Hydrogen peroxide-mediated synthesis of 2,4-subtituted quinazolines via one-pot three-component reactions under metal-free conditions

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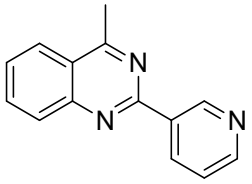
Supporting information

Materials and instrumentation

All reagents and starting materials were obtained commercially from Sigma-Aldrich, Acros and Merck, and were used as received without any further purification unless otherwise noted. Gas chromatographic (GC) analyses were performed using a Shimadzu GC 2010-Plus equipped with a flame ionization detector (FID) and an SPB-5 column (length = 30 m, inner diameter = 0.25 mm, and film thickness = 0.25 μm). The

temperature program for GC analysis held samples at 100 ^{o}C for 1 min; heated them from 100 to 280 ^{o}C at 40 ^{o}C/min; held them at 280 ^{o}C for 4.5 min. The GC yield was calculated using diphenyl ether as the internal standard. The ^{1}H NMR and ^{13}C NMR were recorded on Bruker AV 500 spectrometers using residual solvent peak as a reference.

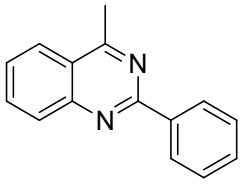
Characterization data of products



4-methyl-2-(pyridin-3-yl)quinazoline (3aa)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:3 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.6): Light yellow solid, 88% yield (19.4 mg).

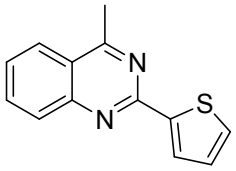
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 9.81 (d, *J* = 1.5 Hz, 1H), 8.87 (dt, *J* = 8.0 Hz, 2.0 Hz, 1H), 8.72 (dd, *J* = 5.0 Hz, 1.5 Hz, 1H), 8.09 - 8.05 (m, 2H), 7.87 (ddd, *J* = 8.5, 7.0 Hz, 1.0 Hz, 1H), 7.60 (ddd, *J* = 8.5 Hz, 7.0 Hz, 1 Hz, 1H), 7.44 (ddd, *J* = 8.0, 5.0, 1.0 Hz, 1H), 3.01 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 168.5, 158.2, 150.8, 150.2, 150.1, 135.8, 133.8, 133.7, 129.2, 127.3, 125.0, 123.3, 123.2, 21.9.



4-methyl-2-phenylquinazoline (3ab)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:5 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.45): Yellow solid, 89% yield (19.5 mg).

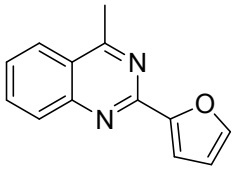
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.63 – 8.60 (m, 2H), 8.08 – 8.06 (m, 2H), 7.85 (ddd, *J* = 8.5, 7.0 Hz, 1.5 Hz, 1H), 7.58 – 7.46 (m, 4H), 3.01 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 168.3, 160.1, 150.3, 138.2, 133.5, 130.4, 129.2, 128.6, 128.5, 126.8, 124.9, 123.0, 22.0.



4-methyl-2-(thiophen-2-yl)quinazoline (3ac)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.5): Yellow liquid, 75% yield (16.9 mg).

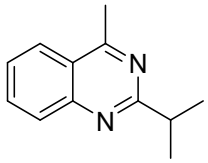
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.16 (d, *J* = 3 Hz, 1H), 8.03 – 8.00 (m, 2H), 7.82 (ddd, *J* = 8.5 Hz, 7.0 Hz, 1.5 Hz, 1H), 7.53 (ddd, *J* = 8.0 Hz, 7.0 Hz, 1.5 Hz, 1H), 7.49 (dd, *J* = 5.0 Hz, 1.5 Hz, 1H), 7.18 (dd, *J* = 5.0 Hz, 4.0 Hz, 1H), 2.96 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 168.5, 156.9, 150.0, 144.0, 133.7, 129.7, 129.2, 128.6, 128.2, 126.6, 125.0, 122.8, 21.8.



2-(furan-2-yl)-4-methylquinazoline (3ad)

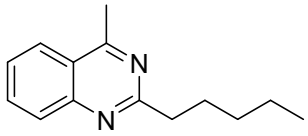
Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.34): Brown liquid, 74% yield (15.5 mg).

^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.05 – 8.03 (m, 2H), 7.84 (ddd, *J* = 8.5 Hz, 7.0 Hz, 1.5 Hz, 1H), 7.68 (dd, *J* = 2.0 Hz, 1.0 Hz, 1H), 7.56 (ddd, *J* = 8.0 Hz, 7.0 Hz, 1.0 Hz, 1H), 7.46 (dd, *J* = 3.5 Hz, 1.0 Hz, 1H), 6.60 (dd, *J* = 3.5 Hz, 2.0 Hz, 1H), 2.98 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 168.7, 153.3, 152.7, 150.0, 145.1, 133.8, 128.9, 126.8, 125.0, 122.8, 113.8, 112.1, 21.8.



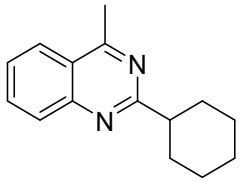
2-isopropyl-4-methylquinazoline (3ae)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37 – 63 μm, ethyl acetate/hexane = 1:10 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.5): Yellow liquid, 70% yield (13.0 mg). ^{1}H-NMR (500MHz, $CDCl_3$) δ(ppm) 8.05 (d, *J* = 8.5 Hz, 1H), 7.97 (d, *J* = 8.5 Hz, 1H), 7.84 – 7.80 (m, 1H), 7.56 – 7.53 (m, 1H), 3.32 (m, 1H), 2.93 (s, 3H), 1.42 (d, *J* = 6.5, 6H). ^{13}C-NMR ($CDCl_3$, 125 MHz) δ(ppm) 170.8, 168.0, 149.9, 133.2, 128.6, 126.4, 124.8, 122.6, 37.8, 21.7



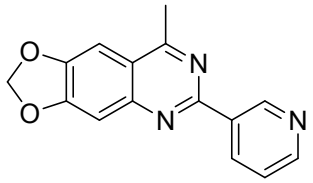
4-methyl-2-pentylquinazoline (3af)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37 – 63 μm, ethyl acetate/hexane = 1:10 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.55): Yellow liquid, 75% yield (16.0mg). ^{1}H-NMR (500MHz, $CDCl_3$) δ(ppm) 8.06 (d, *J* = 8.0 Hz, 1H), 7.97 (d, *J* = 8.5 Hz, 1H), 7.84 (t, *J* = 7.5 Hz, 7.0 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 7.0 Hz, 1H), 3.05 (m, 2H), 2.94 (s, 3H), 1.90 (m, 2H), 1.40 (m, 4H), 0.91 (t, *J* = 7.0 Hz, 7.0 Hz, 3H).^{13}C-NMR ($CDCl_3$, 125 MHz) δ(ppm) 168.2, 167.0, 149.8, 133.5, 128.4, 126.5, 124.9, 122.4, 40.0, 31.8, 28.8, 22.6, 21.7, 14.0.



2-cyclohexyl-4-methylquinazoline (3ag)

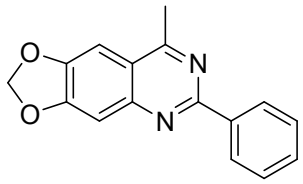
Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37 – 63 μm, ethyl acetate/hexane = 1:10 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.6): Light yellow solid, 85% yield (19.2 mg). ^{1}H-NMR (500MHz, $CDCl_3$) δ(ppm) 8.01(d, *J* = 8.0 Hz, 1H), 7.95 (d, *J* = 8.5 Hz, 1H), 7.81 – 7.78 (m, 1H), 7.53 – 7.50 (m, 1), 3.01 -2.96 (m, 1H), 2.91 (s, 3H), 2.08 – 2.05 (m, 2H), 1.90 – 1.86 (m, 2H), 1.81 – 1.73 (m, 3H), 1.48 – 1.42 (m, 2H), 1.39 – 1.34 (m, 2H). ^{13}C-NMR ($CDCl_3$, 125 MHz) δ(ppm) 170.0, 167.8, 150.0, 133.1, 128.6, 126.3, 124.8, 122.6, 47.8, 31.9, 26.3, 21.7.



8-methyl-6-(pyridin-3-yl)-[1,3]dioxolo[4,5-*g*]quinazoline (3ba)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.3): Light yellow solid, 84% yield (22.3 mg).

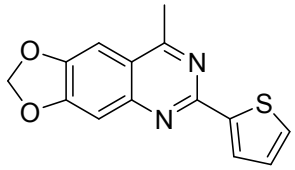
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 9.75 (s, 1H), 8.84 (dt, *J* = 8.0, 1.9 Hz, 1H), 8.69 (dd, *J* = 4.9, 1.7 Hz, 1H), 7.46 – 7.43 (m, 1H), 7.33 (s, 1H), 7.29 (s, 1H), 6.16 (s, 2H), 2.89 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 165.5, 157.0, 153.7, 149.9, 149.7, 149.4, 148.4, 135.9, 134.2, 123.4, 120.1, 105.4, 102.2, 100.3, 22.1.



8-methyl-6-phenyl-[1,3]dioxolo[4,5-*g*]quinazoline (3bb)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.42): Light yellow solid, 88% yield (23.2 mg).

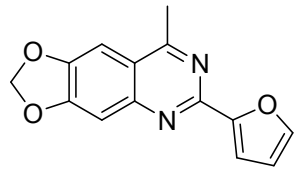
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.53 (d, *J* = 8.0 Hz, 2H), 7.50 – 7.44 (m, 4H), 7.31 – 7.24 (m, 1H), 6.11 (d, *J* = 5.5 Hz, 2H), 2.85 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 165.2, 159.2, 153.4, 147.9, 138.3, 136.2, 130.1, 130.0, 129.4, 128.2, 105.4, 102.1, 99.2, 22.1.



8-methyl-6-(thiophen-2-yl)-[1,3]dioxolo[4,5-*g*]quinazoline (3bc)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.26): Yellow solid, 85% yield (22.9 mg).

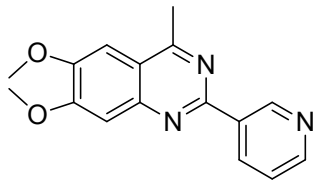
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.08 (d, *J* = 2.0 Hz, 1H), 7.44 (dd, *J* = 5.0, 1.0 Hz, 1H), 7.29 (s, 1H), 7.25 (s, 1H), 7.15 (dd, *J* = 5.0 Hz, 3.5 Hz, 1H), 6.13 (s, 2H), 2.84 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 153.6, 147.8, 129.0, 128.4, 128.1, 119.4, 105.0, 102.1, 100.5, 22.0.



6-(furan-2-yl)-8-methyl-[1,3]dioxolo[4,5-*g*]quinazoline (3bd)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.3): Brown liquid, 72% yield (18.3 mg).

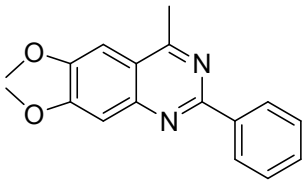
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 7.64 (dd, *J* = 1.5 Hz, 1.0 Hz, 2H), 7.38 – 7.36 (m, 2H), 6.57 (dd, *J* = 3.5 Hz, 2.0 Hz, 1H), 6.14 (s, 2H), 2.86 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 165.6, 153.7, 149.4, 148.0, 144.7, 119.5, 112.8, 112.0, 105.2, 102.1, 100.5, 22.0.



6,7-dimethoxy-4-methyl-2-(pyridin-3-yl)quinazoline (3ca)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.35): Light yellow solid, 82% yield (23.0 mg).

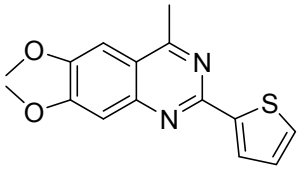
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 9.75 (s, 1H), 8.82 (dt, *J* = 8.0 Hz, 2.0 Hz, 1H), 8.69 (dd, *J* = 5.0 Hz, 1.5 Hz, 1H), 7.43 (ddd, *J* = 8.0 Hz, 5.0 Hz, 1.0 Hz, 1H), 7.35 (s, 1H), 7.20 (s, 1H), 4.08 (s, 3H), 4.05 (s, 3H), 2.93 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 165.0, 157.1, 155.7, 150.2, 149.6, 148.1, 135.6, 134.2, 123.3, 118.6, 107.4, 102.3, 56.4, 56.1, 21.9.



6,7-dimethoxy-4-methyl-2-phenylquinazoline (3cb)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.45): Light yellow solid, 85% yield (23.8 mg).

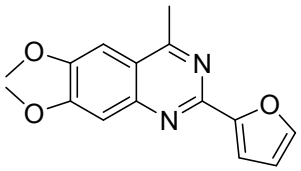
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.54 (d, *J* = 6.5 Hz, 2H), 7.51 – 7.48 (m, 2H), 7.46 – 7.44 (m, 1H), 7.34 (s, 1H), 7.14 (s, 1H), 4.04 (s, 3H), 4.01 (s, 3H), 2.89 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 164.7, 159.1, 155.5, 149.7, 148.1, 138.6, 129.9, 128.4, 128.1, 118.3, 107.4, 102.3, 56.3, 56.1, 21.9.



6,7-dimethoxy-4-methyl-2-(thiophen-2-yl)quinazoline (3cc)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.27): Yellow solid, 80% yield (10.6 mg).

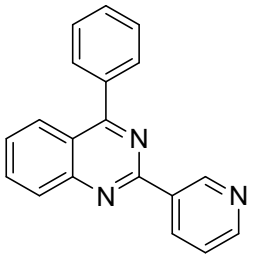
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.07 (dd, *J* = 3.5 Hz, 1.0 Hz, 1H), 7.44 (dd, *J* = 5.0 Hz, 1.5 Hz, 1H), 7.30 (s, 1H), 7.16 – 7.14 (m, 2H), 4.05 (s, 3H), 4.03 (s, 3H), 2.88 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 165.0, 156.1, 155.7, 149.6, 147.9, 128.8, 128.1, 118.1, 107.1, 102.6, 56.4, 56.1, 21.8.



2-(furan-2-yl)-6,7-dimethoxy-4-methylquinazoline (3cd)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:2 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.32): Brown liquid, 76% yield (20.5 mg).

