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Copper-Catalyzed Radical Cascade Cyclization for Synthesis of CF_3-Containing Tetracyclic Benzimidazo[2,1-*a*]iso-quinolin-6(5*H*)-ones

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Supporting Information

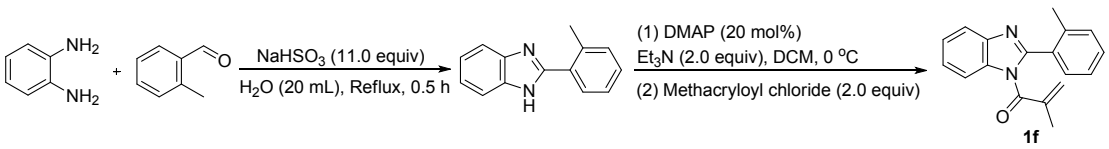
Contents

1. General Conditions

All reagents were purchased from commercial sources and used without further purification. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on a Bruker Ascend™ 400 or a Bruker Ascend™ 600 spectrometer in deuterated solvents containing TMS as an internal reference standard. All high-resolution mass spectra (HRMS) were measured on a mass spectrometer by using electrospray ionization orthogonal acceleration time-of-flight (ESI-OA-TOF), and the purity of all samples used for HRMS (>95%) was confirmed by ^{1}H NMR and ^{13}C NMR spectroscopic analysis. Melting points were measured on a melting point apparatus equipped with a thermometer and were uncorrected. All the reactions were monitored by thin-layer chromatography (TLC) using GF254 silica gel-coated TLC plates. Purification by flash column chromatography was performed over SiO_2 (silica gel 200−300 mesh).

2. Typical Experimental Procedure

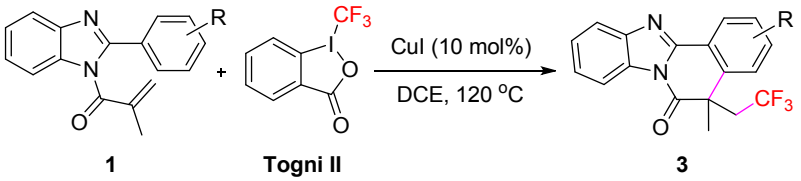
2.1 The general procedure for the synthesis of 1[1] (1f as an example):



Step 1: In a round-bottomed flask (50 mL) equipped with a magnetic stirrer, a mixture of *o*-methylbenzaldehyde (5.0 mmol, 578 μL) and $NaHSO_3$ (11.0 eq, 5.73 g) in H_2O (20.0 mL) was prepared. When the mixture reached refluxing temperature, *o*-phenylenediamine (5.0 mmol, 541 mg) were added. The resulting mixture was stirred for appropriate time. After completion of the reaction, the reaction mixture was vacuum filtered after cooling to room temperature by a glass funnel. The residues were washed by water (20 mL × 2), dried in air dry oven to give the corresponding product.

Step 2: To the solution of 2-(*o*-tolyl)-1*H*-benzo[*d*]imidazole (3 mmol, 625 mg) and DMAP (0.6 mmol, 73 mg) in DCM (0.5 M) was added Et_3N (6 mmol, 834μL) and methacryloyl chloride (6 mmol, 581 μL) at 0 °C. The solution was warmed up to room temperature and stirred for 12 h. The reaction was complete according to TLC analysis, and water (20 mL) was added to the mixture, which was extracted with CH_2Cl_2 (15 mL × 3). Then the organic solvent was concentrated in vacuo. The residue was purified by flash column chromatography with Ethyl acetate and Petroleum ether as eluent to give **1f**.

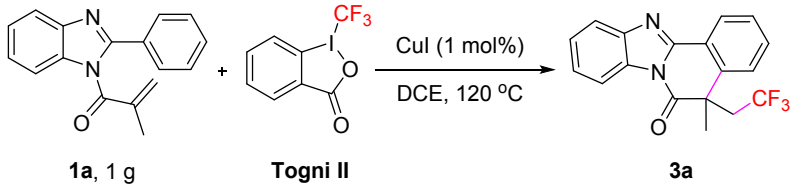
2.2 The general procedure for the synthesis of 3



1 **Togni II** **3**

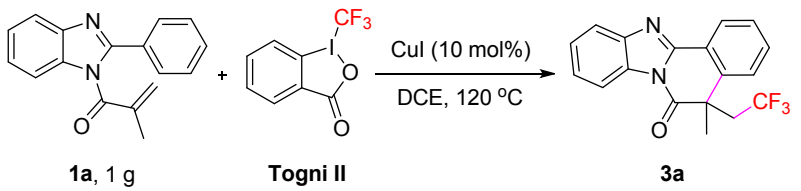
With no precautions to exclude air or moisture, the screw-cap test tube (15 mL) with a stir bar was charged with olefinic amide **1** (0.3 mmol), **Togni-II** (2.0 equiv, 189.6 mg), CuI (10 mol%, 5.7 mg) and DCE (2 mL). Then the mixture was stirred at 120 °C for appropriate time. After the completion of the reaction, the mixture was quenched by $NaHCO_3$ (sat. aq. 15 mL) and extracted with CH_2Cl_2 (5 mL × 3). Then the organic solvent was concentrated in vacuo and residue was purified by flash column chromatography with ethyl acetate and Petroleum ether as eluent to give **3**.

2.3 Gram scale-up reactions.



1a, 1 g **Togni II** **3a**

With no precautions to exclude air or moisture, the screw-cap test tube (150 mL) with a stir bar was charged with 2-methyl-1-(2-phenyl-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1a** (3.82 mmol, 1002 mg), **Togni-II** (1.5 equiv, 1811 mg), CuI (1 mol%, 7.3 mg) and DCE (40 mL). Then the mixture was stirred at 120 °C for 4 h. After the completion of the reaction, the mixture was quenched by $NaHCO_3$ (sat. aq. 150 mL) and extracted with CH_2Cl_2 (50 mL × 3). Then the organic solvent was concentrated in vacuo. The residue was purified by flash column chromatography with Ethyl acetate and Petroleum ether as eluent to give **3a** (542.6 mg, 43% yield).

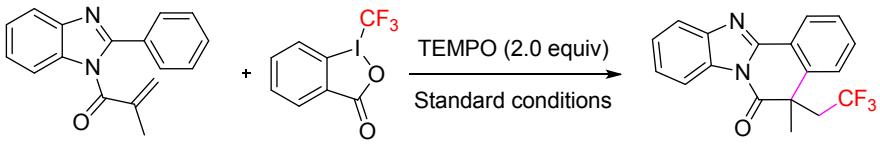


1a, 1 g **Togni II** **3a**

With no precautions to exclude air or moisture, the screw-cap test tube (150 mL) with a stir bar was charged with 2-methyl-1-(2-phenyl-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1a** (3.82 mmol, 1002 mg), **Togni-II** (1.5 equiv, 1811 mg), CuI (10 mol%, 73 mg) and DCE (40 mL). Then the mixture was stirred at 120 °C for 4 h. After the completion of the reaction, the mixture was quenched by $NaHCO_3$ (sat. aq. 150 mL) and extracted with CH_2Cl_2 (50 mL × 3). Then the organic solvent was concentrated in vacuo. The residue was purified by flash column chromatography with Ethyl acetate and Petroleum ether as eluent to give **3a** (908.5 mg, 72% yield).

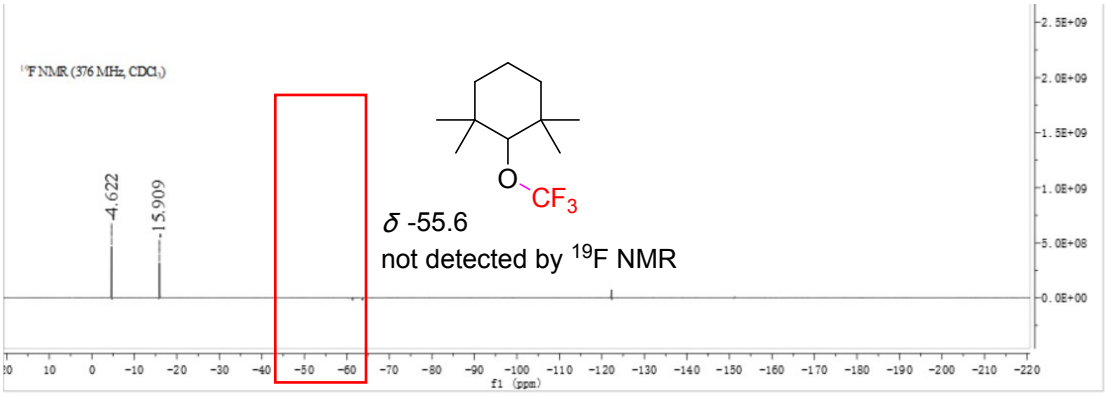
3. Control experiment

3.1 Control experiment in the presence of TEMPO

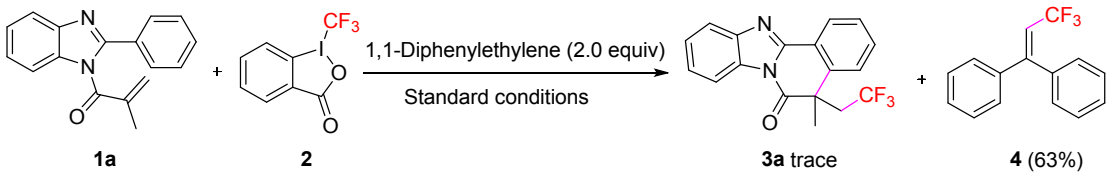


1a **2** **3a** (0%)

With no precautions to exclude air or moisture, the screw-cap test tube (15 mL) with a stir bar was charged with 2-methyl-1-(2-phenyl-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1a** (0.3 mmol, 78.7 mg), **Togni-II** (2.0 equiv, 189.6 mg), CuI (10 mol%, 5.7 mg), radical scavenger TEMPO (2.0 equiv, 93.8 mg) and DCE (2 mL). Then the mixture was stirred at 120 °C for 1 h and then monitored by TLC. No desired product **3a** was detected, suggesting that this transformation might occur *via* a radical process. The **TEMPO-CF_3** singal wasn't detected by ^{19}F NMR analysis.[2]



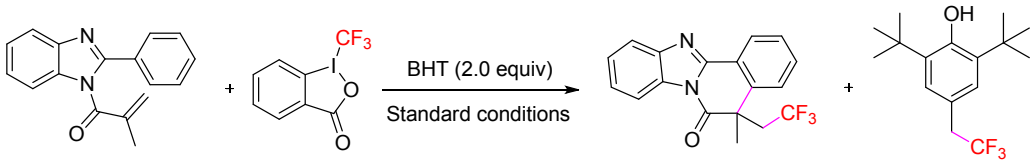
3.2 Control experiment in the presence of ethene-1,1-diyldibenzene



1a **2** **3a** trace **4** (63%)

With no precautions to exclude air or moisture, the screw-cap test tube (15 mL) with a stir bar was charged with 2-methyl-1-(2-phenyl-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1a** (0.3 mmol, 78.7 mg), **Togni-II** (2.0 equiv, 189.6 mg), CuI (10 mol%, 5.7 mg), the radical scavenger 1,1-Diphenylethylene (2.0 equiv, 108.2 mg) and DCE (2 mL). Then the mixture was stirred at 120 °C for 1 h and monitored by TLC. Only trace of product **3a** was obtained and 63% of (3,3,3-trifluoroprop-1-ene-1,1-diyl)dibenzene **4** was obtained, suggesting that this transformation might occur *via* a radical process.

3.3 Control experiment in the presence of BHT

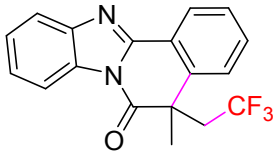


1a **2** **3a** (41%) **5** (28%)

With no precautions to exclude air or moisture, the screw-cap test tube (15 mL) with a stir bar was charged with 2-methyl-1-(2-phenyl-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1a** (0.3 mmol, 78.7 mg), **Togni-II** (2.0 equiv, 189.6 mg), CuI (10 mol%, 5.7 mg), the radical scavenger BHT (2.0 equiv, 132.2 mg) and DCE (2 mL). Then the mixture was stirred at 120 °C for 1 h and monitored by TLC. Only 41% of product **3a** was obtained and 28% of 2,6-di-*tert*-butyl-4-(2,2,2-trifluoroethyl)phenol **5** was obtained, suggesting that this transformation might occur *via* a radical process.

4. Product Characterization

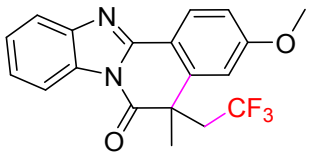
5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3a)



2-methyl-1-(2-phenyl-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1a** (78.7mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (115 – 117 ^{o}C) in 74% yield.

Rf (petroleum ether: EtOAc = 20:1): 0.15; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.52 (d, J = 7.6 Hz, 1H), 8.36 (d, J = 7.6 Hz, 1H), 7.83 (d, J = 7.6 Hz, 1H), 7.59 (t, J = 7.6 Hz, 1H), 7.53 (t, J = 7.6 Hz, 1H), 7.50 – 7.40 (m, 3H), 3.55 – 3.40 (m, 1H), 3.01 – 2.89 (m, 1H), 1.76 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.13, 149.37, 144.16, 138.54, 131.77, 131.52, 128.57, 126.59, 126.45, 126.29, 125.97, 125.04 (q, J = 277.0 Hz), 122.52, 120.08, 115.83, 45.40 (q, J = 2.0 Hz), 44.31 (q, J = 27.7 Hz), 31.08; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -61.35; **HRMS** (ESI) calcd for $C_{18}H_{14}F_3N_2O$ $[M+H]^+$: 331.1053, found: 331.1057.

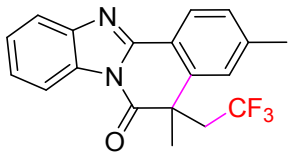
3-methoxy-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3b)



1-(2-(4-methoxyphenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1b** (87.7mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (133 – 135 ^{o}C) in 72% yield.

Rf (petroleum ether: EtOAc = 20:1): 0.2; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.45 (d, J = 8.7 Hz, 1H), 8.32 (d, J = 7.8 Hz, 1H), 7.78 (d, J = 7.8 Hz, 1H), 7.45 – 7.37 (m, 2H), 7.07 (dd, J = 8.7, 2.3 Hz, 1H), 6.93 (d, J = 2.3 Hz, 1H), 3.91 (s, 3H), 3.51 – 3.41 (m, 1H), 2.94 – 2.85 (m, 1H), 1.74 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.12, 162.52, 149.56, 144.25, 140.62, 131.42, 128.42, 126.17, 125.45, 125.03 (q, J = 278.8 Hz), 119.64, 115.67, 115.35, 114.10, 112.53, 55.75, 45.50 (q, J = 2.0 Hz), 44.09 (q, J = 28.0 Hz), 31.20; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.28; **HRMS** (ESI) calcd for $C_{19}H_{16}F_3N_2O_2$ $[M+H]^+$: 361.1158, found: 361.1158.

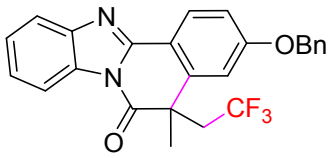
3,5-dimethyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3c)



2-methyl-1-(2-(*p*-tolyl)-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1c** (82.9mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (169 – 171 ^{o}C) in 74% yield.

Rf (petroleum ether: EtOAc = 20:1): 0.3; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.39 (d, J = 8.0 Hz, 1H), 8.35 – 8.33 (m, 1H), 7.81 (d, J = 7.2 Hz, 1H), 7.46 – 7.40 (m, 2H), 7.34 (d, J = 8.0 Hz, 1H), 7.25 (s, 1H), 3.50 – 3.41 (m, 1H), 2.97 – 2.89 (m, 1H), 2.47 (s, 3H), 1.74 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.26, 149.64, 144.28, 142.39, 138.63, 131.52, 129.66, 126.96, 126.40, 126.18, 125.69, 125.09 (q, J = 278.7 Hz), 119.92, 115.76, 45.32 (q, J = 2.1 Hz), 44.06 (q, J = 27.7 Hz), 31.08, 22.01; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.33; **HRMS** (ESI) calcd for $C_{19}H_{16}F_3N_2O$ $[M+H]^+$: 345.1209, found: 345.1201.

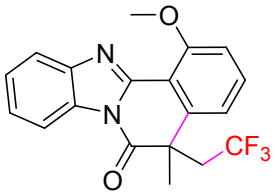
3-(benzyloxy)-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinol-in-6(5*H*)-one (3d)



1-(2-(4-(benzyloxy)phenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1d** (110.5mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (95 – 97 ^{o}C) in 67% yield.

Rf (petroleum ether: EtOAc = 30:1): 0.15; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.46 (d, J = 8.7 Hz, 1H), 8.33 (d, J = 7.8 Hz, 1H), 7.79 (d, J = 7.8 Hz, 1H), 7.47 – 7.36 (m, 7H), 7.16 (dd, J = 8.7, 2.2 Hz, 1H), 7.01 (s, 1H), 5.21 – 5.14 (m, 2H), 3.49 – 3.40 (m, 1H), 2.89 – 2.79 (m, 1H), 1.72 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.06, 161.62, 149.47, 144.30, 140.60, 136.03, 131.42, 128.90, 128.54, 128.36, 127.71, 126.14, 125.00 (q, J = 278.3 Hz), 125.44, 119.68, 115.65, 115.60, 115.00, 113.44, 70.60, 45.45 (q, J = 1.8 Hz), 44.09 (q, J = 27.7 Hz), 31.10; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -61.31; **HRMS** (ESI) calcd for $C_{25}H_{20}F_3N_2O_2$ $[M+H]^+$: 437.1471, found: 437.1463.

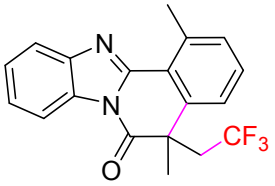
1-methoxy-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3e)



1-(2-(2-methoxyphenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1e** (87.7mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (155 – 157 °C) in 68% yield.

Rf (petroleum ether: EtOAc = 4:1): 0.2; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.39 – 8.33 (m, 1H), 7.93 – 7.87 (m, 1H), 7.51 (t, J = 8.1 Hz, 1H), 7.45 – 7.39 (m, 2H), 7.09 – 7.05 (m, 2H), 4.13 (s, 3H), 3.50 – 3.40 (m, 1H), 2.96 – 2.86 (m, 1H), 1.75 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.03, 159.00, 147.48, 144.47, 141.07, 132.13, 130.38, 125.98, 125.92, 125.03 (q, J = 278.9 Hz), 120.70, 118.88, 115.60, 111.88, 111.12, 56.76, 45.18 (q, J = 1.7 Hz), 44.37 (q, J = 27.6 Hz), 31.39; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.27; **HRMS** (ESI) calcd for $C_{19}H_{16}F_3N_2O_2$ $[M+H]^+$: 361.1158, found: 361.1159.

1,5-dimethyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3f)

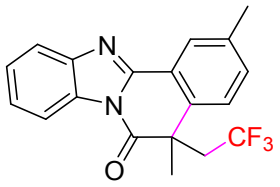


2-methyl-1-(2-(*o*-tolyl)-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1f** (82.9mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (154 – 156 °C) in 70% yield.

Rf (petroleum ether: EtOAc = 100:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.43 – 8.35 (m, 1H), 7.88 – 7.82 (m, 1H), 7.48 – 7.42 (m, 3H), 7.38 – 7.32 (m, 2H), 3.55 – 3.42 (m, 1H), 3.07 (s, 3H), 3.00 – 2.88 (m, 1H), 1.76 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.32, 149.60, 144.32, 140.38,

139.60, 131.84, 130.68, 130.37, 125.99, 125.14 (q, J = 278.6 Hz), 124.51, 121.12, 120.38, 115.88, 45.35 (q, J = 1.9 Hz), 44.34 (q, J = 27.6 Hz), 31.63, 24.90; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.20; **HRMS** (ESI) calcd for $C_{19}H_{16}F_3N_2O$ $[M+H]^+$: 345.1209, found: 345.1193.

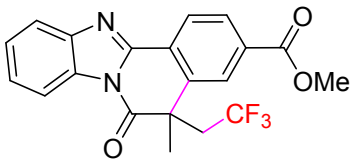
2,5-dimethyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3g)



2-methyl-1-(2-(*m*-tolyl)-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1g** (82.9mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (107 – 109 ^{o}C) in 63% yield.

Rf (petroleum ether: EtOAc = 15:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.37 – 8.32 (m, 2H), 7.84 – 7.80 (m, 1H), 7.46 – 7.42 (m, 2H), 7.41 – 7.33 (m, 2H), 3.51 – 3.40 (m, 1H), 2.96 – 2.86 (m, 1H), 2.47 (s, 3H), 1.73 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.36, 149.60, 144.16, 138.63, 135.70, 132.88, 131.55, 126.53, 126.50, 126.23, 125.86, 125.08 (q, J = 278.6 Hz), 122.22, 120.01, 115.82, 45.16 (q, J = 1.4 Hz), 43.99 (q, J = 27.6 Hz), 31.13, 21.10; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -61.33; **HRMS** (ESI) calcd for $C_{19}H_{16}F_3N_2O$ $[M+H]^+$: 345.1209, found: 345.1210.

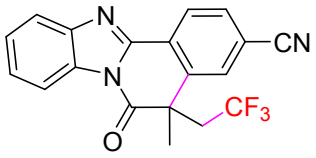
methyl 5-methyl-6-oxo-5-(2,2,2-trifluoroethyl)-5,6-dihydrobenzo[4,5]imidazo[2,1-*a*]isoquinoline-3-carboxylate (3h)



Methyl 4-(1-methacryloyl-1H-benzo[d]imidazol-2-yl)benzoate **1h** (96.1 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (182 – 184 ^{o}C) in 72% yield.

Rf (petroleum ether: EtOAc = 20:1): 0.2; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.58 (d, J = 8.0 Hz, 1H), 8.38 – 8.34 (m, 1H), 8.18 – 8.14 (m, 2H), 7.87 – 7.83 (m, 1H), 7.50 – 7.45 (m, 2H), 3.98 (s, 3H), 3.55 – 3.44 (m, 1H), 3.08 – 2.98 (m, 1H), 1.80 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 170.64, 166.04, 148.28, 144.14, 138.68, 132.85, 131.52, 129.36, 128.11, 126.59, 126.55, 126.27, 124.96 (q, J = 279.1 Hz), 120.42, 115.93, 52.78, 45.44 (q, J = 1.9 Hz), 44.07 (q, J = 28.0 Hz), 30.83; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.39; **HRMS** (ESI) calcd for $C_{20}H_{16}F_3N_2O_3$ $[M+H]^+$: 389.1108, found: 389.1079.

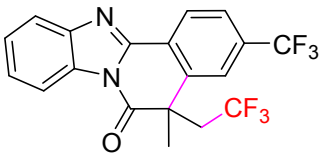
5-methyl-6-oxo-5-(2,2,2-trifluoroethyl)-5,6-dihydrobenzo[4,5]imidazo[2,1-*a*]isoq-uinoline-3-carbonitrile (3i)



4-(1-methacryloyl-1*H*-benzo[*d*]imidazol-2-yl)benzonitrile **1i** (86.2 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (201 – 203 °C) in 64% yield.

Rf (petroleum ether: EtOAc = 15:1): 0.2; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.63 (d, J = 8.1 Hz, 1H), 8.39 – 8.34 (m, 1H), 7.88 – 7.84 (m, 1H), 7.81 – 7.78 (m, 2H), 7.52 – 7.48 (m, 2H), 3.57 – 3.48 (m, 1H), 2.98 – 2.88 (m, 1H), 1.80 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.76, 147.30, 144.13, 139.44, 131.71, 131.52, 130.81, 127.20, 127.05, 126.80, 126.57, 124.81 (q, J = 278.8 Hz), 120.68, 117.89, 115.99, 115.14, 45.36 (q, J = 2.1 Hz), 44.08 (q, J = 27.9 Hz), 30.75; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.33; **HRMS** (ESI) calcd for $C_{19}H_{13}F_3N_3O$ $[M+H]^+$: 356.1005, found: 356.0970.

5-methyl-5-(2,2,2-trifluoroethyl)-3-(trifluoromethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3j)

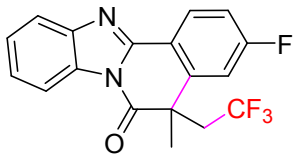


2-methyl-1-(2-(4-(trifluoromethyl)phenyl)-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1j** (99.1 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure.

The crude product was purified by column chromatography to afford the title compound as a white solid (147 – 149 °C) in 56% yield.

Rf (petroleum ether: EtOAc = 40:1): 0.2; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.65 (d, J = 8.2 Hz, 1H), 8.41 – 8.34 (m, 1H), 7.89 – 7.84 (m, 1H), 7.79 (d, J = 8.3 Hz, 1H), 7.71 (s, 1H), 7.52 – 7.46 (m, 2H), 3.58 – 3.48 (m, 1H), 3.02 – 2.92 (m, 1H), 1.80 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 170.24, 147.83, 144.09, 139.13, 133.39 (q, J = 32.9 Hz),131.52, 127.15, 126.71, 126.65, 125.83, 125.46 (q, J = 3.8 Hz), 124.88 (q, J = 278.8 Hz), 123.75 (q, J = 3.5 Hz), 123.61 (q, J = 272.7 Hz), 120.52, 115.96, 45.53 (q, J = 1.8 Hz), 44.02 (q, J = 27.8 Hz), 30.90; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.40, -62.99; **HRMS** (ESI) calcd for $C_{19}H_{13}F_6N_2O$ $[M+H]^+$: 399.0927, found: 399.0928.

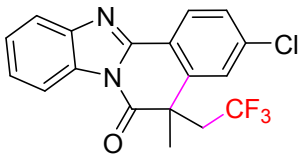
3-fluoro-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3k)



1-(2-(4-fluorophenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1k** (84.1 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (127 – 129 °C) in 53% yield.

Rf (petroleum ether: EtOAc = 30:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.57 – 8.49 (m, 1H), 8.37 – 8.31 (m, 1H), 7.84 – 7.78 (m, 1H), 7.49 – 7.40 (m, 2H), 7.28 – 7.21 (m, 1H), 7.18 – 7.13 (m, 1H), 3.55 – 3.40 (m, 1H), 2.94 – 2.79 (m, 1H), 1.76 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.48, 164.87 (d, J = 253.5 Hz), 148.57, 144.15, 141.28 (d, J = 7.7 Hz), 131.45, 129.02 (d, J = 9.0 Hz), 126.39, 126.02, 124.92 (q, J = 278.7 Hz), 120.08, 119.12 (d, J = 2.9 Hz), 116.67 (d, J = 22.4 Hz), 115.80, 113.65 (d, J = 23.5 Hz), 45.54 (q, J = 1.8 Hz), 44.16 (q, J = 28.0 Hz). 30.93; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -61.42, -106.25; **HRMS** (ESI) calcd for $C_{18}H_{13}F_4N_2O$ $[M+H]^+$: 349.0959, found: 349.0923.

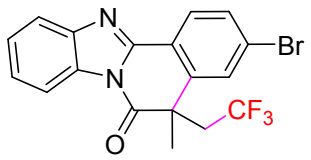
3-chloro-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3l)



1-(2-(4-chlorophenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1l** (89.0 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (194 – 196 °C) in 61% yield.

Rf (petroleum ether: EtOAc = 40:1): 0.2; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.45 (d, J = 8.4 Hz, 1H), 8.35 – 8.32 (m, 1H), 7.83 – 7.80 (m, 1H), 7.52 – 7.48 (m, 1H), 7.48 – 7.43 (m, 3H), 3.52 – 3.42 (m, 1H), 2.94 – 2.84 (m, 1H), 1.76 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.34, 148.43, 144.13, 140.23, 138.04, 131.47, 129.21, 127.84, 126.83, 126.43, 126.20, 124.91 (q, J = 278.8 Hz), 121.22, 120.18, 115.82, 45.37 (q, J = 2.1 Hz), 44.10 (q, J = 27.9 Hz), 30.83; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.37; **HRMS** (ESI) calcd for $C_{18}H_{13}ClF_3N_2O$ $[M+H]^+$: 365.0663, found: 365.0626.

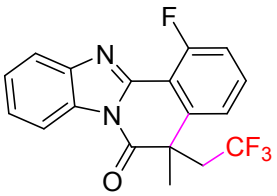
3-bromo-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3m)



1-(2-(4-bromophenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1m** (102.4 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (244 – 246 °C) in 52% yield.

Rf (petroleum ether: EtOAc = 30:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.41 – 8.32 (m, 2H), 7.84 – 7.80 (m, 1H), 7.69 – 7.64 (m, 1H), 7.63 (s, 1H), 7.49 – 7.42 (m, 2H), 3.54 – 3.40 (m, 1H), 2.97 – 2.82 (m, 1H), 1.77 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.30, 148.51, 144.15, 140.35, 132.10, 131.50, 129.81, 127.90, 126.47, 126.32, 126.25, 121.66, 124.92 (q, J = 279.0 Hz), 120.22, 115.84, 45.32 (q, J = 2.1 Hz), 44.12 (q, J = 27.9 Hz), 30.87; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -61.37; **HRMS** (ESI) calcd for $C_{18}H_{13}BrF_3N_2O$ $[M+H]^+$: 409.0158, found: 409.0120.

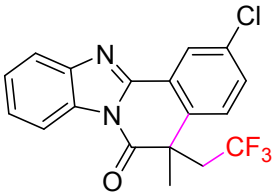
1-fluoro-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3n)



1-(2-(2-fluorophenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1n** (84.1 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg), CuI (30 mol%, 17.1 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a light yellow solid (226 – 228 °C) in 53% yield.

Rf (petroleum ether: EtOAc = 10:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.41 – 8.34 (m, 1H), 7.97 – 7.90 (m, 1H), 7.60 – 7.52 (m, 1H), 7.50 – 7.43 (m, 2H), 7.32 – 7.26 (m, 2H), 3.56 – 3.41 (m, 1H), 3.00 – 2.86 (m, 1H), 1.78 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 170.46, 160.67 (d, J = 263.0 Hz), 145.48 (d, J = 8.7 Hz), 144.40 (d, J = 2.0 Hz), 140.95, 132.37 (d, J = 9.3 Hz), 130.47, 126.53, 126.38, 124.94 (q, J = 278.6 Hz), 122.61 (d, J = 3.4 Hz), 120.83, 116.45 (d, J = 21.4 Hz), 115.73, 112.05 (d, J = 10.0 Hz), 45.27 (q, J = 1.8 Hz), 44.32 (q, J = 28.1 Hz), 31.29; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -61.27, -106.45; **HRMS** (ESI) calcd for $C_{18}H_{13}F_4N_2O$ $[M+H]^+$: 349.0959, found: 349.0949.

2-chloro-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3o)

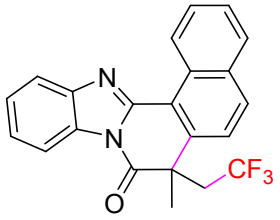


1-(2-(3-chlorophenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1o** (89.0 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (165 – 167 °C) in 75% yield.

Rf (petroleum ether: EtOAc = 20:1): 0.3; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.52 (s, 1H), 8.37 – 8.32 (m, 1H), 7.86 – 7.81 (m, 1H), 7.57 – 7.52 (m, 1H), 7.50 – 7.45 (m, 2H), 7.40 (d, J = 8.5 Hz, 1H), 3.54 – 3.42 (m, 1H), 2.95 – 2.84 (m, 1H), 1.75 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.57, 148.04, 144.03, 136.76, 134.93, 131.83, 131.50, 128.16, 126.50, 126.38, 126.12, 124.94 (q, J = 278.9 Hz), 124.18, 120.32, 115.86, 45.23 (q, J = 2.1 Hz), 43.96 (q, J = 27.8 Hz), 30.97; **^{19}F NMR**

(376 MHz, $CDCl_3$): δ -61.31; **HRMS** (ESI) calcd for $C_{18}H_{13}ClF_3N_2O$ $[M+H]^+$: 365.0663, found: 365.0654.

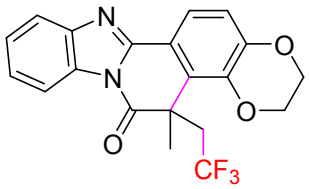
7-methyl-7-(2,2,2-trifluoroethyl)benzo[*h*]benzo[4,5]imidazo[2,1-*a*]isoquinolin-8(7*H*)-one (3p)



2-methyl-1-(2-(naphthalen-1-yl)-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1p** (93.7 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (238 – 240 °C) in 55% yield.

Rf (petroleum ether: EtOAc = 100:1): 0.1; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 10.55 (d, J = 8.7 Hz, 1H), 8.46 – 8.43 (m, 1H), 8.05 (d, J = 8.7 Hz, 1H), 7.97 – 7.94 (m, 1H), 7.93 (d, J = 7.8 Hz, 1H), 7.86 – 7.81 (m, 1H), 7.66 (t, J = 7.8 Hz, 1H), 7.54 (d, J = 8.7 Hz, 1H), 7.52 – 7.47 (m, 2H), 3.61 – 3.51 (m, 1H), 3.12 – 3.02 (m, 1H), 1.82 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.25, 149.56, 144.24, 139.05, 133.12, 132.66, 130.50, 129.05, 128.60, 128.39, 127.31, 126.28, 126.21, 125.07 (q, J = 279.2 Hz), 123.20, 120.44, 117.98, 115.90, 45.68 (q, J = 1.6 Hz), 43.85 (q, J = 28.0 Hz), 31.01; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -61.57; **HRMS** (ESI) calcd for $C_{22}H_{16}F_3N_2O$ $[M+H]^+$: 381.1209, found: 381.1189.

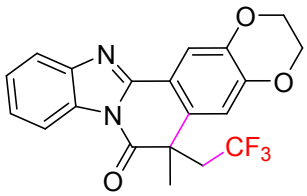
7-methyl-7-(2,2,2-trifluoroethyl)-4,5-dihydrobenzo[4,5]imidazo[2,1-*a*][1,4]dioxino[2,3-*f*]isoquinolin-8(7*H*)-one (3q)



1-(2-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1q** (96.1 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (149 – 151 °C) in 41% yield.

