Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry. This journal is © The Royal Society of Chemistry 2024

Supplementary Information

Tempo Promoted Tandem Reaction of 2-

Aminobenzophenones and Benzylamines under

Electrochemical Conditions

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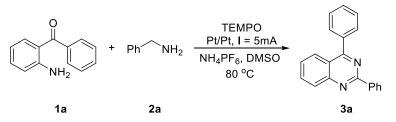
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Part I Experiment Section

1.1 General Information

¹H NMR and ¹³C NMR were recorded on a 500 MHz Nuclear Magnetic Resonance Spectrometer (¹H NMR: 500 MHz, ¹³C NMR: 126 MHz) using TMS as the internal reference. Chemical shifts were expressed as δ with respect to TMS and coupling constants *J* are expressed in Hz. HRMS (ESI) were recorded on a Q-TOF Premier. Commercially available compounds were used without further purification. Solvents were purified according to the standard procedures unless otherwise noted.

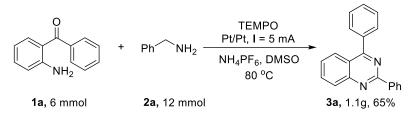
1.2 General Procedures for the Synthesis of Quinazolines



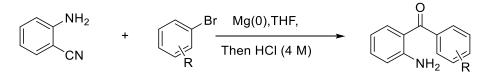
1a (2-amino benzophenone,0.3 mmol, 59.2 mg), ammonium hexafluorophosphate (NH₄PF₆, 0.6 mmol, 97.8 mg), and TEMPO (2,2,6,6-tetramethylpiperidinooxy, 0.06 mmol, 4.7 mg) were added to a 10 mL reaction tube, followed by addition of 3 mL dimethylsulfoxide using a plastic pipette. Then add **2a** (benzylamine, 0.6mmol, 65.5 μ L) with a microsyringe and use the Pt/Pt as electrodes. The reaction mixture was heated at 80 °C while stirring and monitoring the voltage. When the reaction was completed, the reaction solution was extracted three times with water and ethyl acetate, and the combined organic phases were dried over anhydrous sodium sulfate and evaporated in vacuo to obtain the crude product. The residue was purified by flash column chromatography (petroleum ether/ethyl acetate) to give **3a**.

1.3 Gram-scale experiment

1a (2-aminobenzophenone, 6 mmol, 1.183 g), NH₄PF₆ (12 mmol, 1.956 g), and TEMPO (2,2,6,6-tetramethylpiperidinooxy, 1.2 mmol, 94 mg) were added to a 100 mL reaction flask, followed by addition of 60 mL DMSO using a plastic pipette. Then add 2a (benzylamine, 12 mmol, 1.31 mL) with a pipette and use the Pt/Pt as electrodes. The reaction mixture was heated at 80 °C while stirring and monitoring the voltage. When the reaction was completed, the reaction solution was extracted three times with water and ethyl acetate, and the combined organic phases were dried over anhydrous sodium sulfate and evaporated in vacuo to obtain the crude product. The residue was purified by flash column chromatography (petroleum ether/ethyl acetate) to give 3a (1.1g, 65%).

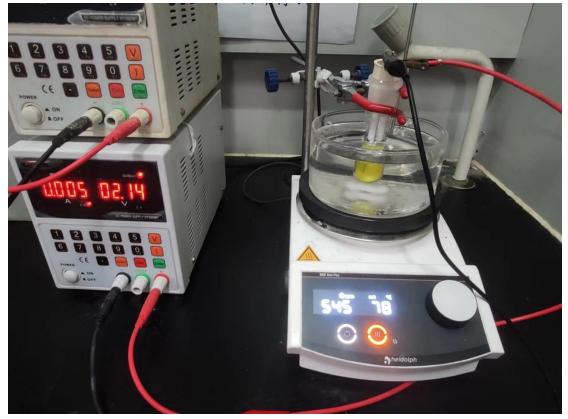


1.4 Steps in the synthesis of substrates



The magnesium strip (1.1 equiv.) and dry THF (1 M) were added to a flame-dried round-bottomed flask. Following the dropwise addition of aryl bromide (1.0 equiv.), the reaction mixture was kept under reflux conditions for 2 hours. Then 2-amino benzonitrile (0.2 to 0.25 eq.) was added. The reaction was heated to $60 \,^{\circ}\text{C}$ and continued for about 12 hours. After consumption of the material, the reaction was quenched with a solution of 4 M hydrochloric acid in EtOH at 0 $^{\circ}\text{C}$ and then heated at reflux overnight. Concentrated in a vacuum and quenched with saturated sodium bicarbonate followed by extraction with EtOAc (×3). The organic layers were combined, dried, and concentrated in anhydrous sodium sulfate to give the crude product. The crude mixture was then purified by column chromatography using petroleum ether/ethyl acetate as eluent to give the target product.

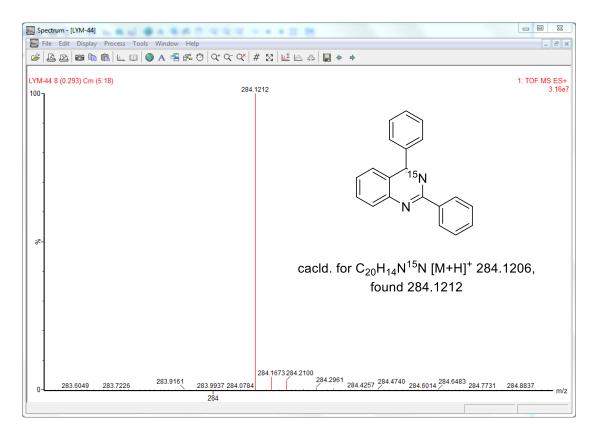
1.5 The electrochemical reaction setting and the parameters of the electrochemical reactor



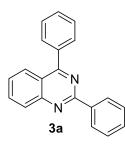
1.5.1 The picture of the electrochemical reaction setting

1.5.2 The parameters of the electrochemical reactor		
Model number	HY3005MT	
Output voltage	0-30VDC	
Output current	0-5VDC	
Ripple and noise	$CV \le 5 \text{ mV r ms}$ $CC \le 20 \text{ mA}$	
Input voltage	220VAC+10% 50HZ	
Power supply effect	CV≤0.1%+3 mV CC≤0.5%+3mA	
load effect	CV≤0.05%+3 mV CC≤0.5%+10mA	
Monitor	Dual 4-digit LED display	
Protection	Current limit, short circuit, overload and overheat protection	
Operating environment	0~+40°C Relative humidity:<90%	
Locking function	Have	
Anti-impact output control	Have	
Voltage indication accuracy	$LED \pm 0.5\% + 5$ characters	
Current indicating accuracy	LED±1%+5 characters	
Volume	24 length x 13 width x 15 height CM	
Weight	2KG	

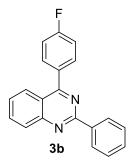
Part II HRMS data for ¹⁵N-labelled quinazoline



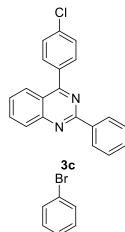
Part III The Analytical Data of Products



2,4-diphenylquinazoline(3a¹): ¹H NMR (500 MHz, CDCl₃) δ 8.55 (d, J = 7.2 Hz ,2H), 7.93 (d, J = 8.4 Hz, 1H), 7.85 (d, J = 8.4 Hz, 1H), 7.70 – 7.64 (m, 2H), 7.62 – 7.55 (m, 1H), 7.41 – 7.28 (m, 6H), 7.27 – 7.21 (m, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 168.3, 160.2, 152.0, 138.3, 137.7, 133.6, 130.7, 130.3, 130.0, 129.2, 128.8, 128.6, 127.1, 121.7.