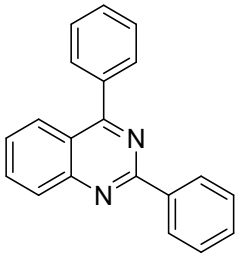
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 7.65 (dd, *J* = 2.0 Hz, 1.0 Hz, 1H), 7.42 (s, 1H), 7.38 (d, *J* = 3.5 Hz, 1H), 7.18 (s, 1H), 6.58 (dd, *J* = 3.5 Hz, 1.5 Hz, 1H), 4.04 (d, *J* = 4.0 Hz, 6H), 2.90 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 165.2, 155.9, 152.7, 152.4, 149.8, 147.7, 144.6, 118.1, 112.6, 112.0, 107.3, 102.5, 56.4, 56.1, 21.8.



4-phenyl-2-(pyridin-3-yl)quinazoline (3da)

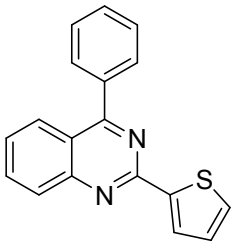
Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:5 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.53): Light yellow solid, 84% yield (23.8 mg).

^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 9.89 (s, 1H), 8.97 (dt, *J* = 8.0 Hz, 1.9 Hz, 1H), 8.73 (d, *J* = 3.5 Hz, 1H), 8.16 (ddd, *J* = 8.0 Hz, 6.5, 1.5 Hz, 2H), 7.93 – 7.87 (m, 3H), 7.62 – 7.57 (m, 4H), 7.47 (dd, *J* = 8.0 Hz, 4.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 168.6, 158.2, 151.8, 150.6, 150.0, 137.3, 136.2, 133.9, 133.8, 130.1, 130.1, 129.2, 128.6, 127.6, 127.1, 123.4, 121.9.



2,4-diphenylquinazoline (3db)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:5 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.6): Light yellow solid, 85% yield (24.0 mg). ^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.70 – 8.68 (m, 2H), 8.15 (dt, *J* = 8.5 Hz, 1 Hz, 1H), 8.09 (dd, *J* = 8.5 Hz, 1.5 Hz, 1H), 7.87 – 7.83 (m, 3H), 7.58 – 7.55 (m, 3H), 7.53 – 7.48 (m, 4H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 168.3, 160.2, 151.9, 138.2, 137.7, 133.5, 130.5, 130.2, 129.9, 129.1, 128.7, 128.5, 127.0, 121.7.

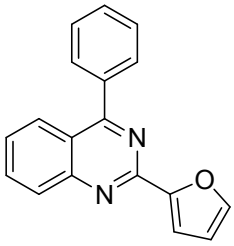


4-phenyl-2-(thiophen-2-yl)quinazoline (3dc)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:5 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.44): Yellow liquid, 78% yield (22.5 mg).

^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.20 (dd, *J* = 3.5 Hz, 1.0 Hz, 1H), 8.07 – 8.04 (m, 2H), 7.85 – 7.81 (m, 3H), 7.58 – 7.55 (m, 3H), 7.49 – 7.4 (m, 2H), 7.17 (dd, *J* = 5.0 Hz,

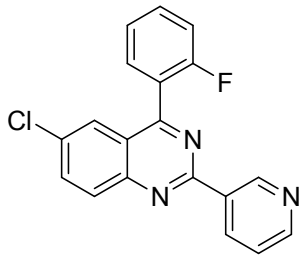
3.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 168.4, 157.2, 151.8, 144.1, 137.3, 133.7, 130.2, 130.0, 129.8, 129.4, 128.6, 128.5, 128.2, 127.1, 126.7, 121.5.



2-(furan-2-yl)-4-phenylquinazoline (3dd)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:5 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.5): Brown liquid, 68% yield (18.5 mg).

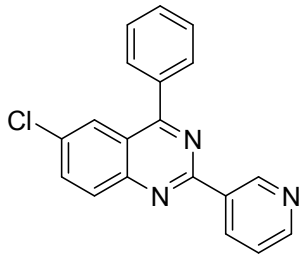
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 8.18 (dt, *J* = 8.5 Hz, 1 Hz, 1H), 8.07 (ddd, *J* = 8.5 Hz, 1.5 Hz, 0.5 Hz, 1H), 7.88 (ddd, *J* = 8.5 Hz, 7.0 Hz, 1.5 Hz, 1H), 7.83 – 7.81 (m, 2H), 7.69 (dd, *J* = 2.0 Hz, 1.0 Hz, 1H), 7.59 – 7.56 (m, 3H), 7.54 – 7.50 (m, 2H), 6.60 (dd, *J* = 3.5 Hz, 1.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 168.9, 153.5, 152.7, 151.5, 145.3, 137.2, 133.9, 130.0, 130.04, 128.8, 128.5, 127.1, 127.0, 121.6, 114.3, 112.2.



6-chloro-4-(2-fluorophenyl)-2-(pyridin-3-yl)quinazoline (3ea)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:4 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.46): White solid, 69% yield (23.0 mg).

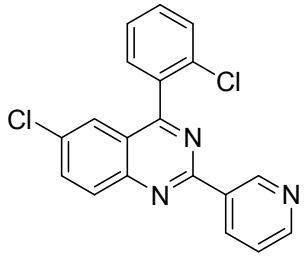
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 9.82 (s, 1H), 8.86 (dt, *J* = 8.0 Hz, 2.0 Hz, 1H), 8.72 (d, *J* = 5.0 Hz, 1H), 8.09 (d, *J* = 9.0 Hz, 1H), 7.82 (dd, *J* = 9.0, 2.0 Hz, 1H), 7.62 – 7.60 (m, 2H), 7.55 – 7.47 (m, 3H), 7.42 (dd, *J* = 8.0, 4.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 166.6, 158.8, 151.3, 150.3, 149.7, 135.9, 135.6, 133.2, 132.8, 131.6, 130.2, 127.1, 125.6, 123.3, 123.0. ^{19}F NMR ($CDCl_3$, 470 MHz, F) δ(ppm) −112.9.



6-chloro-4-phenyl-2-(pyridin-3-yl)quinazoline (3fa)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:4 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.5): White solid, 73% yield (23.2 mg).

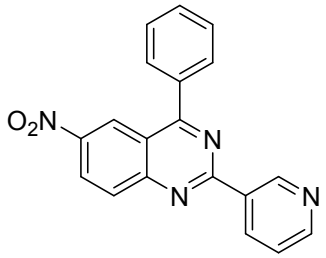
^{1}H-NMR (500 MHz, $CDCl_3$) δ(ppm) 9.87 (s, 1H), 8.94 (dt, *J* = 8.0 Hz,2.0 Hz, 1H), 8.76 (s, 1H), 8.12 – 8.11 (m, 2H), 7.87 – 7.84 (m, 3H), 7.64 – 7.62 (m, 3H), 7.47 (dd, *J* = 8.0 Hz, 4.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ(ppm) 167.9, 151.2, 150.4, 150.2, 136.8, 136.1, 134.8, 133.3, 130.9, 130.4, 130.0, 128.8, 125.9, 123.5, 122.4.



6-chloro-4-(2-chlorophenyl)-2-(pyridin-3-yl)quinazoline (3ga)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:4 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.54): White solid, 70% yield (24.6 mg).

^{1}H-NMR (500 MHz, DMSO-d_6) δ(ppm) 9.66 (d, J = 2.0 Hz, 1H), 8.85 (dt, J = 8.0 Hz, 2.0 Hz, 1H), 8.78 (dd, J = 5.0 Hz, 1.5 Hz, 1H), 8.24 (d, J = 9.0 Hz, 1H), 8.13 (dd, J = 9.0 Hz, 2.5 Hz, 1H), 7.82 (td, J = 7.5 Hz, 2.0 Hz, 1H), 7.78 – 7.74 (m, 2H), 7.64 (dd, J = 8.0 Hz, 5.0 Hz, 1H), 7.55 – 7.50 (m, 2H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ(ppm) 150.9, 148.9, 135.2, 135.0, 132.3, 132.2, 132.2, 131.3, 130.5, 124.7, 124.7, 124.6, 123.3, 115.8, 115.7.



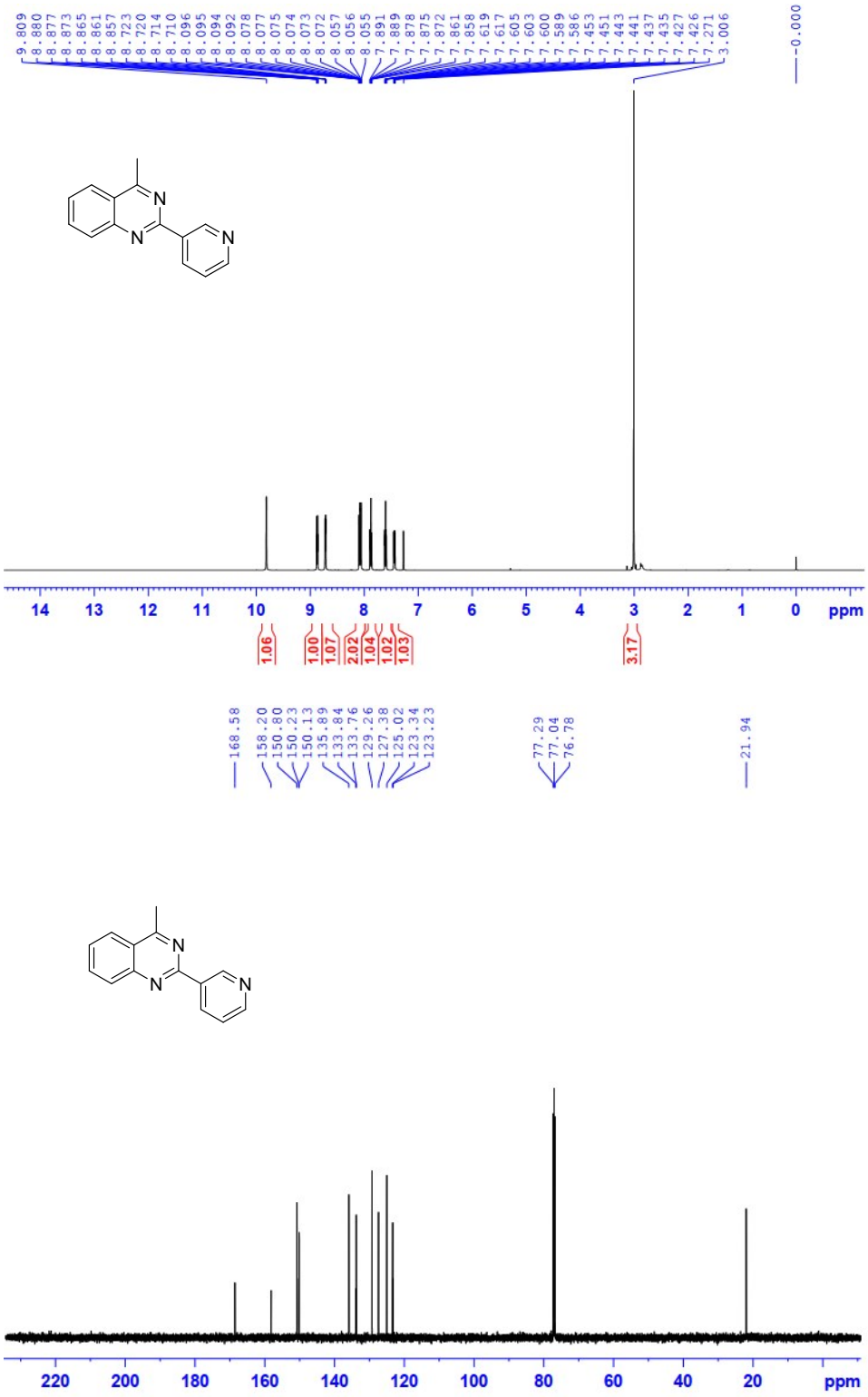
6-nitro-4-phenyl-2-(pyridin-3-yl)quinazoline (3ha)

Prepared as shown in the general experimental procedure and purified on silica gel (230-400 mesh or 37-63 μm, ethyl acetate/hexane = 1:4 (v./v.), TLC silica gel 60 F_{254}, R_f = 0.35): White solid, 61% yield (20.0 mg).

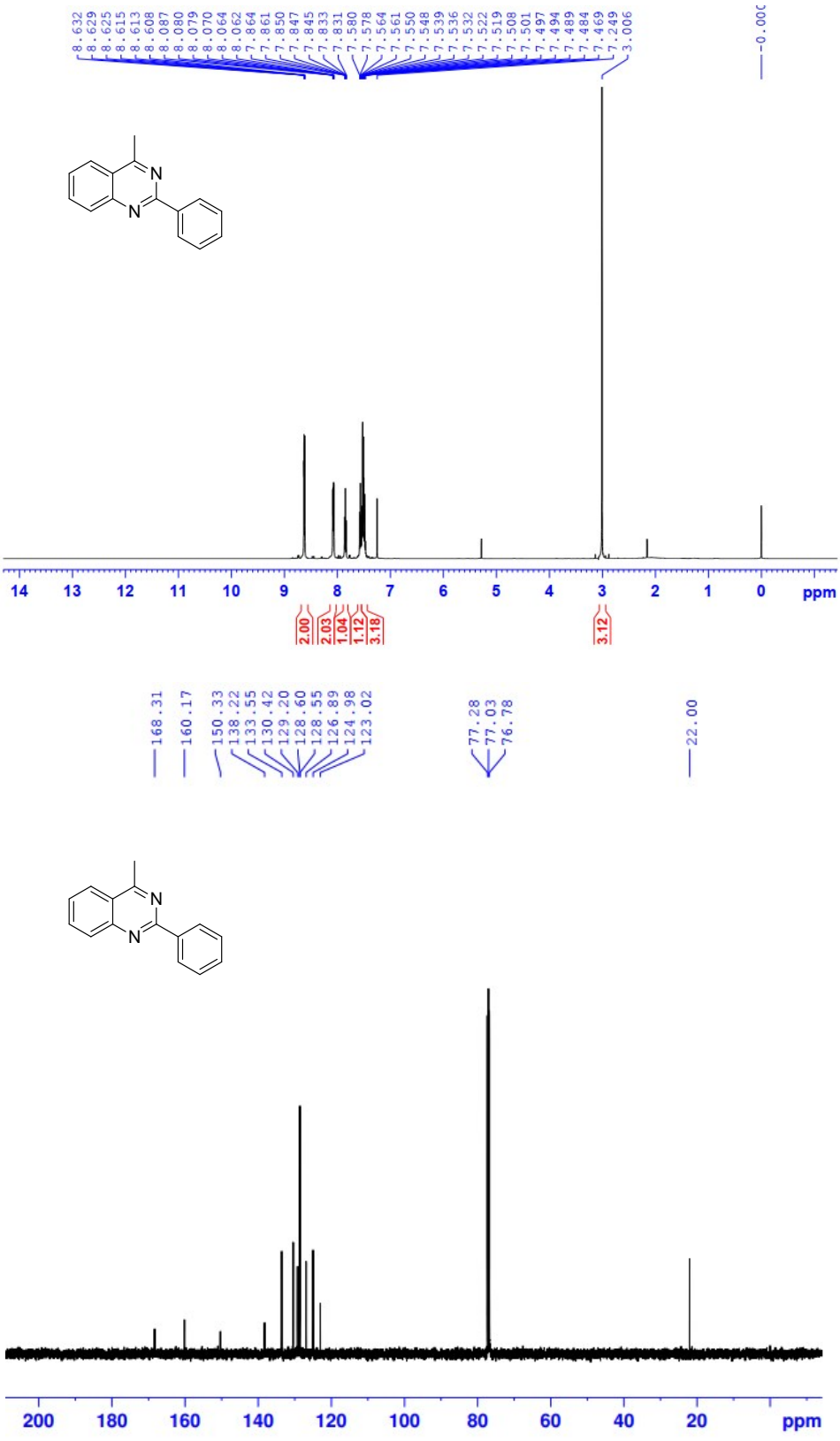
^{1}H-NMR (500 MHz, DMSO-d_6) δ(ppm) 9.74 (dd, J = 2.0 Hz, 1.0 Hz, 1H), 8.91 – 8.86 (m, 2H), 8.79 (dd, J = 5.0 Hz, 1.5 Hz, 1H), 8.73 (dd, J = 9.0 Hz, 2.5 Hz, 1H), 8.35 (d, J = 9.0 Hz, 1H), 8.01 – 7.99 (m, 2H), 7.76 – 7.72 (m, 3H), 7.64 (ddd, J = 8.0 Hz, 5.0 Hz, 1.0 Hz, 1H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ(ppm) 170.5, 153.4, 152.1, 149.7, 145.6, 135.9, 135.7, 132.2, 131.0, 130.7, 130.2, 128.9, 127.7, 124.0, 123.9.

