Electronic Supplementary Material (ESI) for Green Chemistry.

Visible-light-induced deoxygenative C2-sulfonylation of quinoline *N*-oxides with sulfinic acids

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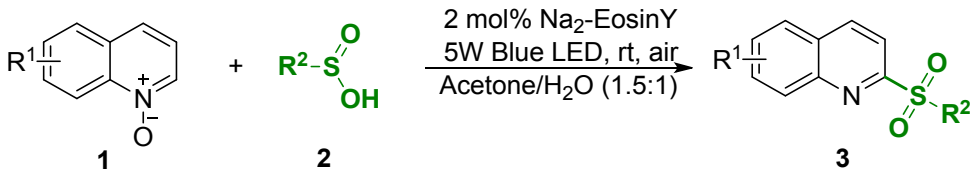
1. General information

Unless otherwise specified, all reagents and solvents were obtained from commercial suppliers and used without further purification. All reagents were weighed and handled in air at room temperature. ^{1}H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra were recorded at 100 MHz by using a Bruker Avance 400 spectrometer. Chemical shifts were calibrated using residual undeuterated solvent as an internal reference (^{1}H NMR: $CDCl_3$ 7.26 ppm, ^{13}C NMR: $CDCl_3$ 77.0 ppm). The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, brs = broad singlet. Mass spectra were performed on a spectrometer operating on ESI-TOF. There is about 3.0 cm distance between the reactor and LEDs.

2. Experimental Section

2.1 General procedure for the synthesis of 2-Sulfonylquinolines



To a solution of sulfinic acid (0.4 mmol), and Na_2-EosinY (0.004 mmol)) in acetone/H_2O (1 mL, v_1/v_2 =1.5/1) was added quinoline *N*-oxide (0.2 mmol). The reaction mixture was open to the air and stirred at room temperature under the irradiation of 5W blue LED lamps for about 12h. After completion of the reaction, the resulting mixture was extracted with EtOAc and the solvent was then removed under vacuum. The residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **3**.

2.2 Preparation of 2-*d*1-Quinoline-*N*-Oxide

D_2O (1.5 mL), NaOH (200 mg, 5 mmol), quinoline-*N*-oxide (258 mg, 2.0 mmol) were weighed into 30-mL pressure tube sealed with rubber plugs. The reaction mixture was stirred at 100 ℃ for overnight. After cooling to room temperature, the mixture was then extracted with EtOAc (3 x 10 mL). The combined organic phase was washed with saturated NaCl solution (3 x 5 mL), dried over $MgSO_4$, and filtered. EtOAc was removed under reduced pressure to obtain the crude product 2-d_1-quinoline-*N*-Oxide. It was further purified by flash column chromatography and percentage of *d* - incorporation was determined by ^{1}H NMR. Peak areas at 8.74 ppm and 8.53 ppm were compared to obtain the deuterium incorporation. Deuterium incorporation was detected to be 92% by ^{1}H NMR (see ^{1}H spectrum, Figure S1).

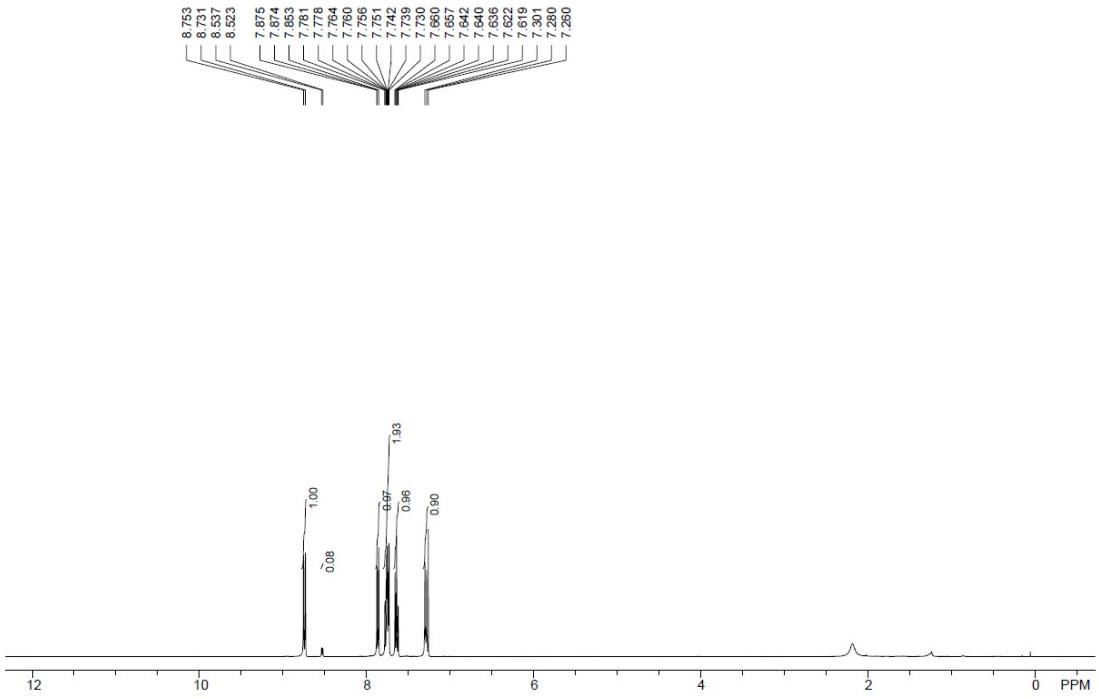
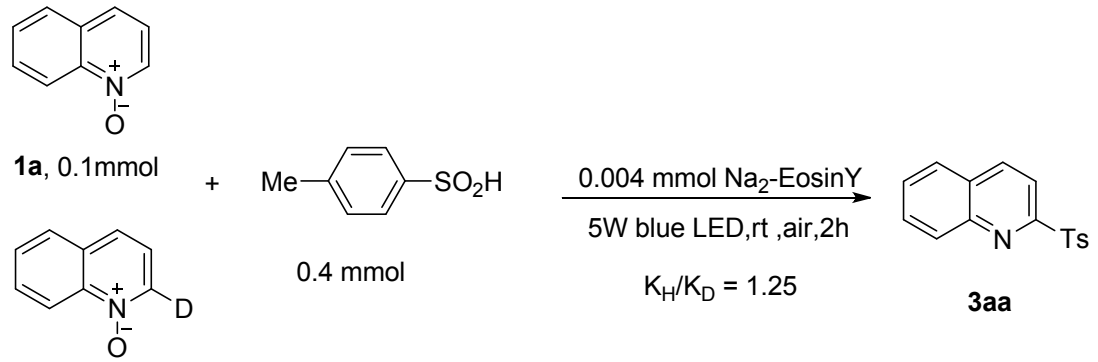


Figure S1

2.3 KIE Experiment



To a solution of sulfinic acid (0.4 mmol), and Na_2-EosinY (0.004 mmol)) in acetone/H_2O (1 mL, v_1/v_2 =1.5/1) was added Quinoline *N*-oxide **1a** and 2-d_1-quinoline *N*-oxide **[D_1]-1a** (1:1，totally 0.2 mmol, deuteration ratio has been calculated). The reaction mixture was open to the air and stirred at room temperature under the irradiation of 5W blue LED lamps for 2h, then the residual starting material (mixture of 2-d_1-quinoline-*N*-oxide and quinoline -*N*-oxide) was recovered by column chromatography on silica gel (200-300 mesh), which was characterized by ^{1}H NMR spectroscopy (Figure S2). Peak areas at 8.74 ppm and 8.52 ppm were compared to give the ratio (0.56:0.44) of 2-d_1-quinoline N-oxide to quinoline N-oxide in residual material. k_H/k_D was calculated using the following expression[1]:

$$K_H/K_D = \frac{M / 2 - 0.44m}{M / 2 - 0.56m}$$

M, m represent the amount of 2-d_1-quinoline N-oxide and quinoline *N*-oxide in reaction staring material and residual material, respectively. Here, M = 29, m = 14, which corresponds to k_H/k_D = 1.25.

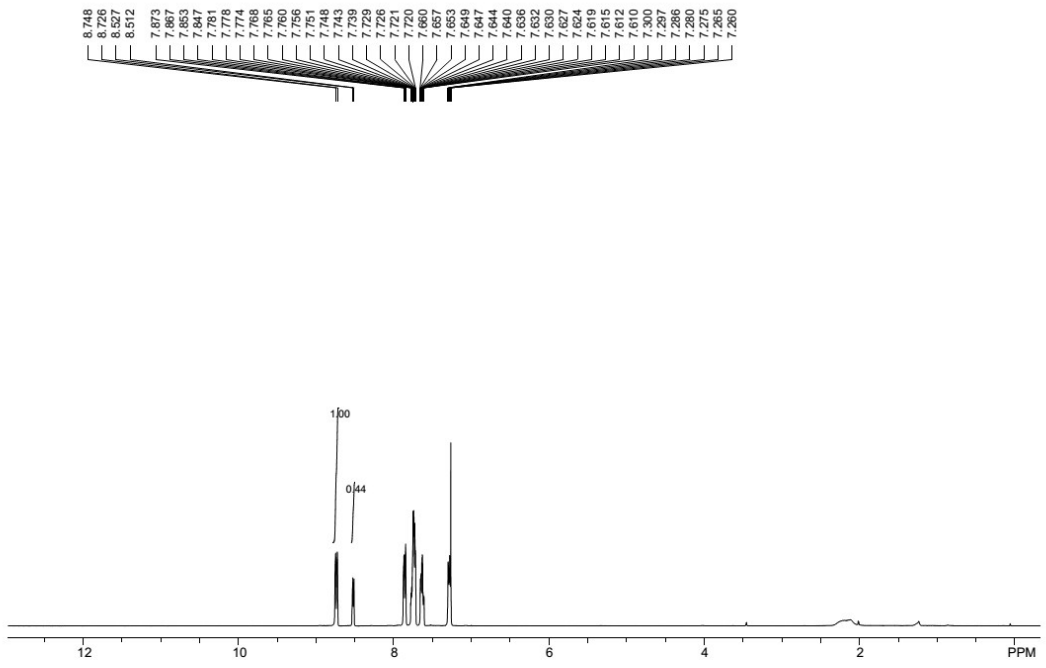
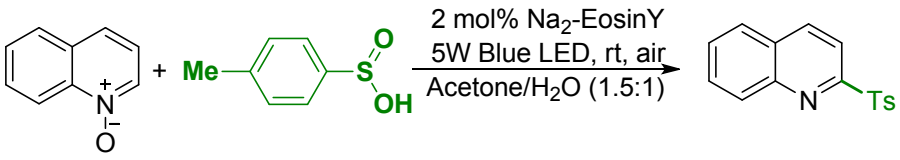


Figure S2

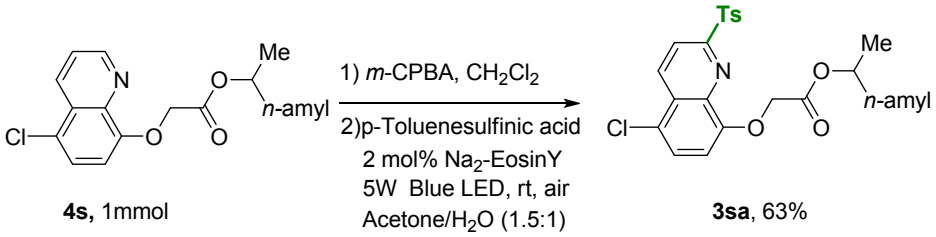
2.4 Gram-scale synthesis of 3aa



1a, 5 mmol, 0.73 g **3aa**, 1.12 g, yield 79%

To a solution of 4-methylbenzenesulfinic acid (1.56 g, 10 mmol), and Na_2-EosinY (69.1 mg, 0.1 mmol)) in acetone/H_2O (25 mL, v_1/v_2 =1.5/1) was added quinoline *N*-oxide (0.73 g , 5 mmol). The reaction mixture was open to the air and stirred at room temperature under the irradiation of 5W blue LED lamps for about 12h. After completion of the reaction, the resulting mixture was extracted with EtOAc (15 mL× 3) and the solvent was then removed under vacuum. The residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give 1.12 gram of **3aa,** yield 79%.

2.5 Synthesis of sulfonated cloquintocet-mexyl (3sa)



4s, 1mmol

3sa, 63%

A solution of cloquintocet-mexyl **4s** (0.34 g, 1.0 mmol) in DCM (10 mL) was stirred at 0 °C for 5 min. Then m-CPBA (3-chloroperbenzoic acid, 1.5 mmol） was added to the solution through several times. The mixture was stirred at 25 °C for 12 h and a saturated aqueous $NaHCO_3$ solution (10 mL) was added. The resulting solution was extracted with DCM (10 mL × 2). Then it was dried by Na_2SO_4 and concentrated under reduced pressure to obtain the crude product cloquintocet-mexyl *N*-oxide and used without further purification.

The above-synthesized crude product cloquintocet-mexyl *N*-oxide was added to a solution of 4-methylbenzenesulfinic acid (0.31 g, 2 mmol), and Na_2-EosinY (13.8 mg, 0.02 mmol)) in acetone/H_2O (5 mL, v_1/v_2 =1.5/1). The reaction mixture was open to the air and stirred at room temperature under the irradiation of 5W blue LED lamps for about 12 h. After completion of the reaction, the resulting mixture was extracted with EtOAc (10 mL× 3) and the solvent was then removed under vacuum. The residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give 0.31 gram of **3sa,** yield 63%.

2.6 Cyclic voltammetry measurement

CV measurements were performed on a CHI-660B workstation (Shanghai Chenhua Instruments Co., China) with the three-electrode system using a glassy carbon working electrode, a platinum wire counter electrode, an Ag/AgCl as a reference electrode and TBATFB 0.1 M as supporting electrolyte. The potentials were achieved relative to the Fc/Fc+ redox couple with ferrocene as internal standard. The measurements were carried out as follows: a 0.1 M solution of TBATFB in acetone/H_2O (1.5:1) was added to the measuring cell and the solution was degassed by argon purge for 5 min. After recording the baseline the electroactive compound was added (0.01 M) and the solution was again degassed a stream of argon for 5 min. The cyclic voltammogram was recorded with one to three scans. Afterwards ferrocene (2.20 mg, 12.0 μmol) was added to the solution which was again degassed by argon purge for 5 min and the final measurement was performed with three scans.

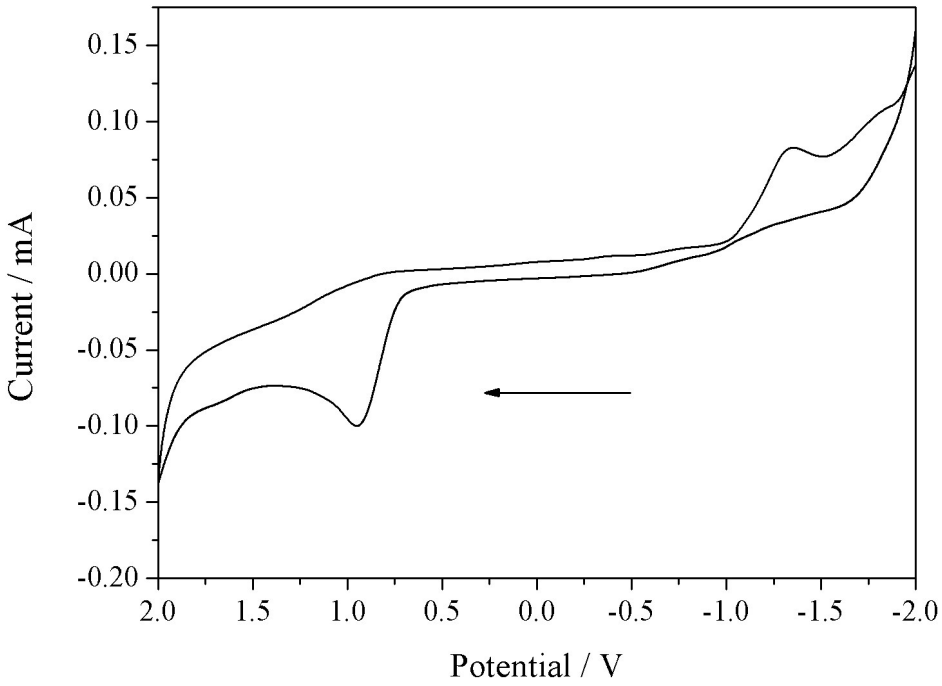


Figure S3. Cyclic voltammogram of 4-methylbenzenesulfinic acid (2a) in acetone/H_2O (1.5:1) under argon (scan direction indicated by black arrow). The irreversible peak at -1.35 V is the reduction of **2a**.

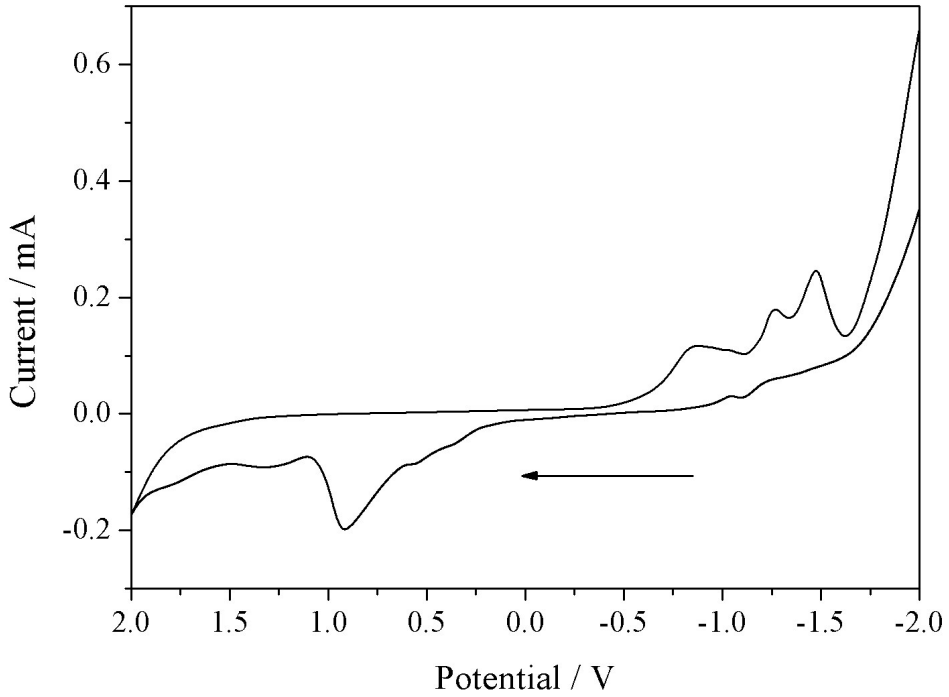
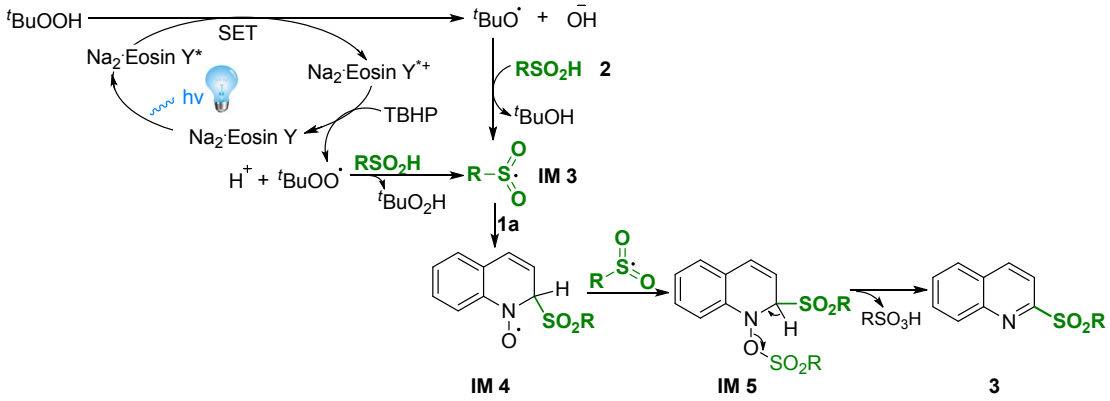


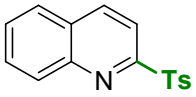
Figure S4. Cyclic voltammogram of Na_2·eosin Y in acetone/H_2O (1.5:1) under argon (scan direction indicated by black arrow). The irreversible peak at +0.91 V is the oxidation of Na_2·eosin Y.

2.7 Possible mechanism with TBHP as the oxidant



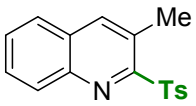
3. Characterization data of products

2-tosylquinoline **(3aa)**[2]



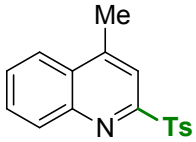
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (d, *J* = 8.8 Hz, 1 H), 8.20 – 8.16 (m, 2 H), 8.03 – 8.00 (m, 2 H), 7.86 (d, *J* = 8.4 Hz, 1 H), 7.79 – 7.75 (m, 1 H), 7.66 – 7.63 (m, 1 H), 7.32 (d, *J* = 8.0 Hz, 2 H), 2.39 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.3, 147.4, 144.8, 138.7, 136.1, 130.9, 130.3, 129.7, 129.1, 129.0, 128.7, 127.6, 117.6, 21.6.

3-methyl-2-tosylquinoline **(3ba)**[3]



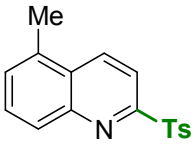
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.03 (s, 1 H), 7.94 – 7.90 (m, 3 H), 7.74 – 7.72 (m, 1 H), 7.65 – 7.60 (m, 1 H), 7.58 – 7.54 (m, 1 H), 7.34 (d, *J* = 8.0 Hz, 2 H), 2.84 (s, 3 H), 2.44 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 156.9, 144.6, 144.4, 139.8, 135.7, 129.8, 129.6, 129.3, 129.3, 129.0, 128.9, 128.4, 126.6, 21.6, 18.8.

4-methyl-2-tosylquinoline **(3ca)**[4]



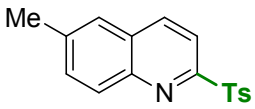
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.15 (dd, J_1 = 8.8 Hz, J_2 = 0.8 Hz,1 H), 8.02 – 7.98 (m, 4 H), 7.76 – 7.72 (m, 1 H), 7.66 – 7.62 (m, 1 H), 7.31 (d, *J* = 7.6 Hz, 2 H), 2.77 (s, 3 H), 2.38 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.9, 147.8, 147.2, 144.6, 136.2, 131.0, 130.4, 129.7, 128.9, 128.8, 128.7, 123.7, 118.0, 21.6, 19.1.

5-methyl-2-tosylquinoline **(3da)**[5]



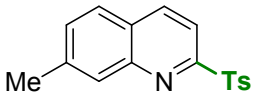
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.51 (dd, J_1 = 8.8 Hz, J_2 = 0.8 Hz, 1 H), 8.20 (d, *J* = 8.4 Hz, 1 H), 8.01 (d, *J* = 8.0 Hz, 2 H), 8.02 – 8.00 (m, 3 H), 7.66 – 7.62 (m, 1 H), 7.49 – 7.44 (m, 1 H), 7.31 (d, *J* = 8.4 Hz, 2 H), 2.68 (s, 3 H), 2.39 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.8, 147.8, 144.7, 136.2, 135.1, 134.7, 130.6, 129.7, 129.4, 129.0, 128.6, 128.2, 117.2, 21.6, 18.6.

6-methyl-2-tosylquinoline **(3ea)**[6]



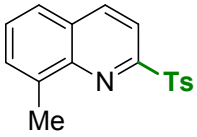
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.25 (d, *J* = 8.4 Hz, 1 H), 8.14 (d, *J* = 8.4 Hz, 1 H), 8.06 (d, *J* = 8.4 Hz, 1 H), 8.01 (d, *J* = 8.0 Hz, 2 H), 7.62 – 7.59 (m, 2 H), 7.32 (d, *J* = 8.0 Hz, 2 H), 2.54 (s, 3 H), 2.39 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.4, 146.1, 144.6, 139.6, 137.7, 136.3, 133.3, 130.0, 129.7, 129.0, 128.9, 126.4, 117.7, 21.8, 21.6.

7-methyl-2-tosylquinoline **(3fa)**[7]



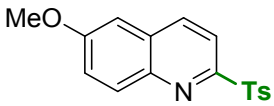
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.30 (d, *J* = 8.8 Hz, 1 H), 8.12 (d, *J* = 8.8 Hz, 1 H), 8.01 (d, *J* = 8.4 Hz, 2 H), 7.95 (s, 1 H), 7.75 (d, *J* = 8.4 Hz, 1 H), 7.47 (d, *J* = 8.0 Hz, 1 H), 7.32 (d, *J* = 8.0 Hz, 2 H), 2.54 (s, 3 H), 2.39 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.2, 147.7, 144.7, 141.6, 138.2, 136.2, 131.5, 129.7, 129.2, 129.0, 127.2, 126.9, 116.8, 21.8, 21.6.

8-methyl-2-tosylquinoline **(3ga)**[2]



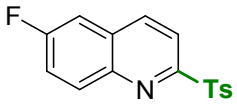
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.30 (d, *J* = 8.8 Hz, 1 H), 8.17 (d, *J* = 8.4 Hz, 1 H), 8.06 – 8.04 (m, 2 H), 7.65 (d, *J* = 8.0 Hz, 1 H), 7.56 – 7.54 (m, 1 H), 7.50 – 7.46 (m, 1 H), 7.32 (d, *J* = 8.0 Hz, 2 H), 2.66 (s, 3 H), 2.39 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.1, 146.2, 144.6, 138.6, 135.8, 130.8, 129.4, 129.2, 128.8, 128.7, 128.7, 125.4, 116.6, 21.5, 17.4.