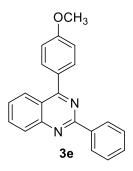


4-(4-fluorophenyl)-2-phenylquinazoline(3b⁷): ¹H NMR (500 MHz, CDCl₃) δ 8.71 – 8.66 (m, 2H), 8.19 (d, *J* = 6.8 Hz, 1H), 8.09 – 8.06 (m, 1H), 7.92 – 7.89 (m, 1H), 7.76 (q, *J* = 8.5 Hz, 3H), 7.58 – 7.51 (m, 4H), 7.25 (s, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 167.3, 164.0 (*J*_{C-F}= 135.4 Hz), 160.2, 151.9, 137.9, 136.5, 132.3 (*J*_{C-F} = 8.3 Hz), 131.8, 130.7, 129.2, 128.6, 127.3, 126.6, 124.7, 121.4 (*J*_{C-F} = 21.7 Hz). ¹⁹F NMR (471 MHz, CDCl₃) δ 110.6.



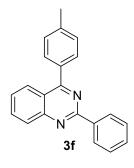
4-(4-chlorophenyl)-2-phenylquinazoline(3c⁶): ¹H NMR (500 MHz, CDCl₃) δ 8.7-8.6 (m, 2H), 8.14 (d, *J* = 8.5 Hz, 1H), 8.03 (d, *J* = 8.3 Hz, 1H), 7.88 – 7.78 (m, 2H), 7.60 – 7.47 (m, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 167.1, 160.2, 152.0, 138.0, 136.3, 136.1, 133.7, 131.6, 130.7, 129.3, 128.9, 128.7, 128.6, 127.3, 126.6, 121.5

4-(4-bromophenyl)-2-phenylquinazoline(3d¹): ¹H NMR (500 MHz, CDCl₃) δ 8.68 – 8.66 (m, 2H), 8.15 (d, *J* = 8.4 Hz, 1H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.89 – 7.87 (m, 1H), 7.75 (q, *J* = 8.5 Hz, 4H), 7.56 – 7.49 (m, 4H). ¹³C NMR (126 MHz, CDCl₃) δ 167.1, 160.3, 152.1, 138.0, 136.6, 133.8, 131.83, 131.77, 130.7, 129.3, 128.7, 128.6, 127.3, 126.6, 124.7, 121.5.

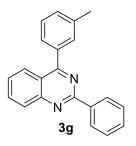


3d

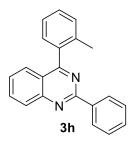
4-(4-methoxyphenyl)-2-phenylquinazoline($3e^7$): ¹H NMR (500 MHz, CDCl₃) δ 8.71 (d, *J* = 6.8 Hz, 2H), 8.18 (d, *J* = 7.9 Hz, 2H), 7.92 – 7.87 (m, 3H), 7.58 – 7.50 (m, 4H), 7.13 (d, *J* = 8.7 Hz, 2H), 3.94 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 168.0, 161.4, 160.0, 133.6, 132.0, 130.6, 130.1, 128.93, 128.91, 128.90, 128.8, 128.6, 127.1, 127.0, 121.6, 114.1, 55.5.



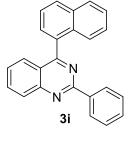
2-Phenyl-4-(p-tolyl)quinazoline(3f⁵): ¹H NMR (500 MHz, CDCl₃) δ 8.73 – 8.66 (m, 1H), 8.19 – 8.13 (m, 1H), 7.90 – 7.84 (m, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.57 – 7.46 (m, 2H), 7.40 (d, *J* = 7.9 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 168.4, 160.2, 152.0, 140.2, 138.3, 134.9, 133.5, 130.5, 130.2, 129.3, 129.1, 128.7, 128.5, 127.1, 126.9, 121.8, 21.5.

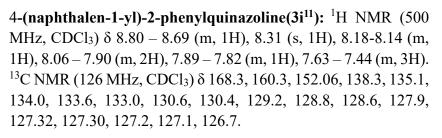


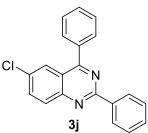
2-phenyl-4-(m-tolyl)quinazoline(3g⁵): ¹H NMR (500 MHz, CDCl₃) δ 8.73 – 8.68 (m, 2H), 8.19 (d, *J* = 8.3 Hz, 1H), 8.13 (d, *J* = 7.8 Hz, 1H), 7.91 – 7.87 (m, 1H), 7.69 (d, *J* = 9.5 Hz, 1H), 7.66 (d, *J* = 7.6 Hz, 1H), 7.58 – 7.47 (m, 5H), 7.40 (d, *J* = 7.6 Hz, 1H), 2.51 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 168.8, 160.2, 151.7, 138.4, 138.1, 137.6, 133.7, 130.8, 130.7, 130.6, 129.0, 128.8, 128.6, 128.4, 127.4, 127.2, 127.1, 121.8, 21.6.



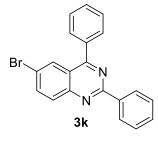
2-(2-methylphenyl)-4-phenylquinazoline(3h⁵): ¹H NMR (500 MHz, CDCl₃) δ 8.67 – 8.65 (m, 2H), 8.17 (d, J = 8.4 Hz, 1H), 7.90 – 7.88 (m, 1H), 7.68 (d, J = 8.3 Hz, 1H), 7.54 – 7.48 (m, 4H), 7.48 – 7.43 (m, 1H), 7.40 (t, J = 9.1 Hz, 2H), 7.37 (t, J = 7.2 Hz, 1H), 2.24 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 169.9, 160.3, 151.4, 138.2, 136.9, 136.5, 133.8, 130.8, 130.6, 129.7, 129.3, 129.0, 128.8 128.6, 127.12, 127.08, 125.7, 122.7, 20.1.



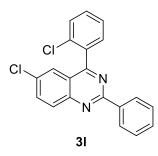




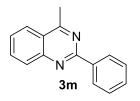
6-chlorine-2,4-diphenylquinazoline(3j⁴): ¹H NMR (500 MHz, CDCl₃) δ 8.64 – 8.58 (m, 2H), 8.05 – 8.02 (m, 2H), 7.83 – 7.79 (m, 2H), 7.76 – 7.74 (m, 1H), 7.59 – 7.52 (m, 3H), 7.45 (q, *J* = 5.0 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 166.6, 159.4, 149.4, 136.7, 136.5, 133.5, 131.6, 129.8, 129.2, 129.0, 127.73, 127.67, 127.6, 124.8, 121.2.



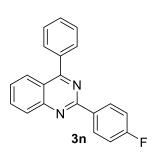
6-bromo-2,4-diphenylquinazoline(3k⁸): ¹H NMR (500 MHz, CDCl₃) δ 8.67 (s, 2H), 8.25 (s, 1H), 8.11 – 7.79 (m, 4H), 7.56 (d, *J* = 49.0 Hz, 6H). ¹³C NMR (126 MHz, CDCl₃) δ 167.5, 160.5, 150.7, 137.7, 137.1, 130.9, 130.9, 130.3, 130.1, 129.1, 128.8, 128.8, 128.6, 122.7, 120.7.



6-chloro-4-(2-chlorophenyl)-2-phenylquinazoline(31²): ¹H NMR (500 MHz, CDCl₃) δ 8.67 – 8.62 (m, 2H), 8.12 (d, *J* = 9.0 Hz, 1H), 7.81 (q, *J* = 9.0 Hz, 1H), 7.63 – 7.58 (m, 2H), 7.55 – 7.46 (m, 6H). ¹³C NMR (126 MHz, CDCl₃) δ 166.5, 160.7, 149.8, 137.6, 135.9, 135.0, 132.9, 132.9, 131.1, 130.92, 130.90, 130.75, 130.2, 128.8, 128.7, 127.1, 125.6, 122.9.



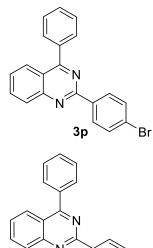
4-methyl-2-phenylquinazoline(3m³): ¹H NMR (500 MHz, CDCl₃) δ 8.50 – 8.46 (m, 2H), 7.85 (d, *J* = 8.4 Hz, 1H), 7.75 (d, *J* = 8.3 Hz, 1H), 7.60-7.57 (m, 1H), 7.41 – 7.23 (m, 4H), 2.74 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 168.2, 160.1, 150.4, 138.4, 133.5, 130.4, 129.2, 128.64, 128.59, 126.8, 124.9, 123.0, 22.00.



