

Heterogeneous oxidative synthesis of quinazolines over OMS-2 under ligand-free conditions

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1 General Information

All reagents were purchased from commercial suppliers and used without further purification. All experiments were carried out under air or using O₂ balloon. Flash chromatography was carried out with Merck silica gel 60 (200-300 mesh). Analytical TLC was performed with Merck silica gel 60 F254 plates, and the products were visualized by UV detection. ¹H NMR and ¹³C NMR (400 and 100 MHz respectively) spectra were recorded in CDCl₃. Chemical shifts (δ) are reported in ppm using TMS as internal standard, and spin-spin coupling constants (J) are given in Hz.

2. Catalyst Characterization

Nitrogen adsorption-desorption measurements were performed at 76 K using an ASAP 2020M analyzer utilizing the BET model for the calculation of specific surface areas. The element contents of samples were determined by ICP-AES (ContrAA 700). The crystal phase and composition were determined by power X-ray diffraction using a X-Pert PRO X-ray diffractometer with Cu Ka radiation in the 2 θ range of 10–90°. The morphologies of the samples were characterized by a TF20 transmission electron microscope and SM-5600LV or Quanta 250 FEG scanning electron microscope. The X-ray photoelectron spectroscopy (XPS) measurements were performed on a Kratos AXIS Ultra DLD high performance electron spectrometer

using nonmonochromatized Al K α excitation source ($h\nu = 1486.6$ eV). Binding energies were calibrated by using the contaminant carbon (C 1s = 284.6 eV).

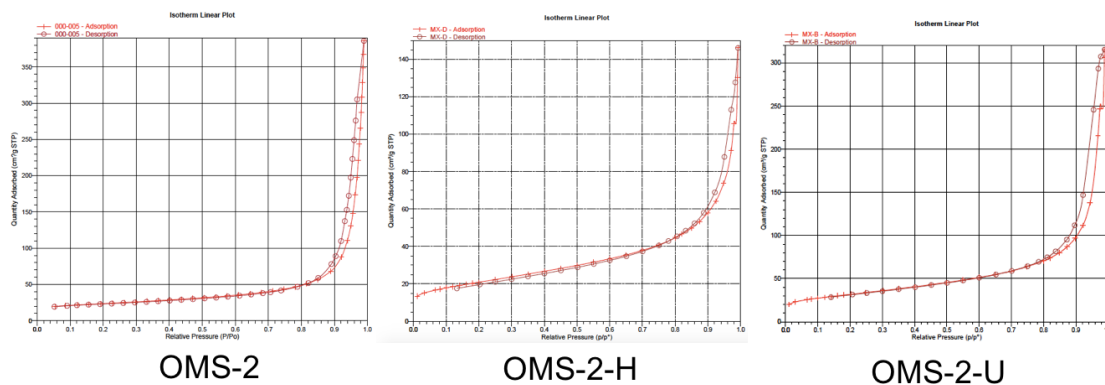


Figure S1. N₂ adsorption–desorption isotherms for OMS-2, OMS-2-H and OMS-2-U.

Table S1. Results of ICP-AES.

Catalyst	Content (wt%)	
	Mn	K
OMS-2	54.4	4.65
OMS-2-H	52.5	4.75
OMS-2-U	58.5	2.71

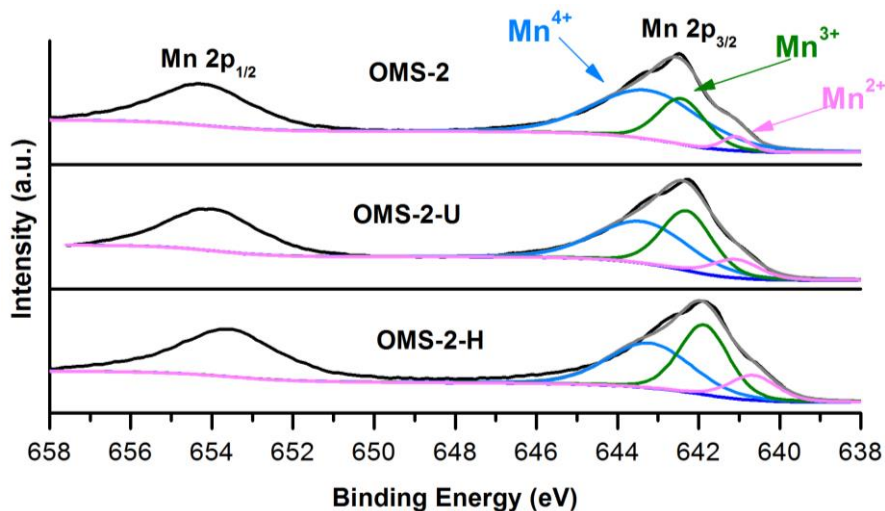
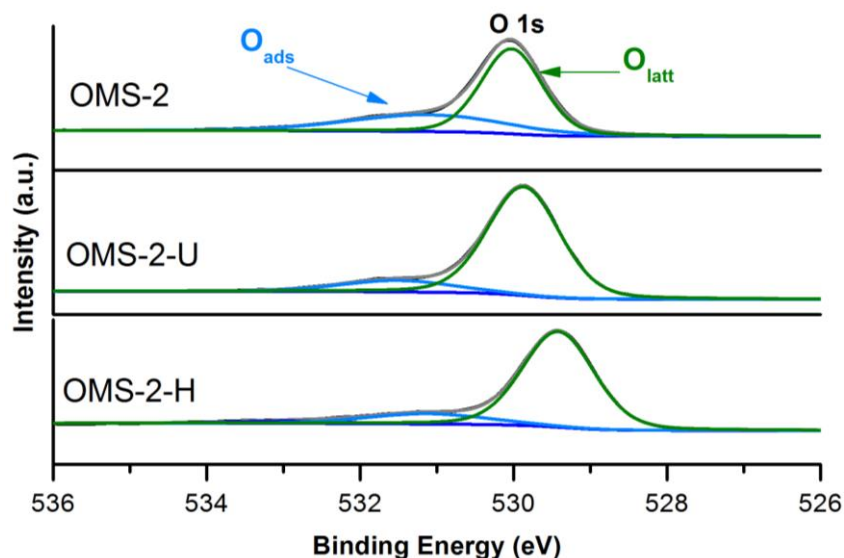


Figure S2. XPS spectra of Mn 2p for OMS-2-based materials.

Table S2. XPS results of Mn 2p_{3/2} for OMS-2-based materials.

Catalyst	Binding energy of Mn species in Mn 2p _{3/2} (eV)			Mn species (%)		
	Mn ⁴⁺	Mn ³⁺	Mn ²⁺	Mn ⁴⁺	Mn ³⁺	Mn ²⁺
OMS-2	643.3	642.2	641.0	66.8	28.1	5.1
OMS-2-U	643.4	642.3	641.1	50.2	39.2	10.6
OMS-2-H	643.2	641.9	640.7	44.6	40.7	14.7

**Figure S3.** XPS spectra of O 1s for OMS-2-based materials.**Table S3.** XPS results of O 1s for OMS-2-based materials.

Catalyst	Binding energy of oxygen species in O 1s (eV)		oxygen species (%)	
	Adsorbed O	Lattice O	Adsorbed O	Lattice O
OMS-2	531.1	530.0	33.6	66.4
OMS-2-U	531.5	529.9	14.1	85.9
OMS-2-H	531.1	529.4	17.8	82.2

3. Hot filtration experiment

After the reaction run for 2 h, the NMR yield of **3a** was roughly 38%. Then, the catalyst was simply filtered and the filtrate was allowed to react for further 10 h. It was found that the reaction did not occur anymore and the yield of **3a** did not increase (Figure S3). And

inductively coupled plasma-atomic emission spectroscopy (ICP-AES) was used to analyze the reaction solution after the filtration of OMS-2-U and Mn species were barely detected. which suggested the supported manganese did not leach from OMS-2-U. Furthermore, the retrieved heterogeneous catalyst that was washed and dried at 120 °C for 5 h could be used efficiently in the next run.

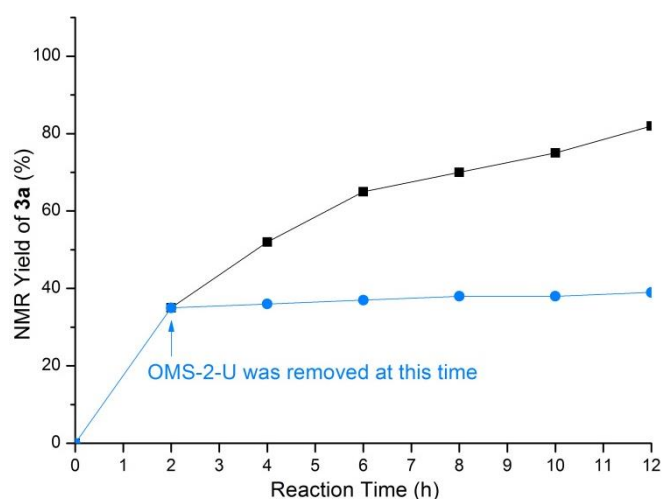
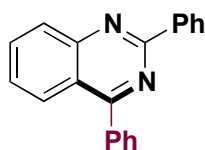


Figure S4. Hot-filtration experiment. Anisole was used as internal standard for measuring yield by NMR.

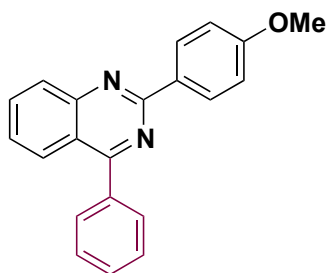
4. Spectrum Data of Products

2,4-diphenylquinazoline^[1] (3a)



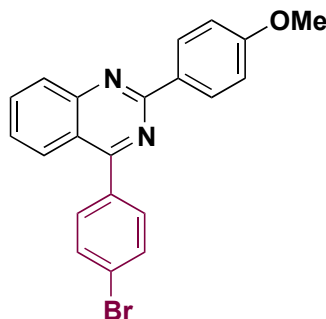
Yellow solid, isolated yield 82%, m.p. 114-115 °C. ¹H NMR (400MHz, CDCl₃): δ = 8.70 (d, *J* = 7.9, 1.6 Hz, 2H), 8.15 (t, *J* = 11.1, 8.7 Hz, 2H), 7.91-7.89 (m, 3H), 7.61-7.60 (m, 3H), 7.56-7.51 (m, 4H). The data of ¹³C NMR was in agreement with the corresponding product reported in reference.¹

2-(4-methoxyphenyl)-4-phenylquinazoline^[2] (3b)



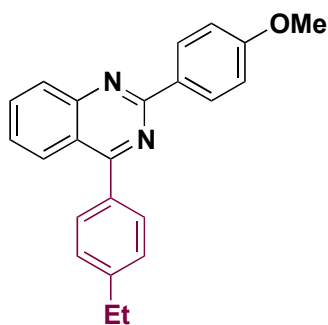
Yellow solid, isolated yield 85%, m.p. 141-142 °C. ¹H NMR (400MHz, CDCl₃): δ = 8.67 (d, *J* = 8.7 Hz, 2H), 8.10 (t, *J* = 8.7 Hz, 2H), 7.94-7.81 (m, 3H), 7.59 (d, *J* = 3.4 Hz, 3H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.05 (d, *J* = 8.8 Hz, 2H), 3.90 (s, 3H); ¹³C NMR (100MHz, CDCl₃): δ = ¹³C NMR (101 MHz, CDCl₃) δ = 168.0, 161.7, 159.9, 152.0, 137.7, 133.3, 130.8, 130.2, 130.1, 129.7, 128.8, 128.4, 126.9, 126.4, 121.3, 113.8, 55.3.

4-(4-bromophenyl)-2-(4-methoxyphenyl)quinazoline (3c)



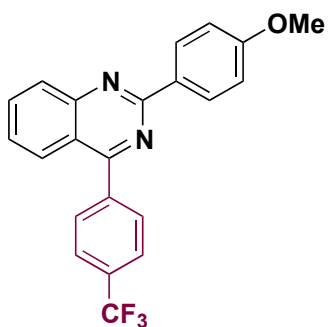
White solid, isolated yield 81%, m.p. 152-154 °C. ¹H NMR (400MHz, CDCl₃): δ = 8.63 (d, *J* = 8.5 Hz, 2H), 8.07 (dd, *J* = 30.6, 8.5 Hz, 2H), 7.87 (t, *J* = 7.6 Hz, 1H), 7.75 (q, *J* = 8.6 Hz, 4H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.04 (d, *J* = 8.4 Hz, 2H), 3.90 (s, 3H); ¹³C NMR (100MHz, CDCl₃): δ = 166.8, 161.7, 159.8, 151.9, 136.5, 133.5, 131.6, 131.6, 130.5, 130.1, 128.9, 126.6, 126.4, 124.4, 120.9, 113.7, 55.2. HRMS (ESI) calcd for C₂₁H₁₅BrN₂O ([M+H]⁺) 391.0434, found 391.0445.

4-(4-ethylphenyl)-2-(4-methoxyphenyl)quinazoline (3d)



White solid, isolated yield 91%, m.p. 138-140 °C. ^1H NMR (400MHz, CDCl_3): δ = 8.67 (d, J = 8.9 Hz, 2H), 8.11 (t, J = 9.0 Hz, 2H), 7.82 (d, J = 11.6 Hz, 3H), 7.50-7.37 (m, 3H), 7.04 (d, J = 9.0 Hz, 2H), 3.88 (s, 3H), 2.79 (q, J = 7.6 Hz, 2H), 1.34 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100MHz, CDCl_3): δ = 168.1, 161.6, 159.9, 151.9, 146.3, 135.1, 133.3, 130.9, 130.2, 130.2, 128.8, 128.0, 127.0, 126.3, 121.3, 113.7, 55.3, 28.8, 15.5. HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}$ ($[\text{M}+\text{H}]^+$) 341.1694, found 341.1685.

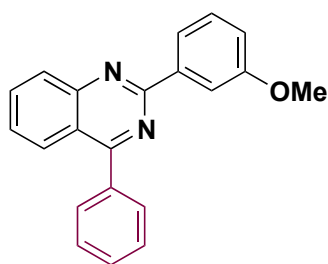
2-(4-methoxyphenyl)-4-(4-(trifluoromethyl)phenyl)quinazoline (3e)



White solid, isolated yield 75%, m.p. 129-131 °C. ^1H NMR (400MHz, CDCl_3): δ = 8.63 (d, J = 8.8 Hz, 2H), 8.11 (d, J = 8.5 Hz, 1H), 7.97 (d, J = 8.1 Hz, 3H), 7.85 (d, J = 8.0 Hz, 3H), 7.50 (t, J = 7.6 Hz, 1H), 7.03 (d, J = 8.8 Hz, 2H), 3.88 (s, 3H); ^{13}C NMR (100MHz, CDCl_3): δ = 166.3,

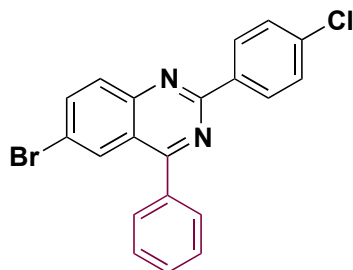
161.6, 159.7, 151.7, 140.9, 133.4, 131.5, 131.2, 130.3, 130.2, 130.1, 130.0, 128.8, 126.6, 126.0, 125.2, 125.2, 125.1, 125.1, 120.8, 113.6, 55.1. HRMS (ESI) calcd for $C_{22}H_{15}F_3N_2O$ ($[M+H]^+$) 381.1134, found 381.1121.

