Supplementary Information

Catalyst-free Photoinduced Radical Sulfonylation/Cyclization of Unactivated Alkenes toward Sulfone-containing Quinazolinones

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1 Supplementary Notes

Chemicals were purchased from commercial suppliers and used without further purification unless otherwise stated. Analytical thin layer chromatography (TLC) was performed on precoated silica gel 60 GF254 plates. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040-0.063 mm). Visualization on TLC was achieved by use of UV light (254 nm). ^{1}H and ^{13}C NMR spectra were recorded on Bruker 400 MHz spectrometer in $CDCl_3$ with tetramethylsilane (TMS) as internal standard. The chemical shifts are expressed in ppm and coupling constants are given in Hz. Data for ^{1}H NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet; d = doublet; t = triplet; q = quartet; p = pentet; m = multiplet; brs = broad singlet), coupling constant (Hz), integration. Data for ^{13}C NMR and ^{19}F NMR are reported in terms of chemical shift (δ, ppm). High resolution mass spectroscopy (HRMS) analyses were performed at a Q-Exactive (Thermo Scientific) Inc mass instrument (HESI).

2 Supplementary Methods

2.1 General procedures for the preparation of products 3

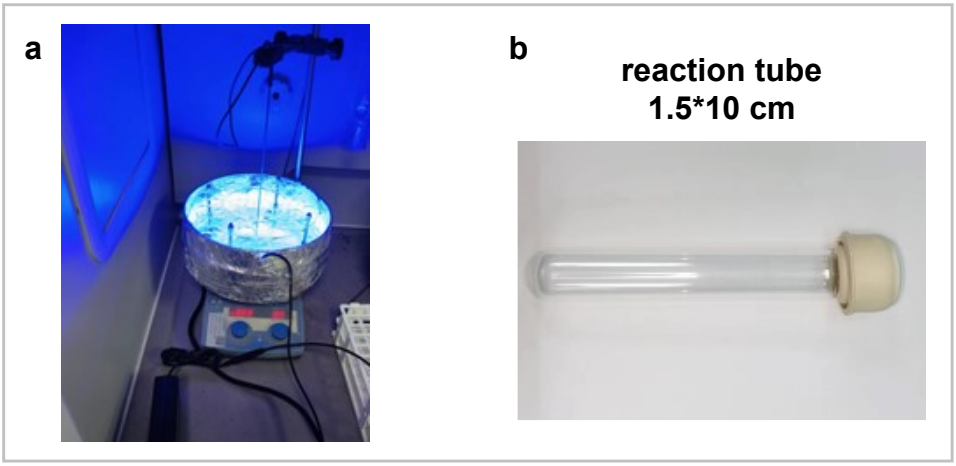
Set-up of the photoredox reaction: All photoredox reactions were carried out in the apparatus shown below.

Light source: 35 W LED strip, Greethink (Manufacturer), GT-5050-Blue (Model)

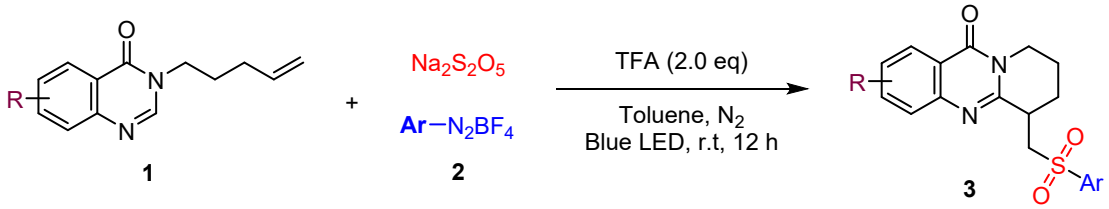
Wavelength of peak intensity: 460-470 nm

Material of the irradiation vessel: borosilicate glass

Distance of the irradiation vessel from the light source: approximately 3 cm.



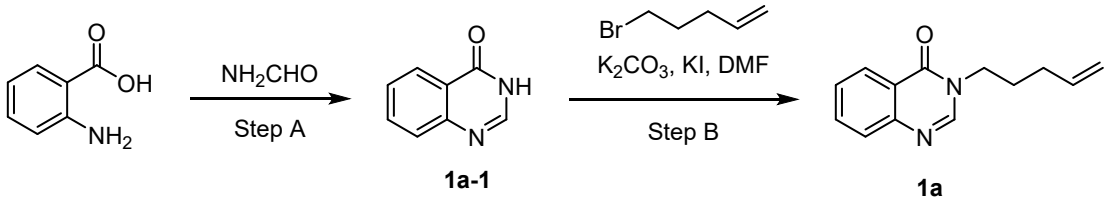
Supplementary Figure 1. Set-up of the photoredox reaction: **a** reaction device, **b** reaction tube.



Quinazolin-4(3H)-one **1** (42.8 mg, 0.2 mmol), $Na_2S_2O_5$ (57.0 mg, 0.3 mmol) and aryldiazonium salt **2** (61.8 mg, 0.3 mmol) were added to an oven-dried reaction vial equipped with a magnetic stirring bar. The reaction vessel was sealed with a rubber stopper, evacuated and refilled three times with N_2. Toluene (2.0 mL) and trifluoroacetic acid (45.6 mg, 0.4 mmol) were then added sequentially via syringe to the above system. The reaction mixture was stirred and irradiated with a 35 W blue LED light at room temperature for 12 h. After completion of the reaction monitored by

TLC, 1 mL of saturated sodium carbonate solution was added and stirred for 5 minutes. The reaction solution was diluted with 10 mL of water and extracted with dichloromethane (15 mL x 3). The combined organic phase was concentrated, and the resulting residue was purified flash chromatography on silica gel eluted with PE/EA (5/1) to afford the corresponding products **3**.

2.2 Procedure for the Synthesis of Substrates 1 (with 1a as an example)[1]

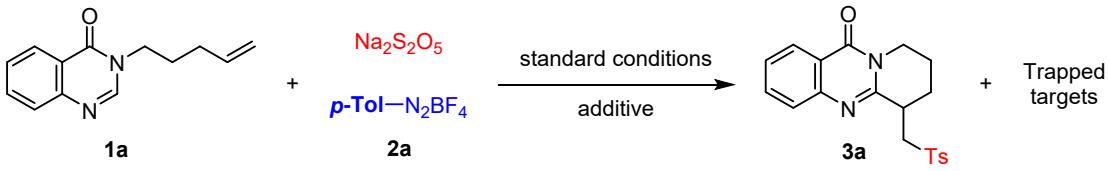


Step A: Synthesis of quinazoline-4(3*H*)-one 1a-1: To a 10 mL round-bottomed flask was added anthranilic acid (686mg, 5 mmol) and formamide (2.0 mL, 50 mmol). The reaction mixture was stirred at 130°C for 4 h. After complete consumption of anthranilic acid, the reaction mixture was cooled to room temperature and then poured into ice water. The resulting light precipitates were filtered and washed three times with water (100 mL) and dried under vacuum to give quinazoline-4(3*H*)-one, which was used for the next step without further purification.

Step B: Synthesis of quinazoline-4(3*H*)-one derivative 1a: A 150 mL round-bottom flask was charged with quinazolin-4(3*H*)-one (731 mg, 5 mmol), potassium carbonate (1.38 g, 10 mmol), and DMF (50 mL). The resulting mixture was heated to 80 ℃ with stirring for 30 minutes, then KI (83, 5 mmol) was added. After further stirring for 15 minutes, the solution of brominated olefins (711 *u*L, 6 mmol) in DMF (5 mL) was added dropwise to the mixture. The reaction mixture was heated to 60 ℃ in an oil bath and stirred for 3 h. After completion of the reaction monitored by TLC, the mixture was cooled to room temperature, diluted with water and extracted three times with ethyl acetate. The combined organic layer was washed with brine, dried with anhydrous Na_2SO_4 and filtered. The filtrate was concentrated under vacuo. The crude product was purified by flash chromatography on silica gel (PE:EA =10:1) to afford product **1a** as a yellow solid.

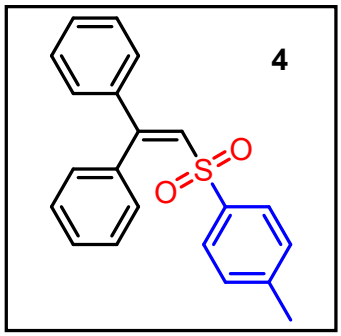
2.3. Mechanistic studies

2.3.1 Radical trapping experiments



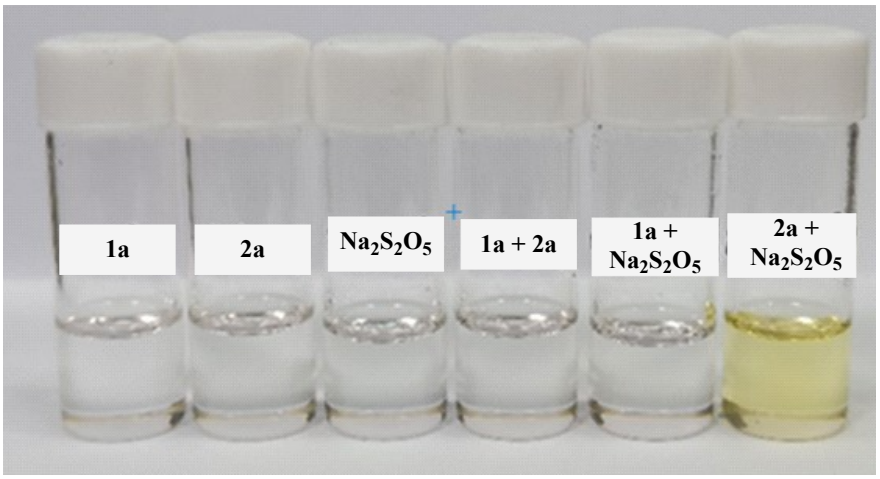
Entry	Additive	**3a**	Trapped targets
1	TEMPO	0	0
2	1,1-Diphenylethylene	0	4, 35%

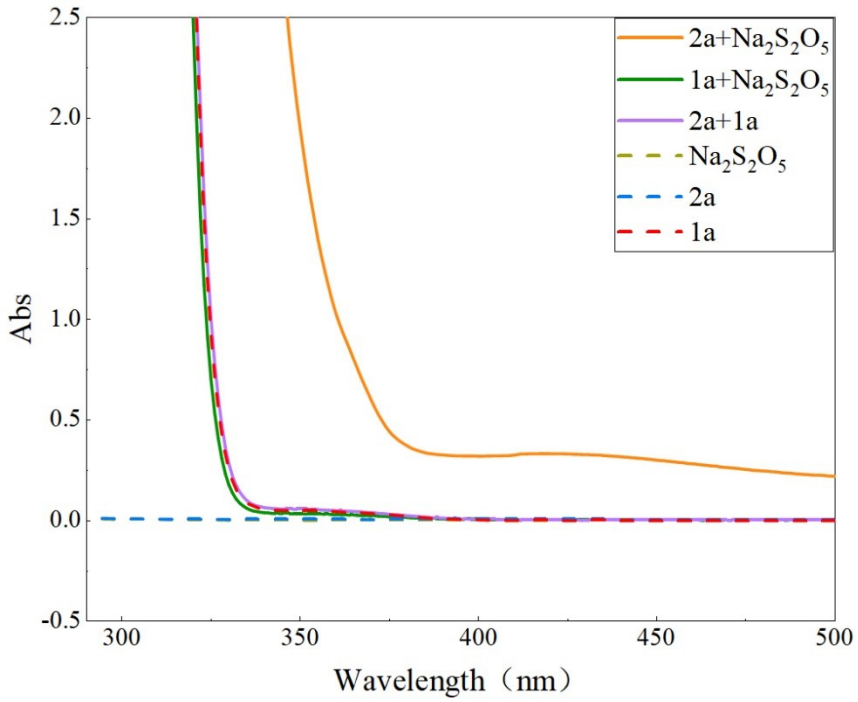
The synthesis of 4 is carried out under standard conditions with the addition of a radical scavenger (3.0 equiv). When the reaction is completed (monitored by TLC), the mixture is purified by flash chromatography on silica gel to afford the product **3a** and trapped targets. The compound structure is determined by NMR.



