Supporting information for

I_2-Catalyzed Oxidative Dehydrogenative Tandem Cyclization of 2-Methylquinolines, Arylamines and 1,4-Dioxane

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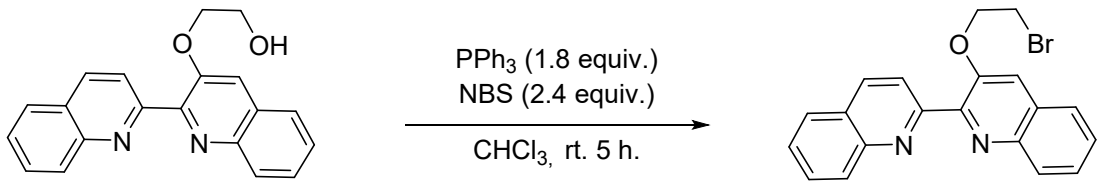
General information:

All reactions were carried out under an atmosphere of air unless otherwise noted. Column chromatography was performed using silica gel (200-300 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker-AV (400 and 100 MHz, respectively) instrument internally referenced to tetramethylsilane (TMS) or chloroform signals. Mass spectra was measured on bruker 15T HRMS instrument (maldi). Melting points were measured with a YUHUA X-5 melting point instrument and were uncorrected. All reagents were obtained from commercial suppliers and used without further purification.

General procedure for preparation (4a):

A 10 mL sealed tube was added 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), aniline (**2a**, 18 μL, 0.2 mmol), 70%TBHP (62 μL, 0.44 mmol), 1,4-dioxane (1.5 mL) by syringe. The reaction vessel was stirred at 110 °C for 16 h. After cooling to room temperature, the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc =1:1) to yield the desired product **4a** as yellow oil (38.5 mg, 60% yield).

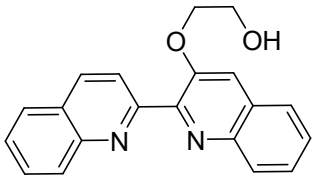
Preparation of 3-(2-bromoethoxy)-2,2'-biquinoline (4ab):[1]



4a **4ab**

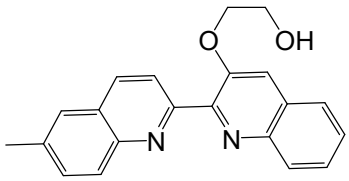
NBS (40.6 mg, 0.226 mmol), **4a** (29.4 mg, 0.093 mmol) and 1 mL $CHCl_3$ were added to a 10 mL reaction tube. After the reaction vessel was stirred for 30 minutes at room temperature, PPh_3 (44.6 mg, 0.170 mmol) was added to the reaction vessel and the sealed tube stirred for 5h at room temperature. The volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc =1:1) to yield the desired product **4ab** as yellow liquid (33.3 mg, 95% yield).

2-([2,2'-biquinolin]-3-yloxy)ethan-1-ol (4a)



^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.39 (d, J = 8.4, 1H), 8.26 (d, J = 8.4 Hz, 1H), 8.20 (d, J = 8.4 1H), 8.15 (d, J = 8.0 Hz, 1H), 7.90 (d, J = 8.4 Hz, 1H), 7.81-7.75 (m, 3H), 7.65-7.55 (m, 3H), 4.52 (t, J = 4.2 Hz, 2H), 4.00 (t, J = 4.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.4, 151.8, 150.8, 146.8, 143.5, 137.3, 130.1, 129.5, 129.2, 128.7, 127.7, 127.6, 127.6, 127.6, 127.2, 126.4, 122.6, 119.0, 73.2, 60.7; HRMS (maldi, m/z): calcd. for $C_{20}H_{17}N_2O_2$ $[M+H]^+$ 317.1290, found 317.1291.

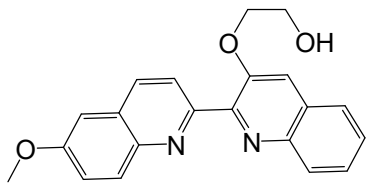
2-((6'-methyl-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4b)



The reaction was conducted with 6-methyl-quinolin (**1b**, 94.4 mg, 0.6 mmol) and aniline (**2a**, 18 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4b** as reddish-brown oil (24.5 mg, 37%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.29 (d, J = 8.8 Hz, 1H), 8.17-8.13 (m, 3H), 7.78 (d, J = 8.0 Hz, 1H), 7.73 (s, 1H), 7.65-7.54 (m, 4H), 6.62 (brs, 1H), 4.52 (t, J = 4.4 Hz, 2H), 4.00 (t, J = 4.4 Hz, 2H), 2.57 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 155.5, 151.9, 151.0, 145.4, 143.5, 137.2, 136.7, 132.4, 129.5, 129.2, 128.4, 127.8, 127.6, 127.6, 126.4, 126.4, 122.6, 119.1, 73.3, 60.7, 21.6; HRMS (maldi, m/z): calcd. for $C_{21}H_{19}N_2O_2$ $[M+H]^+$ 331.1447, found 331.1445.

2-((6'-methoxy-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4c)

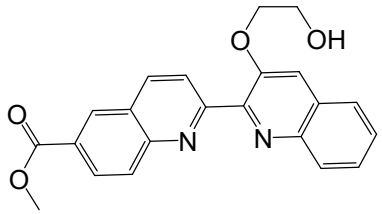


The reaction was conducted with 6-methoxyquinaldine (**1c,** 103.9 mg, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and aniline (**2a**, 18 μL, 0.2 mmol). The residue was purified by column

chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4c** as reddish-brown oil (32.4 mg, 47%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.27 (d, J = 8.8 Hz, 1H), 8.18-8.13 (m, 3H), 7.78 (d, J = 8.0 Hz, 1H), 7.73 (s, 1H), 7.64-7.61 (m, 1H), 7.55 (t, J = 7.4 Hz, 1H), 7.41 (dd, J = 2.4, 9.2 Hz, 1H), 7.14 (d, J = 2.4 Hz, 1H), 6.61 (brs, 1H), 4.51 (t, J = 4.0 Hz, 2H), 4.01-3.97 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 158.3, 154.0, 151.9, 150.9, 143.5, 142.9, 136.0, 130.2, 129.4, 129.1, 129.0, 127.6, 127.5, 126.5, 122.9, 119.1, 104.9, 73.3, 60.7, 55.5. HRMS (maldi, m/z): calcd. for $C_{21}H_{19}N_2O_3$ $[M+H]^+$ 347.1396, found 347.1395.

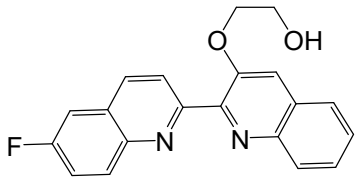
methyl 3'-(2-hydroxyethoxy)-[2,2'-biquinoline]-6-carboxylate (4d)



The reaction was conducted with 2-methylquinoline-6-carboxylate (**1d**, 120.7 mg, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and aniline (**2a**, 18 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4d** as yellow oil (24.8 mg, 33%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.67 (s, 1H), 8.49 (d, J = 8.8 Hz, 1H), 8.36-8.28 (m, 3H), 8.14 (d, J = 8.4 Hz, 1H), 7.81 (t, J = 8.0 Hz, 1H), 7.76 (s, 1H), 7.67-7.57 (m, 2H), 4.53 (t, J = 4.2 Hz, 2H), 4.02-4.00 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 166.5, 158.6, 151.9, 150.2, 148.8, 143.6, 138.5, 130.7, 129.6, 129.6, 129.4, 129.2, 128.6, 127.9, 127.8, 126.9, 126.4, 123.5, 119.1, 73.2, 60.7, 52.5; HRMS(maldi, m/z): calcd. for $C_{22}H_{19}N_2O_4$ $[M+H]^+$ 375.1435, found 375.1344.

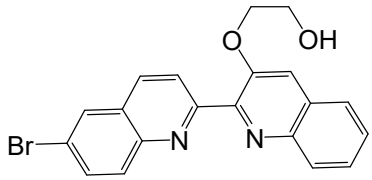
2-((6'-fluoro-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4e)



The reaction was conducted with 6-fluoro-2-methylquinoline (**1e**, 96.7 mg, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and aniline (**2a**, 18 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 2:1) afforded the product **4e** as reddish-brown oil (28.6 mg, 43%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.38 (d, J = 8.4 Hz, 1H), 8.19 (d, J = 8.4 Hz, 1H), 8.14 (d, J = 8.4 Hz, 1H), 7.93-7.88 (m, 2H), 7.80 (d, J = 8.4 Hz, 1H), 7.76 (s, 1H), 7.64 (t, J = 7.0 Hz, 1H), 7.58 (t, J = 7.0 Hz, 1H), 7.43-7.48 (m, 1H), 4.52 (t, J = 4.2 Hz, 2H), 4.01 (t, J = 4.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 163.4 (d, J = 249.1 Hz), 157.5, 151.9, 150.4, 147.8 (d, J = 12.4 Hz), 143.6, 137.3, 129.6 (d, J = 9.9 Hz), 129.6, 129.3, 127.8 (d, J = 8.5 Hz), 126.4, 124.8, 122.0, 122.0, 119.2, 117.8 (d, J = 25.3 Hz), 112.6(d, J = 20.7 Hz), 73.4, 60.7. HRMS (maldi, m/z): calcd. for $C_{20}H_{16}N_2O_2F$ $[M+H]^+$ 335.1196, found 335.1194.

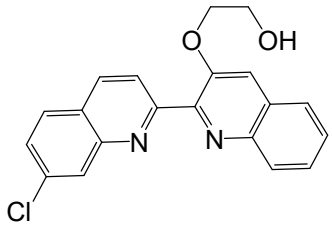
2-((6'-bromo-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4f)



The reaction was conducted with 6-bromo-2-methylquinoline (**1f**, 133.2 mg, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and aniline (**2a**, 18 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 2:1) afforded the product **4f** as yellow oil (39.6 mg, 50%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.27 (q, J = 8.9 Hz, 2H), 8.13 (q, J = 4.3 Hz, 2H), 8.06 (d, J = 1.6 Hz, 1H), 7.84-7.79 (m, 2H), 7.74 (s, 1H), 7.66-7.56 (m, 2H), 6.25 (brs, 1H), 4.52 (t, J = 4.4 Hz, 2H), 4.00 (t, J = 4.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.8, 151.8, 150.2, 145.4, 143.5, 136.3, 133.5, 130.5, 129.6, 129.6, 129.3, 128.8, 127.8, 127.7, 126.4, 123.6, 121.2, 119.0, 73.1, 60.7. HRMS (maldi, m/z): calcd. for $C_{20}H_{16}N_2O_2Br$ $[M+H]^+$ 395.0395, found 395.0395.

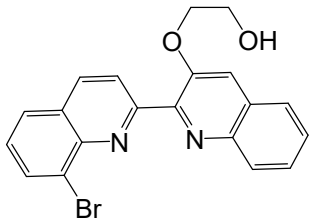
2-((7'-chloro-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4g)



The reaction was conducted with 7-chloro-2-methylquinoline (**1g**, 106.6 mg, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and aniline (**2a**, 18 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 2:1) afforded the product **4g** as brown oil (41.8 mg, 60%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.36 (d, J = 8.4 Hz, 1H), 8.26 (d, J = 1.2 Hz, 1H), 8.21 (d, J = 8.8 Hz, 1H), 8.13 (d, J = 8.4 Hz, 1H), 7.84-7.79 (m, 2H), 7.75 (s, 1H), 7.66-7.62 (m, 1H), 7.59-7.54 (m, 2H), 6.25 (brs, 1H), 4.52 (t, J = 4.2 Hz, 2H), 4.00 (t, J = 4.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 157.5, 151.8, 150.3, 147.2, 143.5, 137.1, 136.0, 129.5, 129.3, 128.8, 128.3, 127.8, 127.7, 127.7, 126.4, 126.1, 122.9, 119.1, 73.2, 60.7; HRMS (maldi, m/z): calcd. for $C_{20}H_{17}N_2O_2Cl$ $[M+H]^+$ 351.0900, found 351.0899.

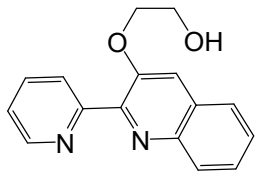
2-((8'-amino-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4h)



The reaction was conducted with 8-bromo-2-methylquinoline (**1h**, 133.2 mg, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and aniline (**2a**, 18 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 2:1) afforded the product **4h** as brown oil (35.4 mg, 45%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.33 (d, J = 8.4 Hz, 1H), 8.18-8.09 (m, 3H), 7.87 (d, J = 7.6 Hz, 1H), 7.80 (d, J = 7.6 Hz, 1H), 7.65-7.57 (m, 3H), 7.45 (t, J = 7.6 Hz, 1H), 4.34 (t, J = 4.4 Hz, 2H), 3.99 (t, J = 4.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 157.8, 151.4, 150.3, 144.5, 143.1, 137.2, 133.5, 129.5, 129.3, 129.0, 127.7, 127.6, 127.3, 126.4, 125.1, 123.5, 115.7, 70.3, 61.0; HRMS (maldi, m/z): calcd. for $C_{20}H_{17}N_2O_2Br$ $[M+H]^+$ 395.0395, found 395.0394.

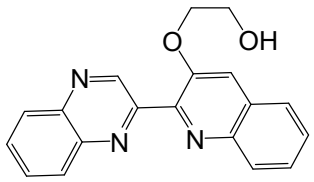
2-((2-(pyridin-2-yl)quinolin-3-yl)oxy)ethan-1-ol (4i)



The reaction was conducted with 2-methylpyridine (**1i**, 59 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and aniline (**2a**, 18 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 2:1) afforded the product **4i** as yellow solid (26.5 mg, 41%), melting point (m.p.): 123.8-125.0 °C.

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.67 (d, J = 4.8 1H), 8.13-8.08 (m, 2H), 7.96-7.92 (m, 1H), 7.78 (d, J = 7.6 1H), 7.71 (s, 1H), 7.64-7.54 (m, 2H), 7.41 (t, J = 6.2 1H), 4.48 (t, J = 4.4 Hz, 2H), 3.94 (t, J = 4.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.5, 151.7, 150.6, 147.9, 143.5, 137.6, 129.5, 129.2, 127.6, 127.5, 126.4, 125.1, 123.6, 119.0, 73.5, 60.7; HRMS (maldi, m/z): calcd. for $C_{16}H_{15}N_2O_2$ $[M+H]^+$ 269.1290, found 269.1288.

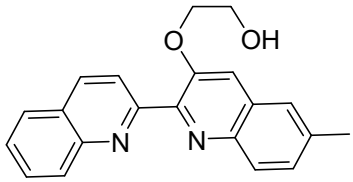
2-((2-(quinoxalin-2-yl)quinolin-3-yl)oxy)ethan-1-ol (4j)



The reaction was conducted with 2-methylquinoxaline (**1j**, 77 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and aniline (**2a**, 18 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 2:1) afforded the product **4j** as yellow solid (18.2 mg, 30%), melting point (m.p.): 85.0-87.0 °C.

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 9.68 (s, 1H), 8.25-8.18 (m, 3H), 7.85-7.80 (m, 3H), 7.74 (s, 1H), 7.68-7.58 (m, 2H), 5.45 (brs, 1H), 4.50 (t, J = 4.4 Hz, 2H), 4.03 (t, J = 4.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 152.2, 150.6, 147.9, 146.8, 143.6, 142.0, 140.7, 130.5, 129.8, 129.4, 129.3, 129.1, 128.2, 127.9, 126.4, 118.3, 72.6, 60.8; HRMS (maldi, m/z): calcd. for $C_{19}H_{15}N_3NaO_2$ $[M+Na]^+$ 340.1062, found 340.1056.

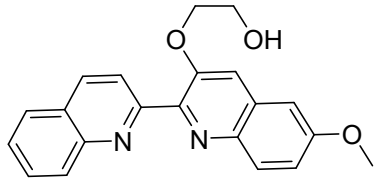
2-((6-methyl-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4k)



The reaction was conducted with 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and 4-methyl- benzenamine (**2b**, 21.4 mg, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4k** as yellow-brown oil (36.4 mg, 55%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.38 (d, J = 8.4 Hz，1H), 8.26 (d, J = 8.4 Hz, 1H), 8.20 (d, J = 8.8 Hz，1H), 8.03 (d, J = 8.4 Hz, 1H), 7.90 (d, J = 8.0 Hz, 1H), 7.77 (t, J = 7.6 Hz，1H), 7.66 (s, 1H), 7.60 (t, J = 7.6 Hz，1H), 7.55 (s, 1H), 7.46 (dd, J = 1.6, 8.8 Hz, 1H), 4.51 (t, J = 4.4 Hz, 2H), 4.00 (t, J = 4.2 Hz, 2H), 2.56 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.6, 152.0, 149.8, 146.9, 142.2, 137.8, 137.3, 130.0, 130.0, 129.3, 129.2 128.8, 127.7, 127.6, 127.1, 125.3, 122.6, 118.6, 73.3, 60.7, 21.7; HRMS (maldi, m/z): calcd. for $C_{21}H_{19}N_2O_2$ $[M+H]^+$ 331.1447, found 331.1445.

2-((6-methoxy-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4l)

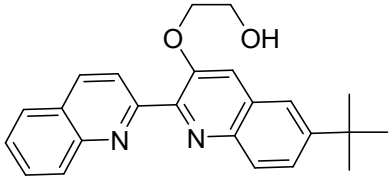


The reaction was conducted with 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and p-anisidine (**2c**, 24.6 mg, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4l** as reddish-brown oil (15.0 mg, 20%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.37 (d, J = 8.4 Hz, 1H), 8.25 (d, J = 8.8 Hz, 1H), 8.20 (d, J = 8.8 Hz, 1H), 8.02 (d, J = 8.8 Hz, 1H), 7.89 (d, J = 8.0 Hz, 1H), 7.76 (t, J = 7.6 Hz, 1H), 7.65 (s, 1H), 7.59 (t, J = 7.4 Hz, 1H), 7.29 (d, J = 2.8 Hz, 1H), 7.05 (d, J = 2.8 Hz, 1H), 4.51 (t, J = 4.4 Hz, 2H), 4.00 (t, J = 4.2 Hz, 2H), 3.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 158.8, 156.6, 152.4, 148.0, 146.9, 139.6, 137.3, 131.0, 130.6, 130.0, 128.7, 127.7, 127.6, 127.0, 122.6, 120.4, 118.3, 104.0, 73.3, 60.7, 55.6; HRMS (maldi, m/z): calcd. for $C_{21}H_{19}N_2O_3$ $[M+H]^+$

347.1396, found 347.1395.

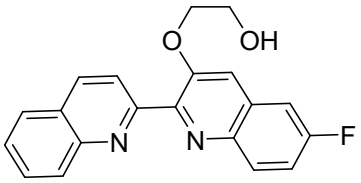
2-((6-(tert-butyl)-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4m)



The reaction was conducted with 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and 4-tert-butylaniline (**2d**, 32 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4m** as yellow oil (34.4 mg, 46%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.38 (d, J = 8.4 Hz, 1H), 8.26 (d, J = 8.4 Hz, 1H), 8.20 (d, J = 8.4 Hz, 1H), 8.08 (d, J = 8.8 Hz, 1H), 7.90 (d, J = 8.0 Hz, 1H), 7.79-7.71 (m, 4H), 7.60 (t, J = 7.6 Hz, 1H), 6.65 (brs, 1H), 4.53 (t, J = 4.4 Hz, 2H), 4.00 (t, J = 4.4 Hz, 2H), 1.45 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.6, 151.9 , 150.7, 150.1, 146.9, 142.2, 137.4, 130.0, 129.0, 128.8, 127.7, 127.6, 127.1, 126.8, 122.6, 121.5, 119.5, 73.4, 60.7, 35.1, 31.2; HRMS (maldi, m/z): calcd. for $C_{24}H_{25}N_2O_2$ $[M+H]^+$ 373.1916, found 373.1915.

2-((6-fluoro-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4n)

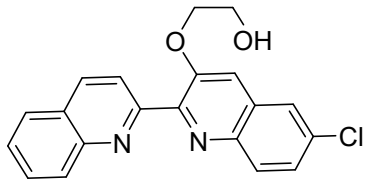


The reaction was conducted with 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and 4-fluoroaniline (**2e**, 19 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4n** as brown oil (36.0 mg, 54%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.39 (d, J = 8.8 Hz, 1H), 8.25 (d, J = 8.8 Hz, 1H), 8.18 (d, J = 8.8 Hz, 1H), 8.15-8.11 (m, 1H), 7.90 (d, J = 8.0 Hz, 1H), 7.78 (t, J = 7.6 Hz, 1H), 7.67 (s, 1H), 7.61 (t, J = 7.4 Hz, 1H), 7.40 (d, J = 8.4 Hz, 2H), 4.51 (t, J = 4.4 Hz, 2H), 4.01 (t, J = 4.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 161.9 (d, J = 247.7 Hz), 156.2, 152.6, 150.1, 146.9,

140.5, 137.4, 132.1 (d, J = 9.8 Hz), 130.2, 130.1, 128.8, 127.7, 1267.6, 127.2, 122.4, 118.2 (d, J = 5.3 Hz), 117.8 (d, J = 25.6 Hz), 109.5 (d, J = 22.1 Hz), 73.3, 60.7; HRMS (maldi, m/z): calcd. for $C_{20}H_{16}N_2O_2F$ $[M+H]^+$ 335.1196, found 335.1194.

