Supporting Information

Redox-Neutral Couplings between Amides and Alkynes via Cobalt(III)-Catalyzed C-H Activation

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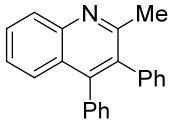
I. General Remarks

All chemicals were obtained from commercial sources and were used as received unless otherwise noted. Diphenylacetylenes,[1] $[Cp^*CoCl_2]_2$[2] were prepared by following literature reports. Reactions for synthesis of quinolines were carried out under air. NMR Spectra were recorded on a 400 MHz NMR spectrometer in the solvent indicated. The chemical shift is given in dimensionless δ values and is frequency referenced relative to TMS in ^{1}H and ^{13}C NMR spectroscopy. HRMS data were obtained on a Thermo Scientific LTQ Orbitrap Discovery spectrometer (Bremen, Germany). Column chromatography was performed on silica gel (300-400 mesh) using ethyl acetate (EA)/petroleum ether (PE) and ethyl acetate (EA)/triethylamine.

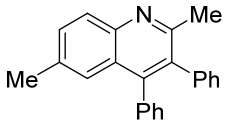
II. General procedures for the synthesis of compound 3

Amides (0.2 mmol), alkynes (0.24 mmol), $[Cp^*CoCl_2]_2$ (6 mol %), $AgNTf_2$ (0.2 mmol), and DCE (4.0 mL) were charged into the pressure tube. The reaction mixture was stirred under air at 130 ºC for 16 h. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using PE/EA and EA/triethylamine to afford the product **3**.



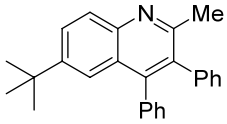
2-Methyl-3,4-diphenylquinoline (**3aa)**

3aa was obtained according to the general procedure in 93% yield (54.8 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, J = 8.4 Hz, 1H), 7.68 (t, J = 7.6 Hz, 1H), 7.50 (d, J = 8.3 Hz, 1H), 7.38 (t, J = 7.6 Hz, 1H), 7.28-7.14 (m, 6H), 7.10-7.05 (m, 4H), 2.54 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 147.1, 146.6, 138.7, 136.8, 134.1, 130.1, 130.1, 129.1, 128.6, 127.9, 127.7, 127.2, 126.8, 126.6, 126.3, 125.9, 25.5. HRMS: $[M + H]^+$ calculated for $C_{22}H_{18}N^+$: 296.1434, found: 296.1435.



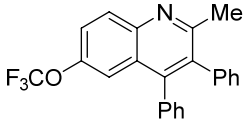
2,6-Dimethyl-3,4-diphenylquinoline **(3ba)**

3ba was obtained according to the general procedure in 62% yield (38.6 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 8.5 Hz, 1H), 7.52 (dd, J = 8.6, 1.6 Hz, 1H), 7.27-7.12 (m, 7H), 7.11-7.01 (m, 4H), 2.52 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 145.9, 145.7, 138.8, 137.0, 135.6, 134.0, 131.3, 130.1, 130.0, 128.4, 127.8, 127.6, 127.1, 126.7, 126.2, 125.3, 25.3, 21.7. HRMS: $[M + H]^+$ calculated for $C_{23}H_{20}N^+$: 310.1590, found: 310.1594.



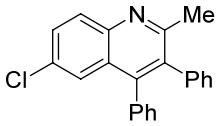
6-(Tert-butyl)-2-methyl-3,4-diphenylquinoline (**3ca)**

3ca was obtained according to the general procedure in 94% yield (66.1 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.9 Hz, 1H), 7.79 (dd, J = 8.9, 2.1 Hz, 1H), 7.43 (d, J = 1.7 Hz, 1H), 7.27-7.13 (m, 6H), 7.12-7.07 (m, 2H), 7.07-7.02 (m, 2H), 2.52 (s, 3H), 1.27 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.1, 148.6, 146.6, 145.6, 139.0, 136.9, 134.0, 130.2, 130.1, 128.2, 128.0, 127.9, 127.6, 127.2, 126.7, 125.8, 121.6, 34.9, 31.1, 25.4. HRMS: $[M + H]^+$ calculated for $C_{23}H_{17}F_3NO^+$: 352.2060, found: 352.2061.



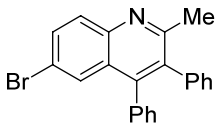
2-Methyl-3,4-diphenyl-6-(trifluoromethoxy)quinoline (**3da**)

3da was obtained according to the general procedure in 63% yield (47.5 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, J = 9.0 Hz, 1H), 7.56 (d, J = 8.5 Hz, 1H), 7.32-7.22 (m, 7H), 7.13-7.00 (m, 4H), 2.54 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 146.6, 146.56, 146.5, 145.4, 138.2, 136.0, 135.0, 130.8, 130.0, 129.9, 128.0, 127.9, 127.6, 127.1, 126.7, 123.0, 117.5, 25.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ - 57.79. HRMS: $[M + H]^+$ calculated for $C_{23}H_{17}F_3NO^+$: 380.1257, found: 380.1257.



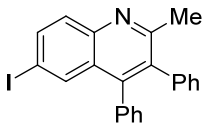
6-Chloro-2-methyl-3,4-diphenylquinoline (**3ea**)

3ea was obtained according to the general procedure in 88% yield (58.0 mg). brown solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 8.9 Hz, 1H), 7.61 (dd, J = 8.9, 2.2 Hz, 1H), 7.46 (d, J = 2.2 Hz, 1H), 7.29-7.14 (m, 6H), 7.10-7.01 (m, 4H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.3, 145.9, 145.5, 138.3, 136.1, 134.9, 131.7, 130.4, 130.02, 130.0, 129.9, 128.0, 127.9, 127.6, 127.1, 127.0, 125.4, 25.5. HRMS: $[M + H]^+$ calculated for $C_{22}H_{17}ClN^+$: 330.1044, found: 330.1045.



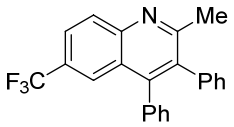
6-Bromo-2-methyl-3,4-diphenylquinoline (**3fa**), 79% yield

3fa was obtained according to the general procedure in 79% yield (59.1 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 8.9 Hz, 1H), 7.74 (dd, J = 8.9, 2.1 Hz, 1H), 7.63 (d, J = 2.1 Hz, 1H), 7.29-7.14 (m, 6H), 7.10-7.01 (m, 4H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 145.8, 145.7, 138.2, 136.0, 134.9, 132.6, 130.5, 130.0, 129.9, 128.7, 128.0, 127.9, 127.6, 127.6, 127.0, 119.9, 25.5. HRMS: $[M + H]^+$ calculated for $C_{22}H_{17}BrN^+$: 374.0539, found: 374.0539.



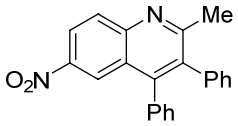
6-Iodo-2-methyl-3,4-diphenylquinoline (**3ga**)

3ga was obtained according to the general procedure in 88% yield (74.5 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.90 (m, 1H), 7.88-7.79 (m, 2H), 7.31-7.13 (m, 6H), 7.11-6.98 (m, 4H), 2.51 (s, 3H) ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 146.1, 145.52, 138.2, 137.9, 136.0, 135.3, 134.8, 130.5, 130.1, 129.9, 128.2, 128.0, 127.9, 127.6, 127.0, 91.6, 25.6. HRMS: $[M + H]^+$ calculated for $C_{22}H_{17}IN^+$: 422.0400, found: 422.0400.



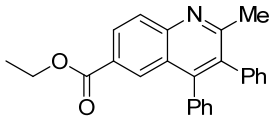
2-Methyl-3,4-diphenyl-6-(trifluoromethyl)quinoline (**3ha**)

3ha was obtained according to the general procedure in 72% yield (51.8 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, J = 8.7 Hz, 1H), 7.86 (dd, J = 8.8, 1.6 Hz, 1H), 7.82 (s, 1H), 7.31-7.17 (m, 6H), 7.11-7.03 (m, 4H), 2.57 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.5, 148.0, 147.4, 138.0, 135.7, 135.3, 128.0, 129.9, 129.8, 128.0, 127.9, 127.7, 127.6 (q, $J_{C\text{-}F}$ = 32.5 Hz), 127.1, 125.5, 124.8 (q, $J_{C\text{-}F}$ = 2.9 Hz), 124.1 (q, $J_{C\text{-}F}$ = 273.6 Hz), 124.5 (q, $J_{C\text{-}F}$ = 4.5 Hz), 25.6. HRMS: $[M + H]^+$ calculated for $C_{23}H_{17}F_3N^+$: 364.1308, found: 364.1309.



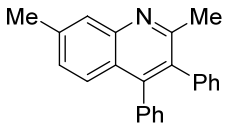
2-Methyl-6-nitro-3,4-diphenylquinoline (**3ia**)

3ia was obtained according to the general procedure in 34% yield (23.0 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49-8.42 (m, 2H), 8.21 (d, J = 9.0 Hz, 1H), 7.33-7.21 (m, 6H), 7.11-7.04 (m, 4H), 2.59 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 149.2, 148.4, 145.2, 137.6, 136.0, 135.2, 130.5, 130.0, 129.7, 128.2, 128.19, 128.1, 127.4, 125.6, 123.8, 122.7, 25.9. HRMS: $[M + H]^+$ calculated for $C_{22}H_{17}N_2O_2^+$: 341.1285, found: 341.1285.



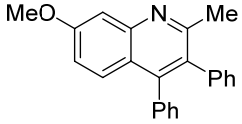
Ethyl 2-methyl-3,4-diphenylquinoline-6-carboxylate (**3ja**)

3ja was obtained according to the general procedure in 90% yield (66.3 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30-8.27 (m, 2H), 8.13 (d, J = 8.5 Hz, 1H), 7.25-7.18 (m, 6H), 7.10-7.05 (m, 4H), 4.34 (q, J = 6.5 Hz, 2H), 2.56 (s, 3H), 1.34 (t, J = 6.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 160.5, 149.1, 147.9, 138.3, 136.1, 135.0, 130.2, 130.1, 130.0, 129.1, 128.8, 128.1, 128.0, 127.9, 127.7, 127.1, 125.8, 61.3, 25.9, 14.4. HRMS: $[M + H]^+$ calculated for $C_{25}H_{22}NO_2^+$: 368.1645, found: 368.1647.



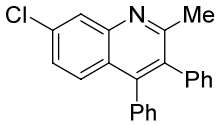
2,7-Dimethyl-3,4-diphenylquinoline (**3ka)**

3ka was obtained according to the general procedure in 75% yield (46.5 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.38 (d, J = 8.5 Hz, 1H), 7.28-7.12 (m, 7H), 7.10-7.02 (m, 4H), 2.55 (s, 3H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 147.3, 146.4, 139.4, 138.8, 137.0, 133.3, 130.2, 130.1, 128.1, 127.9, 127.8, 127.7, 127.2, 126.8, 126.3, 124.3, 25.5, 21.7. HRMS: $[M + H]^+$ calculated for $C_{23}H_{20}N^+$: 310.1590, found: 310.1589.



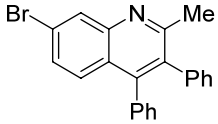
7-Methoxy-2-methyl-3,4-diphenylquinoline (**3la**)

3la was obtained according to the general procedure in 58% yield (37.7 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, J = 2.5 Hz, 1H), 7.38 (d, J = 9.2 Hz, 1H), 7.24-7.12 (m, 6H), 7.08-7.02 (m, 5H), 3.97 (s, 3H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.5, 158.0, 148.7, 146.6, 138.7, 136.9, 132.0, 130.2, 130.0, 127.8, 127.7, 127.6, 127.1, 126.7, 121.2, 118.7, 106.7, 55.5, 25.2. HRMS: $[M + H]^+$ calculated for $C_{23}H_{20}NO^+$: 326.1539, found: 326.1540.



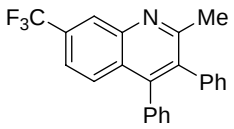
7-Chloro-2-methyl-3,4-diphenylquinoline (**3ma**)

3ma was obtained according to the general procedure in 66% yield (43.4 mg). pink solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, J = 1.9 Hz, 1H), 7.43 (d, J = 8.9 Hz, 1H), 7.31 (dd, J = 8.9, 1.8 Hz, 1H), 7.28-7.14 (m, 6H), 7.07-7.05 (m, 4H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.2, 147.5, 146.6, 138.3, 136.3, 135.0, 134.3, 130.0, 129.9, 128.1, 128.0, 127.8, 127.6, 127.5, 127.0, 126.8, 124.8, 25.5. HRMS: $[M + H]^+$ calculated for $C_{22}H_{17}ClN^+$: 330.1044, found: 330.1045.



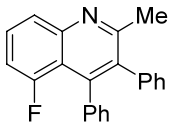
7-Bromo-2-methyl-3,4-diphenylquinoline (**3na**)

3na was obtained according to the general procedure in 60% yield (44.6 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (d, J = 1.8 Hz, 1H), 7.45 (dd, J = 8.9, 1.5 Hz, 1H), 7.36-7.34 (m, 1H), 7.27-7.15 (m, 6H), 7.07-7.03 (m, 4H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.2, 147.7, 146.7, 138.3, 136.3, 134.4, 131.0, 130.0, 129.9, 129.3, 128.1, 128.0, 127.9, 127.5, 127.0, 125.1, 123.3, 25.5. HRMS: $[M + H]^+$ calculated for $C_{22}H_{17}BrN^+$: 374.0539, found: 374.0541.



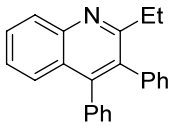
2-Methyl-3,4-diphenyl-7-(trifluoromethyl)quinoline (**3oa**)

3oa was obtained according to the general procedure in 62% yield (45.4 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (s, 1H), 7.62 (d, J = 8.8 Hz, 1H), 7.55 (dd, J = 8.8, 1.7 Hz, 1H), 7.30-7.16 (m, 3H), 7.09-7.05 (m, 2H), 2.57 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 146.5, 146.1, 138.0, 136.1, 135.9, 130.8 (q, $J_{C\text{-}F}$ = 32.7 Hz), 130.0, 129.8, 128.1, 128.01, 128.0 127.9, 127.6, 127.2, 126.5 (q, $J_{C\text{-}F}$ = 4.4 Hz), 124.0 (q, $J_{C\text{-}F}$ = 273.4 Hz), 121.4 (q, $J_{C\text{-}F}$ = 3.0 Hz), 25.6. HRMS: $[M + H]^+$ calculated for $C_{23}H_{17}F_3N^+$: 364.1308, found: 364.1309.



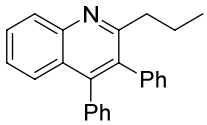
5-Fluoro-2-methyl-3,4-diphenylquinoline (**3pa**), major : minor = 1.2 : 1

3pa was obtained according to the general procedure in 81% yield (50.5 mg). yellow solid; selected major signals,: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 8.5 Hz, 1H), 7.74 (dd, J = 10.1, 2.6 Hz, 1H), 7.60 (td, J = 8.1, 5.4 Hz, 1H), 7.48 (dd, J = 9.2, 6.2 Hz, 1H), 7.28-7.12 (m, 13H), 7.11-6.97 (m, 9H), 2.53 (s, 2H), 2.50 (s, 3H). Selected major signals ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0 (d, $J_{C\text{-}F}$ = 250.6 Hz), 159.9, 159.3, 157.3, 148.5, 148.1 (d, $J_{C\text{-}F}$ = 12.8 Hz), 138.4, 136.6, 133.5 (d, $J_{C\text{-}F}$ = 2.6 Hz), 130.1, 128.9, 128.87, 127.9, 127.1, 125.1 (d, $J_{C\text{-}F}$ = 4.2 Hz), 116.1 (d, $J_{C\text{-}F}$ = 24.9 Hz), 112.3 (d, $J_{C\text{-}F}$ = 20.2 Hz), 25.5. HRMS: $[M + H]^+$ calculated for $C_{22}H_{17}FN^+$: 314.1340, found: 314.1338.



2-Ethyl-3,4-diphenylquinoline (**3qa**)

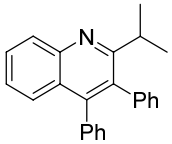
3qa was obtained according to the general procedure in 78% yield (48.0 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, J = 8.4 Hz, 1H), 7.69-7.65 (m, 1H), 7.48 (dd, J = 8.4, 0.8 Hz, 1H), 7.38-7.34 (m, 1H), 7.26-7.12 (m, 6H), 7.08-7.06 (m, 4H), 2.84 (q, J = 7.5 Hz, 2H), 1.23 (t, J = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.3, 147.3, 146.9, 138.5, 137.0, 133.8, 130.3, 130.1, 129.1, 128.9, 127.8, 127.7, 127.2, 126.8, 126.6, 126.2, 125.9, 30.7, 13.8. HRMS: $[M + H]^+$ calculated for $C_{23}H_{20}N^+$: 310.1590, found: 310.1592.



3,4-Diphenyl-2-propylquinoline (**3ra**)

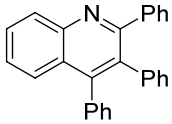
3ra was obtained according to the general procedure in 81% yield (52.7 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, J = 8.4 Hz, 1H), 7.67 (t, J = 7.6 Hz, 1H), 7.48 (d, J = 8.2 Hz, 1H), 7.37 (t, J = 7.6 Hz, 1H), 7.26-7.12 (m, 6H), 7.09-7.05 (m, 4H), 2.78 (t, J = 7.8 Hz, 2H),

1.78-1.65 (m, 2H), 0.86 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.2, 147.2, 146.8, 138.5, 137.0, 133.9, 130.3, 130.1, 129.0, 128.8, 127.63, 127.6, 127.1, 126.7, 126.6, 126.1, 125.8, 39.3, 22.9, 14.2. HRMS: $[M + H]^+$ calculated for $C_{24}H_{22}N^+$: 324.1747, found: 324.1747.



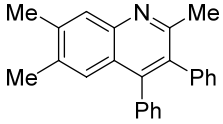
2-Isopropyl-3,4-diphenylquinoline (**3sa**)

3sa was obtained according to the general procedure in 61% yield (39.7 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, J = 8.4 Hz, 1H), 7.70-7.63 (m, 1H), 7.45 (d, J = 7.7 Hz, 1H), 7.38-7.33 (m, 1H), 7.24-7.15 (m, 6H), 7.09-7.04 (m, 4H), 3.14 (hept, J = 6.8 Hz, 1H), 1.29 (d, J = 6.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.6, 147.5, 146.6, 138.7, 137.3, 133.3, 130.3, 130.1, 129.2, 128.7, 127.6, 127.6, 127.0, 126.6, 126.5, 126.0, 125.7, 32.8, 22.4. HRMS: $[M + H]^+$ calculated for $C_{24}H_{22}N^+$: 324.1747, found: 324.1748.



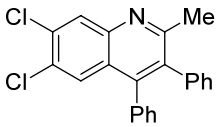
2,3,4-Triphenylquinoline (**3ta**)

3ta was obtained according to the general procedure in 60% yield (42.6 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (d, J = 8.4 Hz, 1H), 7.75-7.66 (m, 1H), 7.58 (d, J = 8.3 Hz, 1H), 7.46-7.35 (m, 3H), 7.30-7.16 (m, 6H), 7.13-7.11 (m, 2H), 7.02-6.94 (m, 3H), 6.90-6.87 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.9, 147.5, 147.2, 141.1, 138.2, 136.8, 132.8, 131.2, 130.2, 129.8, 129.6, 129.2, 127.7, 127.5, 127.5, 127.2, 127.1, 126.5, 126.5, 126.4, 126.2. HRMS: $[M + H]^+$ calculated for $C_{27}H_{20}N^+$: 358.1590, found: 358.1593.



2,6,7-Trimethyl-3,4-diphenylquinoline (**3wa**), major : minor = 17 : 1

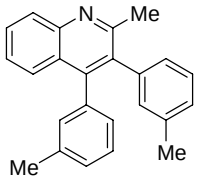
3wa was obtained according to the general procedure in 54% yield (34.9 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (s, 1H), 7.30-7.11 (m, 7H), 7.08-7.03 (m, 4H), 2.50 (s, 3H), 2.46 (s, 3H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 146.2, 145.7, 139.3, 139.0, 137.1, 135.7, 133.3, 130.2, 130.15, 128.2, 127.8, 127.7, 127.1, 126.7, 125.7, 124.7, 25.3, 20.3, 20.2. HRMS: $[M + H]^+$ calculated for $C_{24}H_{22}N^+$: 324.1747, found: 324.1749.



6,7-Dichloro-2-methyl-3,4-diphenylquinoline (**3xa),** major : minor = 20 : 1

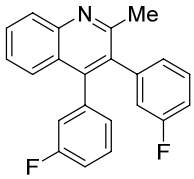
3xa was obtained according to the general procedure in 68% yield (49.3 mg). yelow solid; ^{1}H

NMR (400 MHz, $CDCl_3$) δ 8.22 (s, 1H), 7.58 (s, 1H), 7.33-7.15 (m, 6H), 7.06-7.02 (m, 4H), 2.51 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 145.8, 145.7, 138.0, 135.7, 135.1, 133.5, 130.3, 129.9, 129.8, 129.8, 128.1, 128.0, 127.8, 127.4, 127.2, 125.8, 25.5. HRMS: $[M + H]^+$ calculated for $C_{22}H_{16}Cl_2N^+$: 364.0654, found: 364.0654.



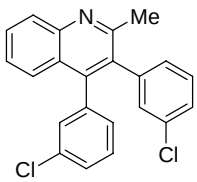
2-Methyl-3,4-di-*m*-tolylquinoline (**3ab**)

3ab was obtained according to the general procedure in 67% yield (43.4 mg). yelow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, J = 8.4 Hz, 1H), 7.79-7.70 (m, 1H), 7.59 (d, J = 7.7 Hz, 1H), 7.47-7.43 (m, 1H), 7.25-7.14 (m, 2H), 7.10-7.04 (m, 2H), 7.02-6.90 (m, 4H), 2.63 (s, 3H), 2.32 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 147.0, 146.7, 138.6, 137.3, 137.2, 137.1, 137.0, 136.7, 134.1, 134.1, 130.8, 130.74, 130.72, 130.7, 128.9, 128.5, 127.8, 127.8, 127.7, 127.6, 127.5, 127.4, 127.2, 127.1, 127.09, 127.06, 126.7, 126.5, 126.4, 125.7, 25.4, 21.3, 21.27. HRMS: $[M + H]^+$ calculated for $C_{24}H_{22}N^+$: 324.1747, found: 324.1750.



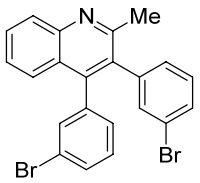
3,4-Bis(3-fluorophenyl)-2-methylquinoline (**3ac**)

3ac was obtained according to the general procedure in 85% yield (56.6 mg). yelow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, J = 8.4 Hz, 1H), 7.77 (t, J = 7.2 Hz, 1H), 7.57-7.44 (m, 2H), 7.35-7.25 (m, 2H), 7.06-6.83 (m, 6H), 2.62 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4 (d, $J_{C\text{-}F}$ = 247.9 Hz) 162.36 (d, $J_{C\text{-}F}$ =247.7 Hz), 162.2 (d, J = 247.5 Hz), 162.19 (d, $J_{C\text{-}F}$ = 248.2 Hz), 157.3, 147.2, 145.2 (d, $J_{C\text{-}F}$ = 3.1 Hz), 140.5 (d, $J_{C\text{-}F}$ = 7.8 Hz), 138.6(d, $J_{C\text{-}F}$ = 7.8 Hz), 132.7, 129.8, 129.7 (d, $J_{C\text{-}F}$ = 1.4 Hz), 129.6, 129.5, 129.4, 128.8, 126.3, 126.2, 125.8, 125.79, 125.77, 125.7, 117.03 (d, $J_{C\text{-}F}$ = 22.6 Hz), 117.0 (d, $J_{C\text{-}F}$ = 22.0 Hz), 116.9 (d, $J_{C\text{-}F}$ = 21.5 Hz), 114.51 (d, $J_{C\text{-}F}$ =21.4 Hz), 114.5 (d, $J_{C\text{-}F}$ =21.1 Hz), 114.1 (d, $J_{C\text{-}F}$ = 20.9 Hz), 25.2. HRMS: $[M + H]^+$ calculated for $C_{22}H_{16}F_2N^+$: 332.1245, found: 332.1248.



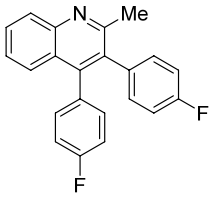
3,4-Bis(3-chlorophenyl)-2-methylquinoline (**3ad**)

3ad was obtained according to the general procedure in 94% yield (68.7 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, J = 8.4 Hz, 1H), 7.72-7.68 (m, 1H), 7.48-7.38 (m, 2H), 7.26-7.06 (m, 6H), 6.99-6.94 (m, 2H), 2.54 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3, 147.2, 145.2, 140.1, 138.3, 134.1, 134.0, 133.97, 133.9, 132.6, 130.0, 129.98, 129.92, 129.9, 129.6, 129.5, 129.4, 129.36, 129.2, 128.9, 128.3, 128.2, 127.8, 127.4, 126.4, 126.3, 125.7, 25.4. HRMS: $[M + H]^+$ calculated for $C_{22}H_{16}Cl_2N^+$: 364.0564, found: 364.0563.



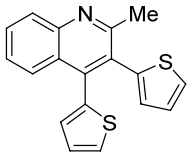
3,4-Bis(3-bromophenyl)-2-methylquinoline (**3ae**)

3ae was obtained according to the general procedure in 58% yield (52.4 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, J = 8.4 Hz, 1H), 7.73-7.69 (m, 1H), 7.47-7.43 (m, 2H), 7.40 (d, J = 8.0 Hz, 1H), 7.35 (d, J = 8.0 Hz, 1H), 7.30-7.24 (m, 2H), 7.19-7.10 (m, 2H), 7.06-6.97 (m, 2H), 2.55 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3, 147.2, 145.1, 140.4, 140.3, 138.5, 138.45, 132.9, 132.8, 132.6, 130.7, 130.4, 129.8, 129.7, 129.61, 129.6, 129.4, 128.8, 128.7, 128.6, 126.4, 126.3, 125.7, 122.3, 122.1, 122.0, 25.4. HRMS: $[M + H]^+$ calculated for $C_{22}H_{16}Br_2N^+$: 451.9644, found: 451.9641.



3,4-Bis(4-fluorophenyl)-2-methylquinoline (**3af**)

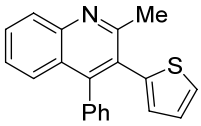
3af was obtained according to the general procedure in 87% yield (57.9 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, J = 8.4 Hz, 1H), 7.74-7.66 (m, 1H), 7.48-7.39 (m, 2H), 7.06-6.92 (m, 8H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7 (d, $J_{C\text{-}F}$ = 248.1 Hz), 161.7 (d, $J_{C\text{-}F}$ = 247.6 Hz), 157.8, 147.2, 145.9, 134.4 (d, $J_{C\text{-}F}$ = 3.6 Hz), 133.3, 132.6 (d, $J_{C\text{-}F}$ = 3.6 Hz), 131.7 (d, $J_{C\text{-}F}$ = 8.1 Hz), 131.6 (d, $J_{C\text{-}F}$ = 8.1 Hz), 129.4, 128.8, 126.3, 126.2, 126.1, 115.2 (d, $J_{C\text{-}F}$ = 21.5 Hz), 115.0 (d, $J_{C\text{-}F}$ = 21.6 Hz), 25.4. HRMS: $[M + H]^+$ calculated for $C_{22}H_{16}F_2N^+$: 332.1245, found: 332.1248.



2-Methyl-3,4-di(thiophen-2-yl)quinoline (**3ag**)

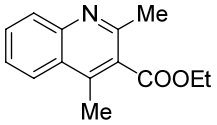
3ag was obtained according to the general procedure in 96% yield (58.9 mg). yelow solid; ^{1}H

NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 8.4 Hz, 1H), 7.77 (d, J = 8.4 Hz, 1H), 7.70 (t, J = 7.6 Hz, 1H), 7.44 (t, J = 7.6 Hz, 1H), 7.35 (d, J = 5.0 Hz, 1H), 7.29 (d, J = 5.1 Hz, 1H), 7.03-6.99 (m, 1H), 6.98-6.96 (m, 2H), 6.86 (dd, J = 3.3, 0.7 Hz, 1H), 2.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.6, 147.4, 141.9, 138.9, 136.6, 129.8, 129.6, 128.8, 128.7, 128.3, 127.2, 126.7, 126.7, 126.6, 126.58, 126.5, 126.4, 25.4. HRMS: $[M + H]^+$ calculated for $C_{18}H_{14}NS_2^+$: 308.0562, found: 308.0563.



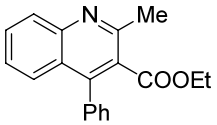
2-Methyl-4-phenyl-3-(thiophen-2-yl)quinoline (**3ah**), major : minor = 5 : 1

3ah was obtained according to the general procedure in 88% yield (52.9 mg). yelow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, J = 8.3 Hz, 1H), 7.76 (t, J = 7.4 Hz, 1H), 7.56 (d, J = 8.4 Hz, 1H), 7.45 (t, J = 7.5 Hz, 1H), 7.36 (t, J = 7.5 Hz, 3H), 7.31-7.18 (m, 3H), 6.97-6.94 (m, 1H), 6.84 (s, 1H), 2.74 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 148.8, 147.3, 139.1, 136.7, 129.8, 129.8, 129.6, 128.7, 128.5, 128.0, 127.7, 127.5, 126.8, 126.6, 126.2, 126.0, 25.3. HRMS: $[M + H]^+$ calculated for $C_{20}H_{16}NS^+$: 302.0998, found: 302.0999.



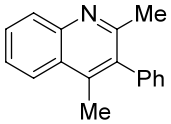
Ethyl 2,4-dimethylquinoline-3-carboxylate (**3ai**), major : minor = 14 : 1

3ai was obtained according to the general procedure in 87% yield (42.2 mg). yelow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 8.02-7.97 (m, 2H), 7.70 (t, J = 7.6 Hz, 1H), 7.53 (t, J = 7.6 Hz, 1H), 4.49 (q, J = 7.1 Hz, 2H), 2.71 (s, 3H), 2.65 (s, 3H), 1.44 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.2, 154.3, 147.1, 141.4, 130.0, 129.3, 128.0, 126.3, 125.8, 124.0, 61.7, 23.8, 15.7, 14.2. HRMS: $[M + H]^+$ calculated for $C_{14}H_{16}NO_2^+$: 230.1176, found: 230.1177.



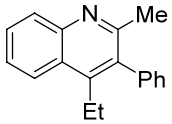
Ethyl 2-methyl-4-phenylquinoline-3-carboxylate (**3aj**), major : minor = 4 : 1

3aj was obtained according to the general procedure in 77% yield (44.8 mg). yelow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 8.1 Hz, 1H), 7.82 (d, J = 8.2 Hz, 1H), 7.73 (t, J = 7.3 Hz, 1H), 7.56 (d, J = 7.2 Hz, 1H), 7.51-7.39 (m, 4H), 7.33-7.31 (m, 2H), 4.12 (q, J = 6.9 Hz, 2H), 2.55 (s, 3H), 0.95 (t, J = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 157.8, 147.0, 138.8, 137.4, 132.0, 129.8, 129.3, 129.0, 128.4, 128.0, 127.0, 124.8, 122.6, 61.6, 24.8, 13.7. HRMS: $[M + H]^+$ calculated for $C_{19}H_{18}NO_2^+$: 292.1332, found: 292.1334.



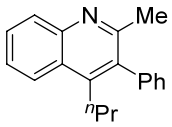
2,4-Dimethyl-3-phenylquinoline (**3ak**), major : minor = 10 : 1

3ak was obtained according to the general procedure in 95% yield (44.5 mg). yelow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.4 Hz, 1H), 7.98 (d, J = 8.3 Hz, 1H), 7.68 (t, J = 7.3 Hz, 1H), 7.54-7.39 (m, 4H), 7.21-7.19 (m, 2H), 2.43 (s, 3H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.6, 146.7, 141.2, 139.6, 134.9, 129.3, 129.2, 128.9, 128.7, 127.4, 126.8, 125.8, 124.1, 25.5, 15.9. HRMS: $[M + H]^+$ calculated for $C_{17}H_{16}N^+$: 234.1277, found: 234.1279.



4-Ethyl-2-methyl-3-phenylquinoline (**3al)**, major : minor = 8 : 1

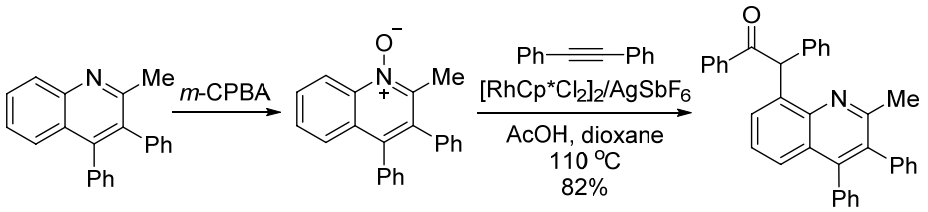
3al was obtained according to the general procedure in 92% yield (45.4 mg). yelow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, J = 8.4 Hz, 1H), 8.01 (d, J = 8.3 Hz, 1H), 7.67 (t, J = 7.6 Hz, 1H), 7.54-7.40 (m, 4H), 7.24-7.20 (m, 2H), 2.81 (q, J = 7.6 Hz, 2H), 2.39 (s, 3H), 1.14 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 147.3, 147.0, 139.4, 134.3, 129.5, 129.2, 128.8, 128.7, 127.4, 125.8, 125.6, 124.1, 25.5, 22.6, 15.1. HRMS: $[M + H]^+$ calculated for $C_{18}H_{18}N^+$: 248.1434, found: 248.1436.