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

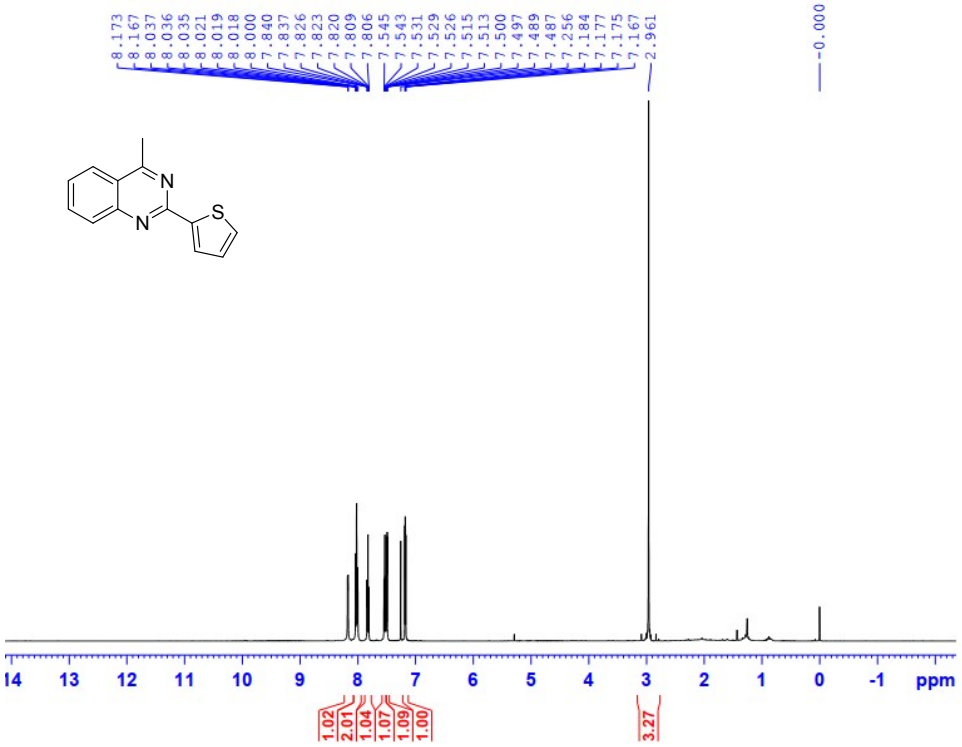
^{1}H and ^{13}C spectra of **3aa**

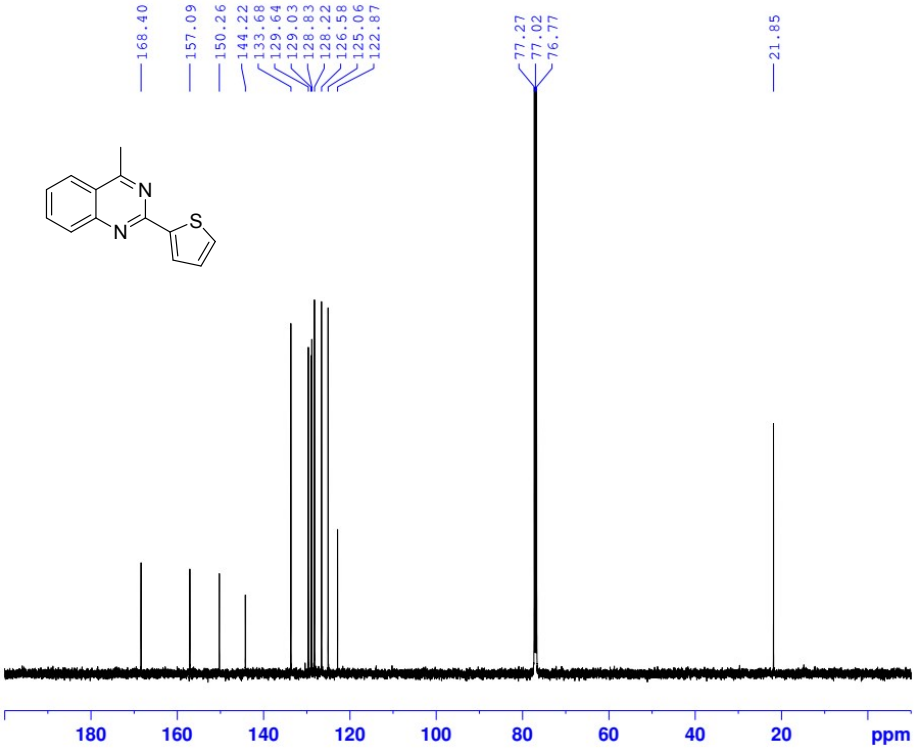


^{1}H and ^{13}C spectra of **3ab**

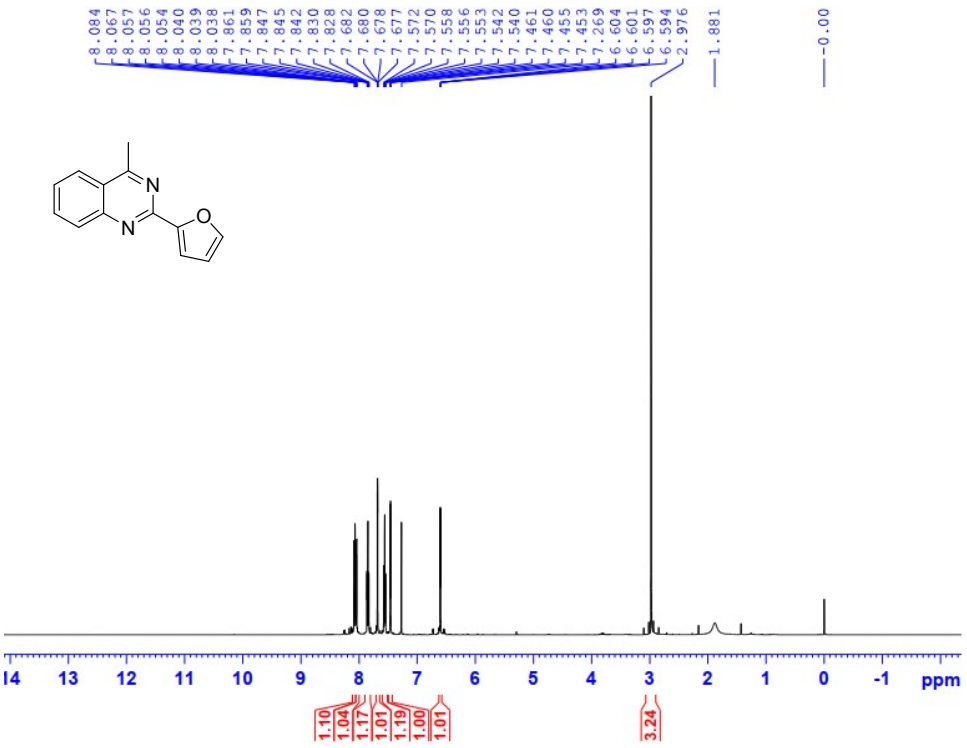


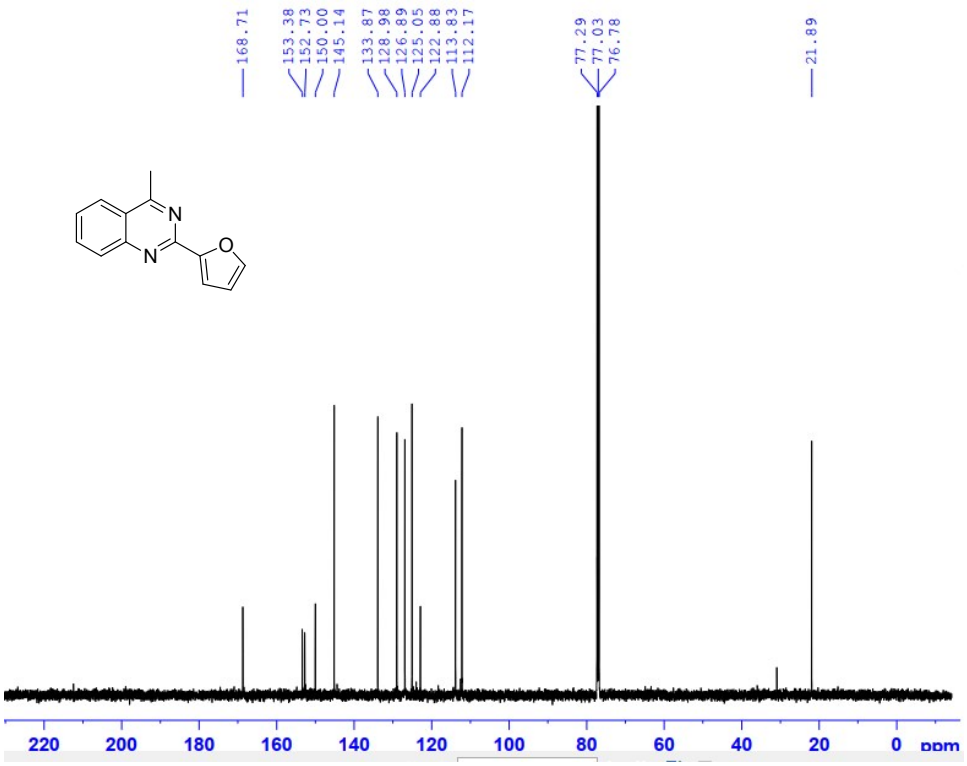
^{1}H and ^{13}C spectra of **3ac**



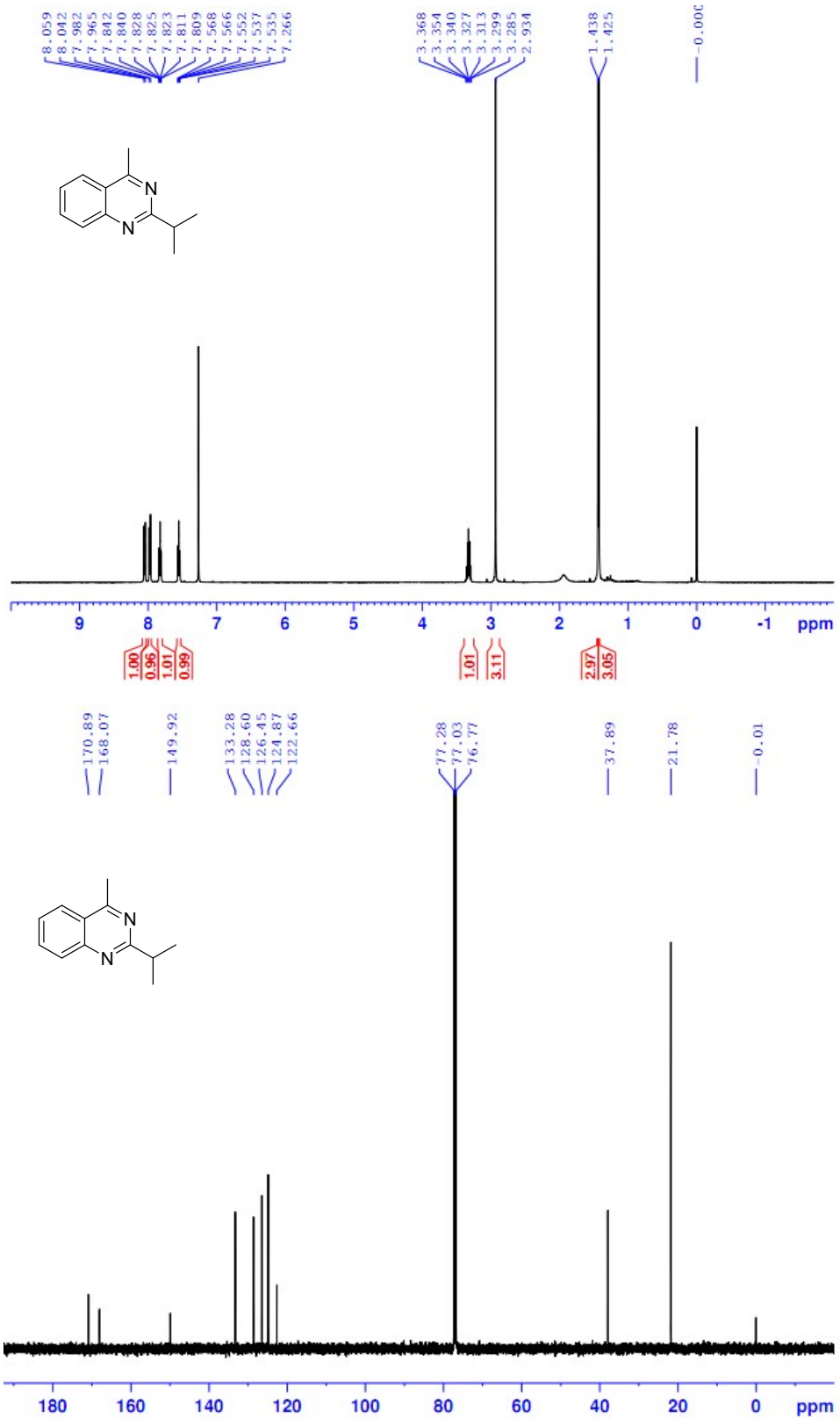


^{1}H and ^{13}C spectra of **3ad**

