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SUPPLEMENTARY INFORMATION

FOR

Deoxygenation of N-heterocyclic *N*-oxides using isopropanol as the recyclable reductant

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GENERAL INFORMATION

General Methods

Experiments involving moisture- and/or air-sensitive compounds were performed in oven- or flame-dried glassware with rubber septa under a positive pressure of nitrogen using standard Schlenk techniques. Nonaqueous reagents were transferred by hypodermic syringe. Heating was accomplished by silicon oil bath using a temperature controller. Brine is defined as a saturated aqueous solution of sodium chloride. Organic solutions were concentrated under reduced pressure at 30 °C (water bath temperature) using a Büchi rotary evaporator, unless otherwise noted. Reactions were magnetically stirred and monitored by thin-layer chromatography (TLC) or GC. Yields refer to chromatographically and spectroscopically (^{1}H NMR) homogeneous (>95%) materials, unless otherwise stated. Melting points were measured using a Stuart SMP50 apparatus and are uncorrected.

Chromatography

TLC was performed on E. Merck 60–F254 precoated plates (0.25 mm), and spots were visualised by UV fluorescence (254 nm) quenching, iodine vapor, and $KMnO_4$ staining. Flash chromatography was carried out with Merck silica gel 60 (200–400 mesh) according to the procedure of Still et al.[1]

NMR & IR Spectroscopy

^{1}H and ^{13}C NMR spectra were recorded with Bruker Avance III Ascend 500 (500 MHz and 126 MHz, respectively) and Avance III HD (400 MHz and 101 MHz, respectively) spectrometers. ^{19}F NMR spectra were obtained on a Bruker Avance III Ascend 500 spectrometer (471 MHz). Chemical shifts of the ^{1}H NMR ($CDCl_3$: 7.26 ppm, MeOD: 4.78 ppm, DMSO-d_6: 2.50 ppm) and ^{13}C NMR ($CDCl_3$: 77.00 ppm, MeOD: 49.15 ppm, DMSO-d_6: 39.50 ppm) spectra were referenced to residual solvent peaks or tetramethylsilane (0.00 ppm) as an internal standard. ^{1}H NMR spectra are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quarter, quin = quintet, sext = sextet, sept = septet, m = multiplet, br = broad), coupling constant (J) in Hz and integration. Infrared (IR) spectra were recorded on a Brucker AVATAR 370 DTGS spectrometer using thin film samples on KBr plates and are reported in terms of frequency of absorption (cm^{-1}).

Mass Spectrometry

Low-resolution mass spectra were acquired using a HP 6890 GC system coupled with a HP 5973 mass selective detector with electron impact (EI) mode or an Agilent 1260 infinity LC-MS system coupled with an Agilent 6120 quadrupole mass spectrometer with electrospray ionisation (ESI) mode. High-resolution mass spectra (HRMS) were obtained from the Organic Chemistry Research Center (OCRC) at Sogang University (Seoul, Korea) using a Bruker Compact ESI-TOF mass spectrometer.

Starting Materials

Unless otherwise stated, all reagents and solvents were purchased at the highest commercial quality from commercial suppliers (Sigma-Aldrich, TCI, Alpha Aesar, Strem, or Acros) and used as received without further purification.

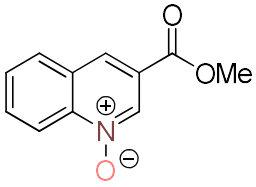
EXPERIMENTAL DATA

Experimental Procedures and Compounds Characterisation Data

Preparation of N-Heterocyclic *N*-Oxides (1)

Unless stated otherwise, N-heterocyclic *N*-oxides used in this study were prepared according to the literature procedures. They were further purified by recrystallisation from an appropriate mixture of solvents, as indicated, and then stored in a desiccator.

Preparation of Methyl Quinoline-3-carboxylate *N*-Oxide (1a)



A 250-mL Schlenk-type flask, equipped with a magnetic stir bar and a cold finger condenser, was charged with 3-quinolinecarboxylic acid (2.86 g, 16.5 mmol, 1.0 equivalent) and $SOCl_2$ (18 mL). The resulting mixture was heated at 60 °C for 4 h. To this mixture was added MeOH (60 mL), NEt_3 (24 mL), and the resulting mixture was stirred overnight at room temperature. The reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with EtOAc (15 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (CH_2Cl_2/MeOH = 10:1) on silica gel to afford methyl quinoline-3-carboxylate (**2a**, 2.9 g, 96 %) as a white solid. A 100-mL round-bottomed flask was charged with methyl quinoline-3-carboxylate (748.8 mg, 4.00 mmol, 1.00 equivalent) and CH_2Cl_2 (13.3 mL), and the resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 1.34 g, 6.00 mmol, 1.5 equivalent) in CH_2Cl_2 (17 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing the pyridine at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 30 minutes at that temperature, the reaction mixture was poured

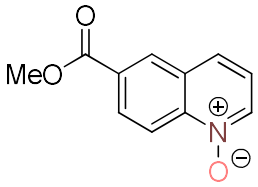
into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc/MeOH = 9:1) on silica gel to afford an off-white solid, which was further purified by recrystallization from a hot mixture of EtOAc and hexanes (1:1, *v*/*v*) to yield the title compound as a white solid (565 mg, 71%). Spectroscopic data matched with those reported in the literature.[4,6]

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.0 (d, *J* = 1.4 Hz, 1H), 8.8–8.7 (m, 1H), 8.4 (s, 1H), 8.0 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.8 (ddd, *J* = 8.5, 6.9, 1.4 Hz, 1H), 7.7 (ddd, *J* = 8.2, 6.9, 1.2 Hz, 1H), 4.0 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.7, 142.9, 134.8, 132.4, 129.4 (d, *J* = 10.0 Hz), 129.0, 127.5, 124.2, 119.8, 52.8.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{11}H_{10}NO_3$) 204.1, found 204.1.

Preparation of Methyl Quinoline-6-carboxylate *N*-Oxide (1b)



A 100-mL round-bottomed flask was charged with methyl quinoline-6-carboxylate (748.8 mg, 4.00 mmol, 1.00 equivalent) and CH_2Cl_2 (13.3 mL), and the resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 1.34 g, 6.00 mmol, 1.50 equivalent) in CH_2Cl_2 (17 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing the pyridine at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 30 minutes at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc/MeOH = 10:1) on silica gel to afford an off-white solid, which was further

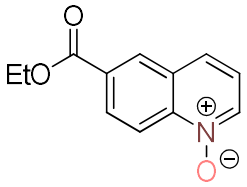
purified by recrystallization from a hot mixture of EtOAc and hexanes (10:1, *v*/*v*) to yield the title compound as a white solid (577 mg, 71%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.7 (d, *J*= 10.9 Hz, 1H), 8.6–8.5 (m, 2H), 8.2 (dt, *J*= 9.2, 1.7 Hz, 1H), 7.8 (d, *J*= 8.3 Hz, 1H), 7.3 (dd, *J*= 8.5, 6.1 Hz, 1H), 3.9 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7, 143.2, 137.1, 131.0, 130.4, 129.9, 129.8, 126.6, 121.9, 120.3, 52.7.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{11}H_{10}NO_3$) 204.1, found 204.1.

Preparation of Ethyl Quinoline-6-carboxylate *N*-Oxide (1c)



A 100-mL schlenk flask, equipped with a magnetic stir bar and a reflux condenser, was charged with 6-quinolinecarboxylic acid (1.4 g, 8.00 mmol, 1.0 equivalent), ethanol (50 mL), and H_2SO_4 (1.0 mL). The resulting mixture was heated to reflux overnight. The mixture was allowed to cool to room temperature, diluted with CH_2Cl_2, and neutralized with an aqueous solution of saturated $NaHCO_3$. The mixture was then extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with water and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (hexenes/EtOAc = 2:1) on silica gel to afford ethyl quinoline-6-carboxylate (**2c**, 1.2 g, 68 %) as a white solid. A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with **2c** (503 mg, 2.50 mmol, 1.0 equivalent) and CH_2Cl_2 (7.0 mL). The resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 647 mg, 3.75 mmol, 1.5 equivalent) in CH_2Cl_2 (10 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing **2c** at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 13 hours at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue

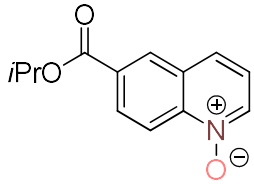
was purified by flash column chromatography (EtOAc/MeOH = 8:1) on silica gel to afford an off-white solid, which was further purified by recrystallization from a hot mixture of EtOAc and hexanes (1:1, *v*/*v*) to yield the title compound as a white solid (364 mg, 67%). Spectroscopic data matched with those reported in the literature.[4,6]

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.77 (d, *J* = 9.1 Hz, 1H), 8.59 (d, *J* = 1.8 Hz, 1H), 8.56 (dd, *J* = 6.0, 1.0 Hz, 1H), 8.31 (dd, *J* = 9.1, 1.8 Hz, 1H), 7.84–7.78 (m, 1H), 7.35 (dd, *J* = 8.4, 6.1 Hz, 1H), 4.44 (q, *J* = 7.1 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 165.2, 143.2, 137.0, 130.8 (two carbons), 129.9, 129.8, 126.4, 121.8, 120.3, 61.7, 14.3.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{12}H_{12}NO_3$) 218.1, found 218.1

Preparation of Isopropyl Quinoline-6-carboxylate *N*-Oxide (1d)



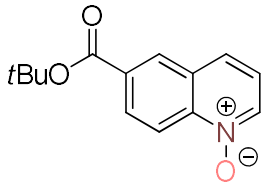
A 100-mL round-bottomed flask was charged with isopropyl quinoline-6-carboxylate (**1d**, 861 mg, 4.00 mmol, 1.00 equivalent) and CH_2Cl_2 (13.3 mL), and the resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 1.34 g, 6.00 mmol, 1.50 equivalent) in CH_2Cl_2 (17 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing the pyridine at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 4 hours at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc/MeOH = 8:1) on silica gel to afford an off-white solid, which was further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to yield the title compound as a white solid (604 mg, 65%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.7 (d, *J* = 9.2 Hz, 1H), 8.5 (dd, *J* = 7.9, 1.7 Hz, 2H), 8.3 (dd, *J* = 9.1, 1.8 Hz, 1H), 7.8 (d, *J* = 8.5 Hz, 1H), 7.3 (dd, *J* = 8.4, 6.1 Hz, 1H), 5.3 (hept, *J* = 6.3 Hz, 1H), 1.4 (d, *J* = 6.4 Hz, 5H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 164.8, 143.3, 137.1, 131.2, 130.8, 130.0, 129.9, 126.6, 121.9, 120.3, 69.5, 22.0.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{13}H_{14}NO_3$) 232.1, found 232.1

Preparation of *tert*-Butyl Quinoline-6-carboxylate *N*-Oxide (1e)



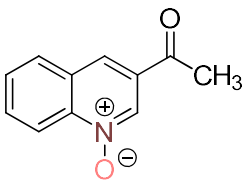
Prepared according to the literature procedure[6] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.97 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.50 (d, *J* = 1.9 Hz, 1H), 8.29–8.21 (m, 2H), 8.10 (d, *J* = 8.8 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 1.63 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 165.2, 152.2, 149.9, 137.2, 130.5, 129.9, 129.5, 129.0, 127.3, 121.6, 81.5, 28.2.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{14}H_{16}NO_3$) 246.1, found 246.1.

Preparation of 3-Acetylquinoline *N*-Oxide (1f)



A 100-mL round-bottomed flask was charged with methyl quinoline-3-carboxylate[4,8] (748.8 mg, 4.00 mmol, 1.00 equivalent) and CH_2Cl_2 (13.3 mL), and the resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 1.34 g, 6.00 mmol, 1.50 equivalent) in CH_2Cl_2 (17 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing the pyridine at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the

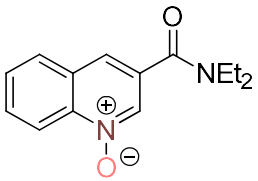
resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 30 minutes at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc/MeOH = 15:1) on silica gel to afford an off-white solid, which was further purified by recrystallization from a hot mixture of EtOAc and hexanes (10:1, *v*/*v*) to yield the title compound as a white solid (546 mg, 67%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.9 (d, *J* = 1.1 Hz, 1H), 8.7 (d, *J* = 8.7 Hz, 1H), 8.2 (s, 1H), 8.0 (d, *J* = 8.1 Hz, 1H), 7.8 (ddd, *J* = 8.5, 6.9, 1.2 Hz, 1H), 7.7 (t, *J* = 7.5 Hz, 1H), 2.7 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 194.3, 143.0, 134.0, 132.7, 130.6, 129.9, 129.7, 129.2, 126.4, 120.0, 26.7.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{11}H_{10}NO_2$) 188.1, found 188.1.

Preparation of *N,N*-Diethyl-3-quinolinecarboxamide *N*-Oxide (1g)



A 25-mL round-bottomed flask was charged with *N*,*N*-diethyl-3-quinolinecarboxamide[9] (114 mg, 0.500 mmol, 1.00 equivalent) and CH_2Cl_2 (1.7 mL), and the resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 168 mg, 0.750 mmol, 0.75 equivalent) in CH_2Cl_2 (1.5 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing the pyridine at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (0.5 mL × 2), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 30 minutes at that temperature, the reaction mixture was poured into water (10 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered,

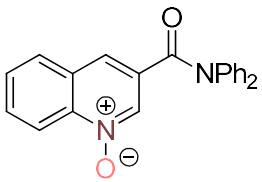
and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc/MeOH = 9:1) on silica gel to afford the title compound as a yellow dense oil (64.4 mg, 53%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.71 (d, *J* = 8.7 Hz, 1H), 8.51 (d, *J* = 1.4 Hz, 1H), 7.87 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.78 (ddd, *J* = 8.6, 6.9, 1.4 Hz, 1H), 7.71 (s, 1H), 7.66 (ddd, *J* = 8.1, 6.8, 1.2 Hz, 1H), 3.45 (d, *J* = 111.4 Hz, 4H), 1.21 (d, *J* = 38.9 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 166.2, 141.5, 133.5, 131.1, 130.9, 129.6, 129.4, 128.5, 123.0, 119.7, 77.3, 77.0, 76.7, 43.5, 39.7, 14.3, 12.8.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{14}H_{17}N_2O_2$) 245.1, found 245.1.

Preparation of *N,N*-Diphenyl-3-quinolinecarboxamide *N*-Oxide (1h)



A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged whit 3-quinolinecarboxylic acid (520 mg, 3.00 mmol, 1.0 equivalent), 1,1'-carbodiimidazole (535 mg, 3.30 mmol, 1.1 equivalent), and dry DMF (4.4 mL, 0.69 M). After stirring in an oil bath (bath temperature = 40 °C) for 24 hours, the mixture was treated with diphenylamine (1.02 g, 6.00 mmol, 2.0 equivalent) followed by DBU (457 mg, 3.00 mmol, 1.0 equivalent). After stirring at 80 °C for 11 hours, the mixture was allowed to cool to room temperature and extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (hexanes/EtOAc = 3:2) on silica gel to afford *N,N*-diphenyl-3-quinolinecarboxamide (**2h**, 407 mg, 42 %) as an off-white solid. A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with **2h** (407 mg, 1.26 mmol, 1.0 equivalent) and CH_2Cl_2 (2.1 mL). The resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 422 mg, 1.88 mmol, 1.5 equivalent) in CH_2Cl_2 (2.1 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing **2h** at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly

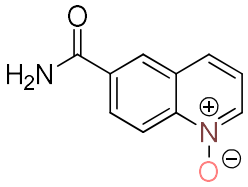
warm to room temperature. After stirring for 12 hours at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed successively with an aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc as a sole eluent) on silica gel to afford an off-white solid, which was further purified by recrystallisation from a hot mixture of EtOAc and hexanes (5:1, *v*/*v*) to yield the title compound as a white solid (305 mg, 71%). All spectroscopic data matched with those reported in the literature.[9]

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 8.7 Hz, 1H), 8.60 (s, 1H), 7.81–7.73 (m, 4H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 5H), 7.28–7.21 (m, 8H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 165.9, 142.8, 141.6, 135.3, 131.7, 130.3, 129.6, 129.3, 129.1, 129.0, 127.3, 126.6, 119.8.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{22}H_{17}N_2O_2$) 341.1, found 341.1.

Preparation of 6-Quinolinecarboxamide *N*-Oxide (1i)



A 50-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with methyl quinoline-6-carboxylate (**2b**, 740 mg, 3.95 mmol, 1.0 equivalent) and methanol (3 mL). The resulting mixture was heated in an oil bath at 45 °C and treated with aqueous ammonia (2.5 mL) via a syringe. After stirring for 10 minutes, the same volume of aqueous ammonia (2.5 mL) was added. The mixture was stirred for 10 minutes before being poured into water. The mixture was extracted with EtOAc (3 × 30 mL). The combined organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (hexanes/EtOAc = 3:1 to 15:1) on silica gel to afford 6-quinolinecarboxamide (**2i**, 286 mg, 42%) as a white solid.

^{1}H NMR (300 MHz, MeOD) δ 8.87 (dd, *J* = 4.4, 1.7 Hz, 1H), 8.41 (dd, *J* = 17.2, 1.9 Hz, 2H), 8.16 (dd, *J* = 8.9, 2.1 Hz, 2H), 8.02 (d, *J* = 8.9 Hz, 1H), 7.54 (dd, *J* = 8.3, 4.3 Hz, 2H).

^{13}C NMR (75 MHz, MeOD) δ 171.3, 153.0, 149.9, 139.4, 133.4, 129.7, 129.5, 129.3, 129.0, 123.3.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{10}H_8N_2O$) 172.1, found 172.1.

A 50-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with **2i** (177 mg, 1.00 mmol, 1.0 equivalent) and CH_2Cl_2 (2 mL). The resulting mixture was cooled in an ice-water bath. In a pear-shaped flask, *m*-CPBA (77%, 690 mg, 4.00 mmol, 1.5 equivalent) was dissolved in CH_2Cl_2 (1.3 mL). The resulting *m*-CPBA solution was added dropwise to the flask containing **2i** at 0 °C via a syringe. After stirring for 5 minutes, the cold reaction mixture was allowed to slowly warm to room temperature and was then treated with CH_2Cl_2 (5 mL). After stirring overnight at room temperature, the reaction mixture was treated with K_2CO_3 (1.66 g, 12.0 mmol, 4.5 equivalent). The resulting mixture was stirred for 3 hours, filtered through a pad of Celite®, and washed with CH_2Cl_2. The filtrate was concentrated using a rotary evaporator. After dissolving the reaction mixture in a 3:1 mixture of CH_2Cl_2 and MeOH, the resulting solution was directly loaded on a silica gel column and eluted with a 3:1 mixture of EtOAc and MeOH to afford 6-quinolinecarboxamide (**1i**, 124 mg, 66%) as a white solid.

^{1}H NMR (400 MHz, DMSO) δ 8.68–8.59 (m, 2H), 8.56 (d, *J* = 9.2 Hz, 1H), 8.33 (s, 1H), 8.22 (dd, *J* = 9.0, 2.0 Hz, 1H), 8.00 (d, *J* = 8.5 Hz, 1H), 7.70 (s, 1H), 7.53 (dd, *J* = 8.5, 6.1 Hz, 1H).

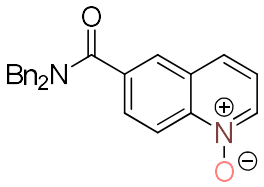
^{13}C NMR (101 MHz, DMSO) δ 166.9, 141.7, 136.4, 134.3, 129.8, 128.7, 128.6, 125.9, 122.6, 119.2.

IR (KBr) 3378, 3199, 1656, 1625, 1611, 1588, 1461, 1407, 1316, 1268, 1025, 915, 857, 794 cm^{-1}.

HRMS (ESI, TOF) *m/z* calculated for $[M + Na]^+$ ($C_{10}H_8N_2NaO_2$) 211.0478, found 211.0478.

Melting point: 202–205 °C (dec.)

Preparation of *N*,*N*-Dibenzyl-6-quinolinecarboxamide *N*-Oxide (1j)



A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with 6-quinolinecarboxylic acid (693 mg, 4.00 mmol, 1.0 equivalent), 1,1'-carbodiimidazole (685 mg, 4.00 mmol, 1.0 equivalent), and dry DMF (5.8 mL, 0.69 M). After stirring in an oil bath (bath temperature = 40 °C) for 2 hours, the mixture was treated with dibenzylamine (2.37 g, 12.0 mmol, 3.0 equivalent)

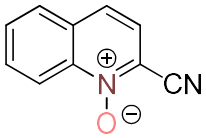
followed by DBU (609 mg, 4.00 mmol, 1.0 equivalent). After stirring at 80 °C for 15 hours, the mixture was allowed to cool to room temperature and extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (hexanes/EtOAc = 1:2) on silica gel to afford *N*,*N*-dibenzyl-6- quinolinecarboxamide (**2j**, 253 mg, 21 %) as an off-white solid. A 25-mL round-bottomed flask equipped with a magnetic stir bar was charged with **2j** (352 mg, 0.500 mmol, 1.0 equivalent) and CH_2Cl_2 (1.7 mL). The resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 168 mg, 0.750 mmol, 1.5 equivalent) in CH_2Cl_2 (1.7 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing **2j** at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 12 hours at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed successively with an aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc as a sole eluent) on silica gel to afford an off-white solid, which was further purified by recrystallisation from a hot mixture of EtOAc and hexanes (3:1, *v*/*v*) to yield the title compound as a white solid (135 mg, 73%). All spectroscopic data matched with those reported in the literature.[8]

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.72 (d, *J* = 9.0 Hz, 1H), 8.48 (d, *J* = 6.0 Hz, 1H), 7.96 (s, 1H), 7.79 (d, *J* = 8.9 Hz, 1H), 7.64 (d, *J* = 8.5 Hz, 1H), 7.43–7.27 (m, 13H), 7.10 (s, 2H), 4.75 (s, 2H), 4.38 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.4, 141.5, 136.6, 136.4, 136.1, 135.8, 130.1, 128.8, 128.7, 128.4, 128.2, 127.7, 127.7, 126.7, 126.5, 125.5, 121.8, 120.4, 51.5, 47.3.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{24}H_{21}N_2O_2$) 369.2, found 369.2

Preparation of 2-Quinolinecarbonitrile *N*-Oxide(1k)



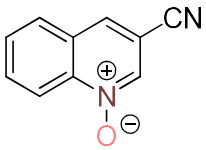
Prepared according to the literature procedure[8] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 8.9 Hz, 1H), 7.92 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.83 (ddd, *J* = 8.7, 7.0, 1.5 Hz, 1H), 7.80–7.70 (m, 2H), 7.52 (d, *J* = 8.7 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 142.2, 131.6, 131.2, 131.1, 128.7, 124.7, 123.5, 121.5, 120.0, 113.0.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{10}H_7N_2O$) 171.1, found 171.1.

Preparation of 3-Quinolinecarbonitrile *N*-Oxide (1l)



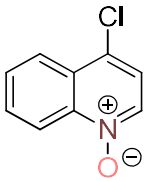
Prepared according to the literature procedure[4,8] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.75 (d, *J* = 8.7 Hz, 1H), 8.63 (s, 1H), 8.09 (s, 1H), 8.01–7.91 (m, 2H), 7.81 (t, *J* = 7.6 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 143.3, 134.9, 133.5, 130.5, 130.0, 129.1, 129.0, 120.1, 114.9, 107.2.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{10}H_7N_2O$) 171.1, found 171.1.

Preparation of 4-Chloroquinoline *N*-Oxide (1m)



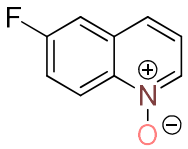
Prepared according to the literature procedure[12] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.75 (dd, *J* = 8.8, 1.2 Hz, 1H), 8.42 (d, *J* = 6.6 Hz, 1H), 8.20 (dd, *J* = 8.4, 1.4 Hz, 1H), 7.82 (ddd, *J* = 8.6, 6.9, 1.4 Hz, 1H), 7.74 (ddd, *J* = 8.3, 6.9, 1.3 Hz, 1H), 7.36 (d, *J* = 6.6 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 142.2, 135.1, 131.1, 129.8, 129.7, 128.1, 125.2, 121.0, 120.4.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ (C_9H_7ClNO) 180.0, found 180.0.

Preparation of 6-Fluoroquinoline *N*-Oxide (1n)



Prepared according to the literature procedure[4,11] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

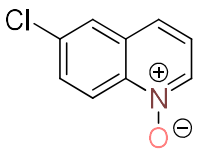
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.76 (dd, *J* = 9.0, 5.2 Hz, 1H), 8.47 (d, *J* = 6.0 Hz, 1H), 7.66 (d, *J* = 8.5 Hz, 1H), 7.53–7.45 (m, 2H), 7.31 (dd, *J* = 8.5, 6.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 161.8 (d, $^1J_{C\text{-}F}$ = 251.8 Hz), 138.7, 135.0, 131.7 (d, *J* = 10.0 Hz), 124.9 (d, $^4J_{C\text{-}F}$ = 5.8 Hz), 122.9 (d, $^3J_{C\text{-}F}$ = 9.3 Hz), 122.2, 120.3 (d, $^2J_{C\text{-}F}$ = 25.5 Hz), 111.5 (d, $^2J_{C\text{-}F}$ = 22.7 Hz).

^{19}F NMR (471 MHz, $CDCl_3$) δ –109.4.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ (C_9H_7FNO) 164.1, found 164.1.

Preparation of 6-Chloroquinoline *N*-Oxide (1o)



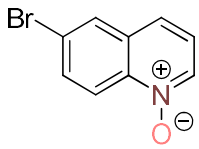
Prepared according to the literature procedure[9] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (d, *J* = 9.3 Hz, 1H), 8.49 (d, *J* = 7.1 Hz, 1H), 7.85 (d, *J* = 2.2 Hz, 1H), 7.71–7.60 (m, 2H), 7.32 (dd, *J* = 8.5, 6.1 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 140.3, 135.8, 135.2, 131.4, 131.3, 126.9, 124.8, 122.4, 121.9.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ (C_9H_7ClNO) 180.0, found 180.0.

Preparation of 6-Bromoquinoline *N*-Oxide (1p)



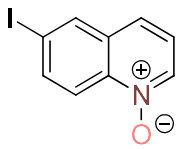
Prepared according to the literature procedure[19] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, v/v) to afford the title compound as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (d, *J* = 9.2 Hz, 1H), 8.51 (d, *J* = 6.0 Hz, 1H), 8.04 (d, *J* = 2.0 Hz, 1H), 7.82 (dd, *J* = 9.2, 2.1 Hz, 1H), 7.63 (d, *J* = 8.5 Hz, 1H), 7.32 (dd, *J* = 8.5, 6.0 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 140.4, 135.8, 133.8, 131.6, 130.1, 124.6, 123.3, 122.2, 121.8.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ (C_9H_7BrNO) 224.0, found 224.0.

Preparation of 6-Iodoquinoline *N*-Oxide (1q)



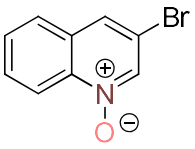
Prepared according to the literature procedure[8] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.49 (d, *J* = 6.1 Hz, 1H), 8.44 (d, *J* = 9.2 Hz, 1H), 8.25 (d, *J* = 1.9 Hz, 1H), 7.97 (dd, *J* = 9.1, 1.9 Hz, 1H), 7.59 (d, *J* = 8.5 Hz, 1H), 7.29 (dd, *J* = 8.5, 6.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 140.9, 139.0, 136.6, 135.8, 132.0, 124.2, 122.0, 121.5, 95.1.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ (C_9H_7INO) 272.0, found 272.0.

Preparation of 3-Bromoquinoline *N*-Oxide (1r)



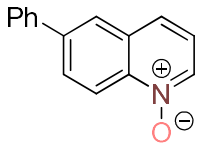
Prepared according to the literature procedure[8] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (dd, *J* = 8.7, 0.9 Hz, 1H), 8.58 (d, *J* = 1.6 Hz, 1H), 7.85 (s, 0H), 7.79–7.68 (m, 2H), 7.63 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 140.6, 137.1, 130.5, 130.3, 129.8, 127.6, 127.4, 119.9, 114.4.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ (C_9H_7BrNO) 224.0, found 224.0.

Preparation of 6-Phenylquinoline *N*-Oxide (1s)



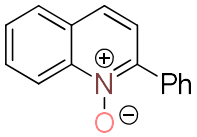
Prepared according to the literature procedure[16] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.81 (d, *J* = 1.7 Hz, 1H), 8.72 (d, *J* = 8.9 Hz, 1H), 7.91–7.85 (m, 2H), 7.75–7.68 (m, 1H), 7.62 (dd, *J* = 7.2, 2.2 Hz, 3H), 7.52–7.46 (m, 2H), 7.43 (dd, *J* = 8.5, 6.2 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 140.4, 136.0, 135.1, 135.0, 130.4, 130.2, 129.4, 129.2, 129.0, 128.4, 127.1, 123.4, 119.8.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{15}H_{12}NO$) 222.1, found 222.1.

Preparation of 2-Phenylquinoline *N*-Oxide (1t)



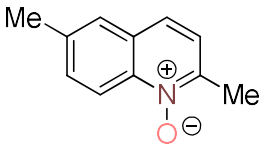
Prepared according to the literature procedure[4,12,14] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.84 (d, *J* = 8.7 Hz, 1H), 7.99–7.93 (m, 2H), 7.83 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.79–7.69 (m, 2H), 7.61 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.53–7.41 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 145.1, 142.3, 133.5, 130.6, 130.6, 129.6, 129.6, 128.5, 128.3, 128.0, 125.3, 123.4, 120.3.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{15}H_{12}NO$) 222.1, found 222.1.

Preparation of 2,6-Dimethylquinoline *N*-Oxide (1u)



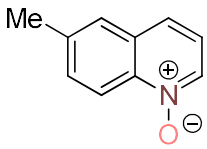
Prepared according to the literature procedure[18] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, *J* = 8.7 Hz, 1H), 7.60–7.53 (m, 3H), 7.28 (s, 1H), 2.71 (s, 3H), 2.53 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.9, 140.1, 137.7, 132.4, 129.3, 126.9, 124.6, 122.9, 119.3, 21.3, 18.6.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{11}H_{12}NO$) 174.1, found 174.1.

Preparation of 6-Methylquinoline *N*-Oxide (1v)



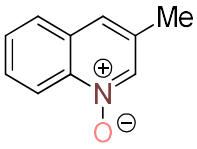
Prepared according to the literature procedure[4,14] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 8.9 Hz, 1H), 8.47 (d, *J* = 5.9 Hz, 1H), 7.65 (d, *J* = 8.5 Hz, 1H), 7.62 (s, 1H), 7.59 (dd, *J* = 8.9, 1.7 Hz, 1H), 7.27–7.23 (m, 1H), 2.54 (s, 3H)

^{13}C NMR (101 MHz, $CDCl_3$) δ 140.1, 139.0, 135.0, 132.6, 130.7, 127.0, 125.5, 120.9, 119.6, 21.4

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{10}H_{10}NO$) 160.1, found 160.1.

Preparation of 3-Methylquinoline *N*-Oxide (1w)



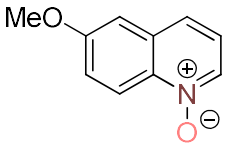
Prepared according to the literature procedure[19] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, *J* = 8.4 Hz, 1H), 8.40 (d, *J* = 1.5 Hz, 1H), 7.75 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.66 (ddd, *J* = 8.6, 6.9, 1.4 Hz, 1H), 7.58 (ddd, *J* = 8.1, 6.9, 1.3 Hz, 1H), 7.50 (s, 1H), 2.43 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.7, 137.0, 131.2, 130.2, 129.3, 128.7, 127.4, 125.4, 119.6, 18.7.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{10}H_{10}NO$) 160.1, found 160.1.

Preparation of 6-Methoxyquinoline *N*-Oxide (1x)



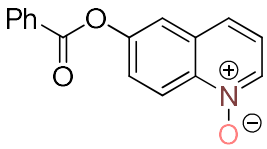
Prepared according to the literature procedure[11] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (d, *J* = 9.6 Hz, 1H), 8.39 (dd, *J* = 6.0, 1.1 Hz, 1H), 7.63 (d, *J* = 8.5 Hz, 1H), 7.42–7.34 (m, 1H), 7.25–7.22 (m, 1H), 7.10 (d, *J* = 2.4 Hz, 1H), 3.94 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.5, 137.3, 133.8, 132.0, 125.0, 122.8, 121.5 (two carbons), 105.8, 55.7.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{10}H_{10}NO_2$) 176.1, found 176.1.

Preparation of 6-Benzoyloxyquinoline *N*-Oxide (1y)



A 100-ml round-bottomed flask, equipped with a magnetic stir bar, was charged with 6-hydroxyquinoline (155 mg, 1.07 mmol, 1.0 equivalent), 4-(*N*,*N*-dimethylamino)pyridine (DMAP, 6.6 mg, 0.054 mmol, 5 mol%), Et_3N (5 mL), and CH_2Cl_2 (25 mL). To this mixture was added dropwise benzoyl chloride (156 mg, 1.07 mmol, 1.0 equivalent) via a syringe at room temperature, and the resulting mixture was stirred for 1 hour. The reaction was quenched by H_2O (1 mL), and the mixture was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed with brine, dried with Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (hexanes/EtOAc = 2:1) on silica gel to afford 6-benzoyloxyquinoline (**2y**, 188 mg, 71 %)

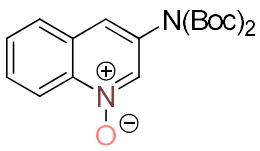
as an off-white solid. A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with **2y** (188 mg, 0.760 mmol, 1.0 equivalent) and CH_2Cl_2 (1.2 mL). The resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 254 mg, 1.13 mmol, 1.5 equivalent) in CH_2Cl_2 (1.3 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing **2y** at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 1 hour at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed successively with an aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc/MeOH = 9:1) on silica gel to afford the title compound as an off-white solid, which was further purified by recrystallisation from a hot mixture of EtOAc and hexanes (5:1, *v*/*v*) to yield the title compound as a white solid (102 mg, 51 %). All spectroscopic data matched with those reported in the literature.[8]

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.80 (d, *J* = 9.5 Hz, 1H), 8.49 (d, *J* = 6.0 Hz, 1H), 8.21 (d, *J* = 6.9 Hz, 2H), 7.76 (d, *J* = 2.6 Hz, 1H), 7.71–7.57 (m, 4H), 7.52 (t, *J* = 7.9 Hz, 2H), 7.29 (dd, *J* = 8.5, 6.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 164.8, 150.8, 139.7, 135.4, 134.1, 131.3, 130.3, 128.9, 128.8, 125.6, 125.5, 122.0, 121.9, 119.2.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{16}H_{12}NO_3$) 266.1, found 266.1.

Preparation of 3-[Bis(*tert*-butoxycarbonyl)amino]quinoline *N*-Oxide (1z)



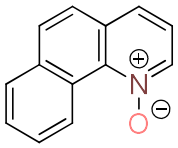
Prepared according to the literature procedure[4,10] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.68 (d, *J* = 8.8 Hz, 1H), 8.36 (d, *J* = 1.8 Hz, 1H), 7.82 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.73 (ddd, *J* = 8.5, 6.9, 1.4 Hz, 1H), 7.62 (ddd, *J* = 8.1, 6.8, 1.2 Hz, 1H), 7.53 (d, *J* = 1.8 Hz, 1H), 1.39 (s, 18H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 150.7, 140.6, 136.2, 133.2, 130.7, 129.3, 129.2, 128.4, 124.7, 119.8, 84.2, 27.9.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{19}H_{25}N_2O_5$) 361.2, found 361.2.

Preparation of Benzo[*h*]quinoline *N*-Oxide (1aa)



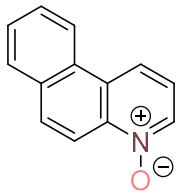
A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with benzo[*h*]quinoline (**2aa**, 269 mg, 1.50 mmol, 1.0 equivalent) and CH_2Cl_2 (5 mL). The resulting mixture was cooled in an ice-water bath. In a pear-shaped flask, *m*-CPBA (77%, 388 mg, 2.25 mmol, 1.5 equivalent) was dissolved in CH_2Cl_2 (2.5 mL). The resulting *m*-CPBA solution was added dropwise to the flask containing **2aa** at 0 °C via a syringe. After stirring for 5 minutes, the cold reaction mixture was allowed to slowly warm to room temperature and stirred overnight. The reaction mixture was re-cooled to 0 °C and treated with an additional *m*-CPBA solution (77%, 129 mg, 0.75 mmol, 0.5 equivalent) in CH_2Cl_2 (2 mL) via a syringe. After stirring for 5 hours, the reaction mixture was poured into water (10 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine. It was then dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue obtained was purified by flash column chromatography (EtOAc/MeOH = 7:1) on silica gel to afford the title compound (**1aa**, 204 mg, 70%) as a white solid. All spectroscopic data matched with those reported in the literature.[20]

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.84 (dd, *J* = 7.3, 2.6 Hz, 1H), 8.63 (dd, *J* = 6.3, 1.3 Hz, 1H), 7.88 (dd, *J* = 7.3, 2.2 Hz, 1H), 7.82–7.66 (m, 4H), 7.58 (d, *J* = 8.9 Hz, 1H), 7.34 (dd, *J* = 8.1, 6.2 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.3, 138.5, 134.1, 131.2, 130.6, 129.1, 128.3, 128.1, 127.7, 126.1, 125.7, 125.0, 121.2.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{13}H_{10}NO$) 196.1, found 196.1.

Preparation of Benzo[*f*]quinoline *N*-Oxide (1ab)



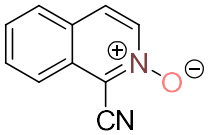
A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with benzo[*f*]quinoline (**2ab**, 269 mg, 1.50 mmol, 1.0 equivalent) and CH_2Cl_2 (5 mL). The resulting mixture was cooled in an ice-water bath. In a pear-shaped flask, *m*-CPBA (77%, 388 mg, 2.25 mmol, 1.5 equivalent) was dissolved in CH_2Cl_2 (2.5 mL). The resulting *m*-CPBA solution was added dropwise to the flask containing **2ab** at 0 °C via a syringe. After stirring for 5 minutes, the cold reaction mixture was allowed to slowly warm to room temperature and stirred overnight. The reaction mixture was re-cooled to 0 °C and treated with an additional *m*-CPBA solution (77%, 129 mg, 0.75 mmol, 0.5 equivalent) in CH_2Cl_2 (2 mL) via a syringe. After stirring for 5 hours, the reaction mixture was poured into water (10 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine. It was then dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue obtained was purified by flash column chromatography (EtOAc/MeOH = 7:1) on silica gel to afford the title compound (**1ab**, 219 mg, 75%) as a white solid. All spectroscopic data matched with those reported in the literature.[25]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, *J* = 10.3 Hz, 1H), 8.59–8.46 (m, 2H), 8.41 (d, *J* = 8.7 Hz, 1H), 7.97 (d, *J* = 9.5 Hz, 1H), 7.93–7.86 (m, 1H), 7.72–7.61 (m, 2H), 7.39 (dd, *J* = 8.5, 6.1 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.1, 136.2, 132.1, 132.1, 129.1, 128.9, 128.6, 128.5, 128.0, 123.3, 121.4, 120.5, 117.2.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{13}H_{10}NO$) 196.1, found 196.1.

Preparation of 1-Isoquinolinecarbonitrile *N*-Oxide (1ac)



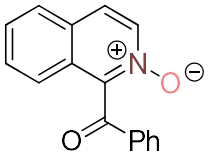
A 100-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with 1-isoquinolinecarbonitrile (**2ac**) (308 mg, 2.00 mmol, 1.00 equivalent) and CH_2Cl_2 (3.0 mL). The resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 672 mg, 3.00 mmol, 1.5 equivalent) in CH_2Cl_2 (4 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing **2ac** at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 12 hours at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue thus obtained was purified by flash column chromatography (EtOAC/MeOH =12:1) on silica gel to afford the title compound as a white solid, which was further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to yield the title compound as a white solid (186 mg, 55%). Spectroscopic data matched with those reported in the literature.[9,24]

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.16 (d, *J* = 7.2 Hz, 1H), 7.99 (dd, *J* = 8.4, 0.9 Hz, 1H), 7.87 (dt, *J* = 8.1, 0.8 Hz, 1H), 7.84 (d, *J* = 7.3 Hz, 1H), 7.80 (ddd, *J* = 8.4, 7.1, 1.2 Hz, 1H), 7.68 (ddd, *J* = 8.2, 7.0, 1.2 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 137.2, 131.9, 130.3, 129.7, 127.7, 127.6, 127.2, 123.2, 121.9, 111.3.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{13}H_7N_2O$) 171.1, found 171.1.

Preparation of 1-Benzoylisoquinoline *N*-Oxide (1ad)



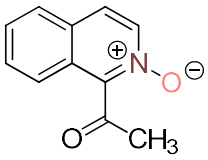
Prepared according to the literature procedure[25] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.13 (d, *J* = 7.2 Hz, 1H), 7.84 (dt, *J* = 8.7, 1.7 Hz, 3H), 7.77 (d, *J* = 7.2 Hz, 1H), 7.59 (dddd, *J* = 8.2, 6.9, 3.5, 1.4 Hz, 2H), 7.54 (td, *J* = 7.6, 6.9, 1.4 Hz, 1H), 7.49 (d, *J* = 8.2 Hz, 1H), 7.47–7.42 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 189.7, 142.4, 136.7, 135.3, 134.6, 130.2, 129.3, 129.2, 129.1, 128.8, 127.5, 127.3, 125.0, 123.2.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{16}H_{12}NO_2$) 250.1, found 250.1.

Preparation of 1-Acetylisoquinoline *N*-Oxide (1ae)



A 100-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with isoquinoline (517 mg, 4.00 mmol, 1.0 equivalent), acetaldehyde (705 mg, 16.0 mmol, 4.0 equivalent), tetra-*n*-butylammonium bromide (387 mg, 1.20 mmol, 0.3 equivalent), $K_2S_2O_8$ (2.16 g, 8.00 mmol, 2.0 equivalent), and 1,2-dichloroethane (33 mL). The resulting mixture was heated in an oil bath (bath temperature = 100 °C) under reflux for 3 hours. The mixture was allowed to cool to room temperature, and the solvent was removed under vacuum. The crude mixture was diluted with CH_2Cl_2, washed successively with an aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography on silica gel (CH_2Cl_2 as a sole eluent) to afford 1-acetylisoquinoline (**2ae**, 437 mg, 64 %) as a white solid. A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with **2ae** (437 mg, 2.55 mmol, 1.0 equivalent) and CH_2Cl_2 (4.0 mL). The resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 1.14 g,

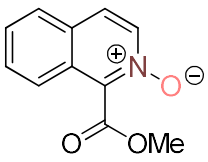
5.11 mmol, 2.0 equivalent) in CH_2Cl_2 (4.5 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing **2ae** at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 17 hours at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with an aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc as a sole eluent) on silica gel to afford an off-white solid, which was further purified by recrystallisation from a hot mixture of hexanes and EtOAc (3:1, *v*/*v*) to yield the title compound as a white solid (209 mg, 44%). Spectroscopic data matched with those reported in the literature.[8]

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.1 Hz, 1H), 7.82–7.74 (m, 1H), 7.67 (dd, *J* = 17.3, 7.6 Hz, 2H), 7.58 (pd, *J* = 6.9, 1.3 Hz, 4H), 2.73 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 197.0, 143.2, 136.4, 130.2, 128.9, 127.1, 126.0, 124.9, 123.0, 29.7.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{11}H_{10}NO_2$) 188.1, found 188.1.

Preparation of Methyl 1-Isoquinolinecarboxylate *N*-Oxide (1af)



Prepared according to the literature procedure[27]. In a 50-mL Schlenk-type flask, equipped with a magnetic stir bar and a reflux condenser, 1-isoquinolinecarboxylic acid (346 mg, 2.00 mmol, 1.0 equivalent), MeOH (20 mL), and H_2SO_4 (1 mL) were combined. The resulting mixture was heated in an oil bath (bath temperature = 80 °C) under reflux overnight. The mixture was allowed to cool to room temperature, diluted with CH_2Cl_2, and neutralised with an aqueous solution of saturated $NaHCO_3$.The mixture was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed successively with water and brine and dried over anhydrous Na_2SO_4. It was filtered and concentrated on a rotary evaporator. The residue obtained was purified by flash column chromatography (Hex/EtOAc = 2:1) on

silica gel to afford methyl 1-isoquinolinecarboxylate (**2af**, 329 mg, 88%) as a white solid. All spectroscopic data matched with those reported in the literature.

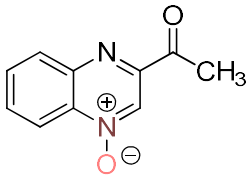
A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with methyl 1-isoquinolinecarboxylate (**2af**, 281 mg, 1.50 mmol, 1.0 equivalent) and CH_2Cl_2 (5 mL). The resulting mixture was cooled in an ice-water bath. In a pear-shaped flask, *m*-CPBA (77%, 388 mg, 2.25 mmol, 1.5 equivalent) was dissolved in CH_2Cl_2 (2.5 mL). The resulting *m*-CPBA solution was added dropwise to the flask containing **2af** at 0 °C via a syringe. After stirring for 5 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring overnight, the reaction mixture was poured into water (10 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine. It was dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc/MeOH = 15:1) on silica gel to afford the title compound (**1af**, 238 mg, 78%) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 7.2 Hz, 1H), 7.75 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.66 (d, *J* = 7.2 Hz, 1H), 7.61–7.50 (m, 3H), 4.09 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 162.4, 138.6, 136.6, 130.2, 129.0, 128.4, 127.1, 126.5, 124.9, 123.0, 53.5.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{11}H_{10}NO_3$) 204.1, found 204.1

Preparation of Methyl 2-Quinoxalinecarboxylate *N*-Oxide (1ag)



A 150-mL round-bottomed flask, equipped with a magnetic stir bar and a reflux condenser, was charged with quinoxaline (651 mg, 5.00 mmol, 1.0 equivalent), acetaldehyde (1.10 g, 20.0 mmol, 4.0 equivalent), tetra-*n*-butylammonium bromide (484 mg, 1.50 mmol, 0.3 equivalent), $K_2S_2O_8$ (2.70 g, 10.0 mmol, 2.0 equivalent), and 1,2-dichloroethane (42 mL). The resulting mixture was heated in an oil bath (bath temperature = 100 °C) under reflux for 2 hours. The mixture was allowed to cool to room temperature, and the solvent was removed under vacuum. The crude mixture was diluted with CH_2Cl_2, washed

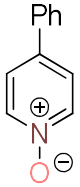
successively with an aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography on silica gel (hexanes/EtOAc = 7:1) to afford 2-acetylquinoxaline (**2ag**) as a white solid (437 mg, 64 %). A 50-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with **2ag** (528 mg, 3.08 mmol, 1.0 equivalent) and CH_2Cl_2 (5.0 mL). The resulting mixture was cooled in an ice-water bath. A separate pear-shaped flask was charged with *m*-CPBA (77%, 1.38 g, 6.16 mmol, 2.0 equivalent) in CH_2Cl_2 (5.3 mL), and the resulting *m*-CPBA solution was added dropwise to the flask containing **2ag** at 0 °C via a syringe. The pear-shaped flask was rinsed with CH_2Cl_2 (1.0 mL × 3), and the resulting solution was added via a syringe. After stirring for 10 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring for 10 hours at that temperature, the reaction mixture was poured into water (20 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (15 mL × 3). The combined organic layer was washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine, dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (hexene/EtOAc = 4:1) on silica gel to afford a yellow solid, which was further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to yield the title compound as a white solid (209 mg, 44%). Spectroscopic data matched with those reported in the literature.[17]

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.86 (s, 1H), 8.55 (dd, *J* = 8.5, 1.7 Hz, 1H), 8.19 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.84 (dddd, *J* = 25.8, 8.4, 7.0, 1.5 Hz, 2H), 2.79 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 197.9, 149.8, 144.4, 138.1, 132.3, 132.3, 132.1, 127.3, 119.2, 25.4.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{10}H_9N_2O_2$) 189.1, found 189.1.

Preparation of 4-Phenylpyridine *N*-Oxide (1ah)



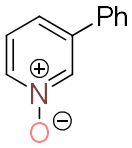
Prepared according to the literature procedure[21] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.29–8.22 (m, 2H), 7.62–7.55 (m, 2H), 7.54–7.40 (m, 5H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.3, 138.9, 136.1, 129.3, 129.2, 126.4, 123.7.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{11}H_{10}NO$) 172.1, found 172.1.

Preparation of 3-Phenylpyridine *N*-Oxide (1ai)



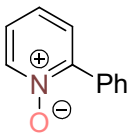
Prepared according to the literature procedure[22] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (2:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.41 (t, *J* = 1.9 Hz, 1H), 8.15 (dt, *J* = 6.4, 1.2 Hz, 1H), 7.51–7.37 (m, 6H), 7.29 (dd, *J* = 8.1, 6.3 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 140.3, 137.6, 137.5, 135.1, 129.4 (two carbons), 126.9, 125.9, 124.6.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{11}H_{10}NO$) 172.1, found 172.1.

Preparation of 2-Phenylpyridine *N*-Oxide (1aj)



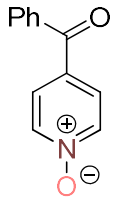
Prepared according to the literature procedure[23] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (dd, *J* = 6.5, 1.3 Hz, 1H), 7.82 (dd, *J* = 8.0, 1.7 Hz, 2H), 7.53–7.39 (m, 4H), 7.30 (td, *J* = 7.7, 1.3 Hz, 1H), 7.22 (ddd, *J* = 7.6, 6.5, 2.2 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 149.3, 140.5, 132.6, 129.6, 129.2, 128.3, 127.4, 125.5, 124.5.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{11}H_{10}NO$) 172.1, found 172.1.

Preparation of 4-Benzoylpyridine *N*-Oxide (1ak)



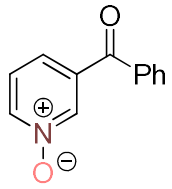
Prepared according to the literature procedure[24] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.26–8.20 (m, 2H), 7.76–7.65 (m, 4H), 7.65–7.58 (m, 1H), 7.50 (tt, *J* = 7.5, 1.5 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 192.1, 139.4 (two carbons), 136.1, 133.3, 132.9, 129.6 (two carbons), 128.8 (two carbons), 127.0 (two carbons).

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{12}H_{10}NO_2$) 200.1, found 200.1.

Preparation of 3-Benzoylpyridine *N*-Oxide (1al)



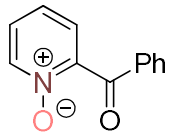
A 25-mL round-bottomed flask, equipped with a magnetic stir bar, was charged with 3-benzoylpyridine (**2al**, 275 mg, 1.50 mmol, 1.0 equivalent) and CH_2Cl_2 (5 mL). The resulting mixture was cooled in an ice-water bath. In a pear-shaped flask, *m*-CPBA (77%, 388 mg, 2.25 mmol, 1.5 equivalent) was dissolved in CH_2Cl_2 (2.5 mL). The resulting *m*-CPBA solution was added dropwise to the flask containing **2al** at 0 °C via a syringe. After stirring for 5 minutes, the cold reaction mixture was allowed to slowly warm to room temperature. After stirring overnight, the reaction mixture was poured into water (10 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (10 mL × 3). The combined organic layer was then washed successively with aqueous solution of saturated $NaHCO_3$, water, and brine. It was dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography (EtOAc/MeOH = 8:1) on silica gel to afford the title compound (**1al**, 63 mg, 21%) as a brown solid. All spectroscopic data matched with those reported in the literature.[26]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (s, 1H), 8.30 (d, *J* = 6.5 Hz, 1H), 7.73 (d, *J* = 7.9 Hz, 2H), 7.58 (dd, *J* = 10.2, 4.2 Hz, 2H), 7.50–7.42 (m, 2H), 7.42–7.32 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 191.6, 141.7, 140.1, 136.6, 135.5, 133.8, 129.9, 128.8, 126.5, 125.9.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{12}H_{10}NO_2$) 200.1, found 200.1.

Preparation of 2-Benzoylpyridine *N*-Oxide (1am)



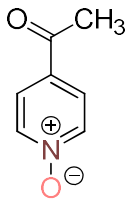
Prepared according to the literature procedure[13] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (ddd, *J* = 4.9, 2.6, 1.1 Hz, 1H), 7.86–7.77 (m, 2H), 7.62–7.53 (m, 1H), 7.44 (tt, *J* = 7.7, 1.8 Hz, 2H), 7.40–7.33 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 189.4, 147.1, 140.0, 135.1, 134.2, 129.3 (two carbons), 128.9 (two carbons), 127.1, 125.7, 125.7.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{12}H_{10}NO_2$) 200.1, found 200.1.

Preparation of 4-Acetylpyridine *N*-Oxide (1an)



Prepared according to the literature procedure[15] and further purified by recrystallisation from a hot mixture of hexanes and EtOAc (1:1, *v*/*v*) to afford the title compound as a white solid.

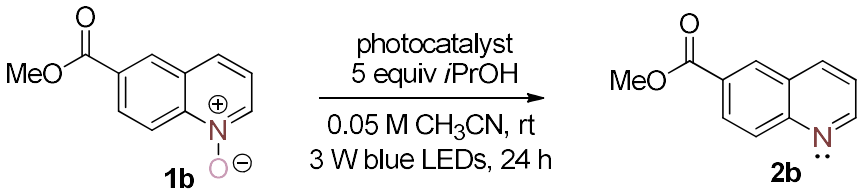
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19 (d, *J* = 6.7 Hz, 2H), 7.76 (d, *J* = 7.3 Hz, 2H), 2.55 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 193.7, 139.6 (two carbons), 132.3, 125.1 (two carbons), 26.3.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_7H_8NO_2$) 138.1, found 138.1.

A Comprehensive Optimisation Study

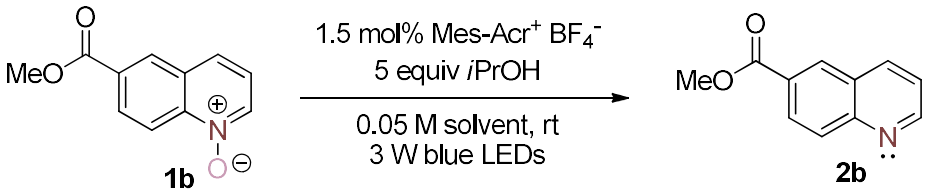
Table S1. Catalyst Screening[a]



Entry	Photocatalyst (mol%)	Yield (%)
1[b]	eosin Y disodium salt (1.0)	11
2[b]	eosin Y disodium salt (3.0)	21
3[b]	eosin Y (1.0)	10
4[b]	eosin Y (3.0)	22
5[b]	rosebengal (1.0)	trace
6[b]	rosebengal (3.0)	6
7[b]	rosebengal (5.0)	10
8[b]	rhodamine B (1.0)	trace
9[b]	rhodamine 6G (1.0)	trace
10	9,10-dicyanoanthracene (1.0)	trace
11	fluorescein (1.0)	trace
12	4CzIPN (1.0) **(3)**	50
13	2,4,6-triphenylpyrylium BF_4^- (1.0) **(4)**	30
14	Mes-Acr^+ BF_4^- (1.0) **(5)**	63
15	Mes-Acr^+ ClO_4^- (1.0) **(6)**	60
16	9-mesityl-10-phenylacridinium$^+$ BF_4^- (1.0)	57

[a]Conditions: **1b** (0.20 mmol), IPA (0.08 mL), and a photocatalyst (1.0–5.0 mol%) in degassed acetonitrile (4 mL, 0.05 M). [b]Under the irradiation of green LEDs.

Table S2. Solvent Screening[a]

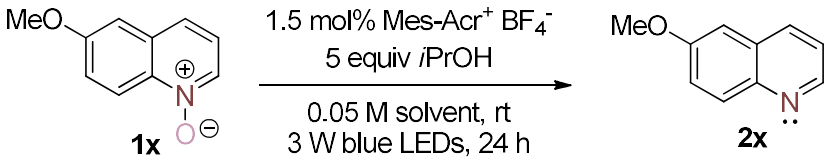


Entry	Solvent	Time (h)	Yield (%)
1	DCM	24	34
2	DCE	24	33
3	MeOH	20	45
4	EtOH	22	40
5	BuOH	23	41
6	toluene	18	82
7	xylene	20	63
8	THF	10	41
9	DMA	23	47
10	DMSO	14	53
11	DMF	24	49
12	acetonitrile	24	63
13	acetone	16	78
14	1,4-dioxane	4.5	58
15	2-butanone	22	56
16	toluene/acetonitrile (7/1)	13	88
17	toluene/DCM (40/1)	13	84
18	toluene/THF (3/1)	12	80
19	toluene/acetone (3/1)	14	83
20	toluene/DMSO (5/1)	24	76
21	toluene/anisole (20/1)	13	81
22	toluene/butanone (3/1)	12	86
23	toluene/acetonitrile (10/1)	13	79
24	toluene/acetonitrile (4/1)	19	84

25	toluene/acetonitrile (1/1)	24	80

[a]Conditions: **1b** (0.20 mmol), IPA (0.08 mL), and a photocatalyst (1.5 mol%) in degassed solvent (4 mL, 0.05 M).

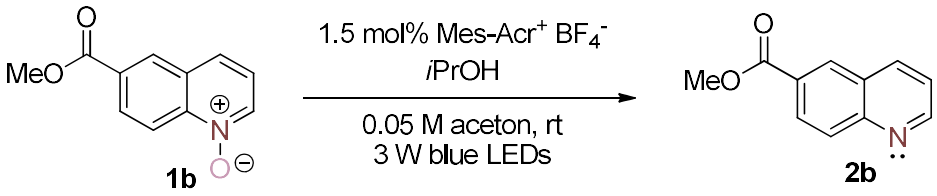
Table S3. Further Solvent Screening[a]



Entry	solvent	Yield (%)
1	toluene/acetonitrile (7/1)	43
2	acetone	54
3	2-butanone	42
4	acetonitrile	33
5	1,4-dioxane	32
6	DCM	34
7	MeOH	39
8	toluene	53
9	THF	38
10	DMF	36

[a]Conditions: **1x** (0.20 mmol), IPA (0.08 mL), and a photocatalyst (1.5 mol%) in degassed solvent (4 mL, 0.05 M).

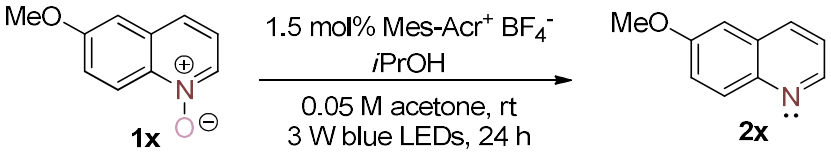
Table S4. Screening the Equivalent of IPA[a]



Entry	Equivalent of IPA	Time (h)	Yield (%)
1	1.1	24	43
2	20	10.5	72
3	40	9	74
4	60	8	77
5	75	7.5	76
6	120	6	66

[a]Conditions: **1b** (0.20 mmol), IPA (1.1–120 equivalent), and a photocatalyst (0.5-1.5 mol%) in degassed acetone (4 mL, 0.05 M).

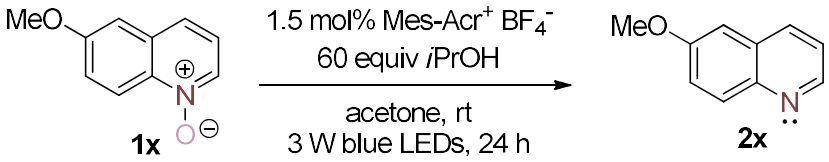
Table S5. Further Screening the Equivalent of IPA[a]



Entry	Equivalent of IPA	Yield (%)
1	1.1	14
2	10	31
3	30	44
4	40	50
5	50	57
6	60	64
7	75	55
8	90	52

[a]Conditions: **1x** (0.20 mmol), IPA (1.1-90 equivalent), and a photocatalyst (1.5 mol%) in degassed solvent (4 mL, 0.05 M).

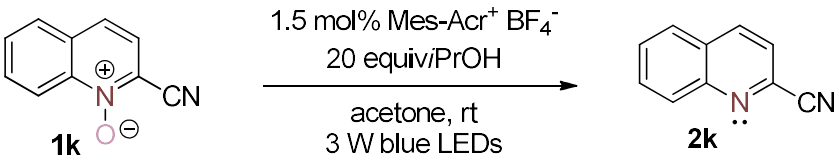
Table S6. Screening the Molarity of Acetone[a]



Entry	Molarity of Acetone	Yield (%)
1	0.3	7
2	0.2	18
3	0.1	33
4	0.07	64
5	0.05	63
6	0.03	58

[a]Conditions: **1x** (0.20 mmol), IPA (0.92 mL), and a photocatalyst (1.5 mol%) in degassed solvent (0.67–6.7 mL, 0.1-0.03 M).

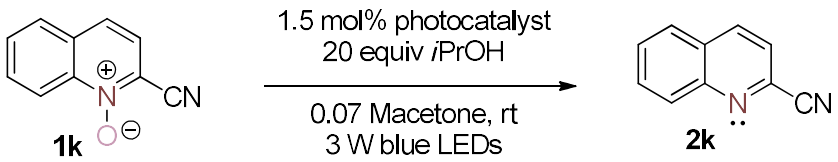
Table S7. Further Screening the Molarity of Acetone[a]



Entry	Molarity of Acetone	Time (h)	Yield (%)
1	0.1	5	73
2	0.07	5	83
`	0.05	6.5	77

[a]Conditions: **1k** (0.20 mmol), IPA (0.30 mL), and a photocatalyst (1.5 mol%) in degassed solvent (2-4 mL, 0.1-0.05 M).

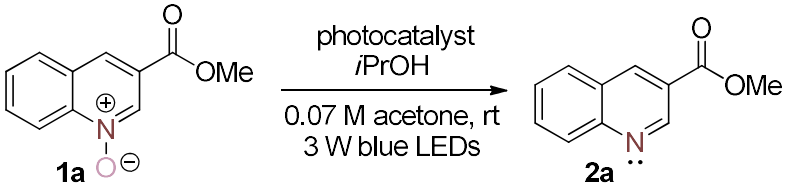
Table S8. Screening Mes-Acr$^+$ Derivatives as a Photocatalyst[a]



Entry	Photocatalyst (mol%)	Time (h)	Yield (%)
1	Mes-Acr$^+$ BF_4^- (1.5) **(5)**	6.5	83
2	Mes-Acr$^+$ ClO_4^- (1.5) **(6)**	7	81
3	9-mesityl-10-phenylacridinium$^+$ BF_4^- (1.5)	11	80
4	Mes-*t*Bu$_2$Acr$^+$ BF_4^- (1.5) **(7)**	5	90
5	10,10′-dimethyl-9,9′-biacridinium^{2+} 2 NO_3^- (1.5)	24	48
6	9-phenylacridine (1.5)	24	51
7	10-methyl-9-phenylacridinium$^+$ ClO_4^- (1.5)	22	73
8	2,7,10-trimethyl-9-(2,4,6-trimethylphenyl)acridinium$^+$ ClO_4^- (1.5)	16	86
9	9-(2,6-dimethylphenyl)-10-methylacridinium$^+$ ClO_4^- (1.5)	24	77
10	9-[1,1′-biphenyl]-2-yl-10-methylacridinium$^+$ ClO_4^- (1.5)	22	80

[a]Conditions: **1k** (0.20 mmol), IPA (0.30 mL), and a photocatalyst (1.5 mol%) in degassed solvent (2.9 mL, 0.07 M).

Table S9. Confirming the Optimised Conditions[a]



Entry	Photocatalyst (mol%)	Equivalent of IPA	Time (h)	Yield (%)
1[b]	eosin Y disodium salt (1.0)	30	24	Trace
2[b]	rosebengal (1.0)	30	24	Trace
3	4CzIPN (1.0) **(3)**	30	7	69
4	2,4,6-triphenylpyrylium BF_4^- (1.0) **(4)**	30	24	42
5	Mes-Acr^+ BF_4^- (1.0) **(5)**	30	8	78
6	Mes-Acr^+ ClO_4^- (1.0) **(6)**	30	8.5	77
7	Mes-*t*Bu_2Acr^+ BF_4^- (1.0) **(7)**	30	4	79
8	Mes-*t*Bu_2Acr^+ BF_4^- (1.0) **(7)**	1.1	22	43
9	Mes-*t*Bu_2Acr^+ BF_4^- (1.0) **(7)**	15	5	76
10	Mes-*t*Bu_2Acr^+ BF_4^- (1.0) **(7)**	4	3	85
11	Mes-*t*Bu_2Acr^+ BF_4^- (1.0) **(7)**	60	3	89
12	Mes-*t*Bu_2Acr^+ BF_4^- (1.0) **(7)**	75	2.5	80
13	Mes-*t*Bu_2Acr^+ BF_4^- (0.5) **(7)**	60	3.5	85
14	Mes-*t*Bu_2Acr^+ BF_4^- (1.5) **(7)**	60	2.5	92

[a]Conditions: **1a** (0.20 mmol), IPA (1.1–75 equivalent), and a photocatalyst (0.5–1.5 mol%) in degassed solvent (2.9 mL, 0.07 M).
[b]Under the irradiation of green LEDs.

General Procedure for the Optimisation Studies (Table 1)

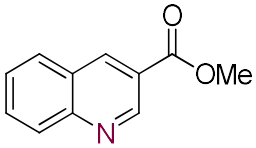
In a 10-mL test tube, equipped with a small magnetic stir bar, methyl quinoline-3-carboxylate *N*-oxide (**1a**, 40.6 mg, 0.200 mmol, 1.00 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 1.7 mg, 3.0 μmol, 1.5 mol%), and isopropanol (IPA, 0.92 mL) were combined. The test tube was fitted with a rubber septum, and acetone (2.90 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 5 minutes, the reaction mixture was irradiated with two 3 W blue LEDs at room temperature for the indicated time (see Table 1). Upon completion of the reaction as determined by TLC, the reaction mixture was concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography on silica gel (hexanes/EtOAc = 3:1) to afford methyl quinoline-3-carboxylate (**2a**) as a white solid.

General Procedure for the Organic Photoredox-Catalysed Deoxygenation Reaction of N-Heterocyclic *N*-Oxide Using Isopropyl Alcohol as a Sustainable Reducing Agent (Table 2)

In a 10-mL test tube, equipped with a small magnetic stir bar, N-heterocyclic *N*-oxide (**1**, 0.200 mmol, 1.00 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 1.7 mg, 3.0 µmol, 1.5 mol%), and IPA (0.92 mL) were combined. The test tube was fitted with a rubber septum, and acetone (2.9 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 5 minutes, the reaction mixture was irradiated with two 3 W blue LEDs at room temperature for the indicated time (Table 2). Upon completion of the reaction as determined by TLC, the reaction mixture was concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography on silica gel (hexanes and EtOAc) to afford the corresponding deoxygenated N-heterocycle (**2**). For N-heterocyclic *N*-oxides **1n**, **1o**, **1p**, **1t**, **1u**, **1v**, **1w**, **1x** and **1ao**, a photoreactor equipped with four 3 W blue LEDs were used under otherwise identical conditions.