2-Methyl-3-phenyl-4-propylquinoline (**3am**), major : minor = 11 : 1

3am was obtained according to the general procedure in 88% yield (46.0 mg). brown solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.3 Hz, 1H), 7.99 (d, J = 8.3 Hz, 1H), 7.70-7.64 (m, 1H), 7.54-7.40 (m, 4H), 7.24-7.19 (m, 2H), 2.78-2.71 (m, 2H), 2.39 (s, 3H), 1.64-1.50 (m, 2H), 0.86 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 147.1, 145.7, 139.4, 134.5, 129.3, 129.3, 128.8, 128.6, 127.4, 125.9, 125.7, 124.2, 31.6, 25.5, 24.2, 14.6. HRMS: $[M + H]^+$ calculated for $C_{19}H_{20}N^+$: 262.1590, found: 262.1593.

III. Derivatization Reactions



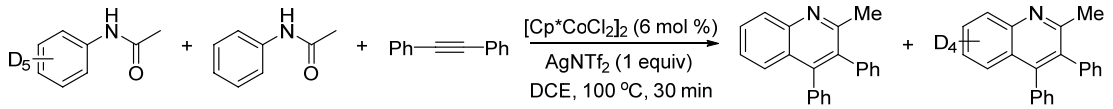
3aa **4**

Quinoline **3aa** was oxidized by *m*-CPBA.[4] The *N*-oxide product was allowed to react with diphenylacetylene by following our previously reported procedures.[5a] 2-(2-Methyl-3,4-diphenylquinolin-8-yl)-1,2-diphenylethan-1-one (**4**), 82% yield, white solid. ^{1}H

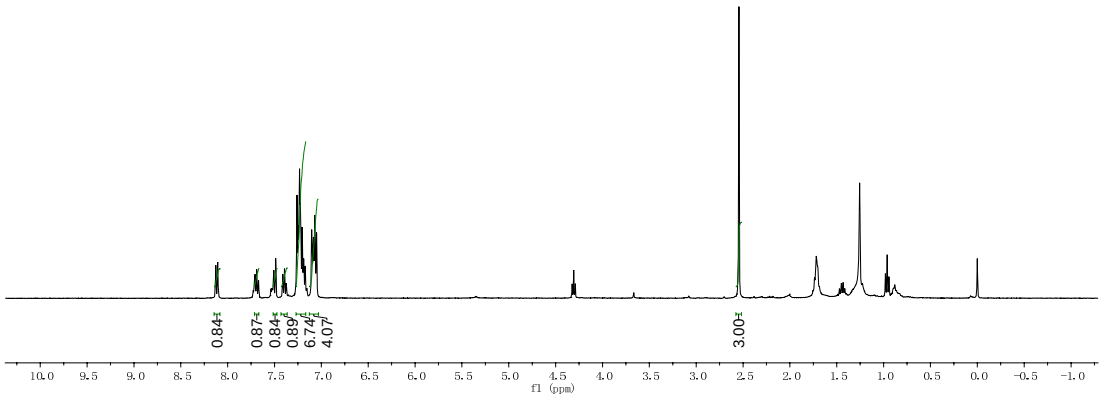
NMR (400 MHz, $CDCl_3$) δ 8.25-8.17 (m, 2H), 7.55-7.52 (m, 2H), 7.51-7.46 (m, 1H), 7.45-7.36 (m, 6H), 7.34-7.11 (m, 9H), 7.10-7.06 (m, 1H), 7.05-6.98 (m, 3H), 2.27 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.9, 156.7, 146.7, 144.2, 139.2, 138.8, 138.0, 137.8, 137.1, 134.0, 132.4, 130.2, 130.1, 130.07, 130.02, 129.1, 128.9, 128.4, 127.9, 127.86, 127.7, 127.6, 127.3, 127.2, 126.8, 126.2, 125.8, 125.5, 54.7, 25.3. HRMS: $[M + H]^+$ calculated for $C_{36}H_{28}NO^+$: 490.2165, found: 490.2168.

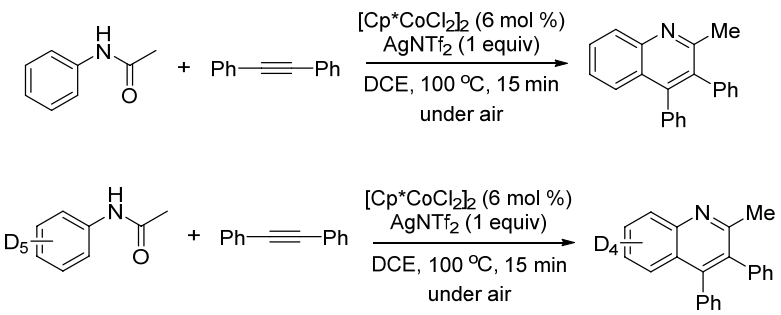
IV. Mechanistic Studies

1. KIE measurements of reaction for quinolines

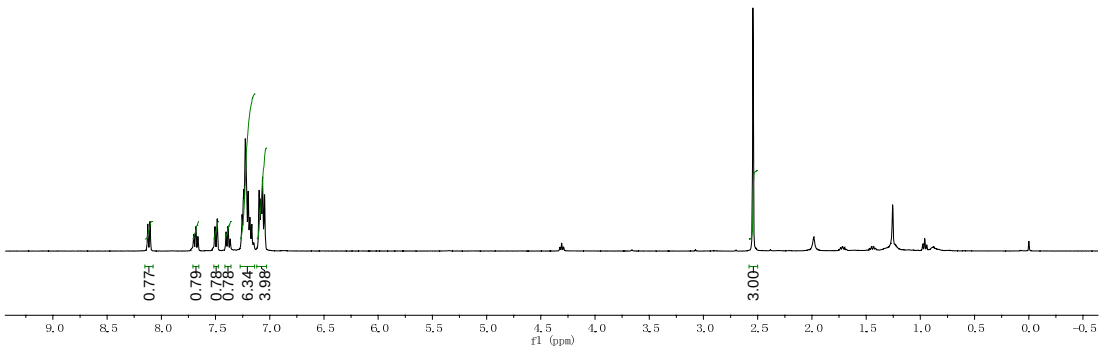


An equimolar mixture of acetanilide (0.2 mmol, 27.0 mg) and acetanilide-d_5 (0.2 mmol, 28.0 mg), diphenylacetylene (0.2 mmol, 35.6 mg), $[Cp^*CoCl_2]_2$ (6.0 mol %, 6.4 mg), $AgNTf_2$ (0.2 mmol, 77.6 mg), and DCE (4.0 mL) were charged into a pressure tube under air. The reaction mixture was stirred at 100 °C for 30 min. The solvent was rapidly removed under reduced pressure and the residue was purified by silica gel chromatography using PE/EA and EA/triethylamine to afford the mixed product. KIE value (k_H/k_D = 5.3) was determined on the basis of ^1H NMR analysis.

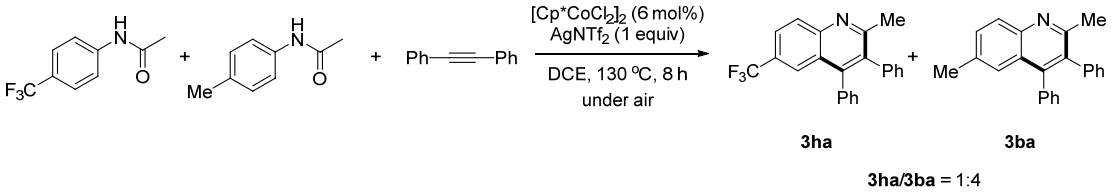




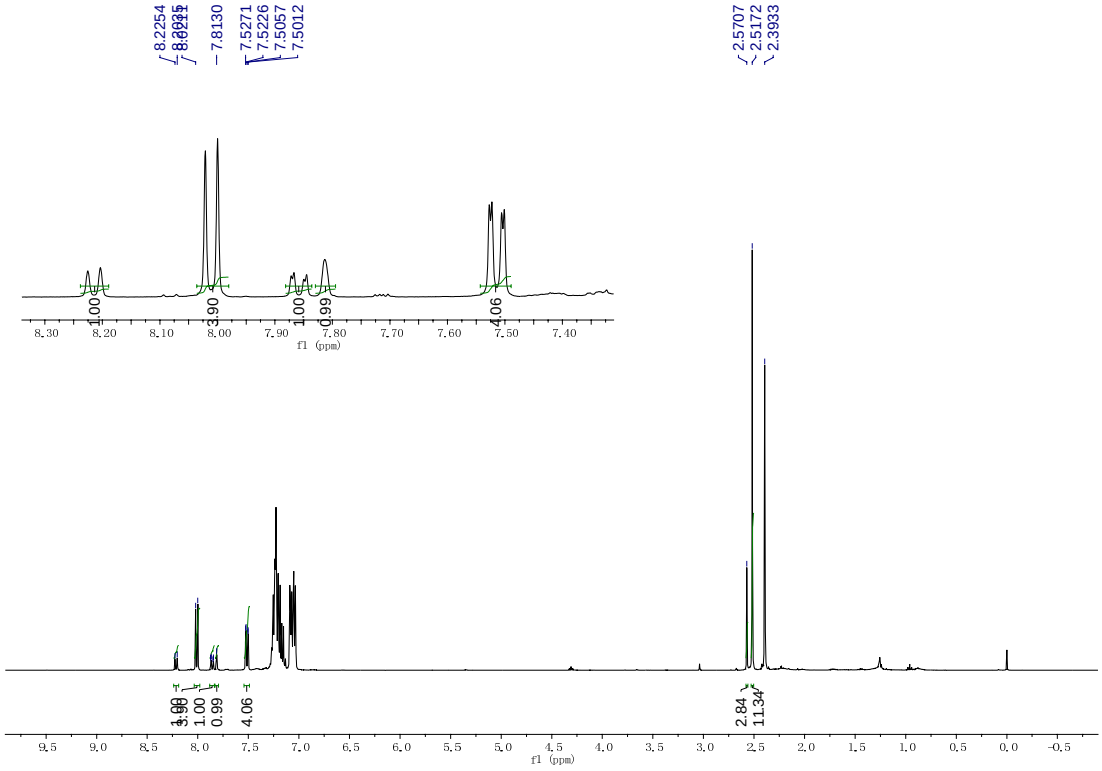
A mixture of acetanilide (0.2 mmol, 27.0 mg), diphenylacetylene (0.2 mmol, 35.6 mg), $[Cp^*CoCl_2]_2$ (6 mol %, 6.4 mg), $AgNTf_2$ (0.2 mmol, 77.6 mg), and DCE (4.0 mL) were charged into a pressure tube under air. In another tube were added a mixture of acetanilide-d_5 (0.2 mmol, 28.0 mg), diphenylacetylene (0.2 mmol, 35.6 mg), $[Cp^*CoCl_2]_2$ (6.0 mol %, 6.4 mg), $AgNTf_2$ (0.2 mmol, 77.6 mg), and DCE (4.0 mL). These two reaction mixtures were stirred side-by-side in the same oil bath at 100 °C for 15 min. These two mixtures were rapidly combined and all the volatiles were rapidly removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA and EA/triethylamine to afford the mixed product. KIE value (k_H/k_D = 3.4) was determined on the basis of ^{1}H NMR analysis.



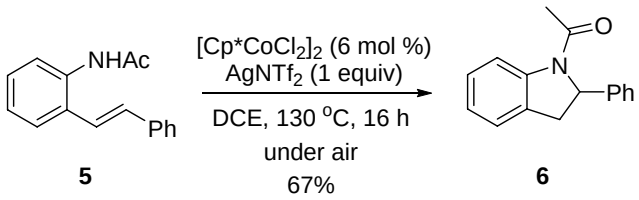
2. Competitive Experiment



An equimolar mixture of acetanilide **1h** (0.2 mmol, 30.0 mg) and **1b** (0.2 mmol, 40.6 mg), diphenylacetylene (0.24 mmol, 42.8 mg), $[Cp^*CoCl_2]_2$ (6.0 mol %, 6.4 mg), $AgNTf_2$ (0.2 mmol, 77.6 mg), and DCE (4.0 mL) were charged into a pressure tube under air. The reaction mixture was stirred at 130 °C for 8 h. The solvent was rapidly removed under reduced pressure and the residue was purified by silica gel chromatography using PE/EA and EA/triethylamine to afford the mixed product. The yield ratio (**3ha**/**3ba** = 1:4) was determined on the basis of ^{1}H NMR analysis.



3. Studies of a possible intermediate



Compound **5** was prepared by following a literature report.[6] **5** was subjected to the reaction under optimized conditions to afford **6**. 1-(2-Phenylindolin-1-yl)ethan-1-one (**6**), 67% yield, white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, J = 8.0 Hz, 1H), 7.32-7.24 (m, 4H), 7.17-7.11 (m, 3H), 7.07-7.03 (m, 1H), 5.37 (d, J = 9.9 Hz, 1H), 3.79 (dd, J = 15.7, 10.4 Hz, 1H), 2.97 (dd, J = 16.0, 1.8 Hz, 1H), 2.03 (s, 3H). HRMS: $[M + H]^+$ calculated for $C_{16}H_{16}NO^+$: 238.1226, found: 238.1227.

The NMR datas agree with those in a literature report.[3]

V. References

(1) Mio, M. J.; Kopel, L. C.; Braun, J. B.; Gadzikwa, T. L.; Hull, K. L.; Brisbois, R. G.; Markworth, C. J.; Grieco, P. A. *Org. Lett*. **2002**, *4*, 3199.

(2) Yoshino, T.; Ikemoto, H.; Matsunaga, S.; Kanai, M. *Angew. Chem., Int. Ed*. **2013**, *52*, 2207.

(3) Saito, K.; Shibata, Y.; Yamanaka, M.; Akiyama, T. *J. Am. Chem. Soc.* **2013**, *135*, 11740.

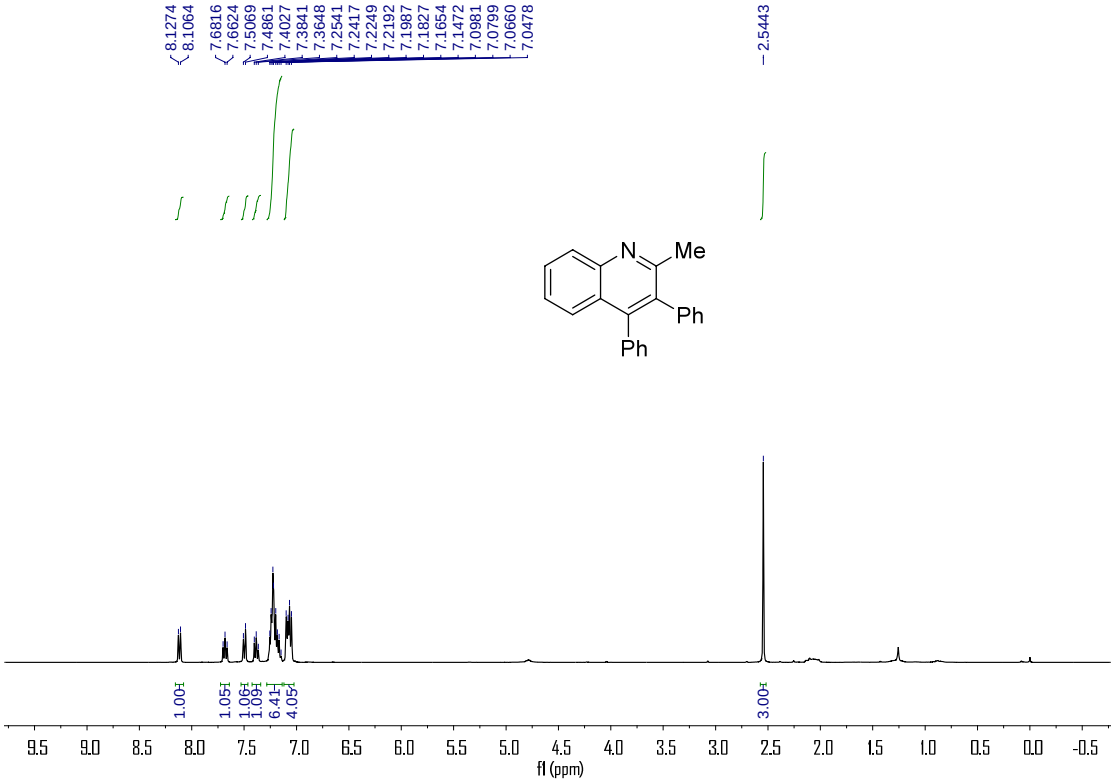
(4) Li, G.; Jia, C.; Sun, K.; Lv, Y.; Zhao, F.; Zhou, K.; Wu, H. *Org. Biomol. Chem*. **2015**, *13*, 3207.

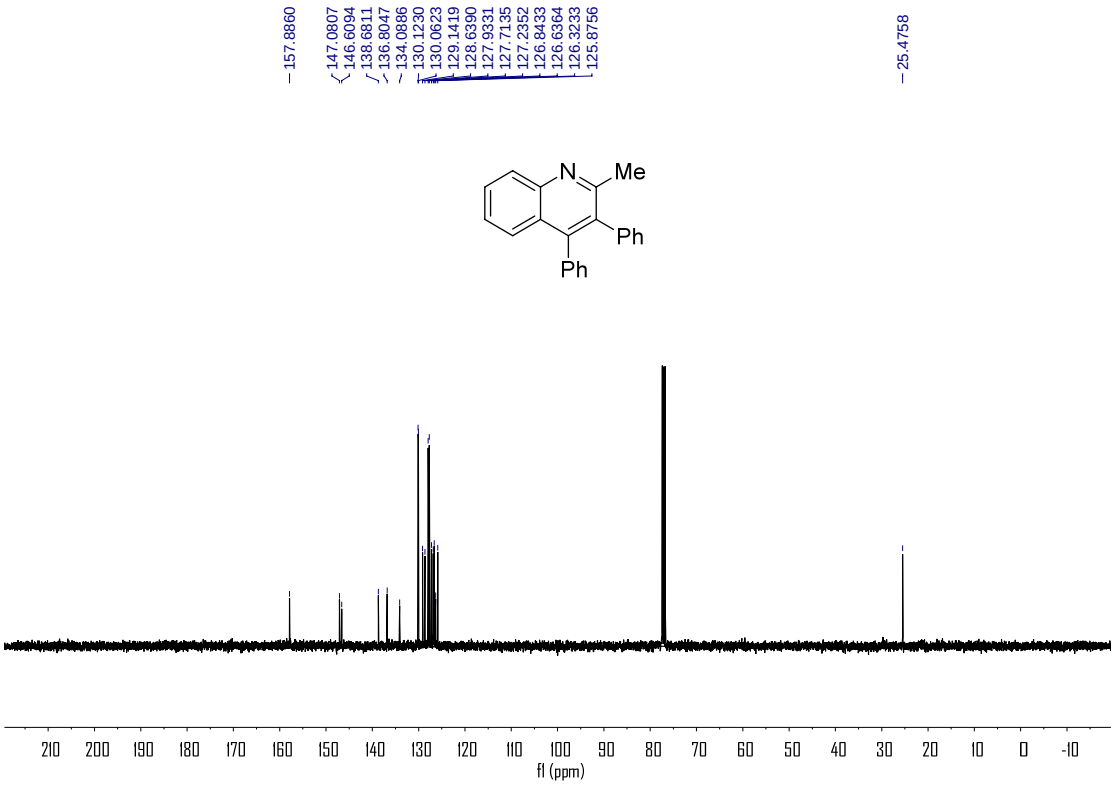
(5) Zhang, X.; Qi, Z.; Li, X. *Angew. Chem., Int. Ed.* **2014**, *53*, 10794.

(6) Patureau, F. W.; Glorius, F. *J. Am. Chem. Soc.* **2010**, *132*, 9982.

VI. NMR Spectra

3aa





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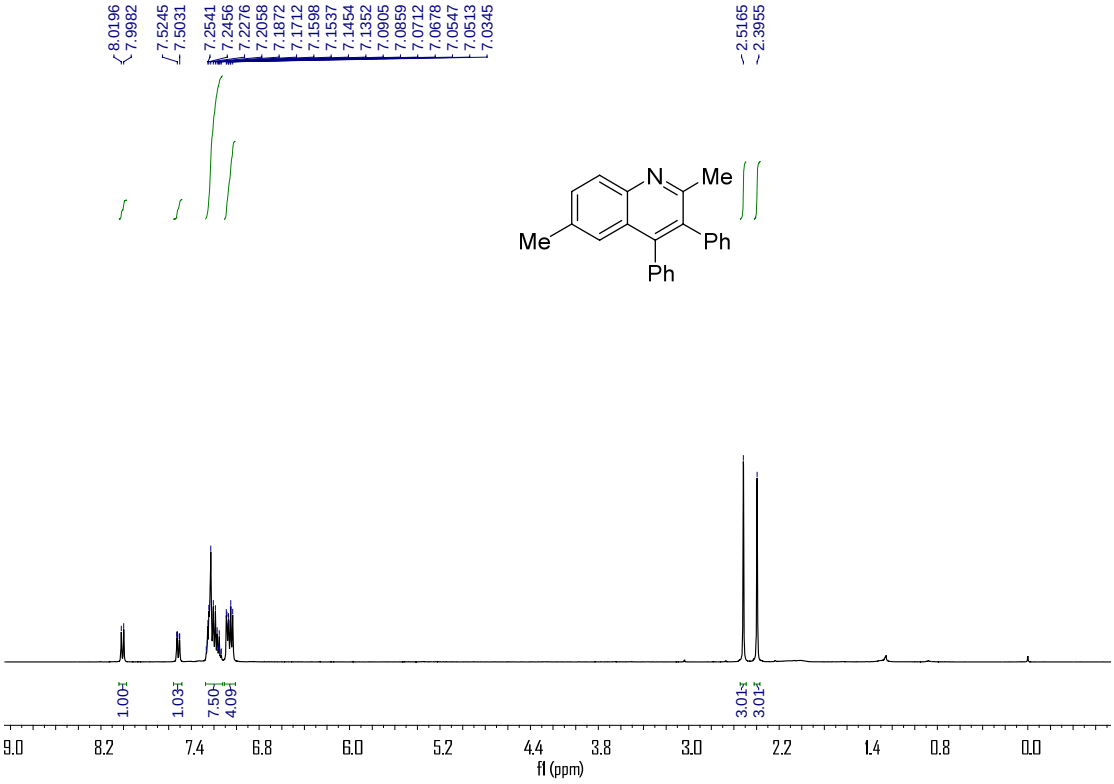
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7.1537
7.1454
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7.0905
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7.0712
7.0678
7.0547
7.0513
7.0345
2.5165
2.3955
N
Me
Me
Ph
Ph
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1.03
7.50
4.09
3.01
3.01
9.0
8.2
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5.2
4.4
3.8
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1.4
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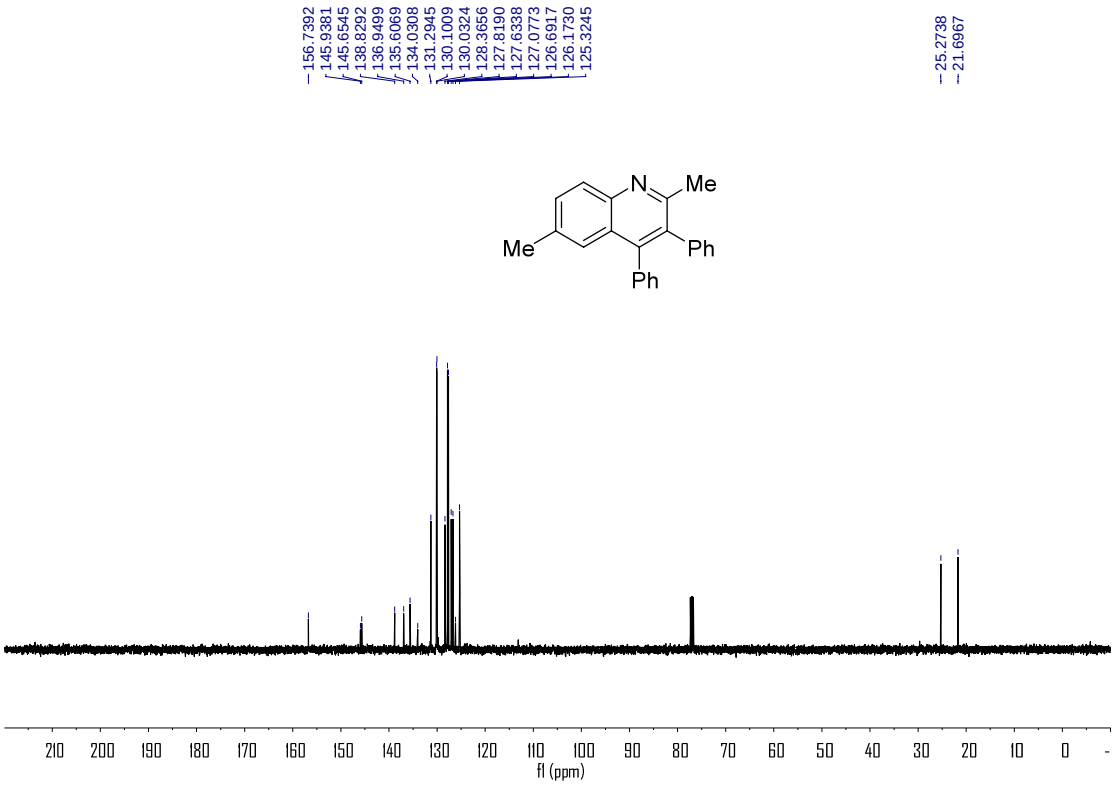
0.0
f1 (ppm)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Me
Ph
Ph
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180
170
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110
100
90
80
70
60
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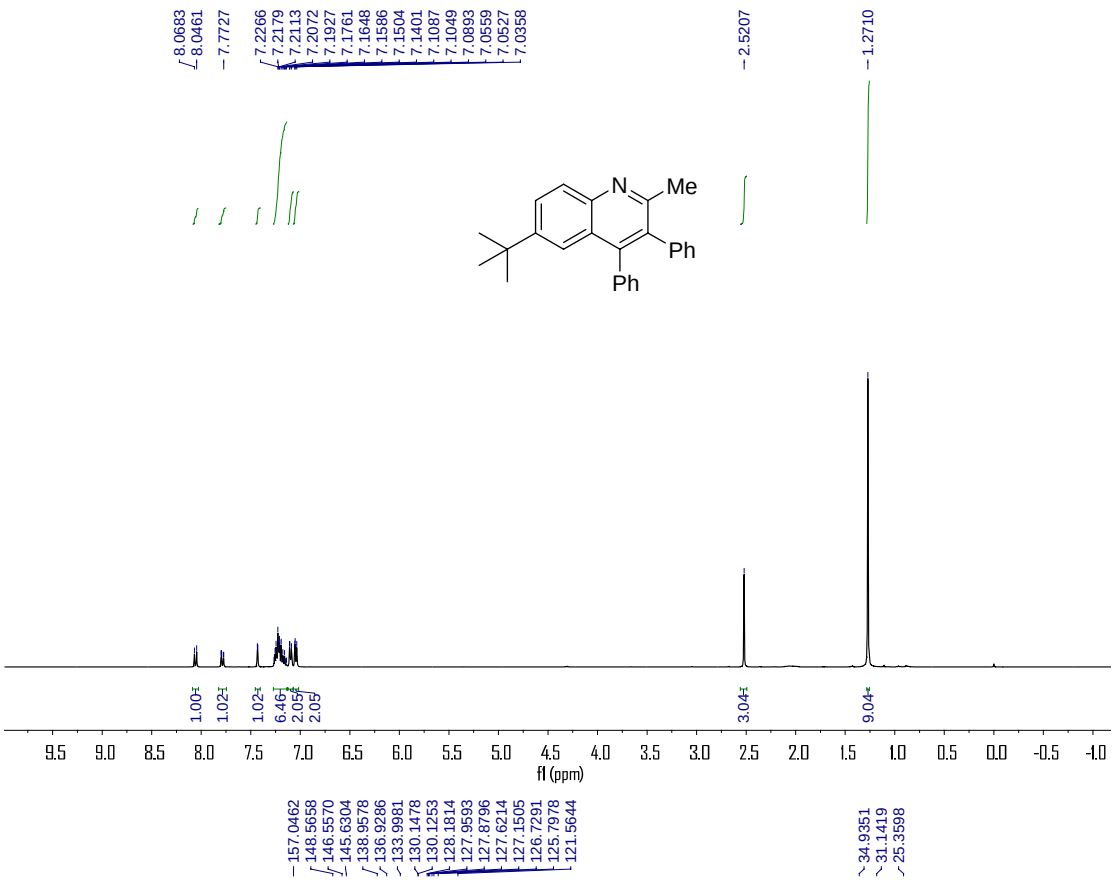
40
30
20
10
0
f1 (ppm)

3ca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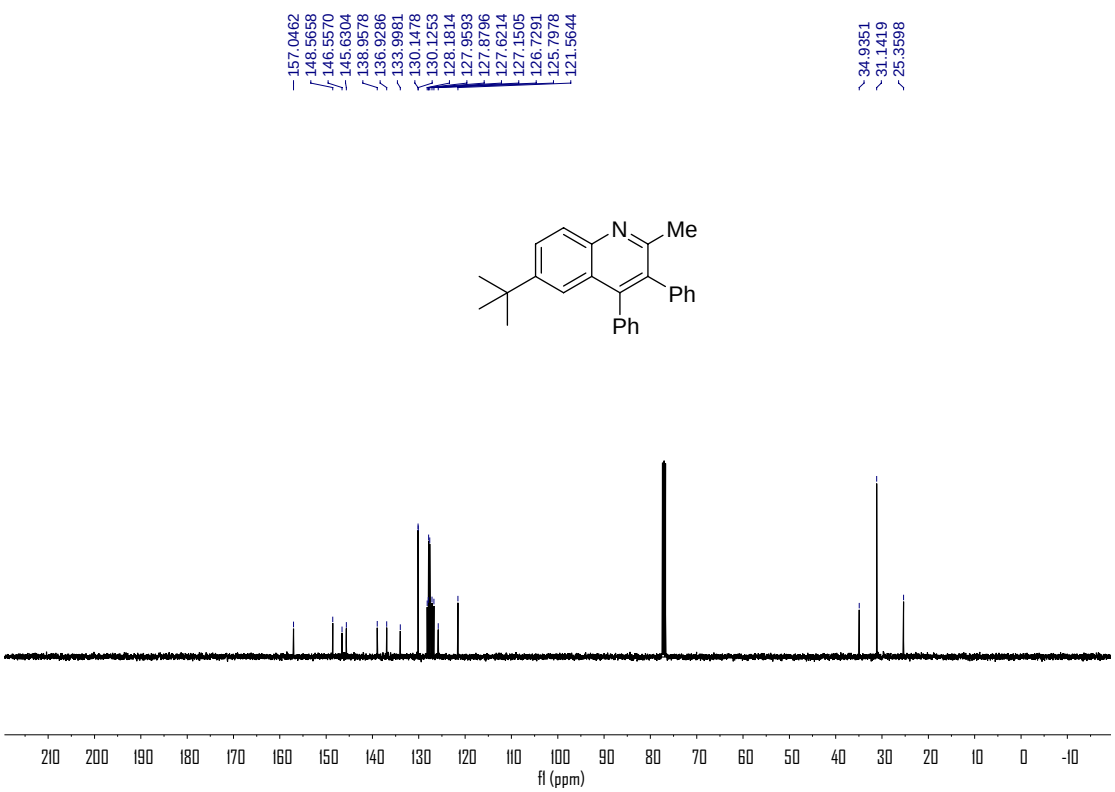
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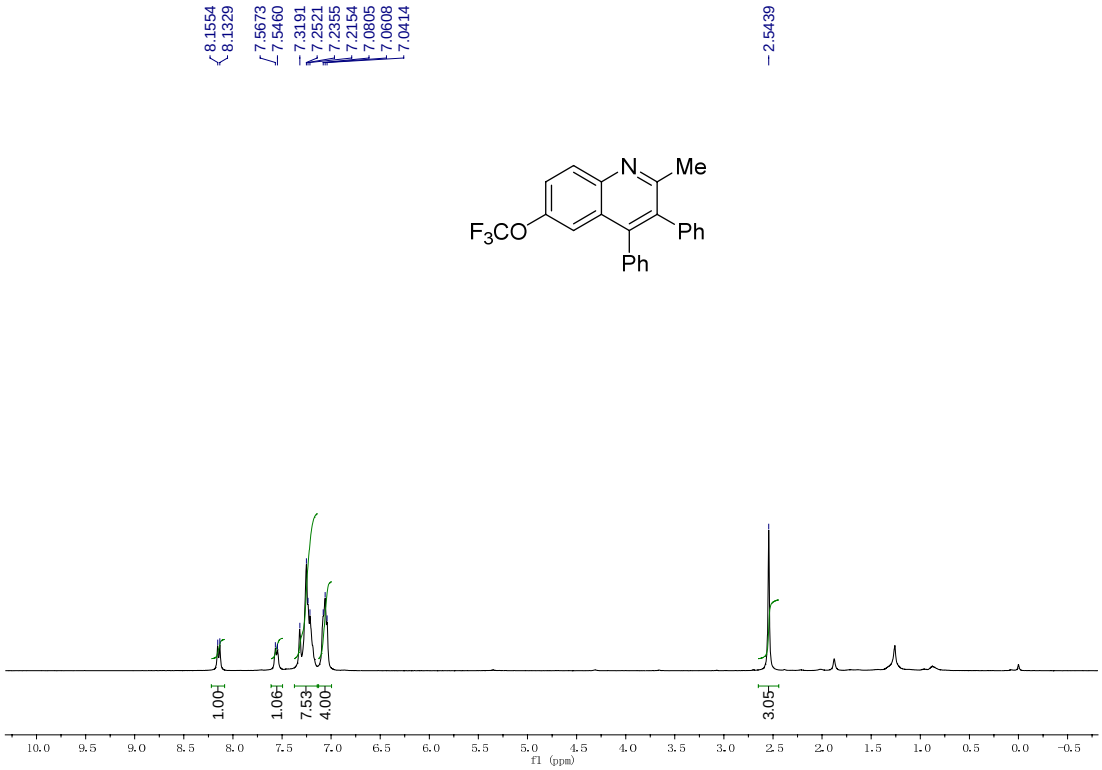
Ph
Ph
f1 (ppm)