2-(4-Fluorophenyl)-4-phenylquinazoline(3n⁶): ¹H NMR (500 MHz, CDCl₃) δ 8.70 (dd, J = 8.6, 5.7 Hz, 2H), 8.14 – 8.10 (m, 2H), 7.93 – 7.82 (m, 3H), 7.60 – 7.58 (m, 3H), 7.55 – 7.52 (m, 1H), 7.25 (s, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 168.5, 164.7 (J_{C-F} = 250.6 Hz), 159.3, 151.8, 137.6, 134.3, 133.7, 130.8 (J_{C-F} = 8.6 Hz), 130.2, 130.1, 129.0, 128.6, 127.1, 121.6, 115.5 (J_{C-F} = 21.6 Hz). ¹⁹F NMR (471 MHz, CDCl₃) δ 110.6.



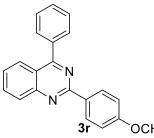
2-(4-Chlorine)-4-phenylquinazoline(30⁹): ¹H NMR (500 MHz, CDCl₃) δ 8.66 (d, J = 8.5 Hz, 2H), 8.18 (d, J = 8.3 Hz, 1H), 8.14 (d, J = 8.4 Hz, 1H), 7.93 – 7.87 (m, 3H), 7.59 – 7.53 (m, 4H), 7.50 (d, J = 8.5 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 168.5, 159.2, 151.8, 137.5, 136.8, 136.7, 133.7, 130.2, 130.1, 129.1, 128.7, 128.6, 127.3, 127.1, 121.7.



3q

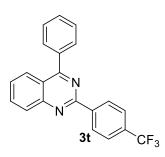
2-(4-bromophenyl)-4-phenylquinazoline(3p⁴): ¹H NMR (500 MHz, CDCl₃) δ 8.60 – 8.57 (m, 2H), 8.20 – 8.11 (m, 2H), 7.92 - 7.85 (m, 3H), 7.67 - 7.54 (m, 6H). ¹³C NMR (126 MHz, CDCl₃) & 168.6, 159.2, 151.7, 137.5, 137.0, 133.8, 131.7, 130.3, 130.2, 130.1, 129.0, 128.6, 127.3, 127.1, 125.4, 121.8.

2-(4-Methylphenyl)-4-phenylquinazoline(3q²): $^{1}\mathrm{H}$ NMR $(500 \text{ MHz}, \text{CDCl}_3) \delta 8.59 \text{ (d, } J = 8.1 \text{ Hz}, 2\text{H}), 8.13 \text{ (d, } J = 6.0 \text{ Hz})$ Hz, 1H), 8.09 (d, J = 8.3 Hz, 1H), 7.88 – 7.83 (m, 3H), 7.59 – 7.57 (m, 3H), 7.51 - 7.48 (m, 1H), 7.32 (d, J = 8.0 Hz, 2H), 2.43(s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 168.3, 160.3, 152.0, 140.8, 137.8, 135.5, 133.5, 130.2, 129.9, 129.3, 129.1, 128.7, 128.5, 127.0, 126.8, 121.6, 21.6.



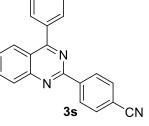
2-(4-methoxyphenyl)-4-phenylquinazoline(3r⁹): $^{1}\mathrm{H}$ NMR (500 MHz, CDCl₃ δ 8.68 - 8.63 (m, 2H), 8.13 - 8.04 (m, 2H), 7.90 – 7.80 (m, 3H), 7.61 – 7.54 (m, 3H), 7.47 (m, 1H), 7.05 - 7.01 (m, 2H), 3.88 (d, J = 8.3 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 168.2, 161.8, 160.1, 152.1 137.8, 133.5, 130.9, 130.4, 130.2, 129.9, 129.0, 128.5, 127.0, 126.6 121.4, 113.9, 55.4.

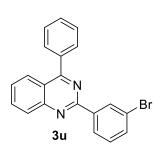
4-(4-phenylquinazolin-2-yl)benzonitrile(3s⁸): ¹H NMR $(500 \text{ MHz}, \text{CDCl}_3) \delta 8.81 \text{ (d}, J = 8.4 \text{ Hz}, 2\text{H}), 8.19-8.15 \text{ (m},$ 2H), 7.96 – 7.90 (m, 1H), 7.90 – 7.85 (m, 2H), 7.79 (d, J = 6.3Hz, 1H), 7.65 – 7.58 (m, 4H). ¹³C NMR (126 MHz, CDCl₃) δ 168.8, 158.2, 151.7, 142.2, 137.3, 134.1, 132.3, 130.3, 130.2, 129.3, 129.1, 128.7, 128.0, 127.2, 127.0, 119.0, 113.7.



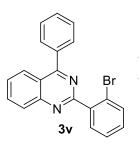
4-Phenyl-2-(4-(trifluoromethyl)phenyl)quinazoline(3t¹⁰): ¹H NMR (500 MHz, CDCl₃) δ 8.79 (d, J = 8.1 Hz, 2H), 8.18 -8.08 (m, 2H), 7.85 (d, J = 3.5 Hz, 3H), 7.74 (d, J = 8.2 Hz, 2H), 7.59 – 7.54 (m, 4H). ¹³C NMR (126 MHz, CDCl₃) δ 168.6, 158.6, 151.9, 141.5, 137.4, 133.8, 132.0 ($J_{C-F} = 32.9$ Hz), 130.2, 130.1, 129.3, 128.9, 128.6, 127.6, 127.1, 125.5, $125.4 (J_{C-F} = 10.7 \text{ Hz}), 124.8 (J_{C-F} = 280.0 \text{ Hz}).$ ¹⁹F (471 MHz, CDCl₃) δ 62.5.

OCH₃





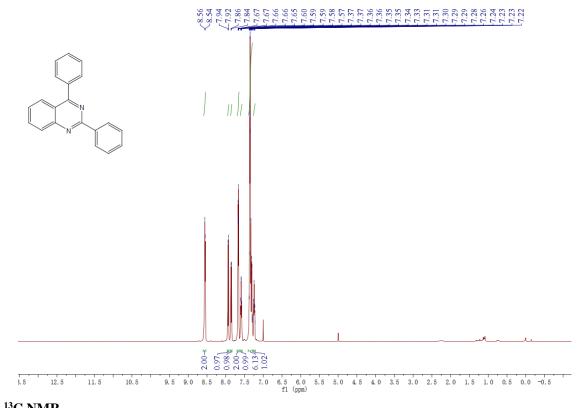
2-(3-bromophenyl)-4-phenylquinazoline(3u²): ¹H NMR (500 MHz, CDCl₃) δ 8.85 (s, 1H), 8.64 (d, *J* = 7.8 Hz, 2H), 8.16 – 8.12 (m, 2H), 7.91 – 7.86 (m, 3H), 7.62 – 7.59 (m, 4H), 7.58 – 7.55 (m, 1H), 7.40 – 7.37 (m, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 168.6, 158.7, 151.8, 140.3, 137.4, 133.8, 133.4, 131.6, 130.2, 130.12, 130.09, 129.2, 128.6, 127.5, 127.3, 127.1, 122.9, 121.9.



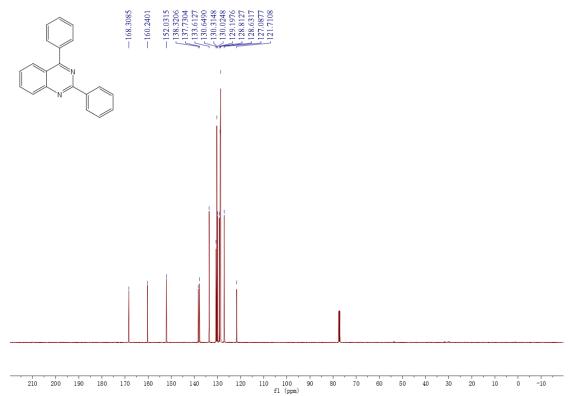
2-(2-bromophenyl)-4-phenylquinazoline(3v^{12}): ¹H NMR (500 MHz, CDCl₃) δ 8.50 (d, J = 8.6 Hz, 2H), 8.10 – 8.03 (m, 2H), 7.83 – 7.79 (m, 3H), 7.57 (d, J = 8.5 Hz, 2H), 7.54 – 7.52 (m, 3H), 7.50 – 7.47 (m, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 167.5, 158.2, 150.7, 136.4, 136.0, 132.7, 130.8, 129.9, 129.2, 129.1, 129.0, 128.0, 127.8, 127.6, 126.3, 126.1, 124.3, 120.7.

