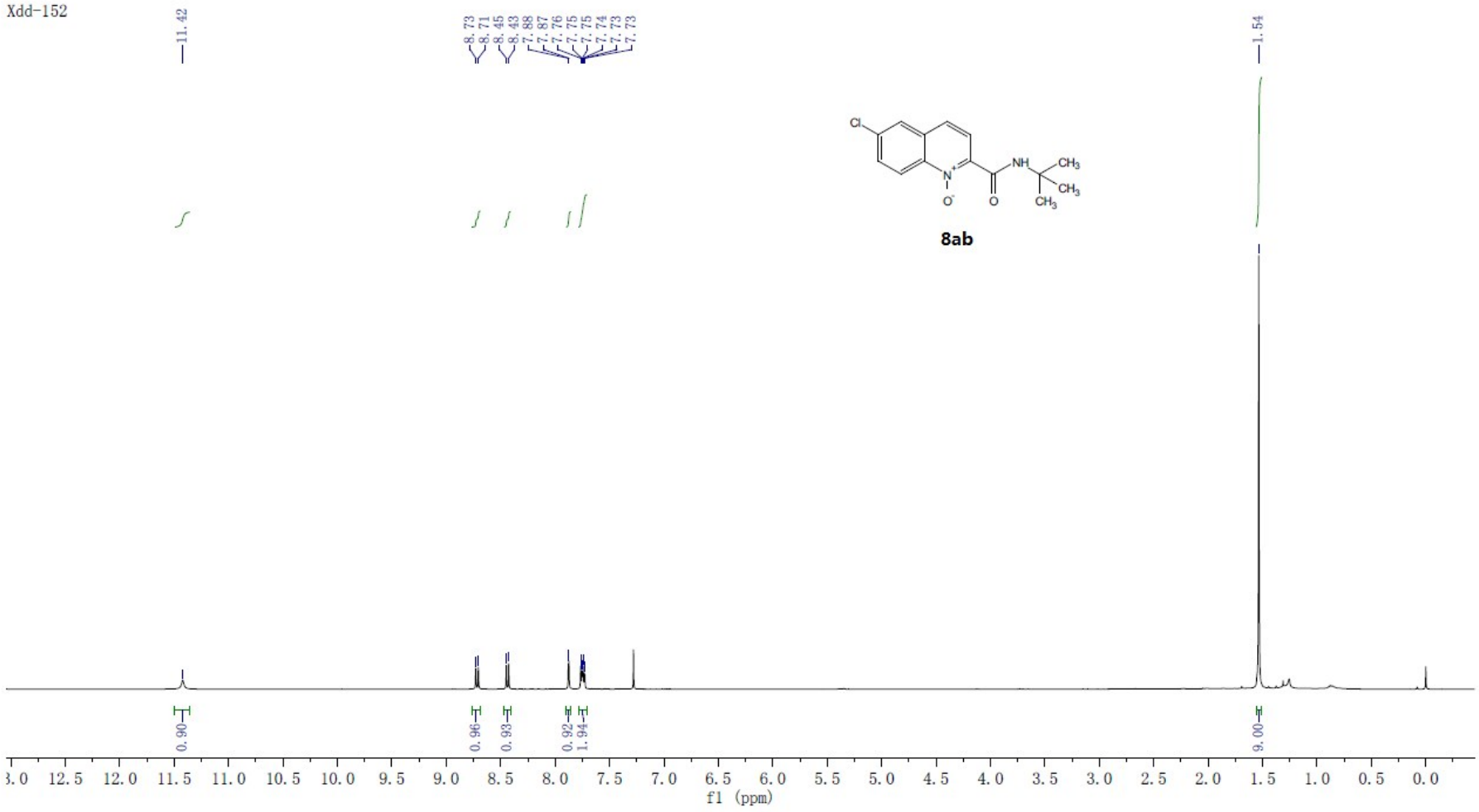

CX-120
11.54
8.57
8.54
8.29
8.27
7.69
7.67
7.56
7.56
7.54
2.48
1.46
H3C
N+
O-
NH
CH3
CH3
CH3
O
8z
0.90
1.04
0.96
1.05
2.09
3.00
9.39
f1 (ppm)

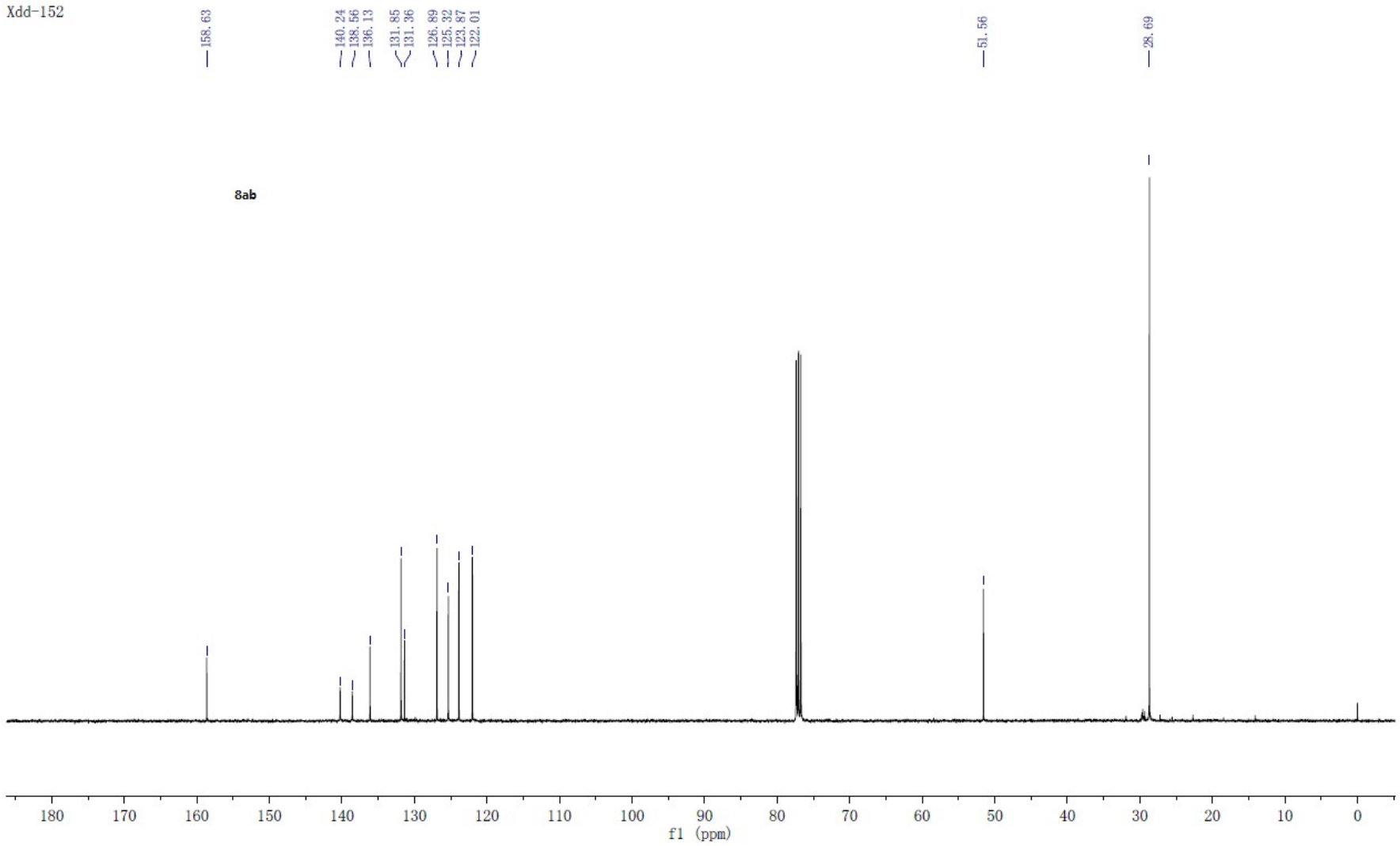

CX-120
159.10
140.17
140.12
137.78
133.33
130.78
127.14
126.16
122.47
119.75
51.40
28.72
21.42
H3C
N+
O-
O
NH
CH3
CH3
CH3
8z
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

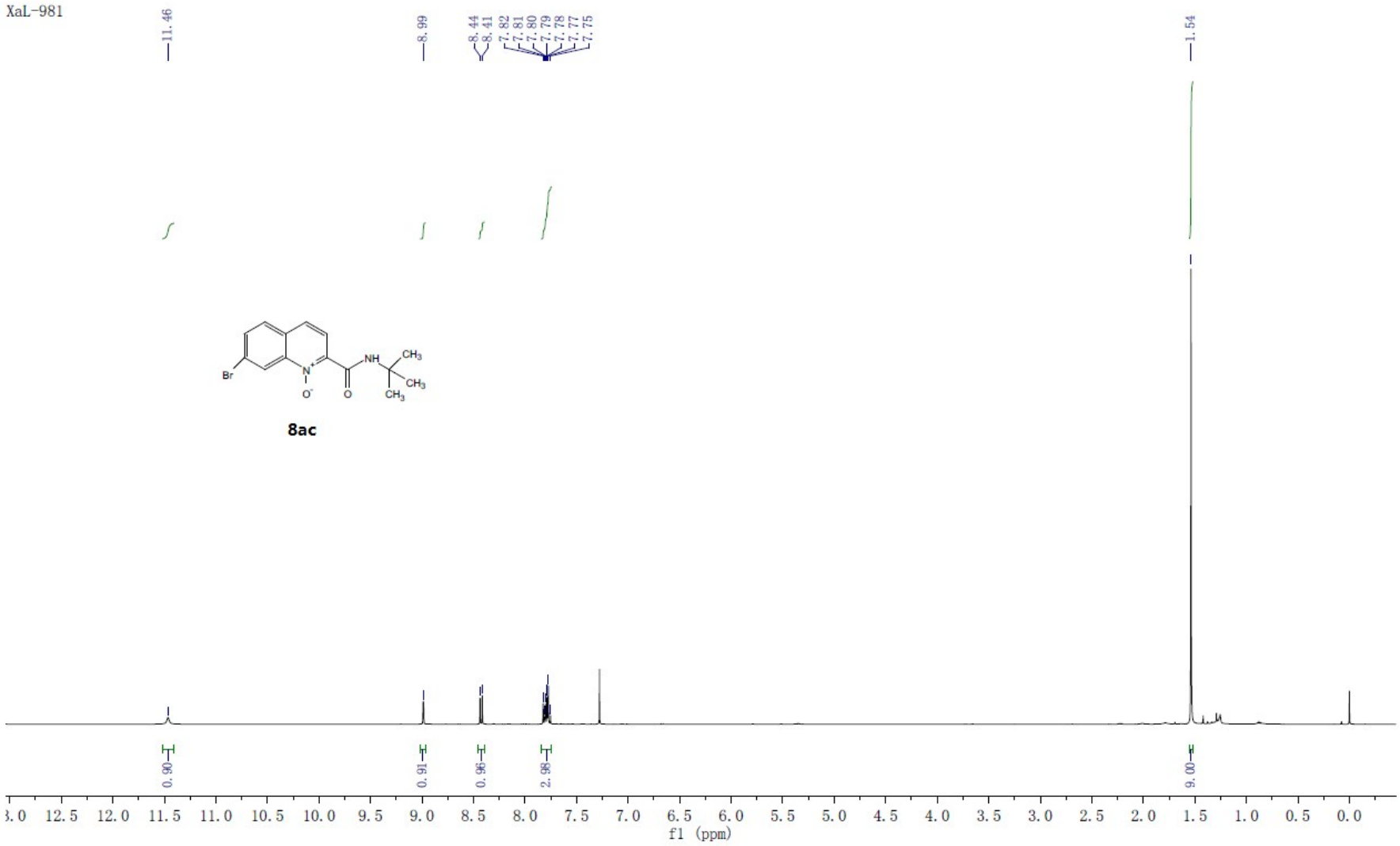

XLC-008
11.55
8.69
8.66
8.37
7.74
7.44
7.42
7.12
3.97
1.53
8aa
0.94
1.08
0.99
0.95
1.10
1.06
3.00
9.27
f1 (ppm)

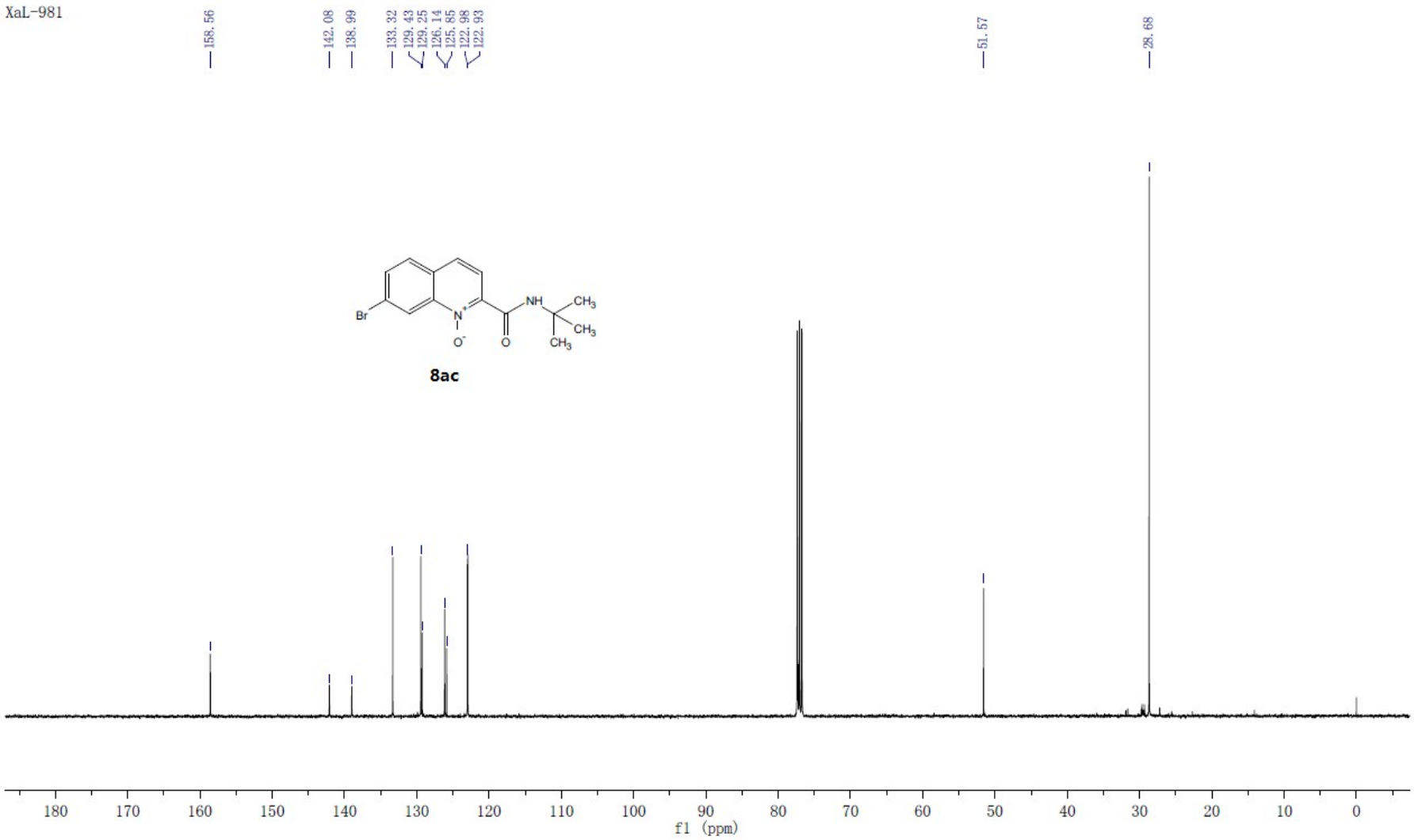
X17198

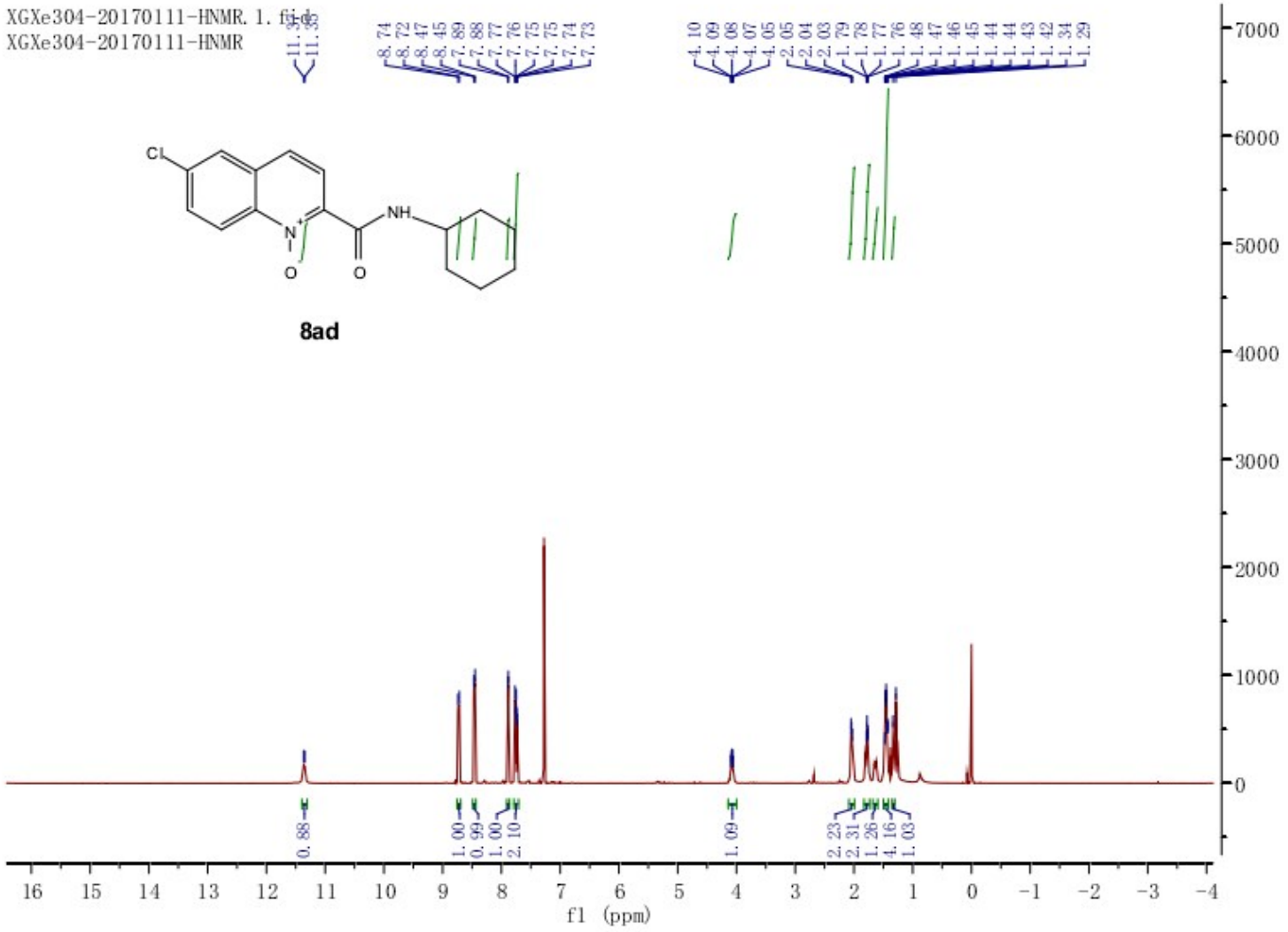

160.24
159.18
137.15
136.78
132.37
125.66
123.50
123.12
121.74
105.87
55.84
51.40
50.18
29.62
28.74
H3C
O
N+
O-
O
NH
CH3
CH3
CH3
8aa
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