Rf (petroleum ether: EtOAc = 10:1): 0.15; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.32 (d, J = 7.6 Hz, 1H), 8.10 (d, J = 8.3 Hz, 1H), 7.77 (d, J = 7.5 Hz, 1H), 7.46 – 7.35 (m, 2H), 7.06 (d, J = 8.5 Hz, 1H), 4.40 – 4.33 (m, 4H), 3.76 – 3.67 (m, 1H), 3.40 – 3.30 (m, 1H), 1.82 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 172.05, 149.71, 146.68, 144.39, 141.57, 131.49, 126.88, 126.13, 125.71 (q, J = 278.3 Hz), 125.37, 120.20, 119.65, 118.70, 116.23, 115.73, 64.25, 64.05, 45.06 (q, J = 1.9 Hz), 40.43 (q, J = 27.4 Hz), 26.46; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -62.90; **HRMS** (ESI) calcd for $C_{20}H_{16}F_3N_2O_3$ $[M+H]^+$: 389.1108, found: 389.1108.

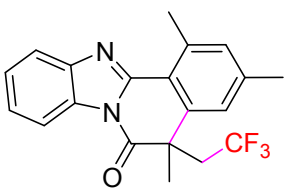
6-methyl-6-(2,2,2-trifluoroethyl)-2,3-dihydrobenzo[4,5]imidazo[2,1-*a*][1,4]dioxino[2,3-*g*]isoquinolin-7(6*H*)-one (3q')



1-(2-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1q** (96.1 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (182 – 184 ^{o}C) in 29% yield.

Rf (petroleum ether: EtOAc = 5:1): 0.2; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.31 (d, J = 7.5 Hz, 1H), 7.97 (s, 1H), 7.78 (d, J = 7.5 Hz, 1H), 7.45 – 7.37 (m, 2H), 6.91 (s, 1H), 4.35 (d, J = 7.4 Hz, 4H), 3.46 – 3.36 (m, 1H), 2.88 – 2.76 (m, 1H), 1.71 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.31, 149.34, 147.23, 144.23, 144.10, 132.42, 131.53, 126.13, 125.50, 125.08 (q, J = 278.8 Hz), 119.75, 116.14, 115.68, 115.19, 114.69, 64.87, 64.37, 45.01, 4.24 (q, J = 27.8 Hz), 31.29; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.32; **HRMS** (ESI) calcd for $C_{20}H_{16}F_3N_2O_3$ $[M+H]^+$: 389.1108, found: 389.1093.

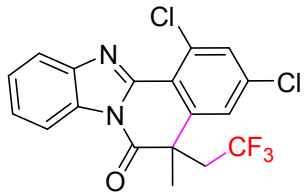
1,3,5-trimethyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3r)



1-(2-(2,4-dimethylphenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1r** (87.1 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (136 – 138 °C) in 79% yield.

Rf (petroleum ether: EtOAc = 100:1): 0.1; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.38 (dd, J = 6.6, 2.4 Hz, 1H), 7.83 (dd, J = 6.6, 2.4 Hz, 1H), 7.46 – 7.39 (m, 2H), 7.17 (s, 1H), 7.12 (s, 1H), 3.51 – 3.40 (m, 1H), 3.02 (s, 3H), 2.96 – 2.89 (m, 1H), 2.43 (s, 3H), 1.74 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.45, 149.83, 144.39, 140.73, 140.19, 139.70, 132.87, 130.67, 125.91, 125.73, 125.17 (q, J = 279.2 Hz), 125.06, 120.19, 118.49, 115.80, 45.27 (q, J = 1.7 Hz), 44.32 (q, J = 27.5 Hz), 31.65, 24.68, 21.69; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -61.16; **HRMS** (ESI) calcd for $C_{20}H_{18}F_3N_2O$ $[M+H]^+$: 359.1366, found: 359.1366.

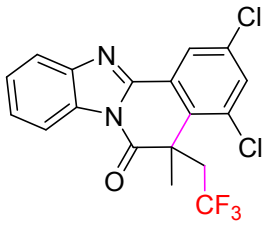
1,3-dichloro-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3s)



1-(2-(2,4-dichlorophenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1s** (99.4 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg), CuI (30 mol%, 17.1 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (240 – 242 °C) in 68% yield.

Rf (petroleum ether: EtOAc = 40:1): 0.2; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.39 – 8.34 (m, 1H), 7.94 – 7.89 (m, 1H), 7.61 (s, 1H), 7.50 – 7.46 (m, 2H), 7.39 (s, 1H), 3.54 – 3.45 (m, 1H), 2.93 – 2.83 (m, 1H), 1.78 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 169.55, 145.96, 143.95, 142.50, 136.75, 134.94, 132.06, 130.55, 126.94, 126.48, 125.88, 124.81 (q, J = 278.7 Hz), 121.03, 119.42, 115.83, 45.58, 44.38 (q, J = 28.2 Hz), 31.26; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -61.21; **HRMS** (ESI) calcd for $C_{18}H_{12}Cl_2F_3N_2O$ $[M+H]^+$: 399.0273, found: 399.0245.

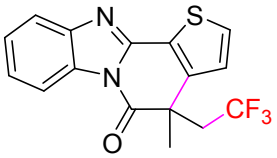
2,4-dichloro-5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3t)



1-(2-(3,5-dichlorophenyl)-1*H*-benzo[*d*]imidazol-1-yl)-2-methylprop-2-en-1-one **1t** (99.4 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (168 – 170 °C) in 69% yield.

Rf (petroleum ether: EtOAc = 50:1): 0.2; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.59 (d, J = 2.1 Hz, 1H), 8.38 – 8.31 (m, 1H), 7.87 – 7.81 (m, 1H), 7.60 (d, J = 2.1 Hz, 1H), 7.52 – 7.45 (m, 2H), 4.04 – 3.93 (m, 1H), 3.48 – 3.37 (m, 1H), 1.97 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 170.78, 147.24, 144.05, 135.52, 134.50, 134.45, 132.80, 131.50, 126.78, 126.65, 126.50, 125.77, 125.30 (q, J = 278.4 Hz), 120.44, 116.00, 46.41 (q, J = 1.7 Hz), 40.15 (q, J = 27.6 Hz), 25.61; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -62.90; **HRMS** (ESI) calcd for $C_{18}H_{12}Cl_2F_3N_2O$ $[M+H]^+$: 399.0273, found: 399.0253.

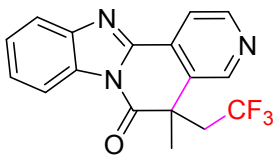
4-methyl-4-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[1,2-*a*]thieno[2,3-*c*]pyridin-5(4*H*)-one (3u)



2-methyl-1-(2-(thiophen-2-yl)-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1u** (80.5 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a yelow solid (137 – 139 °C) in 53% yield.

Rf (petroleum ether: EtOAc = 20:1): 0.1; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.33 – 8.27 (m, 1H), 7.80 – 7.75 (m, 1H), 7.63 (d, J = 5.1 Hz, 1H), 7.46 – 7.39 (m, 2H), 7.11 (d, J = 5.1 Hz, 1H), 3.47 – 3.34 (m, 1H), 2.87 – 2.76 (m, 1H), 1.71 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.60, 146.22, 144.21, 144.02, 131.12, 130.92, 126.23, 125.94, 125.61, 124.91 (q, J = 278.4 Hz), 124.19, 119.96, 115.34, 45.21 (q, J = 2.1 Hz), 44.01 (q, J = 28.0 Hz), 29.81; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -62.12; **HRMS** (ESI) calcd for $C_{16}H_{12}F_3N_2OS$ $[M+H]^+$: 337.0617, found: 337.0609.

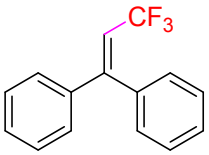
5-methyl-5-(2,2,2-trifluoroethyl)benzo[4,5]imidazo[2,1-*a*][2,6]naphthyridin-6(5*H*)-one (3v)



2-methyl-1-(2-(pyridin-4-yl)-1*H*-benzo[*d*]imidazol-1-yl)prop-2-en-1-one **1v** (79.0 mg, 0.3 mmol) was reacted with **Togni-II** (2.0 equiv, 189.6 mg) according to General Procedure. The crude product was purified by column chromatography to afford the title compound as a white solid (134 – 136 °C) in 56% yield.

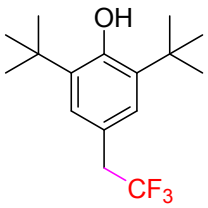
Rf (petroleum ether: EtOAc = 3:1): 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.91 – 8.72 (m, 2H), 8.41 – 8.34 (m, 1H), 8.30 (d, J = 5.1 Hz, 1H), 7.92 – 7.83 (m, 1H), 7.55 – 7.46 (m, 2H), 3.59 – 3.45 (m, 1H), 3.10 – 2.96 (m, 1H), 1.83 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 170.06, 149.43, 148.94, 146.90, 143.99, 132.73, 131.54, 129.50, 127.17, 126.76, 124.88 (q, J = 278.6 Hz), 120.80, 118.73, 116.01, 44.02 (q, J = 1.5 Hz), 43.80 (q, J = 27.9 Hz), 30.52; **^{19}F NMR** (376 MHz, $CDCl_3$): δ -61.36; **HRMS** (ESI) calcd for $C_{17}H_{13}F_3N_3O$ $[M+H]^+$: 332.1005, found: 332.1004.

(3,3,3-trifluoroprop-1-ene-1,1-diyl)dibenzene (4)[3]



^{1}H NMR (600 MHz, $CDCl_3$): δ 7.40 – 7.30 (m, 6H), 7.26 – 7.22 (m, 4H), 6.12 (q, J = 8.3 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 152.61 (q, J = 5.6 Hz), 140.27, 137.41, 129.53, 129.25 (q, J = 1.8 Hz), 128.63, 128.62, 128.18, 128.11, 123.24 (q, J = 270.7 Hz), 115.58 (q, J = 34.0 Hz); **^{19}F NMR** (565 MHz, $CDCl_3$): δ -55.57.

2,6-di-*tert*-butyl-4-(2,2,2-trifluoroethyl)phenol (5)[4]

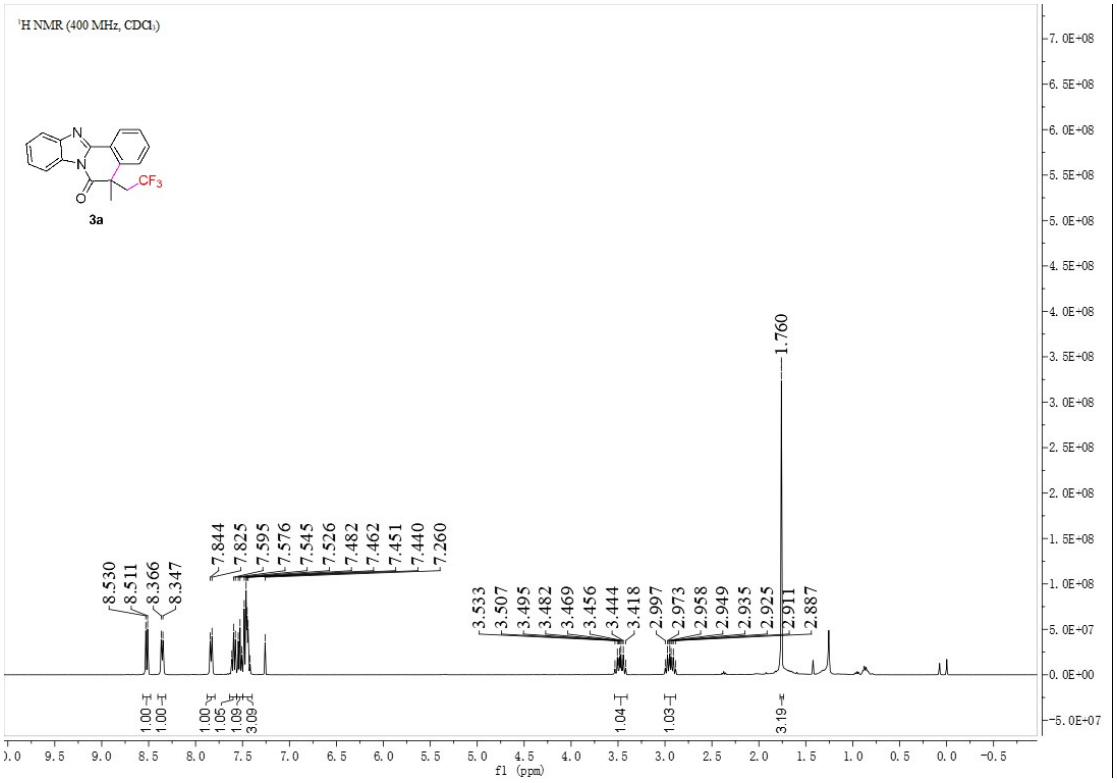


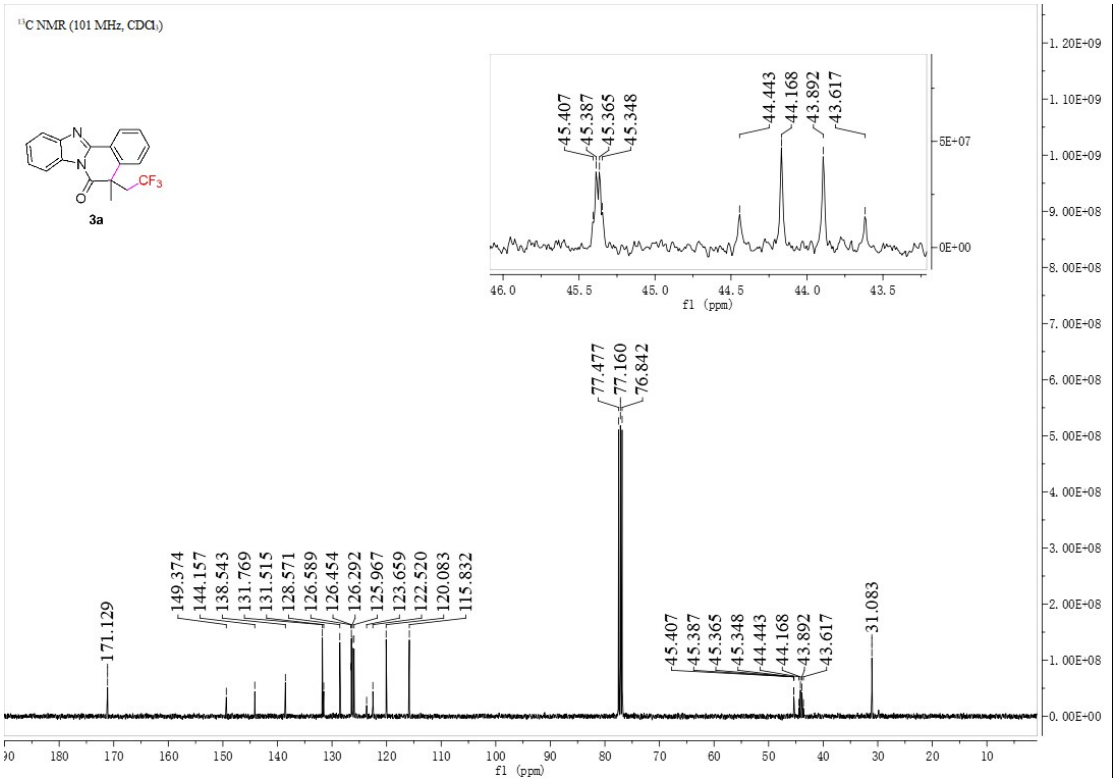
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.34 (s, 2H), 5.50 (s, 1H), 3.54 (q, J = 11.0 Hz, 2H), 1.71 (s, 19H); **^{19}F NMR** (565 MHz, $CDCl_3$): δ -66.14.

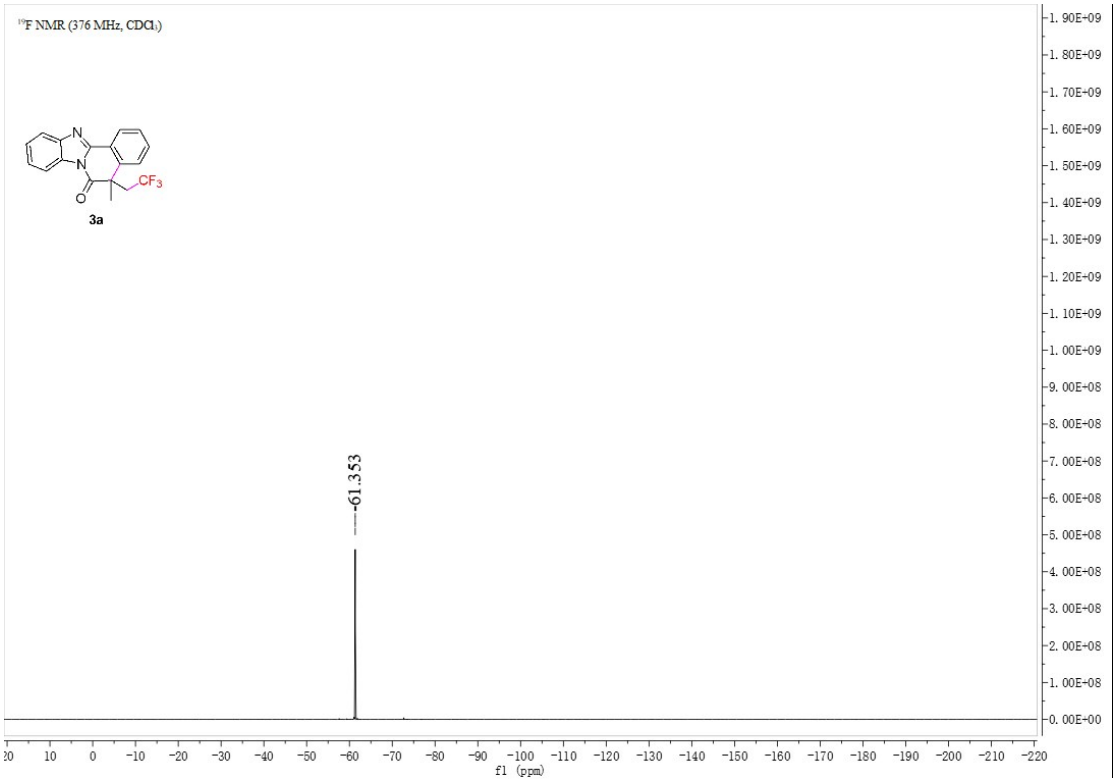
REFERENCES:

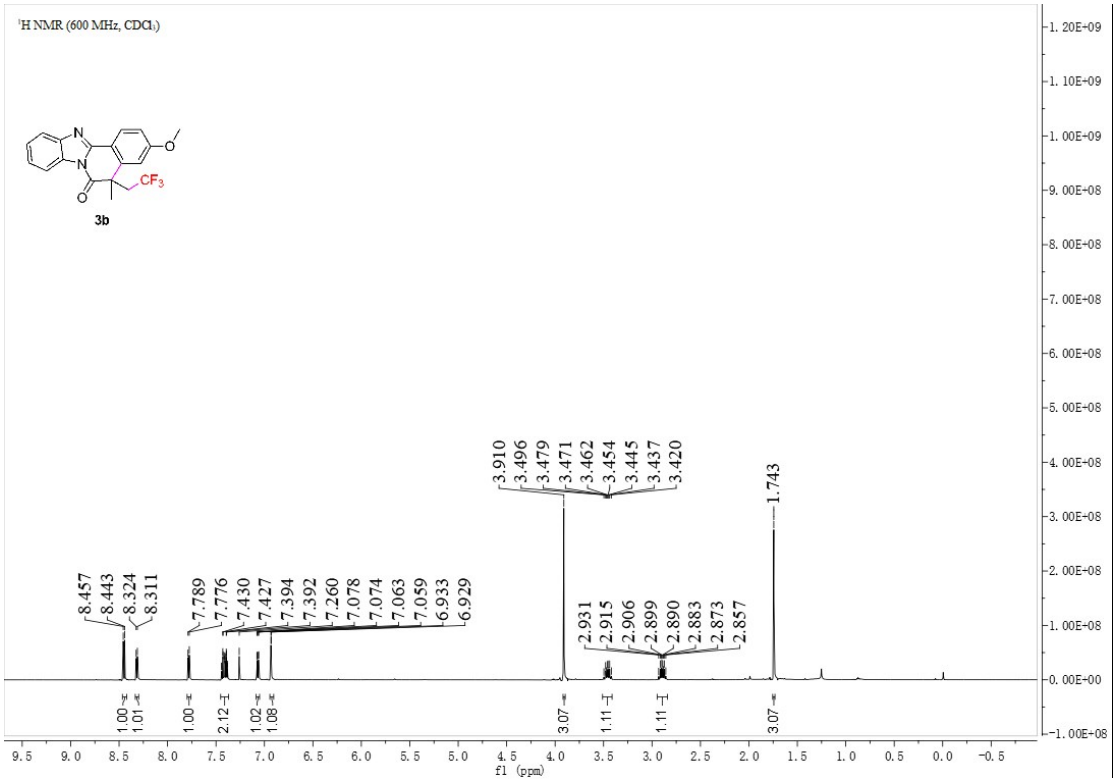
1. Y.-Q. Jiang, S.-H. Jia, X.-Y. Li, Y.-M. Sun, W. Li, W.-W. Zhang and G.-Q. Xu, *Chemical Papers*, 2018, **72**, 1265–1276.
2. (a) P. Gao, X.-B. Yan, T. Tao, F. Yang, T. He, X.-R., Song, X.-Y. Liu and Y.-M., Liang, *Chem. Eur. J.*, 2013, **19**, 14420 – 14424; (b) X. Bao, Q. Wang and J.-P. Zhu, *Nat. Commun.*, 2019, **10**, 769; (c) Z.-Z. Zhu, K. Chen, L.-Z. Yu, X.-Y. Tang and M. Shi, *Org. Lett.*, 2015, **17**, 5994–5997.
3. L.-H. Wu, K. Zhao, Z.-L. Shen and T.-P. Loh, *Org. Chem. Front.*, 2017, **4**, 1872–1875.
4. M. Winter, R. Schütz, A. Eitzinger, A. R. Ofial and M. Waser, *Eur. J. Org. Chem.*, 2020, **2020**, 3812–3817.

5. NMR Spectra of products






^{19}F NMR (376 MHz, CDCl$_3$)
CF$_3$
3a
-61.353
f1 (ppm)


^{1}H NMR (600 MHz, CDCl$_3$)
CF$_3$
3b
8.457
8.443
8.324
8.311
7.789
7.776
7.430
7.427
7.394
7.392
7.260
7.078
7.074
7.063
7.059
6.933
6.929
3.910
3.496
3.479
3.471
3.462
3.454
3.445
3.437
3.420
2.931
2.915
2.906
2.899
2.890
2.883
2.873
2.857
1.743
1.00
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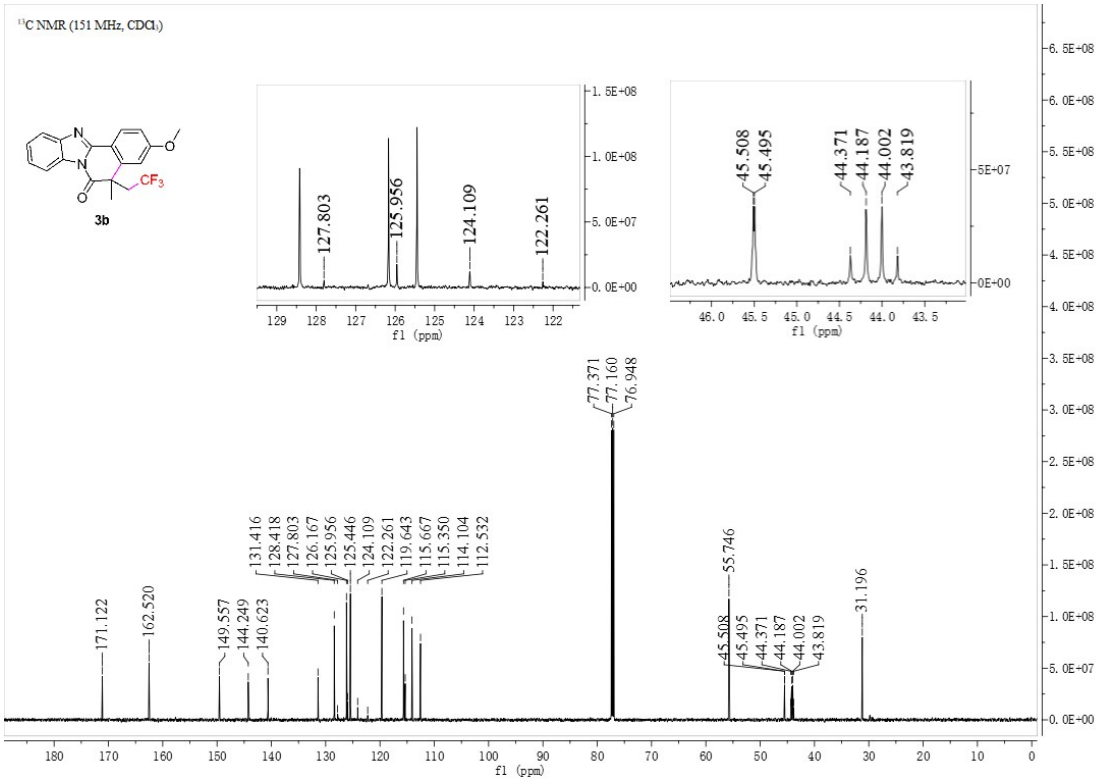
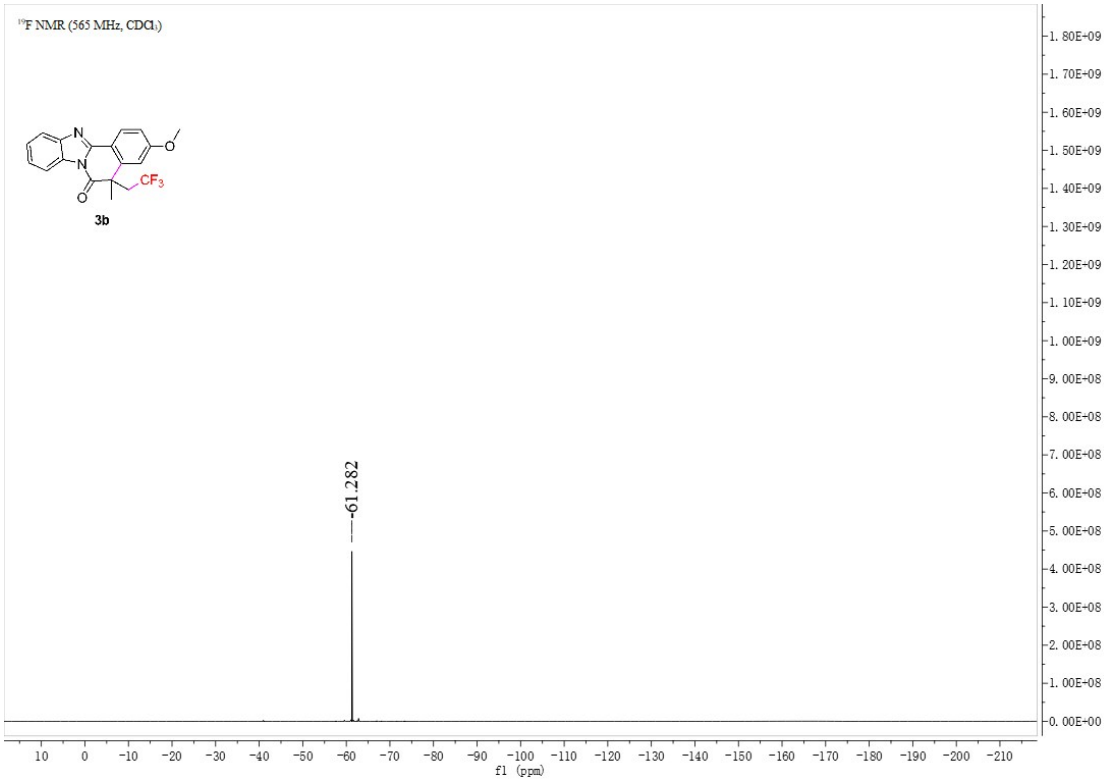
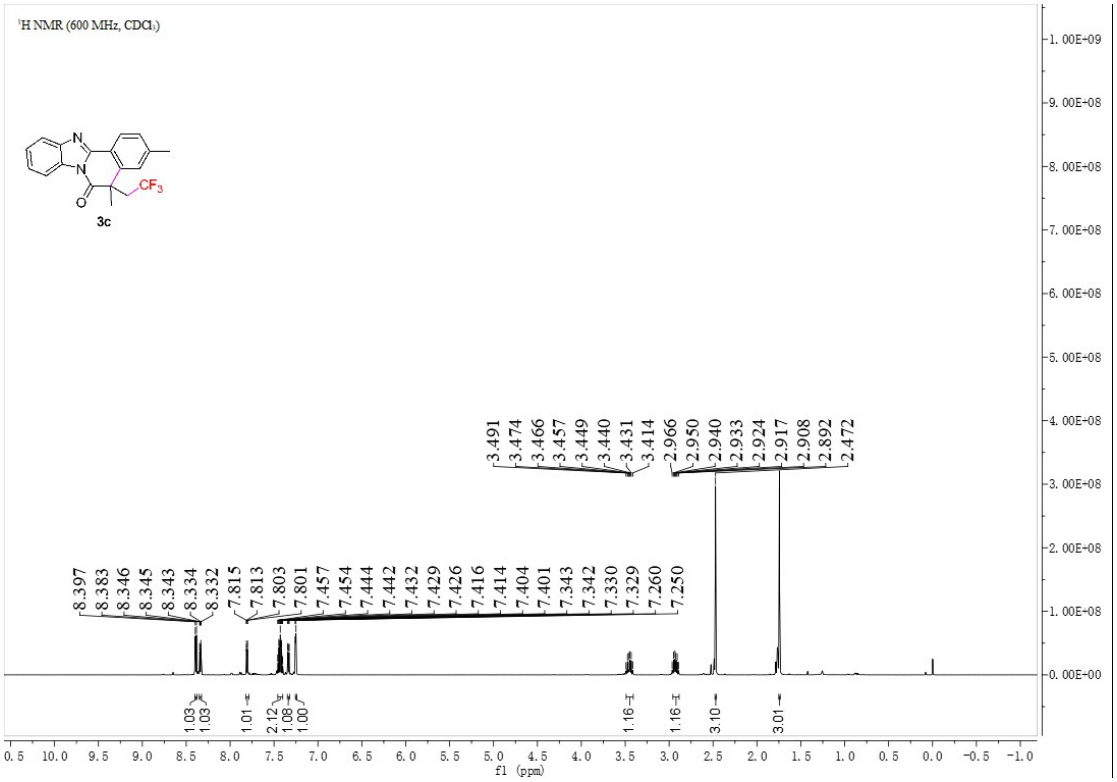
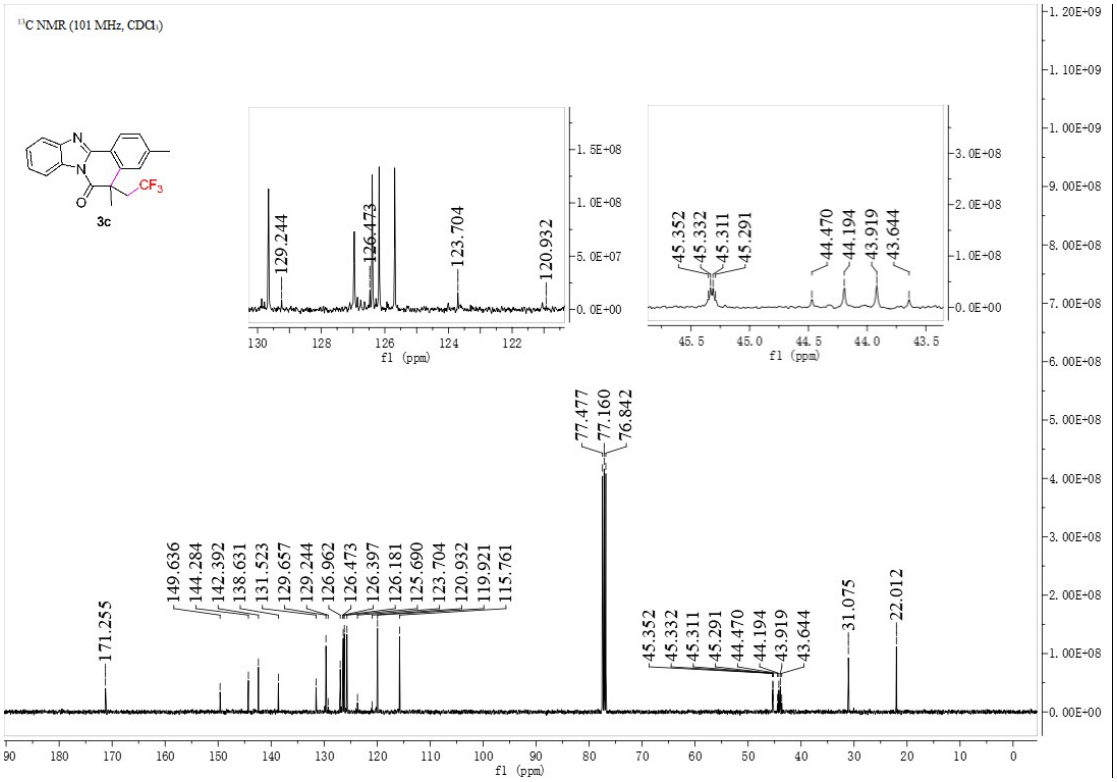
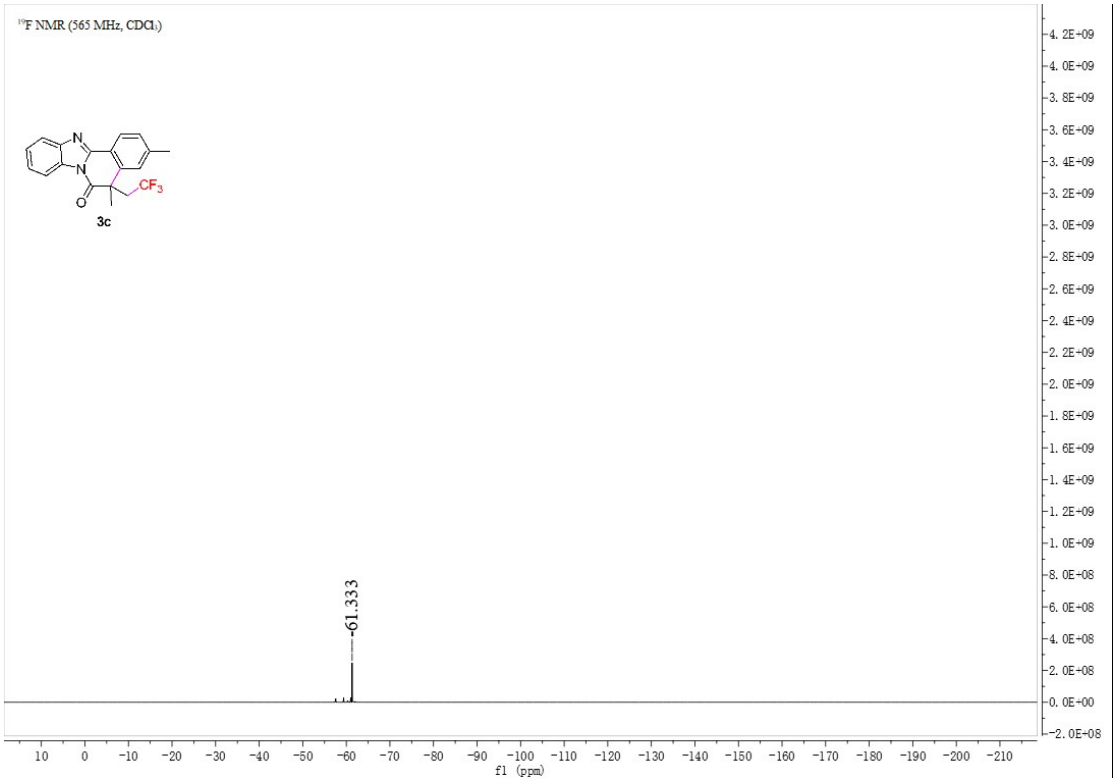
1.00
2.12
1.02
1.08
3.07
1.11
1.11
3.07
f1 (ppm)