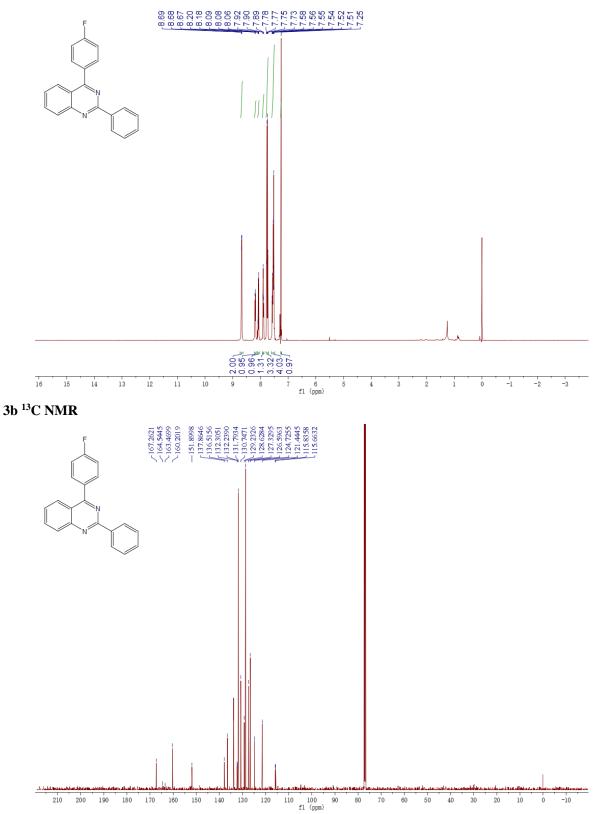
Part IV NMR Spectra

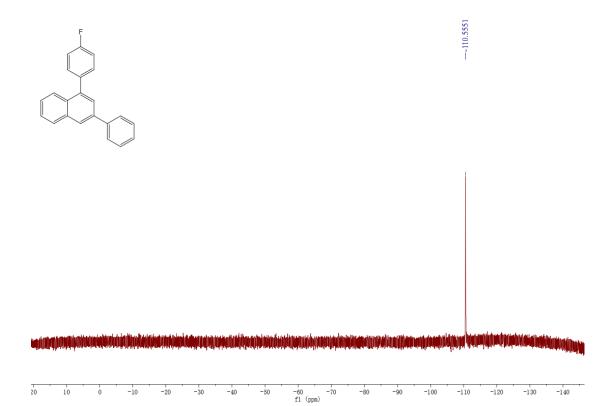
3a ¹H NMR



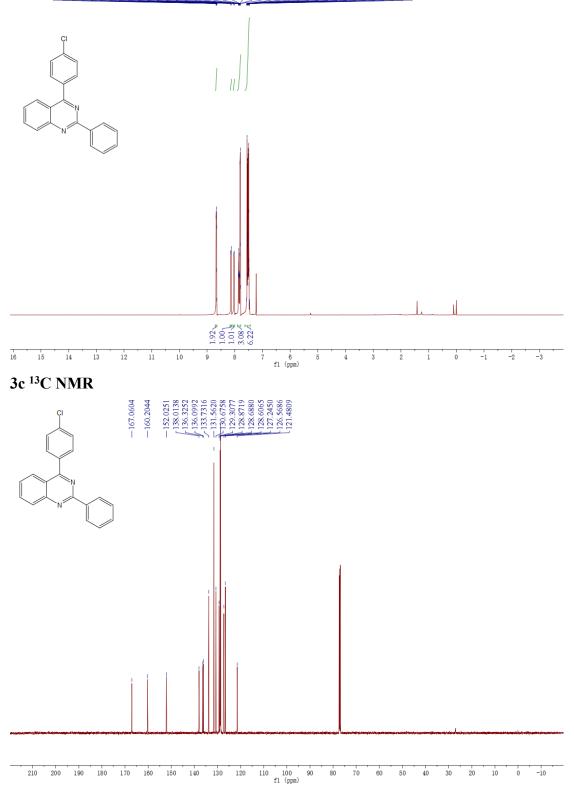
3a ¹³C NMR

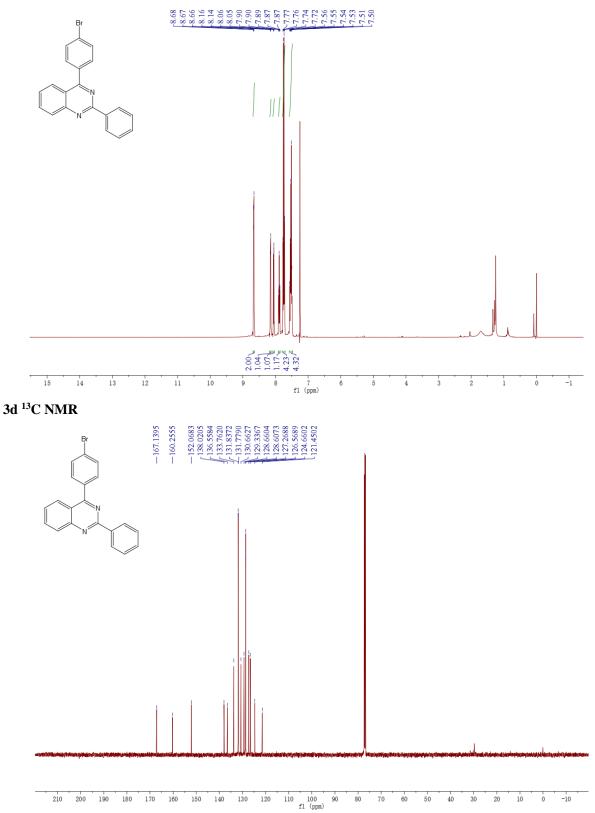




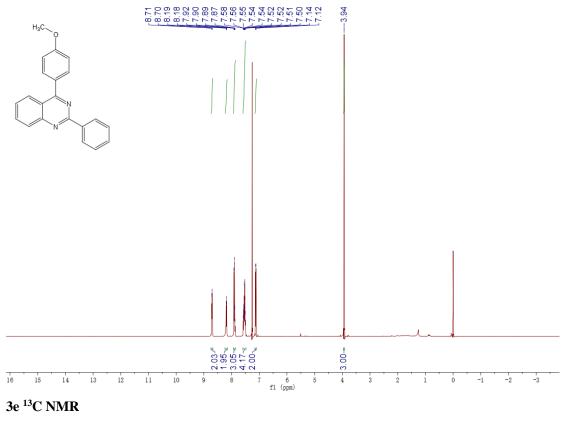


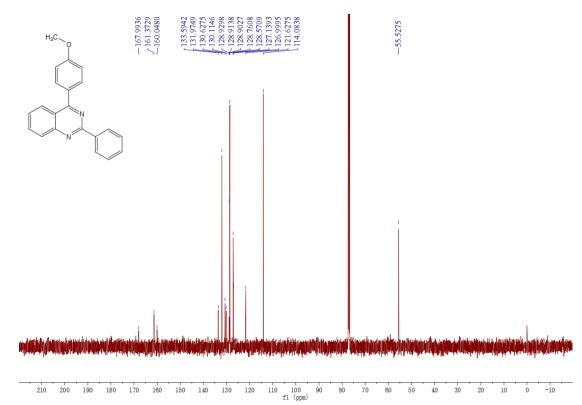
3c¹H NMR

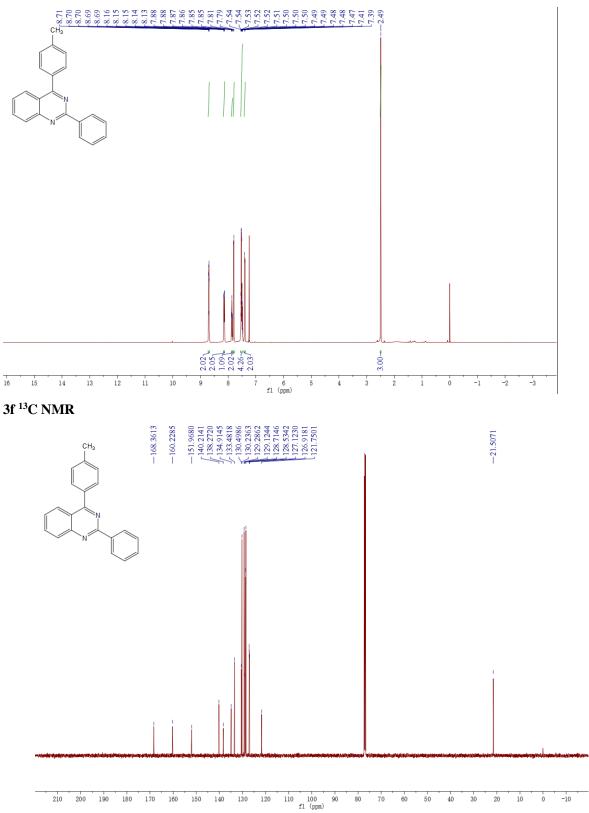