3da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F3CO
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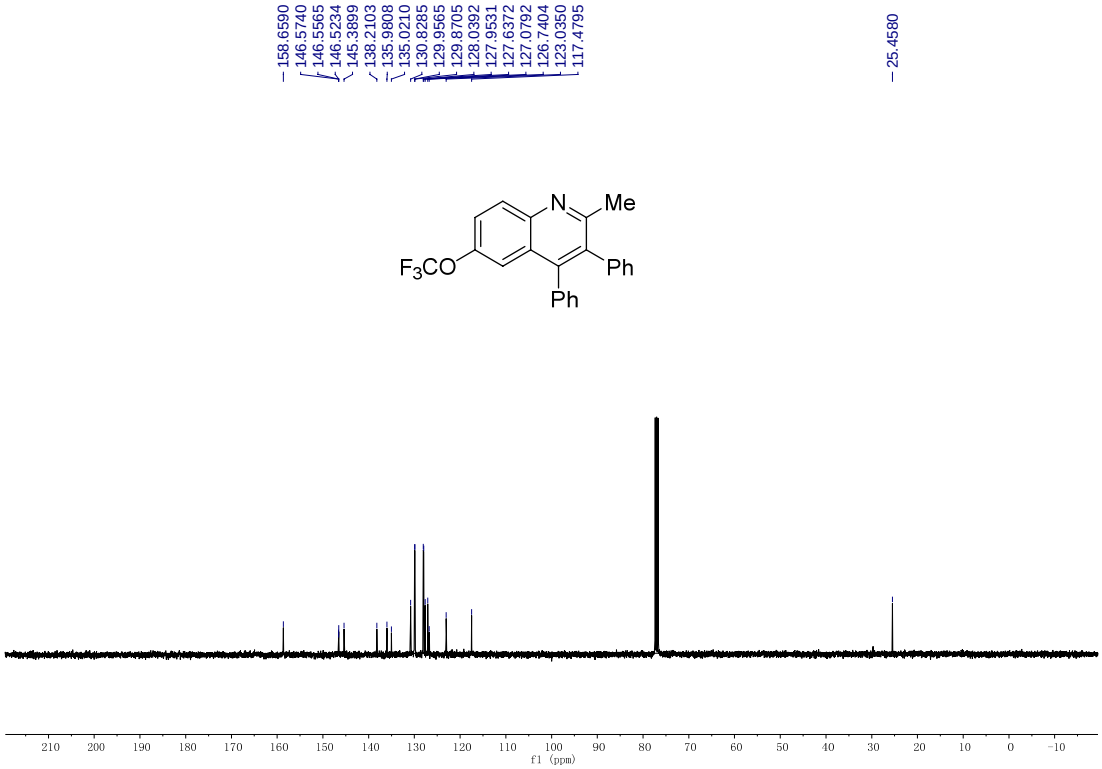
f1 (ppm)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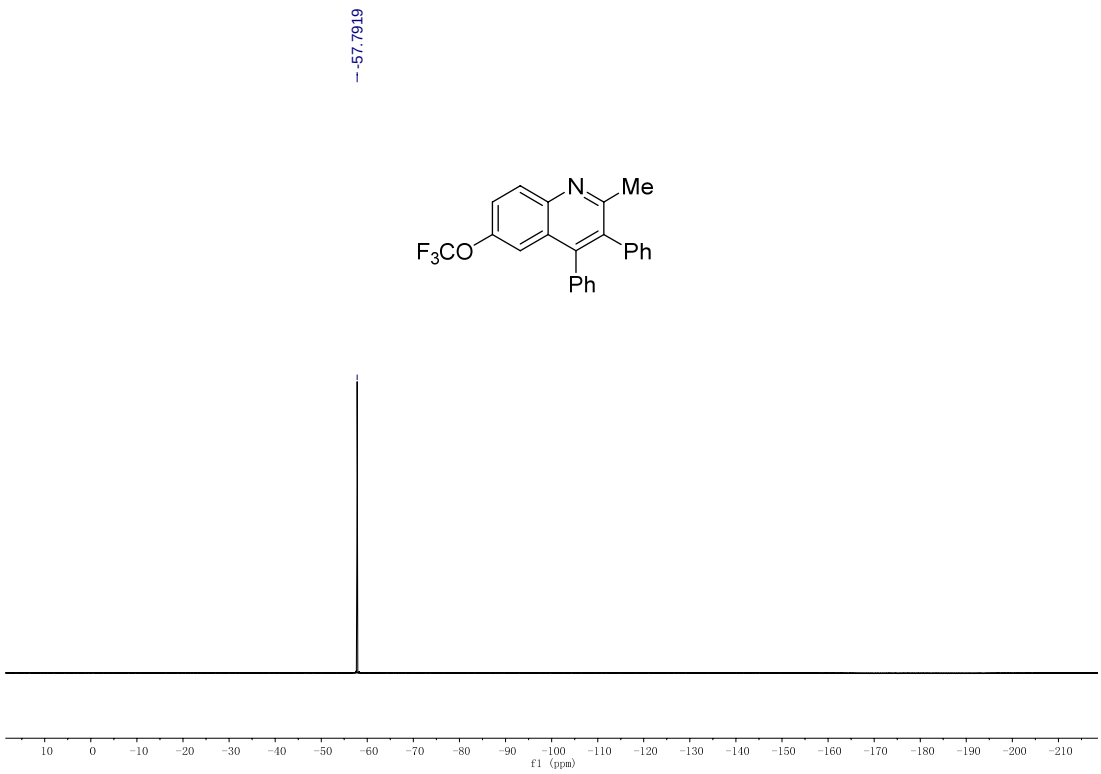
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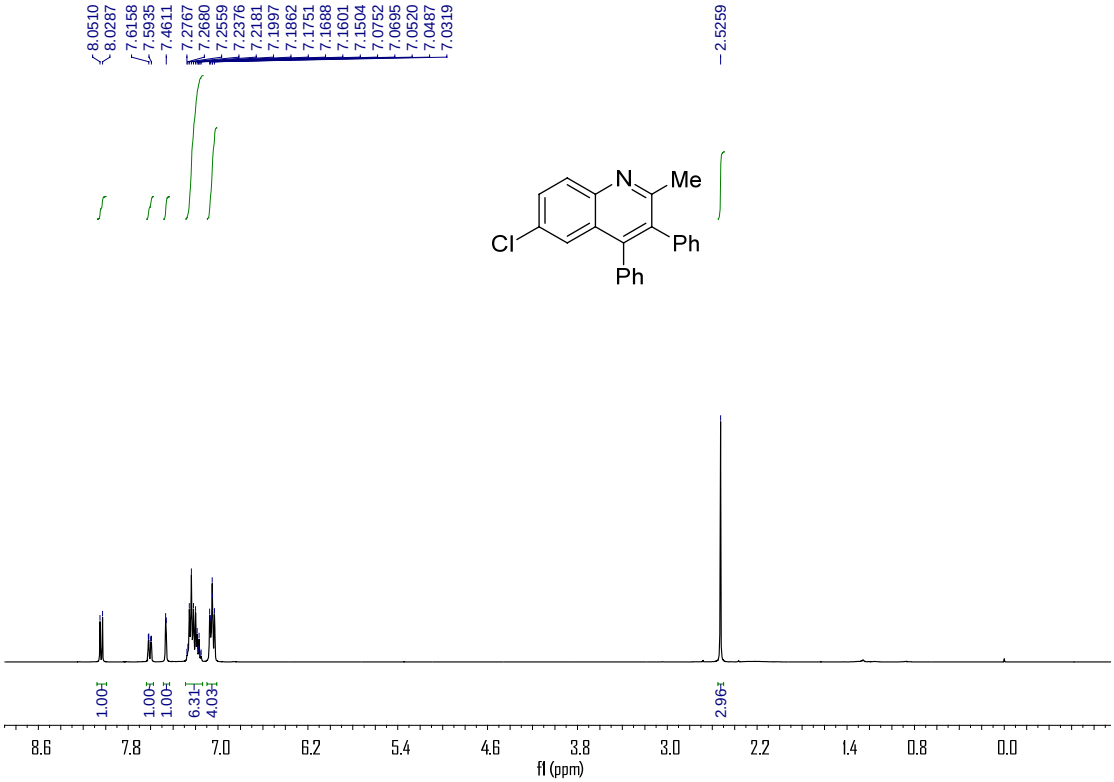
3ea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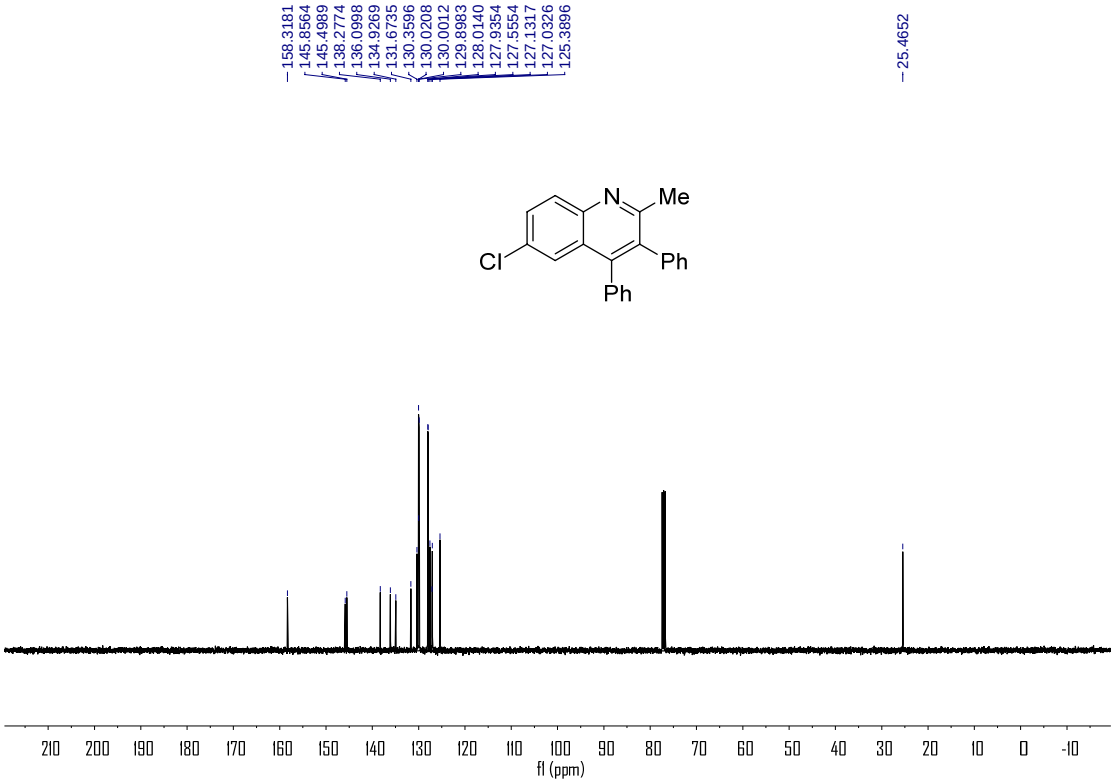
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20
10
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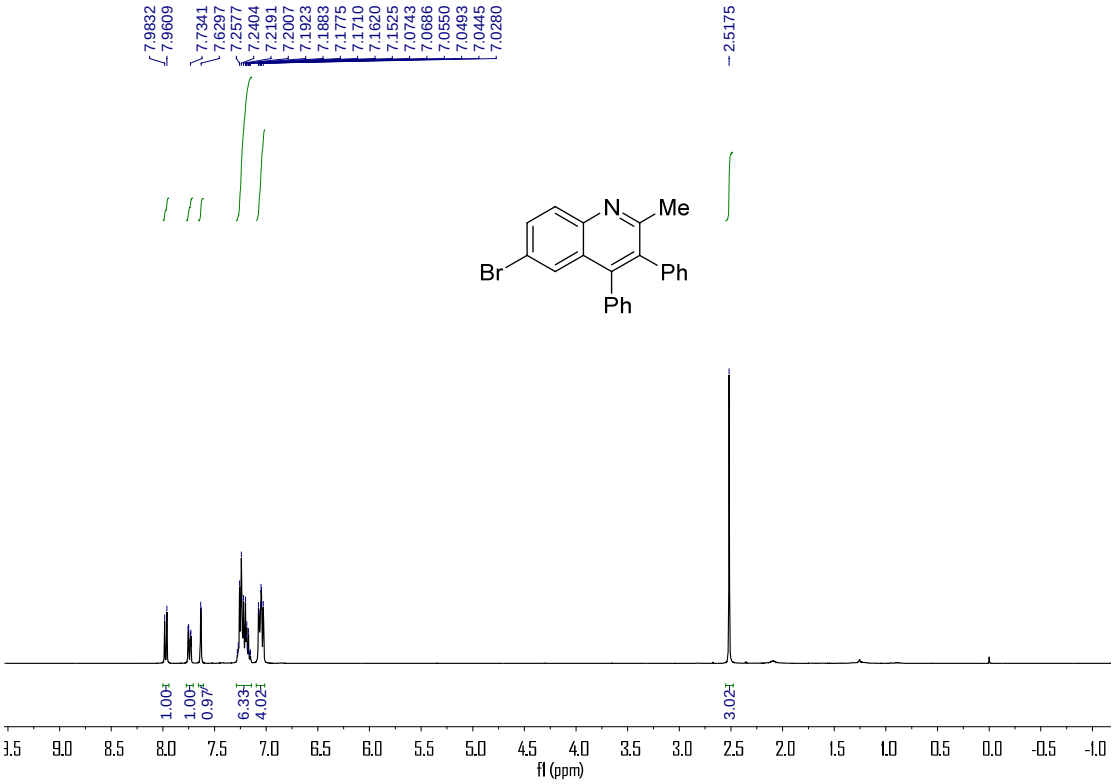
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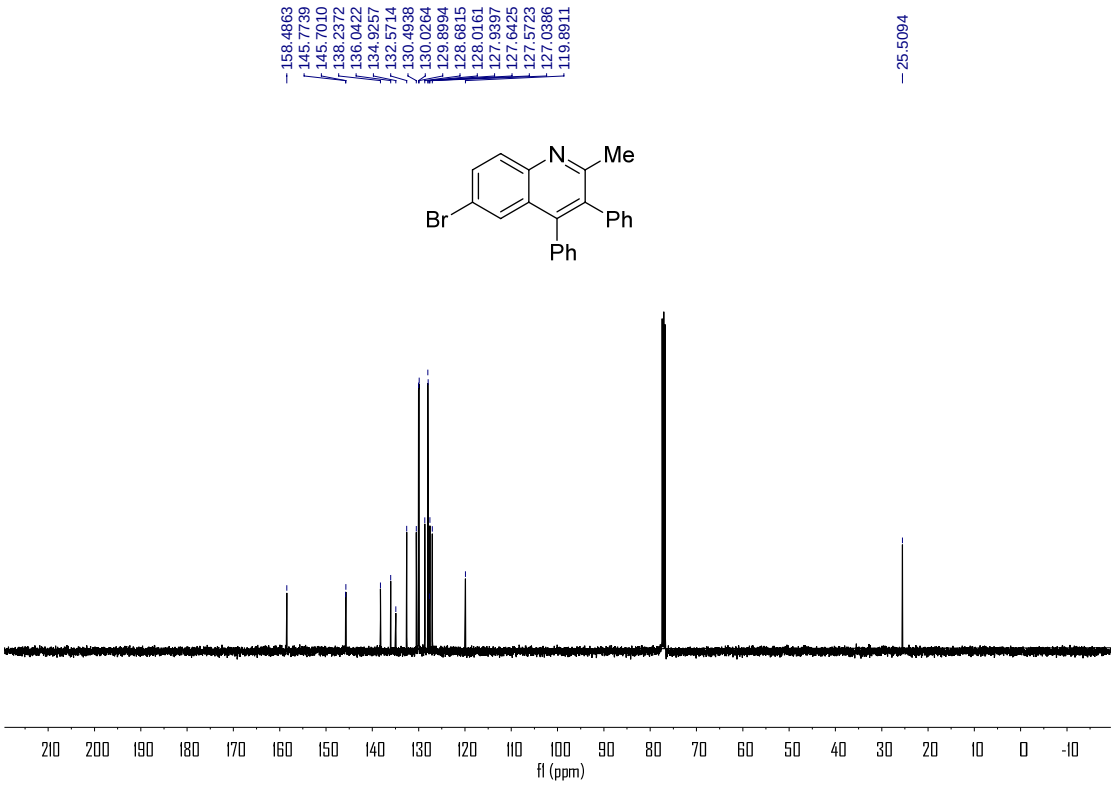
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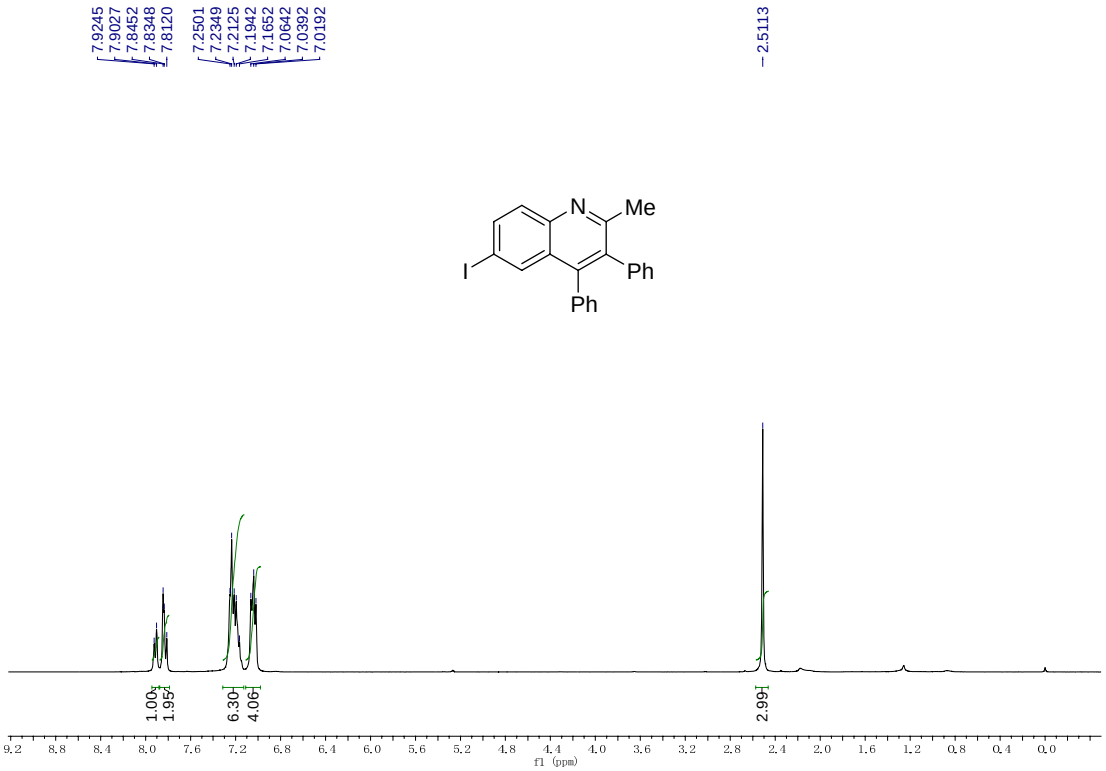
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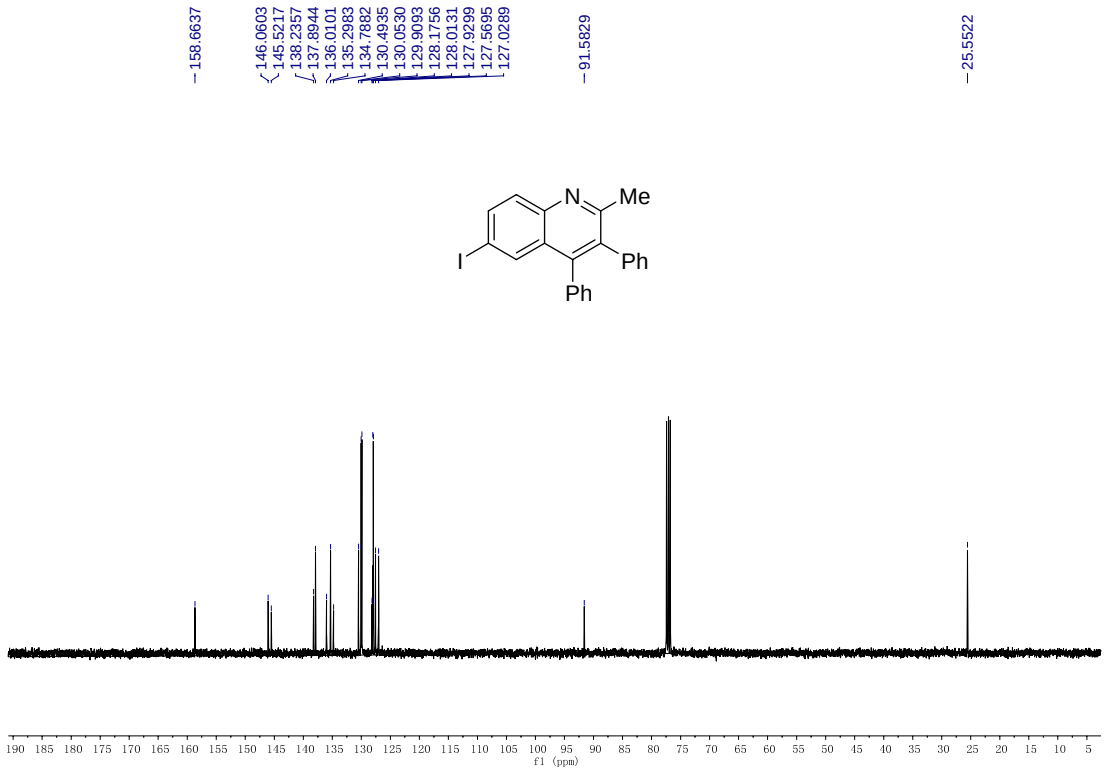
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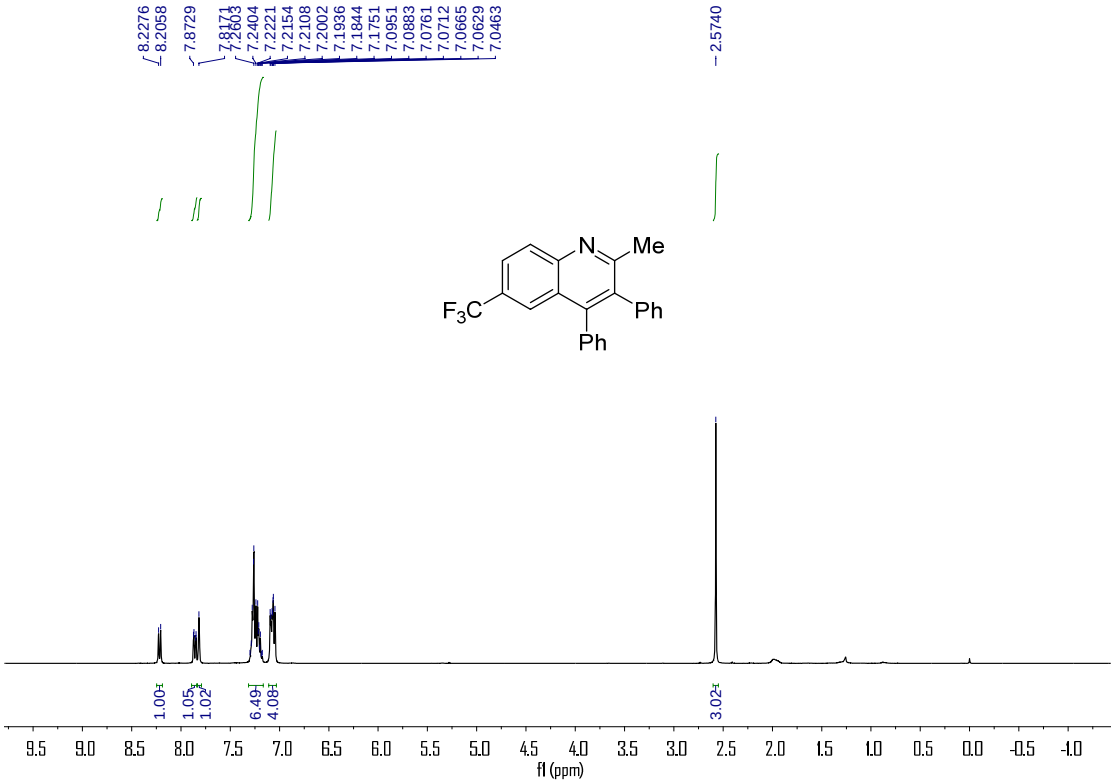
3ha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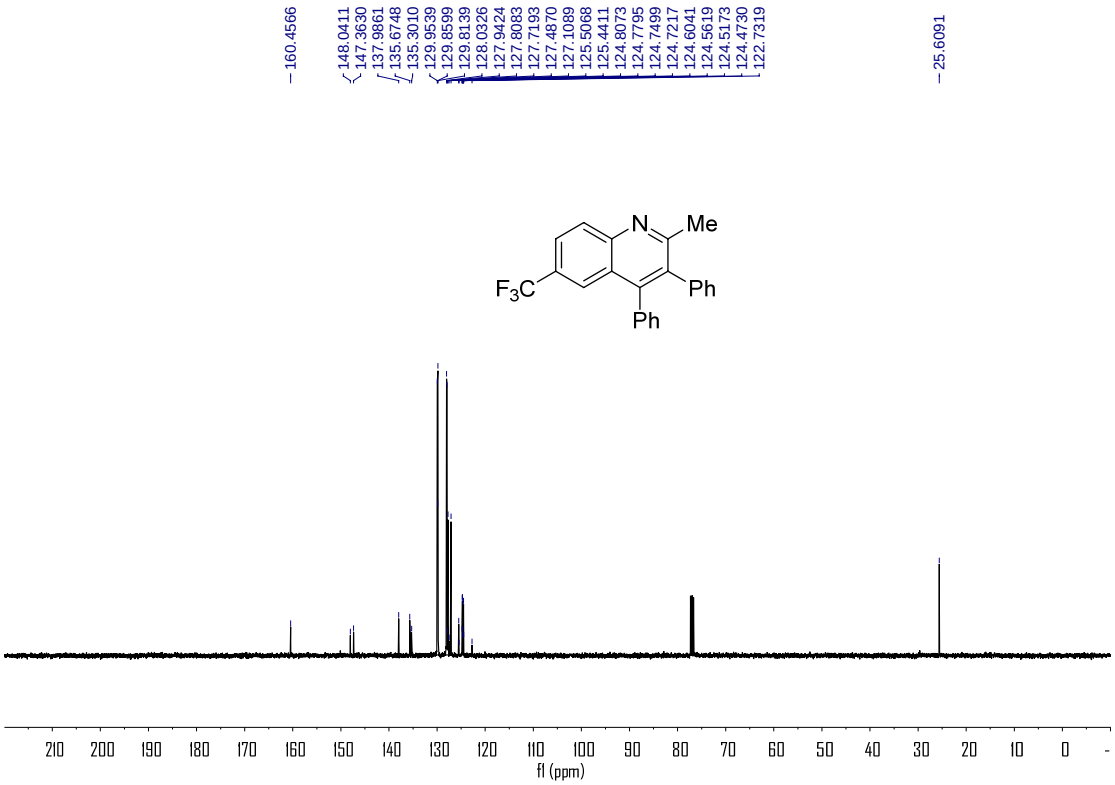
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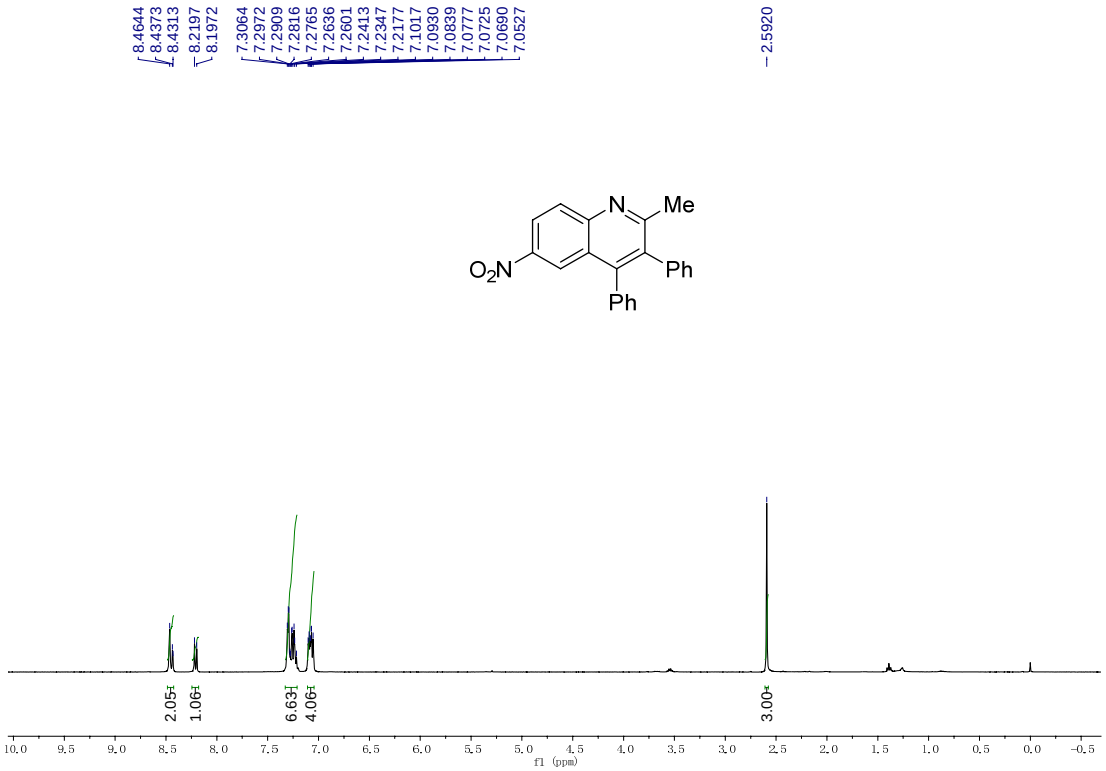
3ia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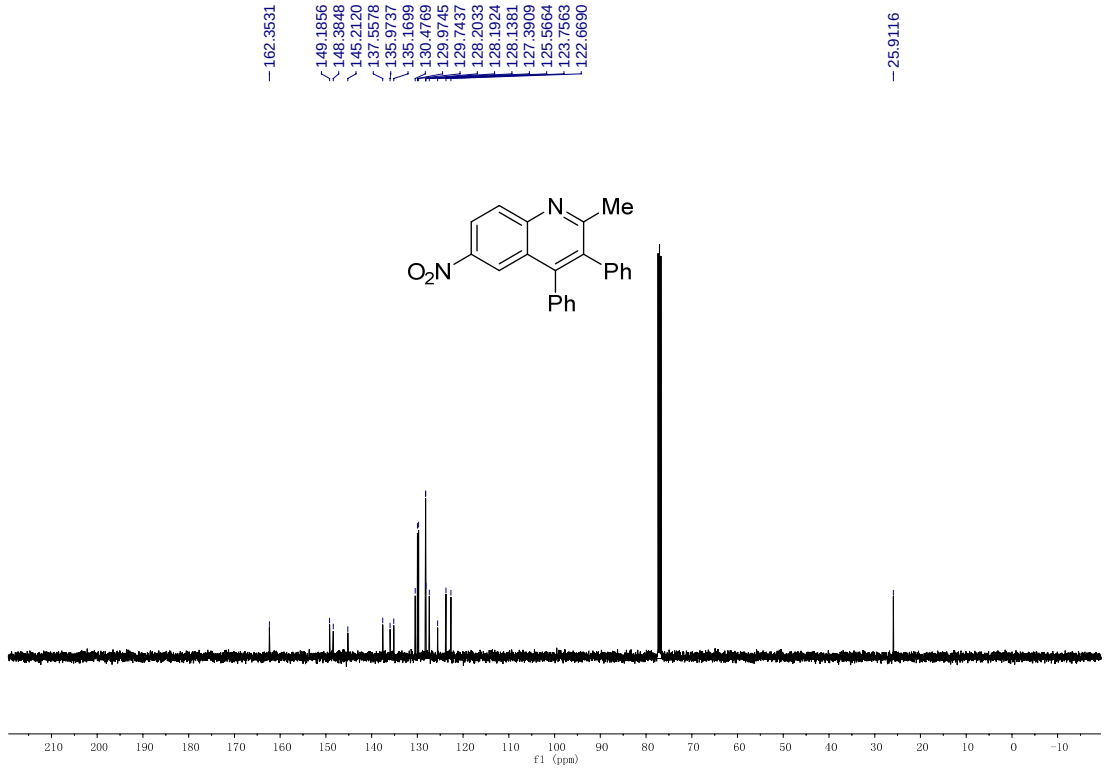
f1 (ppm)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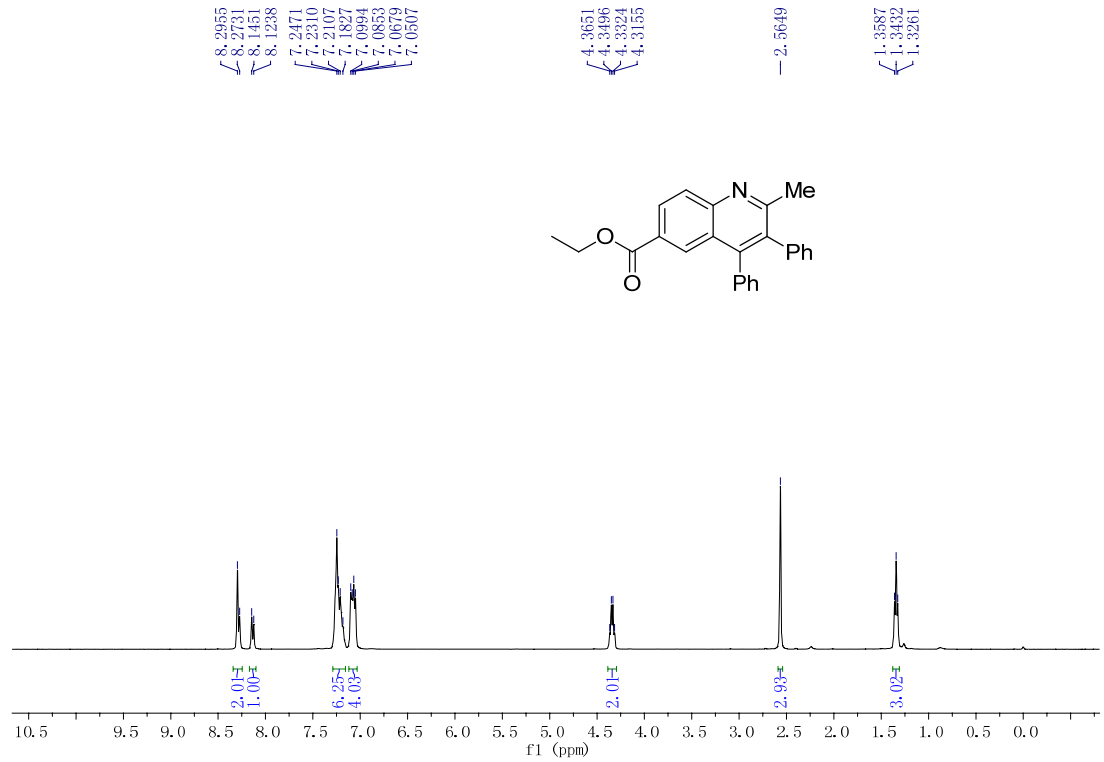
148.3848
145.2120
137.5578
135.9737
135.1699
130.4769
129.9745
129.7437
128.2033
128.1924
128.1381
127.3909
125.5664
123.7563
122.6690
25.9116
N
Me
O2N
Ph
Ph
f1 (ppm)

