SUPPORTING INFORMATION

Cobalt-Catalyzed, Aminoquinoline-Directed Coupling of sp^2 C-H Bonds with Alkenes

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General considerations

Reactions were performed using standard glassware or were run in 2-dram vials with PTFE/Liner screw caps and 8-dram vials using w/polyseal screw caps. Column chromatography was performed on 60Å silica gel (Dynamic Adsorbents Inc.). ^{1}H, ^{13}C, ^{19}F-NMR and 2D-NMR spectra were recorded on JEOL EC-400 and JEOL EC-500 spectrometers using residual solvent peak as a reference. HRMS analysis was performed by chemical ionization (CI) using Micromass Autospec Ultima spectrometer at the Mass Spectrometry Facility of the Department of Chemistry and Biochemistry of University of Texas-Austin. IR- spectra were obtained using a Perkin Elmer Spectrum 100 FT-IR spectrometer. Analytical thin layer chromatography was performed on silica gel IB-F (Baker-flex) by J. T. Baker. All procedures were performed under ambient air unless otherwise noted. Reagents and starting materials were obtained from commercial sources and used without further purification unless otherwise noted. Room temperature is

Substrate synthesis

Amides were synthesized according to literature procedures from 8-aminoquinoline and corresponding acyl chlorides (Procedure **A**, amides: *N*-(quinolin-8-yl)benzamide, 4-trifluoromethyl-*N*-(quinolin-8-yl)benzamide, 4-bromo-*N*-(quinolin-8-yl)benzamide, 4-nitro-*N*-(quinolin-8-yl)benzamide, 3-iodo-*N*-(quinolin-8-yl)benzamide, 4-methyl-*N*-(quinolin-8-yl)benzamide, *N*-(quinolin-8-yl)furan-2-carboxamide), *N*-(quinolin-8-yl)thiophene-2-carboxamide[1] or carboxylic acids (Procedure **B**, amides: 2-methoxy-*N*-(quinolin-8-yl)benzamide and *N*-(quinolin-8-yl)cinnamamide), *N*-(quinolin-8-yl)methacrylamide[2]. *N*-(5-Methoxyquinolin-8-yl)benzamide was prepared from 8-amino-5-methoxyquinoline hydrochloride and benzoyl chloride using Procedure **A**.

Procedure A:

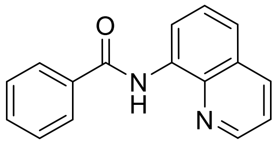
Synthesis of N-(quinolin-8-yl)benzamide is representative.

To a solution of 8-aminoquinoline (3.00 g, 21 mmol) and *N*,*N*-dimethyl-4-aminopyridine (80 mg, 0.65 mmol) in anhydrous CH_2Cl_2 (30 mL) under nitrogen Et_3N (3.3 mL, 24 mmol, 1.2 equiv) was added and resulting solution was cooled to 0 °C. Benzoyl chloride (2.3 mL, 20 mmol) was added dropwise and reaction mixture was stirred at room temperature overnight. The mixture was quenched with water (30 mL) and extracted with CH_2Cl_2 (3 x 20 mL). Combined organic phase was

[1] Nishino, M.; Hirano, K.; Satoh, T.; Miura, M. *Angew. Chem.*, *Int. Ed*. **2013**, *52*, 4457.
[2] Tran, L. D.; Popov, I.; Daugulis, O. *J. Am. Chem. Soc*. **2012**, *134*, 18237.

dried over $MgSO_4$ and filtered. Concentration in vacuum followed by recrystallization from toluene afforded 4.6 g (94%) of *N*-(quinolin-8-yl)benzamide as a white solid.

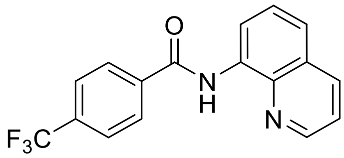
***N*-(Quinolin-8-yl)benzamide**



This compound is known.[1]

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.76 (s, 1H), 8.95 (dd, *J* = 7.6, 1.3 Hz, 1H), 8.85 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.19 (dd, *J* = 8.3, 1.6 Hz, 1H), 8.14-8.04 (m, 2H), 7.68 – 7.52 (m, 5H) and 7.48 (dd, *J* = 8.2, 4.2 Hz, 1H).

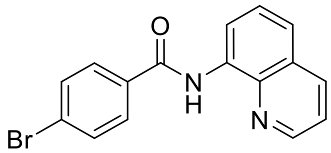
4-Trifluoromethyl-*N*-(quinolin-8-yl)benzamide



8-Aminoquinoline (3.00 g, 21 mmol), *N*,*N*-dimethyl-4-aminopyridine (80 mg, 0.65 mmol), Et_3N (3.3 mL, 24 mmol, 1.2 equiv), 4-trifluoromethylbenzoyl chloride (3.0 mL, 20 mmol), CH_2Cl_2 (30 mL). Recrystallization from hexanes/EtOAc 4:1 afforded 5.50 g (88%) of 4-trifluoromethyl-*N*-(quinolin-8-yl)benzamide as a white solid. This compound is known.[1]

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.80 (s, 1H), 8.92 (dd, *J* = 7.2, 1.7 Hz, 1H), 8.86 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.25 – 8.16 (m, 3H), 7.82 (d, *J* = 8.1 Hz, 2H), 7.65 – 7.56 (m, 2H) and 7.51 (dd, *J* = 8.3, 4.2 Hz, 1H).

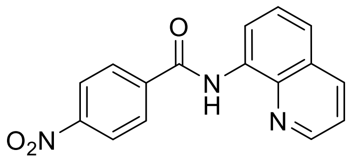
4-Bromo-*N*-(quinolin-8-yl)benzamide



8-Aminoquinoline (3.00 g, 21 mmol), *N*,*N*-dimethyl-4-aminopyridine (80 mg, 0.65 mmol), Et_3N (3.3 mL, 24 mmol, 1.2 equiv), 4-bromobenzoyl chloride (4.39 g, 20 mmol), CH_2Cl_2 (30 mL). Recrystallization from toluene afforded 5.84 g (89%) of 4-bromo-*N*-(quinolin-8-yl)benzamide as a white solid. This compound is known.[2]

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.71 (s, 1H), 8.90 (dd, *J* = 7.4, 1.5 Hz, 1H), 8.85 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.19 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.97 – 7.91 (m, 2H), 7.71 – 7.65 (m, 2H), 7.62 – 7.53 (m, 2H) and 7.48 (dd, *J* = 8.3, 4.2 Hz, 1H).

4-Nitro-*N*-(quinolin-8-yl)benzamide

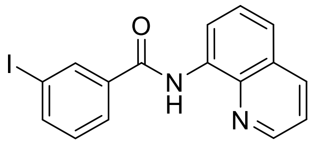


8-Aminoquinoline (3.00 g, 21 mmol), *N*,*N*-dimethyl-4-aminopyridine (80 mg, 0.65 mmol), Et_3N (3.3 mL, 24 mmol, 1.2 equiv), 4-nitrobenzoyl chloride (3.71 g, 20 mmol), CH_2Cl_2 (30 mL).

Recrystallization from hexanes/EtOAc 4:1 afforded 5.67 g (97%) of 4-nitro-*N*-(quinolin-8-yl)benzamide as a yellow solid. This compound is known.[3]

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.83 (s, 1H), 8.91 (dd, *J* = 6.4, 2.5 Hz, 1H), 8.87 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.45 – 8.36 (m, 2H), 8.26-8.22 (m, 3H), 7.64-7.60 (m, 2H) and 7.52 (dd, *J* = 8.3, 4.2 Hz, 1H).

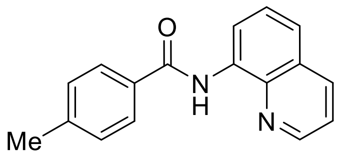
3-Iodo-*N*-(quinolin-8-yl)benzamide



8-Aminoquinoline (1.50 g, 10.5 mmol), *N*,*N*-dimethyl-4-aminopyridine (40 mg, 0.33 mmol), Et_3N (1.65 mL, 12 mmol, 1.2 equiv), 3-iodobenzoyl chloride (2.67 g, 10 mmol), CH_2Cl_2 (15 mL). Recrystallization from hexanes/EtOAc 2:1 afforded 3.46 g (93%) of 3-iodo-*N*-(quinolin-8-yl)benzamide as a white solid. This compound is known.[4]

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.67 (s, 1H), 8.90 (dd, *J* = 7.4, 1.6 Hz, 1H), 8.86 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.41 (t, *J* = 1.7 Hz, 1H), 8.20 (dd, *J* = 8.3, 1.6 Hz, 1H), 8.02 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.91 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.63 – 7.54 (m, 2H), 7.49 (dd, *J* = 8.2, 4.2 Hz, 1H) and 7.29 (t, *J* = 7.8 Hz, 1H).

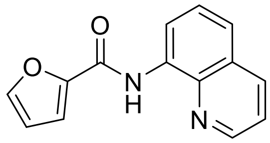
4-Methyl-*N*-(quinolin-8-yl)benzamide



8-Aminoquinoline (6.00 g, 42 mmol), *N*,*N*-dimethyl-4-aminopyridine (160 mg, 1.3 mmol), Et_3N (6.6 mL, 48 mmol, 1.2 equiv), 4-toluoyl chloride (5.3 mL, 40 mmol), CH_2Cl_2 (60 mL). Recrystallization from toluene afforded 9.0 g (86%) of amide 4-methyl-*N*-(quinolin-8-yl)benzamide as a white solid. This compound is known.[5]

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.73 (s, 1H), 8.94 (dd, *J* = 7.6, 1.3 Hz, 1H), 8.85 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.18 (dd, *J* = 8.3, 1.7 Hz, 1H), 8.02 – 7.96 (m, 2H), 7.59 (t, *J* = 7.9 Hz, 1H), 7.53 (dd, *J* = 8.3, 1.3 Hz, 1H), 7.47 (dd, *J* = 8.2, 4.2 Hz, 1H), 7.35 (d, *J* = 7.8 Hz, 2H), 2.45 (s, 3H).

***N*-(Quinolin-8-yl)furan-2-carboxamide**



8-Aminoquinoline (1.50 g, 10.5 mmol), *N*,*N*-dimethyl-4-aminopyridine (40 mg, 0.33 mmol), Et_3N (1.65 mL, 12 mmol, 1.2 equiv), 2-furoyl chloride (1.0 mL, 10 mmol), CH_2Cl_2 (15 mL). Recrystallization from hexanes/EtOAc 4:1

[3] Truong, T.; Klimovica, K.; Daugulis, O. *J. Am. Chem. Soc*. **2013**, *135*, 9342.

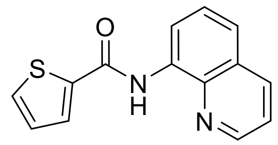
[4] Zaitsev, V. G.; Shabashov, D.; Daugulis, O. *J. Am. Chem. Soc*. **2005**, *127*, 13154.

[5] Suess, A. M.; Ertem, M. Z.; Cramer, C. J.; Stahl, S. S. *J. Am.Chem. Soc.* **2013**, *135*, 9797.

afforded 1.93 g (81%) of *N*-(quinolin-8-yl)furan-2-carboxamide as a white solid. This compound is known.[1]

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.77 (s, 1H), 8.96 – 8.80 (m, 2H), 8.16 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.62 (dd, *J* = 1.7, 0.7 Hz, 1H), 7.59 – 7.51 (m, 2H), 7.46 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.31 (dd, *J* = 3.5, 0.7 Hz, 1H) and 6.58 (dd, *J* = 3.5, 1.7 Hz, 1H).

_N_-(Quinolin-8-yl)thiophene-2-carboxamide



8-Aminoquinoline (1.50 g, 10.5 mmol), *N*,*N*-dimethyl-4-aminopyridine (40 mg, 0.33 mmol), Et_3N (1.65 mL, 12 mmol, 1.2 equiv), 2-thiophenecarbonyl chloride (1.1 mL, 10 mmol), CH_2Cl_2 (30 mL). Recrystallization from hexanes/EtOAc 4:1 afforded 2.33 g (92%) of *N*-(quinolin-8-yl)thiophene-2-carboxamide as a white solid. This compound is known.[6]

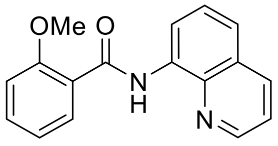
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.61 (s, 1H), 8.90 – 8.80 (m, 2H), 8.18 (dd, J = 8.3, 1.6 Hz, 1H), 7.84 (dd, J = 3.7, 1.1 Hz, 1H), 7.62 – 7.56 (m, 2H), 7.54 (dd, J = 8.3, 1.4 Hz, 1H), 7.48 (dd, J = 8.2, 4.2 Hz, 1H), 7.19 (dd, J = 5.0, 3.7 Hz, 1H).

Procedure B:

Synthesis of 2-methoxy-N-(quinolin-8-yl)benzamide is representative.

o-Anisic acid (2.28 g, 15 mmol, 1.5 equiv) and Et_3N (4.8 mL, 35 mmol, 3.5 equiv) were dissolved in CH_2Cl_2 (30 mL), flask was flushed with nitrogen and the resulting mixture was cooled to 0 °C. Ethyl chloroformate (1.4 mL, 15 mmol, 1.5 equiv) was added dropwise and solution was stirred at 0 °C for 30 minutes followed by dropwise addition of 8-aminoquinoline (1.44 g, 10 mmol) solution in CH_2Cl_2 (10 mL). The resulting suspension was warmed up to room temperature and stirred overnight. After completion, water (30 mL) was added to the reaction mixture and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2 x 30 mL). The combined organic phase was dried over $MgSO_4$ and filtered, followed by evaporation of solvent. Purification by column chromatography on silica gel (hexanes/EtOAc from 4:1 to 2:1) afforded 1.97 g (71%) of 2-methoxy-*N*-(quinolin-8-yl)benzamide as a white solid.

2-Methoxy-_N_-(quinolin-8-yl)benzamide

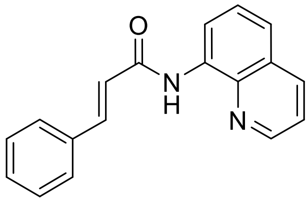


This compound is known.[5]

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 12.35 (s, 1H), 9.04 (dd, *J* = 7.7, 1.1 Hz, 1H), 8.85 (dd, *J* = 4.1, 1.6 Hz, 1H), 8.36 (dd, *J* = 7.8, 1.8 Hz, 1H), 8.15 (dd,

J = 8.2, 1.6 Hz, 1H), 7.57 (t, J = 7.9 Hz, 1H), 7.54 – 7.47 (m, 2H), 7.43 (dd, J = 8.2, 4.2 Hz, 1H), 7.14 (t, J = 7.5 Hz, 1H), 7.06 (d, J = 8.3 Hz, 1H) and 4.18 (s, 3H).

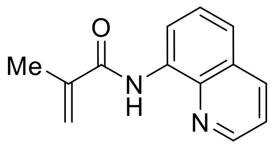
_N_-(Quinolin-8-yl)cinnamamide



Cinnamic acid (2.22 g, 15 mmol, 1.5 equiv), Et_3N (4.8 mL, 35 mmol, 3.5 equiv), ethyl chloroformate (1.4 mL, 15 mmol, 1.5 equiv), 8-aminoquinoline (1.44 g, 10 mmol), CH_2Cl_2 (40 mL). Purification by column chromatography on silica gel (hexanes/EtOAc from 4:1 to 2:1) afforded 1.85 g (67%) of *N*-(quinolin-8-yl)cinnamamide as a white solid. This compound is known.[6]

^{1}H-NMR (400 MHz, $CDCl_3$, ppm) δ 10.02 (s, 1H), 8.92 (dd, J = 7.5, 1.5 Hz, 1H), 8.85 (dd, J = 4.2, 1.7 Hz, 1H), 8.19 (dd, J = 8.3, 1.6 Hz, 1H), 7.83 (d, J = 15.6 Hz, 1H), 7.65 – 7.51 (m, 4H), 7.48 (dd, J = 8.3, 4.2 Hz, 1H), 7.46 – 7.38 (m, 3H) and 6.82 (d, J = 15.5 Hz, 1H).

_N_-(Quinolin-8-yl)methacrylamide



Methacrylic acid (0.85 mL, 10 mmol), Et_3N (1.7 mL, 12 mmol, 1.2 equiv), ethyl chloroformate (0.96 mL, 10 mmol, 1 equiv), 8-aminoquinoline (1.44 g, 10 mmol), CH_2Cl_2 (30 mL). Purification by column chromatography on silica gel (hexanes/EtOAc 4:1) afforded 1.51 g (71%) of *N*-(quinolin-8-yl)methacrylamide as a colorless oil. R_f = 0.67 (hexanes/EtOAc 4:1).

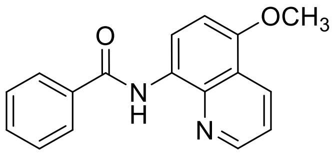
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.36 (s, 1H), 8.94 – 8.65 (m, 2H), 8.14 (dd, J = 8.2, 1.5 Hz, 1H), 7.60 – 7.48 (m, 2H), 7.44 (dd, J = 8.2, 4.2 Hz, 1H), 6.05 (s, 1H), 5.55 (s, J = 0.5 Hz, 1H), 2.19 (s, J = 0.8 Hz, 3H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 166.4, 148.2, 140.7, 138.6, 136.3, 134.4, 127.9, 127.4, 121.6, 121.5, 120.6, 116.4, 18.7.

HRMS calcd. for $C_{13}H_{12}N_2O$ $[M]^+$: 212.0950; found: 212.0946.

FT-IR (neat, cm^{-1}) υ 3358, 1717, 1669, 1525, 1485, 1378, 1323.

_N_-(5-Methoxyquinolin-8-yl)benzamide



Compound was prepared according to Procedure **A** from 8-amino-5-methoxyquinoline hydrochloride (200 mg, 0.95 mmol), *N*,*N*-dimethyl-4-aminopyridine (4.2 mg, 0.035 mmol), Et_3N (0.28 mL, 2 mmol, 2.1

[6] Ano, Y.; Tobisu, M.; Chatani, N. *Org. Lett.* **2012**, *14*, 354.

equiv), benzoyl chloride (0.1 mL, 0.9 mmol), and CH_2Cl_2 (10 mL). Purification by column chromatography on silica gel (hexanes/EtOAc from 4:1 to 1:1) afforded 210 mg (80%) of *N*-(5-methoxyquinolin-8-yl)benzamide as a white solid. This compound is known.[7]

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 10.48 (s, 1H), 8.98 – 8.70 (m, 2H), 8.55 (d, *J* = 8.1 Hz, 1H), 8.06 (d, *J* = 6.4 Hz, 2H), 7.63 – 7.36 (m, 4H), 6.85 (d, *J* = 8.5 Hz, 1H), 3.97 (s, 3H).

Cobalt-catalyzed sp^2 C-H functionalization

1. Optimization of cobalt-catalyzed alkylation/cyclization

1.1. Catalyst

General procedure for catalyst optimization experiments.

2-Dram vial with a screw cap (PTFE/Liner) was charged with 4-methyl-*N*-(quinolin-8-yl)benzamide (26.2 mg, 0.1 mmol), $AgNO_3$ (34 mg, 0.2 mmol, 2 equiv), NaOPiv (24.8 mg, 0.2 mmol, 2 equiv), catalyst (0.02 mmol, 20 mol%), styrene (14 μL, 0.12 mmol, 1.2 equiv), and CF_3CH_2OH (1 mL). Resulting mixture was heated at 80 °C for 12 h, then cooled to room temperature and analyzed by TLC (hexanes/EtOAc 4:1, hexanes/EtOAc 1:1) and ^{1}H-NMR spectroscopy.

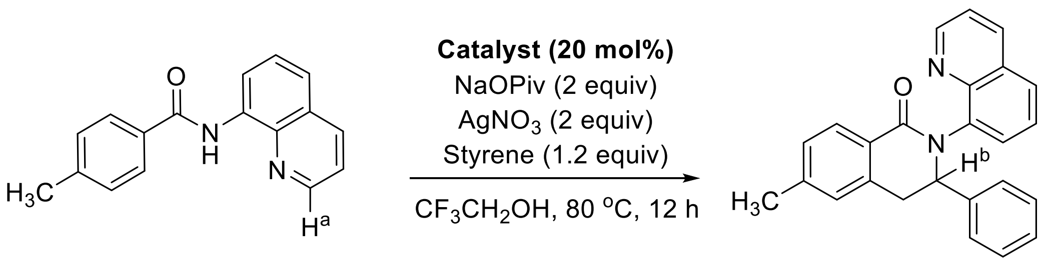


Table S1. Catalyst Screening

Entry	Catalyst	Substrate : Product ratio[a]
1[b]	Co(OAc)$_2$	1:2 (16%)[c]
2	CoCl$_2$	1:2
3[b]	**Co(acac)$_2$**	**1:20 (66%)[c]**
4	-	1:0

[a] Determined by ^{1}H-NMR spectroscopy as H^a/H^b integration ratio. [b] 16 h. [c] NMR yield using 1,1,2-trichloroethane as internal standard in parentheses.

[7] Allu, S.; Swamy, K. C. K. *J. Org. Chem*. **2014**, *79*, 3963.

1.2. Oxidant

General procedure for oxidant optimization experiments.

2-Dram vial with a screw cap (PTFE/Liner) was charged with 4-methyl-*N*-(quinolin-8-yl)benzamide (26.2 mg, 0.1 mmol), oxidant (0.05 - 0.2 mmol, 0.5 - 2 equiv), NaOPiv (24.8 mg, 0.2 mmol, 2 equiv), $Co(acac)_2$ (5.2 mg, 0.02 mmol, 20 mol%), styrene (14 µL, 0.12 mmol, 1.2 equiv), and CF_3CH_2OH (1 mL). Resulting mixture was heated at 80 °C for 5 – 16 h, cooled to room temperature and analyzed by TLC (hexanes/EtOAc 4:1, hexanes/EtOAc 1:1) and ^1H-NMR spectroscopy.

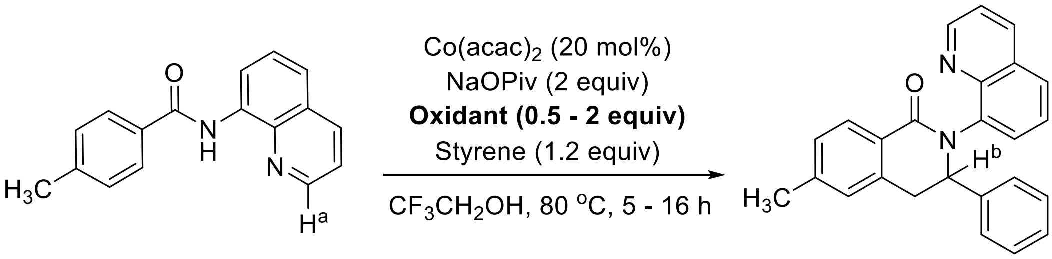


Table S2. Oxidant Screening

Entry	Oxidant	Time, h	Substrate : Product ratio[a]
1	$PhI(OAc)_2$ (2 equiv)	12	5:1
2	$Mn(acac)_3$ (2 equiv)	12	1:2
3	O_2	16	5:1
4	$Mn(OAc)_2$ (1 equiv)	16	1:9
5	$Mn(OAc)_2$ (1 equiv)/O_2	12	1:9
6	$Mn(OAc)_2$ (0.5 equiv)	16	1:2 (41%)[b]
7	**$Mn(OAc)_3$*$2H_2O$ (2 equiv)**	**12**	**1:>99 (93%)[b]**
8	$Mn(OAc)_3$*$2H_2O$ (2 equiv)	5	1:20
9	$Mn(OAc)_3$*$2H_2O$ (1.5 equiv)	5	1:20
10	**$Mn(OAc)_3$*$2H_2O$ (1 equiv)**	**5**	**1:20**
11	**$Mn(OAc)_3$*$2H_2O$ (0.5 equiv)**	**16**	**1:20 (90%)[b]**
12	-	16	5:1 (15%)[b]
13[c]	$Mn(OAc)_3$*$2H_2O$ (0.5 equiv)	16	1:0 (<1%)[b]
14[d]	$Mn(OAc)_3$*$2H_2O$ (0.5 equiv)/ inert atmosphere	12	4:1

[a] Determined by ^1H-NMR spectroscopy as H^a/H^b integration ratio. [b] NMR yield using 1,1,2-trichloroethane as internal standard in parentheses. [c] Reaction was performed without $Co(acac)_2$. [d] Deoxygenated CF_3CH_2OH was used under inert atmosphere.

1.3. Temperature

General procedure for temperature optimization experiments.

2-Dram vial with a screw cap (PTFE/Liner) was charged with 4-methyl-*N*-(quinolin-8-yl)benzamide (26.2 mg, 0.1 mmol), $Mn(OAc)_3*2H_2O$ (0.05 mmol, 0.5 equiv), NaOPiv (24.8 mg, 0.2 mmol, 2 equiv), $Co(acac)_2$ (5.2 mg, 0.02 mmol, 20 mol%), styrene (14 μL, 0.12 mmol, 1.2 equiv), and CF_3CH_2OH (1 mL). Resulting mixture was stirred at indicated temperature for 16 h, cooled to room temperature and analyzed by TLC (hexanes/EtOAc 4:1, hexanes/EtOAc 1:1) and ^{1}H-NMR spectroscopy.

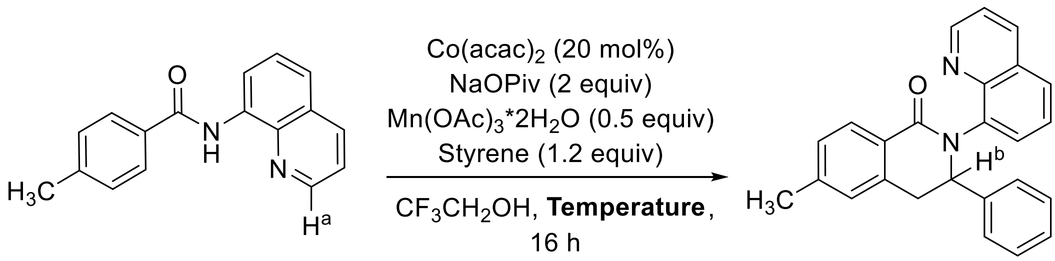


Table S3. Temperature Screening

Entry	Temperature, °C	Substrate : Product ratio[a]
1	60	1:99
2	40	1:99
3	**rt**	**1:99 (93%)[b]**

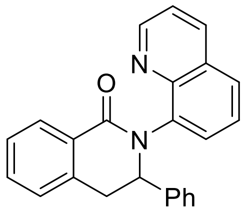
[a] Determined by ^{1}H-NMR spectroscopy as H^a/H^b integration ratio. [b] NMR yield using 1,1,2-trichloroethane as internal standard in parentheses.

2. Cobalt-catalyzed sp^2 C-H alkylation/cyclization and characterization of products

General procedure for cobalt-catalyzed sp^2 C-H alkylation/cyclization.

A 8 dram vial (septum with needle) equipped with a magnetic stir bar was charged with amide (0.5 mmol), alkyne (0.6 mmol, 1.2 equiv), $Co(acac)_2$ (0.1 – 0.25 mmol, 20 - 50 mol%), NaOPiv (1 mmol, 2 equiv), $Mn(OAc)_3*2H_2O$ (0.25 - 0.5 mmol, 0.5 – 1 equiv), and CF_3CH_2OH (5 mL). Reaction mixture was stirred at room temperature (or heated at 80 °C) for indicated time, monitored by TLC (after 2 h, 6 h, 12 h, 16 h, and 20 h to determine the completion time). Reaction solvent was evaporated, product was purified using column chromatography on silica gel using appropriate eluent. After purification product was dried under reduced pressure.

3-Phenyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 2, Entry 1)



N-(Quinolin-8-yl)benzamide (124 mg, 0.5 mmol), styrene (69 µL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (26 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3*2H_2O$ (67 mg, 0.25 mmol, 0.5 equiv), and CF_3CH_2OH (5 mL), 12 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 132 mg (75%) of a white solid was obtained. R_f = 0.43 (hexanes/EtOAc 1:1), mp 166 – 168 °C (Et_2O).

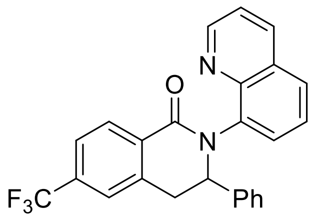
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.96 (d, *J* = 2.8 Hz, 1H), 8.23 (dd, *J* = 7.6, 0.9 Hz, 1H), 8.16 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 8.1 Hz, 1H), 7.54 (dd, *J* = 7.3, 1.2 Hz, 1H), 7.49 – 7.33 (m, 4H), 7.25 – 7.05 (m, 6H), 5.46 (d, *J* = 5.9 Hz, 1H), 4.25 (bs, 1H), 3.23 (bs, 1H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 165.1, 150.4, 144.2, 140.8, 139.1, 136.4, 136.3, 132.1, 130.1, 129.8, 129.5, 128.4, 128.2, 127.7, 127.5, 127.4, 127.0, 126.9, 126.0, 121.3, 63.1, 36.3.

HRMS calcd. for $C_{24}H_{18}N_2O$ $[M]^+$: 350.1419; found: 350.1415.

FT-IR (neat, cm^{-1}) υ 1644, 1424.

3-Phenyl-2-(quinolin-8-yl)-6-(trifluoromethyl)-3,4-dihydroisoquinolin-1(2H)-one (Table 2, Entry 2)



4-Trifluoromethyl-*N*-(quinolin-8-yl)benzamide (158 mg, 0.5 mmol), styrene (69 µL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3*2H_2O$ (67 mg, 0.25 mmol, 0.5 equiv), and CF_3CH_2OH (5 mL), 12 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 146 mg (70%) of a white solid was obtained. R_f = 0.65 (hexanes/EtOAc 1:1), mp 160 – 162 °C (Et_2O).

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.98 (d, *J* = 2.8 Hz, 1H), 8.36 (d, *J* = 8.1 Hz, 1H), 8.15 (d, *J* = 7.9 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.54 (dd, *J* = 7.3, 1.1 Hz, 1H), 7.47 – 7.35 (m, 3H), 7.24 – 7.09 (m, 5H), 5.48 (d, *J* = 6.1 Hz, 1H), 4.29 (bs, 1H), 3.29 (bs, 1H).

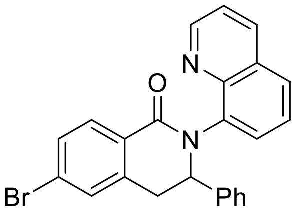
^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 163.9, 150.6, 144.0, 140.1 (m), 138.6, 137.4, 136.4, 133.6 (q, J_{C-F} = 32.2 Hz), 132.9, 129.9, 129.5, 129.0, 128.5, 128.0, 127.7, 126.8, 126.0, 124.6, 123.9, 123.7 (q, J_{C-F} = 272.3 Hz), 121.5, 62.8, 36.1.

^{19}F-NMR (470 MHz, $CDCl_3$, ppm) δ -62.69.

HRMS calcd. for $C_{25}H_{17}N_2OF_3$ $[M]^+$: 418.1293; found: 418.1288.

FT-IR (neat, cm^{-1}) υ 1643, 1423, 1326.

6-Bromo-3-phenyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 2, Entry 3)



4-Bromo-*N*-(quinolin-8-yl)benzamide (158 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (67 mg, 0.25 mmol, 0.5 equiv), and CF_3CH_2OH (5 mL), 12 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 136 mg (64%) of a white solid was obtained. R_f = 0.55 (hexanes/EtOAc 1:1), mp 186 – 188 °C (Et_2O).

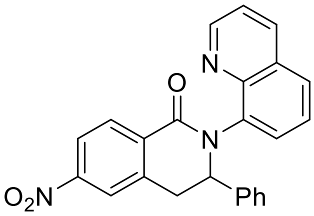
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.95 (s, 1H), 8.17-8.08 (m, 2H), 7.74 (d, *J* = 7.7 Hz, 1H), 7.54-7.50 (m, 2H), 7.44-7.40 (m, 2H), 7.35 – 7.28 (m, 1H), 7.26 – 7.09 (m, 5H), 5.44 (bs, 1H), 4.24 (bs, 1H), 3.18 (bs, 1H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 164.4, 150.5, 140.3, 139.6, 138.8, 138.6, 136.4, 130.5, 130.4, 130.2, 130.0, 129.5, 128.8, 128.4, 127.9, 127.6, 126.8, 126.0, 121.4, 119.9, 63.0, 36.0.

HRMS calcd. for $C_{24}H_{17}N_2O^{81}Br$ $[M]^+$: 430.0504; found: 430.0505.

FT-IR (neat, cm^{-1}) υ 1651, 1593, 1471, 1422.

6-Nitro-3-phenyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 2, Entry 4)



4-Nitro-*N*-(quinolin-8-yl)benzamide (147 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (67 mg, 0.25 mmol, 0.5 equiv), and CF_3CH_2OH (5 mL), 20 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 140 mg (71%) of a yellow solid was obtained. R_f = 0.45 (hexanes/EtOAc 1:1), mp 177 – 179 °C (Hexanes/Et_2O 1:1).

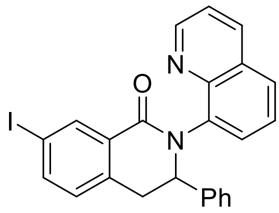
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 9.11 – 8.86 (m, 1H), 8.37 (d, *J* = 7.2 Hz, 1H), 8.31 – 8.14 (m, 2H), 8.07 – 7.93 (m, 1H), 7.80 (d, *J* = 6.7 Hz, 1H), 7.56 (d, *J* = 6.2 Hz, 1H), 7.51 – 7.40 (m, 2H), 7.26 – 7.09 (m, 5H), 5.54 (bs, 1H), 4.36 (bs, 1H), 3.32 (d, *J* = 10.5 Hz, 1H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 163.2, 150.6, 149.8, 143.8, 139.6, 138.3, 136.4, 135.1, 129.8, 129.5, 128.5, 128.2, 127.8, 126.6, 126.0, 122.7, 122.0, 121.6, 62.9, 36.2.

HRMS calcd. for $C_{24}H_{17}N_3O_3$ $[M]^+$: 395.1270; found: 395.1271.

FT-IR (neat, cm^{-1}) υ 1661, 1526, 1494, 1349.

7-Iodo-3-phenyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 2, Entry 5)



3-Iodo-*N*-(quinolin-8-yl)benzamide (187 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), Co(acac)$_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), Mn(OAc)$_3$*2H$_2$O (67 mg, 0.25 mmol, 0.5 equiv), and CF$_3$CH$_2$OH (5 mL), 16 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 144 mg (61%) of a white solid was obtained. R$_f$ = 0.63 (hexanes/EtOAc 1:1), mp 214 – 216 °C (Et$_2$O).

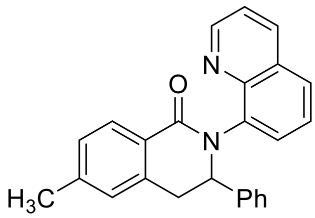
^{1}H-NMR (500 MHz, CDCl$_3$, ppm) δ 8.95 (d, *J* = 2.7 Hz, 1H), 8.54 (d, *J* = 1.7 Hz, 1H), 8.16 (d, *J* = 8.0 Hz, 1H), 7.80 – 7.71 (m, 2H), 7.52 (dd, *J* = 7.3, 1.1 Hz, 1H), 7.45 – 7.40 (m, 2H), 7.23 – 7.11 (m, 5H), 6.89 (d, *J* = 8.0 Hz, 1H), 5.43 (dd, *J* = 6.1, 4.1 Hz, 1H), 4.17 (bs, 1H), 3.17 (d, *J* = 14.0 Hz, 1H).

^{13}C-NMR (125 MHz, CDCl$_3$, ppm) δ 163.6, 150.5, 140.9, 140.3, 138.7, 137.2, 136.4, 136.1, 131.6, 130.0, 129.5, 128.4, 127.9, 127.5, 126.8, 126.0, 121.4, 91.8, 63.0, 36.0.

HRMS calcd. for $C_{24}H_{17}N_2OI$ $[M]^+$: 476.0386; found: 476.0379.

FT-IR (neat, cm^{-1}) υ 1644, 1494, 1425.

6-Methyl-3-phenyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 2, Entry 6)



4-Methyl-*N*-(quinolin-8-yl)benzamide (131 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), Co(acac)$_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), Mn(OAc)$_3$*2H$_2$O (67 mg, 0.25 mmol, 0.5 equiv), and CF$_3$CH$_2$OH (5 mL), 12 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 146 mg (80%) of a white solid was obtained. R$_f$ = 0.43 (hexanes/EtOAc 1:1), mp 203 – 205 °C (Et$_2$O).

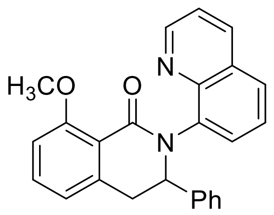
^{1}H-NMR (500 MHz, CDCl$_3$, ppm) δ 8.95 (d, *J* = 2.9 Hz, 1H), 8.19 – 8.07 (m, 2H), 7.72 (d, *J* = 8.1 Hz, 1H), 7.54 (d, *J* = 7.2 Hz, 1H), 7.46 – 7.37 (m, 2H), 7.25 – 7.08 (m, 6H), 6.95 (s, 1H), 5.44 (dd, *J* = 5.8, 4.4 Hz, 1H), 4.21 (bs, 1H), 3.17 (d, *J* = 12.5 Hz, 1H), 2.36 (s, *J* = 15.4 Hz, 3H).

^{13}C-NMR (125 MHz, CDCl$_3$, ppm) δ 165.2, 150.3, 144.2, 142.6, 141.0, 139.2, 136.6, 136.4, 130.2, 129.5, 128.4, 128.2, 128.1, 127.8, 127.6, 127.3, 127.2, 126.9, 126.0, 121.3, 63.1, 36.2, 21.6.

HRMS calcd. for $C_{25}H_{20}N_2O$ $[M]^+$: 364.1576; found: 364.1571.

FT-IR (neat, cm^{-1}) υ 1644, 1593, 1423.

8-Methoxy-3-phenyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 2, Entry 7)



2-Methoxy-*N*-(quinolin-8-yl)benzamide (139 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 24 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1, then EtOAc) 162 mg (85%) of a white solid was obtained. R_f = 0.13 (hexanes/EtOAc 1:1), mp 200 – 202 °C (Et_2O).

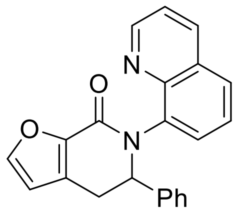
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 9.01 – 8.87 (m, 1H), 8.16 (d, *J* = 6.8 Hz, 1H), 7.73 (d, *J* = 7.8 Hz, 1H), 7.59 (d, *J* = 6.6 Hz, 1H), 7.47 – 7.38 (m, 2H), 7.33 – 7.25 (m, 3H), 7.21 – 7.12 (m, 3H), 6.89 (d, *J* = 8.3 Hz, 1H), 6.69 (d, *J* = 6.9 Hz, 1H), 5.29 (bs, 1H), 4.40 (bs, 1H), 3.92 (s, 3H), 3.14 (bs, 1H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 163.4, 160.1, 150.2, 144.4, 140.6, 139.8, 139.6, 136.1, 132.7, 130.1, 129.4, 128.1, 127.5, 127.0, 126.8, 125.9, 121.1, 119.9, 118.4, 111.3, 62.5, 56.2, 37.3.

HRMS calcd. for $C_{25}H_{20}N_2O_2$ $[M]^+$: 380.1525; found: 380.1524.

FT-IR (neat, cm^{-1}) υ 1644, 1593, 1433, 1238.

5-Phenyl-6-(quinolin-8-yl)-5,6-dihydrofuro[2,3-c]pyridin-7(4H)-one (Table 2, Entry 8)



N-(Quinolin-8-yl)furan-2-carboxamide (119 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 20 h, RT. After column chromatography (gradient hexanes/EtOAc from 2:1 to 1:1, then EtOAc) 154 mg (81%) of a white solid was obtained. R_f = 0.18 (hexanes/EtOAc 1:1), mp 232 – 234 °C (Et_2O).

