

Photocatalyst-free visible-light-promoted quinazolinone synthesis at room temperature utilizing aldehydes generated *in situ* via C=C bond cleavage

Zongbo Xie*, Jin Lan, Liyuan Yan, Xuehua Chen, Qian Li, Jia Meng and Zhanggao Le*

1. General information

Column chromatography was generally performed on silica gel (200-300 mesh) and reactions were monitored by thin layer chromatography (TLC) visualize the course of the reactions. The ^1H NMR and ^{13}C NMR data were recorded on Bruker 500 M spectrometer. Benzaldehyde was demonstrated by HPLC using n-hexane: isopropyl alcohol (99:1).

2. Setup for photocatalytic reactions

The reaction setup is depicted in **Figure S1**. 18W LED lights are available for purchase. During the first experiment the temperature was monitored inside the crystallizing dish and did not exceed the room temperature (20–30 °C). Magnetic stirring was performed with 200 rpm.



Figure S1: LED reaction setup.

3. Analytical data of the products

2-phenylquinazolin-4(3H)-one (3a)¹: White solid. m.p: 241.2-243.9 °C. ^1H NMR (500 MHz, DMSO- d_6) δ 12.55 (s, 1H), 8.21 – 8.14 (m, 3H), 7.85 (ddd, J = 8.5, 7.0, 1.7 Hz, 1H), 7.75 (dd, J = 8.2, 1.0 Hz, 1H), 7.61 – 7.48 (m, 4H); ^{13}C NMR (126 MHz, DMSO) δ 162.7, 152.8, 149.2, 135.1, 133.2, 131.9, 129.1, 128.2, 128.0, 127.1, 126.3, 121.5.

2-(p-tolyl)quinazolin-4(3H)-one (3b)¹: White solid. m.p: 238.3-241.0 °C. ^1H NMR (500 MHz, DMSO- d_6) δ 12.47 (s, 1H), 8.16 (d, J = 7.9 Hz, 1H), 8.11 (d, J = 8.1 Hz, 2H), 7.88 – 7.79 (m, 1H), 7.73 (d, J = 8.1 Hz, 1H), 7.51 (t, J = 7.6 Hz, 1H), 7.36 (dd, J = 7.8, 3.2 Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (126 MHz, DMSO) δ 162.8, 152.7, 149.3, 141.9, 135.0, 130.4, 129.7, 128.2, 127.8, 126.8, 126.3, 121.4, 21.5.

2-(4-methoxyphenyl)quinazolin-4(3H)-one (3c)¹: White solid. m.p: 247.3-250.2 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 12.42 (s, 1H), 8.22 (d, *J* = 8.7 Hz, 2H), 8.16 (d, *J* = 7.7 Hz, 1H), 7.82 (t, *J* = 7.4 Hz, 1H), 7.72 (d, *J* = 8.1 Hz, 1H), 7.50 (t, *J* = 7.4 Hz, 1H), 7.10 (d, *J* = 8.6 Hz, 2H), 3.86 (s, 3H); ¹³C NMR (126 MHz, DMSO) δ 162.8, 162.3, 152.3, 149.4, 135.0, 129.9, 127.8, 126.6, 126.3, 125.3, 121.2, 114.5, 55.9.

2-(4-chlorophenyl)quinazolin-4(3H)-one (3d)¹: White solid. m.p: 294.6-297.7 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 12.61 (s, 1H), 8.21 (d, *J* = 8.6 Hz, 2H), 8.16 (d, *J* = 7.4 Hz, 1H), 7.85 (t, *J* = 7.6 Hz, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.63 (d, *J* = 8.6 Hz, 2H), 7.54 (t, *J* = 7.4 Hz, 1H); ¹³C NMR (126 MHz, DMSO) δ 162.6, 151.8, 149.1, 136.8, 135.2, 132.0, 130.1, 129.2, 128.0, 127.3, 126.3, 121.5.

2-([1,1'-biphenyl]-4-yl)quinazolin-4(3H)-one (3e)²: White solid. m.p: 266.6-269.7 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 12.62 (s, 1H), 8.31 (d, *J* = 8.5 Hz, 2H), 8.18 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.88 (d, *J* = 8.6 Hz, 3H), 7.83 – 7.75 (m, 3H), 7.52 (t, *J* = 8.2 Hz, 3H), 7.44 (d, *J* = 7.4 Hz, 1H); ¹³C NMR (126 MHz, DMSO) δ 162.7, 152.4, 149.2, 143.3, 139.4, 135.1, 132.0, 129.6, 128.9, 128.7, 128.0, 127.3, 127.2, 127.1, 126.4, 121.5.

2-(naphthalen-2-yl)quinazolin-4(3H)-one (3f)¹: White solid. m.p: 246.9-249.2 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 12.70 (s, 1H), 8.83 (s, 1H), 8.31 (s, 1H), 8.19 (d, *J* = 7.2 Hz, 1H), 8.05 (dd, *J* = 29.6, 7.6 Hz, 3H), 7.93 – 7.73 (m, 2H), 7.60 (d, *J* = 47.3 Hz, 3H); ¹³C NMR (126 MHz, DMSO) δ 162.7, 152.7, 149.3, 135.1, 134.6, 132.8, 130.4, 129.4, 128.6, 128.6, 128.4, 128.1, 128.0, 127.4, 127.1, 126.4, 125.0, 121.5.

2-(pyridin-2-yl)quinazolin-4(3H)-one (3g)³: White solid. m.p: 271.3-275.6 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.81 (s, 1H), 8.77 (d, *J* = 4.5 Hz, 1H), 8.46 (d, *J* = 7.9 Hz, 1H), 8.21 (d, *J* = 8.9 Hz, 1H), 8.08 (td, *J* = 7.8, 1.6 Hz, 1H), 7.93 – 7.85 (m, 1H), 7.81 (d, *J* = 7.9 Hz, 1H), 7.71 – 7.64 (m, 1H), 7.63 – 7.55 (m, 1H); ¹³C NMR (126 MHz, DMSO) δ 161.2, 150.4, 149.4, 149.1, 148.9, 138.5, 135.2, 128.2, 127.7, 127.0, 126.6, 122.6, 122.5.

(E)-2-styrylquinazolin-4(3H)-one (3h)¹: White solid. m.p: 245.5-248.4 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 12.37 (s, 1H), 8.12 (d, *J* = 7.9 Hz, 1H), 7.96 (d, *J* = 16.2 Hz, 1H), 7.82 (t, *J* = 7.6 Hz, 1H), 7.68 (t, *J* = 7.6 Hz, 3H), 7.48 (q, *J* = 8.0 Hz, 3H), 7.43 (d, *J* = 7.1 Hz, 1H), 7.02 (d, *J* = 16.2 Hz, 1H); ¹³C NMR (126 MHz, DMSO) δ 162.2, 151.9, 149.5, 138.7, 135.4, 135.0, 130.3, 129.6, 128.1, 127.6, 126.7, 126.3, 121.5.

6-chloro-2-(p-tolyl)quinazolin-4(3H)-one (3i)⁴: White solid. m.p: 302.1-304.9 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 12.66 (s, 1H), 8.10 (s, 3H), 7.90 – 7.82 (m, 1H), 7.75 (d, *J* = 8.5 Hz, 1H), 7.37 (d, *J* = 6.8 Hz, 2H), 2.40 (s, 3H); ¹³C NMR (126 MHz, DMSO) δ 161.8, 153.2, 142.2, 135.2, 132.0, 131.0, 130.1, 130.1, 129.7, 128.2, 125.3, 21.5.

7-methoxy-2-(p-tolyl)quinazolin-4(3H)-one (3j)⁵: White solid. m.p: 266.5-269.7 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 12.47 (s, 1H), 8.08 (d, *J* = 8.2 Hz, 2H), 7.69 (d, *J* = 8.9 Hz, 1H), 7.54 (d, *J* = 3.0 Hz, 1H), 7.44 (dd, *J* = 8.9, 3.0 Hz, 1H), 7.35 (d, *J* = 8.1 Hz, 2H), 3.89 (s, 3H), 2.39 (s, 3H); ¹³C NMR (126 MHz, DMSO) δ 162.5, 158.1, 150.5, 143.8, 141.5, 130.4, 129.7, 127.9, 124.6, 122.1, 106.3, 56.1, 21.4.

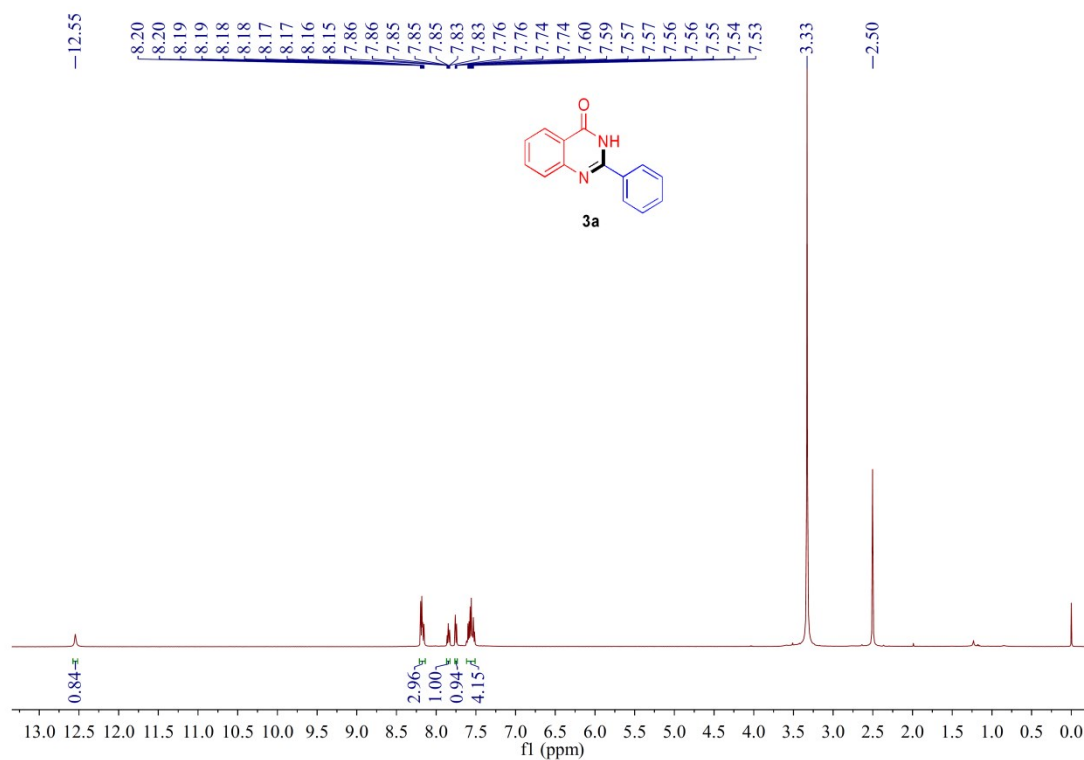
3-methyl-2-(p-tolyl)quinazolin-4(3H)-one (3k)⁶: White solid. m.p: 139.2-144.1 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 8.18 (d, *J* = 7.9 Hz, 1H), 7.87 – 7.77 (m, 1H), 7.71 – 7.63 (m, 1H), 7.60 – 7.55 (m, 2H), 7.53 (d, *J* = 8.4 Hz, 1H), 7.35 (d, *J* = 8.1 Hz, 2H), 2.41 (s, 3H); ¹³C NMR (126 MHz, DMSO) δ 162.2, 156.7, 147.6, 140.0, 134.7, 133.1, 129.4, 128.8, 127.6, 127.2, 126.5, 120.5, 34.4, 21.4.