3ja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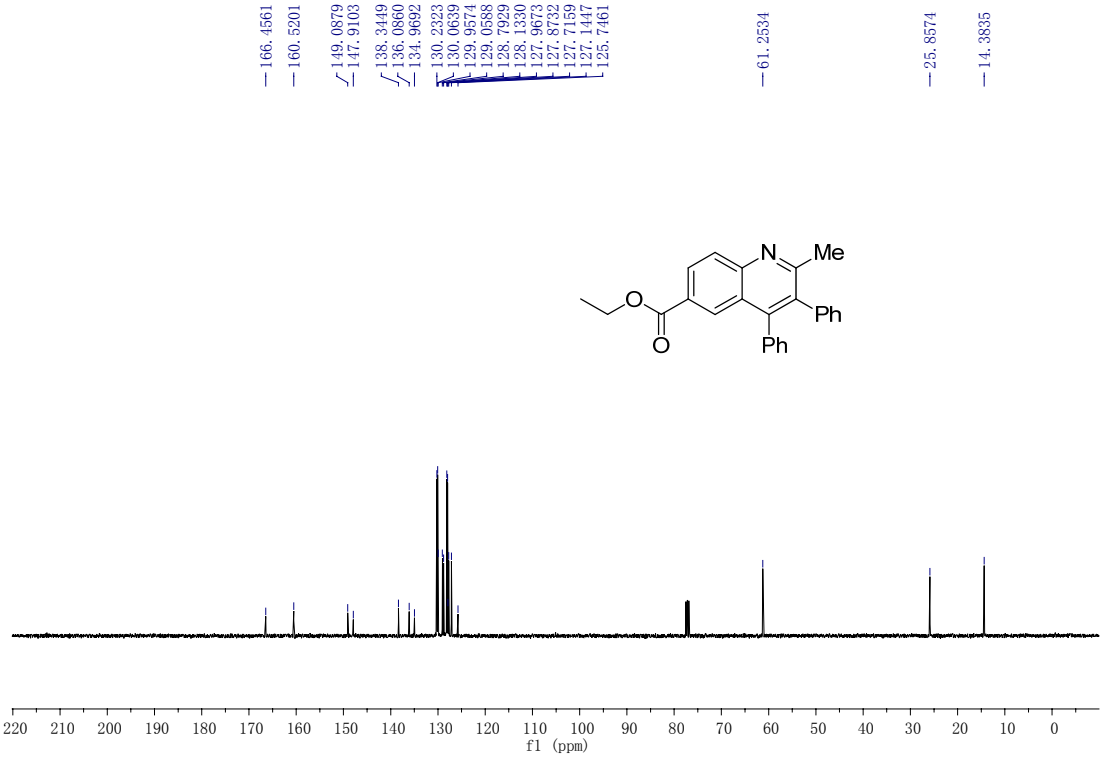
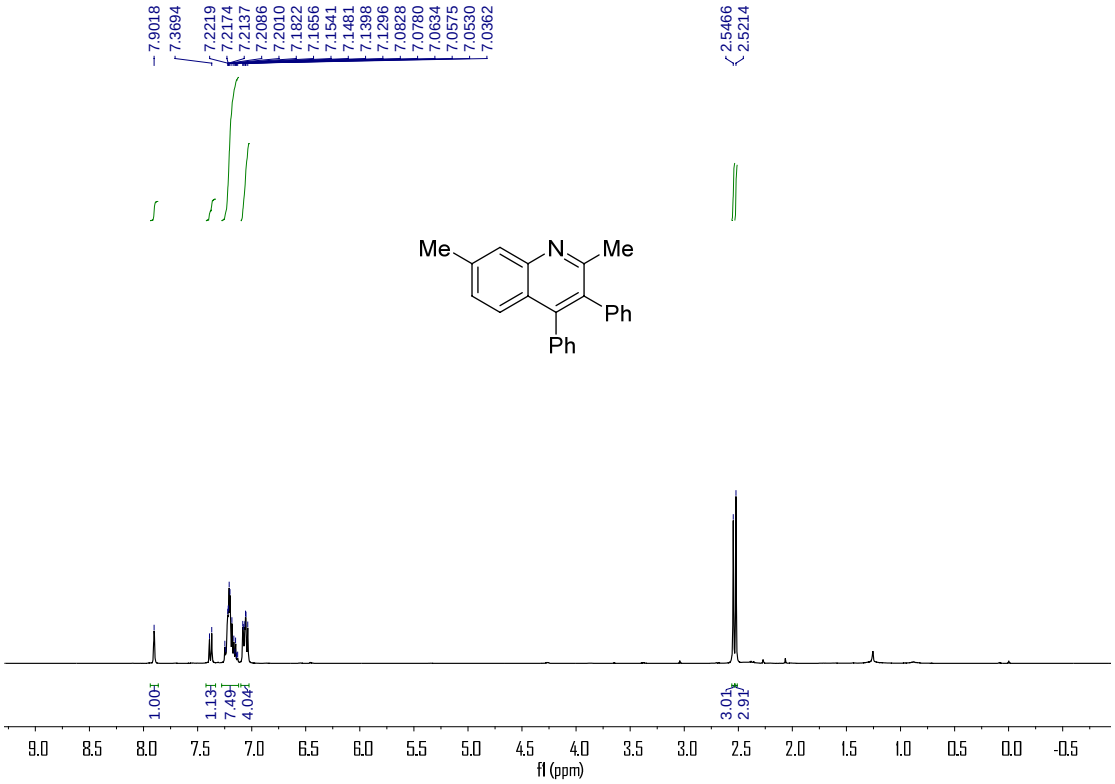
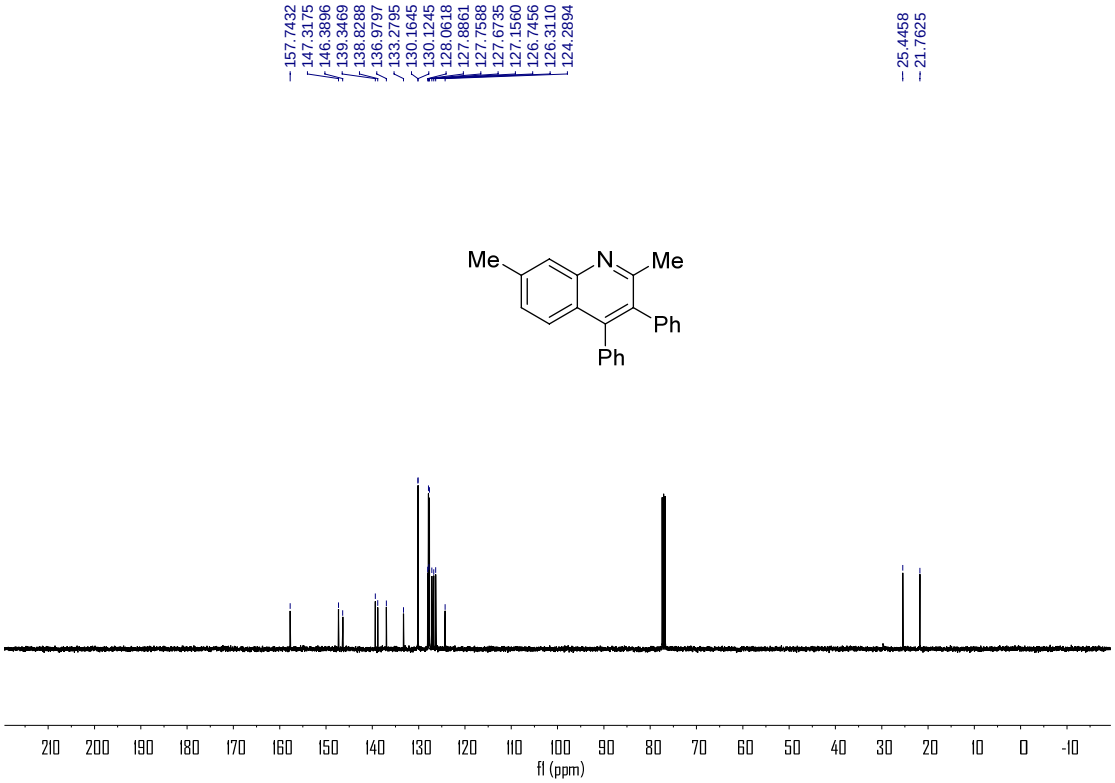
8.2731
8.1451
8.1238
7.2471
7.2310
7.2107
7.1827
7.0994
7.0853
7.0679
7.0507
4.3651
4.3496
4.3324
4.3155
2.5649
1.3587
1.3432
1.3261
N
Me
O
O
Ph
Ph
2.01
1.00
6.25
4.03
2.01
2.93
3.02
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

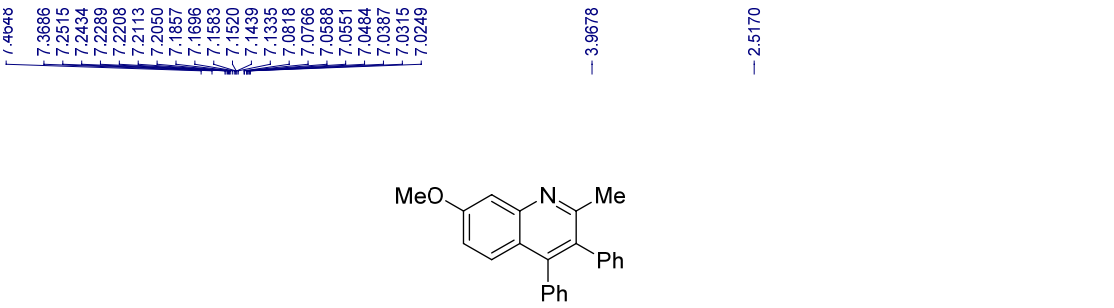

166.4561
160.5201
149.0879
147.9103
138.3449
136.0860
134.9692
130.2323
130.0639
129.9574
129.0588
128.7929
128.1330
127.9673
127.8732
127.7159
127.1447
125.7461
61.2534
25.8574
14.3835
N
Me
O
O
Ph
Ph
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

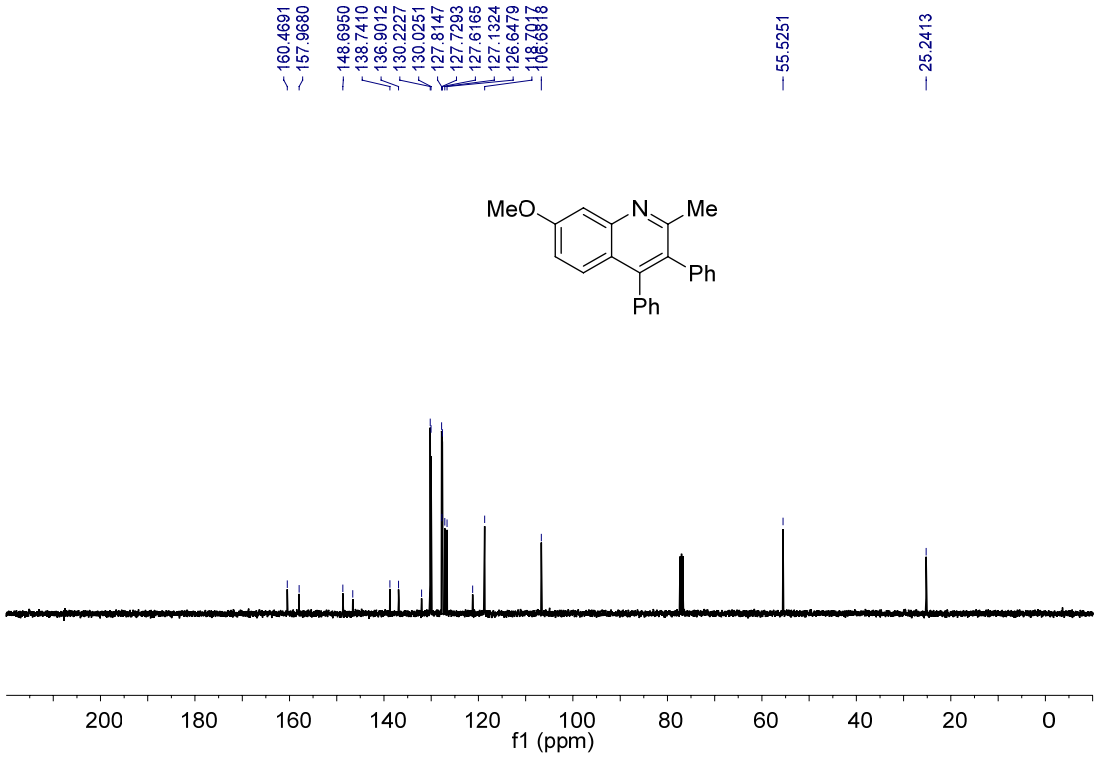
3ka


7.9018
7.3694
7.2219
7.2174
7.2137
7.2086
7.2010
7.1822
7.1656
7.1541
7.1481
7.1398
7.1296
7.0828
7.0780
7.0634
7.0575
7.0530
7.0362
2.5466
2.5214
Me
N
Me
Ph
Ph
1.00
1.13
7.49
4.04
3.01
2.91
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
f1 (ppm)

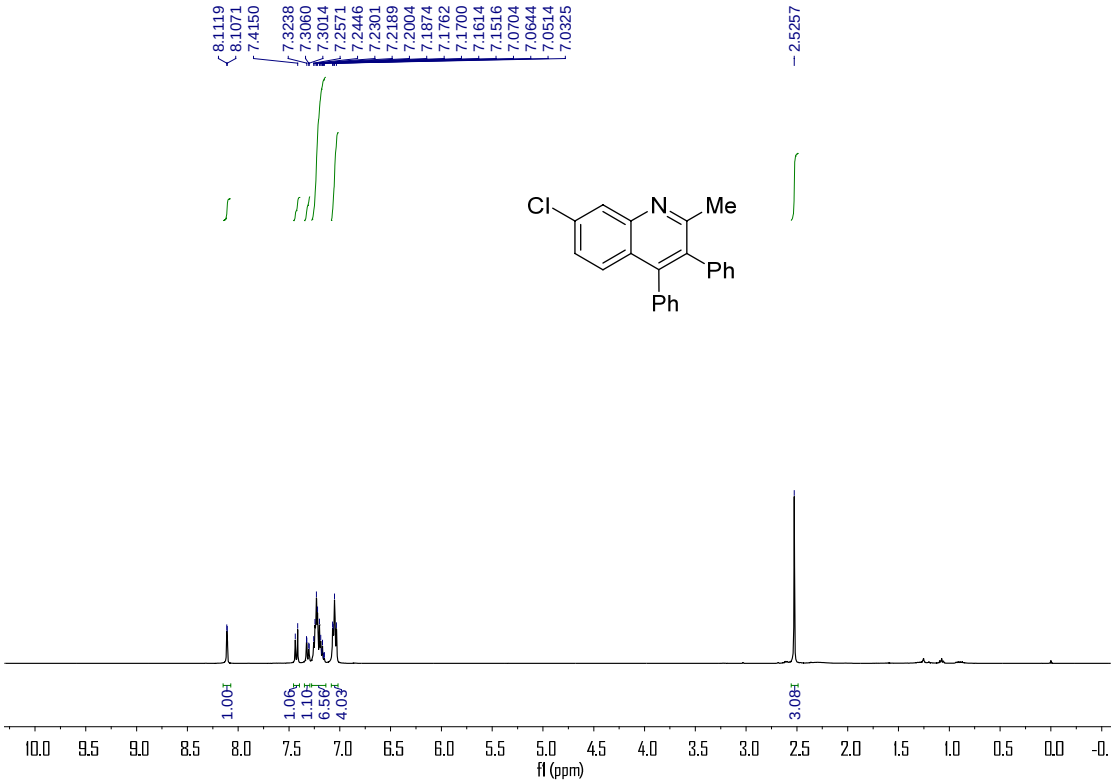

157.7432
147.3175
146.3896
139.3469
138.8288
136.9797
133.2795
130.1645
130.1245
128.0618
127.8861
127.7588
127.6735
127.1560
126.7456
126.3110
124.2894
25.4458
21.7625
Me
N
Me
Ph
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

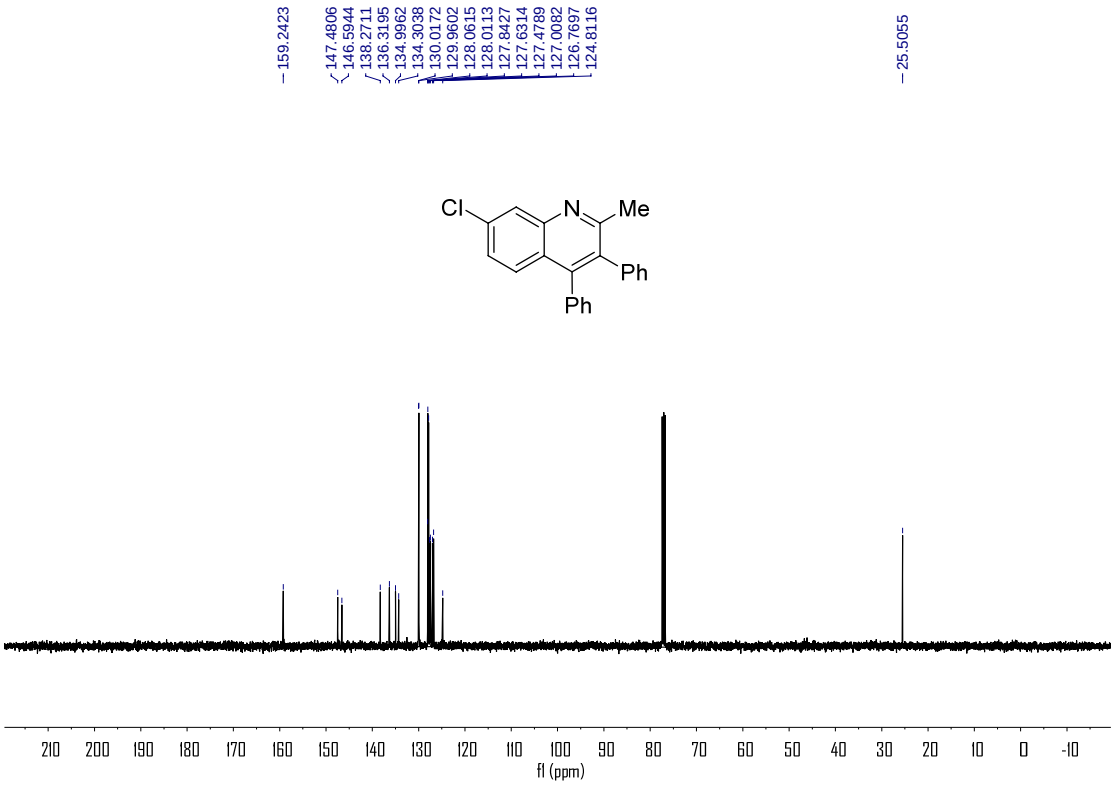
3la


7.4648
7.3686
7.2515
7.2434
7.2289
7.2208
7.2113
7.2050
7.1857
7.1696
7.1583
7.1520
7.1439
7.1335
7.0818
7.0766
7.0588
7.0551
7.0484
7.0387
7.0315
7.0249
3.9678
2.5170
MeO
N
Me
Ph
Ph
1.00
1.04
6.09
5.12
3.09
3.04
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
f1 (ppm)

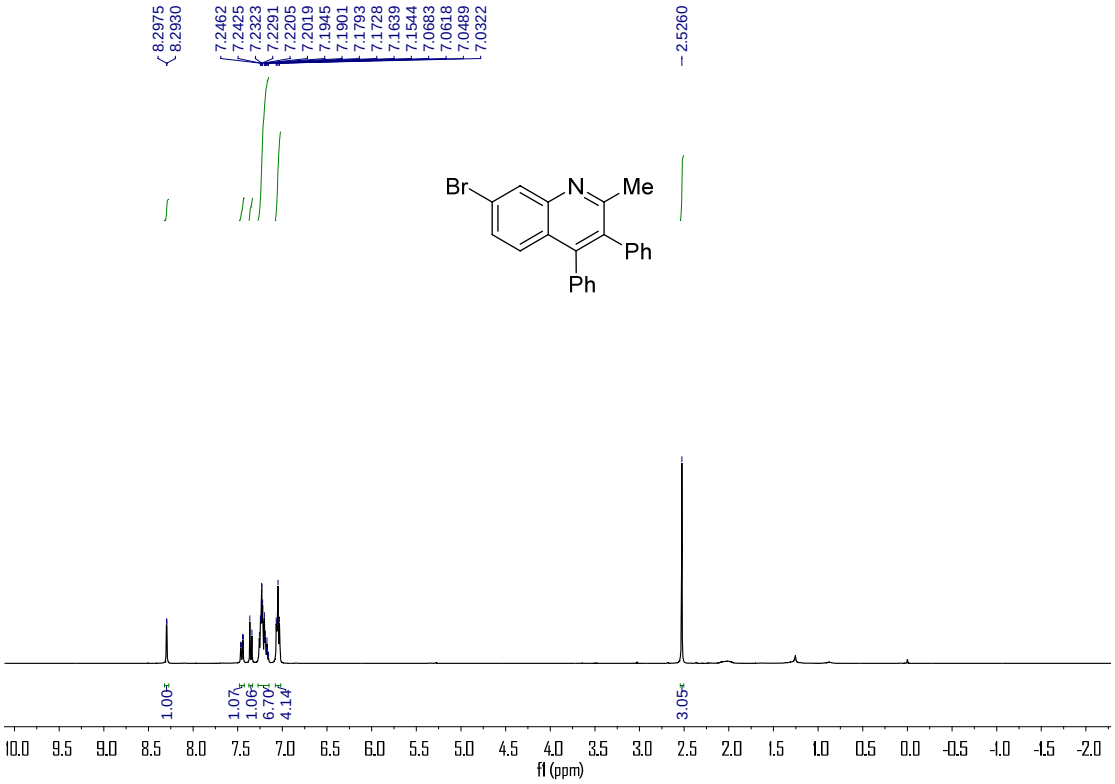

160.4691
157.9680
148.6950
138.7410
136.9012
130.2227
130.0251
127.8147
127.7293
127.6165
127.1324
126.6479
118.7017
108.8818
55.5251
25.2413
MeO
N
Me
Ph
Ph
200 180 160 140 120 100 80 60 40 20 0
f1 (ppm)

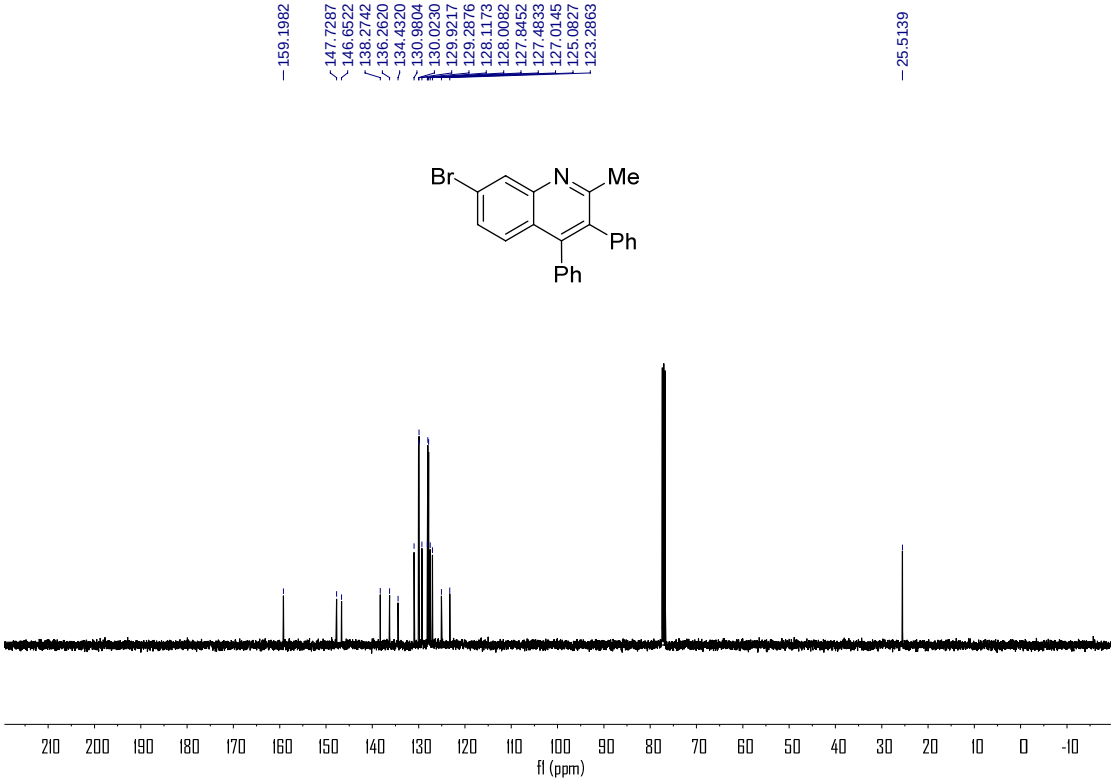
3ma


8.1119
8.1071
7.4150
7.3238
7.3060
7.3014
7.2571
7.2446
7.2301
7.2189
7.2004
7.1874
7.1762
7.1700
7.1614
7.1516
7.0704
7.0644
7.0514
7.0325
2.5257
Cl
N
Me
Ph
Ph
1.00
1.06
1.10
6.56
4.03
3.08
f1 (ppm)

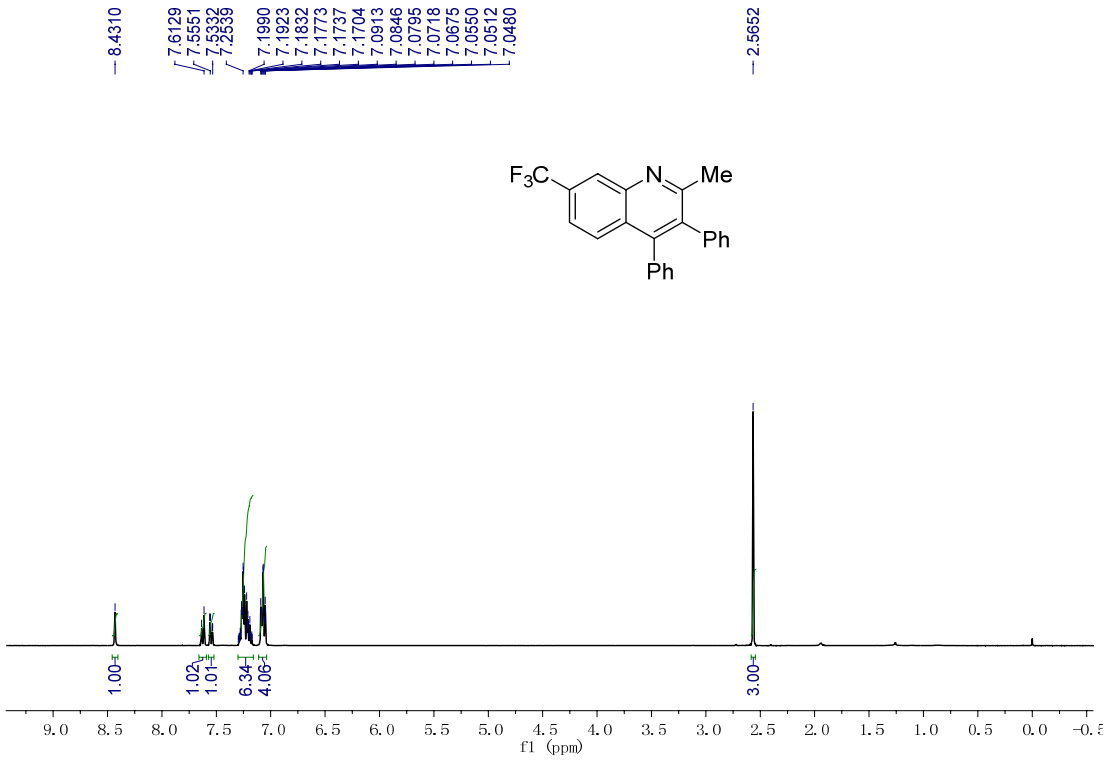

159.2423
147.4806
146.5944
138.2711
136.3195
134.9962
134.3038
130.0172
129.9602
128.0615
128.0113
127.8427
127.6314
127.4789
127.0082
126.7697
124.8116
25.5055
Cl
N
Me
Ph
Ph
f1 (ppm)

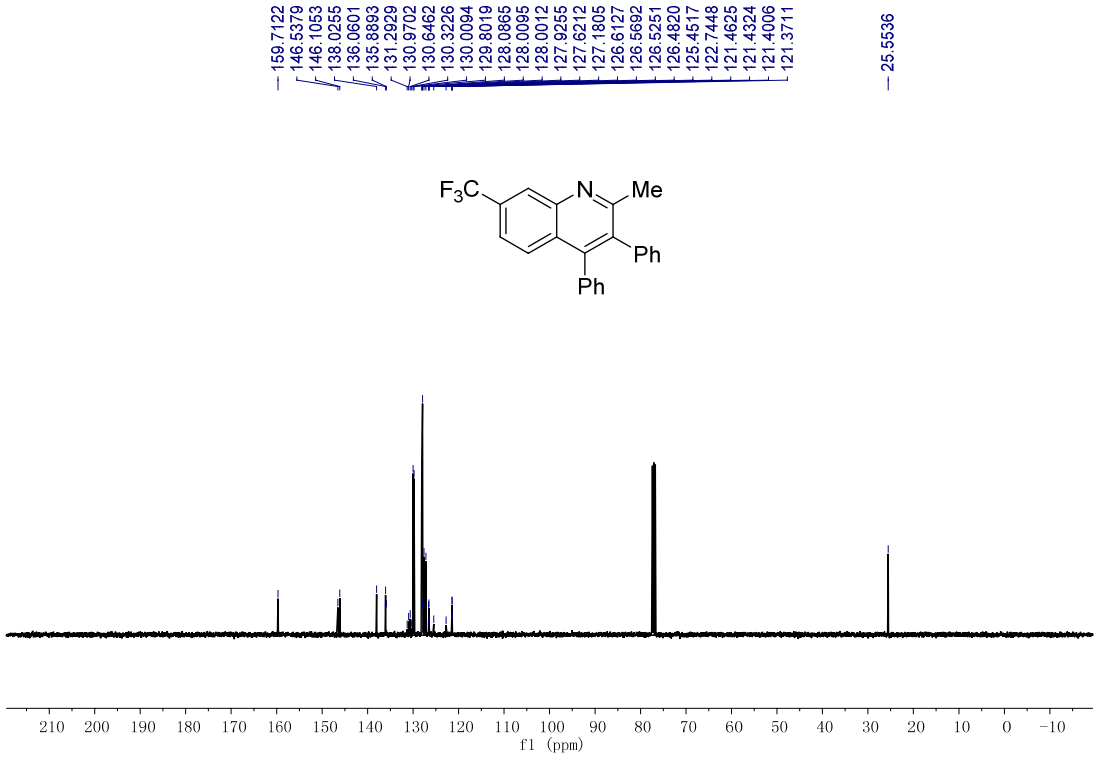
3na


8.2975
8.2930
7.2462
7.2425
7.2323
7.2291
7.2205
7.2019
7.1945
7.1901
7.1793
7.1728
7.1639
7.1544
7.0683
7.0618
7.0499
7.0322
2.5260
Br
N
Me
Ph
Ph
1.00
1.07
1.06
6.70
4.14
3.05
f1 (ppm)


