Supplementary Information

Rhodium(I)-Catalyzed Mono-Selective C–H Alkylation of Benzenesulfonamides with Terminal Alkenes

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1. General information

All chemicals were measured and added to a J-Young Schlenk tube or a sealed vial under an atmosphere of air. The reaction vial was then closed and kept in an oil bath. ^{1}H NMR (400 MHz), ^{13}C{^{1}H} NMR (101 MHz), and ^{19}F NMR (376 Hz) spectra were recorded on a JEOL ECS-400 spectrometer in $CDCl_3$ with tetramethylsilane as an internal standard. All ^{1}H NMR chemical shifts were recorded in ppm (δ) and referenced to tetramethylsilane. All ^{13}C{^{1}H} NMR chemical shifts are given in ppm (δ) relative to carbon resonances in $CDCl_3$ at δ 77.16. Data are reported as follows: chemical shifts in ppm (δ), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet), coupling constant (Hz), and integration. Infrared spectra (IR) were obtained using a JASCO FT/IR-4200 spectrometer; absorptions are reported in reciprocal centimetres with the following relative intensities: s (strong), m (medium), or w (weak). High resolution mass spectra (HRMS) were obtained using a JEOL JMS-700 spectrometer and recorded by EI using a double-focusing mass spectrometer. Melting points were determined using a Stanford Research Systems apparatus. Flash column chromatography was performed using SiO_2 F60 (0.040–0.0663 nm, 230-400 mesh). Some compounds were isolated by LC-908 HPLC (GPC) or HPLC (Phenomenex Luna 5u Silica (2) 100 × 21.20 mm column with hexane/EtOAc as an eluent).

2. Materials

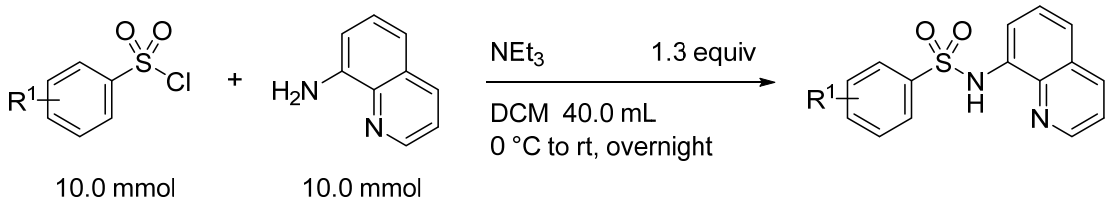
$[Rh(OAc)(cod)]_2$ was prepared from $RhCl_3.H_2O$ by following the literature procedure.[1] Sulfonamide starting materials of Table S1 were synthesized according to the literature procedure.[2] All the chemicals were used as it is received without further purification.

Solvents (DCM, toluene, EtOAc, hexane, and $CDCl_3$) were used without further purification.

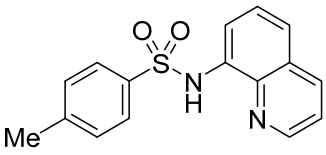
General procedure for the preparation of sulfonamide starting material

In a dry three neck round bottom flask, 8-aminoqunoline (1.441 g, 10.0 mmol, 1.0 equiv) and triethylamine (1.8 mL, 13.0 mmol, 1.3 equiv) was dissolved in CH_2Cl_2 (25 mL). After cooling the reaction mixture to 0 °C, suitable sulfonyl chloride (10.0 mmol, 1.0 equiv) in 15 mL of CH_2Cl_2 was added portion wise about 10 minutes of period. After that, the resulting mixture was allowed to warm up to room temperature and stirred for overnight. After completion of the reaction, the crude mixture was washed with saturated aqueous $NaHCO_3$ (20 mL) and brine solution (20 mL). The aqueous layer was washed with 3 x 20 mL of CH_2Cl_2. Then the organic phase was dried over anhydrous Na_2SO_4 and the volatiles were evaporated to dryness. The resulting crude mixture was purified by flash chromatography on silica gel (eluent: hexane/EtOAc/acetone = 10/3/1).

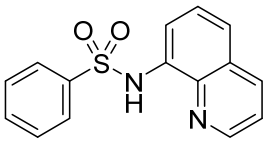
Table S1. Synthesized sulfonamide starting materials



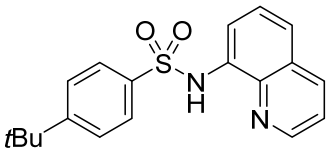
10.0 mmol 10.0 mmol



1a 85%



1b 74%



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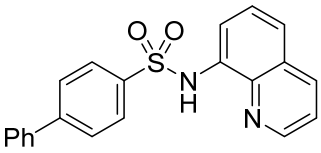
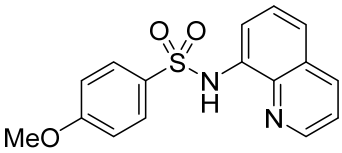
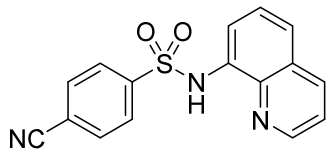


1d 70%



1e 90%



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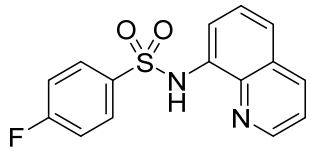
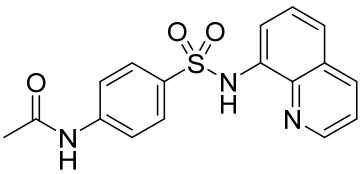
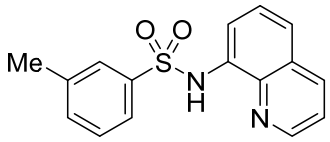


1g 77%



1h 91%



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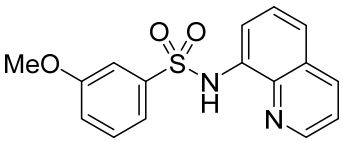

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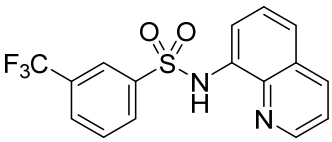

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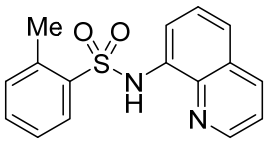

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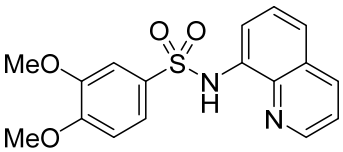

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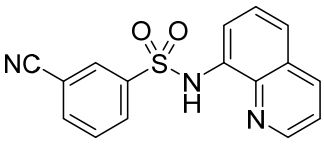
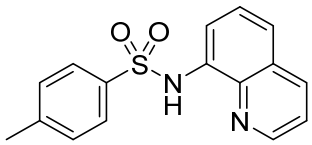


1n 68%

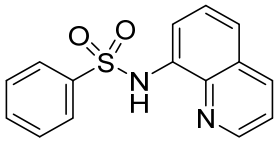
3. Spectral data of starting materials

4-methyl-*N*-(quinolin-8-yl)benzenesulfonamide (1a)



Yield – 2.531 g, 85%. R_f - 0.17 (hexane/EtOAc = 5/1). White solid. MP – 156 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.22 (s, 1H), 8.75 (d, *J* = 4.1 Hz, 1H), 8.08 (d, *J* = 8.3 Hz, 1H), 7.86 – 7.75 (m, 3H), 7.49 – 7.36 (m, 3H), 7.14 (d, *J* = 8.0 Hz, 2H), 2.28 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 148.7, 143.8, 138.5, 136.5, 136.3, 133.9, 129.6, 128.3, 127.3, 126.9, 122.1, 122.0, 114.9, 21.5. IR (neat, ν/cm^{-1}) 3261 m, 3050 w, 2923 w, 2854 w, 1503 s, 1470 m, 1366 s, 1305 s, 1159 s, 1087 s, 920 m, 825 m, 791 s, 759 m, 662 s. HRMS (EI$^+$) *m/z*: $[M]^+$ Calcd for $C_{16}H_{14}N_2O_2S$ 298.0776; found 298.0774.

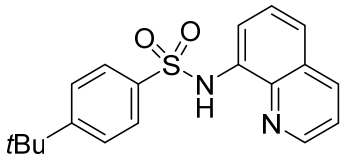
N-(quinolin-8-yl)benzenesulfonamide (1b)



Yield – 2.115 g, 74%. R_f - 0.31 (hexane/EtOAc = 10/3). White solid. MP – 134 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.25 (s, 1H), 8.74 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.07 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.97 – 7.87 (m, 2H), 7.83 (dd, *J* = 6.5, 2.3 Hz, 1H), 7.49 – 7.30 (m, 6H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 148.8, 139.4, 138.5, 136.3, 133.7, 133.0, 128.9,

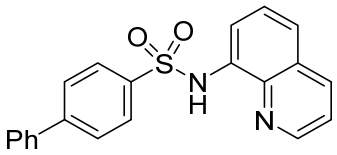
128.2, 127.2, 126.9, 122.3, 122.0, 115.1. IR (neat, ν/cm^{-1}) 3216 w, 3069 w, 2927 w, 1503 m, 1470 w, 1356 m, 1306 m, 1164 s, 1089 m, 906 s, 786 m, 727 s, 685 s. HRMS (EI$^+$) *m/z*: $[M]^+$ Calcd for $C_{15}H_{12}N_2O_2S$ 284.0619; found 284.0621.

4-(*tert*-butyl)-*N*-(quinolin-8-yl)benzenesulfonamide (1c)



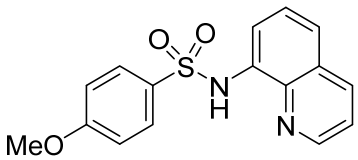
Yield – 2.456 g, 72%. R_f - 0.24 (hexane/EtOAc = 5/1). White crystalline solid. MP – 174 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.24 (s, 1H), 8.75 (dd, *J* = 4.1, 1.5 Hz, 1H), 8.09 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.89 – 7.76 (m, 3H), 7.49 – 7.33 (m, 5H), 1.23 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 156.7, 148.7, 138.5, 136.5, 136.3, 134.0, 128.2, 127.1, 127.0, 126.0, 122.0, 121.9, 114.7, 35.1, 31.0. IR (neat, ν/cm^{-1}) 3208 m, 3062 w, 2961 m, 2904 w, 2968 w, 1503 s, 1469 m, 1413 m, 1333 s, 1167 s, 1085 s, 1057 m, 919 s, 824 m, 789 s, 755 s, 672 s. HRMS (EI$^+$) *m/z*: $[M]^+$ Calcd for $C_{19}H_{20}N_2O_2S$ 340.1245; found 340.1244.

***N*-(quinolin-8-yl)-[1,1'-biphenyl]-4-sulfonamide (1d)**



Yield – 2.528 g, 70%. R_f - 0.17 (hexane/EtOAc = 5/1). White crystaline solid. MP – 158 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.29 (s, 1H), 8.76 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.09 (dd, *J* = 8.3, 1.6 Hz, 1H), 8.00 – 7.92 (m, 2H), 7.87 (dd, *J* = 5.4, 3.5 Hz, 1H), 7.59 – 7.51 (m, 2H), 7.50 – 7.30 (m, 8H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 148.8, 145.8, 139.2, 138.6, 138.0, 136.4, 133.8, 129.0, 128.5, 128.3, 127.8, 127.6, 127.3, 127.0, 122.3, 122.1, 115.2. IR (neat, ν/cm^{-1}) 3259 w, 3065 w, 3032 w, 1594 m, 1503 s, 1370 s, 1308 s, 1163 s, 1090 s, 921 m, 792 m, 759 s, 695 m, 673 s. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{21}H_{16}N_2O_2S$ 360.0932; found 360.0929.

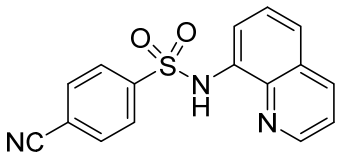
4-methoxy-*N*-(quinolin-8-yl)benzenesulfonamide (1e)



Yield – 2.830 g, 90%. R_f - 0.14 (hexane/EtOAc = 5/1). White solid. MP – 93 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.19 (s, 1H), 8.75 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.08 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.88 – 7.75 (m, 3H), 7.49 – 7.36 (m, 3H), 6.85 – 6.76 (m, 2H), 3.74 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 163.1, 148.7, 138.6, 136.4, 134.0, 131.1, 129.5, 128.3, 126.9, 122.1, 122.0, 115.0, 114.1, 55.6. IR (neat, ν/cm^{-1}) 3260 w, 3069 w, 3012 w,

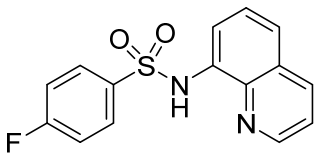
2944 w, 1595 s, 1578 m, 1501 s, 1413 m, 1367 s, 1306 s, 1259 s, 1155 s, 1089 s, 1025 m, 919 m, 826 s, 792 s, 758 m, 667 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{16}H_{14}N_2O_3S$ 314.0725; found 314.0730.

4-cyano-*N*-(quinolin-8-yl)benzenesulfonamide (1f)



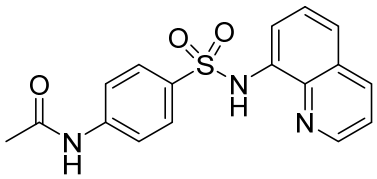
Yield – 2.165 g, 70%. R_f - 0.26 (hexane/EtOAc = 1/1). White solid. MP – 129 °C. ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.29 (s, 1H), 8.75 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.13 (dd, *J* = 8.3, 1.5 Hz, 1H), 8.03 – 7.95 (m, 2H), 7.86 (dd, *J* = 7.4, 1.3 Hz, 1H), 7.69 – 7.57 (m, 2H), 7.56 – 7.40 (m, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 149.1, 143.5, 138.6, 136.6, 132.9, 132.8, 128.4, 127.9, 126.9, 123.3, 122.3, 117.3, 116.7, 116.1. IR (neat, ν/cm^{-1}) 3252 w, 3095 w, 3070 w, 3044 w, 2234 m, 1503 s, 1470 m, 1413 s, 1374 s, 1309 s, 1165 s, 1089 s, 1059 w, 925 m, 826 s, 792 s, 756 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{16}H_{11}N_3O_2S$ 309.0572; found 309.0568.

4-fluoro-*N*-(quinolin-8-yl)benzenesulfonamide (1g)



Yield – 2.322 g, 77%. R_f - 0.31 (hexane/EtOAc = 5/1). White solid. MP – 139 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.22 (s, 1H), 8.75 (dd, *J* = 4.0, 0.8 Hz, 1H), 8.11 (dd, *J* = 8.2, 0.8 Hz, 1H), 7.97 – 7.88 (m, 2H), 7.88 – 7.79 (m, 1H), 7.53 – 7.39 (m, 3H), 7.08 – 6.97 (m, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 165.2 (d, *J* = 255.1 Hz), 148.9, 138.6, 136.4, 135.4 (d, *J* = 3.2 Hz), 133.6, 130.0 (d, *J* = 9.5 Hz), 128.3, 126.9, 122.6, 122.2, 116.2 (d, *J* = 22.5 Hz), 115.51. ^{19}F NMR (376 MHz, $CDCl_3$, 30 °C) δ -104.67. IR (neat, ν/cm^{-1}) 3259 w, 3106 w, 3071 w, 1592 m, 1503 s, 1413 m, 1372 s, 1236 m, 1170 s, 1156 s, 1089 s, 923 m, 839 s, 824 s, 792 s, 758 m, 667 m. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{15}H_{11}FN_2O_2S$ 302.0525; found 302.0531.

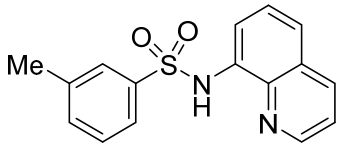
_N_-(4-(_N_-(quinolin-8-yl)sulfamoyl)phenyl)acetamide (1h)



Yield – 3.108 g, 91%. R_f - 0.03 (hexane/EtOAc = 5/1). White solid. MP – 194 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.23 (s, 1H), 8.75 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.10 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.87 – 7.77 (m, 3H), 7.55 – 7.32 (m, 6H), 2.13 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 168.6, 148.8, 142.1, 138.6, 136.4, 134.1, 133.7, 128.7, 128.3, 126.9, 122.3, 122.1, 119.1, 115.2, 24.8. IR (neat, ν/cm^{-1}) 3359 w, 3242 w, 3187 w, 3109 w, 3054

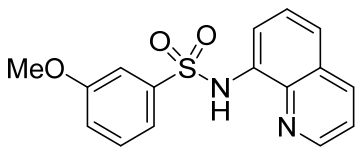
w, 2925 w, 1682 s, 1592 s, 1528 s, 1503 s, 1311 s, 1159 s, 1089 s, 921 m, 826 s, 791 s, 749 m, 728 s. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{17}H_{15}N_3O_3S$ 341.0834; found 341.0839.

3-methyl-*N*-(quinolin-8-yl)benzenesulfonamide (1i)



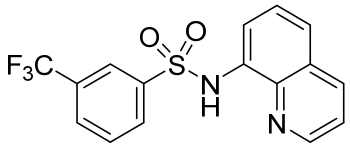
Yield – 2.838 g, 95%. R_f - 0.34 (hexane/EtOAc = 5/1). White solid. MP –94 °C. ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.24 (s, 1H), 8.79 – 8.72 (m, 1H), 8.13 – 8.04 (m, 1H), 7.81 (dd, *J* = 6.1, 2.7 Hz, 1H), 7.77 – 7.66 (m, 2H), 7.49 – 7.36 (m, 3H), 7.25 – 7.20 (m, 2H), 2.29 (s, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 148.7, 139.29, 139.22, 138.5, 136.3, 133.8, 128.8, 128.2, 127.6, 126.9, 124.4, 122.1, 122.0, 115.0, 21.3. IR (neat, ν/cm^{-1}) 3259 w, 3060 w, 2960 w, 2922 w, 1503 s, 1412 m, 1368 s, 1334 m, 1307 s, 1156 s, 1087 s, 922 m, 824 m, 789 s, 756 m, 687 s. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{16}H_{14}N_2O_2S$ 298.0776; found 298.0773.

3-methoxy-*N*-(quinolin-8-yl)benzenesulfonamide (1j)



Yield – 3.110 g, 99%. R_f - 0.11 (hexane/EtOAc = 5/1). White solid. MP – 105 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.24 (s, 1H), 8.79 – 8.69 (m, 1H), 8.09 (d, *J* = 8.3 Hz, 1H), 7.84 (d, *J* = 6.8 Hz, 1H), 7.54 – 7.34 (m, 5H), 7.28 – 7.19 (m, 1H), 7.00 – 6.88 (m, 1H), 3.71 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 159.7, 148.8, 140.5, 138.6, 136.4, 133.8, 130.0, 128.2, 126.9, 122.3, 122.1, 119.58, 119.53, 115.3, 111.7, 55.6. IR (neat, ν/cm^{-1}) 3253 w, 3070 w, 3009 w, 2938 w, 1503 s, 1470 s, 1370 s, 1308 s, 1243 s, 1159 s, 1088 s, 1037 m, 825 s, 790 s, 758 s, 687 s. HRMS (EI$^+$) *m/z*: $[M]^+$ Calcd for $C_{16}H_{14}N_2O_3S$ 314.0725; found 314.0726.

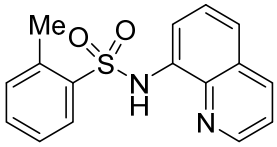
_N_-(quinolin-8-yl)-3-(trifluoromethyl)benzenesulfonamide (1k)



*N-(Quinolin-8-yl)-3-(trifluoromethyl)benzene-sulfonamide (**1k**).* Yield – 2.818 g, 80%. R_f - 0.34 (hexane/EtOAc = 5/1). White solid. MP – 65 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.25 (s, 1H), 8.75 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.16 (s, 1H), 8.11 (dd, *J* = 8.3, 1.5 Hz, 1H), 8.05 (d, *J* = 7.9 Hz, 1H), 7.86 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.55 – 7.37 (m, 4H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 149.0, 140.5, 138.8, 136.4, 133.2, 131.5 (q, *J* = 33.6 Hz), 130.4, 129.7, 129.6 (q, *J* = 3.5 Hz), 128.3, 126.9, 124.5 (q, *J* = 3.5

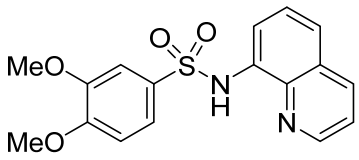
Hz), 123.19, 123.15 (q, J = 274.2 Hz), 122.2, 116.3. ^{19}F NMR (376 MHz, $CDCl_3$, 30 °C) δ -62.90. IR (neat, ν/cm^{-1}) 3254 w, 3074 w, 2924 w, 1503 s, 1414 m, 1374 s, 1325 s, 1309 s, 1163 s, 1128 s, 1103 s, 1070 s, 925 m, 824 m, 791 s, 693 s. HRMS (EI$^+$) *m/z*: $[M]^+$ Calcd for $C_{16}H_{11}F_3N_2O_2S$ 352.0493; found 352.0491.

2-methyl-*N*-(quinolin-8-yl)benzenesulfonamide (1l)



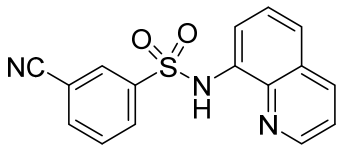
Yield – 2.596 g, 87%. R_f - 0.16 (hexane/EtOAc = 5/1). Off-white solid. MP – 109 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.35 (s, 1H), 8.84 – 8.74 (m, 1H), 8.15 – 8.04 (m, 2H), 7.67 (dd, J = 7.3, 1.2 Hz, 1H), 7.48 – 7.29 (m, 4H), 7.29 – 7.20 (m, 1H), 7.17 (d, J = 7.6 Hz, 1H), 2.74 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 148.7, 138.3, 137.5, 137.2, 136.4, 133.7, 133.1, 132.7, 130.3, 128.3, 126.9, 126.0, 122.1, 121.8, 114.1, 20.3. IR (neat, ν/cm^{-1}) 3279 w, 3064 w, 2968 w, 2929 w, 1503 s, 1470 s, 1411 s, 1364 s, 1308 s, 1165 s, 922 m, 792 m, 754 s, 692 m. HRMS (EI$^+$) *m/z*: $[M]^+$ Calcd for $C_{16}H_{14}N_2O_2S$ 298.0776; found 298.0774.

3,4-dimethoxy-*N*-(quinolin-8-yl)benzenesulfonamide (1m)



Yield – 2.894 g, 84%. R_f - 0.08 (hexane/EtOAc = 5/1). White solid. MP – 134 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.18 (s, 1H), 8.75 (d, *J* = 3.3 Hz, 1H), 8.10 (d, *J* = 8.0 Hz, 1H), 7.84 (dd, *J* = 6.8, 1.9 Hz, 1H), 7.52 (dd, *J* = 8.5, 1.9 Hz, 1H), 7.49 – 7.38 (m, 3H), 7.32 (d, *J* = 1.9 Hz, 1H), 6.76 (d, *J* = 8.5 Hz, 1H), 3.81 (s, 3H), 3.77 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 152.8, 148.9, 148.8, 138.8, 136.4, 134.0, 131.1, 128.3, 126.9, 122.3, 122.0, 121.5, 115.6, 110.3, 109.6, 56.2, 56.1. IR (neat, ν/cm^{-1}) 3256 w, 3077 w, 3008 w, 2964 w, 2935 w, 1587 m, 1504 s, 1469 m, 1410 m, 1368 m, 1333 m, 1308 m, 1262 s, 1156 s, 1139 s, 1089 s, 1020 s, 920 m, 825 m, 792 s, 731 m. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{17}H_{16}N_2O_4S$ 344.0831; found 344.0835.

3-cyano-*N*-(quinolin-8-yl)benzenesulfonamide (1n)

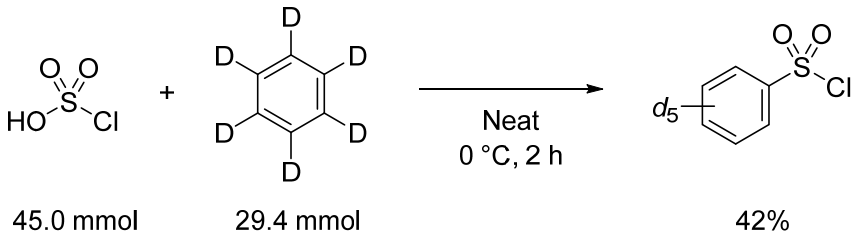


Yield – 2.105 g, 68%. R_f - 0.08 (hexane/EtOAc = 5/1). White solid. MP – 143 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.26 (s, 1H), 8.77 (dd, *J* = 4.3, 1.6 Hz, 1H), 8.20 – 8.15 (m, 1H), 8.15 – 8.07 (m, 2H), 7.86 (dd, *J* = 7.4, 1.2 Hz, 1H), 7.69 (dt, *J* = 7.7, 1.2 Hz, 1H), 7.57 – 7.39 (m, 4H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 149.1, 141.1, 138.7, 136.6, 136.1, 132.9, 131.2, 130.9, 130.0, 128.4, 127.0, 123.3, 122.3, 117.1, 116.2, 113.6. IR (neat,

ν/cm^{-1}) 3252 w, 3072 w, 2235 m, 1503 s, 1414 s, 1374 s, 1309 s, 1205 m, 1159 s, 1089 s, 927 s, 825 s, 792 s, 682 s. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{16}H_{11}N_3O_2S$ 309.0572; found 309.0573.

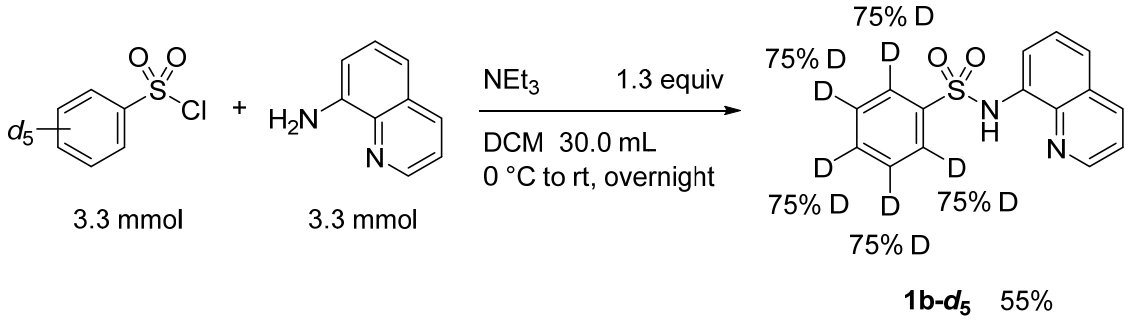
4. Preparation of deuterated sulfonamide 1b-d_5

In an oven dried two-neck round bottom flask chlorosulfonic acid (3.0 mL, 45.0 mmol, 1 equiv) was taken and into that benzene-d_6 (2.6 mL, 29.4 mmol, 0.65 equiv) was added portionwise 0 °C. After complete addition, the ice bath was removed and the resulting mixture was stirred another 2 h at room temperature. Then, the mixture was diluted with 40 mL of CH_2Cl_2 and poured onto 50 mL of ice water. The organic layer was separated and washed with water (2 X 25 mL), dried (Na_2SO_4). Later the organic phase was evaporated to give a colorless oil. Purification was carried out by column chromatography (eluent-EtOAc/hexane) to give 2.3 g (42%) of benzenesulfonyl chloride-d_5 as clear colorless oil.[3]

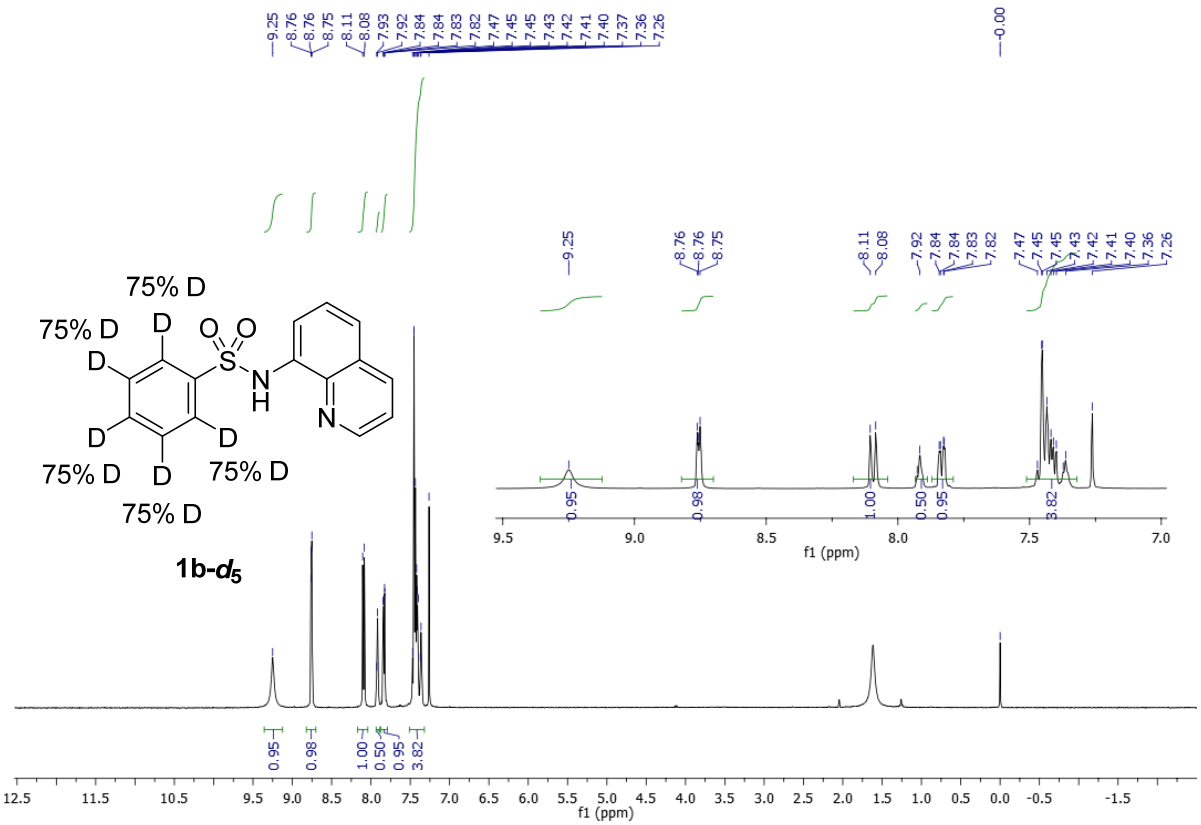


Benzenesulfonyl chloride-d_5 was used for the preparation of sulfonamide **1c-d_5**. In a dry three neck round bottom flask, 8-aminoqunoline (476 mg, 3.3 mmol, 1.0 equiv) and triethylamine (0.6 mL, 4.29 mmol, 1.3 equiv) was dissolved in CH_2Cl_2 (20 mL). After cooling the reaction mixture to 0 °C, benzenesulfonyl chloride-d_5 (600 mg, 3.3 mmol, 1.0

equiv) in 10 mL of CH_2Cl_2 was added dropwise about 10 minutes of period. After that, the resulting mixture was allowed to warm up to room temperature and stirred for overnight. After completion of the reaction, the crude mixture was washed with saturated aqueous $NaHCO_3$ (20 mL). The aqueous layer was washed with 3 x 20 mL of CH_2Cl_2. Then, the organic phase was dried over anhydrous Na_2SO_4 and the volatiles were evaporated to dryness. The resulting crude mixture was purified by flash chromatography on silica gel (eluent: hexane/EtOAc/acetone = 10/3/1) to yield of 521 mg (55 % yield) of sulfonamide **1b-*d*$_5$**.



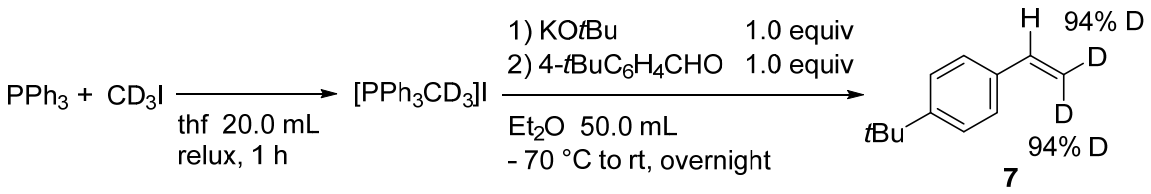
N-(quinolin-8-yl)benzene-d5-sulfonamide (**1b-d$_5$**). Yield 521 mg, 55%. R_f - 0.31 (hexane/EtOAc = 10/3). White solid. MP – 133 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.25 (s, 1H), 8.82 – 8.70 (m, 1H), 8.09 (d, *J* = 8.3 Hz, 1H), 7.95 – 7.79 (m, 0.5H), 7.87 – 7.79 (m, 1H), 7.51 – 7.32 (m, 3H of quinoline moiety, and 0.82 H of arene moiety).



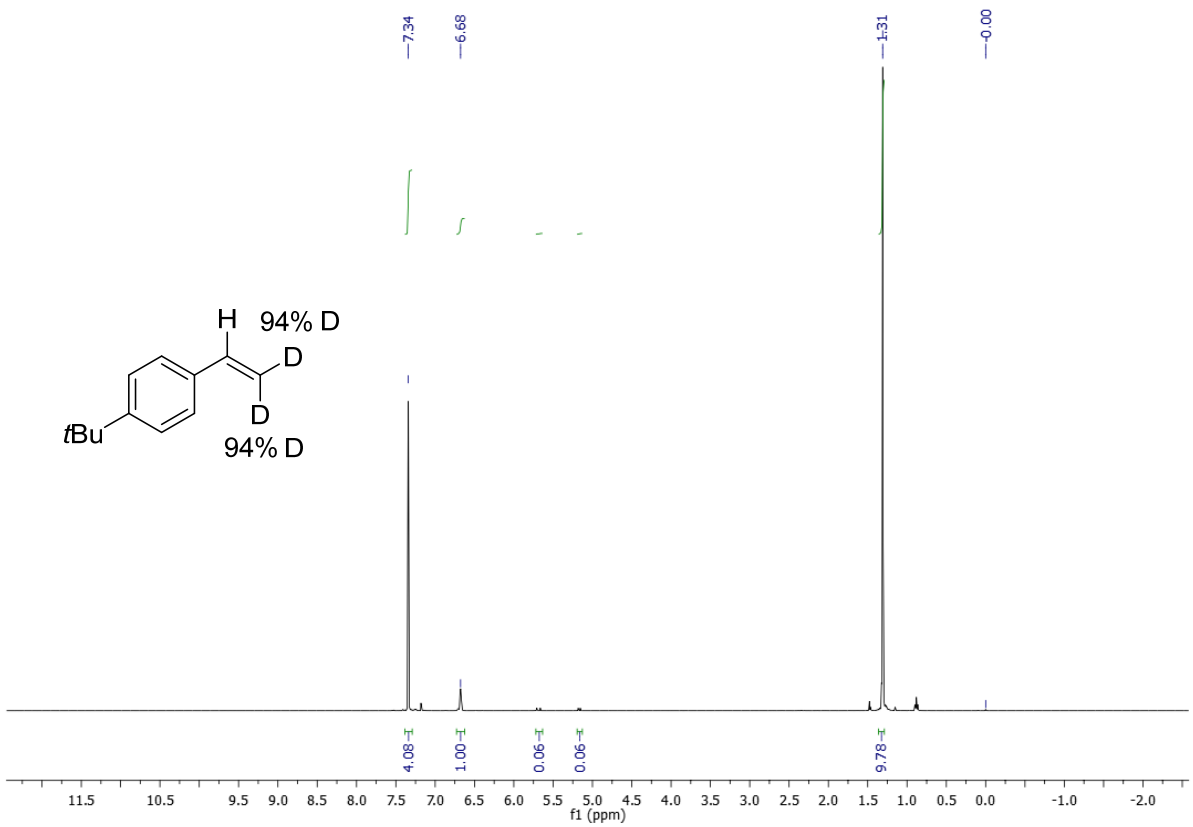
5. Preparation of α-deuterated styrene derivative 7

In a J-Young Schlenk tube, PPh_3 (3.934 g, 15.0 mmol, 1 equiv) in 20 mL THF was added, and CD_3I (0.93 ml, 15.0 mmol, 1 equiv) was then added dropwise into the solution, which resulted in the immediate appearance of a white suspension. After the addition was complete, the reaction mixture was refluxed for 1 h. During the cooling of the reaction mixture to room temperature, a large amount of a white precipitate was formed, and the resulting white solid was isolated on a filter, washed with Et_2O (3 x 20 ml) and dried under vacuum to give 6.048 g of $[PPh_3(CD_3)]I$ (99% yield). After confirming that a >95% D atom incorporation had occurred by NMR spectroscopy, the resulting $[PPh_3(CD_3)]I$ (6.048 g, 14.9 mmol, 1 equiv) was suspended in Et_2O (50 ml), and *tert*-BuOK (1.666 g, 14.9 mmol, 1 equiv) was added into the reaction mixture under an inert atmosphere, which resulted in

the color of reaction mixture changing from a white to a yellow suspension. The resulting reaction mixture was stirred at room temperature. After 2 h, 4-*tert*-butylbenzaldehyde (2.5 ml, 14.9 mmol, 1 equiv) was added dropwise at - 70 °C to give a white suspension. The reaction temperature was allowed to warm to room temperature and was then stirred overnight. After adding water (30 ml) to the suspension, the product was extracted with Et_2O (3 x 30 mL). The combined organic phases were washed with brine (10 ml), and dried over Na_2SO_4. The organic phase was concentrated in vacuo and the product was purified by silica gel chromatography using pentane as the eluent to yield the product **7** in 77% yield.[4]



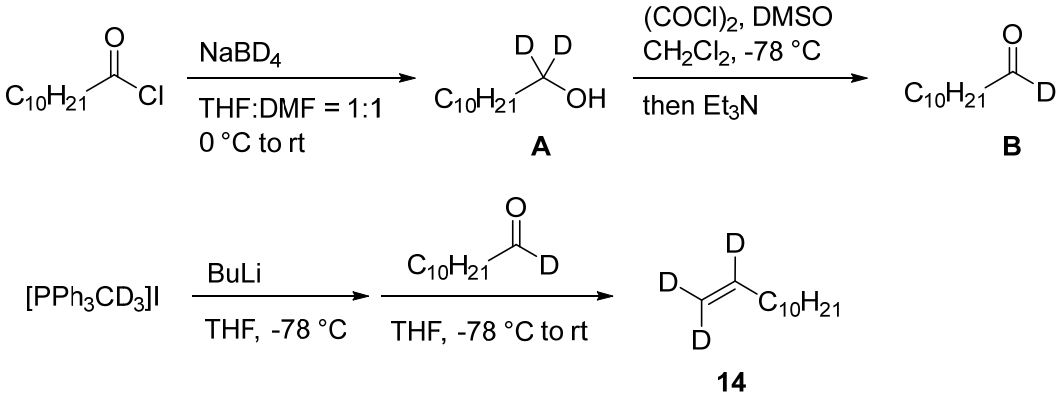
1-(tert-butyl)-4-(vinyl-2,2-d_2)benzene (**7**). Yield 1.862 g, 77%. R_f - 0.58 (hexane). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 7.34 (s, 4H), 6.68 (s, 1H), 5.72 – 5.63 (m, 0.6H), 5.19 – 5.13 (m, 0.6H), 1.31 (s, 9H).



6. Preparation of deuterated olefins 10 and 14

To a stirred solution of sodium borodeuteride (1.2 g, 26 mmol) in THF/DMF (30 mL, 1:1), the acid chloride (8.8 g, 43 mmol) in THF/DMF (30 mL, 1:1) was added at 0 °C under nitrogen. The solution was magnetically stirred at 0 °C for 2 h and the resulting mixture was then allowed to warm to room temperature and was then stirred overnight. The solution containing the crude product was washed with 1 M aqueous HCl and diethyl ether. The organic phase was dried over anhydrous $MgSO_4$ and the solvent removed by evaporation. The resulting crude product was purified by flash chromatography on silica gel (eluent: hexane/EtOAc = 5/1) and distilled to afford desired alcohol **A** as a colorless oil in 58% yield (4.3 g, 25 mmol).[5]

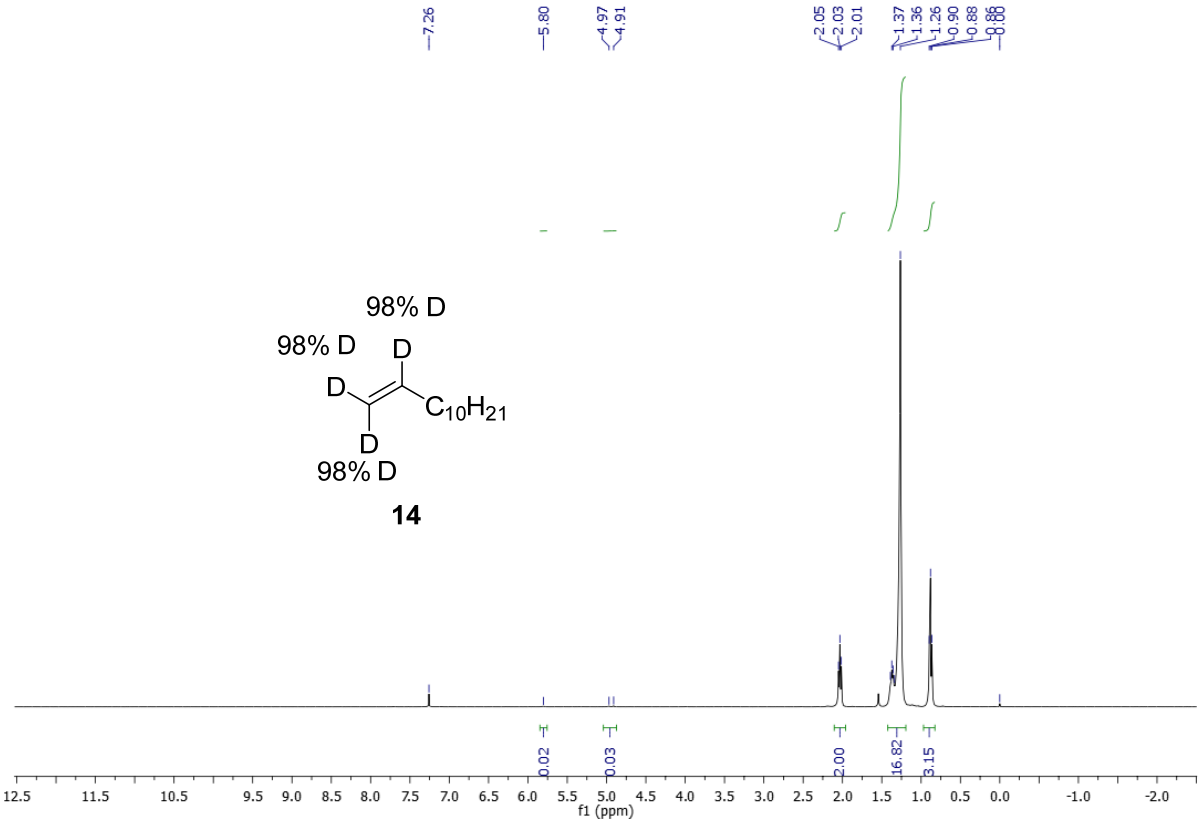
Next, a solution of oxalyl chloride (0.94 mL, 10.8 mmol) in DCM (30 mL), DMSO (0.76 mL, 10.8 mmol) was added dropwise at -78 °C under nitrogen. The solution was stirred at -78 °C for 5 min, and the alcohol (1.7 g, 9.8 mmol) in DCM (10 mL) was then added at -78 °C. The mixture was stirred at -78 °C for 15 min, and triethylamine (6.8 mL, 50 mmol) was then added. After 10 min, the resulting mixture was allowed to warm to room temperature and stirred for an additional 1 h. The solution containing the crude product was washed with saturated aqueous NH_4Cl and brine. The organic phase was dried over anhydrous $MgSO_4$ and the solvent was removed by evaporation. The resulting crude product was purified by flash chromatography on silica gel (eluent: hexane/EtOAc = 10/1) to afford the desired aldehyde **B** as a colorless oil in 79% yield (1.3 g, 7.7 mmol).



To a solution of $[PPh_3(CD_3)]I$ (3.1 g, 7.5 mmol) in THF (30 mL), BuLi (1.6 M in hexane, 4.7 mL, 7.5 mmol) was added at -78 °C under nitrogen. The solution was stirred at -78 °C for 15 min, allowed to warm to room temperature and then stirred for an additional 10 min. The mixture was cooled to -78 °C, and the aldehyde (1.3 g, 7.5 mmol) was slowly added. After 15 min, the resulting mixture was allowed to warm to room temperature and stirred for 15 h. The solution containing the crude product was filtered through a celite pad with

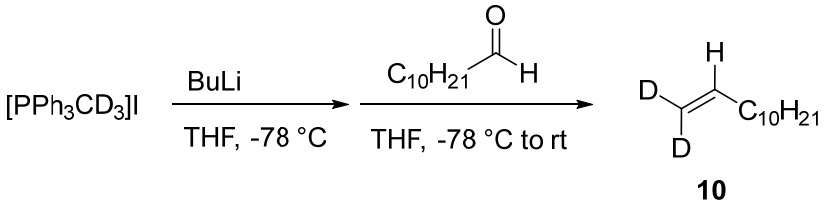
hexane and the filtrate was evaporated. The resulting crude product was purified by flash chromatography on silica gel (eluent: pentane) to afford the desired deuterated alkene **14** (98% D) as a colorless oil in 64% yield (0.83 g, 4.8 mmol).

dodec-1-ene-1,1,2-d$_3$ (15). Yield 0.830 g, 64%. R_f - 0.69 (hexane). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 2.03 (t, J = 7.3 Hz, 2H), 1.26 – 1.39 (m, 16H), 0.88 (t, J = 6.8 Hz, 3H).



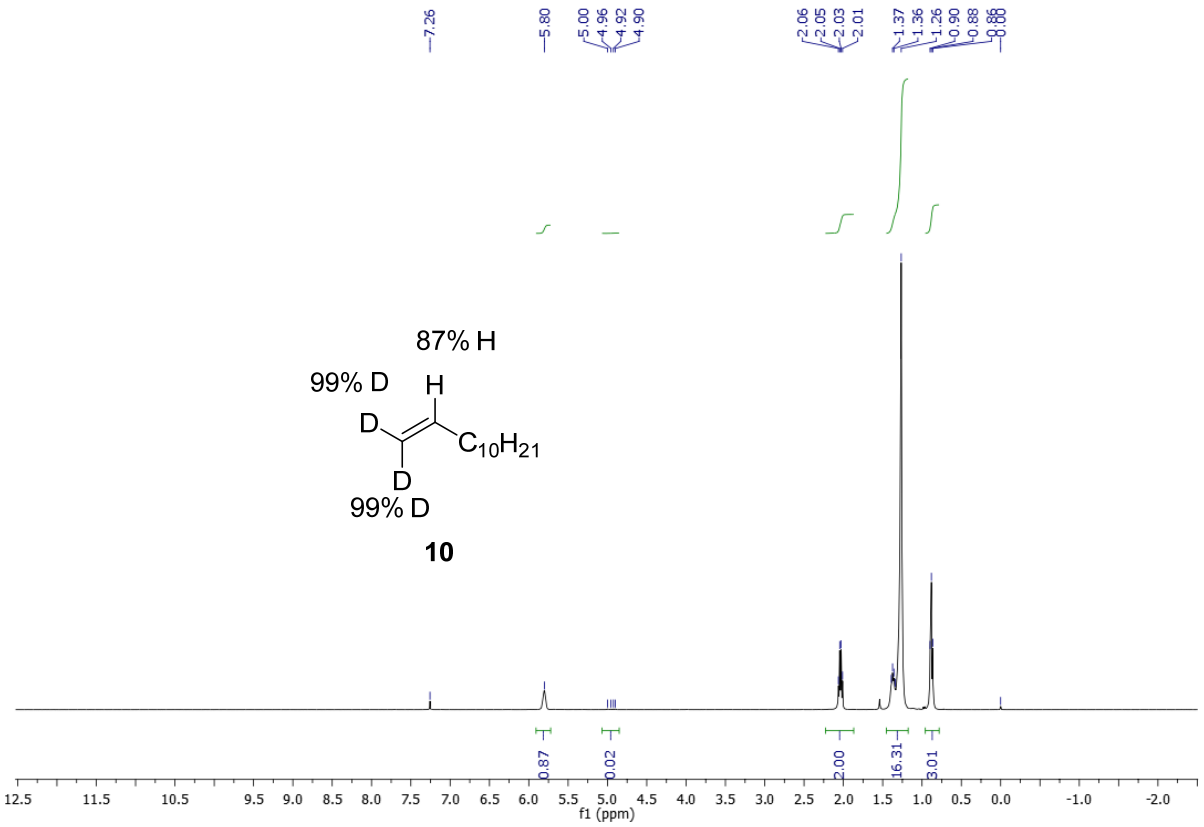
To a solution of [PPh$_3$(CD$_3$)]I (3.1 g, 7.5 mmol) in THF (30 mL), BuLi (1.6 M in hexane, 4.7 mL, 7.5 mmol) was added at -78 °C under nitrogen. The solution was stirred at -78 °C for 15 min, allowed to warm to room temperature and then stirred for an additional 10 min. The mixture was cooled to -78 °C, and undecanal (1.3 g, 7.5 mmol) was slowly added. After 15 min, the resulting mixture was allowed to warm to room temperature and then

stirred for an additional 15 h. The solution containing the crude product was filtered through a celite pad with hexane and the filtrate was evaporated. The resulting crude product was purified by flash chromatography on silica gel (eluent: pentane) to afford the desired deuterated alkene **10** (99% D) as a colorless oil.



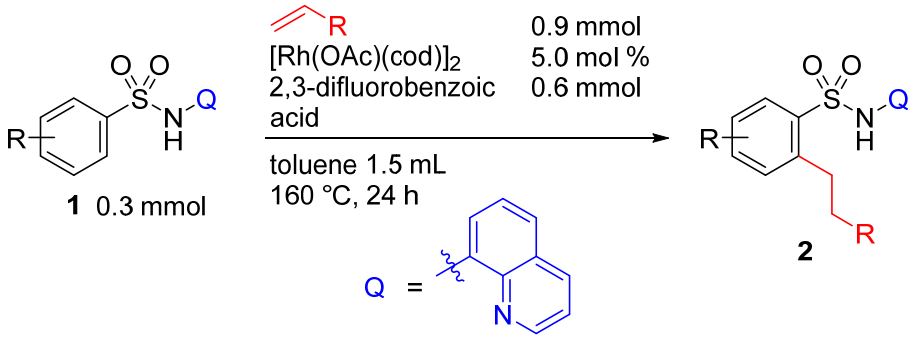
10

dodec-1-ene-1,1-d$_2$ (18). Yield 0.782 g, 60%. R_f - 0.69 (hexane). Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 5.80 (bs, 0.87H), 2.04 (dd, J = 14.1, 7.0 Hz, 2H), 1.45 – 1.18 (m, 16H), 0.88 (t, J = 6.8 Hz, 3H).



7. General procedure for the *ortho*-alkylation of sulfonamide derivatives

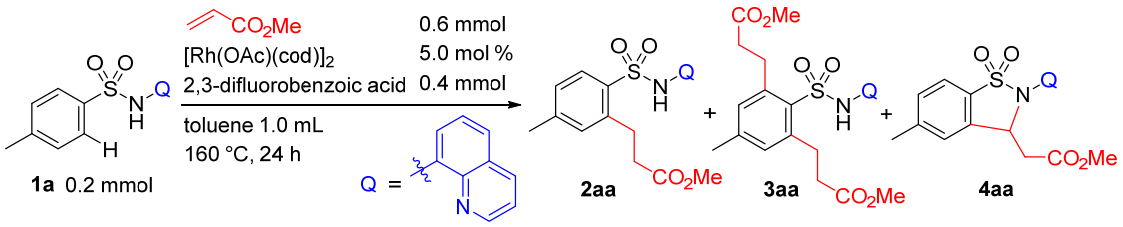
In an oven dried J-Young Schlenk or sealed tube, $[Rh(OAc)(cod)]_2$ (8.1 mg, 0.015 mmol, 0.05 equiv), the sulfonamide (0.3 mmol, 1.0 equiv), and the acid additive (0.6 mmol, 2.0 equiv) were added, after which,1.5 mL of toluene was added. An alkene (0.9 mmol, 3.0 equiv) was then added and the reaction mixture was heated for 24 h at 160 °C in an oil bath. The crude mixture was transferred to a flask and dried under vacuum, and then directly loaded on a column containing NH-silica (eluent- Hexane/EtOAc/acetone). If required, the product was purified further by LC-908 HPLC (GPC).



8. Optimization of Rh-catalyzed *ortho*-alkylation of sulfonamide derivatives

In an oven dried J-Young Schlenk or sealed tube, $[Rh(OAc)(cod)]_2$ (8.1 mg, 0.015 mmol, 0.05 equiv), the sulfonamide (0.3 mmol, 1.0 equiv), and the acid additive (0.6 mmol, 2.0 equiv) were added, after which,1.5 mL of toluene was added. An alkene (0.9 mmol, 3.0 equiv) was then added and the reaction mixture was heated for 24 h at 160 °C in an oil bath. The crude mixture was transferred to a flask and dried under vacuum, and then directly loaded on a column containing NH-silica (eluent- Hexane/EtOAc/acetone). If required, the product was purified further by LC-908 HPLC (GPC).

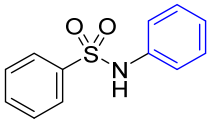
Table S2. Optimization of the Rh-Catalyzed *othro*-Alkylation of Sulfonamide 1a[a]



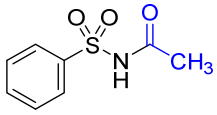
entry	deviation of reaction conditions from standard	yield (%)[b]		
		2aa	**3aa**	**4aa**
1	–	75 (70)	9 (8)	trace
2	1 equiv of 2,3-difluorobenzoic acid	66	8	5
3	3 equiv of 2,3-difluorobenzoic acid	77 (70)	13 (11)	trace
4	1 equiv of *o*-toluic acid	62	11	6
5	1 equiv of pivalic acid	61	9	4
6	1 equiv of acetic acid	58	7	4
7	1 equiv of methyl acrylate	38	3	trace
8	Under N_2	69(65)	10(7)	trace
9	No acid additive	20	trace	trace
10	No catalyst	n.d.	n.d.	n.d.
11	$[RhCl_2Cp^*]_2$ as catalyst	4	n.d.	n.d.
12	$[RhCl_2Cp^*]_2$ as catalyst and $AgSbF_6$ (20 mol%)	trace	trace	trace
13	$[RhCl_2Cp^*]_2$ as catalyst and Zn powder (1 equiv)	n.d.	n.d.	n.d.
14	$[Rh(OAc)_2]_2$ as catalyst	61	8	8
15	$[Rh(cod)Cl]_2$ as catalyst	59	4	4
16	$RhCl(PPh_3)_3$ (10 mol%) as catalyst	36	7	8
17	$Rh(CO)_2(acac)$ (10 mol%) as catalyst	60	3	9
18	$Rh(CO)Cl(PPh_3)_2$ (10 mol%) as catalyst	18	7	4
19	$[Rh(cod)_2]BF_4$ (10 mol%) as catalyst	31	trace	trace
20	**A** as substrate	n.d.	n.d.	n.d.
21[c]	**B** as substrate	<5%	n.d.	n.d.

[a]Reaction conditions: **1a** (0.20 mmol), methyl acrylate (0.60 mmol), 2,3-difluorobenzoic acid (0.4 mmol), and $[Rh(OAc)(cod)]_2$ (5 mol %) in toluene (1.0 mL) at 160 °C for 24 h. [b]Yields are ^{1}H NMR yields. Isolated yields are given in parenthesis. [c]A complicated reaction mixture was

obtained. n.d. refers to not determined.



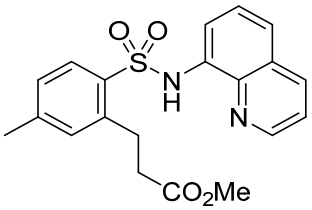
A



B

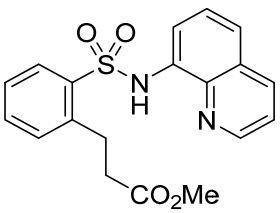
9. Spectral data of products

methyl 3-(5-methyl-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2aa)



Yield – 80.4 mg, 70%. R_f - 0.14 (hexane/EtOAc = 5/2). Off-white solid. MP – 137 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.32 (s, 1H), 8.78 (dd, *J* = 4.1, 1.2 Hz, 1H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.94 (d, *J* = 8.0 Hz, 1H), 7.66 (dd, *J* = 7.3, 1.2 Hz, 1H), 7.47 – 7.31 (m, 3H), 7.10 – 6.96 (m, 2H), 3.68 (s, 3H), 3.39 (t, *J* = 7.8 Hz, 2H), 2.70 (t, *J* = 7.8 Hz, 2H), 2.27 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.2, 148.8, 144.0, 139.9, 138.3, 136.3, 134.6, 133.8, 132.4, 130.4, 128.2, 127.2, 126.8, 122.1, 121.8, 114.3, 51.7, 35.3, 28.3, 21.3. IR (neat, ν/cm^{-1}) 3274 w, 3003 w, 2951 w, 2925 w, 2854 w, 1737 s,1503 s, 1413 m, 1366 s, 1308 m, 1203 m, 1161 s, 1059 m, 921 m, 825 m, 793 m, 658 m. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{20}H_{20}N_2O_4S$ 384.1144; found 384.1142.

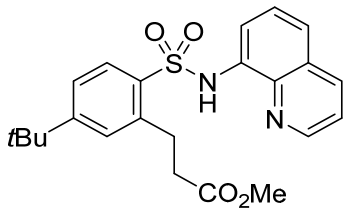
methyl 3-(2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2ba)



Yield – 68.4 mg, 62%. R_f - 0.17 (hexane/EtOAc = 5/2). Off-white solid. MP – 81 °C. ^{1}H

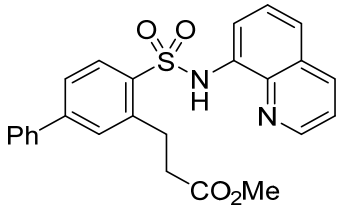
NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.34 (s, 1H), 8.77 (d, *J* = 3.6 Hz, 1H), 8.08 (d, *J* = 8.0 Hz, 1H), 8.04 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 7.3 Hz, 1H), 7.48 – 7.32 (m, 4H), 7.30 – 7.17 (m, 2H), 3.68 (s, 3H), 3.45 (t, *J* = 7.8 Hz, 2H), 2.71 (t, *J* = 7.8 Hz, 2H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.1, 148.8, 140.1, 138.3, 137.5, 136.3, 133.6, 133.2, 131.6, 130.0, 128.2, 126.8, 126.6, 122.1, 122.0, 114.5, 51.7, 35.2, 28.3. IR (neat, ν/cm^{-1}) 3270 w, 3064 w, 2952 w, 2925 w, 1733 s, 1503 s, 1470 m, 1363 m, 1307 s, 1159 s, 1087 m, 922 m, 824 s, 791 s, 755 s, 687 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{19}H_{18}N_2O_4S$ 370.0987; found 370.0990.

methyl 3-(5-(*tert*-butyl)-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2ca)



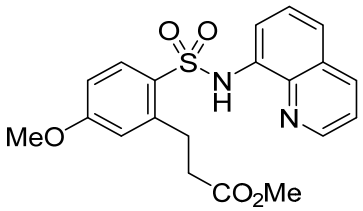
Yield – 76 mg, 59%. R_f - 0.22 (hexane/EtOAc = 5/2). Off-white solid. MP – 123 °C. ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.33 (s, 1H), 8.78 (dt, *J* = 4.2, 1.4 Hz, 1H), 8.09 (dt, *J* = 8.4, 1.4 Hz, 1H), 7.96 (d, *J* = 8.4 Hz, 1H), 7.75 – 7.62 (m, 1H), 7.47 – 7.34 (m, 3H), 7.30 – 7.20 (m, 2H), 3.68 (s, 3H), 3.43 (t, *J* = 7.8 Hz, 2H), 2.73 (t, *J* = 7.8 Hz,, 2H), 1.23 (s, 9H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.2, 156.8, 148.7, 139.6, 138.2, 136.3, 134.7, 133.8, 129.9, 128.9, 128.2, 126.9, 123.6, 122.0, 121.7, 114.2, 51.7, 35.5, 34.9, 30.9, 28.6. IR (neat, ν/cm^{-1}) 3281 w, 2959 w, 2871 w, 1736 s, 1596 w, 1503 s, 1412 m, 1361 s, 1308 s, 1163 s, 1088 m, 922 m, 825 s, 792 s, 757 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{23}H_{26}N_2O_4S$ 426.1613; found 426.1617.

methyl 3-(4-(*N*-(quinolin-8-yl)sulfamoyl)-[1,1'-biphenyl]-3-yl)propanoate (2da)



Yield – 79.8 mg, 60%. R_f - 0.34 (hexane/EtOAc = 5/2). Off-white solid. MP – 112 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.39 (s, 1H), 8.83 – 8.73 (m, 1H), 8.15 – 8.09 (m, 2H), 7.76 – 7.67 (m, 1H), 7.53 – 7.28 (m, 10H), 3.68 (s, 3H), 3.50 (t, *J* = 7.8 Hz, 2H), 2.77 (t, *J* = 7.8 Hz, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.1, 148.8, 145.8, 140.5, 138.9, 138.3, 136.3, 136.1, 133.7, 130.7, 130.3, 129.0, 128.5, 128.2, 127.2, 126.9, 125.0, 122.1, 122.0, 114.5, 51.8, 35.3, 28.5. IR (neat, ν/cm^{-1}) 3272 w, 3062 w, 2951 w, 1733 s, 1597 m, 1503 s, 1412 m, 1362 m, 1308 m, 1161 s, 1087 m, 921 m, 825 m, 792 m, 759 s, 696 m, 666 s. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{25}H_{22}N_2O_4S$ 446.1300; found 446.1303.

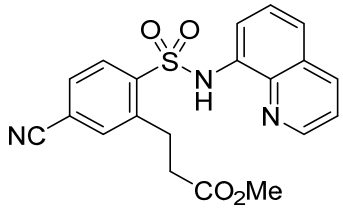
methyl 3-(5-methoxy-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2ea)



Yield – 85.2 mg, 71%. R_f - 0.24 (hexane/EtOAc = 5/2). Off-white solid. MP – 133 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.30 (s, 1H), 8.77 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.08 (dd, *J* = 8.3, 1.5 Hz, 1H), 8.01 (d, *J* = 8.8 Hz, 1H), 7.65 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.45 – 7.32 (m, 3H), 6.79 – 6.65 (m, 2H), 3.74 (s, 3H), 3.68 (s, 3H), 3.39 (d, *J* = 7.8 Hz, 2H), 2.70 (d, *J* = 7.8 Hz, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.1, 163.0, 148.7, 142.3, 138.3, 136.3, 133.8, 132.8, 129.1, 128.2, 126.8, 122.0, 121.8, 117.2, 114.3, 111.0, 55.5, 51.7, 35.1, 28.4. IR (neat, ν/cm^{-1}) 3278 w, 3074 w, 2951 w, 2844 w, 1733 s, 1596 s, 1503 s, 1332 m,

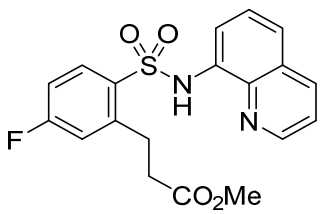
1306 s, 1159 s, 1132 m, 1065 s, 920 m, 825 s, 792 s, 662 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{20}H_{20}N_2O_5S$ 400.1093; found 400.1088.

methyl 3-(5-cyano-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2fa)



Yield – 56.1 mg, 47%. R_f - 0.14 (hexane/EtOAc = 5/2). Off-white solid. MP – 76 °C. ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.31 (s, 1H), 8.80 (d, *J* = 3.9 Hz, 1H), 8.14 (d, *J* = 8.2 Hz, 1H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.71 (d, *J* = 7.5 Hz, 1H), 7.57 (s, 1H), 7.54 – 7.39 (m, 4H), 3.71 (s, 3H), 3.48 (t, *J* = 7.8 Hz, 2H), 2.76 (t, *J* = 7.8 Hz, 2H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 172.5, 149.1, 141.9, 141.6, 138.4, 136.5, 135.1, 132.9, 130.5, 130.1, 128.3, 126.8, 122.9, 122.3, 117.2, 116.8, 115.3, 52.0, 34.6, 28.0. IR (neat, ν/cm^{-1}) 3264 w, 3073 w, 2952 w, 2848 w, 2235 m, 1734 s, 1503 s, 1470 m, 1413 m, 1370 m, 1309 m, 1170 s, 827 m, 793 m, 768 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{20}H_{17}N_3O_4S$ 395.0940; found 395.0945.

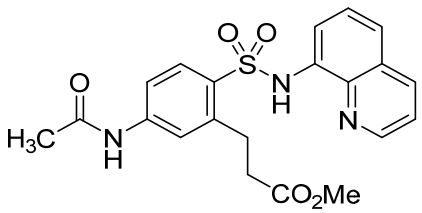
methyl 3-(5-fluoro-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2ga)



Yield – 69.8 mg, 60%. R_f - 0.13 (hexane/EtOAc = 5/2). White solid. MP – 78 °C. ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.33 (s, 1H), 8.81 – 8.73 (m, 1H), 8.11 (dd, *J* = 8.3, 1.2 Hz, 1H), 8.06 (dd, *J* = 8.8, 5.7 Hz, 1H),7.67 (dd, *J* = 7.4, 1.2 Hz, 1H), 7.48 – 7.35 (m, 3H), 7.03

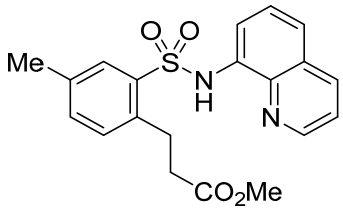
– 6.82 (m, 2H), 3.69 (s, 3H), 3.44 (t, *J* = 7.8 Hz, 2H), 2.71 (t, *J* = 7.8 Hz, 2H). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 172.8, 165.0 (d, *J* = 255.2 Hz), 148.9, 143.6 (d, *J* = 8.7 Hz), 138.4, 136.4, 133.4, 133.1 (d, *J* = 9.5 Hz), 128.3, 126.8, 122.3 (d, *J* = 12.6 Hz), 118.5 (d, *J* = 22.4 Hz), 114.7, 113.5 (d, *J* = 21.8 Hz), 51.9, 34.7, 28.2. ^{19}F NMR (376 MHz, $CDCl_3$, 30 °C) δ -104.97. IR (neat, ν/cm^{-1}) 3272 w, 3074 w, 2952 w, 2851 w, 1735 s, 1580 m, 1503 s, 1366 s, 1308 s, 1232 m, 1160 s, 1060 m, 922 m, 825 m, 792 m, 770 s. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{19}H_{17}FN_2O_4S$ 388.0893; found 388.0889.

methyl 3-(5-acetamido-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2ha)



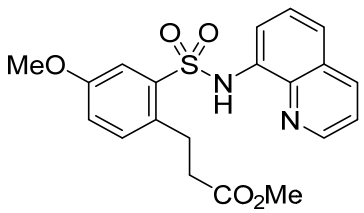
Yield – 63.7 mg, 50%. R_f - 0.06 (hexane/EtOAc = 5/2). Pale yellow solid. MP – 177 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.28 (s, 1H), 8.75 (dd, *J* = 4.2, 1.4 Hz, 1H), 8.25 – 8.02 (m, 2H), 7.93 (d, *J* = 8.6 Hz, 1H), 7.65 – 7.58 (m, 1H), 7.52 – 7.31 (m, 5H), 3.65 (s, 3H), 3.35 (t, *J* = 7.8 Hz, 2H), 2.65 (t, *J* = 7.8 Hz, 2H), 2.07 (s, 3H). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.2, 169.2, 148.8, 142.7, 141.3, 138.3, 136.4, 133.5, 131.6, 128.2, 126.8, 122.1, 121.5, 116.7, 114.5, 51.8, 35.0, 28.3, 24.6. IR (neat, ν/cm^{-1}) 3277 w, 3106 w, 3054 w, 2952 w, 2926 w, 2852 w, 1734 s, 1702 s, 1581 s, 1528 s, 1503 s,1409 s, 1366 s, 1307 s, 1160 s, 1059 m, 922 m, 826 s, 793 s, 758 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{21}H_{21}N_3O_5S$ 427.1202; found 427.1195.

methyl 3-(4-methyl-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2ia)



Yield – 73.9 mg, 64%. R_f - 0.21 (hexane/EtOAc = 5/2). Off-white solid. MP – 130 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.31 (s, 1H), 8.78 (dd, *J* = 4.2, 1.4 Hz, 1H), 8.09 (dd, *J* = 8.3, 1.4 Hz, 1H), 7.88 (d, *J* = 1.0 Hz, 1H), 7.67 (dd, *J* = 7.2, 1.4 Hz, 1H), 7.47 – 7.35 (m, 3H), 7.22 – 7.08 (m, 2H), 3.67 (s, 3H), 3.39 (t, *J* = 7.8 Hz, 2H), 2.68 (t, *J* = 7.8 Hz, 2H), 2.27 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.2, 148.7, 138.4, 137.2, 136.9, 136.6, 136.3, 133.9, 133.7, 131.5, 130.5, 128.2, 126.9, 122.1, 122.0, 114.6, 51.7, 35.3, 27.9, 20.9. IR (neat, ν/cm^{-1}) 3274 w, 2952 w, 2925 w, 2854 w, 1735 s, 1578 w, 1503 s, 1469 m, 1411 m, 1362 s, 1333 m, 1307 s, 1157 s, 1087 m, 923 m, 825 s, 792 s, 757 m, 697 m. HRMS (EI$^+$) *m/z*: $[M]^+$ Calcd for $C_{20}H_{20}N_2O_4S$ 384.1144; found 384.1148.

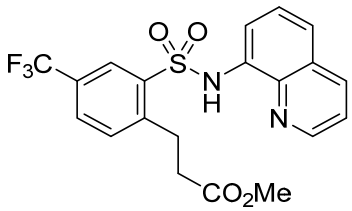
methyl 3-(4-methoxy-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2ja)



Yield – 75.4 mg, 63%. R_f - 0.23 (hexane/EtOAc = 5/2). Off-white solid. MP – 70 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.24 (s, 1H), 8.74 – 8.63 (m, 1H), 8.00 (d, *J* = 8.2 Hz, 1H), 7.62 (d, *J* = 7.3 Hz, 1H), 7.45 (d, *J* = 2.4 Hz, 1H), 7.38 – 7.24 (m, 3H), 7.06 (d, *J* = 8.4 Hz, 1H), 6.80 (dd, *J* = 8.4, 2.4 Hz, 1H), 3.58 (s, 3H), 3.58 (s, 3H), 3.27 (t, *J* = 7.8 Hz, 2H), 2.59 (t, *J* = 7.8 Hz, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.2, 157.7, 148.8, 138.38, 138.35, 136.3, 133.6, 132.8, 131.6, 128.2, 126.8, 122.18, 122.12, 119.0, 115.0, 114.7, 55.6, 51.6, 35.4, 27.5. IR (neat, ν/cm^{-1}) 3271 w, 3004 w, 2950 w, 1735 s, 1605

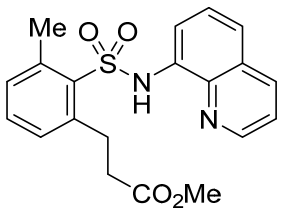
m, 1502 s, 1469 m, 1412 m, 1364 s, 1240 s, 1158 s, 1087 m, 1036 m, 924 m, 825 s, 792 s, 757 m, 695 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{20}H_{20}N_2O_5S$ 400.1093; found 400.1090.

methyl 3-(2-(*N*-(quinolin-8-yl)sulfamoyl)-4-(trifluoromethyl)phenyl)propanoate (2ka)



Yield – 67.2 mg, 51%. R_f - 0.37 (hexane/EtOAc = 5/2). White solid. MP – 137 °C. ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.37 (s, 1H), 8.80 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.28 (s, 1H), 8.12 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.73 (dd, *J* = 7.5, 1.1 Hz, 1H), 7.64 – 7.57 (m, 1H), 7.53 – 7.37 (m, 4H), 3.69 (s, 3H), 3.49 (t, *J* = 7.8 Hz, 2H), 2.74 (t, *J* = 7.8 Hz, 4H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 172.8, 149.1, 144.3, 138.7, 138.5, 136.5, 133.1, 132.4, 129.6 (q, *J* = 3.4 Hz), 129.1 (q, *J* = 33.8 Hz), 128.3, 127.1 (q, *J* = 3.7 Hz), 126.9, 123.2 (q, *J* = 274.0 Hz), 122.9, 122.3, 115.5, 51.9, 34.8, 28.3. ^{19}F NMR (376 MHz, $CDCl_3$, 30 °C) δ – 62.75. IR (neat, ν/cm^{-1}) 3268 w, 3014 w, 2971 w, 2952 w, 1739 s, 1413 m, 1368 s, 1330 s, 1217 s, 1165 s, 1131 s, 1086 m, 826 m, 772 s. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{20}H_{17}F_3N_2O_4S$ 438.0861; found 438.0865.