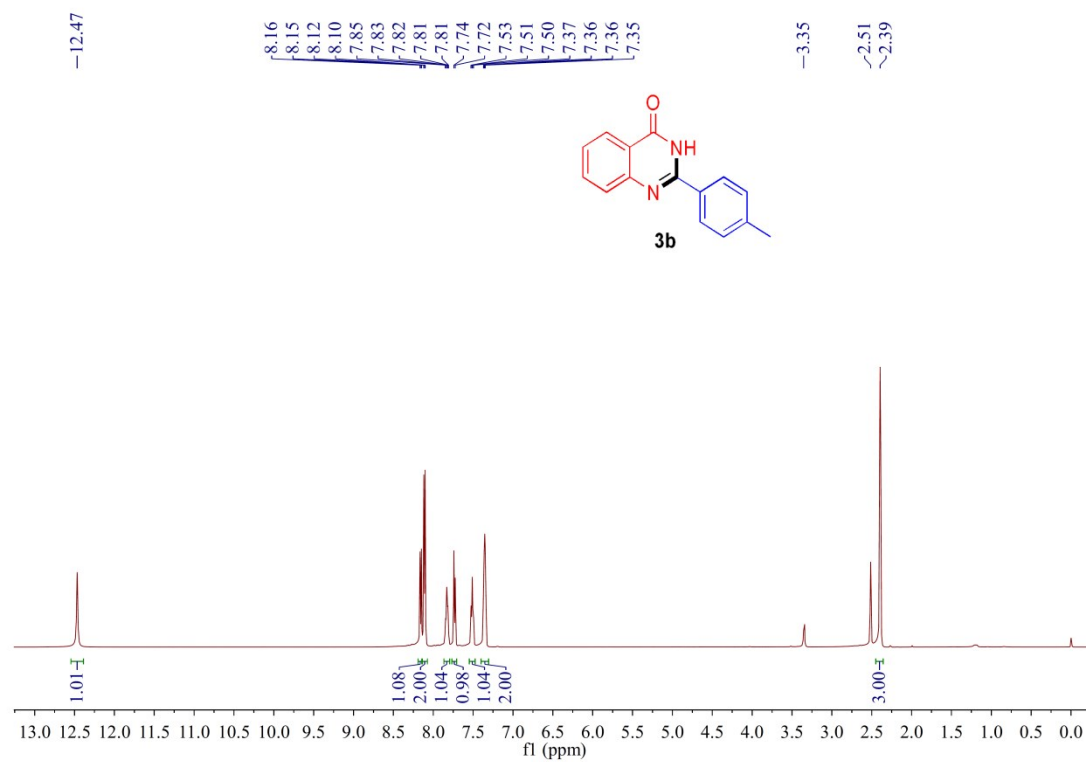
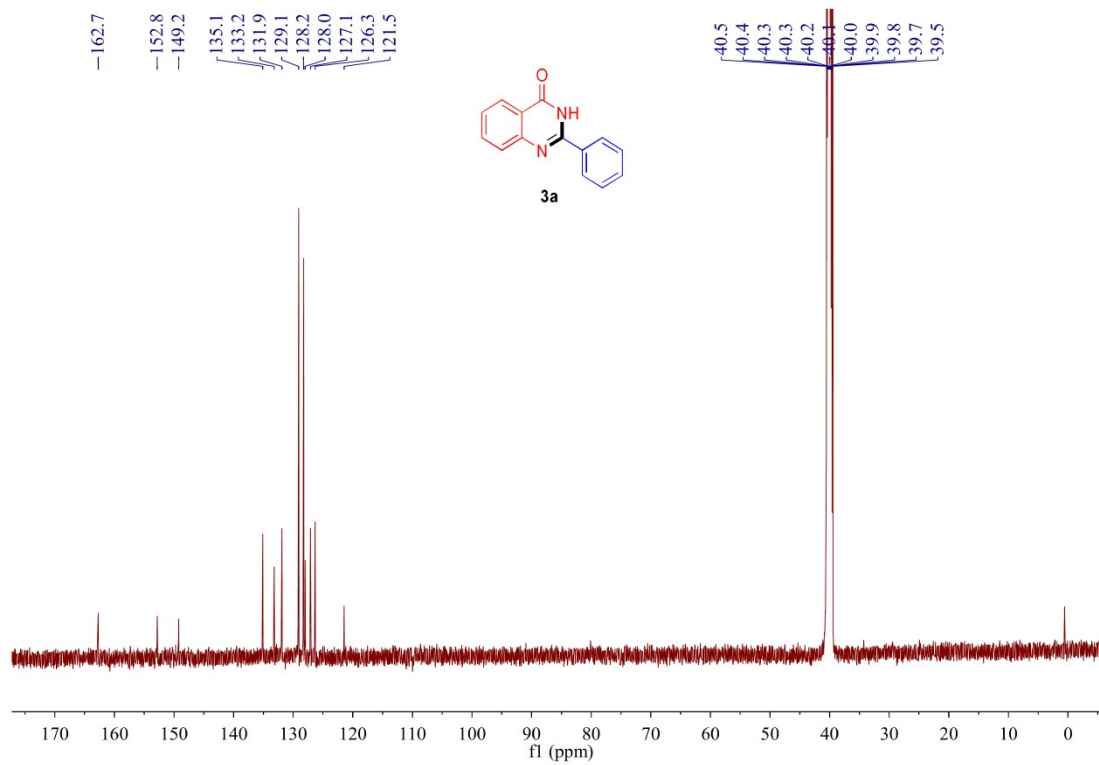
3-methyl-2-phenylquinazolin-4(3H)-one (3l)⁷: White solid. m.p: 133.3-135.5 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 8.19 (d, *J* = 7.3 Hz, 1H), 7.89 – 7.80 (m, 1H), 7.68 (d, *J* = 7.6 Hz, 3H), 7.56 (s, 4H), 3.37 (s, 3H); ¹³C NMR (126 MHz, DMSO) δ 162.1, 156.6, 147.5, 135.9, 134.8, 130.3, 128.9, 128.7, 127.7, 127.4, 126.6, 120.6, 34.4.

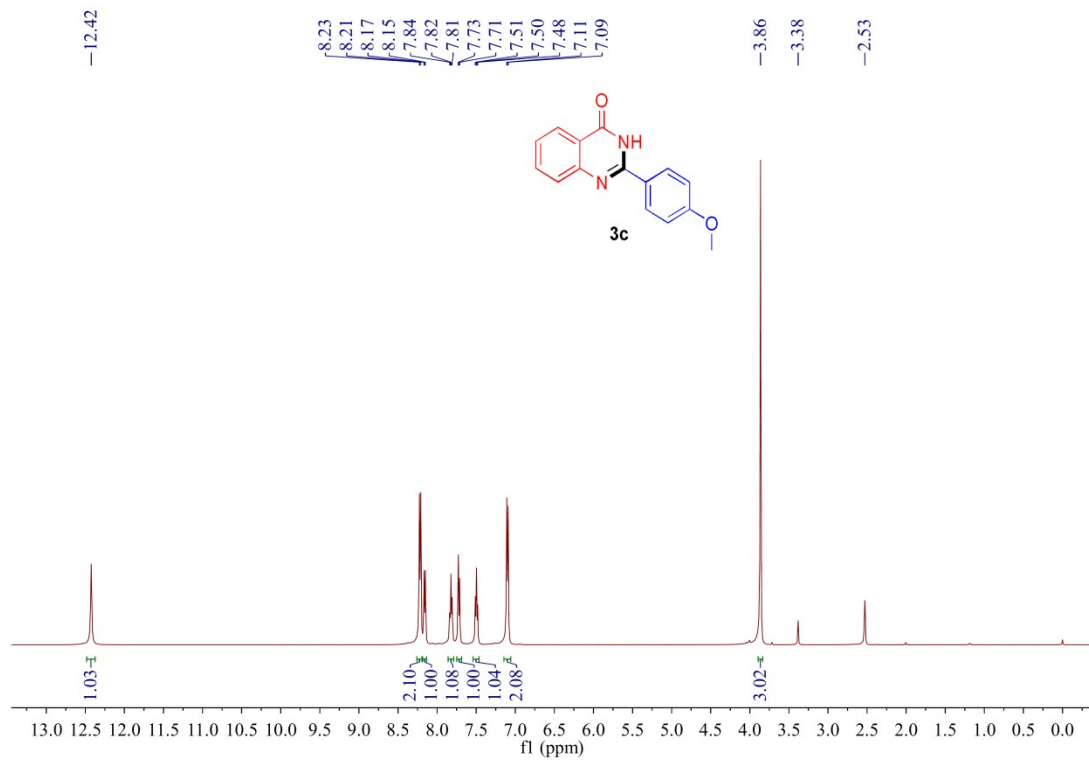
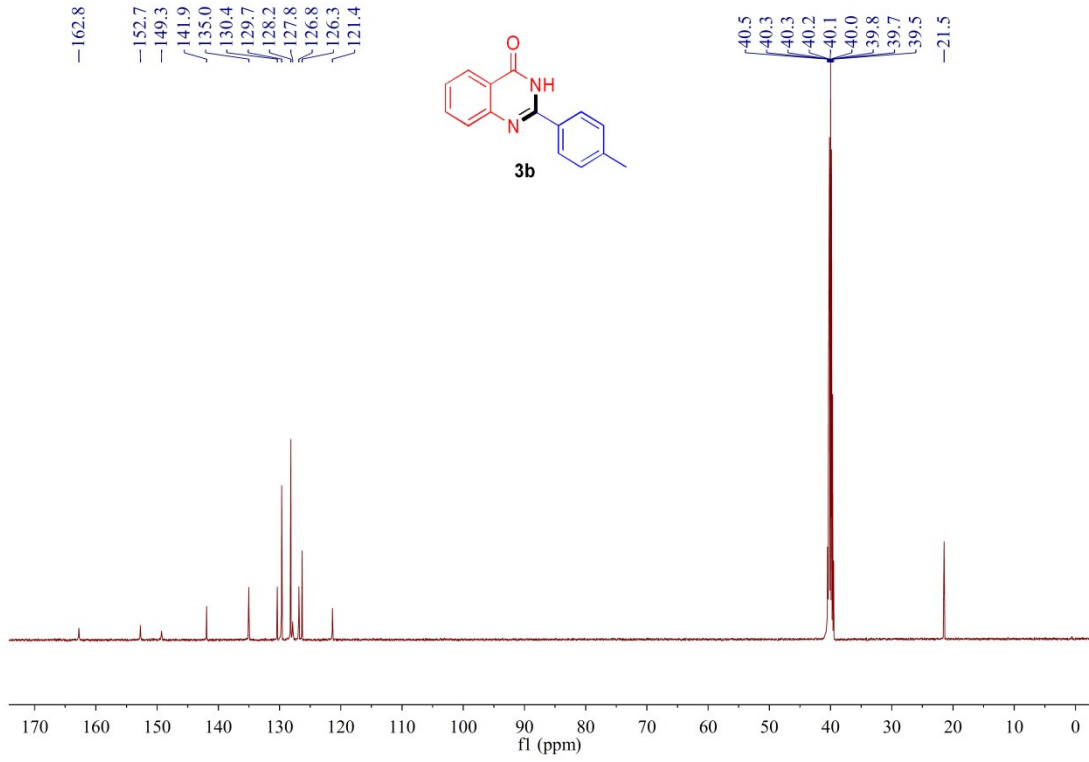
Benzophenone (3m)⁸: White solid. m.p: 48.3-49.7 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.77 – 7.73 (m, 4H), 7.70 (t, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.7 Hz, 4H); ¹³C NMR (126 MHz, DMSO) δ 196.3, 137.5, 133.2, 130.1, 129.0.