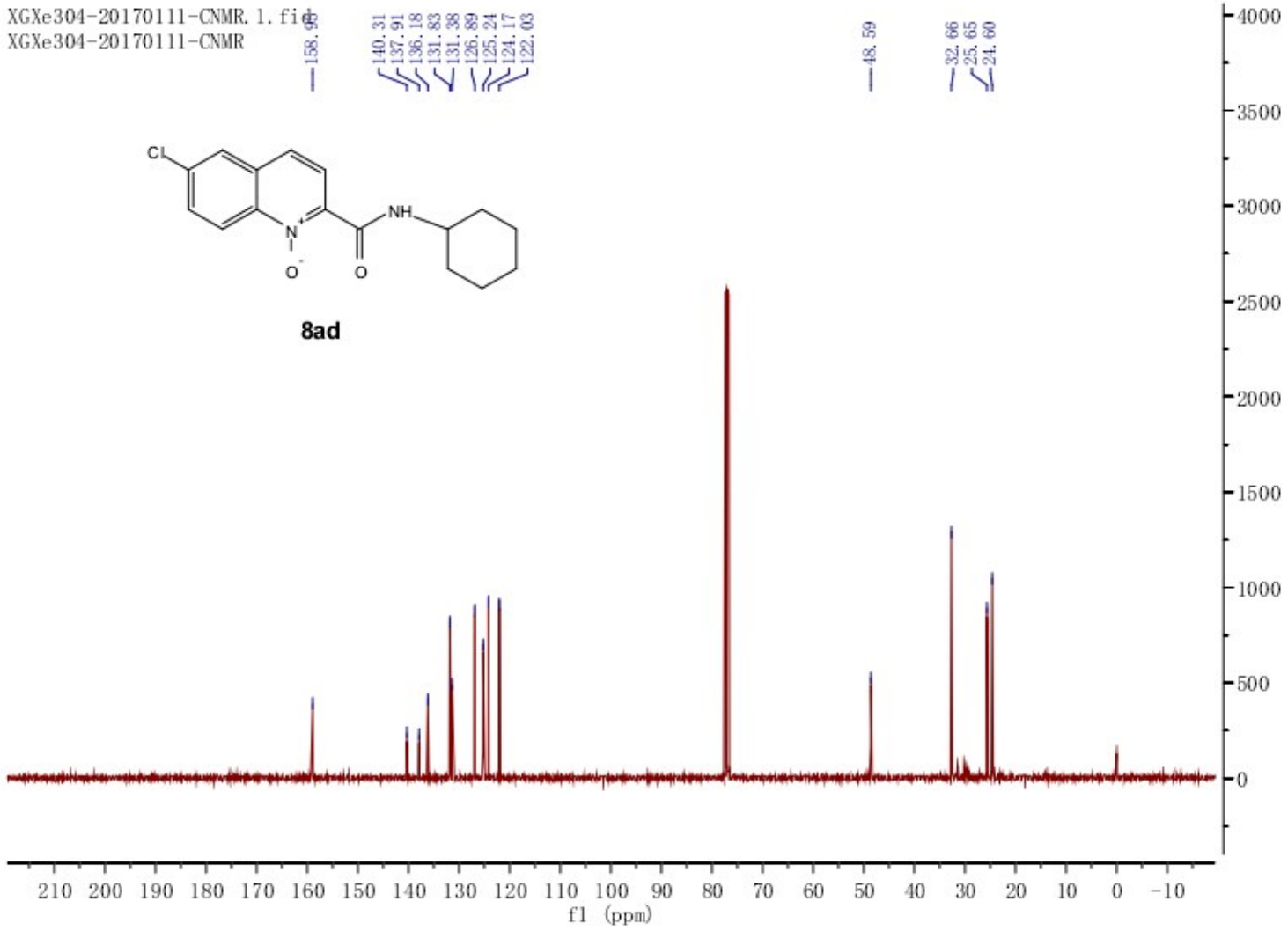

Xdd-152
11.42
8.73
8.71
8.45
8.43
7.88
7.87
7.76
7.75
7.75
7.74
7.73
7.73
1.54
Cl
N+
O-
O
NH
CH3
CH3
CH3
8ab
0.90
0.96
0.93
0.92
1.94
9.00
f1 (ppm)

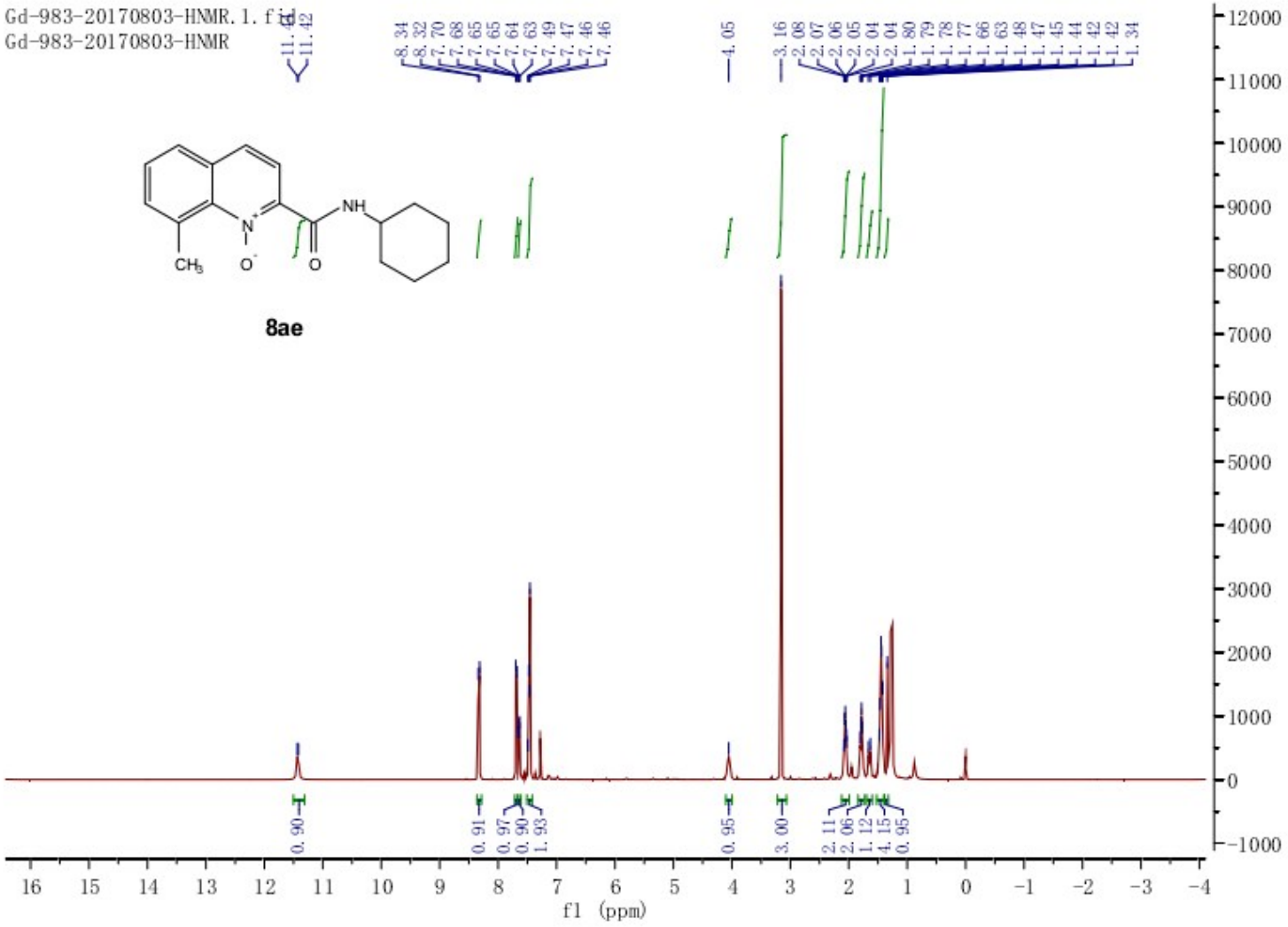

Xdd-152
158.63
140.24
138.56
136.13
131.85
131.36
126.89
125.32
123.87
122.01
51.56
28.69
8ab
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

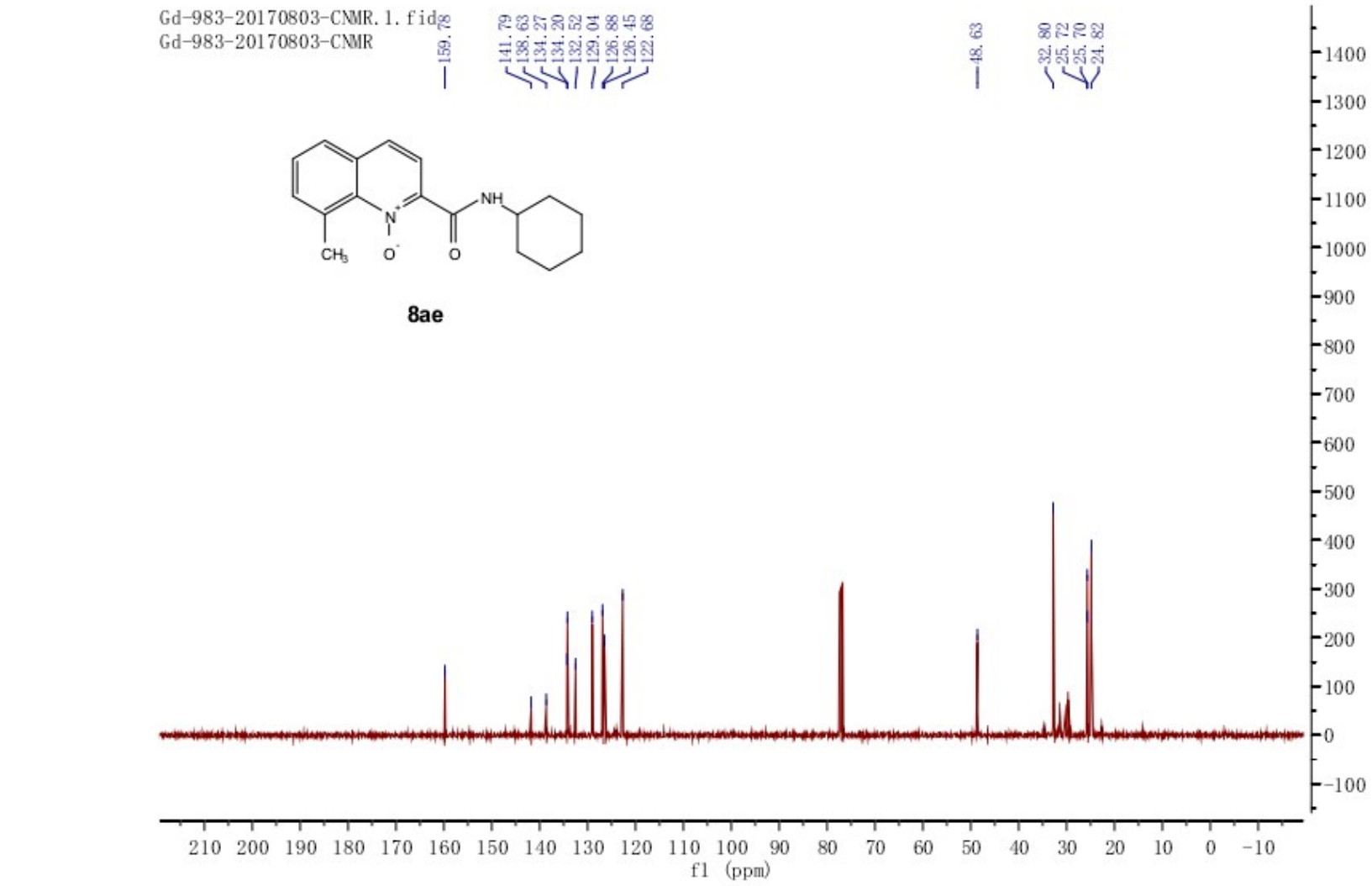

XaL-981
11.46
8.99
8.44
8.41
7.82
7.81
7.80
7.79
7.78
7.77
7.75
1.54
Br
N+
O-
O
NH
CH3
CH3
CH3
8ac
0.90
0.91
0.96
2.98
9.00
f1 (ppm)

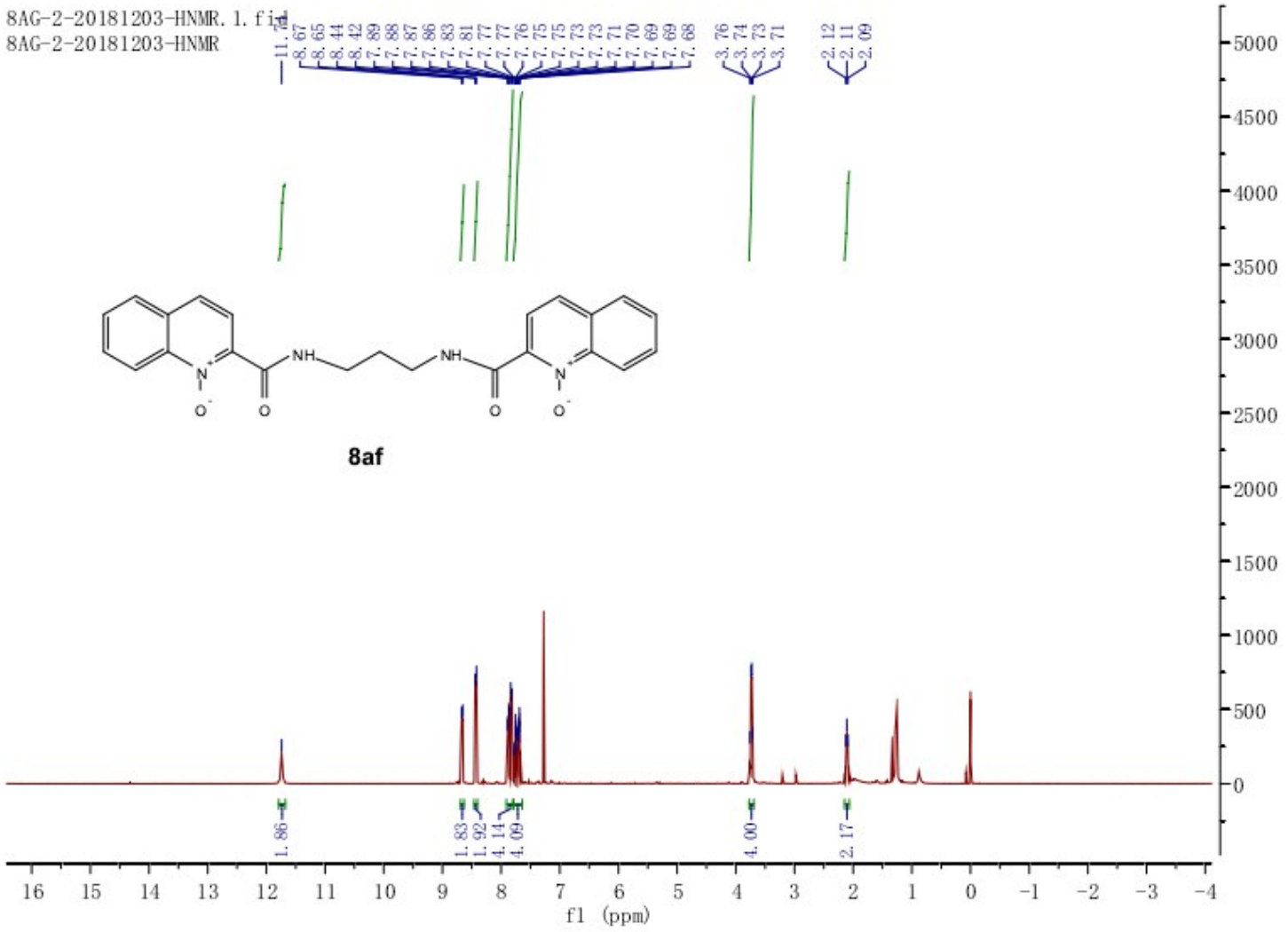

XaL-981
158.56
142.08
138.99
133.32
129.43
129.25
126.14
125.85
122.98
122.93
51.57
28.68
Br
N+
O-
O
NH
CH3
CH3
CH3
8ac
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

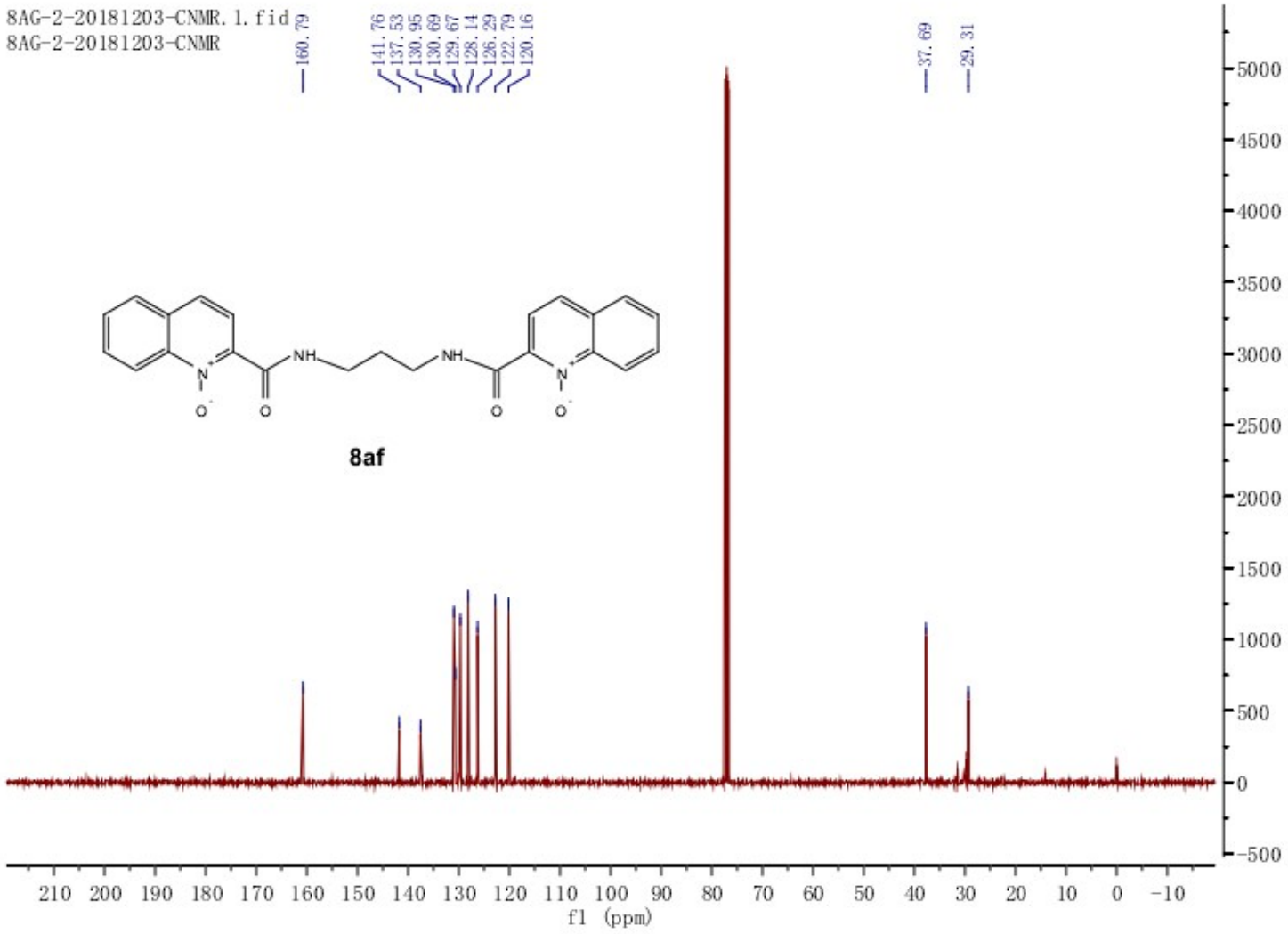

XGXe304-20170111-HNMR.1.fid
XGXe304-20170111-HNMR
11.36
11.35
8.74
8.72
8.47
8.45
7.89
7.88
7.77
7.76
7.75
7.74
7.73
4.10
4.09
4.08
4.07
4.05
2.05
2.04
2.03
1.79
1.78
1.77
1.76
1.48
1.47
1.46
1.45
1.44
1.44
1.43
1.42
1.34
1.29
Cl
N
O
NH
O
8ad
7000
6000
5000
4000
3000
2000
1000
0
0.88
1.00
0.99
1.00
2.10
1.09
2.23
2.31
1.26
4.16
1.03
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
−4
f1 (ppm)