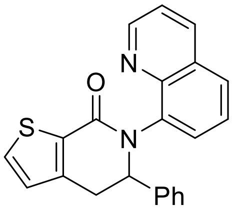
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.94 (d, *J* = 2.3 Hz, 1H), 8.11 (d, *J* = 8.1 Hz, 1H), 7.67 (d, *J* = 8.1 Hz, 1H), 7.60 – 7.48 (m, 2H), 7.43 – 7.34 (m, 2H), 7.26 – 7.10 (m, 5H), 6.37 (s, 1H), 5.61 (t, *J* = 5.6 Hz, 1H), 3.83 (d, *J* = 9.9 Hz, 1H), 3.15 (d, *J* = 15.8 Hz, 1H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 158.3, 150.2, 146.3, 144.3, 143.4, 140.8, 137.6, 136.3, 130.8, 129.3, 128.2, 127.6, 127.5, 127.4, 127.0, 125.9, 121.2, 110.6, 64.8, 30.1.

HRMS calcd. for $C_{22}H_{16}N_2O_2$ $[M]^+$: 340.1212; found: 340.1210.

FT-IR (neat, cm^{-1}) υ 1643, 1593, 1433.

5-Phenyl-6-(quinolin-8-yl)-5,6-dihydrothieno[2,3-c]pyridin-7(4H)-one (Table 2, Entry 9)



N-(Quinolin-8-yl)thiophene-2-carboxamide (127 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 24 h, RT. After column chromatography (gradient hexanes/EtOAc from 2:1 to 1:1, then EtOAc) 134 mg (75%) of a white solid was obtained. R_f = 0.35 (hexanes/EtOAc 1:1), mp 217 – 219 °C (Et_2O).

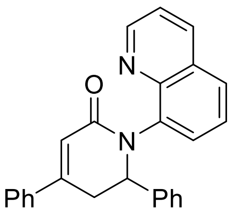
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.95 (d, *J* = 2.7 Hz, 1H), 8.12 (d, *J* = 7.9 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.62 – 7.46 (m, 2H), 7.45 – 7.34 (m, 2H), 7.26 – 7.11 (m, 5H), 6.90 (d, *J* = 4.8 Hz, 1H), 5.60 (t, *J* = 5.5 Hz, 1H), 3.96 (bs, 1H), 3.28 (d, *J* = 13.1 Hz, 1H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 161.6, 150.3, 144.3, 142.3, 140.9, 138.1, 136.3, 132.0, 131.5, 130.7, 129.4, 128.2, 127.6, 127.4, 127.0, 126.9, 125.9, 121.2, 64.6, 33.1.

HRMS calcd. for $C_{22}H_{16}N_2OS$ $[M]^+$: 356.0983; found: 356.0986.

FT-IR (neat, cm^{-1}) υ 1664, 1414, 1259, 1141, 1030.

4,6-Diphenyl-1-(quinolin-8-yl)-5,6-dihydropyridin-2(1H)-one (4, Scheme 1)



N-(Quinolin-8-yl)cinnamamide (137 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 24 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 1:1, then EtOAc) 116 mg (62%) of a yellow oil was obtained. R_f = 0.71 (EtOAc).

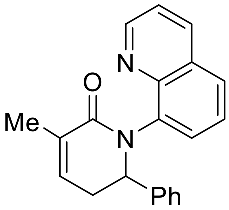
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.99 (s, 1H), 8.14 (d, *J* = 8.0 Hz, 1H), 7.70 (d, *J* = 8.1 Hz, 1H), 7.56 (d, *J* = 7.2 Hz, 1H), 7.53 – 7.49 (m, 2H), 7.43 – 7.35 (m, 5H), 7.34 – 7.30 (m, 2H), 7.23 – 7.19 (m, 2H), 7.18 – 7.14 (m, 1H), 6.62 (s, 1H), 5.61 (bs, 1H), 3.81 (bs, 1H), 3.17 (d, *J* = 13.9 Hz, 1H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 159.2, 149.8, 148.2, 144.0, 141.0, 138.1, 137.7, 136.5, 130.6, 129.5, 129.4, 128.6, 128.3, 127.6, 127.5, 127.2, 126.0, 125.9, 121.3, 120.3, 62.6, 35.1.

HRMS calcd. for $C_{26}H_{20}N_2O$ $[M]^+$: 376.1576; found: 376.1569.

FT-IR (neat, cm^{-1}) υ 1651, 1428, 1244.

3-Methyl-6-phenyl-1-(quinolin-8-yl)-5,6-dihydropyridin-2(1H)-one (6, Scheme 1)



N-(Quinolin-8-yl)methacrylamide (106 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), Co(acac)$_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), Mn(OAc)$_3$*2H$_2$O (134 mg, 0.5 mmol, 1 equiv), and CF$_3$CH$_2$OH (5 mL), 30 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 1:1, then EtOAc) 115 mg (73%) of a white solid was obtained. R$_f$ = 0.23 (hexanes/EtOAc 1:1), mp 170 – 172 °C (Et$_2$O).

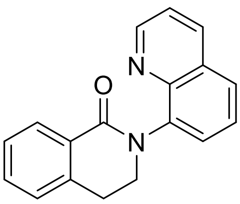
^{1}H-NMR (500 MHz, CDCl$_3$, ppm) δ 8.97 (dd, *J* = 4.1, 1.6 Hz, 1H), 8.11 (d, *J* = 8.1 Hz, 1H), 7.67 (d, *J* = 8.1 Hz, 1H), 7.50 (d, *J* = 7.2 Hz, 1H), 7.42 – 7.33 (m, 2H), 7.31 – 7.17 (m, 5H), 6.34 (s, 1H), 5.34 (dd, *J* = 6.4, 5.3 Hz, 1H), 3.44 (bs, 1H), 2.64 (d, *J* = 16.6 Hz, 1H), 2.04 (s, 3H).

^{13}C-NMR (125 MHz, CDCl$_3$, ppm) δ 166.2, 150.3, 144.3, 141.4, 138.9, 136.2, 132.5, 131.9, 130.4, 129.4, 128.1, 127.4, 127.3, 125.9, 121.2, 62.7, 32.4, 17.3.

HRMS calcd. for $C_{21}H_{18}N_2O$ [M]$^+$: 314.1419; found: 314.1418.

FT-IR (neat, cm^{-1}) υ 3062, 1668, 1626, 1556, 1370, 1264, 1137, 1030.

2-(Quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 3, Entry 1)



N-(Quinolin-8-yl)benzamide (124 mg, 0.5 mmol), Co(acac)$_2$ (26 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), Mn(OAc)$_3$*2H$_2$O (134 mg, 0.5 mmol, 1 equiv), and CF$_3$CH$_2$OH (5 mL) were mixed at room temperature. The solution was purged with ethylene gas for 5 minutes. The vial was equipped with ethylene balloon and stirred for 22 h at RT. The reaction was checked by TLC 3-4 times during the reaction. This is important as oxygen required for the reaction is introduced when the vial is opened to air. After each opening, the reaction mixture was purged with ethylene for 1 minute and the vial was equipped with ethylene balloon. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 98 mg (72%) of a colorless oil was obtained. R$_f$ = 0.30 (hexanes/EtOAc 1:1).

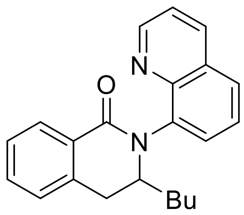
^{1}H-NMR (500 MHz, CDCl$_3$, ppm) δ 8.93 – 8.84 (m, 1H), 8.26 – 8.12 (m, 2H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.77 (d, *J* = 7.1 Hz, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.47 (t, *J* = 7.3 Hz, 1H), 7.45 – 7.33 (m, 2H), 7.28 (d, *J* = 7.4 Hz, 1H), 4.28 (bs, 2H), 3.29 (bs, 2H).

^{13}C-NMR (125 MHz, CDCl$_3$, ppm) δ 165.0, 150.2, 144.0, 140.5, 139.0, 136.3, 131.8, 129.9, 129.6, 128.9, 128.7, 127.7, 127.0, 126.9, 126.4, 121.5, 50.2, 28.7.

HRMS calcd. for $C_{18}H_{15}N_2O$ [MH]$^+$: 275.1184; found: 375.1178.

FT-IR (neat, cm^{-1}) υ 1650, 1593, 1472, 1419.

3-Butyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 3, Entry 2)



N-(quinolin-8-yl)benzamide (124 mg, 0.5 mmol), 1-hexene (75 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (26 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3*2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 16 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 146 mg (89%, 10:1 mixture of isomers inseparable by flash column chromatography, structure of major isomer shown) of a colorless oil was obtained. R_f = 0.53 (hexanes/EtOAc 1:1).

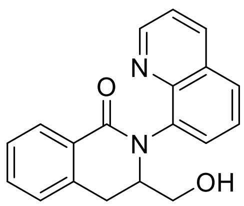
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.90 (d, *J* = 1.9 Hz, 1H), 8.22 – 8.11 (m, 2H), 7.82 (d, *J* = 8.2 Hz, 1H), 7.78 (d, *J* = 6.8 Hz, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.47 (t, *J* = 7.5 Hz, 1H), 7.43 – 7.34 (m, 2H), 7.28 (d, *J* = 7.4 Hz, 1H), 4.15 (d, *J* = 3.7 Hz, 1H), 3.99 (bs, 1H), 2.97 (d, *J* = 14.1 Hz, 1H), 1.73 – 1.53 (m, 2H), 1.42 – 1.00 (m, 4H), 0.73 (t, *J* = 6.5 Hz, 3H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 164.2, 150.3, 144.3, 138.7, 137.5, 136.2, 131.8, 130.4, 129.8, 129.6, 128.3, 127.7, 127.6, 126.7, 126.0, 121.3, 59.2, 32.0, 31.6, 28.4, 22.4, 13.8.

HRMS calcd. for $C_{22}H_{22}N_2O$ $[M]^+$: 330.1732; found: 330.1737.

FT-IR (neat, cm^{-1}) υ 2954, 2926, 1647, 1461, 1420, 1244.

3-(Hydroxymethyl)-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 3, Entry 3)



N-(quinolin-8-yl)benzamide (124 mg, 0.5 mmol), allyl alcohol (41 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (26 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3*2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 30 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 1:1, then EtOAc) 84 mg (55%) of a yellow oil was obtained. R_f = 0.53 (EtOAc).

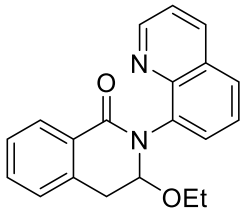
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.95 – 8.86 (m, 1H), 8.29 (d, *J* = 7.1 Hz, 1H), 8.11 (d, *J* = 7.6 Hz, 1H), 7.89 (d, *J* = 7.1 Hz, 1H), 7.75 (d, *J* = 7.1 Hz, 1H), 7.65 (t, *J* = 7.1 Hz, 1H), 7.55 – 7.43 (m, 2H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 4.19 (bs, 1H), 3.91 – 3.63 (m, 2H), 3.57 (d, *J* = 7.3 Hz, 1H), 3.17 (d, *J* = 16.1 Hz, 1H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 165.0, 150.3, 140.3, 137.7, 137.2, 132.2, 129.9, 129.2, 129.1, 128.6, 128.4, 127.3, 127.0, 126.9, 121.9, 121.7, 64.5, 62.3, 32.3.

HRMS calcd. for $C_{19}H_{17}N_2O_2$ $[MH]^+$: 305.1290; found: 305.1287.

FT-IR (neat, cm^{-1}) υ 3365, 1636, 1417, 1038.

3-Ethoxy-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 3, Entry 4)



N-(quinolin-8-yl)benzamide (124 mg, 0.5 mmol), ethyl vinyl ether (58 μL, 0.6 mmol, 1.2 equiv), Co(acac)$_2$ (26 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), Mn(OAc)$_3$*2H$_2$O (134 mg, 0.5 mmol, 1 equiv), and CF$_3$CH$_2$OH (5 mL), 16 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 143 mg (90%) of a colorless oil was obtained.

R$_f$ = 0.43 (hexanes/EtOAc 1:1).

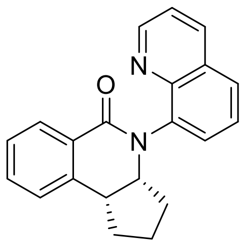
^{1}H-NMR (500 MHz, CDCl$_3$, ppm) δ 8.90 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.27 – 8.11 (m, 2H), 7.95 (d, *J* = 6.8 Hz, 1H), 7.85 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.63 (dd, *J* = 8.1, 7.4 Hz, 1H), 7.50 (td, *J* = 7.5, 1.4 Hz, 1H), 7.42 (dd, *J* = 8.3, 4.1 Hz, 1H), 7.38 (t, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 7.5 Hz, 1H), 5.36 (dd, *J* = 4.1, 2.2 Hz, 1H), 4.07 (d, *J* = 15.0 Hz, 1H), 3.57 – 3.48 (m, 1H), 3.42 – 3.34 (m, 1H), 3.23 (dd, *J* = 16.1, 1.9 Hz, 1H), 0.99 (t, *J* = 7.0 Hz, 3H).

^{13}C-NMR (125 MHz, CDCl$_3$, ppm) 164.3, 150.4, 144.1, 136.5, 136.3, 132.1, 130.3, 129.5, 129.0, 128.5, 127.8, 127.7, 126.8, 126.2, 121.4, 119.9, 88.6, 66.1, 33.8, 15.3.

HRMS calcd. for $C_{20}H_{18}N_2O_2$ $[M]^+$: 318.1368; found: 318.1356.

FT-IR (neat, cm^{-1}) υ 1644, 1471, 1430, 1062.

(3a*R,9b*R**)-4-(Quinolin-8-yl)-2,3,3a,4-tetrahydro-1H-cyclopenta[c]isoquinolin-5(9bH)-one (Table 3, Entry 5)**



N-(quinolin-8-yl)benzamide (124 mg, 0.5 mmol), cyclopentene (66 μL, 0.6 mmol, 1.2 equiv), Co(acac)$_2$ (26 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), Mn(OAc)$_3$*2H$_2$O (134 mg, 0.5 mmol, 1 equiv), and CF$_3$CH$_2$OH (5 mL), 20 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 138 mg (88%) of a white solid was obtained.

R$_f$ = 0.38 (hexanes/EtOAc 1:1), mp 157 – 159 °C (Hexanes/Et$_2$O 4:1).

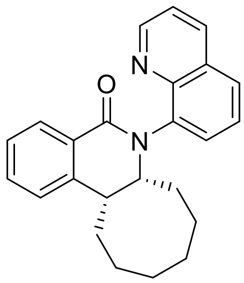
^{1}H-NMR (500 MHz, CDCl$_3$, ppm) δ 8.92 – 8.82 (m, 1H), 8.26 – 8.13 (m, 2H), 7.84 (d, *J* = 7.4 Hz, 1H), 7.73 – 7.65 (m, 1H), 7.60 (t, *J* = 7.2 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.44 – 7.28 (m, 3H), 5.00 (bs, 1H), 3.49 (bs, 1H), 2.37 – 2.18 (m, 1H), 2.19 – 2.02 (m, 1H), 1.97 – 1.81 (m, 1H), 1.81 – 1.56 (m, 3H).

^{13}C-NMR (125 MHz, CDCl$_3$, ppm) δ 164.9, 150.3, 144.4, 141.3, 138.0, 136.3, 132.0, 130.6, 129.6, 129.0, 127.9, 127.5, 126.9, 126.6, 126.1, 121.3, 63.0, 42.4, 32.8, 32.2, 22.1.

HRMS calcd. for $C_{21}H_{18}N_2O$ $[M]^+$: 314.1419; found: 314.1412.

FT-IR (neat, cm^{-1}) υ 1645, 1593, 1426.

(6a*R,12a*R**)-6-(Quinolin-8-yl)-6a,7,8,9,10,11,12,12a-octahydrocycloocta[c]isoquinolin-5(6H)-one (Table 3, Entry 6)**



N-(quinolin-8-yl)benzamide (124 mg, 0.5 mmol), *cis*-cyclooctene (78 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (26 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 20 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 2:1) 130 mg (73%) of a colorless oil was obtained. R_f = 0.48 (hexanes/EtOAc 1:1).

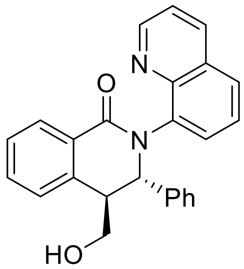
^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.92 (d, *J* = 2.1 Hz, 1H), 8.24 – 8.11 (m, 2H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.76 (d, *J* = 6.7 Hz, 1H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.46 – 7.29 (m, 3H), 4.52 (bs, 1H), 4.05 (bs, 1H), 2.27 – 2.02 (m, 2H), 1.98 – 1.86 (m, 1H), 1.81 – 1.57 (m, 4H), 1.53 – 1.08 (m, 5H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 164.9, 150.4, 144.6, 142.6, 138.6, 136.2, 131.9, 130.1, 129.5, 129.4, 128.3, 127.7, 126.4, 126.0, 125.0, 121.3, 60.1, 39.7, 28.7, 28.5, 27.6, 26.1, 24.7, 24.6.

HRMS calcd. for $C_{24}H_{25}N_2O$ $[MH]^+$: 357.1967; found: 357.1968.

FT-IR (neat, cm^{-1}) υ 2919, 2851, 1648, 1600, 1472.

(3*S,4*S**)-4-(Hydroxymethyl)-3-phenyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (Table 3, Entry 7)**



N-(quinolin-8-yl)benzamide (124 mg, 0.5 mmol), *trans*-cinnamyl alcohol (77 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (64.3 mg, 0.25 mmol, 50 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 20 h, 80 °C. After column chromatography (gradient hexanes/EtOAc from 2:1 to 1:1, then EtOAc) 100 mg (53%) of a white solid was obtained. R_f = 0.58 (EtOAc), mp 212 – 214 °C (Et_2O).

(Table 3, Entry 8)

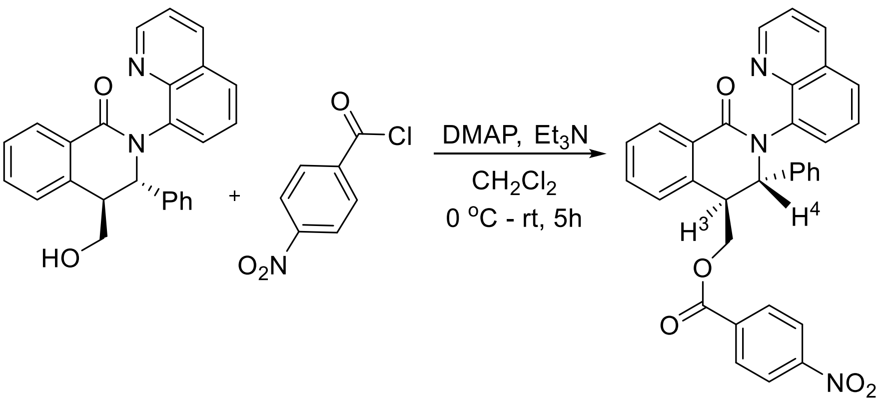
N-(quinolin-8-yl)benzamide (124 mg, 0.5 mmol), *cis*-cinnamyl alcohol (81 mg, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (64.3 mg, 0.25 mmol, 50 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 24 h, 80 °C. After column chromatography (gradient hexanes/EtOAc from 2:1 to 1:1, then EtOAc) 98 mg (52%) of a white solid was obtained.

Cis-cinnamyl alcohol was partially converted to trans-isomer as observed by ^{1}H-NMR spectra of crude reaction mixture.

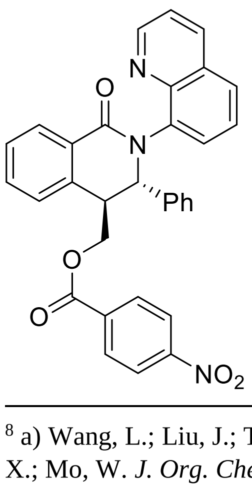
[1]H-NMR (500 MHz, $CDCl_3$, ppm) δ 9.02 – 8.88 (m, 1H), 8.32 – 8.24 (m, 2H), 7.79 (d, *J* = 7.6 Hz, 1H), 7.55 – 7.42 (m, 5H), 7.21 – 7.10 (m, 6H), 5.39 (bs, 1H), 4.28 (d, *J* = 10.3 Hz, 1H), 4.13 (bs, 1H), 3.27 (bs, 1H).
[13]C-NMR (125 MHz, $CDCl_3$, ppm) δ 164.9, 149.9, 143.1, 140.6, 138.3, 137.3, 136.9, 132.5, 131.8, 129.9, 129.8, 128.4, 128.1, 128.0, 127.9, 127.5, 127.4, 126.7, 126.5, 121.5, 67.7, 65.6, 49.3.
HRMS calcd. for $C_{25}H_{21}N_2O_2$ $[MH]^+$: 381.1603; found: 381.1610.
FT-IR (neat, cm^{-1}) υ 1643, 1593, 1424.

Determination of (3*S,4*S**)-4-(hydroxymethyl)-3-phenyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one relative configuration**

Configuration of 4-nitrobenzoyl derivative was determined by [1]H-NMR spectroscopy based on scalar coupling constant between protons H-3 and H-4 (typically, for *cis*-configuration $J_{3,4}$ = 4.2 – 6.0 Hz, for *trans*-configuration $J_{3,4}$ = 0-2 Hz).[8]



((3*S,4*S**)-1-Oxo-3-phenyl-2-(quinolin-8-yl)-1,2,3,4-tetrahydroisoquinolin-4-yl)methyl 4-nitrobenzoate**



To a solution of (3*S**,4*S**)-4-(hydroxymethyl)-3-phenyl-2-(quinolin-8-yl)-3,4-dihydroisoquinolin-1(2H)-one (50 mg, 0.13 mmol) and *N*,*N*-dimethyl-4-aminopyridine (1.6 mg, 0.013 mmol, 10 mol%) in anhydrous CH_2Cl_2 (5 mL) under nitrogen Et_3N (22 μL, 0.16 mmol, 1.2 equiv) was added. The resulting solution was cooled to 0 °C. 4-Nitrobenzoyl chloride (24 mg, 0.13 mmol) was added as a solid and reaction mixture was stirred at room temperature for 5 h. The mixture was quenched with water (10 mL) and extracted with CH_2Cl_2 (3 x

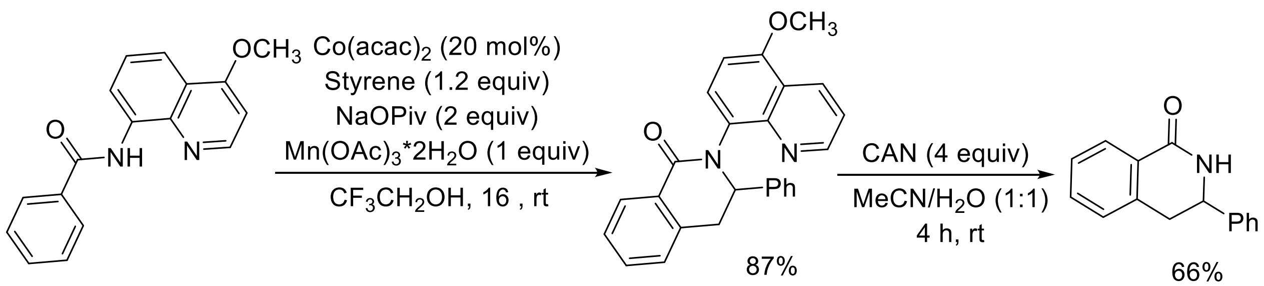
[8] a) Wang, L.; Liu, J.; Tian, H.; Qian, C.; Sun, J. *Adv. Synth. Catal*. **2005**, *347*, 689. b) Chen, W.; Cui, J.; Zhu, Y.; Hu, X.; Mo, W. *J. Org. Chem*., **2012**, *77*, 1585.

10 mL). Combined organic phase was dried over $MgSO_4$ and filtered. Concentration in vacuum followed by purification by flash column chromatography (eluent hexanes/EtOAc 2:1) afforded 46 mg (68%) of ((3*S**,4*S**)-1-oxo-3-phenyl-2-(quinolin-8-yl)-1,2,3,4-tetrahydroisoquinolin-4-yl)methyl 4-nitrobenzoate as a white solid. R_f = 0.79 (Hexanes/EtOAc 1:1)), mp 280 – 282 °C (Et_2O).

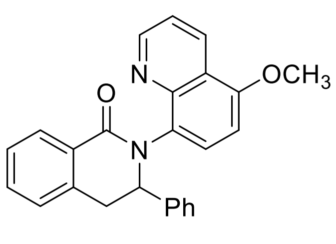
^{1}H-NMR (400 MHz, $CDCl_3$, ppm) δ 8.82 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.35 – 8.31 (m, 1H), 8.28 – 8.21 (m, 2H), 8.20 – 8.09 (m, 3H), 7.78 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.55 – 7.47 (m, 2H), 7.47 – 7.39 (m, 2H), 7.36 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.30 – 7.15 (m, 6H), 5.68 (t, *J* = 10.1 Hz, 1H), 5.40 (s, 1H), 4.95 (dd, *J* = 10.9, 5.5 Hz, 1H), 3.64 (dd, *J* = 9.1, 5.5 Hz, 1H). Since coupling between H^3 and H^4 is not observed (<0.5 Hz), we assign trans configuration to the substance.

^{13}C-NMR (100 MHz, $CDCl_3$, ppm) δ 164.6, 164.0, 150.5, 150.2, 144.3, 140.4, 138.5, 136.3, 135.6, 135.2, 132.6, 130.5, 129.6, 129.5, 129.4, 128.8, 128.7, 128.6, 128.4, 128.2, 127.8, 126.6, 126.2, 123.6, 121.5, 68.6, 64.3, 46.5.

Removal of directing group



2-(5-Methoxyquinolin-8-yl)-3-phenyl-3,4-dihydroisoquinolin-1(2H)-one (8, Scheme 2)

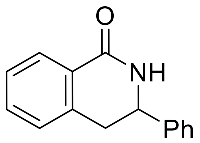


N-(5-Methoxyquinolin-8-yl)benzamide (139 mg, 0.5 mmol), styrene (69 μL, 0.6 mmol, 1.2 equiv), $Co(acac)_2$ (25.7 mg, 0.1 mmol, 20 mol%), NaOPiv (124 mg, 1 mmol, 2 equiv), $Mn(OAc)_3$*$2H_2O$ (134 mg, 0.5 mmol, 1 equiv), and CF_3CH_2OH (5 mL), 16 h, RT. After column chromatography (gradient hexanes/EtOAc from 4:1 to 1:1) 166 mg (87%) of a white solid was obtained. R_f = 0.40 (hexanes/EtOAc 1:1), mp 229 – 231 °C (Et_2O).

^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ 8.95 (dd, *J* = 4.0, 1.4 Hz, 1H), 8.56 (d, *J* = 8.0 Hz, 1H), 8.24 (dd, *J* = 7.4, 0.8 Hz, 1H), 7.45 – 7.35 (m, 4H), 7.23 – 7.09 (m, 6H), 6.71 (d, *J* = 8.3 Hz, 1H), 5.40 (dd, *J* = 6.1, 4.0 Hz, 1H), 4.25 (d, *J* = 10.7 Hz, 1H), 3.93 (s, 3H), 3.19 (d, *J* = 14.9 Hz, 1H).

^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ 165.3, 154.5, 150.6, 144.6, 141.0, 136.6, 132.0, 131.5, 131.1, 130.0, 129.9, 128.3, 128.2, 127.5, 127.3, 126.9, 121.7, 120.4, 103.4, 63.1, 55.7, 36.2.
HRMS calcd. for $C_{25}H_{20}N_2O_2$ $[M]^+$: 380.1525; found: 380.1523.
FT-IR (neat, cm^{-1}) υ 1644, 1592, 1433.

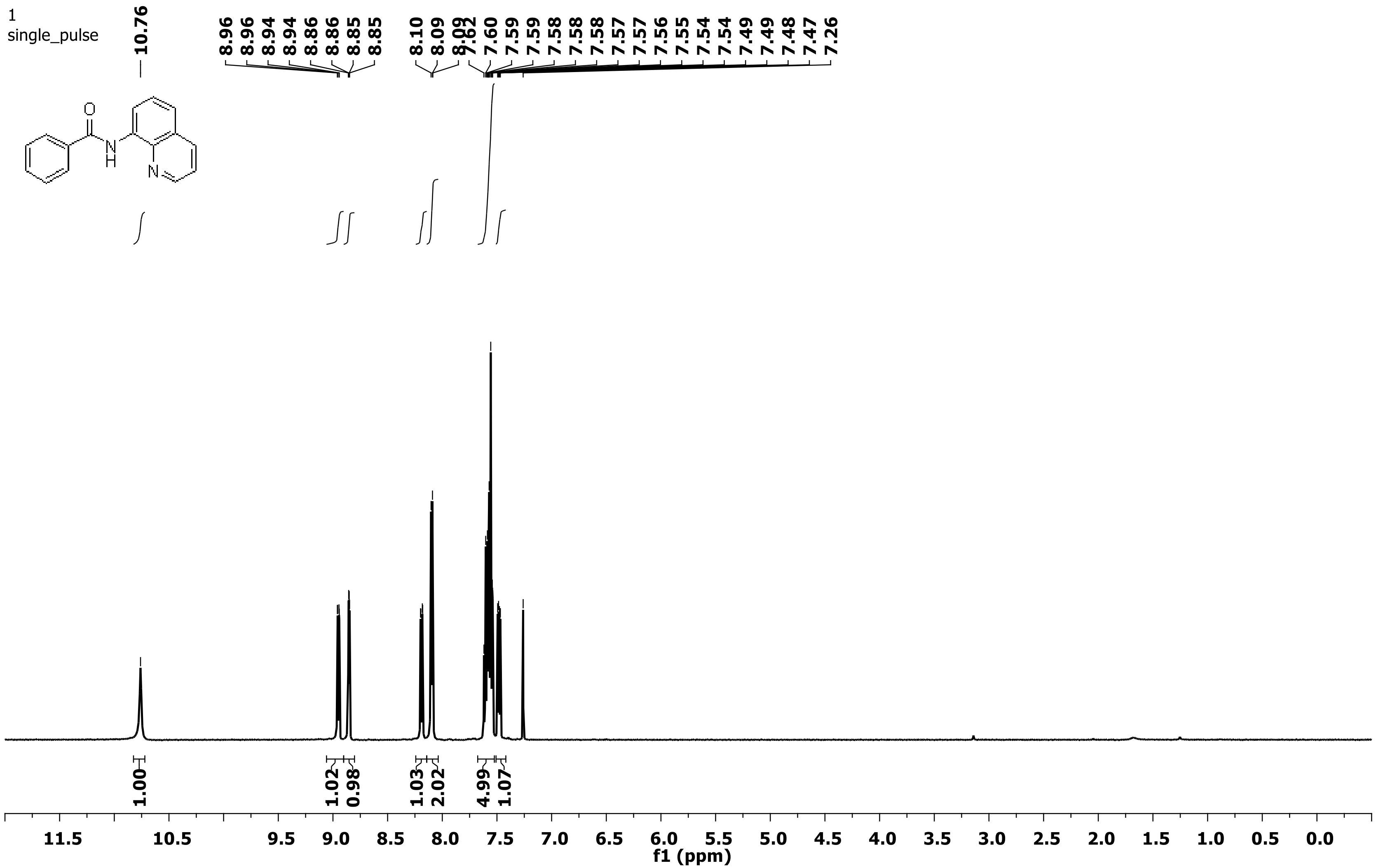
3-Phenyl-3,4-dihydroisoquinolin-1(2H)-one (9, Scheme 2)

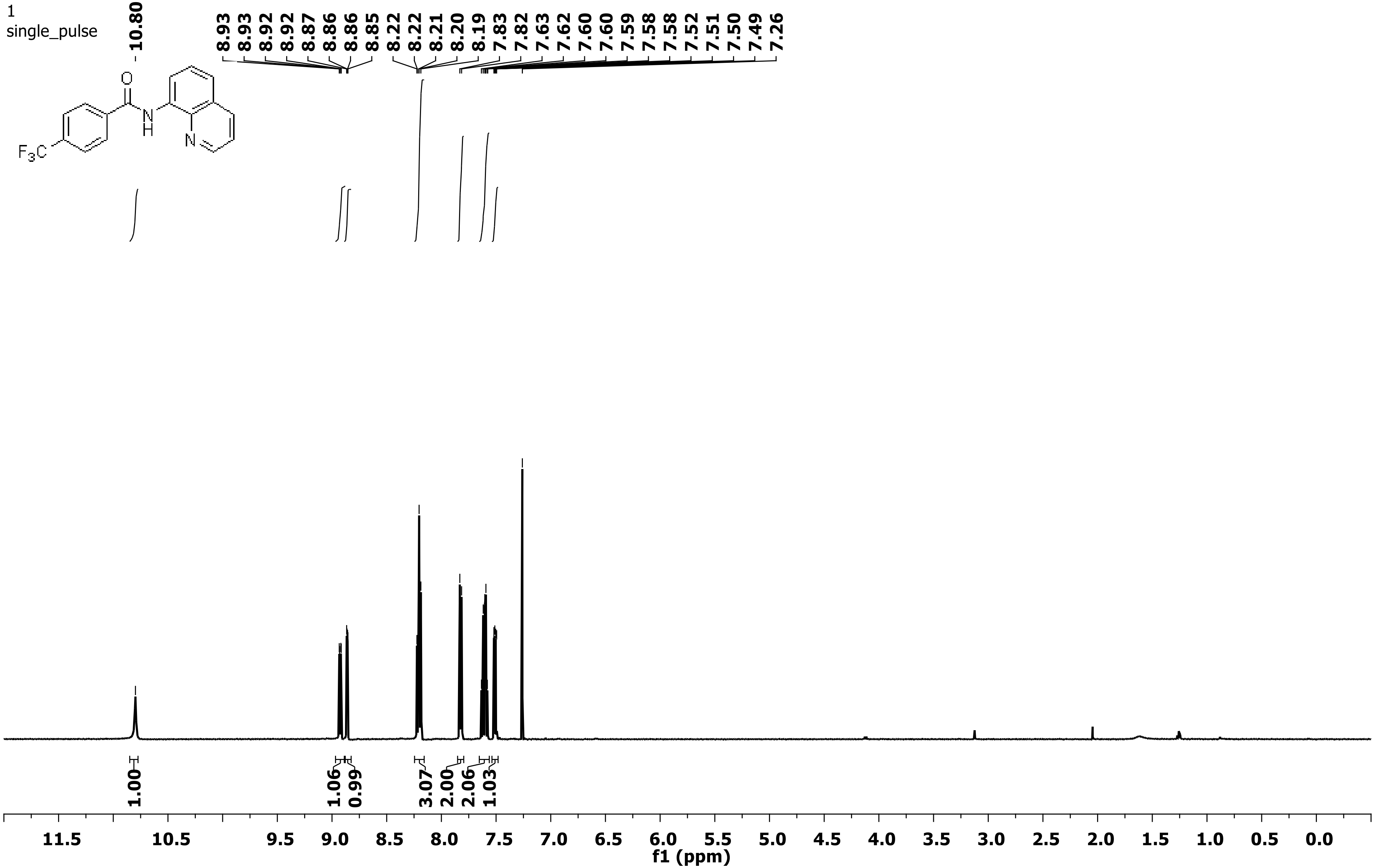


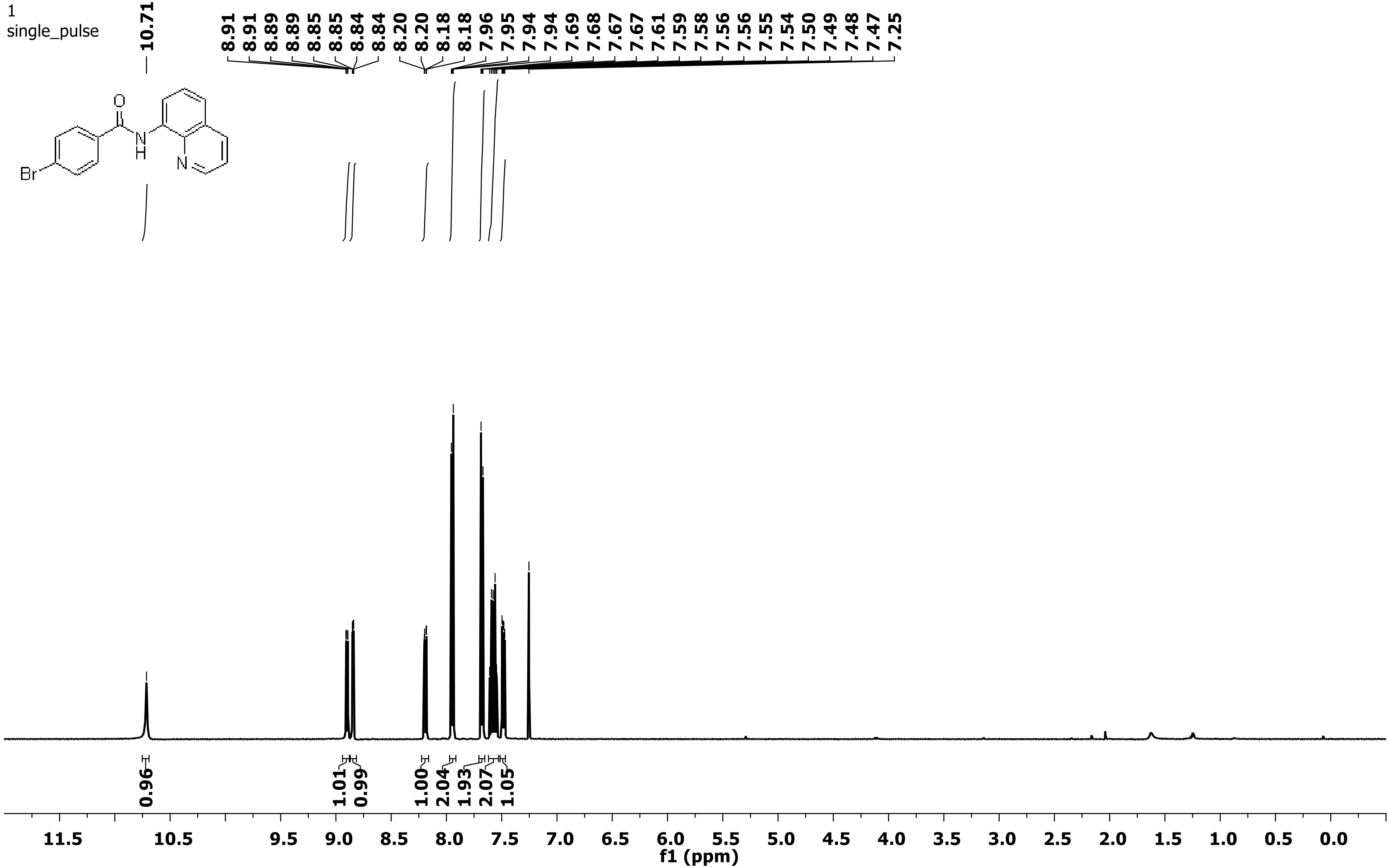
To a solution of 2-(5-methoxyquinolin-8-yl)-3-phenyl-3,4-dihydroisoquinolin-1(2H)-one (100 mg, 0.26 mmol) in MeCN (2.6 mL) at RT was added solution of CAN (570 mg, 1.04 mmol, 4 equiv) in H_2O (2.6 mL). Resulting solution was stirred at RT for 4 h, H_2O (15 mL) was added and the reaction mixture was extracted with EtOAc (3 x 15 mL). Combined organic phase was washed with 10% Na_2SO_3 solution (2 x 15 mL) and brine (15 mL), dried over $MgSO_4$, filtered, solvent was evaporated. After column chromatography (hexanes/EtOAc from 2:1) 38 mg (66%) of a colorless oil was obtained. This compound is known.[9]
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, J = 7.7, 1.3 Hz, 1H), 7.47 (td, J = 7.5, 1.5 Hz, 1H), 7.42 – 7.37 (m, 6H), 7.19 (d, J = 7.5 Hz, 1H), 6.24 (s, 1H), 4.87 (ddd, J = 11.1, 4.7, 1.1 Hz, 1H), 3.21 (dd, J = 15.7, 11.1 Hz, 1H), 3.13 (dd, J = 15.7, 4.5 Hz, 1H).

