Supporting Information

Copper-Catalysed Aerobic Oxidative Esterification of *N*-Heteroaryl Methanes with Alcohols

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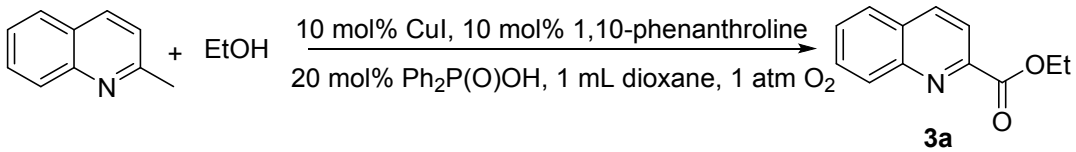
General information

All reactions were carried out in oven-dried Schlenk tubes under O_2 atmosphere. Dry solvents were obtained by purification according to standard methods. Reagents were used as received unless otherwise noted. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR data were obtained on a Bruker-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C, and 162 MHz for ^{31}P NMR spectroscopy). Data are reported as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q= quartet, m = multiplet), Coupling constants (J) are reported in hertz. Mass spectra were measured on a Shimadzu GCMS-QP2010 Plus spectrometer (EI).

A typical procedure for the synthesis of esters

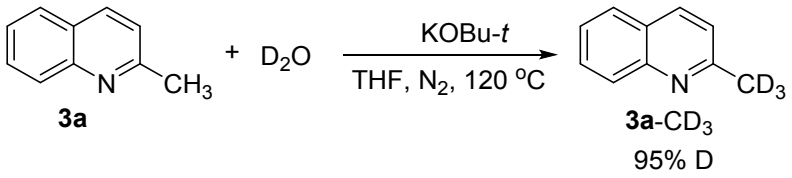


3a

Under O_2 atmosphere, 0.2 mmol 2-methylquinoline, 1mL EtOH, 10 mol% CuI and 10 mol% 1,10-phenanthroline, 20 mol% $Ph_2P(O)OH$, 1 mL dioxane were charged into a 10 mL schlenk tube, and then the mixture was stirred at 120 ^{o}C for 24 h. After removal of the volatiles, the residues were passed through a short silica chromatography (particle size 37–54 μm, petroleum ether/ethyl acetate as eluent) to

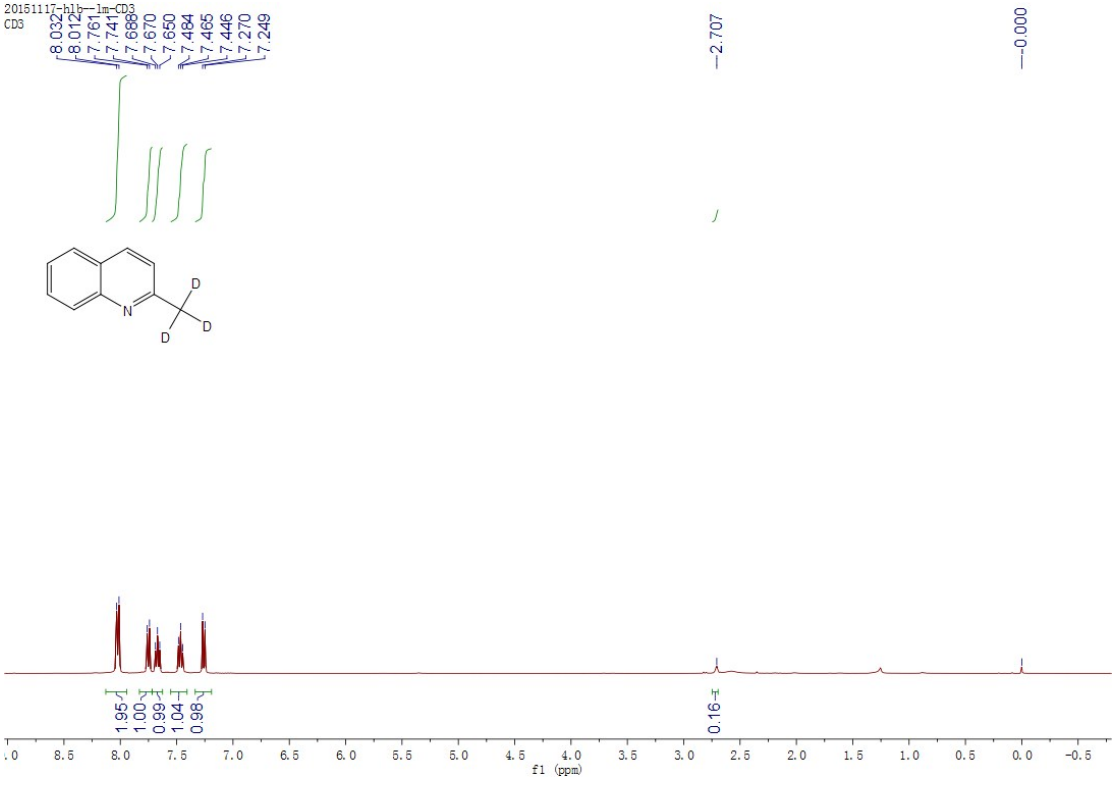
afford analytically pure product **3a**.

Preparation of 2-CD_3-quinolines

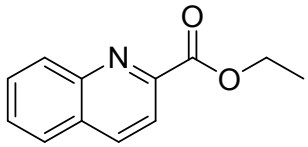


3a **3a**-CD_3 95% D

Under N_2 atmosphere, 4 mmol KOBu-t, 1 mmol 2-methylquinoline, 3 mL D_2O and 0.3 mL THF were charged into a 25 mL glass tube, the mixture was heated at 120 °C for 16 h. The solution was extracted by ethyl acetate (10 mL), dried by Na_2SO_4 and evaporated under high vacuum to give an oil mixture. Redeuteration of the resulting oil mixture produced the 2-CD_3-quinoline in 68% yield with ca. 95% D.

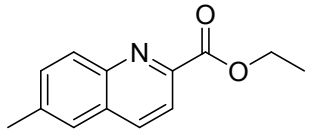


Characterization data of product 3



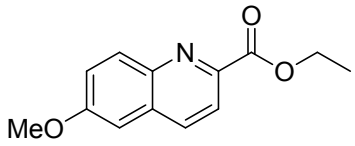
3a[1]

Following the general procedure, **3a** was isolated as a yellow liquid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.31 (d, J = 8.8 Hz, 1H), 8.29 (d, J = 8.8 Hz, 1H), 8.18 (d, J = 8.4 Hz, 1H), 7.86 (d, J = 8.4 Hz, 1H), 7.78 (t, J = 8.0 Hz, 1H), 7.63 (t, J = 8.0 Hz, 1H); 4.56 (q, J = 7.2 Hz, 2H); 1.49 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.4, 148.2, 147.6, 137.2, 130.7, 130.2, 129.3, 128.5, 127.5, 121.0, 62.2, 14.4.



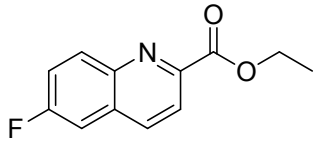
3b[1]

Following the general procedure, **3b** was isolated as a white solid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.14-8.22 (m, 3H), 7.63 (s, 1H), 7.62 (d, J = 8.4 Hz, 1H), 4.56 (q, J = 7.2 Hz, 2H), 2.57 (s, 3H), 1.50 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.5, 147.4, 146.2, 138.9, 136.4, 132.6, 130.4, 129.4, 126.3, 121.1, 62.1, 21.8, 14.4.



3c[1]

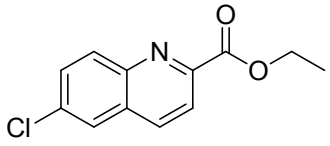
Following the general procedure, **3c** was isolated as a yellow solid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.21 (d, J = 9.2 Hz, 1H), 8.17 (s, 2H), 7.44 (dd, J = 2.8 Hz, 9.2 Hz, 1H), 7.11 (d, J = 2.8 Hz, 1H), 4.56 (q, J = 7.2 Hz, 2H), 3.97 (s, 3H); 1.50 (t, J = 7.2 Hz, 3H) ^{13}C NMR ($CDCl_3$, 100 MHz, TMS) δ 165.6, 159.4, 145.8, 143.8, 135.6, 132.3, 130.8, 123.4, 121.5, 104.6, 62.1, 55.6, 14.4.



3d[2]

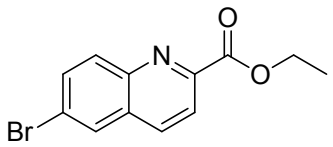
Following the general procedure, **3d** was isolated as a yellow solid.^{1}H NMR ($CDCl_3$, 400 MHz, TMS) δ 8.32 (dd, J = 5.6 Hz, 9.6 Hz, 1H), 8.22 (dd, J = 8.4 Hz, 20.0 Hz, 1H), 8.22 (d, J = 11.6 Hz, 1H), 7.56 (ddd, J = 2.4 Hz, 8.8 Hz, 11.2 Hz, 1H), 7.49 (dd, J = 2.4 Hz, 8.8 Hz, 1H), 4.57 (q, J = 7.2 Hz, 2H), 1.50 (t, J = 7.2 Hz, 3H); ^{13}C NMR

($CDCl_3$, 100 MHz, TMS) δ 165.2, 162.8 (d, J_{F-C} = 250.5 Hz), 147.7 (d, J_{F-C} = 2.9 Hz), 144.7, 136.5 (d, J_{F-C} = 5.7 Hz), 133.5 (d, J_{F-C}= 9.5 Hz), 130.1 (d, J_{F-C} = 10.4 Hz), 121.7, 120.8 (d, J_{F-C} = 25.9 Hz), 110.5 (d, J_{F-C} = 21.8 Hz), 62.3, 14.3.



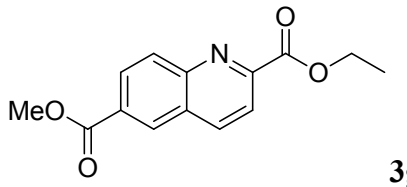
3e[2]

Following the general procedure, **3h** was isolated as a yellow solid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.25 (d, J = 8.8 Hz, 1H), 8.21 (s, 2H), 7.86 (d, J = 2.4 Hz, 1H), 7.72 (dd, J = 2.4 Hz, 8.8 Hz, 1H), 4.56 (q, J = 7.2 Hz, 2H), 1.50 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.1, 148.5, 146.0, 136.3, 134.6, 132.3, 131.3, 129.8, 126.2, 121.9, 62.4, 14.4.



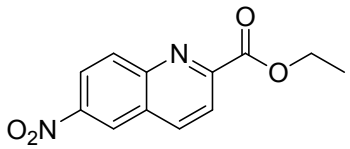
3f[2]

Following the general procedure, **3f** was isolated as a yellow solid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.19 (s, 2H), 8.17 (d, J = 9.2 Hz, 1H), 8.03 (d, J = 2.0 Hz, 1H), 7.84 (dd, J = 2.0 Hz, 9.2 Hz, 1H), 4.56 (q, J = 7.2 Hz, 2H), 1.49 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS) δ 165.1, 148.5, 146.1, 136.2, 133.8, 132.3, 130.2, 129.6, 122.9, 121.9, 62.4, 14.3.



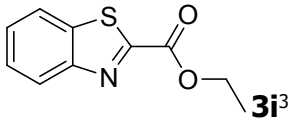
3g

Following the general procedure, **3g** was isolated as a white solid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.63 (s, 1H), 8.41 (d, J = 8.8 Hz, 1H), 8.36 (s, 2H), 8.24 (d, J = 8.4 Hz, 1H), 4.58 (q, J = 7.2 Hz, 2H), 4.01 (s, 3H), 1.51 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.2, 165.0, 150.2, 149.3, 138.6, 131.0, 130.5, 129.8, 129.6, 128.4, 121.7, 62.5, 52.6, 14.3. HRMS Calcd. for $C_{14}H_{13}O_4$ (M+) 259.0845, found 259.0841, M.P. 96.1-98.3 ^{o}C.



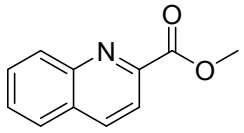
3h

Following the general procedure, **3h** was isolated as a yellow solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.86 (d, J = 2.0 Hz, 1H), 8.54 (d, J = 8.0 Hz, 2H), 8.46 (d, J = 9.6 Hz, 1H), 8.34 (d, J = 8.8 Hz, 1H), 4.60 (q, J = 7.2 Hz, 2H), 1.52 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 164.6, 151.4, 149.4, 146.7, 139.3, 132.6, 128.0, 124.2, 123.6, 122.7, 62.8, 14.3. HMS:246.0623, bp: 163.0-164.2



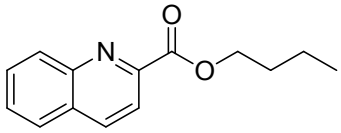
3i[3]

Following the general procedure, **3i** was isolated as a white solid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.27 (d, J = 7.6 Hz, 1H), 7.99 (d, J = 7.6 Hz, 1H), 7.54-7.62 (m, 2H), 4.57 (q, J = 7.2 Hz, 2H), 1.51 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.7, 158.6, 153.2, 136.8, 127.5, 127.1, 125.5, 122.1, 63.1, 14.3.



3j[4]

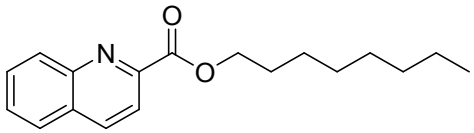
Following the general procedure, **3j** was isolated as a yellow solid. ^{1}H NMR ($CDCl_3$, 400 MHz, TMS) δ 8.28-8.32 (m, 2H), 8.20 (d, J = 8.4 Hz, 1H), 7.87 (d, J = 8.4 Hz, 1H), 7.79 (t, J = 7.6 Hz, 1H), 7.65 (t, J = 7.6 Hz, 1H), 4.09 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS) δ 165.9, 147.9, 147.5, 137.3, 130.7, 130.3, 129.3, 128.6, 127.6, 121.0, 53.2.