6-methoxy-2-tosylquinoline **(3ha)**[3]



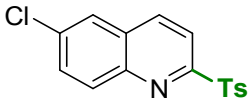
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.21 (d, *J* = 8.4 Hz, 1 H), 8.14 (d, *J* = 8.4 Hz, 1 H), 8.05 (d, *J* = 9.2 Hz, 1 H), 8.00 (d, *J* = 8.4 Hz, 2 H), 7.43 – 7.40 (m, 1 H), 7.32 (d, *J* = 8.0 Hz, 2 H), 7.08 (d, *J* = 2.8 Hz, 1 H), 3.94 (s, 3 H), 2.39 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.8, 155.7, 144.6, 143.6, 136.8, 136.5, 131.8, 130.4, 129.7, 128.9, 124.2, 118.2, 104.5, 55.7, 21.6.

6-fluoro-2-tosylquinoline **(3ia)**[3]



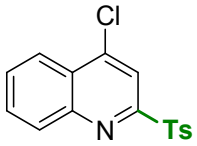
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.30 (d, *J* = 8.8 Hz, 1 H), 8.19 – 8.12 (m, 2 H), 7.99 (d, *J* = 8.0 Hz, 2 H), 7.54 – 7.44 (m, 2 H), 7.31 (d, *J* = 8.0 Hz, 2 H), 2.37 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 161.7 ($J_{C\text{-}F}$ = 251.6 Hz), 157.7 ($J_{C\text{-}F}$ = 3.6 Hz), 144.8, 144.3, 138.0 ($J_{C\text{-}F}$ = 5.8 Hz), 135.8, 132.9 ($J_{C\text{-}F}$ = 9.5 Hz), 129.7, 129.6, 128.9, 121.5 ($J_{C\text{-}F}$ = 26.3 Hz), 118.3, 110.7 ($J_{C\text{-}F}$ = 21.9 Hz), 21.5; ^{19}F NMR (376 MHz, $CDCl_3$): δ = - 108.3.

6-chloro-2-tosylquinoline **(3ja)**[2]



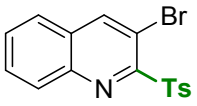
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.28 (d, *J* = 8.4 Hz, 1 H), 8.20 (d, *J* = 8.4 Hz, 1 H), 8.09 (d, *J* = 9.2 Hz, 1 H), 8.00 (d, *J* = 8.4 Hz, 2 H), 7.85 (d, *J* = 3.0 Hz, 1 H), 7.70 (dd, J_1 = 9.2 Hz, J_2 = 2.0 Hz, 1 H), 7.33 (d, *J* = 8.0 Hz, 2 H), 2.40 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.6, 145.7, 145.0, 137.7, 135.8, 135.2, 132.0, 131.9, 129.8, 129.3, 129.1, 126.3, 118.6, 21.6.

4-chloro-2-tosylquinoline **(3ka)**[8]



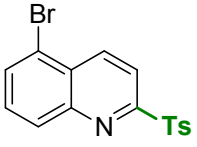
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.28 (s, 1 H), 8.26 (dd, J_1 = 8.4 Hz, J_2 = 0.8 Hz, 1 H), 8.20 – 8.18 (m, 1 H), 8.03 – 8.00 (m, 2 H), 7.86 – 7.81 (m, 1 H), 7.77 – 7.73 (m, 1 H), 7.34 (d, *J* = 8.0 Hz, 2 H), 2.41 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.2, 148.1, 145.2, 145.2, 135.6, 131.7, 130.8, 130.1, 129.9, 129.2, 127.0, 124.2, 117.9, 21.7.

3-bromo-2-tosylquinoline **(3la)**[4]



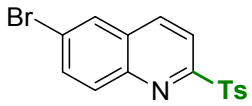
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.51 (s, 1 H), 7.99 – 7.95 (m, 3 H), 7.78 – 7.73 (m, 2 H), 7.67 (d, *J* = 8.0 Hz, 1 H), 7.36 (d, *J* = 8.4 Hz, 2 H), 2.46 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 154.4, 144.9, 144.4, 142.9, 134.9, 131.0, 130.2, 130.0, 129.8, 129.7, 129.4, 126.5, 111.4, 21.7.

5-bromo-2-tosylquinoline **(3ma)**[9]



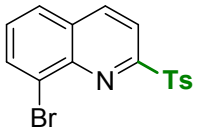
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.75 (dd, J_1 = 8.8 Hz, J_2 = 0.4 Hz, 1 H), 8.28 (d, J= 8.8 Hz, 1 H), 8.15 – 8.13 (m, 1 H), 8.02 – 8.00 (m, 2 H), 7.93 – 7.91 (m, 1 H), 7.65 – 7.61 (m, 1 H), 7.34 (d, J = 8.0 Hz, 2 H), 2.41 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.2, 148.1, 145.0, 138.5, 135.7, 132.7, 131.1, 130.3, 129.8, 129.1, 128.7, 121.8, 118.8, 21.7.

6-bromo-2-tosylquinoline **(3na)**[3]



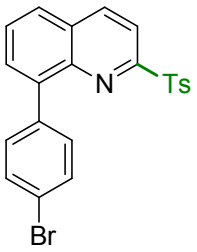
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.27 (d, J = 8.4 Hz, 1 H), 8.21 (d, J = 8.4 Hz, 1 H), 8.04 – 7.99 (m, 4 H), 7.83 (dd, J_1 = 9.2 Hz, J_2 = 2.0 Hz, 1 H), 7.34 (d, J = 8.0 Hz, 2 H), 2.41 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.7, 145.9, 145.0, 137.6, 135.7, 134.5, 131.9, 129.8, 129.7, 129.1, 123.5, 118.6, 21.7.

8-bromo-2-tosylquinoline **(3oa)**[9]



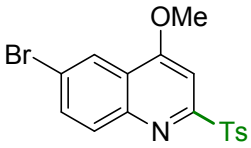
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (d, J= 8.8 Hz, 1 H), 8.25 (d, J= 8.4 Hz, 1 H), 8.13 (d, J= 8.4 Hz, 2 H), 8.07 (d, J= 7.6 Hz, 1 H), 7.82 (d, J= 8.4 Hz, 1 H), 7.47 (t, J= 8.0 Hz, 1 H), 7.36 (d, J= 8.0 Hz, 2 H), 2.42 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.4, 145.0, 144.4, 139.2, 135.2, 134.5, 129.9, 129.8, 129.5, 129.3, 127.4, 125.8, 117.7, 21.7.

8-(4-bromophenyl)-2-tosylquinoline **(3pa)**



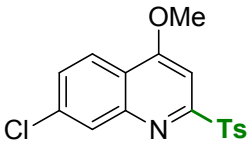
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.42 (d, J = 8.4 Hz, 1 H), 8.24 (d, J = 8.4 Hz, 1 H), 7.87 – 7.85 (m, 3 H), 7.75 (dd, J_1 = 7.2 Hz, J_2 = 1.2 Hz, 1 H), 7.67 (t, J = 7.6 Hz, 1 H), 7.47 – 7.45 (m, 2 H), 7.29 – 7.27 (m, 4 H), 2.47 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.5, 144.8, 144.2, 139.9, 139.0, 136.8, 134.8, 132.2, 131.1, 130.7, 129.6, 129.4, 129.2, 128.8, 127.6, 121.6, 116.6, 21.7； IR (in KBr): ν = 3416, 1578, 1489, 1298, 1135, 1010, 968, 821, 681(cm^{-1})； HRMS (ESI) m/z calcd. for $C_{22}H_{17}BrNO_2S[M+H]^+$: 438.0158, found 438.0163.

6-bromo-4-methoxy-2-tosylquinoline **(3qa)**



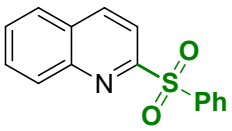
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.36 (d, *J* = 2.0 Hz, 1 H), 8.00 (d, *J* = 8.0 Hz, 2 H), 7.93 (d, *J* = 9.2 Hz, 1 H), 7.80 – 7.77 (m, 1 H), 7.59 (s, 1 H), 7.34 (d, *J* = 8.0 Hz, 2 H), 4.15 (s, 3 H), 2.42 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 163.4, 160.0, 146.9, 144.9, 135.7, 134.6, 131.5, 129.8, 129.1, 124.6, 122.7, 122.4, 97.6, 56.7, 21.7; IR (in KBr): ν = 3419, 2930, 1571, 1418, 1327, 819, 693 (cm^{-1}); HRMS (ESI) m/z calcd. for $C_{17}H_{15}BrNO_3S[M+H]^+$: 391.9951, found 391.9948.

7-chloro-4-methoxy-2-tosylquinoline **(3ra)**



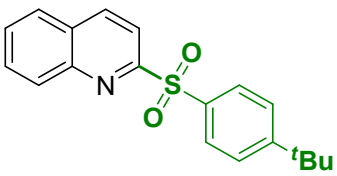
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.13 (d, *J* = 9.2 Hz, 1 H), 8.06 (d, *J* = 2.0 Hz, 1 H), 8.00 (d, *J* = 8.8 Hz, 2 H), 7.58 (s, 1 H), 7.52 (dd, J_1 = 8.8 Hz, J_2 = 2.0 Hz, 1 H), 7.34 (d, *J* = 8.0 Hz, 1 H), 4.15 (s, 3 H), 2.42 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 164.4, 160.9, 148.7, 144.9, 137.2, 135.6, 129.7, 129.2, 128.9, 128.7, 123.4, 120.0, 97.1, 56.7, 21.7; IR (in KBr): ν = 2924, 1517, 1415, 1331, 1154, 1116, 819, 694 (cm^{-1}); HRMS (ESI) m/z calcd. for $C_{17}H_{15}ClNO_3S[M+H]^+$: 348.0456, found 348.0452.

2-(phenylsulfonyl)quinoline **(3ab)**[4]



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.39 (d, *J* = 8.8 Hz, 1 H), 8.23 – 8.14 (m, 4 H), 7.88 (d, *J* = 8.4 Hz, 1 H), 7.81 – 7.77 (m, 1 H), 7.68 – 7.65 (m, 1 H), 7.61 – 7.59 (m, 1 H), 7.56 – 7.52 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.1, 147.4, 139.1, 138.7, 133.7, 131.0, 130.4, 129.2, 129.1, 129.0, 128.8, 127.7, 117.7.

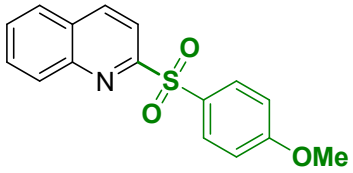
2-((4-(tert-butyl)phenyl)sulfonyl)quinoline **(3ac)**[4]



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.37 (d, *J* = 8.8 Hz, 1 H), 8.20 (d, *J* = 8.8 Hz, 2 H), 8.06 (d, *J* = 8.8 Hz, 2 H), 7.87 (d, *J* = 8.4 Hz, 1 H), 7.81 – 7.70 (m, 1 H), 7.68 – 7.64 (m, 1 H), 7.54 (d, *J* = 8.8 Hz, 2 H), 1.31 (s, 9 H); ^{13}C

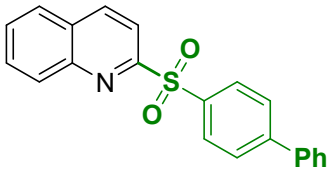
NMR (100 MHz, $CDCl_3$): δ = 158.4, 157.7, 147.5, 138.6, 136.1, 130.9, 130.5, 129.1, 128.9, 128.8, 127.7, 126.2, 117.8, 35.2, 31.0.

2-((4-methoxyphenyl)sulfonyl)quinoline **(3ad)**[6]



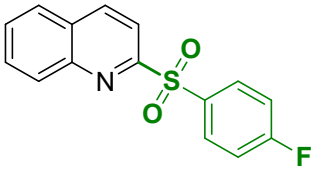
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.35 (d, *J* = 8.0 Hz, 1 H), 8.19 – 8.15 (m, 2 H), 8.07 (d, *J* = 8.8 Hz, 2 H), 7.86 (d, *J* = 8.0 Hz, 1 H), 7.77 (t, *J* = 7.6 Hz, 1 H), 7.64 (t, *J* = 7.6 Hz, 1 H), 6.99 (d, *J* = 8.8 Hz, 2 H), 3.84 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 163.9, 158.6, 147.4, 138.6, 131.2, 130.9, 130.5, 130.3, 129.0, 128.7, 127.6, 117.5, 114.3, 55.6.

2-([1,1'-biphenyl]-4-ylsulfonyl)quinolone **(3ae)**[3]



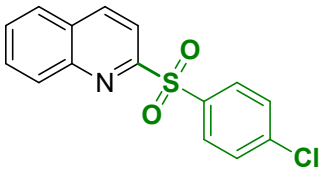
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.40 (d, *J* = 8.8 Hz, 1 H), 8.25 (d, *J* = 8.4 Hz, 1 H), 8.23 – 8.19 (m, 3 H), 7.89 (d, *J* = 8.4 Hz, 1 H), 7.82 – 7.78 (m, 1 H), 7.75 – 7.73 (m, 2 H), 7.69 – 7.65 (m, 1 H), 7.58 – 7.56 (m, 2 H), 7.47 – 7.38 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.2, 147.5, 146.7, 139.2, 138.8, 137.6, 131.0, 130.4, 129.6, 129.2, 129.0, 128.9, 128.6, 127.7, 127.7, 127.4, 117.7.

2-((4-fluorophenyl)sulfonyl)quinoline **(3af)**[3]



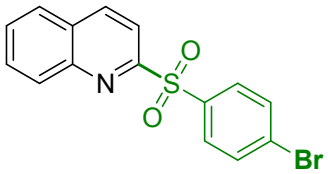
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.46 (d, *J* = 8.8 Hz, 1 H), 8.28 – 8.20 (m, 4 H), 7.95 (dd, J_1 = 8.4 Hz, J_2 = 0.8 Hz, 1 H), 7.88 - 7.84 (m, 1 H), 7.75 – 7.71 (m, 1 H), 7.33 – 7.26 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 165.9 (J_{C-F} = 254.5 Hz), 157.9, 147.4, 138.8, 134.9, 132.0 (J_{C-F} = 9.5 Hz), 131.1, 130.3, 129.3, 128.8, 127.7, 117.4, 116.4 (J_{C-F} = 22.6 Hz); ^{19}F NMR (376 MHz, $CDCl_3$): δ = - 103.4.

2-((4-chlorophenyl)sulfonyl)quinoline **(3ag)**[10]



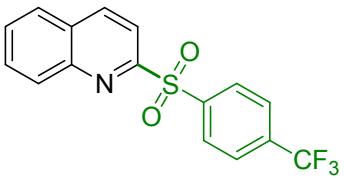
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.40 (d, J = 8.4 Hz, 1 H), 8.20 (d, J = 8.4 Hz, 1 H), 8.15 (dd, J_1 = 8.8 Hz, J_2 = 0.8 Hz 1 H), 8.10 – 8.06 (m, 2 H), 7.89 (dd, J_1 = 8.4 Hz, J_2 = 1.2 Hz 1 H), 7.82 – 7.78 (m, 1 H), 7.69 – 7.65 (m, 1 H), 7.52 – 7.49 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.7, 147.4, 140.5, 138.9, 137.4, 131.1, 130.5, 130.3, 129.4, 129.3, 128.9, 127.7, 117.5.

2-((4-bromophenyl)sulfonyl)quinoline **(3ah)**[6]



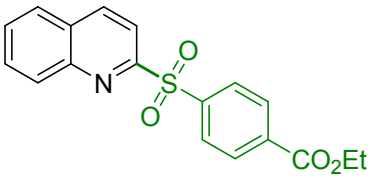
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.40 (d, J = 8.8 Hz, 1 H), 8.20 (d, J = 8.4 Hz, 1 H), 8.15 (d, J = 8.4 Hz, 1 H), 8.00 (d, J = 8.8 Hz, 2 H), 7.89 (d, J = 8.0 Hz, 1 H), 7.80 (t, J = 8.0 Hz, 1 H), 7.69 – 7.67 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.7, 147.4, 138.9, 138.0, 132.4, 131.1, 130.6, 130.3, 129.4, 129.2, 128.9, 127.7, 117.5.

2-((4-(trifluoromethyl)phenyl)sulfonyl)quinoline **(3ai)**[3]



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.42 (d, J = 8.4 Hz, 1 H), 8.29 (d, J = 8.4 Hz, 2 H), 8.23 (d, J = 8.8 Hz, 1 H), 8.14 (d, J = 8.4 Hz, 1 H), 7.90 (d, J = 8.0 Hz, 1 H), 7.82 – 7.78 (m, 3 H), 7.70 – 7.66 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.3, 147.4, 142.6, 139.0, 135.2 ($J_{C\text{-}F}$ = 32.8), 131.2, 130.3, 129.7, 129.5, 128.9, 127.7, 126.1 ($J_{C\text{-}F}$ = 3.7), 123.1 ($J_{C\text{-}F}$ = 271.2), 117.5; ^{19}F NMR (376 MHz, $CDCl_3$): δ = - 63.2.

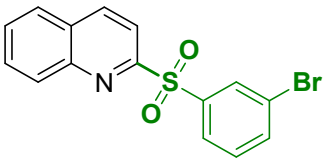
ethyl 4-(quinolin-2-ylsulfonyl)benzoate **(3aj)**[5]



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.41 (d, J = 8.4 Hz, 1 H), 8.24 – 8.18 (m, 5 H), 8.14 (d, J = 8.4 Hz, 1 H), 7.89 (d, J = 8.0 Hz, 1 H), 7.82 – 7.78 (m, 1 H), 7.69 – 7.66 (m, 1 H), 4.39 (q, J = 7.2 Hz, 2 H), 1.39 (t, J = 7.2 Hz, 3 H); ^{13}C

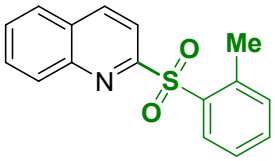
NMR (100 MHz, $CDCl_3$): δ = 165.1, 157.6, 147.5, 142.8, 138.9, 135.1, 131.2, 130.4, 130.1, 129.4, 129.1, 128.9, 127.7, 117.6, 61.7, 14.2.

***2-((3-bromophenyl)sulfonyl)quinoline* (3ak)[11]**



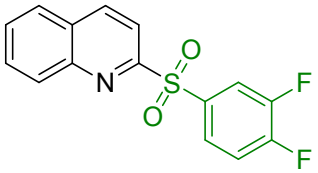
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.41 (d, *J* = 8.4 Hz, 1 H), 8.28 (t, *J* = 2.0 Hz, 1 H), 8.22 (d, *J* = 8.8 Hz, 1 H), 8.18 (d, *J* = 8.4 Hz, 1 H), 8.10 – 8.07 (m, 1 H), 7.90 (d, *J* = 8.4 Hz, 1 H), 7.83 – 7.79 (m, 1 H), 7.74 – 7.67 (m, 2 H), 7.40 (t, *J* = 8.0 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.5, 147.5, 140.9, 138.9, 136.8, 131.8, 131.2, 130.5, 130.4, 129.4, 128.9, 127.7, 127.7, 123.0, 117.6.

***2-(o-tolylsulfonyl)quinoline* (3al)[6]**



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.38 (d, *J* = 8.4 Hz, 1 H), 8.31 (dd, J_1 = 8.0 Hz, J_2 = 1.6 Hz, 1 H), 8.17 (d, *J* = 8.4 Hz, 1 H), 8.10 (d, *J* = 8.4 Hz, 1 H), 7.88 (dd, J_1 = 8.0 Hz, J_2 = 1.2 Hz, 1 H), 7.78 – 7.75 (m, 1 H), 7.65 – 7.63 (m, 1 H), 7.51 – 7.47 (m, 1 H), 7.41 (t, *J* = 7.6 Hz, 1 H), 7.24 (d, J = 7.2 Hz, 1 H), 2.56 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.1, 147.1, 139.1, 138.6, 137.1, 133.9, 132.4, 130.9, 130.6, 130.3, 129.1, 128.8, 127.7, 126.3, 117.7, 20.7.

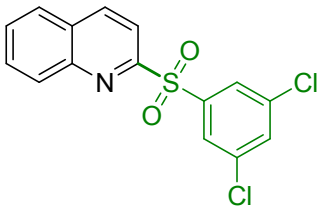
***2-((3,4-difluorophenyl)sulfonyl)quinolone* (3am)**



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.41 (d, *J* = 8.0 Hz, 1 H), 8.19 (d, *J* = 8.4 Hz, 1 H), 8.13 (dd, J_1 = 8.4 Hz, J_2 = 1.2 Hz, 1 H), 8.02 –7.97 (m, 1 H), 7.94 – 7.88 (m, 2 H), 7.82 – 7.77 (m, 1 H), 7.69 – 7.65 (m, 1 H), 7.35 – 7.29 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.3, 153.9 (dd, J_1 = 257.4 Hz, J_2 = 12.4 Hz, 1C), 150.1 (dd, J_1 = 253.8 Hz, J_2 = 13.9 Hz, 1C), 147.3, 139.0, 131.2, 130.2, 129.4, 128.9, 127.7, 126.4 (dd, J_1 = 8.0 Hz, J_2 = 4.3 Hz, 1C), 119.0 (dd, J_1 = 19.7 Hz, J_2 = 2.2 Hz, 1C), 118.3, 118.1, 117.3; ^{19}F NMR (376 MHz, $CDCl_3$): δ = - 127.6 (d, *J* = 20.3, 1F), -133.6 (d, *J* = 20.3, 1F); IR (in KBr): ν = 3038, 1932, 1670, 1497, 1327, 1268, 907, 833, 693 (cm^{-1})； HRMS (ESI)

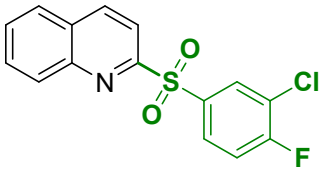
m/z calcd. for $C_{15}H_{10}F_2NO_2S[M+H]^+$: 306.0395, found 306.0393.

2-((3,5-dichlorophenyl)sulfonyl)quinolone **(3an)**



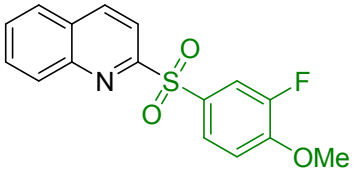
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.42 (dd, J_1 = 8.8 Hz, J_2 = 0.4 Hz, 1 H), 8.20 (d, J = 8.8 Hz, 1 H), 8.16 (dd, J_1 = 8.4 Hz, J_2 = 0.8 Hz, 1 H), 8.01 (d, J = 1.6 Hz, 2 H), 7.90 (dd, J_1 = 8.4 Hz, J_2 = 1.2 Hz, 1 H), 7.82 – 7.79 (m, 1 H), 7.71 – 7.67 (m, 1 H), 7.54 (t, J = 2.0 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 156.9, 147.4, 141.9, 139.1, 135.9, 133.7, 131.3, 130.3, 129.6, 129.0, 127.7, 127.4, 117.5； IR (in KBr): ν = 2924, 1517, 1415, 1331, 1154, 1116, 909, 819, 694(cm^{-1})；HRMS (ESI) m/z calcd. for $C_{15}H_{10}Cl_2NO_2S[M+H]^+$: 337.9804, found 337.9798.

2-((3-chloro-4-fluorophenyl)sulfonyl)quinoline **(3ao)**[5]



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.41 (d, J = 8.4 Hz, 1 H), 8.23 – 8.19 (m, 2 H), 8.14 (d, J= 8.4 Hz, 1 H), 8.08 – 8.04 (m, 1 H), 7.90 (d, J = 8.4 Hz, 1 H), 7.83 – 7.87 (m, 1 H), 7.70 – 7.66 (m, 1 H), 7.30 (t, J = 8.4 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 161.3 ($J_{C\text{-}F}$ = 256.6 Hz), 157.4, 147.4, 139.0, 135.9 ($J_{C\text{-}F}$ = 3.6 Hz),, 132.0 ($J_{C\text{-}F}$ = 1.5 Hz), 131.2, 130.2, 129.7 ($J_{C\text{-}F}$ = 8.8 Hz), 129.5, 128.9, 127.7, 122.5 ($J_{C\text{-}F}$ = 18 Hz), 117.5, 117.4 ($J_{C\text{-}F}$ = 21.8 Hz), 117.4; ^{19}F NMR (376 MHz, $CDCl_3$): δ = - 105.5.

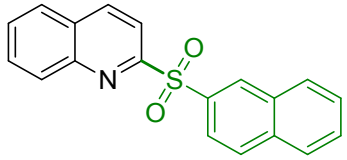
2-((3-fluoro-4-methoxyphenyl)sulfonyl)quinolone **(3ap)**



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.38 (d, J = 8.0 Hz, 1 H), 8.19 – 8.15 (m, 2 H), 7.93 – 7.77 (m, 4 H), 7.68 – 7.64 (m, 1 H), 7.08 – 7.04 (m, 1 H), 3.92 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.0, 151.6 (d, $J_{C\text{-}F}$ = 250.8), 152.4 (d, $J_{C\text{-}F}$ = 10.2), 147.4, 138.8, 131.0, 130.3, 129.2, 128.8, 127.7, 126.5 (d, $J_{C\text{-}F}$ = 3.6), 117.1, 117.4, 116.9, 112.8 (d, $J_{C\text{-}F}$ = 2.2), 56.4; ^{19}F NMR (376 MHz, $CDCl_3$): δ = - 131.8; IR (in KBr): ν = 3421, 1600, 1504, 1430,

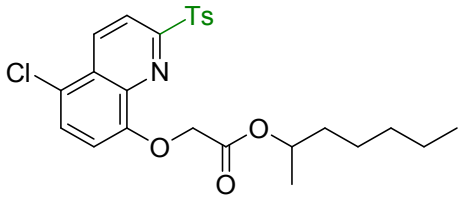
1329, 1283, 1165, 1010, 819, 686(cm^{-1})；HRMS (ESI) m/z calcd. for $C_{16}H_{13}FNO_3S[M+H]^+$: 318.0595, found 318.0598.

