

Supporting Information

Synthesis of quinazoline by decarboxylation of 2-aminobenzylamine and α -keto acid under visible light catalysis

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1. Experimental section

1.1 Instruments and reagents

All major chemicals and solvents were obtained from commercial sources and used without further purification. ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance III-500 spectrometer (Swiss Bruker, Switzerland).

High-Resolution Mass Spectra (HRMS) were recorded on Thermo Scientific Q-TOF Mass Spectrometer (Bruker).

1.2 Preparation of substituted α -keto acids

All submitted α - keto acids were synthesized from oxidation of corresponding methyl ketones with SeO_2 as reported in the literature.^[1]

1.3 General Methods for the Synthesis of Quinazolines derivatives

2-Aminobenzylamine (0.2 mmol), keto acid (0.4 mmol) and Rose bengal (x mol%), were added to a 10 ml test tube dried in an oven, then DMF (2 ml) was added to the mixture, irradiated with 18 W blue LED for 16 h in an air atmosphere at room temperature, and stirred at a rotation rate of 300-400 rpm. The reaction process was monitored by TLC (petroleum ether / ethyl acetate = 6:1, v / v). After the reaction, dilute the solution with EtOAc (2 ml), wash with saturated salt water and ethyl acetate, and concentrate under reduced pressure. The product was then purified by column chromatography (PE: EtOAc = 6:1).

2. Fluorescence Quenching Experiments.

- 1) Excitation spectrum. The excitation spectrum was performed on a Shimadzu RF-540 fluorescence spectrophotometer. Rose Bengal (RB) (0.5 mmol/L) in 2 mL DMF were subjected. The result from this experiment indicated that the optimum excitation wavelength for the sample was 473 nm and the optimum emission wavelength was 602 nm (Figure 1).

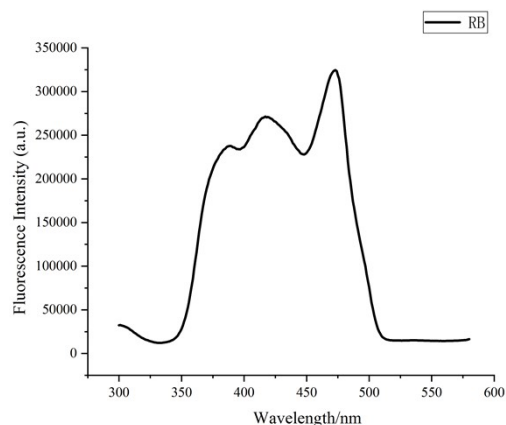


Figure S1. Excitation spectrum

- 2) The fluorescence quenching experiments of RB with 1a and 2a. The fluorescence quenching experiment was taken using a Shimadzu RF-540 fluorescence spectrophotometer. The experiments were carried out in 0.5 mmol/L of Rose Bengal/DMF at 25°C and the excitation wavelength was 473 nm. The concentrations of quenchers (2-aminobenzylamine 1a and benzoylformic acid 2a) in DMF were 0, 5 mmol/L, 10 mmol/L, 15 mmol/L, 20 mmol/L, 25 mmol/L, and 30 mmol/L.

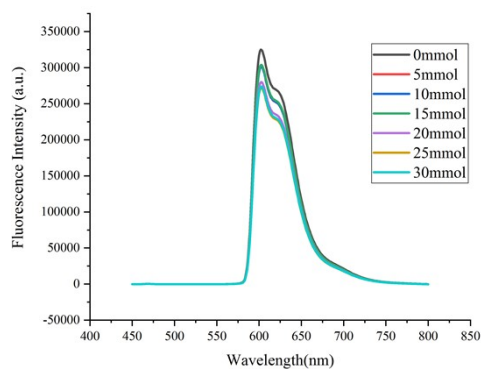


Figure S2. Stern–Volmer quenching experiment of RB in DMF by 1a

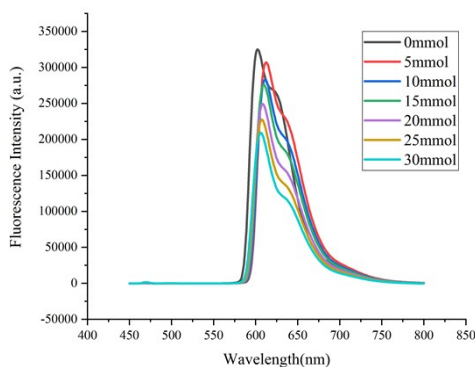


Figure S3. Stern–Volmer quenching experiment of RB in DMF by 2a

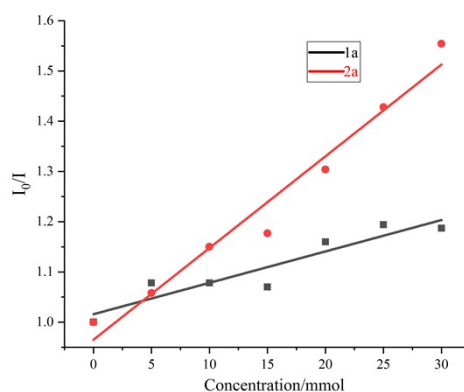


Figure S4. Stern–Volmer quenching experiment of RB in DMF by 1a and 2a

3. Cyclic Voltammetry (CV)

Cyclic voltammograms of 1a (0.1 M) and 2a (0.1 M) were performed in a three-electrode cell connected to an electrochemical cell at room temperature. The working electrode was a steady glassy carbon disk electrode while the counter electrode was a platinum wire. Ag/AgCl electrode was used as the reference electrode. A 0.1 M solution of ${}^n\text{Bu}_4\text{NBF}_4$ in DMF was added into the electrochemical cell in cyclic voltammetry experiments.

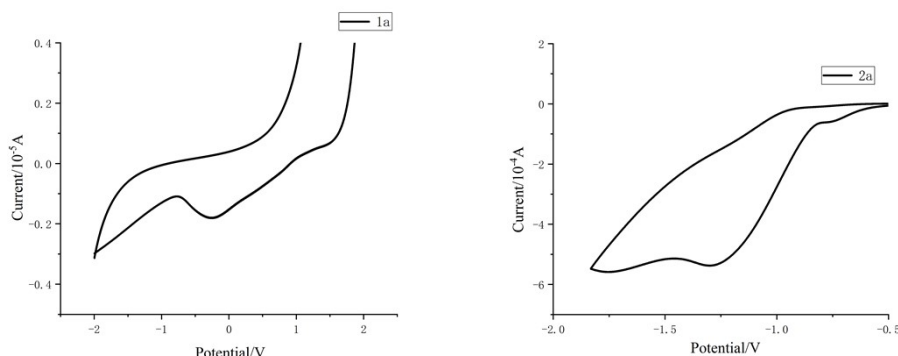


Figure S5. Cyclic voltammograms of reaction substrates.

As shown in the figure, an obvious reduction peak $E_{p2a} = -1.30\text{V}$ can be detected, which means that 2a first experienced an electron transfer process.

4. Characterization of compounds

2-Phenylquinazoline (3aa)^[2]

White solid; m.p: 100.8-101.4 °C

¹H NMR (500 MHz, CDCl₃) δ 9.47 (s, 1H), 8.62 (d, $J = 6.7$ Hz, 2H), 8.09 (d, $J = 8.4$ Hz, 1H), 7.91 (dd, $J = 13.2, 7.8$ Hz, 2H), 7.61 (t, $J = 7.5$ Hz, 1H), 7.58 – 7.51 (m, 3H).
¹³C NMR (126 MHz, CDCl₃) δ 161.04, 160.51, 150.75, 138.00, 134.13, 130.62, 128.64, 128.63, 128.56, 127.28, 127.13, 123.59.

2-(*p*-Tolyl)quinazoline (3ab)^[3]

Yellow solid; m.p: 105.3-107.4 °C

¹H NMR (500 MHz, CDCl₃) δ 9.44 (s, 1H), 8.51 (d, $J = 8.2$ Hz, 2H), 8.07 (d, $J = 8.4$ Hz, 1H), 7.89 (t, $J = 9.1$ Hz, 2H), 7.58 (t, $J = 7.1$ Hz, 1H), 7.34 (d, $J = 8.0$ Hz, 2H), 2.44 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 161.13, 160.49, 150.78, 140.94, 135.28, 134.12, 129.46, 128.56, 128.54, 127.16, 127.09, 123.52, 21.56.

2-(4-Methoxyphenyl)quinazoline (3ac)^[3]

Yellow solid; m.p: 92.0-94.0 °C.

¹H NMR (500 MHz, CDCl₃) δ 9.47 – 9.29 (m, 1H), 8.73 – 8.45 (m, 2H), 8.04 (d, $J = 8.3$ Hz, 1H), 7.88 (d, $J = 8.3$ Hz, 2H), 7.57 (d, $J = 7.7$ Hz, 1H), 7.05 (d, $J = 6.9$ Hz, 2H), 3.95 – 3.78 (m, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 161.86, 160.84, 160.46, 150.80, 134.11, 130.68, 130.25, 128.39, 127.18, 126.85, 123.32, 114.01, 55.43.

2-(3,4-Dimethoxyphenyl)quinazoline (3ad)^[3]

White solid; m.p: 107.7-113.8 °C.

¹H NMR (500 MHz, CDCl₃) δ 9.42 (s, 1H), 8.29 – 8.23 (m, 1H), 8.20 (s, 1H), 8.07 (d, *J* = 8.4 Hz, 1H), 7.89 (t, *J* = 9.6 Hz, 2H), 7.58 (t, *J* = 7.9 Hz, 1H), 7.02 (d, *J* = 8.5 Hz, 1H), 4.07 (s, 3H), 3.98 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 159.63, 159.40, 150.38, 148.01, 133.14, 129.71, 127.29, 126.15, 125.91, 122.30, 121.02, 110.02, 109.80, 55.01, 54.96.

2-(4-Fluorophenyl)quinazoline (3ae)^[2]

Yellow solid; m.p: 133.2-133.4 °C

¹H NMR (500 MHz, CDCl₃) δ 9.43 (s, 1H), 8.62 (dd, *J* = 8.5, 5.7 Hz, 2H), 8.06 (d, *J* = 8.3 Hz, 1H), 7.90 (t, *J* = 8.0 Hz, 2H), 7.60 (t, *J* = 7.5 Hz, 1H), 7.20 (t, *J* = 8.6 Hz, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 165.69, 163.70, 160.56, 160.11, 150.71, 134.28, 130.73, 130.66, 128.53, 127.33, 127.18, 123.49, 115.69, 115.51.

2-(4-Chlorophenyl)quinazoline (3af)^[2]

Yellow solid; m.p: 134.6-135.8 °C

¹H NMR (500 MHz, CDCl₃) δ 9.43 (s, 1H), 8.56 (d, *J* = 7.8 Hz, 2H), 8.06 (d, *J* = 8.3 Hz, 1H), 7.90 (t, *J* = 7.6 Hz, 2H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.49 (d, *J* = 8.5 Hz, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 160.56, 160.02, 150.67, 136.85, 136.50, 134.32, 129.92, 128.85, 128.59, 127.51, 127.19, 123.62.