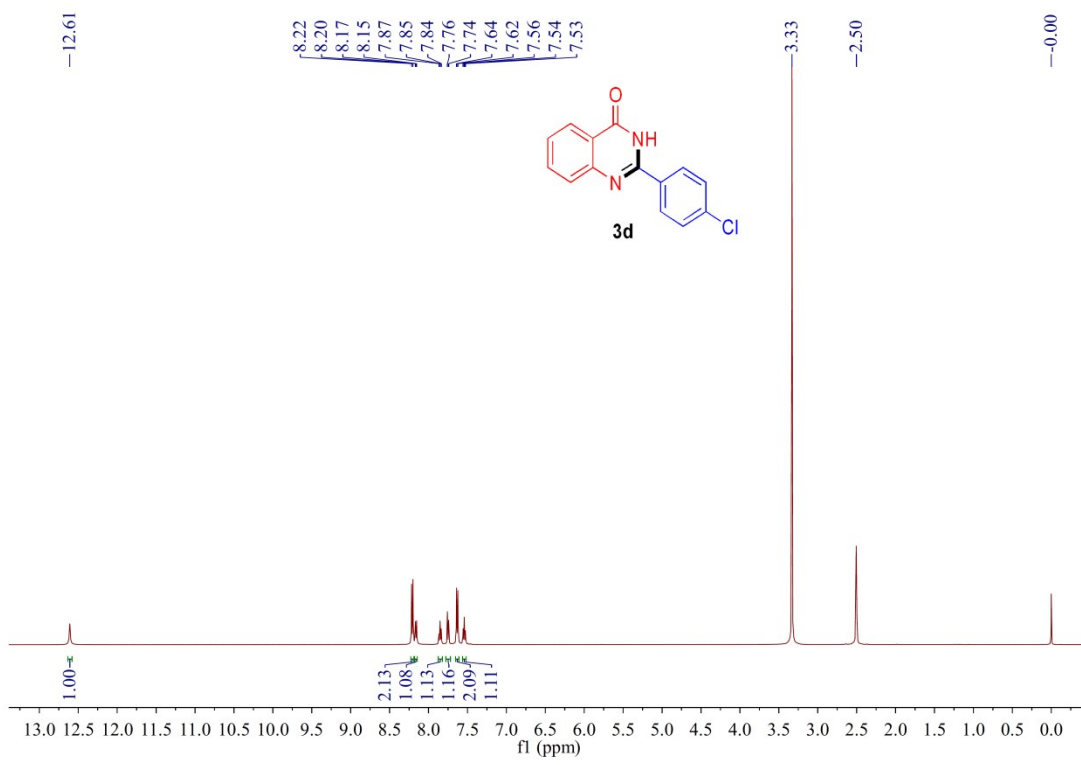
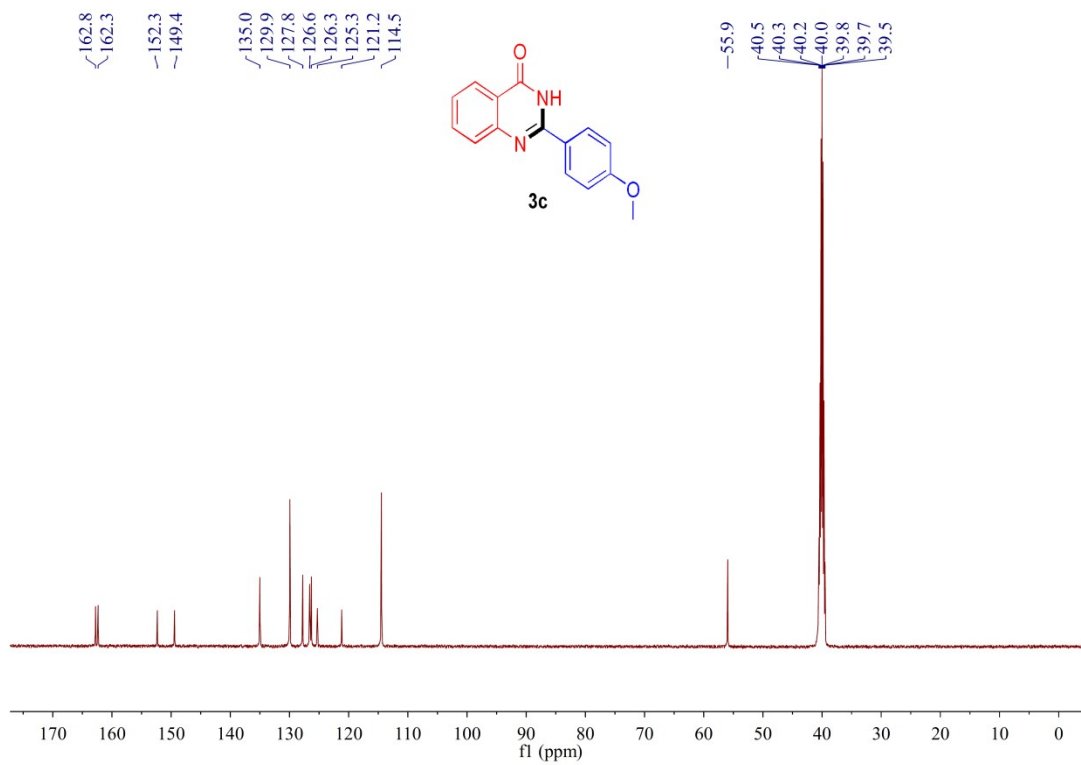
2-phenyl-2,3-dihydroquinazolin-4(1H)-one (intermediates)⁹: White solid. m.p: 225.3-227.1 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 8.27 (s, 1H), 7.60 (d, *J* = 7.7 Hz, 1H), 7.49 (d, *J* = 7.1 Hz, 2H), 7.37 (dt, *J* = 15.2, 7.0 Hz, 3H), 7.28 – 7.17 (m, 1H), 7.10 (s, 1H), 6.74 (d, *J* = 8.1 Hz, 1H), 6.67 (t, *J* = 7.5 Hz, 1H), 5.75 (s, 1H); ¹³C NMR (126 MHz, DMSO) δ 164.0, 148.3, 142.1, 133.8, 128.9, 128.8, 127.8, 127.3, 117.6, 115.4, 114.9, 67.0.

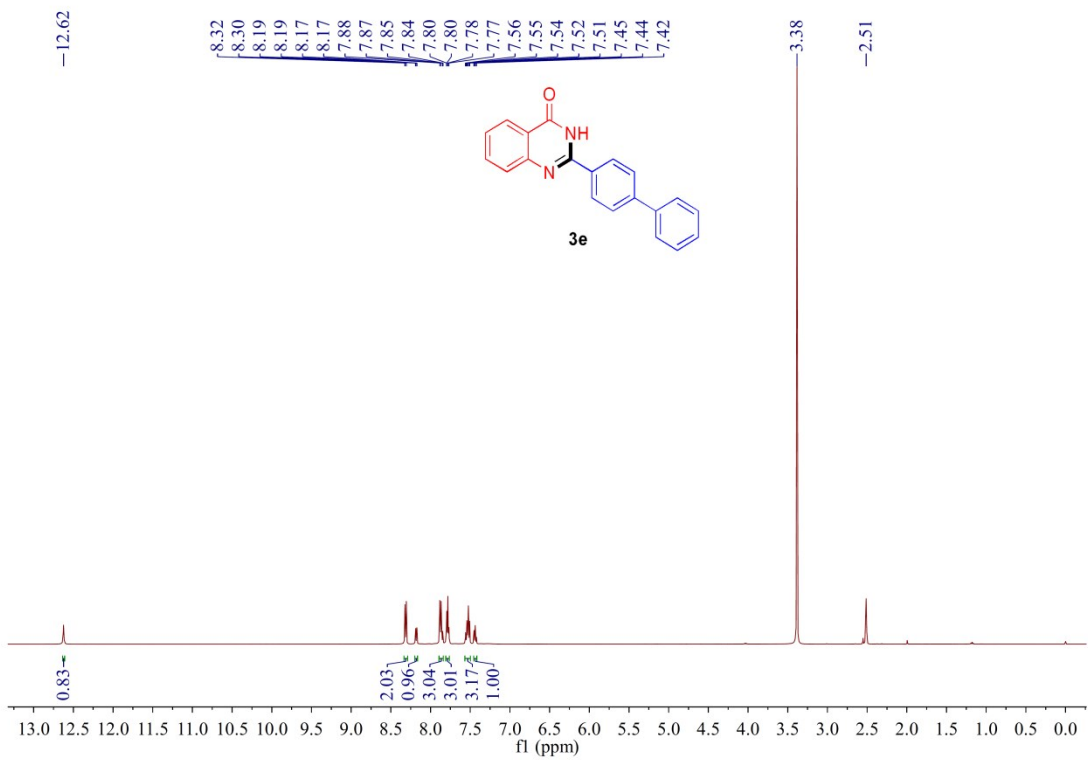
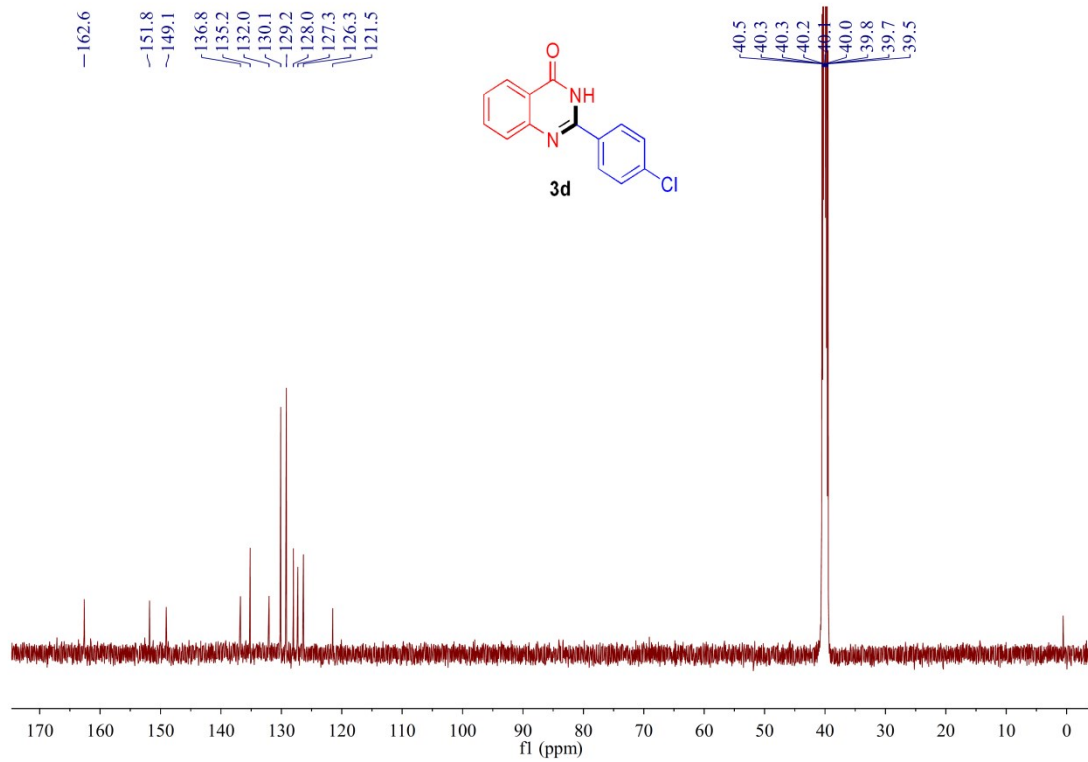
4. Spectrum