2-(3-methoxyphenyl)-4-phenylquinazoline^[2] (3f)



Yellow solid, isolated yield 86%, m.p. 132-134 °C. 1H NMR (400MHz, $CDCl_3$): δ = 8.30 (dd, J = 16.4, 4.4 Hz, 2H), 8.24-8.08 (m, 2H), 7.98-7.83 (m, 3H), 7.65-7.52 (m, 4H), 7.45 (t, J = 7.9 Hz, 1H), 7.07 (dd, J = 8.2, 1.7 Hz, 1H), 3.95 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$): δ = 168.2, 159.8, 151.9, 139.6, 137.6, 137.3, 133.4, 130.1, 129.8, 129.4, 129.1, 128.4, 127.0, 126.9, 122.1, 121.6, 121.2, 116.7, 113.3, 55.4.

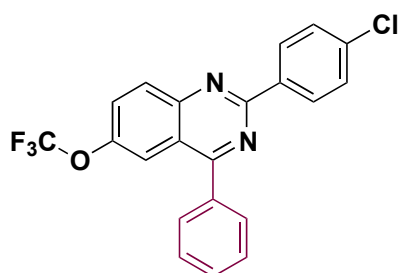
6-bromo-2-(4-chlorophenyl)-4-phenylquinazoline^[1] (3g)



Yellow solid, isolated yield 78%, m.p. 155-156 °C. 1H NMR (400MHz, $CDCl_3$): δ = 8.61 (d, J = 8.7 Hz, 2H), 8.25 (d, J = 1.9 Hz, 1H), 7.95 (dt, J = 9.0, 5.5 Hz, 2H), 7.89-7.79 (m, 2H), 7.67-7.57 (m, 3H), 7.52-7.42 (m,

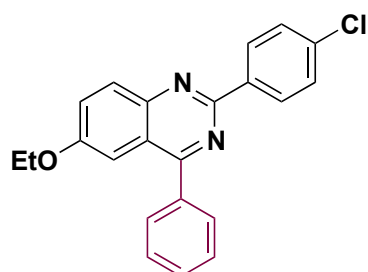
2H); ^{13}C NMR (100MHz, CDCl_3): δ = 167.4, 159.5, 150.6, 137.1, 137.0, 136.9, 136.2, 130.8, 130.2, 130.1, 130.0, 129.1, 128.7, 128.7, 122.6, 120.8.

2-(4-chlorophenyl)-4-phenyl-6-(trifluoromethoxy)quinazoline (3h)



Colorless solid, isolated yield 82%, m.p. 145-146 °C. ^1H NMR (400MHz, CDCl_3): δ = 8.66-8.58 (m, 2H), 8.16 (d, J = 9.2 Hz, 1H), 8.00-7.91 (m, 1H), 7.90-7.82 (m, 2H), 7.78-7.71 (m, 1H), 7.67-7.59 (m, 3H), 7.50-7.44 (m, 2H); ^{13}C NMR (100MHz, CDCl_3): δ = ^{13}C NMR (101 MHz, CDCl_3) δ 168.3, 159.7, 150.3, 147.0, 147.1, 137.0, 136.8, 136.1, 131.4, 130.4, 130.0, 129.9, 128.7, 128.7, 127.7, 124.3, 121.7, 121.6, 119.2, 117.6, 116.6. HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{12}\text{ClF}_3\text{N}_2\text{O}$ ($[\text{M}+\text{H}]^+$) 401.0632, found 401.0635

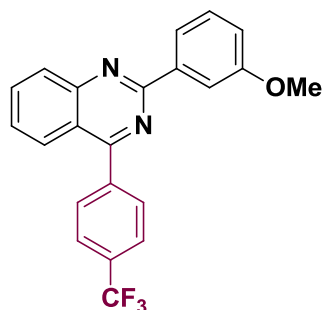
2-(4-chlorophenyl)-6-ethoxy-4-phenylquinazoline (3i)



Pale yellow solid, isolated yield 86%, m.p. 144-146 °C. ^1H NMR (400MHz, CDCl_3): δ = 8.60 (d, J = 8.6 Hz, 2H), 8.03 (d, J = 9.2 Hz, 1H),

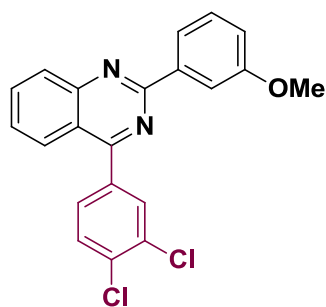
7.90-7.83 (m, 2H), 7.59 (dd, $J = 5.4, 1.8$ Hz, 3H), 7.54 (dd, $J = 9.2, 2.7$ Hz, 1H), 7.49-7.44 (m, 2H), 7.36 (d, $J = 2.7$ Hz, 1H), 4.05 (q, $J = 7.0$ Hz, 2H), 1.45 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (100MHz, CDCl_3): $\delta = 166.5, 157.5, 157.5, 147.9, 137.9, 136.9, 136.1, 130.5, 129.8, 129.7, 129.6, 128.6, 128.5, 126.5, 122.5, 105.1, 63.9, 14.5$. HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{17}\text{ClN}_2\text{O}$ ($[\text{M}+\text{H}]^+$) 361.1011, found 361.1021.

2-(3-methoxyphenyl)-4-(4-(trifluoromethyl)phenyl)quinazoline (3j)



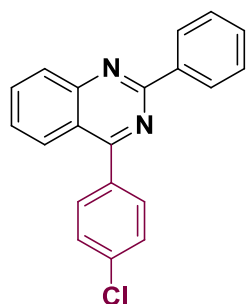
White solid, isolated yield 88%, m.p. 132-134 °C. ^1H NMR (400MHz, CDCl_3): $\delta = 8.32\text{-}8.21$ (m, 2H), 8.19 (d, $J = 8.3$ Hz, 1H), 8.04-7.98 (m, 3H), 7.95-7.82 (m, 3H), 7.58 (t, $J = 7.6$ Hz, 1H), 7.45 (t, $J = 7.9$ Hz, 1H), 7.07 (d, $J = 7.7$ Hz, 1H), 3.95 (s, 3H); ^{13}C NMR (100MHz, CDCl_3): $\delta = 166.8, 160.1, 160.0, 152.0, 141.1, 139.3, 133.8, 130.5, 130.4, 129.6, 129.4, 127.4, 126.3, 125.7, 125.6, 125.5, 121.5, 121.2, 117.0, 113.4, 55.4$. HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{15}\text{F}_3\text{N}_2\text{O}$ ($[\text{M}+\text{H}]^+$) 381.1256, found 381.1265.

4-(3,4-dichlorophenyl)-2-(3-methoxyphenyl)quinazoline (3k)



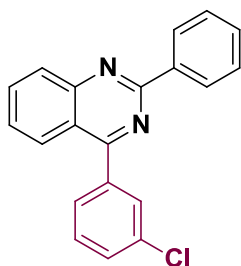
White solid, isolated yield 95%, m.p. 162-164 °C. ^1H NMR (400MHz, CDCl_3): δ = 8.32-8.20 (m, 2H), 8.16 (d, J = 8.2 Hz, 1H), 8.07-7.95 (m, 2H), 7.89 (t, J = 7.6 Hz, 1H), 7.68 (dd, J = 19.4, 8.2 Hz, 2H), 7.57 (t, J = 7.5 Hz, 1H), 7.44 (t, J = 7.8 Hz, 1H), 7.06 (d, J = 8.0 Hz, 1H), 3.94 (s, 3H); ^{13}C NMR (100MHz, CDCl_3): δ = 165.6, 159.9, 152.0, 139.2, 137.4, 134.4, 133.8, 133.0, 131.9, 130.6, 129.6, 129.4, 129.3, 127.5, 126.1, 121.3, 121.2, 116.9, 113.4, 55.4. HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}$ ($[\text{M}+\text{H}]^+$) 381.0574, found 381.0565.

4-(4-chlorophenyl)-2-phenylquinazoline^[1] (3l)



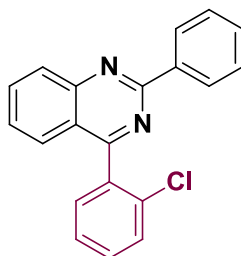
White solid, isolated yield 90%, m.p. 143-145 °C. ^1H NMR (400MHz, CDCl_3): δ = 8.68 (d, J = 7.5 Hz, 2H), 8.17 (d, J = 8.5 Hz, 1H), 8.08 (d, J = 8.3 Hz, 1H), 7.94-7.82 (m, 3H), 7.63-7.47 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ = 167.1, 160.2, 152.0, 138.1, 136.3, 136.1, 133.7, 131.5, 130.6, 129.3, 128.8, 128.6, 128.5, 127.2, 126.5, 121.4.

4-(3-chlorophenyl)-2-phenylquinazoline^[1] (3m)



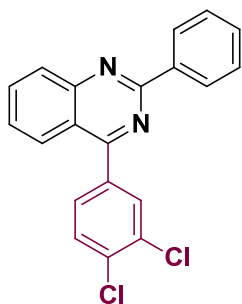
Pale yellow solid, isolated yield 88%, m.p. 139-141 °C. ¹H NMR (400MHz, CDCl₃): δ = 8.70 (d, *J* = 7.5 Hz, 2H), 8.17 (d, *J* = 8.5 Hz, 1H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.94-7.85 (m, 2H), 7.75 (d, *J* = 6.6 Hz, 1H), 7.60-7.48 (m, 6H); ¹³C NMR (100 MHz, CDCl₃): δ = 166.8, 160.2, 152.05, 139.4, 137.8, 134.7, 133.8, 130.6, 130.1, 129.9, 129.8, 129.3, 128.6, 128.5, 128.3, 127.3, 126.5, 121.4.

4-(2-chlorophenyl)-2-phenylquinazoline (3n)



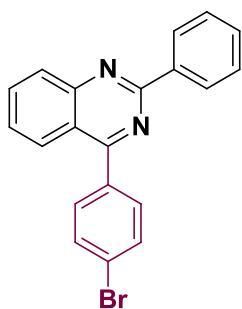
Pale yellow solid, isolated yield 56%, m.p. 139-141 °C. ¹H NMR (400MHz, CDCl₃): δ = 8.67 (d, *J* = 7.7 Hz, 2H), 8.17 (d, *J* = 8.4 Hz, 1H), 7.89 (t, *J* = 7.7 Hz, 1H), 7.66 (d, *J* = 8.3 Hz, 1H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.53-7.47 (m, 7H); ¹³C NMR (100 MHz, CDCl₃): δ = 167.2, 160.5, 151.3, 138.1, 136.6, 133.9, 133.2, 131.1, 130.6, 130.0, 129.0, 128.7, 128.5, 127.1, 126.9, 126.8, 122.3. HRMS (ESI) calcd for C₂₀H₁₃ClN₂ ([M+H]⁺) 317.0856, found 317.0847.

4-(3,4-dichlorophenyl)-2-phenylquinazoline (3o)



White solid, isolated yield 92%, m.p. 165-168 °C. ^1H NMR (400MHz, CDCl_3): δ = 8.67 (d, J = 7.0 Hz, 2H), 8.17 (d, J = 8.4 Hz, 1H), 8.08 – 7.96 (m, 2H), 7.91 (t, J = 7.4 Hz, 1H), 7.70 (dd, J = 19.3, 8.0 Hz, 2H), 7.62-7.48 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 175.4, 165.7, 160.2, 152.1, 137.8, 137.5, 134.3, 133.9, 131.9, 130.7, 130.5, 129.4, 129.3, 128.6, 128.6, 127.4, 126.1, 121.2. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{12}\text{Cl}_2\text{N}_2$ ($[\text{M}+\text{H}]^+$) 351.0443, found 351.0449.

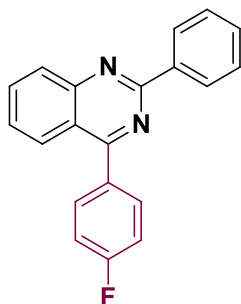
4-(4-bromophenyl)-2-phenylquinazoline^[3] (3p)



White solid, isolated yield 87%, m.p. 165-168 °C. ^1H NMR (400MHz, CDCl_3): δ = 8.73 (d, J = 7.9 Hz, 2H), 8.21 (d, J = 8.4 Hz, 1H), 8.10 (d, J = 8.4 Hz, 1H), 7.93 (t, J = 7.7 Hz, 1H), 7.80 (q, J = 8.8 Hz, 4H), 7.63-7.53 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ = 167.0, 160.2, 152.0, 138.0, 136.5, 133.7, 131.8, 131.7, 130.6, 129.3, 128.6, 128.5, 127.2,

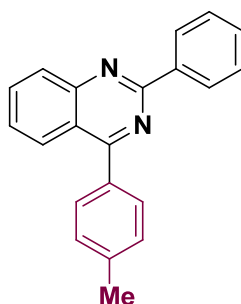
126.5, 124.6, 121.4.

4-(4-fluorophenyl)-2-phenylquinazoline^[3] (3q)



White solid, isolated yield 90%, m.p. 165-168 °C. ¹H NMR (400MHz, CDCl₃): δ = 8.74 (d, J = 7.8 Hz, 2H), 8.21 (d, J = 8.4 Hz, 1H), 8.13 (d, J = 8.4 Hz, 1H), 7.96-7.91 (m, 3H), 7.63-7.52 (m, 4H), 7.34 (t, J = 8.5 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 167.1, 165.2, 162.7, 160.1, 152.0, 138.1, 133.8, 133.6, 132.2, 132.1, 130.5, 129.2, 128.6, 128.6, 128.4, 127.1, 126.7, 121.5, 115.8, 115.5.

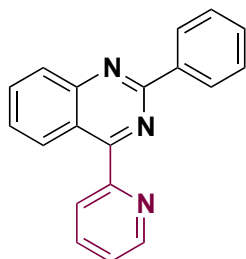
2-phenyl-4-(p-tolyl)quinazoline^[1] (3r)



Pale yellow solid, isolated yield 75%, m.p. 115-117 °C. ¹H NMR (400MHz, CDCl₃): δ = 8.75 (d, J = 7.6 Hz, 2H), 8.20 (d, J = 8.5 Hz, 2H), 7.93 (t, J = 7.7 Hz, 1H), 7.86 (d, J = 7.9 Hz, 2H), 7.63-7.52 (m, 4H), 7.46 (d, J = 7.7 Hz, 2H), 2.55 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 168.3, 160.2, 152.0, 140.1, 138.3, 133.4, 130.4, 130.2, 129.2, 129.1, 128.6,

128.5, 127.1, 126.8, 121.7, 21.4.