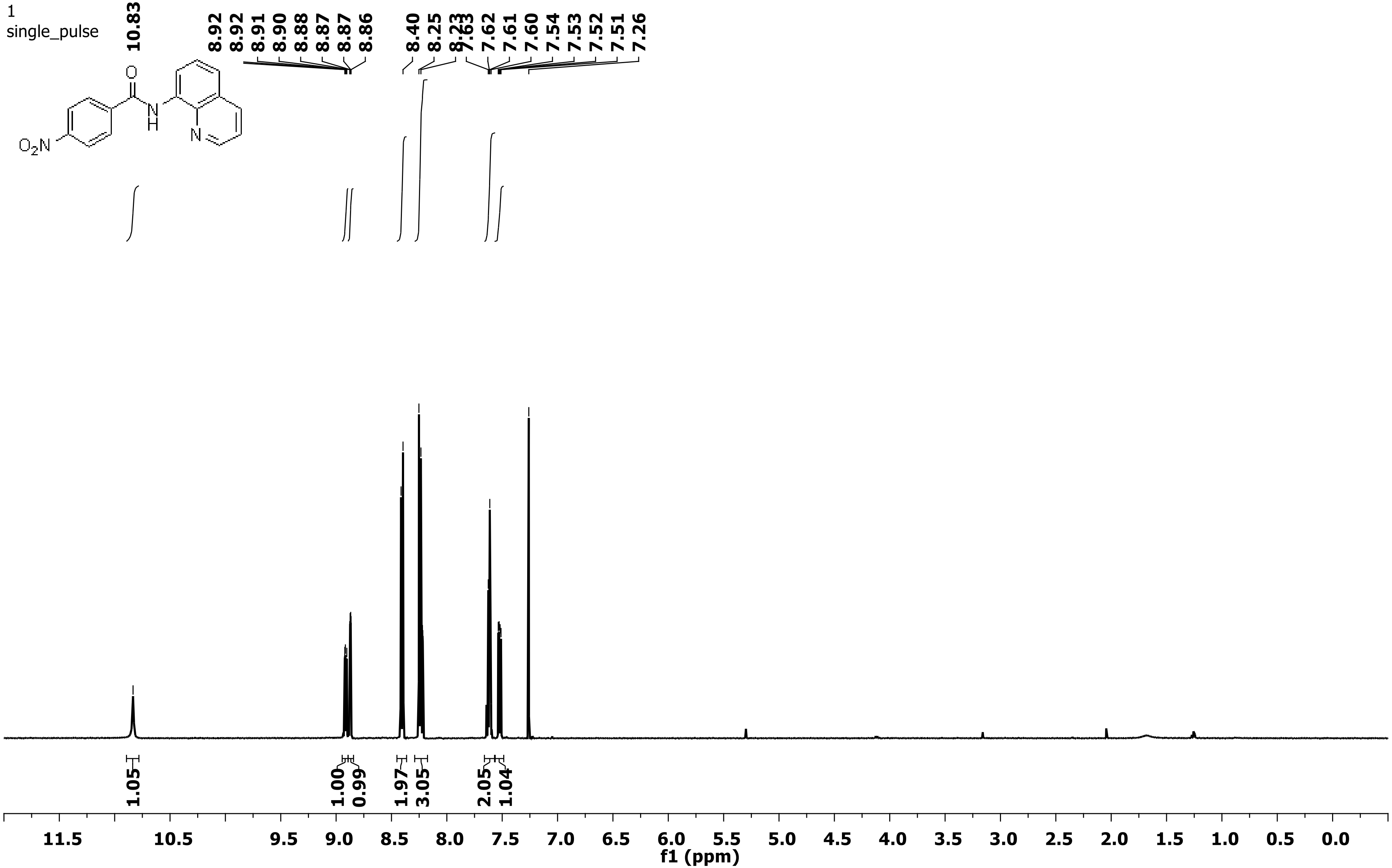
[9] Rakshit, S.; Grohmann, C.; Besset, T.; Glorius, F. *J. Am. Chem. Soc.* **2011**, *133*, 2350.

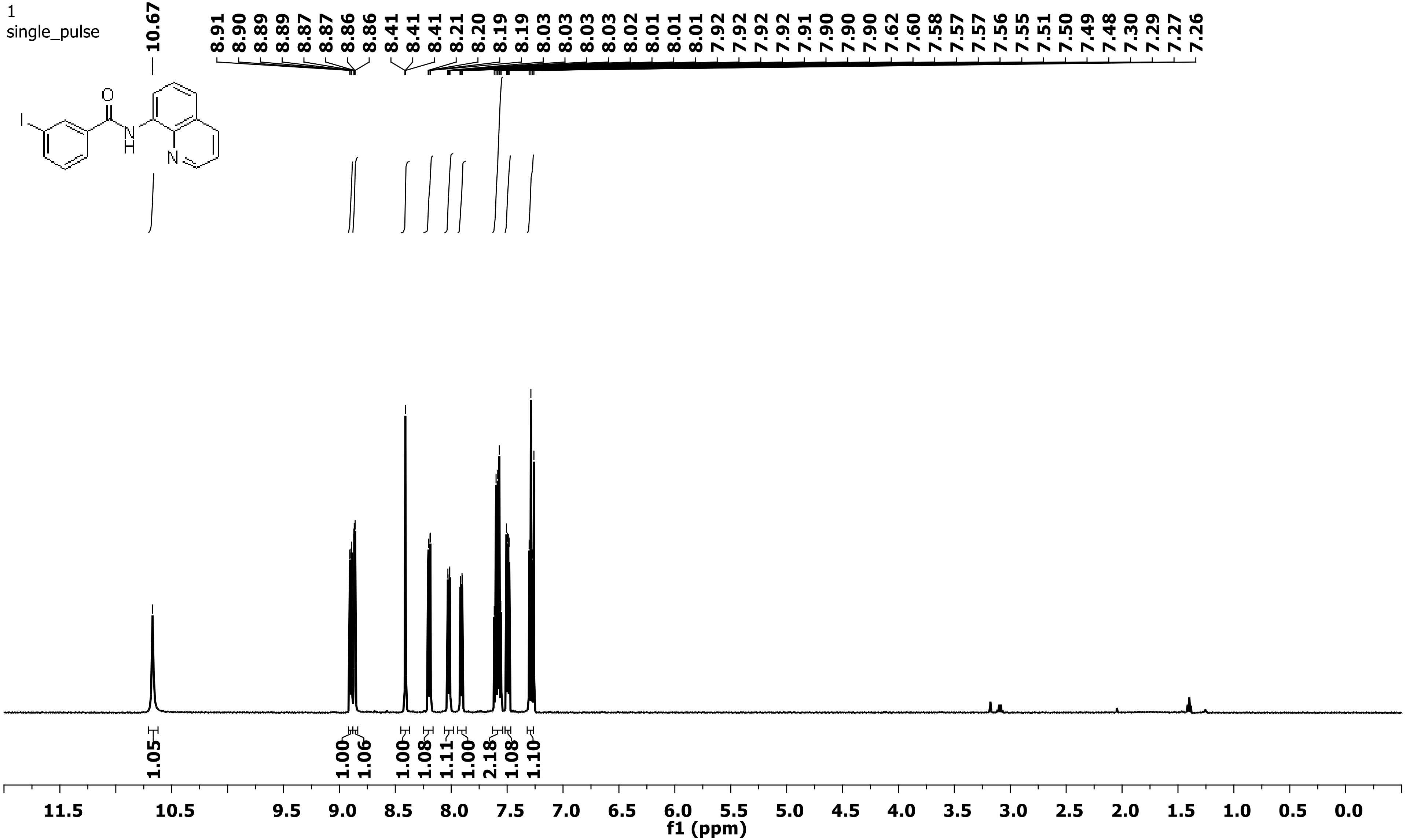
NMR spectra

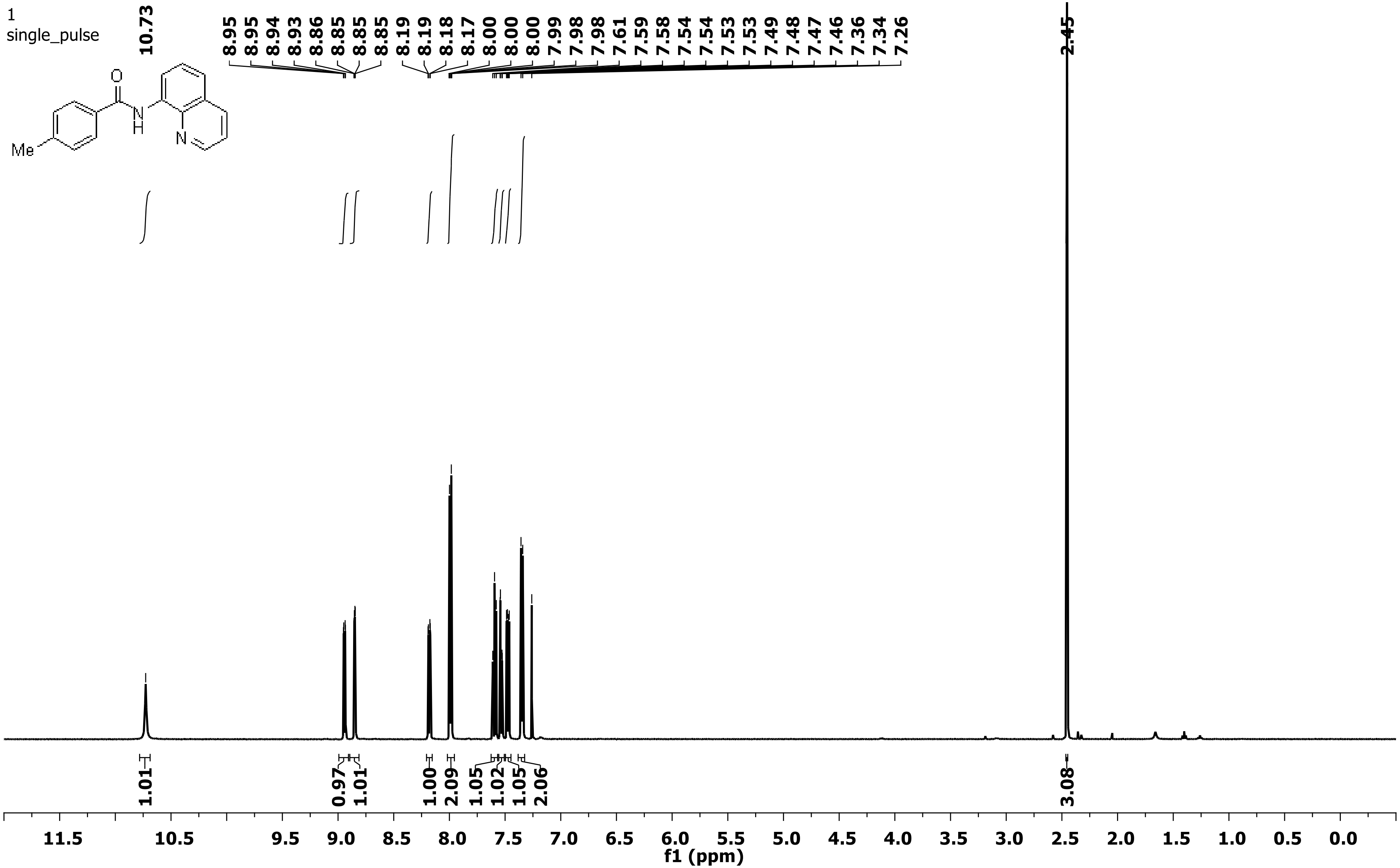

1
single_pulse
10.76
8.96 8.96 8.94 8.94 8.86 8.86 8.85 8.85
8.10 8.09 8.09 7.62 7.60 7.59 7.59 7.58 7.58 7.58 7.57 7.57 7.56 7.55 7.54 7.54 7.49 7.49 7.48 7.47 7.26
1.00
1.02 0.98
1.03 2.02
4.99 1.07
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

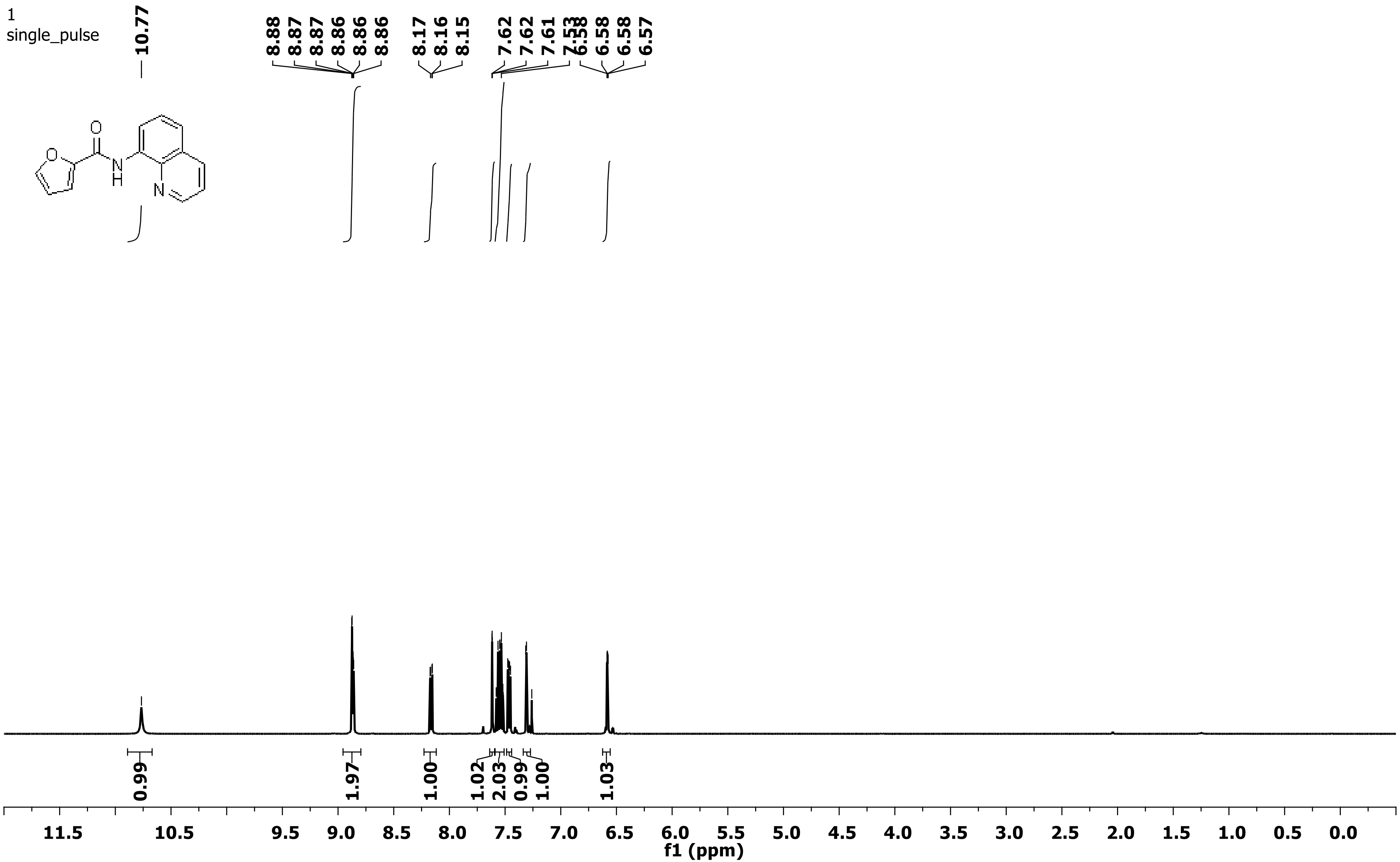

1
single_pulse
10.80
8.93 8.93 8.92 8.92 8.87 8.86 8.86 8.85 8.22 8.22 8.21 8.20 8.19 7.83 7.82 7.63 7.62 7.60 7.60 7.59 7.58 7.58 7.52 7.51 7.50 7.49 7.26
O
N
H
N
F3C
1.00
1.06
0.99
3.07
2.00
2.06
1.03
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

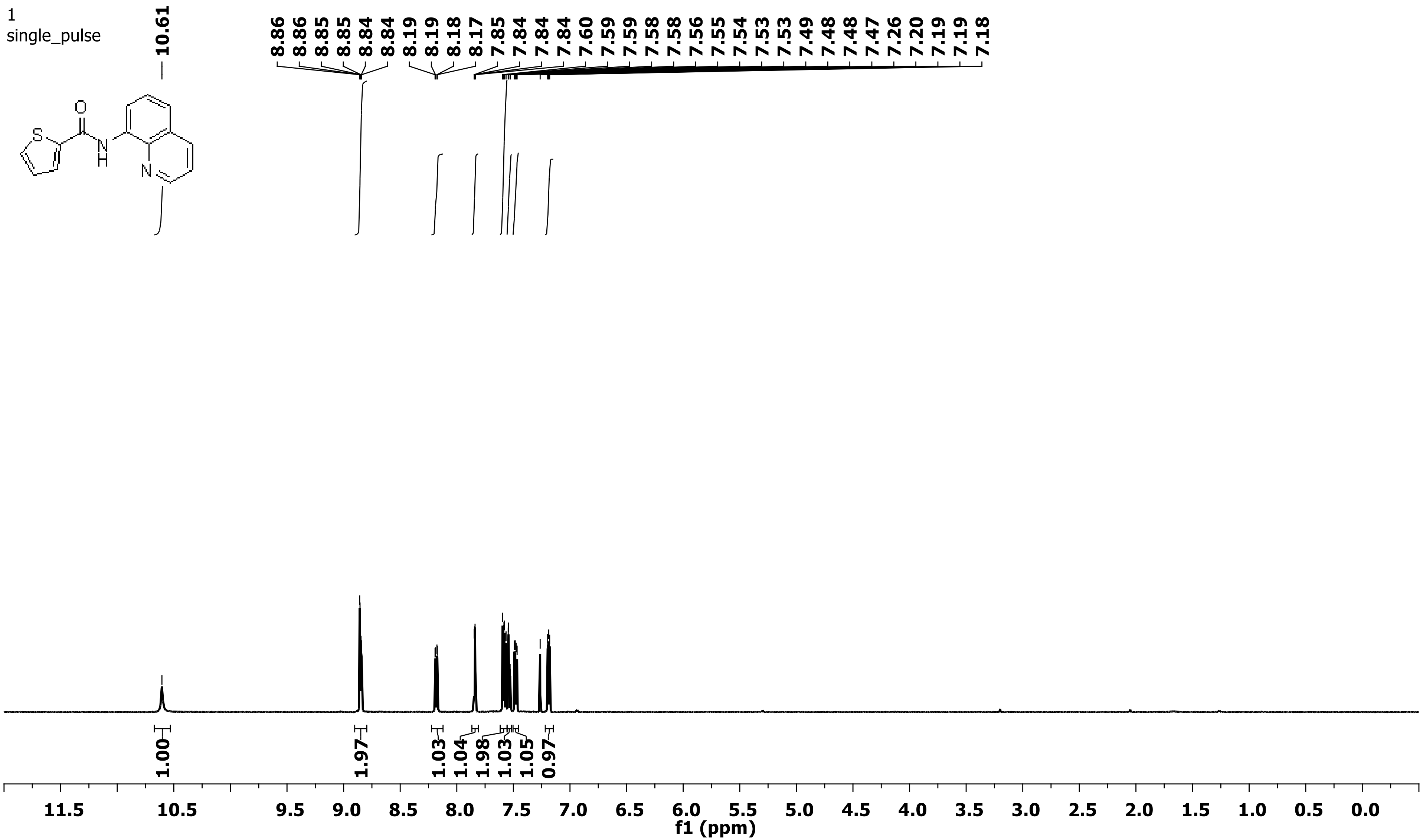

1
single_pulse
10.71
8.91 8.91 8.89 8.89 8.85 8.85 8.84 8.84 8.20 8.20 8.18 8.18 7.96 7.95 7.94 7.94 7.69 7.68 7.67 7.67 7.61 7.59 7.58 7.56 7.56 7.55 7.54 7.50 7.49 7.48 7.47 7.25
O
N
H
N
Br
0.96
1.01
0.99
1.00
2.04
1.93
2.07
1.05
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

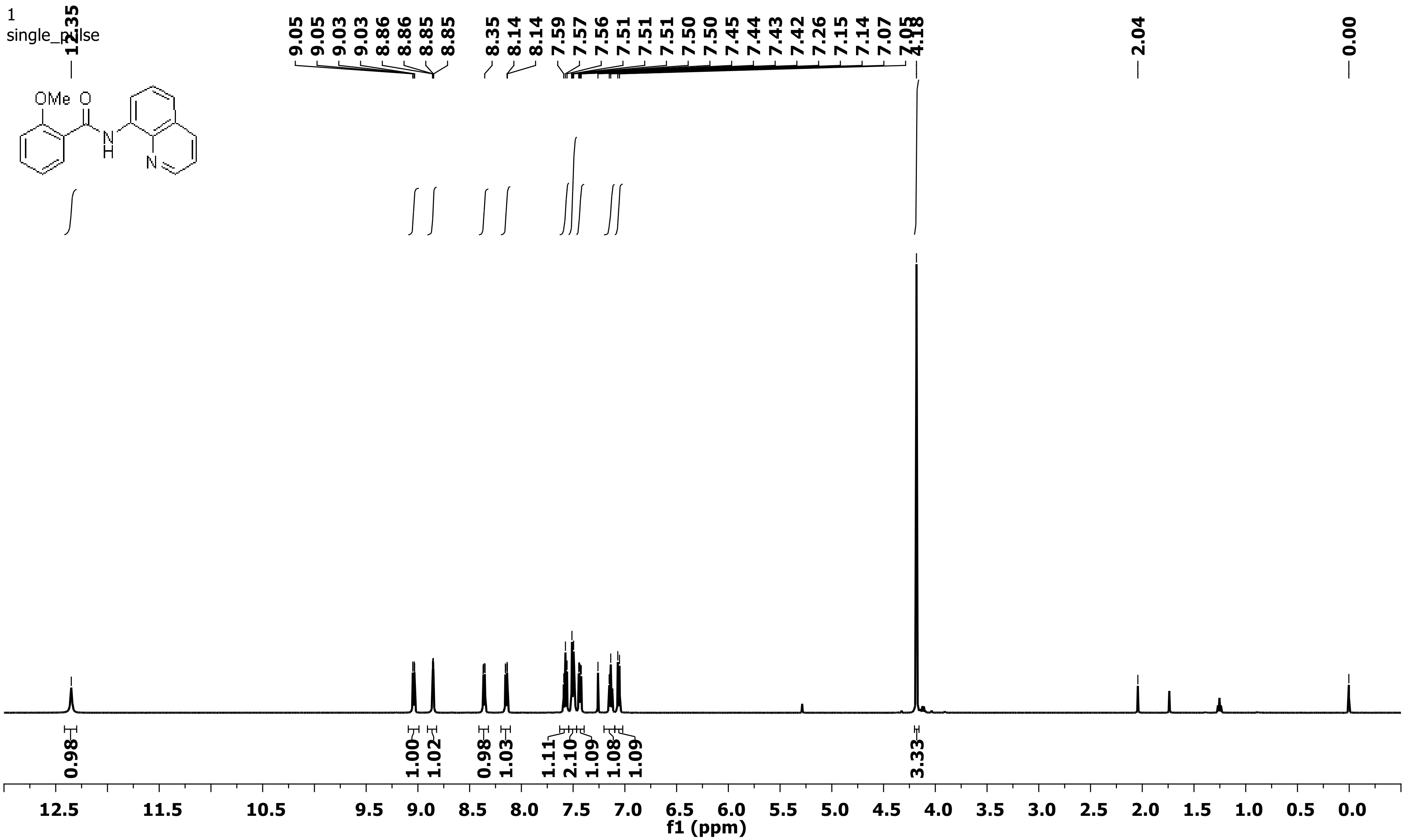

1
single_pulse
10.83
8.92
8.92
8.91
8.90
8.88
8.87
8.87
8.86
8.40
8.25
8.23
7.63
7.62
7.61
7.60
7.54
7.53
7.52
7.51
7.26
O
N
H
N
O2N
1.05
1.00
0.99
1.97
3.05
2.05
1.04
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

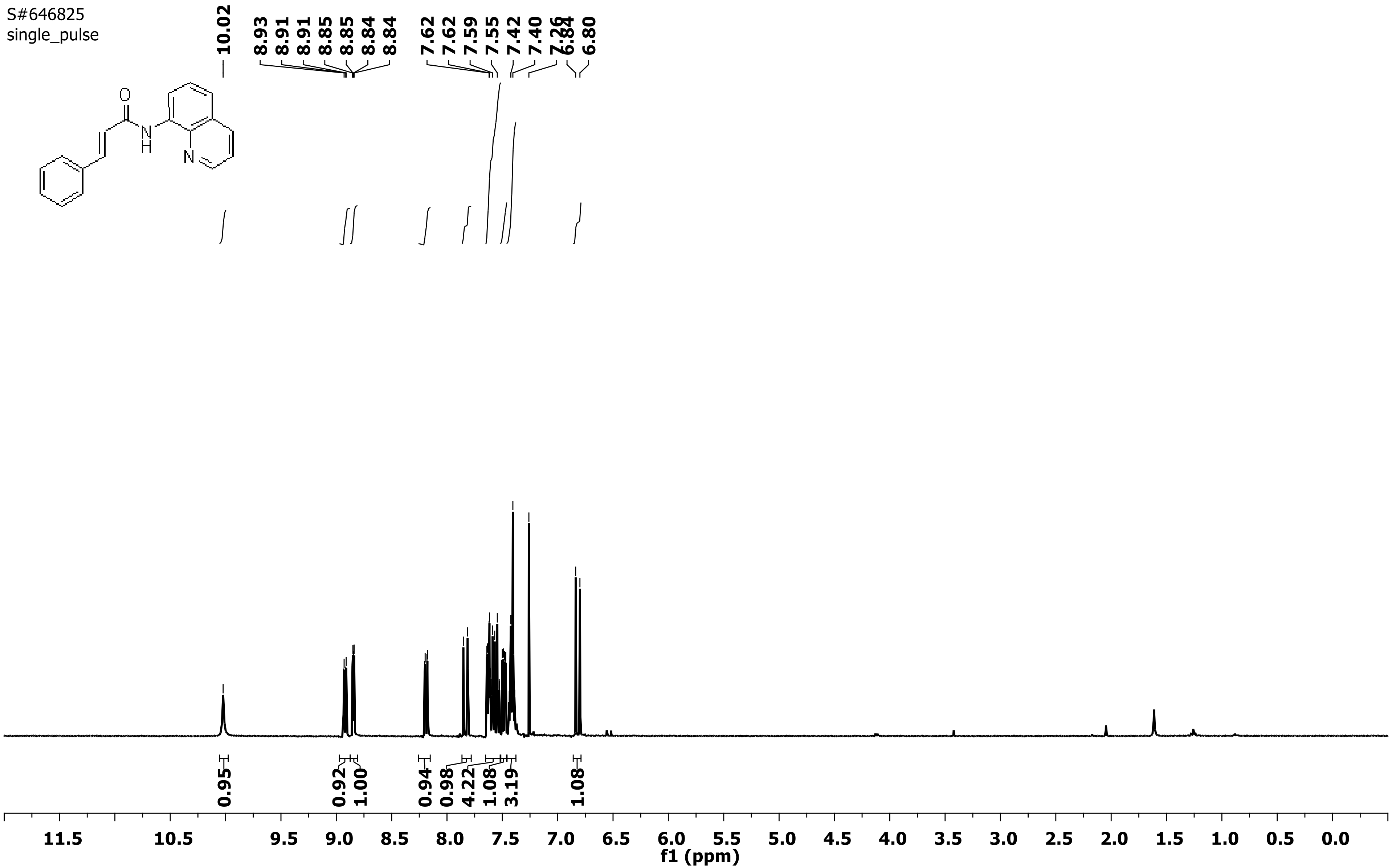

1
single_pulse
10.67
8.91 8.90 8.89 8.89 8.87 8.87 8.86 8.86 8.41 8.41 8.41 8.21 8.20 8.19 8.19 8.03 8.03 8.03 8.03 8.02 8.01 8.01 8.01 7.92 7.92 7.92 7.92 7.91 7.90 7.90 7.90 7.62 7.60 7.58 7.57 7.57 7.56 7.55 7.51 7.50 7.49 7.48 7.30 7.29 7.27 7.26
1.05
1.00
1.06
1.00
1.08
1.11
1.00
2.18
1.08
1.10
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)

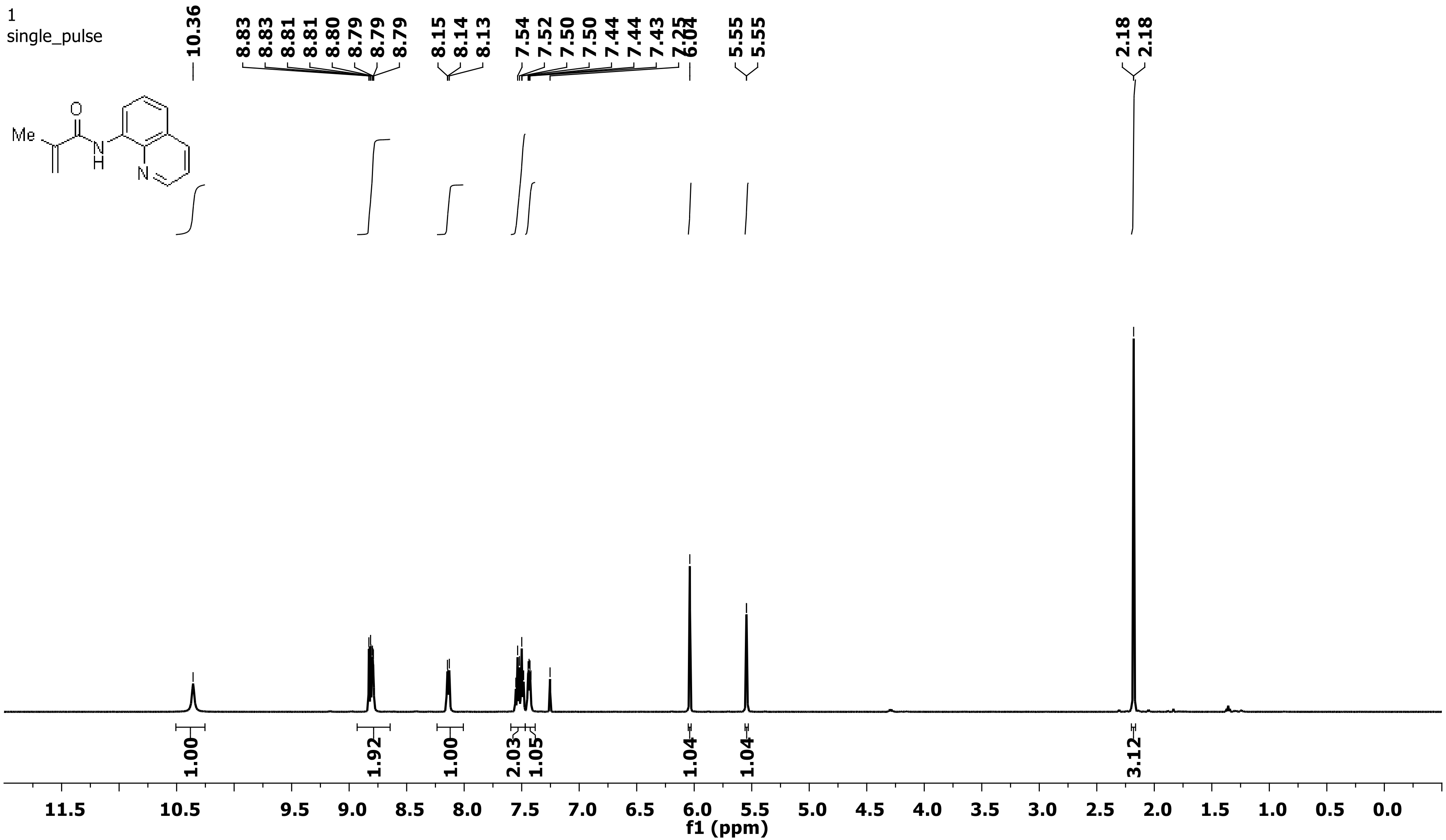

1
single_pulse
O
N
H
N
Me
10.73
8.95
8.95
8.94
8.93
8.86
8.85
8.85
8.85
8.19
8.19
8.18
8.17
8.00
8.00
8.00
7.99
7.98
7.98
7.61
7.59
7.58
7.54
7.54
7.53
7.53
7.49
7.48
7.47
7.46
7.36
7.34
7.26
2.45
1.01
0.97
1.01
1.00
2.09
1.05
1.02
1.02
1.05
2.06
3.08
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

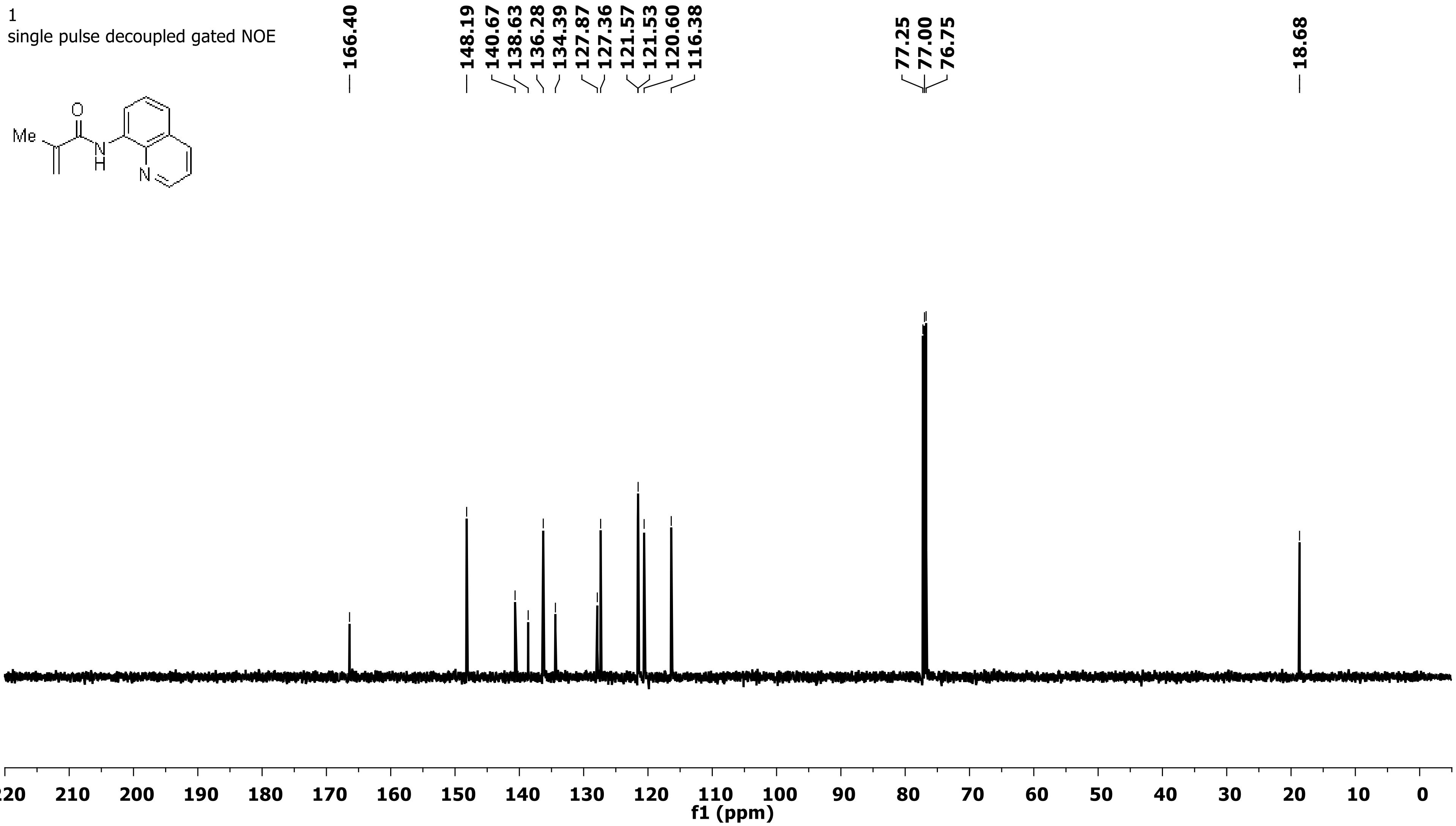

1
single_pulse
10.77
8.88
8.87
8.87
8.86
8.86
8.86
8.17
8.16
8.15
7.62
7.62
7.61
7.53
6.58
6.58
6.58
6.57
N
H
O
O
N
0.99
1.97
1.00
1.02
2.03
0.99
1.00
1.03
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

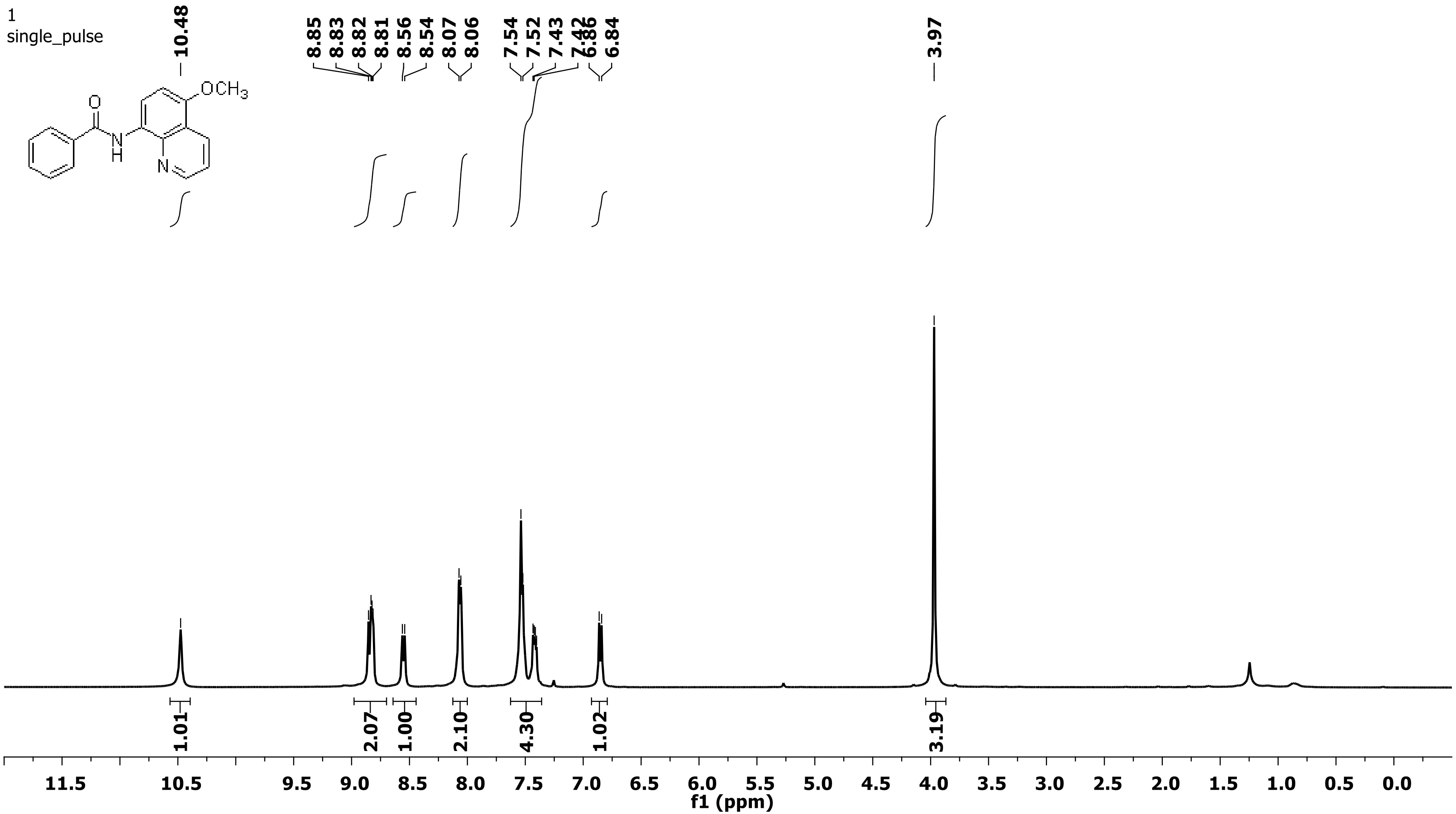

1
single_pulse
10.61
8.86 8.86 8.85 8.85 8.84 8.84 8.19 8.19 8.18 8.17 7.85 7.84 7.84 7.84 7.60 7.59 7.59 7.58 7.58 7.56 7.55 7.54 7.53 7.53 7.49 7.48 7.48 7.47 7.26 7.20 7.19 7.19 7.18
1.00 1.97 1.03 1.04 1.98 1.03 1.05 0.97
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