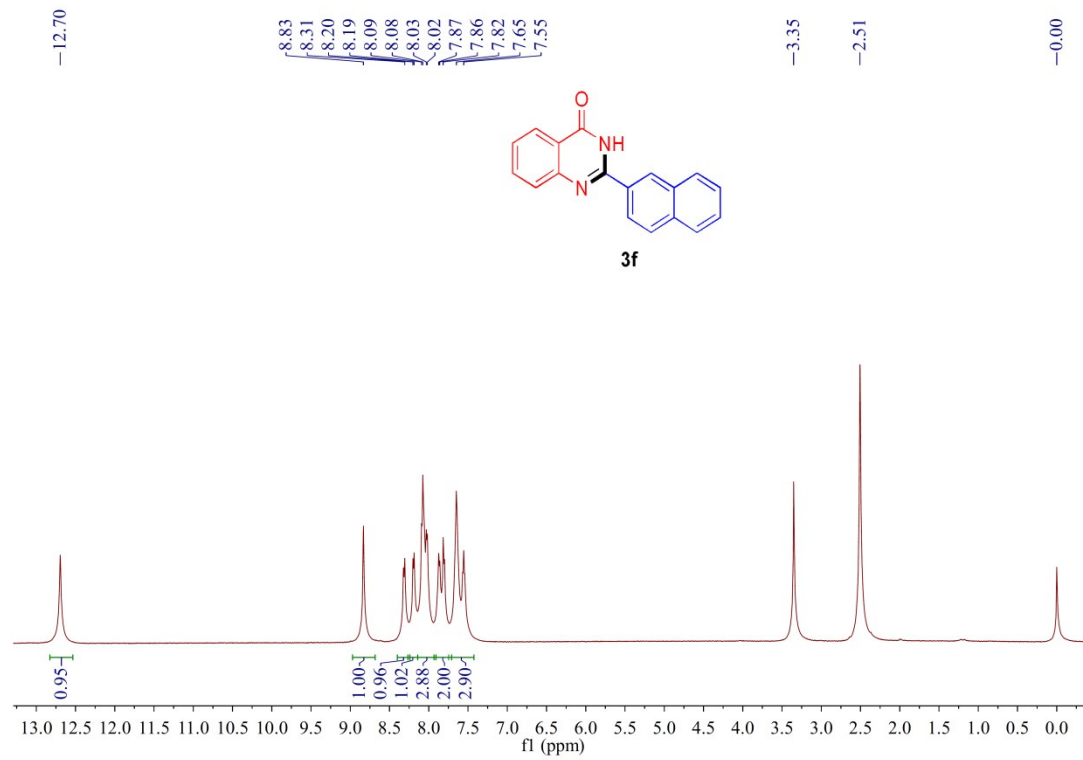
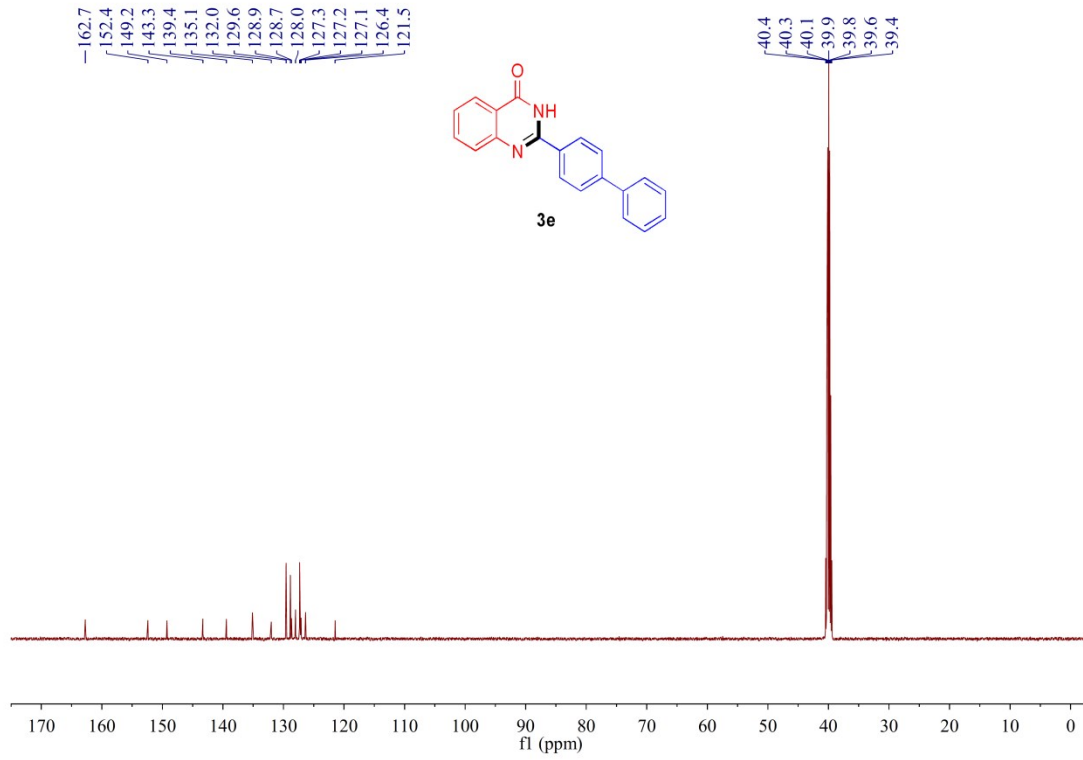


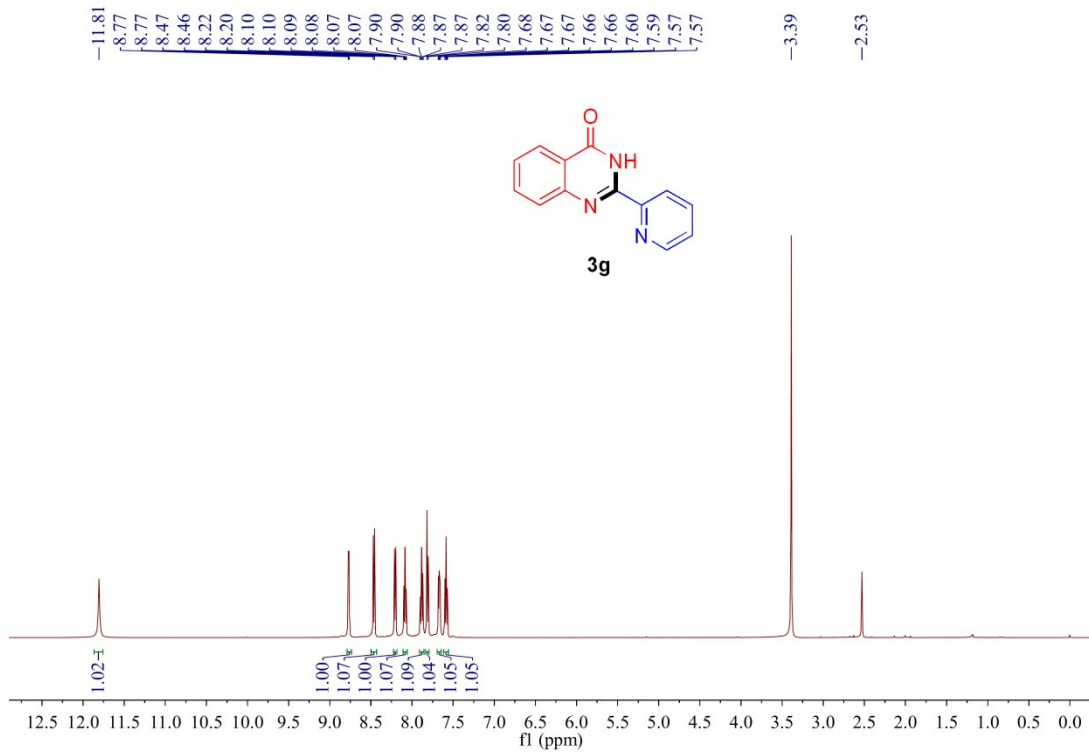
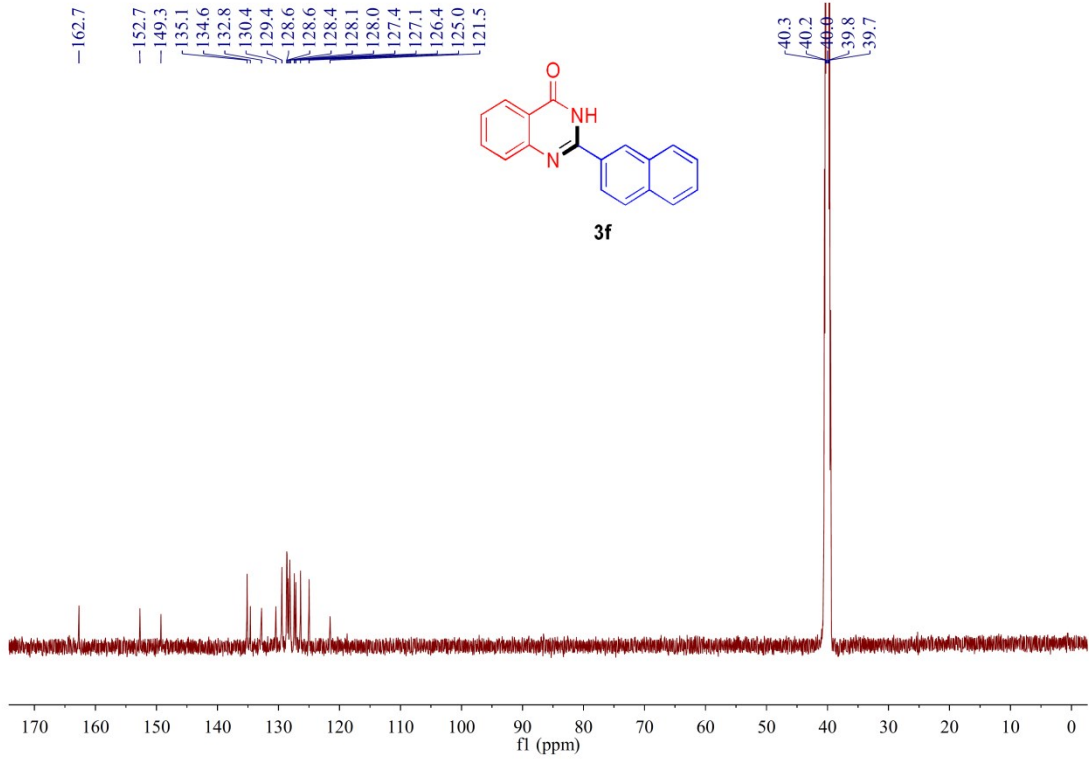