***2-(naphthalen-2-ylsulfonyl)quinoline* (3aq)**[4]



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.75 (d, *J* = 2.0 Hz, 1 H), 8.38 (d, *J* = 8.4 Hz, 1 H), 8.27 (d, *J* = 8.4 Hz, 1 H), 8.16 (d, *J* = 8.4 Hz, 1 H), 8.08 (dd, J_1 = 8.4 Hz, J_2 = 1.6 Hz, 1 H), 8.00 (d, *J* = 8.0 Hz, 1 H), 7.95 (d, *J* = 8.4 Hz, 1 H), 7.88 – 7.85 (m, 2 H), 7.78 – 7.74 (m, 1 H), 7.66 – 7.57 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.1, 147.4, 138.7, 136.0, 135.3, 132.1, 131.0, 130.8, 130.3, 129.5, 129.3, 129.2, 128.8, 127.9, 127.6, 127.5, 123.7, 117.8.

***heptan-2-yl 2-((5-chloro-2-tosylquinolin-8-yl)oxy)acetate* (3sa)**



^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.74 (d, *J* = 8.8 Hz, 1 H), 8.30 (d, *J* = 8.8 Hz, 1 H), 8.06 (d, *J* = 8.4 Hz, 2 H), 7.60 (d, *J* = 8.4 Hz, 1 H), 7.35 (d, *J* = 8.0 Hz, 2 H), 7.11 (d, *J* = 8.04 Hz, 1 H), 5.04 – 4.99 (m, 1 H), 4.85 (s, 2 H), 2.41 (s, 3 H), 1.51 – 1.46 (m, 2 H), 1.25 – 1.23 (s, 9 H), 0.86 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.1, 153.5, 145.0, 140.3, 136.1, 135.7, 129.8, 129.4, 128.9, 127.8, 124.0, 118.6, 114.6, 72.6, 67.8, 35.8, 31.5, 25.0, 22.5, 21.7, 19.9, 13.9; IR (in KBr): ν = 3425, 2927, 1765, 1570, 1415, 1318, 1209, 1114, 813, 696 (cm^{-1})；HRMS (ESI) m/z calcd. for $C_{25}H_{29}ClNO_5S[M+H]^+$: 490.1449, found 490.1445.

4. References

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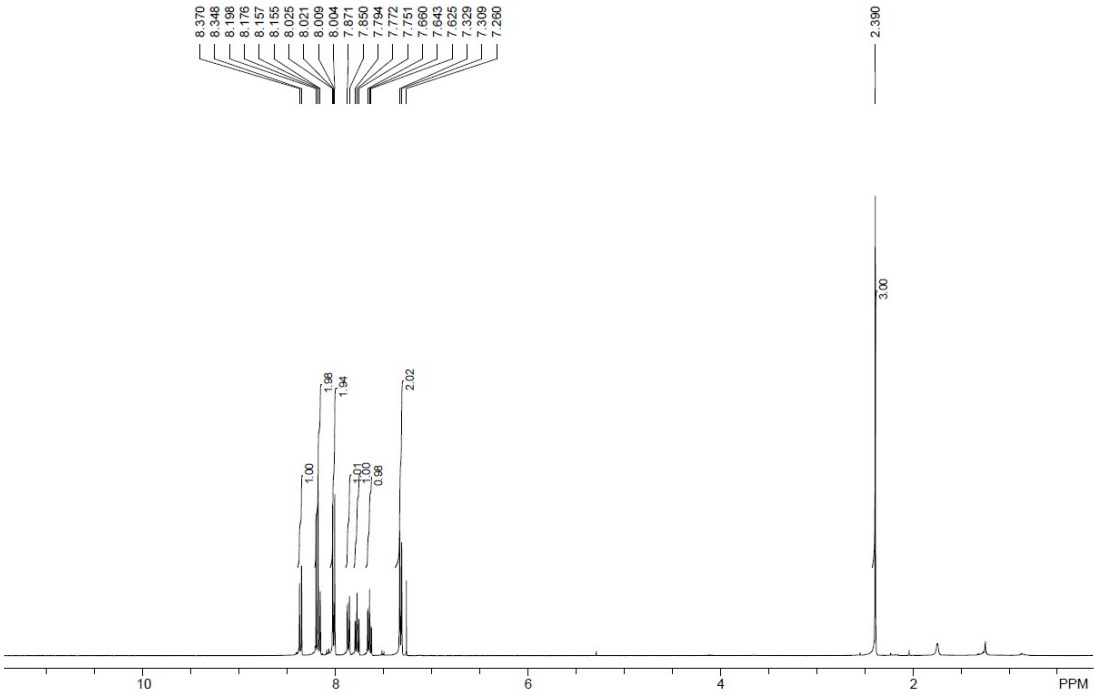
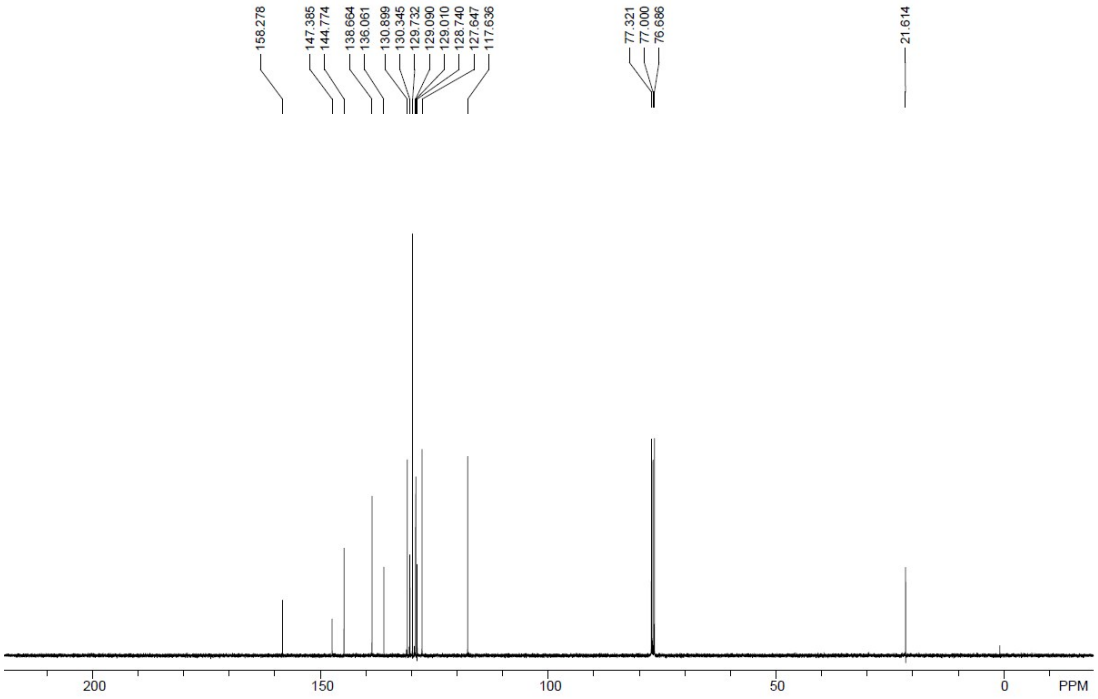
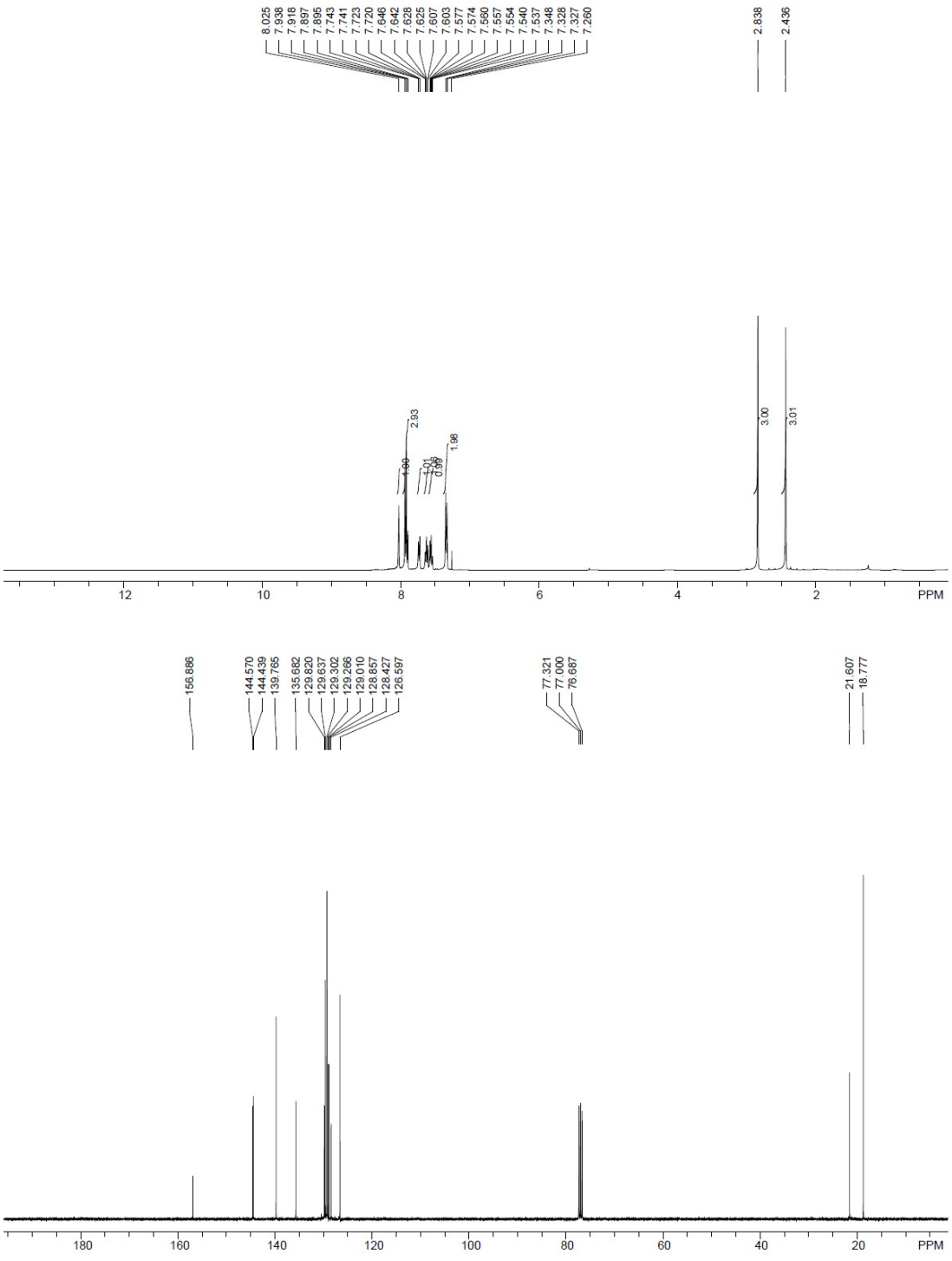
5. ^{1}H and ^{13}C NMR spectra of products