159.1982
147.7287
146.6522
138.2742
136.2620
134.4320
130.9804
130.0230
129.9217
129.2876
128.1173
128.0082
127.8452
127.4833
127.0145
125.0827
123.2863
25.5139
Br
N
Me
Ph
Ph
f1 (ppm)

3oa


8.4310
7.6129
7.5551
7.5332
7.2539
7.1990
7.1923
7.1832
7.1773
7.1737
7.1704
7.0913
7.0846
7.0795
7.0718
7.0675
7.0550
7.0512
7.0480
2.5652
F3C
N
Me
Ph
Ph
1.00
1.02
1.01
6.34
4.06
3.00
f1 (ppm)


159.7122
146.5379
146.1053
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136.0601
135.8893
131.2929
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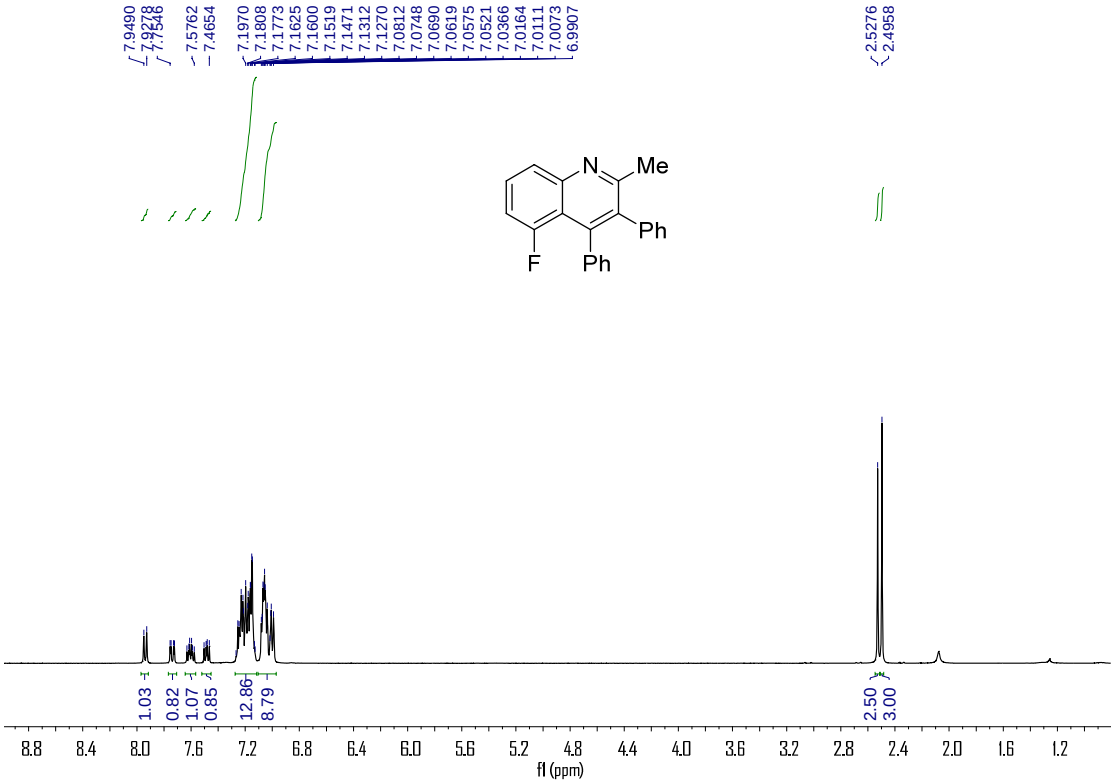
130.6462
130.3226
130.0094
129.8019
128.0865
128.0095
128.0012
127.9255
127.6212
127.1805
126.6127
126.5692
126.5251
126.4820
125.4517
122.7448
121.4625
121.4324
121.4006
121.3711
25.5536
F3C
N
Me
Ph
Ph
f1 (ppm)

3pa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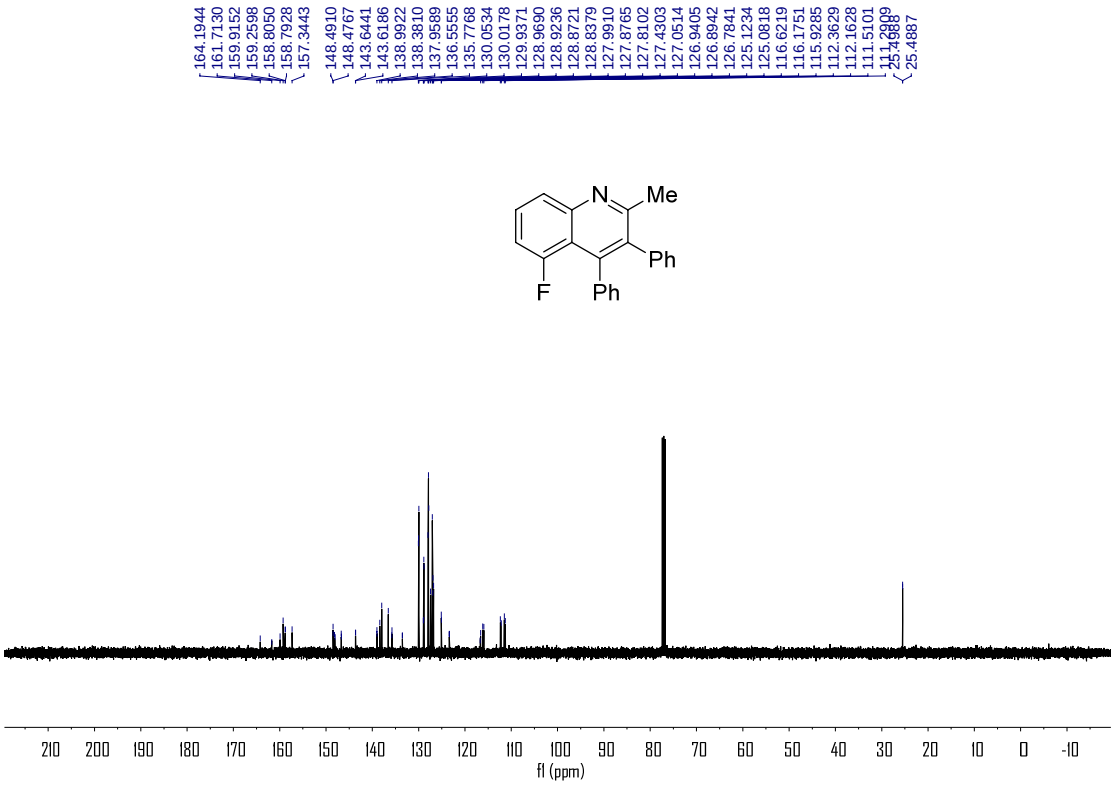
7.9278
7.7546
7.5762
7.4654
7.1970
7.1808
7.1773
7.1625
7.1600
7.1519
7.1471
7.1312
7.1270
7.0812
7.0748
7.0690
7.0619
7.0575
7.0521
7.0366
7.0164
7.0111
7.0073
6.9907
2.5276
2.4958
N
Me
Ph
F
Ph
1.03
0.82
1.07
0.85
12.86
8.79
2.50
3.00
8.8
8.4
8.0
7.6
7.2
6.8
6.4
6.0
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
f1 (ppm)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158.8050
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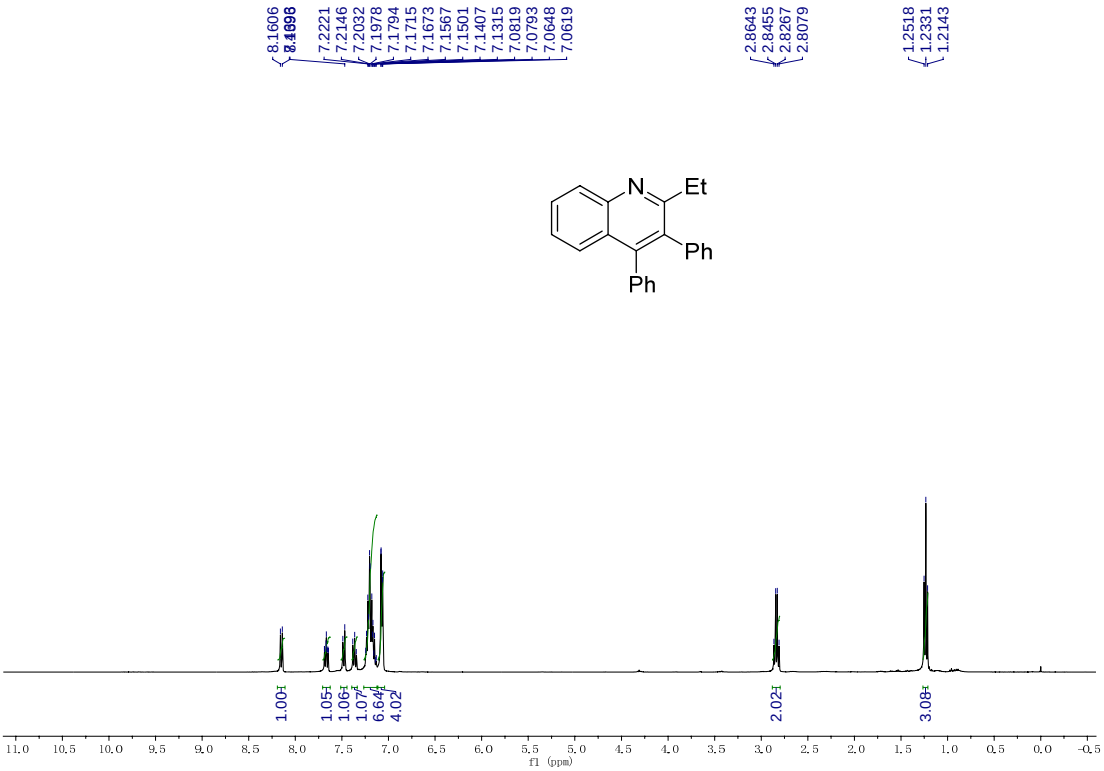
157.3443
148.4910
148.4767
143.6441
143.6186
138.9922
138.3810
137.9589
136.5555
135.7768
130.0534
130.0178
129.9371
128.9690
128.9236
128.8721
128.8379
127.9910
127.8765
127.8102
127.4303
127.0514
126.9405
126.8942
126.7841
125.1234
125.0818
116.6219
116.1751
115.9285
112.3629
112.1628
111.5101
25.4887
N
Me
Ph
F
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

3qa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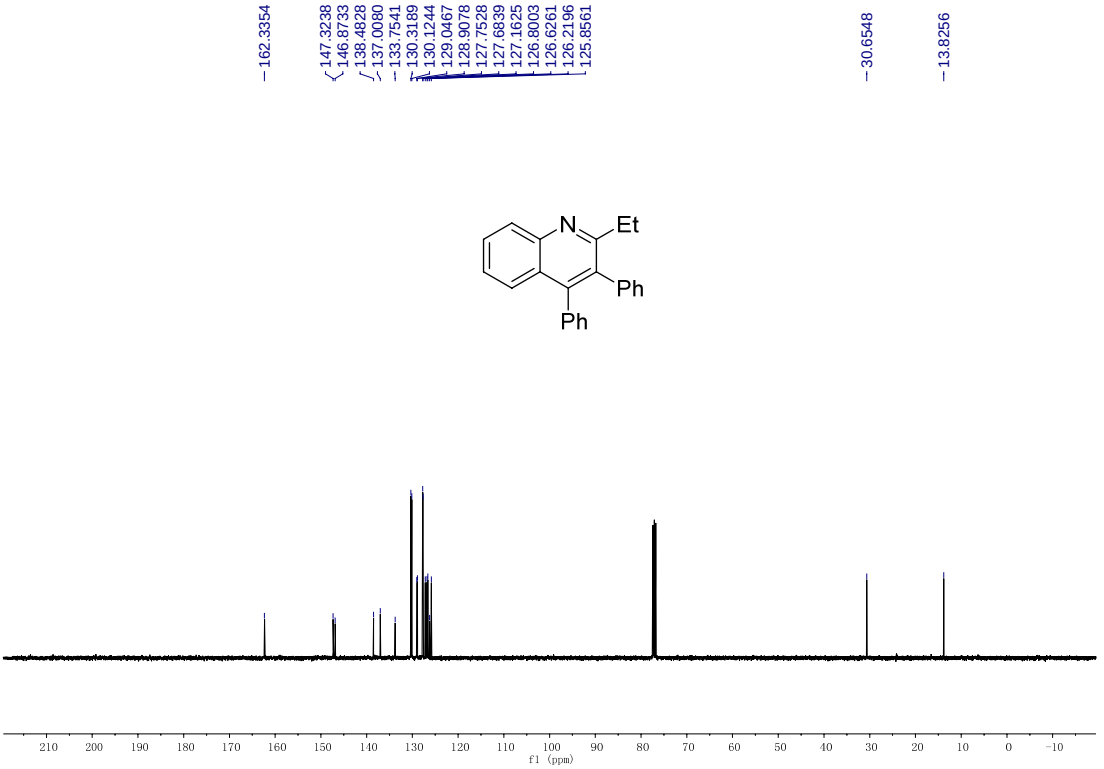
7.2221
7.2146
7.2032
7.1978
7.1794
7.1715
7.1673
7.1567
7.1501
7.1407
7.1315
7.0819
7.0793
7.0648
7.0619
2.8643
2.8455
2.8267
2.8079
1.2518
1.2331
1.2143
N
Et
Ph
Ph
1.00
1.05
1.06
1.07
6.64
4.02
2.02
3.08
f1 (ppm)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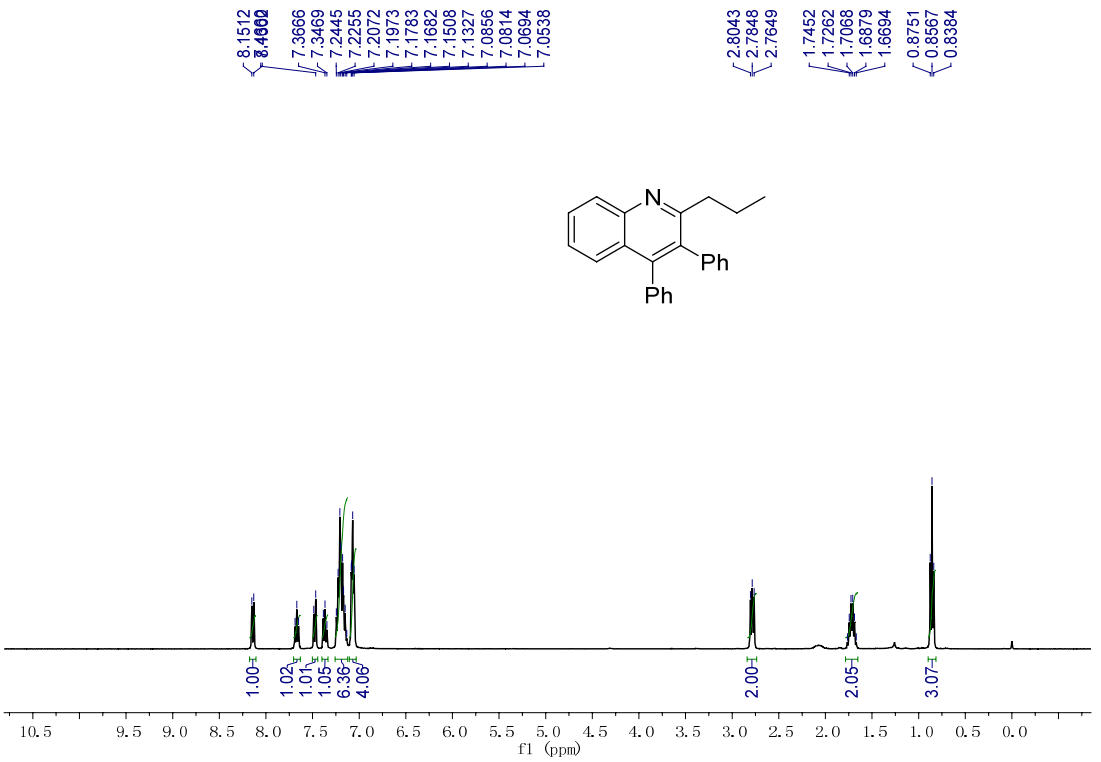
133.7541
130.3189
130.1244
129.0467
128.9078
127.7528
127.6839
127.1625
126.8003
126.6261
126.2196
125.8561
30.6548
13.8256
N
Et
Ph
Ph
f1 (ppm)

3ra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7.3469
7.2445
7.2255
7.2072
7.1973
7.1783
7.1682
7.1508
7.1327
7.0856
7.0814
7.0694
7.0538
2.8043
2.7848
2.7649
1.7452
1.7262
1.7068
1.6879
1.6694
0.8751
0.8567
0.8384
N
Ph
Ph
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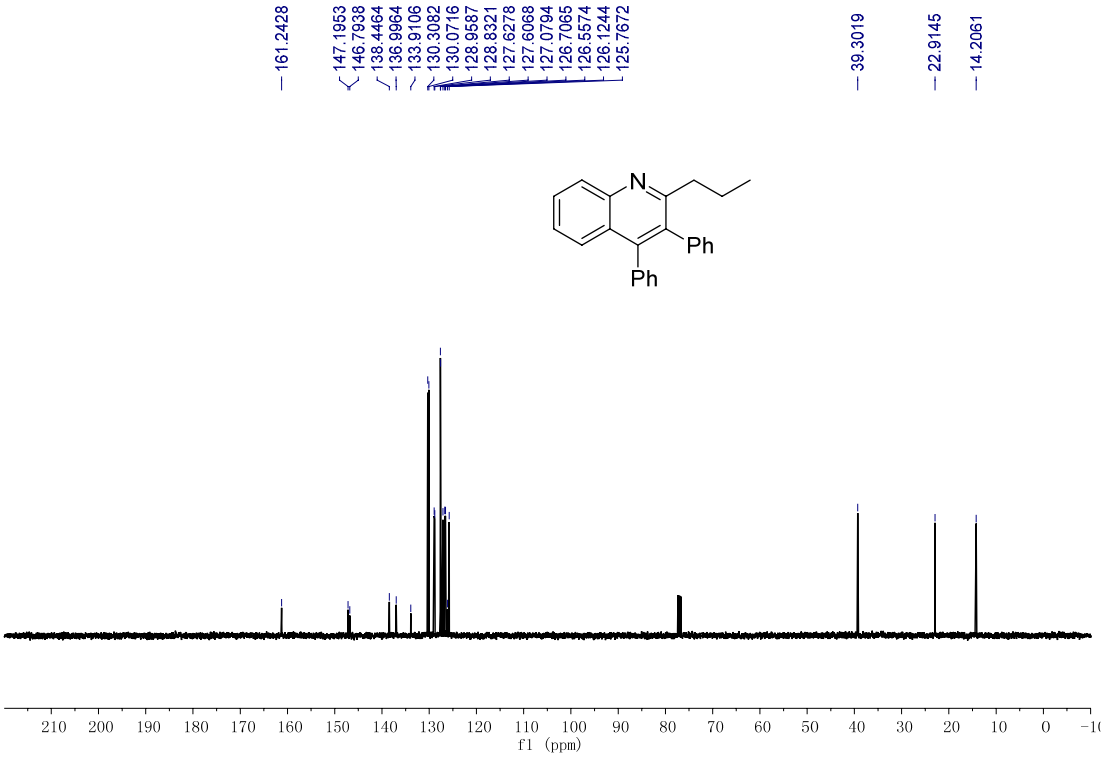
1.02
1.01
1.05
6.36
4.06
2.00
2.05
3.07
f1 (ppm)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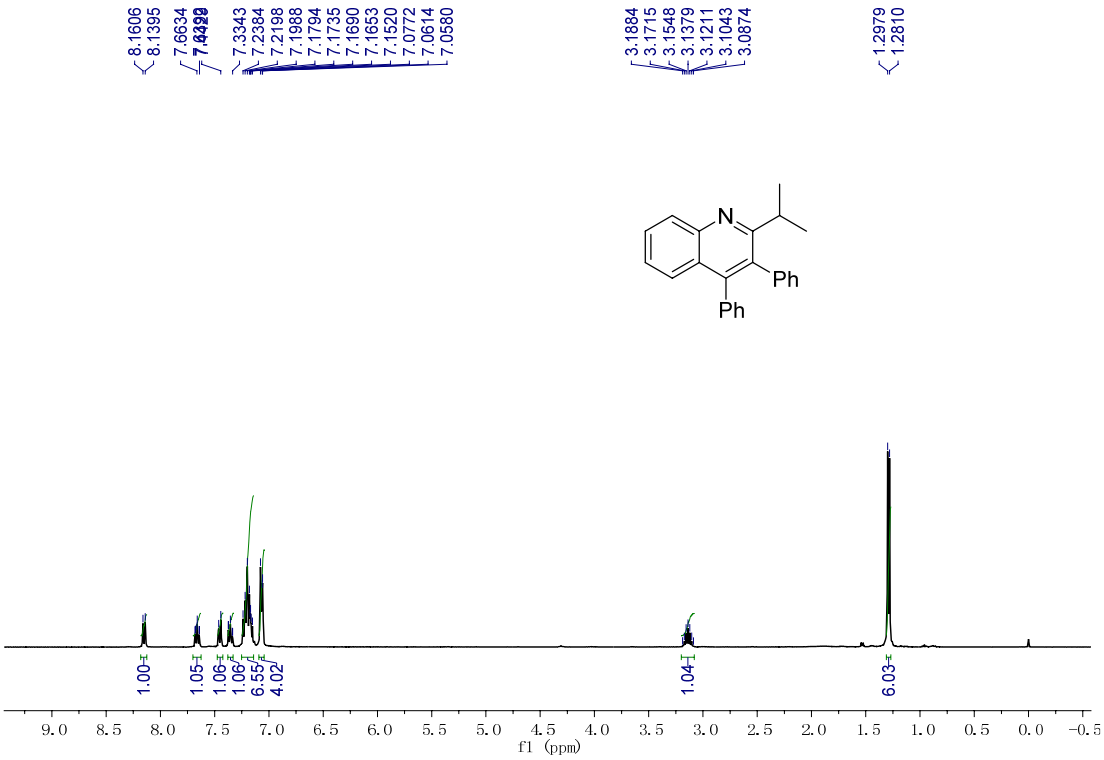
146.7938
138.4464
136.9964
133.9106
130.3082
130.0716
128.9587
128.8321
127.6278
127.6068
127.0794
126.7065
126.5574
126.1244
125.7672
39.3019
22.9145
14.2061
N
Ph
Ph
f1 (ppm)

3sa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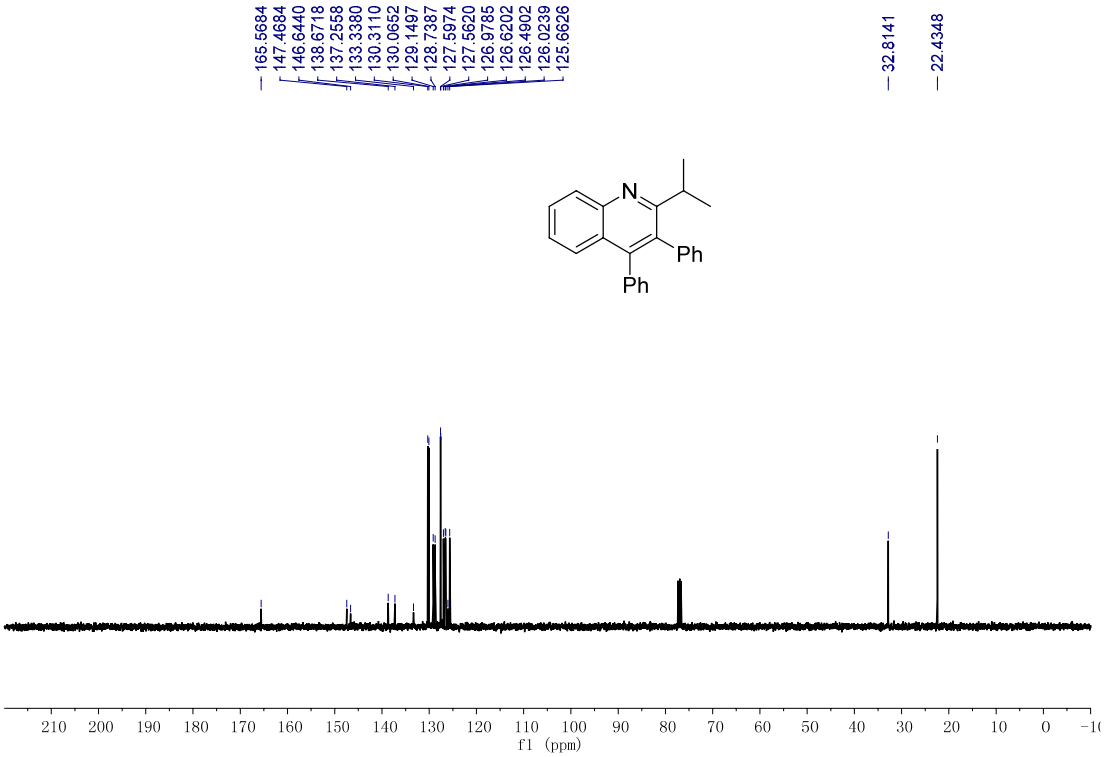
8.1395
7.6634
7.3343
7.2384
7.2198
7.1988
7.1794
7.1735
7.1690
7.1653
7.1520
7.0772
7.0614
7.0580
3.1884
3.1715
3.1548
3.1379
3.1211
3.1043
3.0874
1.2979
1.2810
N
Ph
Ph
1.00
1.05
1.06
1.06
6.55
4.02
1.04
6.03
f1 (ppm)


165.5684
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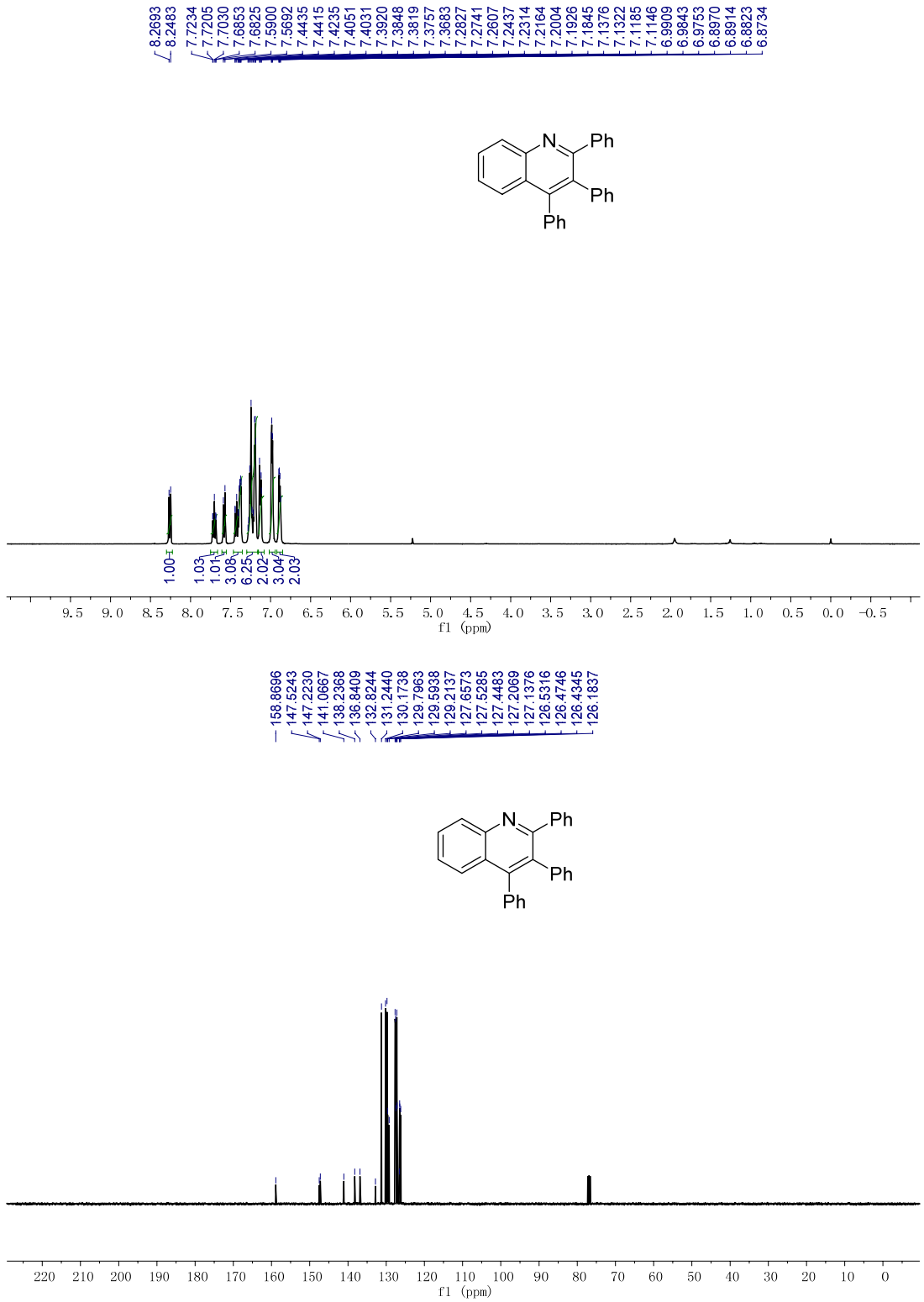
146.6440
138.6718
137.2558
133.3380
130.3110
130.0652
129.1497
128.7387
127.5974
127.5620
126.9785
126.6202
126.4902
126.0239
125.6626
32.8141
22.4348
N
Ph
Ph
f1 (ppm)