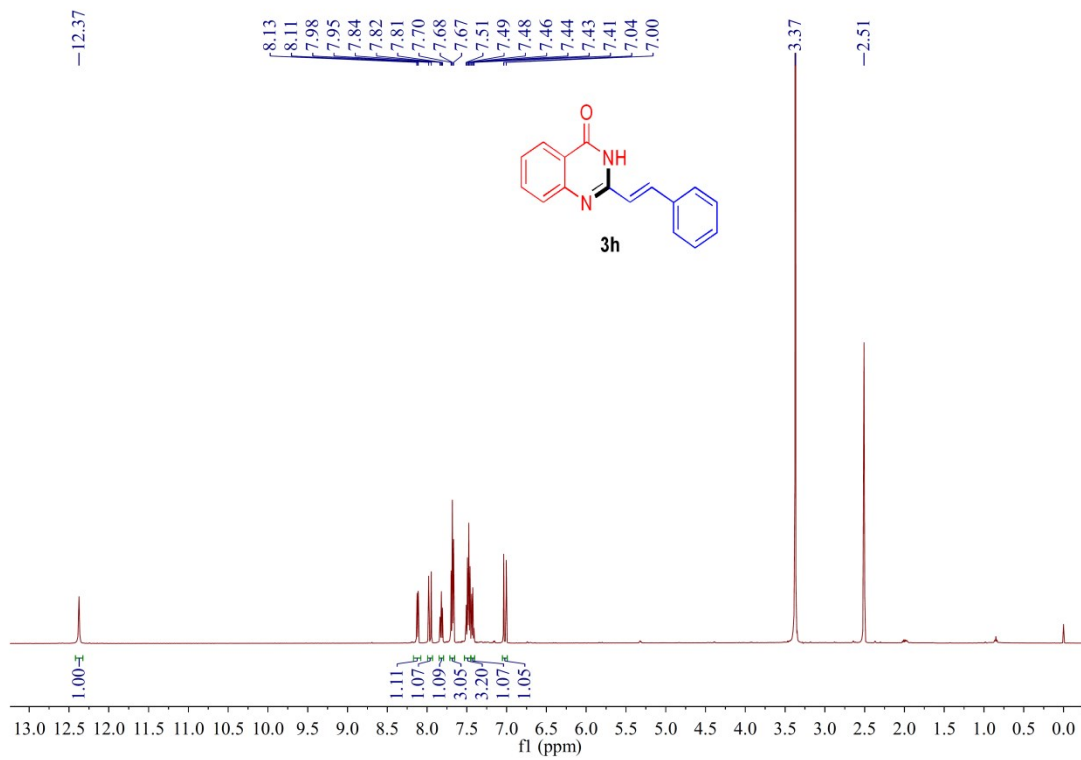
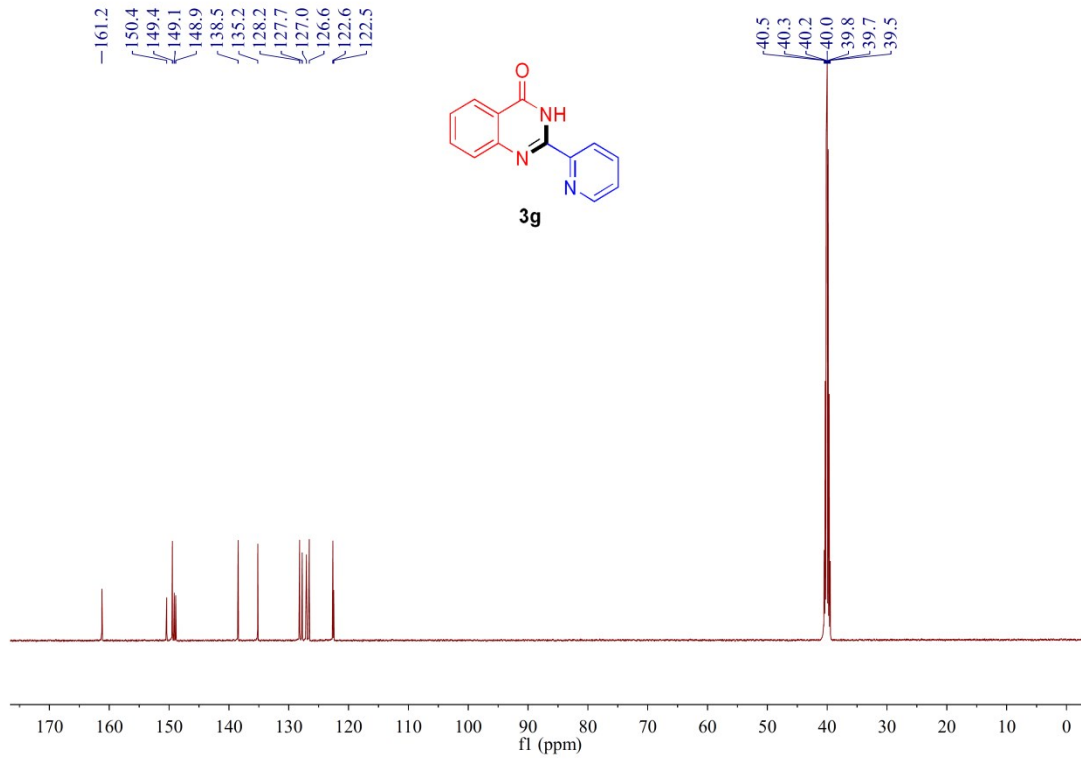


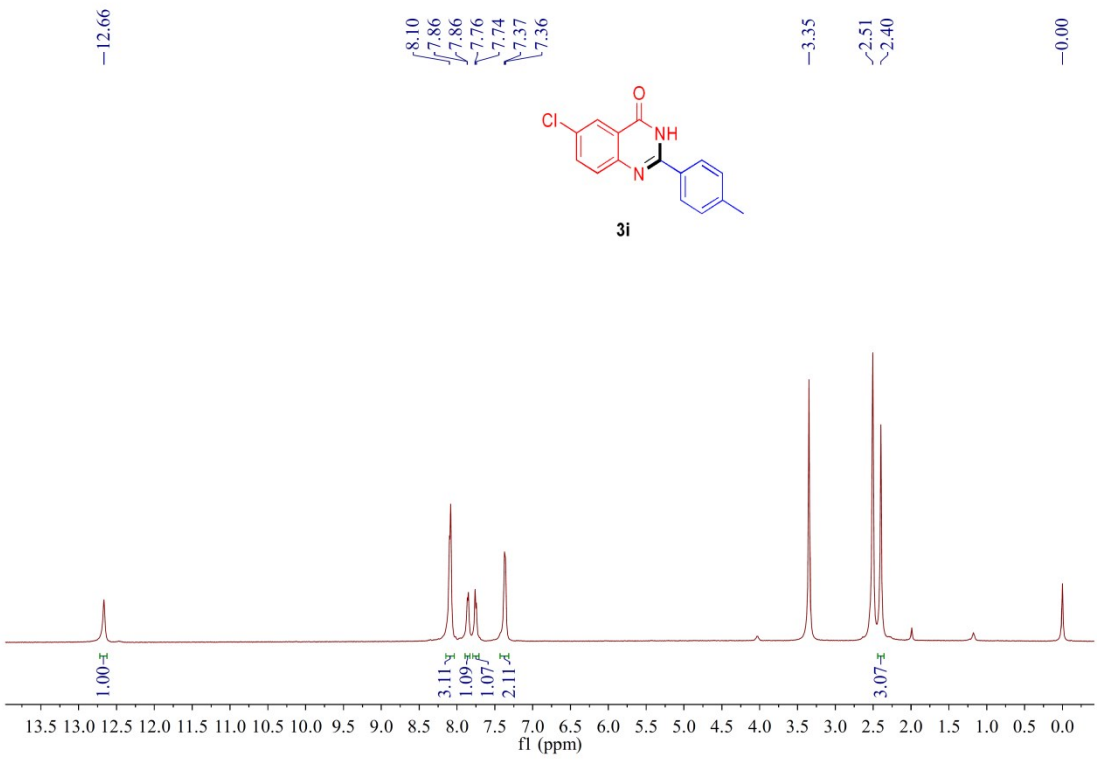
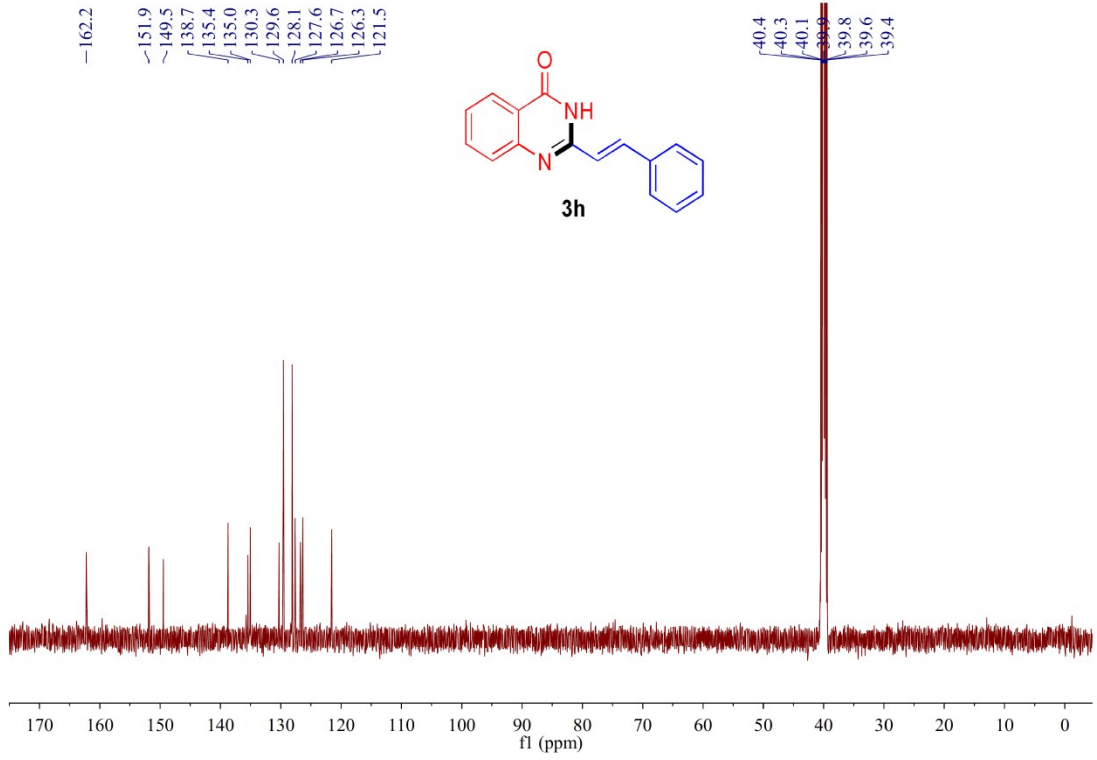