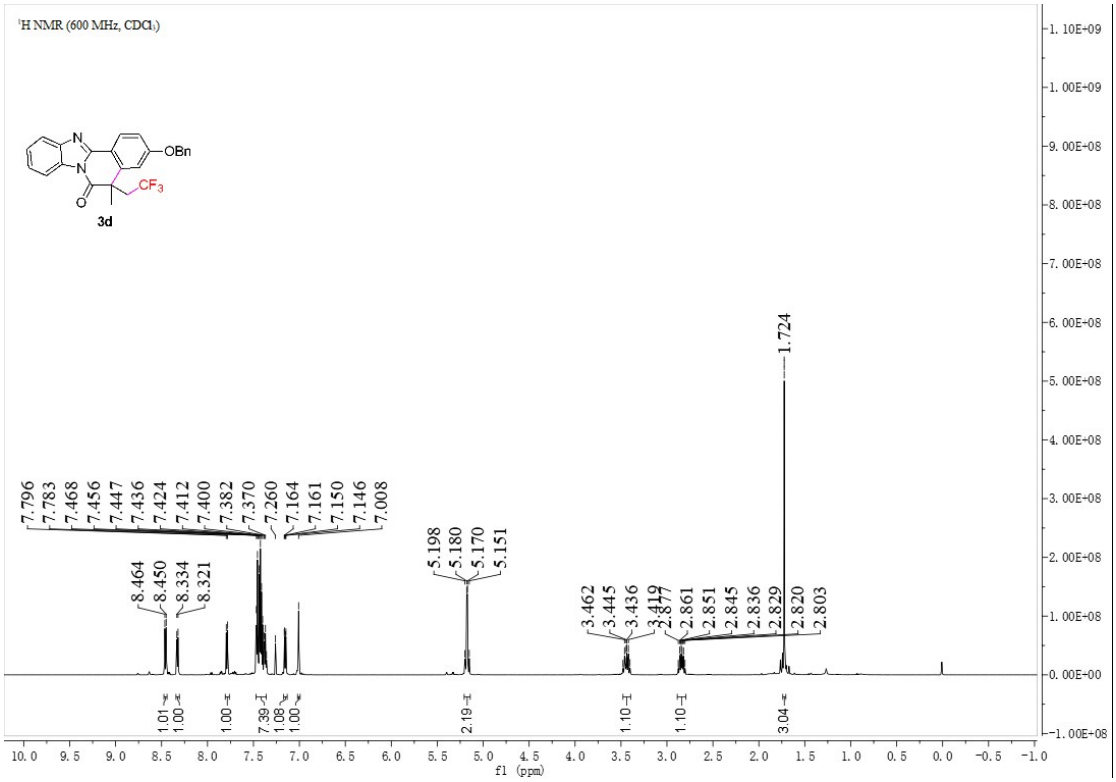

13C NMR (151 MHz, CDCl3)
3b
127.803
125.956
124.109
122.261
45.508
45.495
44.371
44.187
44.002
43.819
f1 (ppm)
77.371
77.160
76.948
131.416
128.418
127.803
126.167
125.956
125.446
124.109
122.261
119.643
115.667
115.350
114.104
112.532
171.122
162.520
149.557
144.249
140.623
55.746
31.196

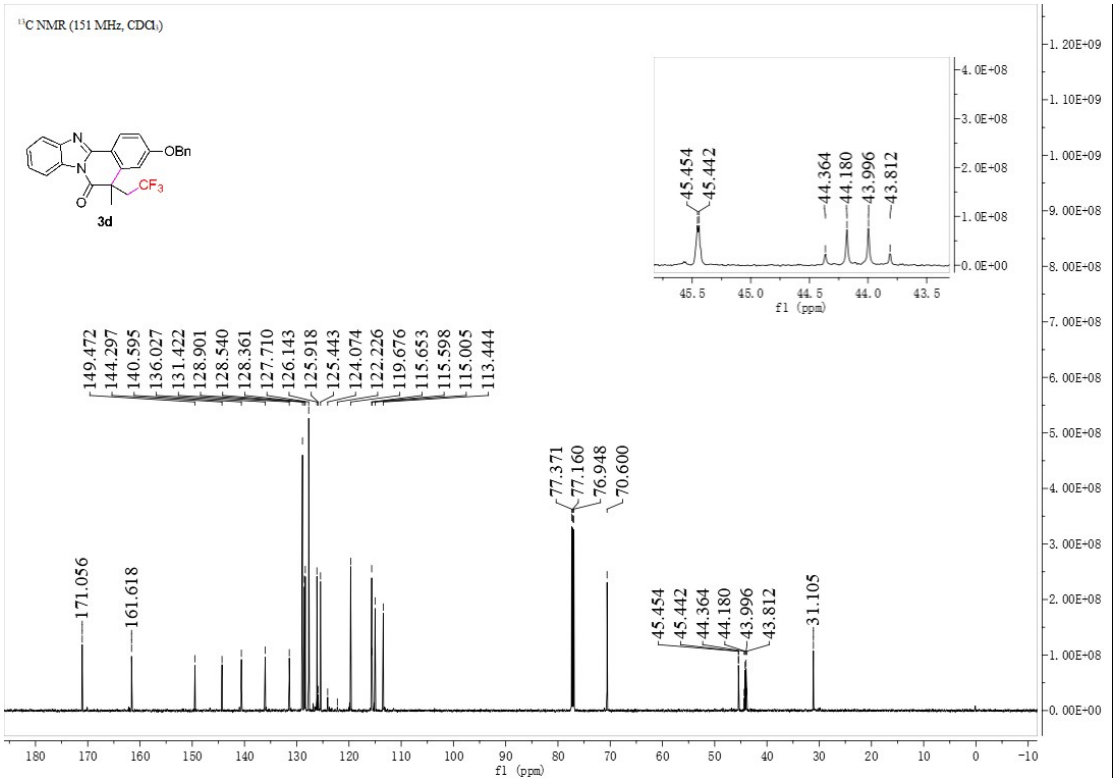

19F NMR (565 MHz, CDCl3)
3b
-61.282
f1 (ppm)

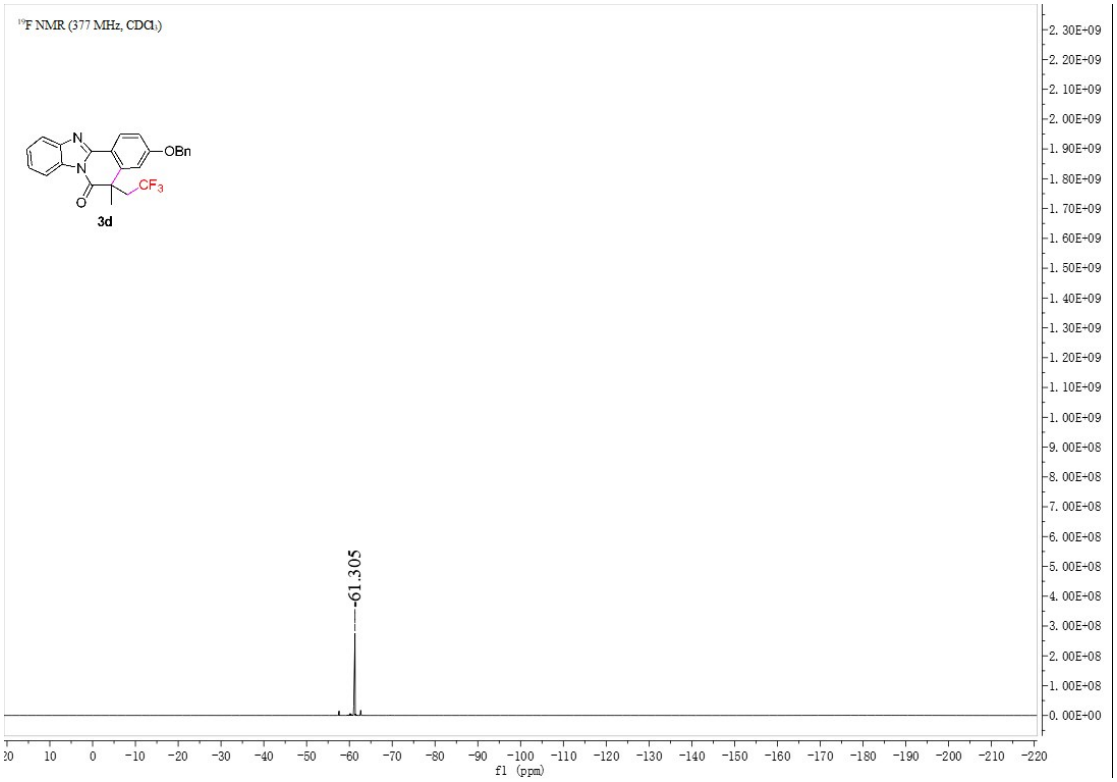

^{1}H NMR (600 MHz, CDCl$_3$)
3c
8.397
8.383
8.346
8.345
8.343
8.334
8.332
7.815
7.813
7.803
7.801
7.457
7.454
7.444
7.442
7.432
7.429
7.426
7.416
7.414
7.404
7.401
7.343
7.342
7.330
7.329
7.260
7.250
3.491
3.474
3.466
3.457
3.449
3.440
3.431
3.414
2.966
2.950
2.940
2.933
2.924
2.917
2.908
2.892
2.472
1.03
1.03
1.01
2.12
1.08
1.00
1.16
1.16
3.10
3.01
f1 (ppm)

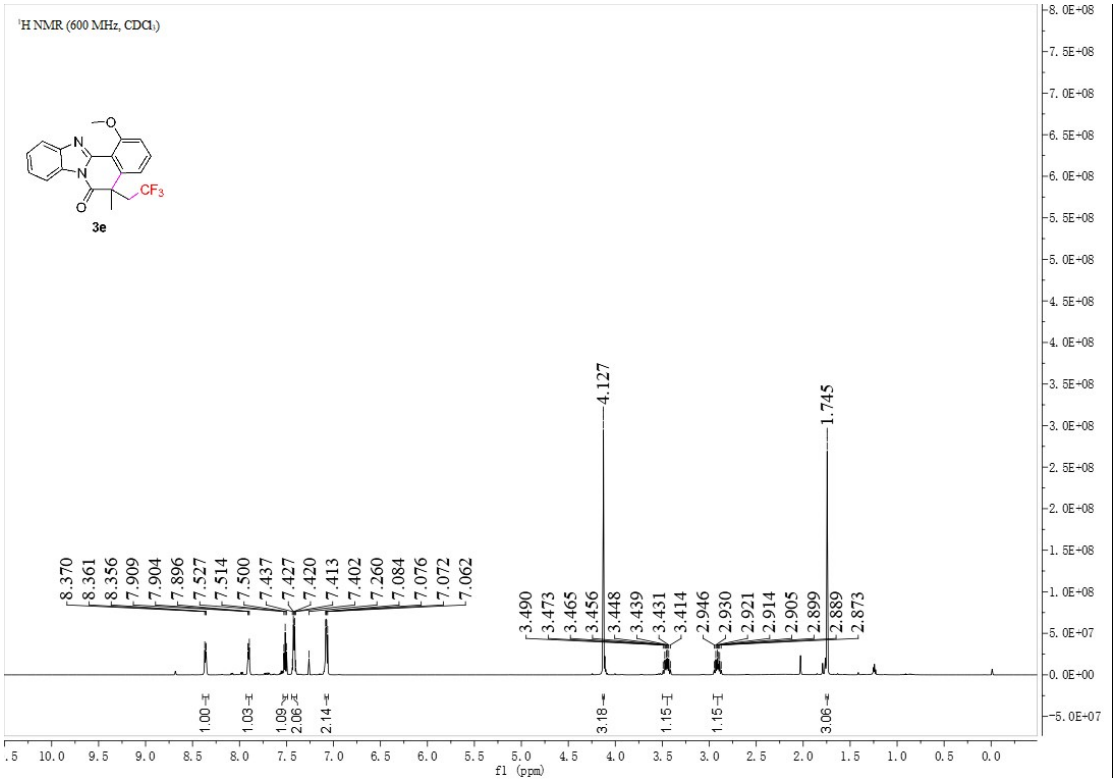

^{13}C NMR (101 MHz, CDCl$_3$)
3c
129.244
126.473
123.704
120.932
45.352
45.332
45.311
45.291
44.470
44.194
43.919
43.644
f1 (ppm)
77.477
77.160
76.842
149.636
144.284
142.392
138.631
131.523
129.657
129.244
126.962
126.473
126.397
126.181
125.690
123.704
120.932
119.921
115.761
171.255
31.075
22.012
f1 (ppm)

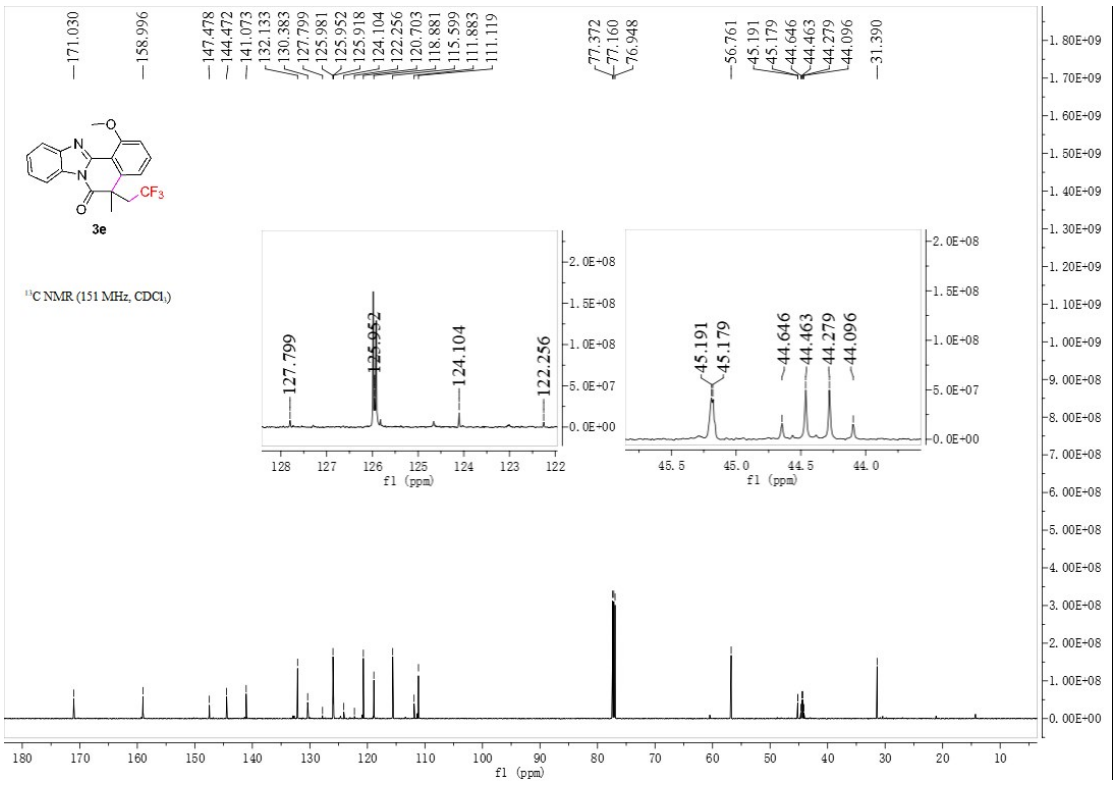

^{19}F NMR (565 MHz, CDCl$_3$)
3c
-61.333
f1 (ppm)

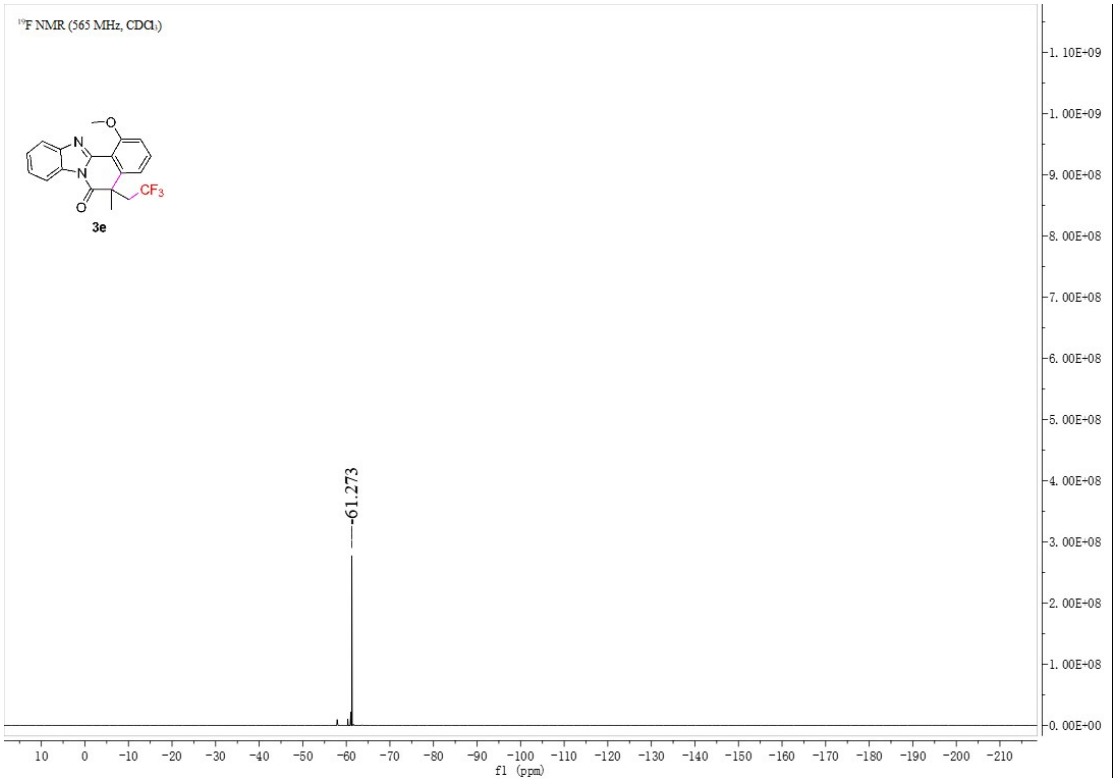

^{1}H NMR (600 MHz, CDCl$_3$)
3d
7.796
7.783
7.468
7.456
7.447
7.436
7.424
7.412
7.400
7.382
7.370
7.260
7.164
7.161
7.150
7.146
7.008
8.464
8.450
8.334
8.321
5.198
5.180
5.170
5.151
3.462
3.445
3.436
3.419
2.877
2.861
2.851
2.845
2.836
2.829
2.820
2.803
1.724
1.01
1.00
1.00
7.39
1.08
1.00
2.19
1.10
1.10
3.04
f1 (ppm)


13C NMR (151 MHz, CDCl3)
OBn
CF3
O
3d
45.454
45.442
44.364
44.180
43.996
43.812
f1 (ppm)
149.472
144.297
140.595
136.027
131.422
128.901
128.540
128.361
127.710
126.143
125.918
125.443
124.074
122.226
119.676
115.653
115.598
115.005
113.444
77.371
77.160
76.948
70.600
171.056
161.618
45.454
45.442
44.364
44.180
43.996
43.812
31.105
f1 (ppm)


19F NMR (377 MHz, CDCl3)
OBn
CF3
O
3d
-61.305
f1 (ppm)


¹H NMR (600 MHz, CDCl₃)
3e
8.370
8.361
8.356
7.909
7.904
7.896
7.527
7.514
7.500
7.437
7.427
7.420
7.413
7.402
7.260
7.084
7.076
7.072
7.062
3.490
3.473
3.465
3.456
3.448
3.439
3.431
3.414
2.946
2.930
2.921
2.914
2.905
2.899
2.889
2.873
4.127
1.745
1.00
1.03
1.09
2.08
2.14
3.18
1.15
1.15
3.06
f1 (ppm)


171.030
158.996
147.478
144.472
141.073
132.133
130.383
127.799
125.981
125.952
125.918
124.104
122.256
120.703
118.881
115.599
111.883
111.119
77.372
77.160
76.948
56.761
45.191
45.179
44.646
44.463
44.279
44.096
31.390
3e
¹³C NMR (151 MHz, CDCl₃)
127.799
125.952
124.104
122.256
45.191
45.179
44.646
44.463
44.279
44.096
f1 (ppm)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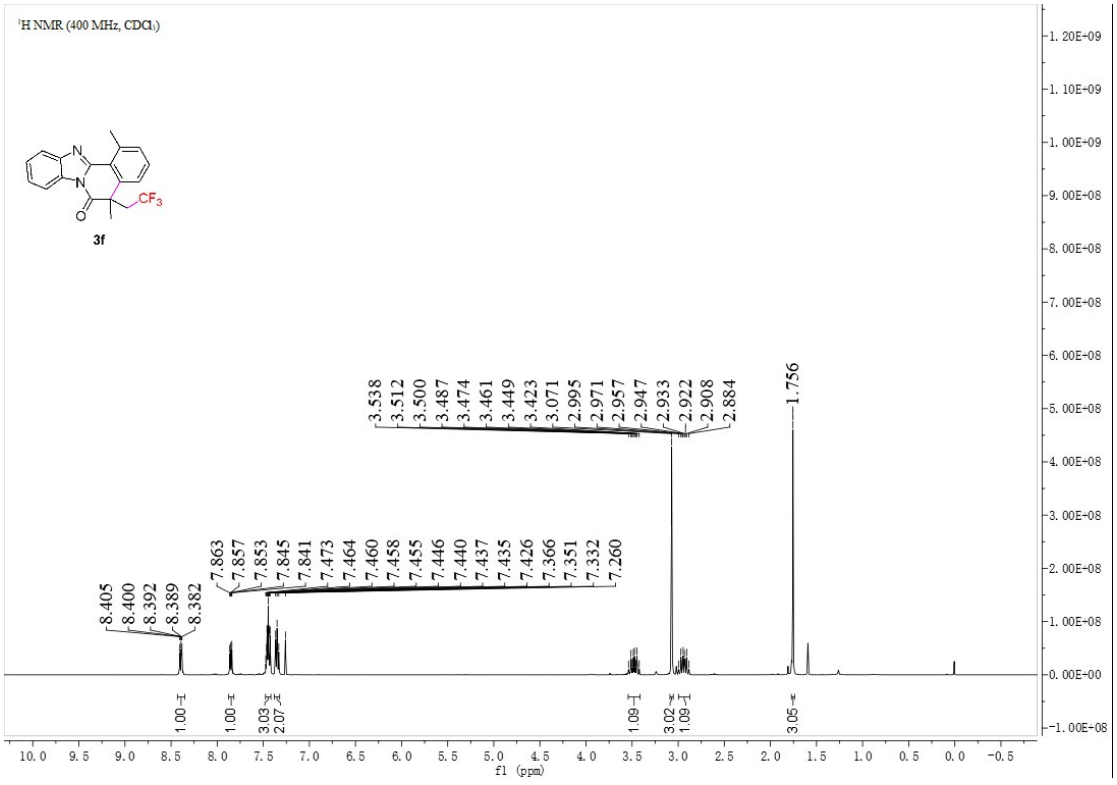
3e
-61.273
f1 (ppm)


^{1}H NMR (400 MHz, CDCl$_3$)
3f
8.405
8.400
8.392
8.389
8.382
7.863
7.857
7.853
7.845
7.841
7.473
7.464
7.460
7.458
7.455
7.446
7.440
7.437
7.435
7.426
7.366
7.351
7.332
7.260
3.538
3.512
3.500
3.487
3.474
3.461
3.449
3.423
3.071
2.995
2.971
2.957
2.947
2.933
2.922
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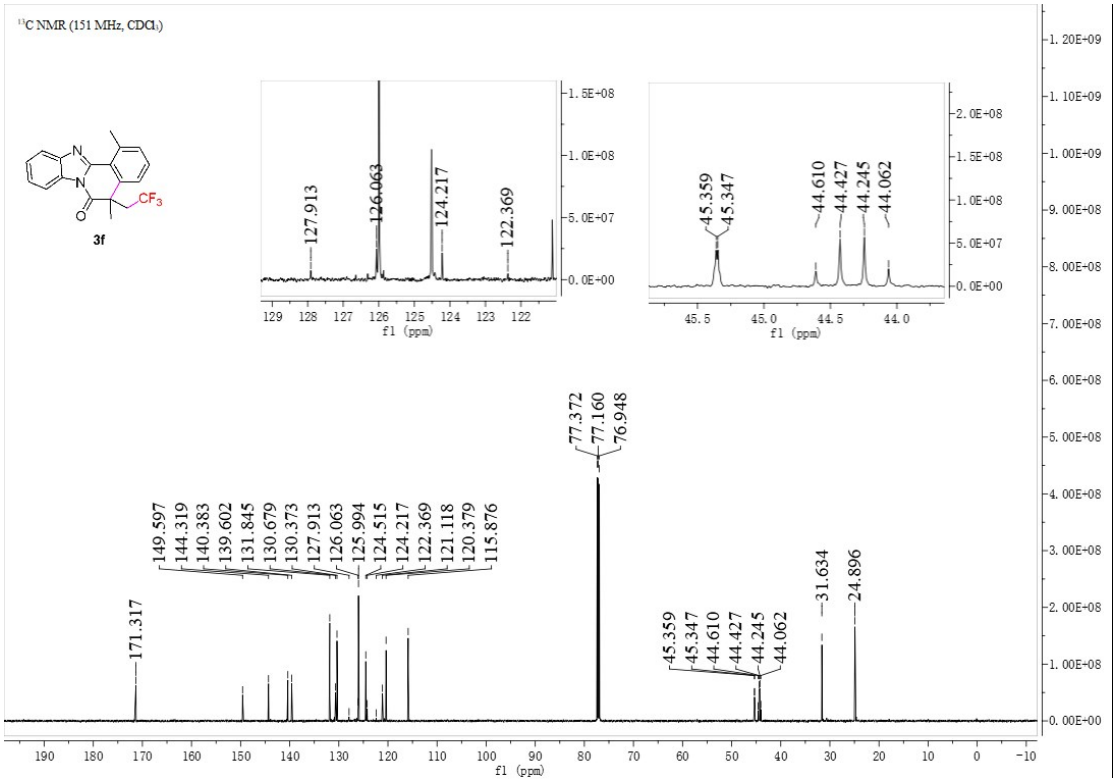
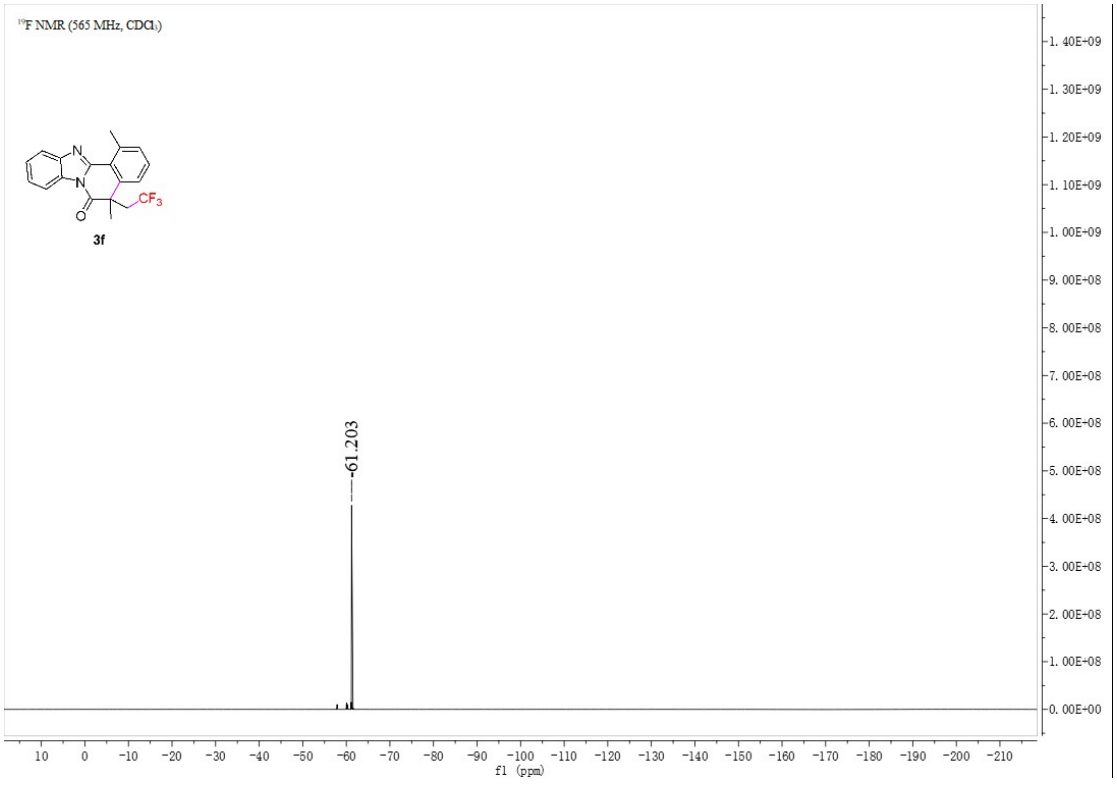
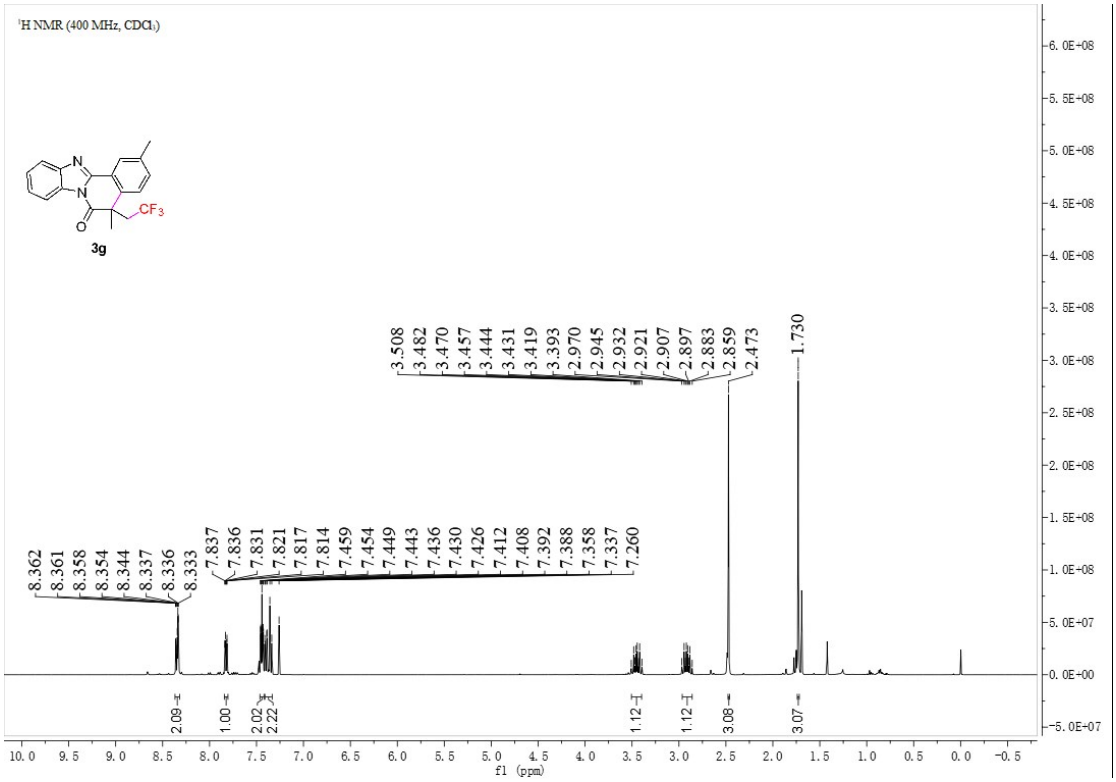
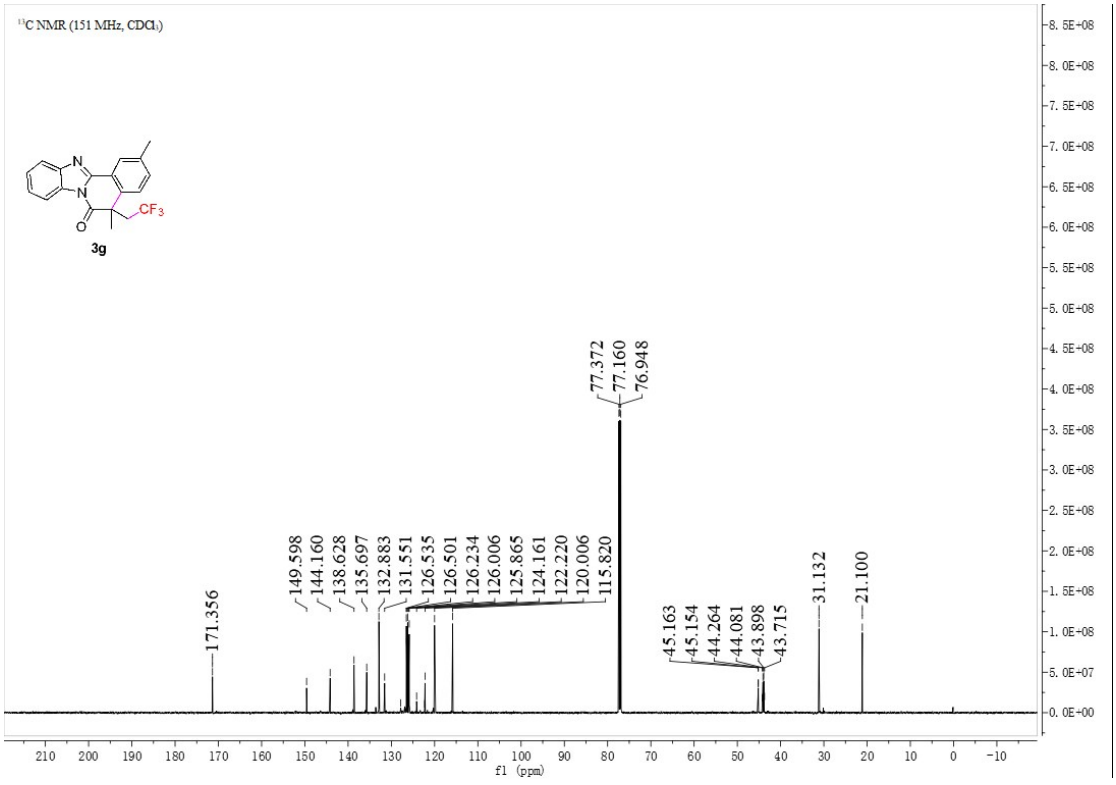
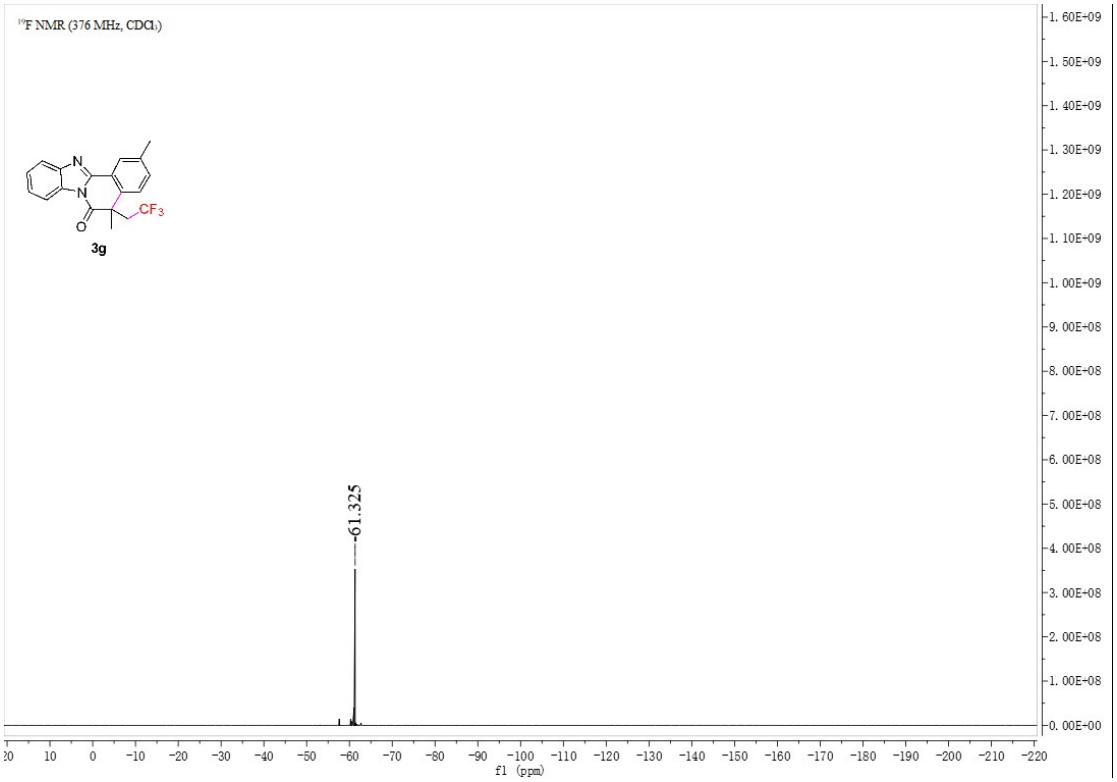
1.09
3.02
1.09
3.05
f1 (ppm)