3ta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7.6853
7.6825
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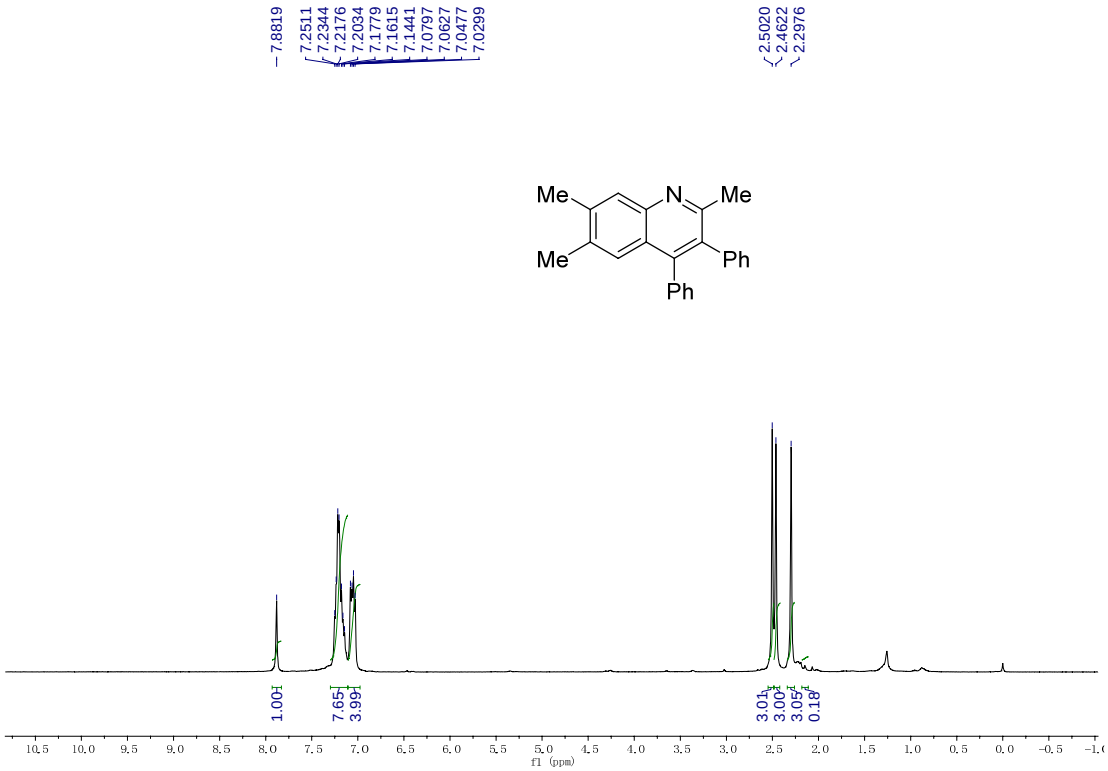
7.5692
7.4435
7.4415
7.4235
7.4051
7.4031
7.3920
7.3848
7.3819
7.3757
7.3683
7.2827
7.2741
7.2607
7.2437
7.2314
7.2164
7.2004
7.1926
7.1845
7.1376
7.1322
7.1185
7.1146
6.9909
6.9843
6.9753
6.8970
6.8914
6.8823
6.8734
N
Ph
Ph
Ph
1.00
1.03
1.01
3.08
6.25
2.02
3.04
2.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
158.8696
147.5243
147.2230
141.0667
138.2368
136.8409
132.8244
131.2440
130.1738
129.7963
129.5938
129.2137
127.6573
127.5285
127.4483
127.2069
127.1376
126.5316
126.4746
126.4345
126.1837
N
Ph
Ph
Ph
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3wa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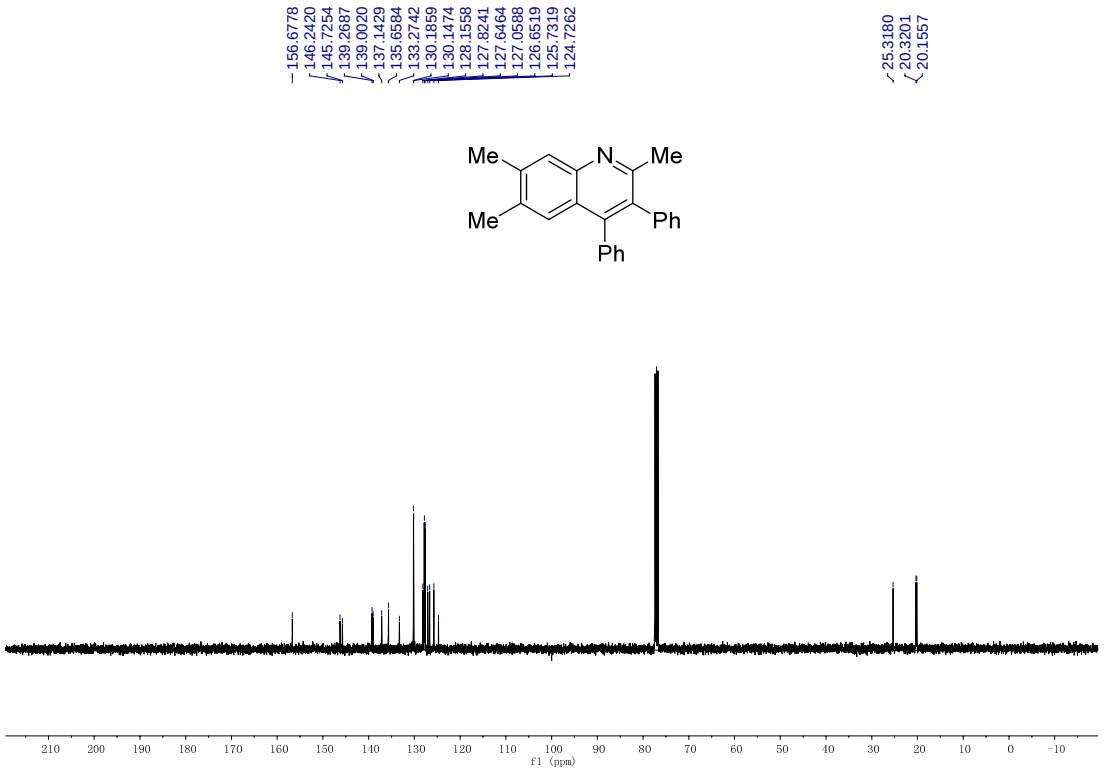
7.2344
7.2176
7.2034
7.1779
7.1615
7.1441
7.0797
7.0627
7.0477
7.0299
2.5020
2.4622
2.2976
Me
N
Me
Me
Ph
Ph
1.00
7.65
3.99
3.01
3.00
3.05
0.18
f1 (ppm)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145.7254
139.2687
139.0020
137.1429
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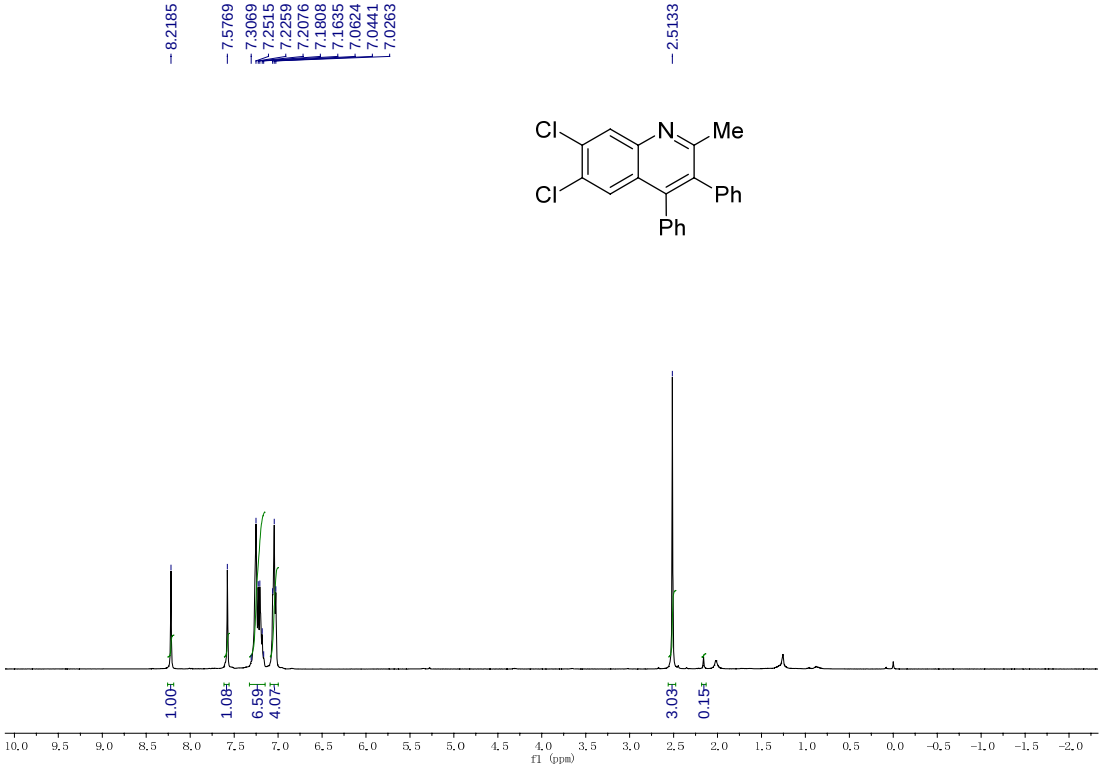
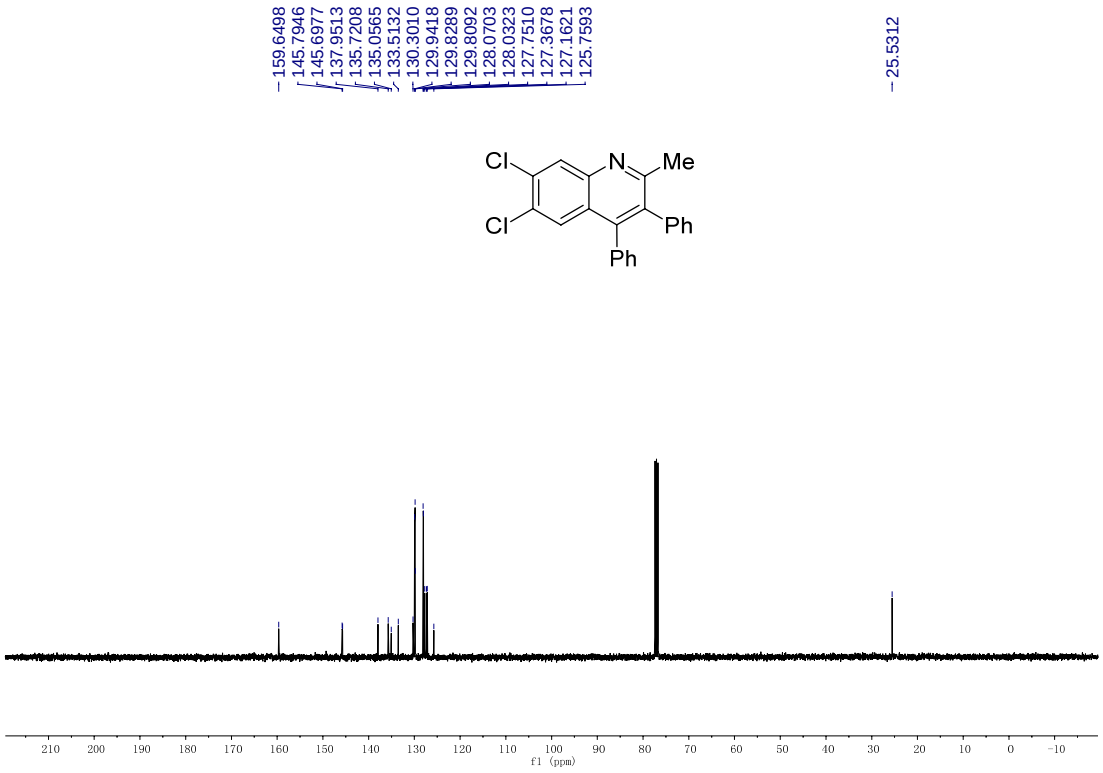
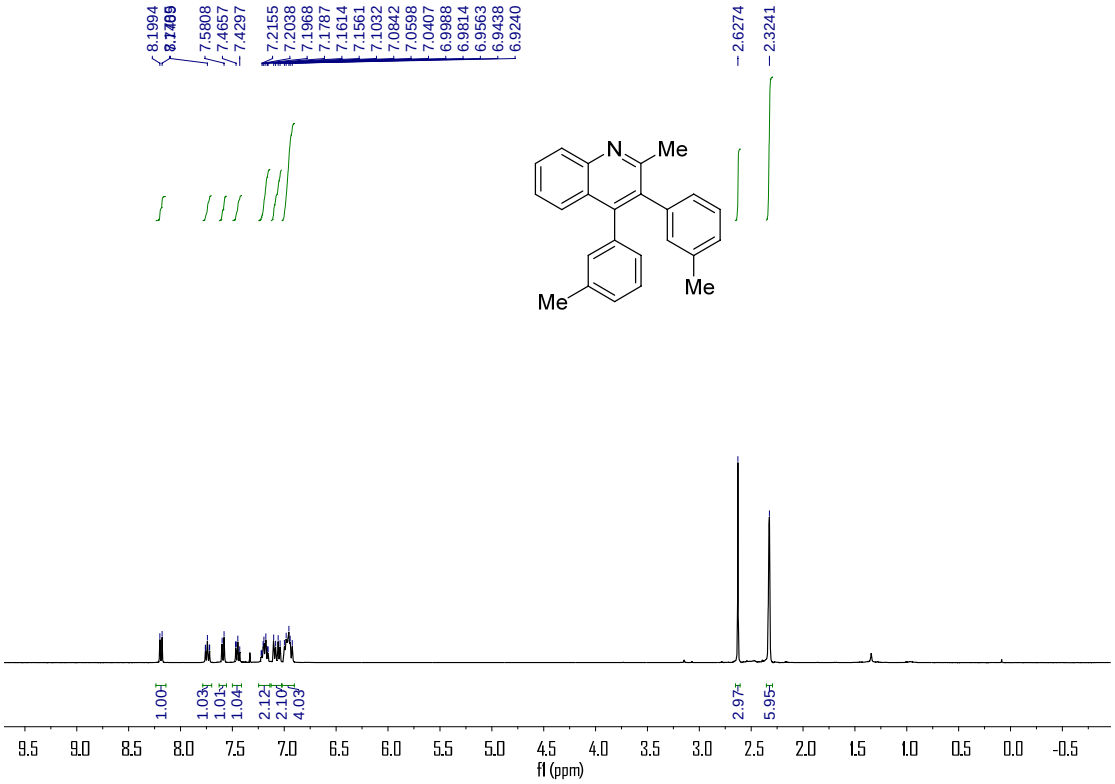
133.2742
130.1859
130.1474
128.1558
127.8241
127.6464
127.0588
126.6519
125.7319
124.7262
25.3180
20.3201
20.1557
Me
N
Me
Me
Ph
Ph
f1 (ppm)

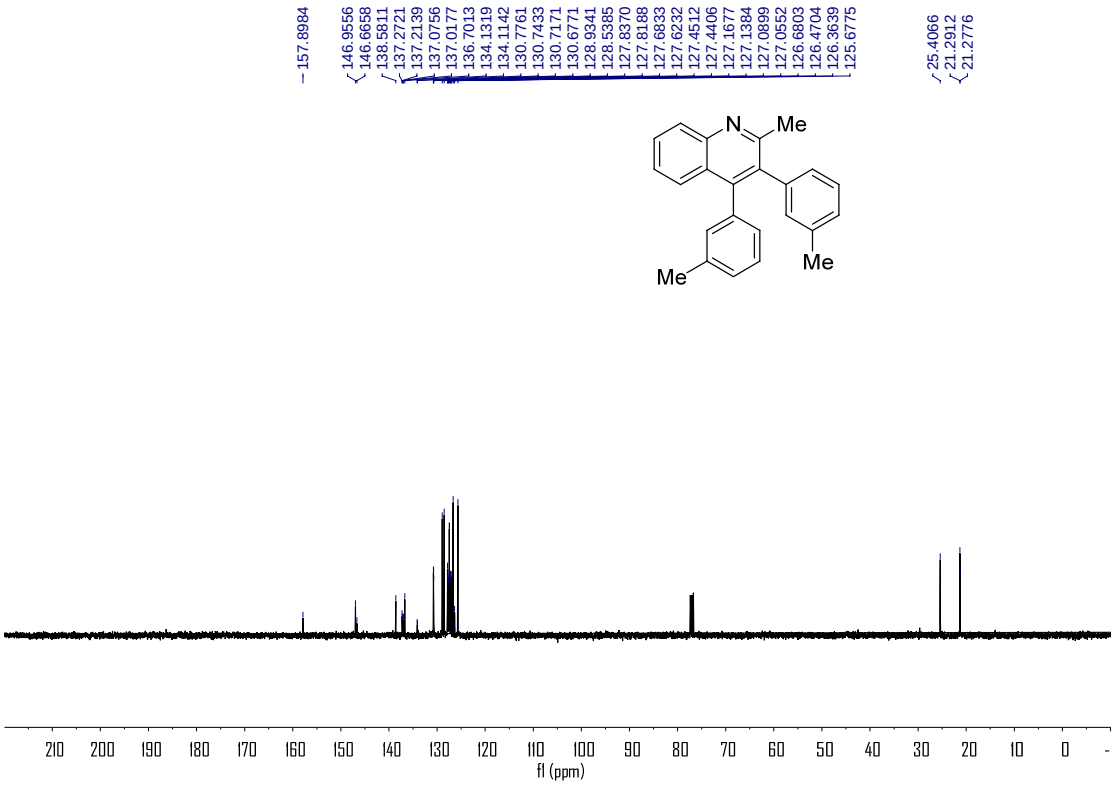
3xa


8.2185
7.5769
7.3069
7.2515
7.2259
7.2076
7.1808
7.1635
7.0624
7.0441
7.0263
2.5133
Cl
N
Me
Cl
Ph
Ph
1.00
1.08
6.59
4.07
3.03
0.15
f1 (ppm)

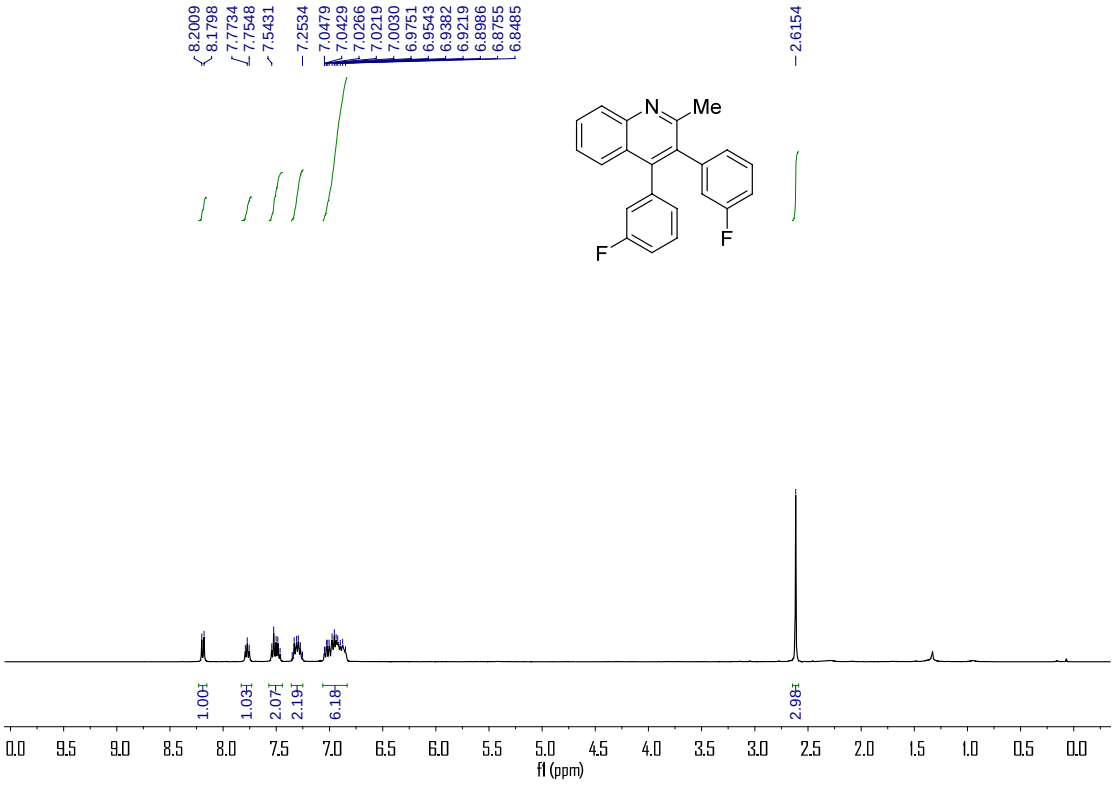

159.6498
145.7946
145.6977
137.9513
135.7208
135.0565
133.5132
130.3010
129.9418
129.8289
129.8092
128.0703
128.0323
127.7510
127.3678
127.1621
125.7593
25.5312
Cl
N
Me
Cl
Ph
Ph
f1 (ppm)

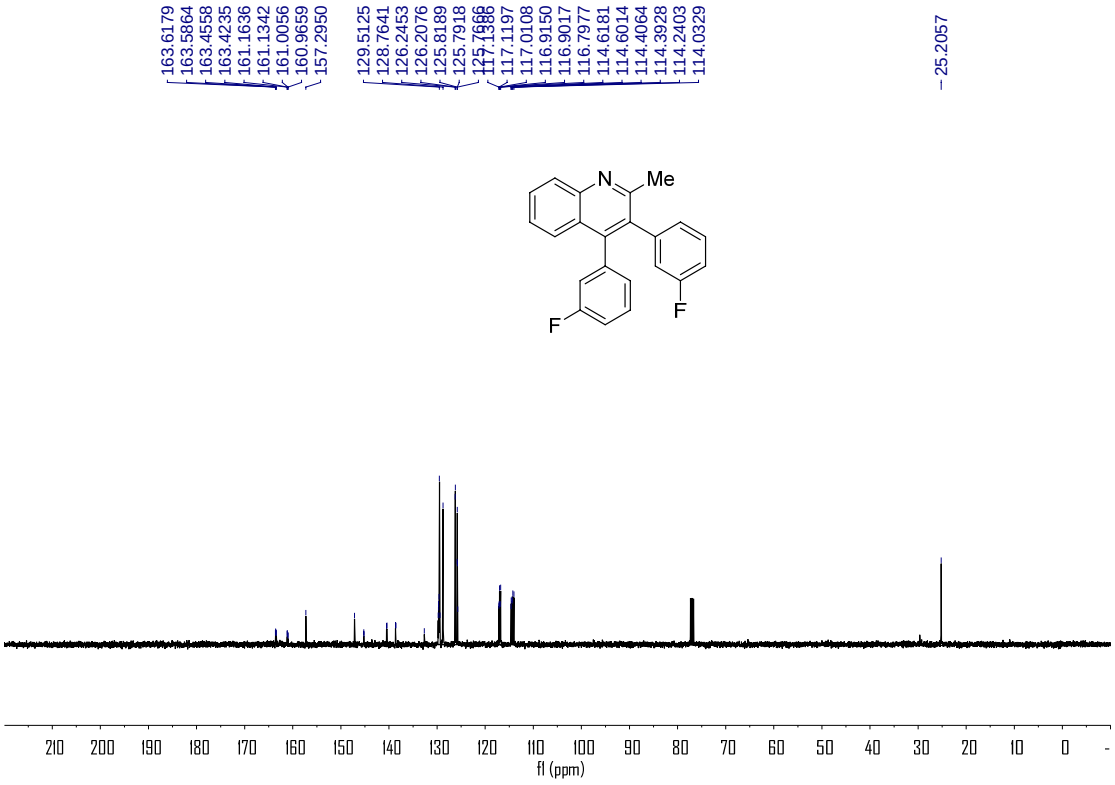
3ab


8.1994
8.1409
7.5808
7.4657
7.4297
7.2155
7.2038
7.1968
7.1787
7.1614
7.1561
7.1032
7.0842
7.0598
7.0407
6.9988
6.9814
6.9563
6.9438
6.9240
2.6274
2.3241
N
Me
Me
Me
1.00
1.03
1.01
1.04
2.12
2.10
4.03
2.97
5.95
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
f1 (ppm)

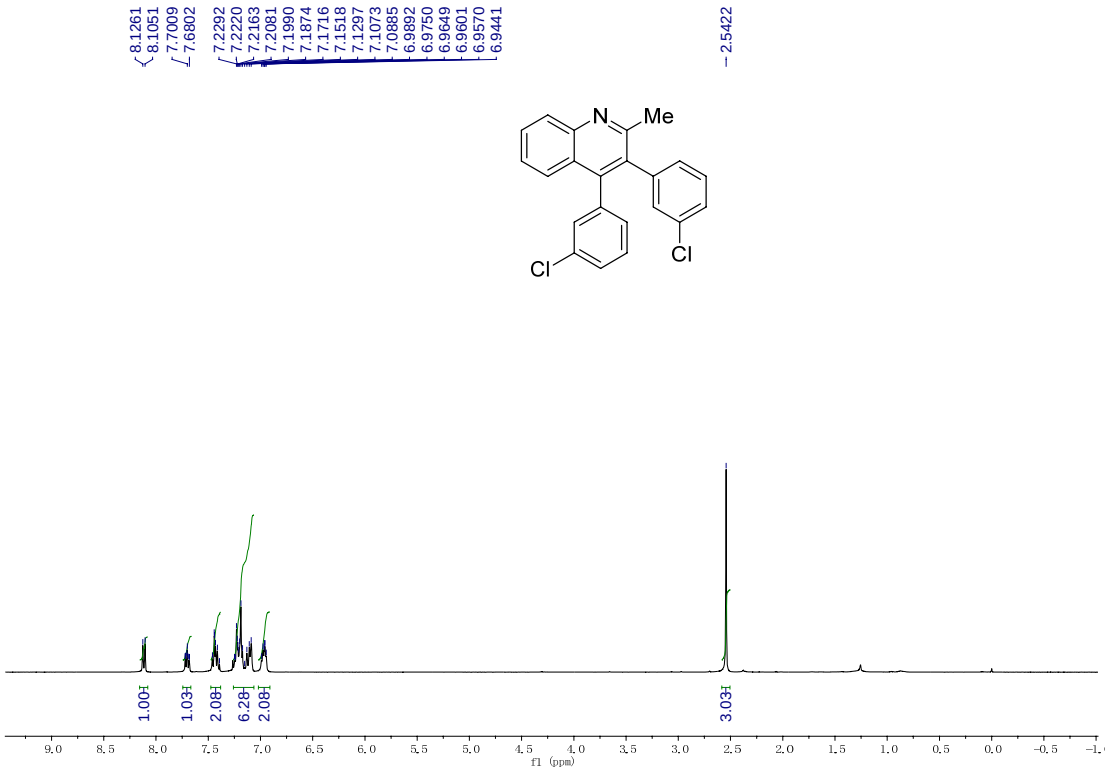

157.8984
146.9556
146.6658
138.5811
137.2721
137.2139
137.0756
137.0177
136.7013
134.1319
134.1142
130.7761
130.7433
130.7171
130.6771
128.9341
128.5385
127.8370
127.8188
127.6833
127.6232
127.4512
127.4406
127.1677
127.1384
127.0899
127.0552
126.6803
126.4704
126.3639
125.6775
25.4066
21.2912
21.2776
N
Me
Me
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

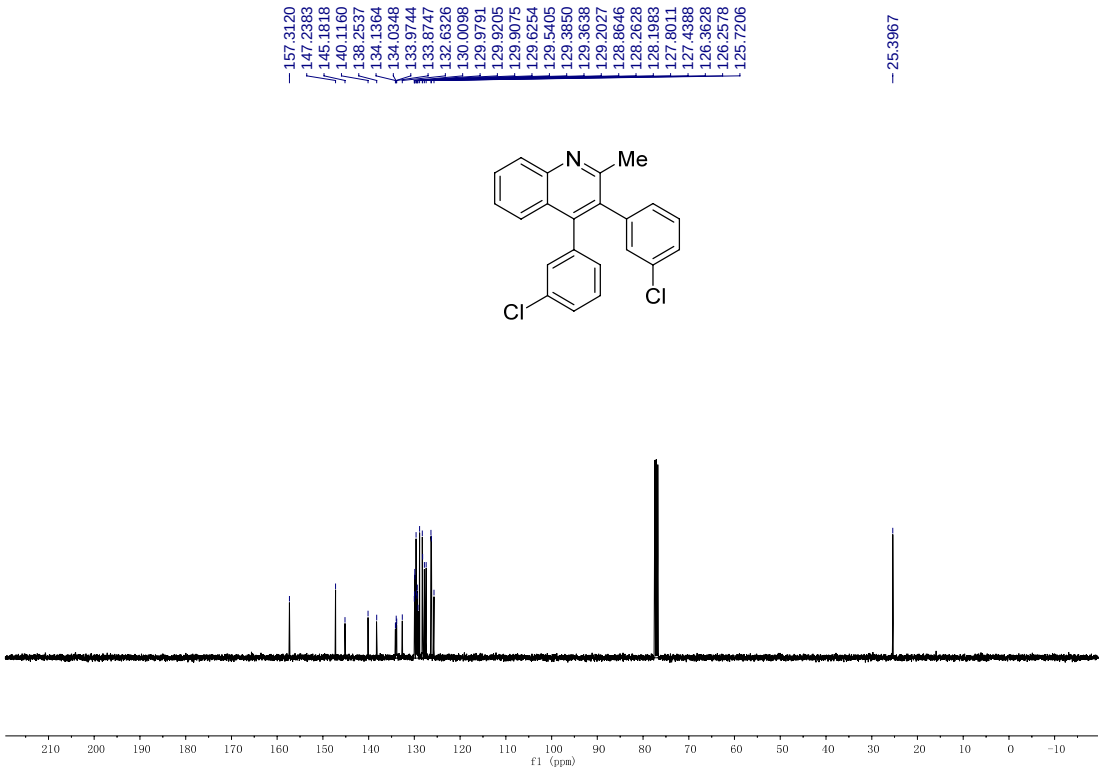
3ac


8.2009
8.1798
7.7734
7.7548
7.5431
7.2534
7.0479
7.0429
7.0266
7.0219
7.0030
6.9751
6.9543
6.9382
6.9219
6.8986
6.8755
6.8485
2.6154
N
Me
F
F
1.00
1.03
2.07
2.19
6.18
2.98
f1 (ppm)

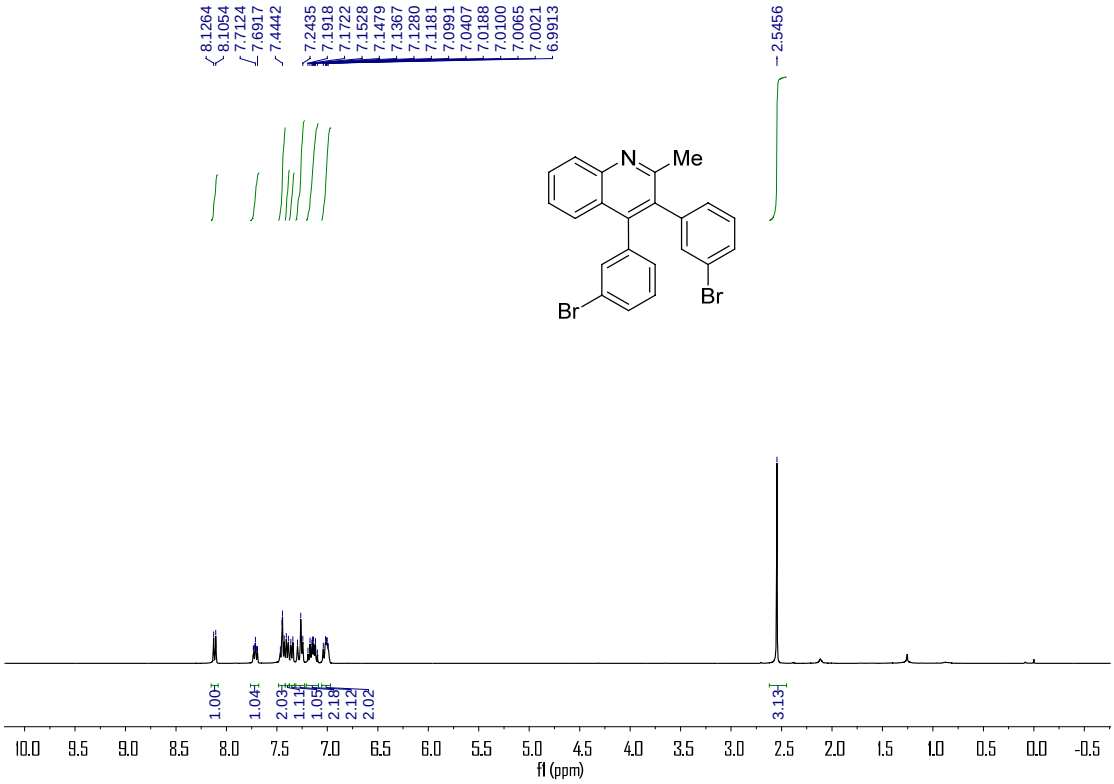

163.6179
163.5864
163.4558
163.4235
161.1636
161.1342
161.0056
160.9659
157.2950
129.5125
128.7641
126.2453
126.2076
125.8189
125.7918
117.1197
117.0108
116.9150
116.9017
116.7977
114.6181
114.6014
114.4064
114.3928
114.2403
114.0329
25.2057
N
Me
F
F
f1 (ppm)

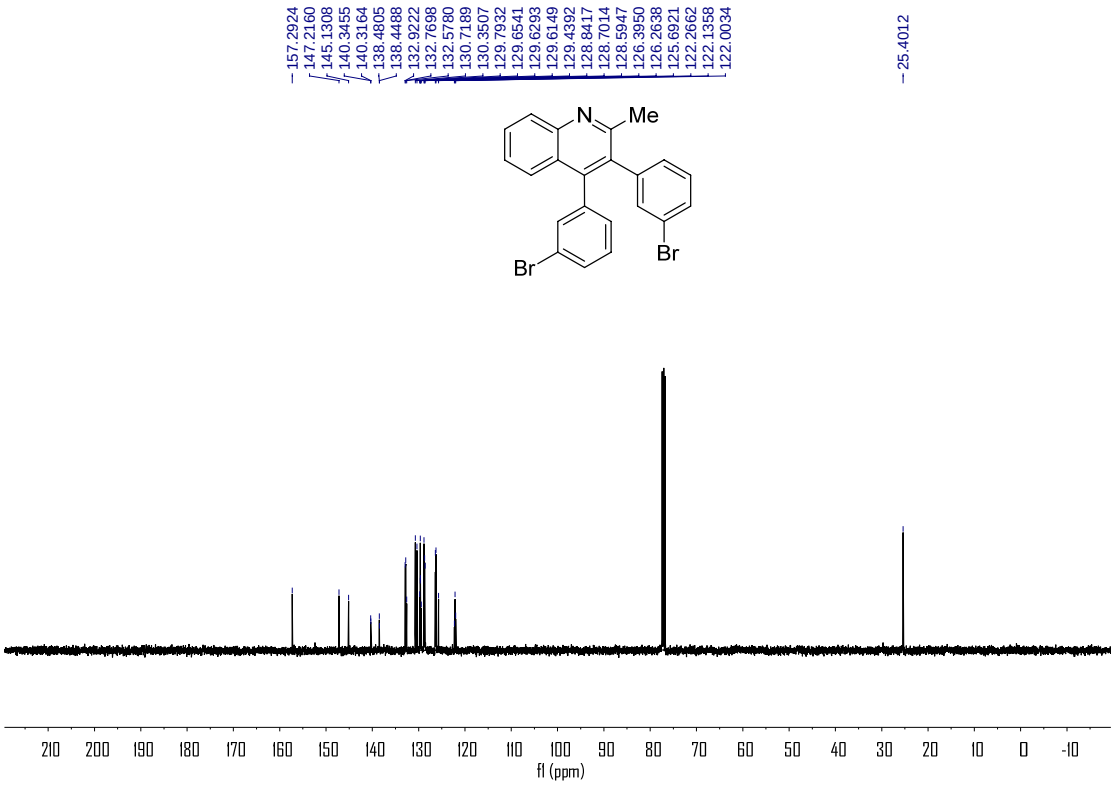
3ad


8.1261
8.1051
7.7009
7.6802
7.2292
7.2220
7.2163
7.2081
7.1990
7.1874
7.1716
7.1518
7.1297
7.1073
7.0885
6.9892
6.9750
6.9649
6.9601
6.9570
6.9441
2.5422
N
Me
Cl
Cl
1.00
1.03
2.08
6.28
2.08
3.03
f1 (ppm)