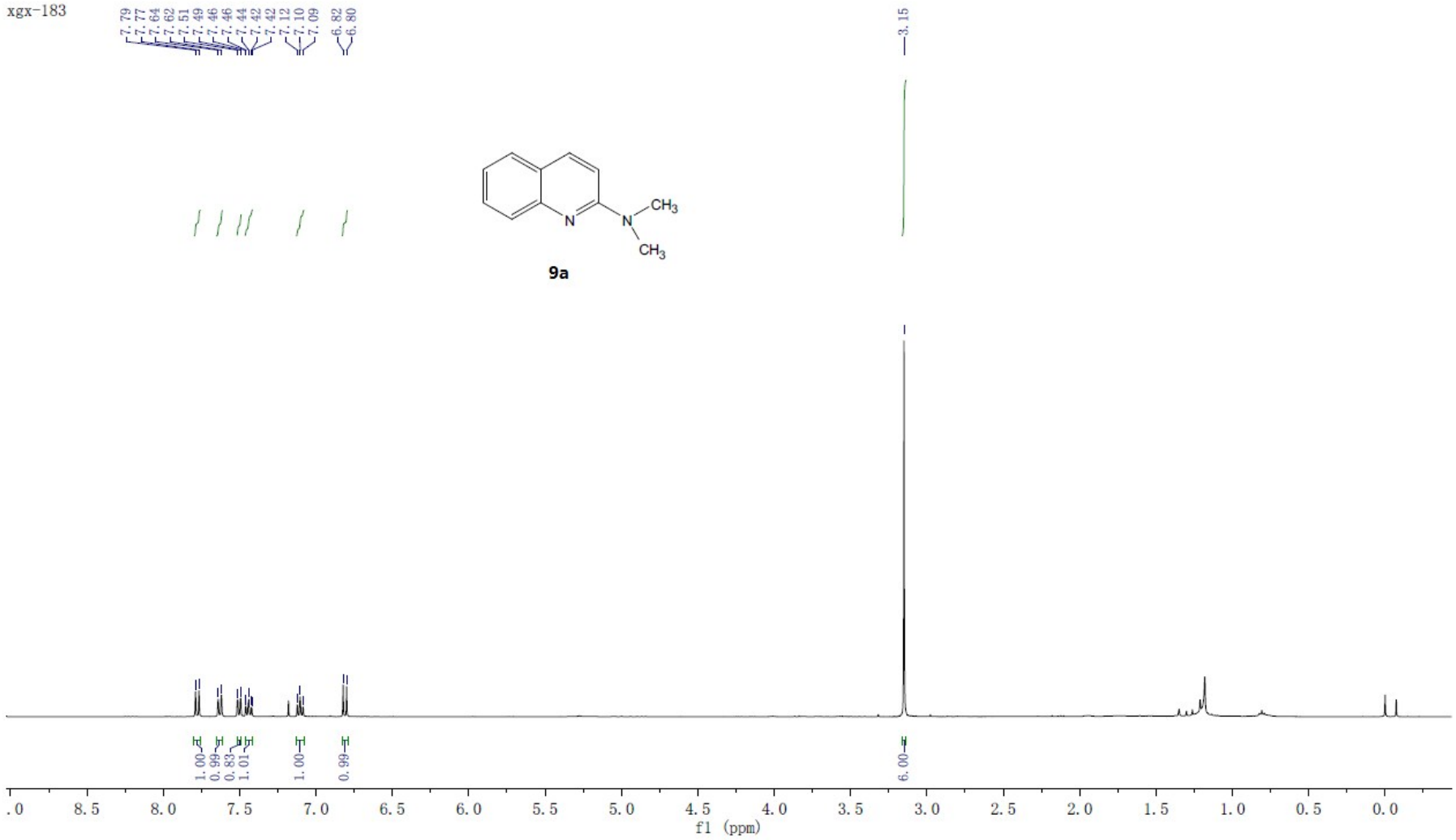

XGXe304-20170111-CNMR.1.fid
XGXe304-20170111-CNMR
158.95
140.31
137.91
136.18
131.83
131.38
126.89
125.24
124.17
122.03
48.59
32.66
25.65
24.60
Cl
N+
O-
NH
O
8ad
4000
3500
3000
2500
2000
1500
1000
500
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

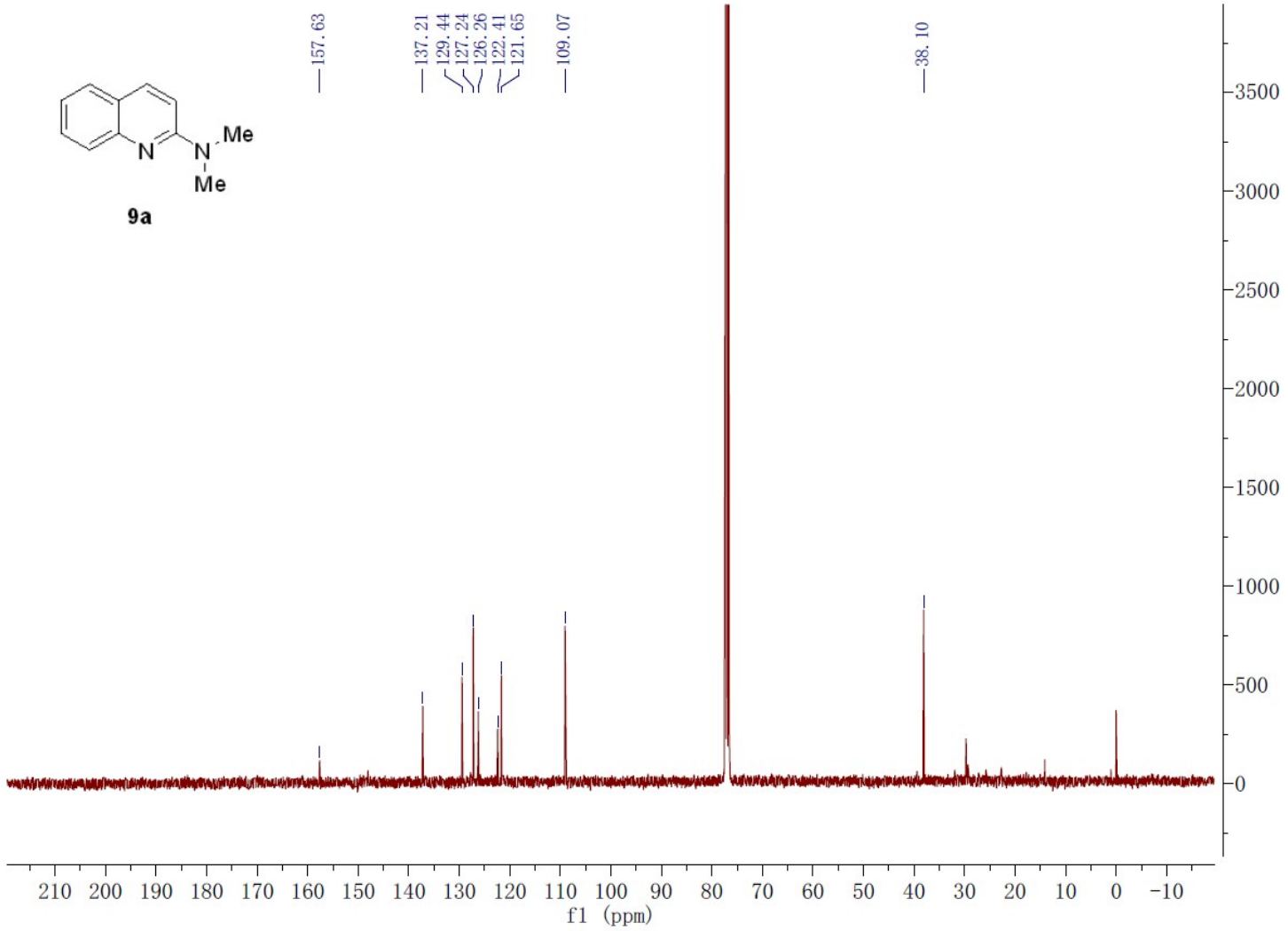

Gd-983-20170803-HNMR.1.fid
Gd-983-20170803-HNMR
11.42
8.34
8.32
7.70
7.68
7.65
7.65
7.64
7.63
7.49
7.47
7.46
7.46
4.05
3.16
2.08
2.07
2.06
2.05
2.04
2.04
1.80
1.79
1.78
1.77
1.66
1.63
1.48
1.47
1.45
1.44
1.42
1.42
1.34
NH
N
O
CH3
O
8ae
0.90
0.91
0.97
0.90
1.93
0.95
3.00
2.11
2.06
1.12
4.15
0.95
f1 (ppm)

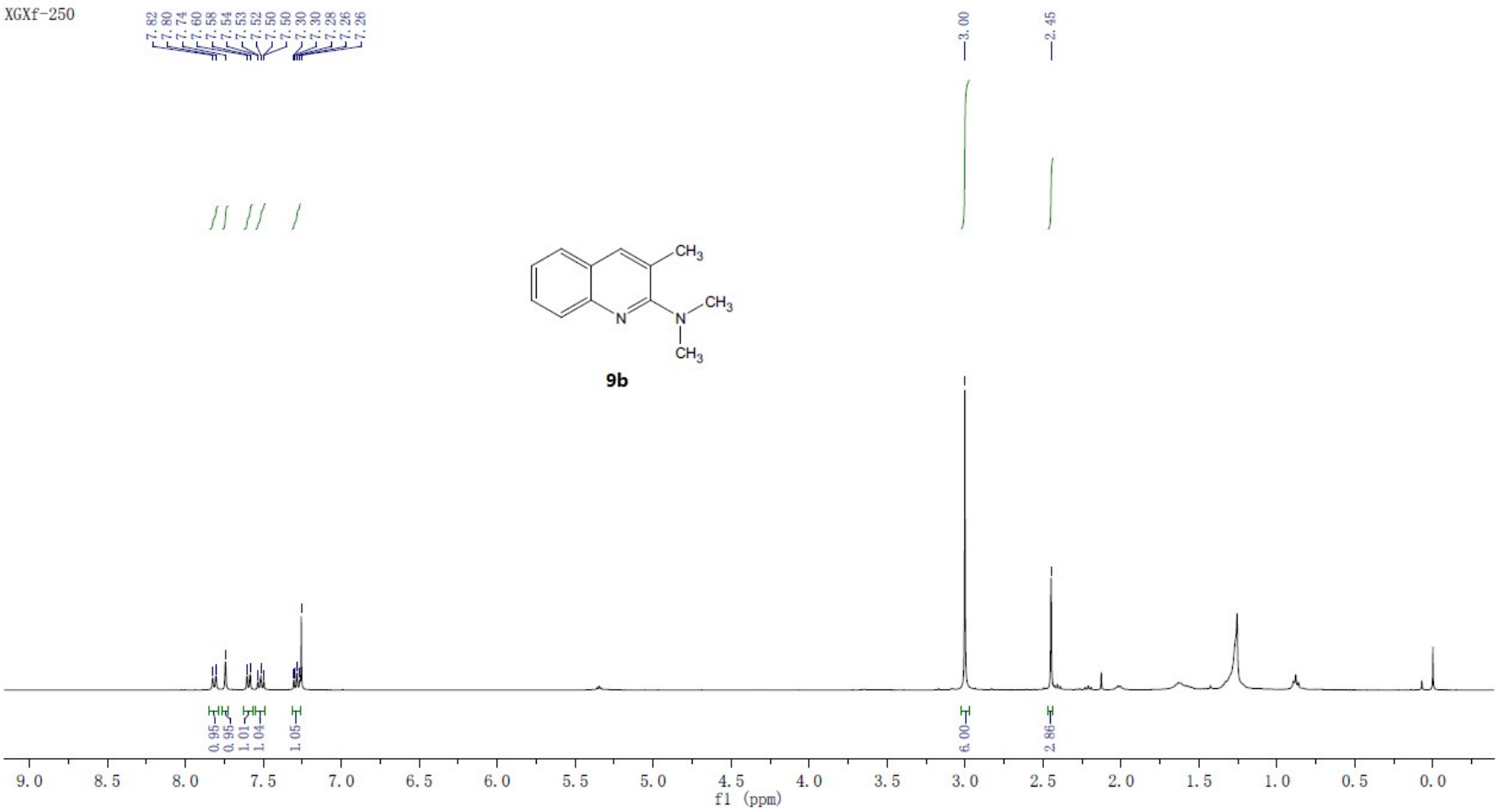

Gd-983-20170803-CNMR.1.fid
Gd-983-20170803-CNMR
159.78
141.79
138.63
134.27
134.20
132.52
129.04
126.88
126.45
122.68
48.63
32.80
25.72
25.70
24.82
NH
N+
O-
CH3
O
8ae
f1 (ppm)

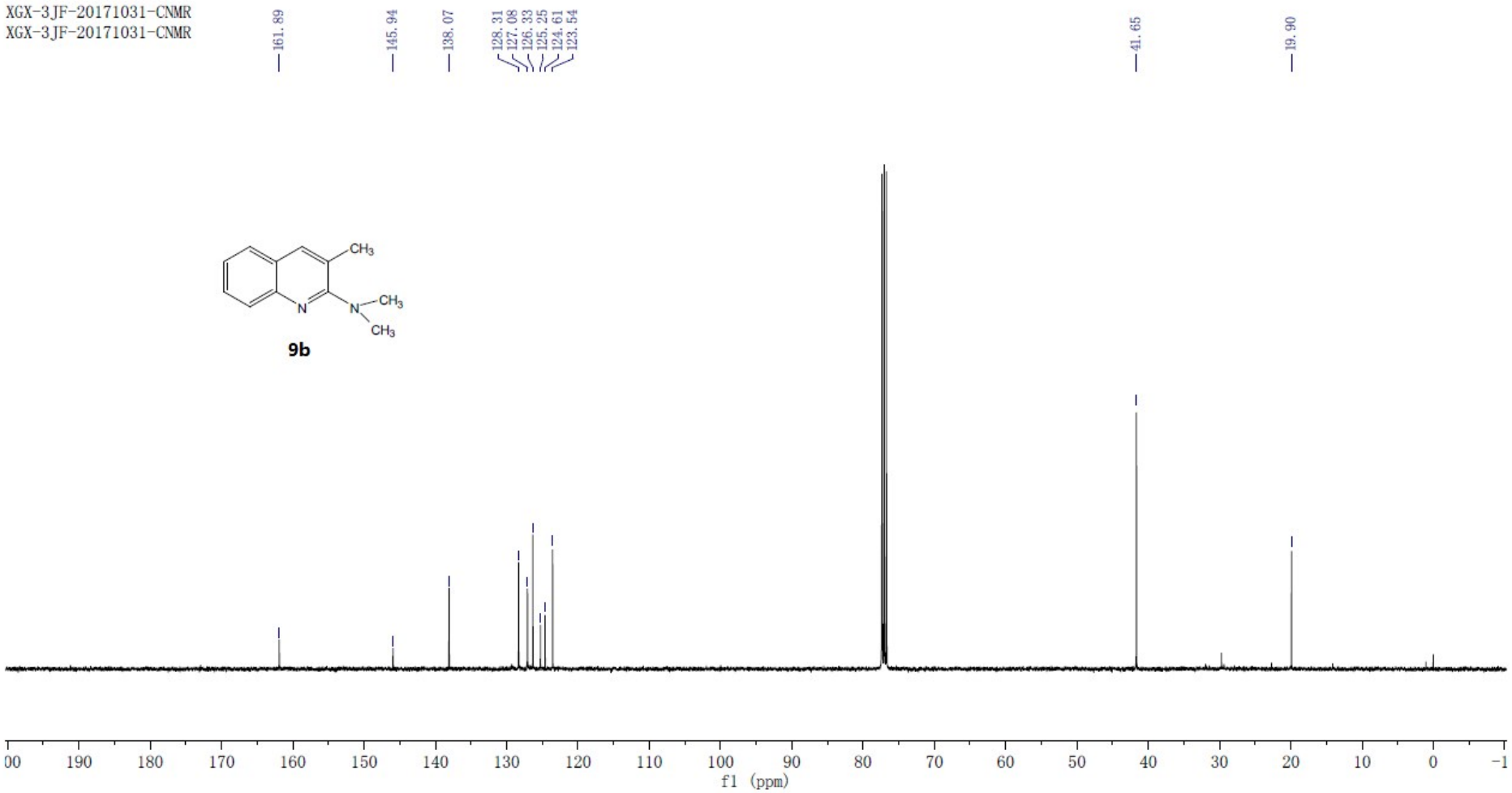

8AG-2-20181203-HNMR.1.fid
8AG-2-20181203-HNMR
11.74
8.67
8.65
8.44
8.42
7.89
7.88
7.87
7.86
7.83
7.81
7.77
7.77
7.76
7.75
7.75
7.73
7.73
7.71
7.70
7.69
7.69
7.68
3.76
3.74
3.73
3.71
2.12
2.11
2.09
NH
NH
O
O
O
O
8af
1.86
1.83
1.92
4.14
4.09
4.00
2.17
f1 (ppm)

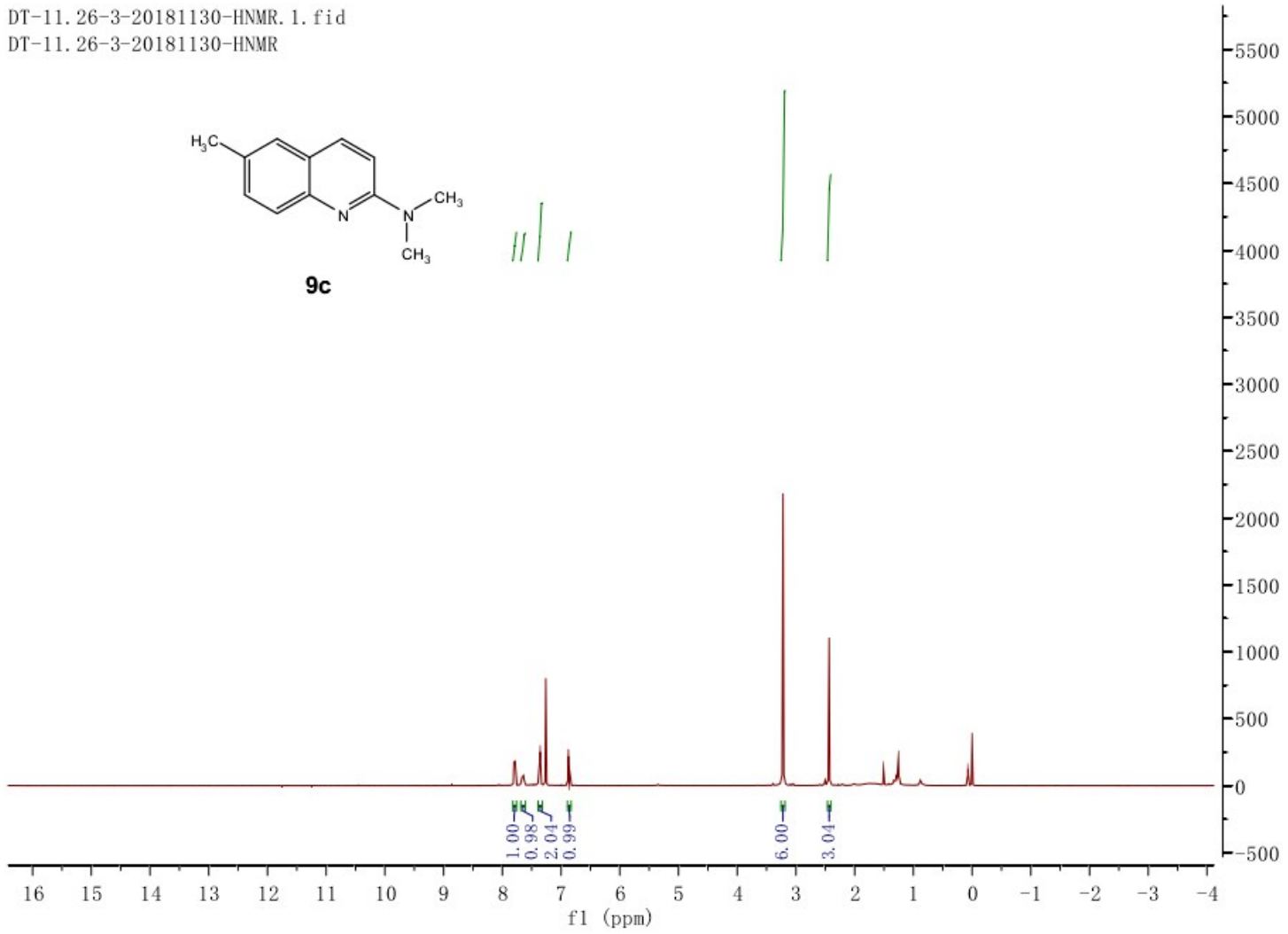

8AG-2-20181203-CNMR.1.fid
8AG-2-20181203-CNMR
160.79
141.76
137.53
130.95
130.69
129.67
128.14
126.29
122.79
120.16
37.69
29.31
NH
NH
8af
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