2-(4-Bromophenyl)quinazoline (3ag)^[2]

White solid; m.p: 137.2-137.9 °C

¹H NMR (500 MHz, CDCl₃) δ 9.42 (s, 1H), 8.48 (d, *J* = 8.4 Hz, 2H), 8.05 (d, *J* = 8.7 Hz, 1H), 7.90 (t, *J* = 7.3 Hz, 2H), 7.66 – 7.59 (m, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 160.56, 159.99, 150.66, 136.95, 134.32, 131.81, 130.17, 128.61, 127.53, 127.19, 125.44, 123.64.

2-(3-Bromophenyl)quinazoline (3ah)^[2]

White solid; m.p: 144.0-146.4 °C

¹H NMR (500 MHz, CDCl₃) δ 9.46 (s, 1H), 8.78 (t, *J* = 1.6 Hz, 1H), 8.55 (d, *J* = 7.8 Hz, 1H), 8.09 (d, *J* = 8.5 Hz, 1H), 7.93 (d, *J* = 7.4 Hz, 2H), 7.63 (d, *J* = 8.0 Hz, 2H), 7.40 (t, *J* = 7.9 Hz, 1H).

¹³C NMR (126 MHz, CDCl₃) δ 160.61, 159.52, 150.62, 139.99, 134.45, 133.53, 131.60, 130.19, 128.66, 127.75, 127.23, 127.13, 123.75, 122.98.

2-(4-(Trifluoromethyl)phenyl)quinazoline (3ai)^[2]

Yellow solid; m.p: 128.6-130.4 °C

¹H NMR (500 MHz, CDCl₃) δ 9.50 (s, 1H), 8.75 (d, *J* = 8.2 Hz, 2H), 8.12 (d, *J* = 8.4 Hz, 1H), 7.96 (dd, *J* = 11.9, 8.2 Hz, 2H), 7.79 (d, *J* = 8.2 Hz, 2H), 7.67 (t, *J* = 7.5 Hz, 1H).

^{13}C NMR (126 MHz, CDCl_3) δ 160.70, 159.62, 150.67, 141.28, 134.48, 132.02, 128.86, 128.78, 127.94, 127.24, 125.57, 125.55, 123.86.

2-([1,1'-Biphenyl]-4-yl)quinazoline (3aj)^[2]

White solid; m.p. 143.2-144.2 °C

^1H NMR (500 MHz, CDCl_3) δ 9.46 (s, 1H), 8.69 (d, $J = 8.4$ Hz, 2H), 8.10 (d, $J = 8.3$ Hz, 1H), 7.94 – 7.86 (m, 2H), 7.78 (d, $J = 8.4$ Hz, 2H), 7.73 – 7.66 (m, 2H), 7.59 (s, 1H), 7.47 (t, $J = 7.6$ Hz, 2H), 7.39 (d, $J = 7.3$ Hz, 1H).

^{13}C NMR (126 MHz, CDCl_3) δ 160.76, 160.57, 150.76, 143.32, 140.61, 136.89, 134.27, 129.11, 128.89, 128.61, 127.74, 127.40, 127.34, 127.24, 123.61.

2-(Naphthalen-2-yl)quinazoline (3ak)^[2]

White solid; m.p: 138.8-139.7 °C

^1H NMR (500 MHz, CDCl_3) δ 9.51 (s, 1H), 9.16 (s, 1H), 8.73 (dd, $J = 8.6, 1.7$ Hz, 1H), 8.14 (d, $J = 8.4$ Hz, 1H), 8.07 – 8.02 (m, 1H), 8.00 (d, $J = 8.6$ Hz, 1H), 7.96 – 7.87 (m, 3H), 7.62 (t, $J = 7.9$ Hz, 1H), 7.59 – 7.50 (m, 2H).

^{13}C NMR (126 MHz, CDCl_3) δ 160.99, 160.58, 150.81, 135.30, 134.72, 134.30, 133.44, 129.33, 129.03, 128.64, 128.35, 127.76, 127.40, 127.24, 127.17, 126.29, 125.43, 123.65, 77.32, 77.06, 76.81.

2-(Thiophen-2-yl)quinazoline (3al)^[2]

White solid; m.p: 132.7-138.8 °C

^1H NMR (500 MHz, CDCl_3) δ 9.35 (s, 1H), 8.16 (d, $J = 3.7$ Hz, 1H), 8.01 (d, $J = 8.8$ Hz, 1H), 7.92 – 7.83 (m, 2H), 7.60 – 7.50 (m, 2H), 7.22 – 7.17 (m, 1H).

^{13}C NMR (126 MHz, CDCl_3) δ 160.61, 157.83, 150.58, 143.78, 134.45, 130.04, 129.32, 128.46, 128.16, 127.32, 127.07, 123.38.

2-Phenylquinazolin-4(3H)-one (3ba)^[4]

White solid; m.p: 235.6-236.8 °C

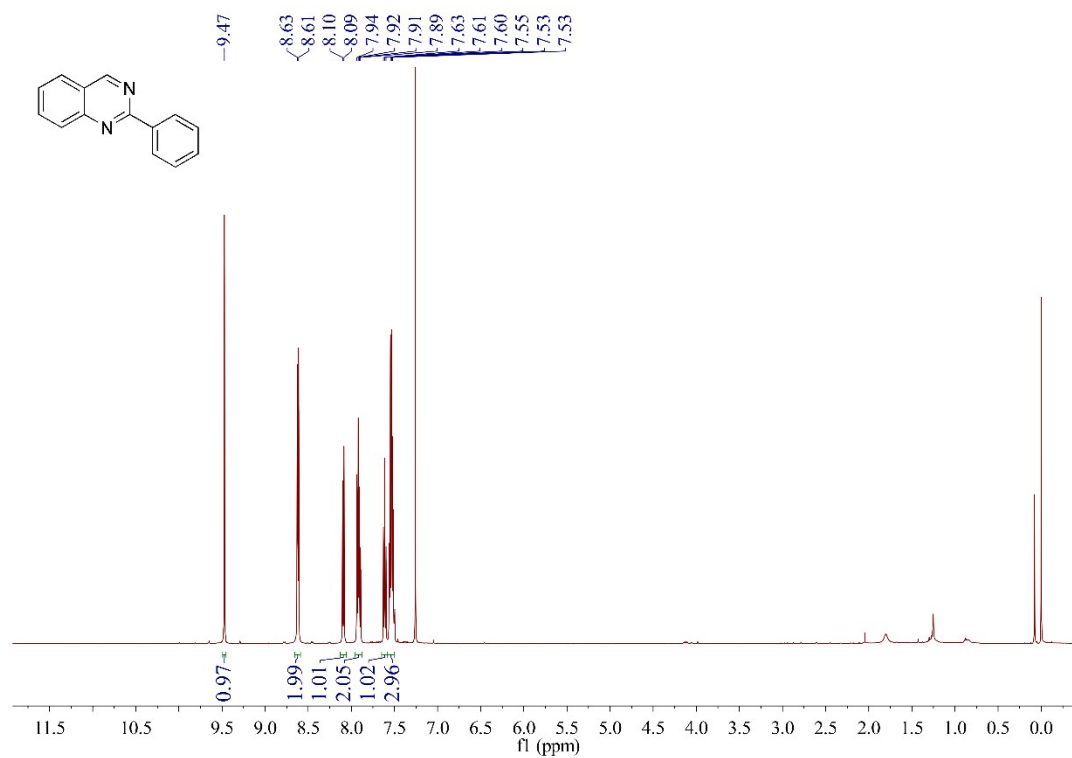
^1H NMR (400 MHz, DMSO) δ 12.57 (s, 1H), 8.19 (t, $J = 8.5$ Hz, 3H), 7.85 (d, $J = 7.0$ Hz, 1H), 7.76 (d, $J = 7.9$ Hz, 1H), 7.63 – 7.50 (m, 4H).

^{13}C NMR (101 MHz, DMSO) δ 162.74, 152.85, 149.10, 135.05, 133.18, 131.86, 129.72, 129.06, 128.25, 127.86, 127.04, 126.33, 121.45.

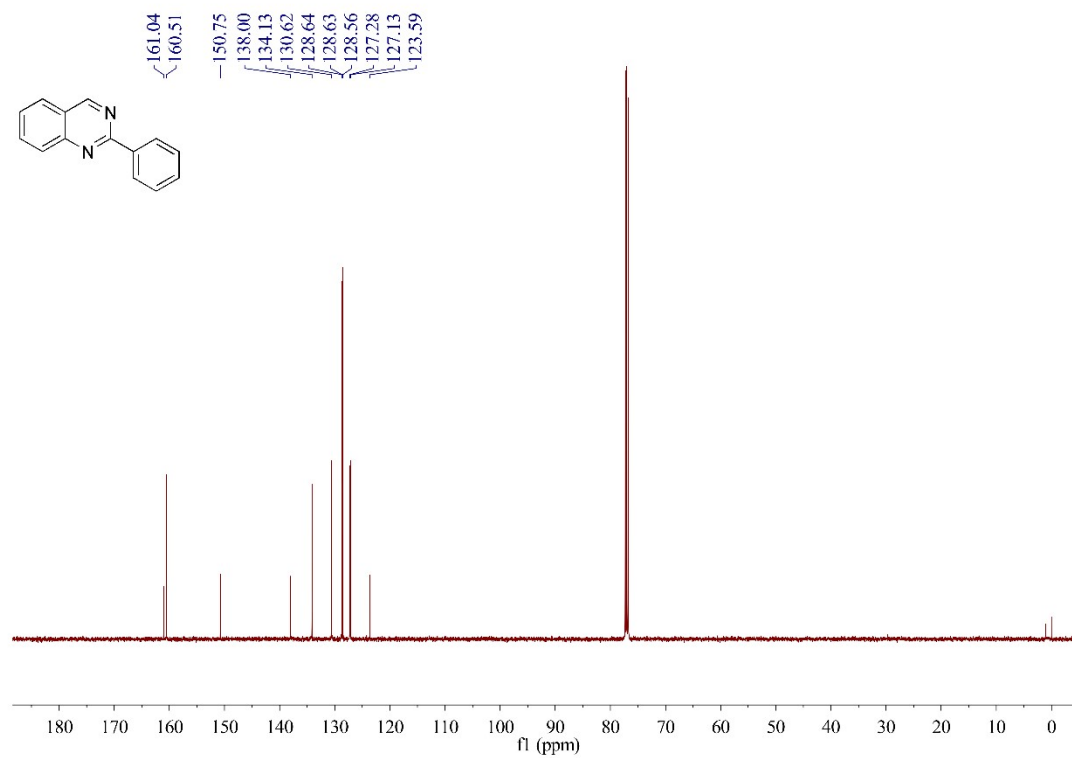
HRMS: m/z calcd for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_2$ $[\text{M} + \text{H}]^+$: 255.11280; found: 255.11215.

5. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra for target products

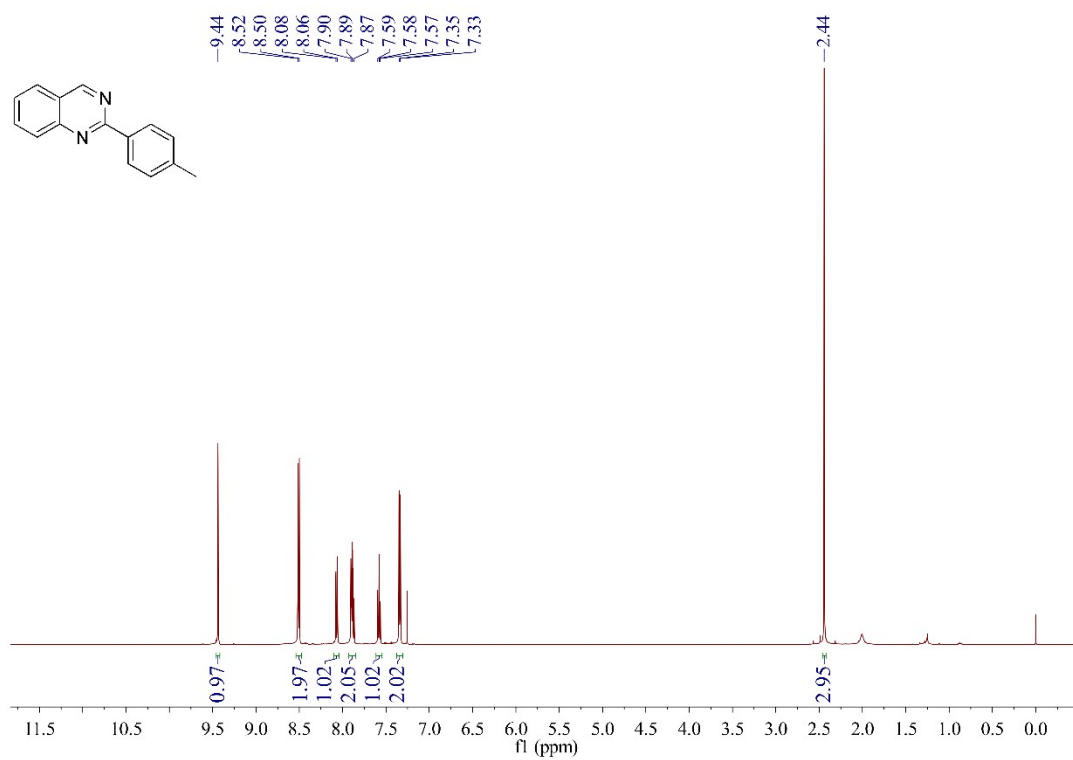
$^1\text{H-NMR}$ spectra of compound **3aa**



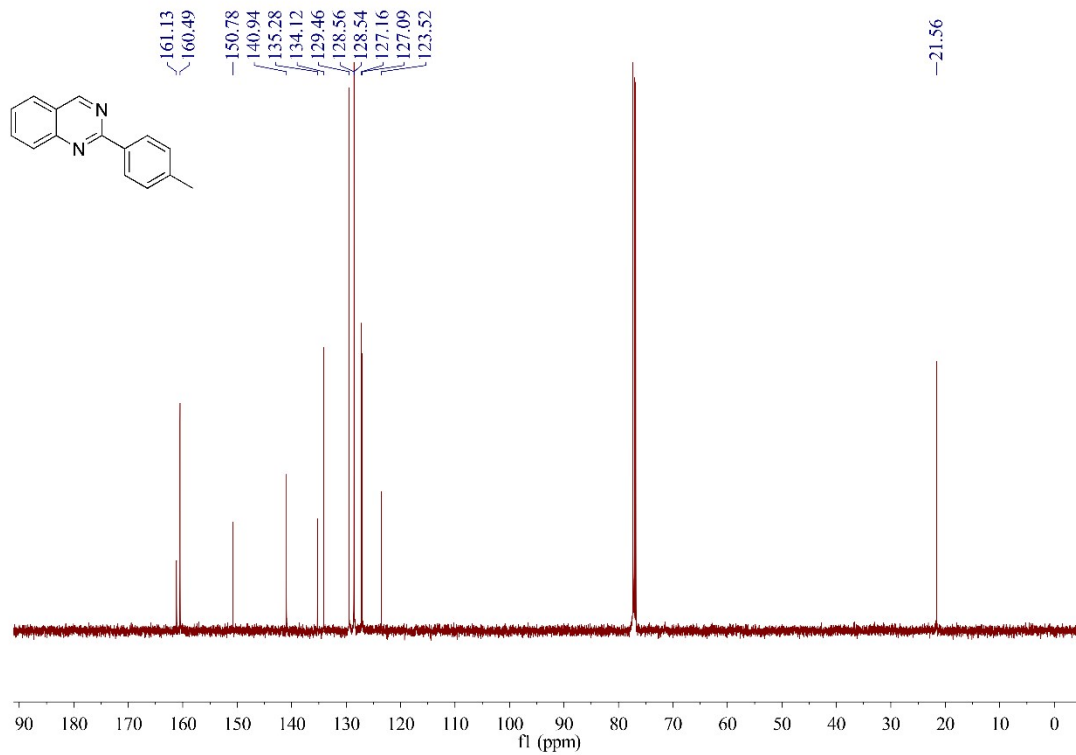
$^{13}\text{C-NMR}$ spectra of compound **3aa**