3k[6]

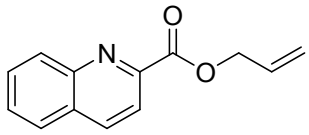
Following the general procedure, **3k** was isolated as a yellow liquid.^{1}H NMR($CDCl_3$, 400 MHz) δ 8.32 (d, J = 8.4 Hz, 1H), 8.28 (d, J = 8.4 Hz, 1H), 8.16 (d, J = 8.4 Hz, 1H), 7.86 (d, J = 8.4 Hz, 1H), 7.77 (dd, J = 8.4 Hz, 7.2 Hz, 1H), 7.63 (dd, J = 8.0 Hz, 6.8 Hz, 1H), 4.50 (t, J = 6.8 Hz, 2H), 1.82-1.89 (m, 2H), 1.46-1.55 (m, 2H), 1.00 (t, J = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.4, 148.3, 147.6, 137.2, 130.8,

130.2, 129.2, 128.5, 127.5, 121.0, 66.0, 30.7, 19.2, 13.8.



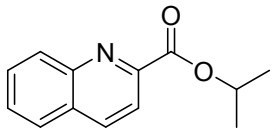
3l

Following the general procedure, **3l** was isolated as a yellow liquid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.32 (d, J = 8.4 Hz, 1H), 8.30 (d, J = 8.4 Hz, 1H), 8.18 (d, J = 8.8 Hz, 1H), 7.88 (d, J = 8.0 Hz, 1H), 7.79 (dd, J = 7.6 Hz, 7.6 Hz, 1H), 7.65 (t, J = 7.2 Hz, 7.6 Hz, 1H), 4.49 (t, J = 6.8 Hz, 2H), 1.84-1.91 (m, 2H), 1.29-1.50 (m, 10H), 0.88 (t, J = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.5, 148.3, 147.7, 137.2, 130.9, 130.2, 129.3, 128.5, 127.5, 121.0, 66.4, 31.8, 29.3, 29.2, 28.7, 25.9, 22.6, 14.1. HRMS Calcd. for $C_{18}H_{23}O_2P$ (M+) 285.1729, found 285.1722



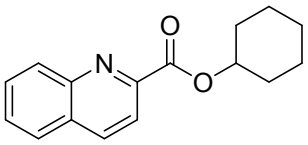
3m[7]

Following the general procedure (9 equiv alcohol, 25 mL glass tube), **3m** was isolated as a yellow liquid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.30-8.33 (m, 2H), 8.20 (d, J = 8.8 Hz, 1H), 7.88 (d, J = 8.4 Hz, 1H), 7.79 (dd, J = 8.4 Hz, 7.2 Hz, 1H), 7.65 (dd, J = 7.2 Hz, 7.6 Hz, 1H), 5.49 (dd, J = 1.2 Hz, 17.2 Hz, 1H), 5.35 (dd, J = 0.8 Hz, 10.4 Hz, 1H), 5.00 (d, J = 6.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.1, 148.0, 147.6, 137.3, 131.8, 130.8, 130.3, 129.3, 128.6, 127.5, 121.1, 119.2, 66.8.



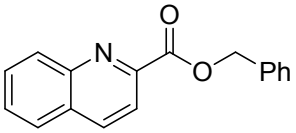
3n[5]

Following the general procedure, **3n** was isolated as a yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, J = 8.8 Hz, 1H), 8.28 (d, J = 8.8 Hz, 1H), 8.15 (d, J = 8.4 Hz, 1H), 7.86 (d, J = 8.4 Hz, 1H), 7.77 (dd, J = 8.4 Hz, 7.2 Hz, 1H), 7.62 (dd, J = 8.0 Hz, 6.8 Hz. 1H), 5.35-5.45 (m, 1H), 1.47 (d, J = 6.4 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 164.8, 148.6, 147.7, 137.1, 130.9, 130.1, 129.2, 128.4, 127.4, 121.0, 69.9, 21.9.



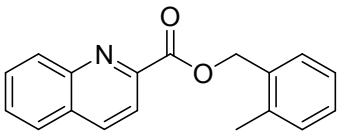
3o

Following the general procedure (9 equiv alcohol, 25 mL glass tube), **3o** was isolated as a yellow liquid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.19-8.25 (m, 2H), 8.07 (d, J = 8.4 Hz, 1H), 7.79 (d, J = 8.4 Hz, 1H), 7.69 (dd, J = 7.6 Hz, 7.6 Hz, 1H), 7.55 (dd, J = 7.6 Hz, 7.6 Hz, 1H), 5.05-5.11 (m, 1H), 1.18-2.01 (m, 10H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 164.6, 148.7, 147.7, 137.1, 130.9, 130.1, 129.2, 128.4, 127.4, 121.0, 74.7, 31.6, 25.4, 23.9; HMS: HRMS Calcd. for $C_{16}H_{17}NO_2$ (M+) 255.1259, found 255.1262



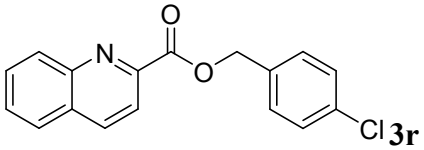
3p

Following the general procedure (9 equiv alcohol, 25 mL glass tube), **3p** was isolated as a yellow liquid. ^{1}H NMR($CDCl_3$, 400 MHz, TMS) δ 8.29-8.32 (m, 2H), 8.16 (d, J = 8.8 Hz, 1H), 7.85 (d, J = 8 Hz, 1H), 7.77 (t, J = 7.6 Hz, 1H), 7.63 (t, J = 7.4 Hz, 1H), 7.52 (d, J = 7.2 Hz, 2H), 7.34-7.41 (m, 3H), 5.53 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.2, 147.9, 147.6, 137.2, 135.7, 130.8, 130.2, 129.3, 128.6, 128.5, 128.4, 127.5, 121.1, 67.7. HRMS Calcd. for $C_{17}H_{13}NO_2$ (M+) 263.0946, found 263.0937



3q

Following the general procedure (9 equiv alcohol, 25 mL glass tube), **3q** was isolated as a yellow liquid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.26-8.32 (m, 2H), 8.15 (d, J = 8.4 Hz, 1H), 7.87 (d, J = 8.0 Hz, 1H), 7.78 (dd, J = 8.0 Hz, 8.4 Hz, 1H), 7.64 (dd, J = 7.2 Hz, 7.6 Hz, 1H), 7.50 (d, J = 8.8 Hz, 1H), 7.22-7.29 (m, 3H), 5.54 (s, 2H), 2.46 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.1, 148.0, 147.7, 137.2, 137.1, 133.7, 130.9, 130.4, 130.3, 129.5, 129.3, 128.7, 128.6, 127.5, 126.1, 121.0, 66.1, 19.1; HRMS Calcd. for $C_{18}H_{18}NO_2$ (M+) 277.1103, found 277.1105.



3r

Following the general procedure (9 equiv alcohol, 25 mL glass tube), **3r** was isolated as a white liquid. ^{1}H NMR($CDCl_3$, 400 MHz) δ 8.29-8.33 (m, 2H), 8.17 (d, J = 8.4 Hz, 1H), 7.88 (d, J = 8.0 Hz, 1H), 7.80 (dd, J = 7.2 Hz, 8.0 Hz, 1H), 7.66 (dd, J = 7.6 Hz, 7.6 Hz, 1H), 7.47 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 5.50 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.2, 147.8, 147.7, 137.3, 134.4, 134.2, 130.8, 130.3, 130.0, 129.4, 128.8, 128.7, 127.5, 121.1, 66.9; HRMS Calcd. for $C_{17}H_{12}NO_2$ (M+) 297.0557, found 297.0561

Reference

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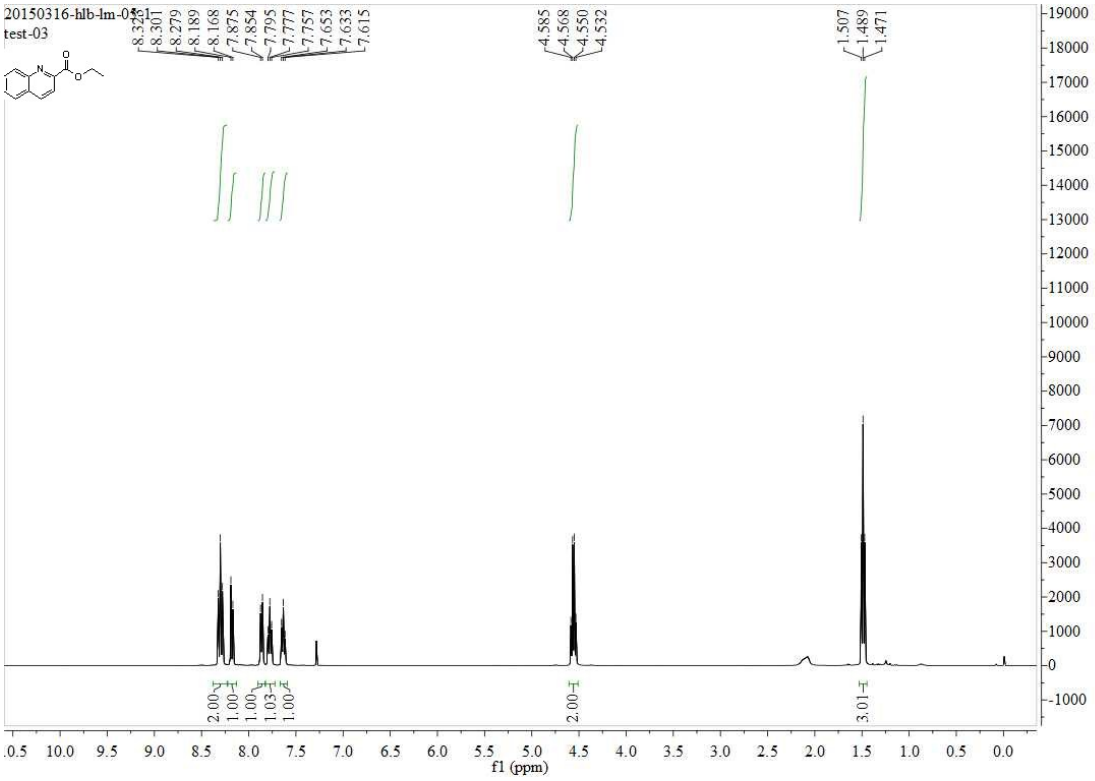
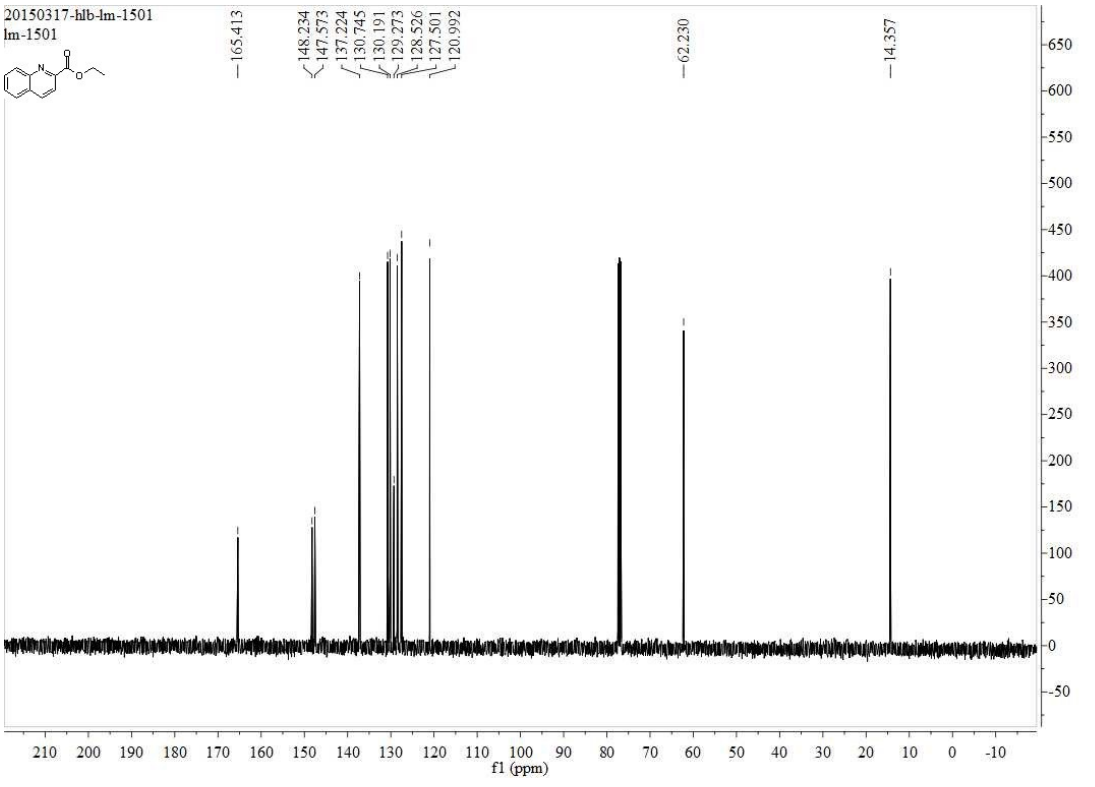
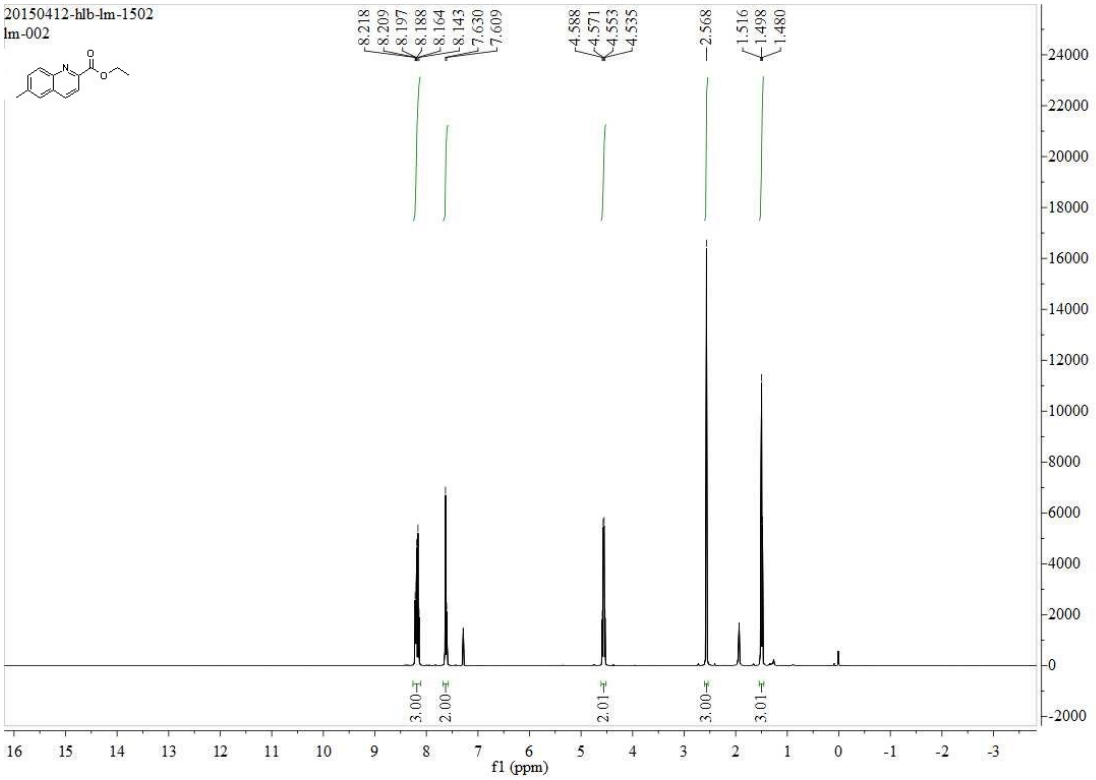
(7) T. Völker, F.Dempwolff, P. L. Graumann and E. Meggers, *Angew. Chem. Int. Ed.*, 2014, **53**, 10536