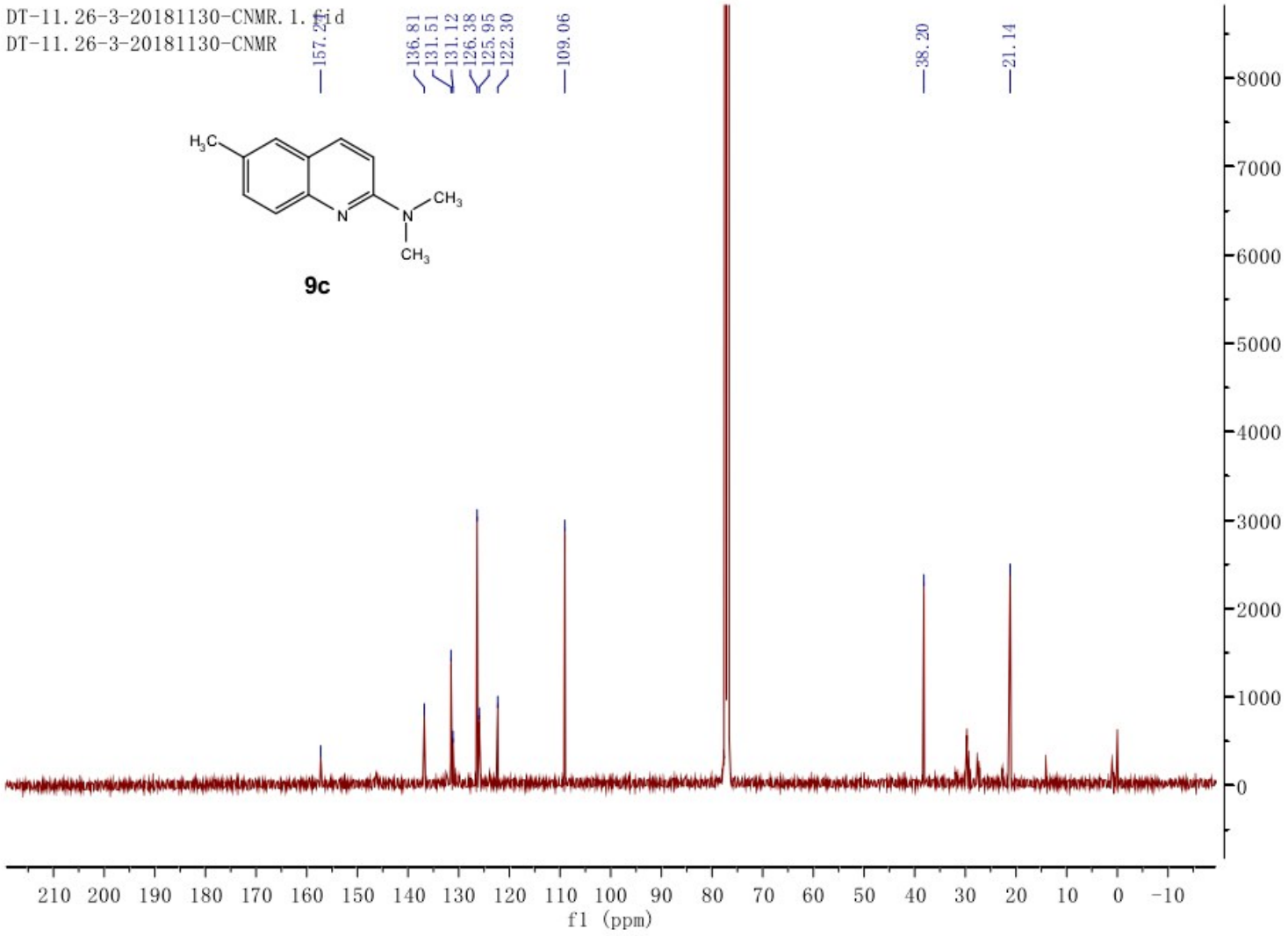

xgx-183
7.79
7.77
7.64
7.62
7.51
7.49
7.46
7.46
7.44
7.42
7.42
7.12
7.10
7.09
6.82
6.80
3.15
CH3
N
N
CH3
9a
1.00
0.99
0.83
1.01
1.00
0.99
6.00
f1 (ppm)

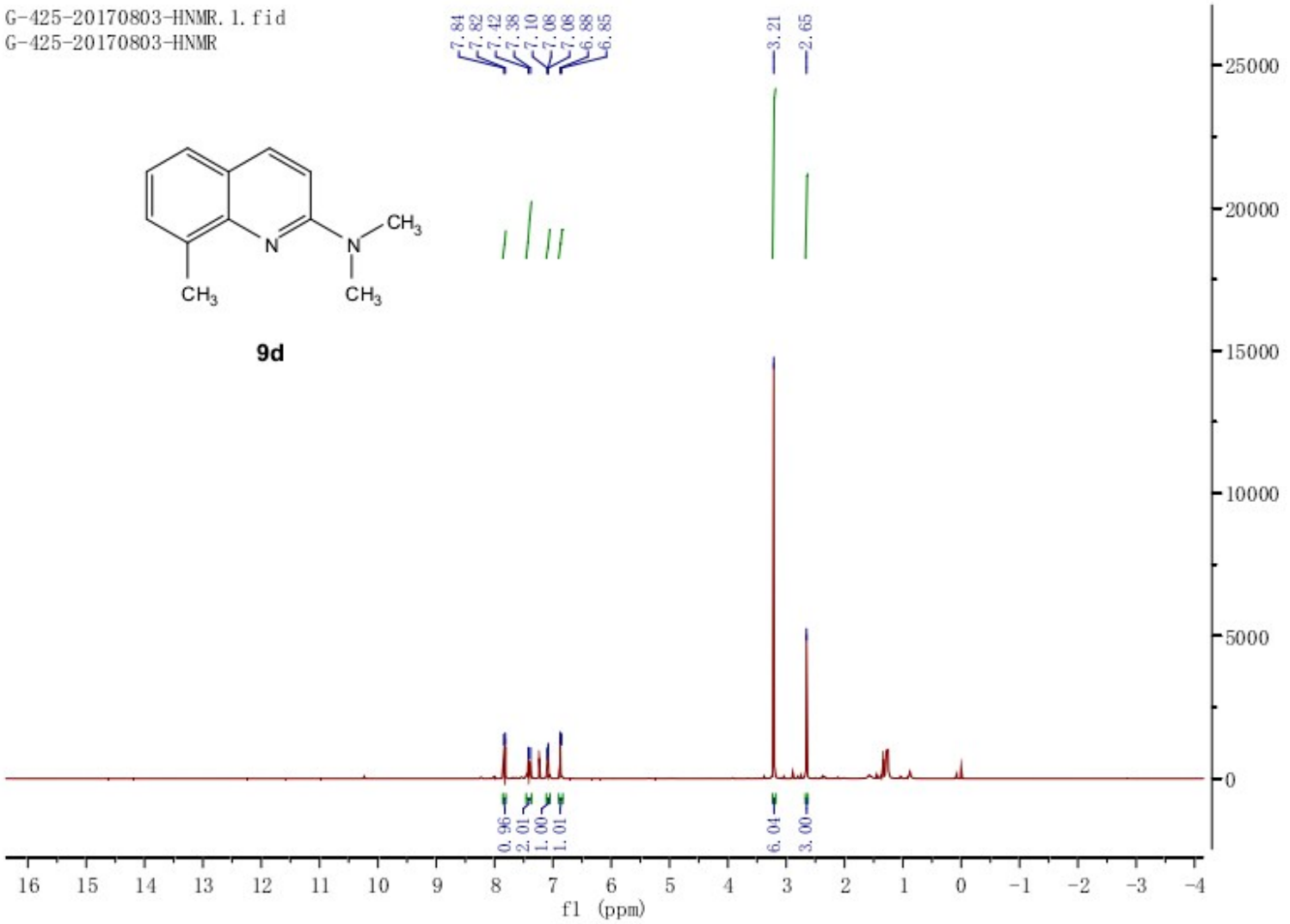

157.63
137.21
129.44
127.24
126.26
122.41
121.65
109.07
38.10
Me
N
N
Me
9a
3500
3000
2500
2000
1500
1000
500
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

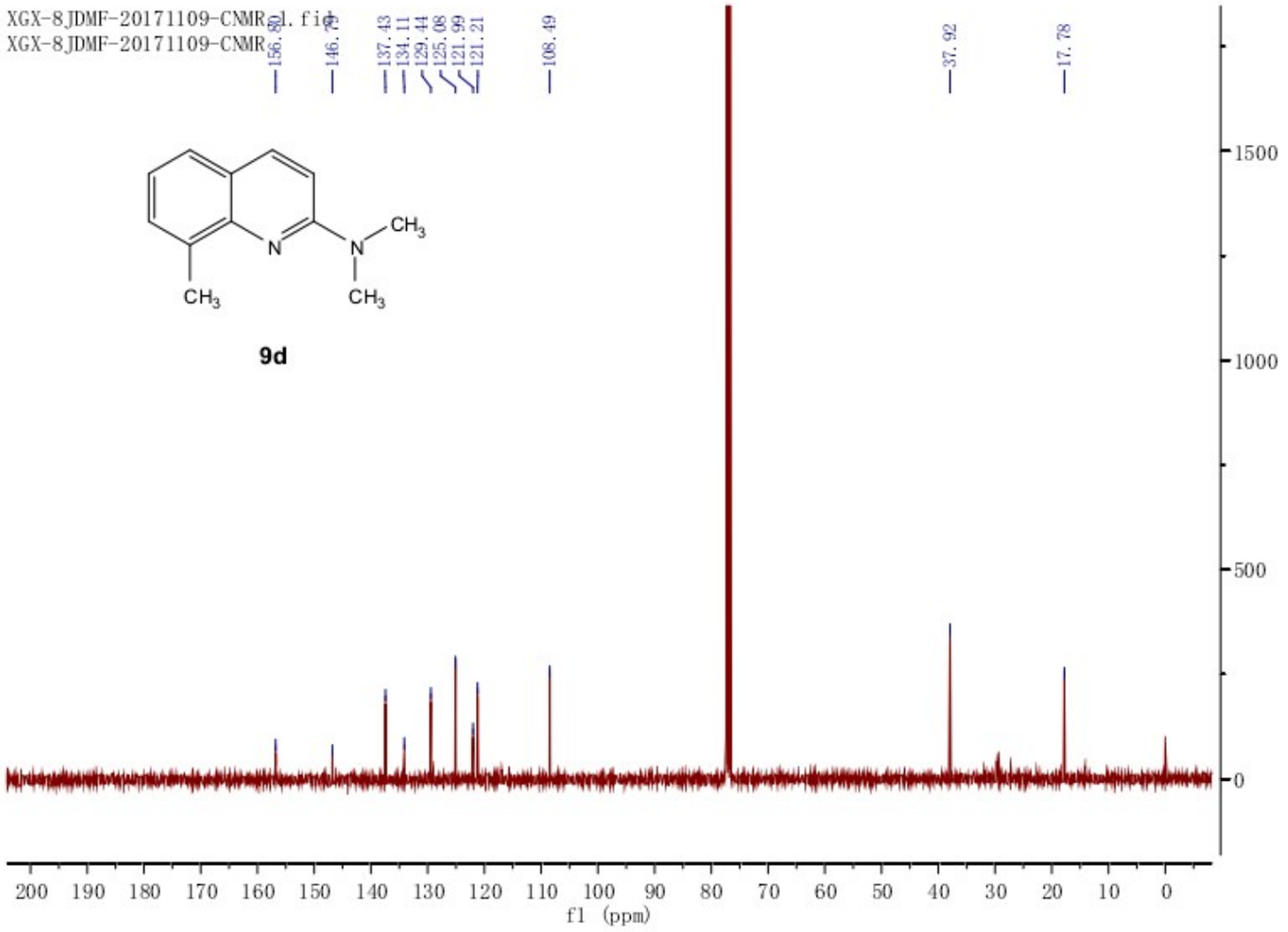

XGXf-250
7.82
7.80
7.74
7.60
7.58
7.54
7.53
7.52
7.50
7.50
7.30
7.30
7.28
7.26
7.26
3.00
2.45
CH3
N
N
CH3
CH3
9b
0.95
0.95
1.01
1.04
1.05
6.00
2.86
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

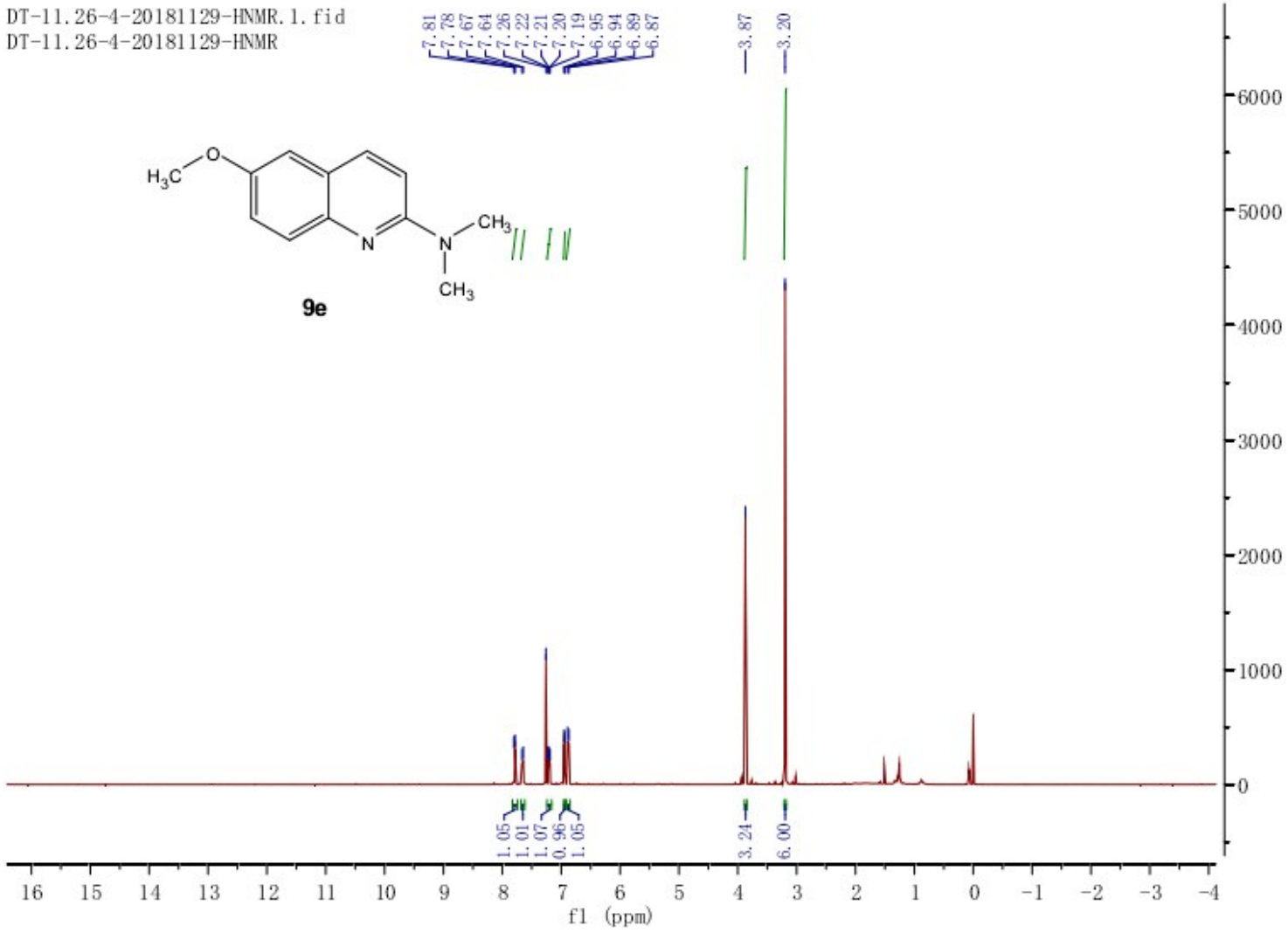

XGX-3JF-20171031-CNMR
XGX-3JF-20171031-CNMR
161.89
145.94
138.07
128.31
127.08
126.33
125.25
124.61
123.54
41.65
19.90
CH3
N
N
CH3
CH3
9b
f1 (ppm)

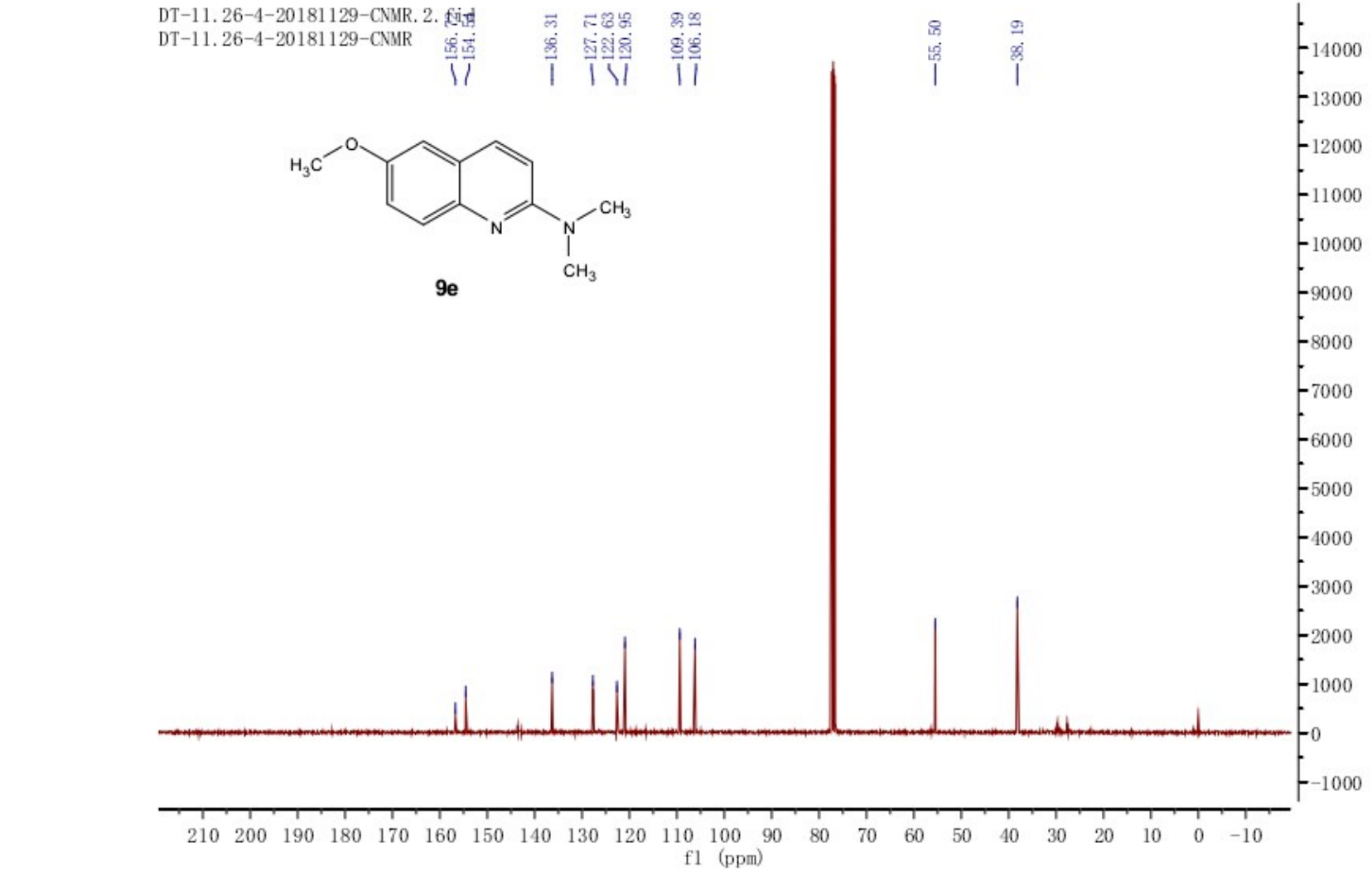

DT-11.26-3-20181130-HNMR.1.fid
DT-11.26-3-20181130-HNMR
H3C
N
N
CH3
CH3
9c
1.00
0.98
2.04
0.99
6.00
3.04
f1 (ppm)