Characterisation Data for the Deoxygenated N-Heterocycles (2)

Preparation of Methyl Quinoline-3-carboxylate (2a)



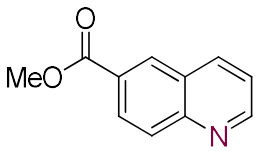
Prepared using methyl quinoline-3-carboxylate *N*-oxide (**1a**, 40.6 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 µmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). Methyl quinoline-3-carboxylate (**2a**, 34.5 mg, 92%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.4 (m, 1H), 8.8 (d, *J* = 2.3 Hz, 1H), 8.2 (d, *J* = 8.4 Hz, 1H), 7.9 (d, *J* = 8.2 Hz, 1H), 7.8 (t, *J* = 7.3 Hz, 1H), 7.6 (t, *J* = 7.5 Hz, 1H), 4.0 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 165.6, 149.6, 149.3, 139.0, 132.0, 129.1, 129.0, 127.5, 126.8, 122.9, 52.4.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{11}H_9NO_2$) 187.1, found 187.1.

Preparation of Methyl Quinoline-6-carboxylate (2b)



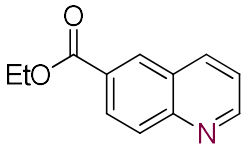
Prepared using methyl quinoline-6-carboxylate *N*-oxide (**1b**, 40.6 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). Methyl quinoline-6-carboxylate (**2b**, 30.1 mg, 80%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.9 (dd, *J* = 4.3, 1.8 Hz, 1H), 8.5 (d, *J* = 2.0 Hz, 1H), 8.2 (dd, *J* = 8.9, 2.0 Hz, 1H), 8.2 (dd, *J* = 8.2, 0.9 Hz, 1H), 8.1 (d, *J* = 8.7 Hz, 1H), 7.4 (dd, *J* = 8.2, 4.3 Hz, 1H), 3.9 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 166.5, 152.5, 150.1, 137.3, 131.0, 129.8, 128.9, 128.1, 127.4, 121.8, 52.4.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{11}H_9NO_2$) 187.1, found 187.1.

Preparation of Ethyl Quinoline-6-carboxylate (2c)



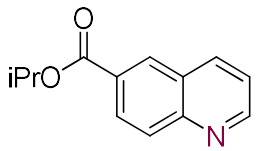
Prepared using ethyl quinoline-6-carboxylate *N*-oxide (**1c**, 43.4 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). Ethyl quinoline-6-carboxylate (**2c**, 32.7 mg, 81%) was isolated (hexanes/EtOAc = 1.5:1) as a pale-yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.0 (dd, *J* = 4.3, 1.8 Hz, 1H), 8.6 (d, *J* = 2.1 Hz, 1H), 8.3 (dd, *J* = 8.9, 2.0 Hz, 1H), 8.3 (dd, *J* = 8.3, 1.9 Hz, 1H), 8.1 (d, *J* = 8.9 Hz, 1H), 7.5 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.5 (q, *J* = 7.1 Hz, 2H), 1.5 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 166.1, 152.4, 150.0, 137.3, 130.9, 129.7, 129.0, 128.5, 127.4, 121.8, 61.4, 14.4.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{12}H_{11}NO_2$) 201.1, found 201.1.

Preparation of Isopropyl Quinoline-6-carboxylate (2d)



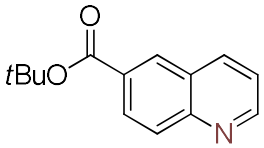
Prepared using isopropyl quinoline-6-carboxylate *N*-oxide (**1d**, 46.2 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). Isopropyl quinoline-6-carboxylate (**2d**, 33.2 mg, 74%) was isolated (hexanes/EtOAc = 1.5:1) as a brown solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.0 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.6 (d, *J* = 2.0 Hz, 1H), 8.3 (dd, *J* = 8.8, 1.9 Hz, 1H), 8.2 (dd, *J* = 8.2, 2.4 Hz, 1H), 8.1 (d, *J* = 8.9 Hz, 1H), 5.3 (hept, *J* = 6.3 Hz, 1H), 1.4 (d, *J* = 6.3 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 165.7, 152.5, 150.1, 137.4, 130.9, 129.8, 129.1, 129.0, 127.5, 121.9, 69.0, 22.1.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{13}H_{13}NO_2$) 215.1, found 215.2.

Preparation of *tert*-Butyl Quinoline-6-carboxylate (2e)



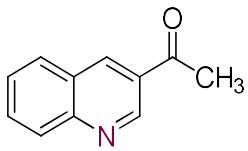
Prepared using *tert*-butyl quinoline-6-carboxylate (**1e**, 49.1 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). *tert*-Butyl quinoline-6-carboxylate (**2e**, 36.7 mg, 80%) was isolated (hexanes/EtOAc = 2:1) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.97 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.50 (d, *J* = 2.0 Hz, 1H), 8.24 (ddd, *J* = 9.2, 4.1, 2.2 Hz, 2H), 8.10 (d, *J* = 8.8 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.3 Hz, 1H), 1.63 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.3, 152.3, 150.0, 137.4, 130.7, 130.1, 129.6, 129.2, 127.5, 121.8, 81.7, 28.3.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{14}H_{15}NO_2$) 229.1, found 229.2.

Preparation of 3-Acetylquinoline (2f)



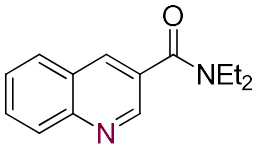
Prepared using 3-acetylquinoline *N*-oxide (**1f**, 37.4 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 3-Acetylquinoline (**2f**, 29.9 mg, 87%) was isolated (hexanes/EtOAc = 1:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.4 (d, *J* = 2.0 Hz, 1H), 8.7 (d, *J* = 2.4 Hz, 1H), 8.1 (d, *J* = 8.4 Hz, 1H), 7.9 (d, *J* = 8.2 Hz, 1H), 7.8 (ddd, *J* = 8.4, 6.8, 1.4 Hz, 1H), 7.6 (t, *J* = 7.5 Hz, 1H), 2.7 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 196.8, 149.8, 149.2, 137.5, 132.2, 129.5, 129.5, 129.3, 127.7, 126.9, 26.9.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{11}H_9NO$) 171.1, found 171.1.

Preparation of *N,N*-Diethyl-3-quinolinecarboxamide (2g)



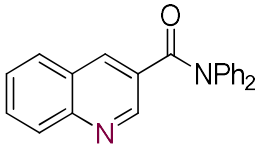
Prepared using *N,N*-diethyl-3-quinolinecarboxamide *N*-oxide (**1g**, 48.9 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). *N,N*-Diethyl-3-quinolinecarboxamide (**2g**, 41.1 mg, 89%) was isolated (hexanes/EtOAc = 1:3) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.90 (s, 1H), 8.16 (s, 1H), 8.09 (d, *J* = 8.5 Hz, 1H), 7.81 (d, *J* = 8.2 Hz, 1H), 7.76–7.69 (m, 1H), 7.59–7.51 (m, 1H), 3.57 (br s, 2H), 3.29 (br s, 2H), 1.24 (br s, *J* = 26.9 Hz, 3H), 1.12 (br s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.7, 148.1, 148.0, 134.1, 130.5, 130.2, 129.4, 128.2, 127.4, 127.1, 43.6, 39.7, 14.4, 13.0.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{14}H_{16}N_2O$) 228.1, found 228.1.

Preparation of *N,N*-Diphenyl-3-quinolinecarboxamide (2h)



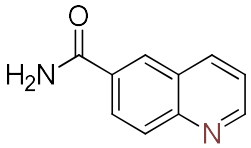
Prepared using *N,N*-diphenyl-3-quinolinecarboxamide *N*-oxide (**1h**, 68.1 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). *N,N*-Diphenyl-3-quinolinecarboxamide (**2h**, 62.9 mg, 97%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.89 (d, *J* = 2.2 Hz, 1H), 8.37 (d, *J* = 2.2 Hz, 1H), 8.04 (d, *J* = 8.4 Hz, 1H), 7.78–7.70 (m, 3H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.32 (t, *J* = 7.7 Hz, 5H), 7.27–7.21 (m, 8H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 168.2, 149.6, 148.1, 143.3, 137.8, 131.0, 129.4, 129.3, 129.2, 128.6, 127.5, 127.3, 127.0, 126.8.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{22}H_{16}N_2O$) 324.1, found 324.1.

Preparation of 6-Quinolinecarboxamide (2i)



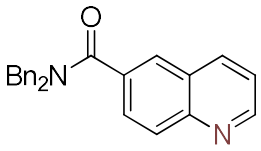
Prepared using 6-quinolinecarboxamide *N*-oxide (**1i**, 37.6 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 6-Quinolinecarboxamide (**2i**, 28.1 mg, 82%) was isolated (hexanes/acetone = 1:3) as a white solid.

^{1}H NMR (300 MHz, MeOD) δ 8.87 (dd, *J* = 4.4, 1.7 Hz, 1H), 8.41 (dd, *J* = 17.2, 1.9 Hz, 2H), 8.16 (dd, *J* = 8.9, 2.1 Hz, 2H), 8.02 (d, *J* = 8.9 Hz, 1H), 7.54 (dd, *J* = 8.3, 4.3 Hz, 2H).

^{13}C NMR (75 MHz, MeOD) δ 171.3, 153.0, 149.9, 139.4, 133.4, 129.7, 129.5, 129.3, 129.0, 123.3.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{10}H_8N_2O$) 172.1, found 172.1.

Preparation of *N*,*N*-Dibenzyl-6-quinolinecarboxamide (2j)



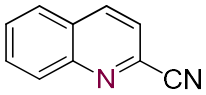
Prepared using *N*,*N*-dibenzyl-6-quinolinecarboxamide *N*-oxide (**1j**, 73.7 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). *N*,*N*-Dibenzyl-6-quinolinecarboxamide (**2j**, 63.5 mg, 90%) was isolated (hexanes/EtOAc = 1:3) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.93 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.12 (d, *J* = 8.4 Hz, 2), 7.97 (d, *J* = 2.0 Hz, 1H), 7.82 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.44–7.29 (m, 9H), 7.15 (d, *J* = 7.9 Hz, 2H), 4.77 (s, 2H), 4.45 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.6, 151.6, 148.4, 136.8, 136.5, 136.3, 134.4, 130.1, 128.9, 128.6, 127.8, 127.7, 127.0, 126.6, 121.9, 51.7, 47.2.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{24}H_{20}N_2O$) 352.2, found 352.2.

Preparation of 2-Quinolinecarbonitrile (2k)



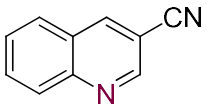
Prepared using 2-quinolinecarbonitrile *N*-oxide (**1k**, 34.0 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.30 mL), and acetone (2.9 mL, 0.07 M). 2-Quinolinecarbonitrile (**2k**, 28.4 mg, 90%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (d, *J* = 8.4 Hz, 1H), 8.18 (d, *J* = 8.5 Hz, 1H), 7.92 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.86 (ddd, *J* = 8.5, 6.9, 1.5 Hz, 1H), 7.73 (t, *J* = 7.6 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 148.3, 137.6, 133.7, 131.4, 130.1, 129.6, 128.8, 127.9, 123.4, 117.7.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{10}H_6N_2$) 154.1, found 154.1.

Preparation of 3-Quinolinecarbonitrile (2l)



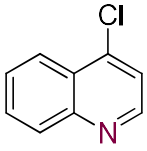
Prepared using 3-quinolinecarbonitrile *N*-oxide (**1l**, 34.0 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 µmol, 1.5 mol%), IPA (0.30 mL), and acetone (2.9 mL, 0.07 M). 3-Quinolinecarbonitrile (**2l**, 24.1 mg, 78%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.02 (d, *J* = 2.1 Hz, 1H), 8.52 (dd, *J* = 2.2, 0.8 Hz, 1H), 8.16 (d, *J* = 9.0 Hz, 1H), 7.93–7.84 (m, 2H), 7.68 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 149.8, 148.8, 141.6, 132.9, 129.9, 128.6, 128.3, 126.3, 117.2, 106.6.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{10}H_6N_2$) 154.1, found 154.1.

Preparation of 4-Chloroquinoline (2m)



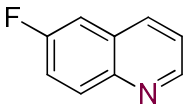
Prepared using 4-chloroquinoline *N*-oxide (**1m**, 35.9 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 µmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 4-Chloroquinoline (**2m**, 24.9 mg, 76%) was isolated (hexanes/EtOAc = 10:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.78 (d, *J* = 4.7 Hz, 1H), 8.22 (dd, *J* = 8.4, 1.5 Hz, 1H), 8.13 (dd, *J* = 8.5, 0.9 Hz, 1H), 7.77 (ddd, *J* = 8.4, 6.8, 1.5 Hz, 1H), 7.64 (ddd, *J* = 8.4, 6.8, 1.3 Hz, 1H), 7.48 (d, *J* = 4.7 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 149.9, 149.1, 142.9, 130.6, 129.9, 127.8, 126.6, 124.3, 121.4.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ (C_9H_6ClN) 163.0, found 163.1.

Preparation of 6-Fluoroquinoline (2n)



Prepared using 6-fluorooquinoline *N*-oxide (**1n**, 32.6 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 6-Fluoroquinoline (**2n**, 20.9 mg, 71%) was isolated (hexanes/EtOAc = 2:1) as a yellow oil.

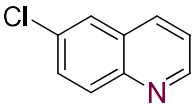
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.88 (dd, *J* = 4.4, 1.4 Hz, 1H), 8.11 (dd, *J* = 9.5, 5.3 Hz, 2H), 7.48 (td, *J* = 8.7, 2.7 Hz, 1H), 7.42 (ddd, *J* = 8.5, 5.3, 3.4 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 160.5 (d, $^{1}J_{C\text{-}F}$ = 248.0 Hz), 149.8 (d, $^{4}J_{C\text{-}F}$ = 2.9 Hz), 145.5, 135.5 (d, $^{4}J_{C\text{-}F}$ = 5.5 Hz), 132.1 (d, $^{3}J_{C\text{-}F}$ = 9.2 Hz), 129.0 (d, $^{3}J_{C\text{-}F}$ = 9.9 Hz), 121.9, 119.9 (d, $^{2}J_{C\text{-}F}$ = 25.7 Hz), 110.8 (d, $^{2}J_{C\text{-}F}$ = 21.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ –113.3.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ (C_9H_6FN) 147.0, found 147.1.

Preparation of 6-Chloroquinoline (2o)



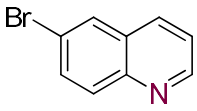
Prepared using 6-chloroquinoline *N*-oxide (**1o**, 35.9 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 6-Chloroquinoline (**2o**, 24.5 mg, 75%) was isolated (hexanes/EtOAc = 3:1) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.90 (dd, *J* = 4.3, 1.8 Hz, 1H), 8.05 (t, *J* = 9.9 Hz, 1H), 7.79 (d, *J* = 2.3 Hz, 1H), 7.64 (dd, *J* = 8.9, 2.4 Hz, 1H), 7.41 (dd, *J* = 8.4, 4.3 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 150.6, 146.6, 135.4, 132.5, 131.1, 130.6, 128.9, 126.5, 122.0.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ (C_9H_6ClN) 163.0, found 163.1.

Preparation of 6-Bromoquinoline (2p)



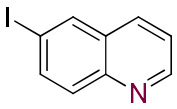
Prepared using 6-bromoquinoline *N*-oxide (**1p**, 44.8 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 6-Bromoquinoline (**2p**, 31.2 mg, 75%) was isolated (hexanes/EtOAc = 3:1) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.91 (dd, *J* = 4.3, 1.7 Hz, 1H), 8.06 (dd, *J* = 8.3, 1.9 Hz, 1H), 8.00–7.95 (m, 2H), 7.77 (dd, *J* = 9.0, 2.3 Hz, 1H), 7.41 (dd, *J* = 8.3, 4.2 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 150.7, 146.8, 135.3, 133.1, 131.2, 129.9, 129.5, 122.0, 120.6.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ (C_9H_6BrN) 207.0, found 207.1.

Preparation of 6-Iodoquinoline (2q)



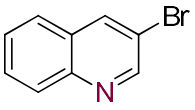
Prepared using 6-iodoquinoline *N*-oxide (**1q**, 54.2 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 6-Iodoquinoline (**2q**, 38.1 mg, 75%) was isolated (hexanes/EtOAc = 10:1) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.89 (d, *J* = 4.5 Hz, 1H), 8.17 (d, *J* = 2.3 Hz, 1H), 7.99 (d, *J* = 8.3 Hz, 1H), 7.95–7.88 (m, 3H), 7.81 (d, *J* = 8.9 Hz, 1H), 7.37 (dd, *J* = 8.3, 4.2 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 151.0, 147.3, 138.2, 136.6, 134.9, 131.3, 130.0, 121.8, 92.2.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ (C_9H_6IN) 255.0, found 254.9.

Preparation of 3-Bromoquinoline (2r)



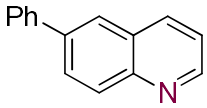
Prepared using 3-bromoquinoline *N*-oxide (**1r**, 44.8 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 3-Bromoquinoline (**2r**, 34.2 mg, 82%) was isolated (hexanes/EtOAc = 10:1) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.90 (d, *J* = 2.4 Hz, 1H), 8.30 (d, *J* = 2.4 Hz, 1H), 8.08 (d, *J* = 9.0 Hz, 1H), 7.72 (td, *J* = 6.1, 5.5, 2.9 Hz, 2H), 7.60–7.53 (m, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 151.4, 146.3, 137.4, 129.9, 129.5, 129.2, 127.7, 127.0, 117.2.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ (C_9H_6BrN) 207.0, found 207.1.

Preparation of 6-Phenylquinoline (2s)



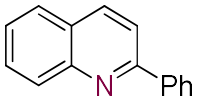
Prepared using 6-phenylquinoline *N*-oxide (**1s**, 44.2 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 6-Phenylquinoline (**2s**, 37.9 mg, 92%) was isolated (hexanes/EtOAc = 10:1) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.20 (s, 1H), 8.30 (s, 1H), 8.16 (d, *J* = 8.5 Hz, 1H), 7.87 (d, *J* = 8.1 Hz, 1H), 7.71 (d, *J* = 7.9 Hz, 3H), 7.61–7.48 (m, 3H), 7.44 (t, *J* = 7.4 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 149.9, 147.2, 137.8, 133.9, 133.3, 129.5, 129.2, 129.2, 128.2, 128.1, 128.0, 127.4, 127.1.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{15}H_{11}N$) 205.1, found 205.1.

Preparation of 2-Phenylquinoline (2t)



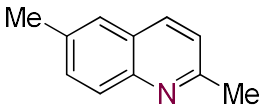
Prepared using 2-phenylquinoline *N*-oxide (**1t**, 44.2 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 2-Phenylquinoline (**2t**, 37.6 mg, 92%) was isolated (hexanes/DCM = 2.5:1) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.22 (s, 1H), 8.21–8.17 (m, 7H), 7.88 (d, *J* = 8.5 Hz, 2H), 7.83 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.74 (ddd, *J* = 8.4, 6.9, 1.6 Hz, 2H), 7.58–7.51 (m, 6H), 7.51–7.44 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.5, 148.4, 139.8, 136.9, 129.9, 129.8, 129.4, 129.0, 127.7, 127.6, 127.3, 126.4, 119.1.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{15}H_{11}N$) 205.1, found 205.1.

Preparation of 2,6-Dimethylquinoline (2u)



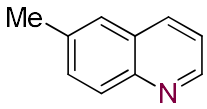
Prepared using 2,6-dimethylquinoline *N*-oxide (**1u**, 34.6 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 2,6-Dimethylquinoline (**2u**, 24.0 mg, 76%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (dd, *J* = 12.8, 8.4 Hz, 2H), 7.54–7.46 (m, 2H), 7.23 (d, *J* = 8.4 Hz, 1H), 2.72 (s, 3H), 2.50 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 158.0, 146.4, 135.8, 135.6, 131.8, 128.3, 126.6, 126.5, 122.1, 25.3, 21.6.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{11}H_{11}N$) 157.1, found 157.2.

Preparation of 6-Methylquinoline (2v)



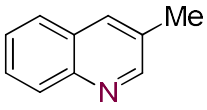
Prepared using 6-methylquinoline *N*-oxide (**1v**, 31.8 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 6-Methylquinoline (**2v**, 22.3 mg, 78%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59 (d, *J* = 8.9 Hz, 1H), 8.41 (dd, *J* = 6.0, 1.1 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.57 (s, 1H), 7.53 (dd, *J* = 8.9, 2.0 Hz, 1H), 7.20 (dd, *J* = 8.5, 6.0 Hz, 1H), 2.49 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 140.1, 139.0, 134.9, 132.6, 130.6, 127.0, 125.4, 120.9, 119.5, 21.4.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{10}H_9N$) 143.1, found 143.2.

Preparation of 3-Methylquinoline (2w)



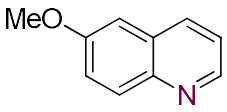
Prepared using 3-methylquinoline *N*-oxide (**1w**, 31.8 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 3-Methylquinoline (**2w**, 21.4 mg, 75%) was isolated (hexanes/EtOAc = 5:1) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.76 (d, *J* = 2.4 Hz, 1H), 8.07 (d, *J* = 8.5 Hz, 1H), 7.92–7.88 (m, 1H), 7.73 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.64 (ddd, *J* = 8.4, 6.8, 1.6 Hz, 1H), 7.50 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 2.50 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 152.4, 146.5, 134.9, 130.6, 129.2, 128.6, 128.2, 127.2, 126.7, 18.9.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{10}H_9N$) 143.1, found 143.2.

Preparation of 6-Methoxyquinoline (2x)



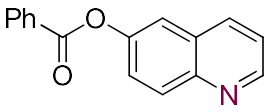
Prepared using 6-methoxyquinoline *N*-oxide (**1x**, 35.0 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 6-Methoxyquinoline (**2x**, 25.1 mg, 79%) was isolated (hexanes/EtOAc = 10:1) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.76 (dd, *J* = 4.4, 1.5 Hz, 1H), 8.09–7.98 (m, 2H), 7.36 (ddd, *J* = 11.3, 8.8, 3.6 Hz, 2H), 7.06 (d, *J* = 2.7 Hz, 1H), 3.92 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.9, 147.8, 144.3, 135.1, 130.8, 129.4, 122.5, 121.5, 105.2, 55.7.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{10}H_9NO$) 159.1, found 159.1.

Preparation of 6-Benzoyloxyquinoline (2y)



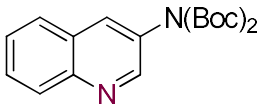
Prepared using 6-bezoyloxyquinoline *N*-oxide (**1y**, 53.0 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 6-Benzoyloxyquinoline (**2y**, 37.6 mg, 76%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.94 (dd, *J* = 4.3, 1.8 Hz, 1H), 8.27 (m, *J* = 7.0, 1.4 Hz, 2H), 8.20 (d, *J* = 9.0 Hz, 1H), 8.15 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.71 (d, *J* = 2.6 Hz, 1H), 7.67 (tt, *J* = 7.0, 1.4 Hz, 1H), 7.61 (dd, *J* = 9.2, 2.6 Hz, 1H), 7.55 (t, *J* = 7.9 Hz, 2H), 7.43 (dd, *J* = 8.2, 4.3 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 165.2, 150.3, 148.9, 146.5, 135.9, 133.9, 131.2, 130.3, 129.4, 128.8, 128.7, 124.9, 121.7, 118.7.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{16}H_{11}NO_2$) 249.1, found 249.1.

Preparation of 3-[Bis(*tert*-butoxycarbonyl)amino]quinoline (2z)



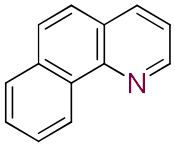
Prepared using 3-[bis(*tert*-butoxycarbonyl)amino]quinoline *N*-oxide (**1z**, 72.1 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 3-[Bis(*tert*-butoxycarbonyl)amino]quinoline (**2z**, 63.7 mg, 93%) was isolated (hexanes/EtOAc = 4:1) as a yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.68 (d, *J* = 2.4 Hz, 1H), 8.09 (d, *J* = 8.4 Hz, 1H), 7.91 (d, *J* = 2.4 Hz, 1H), 7.79 (dd, *J* = 7.5, 1.9 Hz, 1H), 7.70 (tt, *J* = 6.9, 1.7 Hz, 1H), 7.54 (tt, *J* = 6.9, 1.7 Hz, 1H), 1.39 (s, 18H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 151.3, 150.6, 146.8, 133.6, 132.9, 129.8, 129.3, 127.9, 127.8, 127.1, 83.6, 28.0.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{19}H_{24}N_2O_4$) 344.2, found 344.1.

Preparation of Benzo[*h*]quinoline (2aa)



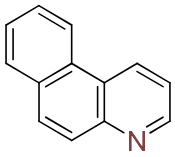
Prepared using benzo[*h*]quinoline *N*-oxide (**1aa**, 39.0 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). Benzo[*h*]quinoline (**2aa**, 29.1 mg, 81%) was isolated (hexanes/EtOAc = 5:1) as a pale yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.32 (dd, *J* = 8.3, 1.1 Hz, 1H), 9.01 (dd, *J* = 4.4, 1.8 Hz, 1H), 8.15 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.91 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.85–7.64 (m, 4H), 7.51 (dd, *J* = 8.1, 4.4 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 148.8, 146.6, 135.8, 133.6, 131.5, 128.2, 127.8, 127.8, 127.1, 126.4, 125.4, 124.4, 121.8.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{13}H_9N$) 179.1, found 179.2.

Preparation of Benzo[*f*]quinoline (2ab)



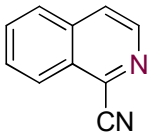
Prepared using benzo[*f*]quinoline *N*-oxide (**1ab**, 39.0 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 µmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). Benzo[*f*]quinoline (**2ab**, 25.9 mg, 72%) was isolated (hexanes/EtOAc = 5:1) as a pale yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.96 (dd, *J* = 4.4, 1.6 Hz, 1H), 8.92 (dd, *J* = 8.4, 1.9 Hz, 1H), 8.60 (d, *J* = 7.9 Hz, 1H), 8.03–7.95 (m, 2H), 7.93 (dd, *J* = 7.4, 2.0 Hz, 1H), 7.73–7.60 (m, 2H), 7.54 (dd, *J* = 8.4, 4.4 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 149.8, 148.3, 131.8, 131.0, 130.8, 129.7, 128.8, 128.3, 127.4, 127.2, 125.5, 122.7, 121.4.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{13}H_9N$) 179.1, found 179.2.

Preparation of 1-Isoquinolinecarbonitrile (2ac)



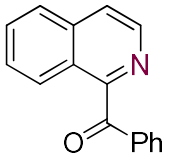
Prepared using 1-isoquinolinecarbonitrile *N*-oxide (**1ac**, 34.0 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 µmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 1-Isoquinolinecarbonitrile (**2ac**, 26.0 mg, 84%) was isolated (hexanes/acetone = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.63 (d, *J* = 5.5 Hz, 1H), 8.33–8.28 (m, 1H), 7.94 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.89 (d, *J* = 5.5 Hz, 1H), 7.80 (dddd, *J* = 18.6, 8.2, 6.9, 1.4 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 143.3, 135.9, 134.8, 131.8, 129.9, 129.3, 127.3, 125.3, 124.5, 115.8.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{10}H_6N_2$) 154.1, found 154.1.

Preparation of 1-Benzoylisoquinoline (2ad)



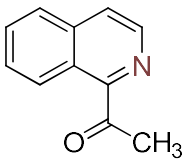
Prepared using 1-benzoylisoquinoline *N*-oxide (**1ad**, 49.9 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 1-Benzoylisoquinoline (**2ad**, 44.4 mg, 84%) was isolated (hexanes/EtOAc = 5:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.60 (d, *J* = 5.6 Hz, 1H), 8.22 (dd, *J* = 8.5, 1.0 Hz, 1H), 8.00–7.95 (m, 2H), 7.91 (d, *J* = 8.2 Hz, 1H), 7.80 (dd, *J* = 5.6, 0.8 Hz, 1H), 7.73 (ddd, *J* = 8.2, 6.9, 1.4 Hz, 1H), 7.60 (dddd, *J* = 8.9, 5.6, 3.1, 1.4 Hz, 2H), 7.50–7.43 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 194.8, 156.5, 141.2, 136.8, 136.7, 133.7, 130.8, 130.8, 128.5, 128.4, 127.2, 126.5, 126.2, 122.7.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{16}H_{11}NO$) 233.1, found 233.1.

Preparation of 1-Acetylisoquinoline (2ae)



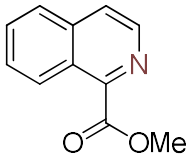
Prepared using 1-acetylisoquinoline *N*-oxide (**1ae**, 37.4 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 1-Acetylisoquinoline (**2ae**, 27.2 mg, 80%) was isolated (hexanes/acetone = 3:1) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.95 (d, *J* = 7.9 Hz, 1H), 8.57 (dd, *J* = 5.5, 1.9 Hz, 1H), 7.88–7.77 (m, 2H), 7.75–7.62 (m, 2H), 2.86 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 202.8, 152.9, 141.2, 137.2, 130.4, 129.2, 127.1, 127.0, 125.9, 124.7, 28.7.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{11}H_9NO$) 171.1, found 171.1.

Preparation of Methyl 1-Isoquinolinecarboxylate (2af)



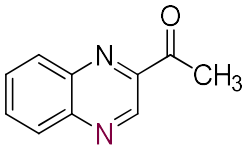
Prepared using Methyl 1-isoquinolinecarboxylate *N*-oxide (**1af**, 40.6 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). Methyl 1-isoquinolinecarboxyate (**2af**, 33.9 mg, 91%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.82 (d, *J* = 8.5 Hz, 1H), 8.61 (dd, *J* = 5.6, 1.6 Hz, 1H), 7.86 (d, *J* = 7.9 Hz, 1H), 7.80 (d, *J* = 5.8 Hz, 1H), 7.75–7.63 (m, 2H), 4.08 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.4, 148.3, 141.7, 137.0, 130.7, 128.9, 127.2, 127.0, 126.5, 124.4, 53.1.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{11}H_9NO_2$) 187.1, found 187.1.

Preparation of Methyl 2-quinoxalinecarboxylate (2ag)



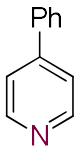
Prepared using methyl 2-quinoxalinecarboxylate *N*-oxide (**1ag**, 37.6 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). Methyl 2-quinoxalinecarboxylate (**2ag**, 25.0 mg, 73%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.47 (s, 1H), 8.16 (ddd, *J* = 13.1, 8.2, 1.8 Hz, 2H), 7.92–7.80 (m, 2H), 2.84 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 199.8, 146.6, 143.9, 143.0, 141.1, 132.2, 130.7, 130.5, 129.4, 25.5.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{10}H_8N_2O$) 172.1, found 172.1.

Preparation of 4-Phenylpyridine (2ah)



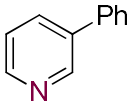
Prepared using 4-phenylpyridine *N*-oxide (**1ah**, 34.2 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 4-Phenylpyridine (**2ah**, 28.2 mg, 90%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.65 (dd, *J* = 4.4, 1.8 Hz, 2H), 7.67–7.59 (m, 2H), 7.55–7.38 (m, 5H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 150.3, 148.3, 138.1, 129.1, 129.1, 127.0, 121.6.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{11}H_9N$) 155.1, found 155.2.

Preparation of 3-Phenylpyridine (2ai)



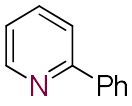
Prepared using 3-phenylpyridine *N*-oxide (**1ai**, 34.2 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 3-Phenylpyridine (**2ai**, 18.8 mg, 61%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.85 (dd, *J* = 2.4, 0.9 Hz, 1H), 8.59 (dd, *J* = 4.8, 1.6 Hz, 1H), 7.86 (ddd, *J* = 7.9, 2.4, 1.6 Hz, 1H), 7.61–7.53 (m, 2H), 7.47 (ddd, *J* = 7.5, 6.7, 1.3 Hz, 2H), 7.43–7.37 (m, 1H), 7.35 (ddd, *J* = 7.9, 4.8, 0.9 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 148.5, 148.4, 137.8, 136.6, 134.3, 129.1, 128.1, 127.2, 123.5.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{11}H_9N$) 155.1, found 155.2.

Preparation of 2-Phenylpyridine (2aj)



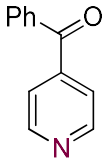
Prepared using 2-phenylpyridine *N*-oxide (**1aj**, 34.2 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 2-Phenylpyridine (**2aj**, 23.0 mg, 74%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.70 (dt, *J* = 4.9, 1.5 Hz, 1H), 8.02–7.96 (m, 2H), 7.79–7.70 (m, 2H), 7.48 (td, *J* = 6.6, 6.1, 1.7 Hz, 1H), 7.45–7.39 (m, 1H), 7.23 (td, *J* = 4.7, 2.3 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 157.6, 149.8, 139.5, 136.9, 129.1, 128.9, 127.0, 122.2, 120.7.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{11}H_9N$) 155.1, found 155.2.

Preparation of 4-Benzoylpyridine (2ak)



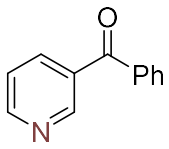
Prepared using 4-benzoylpyridine *N*-oxide (**1ak**, 39.8 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 4-Benzoylpyridine (**2ak**, 33.0 mg, 74%) was isolated (hexanes/EtOAc = 3:1) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.83–8.75 (m, 2H), 7.83–7.75 (m, 2H), 7.62 (tt, *J* = 7.0, 1.4 Hz, 1H), 7.59–7.53 (m, 2H), 7.53–7.45 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 195.2, 150.4 (two carbons), 144.4, 136.0, 133.6, 130.2 (two carbons), 128.7 (two carbons), 122.9 (two carbons).

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{12}H_9NO$) 183.1, found 183.1.

Preparation of 3-Benzoylpyridine (2al)



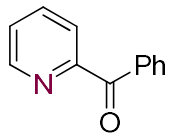
Prepared using 3-benzoylpyridine *N*-oxide (**1al**, 39.8 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 3-Benzoylpyridine (**2al**, 19.9 mg, 54 %) was isolated (hexanes/EtOAc = 3:1) as a brown solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.98 (dd, *J* = 2.2, 1.0 Hz, 1H), 8.80 (dd, *J* = 4.9, 1.8 Hz, 1H), 8.11 (dt, *J* = 7.8, 1.9 Hz, 1H), 7.85–7.78 (m, 2H), 7.68–7.59 (m, 1H), 7.56–7.48 (m, 2H), 7.44 (ddd, *J* = 7.9, 4.9, 1.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 195.0, 152.9, 151.1, 137.3, 136.8, 133.3, 130.1, 128.7, 123.5.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{12}H_9NO$) 183.1, found 183.1.

Preparation of 2-Benzoylpyridine (2am)



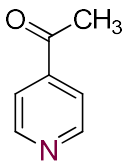
Prepared using 2-benzoylpyridine *N*-oxide (**1am**, 39.8 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 µmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 2-Benzoylpyridine (**2am**, 30.3 mg, 74%) was isolated (hexanes/DCM = 5:4) as a yellow solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.72 (d, *J* = 4.9 Hz, 1H), 8.10–8.01 (m, 3H), 7.89 (tt, *J* = 7.8, 2.4 Hz, 1H), 7.59 (td, *J* = 7.3, 1.2 Hz, 1H), 7.53–7.42 (m, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 193.9, 155.1, 148.6, 137.1, 136.3, 132.9, 131.0 (two carbons), 128.2 (two carbons), 126.2, 124.6.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ ($C_{12}H_9NO$) 183.1, found 183.1.

Preparation of 4-Acetylpyridine (2an)



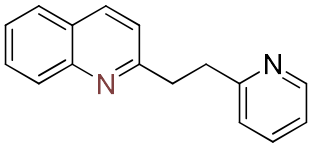
Prepared using 4-acetylpyridine *N*-oxide (**1an**, 27.4 mg, 0.200 mmol, 1.0 equivalent), **7** (1.7 mg, 3.0 µmol, 1.5 mol%), IPA (0.92 mL), and acetone (2.9 mL, 0.07 M). 4-Acetylpyridine (**2an**, 19.3 mg, 80%) was isolated (hexanes/EtOAc = 2:1) as a brown oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.78 (d, *J* = 6.3 Hz, 2H), 7.70 (d, *J* = 6.3 Hz, 2H), 2.60 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 197.4, 151.0 (two carbons), 142.8, 121.3 (two carbons), 26.7.

GC-MS (EI, 70 eV) *m/z* calculated for $[M]^+$ (C_7H_7NO) 121.1, found 121.1.

Preparation of 2-{2-(*N*-Oxidopyridine-2-yl)ethyl}quinoline (1ao')



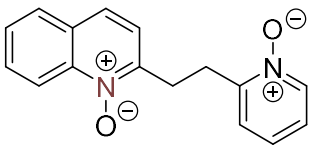
This compound was prepared following a literature procedure.[13] In a 60-mL thick-walled pressure vessel containing a magnetic stir bar, quinaldine (430 mg, 3.00 mmol, 1.0 equivalent), 2-pyridinemethanol (655 mg, 6.00 mmol, 2.0 equivalent), cesium hydroxide monohydrate (1.51 g, 9.00 mmol, 3.0 equivalent), and xylenes (3.0 mL) was combined in the air. The vessel was then fitted with a PTFE cap and immersed into a pre-heated oil bath (bath temperature = 160 °C). After stirring for 3 days, while heating the reaction mixture, the vessel was occasionally open to the air. The reaction mixture was then allowed to cool to room temperature. The reaction mixture was dissolved in a mixture of MeOH and CH_2Cl_2 (1:1, v/v) and poured into water. The mixture was extracted with CH_2Cl_2 (3 × 10 mL), and the combined organic layer was washed with brine (15 mL), dried over anhydrous sodium sulfate, filtered and concentrated on a rotary evaporator. Repetitive purification of the resulting residue by flash column chromatography (hexanes/EtOAc = 3:1 to neat EtOAc to hexanes/acetone = 9:1 to 15:1) on silica gel gave the title compound **1ao'** (91 mg, 13%) as a brown solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61–8.52 (m, 1H), 8.06 (dd, *J* = 12.2, 8.5 Hz, 2H), 7.78 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.70 (ddd, *J* = 8.4, 6.9, 1.6 Hz, 1H), 7.60–7.45 (m, 2H), 7.34–7.26 (m, 1H), 7.21–7.08 (m, 2H), 3.50–3.41 (m, 2H), 3.41–3.31 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 161.8, 161.2, 149.4, 148.1, 136.4, 136.3, 129.5, 129.0, 127.6, 126.9, 125.9, 123.2, 121.7, 121.3, 38.9, 38.1.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{16}H_{15}N_2$) 235.1, found 235.1.

Preparation of 2-{2-(*N*-Oxidopyridine-2-yl)ethyl)}quinoline *N*-oxide (1ao)



This compound was prepared following a literature procedure.[13] To a stirred solution of diheteroarylethane **1ao'** (342 mg, 0.86 mmol, 1.0 equivalent) in CH_2Cl_2 (2.3 mL) at 0 °C (ice-water bath),

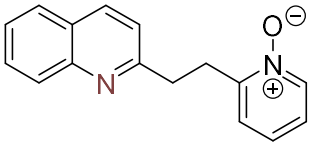
a solution of *m*-CPBA (>99%, 504 mg, 2.92 mmol, 2.0 equivalent) in CH_2Cl_2 (3.5 mL) was added dropwise via cannula over 30 minutes. The reaction mixture was then allowed to slowly warm to room temperature over 30 minutes. After stirring for 2 hours at the same temperature, another solution of *m*-CPBA (>99%, 252 mg, 1.46 mmol, 1.0 equivalent) in CH_2Cl_2 (1.5 mL) was added dropwise to the reaction mixture via cannula. The reaction mixture was then heated under reflux for 24 hours. After cooling to room temperature, the reaction mixture was poured into water and extracted with CH_2Cl_2 (3 × 10 mL). The combined organic layer was successively washed with a saturated solution of $NaHCO_3$ (15 mL), water (15 mL), and brine (15 mL). It was dried over anhydrous Na_2SO_4, filtered, and concentrated on a rotary evaporator. The crude mixture obtained was purified by flash column chromatography (EtOAc/MeOH = 4:1 to 2:1) on silica gel, yielding the title compound (**1ao**, 275 mg, 70%) as a brown solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, *J* = 8.8 Hz, 1H), 8.24 (dd, *J* = 6.0, 1.8 Hz, 1H), 7.82–7.67 (m, 2H), 7.57 (ddd, *J* = 8.2, 6.6, 5.3 Hz, 2H), 7.33–7.20 (m, 2H), 7.17–7.05 (m, 2H), 3.61–3.51 (m, 2H), 3.51–3.40 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 150.9, 147.4, 141.6, 139.6, 130.4, 129.3, 128.1, 128.0, 126.3, 125.8, 125.3, 124.0, 122.4, 119.5, 28.3, 27.2.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{16}H_{15}N_2O_2$) 267.1, found 267.1.

Preparation of 2-{2-(2-Quinolinyl)ethyl}pyridine *N*-oxide (2ao)



In a 10-mL test tube, equipped with a small magnetic stir bar, 2-{2-(2-quinolinyl)ethyl}pyridine *N*-oxide (**1ao**, 53.3 mg, 0.200 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 1.7 mg, 3.0 μmol, 1.5 mol%), and IPA (0.92 mL) were combined. The test tube was fitted with a rubber septum, and acetone (2.9 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 5 minutes, the reaction mixture was irradiated with two 3 W blue LEDs at room temperature for 51 hours. Afterward, the reaction mixture was concentrated on a rotary evaporator. The analysis of the crude reaction mixture by ^{1}H-NMR revealed the presence of a 5.5:1 mixture of mono-*N*-oxide **2ao** and diheteroarylethane **1ao'**. The crude

mixture was purified by flash column chromatography on silica gel (hexanes/EtOAc = 2:1), resulting in the isolation of 2-(2-(2-quinolinyl)ethyl)pyridine (**2ao**, 43 mg, 86%) as a brown oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (dd, *J* = 5.5, 2.3 Hz, 1H), 8.02 (d, *J* = 8.3 Hz, 2H), 7.75 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.66 (ddd, *J* = 8.4, 6.9, 1.6 Hz, 1H), 7.47 (ddd, *J* = 8.0, 6.8, 1.3 Hz, 1H), 7.33–7.27 (m, 1H), 7.24 (dd, *J* = 6.4, 3.5 Hz, 1H), 7.15–7.04 (m, 2H), 3.45 (tt, *J* = 7.8, 3.8 Hz, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 151.5, 147.8, 139.6, 136.4, 129.4, 128.8, 127.6, 126.8, 126.3, 125.9, 125.5, 123.7, 121.5, 34.7, 30.5.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{16}H_{15}N_2O$) 251.1, found 251.1.

Experimental Procedure for Mechanistic studies

Experimental Procedure for Scheme 1a (In the absence of a photocatalyst)

A 10-mL test tube, equipped with a small magnetic stir bar, was charged with 3-quinolinecarbonitrile *N*-oxide (**2l**, 34.0 mg, 0.200 mmol, 1.0 equivalent) and IPA (0.30 mL). The test tube was fitted with a rubber septum, and acetone (2.9 mL, 0.07 M) was added via a syringe under an atmosphere of nitrogen. After degassing by N_2 bubbling at room temperature for 5 minutes, the reaction mixture was irradiated with two 3 W blue LEDs at room temperature for 4 hours. TLC analysis indicated that no conversion of 1**l** into the deoxygenated product **2l** occurred.

Experimental Procedure for Scheme 1b (In the absence of visible light)

In a 10-mL test tube, equipped with a small magnetic stir bar, 6-iodoquinoline *N*-oxide (**1q**, 54.2 mg, 0.200 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 1.7 mg, 3.0 μmol, 1.5 mol%), and IPA (0.92 mL) were combined. The test tube was fitted with a rubber septum and covered with aluminum foil, and acetone (2.9 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 5 minutes, the reaction mixture was stirred in the dark for 3 hours. TLC analysis indicated that no conversion of **1q** into the deoxygenated product **2q** occurred.

Experimental Procedure for Quantum yield(Φ) measurement

(1) Photon-flux measurement:

The photon flux of the photoreactor and quantum yield of this reaction were determined by standard ferrioxalate actinometry[2] following a modified literature procedure.[3] A 0.15 M ferrioxalate solution was prepared by dissolving potassium ferrioxalate trihydrate (736.8 mg, 1.5 mmol) in 10 mL of 0.05 M H_2SO_4. A buffer solution was prepared by dissolving sodium acetate (2.25 g, 27.4 mmol) in 10 mL of 0.5 M H_2SO_4. Then, 4.0 mL of the 0.15 M ferrioxalate solution was added to 4.0 mL vial. The vial was irradiated for 15, 30, and 60 s. After irradiation, 100 μL of the solution was transferred to an aluminum foil covered 10 mL volumetric flask containing 1,10-phenanthroline (15 mg, 0.08 mmol) dissolved in 3.0 mL of the buffer solution. To the flask, water was added to make a total volume of 10 mL. After mixing the reagent

completely, the solution was stored for 1 h in the dark. 400 µL of the solution was transferred to a 1cm path quartz cuvette and the absorbance at λ = 452 nm was measured by using UV-visible spectrophotometer (Fig. S1). This process was repeated for 15, 30, and 60 s and the absorbance of a non-irradiated sample was also measured.

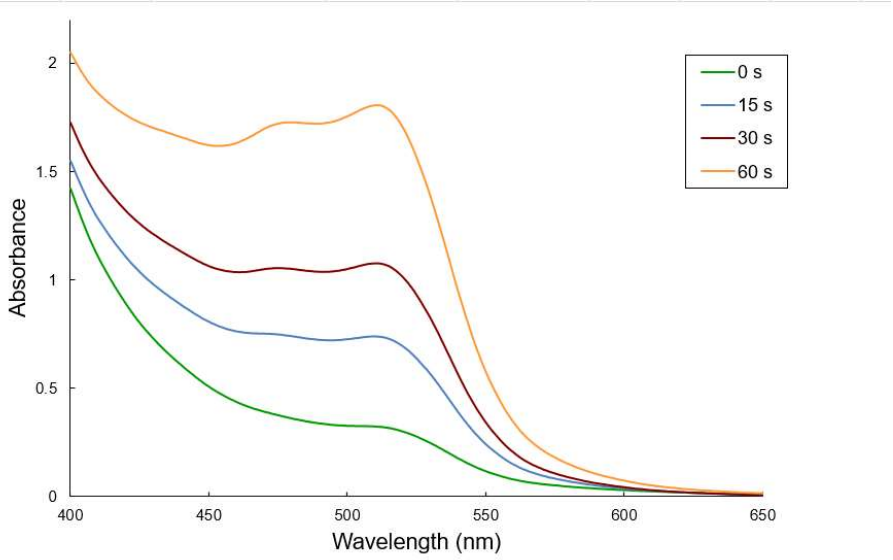


Fig. S1 UV-Vis spectra of ferrioxalate/1,10-phenanthroline solutions after light irradiation

The number of newly generated Fe^{2+} ions (mole) was calculated by following equation:

$$\text{mol } Fe^{2+} = \frac{V_1 V_3 \Delta A(510\ nm)}{V_2 l \epsilon(510 nm)}$$

Where V_1 is the volume of 0.15 M ferrioxalate solution irradiate (4.0 x 10^{-3} L), V_2 is the volume of the aliquot for measurement of the concentration of Fe^{2+} ions (1.0 x 10^{-4} L), V_3 is the final volume after complexation with 1,10- phenanthroline (1.0 x 10^{-2} L), ΔA(510 nm) Is the difference in absorbance at λ = 510 nm between he irradiated and non-irradiated ferrioxalate/1,10 phenanthroline solutions, l is the optical path length of the irradiation (1 cm), and ε(510 nm) is the molar absorptivity of the $Fe(phen)_3^{2+}$ complex at λ = 510 nm (11100 L mol^{-1} cm^{-1}). The moles of Fe^{2+} were plotted as a function of time (Fig. S2).

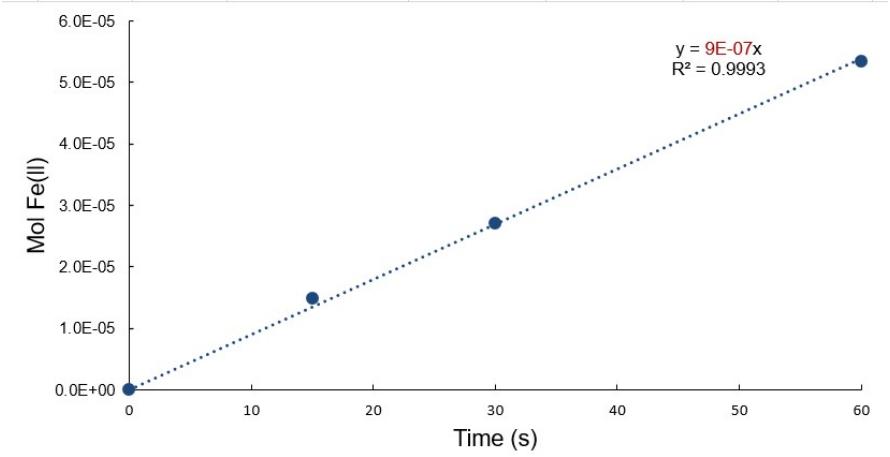


Fig. S2 Moles of Fe^{2+} formed vs. irradiation time

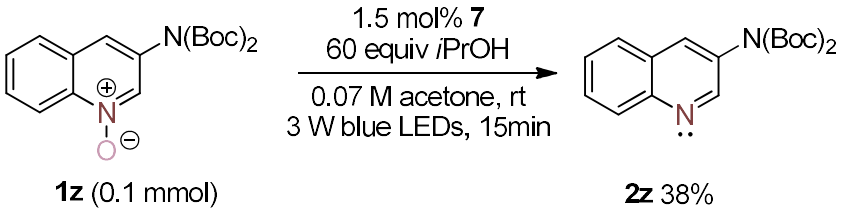
The photon flux was calculated by following equation:

$$\text{photon flux} = \frac{mol\ Fe^{2+}}{\Phi \times t \times f}$$

Where Φ is the quantum yield of the ferrioxalate actinometer (0.845 for a 0.15 M solution at 457 nm), t is the time, and f is the fraction of absorbed light at λ = 452 nm (f = 1–10^{-A} , A(452 nm) of 0.15 M ferrioxalate solution measured in a 1 cm path quartz cuvette by UV-Vis spectroscopy to be >5, therefore f = 1.00).

$$\text{photon flux} = \frac{9.0 \times 10^{-7}\ mol\ s^{-1}}{0.845 \times 1.00} = 1.07 \times 10^{-6}\ einstein\ s^{-1}$$

(2) Quantum yield(*Φ*) measurement:



In a 10-mL test tube, equipped with a small magnetic stir bar, 3-[bis(*tert*-butoxycarbonyl)amino]quinoline *N*-oxide (**1z**, 36.0 mg, 0.100 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 0.9 mg, 1.5 μmol, 1.5 mol%), and IPA (0.46 mL) were combined. The test tube was fitted with a rubber septum, and acetone (1.45 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 5 minutes, the reaction mixture

was irradiated with two 3 W blue LEDs (λ = 452 nm) at room temperature for 900 s (15 minutes). Product yield of **2z** (38%, 3.8 x 10^{-5} mol) was measured by ^{1}H-NMR analysis of the crude mixture in the presence of CH_2Br_2 as an internal standard. The quantum yield (Φ) was the calculated using the following equation.

$$\Phi = \frac{n(product,mol)}{photon\ flux \times t \times f}$$

Where t represents the time (15 minutes = 900 seconds) and f denotes the fraction of light absorbed by the reaction mixture at λ = 452 nm, where $f = 1 - 10^{-A}$ (with A being the absorbance of the reaction mixture at λ = 452 nm measured in a 1 cm path quartz cuvette by a UV-Vis spectrometer; the measured A = 3.333, thus $f \approx 1$). The quantum yield was then calculated to be 0.03 by the following equation.

$$\Phi = \frac{3.8 \times 10^{-5}\ mol}{1.07 \times 10^{-6} einstein\ s^{-1} \times 900\ s \times 1.00} = 0.03$$

The quantum yield studies indicate that this is not a radical-chain propagation mechanism as the Φ<1.

Experimental Procedure for Scheme 1c (Visible Light On/Off Experiment)

Five 10-mL test tubes, each fitted with a magnetic stir bar, were prepared. Methyl 3-quinolinecarboxylate *N*-oxide (**1a**, 40.6 mg, 0.200 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 1.7 mg, 3.0 μmol, 1.5 mol%), and IPA (0.92 mL) were added to the tubes. The tubes were sealed with rubber septa, and acetone (2.9 mL, 0.07 M) was injected using a syringe under a nitrogen atmosphere. After degassing by bubbling N_2 at room temperature for 5 minutes, all tubes were exposed to room temperature irradiation with two 3 W blue LEDs for 50 minutes. Subsequently, one tube was chosen, and its contents were concentrated using a rotary evaporator. The resulting residue was purified via flash column chromatography on silica gel (hexanes/EtOAc = 3:1) to obtain methyl 3-quinolinecarboxylate (**2a**, 19.5 mg, 52%) as a white solid. The remaining four tubes were covered with aluminum foil and kept under stirring with 3 W blue LEDs. After 30 minutes, one tube was selected, and its contents were concentrated using a rotary evaporator. The obtained residue was purified similarly, yielding methyl 3-quinolinecarboxylate (**2a**, 19 mg, 51%) as a white solid. Following the removal of the aluminum foil, the three remaining tubes were stirred under irradiation for 50 minutes. One tube was chosen, and its contents were concentrated and purified as before, resulting in methyl 3-

quinolinecarboxylate (**2a**, 25 mg, 68%) as a white solid. Additionally, two tubes were covered with aluminum foil and irradiated for 30 minutes. The contents of one tube were purified as previously described, yielding methyl 3-quinolinecarboxylate (**2a**, 26 mg, 70%) as a white solid. The other tube, after removing the aluminum foil, was stirred under irradiation for 50 minutes. Similar purification of its contents produced 3-quinolinecarboxylate (**2a**, 34 mg, 92%) as a white solid.

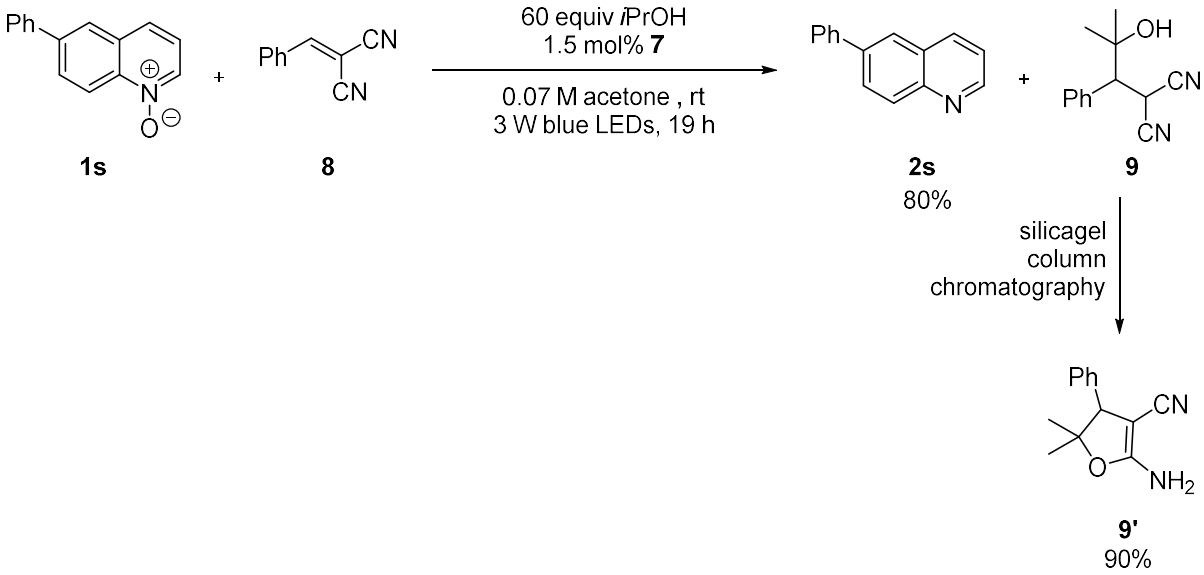
Experimental Procedure for Scheme 1d (employing radical inhibitor)

In a couple of 10-mL test tubes, each equipped with a small magnetic stir bar, 3-acetylquinoline *N*-oxide (**1f**, 37.4 mg, 0.20 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 1.7 mg, 3.0 μmol, 1.5 mol%), and IPA (0.92 mL) were combined. One tube was charged with (2,2,6,6-tetramethylpiperidi*N*-1-yl)oxyl (TEMPO, 188 mg, 1.2 mmol, 6.0 equivalent), and the other tube was charged with 2,2-diphenyl-1-picrylhydrazyl (DPPH, 87 mg, 0.22 mmol, 1.1 equivalent). These test tubes were fitted with rubber septa, and acetone (2.9 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 5 minutes, the reaction mixtures were irradiated with two 3 W blue LEDs at room temperature for 7 hours. In the reaction with TEMPO, the resulting residue was directly purified by flash column chromatography on silica gel (hexanes/EtOAc = 1:1) to afford 3-acetylquinoline (**2f**, 8.9 mg, 26%) as a white solid. For the reaction with DPPH, TLC analysis indicated that no conversion of 1**f** into the deoxygenated product **2f** occurred.

Experimental Procedure for Scheme 1e (radical trapping experiment)

In a 10-mL test tube, equipped with a small magnetic stir bar, methyl quinoline-3-carboxylate *N*-oxide (**1a**, 40.6 mg, 0.200 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 1.7 mg, 3.0 μmol, 1.5 mol%), and benzylidenemalononitrile (**8**, 30.8 mg, 0.200 mmol, 1.0 equivalent), IPA (0.92 mL) were combined. The test tube was fitted with a rubber septum, and CD_3CN (2.9 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 5 minutes, the reaction mixture was irradiated with two 3 W blue LEDs at room temperature for 1 hours. Methyl quinoline-3-carboxylate (**2a**, 60%) and 2-(2-hydroxy-2-methyl-1-phenylpropyl)propanedinitrile (**9**, 50%) were determined by ^1H NMR analysis, utilising dibromomethane as an internal standard.

Experimental Procedure for radical trapping experiment II (footnote 46)



In a 10-mL test tube, equipped with a small magnetic stir bar, 6-phenylquinoline *N*-oxide (**1s**, 44.3 mg, 0.20 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 1.7 mg, 3.0 µmol, 1.5 mol%), and benzylidenemalononitrile (**8**, 30.8 mg, 0.20 mmol, 1.00 equivalent), IPA (0.92 mL) were combined. The test tube was fitted with a rubber septum, and acetone (2.9 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 5 minutes, the reaction mixture was irradiated with two 3 W blue LEDs at room temperature for 19 hours. Upon completion of the reaction as determined by TLC, the reaction mixture was concentrated on a rotary evaporator. The resulting residue was purified by flash column chromatography on silica gel (hexanes/EtOAc = 10:1 to 3:1) to afford 6-phenylquinoline (**2s,** 33.0 mg, 80%) and 2-amino-5,5-dimethyl-4-phenyl-4,5-dihydrofuran-3-carbonitrile (**9'**, 38.4 mg, 90%). The latter compound resulted from the cyclisation of 2-(2-hydroxy-2-methyl-1-phenylpropyl)propanedinitrile (**9**) during the course of column chromatography.

2-Amino-5,5-dimethyl-4-phenyl-4,5-dihydrofuran-3-carbonitirile (9')

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (t, *J* = 7.4 Hz, 2H), 7.29 (d, *J* = 7.4 Hz, 1H), 7.20 (d, *J* = 7.7 Hz, 2H), 4.95 (s, 2H), 4.04 (s, 1H), 1.55 (s, 3H), 0.88 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.0, 138.8, 128.6, 128.4, 127.6, 119.8, 91.4, 57.6, 55.8, 29.2, 24.3.

LC-MS (ESI) *m*/*z* calculated for $[M+1]^+$ ($C_{13}H_{15}N_2O$) 215.1, found 215.1.

Stern-Volmer Luminescence Quenching Experiments (Fig. 2)

Emission Quenching of Mes-*t*Bu$_2$Acr$^+$ (7)

A 150-mL volumetric flask containing the photocatalyst, 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 2.8 mg, 5 μmol) was fitted with a rubber septum and charged with acetone (*ca*. 101 mL) via a syringe. The resulting homogeneous solution was degassed by sparging with nitrogen until its volume reached to 100 mL. The solutions of IPA (100 mM), **1b**, **1x**, and quinoline *N*-oxide (60 mM) in acetone were also prepared similarly. Stern-Volmer luminescence quenching experiments were performed using a Varian Cary eclipse fluorescence spectrophotometer. In each experimentation, the solution of **7** and varying concentrations of quencher (IPA, **1b**, **1x**, or quinoline *N*-oxide) were combined in degassed acetone in 1 cm screw-top quartz cuvettes. For the emission quenching of **7**, the photocatalyst concentration was 20 μM, the solution was excited at 450 nm, and the emission intensity was observed at 510 nm. Plots were constructed according to the Stern-Volmer equation $I_0/I = 1+k_q\tau_0[Q]$. I_0 and I represent the intensities of the emission in the absence and presence of a quencher at 510 nm, respectively.

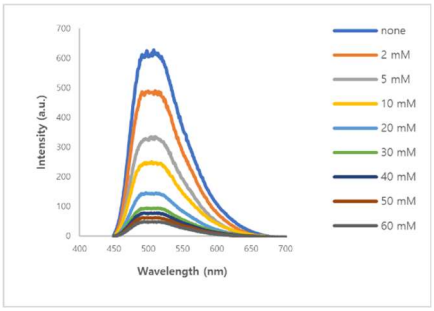


Fig. S3 Emission Quenching of **7** by **1x**

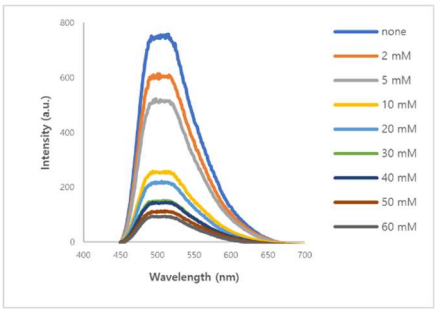


Fig. S5 Emission Quenching of **7** by **1b**

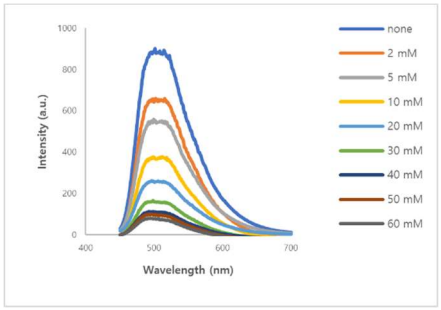


Fig. S4 Emission Quenching of **7** by **quinoline *N*-oxide**

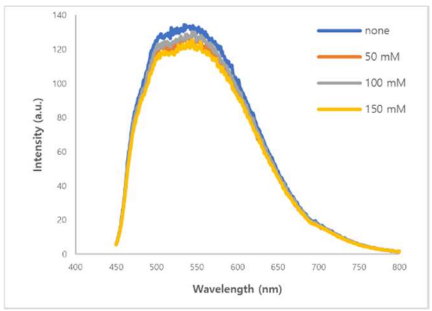


Fig. S6 Emission Quenching of **7** by **IPA**

Cyclic Voltammetry Measurements (Fig. 2)

Cyclic voltammograms were collected with a Gamry Potentiostat. Samples were prepared with 0.004 mmol of substrate in 4 mL of 0.1 M tetra-*n*-butylammonium hexafluorophosphate in anhydrous, degassed acetonitrile. Measurements employed a glassy carbon working electrode, platinum wire counter electrode, Ag/Ag^+ reference electrode, and a scan rate of 100 mV/s. After adding 0.004 mmol ferrocene as an internal standard, the potential of ferrocene was measured under the same conditions. All potentials reported here vs. SCE and been converted by adding + 0.47 V and V to the potentials measured vs. Fc/Fc^+.