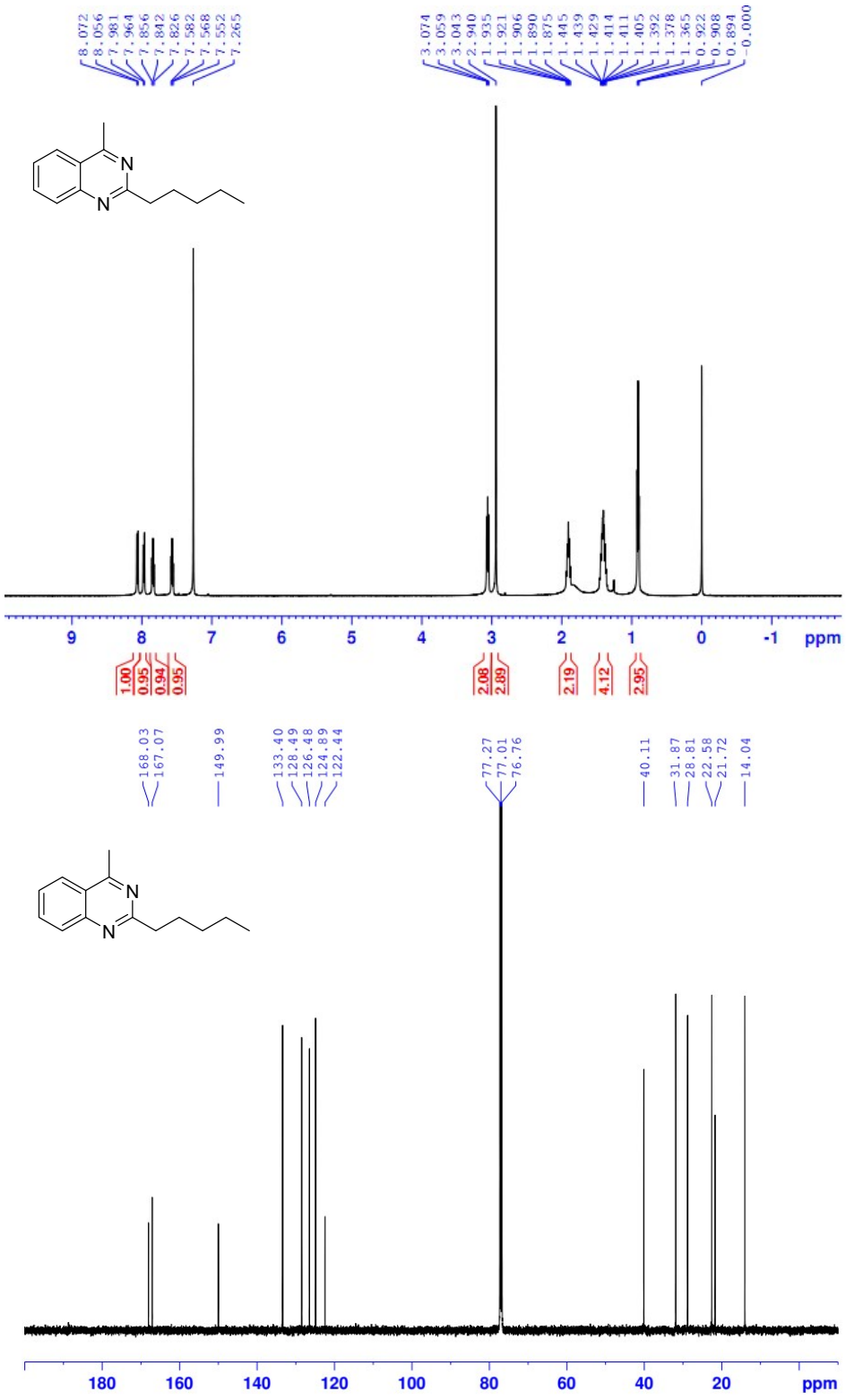




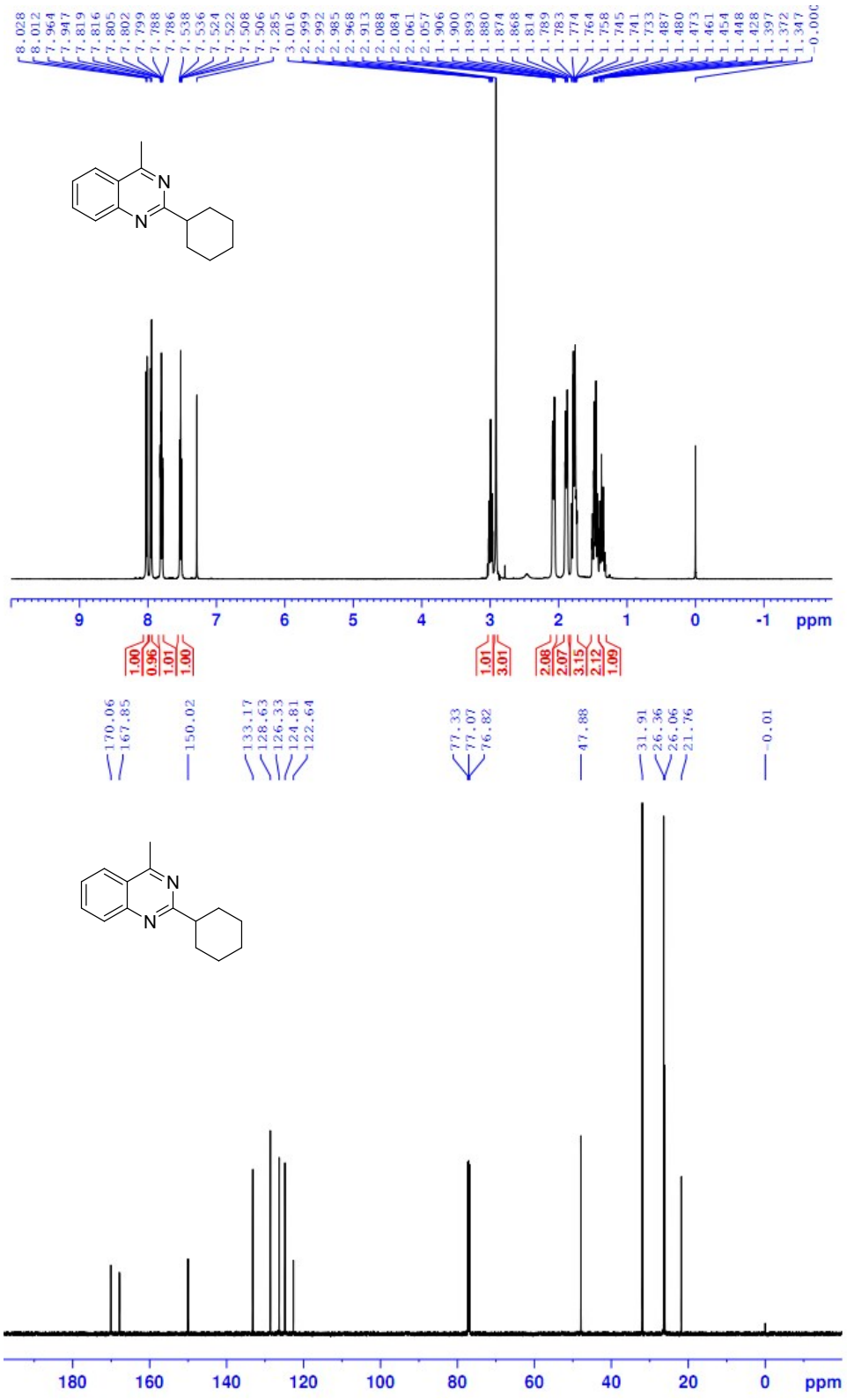
^{1}H and ^{13}C spectra of **3ae**



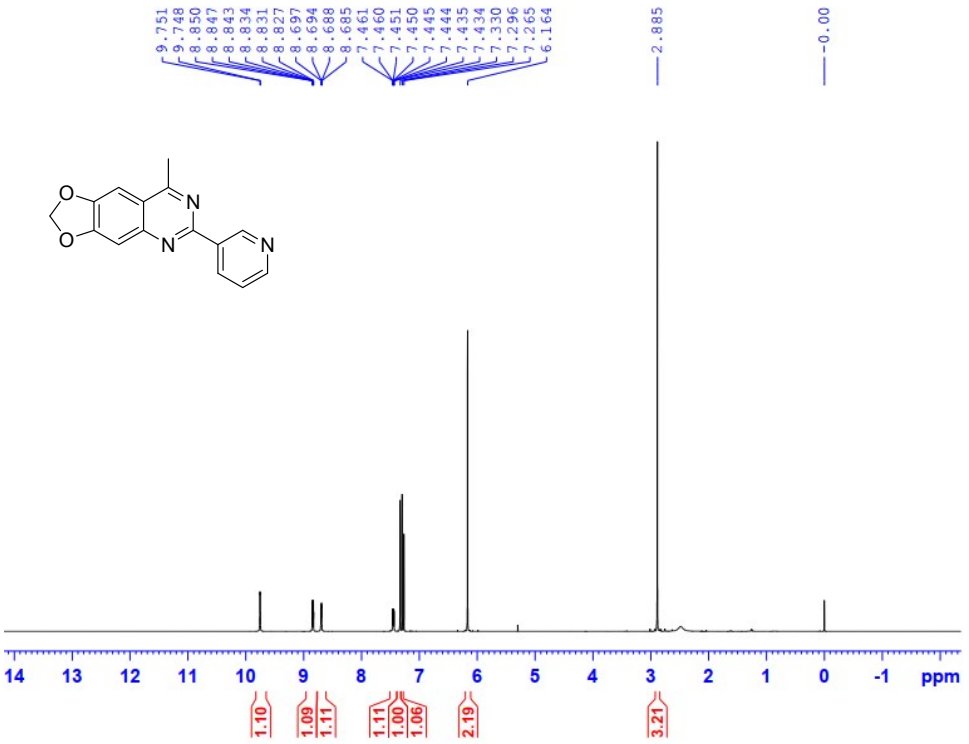
^{1}H and ^{13}C spectra of **3af**

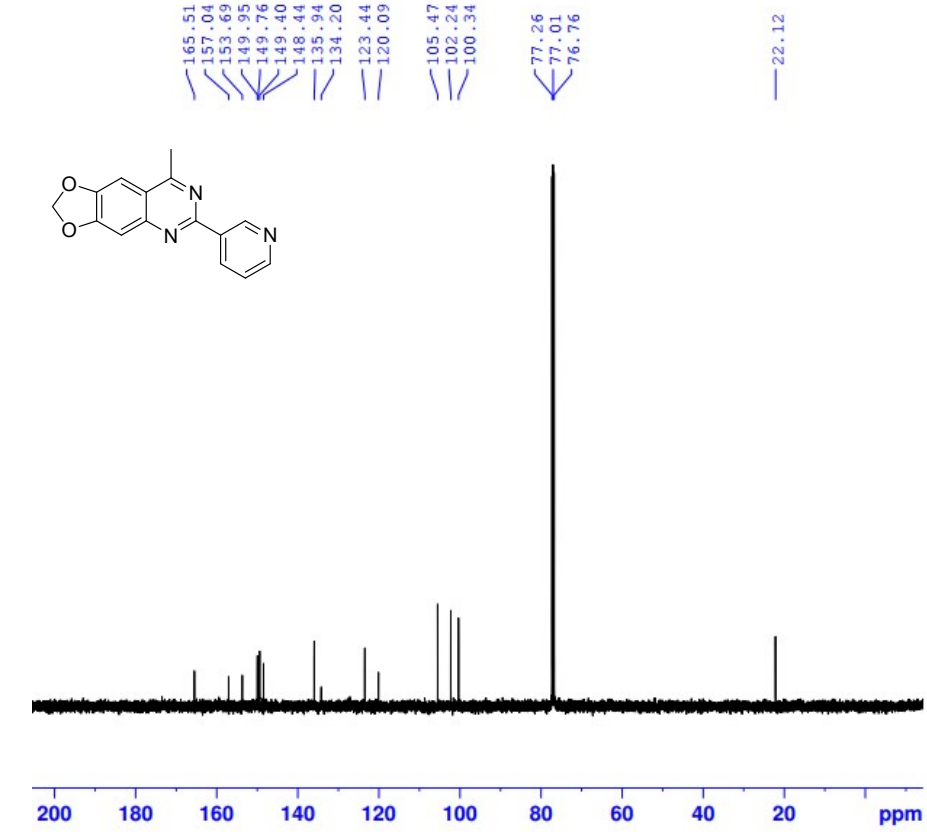


^{1}H and ^{13}C spectra of **3ag**

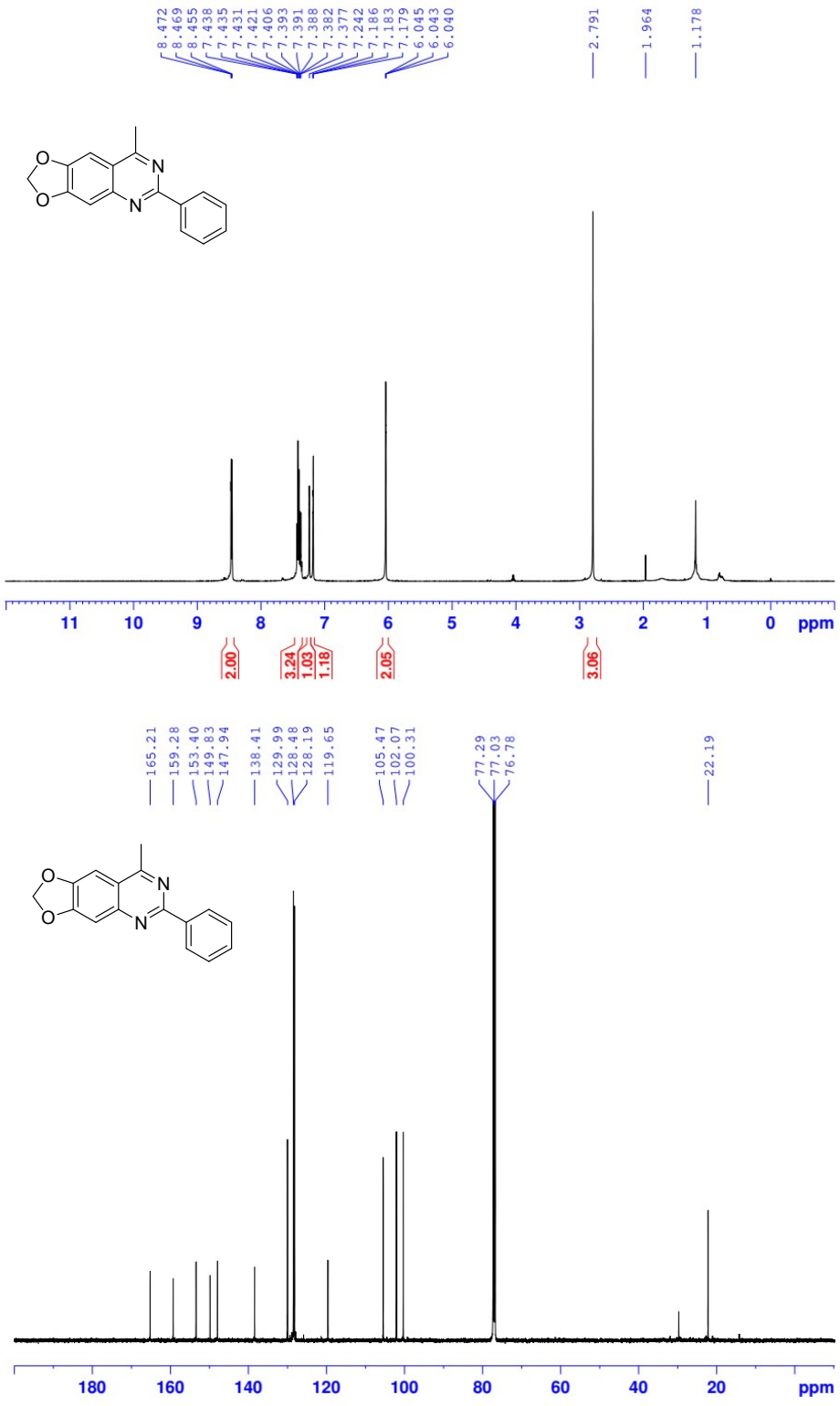


^{1}H and ^{13}C spectra of **3ba**