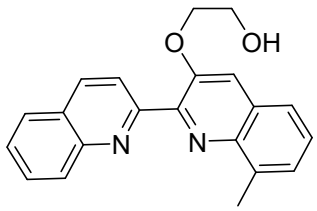
2-((6-chloro-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4o)



The reaction was conducted with 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and 4-chloroaniline (**2f**, 25.5 mg, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4o** as brown oil (26.8 mg, 38%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.38 (d, J = 8.4 Hz, 1H), 8.25 (d, J = 8.4 Hz, 1H), 8.18 (d, J = 8.4 Hz, 1H), 8.06 (d, J = 8.8 Hz, 1H), 7.90 (d, J = 8.0 Hz, 1H), 7.79-7.76 (m, 2H), 7.63-7.59 (m, 2H), 7.55 (dd, J = 2.4, 9.2 Hz, 1H), 4.50 (t, J = 4.2 Hz, 2H), 4.00 (t, J = 4.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.1, 152.6, 151.0, 146.9, 141.7, 137.4, 133.5, 131.1, 130.2, 129.9, 128.8, 128.5, 127.8, 127.6, 127.3, 125.0, 122.4, 117.8, 73.3, 60.7; HRMS (maldi, m/z): calcd. for $C_{20}H_{16}N_2O_2Cl$ $[M+H]^+$ 351.0900, found 351.0899.

2-((8-methyl-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4p)

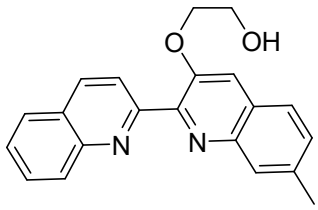


The reaction was conducted with 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and o-toluidine (**2g**, 21 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4p** as yellow-brown oil (24.8 mg, 35%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.38 (d, J = 8.4 Hz, 1H), 8.32 (d, J = 8.8 Hz, 1H), 8.27

(d, J = 8.4 Hz, 1H), 7.90 (d, J = 8.0 Hz, 1H), 7.77 (t, J = 7.2 Hz, 1H), 7.73 (s, 1H), 7.64-7.58 (m, 2H), 7.48-7.43 (m, 2H), 4.54 (t, J = 4.4 Hz, 2H), 4.03 (t, J = 4.4 Hz, 2H), 2.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.9, 151.9, 148.9, 146.7, 142.6, 137.7, 137.1, 130.0, 129.3, 128.7, 127.8, 127.7, 127.5, 127.1 ,124.4, 123.0, 119.6, 73.4, 60.7, 17.9; HRMS (maldi, m/z): calcd. for $C_{21}H_{19}N_2O_2$ $[M+H]^+$ 331.1447, found 331.1445.

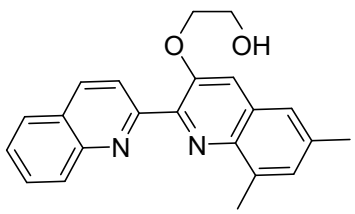
2-((7-methyl-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4q)



The reaction was conducted with 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and m-toluidine (**2h**, 21 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4q** as yellow-brown oil (35.6 mg, 54%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.36 (d, J = 8.4 Hz, 1H), 8.26 (d, J = 8.8 Hz, 1H), 8.19 (d, J = 8.8 Hz, 1H), 7.92 (s, 1H), 7.88 (d, J = 8.0 Hz, 1H), 7.76 (t, J = 7.2 Hz, 1H), 7.70 (s, 1H), 7.67 (d, J = 8.4 Hz, 1H), 7.59 (t, J = 7.6 Hz, 1H), 7.39 (d, J = 8.4 Hz, 1H), 4.50 (t, J = 4.4 Hz, 2H), 3.99 (t, J = 4.2 Hz, 2H), 2.55 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.6, 151.3, 150.5, 146.8, 143.7, 137.7, 137.3, 130.0, 129.9, 128.7, 128.5, 127.7, 127.5, 127.1 ,127.0, 126.0, 122.6, 119.1, 73.2, 60.7, 21.6; HRMS (maldi, m/z): calcd. for $C_{21}H_{19}N_2O_2$ $[M+H]^+$ 331.1447, found 331.1445.

2-((6,8-dimethyl-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4r)

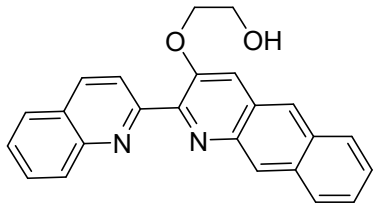


The reaction was conducted with 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and 2,4-dimethyl aniline (**2i**, 25 μL, 0.2 mmol). The residue was purified by column

chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4r** as brown oil (21.7 mg, 32%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.33 (q, J = 8.1 Hz, 2H), 8.27 (d, J = 8.4 Hz, 1H), 7.88 (d, J = 8.0 Hz, 1H), 7.75 (t, J = 7.2 Hz, 1H), 7.63 (s, 1H), 7.58 (t, J = 7.4 Hz, 1H), 7.38 (s, 1H), 7.31 (s, 1H), 6.93 (brs, 1H), 4.52 (t, J = 4.2 Hz, 2H), 4.02 (t, J = 4.2 Hz, 2H), 2.80 (s, 3H), 2.50 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 157.1, 152.0, 148.0, 146.8, 141.3, 137.6, 137.3, 137.0, 130.2, 129.9, 129.5, 128.7, 127.7, 127.5, 127.0, 123.2, 123.0, 119.1, 73.3, 60.7, 21.7, 17.8; HRMS (maldi, m/z): calcd. for $C_{22}H_{21}N_2O_2$ $[M+H]^+$ 345.1603, found 345.1597.

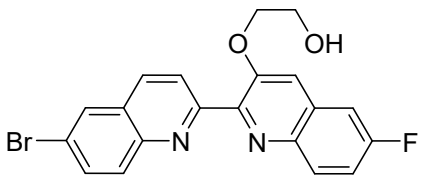
2-((2-(quinolin-2-yl)benzo[g]quinolin-3-yl)oxy)ethan-1-ol (4s)



The reaction was conducted with 2-methylquinoline (**1a**, 80 μL, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and 2-naphthylamine (**2j**, 28.6 mg, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1) afforded the product **4s** as brown oil (28.8 mg, 41%).

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.61-8.58 (m, 2H), 8.40 (d, J = 8.4 Hz, 1H), 8.29 (d, J = 8.4 Hz, 2H), 8.03 (d, J = 8.8 Hz, 1H), 7.97-7.90 (m, 3H), 7.78 (t, J = 7.6 Hz, 1H), 7.74-7.67 (m, 2H), 7.61 (t, J = 7.6 Hz, 1H), 4.66 (t, J = 3.6 Hz, 2H), 4.06 (t, J = 3.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.5, 152.8, 149.0, 146.9, 142.9, 137.3, 132.3, 130.1, 129.1, 128.9, 128.8, 128.8, 128.0, 127.8, 127.7, 127.6, 127.2, 126.9, 126.8, 123.0, 122.7, 116.5, 74.0, 60.9; HRMS (maldi, m/z): calcd. for $C_{24}H_{19}N_2O_2$ $[M+H]^+$ 367.1447, found 367.1446.

2-((6'-bromo-6-fluoro-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4t)

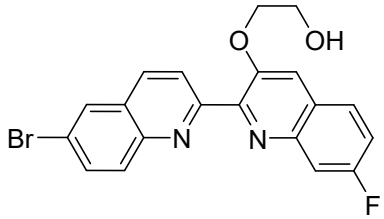


The reaction was conducted with 6-bromo-2-methylquinoline (**1f**, 133.2 mg, 0.6 mmol),

70%TBHP (84 μL, 0.6 mmol) and 4-fluoroaniline (**2e**, 19 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 2:1) afforded the product **4t** as brown solid (35.2 mg, 44%), melting point (m.p.): 171.6−173.0 °C.

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.29 (d, J = 8.8 Hz, 1H), 8.22 (d, J = 8.4 Hz, 1H), 8.12 (d, J = 9.2 Hz, 2H), 8.06 (d, J = 2.0 Hz, 1H), 7.83 (dd, J = 2.0, 8.8 Hz, 1H), 7.73-7.70 (m, 2H), 7.52 (dd, J = 2.0, 8.8 Hz, 1H), 6.22 (brs, 1H), 4.50 (t, J = 4.2 Hz, 2H), 4.00 (t, J = 4.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 163.4 (d, J = 249.1 Hz), 157.5, 151.9, 150.4, 147.8 (d, J = 12.4 Hz), 143.6, 137.3, 129.6 (d, J = 9.9 Hz), 129.6, 129.3, 127.8(d, J = 8.5 Hz), 126.4, 124.8, 122.0, 122.0, 119.2, 117.8 (d, J = 25.3 Hz), 112.6 (d, J = 20.7 Hz), 73.4, 60.7, 21.8; HRMS (maldi, m/z): calcd. for $C_{20}H_{16}N_2O_2BrF$ $[M+H]^+$ 413.0301, found 413.0300.

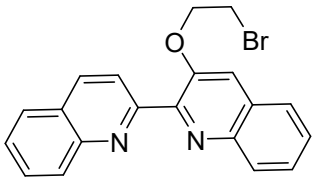
2-((6'-bromo-7-fluoro-[2,2'-biquinolin]-3-yl)oxy)ethan-1-ol (4u)



The reaction was conducted with 6-bromo-2-methylquinoline (**1f**, 133.2 mg, 0.6 mmol), 70%TBHP (84 μL, 0.6 mmol) and 3-fluoroaniline (**2k**, 19 μL, 0.2 mmol). The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 2:1) afforded the product **4u** as brown solid (33.2 mg, 40%), melting point (m.p.): 183.0−185.1 °C.

^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.29 (d, J = 8.8 Hz, 1H), 8.23 (d, J = 8.4 Hz, 1H), 8.14-8.10 (m, 2H), 8.06 (d, J = 2.0 Hz, 1H), 7.83 (dd, J = 2.4, 9.0 Hz, 1H), 7.67 (s, 1H), 7.42-7.38 (m, 2H), 6.22 (s, 1H), 4.50 (t, J = 4.4 Hz, 2H), 4.00 (t, J = 4.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 161.9 (d, J = 246.7 Hz), 156.6, 151.5, 151.1, 145.5, 144.1 (d, J = 12.2 Hz), 136.3, 133.6, 130.6, 129.7, 128.9, 128.1 (d, J = 9.5 Hz), 126.2, 123.5, 121.3, 119.3,118.4 (d, J = 25.4 Hz), 113.2 (d, J = 20.4 Hz), 73.3, 60.7; HRMS (maldi, m/z): calcd. for $C_{20}H_{16}N_2O_2BrF$ $[M+H]^+$ 413.0301, found 413.0301.

3-(2-bromoethoxy)-2,2'-biquinoline (4ab):

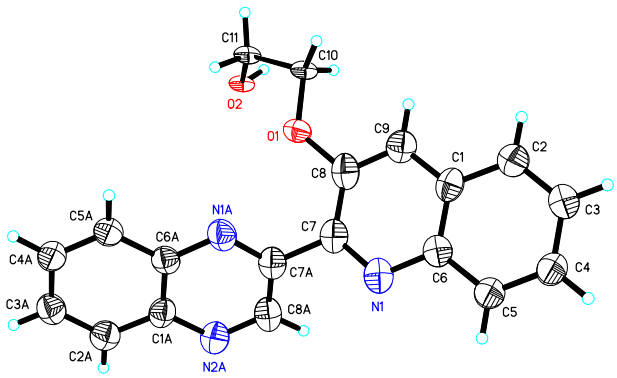


^{1}H NMR (400 MHz, $CDCl_3$, ppm) δ 8.29 (d, J = 8.4, 2H), 8.23 (d, J = 8.4 Hz, 1H), 8.02 (d, J = 8.8 1H), 7.89 (d, J = 8.0 Hz, 1H), 7.79-7.74 (m, 2H), 7.64-7.54 (m, 4H), 4.46 (t, J = 6.2 Hz, 2H), 3.62 (t, J = 6.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 155.9, 151.2, 150.3, 148.1, 143.5, 135.8, 130.1, 129.9, 129.5, 128.9, 127.7, 127.6, 127.5, 127.5, 126.9, 126.3, 122.8, 115.6, 68.7, 28.6; MS (maldi, m/z): calcd. for $C_{20}H_{16}BrN_2O$ $[M+H]^+$ 379.0446, found 379.0337.

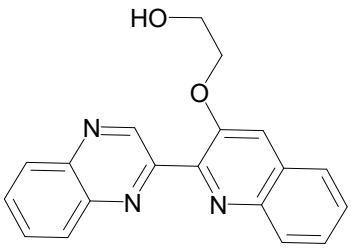
References

[1] (a) A. Martins and M. Lautens, A Palladium-Catalyzed approach to polycyclic sulfur heterocycles, *J. Org. Chem.,* 2008, **73**, 8705 – 8710; (b) A. C. Kruegel, S. Rakshit, X. Li, and D. Sames, Constructing iboga alkaloids via C–H bond functionalization: examination of the direct and catalytic union of heteroarenes and isoquinuclidine Alkenes, *J. Org. Chem.,* 2015, **80**, 2062–2071.

Crystal data and structure refinement for 4j



mo_ddz20083_0m CCDC 2101003



4j

Table 1. Crystal data and structure refinement for **4j**.

Identification code	mo_ddz20083_0m	
Empirical formula	C_{19} H_{15} N_3 O_2	
Formula weight	317.34	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 22.630(7) Å	α= 90° .
	b = 3.8102(11) Å	β= 99.852(8)° .
	c = 18.998(6) Å	γ = 90° .
Volume	1613.9(8) $Å^3$	
Z	4	
Density (calculated)	1.306 Mg/m^3	
Absorption coefficient	0.087 mm^{-1}	
F(000)	664	
Crystal size	0.150 x 0.100 x 0.050 mm^3	
Theta range for data collection	2.591 to 24.996° .	
Index ranges	-26<=h<=25, -4<=k<=4, -22<=l<=22	
Reflections collected	9644	
Independent reflections	1428 [R(int) = 0.0572]	
Completeness to theta = 25.242°	97.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7456 and 0.6177	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1428 / 73 / 157	

Goodness-of-fit on F^2	1.389
Final R indices [I>2sigma(I)]	R1 = 0.1168, wR2 = 0.3369
R indices (all data)	R1 = 0.1616, wR2 = 0.3829
Extinction coefficient	0.026(10)
Largest diff. peak and hole	0.685 and -0.384 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for mo_ddz20083_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	4743(3)	3312(17)	3687(3)	63(2)
N(2)	5786(2)	1795(12)	3954(3)	75(2)
N(1)	5766(2)	-741(10)	2567(2)	72(1)
C(1)	6298(2)	397(12)	3766(3)	67(1)
C(2)	6840(2)	240(13)	4257(3)	74(2)
C(3)	7342(2)	-1146(13)	4049(3)	74(2)
C(4)	7324(2)	-2350(13)	3349(3)	73(2)
C(5)	6805(2)	-2253(13)	2867(3)	67(2)
C(6)	6278(2)	-834(11)	3058(3)	62(1)
C(7)	5279(2)	544(12)	2769(3)	66(2)
C(8)	5286(2)	1911(14)	3468(3)	74(2)
C(9)	5786(2)	1795(12)	3954(3)	75(2)
O(2)	3978(4)	9210(20)	3798(4)	36(2)
C(10)	4695(5)	5210(30)	4332(6)	38(2)
C(11)	4083(4)	6220(30)	4262(5)	37(2)
O(2')	3957(5)	7110(30)	4040(5)	38(2)
C(10')	4741(8)	3980(50)	4430(7)	38(2)
C(11')	4100(11)	4520(80)	4490(16)	38(2)

Table 3. Bond lengths [Å] and angles [°] for mo_ddz20083_0m.

O(1)-C(10')	1.435(14)
O(1)-C(10)	1.442(11)
O(1)-C(8)	1.462(8)

N(2)-C(8)	1.333(7)
N(2)-C(1)	1.378(7)
N(1)-C(7)	1.322(6)
N(1)-C(6)	1.359(6)
C(1)-C(2)	1.410(7)
C(1)-C(6)	1.417(7)
C(2)-C(3)	1.370(7)
C(2)-H(2)	0.9300
C(3)-C(4)	1.401(7)
C(3)-H(3)	0.9300
C(4)-C(5)	1.360(6)
C(4)-H(4)	0.9300
C(5)-C(6)	1.413(6)
C(5)-H(5)	0.9300
C(7)-C(8)	1.424(8)
C(7)-C(7)#1	1.483(9)
O(2)-C(11)	1.434(10)
O(2)-H(2A)	0.8200
C(10)-C(11)	1.423(13)
C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
O(2')-C(11')	1.310(18)
O(2')-H(2')	0.8200
C(10')-C(11')	1.489(18)
C(10')-H(10C)	0.9700
C(10')-H(10D)	0.9700
C(11')-H(11C)	0.9700
C(11')-H(11D)	0.9700

C(10')-O(1)-C(8)	119.3(7)
C(10)-O(1)-C(8)	127.8(6)
C(8)-N(2)-C(1)	119.0(5)
C(7)-N(1)-C(6)	117.8(5)
N(2)-C(1)-C(2)	121.4(5)
N(2)-C(1)-C(6)	118.9(5)
C(2)-C(1)-C(6)	119.7(5)

C(3)-C(2)-C(1)	119.7(5)
C(3)-C(2)-H(2)	120.1
C(1)-C(2)-H(2)	120.1
C(2)-C(3)-C(4)	120.6(5)
C(2)-C(3)-H(3)	119.7
C(4)-C(3)-H(3)	119.7
C(5)-C(4)-C(3)	120.7(4)
C(5)-C(4)-H(4)	119.6
C(3)-C(4)-H(4)	119.6
C(4)-C(5)-C(6)	120.5(5)
C(4)-C(5)-H(5)	119.8
C(6)-C(5)-H(5)	119.8
N(1)-C(6)-C(5)	119.5(5)
N(1)-C(6)-C(1)	121.8(4)
C(5)-C(6)-C(1)	118.7(4)
N(1)-C(7)-C(8)	122.0(5)
N(1)-C(7)-C(7)#1	116.9(6)
C(8)-C(7)-C(7)#1	121.1(5)
N(2)-C(8)-C(7)	120.4(5)
N(2)-C(8)-O(1)	118.0(5)
C(7)-C(8)-O(1)	121.5(5)
C(11)-O(2)-H(2A)	109.5
C(11)-C(10)-O(1)	105.7(9)
C(11)-C(10)-H(10A)	110.6
O(1)-C(10)-H(10A)	110.6
C(11)-C(10)-H(10B)	110.6
O(1)-C(10)-H(10B)	110.6
H(10A)-C(10)-H(10B)	108.7
C(10)-C(11)-O(2)	109.1(9)
C(10)-C(11)-H(11A)	109.9
O(2)-C(11)-H(11A)	109.9
C(10)-C(11)-H(11B)	109.9
O(2)-C(11)-H(11B)	109.9
H(11A)-C(11)-H(11B)	108.3
C(11')-O(2')-H(2')	109.5
O(1)-C(10')-C(11')	105.5(16)
O(1)-C(10')-H(10C)	110.6
C(11')-C(10')-H(10C)	110.7

O(1)-C(10')-H(10D)	110.6
C(11')-C(10')-H(10D)	110.6
H(10C)-C(10')-H(10D)	108.8
O(2')-C(11')-C(10')	100.8(15)
O(2')-C(11')-H(11C)	111.6
C(10')-C(11')-H(11C)	111.6
O(2')-C(11')-H(11D)	111.6
C(10')-C(11')-H(11D)	111.6
H(11C)-C(11')-H(11D)	109.4

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,y,-z+1/2

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for mo_ddz20083_0m. The anisotropic
displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	67(4)	75(4)	47(3)	-11(3)	8(3)	2(3)
N(2)	72(3)	63(3)	88(3)	-7(2)	14(2)	2(2)
N(1)	67(3)	63(3)	84(3)	18(2)	5(2)	-2(2)
C(1)	58(3)	56(3)	89(4)	11(2)	18(2)	-2(2)
C(2)	76(3)	66(3)	79(3)	1(3)	14(3)	-6(3)
C(3)	70(3)	71(4)	78(3)	5(3)	4(2)	-3(2)
C(4)	67(3)	76(4)	77(3)	8(3)	17(3)	6(2)
C(5)	69(3)	66(3)	66(3)	9(2)	13(2)	2(2)
C(6)	65(3)	51(3)	69(3)	13(2)	9(2)	-4(2)
C(7)	62(3)	46(3)	89(4)	5(2)	7(2)	-1(2)
C(8)	64(3)	54(3)	102(4)	3(3)	2(3)	-3(2)
C(9)	72(3)	63(3)	88(3)	-7(2)	14(2)	2(2)
O(2)	58(3)	27(4)	22(3)	13(3)	7(2)	19(3)
C(10)	61(3)	28(4)	25(3)	15(3)	6(2)	20(3)
C(11)	60(3)	27(4)	24(3)	15(3)	6(2)	20(3)
O(2')	60(3)	29(4)	24(3)	13(3)	7(2)	19(3)
C(10')	61(3)	28(4)	25(3)	15(3)	6(2)	20(3)
C(11')	61(3)	28(4)	25(3)	14(3)	6(2)	20(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10 ^{3})
for mo_ddz20083_0m.

	x	y	z	U(eq)
H(2)	6858	1076	4720	88
H(3)	7698	-1290	4376	89
H(4)	7671	-3225	3212	88
H(5)	6798	-3128	2409	80
H(9)	5788	2646	4413	90
H(2A)	4149	10924	3999	53
H(10A)	4809	3721	4748	46
H(10B)	4953	7256	4380	46
H(11A)	3988	6802	4727	45
H(11B)	3826	4293	4065	45
H(2')	4259	8240	4005	57
H(10C)	4975	6062	4586	46
H(10D)	4908	2007	4719	46
H(11C)	3862	2437	4346	45
H(11D)	4055	5170	4971	45

Table 6. Torsion angles [°] for mo_ddz20083_0m.