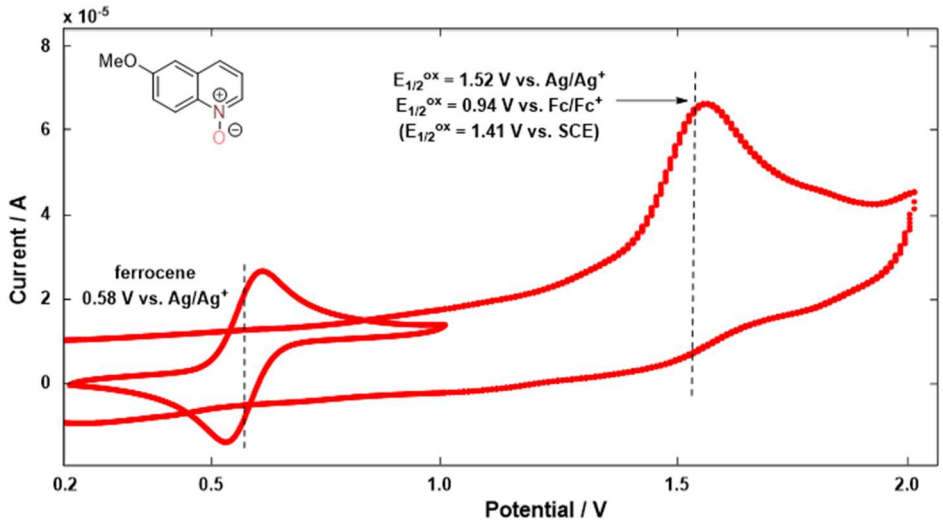


Fig.S7 Cyclic Voltammetry Measurement of **1x**

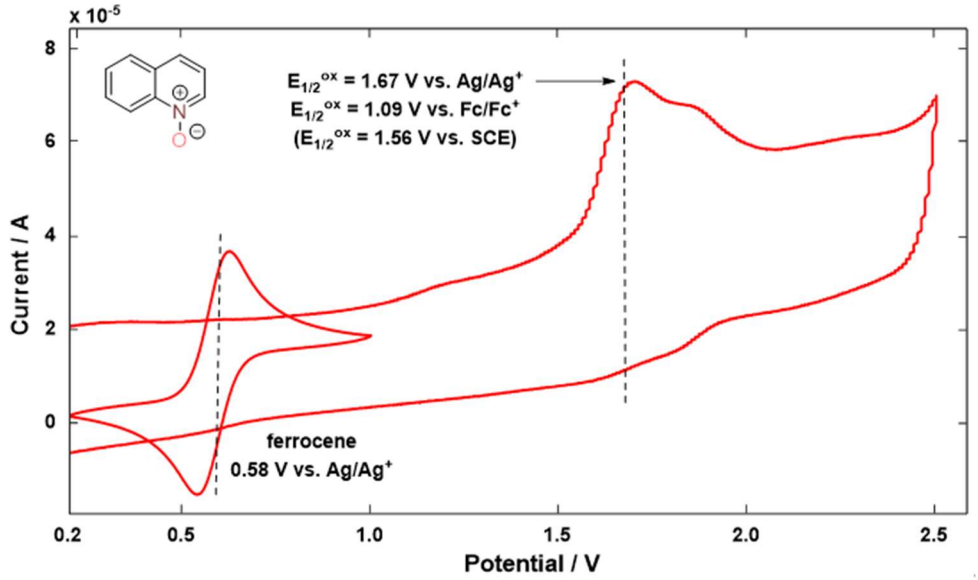


Fig.S8 Cyclic Voltammetry Measurement of quinoline *N*-oxide

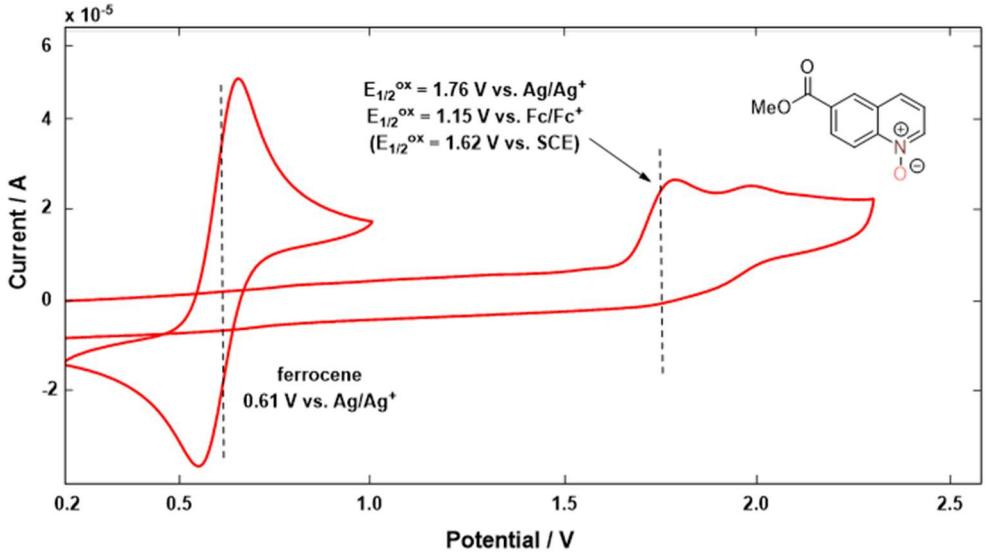
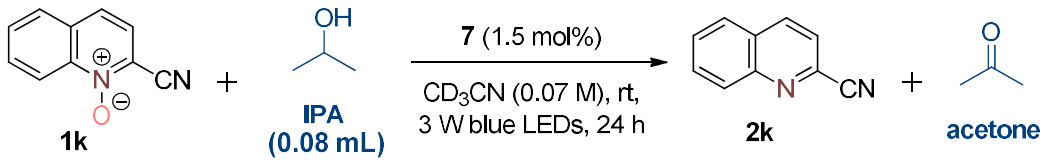


Fig.S9 Cyclic Voltammetry Measurement of **1b**

NMR Spectroscopic Studies for Confirming the Generation of Acetone as a Byproduct



In a 10-mL test tube, equipped with a small magnetic stir bar, 2-quinolinecarbonitrile *N*-oxide (**1k**, 34.0 mg, 0.200 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 1.7 mg, 3.0 μmol, 1.5 mol%), IPA (0.08 mL), and acetonitrile-d_3 (2.9 mL, 0.07 M) were combined. A small portion (*ca*. 0.6 mL) of the resulting mixture was transferred into a J-Young tube via a syringe, and the homogeneous mixture was thoroughly degassed using the freeze-pump-melting method (3 cycles). The reaction mixture was irradiated with two 3 W blue LEDs at room temperature for 24 hours. The disappearance of IPA and the simultaneous generation of acetone were validated by both ^{1}H and ^{13}C NMR analyses, as shown in Fig. S10 and Fig. S11.

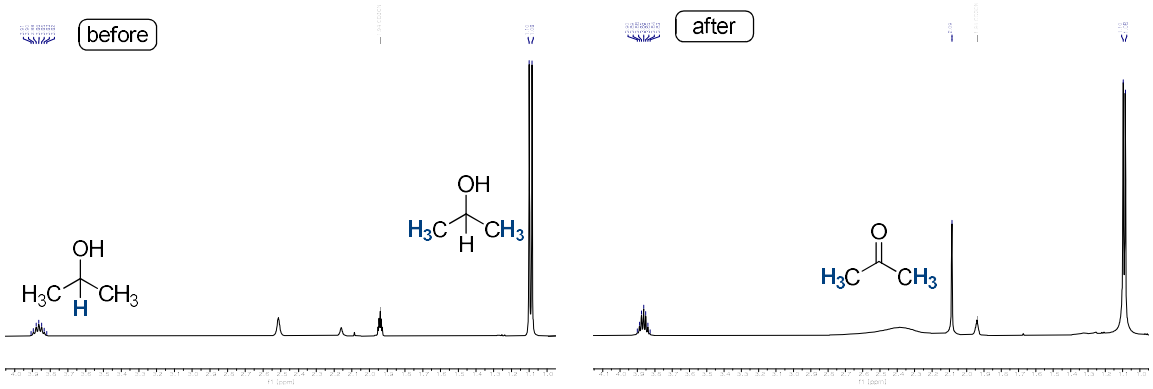


Fig. S10 Acetone Confirm in ^{1}H NMR

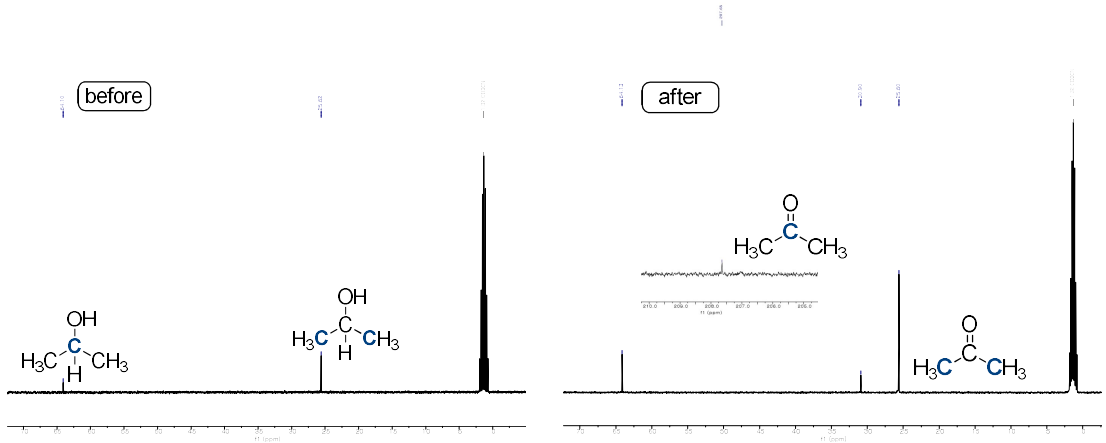


Fig. S11 Acetone Confirm in ^{13}C NMR

Experimental Procedure for Multigram-Scale Deoxygenation Reaction (Scheme 3)

Fig. S12 Gram-scale Photoreactor (light-off)

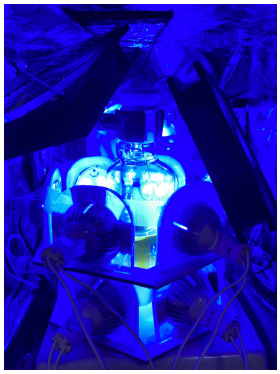


Fig. S13 Gram-scale Photoreactor (light-on)

(a) Recrystallisation

In a 350-mL test tube, equipped with a magnetic stir bar, 2-quinolinecarbonitrile *N*-oxide (**1k**, 1.3 g, 7.5 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 65 mg, 0.11 mmol, 1.5 mol%), and IPA (11.5 mL) were combined. The flask was fitted with a rubber septum, and acetone (107 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 30 minutes, the reaction mixture was irradiated with twelve 3 W blue LEDs at room temperature for 11 hours. Afterward, the reaction mixture was concentrated on a rotary evaporator. The resulting residue was purified by recrystallisation from a hot mixture of hexanes and IPA (1:1, *v*/*v*) to afford 2-quinolinecarbonitrile (**2k**, 0.97 g, 84%) as a pale yellow solid.

In a 350-mL test tube, equipped with a magnetic stir bar, 3-[bis(*tert*-butoxycarbonyl)amino]quinoline *N*-oxide (**1z**, 1.0 g, 3.0 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 25.6 mg, 0.045 mmol, 1.5 mol%), and IPA (14 mL) were combined. The flask was fitted with a rubber septum, and acetone (43 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 30 minutes, the reaction mixture was irradiated with twelve 3 W blue LEDs at room temperature for 23 hours. Afterward, the reaction mixture was concentrated on a rotary evaporator. The resulting residue was purified by recrystallisation from a hot mixture of hexanes and IPA (3:1, *v*/*v*) to afford 3-[bis(*tert*-butoxycarbonyl)amino]quinoline (**2z**, 0.95 g, 92%) as a white solid.

(b) Kugelrohr distillation

In a 350-mL test tube, equipped with a magnetic stir bar, 4-benzoylpyridine *N*-oxide (**1ak**, 1.2 g, 6.0 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 51 mg, 0.09 mmol, 1.5 mol%), and IPA (27.5 mL) were combined. The flask was fitted with a rubber septum, and acetone (86 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 30 minutes, the reaction mixture was irradiated with twelve 3 W blue LEDs at room temperature for 15 hours. Afterward, the reaction mixture was concentrated on a rotary evaporator. The resulting residue was purified by Kugelrohr distillation to afford 4-benzoylpyridine (**2ak**, 1.1 g, 97%) as a brown oil.

In a 350-mL test tube, equipped with a magnetic stir bar, 4-acetylpyridine *N*-oxide (**1an**, 1.1 g, 8.0 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 69 mg, 0.11 mmol, 1.5 mol%), and IPA (37 mL) were combined. The flask was fitted with a rubber septum, and acetone (114 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 30 minutes, the reaction mixture was irradiated with twelve 3 W blue LEDs at room temperature for 15 hours. Afterward, the reaction mixture was concentrated on a rotary evaporator. The resulting residue was purified by Kugelrohr distillation to afford 4-acetylpyridine (**2an**, 0.84 g, 87%) as a white solid.

Experimental Procedure for Recycling Process (Scheme 4)

In a 250-mL round-bottomed flask, equipped with a magnetic stir bar, 4-benzoylpyridine *N*-oxide (**1ak**, 1.02 g, 5.1 mmol, 1.0 equivalent), 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 44 mg, 0.077 mmol, 1.5 mol%), and IPA (25 mL) were combined. The flask was fitted with a rubber septum, and acetone (73 mL, 0.07 M) was added via a syringe under a nitrogen atmosphere. After degassing by N_2 bubbling at room temperature for 30 minutes, the reaction mixture was irradiated with twelve 3 W blue LEDs at room temperature for 17 hours. Afterward, the round-bottomed flask was connected to a distillation still and heated in an oil bath to collect acetone and IPA together. The residue in the round-bottomed flask was then transferred to a small bulb, which was assembled with a Büchi Kugelrohr distillation apparatus to collect a small amount of acetone and IPA together. The resulting

mixture in the bulb was purified by Kugelrohr distillation to afford 4-benzoylpyridine (**2ak**, 0.86 g, 92%) as a white solid. The combined mixture of acetone and IPA (a total of 93 mL) was determined to be approximately 2.7:1 by ^{1}H-NMR spectroscopy. In another 250-mL round-bottomed flask, equipped with a magnetic stir bar, 4-benzoylpyridine *N*-oxide (**1ak**, 1.02 g, 5.1 mmol, 1.0 equivalent) and 9-mesityl-3,6-di-*tert*-butyl-10-phenylacridinium tetrafluoroborate (**7**, 44 mg, 0.076 mmol, 1.5 mol%) were combined. The flask was fitted with a rubber septum, and the recovered mixture of acetone and IPA was added via a syringe under a nitrogen atmosphere. The resulting mixture was treated with a small amount of acetone (3–7 mL) via a syringe. After degassing by N_2 bubbling at room temperature for 30 minutes, the reaction mixture was irradiated with twelve 3 W blue LEDs at room temperature for 15–19 hours. This recycling process was repeated 9 times more, and the results are summarised in Table S10.

Table S10. Recycling Process[a]

Runs	Recovered IPA and acetone (mL)	Amount of acetone added (mL)	Time (h)	Yield (%)
1	91	7	15	92
2	92	6	15	94
3	92	6	15.5	97
4	94	4	16	95
5	94	4	17	95
6	93	5	18	96
7	94	4	18.5	97
8	94	4	18.5	96
9	95	3	19	96
10	94	-	19	96

[a]Conditions: **1ak** (5.1 mmol), IPA (25 mL), and **7**(1.5 mol%) in degassed acetone (73 mL, 0.07 M).

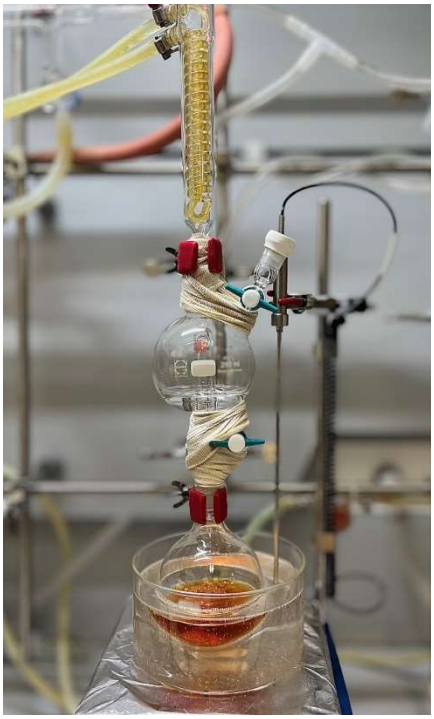


Fig. S14 Recycling Process (simple distillation)

Fig. S15 Recycling Process (Kugelrohr distillation)

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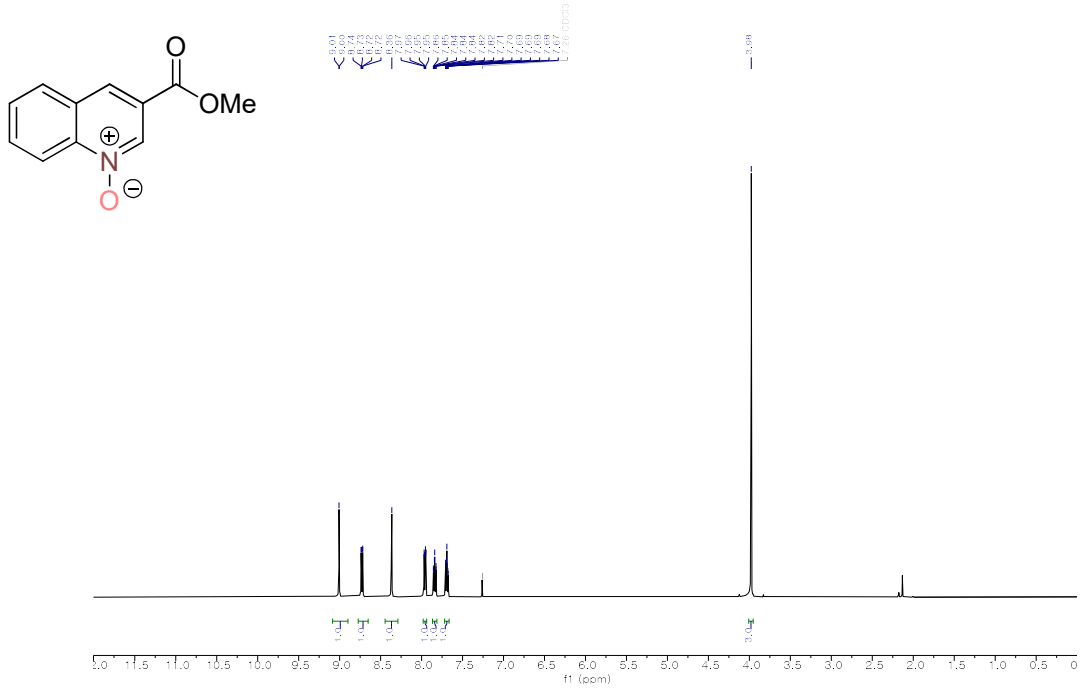
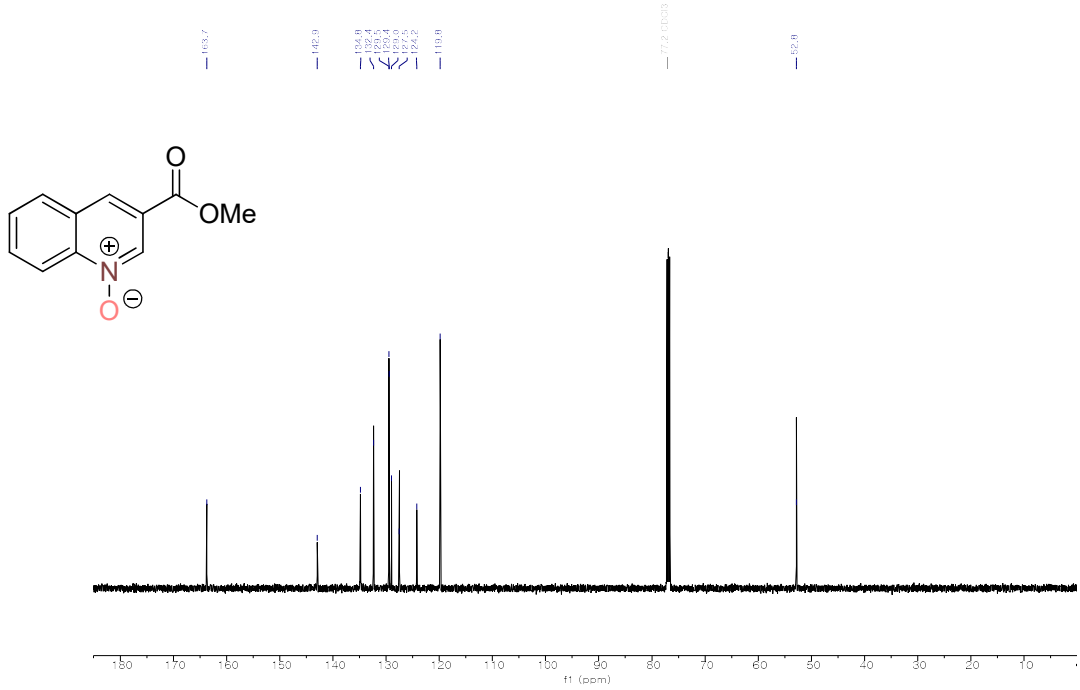
NMR Spectra

^{1}H & ^{13}C NMR Spectra of Methyl Quinoline-3-carboxylate *N*-Oxide (1a)

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

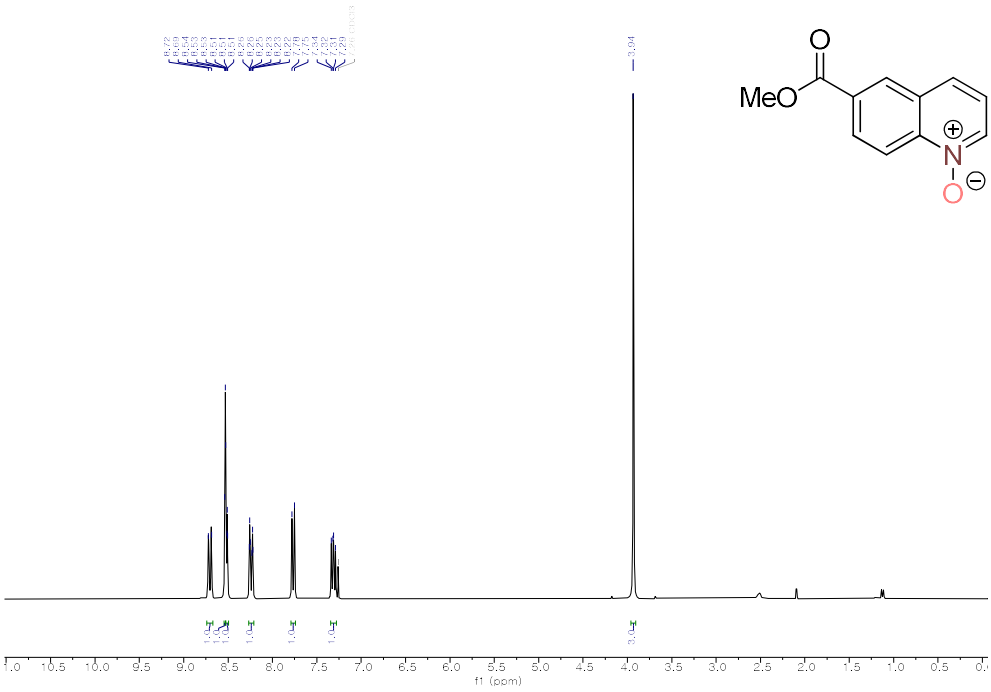


^{13}C NMR (126 MHz, $CDCl_3$)

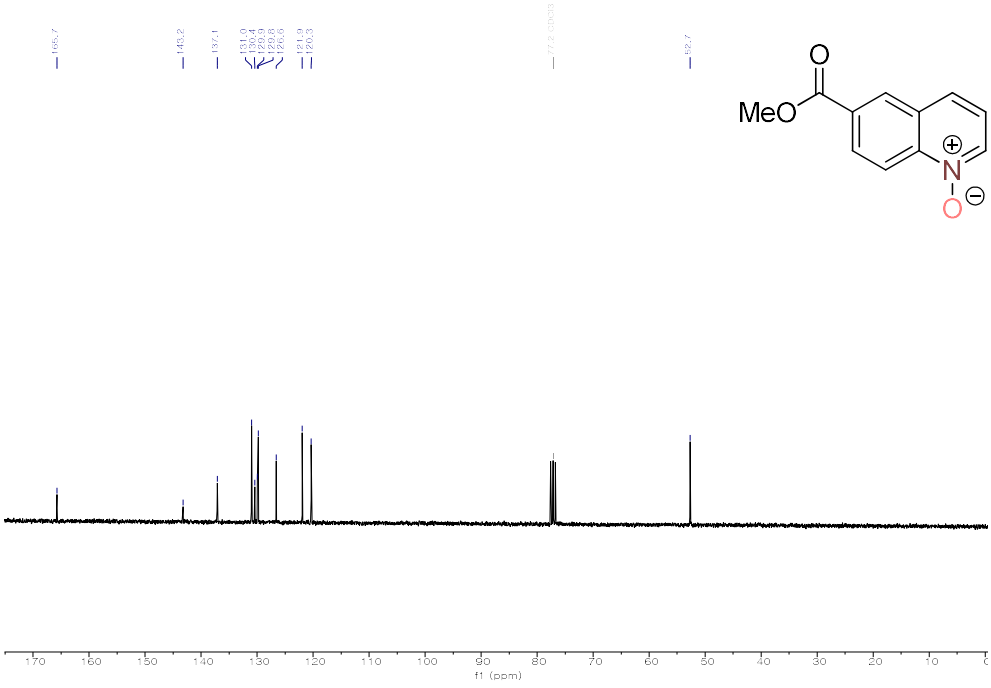


^{1}H & ^{13}C NMR Spectra of Methyl Quinoline-6-carboxylate *N*-Oxide (1b)

^{1}H NMR (300 MHz, $CDCl_3$)

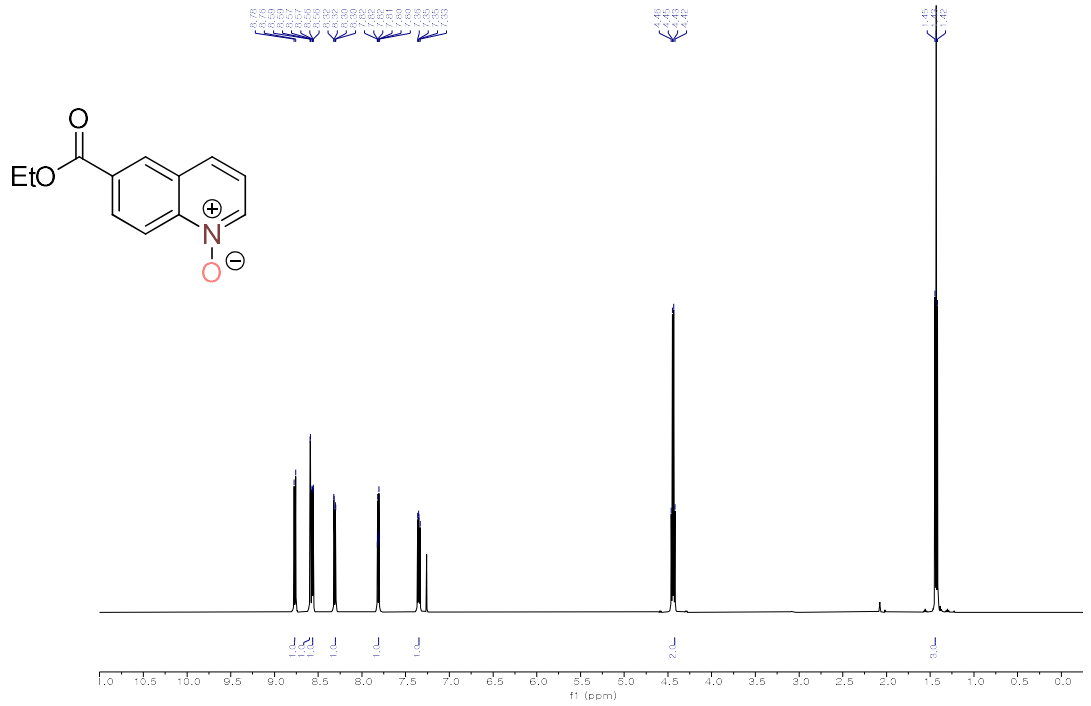


^{13}C NMR (75 MHz, $CDCl_3$)

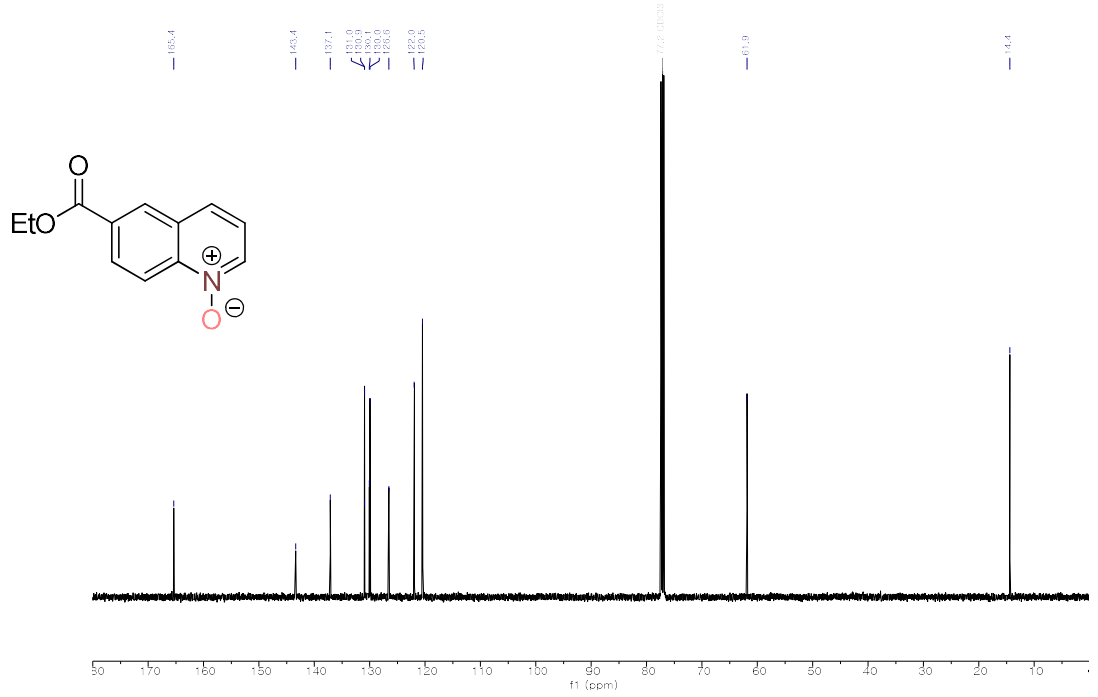


^{1}H & ^{13}C NMR Spectra of Ethyl Quinoline-6-carboxylate *N*-Oxide (1c)

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

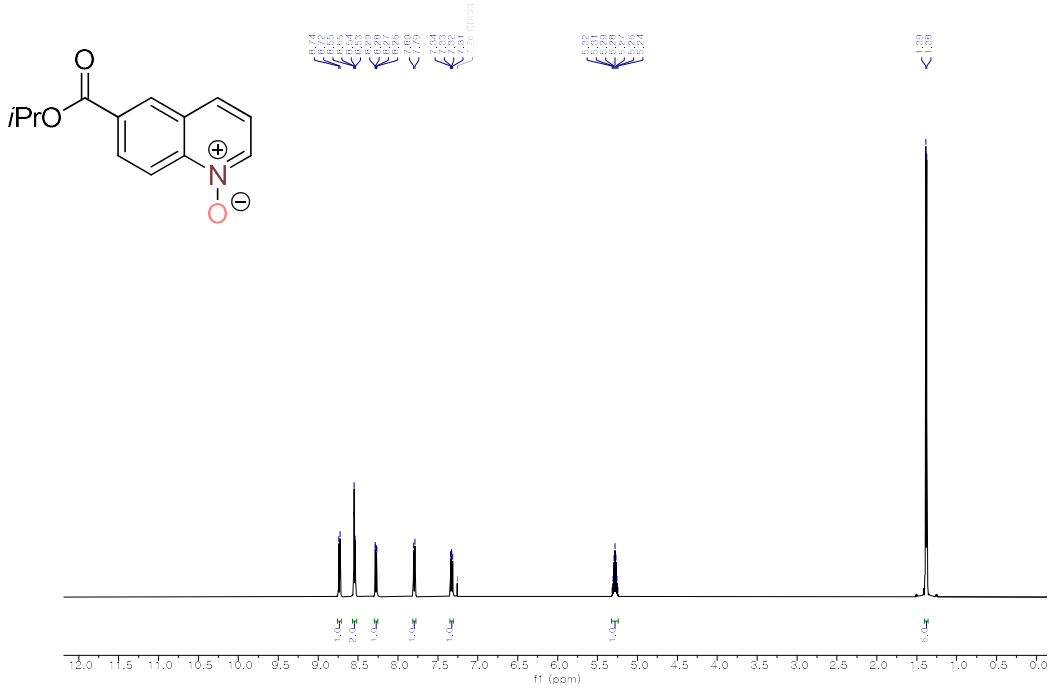


^{13}C NMR (126 MHz, $CDCl_3$)

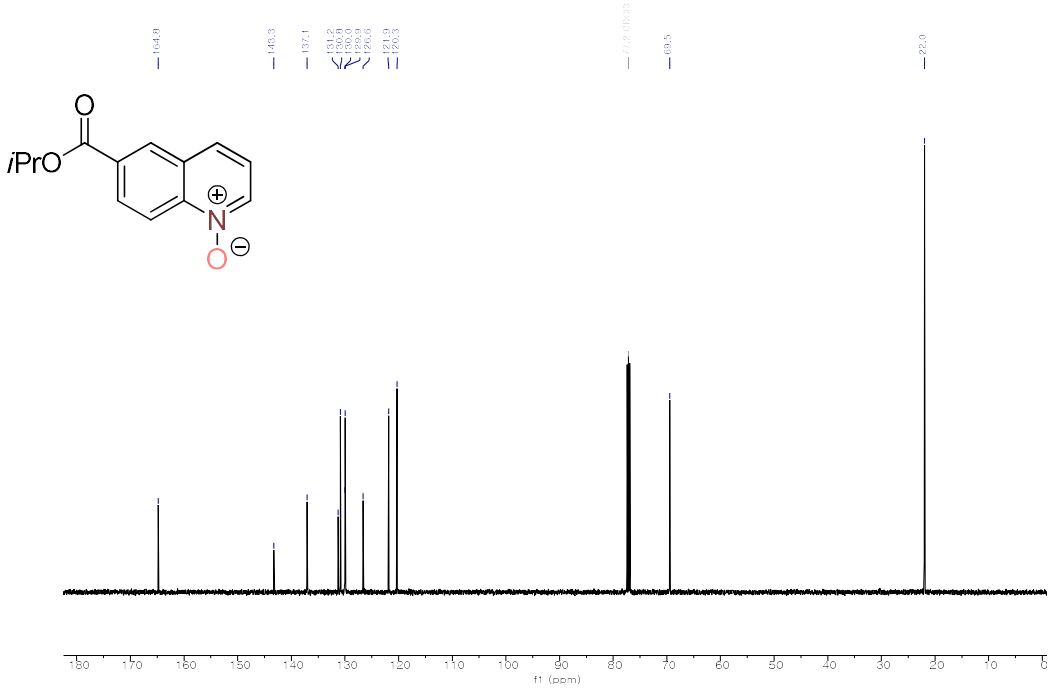


^{1}H & ^{13}C NMR Spectra of Isopropyl Quinoline-6-carboxylate *N*-Oxide (1d)

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

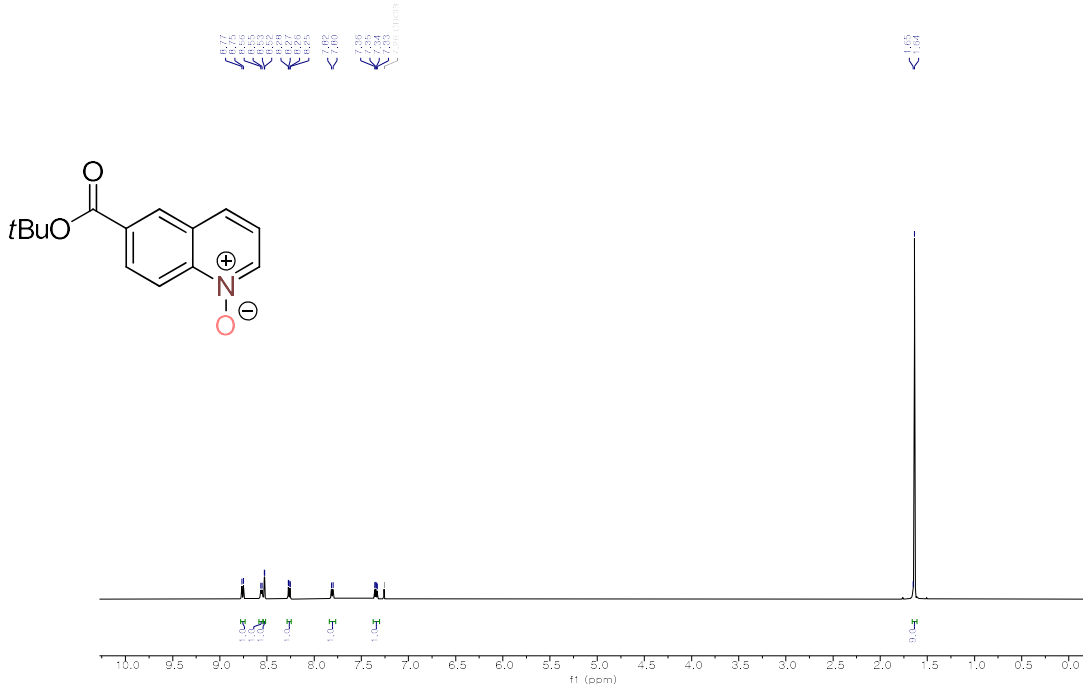


^{13}C NMR (126 MHz, $CDCl_3$)

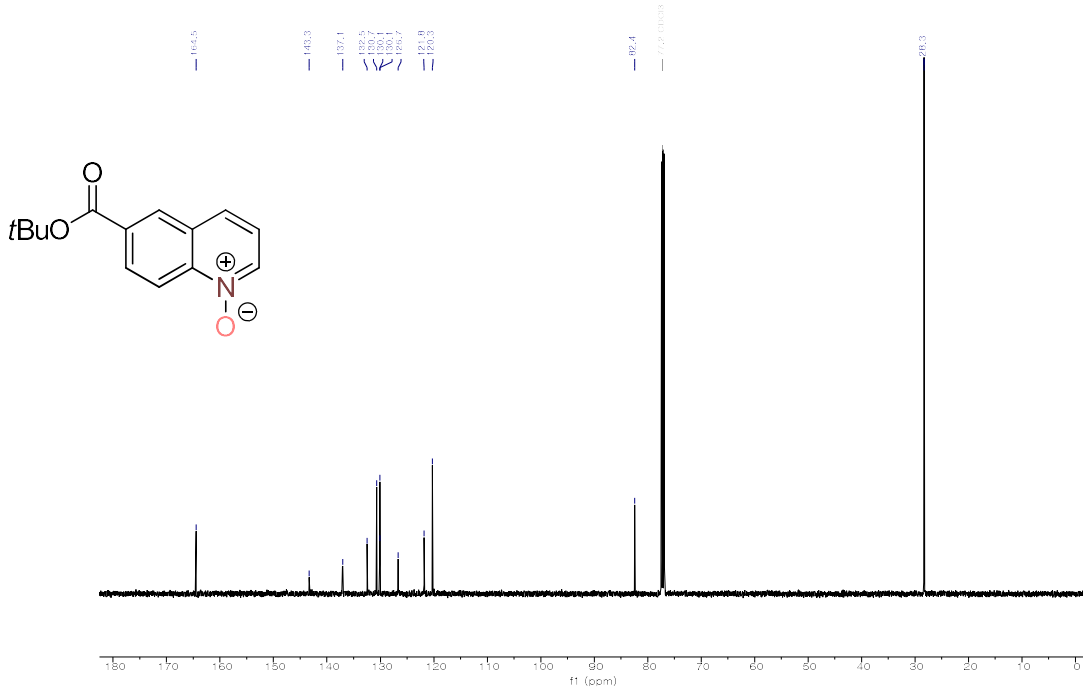


^{1}H & ^{13}C NMR Spectra of *tert*-Butyl Quinoline-6-carboxylate *N*-Oxide (1e)

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

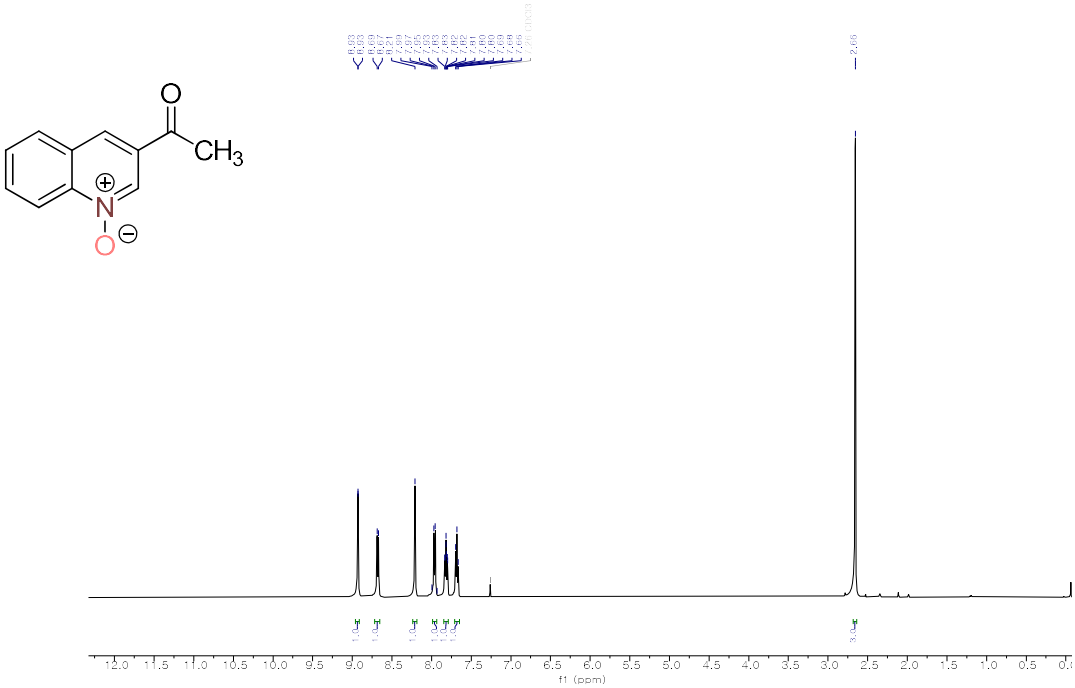


^{13}C NMR (126 MHz, $CDCl_3$)

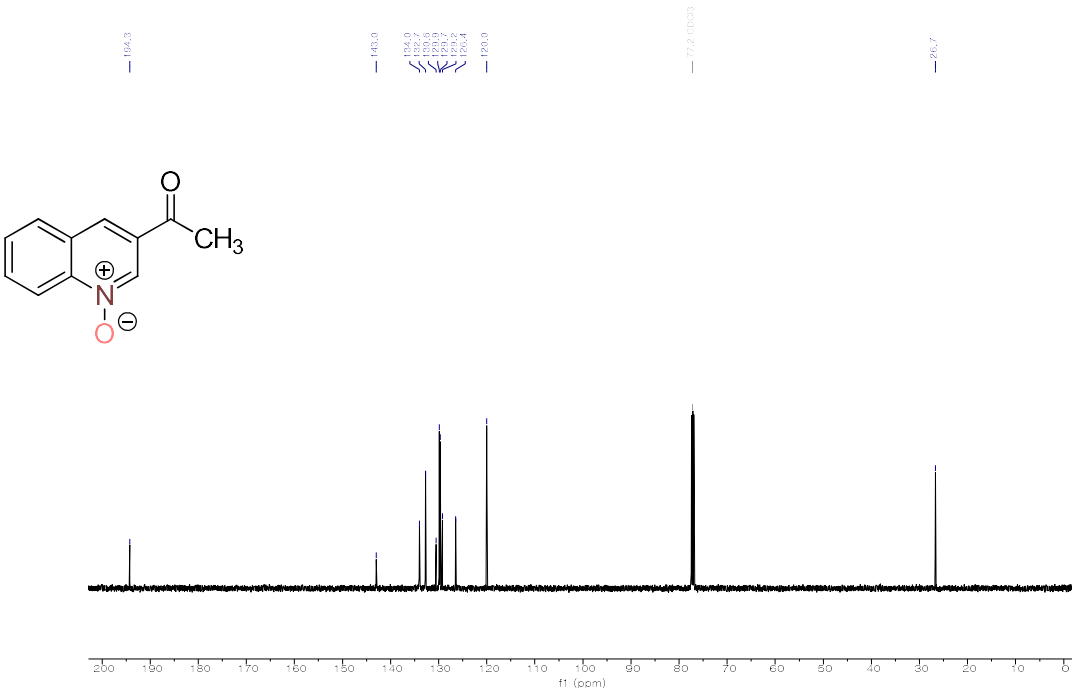


^{1}H & ^{13}C NMR Spectra of 3-Acetylquinoline *N*-Oxide (1f)

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

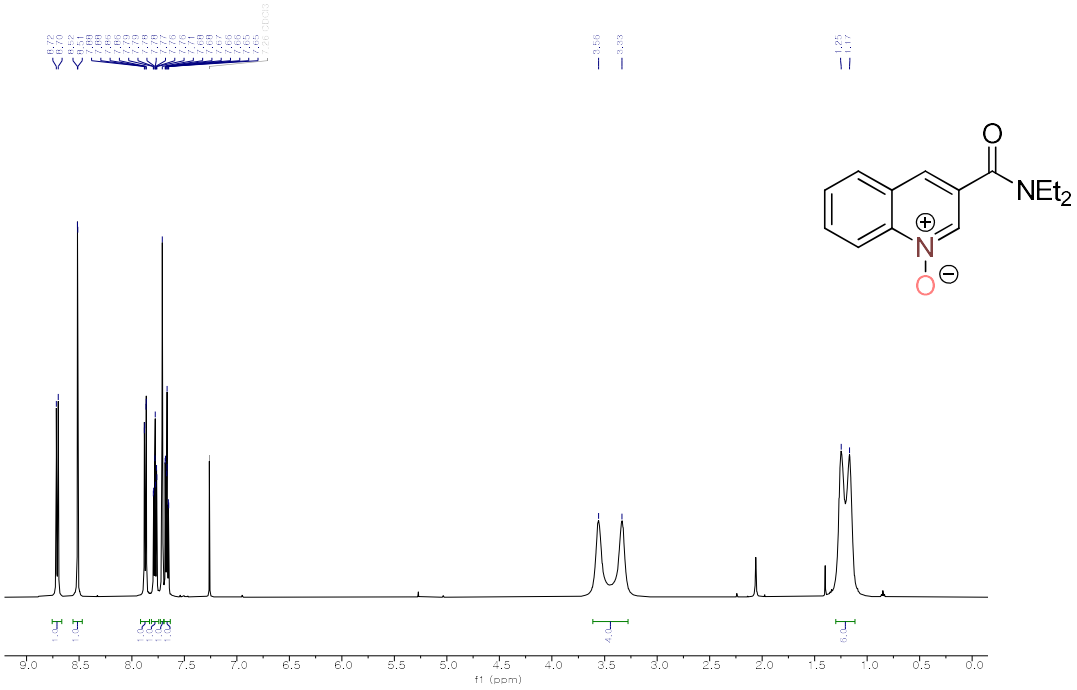


^{13}C NMR (126 MHz, $CDCl_3$)

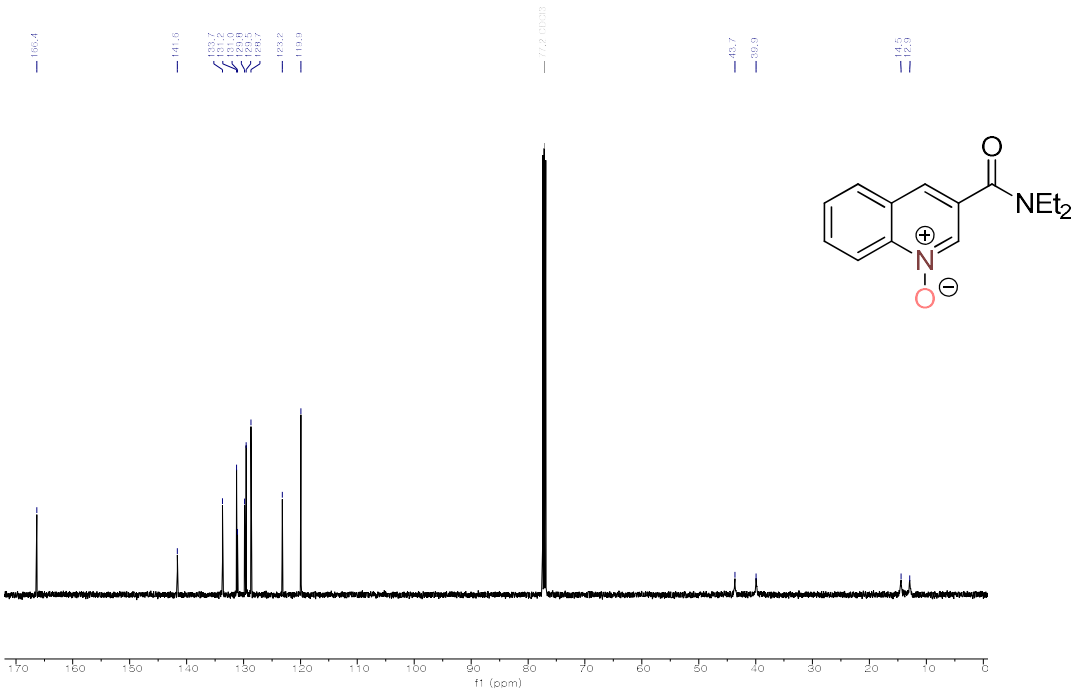


^{1}H & ^{13}C NMR Spectra of *N,N*-Diethyl-3-quinolinecarboxamide *N*-Oxide (1g)

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

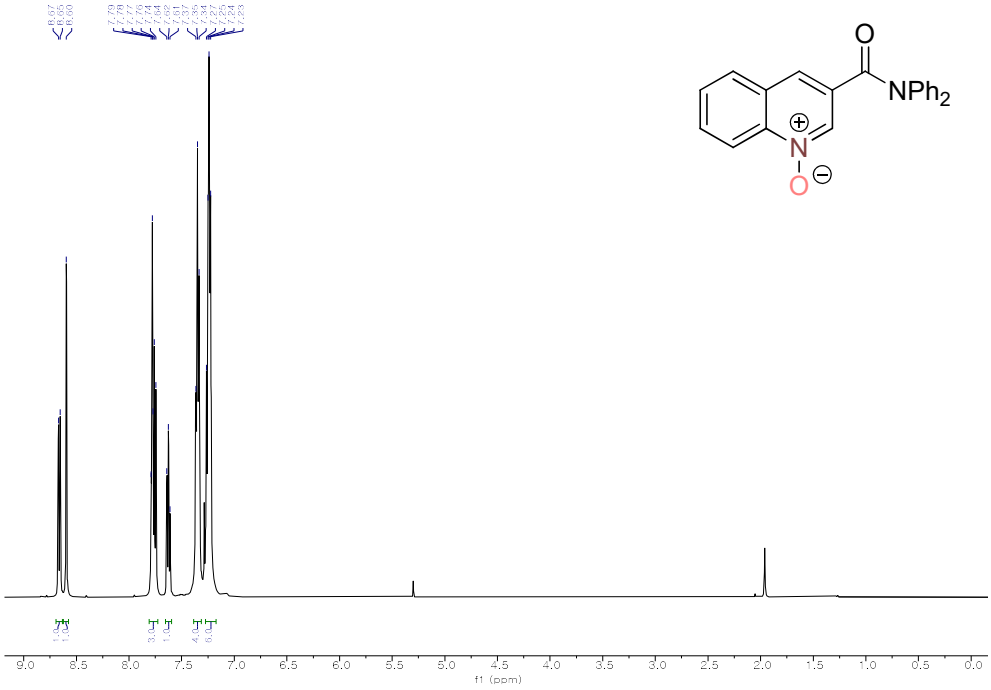


^{13}C NMR (126 MHz, $CDCl_3$)

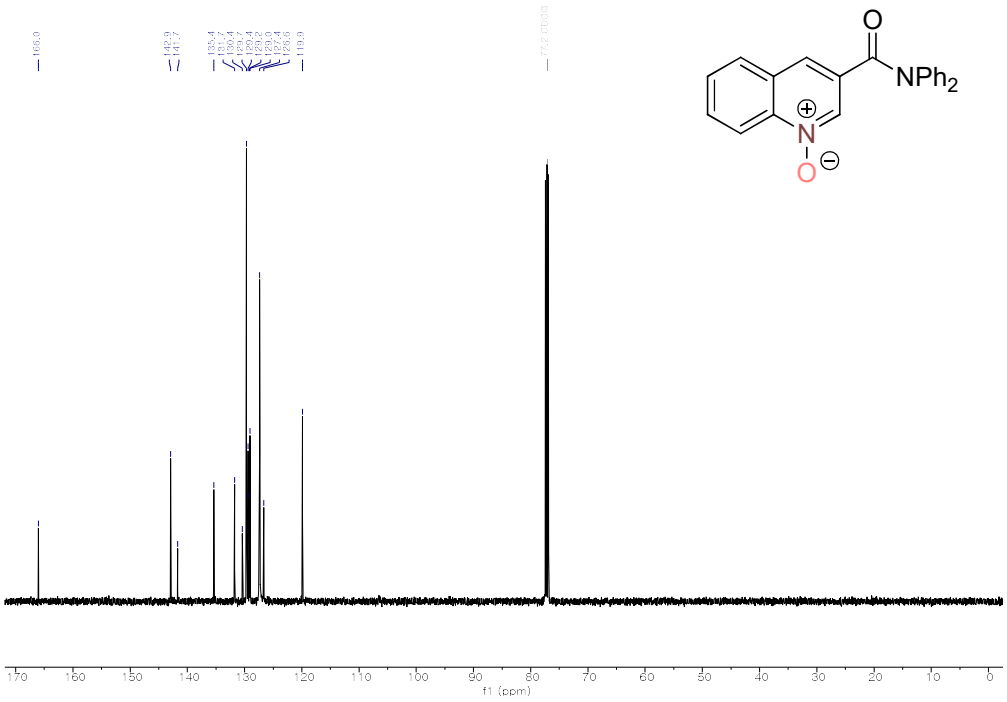


^{1}H & ^{13}C NMR Spectra of *N,N*-Diphenyl-3-quinolinecarboxamide *N*-Oxide (1h)

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

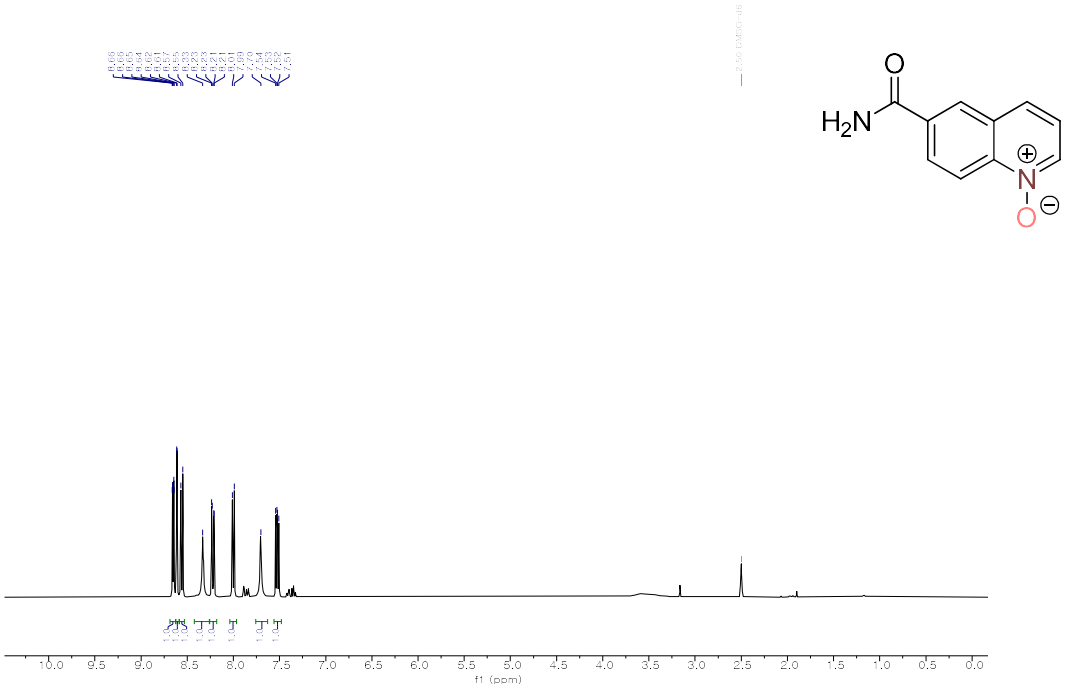


^{13}C NMR (126 MHz, $CDCl_3$)

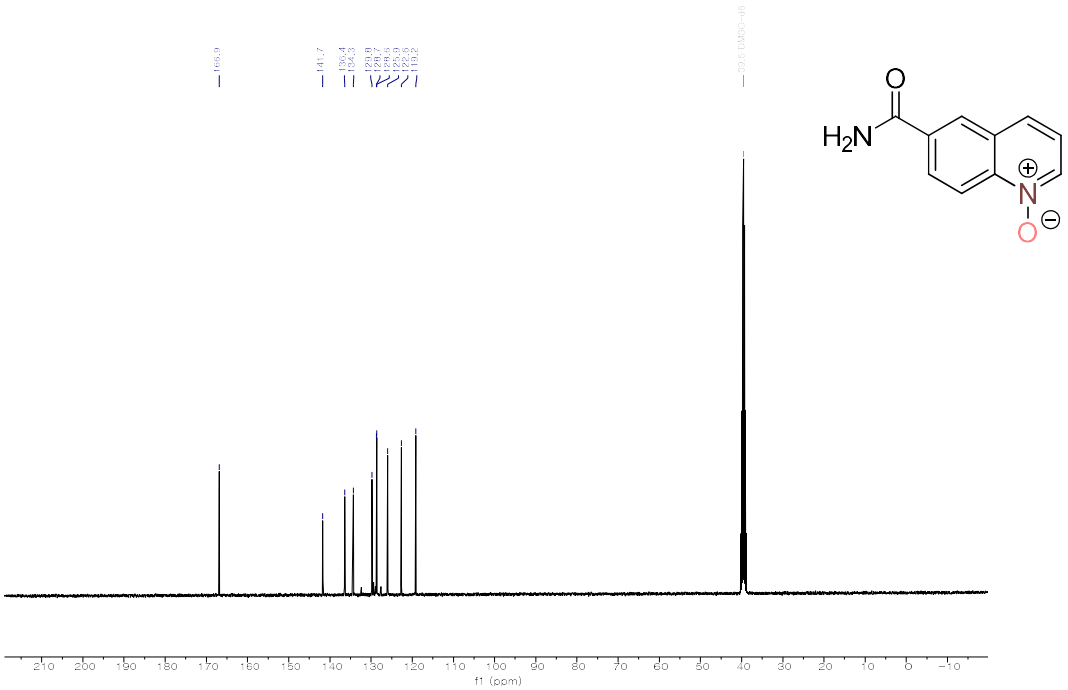


^{1}H & ^{13}C NMR Spectra of 6-Quinolinecarboxamide *N*-Oxide (1i)

^{1}H NMR (400 MHz, DMSO)

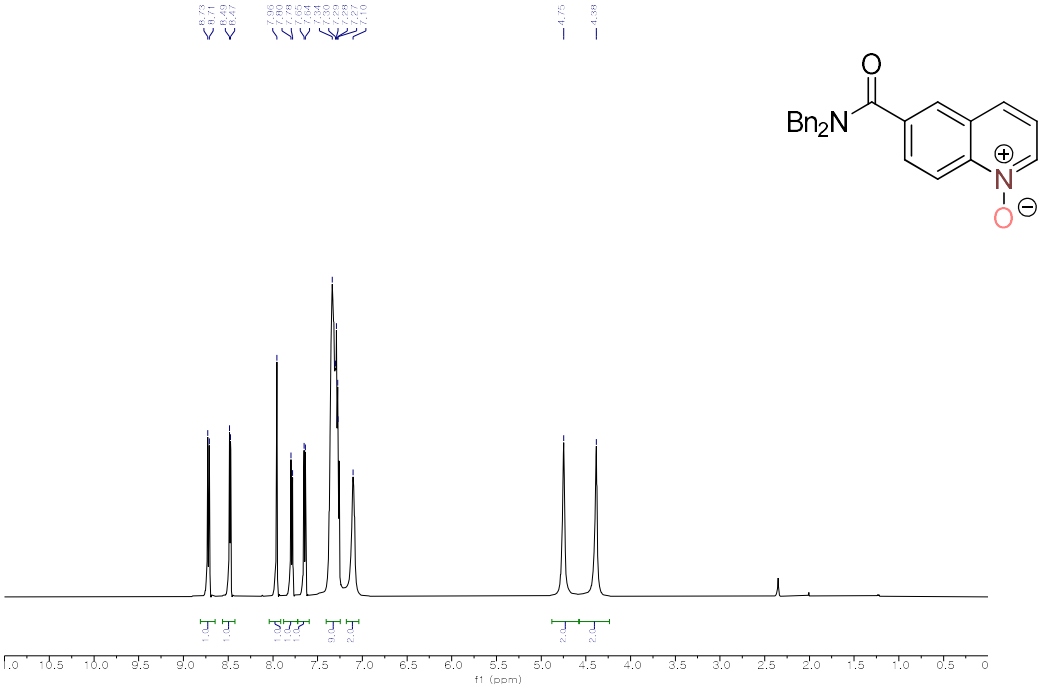


^{13}C NMR (101 MHz, DMSO)

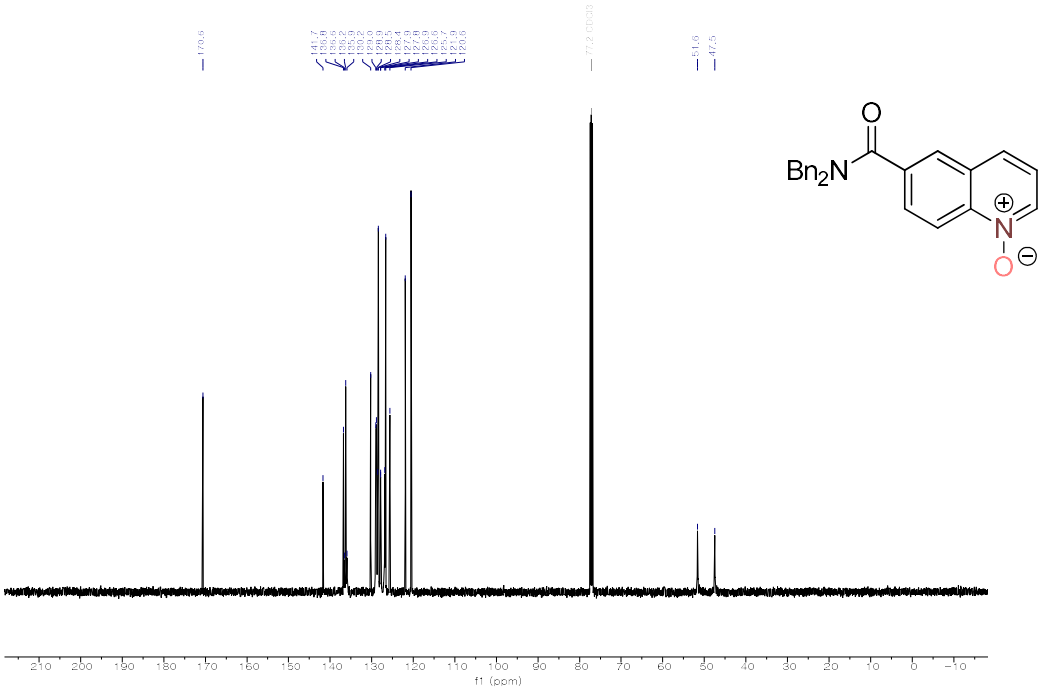


^{1}H & ^{13}C NMR Spectra of *N*,*N*-Dibenzyl-6-quinolinecarboxamide *N*-Oxide (1j)

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

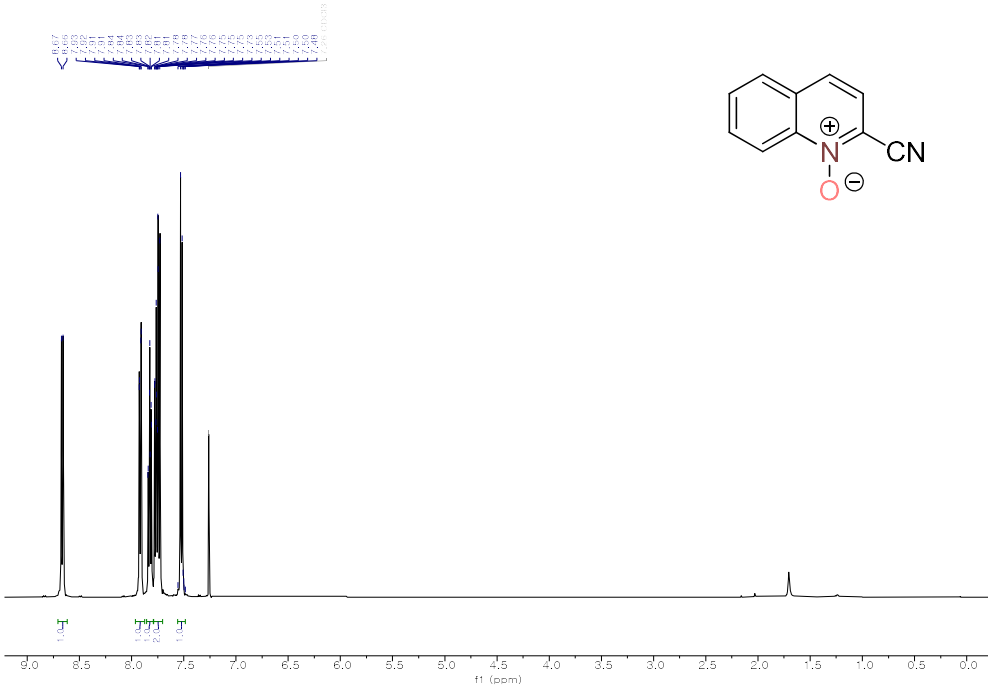


^{13}C NMR (126 MHz, $CDCl_3$)

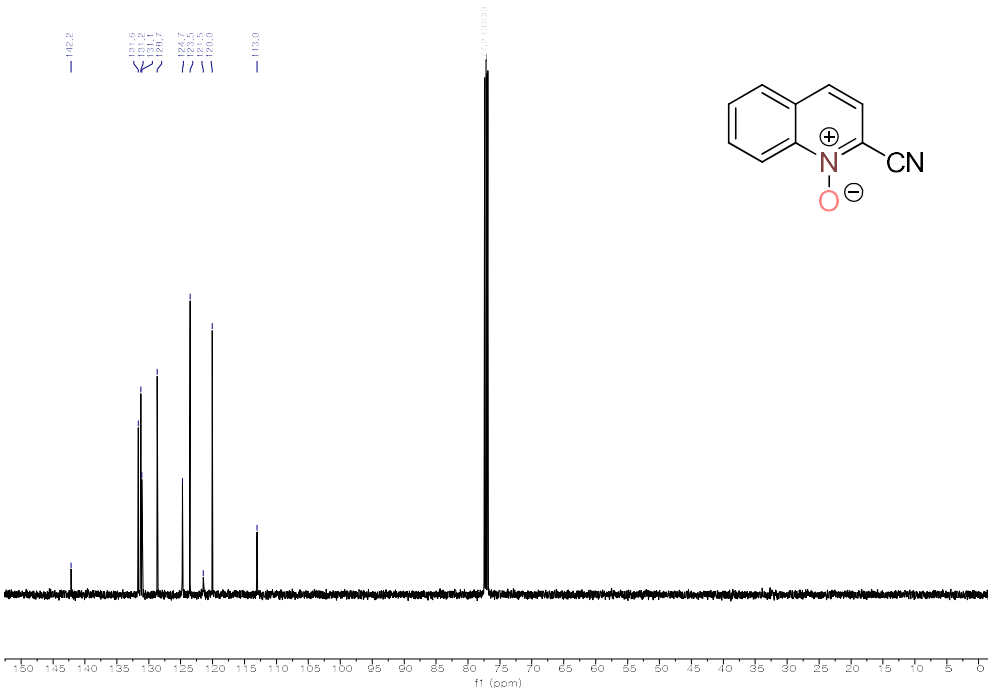


^{1}H & ^{13}C NMR Spectra of 2-Quinolinecarbonitrile *N*-Oxide(1k)