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

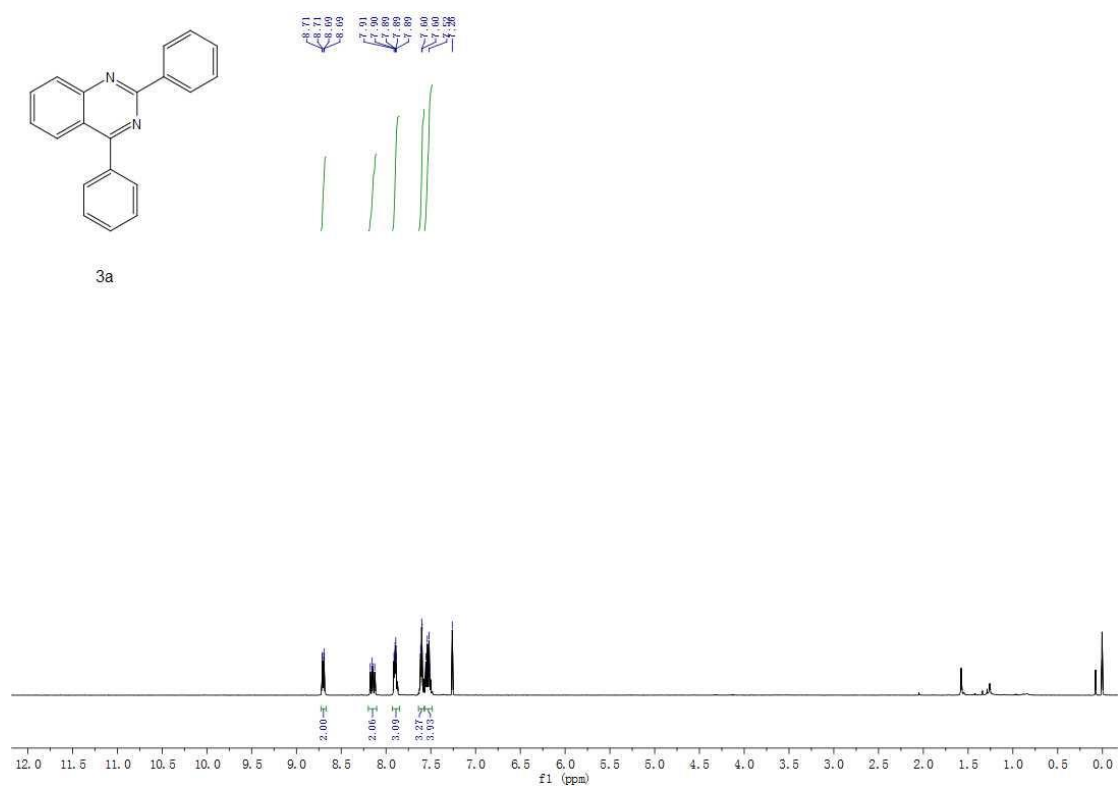


White solid, isolated yield 71%, m.p. 162-163 °C. ^1H NMR (400MHz, CDCl_3): δ = 8.92 (dd, J = 8.5, 0.8 Hz, 1H), 8.85 (dd, J = 4.8, 0.8 Hz, 1H), 8.72 (dd, J = 8.1, 1.6 Hz, 2H), 8.39 (d, J = 7.9 Hz, 1H), 8.18-8.12 (m, 1H), 7.98 (td, J = 7.8, 1.8 Hz, 1H), 7.90 (ddd, J = 8.4, 6.9, 1.4 Hz, 1H), 7.64-7.46 (m, 5H); ^{13}C NMR (100MHz, CDCl_3): δ = 164.1, 159.8, 156.9, 152.5, 148.6, 138.0, 137.1, 133.5, 130.4, 128.9, 128.5, 127.8, 127.4, 125.4, 124.3, 121.4. HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{13}\text{N}_3$ ($[\text{M}+\text{H}]^+$) 284.1332, found 284.1346.

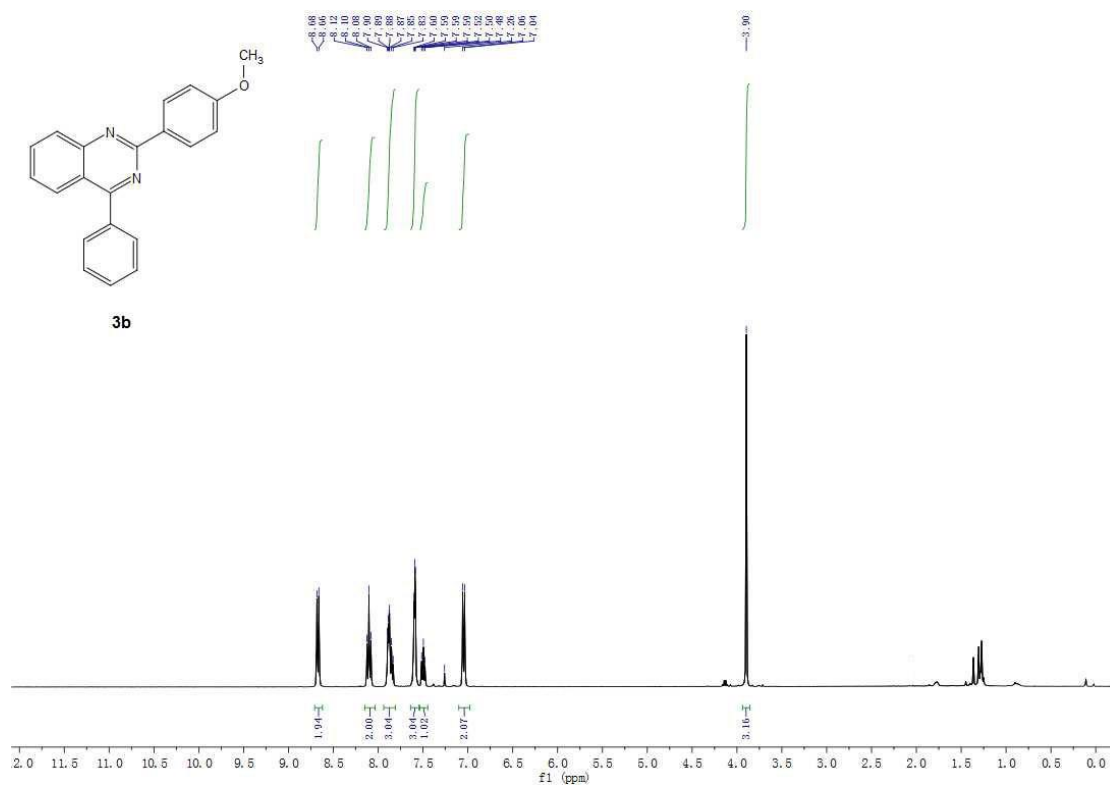
Reference:

- [1] W. Zhang, F. Guo, F. Wang, N. Zhao, L. Liu, J. Li, Z. Wang, *Org. Biomol. Chem.*, 2014, **12**, 5752-5756.
- [2] L. Tang, Y. Yang, L. Wen, S. Zhang, Z. Zha, Z. Wang, *Org. Chem. Front.*, 2015, **2**, 114-118.
- [3] J. Zhang, C. Yu, S. Wang, C. Wan, Z. Wang, *Chem. Commun.*, 2010, **46**, 5244-5246.

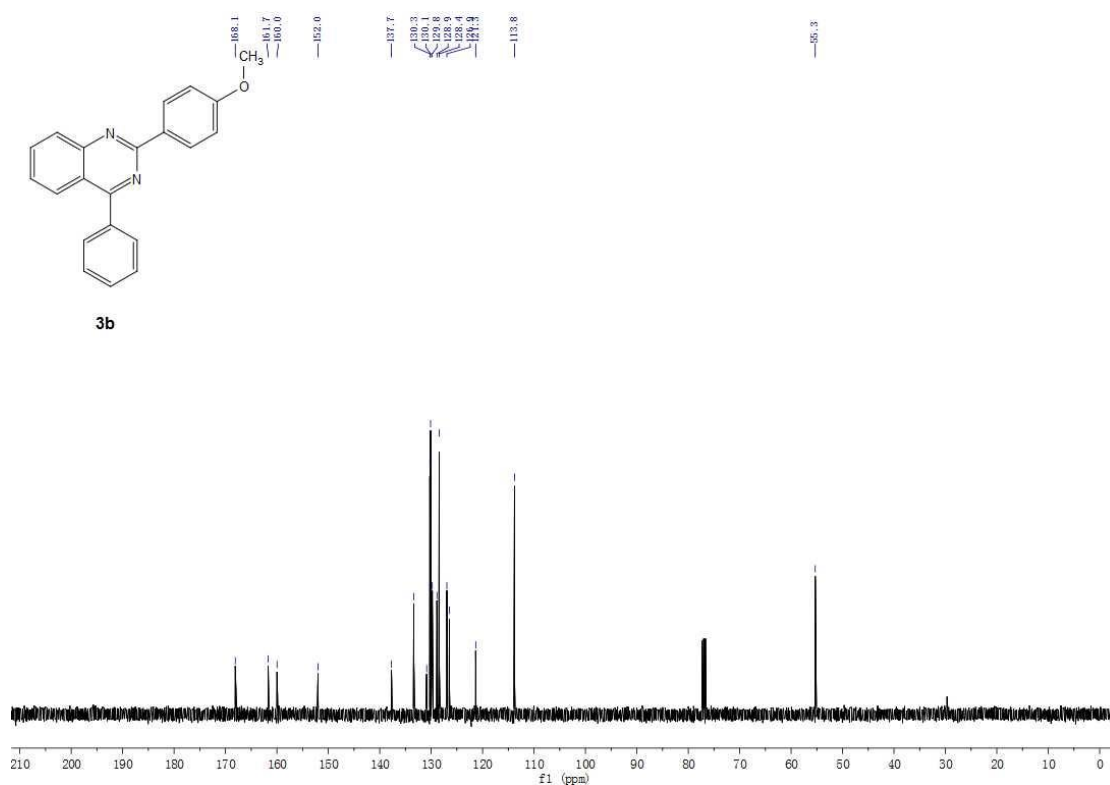
5. Copy of ^1H NMR and ^{13}C NMR Spectra for Products