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.3 Hz, 2H), 7.38-7.35 (m, 2H), 7.32-7.28 (m, 4H), 7.21-7.19 (m, 2H), 7.15 (d, *J* = 8.3 Hz, 2H), 7.11-7.08 (m, 2H), 6.99 (s, 1H), 2.38 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 154.7, 143.8, 139.2, 138.6, 135.6, 130.3, 129.8, 129.4, 129.0, 128.9, 128.6, 128.2, 127.8, 127.7, 21.6. **HRMS (ESI)** calcd for $[M+Na]^+$ $C_{21}H_{18}O_2SNa$, m/z: 357.0920, found: 357.0918.

2.3.2 Ultraviolet-visible (UV-vis) spectra[2]

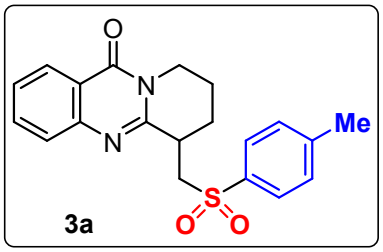




UV–vis spectra of 1a (0.01 M), 2a (0.01 M), $Na_2S_2O_5$(0.01 M) and their EDA complexes in Toluene.

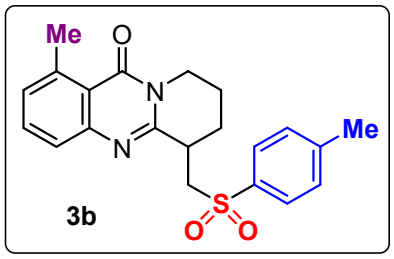
2.4 Characterization data of products 3

6-(Tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3a)



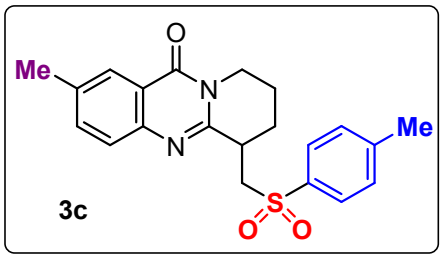
According to the general procedure, **3a** was obtained in 67% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.22 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.84 (d, *J* = 8.3 Hz, 2H), 7.70-7.66 (m, 1H), 7.48 (d, *J* = 8.2 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 4.40 (d, *J* = 11.3 Hz, 1H), 4.30-4.23 (m, 1H), 3.99-3.93 (m, 1H), 3.51-3.40 (m, 2H), 2.68-2.60 (m, 1H), 2.41 (s, 3H), 2.09-1.97 (m, 2H), 1.89-1.79 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.8, 154.2, 144.9, 137.0, 134.2, 123.0, 127.9, 126.7, 126.7, 120.2, 58.1, 41.24, 36.0, 25.3, 21.7, 20.8. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{21}N_2O_3S$, m/z: 369.1267, found: 369.1265.

1-Methyl-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3b)



According to the general procedure, **3b** was obtained in 45% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.83 (d, *J* = 8.0 Hz, 2H), 7.49 (t, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.2 Hz, 1H), 7.15 (d, *J* = 7.3 Hz, 1H), 4.41-4.35 (m, 1H), 4.18-4.11 (m, 1H), 3.95-3.88 (m, 1H), 3.41 (d, *J* = 9.3 Hz, 2H), 2.82 (s, 3H), 2.63-2.57 (m, 1H), 2.41 (s, 3H), 2.07-1.93 (m, 2H), 1.85-1.78 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.3, 153.7, 148.1, 144.8, 140.9, 137.0, 133.2, 129.9, 129.2, 127.9, 125.0, 118.7, 58.2, 41.1, 36.0, 25.32, 2.1, 21.7, 21.0. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{23}N_2O_3S$, m/z: 383.1424, found: 383.1423.

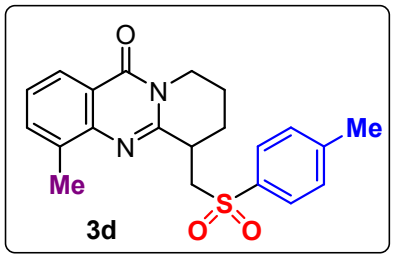
2-Methyl-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3c)



According to the general procedure, **3c** was obtained in 54% yield as yellow solid. **^{1}H NMR**

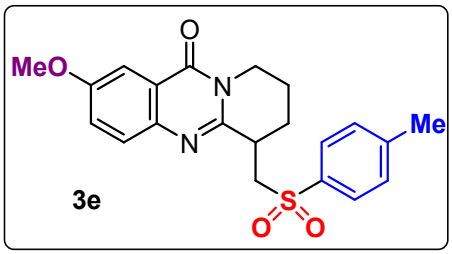
(400 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.83 (d, *J* = 8.1 Hz, 2H), 7.48 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.35 (d, *J* = 8.4 Hz, 1H), 7.32 (d, *J* = 8.1 Hz, 2H), 4.39 (d, *J* = 11.5 Hz, 1H), 4.28-4.22 (m, 1H), 3.96-3.90 (m, 1H), 3.48-3.36 (m, 2H), 2.65-2.58 (m, 1H), 2.44 (s, 3H), 2.40 (s, 3H), 2.04-1.97 (m, 2H), 1.86-1.76 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.8, 153.2, 144.8, 137.0, 136.7, 135.6, 129.9, 127.9, 126.6, 126.0, 119.9, 58.2, 41.2, 36.0, 25.4, 21.7, 21.3, 20.8. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{23}N_2O_3S$, m/z: 383.1424, found: 383.1422.

4-methyl-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3d)



According to the general procedure, **3d** was obtained in 85% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.04 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 7.2 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 1H), 4.48 (dd, *J* = 14.1, 2.2 Hz, 1H), 4.19-4.12 (m, 1H), 4.02-3.96 (m, 1H), 3.48-3.34 (m, 2H), 2.60-2.56 (m, 1H), 2.44 (s, 3H), 2.39 (s, 3H), 2.08-1.94 (m, 2H), 1.87-1.77 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.2, 152.7, 145.0, 144.9, 136.7, 135.2, 134.7, 123.0, 127.9, 126.2, 124.3, 120.0, 58.3, 41.5, 36.5, 25.5, 21.6, 20.9, 17.2. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{23}N_2O_3S$, m/z: 383.1424, found: 383.1421.

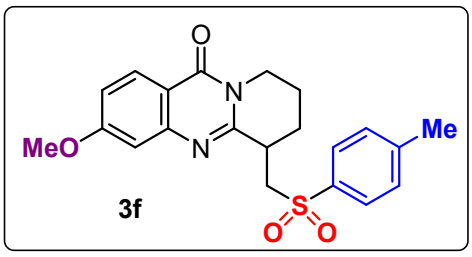
2-Methoxy-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3e)



According to the general procedure, **3e** was obtained in 53% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.84 (d, *J* = 8.1 Hz, 2H), 7.57 (d, *J* = 3.0 Hz, 1H), 7.40 (d, *J* = 8.9 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.28 (dd, *J* = 6.3, 2.7 Hz, 1H), 4.40 (dd, *J* = 13.9, 2.4 Hz, 1H), 4.31-4.24 (m, 1H), 4.00-3.94 (m, 1H), 3.90 (s, 3H), 3.50-3.36 (m, 2H), 2.68-2.58 (m, 1H), 2.42 (s, 3H), 2.11-2.01 (m, 2H), 1.88-1.78

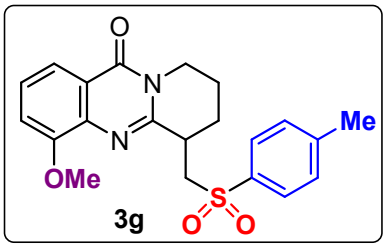
(m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.7, 158.2, 151.7, 144.8, 141.3, 137.1, 123.0, 128.4, 127.9, 124.6, 120.9, 105.8, 58.2, 55.8, 41.4, 35.9, 25.4, 21.7, 20.8. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{23}N_2O_4S$, m/z: 399.1373, found: 399.1374.

3-Methoxy-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3f)



According to the general procedure, **3f** was obtained in 54% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.03 (d, *J* = 8.9 Hz, 1H), 7.78 (d, *J* = 7.9 Hz, 2H), 7.28 (d, *J* = 7.9 Hz, 2H), 6.92 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.78 (s, 1H), 4.31 (d, *J* = 11.5 Hz, 1H), 4.20-4.13 (m, 1H), 3.89-3.85 (m, 1H), 3.81 (s, 3H), 3.42-3.30 (m, 2H), 2.59-2.51 (m, 1H), 2.35 (s, 3H), 1.99-1.88 (m, 2H), 1.80-1.70 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.4, 161.3, 154.9, 148.7, 144.9, 137.1, 123.0, 128.2, 127.9, 116.8, 113.8, 107.3, 58.2, 55.6, 41.1, 36.0, 25.3, 21.7, 20.7. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{23}N_2O_4S$, m/z: 399.1373, found: 399.1375.

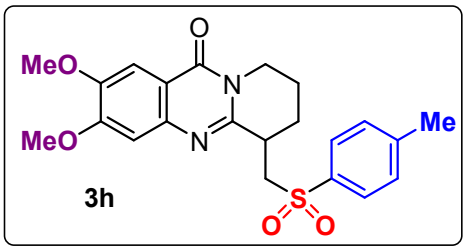
4-Methoxy-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3g)



According to the general procedure, **3g** was obtained in 71% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.83 (d, *J* = 8.0 Hz, 2H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.36 (t, *J* = 8.0 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 7.9 Hz, 1H), 4.32 (dd, *J* = 14.1, 2.9 Hz, 1H), 4.26-4.19 (m, 1H), 4.04-3.97 (m, 1H), 3.95 (s, 3H), 3.67-3.61 (m, 1H), 3.58-3.53 (m, 1H), 2.66-2.55 (m, 1H), 2.39 (s, 3H), 2.11-1.89 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.6, 153.9, 153.6, 144.8, 137.0, 129.8, 128.0, 127.0, 121.4, 118.1, 114.1, 58.0, 56.2, 41.5, 36.4, 25.0, 21.7, 20.8. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{23}N_2O_4S$, m/z: 399.1373, found: 399.1373.

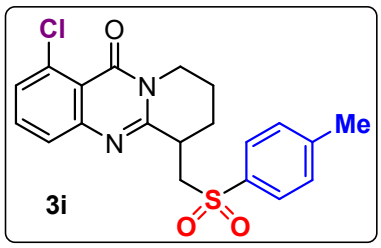
2,3-diMethoxy-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-

11-one (3h)



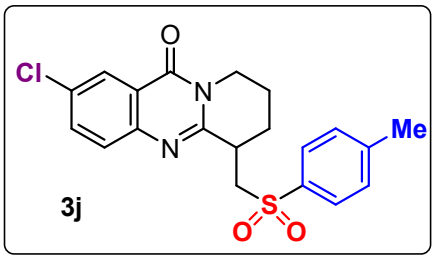
According to the general procedure, **3h** was obtained in 30% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.78 (d, *J* = 8.2 Hz, 2H), 7.46 (s, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.82 (s, 1H), 4.32 (dd, *J* = 14.1, 2.6 Hz, 1H), 4.22-4.16 (m, 1H), 3.93-3.87 (m, 1H), 3.91 (s, 3H), 3.90 (s, 3H), 3.43-3.28 (m, 2H), 2.60-2.52 (m, 1H), 2.36 (s, 3H), 2.02-1.92 (m, 2H), 1.87-1.70 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.1, 154.9, 152.8, 149.0, 144.8, 137.2, 129.9, 127.9, 113.6, 107.1, 105.5, 105.5, 58.3, 56.3, 56.3, 41.3, 35.9, 25.4, 21.6, 20.8. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{22}H_{25}N_2O_5S$, m/z: 429.1479, found: 429.1479.