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

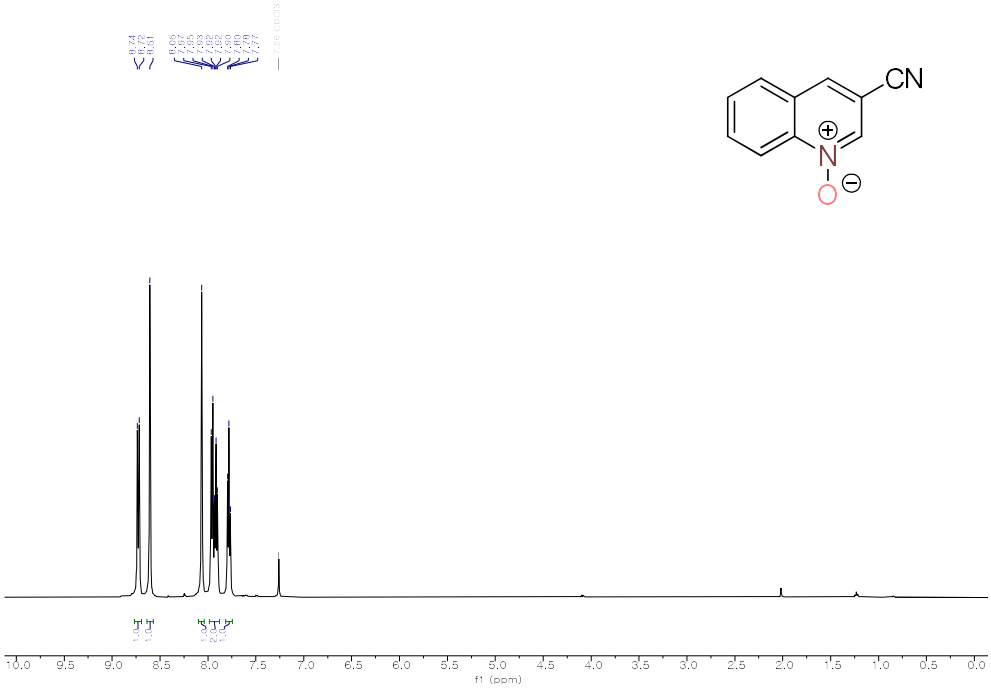


^{13}C NMR (126 MHz, $CDCl_3$)

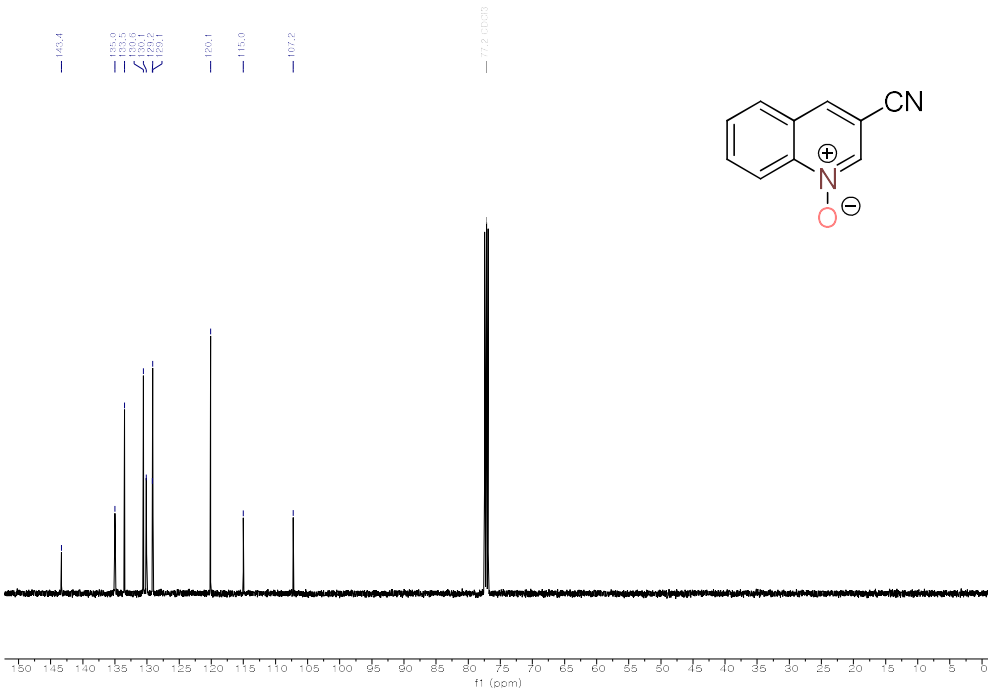


^{1}H & ^{13}C NMR Spectra of 3-Quinolinecarbonitrile *N*-Oxide (1l)

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

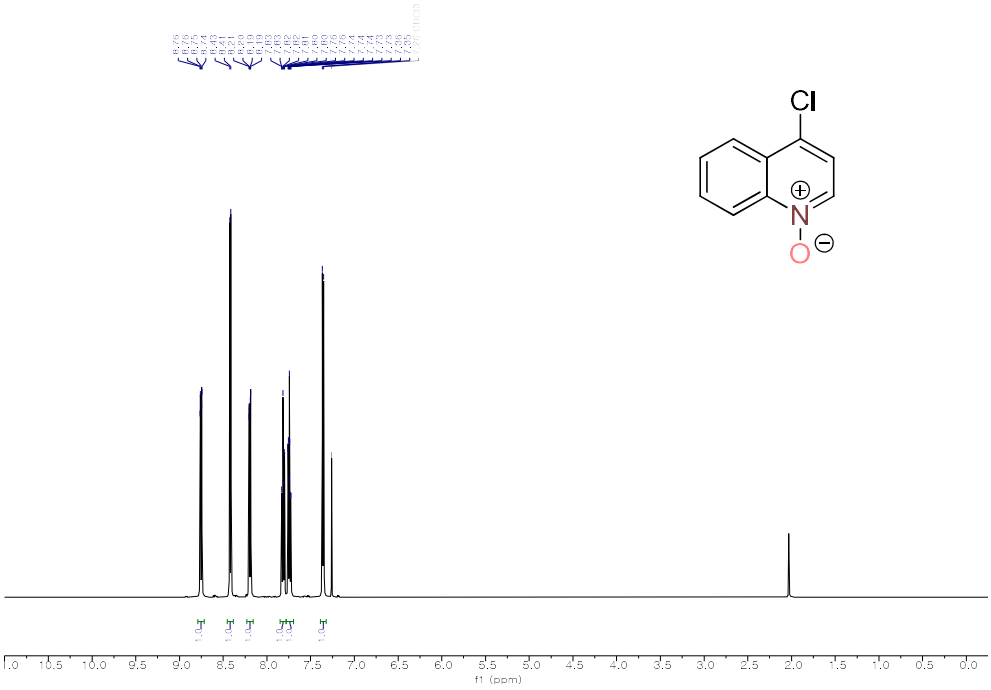


^{13}C NMR (126 MHz, $CDCl_3$)

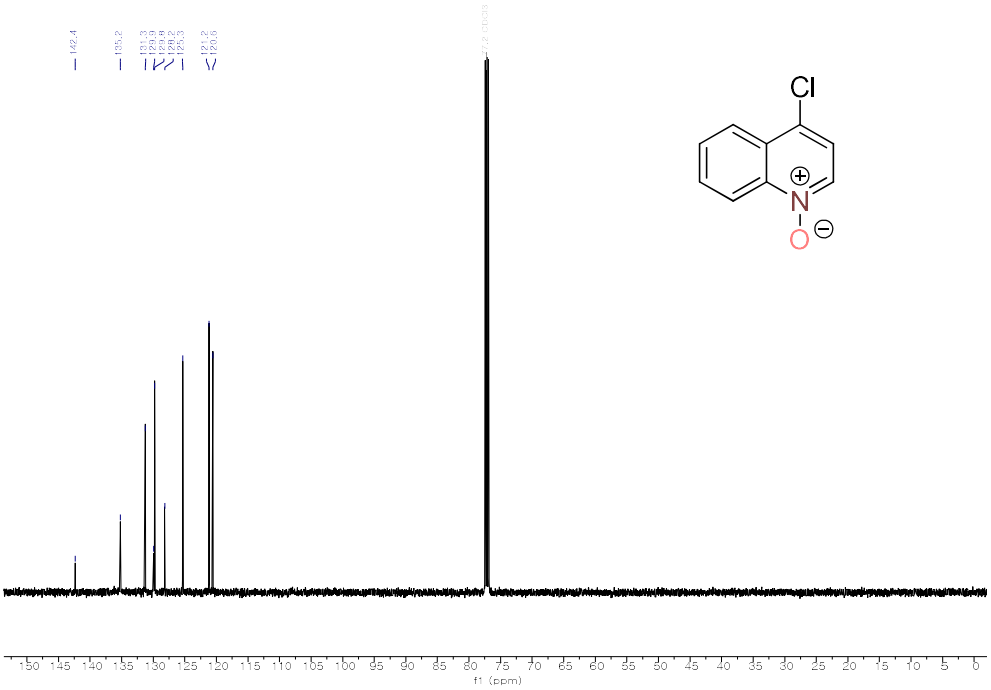


^{1}H & ^{13}C NMR Spectra of 4-Chloroquinoline *N*-Oxide (1m)

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

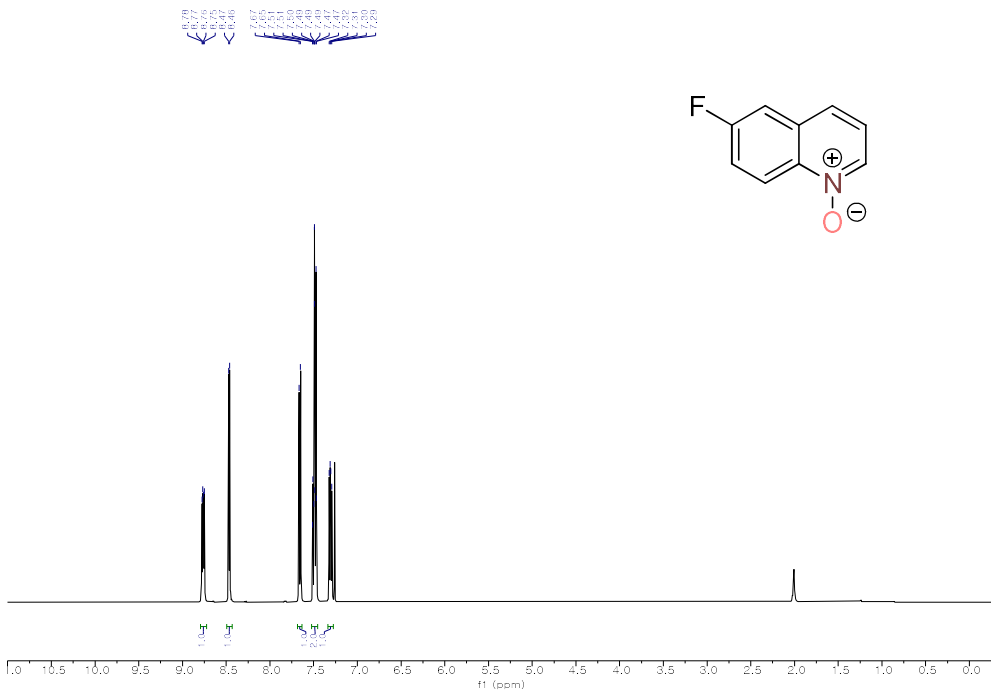


^{13}C NMR (126 MHz, $CDCl_3$)

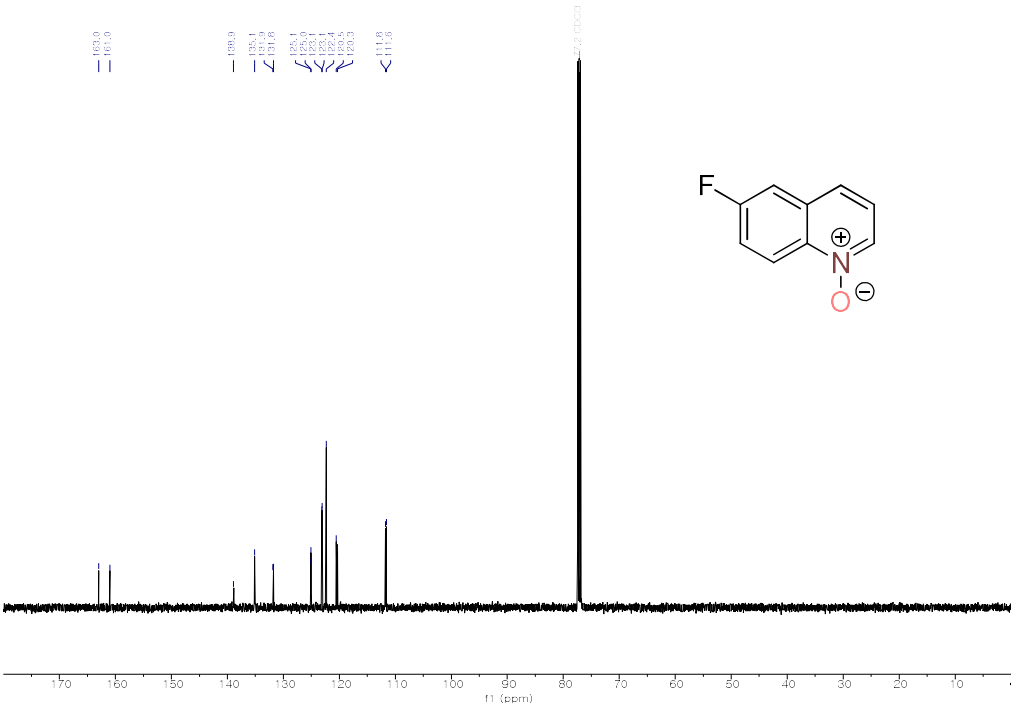


^{1}H & ^{13}C & ^{19}F NMR Spectra of 6-Fluoroquinoline *N*-Oxide (1n)

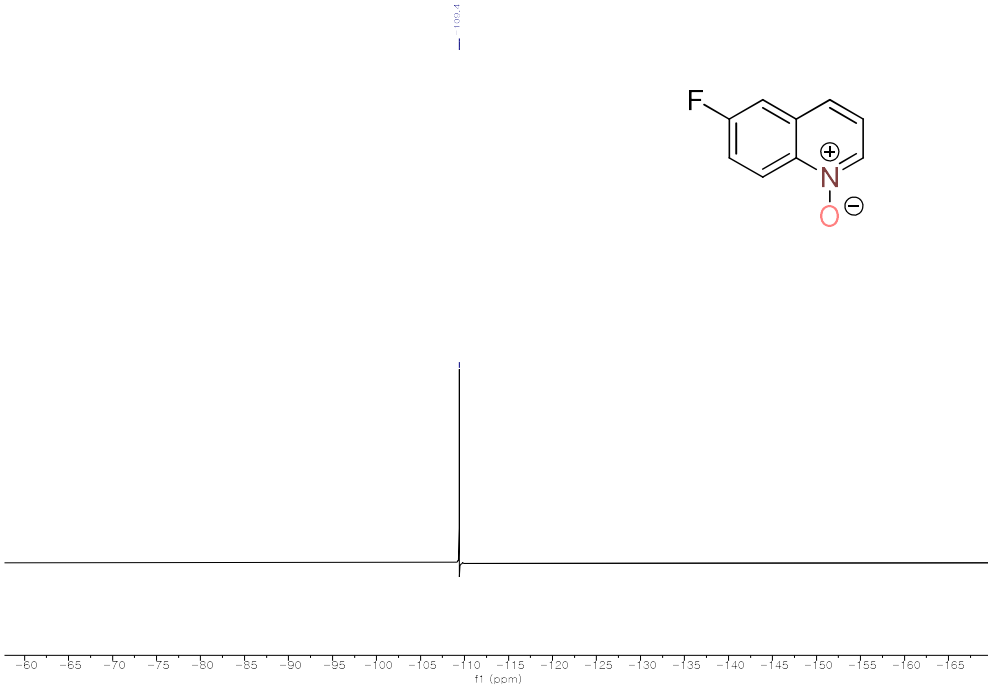
^{1}H NMR (500 MHz, $CDCl_3$)



^{13}C NMR (126 MHz, $CDCl_3$)

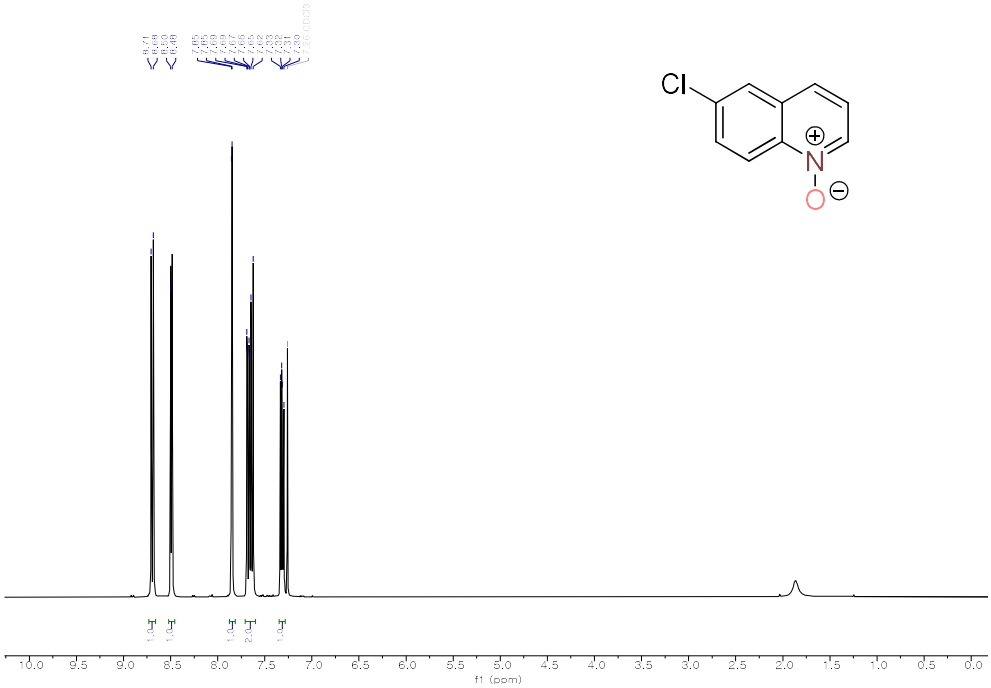


^{19}F NMR (471 MHz, $CDCl_3$)

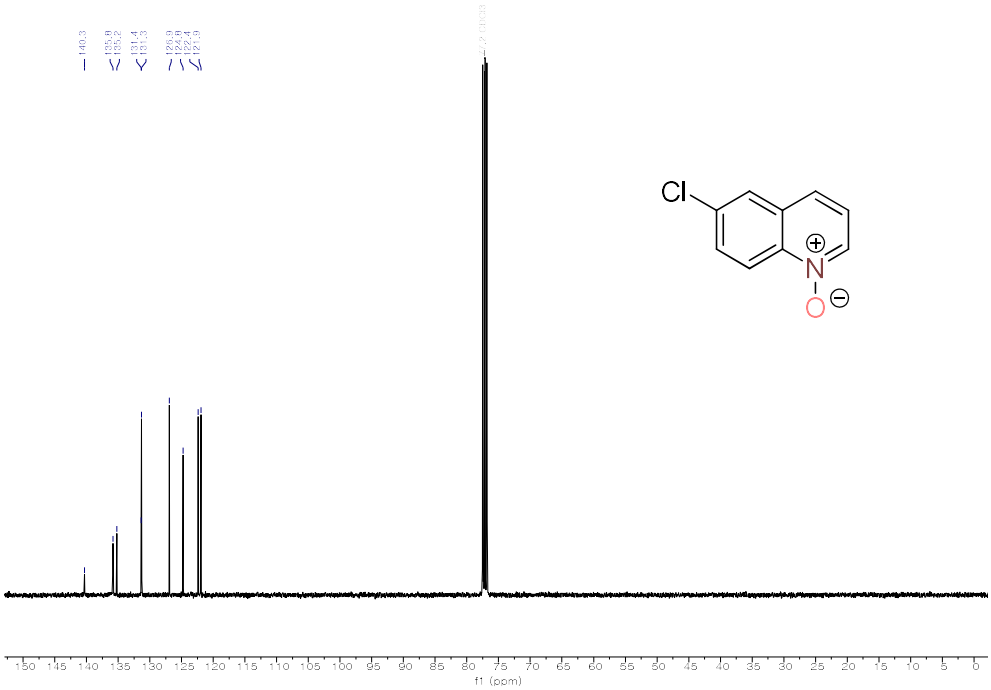


^{1}H & ^{13}C NMR Spectra of 6-Chloroquinoline *N*-Oxide (1o)

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

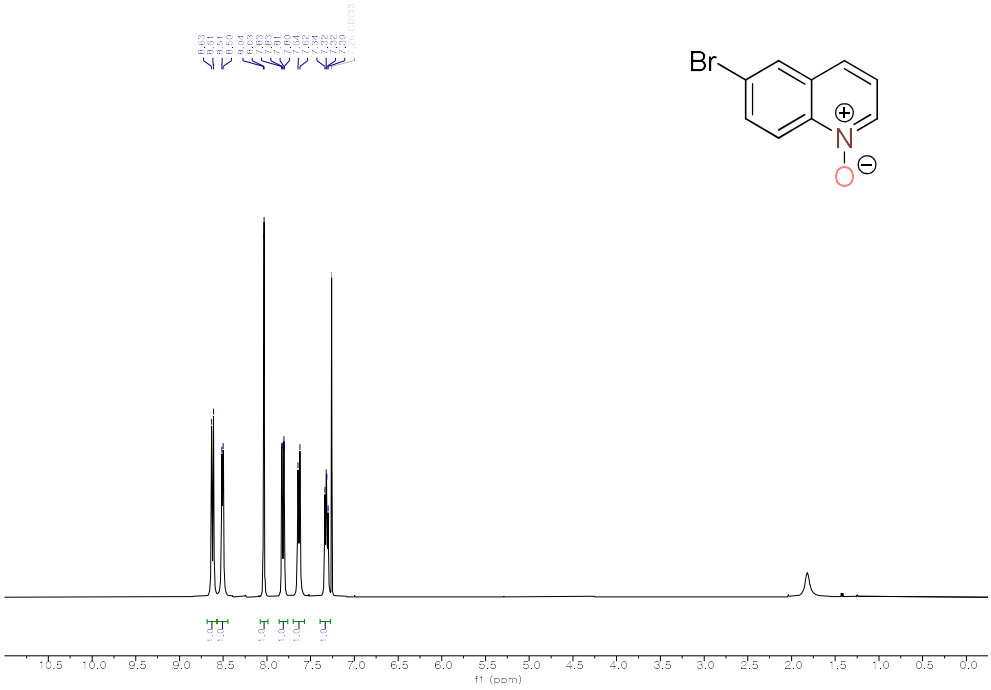


^{13}C NMR (101 MHz, $CDCl_3$)

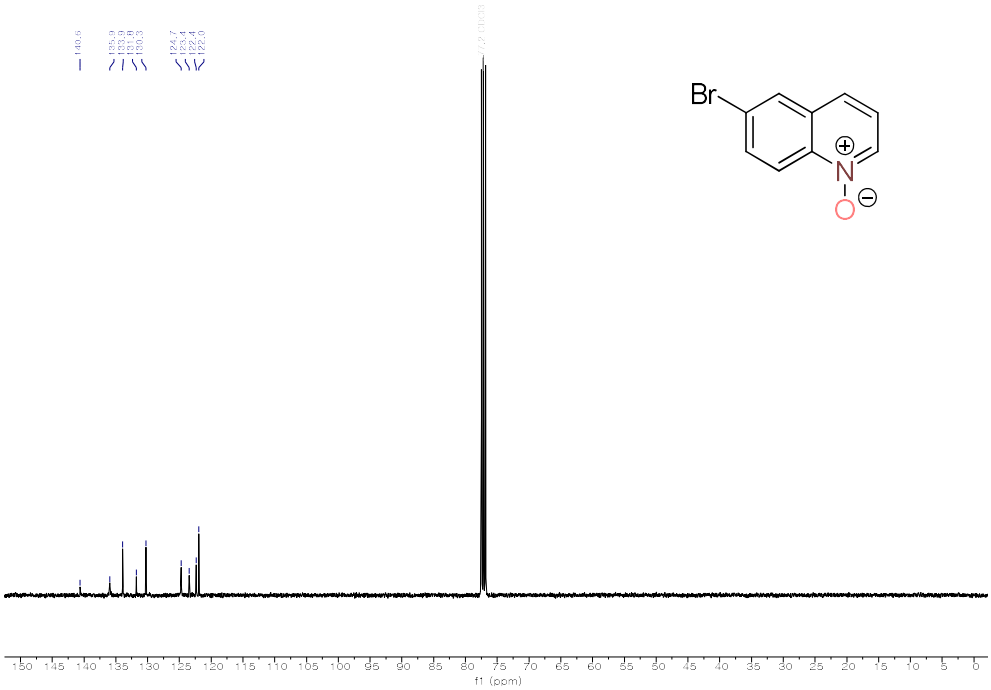


^{1}H & ^{13}C NMR Spectra of 6-Bromoquinoline *N*-Oxide (1p)

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

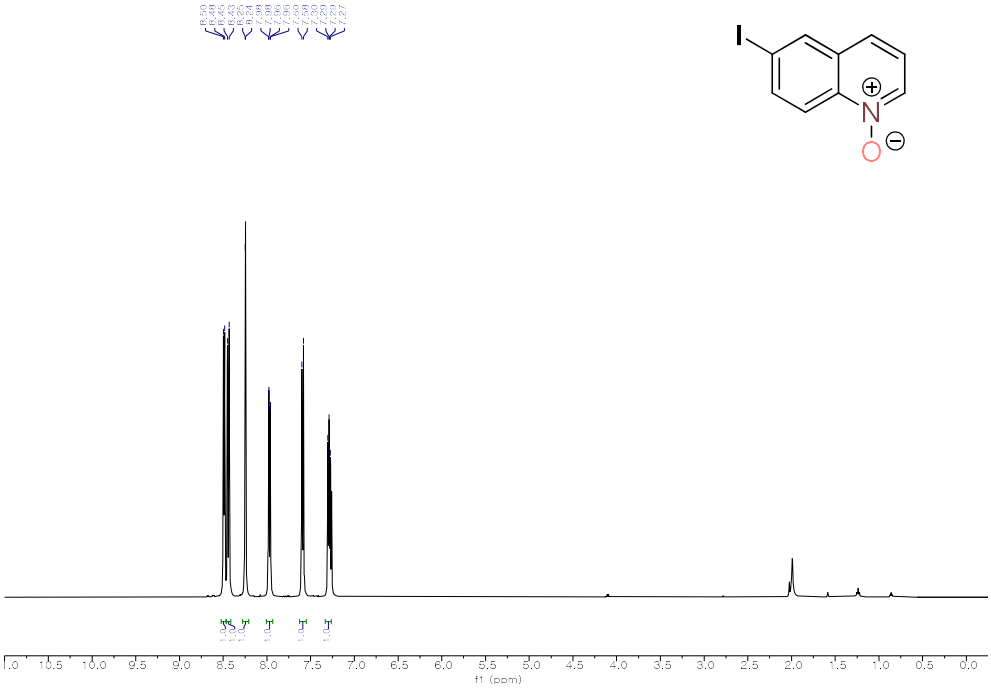


^{13}C NMR (101 MHz, $CDCl_3$)

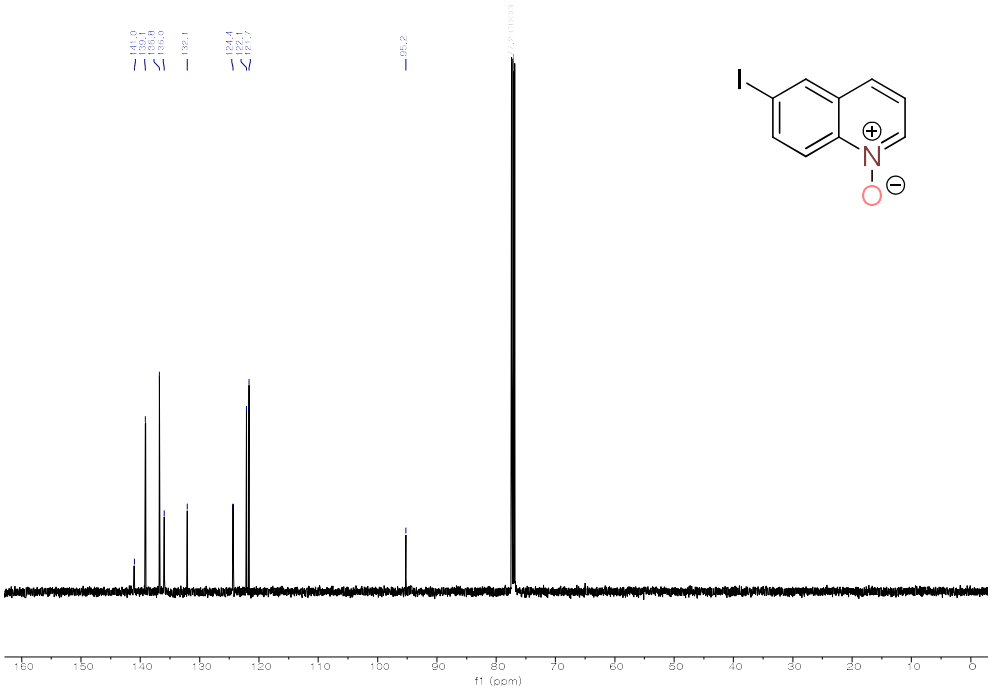


^{1}H & ^{13}C NMR Spectra of 6-Iodoquinoline *N*-Oxide (1q)

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

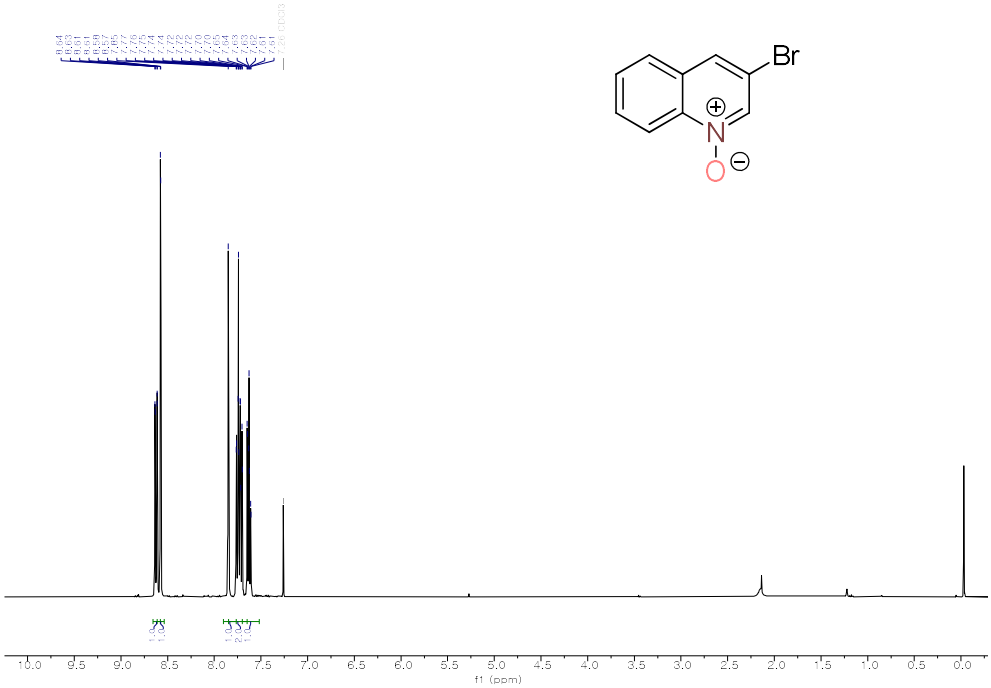


^{13}C NMR (126 MHz, $CDCl_3$)

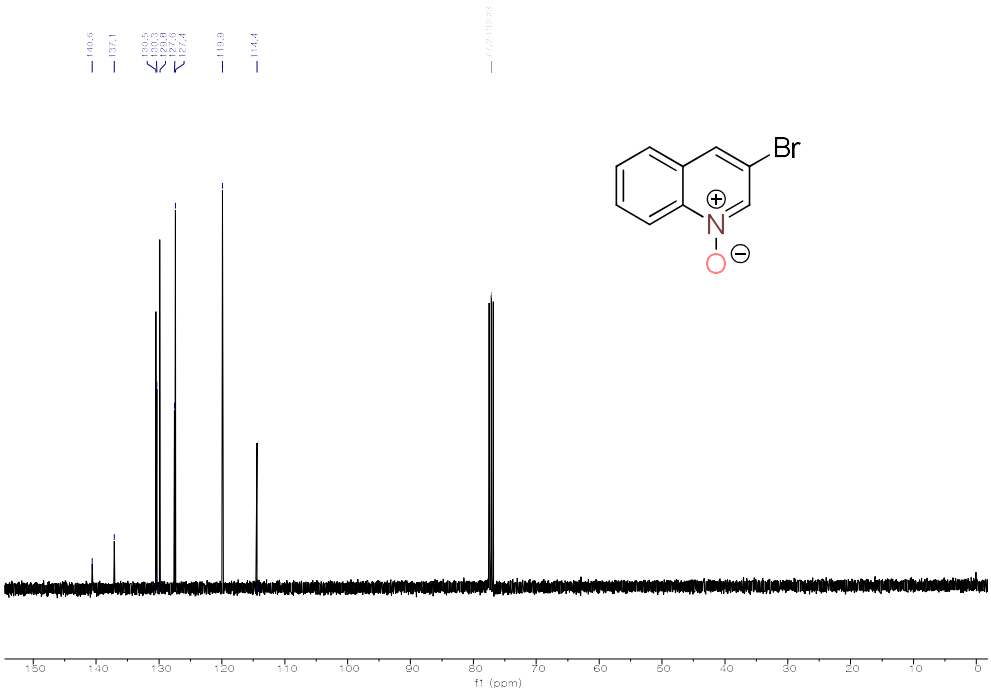


^{1}H & ^{13}C NMR Spectra of 3-Bromoquinoline *N*-Oxide (1r)

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

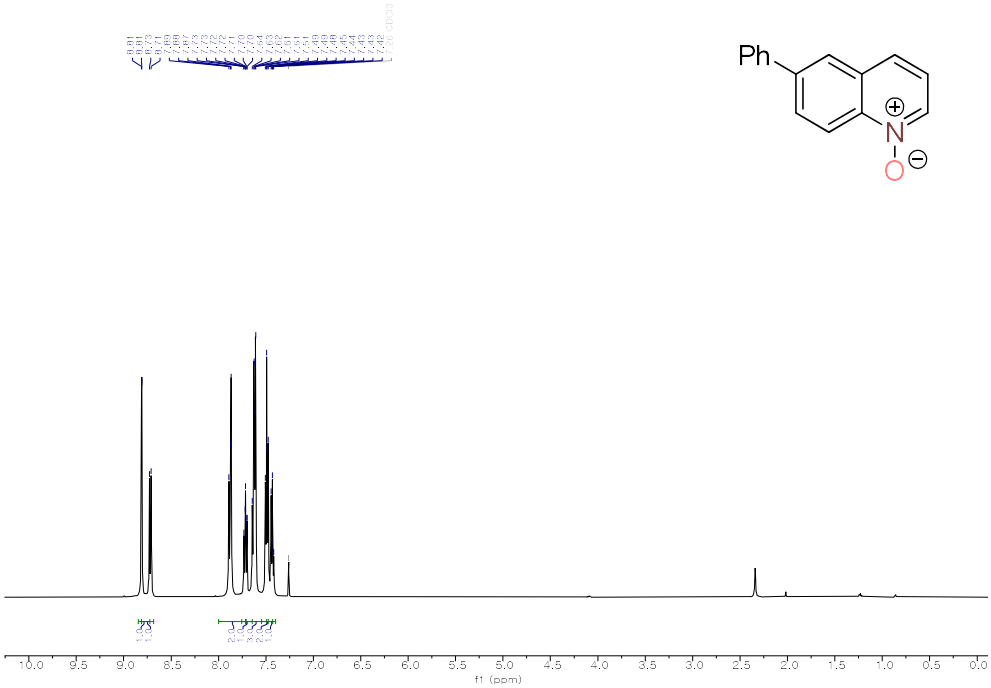


^{13}C NMR (101 MHz, $CDCl_3$)

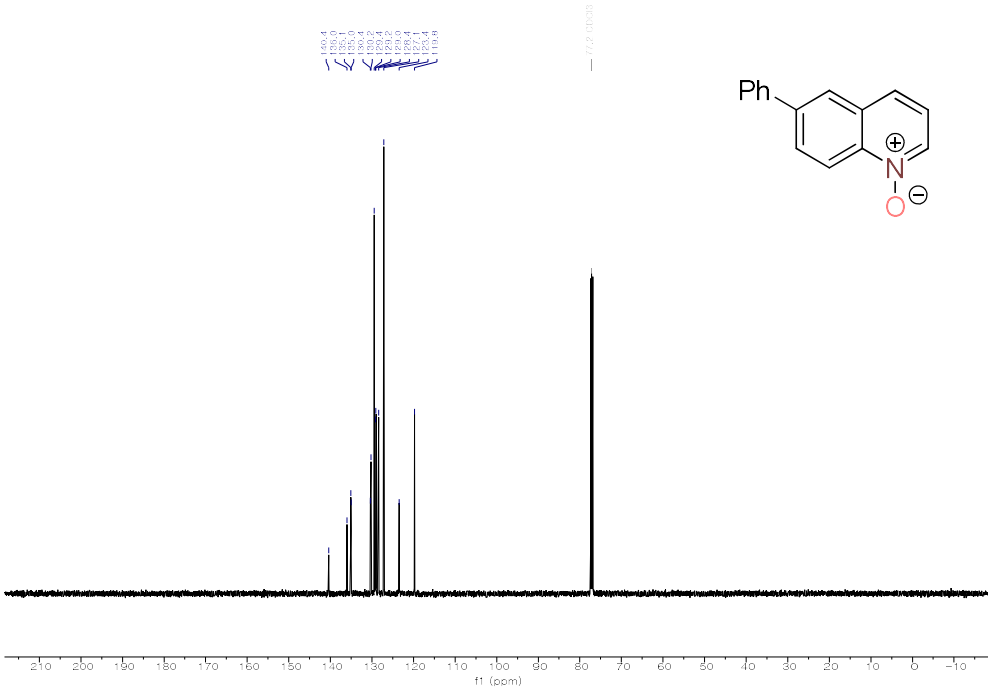


^{1}H & ^{13}C NMR Spectra of 6-Phenylquinoline *N*-Oxide (1s)

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

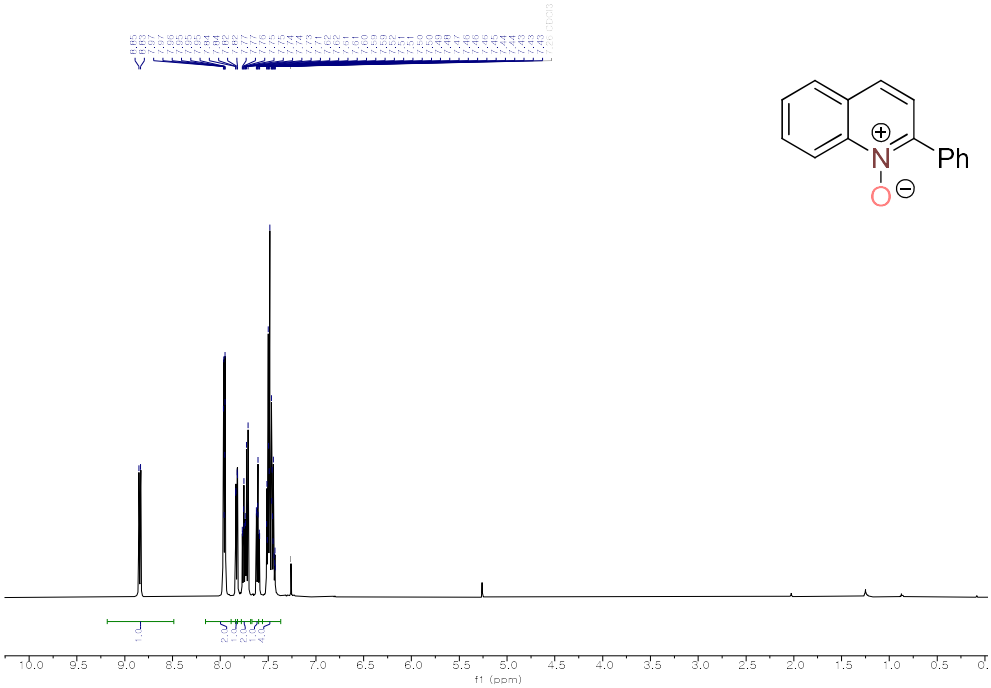


^{13}C NMR (126 MHz, $CDCl_3$)

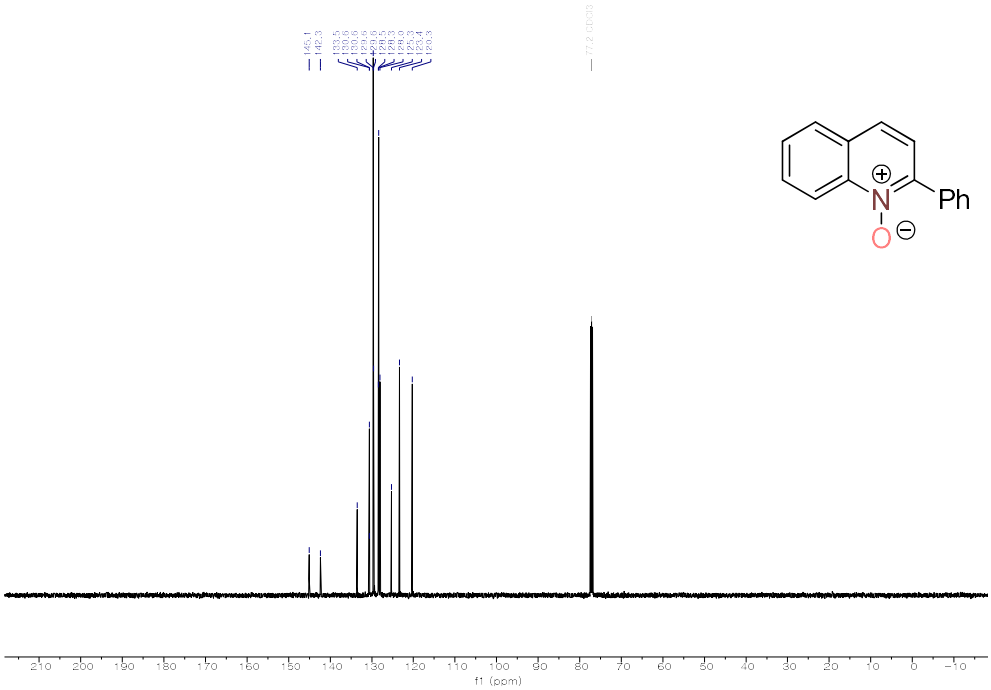


^{1}H & ^{13}C NMR Spectra of 2-Phenylquinoline *N*-Oxide (1t)

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

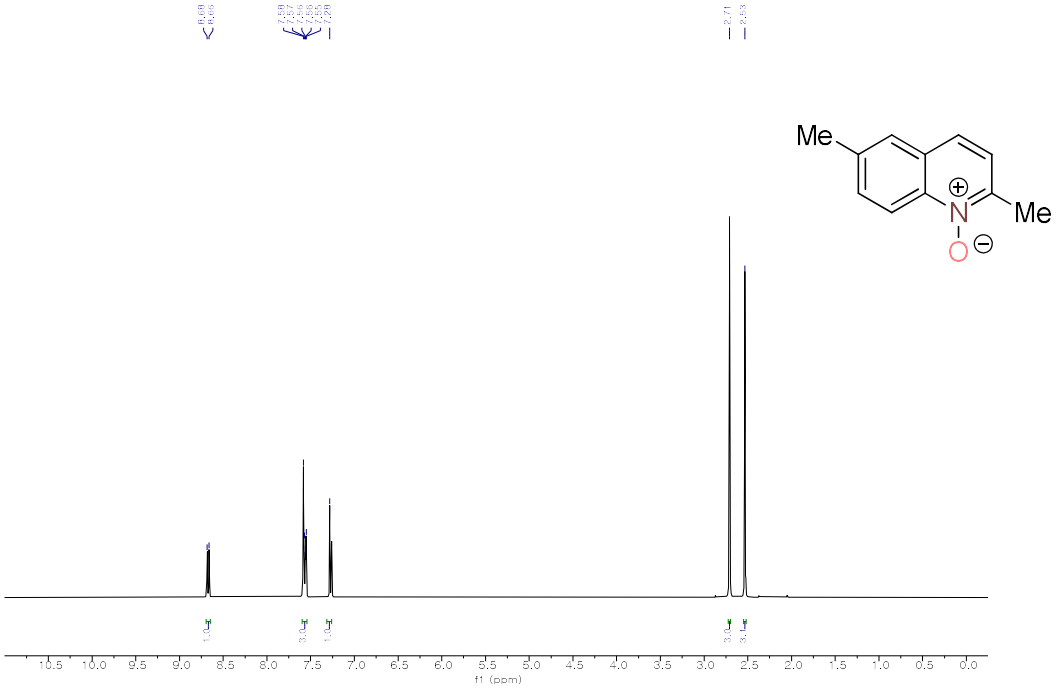


^{13}C NMR (126 MHz, $CDCl_3$)

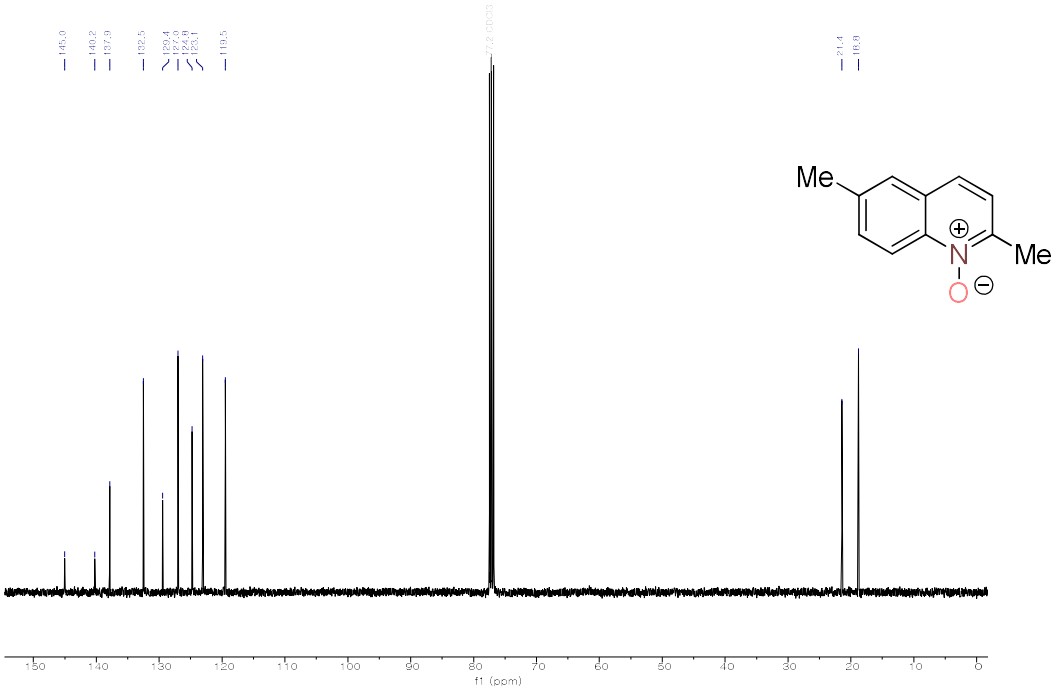


^{1}H & ^{13}C NMR Spectra of 2,6-Dimethylquinoline *N*-Oxide (1u)

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

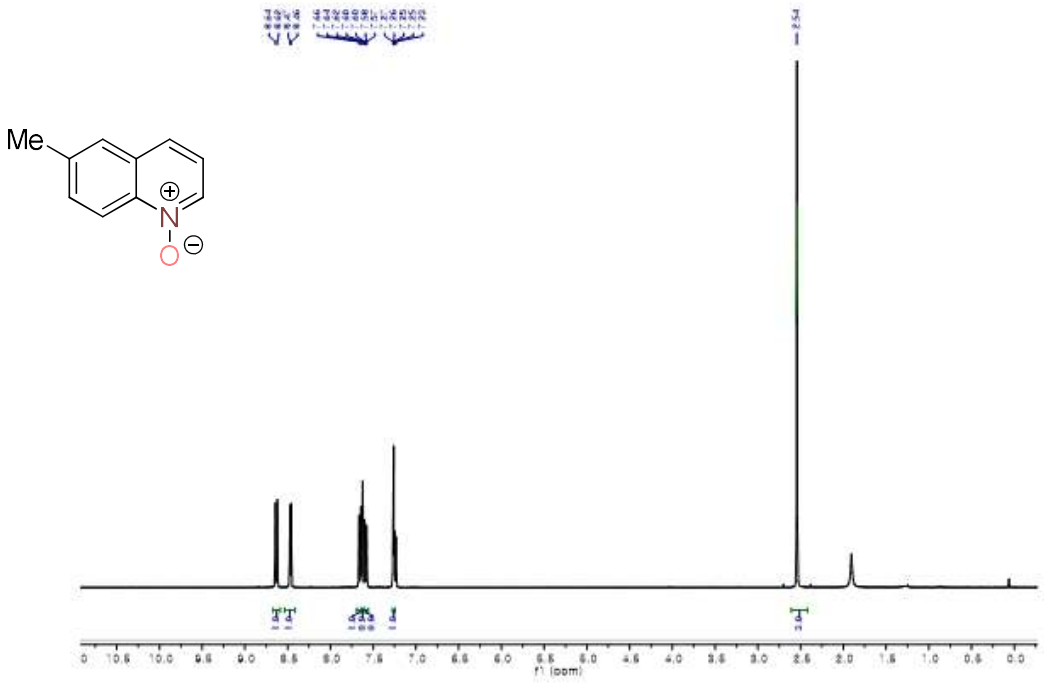


^{13}C NMR (101 MHz, $CDCl_3$)

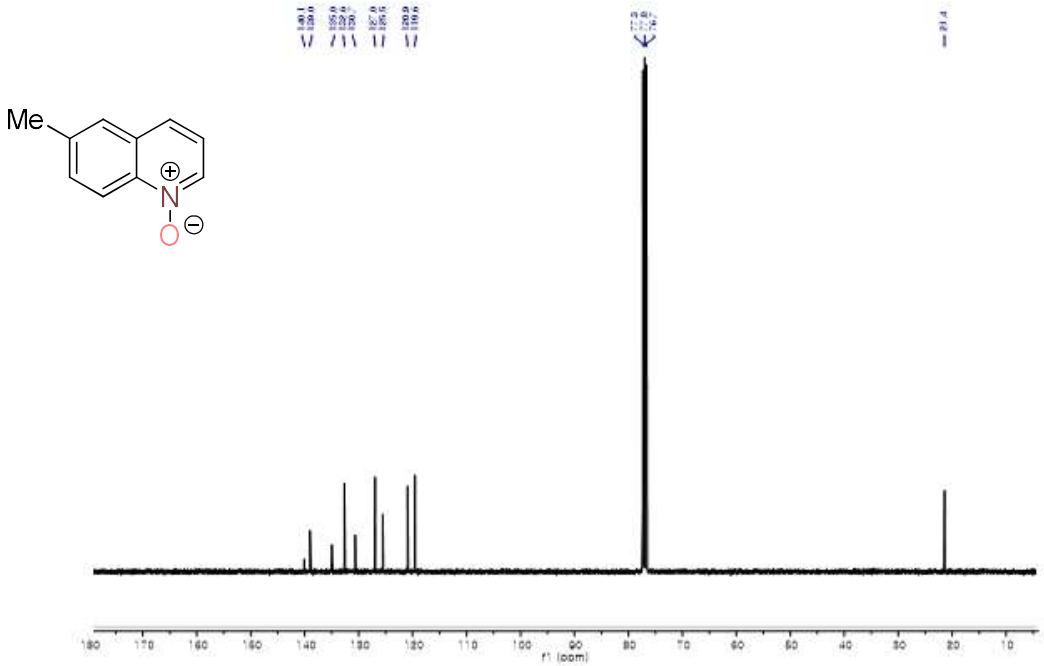


^{1}H & ^{13}C NMR Spectra of 6-Methylquinoline *N*-Oxide (1v)

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

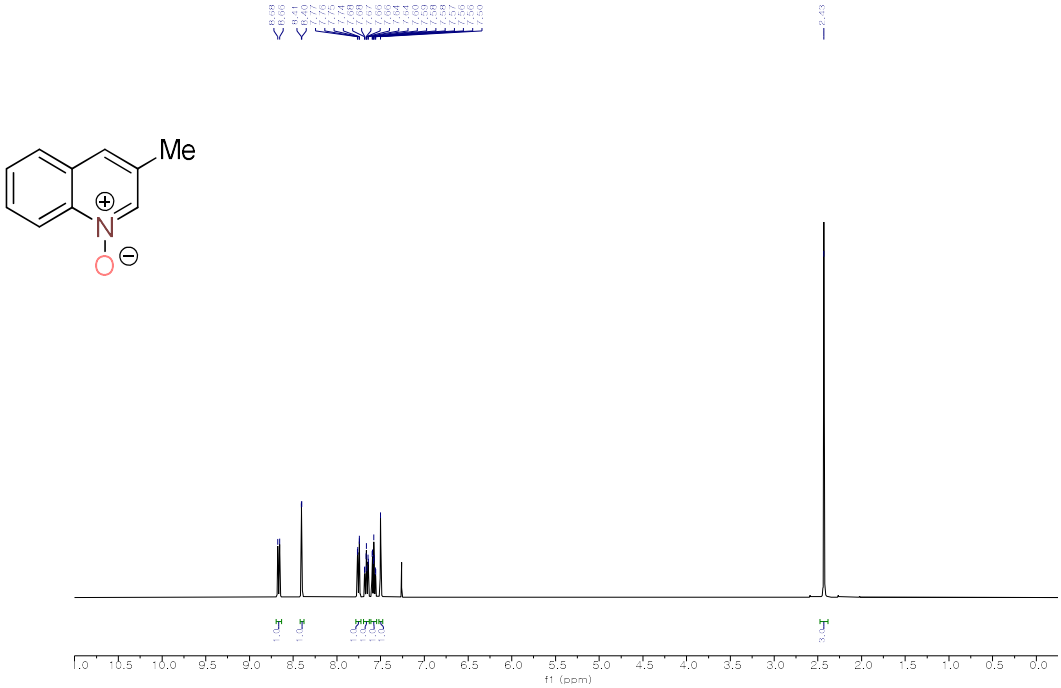


^{13}C NMR (101 MHz, $CDCl_3$)

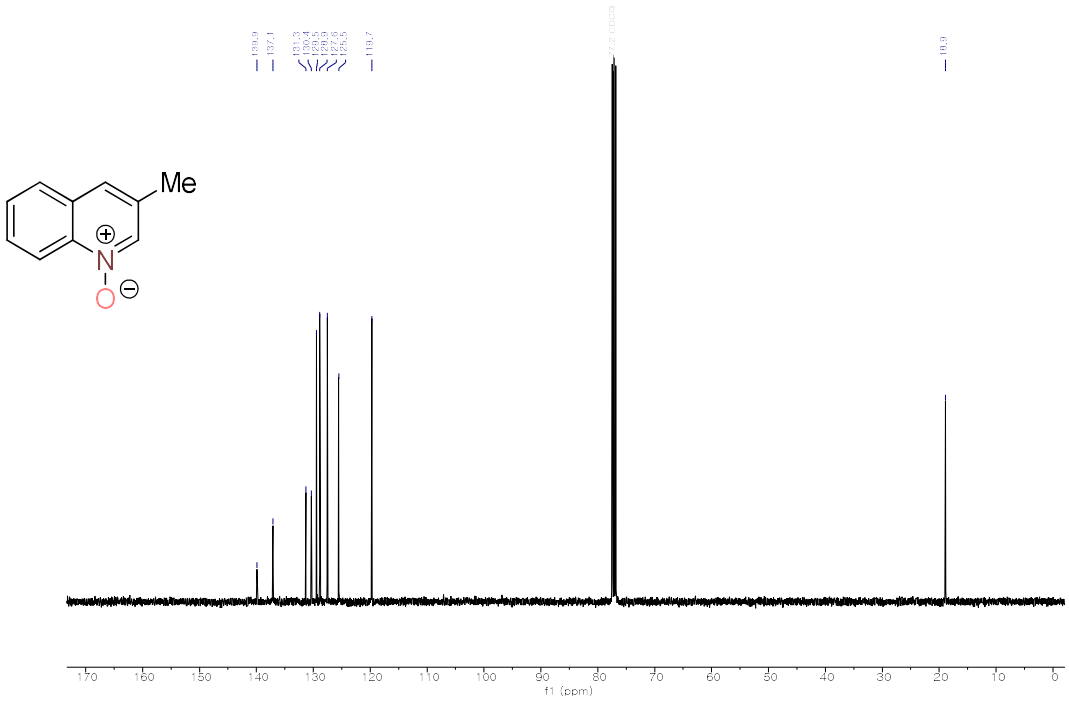


^{1}H & ^{13}C NMR Spectra of 3-Methylquinoline *N*-Oxide (1w)

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

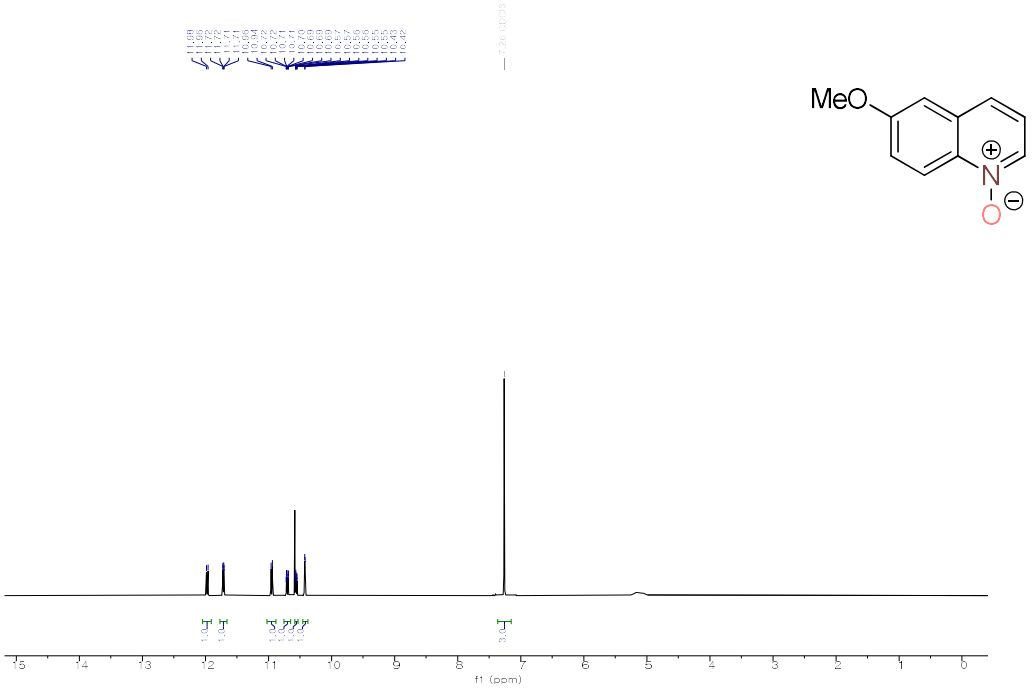


^{13}C NMR (101 MHz, $CDCl_3$)

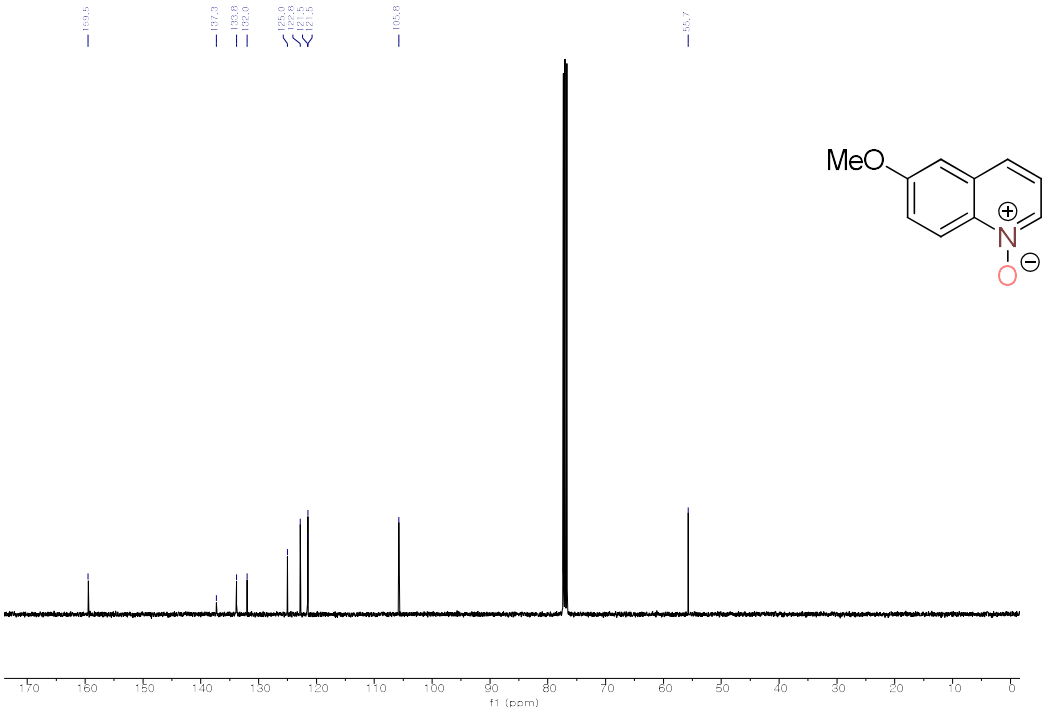


^{1}H & ^{13}C NMR Spectra of 6-Methoxyquinoline *N*-Oxide (1x)

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

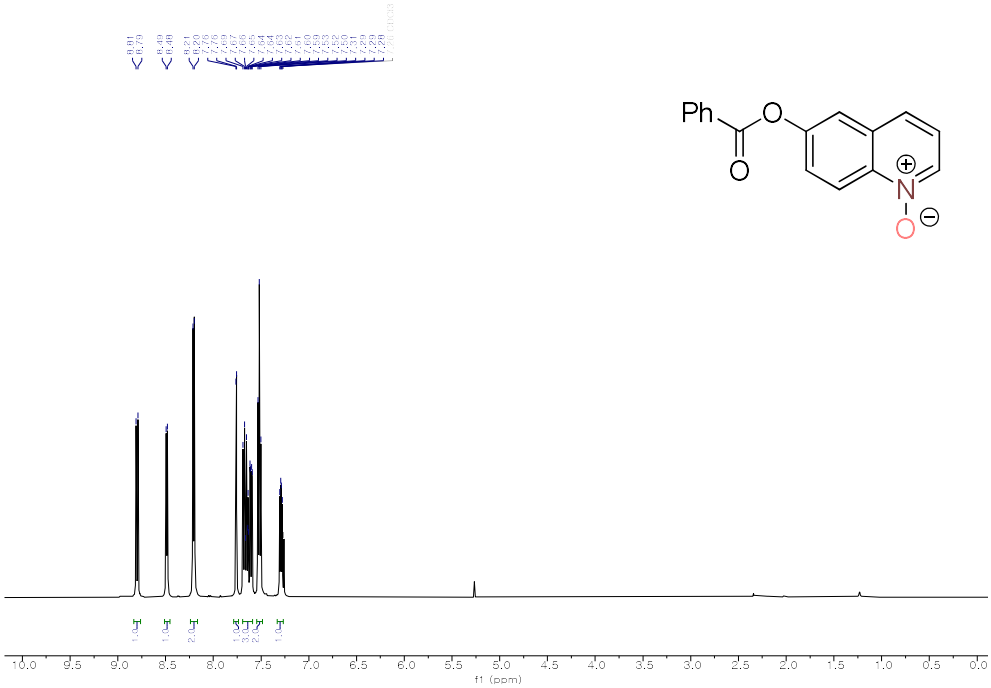


^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H & ^{13}C NMR Spectra of 6-Benzoyloxyquinoline *N*-Oxide (1y)

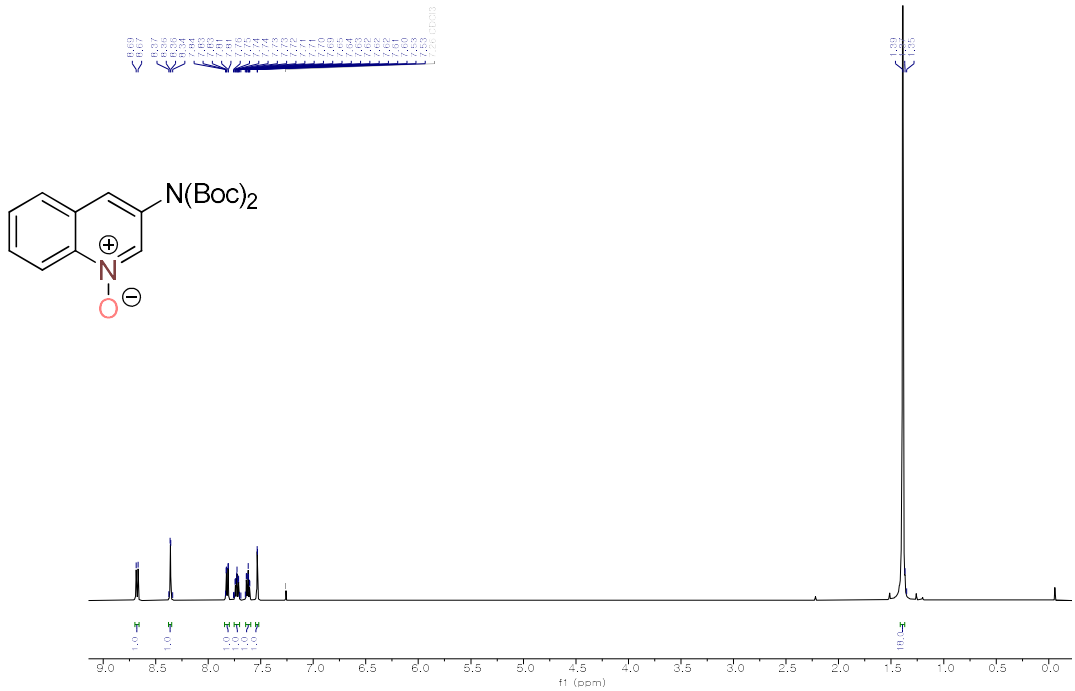
^{1}H NMR (500 MHz, $CDCl_3$)