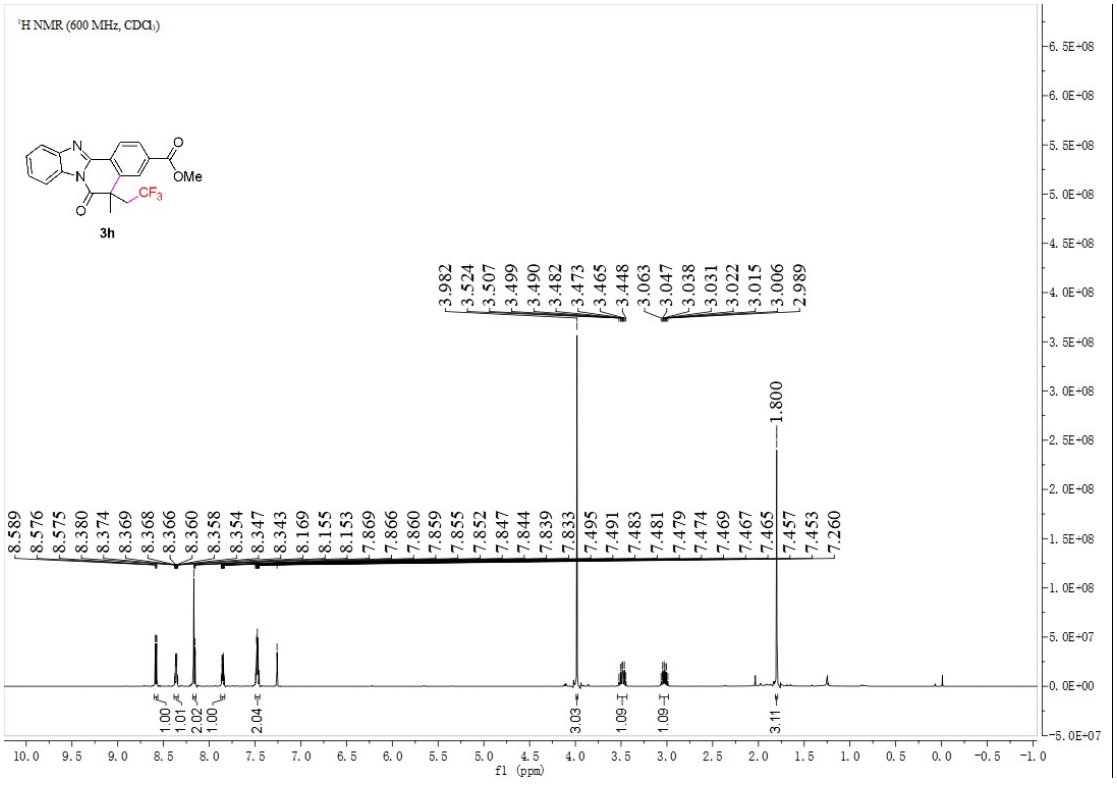

13C NMR (151 MHz, CDCl3)
3f
127.913
126.063
124.217
122.369
45.359
45.347
44.610
44.427
44.245
44.062
f1 (ppm)
77.372
77.160
76.948
149.597
144.319
140.383
139.602
131.845
130.679
130.373
127.913
126.063
125.994
124.515
124.217
122.369
121.118
120.379
115.876
171.317
31.634
24.896
f1 (ppm)

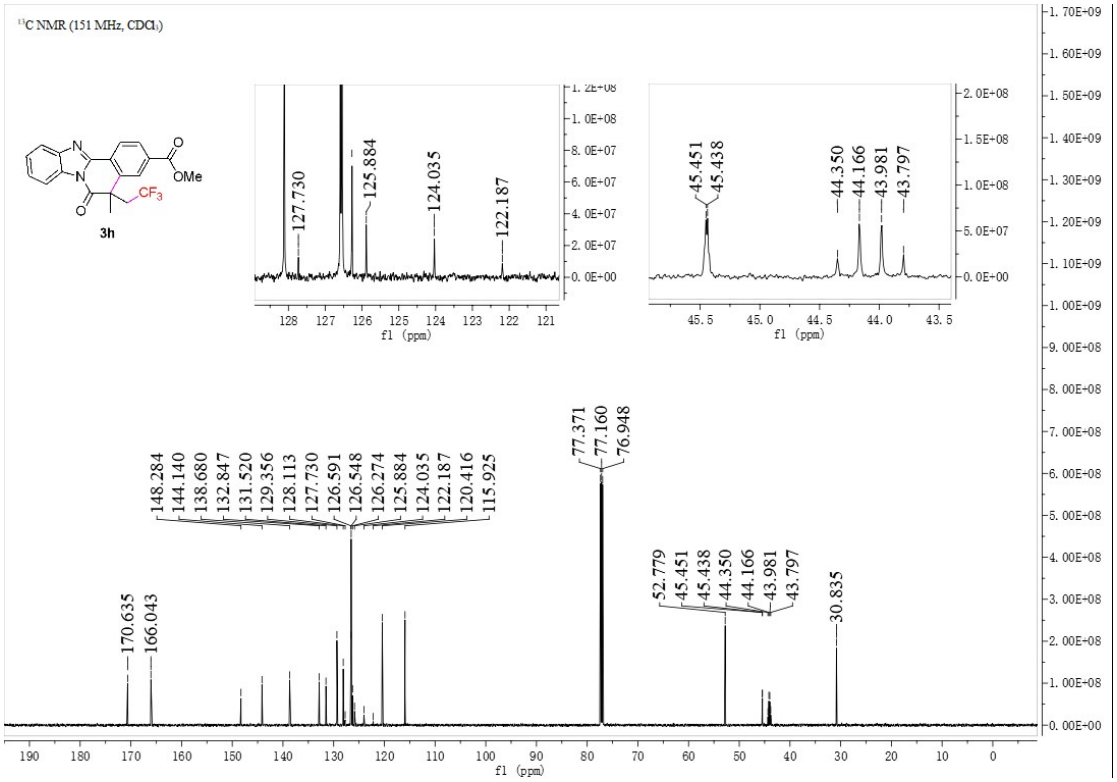

19F NMR (565 MHz, CDCl3)
3f
-61.203
f1 (ppm)

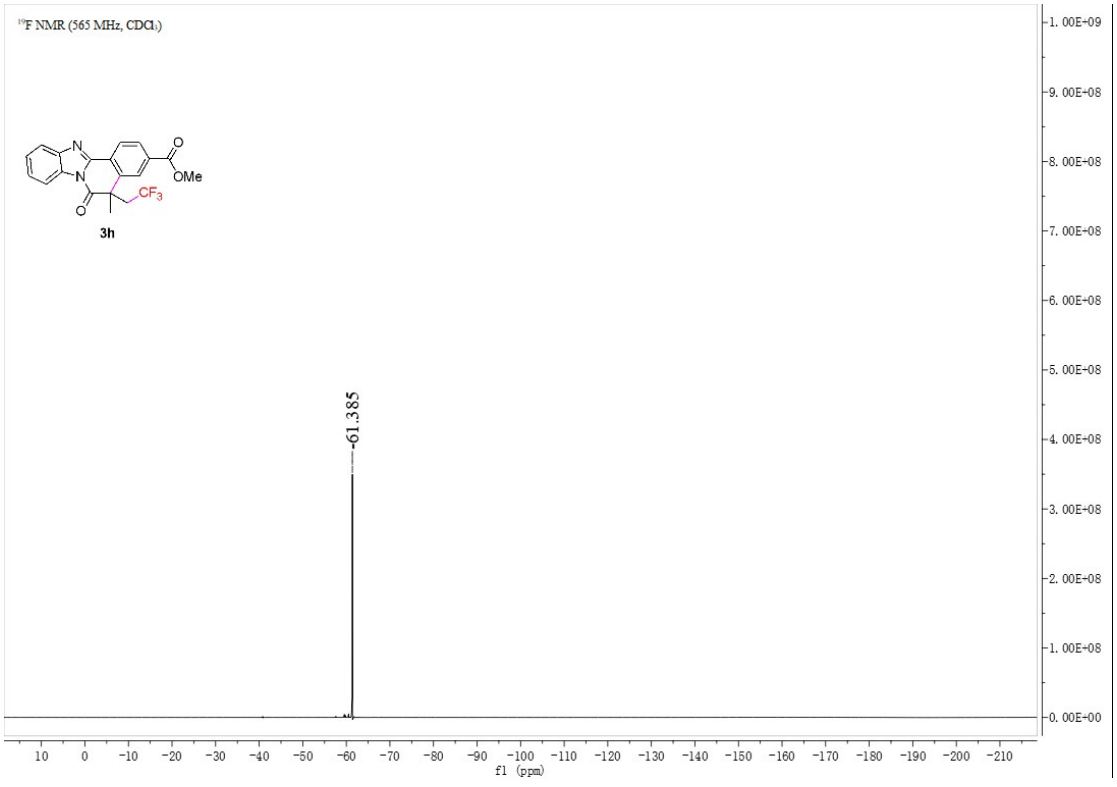

¹H NMR (400 MHz, CDCl₃)
3g
8.362
8.361
8.358
8.354
8.344
8.337
8.336
8.333
7.837
7.836
7.831
7.821
7.817
7.814
7.459
7.454
7.449
7.443
7.436
7.430
7.426
7.412
7.408
7.392
7.388
7.358
7.337
7.260
3.508
3.482
3.470
3.457
3.444
3.431
3.419
3.393
2.970
2.945
2.932
2.921
2.907
2.897
2.883
2.859
2.473
1.730
2.09
1.00
2.02
2.22
1.12
1.12
3.08
3.07
f1 (ppm)

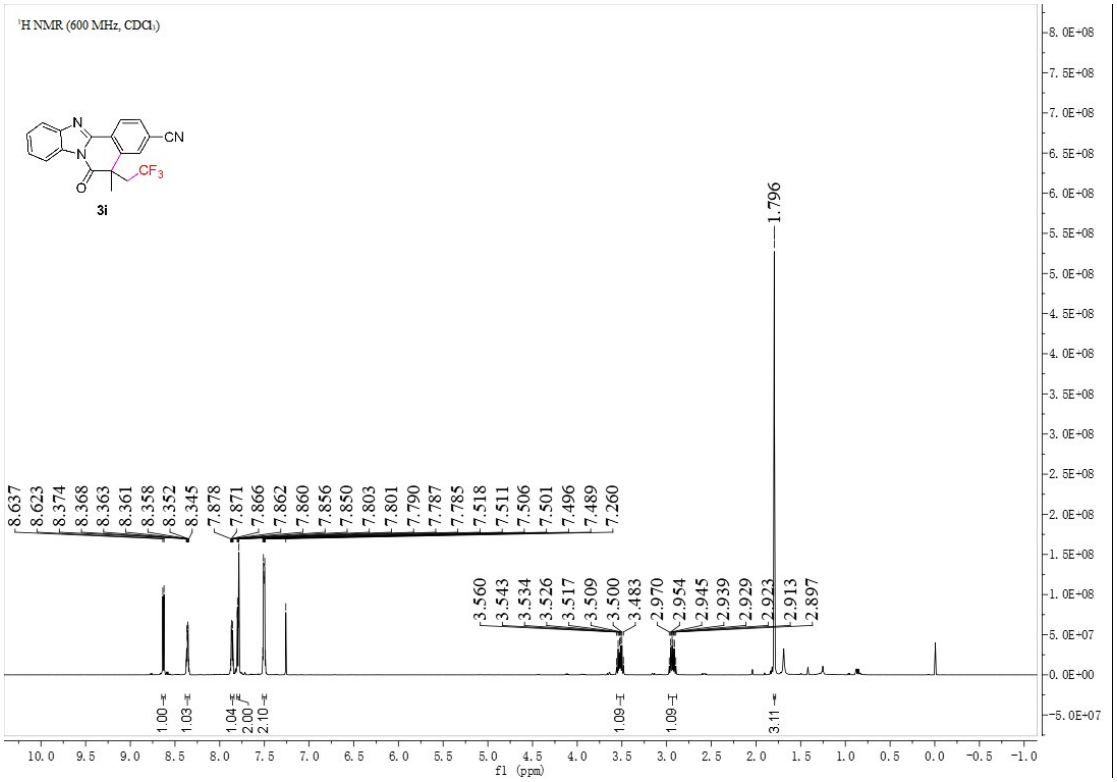

¹³C NMR (151 MHz, CDCl₃)
3g
171.356
149.598
144.160
138.628
135.697
132.883
131.551
126.535
126.501
126.234
126.006
125.865
124.161
122.220
120.006
115.820
77.372
77.160
76.948
45.163
45.154
44.264
44.081
43.898
43.715
31.132
21.100
f1 (ppm)

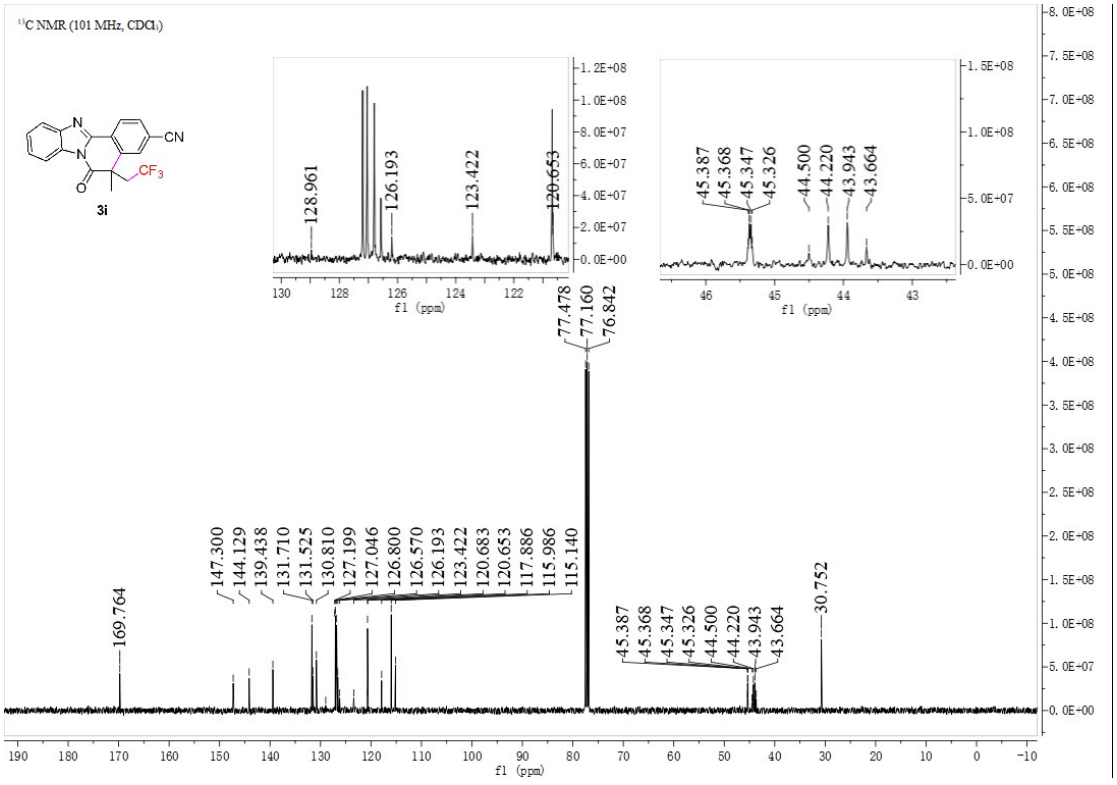

^{19}F NMR (376 MHz, CDCl$_3$)
CF$_3$
3g
-61.325
f1 (ppm)

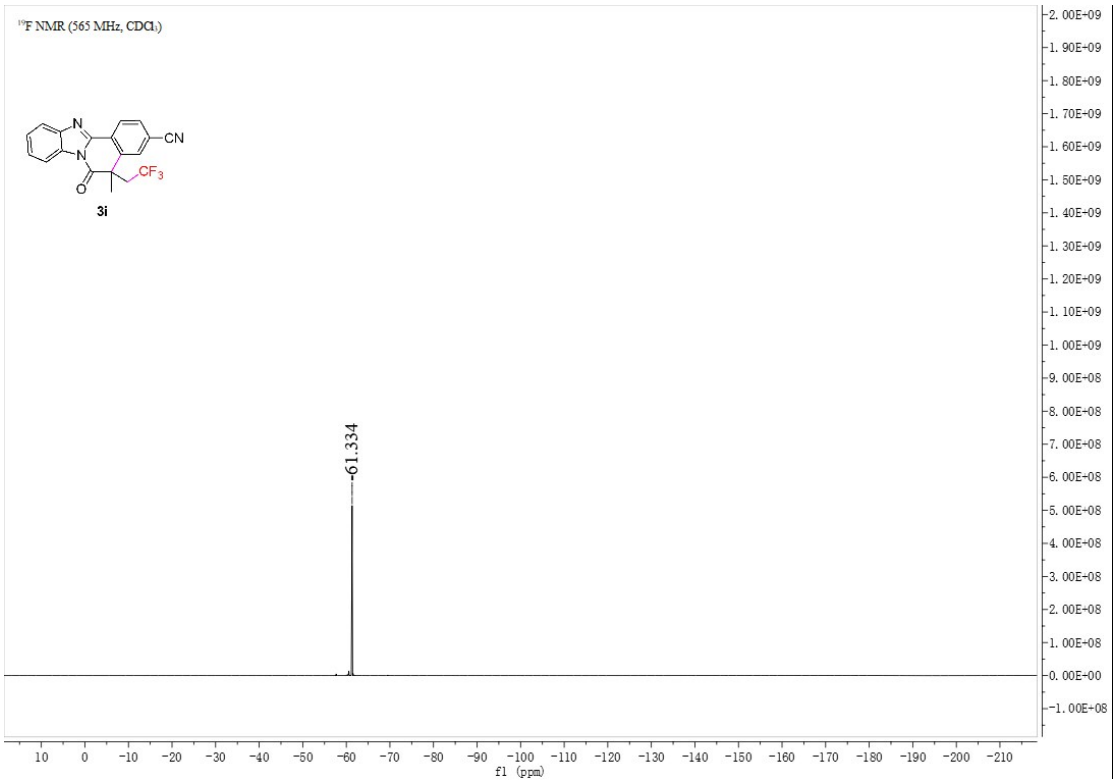

^{1}H NMR (600 MHz, CDCl$_3$)
OMe
CF$_3$
3h
3.982
3.524
3.507
3.499
3.490
3.482
3.473
3.465
3.448
3.063
3.047
3.038
3.031
3.022
3.015
3.006
2.989
1.800
8.589
8.576
8.575
8.380
8.374
8.369
8.368
8.366
8.360
8.358
8.354
8.347
8.343
8.169
8.155
8.153
7.869
7.866
7.860
7.859
7.855
7.852
7.847
7.844
7.839
7.833
7.495
7.491
7.483
7.481
7.479
7.474
7.469
7.467
7.465
7.457
7.453
7.260
1.00
1.01
2.02
1.00
2.04
3.03
1.09
1.09
3.11
f1 (ppm)

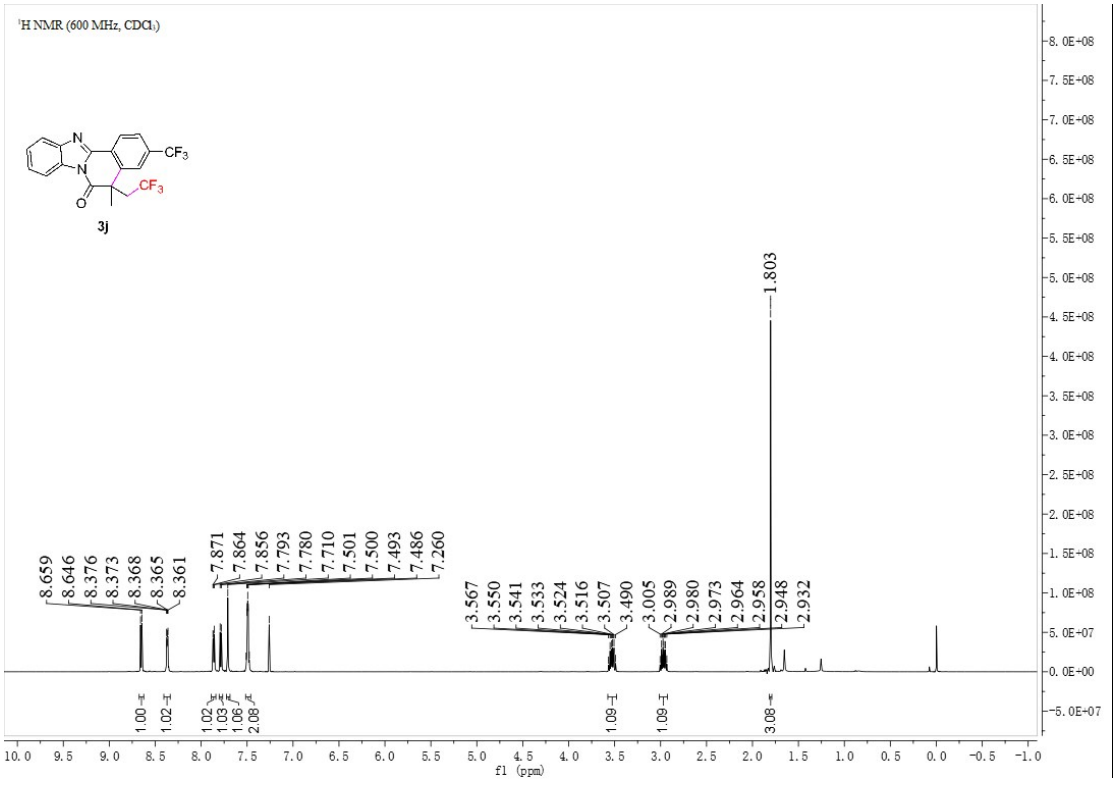

13C NMR (151 MHz, CDCl3)
3h
127.730
125.884
124.035
122.187
45.451
45.438
44.350
44.166
43.981
43.797
148.284
144.140
138.680
132.847
131.520
129.356
128.113
127.730
126.591
126.548
126.274
125.884
124.035
122.187
120.416
115.925
77.371
77.160
76.948
52.779
45.451
45.438
44.350
44.166
43.981
43.797
30.835
170.635
166.043
f1 (ppm)

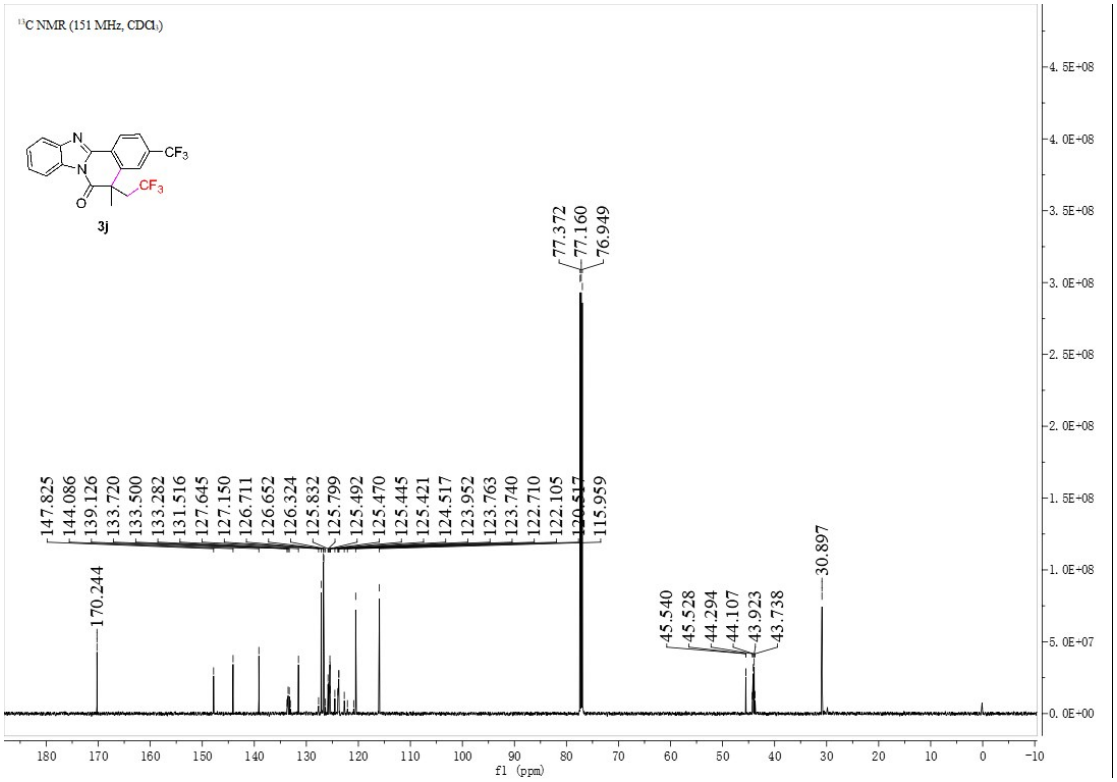

19F NMR (565 MHz, CDCl3)
3h
-61.385
f1 (ppm)

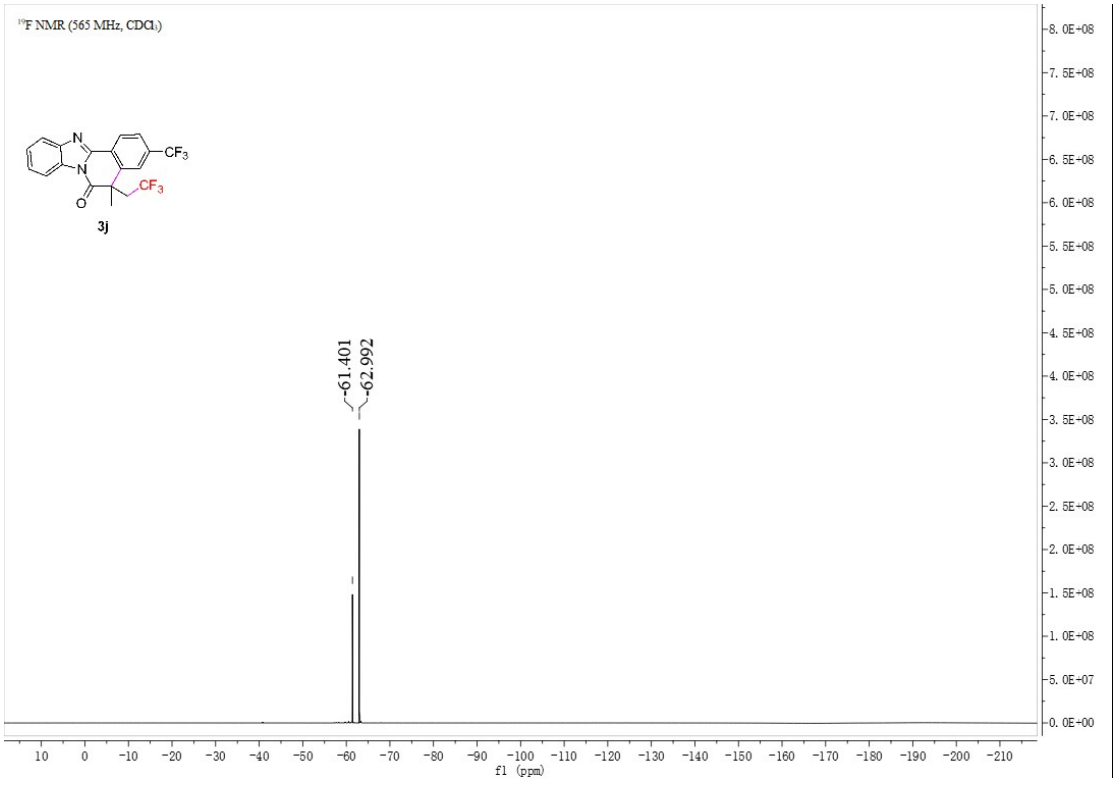

^{1}H NMR (600 MHz, CDCl$_3$)
3i
8.637
8.623
8.374
8.368
8.363
8.361
8.358
8.352
8.345
7.878
7.871
7.866
7.862
7.860
7.856
7.850
7.803
7.801
7.790
7.787
7.785
7.518
7.511
7.506
7.501
7.496
7.489
7.260
3.560
3.543
3.534
3.526
3.517
3.509
3.500
3.483
2.970
2.954
2.945
2.939
2.929
2.923
2.913
2.897
1.796
1.00
1.03
1.04
2.00
2.10
1.09
1.09
3.11
f1 (ppm)