C(8)-N(2)-C(1)-C(2)	179.4(4)
C(8)-N(2)-C(1)-C(6)	0.8(7)
N(2)-C(1)-C(2)-C(3)	-179.6(5)
C(6)-C(1)-C(2)-C(3)	-0.9(7)
C(1)-C(2)-C(3)-C(4)	1.2(8)
C(2)-C(3)-C(4)-C(5)	-1.8(8)
C(3)-C(4)-C(5)-C(6)	2.0(7)
C(7)-N(1)-C(6)-C(5)	178.7(4)
C(7)-N(1)-C(6)-C(1)	0.0(6)
C(4)-C(5)-C(6)-N(1)	179.5(4)
C(4)-C(5)-C(6)-C(1)	-1.7(7)
N(2)-C(1)-C(6)-N(1)	-1.5(7)

C(2)-C(1)-C(6)-N(1)	179.9(4)
N(2)-C(1)-C(6)-C(5)	179.8(4)
C(2)-C(1)-C(6)-C(5)	1.1(7)
C(6)-N(1)-C(7)-C(8)	2.1(7)
C(6)-N(1)-C(7)-C(7)#1	-178.6(3)
C(1)-N(2)-C(8)-C(7)	1.3(7)
C(1)-N(2)-C(8)-O(1)	178.2(5)
N(1)-C(7)-C(8)-N(2)	-2.8(8)
C(7)#1-C(7)-C(8)-N(2)	177.9(4)
N(1)-C(7)-C(8)-O(1)	-179.7(5)
C(7)#1-C(7)-C(8)-O(1)	1.1(7)
C(10')-O(1)-C(8)-N(2)	-9.7(12)
C(10)-O(1)-C(8)-N(2)	12.4(11)
C(10')-O(1)-C(8)-C(7)	167.2(10)
C(10)-O(1)-C(8)-C(7)	-170.6(7)
C(10')-O(1)-C(10)-C(11)	-113(3)
C(8)-O(1)-C(10)-C(11)	175.5(7)
O(1)-C(10)-C(11)-O(2)	-78.3(11)
C(10)-O(1)-C(10')-C(11')	73(3)
C(8)-O(1)-C(10')-C(11')	-166.5(13)
O(1)-C(10')-C(11')-O(2')	-57(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1/2

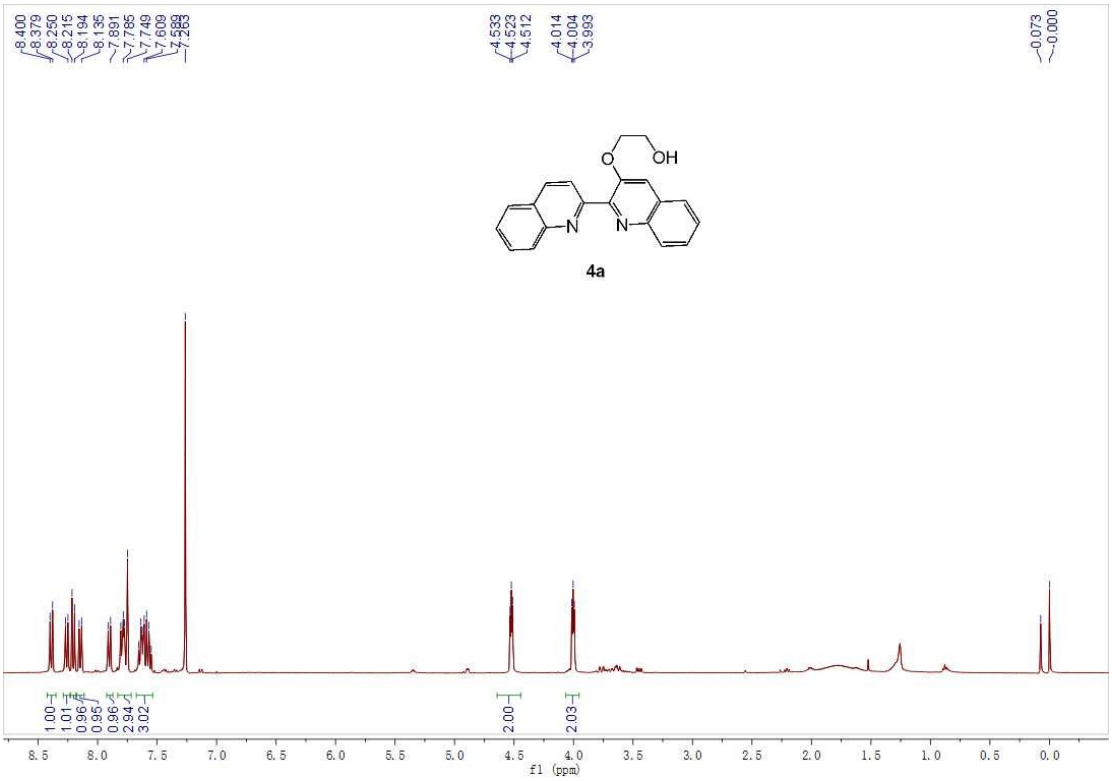
Table 7. Hydrogen bonds for mo_ddz20083_0m [Å and °].

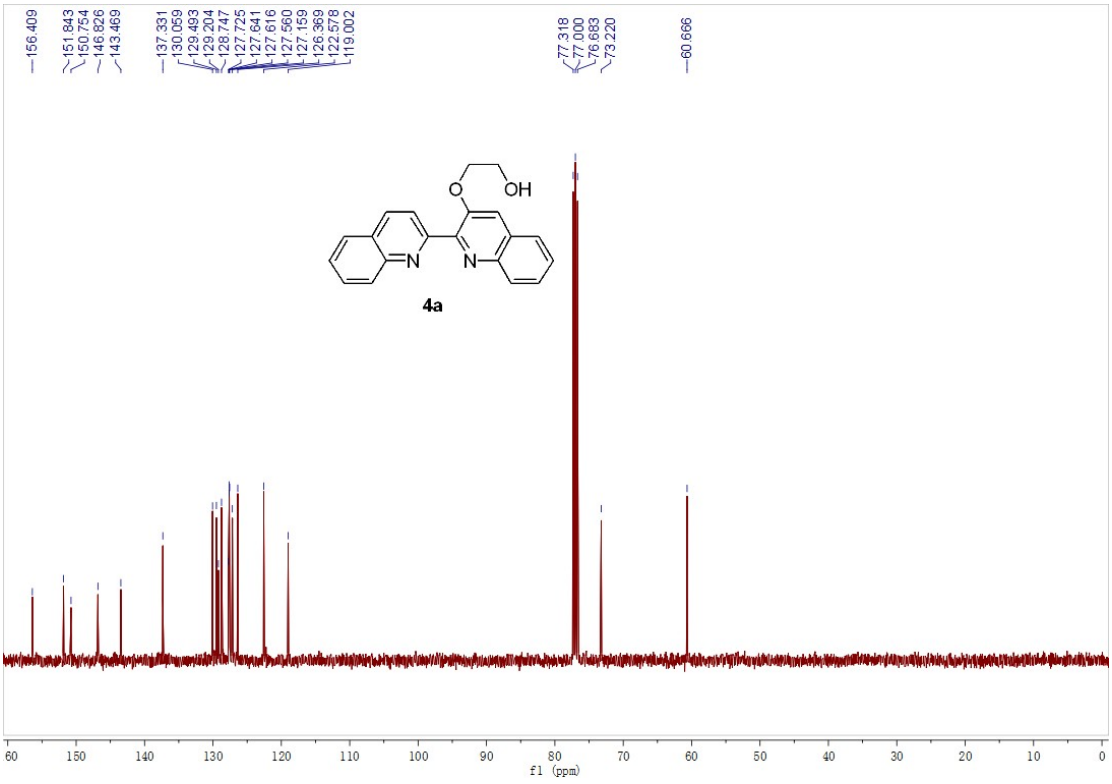
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(11)-H(11A)...N(2)#2	0.97	2.53	3.435(11)	156.0
O(2)-H(2A)...O(1)#3	0.82	1.81	2.369(11)	124.6
C(11)-H(11A)...N(2)#2	0.97	2.53	3.435(11)	156.0
O(2)-H(2A)...O(1)#3	0.82	1.81	2.369(11)	124.6

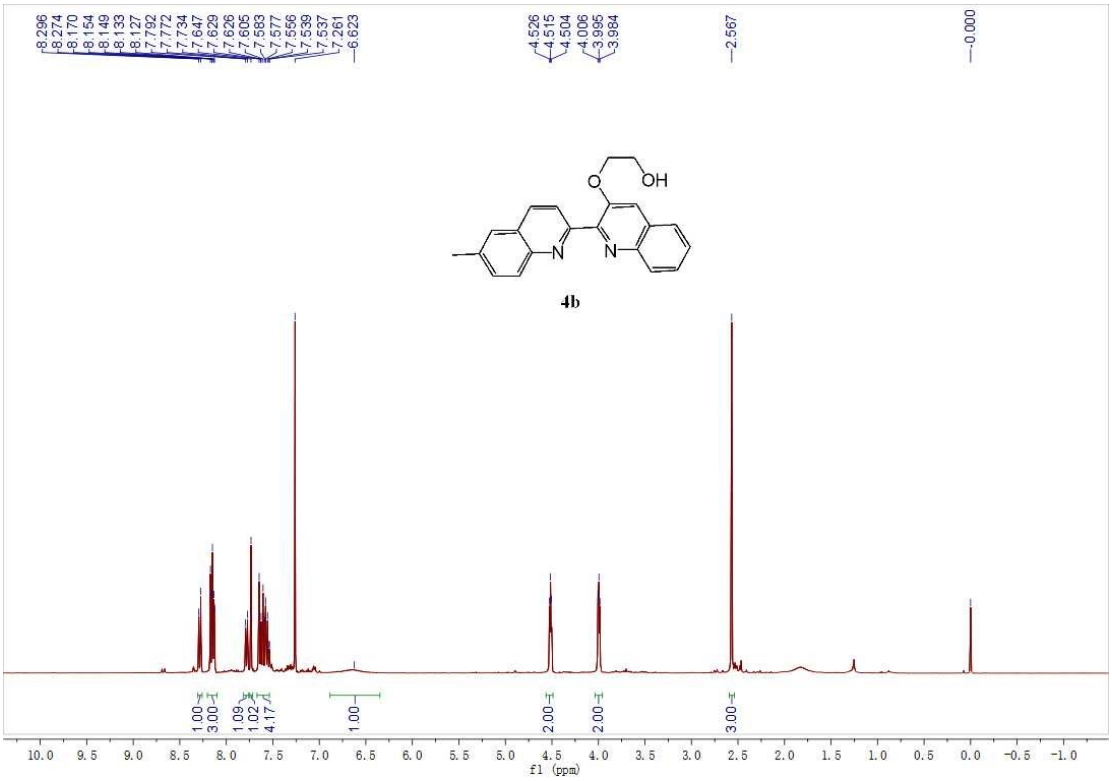
Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+1/2 #2 -x+1,-y+1,-z+1 #3 x,y+1,z