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



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2.00
2.01
3.00
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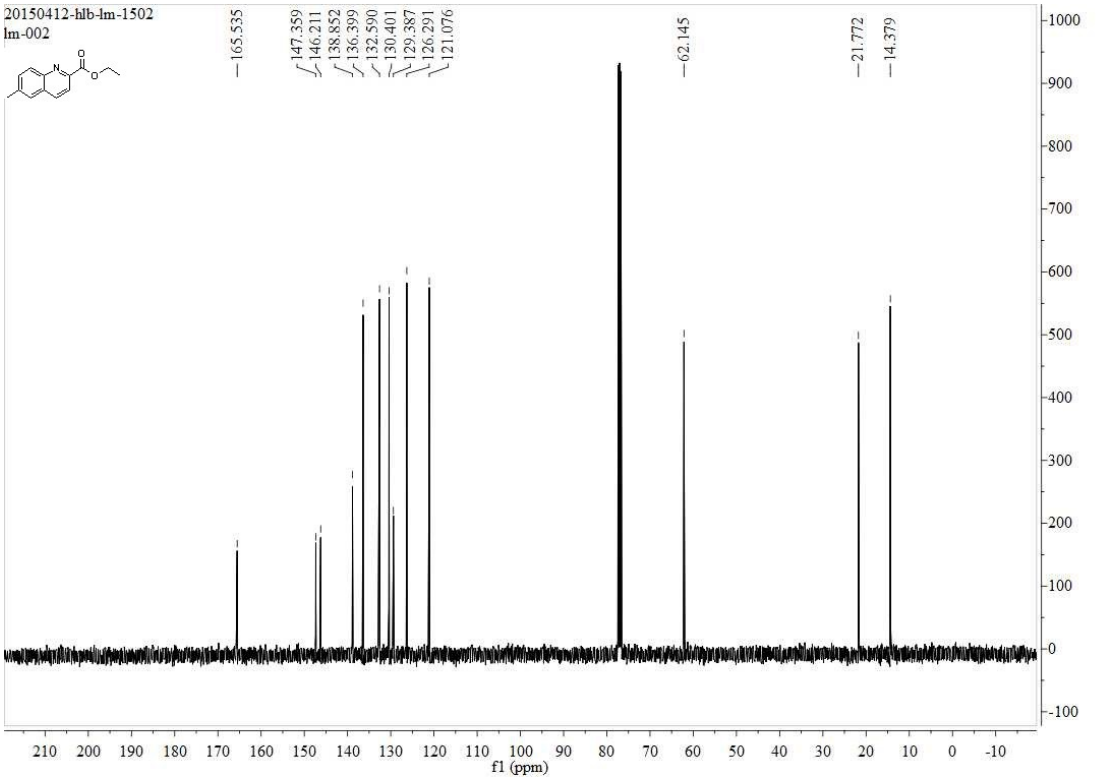
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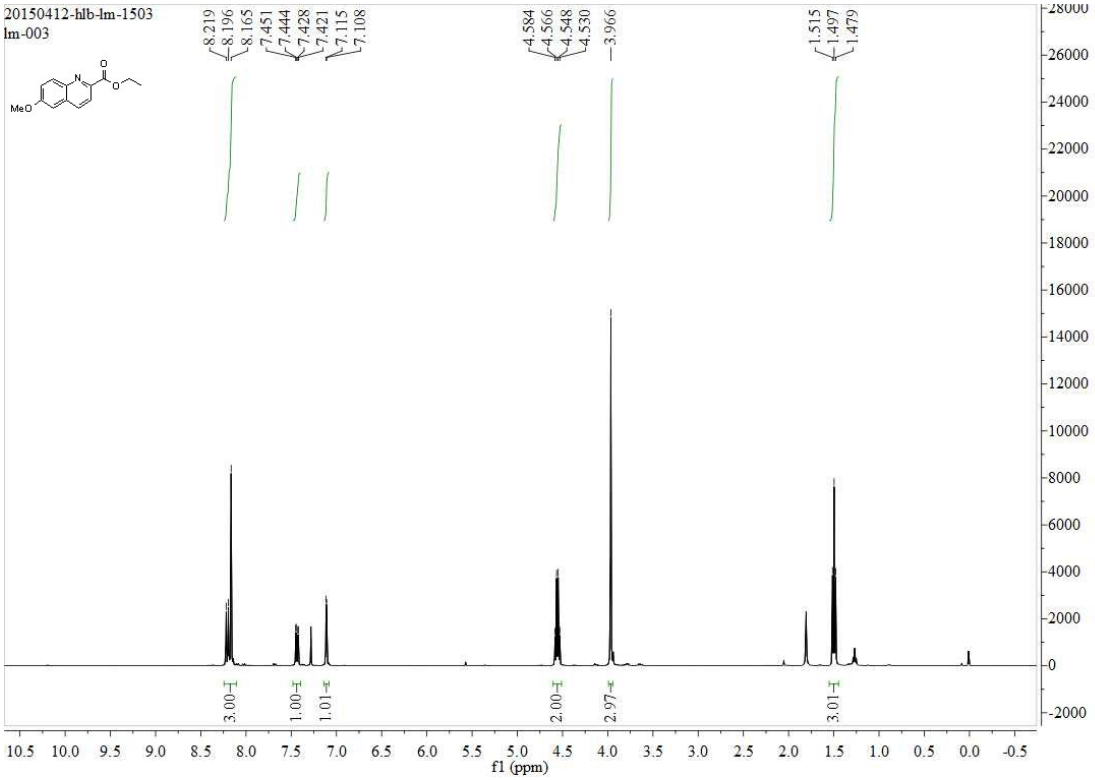
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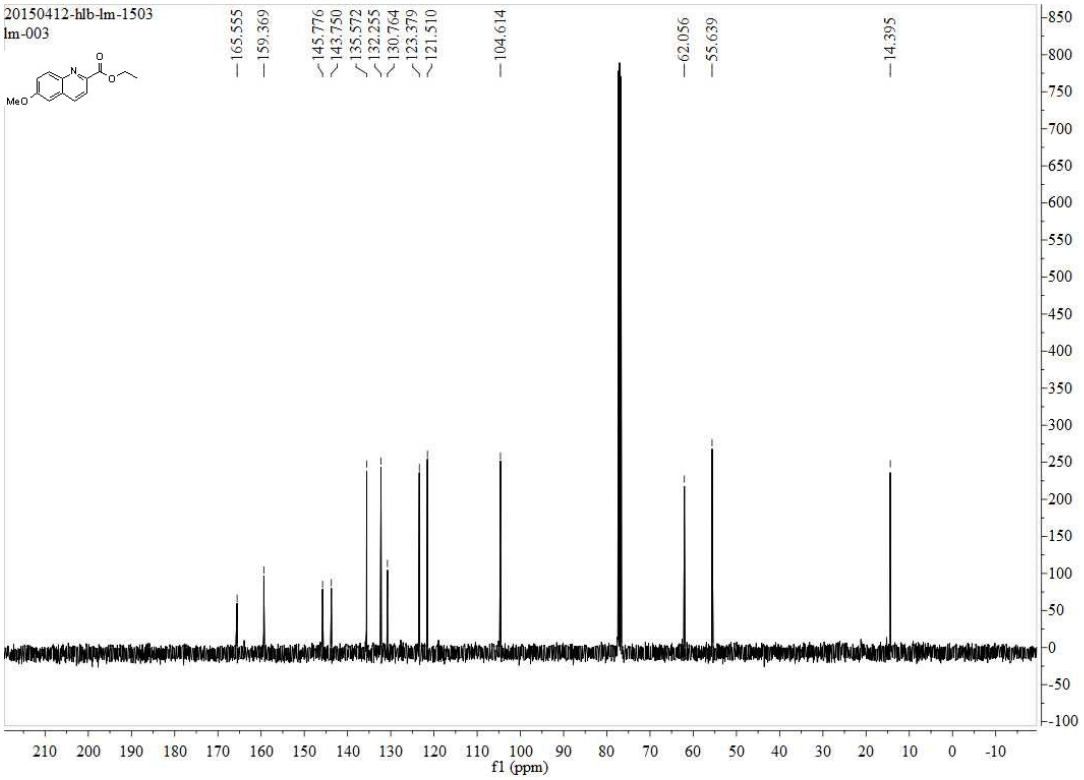
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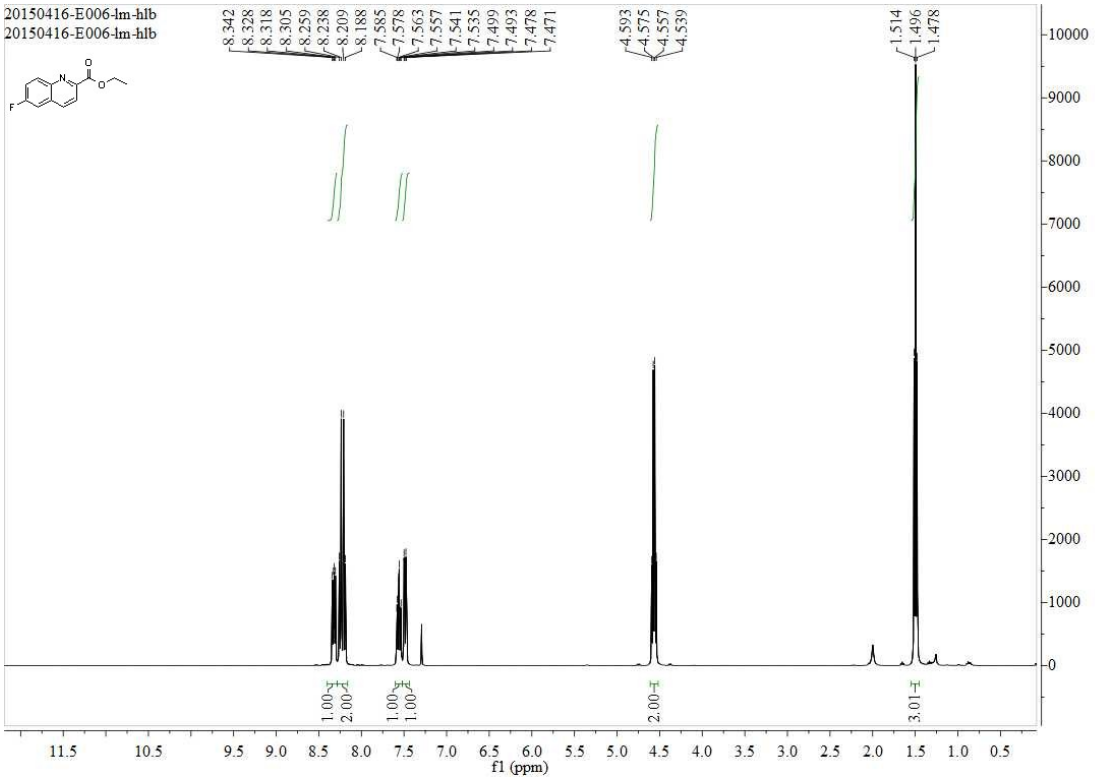
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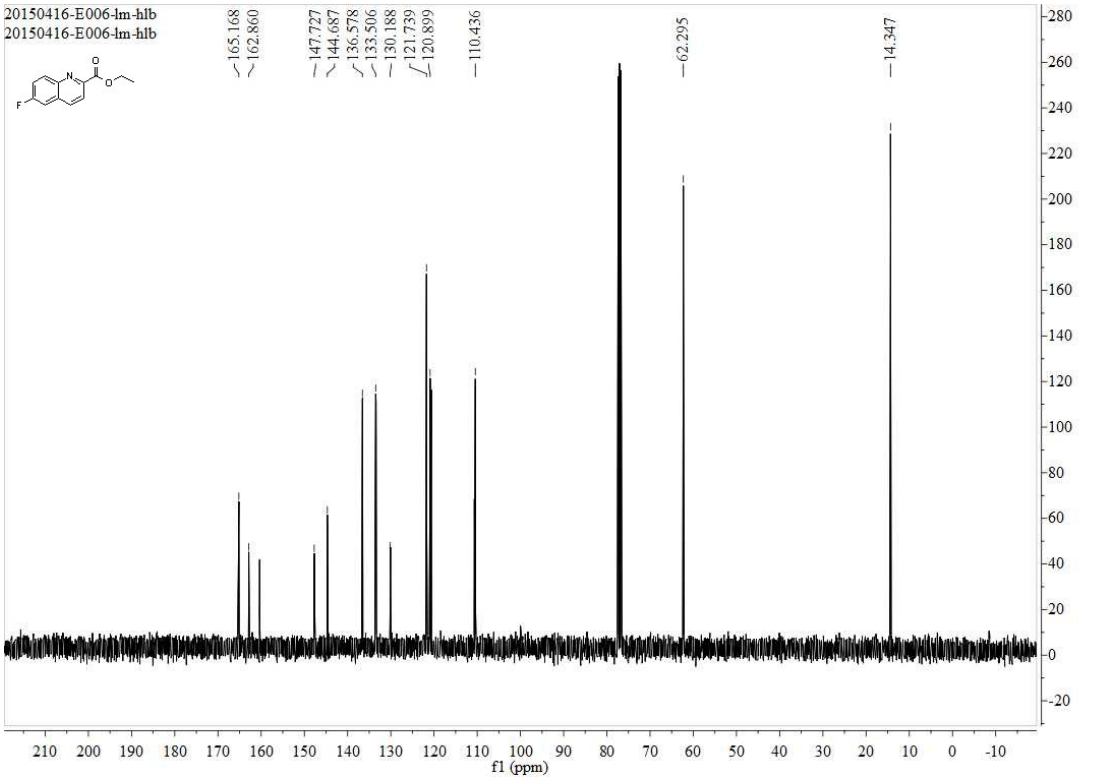
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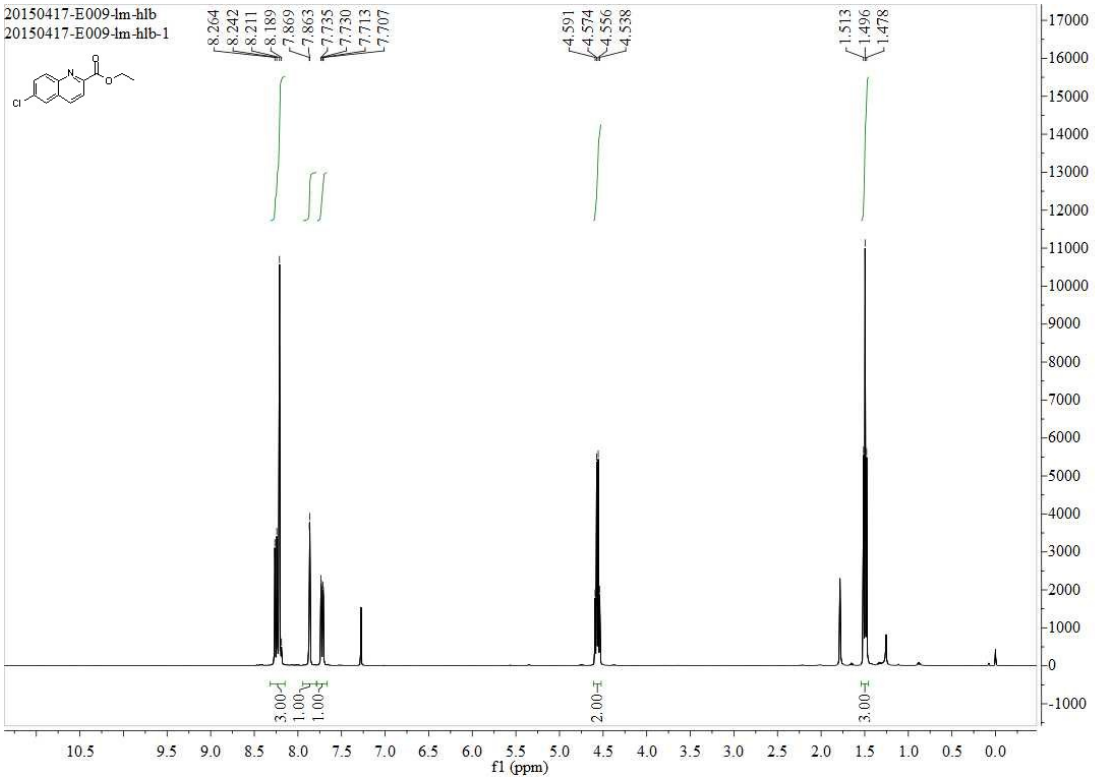
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3.00