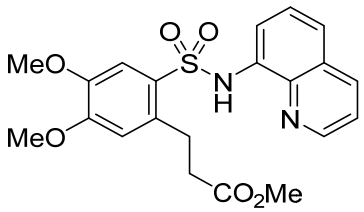
methyl 3-(3-methyl-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2la)



Yield – 53 mg, 46%. R_f - 0.13 (hexane/EtOAc = 5/2). Off-white solid. MP – 61 °C. ^1H

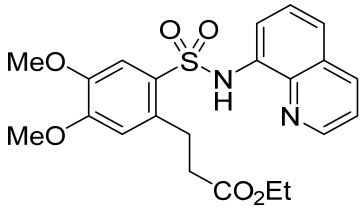
NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.34 (s, 1H), 8.77 (dd, *J* = 4.1, 1.2 Hz, 1H), 8.08 (dd, *J* = 8.2, 0.8 Hz, 1H), 7.59 (dd, *J* = 7.3, 0.8 Hz, 1H), 7.47 – 7.32 (m, 3H), 7.21 (t, *J* = 7.6 Hz, 1H), 7.12 (d, *J* = 7.3 Hz, 1H), 7.05 (d, *J* = 7.3 Hz, 1H), 3.67 (s, 3H), 3.54 (t, *J* = 7.8 Hz, 2H), 2.80 (s, 3H), 2.74 (t, *J* = 7.8 Hz, 2H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.4, 148.7, 142.7, 139.5, 138.5, 136.6, 136.3, 133.8, 132.3, 132.0, 130.8, 128.3, 126.8, 122.1, 122.0, 114.3, 51.7, 36.3, 30.9, 22.9. IR (neat, ν/cm^{-1}) 3286 w, 3061 w, 2950 w, 2851 w, 1733 s, 1579 m, 1503 s, 1410 m, 1362 s, 1307 s, 1161 s, 1087 m, 920 m, 825 s, 791 s, 757 s. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{20}H_{20}N_2O_4S$ 384.1144; found 384.1141.

methyl 3-(4,5-dimethoxy-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2ma)



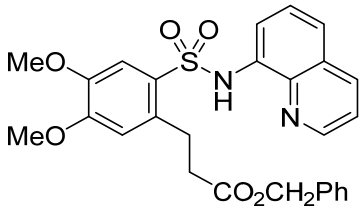
Yield – 95.8 mg, 74%. R_f - 0.11 (hexane/EtOAc = 5/2). Pale yellow solid. MP – 110 °C. ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.29 (s, 1H), 8.83 – 8.72 (m, 1H), 8.10 (dd, *J* = 8.3, 0.9 Hz, 1H), 7.71 (d, *J* = 7.5 Hz, 1H), 7.52 – 7.34 (m, 4H), 6.71 (s, 1H), 3.81 (s, 3H), 3.76 (s, 3H), 3.67 (s, 3H), 3.35 (t, *J* = 7.8 Hz, 2H), 2.70 (t, *J* = 7.8 Hz, 2H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.3, 152.2, 148.8, 146.6, 138.5, 136.3, 134.0, 133.8, 128.8, 128.2, 126.8, 122.1, 122.0, 114.9, 113.9, 113.0, 56.2, 56.0, 51.7, 35.5, 28.2. IR (neat, ν/cm^{-1}) 3268 w, 2952 w, 2929 w, 2850 w, 1732 s, 1573 m, 1504 s, 1469 m, 1306 m, 1265 s, 1146 s, 1088 m, 1048 s, 826 m, 793 s, 758 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{21}H_{22}N_2O_6S$ 430.1199; found 430.1194.

ethyl 3-(4,5-dimethoxy-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2mb)



Yield – 97 mg, 73%. R_f - 0.17 (hexane/EtOAc = 5/2). Off-white solid. MP – 73 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.29 (s, 1H), 8.82 – 8.72 (m, 1H), 8.10 (d, *J* = 8.2 Hz, 1H), 7.72 (d, *J* = 7.3 Hz, 1H), 7.50 (s, 1H), 7.47 – 7.33 (m, 3H), 6.72 (s, 1H), 4.13 (q, *J* = 7.22 Hz, 2H), 3.81 (s, 3H), 3.76 (s, 3H), 3.36 (t, *J* = 7.8 Hz, 2H), 2.69 (t, *J* = 7.8 Hz, 2H), 1.23 (t, *J* = 7.2, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 172.8, 152.2, 148.7, 146.6, 138.4, 136.3, 134.1, 133.8, 128.7, 128.2, 126.7, 122.1, 122.0, 114.8, 113.9, 113.0, 60.5, 56.1, 56.0, 35.7, 28.1, 14.2. IR (neat, ν/cm^{-1}) 3286 w, 3004 w, 2970 w, 2943 w, 1736 s, 1507 s, 1469 m, 1367 s, 1267 m, 1218 s, 1050 m, 795 m, 669 s. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{22}H_{24}N_2O_6S$ 444.1355; found 444.1349.

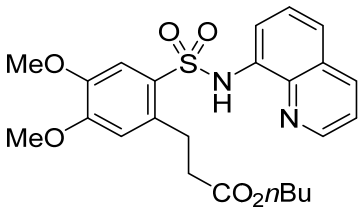
benzyl 3-(4,5-dimethoxy-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2mc)



Yield – 121.2 mg, 80%. R_f - 0.26 (hexane/EtOAc = 5/2). White solid. MP – 88 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.29 (s, 1H), 8.70 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.07 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.71 (dd, *J* = 7.4, 1.3 Hz, 1H), 7.49 (s, 1H), 7.44 – 7.25 (m, 8H), 6.66 (s, 1H), 5.11 (s, 2H), 3.76 (s, 3H), 3.71 (s, 3H), 3.38 (t, *J* = 7.8 Hz, 2H), 2.75 (t, *J* = 7.8 Hz, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 172.6, 152.2, 148.8, 146.6, 138.5, 136.3, 135.9, 133.9, 133.8, 128.7, 128.5, 128.2, 128.1, 126.8, 122.1, 122.0, 114.9, 113.9, 113.0, 66.3,

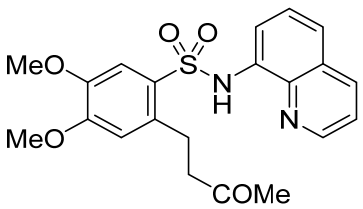
56.2, 56.0, 35.6, 28.1. IR (neat, ν/cm^{-1}) 3269 w, 3006 w, 2937 w, 2847 w, 1733 s, 1504 s, 1360 m, 1265 s, 1222 m, 1146 s, 1087 m, 1047 s, 825 m, 792 m, 755 s, 698 m. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{27}H_{26}N_2O_6S$ 506.1512; found 506.1519.

butyl 3-(4,5-dimethoxy-2-(*N*-(quinolin-8-yl)sulfamoyl)phenyl)propanoate (2md)



Yield – 98.4 mg, 70%. R_f - 0.23 (hexane/EtOAc = 5/2). White solid. MP – 107 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.30 (s, 1H), 8.78 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.11 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.71 (dd, *J* = 7.5, 1.4 Hz, 1H), 7.50 (s, 1H), 7.48 – 7.34 (m, 3H), 6.72 (s, 1H), 4.07 (t, *J* = 6.7 Hz, 2H), 3.81 (s, 3H), 3.77 (s, 3H), 3.35 (t, *J* = 7.8 Hz, 2H), 2.69 (t, *J* = 7.8 Hz, 2H), 1.63 – 1.52 (m, 2H), 1.39 – 1.27 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.0, 152.2, 148.8, 146.6, 138.5, 136.3, 134.1, 133.8, 128.7, 128.2, 126.8, 122.16, 122.10, 114.8, 113.9, 113.0, 64.5, 56.2, 56.1, 35.8, 30.6, 28.2, 19.1, 13.7. IR (neat, ν/cm^{-1}) 3273 w, 2959 w, 2935 w, 2871 w, 1729 s, 1506 s, 1469 m, 1361 m, 1307 m, 1266 s, 1173 s, 1148 s, 1088 m, 1051 s, 826 m, 793 m, 761 m. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{24}H_{28}N_2O_6S$ 472.1668; found 472.1675.

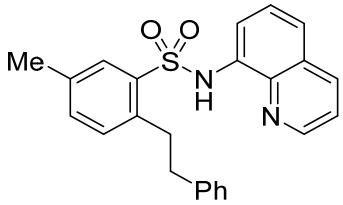
4,5-dimethoxy-2-(3-oxobutyl)-*N*-(quinolin-8-yl)benzenesulfonamide (2me)



Yield – 79.8 mg, 64%. R_f - 0.11 (hexane/EtOAc = 5/2). White solid. MP – 110 °C. ^{1}H NMR

(400 MHz, $CDCl_3$, 30 °C) δ 9.27 (s, 1H), 8.79 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.11 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.70 (dd, *J* = 7.4, 1.0 Hz, 1H), 7.54 – 7.34 (m, 4H), 6.70 (s, 1H), 3.81 (s, 3H), 3.77 (s, 3H), 3.26 (t, *J* = 7.6 Hz, 2H), 2.82 (t, *J* = 7.6 Hz, 2H), 2.11 (s, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 207.9, 152.3, 148.8, 146.6, 138.5, 136.4, 134.8, 133.8, 128.6, 128.3, 126.8, 122.19, 122.14, 114.8, 114.3, 113.0, 56.2, 56.1, 45.3, 30.0, 27.2. IR (neat, ν/cm^{-1}) 3278 w, 3006 w, 2937 w, 1712 s, 1506 s, 1469 m, 1360 s, 1333 m, 1307 m, 1266 s, 1223 m, 1146 s, 1049 s, 826 m, 794 m, 758 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{21}H_{22}N_2O_5S$ 414.1249; found 414.1249.

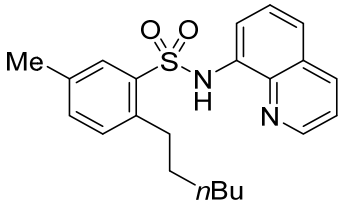
5-methyl-2-phenethyl-*N*-(quinolin-8-yl)benzenesulfonamide (2if)



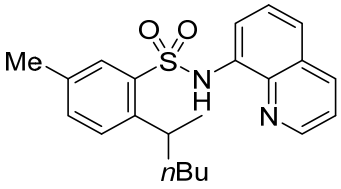
Yield – 69 mg, 57%. R_f - 0.33 (hexane/EtOAc = 5/2). White solid. MP – 124 °C. ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.34 (s, 1H), 8.71 (s, 1H), 8.08 (d, *J* = 7.5 Hz, 1H), 7.93 (s, 1H), 7.69 (d, *J* = 6.6 Hz, 1H), 7.40 – 7.06 (m, 10H), 3.36 (t, *J* = 7.8 Hz, 2H), 2.92 (t, *J* = 7.8 Hz, 2H), 2.29 (s, 3H). Note: Mixture of alkylated and alkenylated product formed in a ratio of 86:14. Several trial has been failed to isolate alkylated product in pure form. Therefore, in the isolated spectra alkenylated product present as an impurity. $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 148.6, 141.5, 138.2, 138.0, 136.9, 136.3, 136.1, 133.8, 131.6, 130.5, 128.6, 128.4, 128.2, 126.9, 126.0, 122.1, 121.8, 114.2, 37.2, 34.6, 20.9. IR (neat, ν/cm^{-1}) 3280 w, 3060 w, 3026 w, 2925 w, 2862 w, 1503 s, 1470 m, 1412 m, 1364 s, 1334

m, 1308 s, 1159 s, 1088 m, 924 m, 823 m, 771 s, 698 s. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{24}H_{22}N_2O_2S$ 402.1402; found 402.1408.

2-Hexyl-5-methyl-N-(quinolin-8-yl)benzenesulfonamide (2ig, linear isomer, major) and 2-(hexan-2-yl)-5-methyl-N-(quinolin-8-yl) benzenesulfonamide (2′ig, branch isomer, minor).



2ig

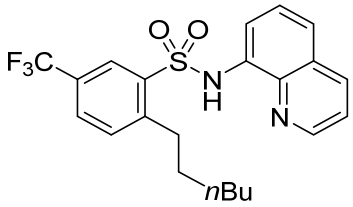


2'ig

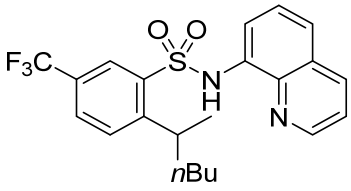
The linear and branch isomer was isolated as a mixture. Yield – 91.5 mg, 79%. R_f - 0.67 (hexane/EtOAc = 5/2). White solid. MP – 89 °C. ^{1}H NMR of linear isomer are as follows: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.30 (s, 1H), 8.81 – 8.71 (m, 1H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.92 (s, 1H), 7.67 – 7.57 (m, 1H), 7.46 – 7.32 (m, 3H), 7.30 – 7.08 (m, 2H), 3.03 (t, *J* = 8.0 Hz, 2H), 2.30 (s, 3H), 1.58 – 1.45 (m, 2H), 1.44 – 1.33 (m, 2H), 1.29 – 1.16 (m, 4H), 0.86 (t, *J* = 7.0 Hz, 3H). Note: Some peaks are overlapped with the minor isomer. Selected ^{1}H NMR peaks of branch isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 7.97 (s, 3H), 3.75 (q, *J* = 6.8 Hz, 1H), 2.33 (s, 3H), 1.11 (d, *J* = 6.8 Hz, 3H), 0.66 (t, *J* = 7.0 Hz, 3H). Combined ^{13}C NMR for both isomers are as follows: ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 148.6, 148.5, 145.2, 139.4, 138.2, 138.0, 136.8, 136.7, 136.3, 135.6, 135.5, 134.2, 133.9, 133.8, 131.2, 130.5, 130.3, 128.2, 128.2, 126.9, 122.0, 121.5, 121.3, 113.8, 113.3, 38.1 (branch isomer), 34.8 (branch isomer), 32.4 (linear isomer), 31.7 (linear isomer), 31.0 (linear isomer), 29.9 (branch isomer), 29.6 (linear isomer), 22.9 (branch isomer), 22.7 (linear isomer), 22.5 (branch isomer), 20.98 (branch isomer), 20.93 (linear isomer), 14.2 (linear

isomer), 13.9 (branch isomer). IR (neat, ν/cm^{-1}) 3288 w, 2955 w, 2926 m, 2858 w, 1503 s, 1470 m, 1412 m, 1364 s, 1308 m, 1160 s, 1088 m, 923 m, 824 m, 791 m, 759 m. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{22}H_{26}N_2O_2S$ 382.1715; found 382.1720.

2-Hexyl-N-(quinolin-8-yl)-5-(trifluoromethyl) benzene-sulfonamide (2kg, linear isomer, major) and 2-(hexan-2-yl)-N-(quinolin-8-yl)-5-(trifluoromethyl)benzenesulfonamide (2′kg, branch isomer, minor).



2kg

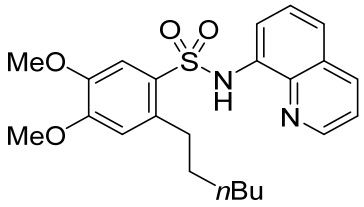


2'kg

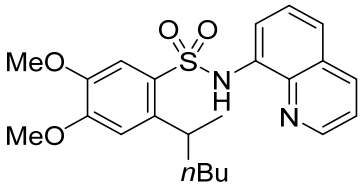
The linear and branch isomer was isolated as a mixture. Yield – 100.5 mg, 76%. R_f - 0.74 (hexane/EtOAc = 5/2). Pale yellow solid. MP – 92 °C. ^{1}H NMR of linear isomer are as follows: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.37 (s, 1H), 8.78 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.34 (d, *J* = 1.0 Hz, 1H), 8.11 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.72 – 7.63 (m, 1H), 7.61 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.50 – 7.33 (m, 4H), 3.14 (t, *J* = 8.0 Hz, 2H), 1.62 – 1.52 (m, 2H), 1.48 – 1.38 (m, 2H), 1.32 – 1.20 (m, 4H), 0.88 (t, *J* = 7.0 Hz, 3H). Note: Some peaks are overlapped with the minor isomer. Selected ^{1}H NMR peaks of branch isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 8.39 (d, *J* = 1.0 Hz, 2H), 3.86 (q, *J* = 6.8 Hz, 1H), 1.15 (d, *J* = 6.8 Hz, 3H), 0.70 (t, *J* = 7.0 Hz, 3H). Combined ^{13}C NMR for both isomers are as follows: ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 152.6, 148.8, 148.7, 146.7, 138.3, 138.1, 138.0, 136.4, 133.3, 131.9, 129.7 (q, *J* = 3.5 Hz), 129.4 (q, *J* = 3.5 Hz), 129.2, 128.37 (q, *J* = 33.6 Hz), 128.35, 127.2 (q, *J* = 3.8 Hz), 126.9, 123.4 (q, *J* = 273.7 Hz), 122.4, 122.2, 122.1, 114.6, 113.9, 38.0 (branch isomer), 35.4 (branch isomer), 32.9 (linear isomer), 31.6 (linear

isomer), 30.8 (linear isomer), 29.8 (branch isomer), 29.5 (linear isomer), 22.9 (branch isomer), 22.7 (linear isomer), 22.2 (branch isomer), 14.2 (linear isomer), 13.9 (branch isomer). ^{19}F NMR (376 MHz, $CDCl_3$, 30 °C) δ – 62.60. IR (neat, ν/cm^{-1}) 3275 w, 2957 w, 2929 w, 2859 w, 1504 s, 1470 m, 1413 m, 1370 m, 1328 s, 1164 s, 1131 s, 1088 m, 1062 w, 925 w, 825 m, 792 m, 771 m. HRMS (EI$^+$) *m/z*: $[M]^+$ Calcd for $C_{22}H_{23}F_3N_2O_2S$ 436.1432; found 436.1436.

2-Hexyl-4,5-dimethoxy-N-(quinolin-8-yl)benzene-sulfonamide (2mg, linear isomer, major) and 2-(hexan-2-yl)-4,5-dimethoxy-N-(quinolin-8-yl) benzenesulfonamide (2′mg, branch isomer, minor)



2mg

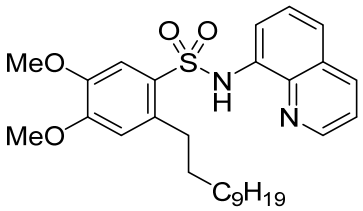


2'mg

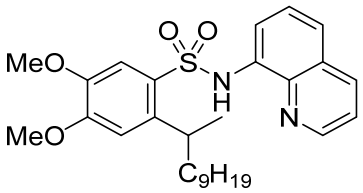
The linear and branch isomer was isolated as a mixture. Yield – 112.9 mg, 88%. R_f - 0.49 (hexane/EtOAc = 5/2). White solid. MP – 80 °C. ^{1}H NMR of linear isomer are as follows: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.28 (s, 1H), 8.77 (dd, *J* = 4.1, 1.3 Hz, 1H), 8.09 (dd, *J* = 8.3, 1.3 Hz, 1H), 7.67 (dd, *J* = 7.3, 1.3 Hz, 1H), 7.55 (s, 1H), 7.47 – 7.33 (m, 3H), 6.66 (s, 1H), 3.83 (s, 3H), 3.80 (s, 3H), 3.00 (t, *J* = 8.0 Hz, 2H), 1.58 – 1.32 (m, 4H), 1.32 – 1.13 (m, 4H), 0.86 (t, *J* = 7.0 Hz, 3H). Note: Some peaks are overlapped with the minor isomer. Selected ^{1}H NMR peaks of branch isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 7.63 (dd, *J* = 7.3, 1.3 Hz, 1H), 7.61 (s, 1H), 6.74 (s, 1H), 3.86 – 3.83 (m, 6H), 3.79 – 3.74 (q, *J* = 6.8 Hz, 1H), 1.10 (d, *J* = 6.8 Hz, 3H), 0.67 (t, *J* = 7.0 Hz, 3H). Combined ^{13}C{^{1}H} NMR for

both isomers are as follows: ^{13}C NMR (101 MHz, $CDCl_3$, 30 °C) δ 152.7, 152.3, 148.6, 148.5, 146.2, 146.1, 142.2, 138.3, 138.1, 136.6, 136.3, 134.0, 128.4, 128.2, 126.8, 122.0, 121.7, 121.4, 114.1, 113.6, 113.4, 113.1, 112.9, 110.0, 56.2, 56.0, 38.2 (branch isomer), 34.8 (branch isomer), 32.7 (linear isomer), 31.7 (linear isomer), 31.3 (linear isomer), 29.8 (branch isomer), 29.5 (linear isomer), 22.9 (branch isomer), 22.6 (linear isomer), 22.4 (branch isomer), 14.1 (linear isomer), 13.9 (branch isomer). IR (neat, ν/cm^{-1}) 3285 w, 2955 w, 2928 w, 2855 w, 1504 s, 1469 m, 1360 m, 1308 m, 1264 s, 1172 m, 1446 s, 1050 s, 920 m, 824 m, 792 m, 757 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{23}H_{28}N_2O_4S$ 428.1770; found 428.1776.

4,5-Dimethoxy-N-(quinolin-8-yl)-2-undecylbenzene-sulfonamide (2mh, linear isomer, major) and 4,5-dimethoxy-N-(quinolin-8-yl)-2-(undecan-2-yl) benzenesulfonamide (2′mh, branch isomer, minor)



2mh

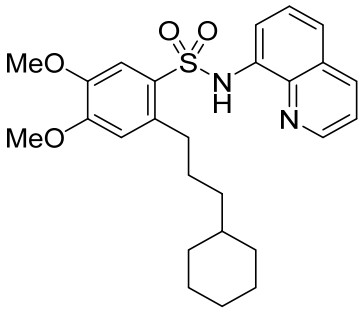


2'mh

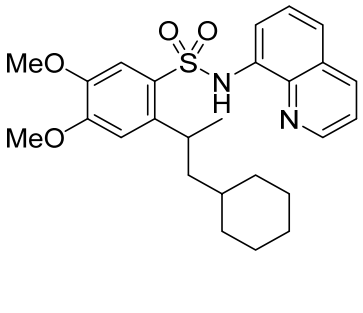
The linear and branch isomer was isolated as a mixture. Yield – 126 mg, 84%. R_f – 0.51 (hexane/EtOAc = 5/2). Off-white solid. MP – 77 °C. ^1H NMR of linear isomer are as follows: ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.29 (s, 1H), 8.76 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.08 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.66 (dd, *J* = 7.2, 1.5 Hz, 1H), 7.56 (s, 1H), 7.43 – 7.33 (m, 3H), 6.66 (s, 1H), 3.82 (s, 3H), 3.81 (s, 3H), 3.00 (t, *J* = 8.0 Hz, 2H), 1.57 – 1.45 (m, 2H), 1.39 (d, *J* = 6.8 Hz, 2H), 1.34 – 1.13 (m, 14H), 0.88 (t, *J* = 7.0 Hz, 3H). Note: Some peaks are overlapped with the minor isomer. Selected ^1H NMR peaks of branch isomer: ^1H NMR

(400 MHz, $CDCl_3$, 30 °C) δ 7.64 – 7.60 (m, 2H), 6.75 (s, 2H), 3.84 (s, 6H), 3.77 (q, *J* = 6.8 Hz, 1H), 1.11 (d, *J* = 6.8 Hz, 3H), 0.86 (d, *J* = 7.0 Hz, 3H). Combined ^{13}C NMR for both isomers are as follows: $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 152.6, 152.2, 148.5, 148.4, 146.1, 146.0, 142.2, 138.2, 138.0, 136.5, 136.3, 133.9, 128.3, 128.1, 126.8, 121.9, 121.6, 121.4, 114.0, 113.4, 113.3, 113.0, 112.8, 109.9, 56.2, 55.9, 38.4 (branch isomer), 34.9 (branch isomer), 32.7 (linear isomer), 31.9 (linear isomer), 31.8 (branch isomer), 31.4 (linear isomer), 29.8, 29.69, 29.65, 29.5, 29.4, 29.38, 29.31, 27.6 (branch isomer), 22.7 (linear isomer), 22.4 (branch isomer), 14.1 (linear isomer). IR (neat, ν/cm^{-1}) 3290 w, 2924 m, 2852 m, 1504 s, 1468 m, 1263 s, 1171 m, 1146 s, 1088 m, 1050 s, 824 m, 792 m, 757 m. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{28}H_{38}N_2O_4S$ 498.2552; found 498.2550.

2-(3-Cyclohexylpropyl)-4,5-dimethoxy-N-(quinolin-8-yl) benzenesulfonamide (2mi, linear isomer, major) and 2-(1-cyclohexylpropan-2-yl)-4,5-dimethoxy-N-(quinolin-8-yl) benzenesulfonamide (2'mi, branch isomer, minor)



2mi

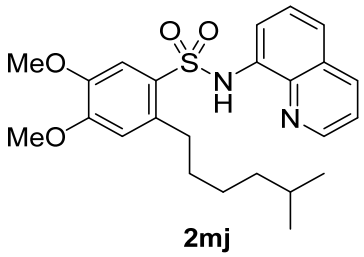


2'mi

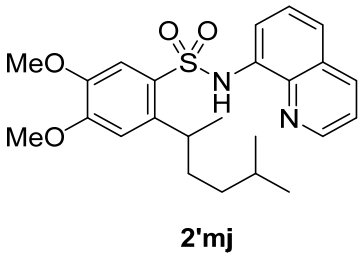
The linear and branch isomer was isolated as a mixture. Yield – 120 mg, 86%. R_f - 0.39 (hexane/EtOAc = 5/2). Off-white solid. MP – 124 °C. ^1H NMR of linear isomer are as follows: ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.28 (s, 1H), 8.82 – 8.75 (m, 1H), 8.10 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.65 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.56 (s, 1H), 7.45 – 7.35 (m, 3H), 6.66

(s, 1H), 3.83 (s, 3H), 3.82 (s, 3H), 2.98 (t, J = 8.0 Hz, 2H), 1.75 – 1.43 (m, 7H), 1.31 – 1.02 (m, 6H), 0.85 – 0.69 (m, 2H). Note: Some peaks are overlapped with the minor isomer. Selected ^{1}H NMR peaks of branch isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 7.62 (dd, J = 7.4, 1.5 Hz, 1H), 7.59 (s, 1H), 6.78 (s, 1H), 3.95 (q, J = 6.8 Hz, 1H), 3.85 (s, 6H). Combined ^{13}C NMR for both isomers are as follows: ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 152.6, 152.2, 148.6, 148.5, 146.1, 146.0, 142.5, 138.3, 138.1, 136.6, 136.3, 134.0, 134.0, 128.3, 128.2, 126.8, 122.0, 121.6, 121.5, 114.0, 113.7, 113.3, 113.0, 112.6, 110.3, 56.2, 56.0, 46.0 (branch isomer), 37.6 (linear isomer), 37.5 (linear isomer), 35.4 (branch isomer), 34.2 (branch isomer), 33.3 (linear isomer), 32.9 (linear isomer), 31.8 (branch isomer), 28.7 (linear isomer), 26.7 (linear isomer), 26.6 (branch isomer), 26.4 (linear isomer), 26.3 (branch isomer), 26.2 (branch isomer), 22.5 (branch isomer). IR (neat, ν/cm^{-1}) 3290 w, 2922 m, 2848 m, 1505 s, 1469 m, 1361 m, 1264 s, 1218 m, 1146 s, 1051 m, 792 m, 770 m. HRMS (EI^{+}) *m/z*: [M]$^{+}$ Calcd for $C_{26}H_{32}N_2O_4S$ 468.2083; found 468.2080.

4,5-Dimethoxy-2-(5-methylhexyl)-N-(quinolin-8-yl) benzenesulfonamide (2mj, linear isomer, major) and 4,5-dimethoxy-2-(5-methylhexan-2-yl)-N-(quinolin-8-yl) benzenesulfonamide (2′mj, branch isomer, minor)



2mj

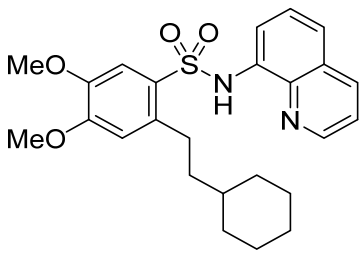


2'mj

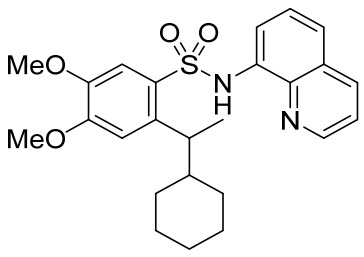
The linear and branch isomer was isolated as a mixture. Yield – 112.5 mg, 85%. R_f - 0.37 (hexane/EtOAc = 5/2). Pale yellow solid. MP – 112 °C. ^{1}H NMR of linear isomer are as follows: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.29 (s, 1H), 8.80 – 8.75 (m, 1H), 8.10 (dd,

J = 8.3, 1.4 Hz, 1H), 7.66 (dd, *J* = 7.3, 1.4 Hz, 1H), 7.56 (s, 1H), 7.46 – 7.34 (m, 3H), 6.66 (s, 1H), 3.83 (s, 3H), 3.81 (s, 3H), 3.01 (t, *J* = 8.0 Hz, 2H), 1.55 – 1.44 (m, 3H), 1.44 – 1.34 (m, 2H), 0.85 (d, *J* = 6.5 Hz, 6H), 0.65 – 0.59 (m, 2H). Note: Some peaks are overlapped with the minor isomer. Selected ^{1}H NMR peaks of branch isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 7.63 (s, 1H), 7.60 (dd, *J* = 7.2, 1.5 Hz, 1H), 6.75 (s, 1H), 3.87 – 3. 84 (m, 6H), 3.74 (q, *J* = 6.8 Hz, 1H), 1.12 (d, *J* = 6.8 Hz, 3H). Combined ^{13}C NMR for both isomers are as follows: $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 152.7, 152.2, 148.6, 148.5, 146.1, 146.0, 142.1, 138.3, 138.0, 136.6, 136.3, 134.0, 128.5, 128.3, 128.2, 126.9, 122.0, 121.7, 121.3, 114.1, 113.3, 113.0, 112.9, 109.8, 56.2, 56.0, 38.8 (linear isomer), 36.5 (branch isomer), 36.1 (branch isomer), 35.0 (branch isomer), 32.8 (linear isomer), 31.7 (linear isomer), 28.1 (branch isomer), 28.0 (linear isomer), 27.7 (linear isomer), 22.79 (branch isomer), 22.73 (linear isomer), 22.4 (branch isomer), 22.3 (branch isomer). IR (neat, ν/cm^{-1}) 3288 w, 2952 m, 2929 m, 2866 w, 1505 s, 1469 m, 1362 m, 1308 m, 1264 s, 1220 m, 1147 s, 1050 m, 825 m, 792 m, 758 m. HRMS (EI^{+}) *m/z*: $[M]^{+}$ Calcd for $C_{24}H_{30}N_2O_4S$ 442.1926; found 442.1926.

2-(2-Cyclohexylethyl)-4,5-dimethoxy-N-(quinolin-8-yl)benzenesulfonamide (2mk, linear isomer, major) and 2-(1-cyclohexylethyl)-4,5-dimethoxy-N-(quinolin-8-yl)benzenesulfonamide (2′mk, branch isomer, minor)



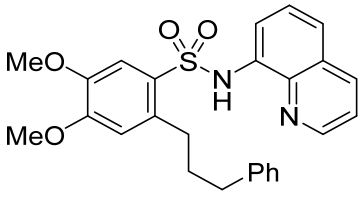
2mk



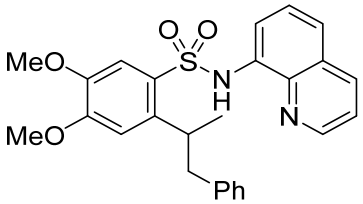
2'mk

The linear and branch isomer was isolated as a mixture. Yield – 84.4 mg, 62%. R_f - 0.20 (hexane/EtOAc = 5/2). Pale yellow solid. MP – 178 °C. ^{1}H NMR of linear isomer are as follows: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.27 (s, 1H), 8.78 (d, *J* = 3.9 Hz, 1H), 8.11 (d, *J* = 8.3 Hz, 1H), 7.66 (d, *J* = 7.3 Hz, 1H), 7.56 (s, 1H), 7.48 – 7.34 (m, 3H), 6.64 (s, 1H), 3.83 (s, 3H), 3.82 (s, 3H), 3.10 – 2.94 (m, 2H), 1.76 – 1.62 (m, 5H), 1.46 – 1.32 (m, 3H), 1.30 – 1.08 (m, 3H), 0.98 – 0.83 (m, 2H). Note: Some peaks are overlapped with the minor isomer. Selected ^{1}H NMR peaks of branch isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 8.73 (d, *J* = 3.9 Hz, 1H), 7.59 (s, 1H), 7.25 – 7.18 (m, 1H), 6.82 (s, 1H), 3.89 (s, 3H), 3.85 (s, 3H). Combined ^{13}C NMR for both isomers are as follows: ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 152.3, 148.6, 148.4, 147.1, 146.2, 141.2, 138.3, 137.0, 136.4, 134.1, 132.0, 128.4, 128.3, 126.9, 123.4, 122.0, 121.7, 121.5, 114.1, 113.7, 113.3, 113.2, 113.0, 110.3, 56.4 (branch isomer), 56.3, 56.1, 41.3 (branch isomer), 38.9, 38.1, 33.3, 32.7 (branch isomer), 30.2, 26.8, 26.5, 26.29 (branch isomer), 26.20 (branch isomer). IR (neat, ν/cm^{-1}) 3284 w, 2923 m, 2849 w, 1505 s, 1467 m, 1360 m, 1308 m, 1262 s, 1220 m, 1146 s, 1049 m, 920 w, 825 m, 793 m, 758 m. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{25}H_{30}N_2O_4S$ 454.1926; found 454.1933.

4,5-Dimethoxy-2-(3-phenylpropyl)-N-(quinolin-8-yl)benzenesulfonamide (2ml, linear isomer, major) and 4,5-dimethoxy-2-(1-phenylpropan-2-yl)-N-(quinolin-8-yl)benzenesulfonamide (2′ml, branch isomer, minor)



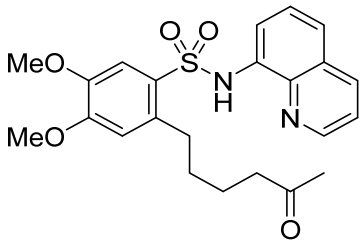
2ml



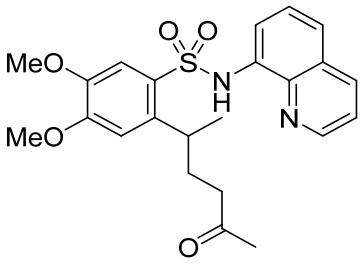
2'ml

The linear and branch isomer was isolated as a mixture. Yield – 124.2 mg, 90%. R_f - 0.20 (hexane/EtOAc = 5/2). White solid. MP – 113 °C. ^{1}H NMR of linear isomer are as follows: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.28 (s, 1H), 8.71 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.08 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.67 (dd, *J* = 7.3, 1.6 Hz, 1H), 7.55 (s, 1H), 7.44 – 7.34 (m, 3H) (peaks overlap with branch isomer), 7.27 – 7.08 (m, 5H) (peaks overlap with branch isomer), 6.61 (s, 1H), 3.79 (s, 3H), 3.78 (s, 3H), 3.08 (t, *J* = 7.8 Hz, 2H), 2.73 (*J* = 7.8 Hz, 2H), 1.94 – 1.83 (m, 2H). Note: Some peaks are overlapped with the minor isomer. Selected ^{1}H NMR peaks of branch isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 7.56 (s, 1H), 6.72 (s, 1H), 3.81 (s, 3H), 3.80 (s, 3H), 3.00 (dd, *J* = 13.3, 4.7 Hz, 1H), 2.62 (t, *J* = 11.4 Hz, 1H), 1.07 (d, *J* = 6.8 Hz, 3H). Combined ^{13}C{^{1}H} NMR for both isomers are as follows: ^{13}C NMR (101 MHz, $CDCl_3$, 30 °C) δ 152.4, 152.3, 148.7, 148.6, 146.3, 146.2, 142.0, 140.9, 139.9, 138.3, 136.3, 136.0, 133.9, 129.5, 128.5, 128.4, 128.3, 128.2, 128.1, 126.8, 126.0, 125.8, 122.0, 121.8, 114.4, 114.3, 113.4, 113.1, 112.9, 110.6, 56.2, 56.06, 56.01 (branch isomer), 44.2 (branch isomer), 36.3 (branch isomer), 36.0 (linear isomer), 32.9 (linear isomer), 32.5 (linear isomer), 20.7 (branch isomer). IR (neat, ν/cm^{-1}) 3276 w, 3024 w, 2934 w, 2849 w, 1505 s, 1469 m, 1308 m, 1264 s, 1220 m, 1171 m, 1146 s, 1050 m, 919 m, 825 m, 792 m, 754 s, 700 m. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{26}H_{26}N_2O_4S$ 462.1613; found 462.1607.

4,5-Dimethoxy-2-(5-oxohexyl)-N-(quinolin-8-yl)benzenesulfonamide (2mm, linear isomer, major) and 4,5-dimethoxy-2-(5-oxohexan-2-yl)-N-(quinolin-8-yl)benzenesulfonamide (2′mm, branch isomer, minor)



2mm

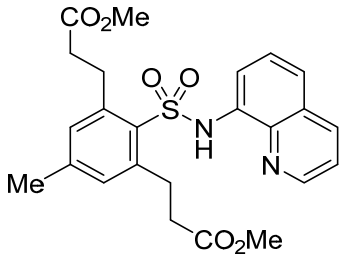


2'mm

The linear and branch isomer was isolated as a mixture. Yield – 85.3 mg, 64%. R_f - 0.09 (hexane/EtOAc = 5/2). Off-white solid. MP – 97 °C. ^{1}H NMR of linear isomer are as follows: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.26 (s, 1H), 8.78 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.11 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.68 (dd, *J* = 7.3, 1.4 Hz, 1H), 7.52 (s, 1H), 7.47 – 7.36 (m, 3H) (overlap with branch isomer), 6.66 (s, 1H), 3.83 (s, 3H), 3.78 (s, 3H), 3.01 (t, *J* = 7.8 Hz, 2H), 2.42 (t, *J* = 7.2 Hz, 2H), 2.12 (s, 3H), 1.74 – 1.64 (m, 2H), 1.63 – 1.53 (m, 2H). Note: Some peaks are overlapped with the minor isomer. Selected ^{1}H NMR peaks of branch isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 7.64 (dd, *J* = 7.3, 1.6 Hz, 1H), 7.61 (s, 1H), 6.73 (s, 1H), 3.85 (s, 3H), 3.84 (s, 3H), 1.86 (s, 3H), 1.08 (d, *J* = 6.8 Hz, 3H). Combined ^{13}C NMR for both isomers are as follows: ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 209.10 (linear isomer), 208.82 (branch isomer), 152.9, 152.3, 148.7, 146.3, 140.6, 138.4, 138.1, 136.4, 135.9, 134.0, 128.8, 128.4, 128.3, 127.0, 126.9, 122.17, 122.10, 121.9, 121.7, 114.4, 113.8, 113.4, 113.0, 112.9, 109.9, 56.2, 56.1, 53.8 (branch isomer), 43.3 (linear isomer), 41.5 (branch isomer), 34.1 (branch isomer), 32.5 (linear isomer), 31.6 (branch isomer), 30.7 (linear isomer), 30.0 (linear isomer), 29.3 (branch isomer), 23.7 (linear isomer), 22.9 (branch isomer). IR (neat, ν/cm^{-1}) 3279 w, 2934 w, 2855 w, 1712 s, 1506 s, 1469 m, 1361 m, 1265 s, 1220 m, 1171 m, 1147 s, 1049 m, 917 w, 826 w, 771 s. HRMS (EI$^+$) m/z: [M]$^+$ Calcd for $C_{23}H_{26}N_2O_5S$ 442.1562; found 442.1567.

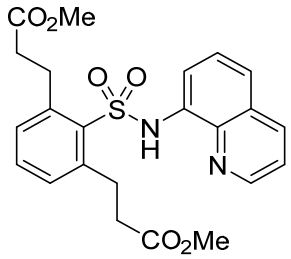
10. Spectral data of double C-H activated products

dimethyl 3,3'-(5-methyl-2-(*N*-(quinolin-8-yl)sulfamoyl)-1,3-phenylene)dipropionate (3aa)



Yield – 12 mg, 8%. R_f - 0.20 (hexane/EtOAc = 5/2). Off-white solid. MP – 139 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.27 (s, 1H), 8.77 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.10 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.59 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.47 – 7.37 (m, 3H), 6.95 (s, 2H), 3.68 (s, 6H), 3.46 (t, *J* = 7.8 Hz, 4H), 2.70 (t, *J* = 7.8 Hz, 4H), 2.23 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.5, 148.8, 143.3, 142.6, 138.8, 136.3, 134.0, 133.8, 132.0, 128.4, 126.9, 122.3, 122.1, 115.2, 51.7, 36.3, 30.7, 21.1. IR (neat, ν/cm^{-1}) 3264 w, 2952 w, 2924 w, 2851 w, 1734 s, 1600 m, 1503 s, 1469 m, 1370 m, 1308 s, 1167 s, 1087 m, 827 m, 792 m, 772 s. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{24}H_{26}N_2O_6S$ 470.1512; found 470.1514.

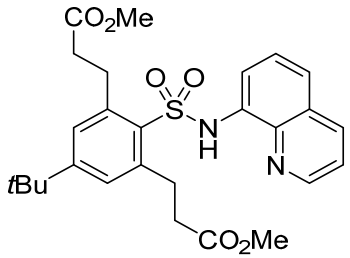
dimethyl 3,3'-(2-(*N*-(quinolin-8-yl)sulfamoyl)-1,3-phenylene)dipropionate (3ba)



Yield – 12.1 mg, 9%. R_f - 0.14 (hexane/EtOAc = 5/2). Off-white solid. MP – 129 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.30 (s, 1H), 8.76 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.10 (dd, *J*

= 8.3, 1.4 Hz, 1H), 7.60 (dd, *J* = 7.5, 1.4 Hz, 1H), 7.48 – 7.37 (m, 3H), 7.32 – 7.23 (m, 1H), 7.15 (d, *J* = 7.5 Hz, 2H), 3.67 (s, 6H), 3.50 (t, *J* = 7.8 Hz, 4H), 2.71 (t, *J* = 7.8 Hz, 4H). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.4, 148.9, 142.7, 138.9, 136.7, 136.3, 133.8, 132.7, 131.2, 128.4, 126.9, 122.5, 122.1, 115.6, 51.7, 36.2, 30.8. IR (neat, ν/cm^{-1}) 3270 w, 3064 w, 2952 w, 2850 w, 1735 s, 1579 m, 1503 s, 1469 m, 1371 m, 1309 m, 1169 s, 1088 m, 827 m, 772 s. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{23}H_{24}N_2O_6S$ 456.1355; found 456.1358.

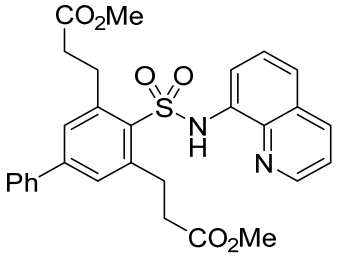
dimethyl-3,3'-(5-(*tert*-butyl)-2-(*N*-(quinolin-8-yl)sulfamoyl)-1,3-phenylene) dipropionate (3ca)



Yield – 13.2 mg, 9%. R_f - 0.21 (hexane/EtOAc = 5/2). White solid. MP – 109 °C. ^1H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.27 (s, 1H), 8.76 (d, *J* = 4.0 Hz, 1H), 8.11 (d, *J* = 8.2 Hz, 1H), 7.62 (d, *J* = 7.2 Hz, 1H), 7.52 – 7.34 (m, 3H), 7.14 (s, 2H), 3.67 (s, 6H), 3.49 (t, *J* = 7.8 Hz, 4H), 2.71 (t, *J* = 7.8 Hz, 4H), 1.22 (s, 9H). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.5, 156.0, 148.7, 142.3, 138.8, 136.4, 134.0, 133.8, 128.49, 128.40, 127.0, 122.3, 122.0, 115.5, 51.7, 36.5, 34.8, 31.0, 30.9. IR (neat, ν/cm^{-1}) 3282 w, 3022 w, 2968 w, 2868 w, 1737 s, 1503 m, 1413 m, 1366 s, 1308 m, 1217 s, 1171 s, 825 w, 772 s. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{27}H_{32}N_2O_6S$ 512.1981; found 512.1989.

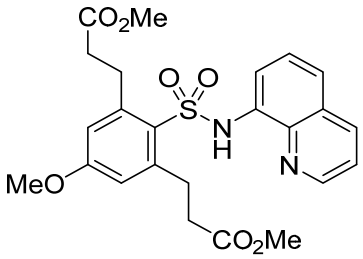
dimethyl 3,3'-(4-(*N*-(quinolin-8-yl)sulfamoyl)-[1,1'-biphenyl]-3,5-diyl)dipropionate

(3da)



Yield – 10.2 mg, 6%. R_f - 0.31 (hexane/EtOAc = 5/2). Off-white solid. MP – 79 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.35 (s, 1H), 8.77 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.11 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.66 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.51 – 7.37 (m, 10H), 3.68 (s, 6H), 3.56 (t, *J* = 7.8 Hz, 4H), 2.77 (t, *J* = 7.8 Hz, 4H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$, 30 °C) δ 173.4, 148.8, 144.9, 143.2, 138.9, 138.6, 136.4, 135.4, 133.8, 129.7, 129.1, 128.7, 128.4, 127.2, 126.9, 122.5, 122.1, 115.7, 51.8, 36.4, 31.0. IR (neat, ν/cm^{-1}) 3276 w, 3061 w, 2951 w, 2852 w, 1733 s, 1595 m, 1503 s, 1412 m, 1308 s, 1167 s, 920 m, 793 m, 768 s, 666 m. HRMS (EI$^+$) *m/z*: [M]$^+$ Calcd for $C_{29}H_{28}N_2O_6S$ 532.1668; found 532.1677.

dimethyl 3,3'-(5-methoxy-2-(*N*-(quinolin-8-yl)sulfamoyl)-1,3-phenylene)dipropionate (3ea)



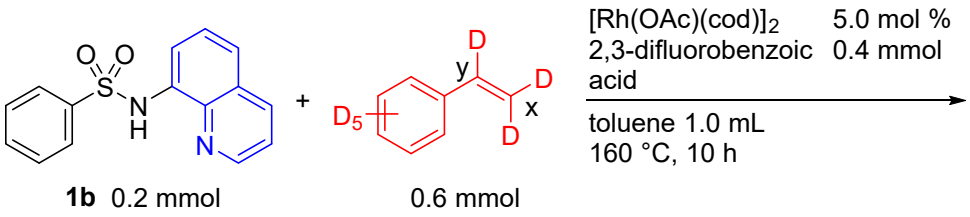
Yield – 8 mg, 5%. R_f - 0.24 (hexane/EtOAc = 5/2). Off-white solid. MP – 117 °C. ^{1}H NMR (400 MHz, $CDCl_3$, 30 °C) δ 9.28 (s, 1H), 8.77 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.11 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.59 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.46 – 7.40 (m, 3H), 6.66 (s, 2H), 3.75 (s, 3H), 3.68 (s, 6H), 3.47 (t, *J* = 7.8 Hz, 4H), 2.71 (t, *J* = 7.8 Hz, 4H). ^{13}C{^{1}H} NMR (101 MHz,

$CDCl_3$, 30 °C) δ 173.4, 161.8, 148.7, 145.3, 138.8, 136.4, 134.0, 128.5, 128.4, 126.9, 122.3, 122.1, 116.2, 115.5, 55.4, 51.8, 36.2, 31.0. IR (neat, ν/cm^{-1}) 3276 w, 2952 w, 2925 w, 2849 w, 1733 s, 1594 s, 1503 s, 1469 m, 1307 s, 1156 s, 1084 s, 1059 m, 826 m, 792 m, 771 s. HRMS (EI^+) *m/z*: $[M]^+$ Calcd for $C_{24}H_{26}N_2O_7S$ 486.1461; found 486.1456.

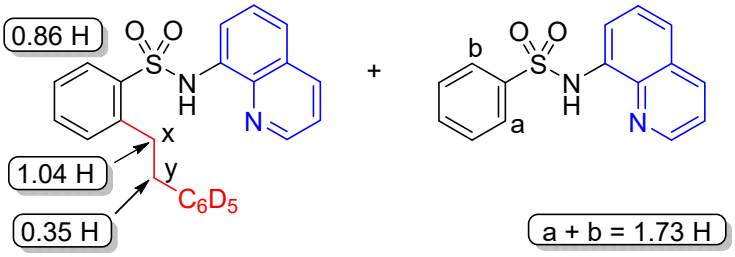
11. Deuterium labelling experiments

In an oven dried J-Young Schlenk or sealed tube, $[Rh(OAc)(cod)]_2$ (8.1 mg, 0.015 mmol, 0.05 equiv), appropriate sulfonamide (0.3 mmol, 1.0 equiv), and acid additive (0.6 mmol, 2.0 equiv) were taken in 1.5 mL of toluene. Into the reaction mixture proper olefins (1.2 mmol, 4.0 equiv or 0.9 mmol, 3.0 equiv) was added and the reaction mixture was kept for 24 h at 160 °C temperature in an oil bath. All the deuterium labelling experiments were performed under atmospheric condition. After completion of the reaction, crude mixture was transferred in a flask and dried under vacuum, and then crude directly loaded in a column contained NH-silica (eluent- Hexane/EtOAc/acetone). Later, the resulted mixture was purified further by LC-908 HPLC (GPC).

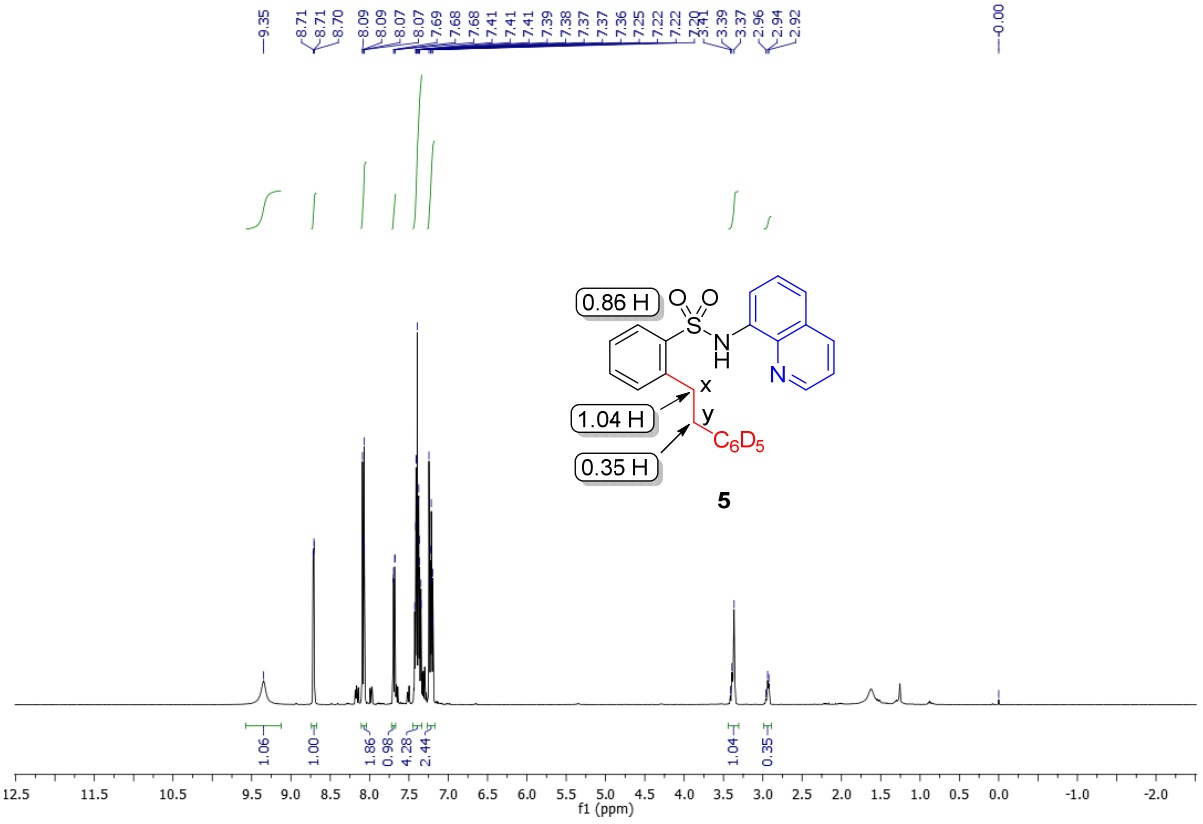
Reaction of sulfonamide 1b and styrene-d_8-

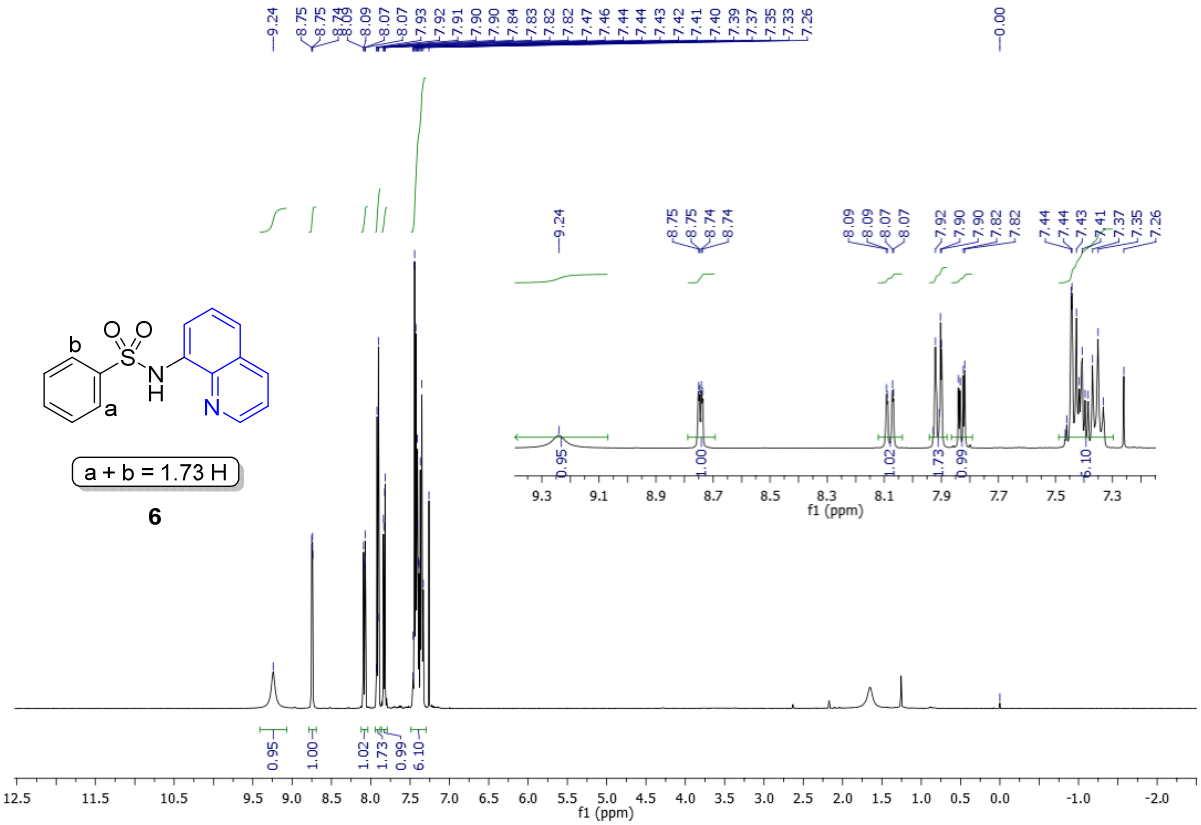


1b 0.2 mmol 0.6 mmol

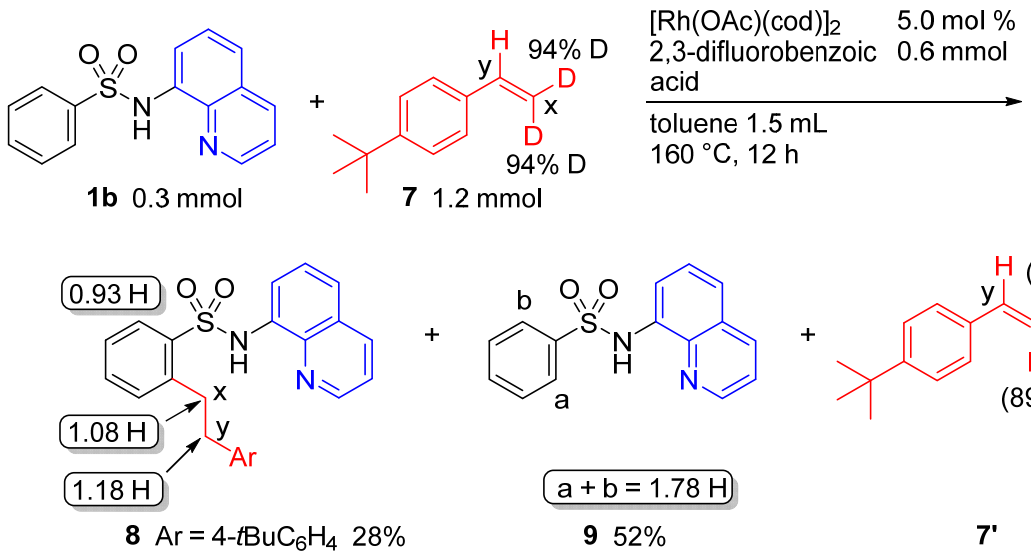


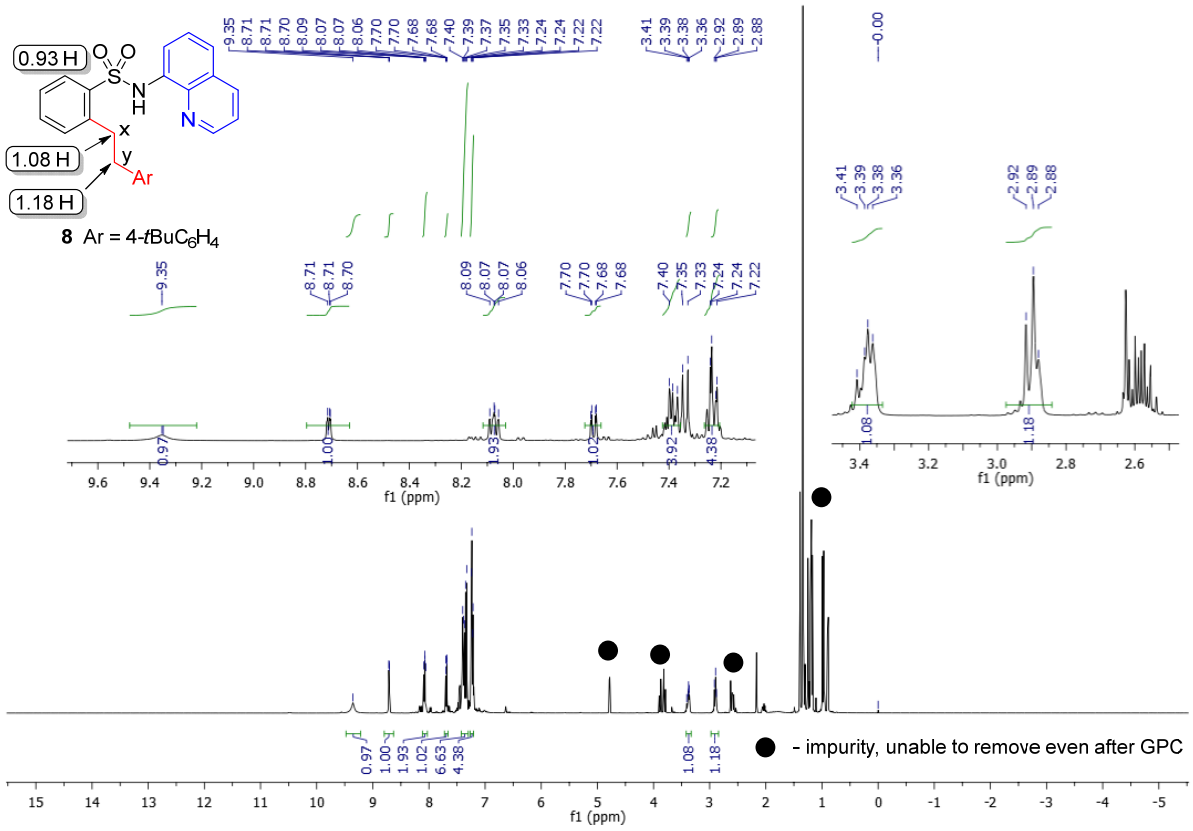
5 31% **6** 48%

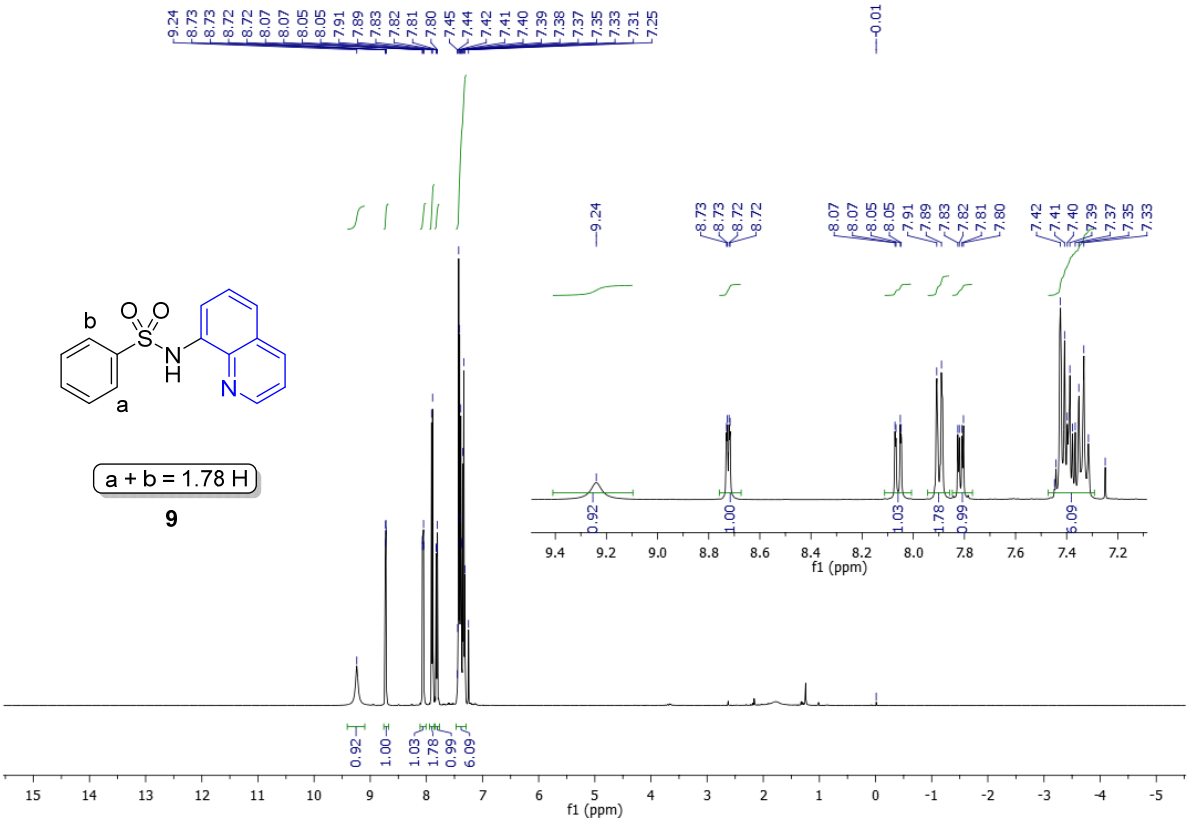


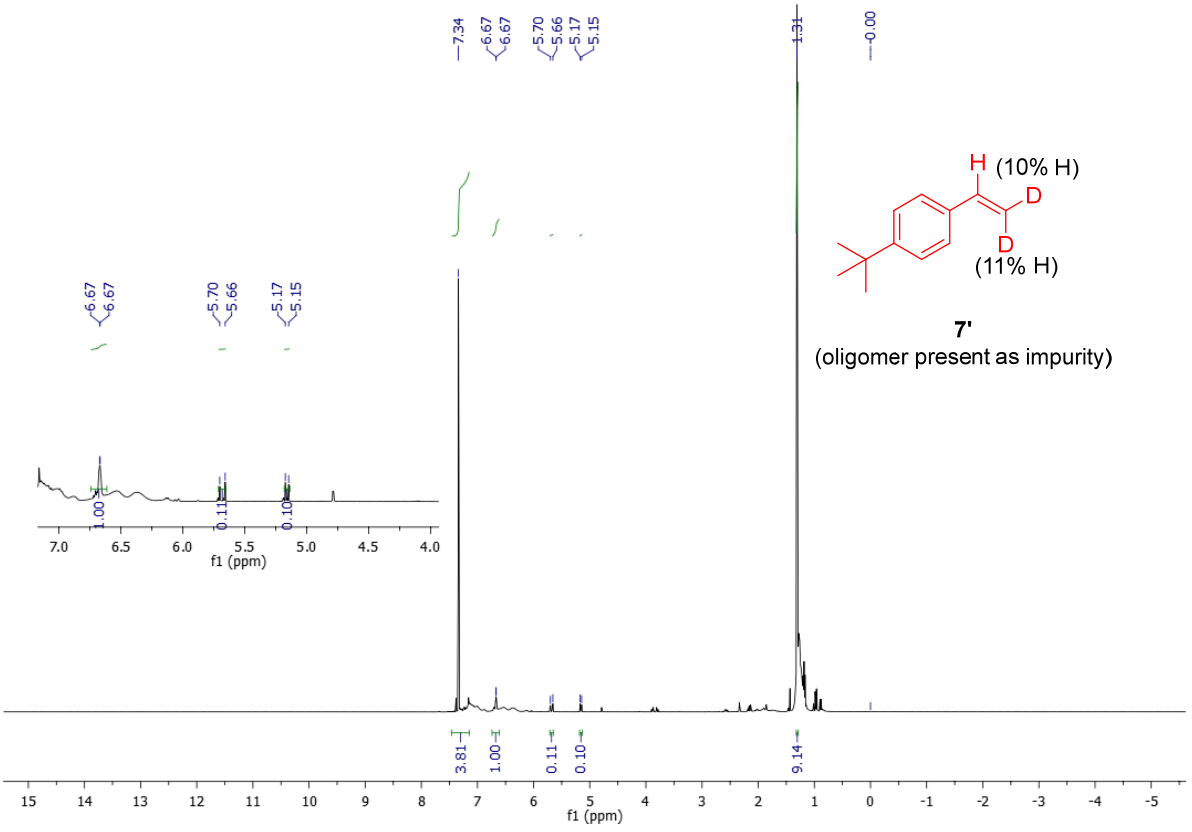

a + b = 1.73 H
6
f1 (ppm)

Reaction of sulfonamide 1b and 7-

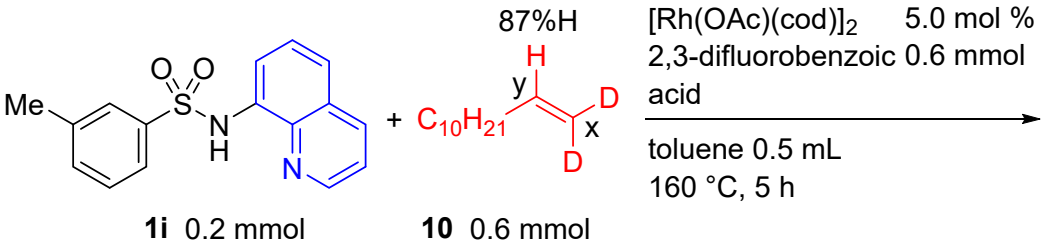


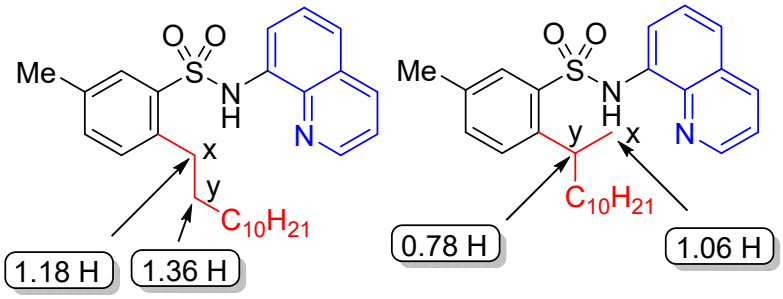


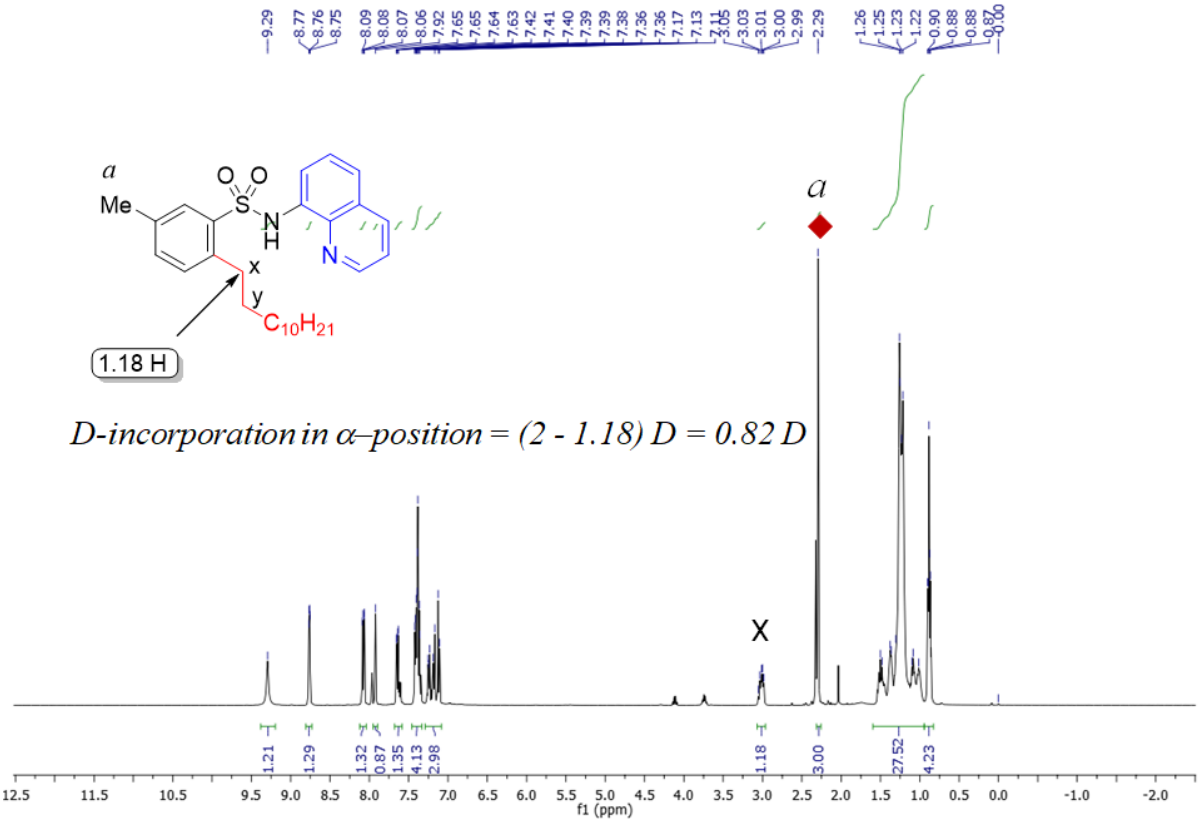

a + b = 1.78 H
9
f1 (ppm)


(10% H)
(11% H)
7'
(oligomer present as impurity)
f1 (ppm)

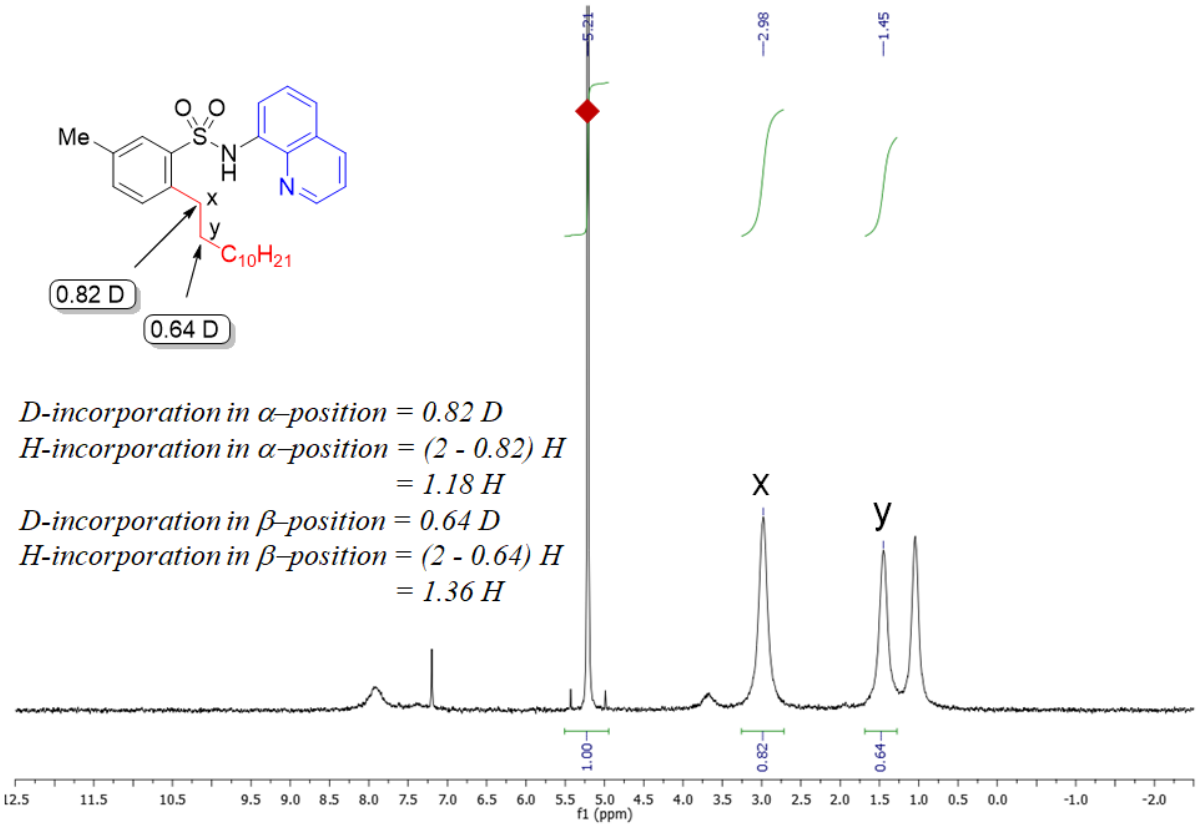
Reaction of sulfonamide 1i and 10-



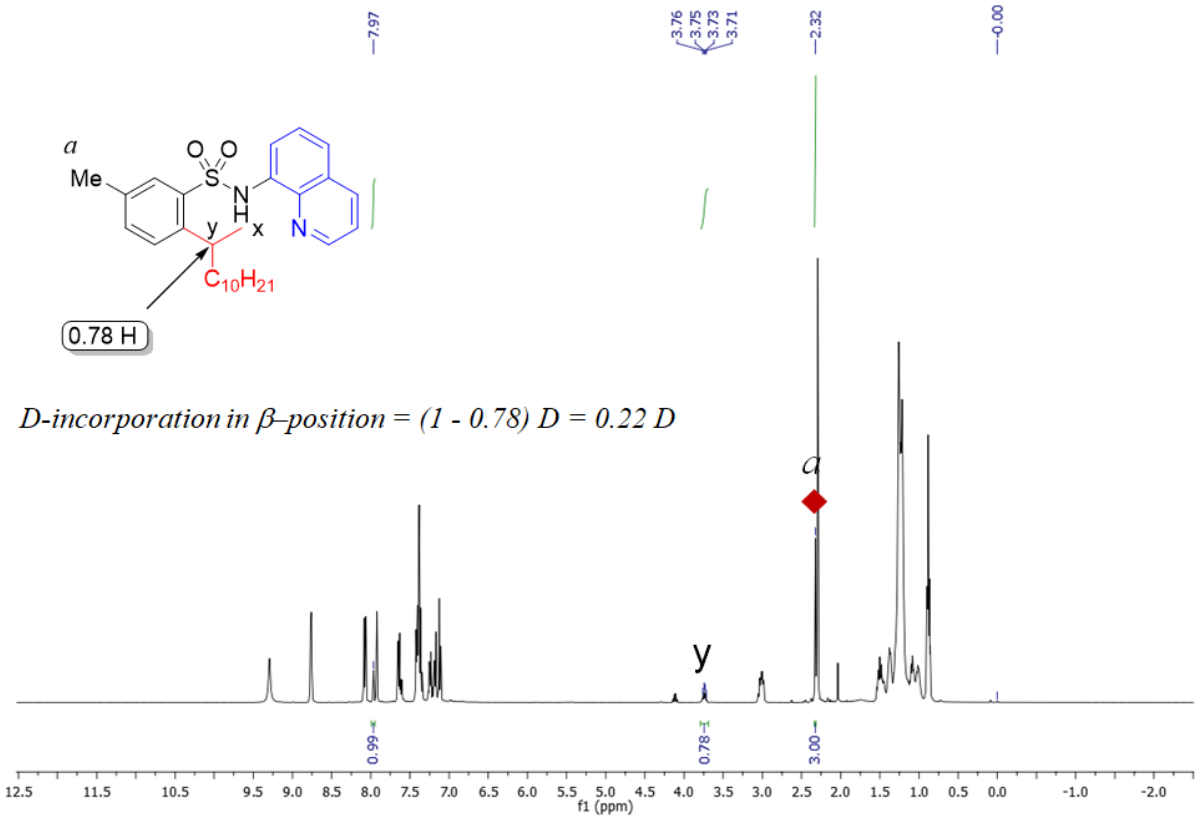




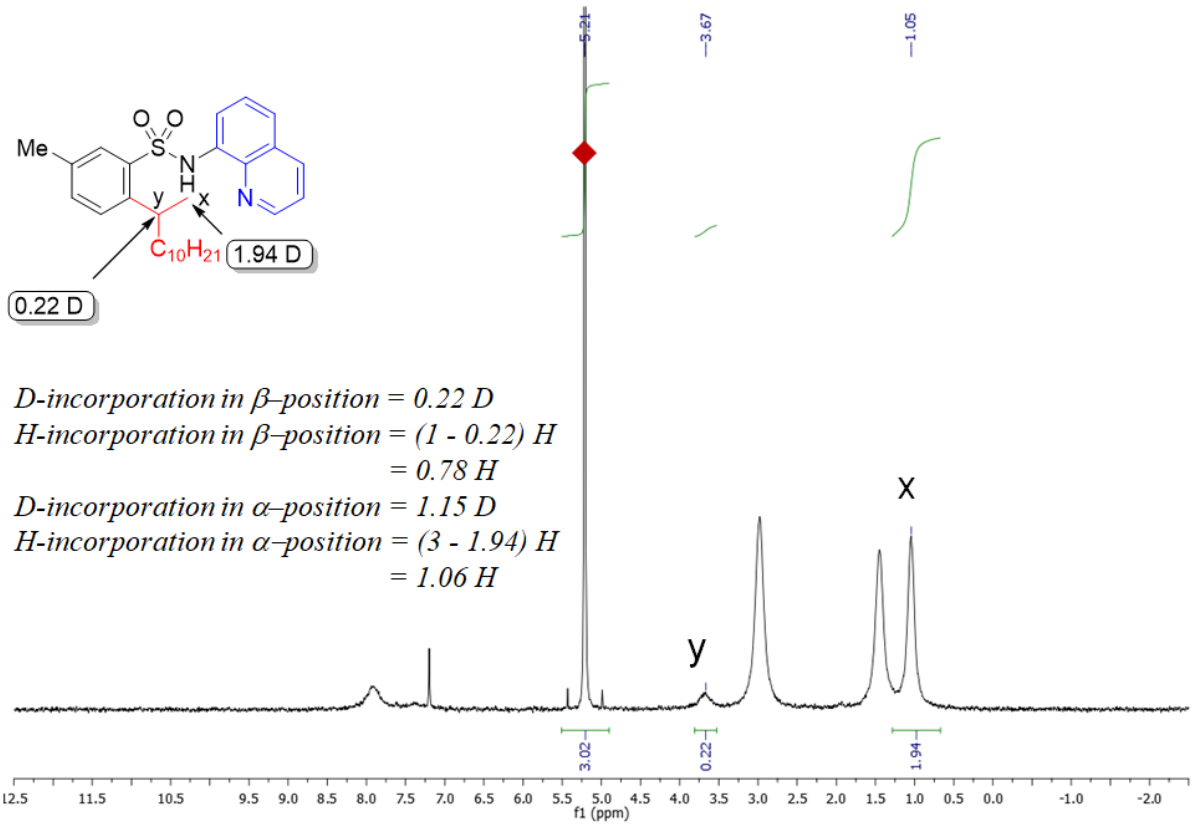
◆ *m*-Me was taken as a standardized peak.