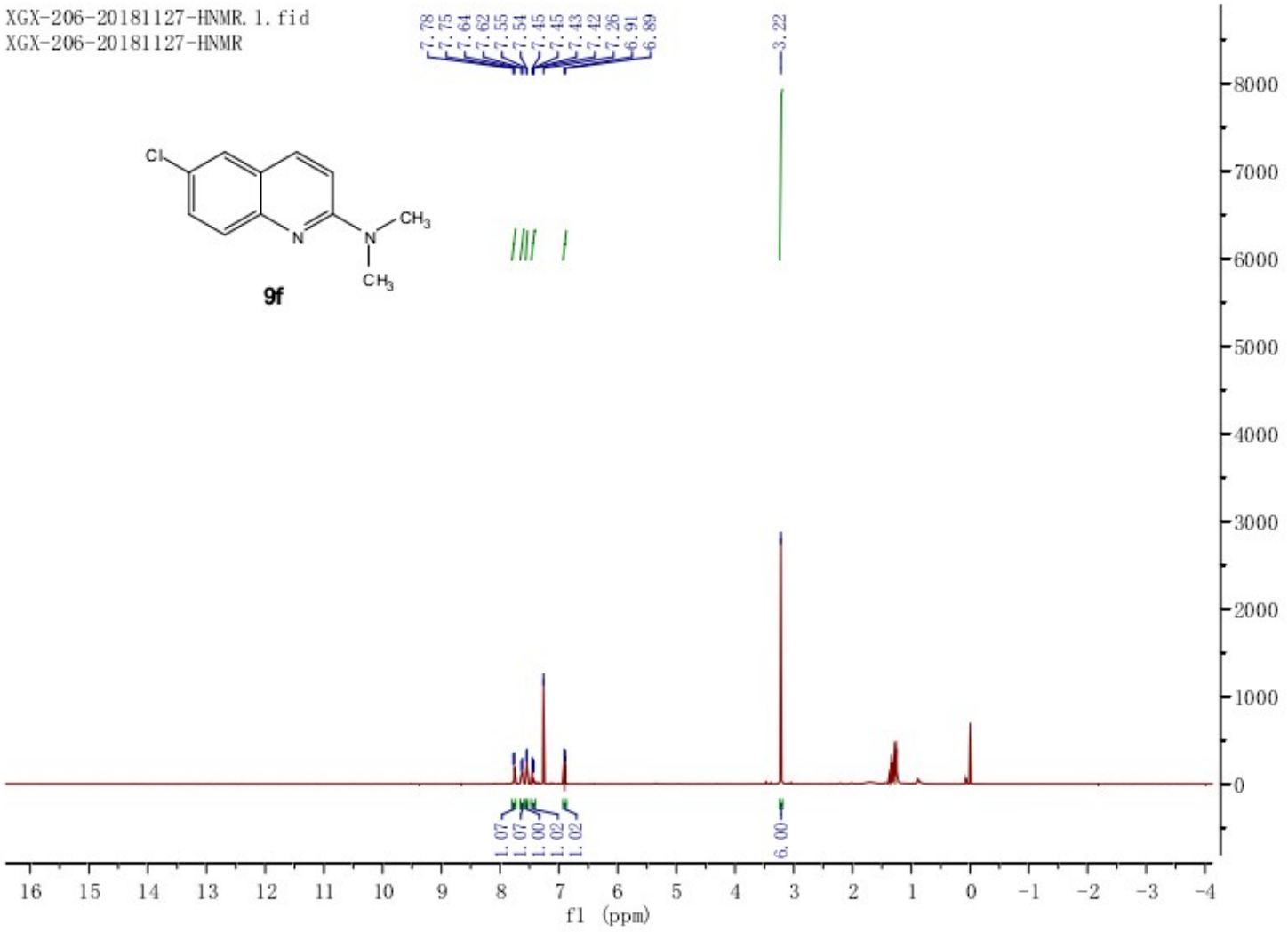

DT-11.26-3-20181130-CNMR.1.fid
DT-11.26-3-20181130-CNMR
157.27
136.81
131.51
131.12
126.38
125.95
122.30
109.06
38.20
21.14
H3C
N
N
CH3
CH3
9c
8000
7000
6000
5000
4000
3000
2000
1000
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

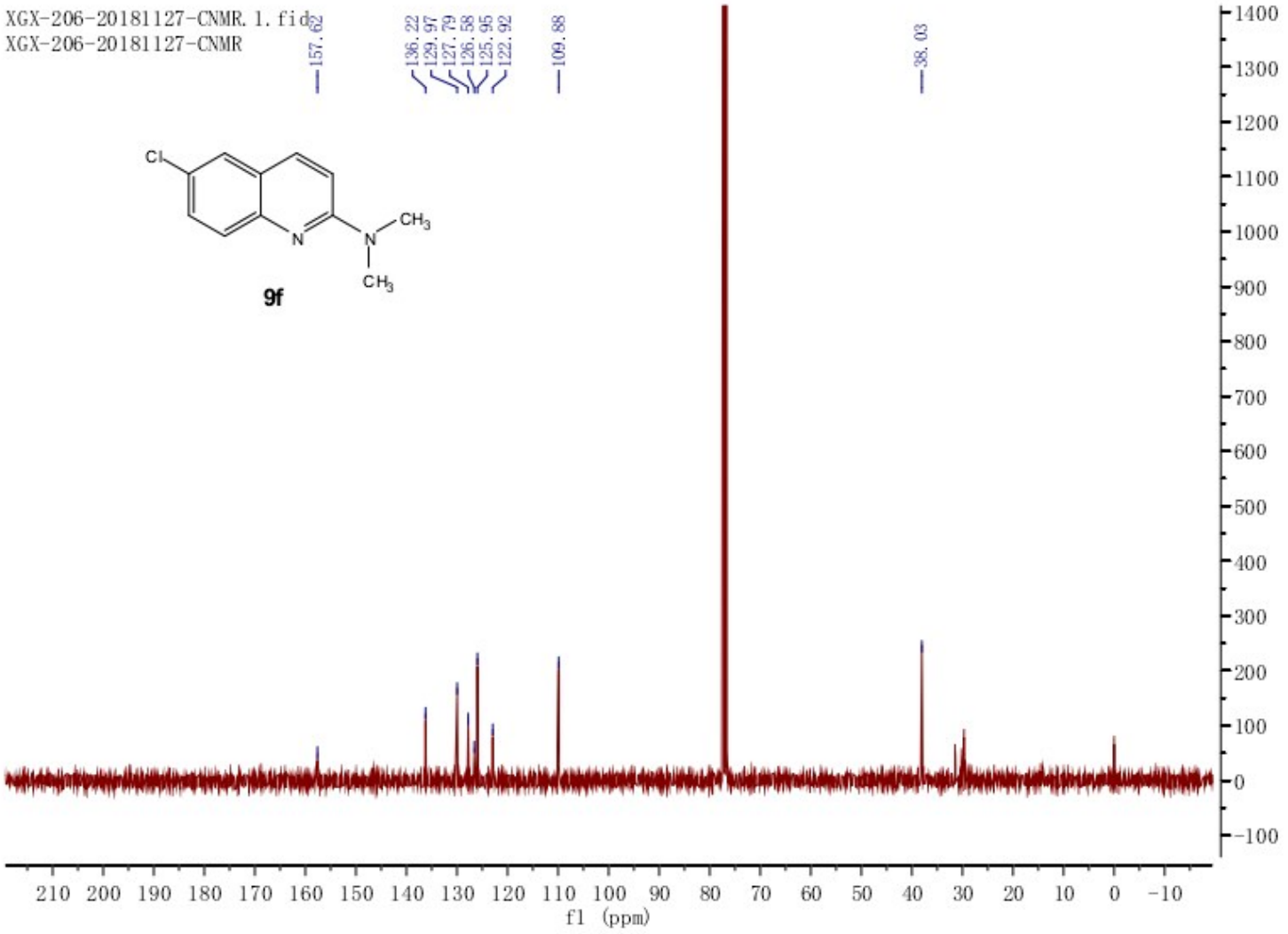

G-425-20170803-HNMR.1.fid
G-425-20170803-HNMR
7.84
7.82
7.42
7.38
7.10
7.08
7.08
6.88
6.85
3.21
2.65
CH3
N
N
CH3
CH3
9d
25000
20000
15000
10000
5000
0
0.96
2.01
1.00
1.01
6.04
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

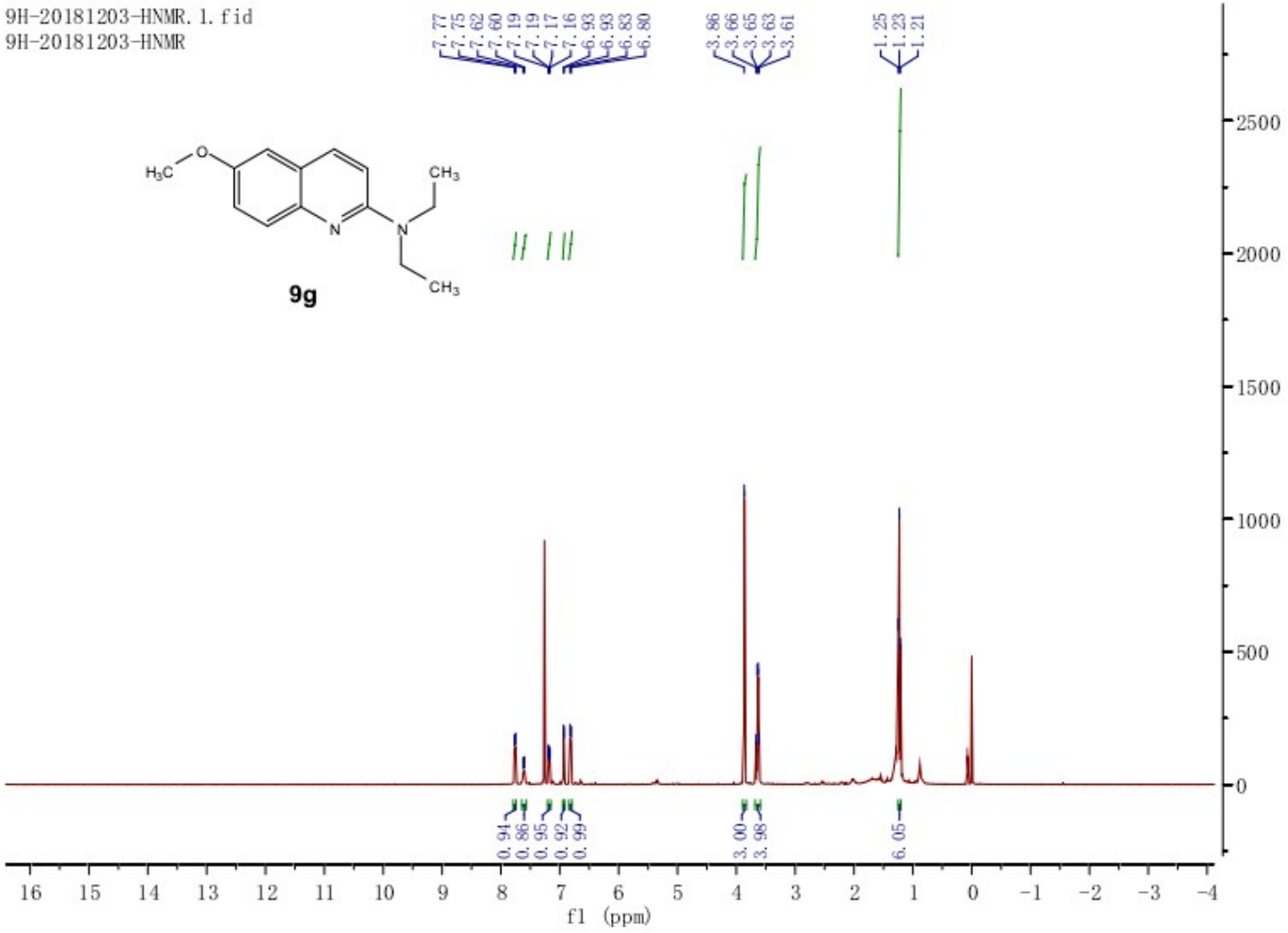

XGX-8JDMF-20171109-CNMR.fid
XGX-8JDMF-20171109-CNMR.fid
156.80
146.79
137.43
134.11
129.44
125.08
121.99
121.21
108.49
37.92
17.78
N
N
CH3
CH3
CH3
9d
1500
1000
500
0
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

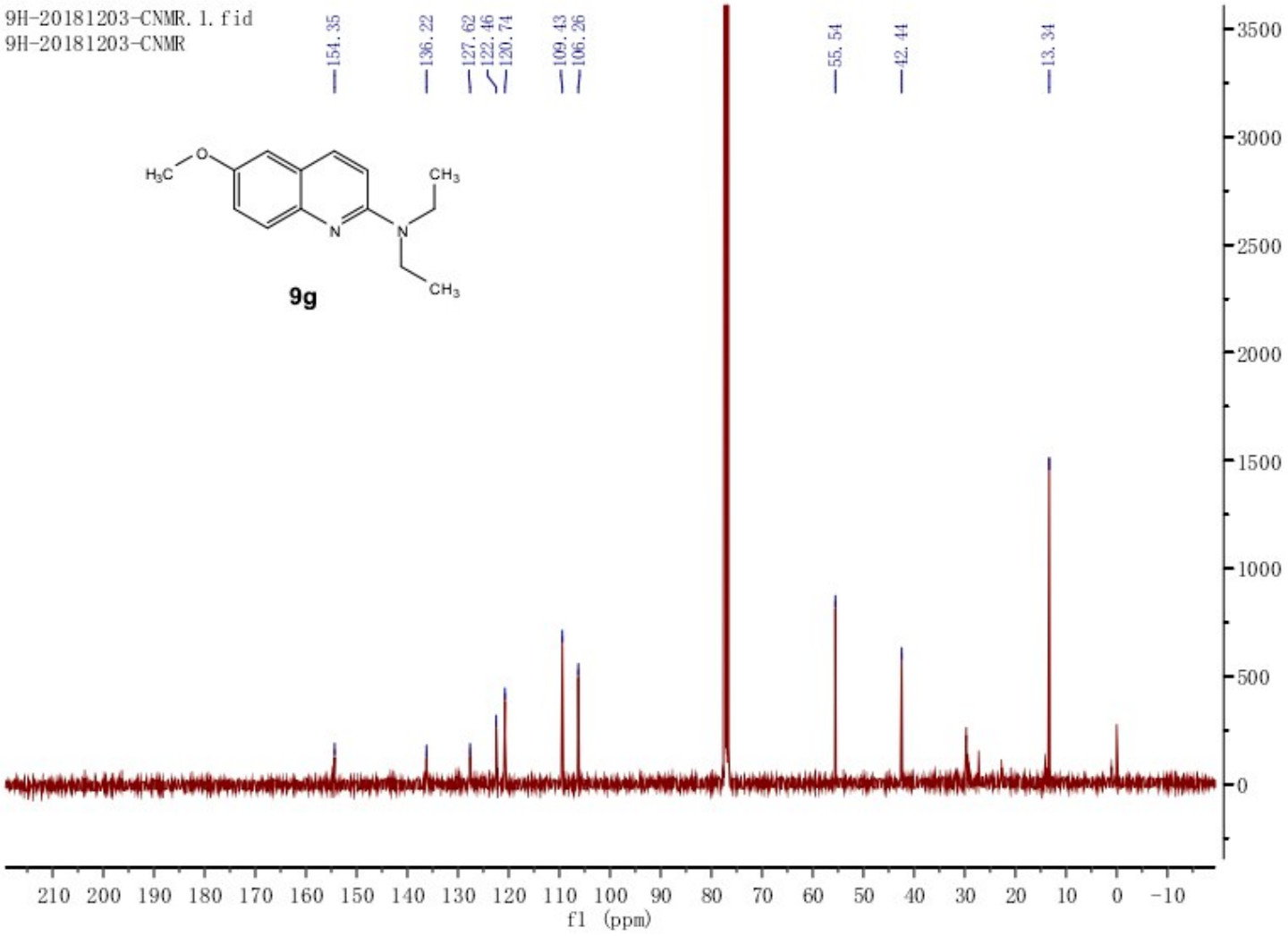

DT-11.26-4-20181129-HNMR.1.fid
DT-11.26-4-20181129-HNMR
7.81
7.78
7.67
7.64
7.26
7.22
7.21
7.20
7.19
6.95
6.94
6.89
6.87
3.87
3.20
O
H3C
N
N
CH3
CH3
9e
6000
5000
4000
3000
2000
1000
0
1.05
1.01
1.07
0.96
1.05
3.24
6.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

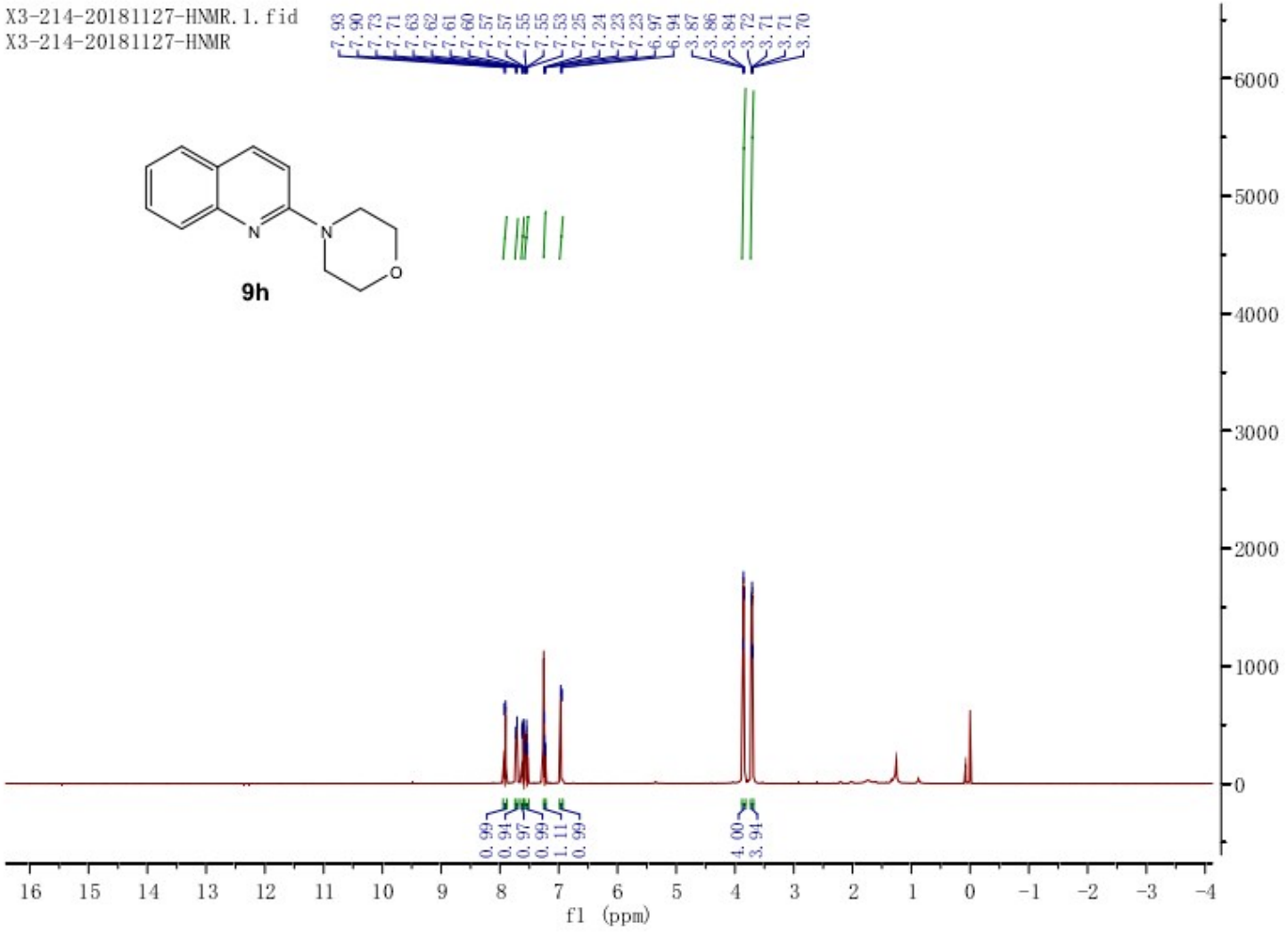

DT-11.26-4-20181129-CNMR.2.6
DT-11.26-4-20181129-CNMR
156.72
154.54
136.31
127.71
122.63
120.95
109.39
106.18
55.50
38.19
O
H3C
N
N
CH3
CH3
9e
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