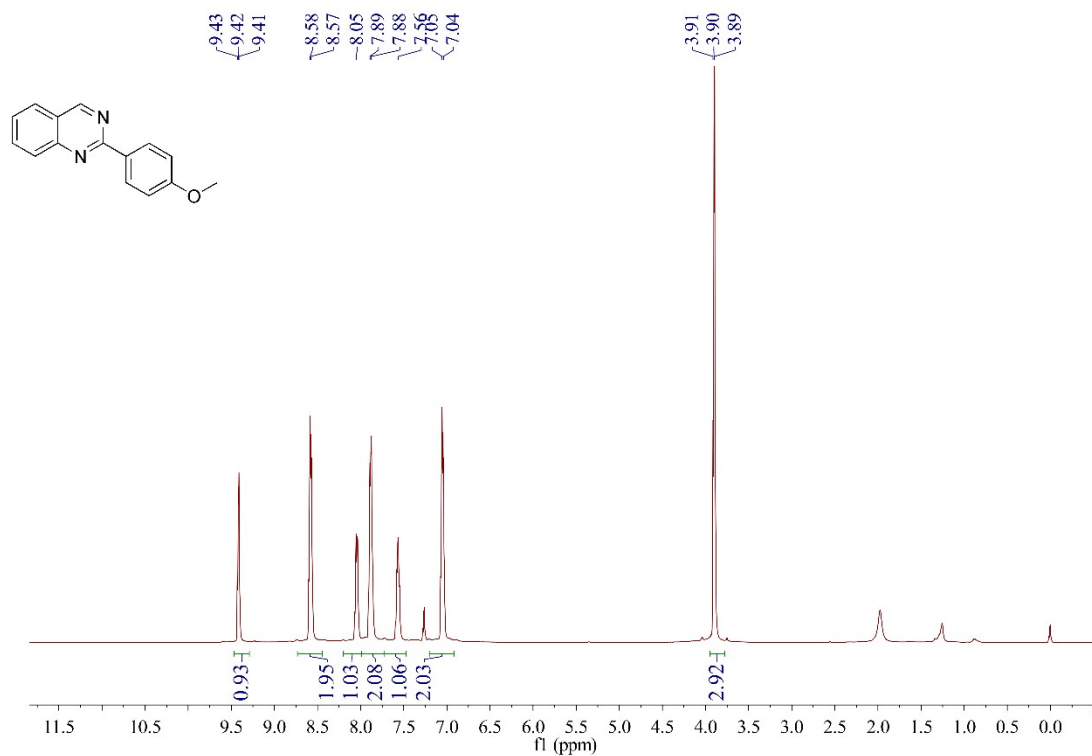
^1H NMR spectra of compound **3ab**



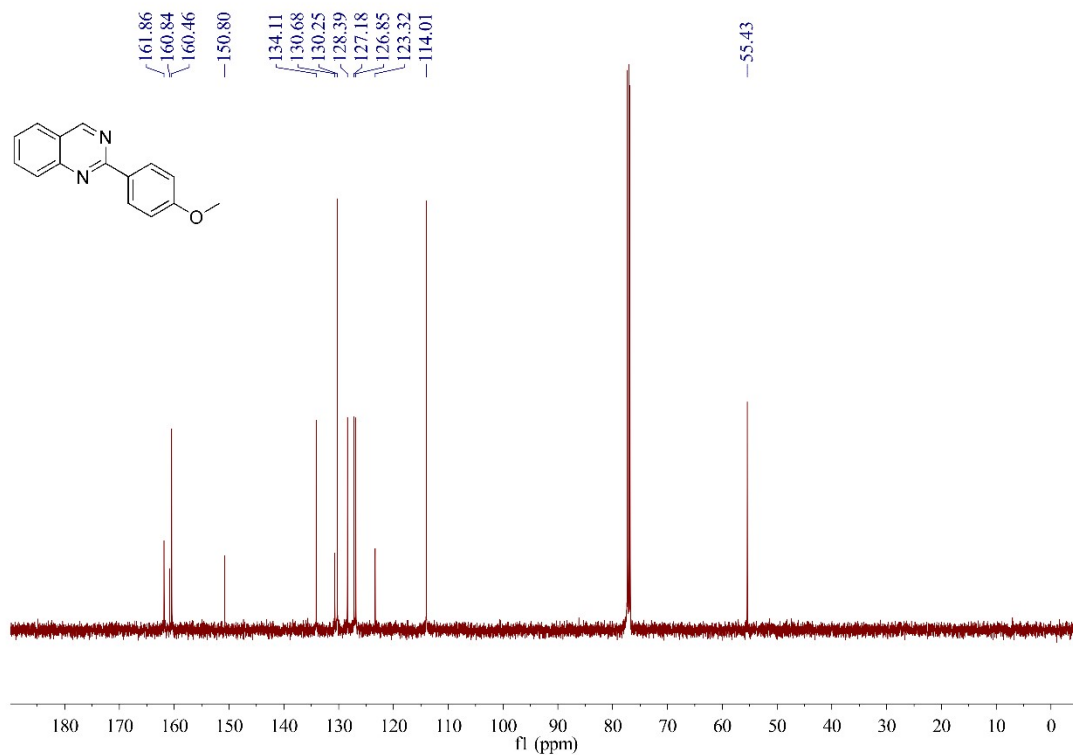
^{13}C NMR spectra of compound **3ab**



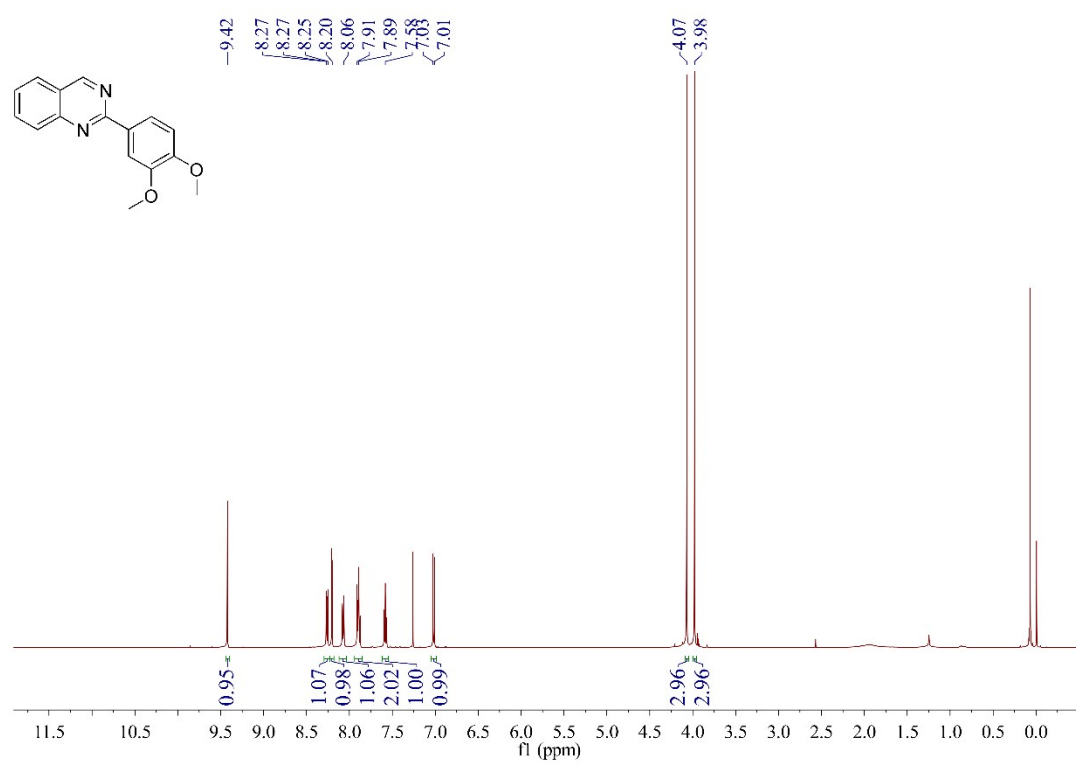
¹H NMR spectra of compound **3ac**



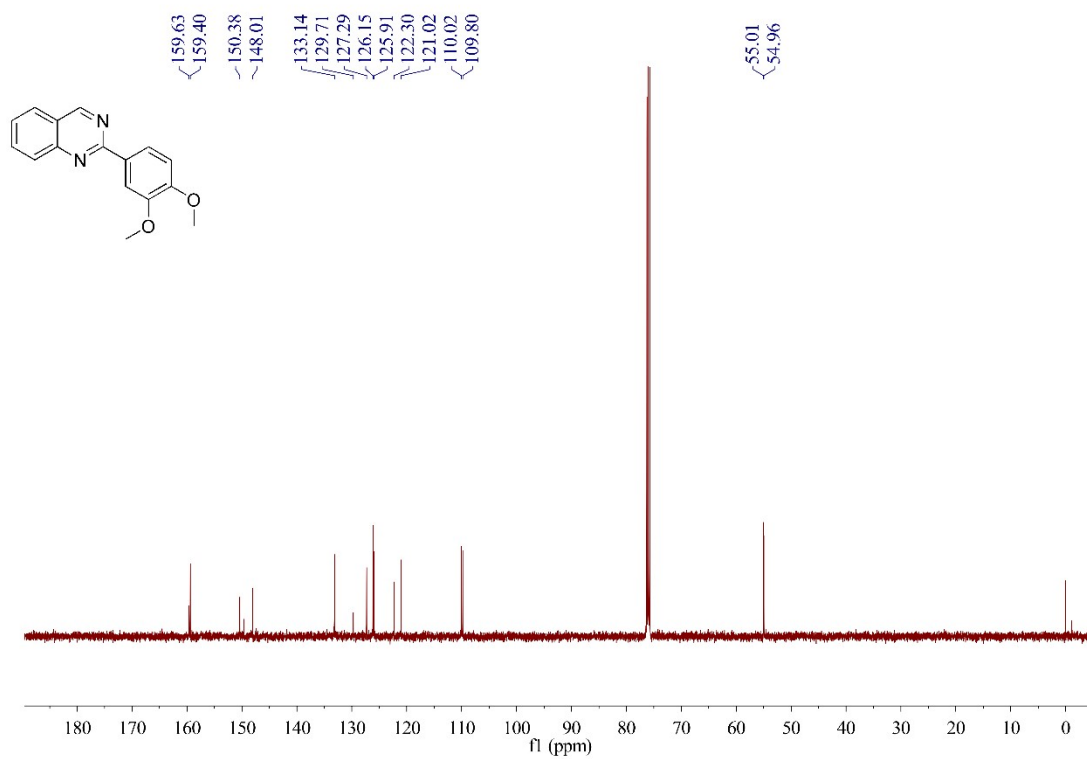
¹³C NMR spectra of compound **3ac**



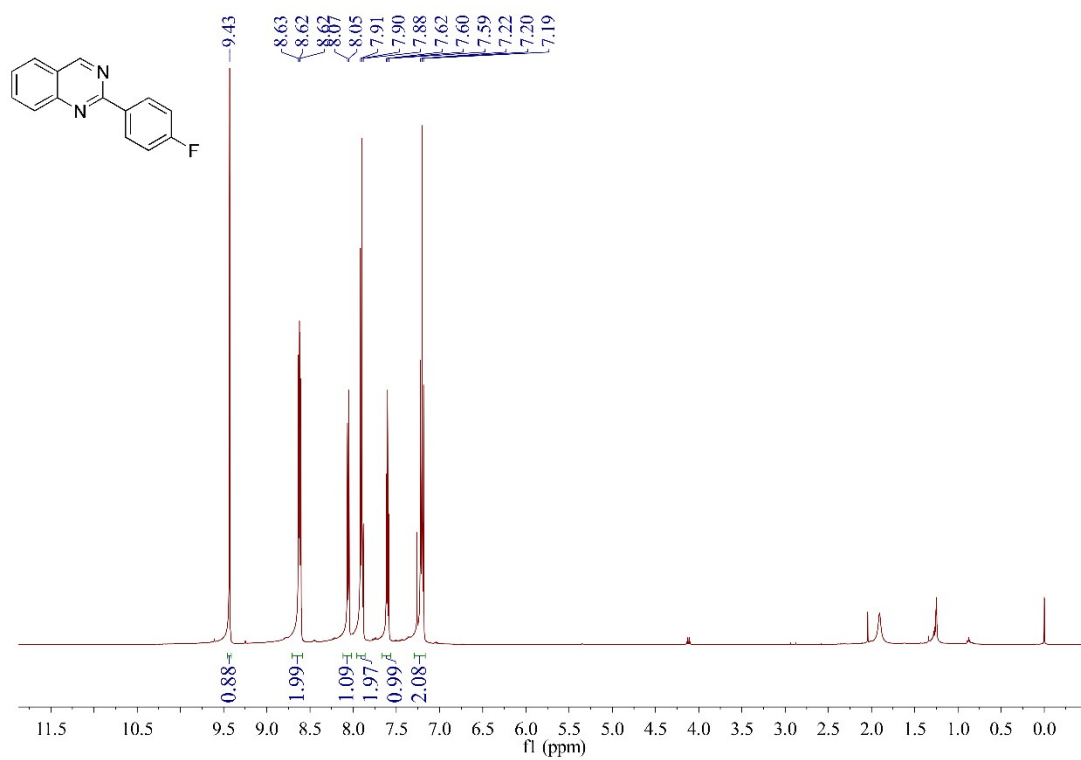
¹H NMR spectra of compound **3ad**



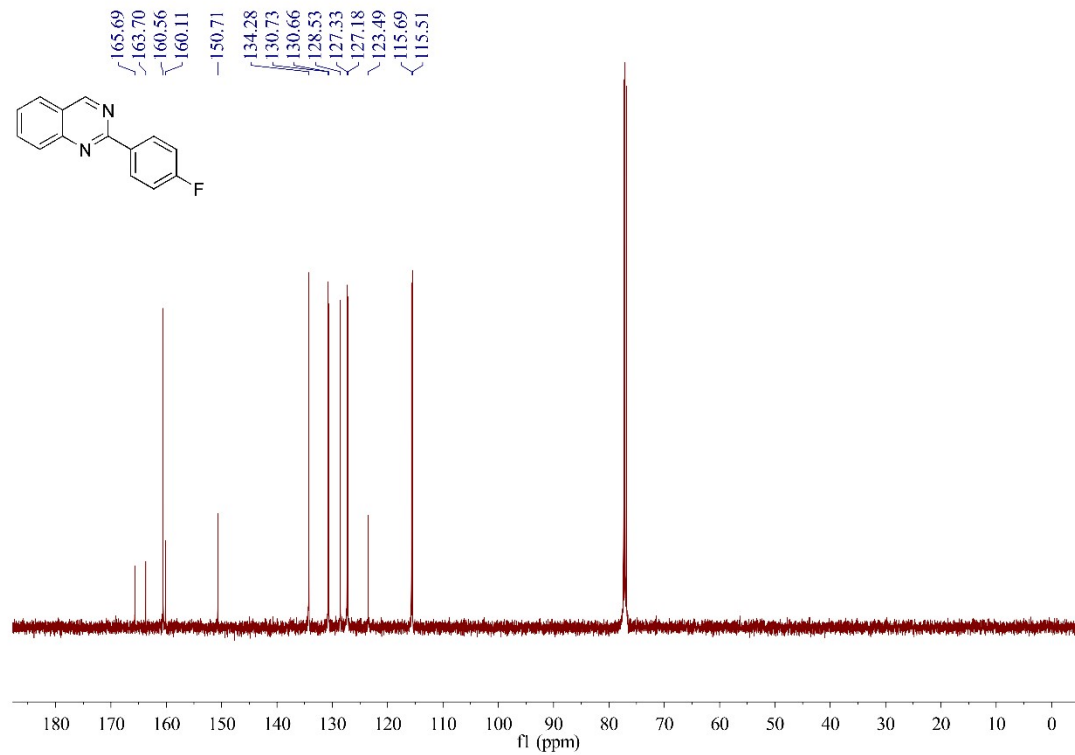
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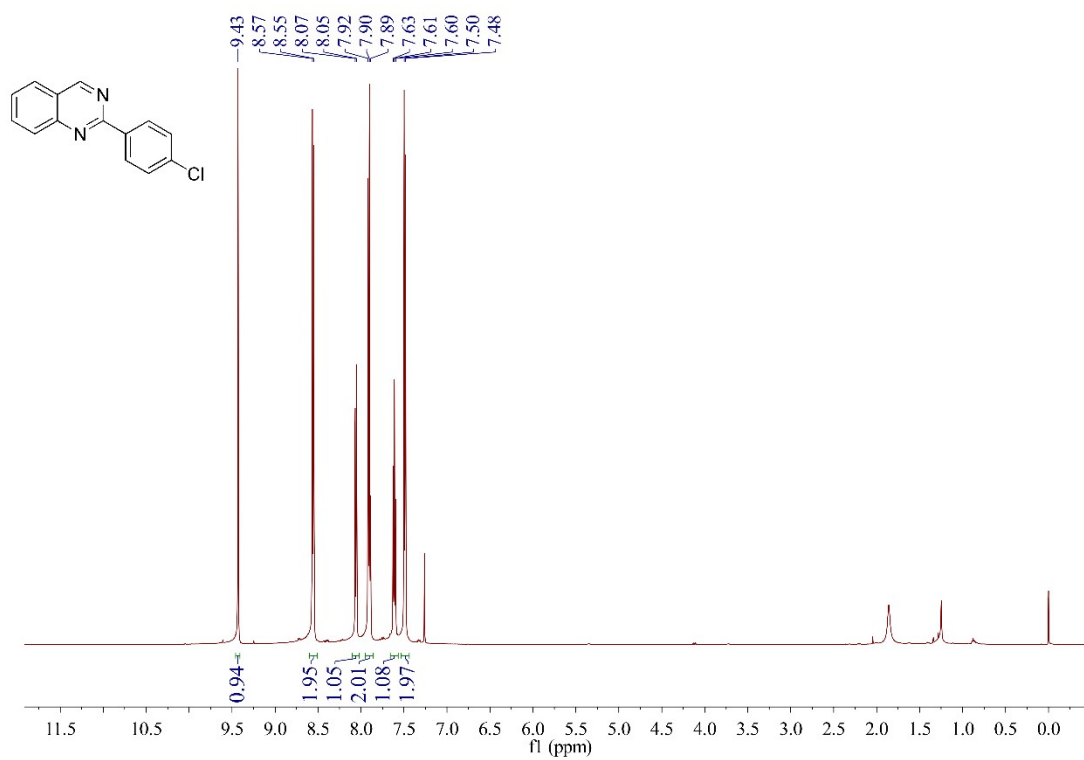
¹H NMR spectra of compound **3ae**