◆ CD_2Cl_2 (internal standard)

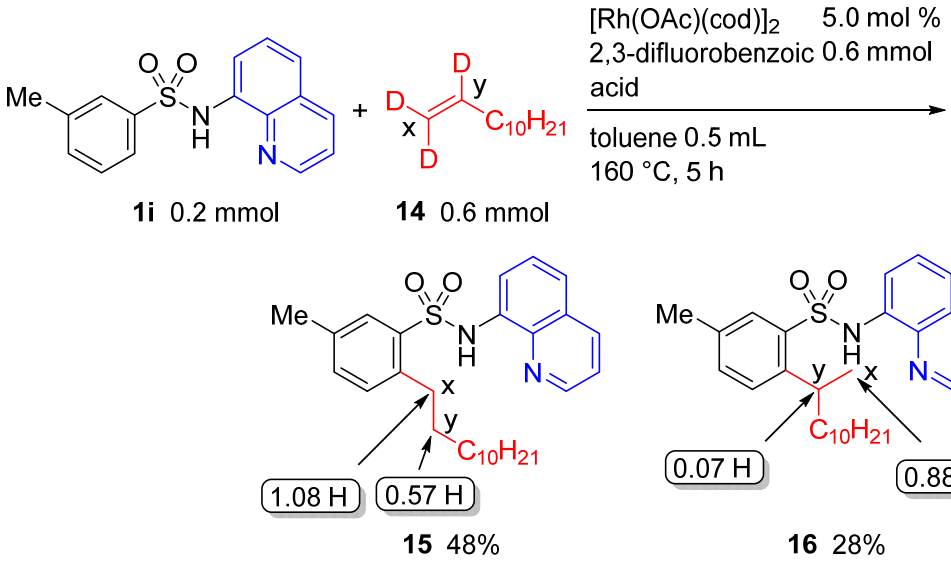


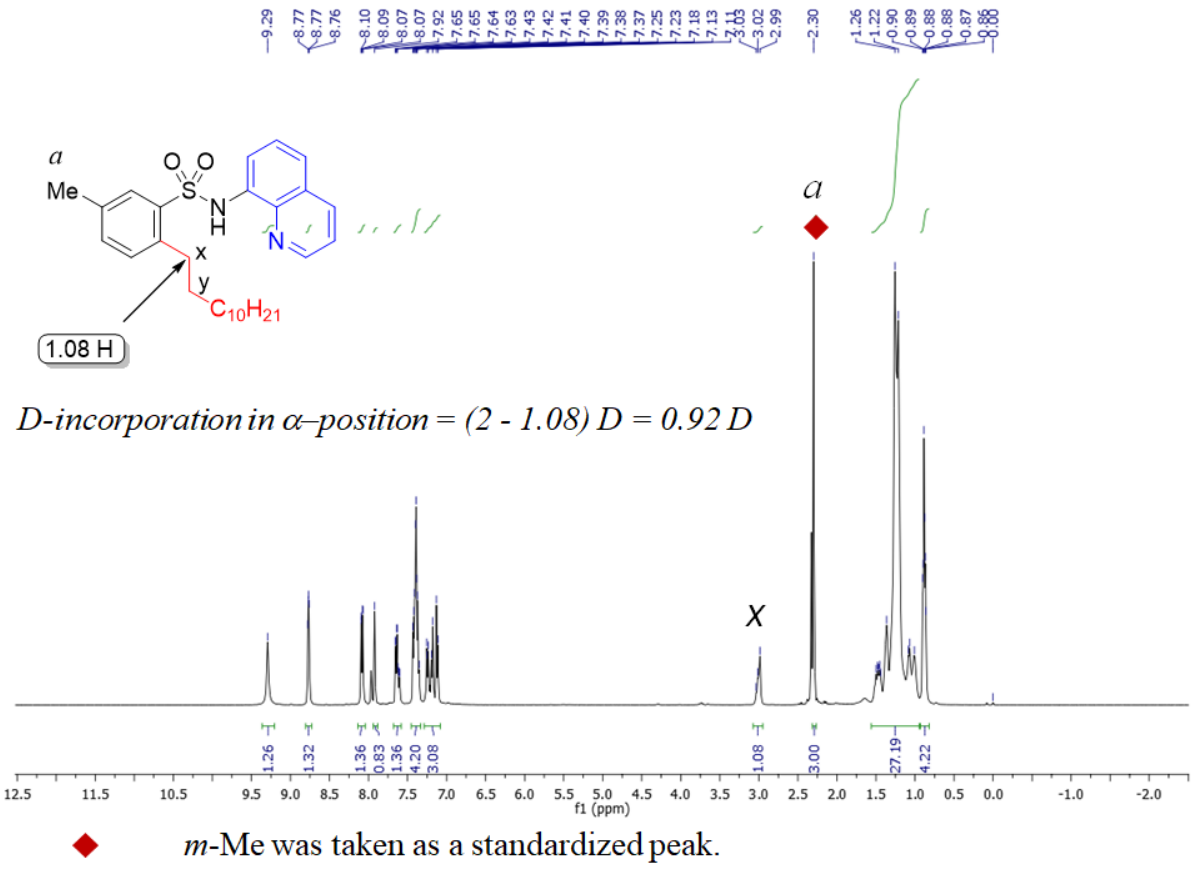
◆ *m*-Me was taken as a standardized peak.

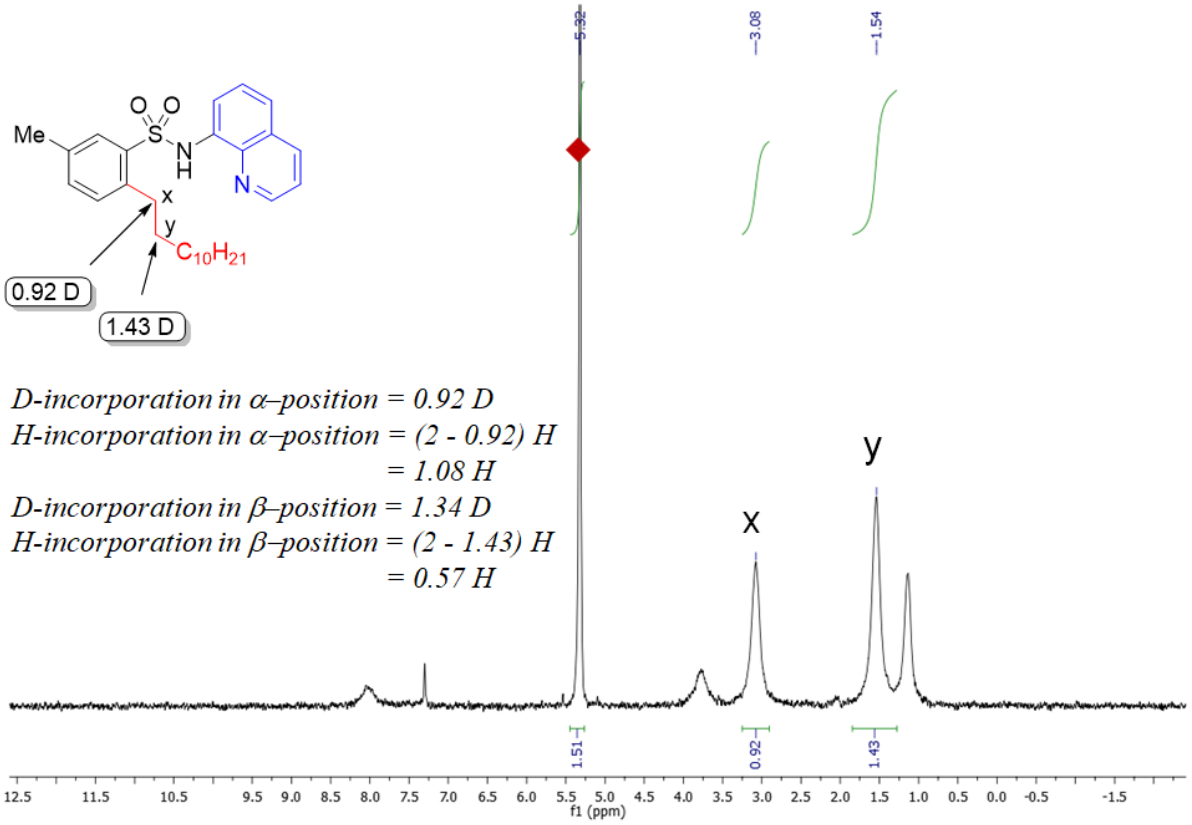


◆ CD_2Cl_2 (internal standard)

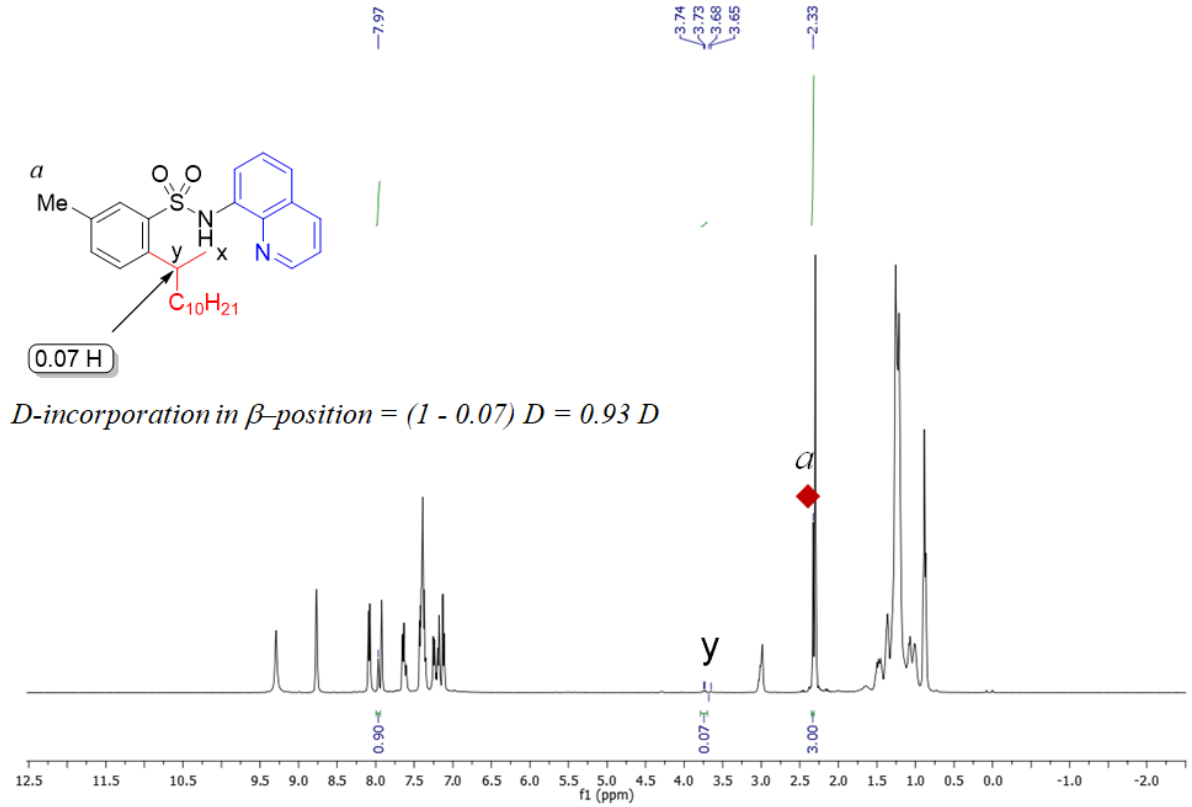
Reaction of sulfonamide 1i and 14-



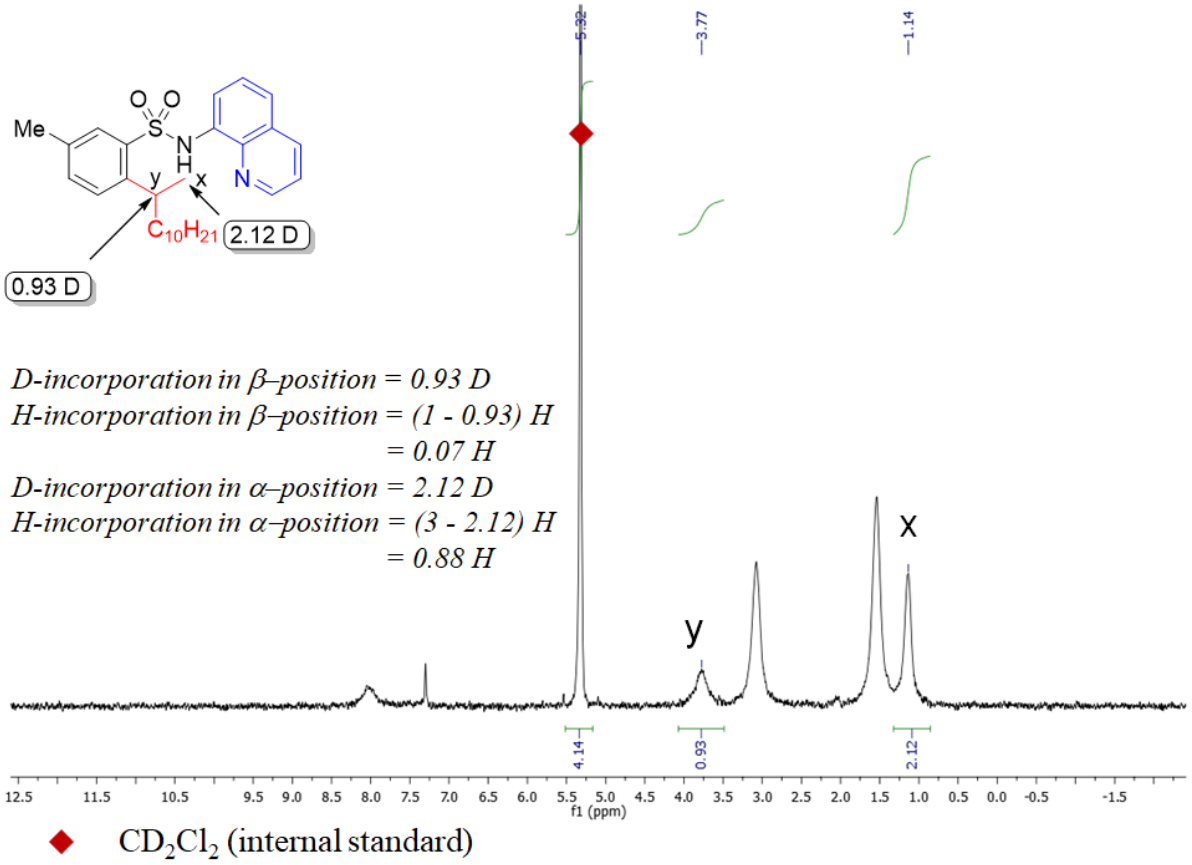




◆ CD_2Cl_2 (internal standard)

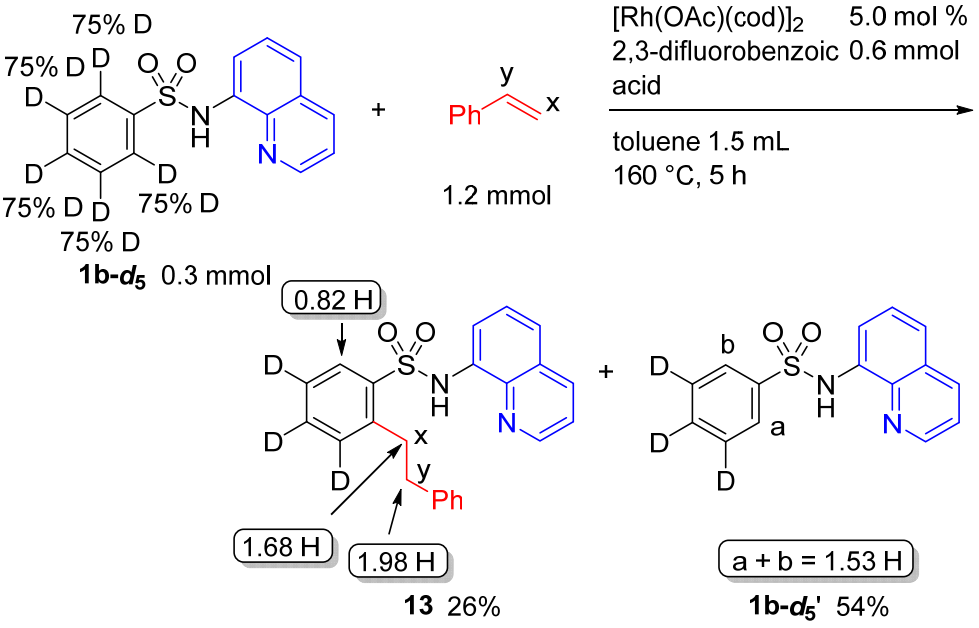


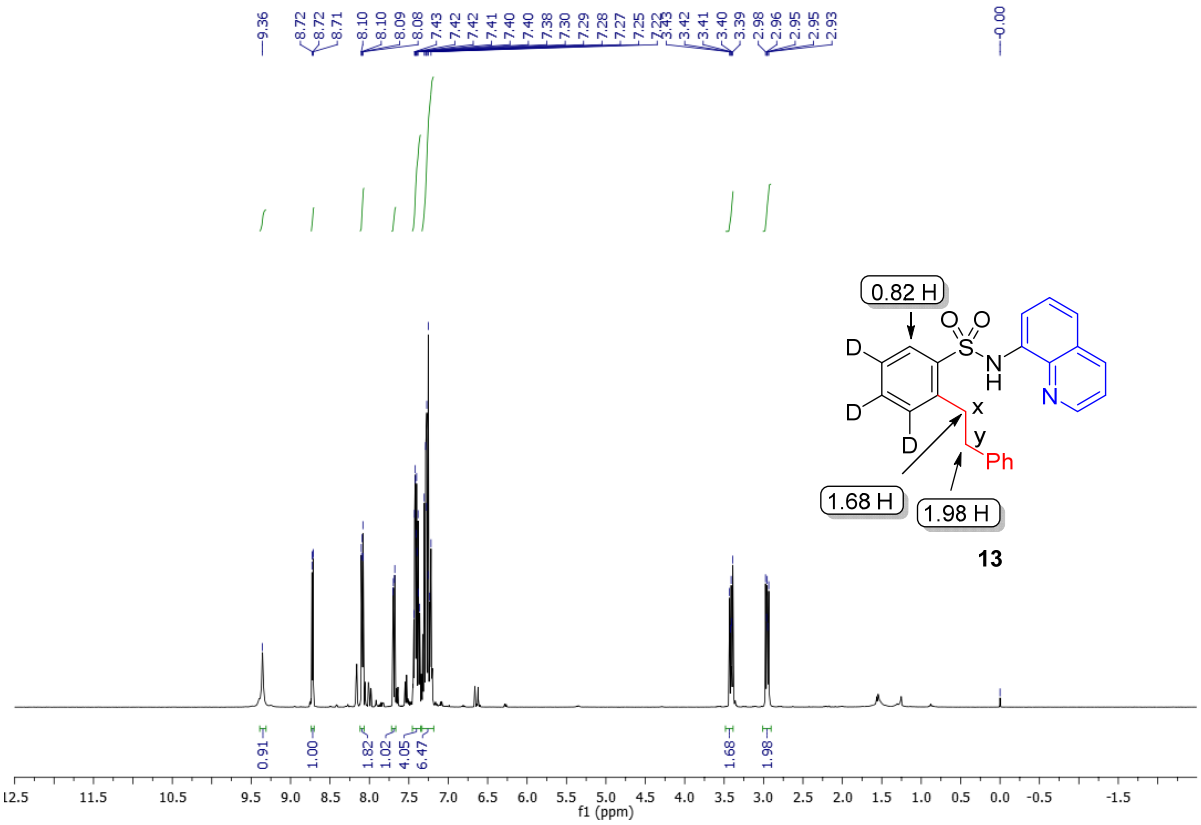
◆ *m*-Me was taken as a standardized peak.

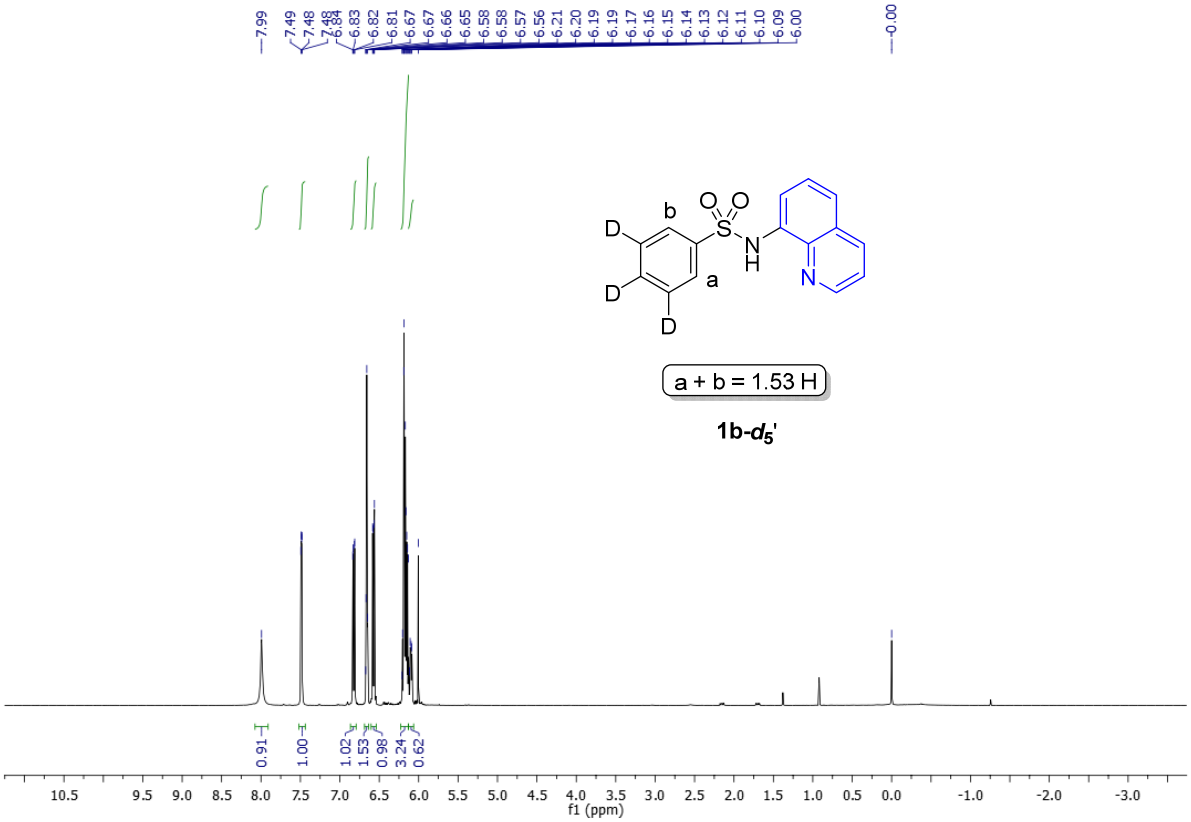


◆ CD_2Cl_2 (internal standard)

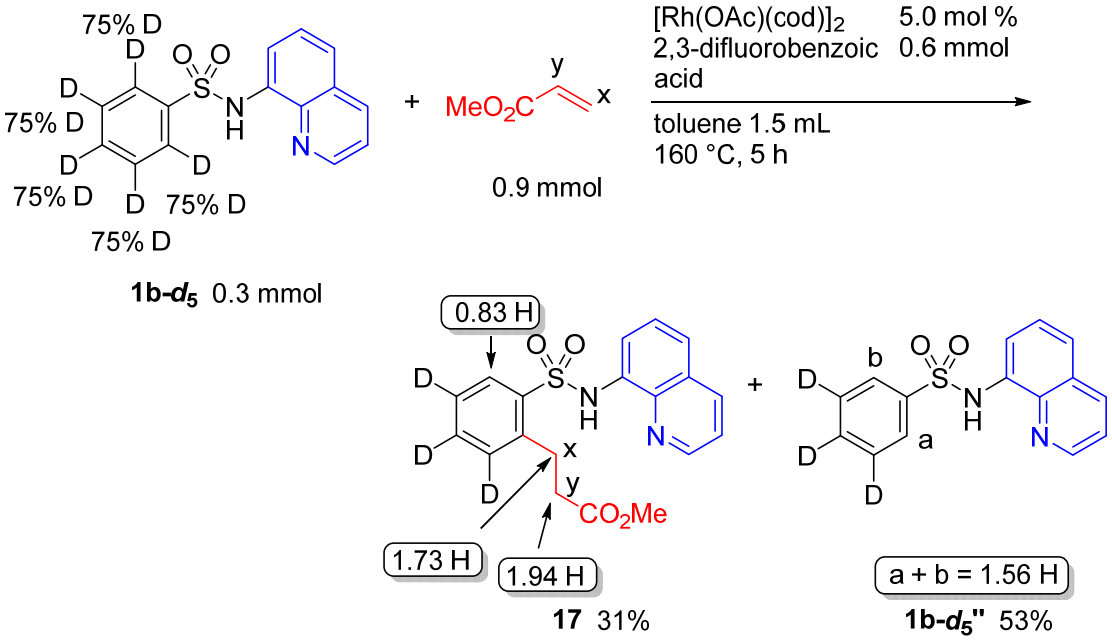
Reaction of sulfonamide 1b-d_5 and styrene-

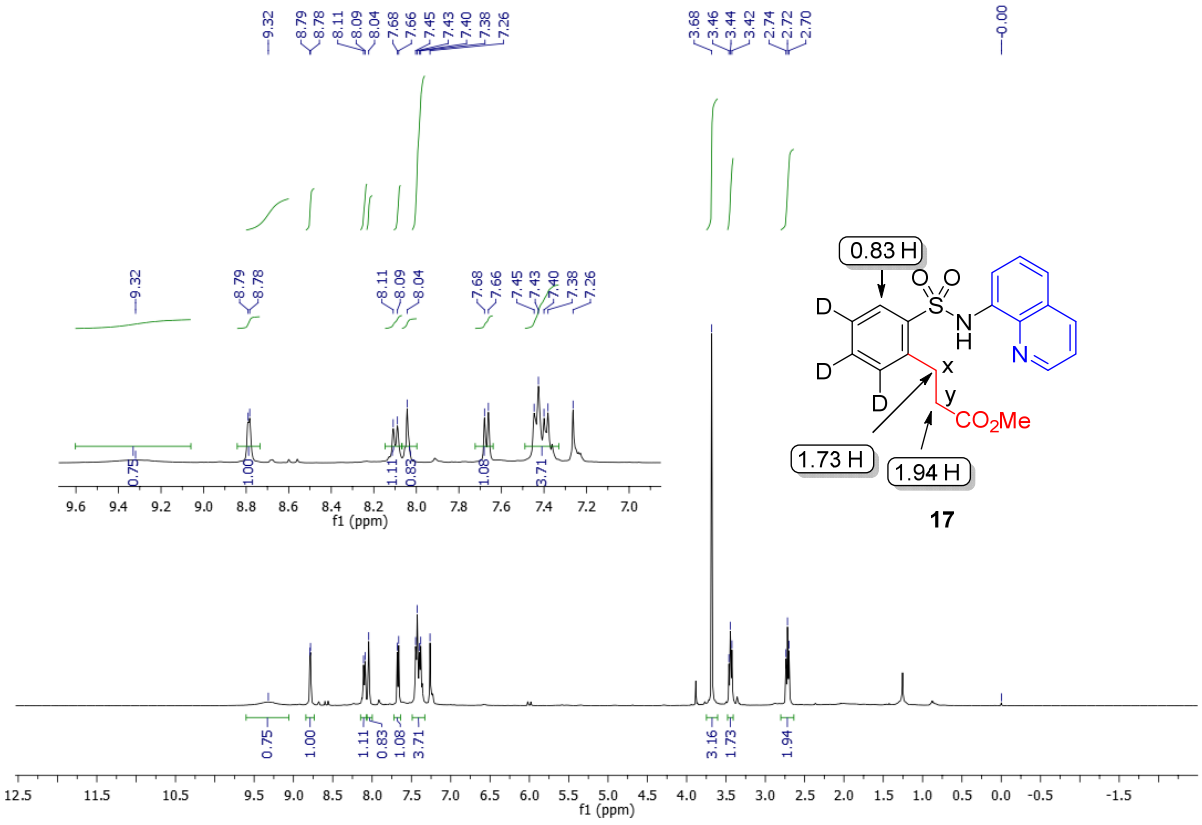


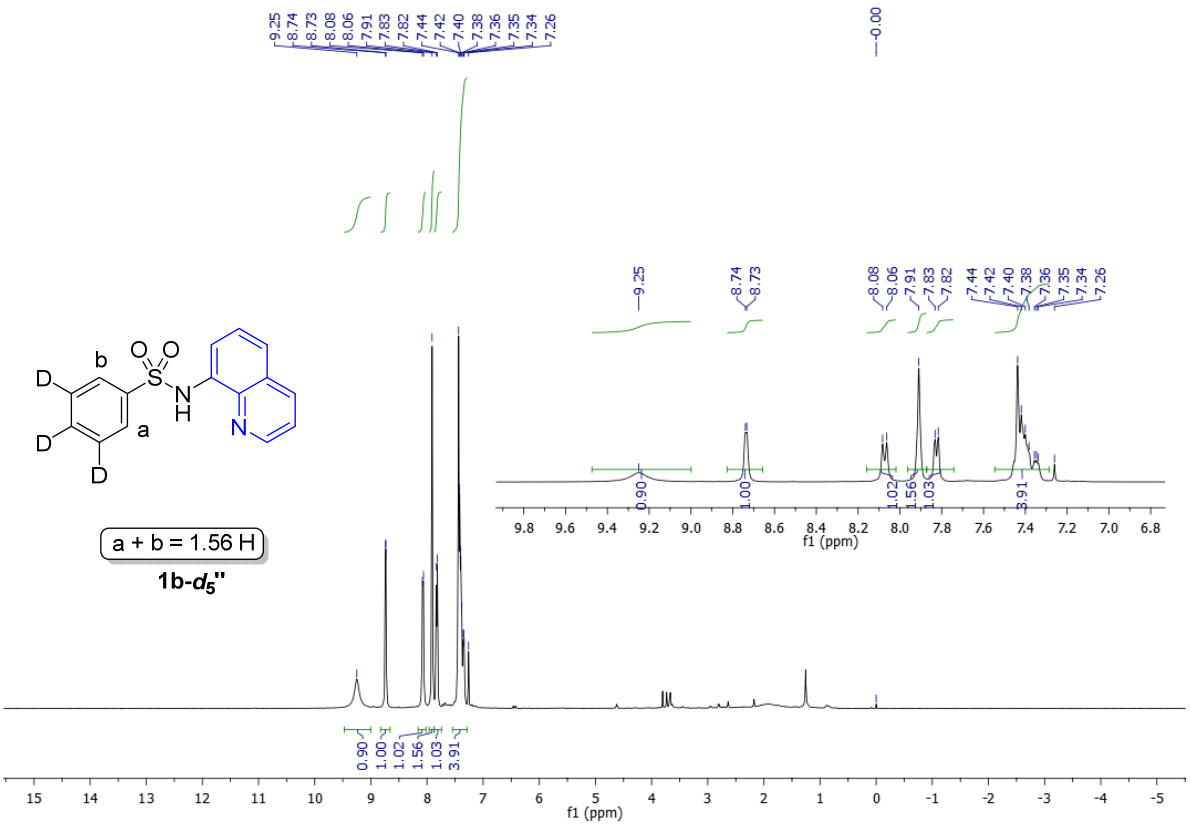



7.99
7.49
7.48
7.48
6.84
6.83
6.82
6.81
6.67
6.67
6.66
6.65
6.58
6.58
6.57
6.56
6.21
6.20
6.19
6.19
6.17
6.16
6.15
6.14
6.13
6.12
6.11
6.10
6.09
6.00
0.00
b
a
D
D
D
O
O
S
N
H
N
a + b = 1.53 H
1b-d5'
0.91
1.00
1.02
1.53
0.98
3.24
0.62
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-1.0
-2.0
-3.0
f1 (ppm)

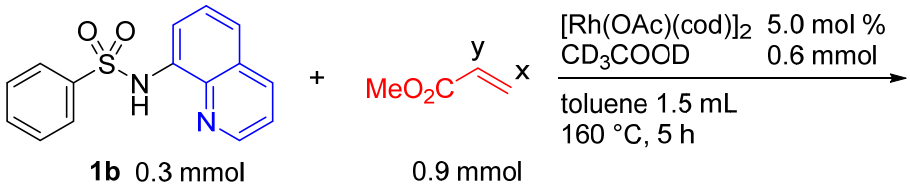
Reaction of sulfonamide 1b-d_5 and methyl acrylate-

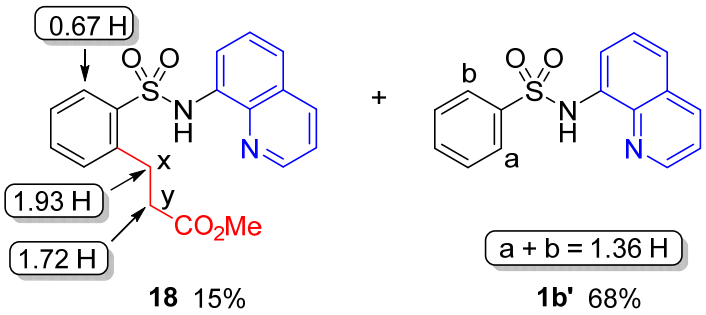


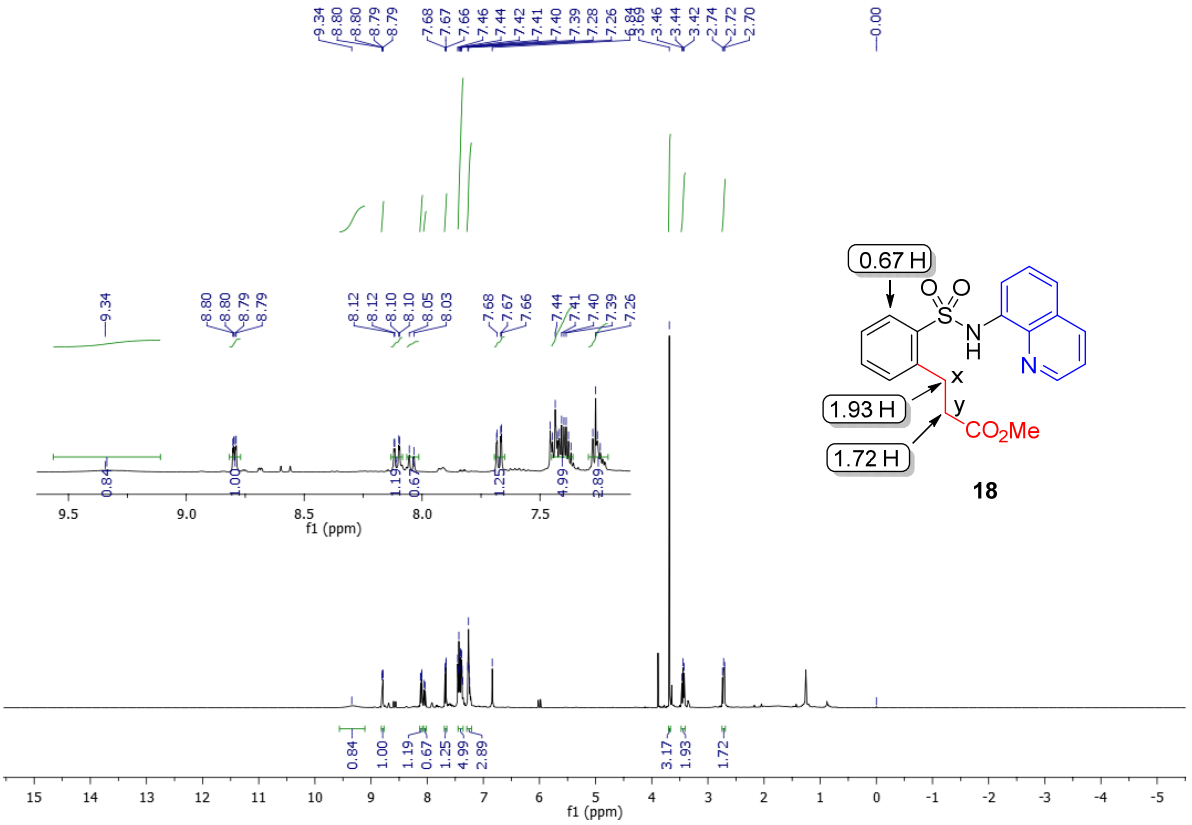


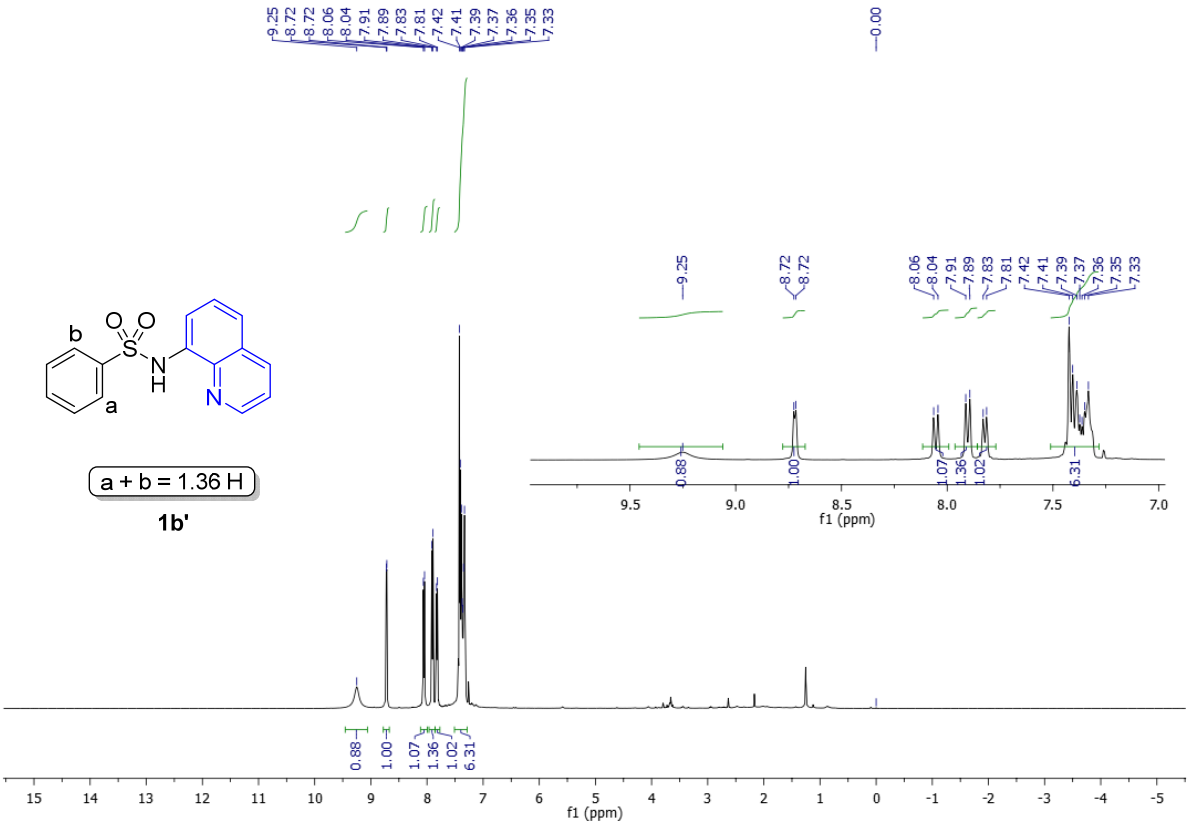

9.25
8.74
8.73
8.08
8.06
7.91
7.83
7.82
7.44
7.42
7.40
7.38
7.36
7.35
7.34
7.26
0.00
b
a
D
D
D
O
O
S
N
H
N
a + b = 1.56 H
1b-d5''
0.90
1.00
1.02
1.56
1.03
3.91
f1 (ppm)

Reaction of sulfonamide 1b and methyl acrylate in the presence of deuterated acid-

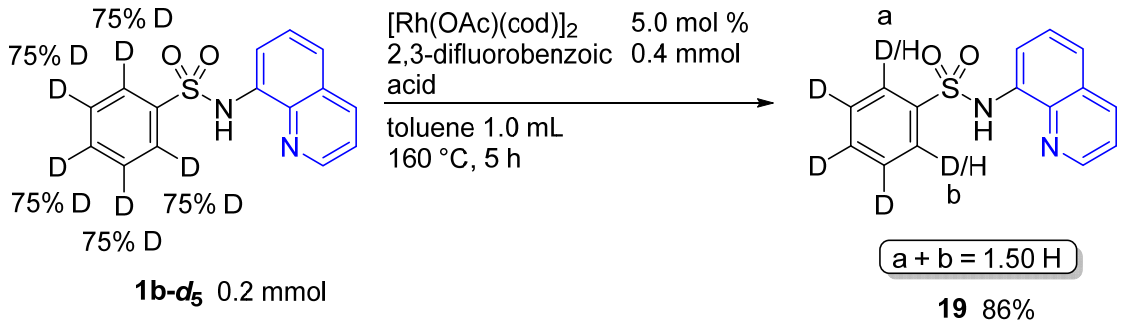






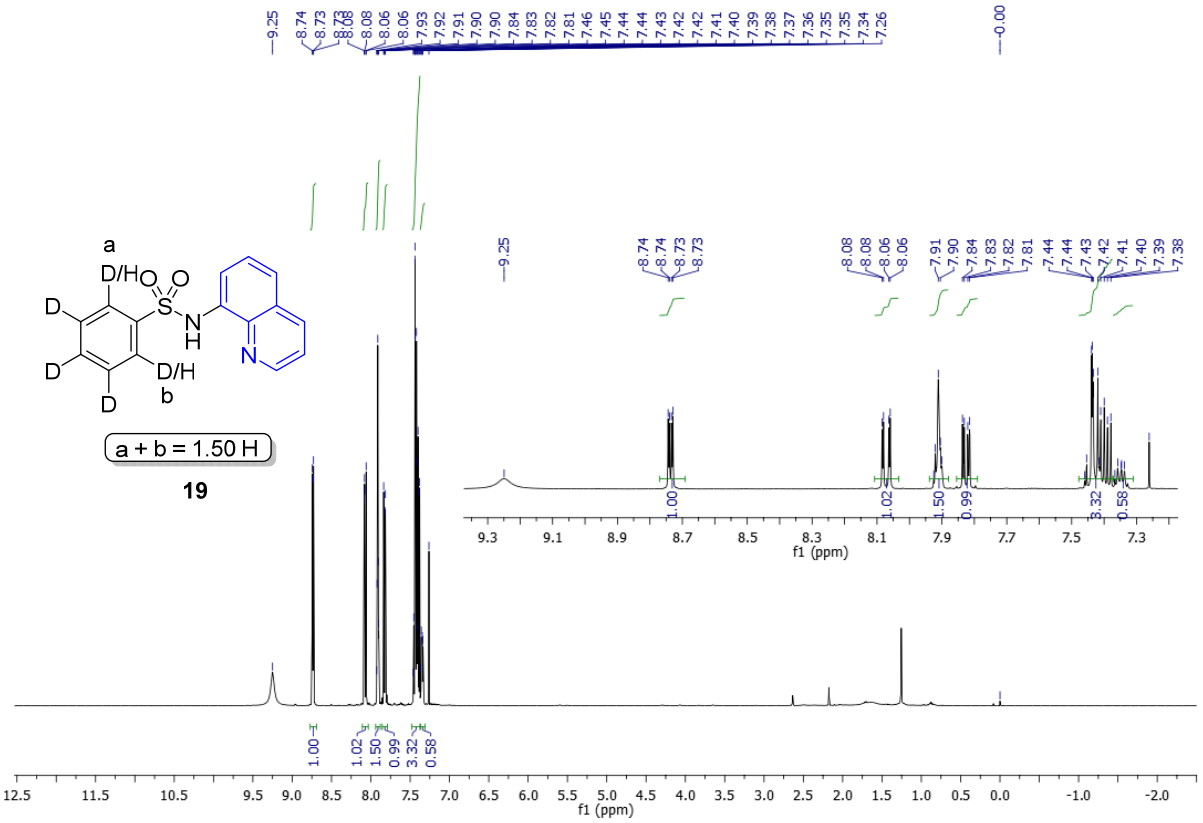

9.25
8.72
8.72
8.06
8.04
7.91
7.89
7.83
7.81
7.42
7.41
7.39
7.37
7.36
7.35
7.33
0.00
b
a
a + b = 1.36 H
1b'
0.88
1.00
1.07
1.36
1.02
6.31
f1 (ppm)

Reaction of sulfonamide 1b-d_5 with 2,3-difluorobenzoic acid-

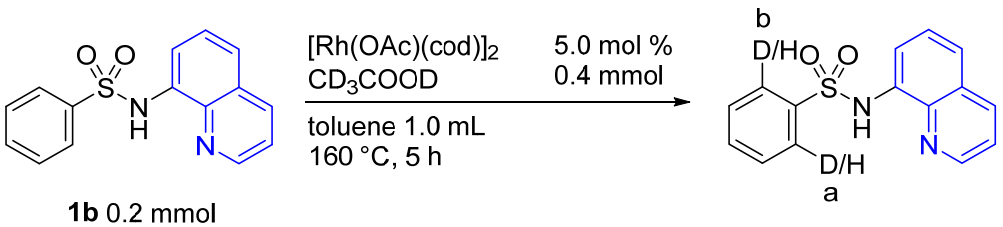


1b-d_5 0.2 mmol

19 86%



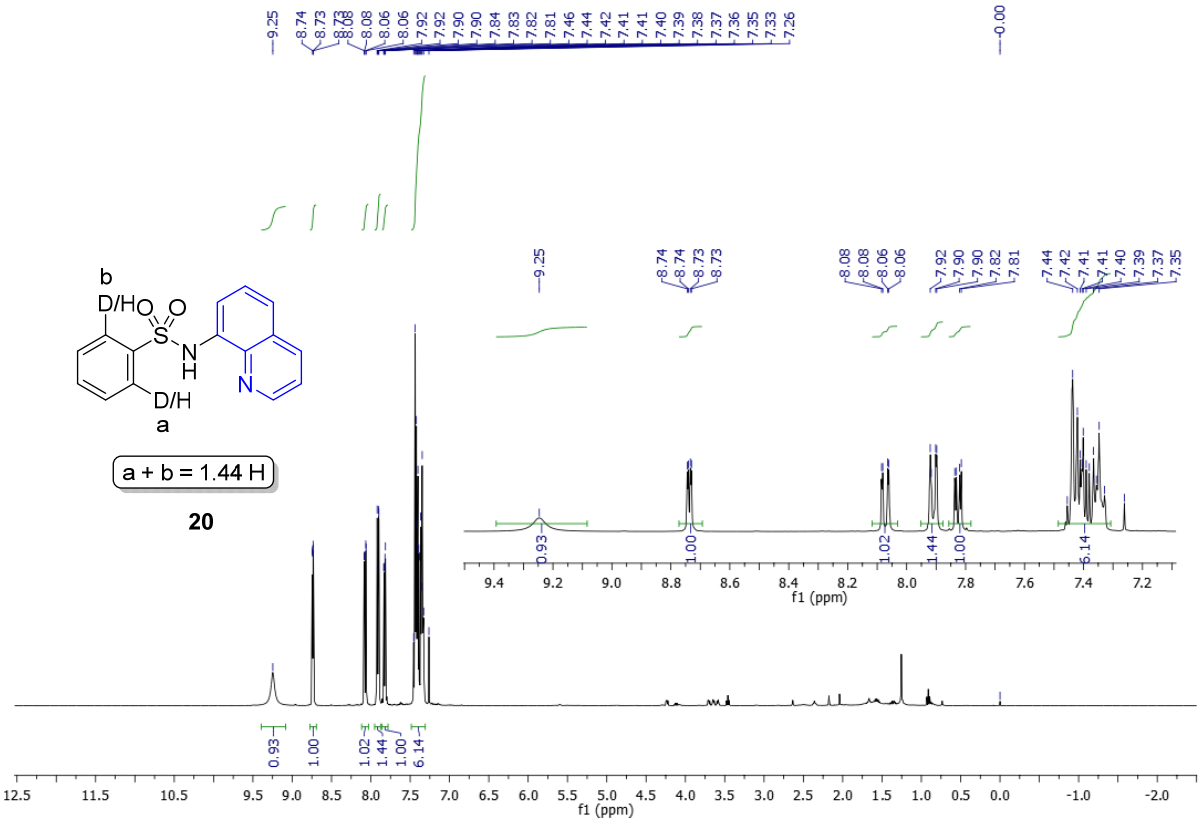
Reaction of sulfonamide 1b with CD_3COOD-



1b 0.2 mmol

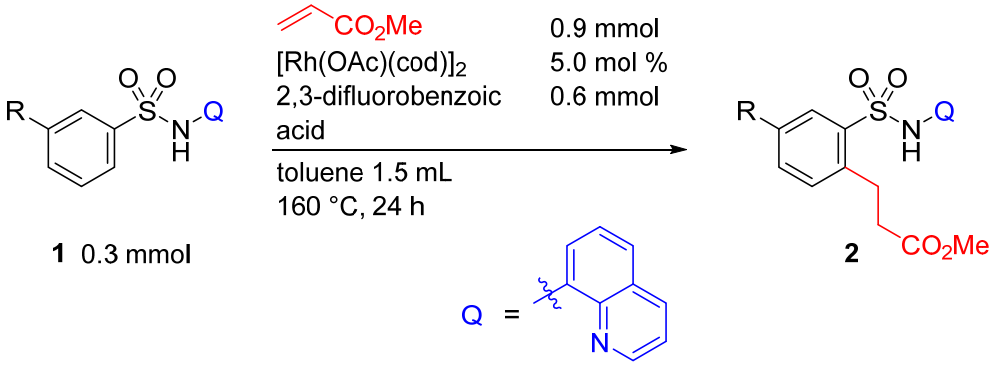
a + b = 1.44 H

20 84%



12. Hammett plot

Four Schelenk tubes containing sulfonamide **1a** (56.9 mg, 0.2 mmol, 1.0 equiv) and *meta*-substituted aryl sulfonamide derivatives **1i**, **1j**, **1k**, and **1n** (-OMe, -Me, -CF_3, and -CN) (0.2 mmol, 1.0 equiv) were reacted with methyl acrylate (54 µL, 51.3 mg. 0.6 mmol, 3.0 equiv) in the presence of $[Rh(OAc)(cod)]_2$ (5.4 mg, 0.01 mmol, 0.05 equiv) and 2,3-difluorobenzoic acid (63.2 mg, 0.4 mmol, 2.0 equiv) as an additive. Then, the reaction mixture was heated for 4 h at 160 °C in an oil bath. The final substrate concentrations for both the substrates were calculated by ^{1}H NMR spectroscopy by using mesitylene (28 µL, 24.2 mg, 0.2 mmol, 1.0 equiv) as an internal standard and then plotted log(conv. of R substituted styrene / conv. of styrene) against σ_ρ values of each substrate to obtain the Hammett plot (Figure S1). Obtained ρ value is negative (- 0.6182), suggested that the transition state is more stabilized by electron donating group substituted aryl sulfonamide, hence, a considerable cationic character projected on aryl sulfonamide in transition state.



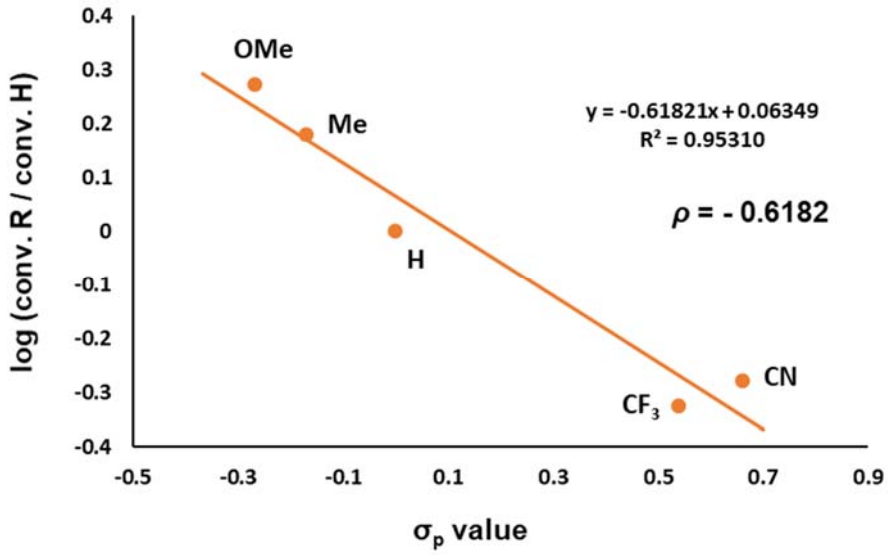
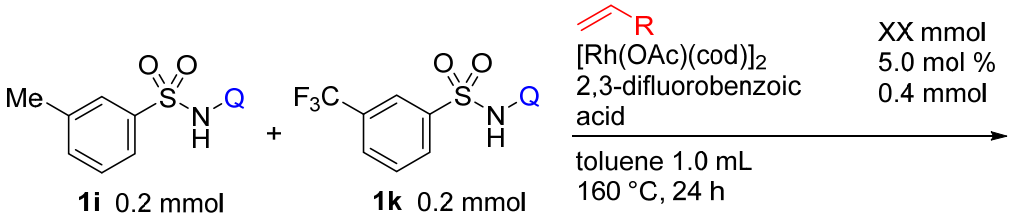
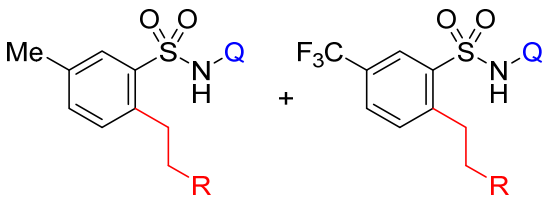


Figure S1. Representative plot of log(conv. of R-substituted sulfonamide / conv. of H-substituted sulfonamide) against σ_p values of each substrate. Obtained ρ value is - 0.6182.

13. Control experiments

Three Schelenk tubes containing sulfonamide **1i** (59.7 mg, 0.2 mmol, 1.0 equiv) and **1k** (70.5 mg, 0.2 mmol, 1.0 equiv) were reacted with three different set of olefins such as methyl acrylate (54 μL, 51.3 mg. 0.6 mmol, 3.0 equiv), styrene (92 μL, 83.6 mg. 0.8 mmol, 4.0 equiv), and 1-hexene (125 μL, 84.1 mg. 1.0 mmol, 5.0 equiv) in the presence of $[Rh(OAc)(cod)]_2$ (5.4 mg, 0.01 mmol, 0.05 equiv) and 2,3-difluorobenzoic acid (63.2 mg, 0.4 mmol, 2.0 equiv) as an additive. Then, the reaction mixture was heated for 24 h at 160 °C in an oil bath. The final product concentrations for both the substrates were calculated by ^{1}H NMR spectroscopy by using mesitylene (28 μL, 24.2 mg, 0.2 mmol, 1.0 equiv) as an internal standard.



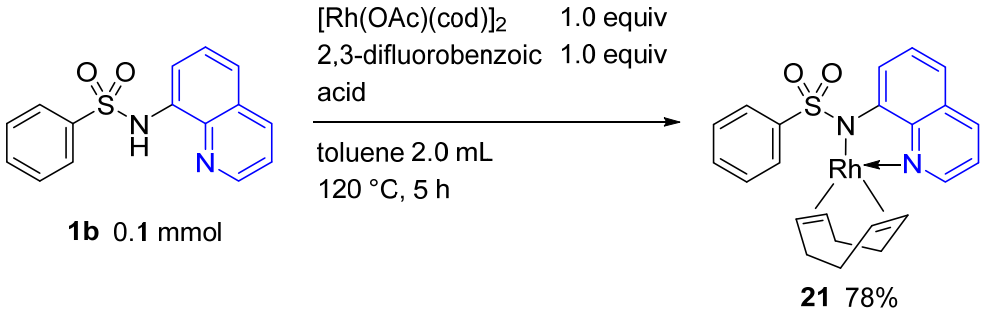


R = CO_2Me; XX = 0.5 mnmol	**2ia**	32%	**2ka**	23%
R = Ph; XX = 0.8 mnmol	**2if**	14%	**2kf**	11%
R = *n*Bu;[a] XX = 1.0 mnmol	**2ig**	55%	**2kg**	41%

[a] mixture of linear and branch isomer.

14. Synthesis of Rh-sulfonamide complex

In a J-young Schlenk tube excess Rh-precursor of $[Rh(OAc)(cod)]_2$ (54 mg, 0.1 mmol, 1.0 equiv) was mixed with sulfonamide **1b** (28.4 mg, 0.1 mmol, 1.0 equiv) and 2,3-difluorobenzoic acid (15.8 mg, 0.1 mmol, 1.0 equiv) in toluene (2 mL) under N_2 atmosphere. The reaction mixture was heated for 5 h at 120 °C. After that, the Schlenk tube was kept as it is without disturbing the solution phase. After 3 days red color crystals appeared, which was isolated and washed with hexane (5 mL).



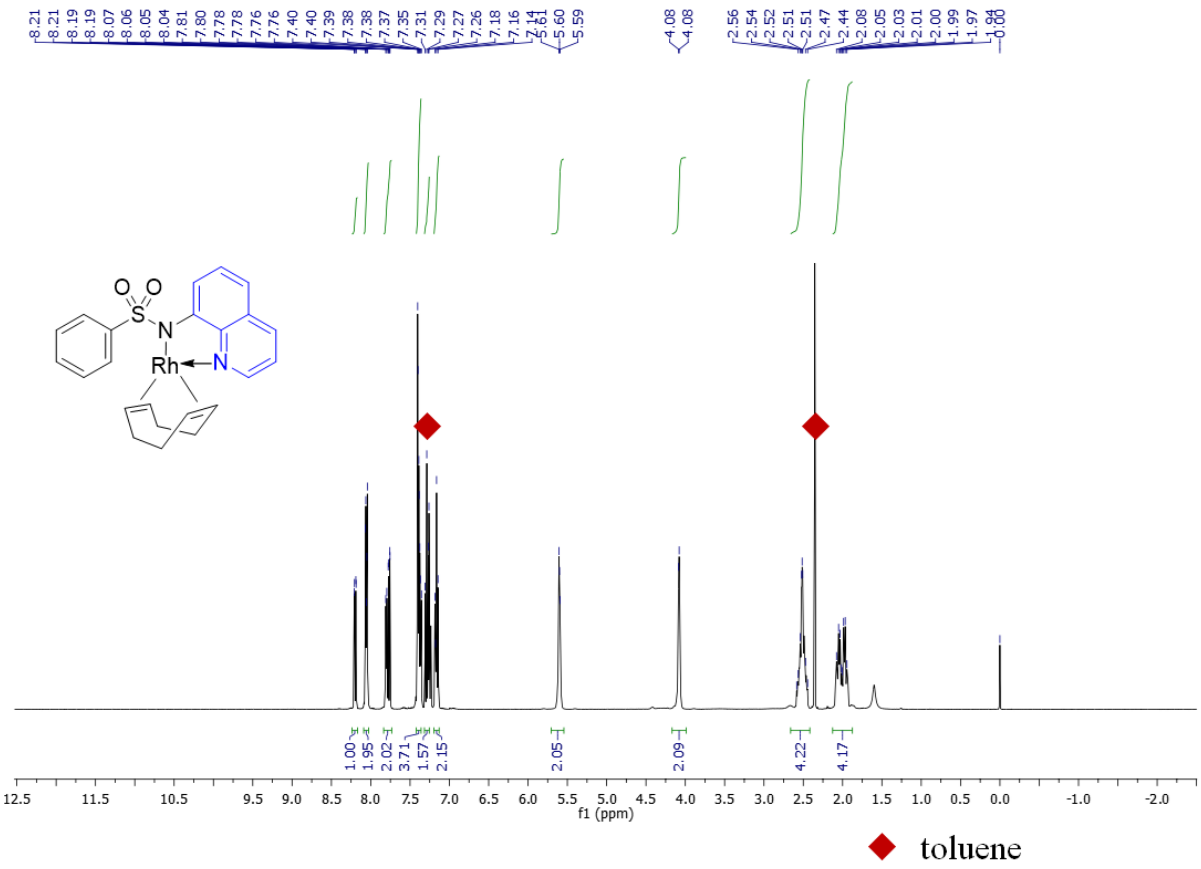


Figure S2. ^{1}H NMR spectra of Rh-sulfonamide complex **21** in $CDCl_3$.

A single crystal of **21** was obtained by the slow evaporation of a solution of benzene at room temperature. An orange prism crystal of **21** having approximate dimensions of 0.350 x 0.200 x 0.200 mm was mounted in a loop using epoxy glue and transferred to a Rigaku XtaLAB P200 diffractometer. Diffraction was measured using multi-layer mirror monochromated Cu-K radiation. X-ray structure details are given below.

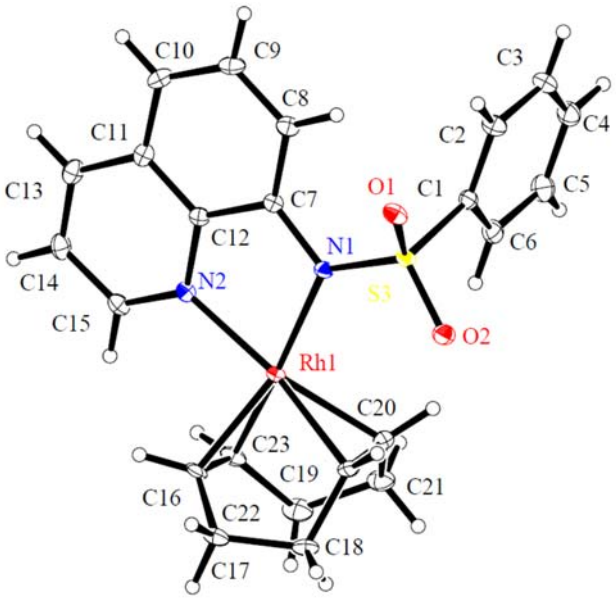


Figure S3. ORTEP Diagram (thermal ellipsoid plot) of Rh-sulfonamide complex **21** in 30% probability level. Numbering of hydrogen atom has eliminated due to clarity. Disordered was observed in solvent molecules. Note: acetonitrile was observed as one of the solvent molecule which incorporated as impurity.