2-tosylquinoline **(3aa)**

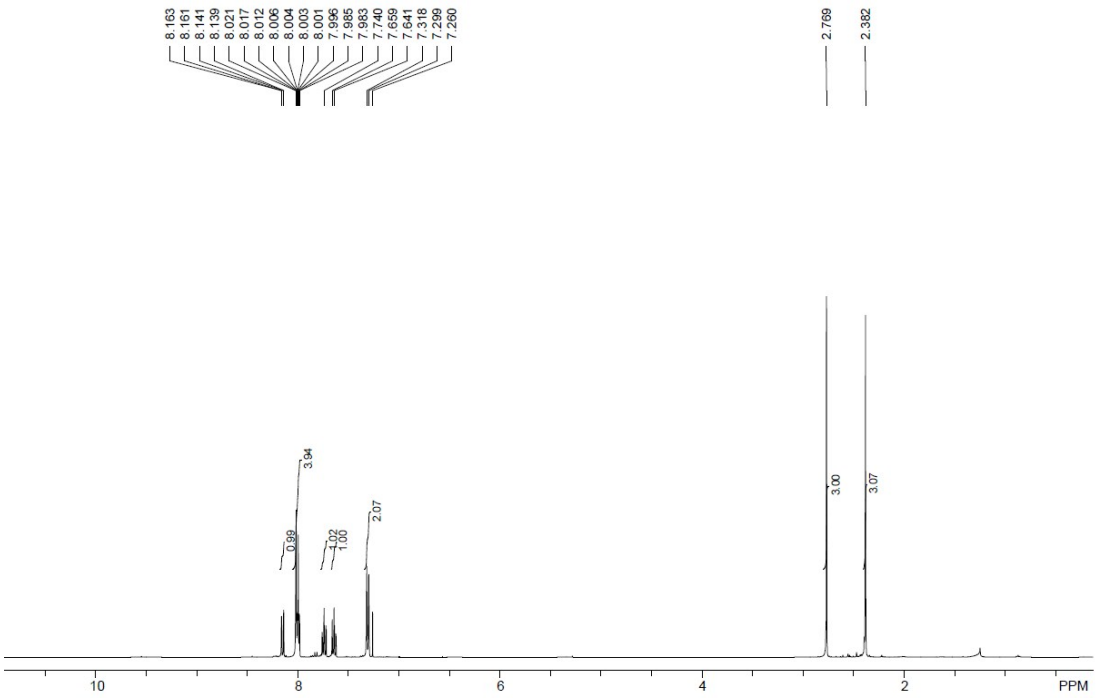


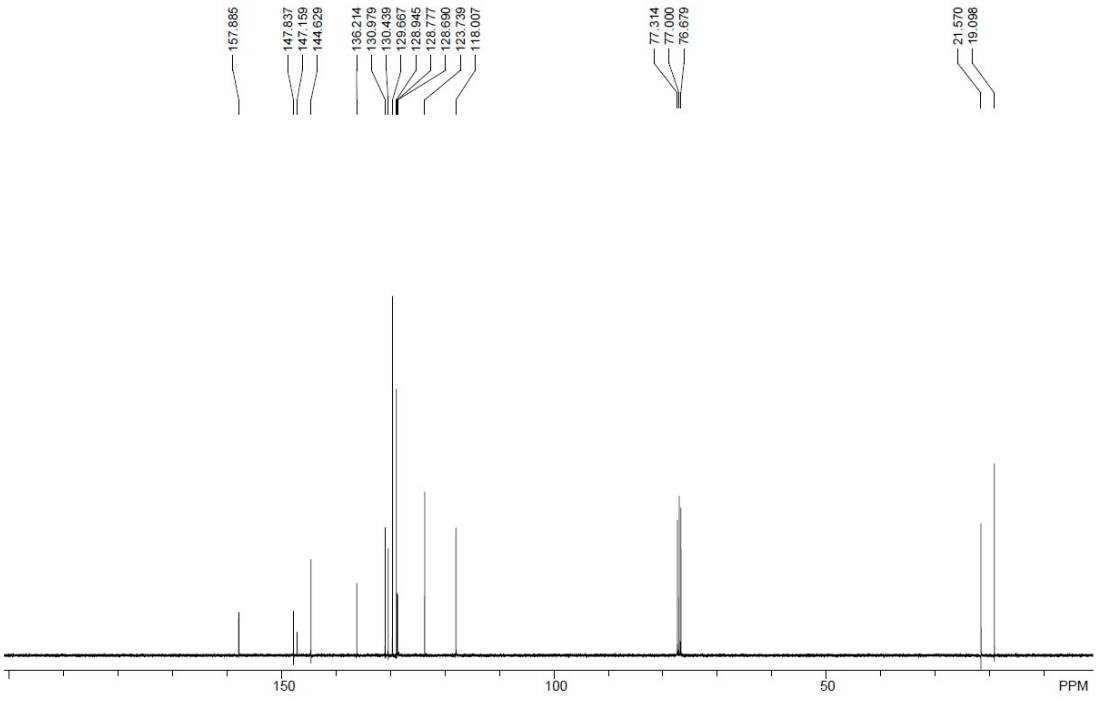


***3-methyl-2-tosylquinoline* (3ba)**

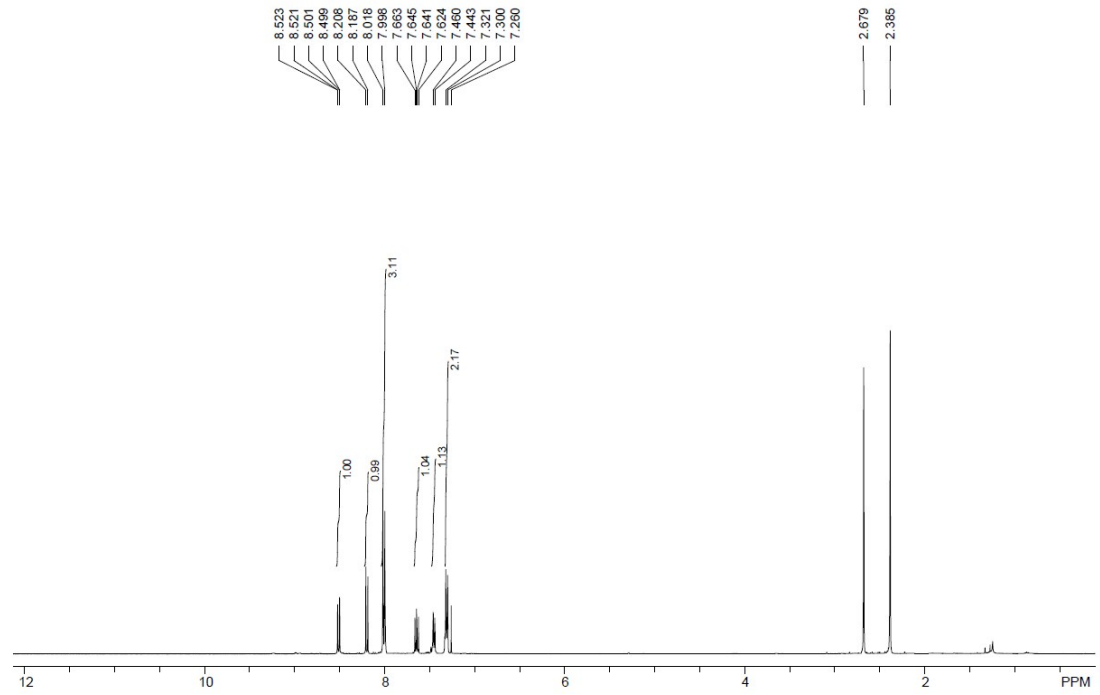


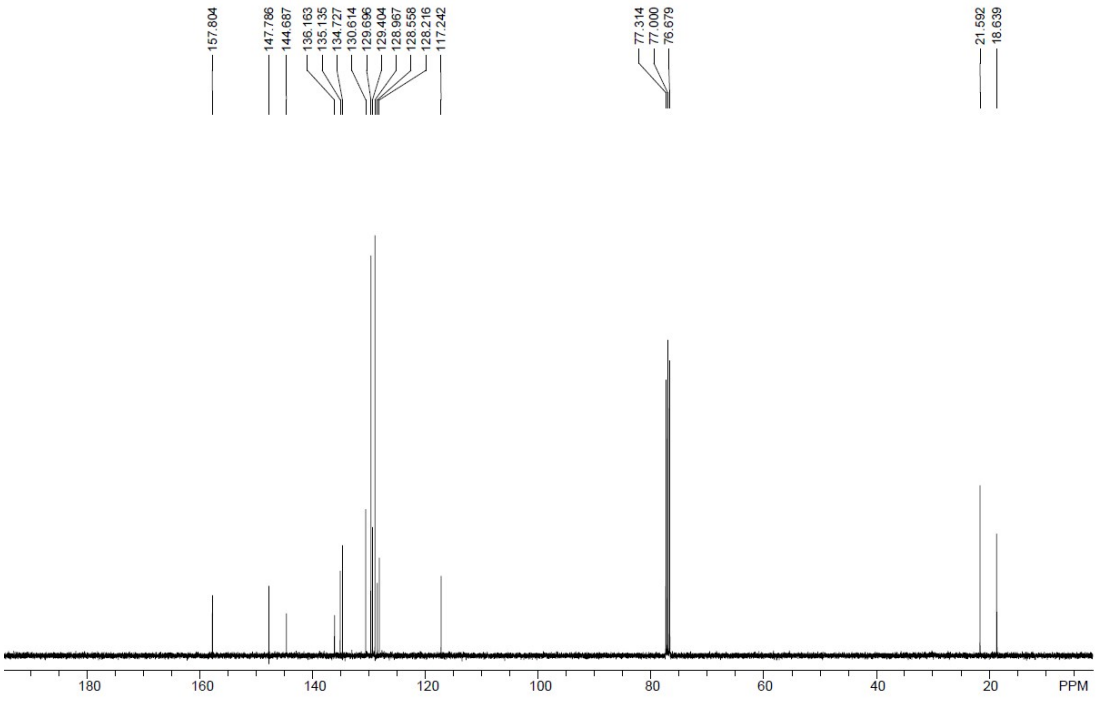
***4-methyl-2-tosylquinoline* (3ca)**



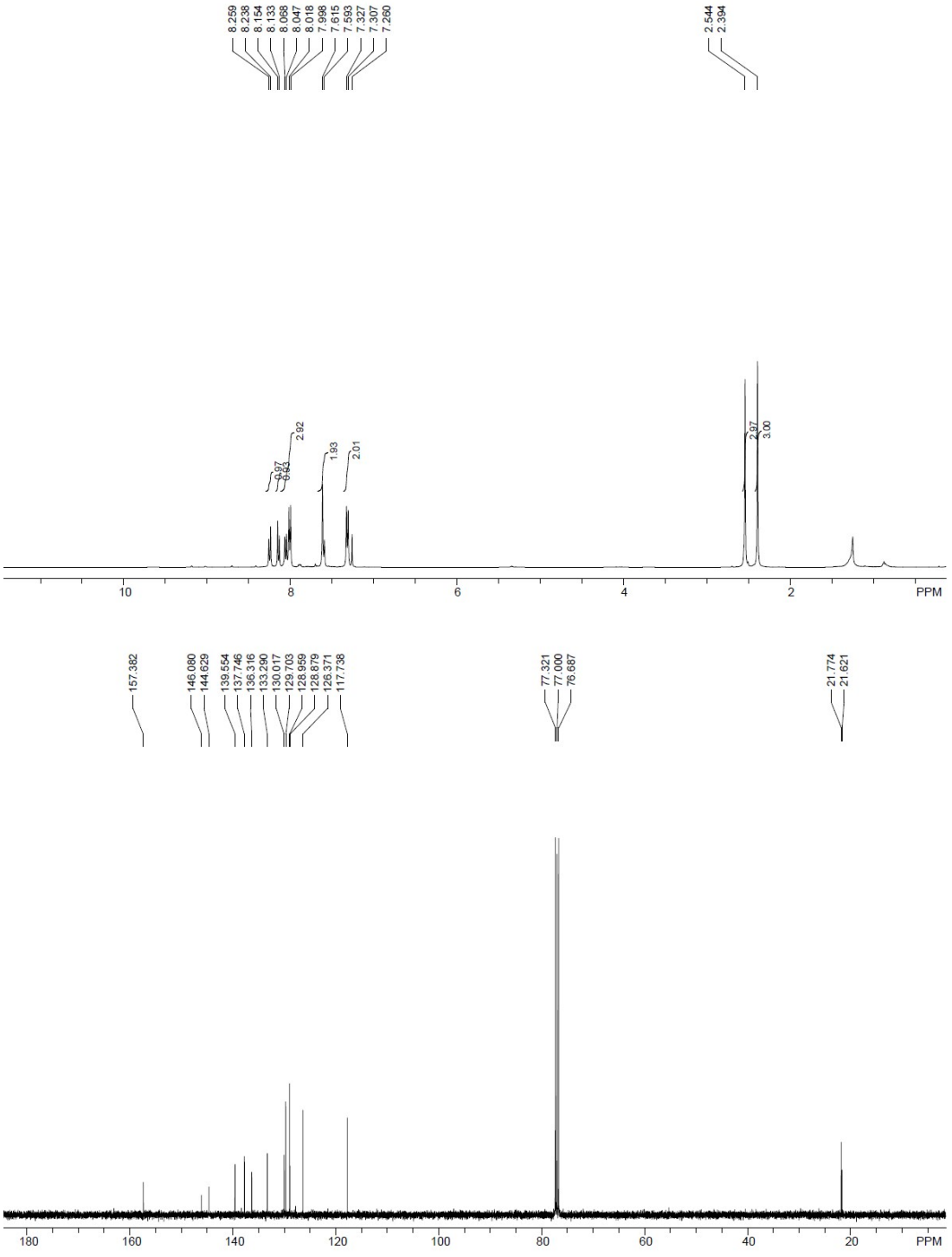


5-methyl-2-tosylquinoline **(3da)**

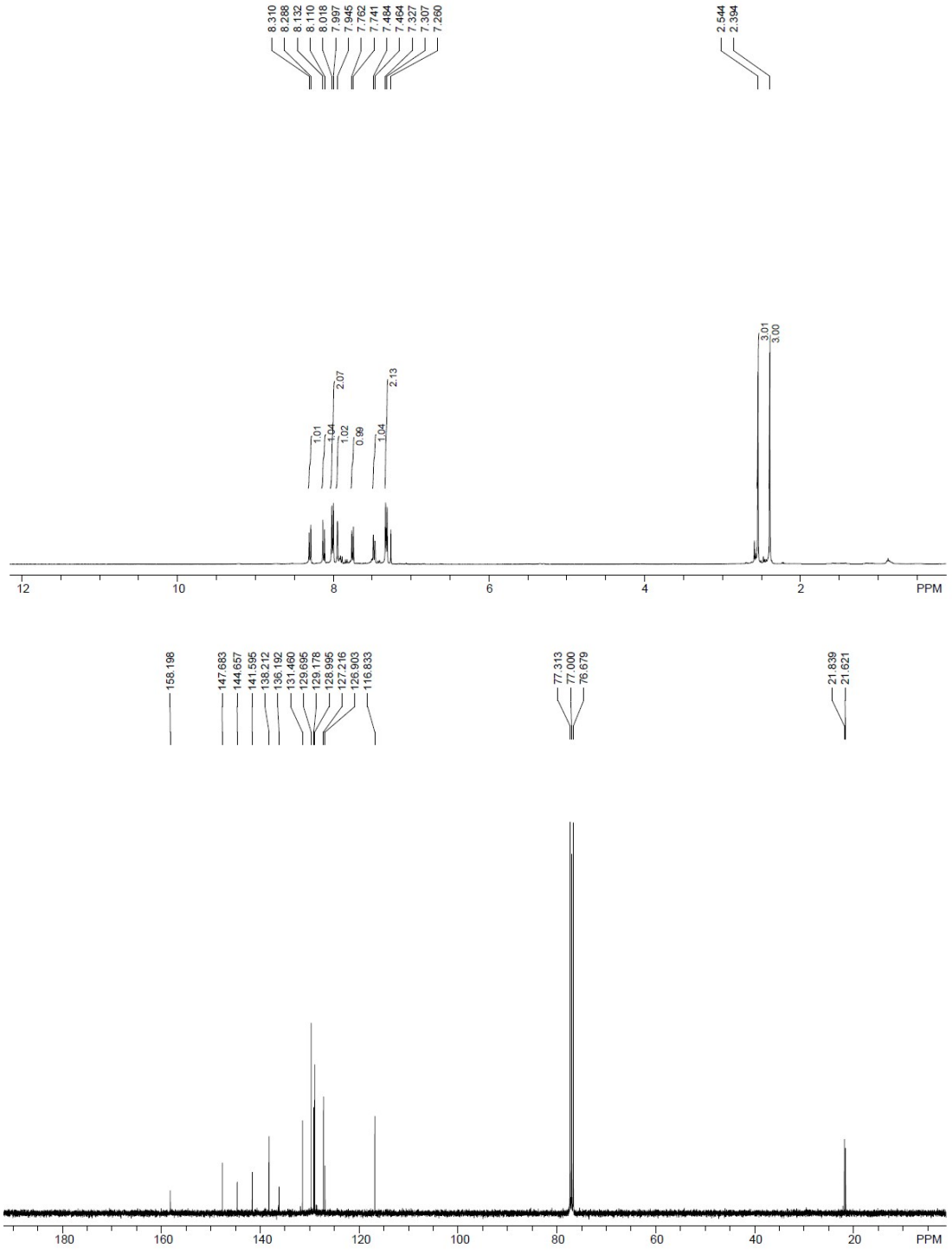




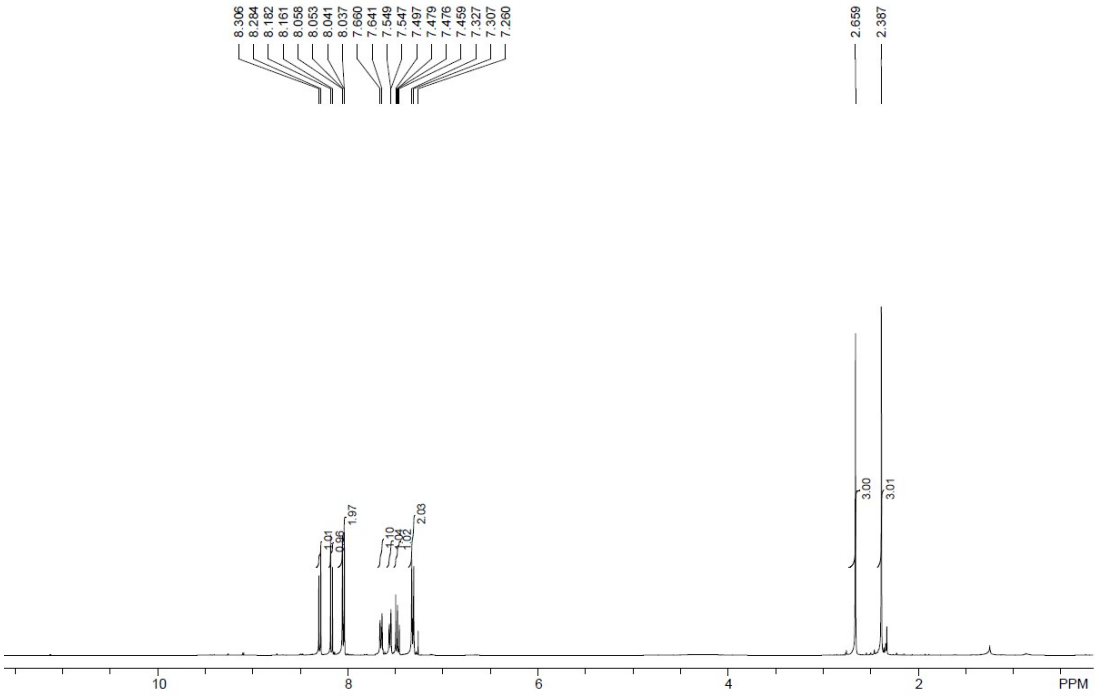
6-methyl-2-tosylquinoline **(3ea)**

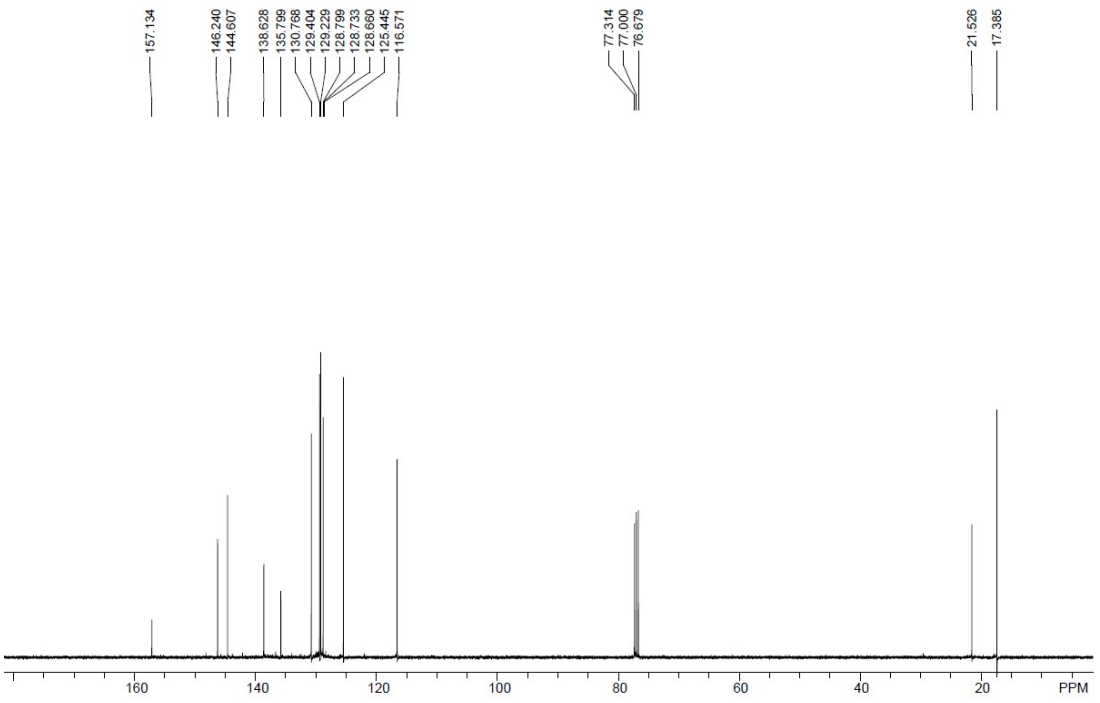


7-methyl-2-tosylquinoline **(3fa)**

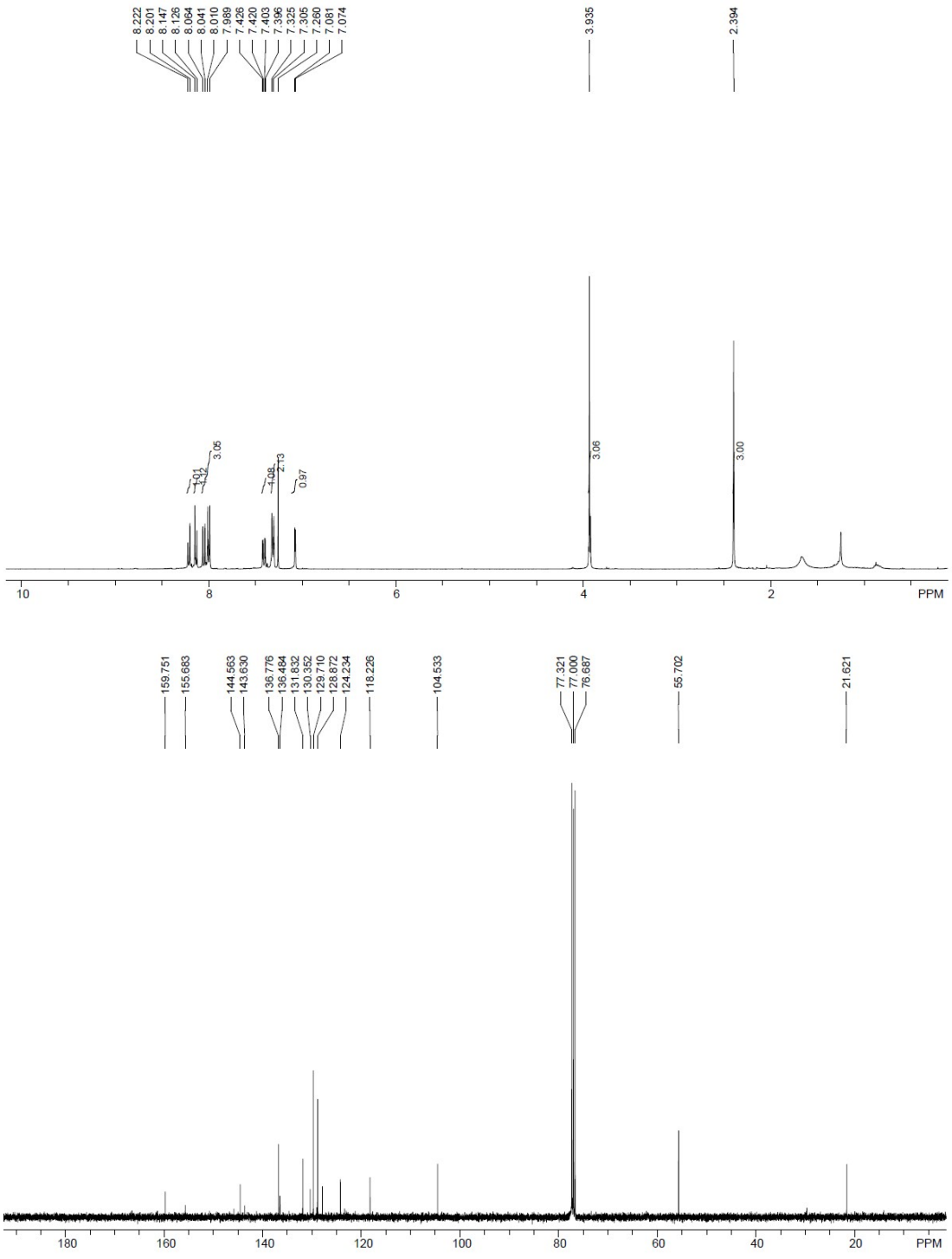


***8-methyl-2-tosylquinoline* (3ga)**

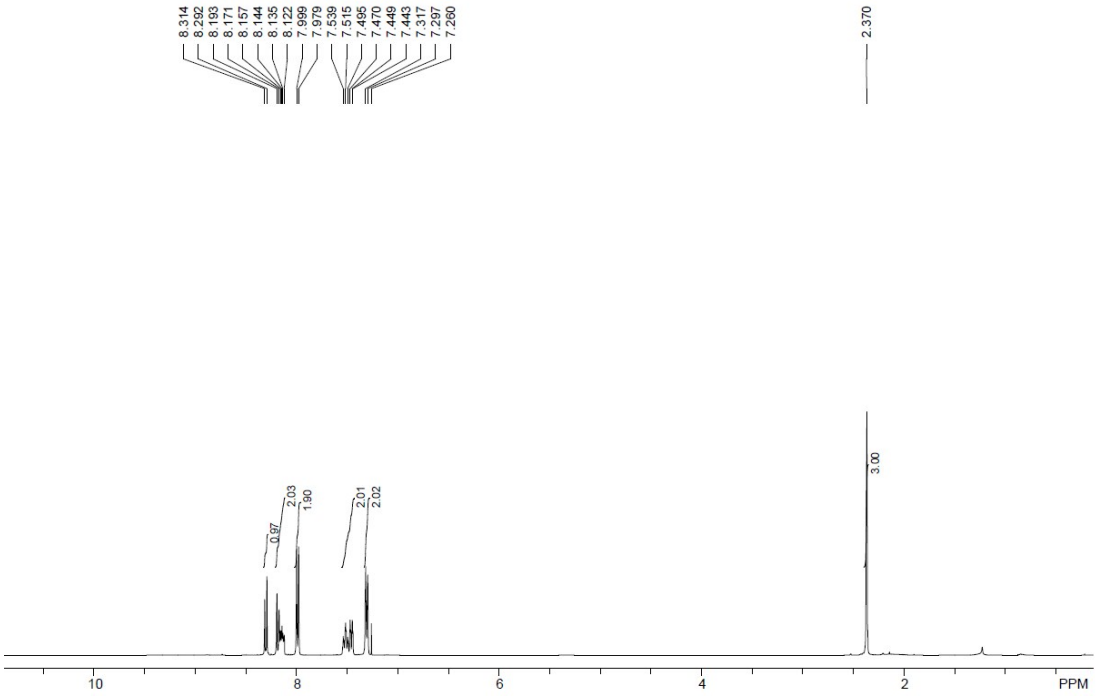


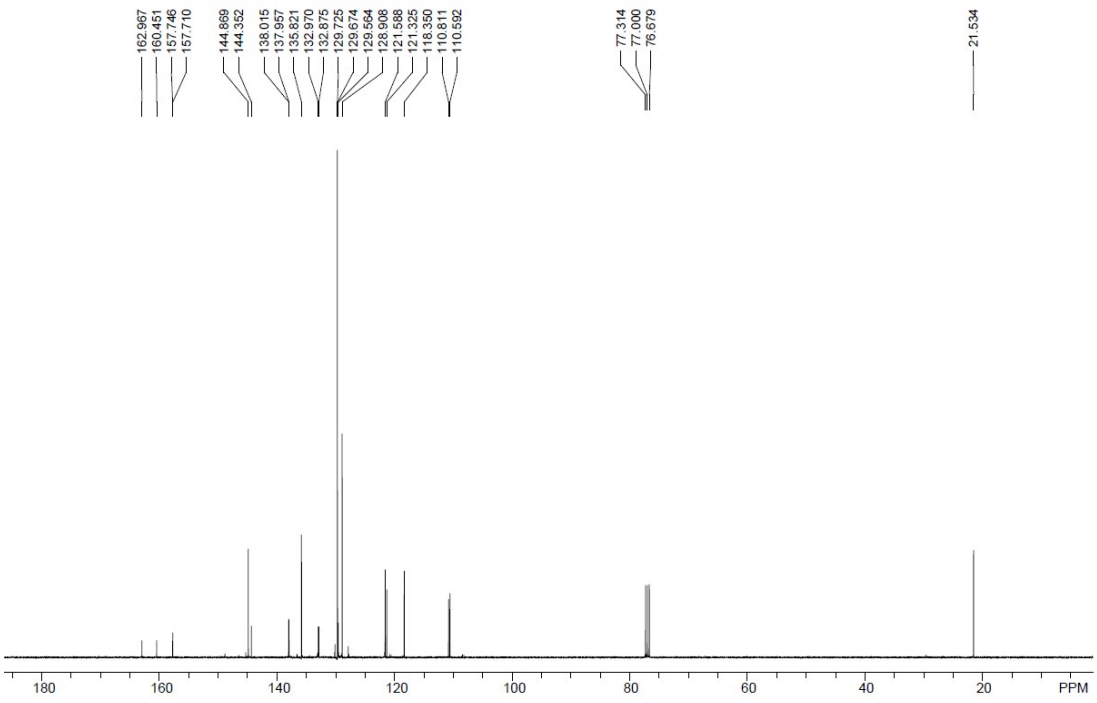