^{1}H NMR and ^{13}C NMR spectra



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4b
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3.00
1.09
1.02
4.17
1.00
2.00
2.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
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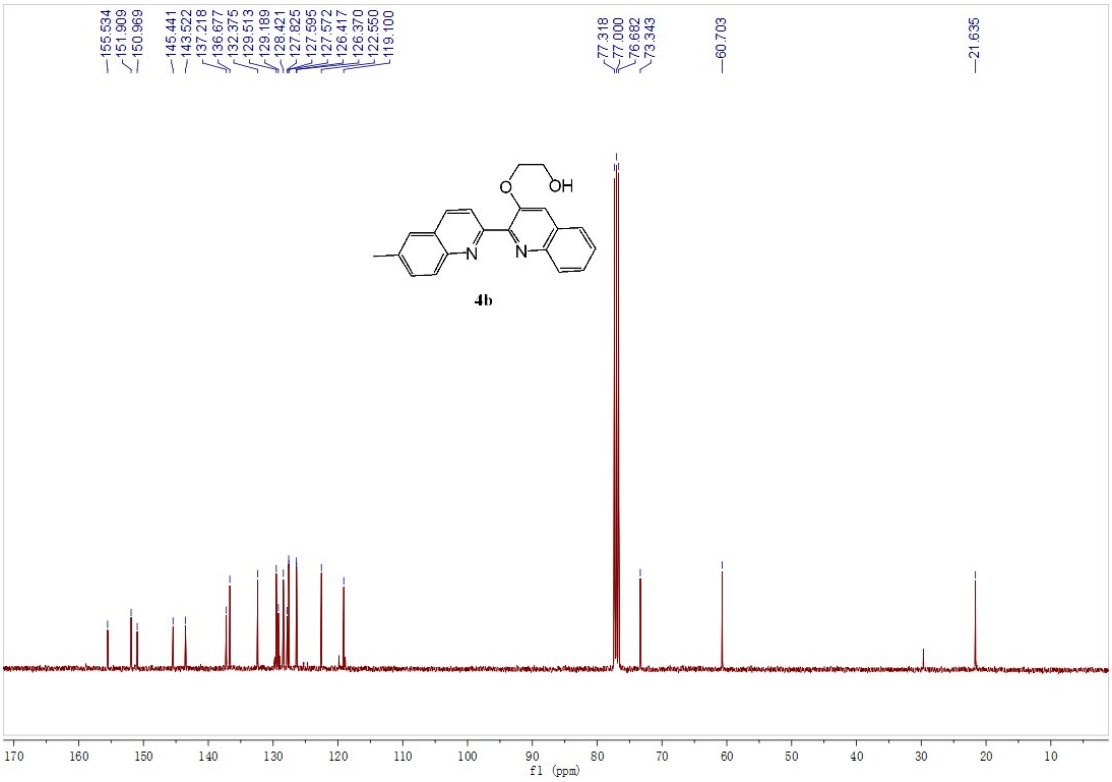
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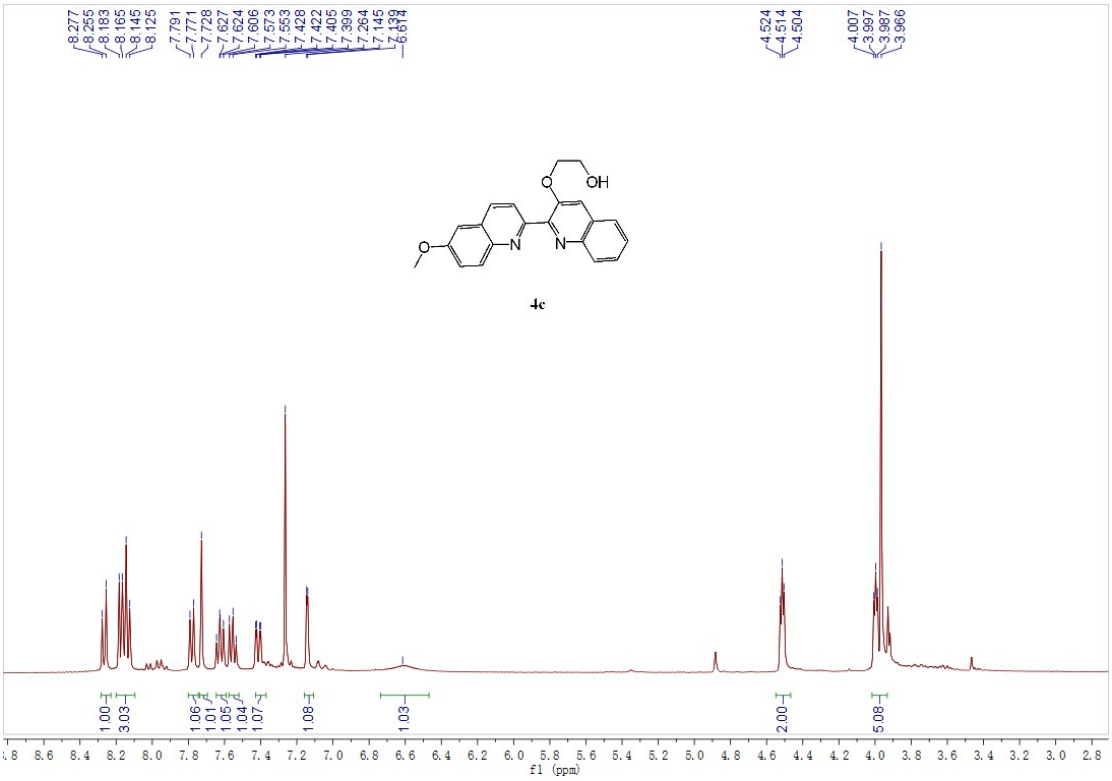
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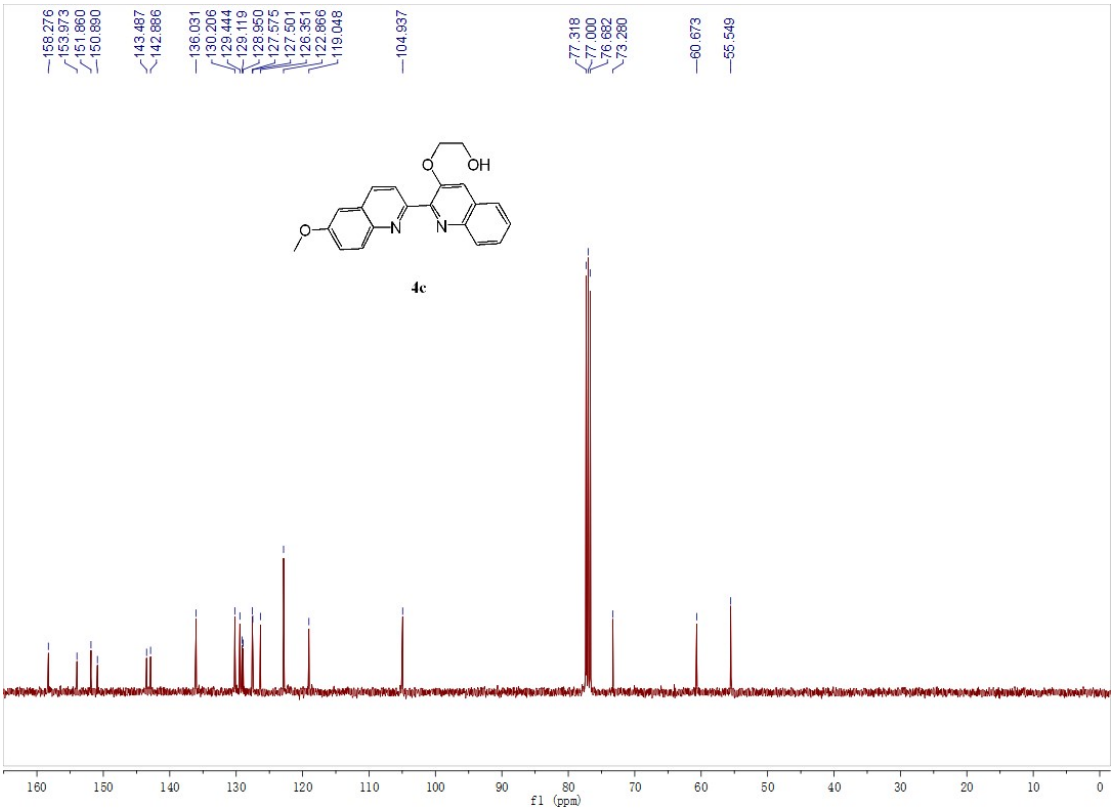
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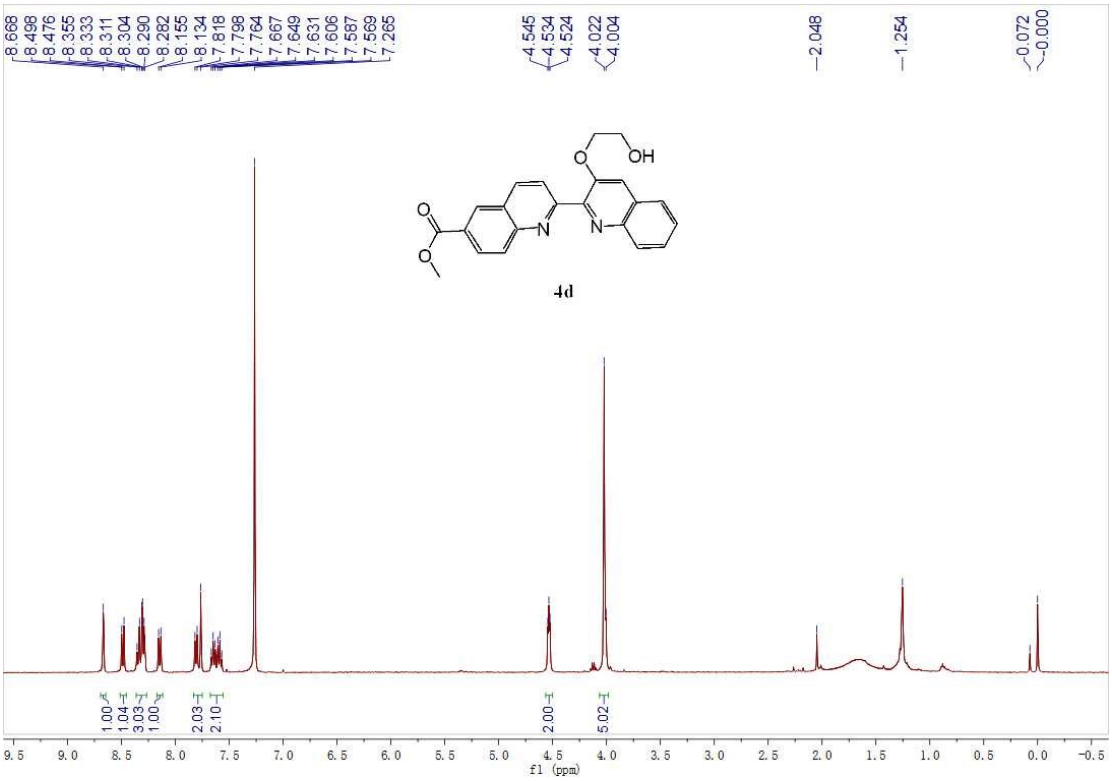
f1 (ppm)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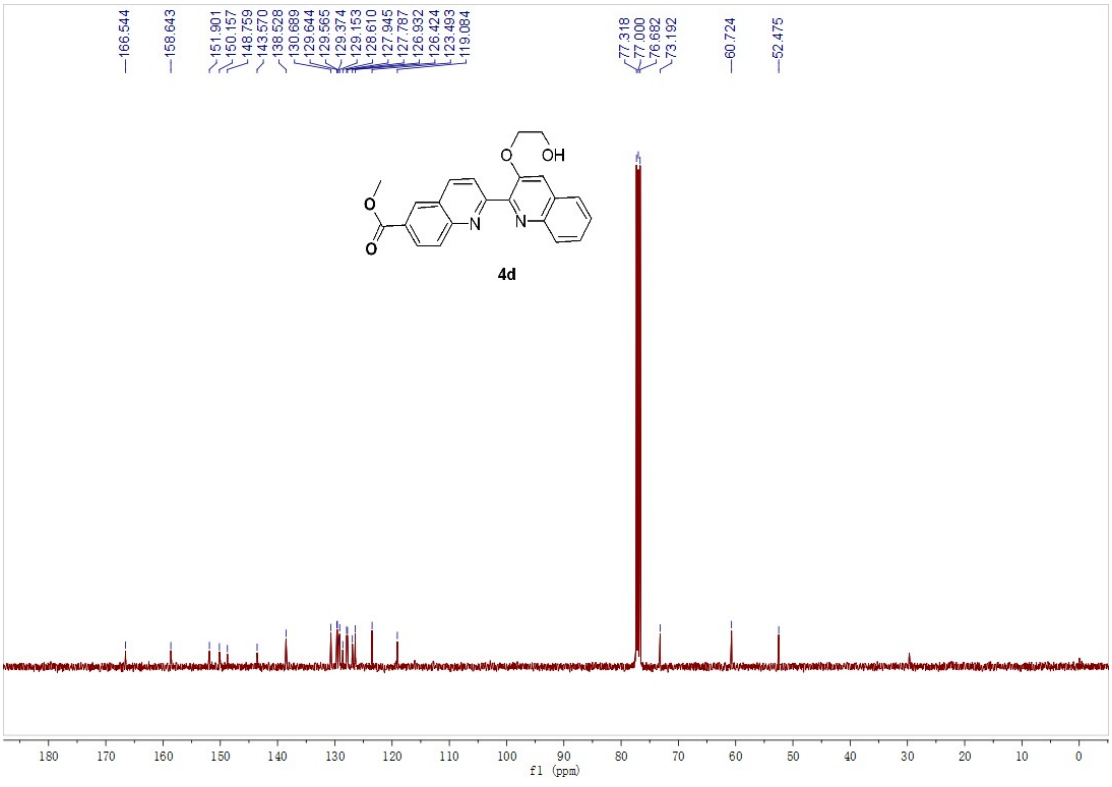
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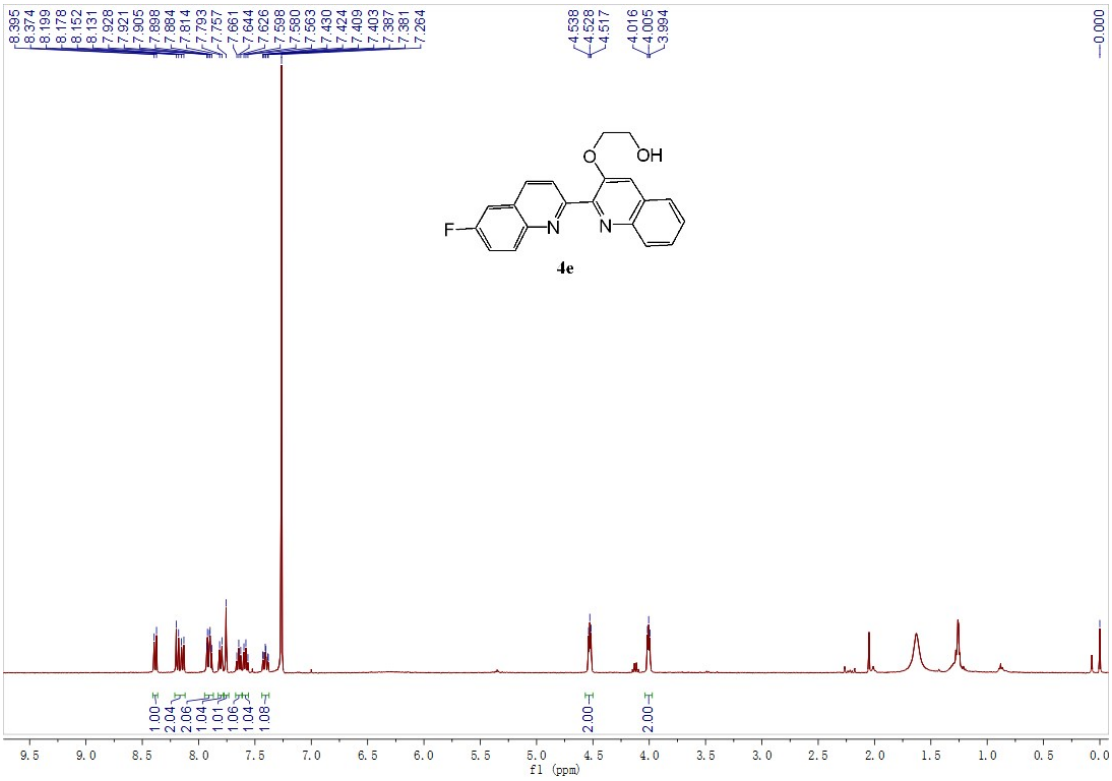
f1 (ppm)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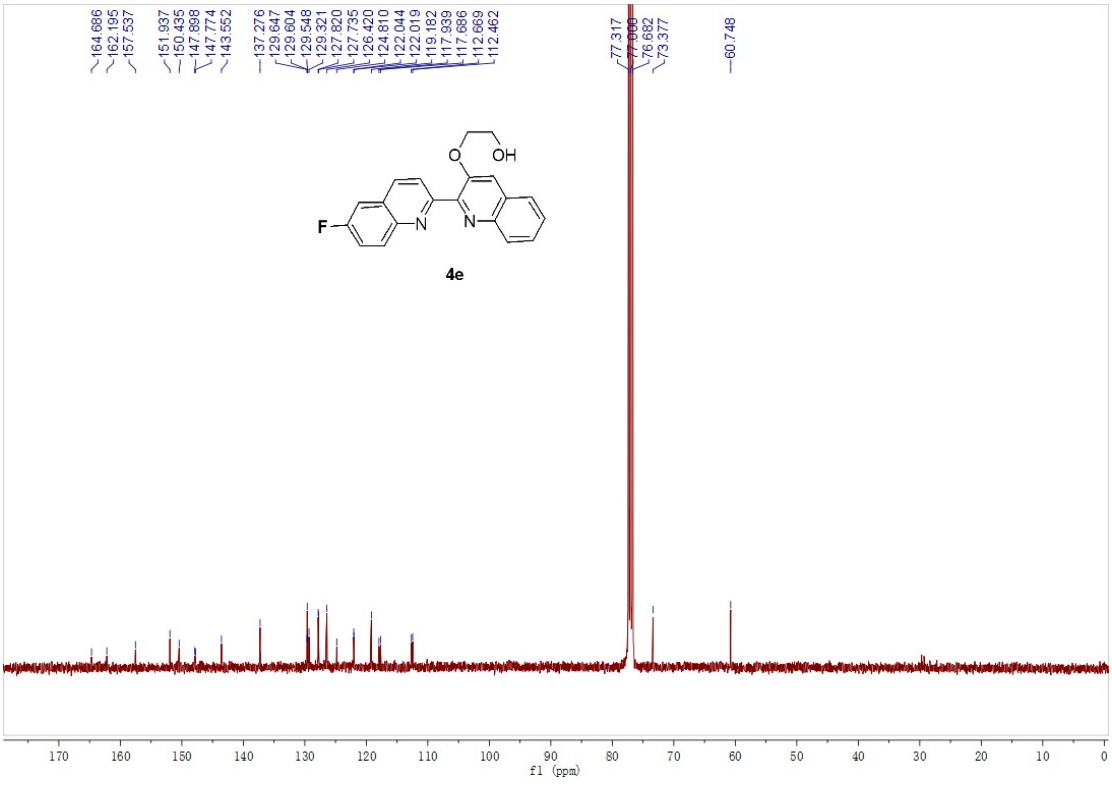
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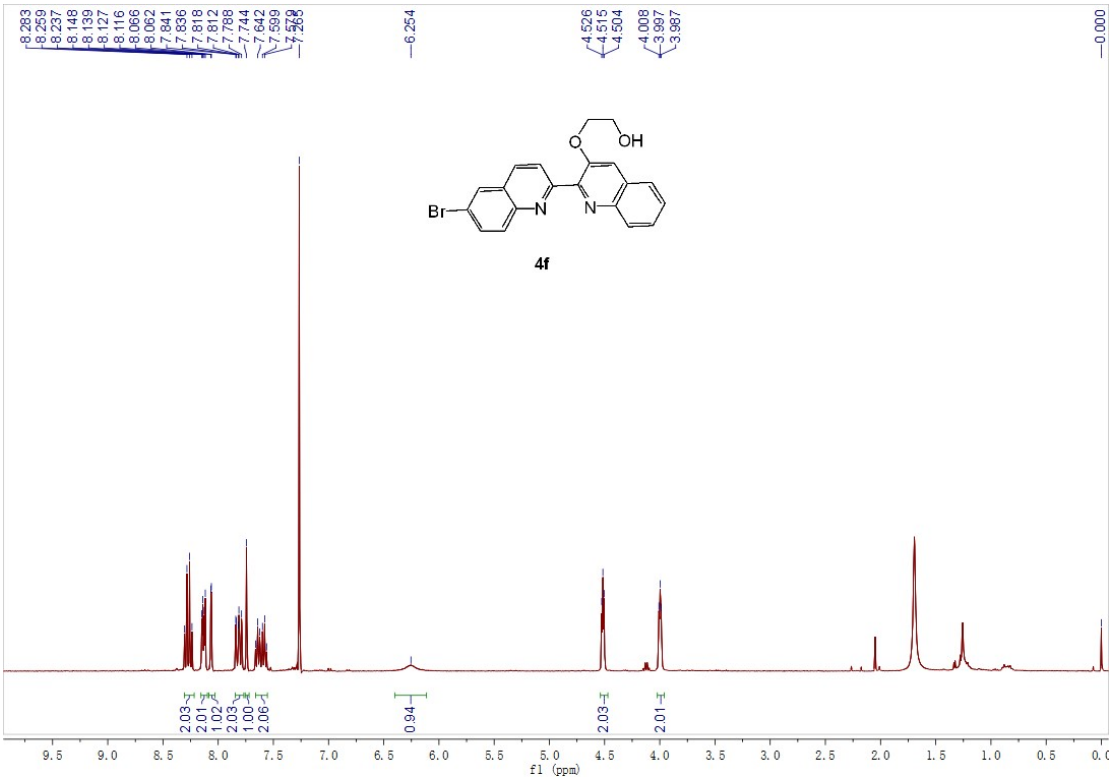
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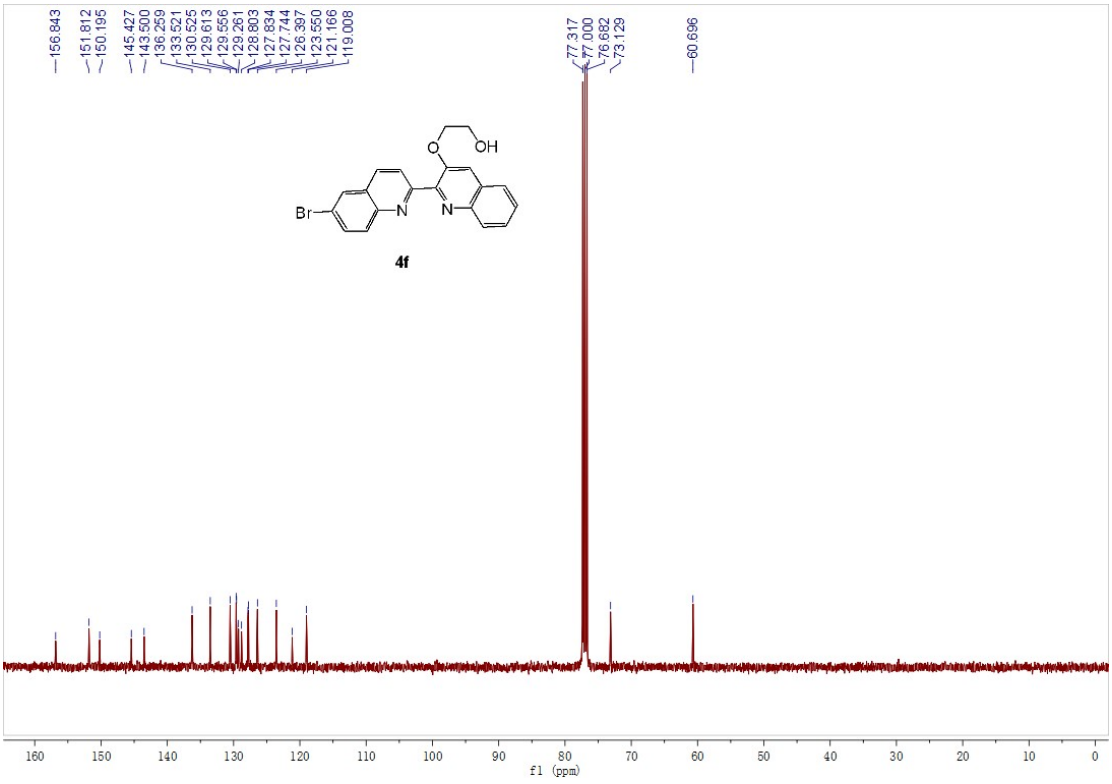
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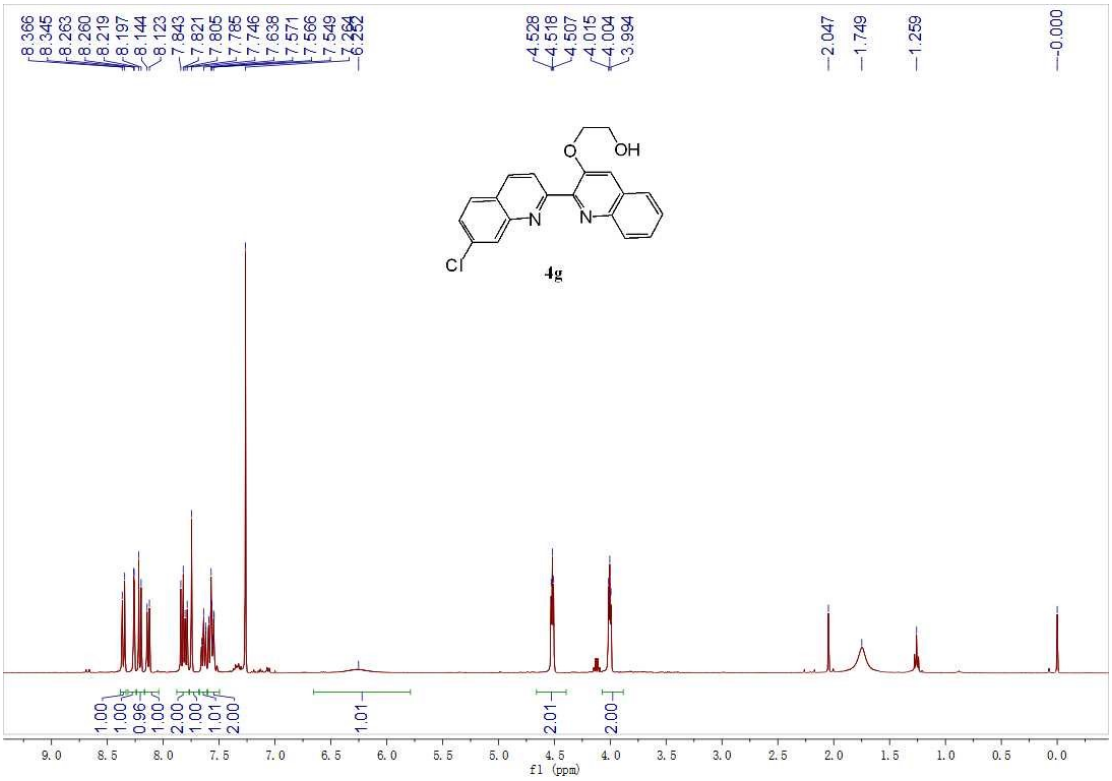