1-Chloro-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3i)



According to the general procedure, **3i** was obtained in 51% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.52 (t, *J* = 8.0 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 4.37-4.30 (m, 1H), 4.18-4.11 (m, 1H), 3.99-3.92 (m, 1H), 3.46-3.41 (m, 2H), 2.63-2.56 (m, 1H), 2.42 (s, 3H), 2.11-1.96 (m, 2H), 1.89-1.80 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 159.8, 154.9, 145.0, 136.9, 134.0, 133.5, 123.0, 129.3, 127.9, 126.0, 117.3, 58.1, 41.6, 36.2, 25.1, 21.7, 20.8. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}ClN_2O_3S$, m/z: 403.0878, found: 403.0878.

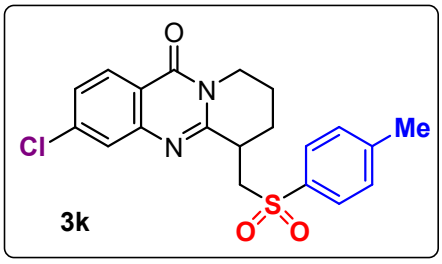
2-Chloro-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3j)



According to the general procedure, **3j** was obtained in 59% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.10 (d, *J* = 2.4 Hz, 1H), 7.77 (d, *J* = 8.0 Hz, 2H), 7.54 (dd, *J* = 8.7, 2.5 Hz, 1H),

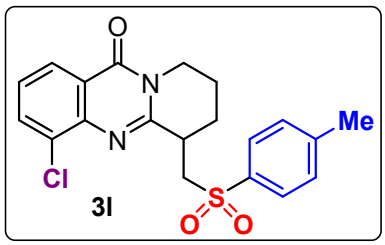
7.35 (d, J = 8.7 Hz, 1H), 7.28 (d, J = 8.0 Hz, 2H), 4.31-4.27 (m, 1H), 4.22-4.15 (m, 1H), 3.93-3.87 (m, 1H), 3.44-3.32 (m, 2H), 2.59-2.55 (m, 1H), 2.36 (s, 3H), 2.03-1.91 (m, 2H), 1.83-1.73 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 160.8, 154.5, 144.9, 137.0, 134.6, 132.3, 130.0, 128.5, 127.9, 126.0, 121.2, 58.1, 41.5, 36.1, 25.2, 21.7, 20.7. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}ClN_2O_3S$, m/z: 403.0878, found: 403.0877.

3-Chloro-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3k)



According to the general procedure, **3k** was obtained in 57% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.15-8.12 (m, 1H), 7.83 (dd, J = 8.3, 2.1 Hz, 2H), 7.44-7.42 (m, 1H), 7.38-7.34 (m, 3H), 4.33 (dd, J = 13.7, 2.5 Hz, 1H), 4.28-4.21 (m, 1H), 4.00-3.93 (m, 1H), 3.50-3.37 (m, 2H), 2.67-2.59 (m, 1H), 2.43 (s, 3H), 2.10-1.98 (m, 2H), 1.91-1.79 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.2, 155.6, 147.5, 145.0, 140.2, 137.1, 123.0, 128.2, 127.9, 127.2, 126.4, 118.6, 58.1, 41.3, 36.3, 25.31, 21.7, 20.7. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}ClN_2O_3S$, m/z: 403.0878, found: 403.0879.

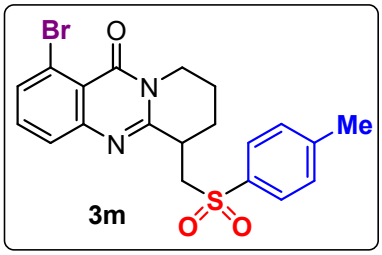
4-Chloro-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3l)



According to the general procedure, **3l** was obtained in 73% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.11 (dd, J = 8.0, 1.4 Hz, 1H), 7.85 (d, J = 8.0 Hz, 2H), 7.75 (dd, J = 7.7, 1.4 Hz, 1H), 7.34-7.30 (m, 3H), 4.47 (dd, J = 14.4, 3.0 Hz, 1H), 4.15-4.02 (m, 2H), 3.60 (dd, J = 14.4, 8.1 Hz, 1H), 3.44-3.39 (m, 1H), 2.64-2.58 (m, 1H), 2.38 (s, 3H), 2.13-2.07 (m, 1H), 1.99-1.89 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.3, 154.9, 144.8, 143.3, 136.6, 134.4, 131.4, 129.9, 128.0, 126.6, 125.5, 121.7, 57.8, 42.0, 36.8, 25.3, 21.6, 21.0. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}ClN_2O_3S$, m/z: 403.0878,

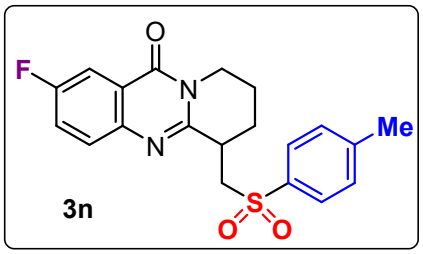
found: 403.0876.

1-Bromo-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3m)



According to the general procedure, **3m** was obtained in 65% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81 (d, *J* = 8.0 Hz, 2H), 7.63 (dd, *J* = 6.6, 2.4 Hz, 1H), 7.43-7.38 (m, 2H), 7.32 (d, *J* = 7.9 Hz, 2H), 4.35-4.29 (m, 1H), 4.16-4.09 (m, 1H), 3.97-3.91 (m, 1H), 3.41 (d, *J* = 9.3 Hz, 2H), 2.60-2.54 (m, 1H), 2.40 (s, 3H), 2.09-1.95 (m, 2H), 1.87-1.78 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 159.9, 154.7, 148.8, 144.9, 136.9, 133.8, 133.1, 123.0, 127.9, 126.9, 121.2, 118.2, 58.0, 41.8, 36.2, 25.1, 21.7, 20.9. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}BrN_2O_3S$, m/z: 447.0373, 449.0352, found: 447.0731, 449.0351.

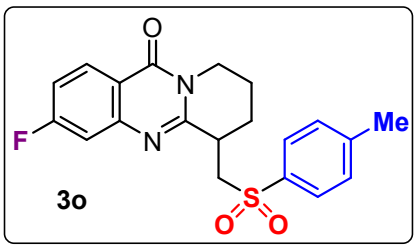
2-Fluoro-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3n)



According to the general procedure, **3n** was obtained in 68% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.84-7.82 (m, 1H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.48 (dd, *J* = 9.0, 4.9 Hz, 1H), 7.39 (td, *J* = 8.6, 2.9 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 4.36 (dd, *J* = 13.7, 2.3 Hz, 1H), 4.25 (dt, *J* = 13.6, 6.5 Hz, 1H), 3.96 (dt, *J* = 13.5, 6.2 Hz, 1H), 3.48-3.37 (m, 2H), 2.67-2.59 (m, 1H), 2.42 (s, 3H), 2.11-1.97 (m, 2H), 1.88-1.78 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.1, 160.7 (d, *J* =246.5 Hz), 153.5, 144.9, 143.2, 137.0, 123.0, 129.2 (d, *J* =8.0 Hz), 127.9, 122.8 (d, *J* =24.1 Hz), 121.4 (d, *J* =8.6 Hz), 111.4 (d, *J* =23.6 Hz), 58.1, 41.4, 36.0, 25.3, 21.7, 20.7. **^{19}F NMR (377 MHz, $CDCl_3$)** δ -112.86. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}FN_2O_3S$, m/z: 387.1173, found:387.1171.

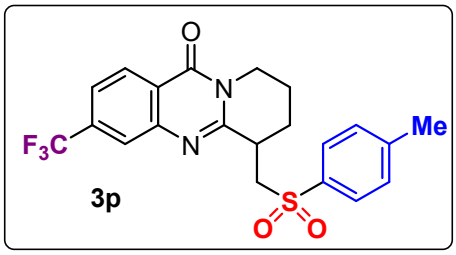
3-Fluoro-6-(tosylmethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one

(3o)



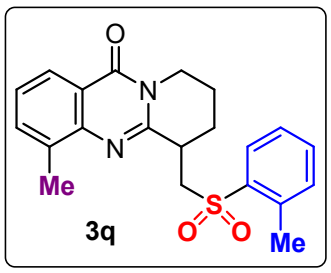
According to the general procedure, **3o** was obtained in 47% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.20 (dd, *J* = 8.8, 6.1 Hz, 1H), 7.82 (d, *J* = 7.9 Hz, 2H), 7.34 (d, *J* = 7.9 Hz, 2H), 7.13-7.05 (m, 2H), 4.35-4.32 (m, 1H), 4.23 (dt, *J* = 13.6, 6.5 Hz, 1H), 3.97-3.91 (m, 1H), 3.47-3.37 (m, 2H), 2.64-2.60 (m, 1H), 2.42 (s, 3H), 2.05-1.99 (m, 2H), 1.88-1.80 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 166.3 (d, *J* =252.3 Hz), 161.04, 155.59, 148.7 (d, *J* =13.1 Hz), 144.93, 137.02, 129.98, 129.4 (d, *J* =10.6 Hz), 127.92, 116.94, 115.4 (d, *J* =23.4 Hz), 112.0 (d, *J* =21.5 Hz), 58.1, 41.3, 36.2, 25.3, 21.7, 20.7. **^{19}F NMR (377 MHz, $CDCl_3$)** δ -103.58. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}FN_2O_3S$, m/z: 387.1173, found:387.1171.

6-(Tosylmethyl)-3-(trifluoromethyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3p)



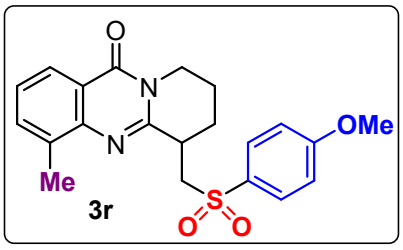
According to the general procedure, **3p** was obtained in 44% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.31 (d, *J* = 8.4 Hz, 1H), 7.82 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 1.8 Hz, 1H), 7.60 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 4.33 (dd, *J* = 14.0, 3.0 Hz, 1H), 4.29-4.24 (m, 1H), 3.98 (dt, *J* = 14.2, 6.2 Hz, 1H), 3.51-3.47 (m, 1H), 3.43 (dd, *J* = 14.0, 8.4 Hz, 1H), 2.68-2.61 (m, 1H), 2.40 (s, 3H), 2.08-2.03 (m, 2H), 1.92-1.82 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 160.9, 155.9, 146.4, 145.0, 137.1, 135.6 (q, *J* = 32.6 Hz), 1230.0, 127.9, 123.4 (q, *J* = 271.5 Hz), 124.3 (q, *J* = 4.0 Hz), 122.5 (q, *J* = 3.5 Hz), 122.39, 58.1, 41.5, 36.3, 25.3, 21.6, 20.7. **^{19}F NMR (377 MHz, $CDCl_3$)** δ -63.18. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{20}F_3N_2O_3S$, m/z: 437.1141, found:437.1141.