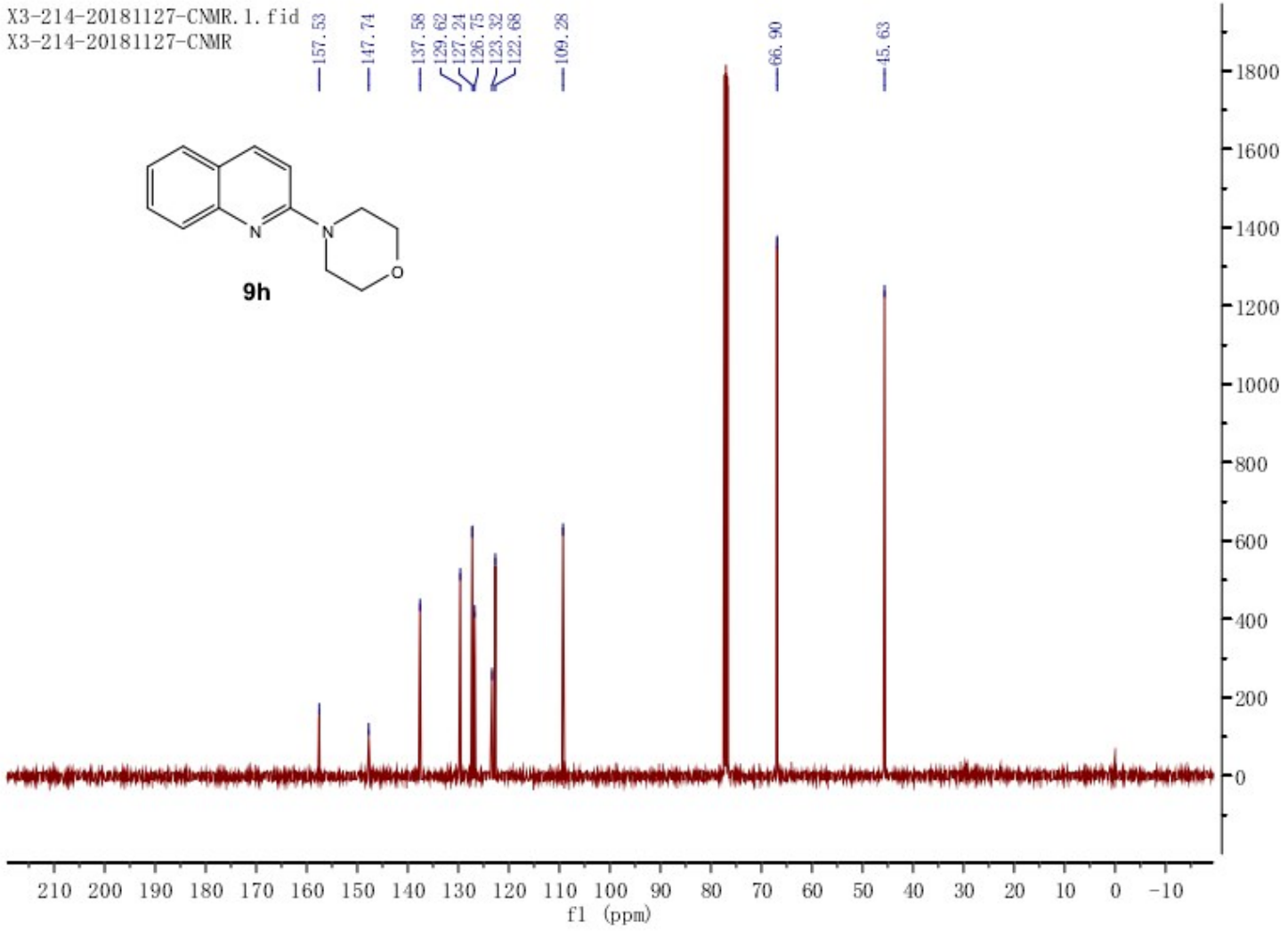

XGX-206-20181127-HNMR.1.fid
XGX-206-20181127-HNMR
7.78
7.75
7.64
7.62
7.55
7.54
7.45
7.45
7.43
7.42
7.26
6.91
6.89
3.22
Cl
N
N
CH3
CH3
9f
8000
7000
6000
5000
4000
3000
2000
1000
0
1.07
1.07
1.00
1.02
1.02
6.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

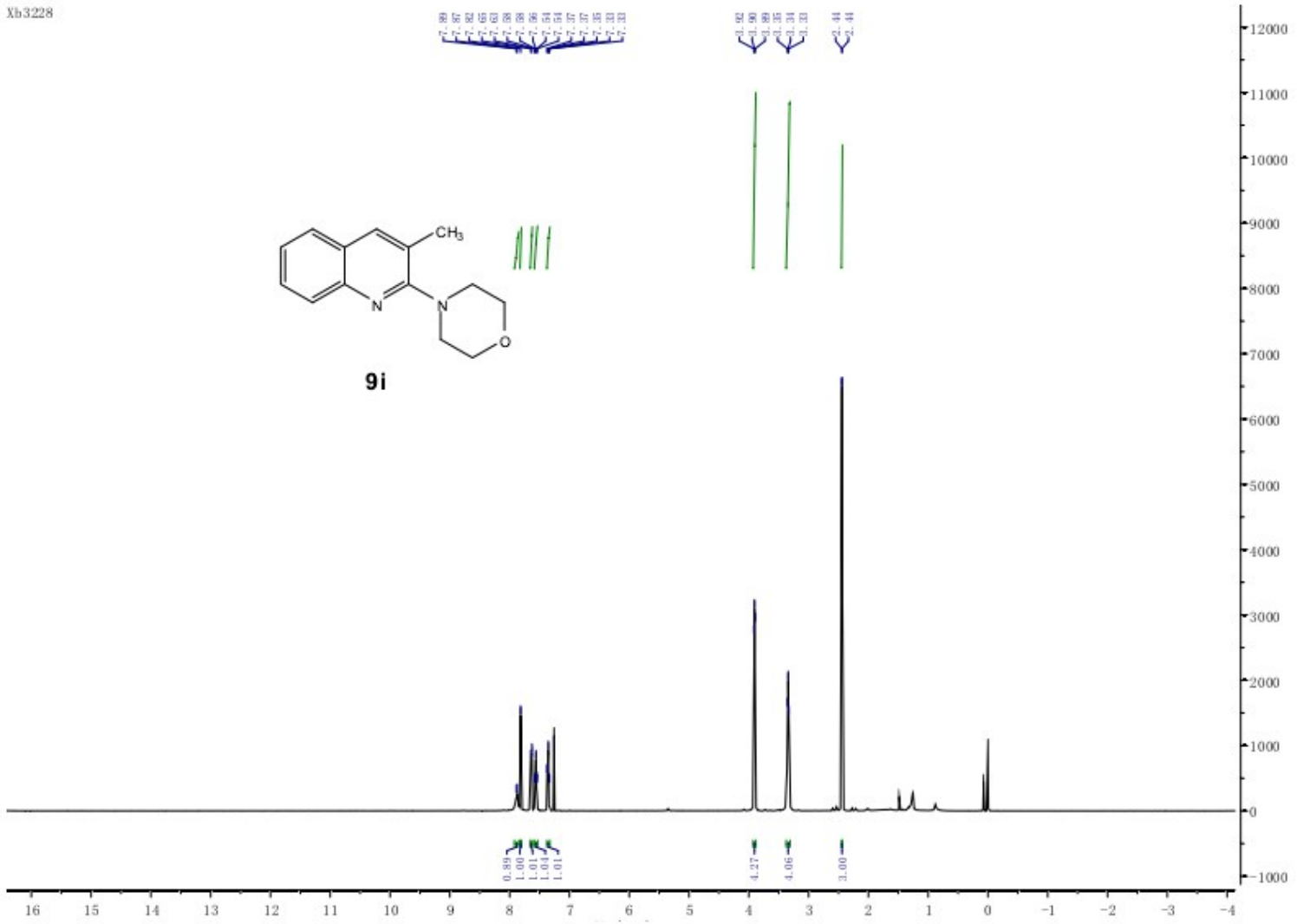

XGX-206-20181127-CNMR.1.fid
XGX-206-20181127-CNMR
157.62
136.22
129.97
127.79
126.58
125.95
122.92
109.88
38.03
Cl
N
N
CH3
CH3
9f
f1 (ppm)

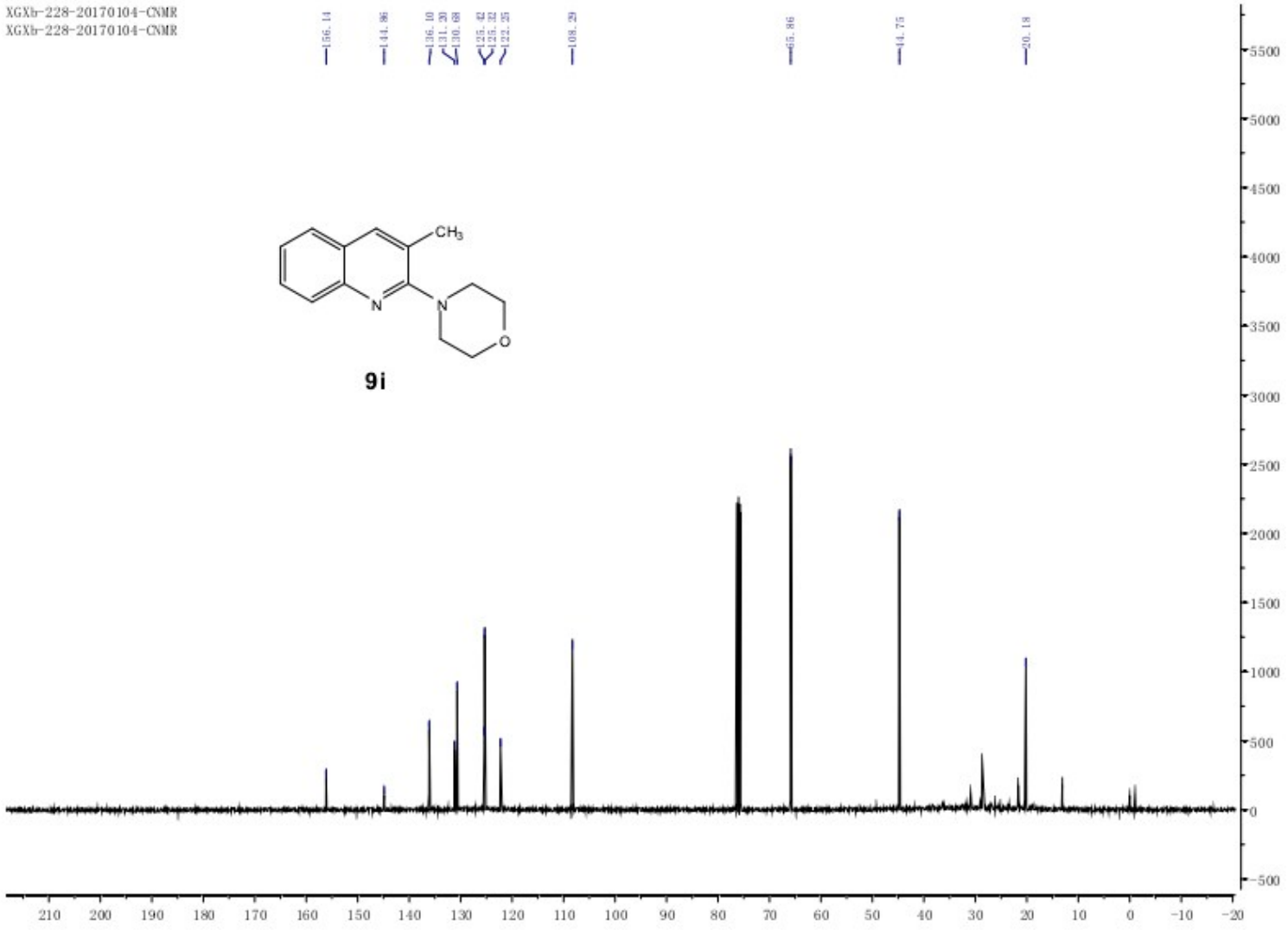

9H-20181203-HNMR.1.fid
9H-20181203-HNMR
7.77
7.75
7.62
7.60
7.19
7.19
7.17
7.16
6.93
6.93
6.83
6.80
3.86
3.66
3.65
3.63
3.61
1.25
1.23
1.21
H3C
O
N
N
CH3
CH3
9g
2500
2000
1500
1000
500
0
0.94
0.86
0.95
0.92
0.99
3.00
3.98
6.05
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

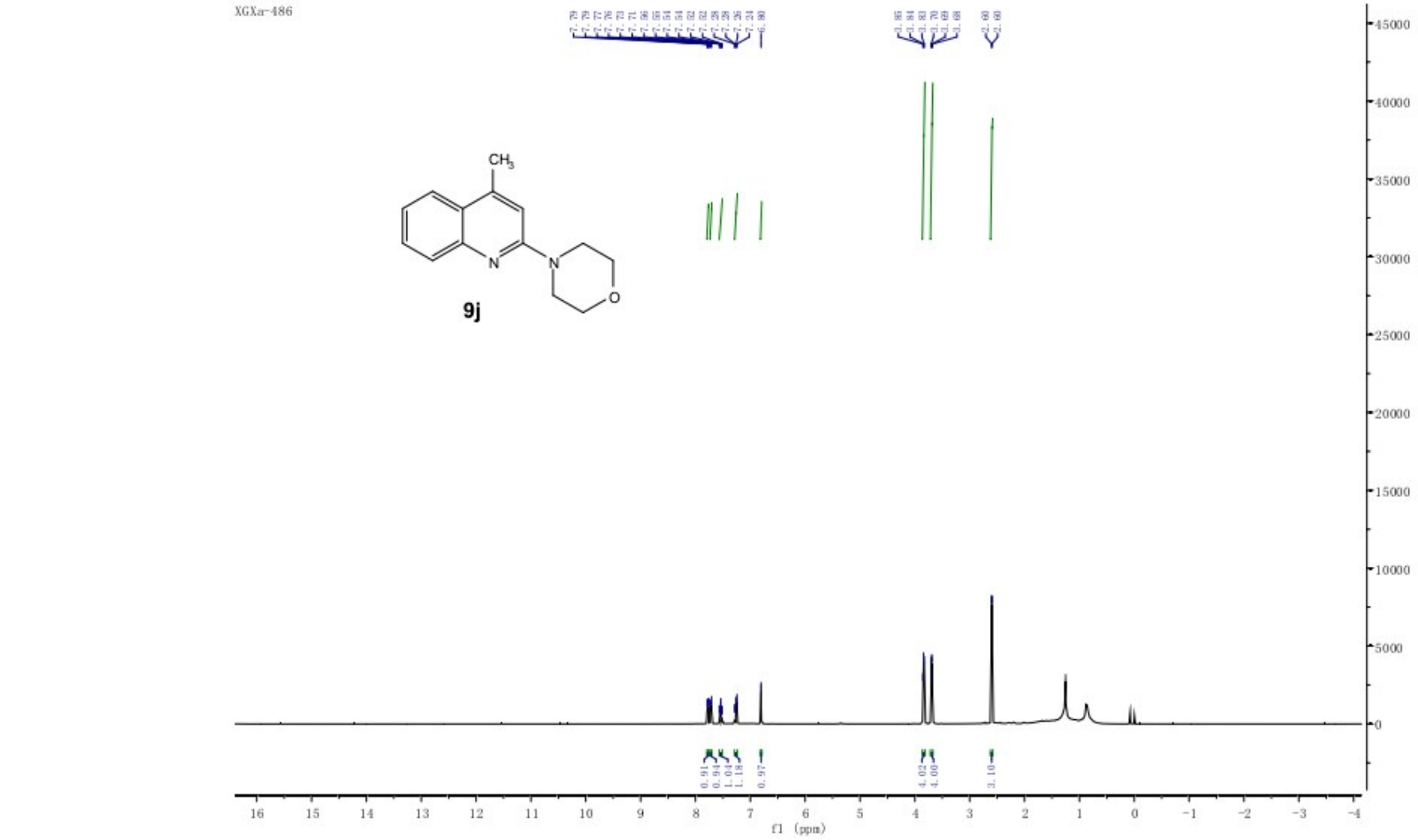

9H-20181203-CNMR.1.fid
9H-20181203-CNMR
154.35
136.22
127.62
122.46
120.74
109.43
106.26
55.54
42.44
13.34
H3C
O
N
N
CH3
CH3
9g
3500
3000
2500
2000
1500
1000
500
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

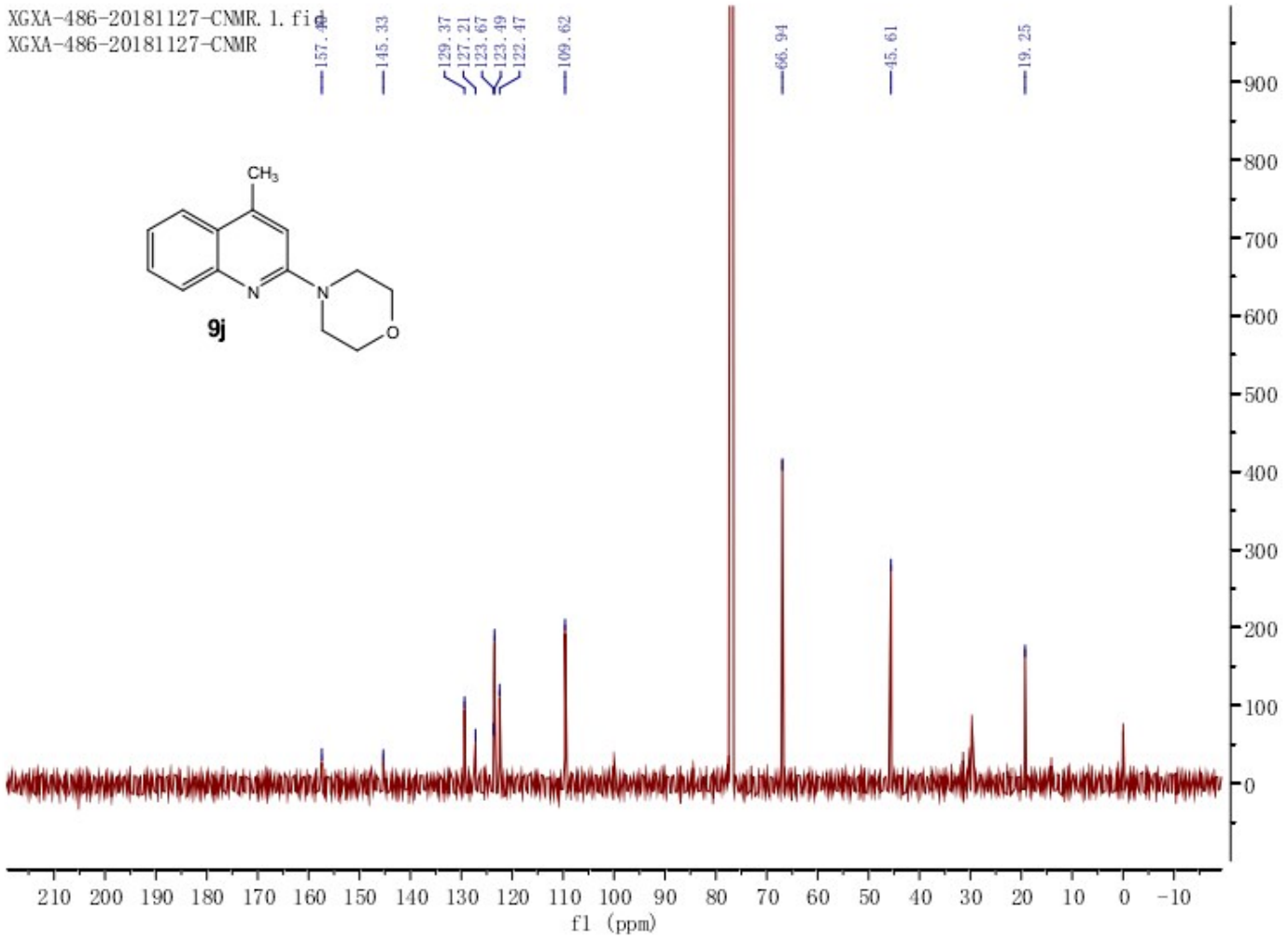

X3-214-20181127-HNMR.1.fid
X3-214-20181127-HNMR
7.93
7.90
7.73
7.71
7.63
7.62
7.61
7.60
7.57
7.57
7.55
7.55
7.53
7.25
7.24
7.23
7.23
6.97
6.94
3.87
3.86
3.84
3.72
3.71
3.71
3.70
9h
6000
5000
4000
3000
2000
1000
0
0.99
0.94
0.97
0.99
1.11
0.99
4.00
3.94
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