CCDC–1878089 contains the supplementary crystallographic data of compound Rh-sulfonamide complex. X-Ray crystallographic structure analysis of sulfonamide coordinated Rh-complex was performed on Rigaku XtaLAB P200 diffractometer using multi-layer mirror monochromated Cu-Kα radiation. The data were collected at a temperature of –150 ± 1 °C to a maximum 2θ value of 148.5°. A total of 4460 oscillation images were collected. A sweep of data was done using ω scans from 1.0 to 29.0° in 0.50° step, at χ=62.0° and ϕ = -57.0°. The exposure rate was 0.1 [sec./°]. The detector swing angle was –42.80°. The structure was solved by direct methods (SIR92)[6] and expanded using Fourier techniques. Some non-hydrogen atoms were refined anisotropically, while

the rest were refined isotropically. Hydrogen atoms were refined using the riding model. The crystal data are mentioned below:

Crystal Data:

Empirical Formula	$C_{101}H_{103}N_9O_8Rh_4S_4$
	$4(C_{23}H_{23}N_2O_2RhS)\ (C_2H_3N)\ (C_7H_8)$
Formula Weight	2110.84
Crystal Color, Habit	orange, prism
Crystal Dimensions	0.350 X 0.200 X 0.200 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 11.8864(2) Å
	b = 11.90490(19) Å
	c = 18.32387(16)Å
	α = 96.2059(10) °
	β = 97.9083(11) °
	γ = 115.6023(16) °
	V = 2275.00(7) $Å^3$
Space Group	P-1 (#2)
Z value	1
D_{calc}	1.541 g/cm^3
F_{000}	1080.00
m(CuKa)	71.294 cm^{-1}

Table S3. Bond lengths (Å)

atom	atom	distance (Å)	atom	atom	distance (Å)
Rh1	N1	2.141(3)	Rh1	N2	2.088(4)
Rh1	C16	2.118(4)	Rh1	C19	2.185(5)
Rh1	C20	2.148(5)	Rh1	C23	2.124(4)
S3	O1	1.448(4)	S3	O2	1.442(3)
S3	N1	1.610(5)	S3	C1	1.781(5)
N1	C7	1.416(6)	N2	C12	1.379(5)
N2	C15	1.333(7)	C1	C2	1.384(6)
C1	C6	1.382(7)	C2	C3	1.392(8)
C3	C4	1.379(8)	C4	C5	1.385(7)
C5	C6	1.390(9)	C7	C8	1.386(5)
C7	C12	1.432(7)	C8	C9	1.403(7)
C9	C10	1.372(8)	C10	C11	1.411(6)
C11	C12	1.413(7)	C11	C13	1.409(8)
C13	C14	1.366(6)	C14	C15	1.395(8)
C16	C17	1.506(8)	C16	C23	1.410(8)
C17	C18	1.530(8)	C18	C19	1.511(6)
C19	C20	1.398(8)	C20	C21	1.514(8)
C21	C22	1.535(9)	C22	C23	1.529(7)

Table S4. Bond angles (°)

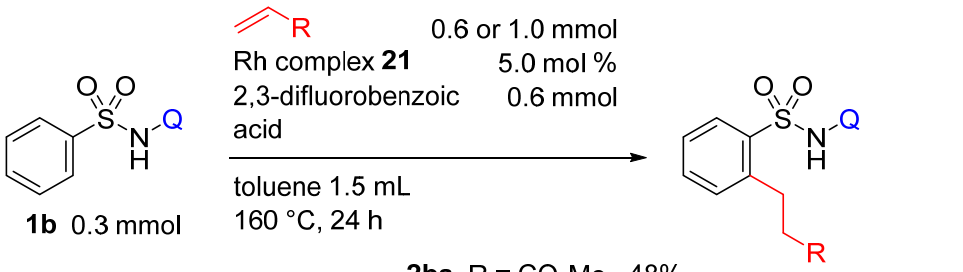
atom	atom	atom	angle (°)	atom	atom	atom	angle (°)
N1	Rh1	N2	78.69(15)	N1	Rh1	C16	155.56(17)
N1	Rh1	C19	102.79(18)	N1	Rh1	C20	99.68(17)
N1	Rh1	C23	162.5(2)	N2	Rh1	C16	92.02(17)
N2	Rh1	C19	163.80(19)	N2	Rh1	C20	158.6(2)
N2	Rh1	C23	93.93(18)	C16	Rh1	C19	80.10(19)
C16	Rh1	C20	97.01(17)	C16	Rh1	C23	38.8(2)
C19	Rh1	C20	37.6(2)	C19	Rh1	C23	88.8(2)
C20	Rh1	C23	81.3(2)	O1	S3	O2	116.3(3)
O1	S3	N1	112.7(2)	O1	S3	C1	106.8(2)
O2	S3	N1	108.3(2)	O2	S3	C1	105.9(2)
N1	S3	C1	106.2(3)	Rh1	N1	S3	129.6(2)
Rh1	N1	C7	113.0(3)	S3	N1	C7	117.4(3)
Rh1	N2	C12	115.2(3)	Rh1	N2	C15	126.8(3)
C12	N2	C15	117.9(4)	S3	C1	C2	121.1(4)
S3	C1	C6	118.2(3)	C2	C1	C6	120.6(5)
C1	C2	C3	119.1(5)	C2	C3	C4	120.7(4)
C3	C4	C5	119.8(6)	C4	C5	C6	120.0(5)
C1	C6	C5	119.8(4)	N1	C7	C8	127.3(5)
N1	C7	C12	115.8(3)	C8	C7	C12	116.9(4)
C7	C8	C9	121.4(5)	C8	C9	C10	122.1(4)
C9	C10	C11	118.6(5)	C10	C11	C12	119.6(5)

Table S4 (Continued). Bond angles (°)

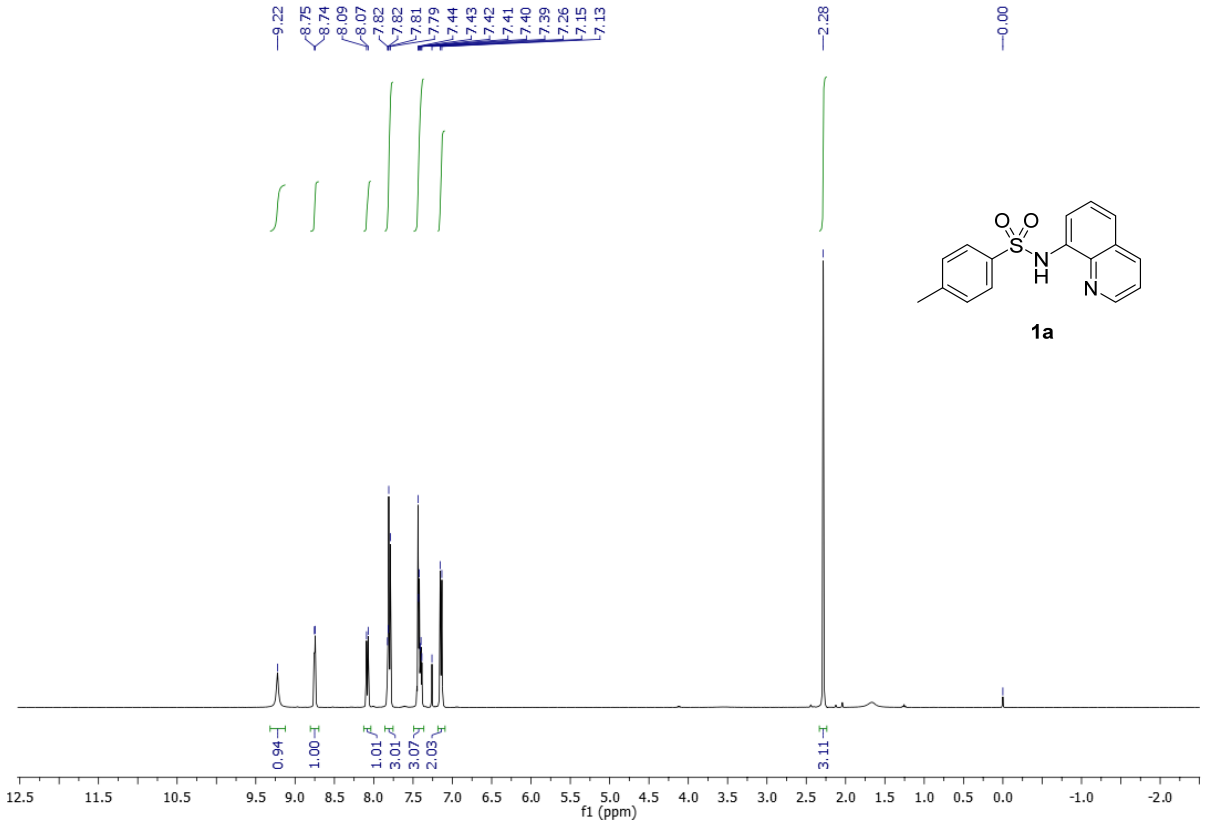
atom	atom	atom	angle (°)	atom	atom	atom	angle (°)
C10	C11	C13	122.0(5)	C12	C11	C13	118.4(4)
N2	C12	C7	117.4(4)	N2	C12	C11	121.3(5)
C7	C12	C11	121.3(4)	C11	C13	C14	119.5(5)
C13	C14	C15	119.0(5)	N2	C15	C14	123.8(4)
Rh1	C16	C17	111.6(4)	Rh1	C16	C23	70.8(3)
C17	C16	C23	126.8(4)	C16	C17	C18	112.1(4)
C17	C18	C19	111.2(4)	Rh1	C19	C18	113.2(4)
Rh1	C19	C20	69.8(3)	C18	C19	C20	124.0(4)
Rh1	C20	C19	72.6(3)	Rh1	C20	C21	109.8(4)
C19	C20	C21	125.4(4)	C20	C21	C22	112.6(4)
C21	C22	C23	111.4(4)	Rh1	C23	C16	70.4(3)
Rh1	C23	C22	114.1(4)	C16	C23	C22	123.5(5)

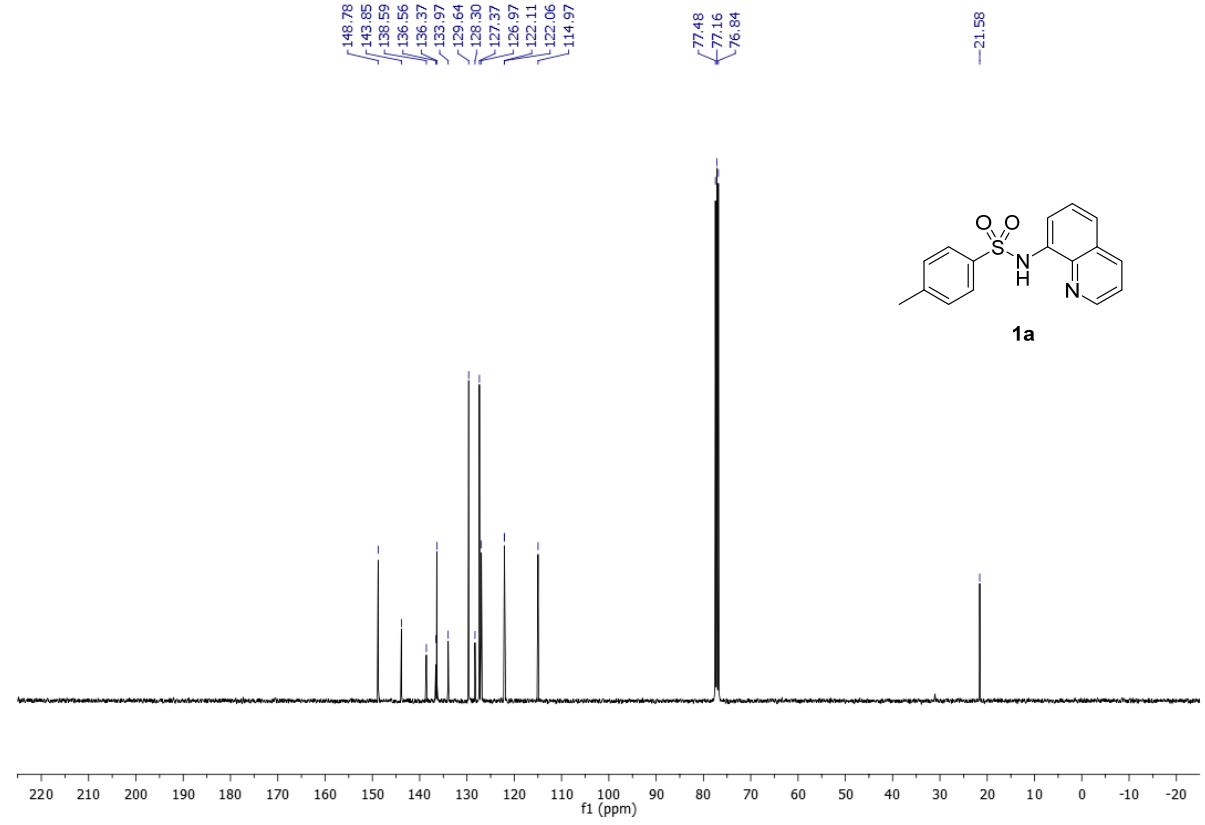
15. Alkylation reaction by using Rh-sulfonamide complex

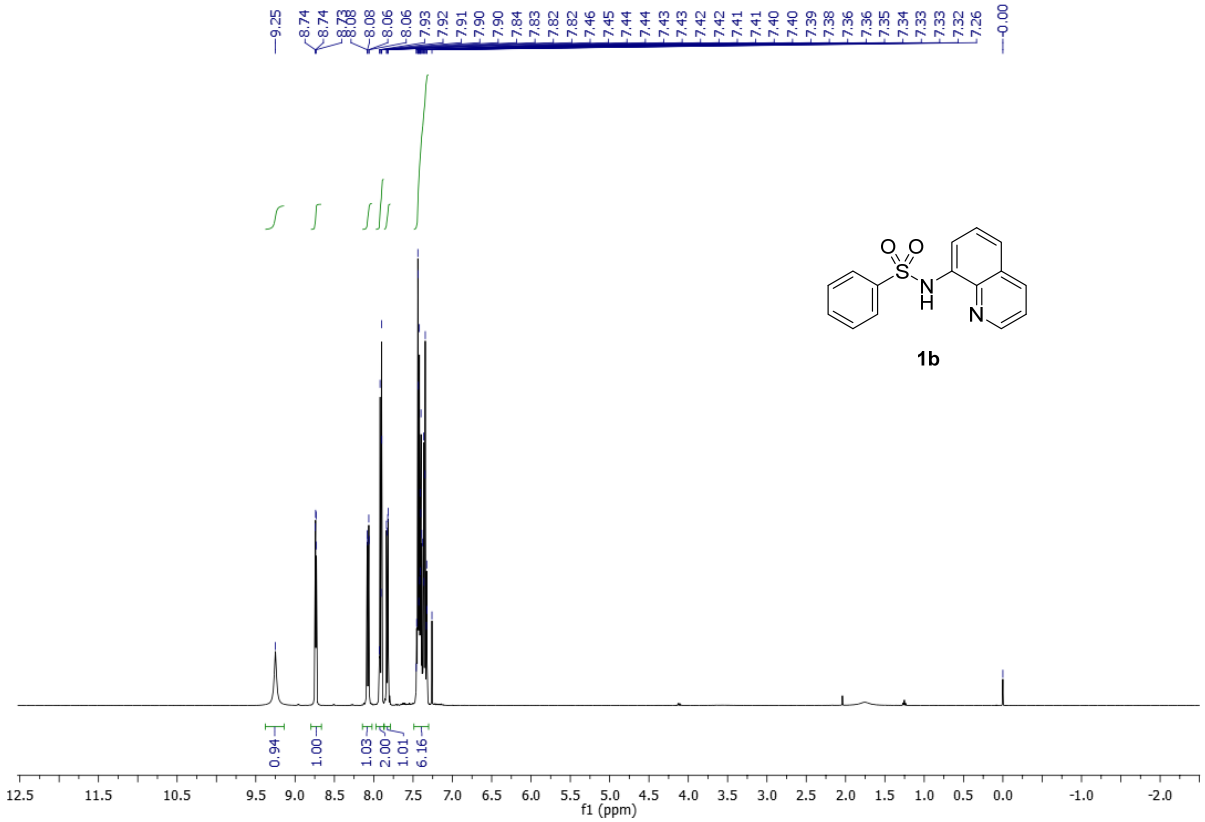
In an oven dried sealed tube, $[Rh(OAc)(cod)]_2$ (5.1 mg, 0.01 mmol, 0.05 equiv), sulfonamide **1b** (56.87 mg, 0.2 mmol, 1.0 equiv), and 2,3-difluorobenzoic acid (63.24 mg, 0.4 mmol, 2.0 equiv) were taken and 1.0 mL of toluene was added inside a glovebox. Into the reaction mixture proper olefin methyl acrylate (54.0 μL, 51.3 mg, 0.6 mmol, 3.0 equiv) or 1-hexene (103.0 μL, 83.8 mg, 1.0 mmol, 5.0 equiv) was added in inert atmosphere and the reaction mixture was kept for 24 h at 160 °C temperature in an oil bath.


R
0.6 or 1.0 mmol
Rh complex 21
5.0 mol %
2,3-difluorobenzoic acid
0.6 mmol
toluene 1.5 mL
160 °C, 24 h
1b 0.3 mmol
2ba R = CO2Me, 48%
2bg R = nBu, 80% (linear/branch = 62/38)

16. NMR spectra of starting materials



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8.06
8.06
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7.92
7.91
7.90
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7.84
7.83
7.82
7.82
7.46
7.45
7.44
7.44
7.43
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.39
7.38
7.36
7.36
7.35
7.34
7.33
7.33
7.32
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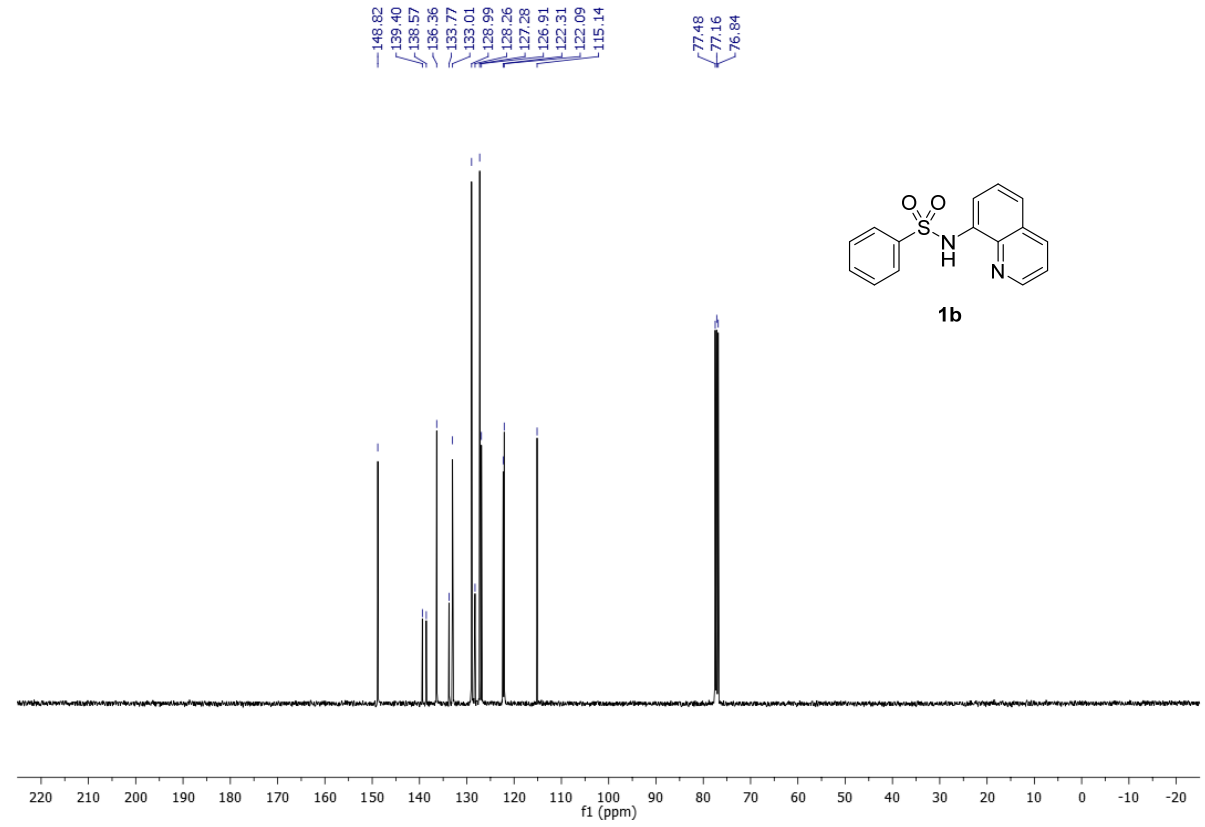
1.00
1.03
2.00
1.01
6.16
f1 (ppm)
1b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133.01
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122.31
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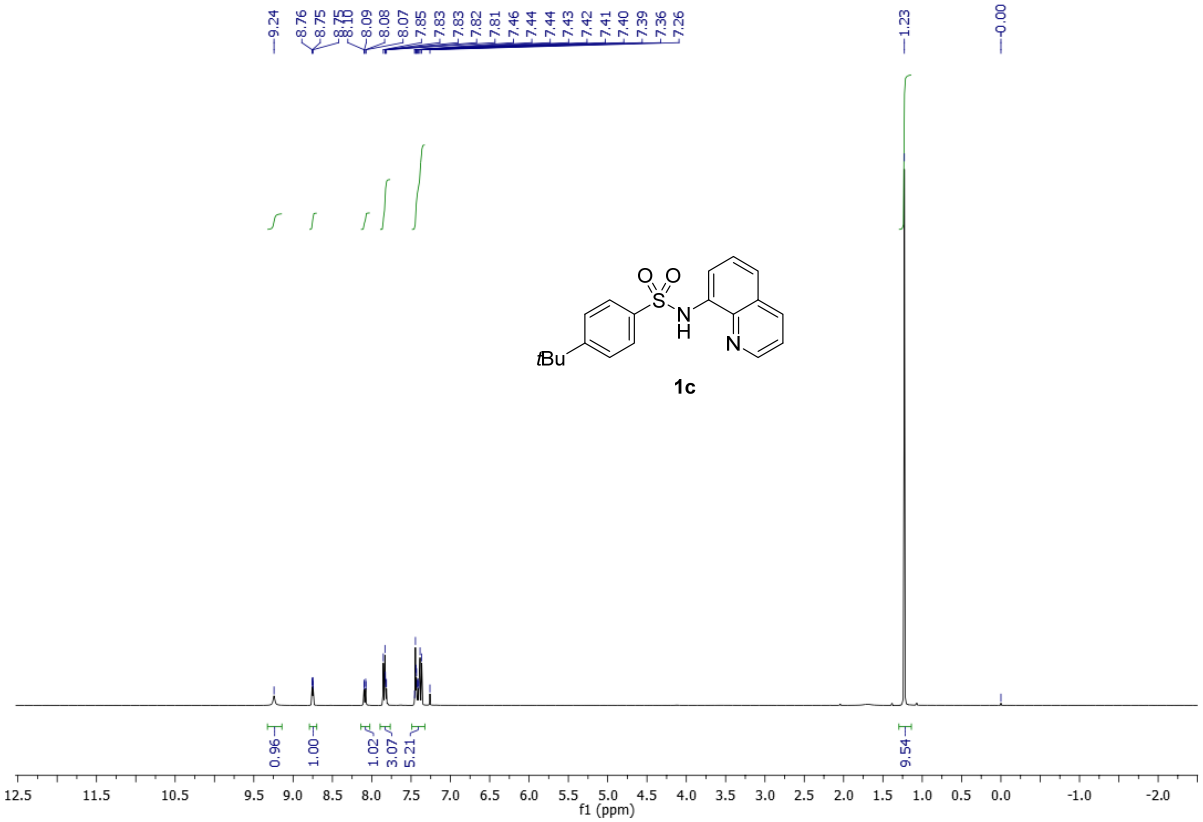
77.48
77.16
76.84
f1 (ppm)
1b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8.08
8.07
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7.82
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7.46
7.44
7.44
7.43
7.42
7.41
7.40
7.39
7.36
7.26
1.23
0.00
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O
S
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H
N
tBu
1c
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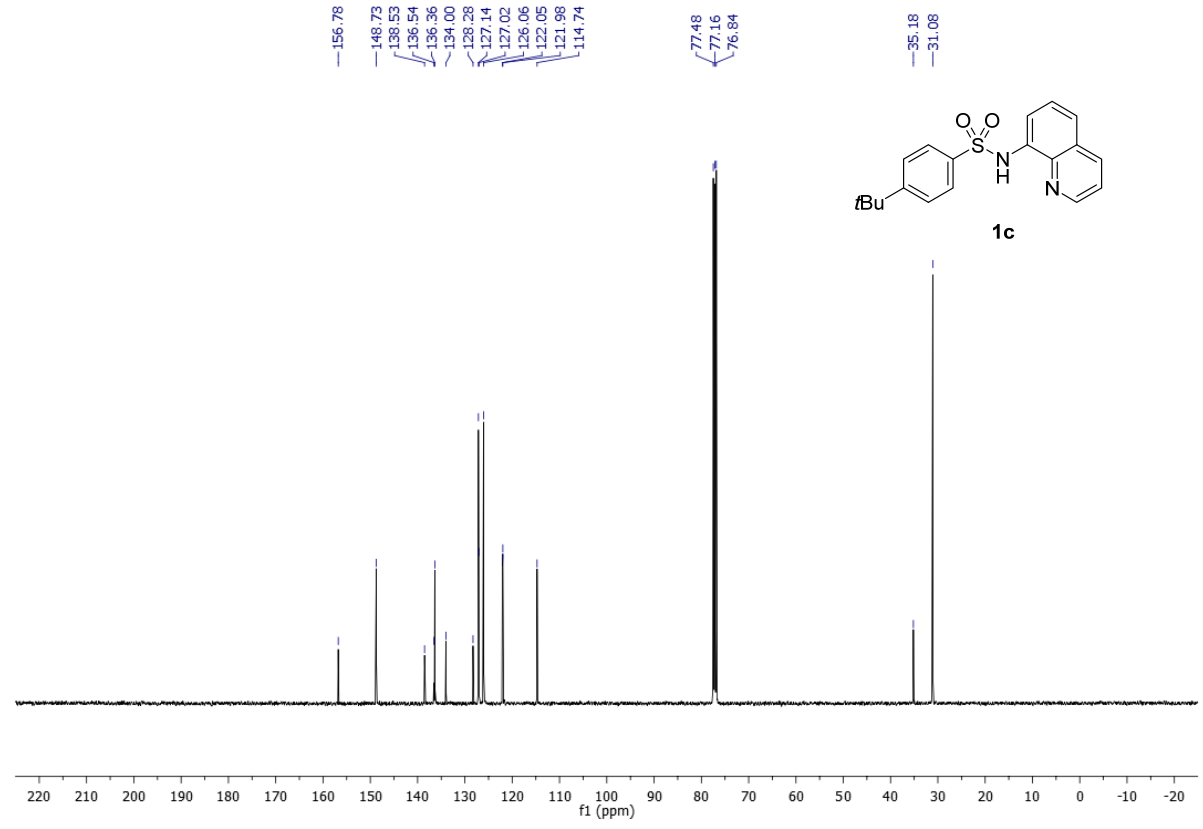
3.07
5.21
9.54
f1 (ppm)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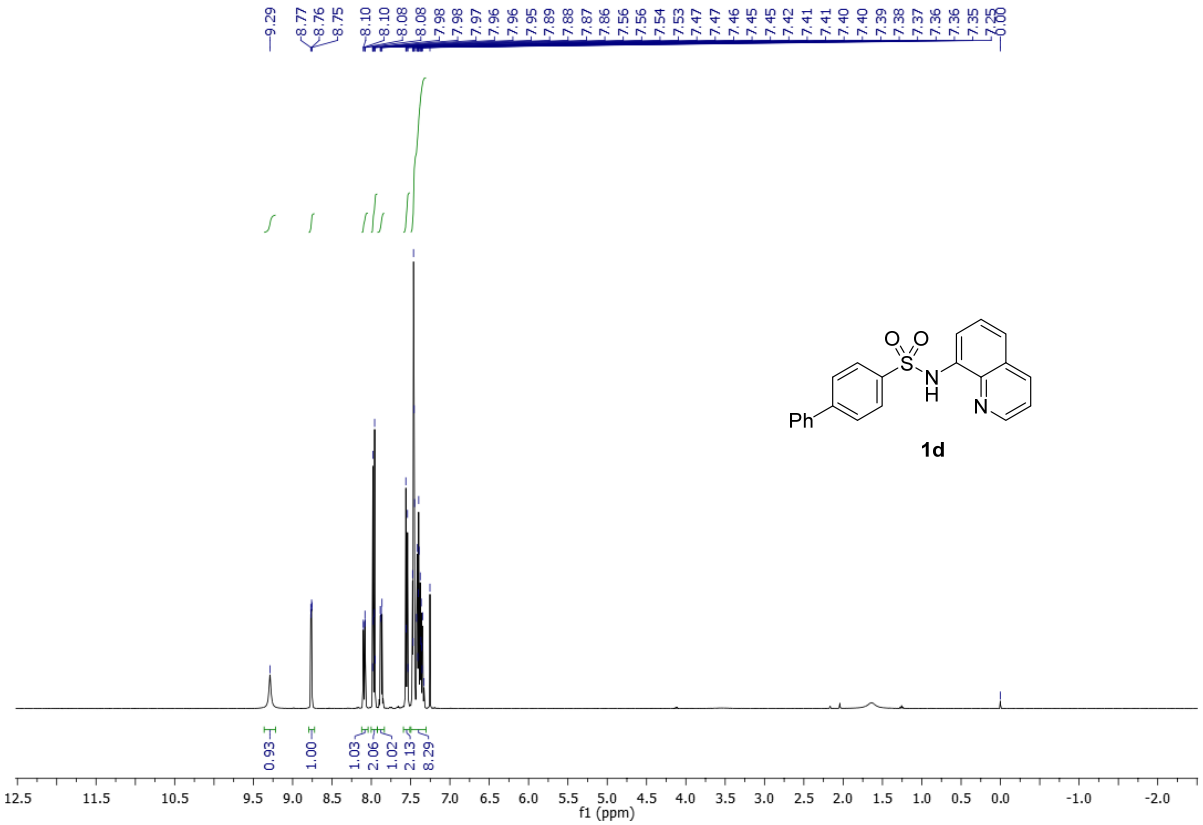
76.84
35.18
31.08
O
O
S
N
H
N
tBu
1c
f1 (ppm)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8.08
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7.96
7.95
7.89
7.88
7.87
7.86
7.56
7.56
7.54
7.53
7.47
7.47
7.46
7.45
7.45
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7.41
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7.39
7.38
7.37
7.36
7.36
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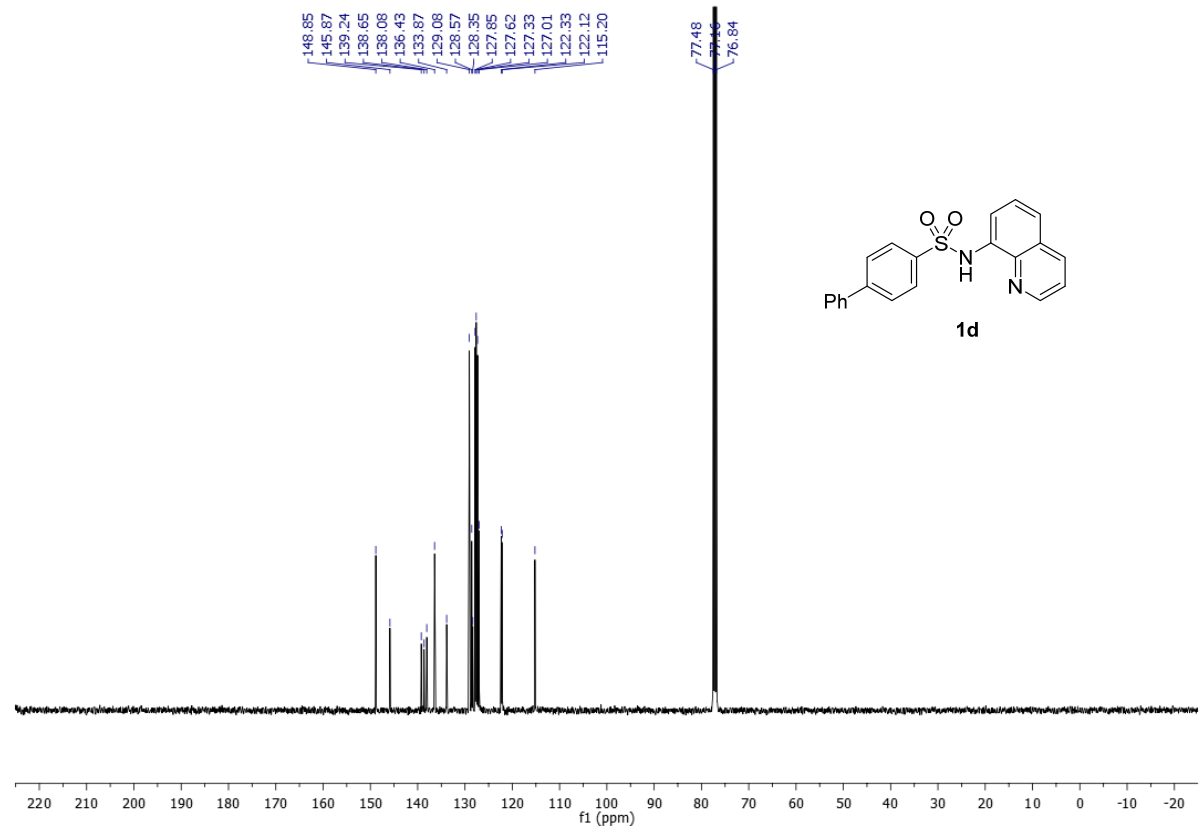
2.13
8.29
f1 (ppm)
Ph
1d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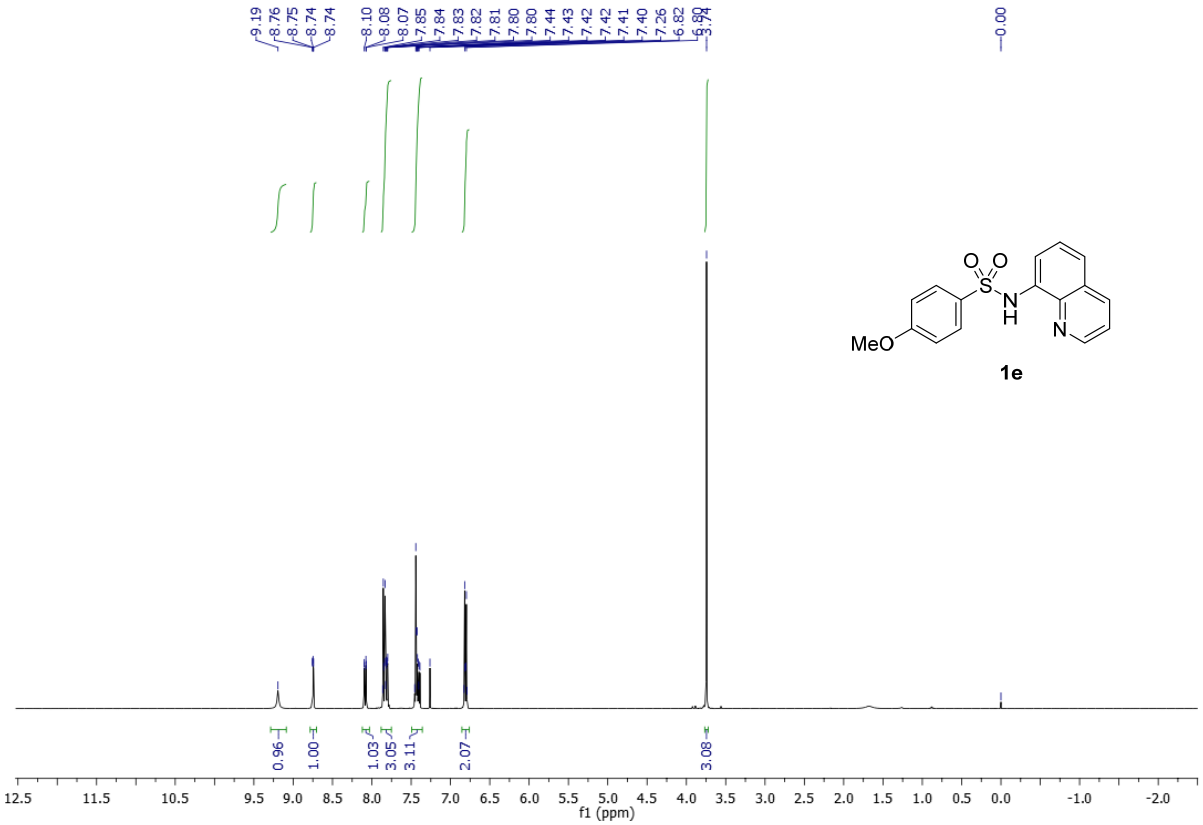
77.16
76.84
f1 (ppm)
Ph
1d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7.80
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7.43
7.42
7.42
7.41
7.40
7.26
6.82
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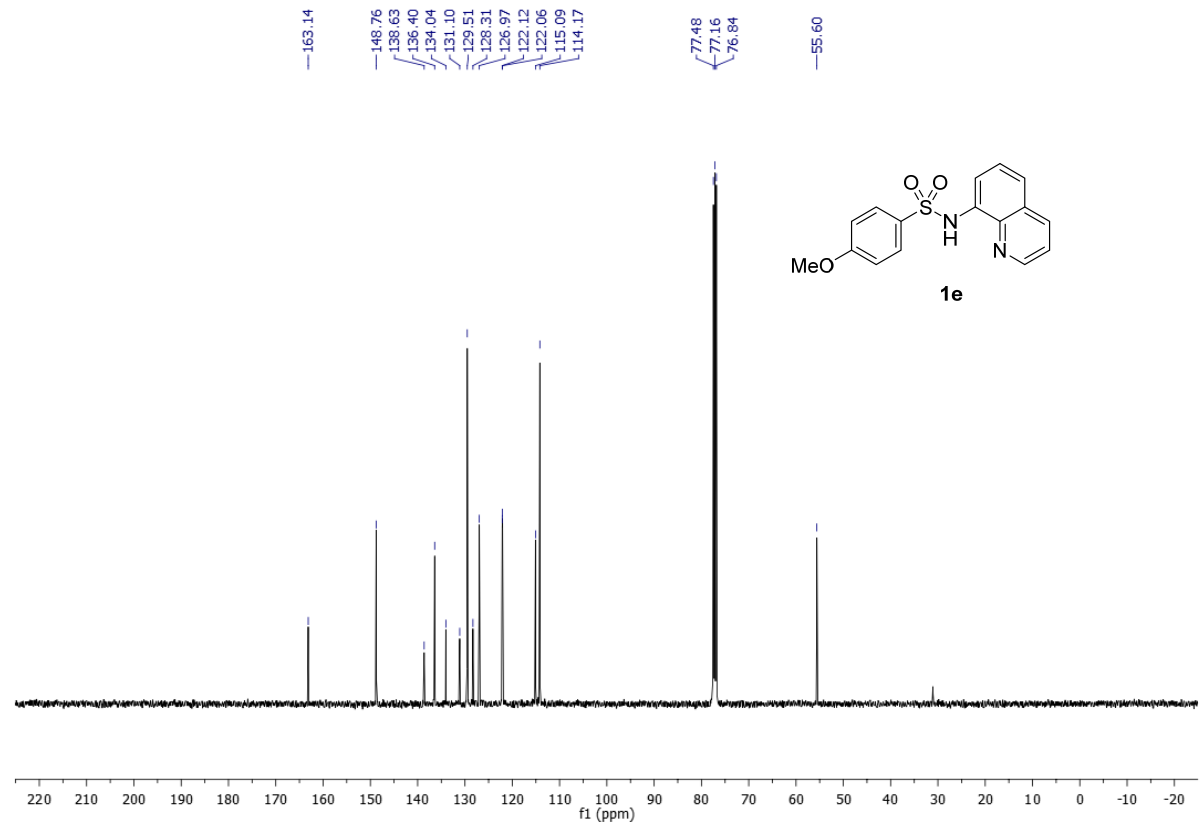
2.07
3.08
MeO
1e
f1 (ppm)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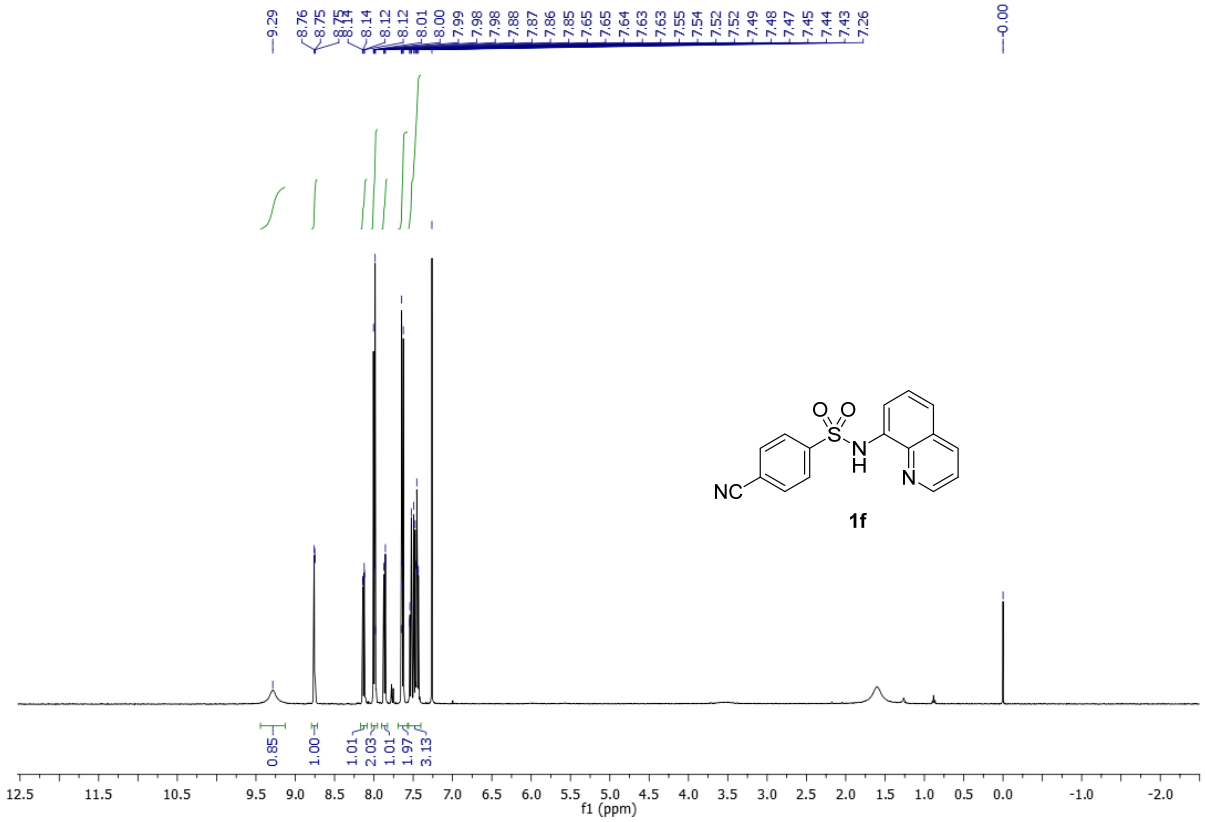
76.84
55.60
MeO
1e
f1 (ppm)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7.65
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7.63
7.55
7.54
7.52
7.49
7.48
7.47
7.45
7.44
7.43
7.26
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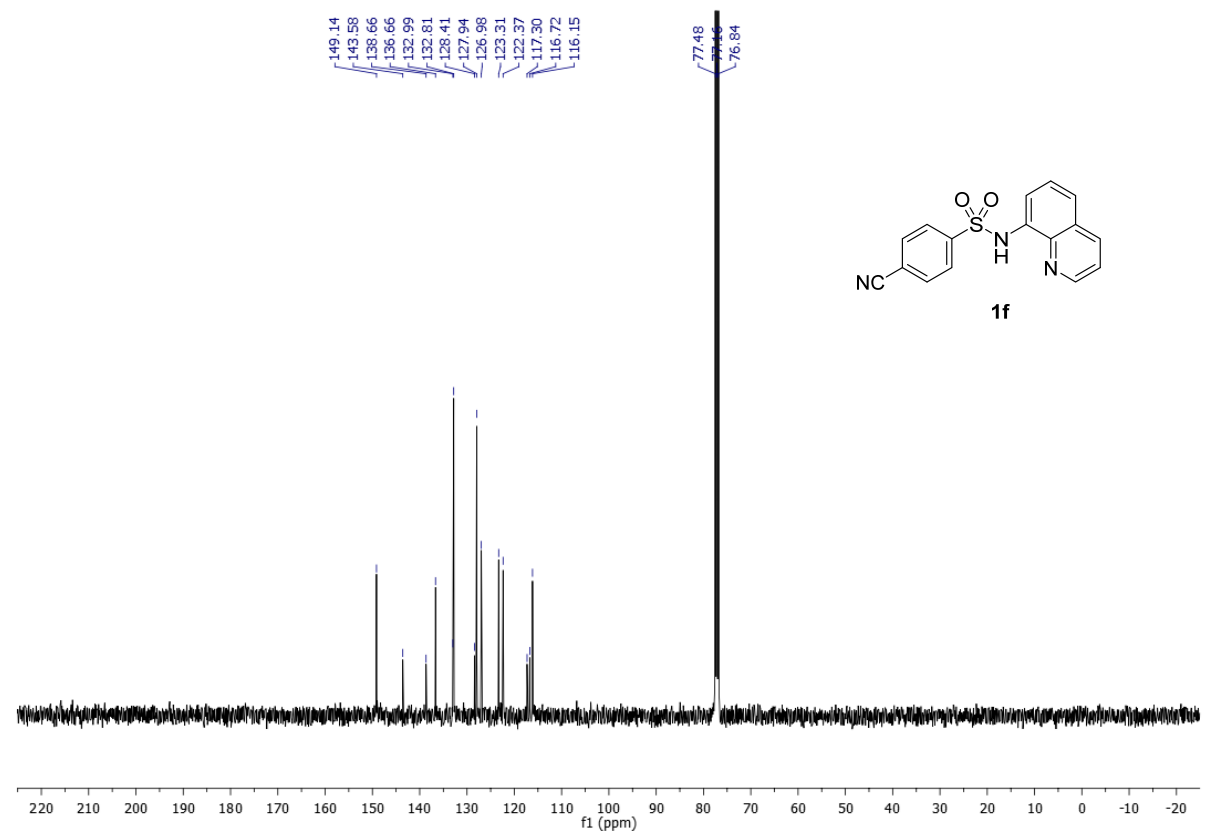
1.00
1.01
2.03
1.01
1.97
3.13
f1 (ppm)
NC
1f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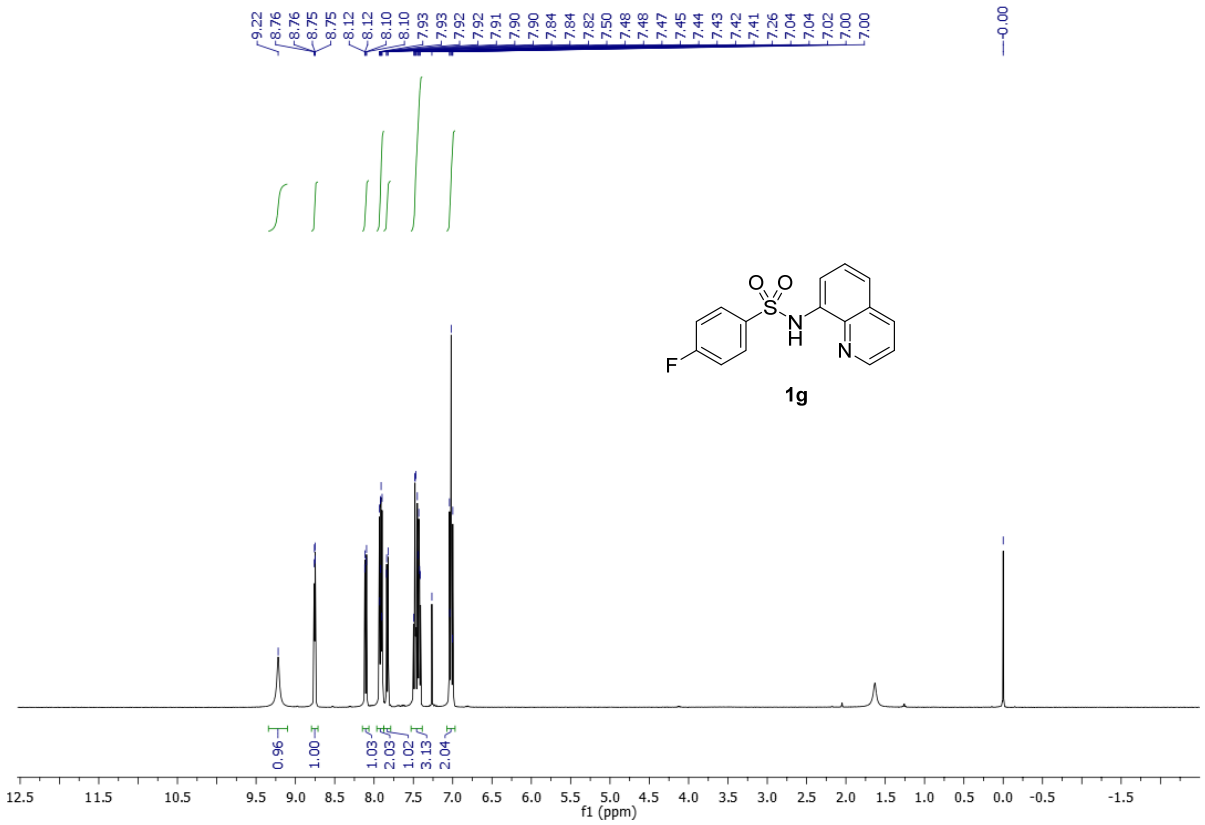
77.16
76.84
f1 (ppm)
NC
1f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7.47
7.45
7.44
7.43
7.42
7.41
7.26
7.04
7.04
7.02
7.00
7.00
0.00
O
O
S
N
H
F
N
1g
0.96
1.00
1.03
2.03
1.02
3.13
2.04
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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2.5
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0.0
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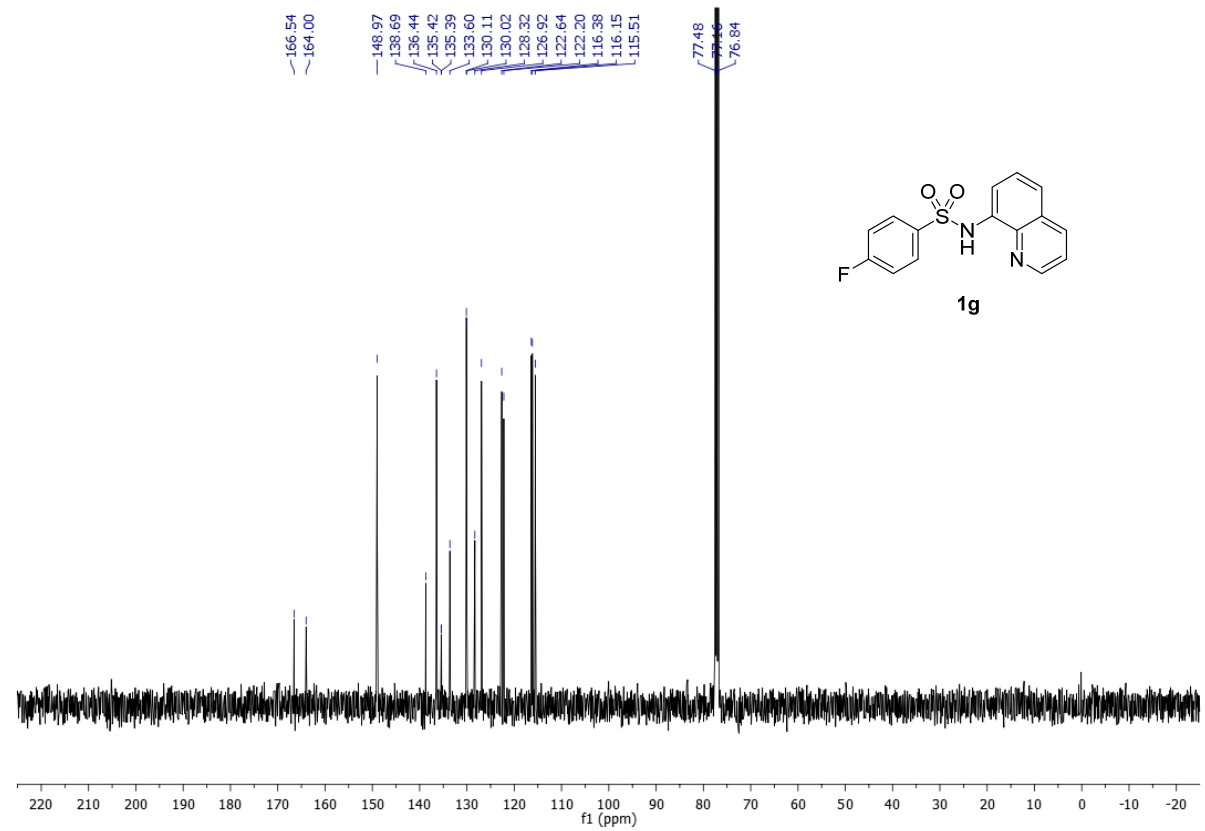
f1 (ppm)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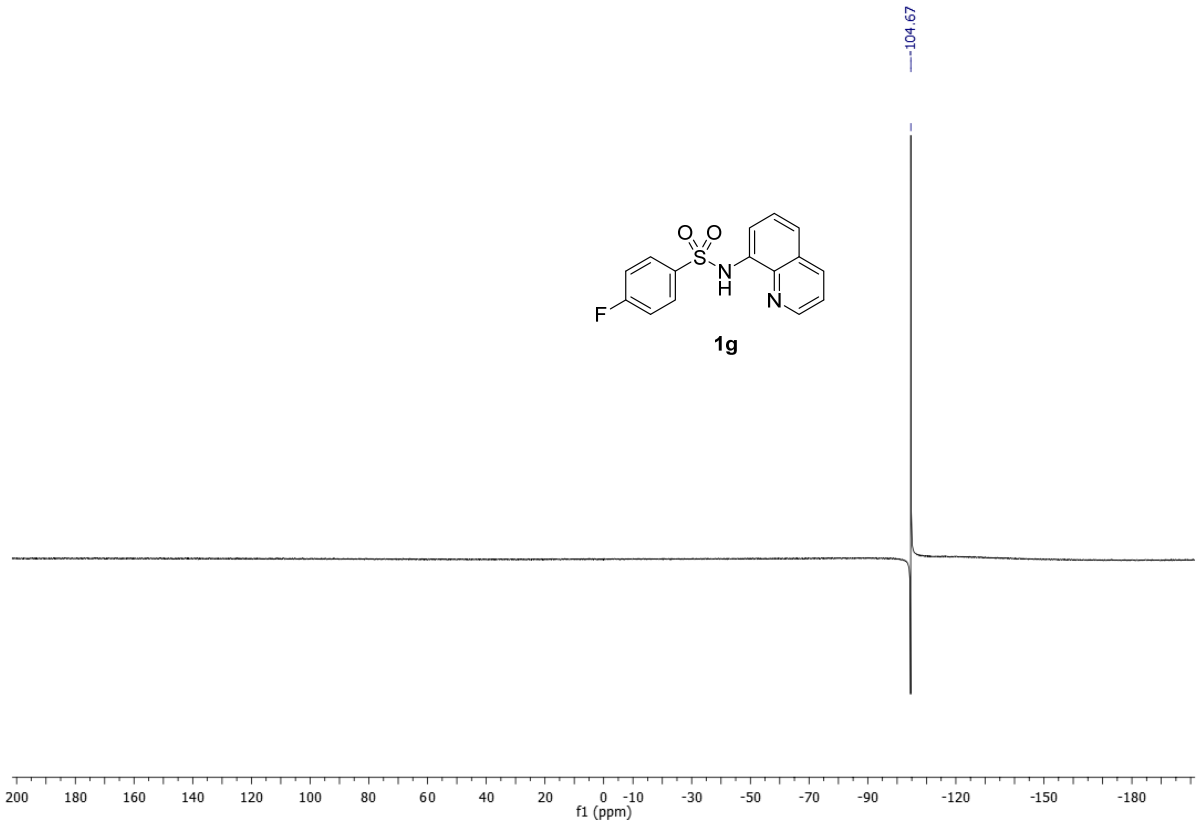
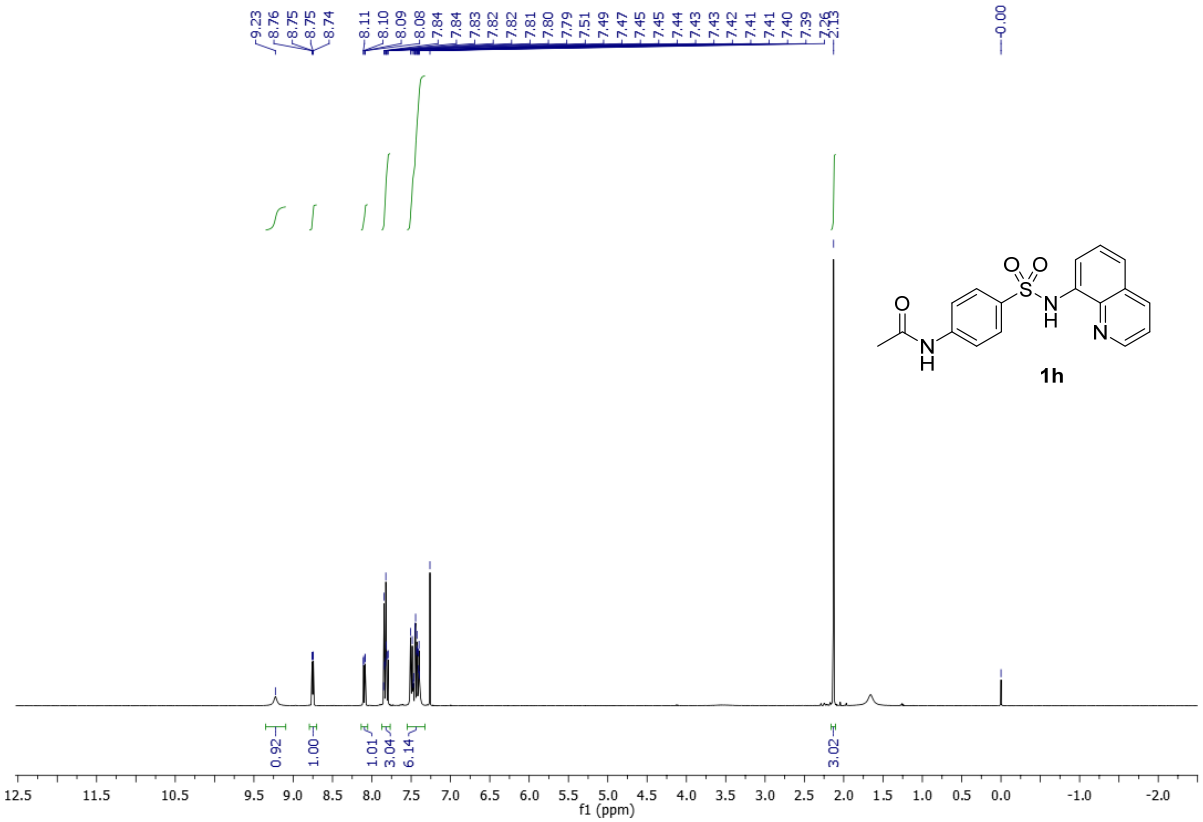
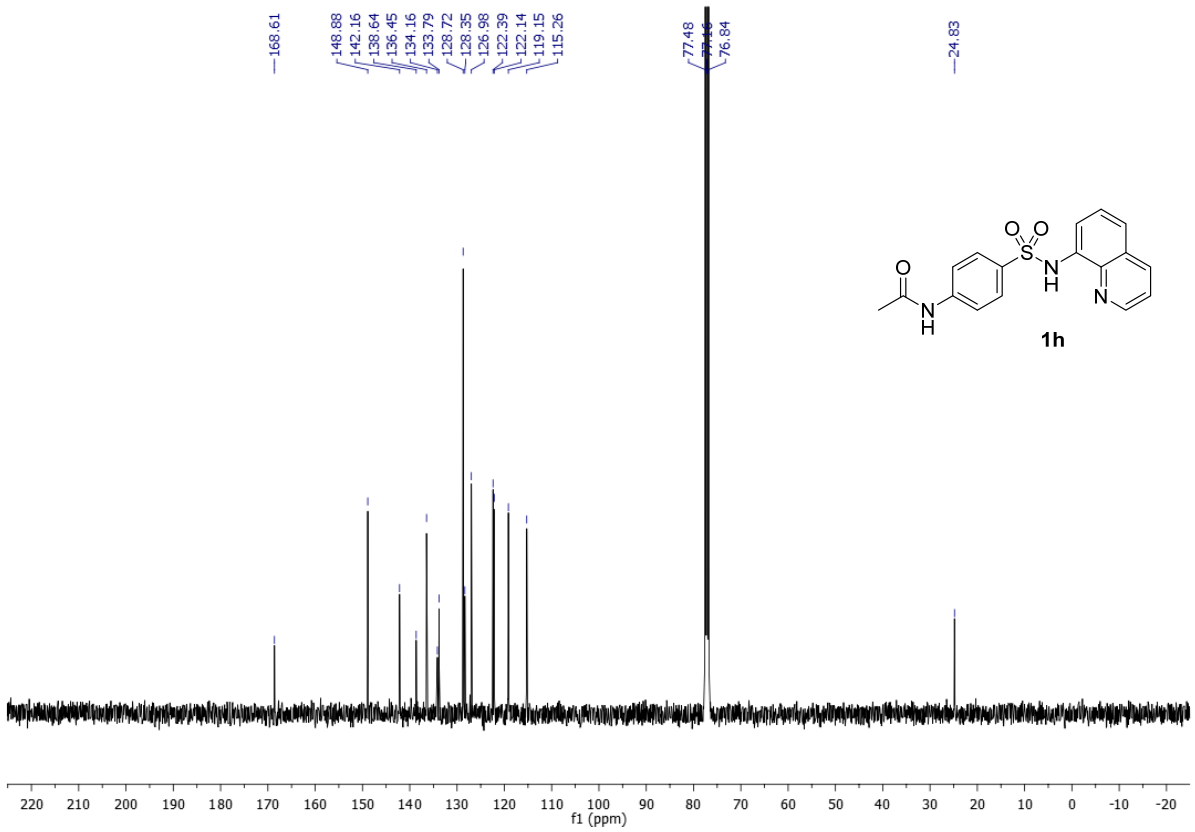
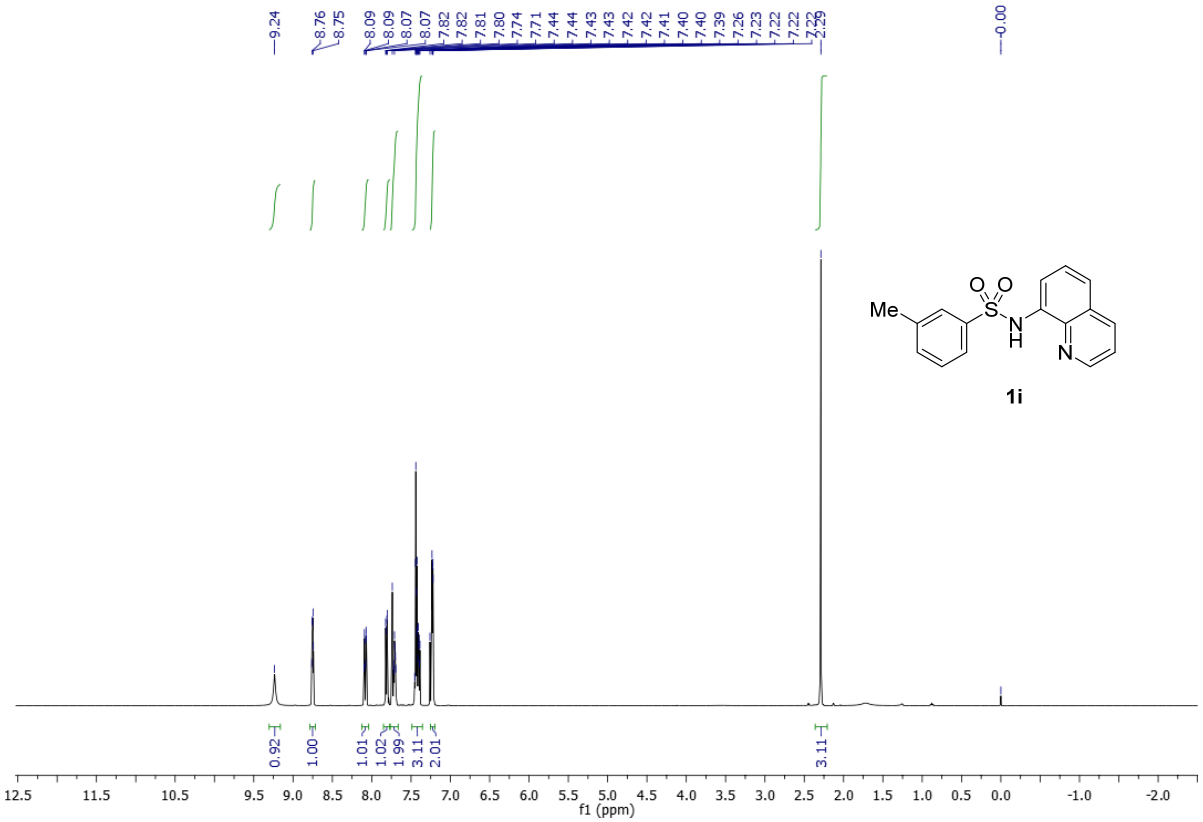
122.20
116.38
116.15
115.51
77.48
77.16
76.84
O
O
S
N
H
F
N
1g
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)


-104.67
1g
200 180 160 140 120 100 80 60 40 20 0 -10 -30 -50 -70 -90 -120 -150 -180
f1 (ppm)

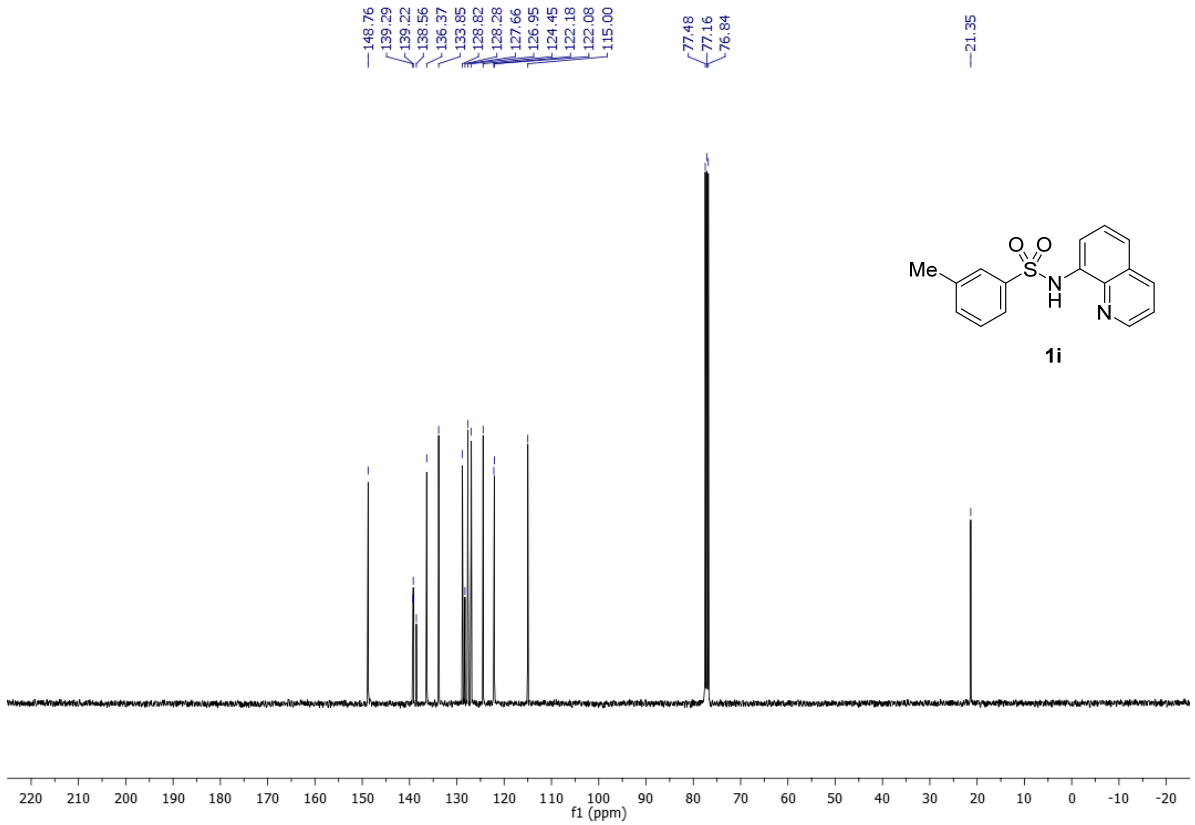

9.23
8.76
8.75
8.75
8.74
8.11
8.10
8.09
8.08
7.84
7.84
7.83
7.82
7.82
7.81
7.80
7.79
7.51
7.49
7.47
7.45
7.45
7.44
7.43
7.43
7.42
7.41
7.41
7.40
7.39
2.13
-0.00
1h
0.92
1.00
1.01
3.04
6.14
3.02
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -1.0 -2.0
f1 (ppm)

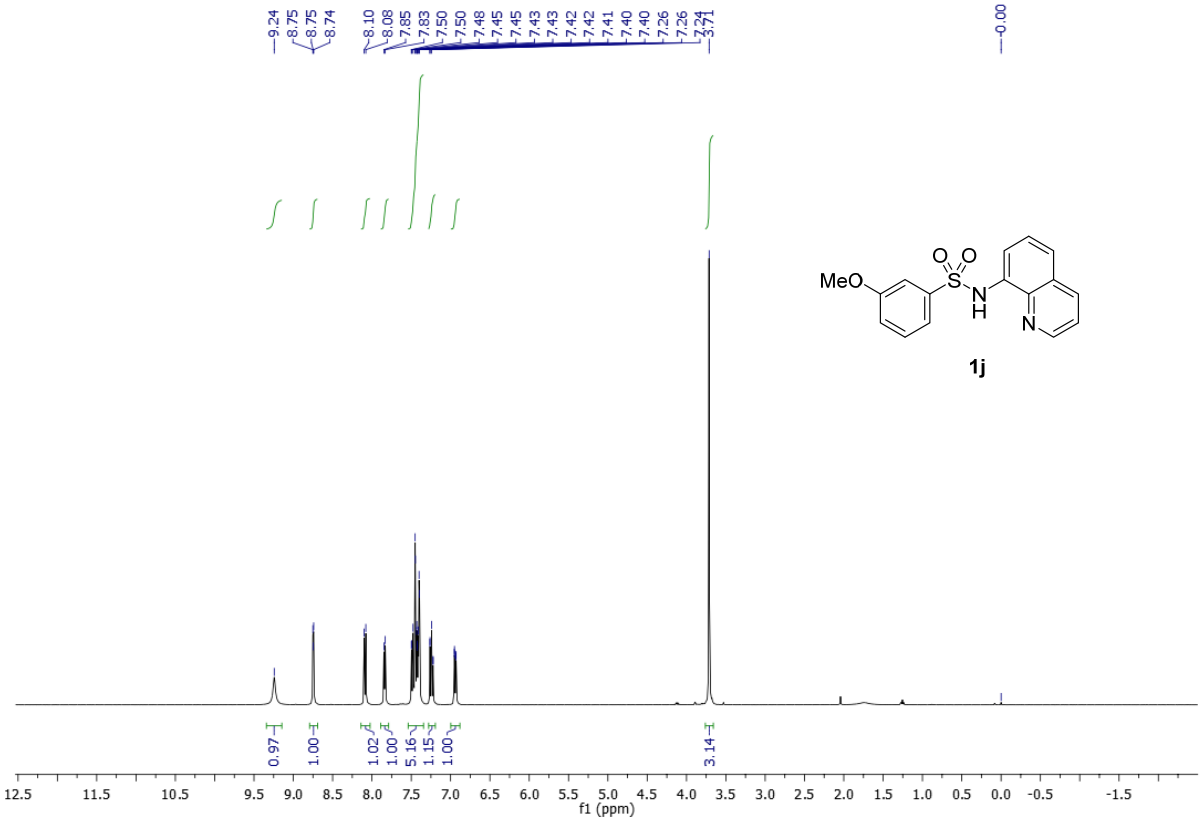

168.61
148.88
142.16
138.64
136.45
134.16
133.79
128.72
128.35
126.98
122.39
122.14
119.15
115.26
77.48
77.16
76.84
24.83
f1 (ppm)