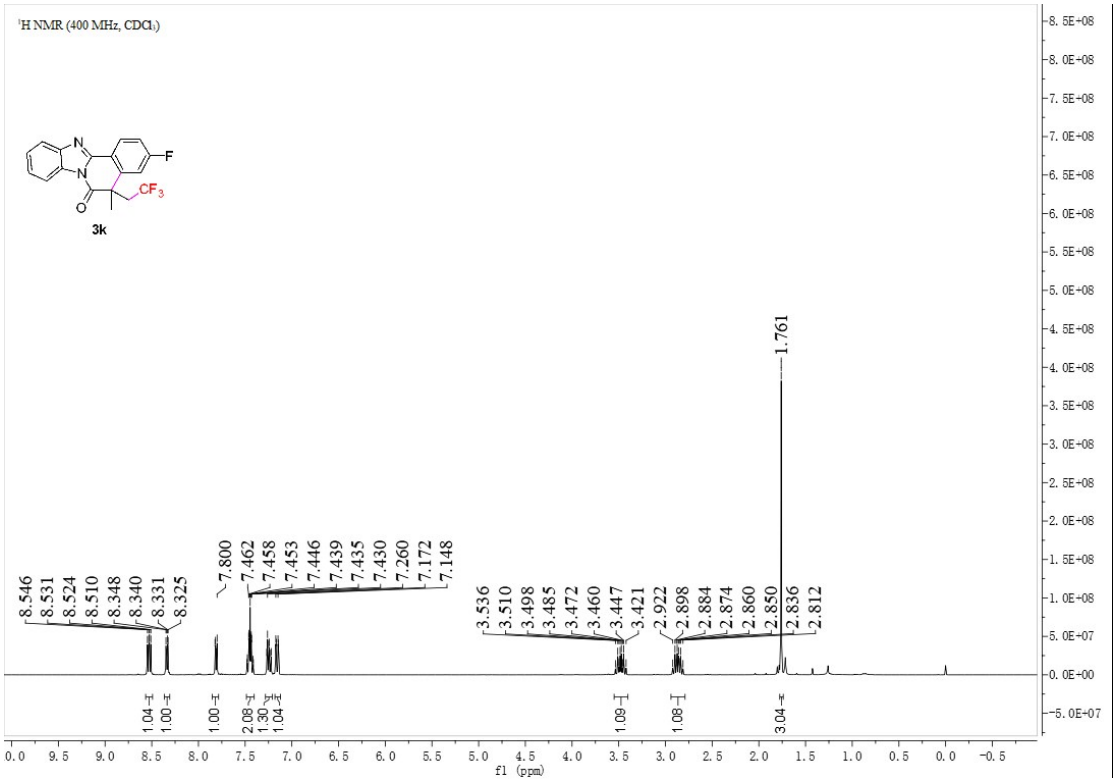

^{13}C NMR (101 MHz, CDCl$_3$)
3i
128.961
126.193
123.422
120.653
45.387
45.368
45.347
45.326
44.500
44.220
43.943
43.664
77.478
77.160
76.842
169.764
147.300
144.129
139.438
131.710
131.525
130.810
127.199
127.046
126.800
126.570
126.193
123.422
120.683
120.653
117.886
115.986
115.140
30.752
f1 (ppm)


^{19}F NMR (565 MHz, CDCl$_3$)
CN
CF$_3$
3i
-61.334
f1 (ppm)


^{1}H NMR (600 MHz, CDCl$_3$)
CF$_3$
CF$_3$
3j
8.659
8.646
8.376
8.373
8.368
8.365
8.361
7.871
7.864
7.856
7.793
7.780
7.710
7.501
7.500
7.493
7.486
7.260
3.567
3.550
3.541
3.533
3.524
3.516
3.507
3.490
3.005
2.989
2.980
2.973
2.964
2.958
2.948
2.932
1.803
1.00
1.02
1.02
1.03
1.06
2.08
1.09
1.09
3.08
f1 (ppm)


13C NMR (151 MHz, CDCl3)
3j
77.372
77.160
76.949
147.825
144.086
139.126
133.720
133.500
133.282
131.516
127.645
127.150
126.711
126.652
126.324
125.832
125.799
125.492
125.470
125.445
125.421
124.517
123.952
123.763
123.740
122.710
122.105
120.517
115.959
170.244
45.540
45.528
44.294
44.107
43.923
43.738
30.897
f1 (ppm)


19F NMR (565 MHz, CDCl3)
3j
-61.401
-62.992
f1 (ppm)


^{1}H NMR (400 MHz, CDCl$_3$)
3k
8.546
8.531
8.524
8.510
8.348
8.340
8.331
8.325
7.800
7.462
7.458
7.453
7.446
7.439
7.435
7.430
7.260
7.172
7.148
3.536
3.510
3.498
3.485
3.472
3.460
3.447
3.421
2.922
2.898
2.884
2.874
2.860
2.850
2.836
2.812
1.761
1.04
1.00
1.00
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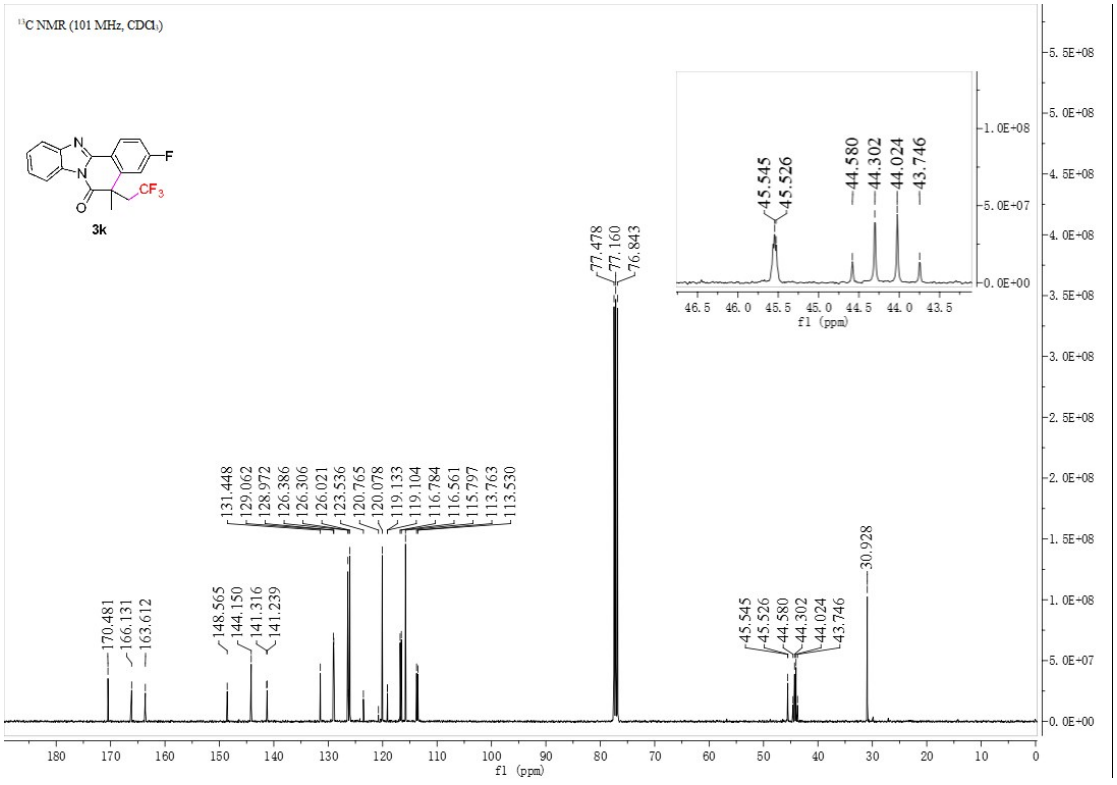
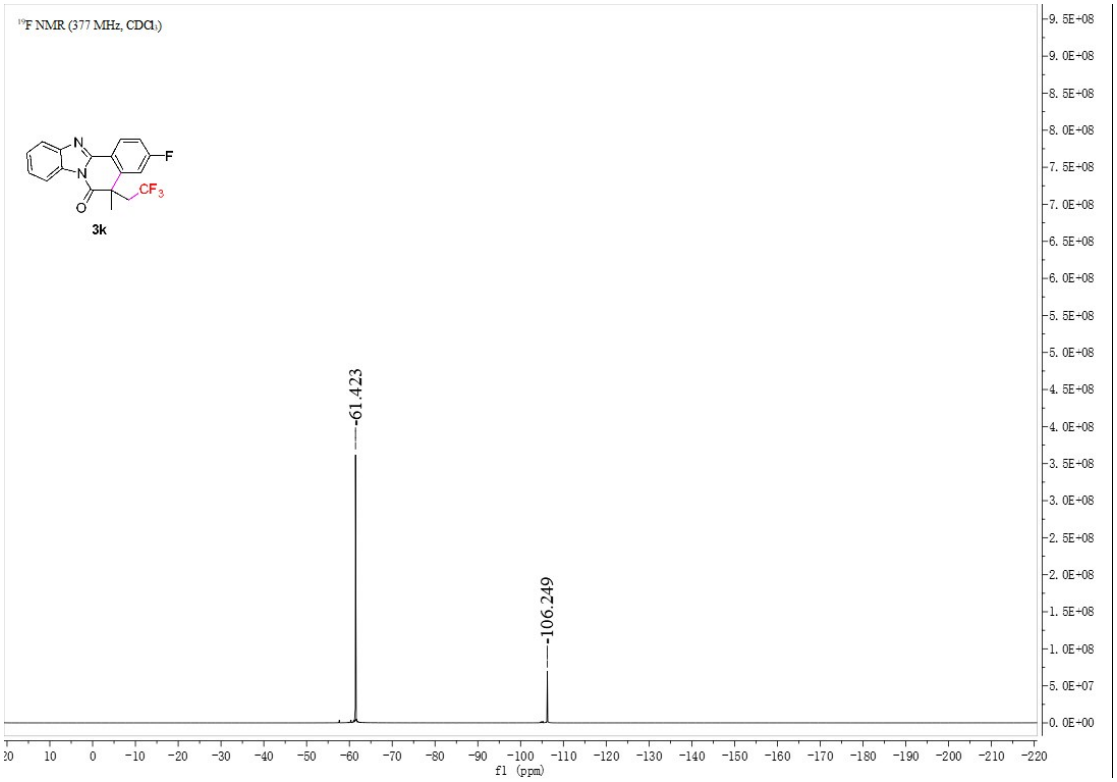
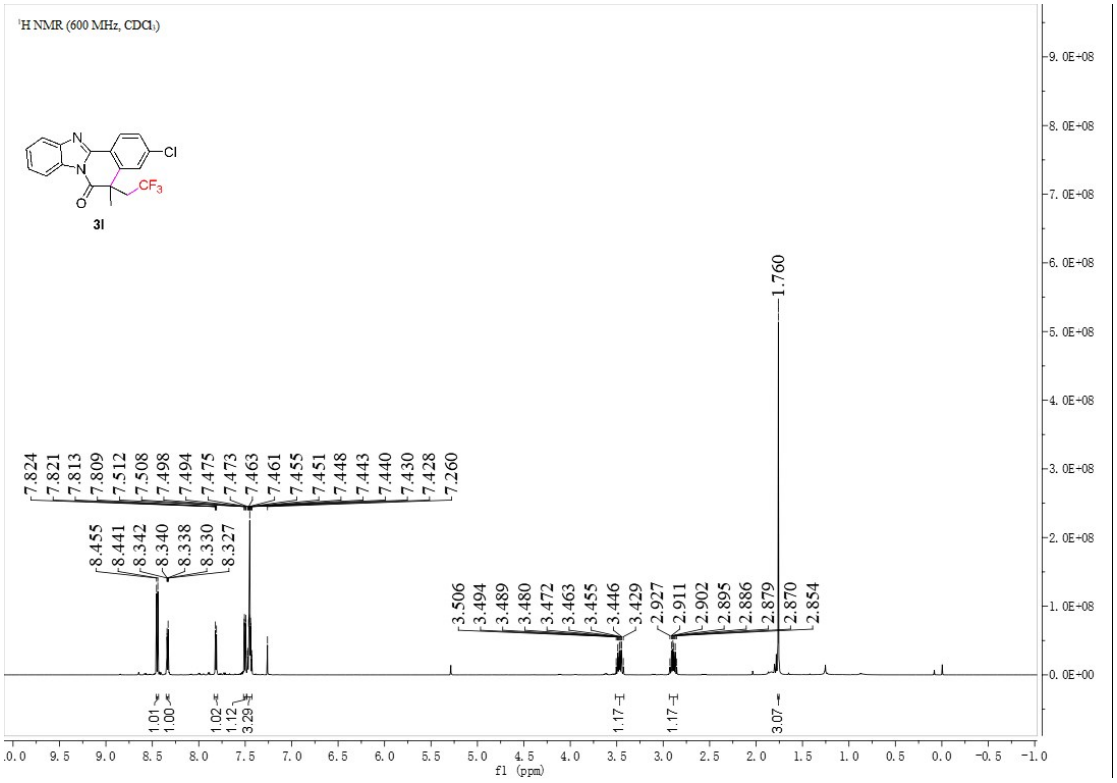
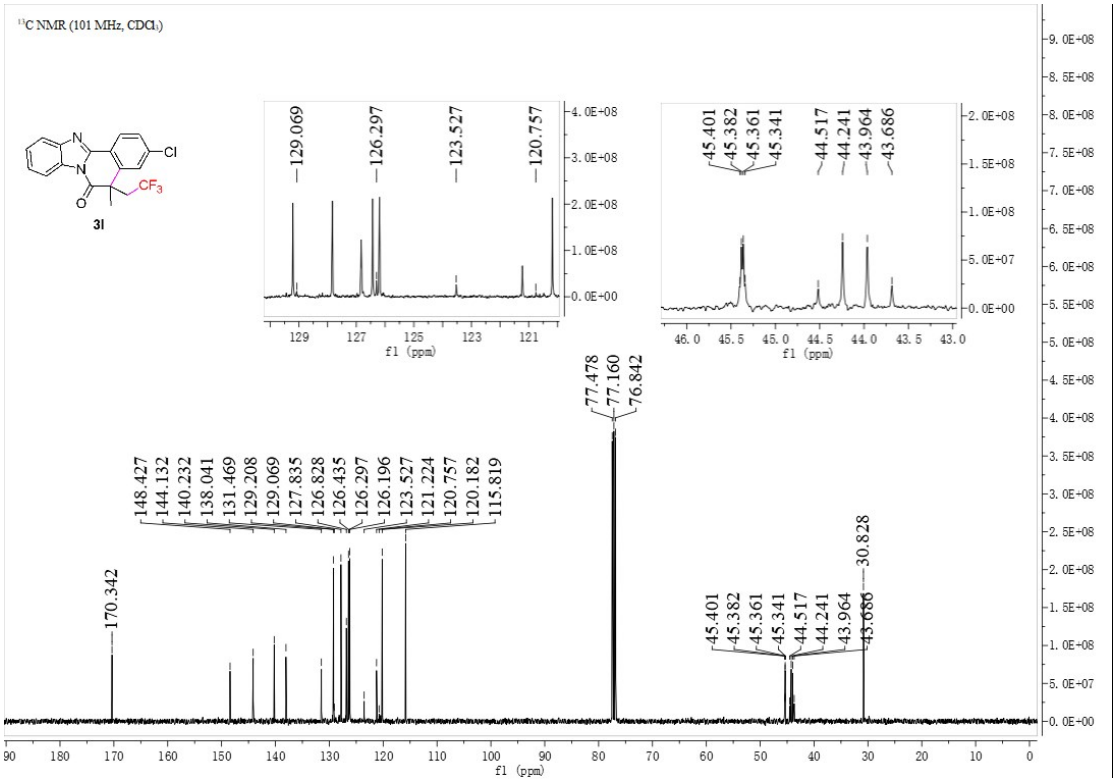
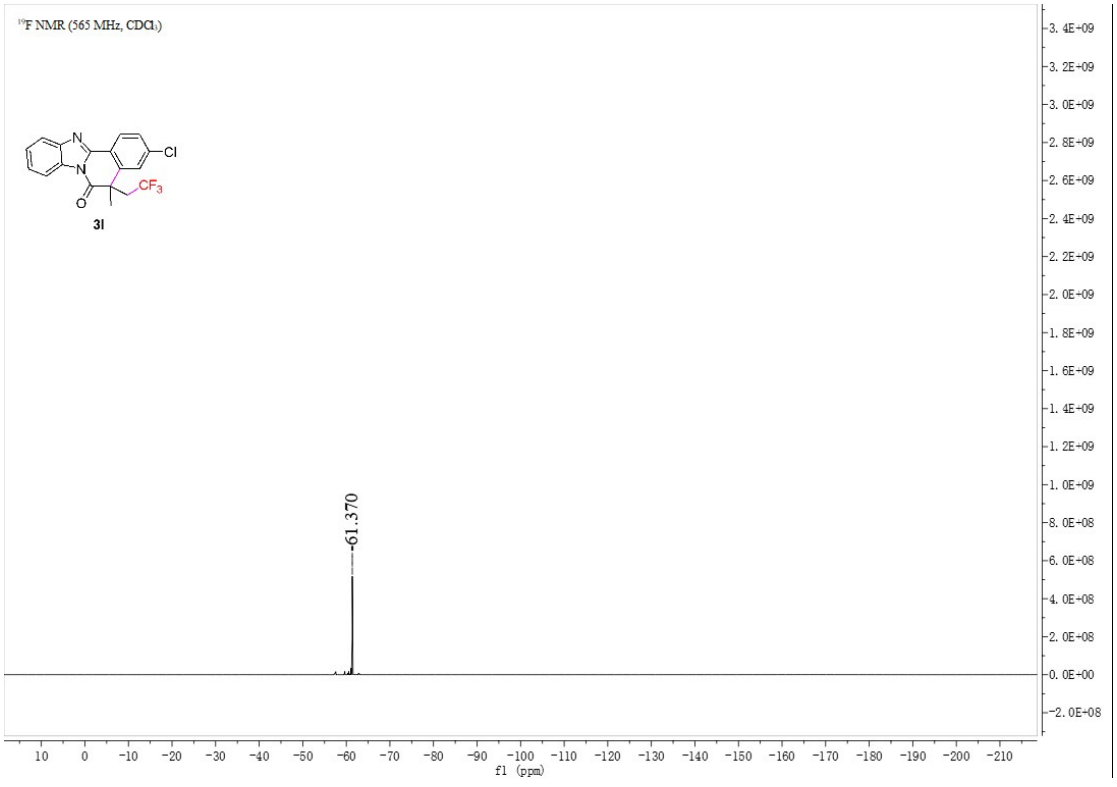
1.04
1.09
1.08
3.04
f1 (ppm)

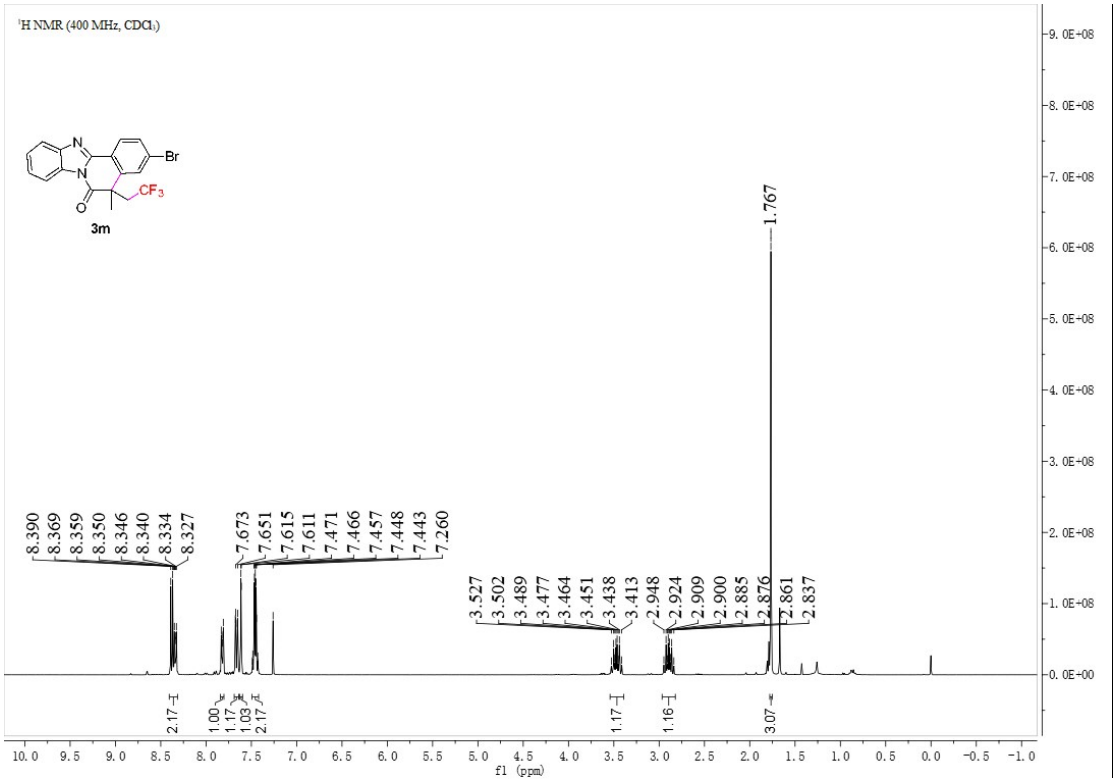

^{13}C NMR (101 MHz, CDCl$_3$)
3k
77.478
77.160
76.843
45.545
45.526
44.580
44.302
44.024
43.746
131.448
129.062
128.972
126.386
126.306
126.021
123.536
120.765
120.078
119.133
119.104
116.784
116.561
115.797
113.763
113.530
170.481
166.131
163.612
148.565
144.150
141.316
141.239
30.928
f1 (ppm)

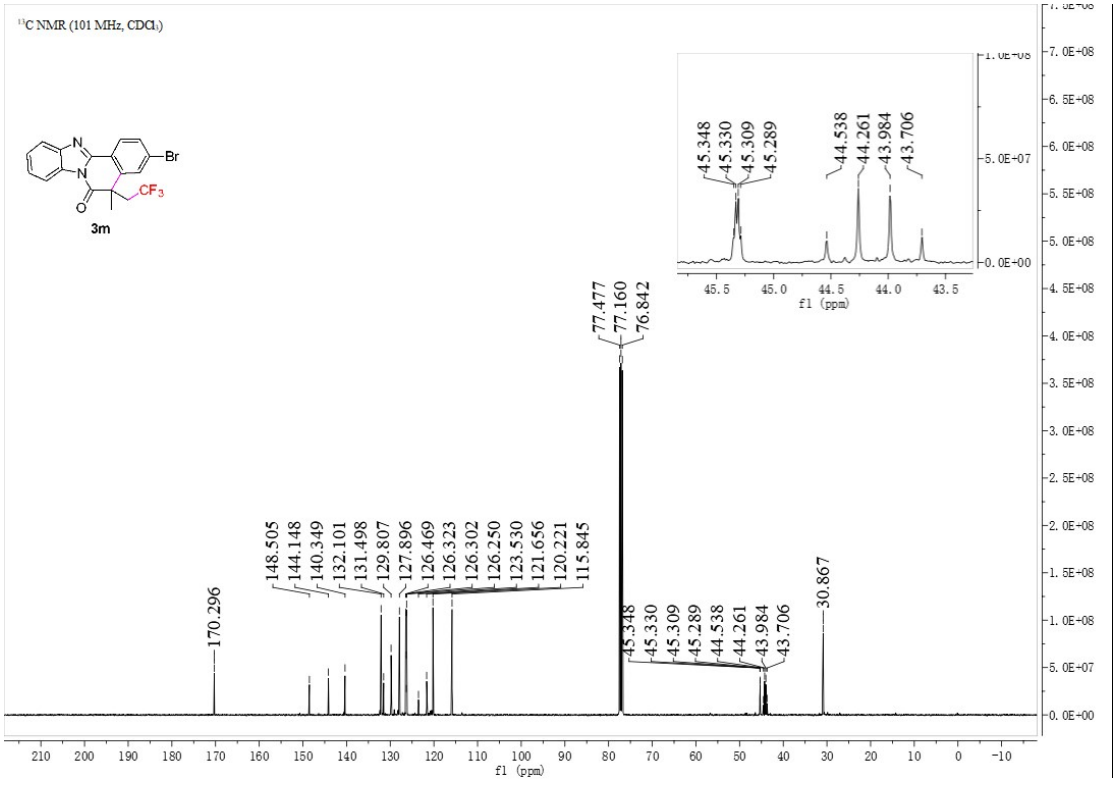

^{19}F NMR (377 MHz, CDCl$_3$)
F
CF$_3$
O
3k
-61.423
-106.249
f1 (ppm)

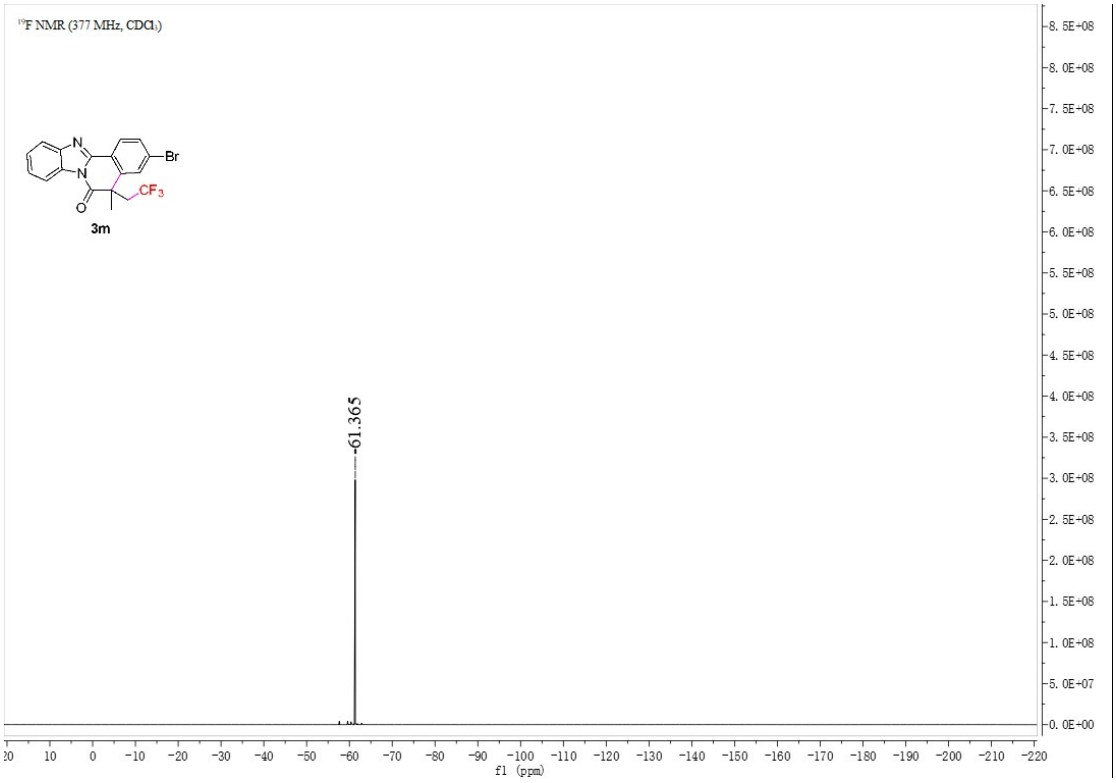

^{1}H NMR (600 MHz, CDCl$_3$)
Cl
CF$_3$
O
3l
7.824
7.821
7.813
7.809
7.512
7.508
7.498
7.494
7.475
7.473
7.463
7.461
7.455
7.451
7.448
7.443
7.440
7.430
7.428
7.260
8.455
8.441
8.342
8.340
8.338
8.330
8.327
3.506
3.494
3.489
3.480
3.472
3.463
3.455
3.446
3.429
2.927
2.911
2.902
2.895
2.886
2.879
2.870
2.854
1.760
1.01
1.00
1.02
1.12
3.29
1.17
1.17
3.07
f1 (ppm)

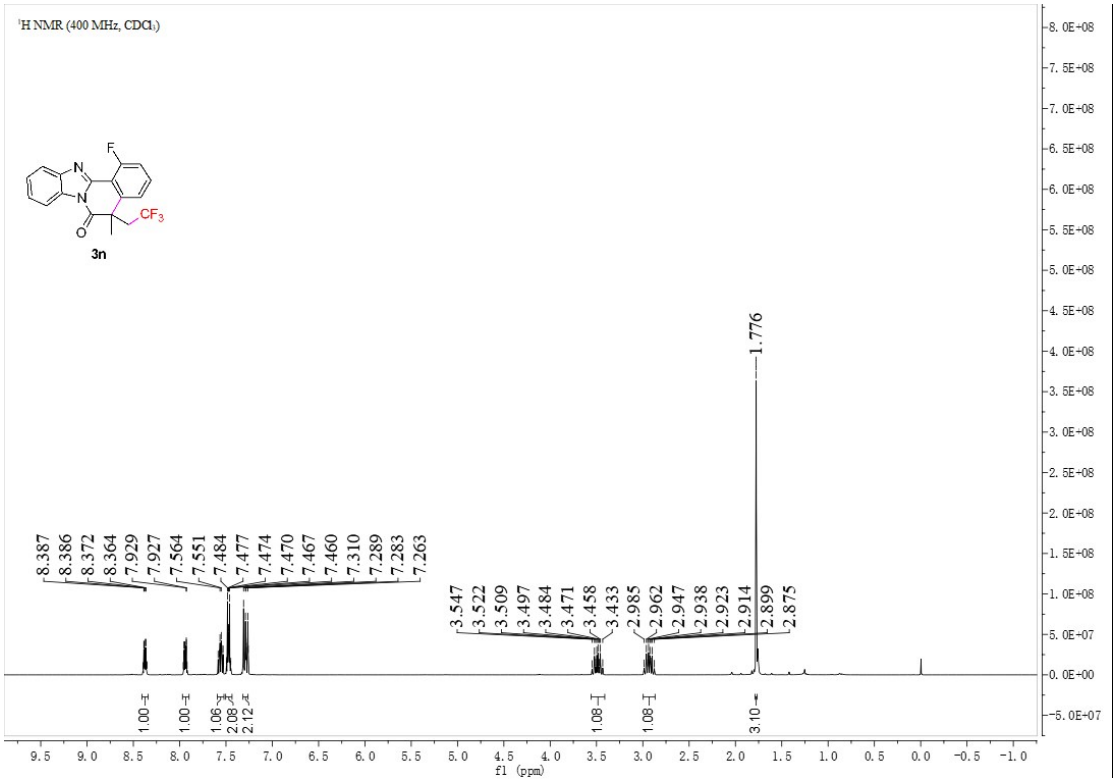

^{13}C NMR (101 MHz, CDCl$_3$)
3l
129.069
126.297
123.527
120.757
45.401
45.382
45.361
45.341
44.517
44.241
43.964
43.686
77.478
77.160
76.842
148.427
144.132
140.232
138.041
131.469
129.208
129.069
127.835
126.828
126.435
126.297
126.196
123.527
121.224
120.757
120.182
115.819
170.342
30.828
f1 (ppm)

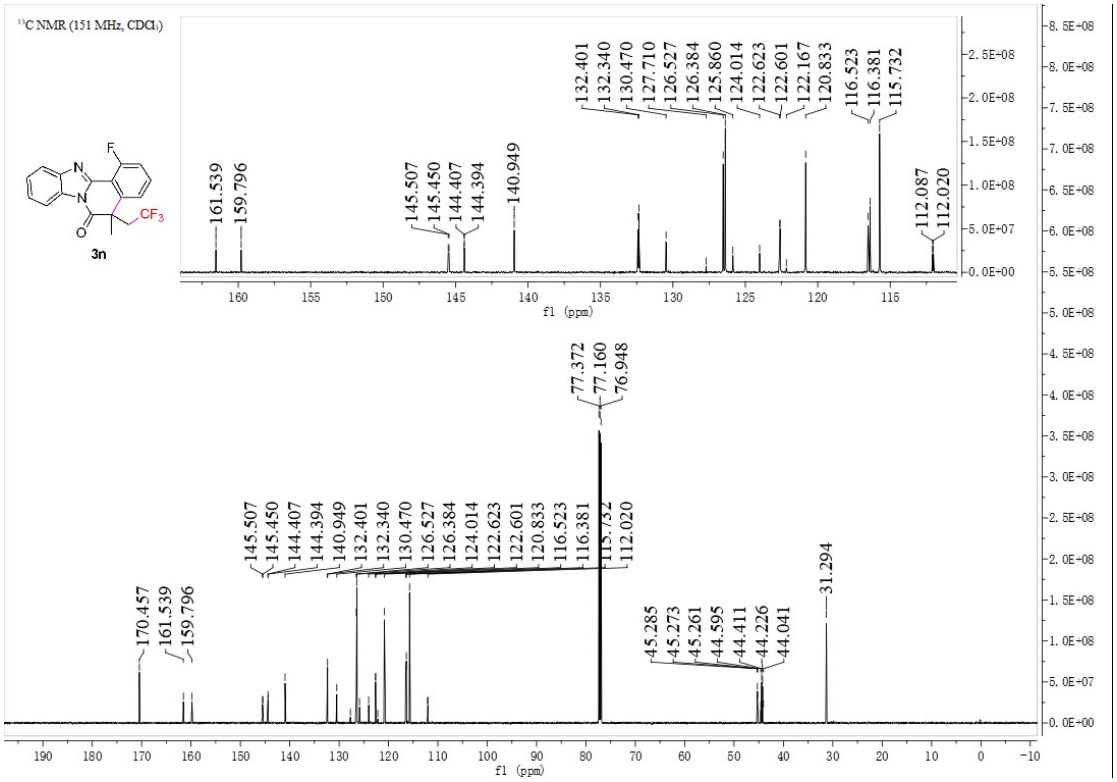

^{19}F NMR (565 MHz, CDCl$_3$)
3l
-61.370
f1 (ppm)