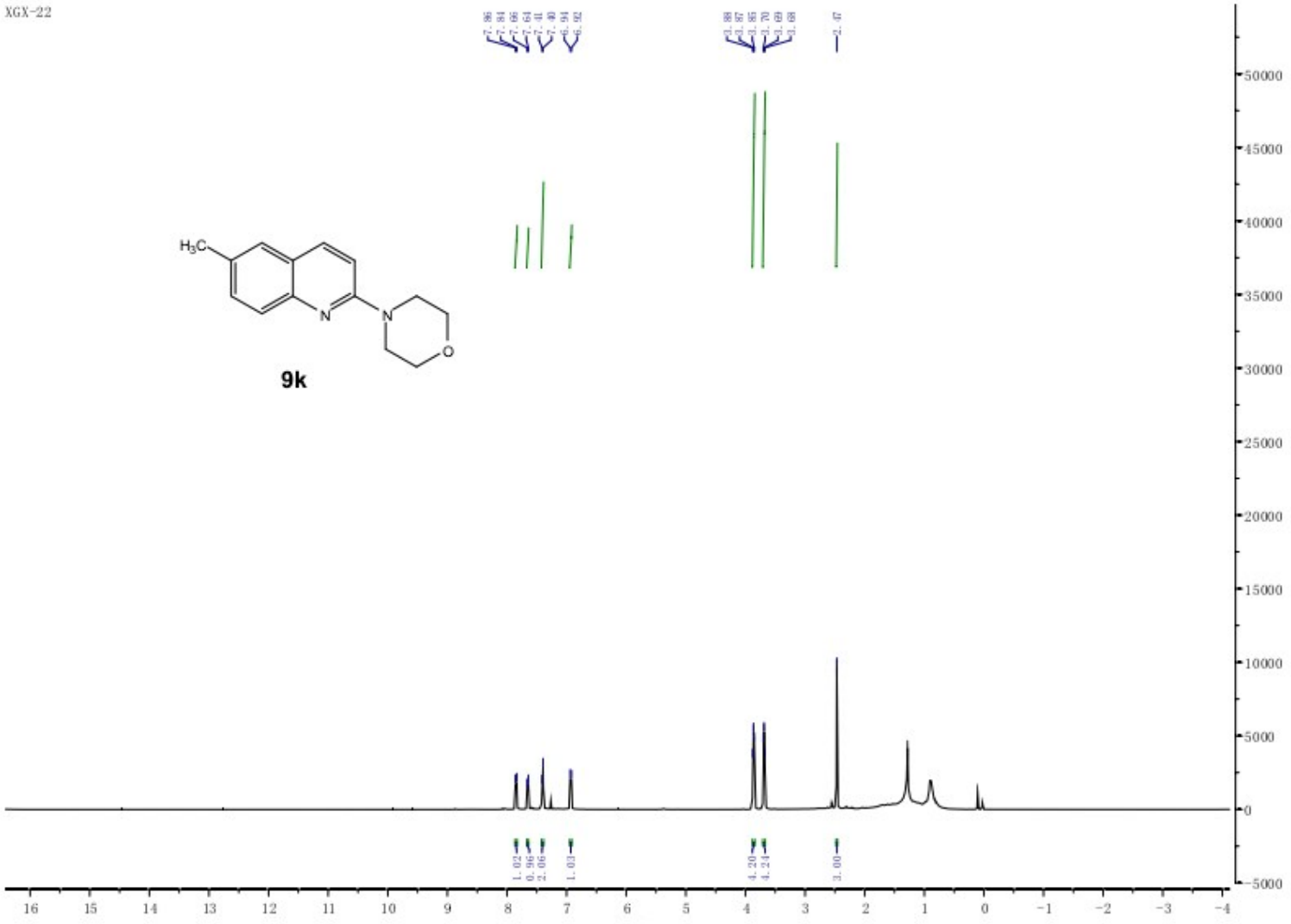

X3-214-20181127-CNMR.1.fid
X3-214-20181127-CNMR
157.53
147.74
137.58
129.62
127.24
126.75
123.32
122.68
109.28
66.90
45.63
9h
1800
1600
1400
1200
1000
800
600
400
200
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

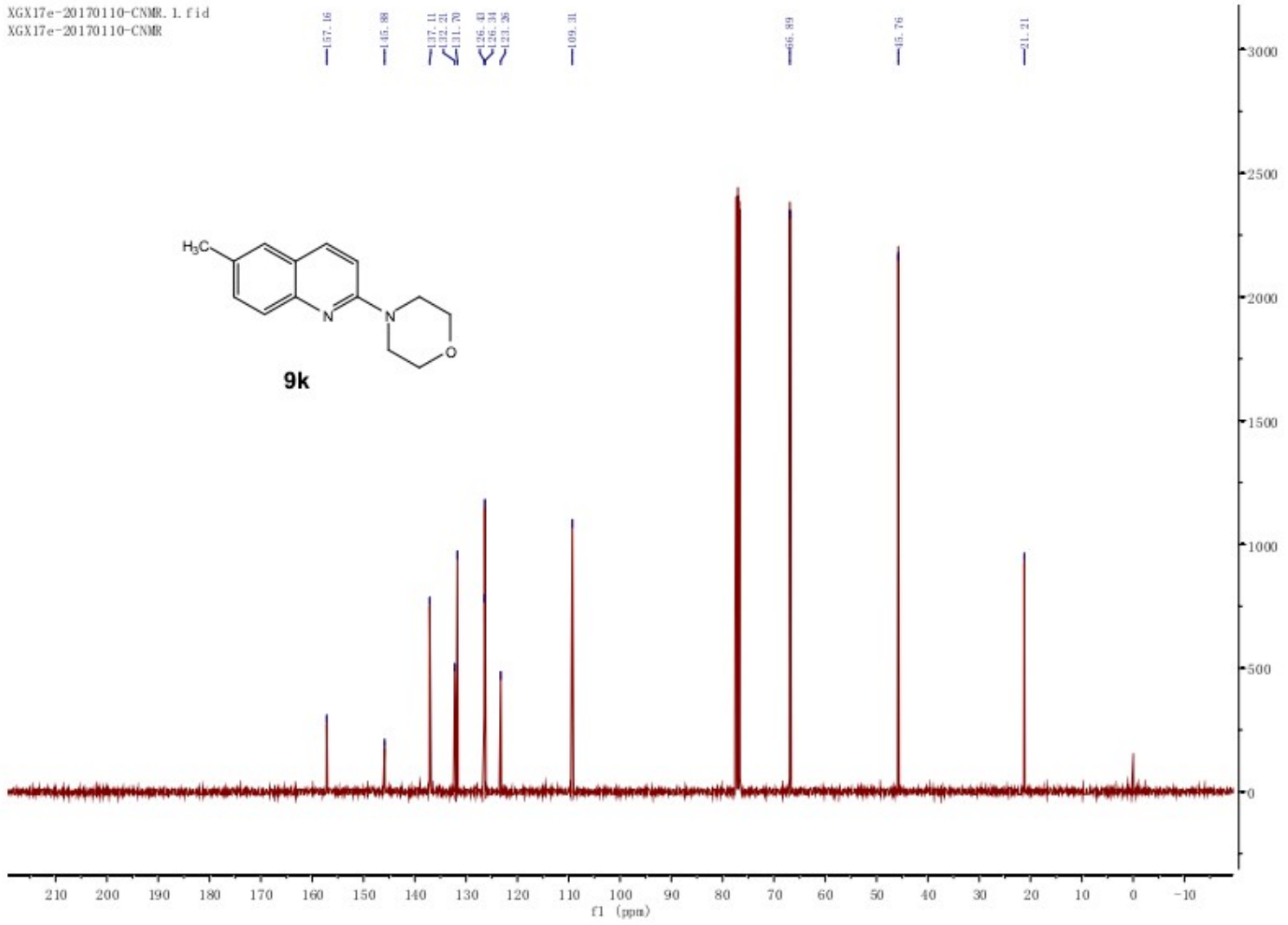

Xb3228
CH3
N
N
O
9i

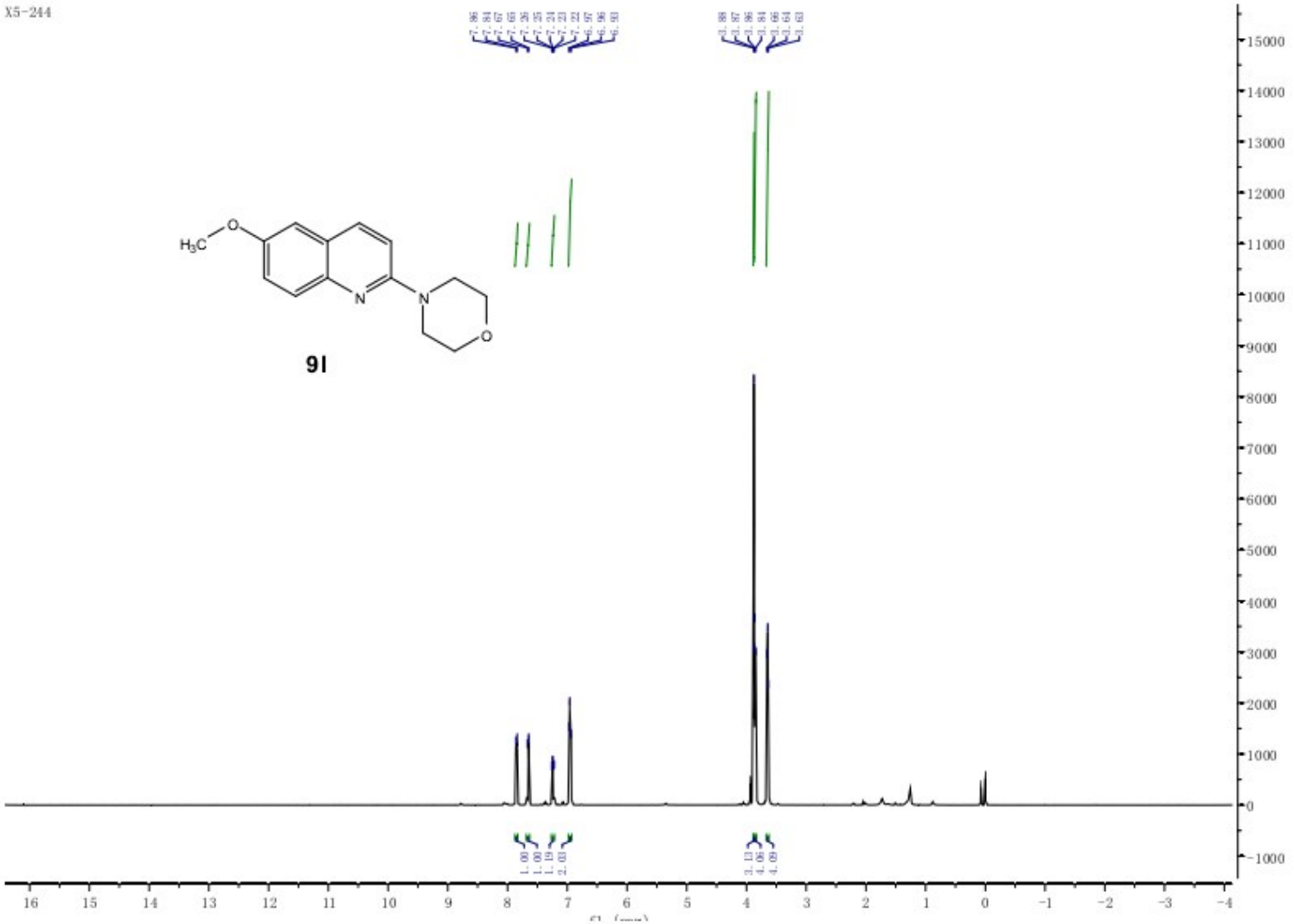

XGXb-228-20170104-CNMR
XGXb-228-20170104-CNMR
156.14
144.86
136.10
131.20
130.68
125.42
125.32
122.25
108.29
65.86
44.75
20.18
CH3
N
N
O
9i

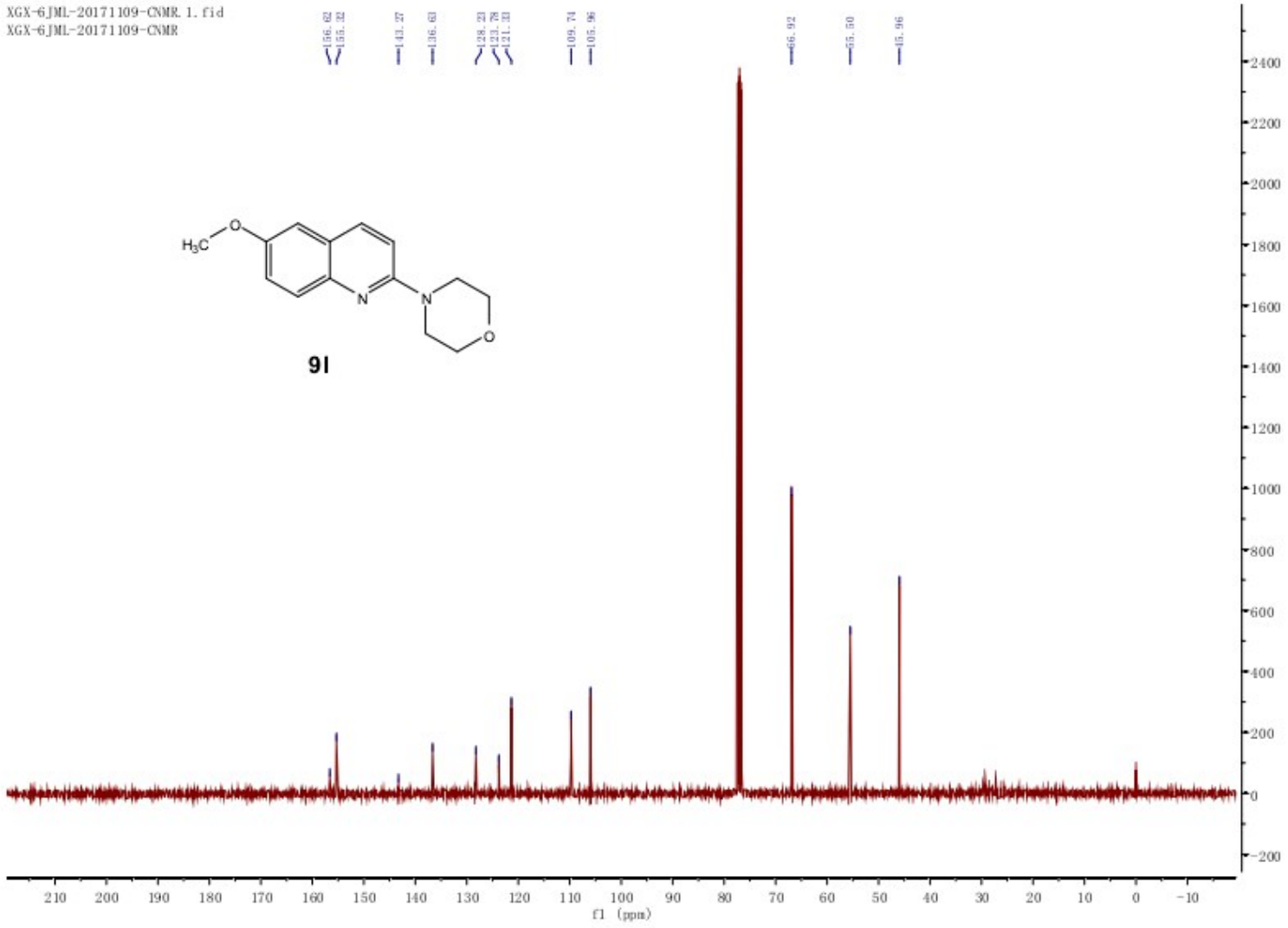

XGXa-486
CH3
N
N
O
9j
f1 (ppm)

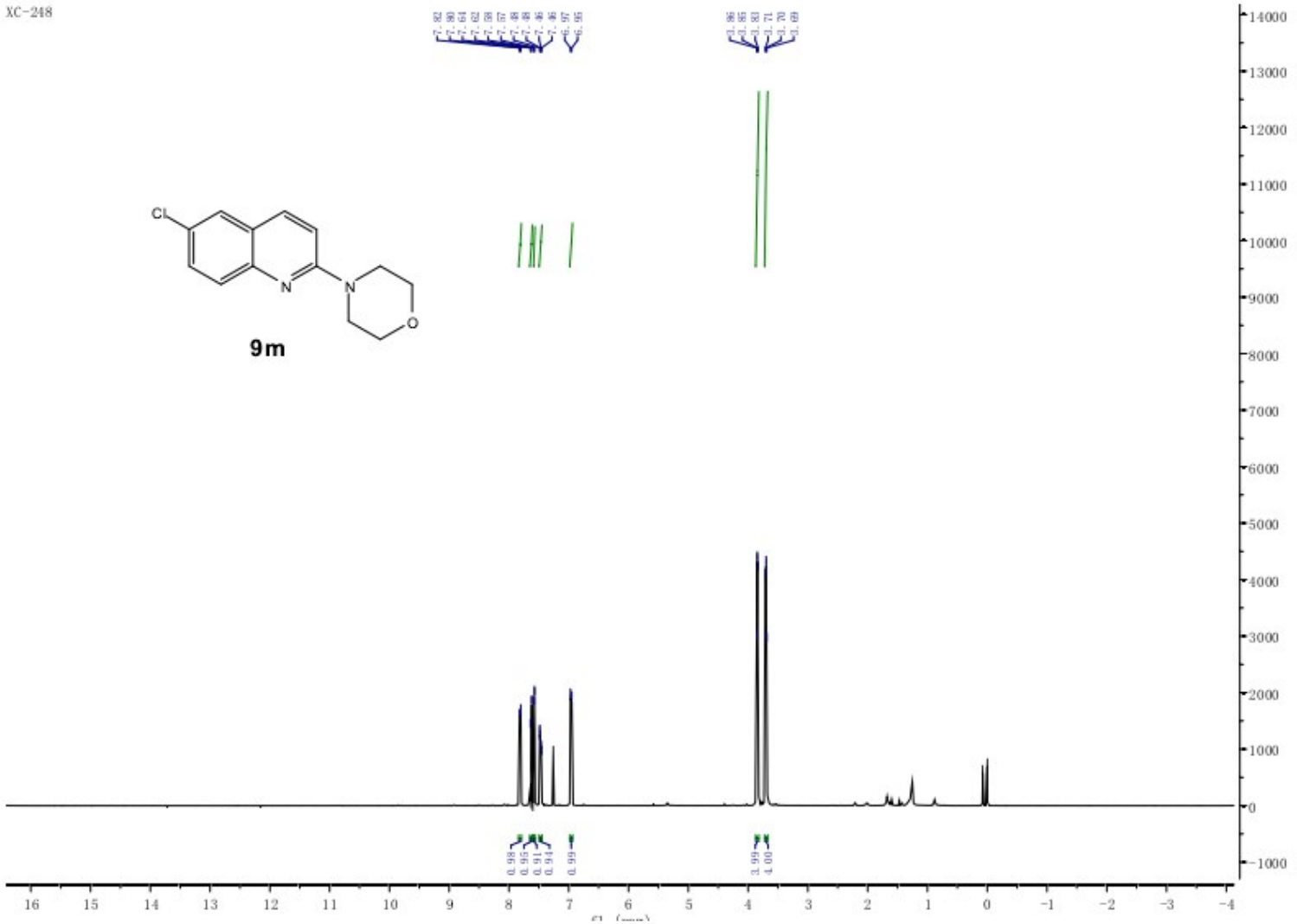

XGXA-486-20181127-CNMR.1.fid
XGXA-486-20181127-CNMR
157.40
145.33
129.37
127.21
123.67
123.49
122.47
109.62
66.94
45.61
19.25
CH3
N
N
O
9j
900
800
700
600
500
400
300
200
100
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