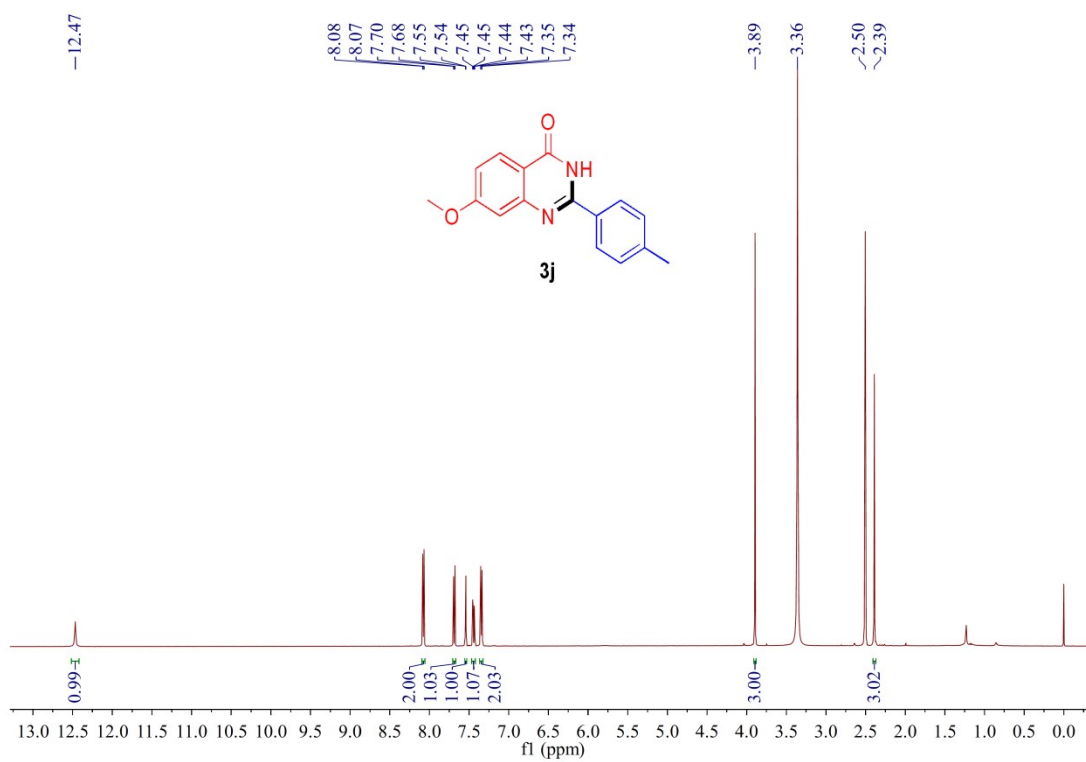
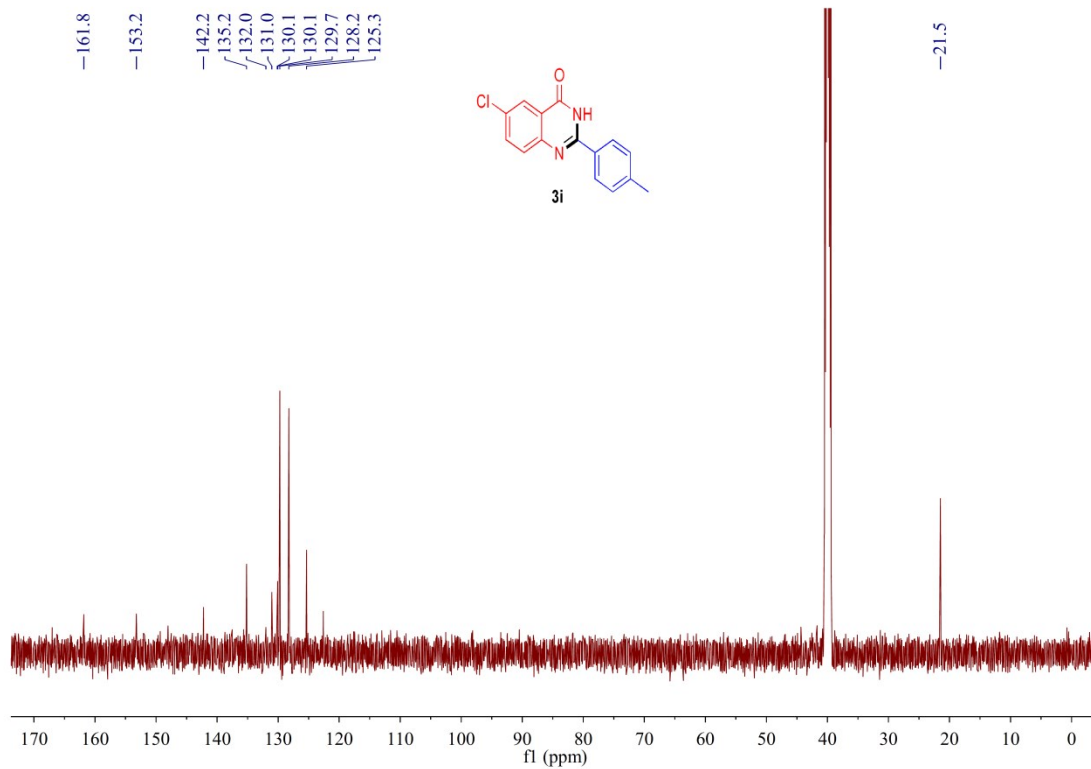


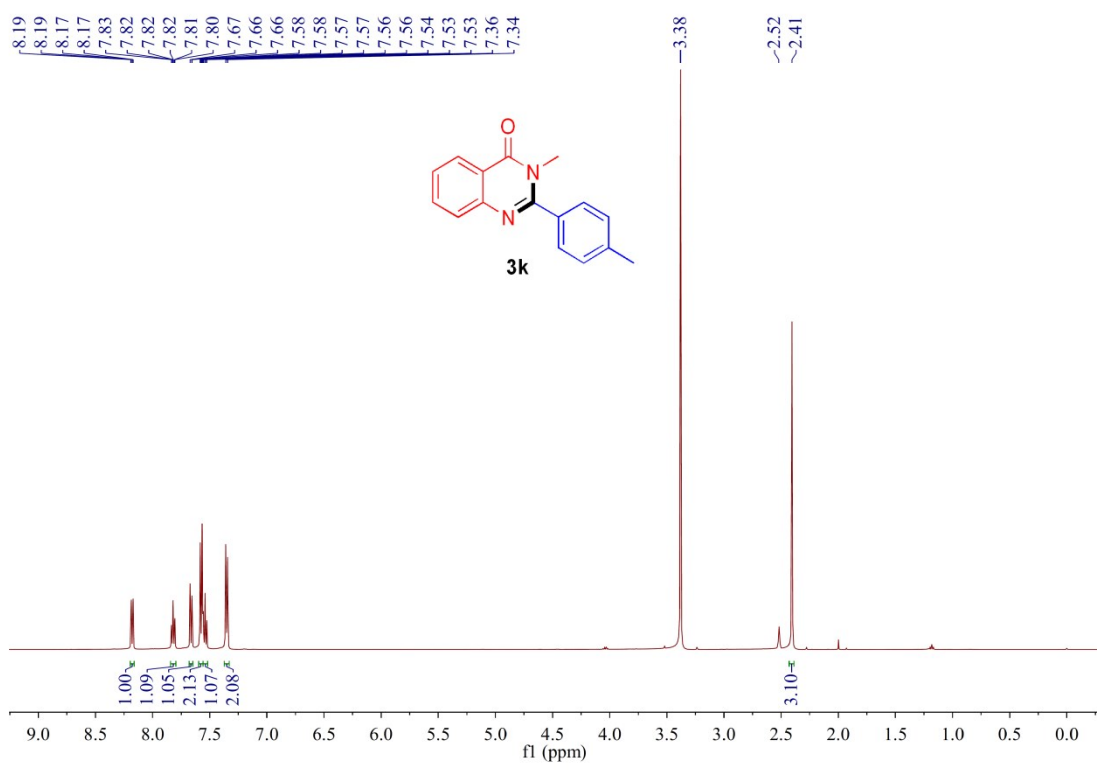
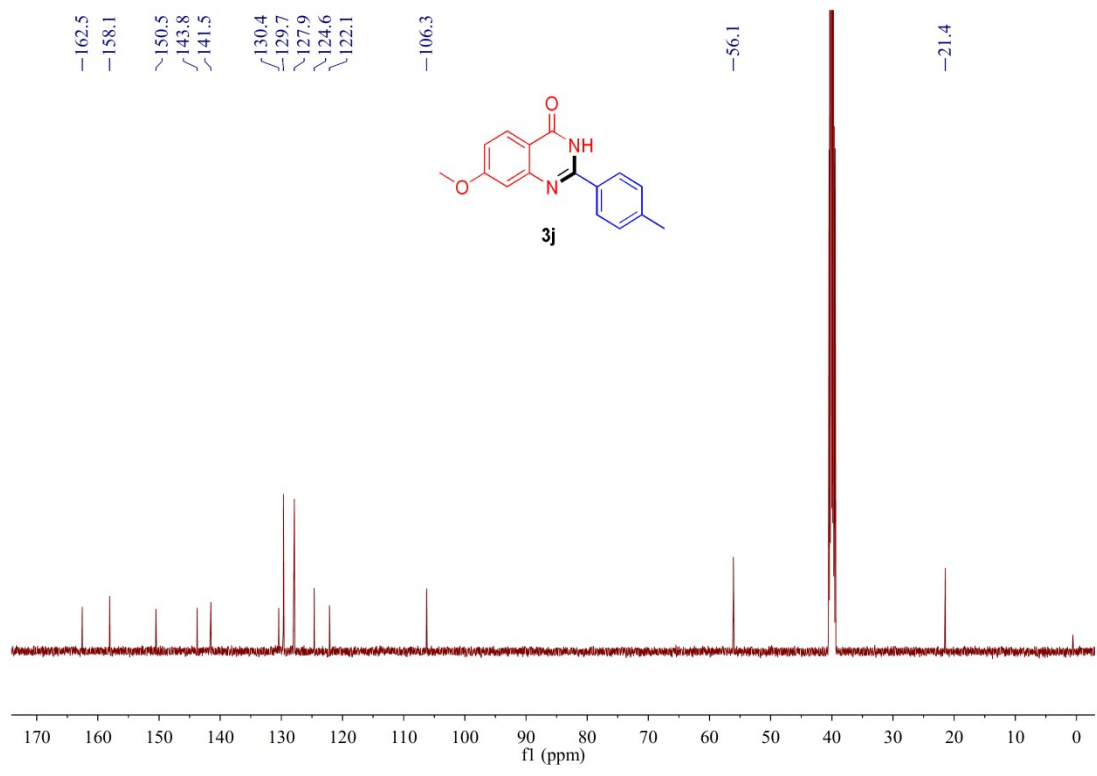