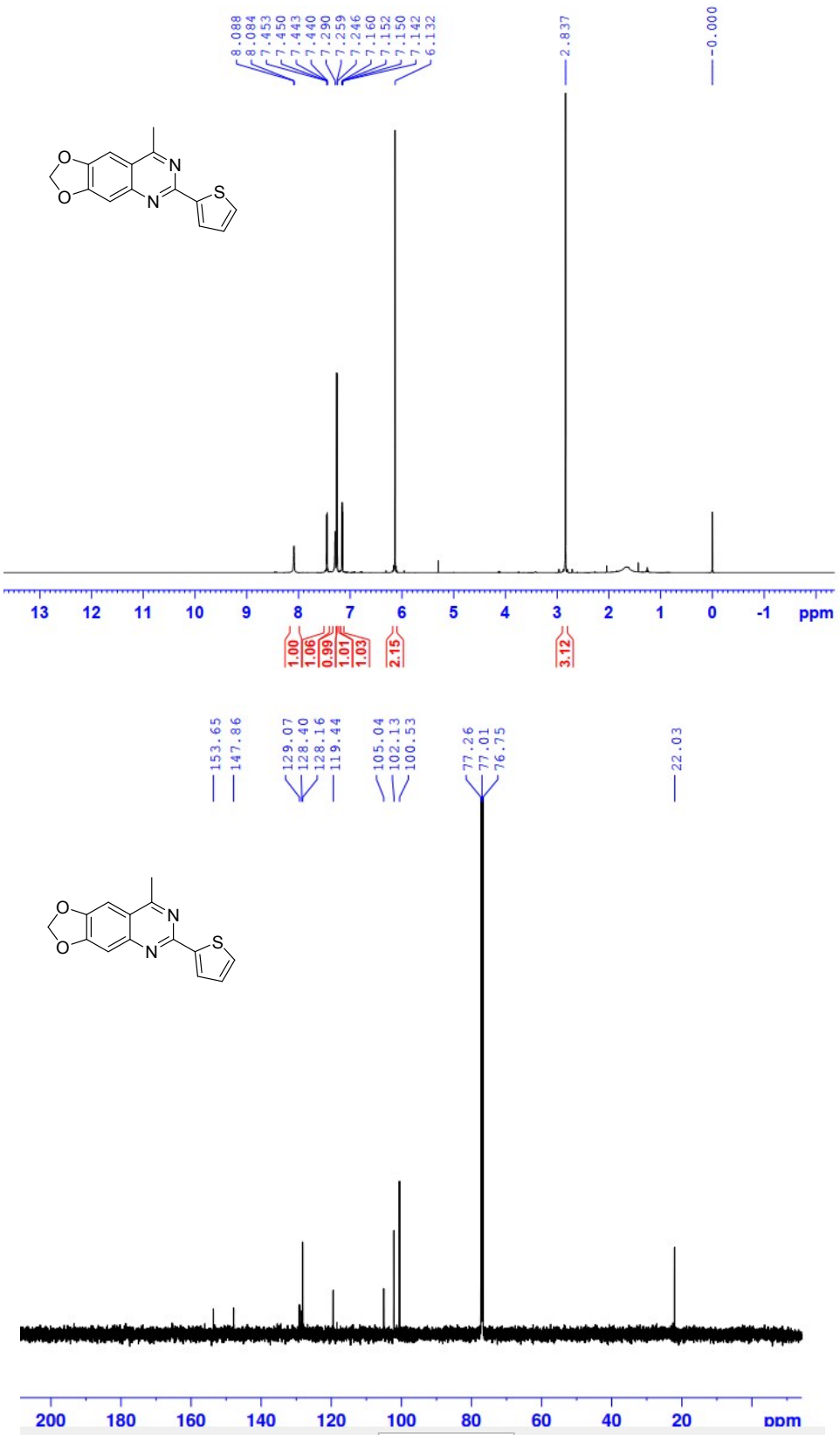




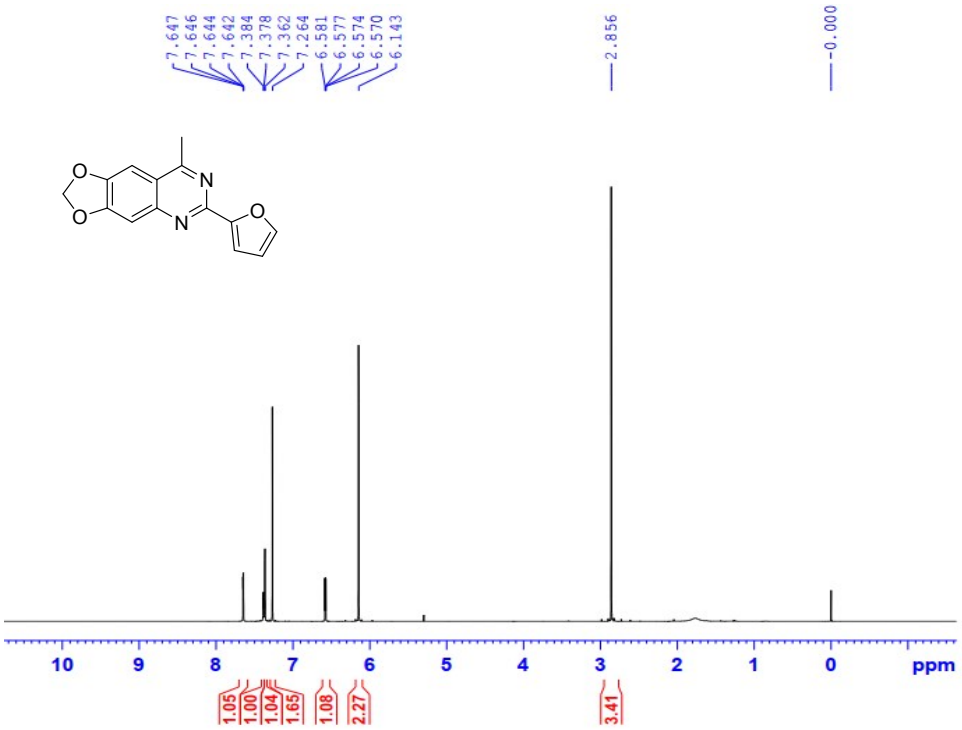
^{1}H and ^{13}C spectra of **3bb**

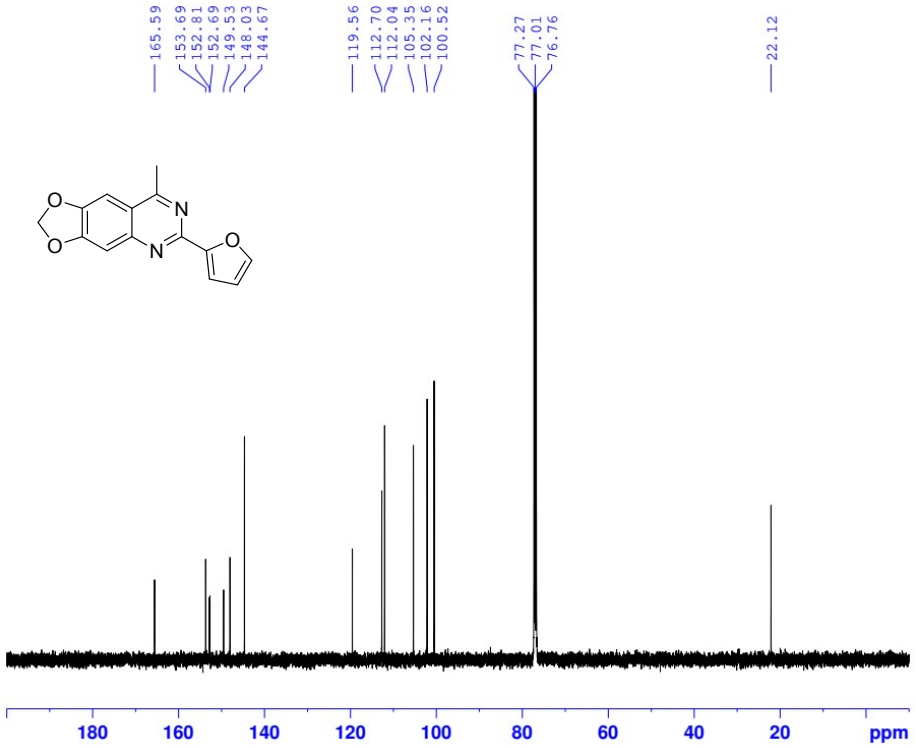


^{1}H and ^{13}C spectra of **3bc**

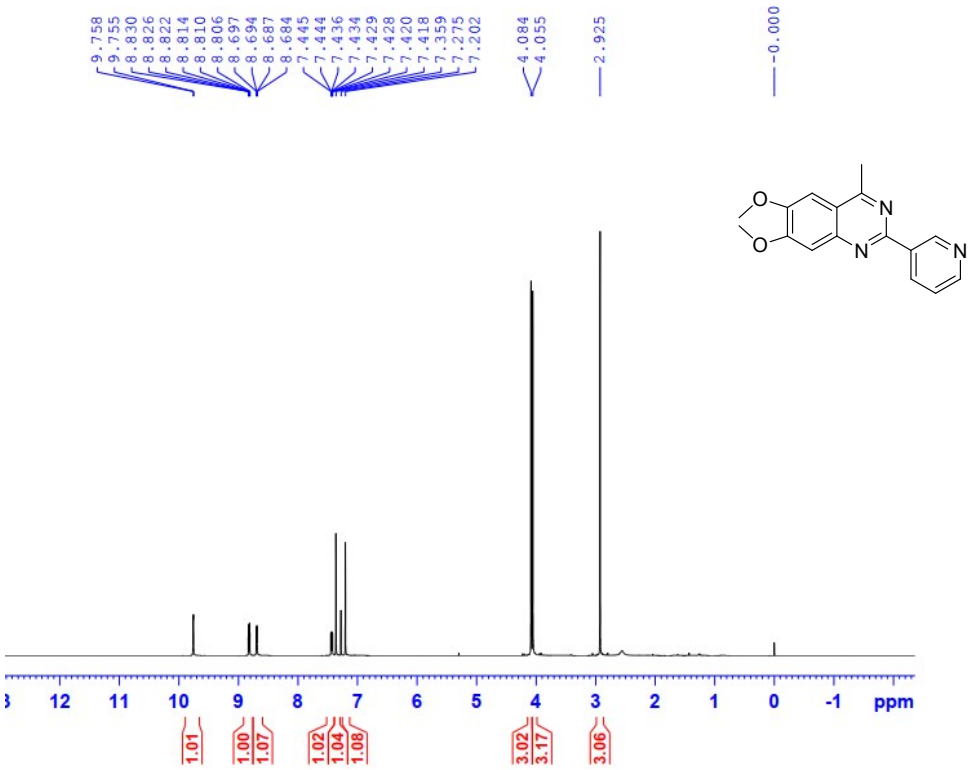


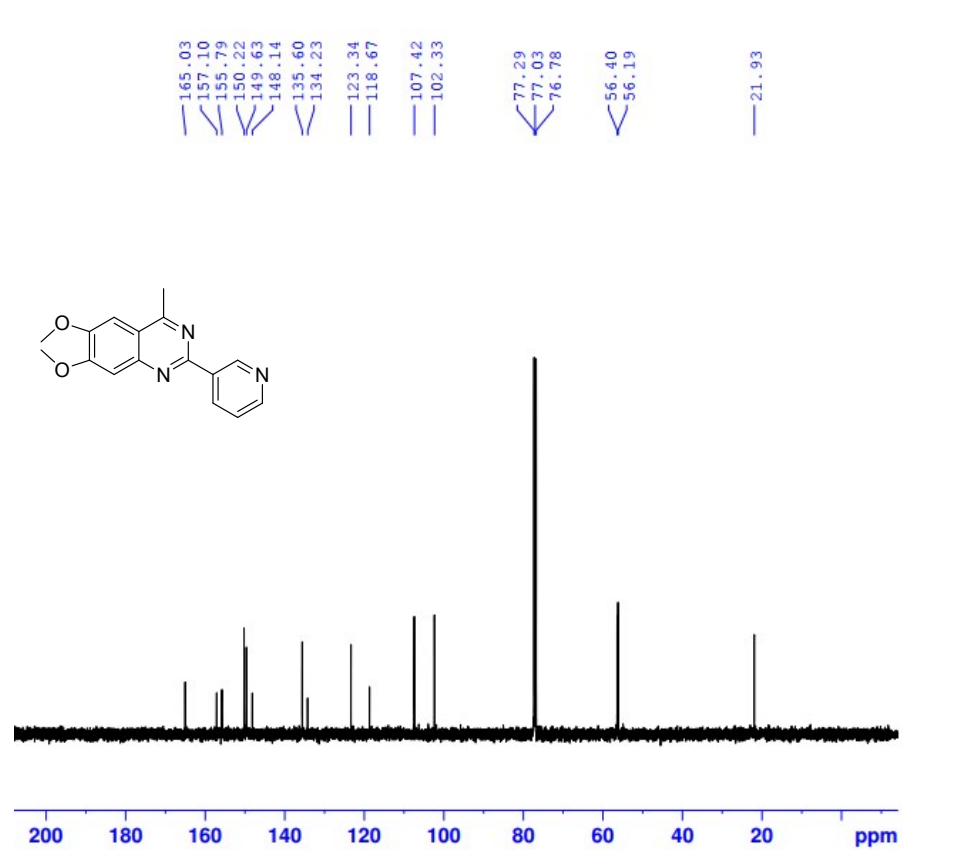
^{1}H and ^{13}C spectra of **3bd**



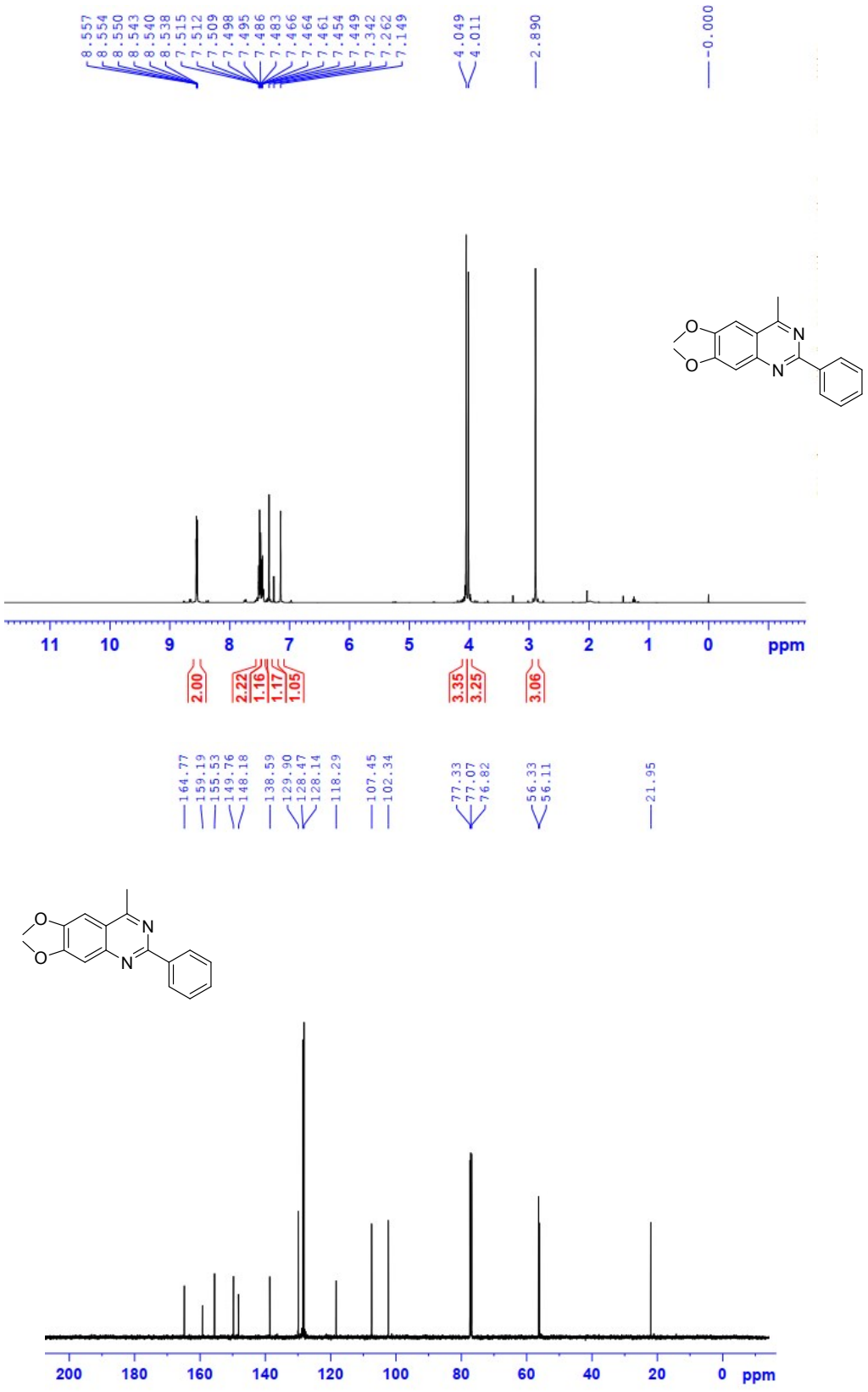


^{1}H and ^{13}C spectra of **3ca**