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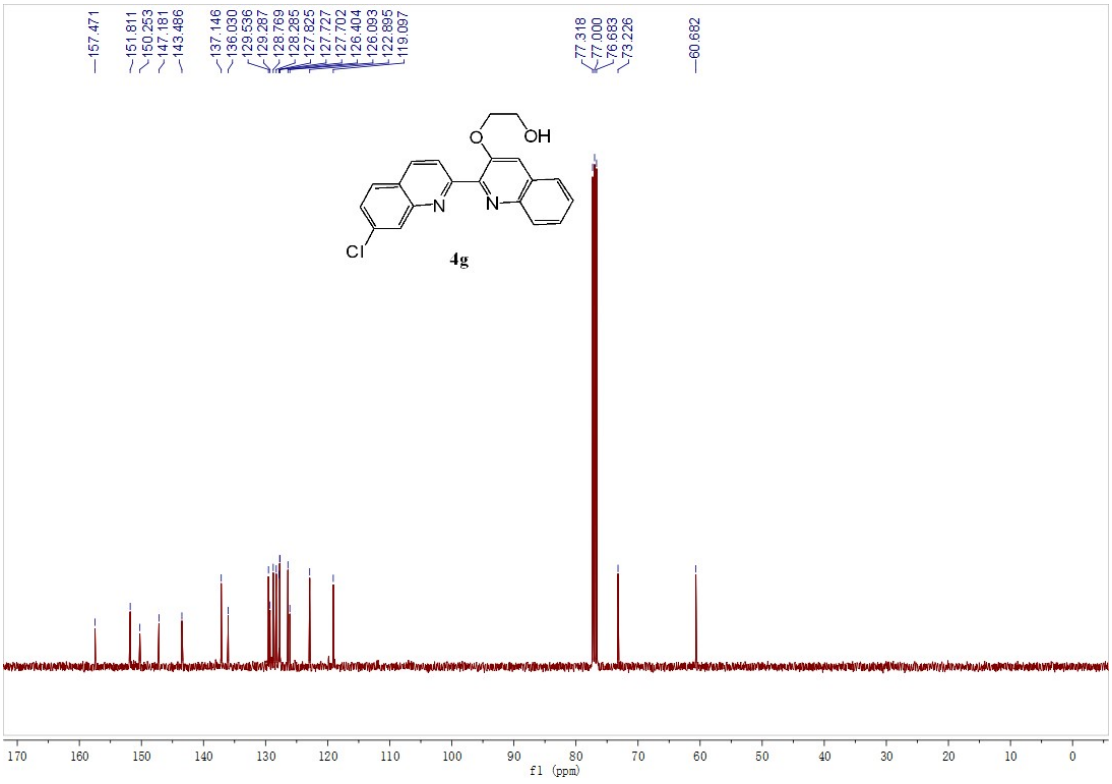
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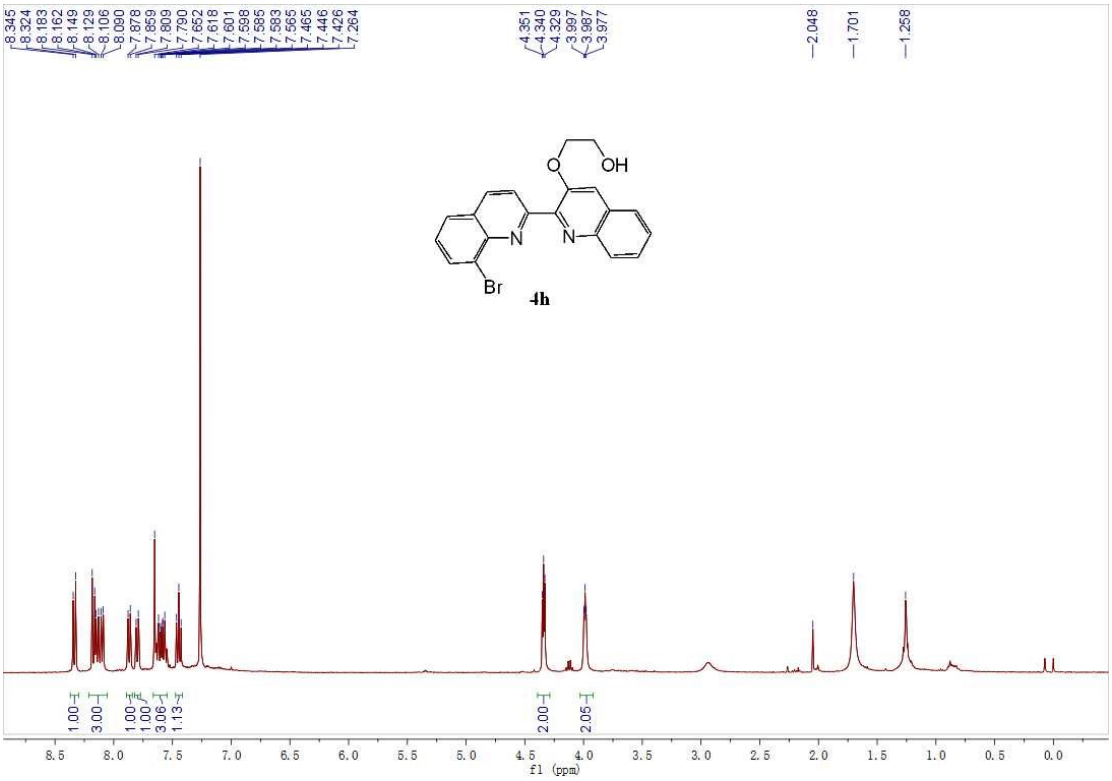
Cl
4g
f1 (ppm)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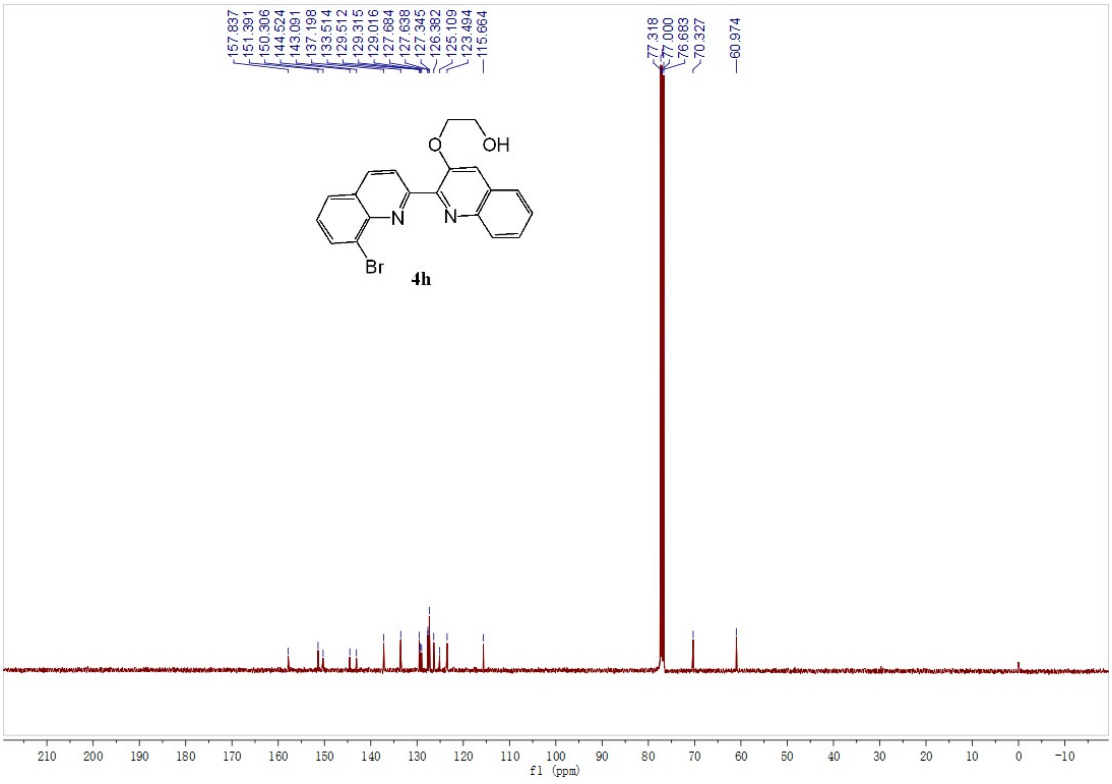
f1 (ppm)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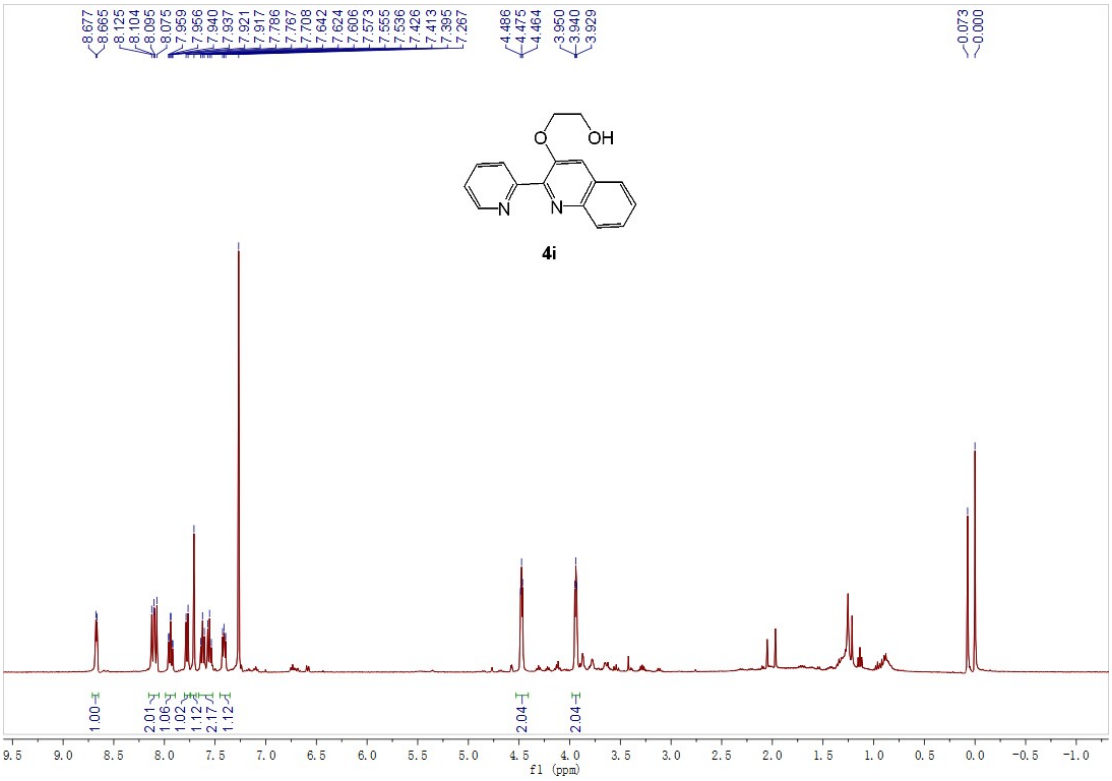
O
OH
N
N
Br
4h
f1 (ppm)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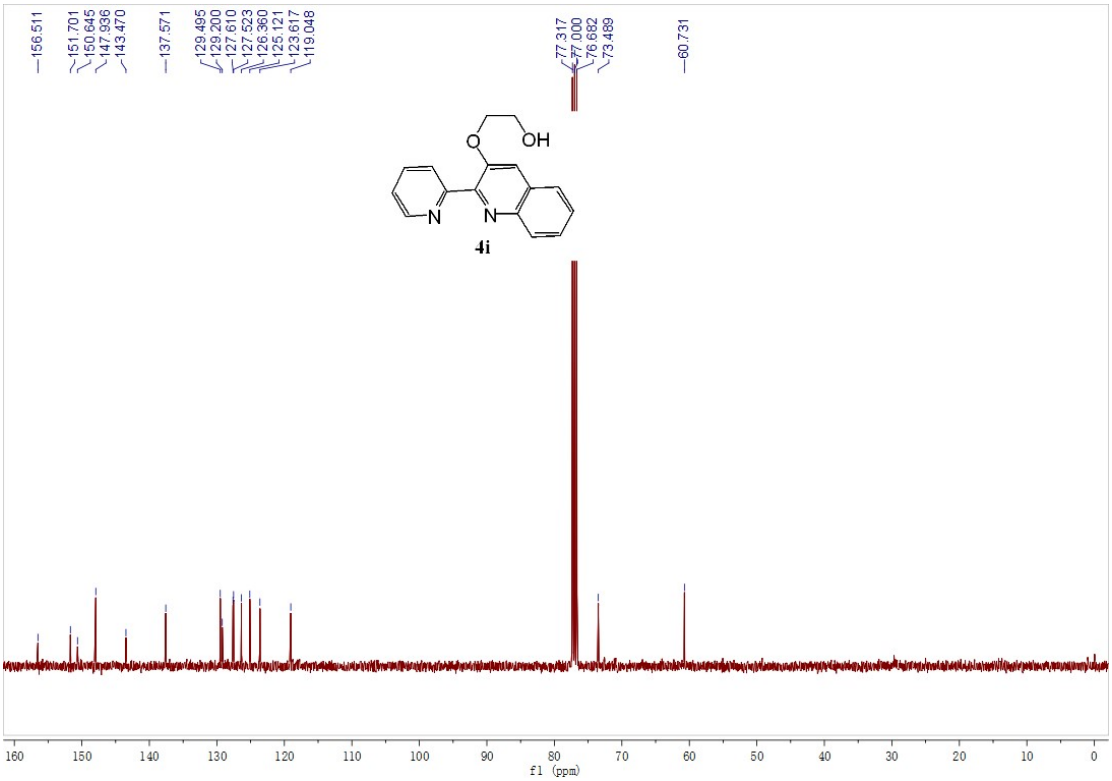
8.665
8.125
8.104
8.095
8.075
7.959
7.956
7.940
7.937
7.921
7.917
7.786
7.767
7.708
7.642
7.624
7.606
7.573
7.555
7.536
7.426
7.413
7.395
7.267
4.486
4.475
4.464
3.950
3.940
3.929
0.073
0.000
O
OH
N
N
4i
1.00
2.01
1.06
1.02
1.12
2.17
1.12
2.04
2.04
f1 (ppm)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150.645
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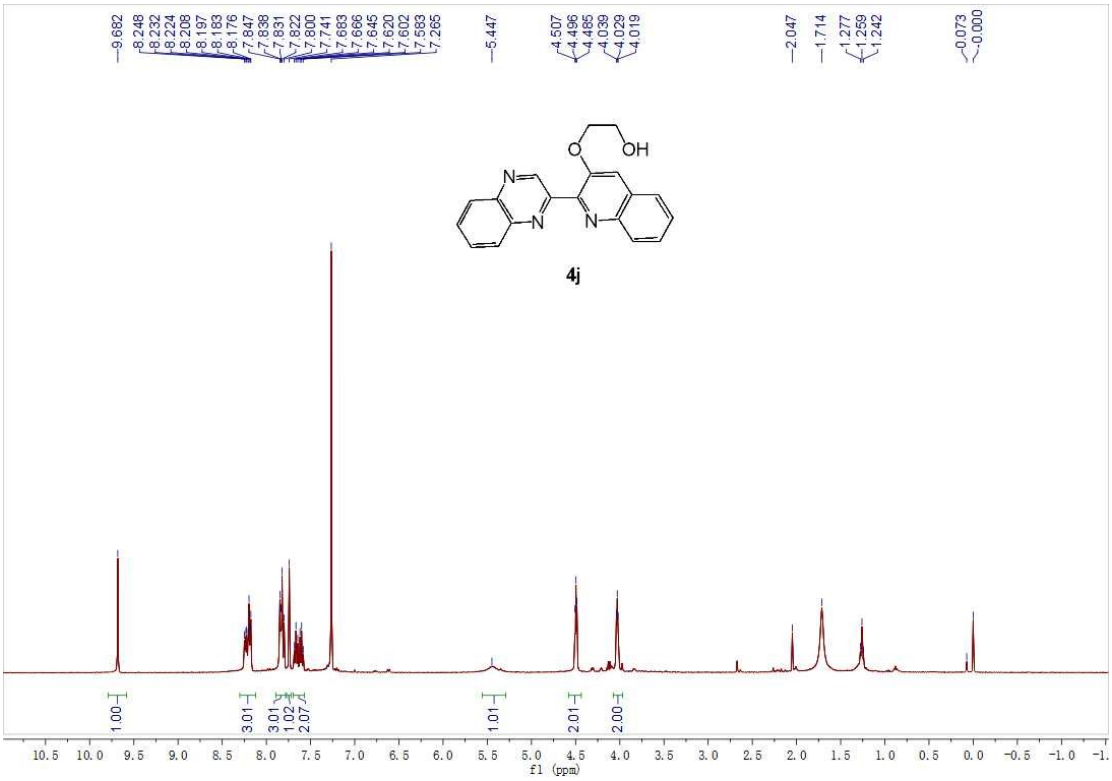
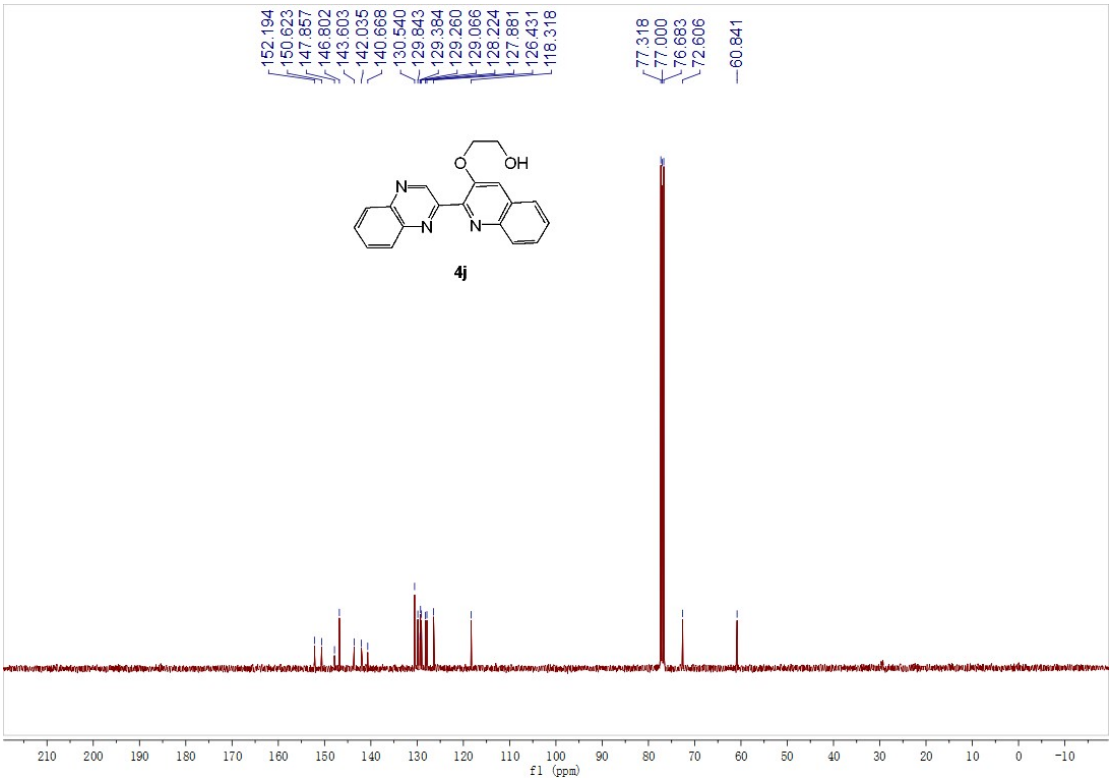
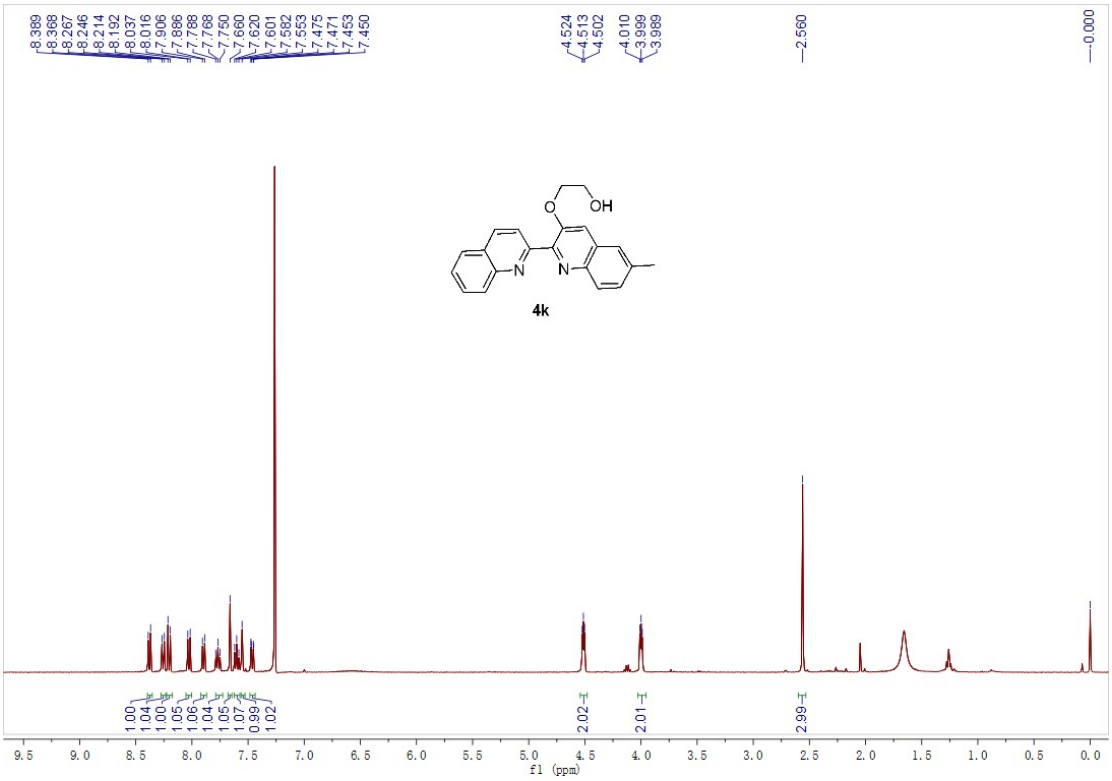
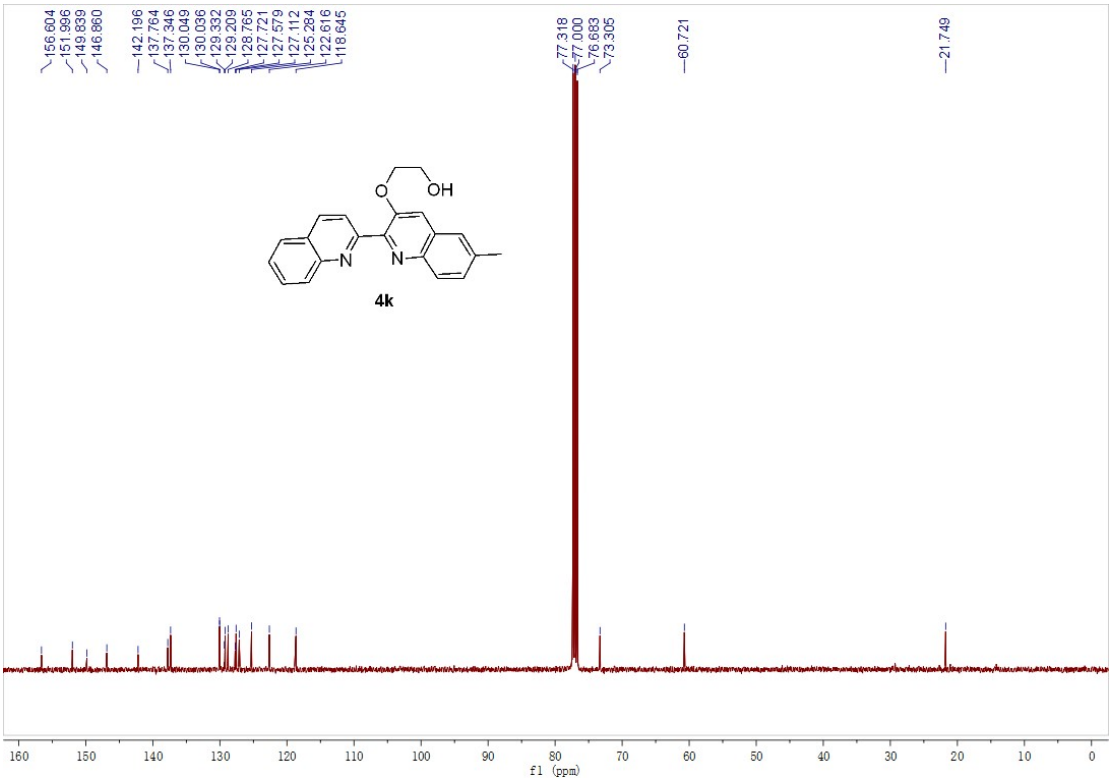
143.470
137.571
129.495
129.200
127.610
127.523
126.360
125.121
123.617
119.048
77.317
77.000
76.682
73.489
60.731
O
OH
N
N
4i
f1 (ppm)