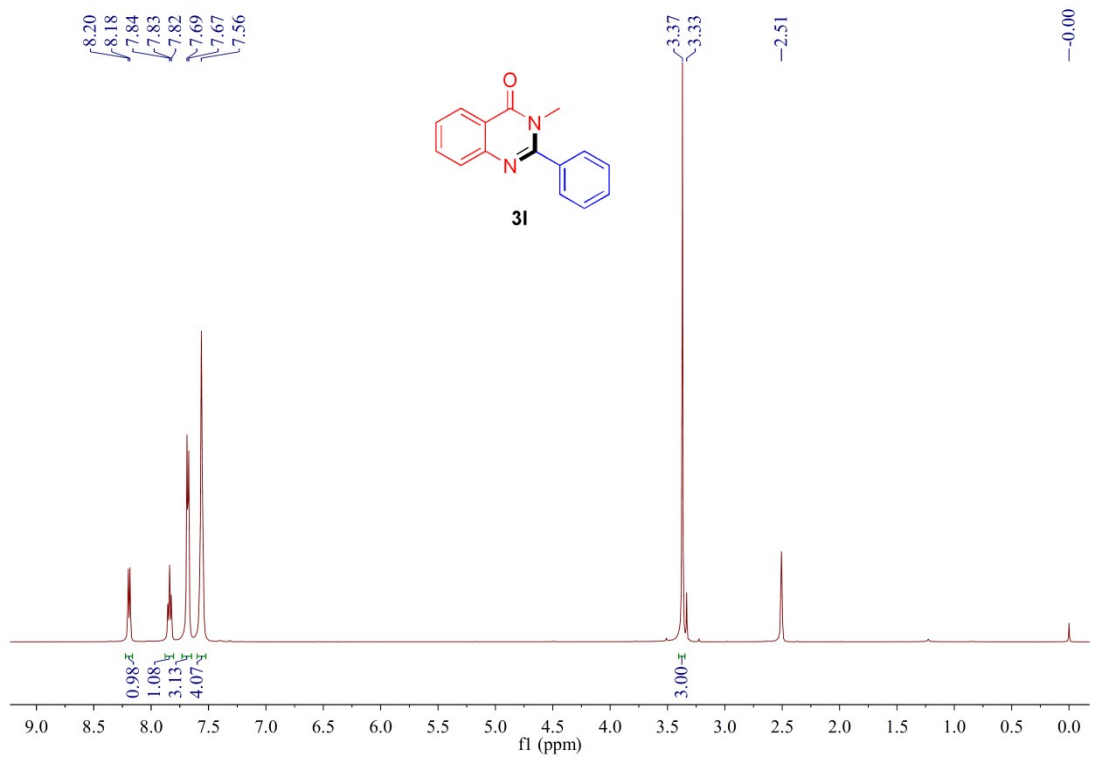
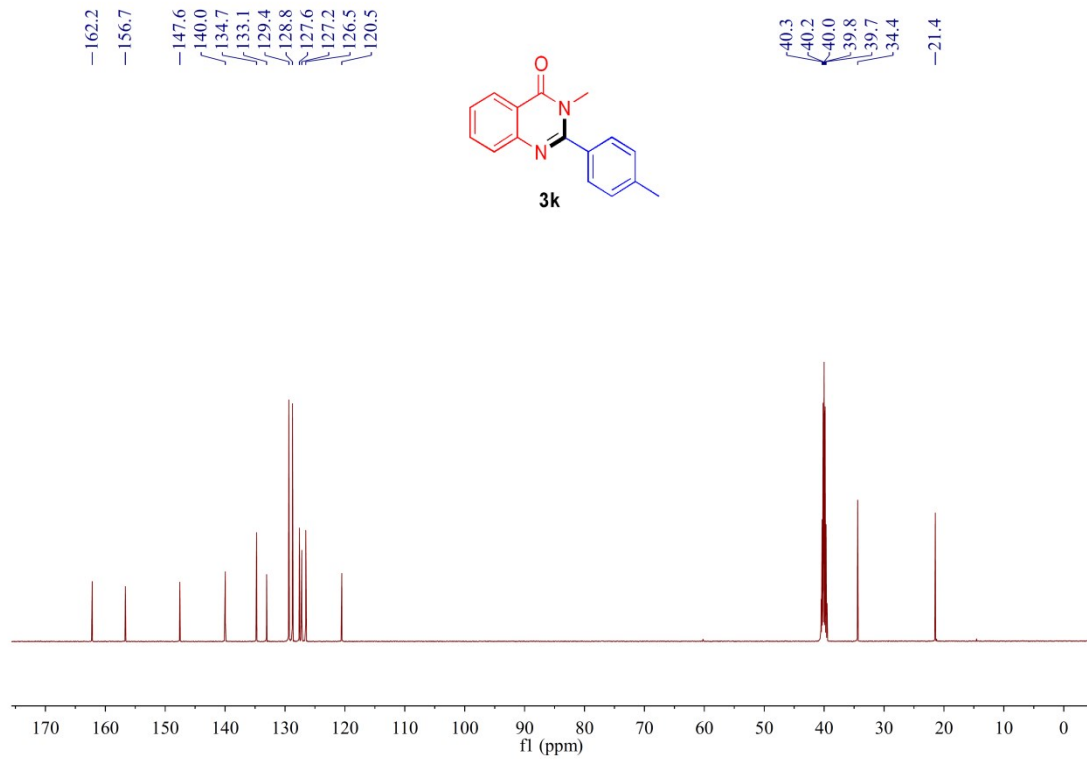


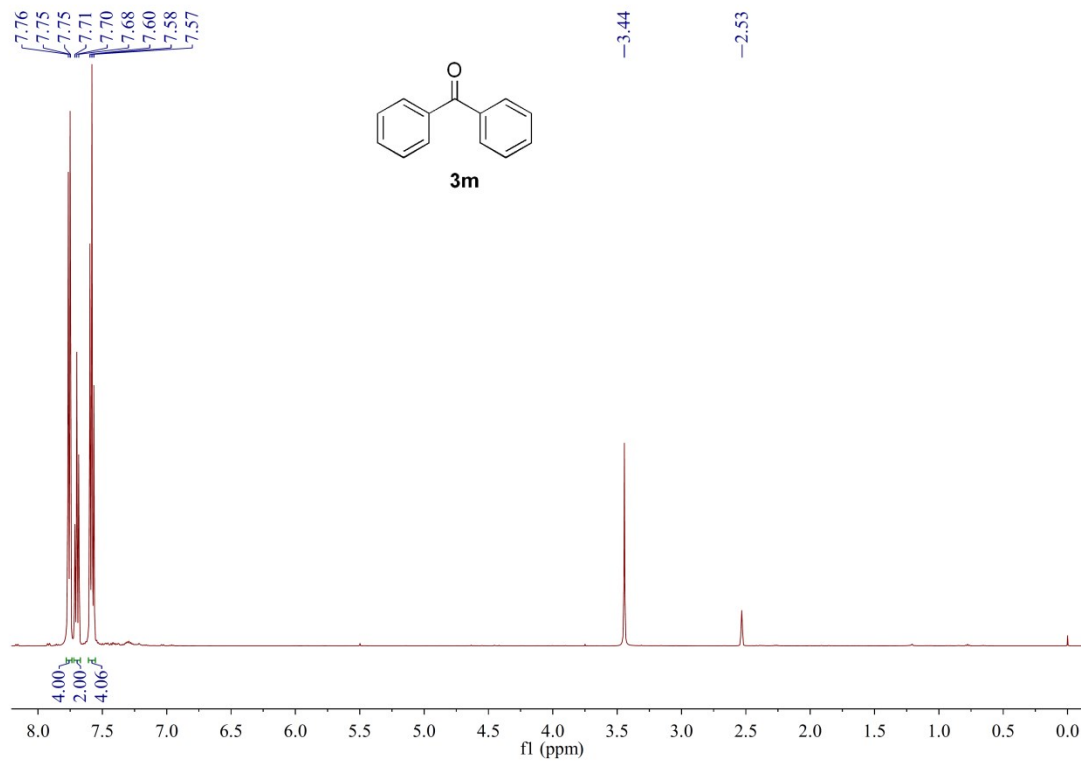
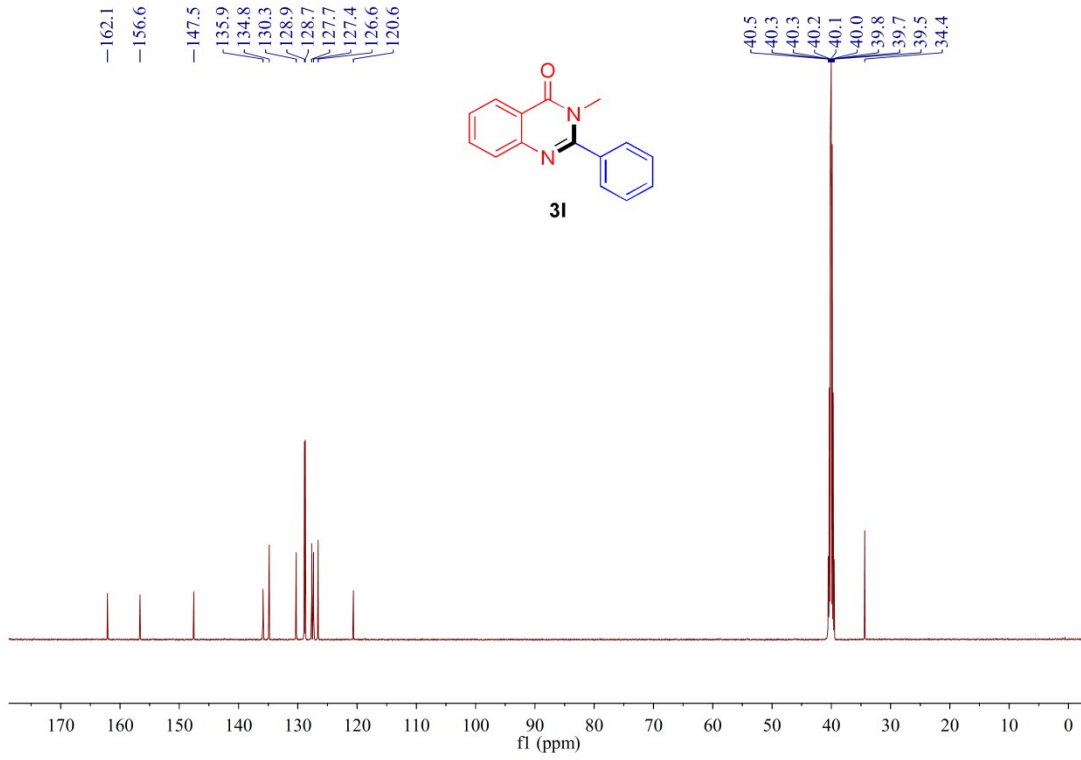