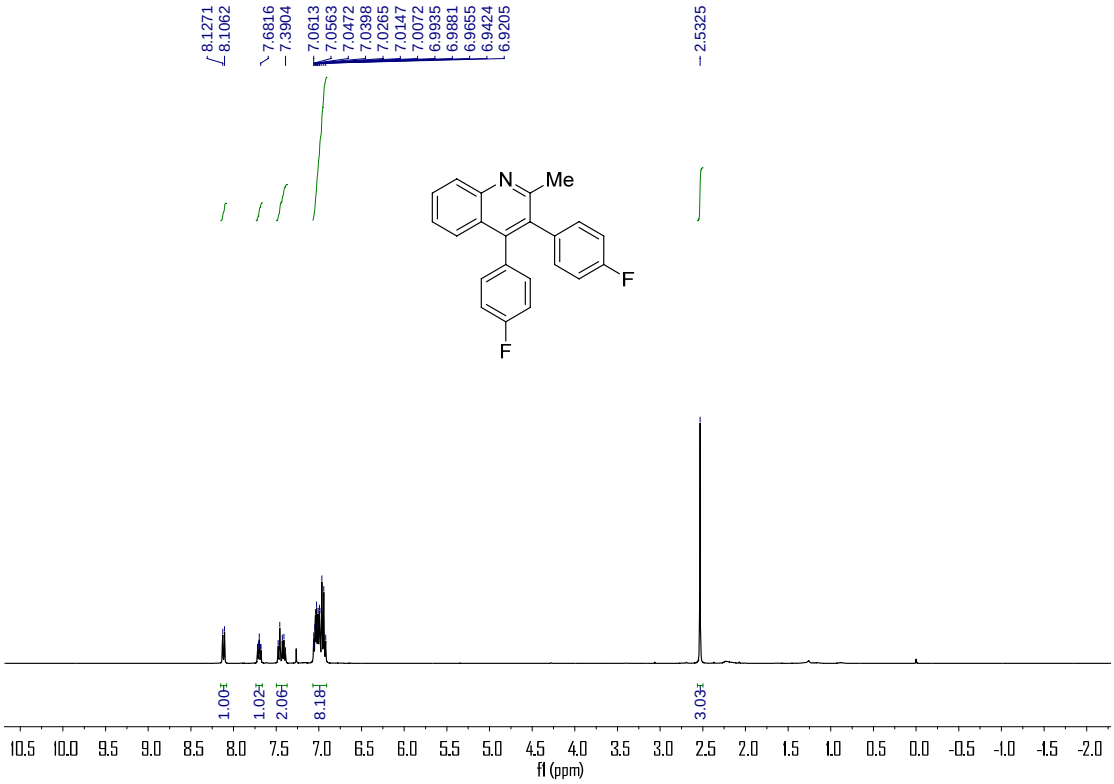

157.3120
147.2383
145.1818
140.1160
138.2537
134.1364
134.0348
133.9744
133.8747
132.6326
130.0098
129.9791
129.9205
129.9075
129.6254
129.5405
129.3850
129.3638
129.2027
128.8646
128.2628
128.1983
127.8011
127.4388
126.3628
126.2578
125.7206
25.3967
N
Me
Cl
Cl
f1 (ppm)

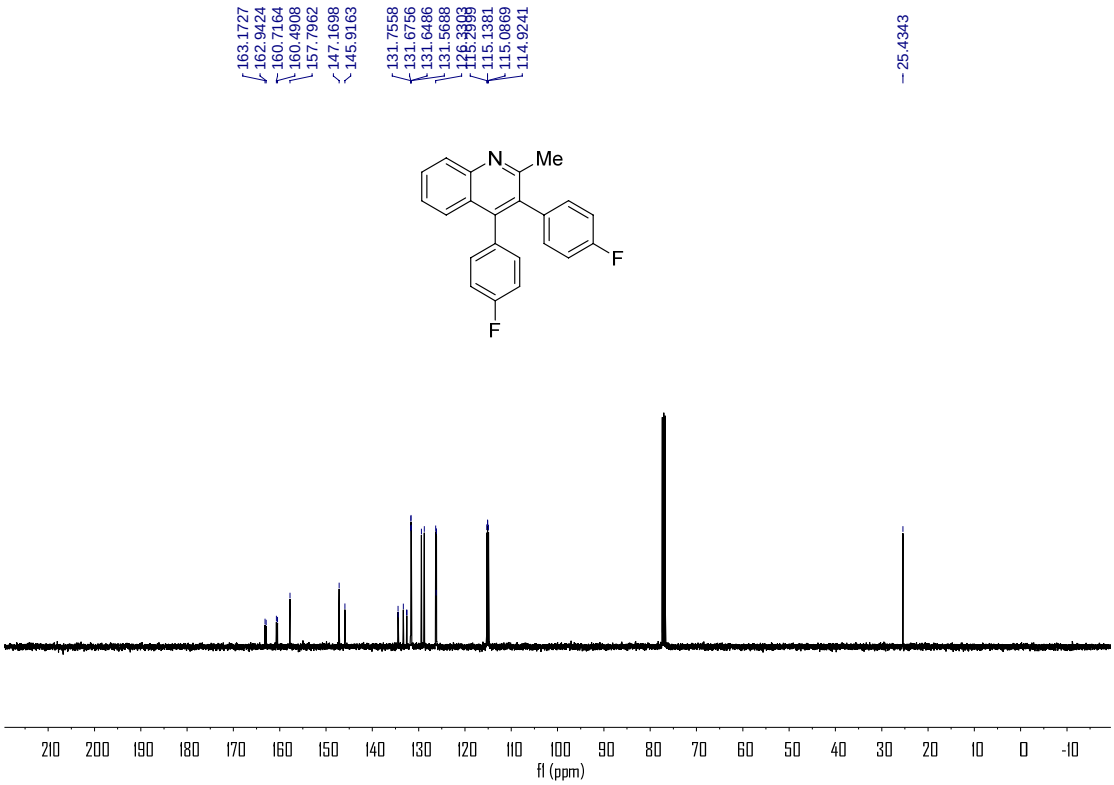
3ae


8.1264
8.1054
7.7124
7.6917
7.4442
7.2435
7.1918
7.1722
7.1528
7.1479
7.1367
7.1280
7.1181
7.0991
7.0407
7.0188
7.0100
7.0065
7.0021
6.9913
2.5456
N
Me
Br
Br
1.00
1.04
2.03
1.11
1.05
2.18
2.12
2.02
3.13
f1 (ppm)

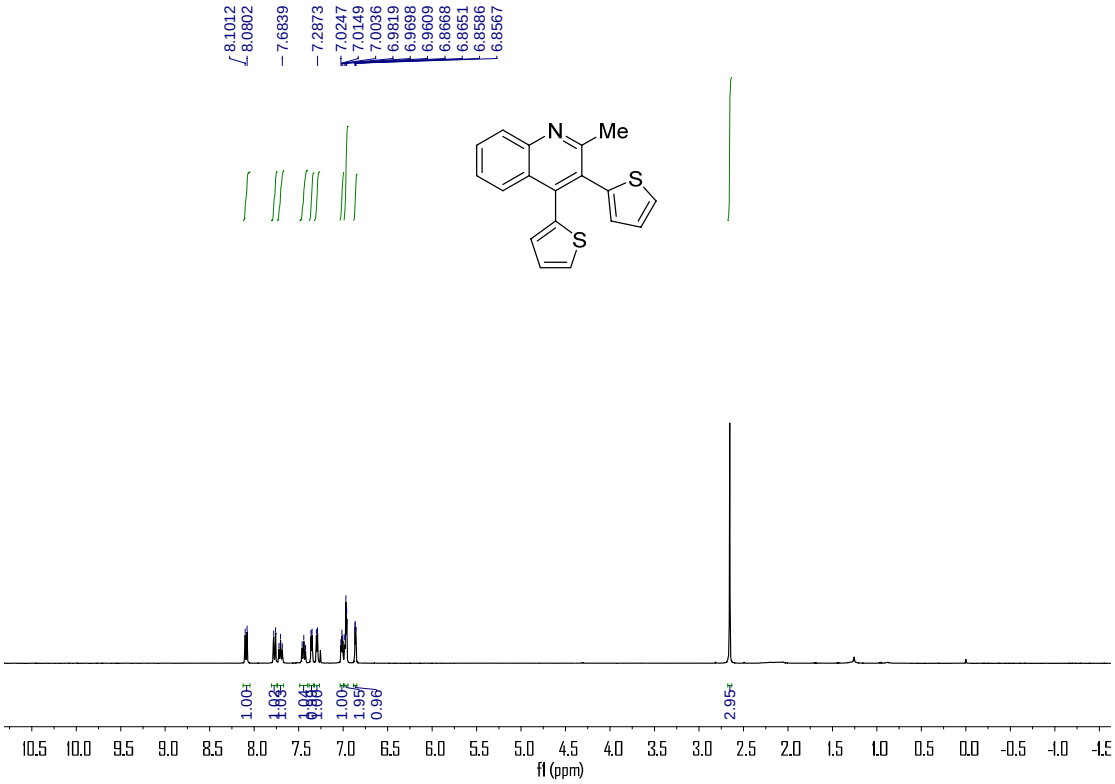

157.2924
147.2160
145.1308
140.3455
140.3164
138.4805
138.4488
132.9222
132.7698
132.5780
130.7189
130.3507
129.7932
129.6541
129.6293
129.6149
129.4392
128.8417
128.7014
128.5947
126.3950
126.2638
125.6921
122.2662
122.1358
122.0034
25.4012
N
Me
Br
Br
f1 (ppm)

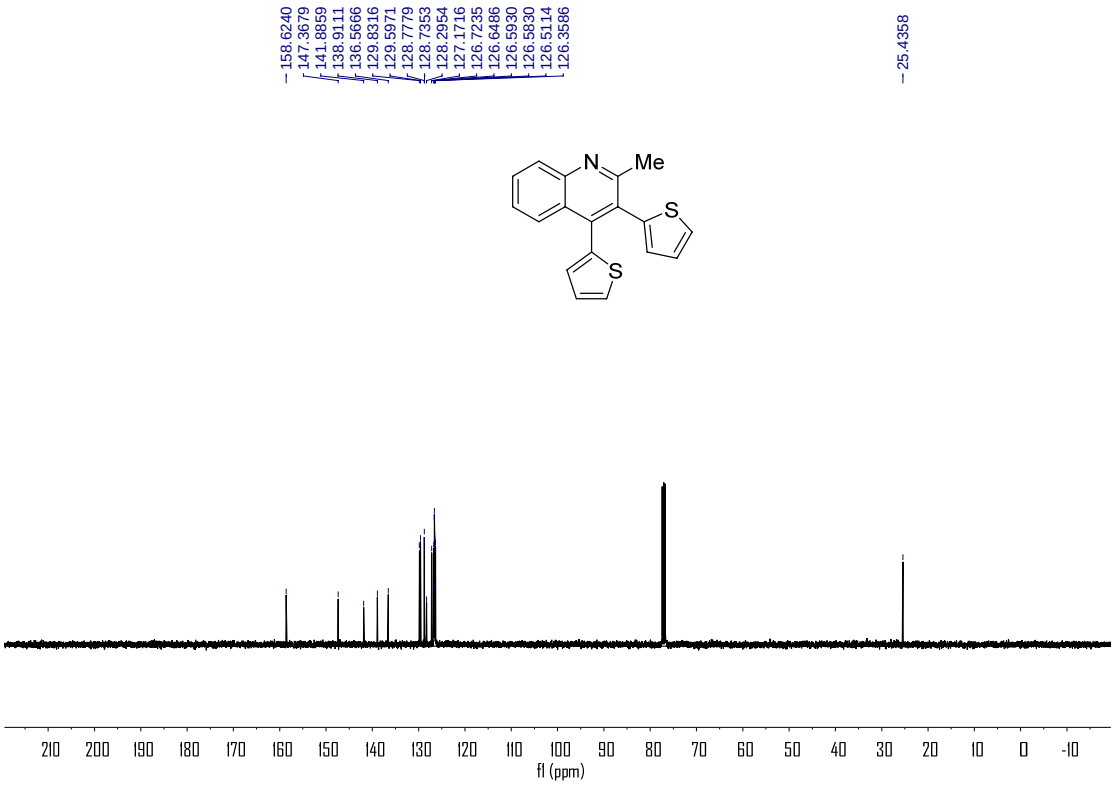
3af


8.1271
8.1062
7.6816
7.3904
7.0613
7.0563
7.0472
7.0398
7.0265
7.0147
7.0072
6.9935
6.9881
6.9655
6.9424
6.9205
2.5325
N
Me
F
F
1.00
1.02
2.06
8.18
3.03
f1 (ppm)

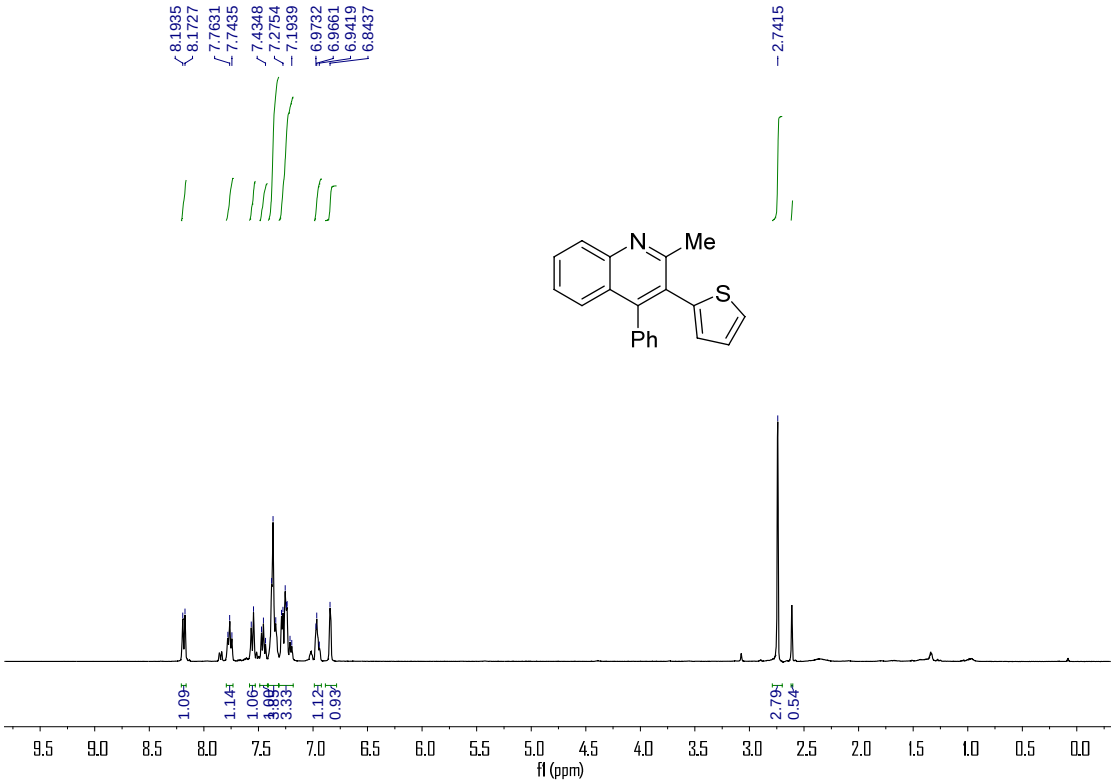

163.1727
162.9424
160.7164
160.4908
157.7962
147.1698
145.9163
131.7558
131.6756
131.6486
131.5688
126.3303
115.1381
115.0869
114.9241
25.4343
N
Me
F
F
f1 (ppm)

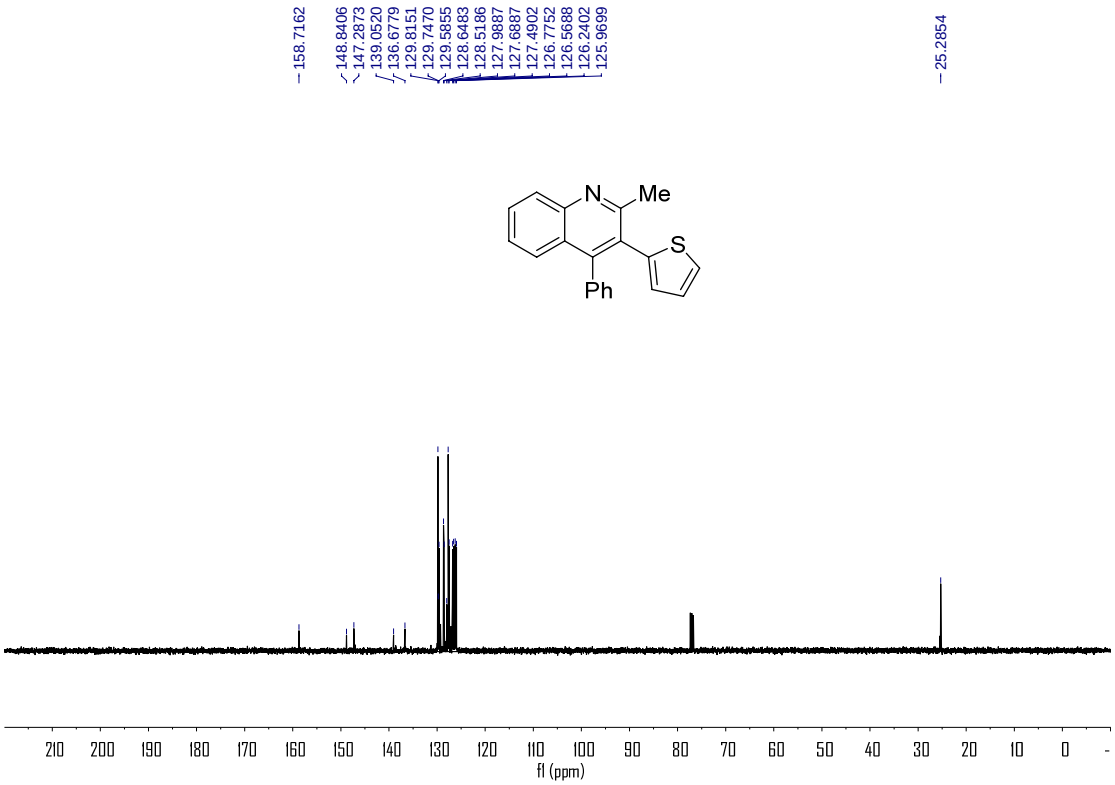
3ag

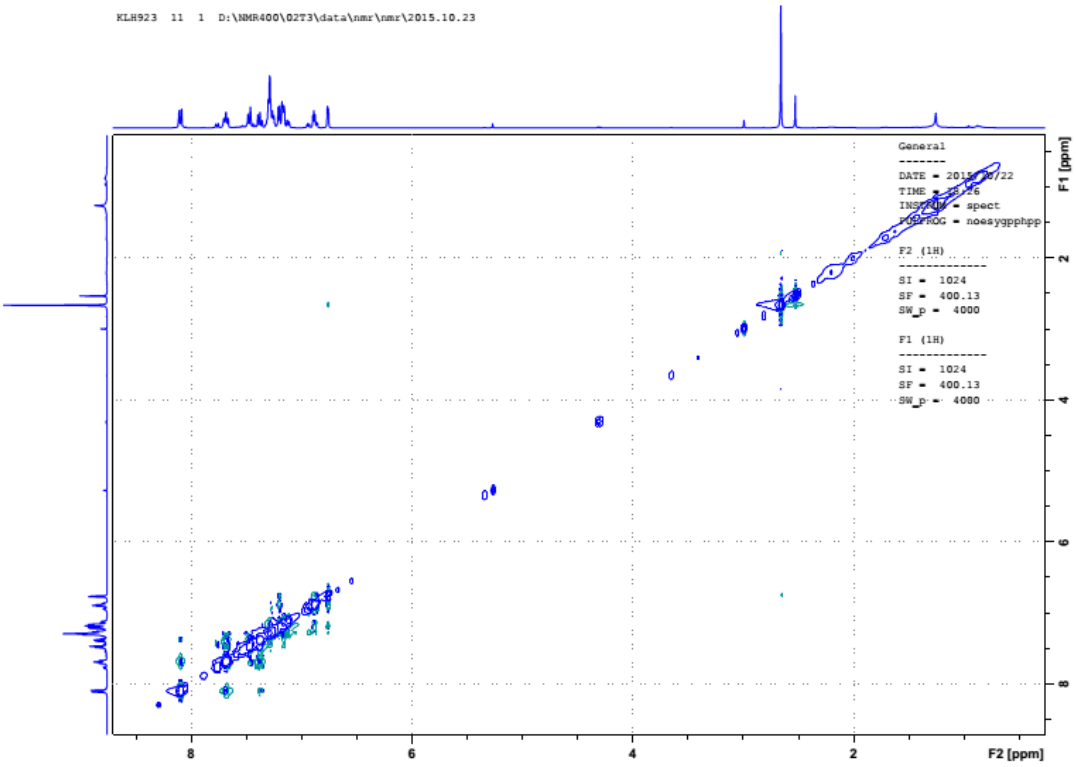

8.1012
8.0802
7.6839
7.2873
7.0247
7.0149
7.0036
6.9819
6.9698
6.9609
6.8668
6.8651
6.8586
6.8567
N
Me
S
S
1.00
1.00
1.95
0.96
2.95
f1 (ppm)


158.6240
147.3679
141.8859
138.9111
136.5666
129.8316
129.5971
128.7779
128.7353
128.2954
127.1716
126.7235
126.6486
126.5930
126.5830
126.5114
126.3586
25.4358
N
Me
S
S
f1 (ppm)

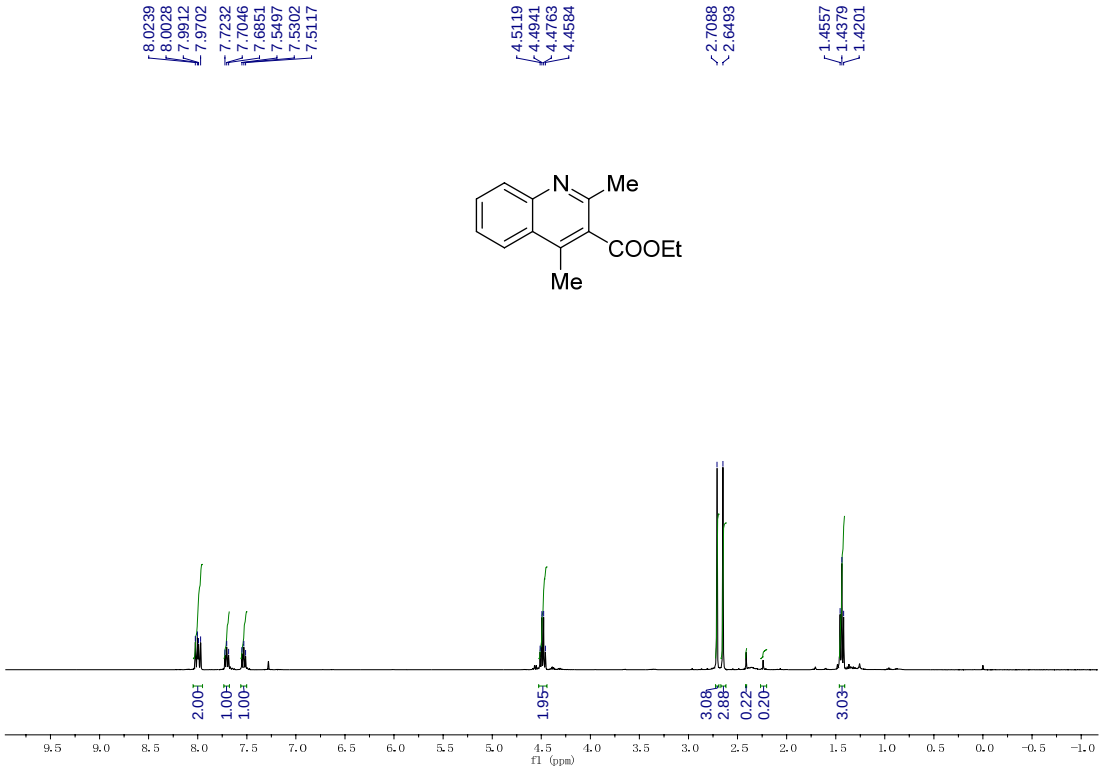
3ah

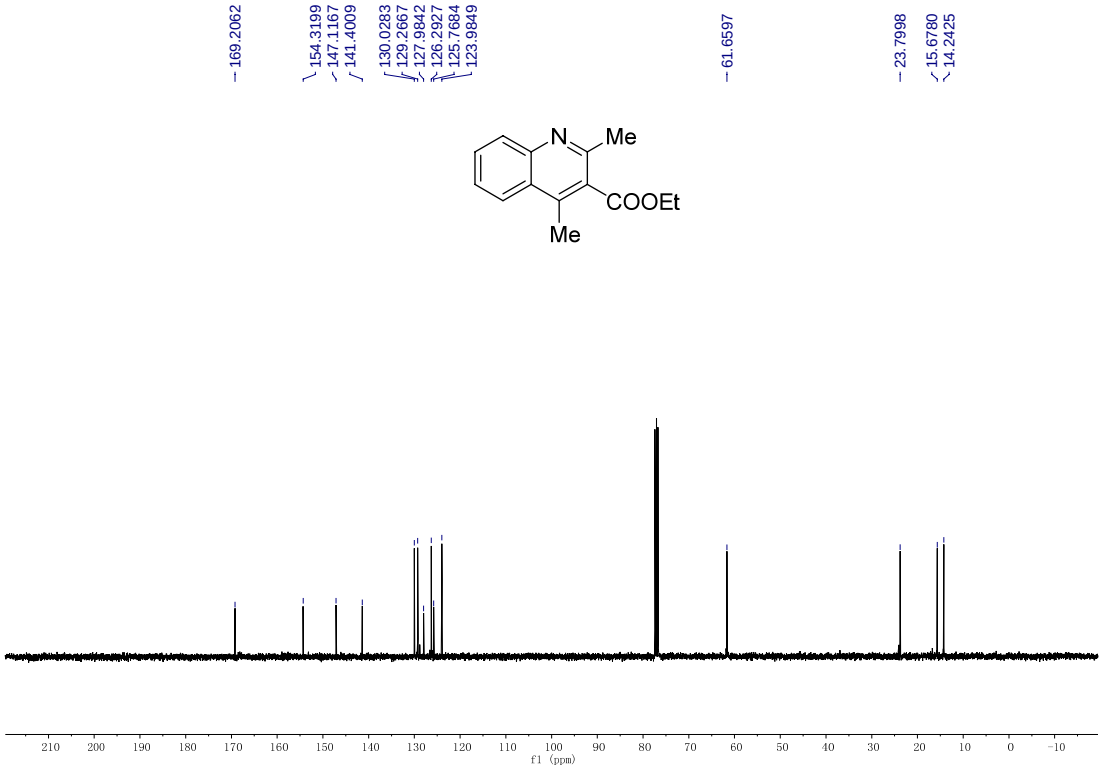

8.1935
8.1727
7.7631
7.7435
7.4348
7.2754
7.1939
6.9732
6.9661
6.9419
6.8437
2.7415
N
Me
S
Ph
1.09
1.14
1.06
3.33
1.12
0.93
2.79
0.54
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

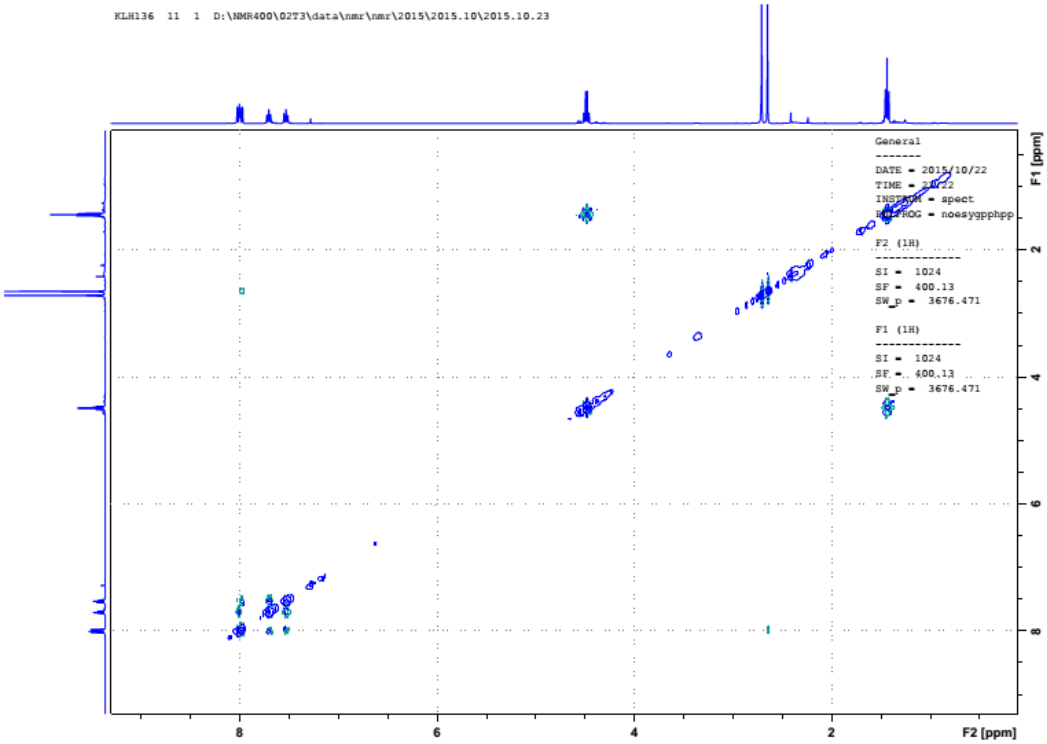

158.7162
148.8406
147.2873
139.0520
136.6779
129.8151
129.7470
129.5855
128.6483
128.5186
127.9887
127.6887
127.4902
126.7752
126.5688
126.2402
125.9699
25.2854
N
Me
S
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


KLH923 11 1 D:\NMR400\0273\data\nmr\nmr\2015.10.23
General
DATE = 2015/10/22
TIME = 15:26
INSTRUM = spect
PULPROG = noesygpphpp
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 4000
F1 (1H)
SI = 1024
SF = 400.13
SW_p = 4000
F2 [ppm]
F1 [ppm]

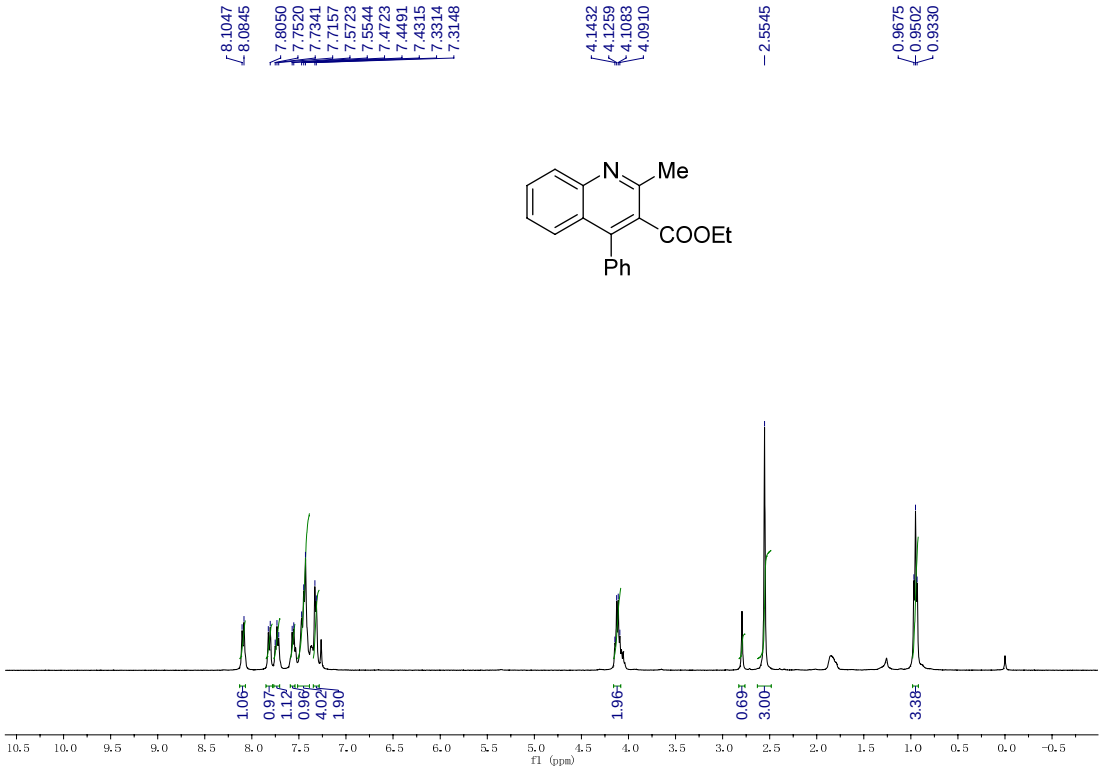
3ai

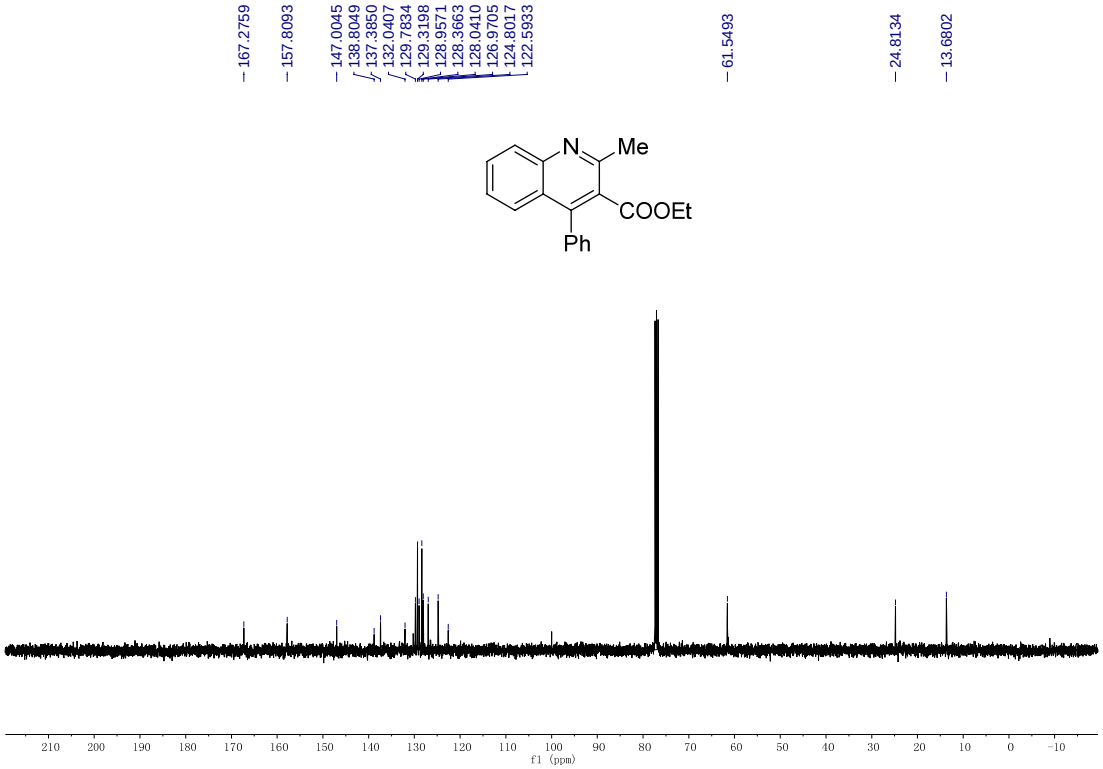

8.0239
8.0028
7.9912
7.9702
7.7232
7.7046
7.6851
7.5497
7.5302
7.5117
4.5119
4.4941
4.4763
4.4584
2.7088
2.6493
1.4557
1.4379
1.4201
N
Me
COOEt
Me
2.00
1.00
1.00
1.95
3.08
2.88
0.22
0.20
3.03
f1 (ppm)