9.682
8.248
8.232
8.224
8.208
8.197
8.183
8.176
7.847
7.838
7.831
7.822
7.800
7.741
7.683
7.666
7.645
7.620
7.602
7.583
7.265
5.447
4.507
4.496
4.485
4.039
4.029
4.019
2.047
1.714
1.277
1.259
1.242
0.073
0.000
OH
N
N
N
4j
1.00
3.01
3.01
1.02
2.07
1.01
2.01
2.00
f1 (ppm)


152.194
150.623
147.857
146.802
143.603
142.035
140.668
130.540
129.843
129.384
129.260
129.066
128.224
127.881
126.431
118.318
77.318
77.000
76.683
72.606
60.841
OH
N
N
N
4j
f1 (ppm)


8.389
8.368
8.267
8.246
8.214
8.192
8.037
8.016
7.906
7.886
7.788
7.768
7.750
7.660
7.620
7.601
7.582
7.553
7.475
7.471
7.453
7.450
4.524
4.513
4.502
4.010
3.999
3.989
2.560
0.000
O
OH
N
N
4k
1.00
1.04
1.00
1.05
1.06
1.04
1.05
1.07
0.99
1.02
2.02
2.01
2.99
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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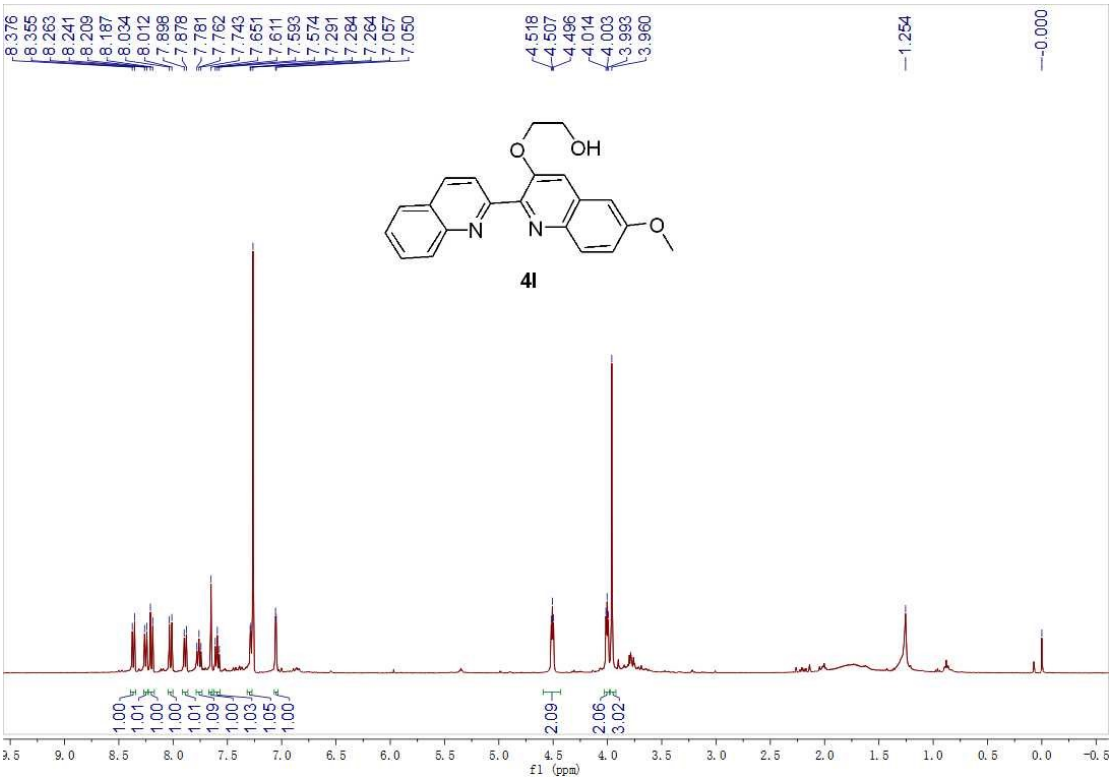
151.996
149.839
146.860
142.196
137.764
137.346
130.049
130.036
129.332
129.209
128.765
127.721
127.579
127.112
125.284
122.616
118.645
77.318
77.000
76.683
73.305
60.721
21.749
O
OH
N
N
4k
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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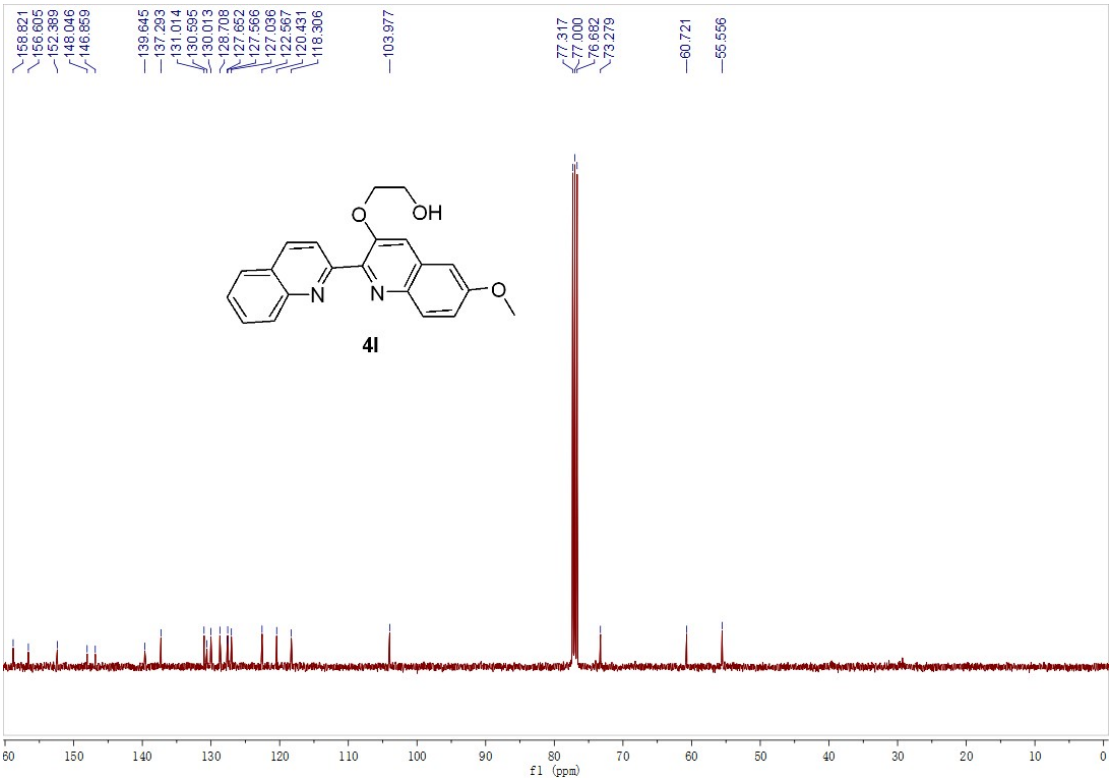
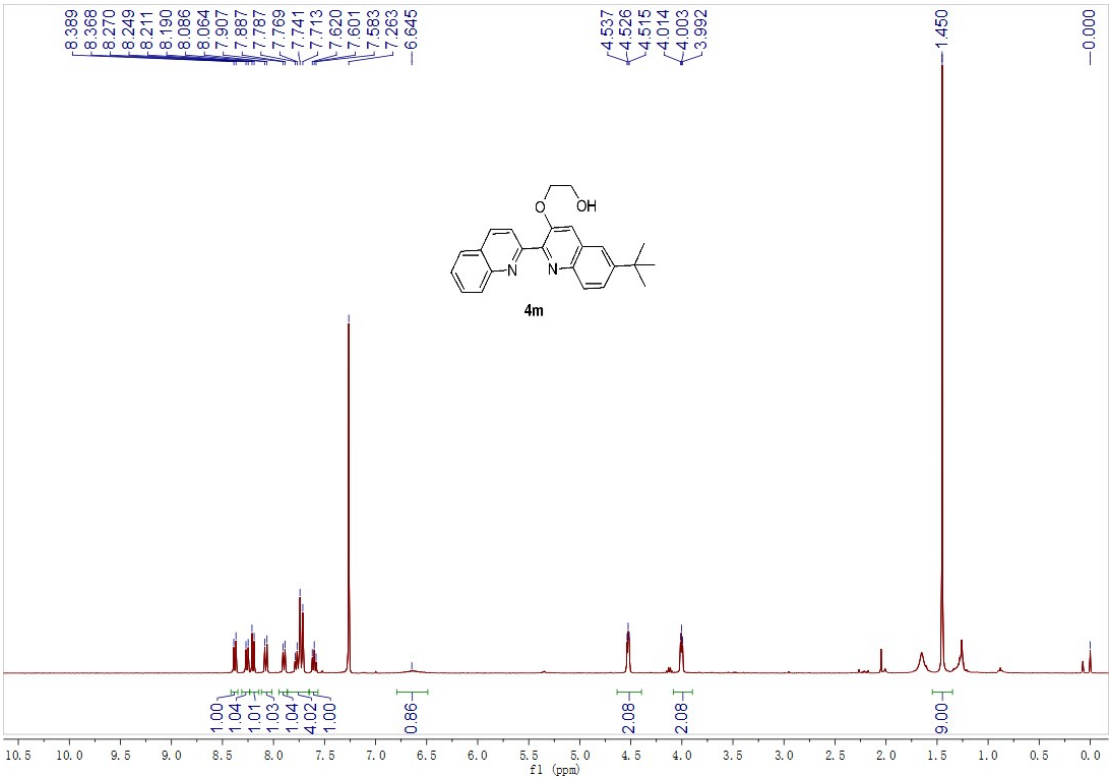
8.355
8.263
8.241
8.209
8.187
8.034
8.012
7.898
7.878
7.781
7.762
7.743
7.651
7.611
7.593
7.574
7.291
7.284
7.264
7.057
7.050
4.518
4.507
4.496
4.014
4.003
3.993
3.960
1.254
0.000
OH
O
N
N
O
4l
1.00
1.01
1.00
1.00
1.01
1.09
1.00
1.03
1.05
1.00
2.09
2.06
3.02
f1 (ppm)


158.821
156.605
152.389
148.046
146.859
139.645
137.293
131.014
130.595
130.013
128.708
127.652
127.566
127.036
122.567
120.431
118.306
103.977
77.317
77.000
76.682
73.279
60.721
55.556
OH
O
N
N
O
4l
f1 (ppm)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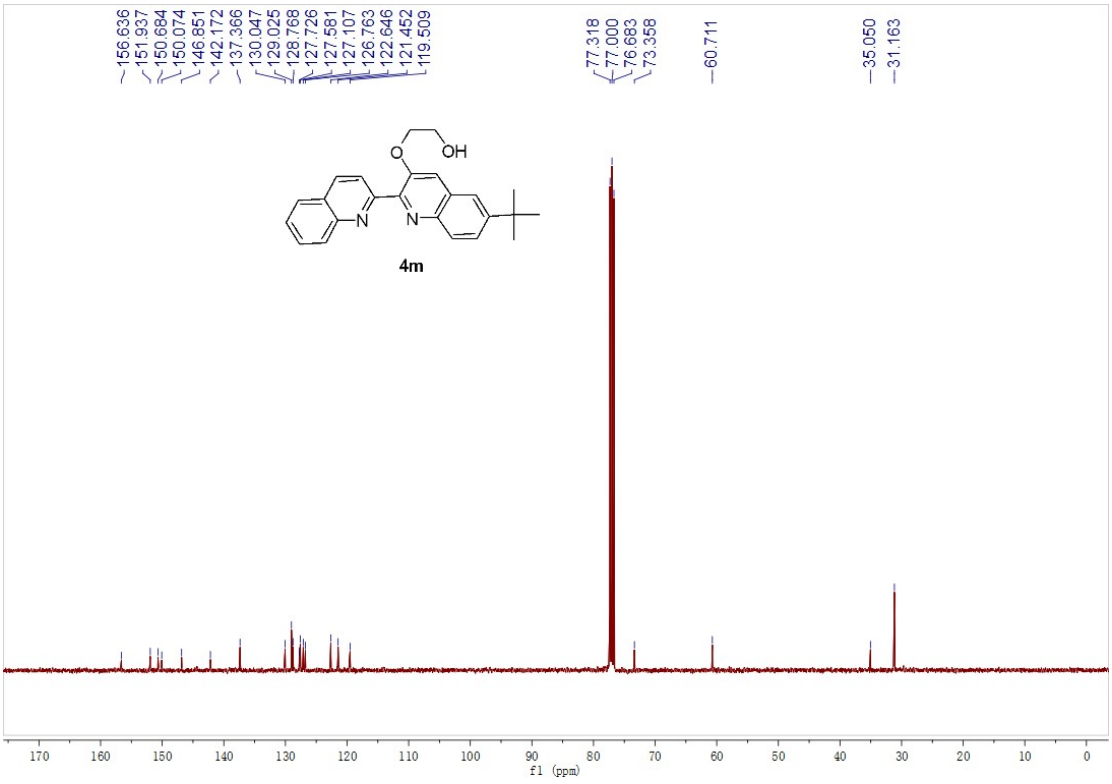
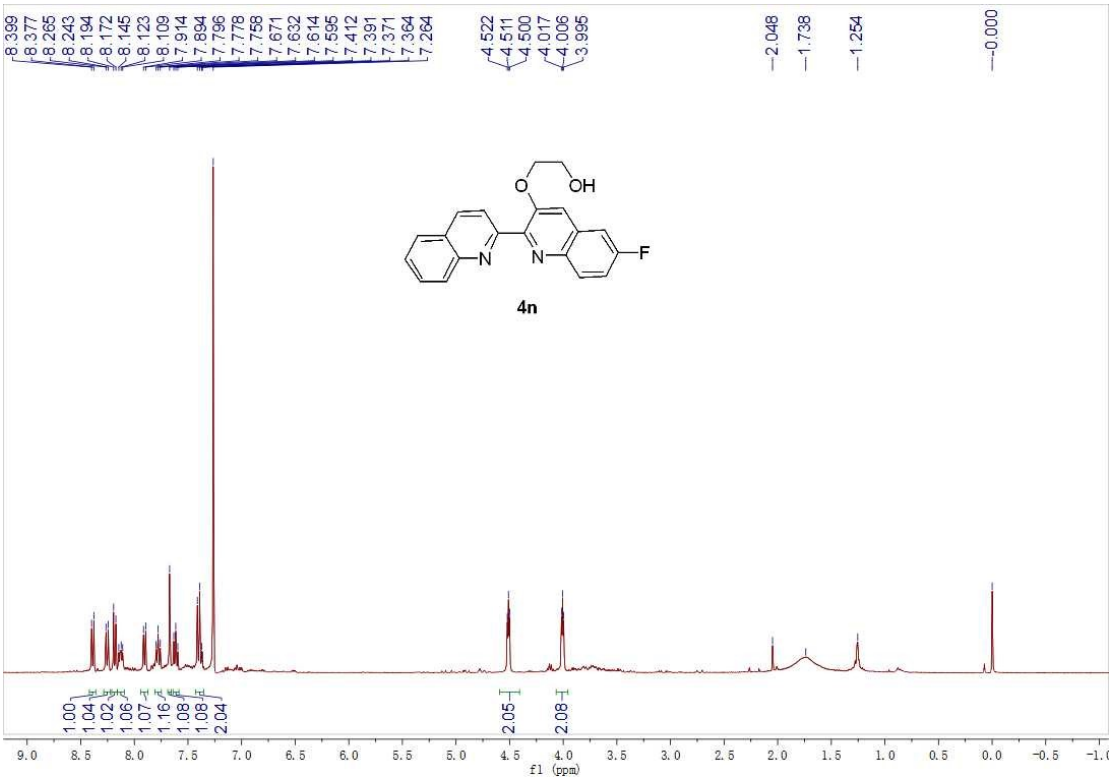
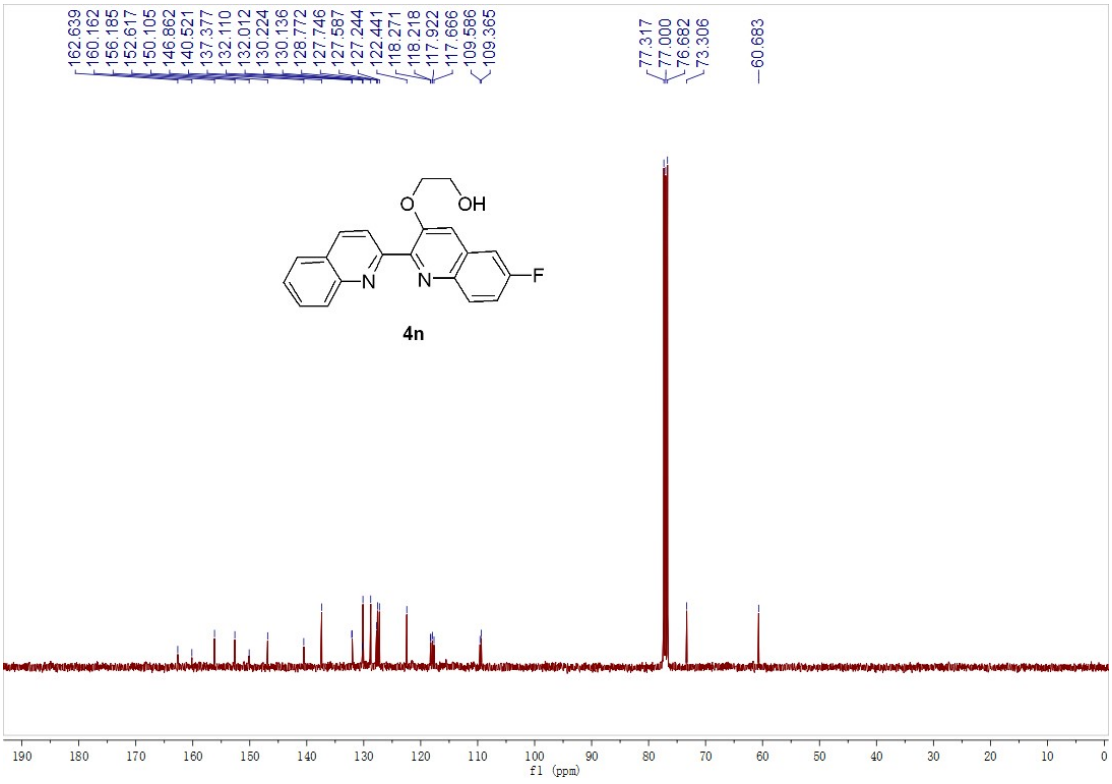
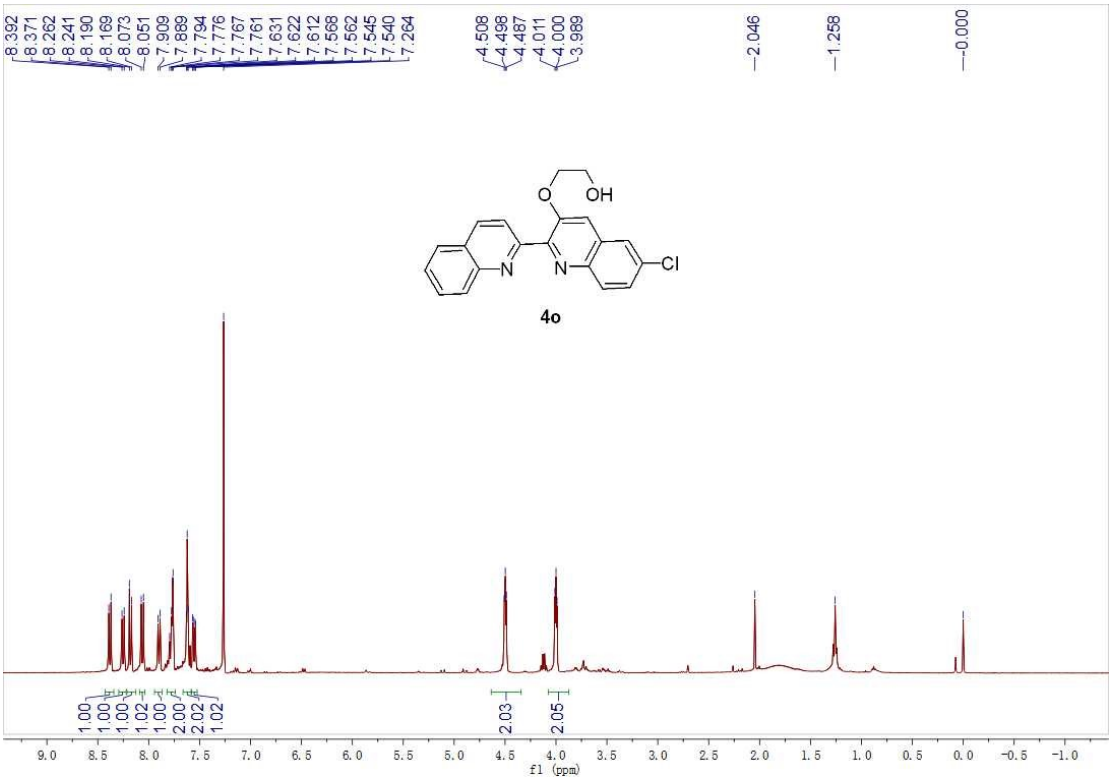
8.368
8.270
8.249
8.211
8.190
8.086
8.064
7.907
7.887
7.787
7.769
7.741
7.713
7.620
7.601
7.583
7.263
6.645
4.537
4.526
4.515
4.014
4.003
3.992
1.450
0.000
OH
N
N
4m
1.00
1.04
1.01
1.03
1.04
4.02
1.00
0.86
2.08
2.08
9.00
f1 (ppm)


156.636
151.937
150.684
150.074
146.851
142.172
137.366
130.047
129.025
128.768
127.726
127.581
127.107
126.763
122.646
121.452
119.509
77.318
77.000
76.683
73.358
60.711
35.050
31.163
OH
N
N
4m
f1 (ppm)


8.399
8.377
8.265
8.243
8.194
8.172
8.145
8.123
8.109
7.914
7.894
7.796
7.778
7.758
7.671
7.632
7.614
7.595
7.412
7.391
7.371
7.364
7.264
4.522
4.511
4.500
4.017
4.006
3.995
2.048
1.738
1.254
0.000
O
OH
N
N
F
4n
1.00
1.04
1.02
1.06
1.07
1.16
1.08
1.08
2.04
2.05
2.08
f1 (ppm)