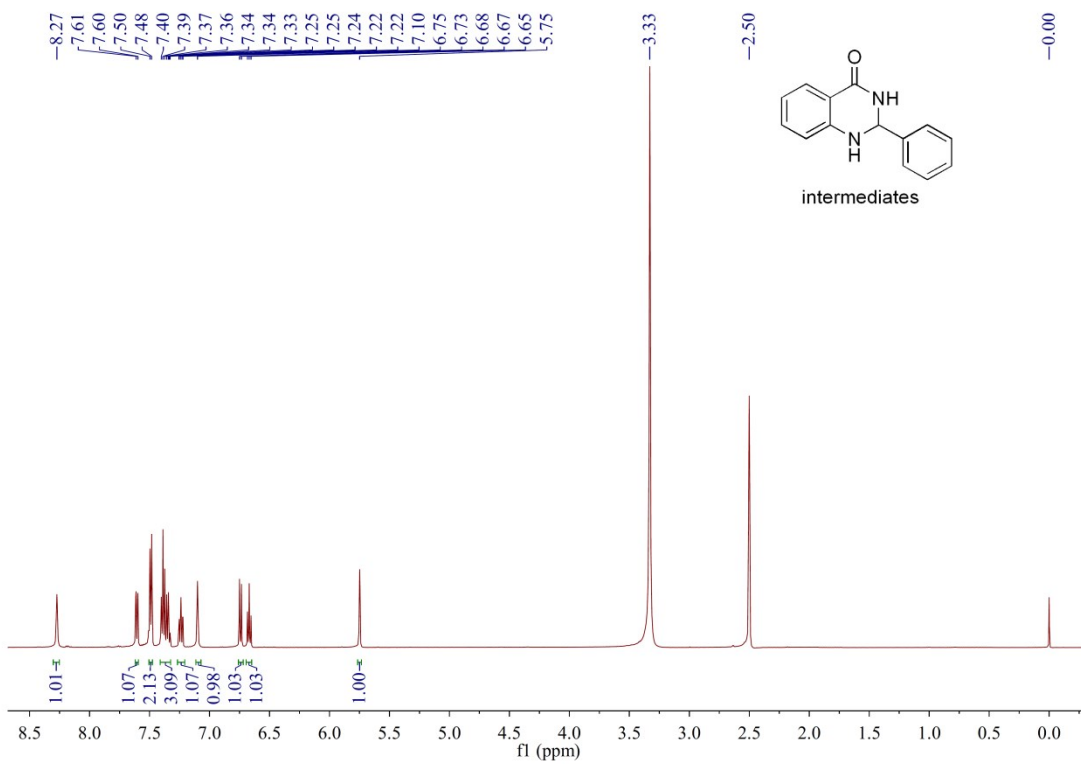
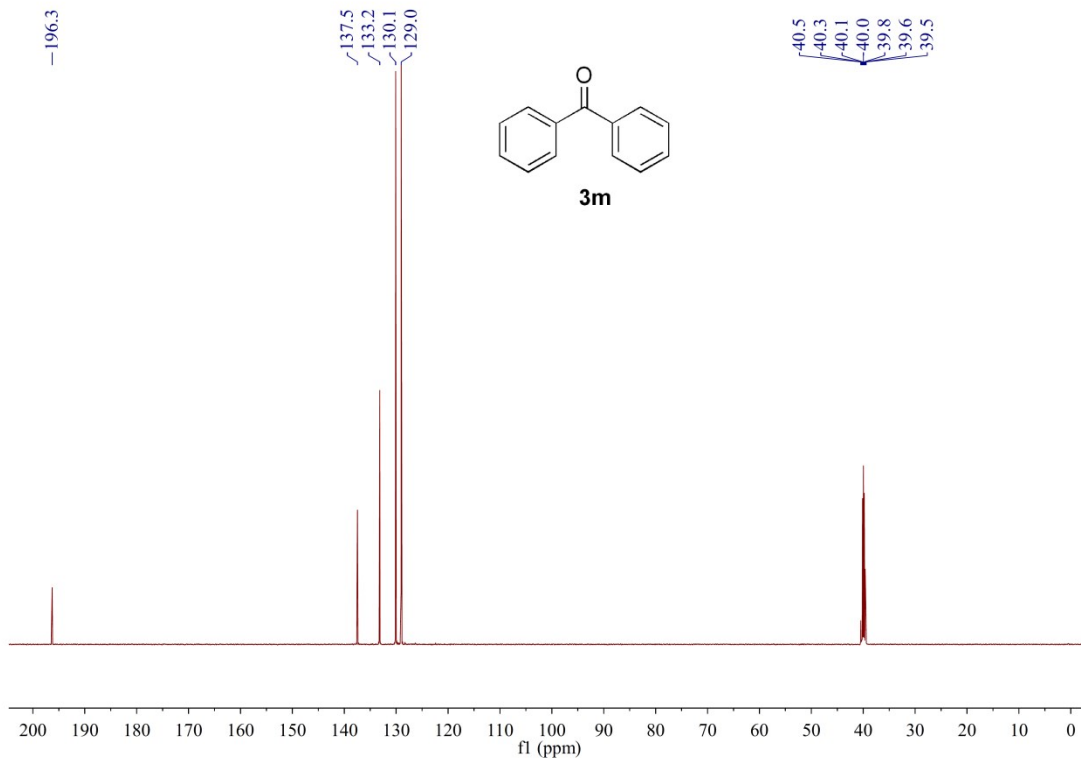


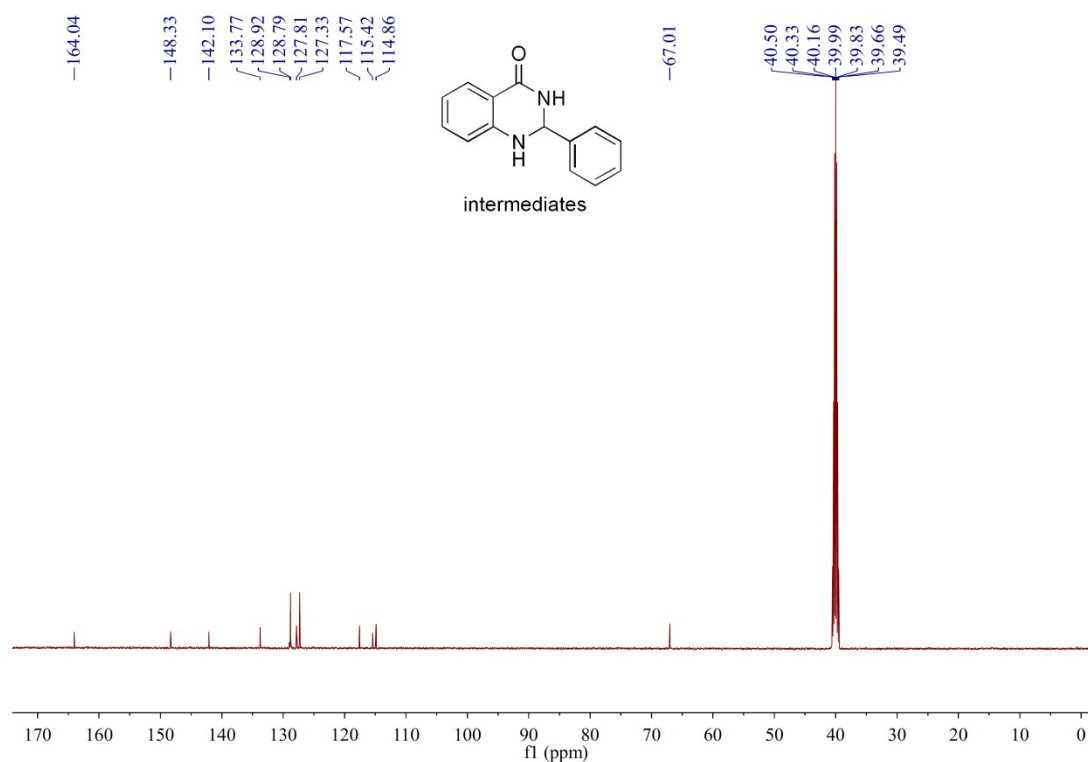




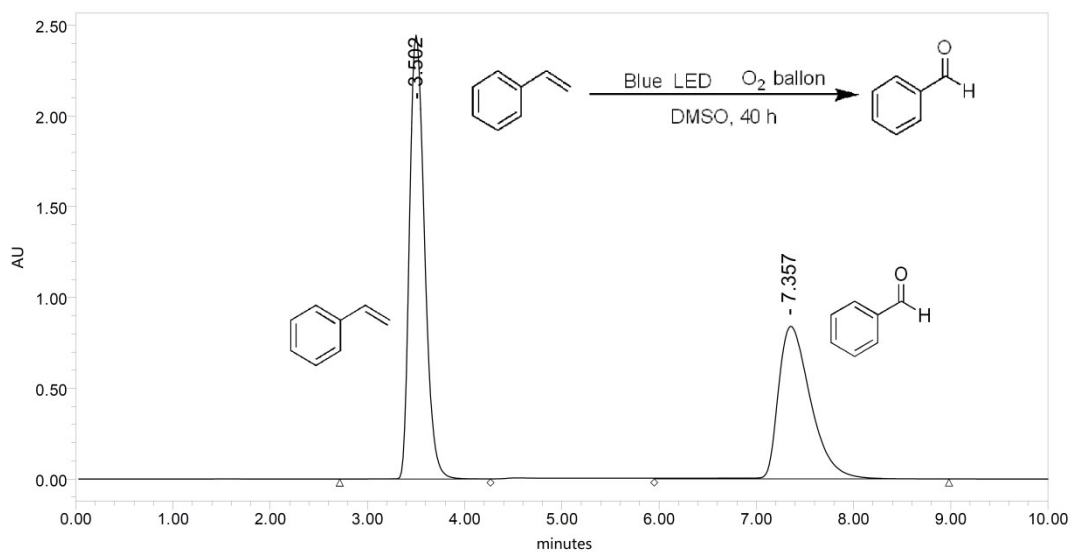








HPLC plots showing the presence of benzaldehyde



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