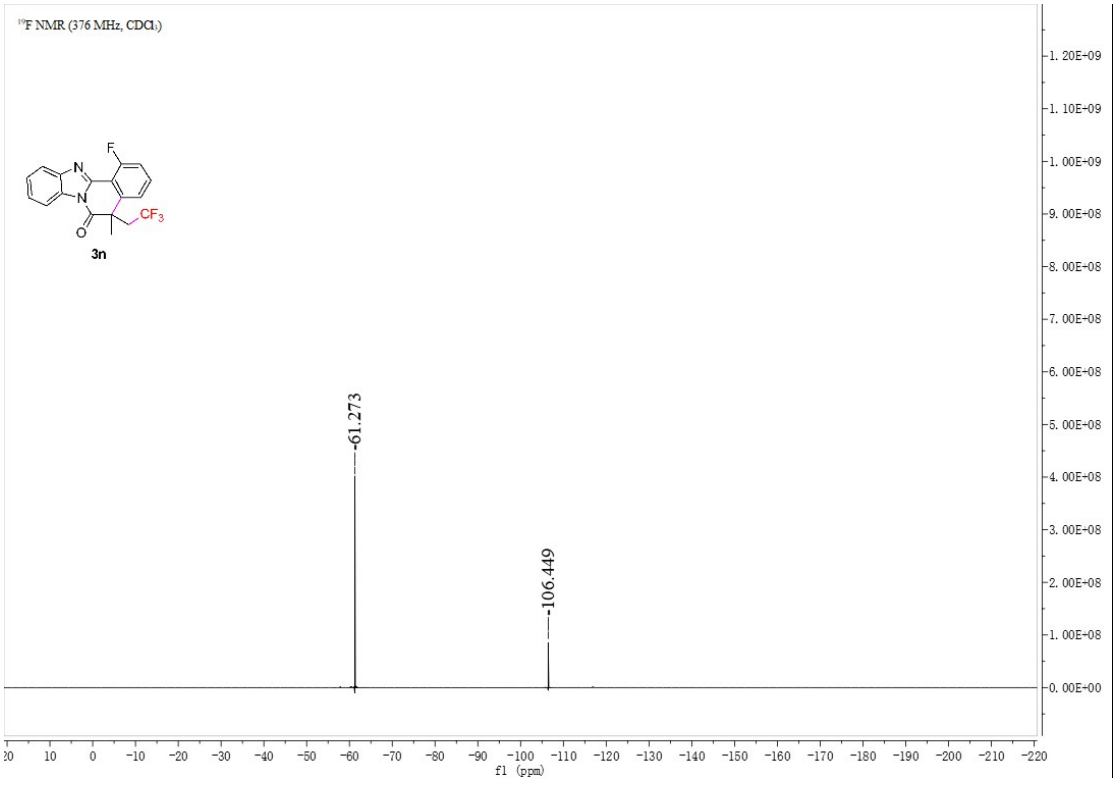

^{1}H NMR (400 MHz, CDCl$_3$)
Br
CF$_3$
O
3m
8.390
8.369
8.359
8.350
8.346
8.340
8.334
8.327
7.673
7.651
7.615
7.611
7.471
7.466
7.457
7.448
7.443
7.260
3.527
3.502
3.489
3.477
3.464
3.451
3.438
3.413
2.948
2.924
2.909
2.900
2.885
2.876
2.861
2.837
1.767
2.17
1.00
1.17
1.03
2.17
1.17
1.16
3.07
f1 (ppm)

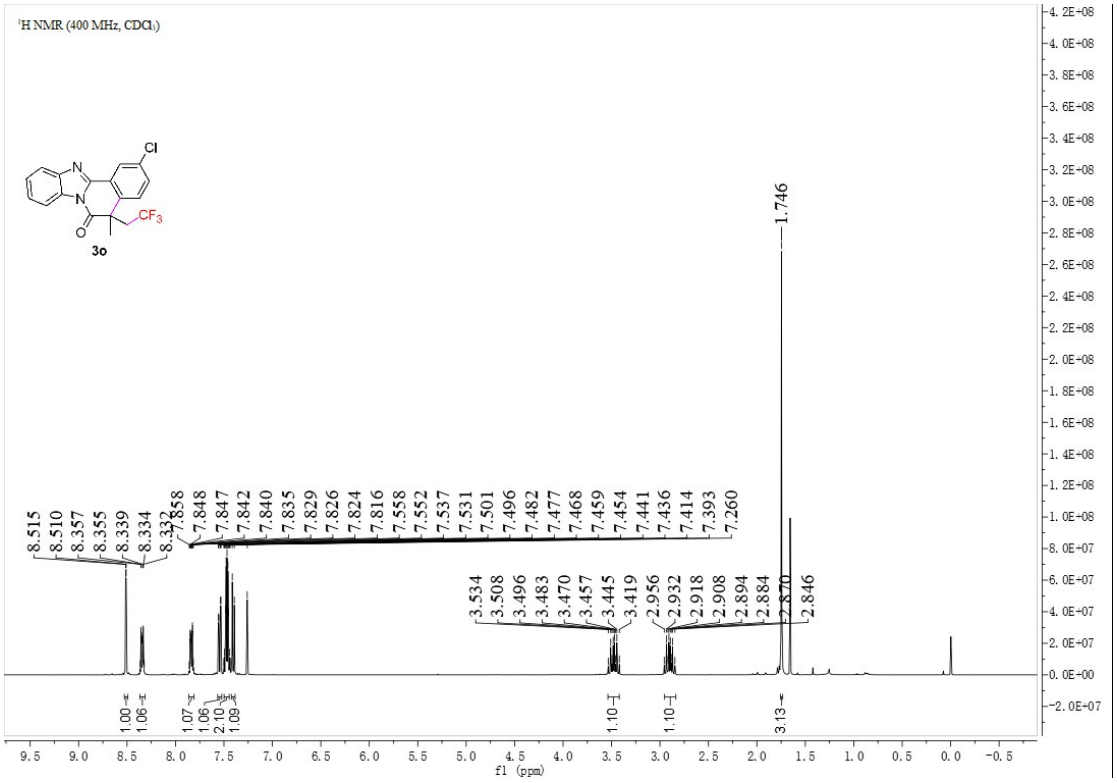

^{13}C NMR (101 MHz, CDCl$_3$)
Br
CF$_3$
O
3m
45.348
45.330
45.309
45.289
44.538
44.261
43.984
43.706
f1 (ppm)
77.477
77.160
76.842
148.505
144.148
140.349
132.101
131.498
129.807
127.896
126.469
126.323
126.302
126.250
123.530
121.656
120.221
115.845
170.296
30.867
f1 (ppm)

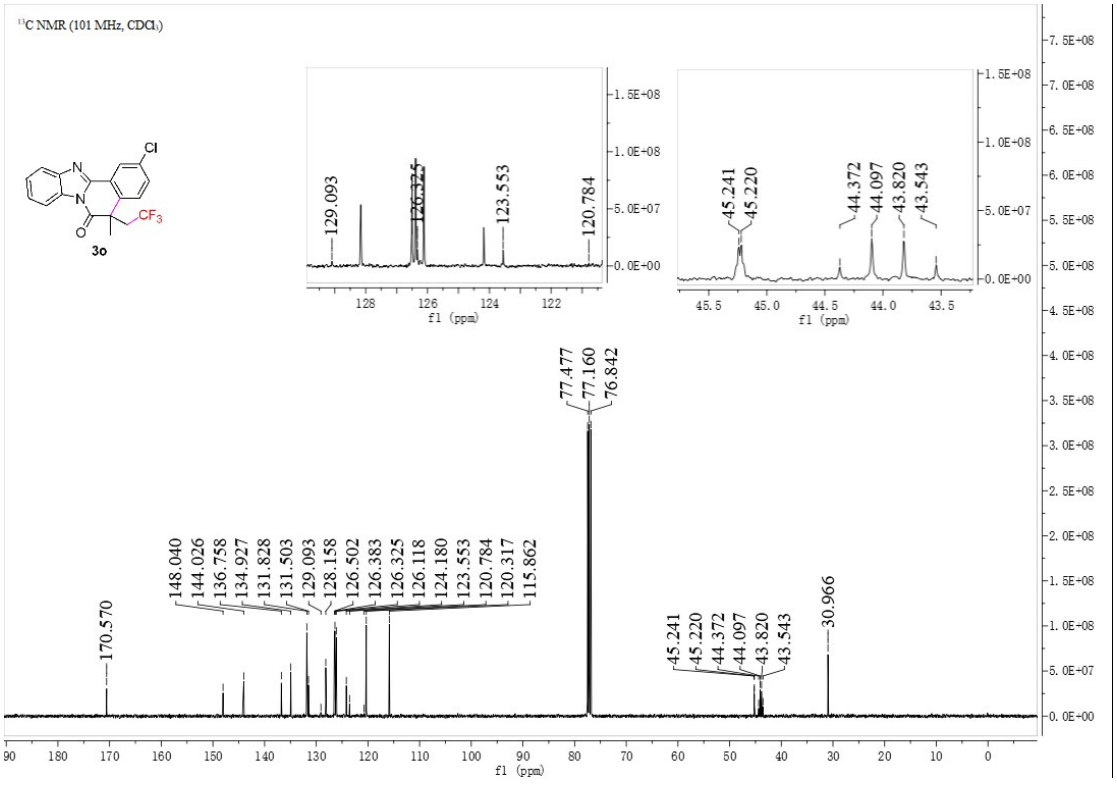

^{19}F NMR (377 MHz, CDCl$_3$)
Br
CF$_3$
O
3m
-61.365
f1 (ppm)

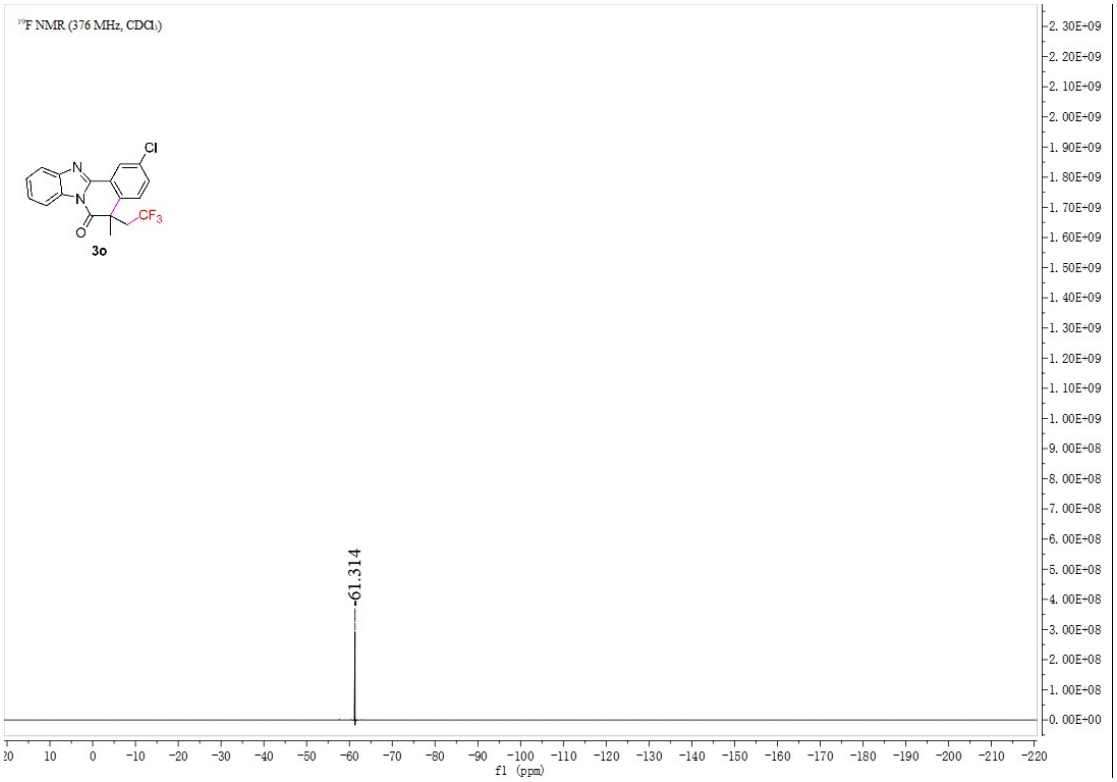

^{1}H NMR (400 MHz, CDCl$_3$)
F
CF$_3$
O
3n
8.387
8.386
8.372
8.364
7.929
7.927
7.564
7.551
7.484
7.477
7.474
7.470
7.467
7.460
7.310
7.289
7.283
7.263
3.547
3.522
3.509
3.497
3.484
3.471
3.458
3.433
2.985
2.962
2.947
2.938
2.923
2.914
2.899
2.875
1.776
1.00
1.00
1.06
2.08
2.12
1.08
1.08
3.10
f1 (ppm)

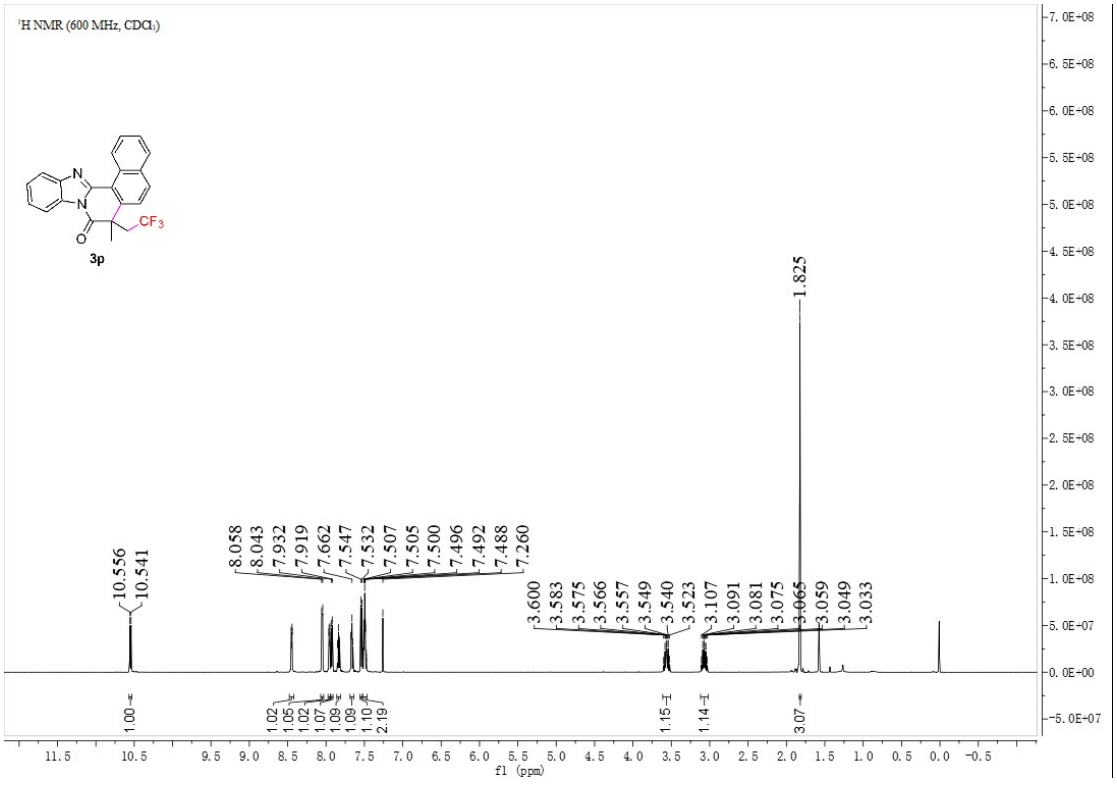

^{13}C NMR (151 MHz, CDCl$_3$)
3n
161.539
159.796
145.507
145.450
144.407
144.394
140.949
132.401
132.340
130.470
127.710
126.527
126.384
125.860
124.014
122.623
122.601
122.167
120.833
116.523
116.381
115.732
112.087
112.020
f1 (ppm)
77.372
77.160
76.948
170.457
31.294
45.285
45.273
45.261
44.595
44.411
44.226
44.041


^{19}F NMR (376 MHz, CDCl$_3$)
3n
-61.273
-106.449
f1 (ppm)


^{1}H NMR (400 MHz, CDCl$_3$)
Cl
N
N
O
CF$_3$
3o
8.515
8.510
8.357
8.355
8.339
8.334
8.332
7.858
7.848
7.847
7.842
7.840
7.835
7.829
7.826
7.824
7.816
7.558
7.552
7.537
7.531
7.501
7.496
7.482
7.477
7.468
7.459
7.454
7.441
7.436
7.414
7.393
7.260
3.534
3.508
3.496
3.483
3.470
3.457
3.445
3.419
2.956
2.932
2.918
2.908
2.894
2.884
2.870
2.846
1.746
1.00
1.06
1.07
1.06
2.10
1.09
1.10
1.10
3.13
f1 (ppm)


^{13}C NMR (101 MHz, CDCl$_3$)
Cl
N
N
O
CF$_3$
3o
129.093
126.325
123.553
120.784
45.241
45.220
44.372
44.097
43.820
43.543
f1 (ppm)
77.477
77.160
76.842
148.040
144.026
136.758
134.927
131.828
131.503
129.093
128.158
126.502
126.383
126.325
126.118
124.180
123.553
120.784
120.317
115.862
170.570
45.241
45.220
44.372
44.097
43.820
43.543
30.966
f1 (ppm)


19F NMR (376 MHz, CDCl3)
Cl
N
N
O
CF3
3o
-61.314
f1 (ppm)


1H NMR (600 MHz, CDCl3)
N
N
O
CF3
3p
10.556
10.541
8.058
8.043
7.932
7.919
7.662
7.547
7.532
7.507
7.505
7.500
7.496
7.492
7.488
7.260
3.600
3.583
3.575
3.566
3.557
3.549
3.540
3.523
3.107
3.091
3.081
3.075
3.065
3.059
3.049
3.033
1.825
1.00
1.02
1.05
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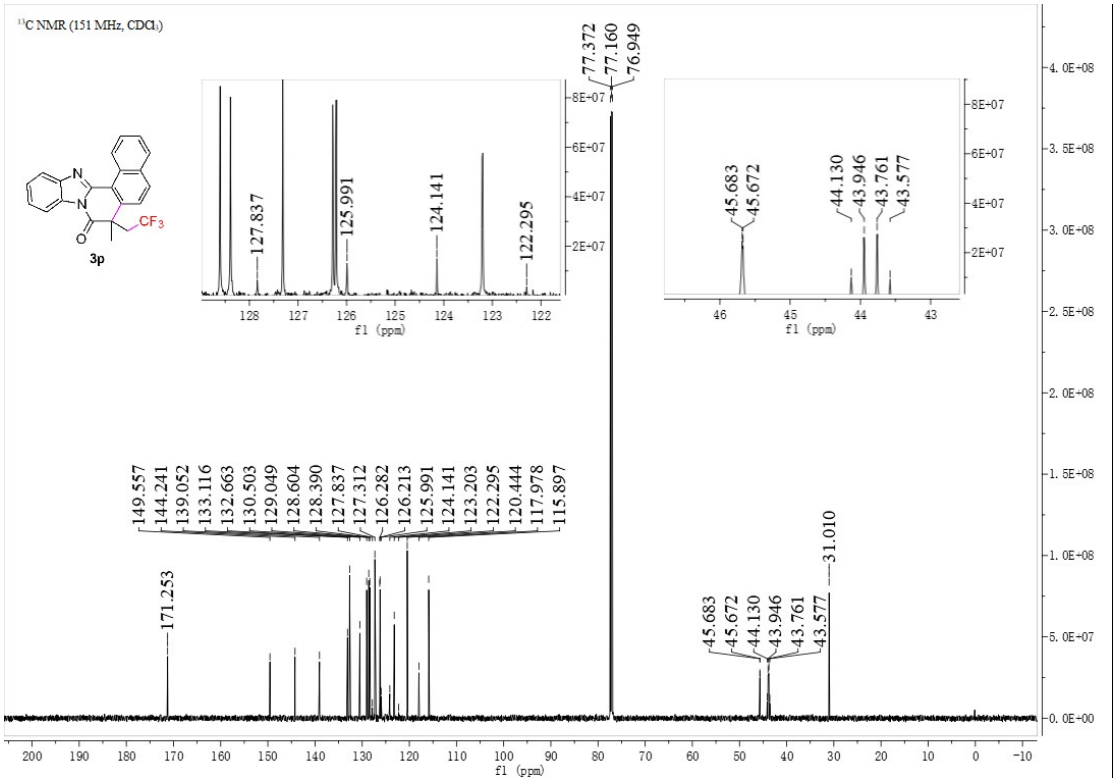
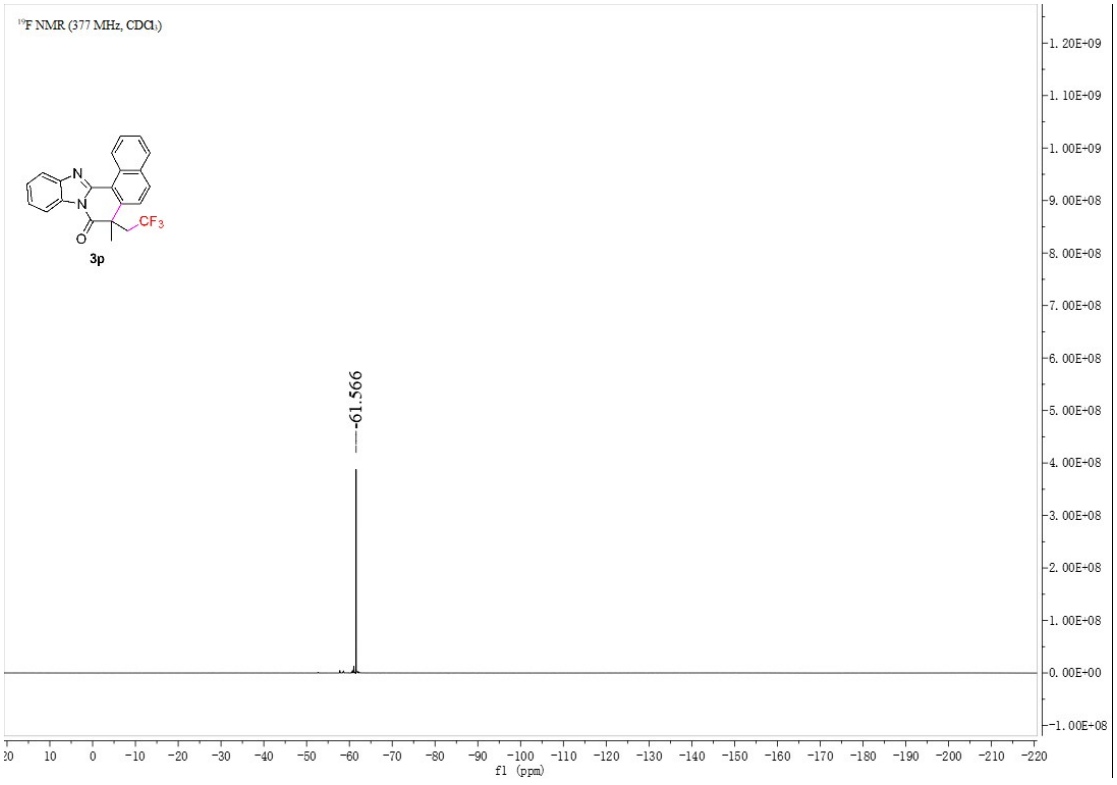
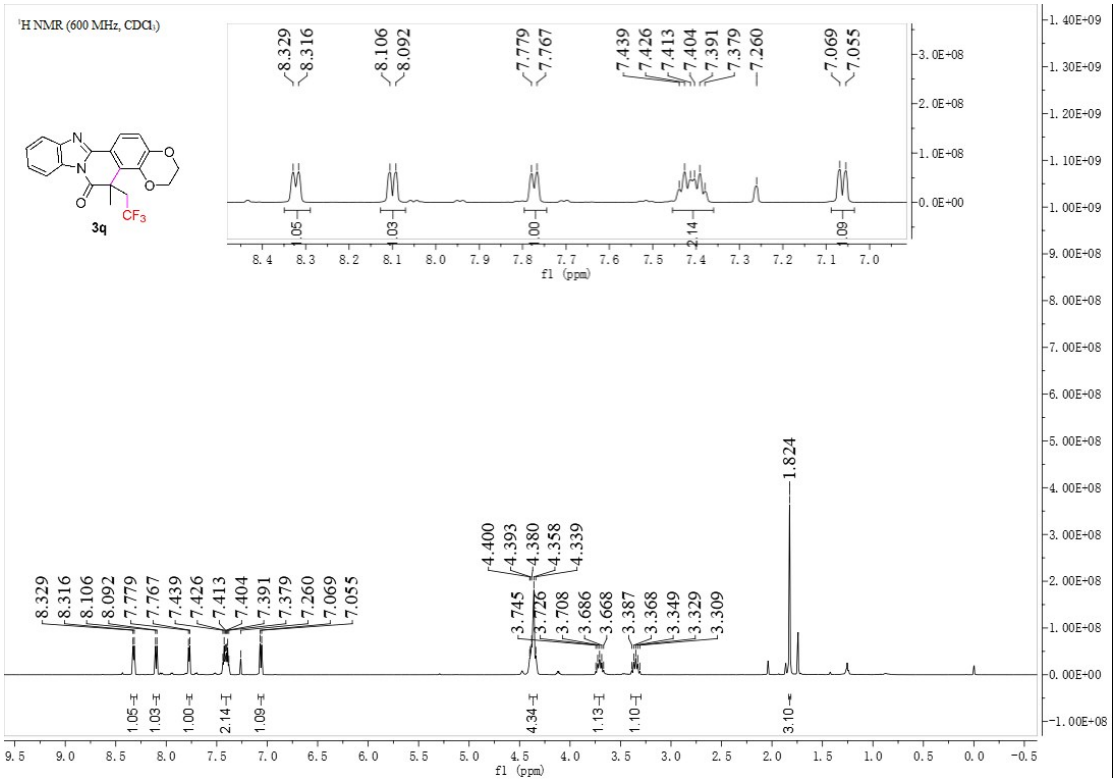
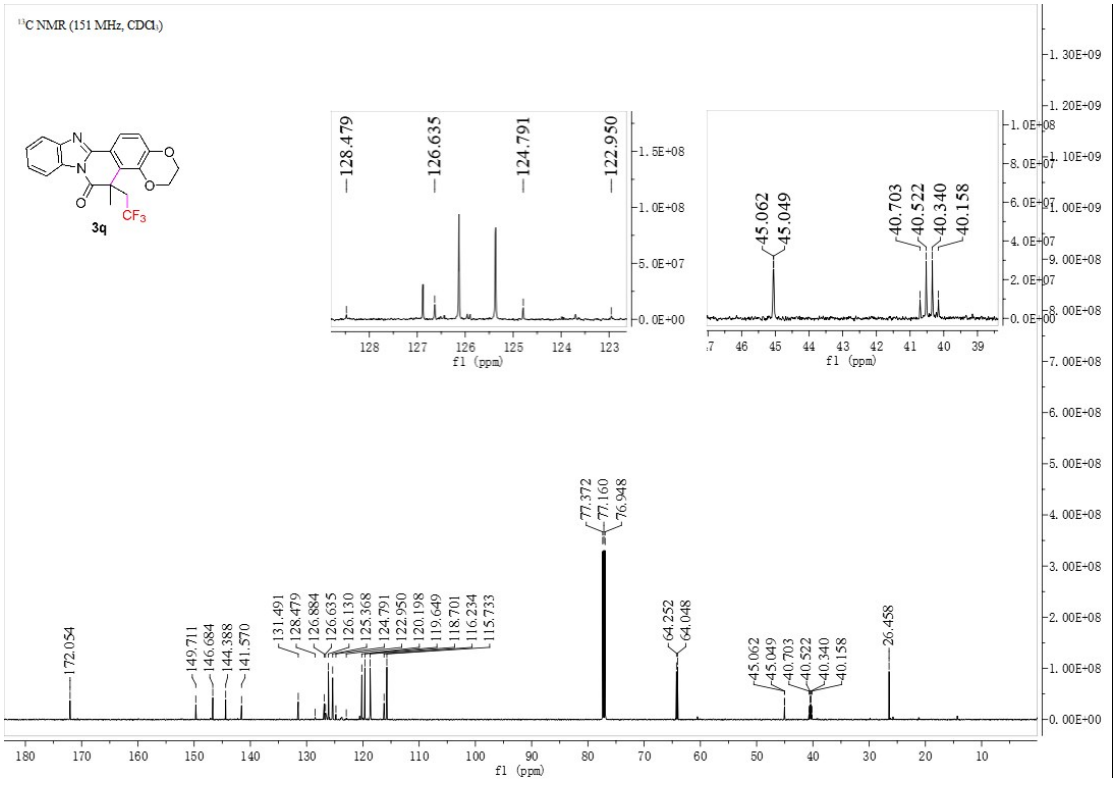
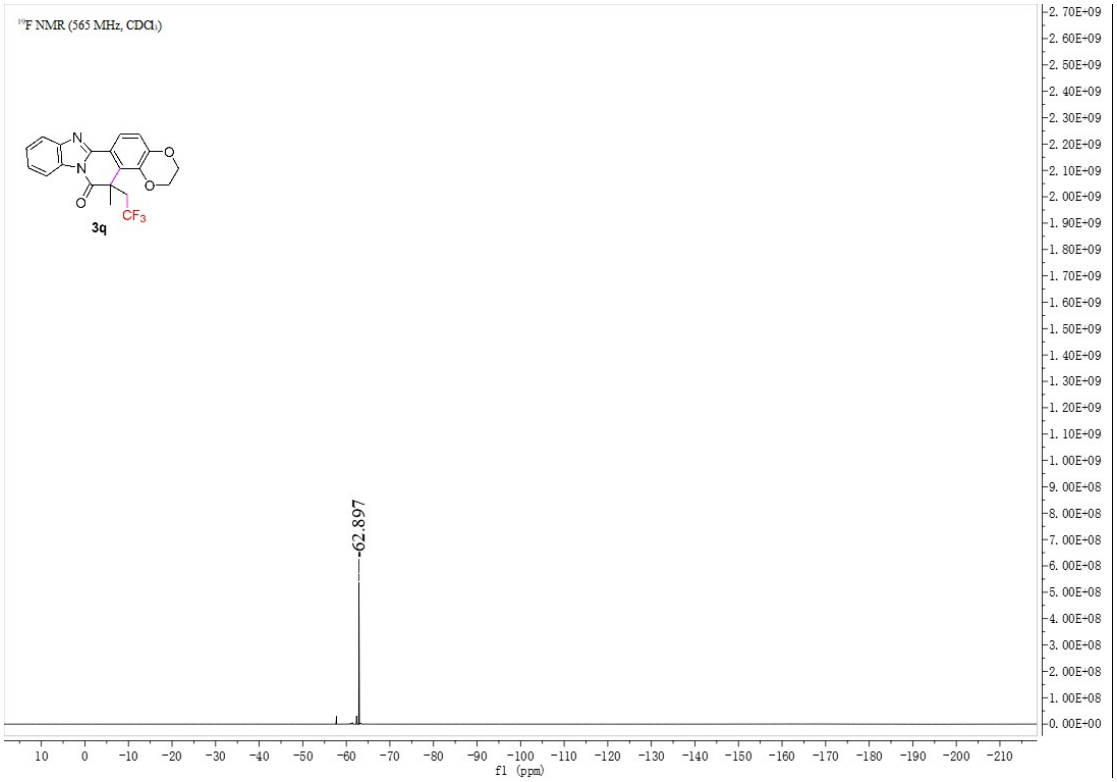
1.07
1.09
1.09
1.10
2.19
1.15
1.14
3.07
f1 (ppm)

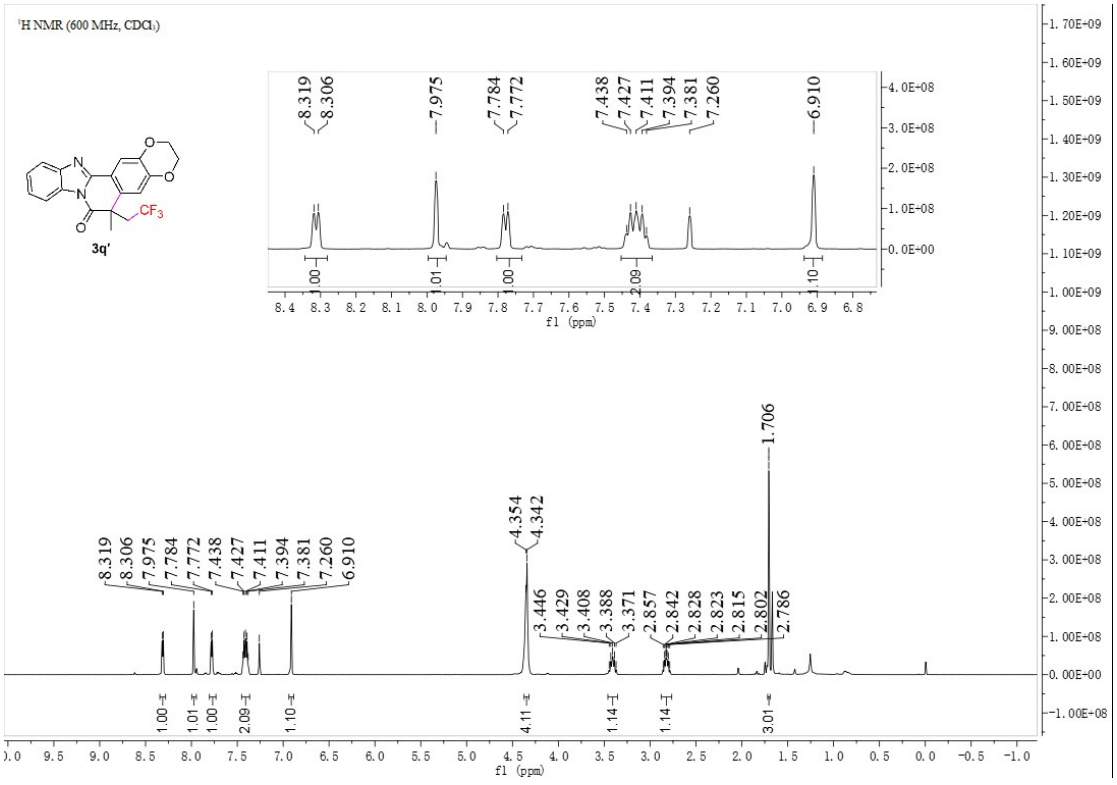

13C NMR (151 MHz, CDCl3)
3p
77.372
77.160
76.949
127.837
125.991
124.141
122.295
45.683
45.672
44.130
43.946
43.761
43.577
149.557
144.241
139.052
133.116
132.663
130.503
129.049
128.604
128.390
127.837
127.312
126.282
126.213
125.991
124.141
123.203
122.295
120.444
117.978
115.897
171.253
31.010
f1 (ppm)