6-methoxy-2-tosylquinoline **(3ha)**

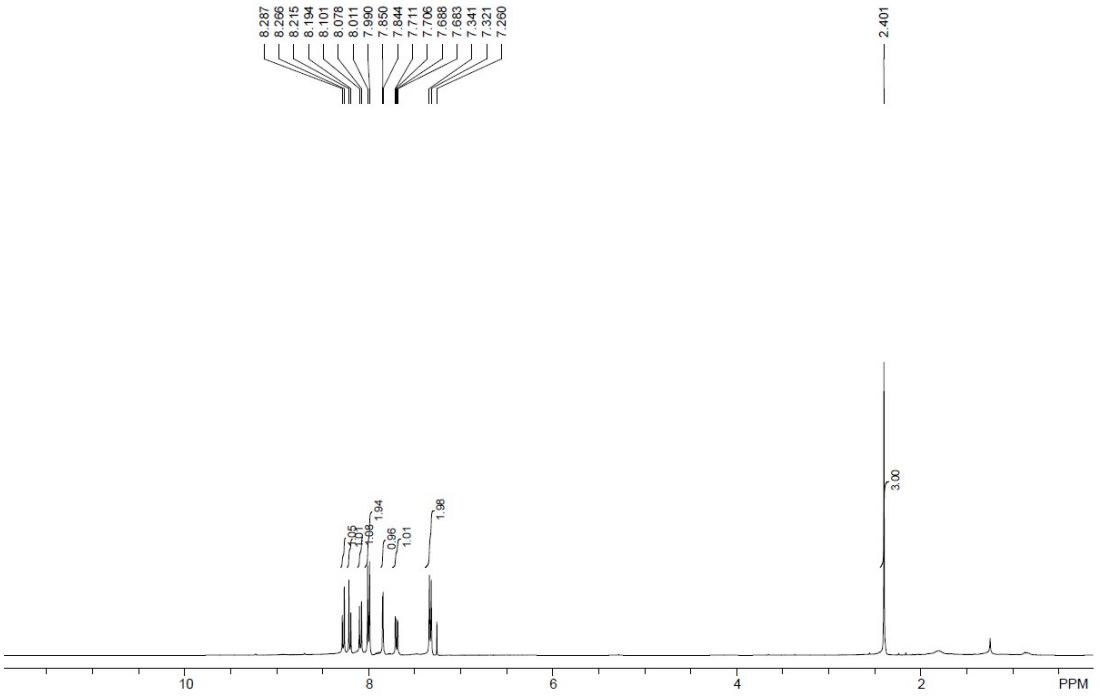


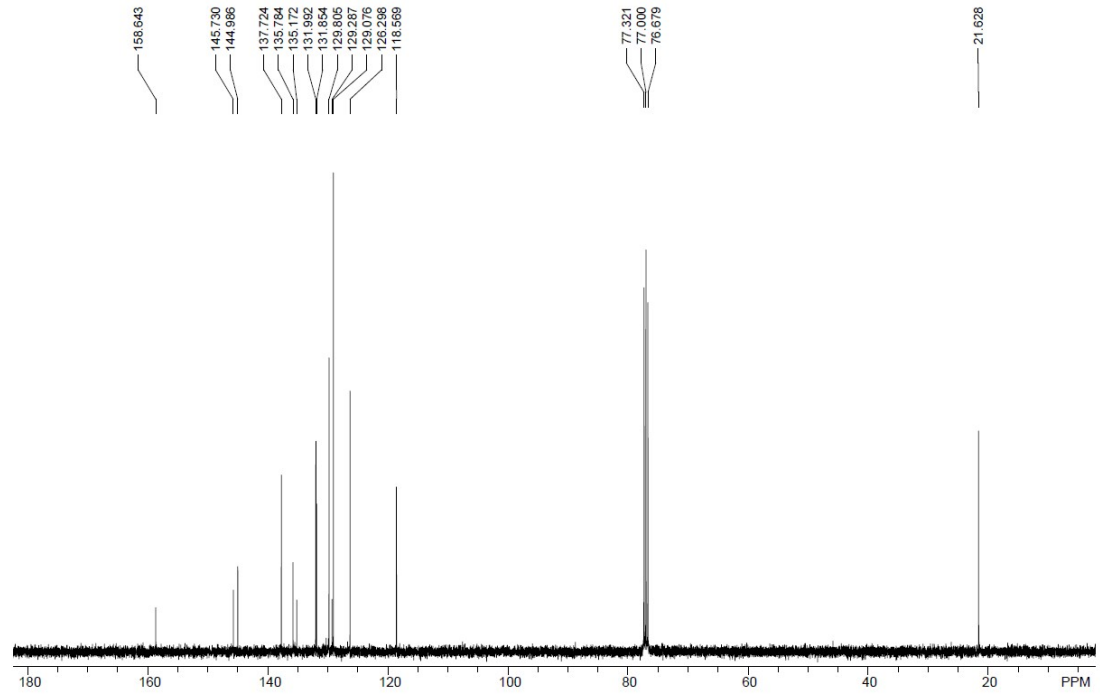
***6-fluoro-2-tosylquinoline* (3ia)**



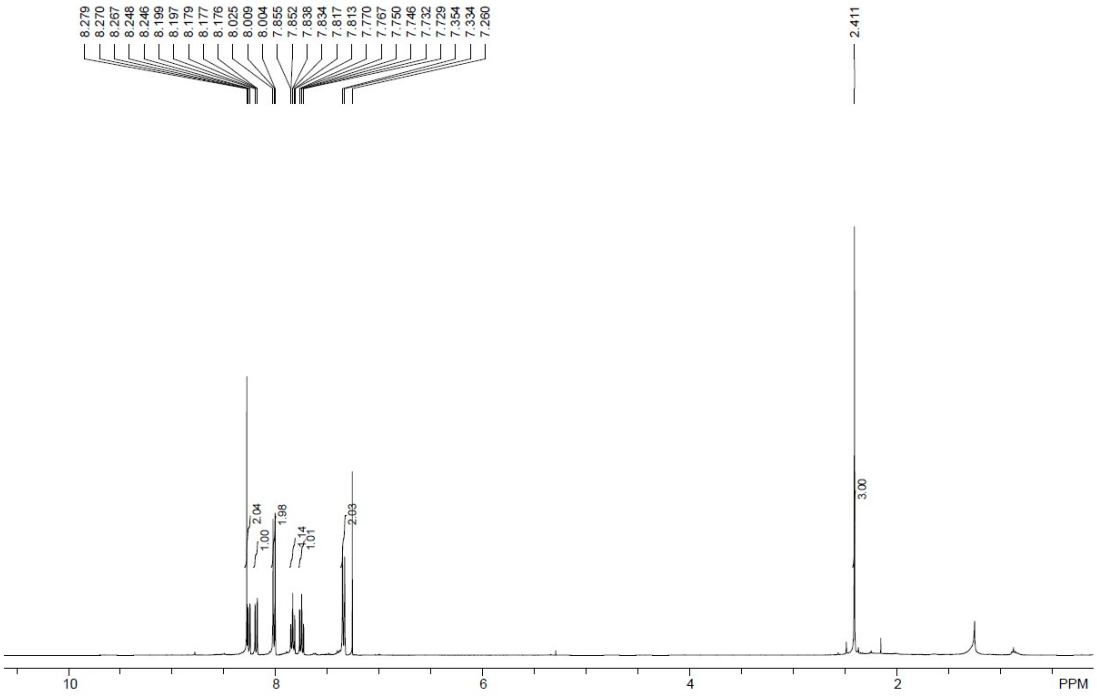


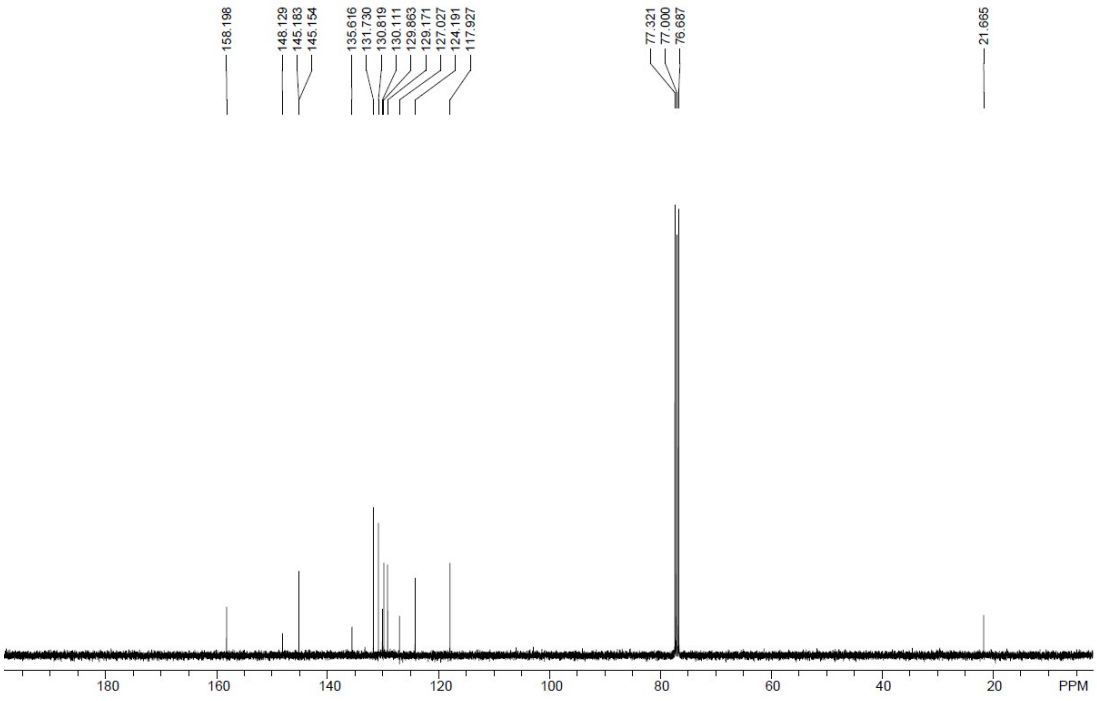
***6-chloro-2-tosylquinoline* (3ja)**



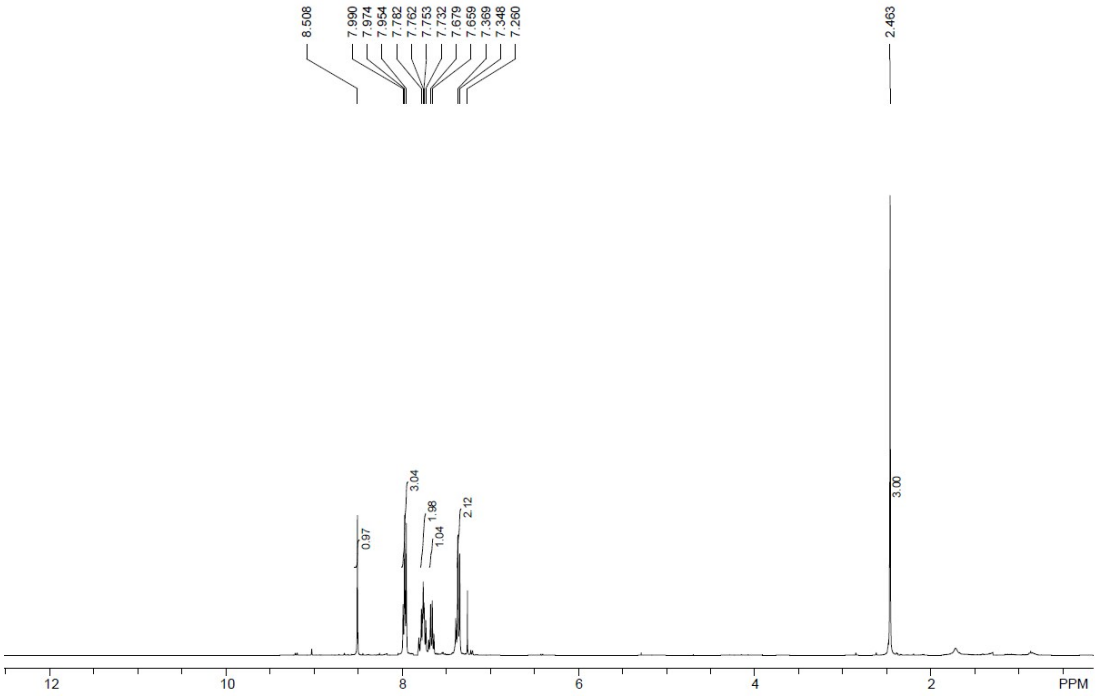


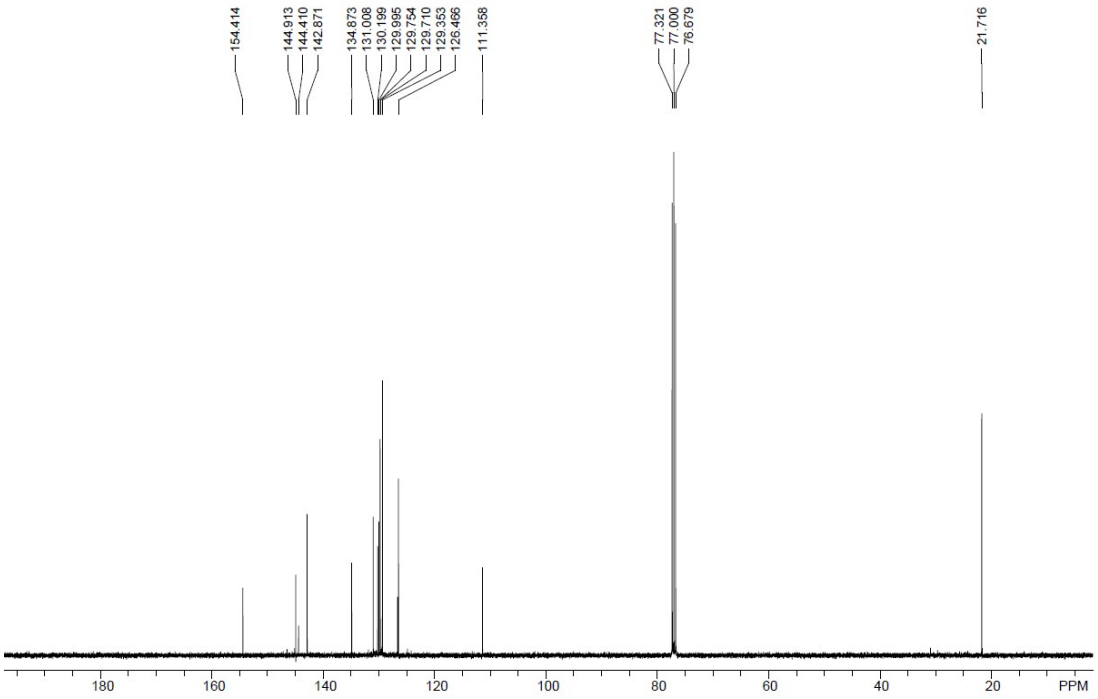
4-chloro-2-tosylquinoline **(3ka)**



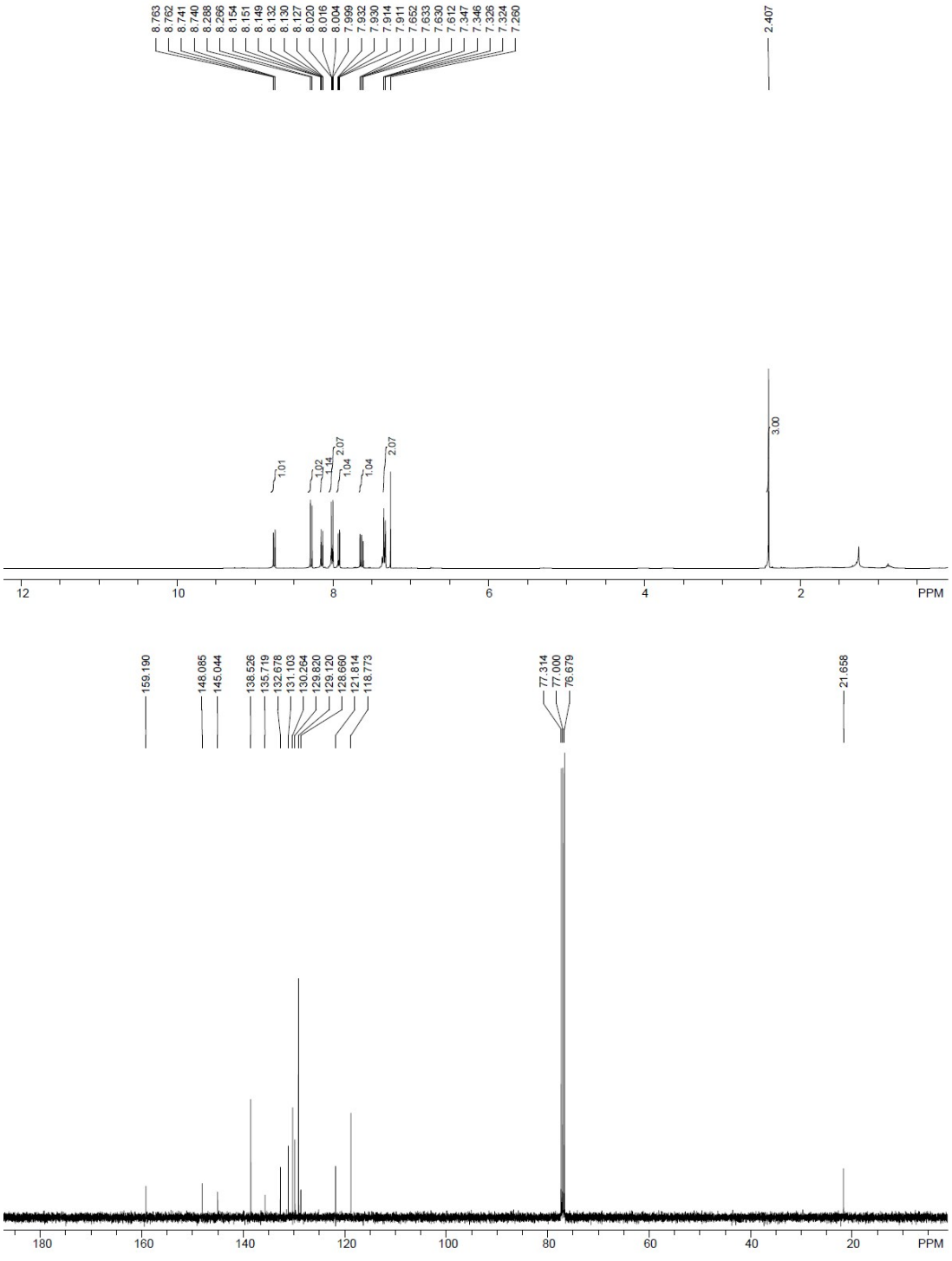


***3-bromo-2-tosylquinoline* (3la)**

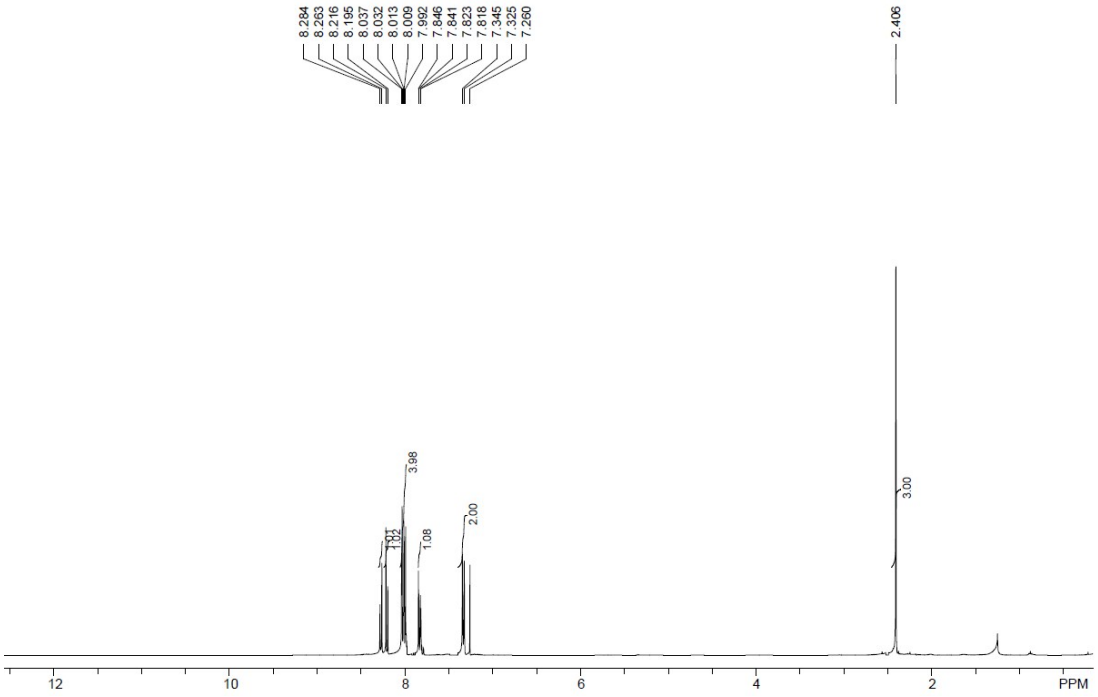


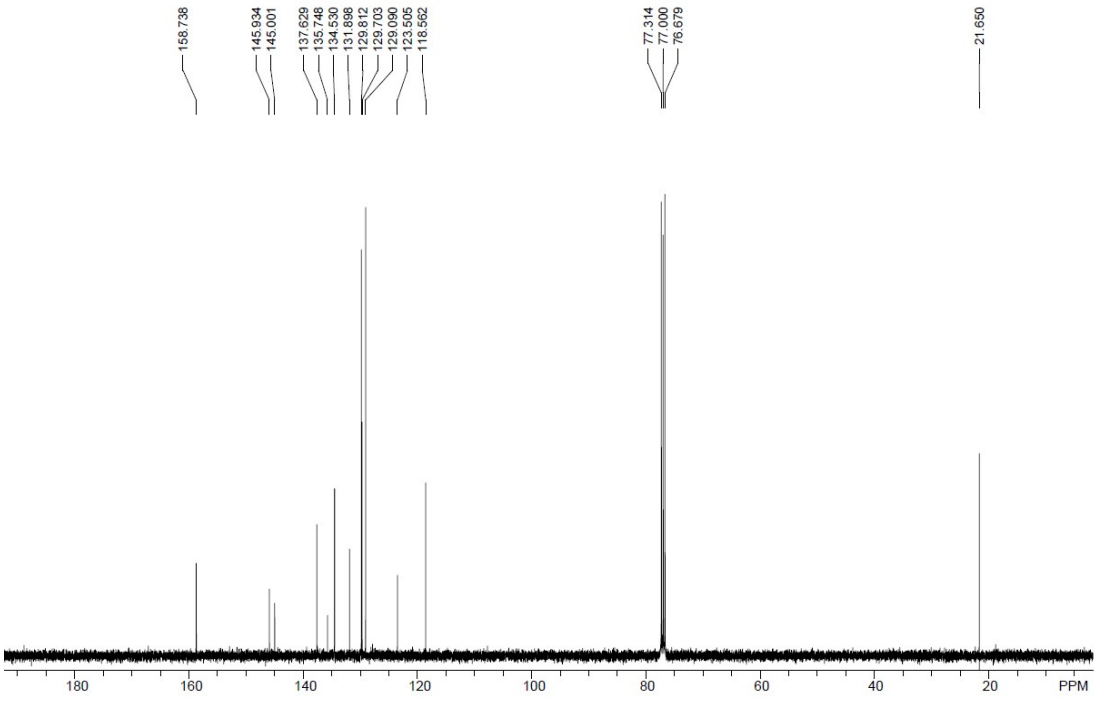


5-bromo-2-tosylquinoline **(3ma)**

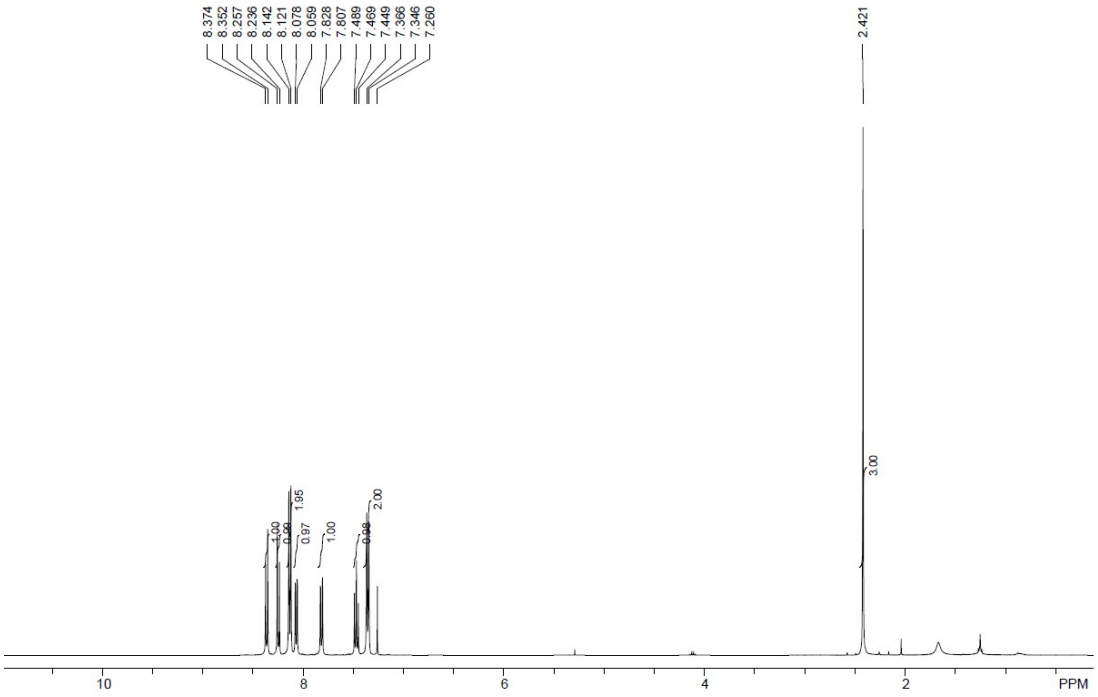


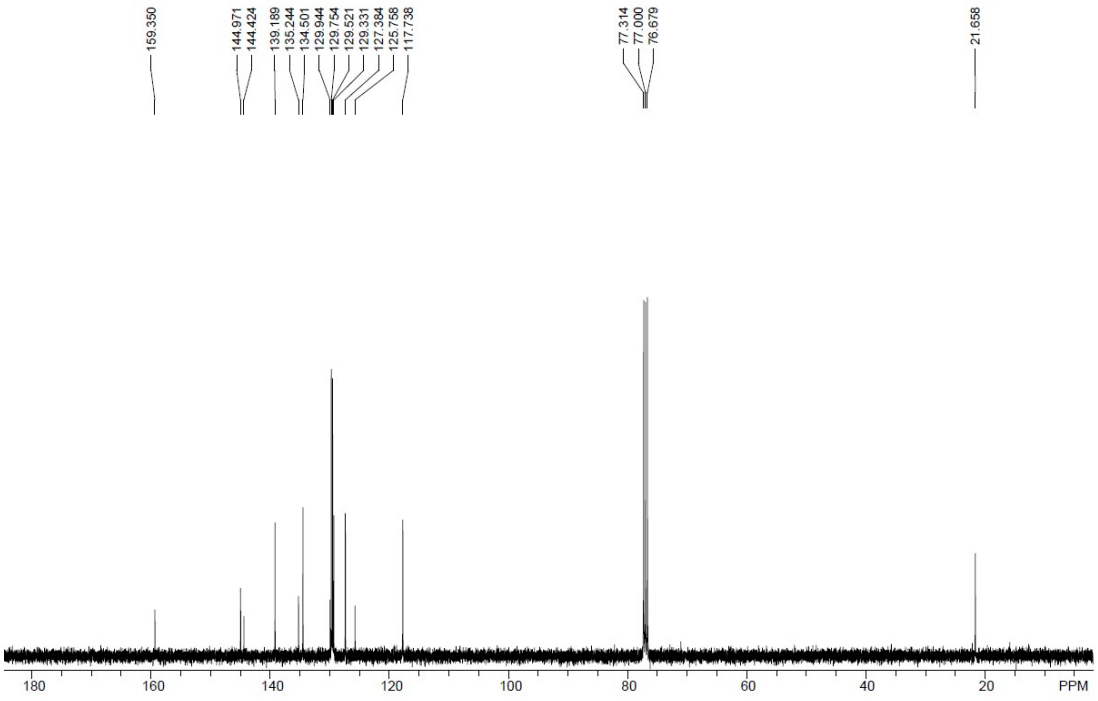
6-bromo-2-tosylquinoline **(3na)**