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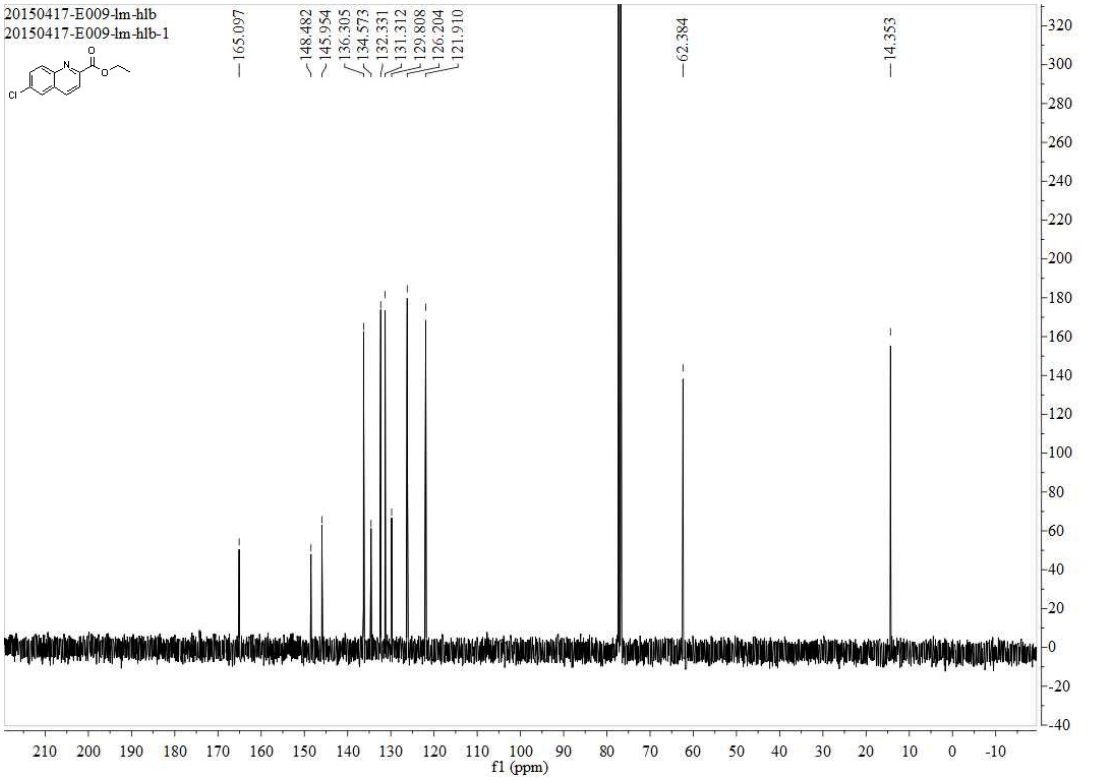
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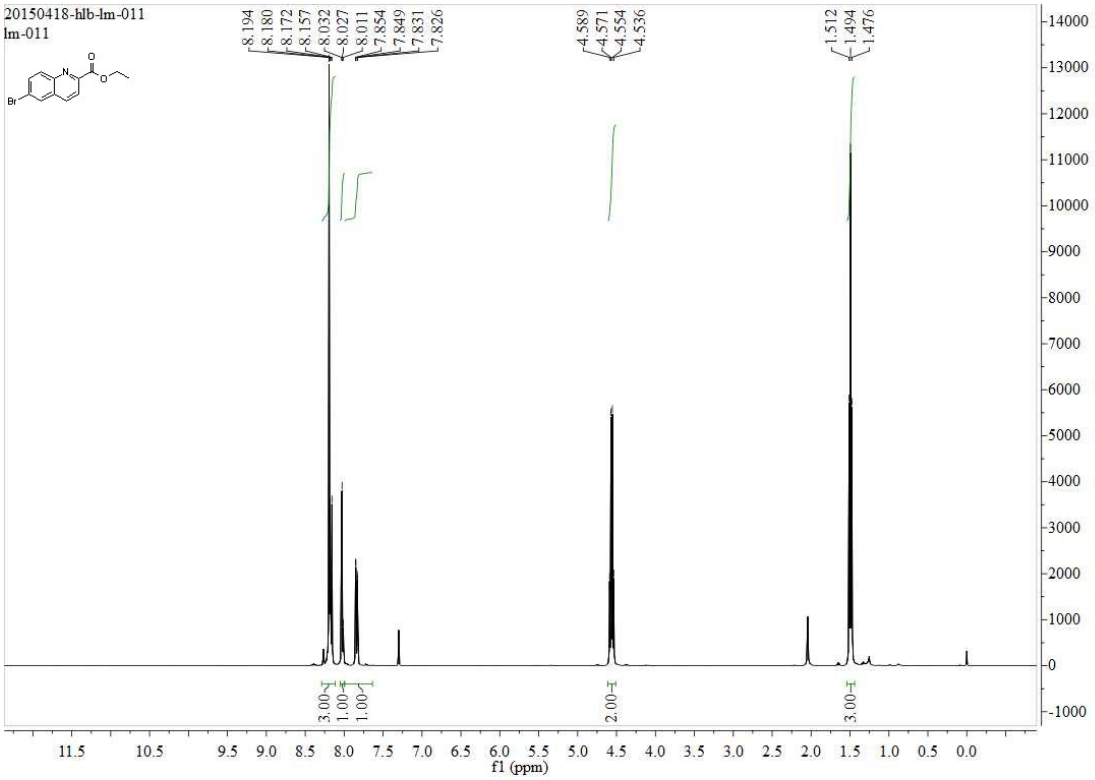
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7.826
4.589
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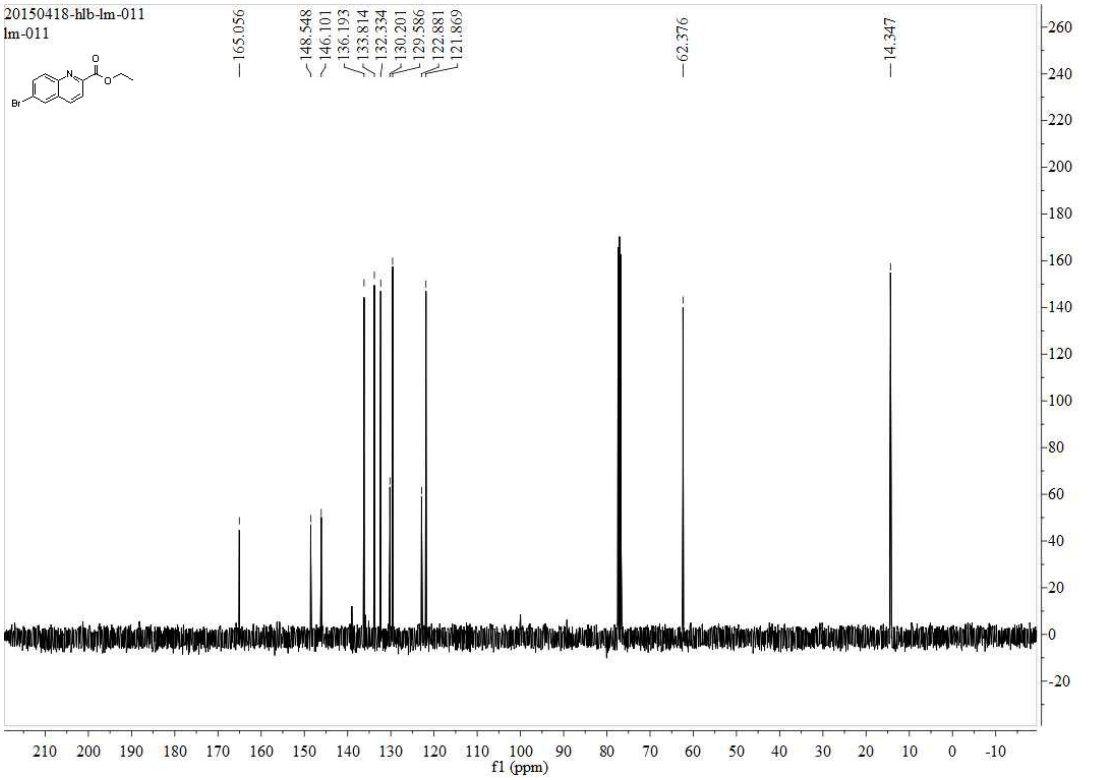
3.00
f1 (ppm)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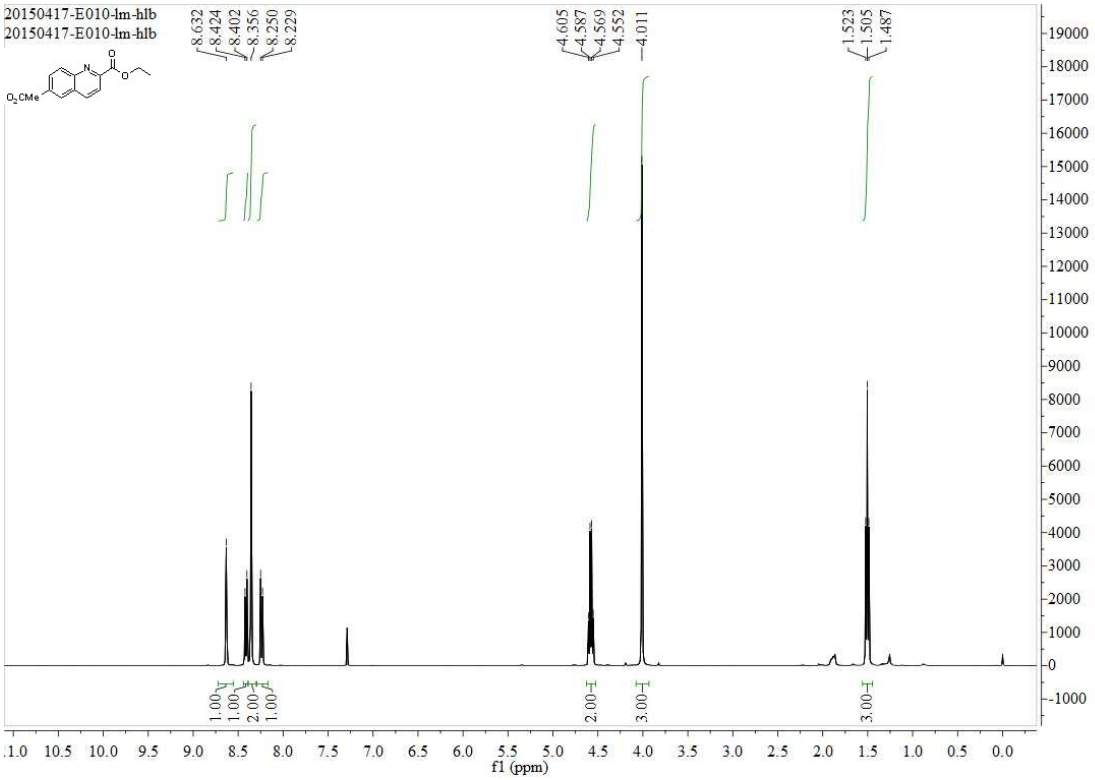
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4.552
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3.00
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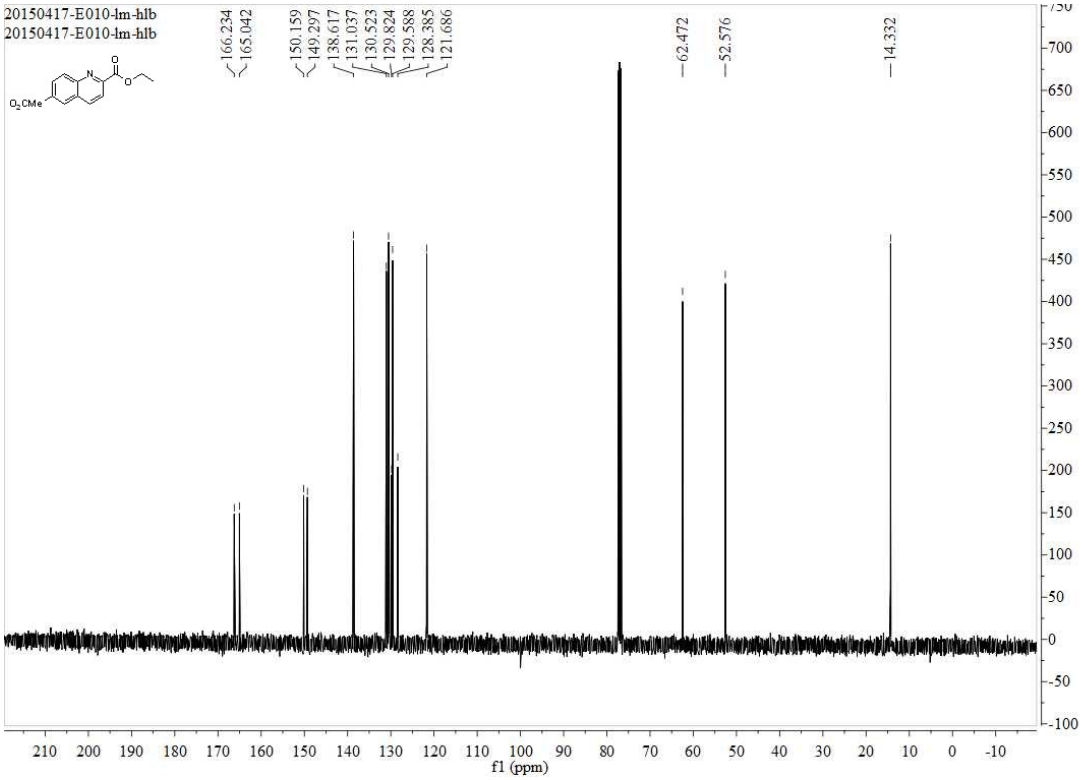
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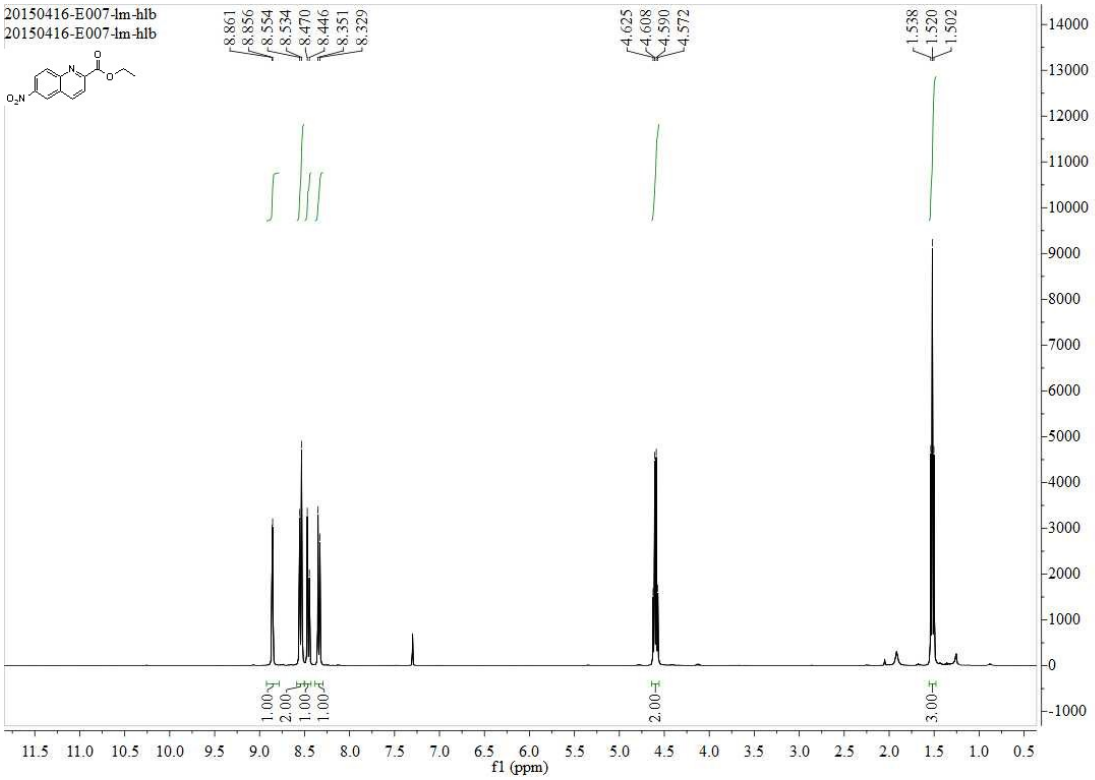
14.332
f1 (ppm)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4.608