1
single_pulse
12.35
9.05 9.05 9.03 9.03 8.86 8.86 8.85 8.85
8.35 8.14 8.14 7.59 7.57 7.56 7.51 7.51 7.50 7.50 7.45 7.44 7.43 7.42 7.26 7.15 7.14 7.07 7.05 4.18
2.04
0.00
OMe O
N
H
N
0.98
1.00 1.02
0.98 1.03
1.11 2.10 1.09 1.08 1.09
3.33
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)


S#646825
single_pulse
10.02
8.93
8.91
8.91
8.85
8.85
8.84
8.84
7.62
7.62
7.59
7.55
7.42
7.40
7.26
6.84
6.80
O
N
H
N
0.95
0.92
1.00
0.94
0.98
4.22
1.08
3.19
1.08
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)


1
single_pulse
10.36
8.83
8.83
8.81
8.81
8.80
8.79
8.79
8.79
8.15
8.14
8.13
7.54
7.52
7.50
7.50
7.44
7.44
7.43
7.25
6.04
5.55
5.55
2.18
2.18
Me
O
N
H
N
1.00
1.92
1.00
2.03
1.05
1.04
1.04
3.12
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)


1
single pulse decoupled gated NOE
Me
O
N
H
N
166.40
148.19
140.67
138.63
136.28
134.39
127.87
127.36
121.57
121.53
120.60
116.38
77.25
77.00
76.75
18.68
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


1
single_pulse
OCH3
O
N
H
N
10.48
8.85
8.83
8.82
8.81
8.56
8.54
8.07
8.06
7.54
7.52
7.43
7.42
6.86
6.84
3.97
1.01
2.07
1.00
2.10
4.30
1.02
3.19
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)

1
single_pulse

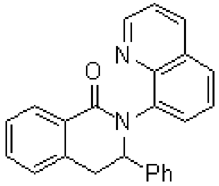
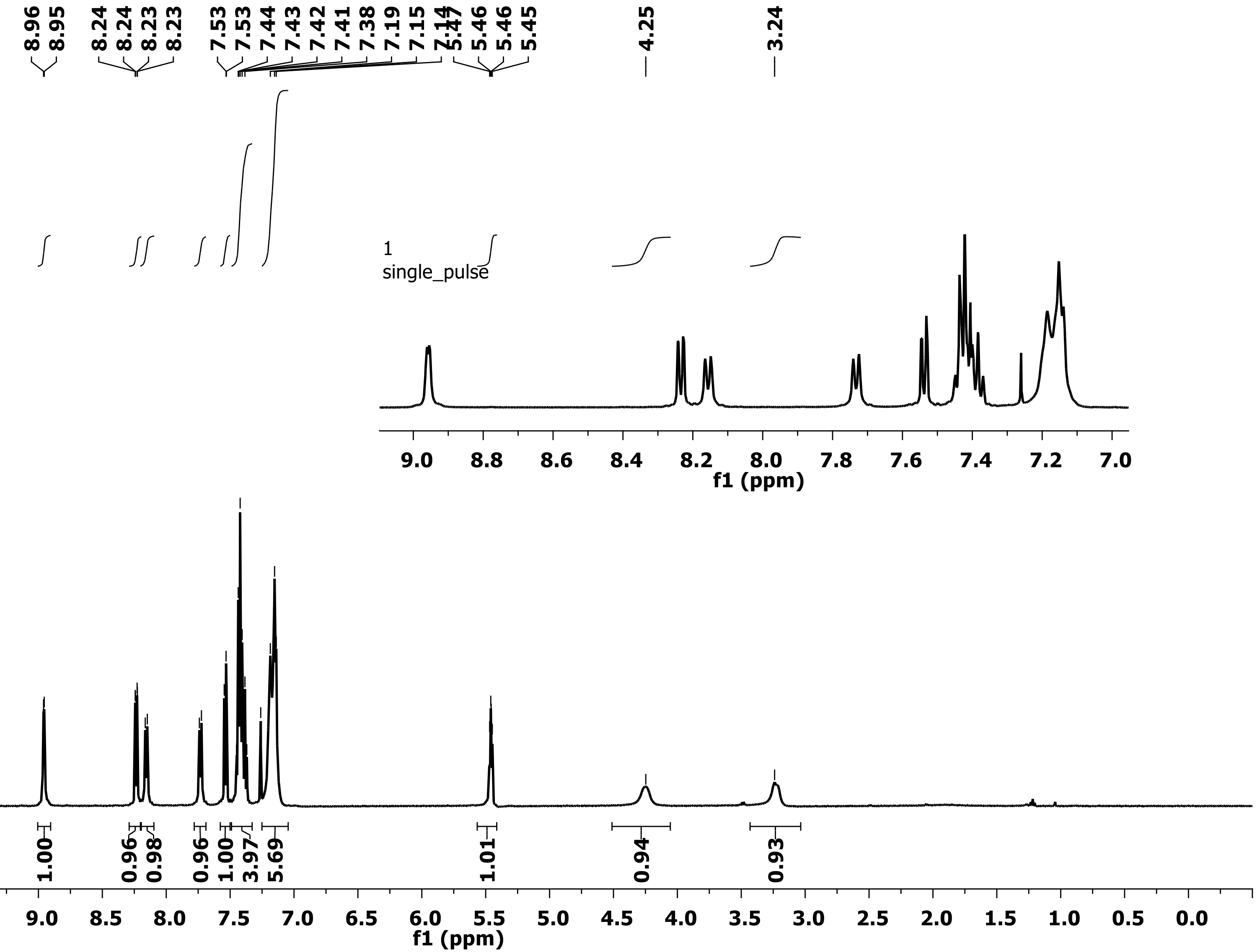


Table 2, Entry 1



1
single pulse decoupled gated NOE

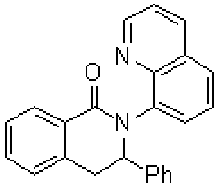
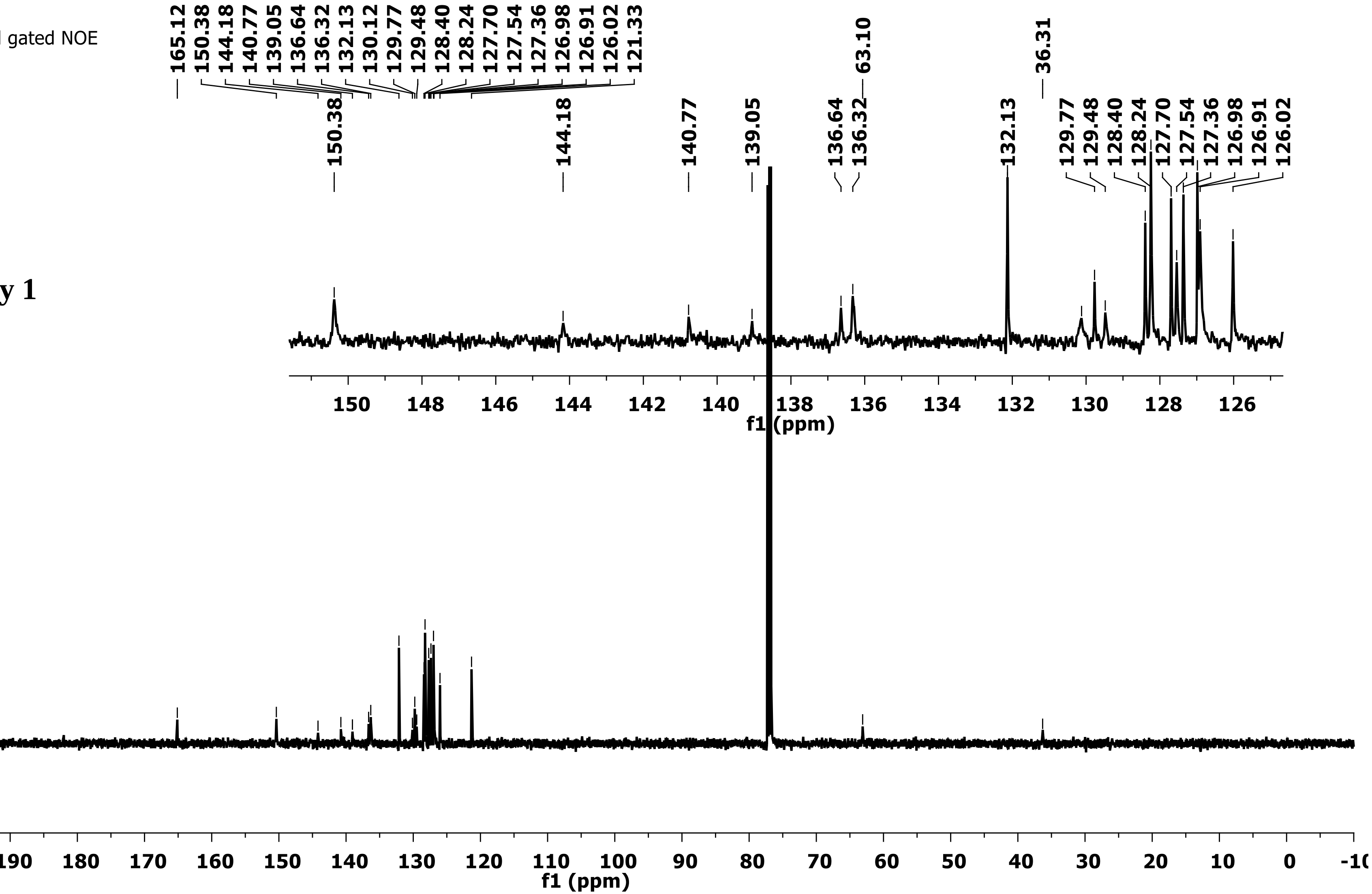


Table 2, Entry 1



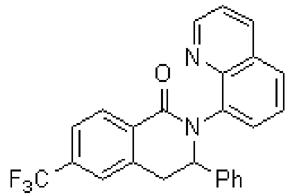
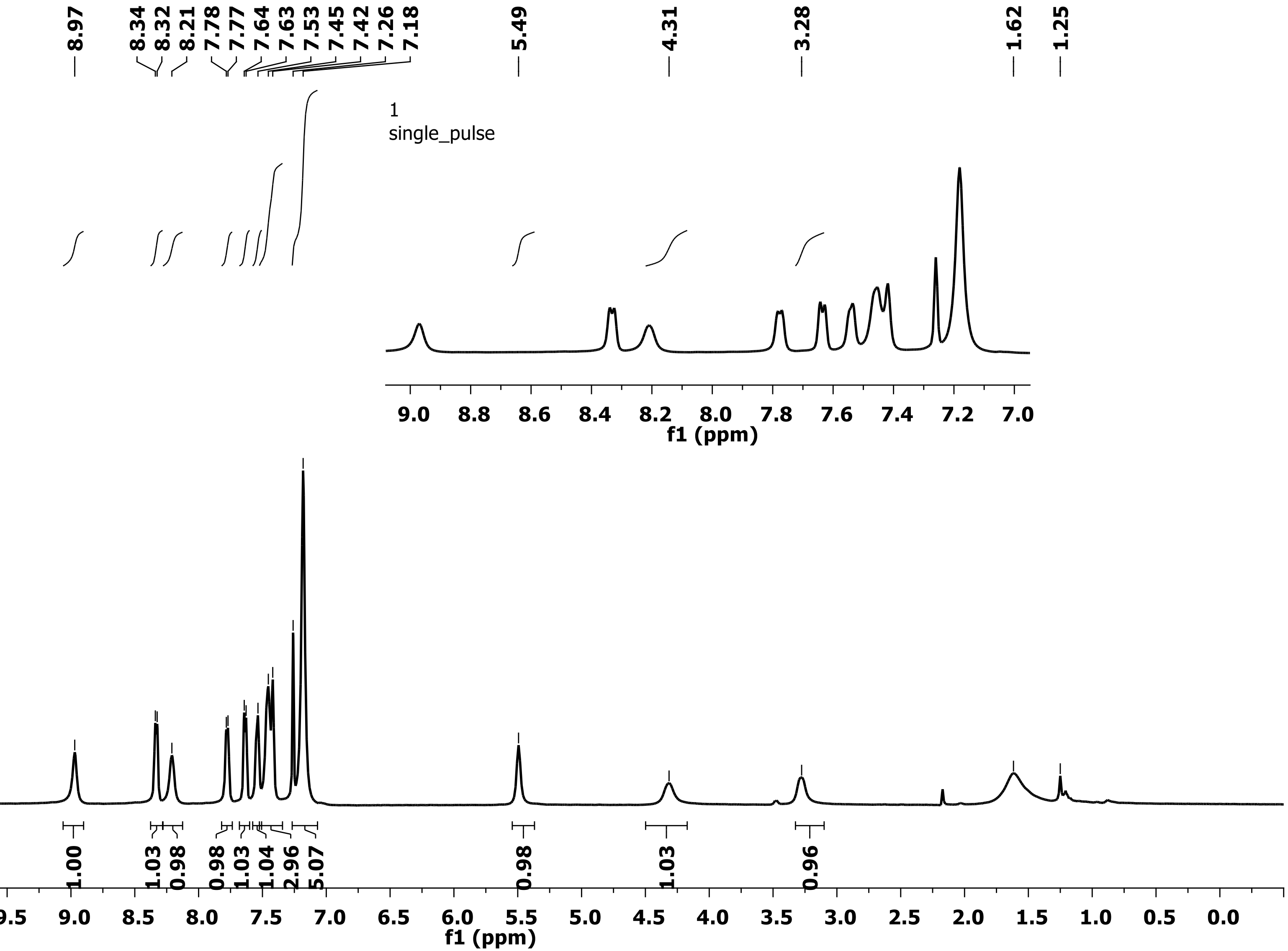
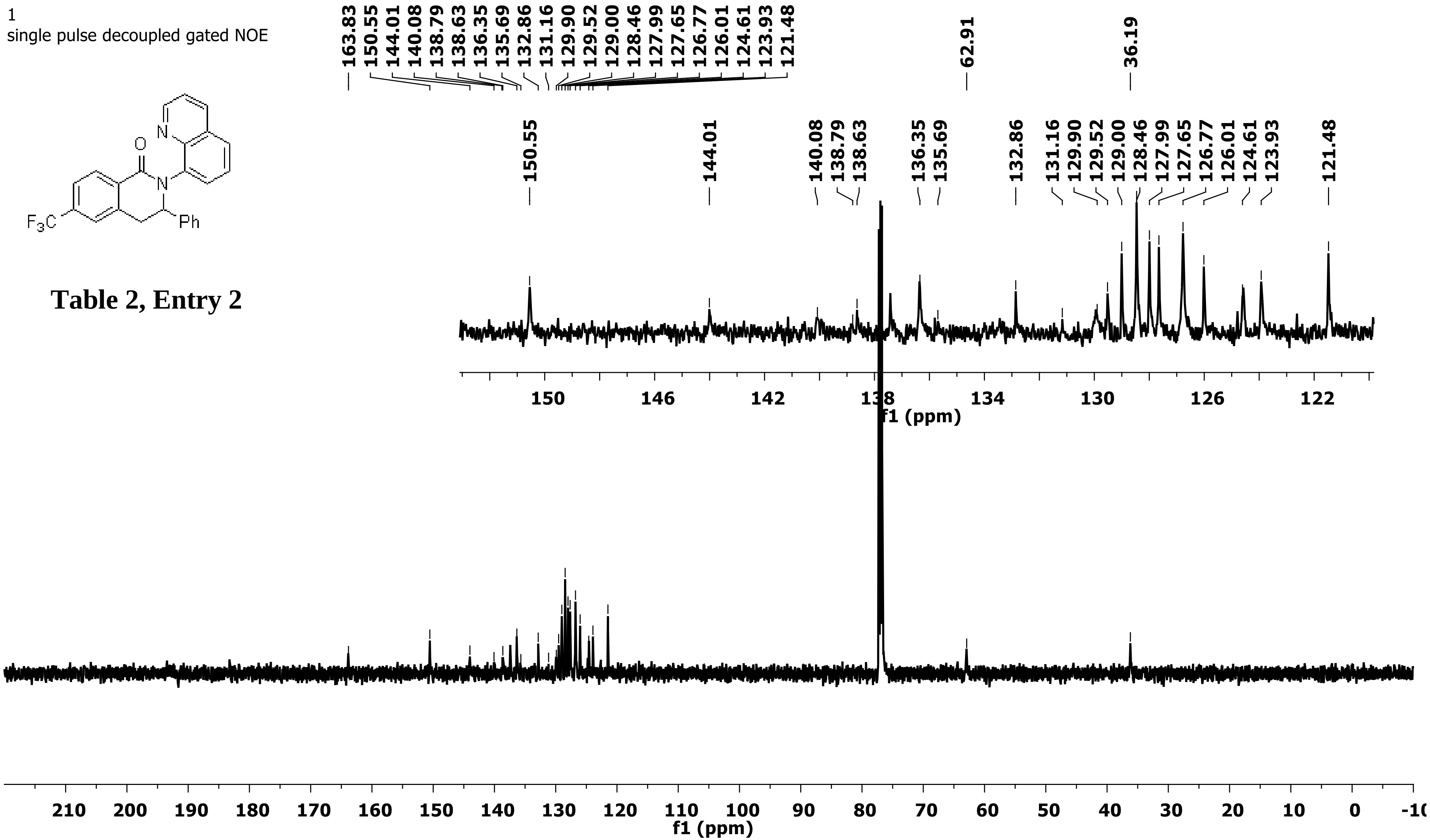


Table 2, Entry 2



1
single pulse decoupled gated NOE

Table 2, Entry 2



1
single_pulse

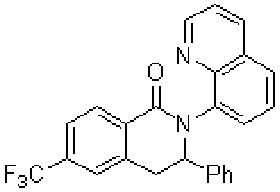
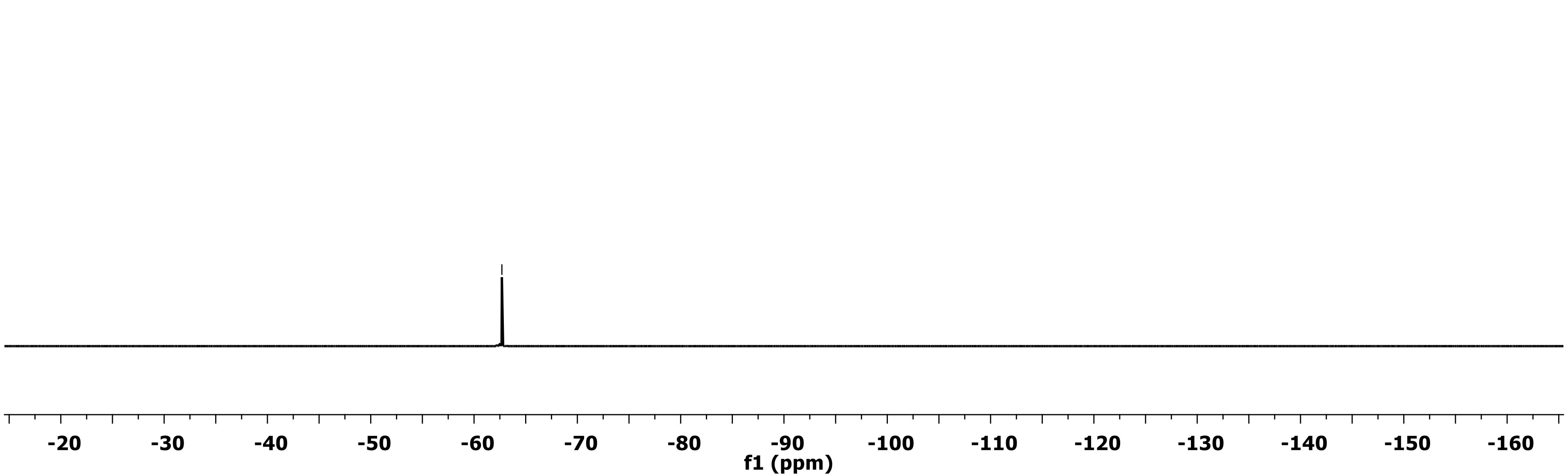


Table 2, Entry 2



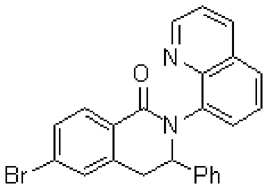
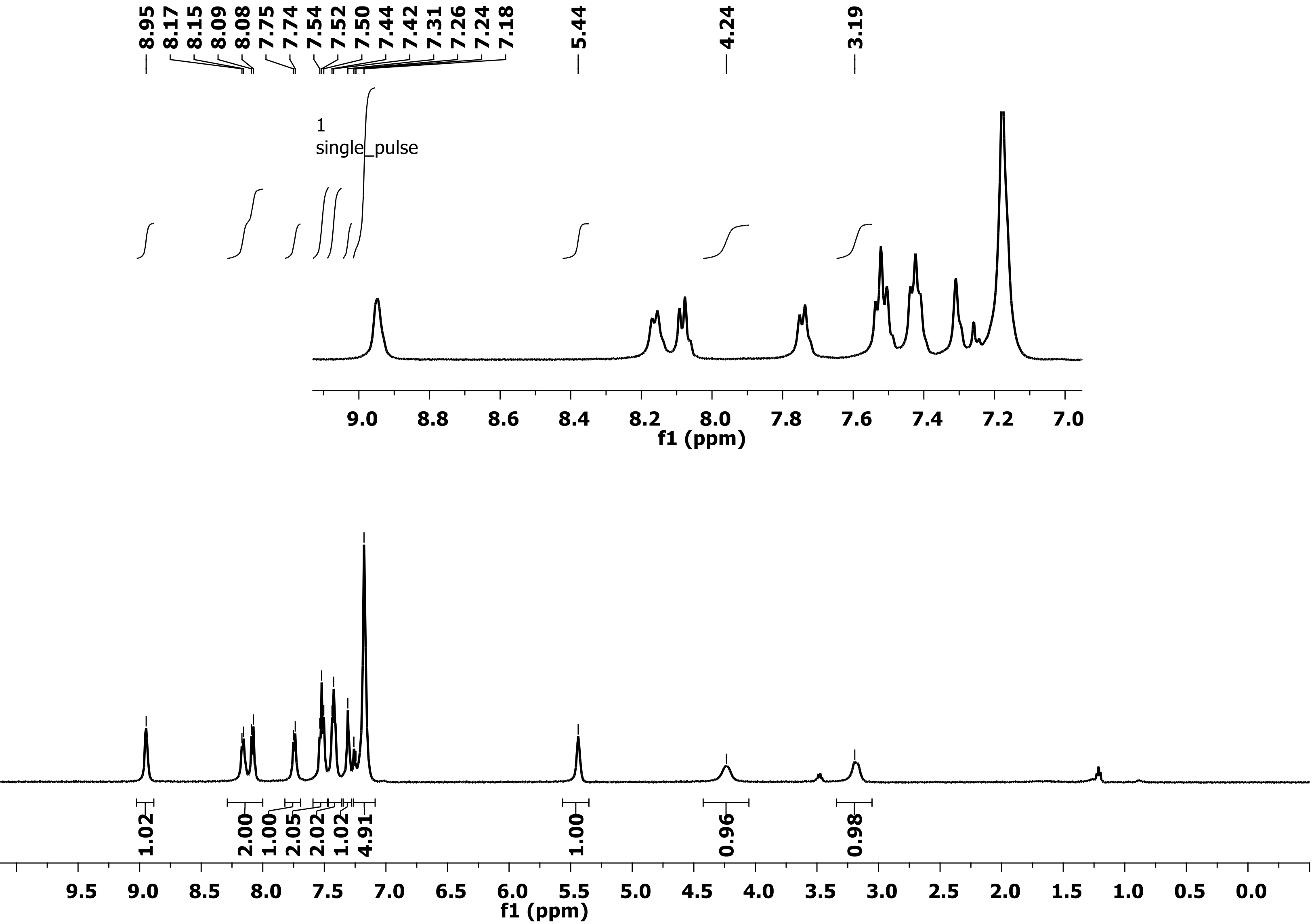


Table 2, Entry 3



1
single pulse decoupled gated NOE

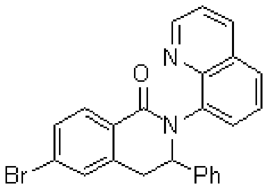
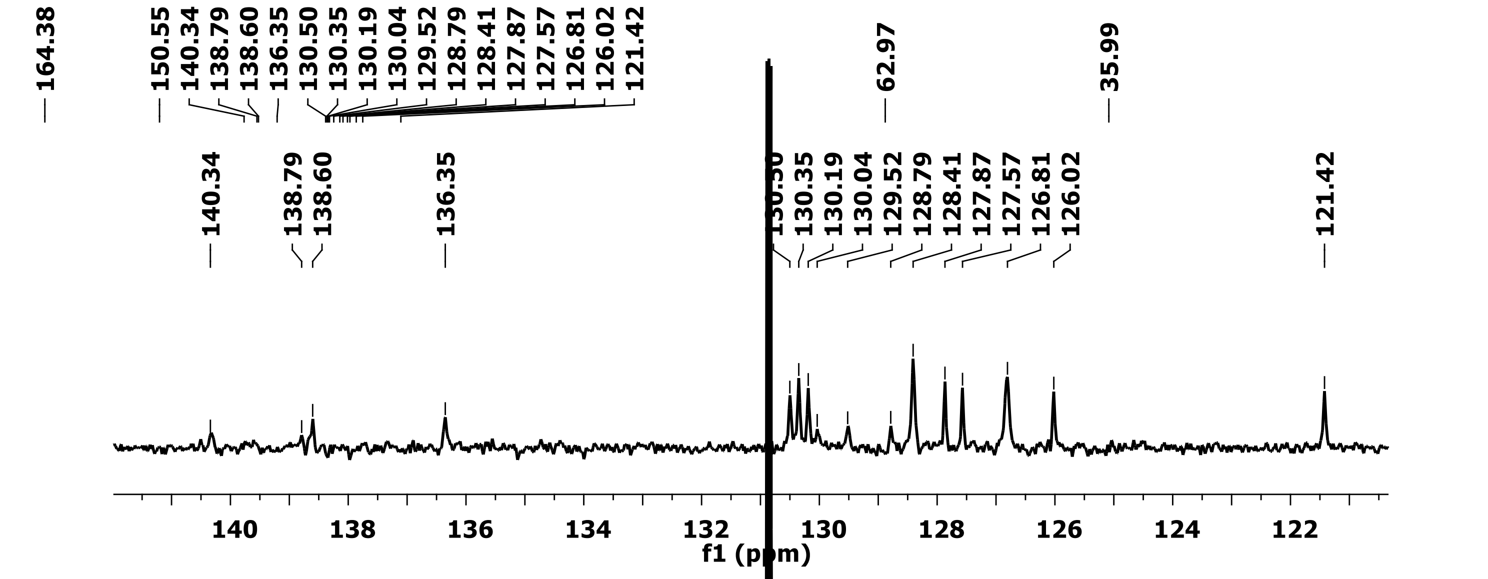
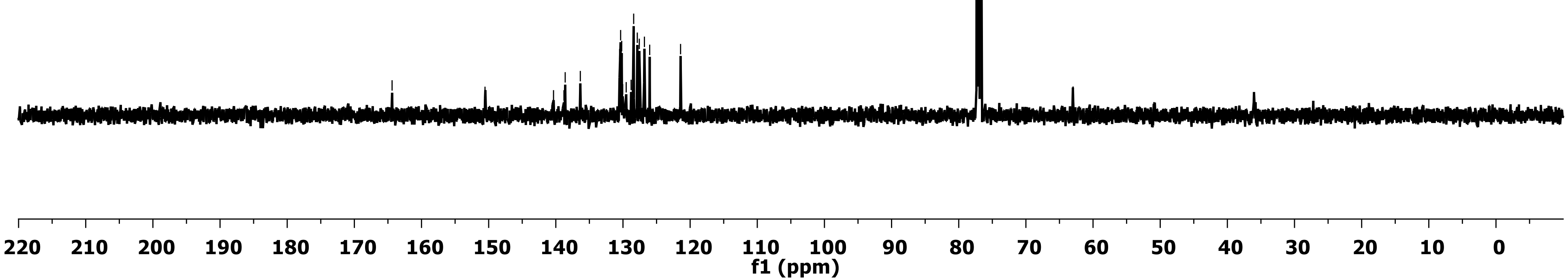
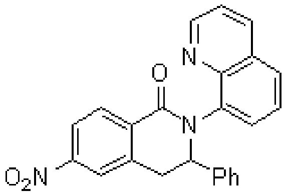


Table 2, Entry 3







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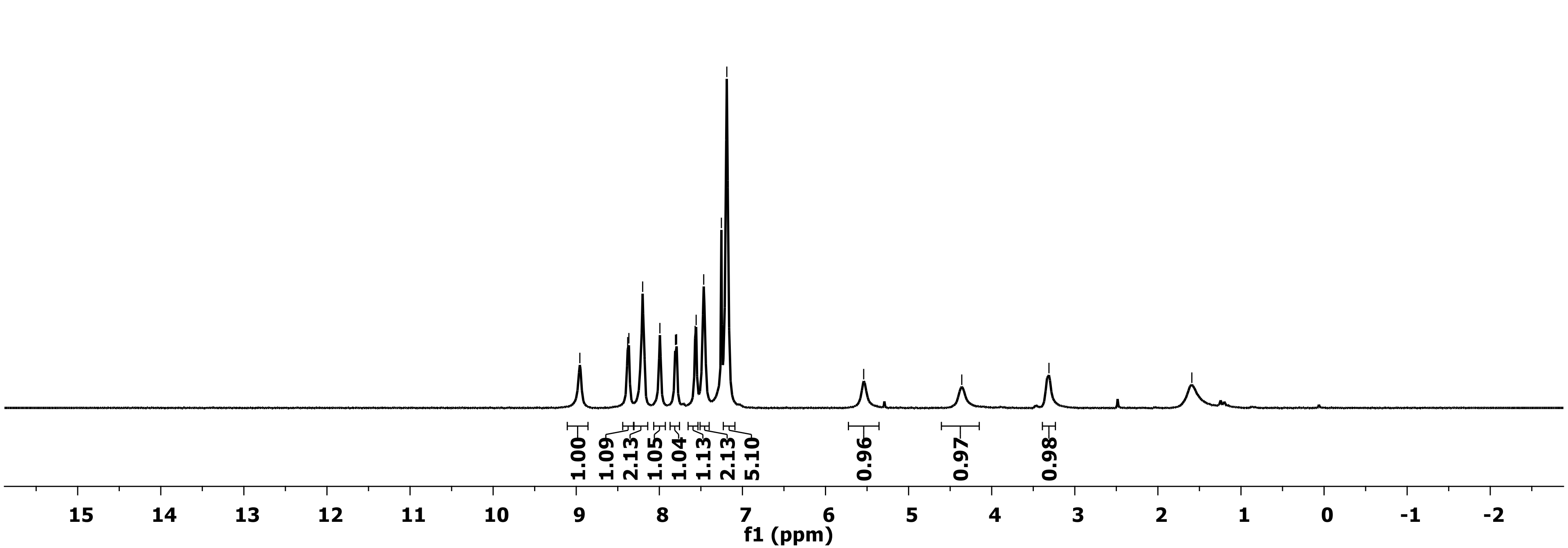
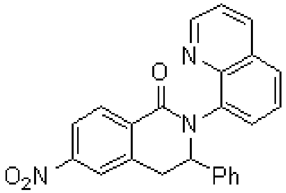


1
single pulse decoupled gated NOE



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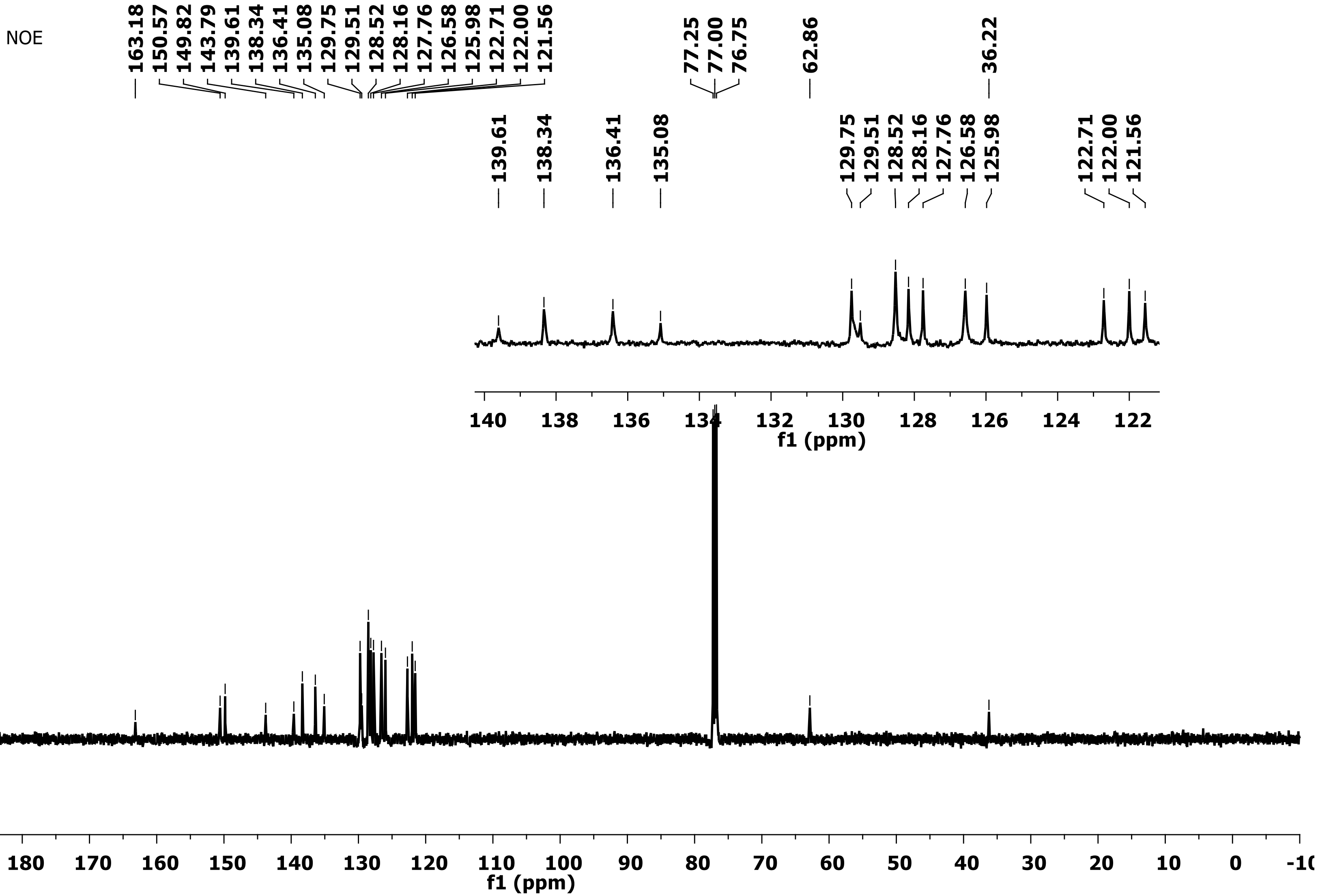
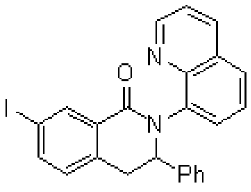


1
single_pulse



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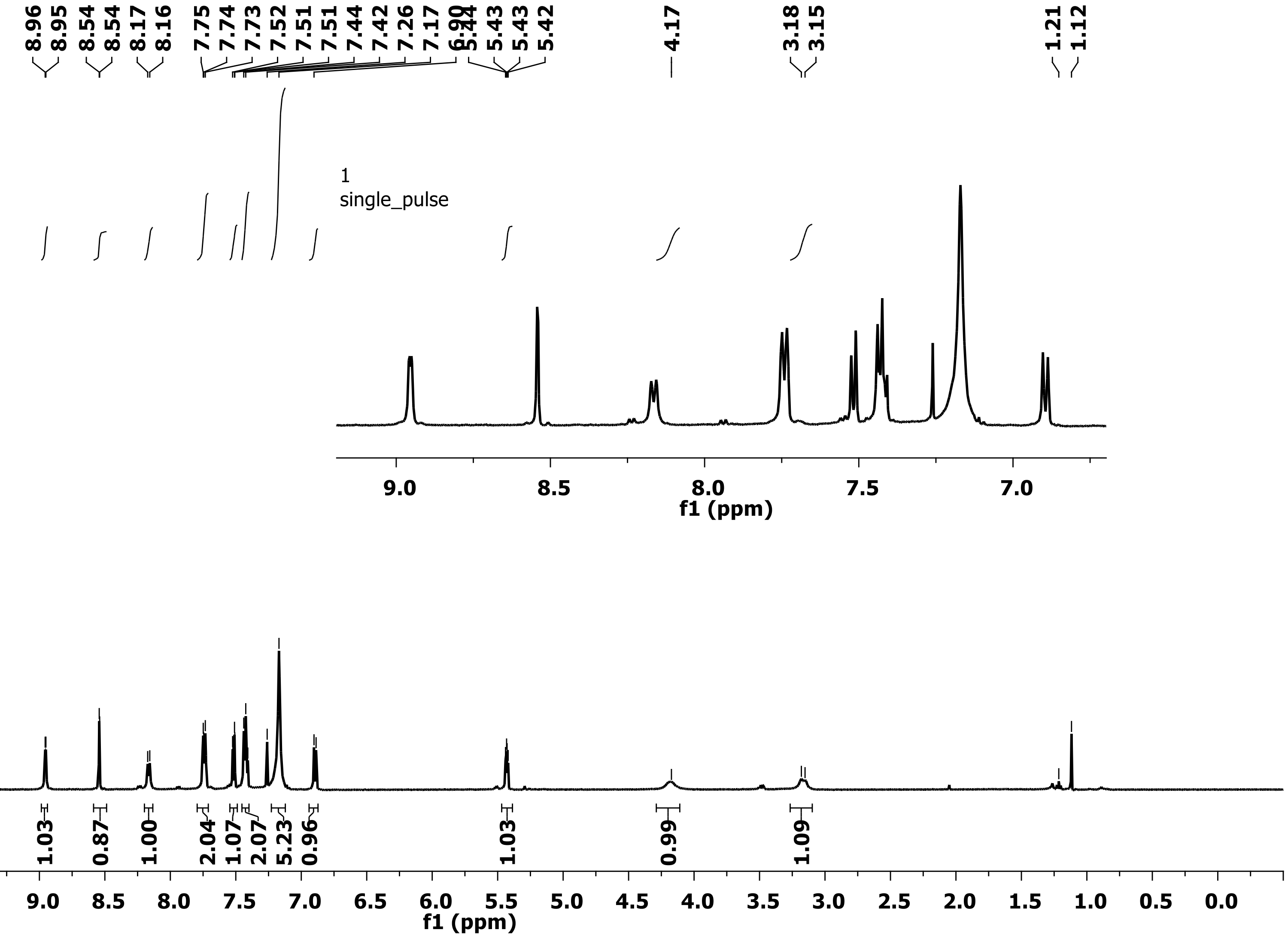