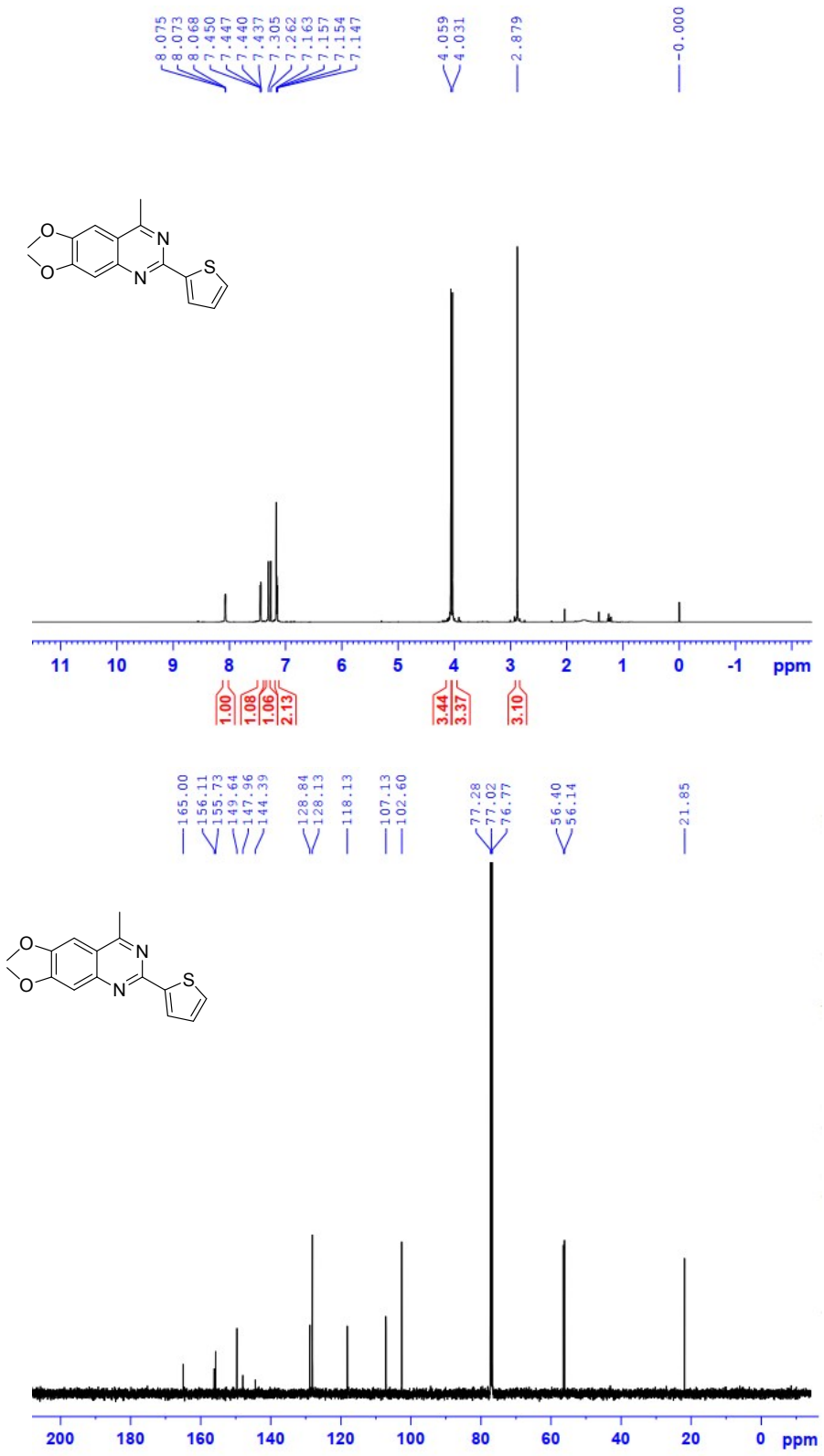




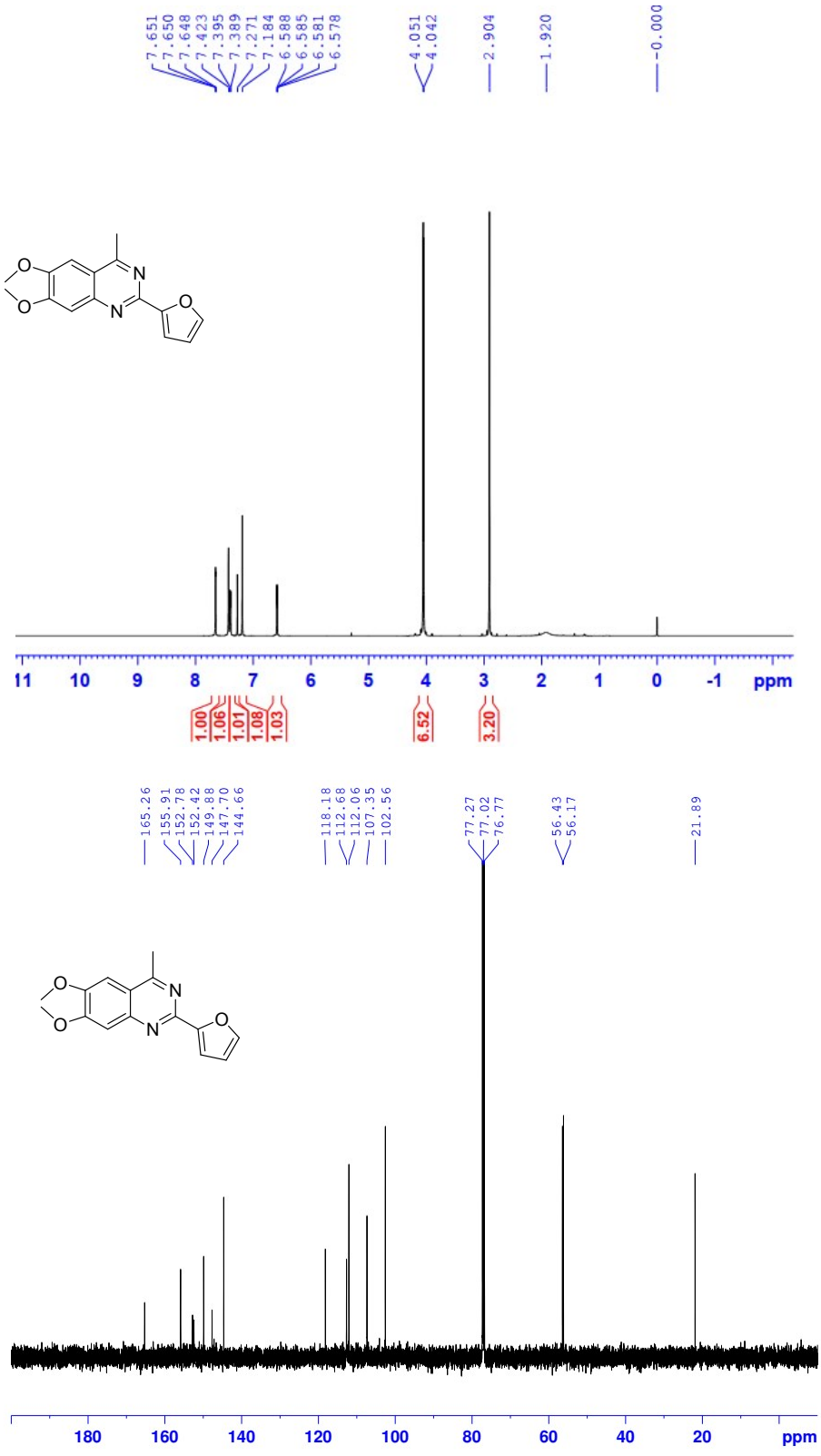
^{1}H and ^{13}C spectra of **3cb**



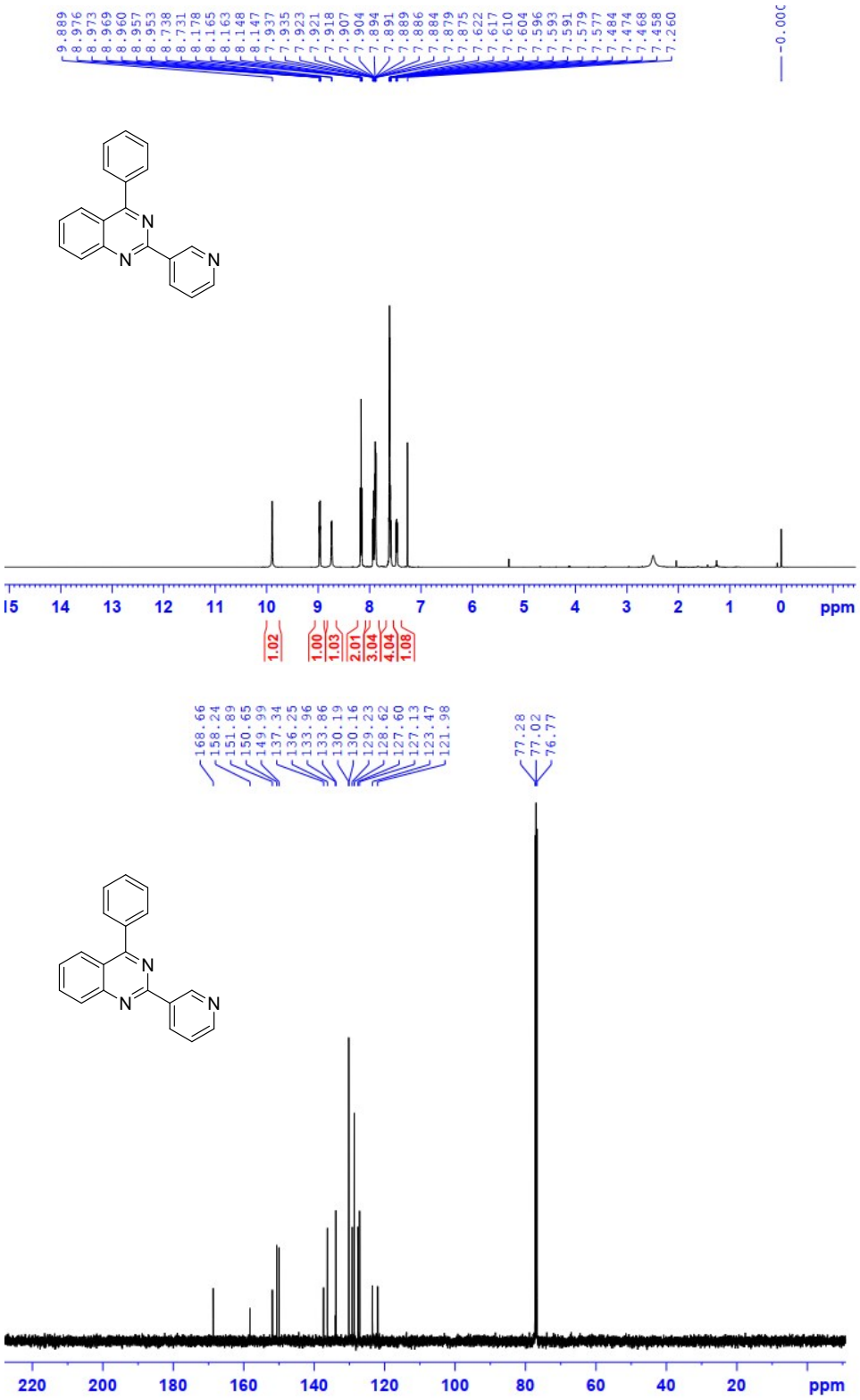
^{1}H and ^{13}C spectra of **3cc**



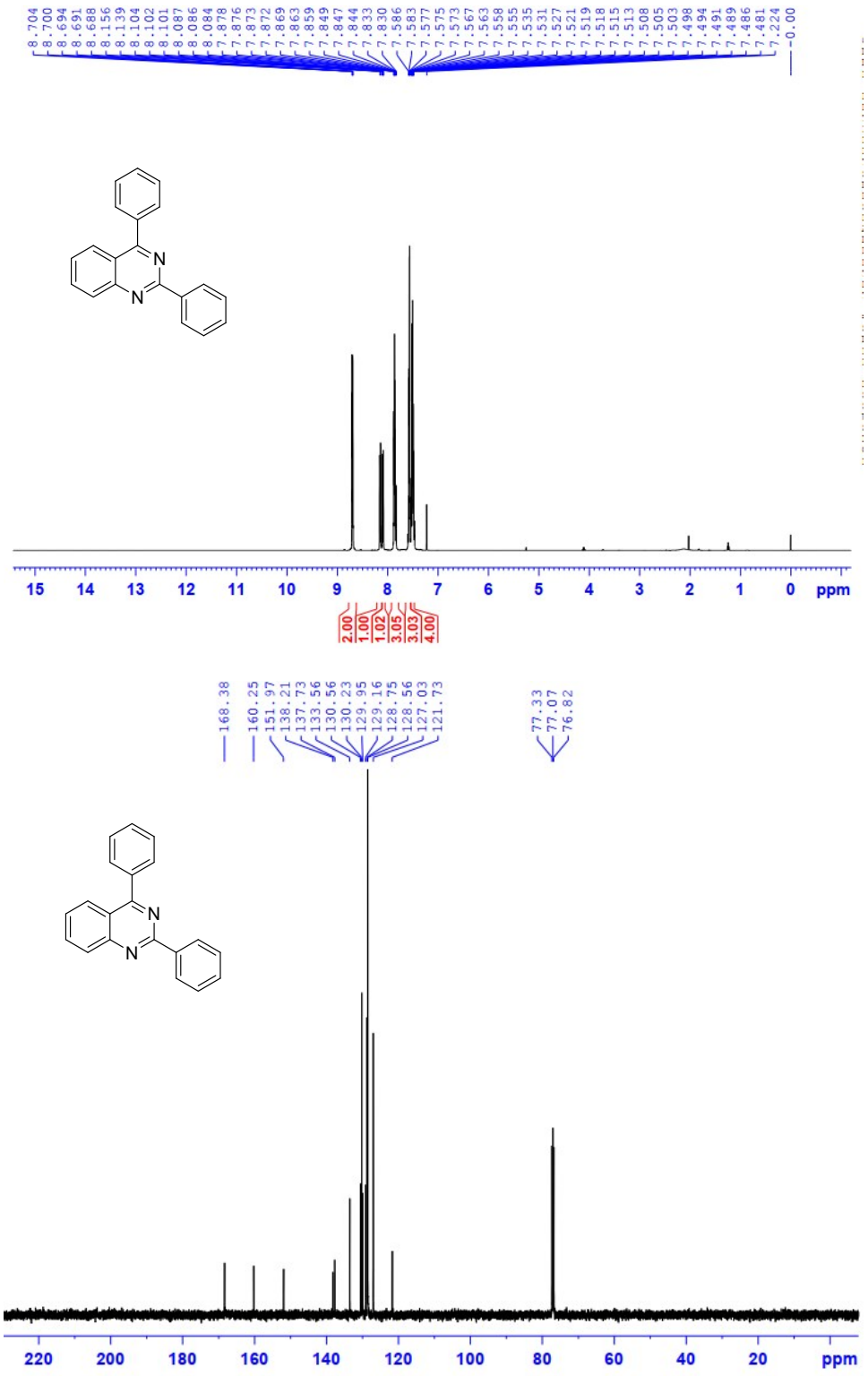
^{1}H and ^{13}C spectra of **3cd**



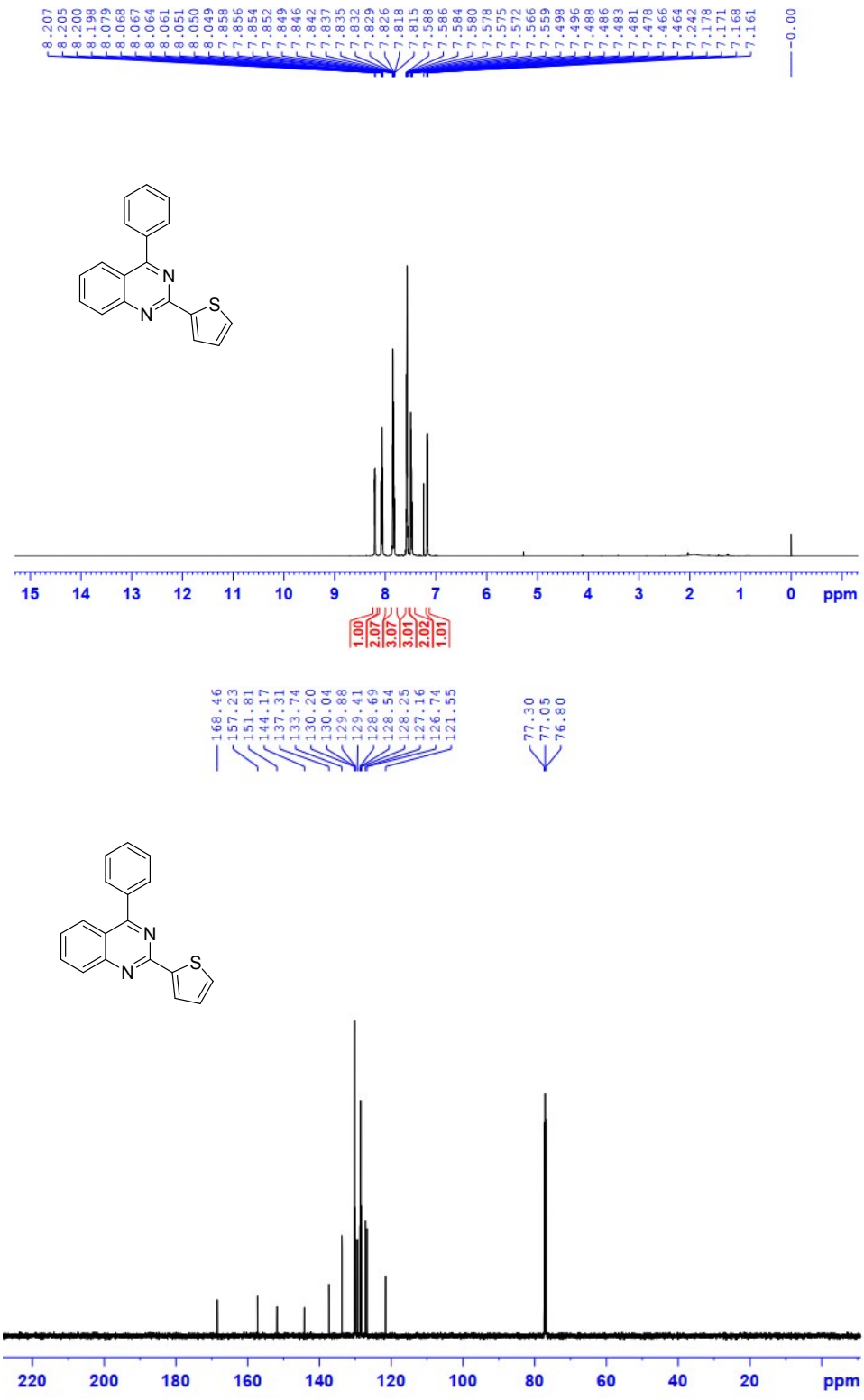
^{1}H and ^{13}C spectra of **3da**



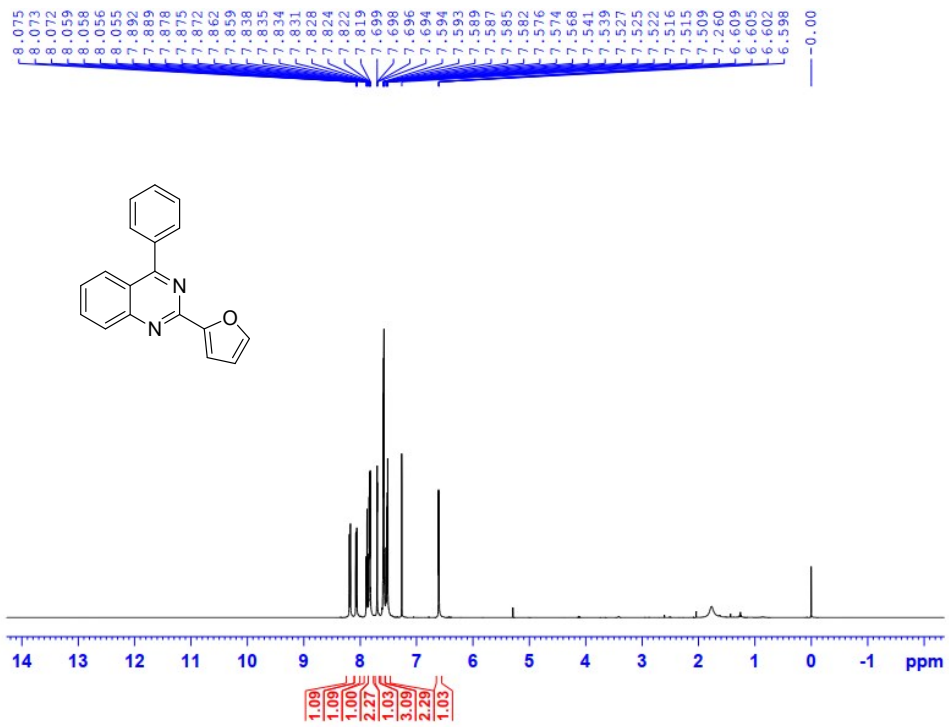