4-Methyl-6-((o-tolylsulfonyl)methyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3q)



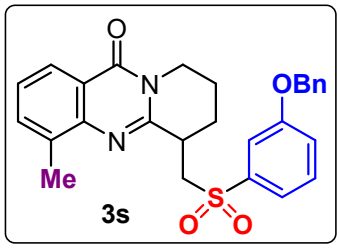
According to the general procedure, **3q** was obtained in 45% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.08-8.03 (m, 2H), 7.54-7.49 (m, 2H), 7.40-7.28 (m, 3H), 4.66 (d, *J* = 13.1 Hz, 1H), 4.19-4.12 (m, 1H), 4.06-3.99 (m, 1H), 3.45-3.36 (m, 2H), 2.80 (s, 3H), 2.66-2.59 (m, 1H), 2.47 (s, 3H), 2.10-1.94 (m, 2H), 1.88-1.79 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.2, 152.9, 145.0, 138.0, 137.7, 135.1, 134.8, 133.9, 132.9, 129.9, 126.7, 126.3, 124.4, 120.1, 57.5, 41.7, 36.3, 25.9, 20.9, 20.4, 17.2. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{23}N_2O_3S$, m/z: 383.1424, found: 383.1422.

6-(((4-Methoxyphenyl)sulfonyl)methyl)-4-methyl-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3r)



According to the general procedure, **3r** was obtained in 74% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.05 (d, *J* = 7.8 Hz, 1H), 7.88 (d, *J* = 8.6 Hz, 2H), 7.52 (d, *J* = 7.2 Hz, 1H), 7.29 (t, *J* = 7.8 Hz, 1H), 6.98 (d, *J* = 8.6 Hz, 2H), 4.48 (d, *J* = 12.9 Hz, 1H), 4.21-4.14 (m, 1H), 4.00 (dt, *J* = 14.0, 5.9 Hz, 1H), 3.84 (s, 3H), 3.48-3.37 (m, 2H), 2.63-2.55 (m, 1H), 2.46 (s, 3H), 2.06-1.97 (m, 2H), 1.89-1.81 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.8, 162.2, 152.8, 145.0, 135.2, 134.7, 131.2, 130.1, 126.2, 124.3, 120.1, 114.5, 58.5, 55.7, 41.5, 36.5, 25.6, 20.9, 17.2. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{23}N_2O_4S$, m/z: 399.1373, found: 399.1372.

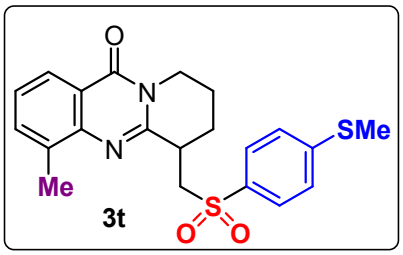
6-(((4-(Benzyloxy)phenyl)sulfonyl)methyl)-4-methyl-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3s)



According to the general procedure, **3s** was obtained in 31% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.06 (d, *J* = 8.4 Hz, 1H), 7.57-7.50 (m, 3H), 7.48-7.28 (m, 7H), 7.20 (dd, *J* = 8.3, 2.4 Hz, 1H), 5.08 (s, 2H), 4.52 (dd, *J* = 14.2, 2.4 Hz, 1H), 4.20-4.13 (m, 1H), 4.02-3.96

(m, 1H), 3.49-3.35 (m, 2H), 2.58-2.53 (m, 1H), 2.45 (s, 3H), 2.07-1.93 (m, 2H), 1.86-1.82 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.2, 159.2, 152.7, 145.0, 140.9, 135.8, 135.2, 134.7, 130.6, 128.7, 128.4, 127.6, 126.3, 124.3, 120.8, 120.2, 120.1, 113.5, 70.4, 58.3, 41.5, 36.4, 25.6, 20.9, 17.2. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{27}H_{27}N_2O_4S$, m/z: 475.1686, found: 475.1683.

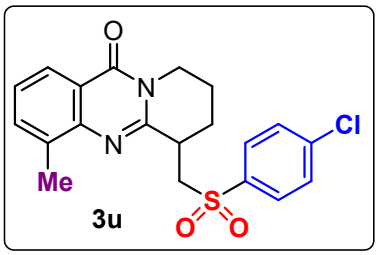
4-Methyl-6-(((4-(methylthio)phenyl)sulfonyl)methyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3t)



3t

According to the general procedure, **3t** was obtained in 32% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.06 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 2H), 7.53 (d, *J* = 6.6 Hz, 1H), 7.32-7.27 (m, 3H), 4.48 (dd, *J* = 14.0, 2.2 Hz, 1H), 4.21-4.14 (m, 1H), 4.04-3.99 (m, 1H), 3.48-3.37 (m, 2H), 2.59 (dt, *J* = 11.6, 5.8 Hz, 1H), 2.50 (s, 3H), 2.45 (s, 3H),2.10-1.94 (m, 2H), 1.89-1.83 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.2, 152.7, 147.5, 145.0, 135.2, 135.2, 134.7, 128.1, 126.2, 125.4, 124.3, 120.0, 58.4, 41.5, 36.5, 25.6, 20.9, 17.2, 14.7. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{23}N_2O_3S_2$, m/z: 415.1145, found: 415.1143.

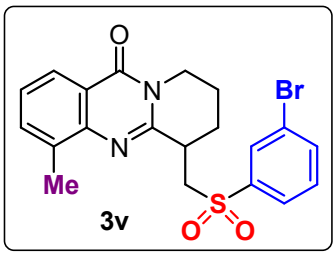
6-(((4-Chlorophenyl)sulfonyl)methyl)-4-methyl-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3u)



3u

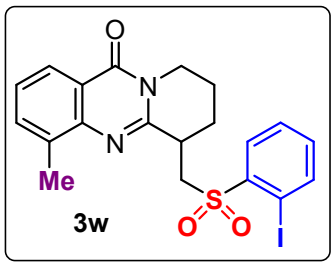
According to the general procedure, **3u** was obtained in 69% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.05 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.89 (d, *J* = 8.5 Hz, 2H), 7.52-7.50 (m, 3H), 7.30 (t, *J* = 7.6 Hz, 1H), 4.49 (d, *J* = 12.5 Hz, 1H), 4.21-4.14 (m, 1H), 4.05-3.98 (m, 1H), 3.49-3.39 (m, 2H), 2.62-2.55 (m, 1H), 2.43 (s, 3H), 2.10-1.98 (m, 2H), 1.89-1.79 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.1, 152.5, 144.9, 140.6, 138.3, 135.0, 134.8, 129.7, 129.3, 126.3, 124.4, 120.1, 58.4, 41.5, 36.4, 25.7, 20.9, 17.1. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}ClN_2O_3S$, m/z: 403.0878, found: 403.0878.

6-(((3-Bromophenyl)sulfonyl)methyl)-4-methyl-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3v)



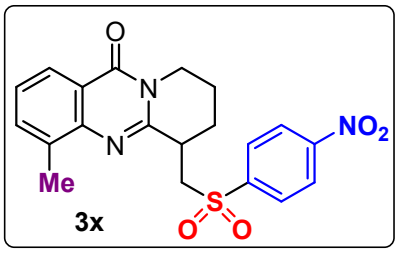
According to the general procedure, **3v** was obtained in 40% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.10 (s, 1H), 8.06 (d, *J* = 7.9 Hz, 1H), 7.89 (d, *J* = 7.7 Hz, 1H), 7.73 (d, *J* = 7.9 Hz, 1H), 7.53 (d, *J* = 7.2 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 1H), 4.51 (d, *J* = 14.2 Hz, 1H), 4.21-4.14 (m, 1H), 4.07-4.02 (m, 1H), 3.54-3.40 (m, 2H), 2.61-2.58 (m, 1H), 2.46 (s, 3H), 2.09-1.86 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.1, 152.4, 144.9, 141.7, 136.9, 135.1, 134.8, 130.9, 126.4, 126.3, 124.4, 123.5, 120.1, 58.4, 41.6, 36.5, 25.7, 21.0, 17.2. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}BrN_2O_3S$, m/z: 447.0373, 449.0352, found: 447.0371, 449.0348.

6-(((2-Iodophenyl)sulfonyl)methyl)-4-methyl-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3w)



According to the general procedure, **3w** was obtained in 40% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.20 (dd, *J* = 8.0, 1.6 Hz, 1H), 8.13 (d, *J* = 7.9 Hz, 1H), 8.07 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.57- 7.52 (m, 2H), 7.33-7.26 (m, 2H), 4.92 (d, *J* = 13.7 Hz, 1H), 4.26-4.19 (m, 1H), 4.04-3.98 (m, 1H), 3.65-3.56 (m, 2H), 2.65-2.60 (m, 1H), 2.50 (s, 3H), 2.11-1.95 (m, 2H), 1.91-1.81 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.2, 143.0, 142.2, 135.3, 134.8, 134.5, 131.4, 128.9, 126.3, 124.4, 120.1, 93.0, 55.9, 41.5, 36.2, 25.7, 20.8, 17.6. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}IN_2O_3S$, m/z: 495.0234, found: 495.0230.

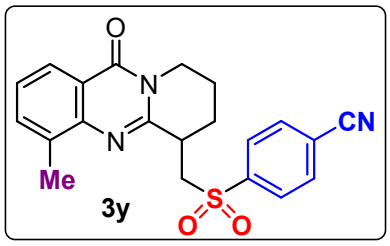
4-Methyl-6-(((4-nitrophenyl)sulfonyl)methyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3x)



According to the general procedure, **3x** was obtained in 47% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.38 (d, *J* = 8.2 Hz, 2H), 8.15 (d, *J* = 8.3 Hz,

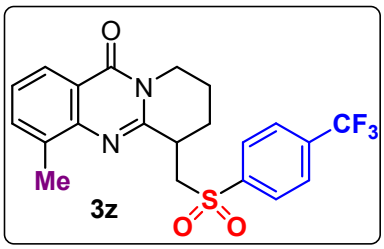
2H), 8.07 (d, J = 7.8 Hz, 1H), 7.53 (d, J = 7.3 Hz, 1H), 7.31 (t, J = 7.6 Hz, 1H), 4.53 (d, J = 14.1 Hz, 1H), 4.26-4.07 (m, 2H), 3.63-3.48 (m, 2H), 2.65-2.60 (m, 1H), 2.42 (s, 3H), 2.14-1.92 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.0, 150.8, 145.4, 135.0, 134.7, 129.4, 128.8, 126.6, 124.6, 124.5, 120.1, 58.5, 41.8, 36.4, 25.8, 21.0, 17.3. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{20}H_{20}N_3O_5S$, m/z: 414.1118, found: 414.1117.

4-(((4-methyl-11-oxo-6,8,9,11-tetrahydro-7H-pyrido[2,1-b]quinazolin-6-yl)methyl)sulfonyl)benzonitrile (3y)



According to the general procedure, **3y** was obtained in 63% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.08-8.05 (m, 3H), 7.83 (d, J = 8.5 Hz, 2H), 7.53 (d, J = 7.2 Hz, 1H), 7.32 (t, J = 7.6 Hz, 1H), 4.50 (dd, J = 14.4, 2.7 Hz, 1H), 4.20-4.02 (m, 2H), 3.55 (dd, J = 14.3, 8.3 Hz, 1H), 3.48-3.41 (m, 1H), 2.63-2.55 (m, 1H), 2.40 (s, 3H), 2.13-1.88 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.1, 152.3, 144.8, 144.0, 134.9, 133.1, 128.6, 126.5, 124.5, 120.1, 117.6, 117.0, 58.3, 41.6, 36.4, 25.8, 21.0, 17.1. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{20}N_3O_3S$, m/z: 394.1220, found: 394.1220.

4-Methyl-6-(((4-(trifluoromethyl)phenyl)sulfonyl)methyl)-6,7,8,9-tetrahydro-11H-pyrido[2,1-b]quinazolin-11-one (3z)



According to the general procedure, **3z** was obtained in 55% yield as yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.09 (d, J = 8.1 Hz, 2H), 8.05 (dd, J = 8.1, 1.5 Hz, 1H), 7.81 (d, J = 8.0 Hz, 2H), 7.51 (d, J = 7.2 Hz, 1H), 7.29 (t, J = 7.6 Hz, 1H), 4.52 (dd, J = 14.1, 2.4 Hz, 1H), 4.20-4.13 (m, 1H), 4.07-4.00 (m, 1H), 3.55-3.40 (m, 2H), 2.64-2.57 (m, 1H), 2.39 (s, 3H), 2.12-1.85 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.1, 152.4, 144.9, 143.3, 135.5 (q, J = 33.1 Hz), 135.0, 134.8, 128.5, 126.6 (q, J = 3.7 Hz), 126.4, 124.4, 123.0 (q, J = 271.5 Hz), 120.1, 58.4, 41.6, 36.4, 25.8, 20.9, 17.1. **^{19}F NMR (377 MHz, $CDCl_3$)** δ -63.18. **HRMS (ESI)** calcd for $[M+H]^+$ $C_{21}H_{20}F_3N_2O_3S$, m/z:

437.1141, found: 437.1138.