1
single pulse decoupled gated NOE

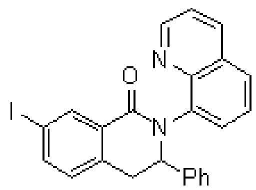
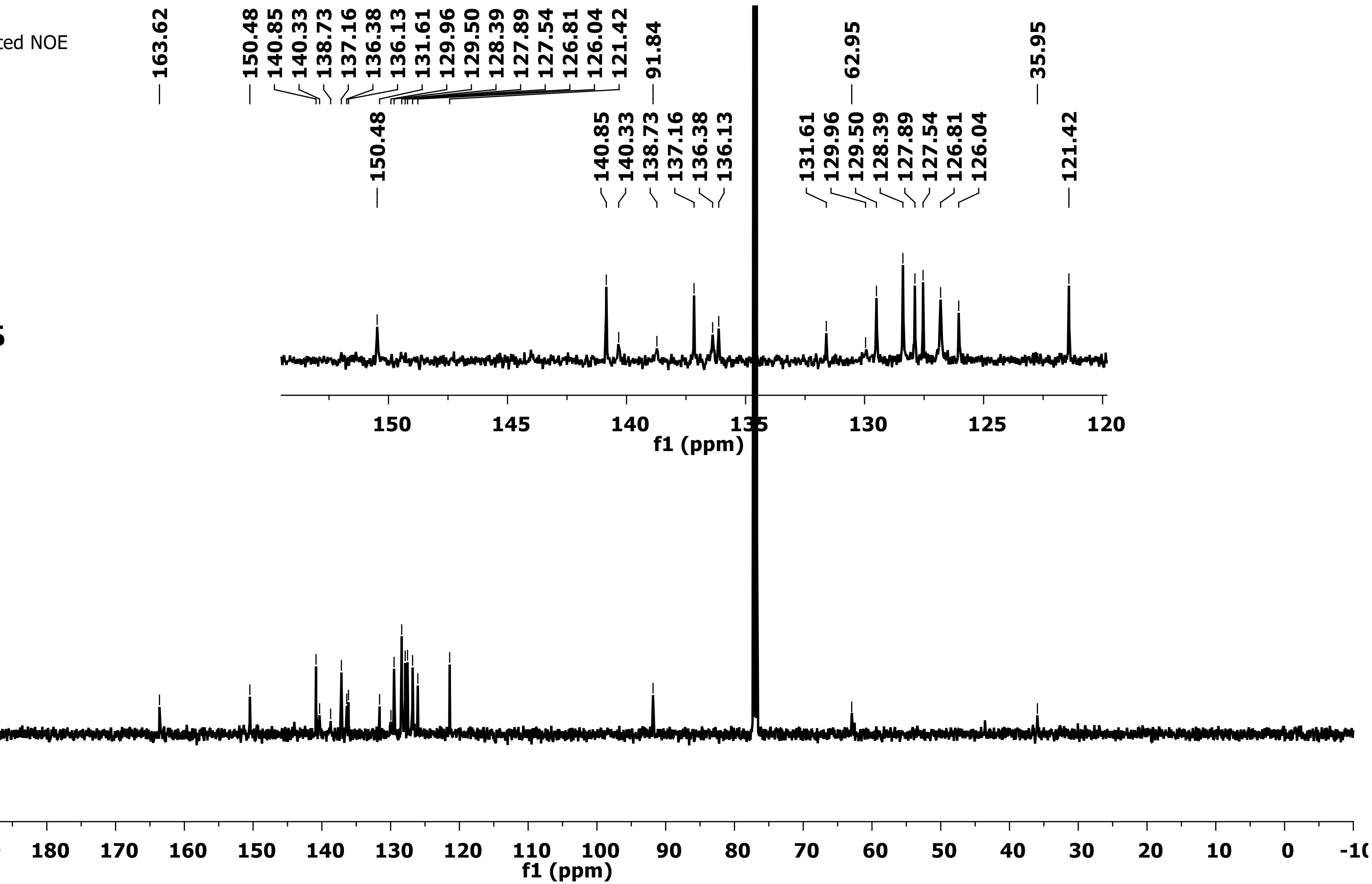


Table 2, Entry 5



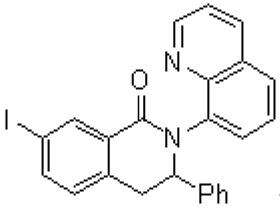
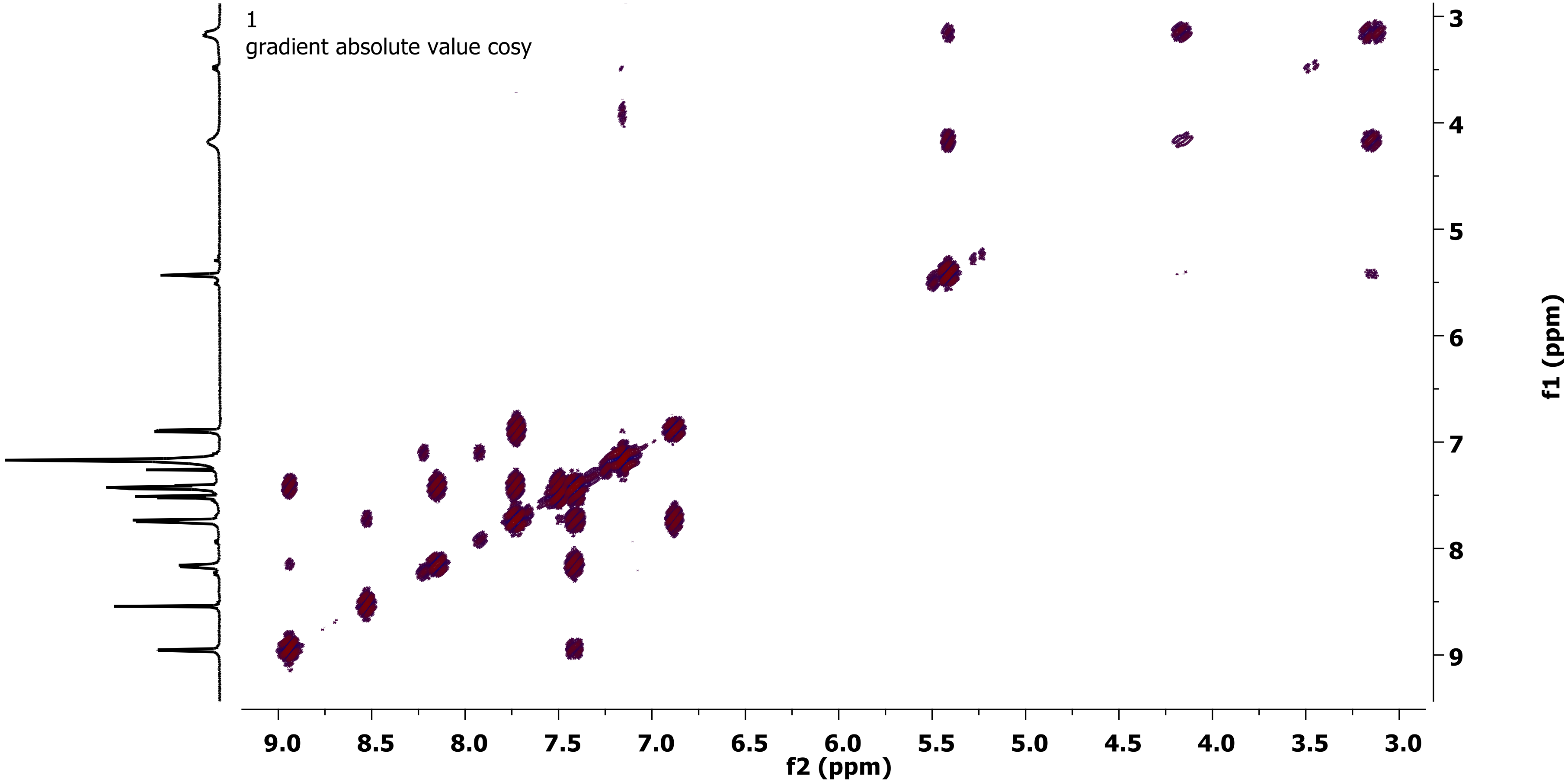


Table 2, Entry 5



1
single_pulse

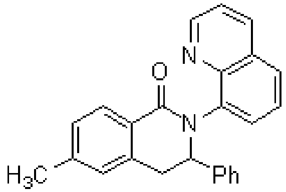
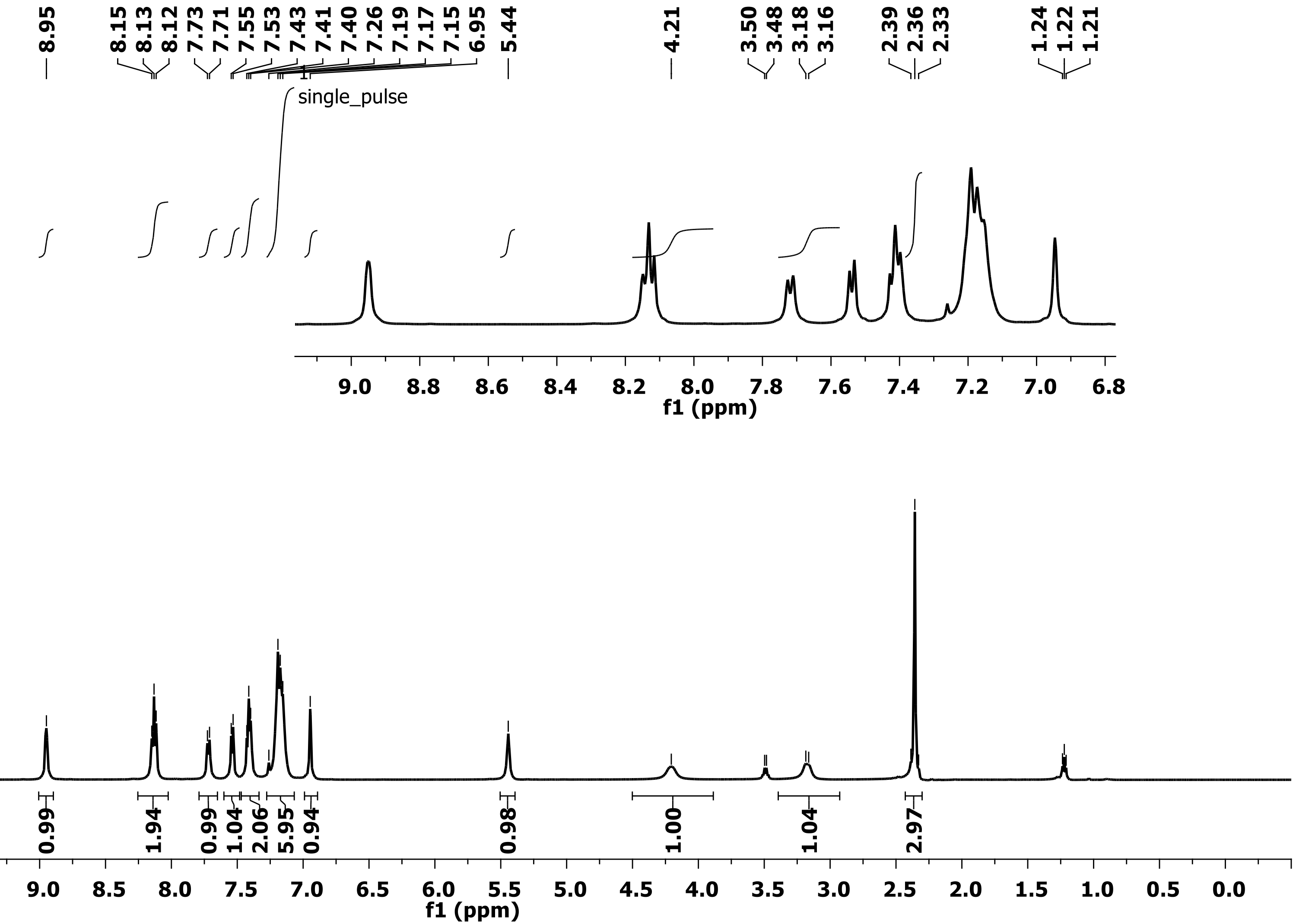


Table 2, Entry 6



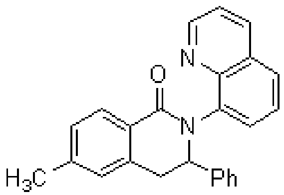
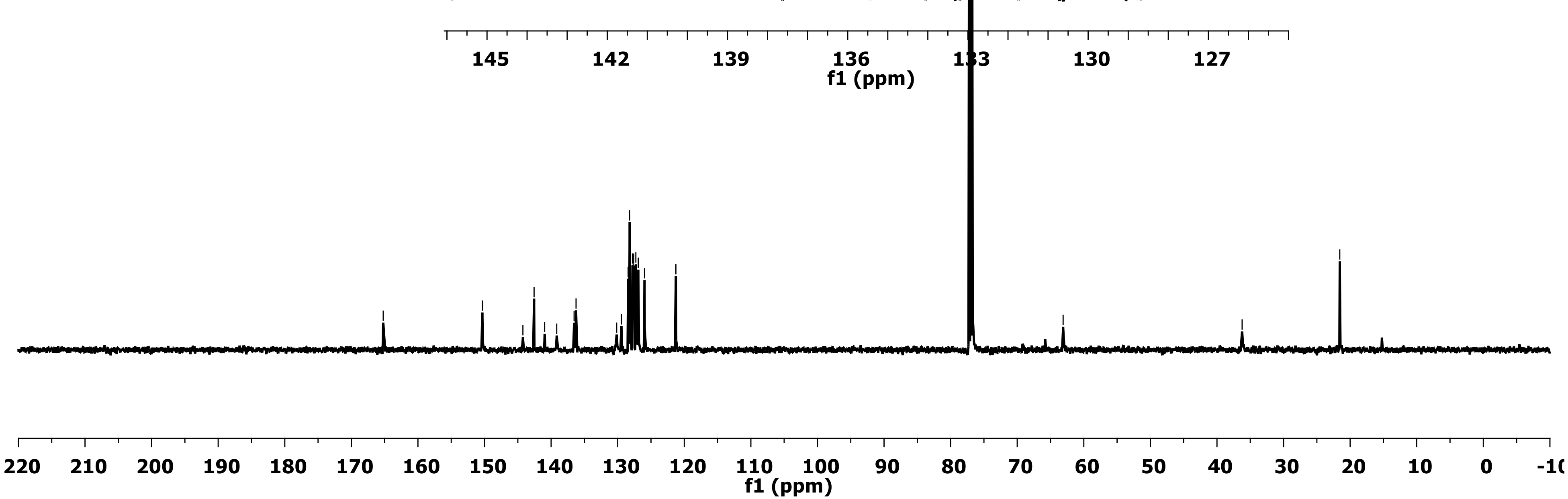


Table 2, Entry 1



1
single_pulse

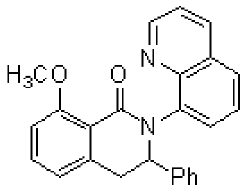
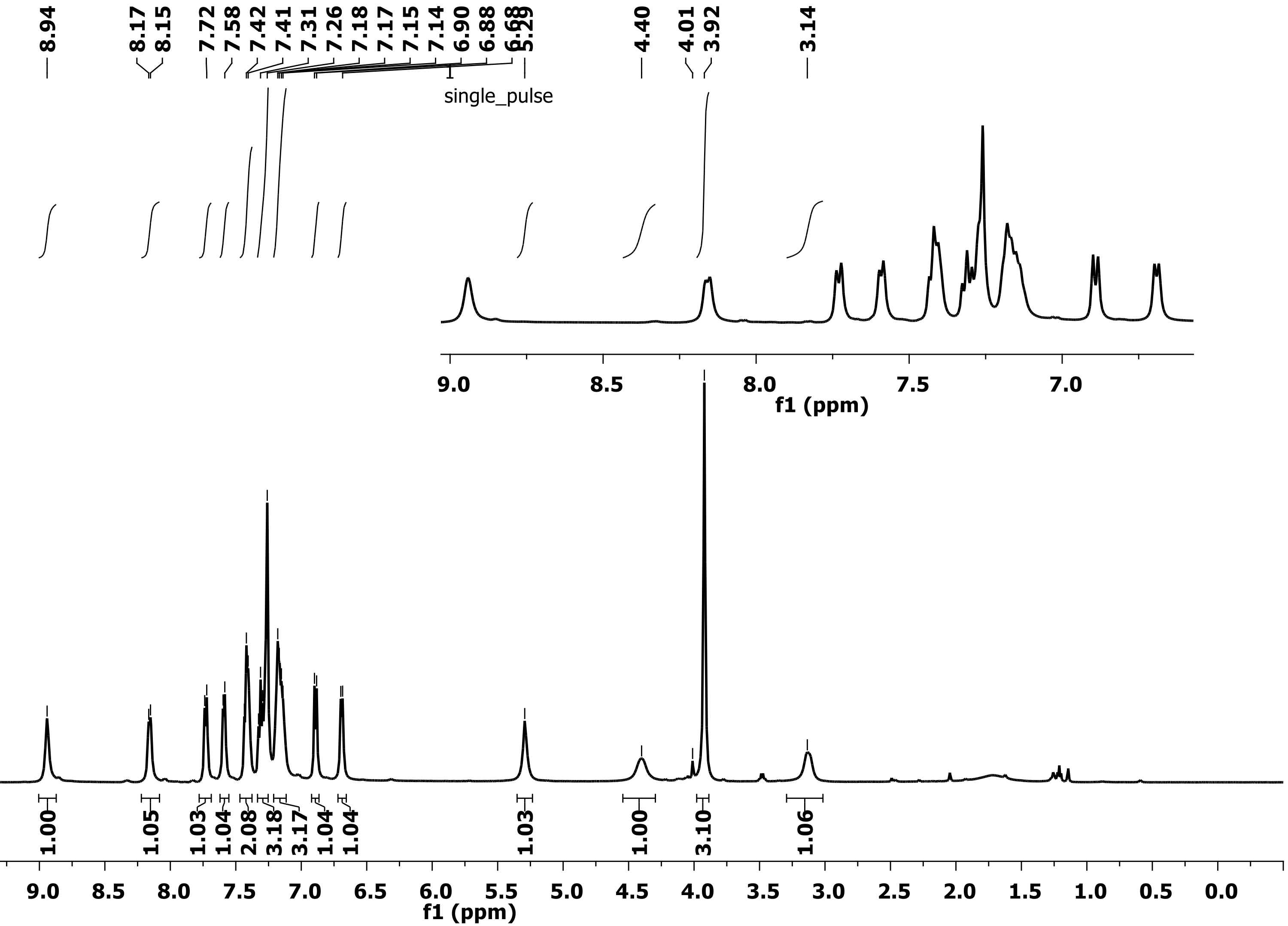


Table 2, Entry 7



1
single pulse decoupled gated NOE

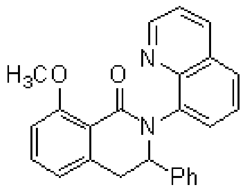
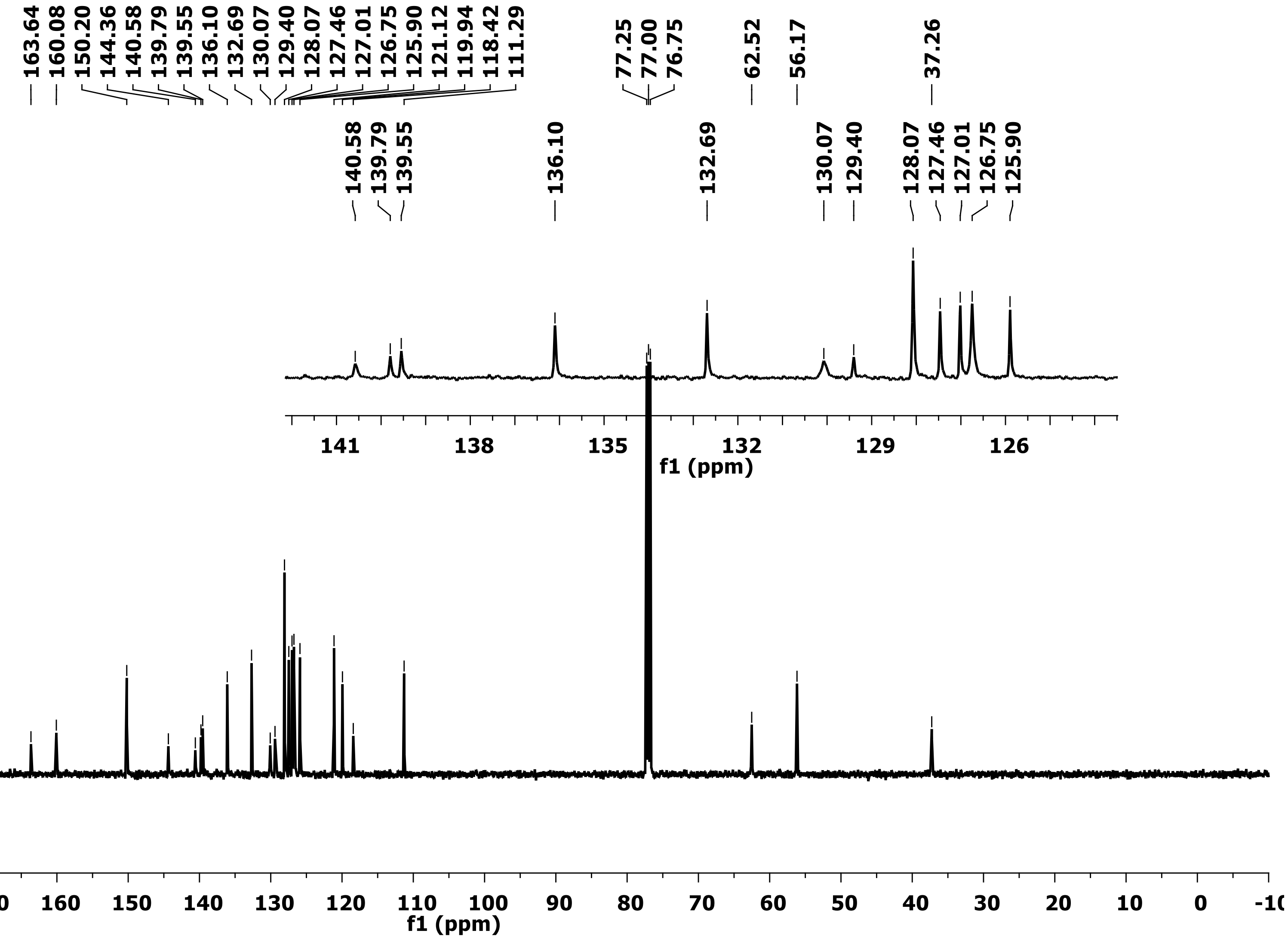


Table 2, Entry 7



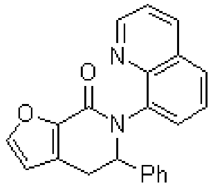
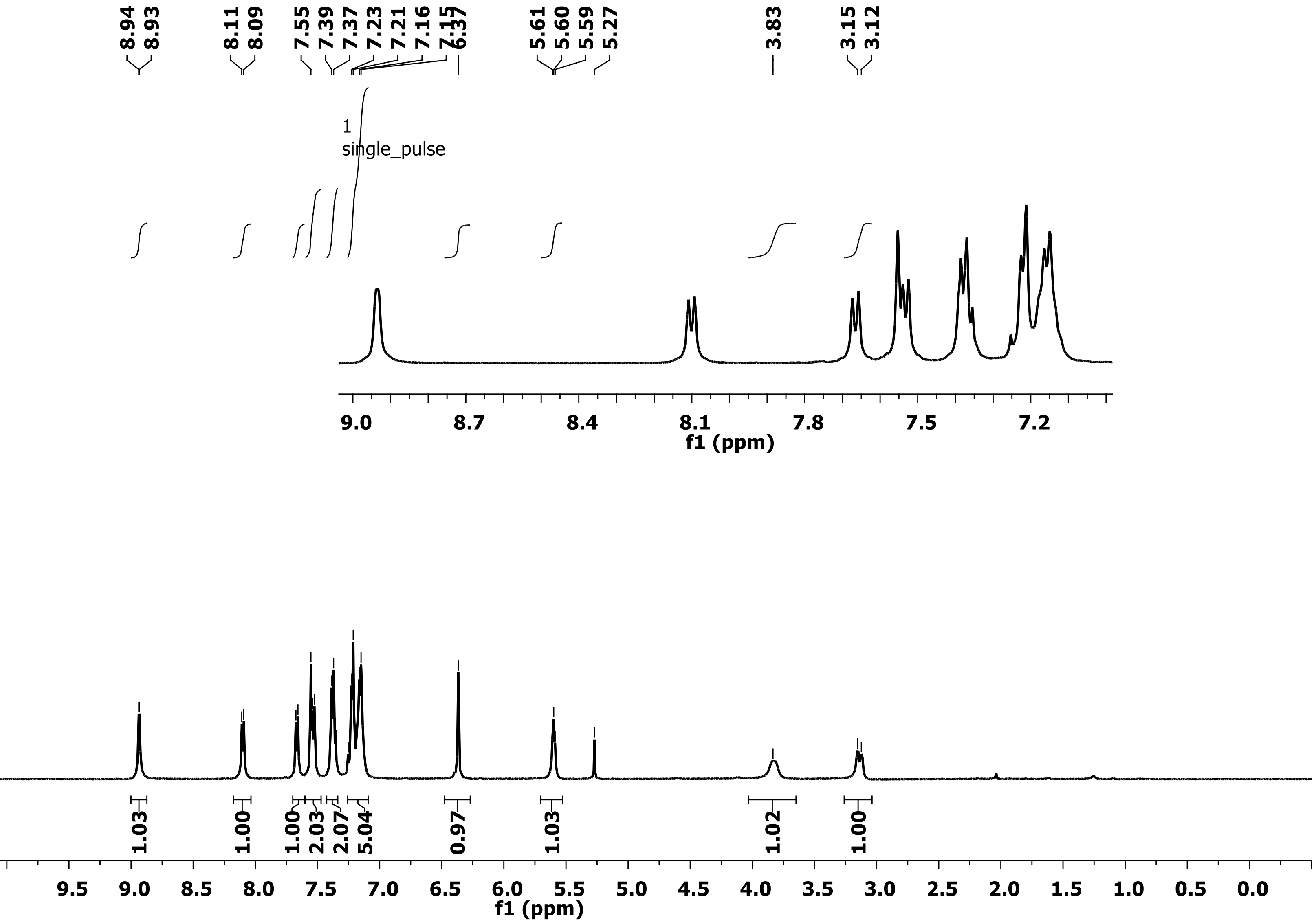


Table 2, Entry 8



1
single pulse decoupled gated NOE

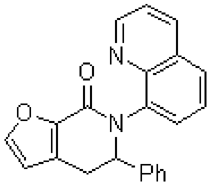
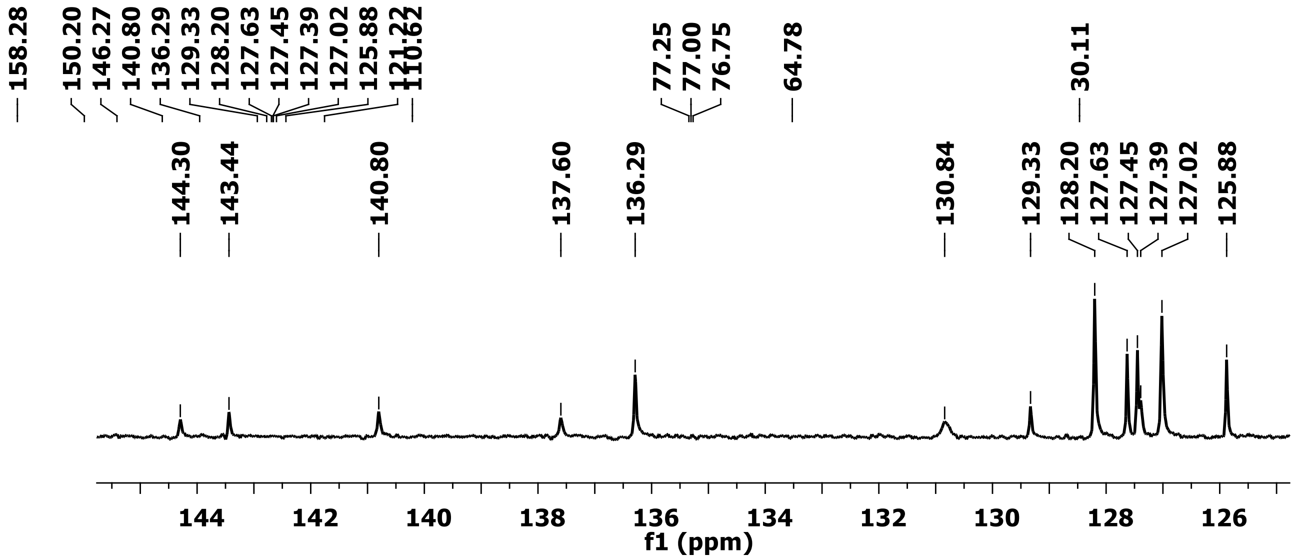
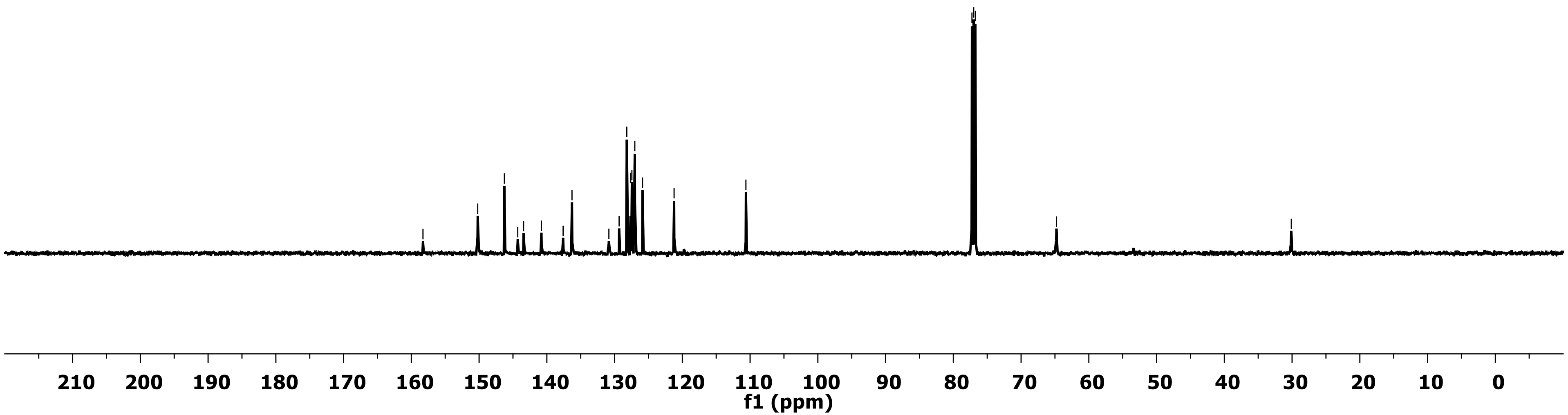


Table 2, Entry 8





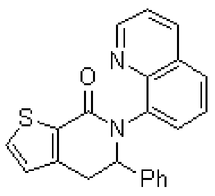
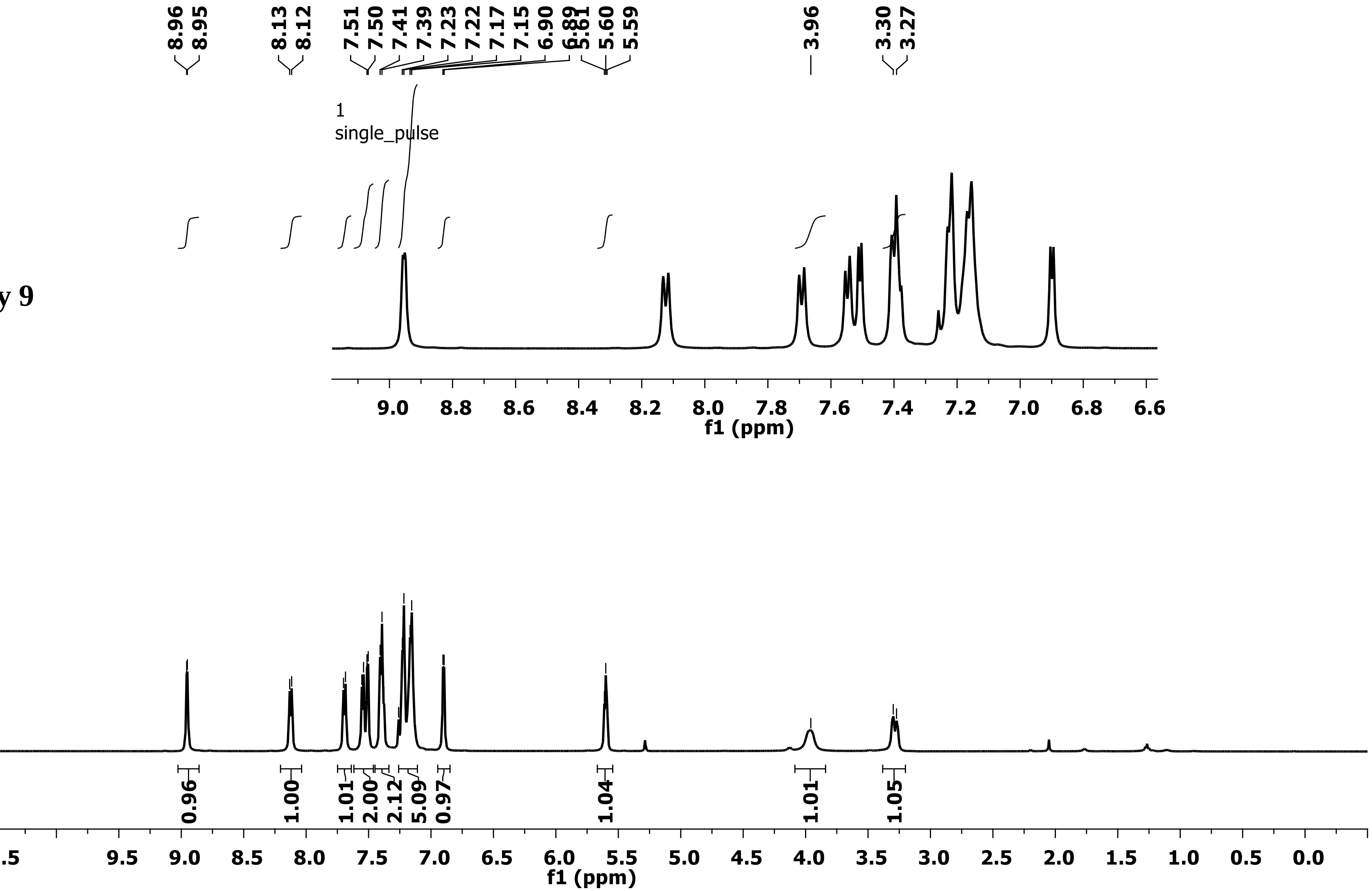


Table 2, Entry 9



1
single pulse decoupled gated NOE

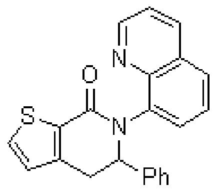
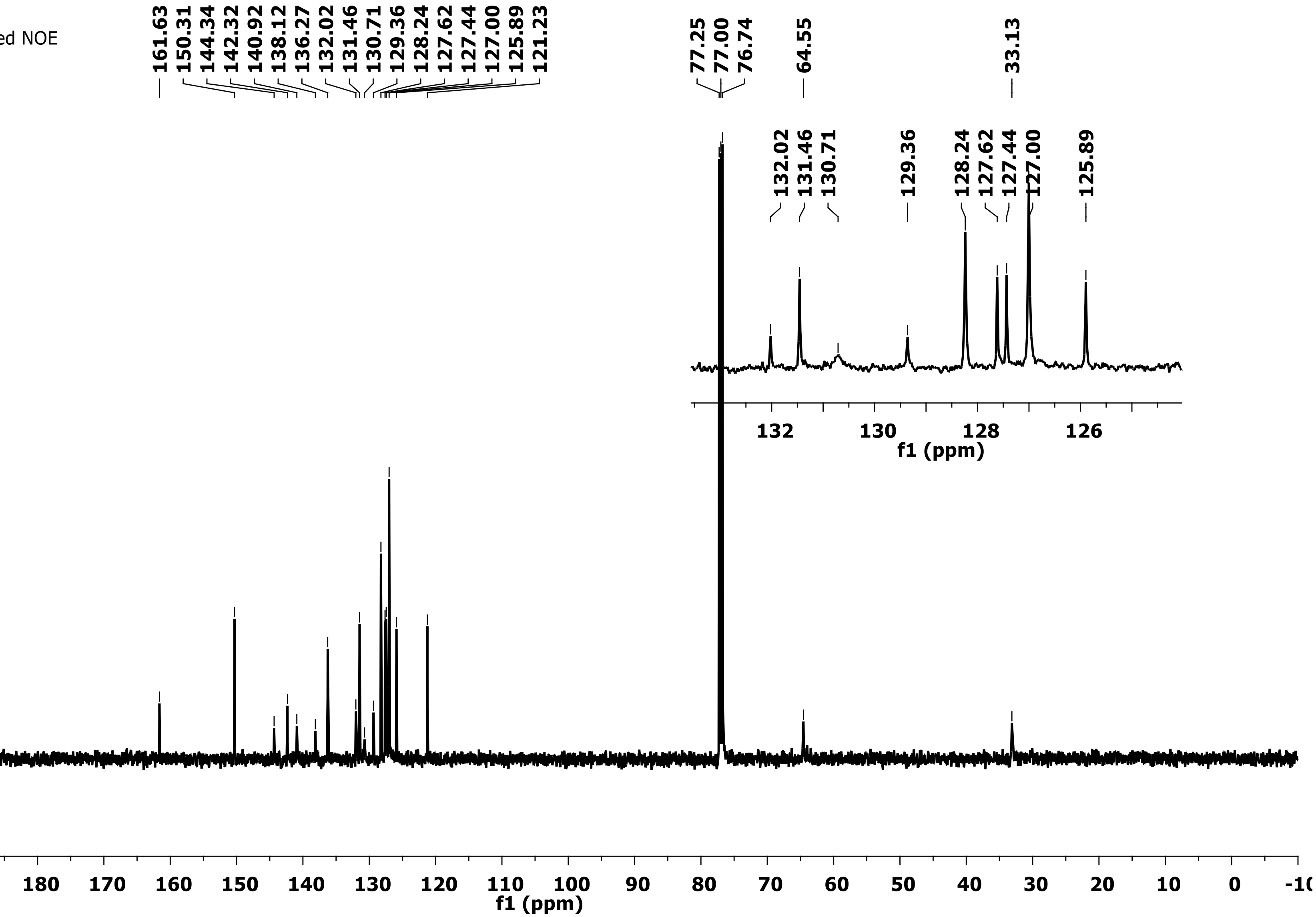
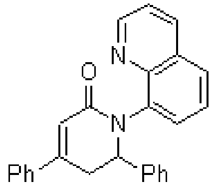