^{1}H and ^{13}C spectra of **3db**

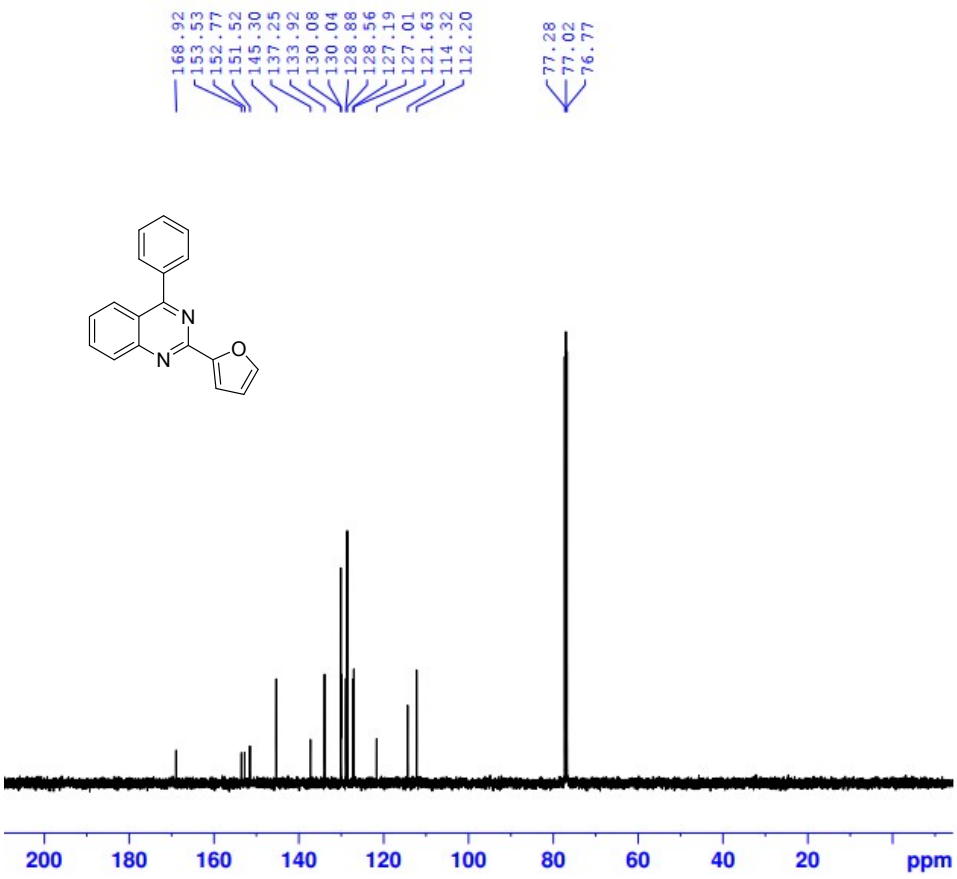


^{1}H and ^{13}C spectra of **3dc**



^{1}H and ^{13}C spectra of **3dd**

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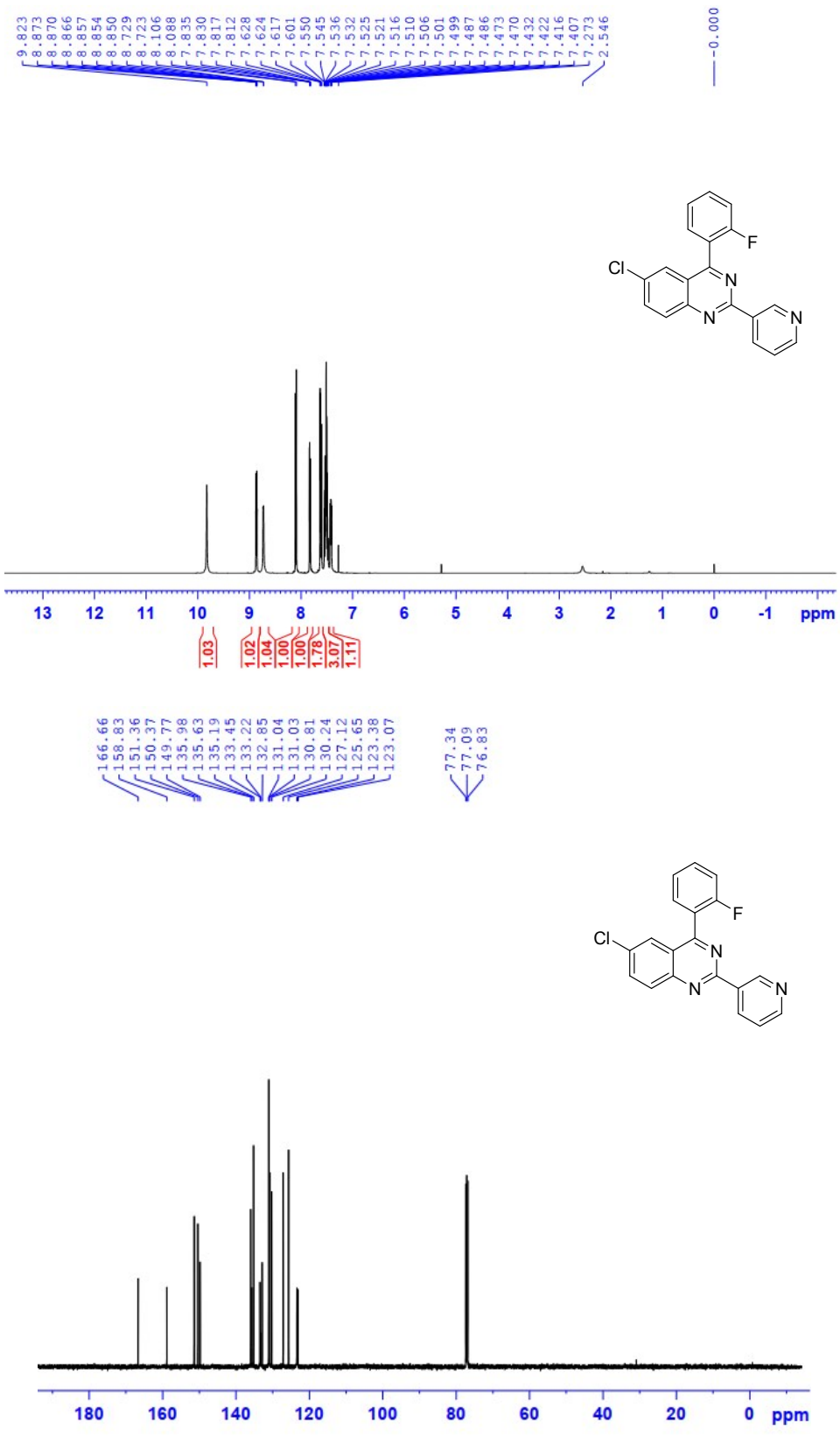
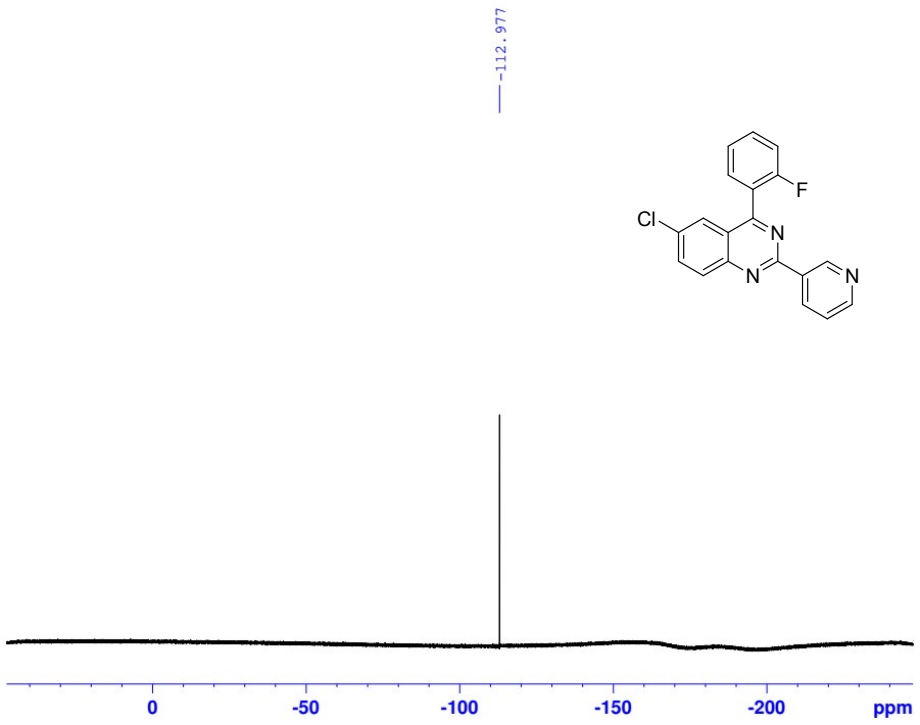
133.92
130.08
130.04
128.88
128.56
127.19
127.01
121.63
114.32
112.20
77.28
77.02
76.77
N
N
O
200
180
160
140
120