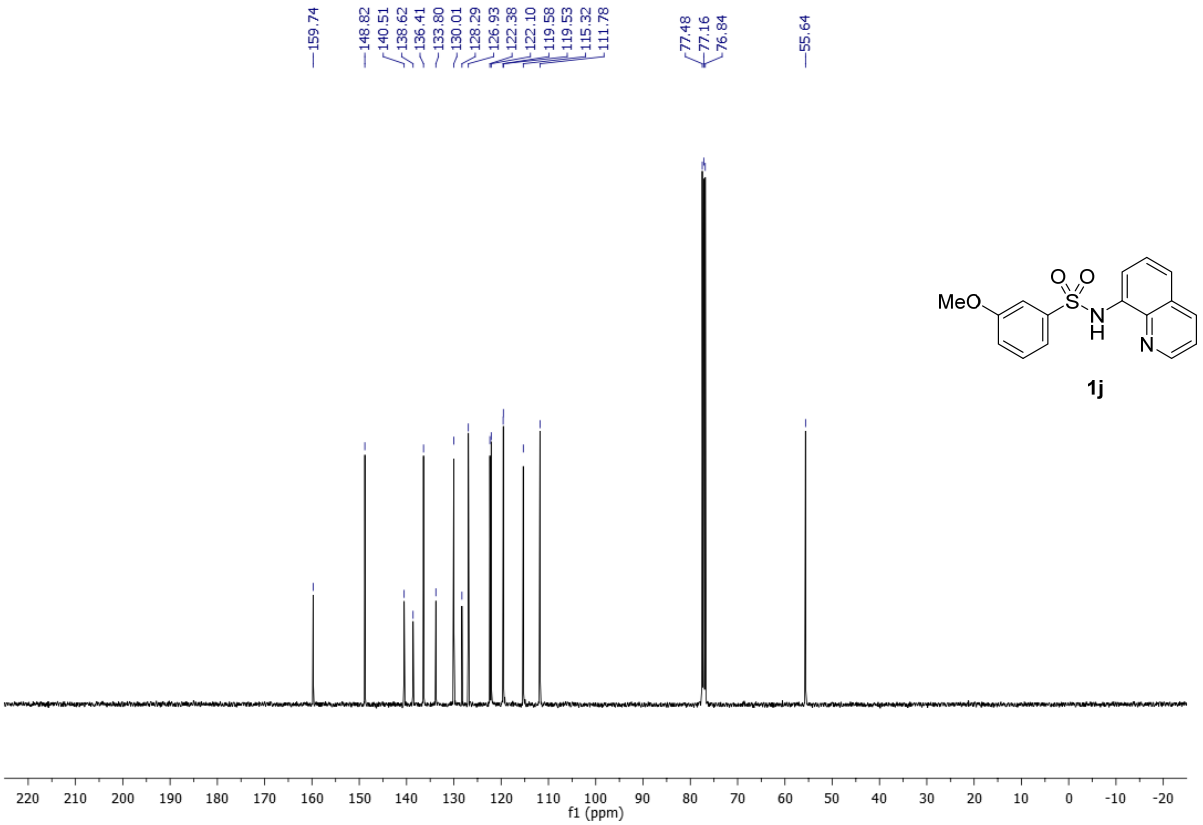
1h

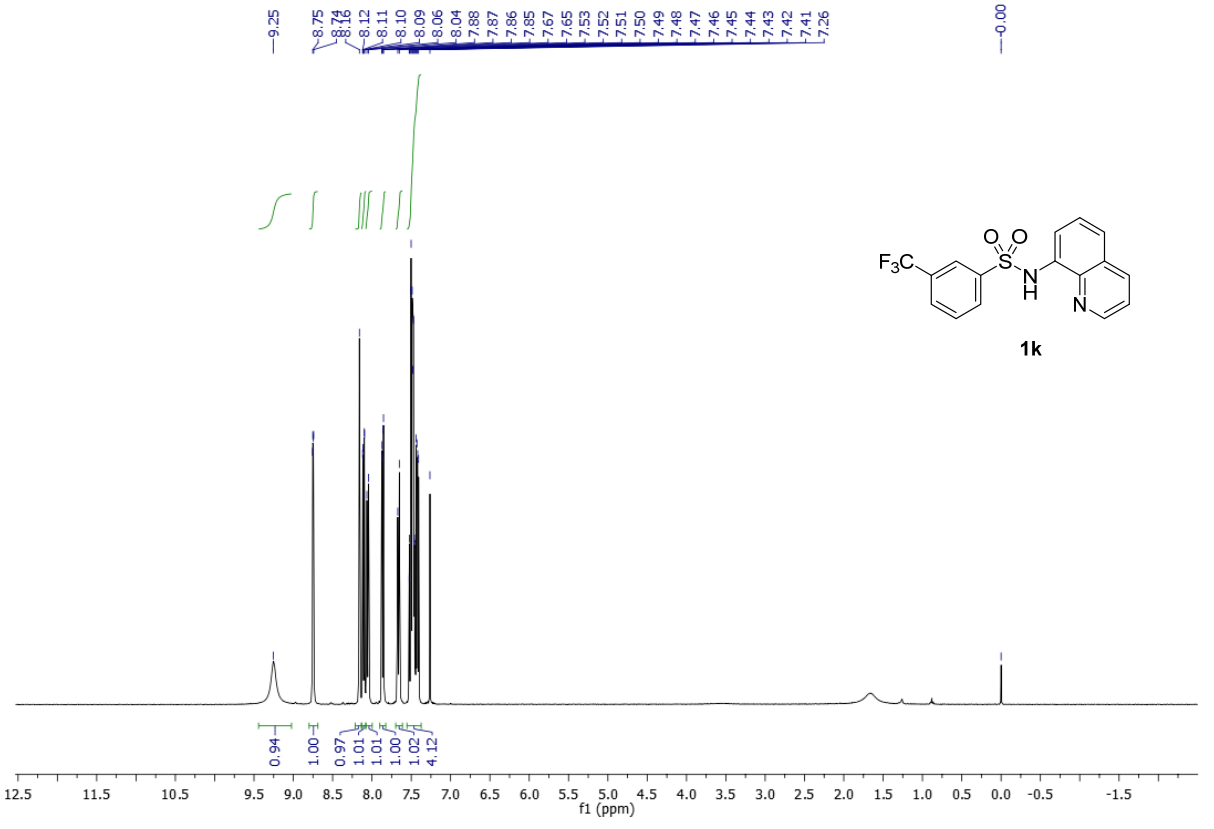

9.24
8.76
8.75
8.09
8.09
8.07
8.07
7.82
7.82
7.81
7.80
7.74
7.71
7.44
7.43
7.43
7.42
7.42
7.41
7.40
7.40
7.39
7.26
7.23
7.22
7.22
2.29
0.00
0.92
1.00
1.01
1.02
1.99
3.11
2.01
3.11
f1 (ppm)

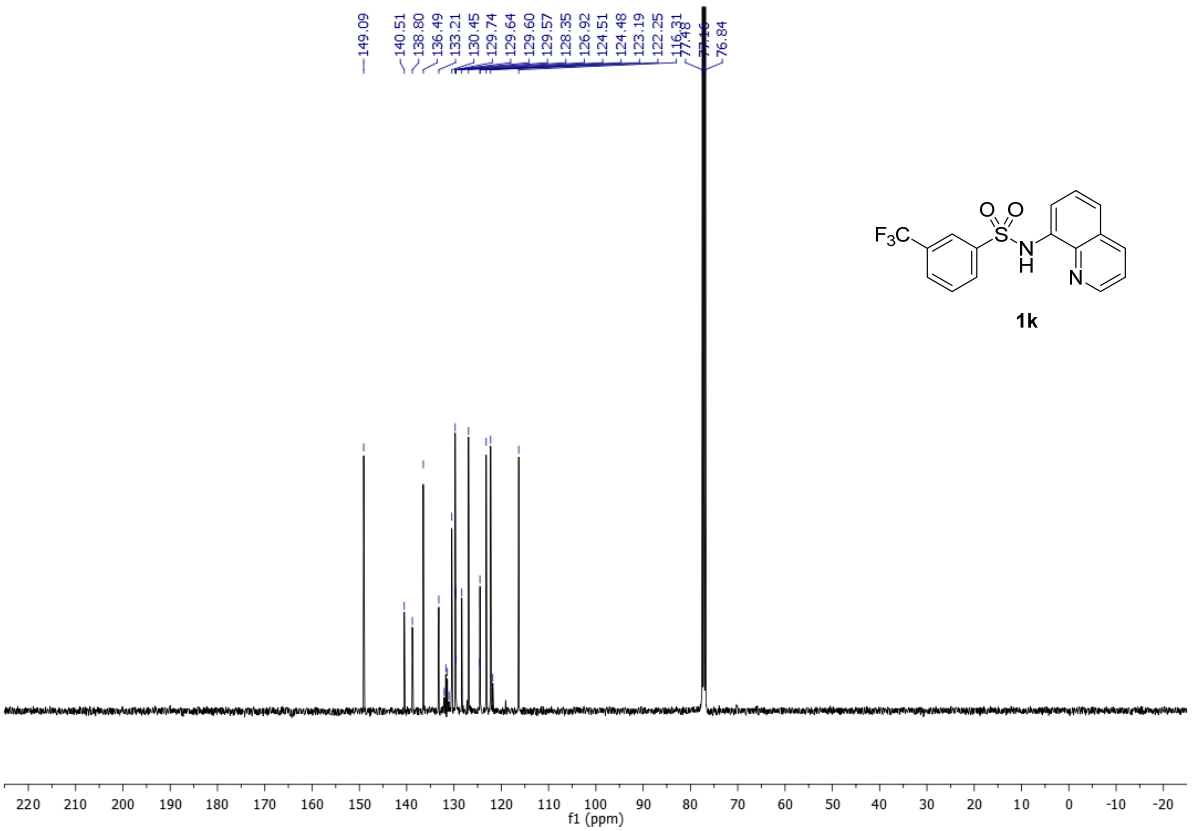
1i

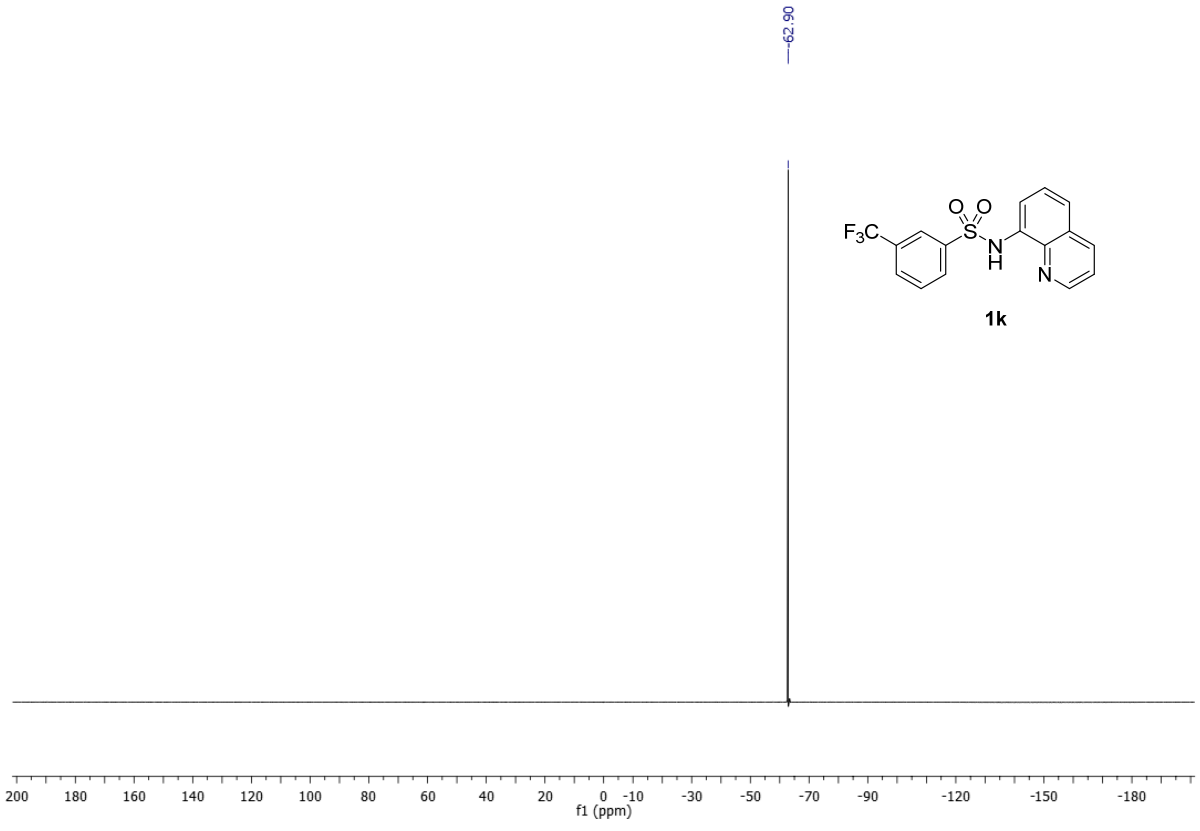

148.76
139.29
139.22
138.56
136.37
133.85
128.82
128.28
127.66
126.95
124.45
122.18
122.08
115.00
77.48
77.16
76.84
21.35
Me
O
O
S
N
H
N
1i
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

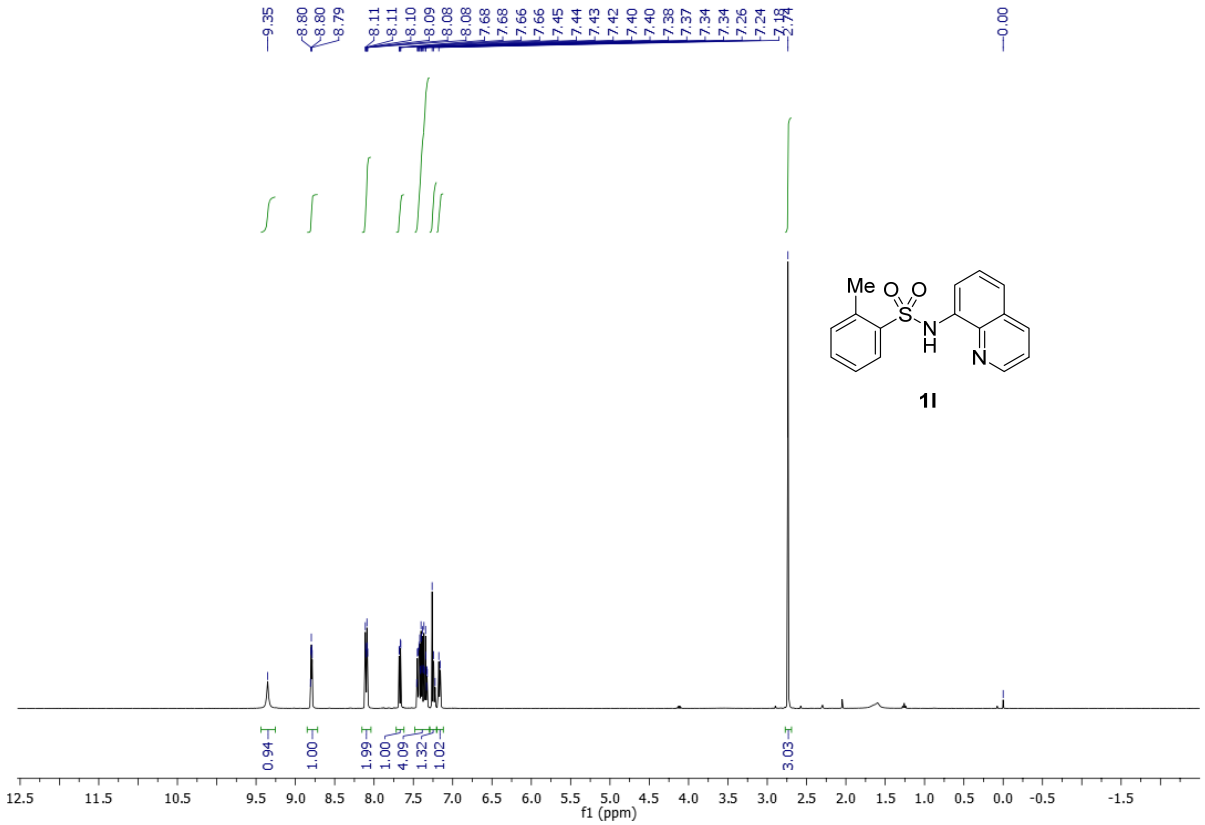

9.24
8.75
8.75
8.74
8.10
8.08
7.85
7.83
7.50
7.50
7.48
7.45
7.45
7.43
7.43
7.42
7.42
7.41
7.40
7.40
7.26
7.24
3.71
0.00
MeO
O
O
S
N
H
N
1j
0.97
1.00
1.02
1.00
5.16
1.15
1.00
3.14
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.5
f1 (ppm)

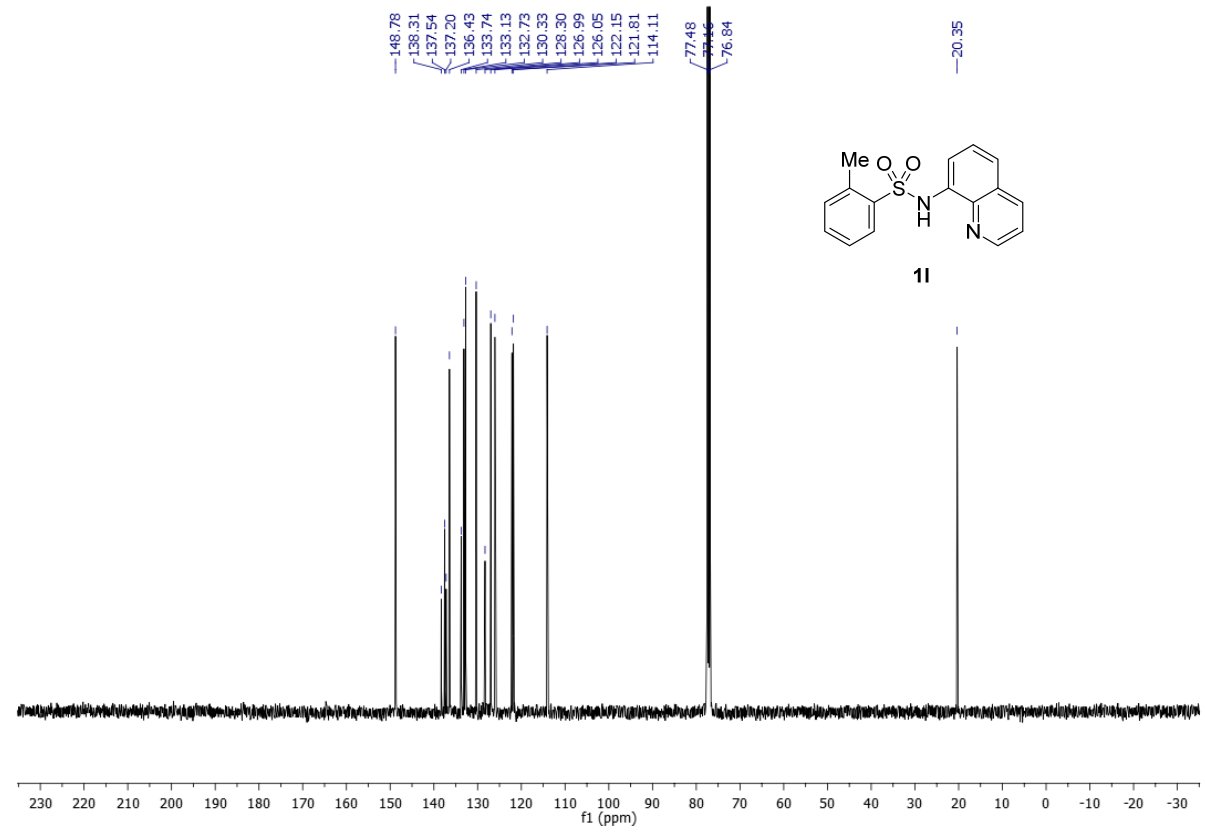

159.74
148.82
140.51
138.62
136.41
133.80
130.01
128.29
126.93
122.38
122.10
119.58
119.53
115.32
111.78
77.48
77.16
76.84
55.64
MeO
O
O
S
N
H
N
1j
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

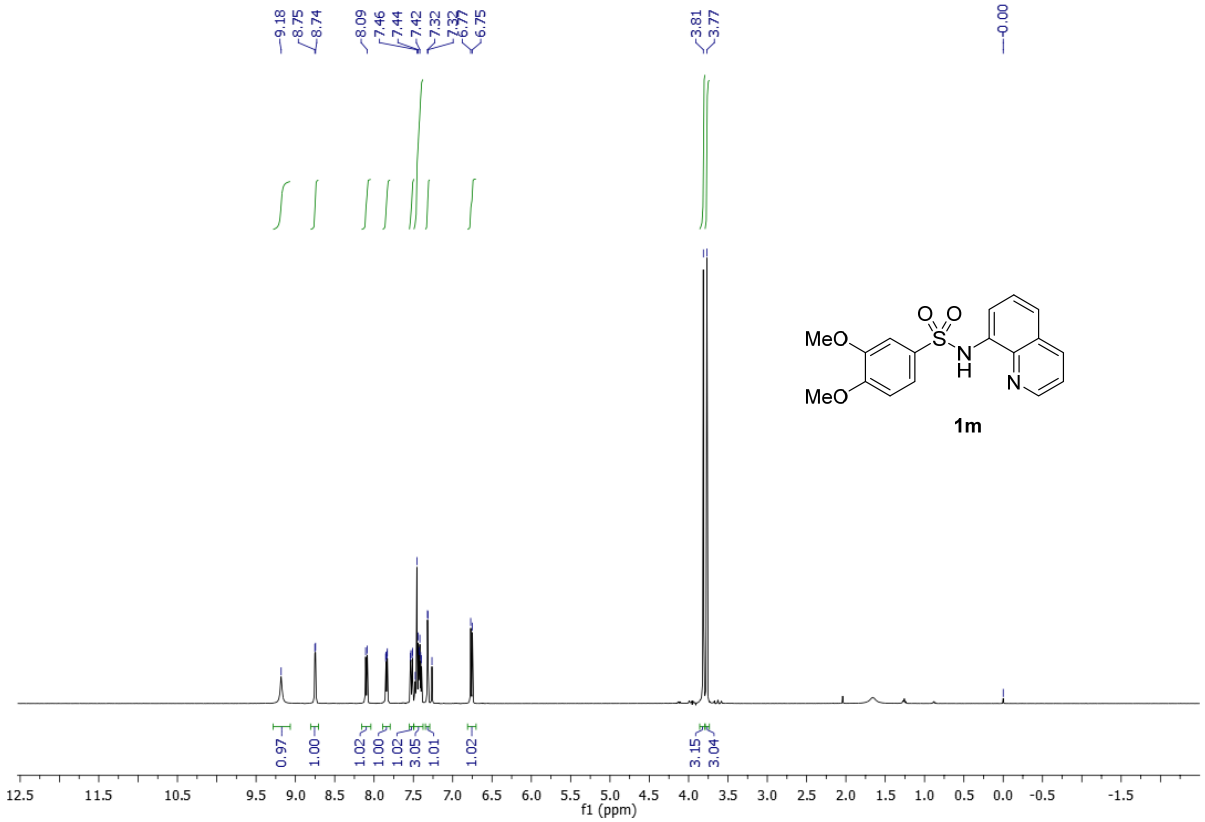

9.25
8.75
8.74
8.18
8.12
8.11
8.10
8.09
8.06
8.04
7.88
7.87
7.86
7.85
7.67
7.65
7.53
7.52
7.51
7.50
7.49
7.48
7.47
7.46
7.45
7.44
7.43
7.42
7.41
7.26
0.00
F3C
O
O
S
N
H
N
1k
0.94
1.00
0.97
1.01
1.01
1.00
1.02
4.12
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.5
f1 (ppm)

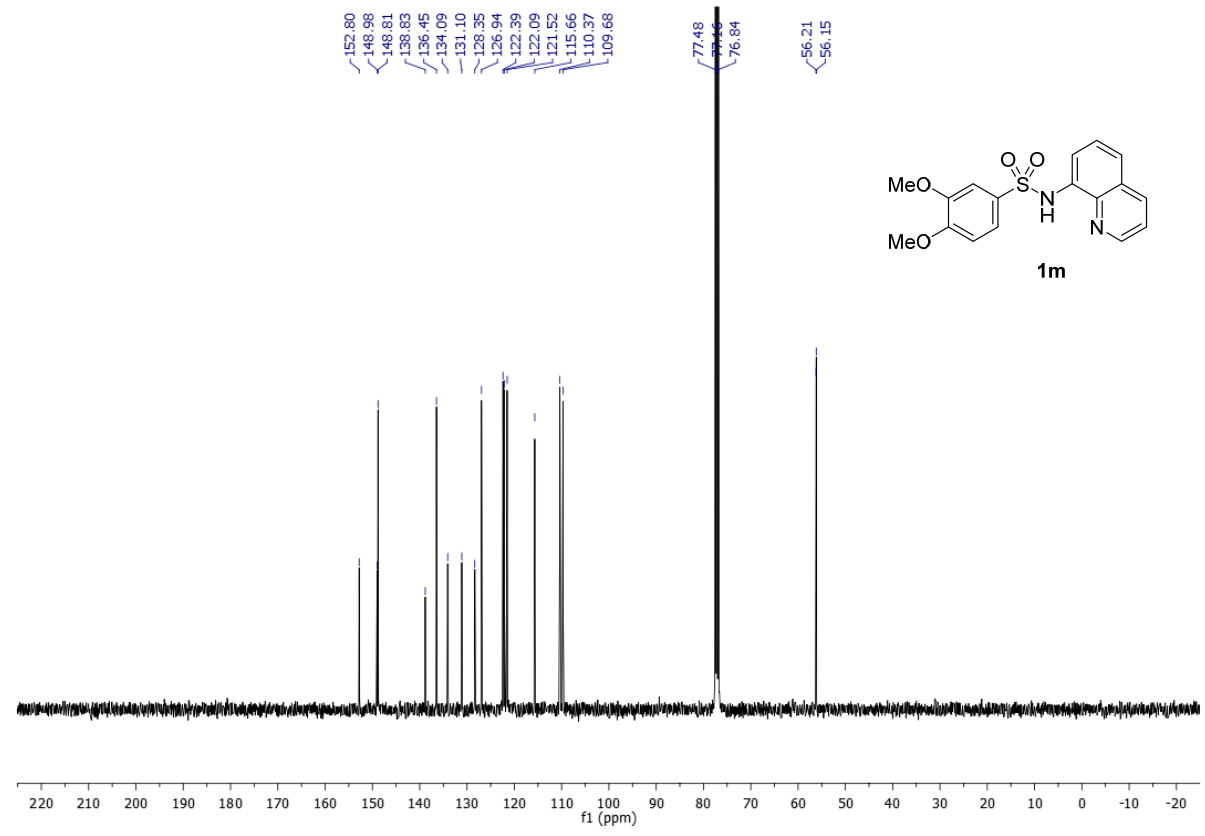

149.09
140.51
138.80
136.49
133.21
130.45
129.74
129.64
129.60
129.57
128.35
126.92
124.51
124.48
123.19
122.25
116.31
77.48
76.84
F3C
O
O
S
N
H
N
1k
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

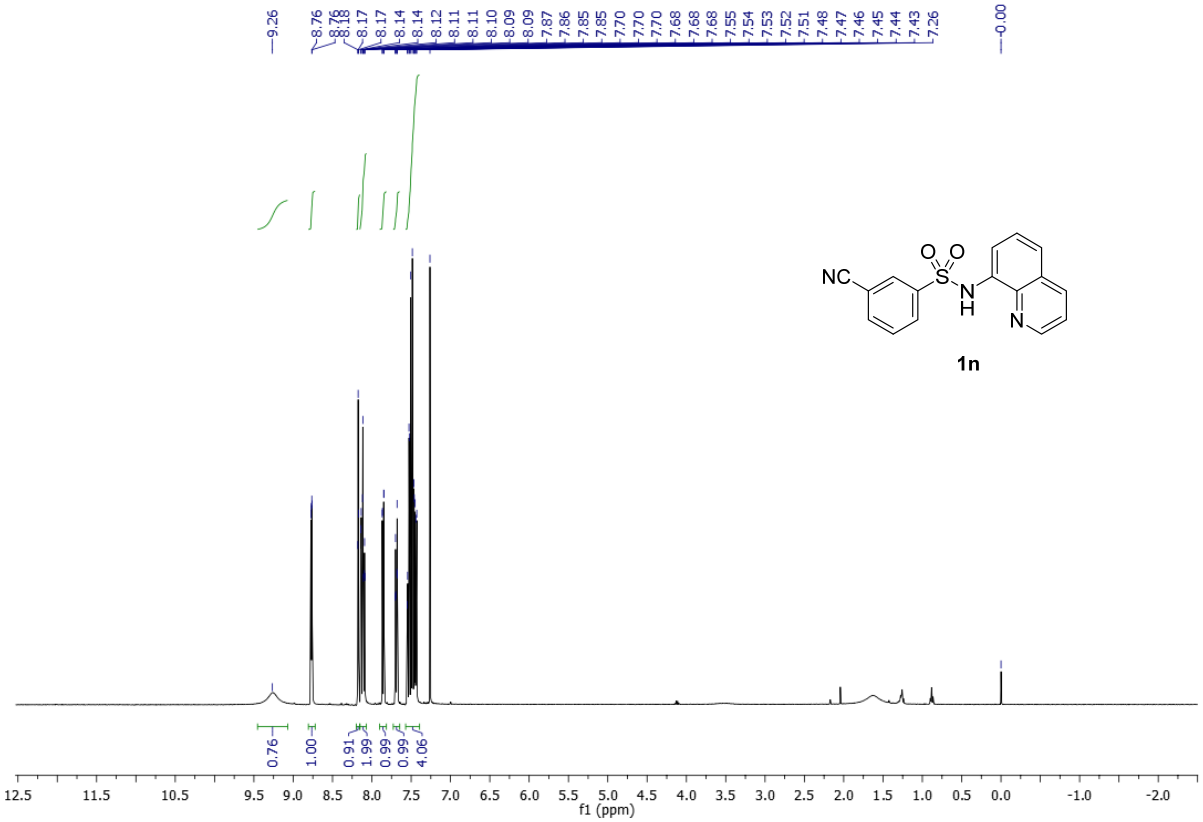

-62.90
F3C
O
O
S
N
H
N
1k
200 180 160 140 120 100 80 60 40 20 0 -10 -30 -50 -70 -90 -120 -150 -180
f1 (ppm)

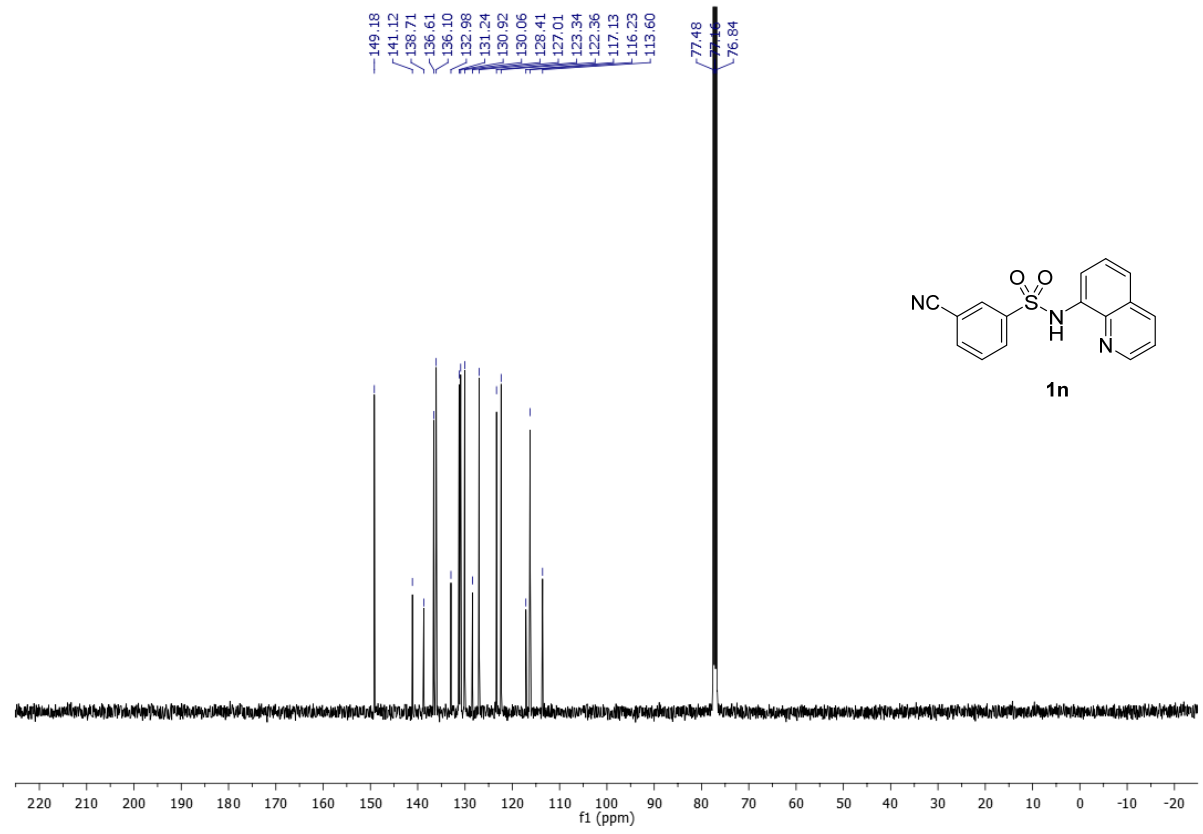

9.35
8.80
8.80
8.79
8.11
8.11
8.10
8.09
8.08
8.08
7.68
7.68
7.66
7.66
7.45
7.44
7.43
7.42
7.40
7.40
7.38
7.37
7.34
7.34
7.26
7.24
2.74
0.00
Me
O
O
S
N
H
N
1l
0.94
1.00
1.99
1.00
4.09
1.32
1.02
3.03
f1 (ppm)


148.78
138.31
137.54
137.20
136.43
133.74
133.13
132.73
130.33
128.30
126.99
126.05
122.15
121.81
114.11
77.48
76.84
20.35
Me
O
O
S
N
H
N
1l
f1 (ppm)

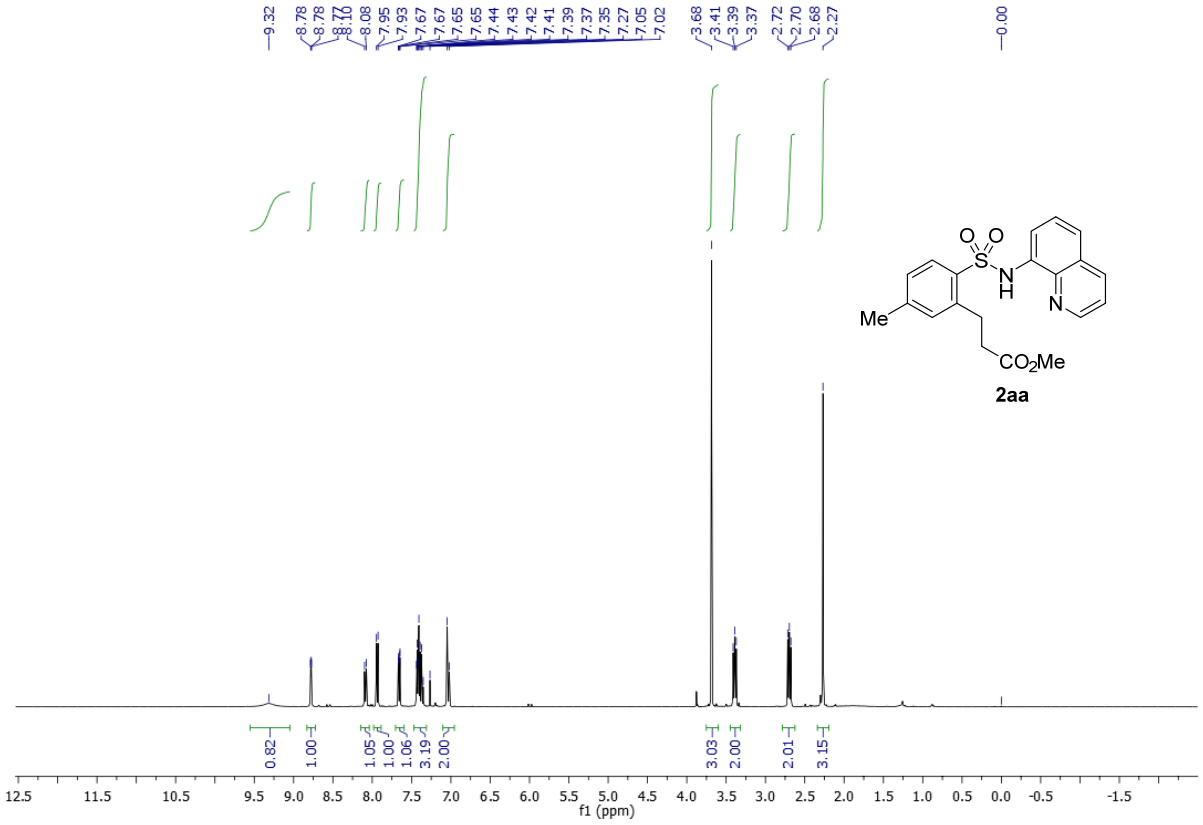

9.18
8.75
8.74
8.09
7.46
7.44
7.42
7.32
6.77
6.75
3.81
3.77
0.00
MeO
MeO
O
O
S
N
H
N
1m
0.97
1.00
1.02
1.00
1.02
3.05
1.01
1.02
3.15
3.04
f1 (ppm)

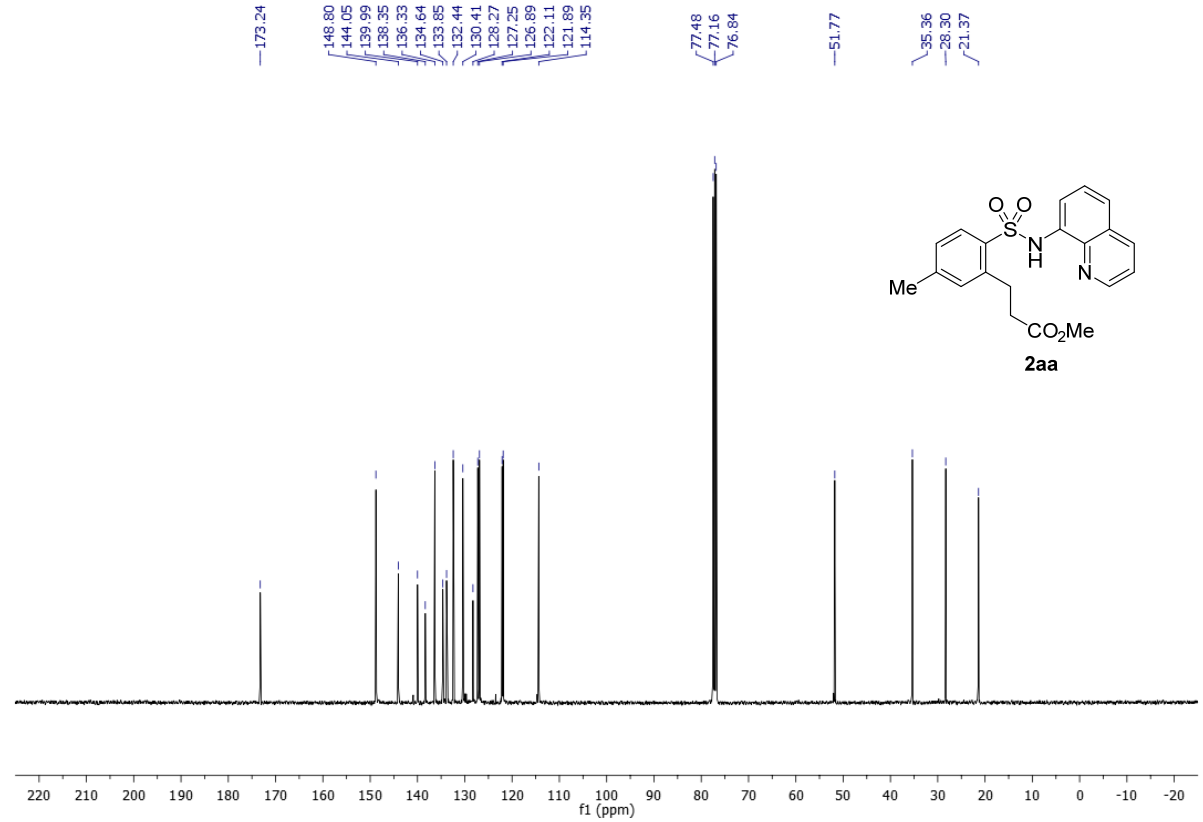

152.80
148.98
148.81
138.83
136.45
134.09
131.10
128.35
126.94
122.39
122.09
121.52
115.66
110.37
109.68
77.48
77.16
76.84
56.21
56.15
MeO
MeO
O
O
S
N
H
N
1m
f1 (ppm)

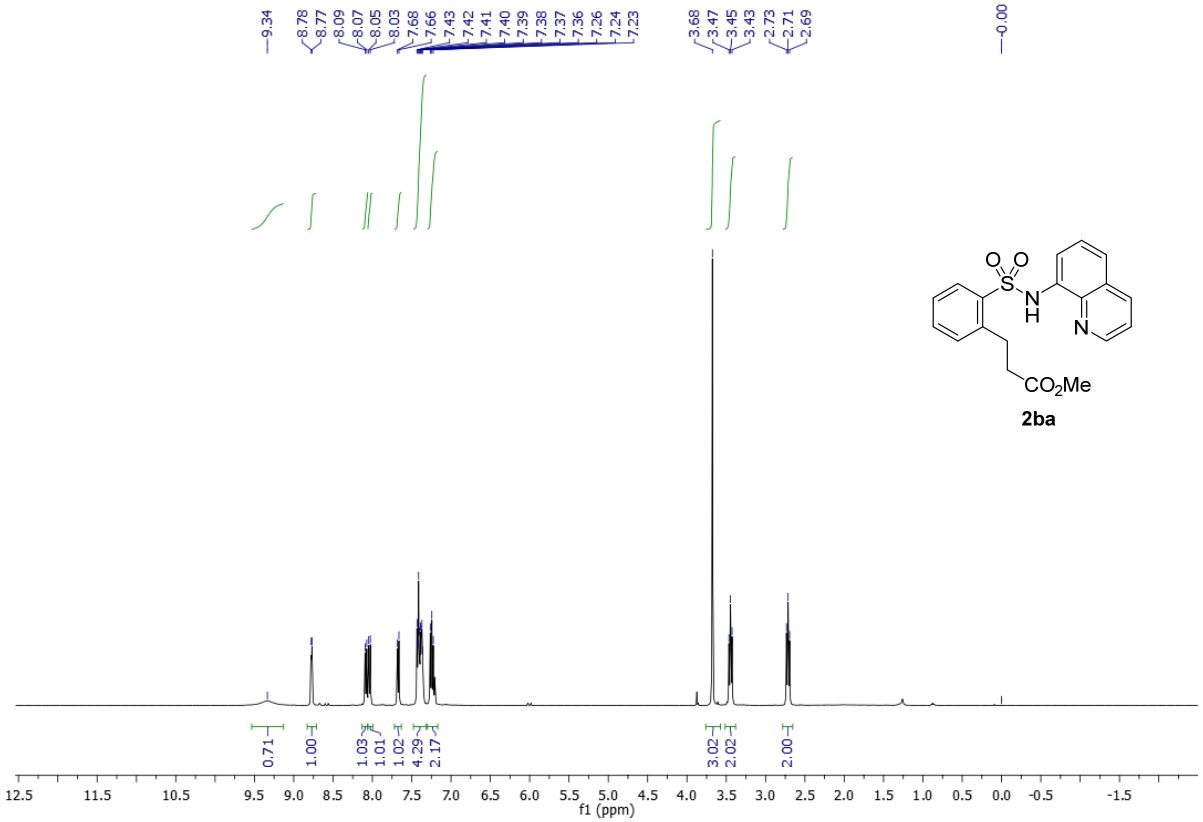

9.26
8.76
8.18
8.17
8.14
8.12
8.11
8.10
8.09
7.87
7.86
7.85
7.70
7.68
7.55
7.54
7.53
7.52
7.51
7.48
7.47
7.46
7.45
7.44
7.43
7.26
0.00
0.76
1.00
0.91
1.99
0.99
0.99
4.06
f1 (ppm)
NC
O
O
S
N
H
N
1n


149.18
141.12
138.71
136.61
136.10
132.98
131.24
130.92
130.06
128.41
127.01
123.34
122.36
117.13
116.23
113.60
77.48
77.16
76.84
f1 (ppm)
NC
O
O
S
N
H
N
1n

17. NMR spectra of products






9.34
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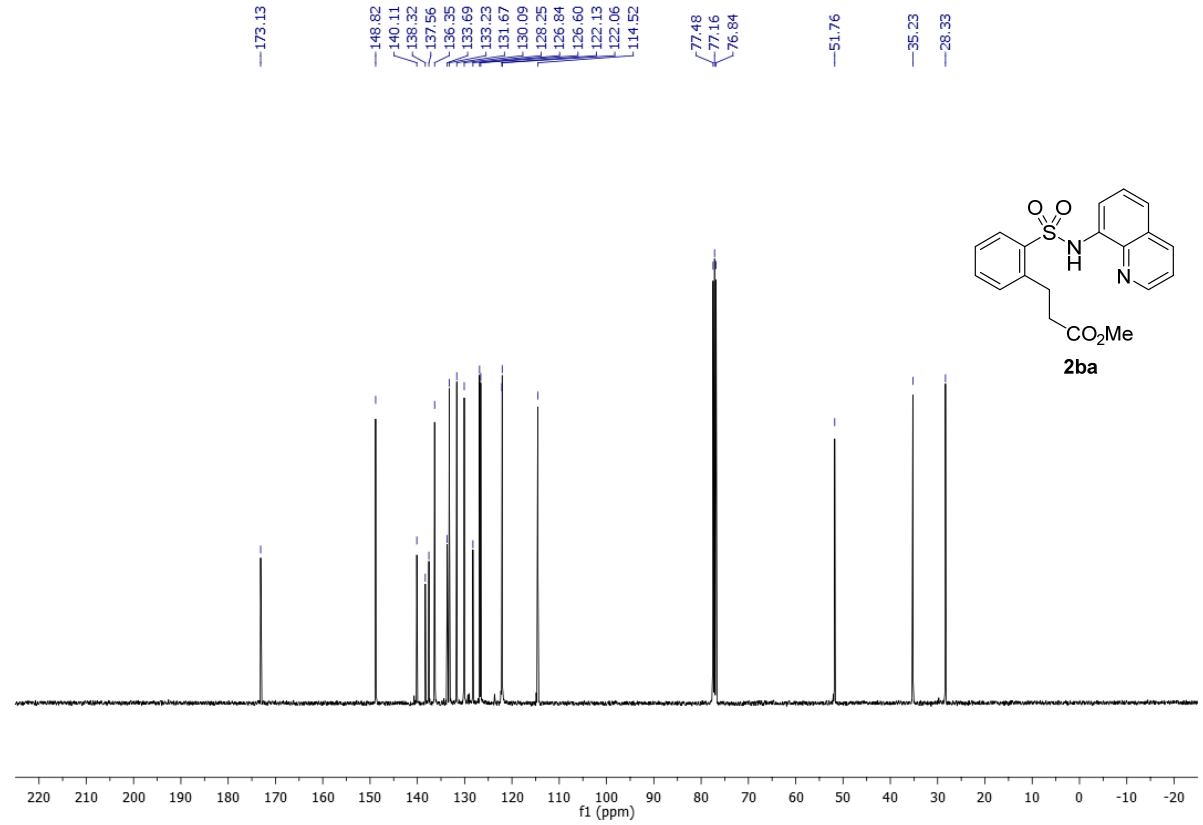
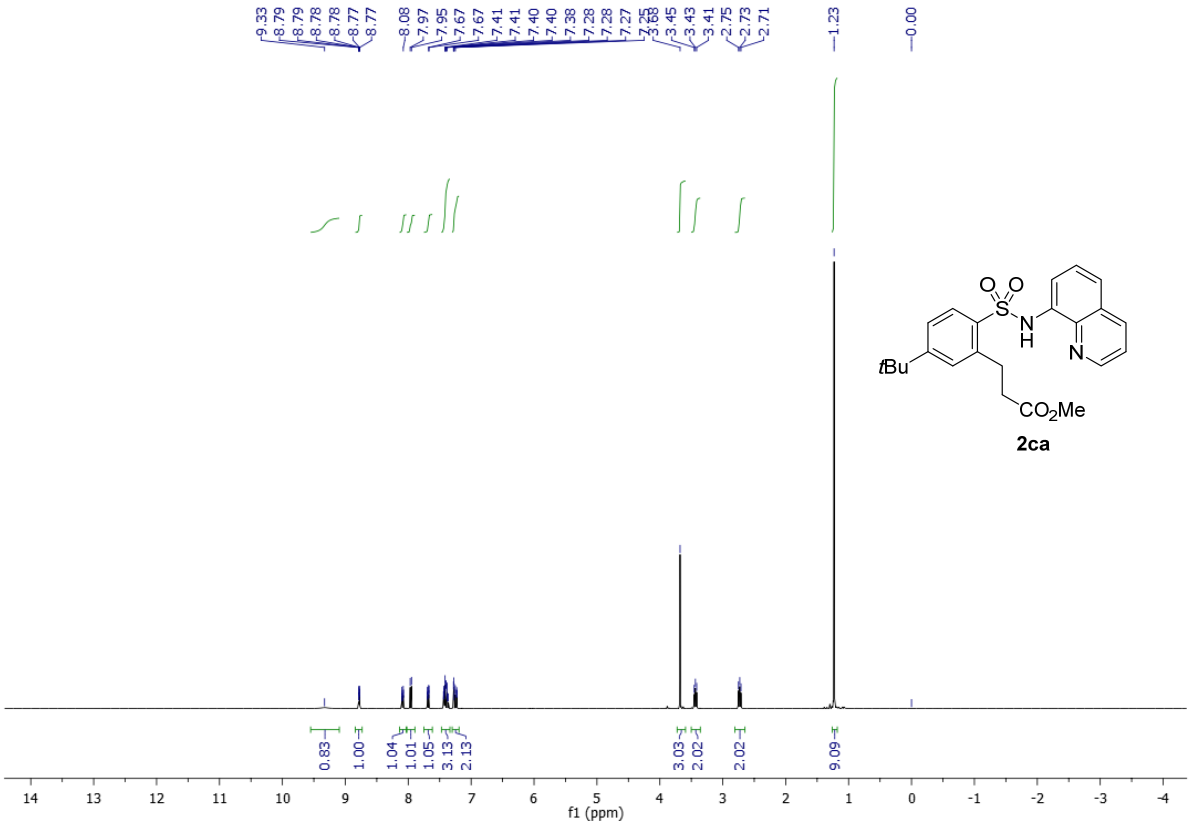
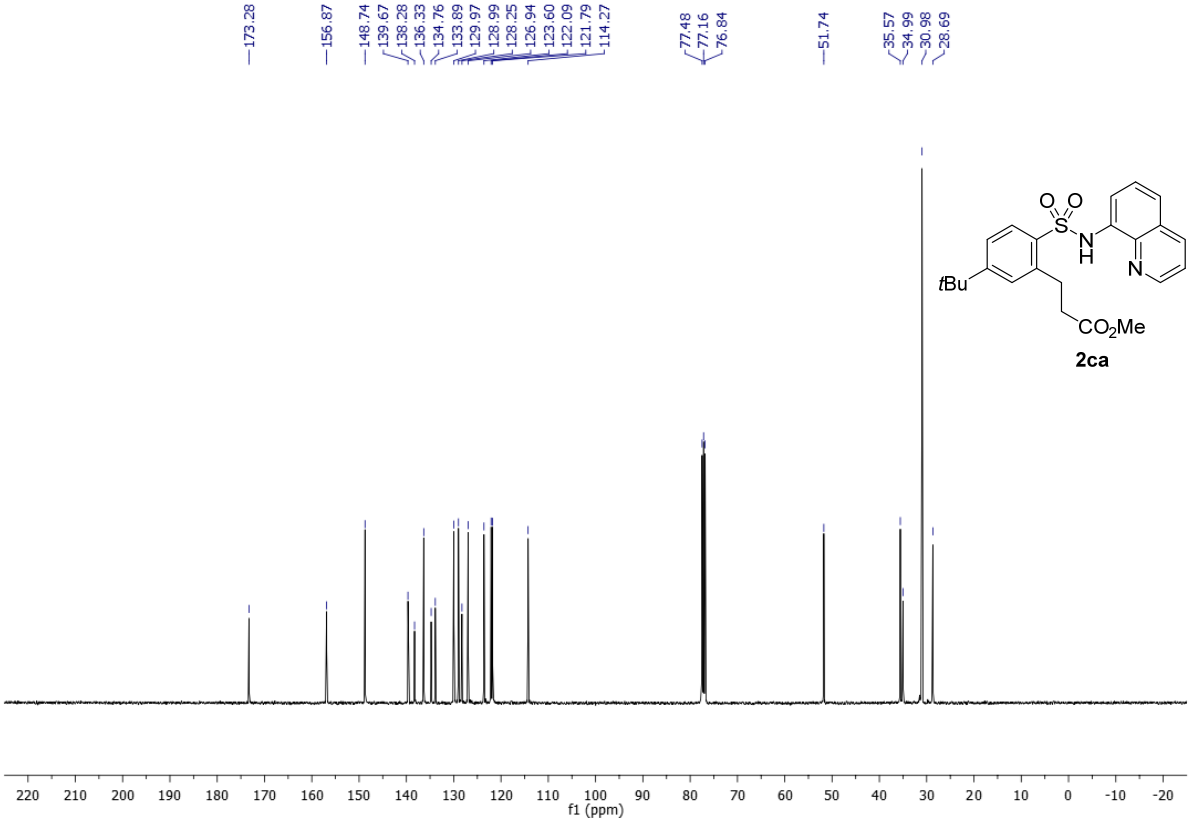
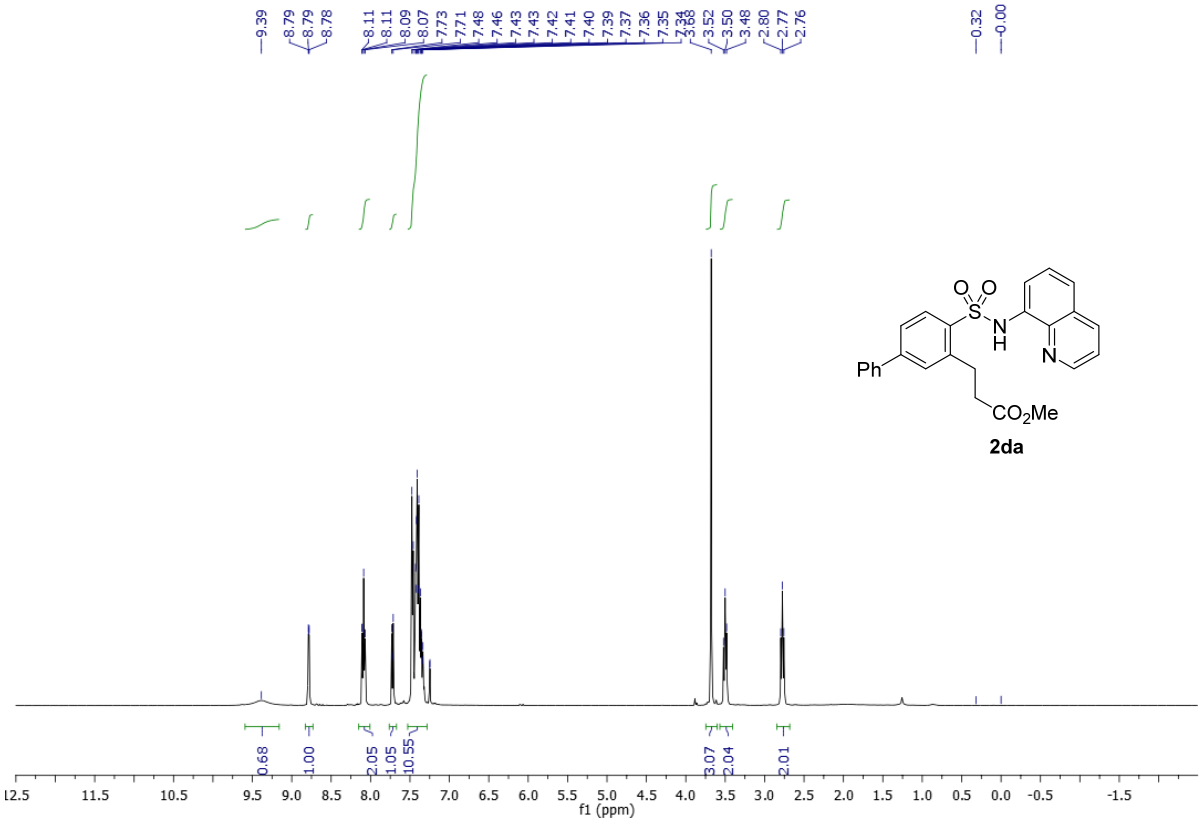
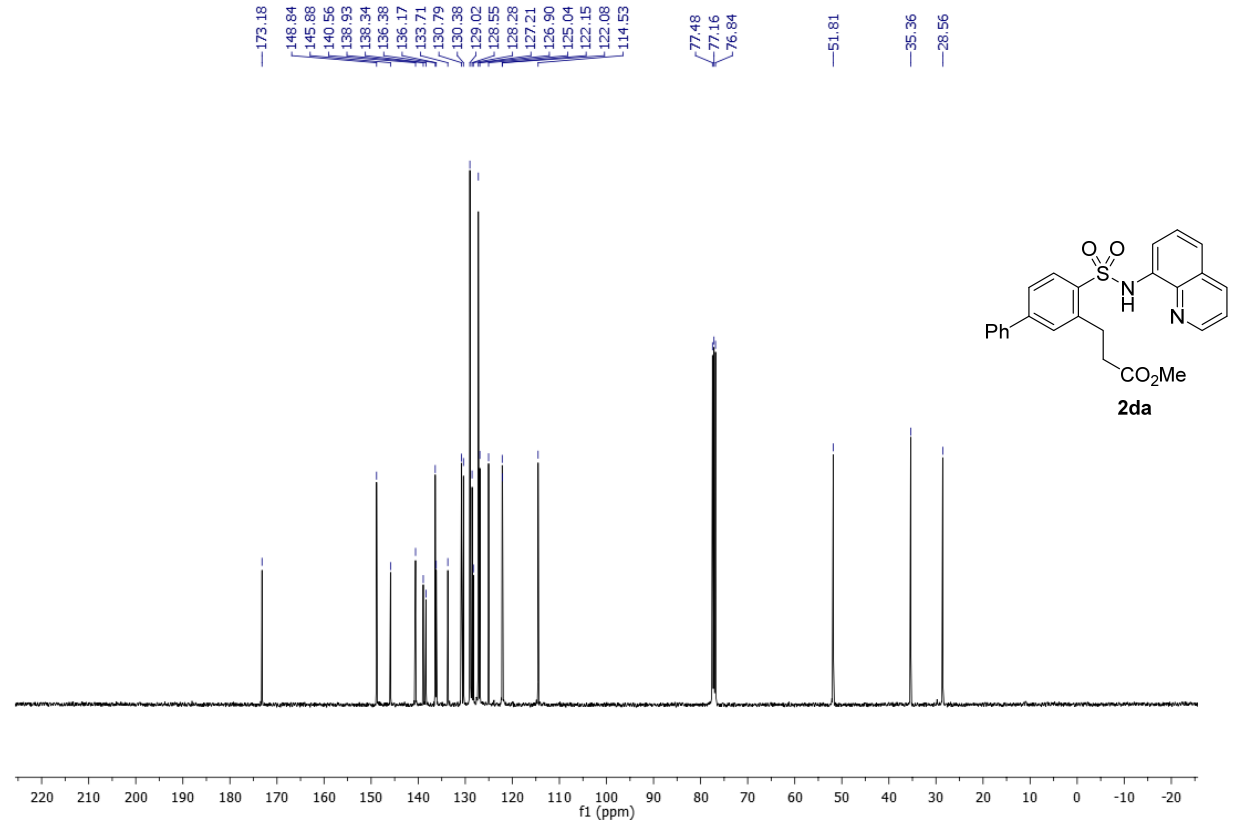
8.77
8.09
8.07
8.05
8.03
7.68
7.66
7.43
7.42
7.41
7.40
7.39
7.38
7.37
7.36
7.26
7.24
7.23
3.68
3.47
3.45
3.43
2.73
2.71
2.69
0.00
0.71
1.00
1.03
1.01
1.02
4.29
2.17
3.02
2.02
2.00
f1 (ppm)
2ba

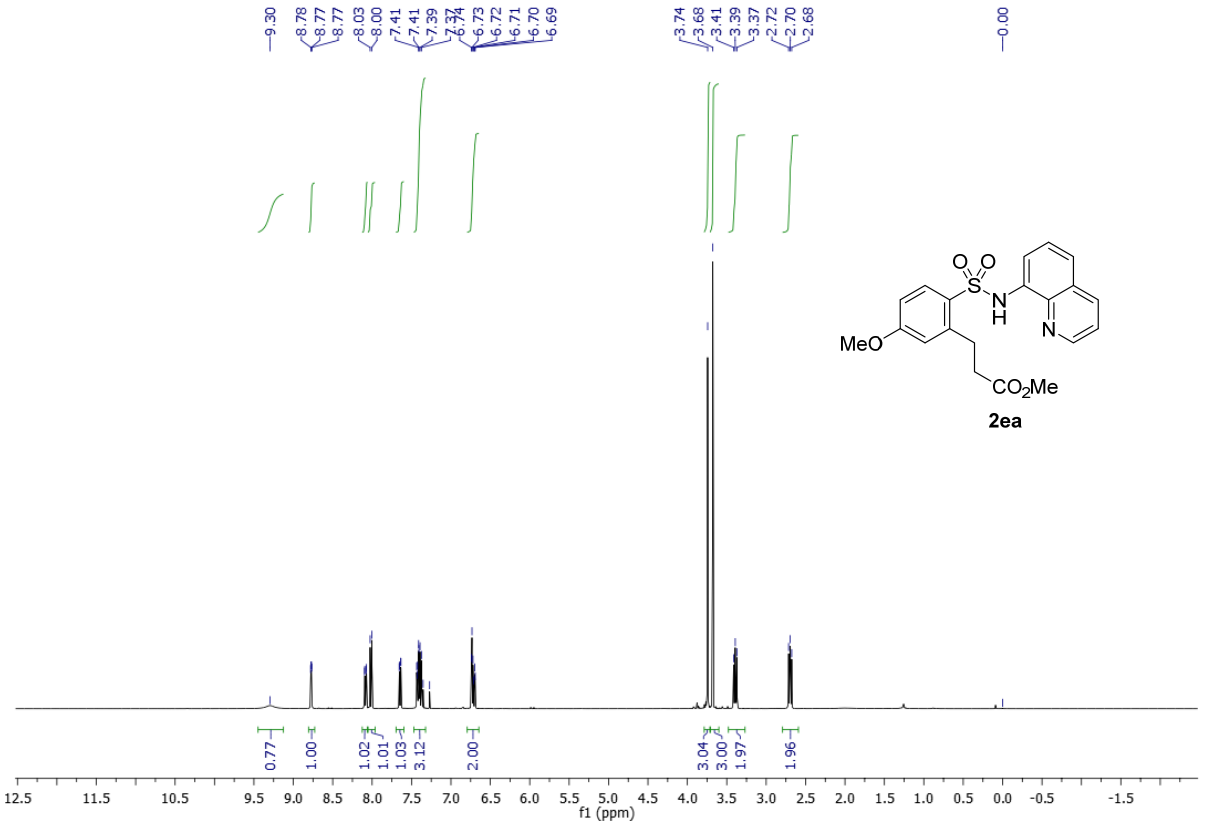

173.13
148.82
140.11
138.32
137.56
136.35
133.69
133.23
131.67
130.09
128.25
126.84
126.60
122.13
122.06
114.52
77.48
77.16
76.84
51.76
35.23
28.33
f1 (ppm)
2ba

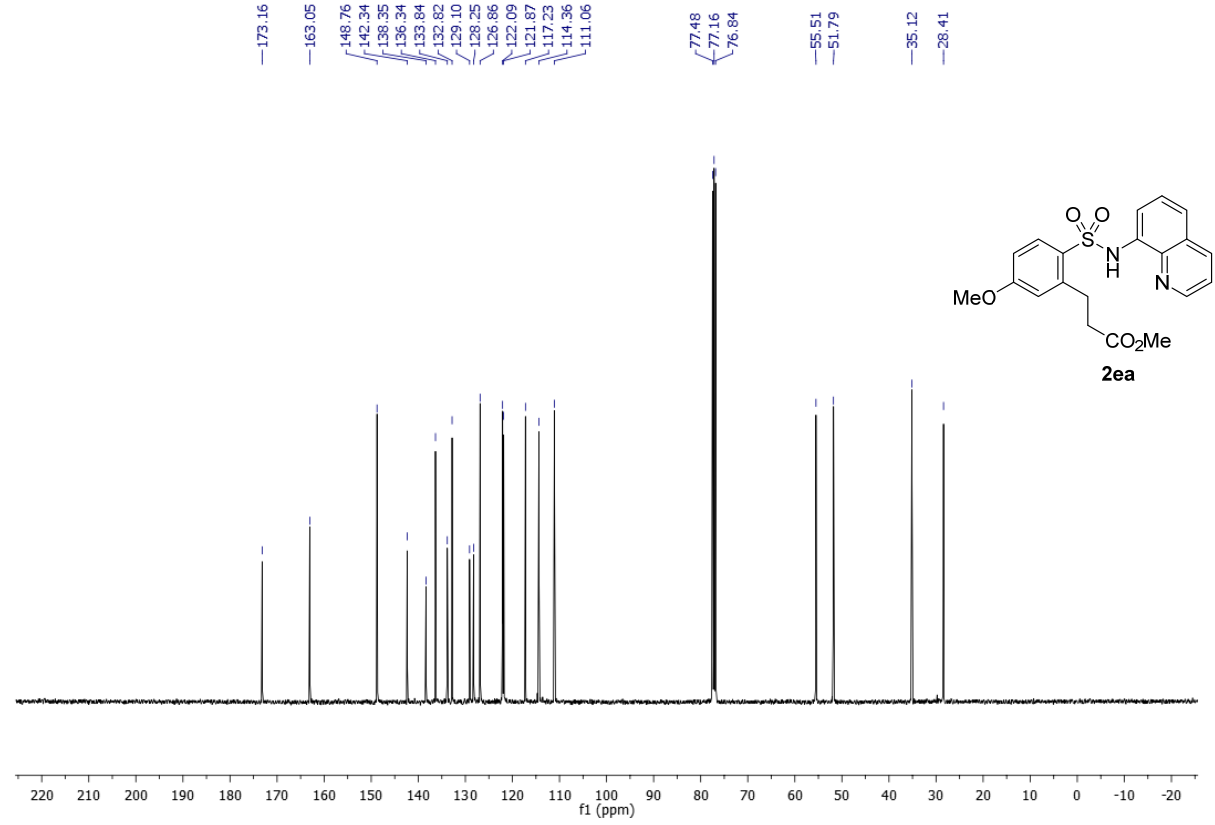

9.33
8.79
8.79
8.78
8.78
8.77
8.77
8.08
7.97
7.95
7.67
7.67
7.41
7.41
7.40
7.40
7.38
7.28
7.27
3.45
3.43
3.41
2.75
2.73
2.71
1.23
0.00
0.83
1.00
1.04
1.01
1.05
3.13
2.13
3.03
2.02
2.02
9.09
f1 (ppm)
tBu
CO2Me
2ca

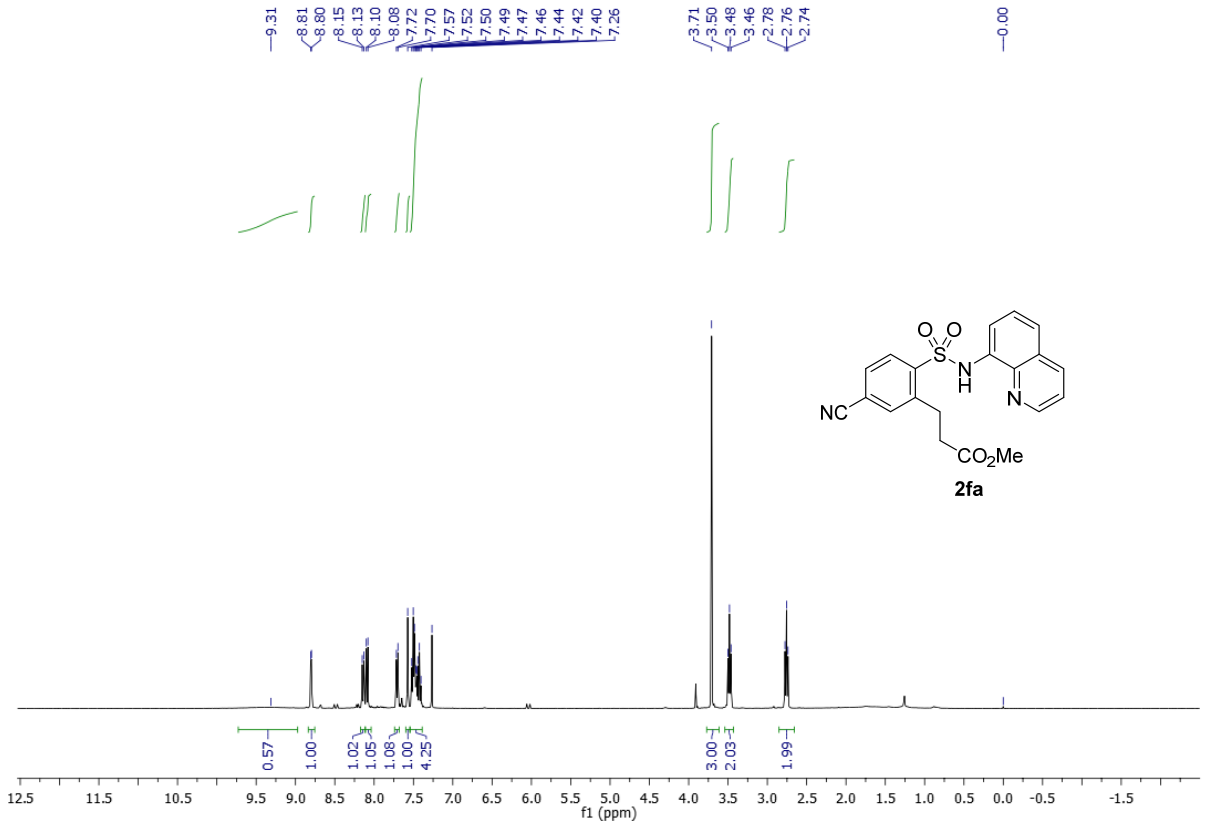

173.28
156.87
148.74
139.67
138.28
136.33
134.76
133.89
129.97
128.99
128.25
126.94
123.60
122.09
121.79
114.27
77.48
77.16
76.84
51.74
35.57
34.99
30.98
28.69
f1 (ppm)
tBu
CO2Me
2ca