2.5 Crystal data and structure refinement for 3m

The crystal structure of compound **3m** has been deposited at the Cambridge Crystallographic Data Centre (**CCDC 2267811**).

The data is available free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html.

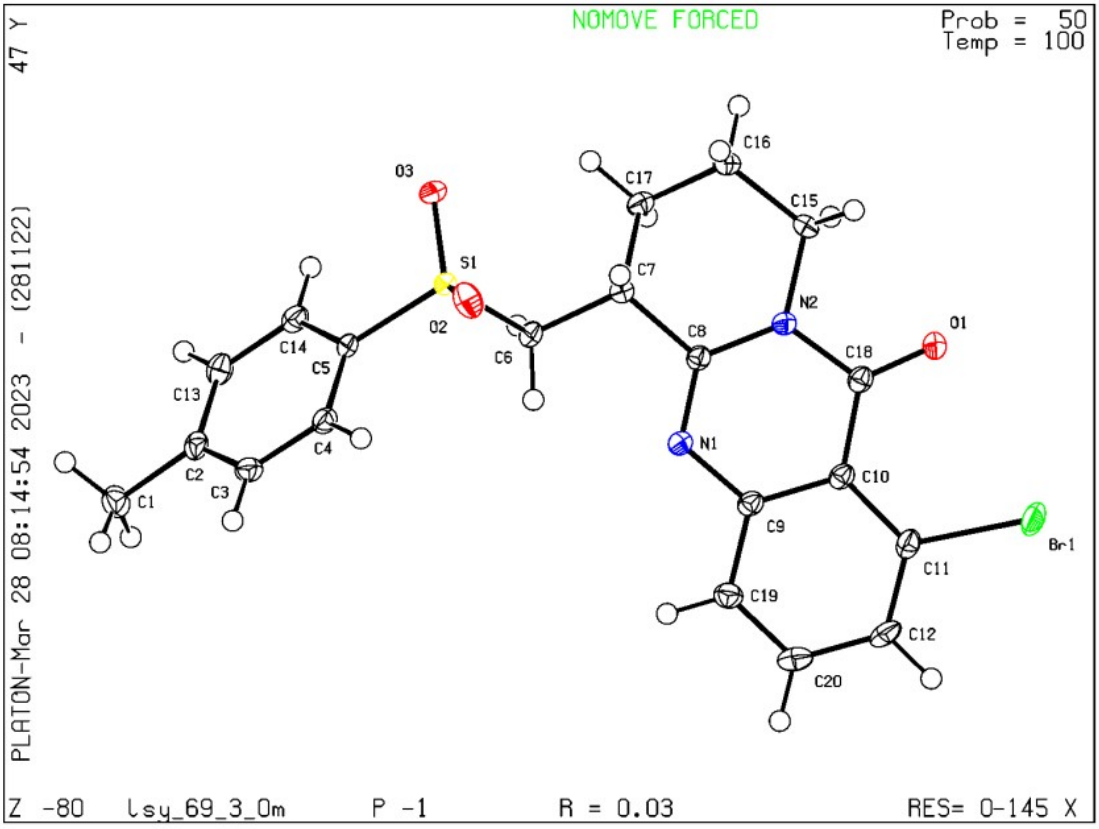


Table 1 Crystal data and structure refinement for 3m.

Identification code	3m
Empirical formula	$C_{20}H_{19}N_2O_3SBr$
Formula weight	447.34
Temperature/K	100.0(2)
Crystal system	triclinic
Space group	P-1
a/Å	4.9018(2)
b/Å	10.5491(5)
c/Å	18.3738(8)
α/°	105.2170(10)
β/°	97.2870(10)
γ/°	93.6630(10)
Volume/Å^3	904.69(7)
Z	2
ρ_{calc}g/cm^3	1.642
μ/mm^{-1}	4.386
F(000)	456.0
Crystal size/mm^3	0.38 × 0.35 × 0.29
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	5.04 to 136.424
Index ranges	$-5 \le h \le 5$, $-12 \le k \le 12$, $-22 \le l \le 22$
Reflections collected	13651
Independent reflections	3269 [R_{int} = 0.0382, R_{sigma} = 0.0317]
Data/restraints/parameters	3269/0/245
Goodness-of-fit on F^2	1.101
Final R indexes [I>=2σ (I)]	R_1 = 0.0332, wR_2 = 0.0884
Final R indexes [all data]	R_1 = 0.0335, wR_2 = 0.0885
Largest diff. peak/hole / e Å^{-3}	0.81/-0.53

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 3m. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br(1)	4059.0(6)	13299.3(2)	9284.0(2)	25.83(12)
S(1)	5558.5(10)	4459.4(5)	6614.5(3)	13.35(14)
O(1)	6675(4)	10936.5(16)	9507.1(9)	22.4(4)
O(2)	7271(3)	5165.6(17)	6236.5(9)	22.3(4)
O(3)	6856(3)	3660.1(16)	7057.2(9)	21.5(4)
N(1)	2856(4)	8263.7(18)	7574.7(10)	14.9(4)
N(2)	6183(4)	8891.3(18)	8680.9(10)	13.7(4)
C(1)	-3806(5)	1135(2)	4308.1(14)	22.1(5)
C(2)	-1407(4)	1936(2)	4869.5(13)	16.2(4)
C(3)	-16(5)	3011(2)	4723.7(13)	16.9(5)
C(4)	2126(5)	3786(2)	5249.1(13)	15.2(4)
C(5)	2908(4)	3467(2)	5926.7(12)	13.0(4)
C(6)	3803(5)	5645(2)	7214.4(13)	16.0(4)
C(7)	5934(4)	6650(2)	7802.5(12)	15.3(4)
C(8)	4880(4)	8002(2)	8022.2(12)	13.5(4)
C(9)	1980(5)	9511(2)	7759.9(12)	14.6(4)
C(10)	3297(4)	10534(2)	8393.0(12)	14.2(4)
C(11)	2331(5)	11791(2)	8506.2(13)	16.8(4)
C(12)	91(5)	12008(2)	8033.3(13)	19.4(5)
C(13)	-557(5)	1627(2)	5550.2(14)	19.0(5)
C(14)	1585(5)	2381(2)	6080.6(13)	16.7(4)
C(15)	8246(5)	8564(2)	9255.0(13)	17.2(5)
C(16)	9237(5)	7209(2)	8995.9(13)	17.9(5)
C(17)	6942(5)	6205(2)	8510.2(13)	17.9(5)
C(18)	5502(4)	10197(2)	8909.5(12)	14.8(4)
C(19)	-299(5)	9747(2)	7280.3(13)	17.6(5)
C(20)	-1235(5)	10974(2)	7426.3(14)	20.2(5)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 3m. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br(1)	42.66(19)	10.18(15)	22.27(17)	2.25(10)	-0.20(12)	4.49(11)
S(1)	14.0(3)	11.6(3)	13.2(3)	1.7(2)	0.19(19)	3.60(19)
O(1)	29.3(9)	14.4(8)	18.4(8)	0.0(7)	-6.0(7)	4.0(7)
O(2)	20.4(8)	26.1(9)	18.0(8)	2.9(7)	3.4(6)	-3.8(7)
O(3)	25.7(9)	16.8(8)	19.5(8)	2.1(7)	-3.7(7)	10.3(7)
N(1)	17.0(9)	12.9(9)	14.5(9)	3.6(7)	1.1(7)	3.4(7)
N(2)	15.8(9)	12.8(9)	12.8(9)	4.1(7)	0.8(7)	2.9(7)
C(1)	17.4(11)	21.1(12)	23.9(12)	1.4(10)	0.3(9)	-0.5(9)
C(2)	14.0(10)	14.2(10)	18.5(11)	-0.5(9)	4.7(8)	3.4(8)
C(3)	19.0(11)	16.9(11)	15.5(11)	5.9(9)	1.4(8)	4.5(9)
C(4)	17.8(11)	12.0(10)	16.3(11)	4.3(8)	2.5(8)	1.9(8)
C(5)	14.2(10)	10.1(10)	13.6(10)	0.7(8)	1.9(8)	3.3(8)
C(6)	16.5(11)	12.8(11)	17.2(11)	0.9(9)	2.0(8)	4.4(8)
C(7)	15.8(10)	13.8(11)	15.2(11)	2.9(9)	-0.1(8)	3.3(8)
C(8)	16.1(10)	12.5(10)	12.4(10)	4.1(8)	2.8(8)	1.8(8)
C(9)	17.5(11)	13.7(11)	13.9(10)	5.4(9)	3.5(8)	3.3(8)
C(10)	17.6(11)	12.6(10)	14.2(10)	5.5(8)	4.2(8)	2.7(8)
C(11)	24.6(11)	11.4(10)	15.0(11)	3.5(8)	4.7(9)	1.9(9)
C(12)	26.7(12)	15.4(11)	21.0(12)	10.0(9)	7.7(9)	9.6(9)
C(13)	22.1(11)	14.1(11)	21.2(12)	4.9(9)	5.3(9)	-0.2(9)
C(14)	21.4(11)	13.9(11)	16.3(11)	5.5(9)	4.8(9)	4.1(9)
C(15)	19.2(11)	16.5(11)	13.8(11)	3.2(9)	-4.3(8)	2.7(9)
C(16)	19.4(11)	18.2(11)	15.4(11)	4.0(9)	-1.0(9)	6.5(9)
C(17)	22.7(11)	14.3(11)	17.4(11)	5.5(9)	0.5(9)	6.1(9)
C(18)	17.1(11)	12.8(11)	15.4(11)	4.9(9)	3.7(8)	1.3(8)
C(19)	18.0(11)	18.8(11)	16.8(11)	6.9(9)	0.5(9)	2.5(9)
C(20)	20.2(11)	24.5(12)	21.1(12)	12.9(10)	4.6(9)	8.7(9)

Table 4 Bond Lengths for 3m.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br(1)	C(11)	1.902(2)	C(4)	C(5)	1.388(3)
S(1)	O(2)	1.4429(17)	C(5)	C(14)	1.391(3)
S(1)	O(3)	1.4402(17)	C(6)	C(7)	1.534(3)
S(1)	C(5)	1.760(2)	C(7)	C(8)	1.519(3)
S(1)	C(6)	1.782(2)	C(7)	C(17)	1.531(3)
O(1)	C(18)	1.216(3)	C(9)	C(10)	1.413(3)
N(1)	C(8)	1.297(3)	C(9)	C(19)	1.409(3)
N(1)	C(9)	1.381(3)	C(10)	C(11)	1.409(3)
N(2)	C(8)	1.372(3)	C(10)	C(18)	1.470(3)
N(2)	C(15)	1.490(3)	C(11)	C(12)	1.382(3)
N(2)	C(18)	1.405(3)	C(12)	C(20)	1.394(4)
C(1)	C(2)	1.506(3)	C(13)	C(14)	1.383(3)
C(2)	C(3)	1.389(3)	C(15)	C(16)	1.515(3)
C(2)	C(13)	1.394(3)	C(16)	C(17)	1.510(3)
C(3)	C(4)	1.386(3)	C(19)	C(20)	1.373(3)