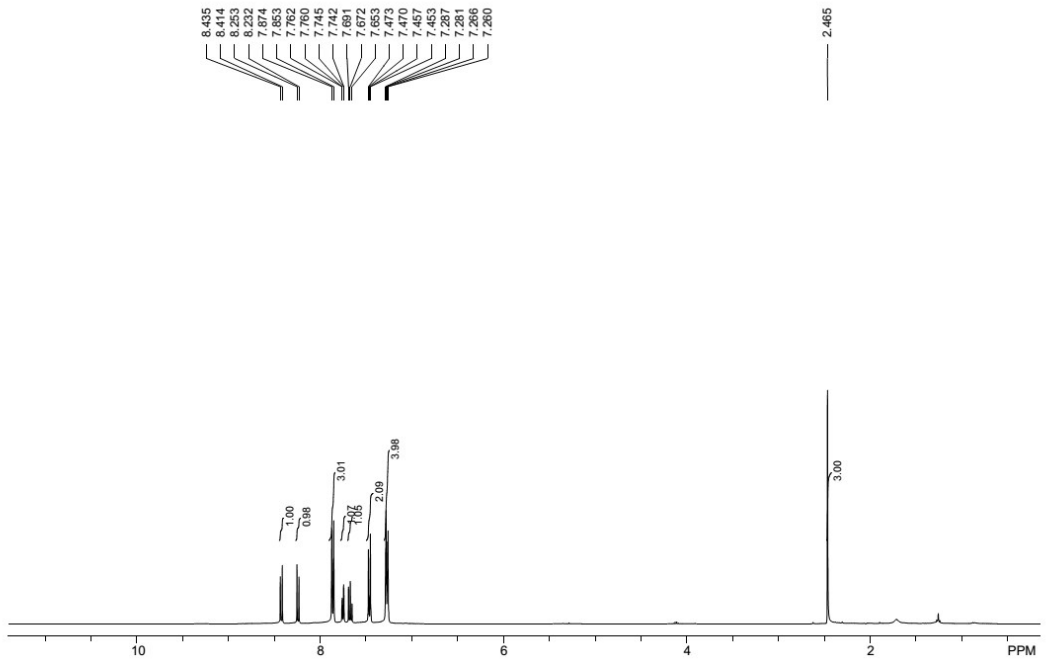


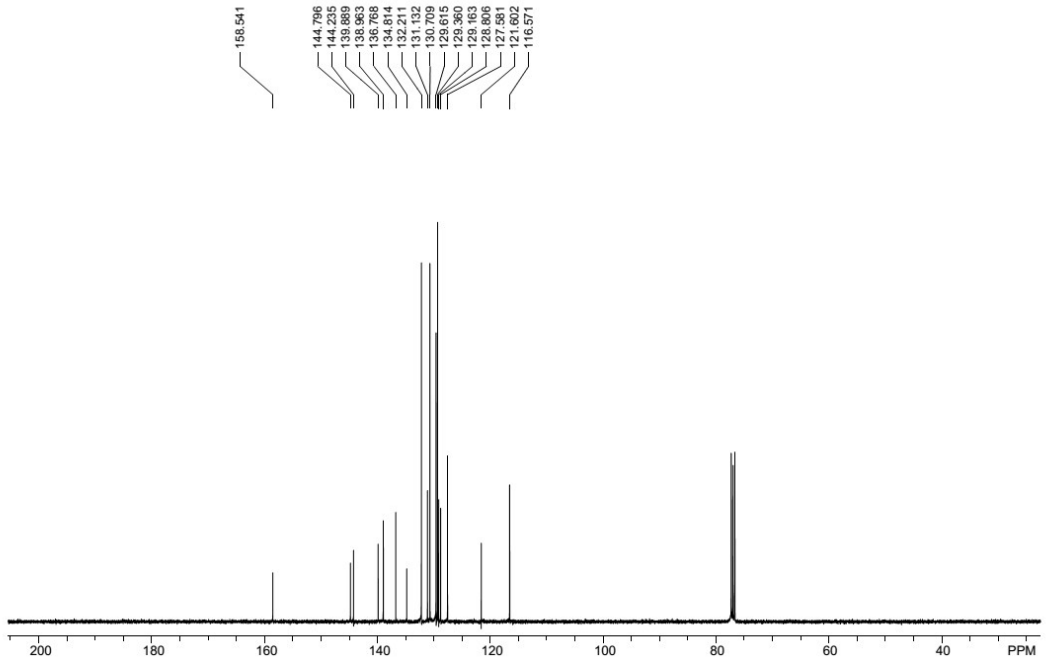
***8-bromo-2-tosylquinoline* (3oa)**



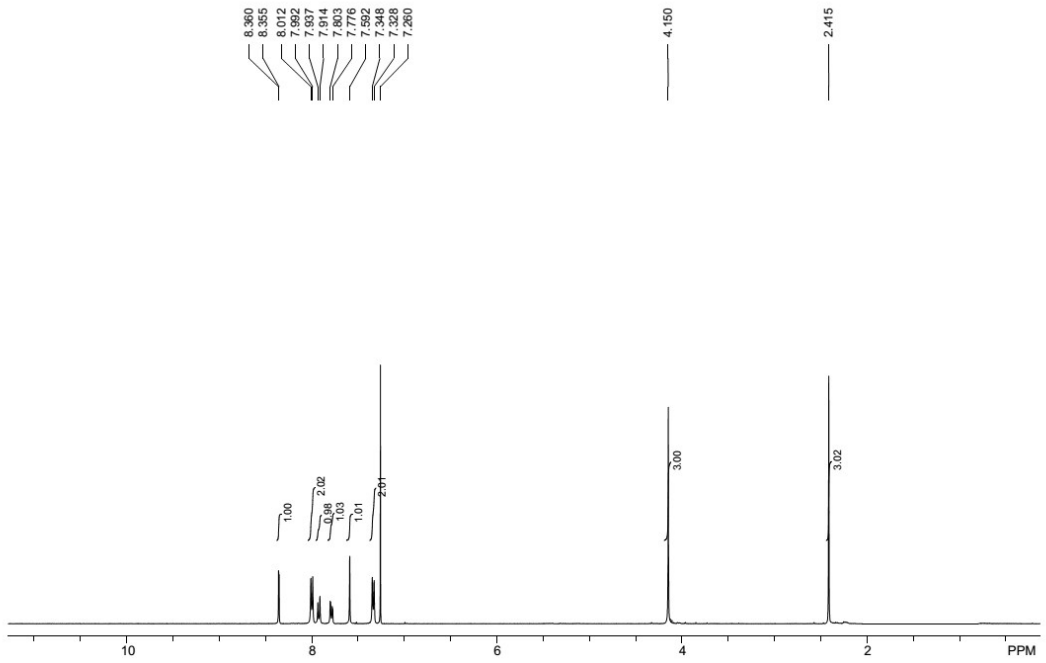


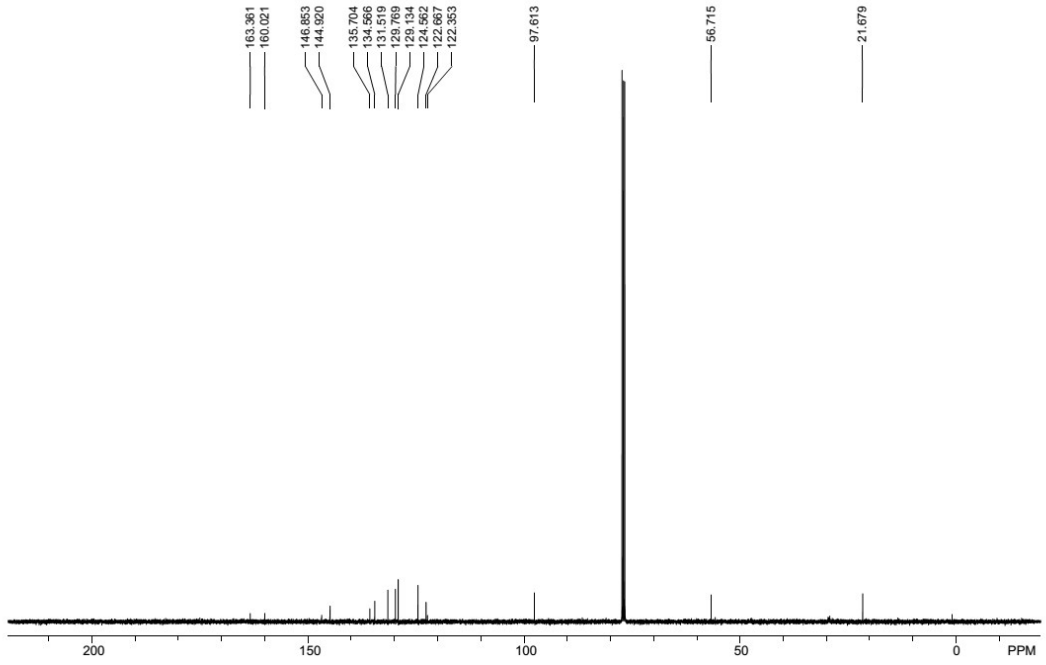
***8-(4-bromophenyl)-2-tosylquinoline* (3pa)**



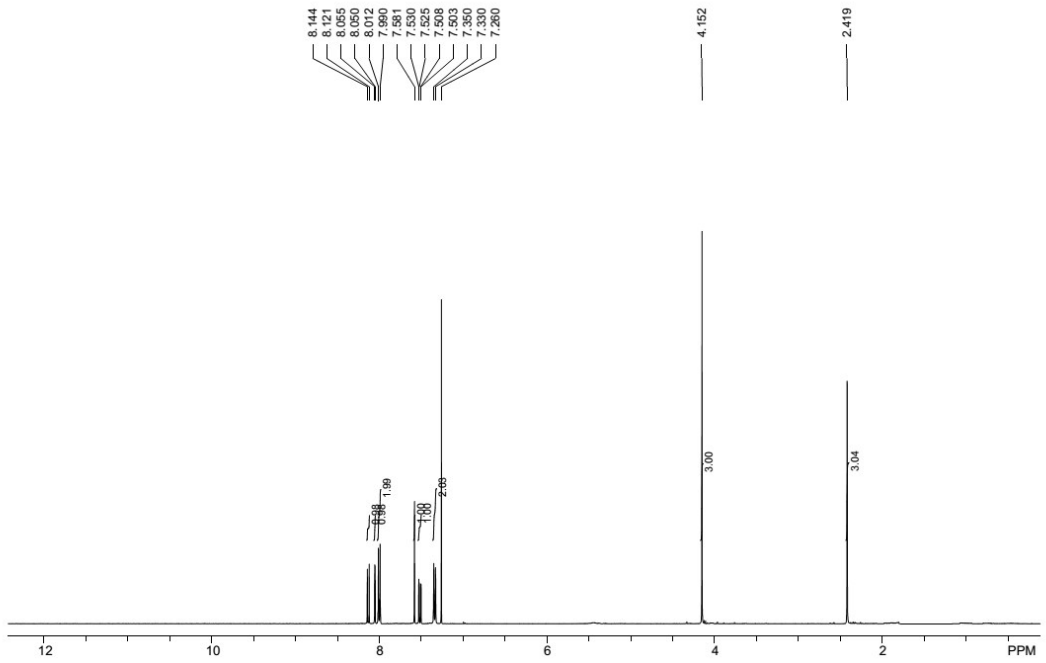


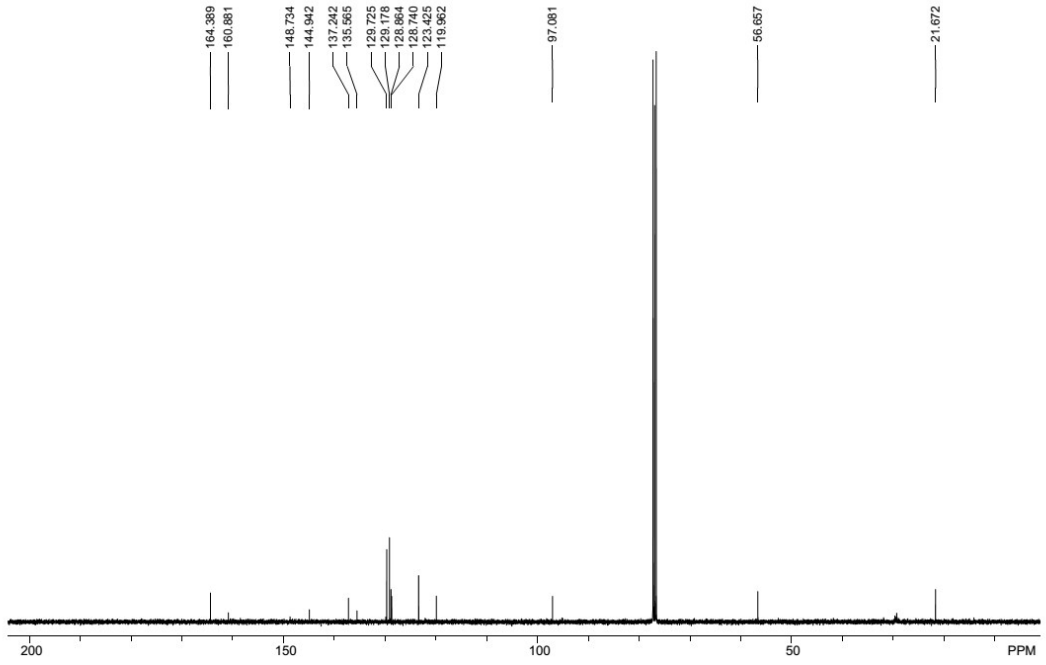
6-bromo-4-methoxy-2-tosylquinoline **(3qa)**



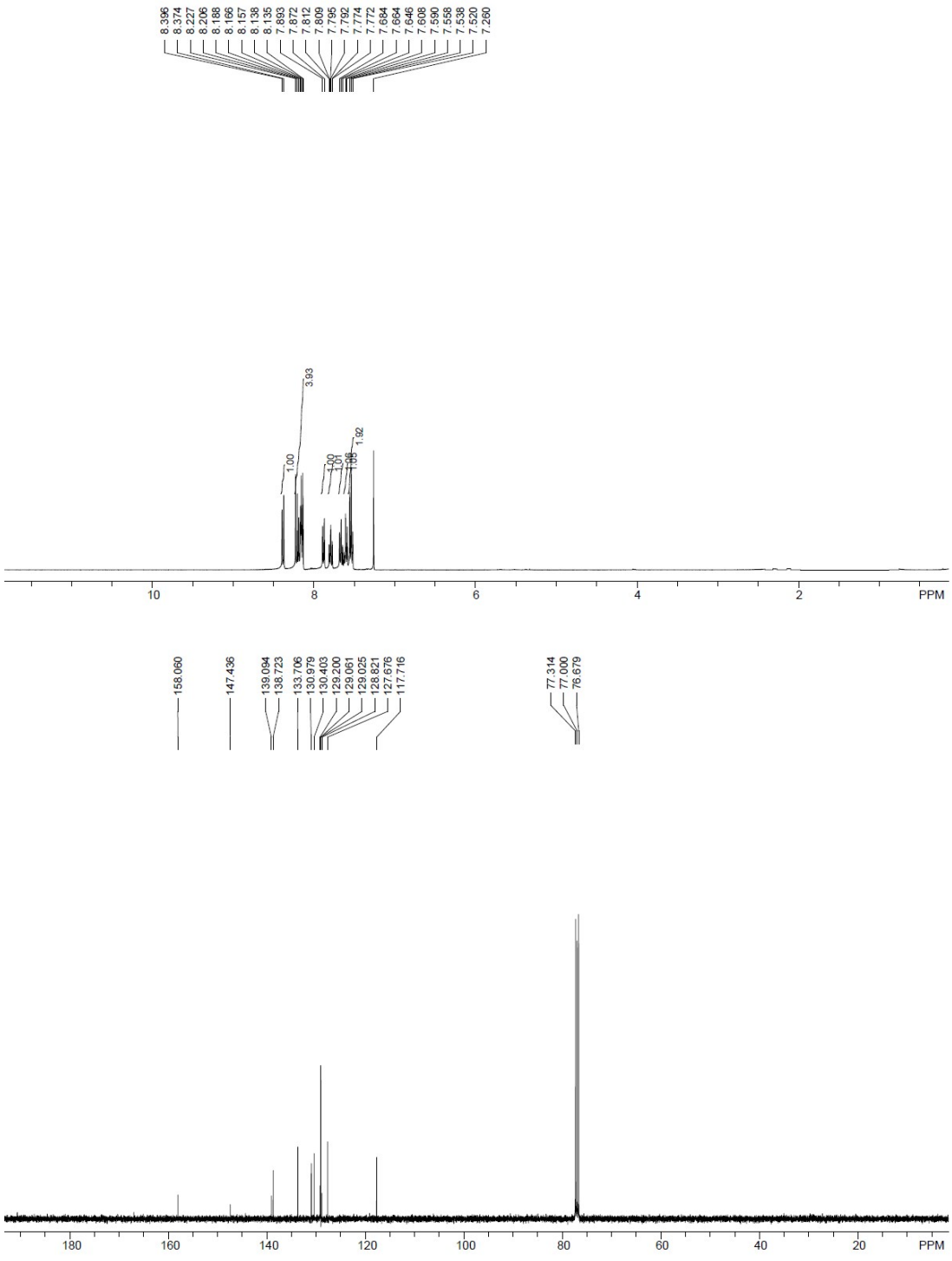


7-chloro-4-methoxy-2-tosylquinoline **(3ra)**

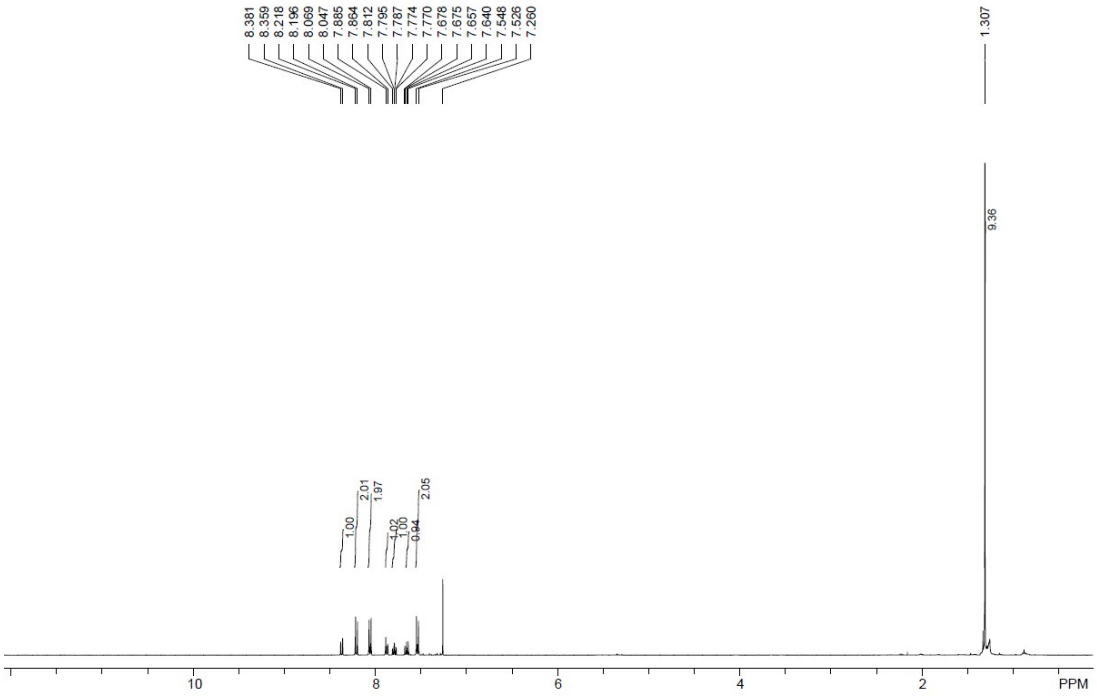


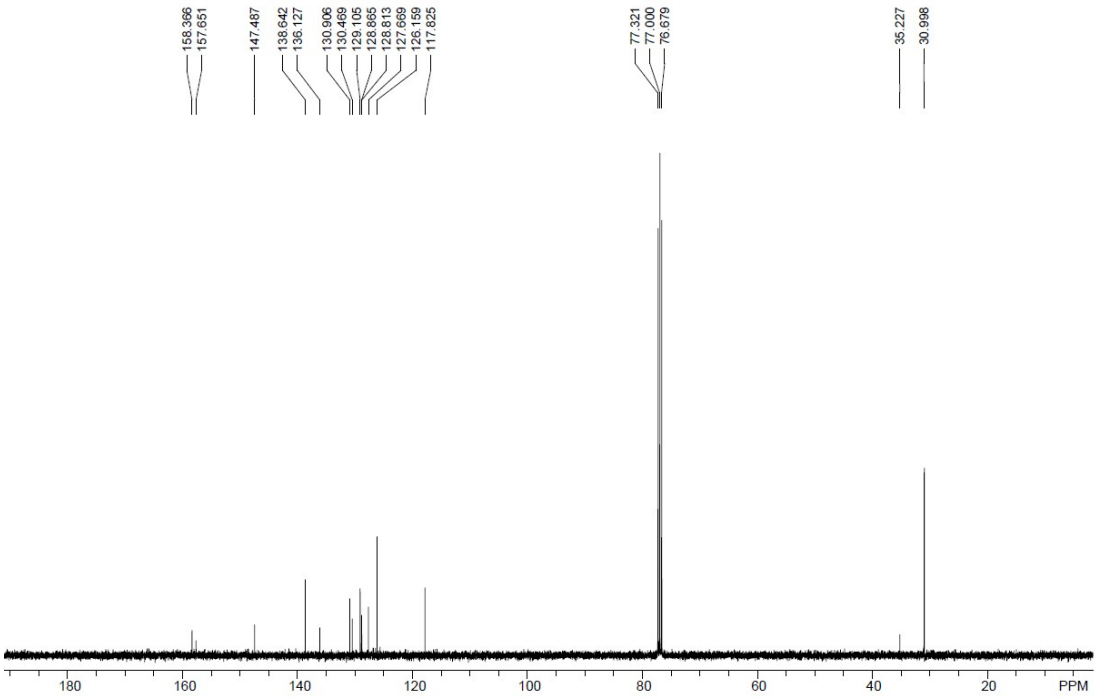


***2-(phenylsulfonyl)quinoline* (3ab)**

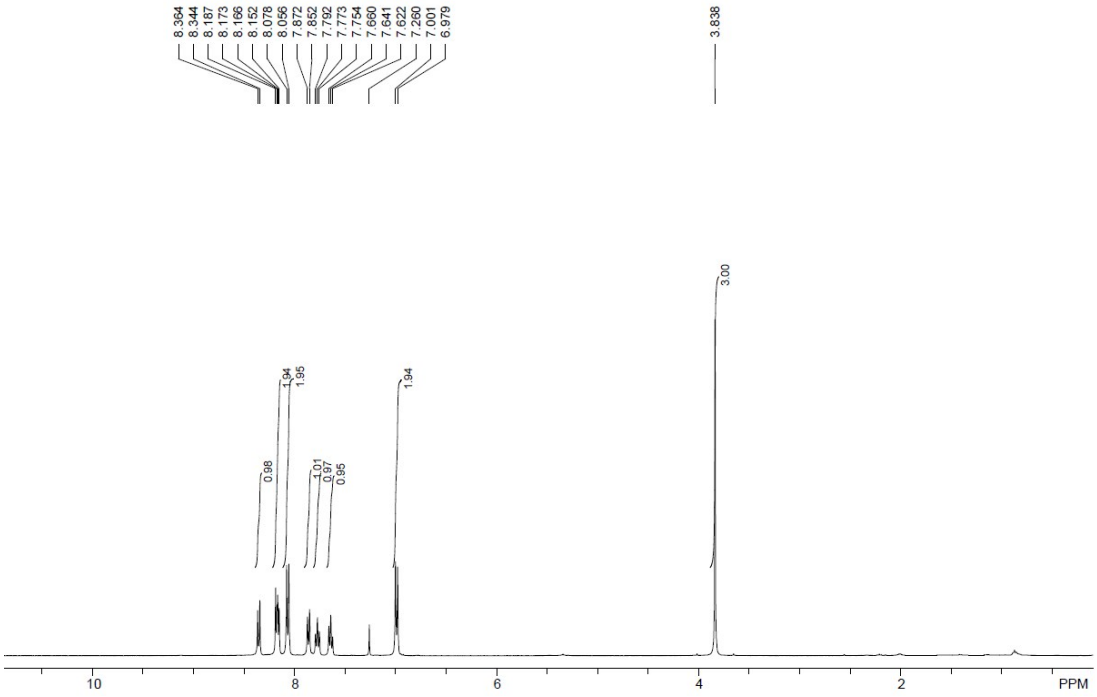


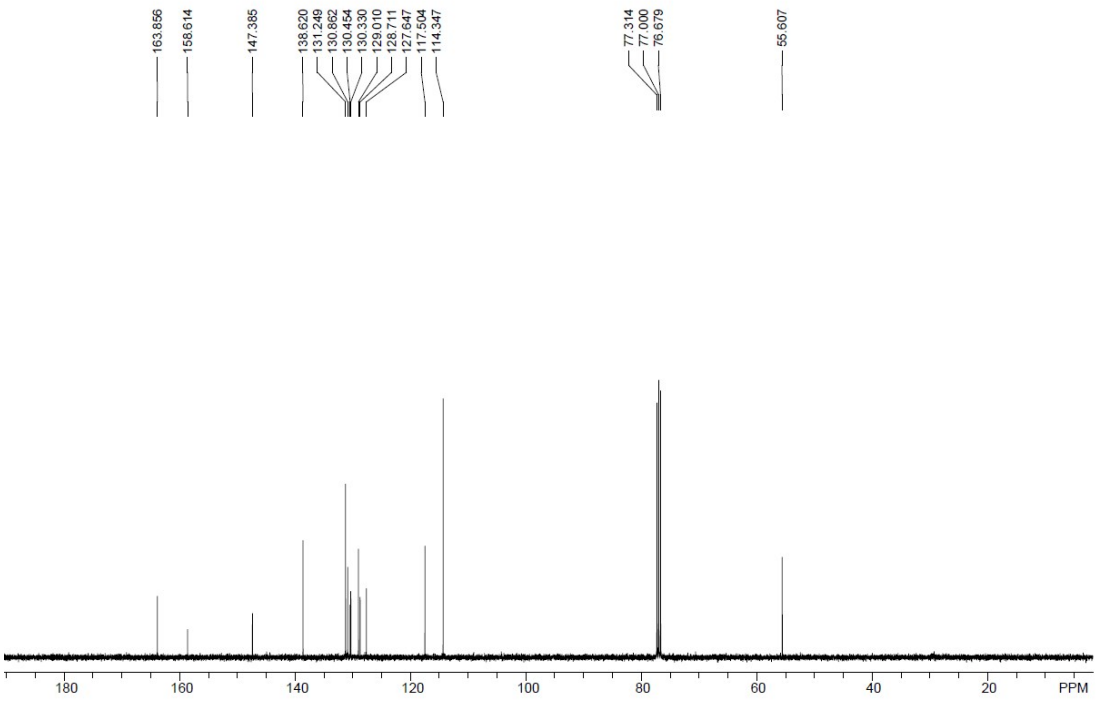
***2-((4-(tert-butyl)phenyl)sulfonyl)quinoline* (3ac)**