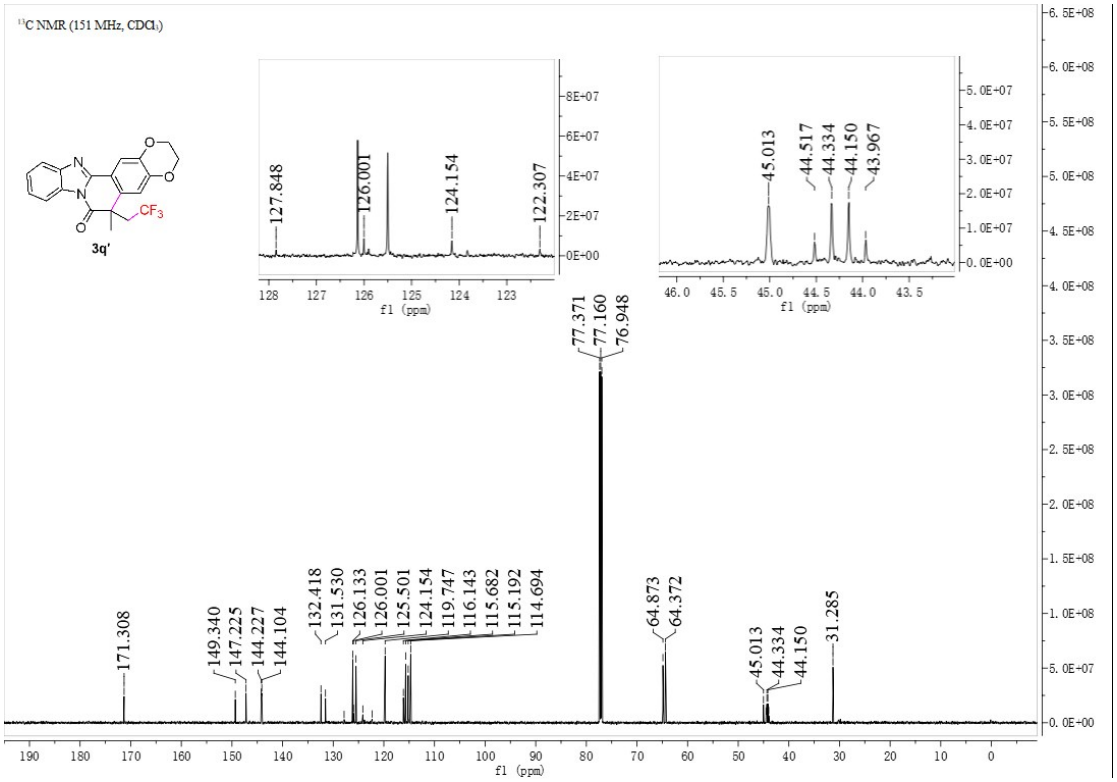

19F NMR (377 MHz, CDCl3)
3p
-61.566
f1 (ppm)

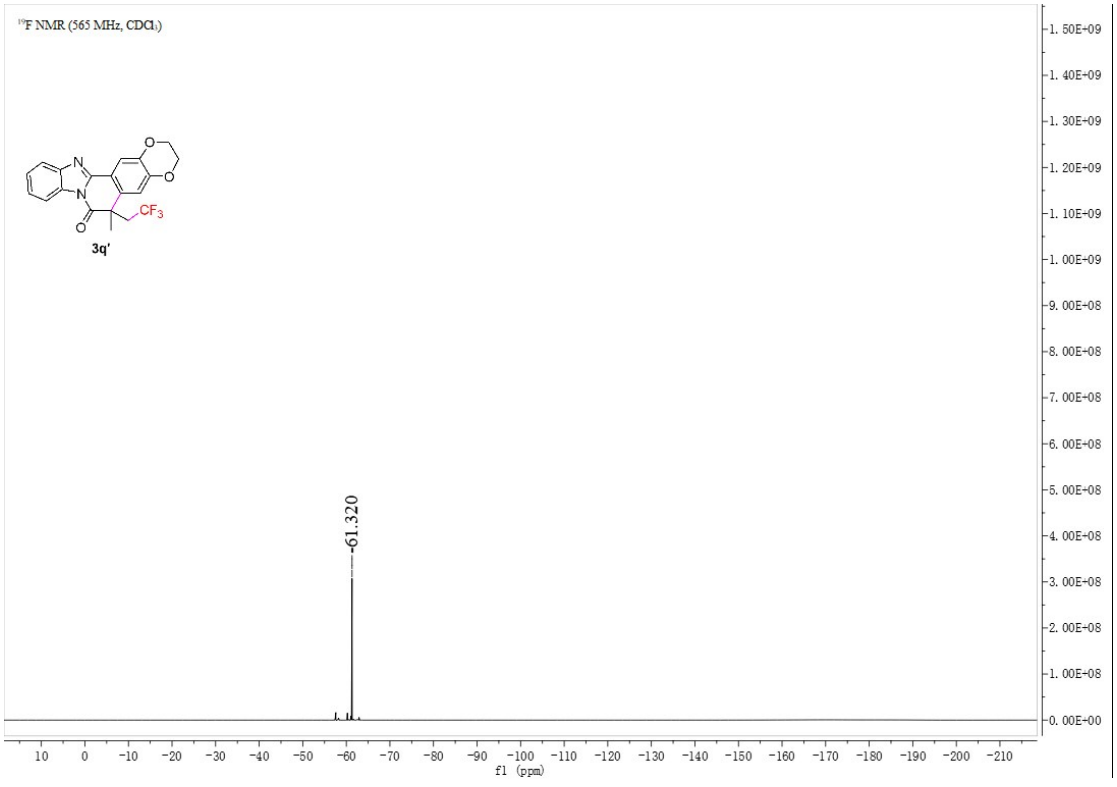

¹H NMR (600 MHz, CDCl₃)
3q
f1 (ppm)

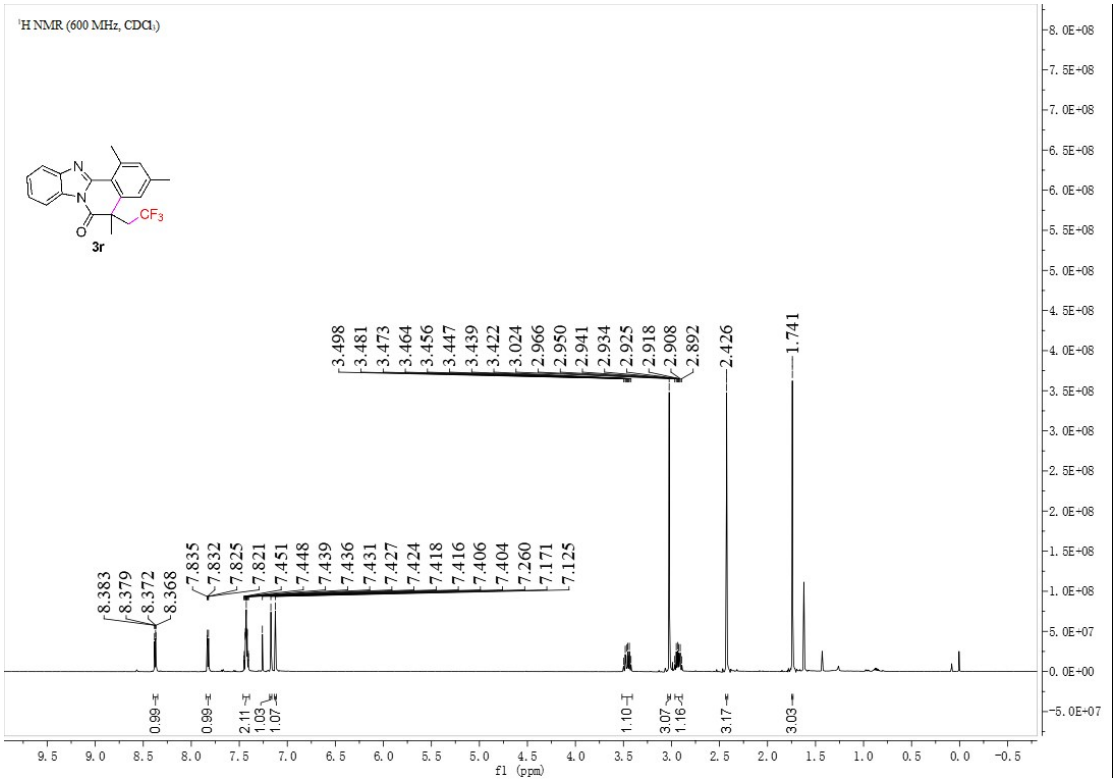

¹³C NMR (151 MHz, CDCl₃)
3q
f1 (ppm)

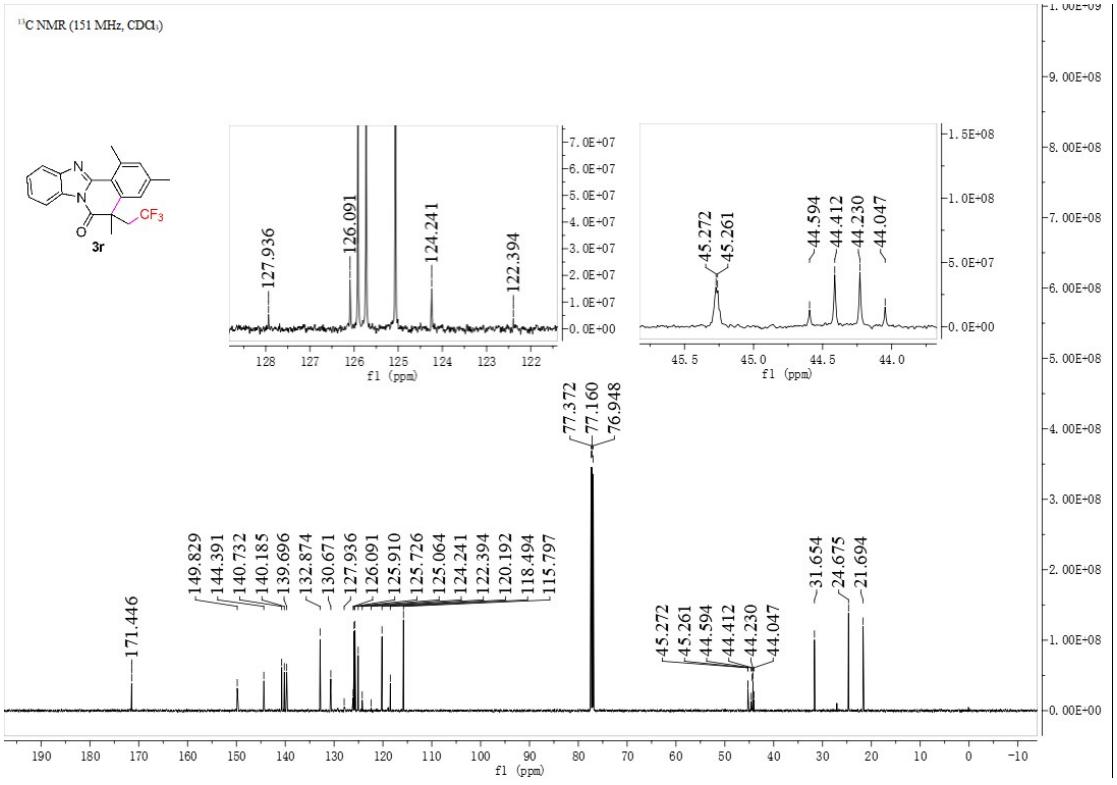

^{19}F NMR (565 MHz, CDCl$_3$)
3q
-62.897
f1 (ppm)

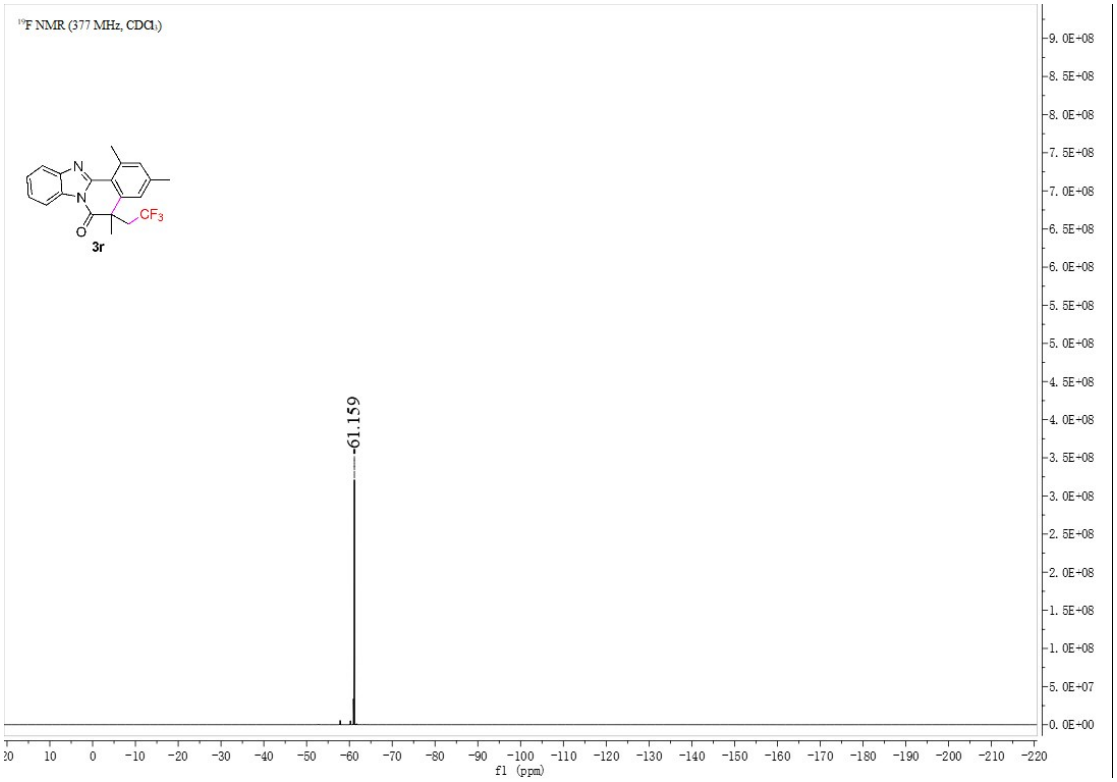

^{1}H NMR (600 MHz, CDCl$_3$)
3q'
8.319
8.306
7.975
7.784
7.772
7.438
7.427
7.411
7.394
7.381
7.260
6.910
4.354
4.342
3.446
3.429
3.408
3.388
3.371
2.857
2.842
2.828
2.823
2.815
2.802
2.786
1.706
f1 (ppm)

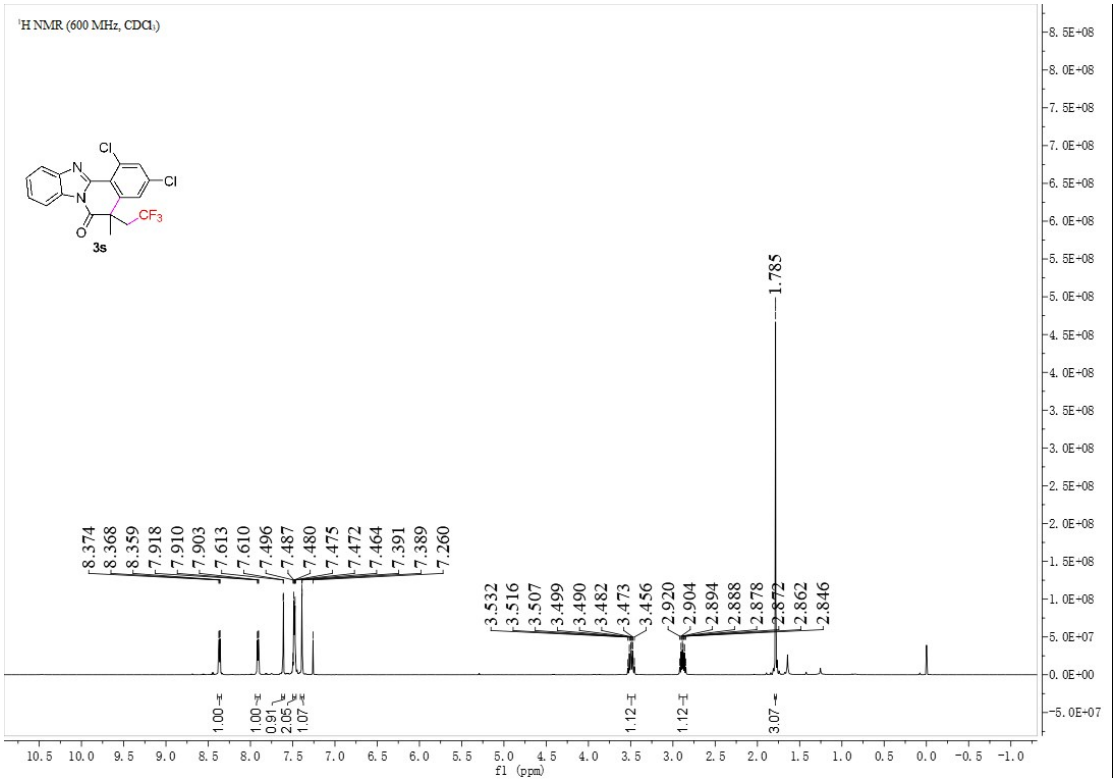

^{13}C NMR (151 MHz, CDCl$_3$)
3q'
127.848
126.001
124.154
122.307
45.013
44.517
44.334
44.150
43.967
77.371
77.160
76.948
171.308
149.340
147.225
144.227
144.104
132.418
131.530
126.133
126.001
125.501
124.154
119.747
116.143
115.682
115.192
114.694
64.873
64.372
45.013
44.334
44.150
31.285
f1 (ppm)

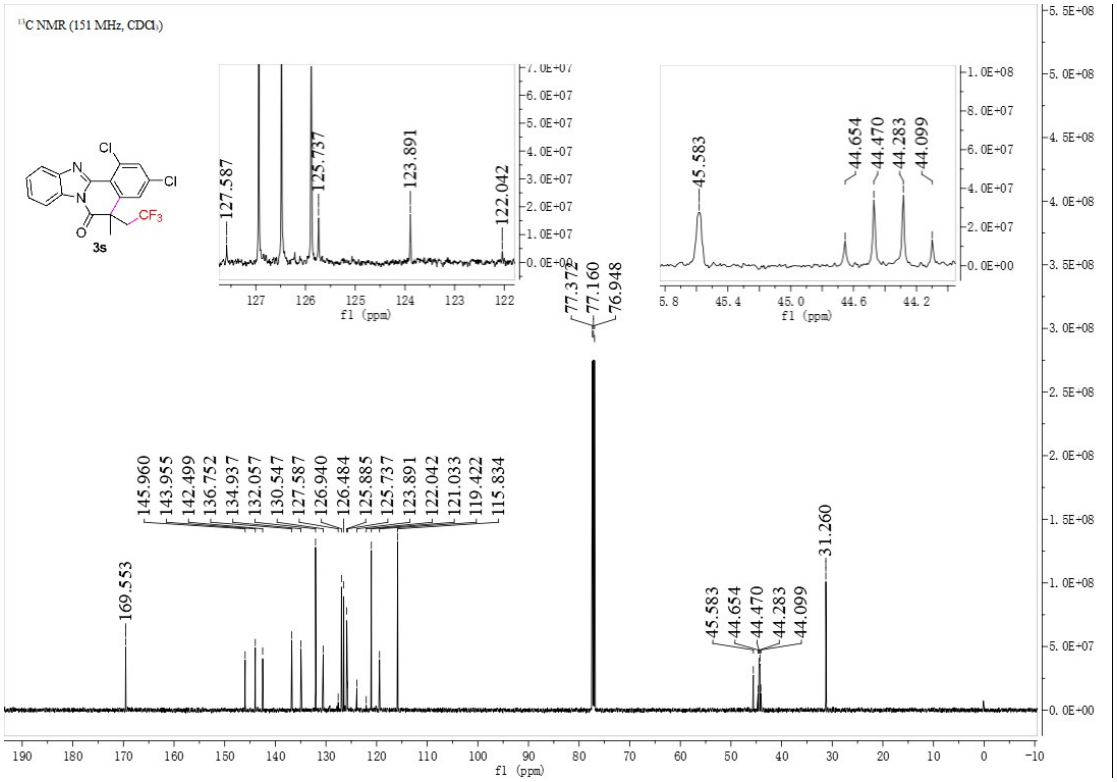

^{19}F NMR (565 MHz, CDCl$_3$)
3q'
-61.320
f1 (ppm)

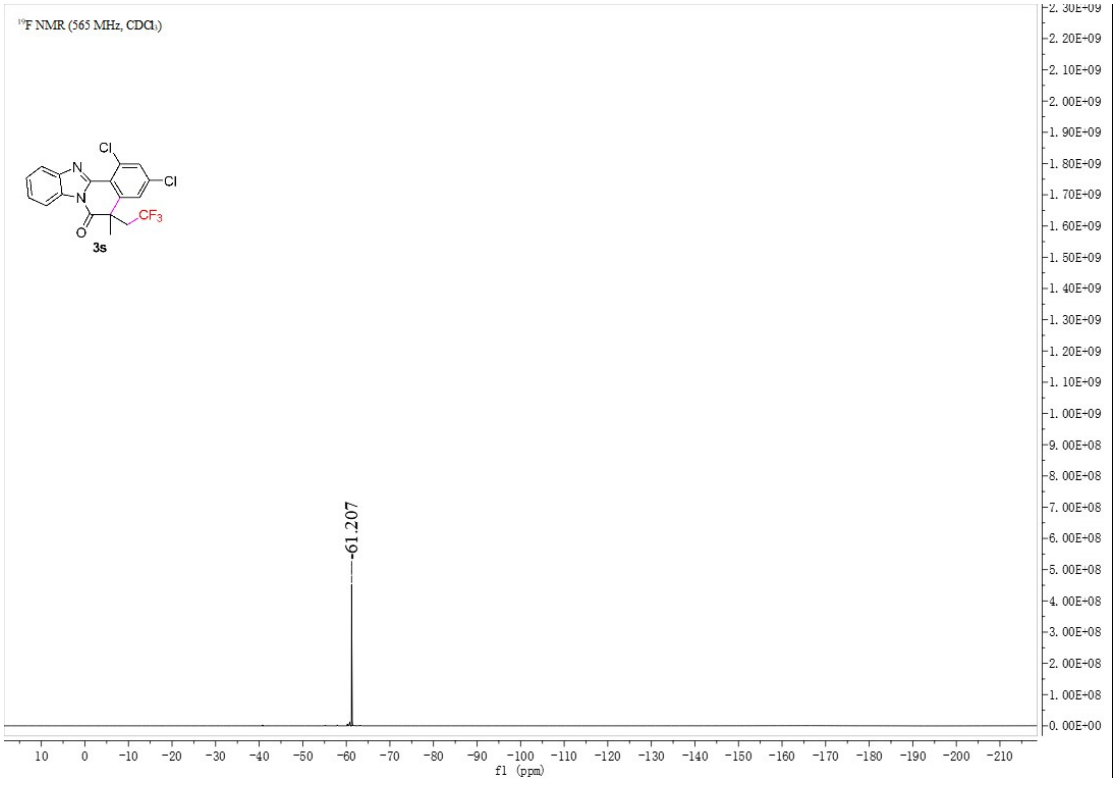

¹H NMR (600 MHz, CDCl₃)
3r
3.498
3.481
3.473
3.464
3.456
3.447
3.439
3.422
3.024
2.966
2.950
2.941
2.934
2.925
2.918
2.908
2.892
2.426
1.741
8.383
8.379
8.372
8.368
7.835
7.832
7.825
7.821
7.451
7.448
7.439
7.436
7.431
7.427
7.424
7.418
7.416
7.406
7.404
7.260
7.171
7.125
0.99
0.99
2.11
1.03
1.07
1.10
3.07
1.16
3.17
3.03
f1 (ppm)

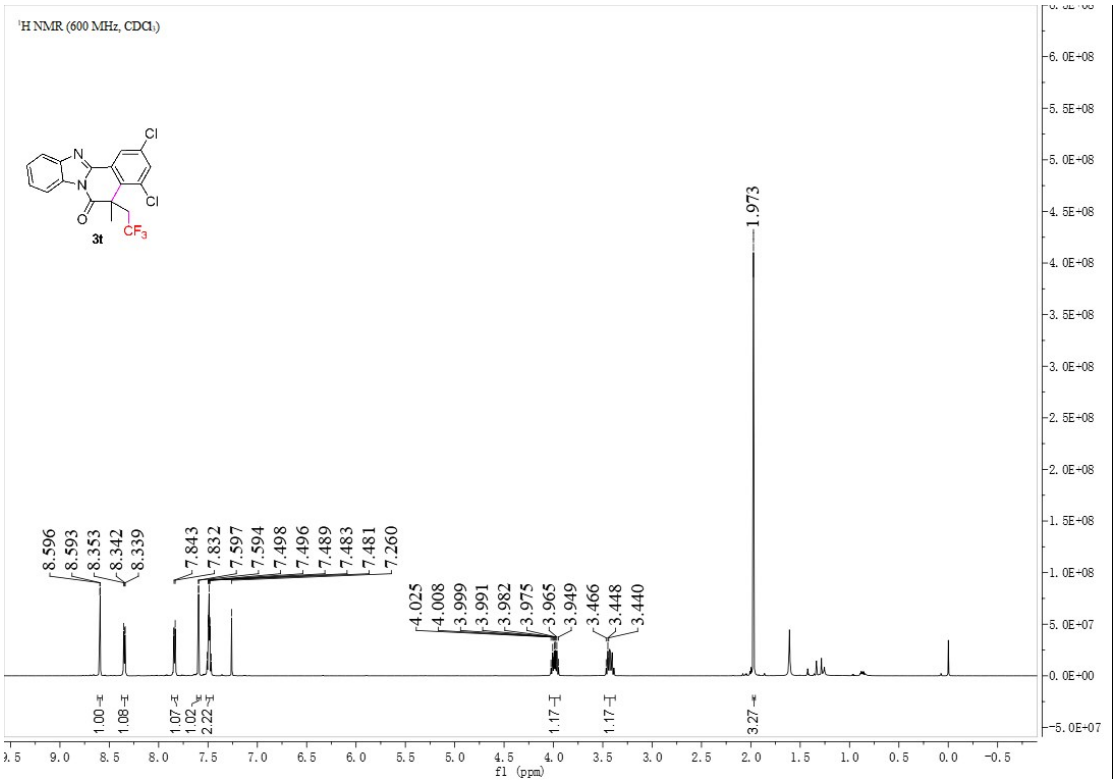

¹³C NMR (151 MHz, CDCl₃)
3r
127.936
126.091
124.241
122.394
45.272
45.261
44.594
44.412
44.230
44.047
77.372
77.160
76.948
171.446
149.829
144.391
140.732
140.185
139.696
132.874
130.671
127.936
126.091
125.910
125.726
125.064
124.241
122.394
120.192
118.494
115.797
31.654
24.675
21.694
f1 (ppm)

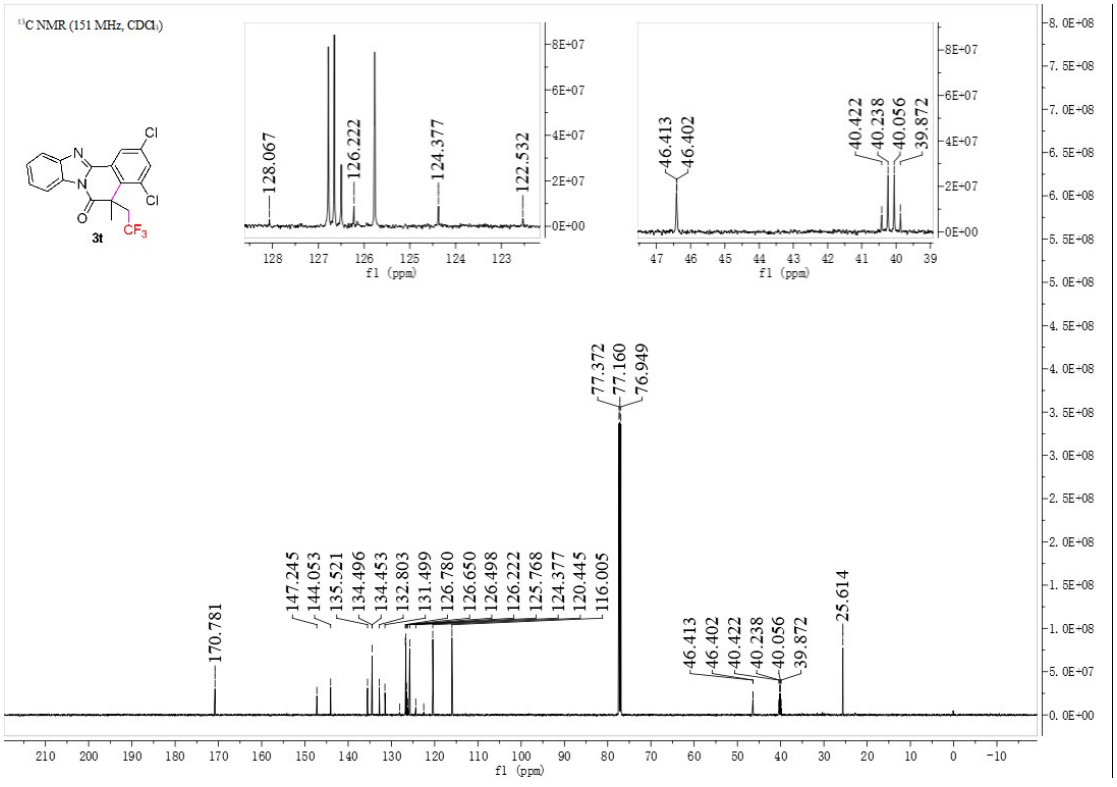

^{19}F NMR (377 MHz, CDCl$_3$)
3r
-61.159
f1 (ppm)

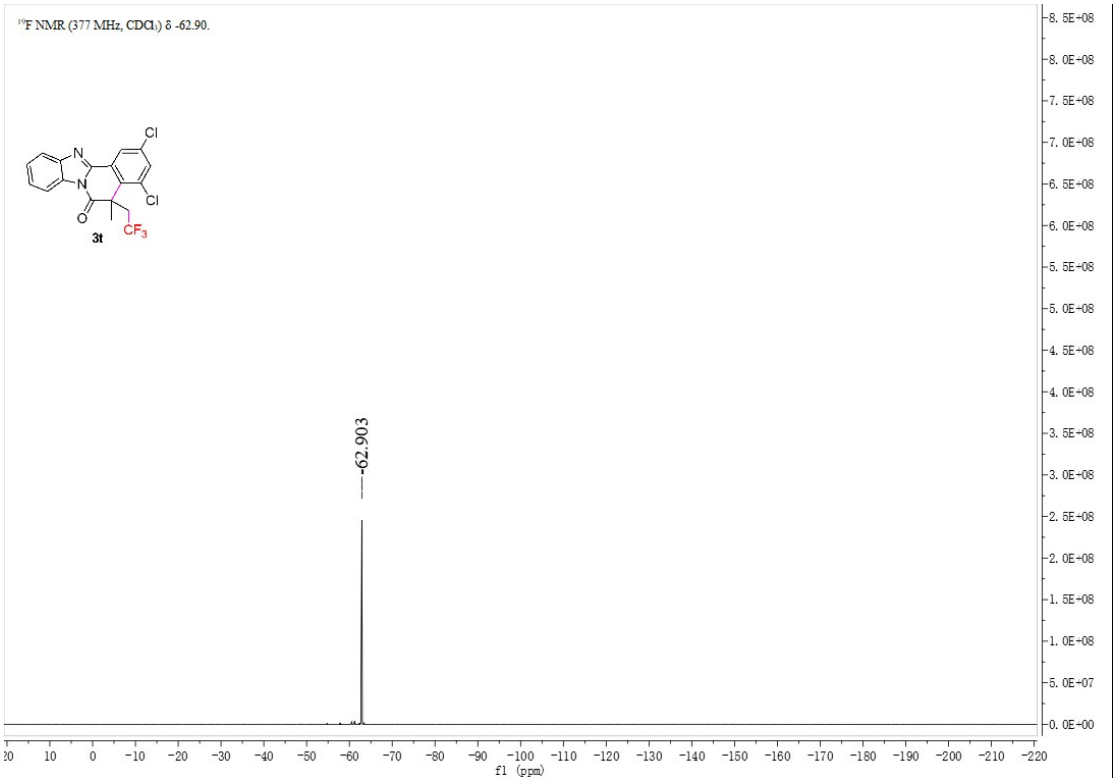

^{1}H NMR (600 MHz, CDCl$_3$)
3s
8.374
8.368
8.359
7.918
7.910
7.903
7.613
7.610
7.496
7.487
7.480
7.475
7.472
7.464
7.391
7.389
7.260
3.532
3.516
3.507
3.499
3.490
3.482
3.473
3.456
2.920
2.904
2.894
2.888
2.878
2.872
2.862
2.846
1.785
1.00
1.00
0.91
2.05
1.07
1.12
1.12
3.07
f1 (ppm)