Table 5 Bond Angles for 3m.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O(2)	S(1)	C(5)	108.33(10)	N(1)	C(8)	C(7)	118.76(19)
O(2)	S(1)	C(6)	107.25(11)	N(2)	C(8)	C(7)	117.51(18)
O(3)	S(1)	O(2)	118.39(11)	N(1)	C(9)	C(10)	122.6(2)
O(3)	S(1)	C(5)	108.84(10)	N(1)	C(9)	C(19)	117.2(2)
O(3)	S(1)	C(6)	108.77(10)	C(19)	C(9)	C(10)	120.2(2)
C(5)	S(1)	C(6)	104.36(10)	C(9)	C(10)	C(18)	117.55(19)
C(8)	N(1)	C(9)	118.60(19)	C(11)	C(10)	C(9)	118.0(2)
C(8)	N(2)	C(15)	124.60(18)	C(11)	C(10)	C(18)	124.4(2)
C(8)	N(2)	C(18)	122.16(18)	C(10)	C(11)	Br(1)	123.05(17)
C(18)	N(2)	C(15)	113.04(18)	C(12)	C(11)	Br(1)	115.66(17)
C(3)	C(2)	C(1)	120.6(2)	C(12)	C(11)	C(10)	121.3(2)
C(3)	C(2)	C(13)	118.6(2)	C(11)	C(12)	C(20)	119.8(2)
C(13)	C(2)	C(1)	120.8(2)	C(14)	C(13)	C(2)	121.3(2)
C(4)	C(3)	C(2)	121.1(2)	C(13)	C(14)	C(5)	118.9(2)
C(3)	C(4)	C(5)	119.1(2)	N(2)	C(15)	C(16)	114.46(18)
C(4)	C(5)	S(1)	120.06(17)	C(17)	C(16)	C(15)	111.60(18)
C(4)	C(5)	C(14)	120.9(2)	C(16)	C(17)	C(7)	108.31(19)
C(14)	C(5)	S(1)	119.00(17)	O(1)	C(18)	N(2)	119.6(2)
C(7)	C(6)	S(1)	109.32(15)	O(1)	C(18)	C(10)	125.3(2)
C(8)	C(7)	C(6)	111.37(18)	N(2)	C(18)	C(10)	115.09(19)
C(8)	C(7)	C(17)	111.13(18)	C(20)	C(19)	C(9)	120.0(2)
C(17)	C(7)	C(6)	113.93(19)	C(19)	C(20)	C(12)	120.8(2)
N(1)	C(8)	N(2)	123.7(2)				

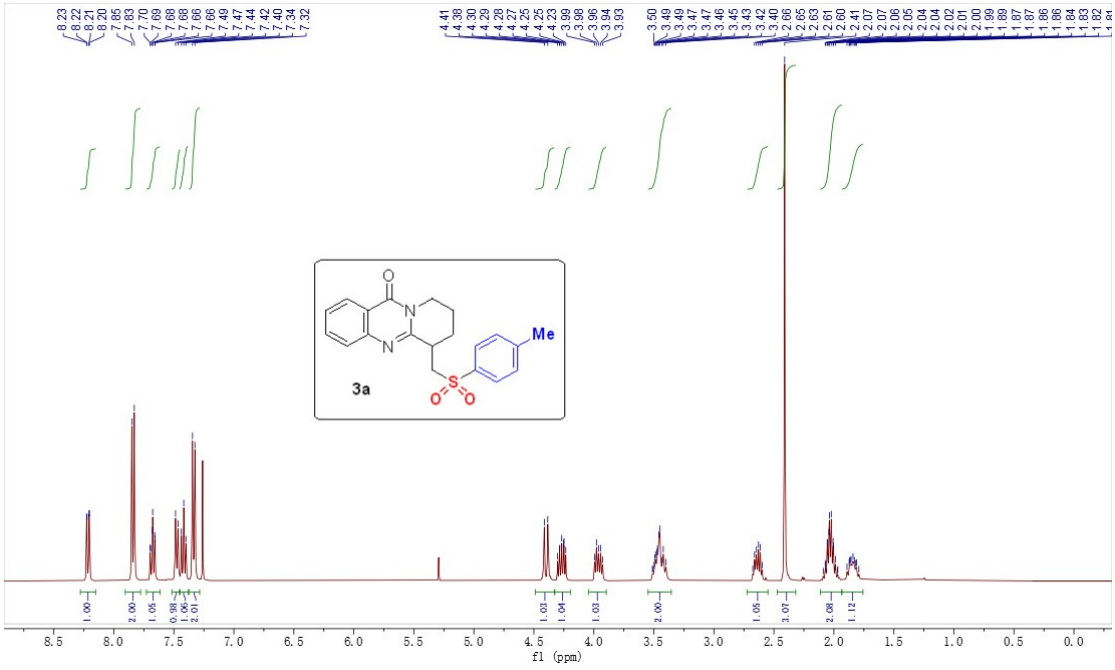
Table 6 Torsion Angles for 3m.

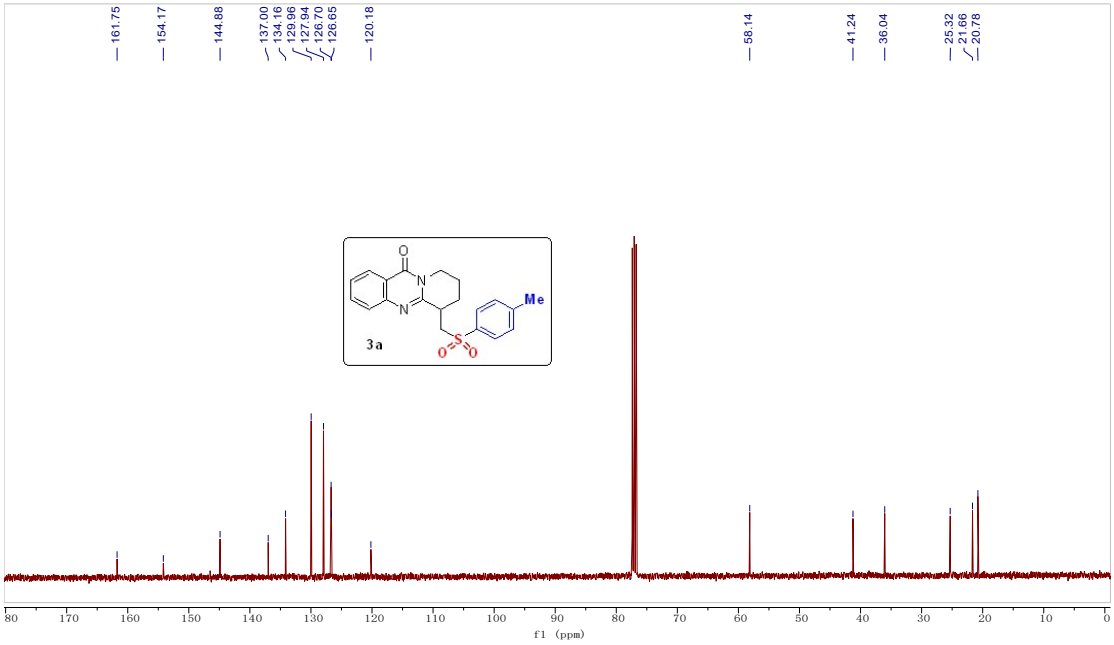
A	B	C	D	Angle/°	A	B	C	D	Angle/°
Br(1)	C(11)	C(12)	C(20)	177.24(17)	C(8)	N(2)	C(18)	O(1)	178.3(2)
S(1)	C(5)	C(14)	C(13)	177.93(17)	C(8)	N(2)	C(18)	C(10)	0.1(3)
S(1)	C(6)	C(7)	C(8)	148.94(15)	C(8)	C(7)	C(17)	C(16)	-59.3(2)
S(1)	C(6)	C(7)	C(17)	-84.4(2)	C(9)	N(1)	C(8)	N(2)	1.1(3)
O(2)	S(1)	C(5)	C(4)	-23.1(2)	C(9)	N(1)	C(8)	C(7)	-177.39(19)
O(2)	S(1)	C(5)	C(14)	158.26(17)	C(9)	C(10)	C(11)	Br(1)	-175.09(16)
O(2)	S(1)	C(6)	C(7)	-60.40(17)	C(9)	C(10)	C(11)	C(12)	2.7(3)
O(3)	S(1)	C(5)	C(4)	-153.04(17)	C(9)	C(10)	C(18)	O(1)	-173.9(2)
O(3)	S(1)	C(5)	C(14)	28.3(2)	C(9)	C(10)	C(18)	N(2)	4.2(3)
O(3)	S(1)	C(6)	C(7)	68.75(17)	C(9)	C(19)	C(20)	C(12)	1.8(3)
N(1)	C(9)	C(10)	C(11)	176.5(2)	C(10)	C(9)	C(19)	C(20)	0.3(3)
N(1)	C(9)	C(10)	C(18)	-6.3(3)	C(10)	C(11)	C(12)	C(20)	-0.7(3)
N(1)	C(9)	C(19)	C(20)	-178.8(2)	C(11)	C(10)	C(18)	O(1)	3.1(4)
N(2)	C(15)	C(16)	C(17)	-36.3(3)	C(11)	C(10)	C(18)	N(2)	-178.8(2)
C(1)	C(2)	C(3)	C(4)	177.5(2)	C(11)	C(12)	C(20)	C(19)	-1.6(3)
C(1)	C(2)	C(13)	C(14)	-177.9(2)	C(13)	C(2)	C(3)	C(4)	-1.7(3)
C(2)	C(3)	C(4)	C(5)	0.9(3)	C(15)	N(2)	C(8)	N(1)	171.4(2)
C(2)	C(13)	C(14)	C(5)	-0.1(3)	C(15)	N(2)	C(8)	C(7)	-10.0(3)
C(3)	C(2)	C(13)	C(14)	1.3(3)	C(15)	N(2)	C(18)	O(1)	3.3(3)
C(3)	C(4)	C(5)	S(1)	-178.31(16)	C(15)	N(2)	C(18)	C(10)	-174.93(18)
C(3)	C(4)	C(5)	C(14)	0.3(3)	C(15)	C(16)	C(17)	C(7)	60.8(2)
C(4)	C(5)	C(14)	C(13)	-0.7(3)	C(17)	C(7)	C(8)	N(1)	-147.1(2)
C(5)	S(1)	C(6)	C(7)	-175.20(15)	C(17)	C(7)	C(8)	N(2)	34.3(3)
C(6)	S(1)	C(5)	C(4)	90.96(19)	C(18)	N(2)	C(8)	N(1)	-3.0(3)
C(6)	S(1)	C(5)	C(14)	-87.71(19)	C(18)	N(2)	C(8)	C(7)	175.56(19)
C(6)	C(7)	C(8)	N(1)	-18.9(3)	C(18)	N(2)	C(15)	C(16)	-174.38(19)
C(6)	C(7)	C(8)	N(2)	162.48(19)	C(18)	C(10)	C(11)	Br(1)	7.9(3)
C(6)	C(7)	C(17)	C(16)	173.88(18)	C(18)	C(10)	C(11)	C(12)	-174.3(2)
C(8)	N(1)	C(9)	C(10)	3.6(3)	C(19)	C(9)	C(10)	C(11)	-2.4(3)
C(8)	N(1)	C(9)	C(19)	-177.4(2)	C(19)	C(9)	C(10)	C(18)	174.74(19)
C(8)	N(2)	C(15)	C(16)	10.7(3)					