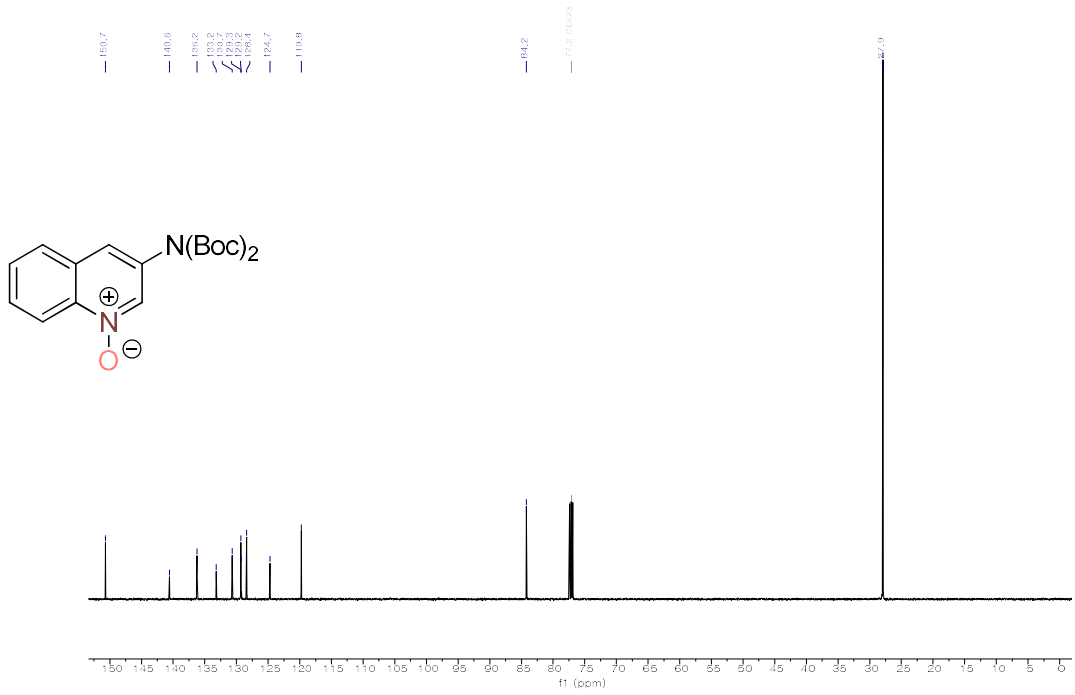
^{13}C NMR (126 MHz, $CDCl_3$)

^{1}H & ^{13}C NMR Spectra of 3-[Bis(*tert*-butoxycarbonyl)amino]quinoline *N*-Oxide (1z)

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

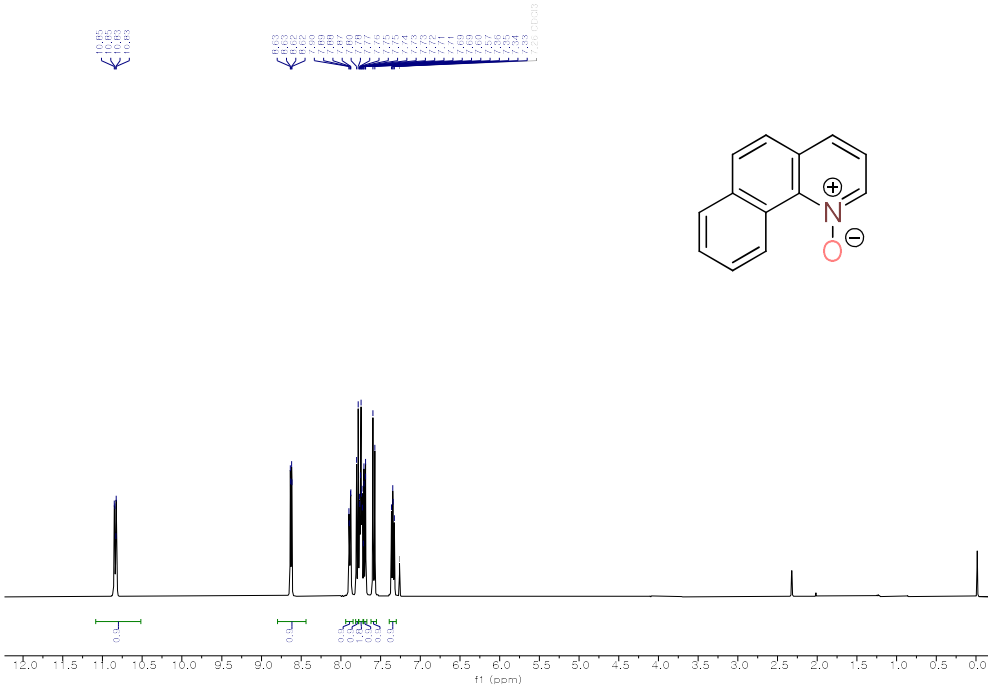


^{13}C NMR (126 MHz, $CDCl_3$)

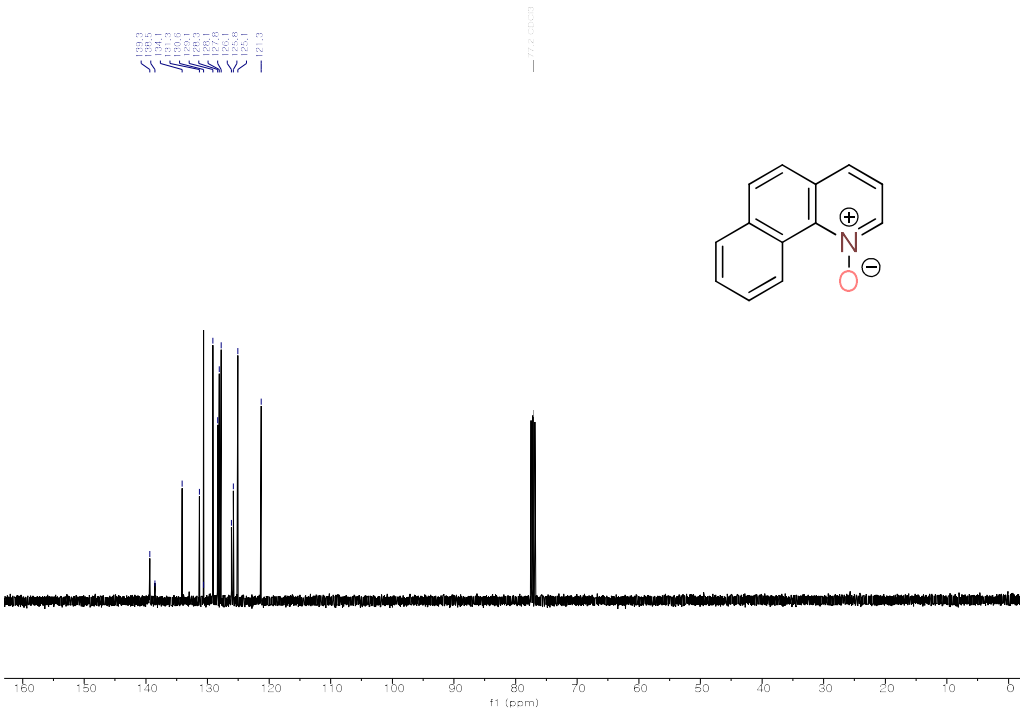


^{1}H & ^{13}C NMR Spectra of Benzo[*h*]quinoline *N*-Oxide (1aa)

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

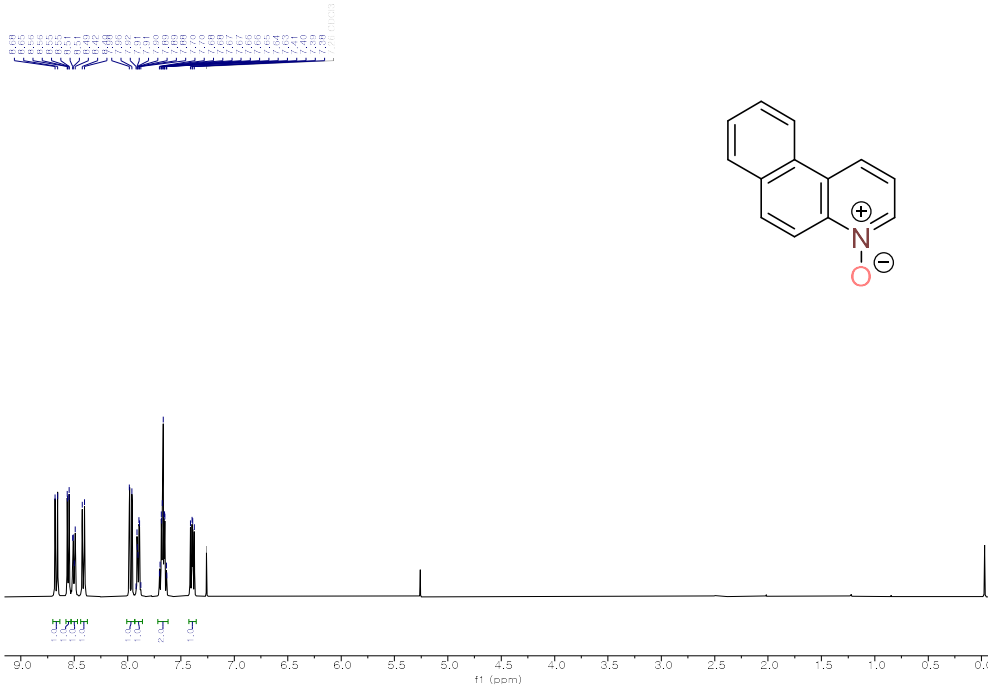


^{13}C NMR (101 MHz, $CDCl_3$)

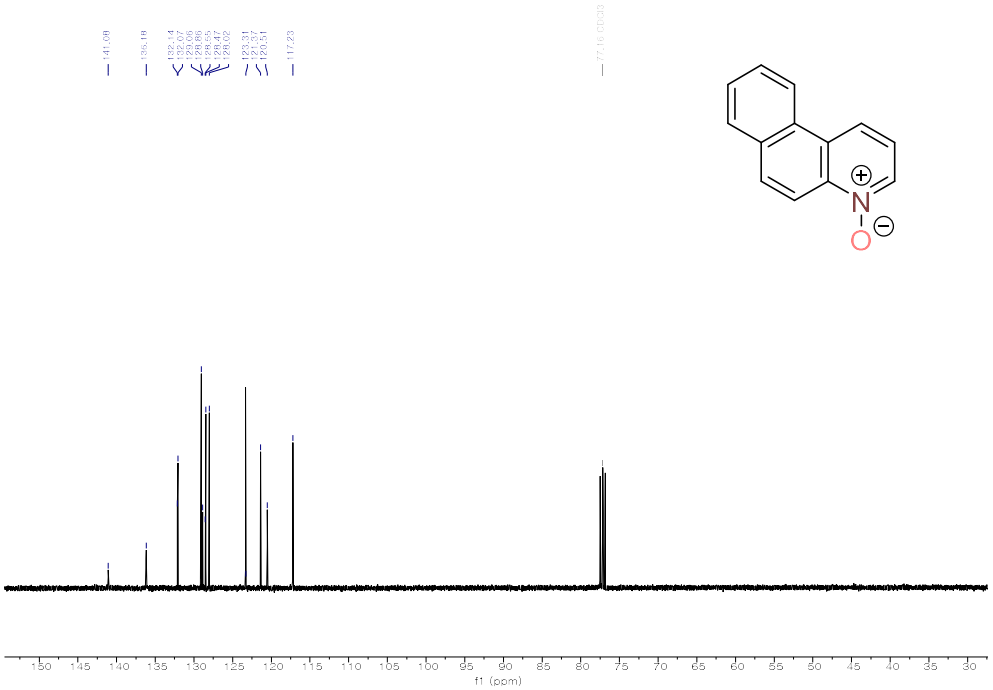


^{1}H & ^{13}C NMR Spectra of Benzo[*f*]quinoline *N*-Oxide (1ab)

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

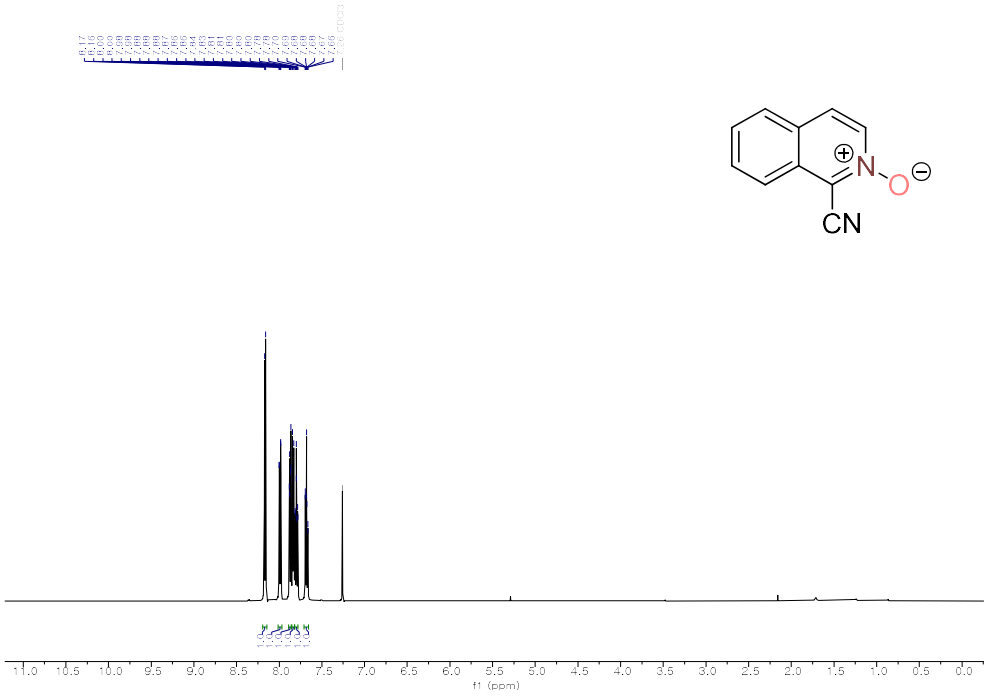


^{13}C NMR (101 MHz, $CDCl_3$)

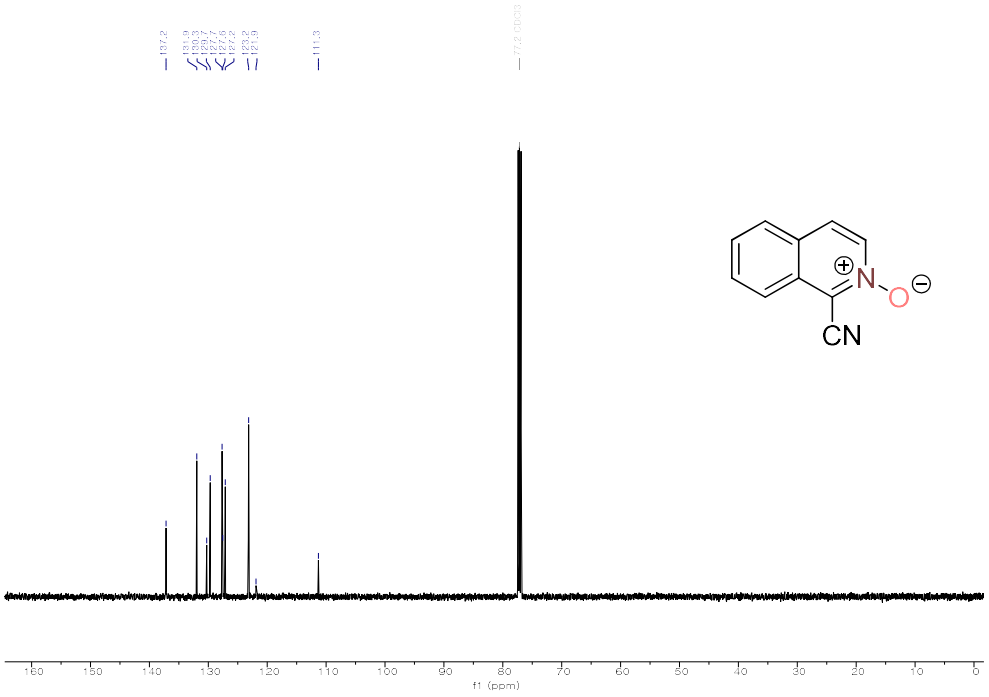


^{1}H & ^{13}C NMR Spectra of 1-Isoquinolinecarbonitrile *N*-Oxide (1ac)

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

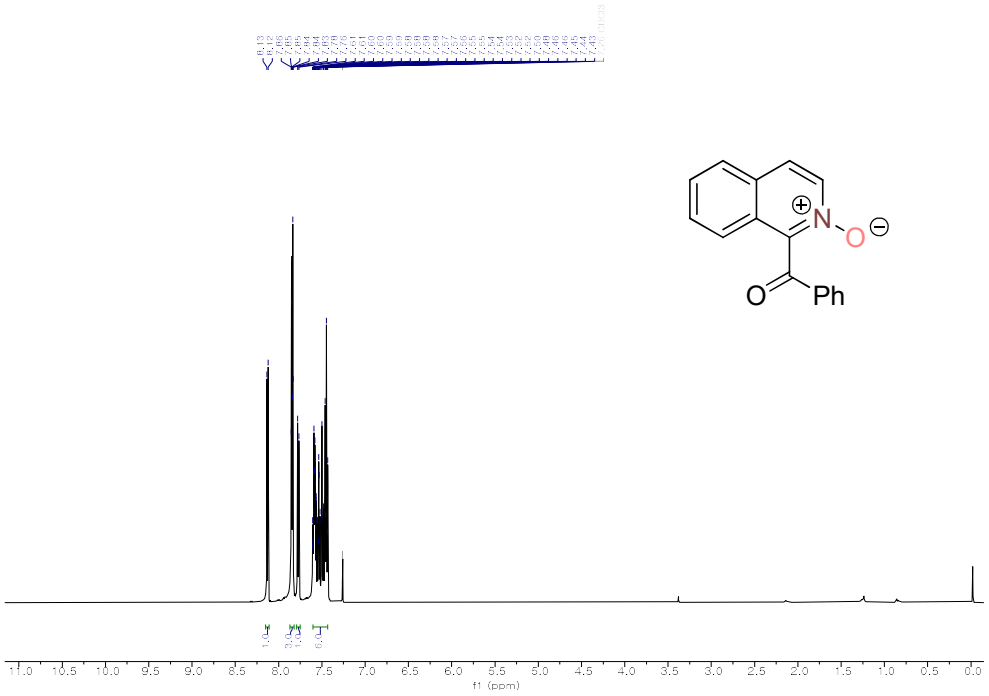


^{13}C NMR (126 MHz, $CDCl_3$)

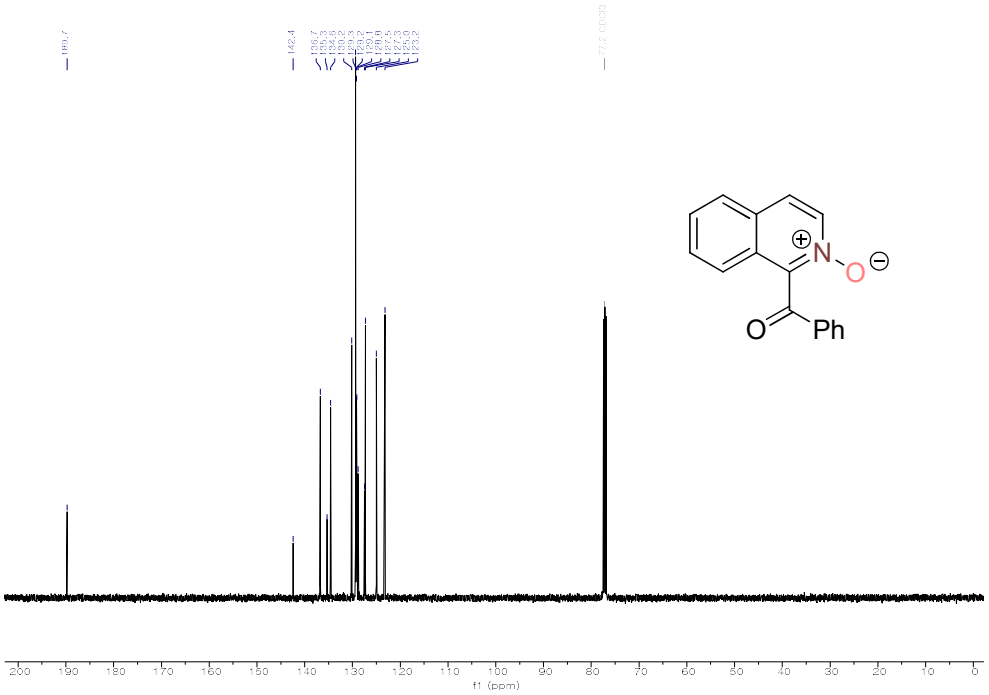


^{1}H & ^{13}C NMR Spectra of 1-Benzoylisoquinoline *N*-Oxide (1ad)

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

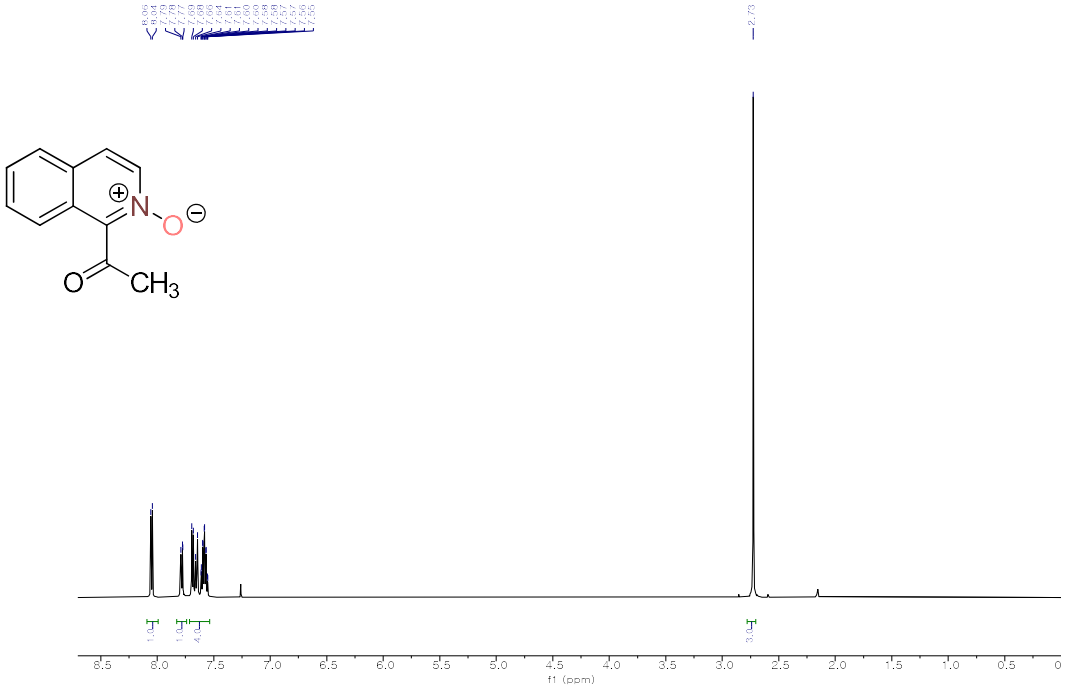


^{13}C NMR (126 MHz, $CDCl_3$)

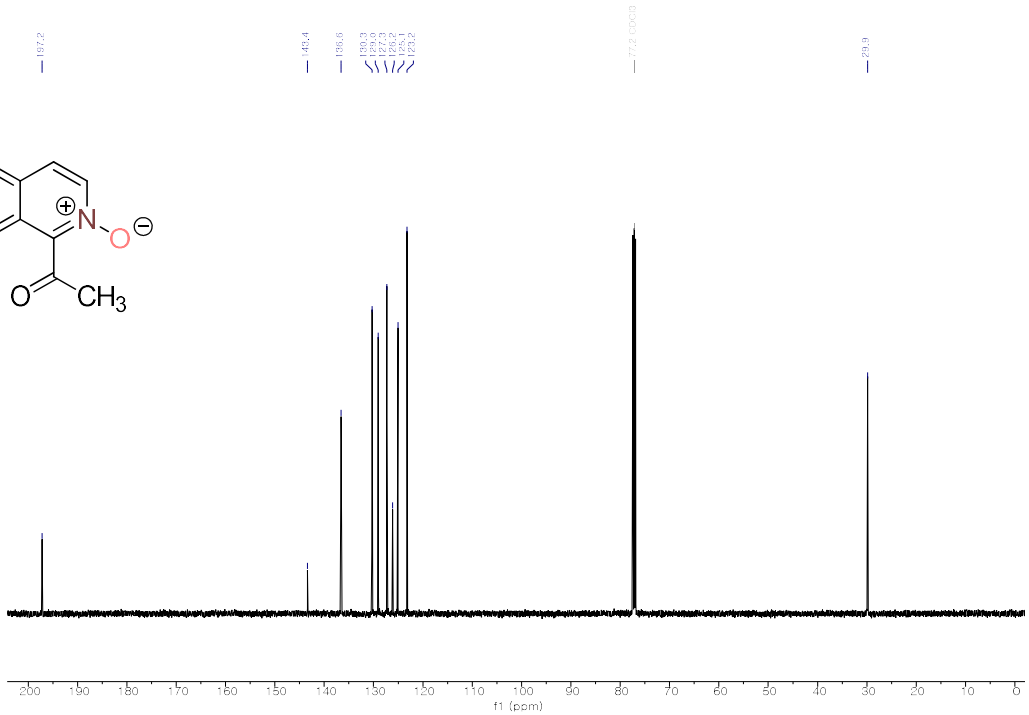


^{1}H & ^{13}C NMR Spectra of 1-Acetylisoquinoline *N*-Oxide (1ae)

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

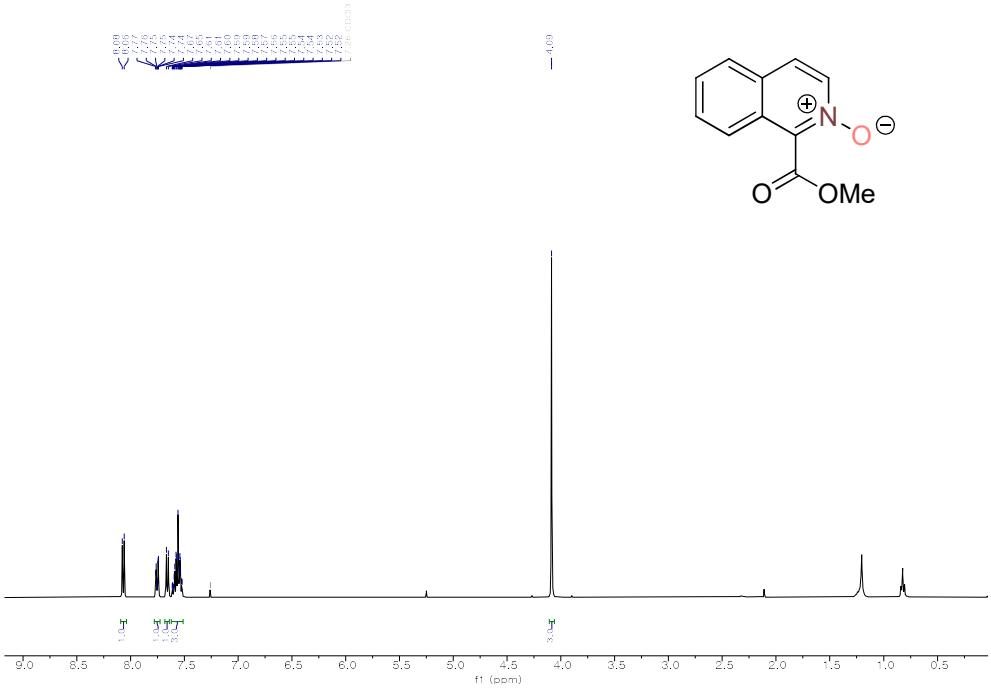


^{13}C NMR (126 MHz, $CDCl_3$)

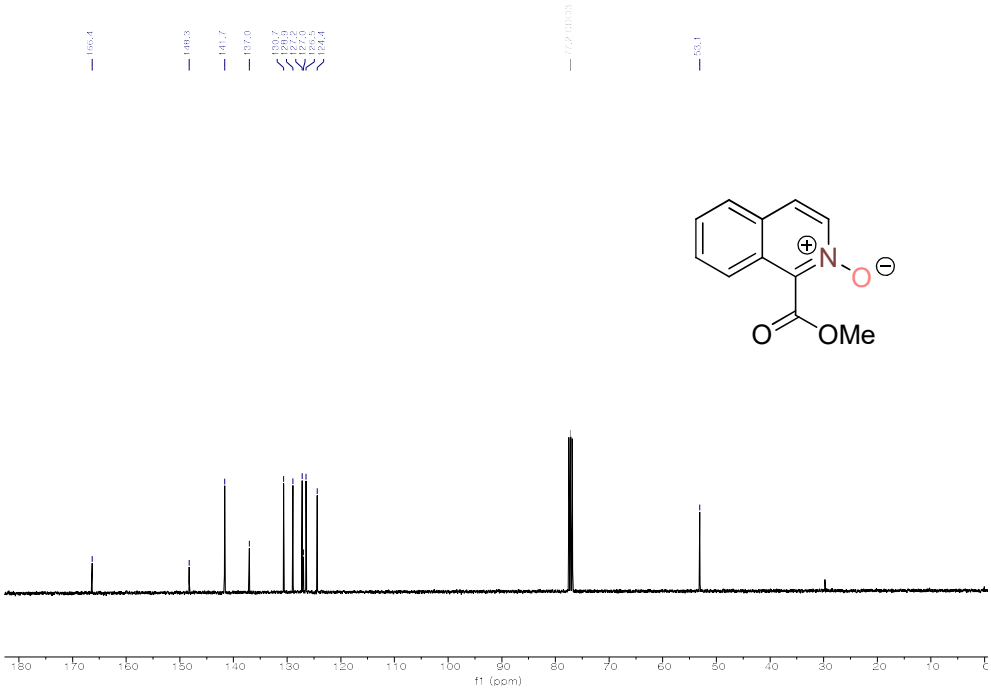


Preparation of Methyl 1-Isoquinolinecarboxylate *N*-Oxide (1af)

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

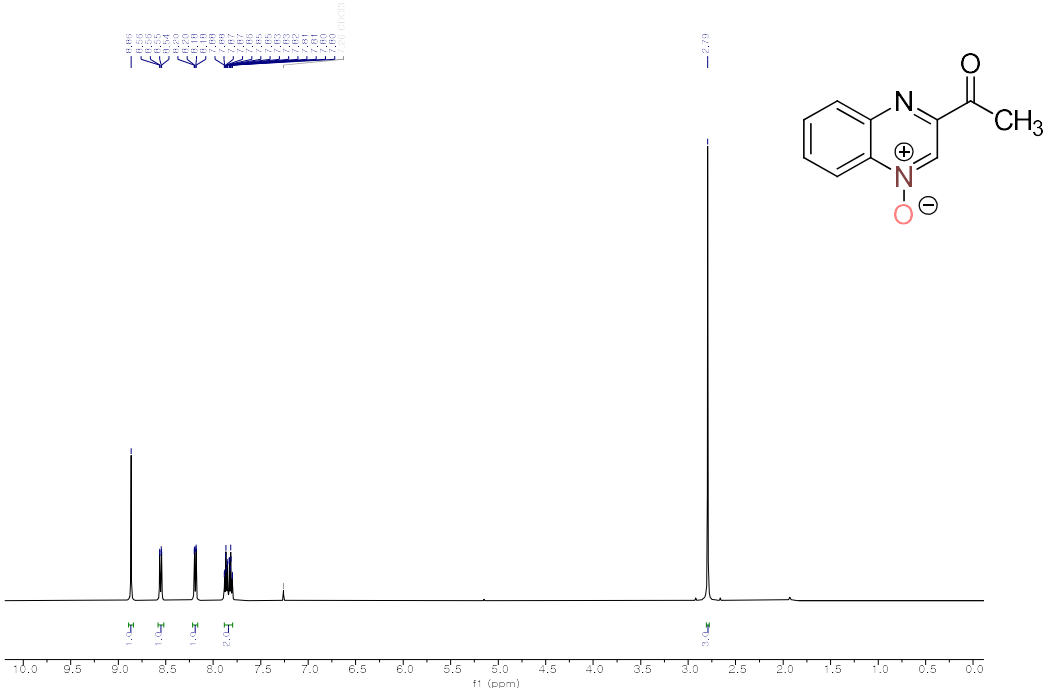


^{13}C NMR (101 MHz, $CDCl_3$)

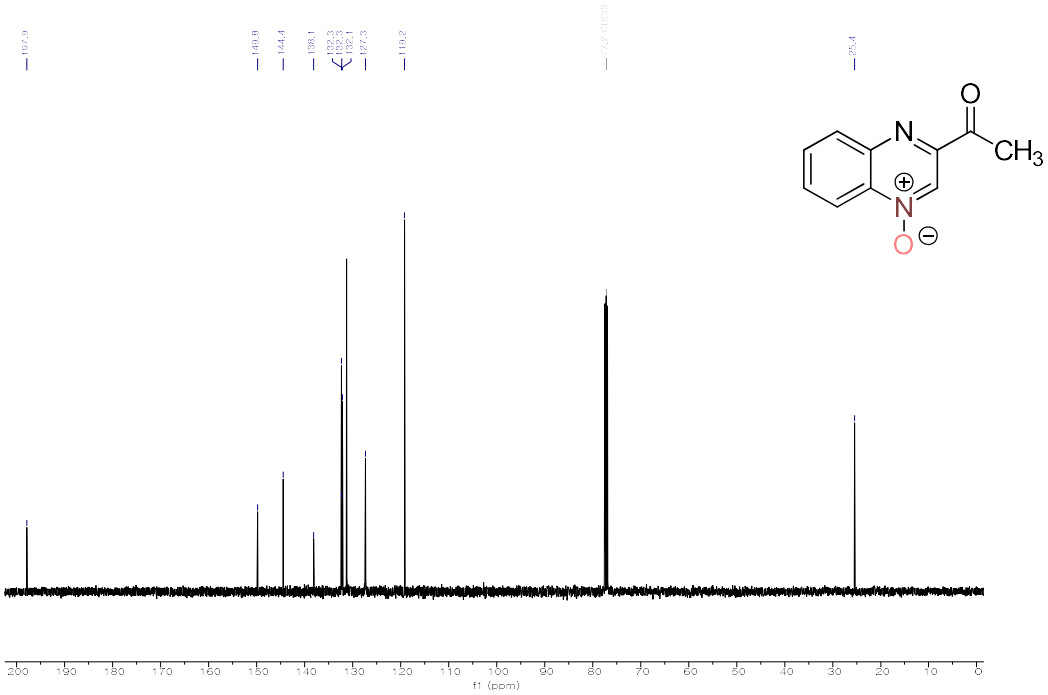


^{1}H & ^{13}C NMR Spectra of Methyl 2-Quinoxalinecarboxylate *N*-Oxide (1ag)

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

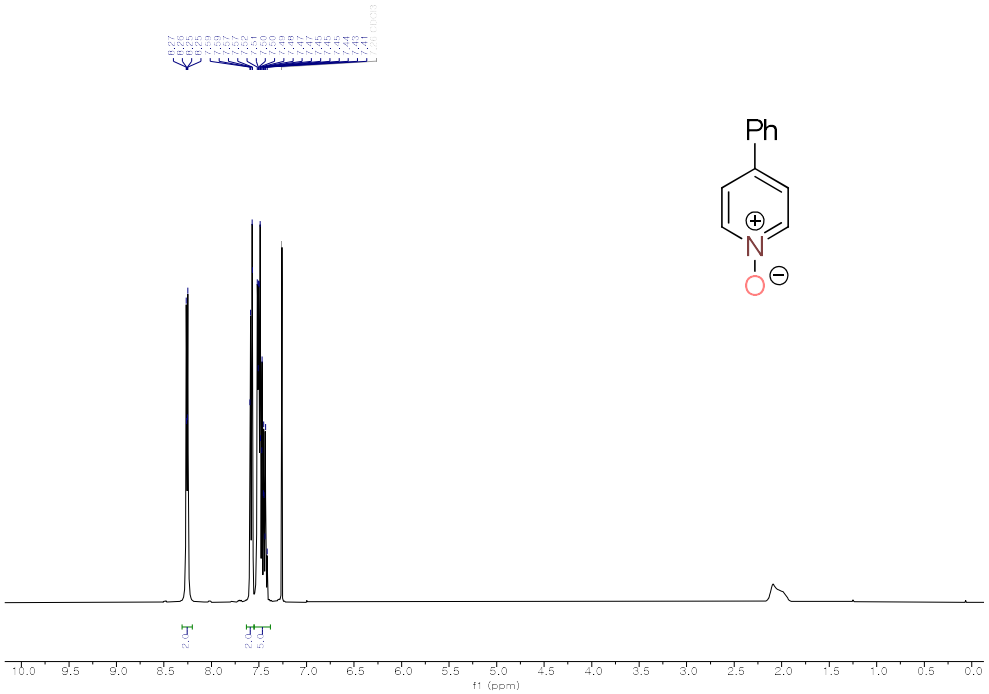


^{13}C NMR (126 MHz, $CDCl_3$)

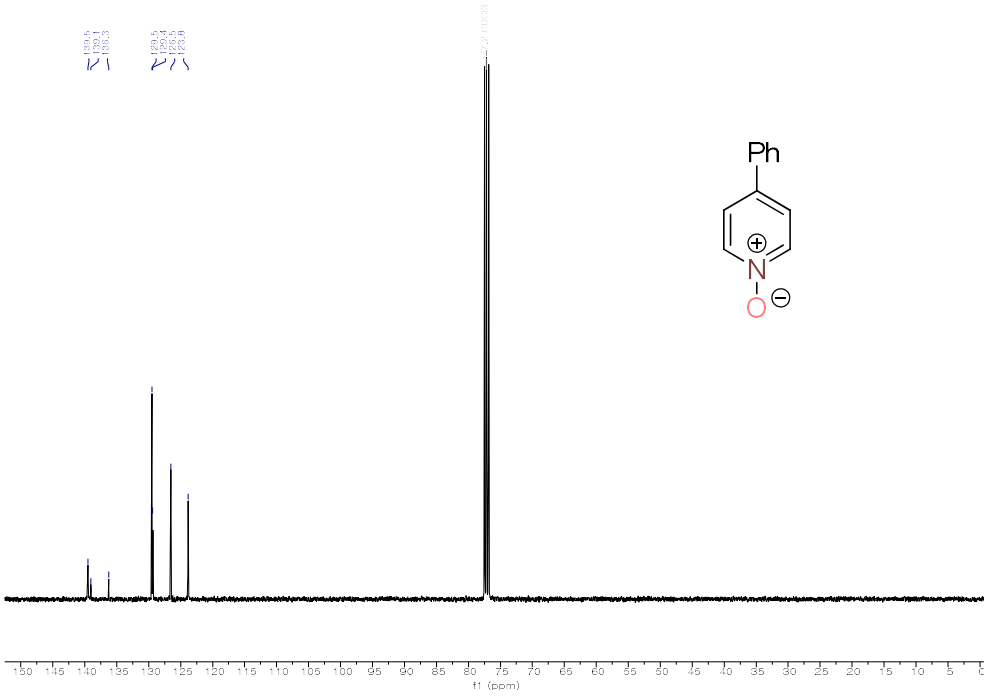


^{1}H & ^{13}C NMR Spectra of 4-Phenylpyridine *N*-Oxide (1ah)

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

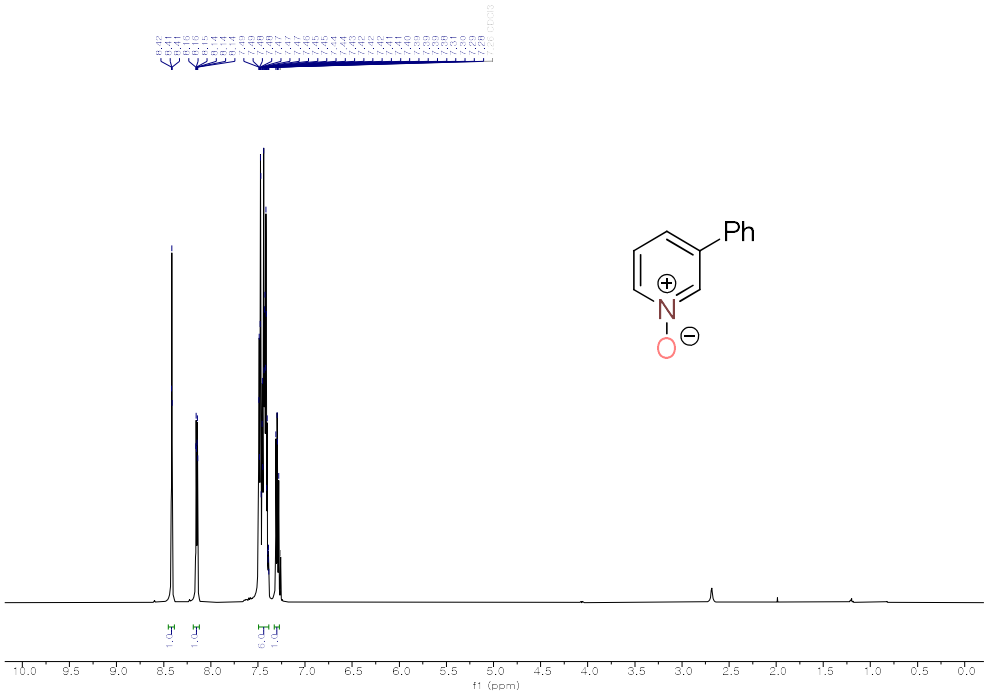


^{13}C NMR (101 MHz, $CDCl_3$)

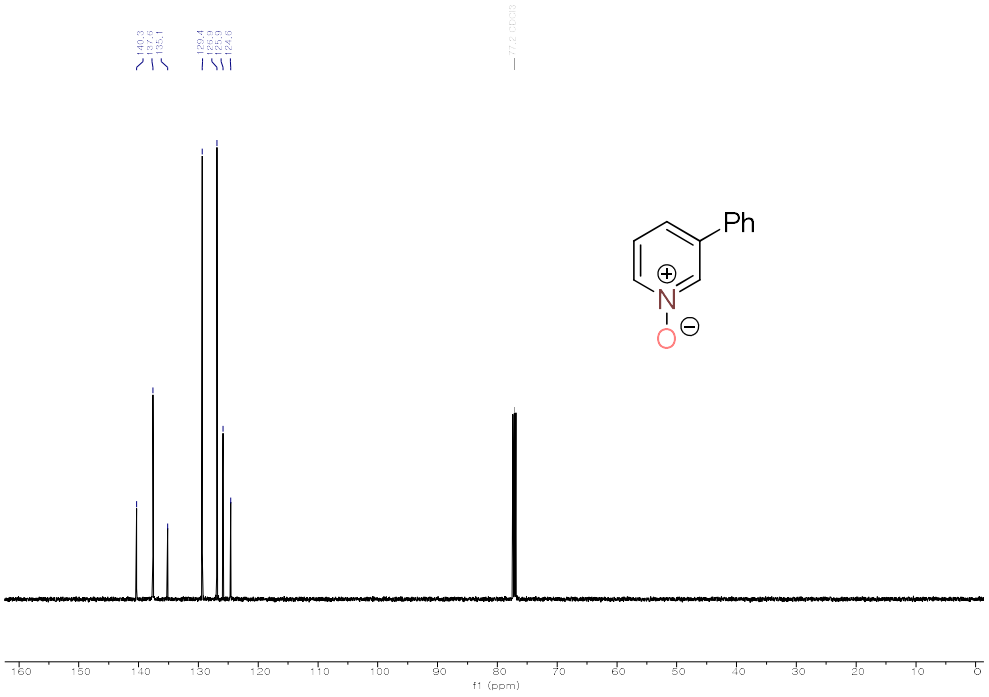


^{1}H & ^{13}C NMR Spectra of 3-Phenylpyridine *N*-Oxide (1ai)

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

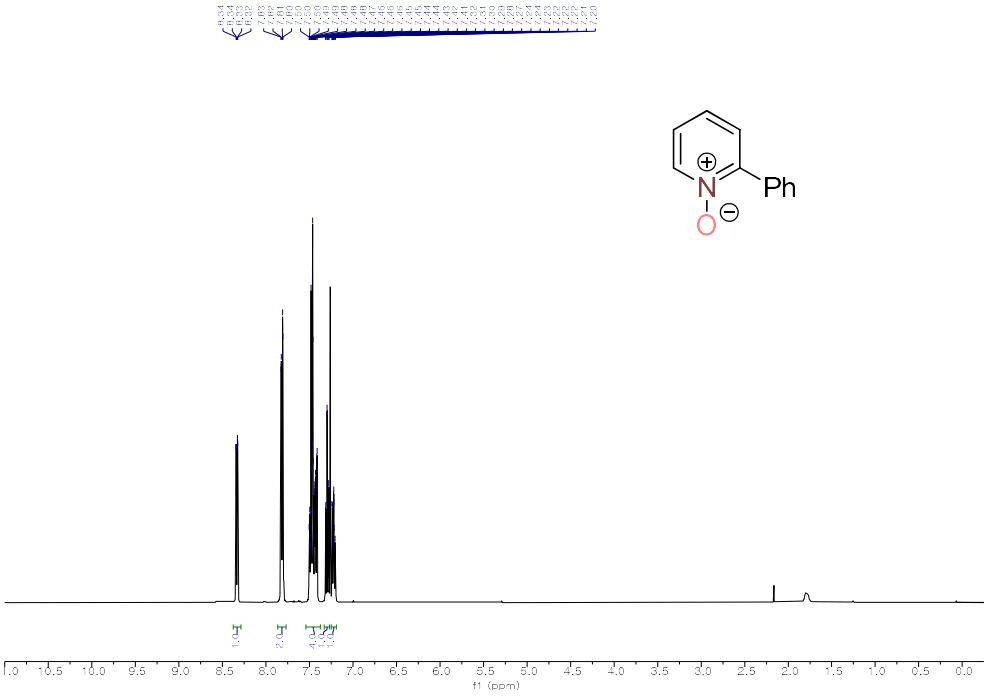


^{13}C NMR (126 MHz, $CDCl_3$)

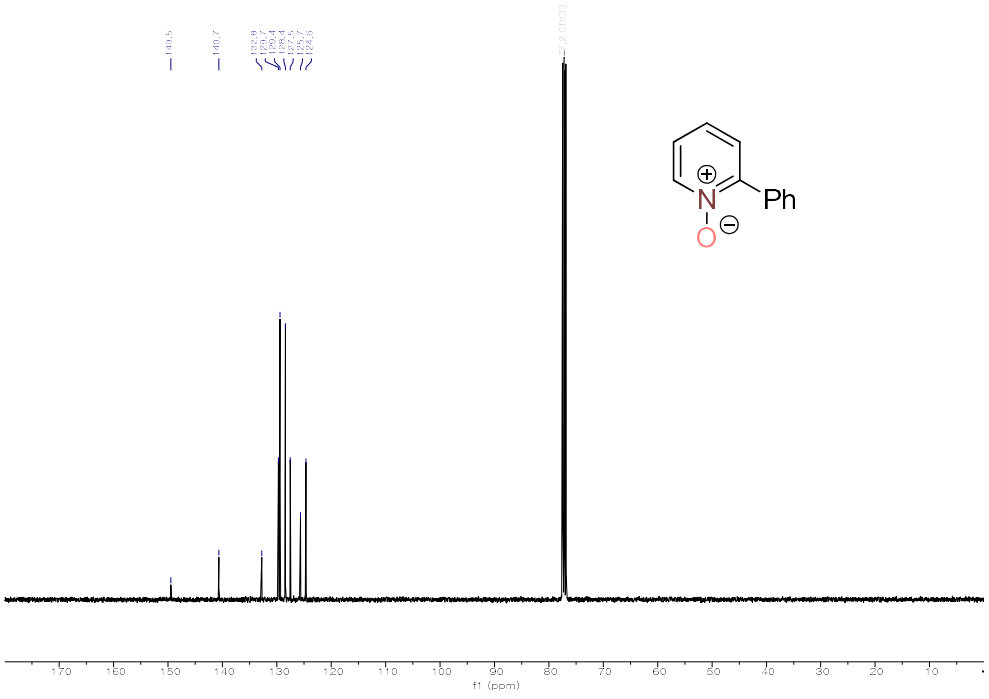


^{1}H & ^{13}C NMR Spectra of 2-Phenylpyridine *N*-Oxide (1aj)

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

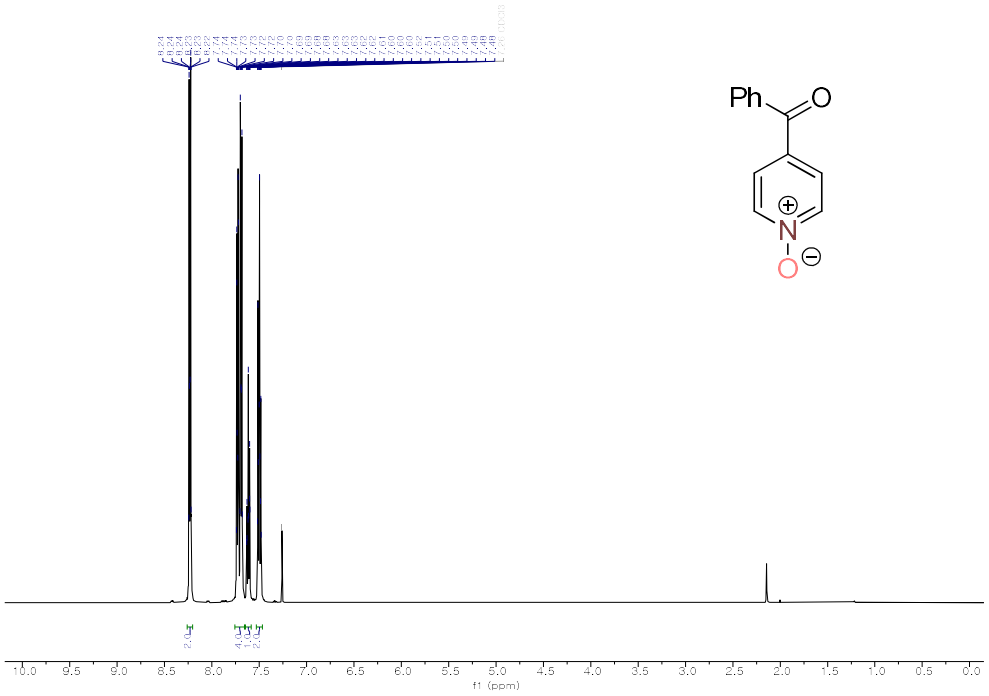


^{13}C NMR (101 MHz, $CDCl_3$)

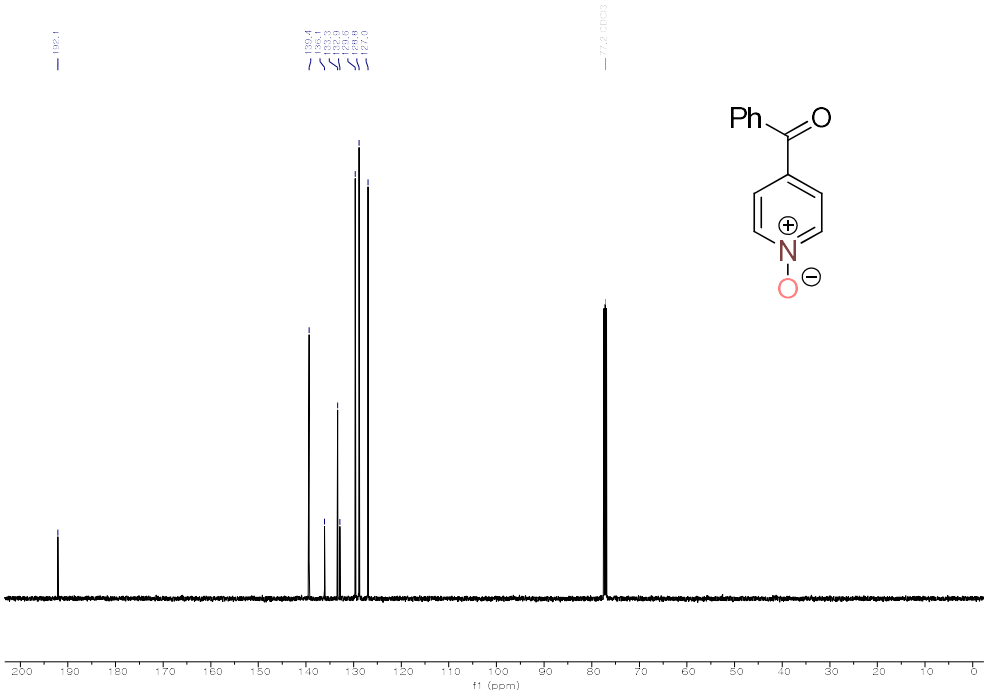


^{1}H & ^{13}C NMR Spectra of 4-Benzoylpyridine *N*-Oxide (1ak)

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

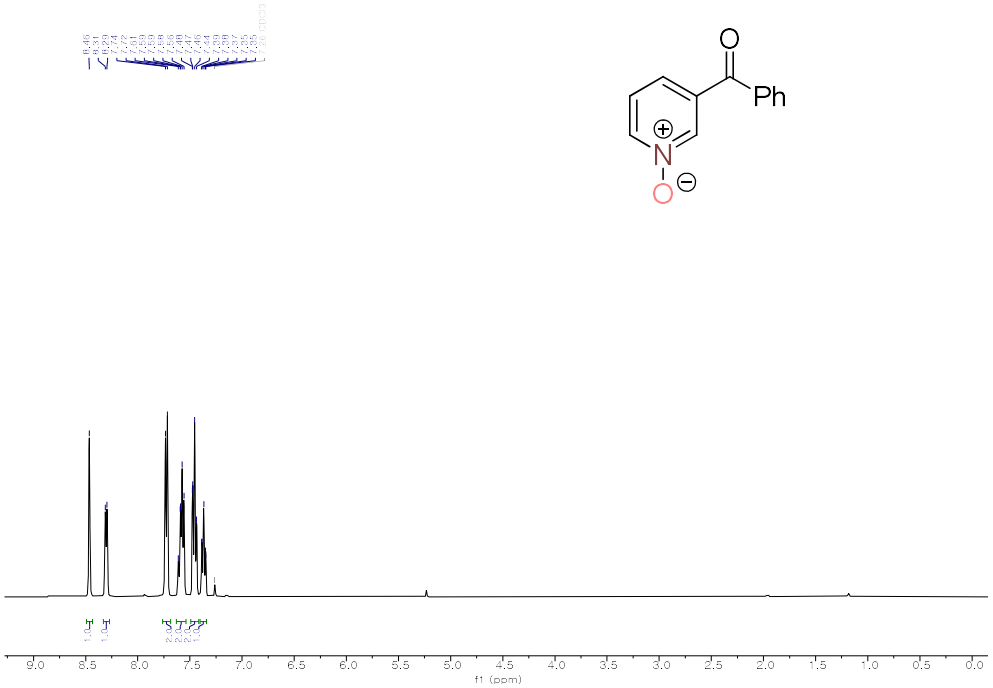


^{13}C NMR (126 MHz, $CDCl_3$)

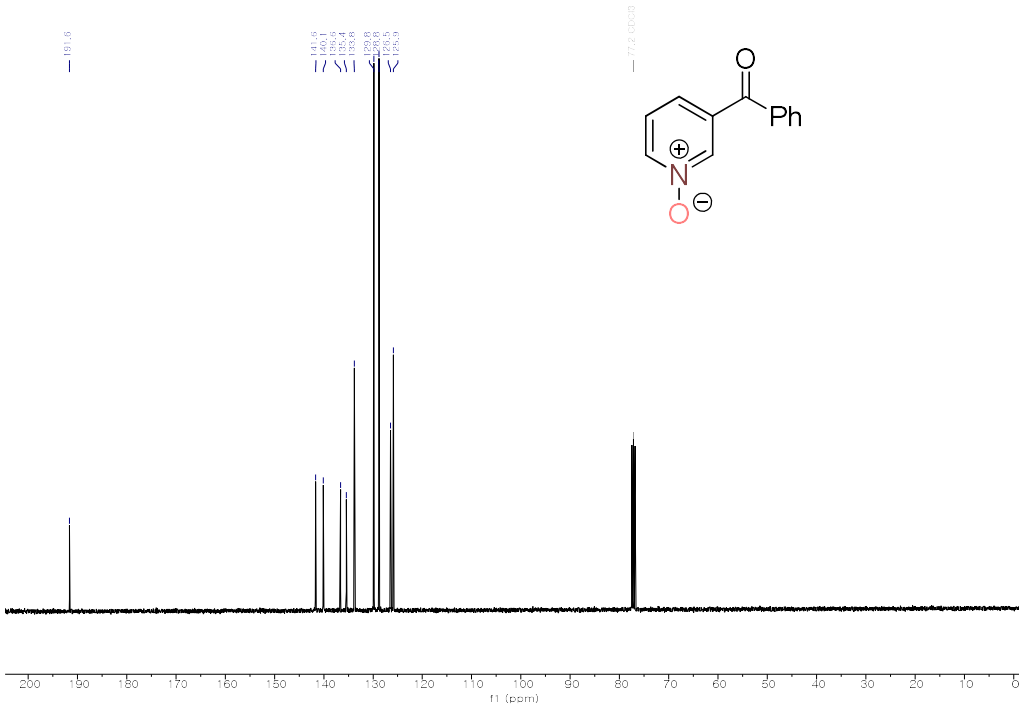


^{1}H & ^{13}C NMR Spectra of 3-Benzoylpyridine *N*-Oxide (1al)

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

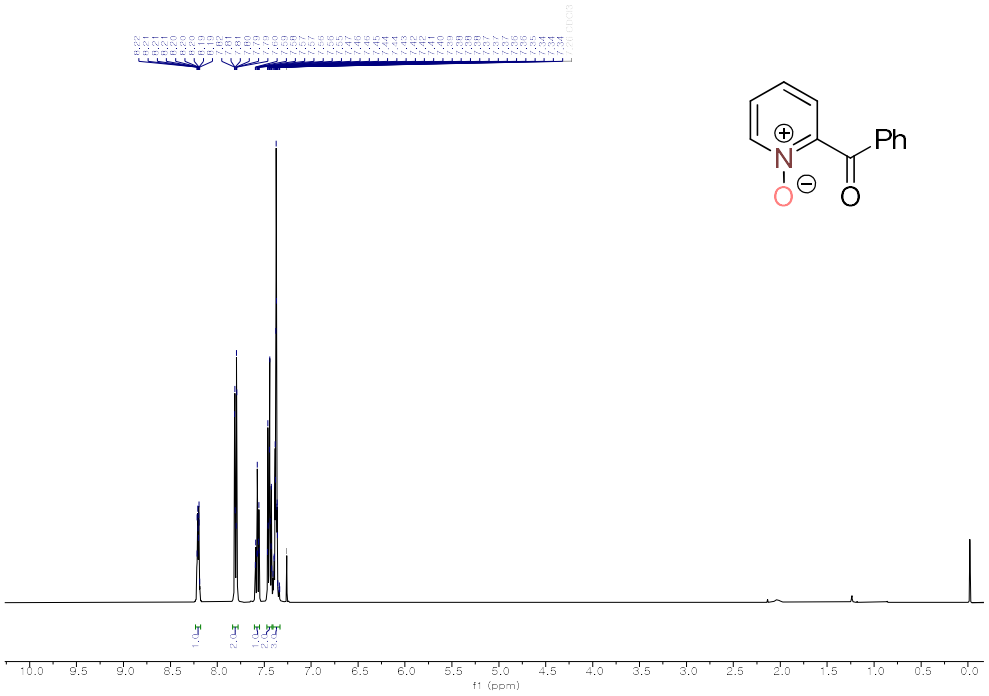


^{13}C NMR (101 MHz, $CDCl_3$)

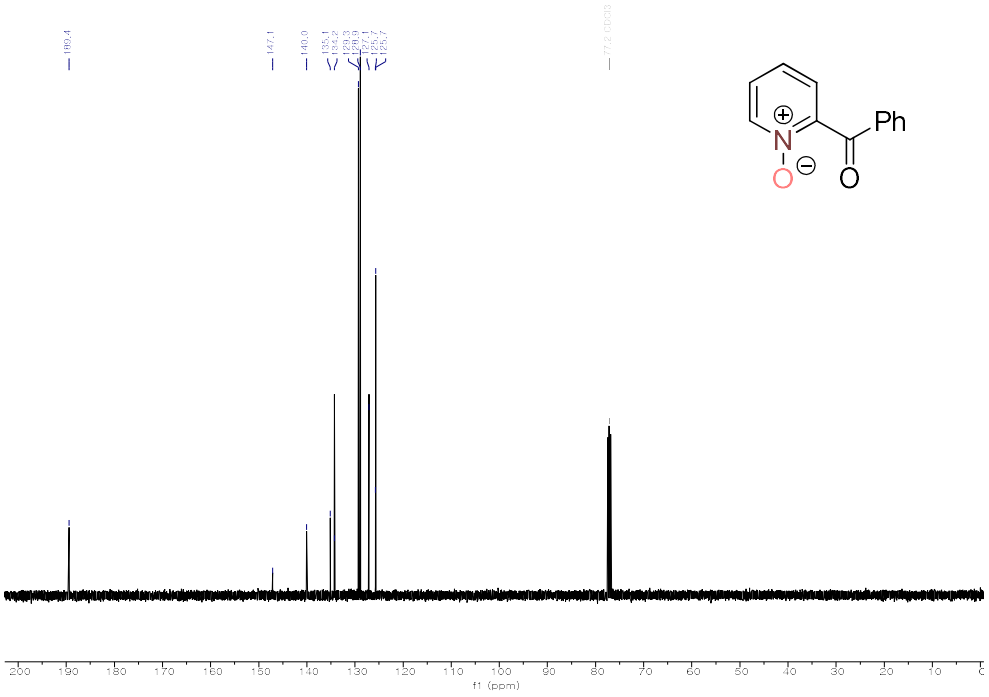


^{1}H & ^{13}C NMR Spectra of 2-Benzoylpyridine *N*-Oxide (1am)

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

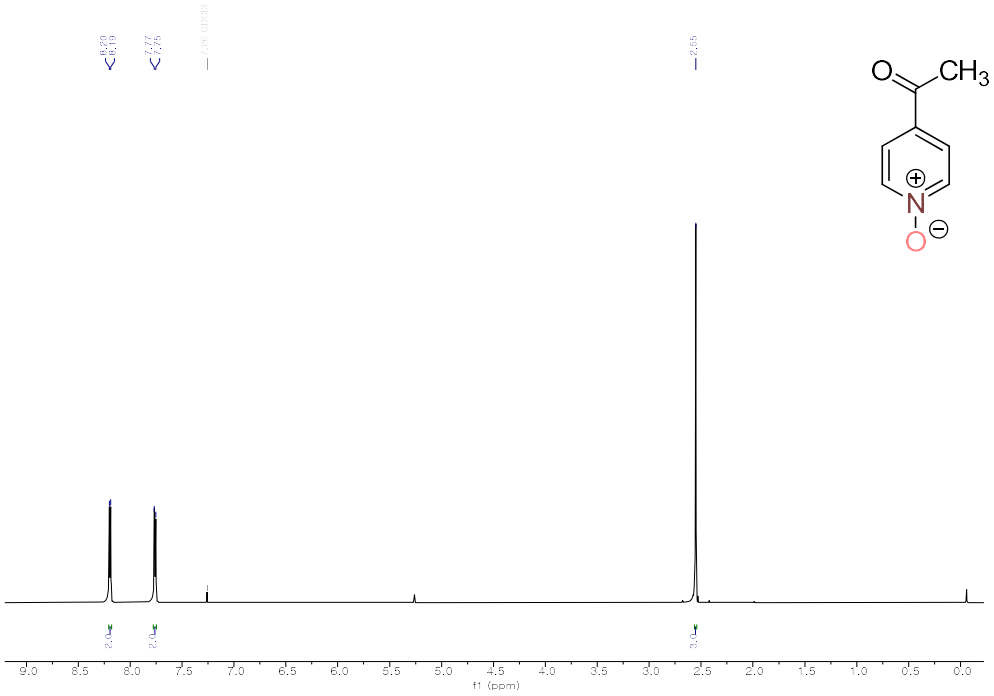


^{13}C NMR (101 MHz, $CDCl_3$)

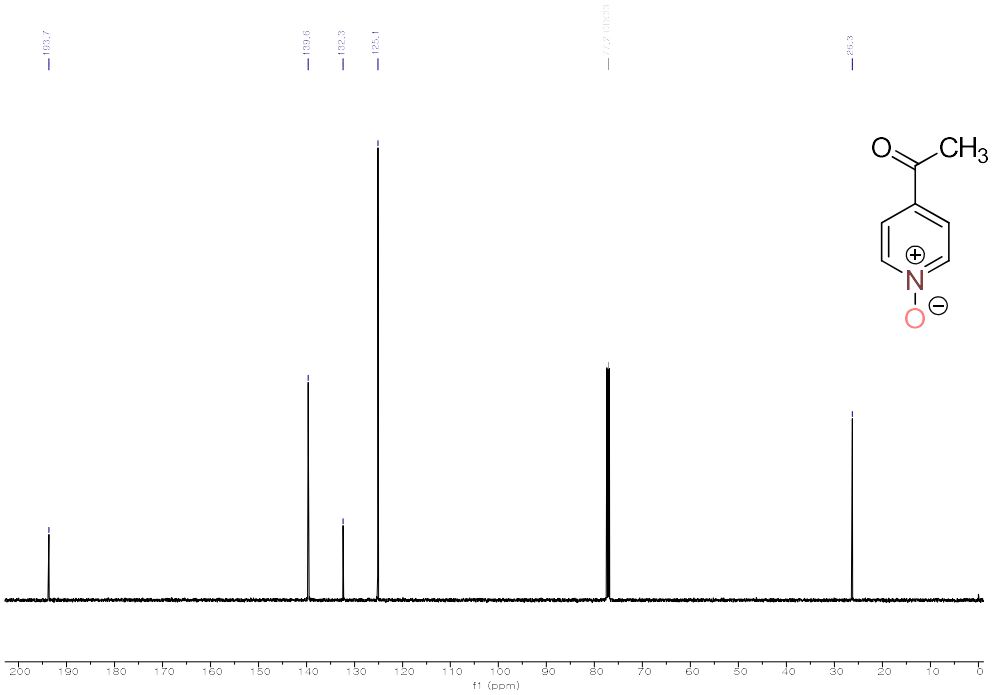


^{1}H & ^{13}C NMR Spectra of 4-Acetylpyridine *N*-Oxide (1an)