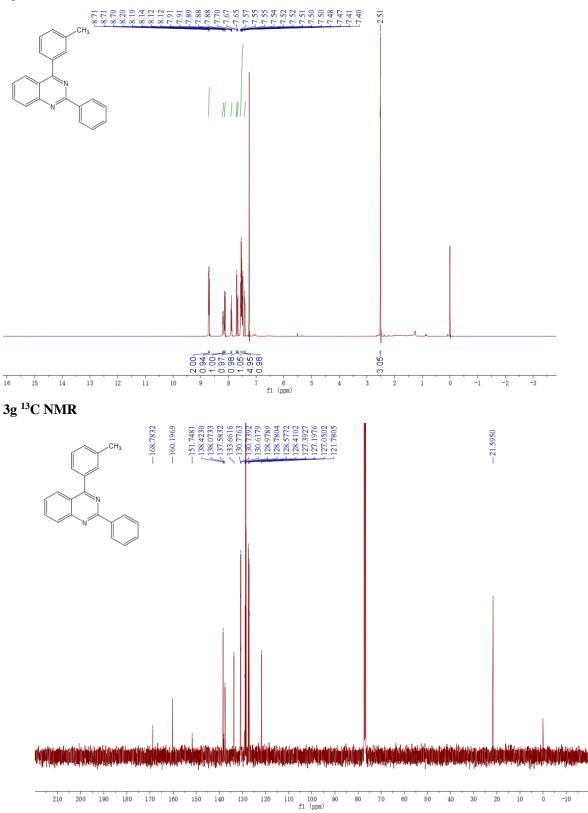


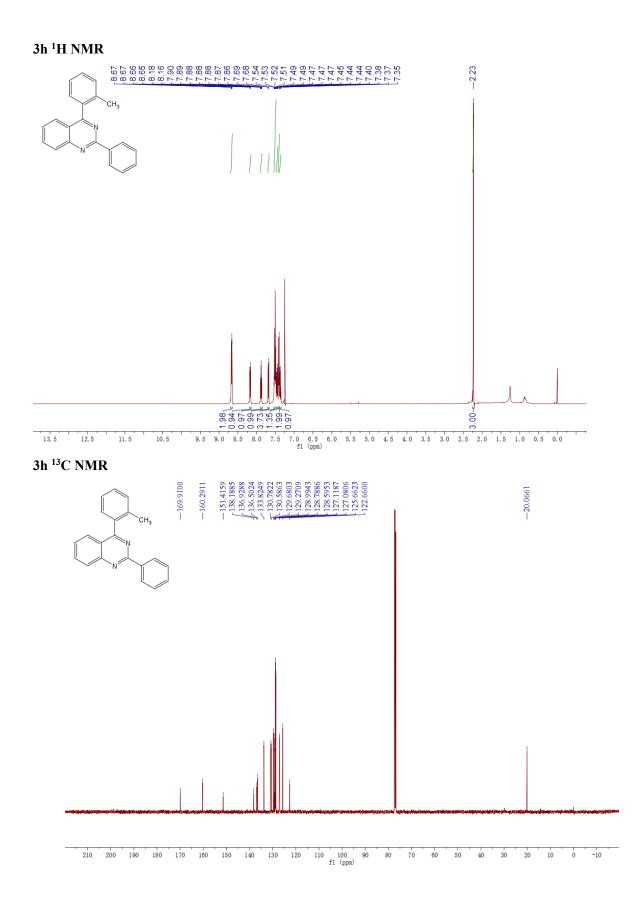


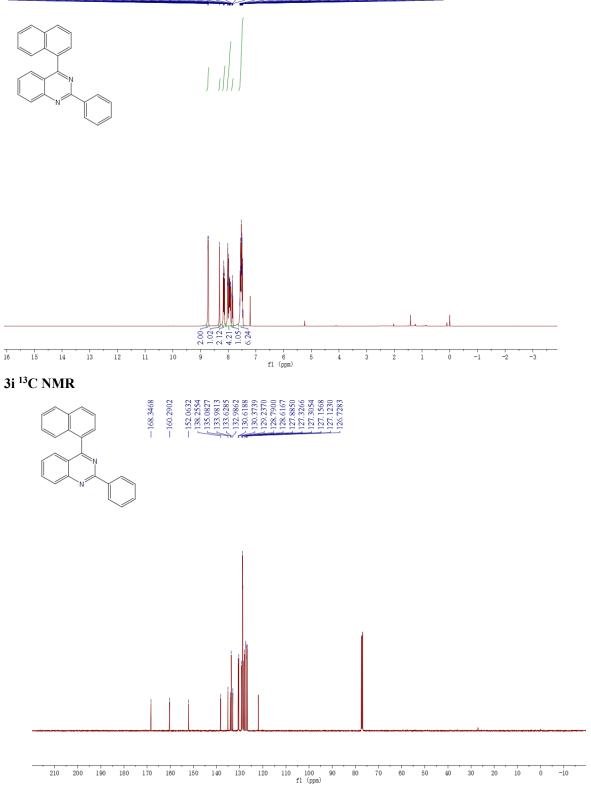




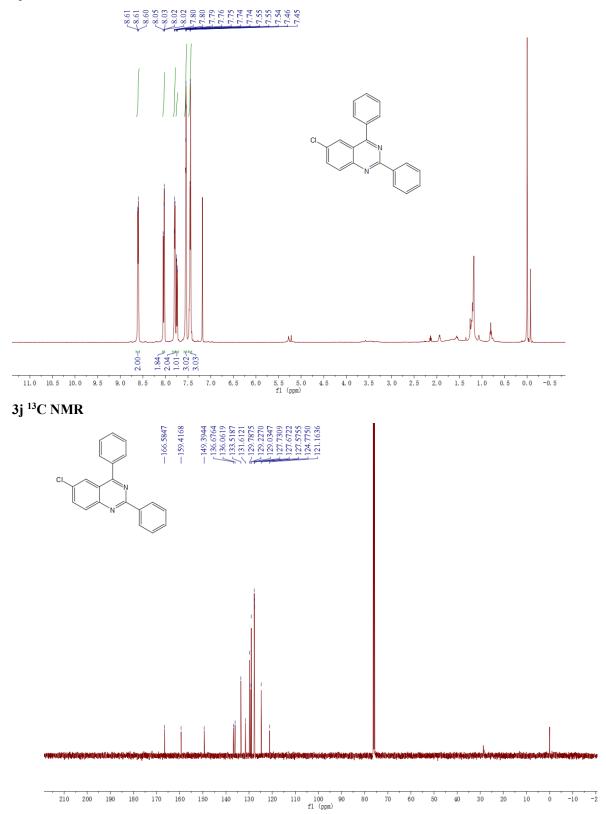




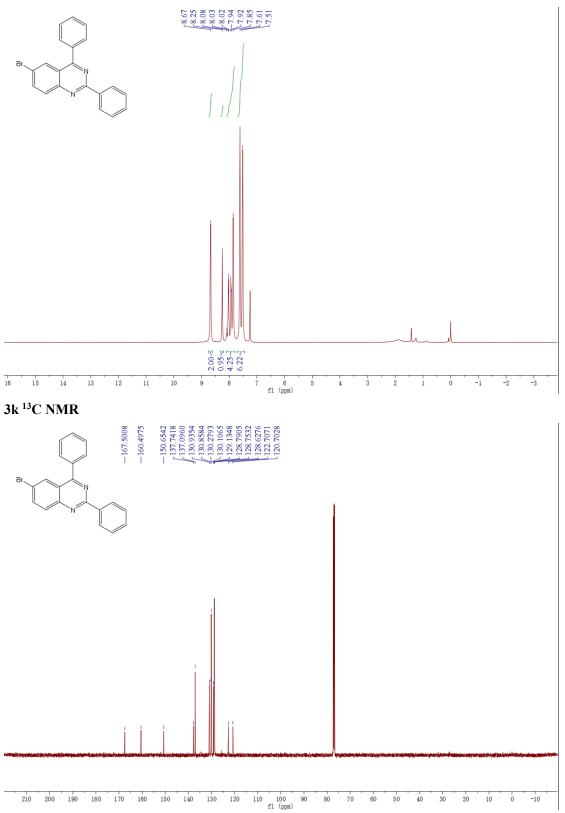




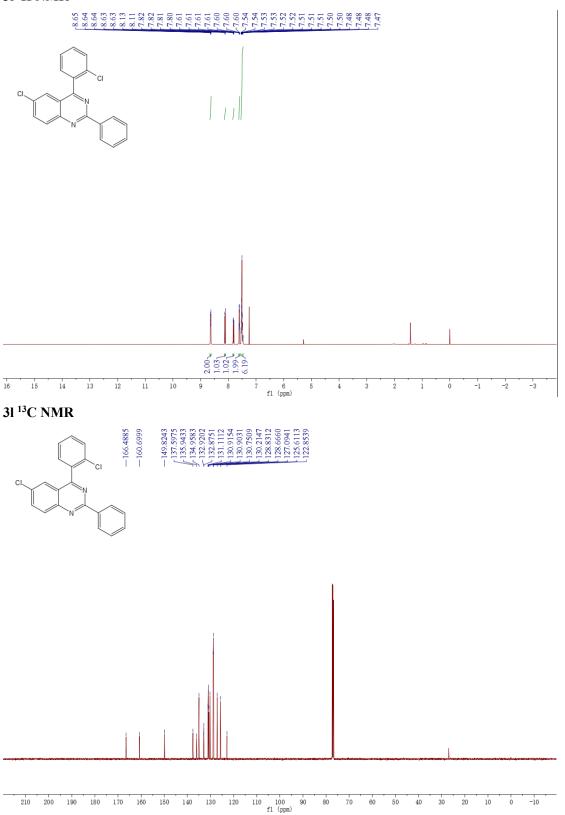
3j¹H NMR



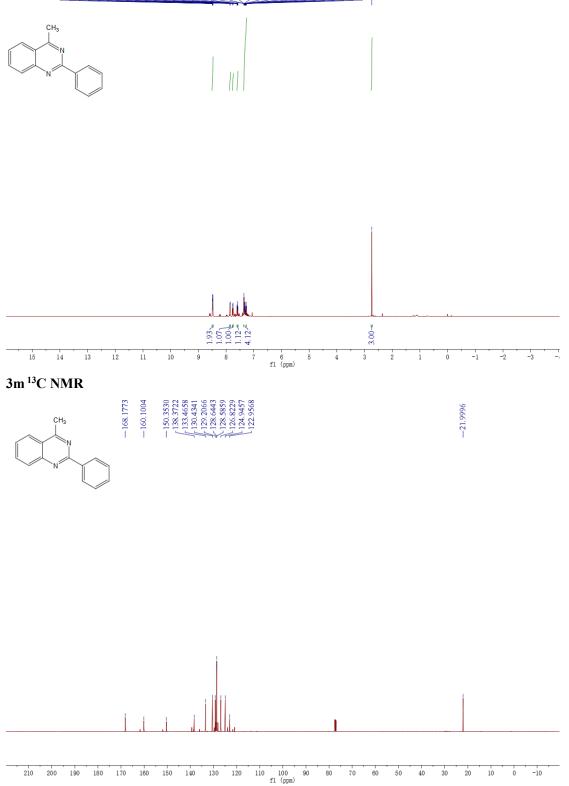


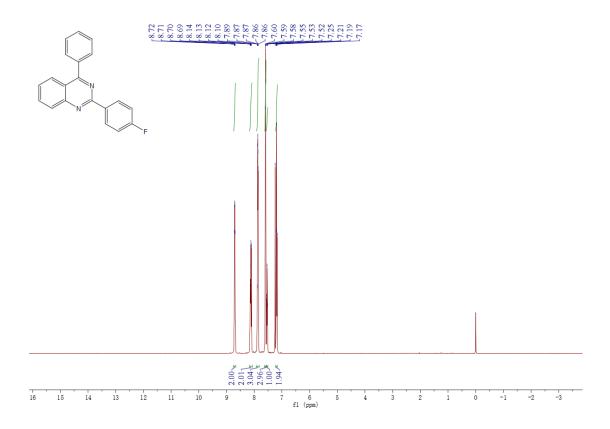