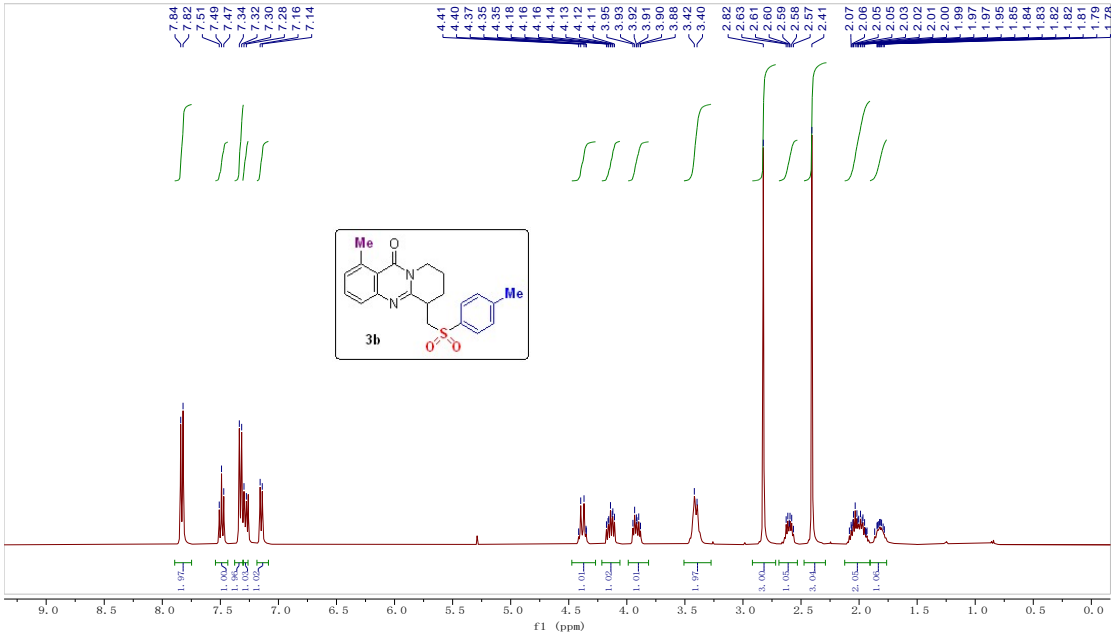
Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 3m.

Atom	*x*	*y*	*z*	U(eq)
H(1A)	-5539.82	1355.1	4501.2	33
H(1B)	-3630.09	192.56	4241.21	33
H(1C)	-3809.68	1335.91	3816.65	33
H(3)	-541.7	3218.95	4256.08	20
H(4)	3046	4526.9	5146.8	18
H(6A)	2649.3	6104.2	6902.9	19
H(6B)	2576.58	5197.08	7478.96	19
H(7)	7585.32	6750.32	7546.4	18
H(12)	-544.2	12859.93	8121.56	23
H(13)	-1468.49	883.52	5651.66	23
H(14)	2143.28	2161.22	6542.83	20
H(15A)	7416.19	8620.13	9725.08	21
H(15B)	9865.17	9235.31	9386.05	21
H(16A)	10772	7260.15	8699.39	21
H(16B)	9953.72	6919.73	9448.34	21
H(17A)	5401.66	6138.32	8802.8	22
H(17B)	7631.27	5325.95	8356.82	22
H(19)	-1186.4	9056.12	6856.23	21
H(20)	-2804.5	11120.9	7110	24