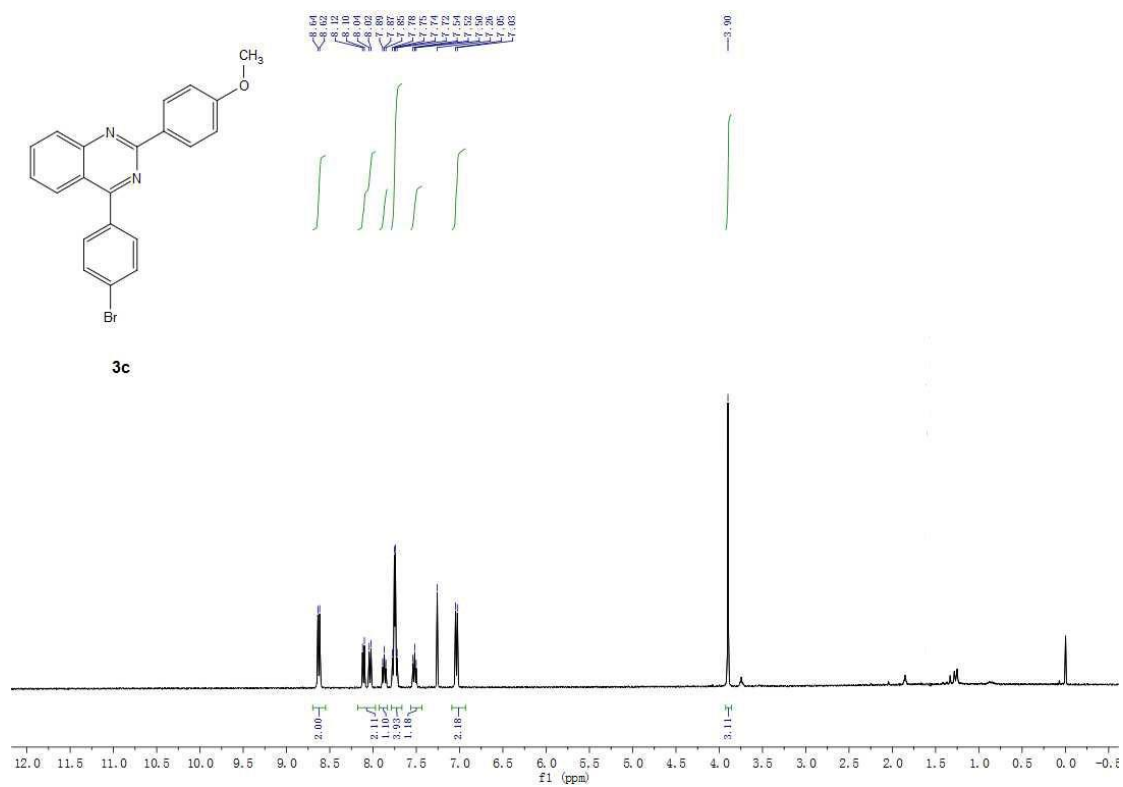
^1H NMR of **3a**



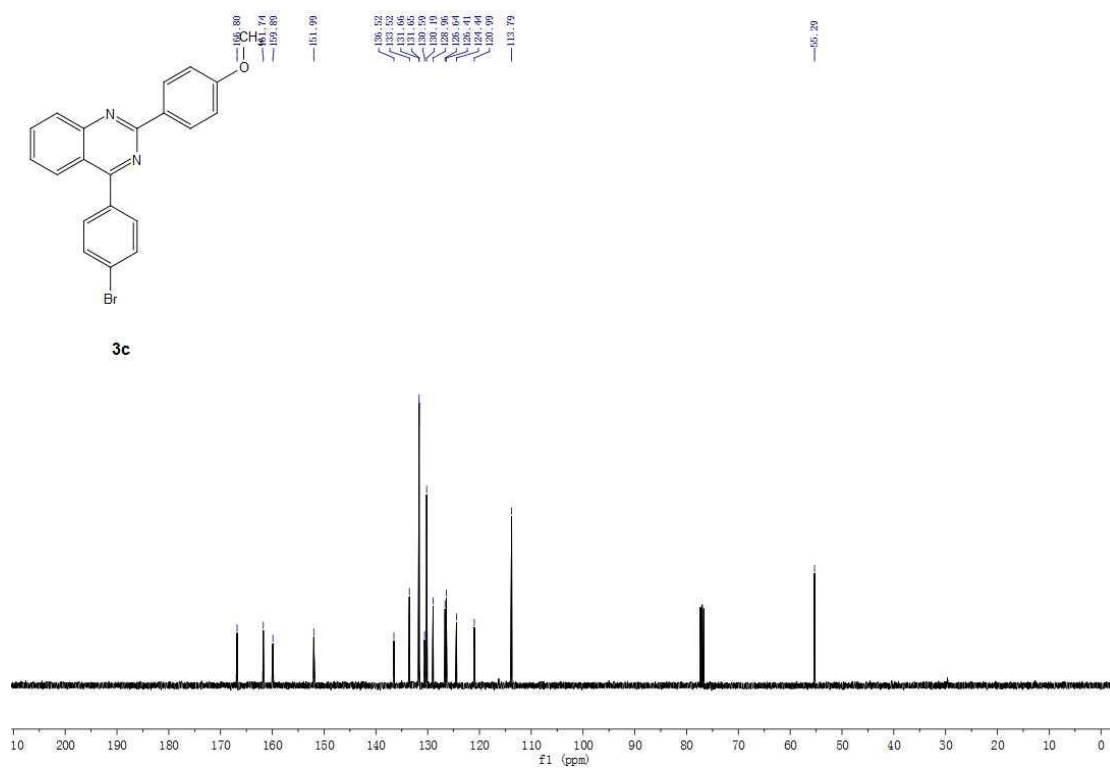
^1H NMR of **3b**



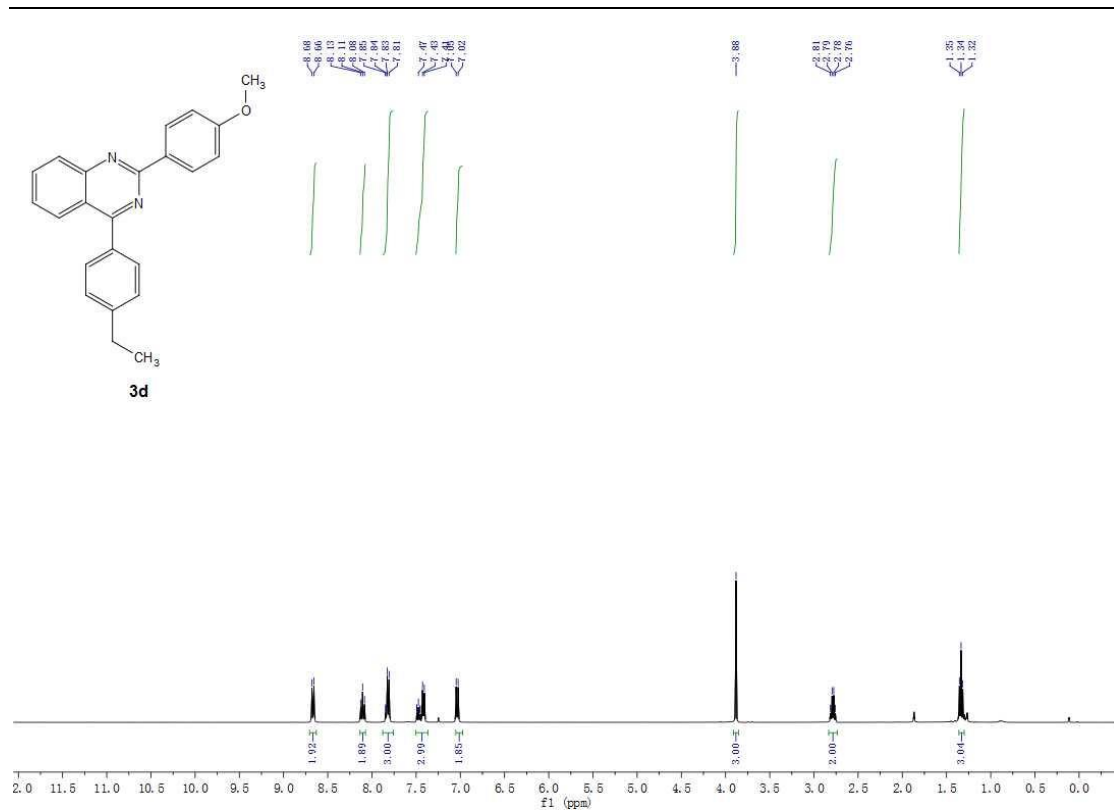
^{13}C NMR of **3b**



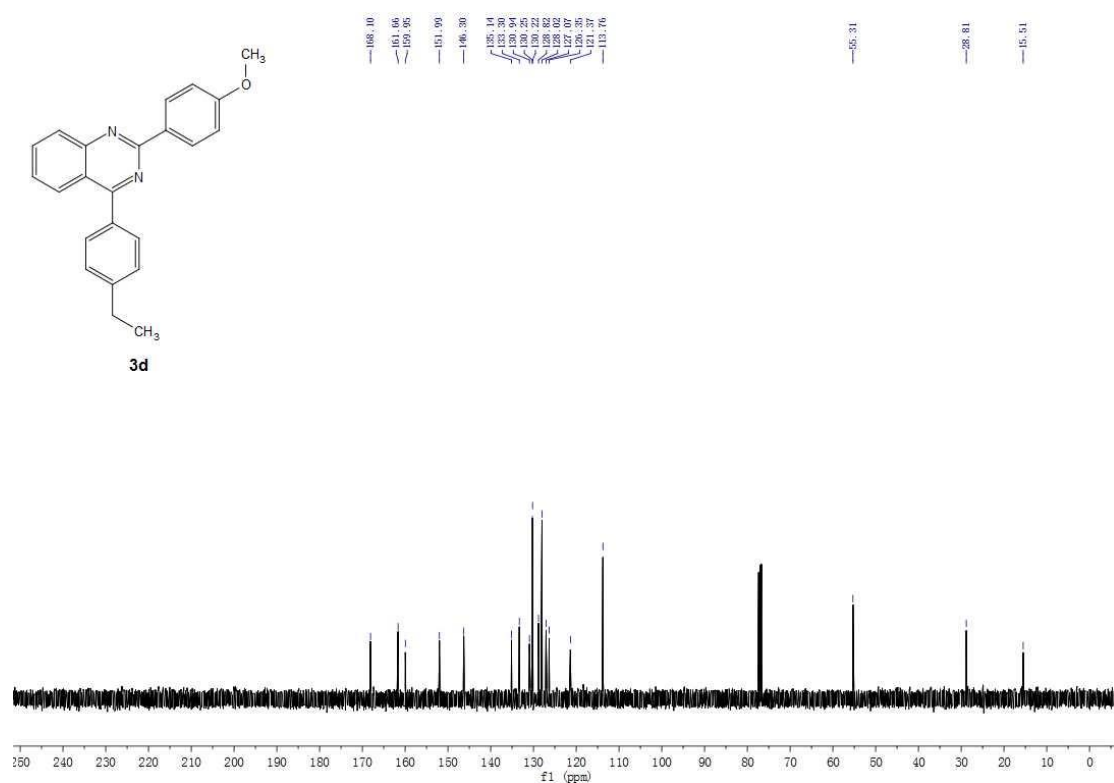
^1H NMR of **3c**



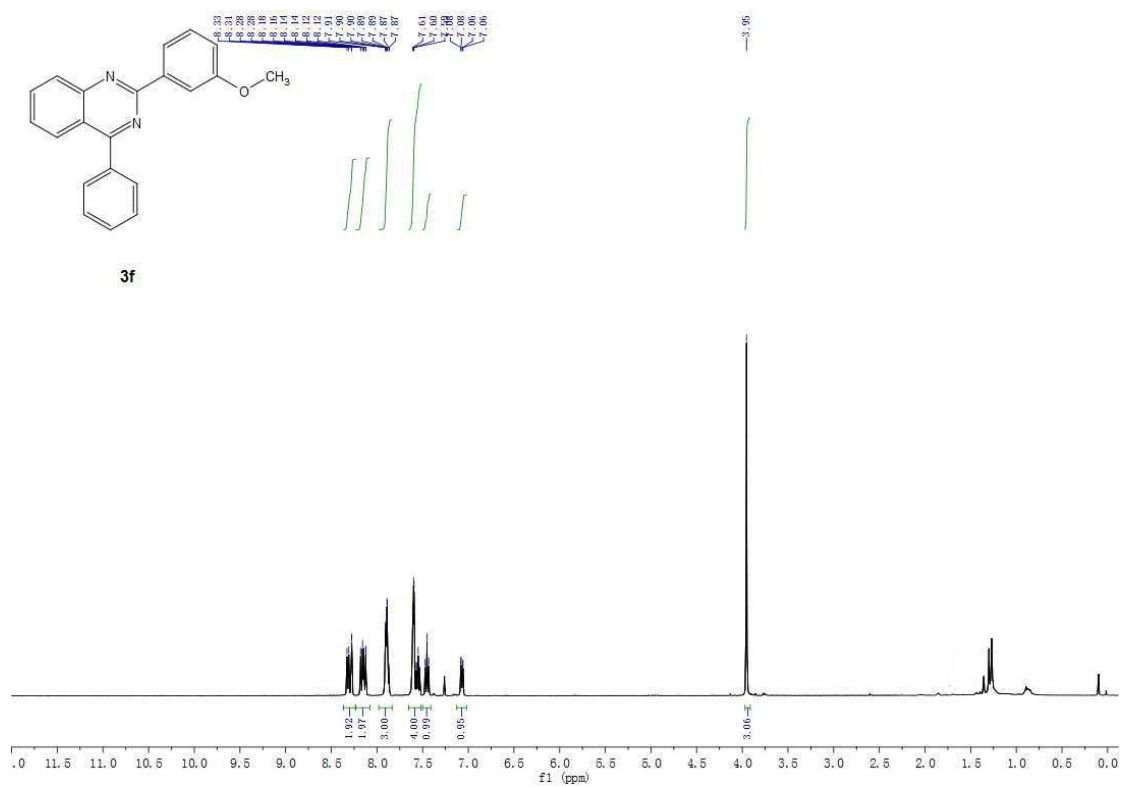
^{13}C NMR of **3c**



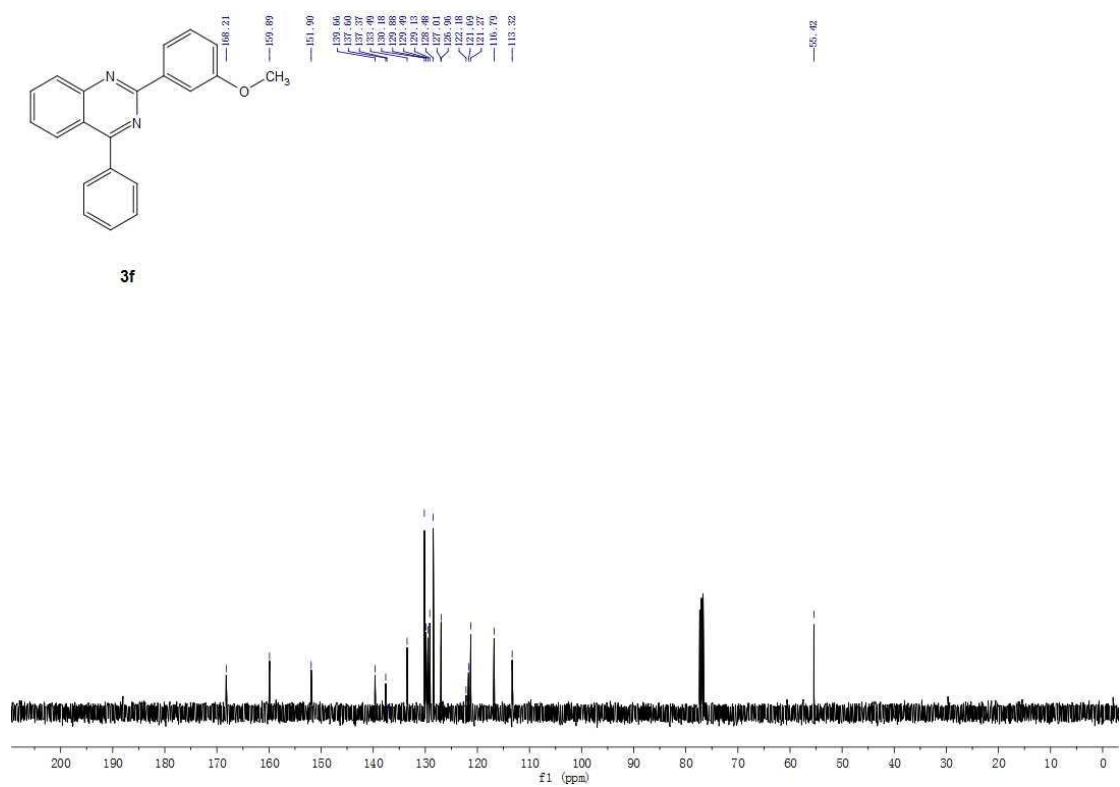