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3.00
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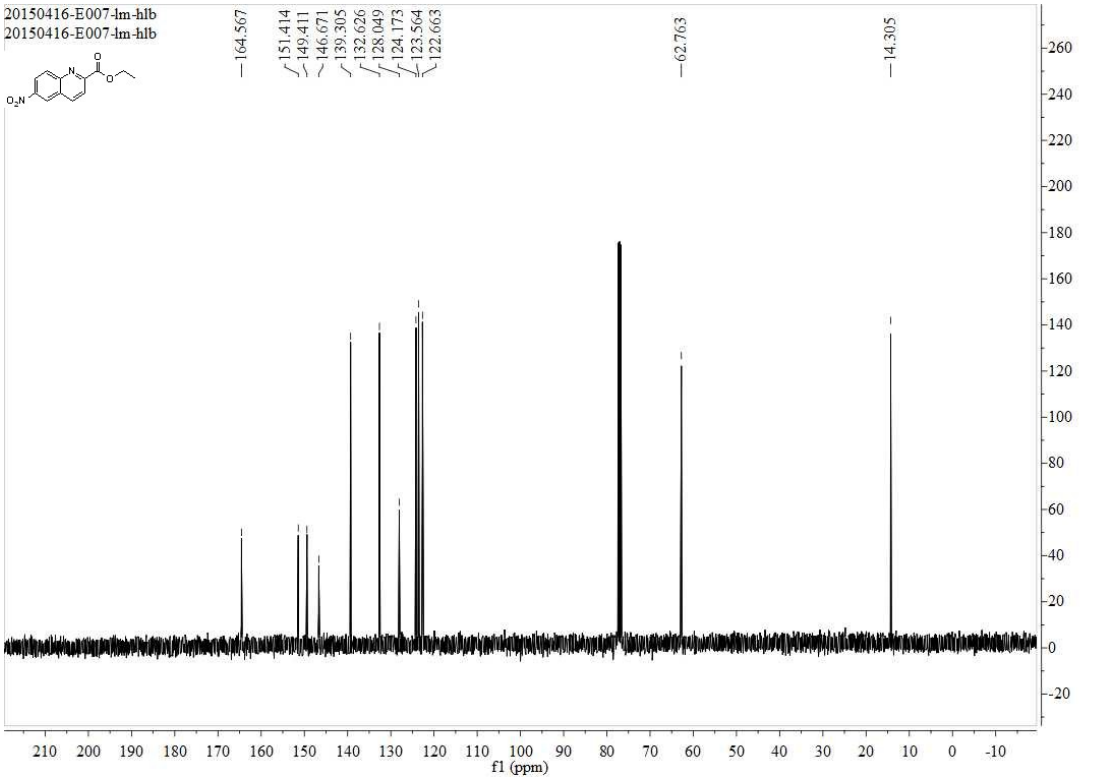
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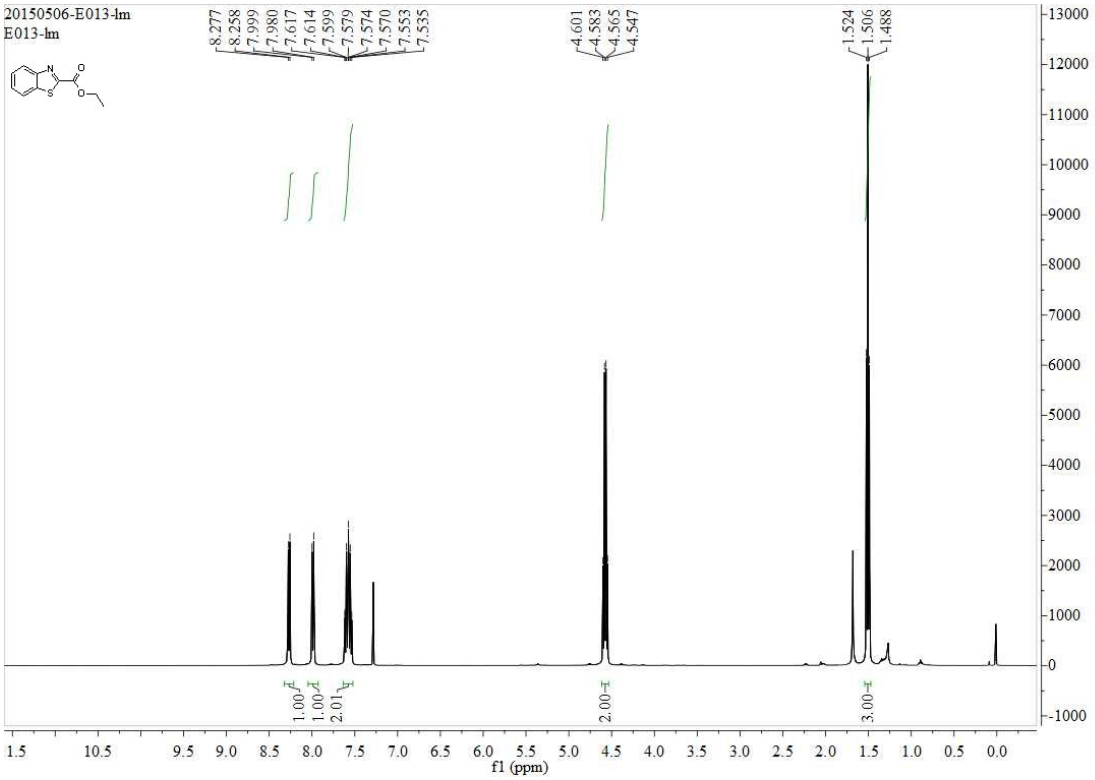
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7.553
7.535
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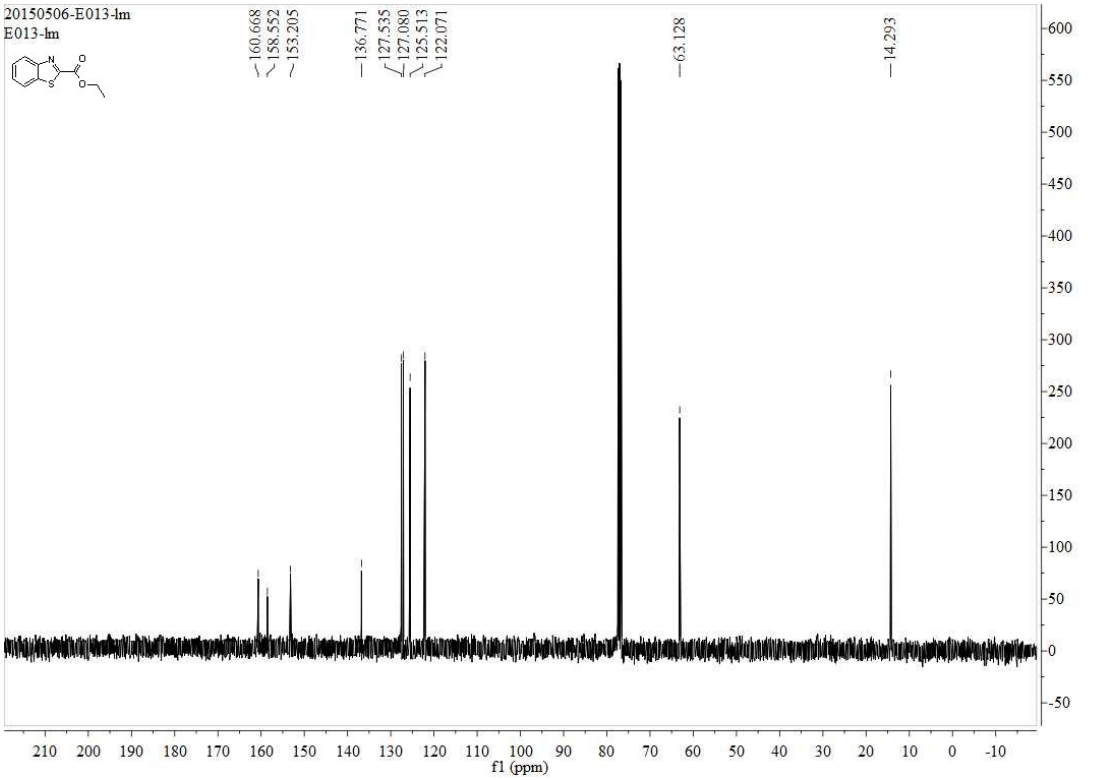
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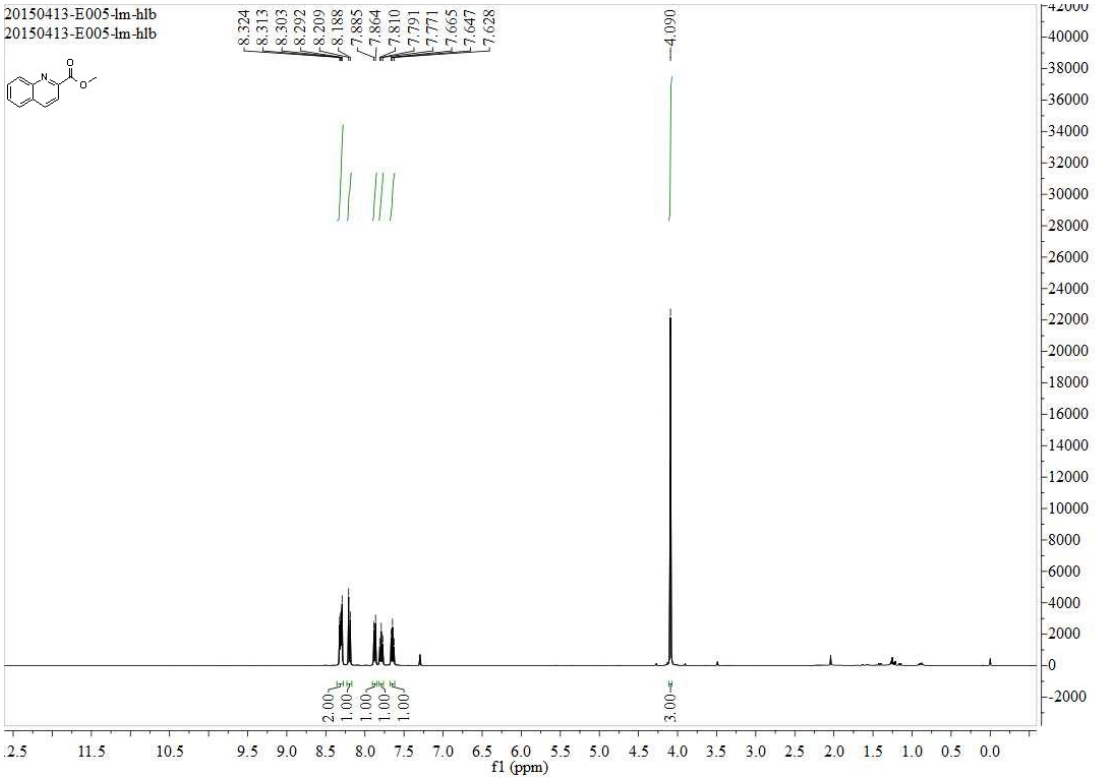
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20150413-E005-lm-hlb
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7.810
7.791
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1.00
1.00
3.00
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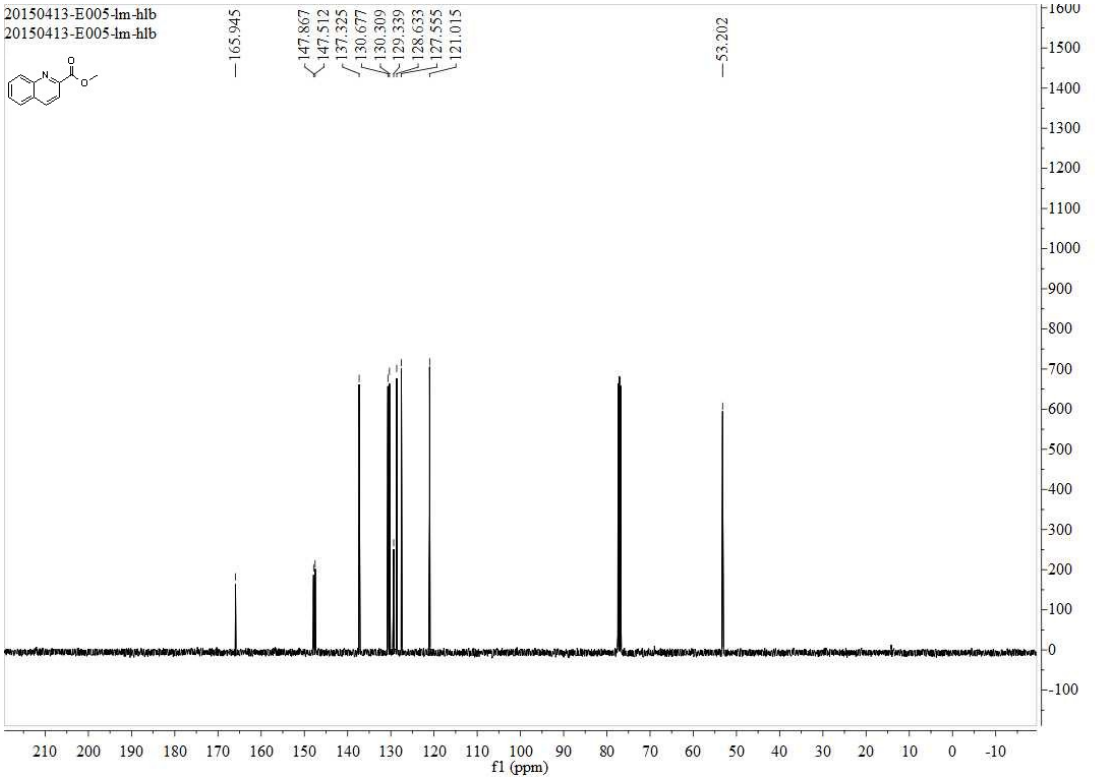