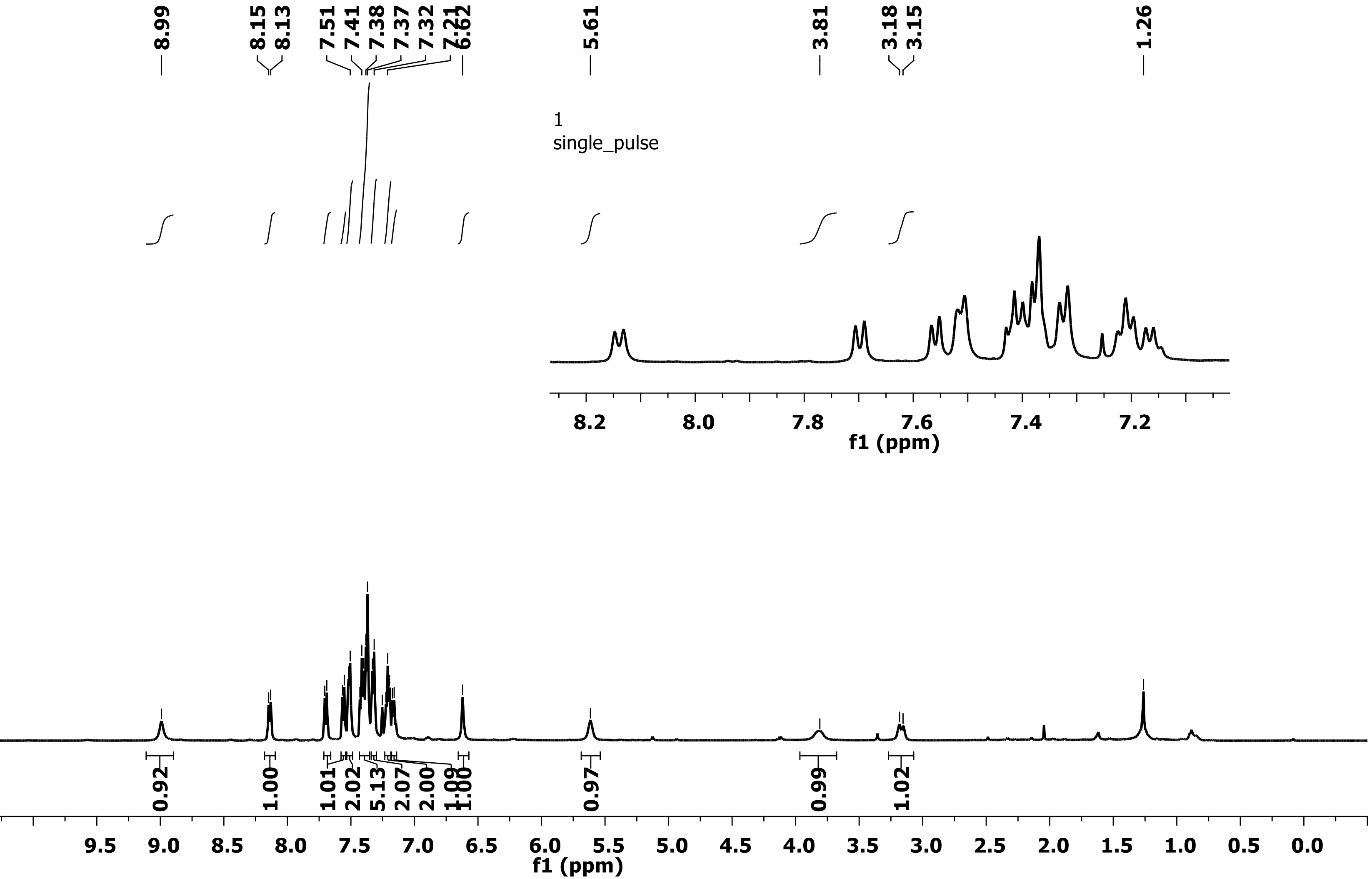
Table 2, Entry 9



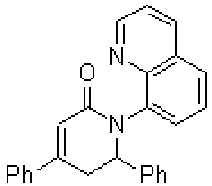


N
O
N
Ph
Ph

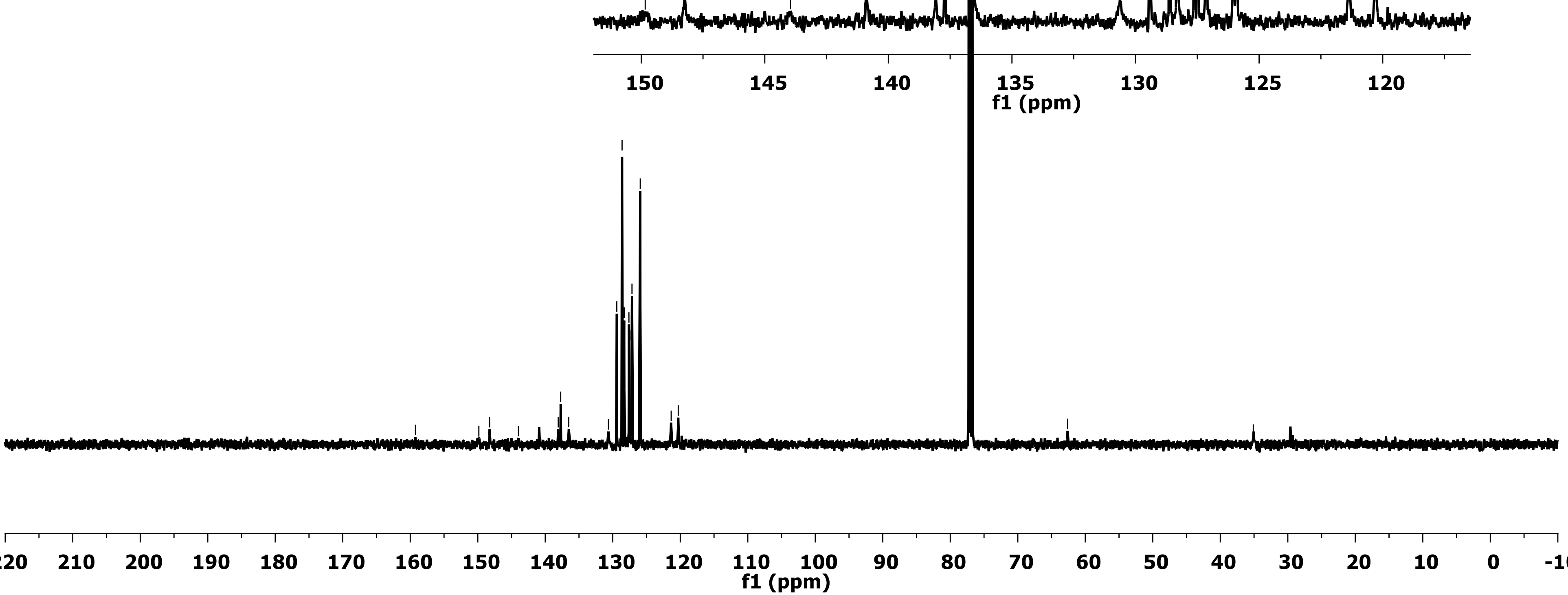
4, Scheme 1



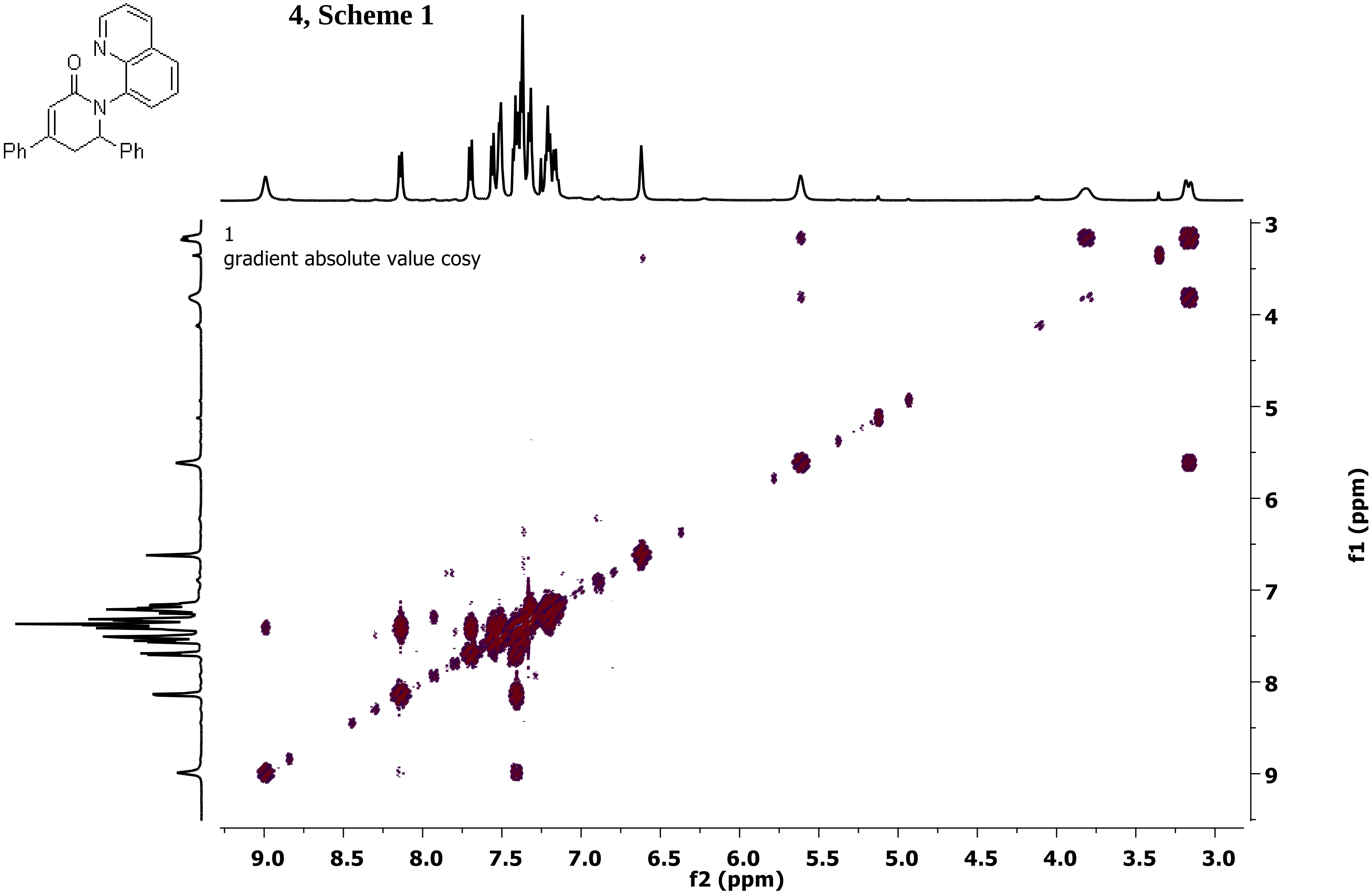
1
single_pulse
8.99
8.15
8.13
7.51
7.41
7.38
7.37
7.32
7.21
6.62
5.61
3.81
3.18
3.15
1.26
0.92
1.00
1.01
2.02
5.13
2.07
2.00
1.00
0.97
0.99
1.02
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
1
single_pulse
8.2 8.0 7.8 7.6 7.4 7.2
f1 (ppm)

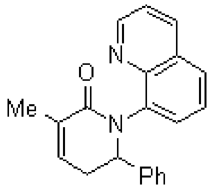


4, Scheme 1

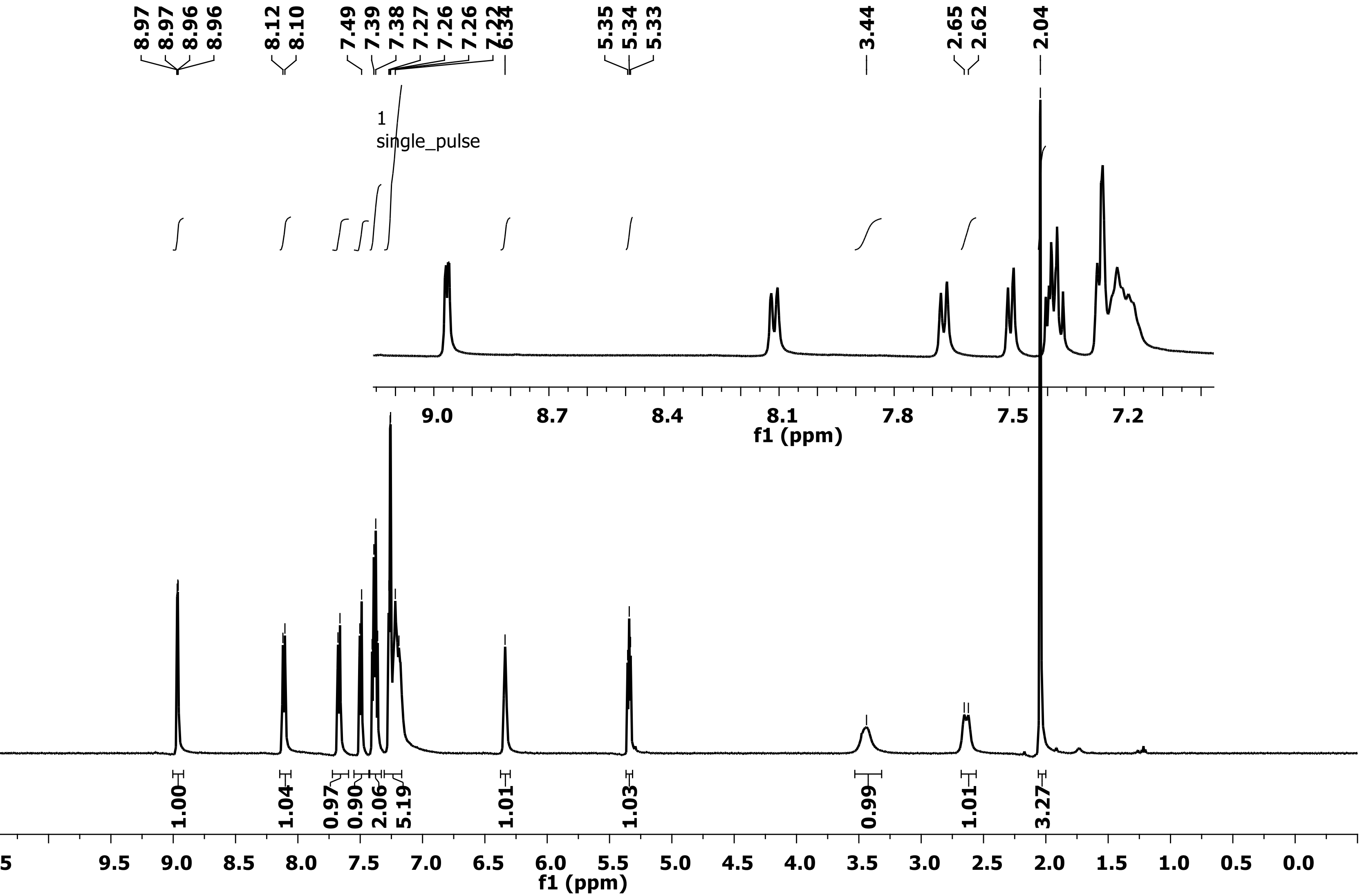

1
single pulse decoupled gated NOE
159.23
149.84
148.25
143.97
140.96
138.08
137.71
136.51
130.64
129.44
129.40
128.62
128.31
127.61
127.48
127.15
126.04
125.93
121.34
120.30
62.63
35.11
f1 (ppm)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
150 145 140 135 130 125 120

4, Scheme 1


N
O
N
Ph
Ph
1
gradient absolute value cosy
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
f2 (ppm)
3
4
5
6
7
8
9
f1 (ppm)

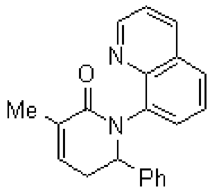


6, Scheme 1

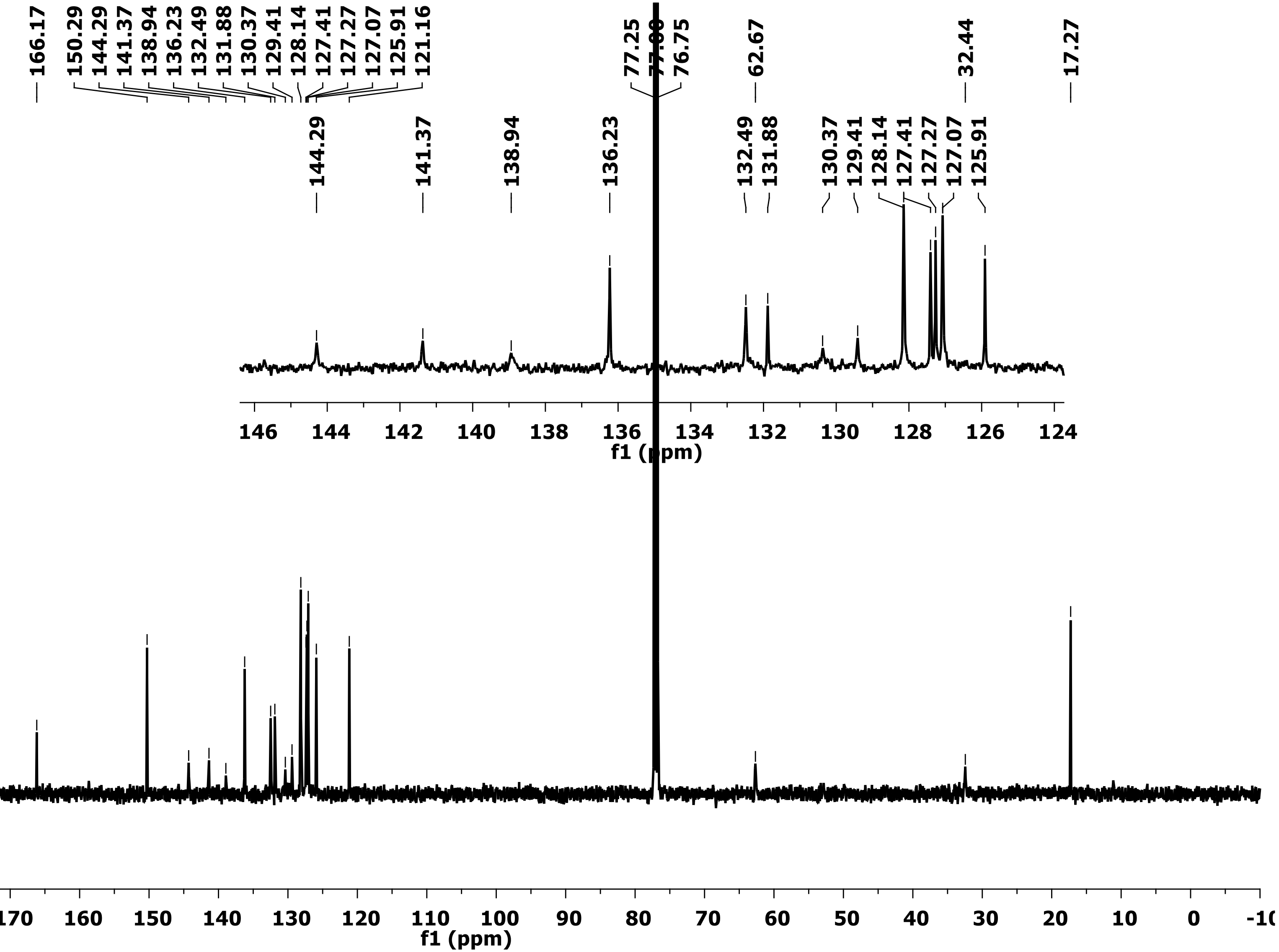


1
single_pulse
8.97
8.97
8.96
8.96
8.12
8.10
7.49
7.39
7.38
7.27
7.26
7.26
7.22
6.34
5.35
5.34
5.33
3.44
2.65
2.62
2.04
1.00
1.04
0.97
0.90
2.06
5.19
1.01
1.03
0.99
1.01
3.27
f1 (ppm)

1
single pulse decoupled gated NOE



6, Scheme 1



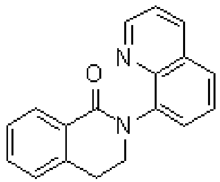
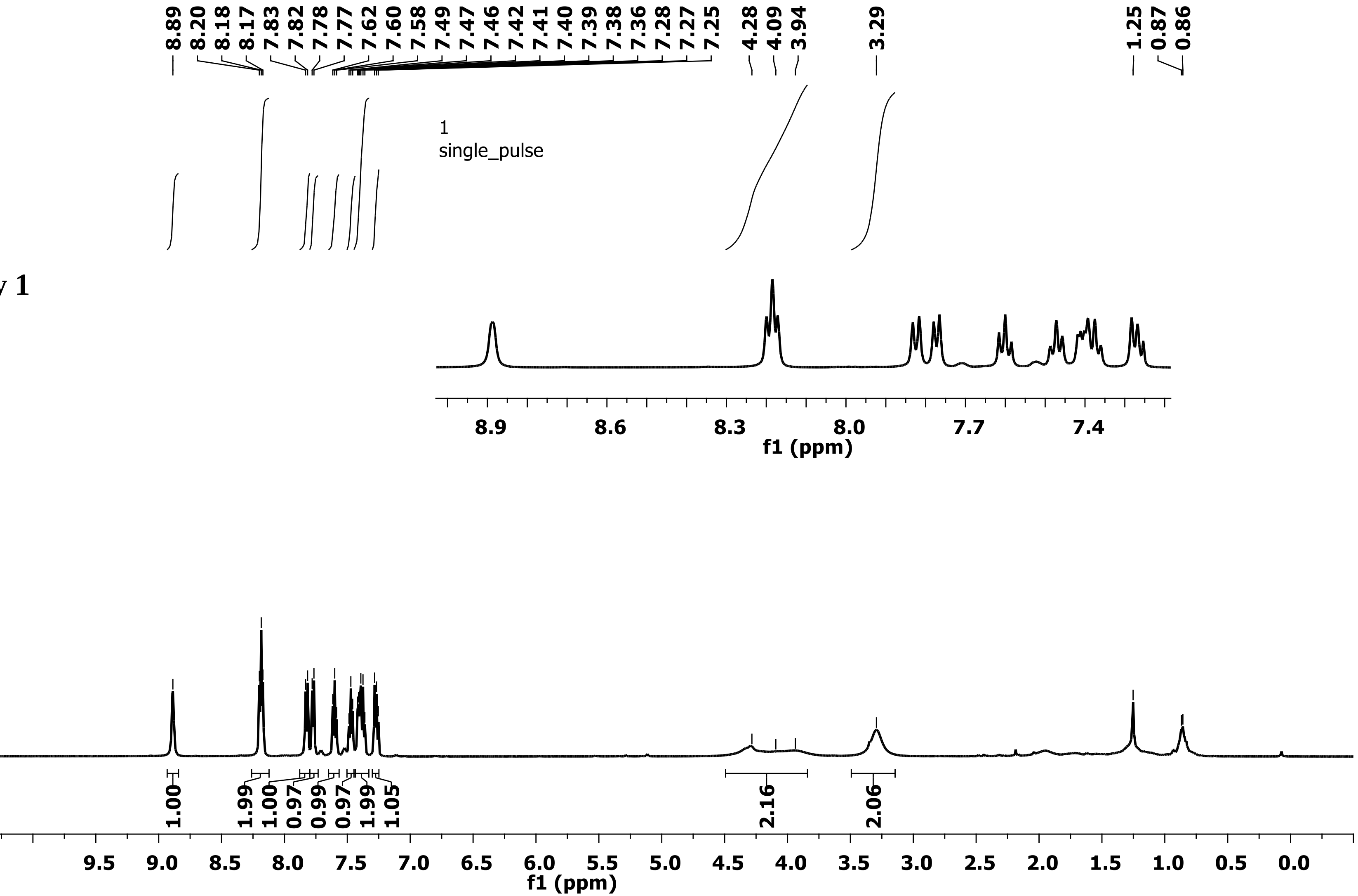


Table 3, Entry 1



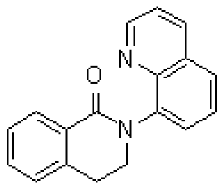
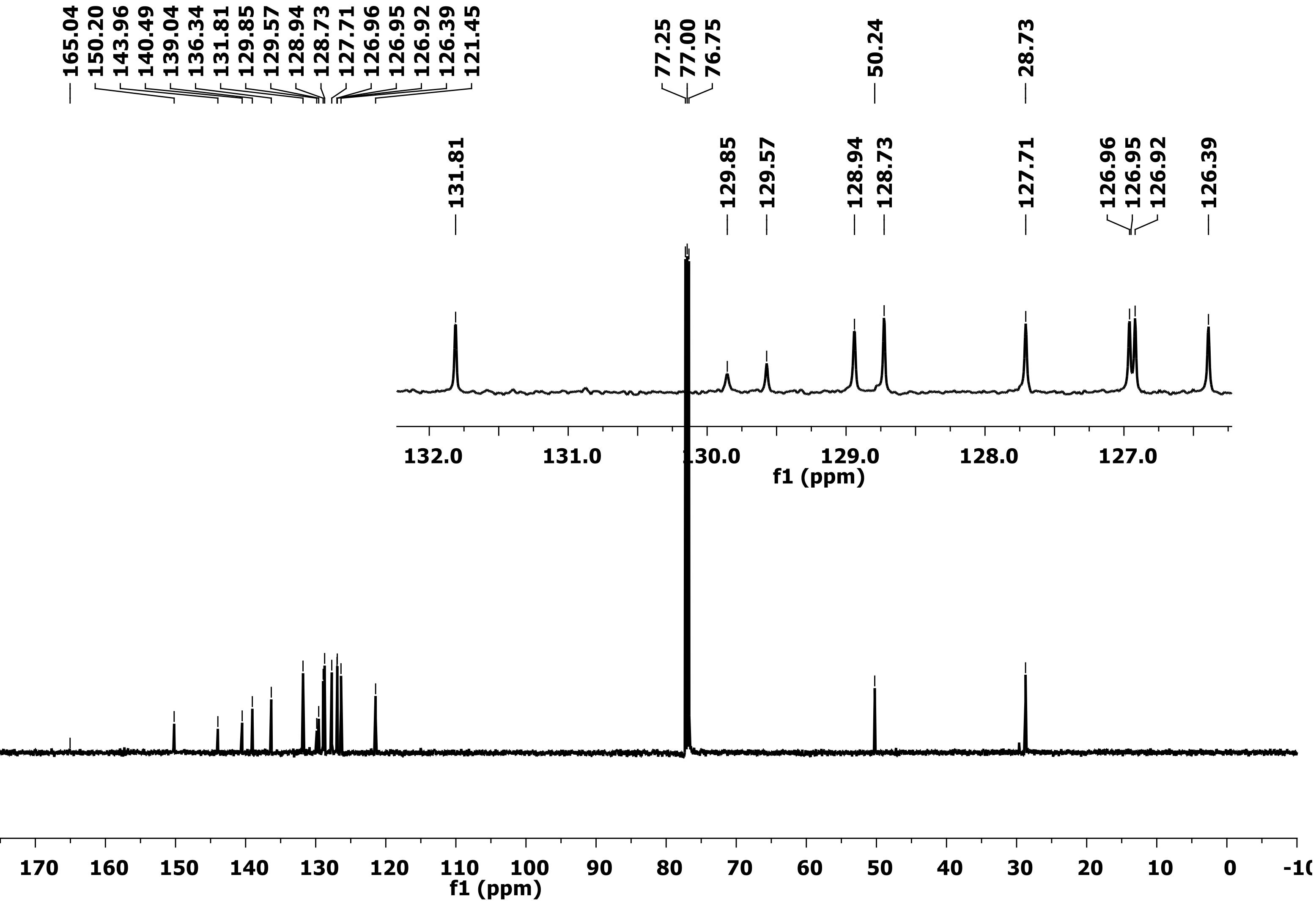


Table 3, Entry 1



1
single_pulse

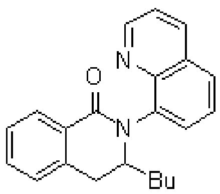
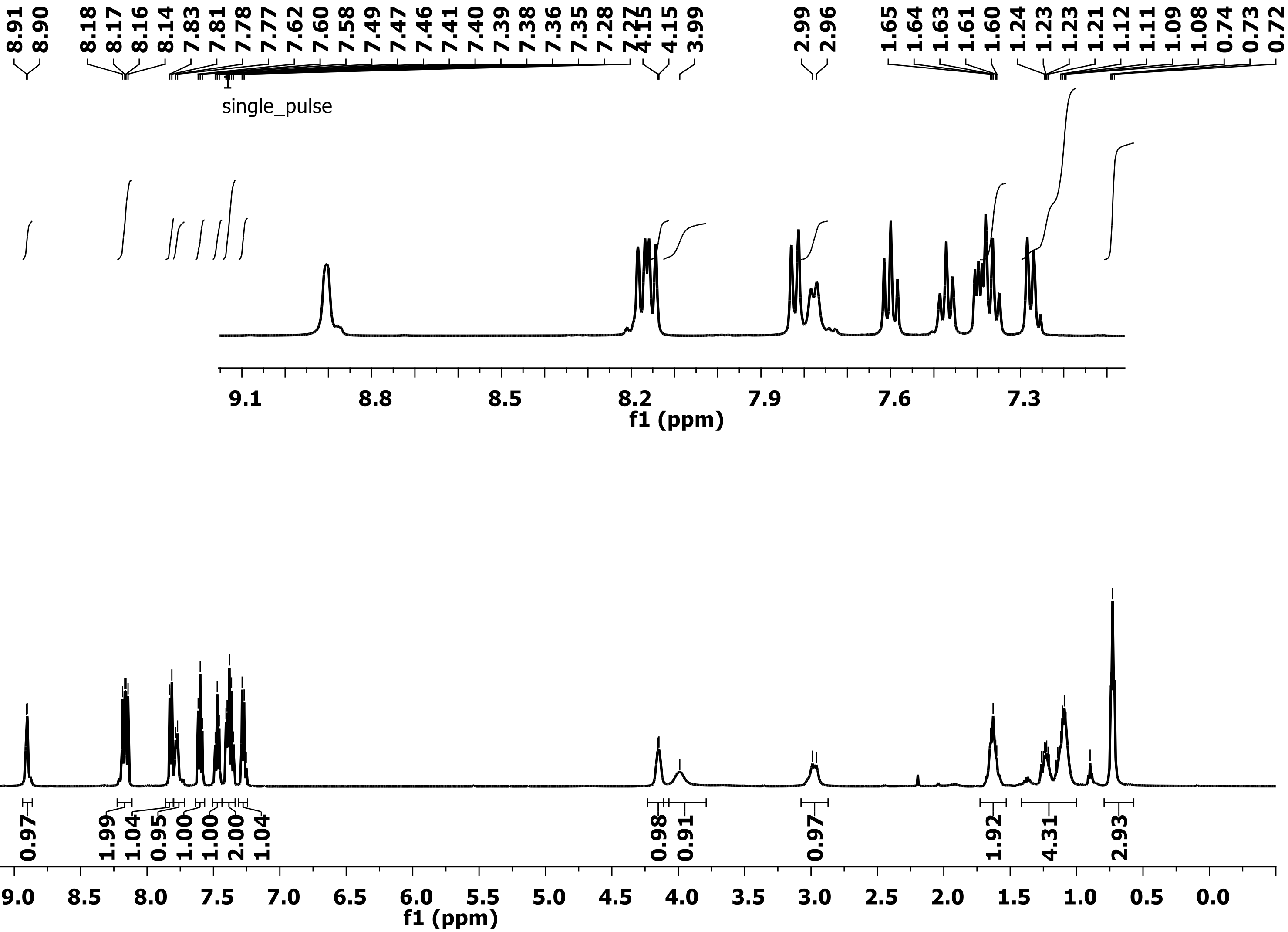


Table 3, Entry 2



1
single pulse decoupled gated NOE

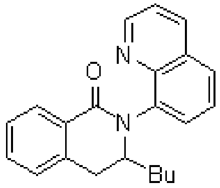
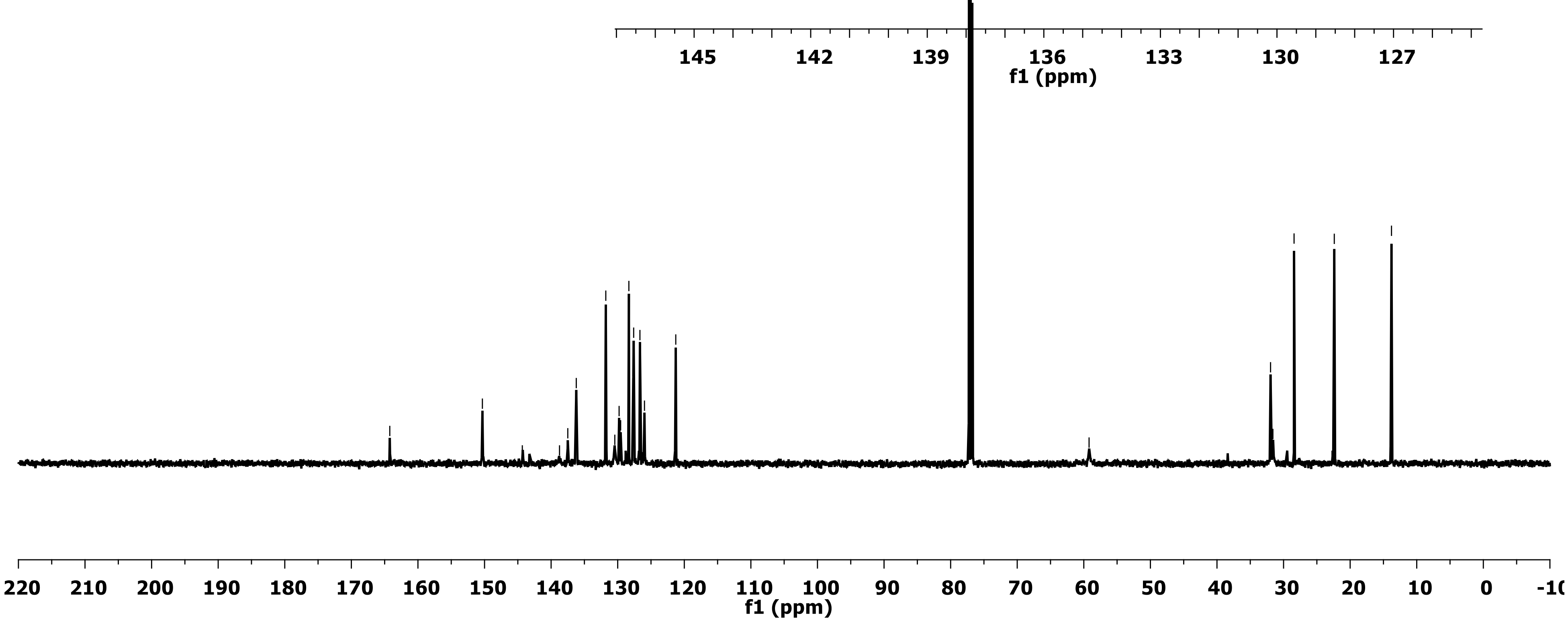
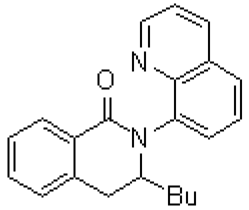


Table 3, Entry 2





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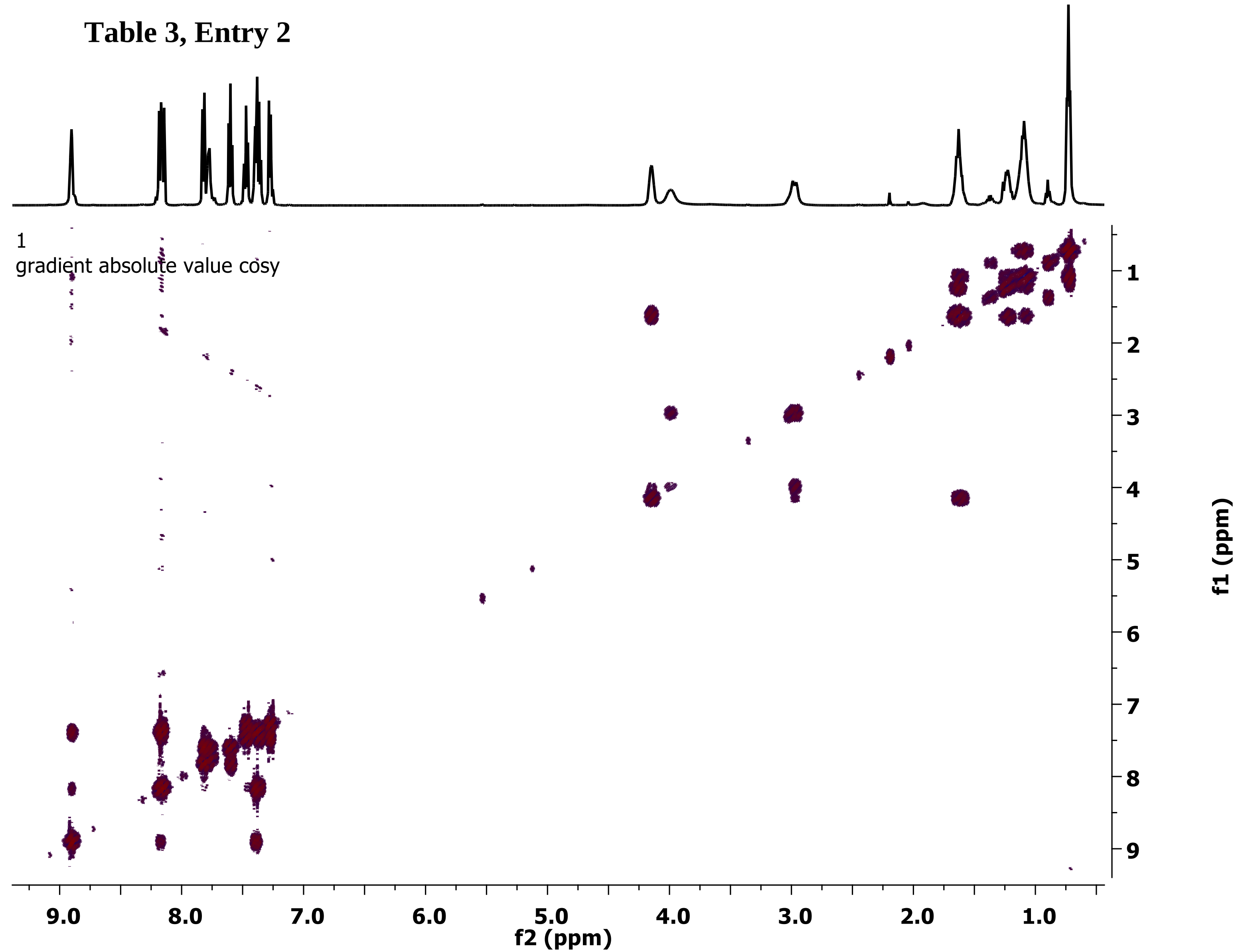


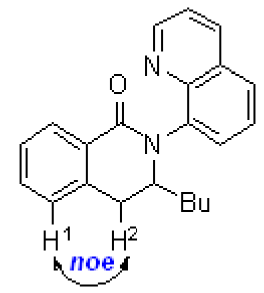
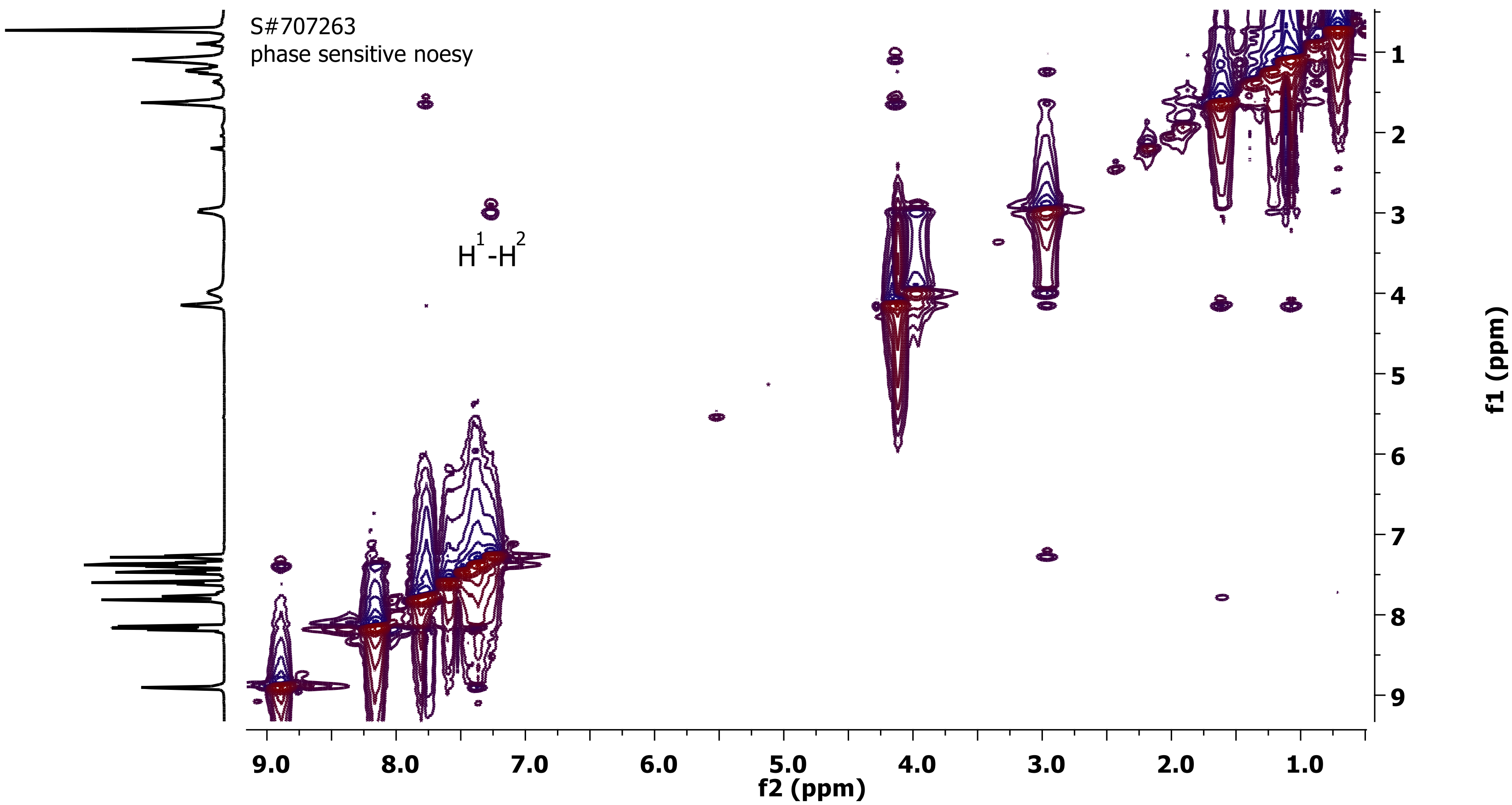