162.639
160.162
156.185
152.617
150.105
146.862
140.521
137.377
132.110
132.012
130.224
130.136
128.772
127.746
127.587
127.244
122.441
118.271
118.218
117.922
117.666
109.586
109.365
77.317
77.000
76.682
73.306
60.683
O
OH
N
N
F
4n
f1 (ppm)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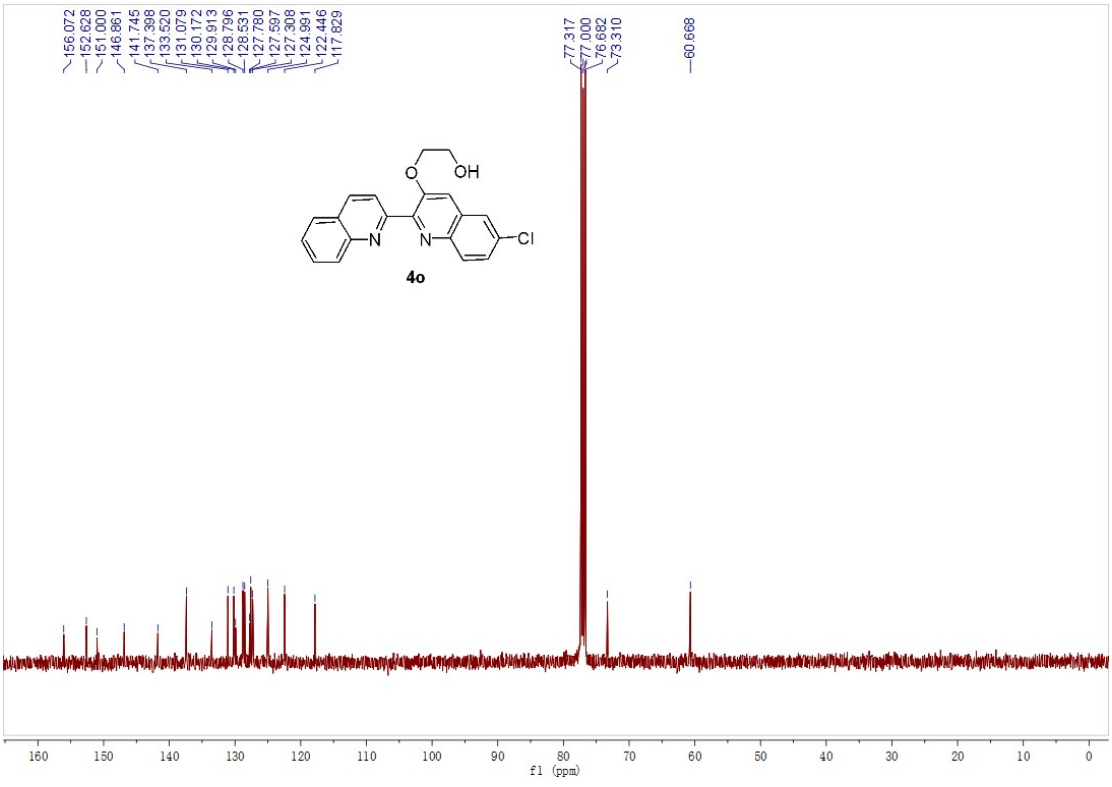
8.169
8.073
8.051
7.909
7.889
7.794
7.776
7.767
7.761
7.631
7.622
7.612
7.568
7.562
7.545
7.540
7.264
4.508
4.498
4.487
4.011
4.000
3.989
2.046
1.258
0.000
O
OH
N
N
Cl
4o
1.00
1.00
1.00
1.02
1.00
2.00
2.02
1.02
2.03
2.05
f1 (ppm)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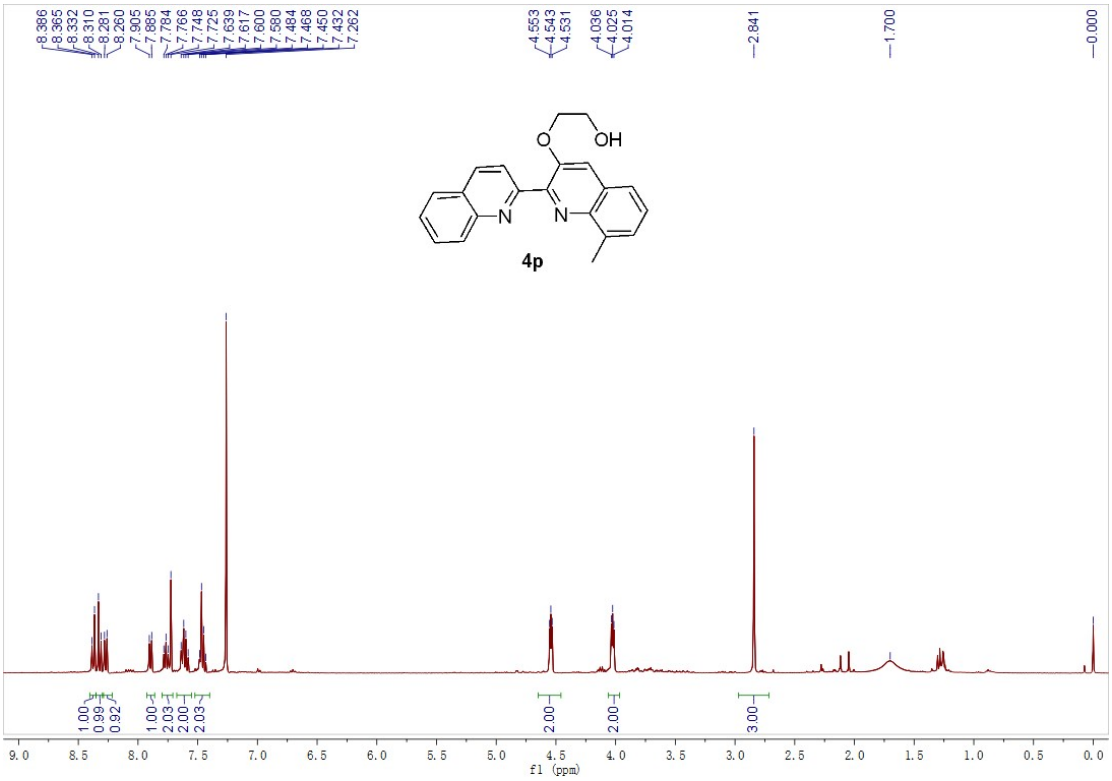
152.628
151.000
146.861
141.745
137.398
133.520
131.079
130.172
129.913
128.796
128.531
127.780
127.597
127.308
124.981
122.446
117.829
77.317
77.000
76.682
73.310
60.668
O
OH
N
N
Cl
4o
f1 (ppm)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8.260
7.905
7.885
7.784
7.766
7.748
7.725
7.639
7.617
7.600
7.580
7.484
7.468
7.450
7.432
7.262
4.553
4.543
4.531
4.036
4.025
4.014
2.841
1.700
0.000
O
OH
N
N
4p
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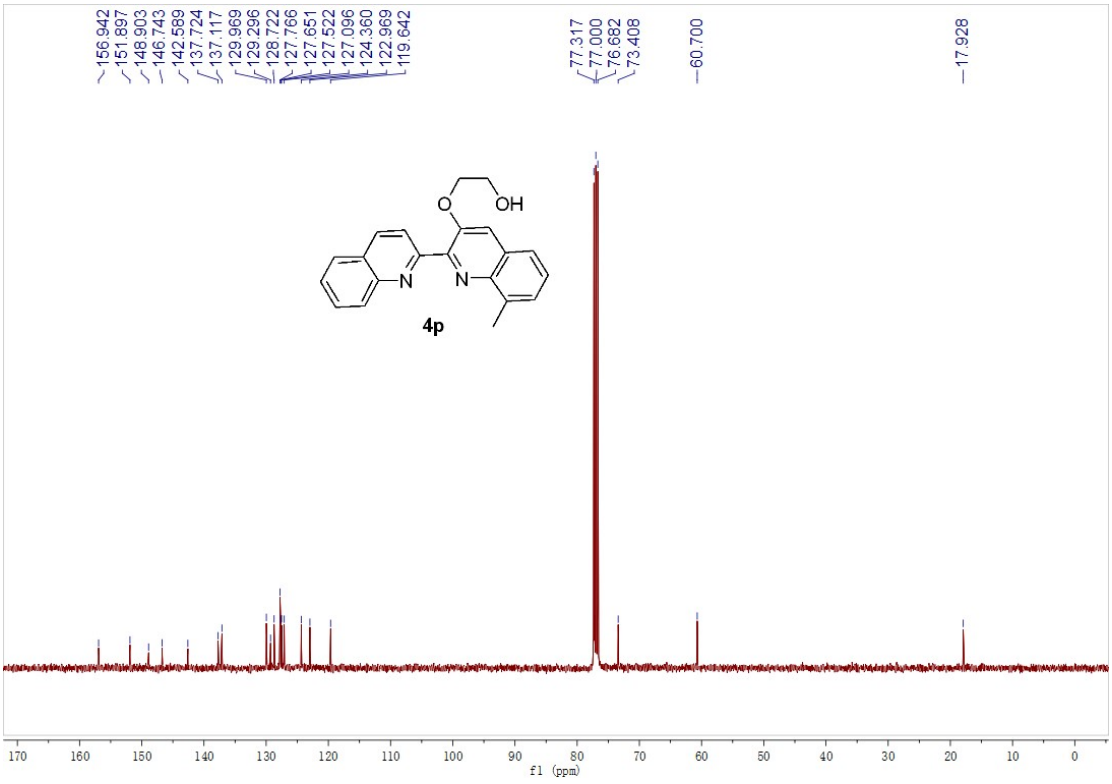
0.99
0.92
1.00
2.03
2.00
2.03
2.00
2.00
3.00
f1 (ppm)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137.117
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129.296
128.722
127.766
127.651
127.522
127.096
124.360
122.969
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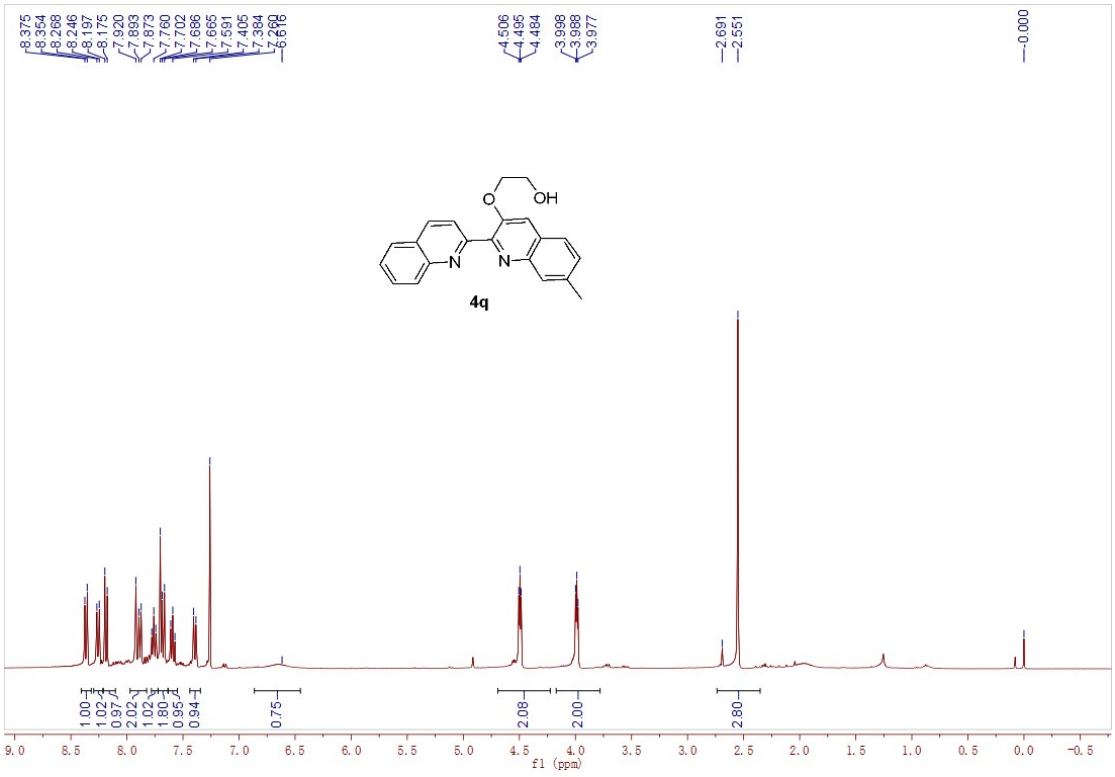
77.000
76.682
73.408
60.700
17.928
O
OH
N
N
4p
f1 (ppm)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8.175
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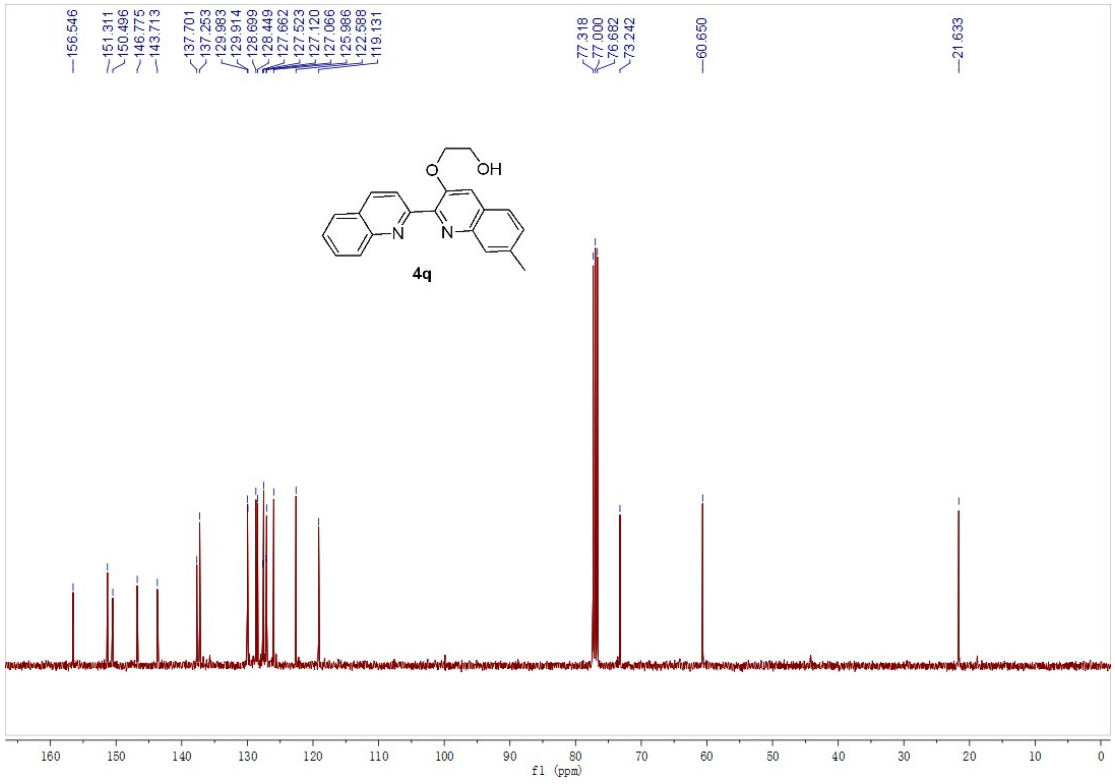
7.893
7.873
7.760
7.702
7.686
7.665
7.591
7.405
7.384
7.260
6.616
4.506
4.495
4.484
3.998
3.988
3.977
2.691
2.551
0.000
OH
N
N
4q
1.00
1.02
0.97
2.02
1.02
1.80
0.95
0.94
0.75
2.08
2.00
2.80
f1 (ppm)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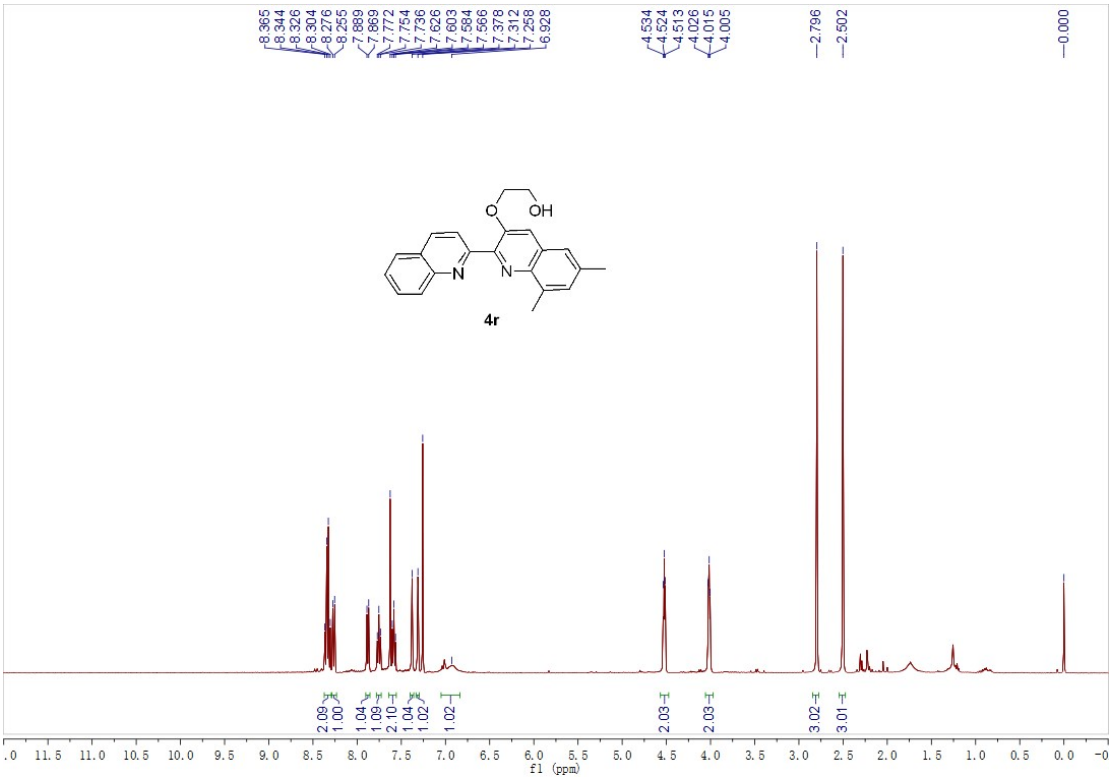
143.713
137.701
137.253
129.983
129.914
128.699
128.449
127.662
127.523
127.120
127.066
125.986
122.588
119.131
77.318
77.000
76.682
73.242
60.650
21.633
OH
N
N
4q
f1 (ppm)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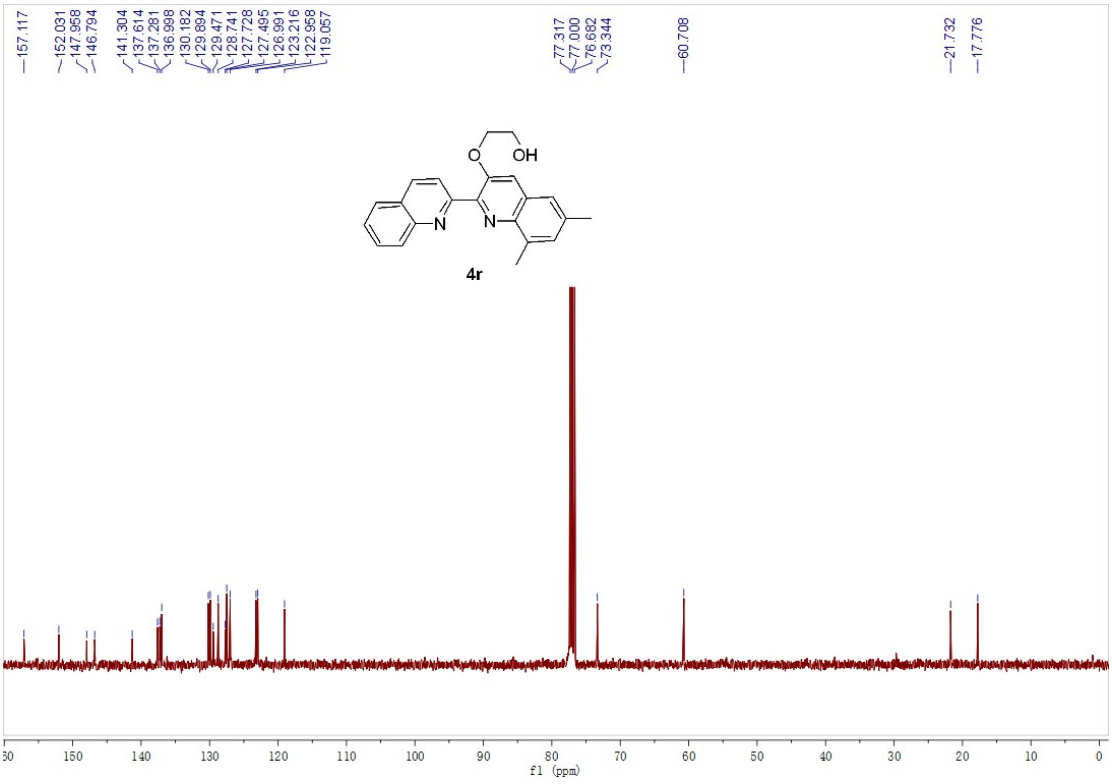
8.255
7.889
7.869
7.772
7.754
7.736
7.626
7.603
7.584
7.566
7.378
7.312
7.258
6.928
4.534
4.524
4.513
4.026
4.015
4.005
2.796
2.502
0.000
O
OH
N
N
4r
2.09
1.00
1.04
1.09
2.10
1.04
1.02
1.02
2.03
2.03
3.02
3.01
f1 (ppm)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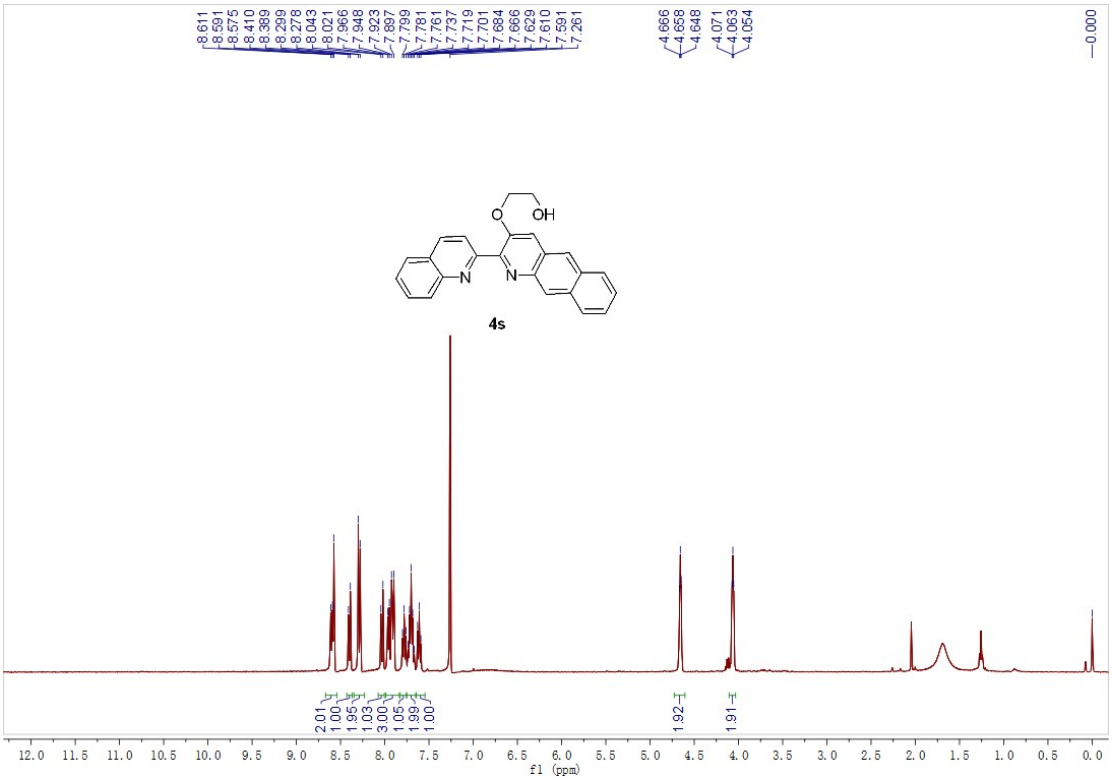
141.304
137.614
137.281
136.998
130.182
129.894
129.471
128.741
127.728
127.495
126.981
123.216
122.958
119.057
77.317
77.000
76.682
73.344
60.708
21.732
17.776
O
OH
N
N
4r
f1 (ppm)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8.299
8.278
8.043
8.021
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7.948
7.923
7.897
7.799
7.781
7.761
7.737
7.719
7.701
7.684
7.666
7.629
7.610
7.591
7.261
4.666
4.658
4.648
4.071
4.063
4.054
0.000
O
OH
N
N
4s
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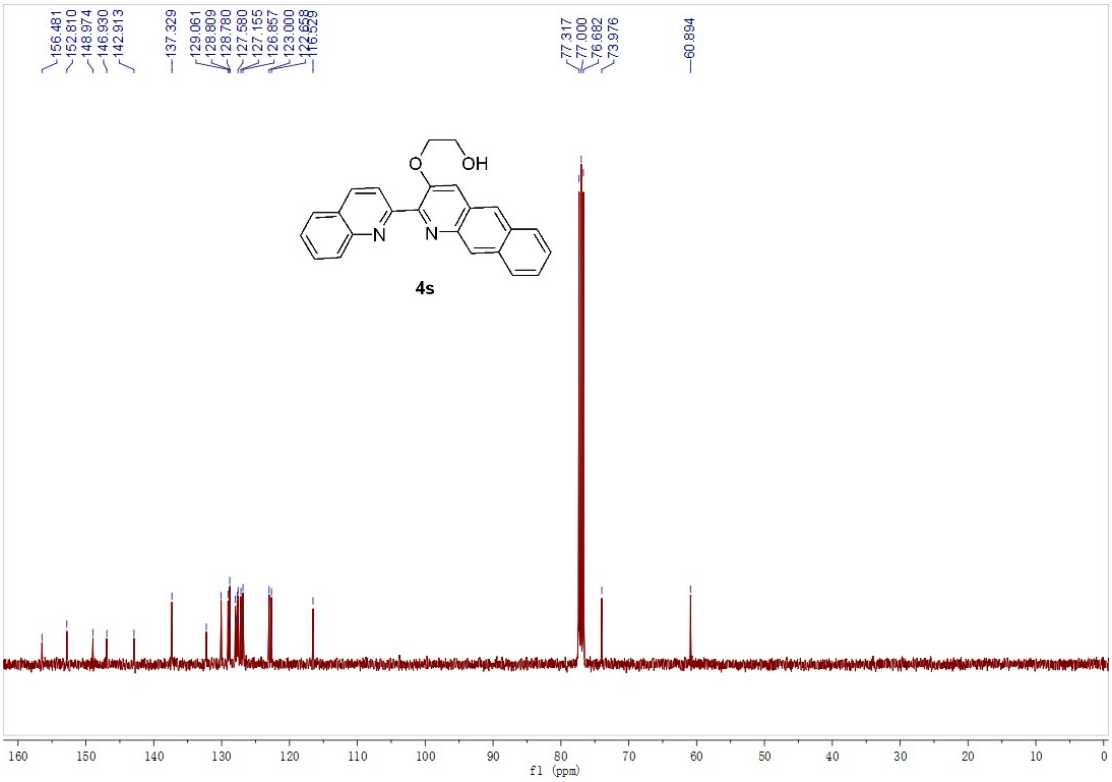
1.95
1.03
3.00
1.05
1.99
1.00
1.92
1.91
f1 (ppm)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128.809
128.780
127.580
127.155
126.857
123.000
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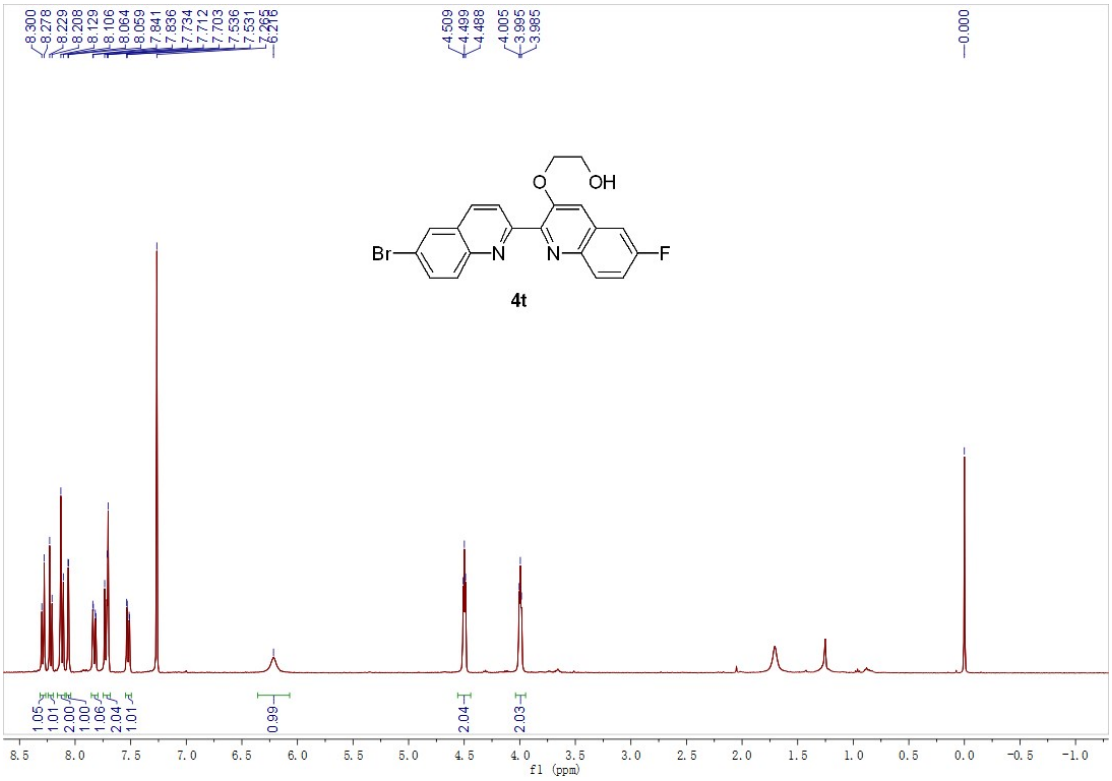
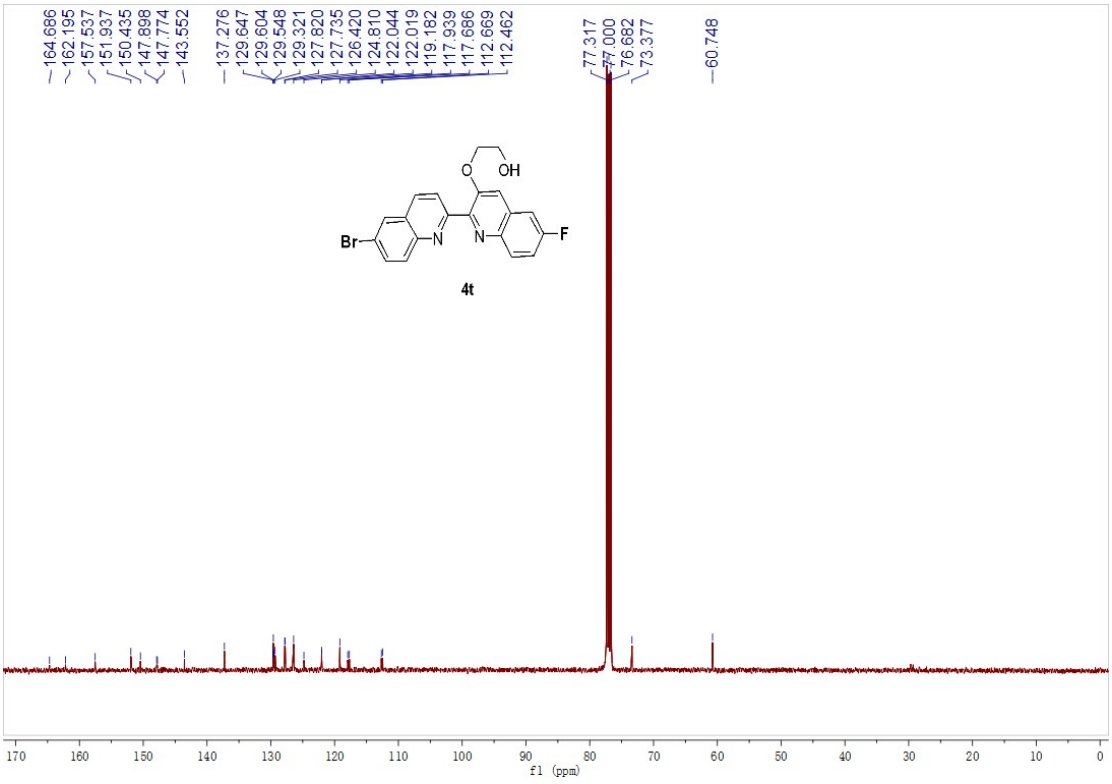
77.317
77.000
76.682
73.976
60.894
O
OH
N
N
4s
f1 (ppm)