^1H NMR of **3d**



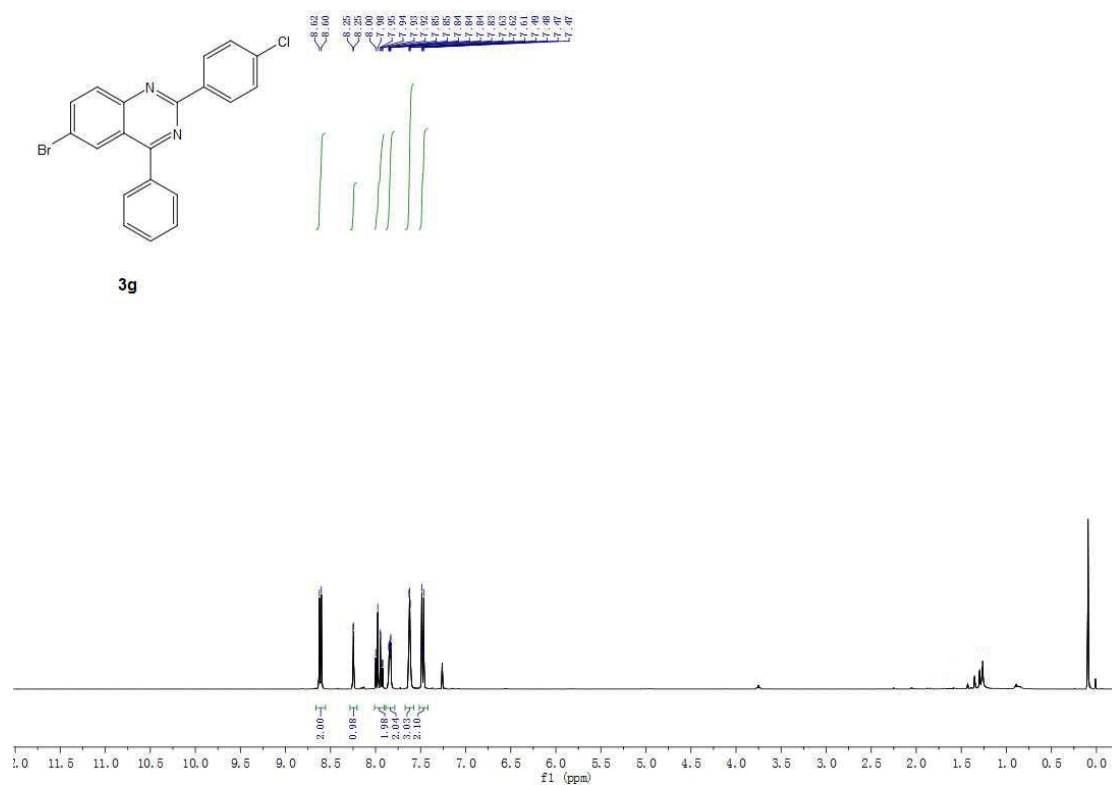
^{13}C NMR of **3d**



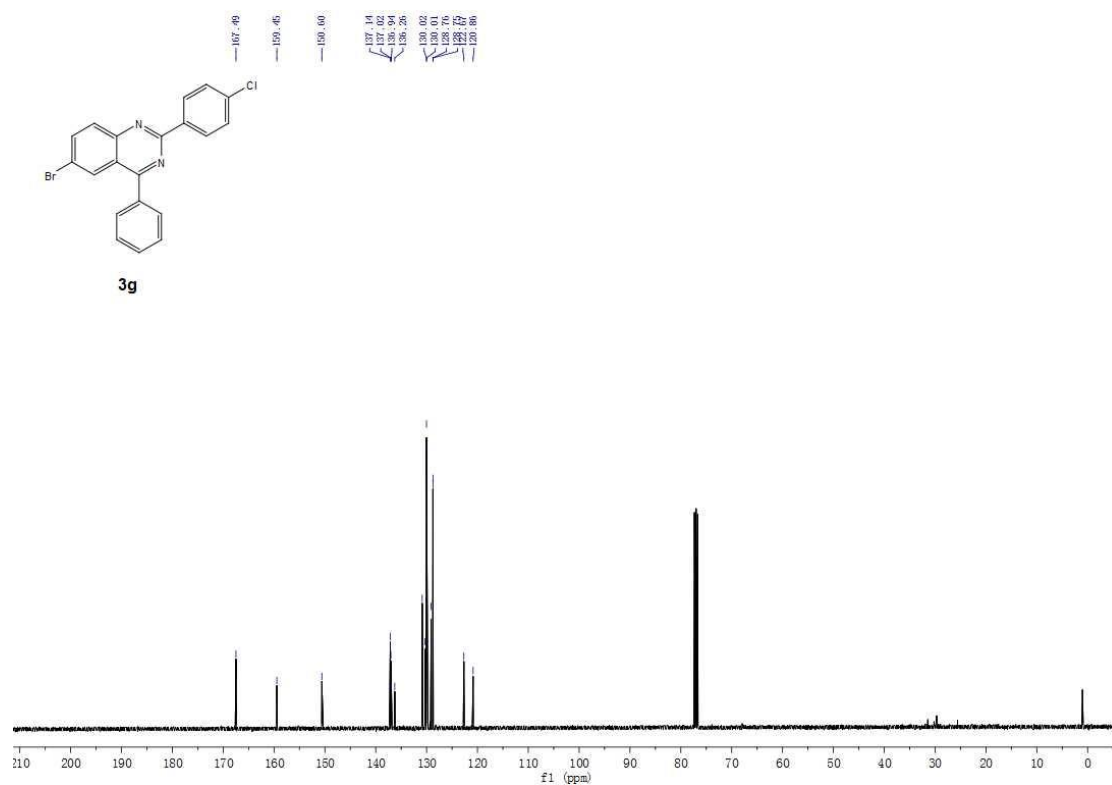
¹H NMR of **3f**



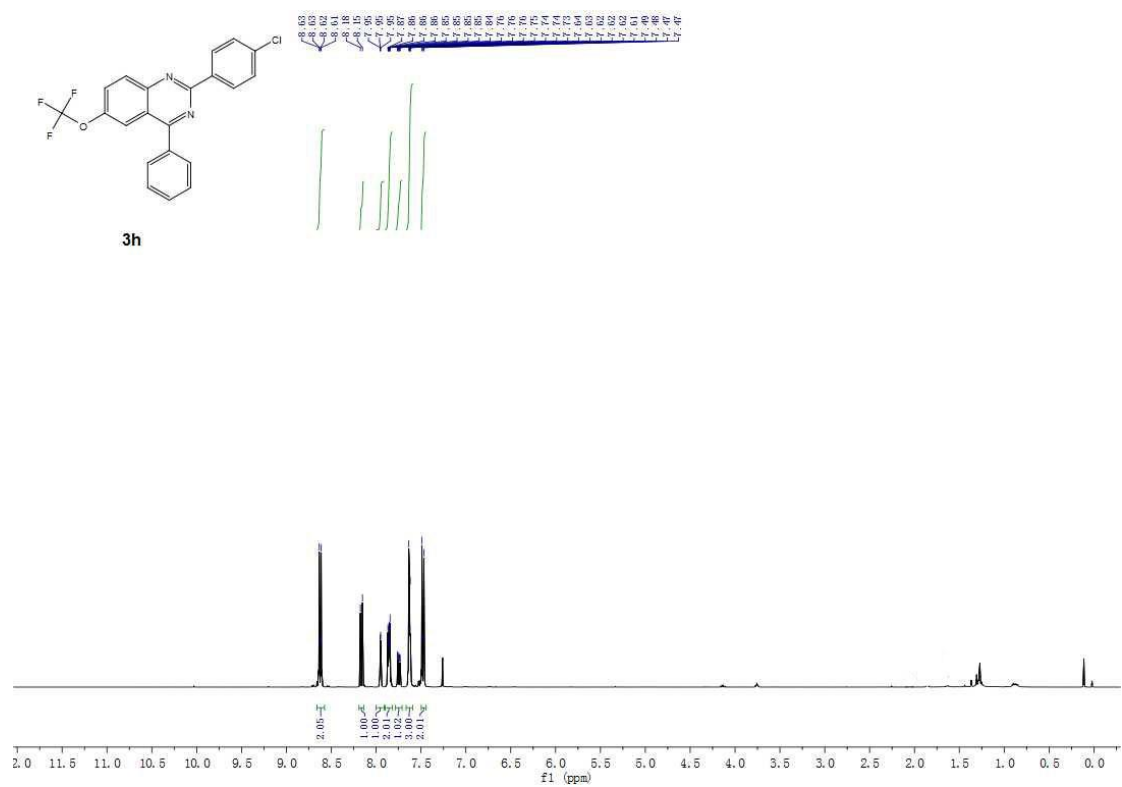
¹³C NMR of **3f**



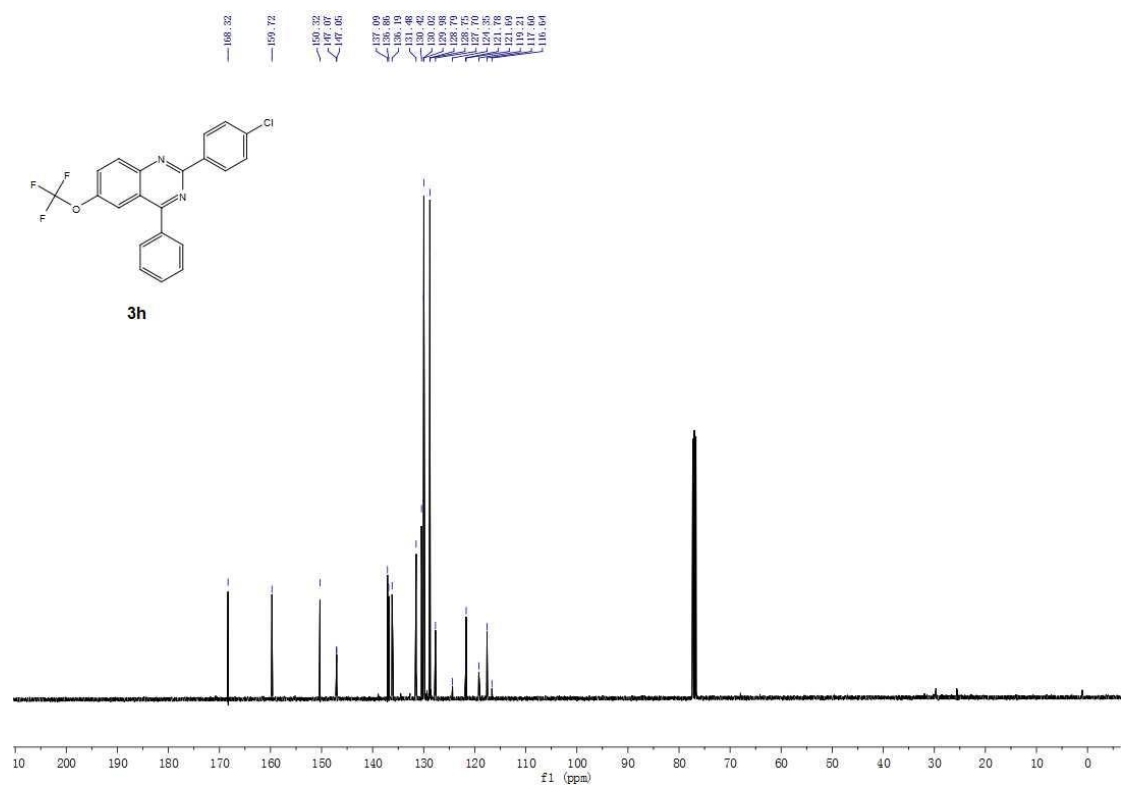
^1H NMR of **3g**



^{13}C NMR of **3g**

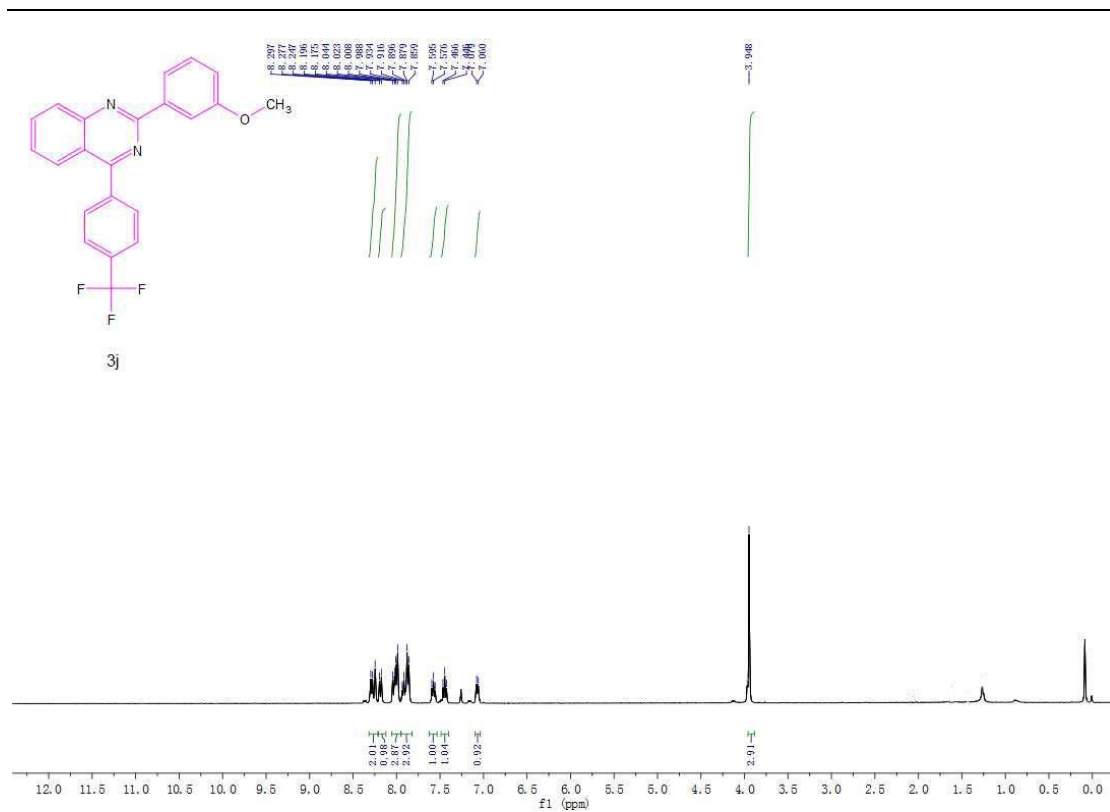


^1H NMR of **3h**