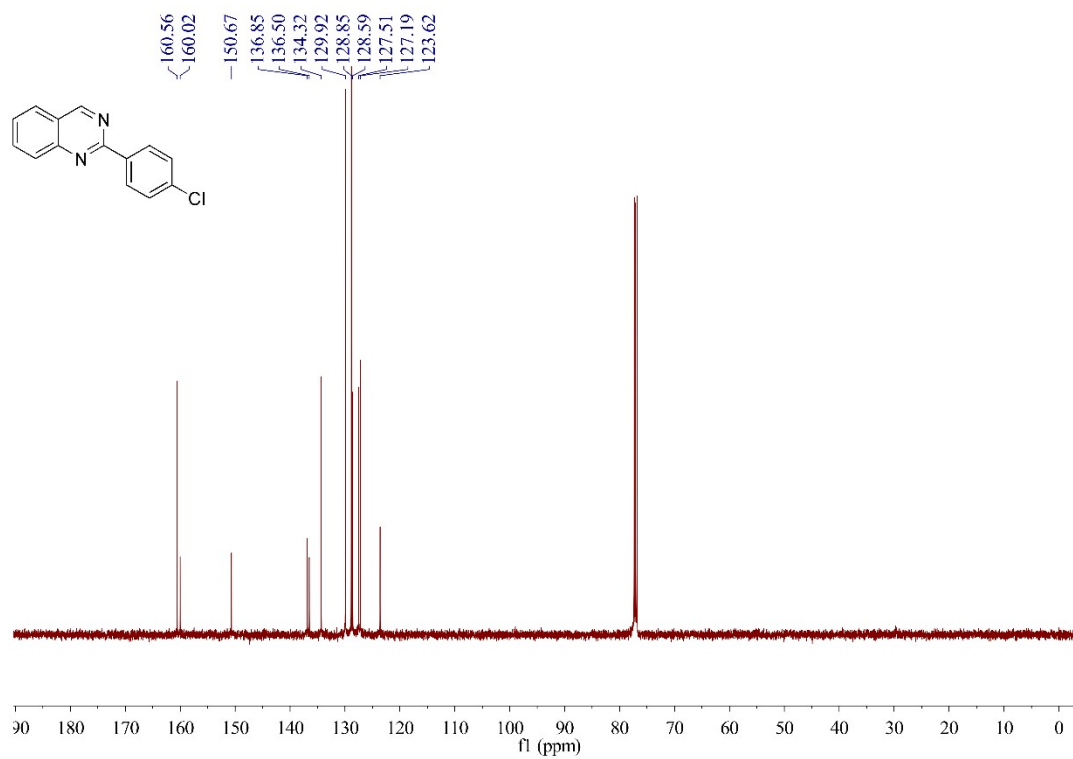
¹³C NMR spectra of compound **3ae**



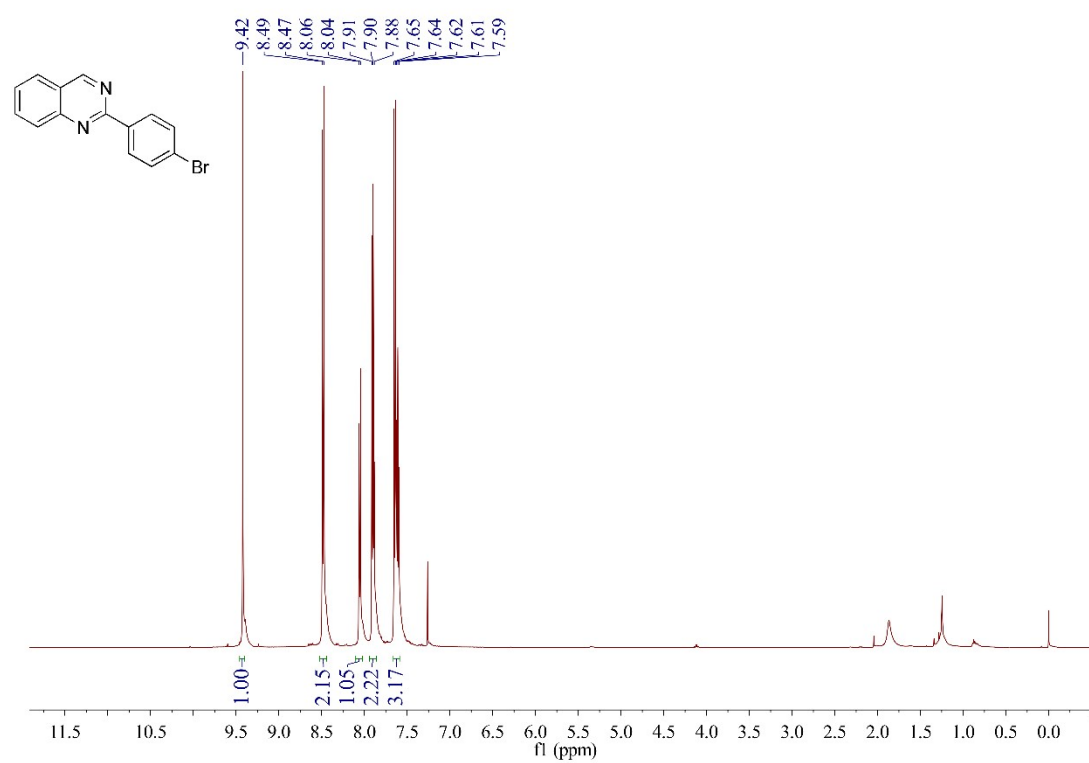
^1H NMR spectra of compound **3af**



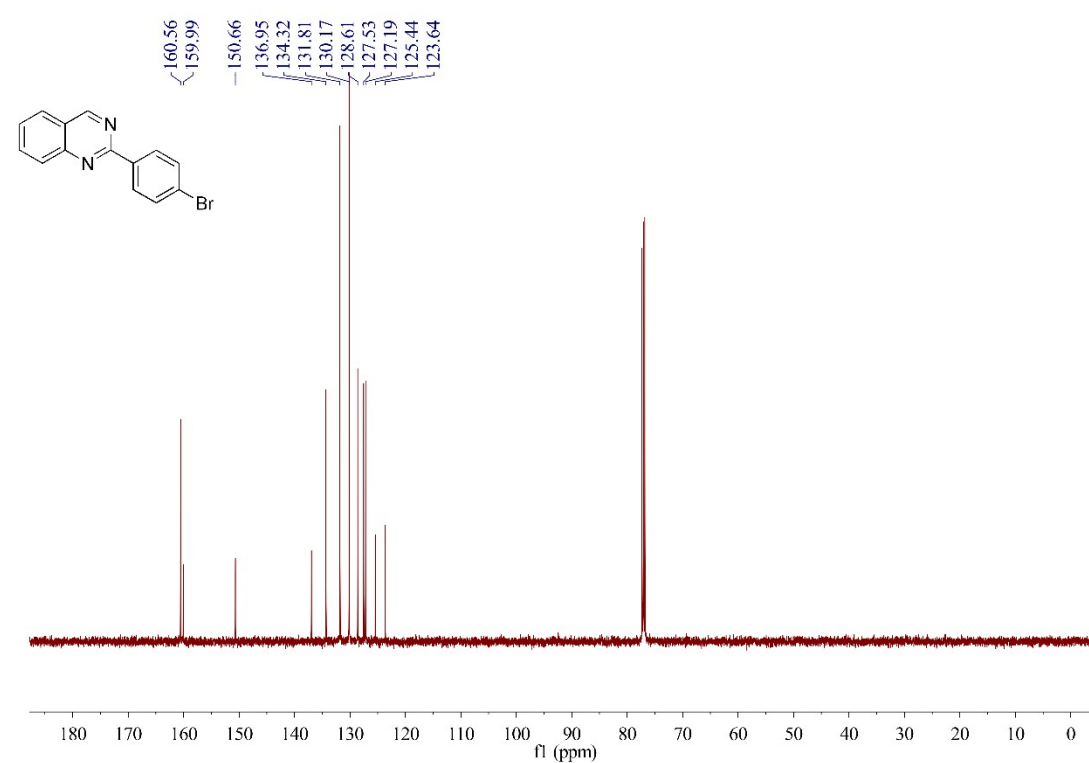
^{13}C NMR spectra of compound **3af**



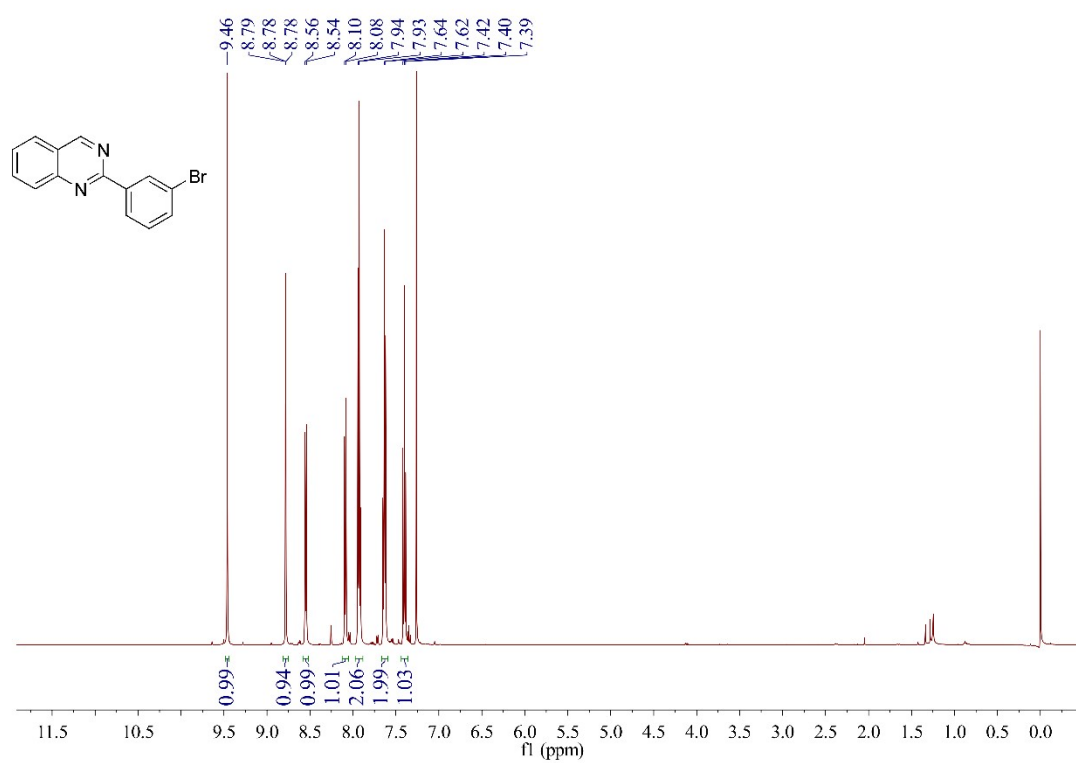
^1H NMR spectra of compound **3ag**



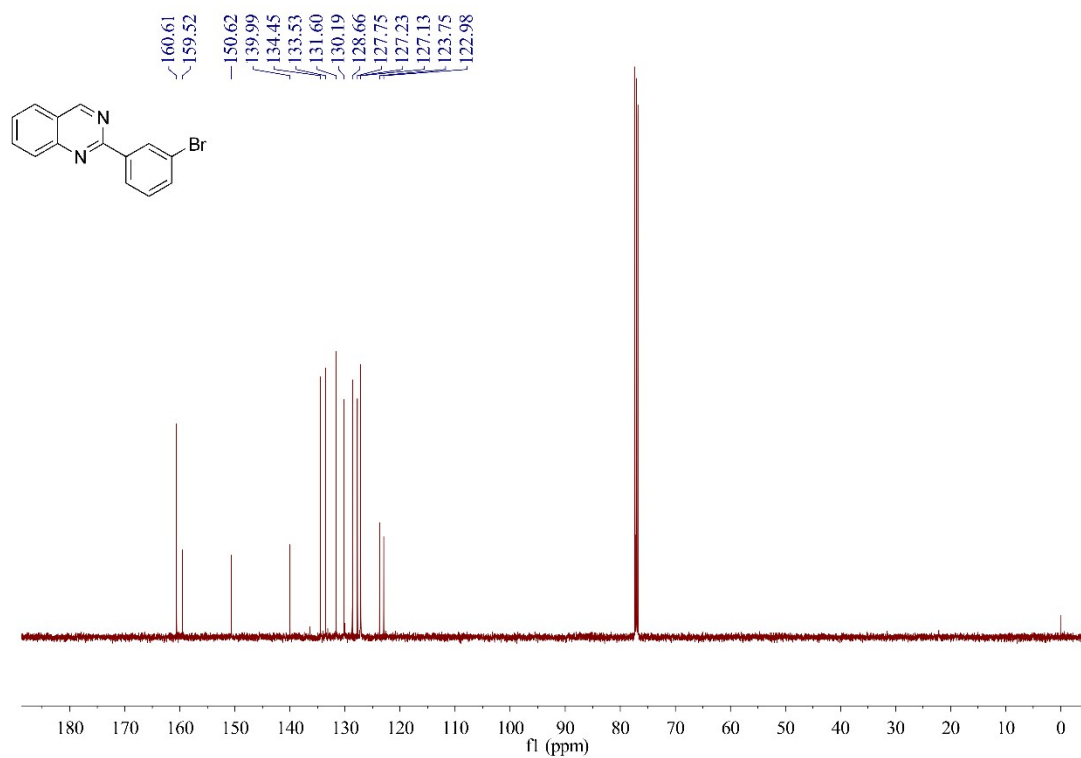
^{13}C NMR spectra of compound **3ag**



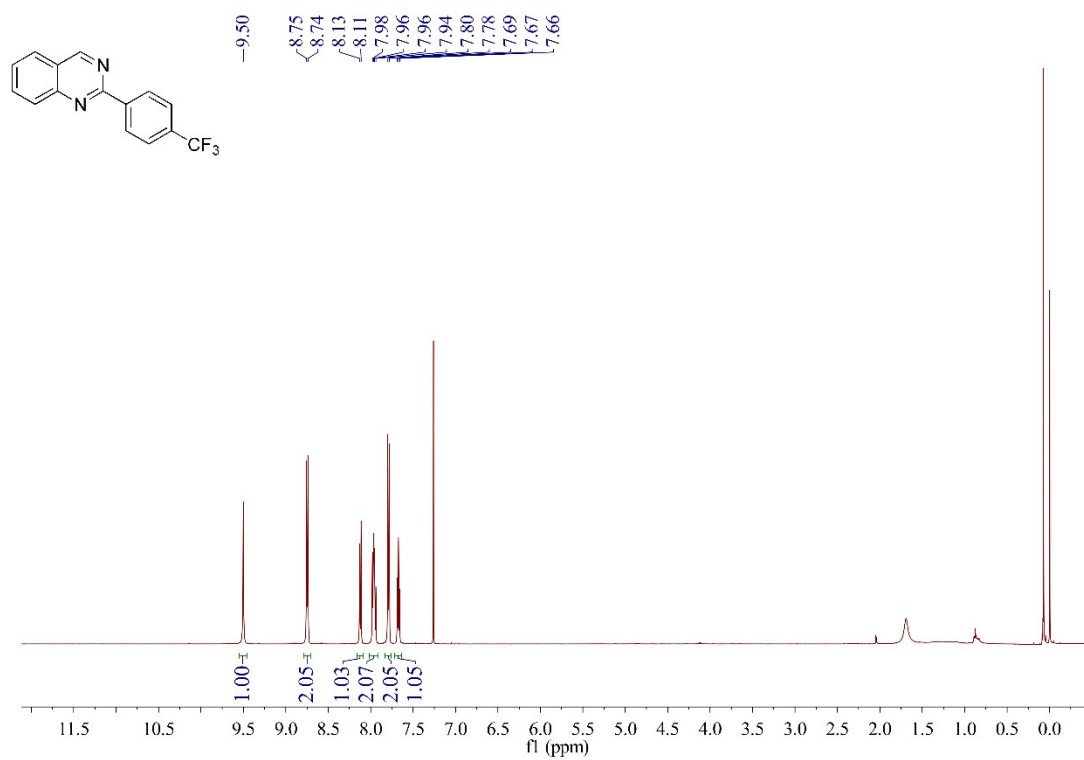