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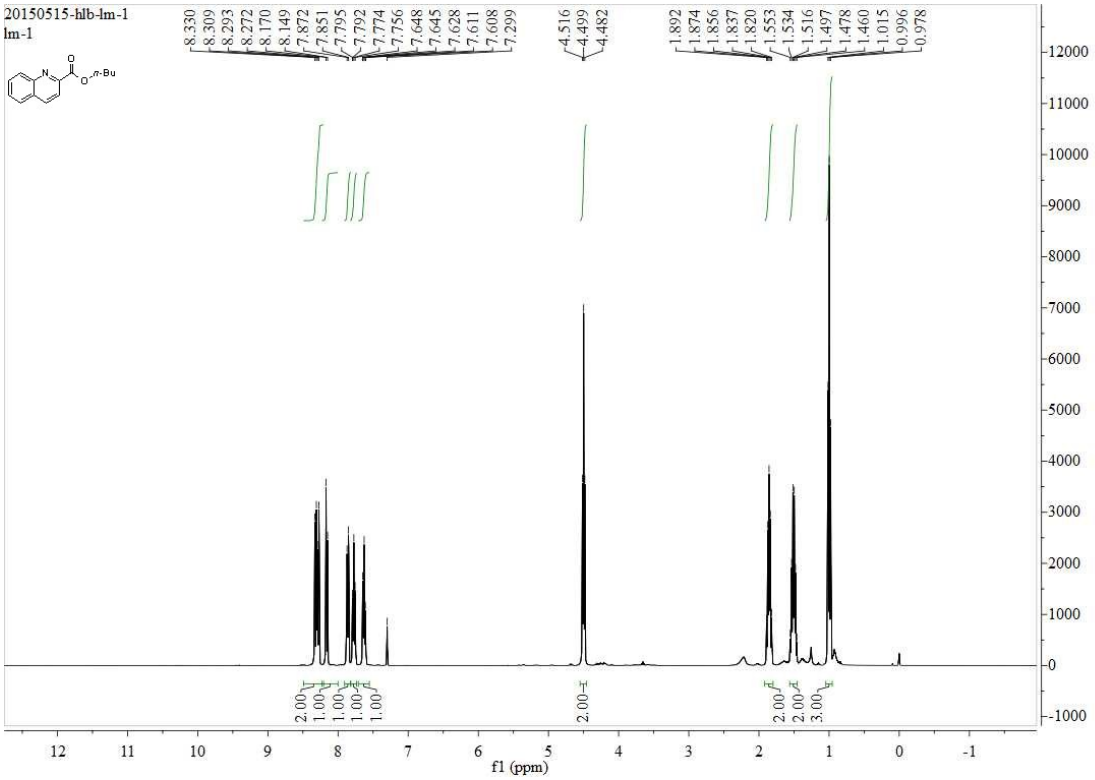
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7.628
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7.608
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4.499
4.482
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1.837
1.820
1.553
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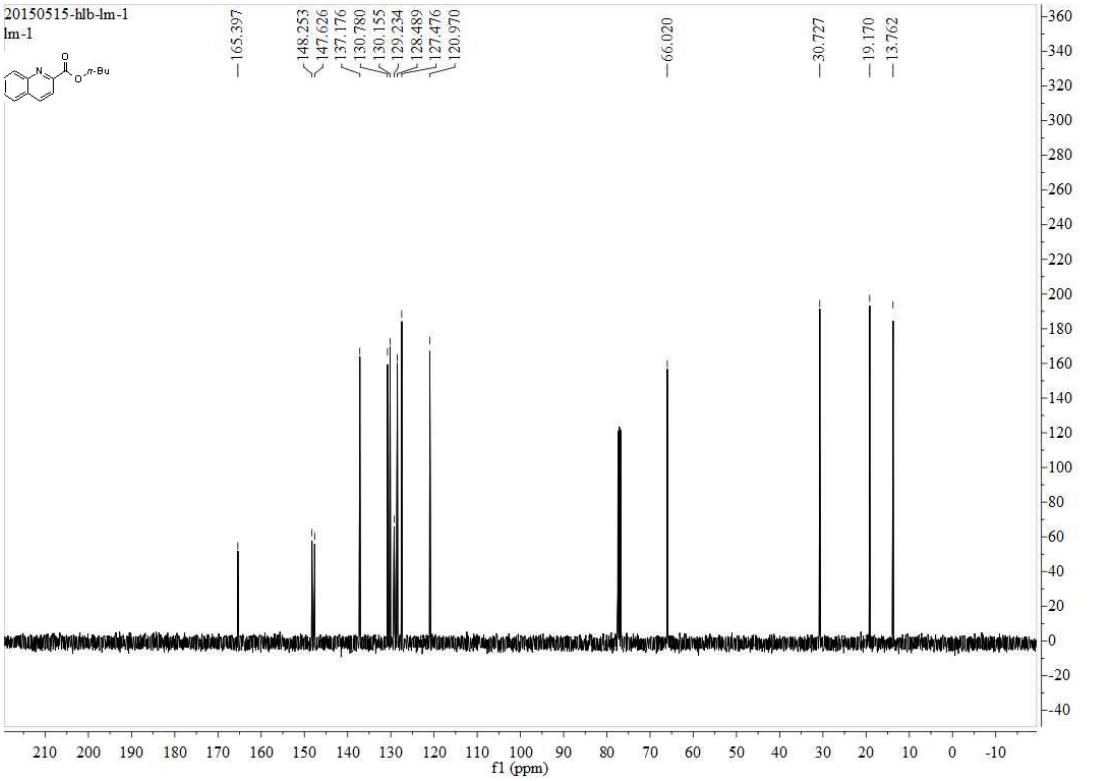
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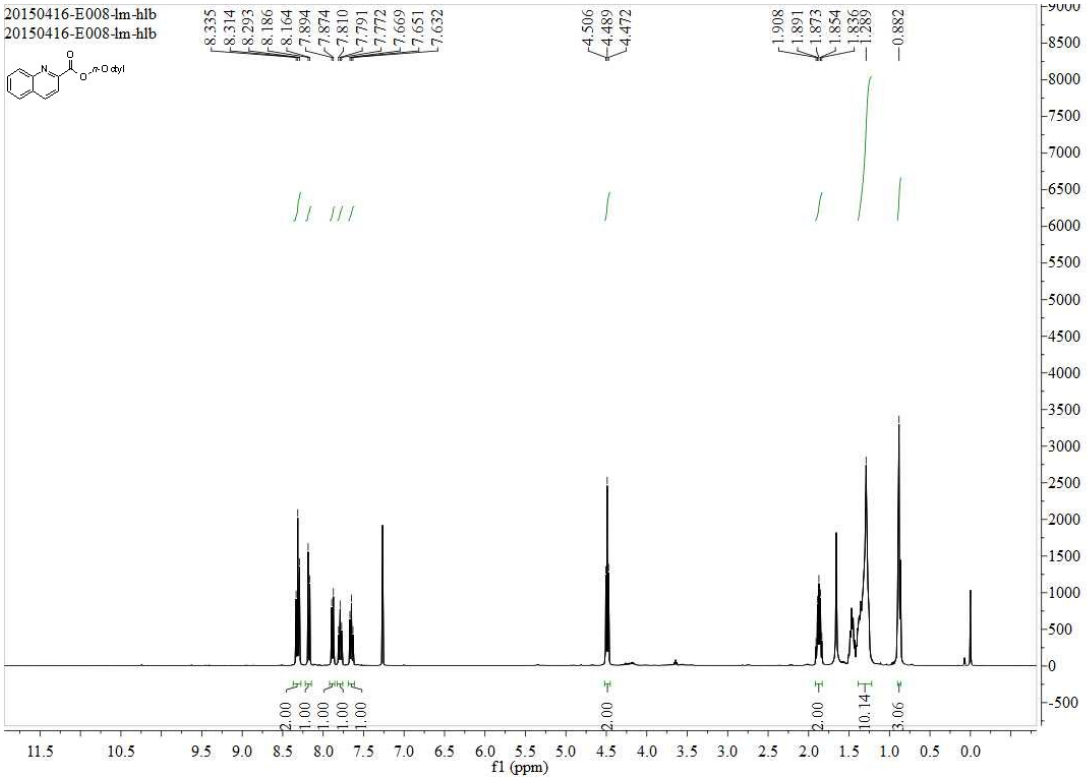
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20150416-E008-lm-hlb
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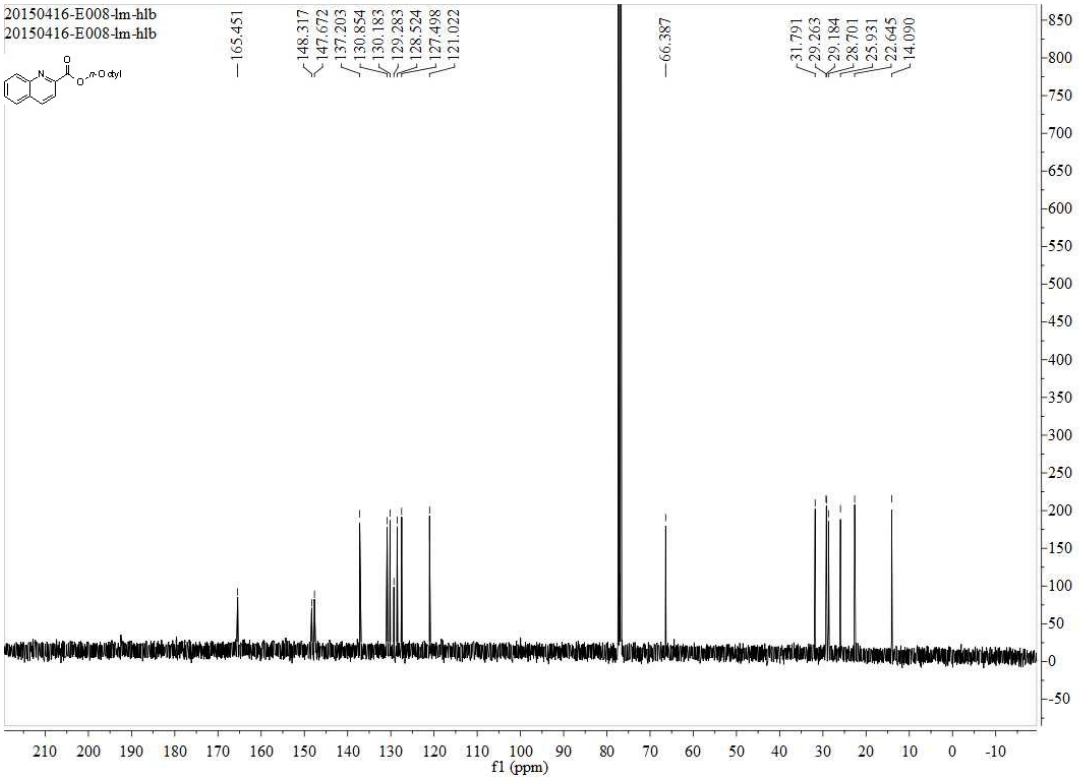