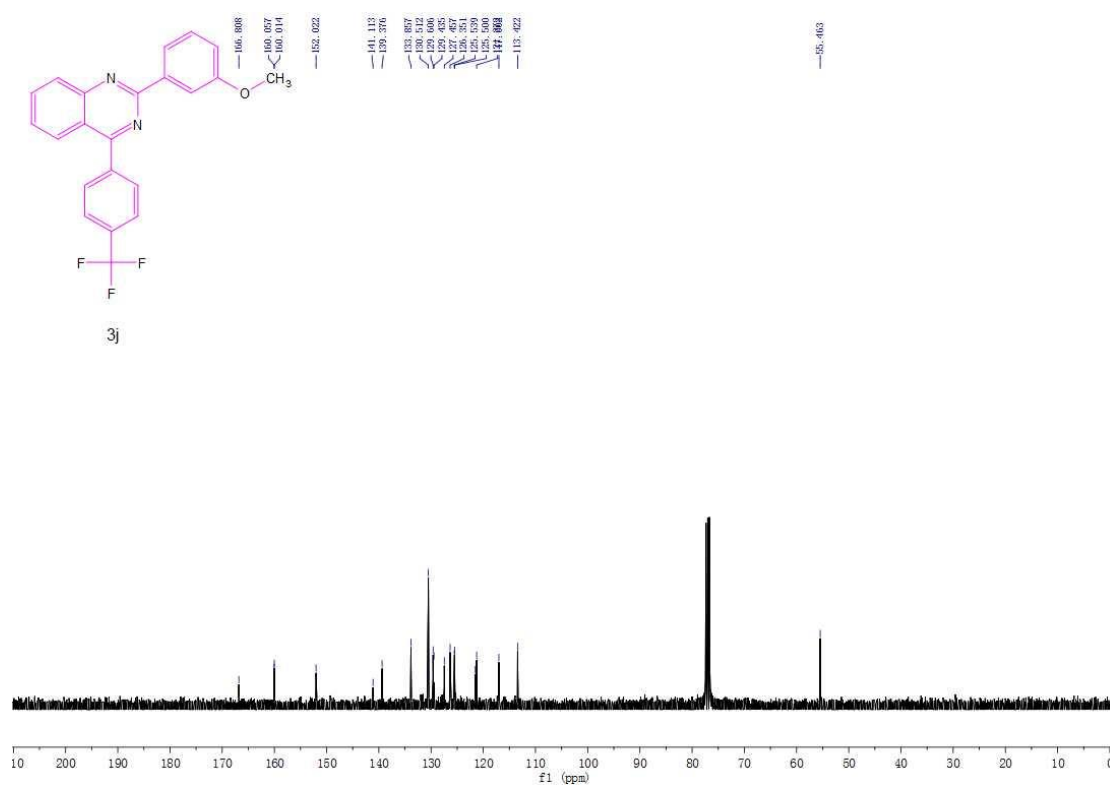


^{13}C NMR of **3h**

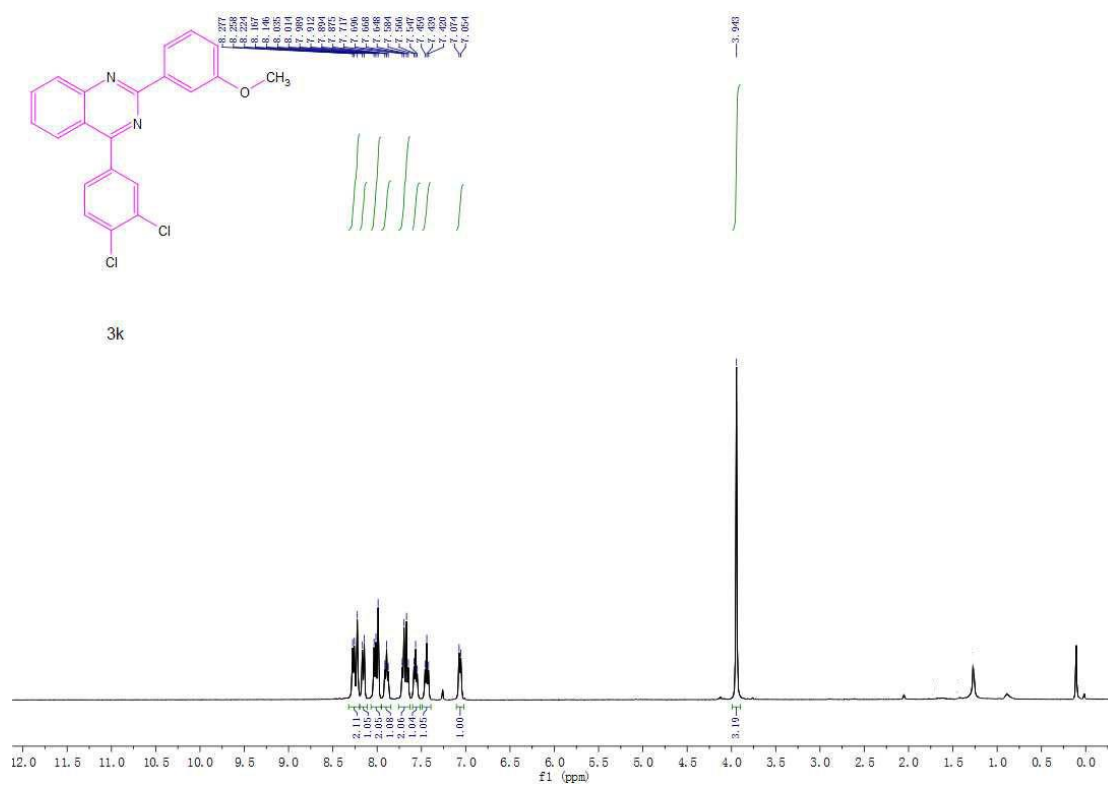




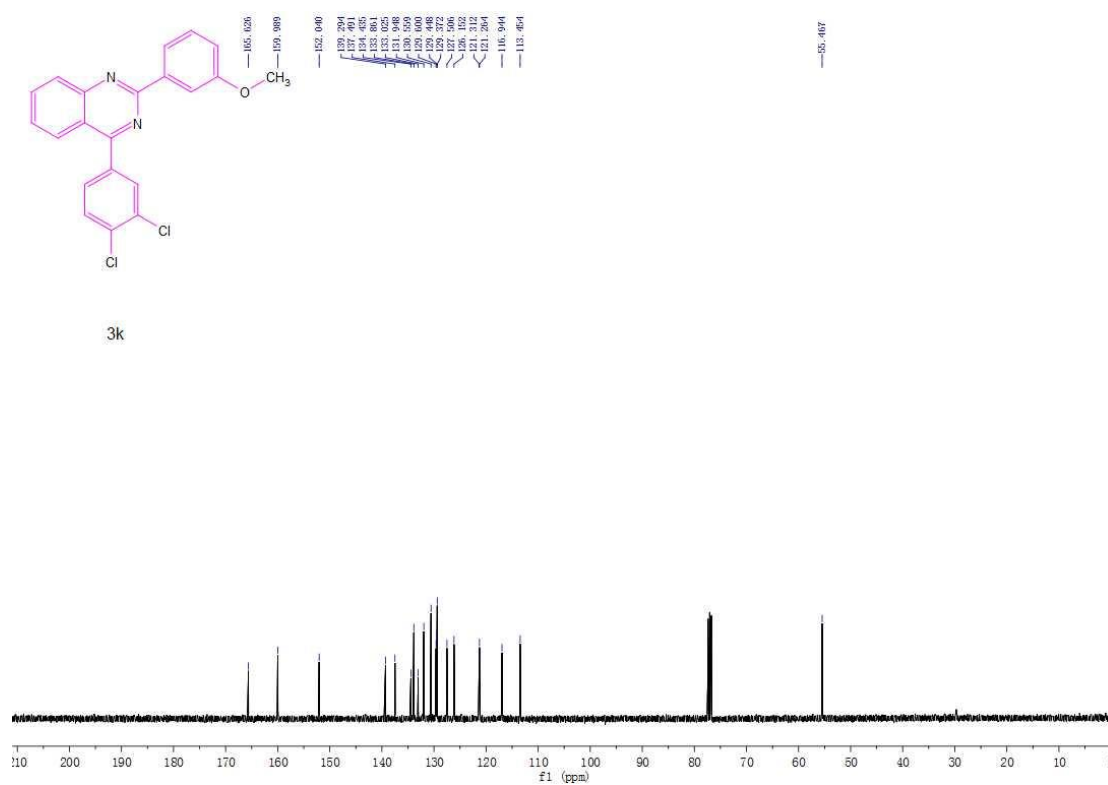
¹H NMR of **3j**



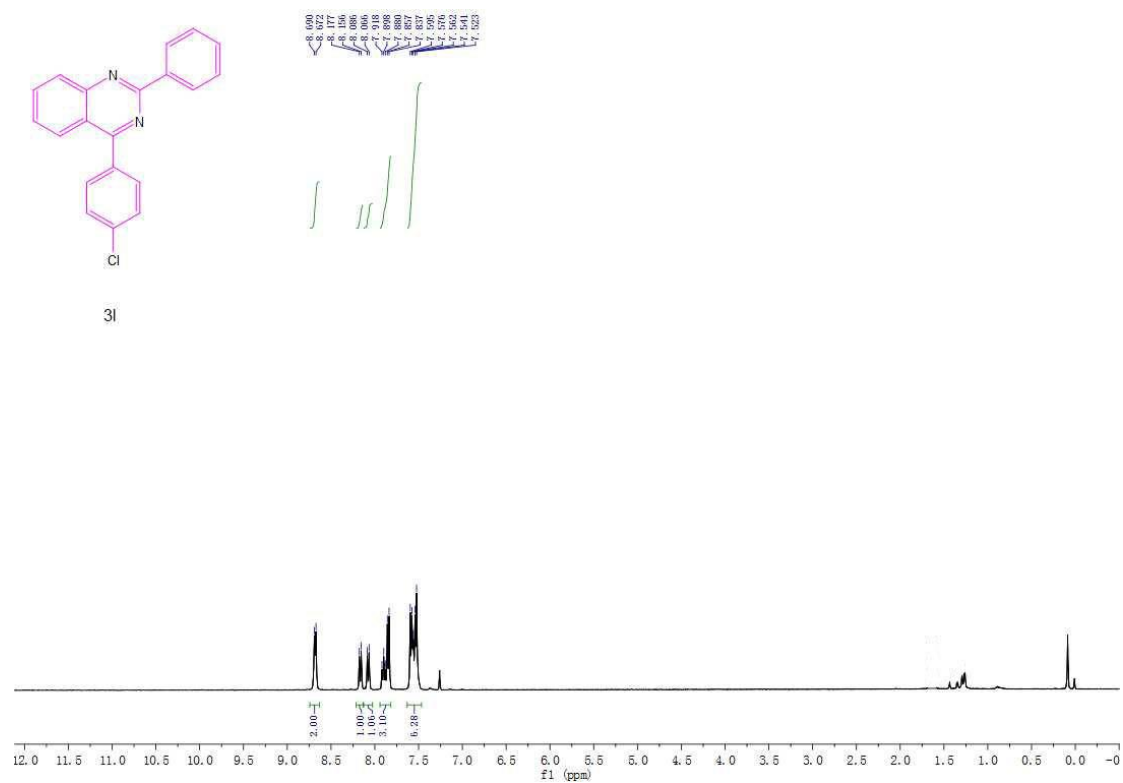
¹³C NMR of **3j**



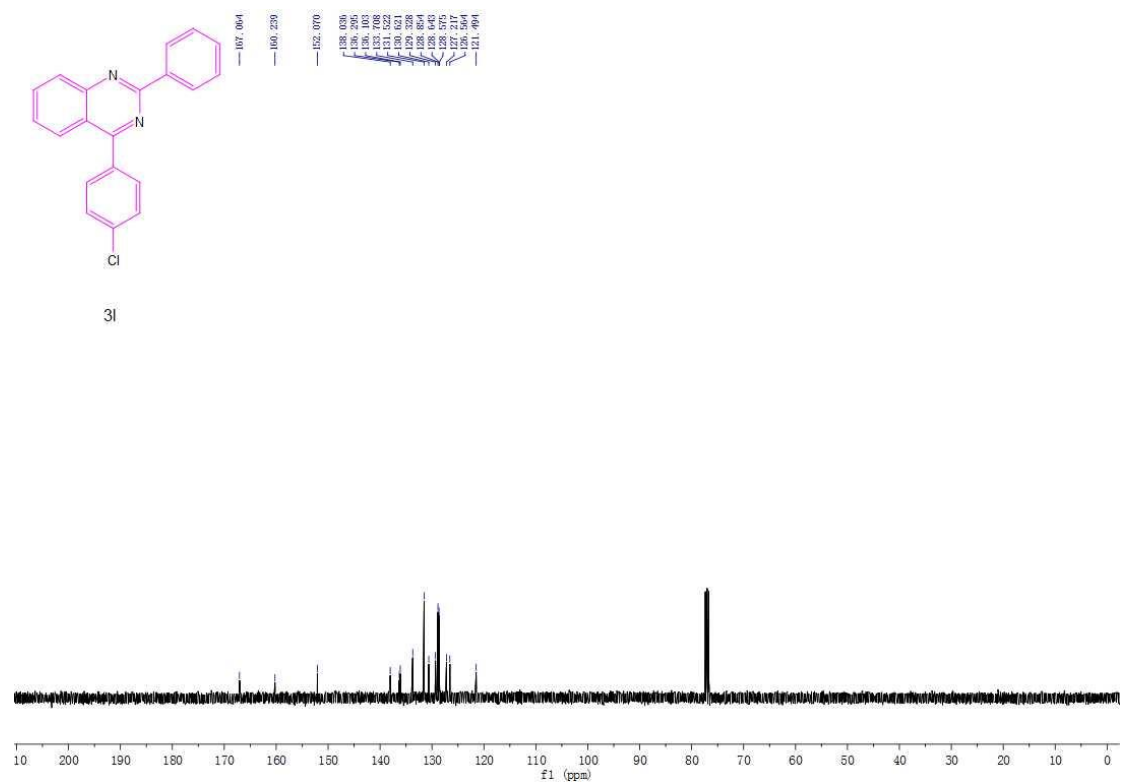
^1H NMR of **3k**



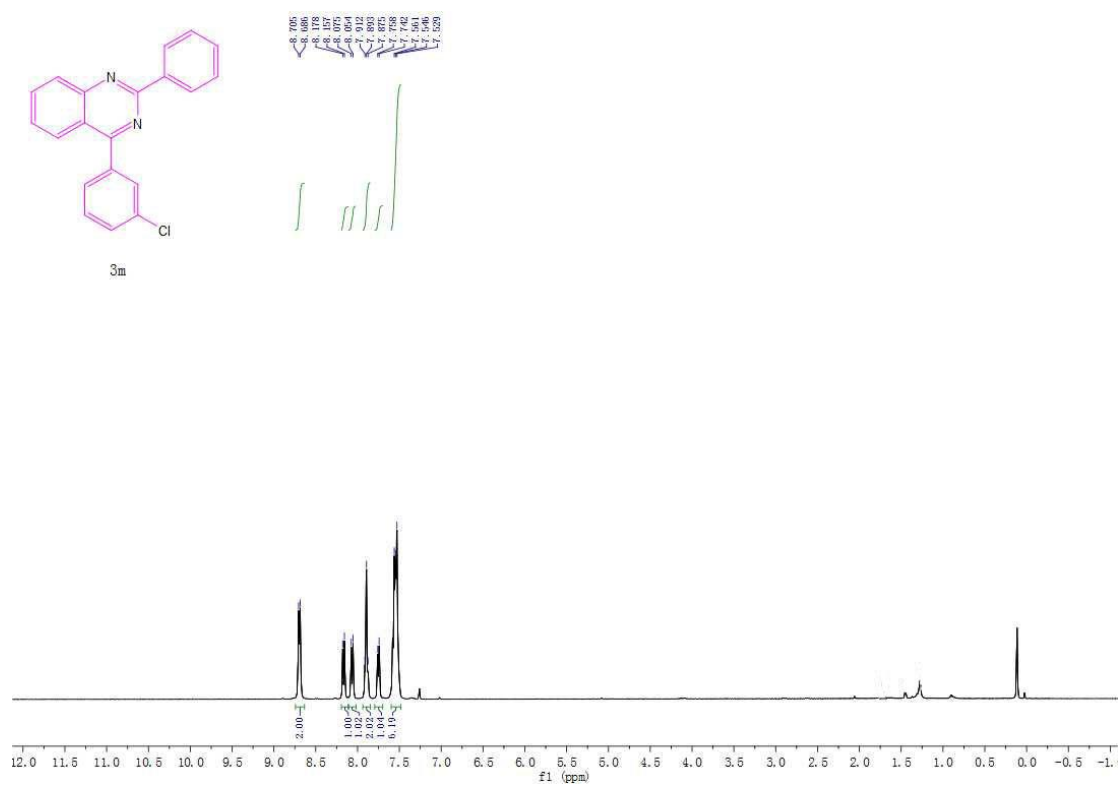
^{13}C NMR of **3k**