¹H NMR spectra of compound **3ah**



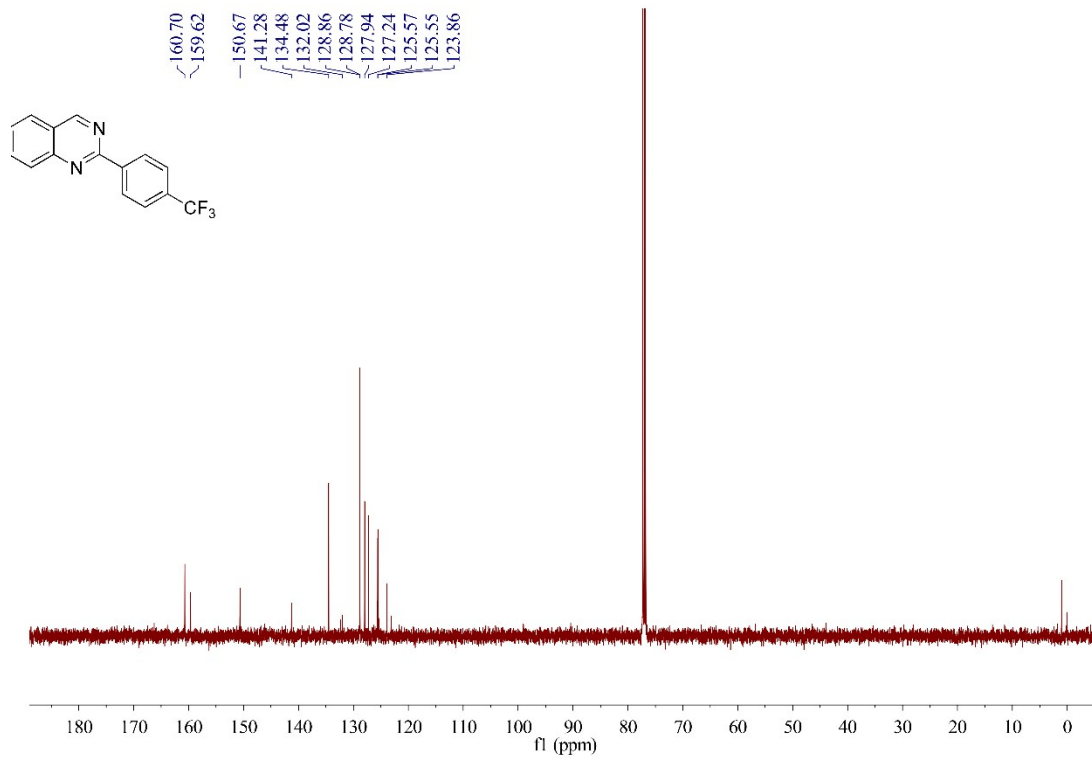
¹³C NMR spectra of compound **3ah**



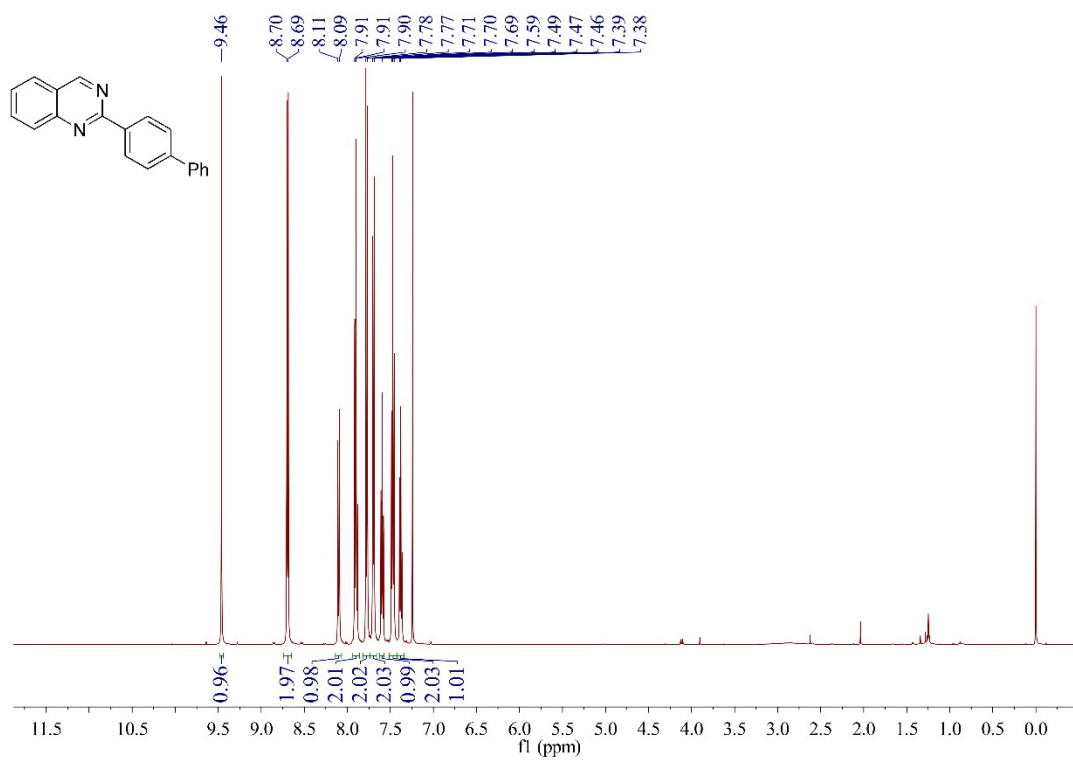
¹H NMR spectra of compound **3ai**



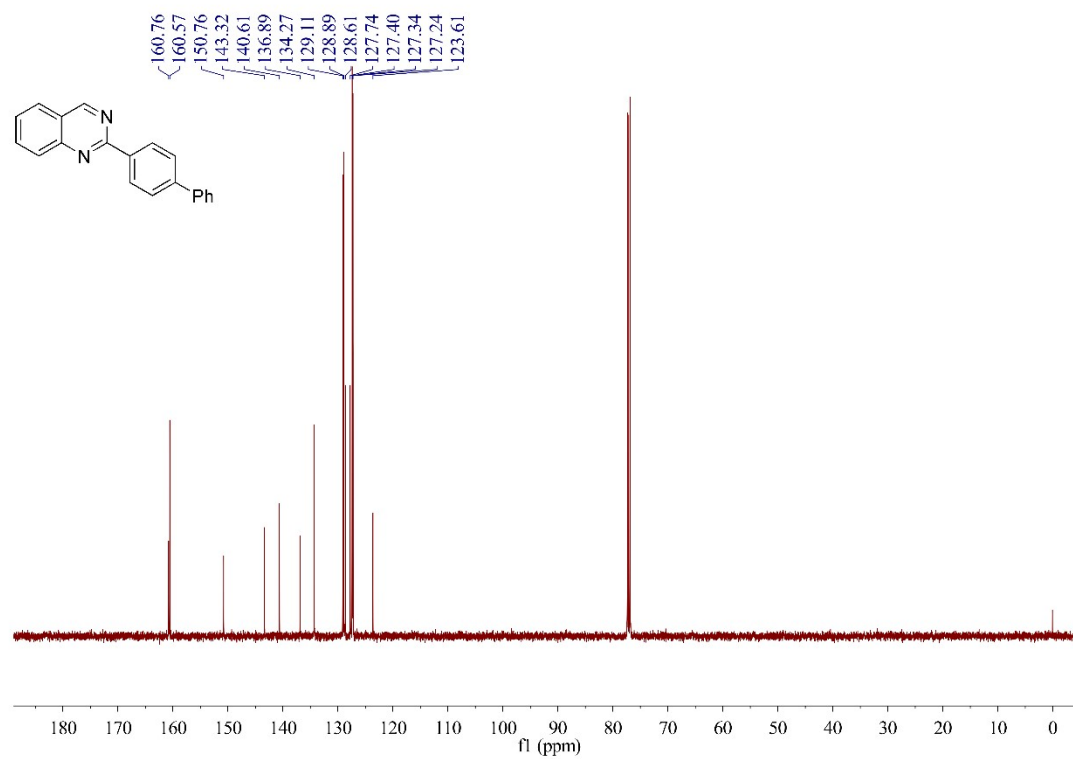
¹³C NMR spectra of compound **3ai**



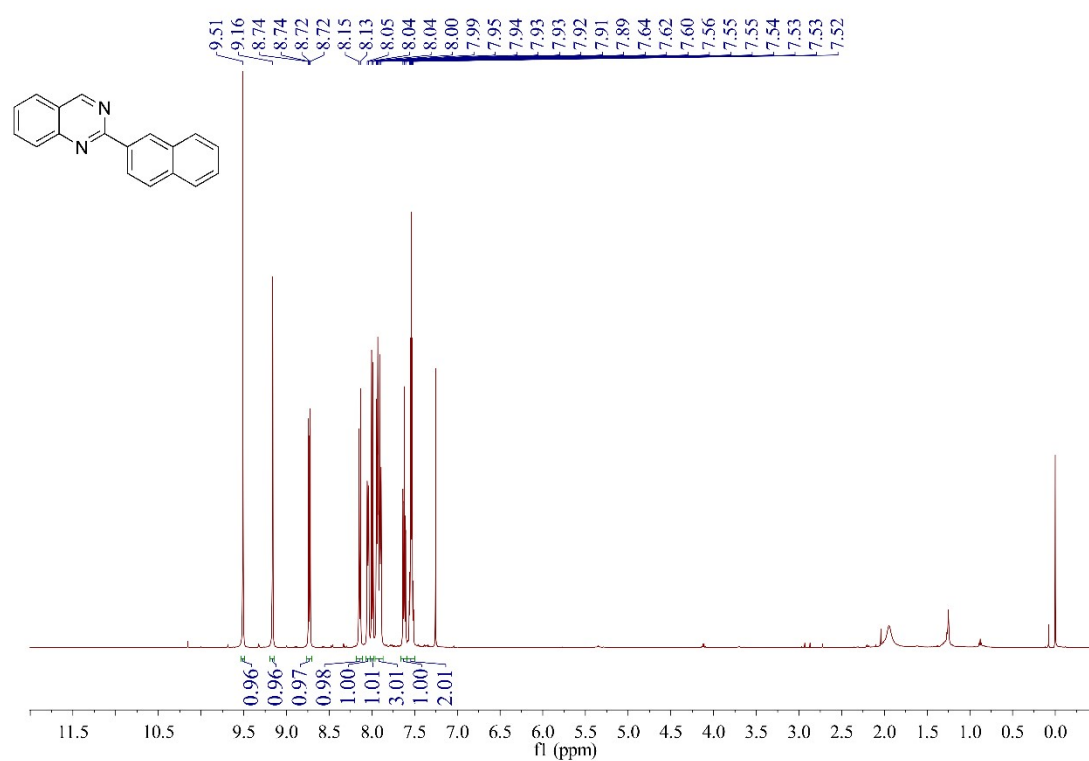
^1H NMR spectra of compound **3aj**



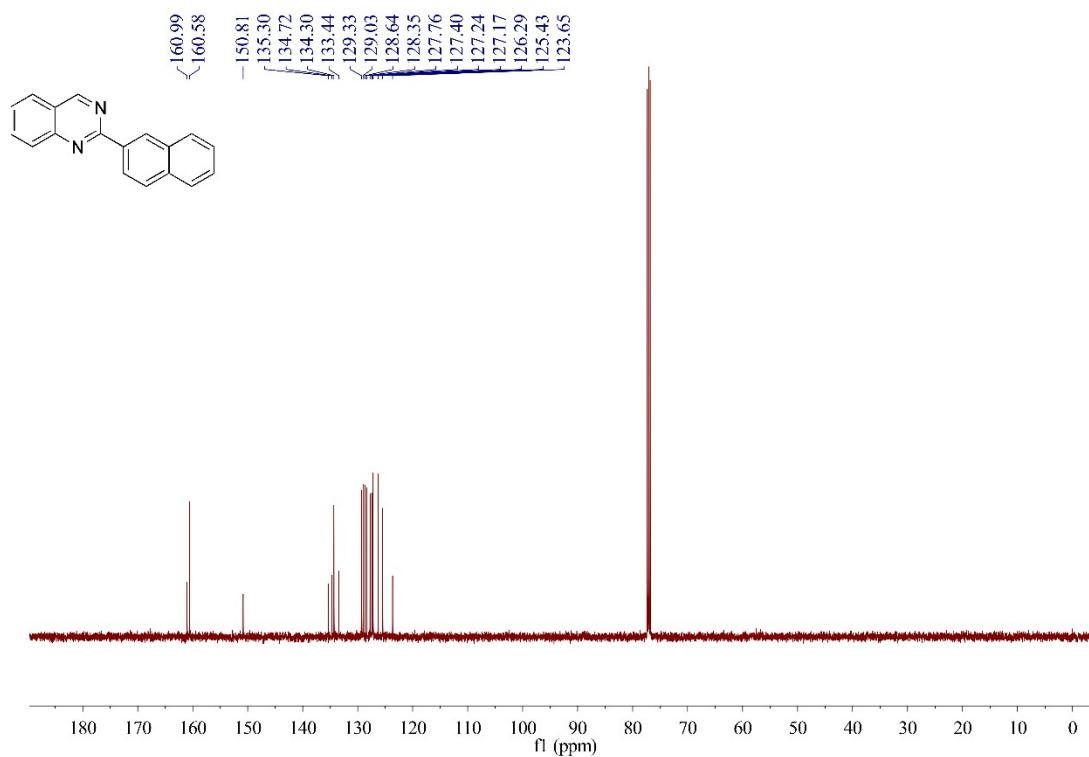
^{13}C NMR spectra of compound **3aj**