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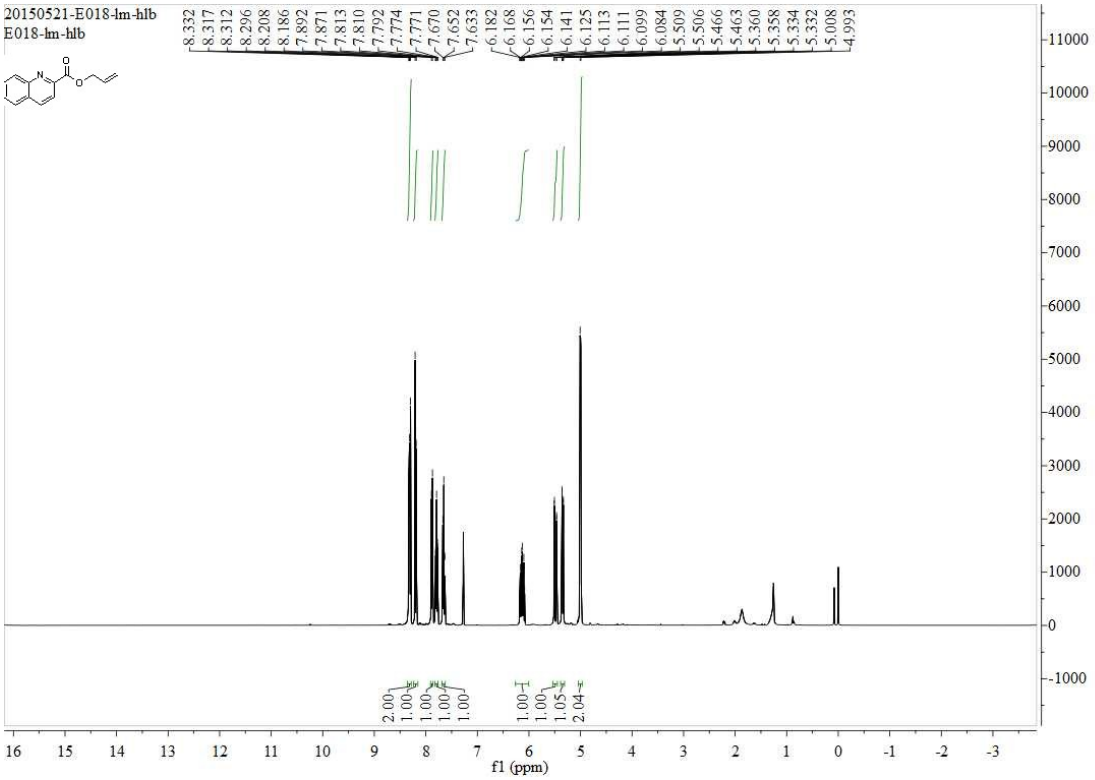
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E018-lm-hlb
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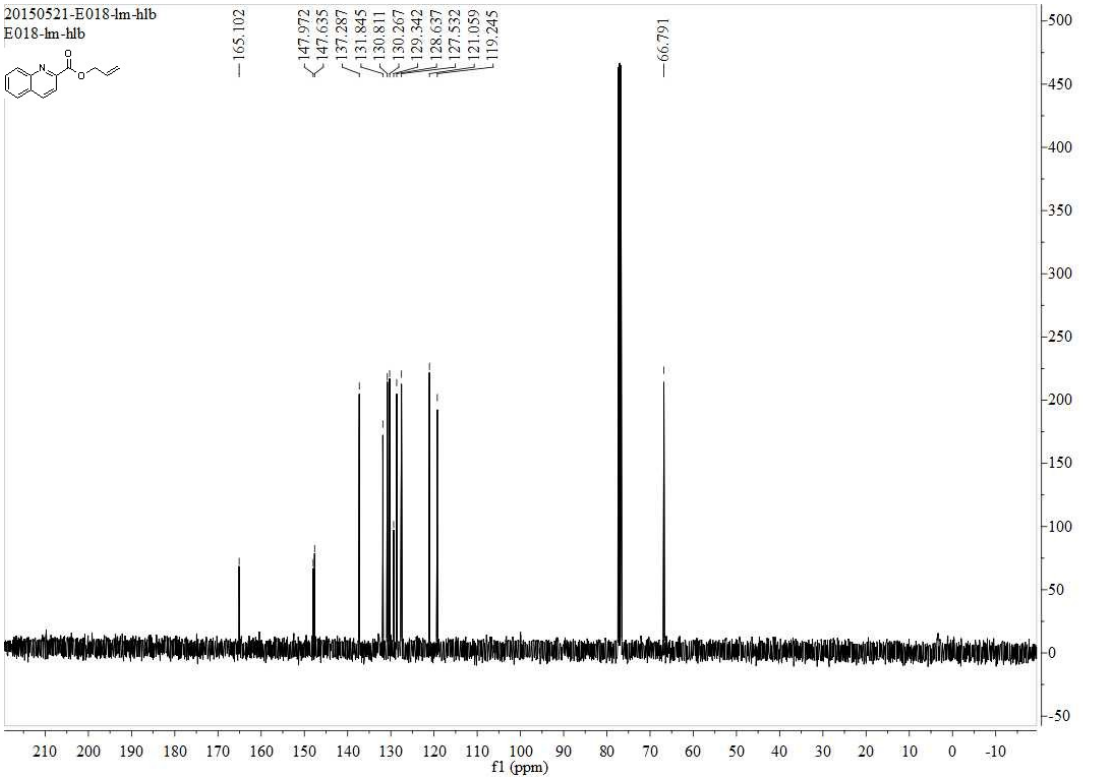
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E018-lm-hlb
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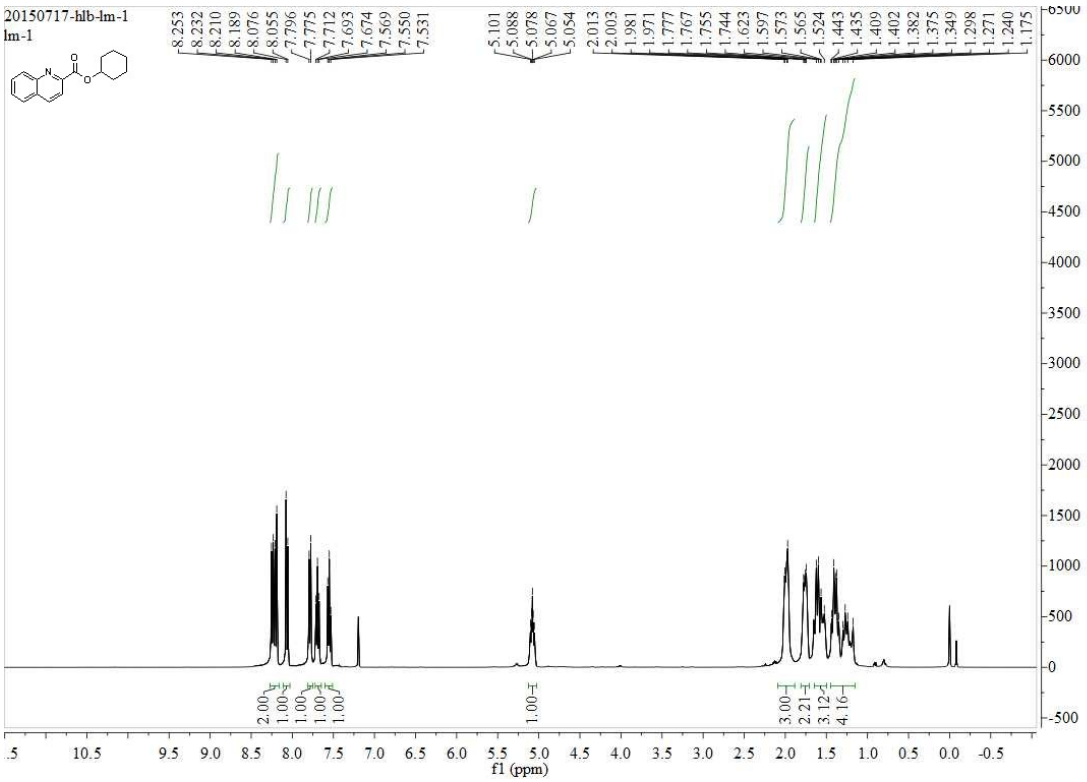
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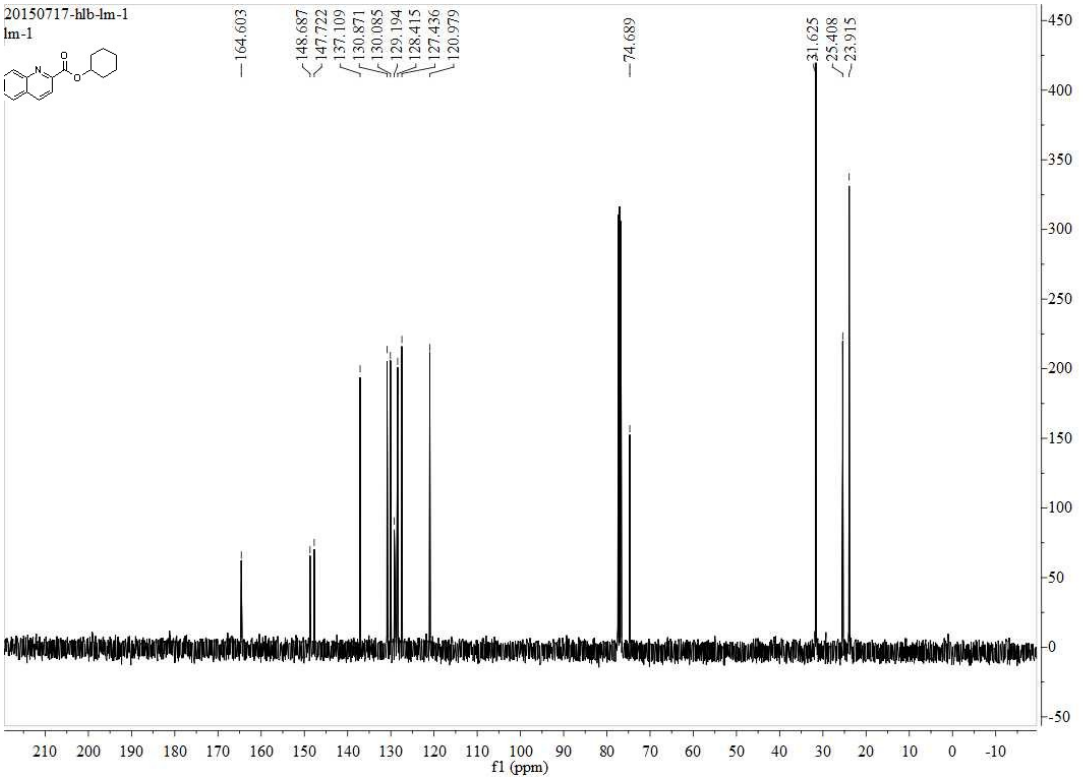
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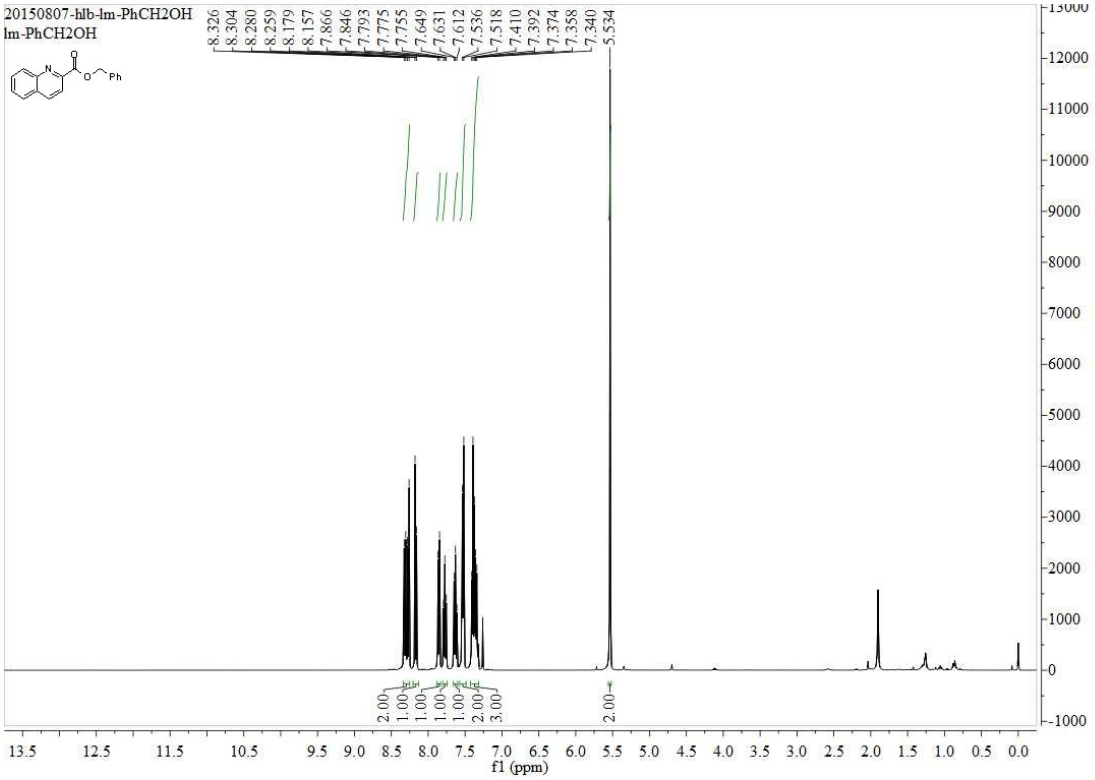
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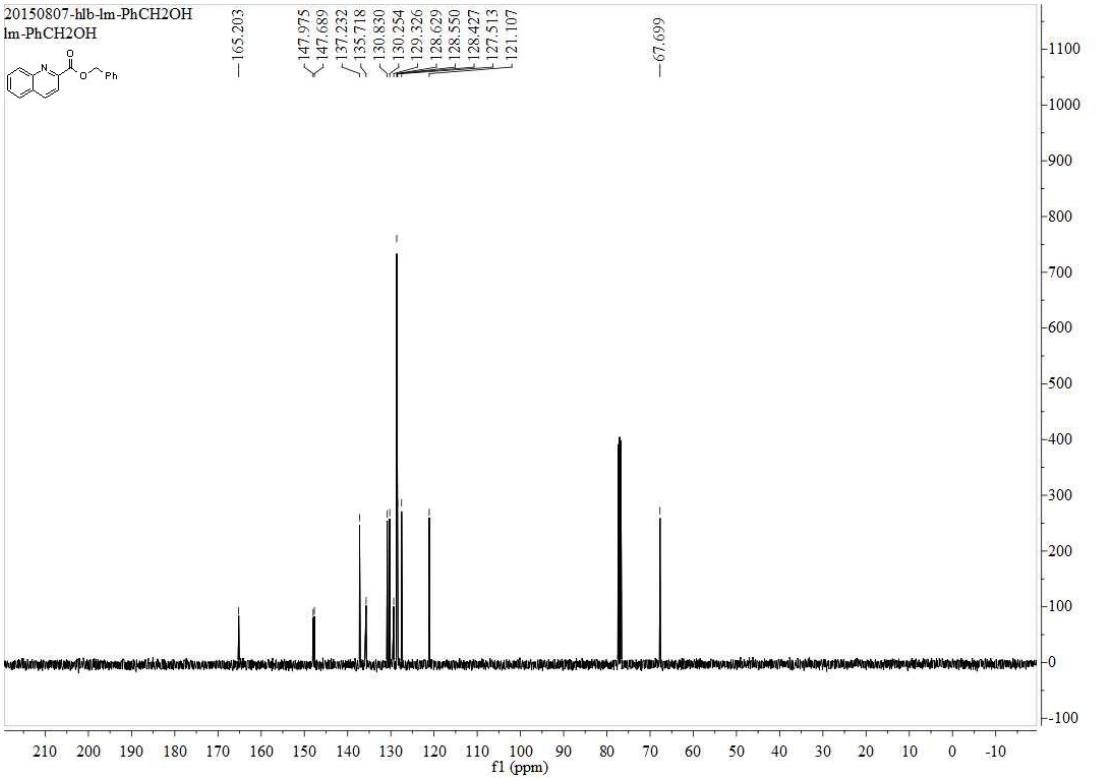
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lm-PhCH2OH
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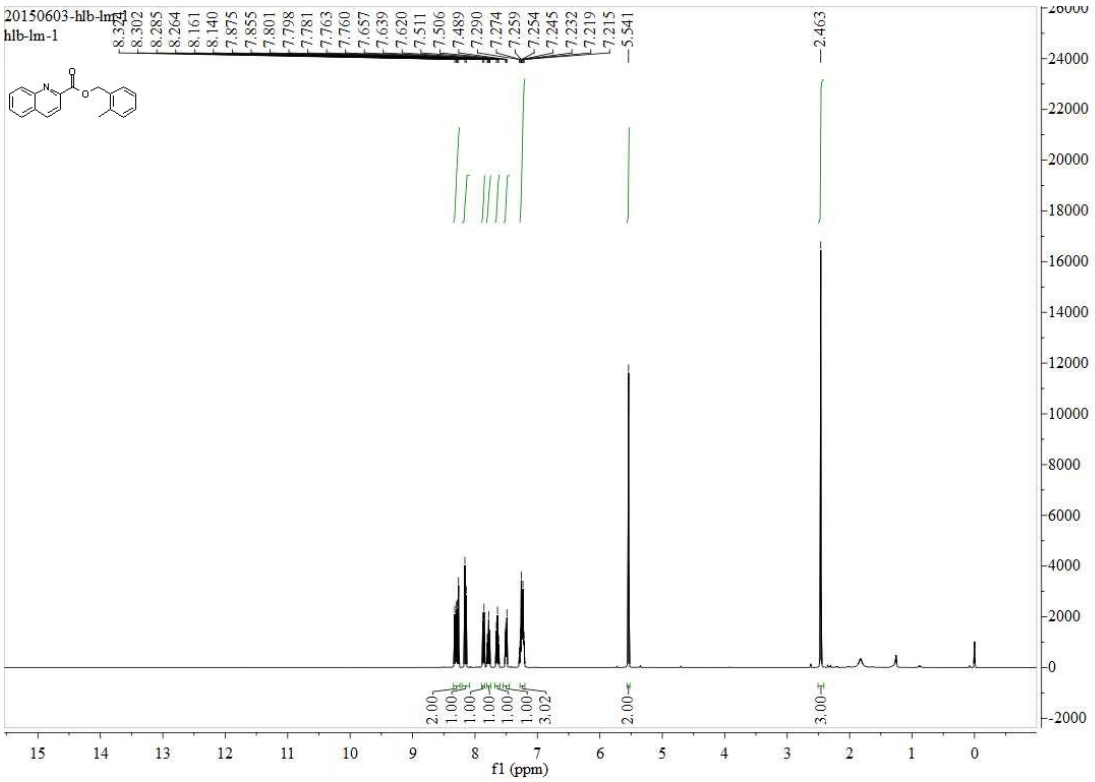