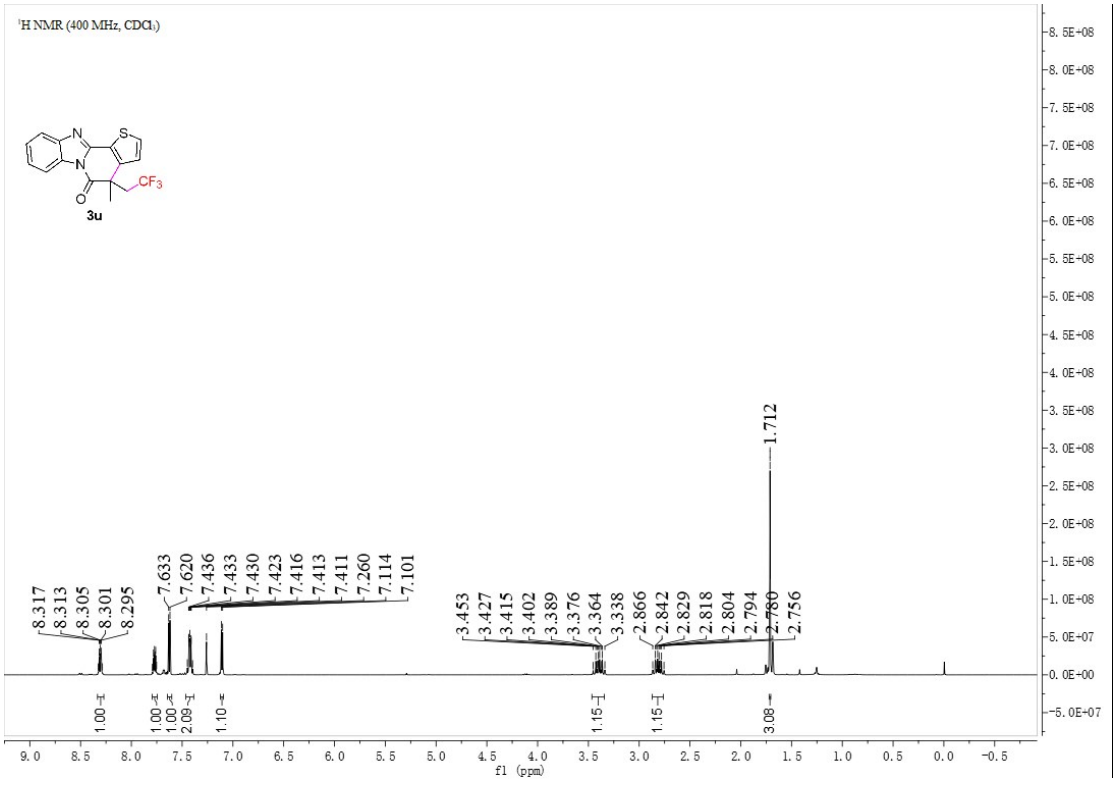

13C NMR (151 MHz, CDCl3)
Cl
Cl
N
N
O
CF3
3s
127.587
125.737
123.891
122.042
77.372
77.160
76.948
45.583
44.654
44.470
44.283
44.099
145.960
143.955
142.499
136.752
134.937
132.057
130.547
127.587
126.940
126.484
125.885
125.737
123.891
122.042
121.033
119.422
115.834
169.553
31.260
f1 (ppm)

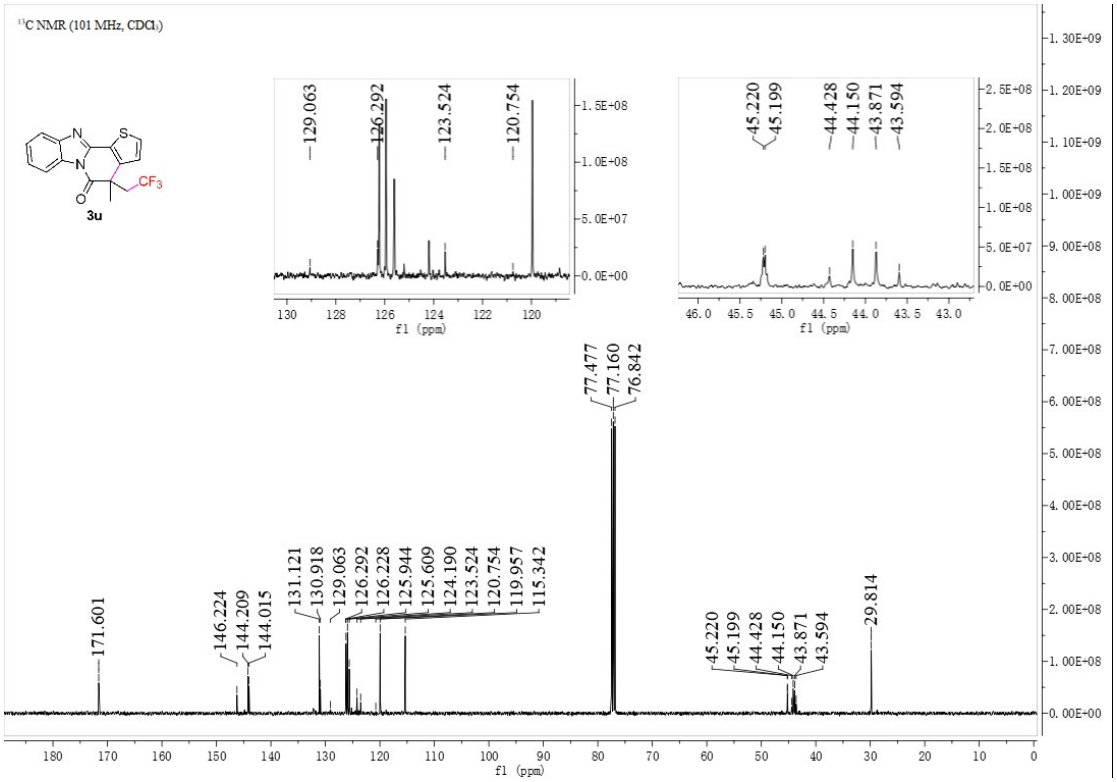

19F NMR (565 MHz, CDCl3)
Cl
Cl
N
N
O
CF3
3s
-61.207
f1 (ppm)

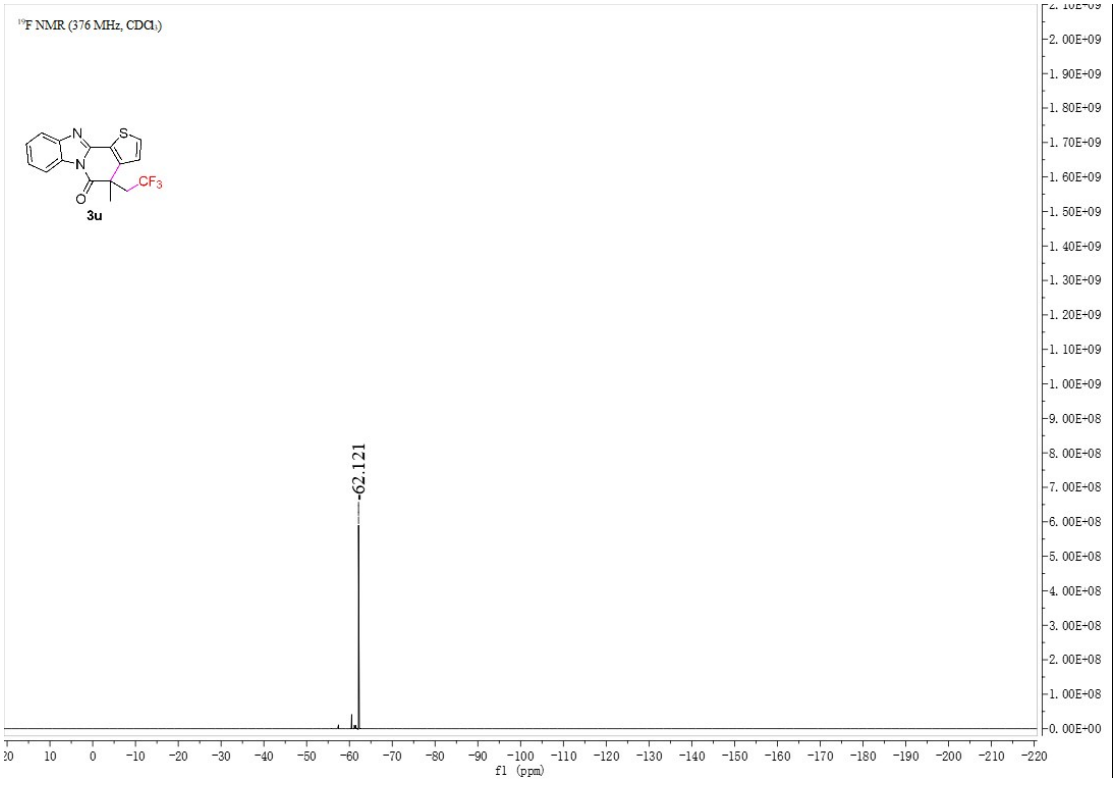

^{1}H NMR (600 MHz, CDCl$_3$)
3t
8.596
8.593
8.353
8.342
8.339
7.843
7.832
7.597
7.594
7.498
7.496
7.489
7.483
7.481
7.260
4.025
4.008
3.999
3.991
3.982
3.975
3.965
3.949
3.466
3.448
3.440
1.973
1.00
1.08
1.07
1.02
2.22
1.17
1.17
3.27
f1 (ppm)

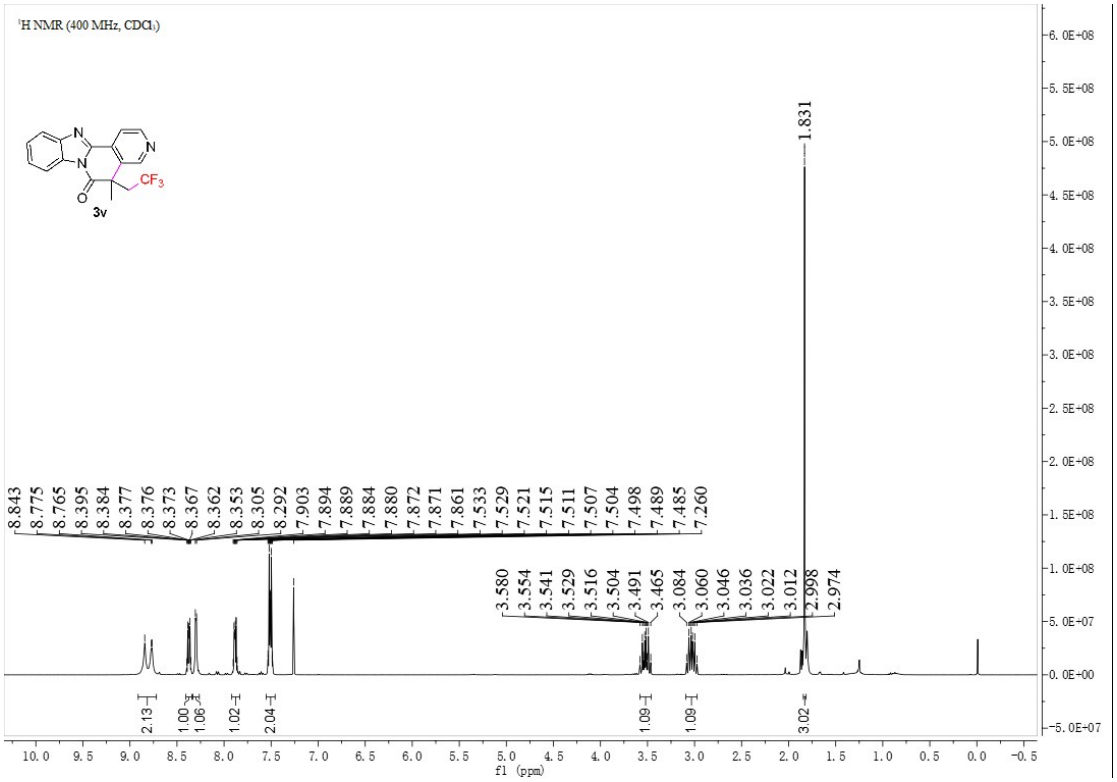

^{13}C NMR (151 MHz, CDCl$_3$)
3t
128.067
126.222
124.377
122.532
46.413
46.402
40.422
40.238
40.056
39.872
f1 (ppm)
77.372
77.160
76.949
170.781
147.245
144.053
135.521
134.496
134.453
132.803
131.499
126.780
126.650
126.498
126.222
125.768
124.377
120.445
116.005
46.413
46.402
40.422
40.238
40.056
39.872
25.614
f1 (ppm)

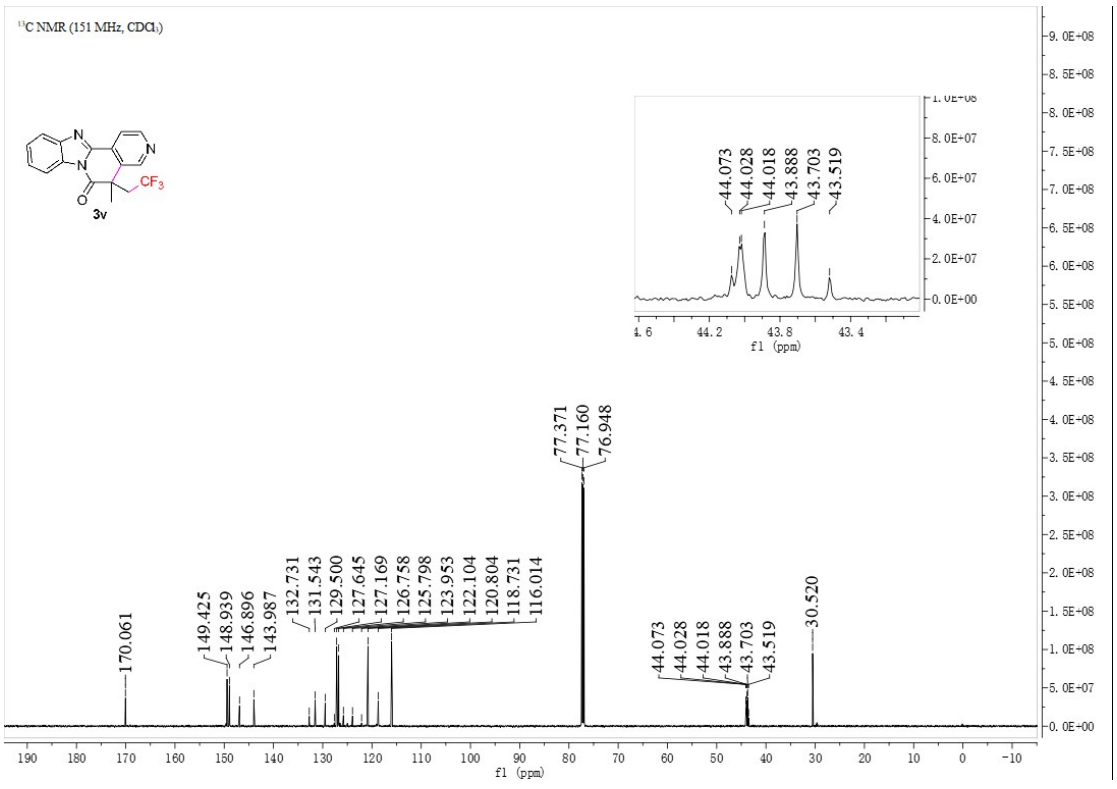

19F NMR (377 MHz, CDCl3) δ -62.90.
Cl
Cl
N
N
O
CF3
3t
-62.903
f1 (ppm)

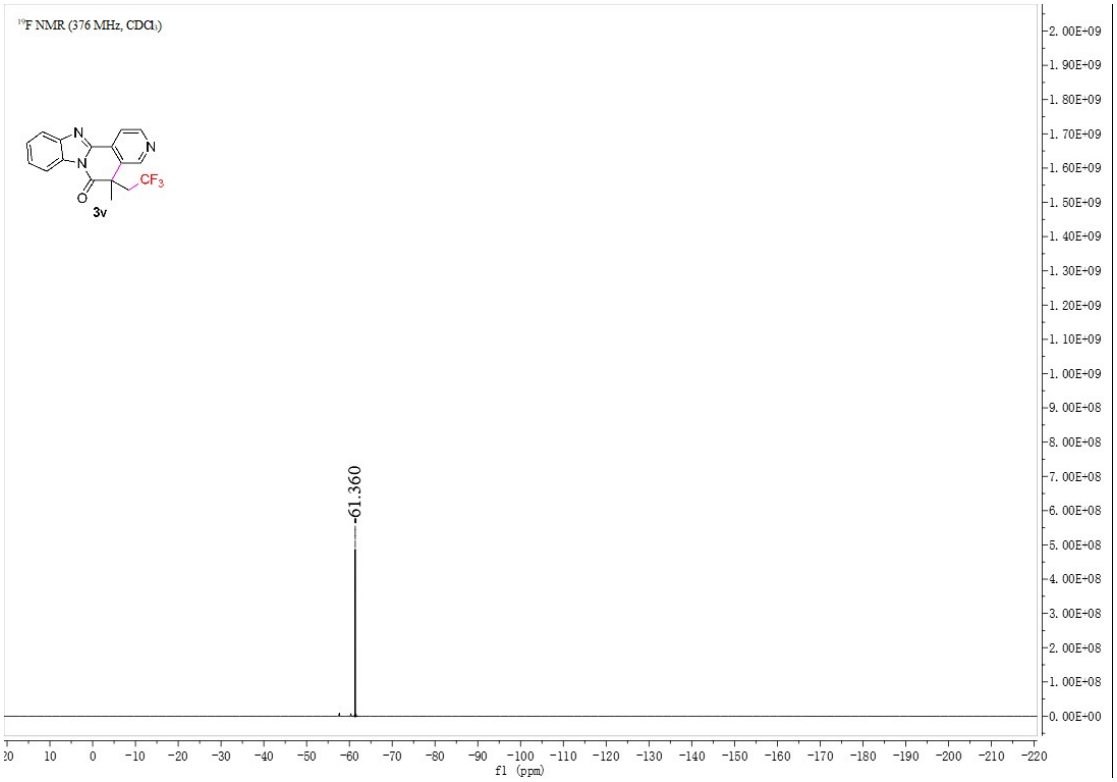

1H NMR (400 MHz, CDCl3)
N
S
N
O
CF3
3u
8.317
8.313
8.305
8.301
8.295
7.633
7.620
7.436
7.433
7.430
7.423
7.416
7.413
7.411
7.260
7.114
7.101
3.453
3.427
3.415
3.402
3.389
3.376
3.364
3.338
2.866
2.842
2.829
2.818
2.804
2.794
2.780
2.756
1.712
1.00
1.00
2.09
1.10
1.15
1.15
3.08
f1 (ppm)

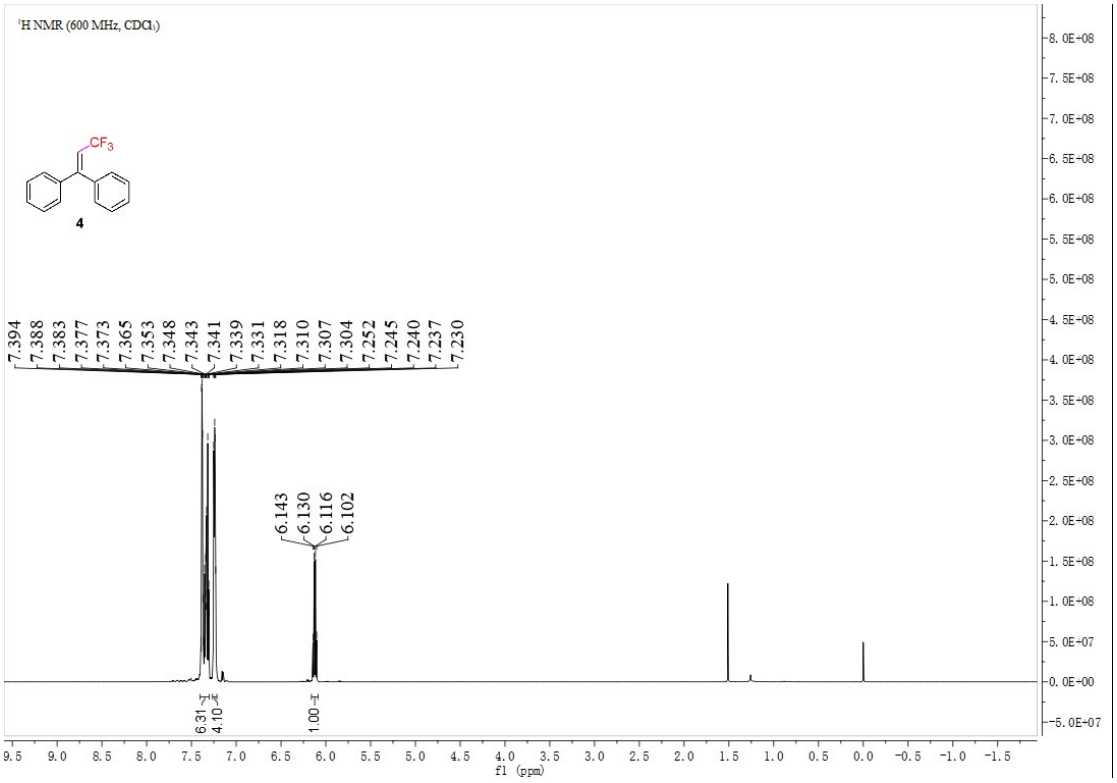

13C NMR (101 MHz, CDCl3)
3u
171.601
146.224
144.209
144.015
131.121
130.918
129.063
126.292
126.228
125.944
125.609
124.190
123.524
120.754
119.957
115.342
77.477
77.160
76.842
45.220
45.199
44.428
44.150
43.871
43.594
29.814
f1 (ppm)

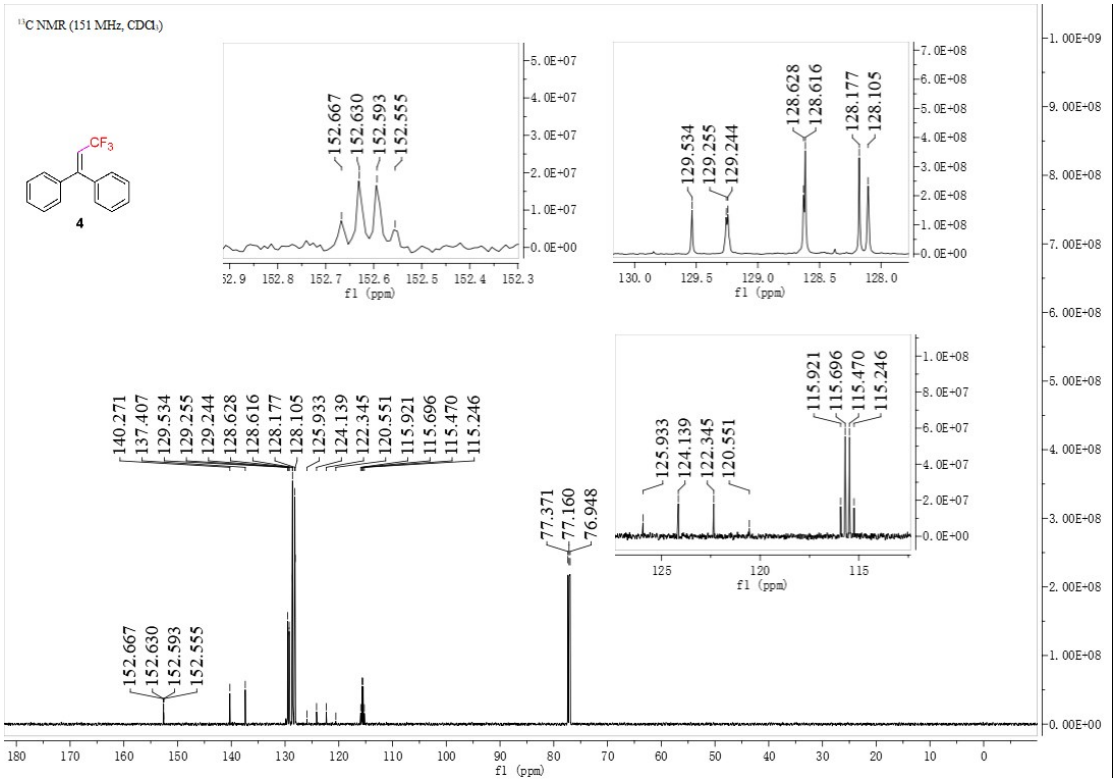

19F NMR (376 MHz, CDCl3)
3u
-62.121
f1 (ppm)

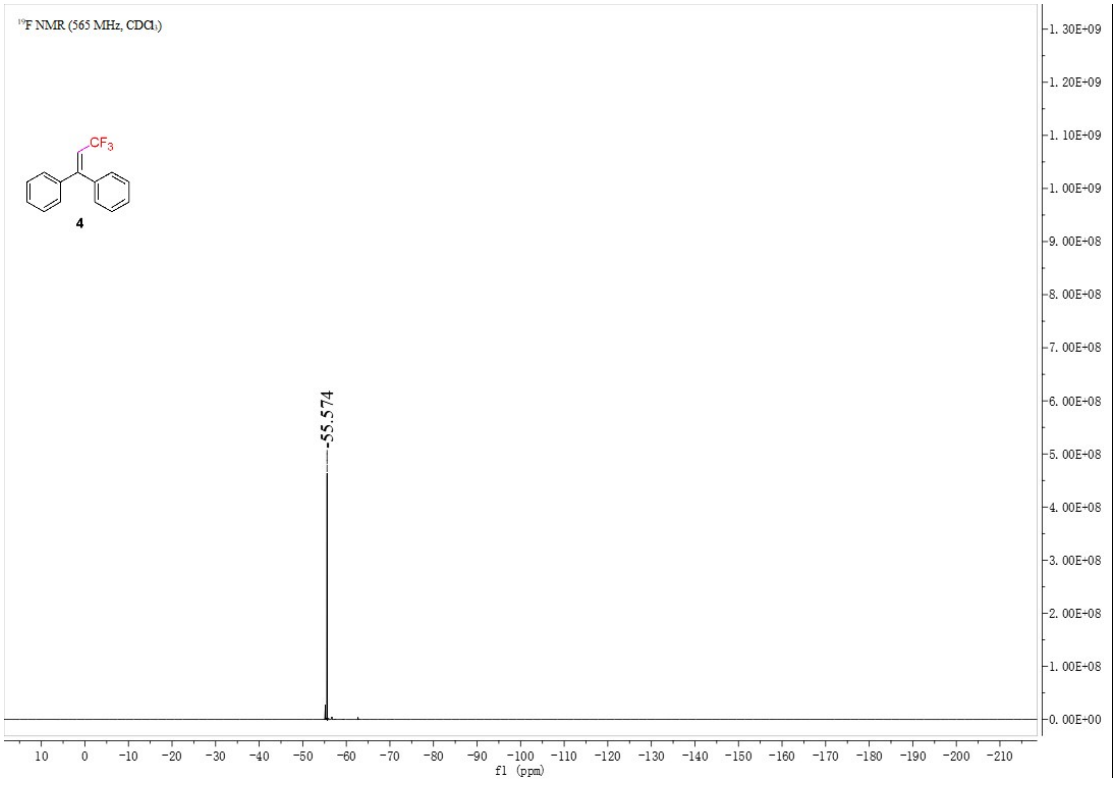

¹H NMR (400 MHz, CDCl₃)
3v
1.831
8.843
8.775
8.765
8.395
8.384
8.377
8.376
8.373
8.367
8.362
8.353
8.305
8.292
7.903
7.894
7.889
7.884
7.880
7.872
7.871
7.861
7.533
7.529
7.521
7.515
7.511
7.507
7.504
7.498
7.489
7.485
7.260
3.580
3.554
3.541
3.529
3.516
3.504
3.491
3.465
3.084
3.060
3.046
3.036
3.022
3.012
2.998
2.974
2.13
1.00
1.08
1.02
2.04
1.09
1.09
3.02
f1 (ppm)

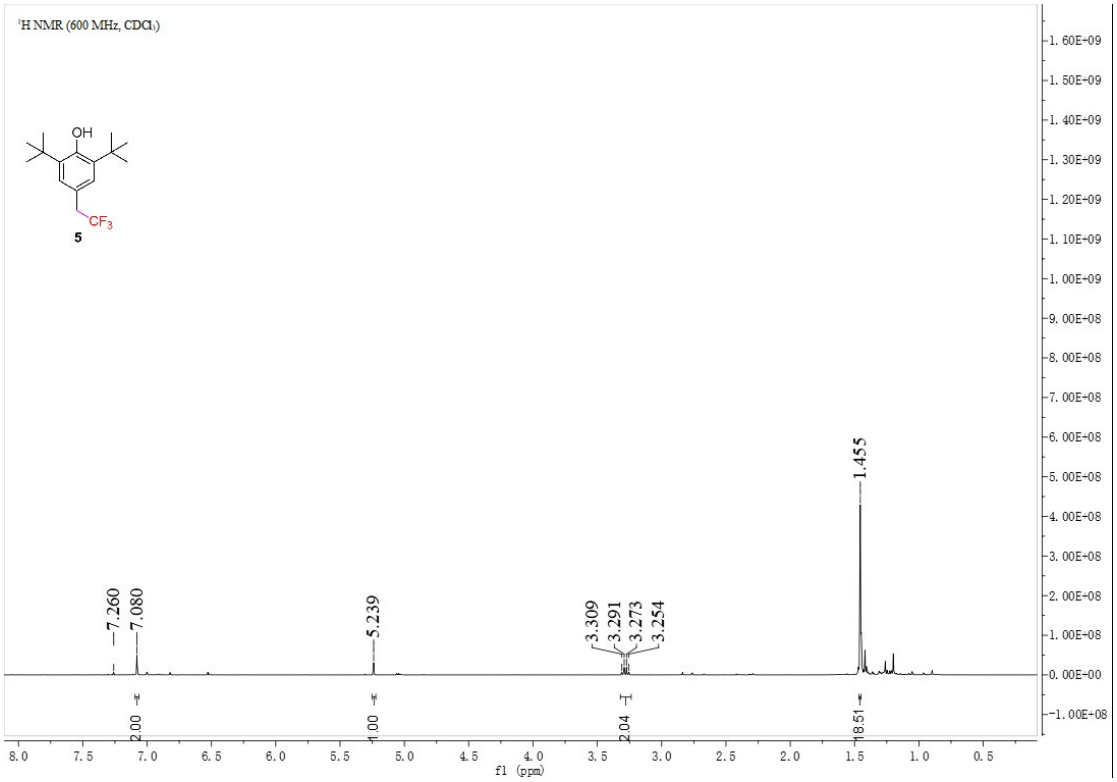

¹³C NMR (151 MHz, CDCl₃)
3v
44.073
44.028
44.018
43.888
43.703
43.519
77.371
77.160
76.948
170.061
149.425
148.939
146.896
143.987
132.731
131.543
129.500
127.645
127.169
126.758
125.798
123.953
122.104
120.804
118.731
116.014
30.520
f1 (ppm)

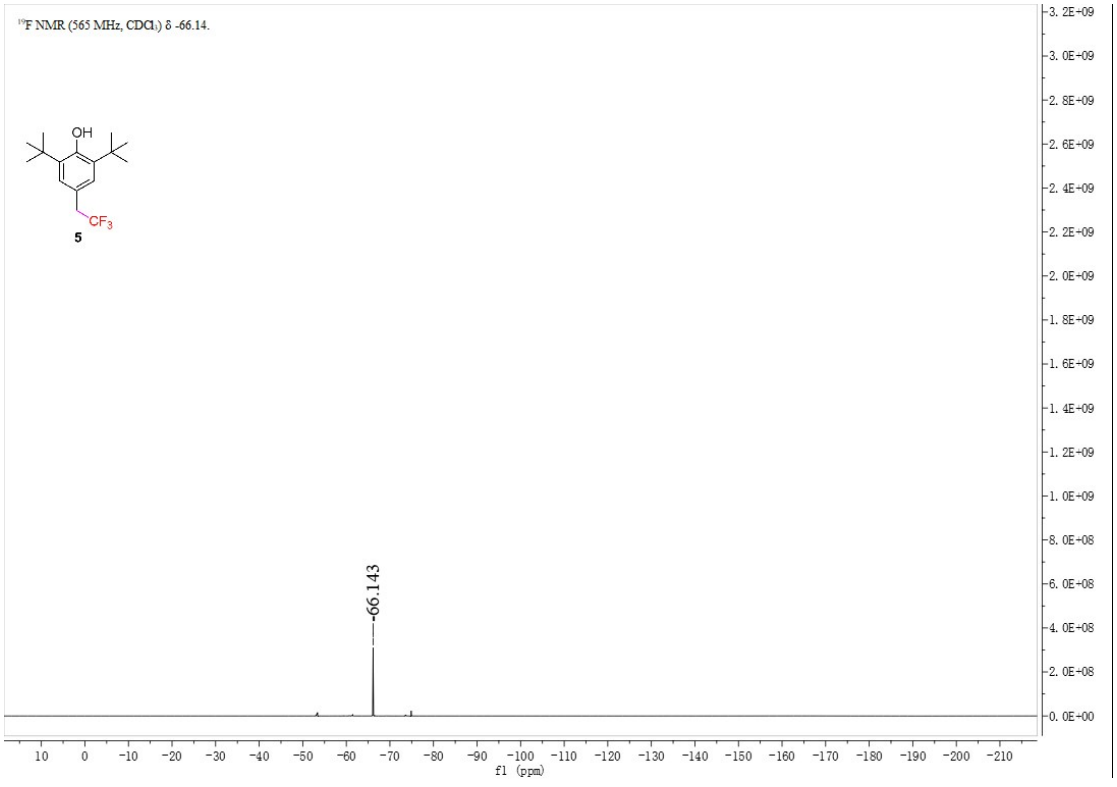

^{19}F NMR (376 MHz, CDCl$_3$)
CF$_3$
3v
-61.360
f1 (ppm)


^{1}H NMR (600 MHz, CDCl$_3$)
CF$_3$
4
7.394
7.388
7.383
7.377
7.373
7.365
7.353
7.348
7.343
7.341
7.339
7.331
7.318
7.310
7.307
7.304
7.252
7.245
7.240
7.237
7.230
6.143
6.130
6.116
6.102
6.31
4.10
1.00
f1 (ppm)


^{13}C NMR (151 MHz, CDCl$_3$)
CF$_3$
4
152.667
152.630
152.593
152.555
129.534
129.255
129.244
128.628
128.616
128.177
128.105
125.933
124.139
122.345
120.551
115.921
115.696
115.470
115.246
140.271
137.407
77.371
77.160
76.948
f1 (ppm)


^{19}F NMR (565 MHz, CDCl$_3$)
CF$_3$
4
-55.574
f1 (ppm)


1H NMR (600 MHz, CDCl3)
OH
CF3
5
7.260
7.080
5.239
3.309
3.291
3.273
3.254
1.455
2.00
1.00
2.04
18.51
f1 (ppm)


19F NMR (565 MHz, CDCl3) δ -66.14.
OH
CF3
5
-66.143
f1 (ppm)