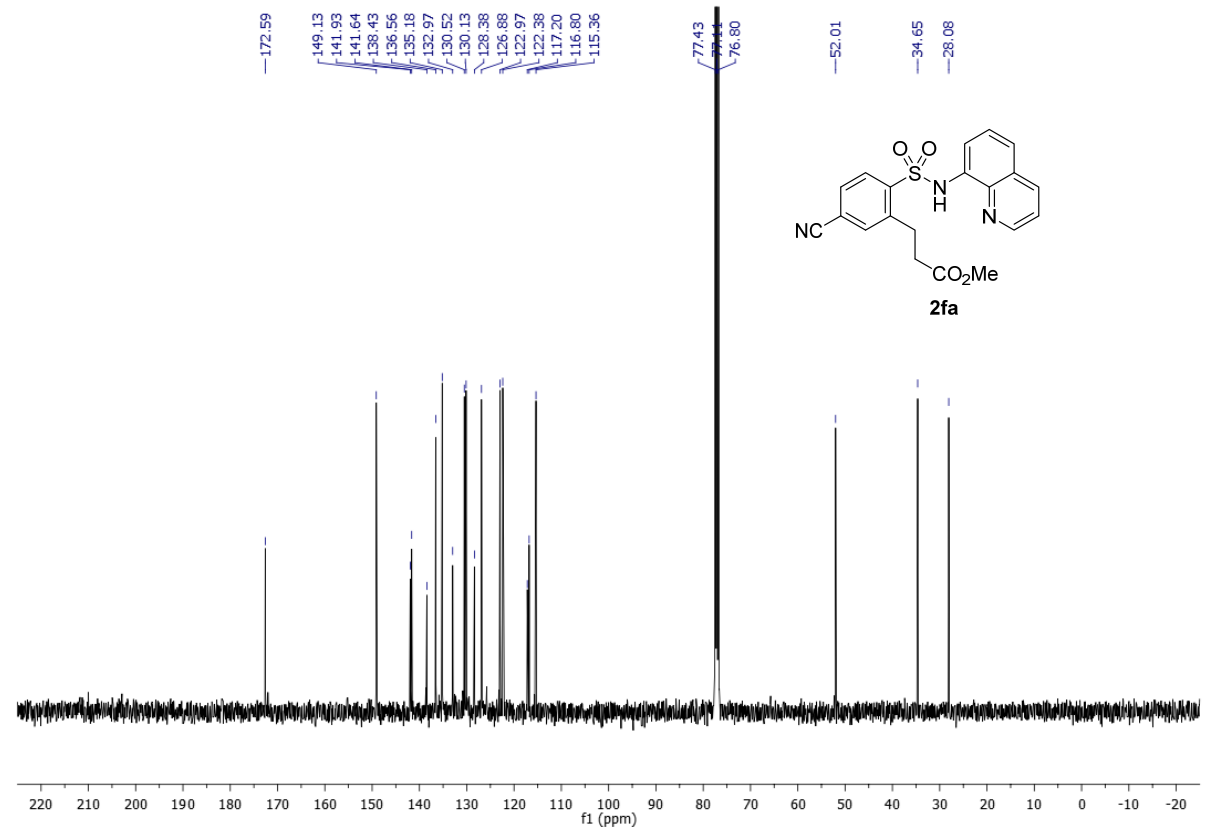

9.39
8.79
8.79
8.78
8.11
8.11
8.09
8.07
7.73
7.71
7.48
7.46
7.43
7.43
7.42
7.41
7.40
7.39
7.37
7.36
7.35
7.34
3.52
3.50
3.48
2.80
2.77
2.76
0.32
0.00
0.68
1.00
2.05
1.05
10.55
3.07
2.04
2.01
f1 (ppm)
Ph
CO2Me
2da

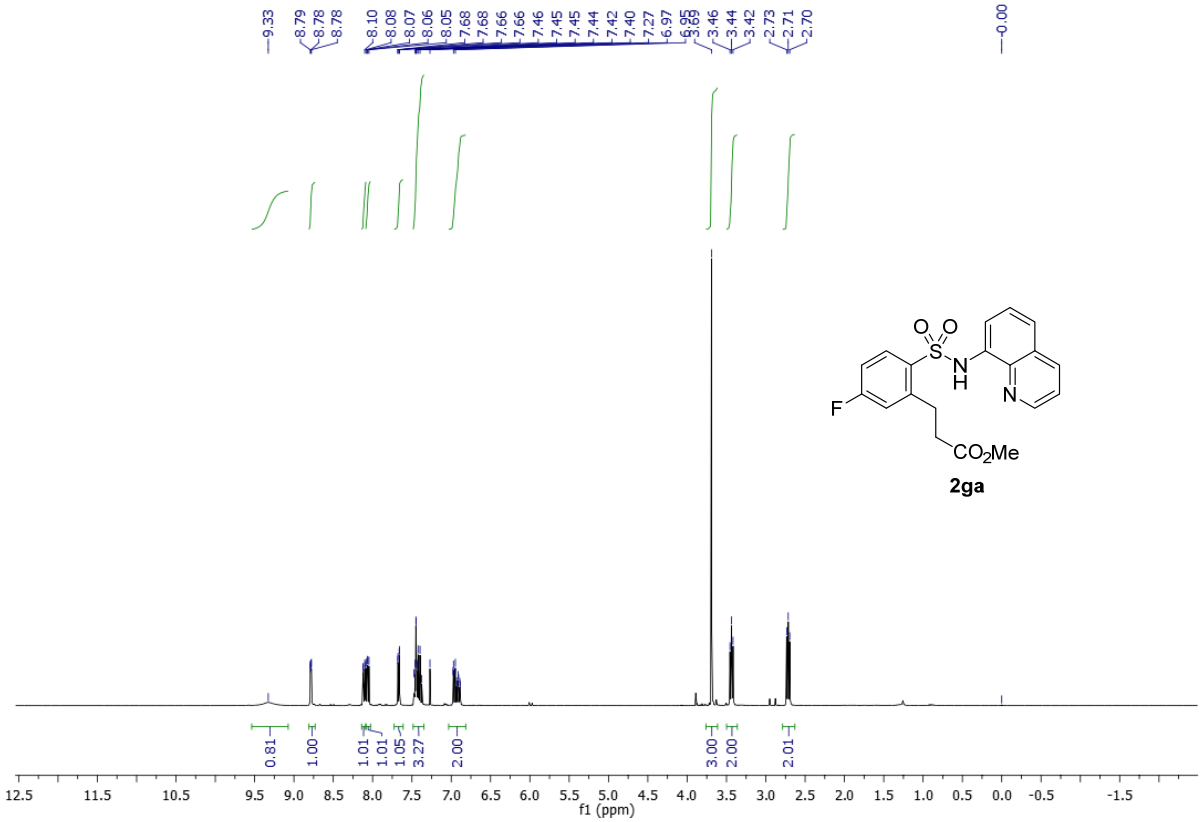

173.18
148.84
145.88
140.56
138.93
138.34
136.38
136.17
133.71
130.79
130.38
129.02
128.55
128.28
127.21
126.90
125.04
122.15
122.08
114.53
77.48
77.16
76.84
51.81
35.36
28.56
f1 (ppm)
Ph
CO2Me
2da


9.30
8.78
8.77
8.77
8.03
8.00
7.41
7.41
7.39
7.37
7.34
6.73
6.72
6.71
6.70
6.69
3.74
3.68
3.41
3.39
3.37
2.72
2.70
2.68
0.00
0.77
1.00
1.02
1.01
1.03
3.12
2.00
3.04
3.00
1.97
1.96
f1 (ppm)
MeO
CO2Me
2ea


173.16
163.05
148.76
142.34
138.35
136.34
133.84
132.82
129.10
128.25
126.86
122.09
121.87
117.23
114.36
111.06
77.48
77.16
76.84
55.51
51.79
35.12
28.41
f1 (ppm)
MeO
CO2Me
2ea


9.31
8.81
8.80
8.15
8.13
8.10
8.08
7.72
7.70
7.57
7.52
7.50
7.49
7.47
7.46
7.44
7.42
7.40
7.26
3.71
3.50
3.48
3.46
2.78
2.76
2.74
0.00
NC
CO2Me
2fa
0.57
1.00
1.02
1.05
1.08
1.00
4.25
3.00
2.03
1.99
f1 (ppm)


172.59
149.13
141.93
141.64
138.43
136.56
135.18
132.97
130.52
130.13
128.38
126.88
122.97
122.38
117.20
116.80
115.36
77.43
77.11
76.80
52.01
34.65
28.08
NC
CO2Me
2fa
f1 (ppm)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8.07
8.06
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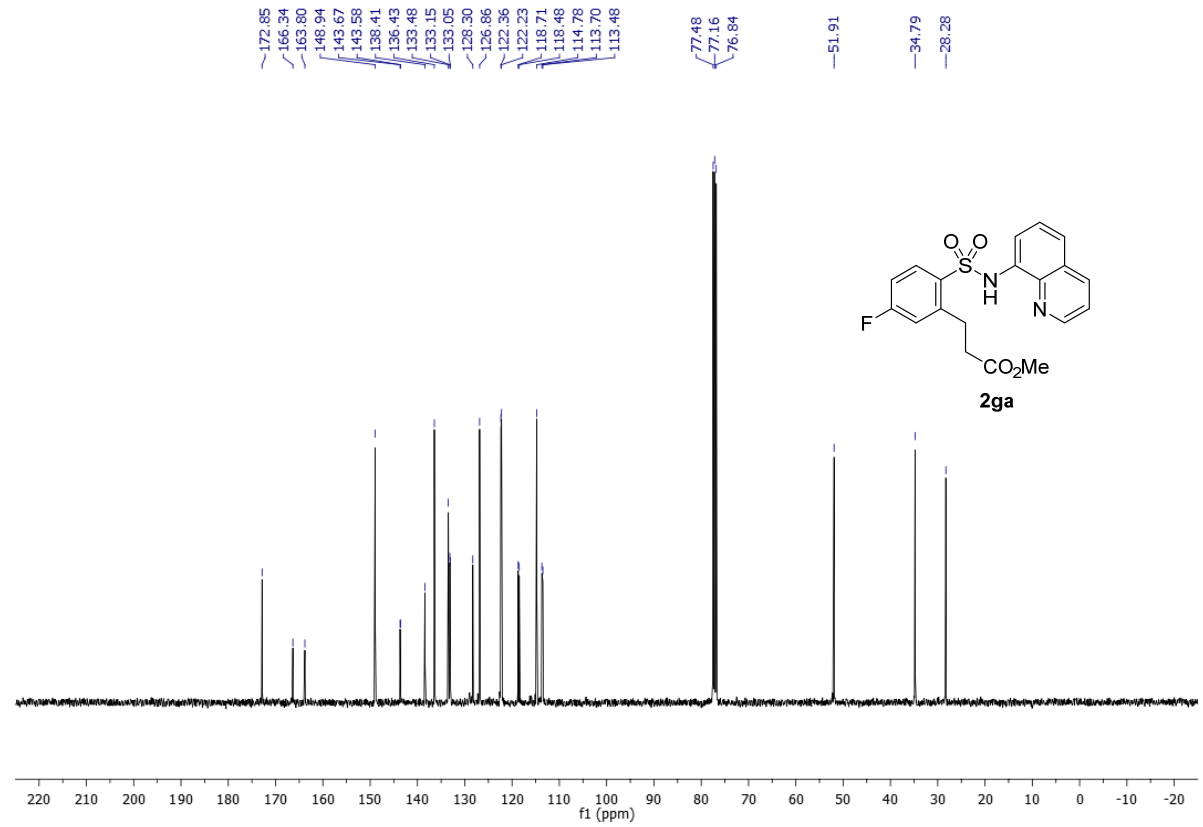
7.68
7.66
7.66
7.46
7.45
7.45
7.44
7.42
7.40
7.27
6.97
3.46
3.44
3.42
2.73
2.71
2.70
0.00
0.81
1.00
1.01
1.01
1.05
3.27
2.00
3.00
2.00
2.01
f1 (ppm)
F
CO2Me
2ga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133.15
133.05
128.30
126.86
122.36
122.23
118.71
118.48
114.78
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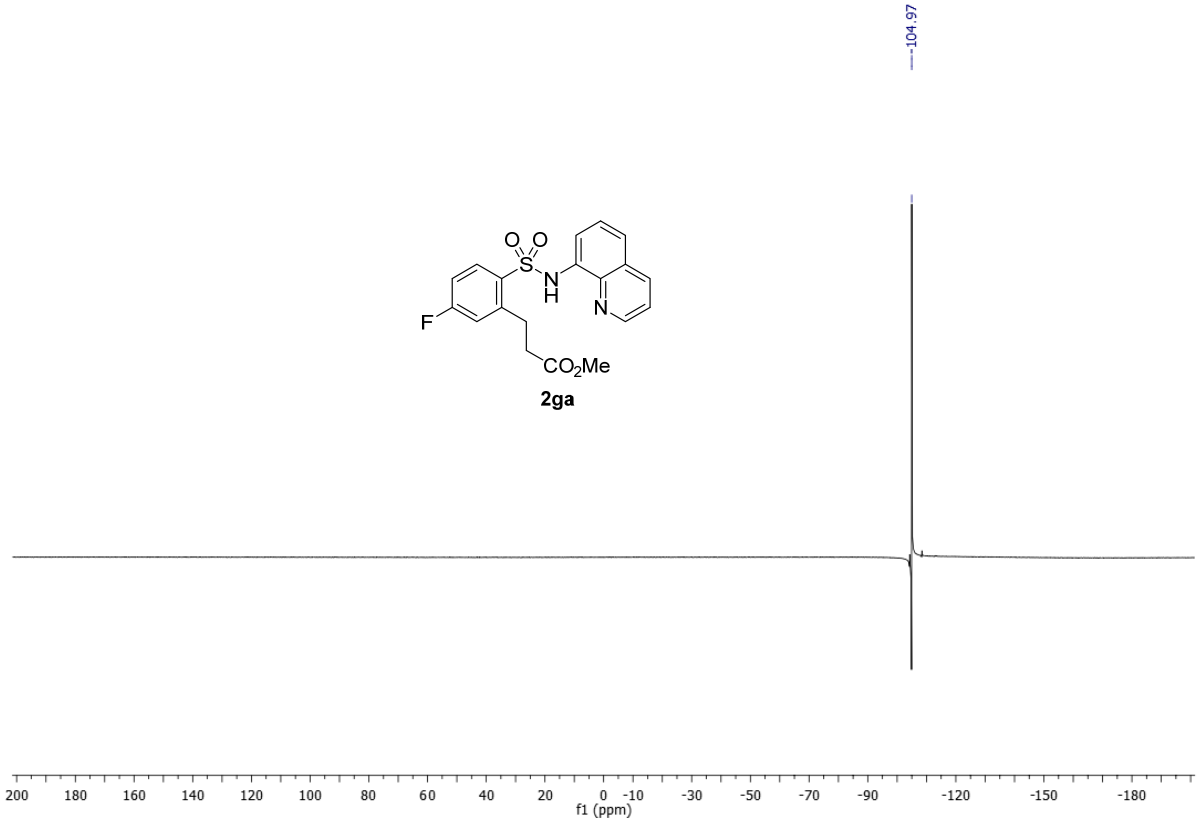
77.48
77.16
76.84
51.91
34.79
28.28
f1 (ppm)
F
CO2Me
2ga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CO2Me
2ga
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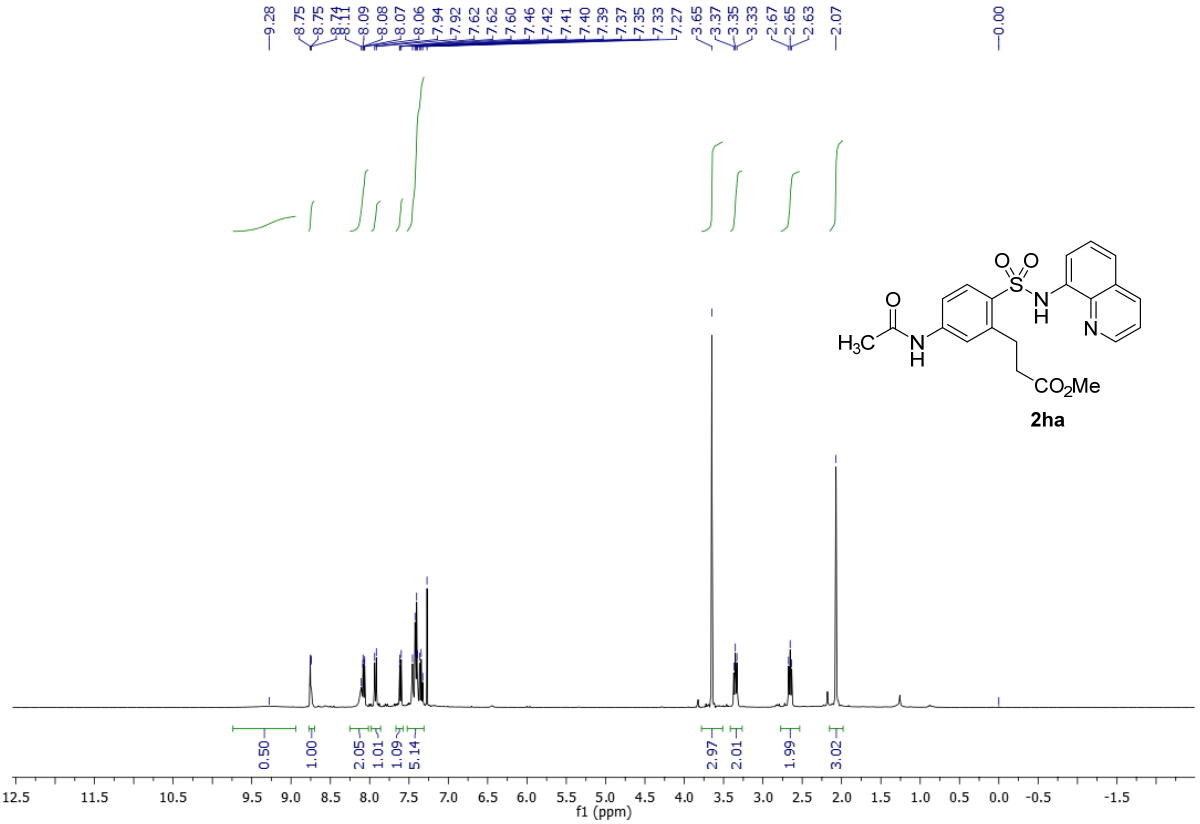
f1 (ppm)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8.07
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7.62
7.62
7.60
7.46
7.42
7.41
7.40
7.39
7.37
7.35
7.33
7.27
3.65
3.37
3.35
3.33
2.67
2.65
2.63
2.07
0.00
H3C
CO2Me
2ha
0.50
1.00
2.05
1.01
1.09
5.14
2.97
2.01
1.99
3.02
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.5
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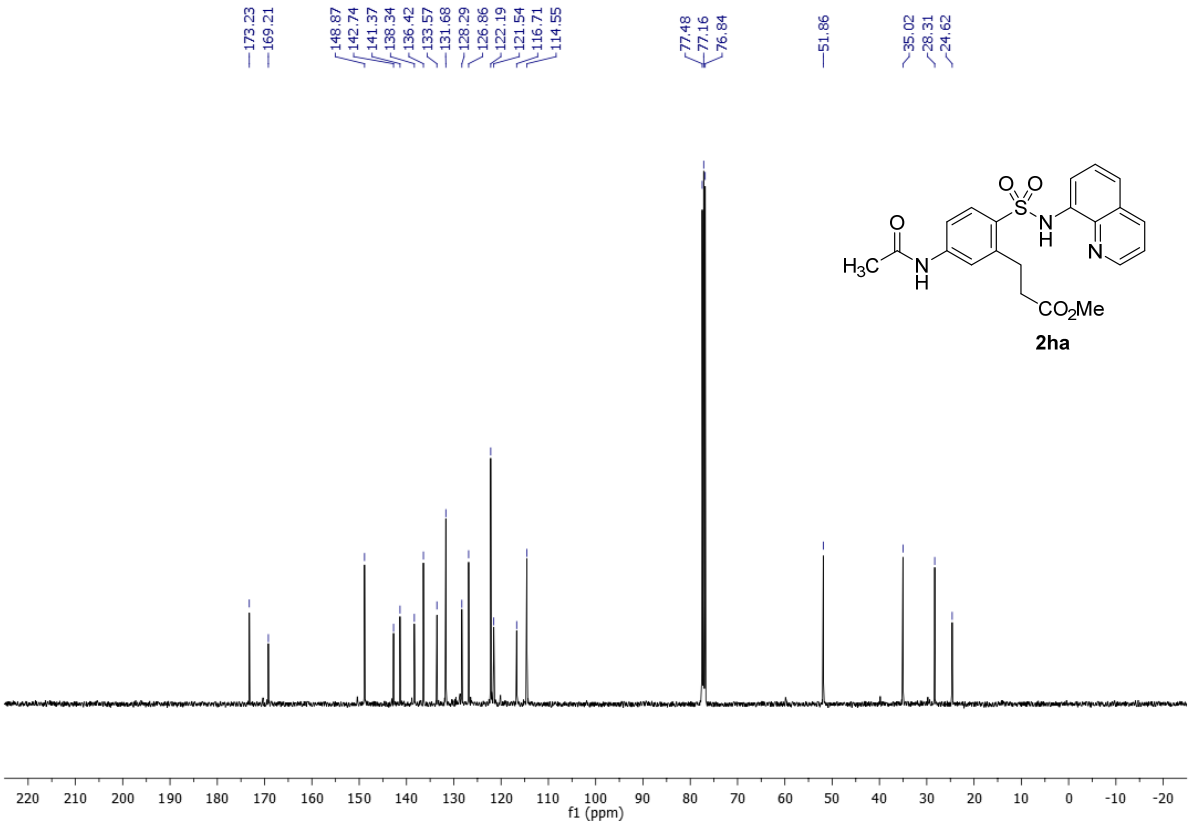
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122.19
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114.55
77.48
77.16
76.84
51.86
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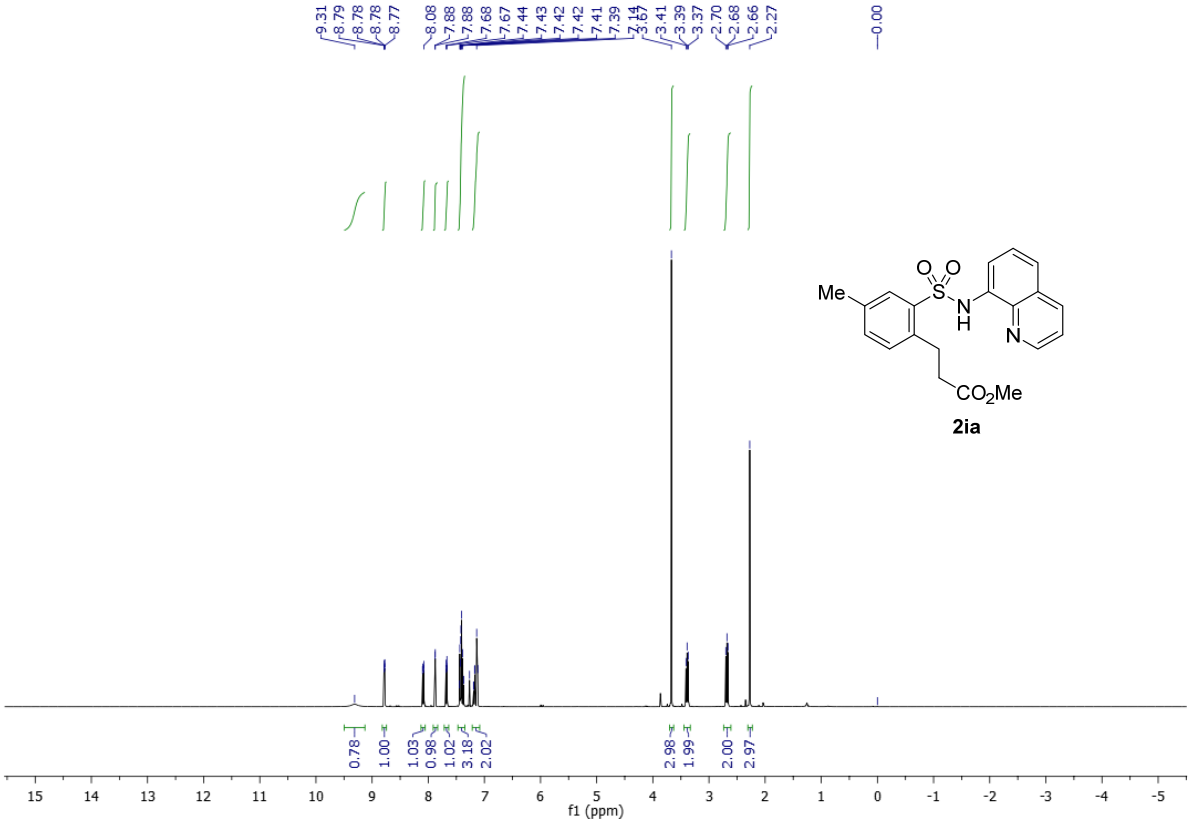
O
O
S
N
H
O
H3C
N
H
N
CO2Me
2ha
f1 (ppm)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7.43
7.42
7.42
7.41
7.39
3.67
3.41
3.39
3.37
2.70
2.68
2.66
2.27
0.00
O
O
S
N
H
Me
N
CO2Me
2ia
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0.98
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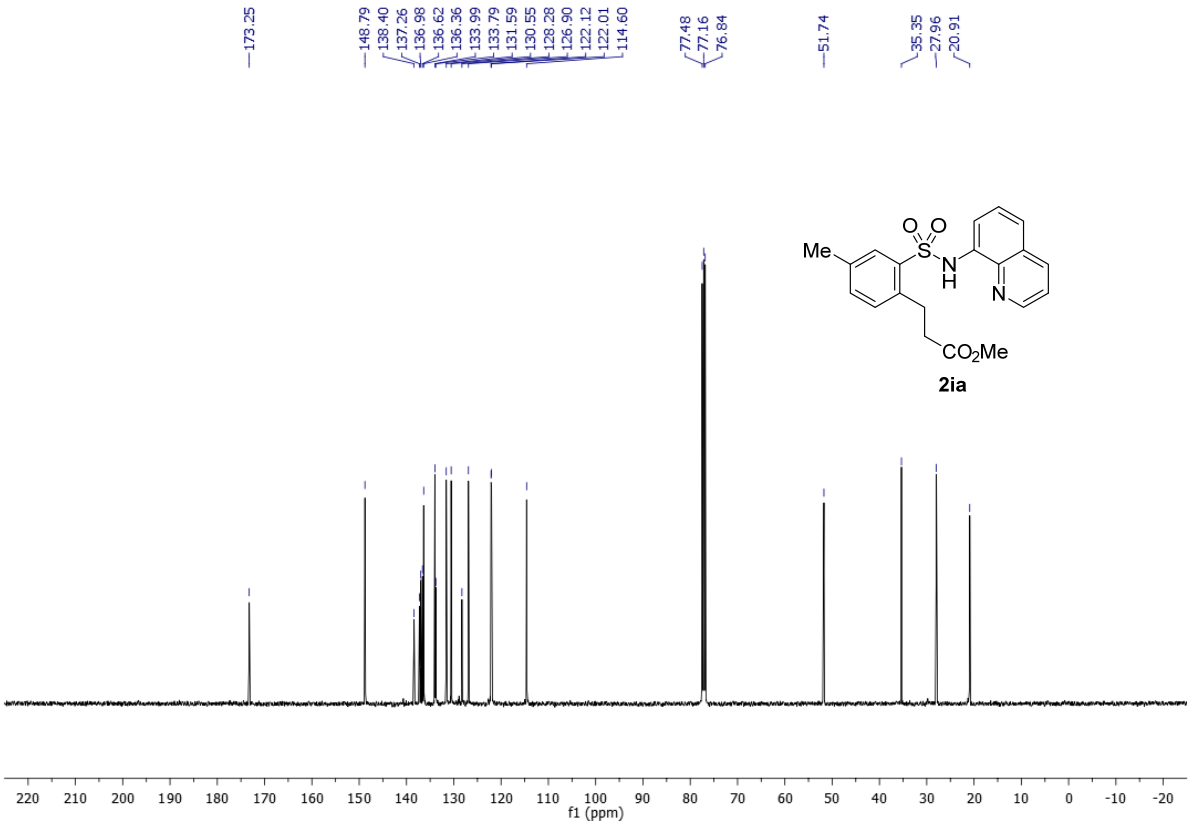
3.18
2.02
2.98
1.99
2.00
2.97
f1 (ppm)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122.01
114.60
77.48
77.16
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27.96
20.91
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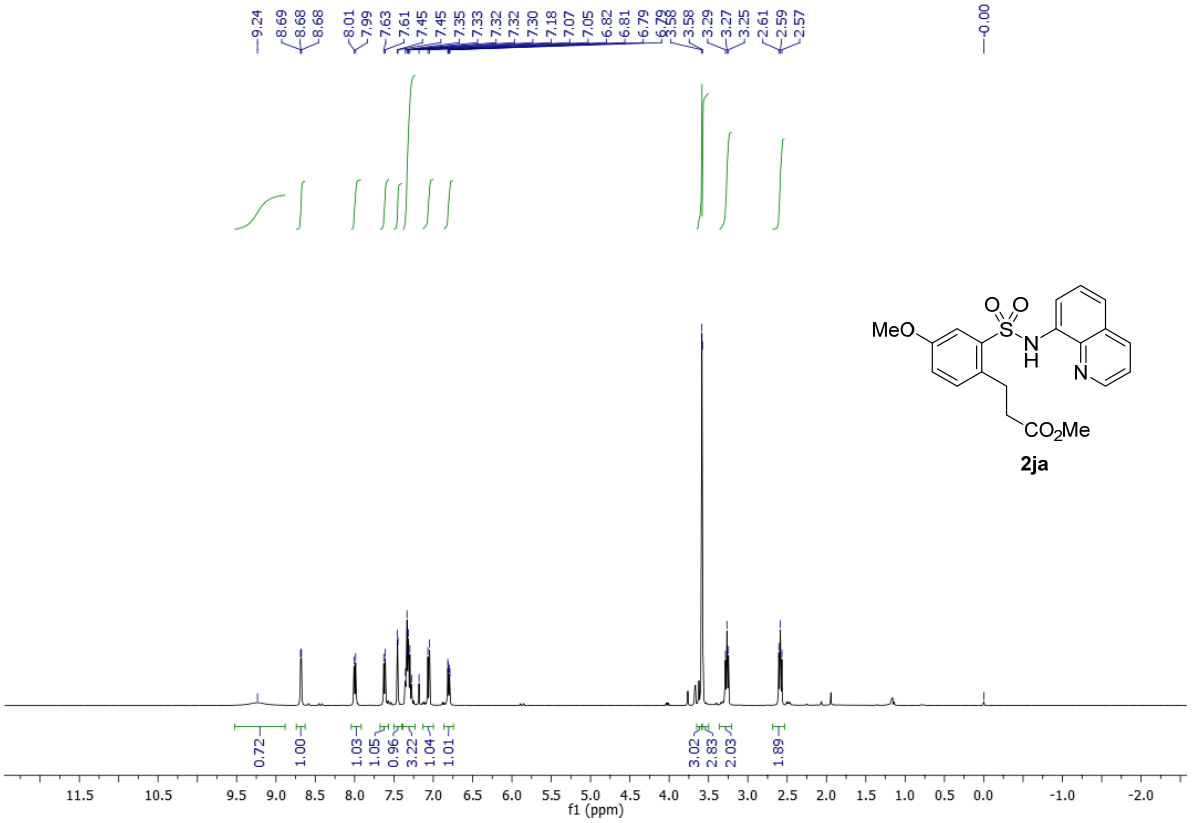
S
N
H
N
CO2Me
2ia
f1 (ppm)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7.32
7.32
7.30
7.18
7.07
7.05
6.82
6.81
6.79
6.78
3.58
3.29
3.27
3.25
2.61
2.59
2.57
0.00
MeO
O
O
S
N
H
N
CO2Me
2ja
0.72
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0.96
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1.04
1.01
3.02
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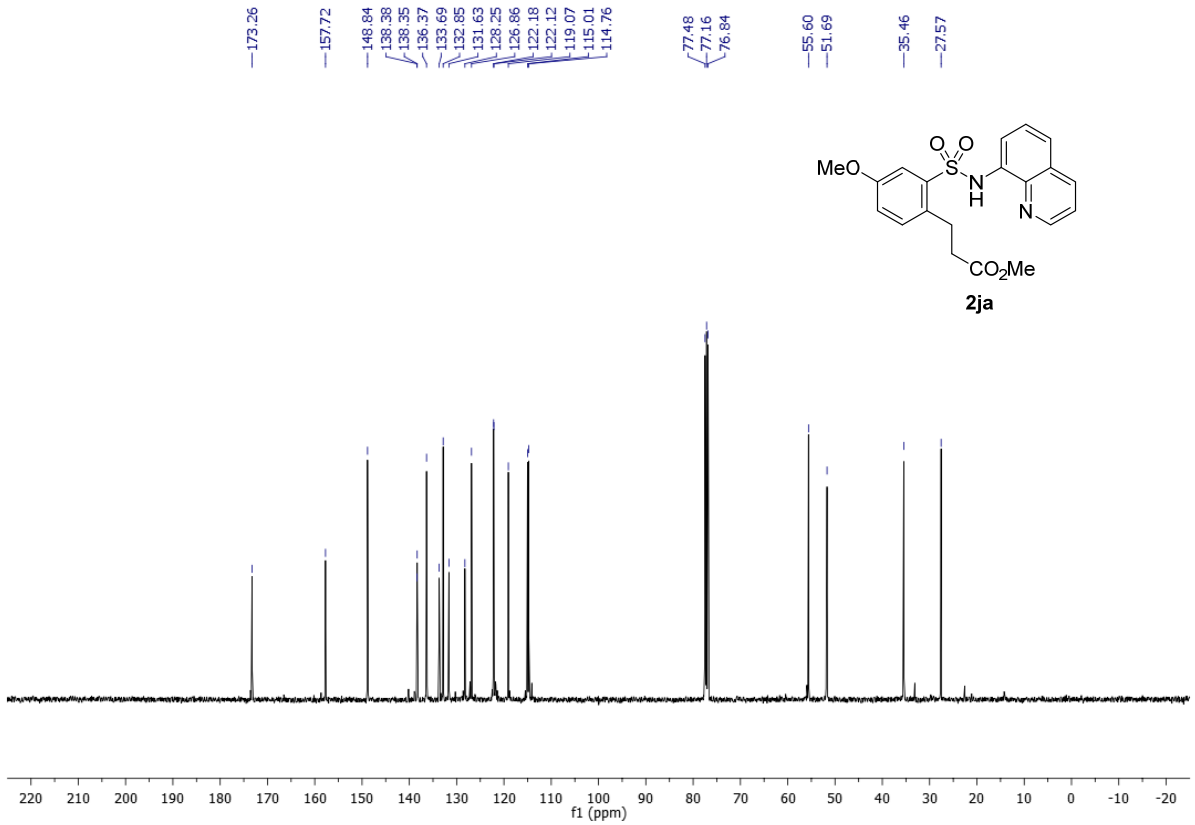
2.03
1.89
f1 (ppm)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126.86
122.18
122.12
119.07
115.01
114.76
77.48
77.16
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27.57
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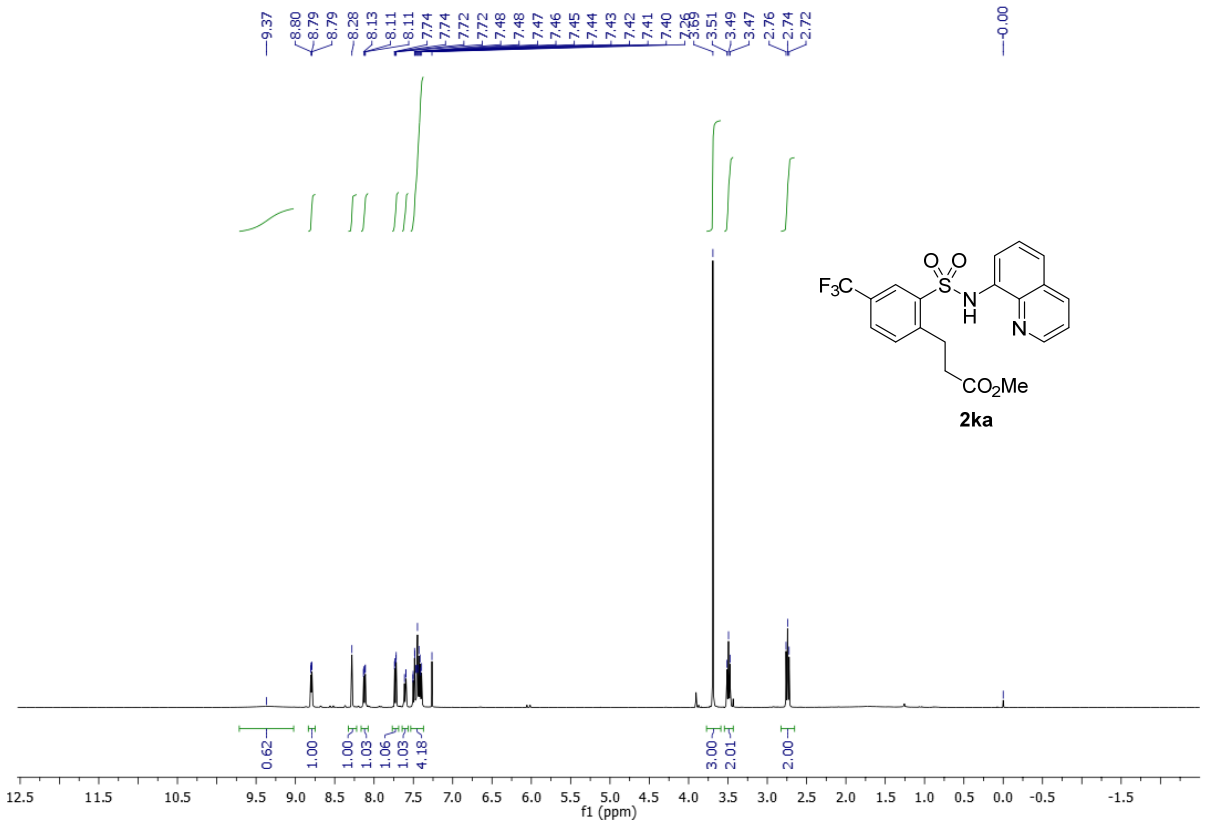
N
H
N
CO2Me
2ja
f1 (ppm)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7.44
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7.42
7.41
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3.51
3.49
3.47
2.76
2.74
2.72
0.00
F3C
O
O
S
N
H
N
CO2Me
2ka
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1.00
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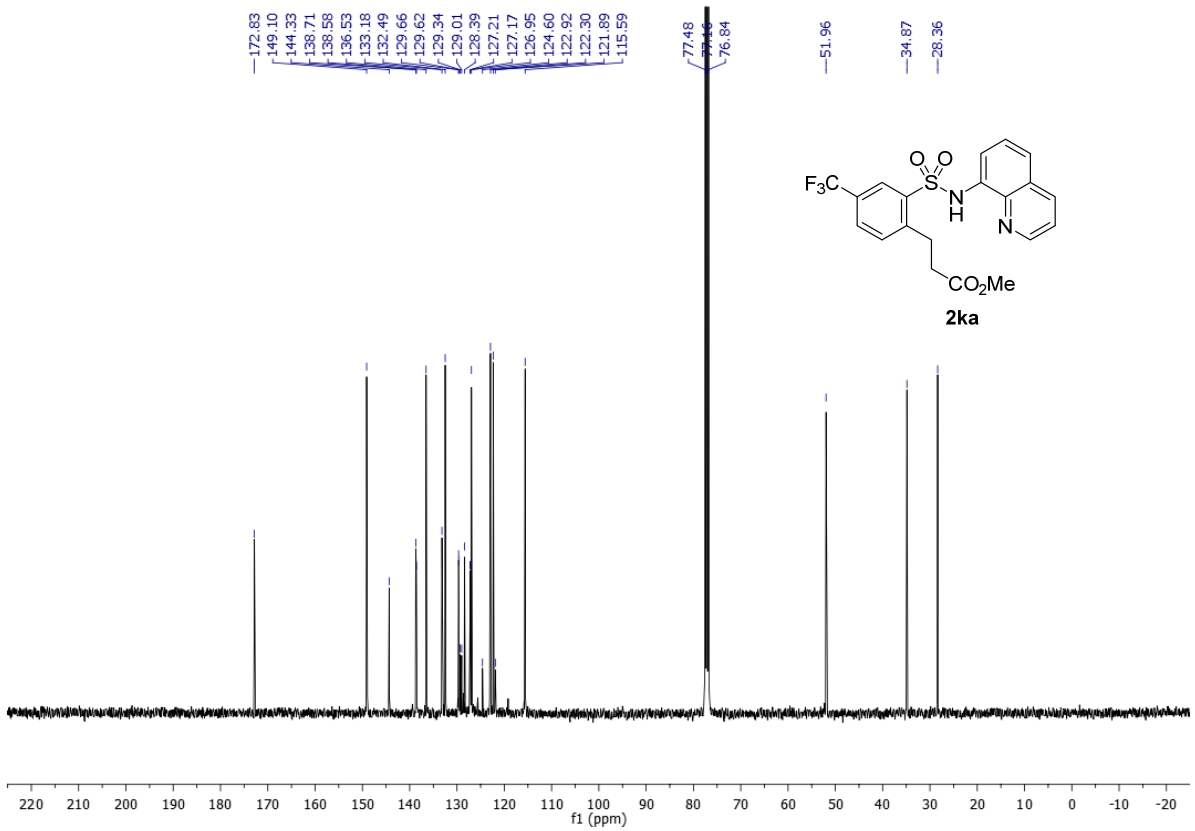
1.03
4.18
3.00
2.01
2.00
f1 (ppm)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127.17
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122.92
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77.16
76.84
51.96
34.87
28.36
F3C
O
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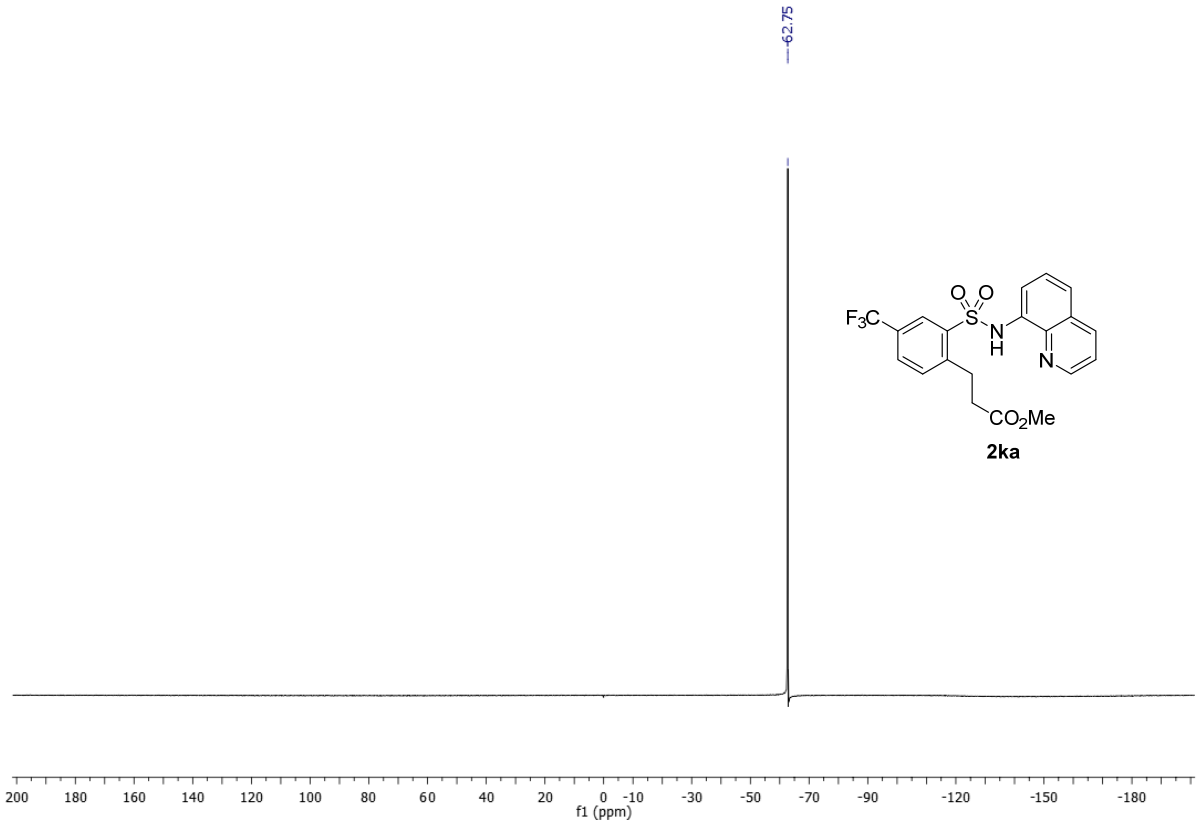
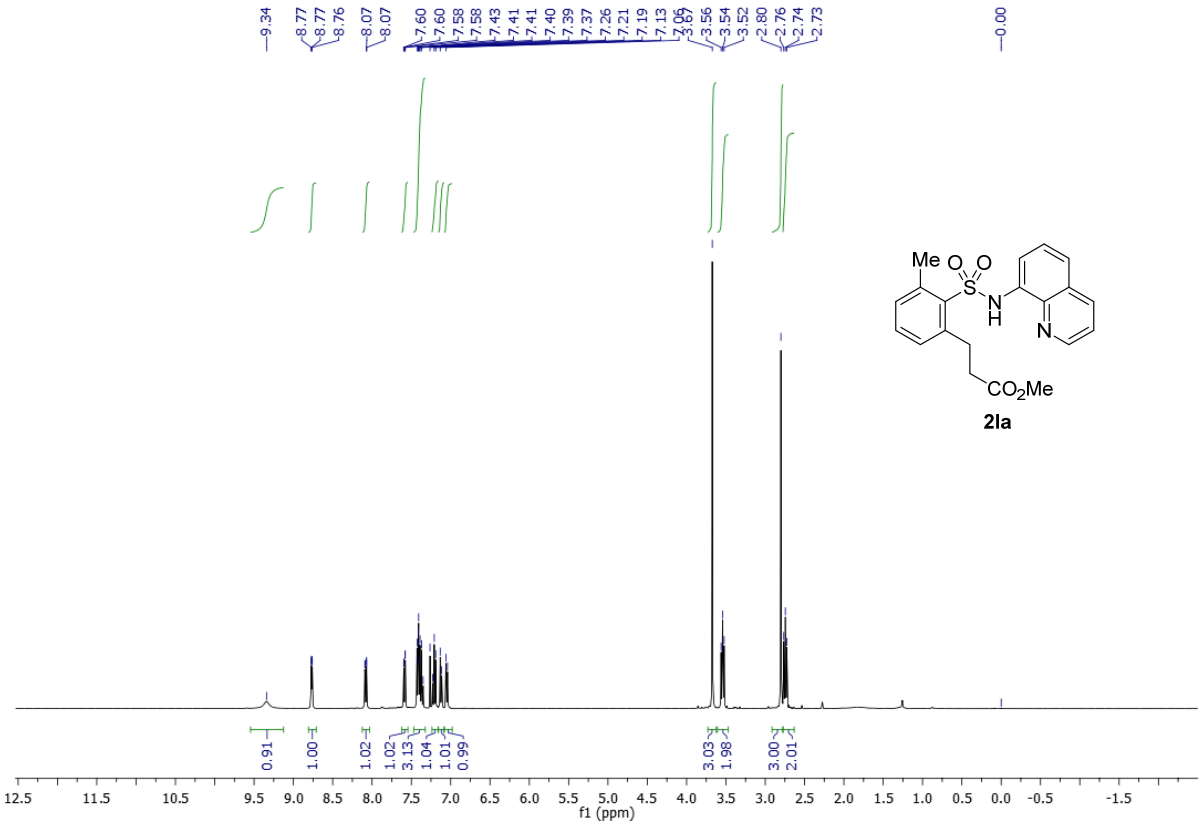
S
N
H
N
CO2Me
2ka
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)