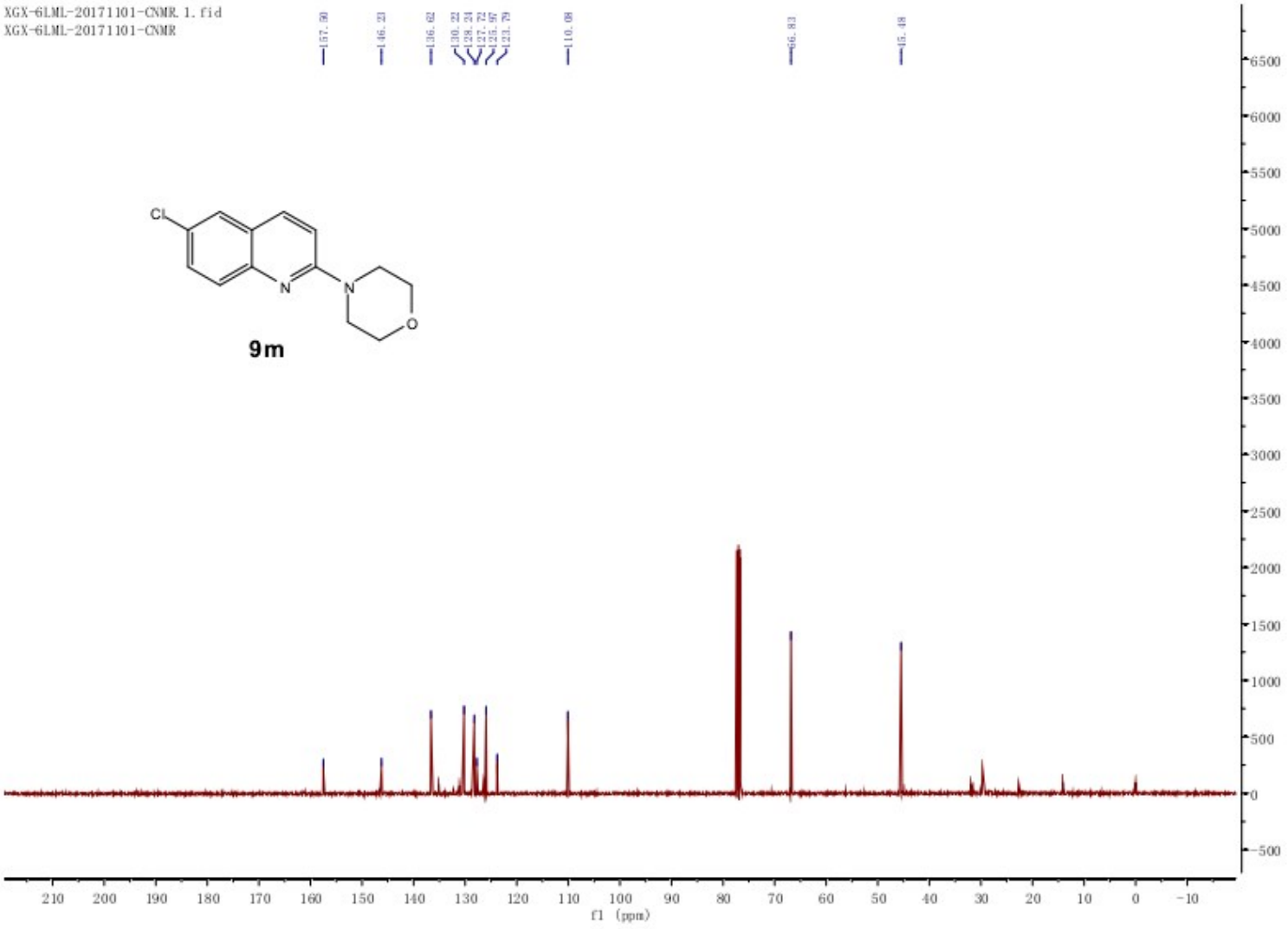

XGX-22
7.98
7.94
7.66
7.64
7.41
7.40
6.94
6.92
3.88
3.87
3.85
3.70
3.69
3.68
2.47
H3C
N
N
O
9k
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
1.02
0.96
2.06
1.03
4.20
4.24
3.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

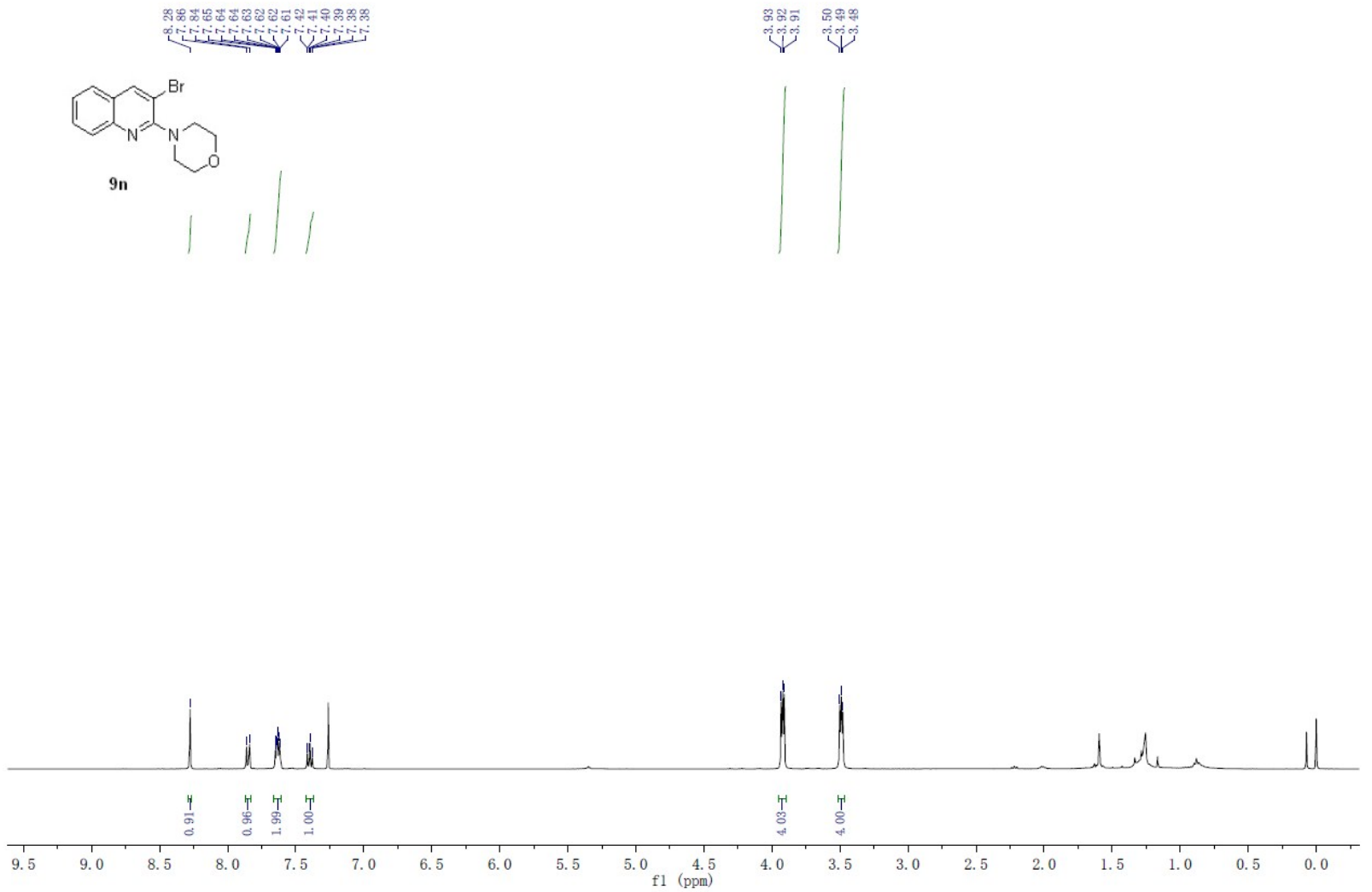

XGX17e-20170110-CNMR.1.fid
XGX17e-20170110-CNMR
157.16
145.88
137.11
132.21
131.70
126.43
126.34
123.36
109.31
66.89
45.76
21.21
H3C
N
N
O
9k
3000
2500
2000
1500
1000
500
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

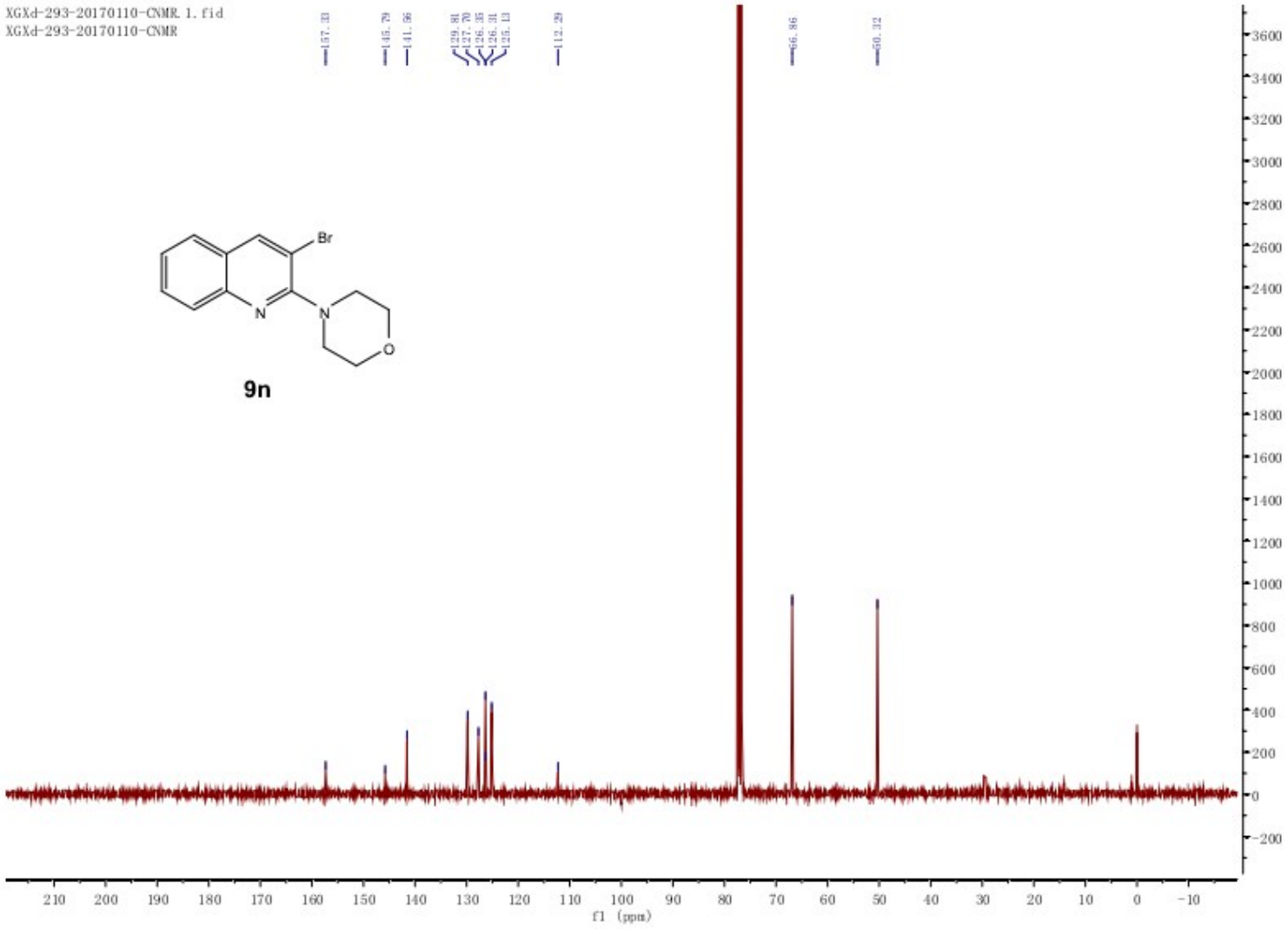

X5-244
H3C
O
N
N
O
9l

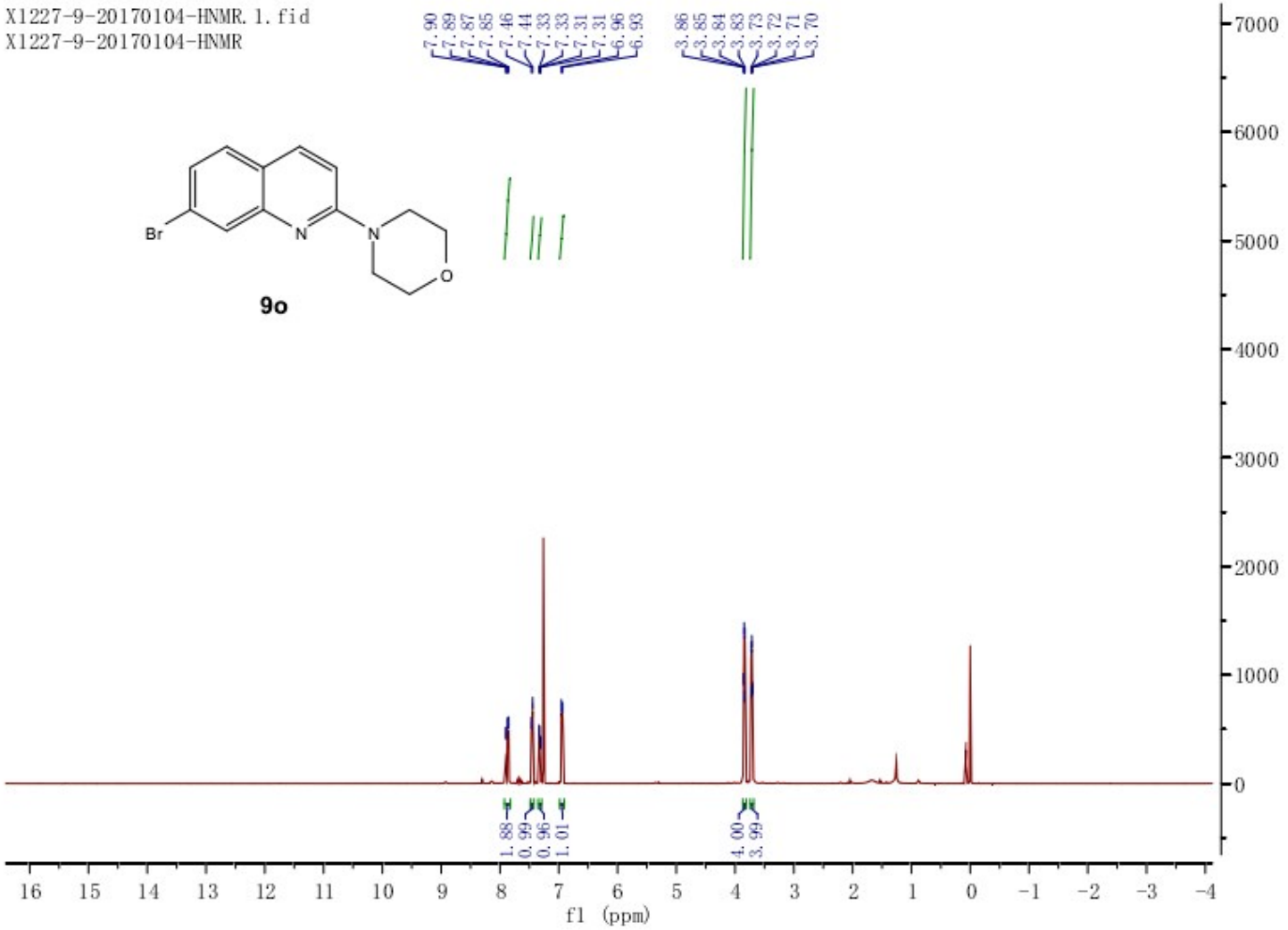

XGX-6JML-20171109-CNMR.1.fid
XGX-6JML-20171109-CNMR
156.62
155.32
143.27
136.63
128.23
123.78
121.33
109.74
105.96
66.92
55.50
45.96
H3C
O
N
N
O
91
f1 (ppm)

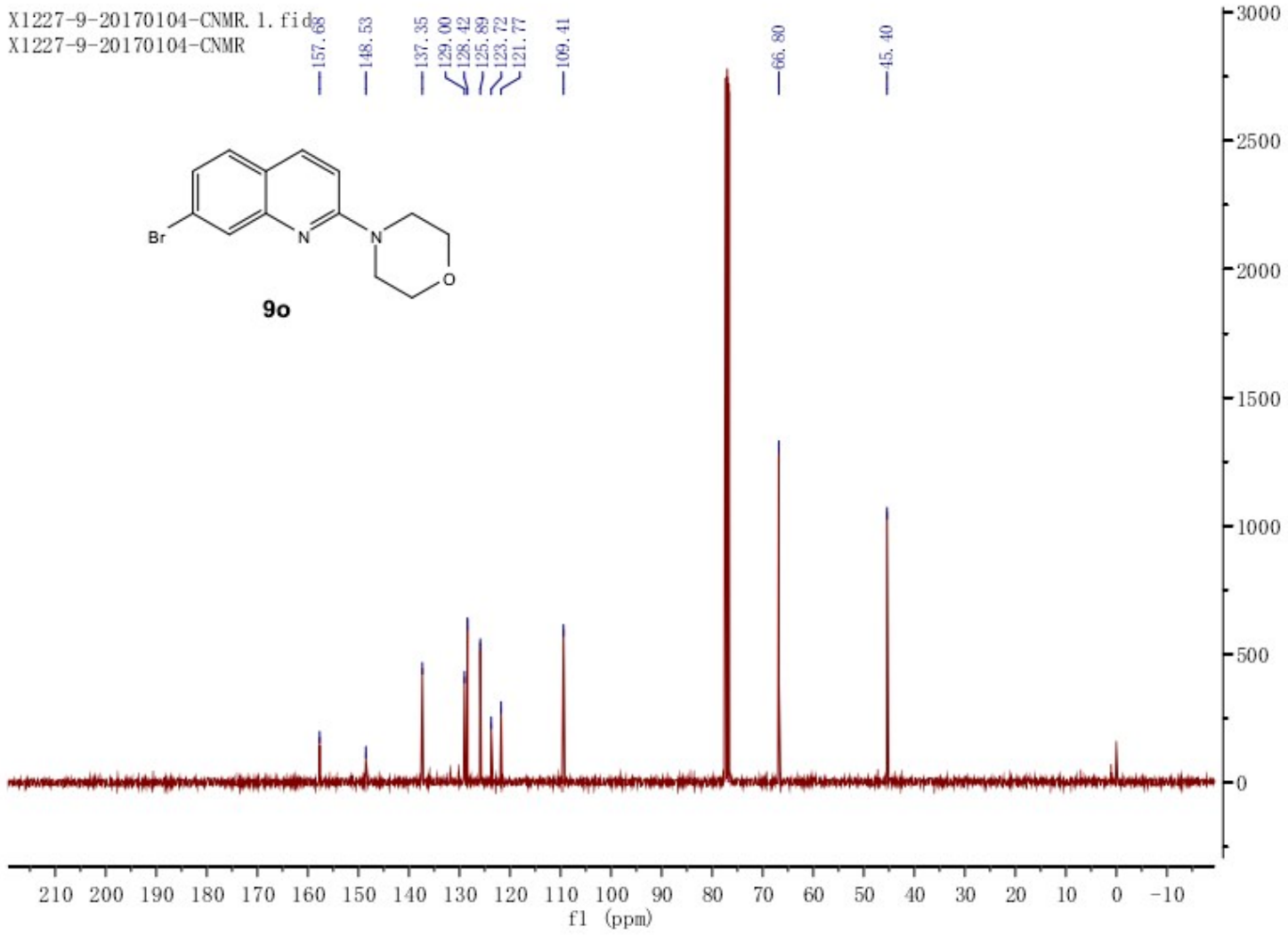

XC-248
Cl
N
N
O
9m


XGX-6LML-20171101-CNMR 1.fid
XGX-6LML-20171101-CNMR
157.90
146.23
136.62
130.22
128.24
127.72
125.97
123.79
110.08
66.83
45.48
Cl
N
N
O
9m
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
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)


8.28
7.86
7.84
7.65
7.64
7.64
7.63
7.62
7.62
7.61
7.42
7.41
7.40
7.39
7.38
7.38
3.93
3.92
3.91
3.50
3.49
3.48
Br
N
N
O
9n
0.91
0.96
1.99
1.00
4.03
4.00
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
f1 (ppm)


XGXd-293-20170110-CNMR 1.fid
XGXd-293-20170110-CNMR
157.33
145.79
141.86
129.81
127.70
126.35
126.31
125.13
112.29
66.86
50.32
Br
N
N
O
9n
f1 (ppm)


X1227-9-20170104-HNMR.1.fid
X1227-9-20170104-HNMR
7.90
7.89
7.87
7.85
7.46
7.44
7.33
7.33
7.31
7.31
6.96
6.93
3.86
3.85
3.84
3.83
3.73
3.72
3.71
3.70
Br
N
N
O
9o
7000
6000
5000
4000
3000
2000
1000
0
1.88
0.99
0.96
1.01
4.00
3.99
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


X1227-9-20170104-CNMR.1.fid
X1227-9-20170104-CNMR
157.68
148.53
137.35
129.00
128.42
125.89
123.72
121.77
109.41
66.80
45.40
Br
N
N
O
9o
3000
2500
2000
1500
1000
500
0
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)