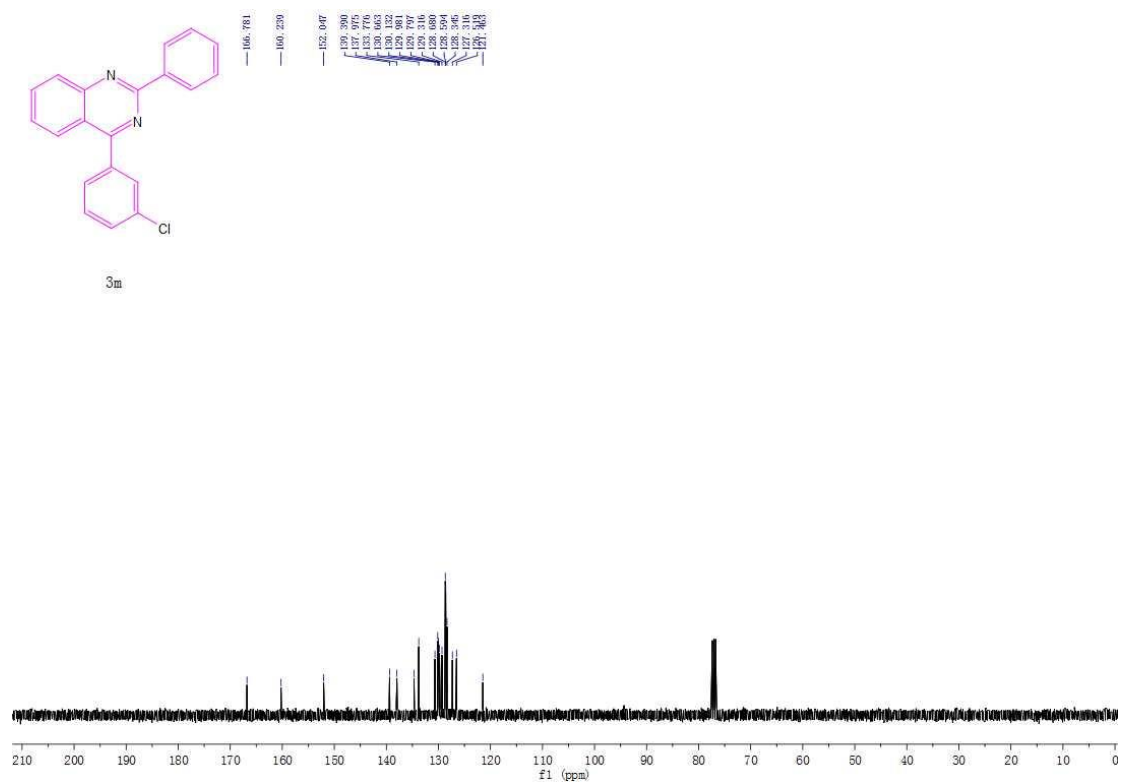
^1H NMR of **3I**



^{13}C NMR of **3I**

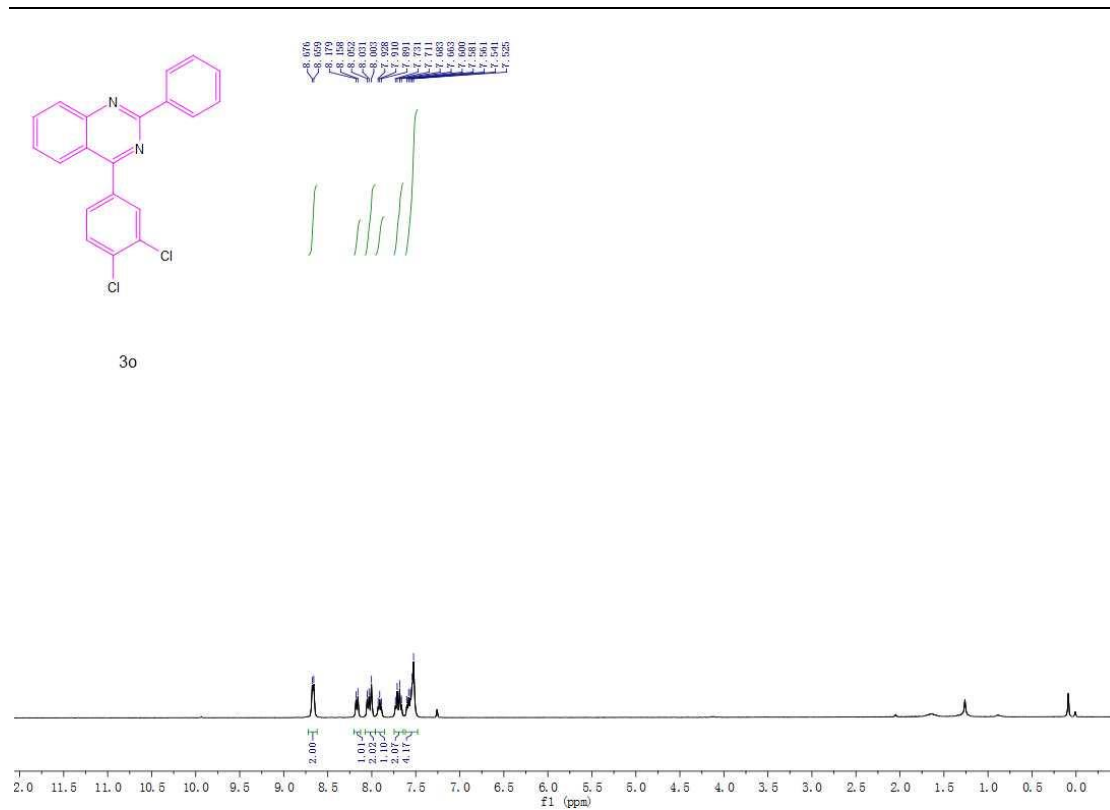


^1H NMR of **3m**

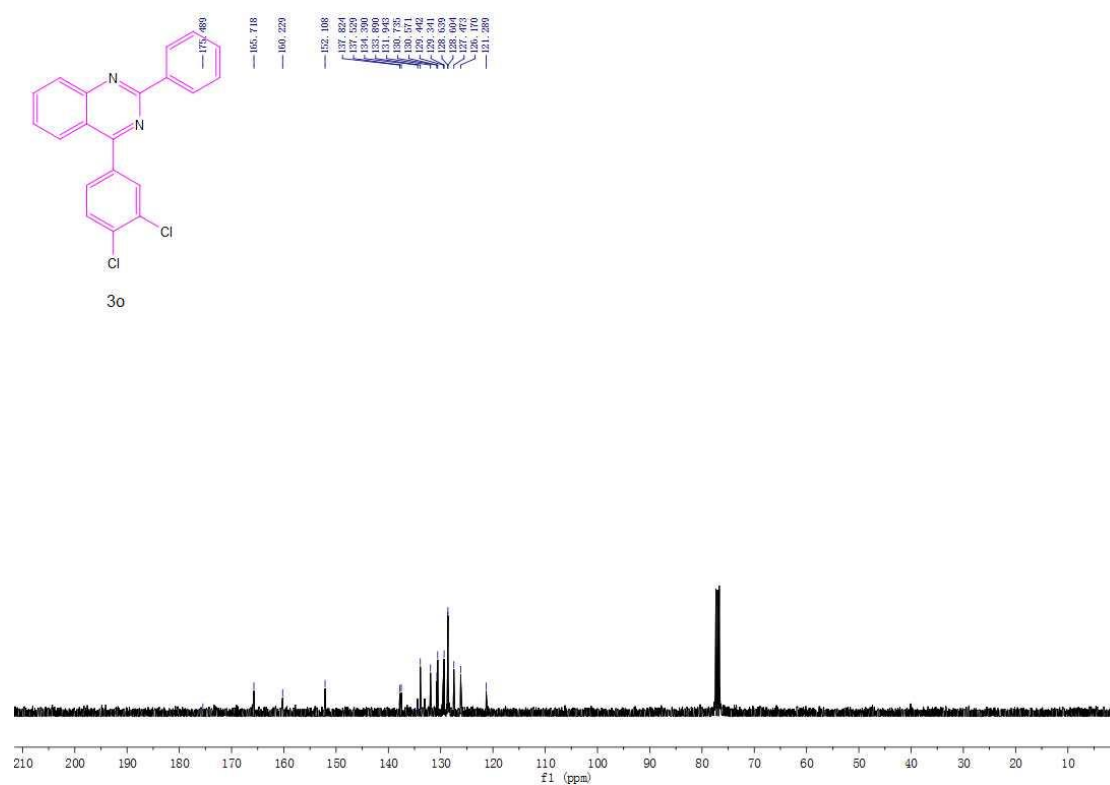


^{13}C NMR of **3m**

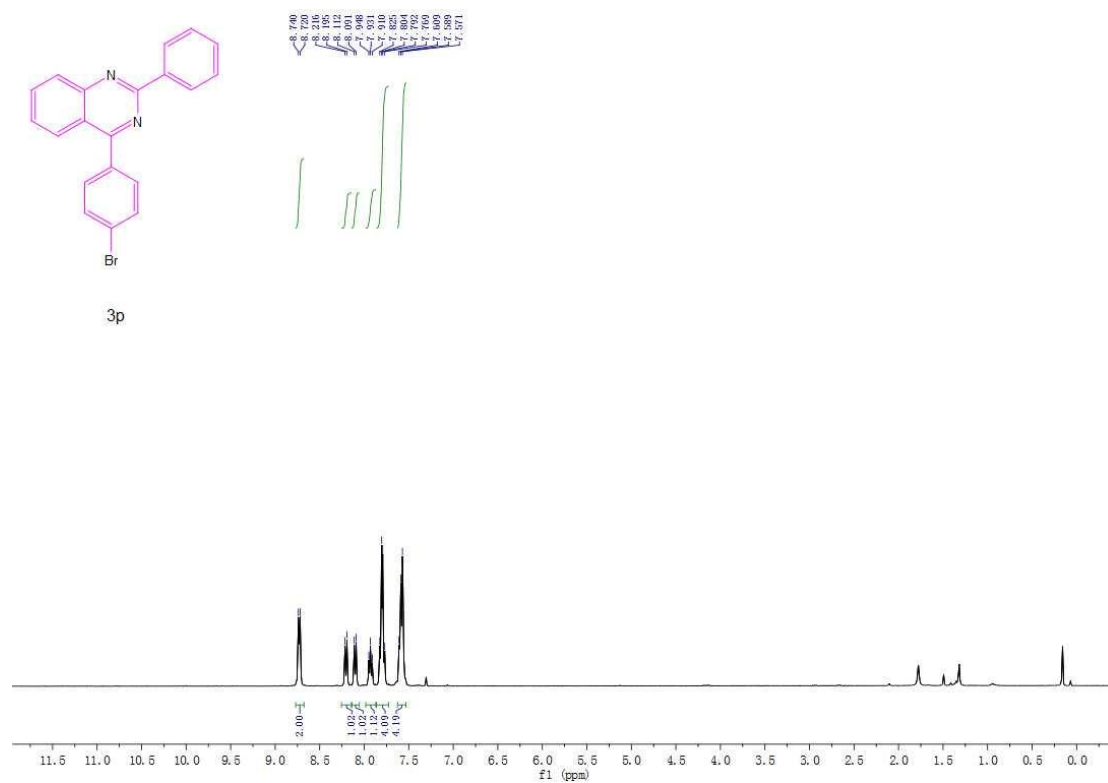




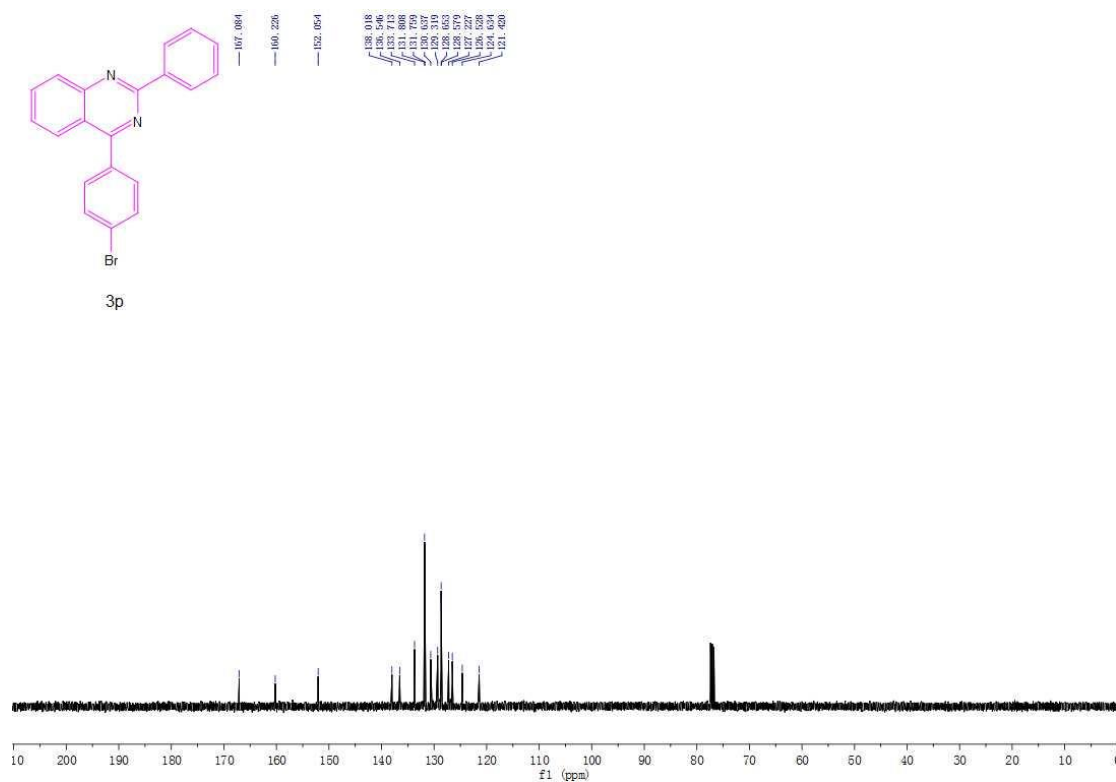
¹H NMR of **3o**



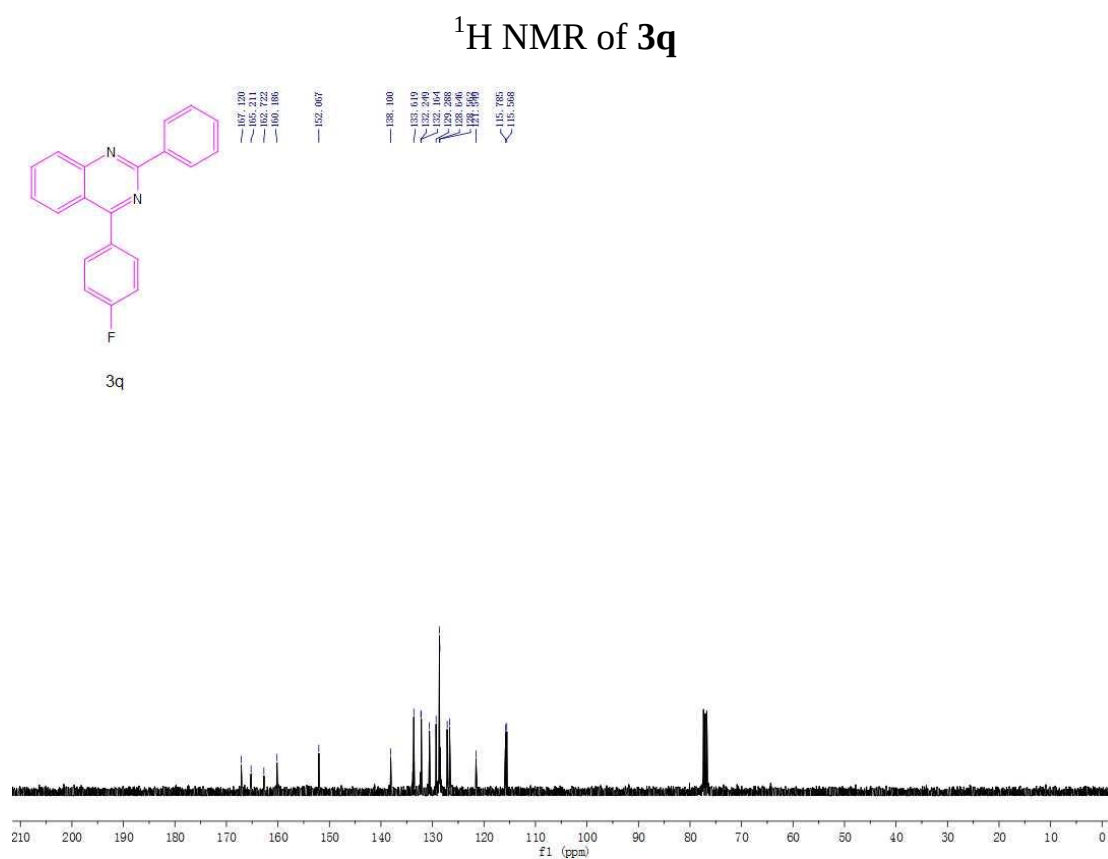