62.75
F3C
O
O
S
N
H
N
CO2Me
2ka
200 180 160 140 120 100 80 60 40 20 0 -10 -30 -50 -70 -90 -120 -150 -180
f1 (ppm)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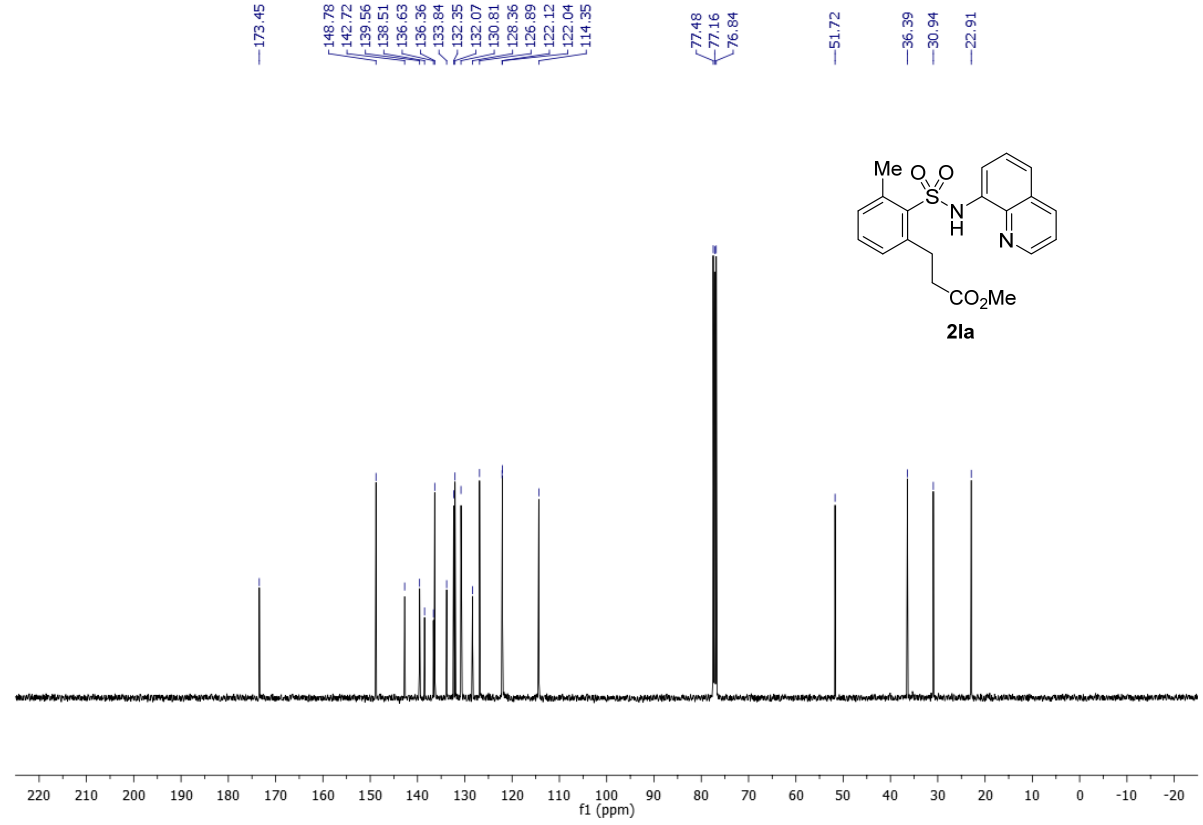
O
S
N
H
N
CO2Me
2la
f1 (ppm)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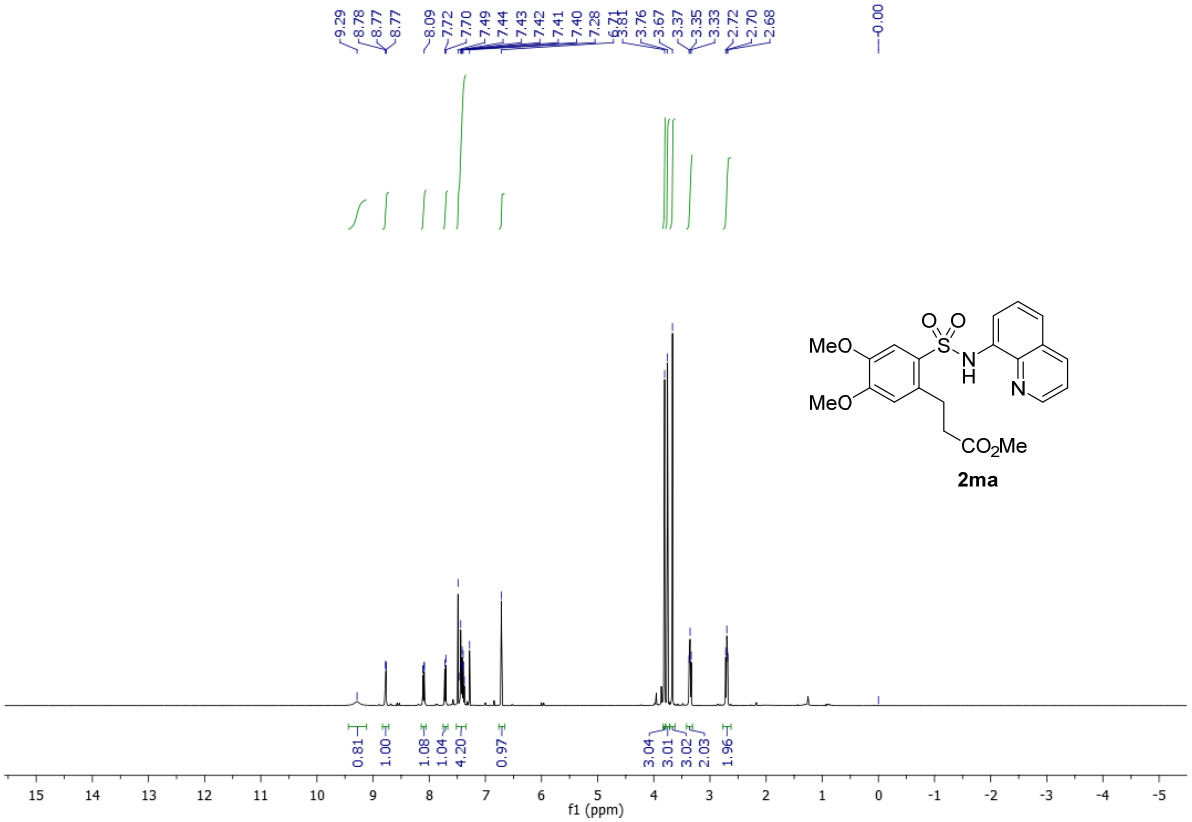
f1 (ppm)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7.43
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7.28
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3.67
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3.33
2.72
2.70
2.68
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MeO
MeO
O
O
S
N
H
N
CO2Me
2ma
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3.02
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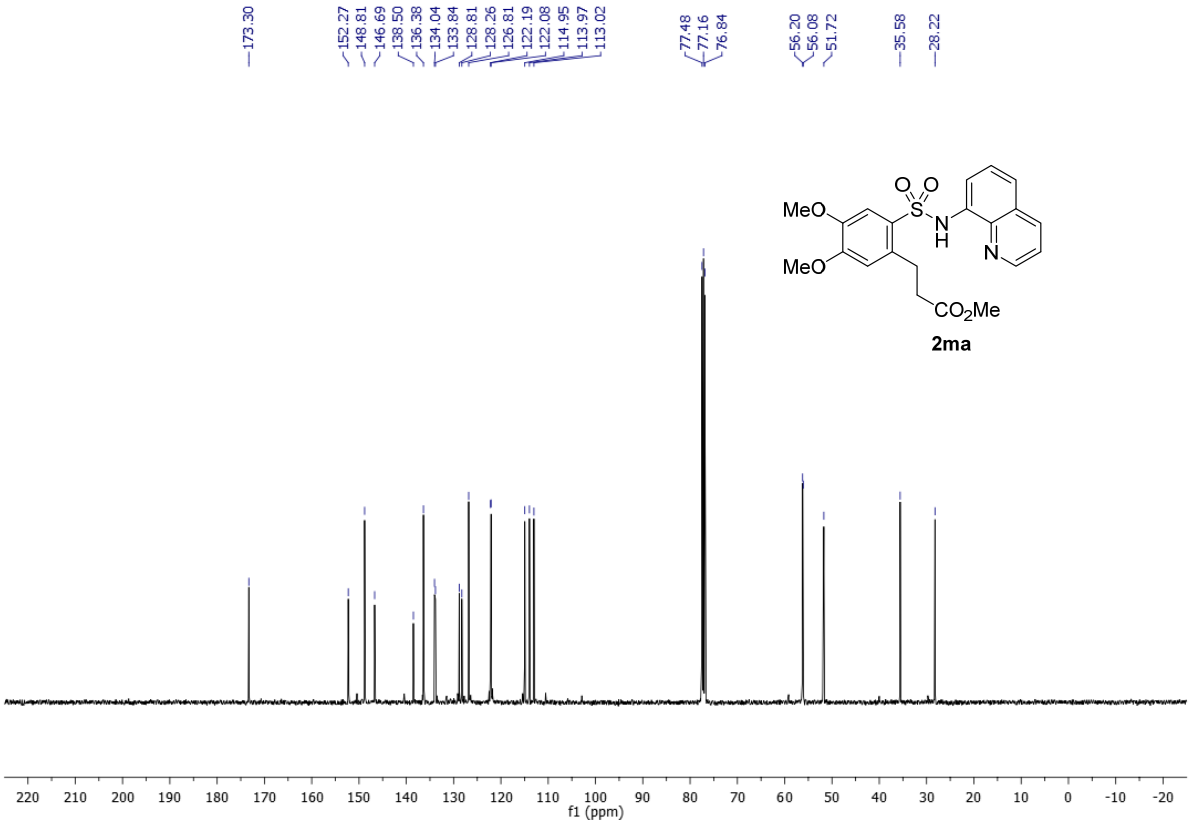
1.96
f1 (ppm)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122.08
114.95
113.97
113.02
77.48
77.16
76.84
56.20
56.08
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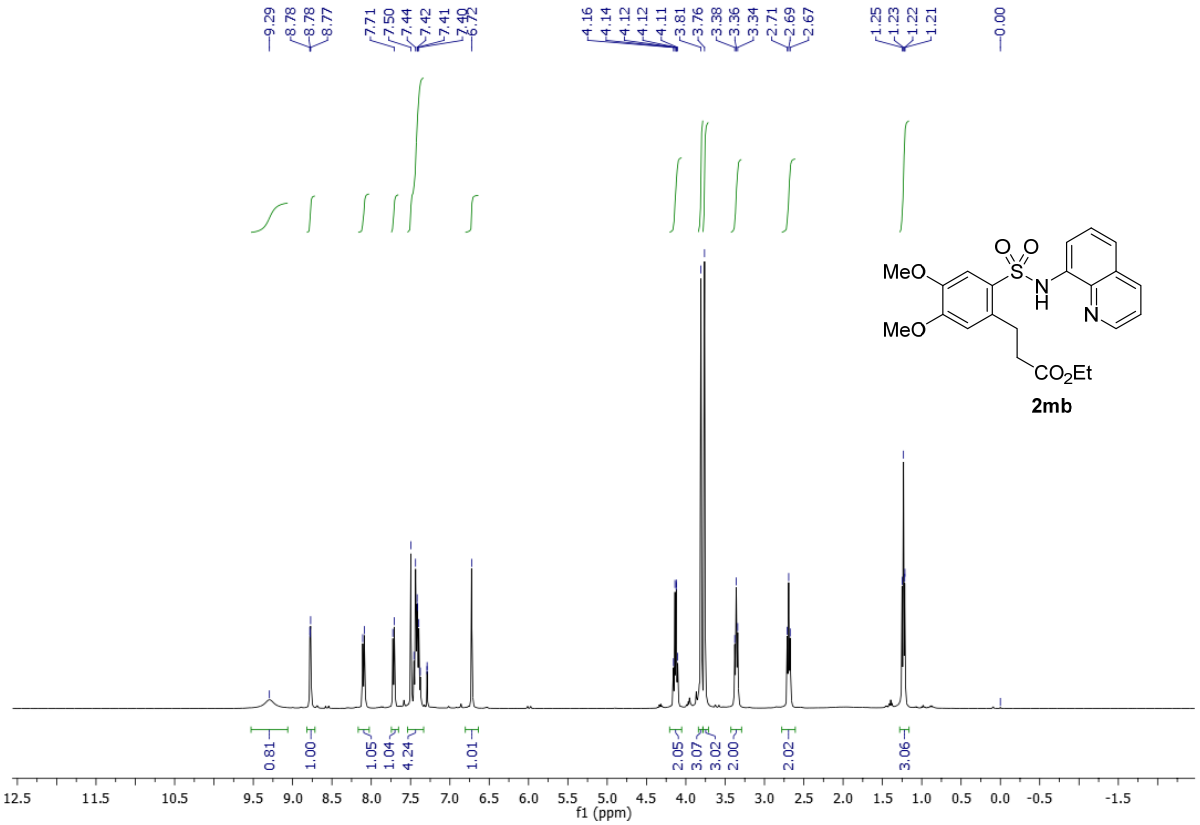
35.58
28.22
MeO
MeO
O
O
S
N
H
N
CO2Me
2ma
f1 (ppm)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8.77
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7.42
7.41
7.40
6.72
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4.14
4.12
4.12
4.11
3.81
3.76
3.38
3.36
3.34
2.71
2.69
2.67
1.25
1.23
1.22
1.21
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MeO
MeO
O
O
S
N
H
N
CO2Et
2mb
0.81
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4.24
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3.07
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2.00
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3.06
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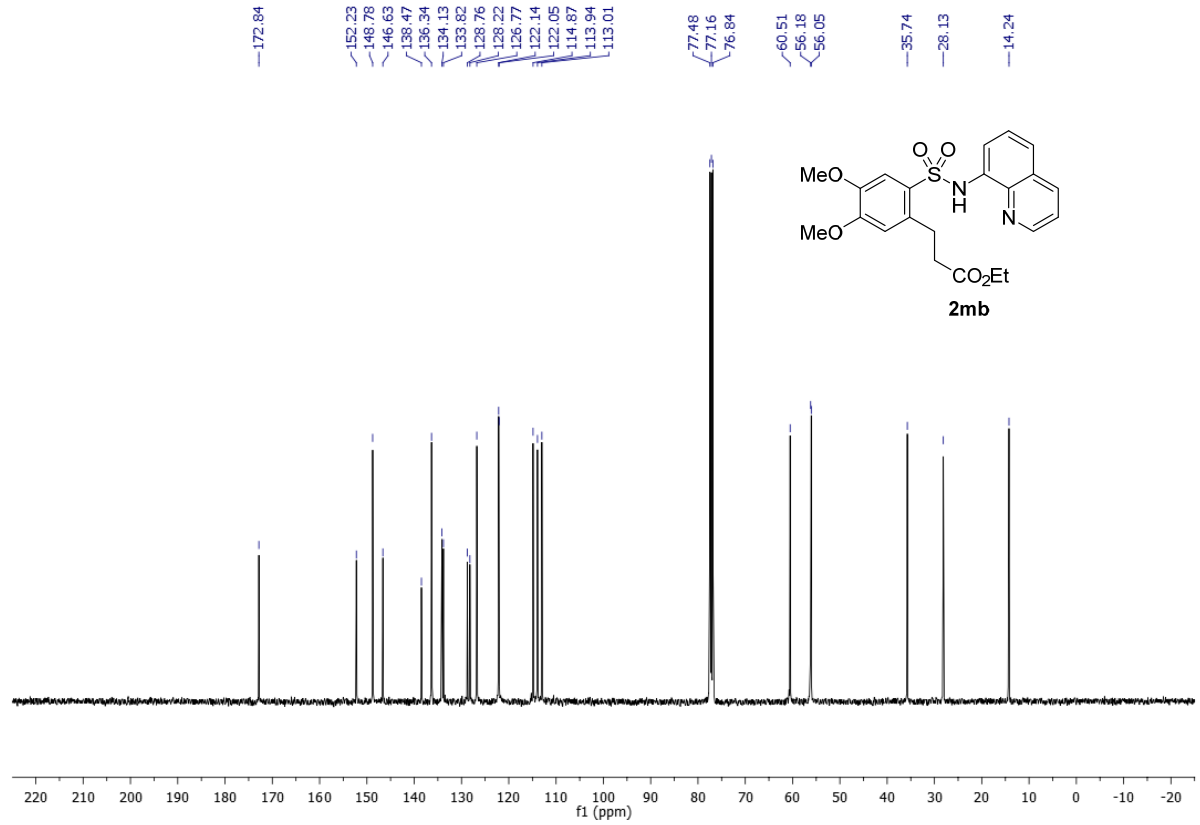
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113.94
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77.16
76.84
60.51
56.18
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14.24
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MeO
O
O
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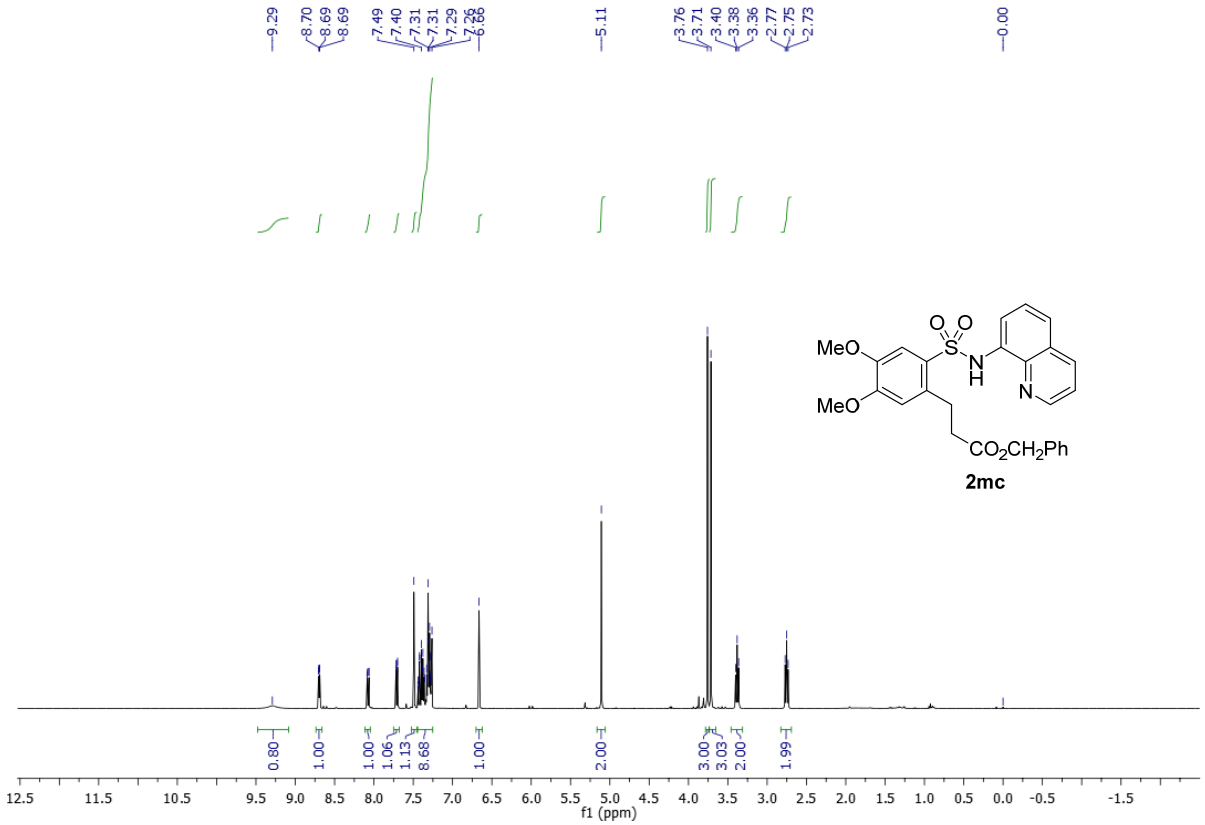
N
H
N
CO2Et
2mb
f1 (ppm)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3.71
3.40
3.38
3.36
2.77
2.75
2.73
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MeO
O
O
S
N
H
N
CO2CH2Ph
2mc
0.80
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3.00
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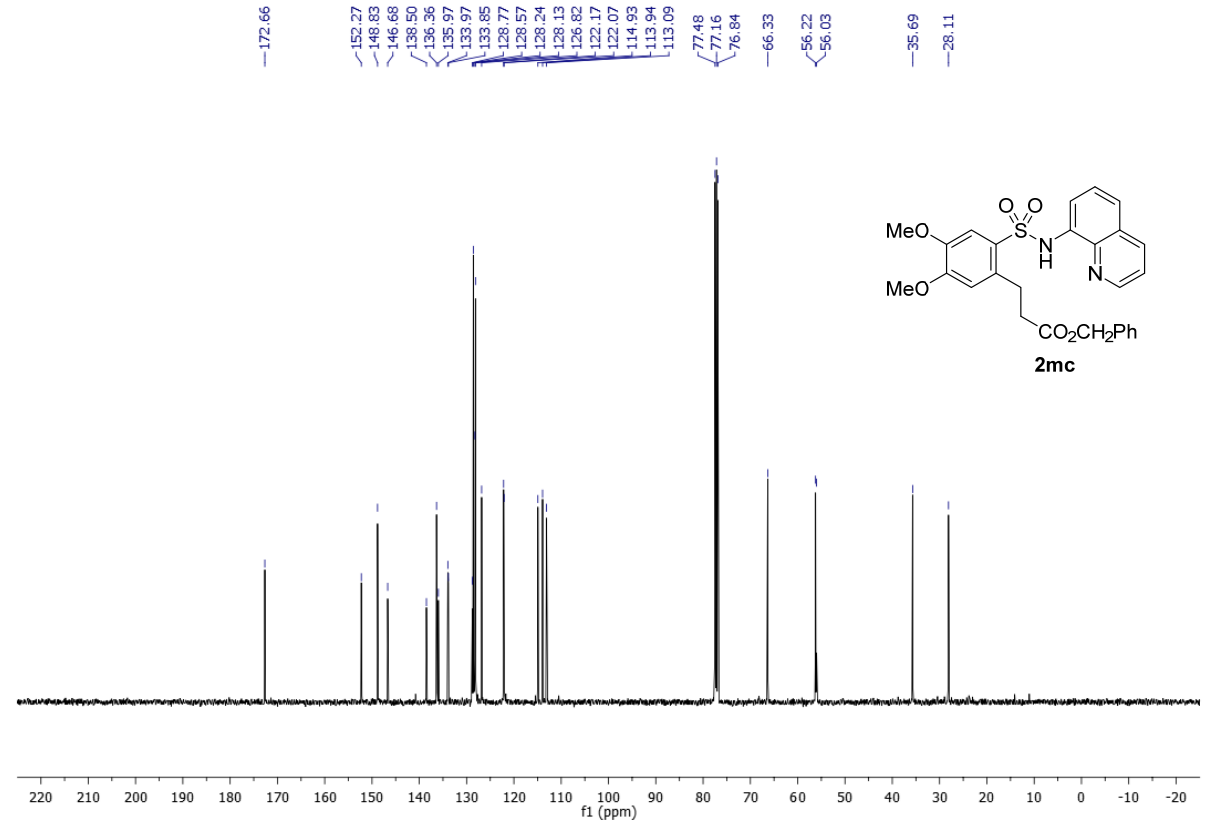
2.00
1.99
f1 (ppm)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122.17
122.07
114.93
113.94
113.09
77.48
77.16
76.84
66.33
56.22
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28.11
MeO
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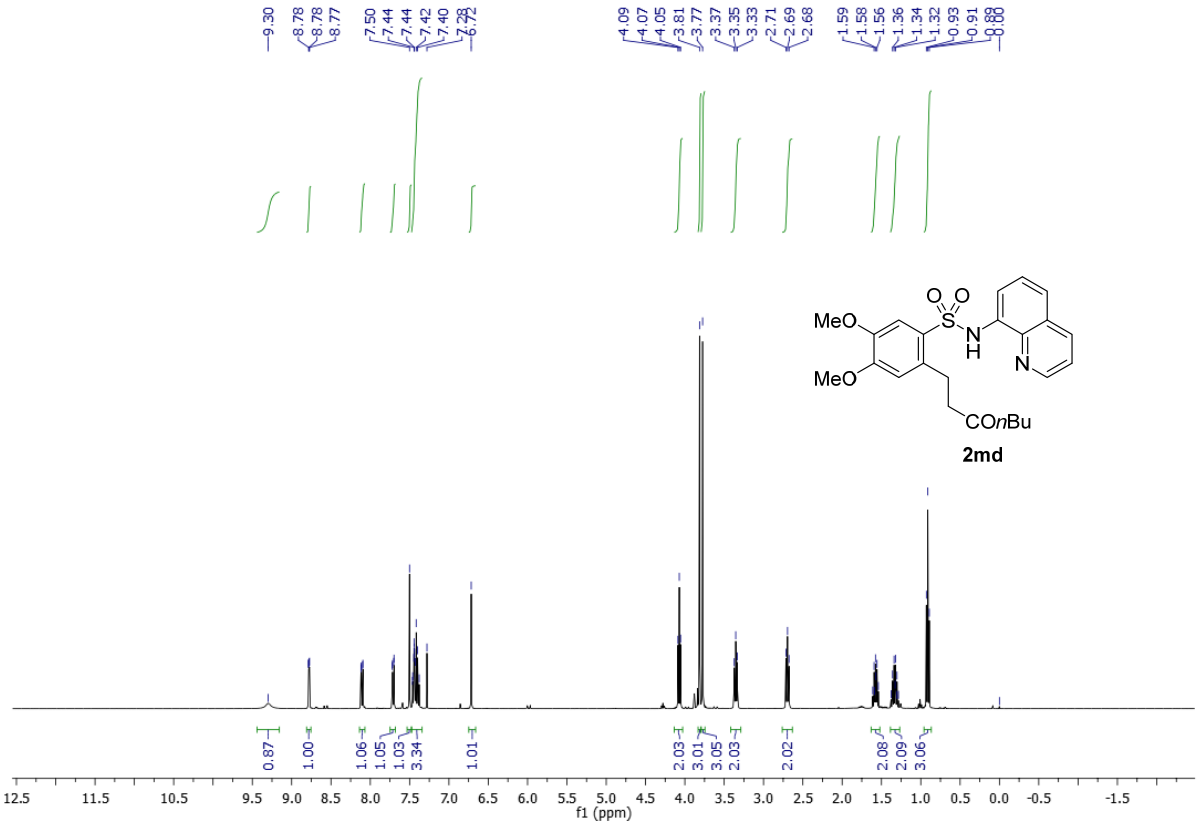
S
N
H
N
CO2CH2Ph
2mc
f1 (ppm)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3.37
3.35
3.33
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2.68
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1.58
1.56
1.36
1.34
1.32
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0.91
0.88
0.00
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MeO
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H
N
COnBu
2md
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1.00
1.06
1.05
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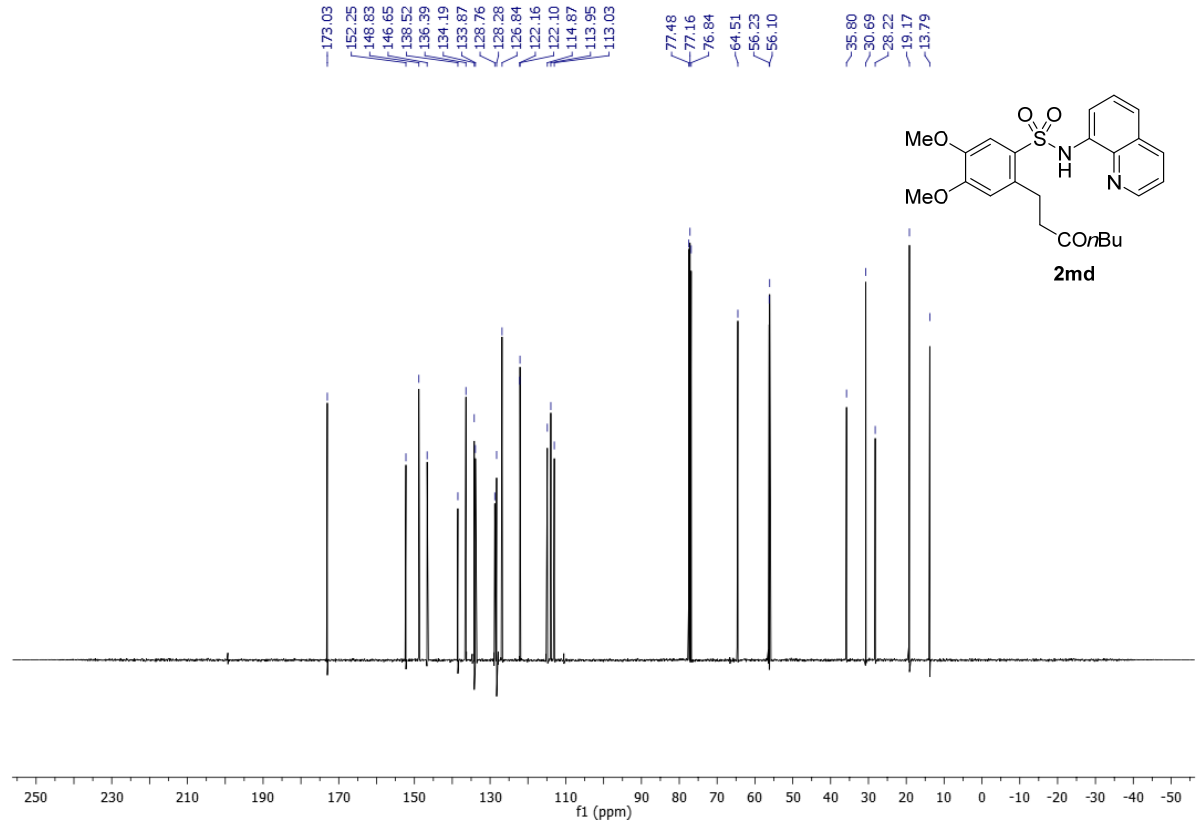
3.06
f1 (ppm)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122.16
122.10
114.87
113.95
113.03
77.48
77.16
76.84
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56.23
56.10
35.80
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13.79
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MeO
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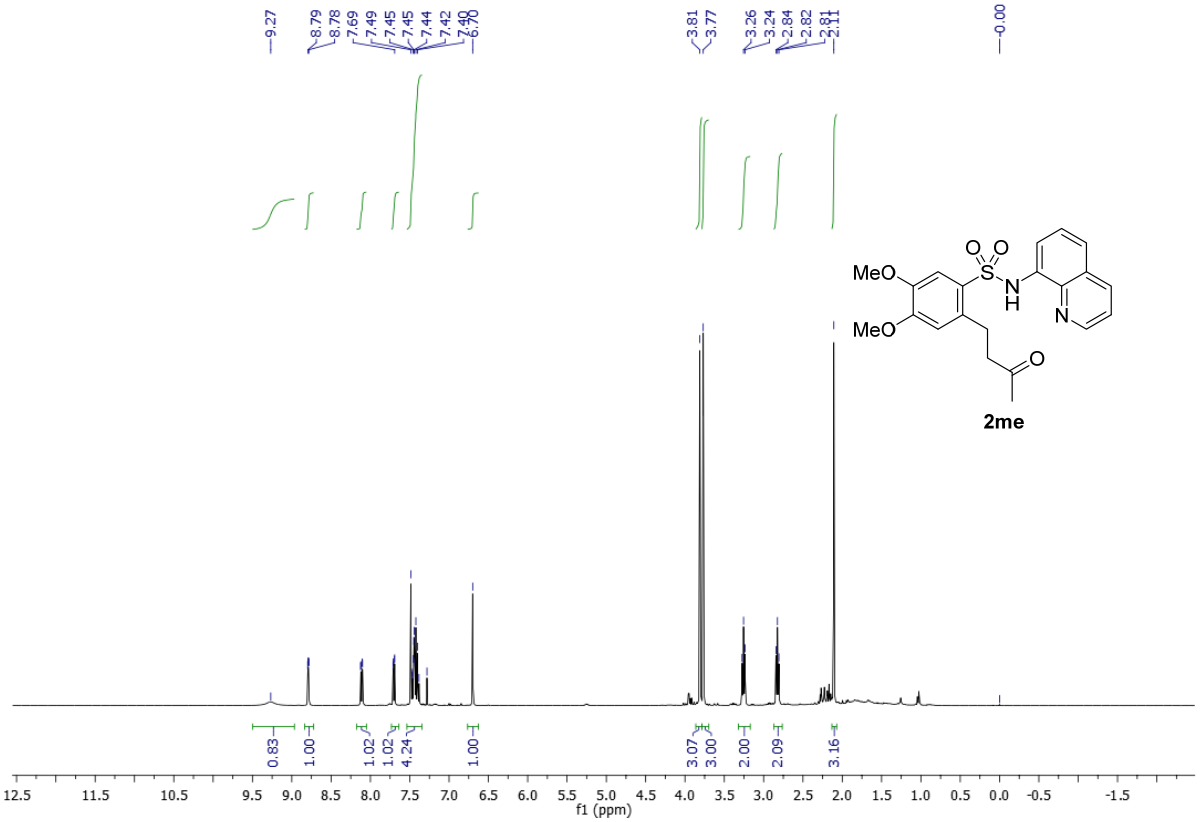
N
H
N
COnBu
2md
f1 (ppm)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6.70
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3.26
3.24
2.84
2.82
2.81
2.11
0.00
MeO
MeO
O
O
S
N
H
N
O
2me
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1.00
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2.00
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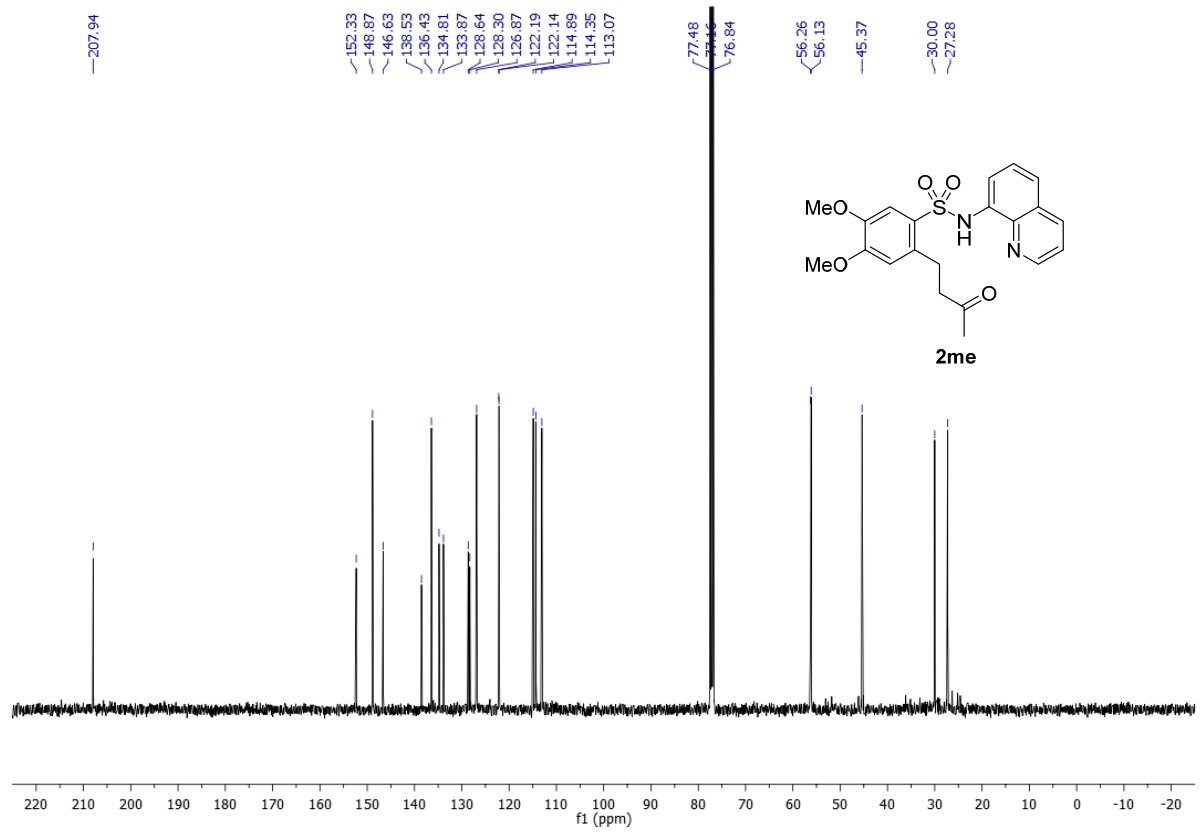
3.16
f1 (ppm)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122.14
114.89
114.35
113.07
77.48
77.16
76.84
56.26
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27.28
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MeO
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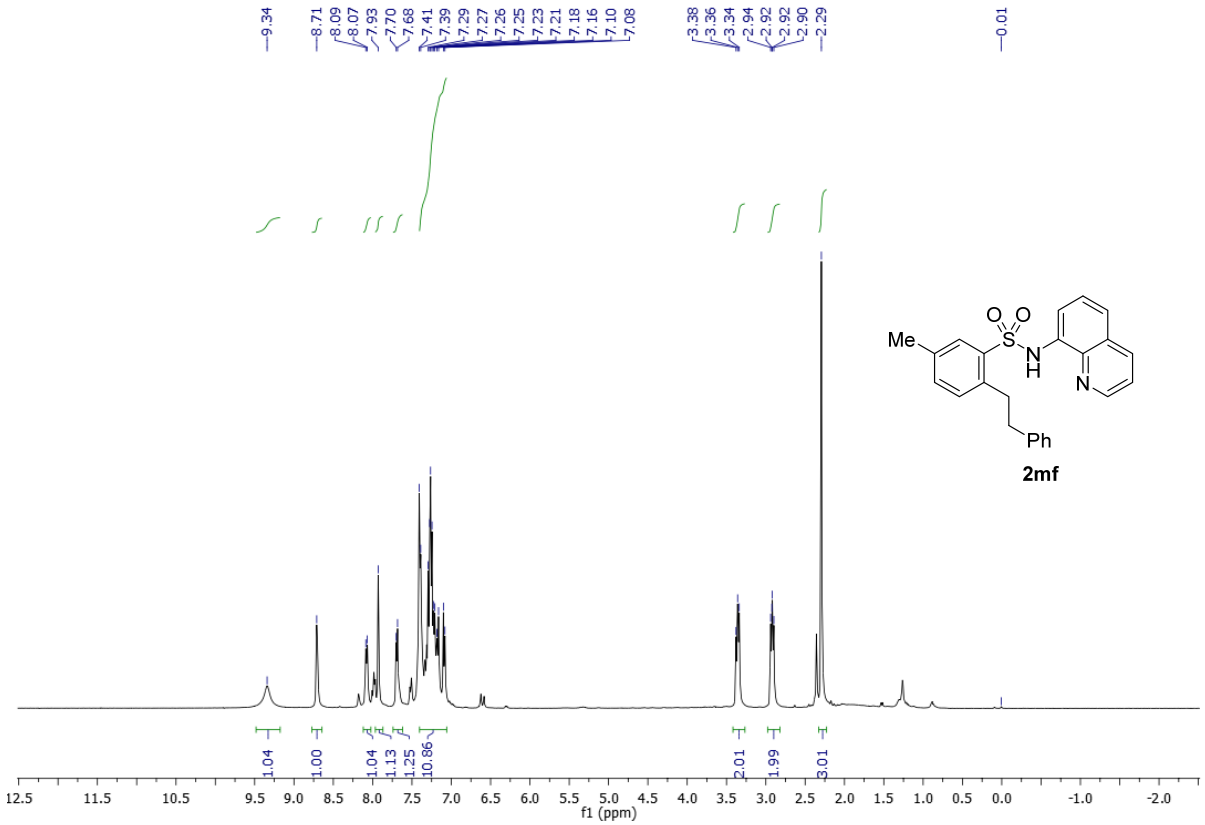
O
S
N
H
N
O
2me
f1 (ppm)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7.23
7.21
7.18
7.16
7.10
7.08
3.38
3.36
3.34
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2.92
2.92
2.90
2.29
0.01
1.04
1.00
1.04
1.13
1.25
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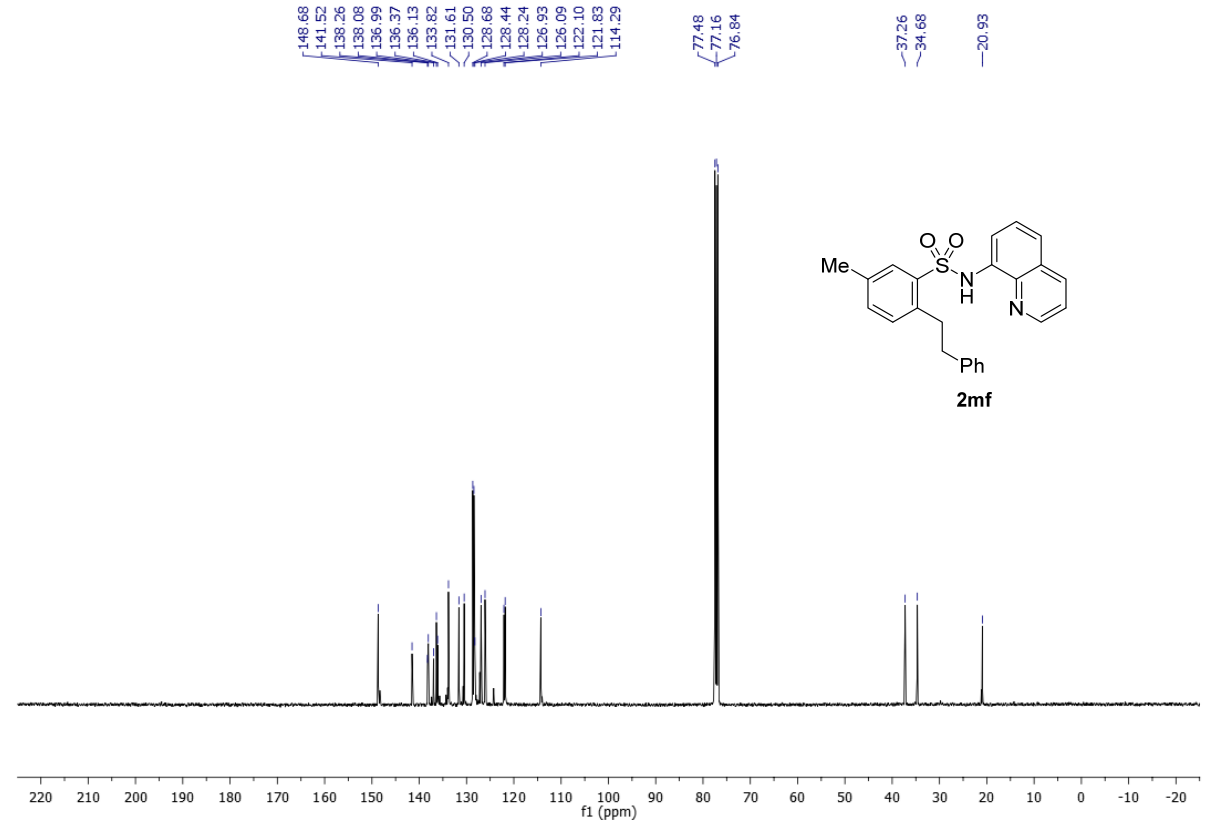
1.99
3.01
f1 (ppm)
Me
O
O
S
N
H
N
Ph
2mf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126.93
126.09
122.10
121.83
114.29
77.48
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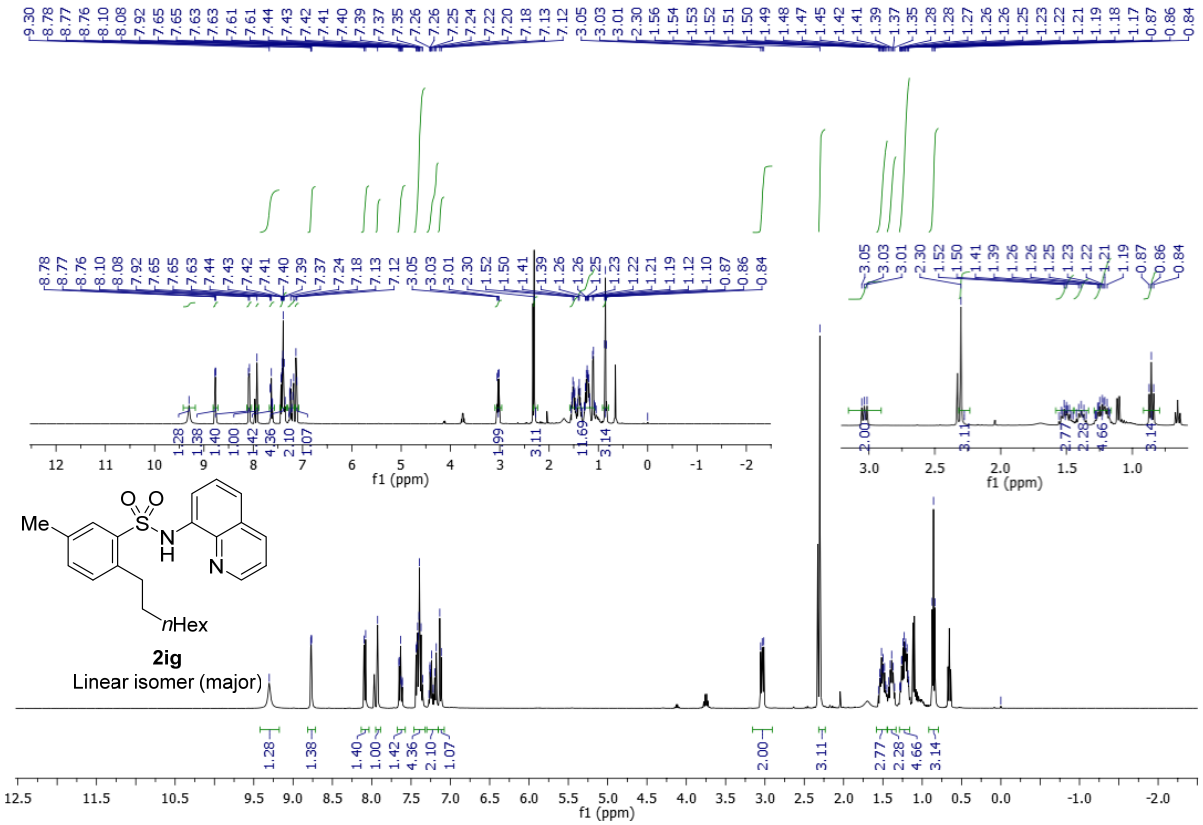
76.84
37.26
34.68
20.93
f1 (ppm)
Me
O
O
S
N
H
N
Ph
2mf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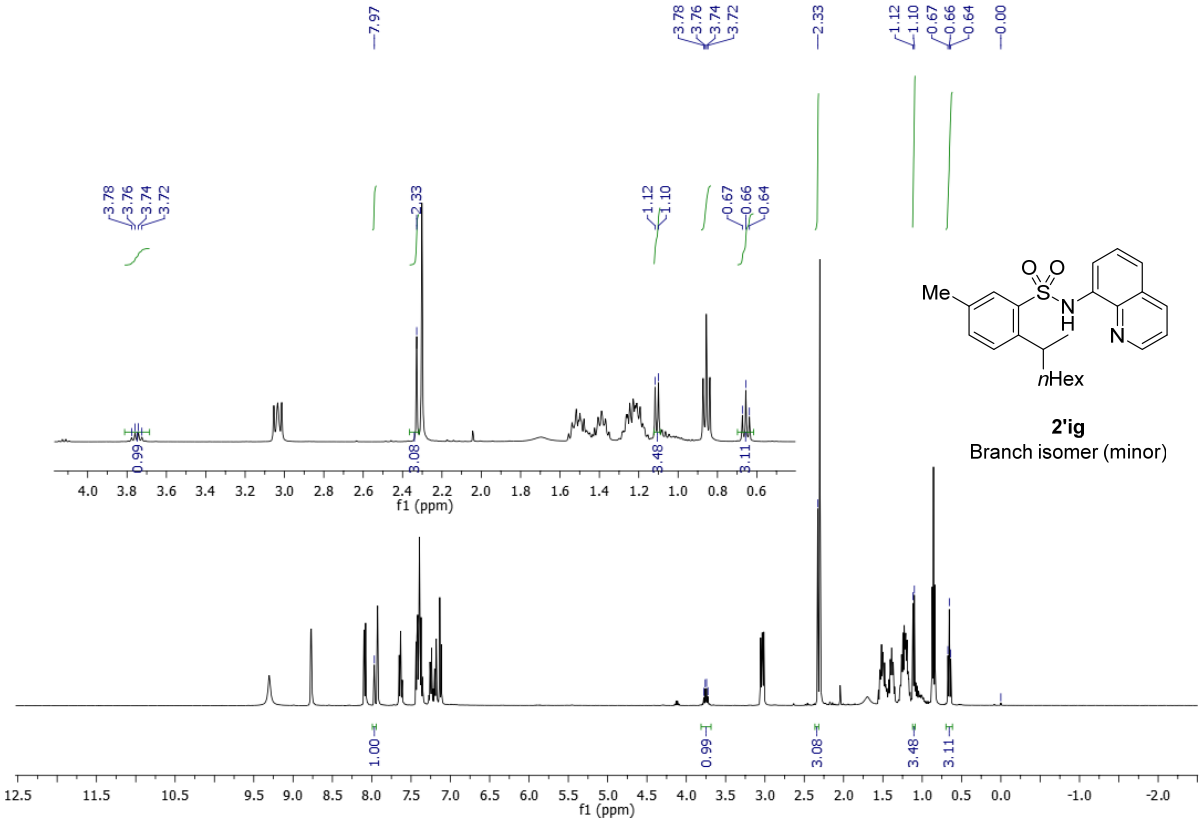
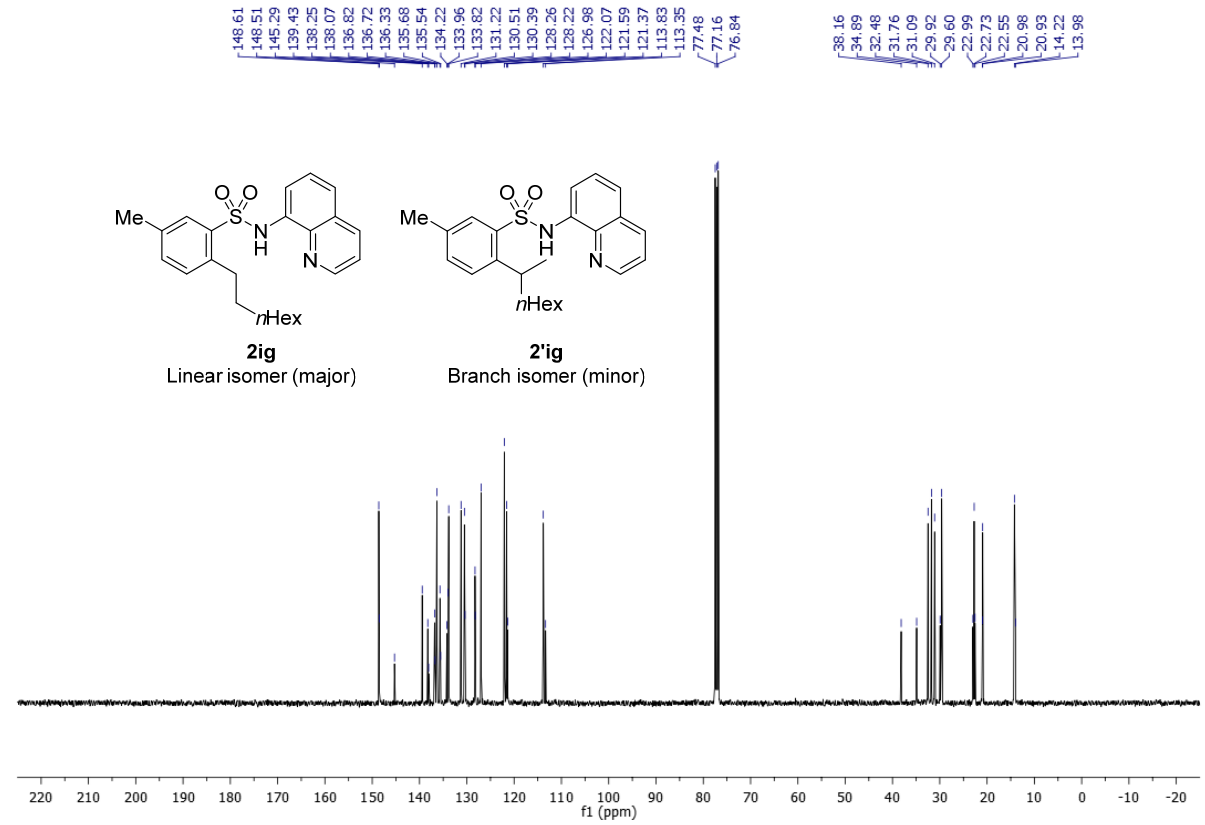
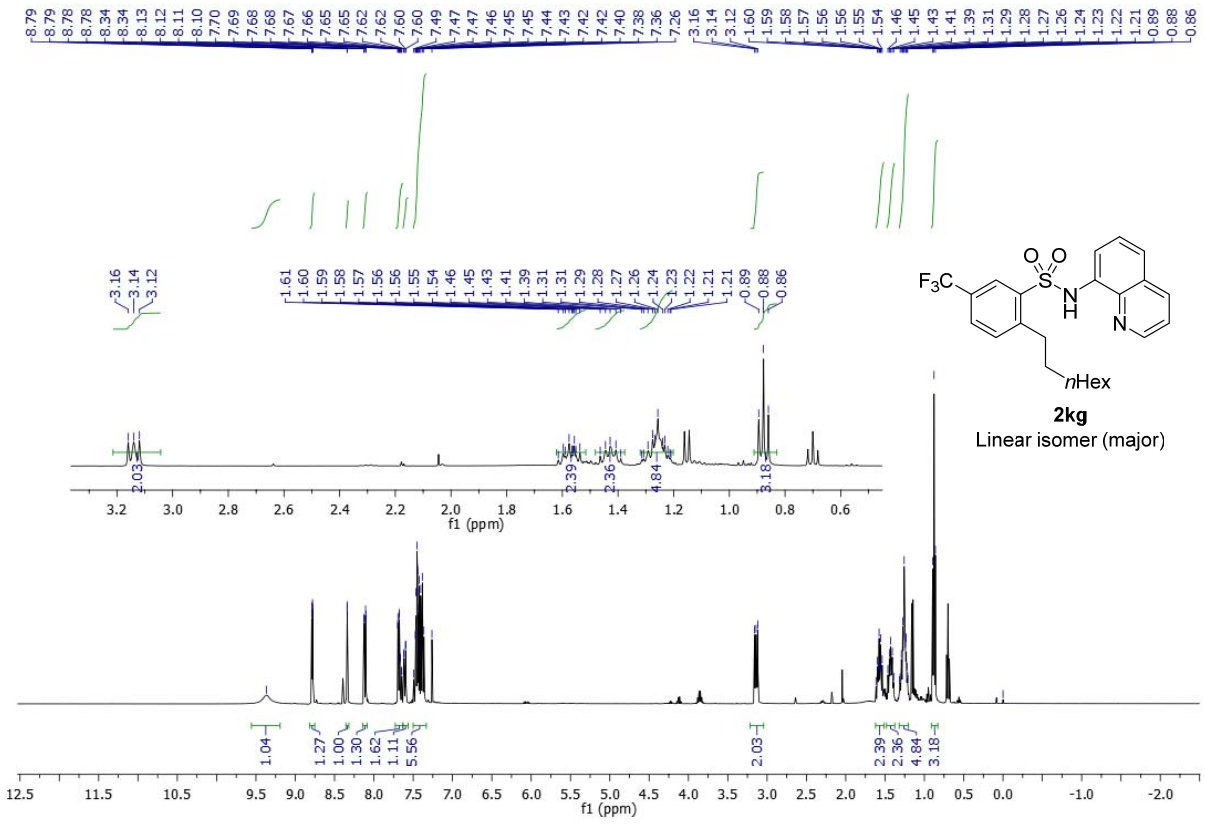
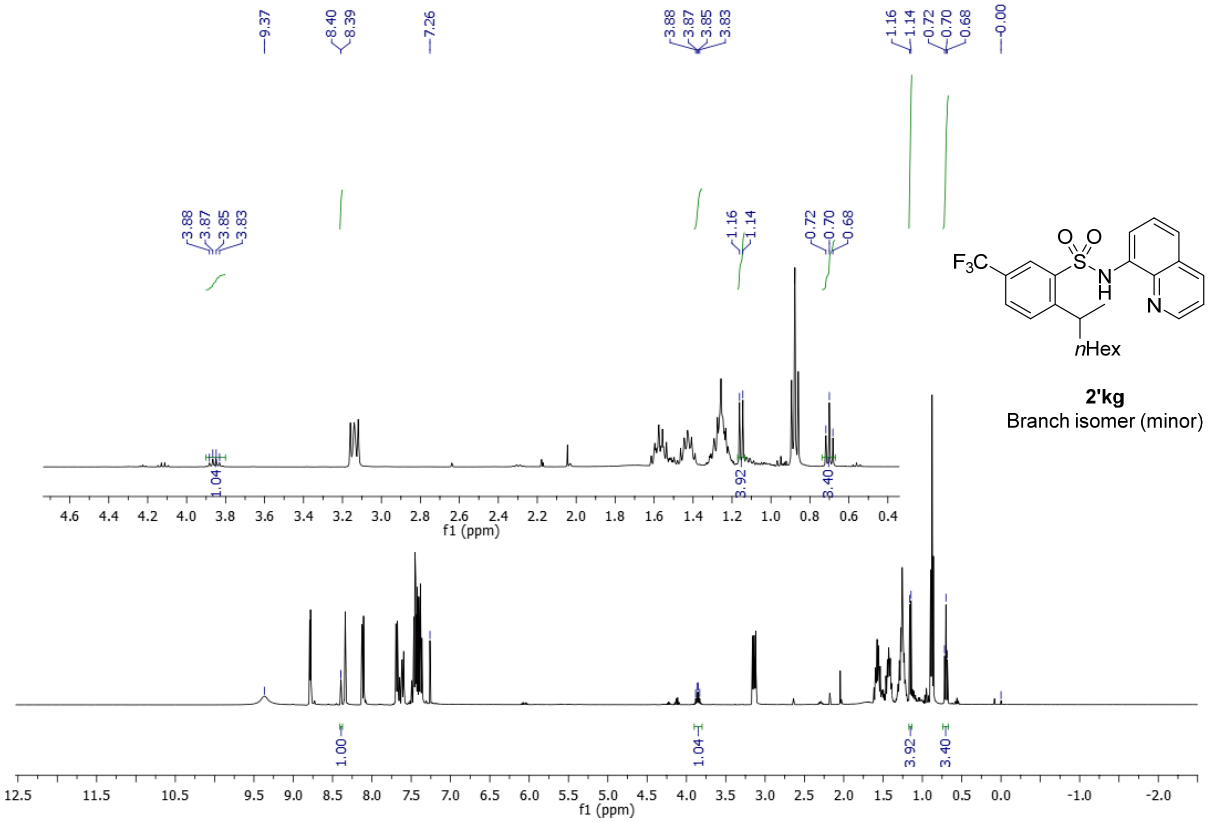
nHex
2ig
Linear isomer (major)
f1 (ppm)

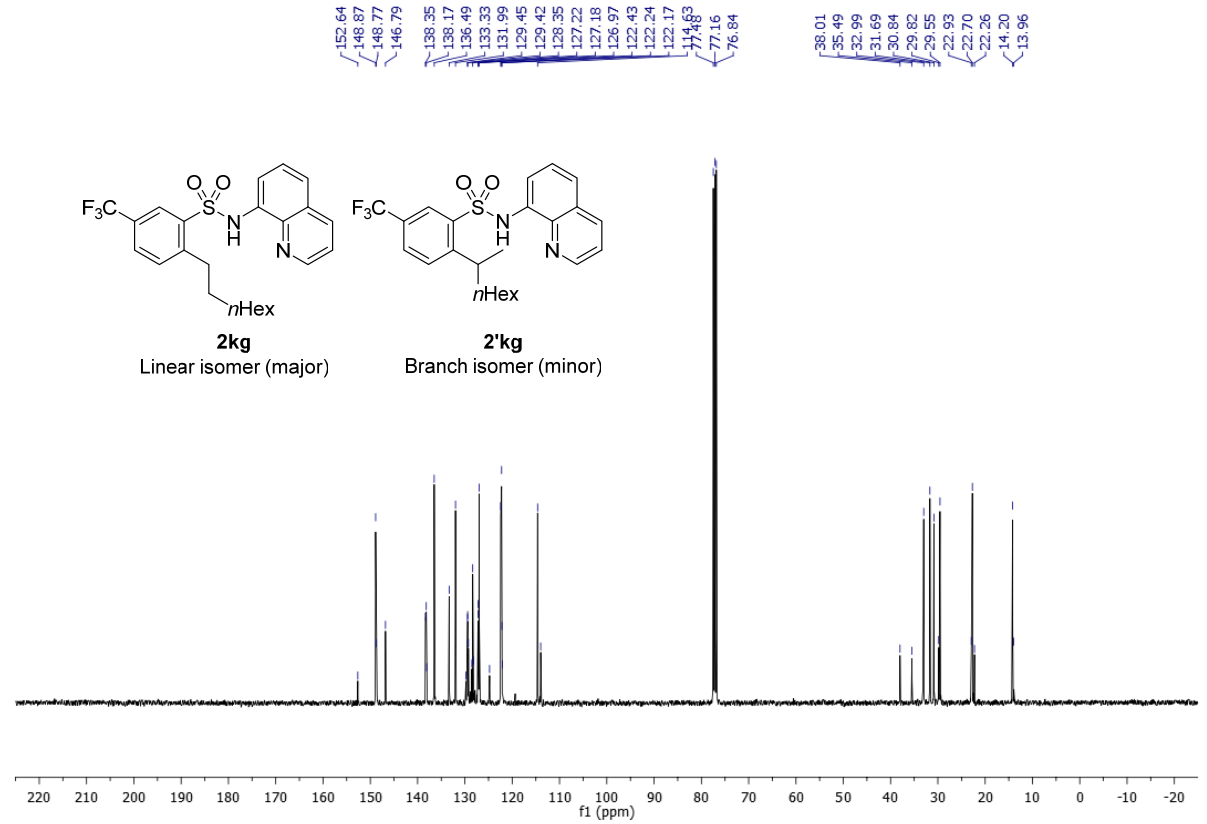

Me
O
O
S
N
H
N
nHex
2'ig
Branch isomer (minor)
f1 (ppm)


148.61
148.51
145.29
139.43
138.25
138.07
136.82
136.72
136.33
135.68
135.54
134.22
133.96
133.82
131.22
130.51
130.39
128.26
128.22
126.98
122.07
121.59
121.37
113.83
113.35
77.48
77.16
76.84
38.16
34.89
32.48
31.76
31.09
29.92
29.60
22.99
22.73
22.55
20.98
20.93
14.22
13.98
Me
O
O
S
N
H
N
nHex
2ig
Linear isomer (major)
2'ig
Branch isomer (minor)
f1 (ppm)


8.79
8.79
8.78
8.78
8.34
8.34
8.13
8.12
8.11
8.10
7.70
7.69
7.68
7.68
7.67
7.66
7.65
7.65
7.62
7.62
7.60
7.60
7.49
7.47
7.47
7.46
7.45
7.45
7.44
7.43
7.42
7.42
7.40
7.38
7.36
7.26
3.16
3.14
3.12
1.60
1.59
1.58
1.57
1.56
1.56
1.55
1.54
1.46
1.45
1.43
1.41
1.39
1.31
1.29
1.28
1.27
1.26
1.24
1.23
1.22
1.21
1.21
0.89
0.88
0.86
F3C
O
O
S
N
H
N
nHex
2kg
Linear isomer (major)
f1 (ppm)

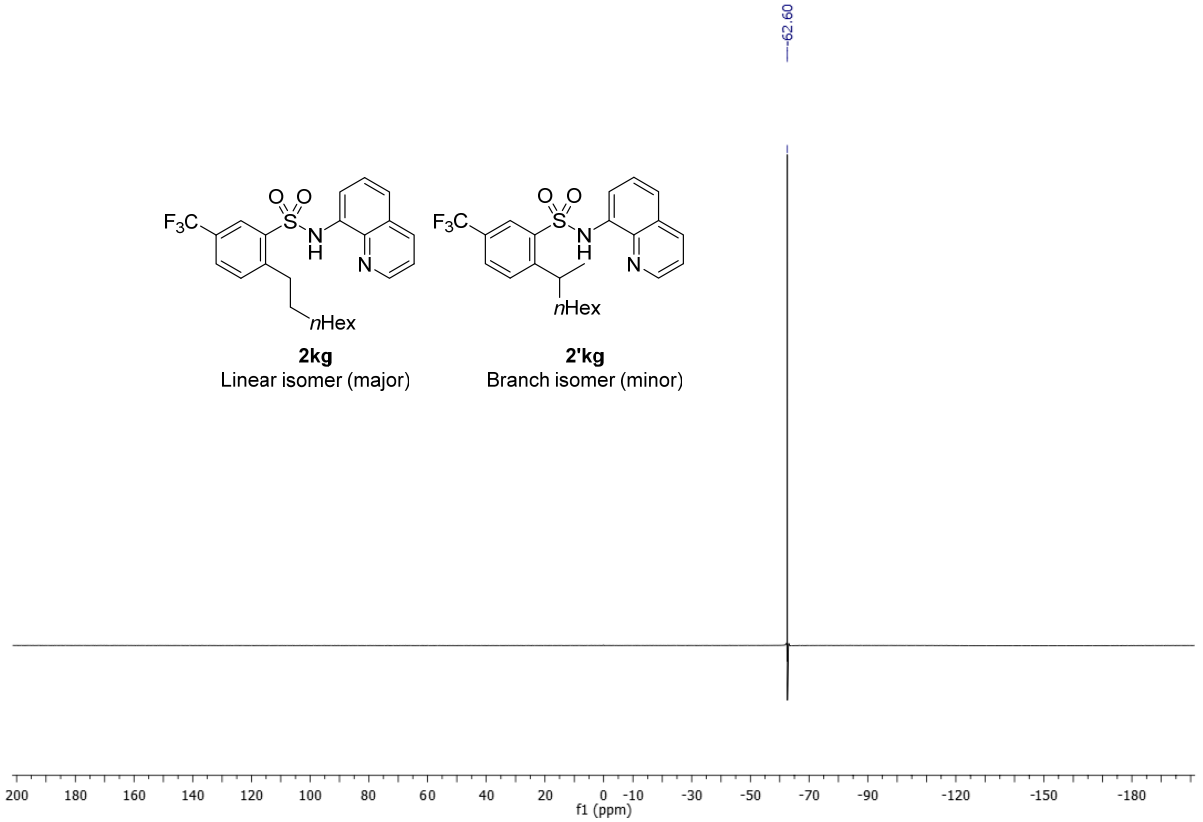


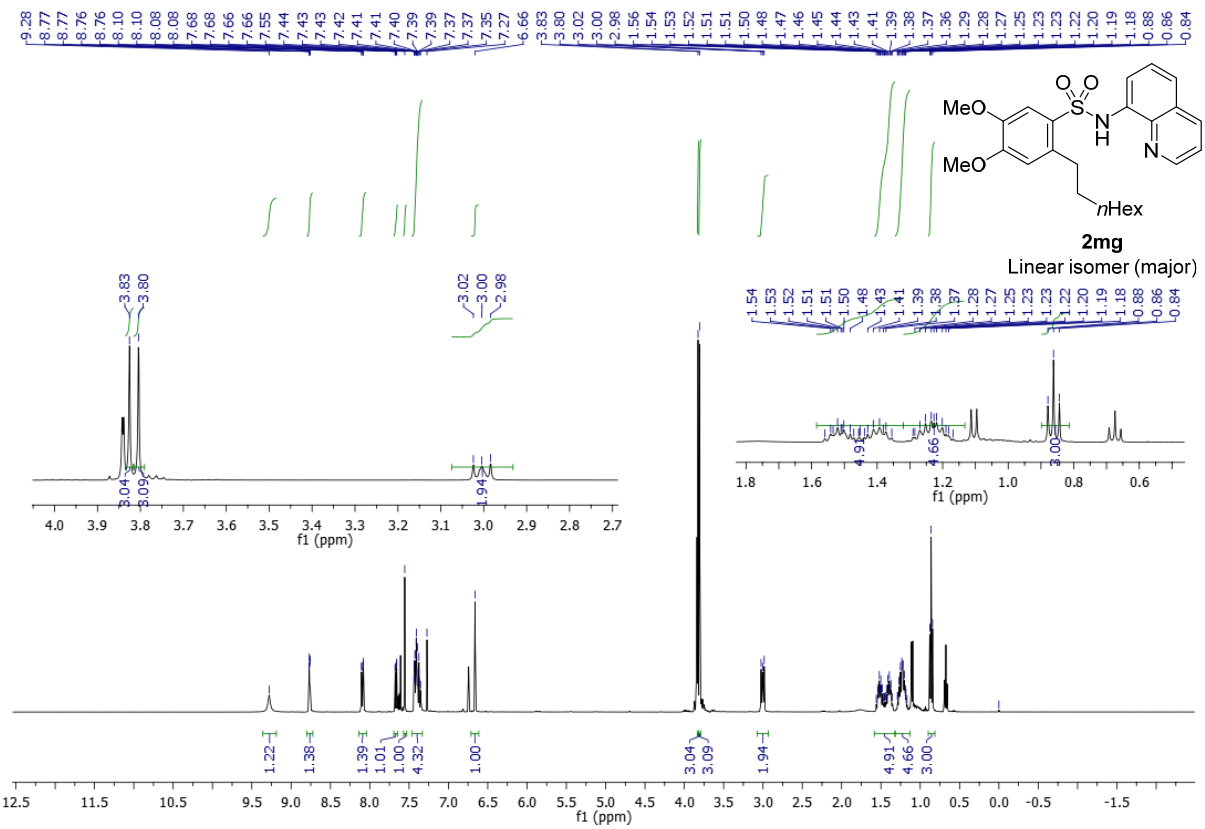
2'kg
Branch isomer (minor)

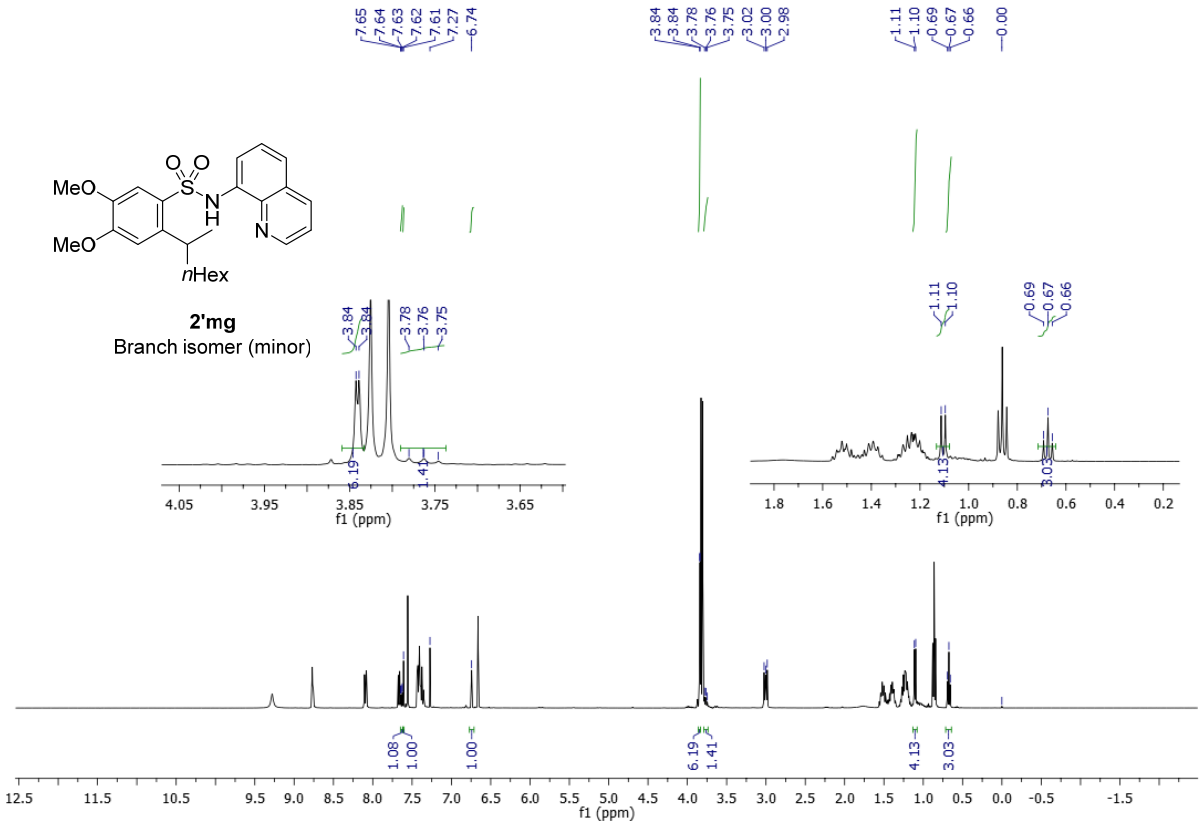


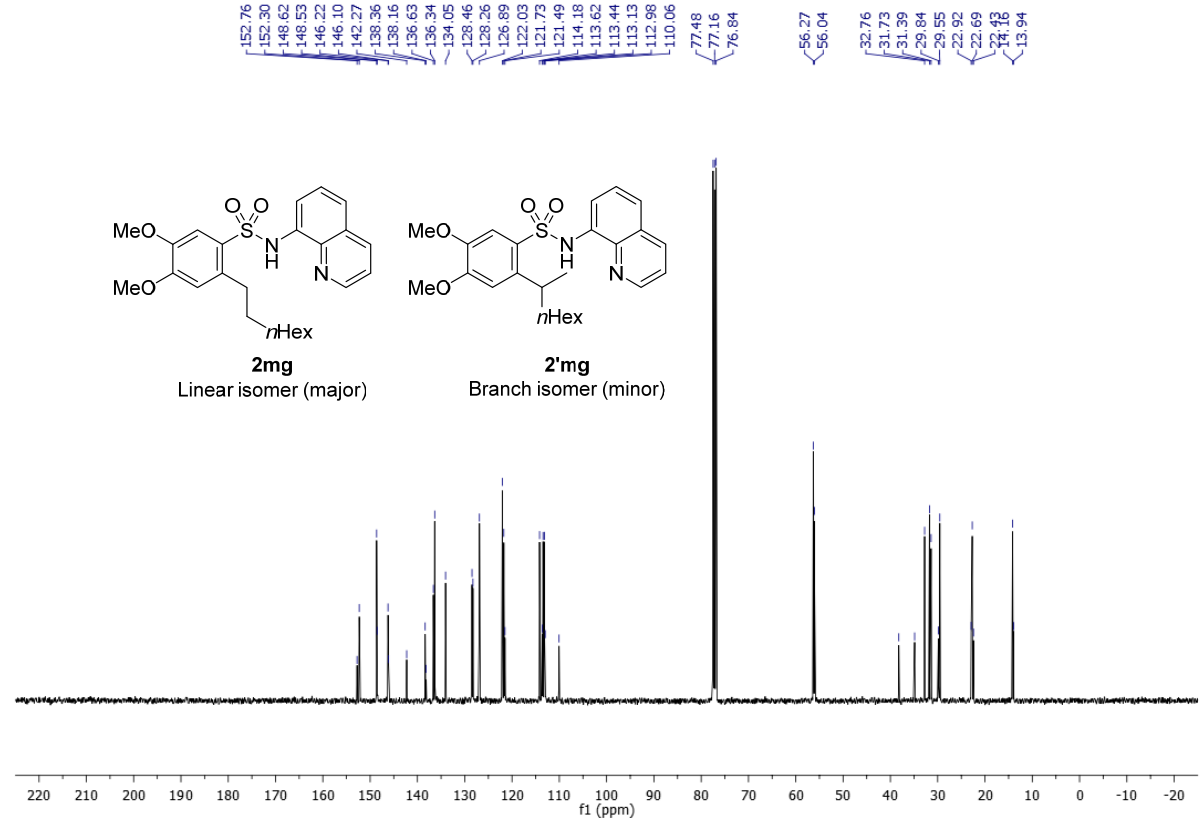
2kg
Linear isomer (major)

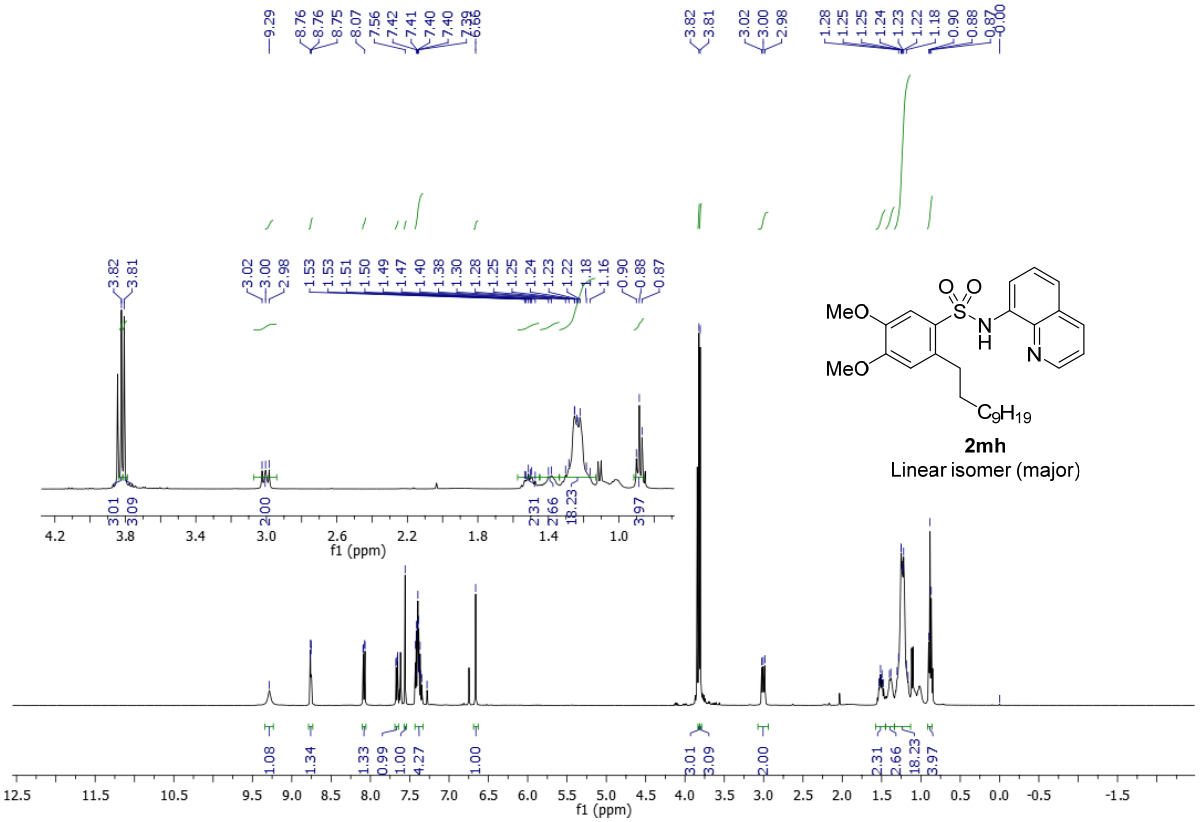
2'kg
Branch isomer (minor)

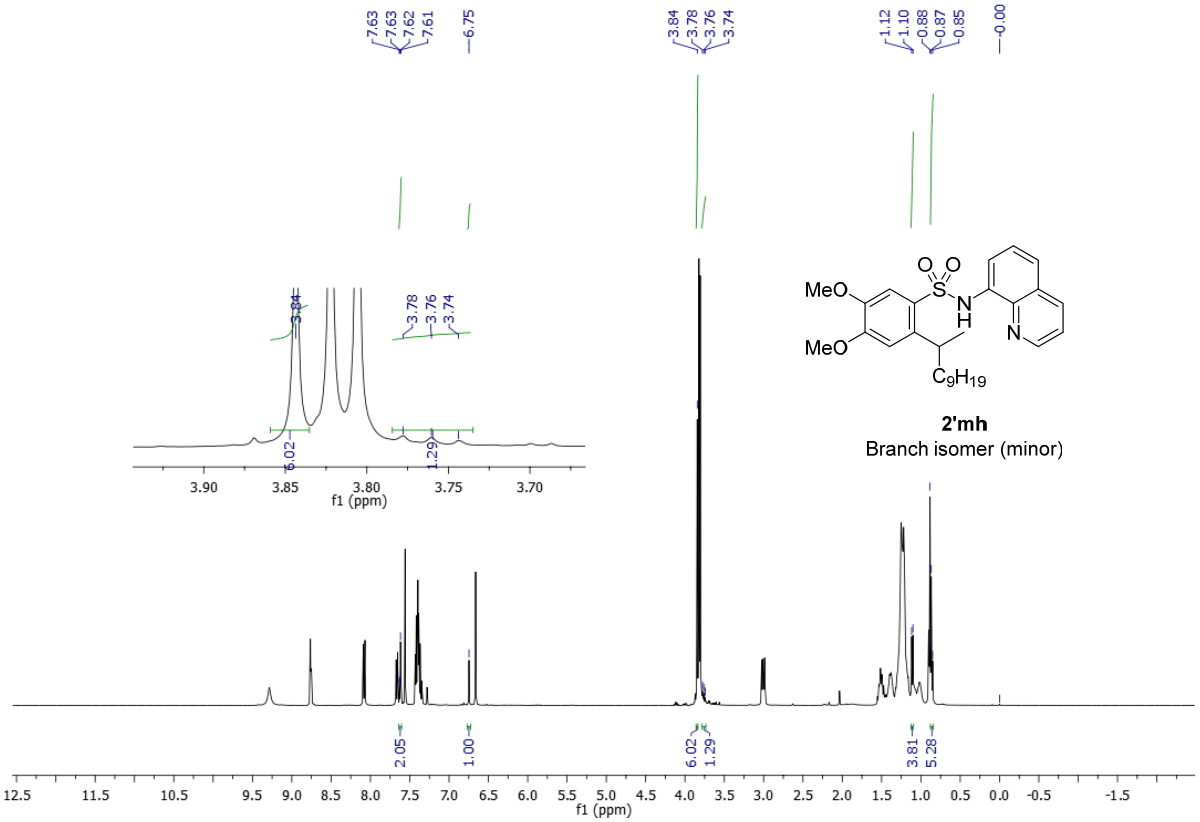

F3C
O
O
S
N
H
N
nHex
2kg
Linear isomer (major)
F3C
O
O
S
N
H
N
nHex
2'kg
Branch isomer (minor)
-62.60
f1 (ppm)

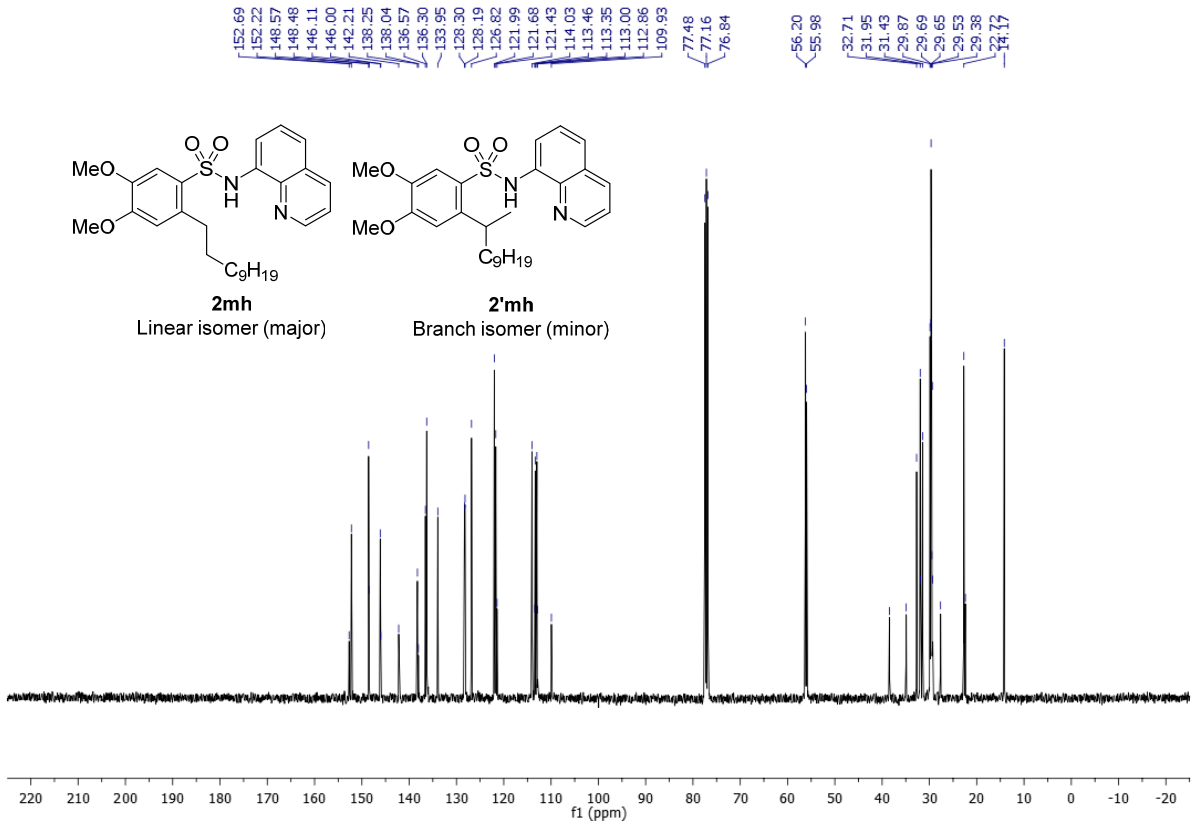

MeO
MeO
O
O
S
N
H
N
nHex
2mg
Linear isomer (major)
f1 (ppm)

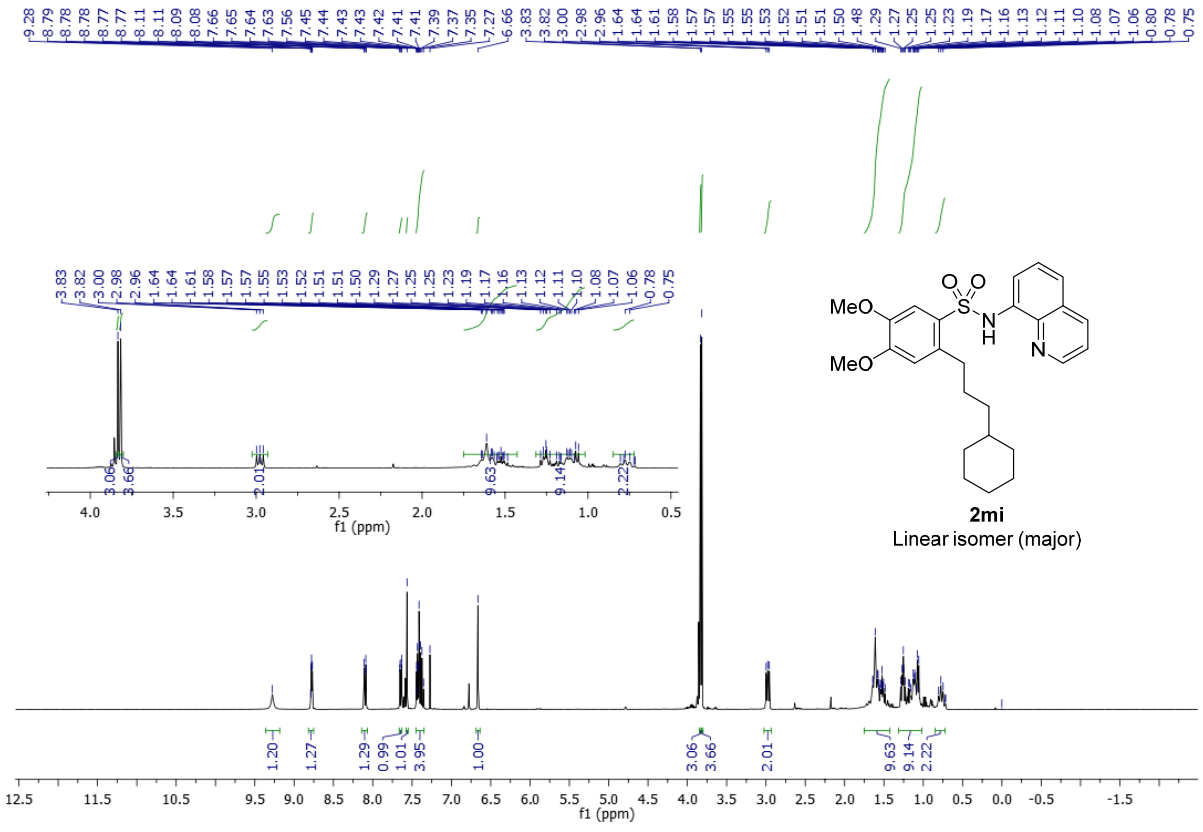

MeO
MeO
nHex
2'mg
Branch isomer (minor)
f1 (ppm)

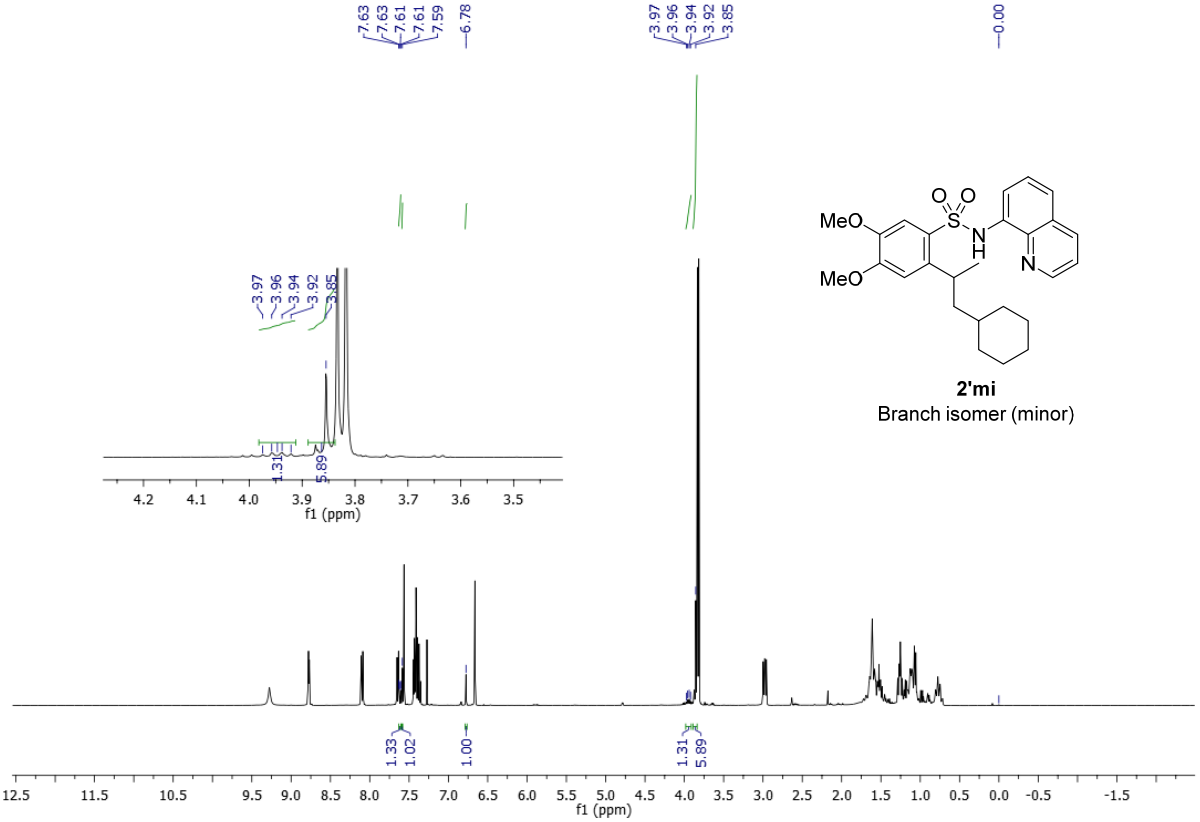

MeO
MeO
nHex
2mg
Linear isomer (major)
MeO
MeO
nHex
2'mg
Branch isomer (minor)
f1 (ppm)

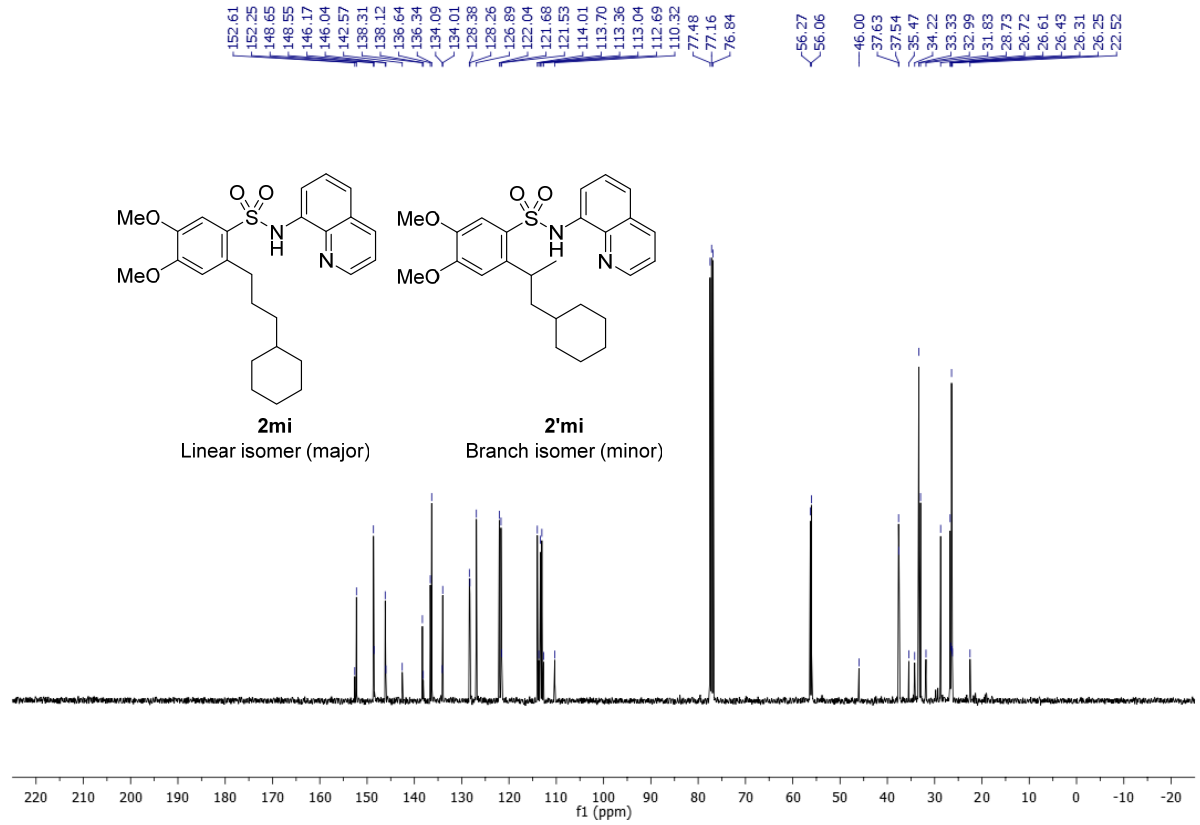

MeO
MeO
O
O
S
N
H
N
C9H19
2mh
Linear isomer (major)
f1 (ppm)

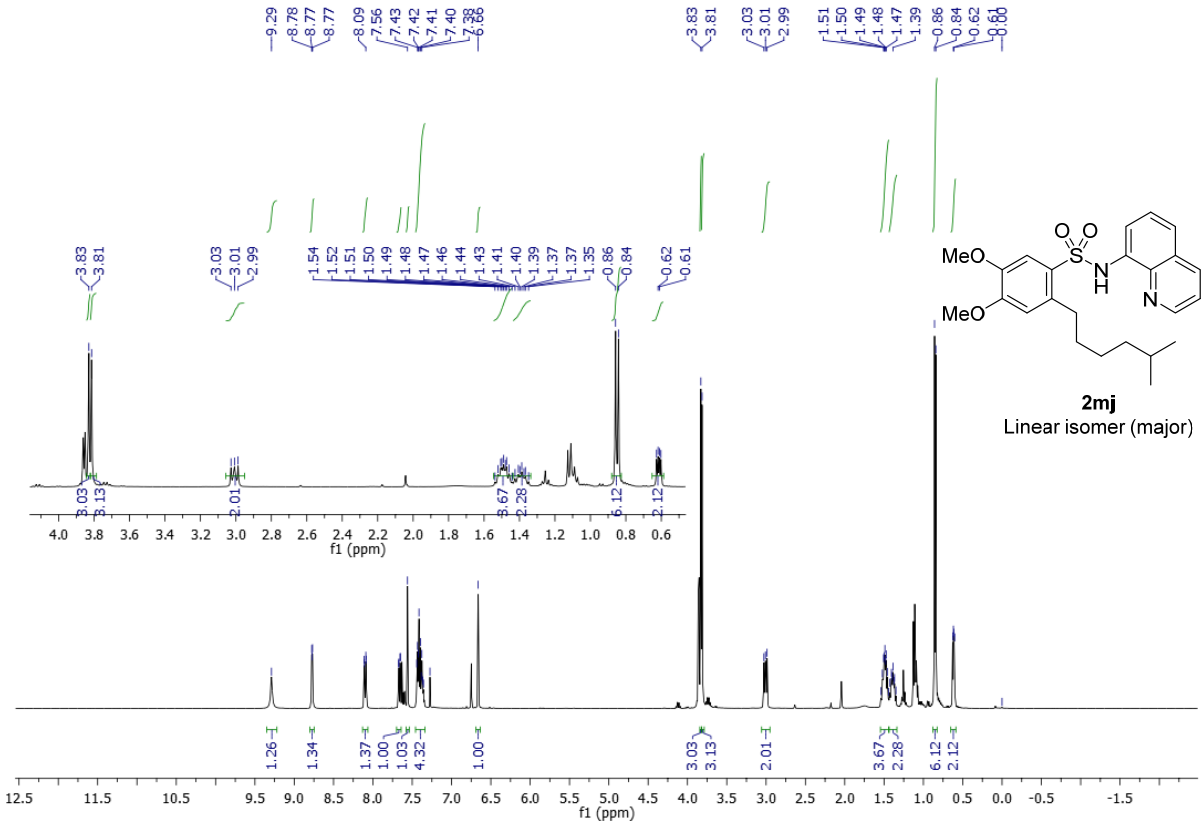

MeO
MeO
O
O
S
N
H
N
C9H19
2'mh
Branch isomer (minor)
f1 (ppm)