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

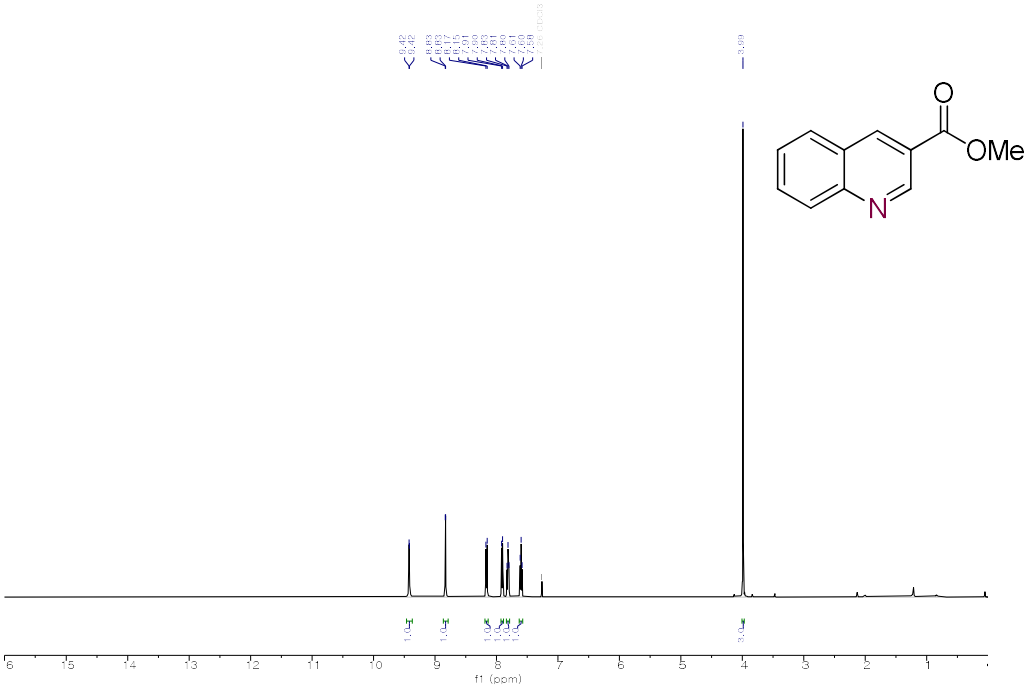


^{13}C NMR (126 MHz, $CDCl_3$)

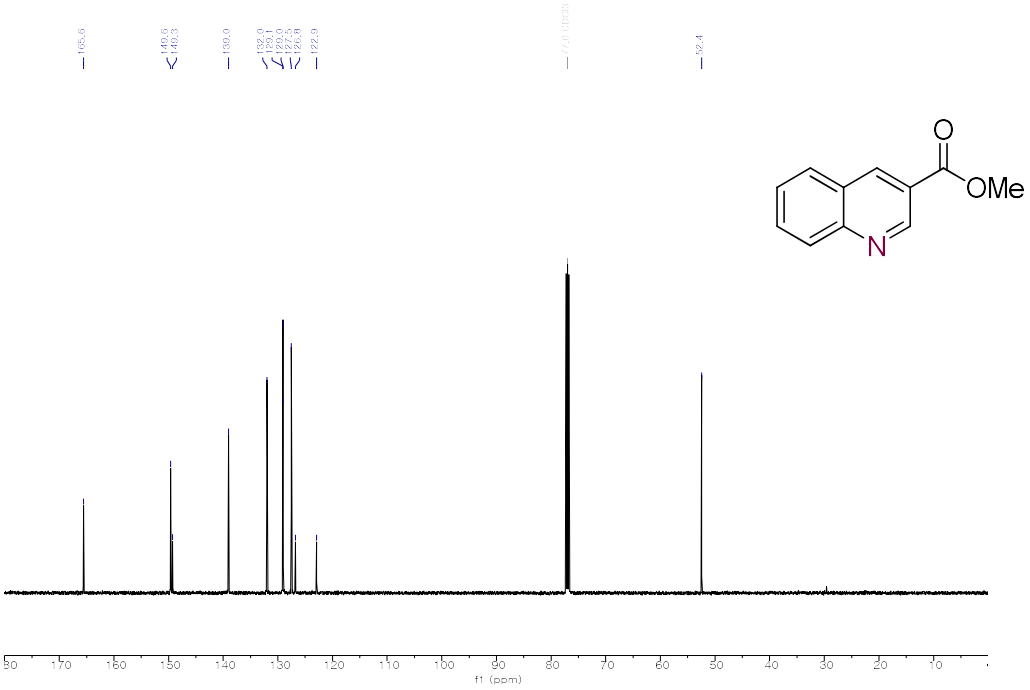


^{1}H & ^{13}C NMR Spectra of Methyl Quinoline-3-carboxylate (2a)

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

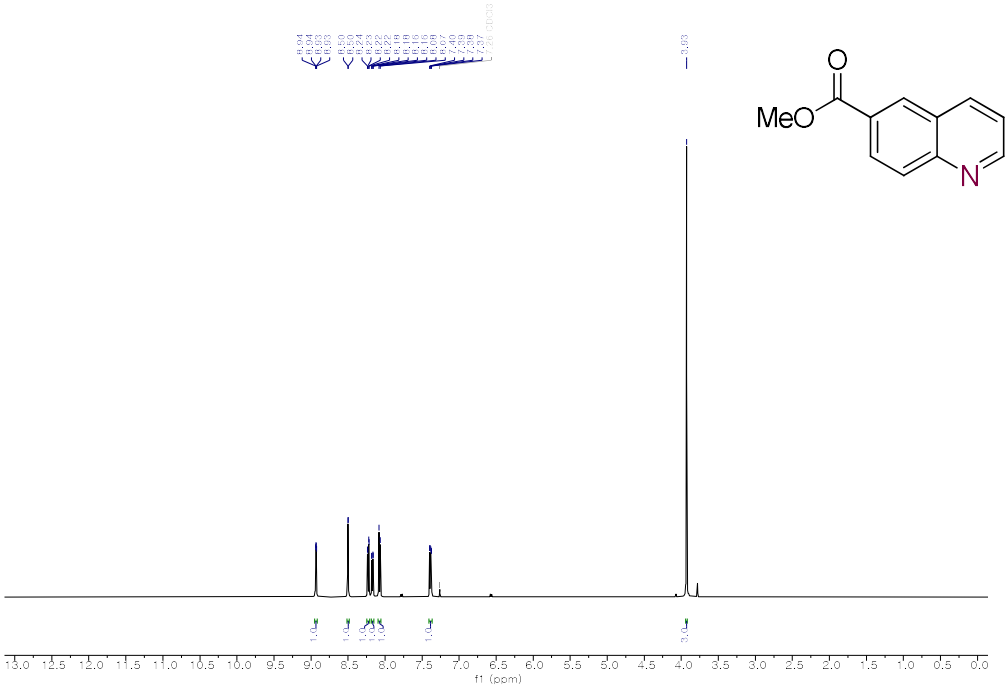


^{13}C NMR (126 MHz, $CDCl_3$

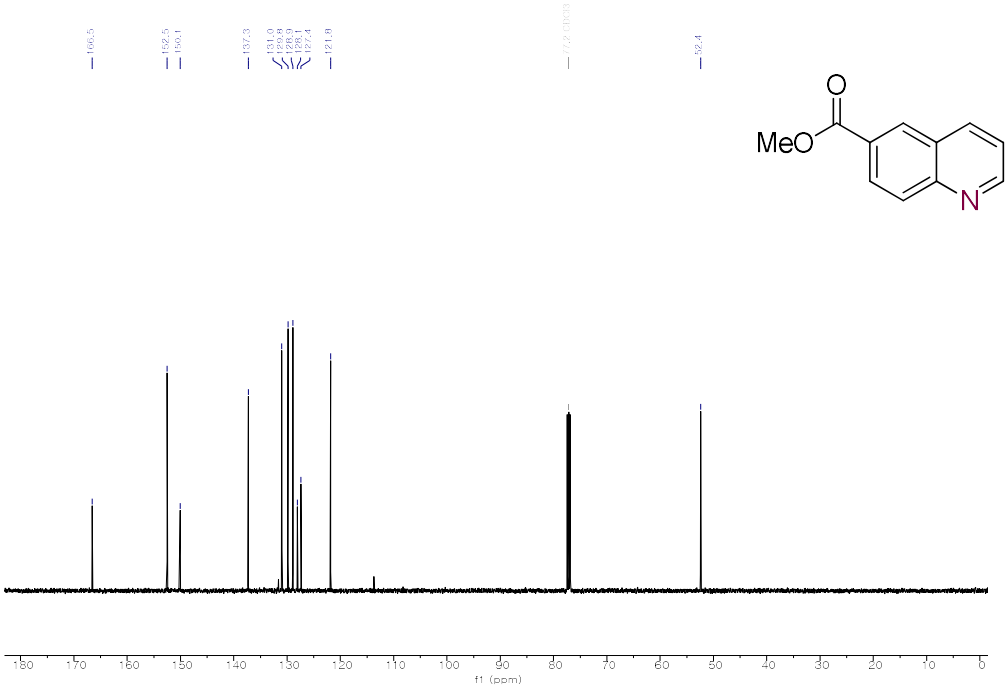


^{1}H & ^{13}C NMR Spectra of Methyl Quinoline-6-carboxylate (2b)

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

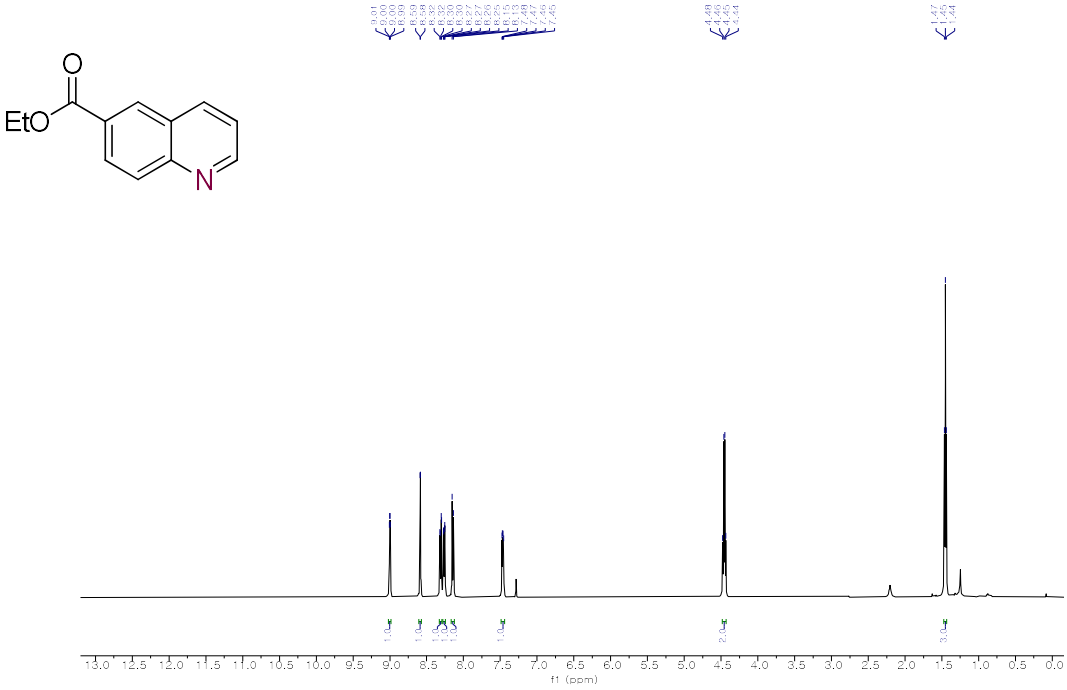


^{13}C NMR (126 MHz, $CDCl_3$) δ

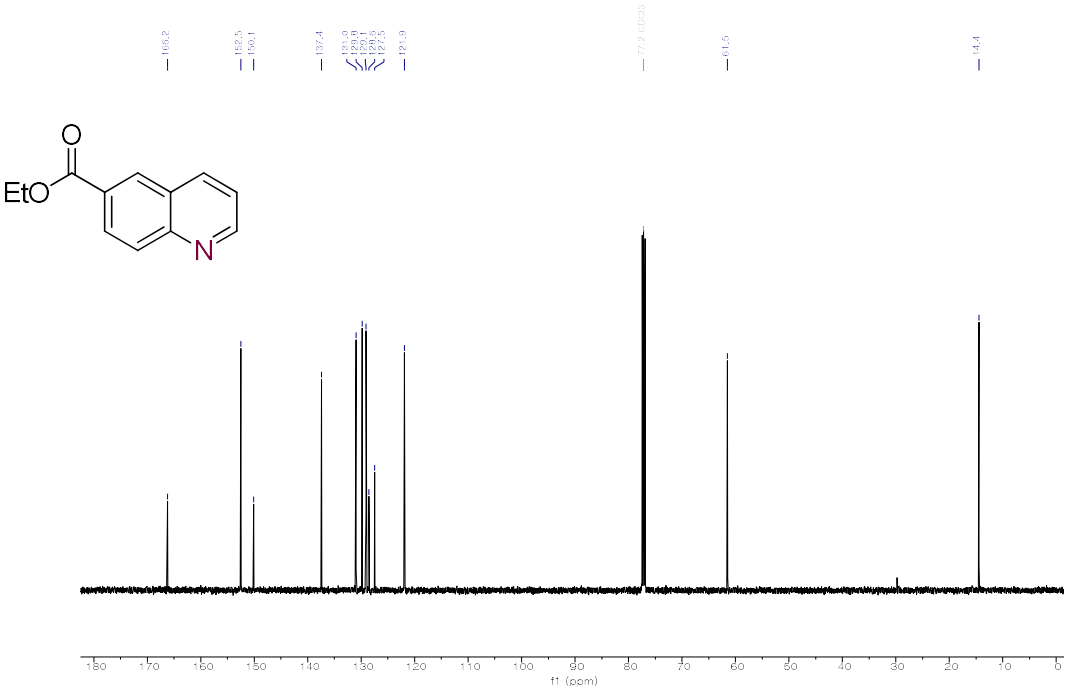


^{1}H & ^{13}C NMR Spectra of Ethyl Quinoline-6-carboxylate (2c)

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

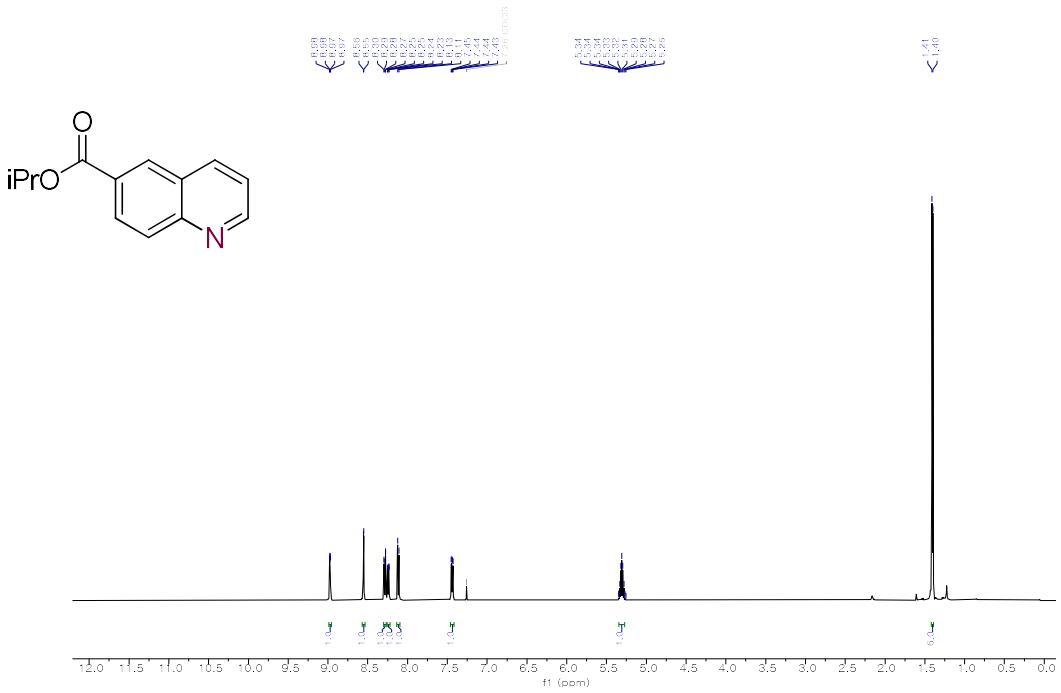


^{13}C NMR (126 MHz, $CDCl_3$)

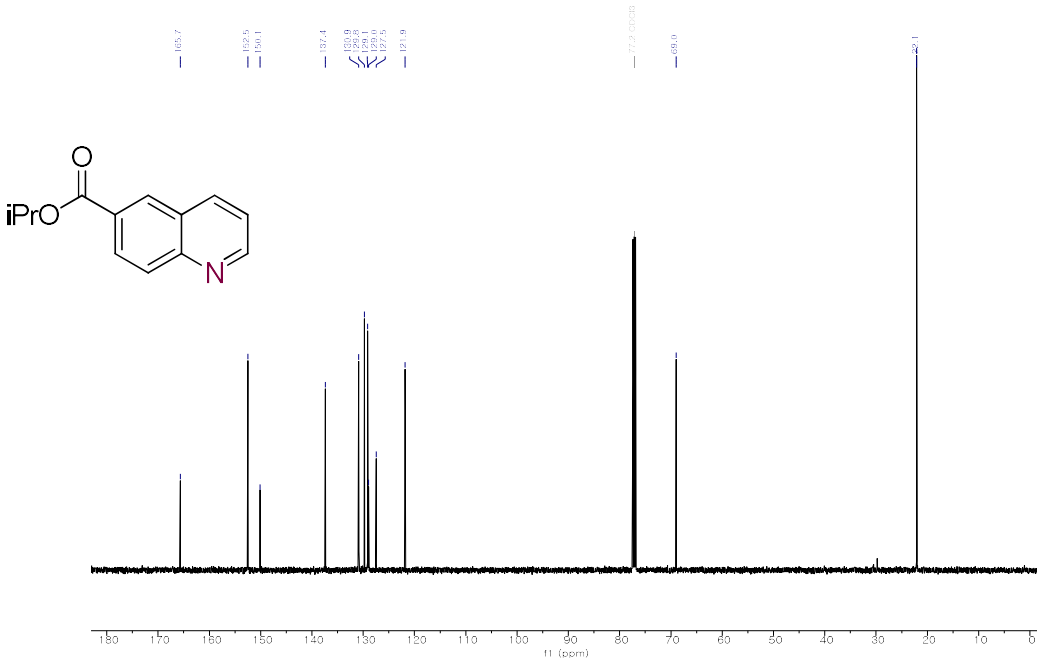


^{1}H & ^{13}C NMR Spectra of Isopropyl Quinoline-6-carboxylate (2d)

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

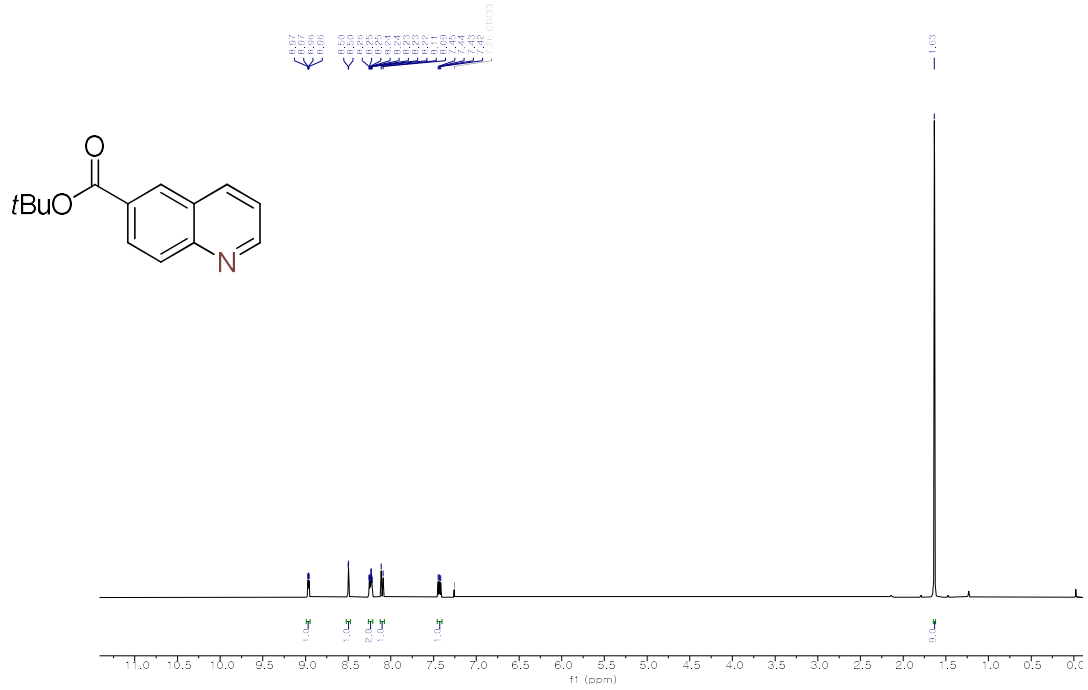


^{13}C NMR (126 MHz, $CDCl_3$)

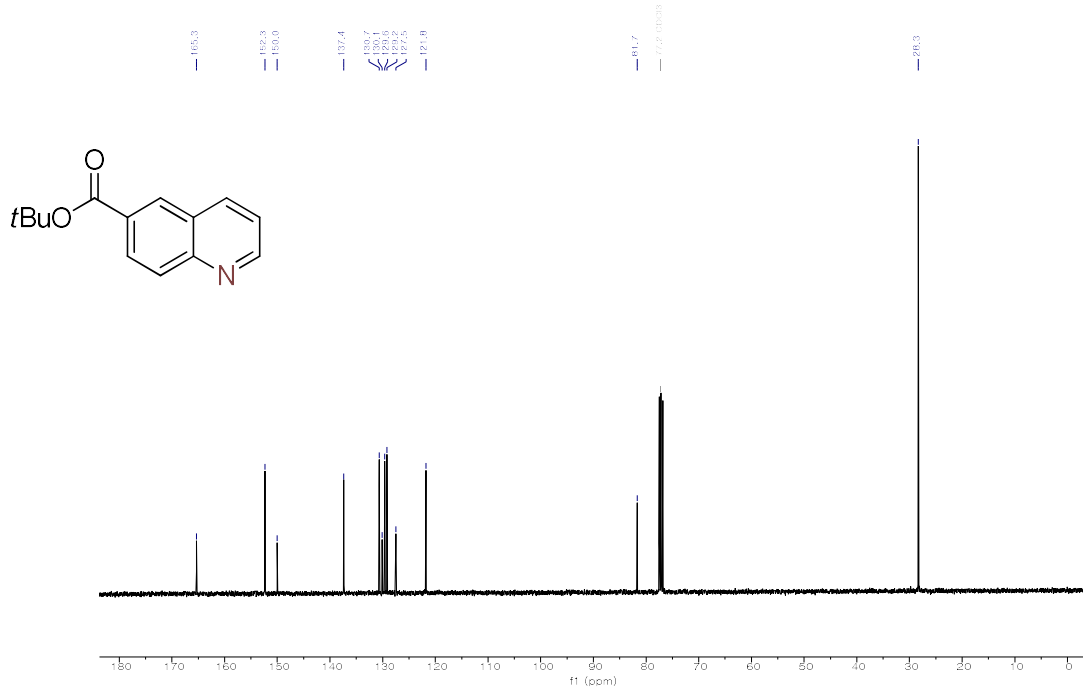


^{1}H & ^{13}C NMR Spectra of *tert*-Butyl Quinoline-6-carboxylate (2e)

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

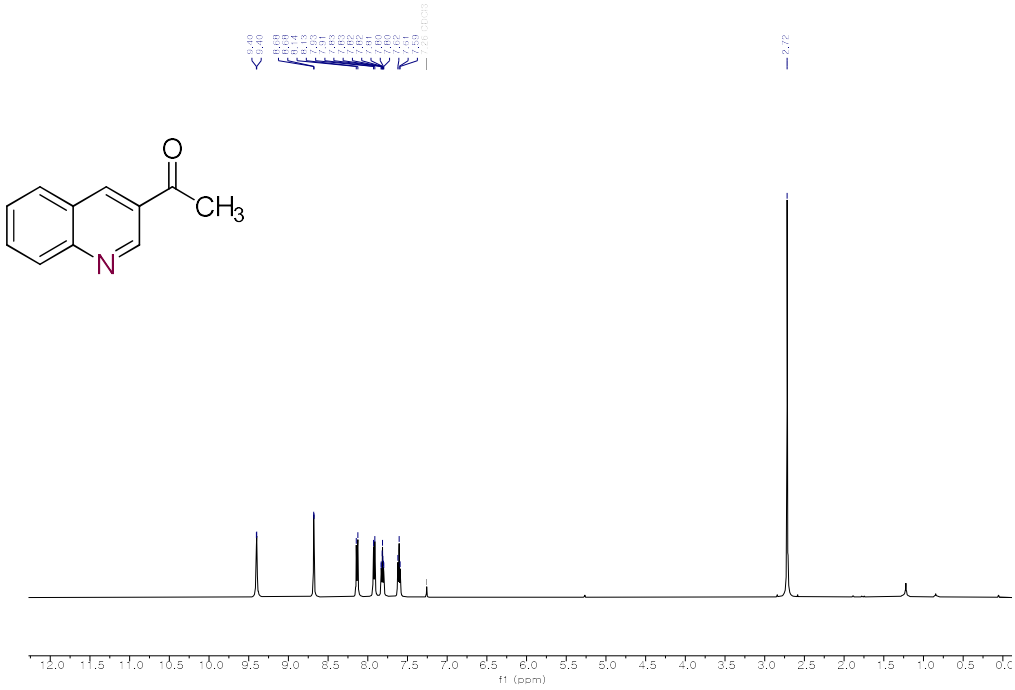


^{13}C NMR (101 MHz, $CDCl_3$)

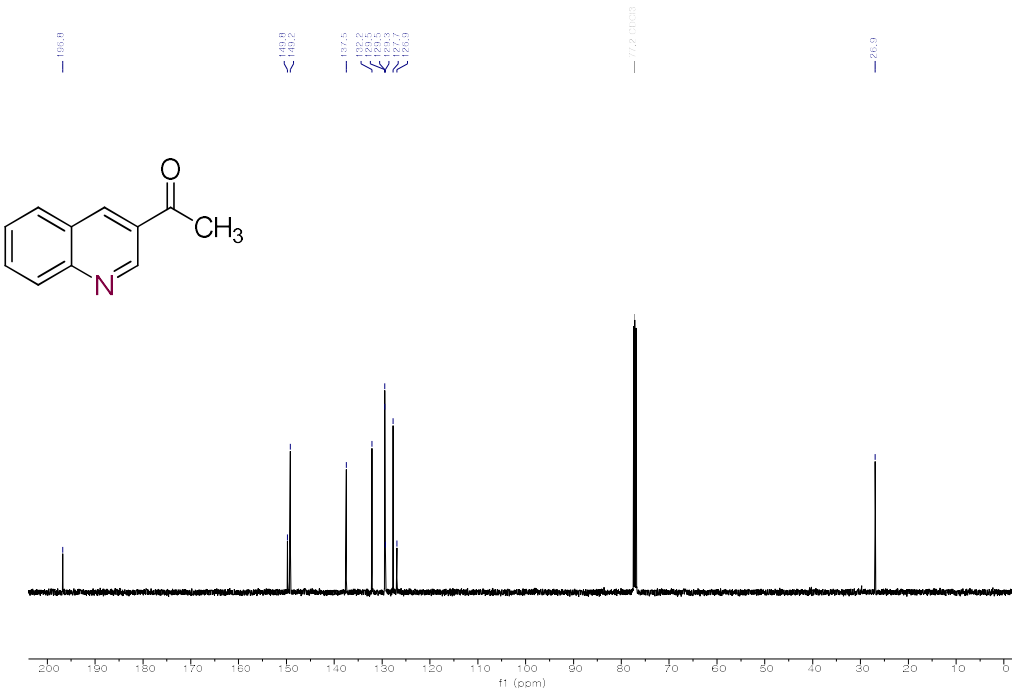


^{1}H & ^{13}C NMR Spectra of 3-Acetylquinoline (2f)

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

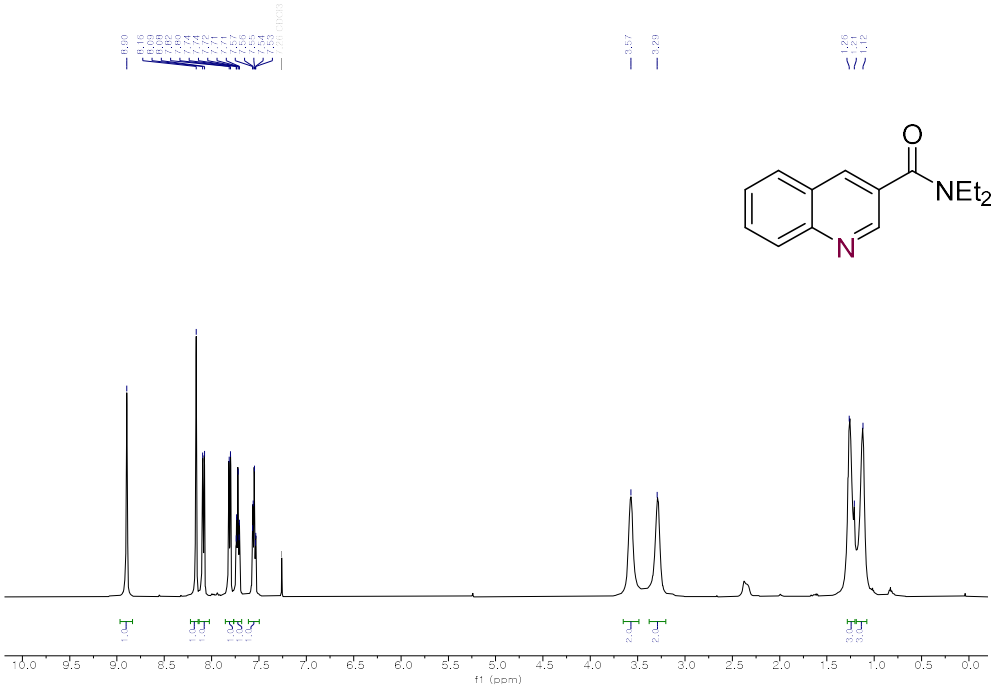


^{13}C NMR (126 MHz, $CDCl_3$)

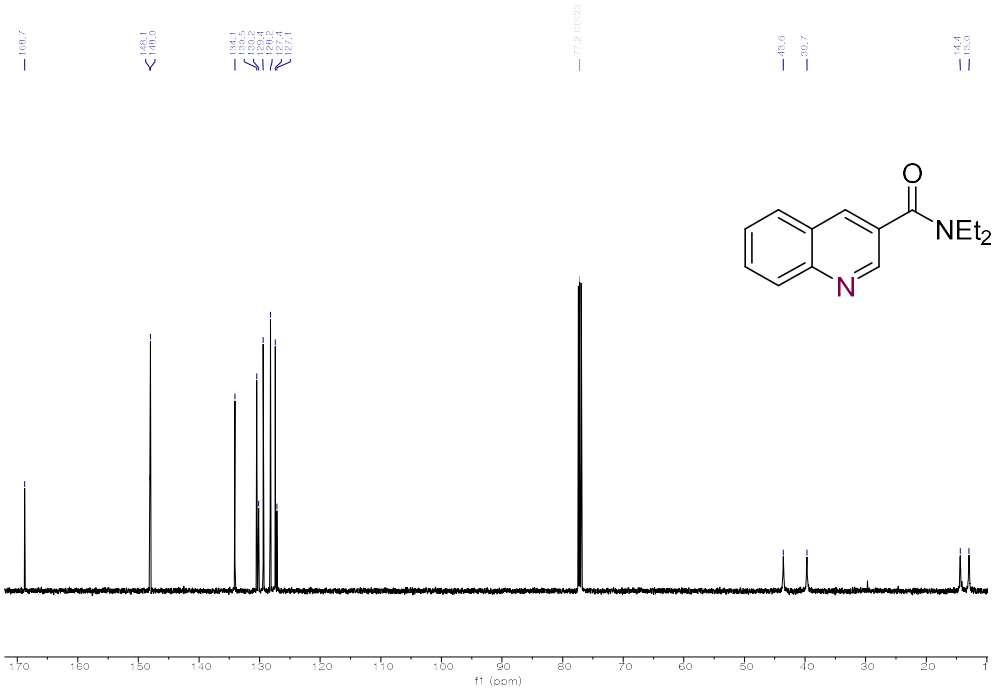


^{1}H & ^{13}C NMR Spectra of *N,N*-Diethyl-3-quinolinecarboxamide (2g)

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

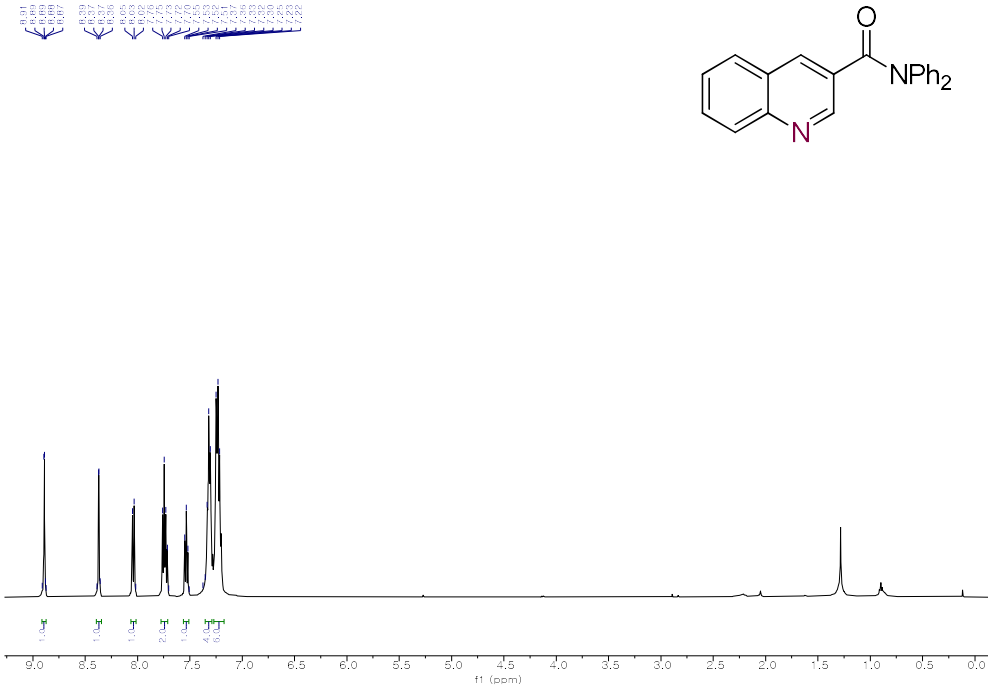


^{13}C NMR (126 MHz, $CDCl_3$)

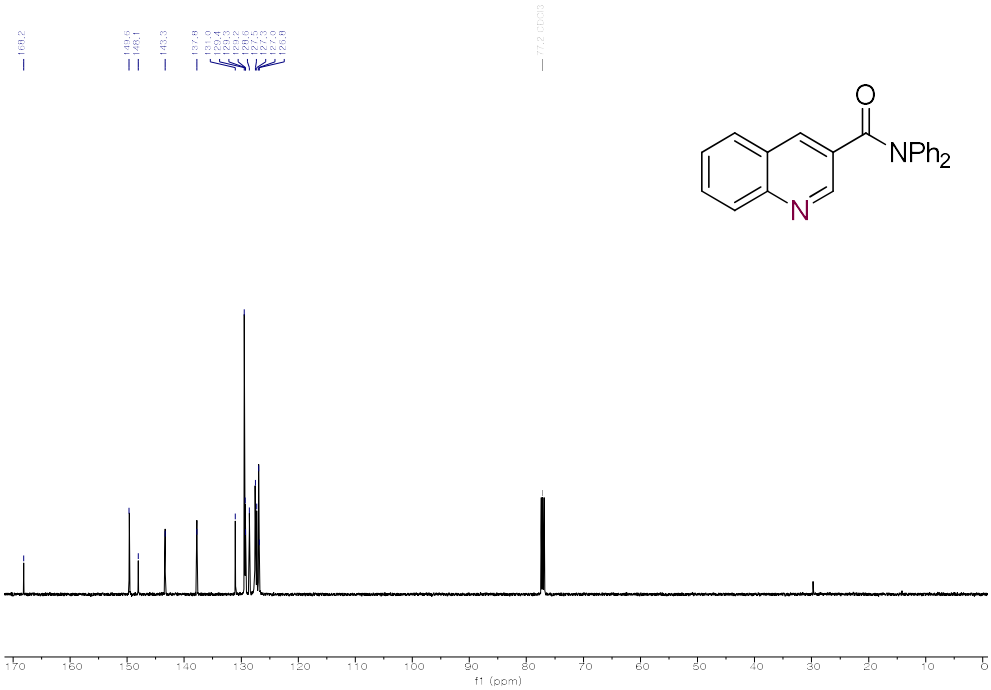


^{1}H & ^{13}C NMR Spectra of *N,N*-Diphenyl-3-quinolinecarboxamide (2h)

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

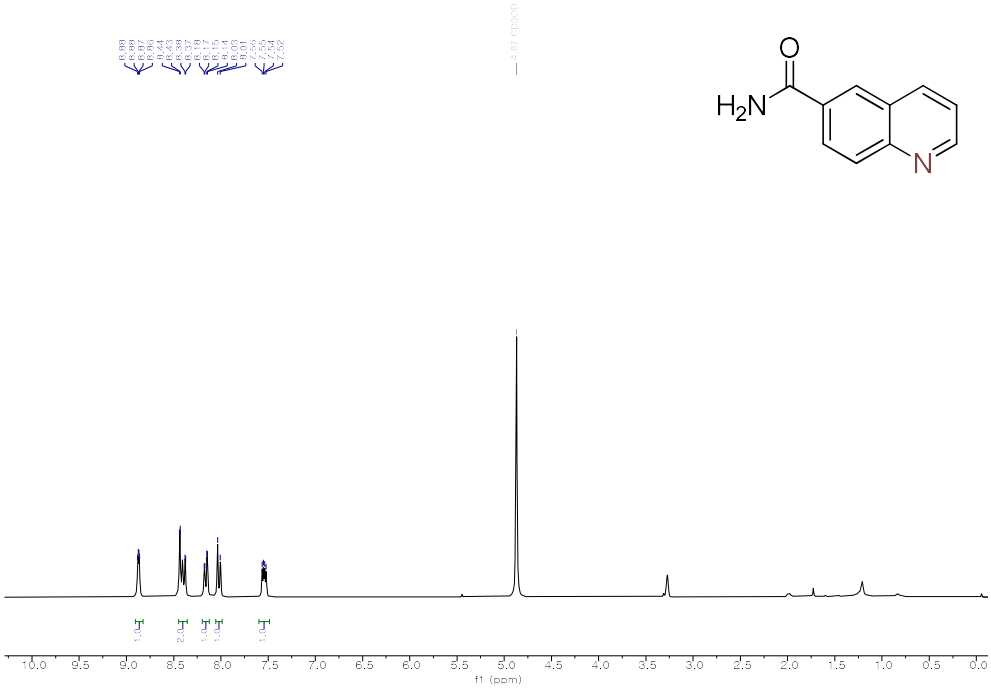


^{13}C NMR (126 MHz, $CDCl_3$)

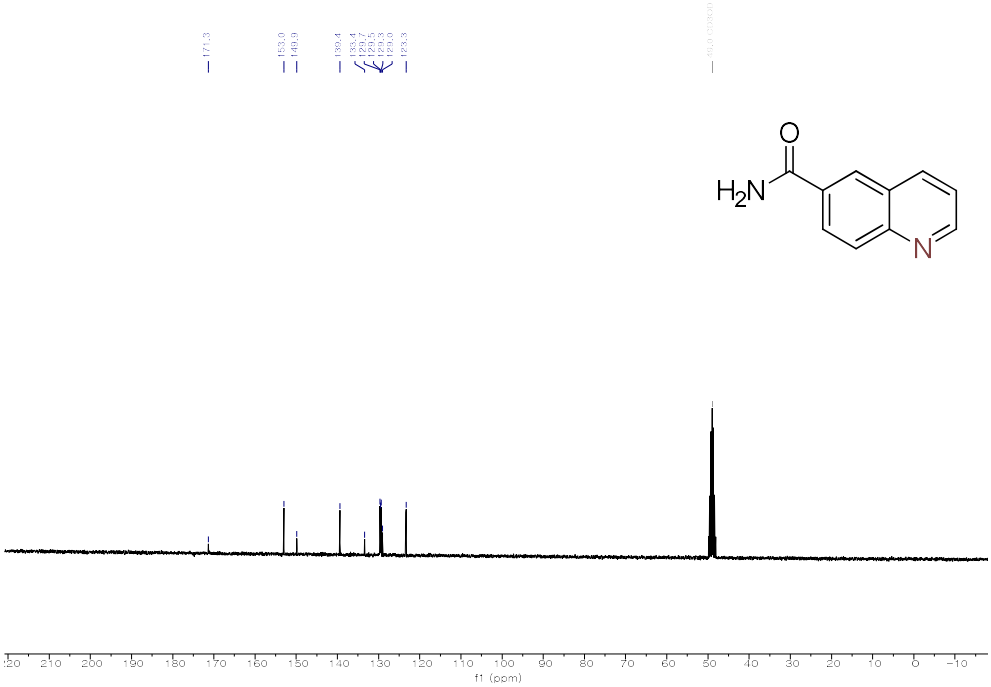


^{1}H & ^{13}C NMR Spectra of 6-Quinolinecarboxamide (2i)

^{1}H NMR (300 MHz, MeOD)

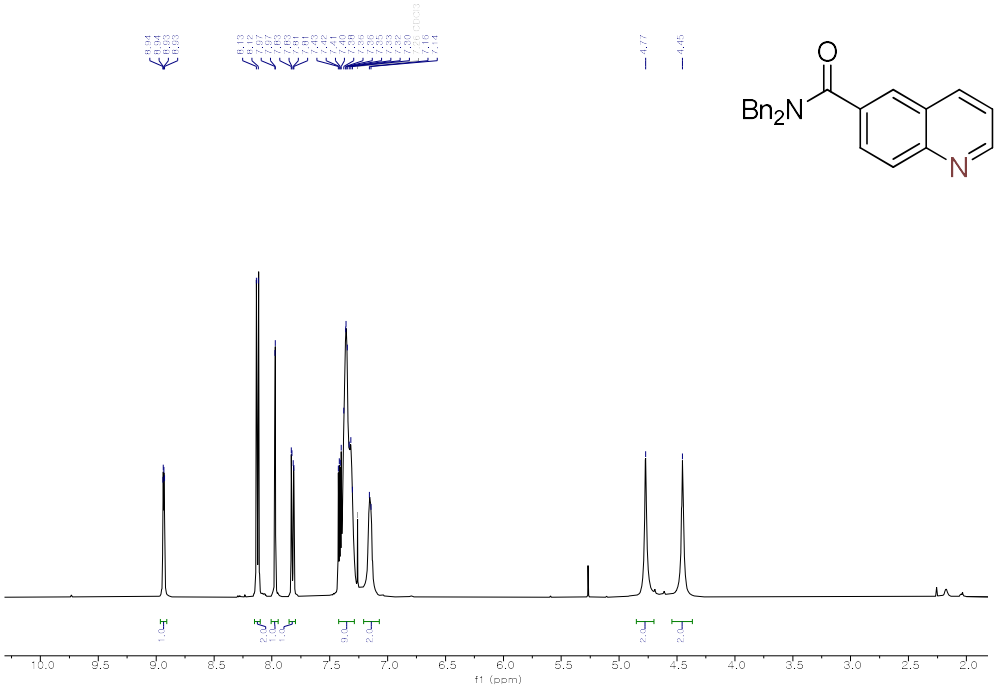


^{13}C NMR (75 MHz, MeOD)

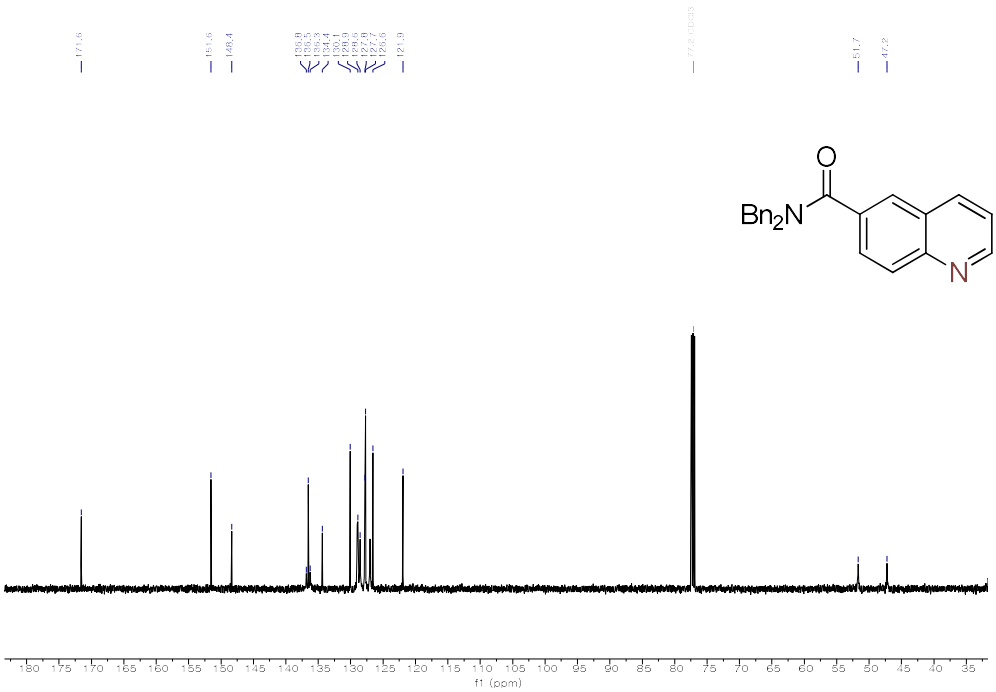


^{1}H & ^{13}C NMR Spectra of *N*,*N*-Dibenzyl-6-quinolinecarboxamide (2j)

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

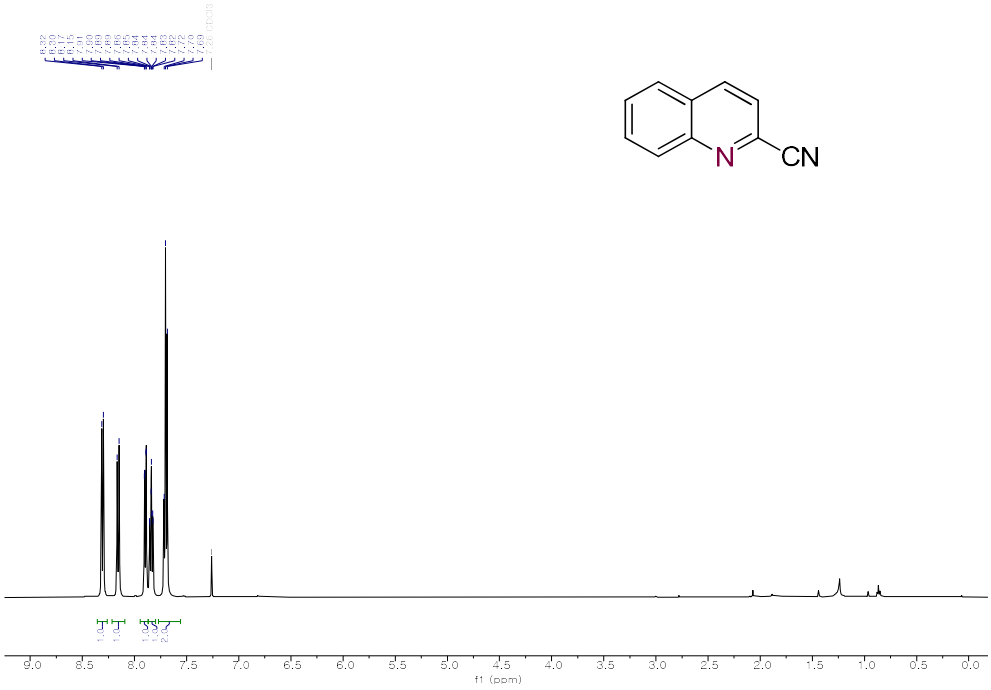


^{13}C NMR (126 MHz, $CDCl_3$)

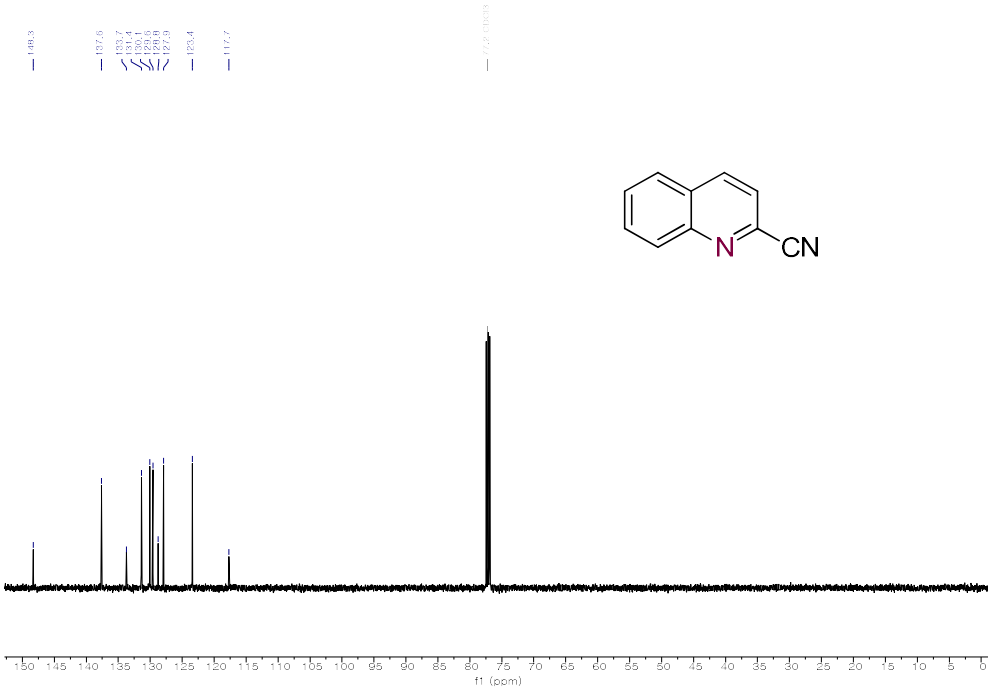


^{1}H & ^{13}C NMR Spectra of 2-Quinolinecarbonitrile (2k)

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

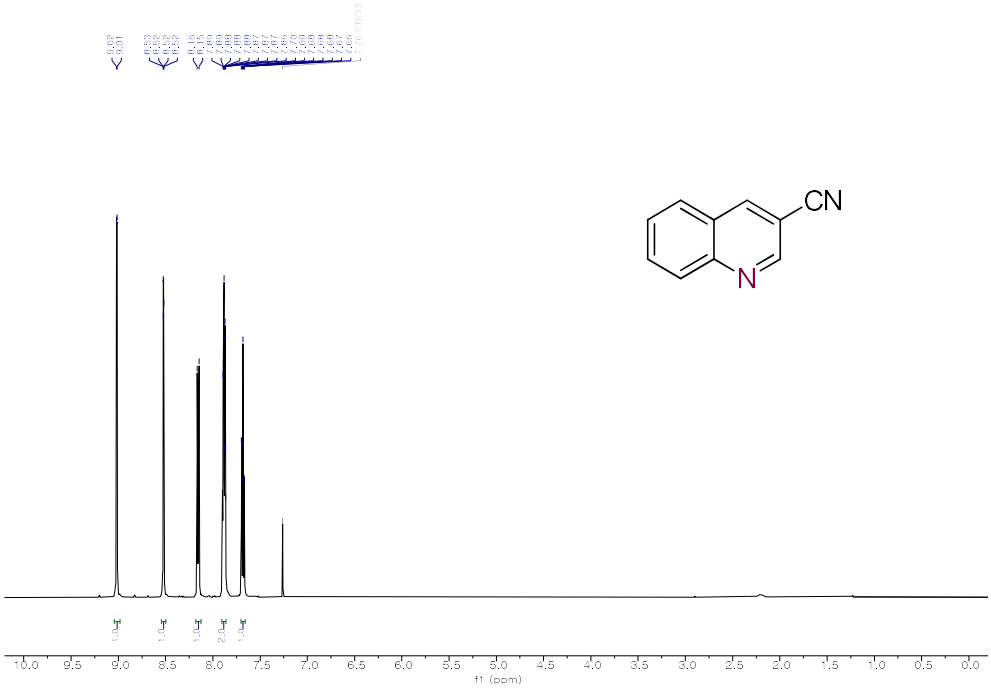


^{13}C NMR (126 MHz, $CDCl_3$)

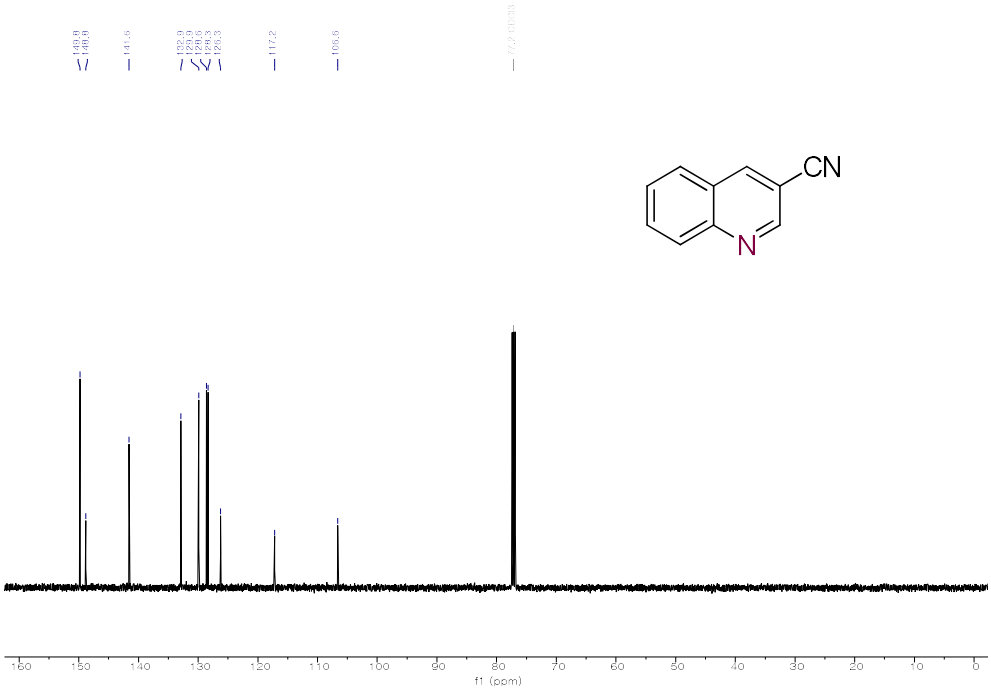


^{1}H & ^{13}C NMR Spectra of 3-Quinolinecarbonitrile (2l)

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

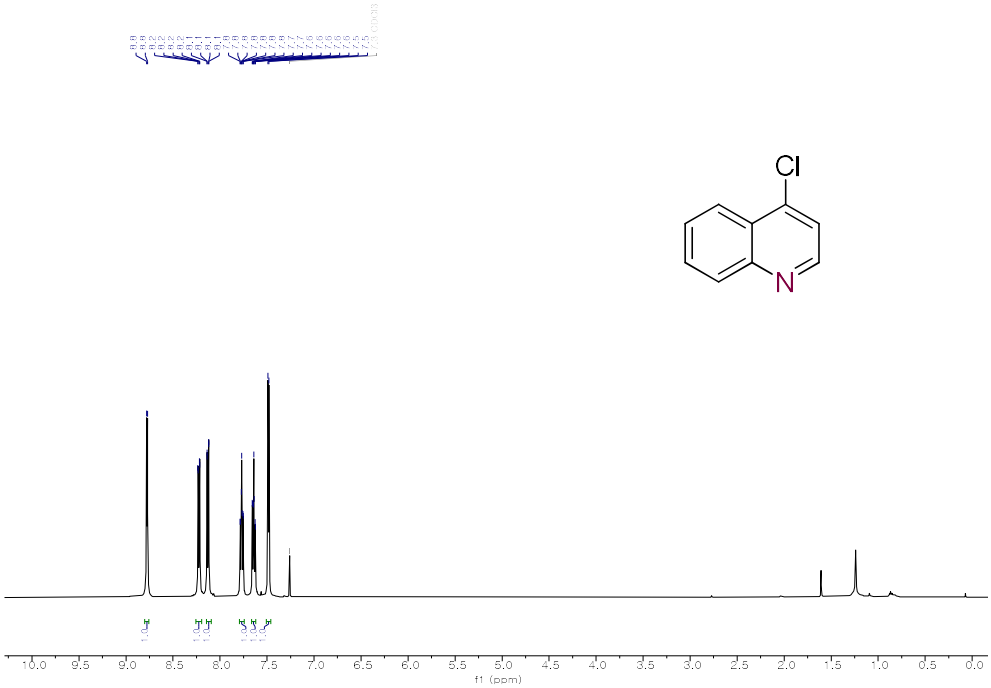


^{13}C NMR (126 MHz, $CDCl_3$)

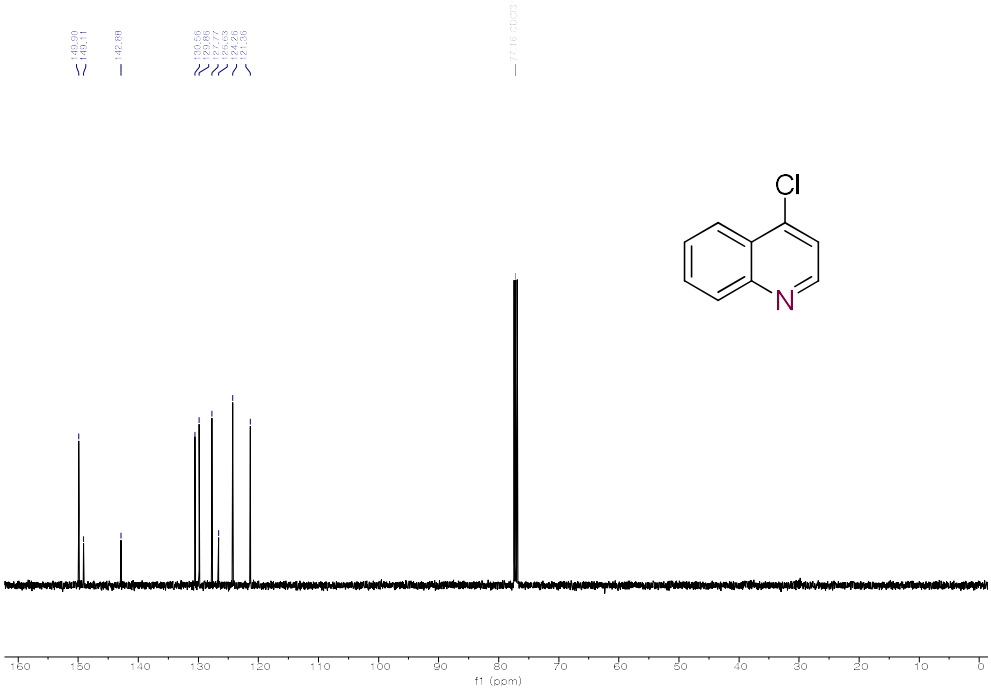


^{1}H & ^{13}C NMR Spectra of 4-Chloroquinoline (2m)

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

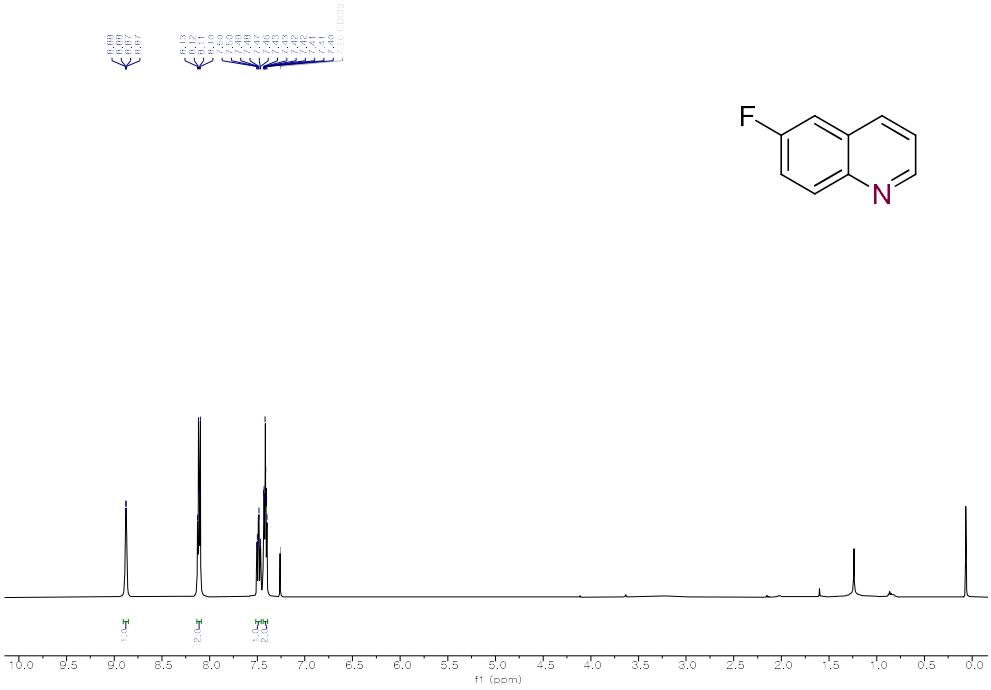


^{13}C NMR (126 MHz, $CDCl_3$)

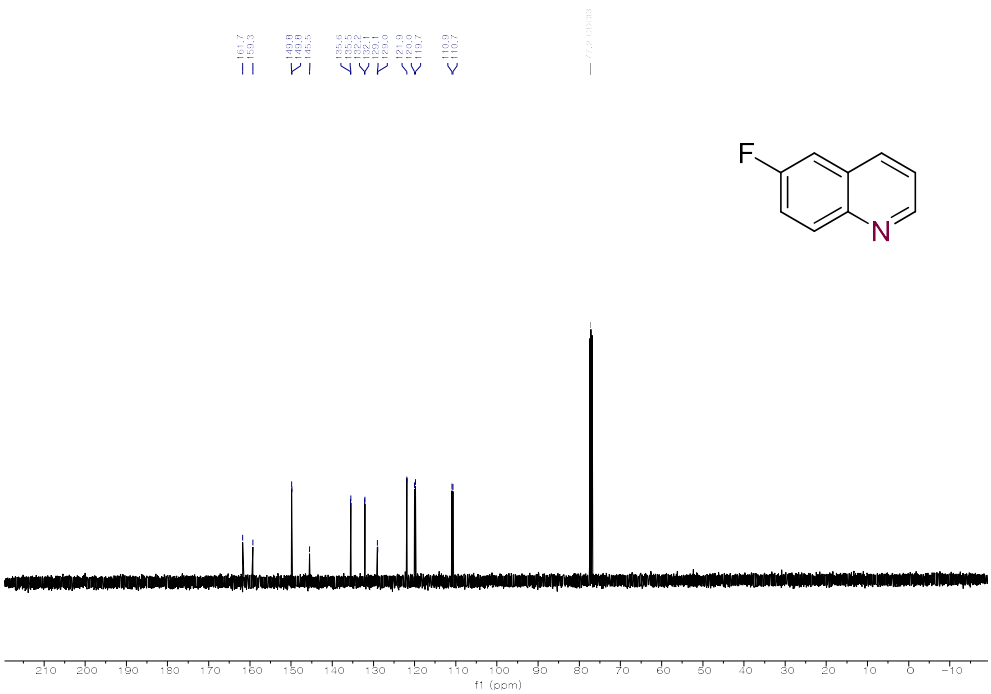


^{1}H & ^{13}C & ^{19}F NMR Spectra of 6-Fluoroquinoline (2n)

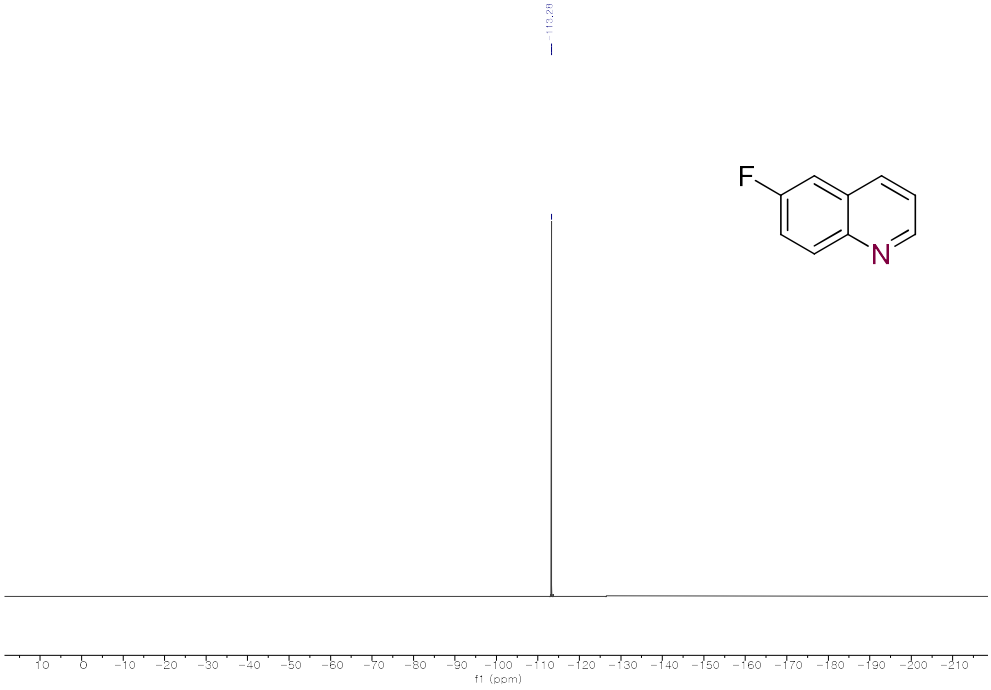
^{1}H NMR (500 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)