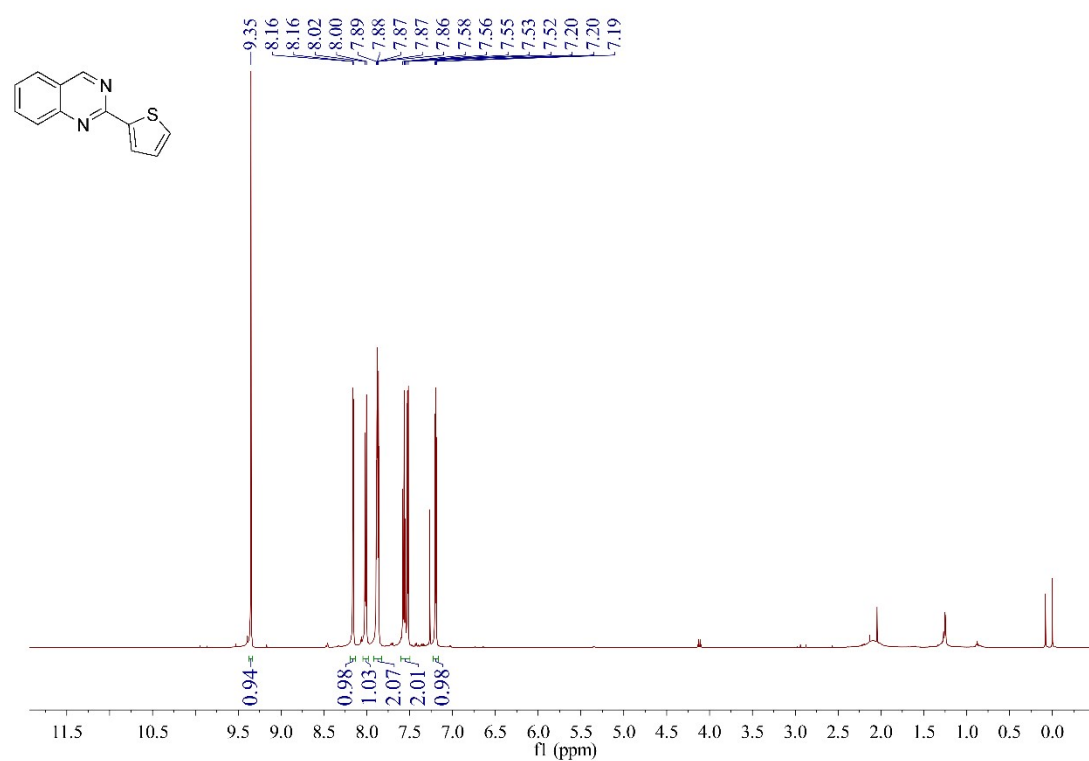
¹H NMR spectra of compound **3ak**



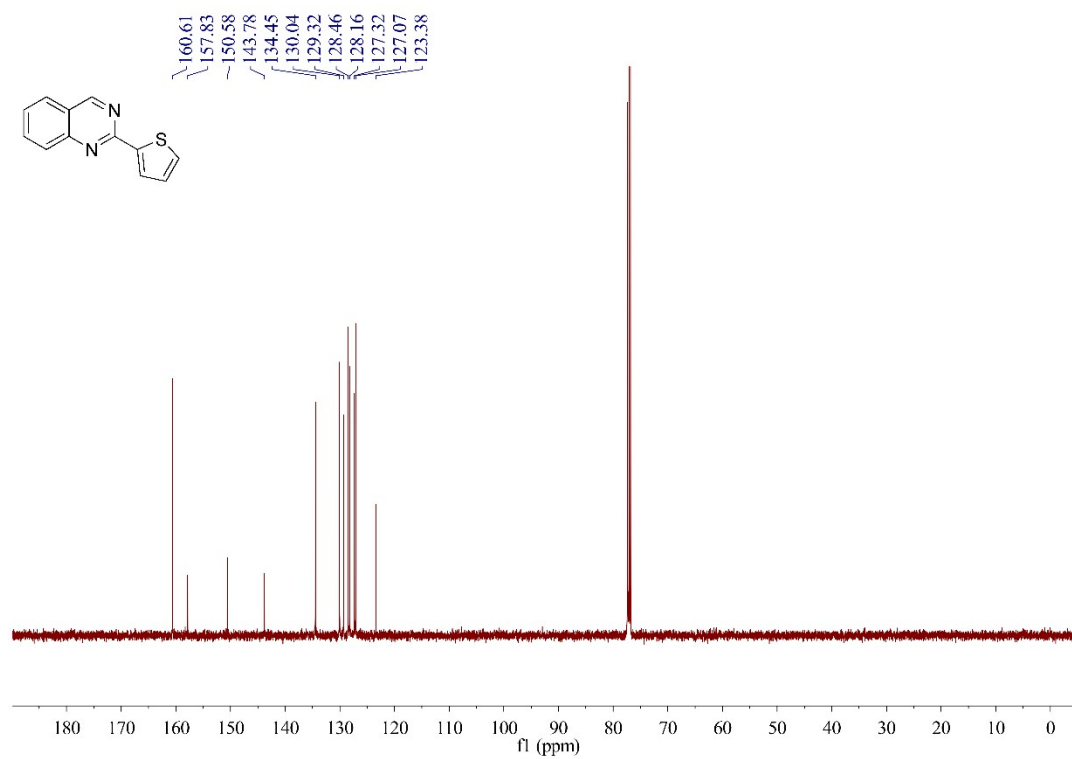
¹³C NMR spectra of compound **3ak**



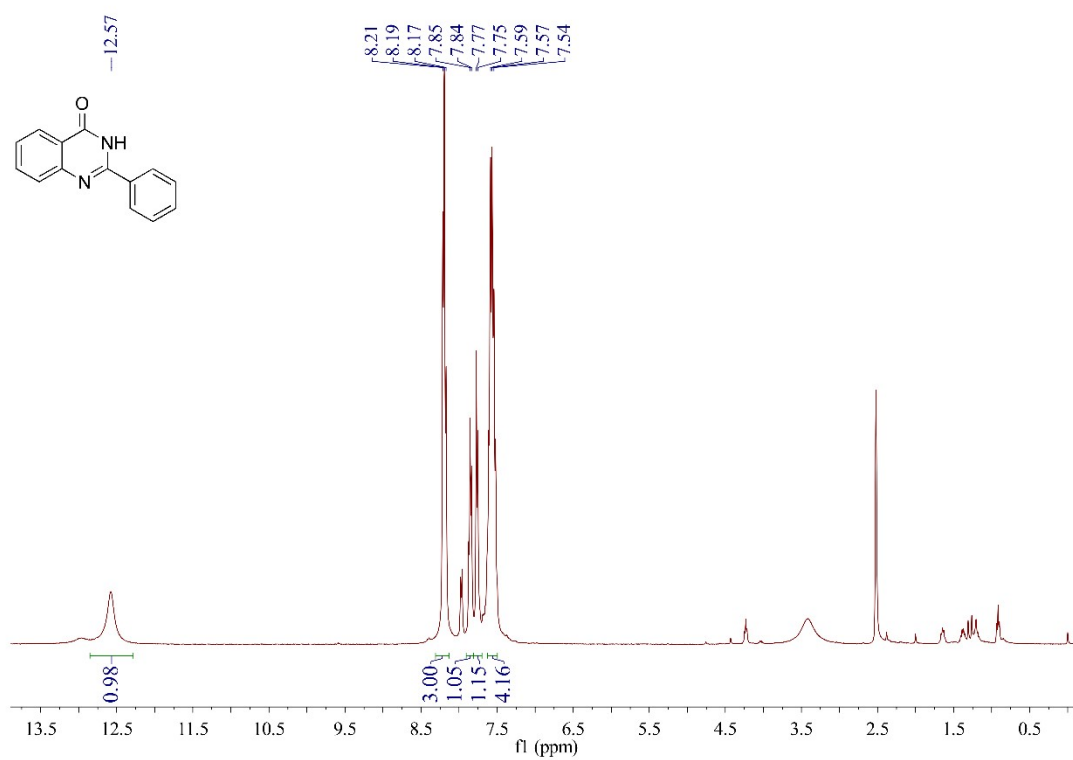
¹H NMR spectra of compound **3al**



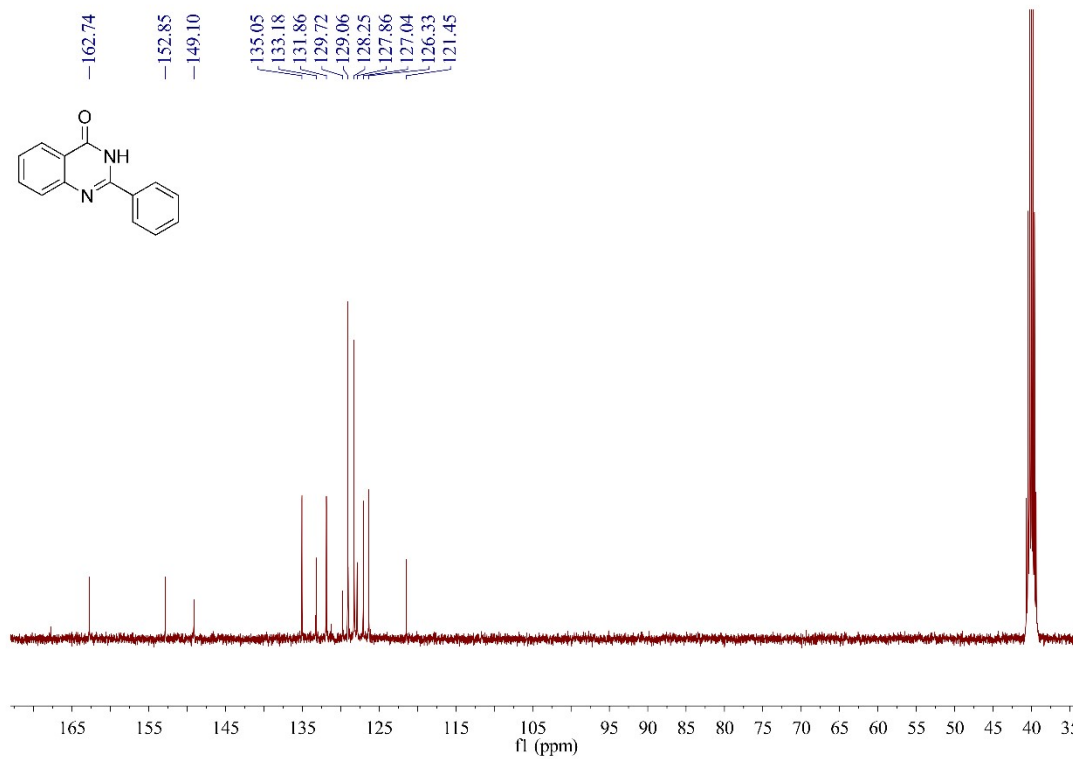
¹³C NMR spectra of compound **3al**



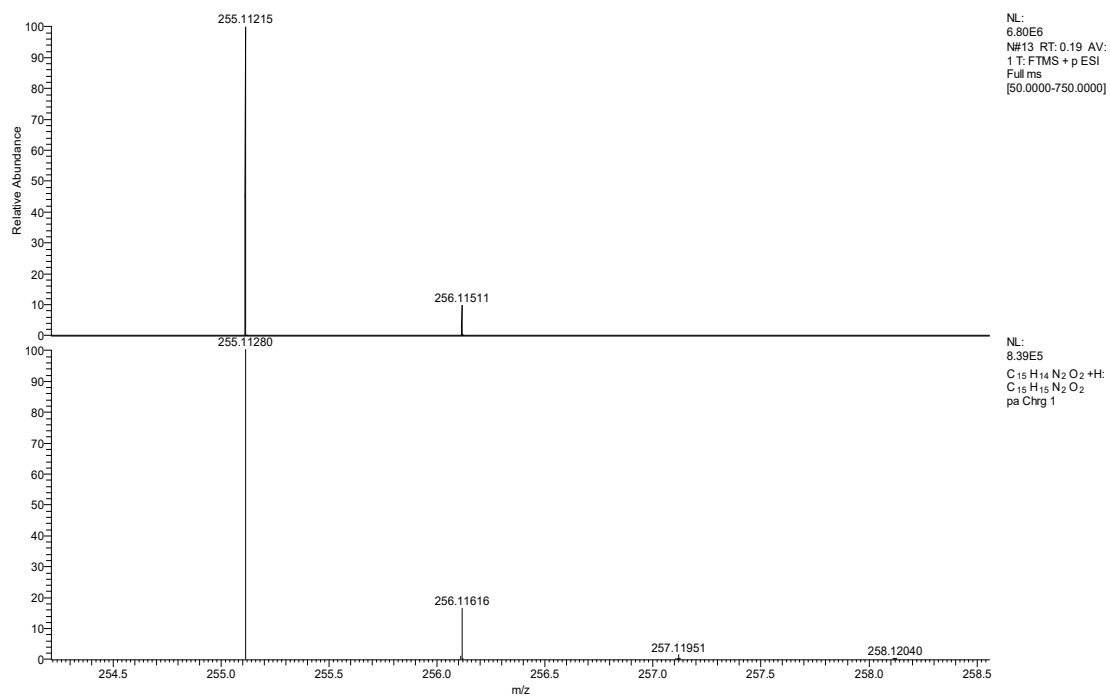
^1H NMR spectra of compound **3ba**



^{13}C NMR spectra of compound **3ba**



HRMS: m/z calcd for C₁₅H₁₄N₂O₂ [M + H]⁺: 255.11280; found: 255.11215.



References

- 1 Li, P. G., Zhu, H., Fan, M., Yan, C., Shi, K., Chi, X. W. and Zou, L. H., *Org Biomol Chem*, 2019, **17**, 5902.
- 2 Zhang, S.-Q., Cui, Y., Guo, B., Young, D. J., Xu, Z. and Li, H.-X., *Tetrahedron*, 2021, **78**, 131825.
- 3 Chen, Z., Chen, J., Liu, M., Ding, J., Gao, W., Huang, X. and Wu, H., *J Org Chem*, 2013, **78**, 11342.
- 4 Hu, Y., Chen, L. and Li, B., *RSC Advances*, 2016, **6**, 65196.