8.300
8.278
8.229
8.208
8.129
8.106
8.064
8.059
7.841
7.836
7.734
7.712
7.703
7.536
7.531
7.265
6.216
4.509
4.499
4.488
4.005
3.995
3.985
0.000
O
OH
Br
N
N
F
4t
1.05
1.01
2.00
1.00
1.06
2.04
1.01
0.99
2.04
2.03
f1 (ppm)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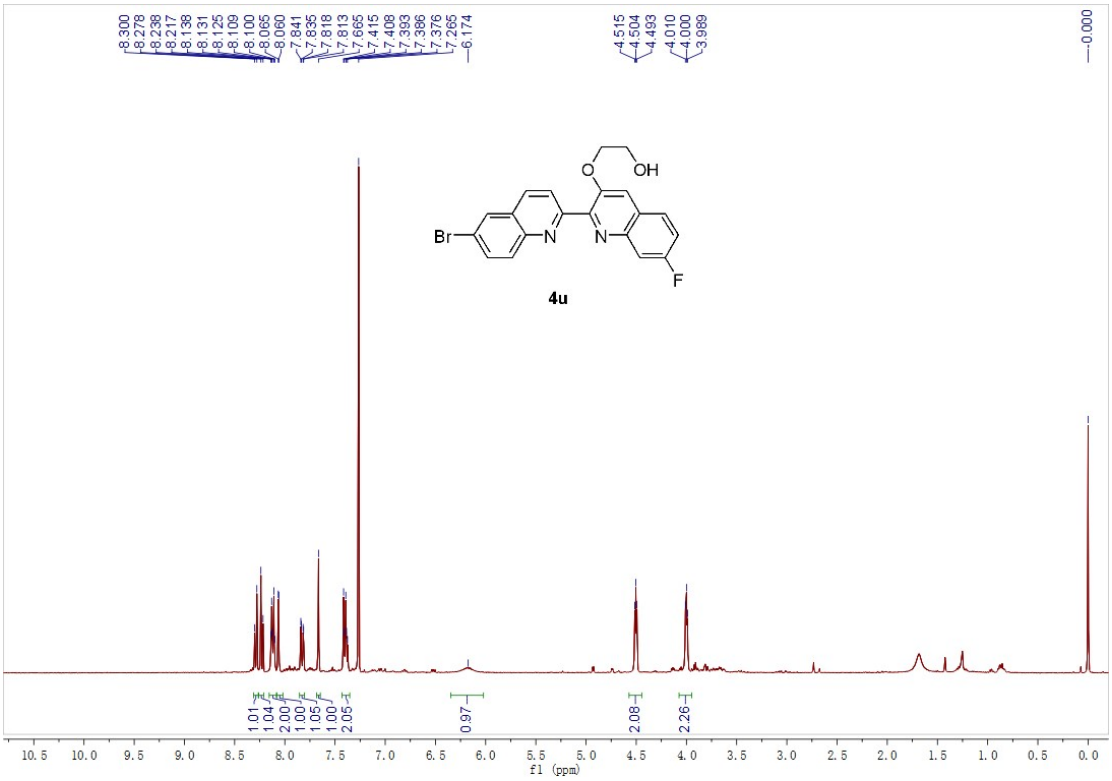
157.537
151.937
150.435
147.898
147.774
143.552
137.276
129.647
129.604
129.548
129.321
127.820
127.735
126.420
124.810
122.044
122.019
119.182
117.939
117.686
112.669
112.462
77.317
77.000
76.682
73.377
60.748
O
OH
Br
N
N
F
4t
f1 (ppm)


8.300
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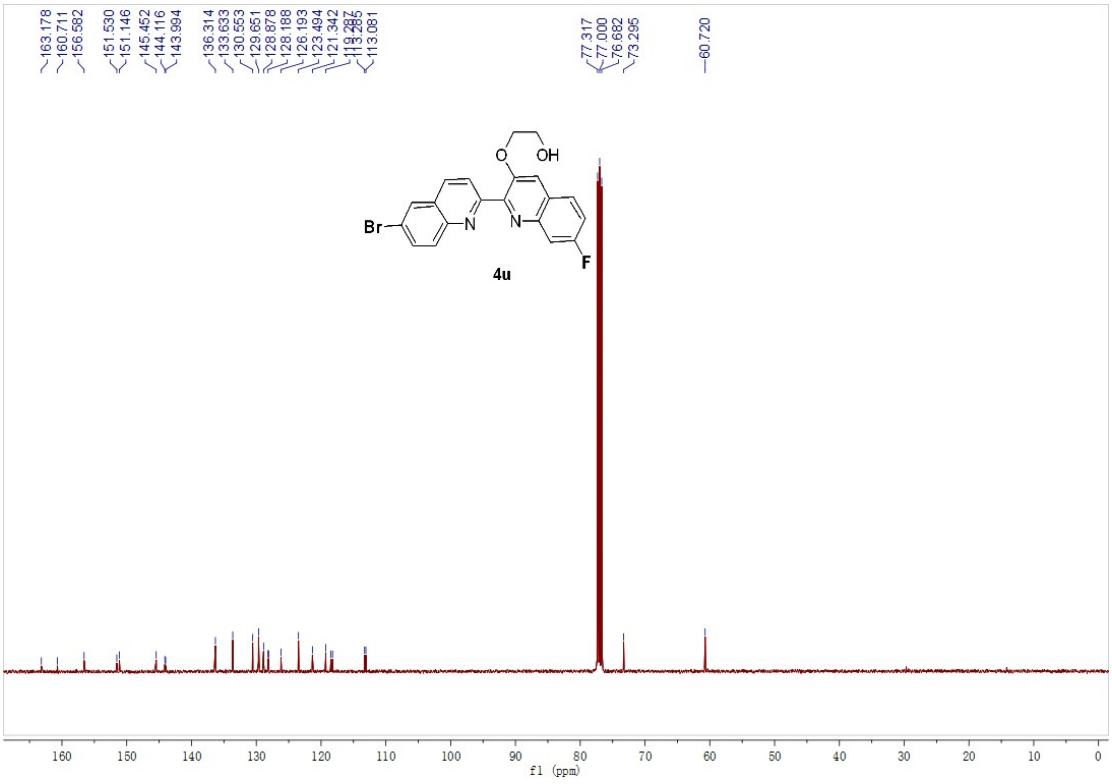
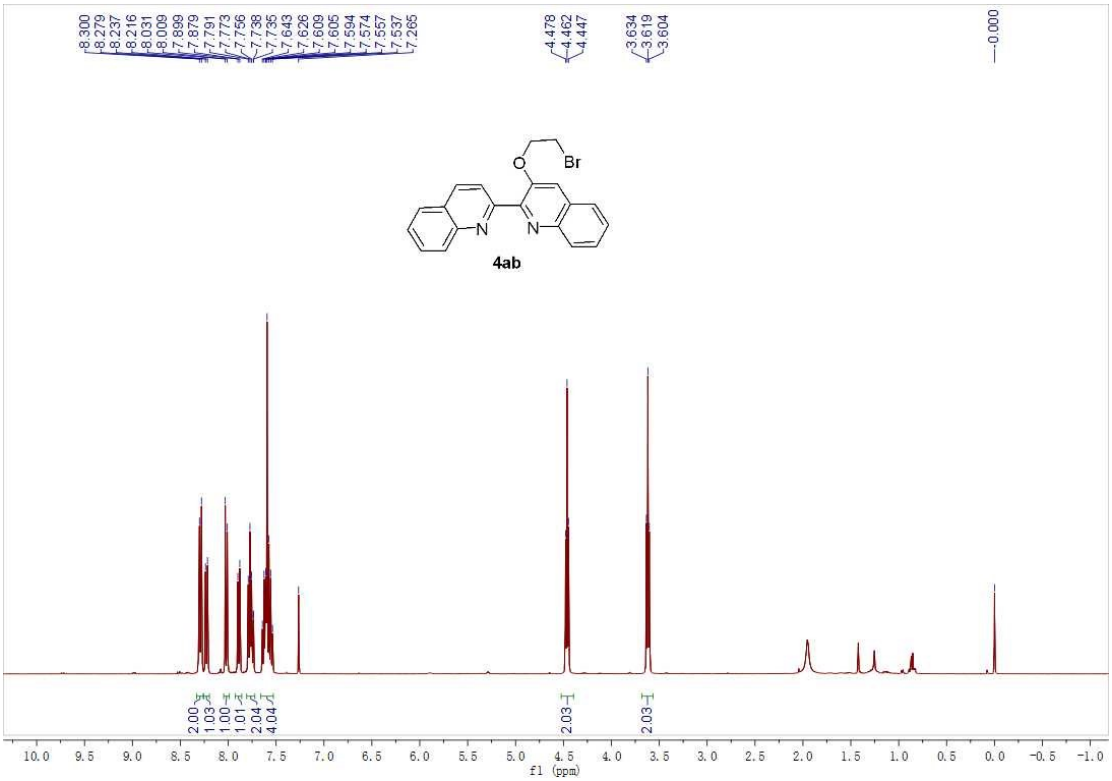
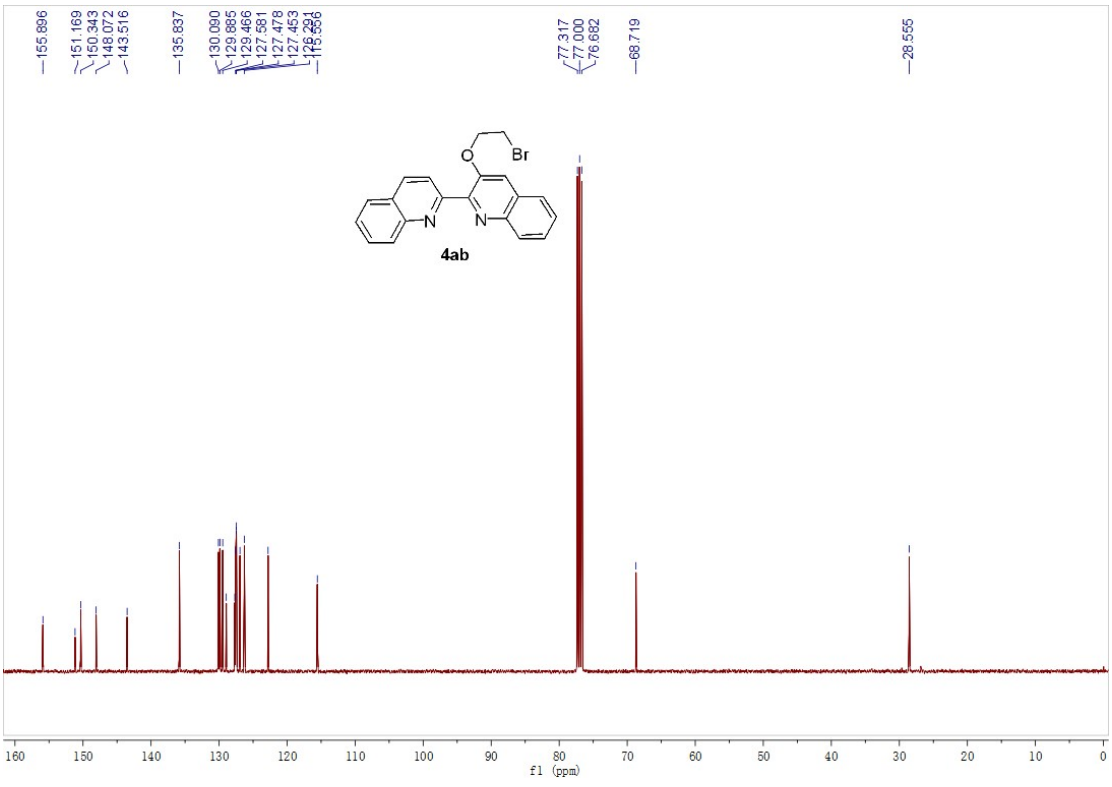
8.217
8.138
8.131
8.125
8.109
8.100
8.065
8.060
7.841
7.835
7.818
7.813
7.665
7.415
7.408
7.393
7.386
7.376
7.265
6.174
4.515
4.504
4.493
4.010
4.000
3.989
0.000
O
OH
Br
N
N
F
4u
1.01
1.04
2.00
1.00
1.05
1.00
2.05
0.97
2.08
2.26
f1 (ppm)


163.178
160.711
156.582
151.530
151.146
145.452
144.116
143.994
136.314
133.633
130.553
129.651
128.878
128.188
126.193
123.494
121.342
113.081
77.317
77.000
76.682
73.295
60.720
O
OH
Br
N
N
F
4u
f1 (ppm)


8.300
8.279
8.237
8.216
8.031
8.009
7.899
7.879
7.791
7.773
7.756
7.738
7.735
7.643
7.626
7.609
7.605
7.594
7.574
7.557
7.537
7.265
4.478
4.462
4.447
3.634
3.619
3.604
0.000
O
Br
N
N
4ab
2.00
1.03
1.00
1.01
2.04
4.04
2.03
2.03
f1 (ppm)


155.896
151.169
150.343
148.072
143.516
135.837
130.090
129.885
129.466
127.581
127.478
127.453
77.317
77.000
76.682
68.719
28.555
O
Br
N
N
4ab
f1 (ppm)