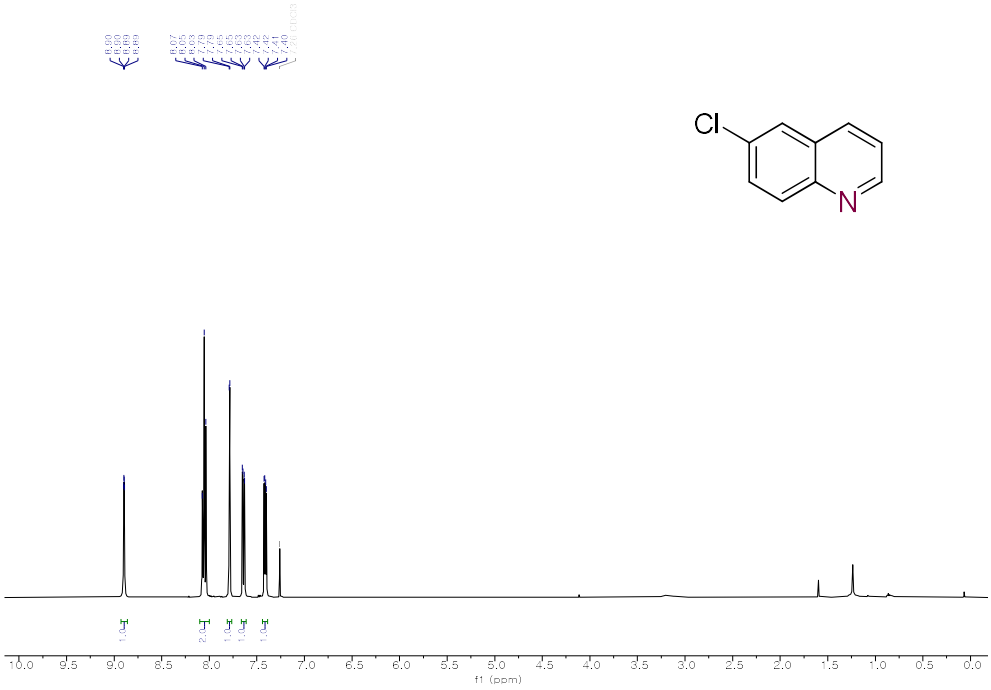


^{19}F NMR (376 MHz, $CDCl_3$)

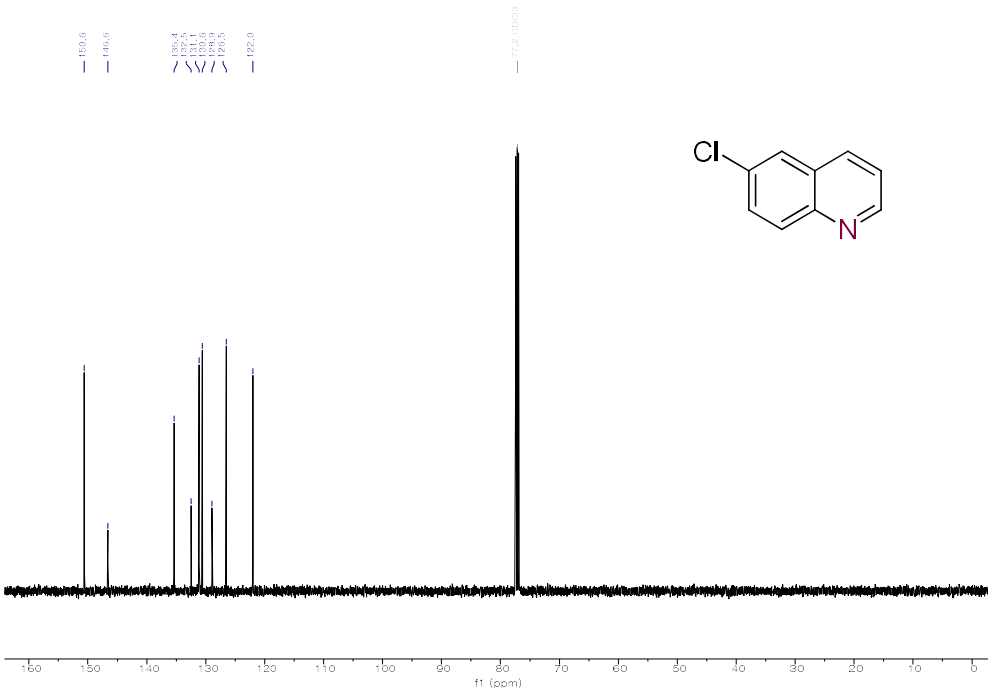


^{1}H & ^{13}C NMR Spectra of 6-Chloroquinoline (2o)

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

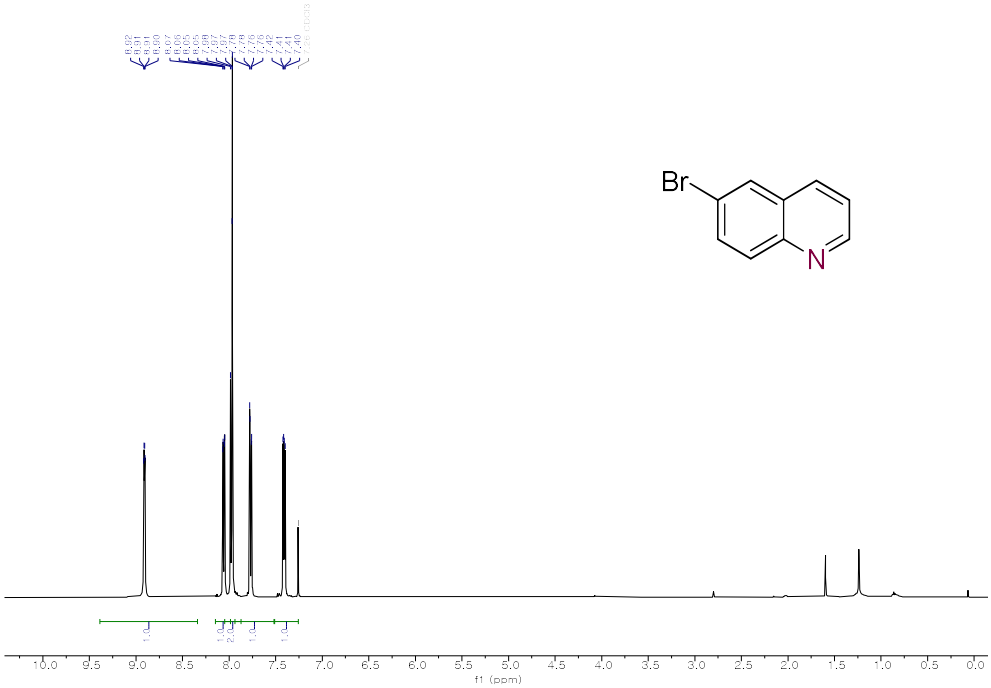


^{13}C NMR (126 MHz, $CDCl_3$)

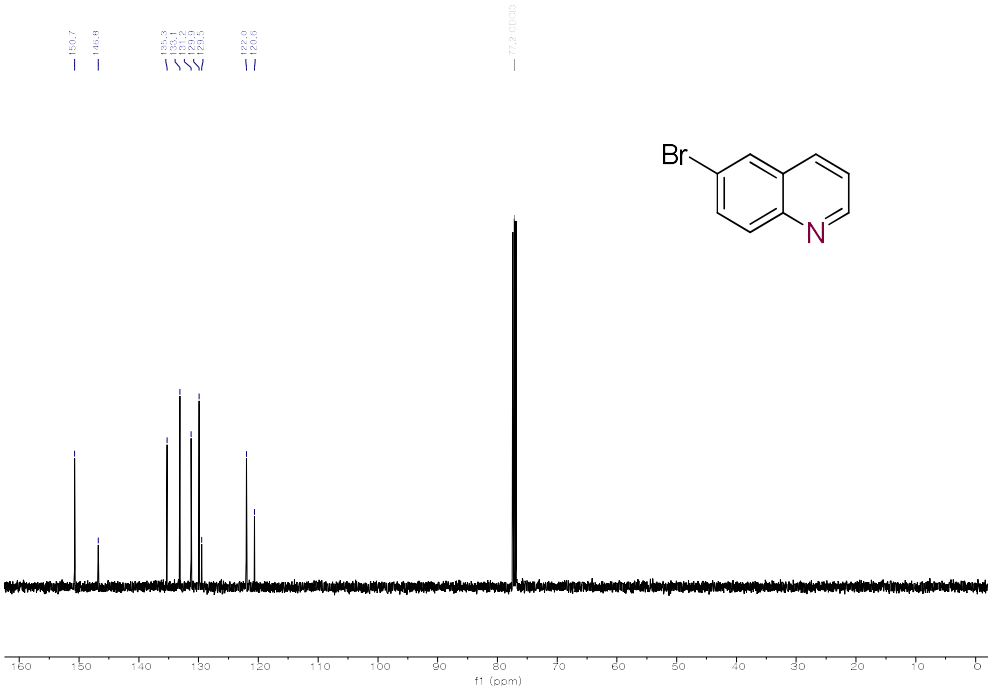


^{1}H & ^{13}C NMR Spectra of 6-Bromoquinoline (2p)

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

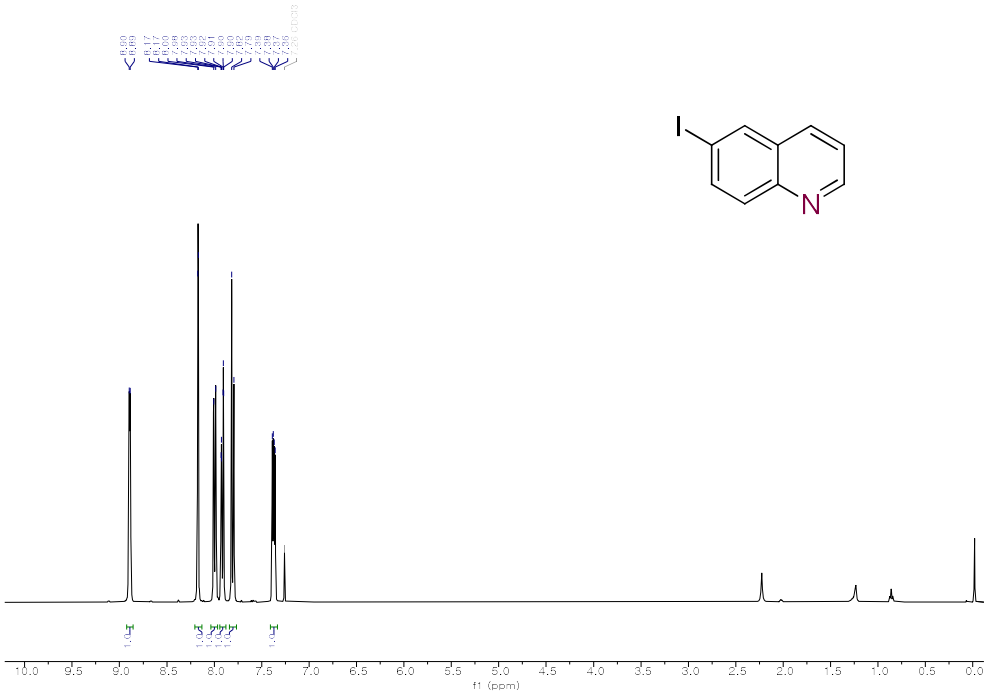


^{13}C NMR (126 MHz, $CDCl_3$)

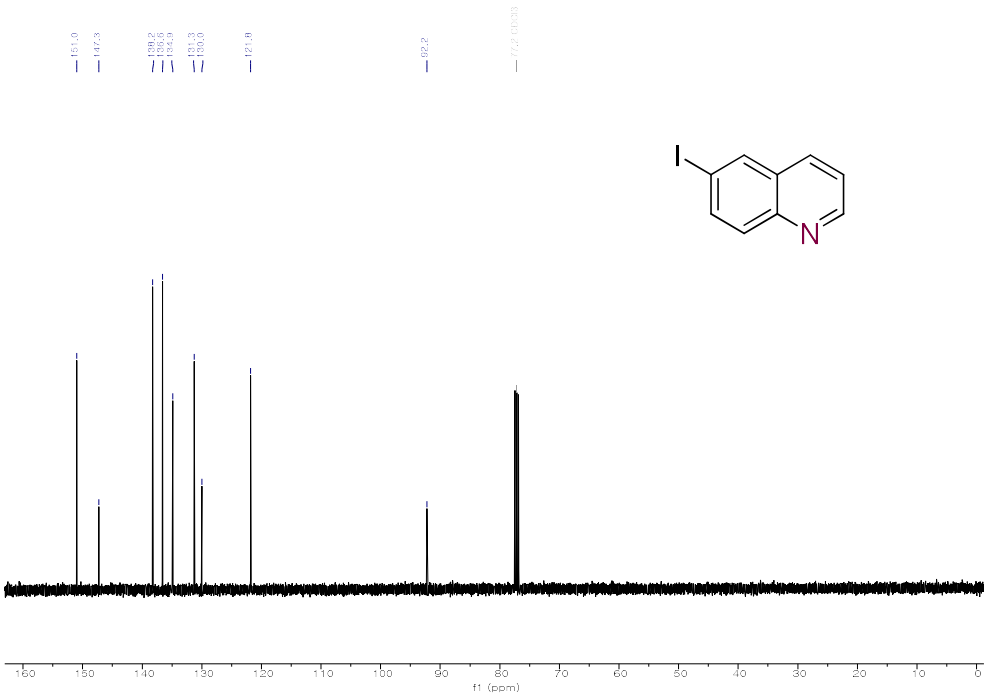


^{1}H & ^{13}C NMR Spectra of 6-Iodoquinoline (2q)

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

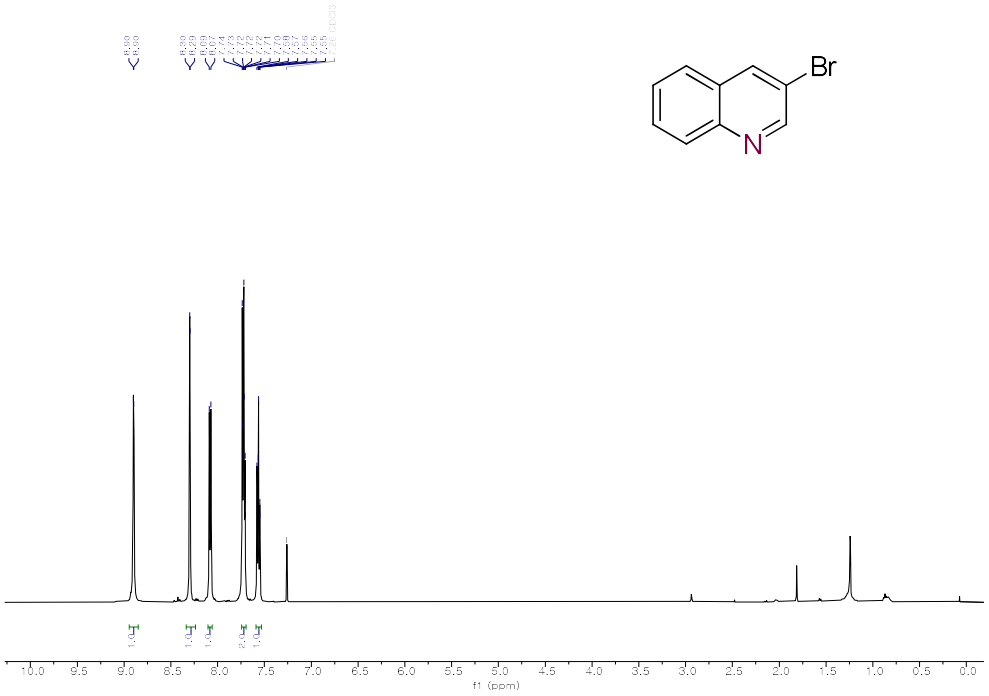


^{13}C NMR (101 MHz, $CDCl_3$)

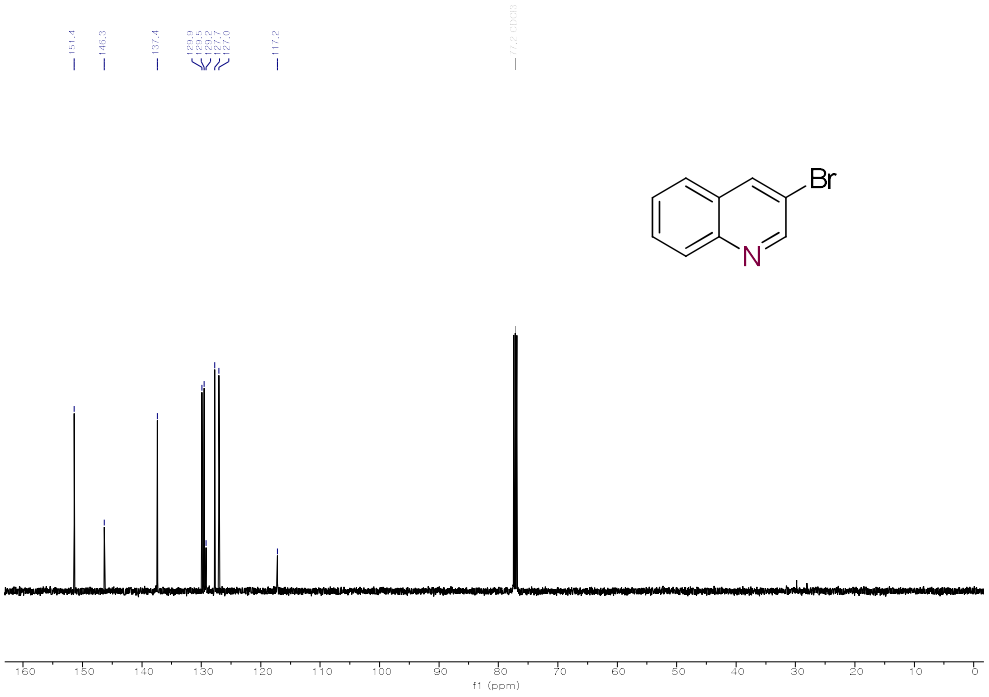


^{1}H & ^{13}C NMR Spectra of 3-Bromoquinoline (2r)

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

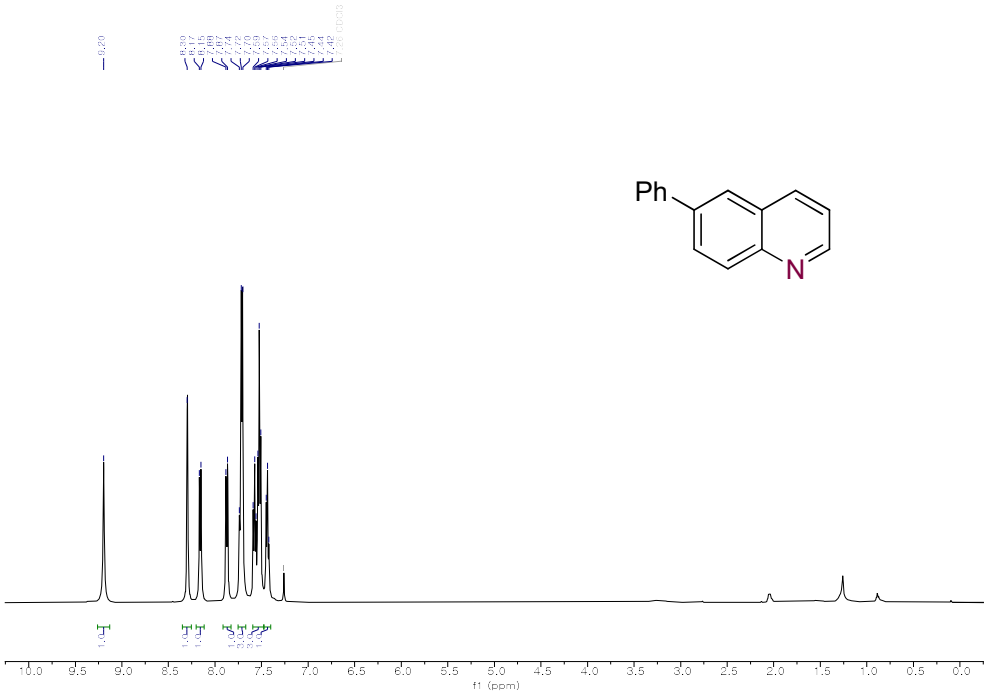


^{13}C NMR (126 MHz, $CDCl_3$)



^{1}H & ^{13}C NMR Spectra of 6-Phenylquinoline (2s)

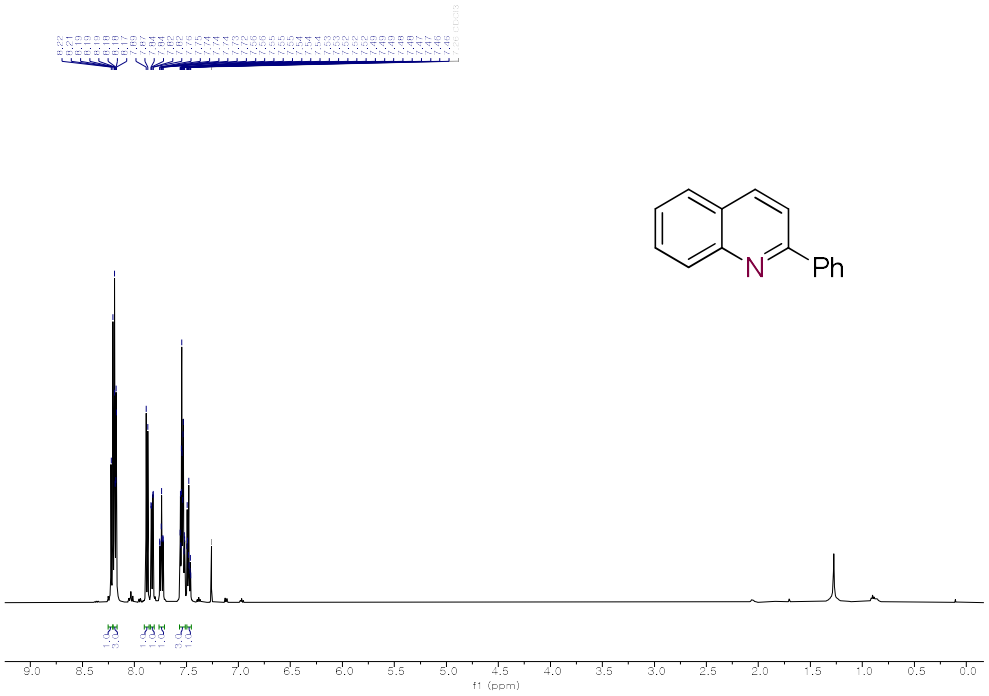
^{1}H NMR (500 MHz, $CDCl_3$)



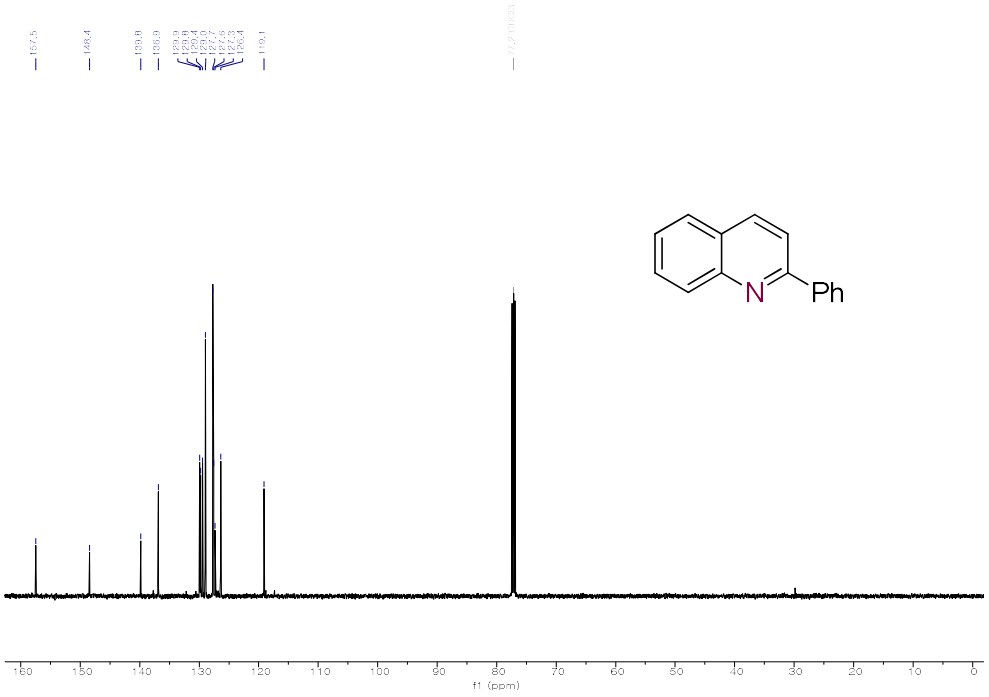
^{13}C NMR (126 MHz, $CDCl_3$)

^{1}H & ^{13}C NMR Spectra of 2-Phenylquinoline (2t)

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

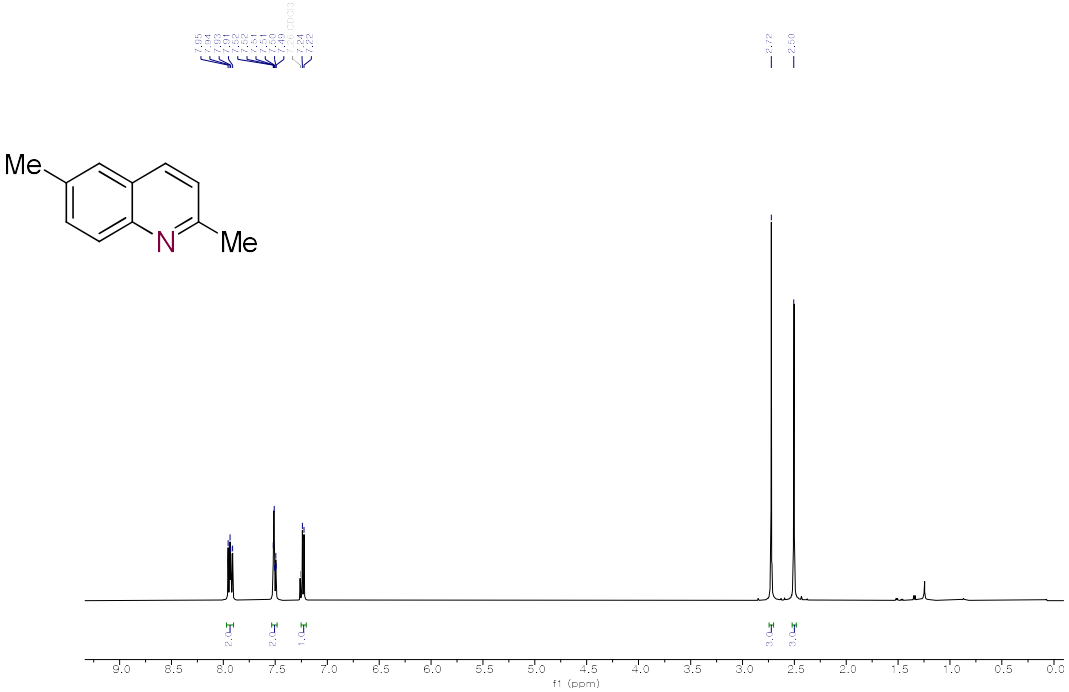


^{13}C NMR (126 MHz, $CDCl_3$)

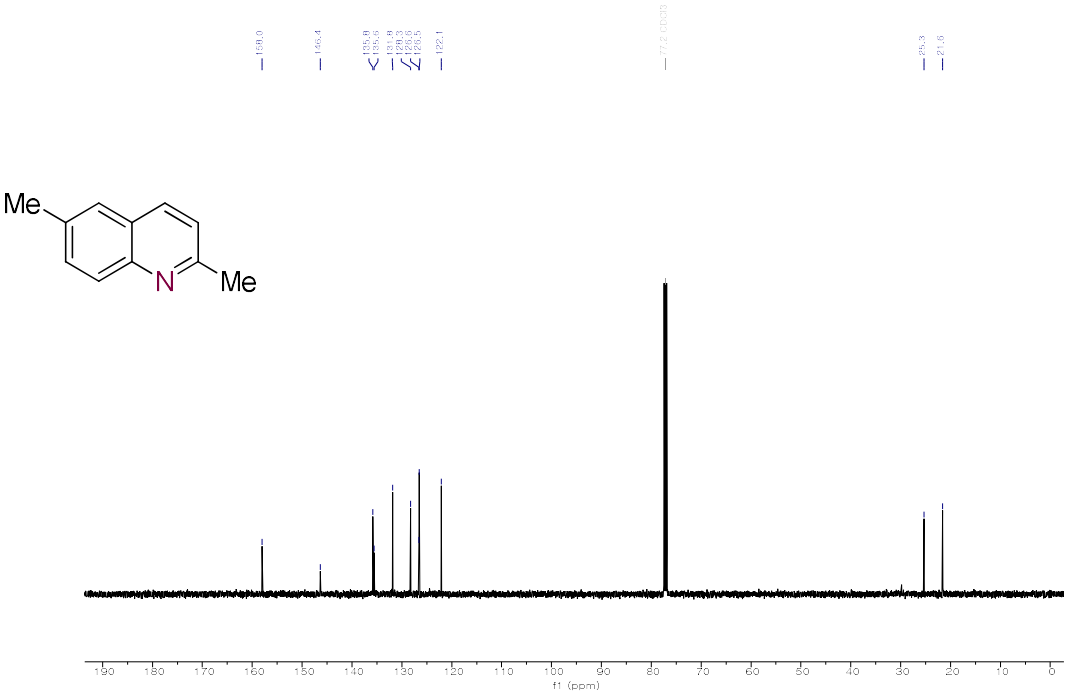


^{1}H & ^{13}C NMR Spectra of 2,6-Dimethylquinoline (2u)

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

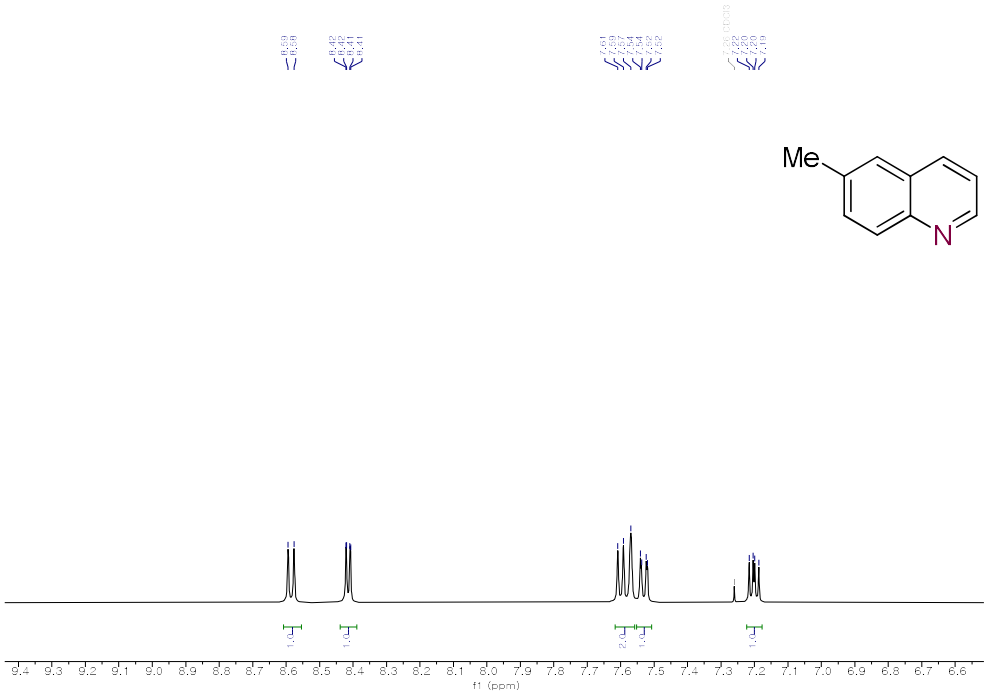


^{13}C NMR (126 MHz, $CDCl_3$)

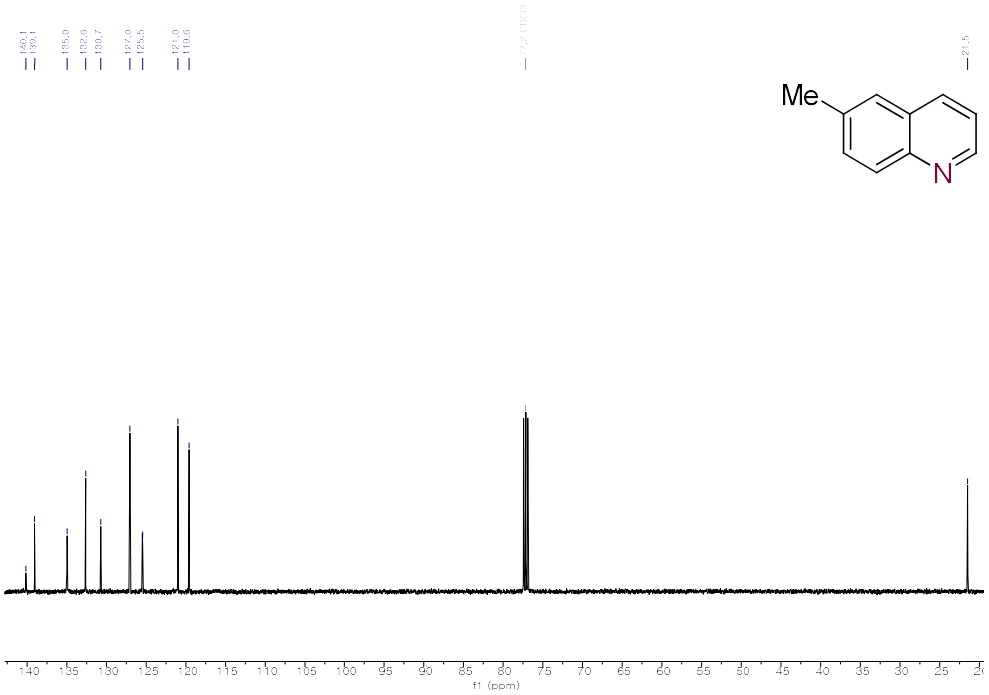


^{1}H & ^{13}C NMR Spectra of 6-Methylquinoline (2v)

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

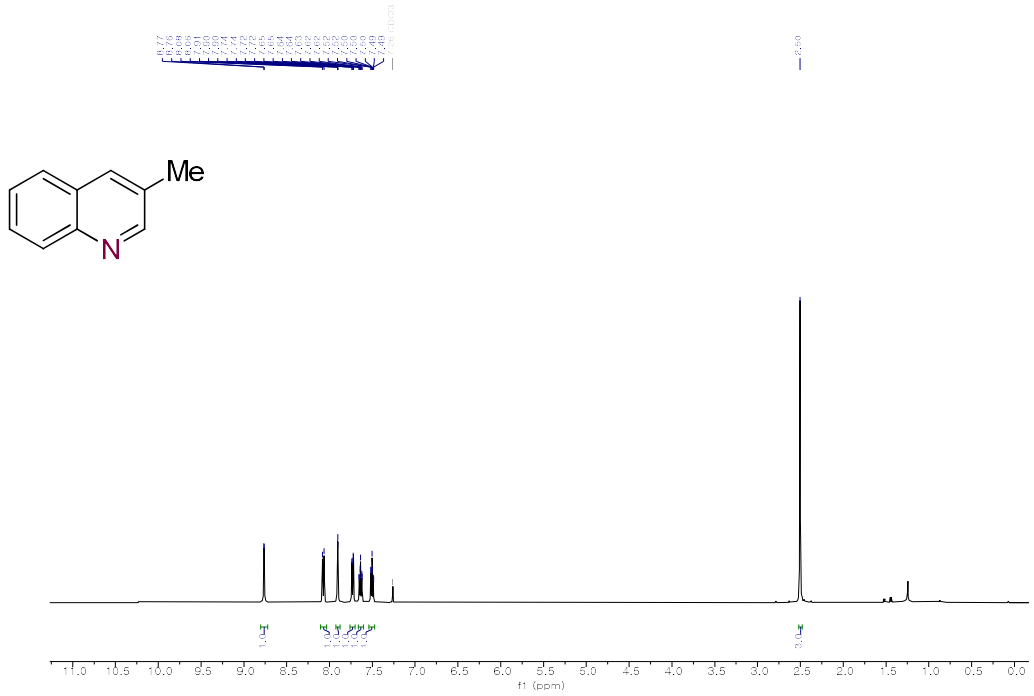


^{13}C NMR (126 MHz, $CDCl_3$)

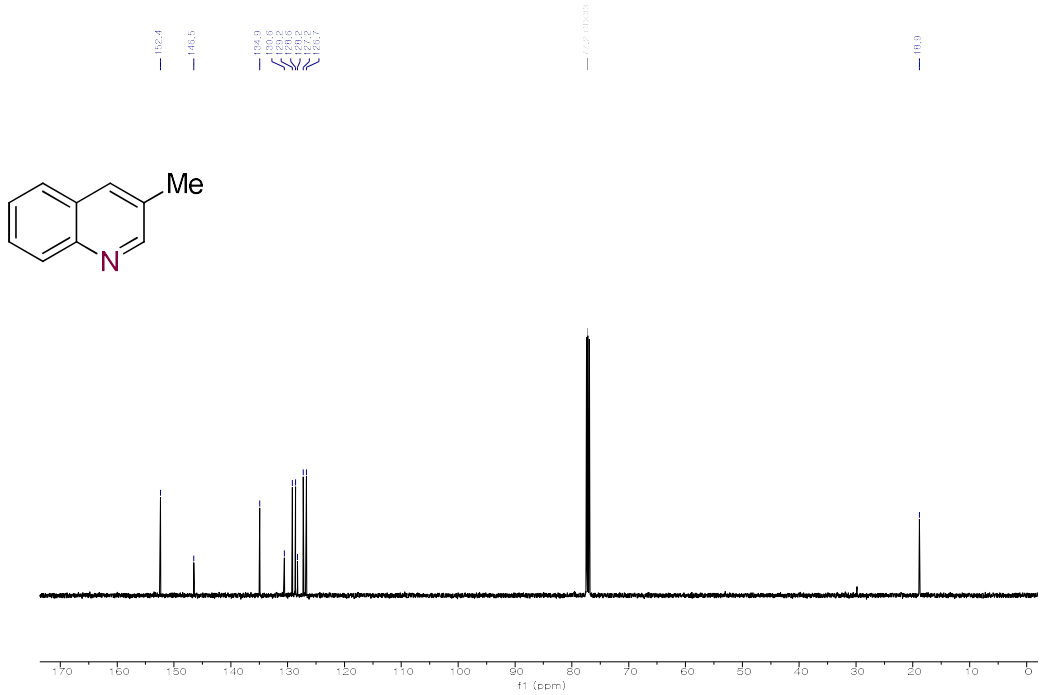


^{1}H & ^{13}C NMR Spectra of 3-Methylquinoline (2w)

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

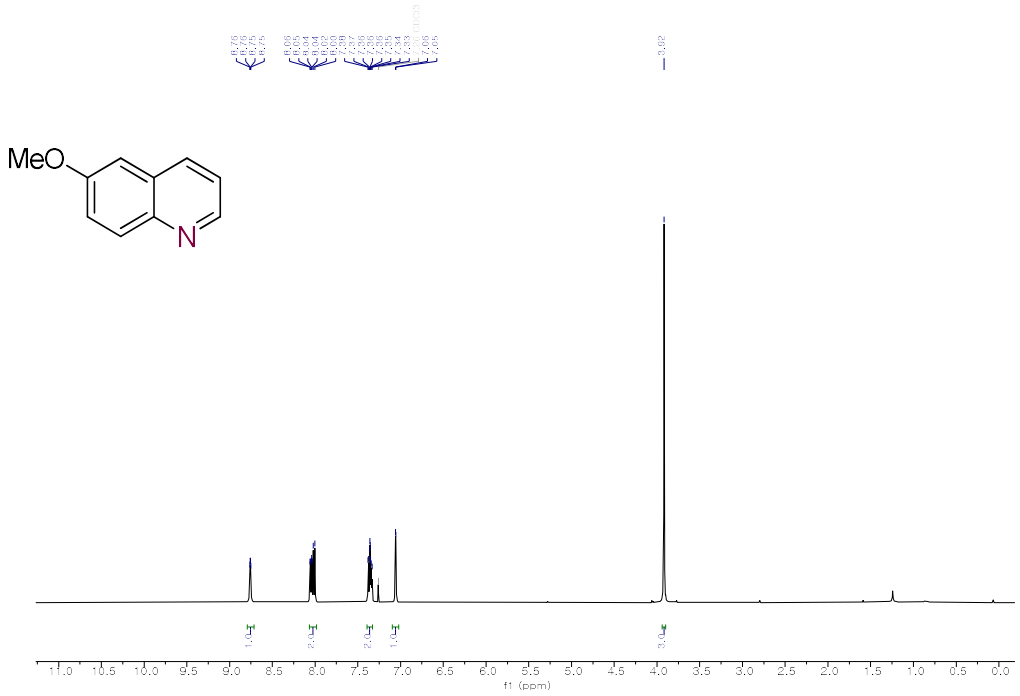


^{13}C NMR (126 MHz, $CDCl_3$)

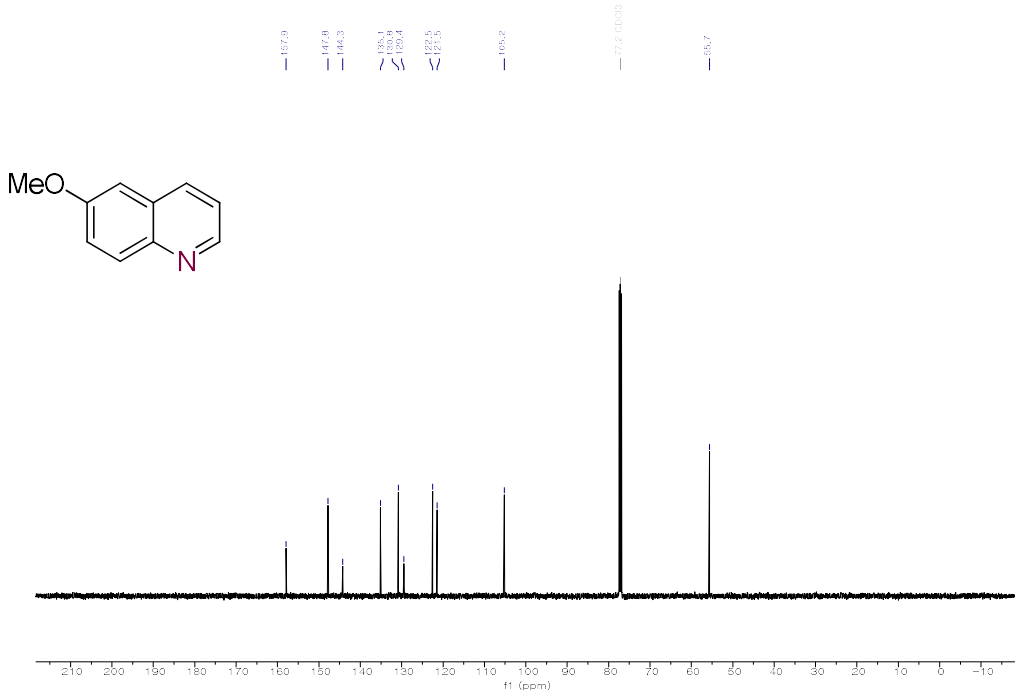


^{1}H & ^{13}C NMR Spectra of 6-Methoxyquinoline (2x)

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

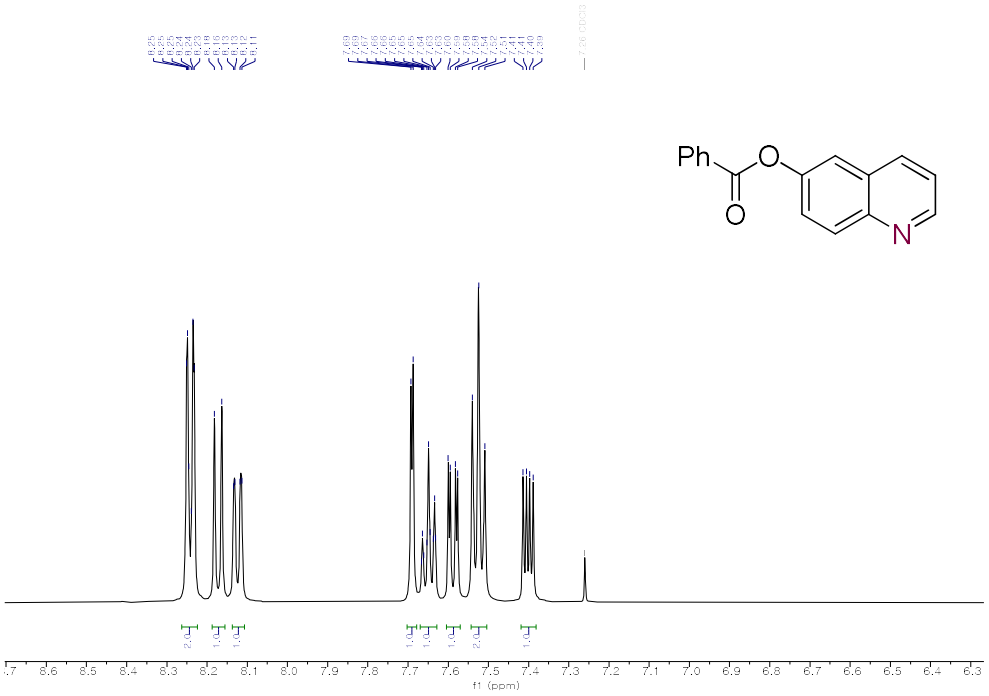


^{13}C NMR (126 MHz, $CDCl_3$)

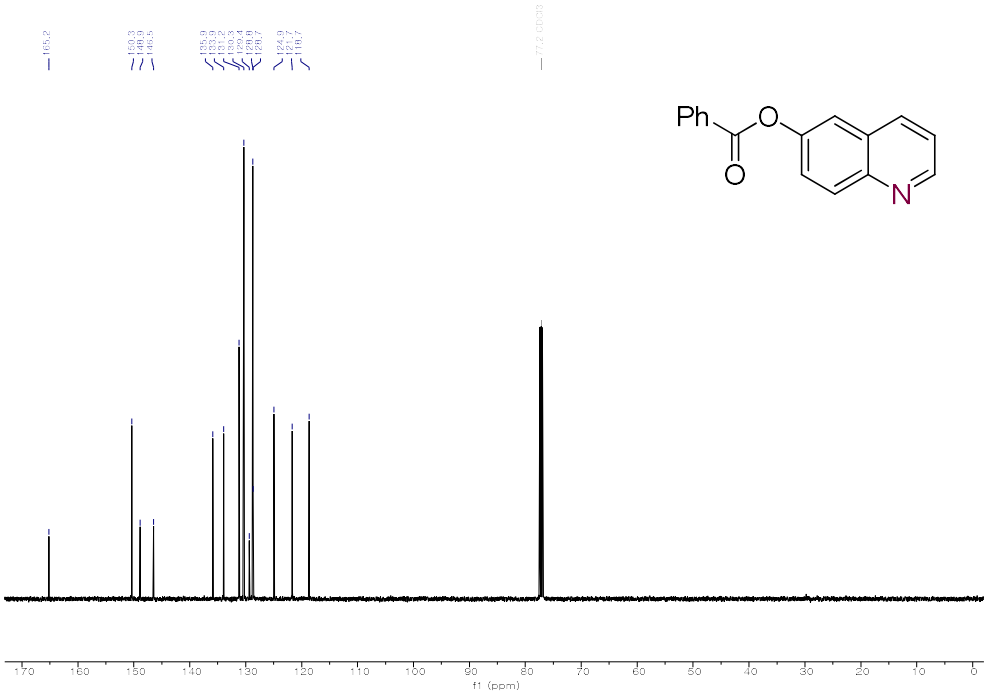


^{1}H & ^{13}C NMR Spectra of 6-Benzoyloxyquinoline (2y)

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

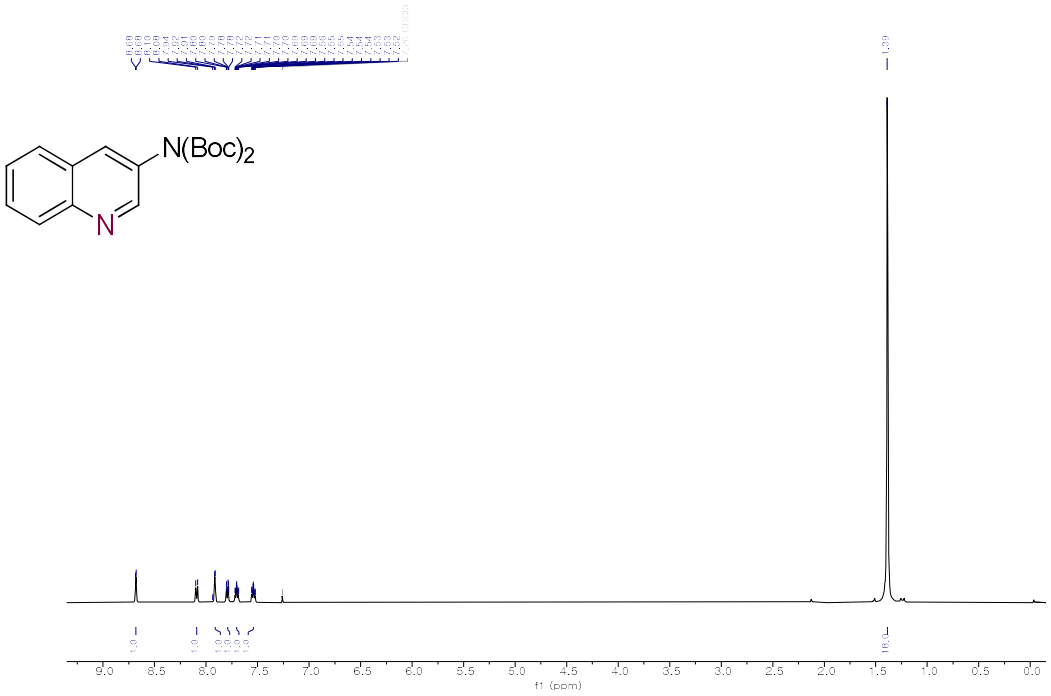


^{13}C NMR (126 MHz, $CDCl_3$)

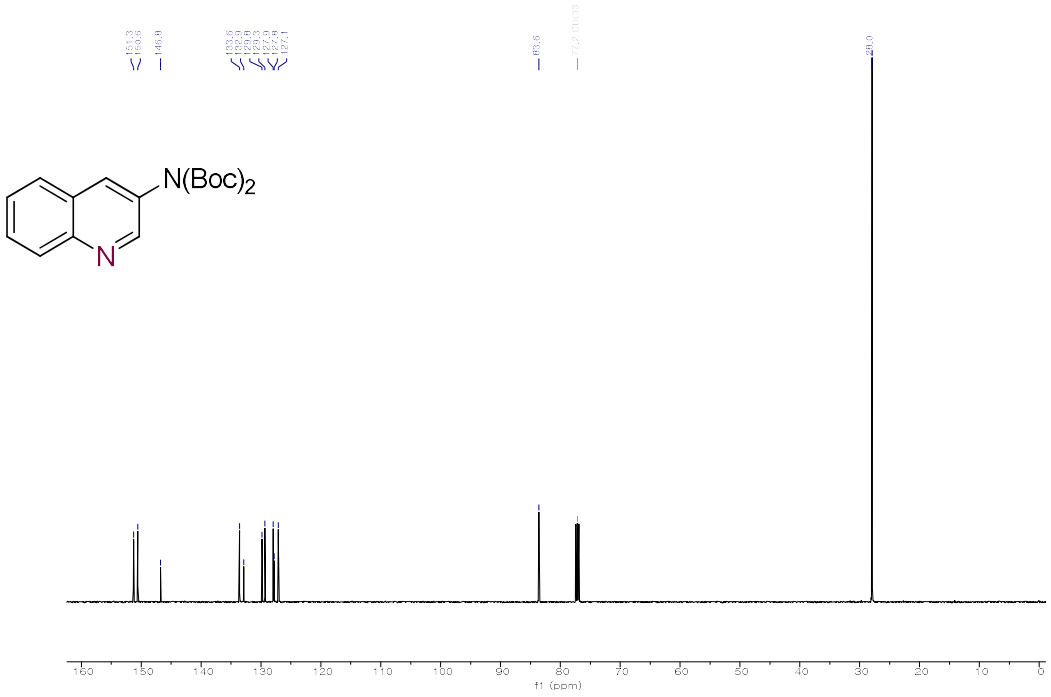


^{1}H & ^{13}C NMR Spectra of 3-[Bis(*tert*-butoxycarbonyl)amino]quinoline (2z)

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

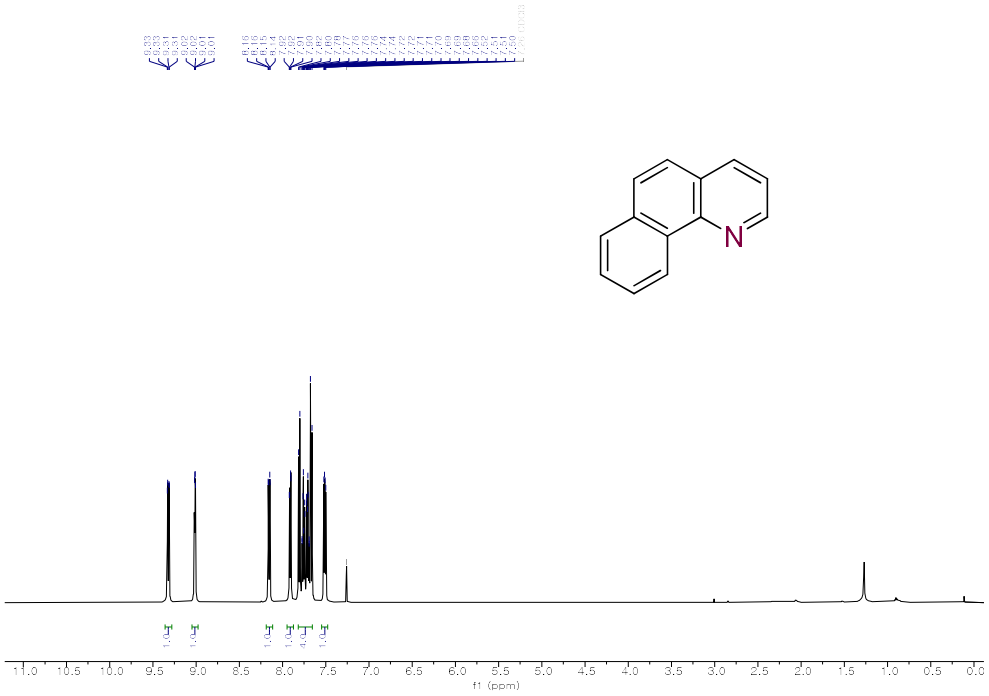


^{13}C NMR (126 MHz, $CDCl_3$)

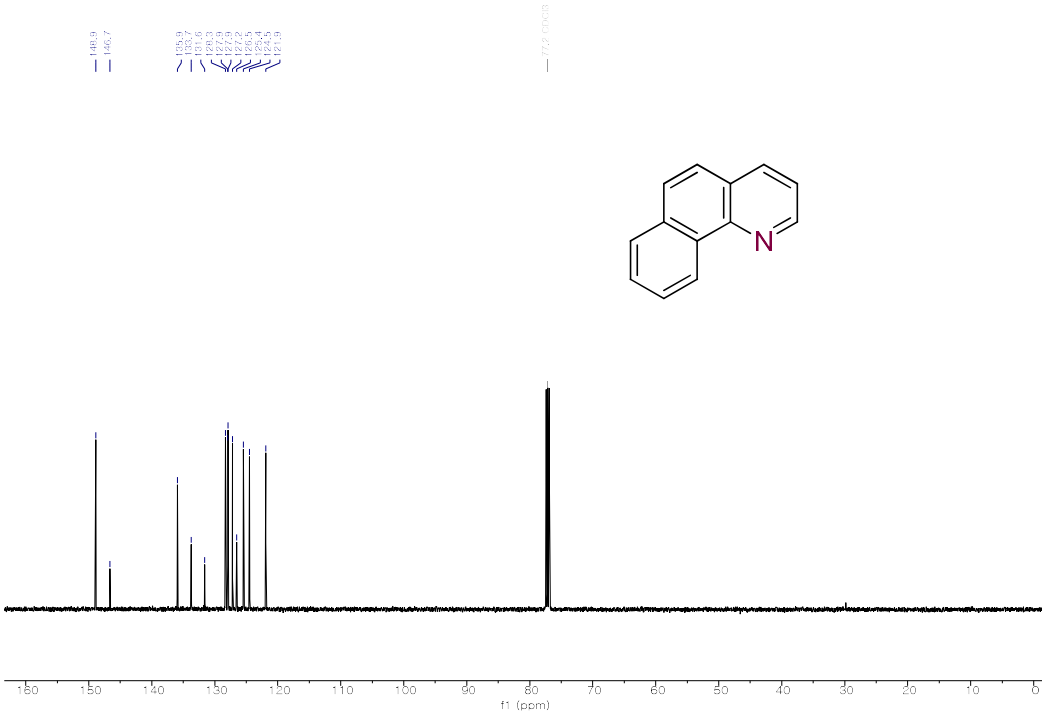


^{1}H & ^{13}C NMR Spectra of Benzo[*h*]quinoline (2aa)

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

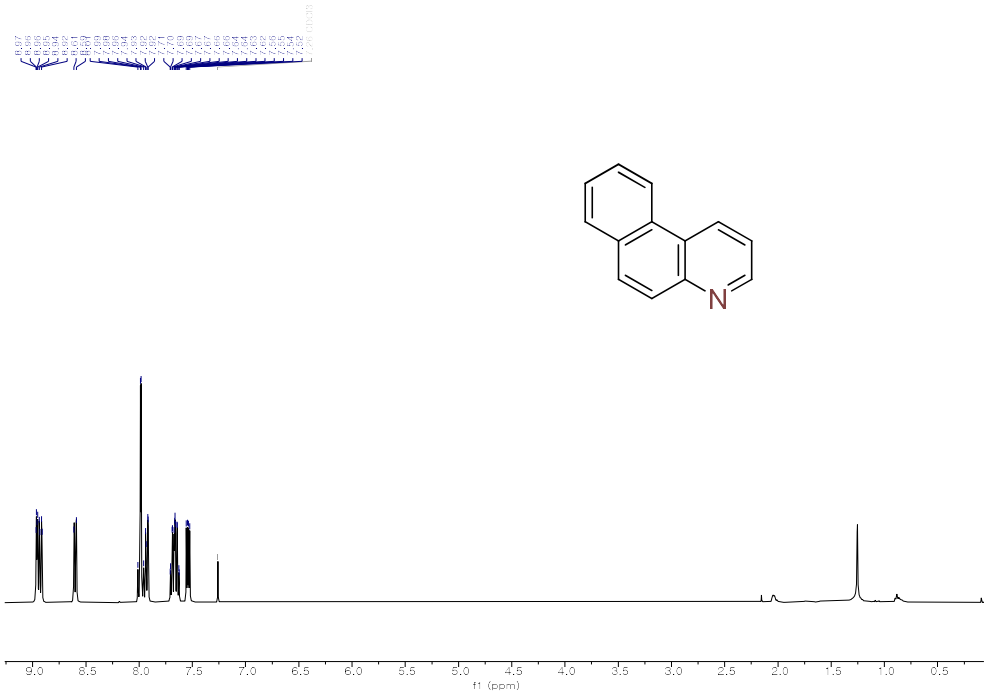


^{13}C NMR (126 MHz, $CDCl_3$)

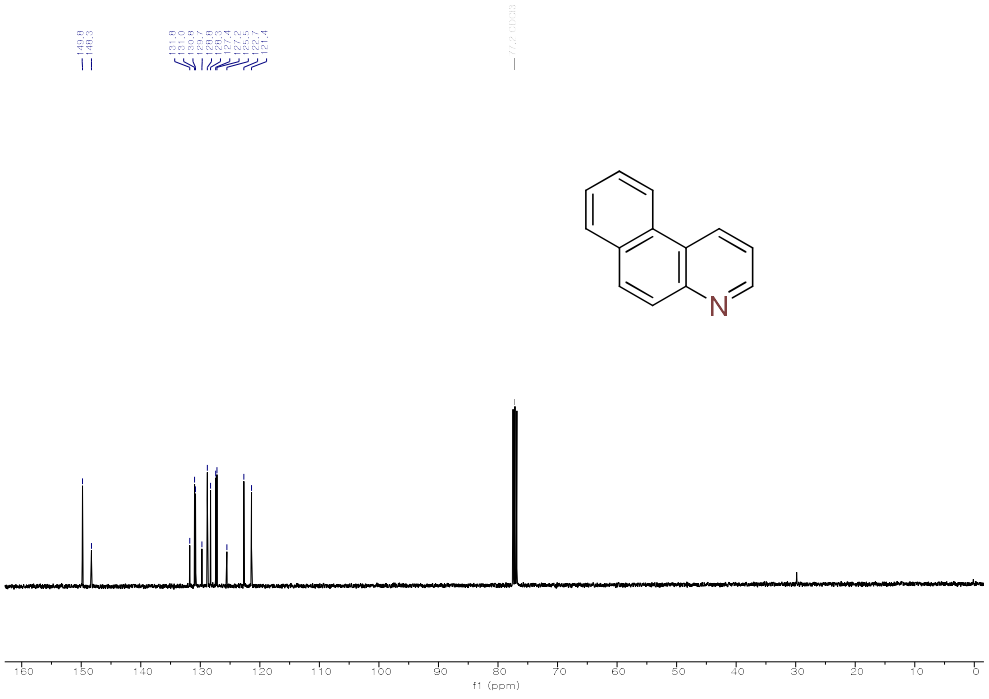


^{1}H & ^{13}C NMR Spectra of Benzo[*f*]quinoline (2ab)

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

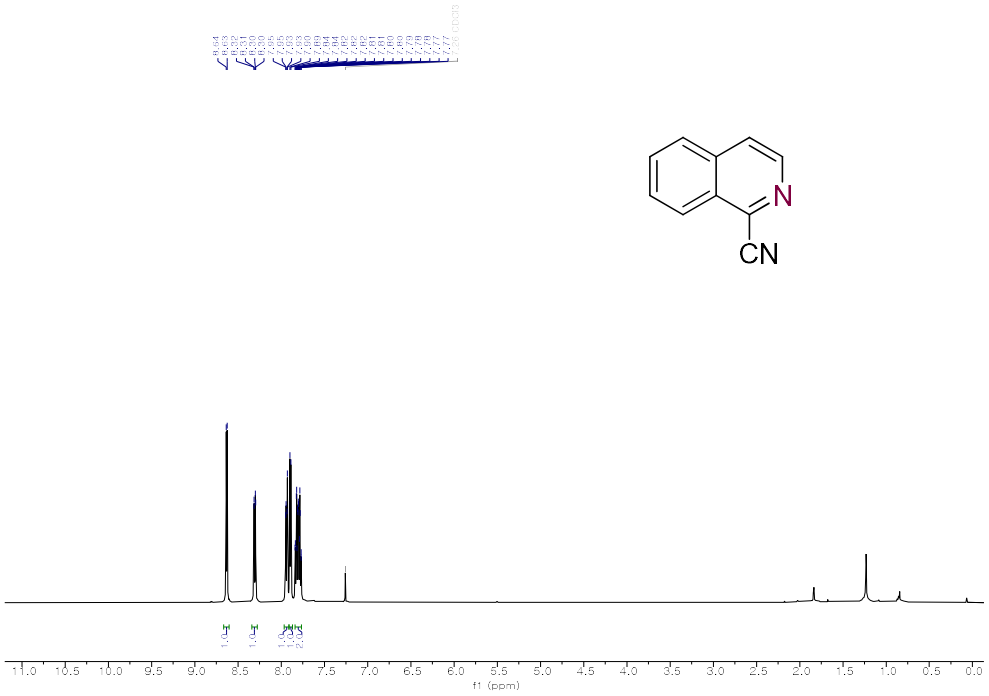


^{13}C NMR (101 MHz, $CDCl_3$)

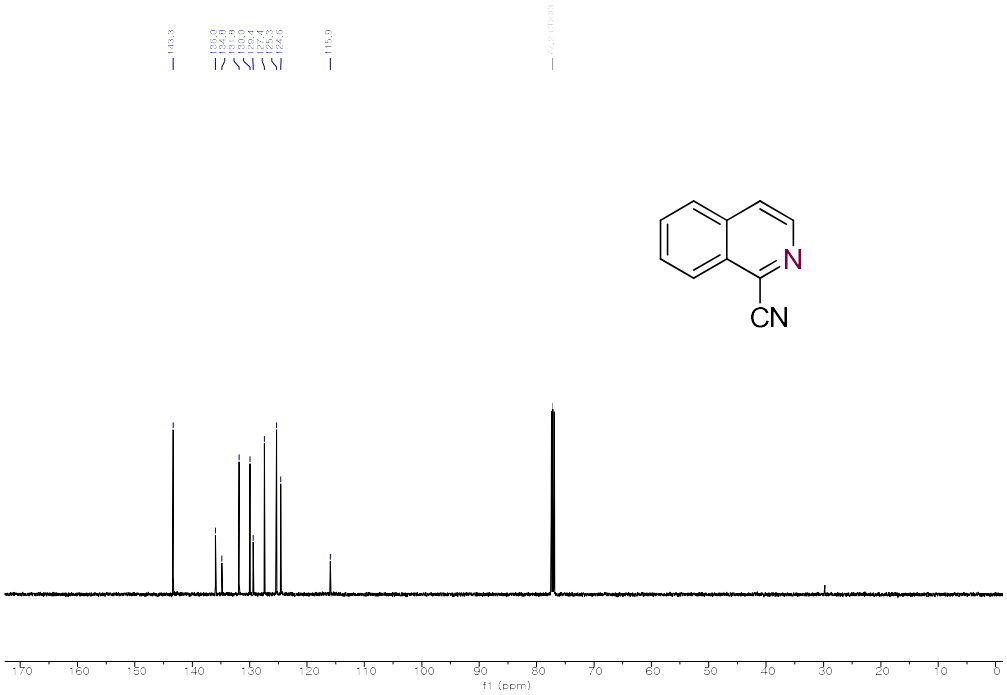


^{1}H & ^{13}C NMR Spectra of 1-Isoquinolinecarbonitrile (2ac)