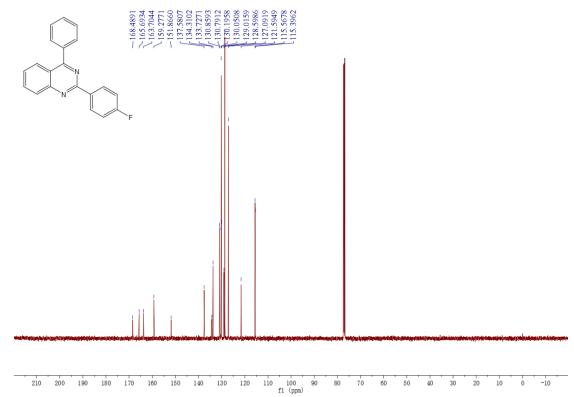




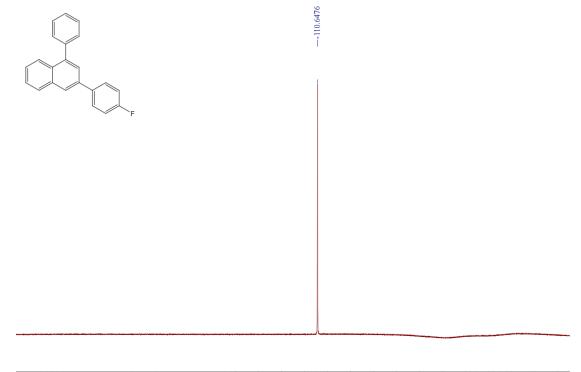




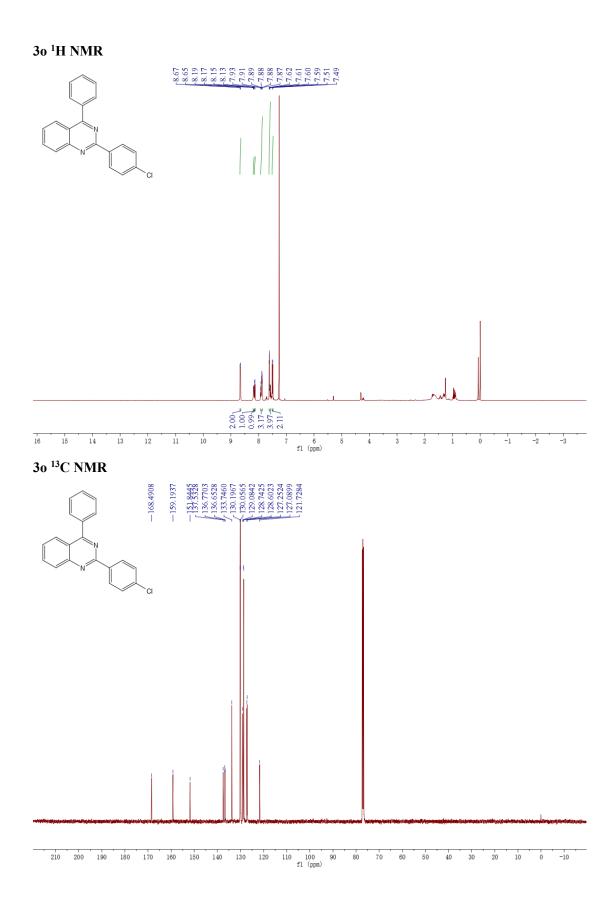
3n ¹³C NMR

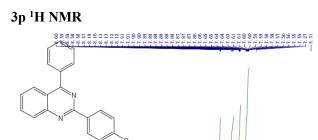


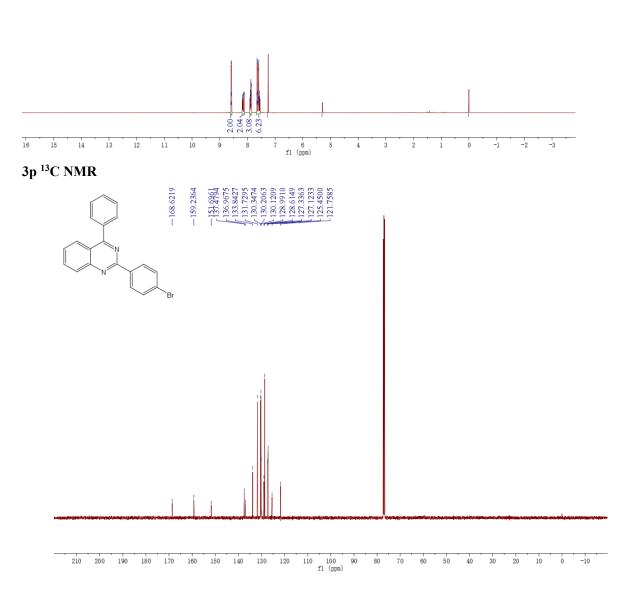
3n¹⁹F NMR



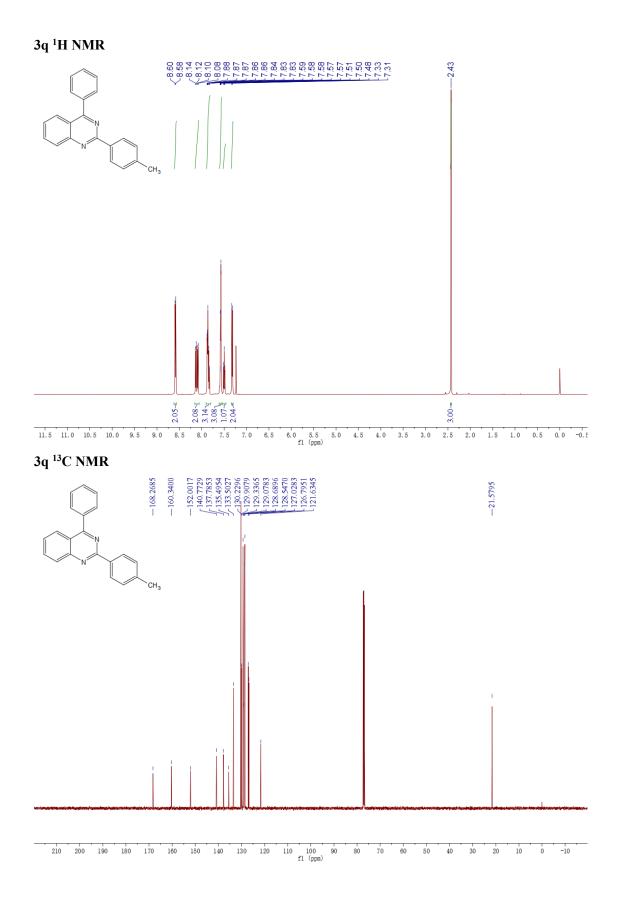
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -21 fl (ppm)