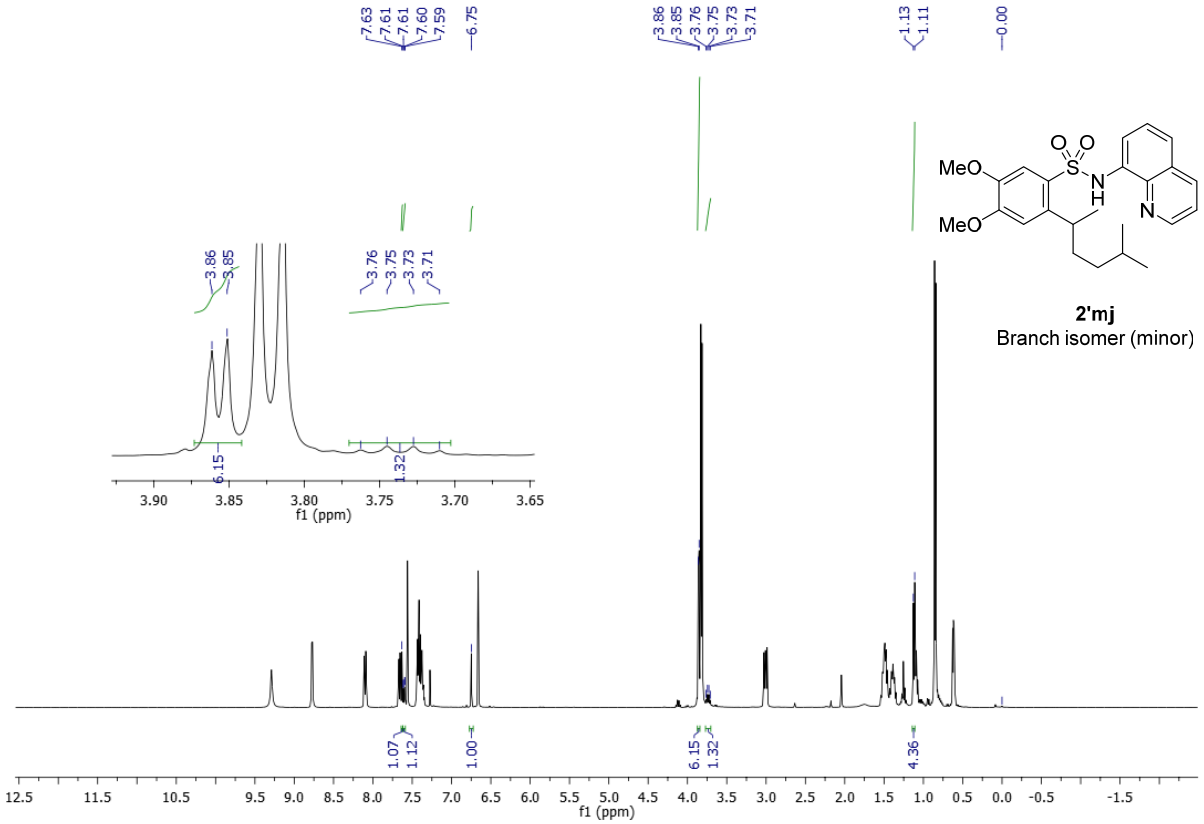

MeO
MeO
C_9H_{19}
2mh
Linear isomer (major)
2'mh
Branch isomer (minor)
f1 (ppm)

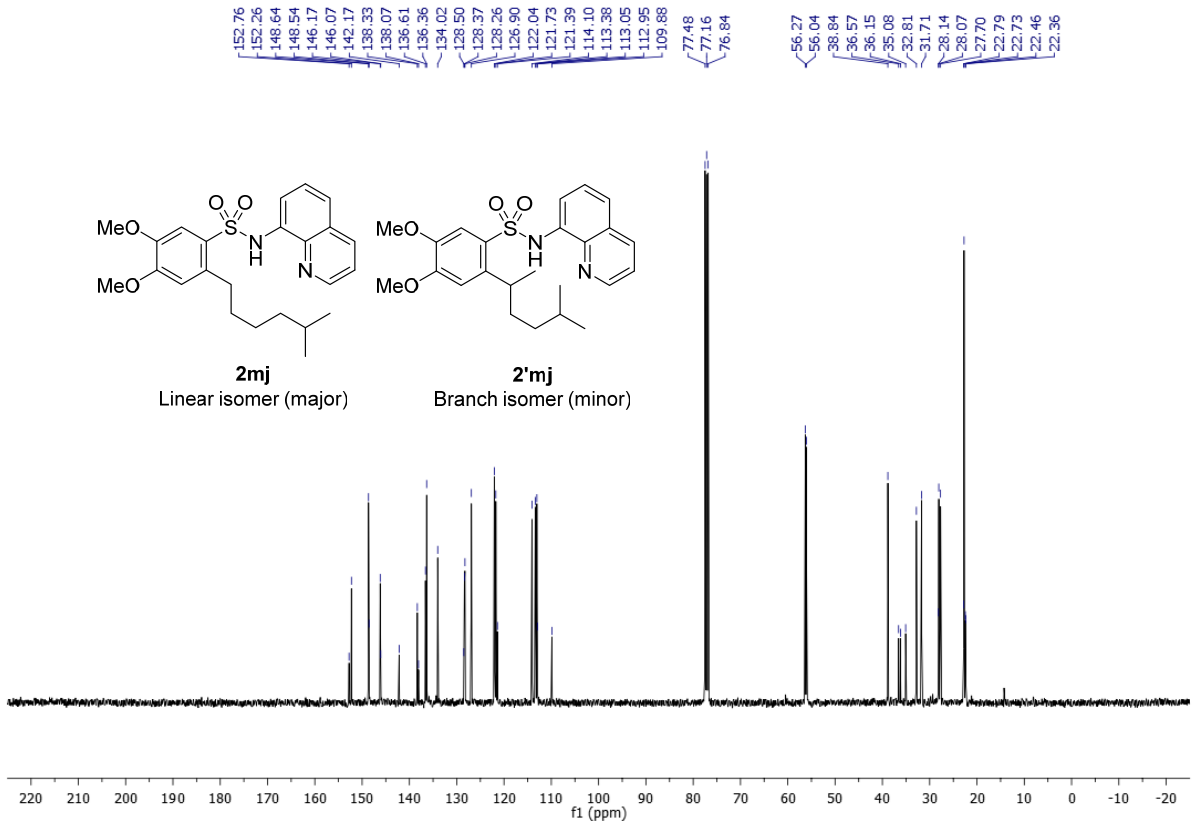

MeO
MeO
2mi
Linear isomer (major)
f1 (ppm)

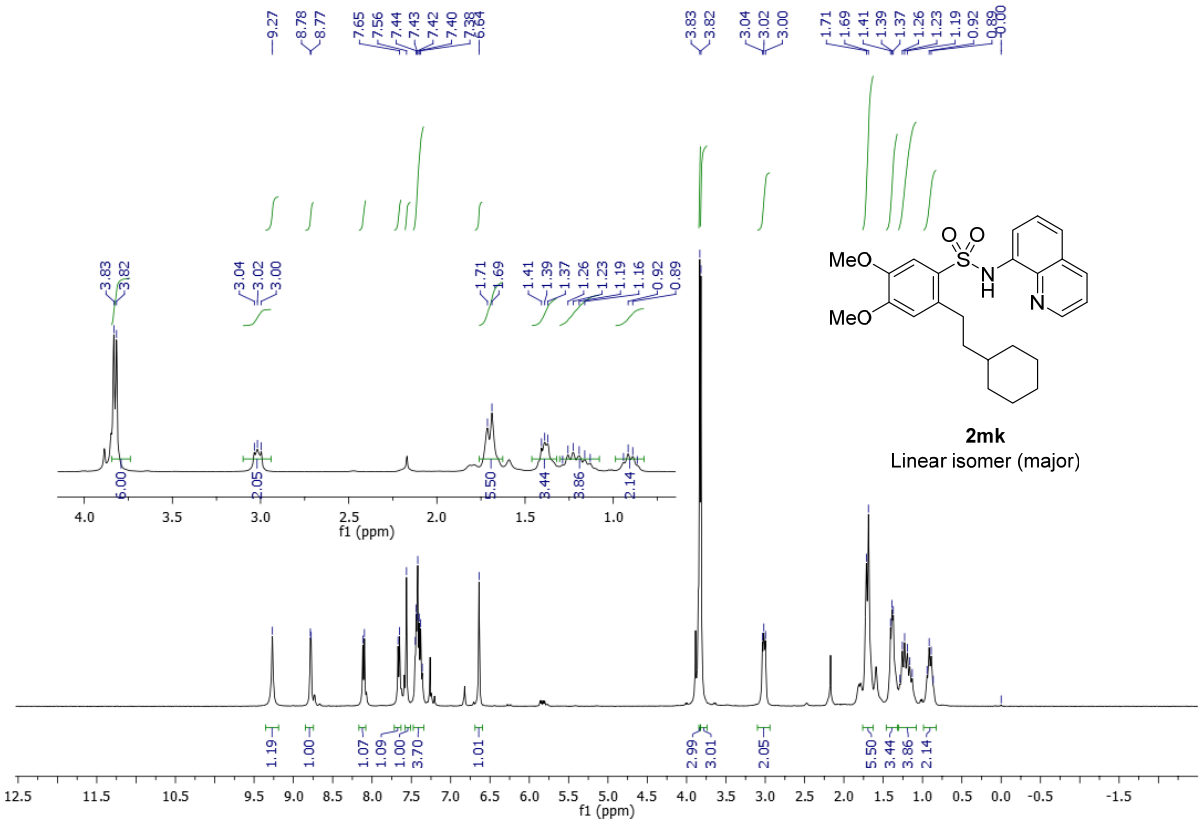

7.63
7.63
7.61
7.61
7.59
6.78
3.97
3.96
3.94
3.92
3.85
0.00
MeO
MeO
O
O
S
N
H
N
2'mi
Branch isomer (minor)
1.33
1.02
1.00
1.31
5.89
f1 (ppm)

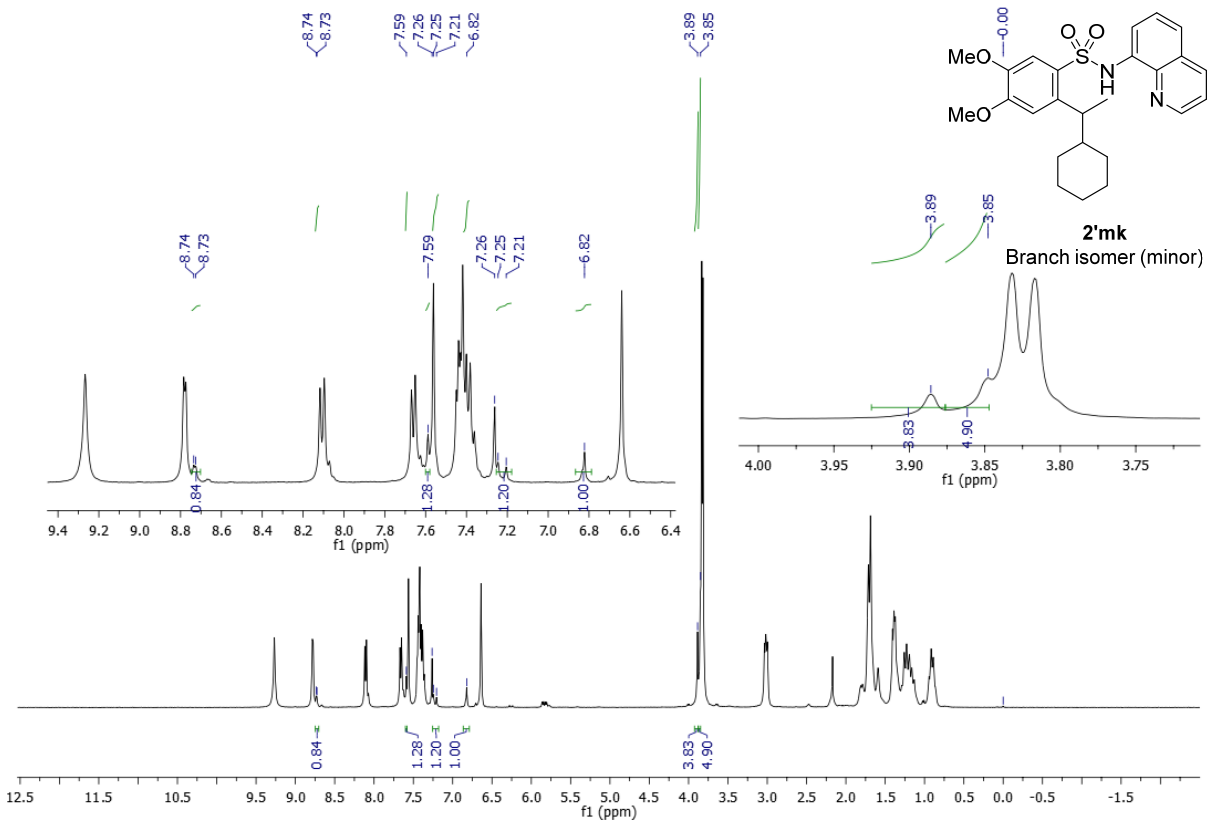

152.61
152.25
148.65
148.55
146.17
146.04
142.57
138.31
138.12
136.64
136.34
134.09
134.01
128.38
128.26
126.89
122.04
121.68
121.53
114.01
113.70
113.36
113.04
112.69
110.32
77.48
77.16
76.84
56.27
56.06
46.00
37.63
37.54
35.47
34.22
33.33
32.99
31.83
28.73
26.72
26.61
26.43
26.31
26.25
22.52
MeO
MeO
O
O
S
N
H
N
2mi
Linear isomer (major)
2'mi
Branch isomer (minor)
f1 (ppm)

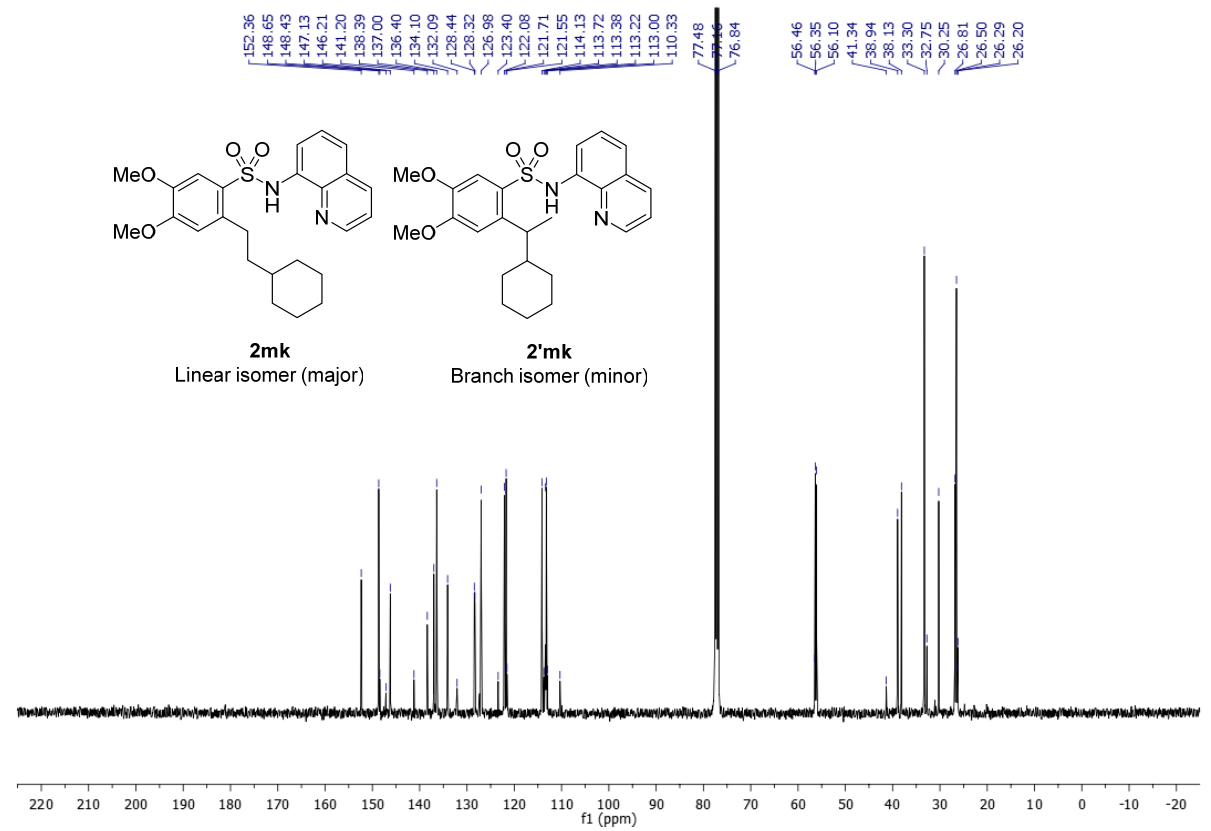

MeO
MeO
O
O
S
N
H
N
2mj
Linear isomer (major)
f1 (ppm)

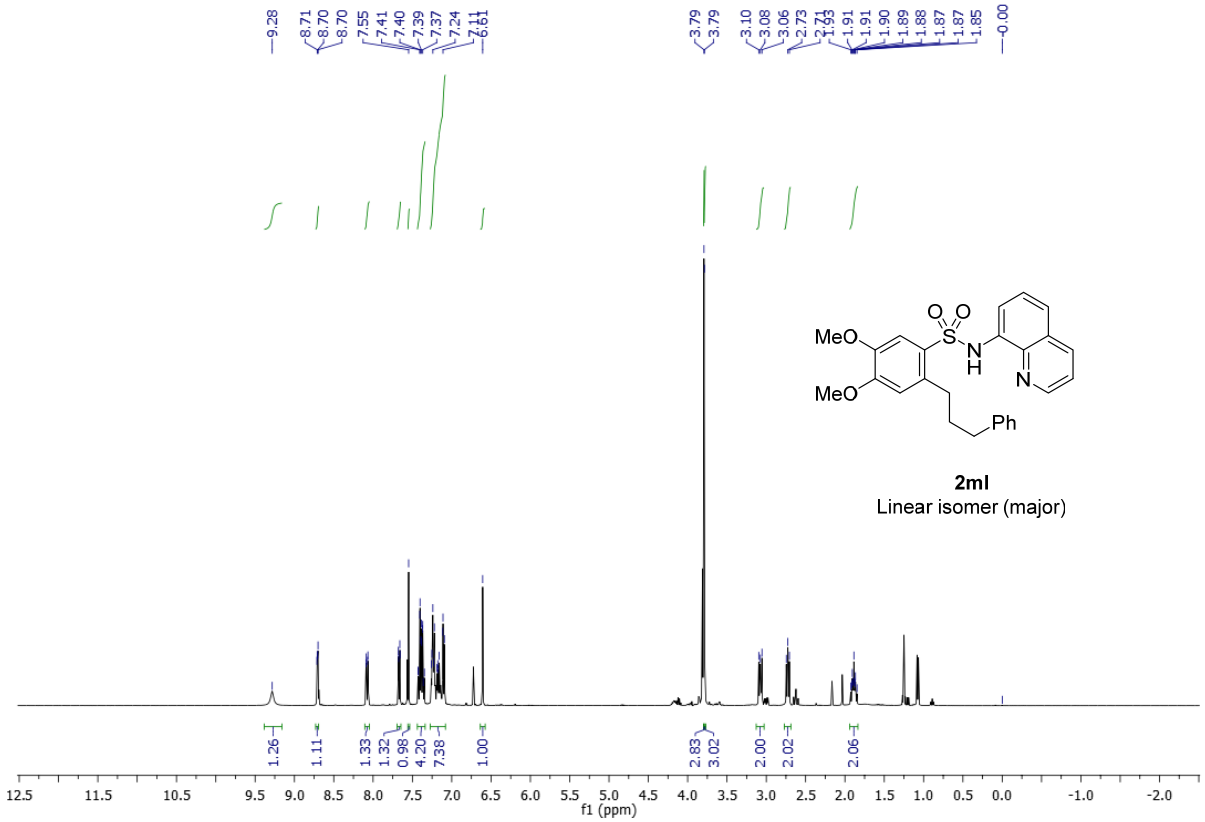

MeO
MeO
O
O
S
N
H
N
2'mj
Branch isomer (minor)
f1 (ppm)


MeO
MeO
O
O
S
N
H
N
2mj
Linear isomer (major)
2'mj
Branch isomer (minor)
f1 (ppm)

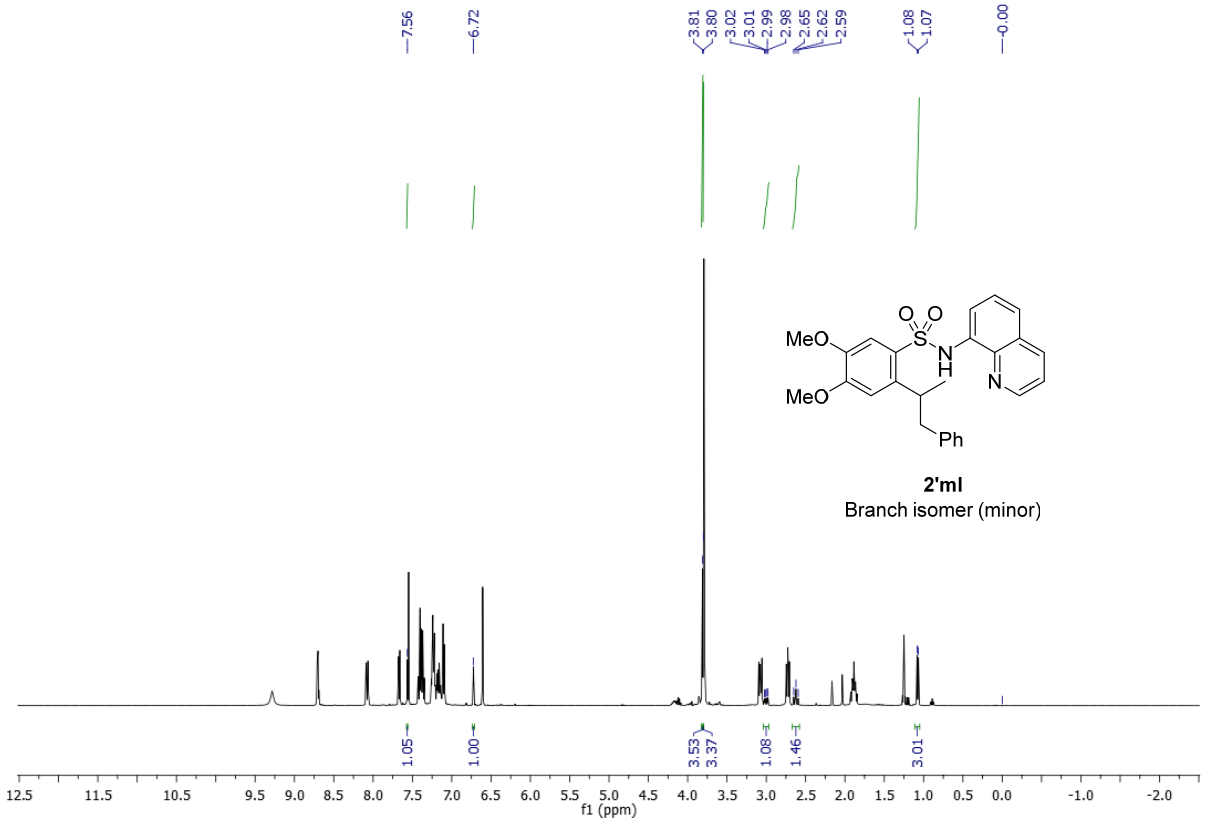

MeO
MeO
2mk
Linear isomer (major)
f1 (ppm)


2'mk
Branch isomer (minor)
f1 (ppm)

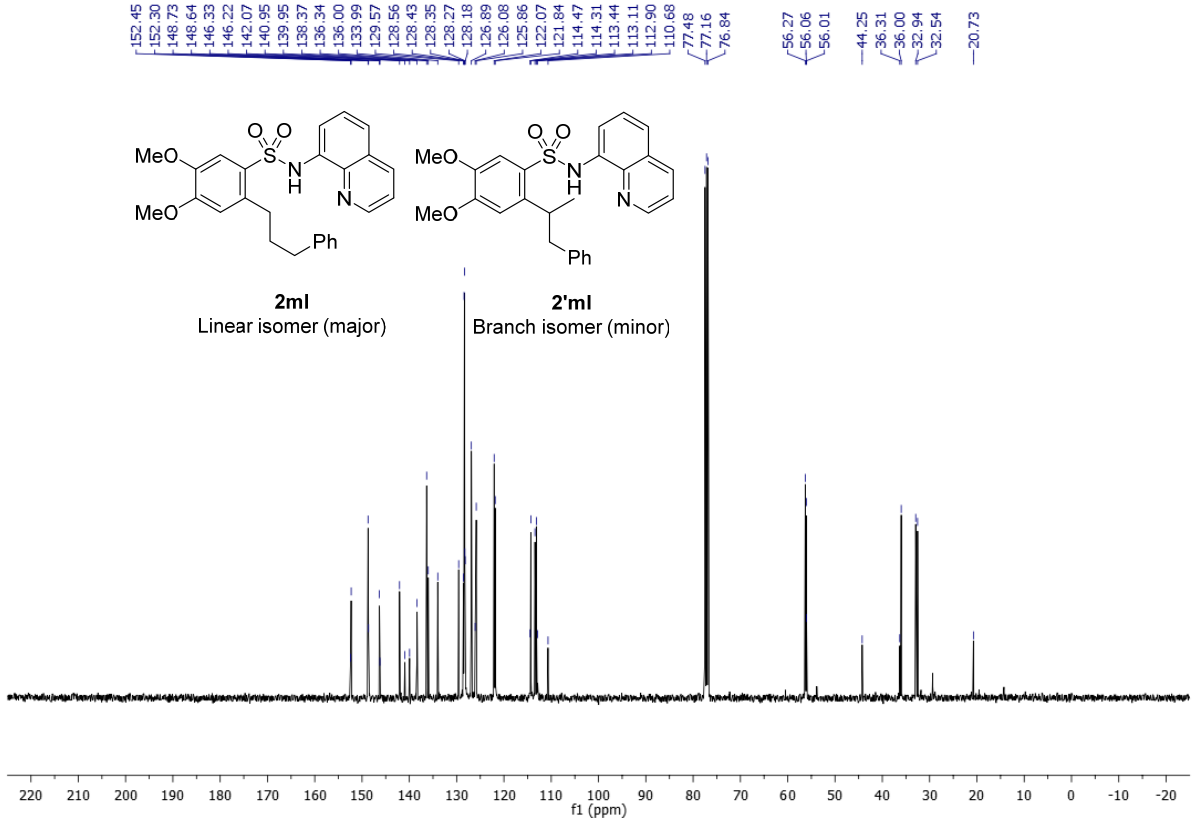

2mk
Linear isomer (major)
2'mk
Branch isomer (minor)
f1 (ppm)

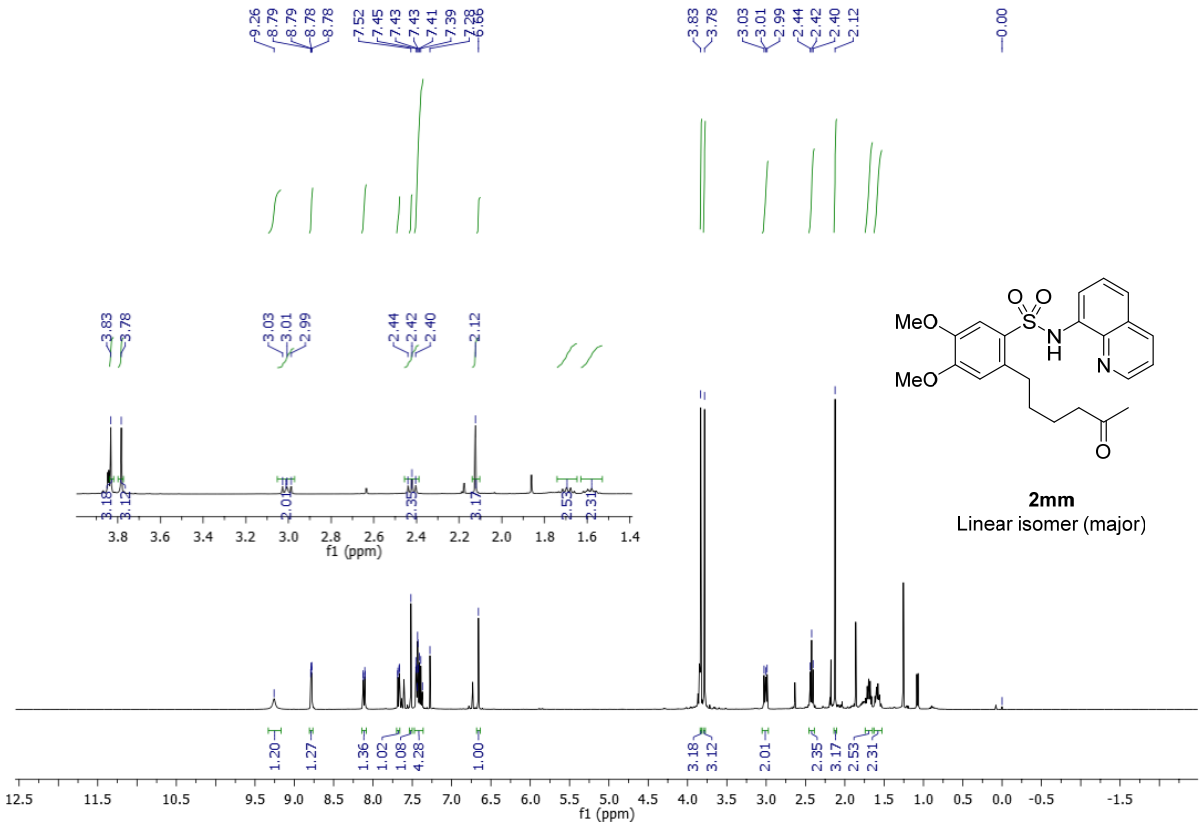


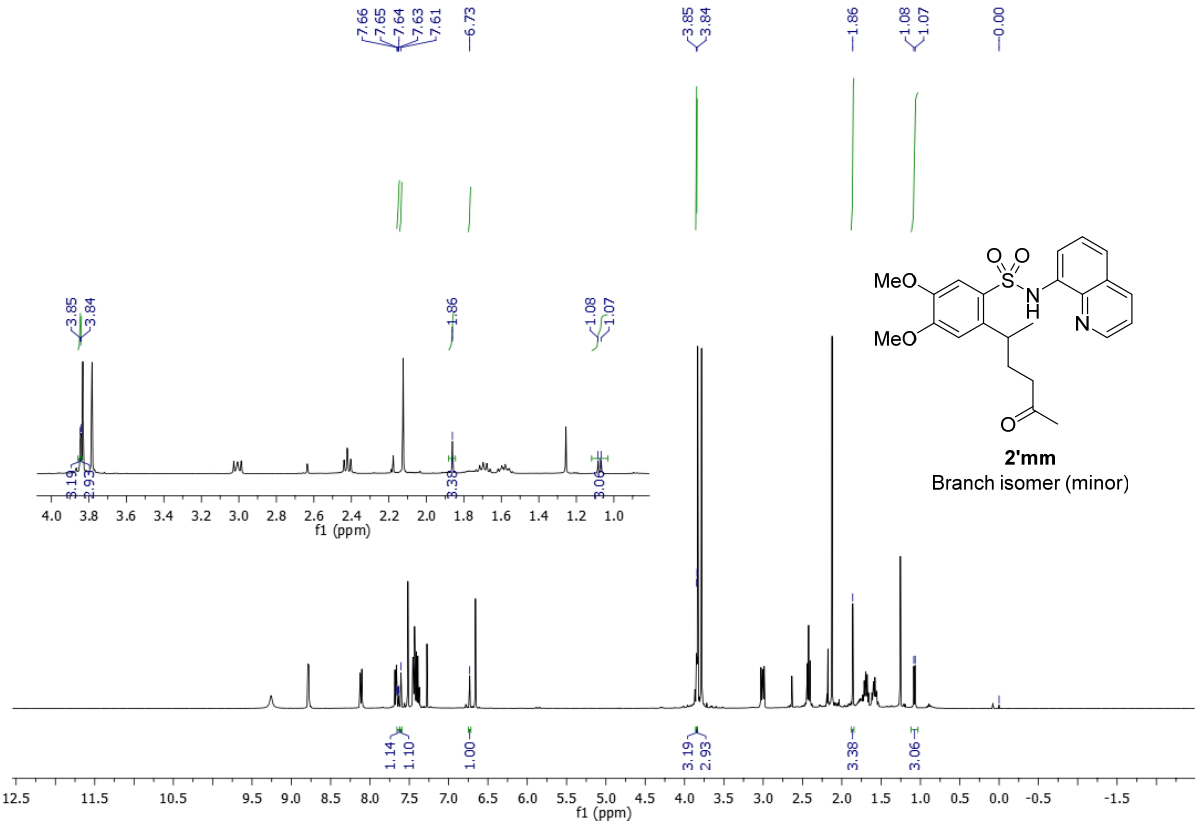
2ml
Linear isomer (major)

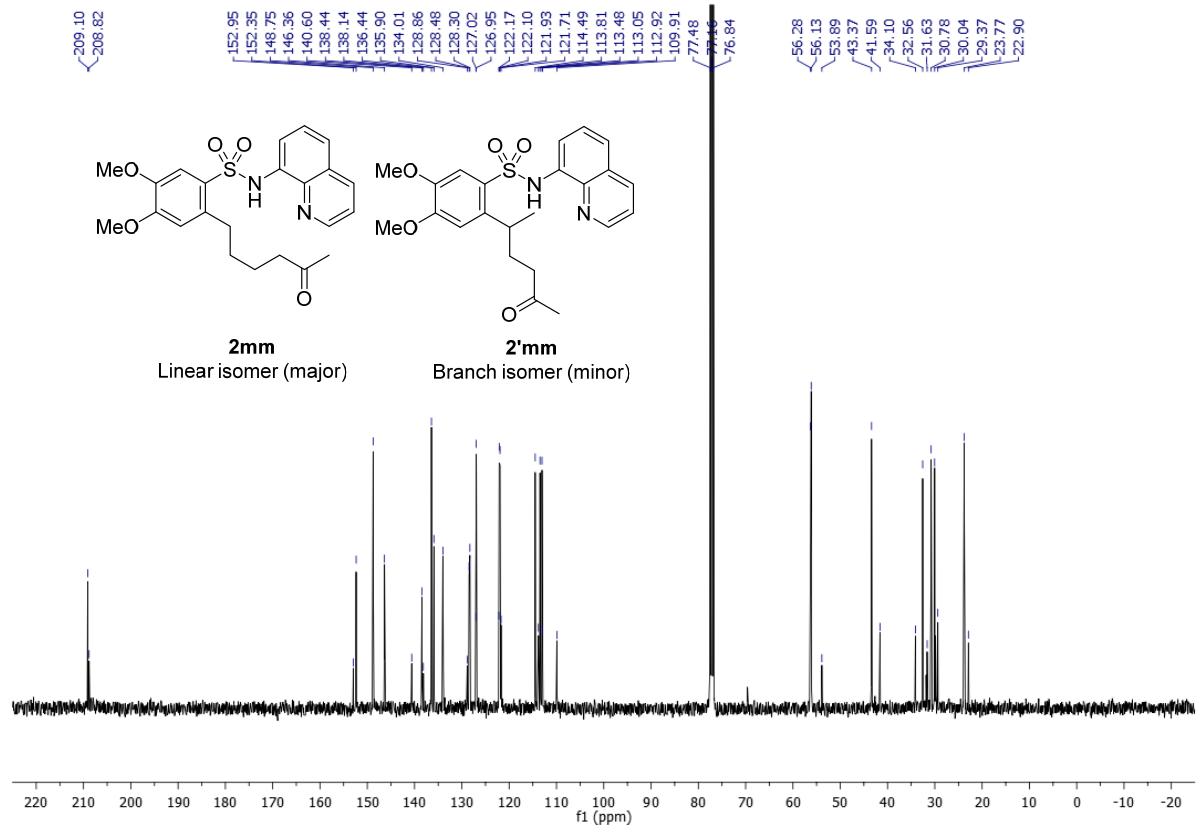


2'ml
Branch isomer (minor)

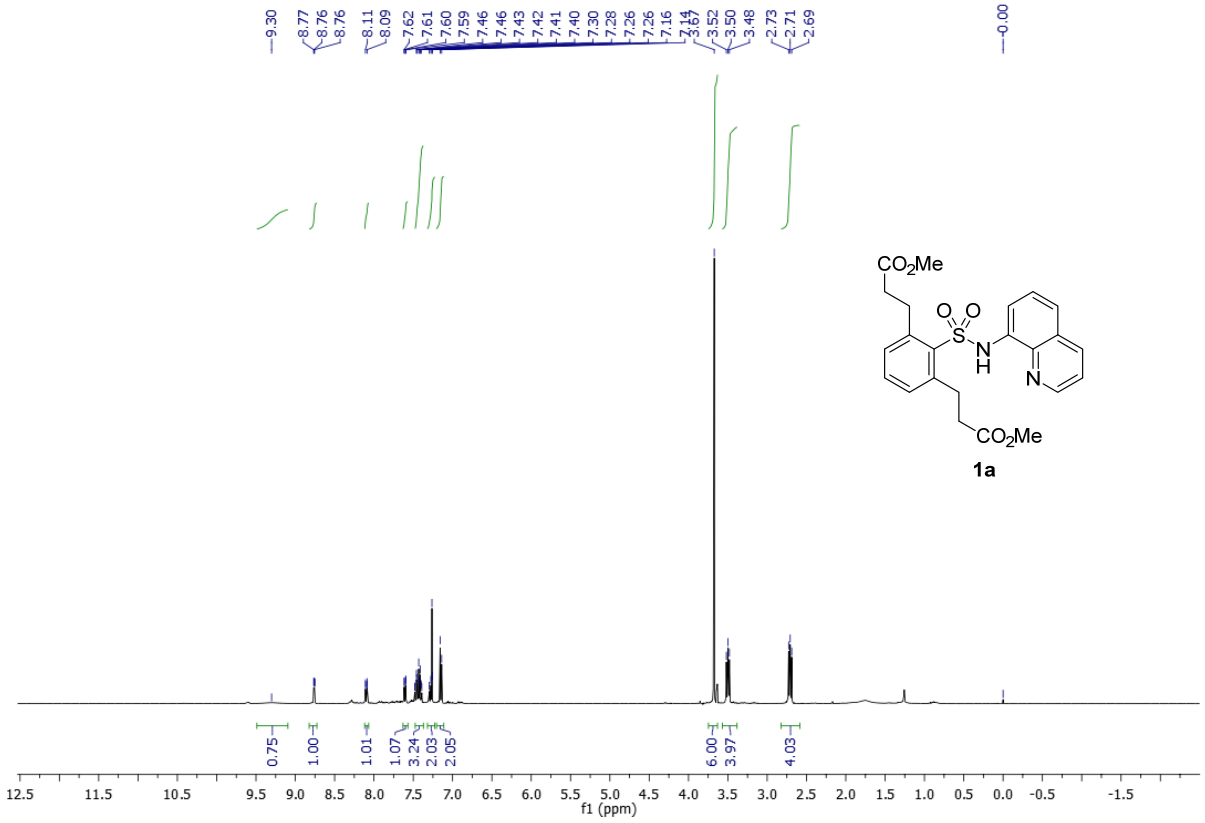

152.45
152.30
148.73
148.64
146.33
146.22
142.07
140.95
139.95
138.37
136.34
136.00
133.99
129.57
128.56
128.43
128.35
128.27
128.18
126.89
126.08
125.86
122.07
121.84
114.47
114.31
113.44
113.11
112.90
110.68
77.48
77.16
76.84
56.27
56.06
56.01
44.25
36.31
36.00
32.94
32.54
20.73
MeO
MeO
Ph
2ml
Linear isomer (major)
2'ml
Branch isomer (minor)
f1 (ppm)

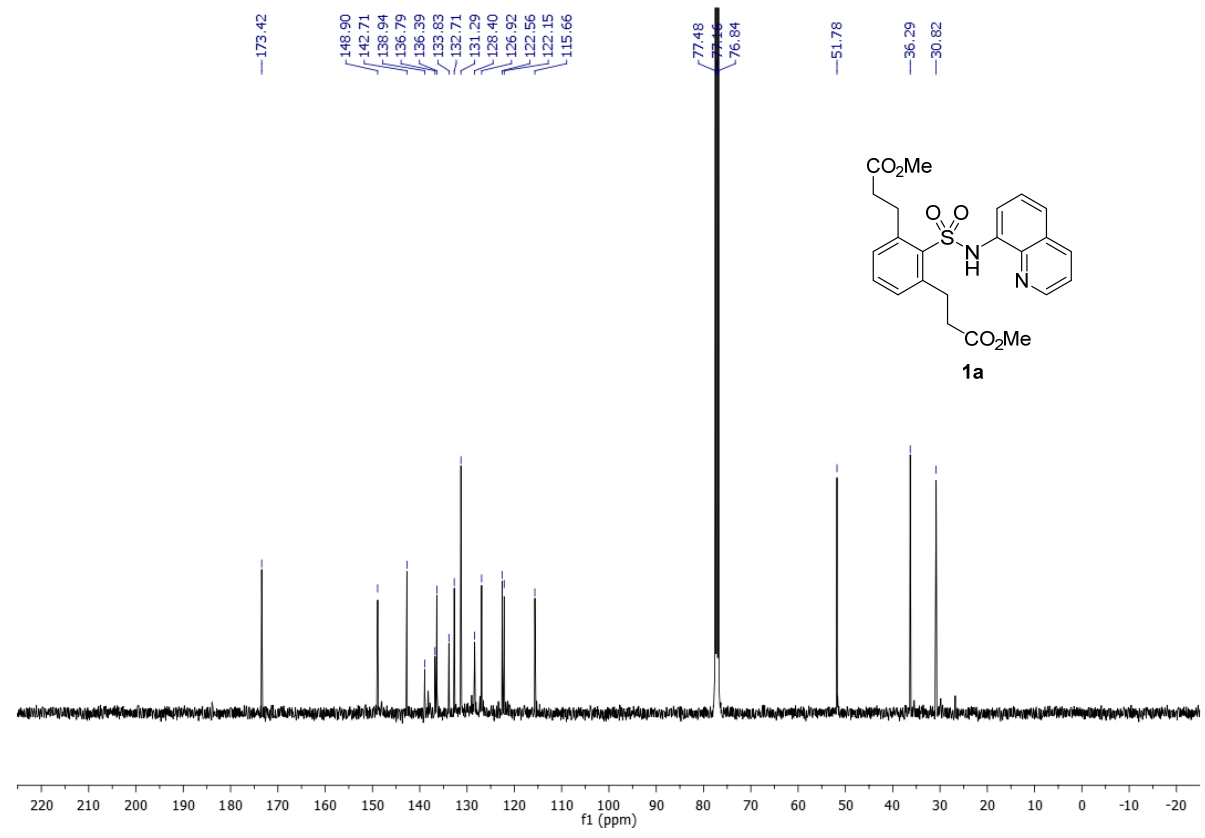

9.26
8.79
8.79
8.78
8.78
7.52
7.45
7.43
7.43
7.41
7.39
7.28
6.66
3.83
3.78
3.03
3.01
2.99
2.44
2.42
2.40
2.12
0.00
MeO
MeO
2mm
Linear isomer (major)
f1 (ppm)

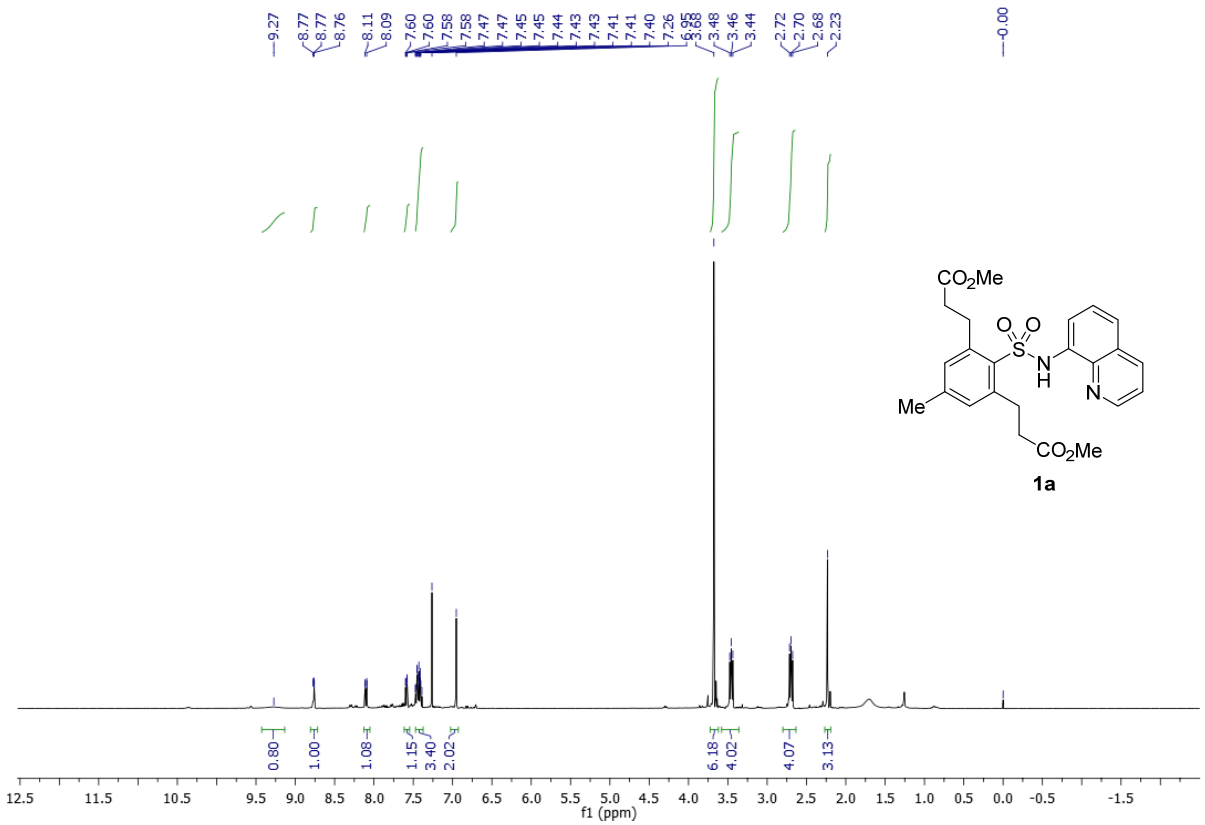

7.66
7.65
7.64
7.63
7.61
6.73
3.85
3.84
1.86
1.08
1.07
0.00
MeO
MeO
O
S
N
H
N
O
O
2'mm
Branch isomer (minor)
f1 (ppm)

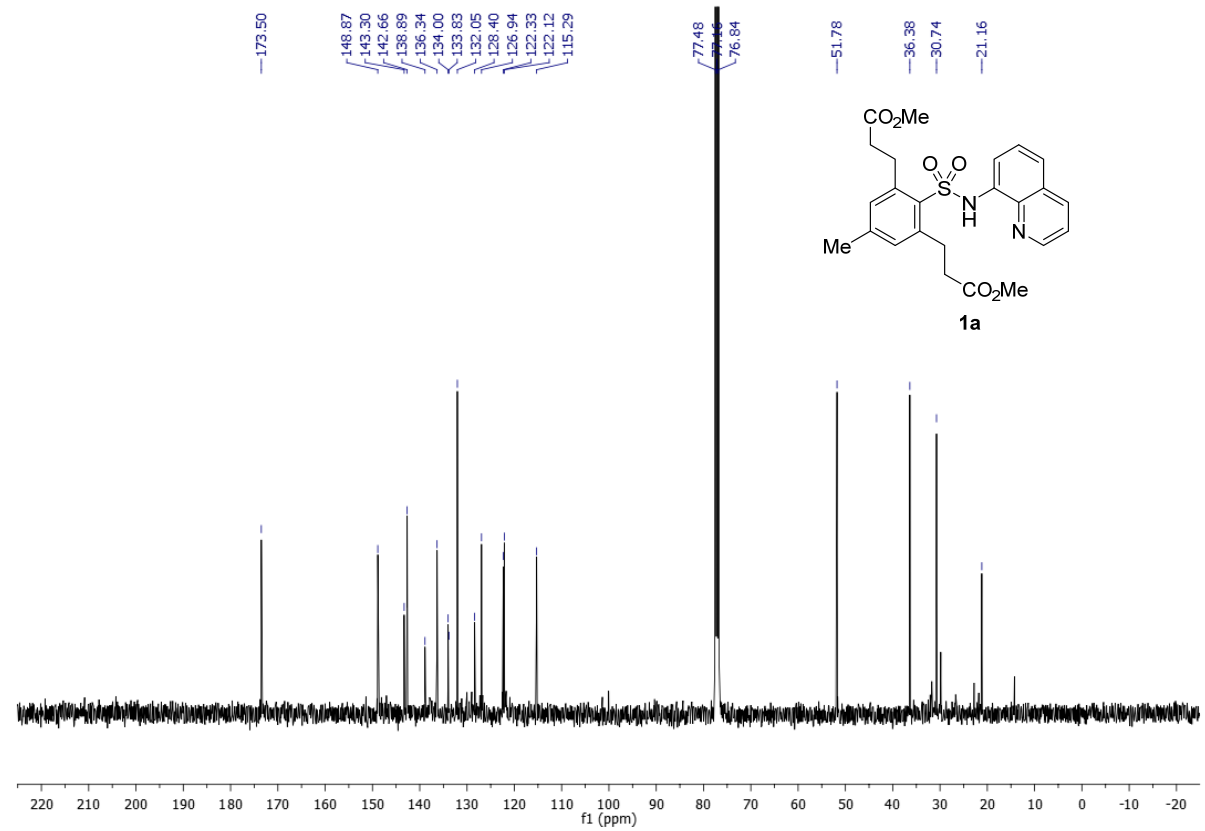

209.10
208.82
152.95
152.35
148.75
146.36
140.60
138.44
138.14
136.44
135.90
134.01
128.86
128.48
128.30
127.02
126.95
122.17
122.10
121.93
121.71
114.49
113.81
113.48
113.05
112.92
109.91
77.48
77.16
76.84
56.28
56.13
53.89
43.37
41.59
34.10
32.56
31.63
30.78
30.04
29.37
23.77
22.90
MeO
MeO
O
S
N
H
N
O
2mm
Linear isomer (major)
2'mm
Branch isomer (minor)
f1 (ppm)

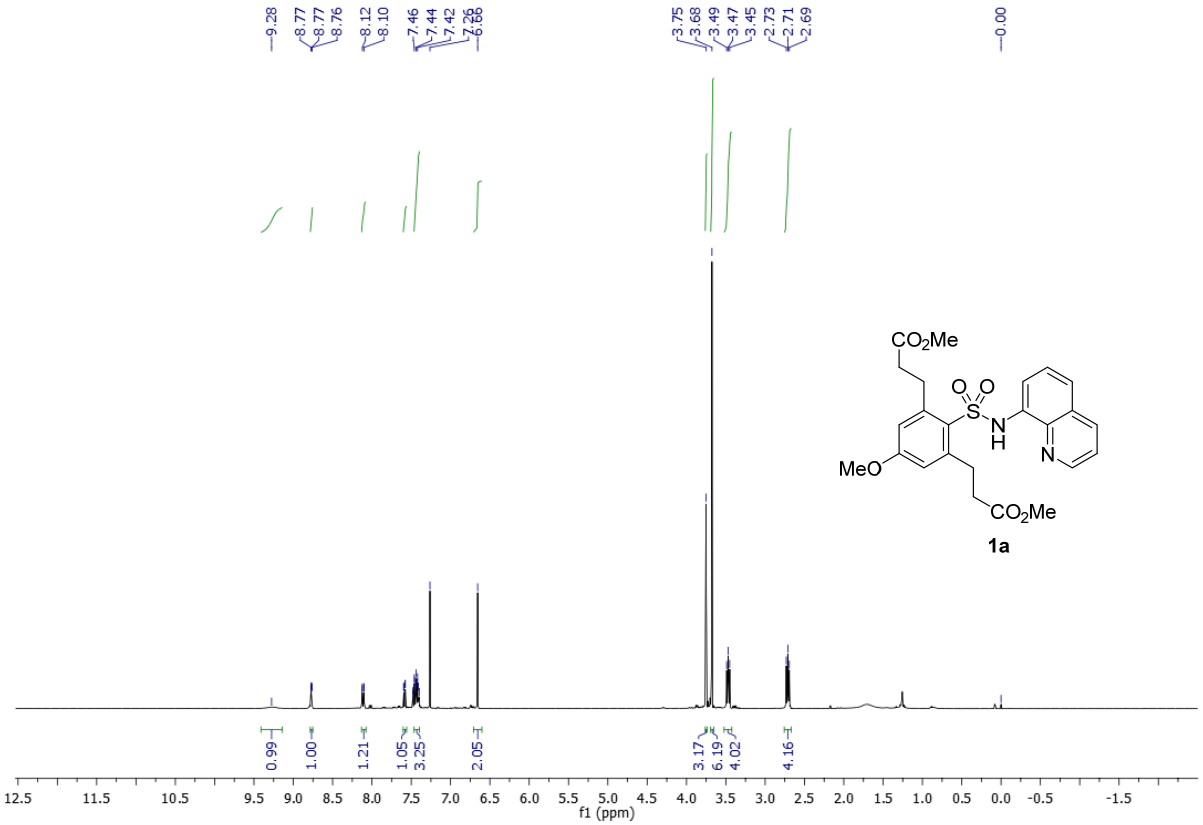
18. NMR spectra of byproducts

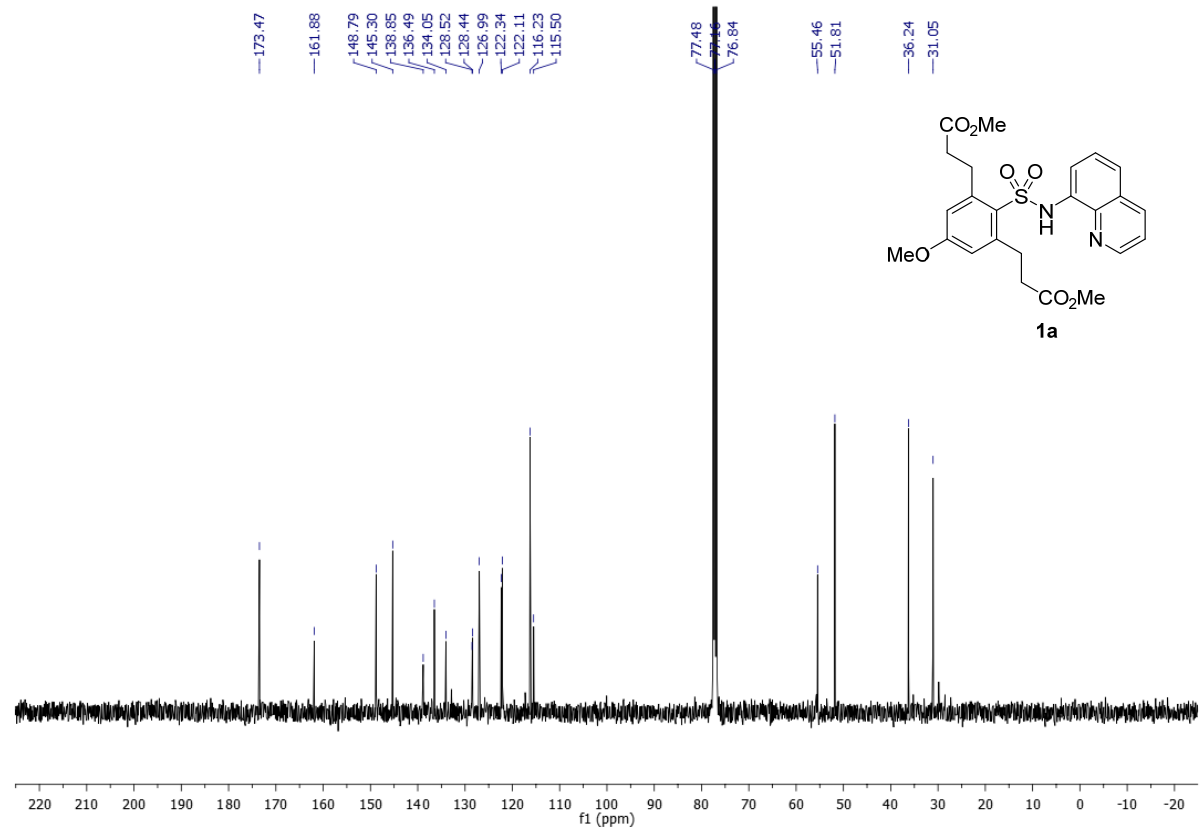


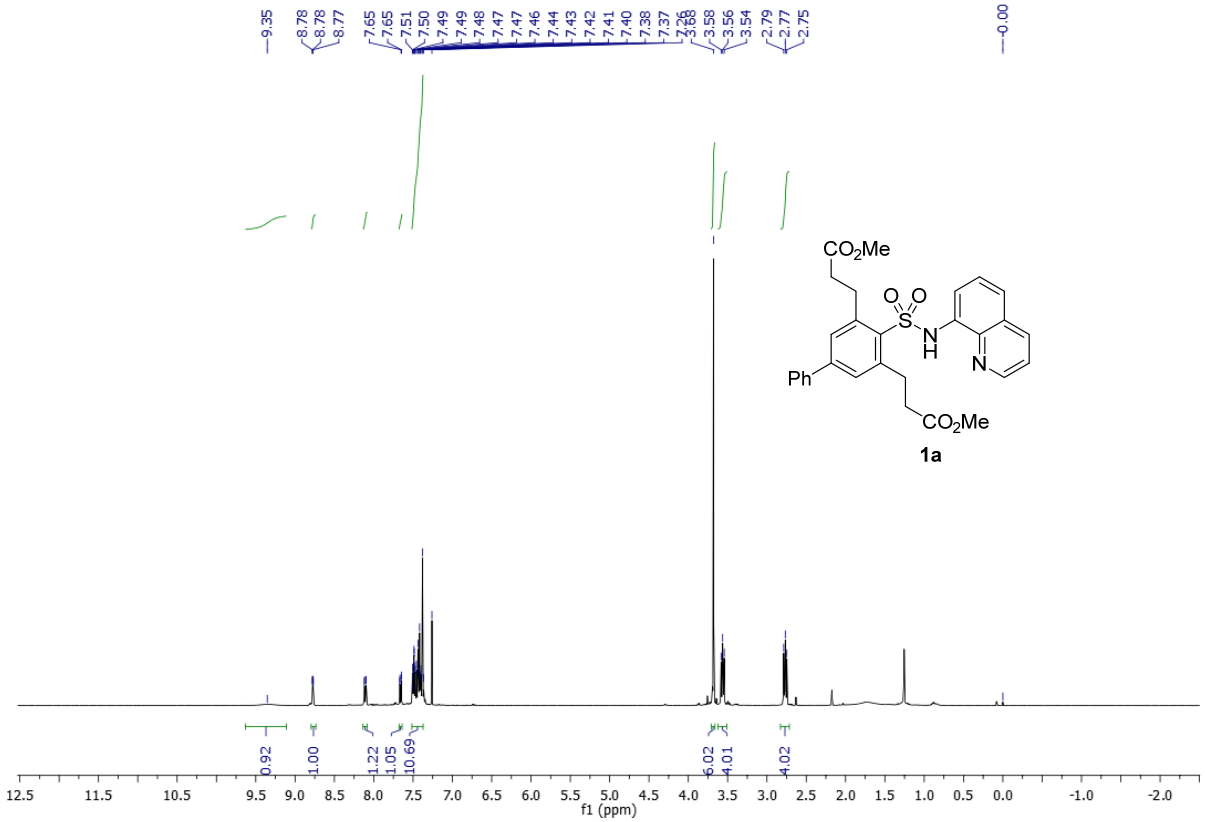


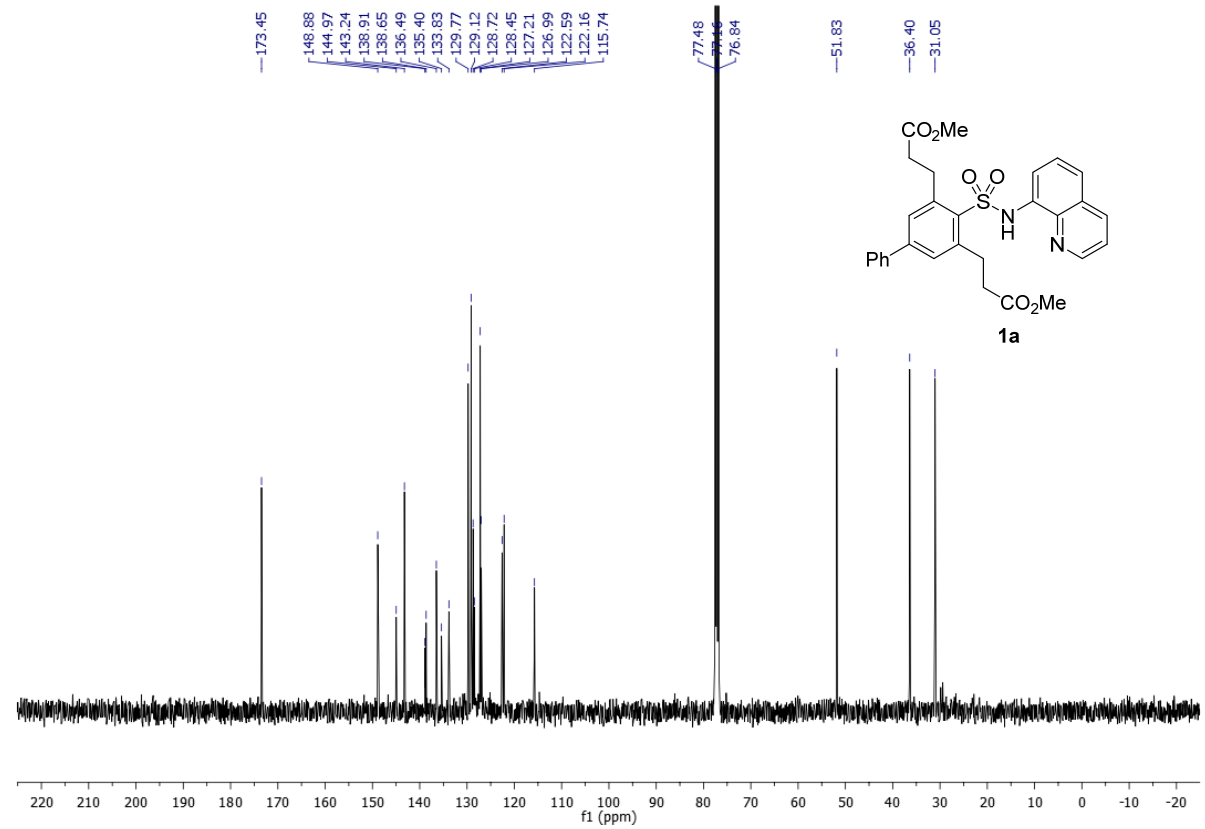

9.27
8.77
8.77
8.76
8.11
8.09
7.60
7.60
7.58
7.58
7.47
7.47
7.45
7.45
7.44
7.43
7.43
7.41
7.41
7.40
7.26
3.48
3.46
3.44
2.72
2.70
2.68
2.23
0.00
CO2Me
O
O
S
N
H
N
Me
CO2Me
1a
0.80
1.00
1.08
1.15
3.40
2.02
6.18
4.02
4.07
3.13
f1 (ppm)

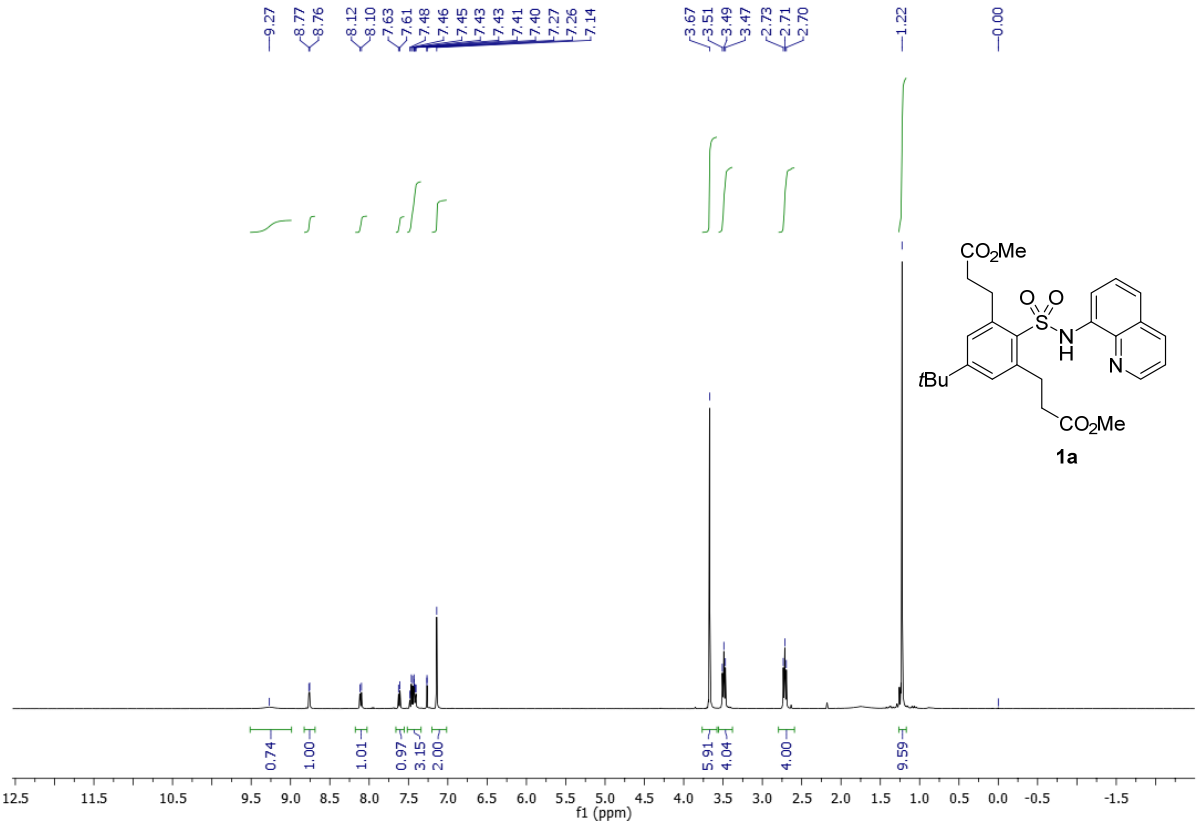

173.50
148.87
143.30
142.66
138.89
136.34
134.00
133.83
132.05
128.40
126.94
122.33
122.12
115.29
77.48
77.16
76.84
51.78
36.38
30.74
21.16
CO2Me
O
O
S
N
H
N
Me
CO2Me
1a
f1 (ppm)

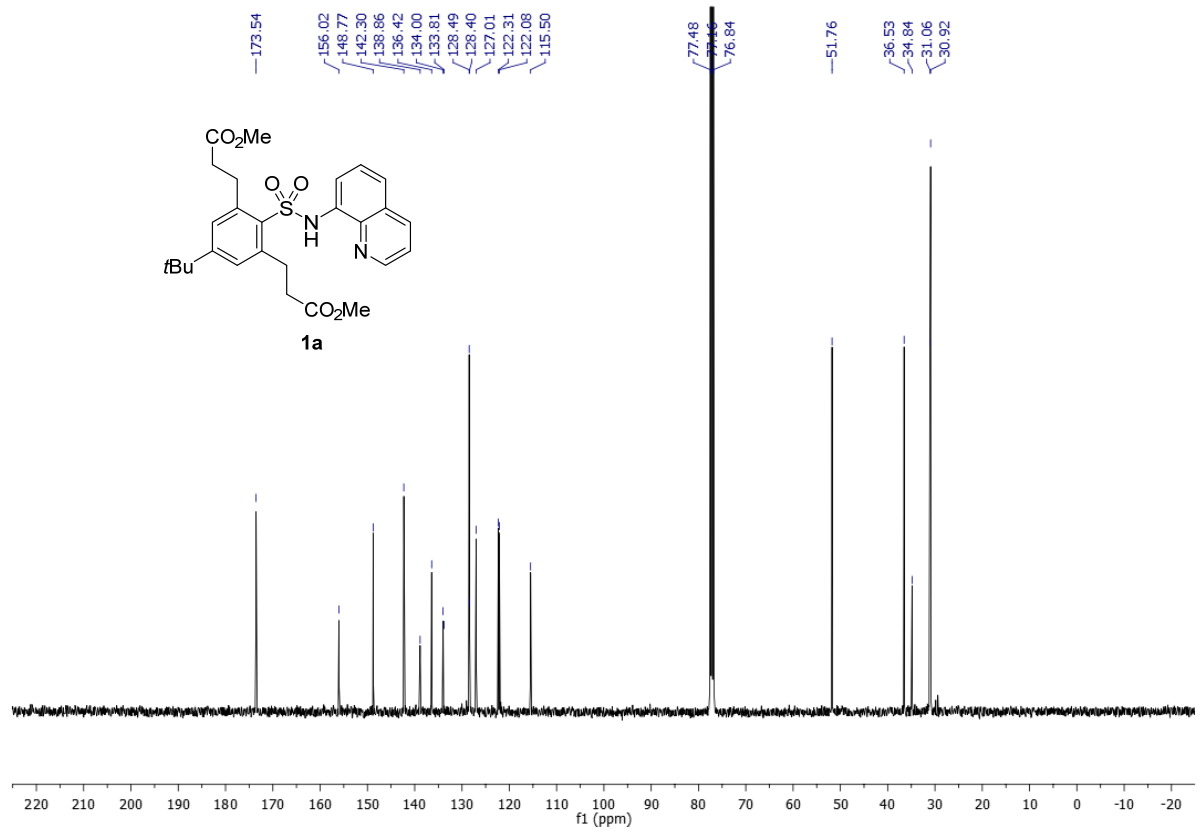

9.28
8.77
8.77
8.76
8.12
8.10
7.46
7.44
7.42
7.26
6.66
3.75
3.68
3.49
3.47
3.45
2.73
2.71
2.69
0.00
CO2Me
O
O
S
N
H
N
MeO
CO2Me
1a
0.99
1.00
1.21
1.05
3.25
2.05
3.17
6.19
4.02
4.16
f1 (ppm)


173.47
161.88
148.79
145.30
138.85
136.49
134.05
128.52
128.44
126.99
122.34
122.11
116.23
115.50
77.48
77.16
76.84
55.46
51.81
36.24
31.05
CO2Me
O
O
S
N
H
N
MeO
CO2Me
1a
f1 (ppm)


9.35
8.78
8.78
8.77
7.65
7.65
7.51
7.50
7.49
7.49
7.48
7.47
7.47
7.46
7.44
7.43
7.42
7.41
7.40
7.38
7.37
7.26
3.68
3.58
3.56
3.54
2.79
2.77
2.75
0.00
CO2Me
O
O
S
N
H
N
Ph
CO2Me
1a
0.92
1.00
1.22
1.05
10.69
6.02
4.01
4.02
f1 (ppm)


173.45
148.88
144.97
143.24
138.91
138.65
136.49
135.40
133.83
129.77
129.12
128.72
128.45
127.21
126.99
122.59
122.16
115.74
77.48
77.16
76.84
51.83
36.40
31.05
CO2Me
O
O
S
N
H
N
Ph
CO2Me
1a
f1 (ppm)


9.27
8.77
8.76
8.12
8.10
7.63
7.61
7.48
7.46
7.45
7.43
7.43
7.41
7.40
7.27
7.26
7.14
3.67
3.51
3.49
3.47
2.73
2.71
2.70
1.22
0.00
CO2Me
O
O
S
N
H
N
tBu
CO2Me
1a
0.74
1.00
1.01
0.97
3.15
2.00
5.91
4.04
4.00
9.59
f1 (ppm)


173.54
156.02
148.77
142.30
138.86
136.42
134.00
133.81
128.49
128.40
127.01
122.31
122.08
115.50
77.48
77.16
76.84
51.76
36.53
34.84
31.06
30.92
CO2Me
O
O
S
N
H
N
tBu
CO2Me
1a
f1 (ppm)

19. References

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