169.2062
154.3199
147.1167
141.4009
130.0283
129.2667
127.9842
126.2927
125.7684
123.9849
61.6597
23.7998
15.6780
14.2425
N
Me
COOEt
Me
f1 (ppm)

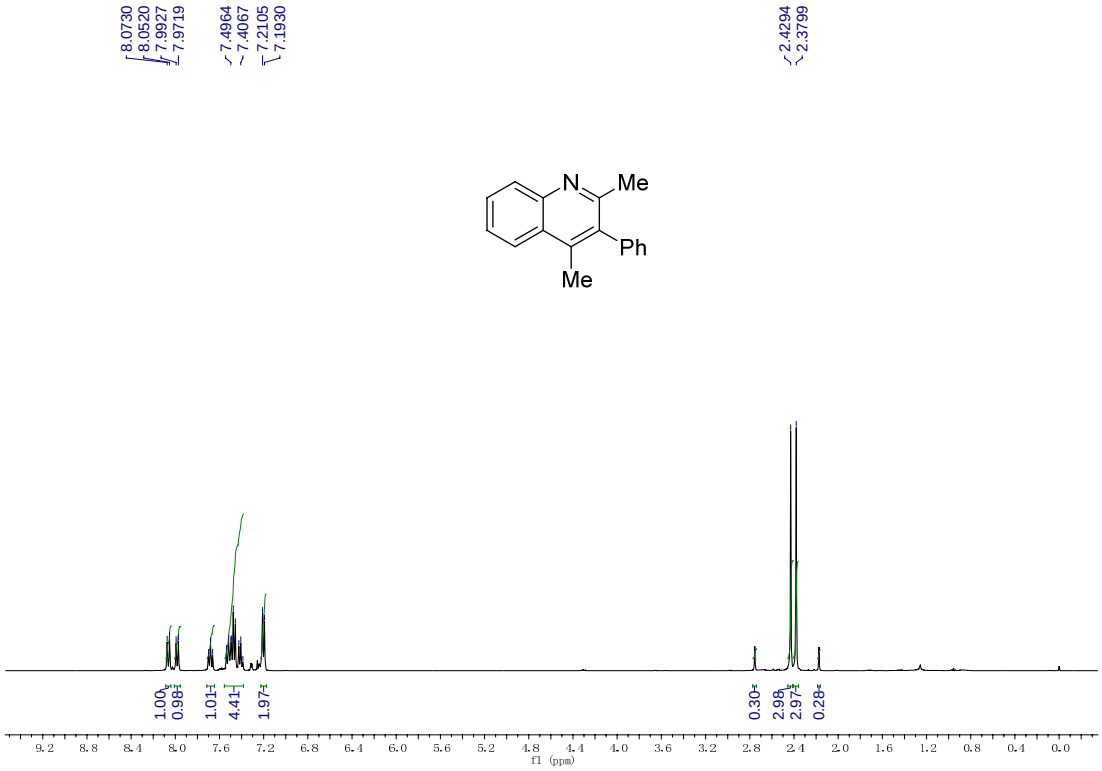

KLH136 11 1 D:\NMR400\02T3\data\nmr\nmr\2015\2015.10\2015.10.23
General
DATE = 2015/10/22
TIME = 20:22
INSTRUM = spect
PULPROG = noesygpphpp
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 3676.471
F1 (1H)
SI = 1024
SF = 400.13
SW_p = 3676.471
F1 [ppm]
F2 [ppm]
8
6
4
2

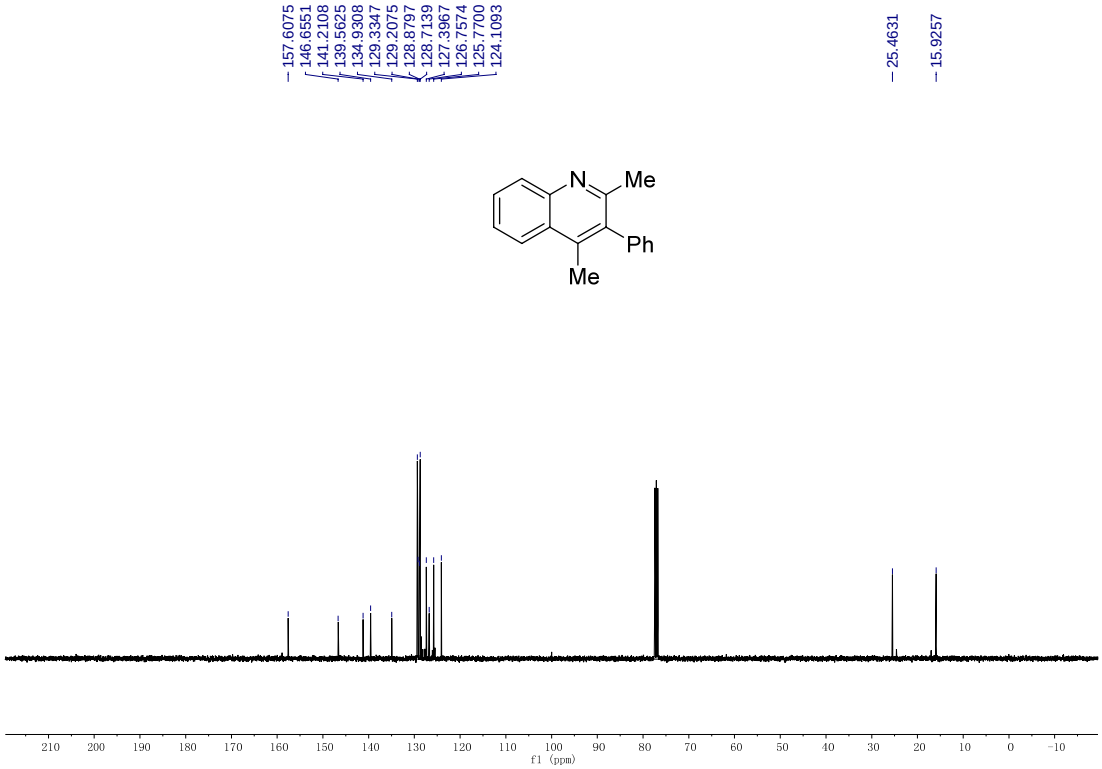
3aj

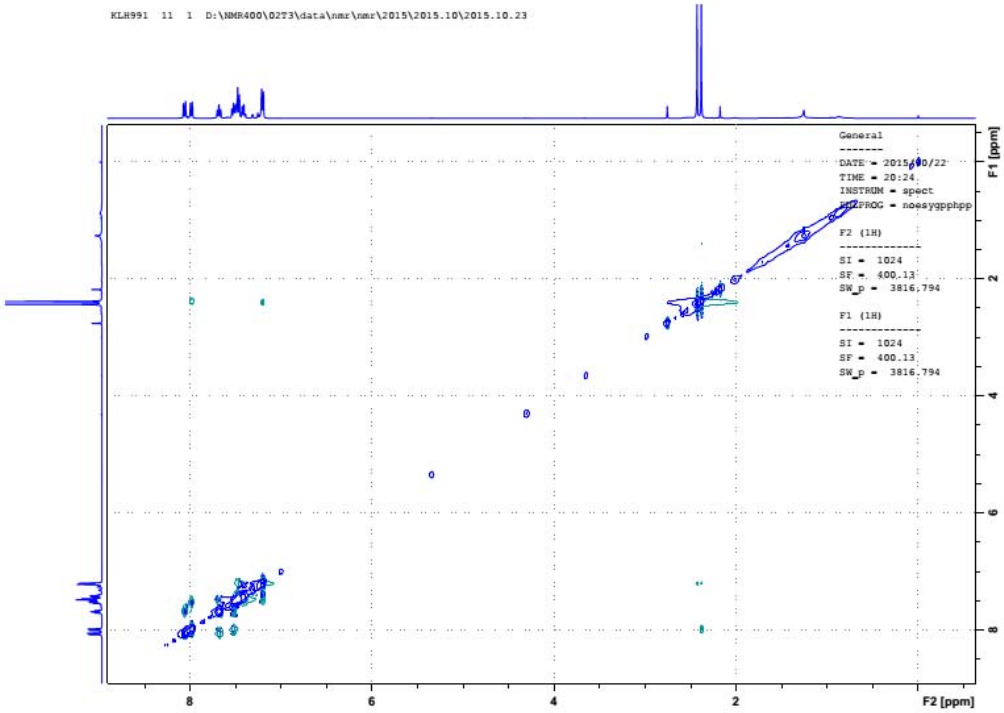

8.1047
8.0845
7.8050
7.7520
7.7341
7.7157
7.5723
7.5544
7.4723
7.4491
7.4315
7.3314
7.3148
4.1432
4.1259
4.1083
4.0910
2.5545
0.9675
0.9502
0.9330
N
Me
COOEt
Ph
1.06
0.97
1.12
0.96
4.02
1.90
1.96
0.69
3.00
3.38
f1 (ppm)


167.2759
157.8093
147.0045
138.8049
137.3850
132.0407
129.7834
129.3198
128.9571
128.3663
128.0410
126.9705
124.8017
122.5933
61.5493
24.8134
13.6802
N
Me
COOEt
Ph
f1 (ppm)

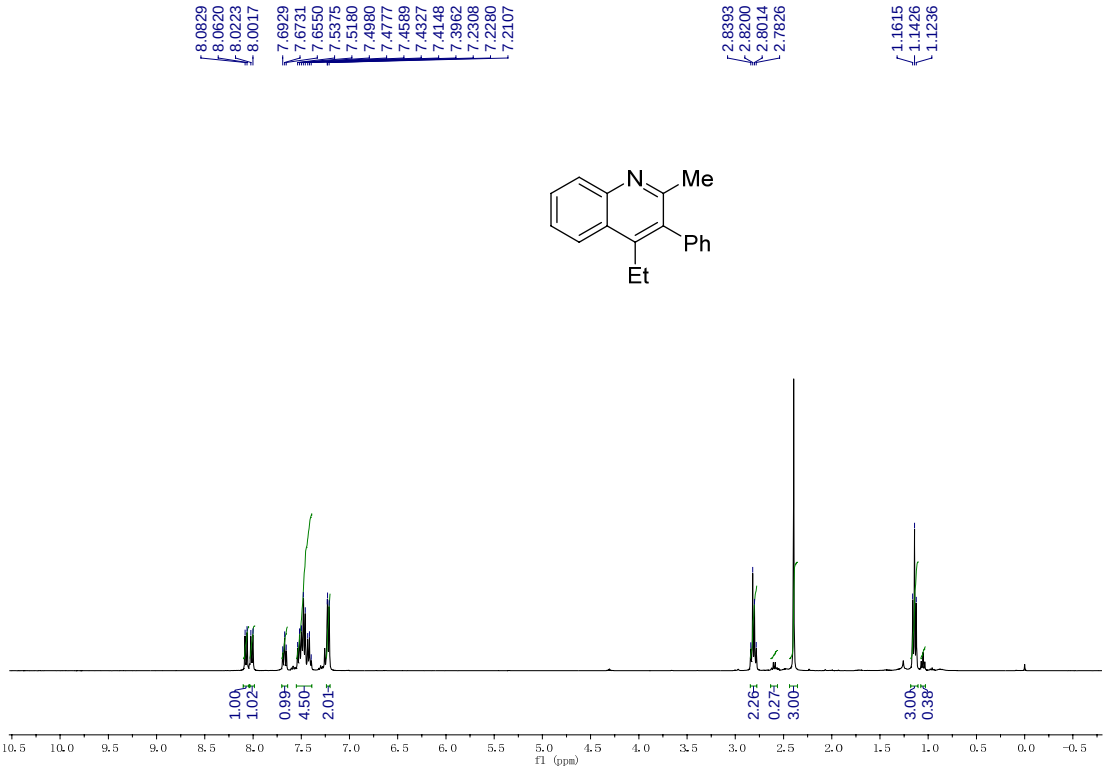
3ak

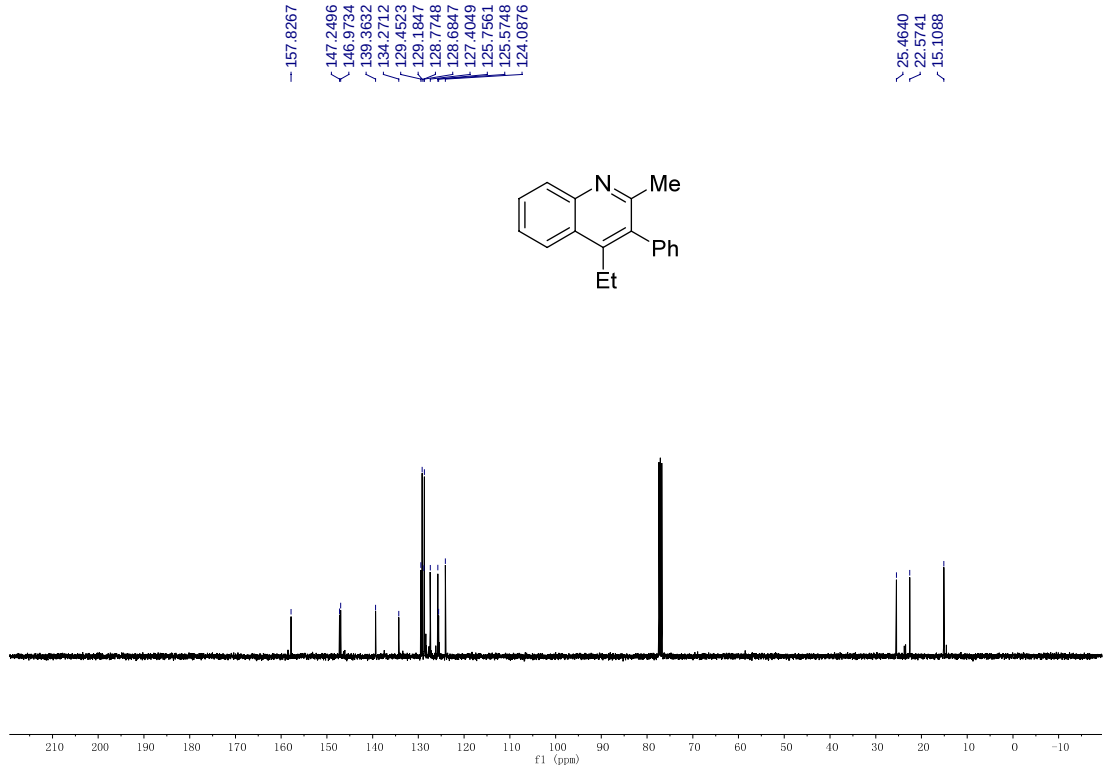

8.0730
8.0520
7.9927
7.9719
7.4964
7.4067
7.2105
7.1930
2.4294
2.3799
N
Me
Ph
Me
1.00
0.98
1.01
4.41
1.97
0.30
2.98
2.97
0.28
f1 (ppm)


157.6075
146.6551
141.2108
139.5625
134.9308
129.3347
129.2075
128.8797
128.7139
127.3967
126.7574
125.7700
124.1093
25.4631
15.9257
N
Me
Ph
Me
f1 (ppm)


KLH991 11 1 D:\NMR400\02T3\data\nmr\nmr\2015\2015.10\2015.10.23
General
DATE = 2015/10/22
TIME = 20:24
INSTRUM = spect
PULPROG = noesygpphpp
F2 (1H)
SI = 1024
SF = 400.13
SW_p = 3816.794
F1 (1H)
SI = 1024
SF = 400.13
SW_p = 3816.794
F1 [ppm]
F2 [ppm]

3al


8.0829
8.0620
8.0223
8.0017
7.6929
7.6731
7.6550
7.5375
7.5180
7.4980
7.4777
7.4589
7.4327
7.4148
7.3962
7.2308
7.2280
7.2107
2.8393
2.8200
2.8014
2.7826
1.1615
1.1426
1.1236
N
Me
Ph
Et
1.00
1.02
0.99
4.50
2.01
2.26
0.27
3.00
3.00
0.38
f1 (ppm)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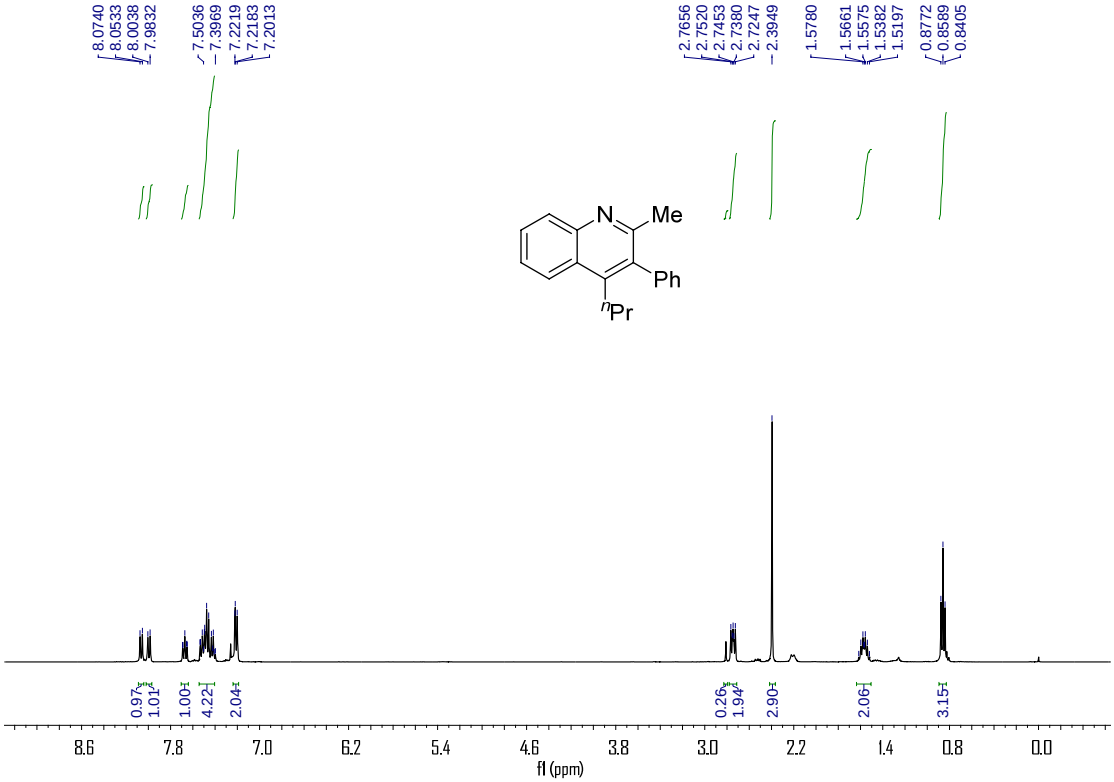
147.2496
146.9734
139.3632
134.2712
129.4523
129.1847
128.7748
128.6847
127.4049
125.7561
125.5748
124.0876
25.4640
22.5741
15.1088
N
Me
Ph
Et
f1 (ppm)

3am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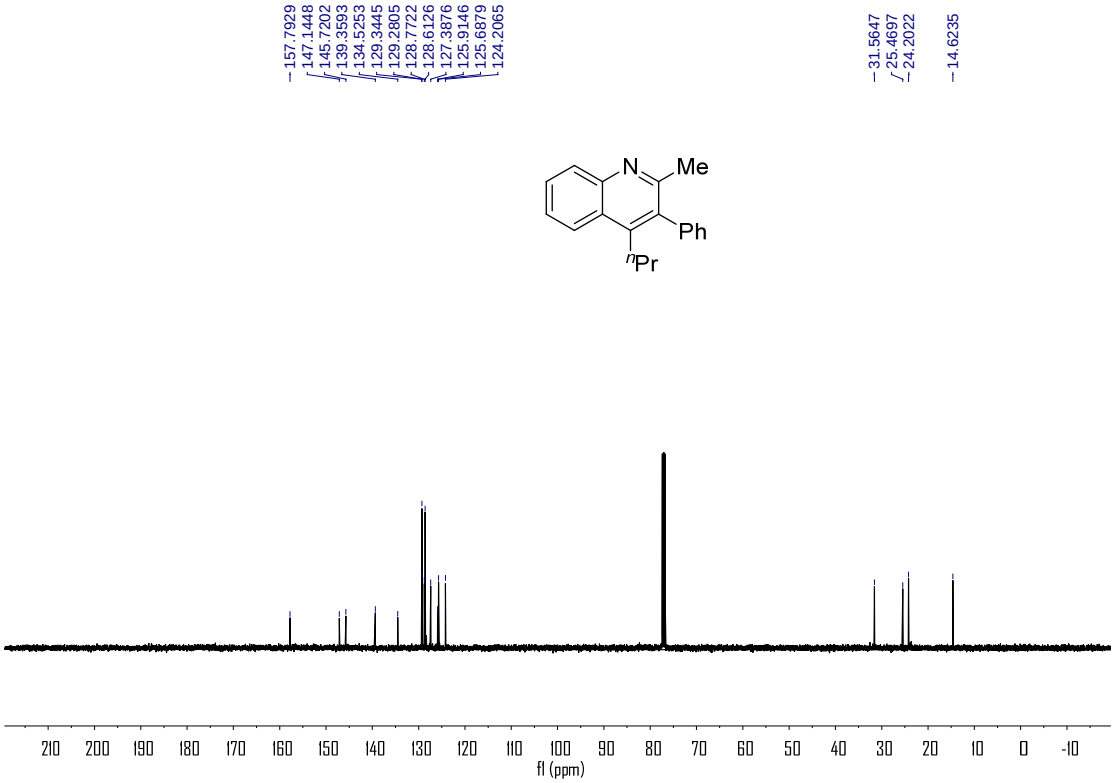
8.0533
8.0038
7.9832
7.5036
7.3969
7.2219
7.2183
7.2013
2.7656
2.7520
2.7453
2.7380
2.7247
2.3949
1.5780
1.5661
1.5575
1.5382
1.5197
0.8772
0.8589
0.8405
N
Me
Ph
nPr
0.97
1.01
1.00
4.22
2.04
0.26
1.94
2.90
2.06
3.15
8.6
7.8
7.0
6.2
5.4
4.6
3.8
3.0
2.2
1.4
0.8
0.0
f1 (ppm)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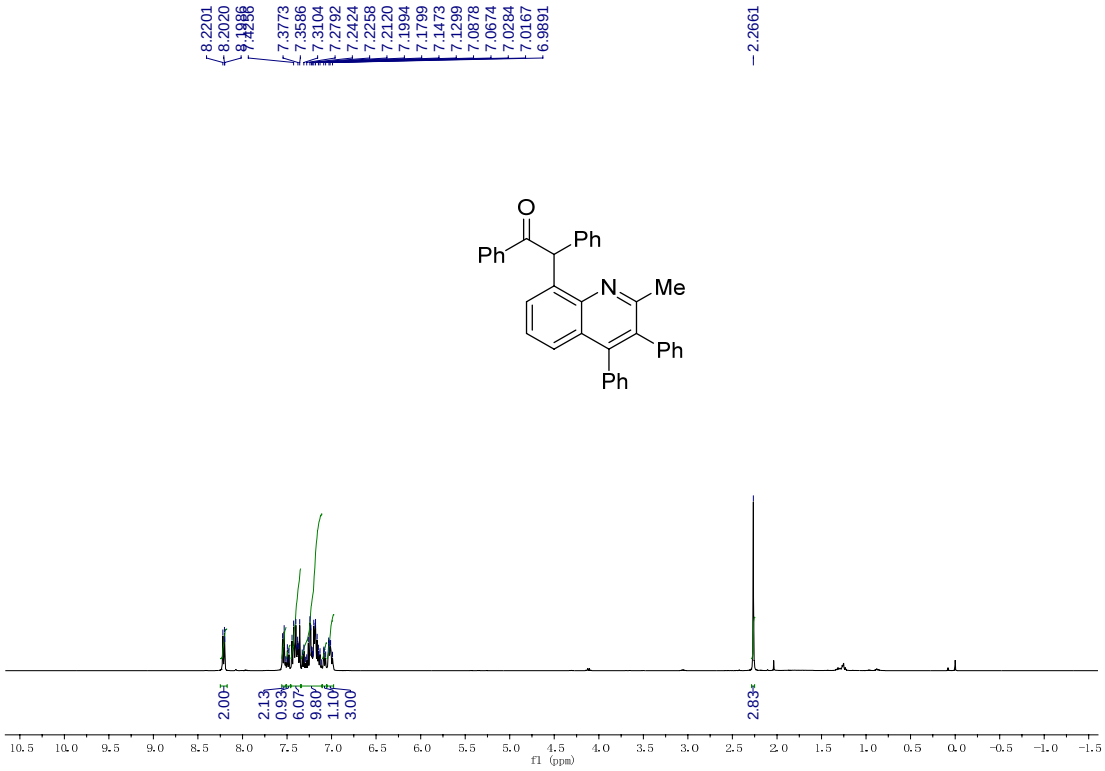
147.1448
145.7202
139.3593
134.5253
129.3445
129.2805
128.7722
128.6126
127.3876
125.9146
125.6879
124.2065
31.5647
25.4697
24.2022
14.6235
N
Me
Ph
nPr
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

4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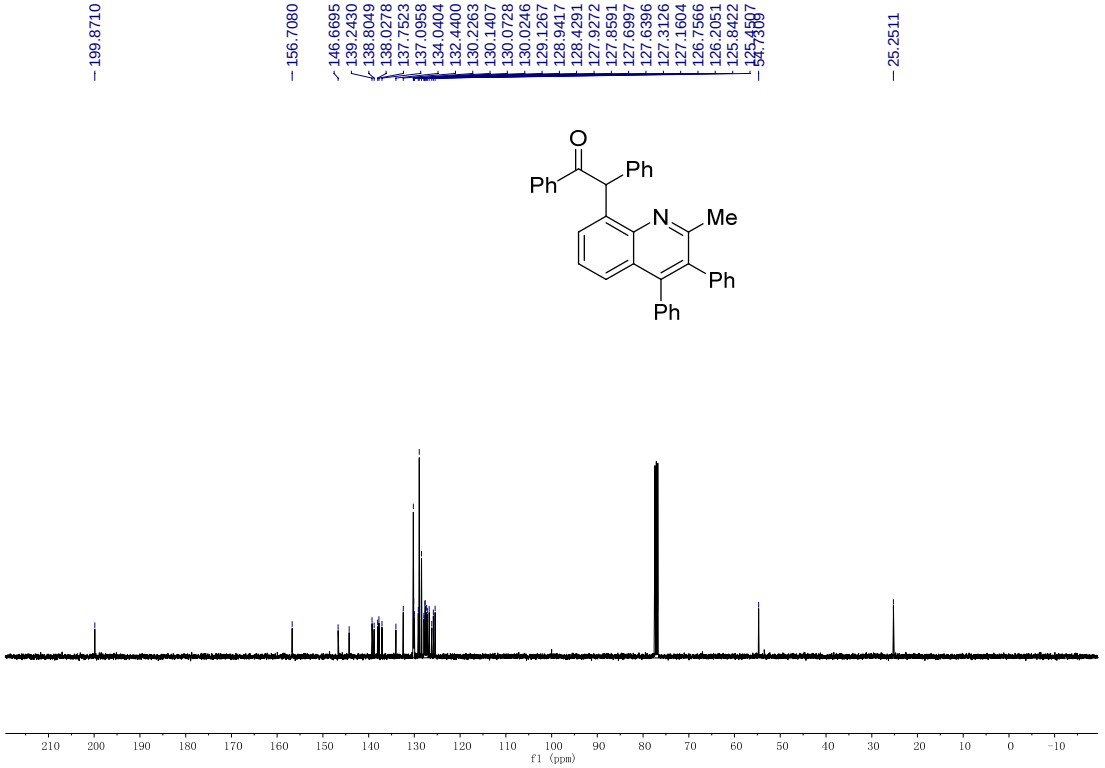
8.2020
7.3773
7.3586
7.3104
7.2792
7.2424
7.2258
7.2120
7.1994
7.1799
7.1473
7.1299
7.0878
7.0674
7.0284
7.0167
6.9891
2.2661
O
Ph
Ph
N
Me
Ph
Ph
2.00
2.13
0.93
6.07
9.80
1.10
3.00
2.83
f1 (ppm)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156.7080
146.6695
139.2430
138.8049
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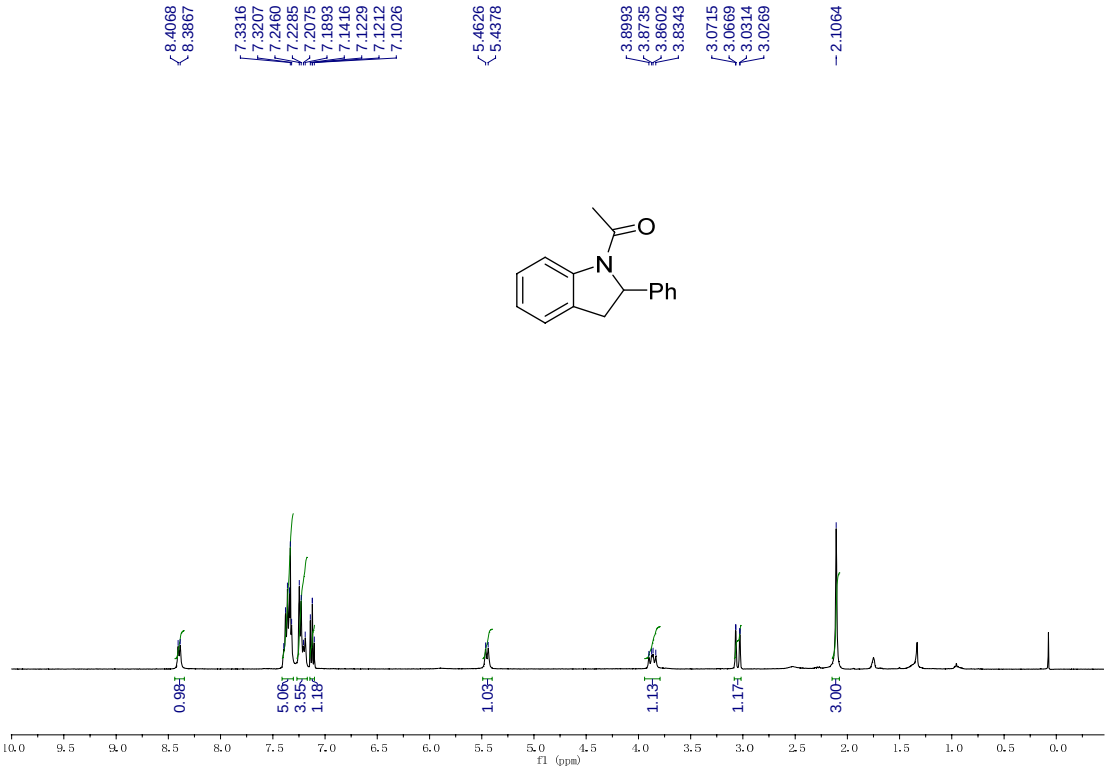
137.7523
137.0958
134.0404
132.4400
130.2263
130.1407
130.0728
130.0246
129.1267
128.9417
128.4291
127.9272
127.8591
127.6997
127.6396
127.3126
127.1604
126.7566
126.2051
125.8422
125.4507
54.7309
25.2511
O
Ph
Ph
N
Me
Ph
Ph
f1 (ppm)

6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8.3867
7.3316
7.3207
7.2460
7.2285
7.2075
7.1893
7.1416
7.1229
7.1212
7.1026
5.4626
5.4378
3.8993
3.8735
3.8602
3.8343
3.0715
3.0669
3.0314
3.0269
2.1064
O
N
Ph
0.98
5.06
3.55
1.18
1.03
1.13
1.17
3.00
f1 (ppm)