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

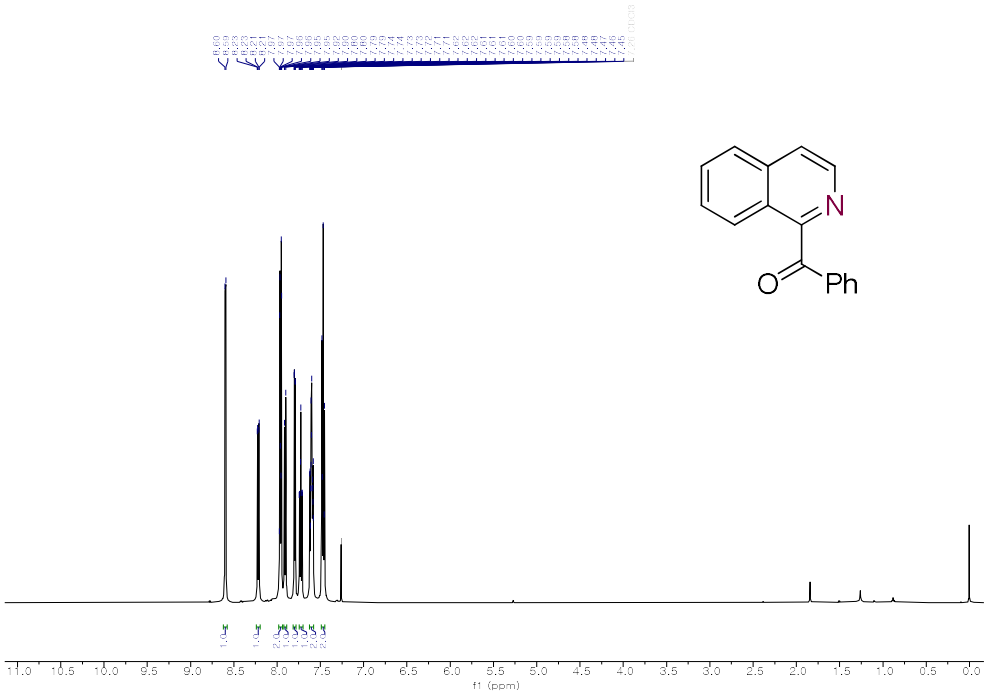


^{13}C NMR (126 MHz, $CDCl_3$)

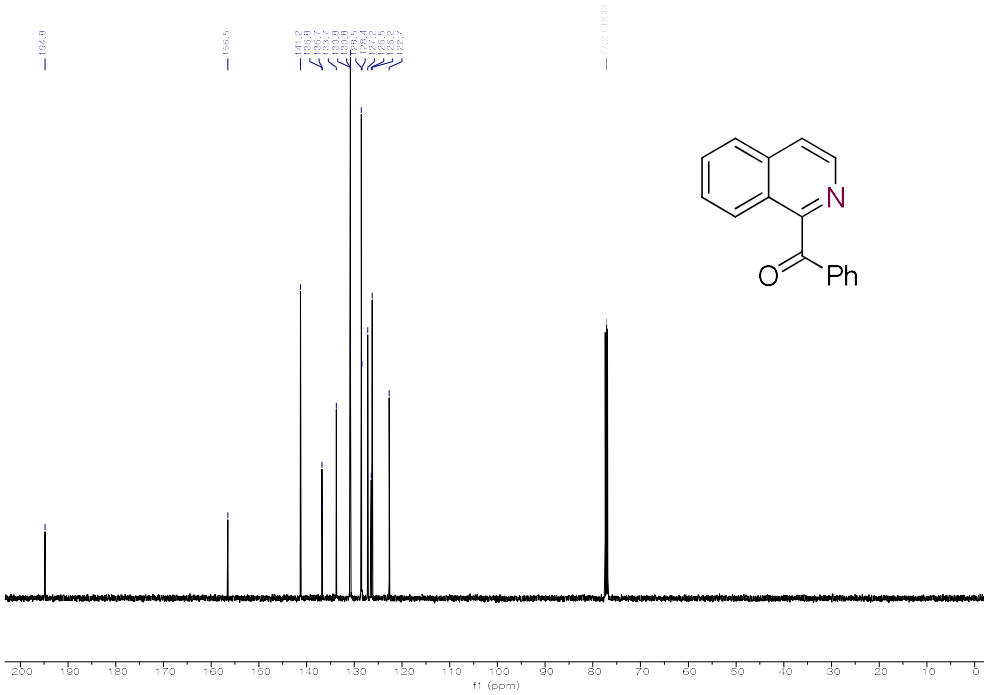


^{1}H & ^{13}C NMR Spectra of 1-Benzoylisoquinoline (2ad)

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

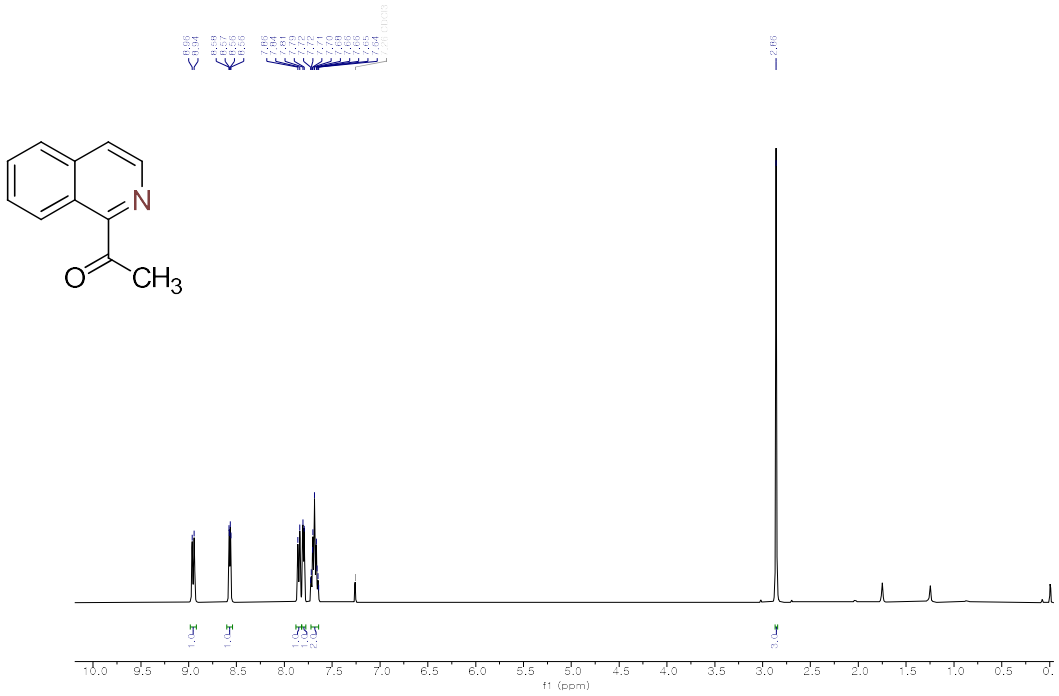


^{13}C NMR (126 MHz, $CDCl_3$)

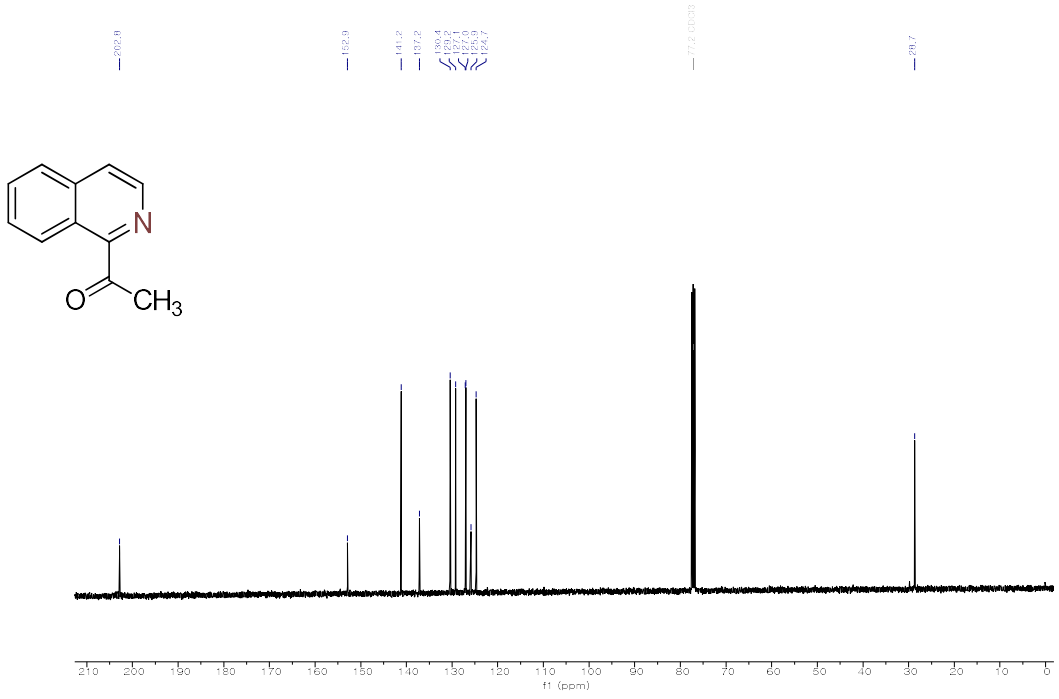


^{1}H & ^{13}C NMR Spectra of 1-Acetylisoquinoline (2ae)

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

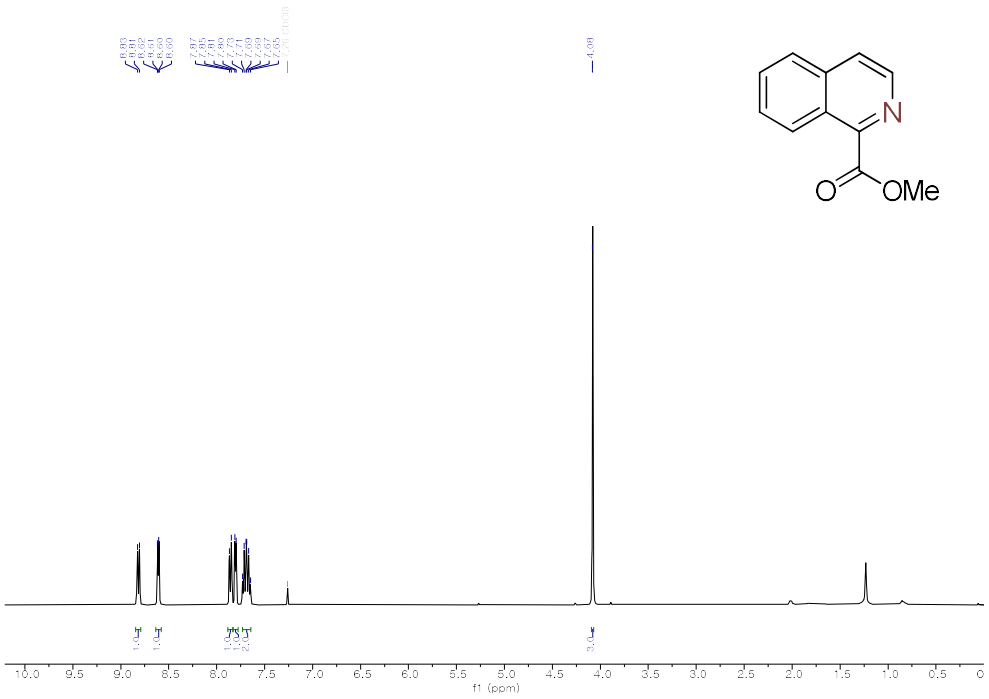


^{13}C NMR (101 MHz, $CDCl_3$)

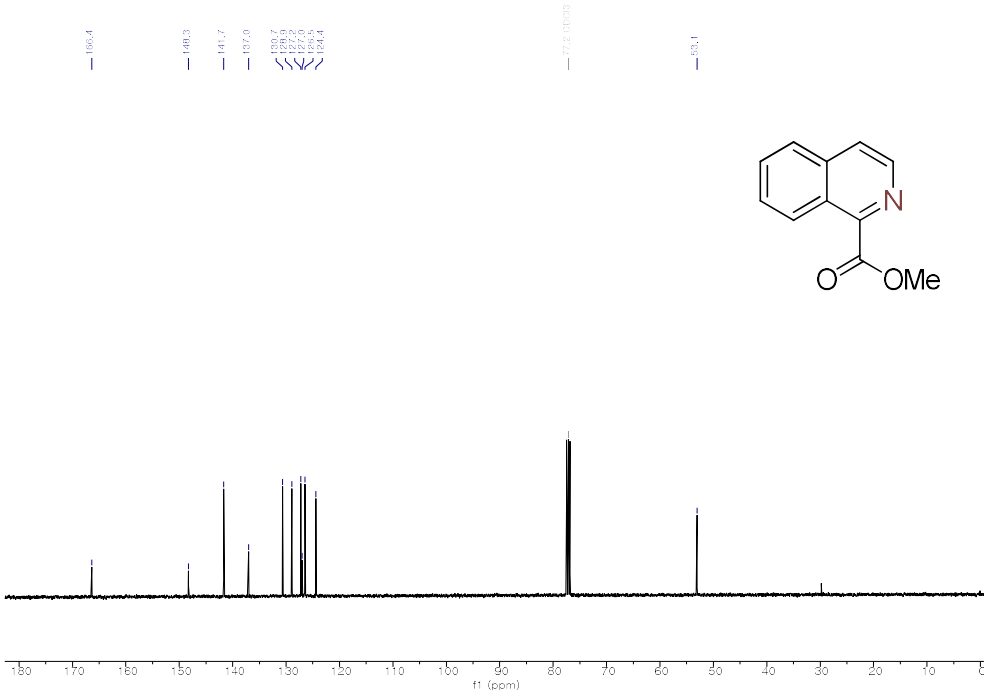


Preparation of Methyl 1-Isoquinolinecarboxylate (2af)

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

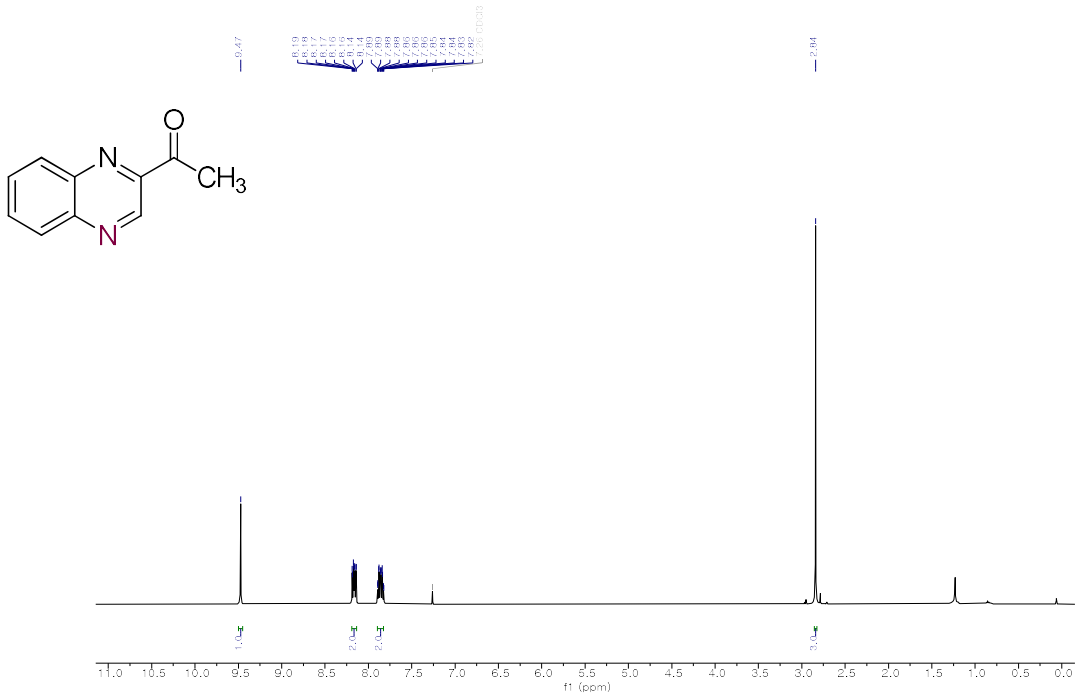


^{13}C NMR (101 MHz, $CDCl_3$)

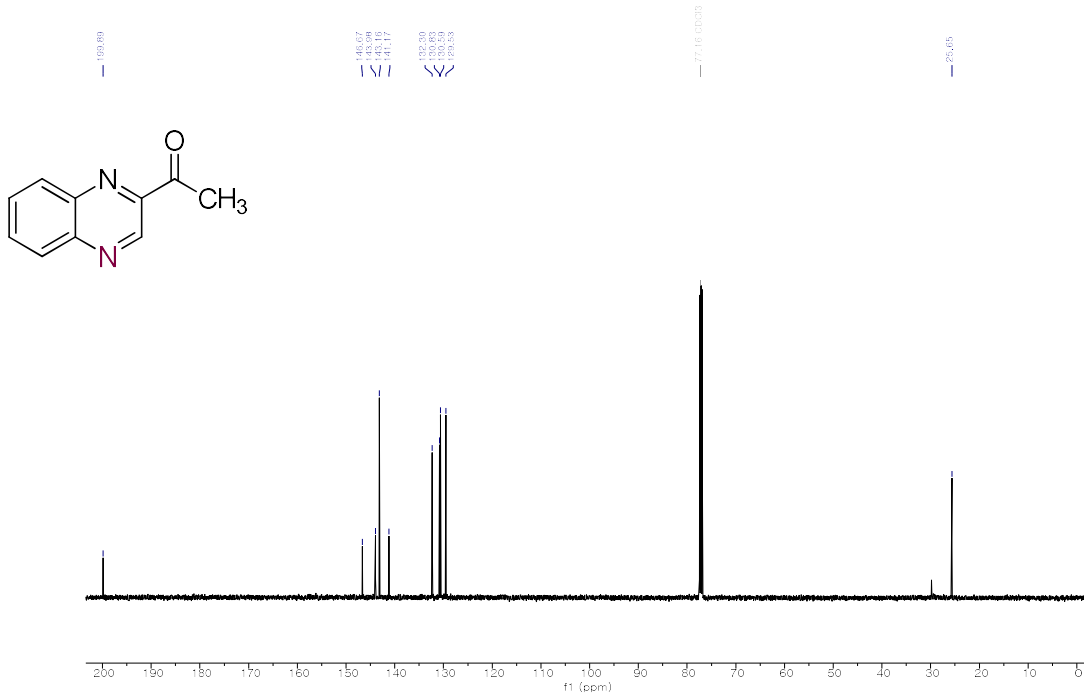


^{1}H & ^{13}C NMR Spectra of Methyl 2-Quinoxalinecarboxylate (2ag)

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

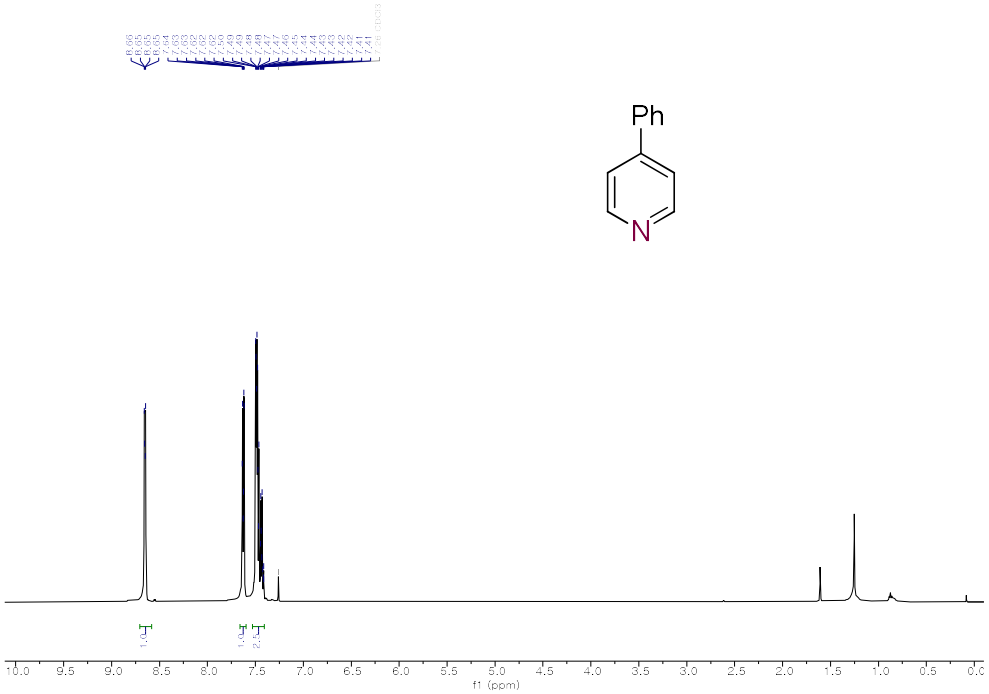


^{13}C NMR (126 MHz, $CDCl_3$)

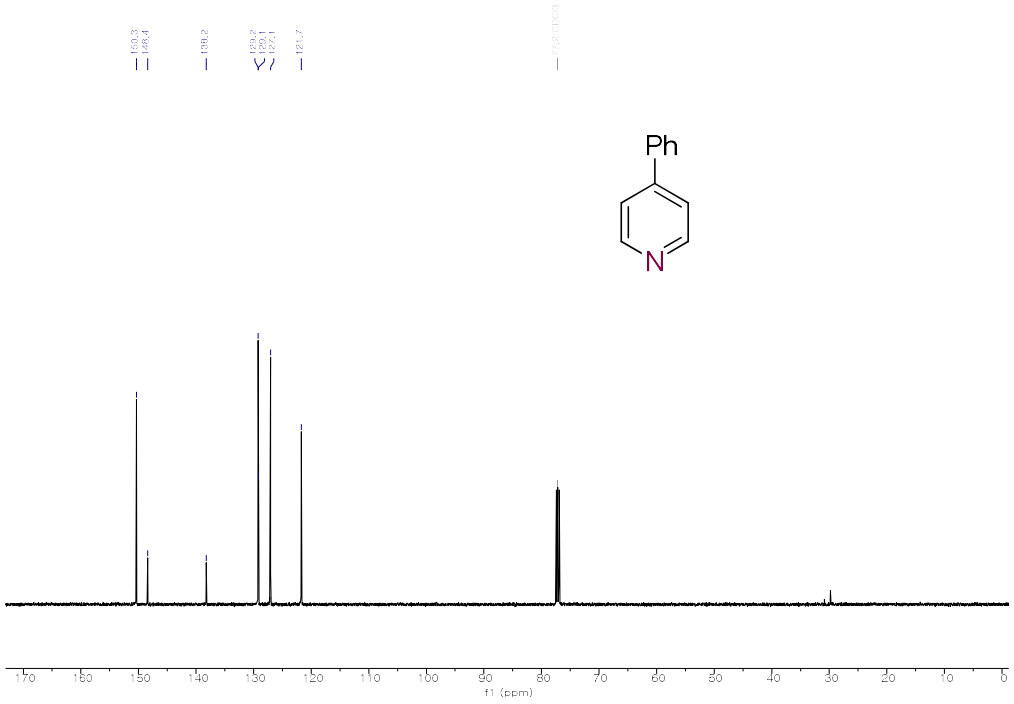


^{1}H & ^{13}C NMR Spectra of 4-Phenylpyridine (2ah)

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

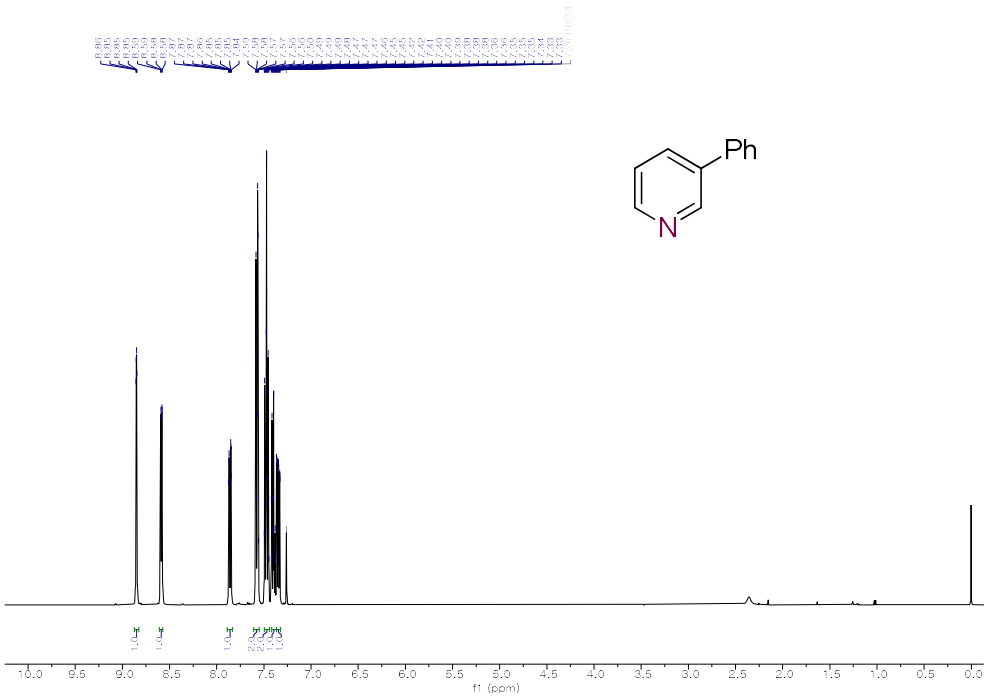


^{13}C NMR (126 MHz, $CDCl_3$)

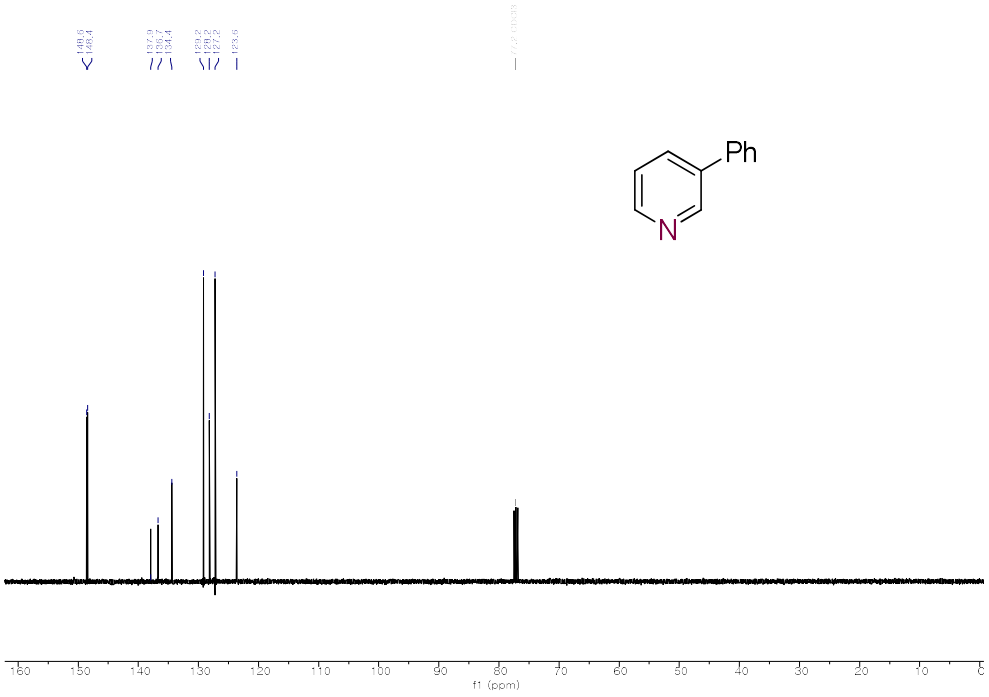


^{1}H & ^{13}C NMR Spectra of 3-Phenylpyridine (2ai)

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

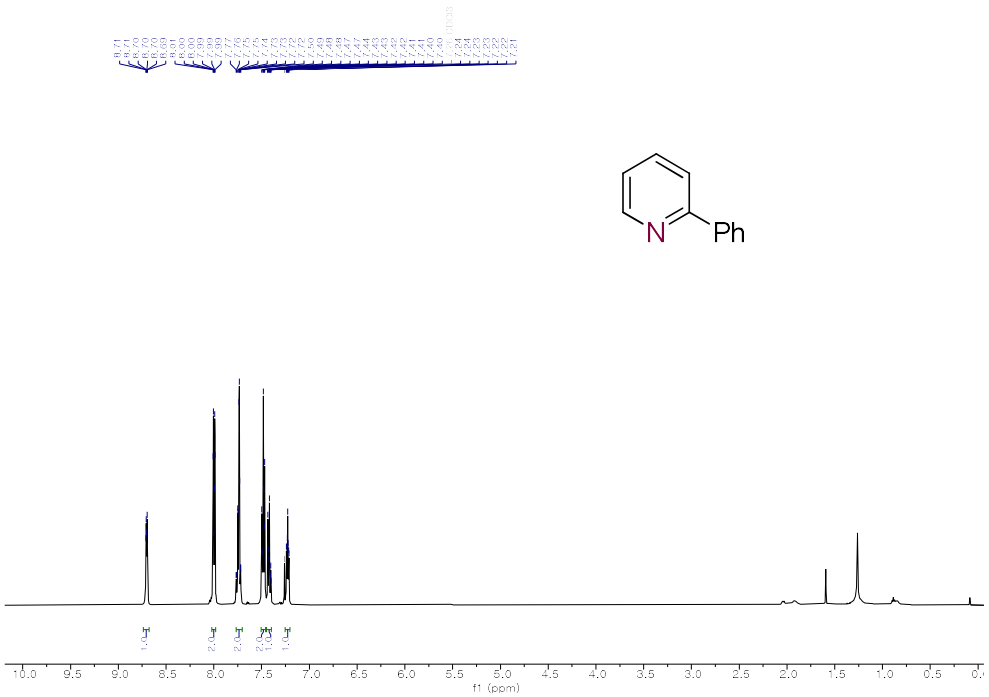


^{13}C NMR (101 MHz, $CDCl_3$)

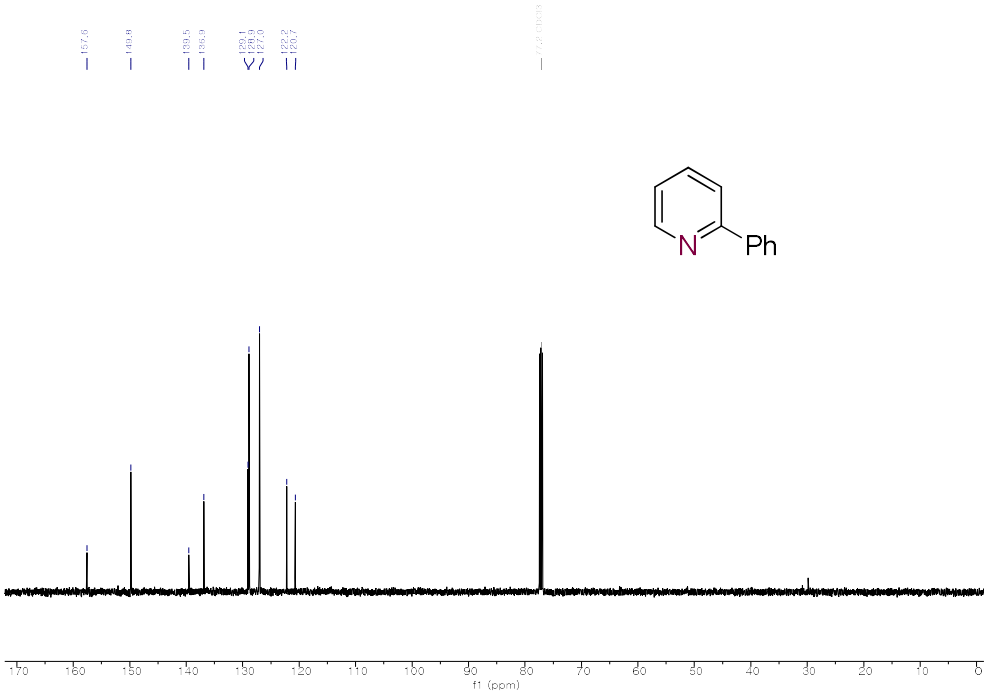


^{1}H & ^{13}C NMR Spectra of 2-Phenylpyridine (2aj)

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

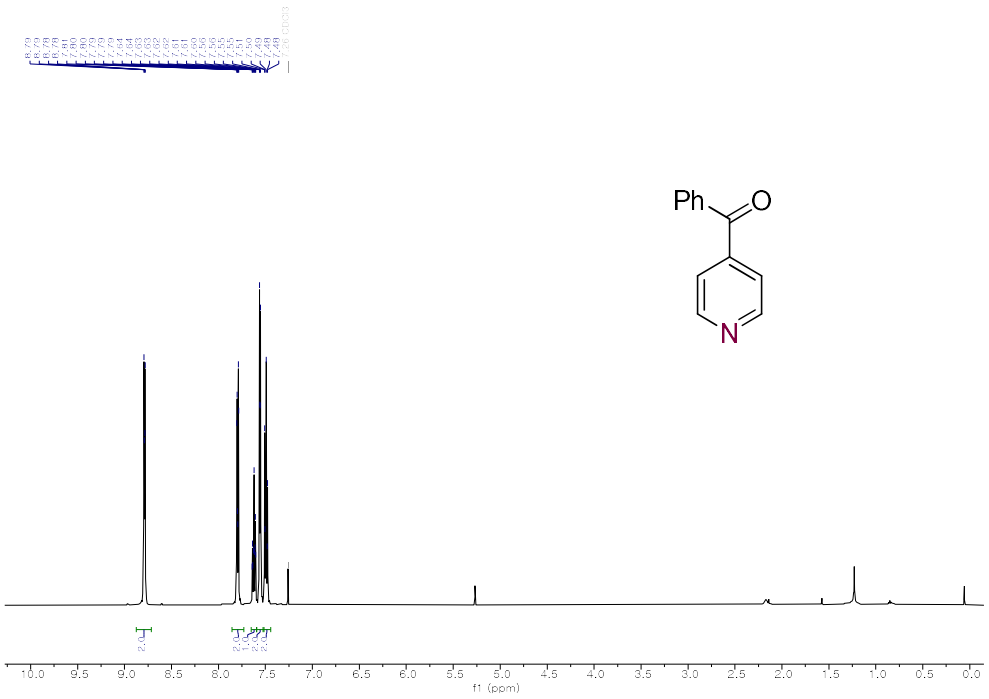


^{13}C NMR (126 MHz, $CDCl_3$)

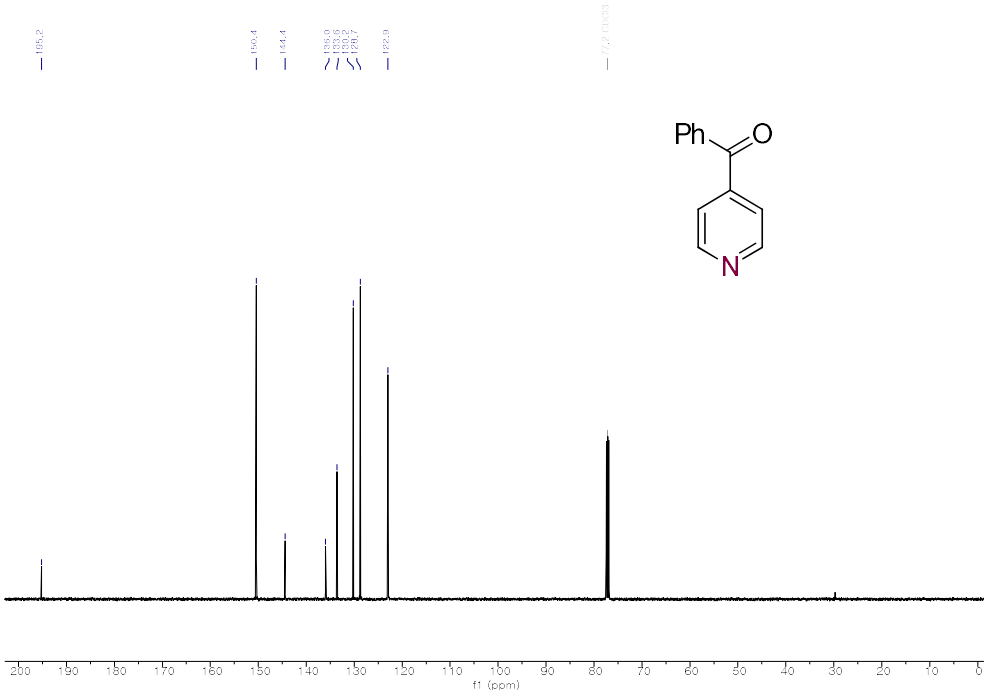


^{1}H & ^{13}C NMR Spectra of 4-Benzoylpyridine (2ak)

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

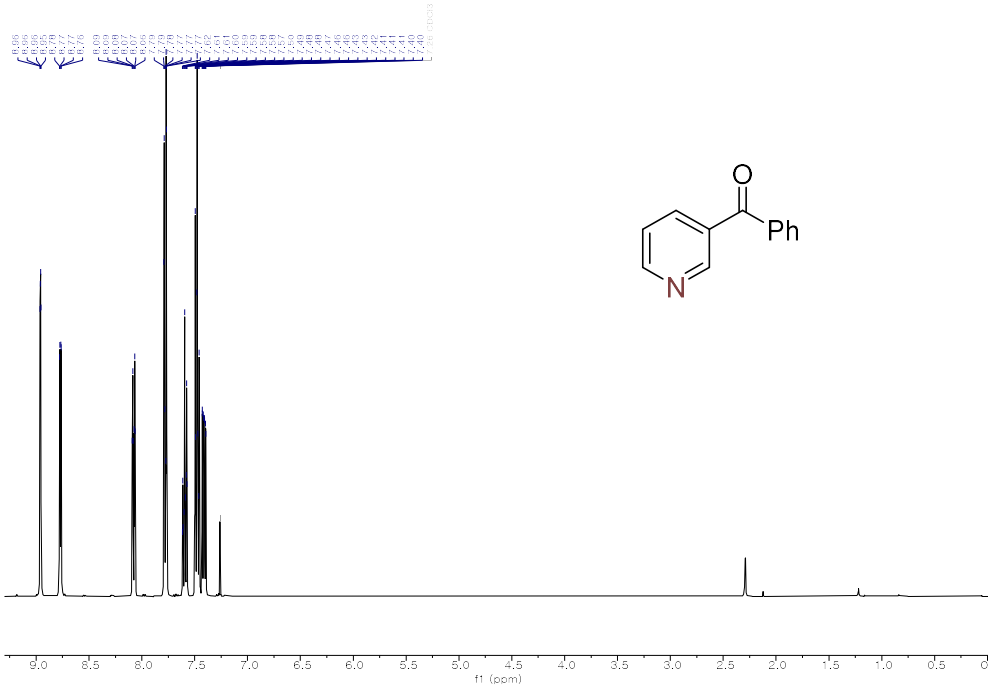


^{13}C NMR (126 MHz, $CDCl_3$)

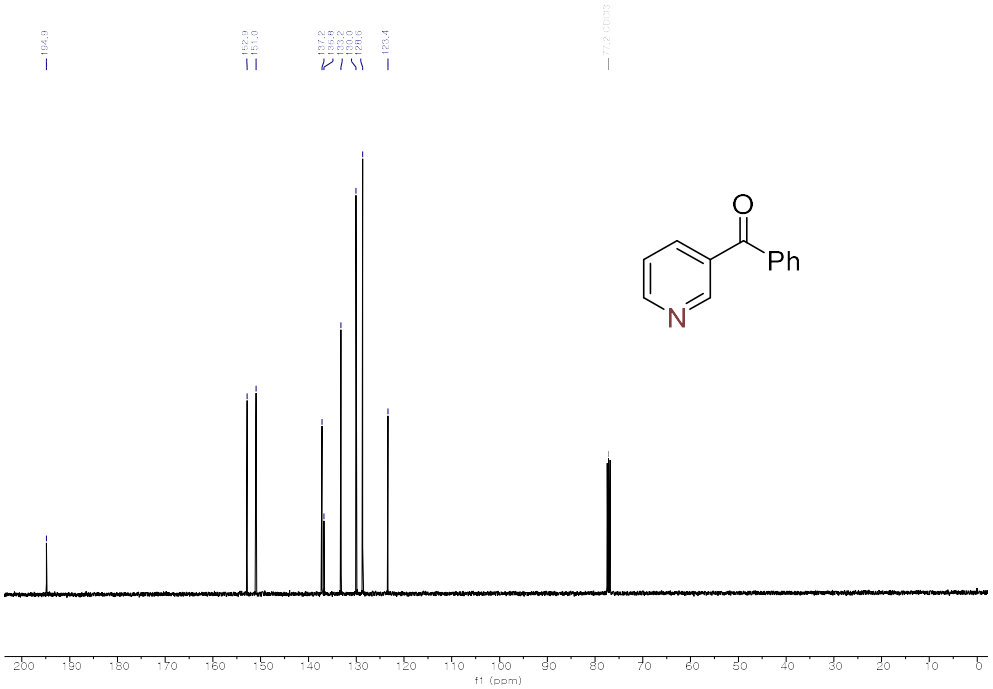


^{1}H & ^{13}C NMR Spectra of 3-Benzoylpyridine (2al)

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

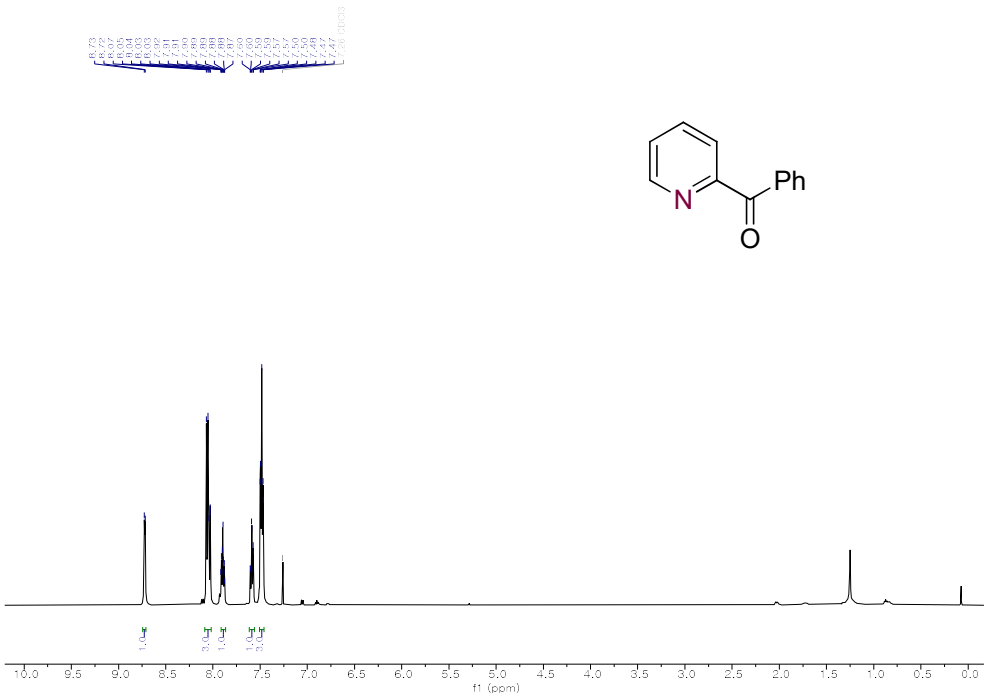


^{13}C NMR (126 MHz, $CDCl_3$)

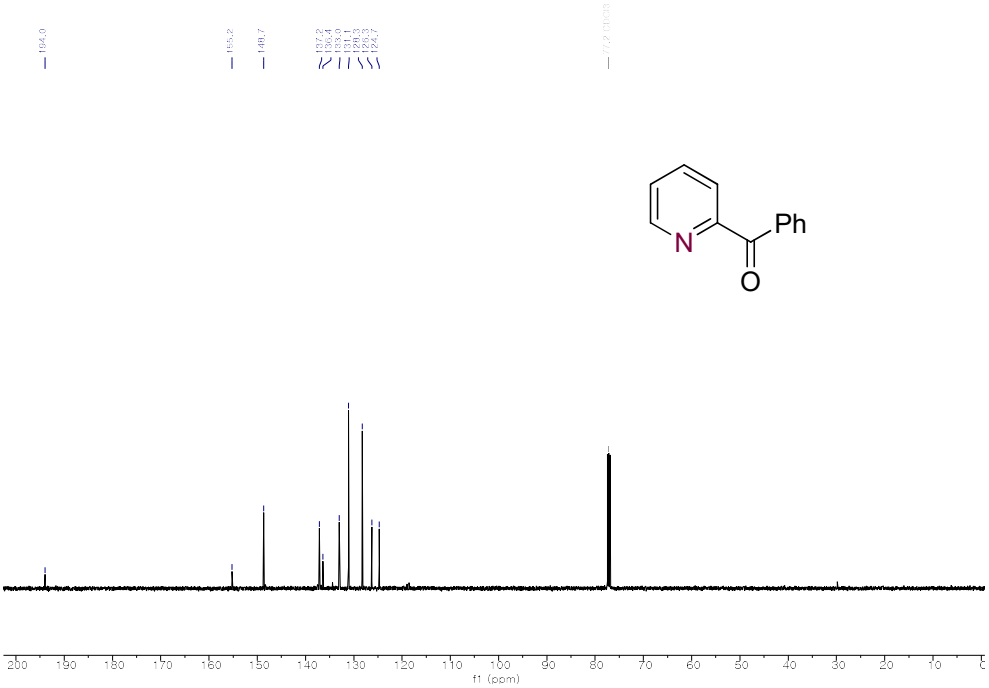


^{1}H & ^{13}C NMR Spectra of 2-Benzoylpyridine (2am)

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

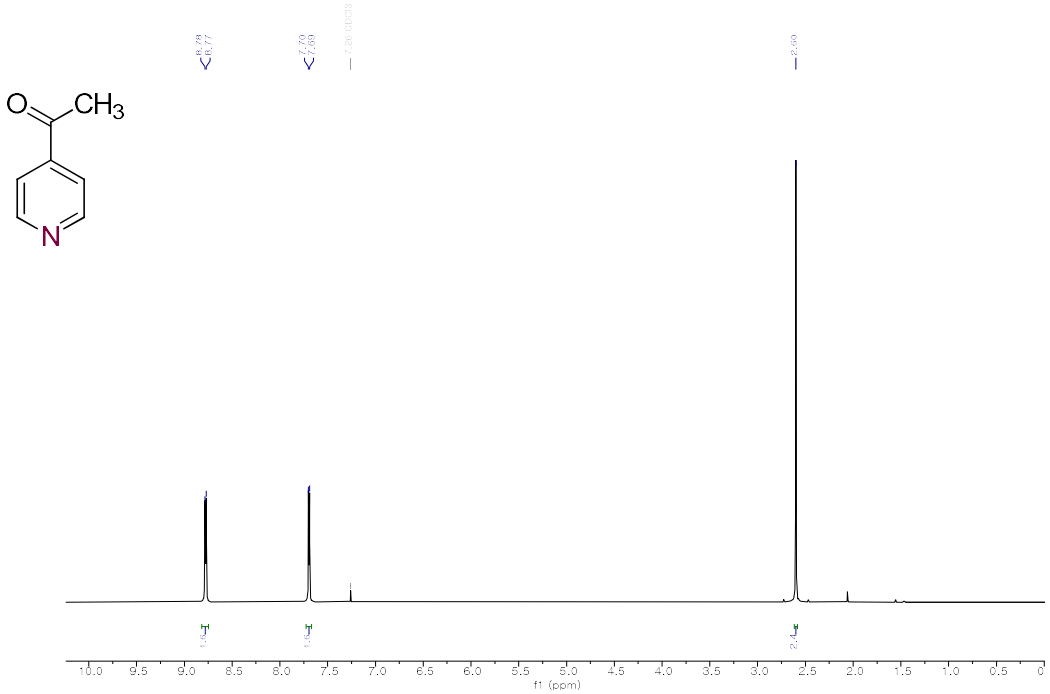


^{13}C NMR (126 MHz, $CDCl_3$)

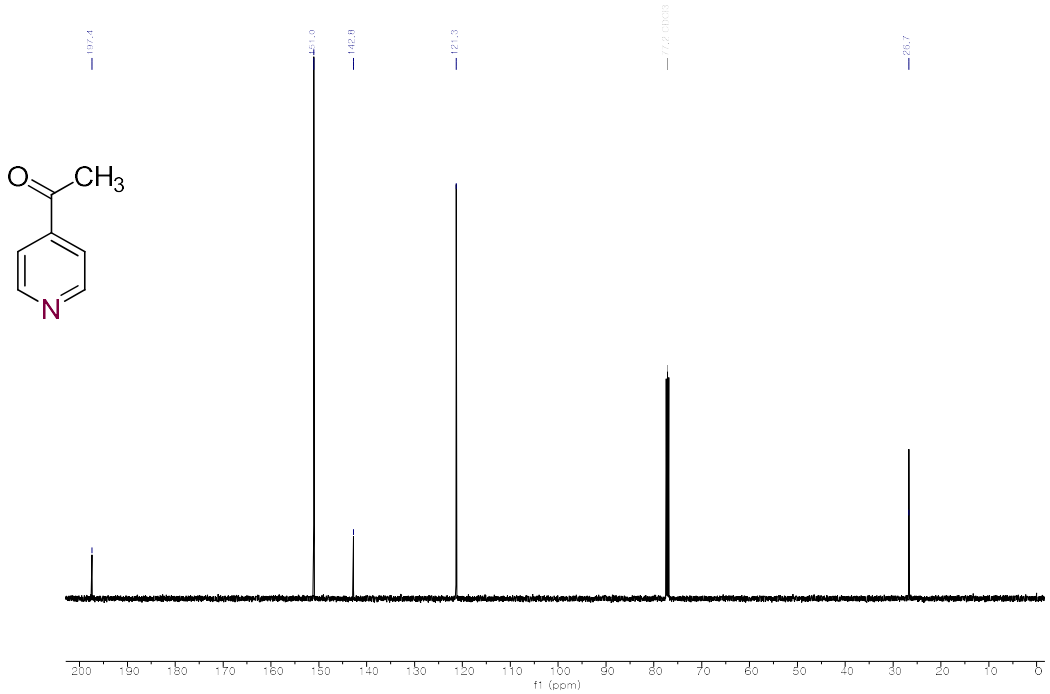


^{1}H & ^{13}C NMR Spectra of 4-Acetylpyridine (2an)

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

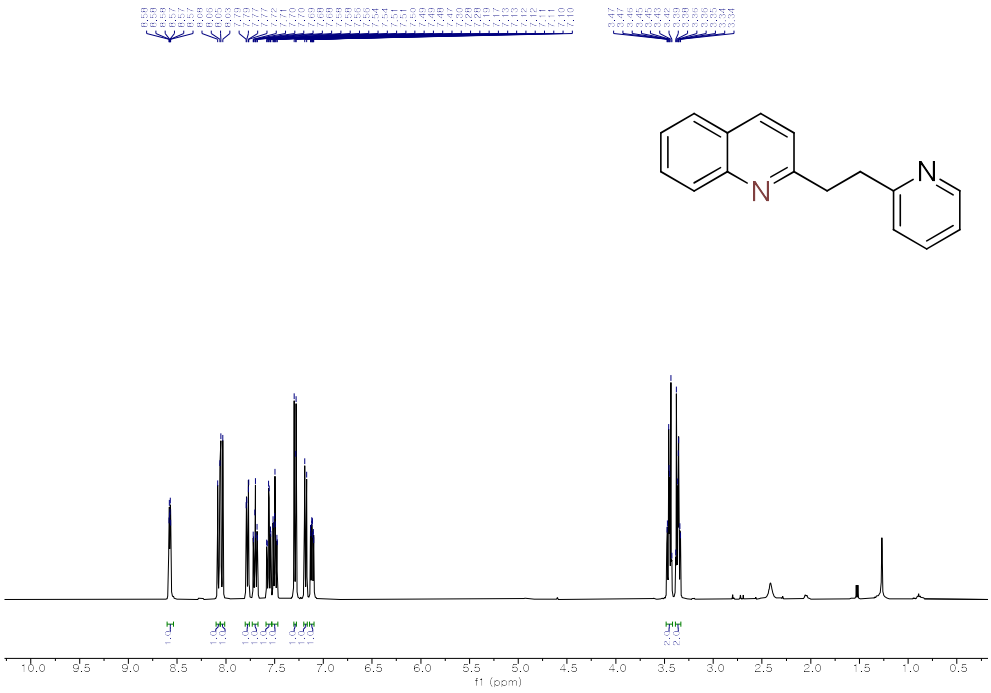


^{13}C NMR (126 MHz, $CDCl_3$)

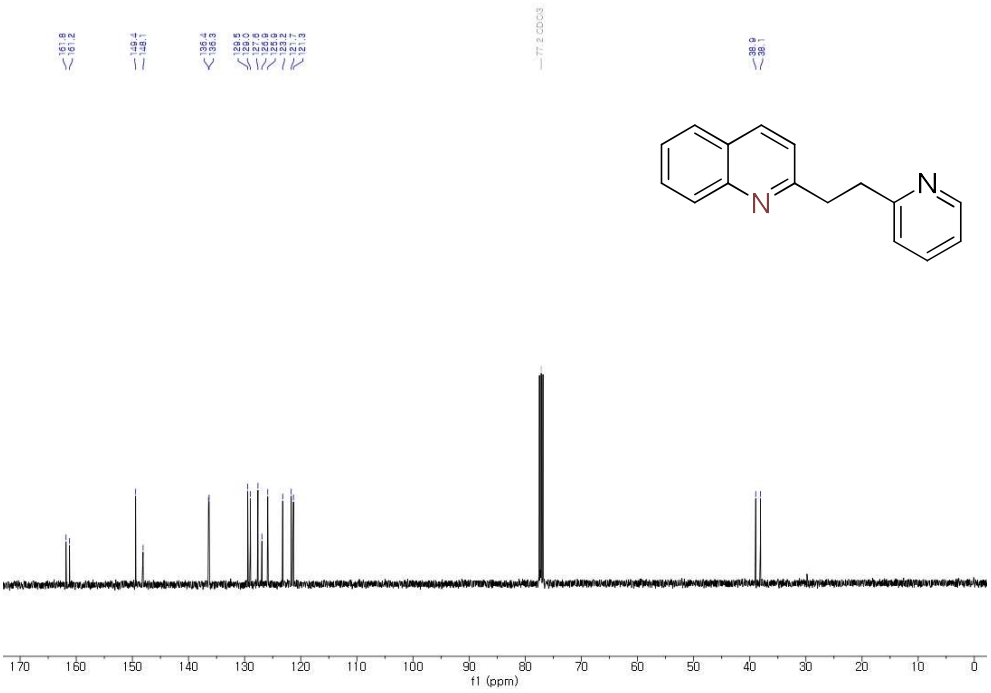


^{1}H & ^{13}C NMR Spectra of 2-{2-(*N*-Oxidopyridine-2-yl)ethyl}quinoline (1ao')

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

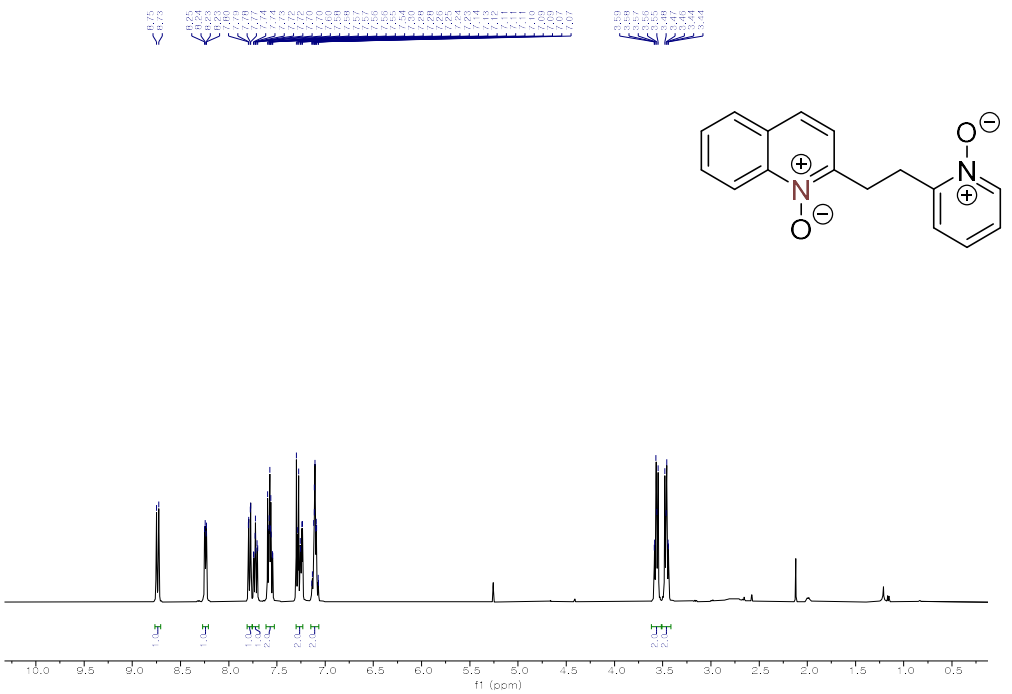


^{13}C NMR (101 MHz, $CDCl_3$)

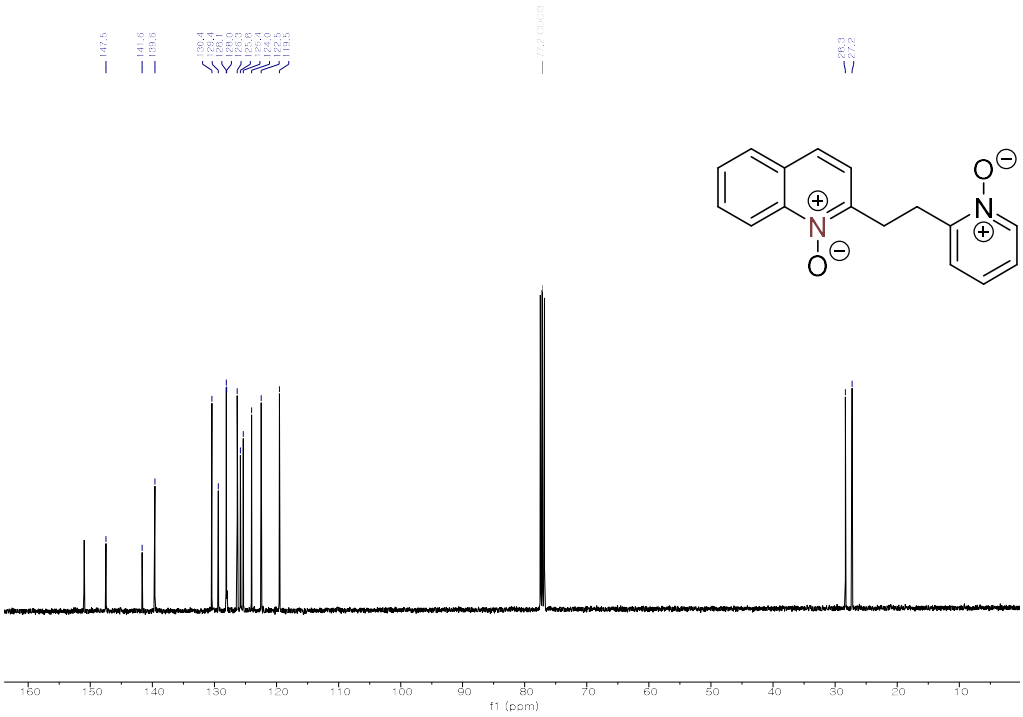


^{1}H & ^{13}C NMR Spectra of 2-{2-(*N*-Oxidopyridine-2-yl)ethyl)}quinoline *N*-oxide (1ao)

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

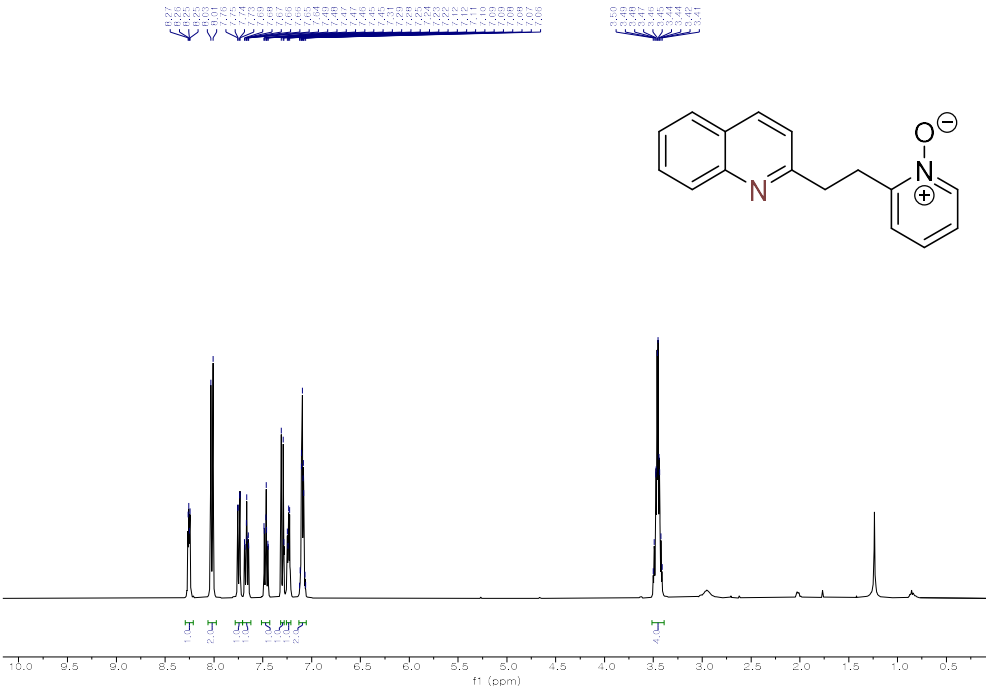


^{13}C NMR (101 MHz, $CDCl_3$)

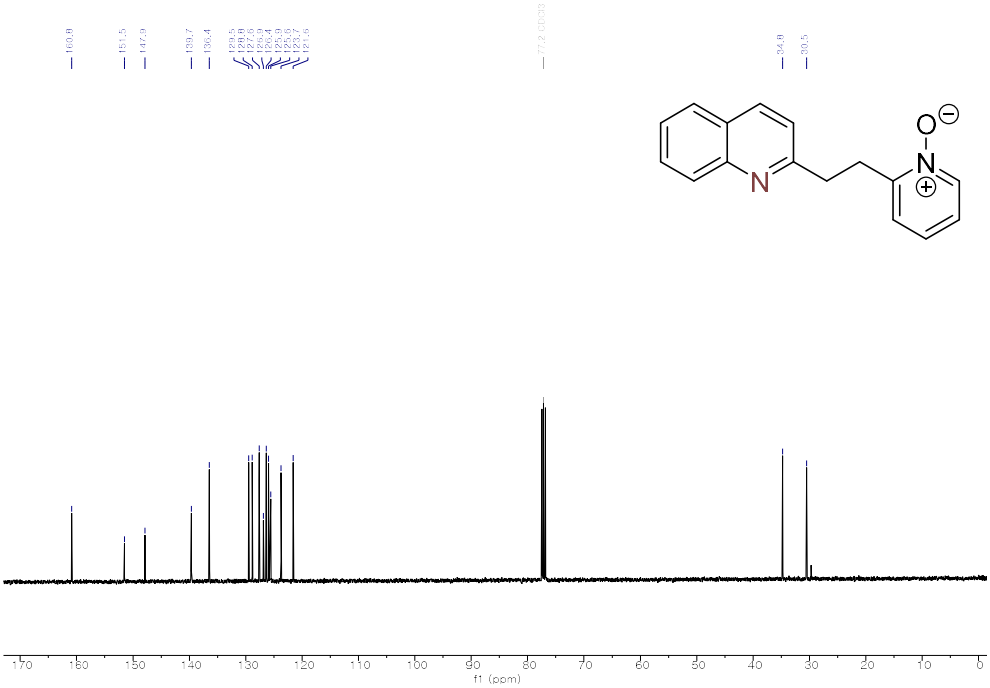


^{1}H & ^{13}C NMR Spectra of 2-{2-(2-Quinolinyl)ethyl}pyridine *N*-oxide (2ao)

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

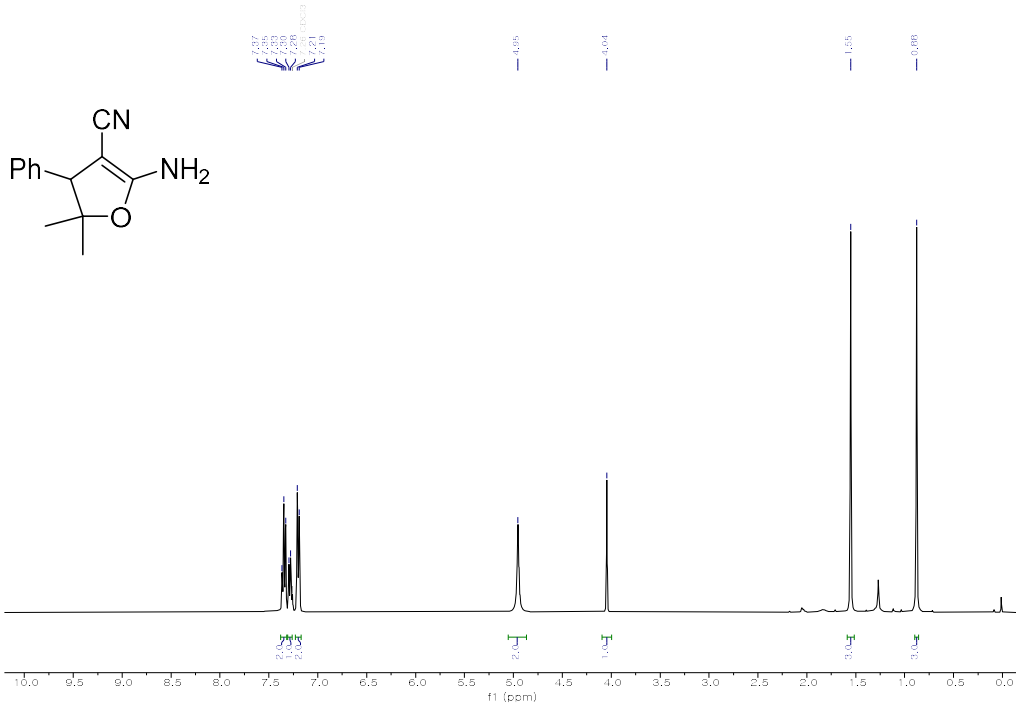


^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H & ^{13}C NMR Spectra of 2-Amino-5,5-dimethyl-4-phenyl-4,5-dihydrofuran-3-carbonitrile (9')

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



^{13}C NMR (101 MHz, $CDCl_3$)