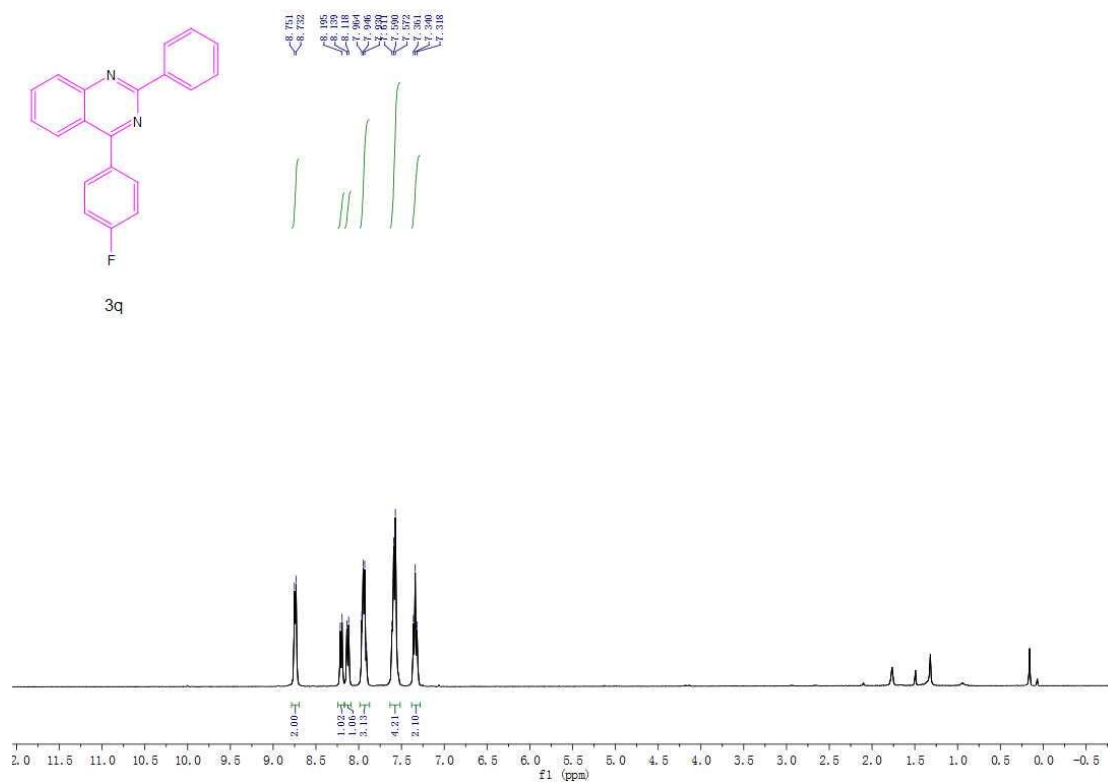
¹³C NMR of **3o**

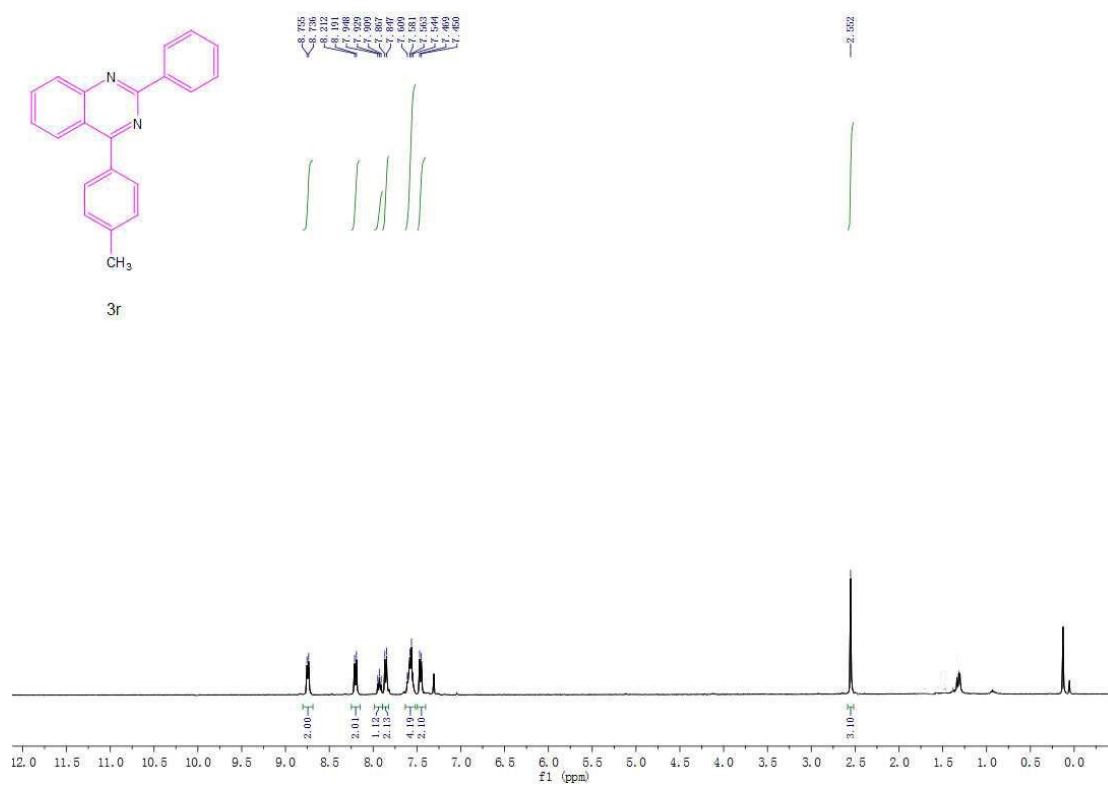


^1H NMR of **3p**

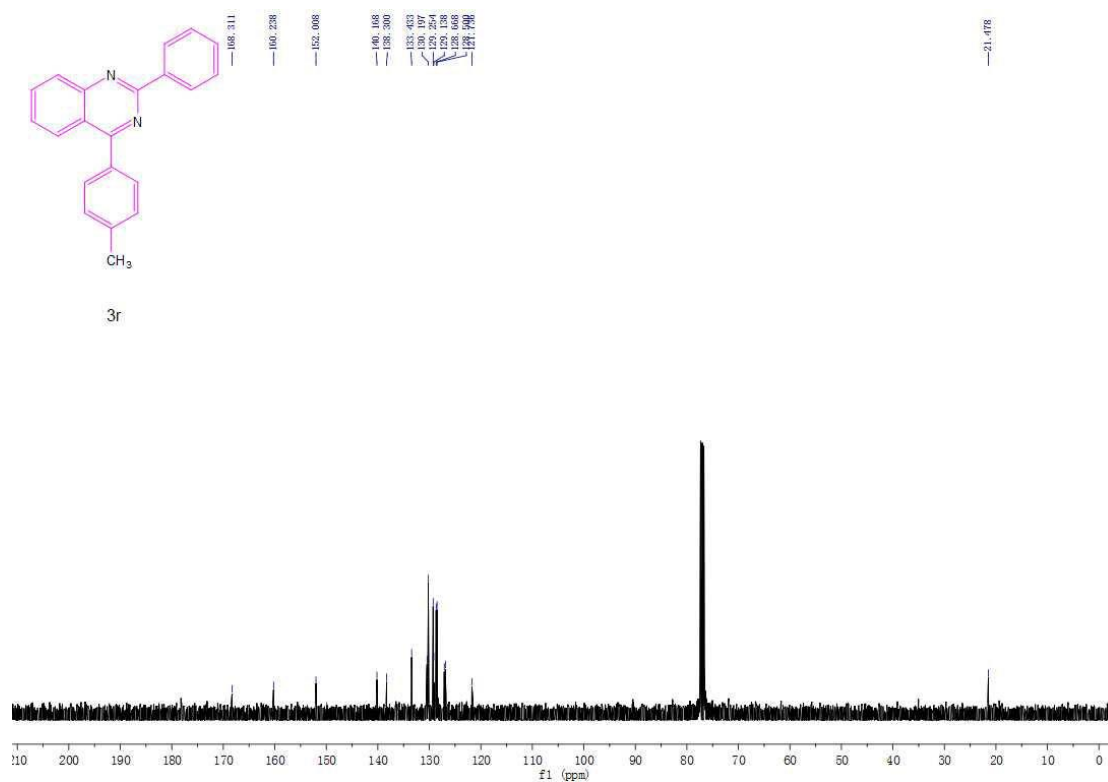


^{13}C NMR of **3p**

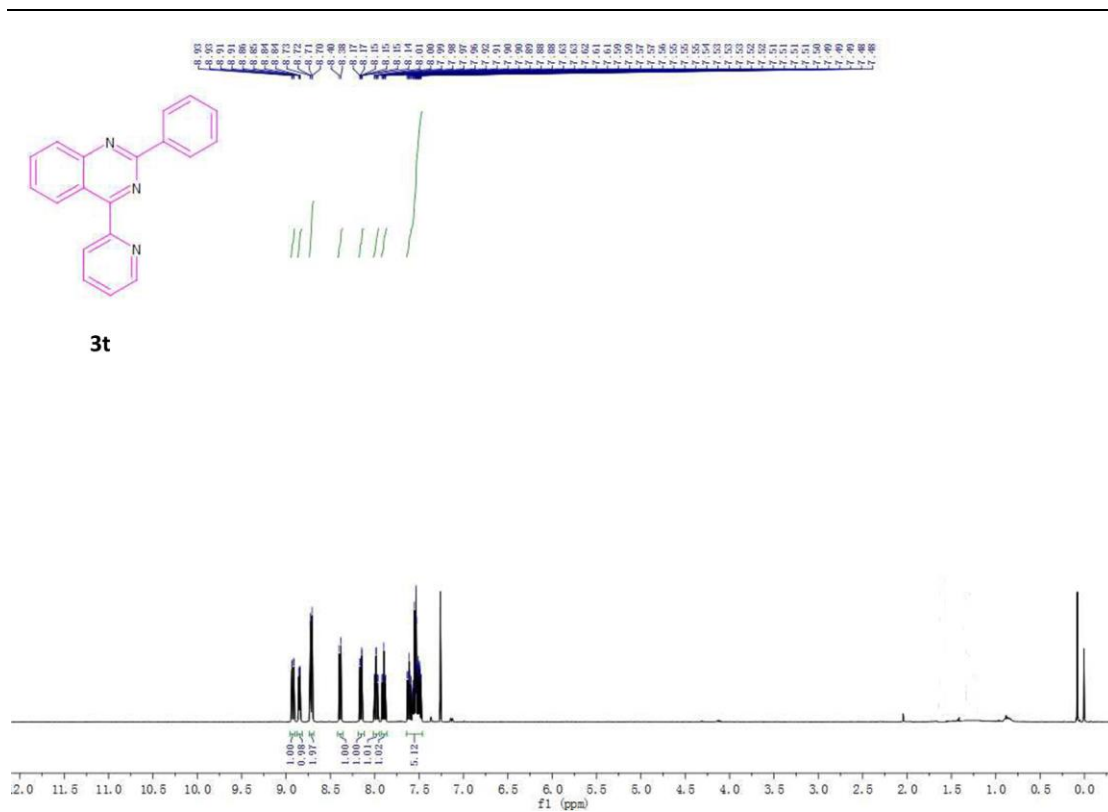




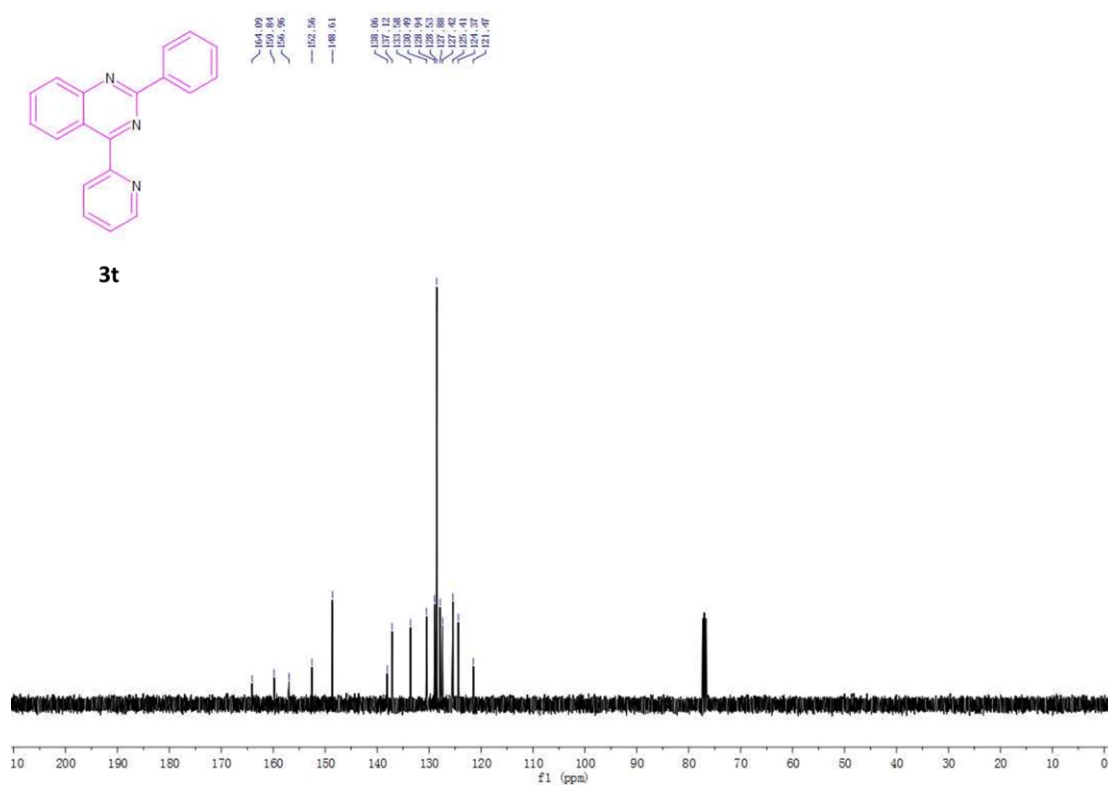
¹H NMR of **3r**



¹³C NMR of **3r**



^1H NMR of **3t**



^{13}C NMR of **3t**