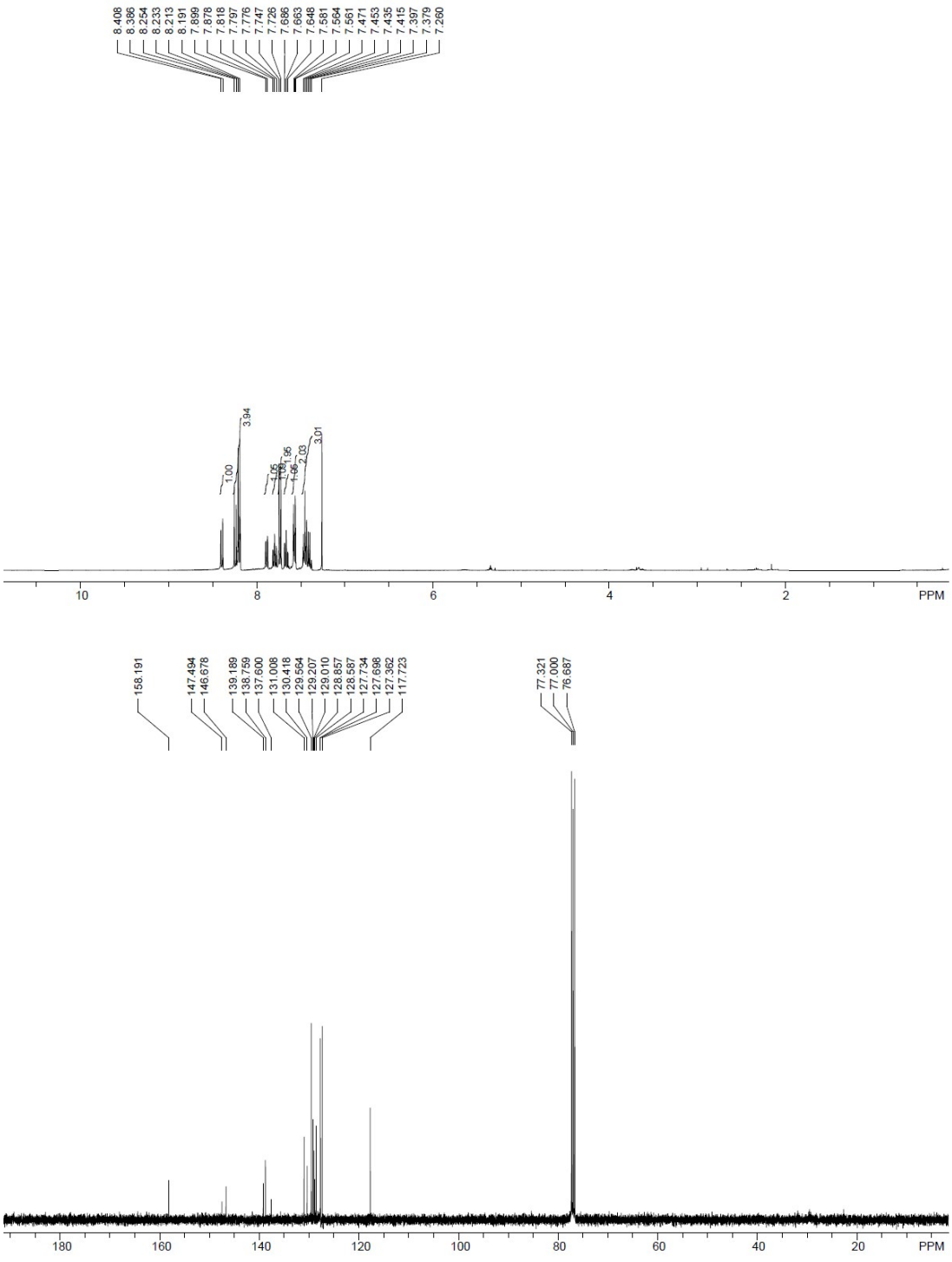


***2-((4-methoxyphenyl)sulfonyl)quinoline* (3ad)**

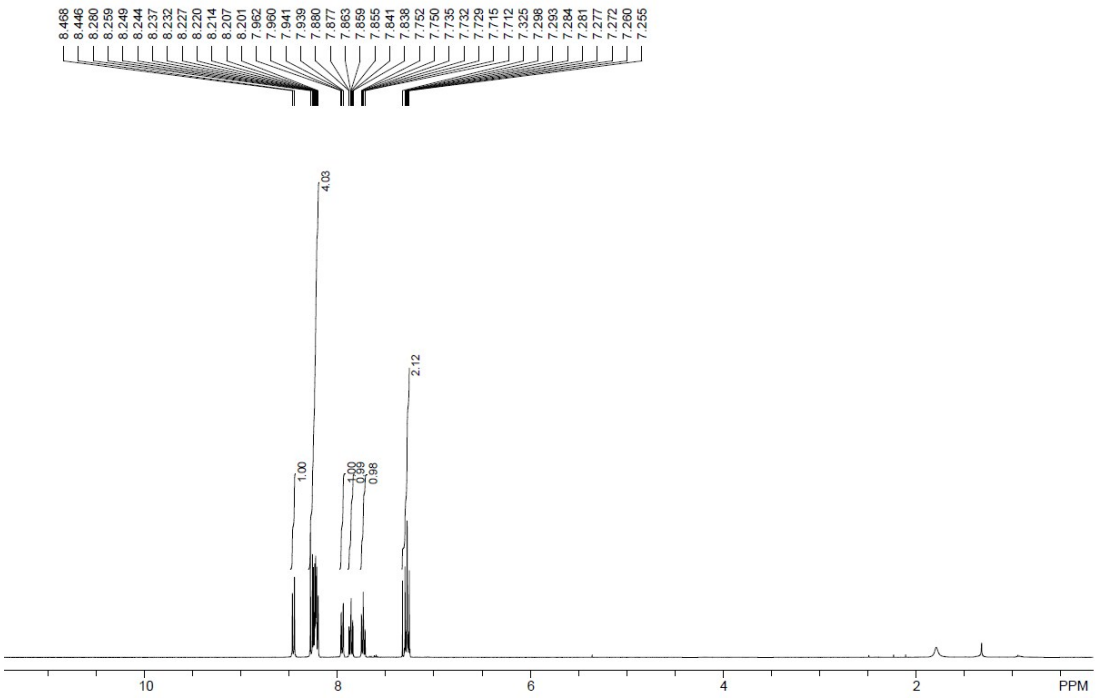


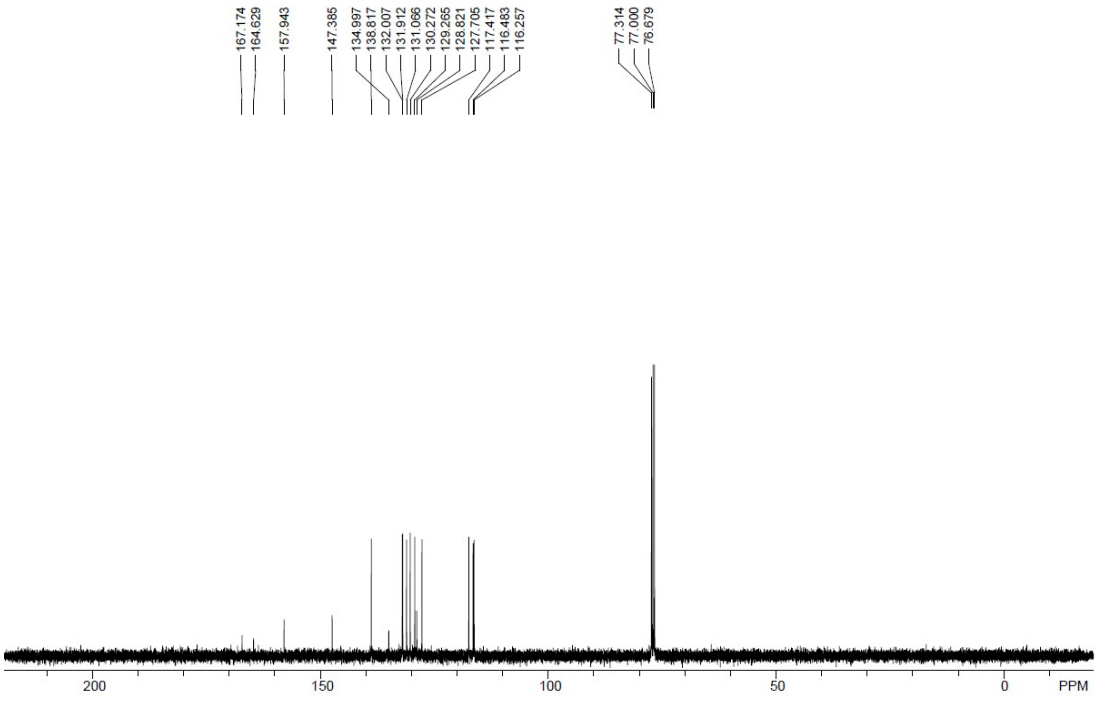


***2-([1,1'-biphenyl]-4-ylsulfonyl)quinolone* (3ae)**

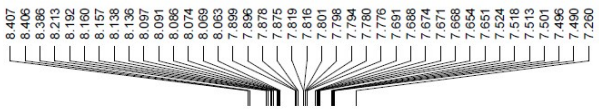


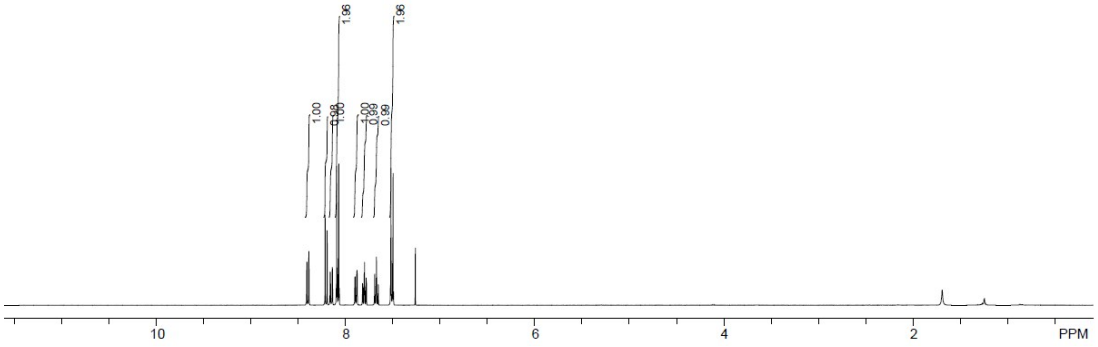
***2-((4-fluorophenyl)sulfonyl)quinoline* (3af)**

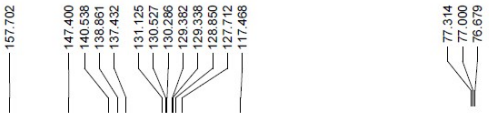


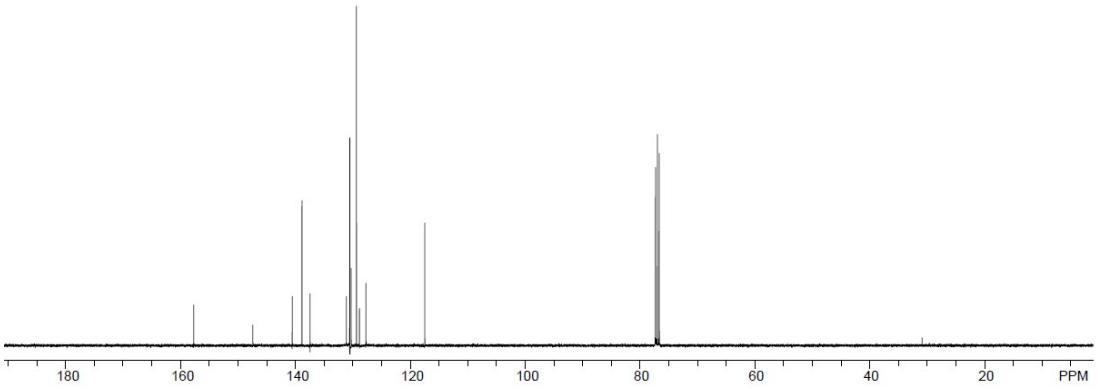


***2-((4-chlorophenyl)sulfonyl)quinoline* (3ag)**

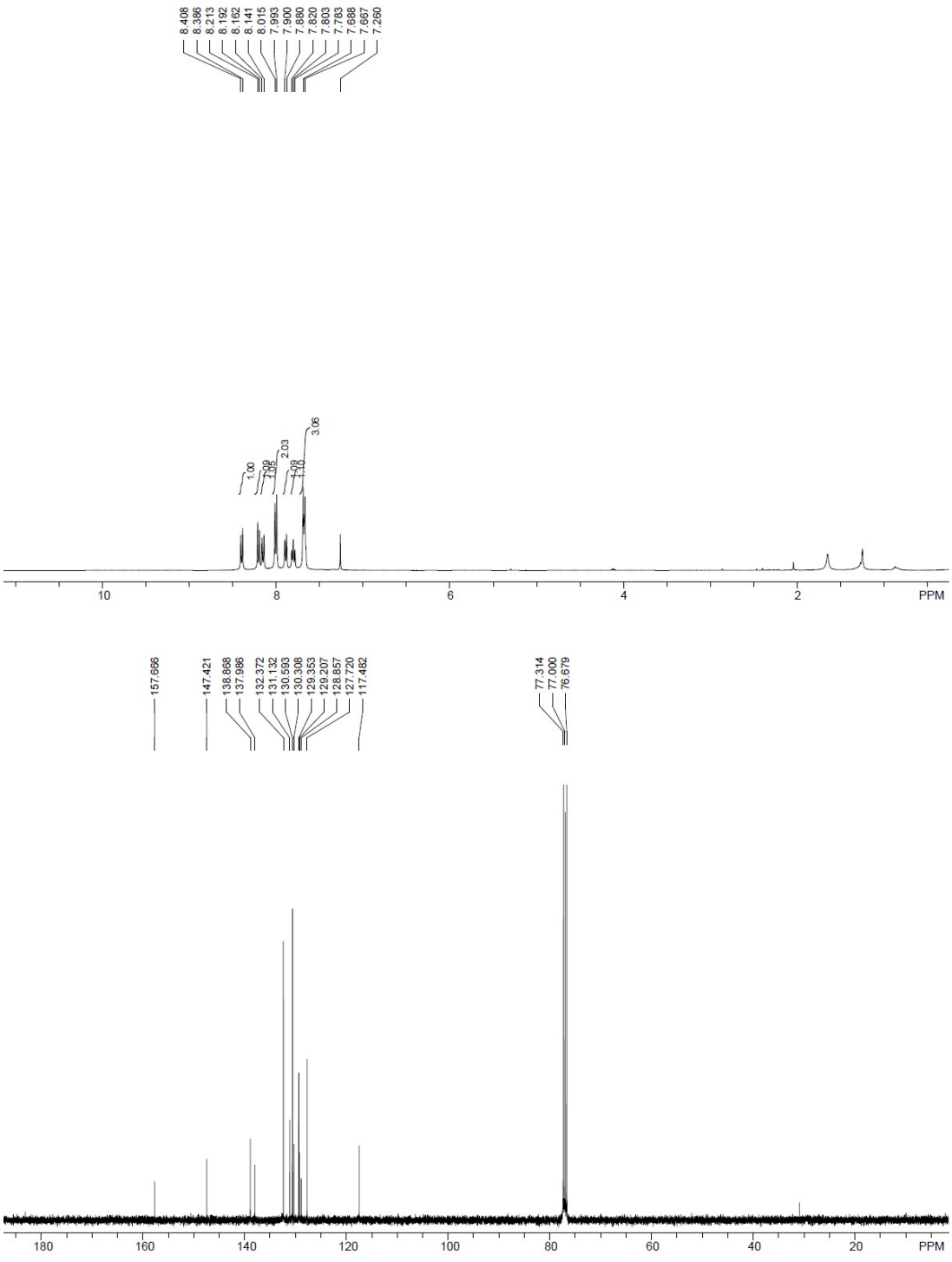




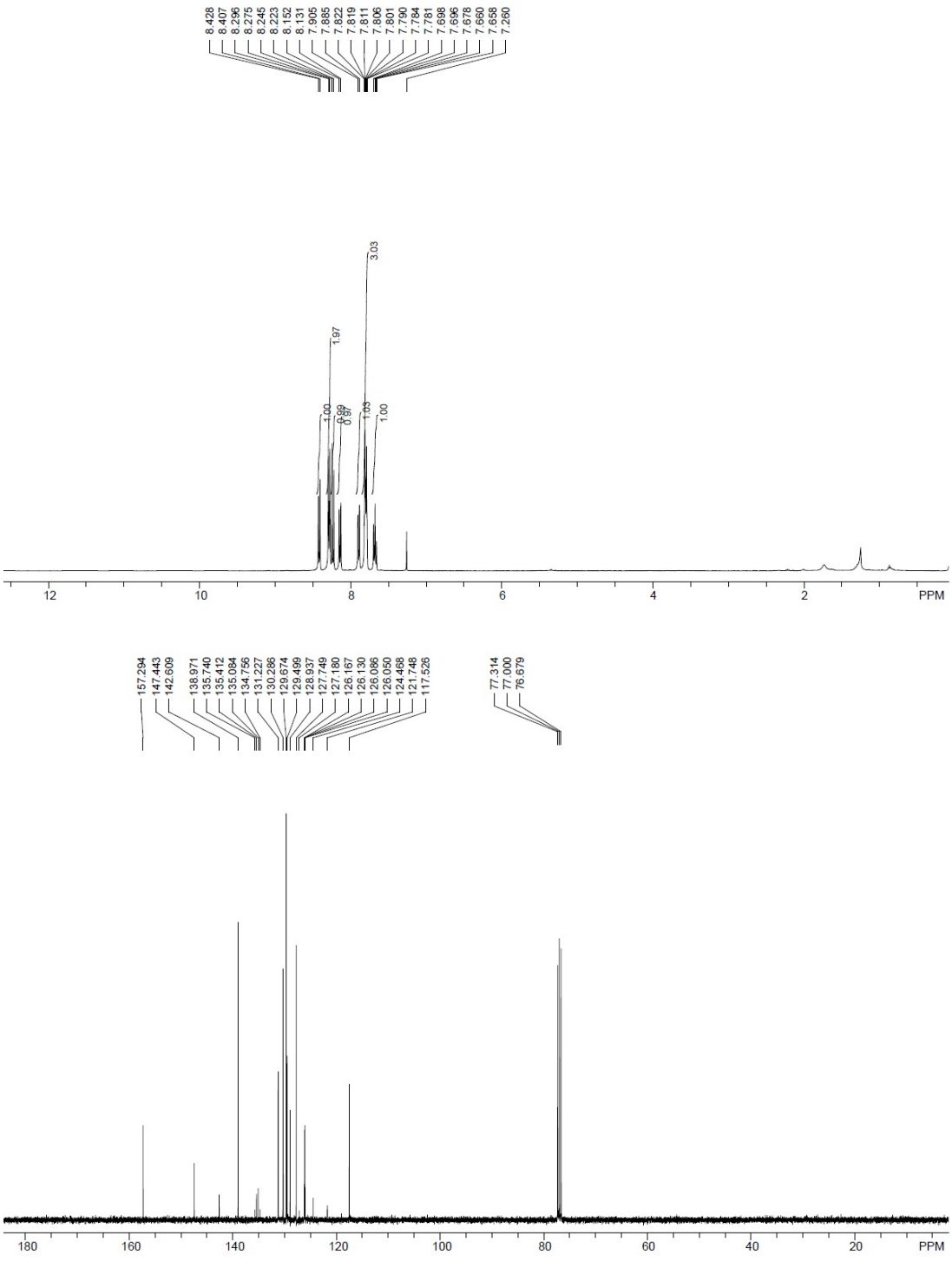




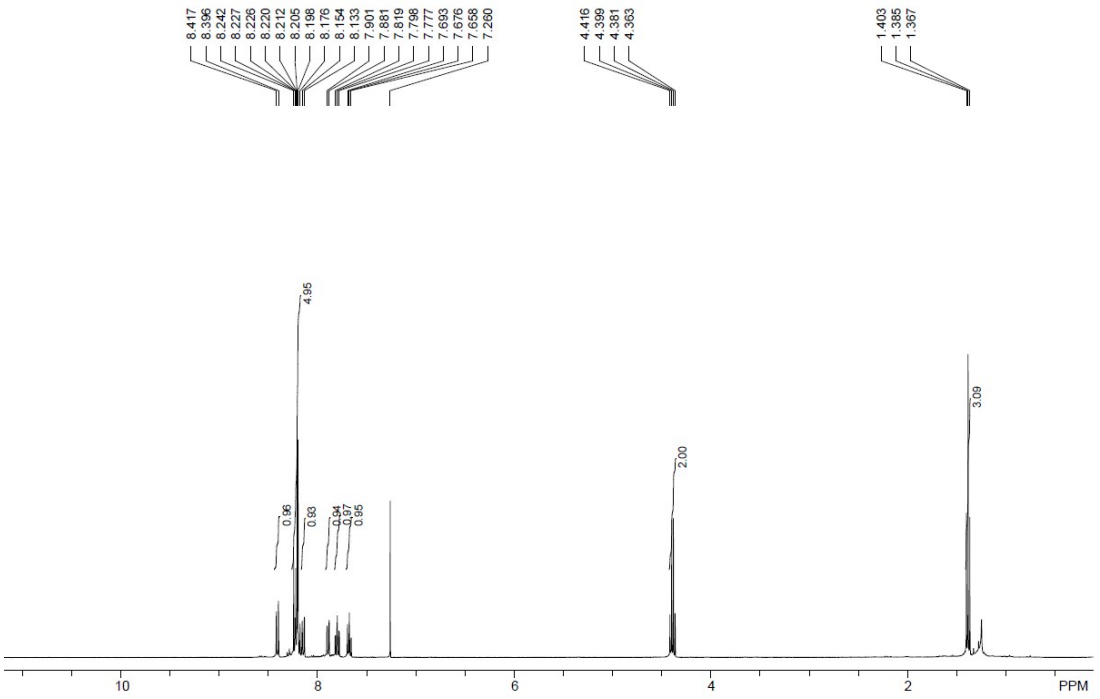
***2-((4-bromophenyl)sulfonyl)quinoline* (3ah)**

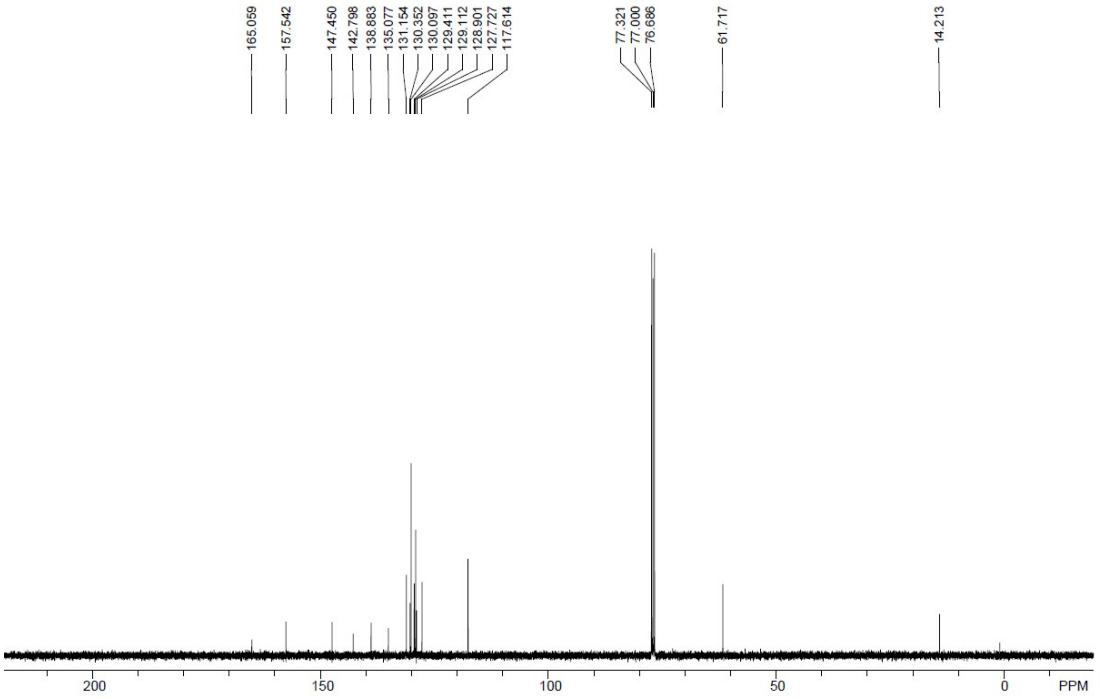


***2-((4-(trifluoromethyl)phenyl)sulfonyl)quinoline* (3ai)**

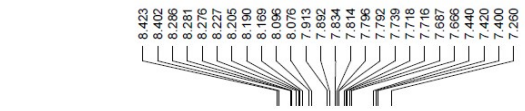


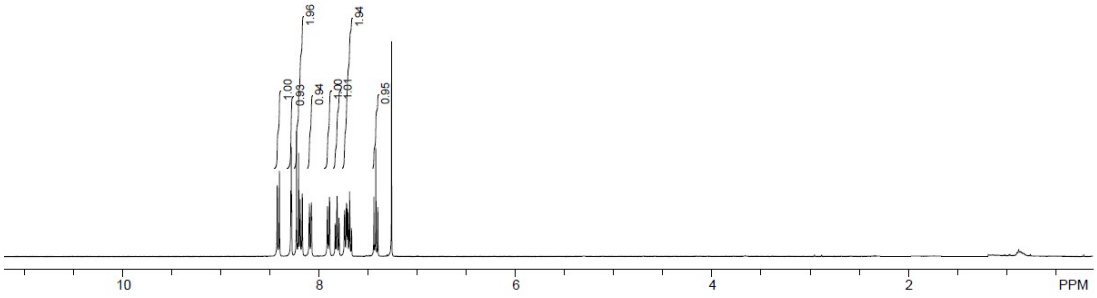
***ethyl 4-(quinolin-2-ylsulfonyl)benzoate* (3aj)**