Table 3, Entry 2



1
single_pulse

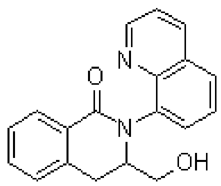
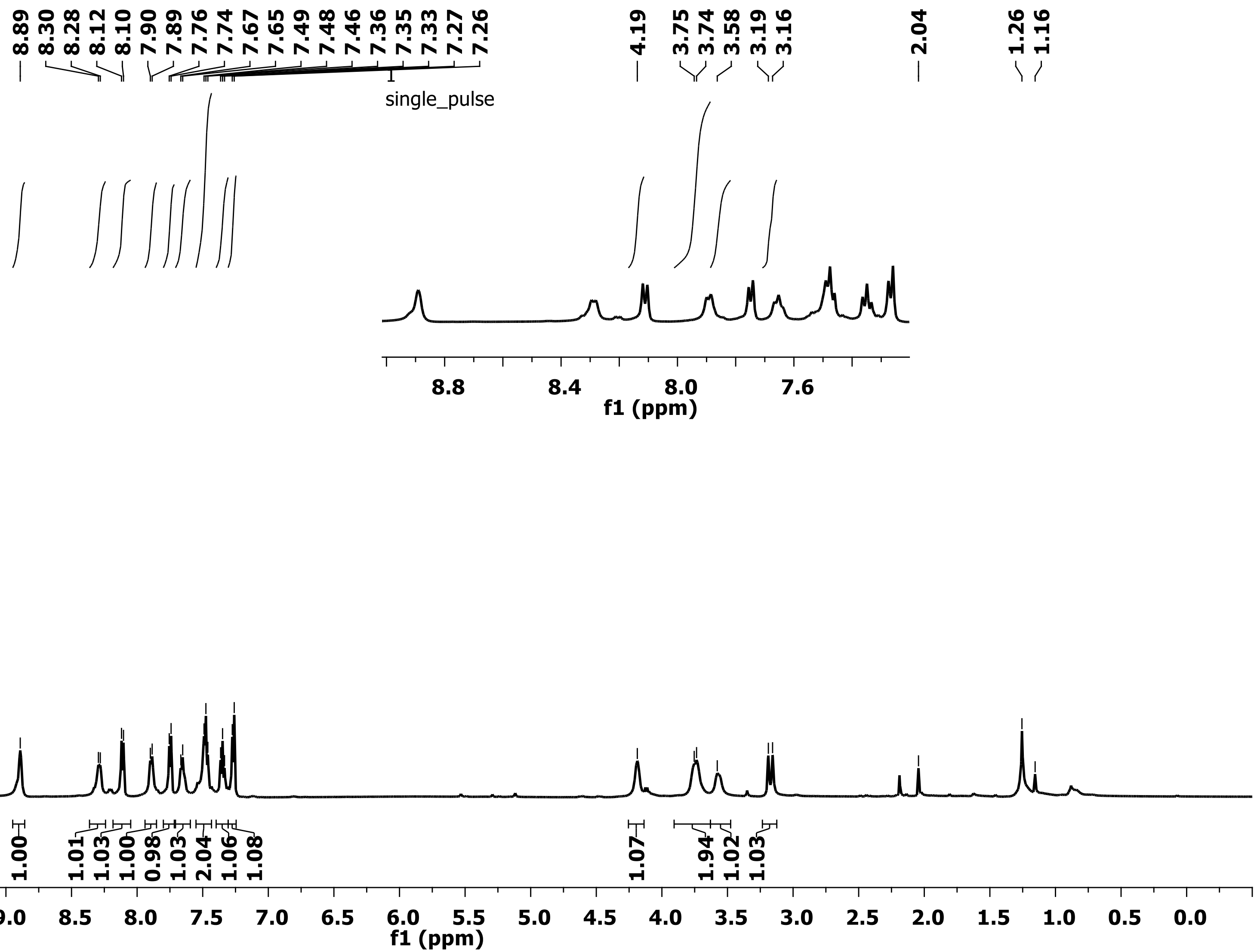
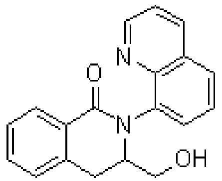


Table 3, Entry 3



1
single pulse decoupled gated NOE



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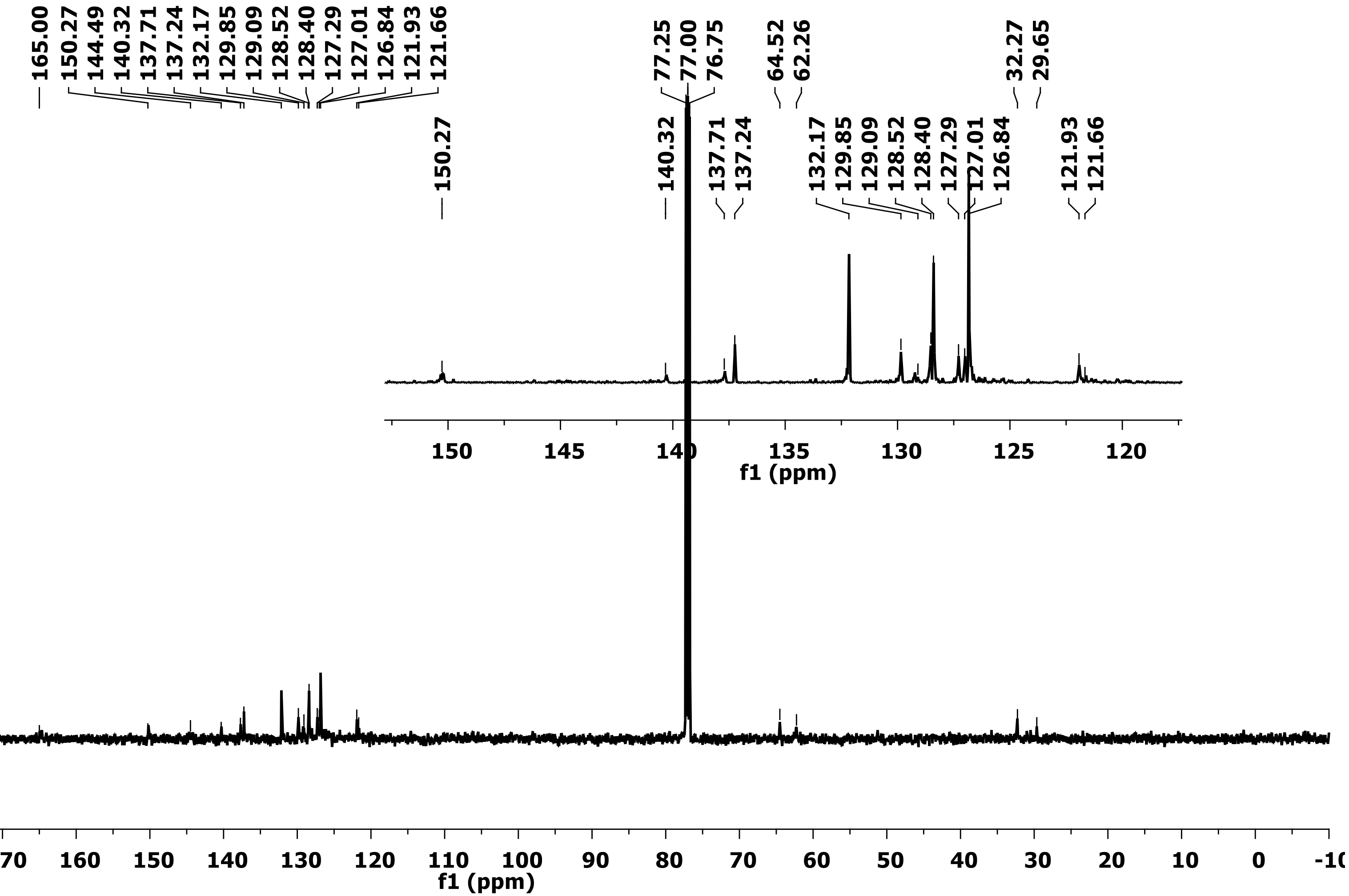

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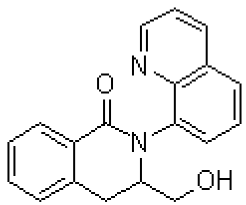
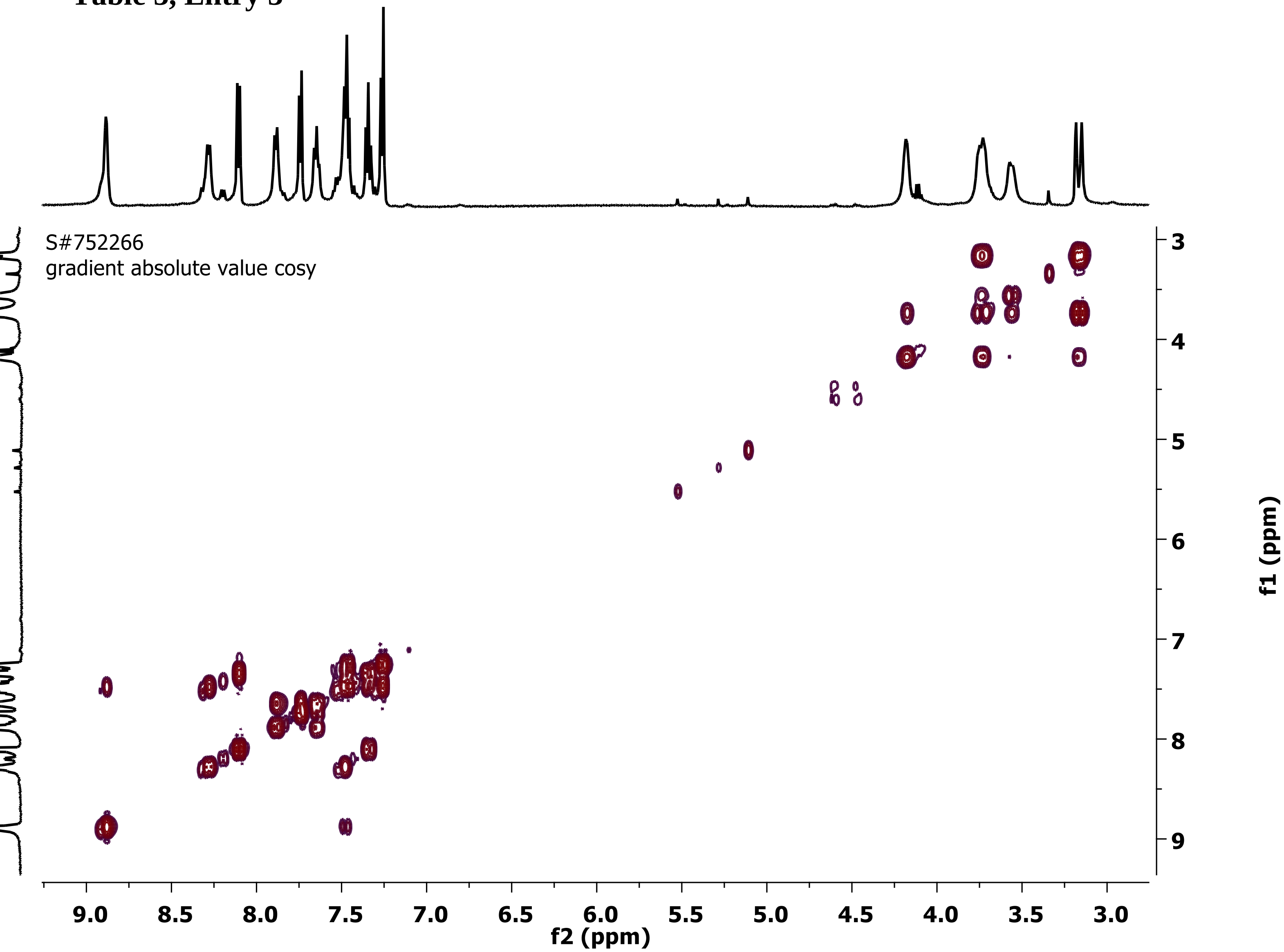




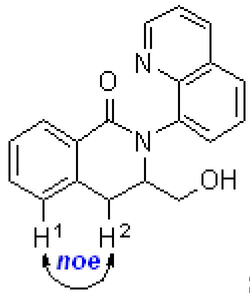


Table 3, Entry 3

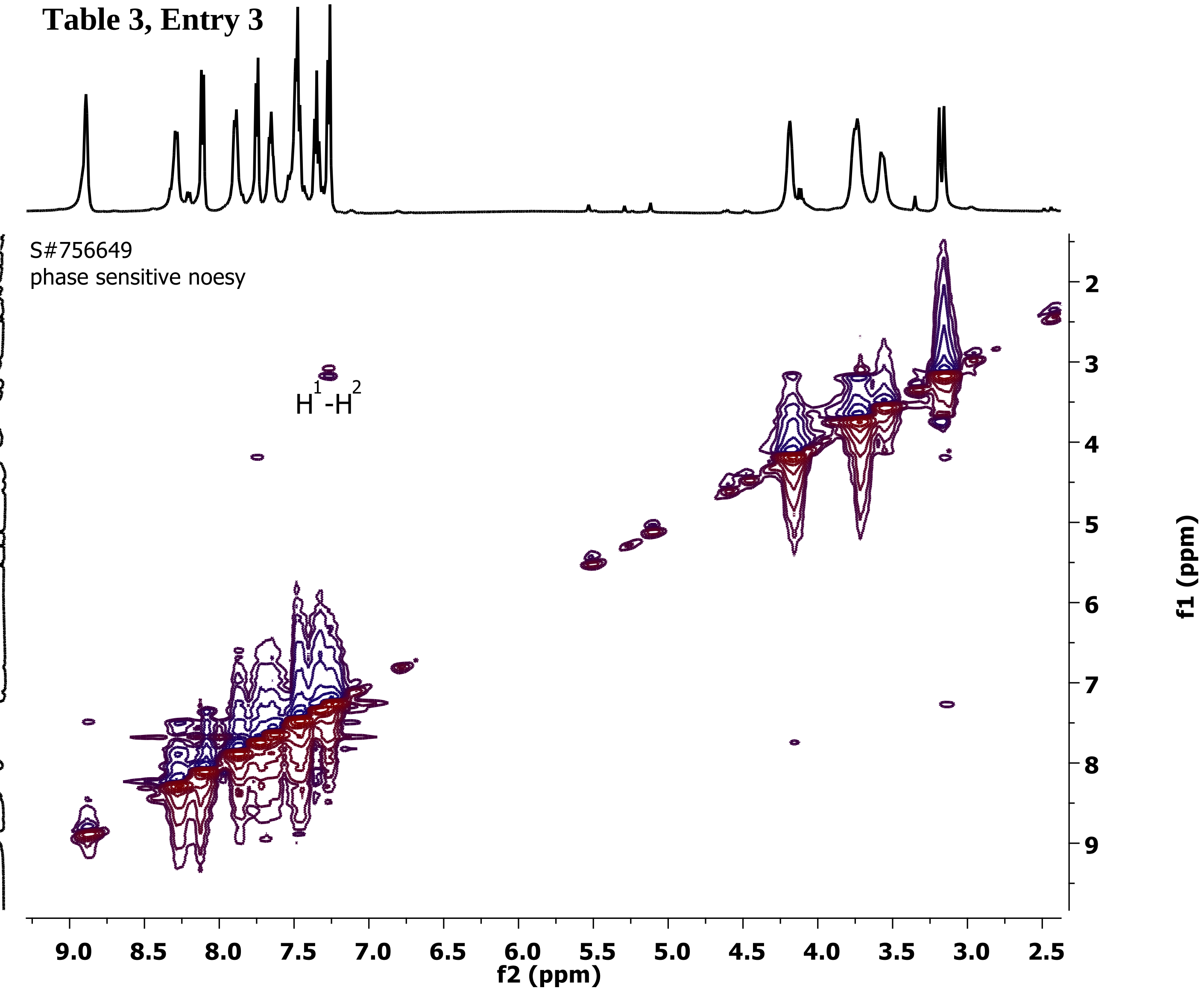
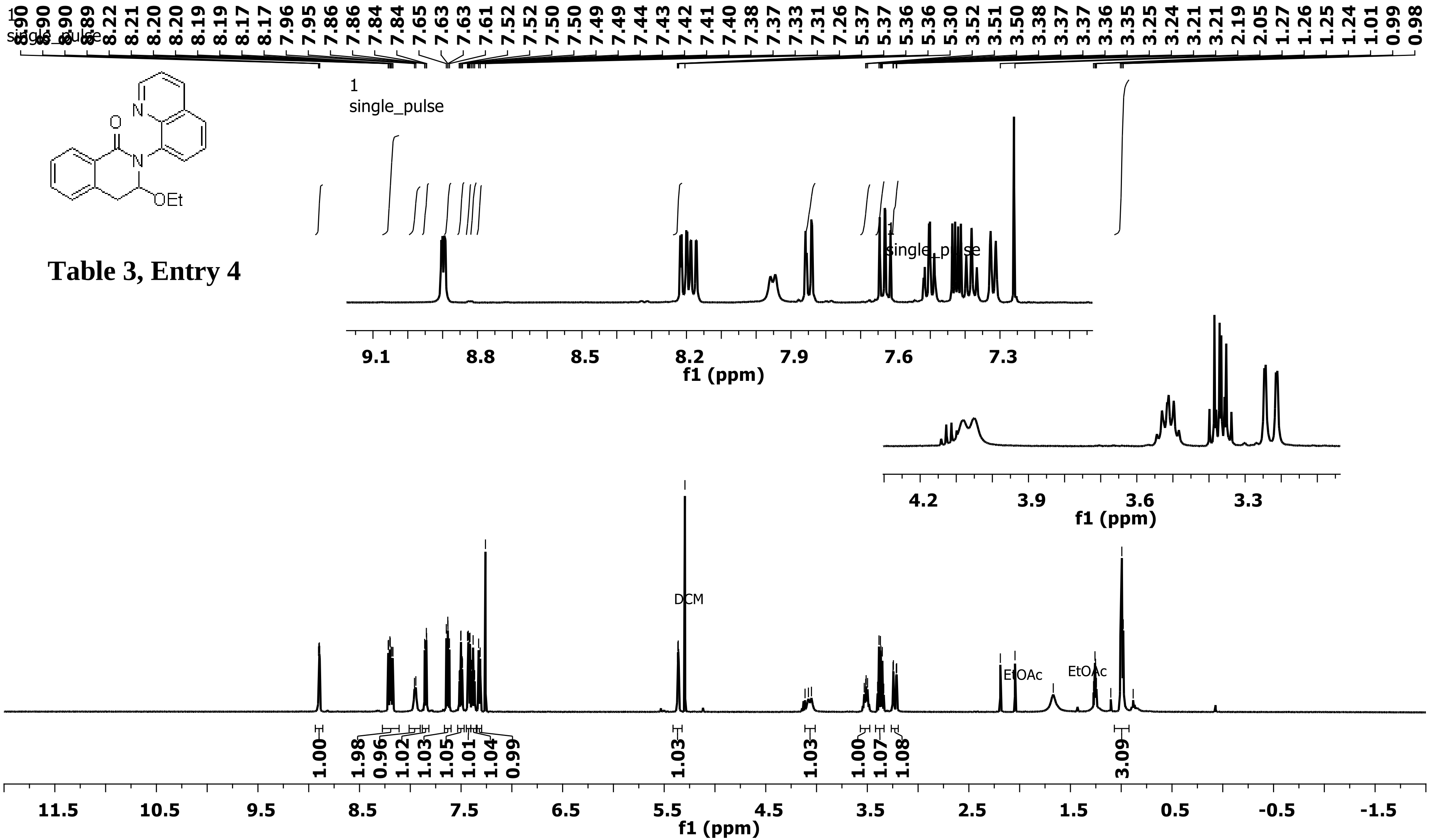


Table 3, Entry 4



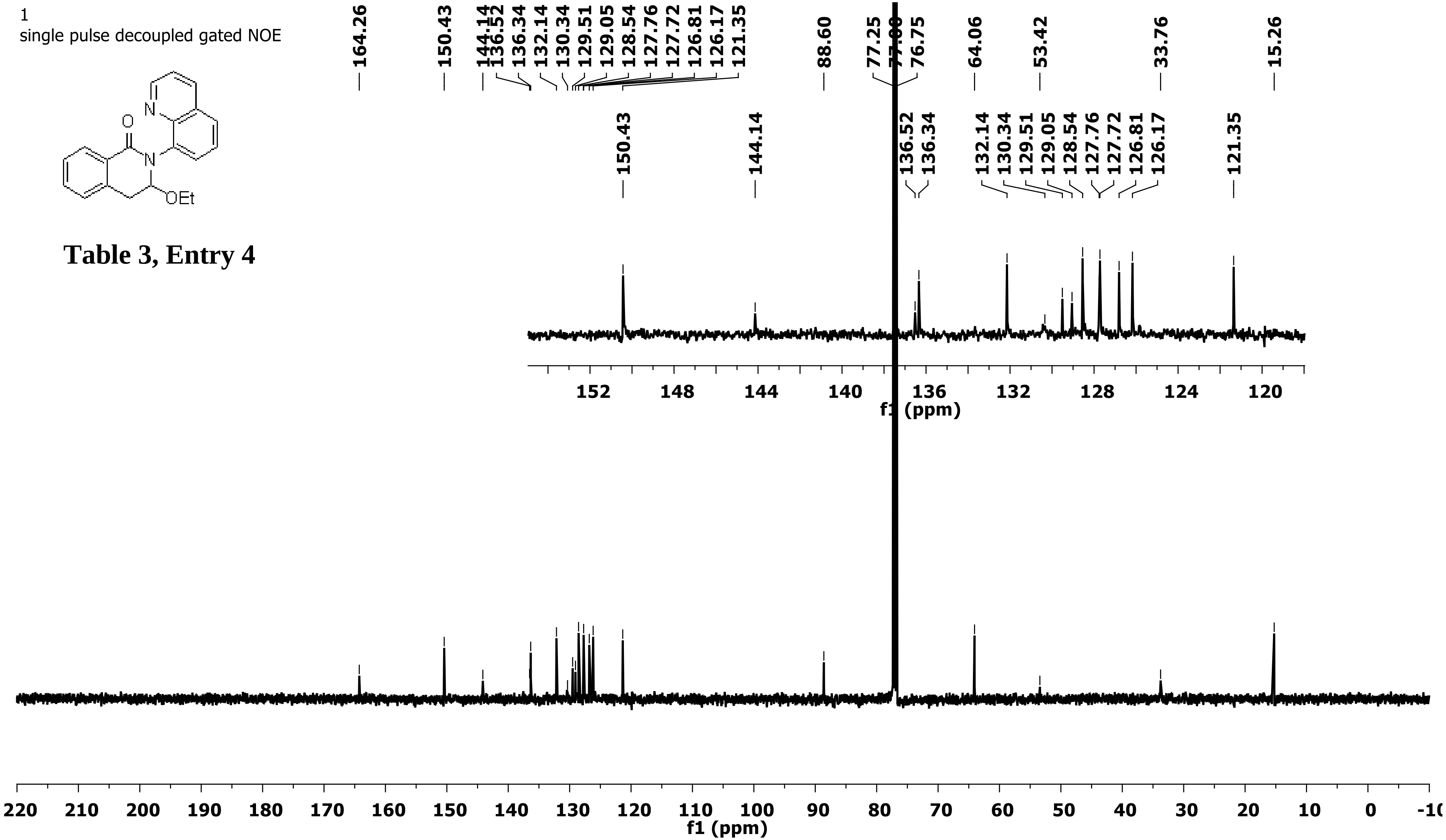
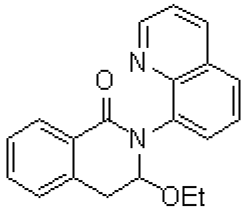


Table 3, Entry 4



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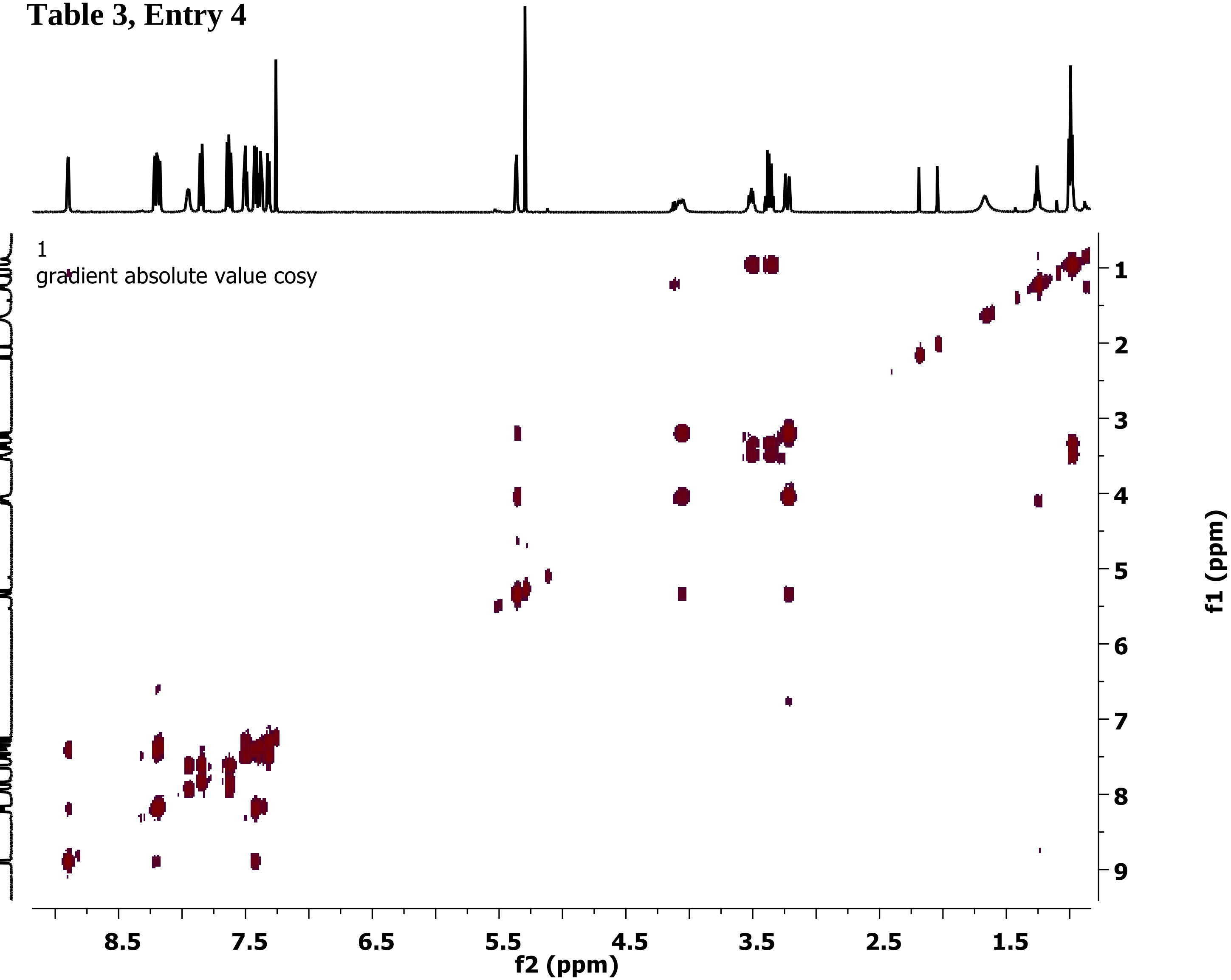


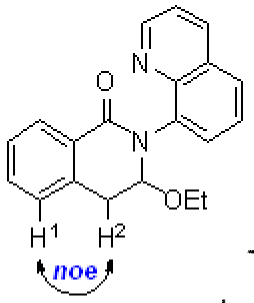
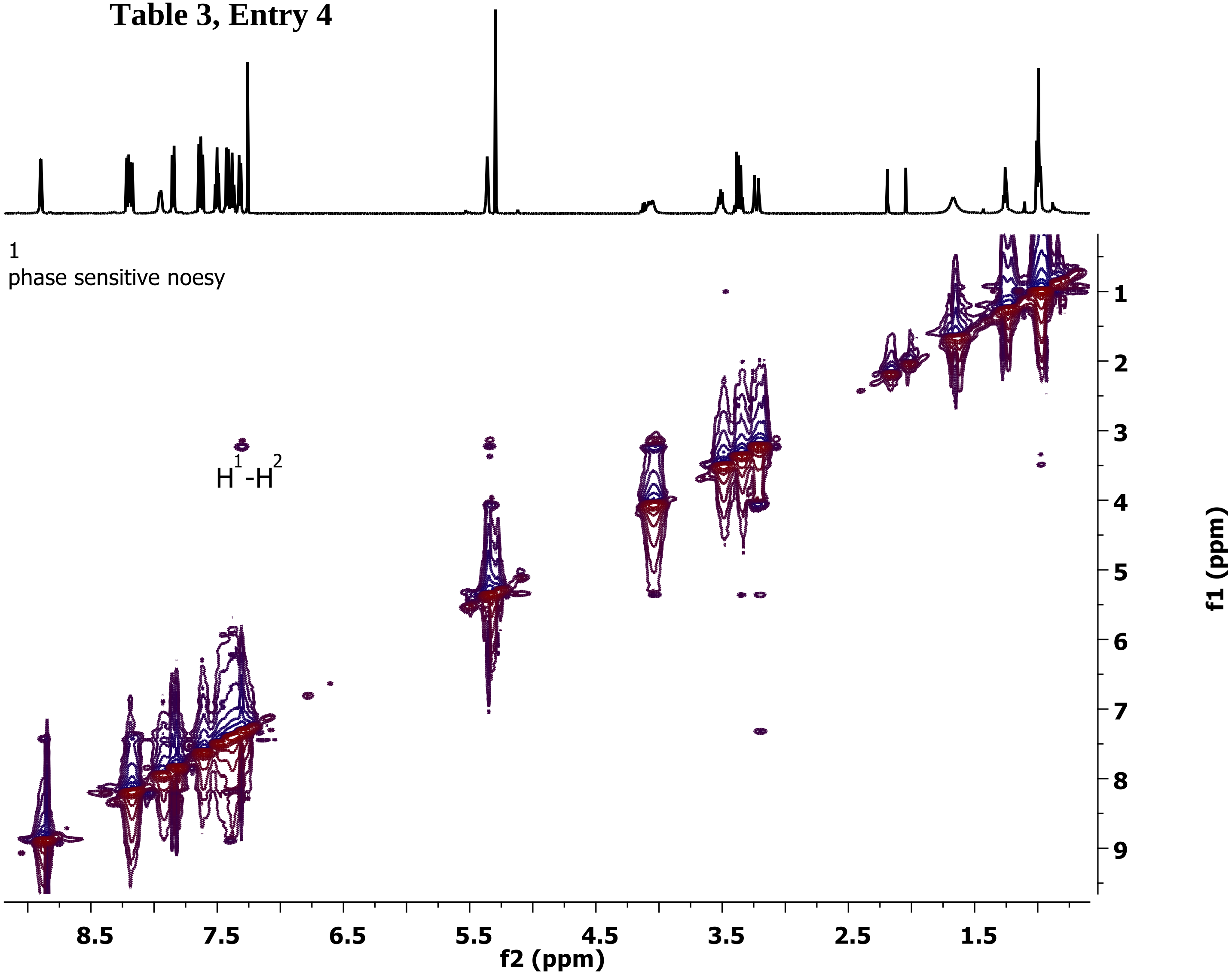


Table 3, Entry 4



1
single_pulse

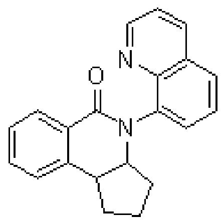
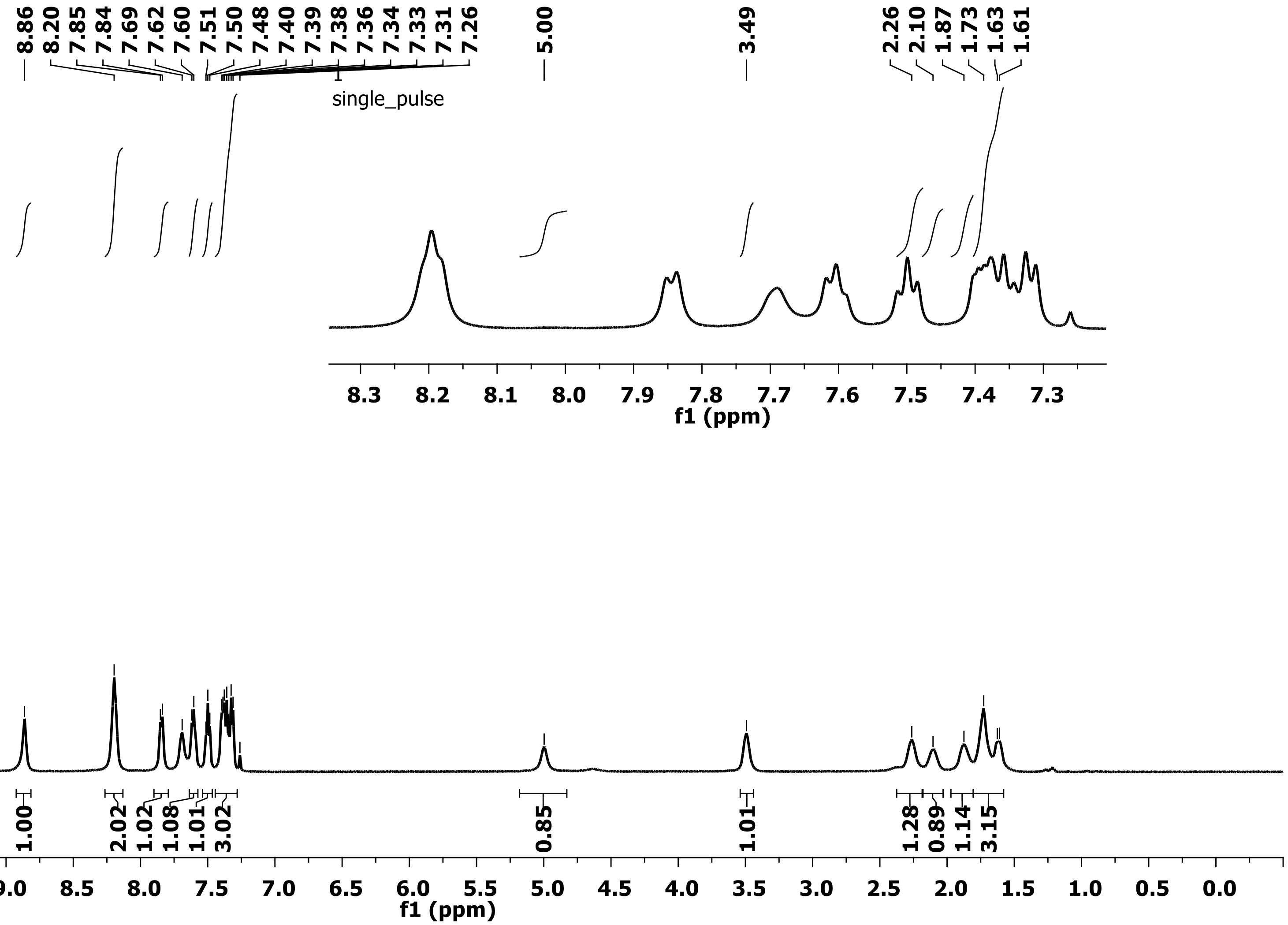


Table 3, Entry 5



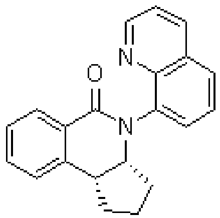
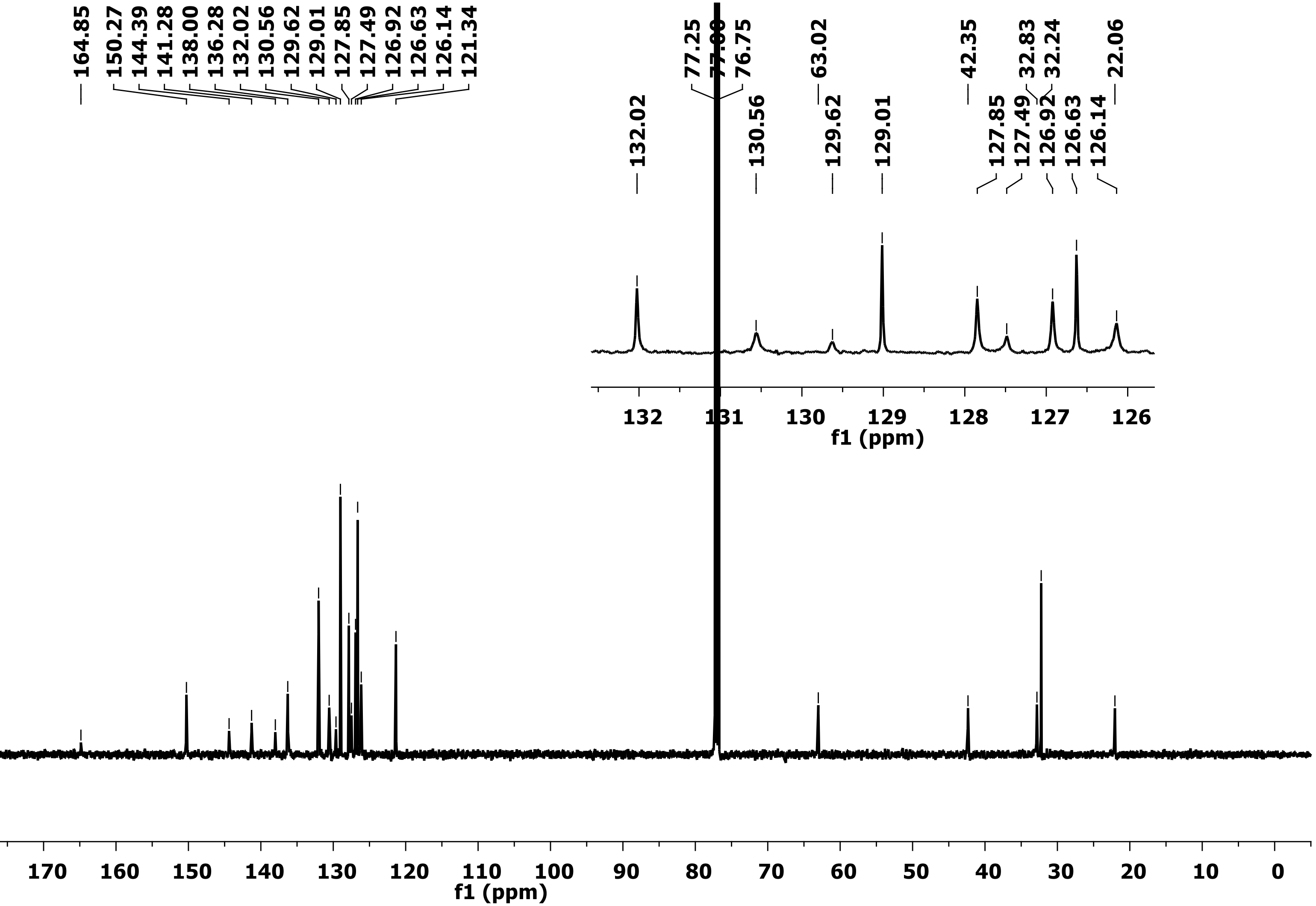