f1 (ppm)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hlb-lm-1
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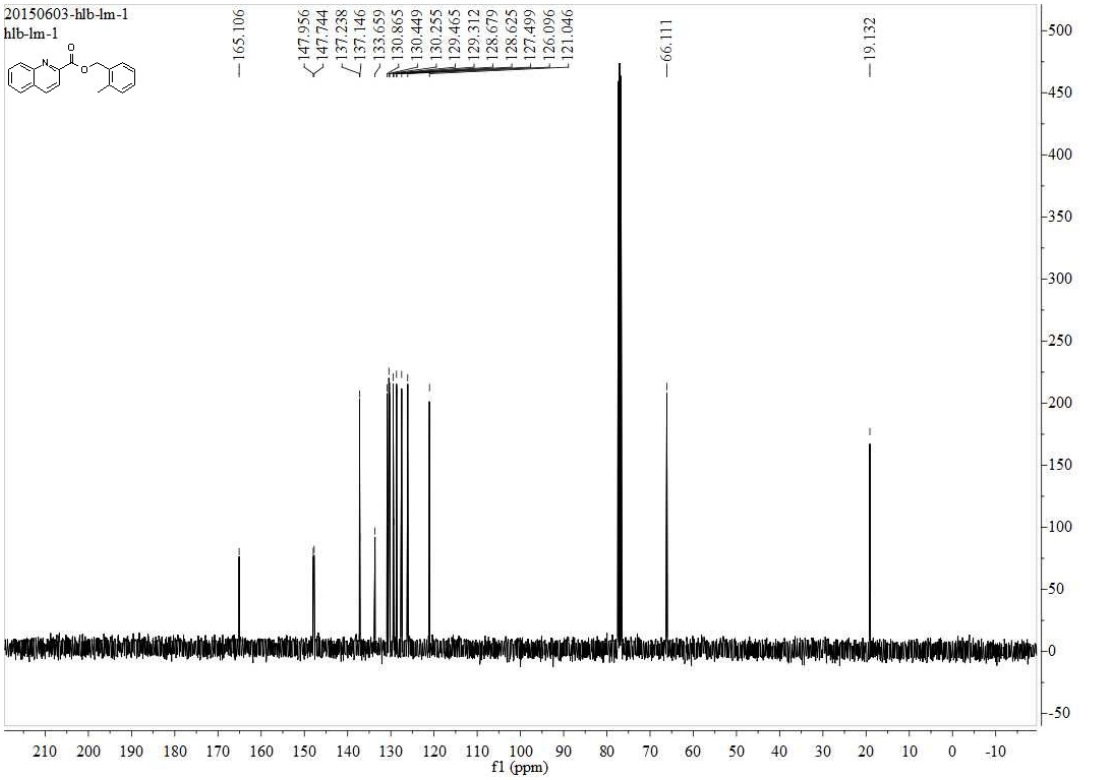
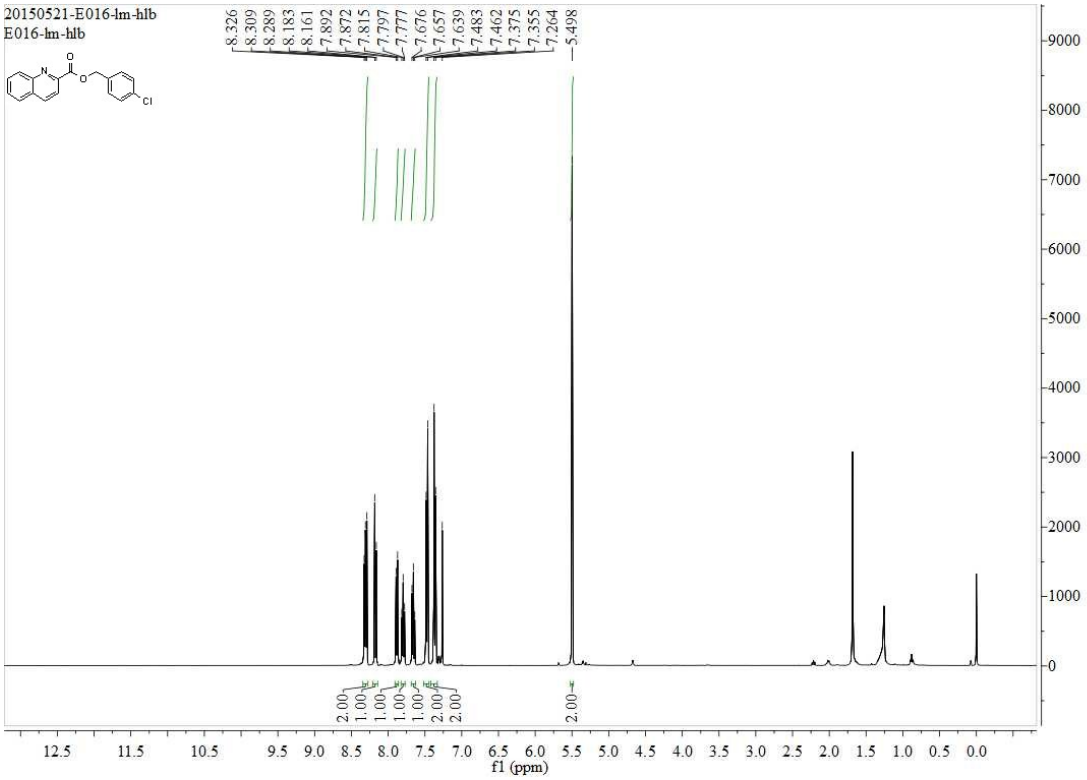
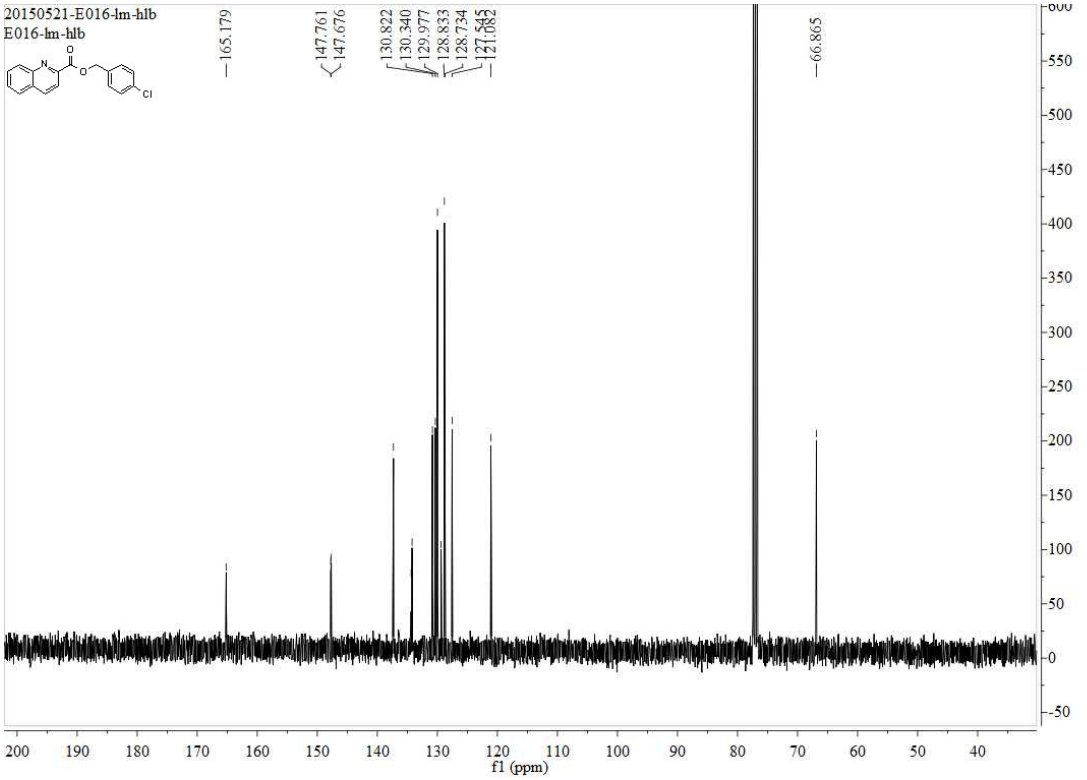
2.00
3.00
f1 (ppm)


20150603-hlb-lm-1
hlb-lm-1
165.106
147.956
147.744
137.238
137.146
133.659
130.865
130.449
130.255
129.465
129.312
128.679
128.625
127.499
126.096
121.046
66.111
19.132
f1 (ppm)


20150521-E016-lm-hlb
E016-lm-hlb
8.326
8.309
8.289
8.183
8.161
7.892
7.872
7.815
7.797
7.777
7.676
7.657
7.639
7.483
7.462
7.375
7.355
7.264
5.498
2.00
1.00
1.00
1.00
1.00
2.00
2.00
2.00
f1 (ppm)


20150521-E016-lm-hlb
E016-lm-hlb
165.179
147.761
147.676
130.822
130.340
129.977
128.833
128.734
66.865
f1 (ppm)