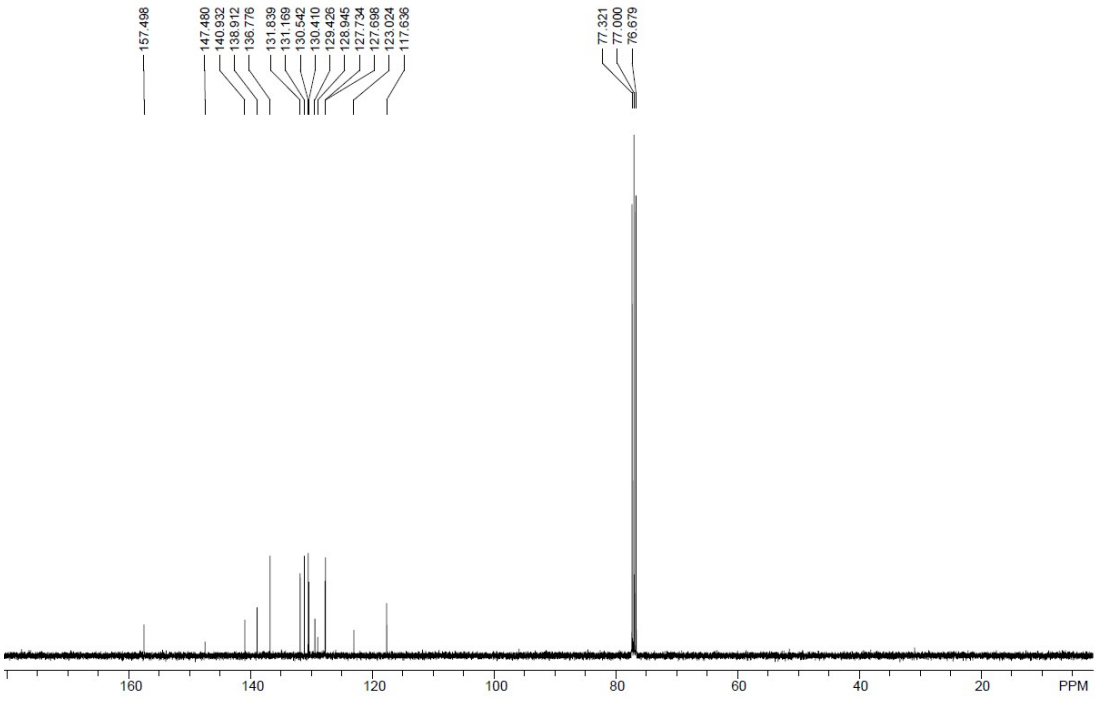




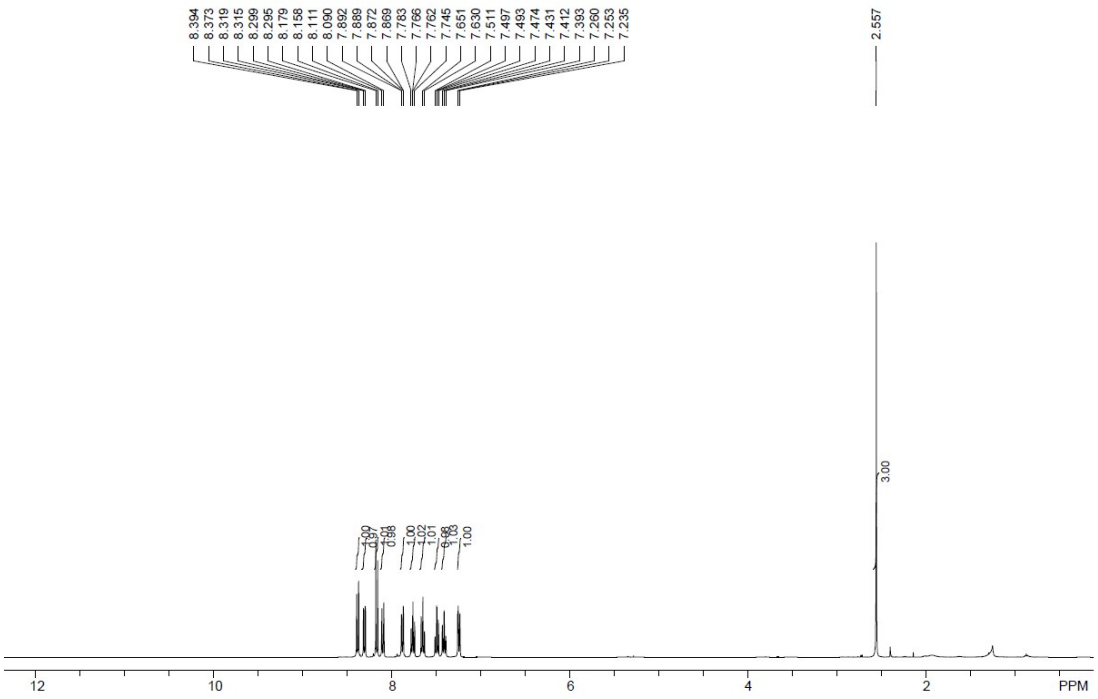
***2-((3-bromophenyl)sulfonyl)quinoline* (3ak)**

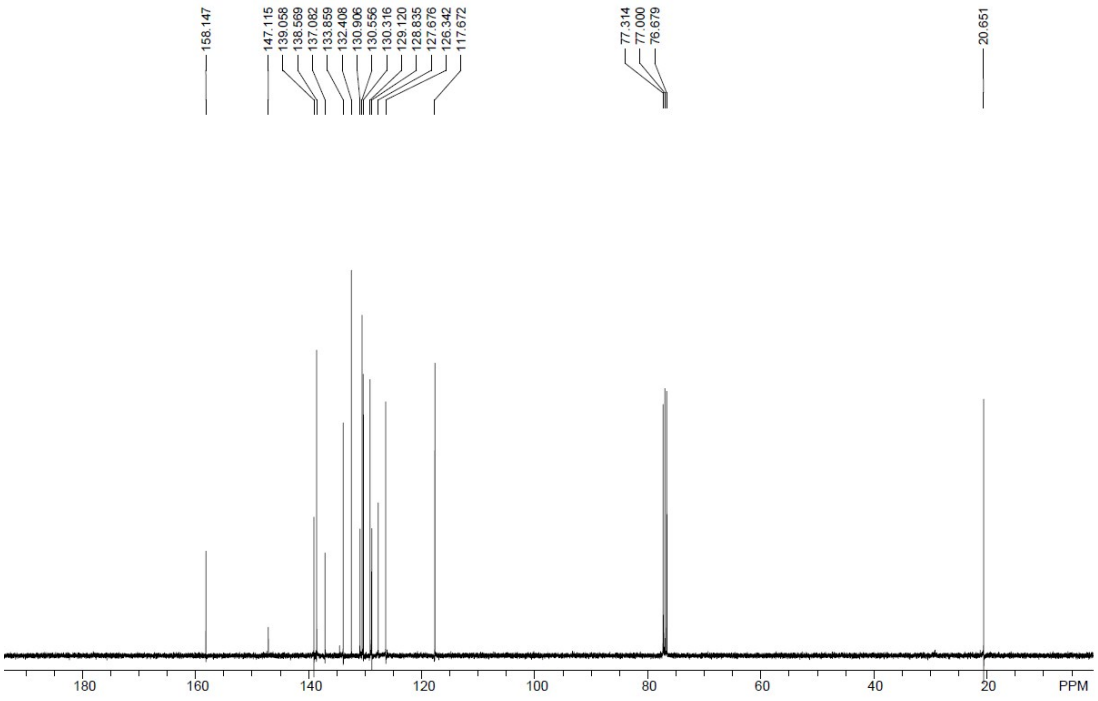




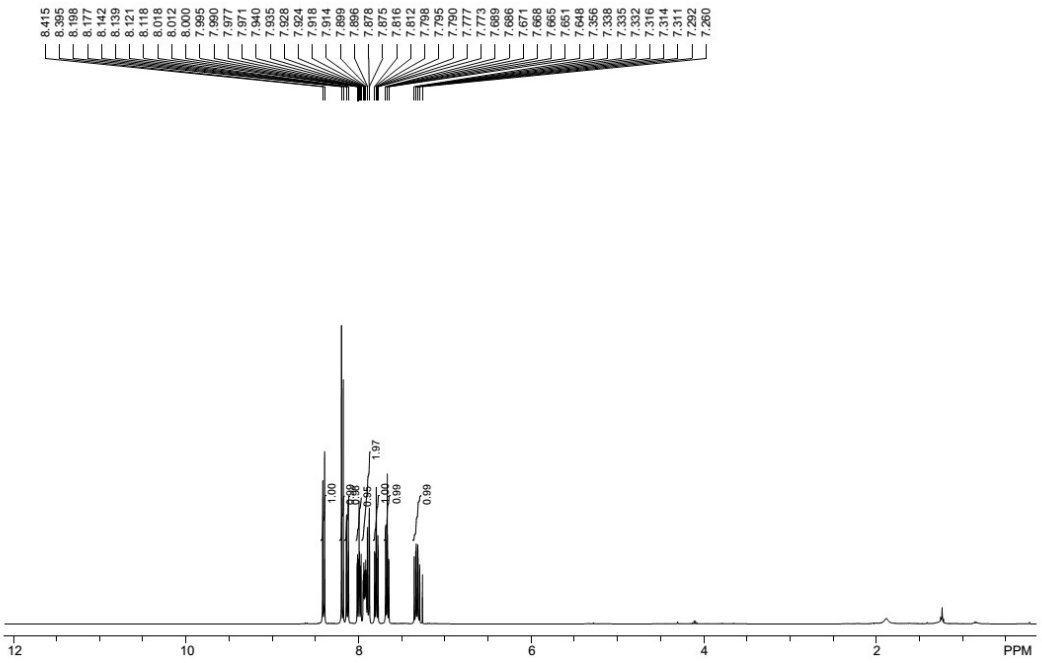


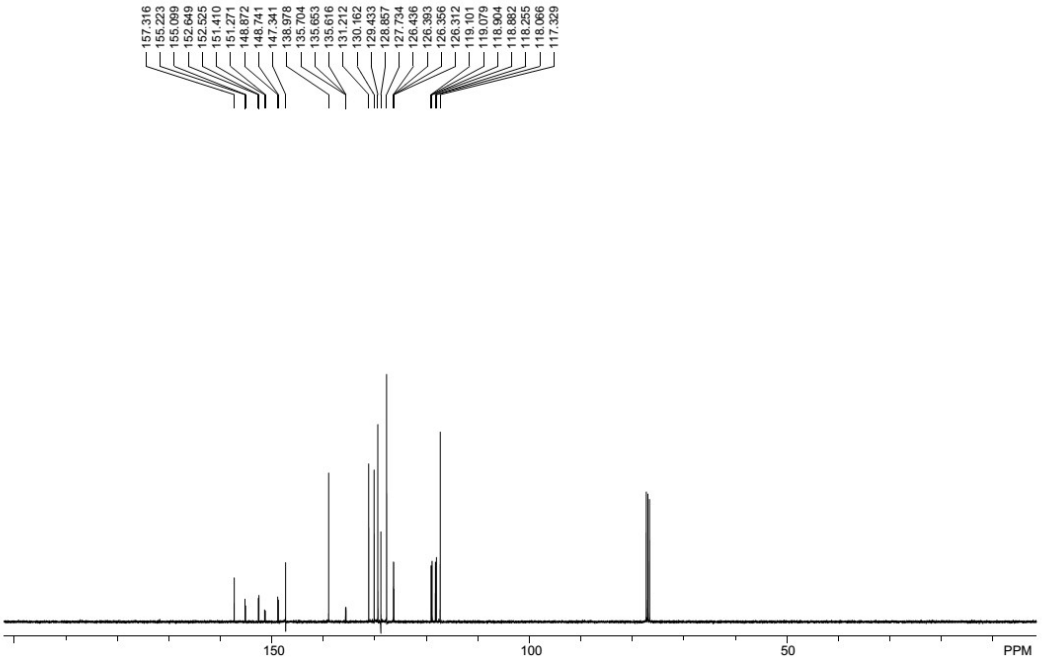
***2-(o-tolylsulfonyl)quinoline* (3al)**



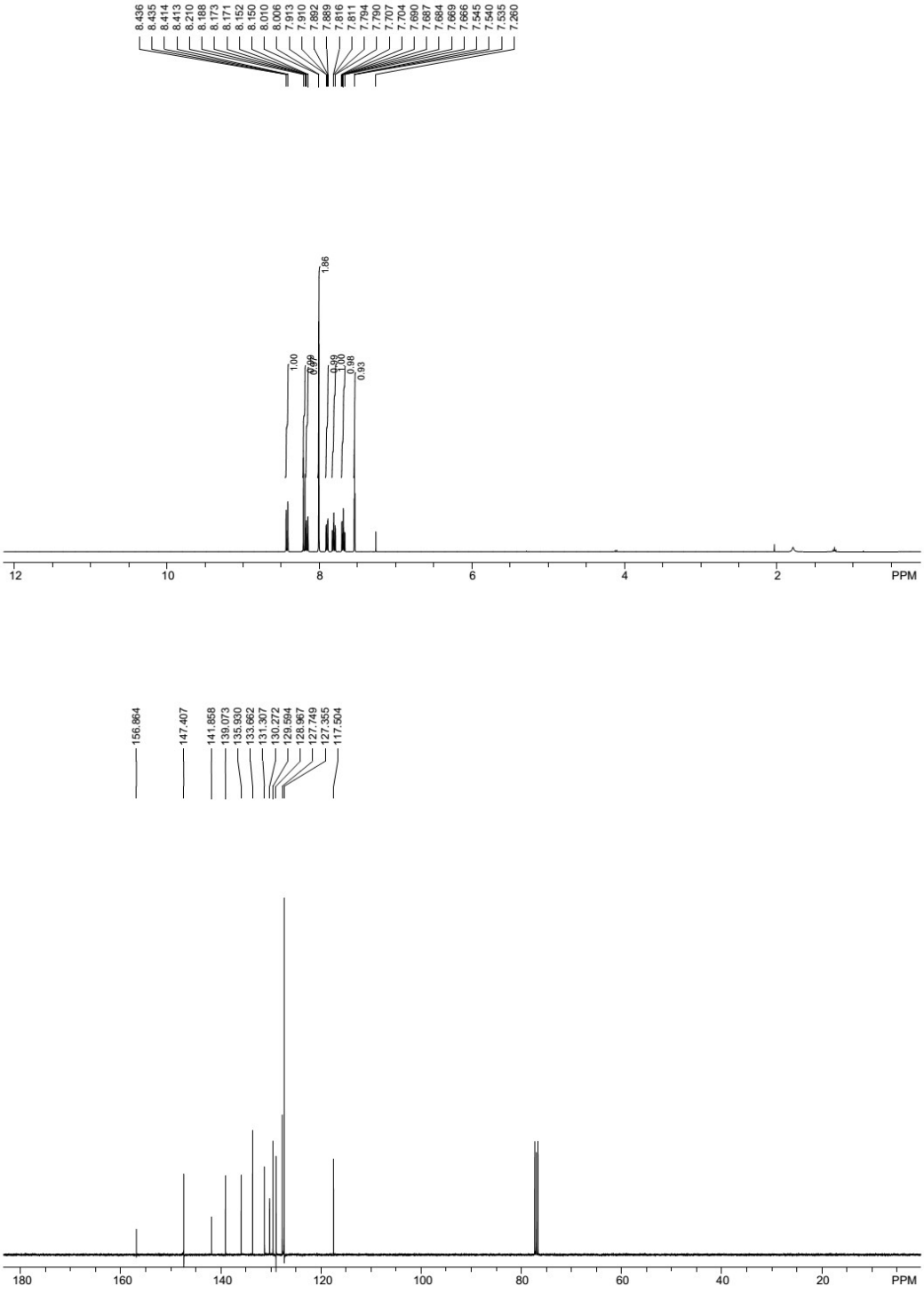


***2-((3,4-difluorophenyl)sulfonyl)quinolone* (3am)**

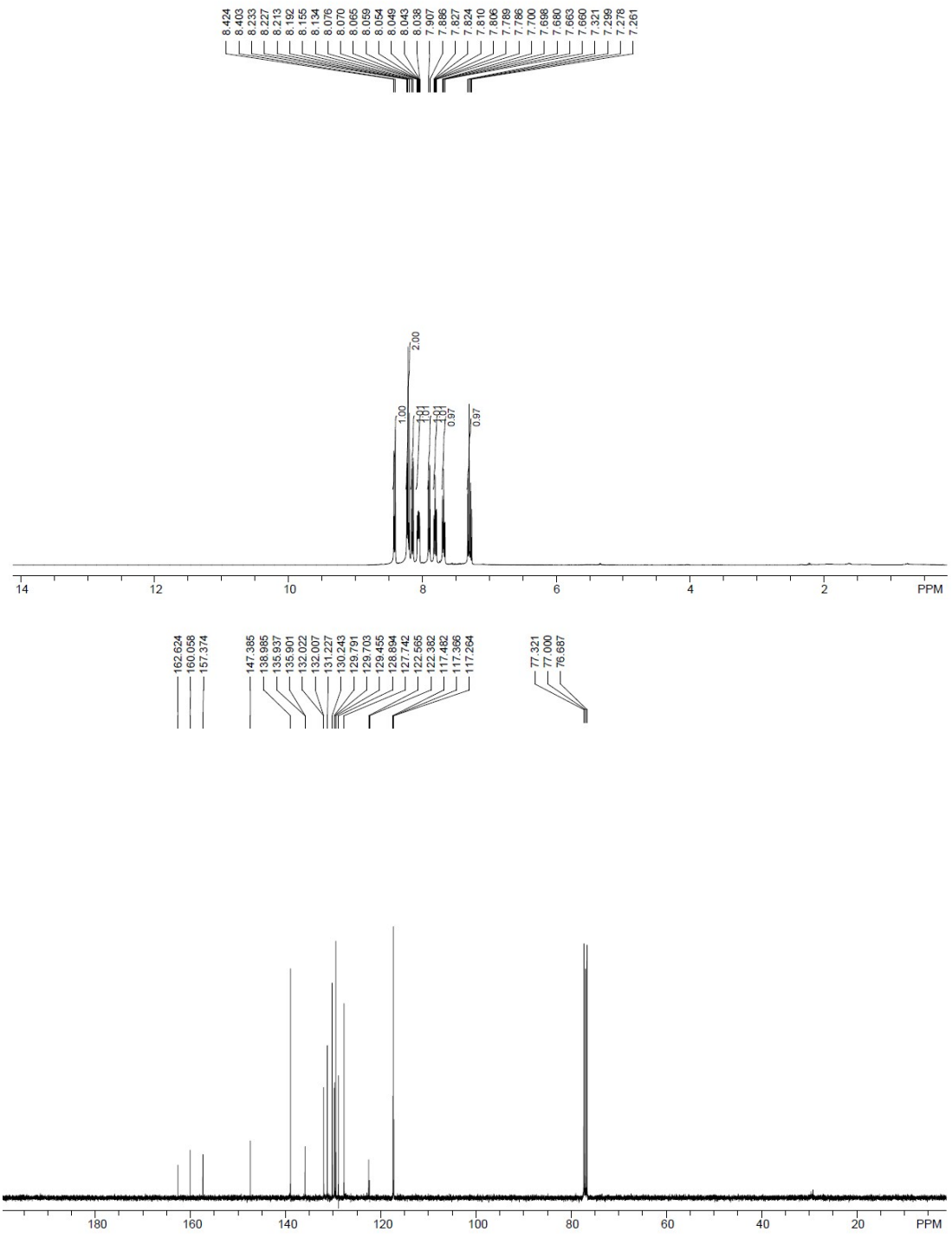




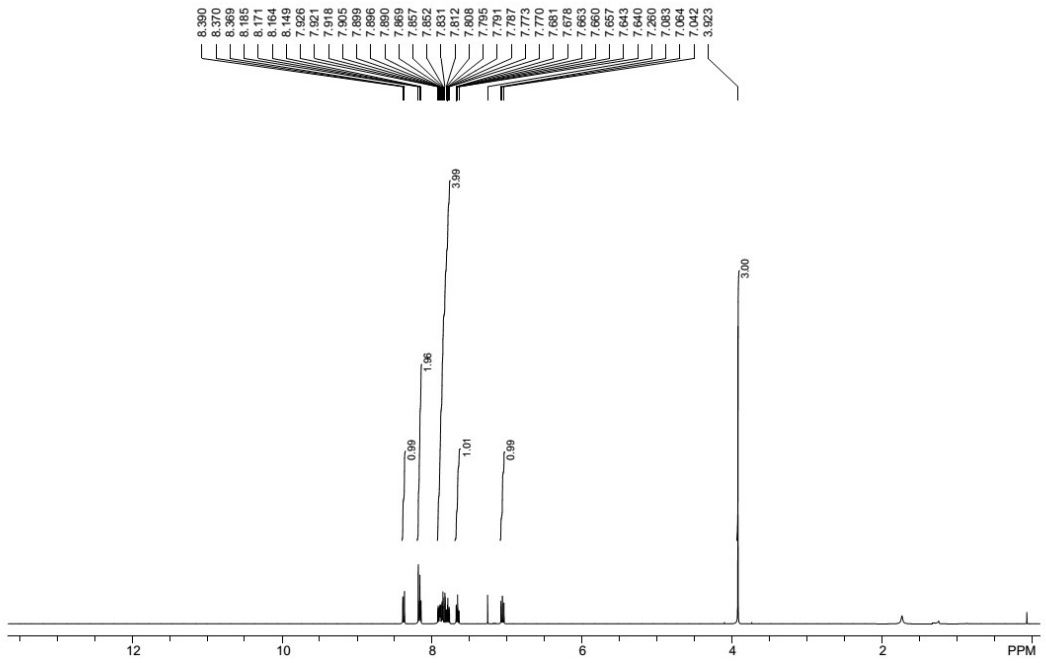
2-((3,5-dichlorophenyl)sulfonyl)quinolone **(3an)**

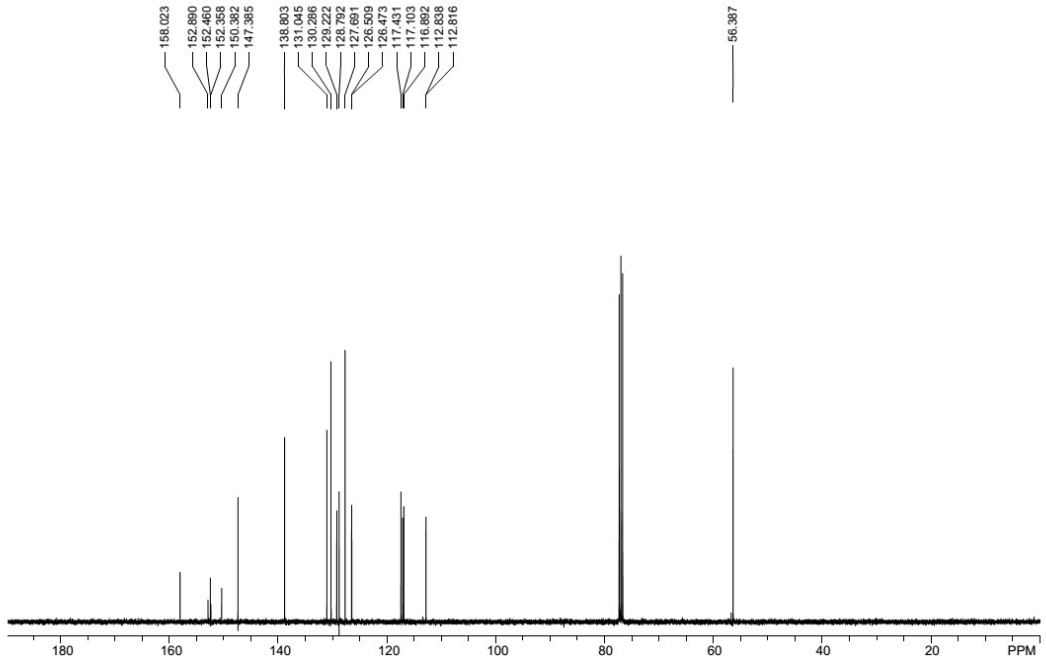


***2-((3-chloro-4-fluorophenyl)sulfonyl)quinoline* (3ao)**

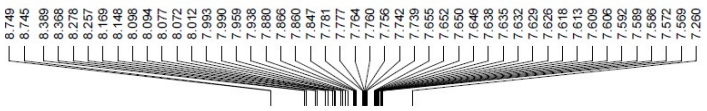


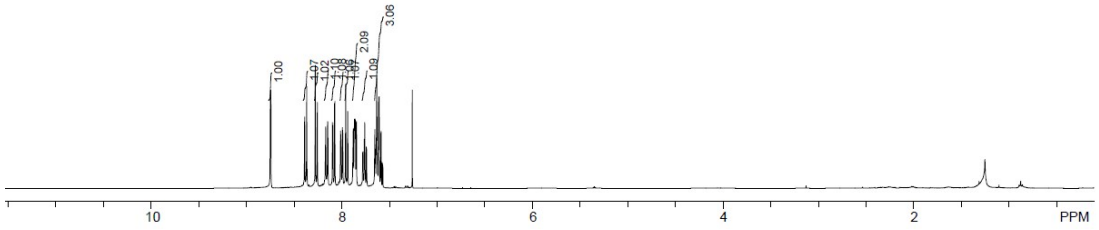
***2-((3-fluoro-4-methoxyphenyl)sulfonyl)quinolone* (3ap)**

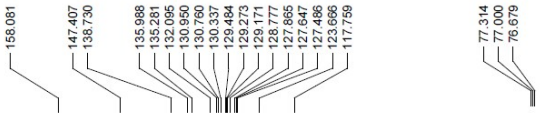


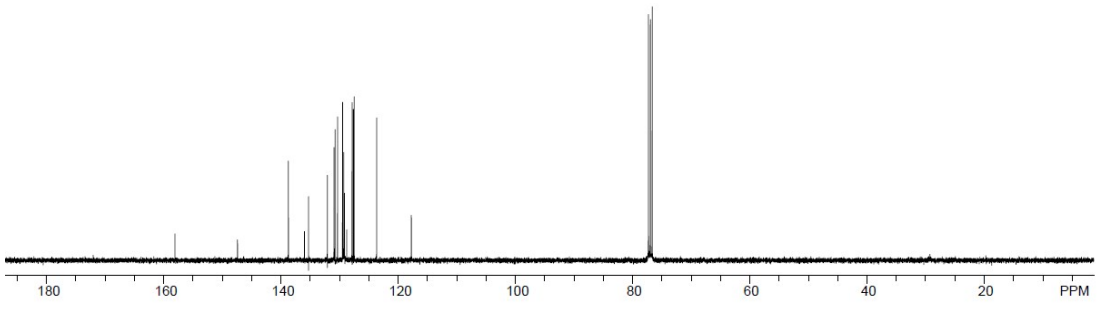


***2-(naphthalen-2-ylsulfonyl)quinoline* (3aq)**









heptan-2-yl 2-((5-chloro-2-tosylquinolin-8-yl)oxy)acetate **(3sa)**