Table 3, Entry 5



1
single_pulse

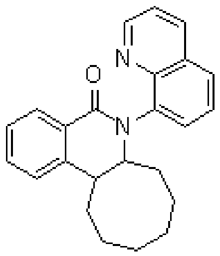
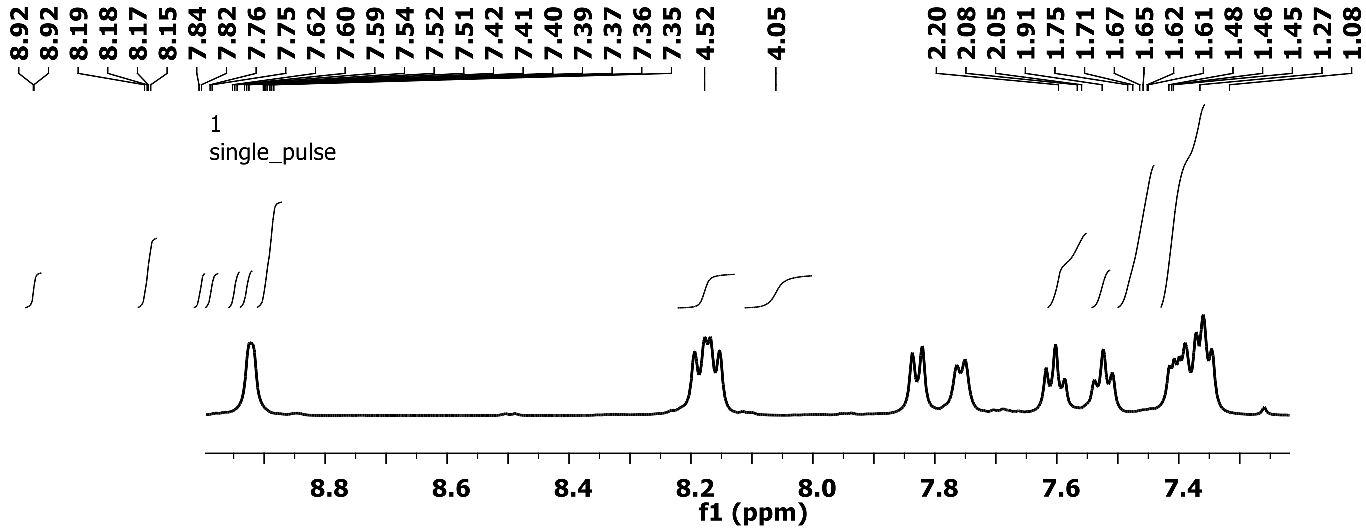
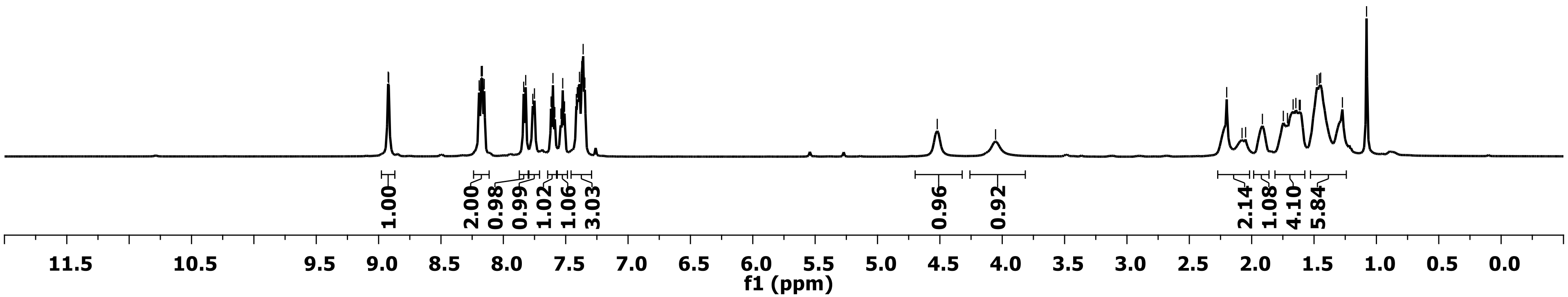


Table 3, Entry 6





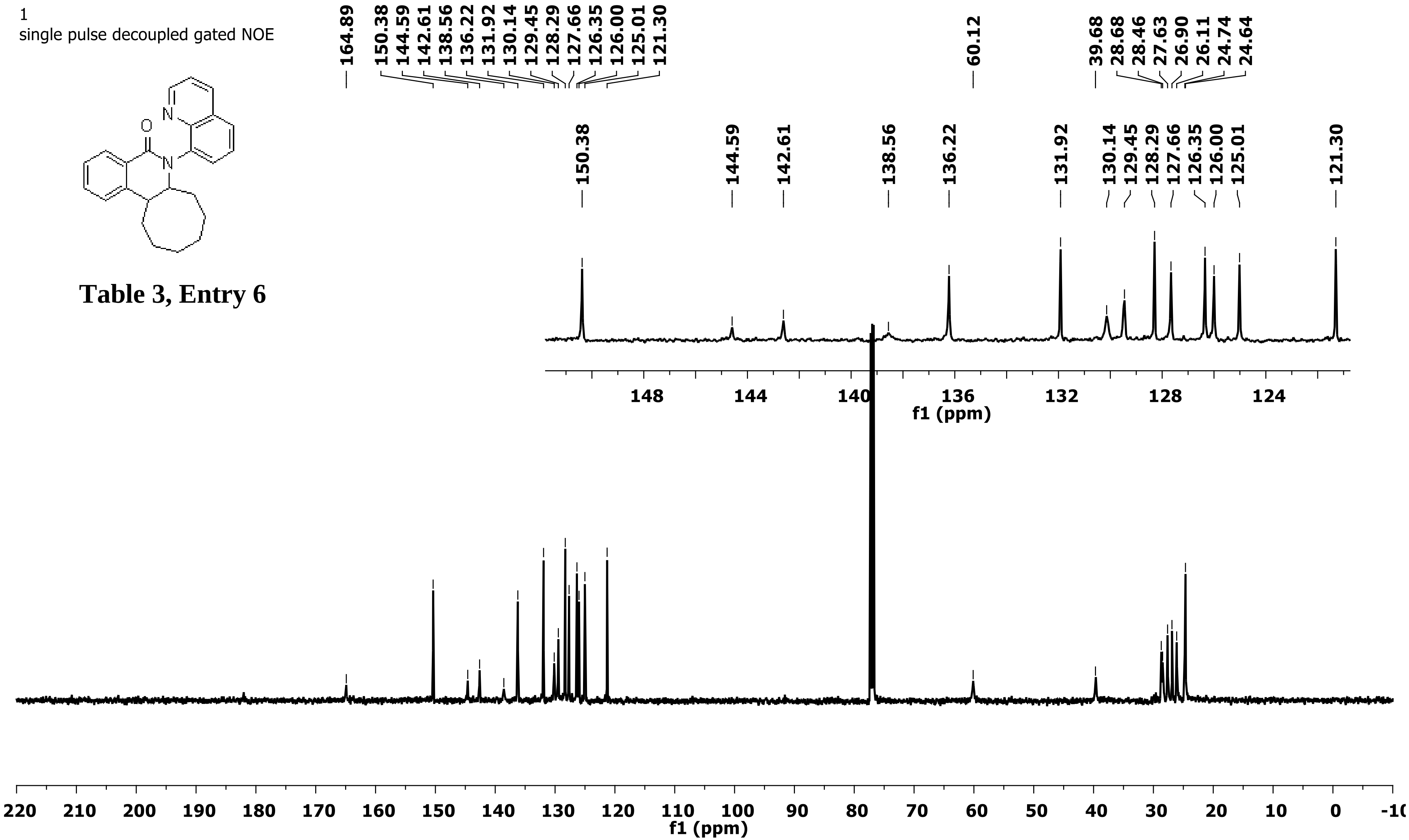


Table 3, Entry 6

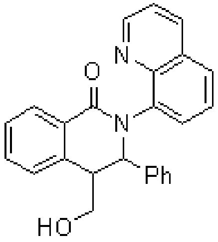
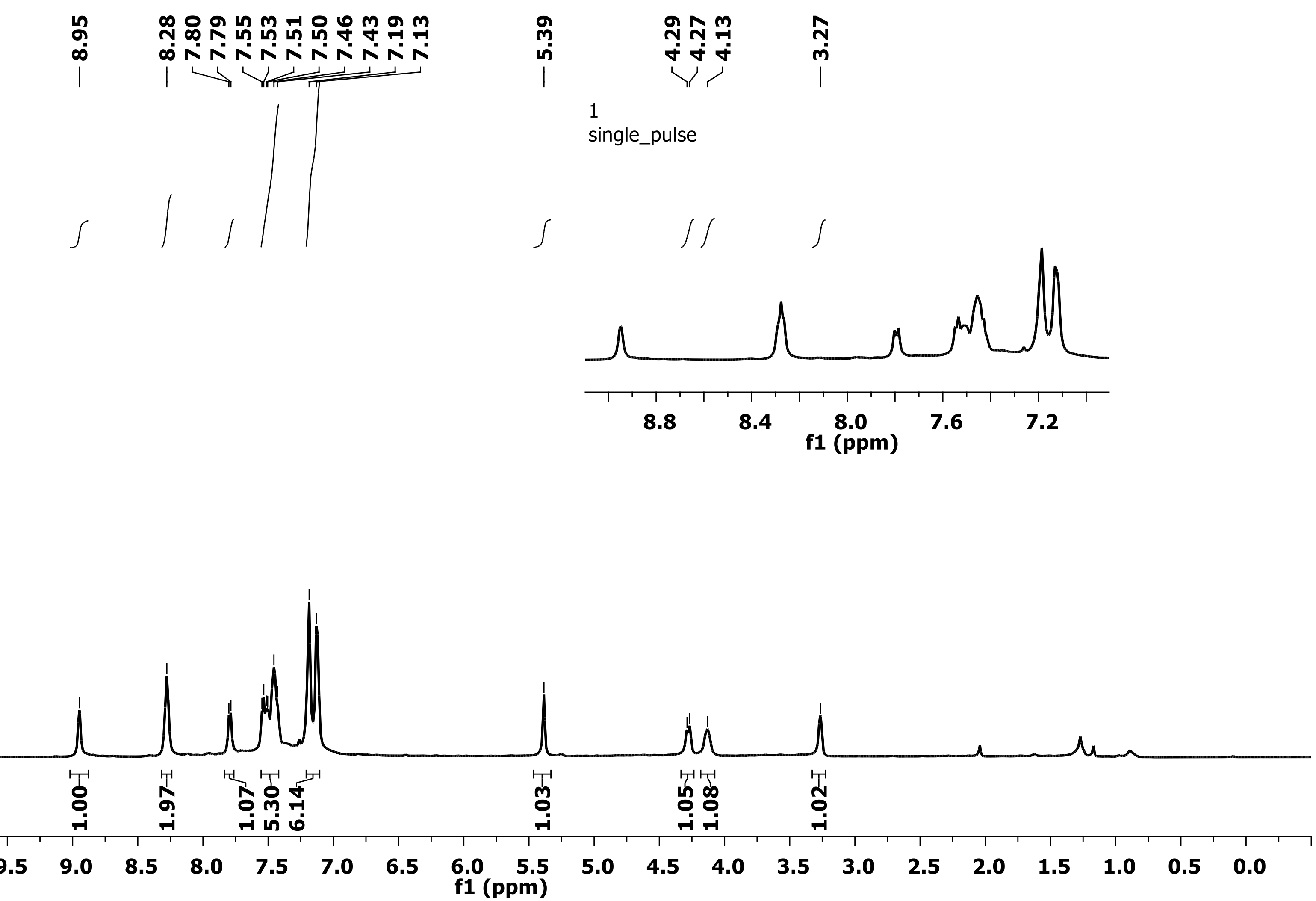


Table 3, Entry 7,8



1
single pulse decoupled gated NOE

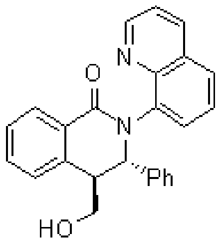


Table 3, Entry 7,8

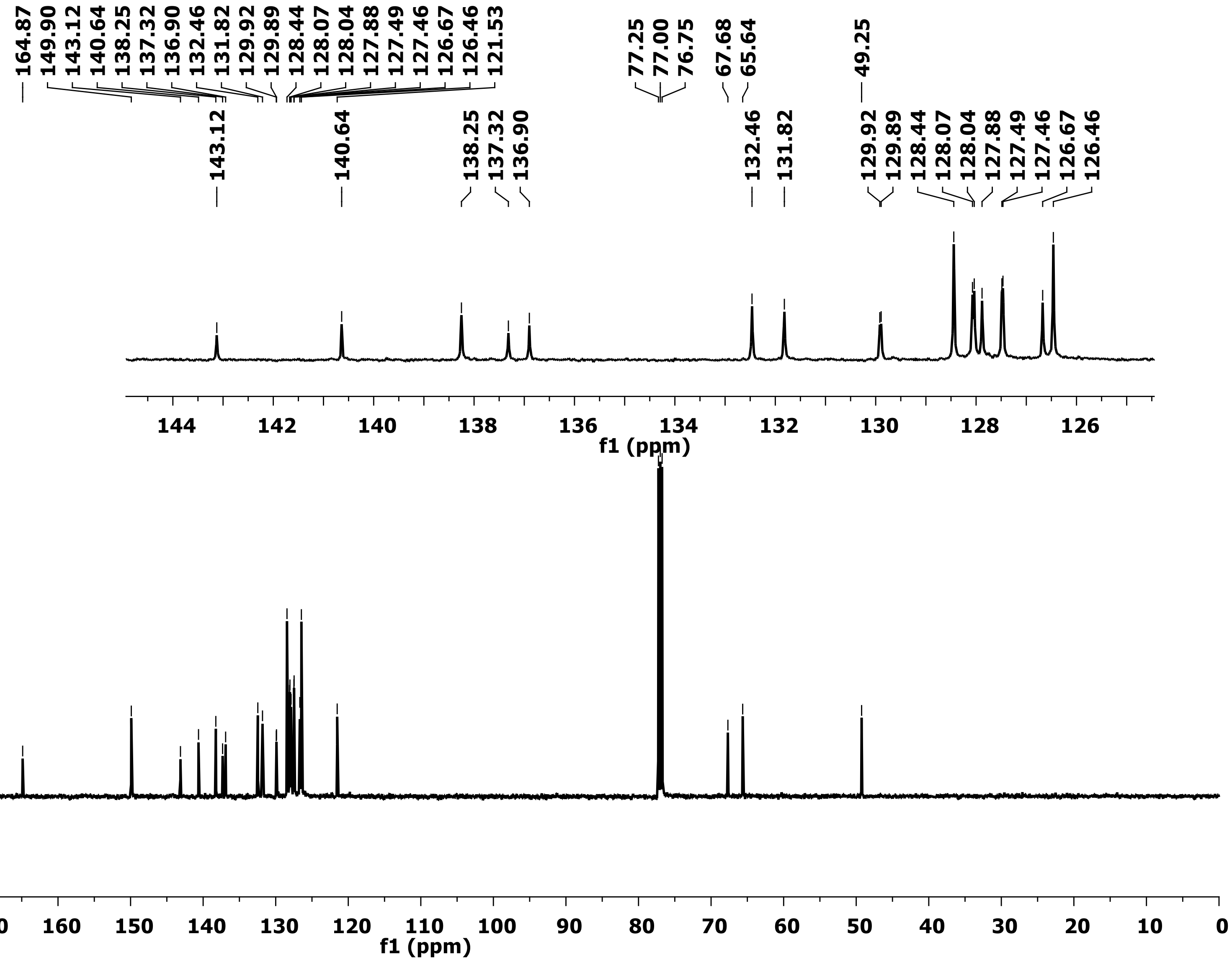
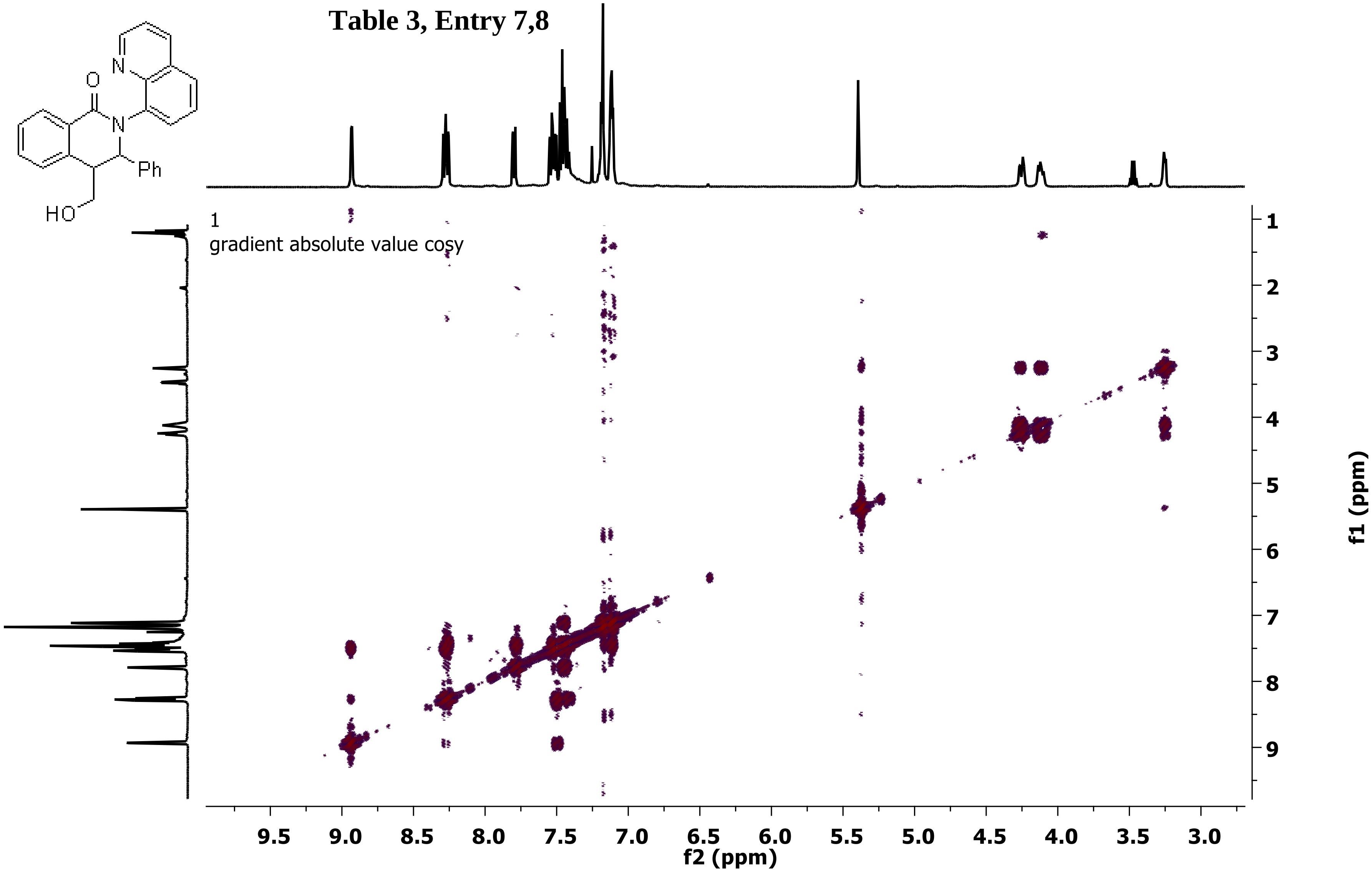


Table 3, Entry 7,8



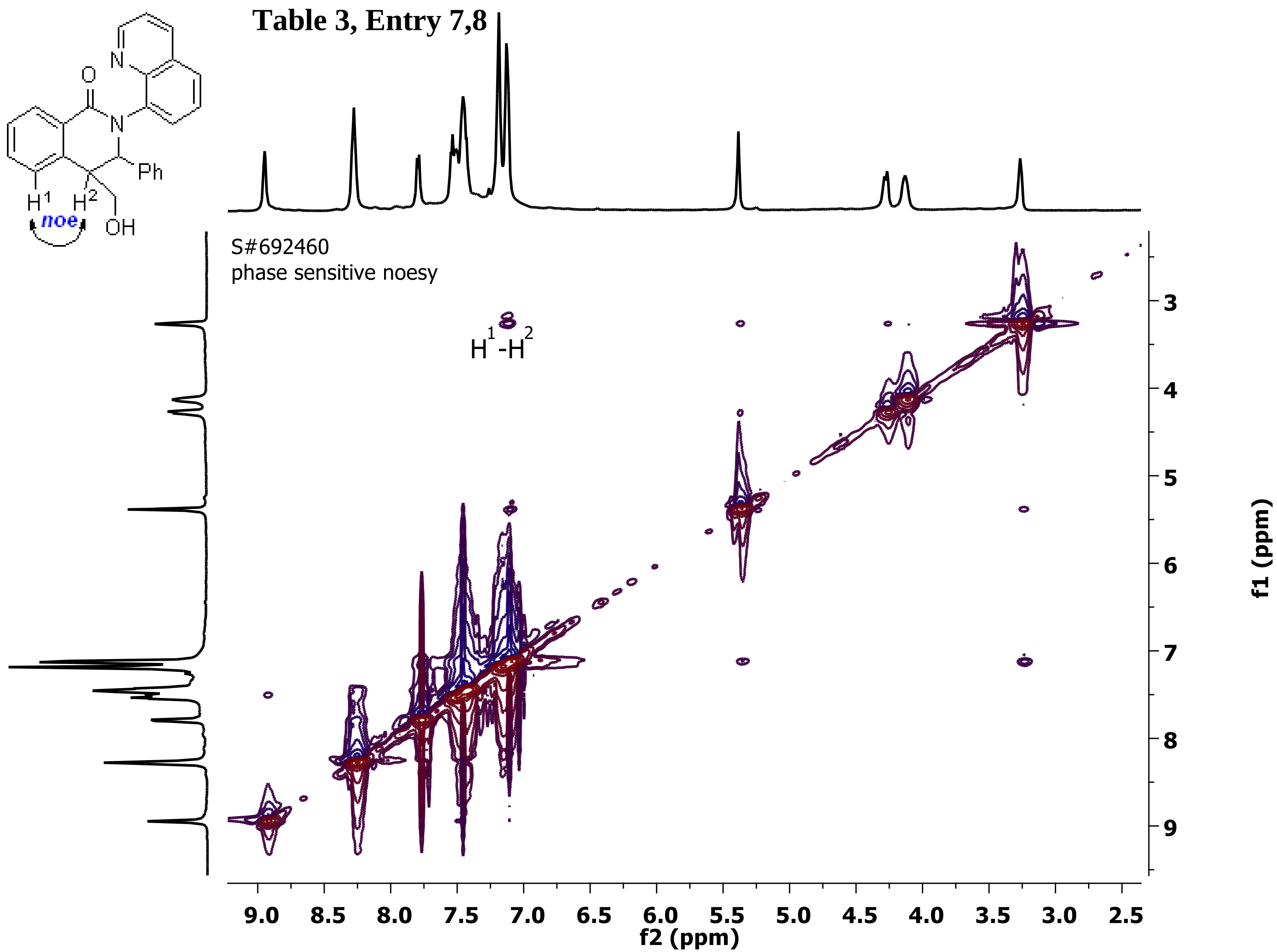
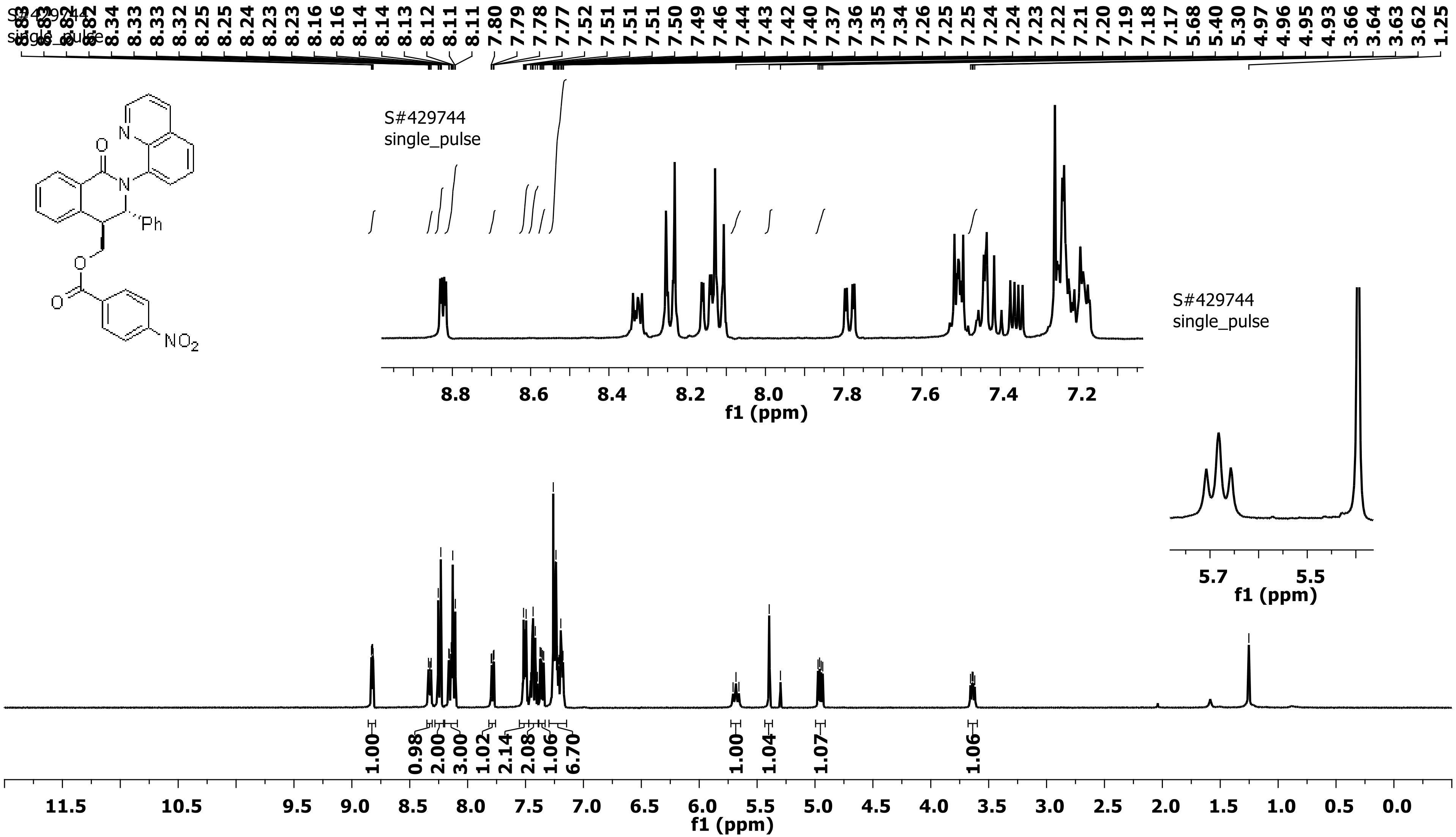
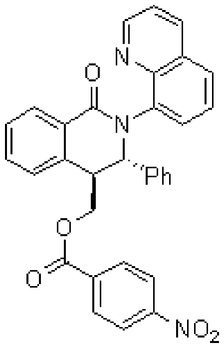
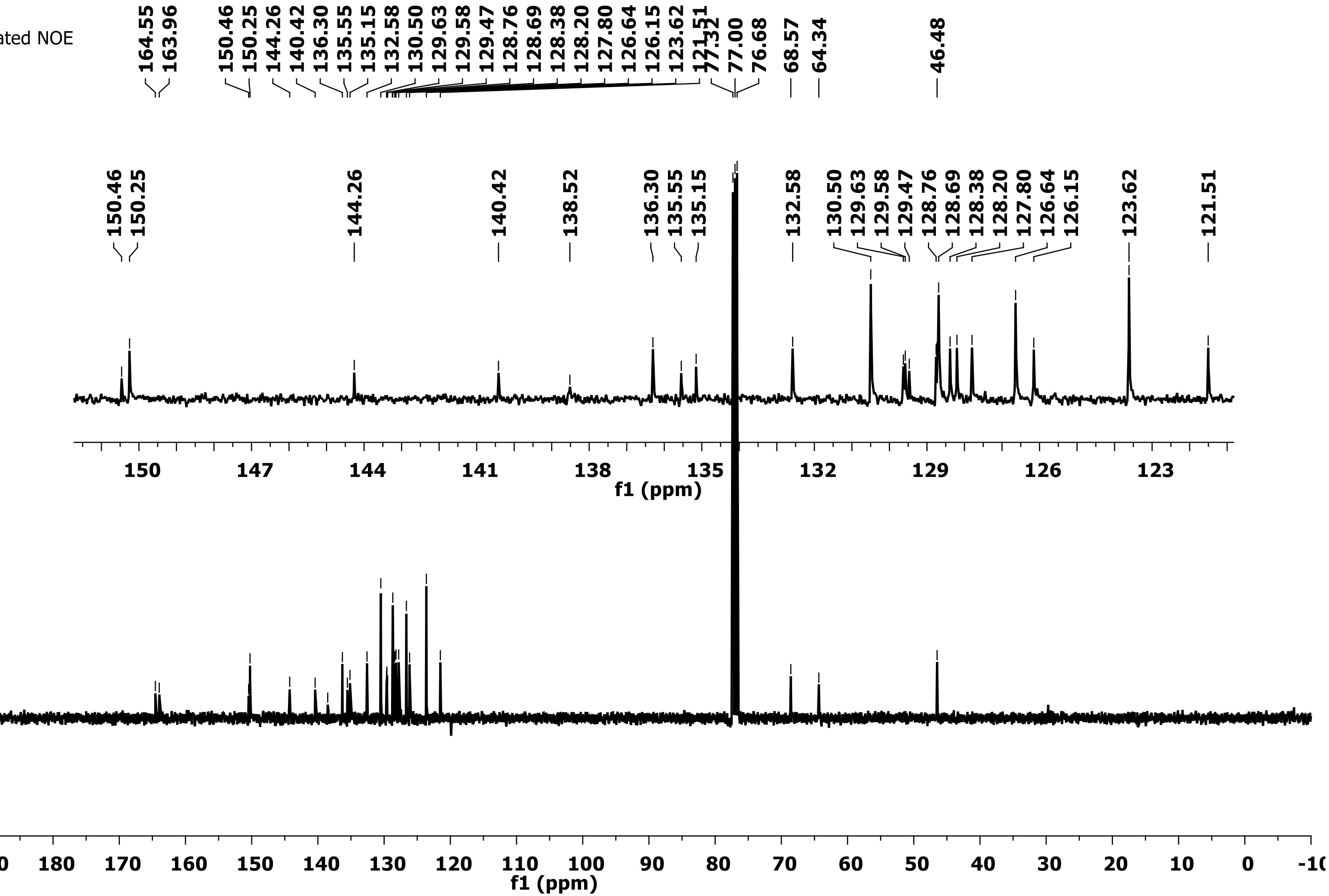


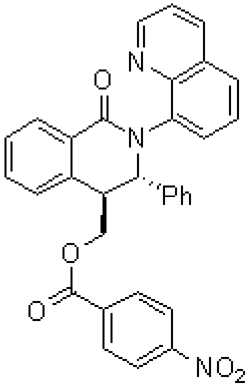
Table 3, Entry 7,8
O
N
N
Ph
H1
H2
noe
OH
S#692460
phase sensitive noesy
H1-H2
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5
f2 (ppm)
3 4 5 6 7 8 9
f1 (ppm)

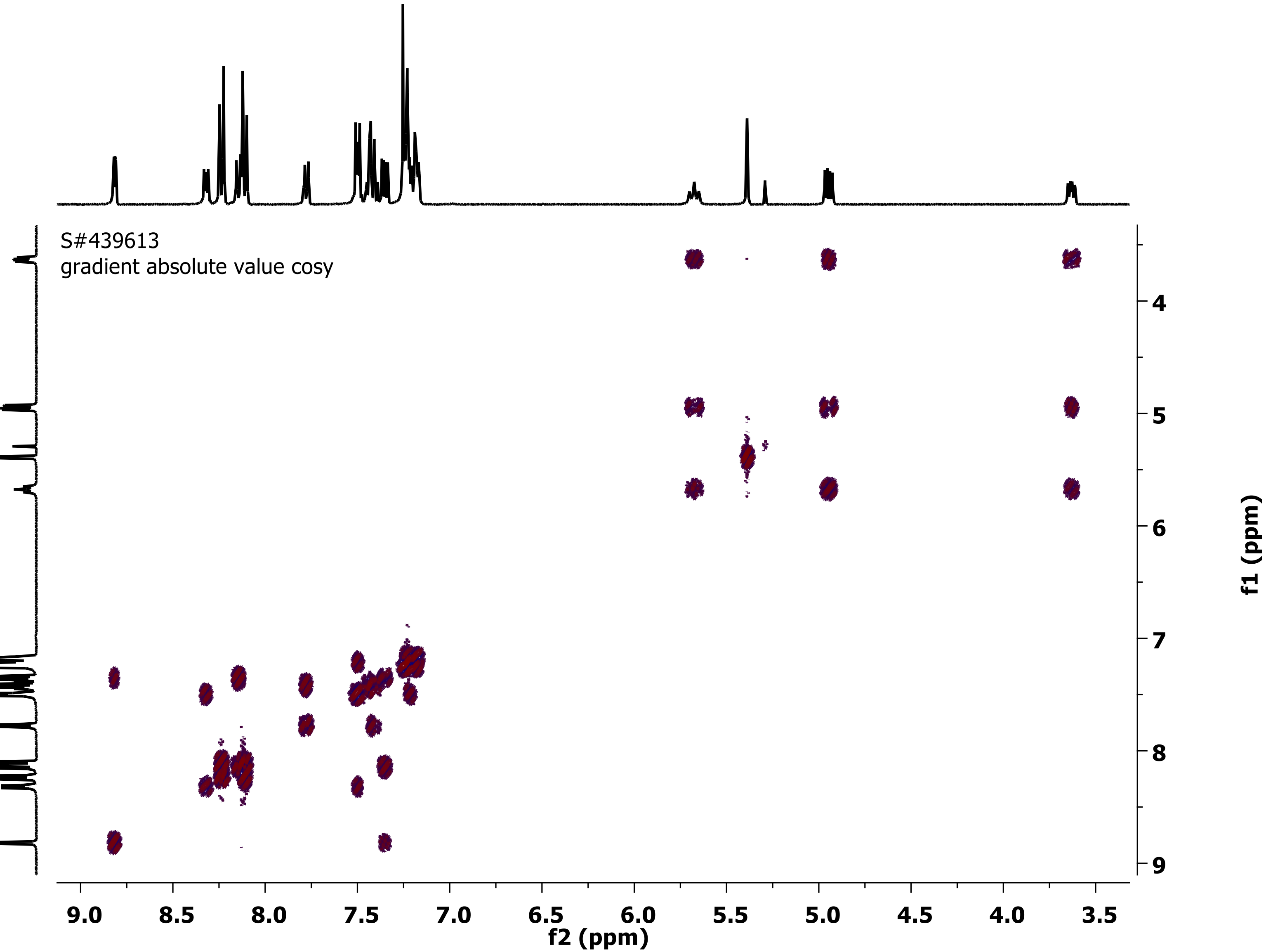

8.83 8.83 8.82 8.82 8.34 8.33 8.33 8.32 8.25 8.25 8.24 8.23 8.23 8.16 8.16 8.14 8.14 8.13 8.12 8.11 8.11 7.80 7.79 7.78 7.77 7.52 7.51 7.51 7.51 7.50 7.49 7.46 7.44 7.43 7.42 7.40 7.37 7.36 7.35 7.34 7.26 7.25 7.25 7.24 7.24 7.23 7.22 7.21 7.20 7.19 7.18 7.17 5.68 5.40 5.30 4.97 4.96 4.95 4.93 3.66 3.64 3.63 3.62 1.25
S#429744
single_pulse
N
O
N
Ph
O
O
NO2
f1 (ppm)
1.00 0.98 2.00 3.00 1.02 2.14 2.08 1.06 6.70 1.00 1.04 1.07 1.06

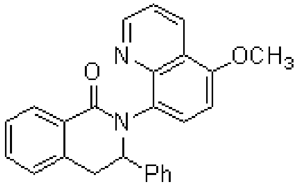
1
single pulse decoupled gated NOE



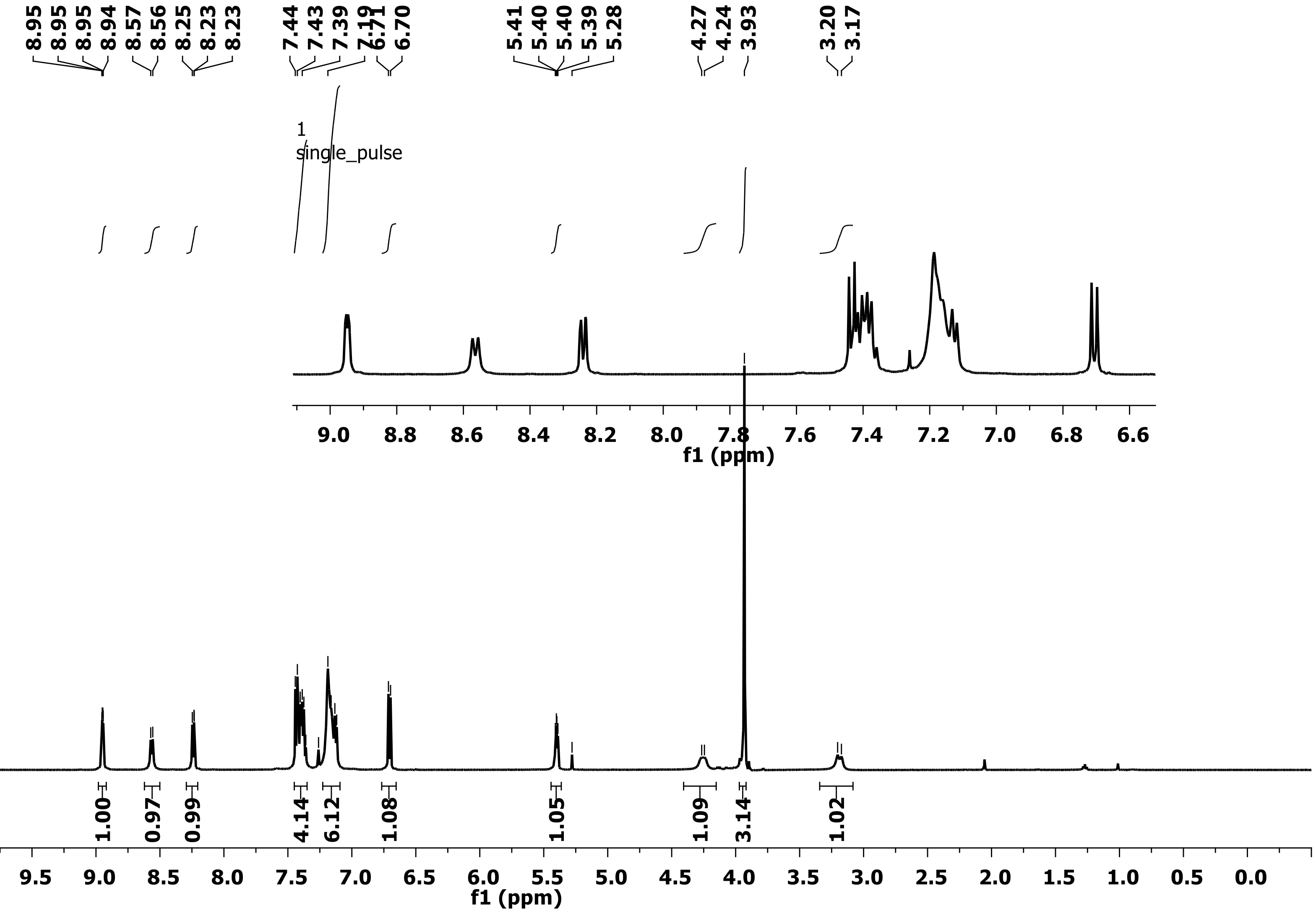



O
N
N
Ph
O
O
NO2


S#439613
gradient absolute value cosy
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
f2 (ppm)
4
5
6
7
8
9
f1 (ppm)

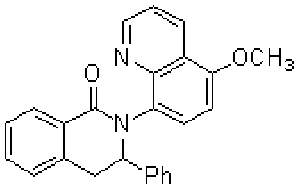


8, Scheme 2



1
single_pulse
8.95 8.95 8.95 8.94 8.57 8.56 8.25 8.23 8.23 7.44 7.43 7.39 7.19 6.71 6.70 5.41 5.40 5.40 5.39 5.28 4.27 4.24 3.93 3.20 3.17
1.00 0.97 0.99 4.14 6.12 1.08 1.05 1.09 3.14 1.02
f1 (ppm)

1
single pulse decoupled gated NOE



8, Scheme 2