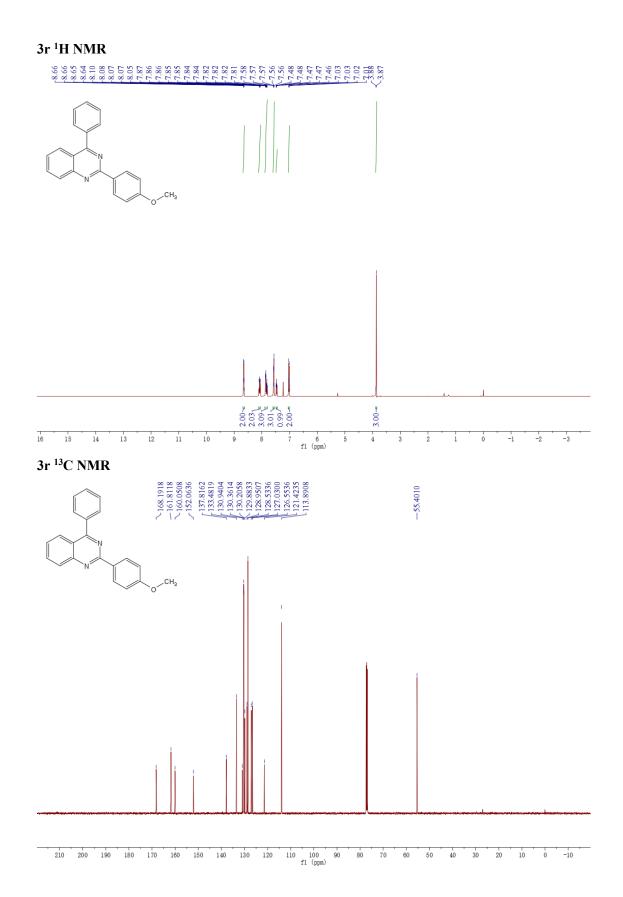






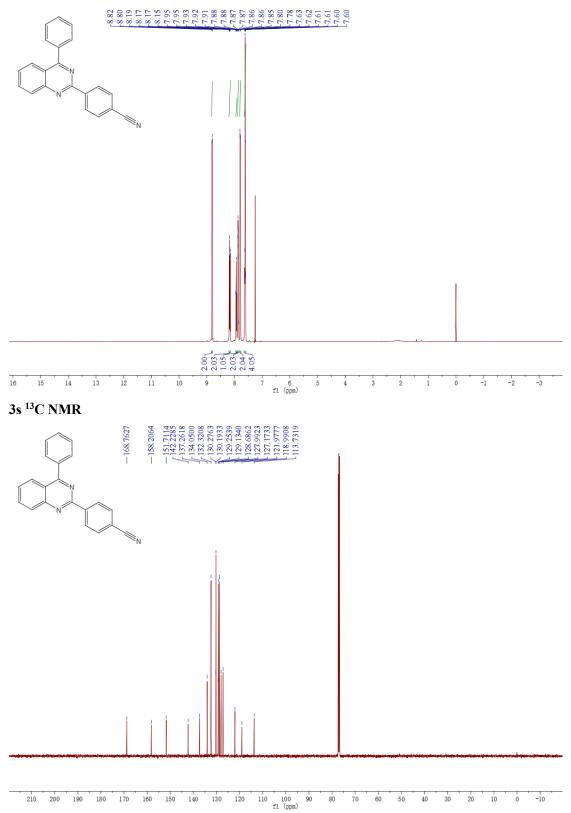
---0.02



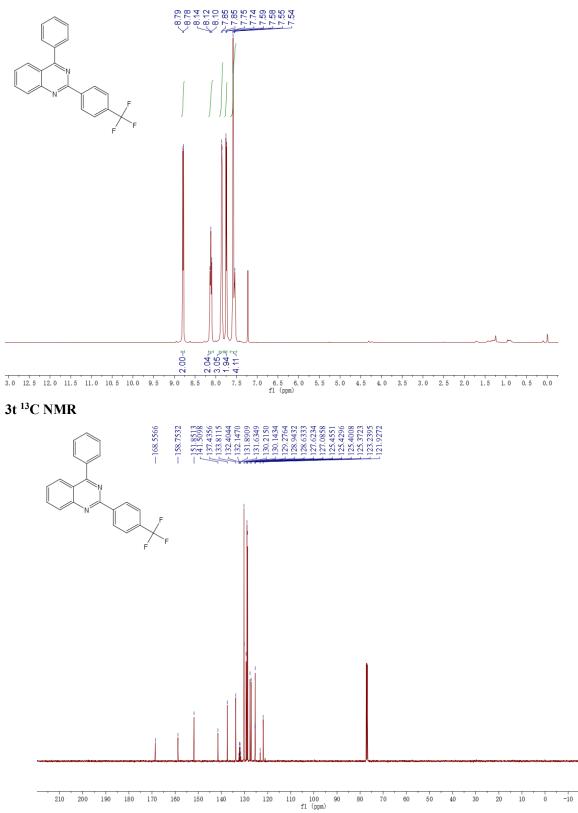


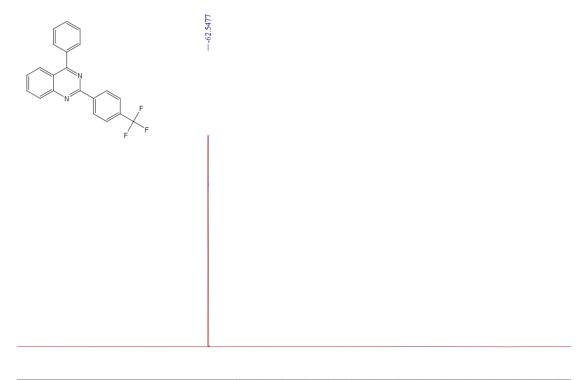
30/37





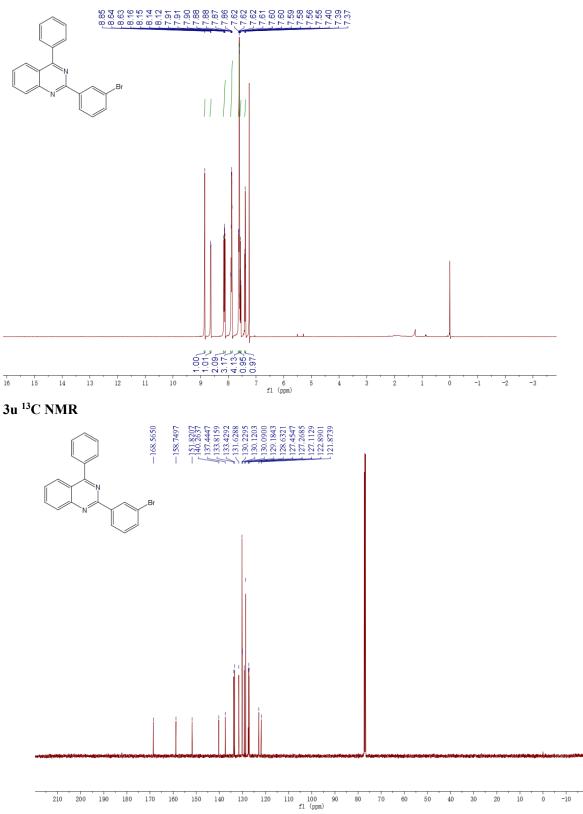




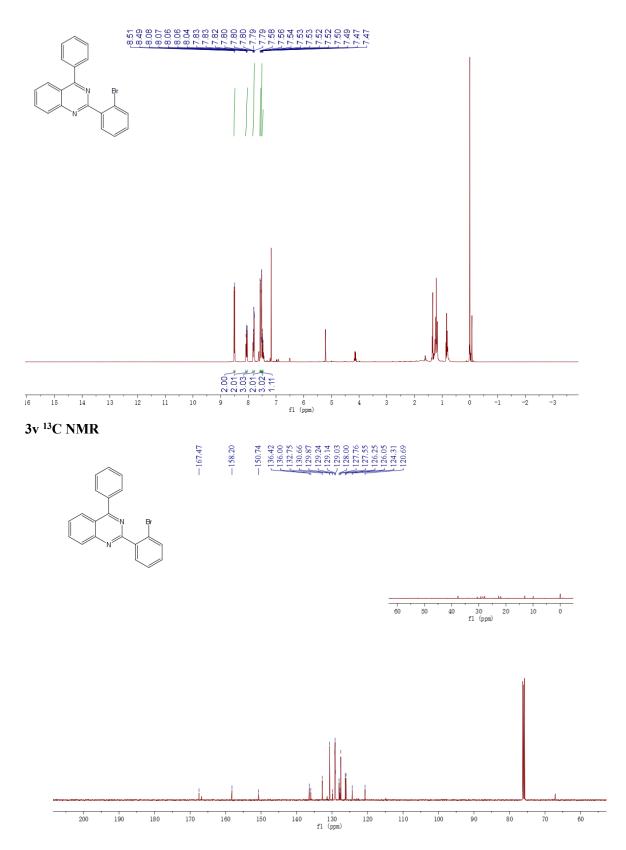


20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -21 fl (ppm)



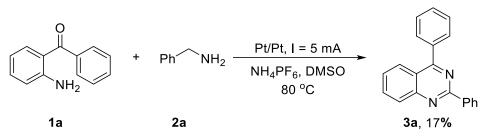


3v¹H NMR



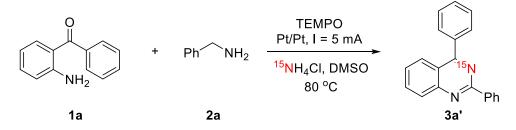
5. Control Experiments

5.1 No TEMPO reaction



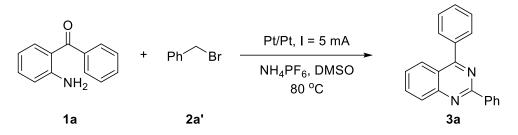
1a (2-aminobenzophenone, 0.3 mmol, 59.2 mg), and NH₄PF₆ (0.6 mmol, 97.8 mg), were added to a 100 mL reaction vial, followed by the addition of 3 mL DMSO using a plastic pipette. Then add **2a** (benzylamine, 0.6 mmol, 65.5 μ L) with a pipette and use the Pt/Pt as electrodes. The reaction mixture was heated at 80°C while stirring and monitoring the voltage. When the reaction was completed, the reaction solution was extracted three times with water and ethyl acetate, and the combined organic phases were dried over anhydrous sodium sulfate and evaporated in vacuo to obtain the crude product. The residue was purified by flash column chromatography (petroleum ether/ethyl acetate) to give **3a**.

5.2 Isotope labeling experiment