100
80
60
40
20
ppm

^{1}H, ^{13}C and ^{19}F spectra of **3ea**

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Cl
N
N
N
0
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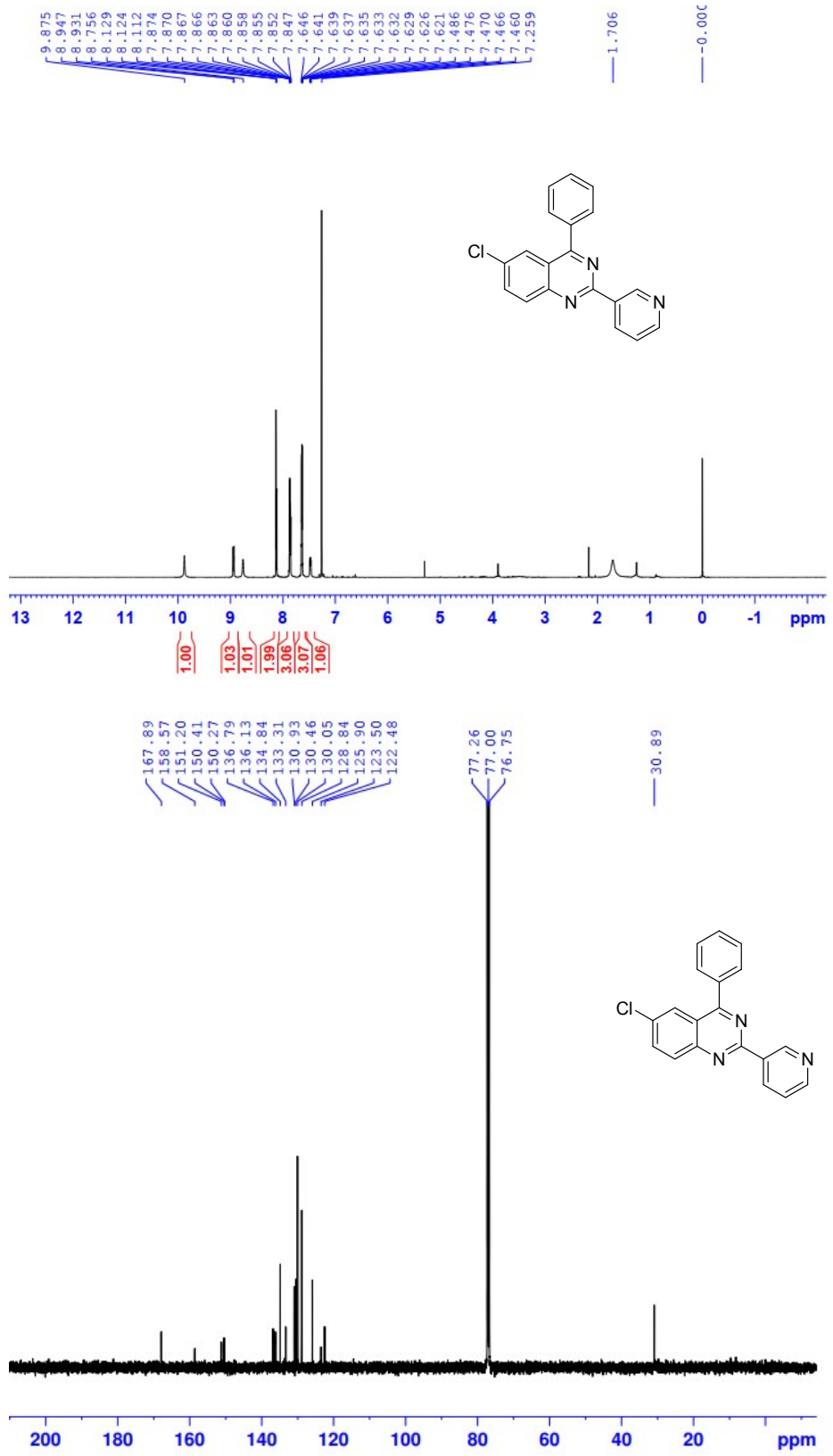
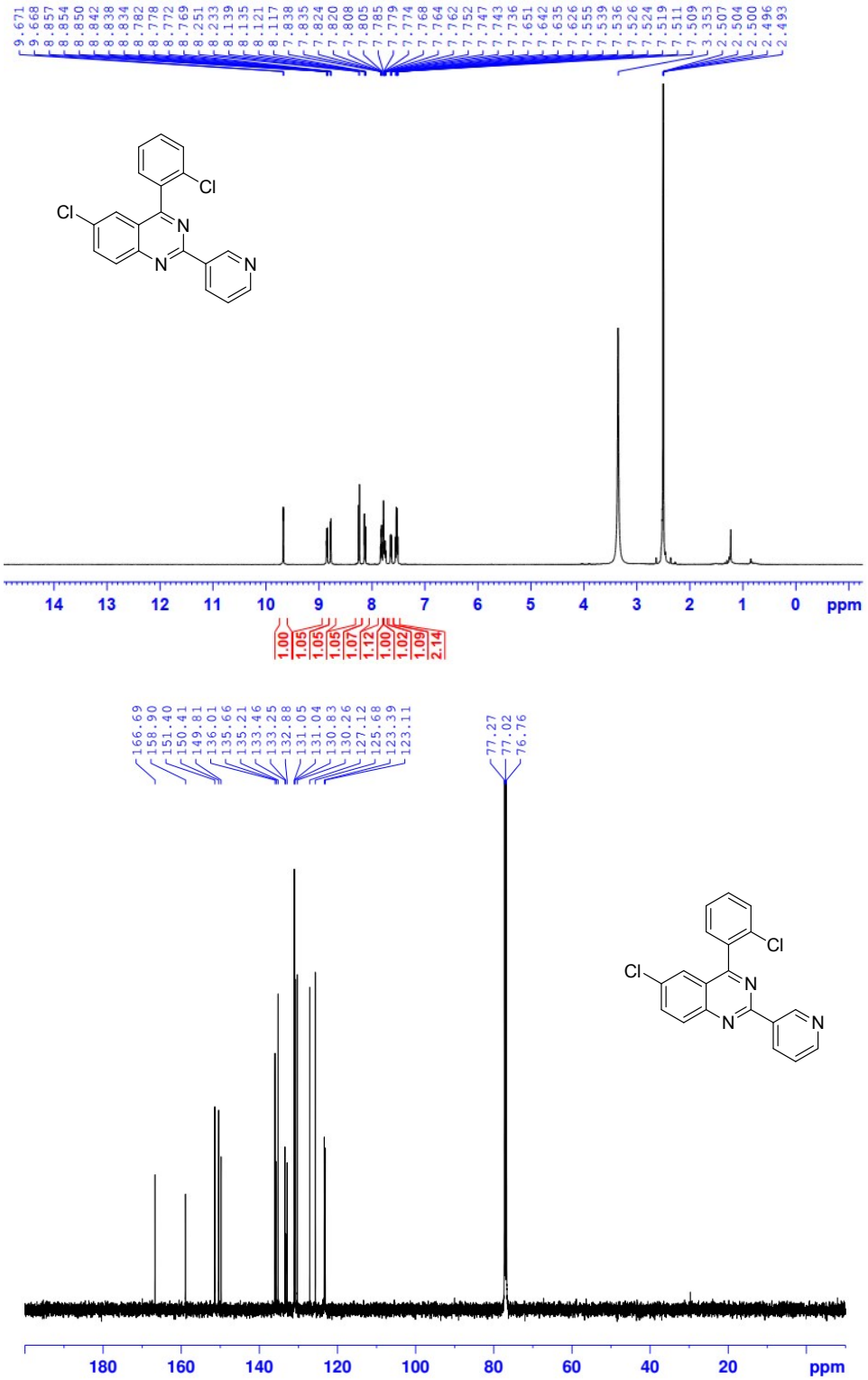
-100
-150
-200
ppm

^{1}H and ^{13}C spectra of **3fa**



^{1}H and ^{13}C spectra of **3ga**



^{1}H and ^{13}C spectra of **3ha**