1a (2-aminobenzophenone, 0.3 mmol, 59.2 mg), ammonium chloride (15 NH₄Cl, 0.6 mmol, 32.4 mg) and TEMPO (2,2,6,6-tetramethylpiperidinooxy, 0.06 mmol, 4.7 mg) were added to a 10 mL reaction vial, followed by addition of 3 mL DMSO using a plastic pipette. Then add **2a** (benzylamine, 0.6 mmol, 65.5 µL) with a pipette and use the Pt/Pt as electrodes. The reaction mixture was heated at 80°C while stirring and monitoring the voltage. When the reaction was completed, the reaction solution was extracted three times with water and ethyl acetate, and the combined organic phases were dried over anhydrous sodium sulfate and evaporated in vacuo to obtain the crude product. The residue was purified by flash column chromatography (petroleum ether/ethyl acetate) to give the ¹⁵N-labelled quinazoline **3a**' (60%).

5.3 Benzylamine replaced by benzylbromide



1a (2-aminobenzophenone, 0.3 mmol, 59.2 mg), NH₄PF₆ (0.6 mmol, 97.8 mg) and TEMPO (2,2,6,6-tetramethylpiperidinooxy, 0.06 mmol, 4.7 mg) were added to a 10 mL reaction vial, followed by addition of 3 mL DMSO using a plastic pipette. Then add **2a**' (benzylbromide, 0.6 mmol, 71.3 μ L) with a pipette and use the Pt/Pt as electrodes. The reaction mixture was heated at 80°C while stirring and monitoring the voltage. When the reaction was completed, the reaction solution was extracted three times with water and ethyl acetate, and the combined organic phases were dried over anhydrous sodium sulfate and evaporated in vacuo to obtain the crude product. The residue was purified by flash column chromatography (petroleum ether/ethyl acetate) to give **3a** (43%)

6. References

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