3 Copies of NMR spectra



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O
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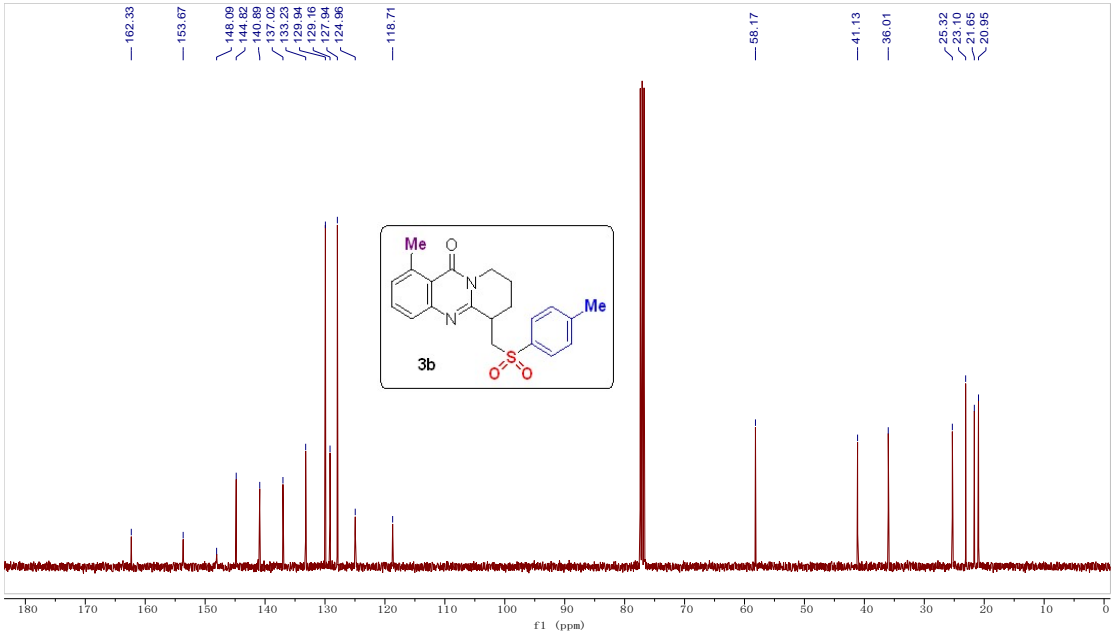
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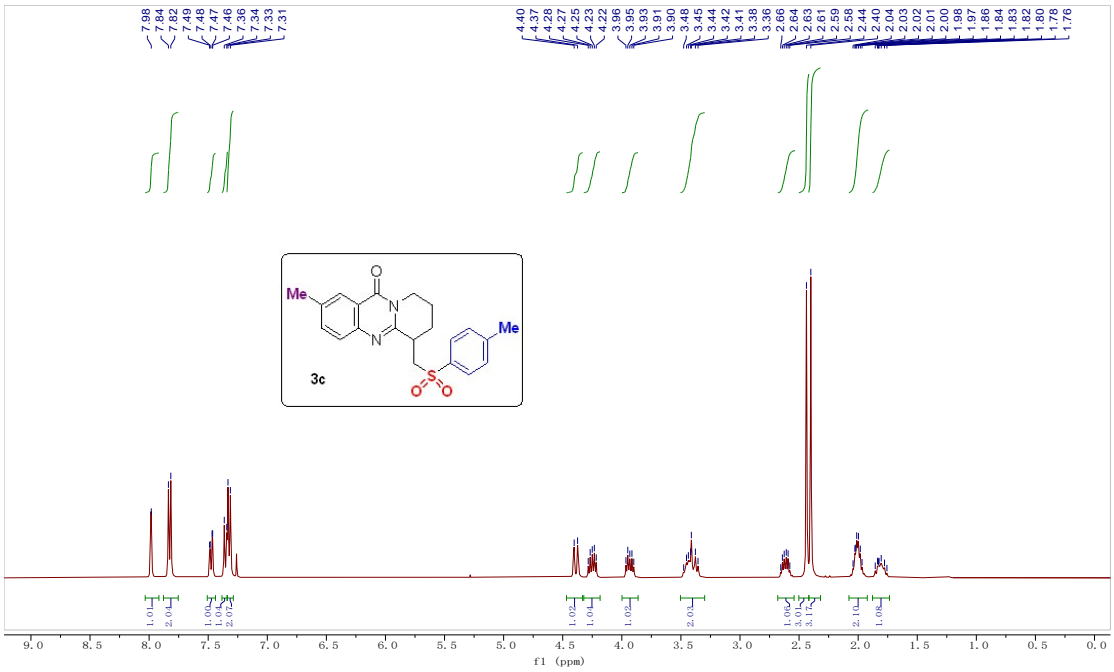
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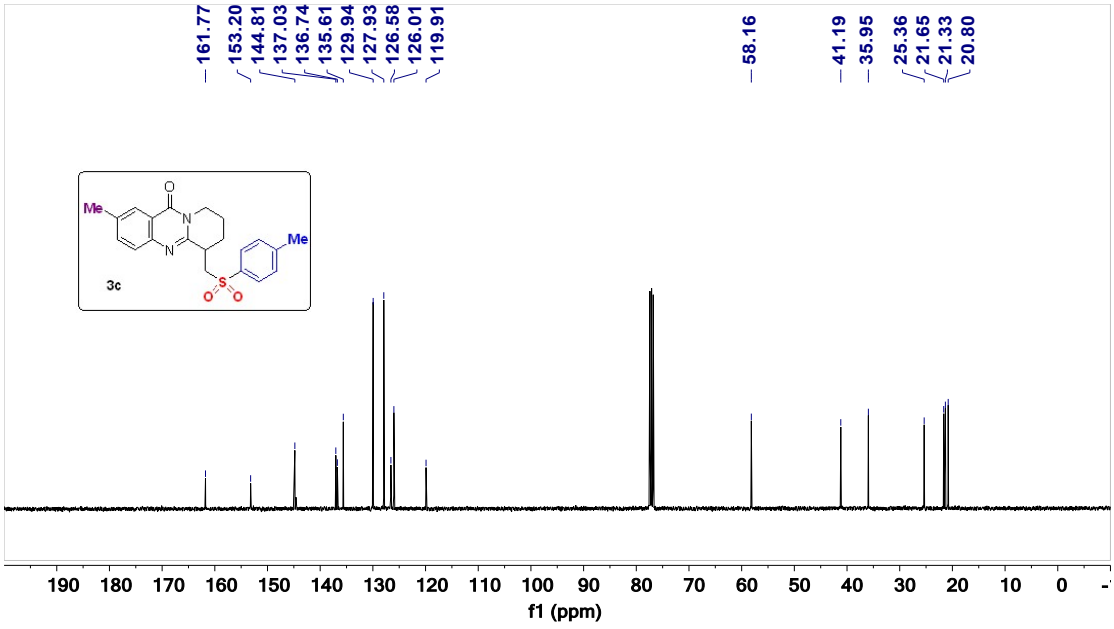
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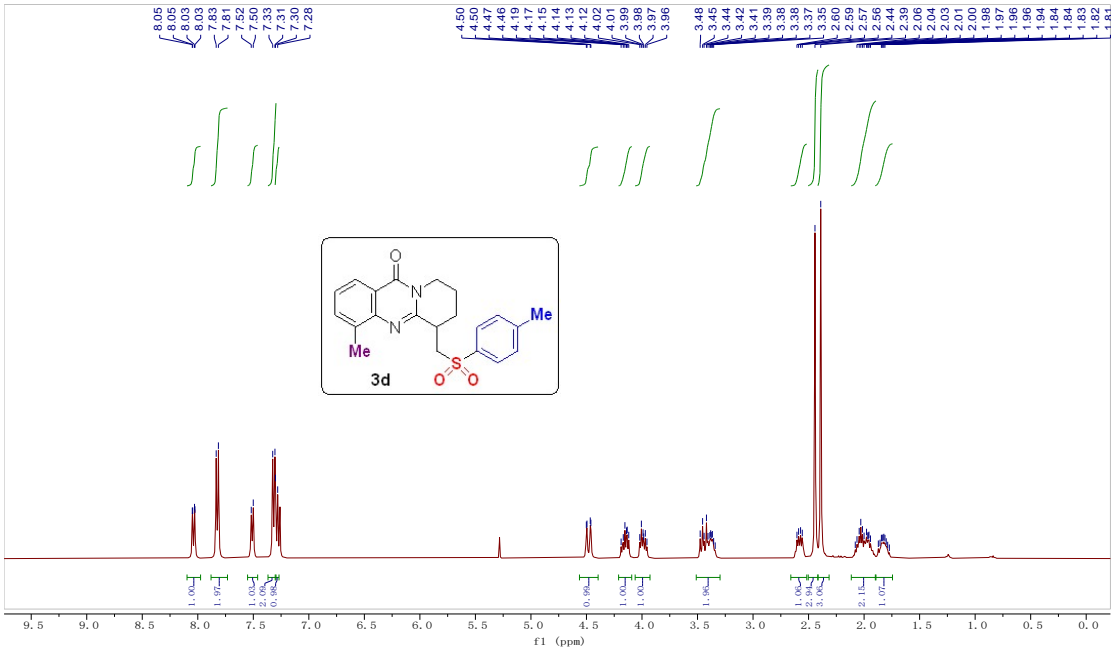
f1 (ppm)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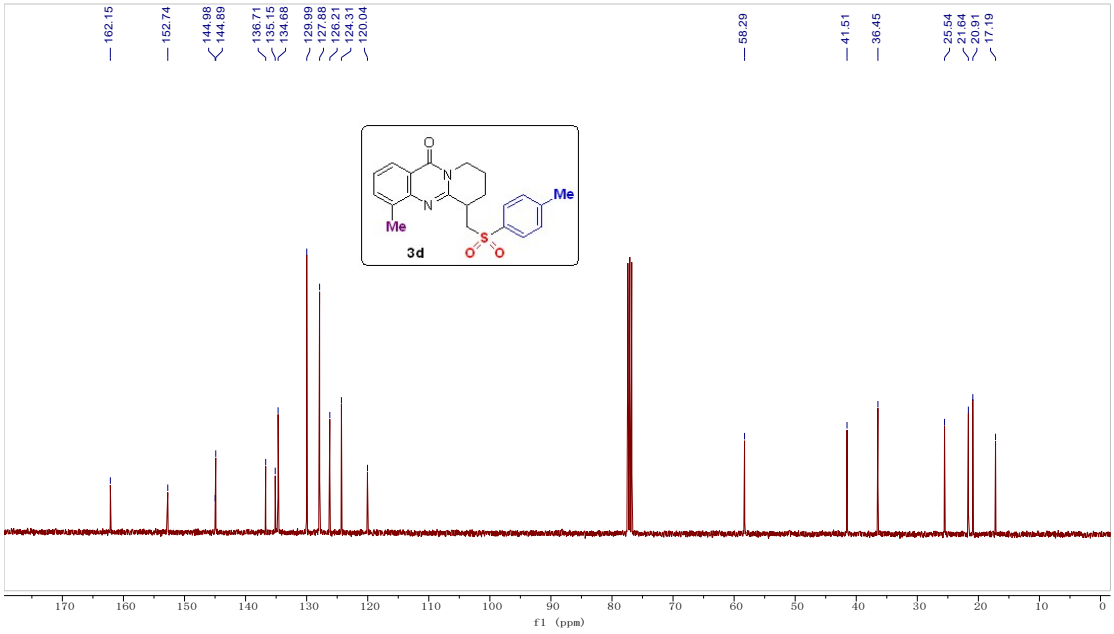
f1 (ppm)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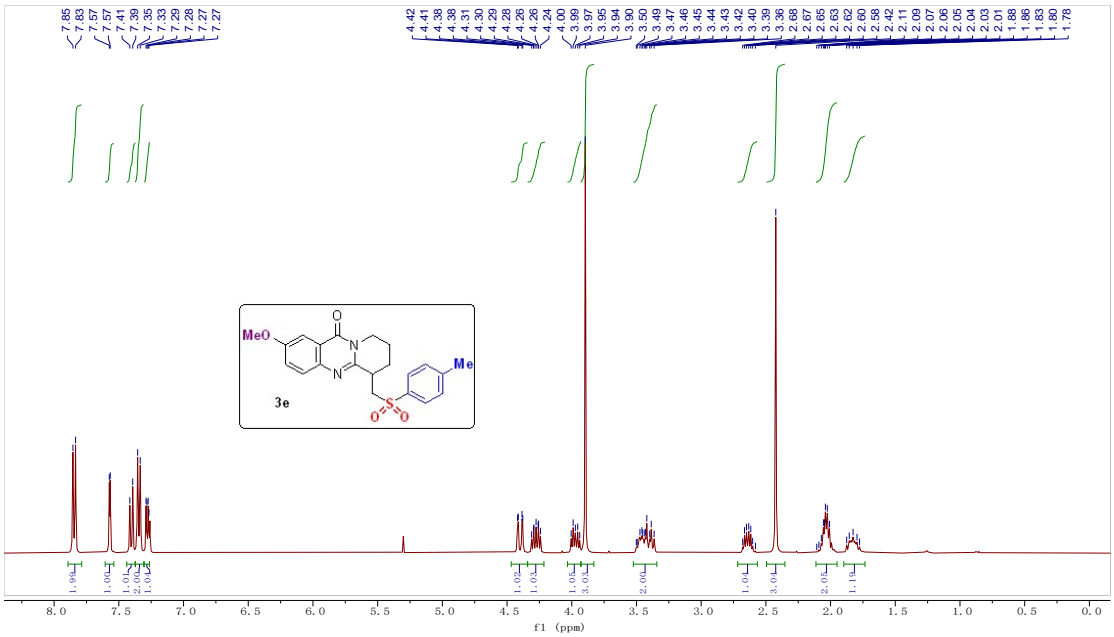
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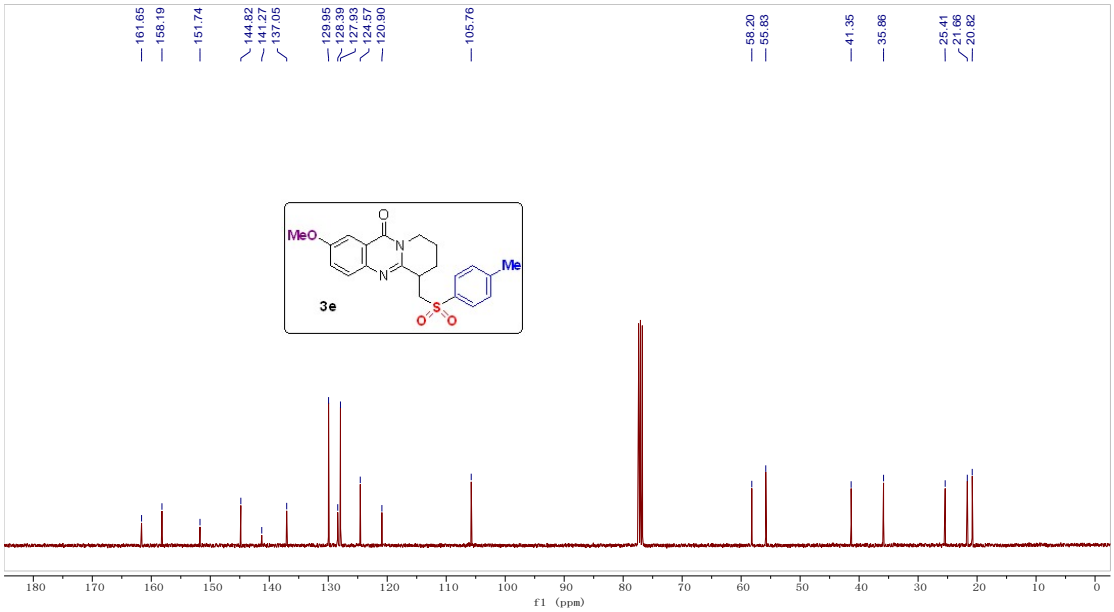
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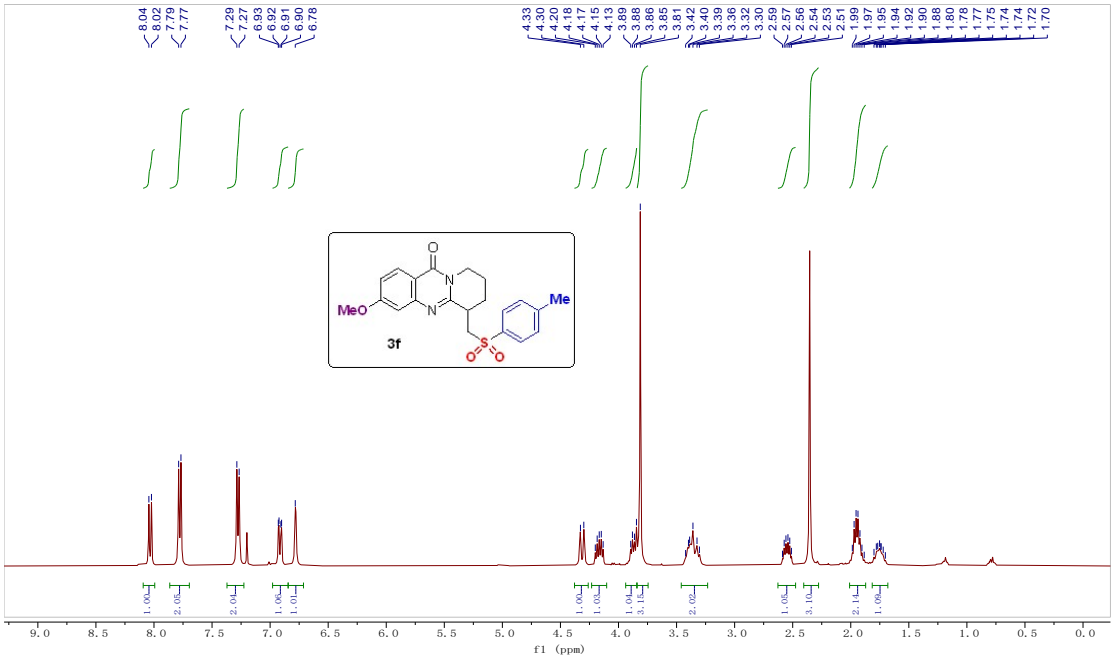
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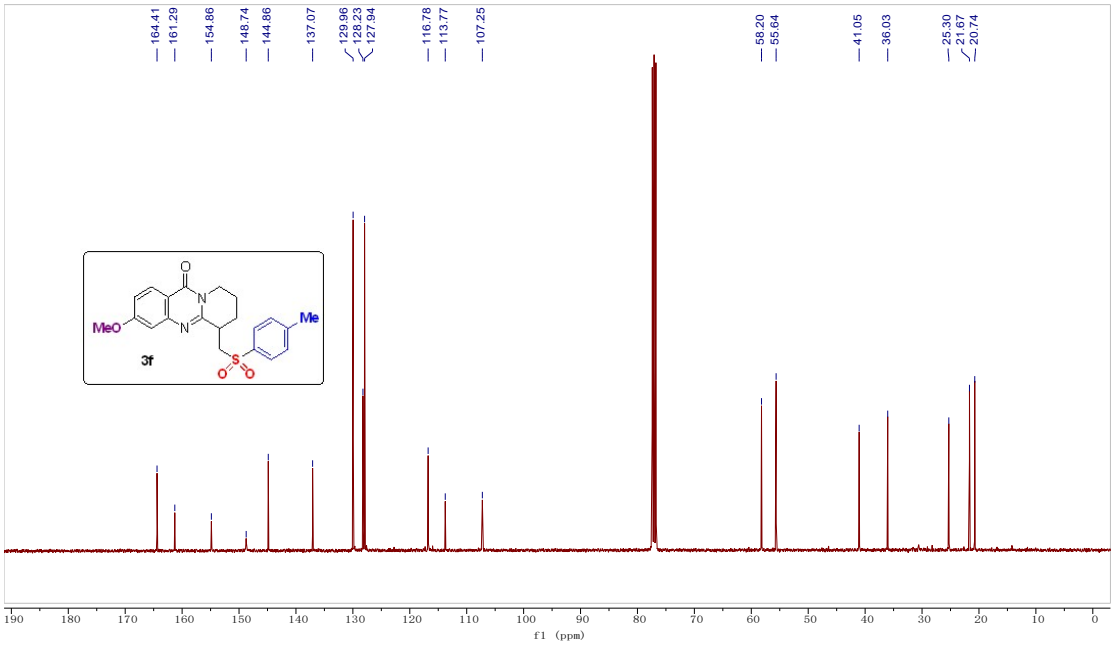
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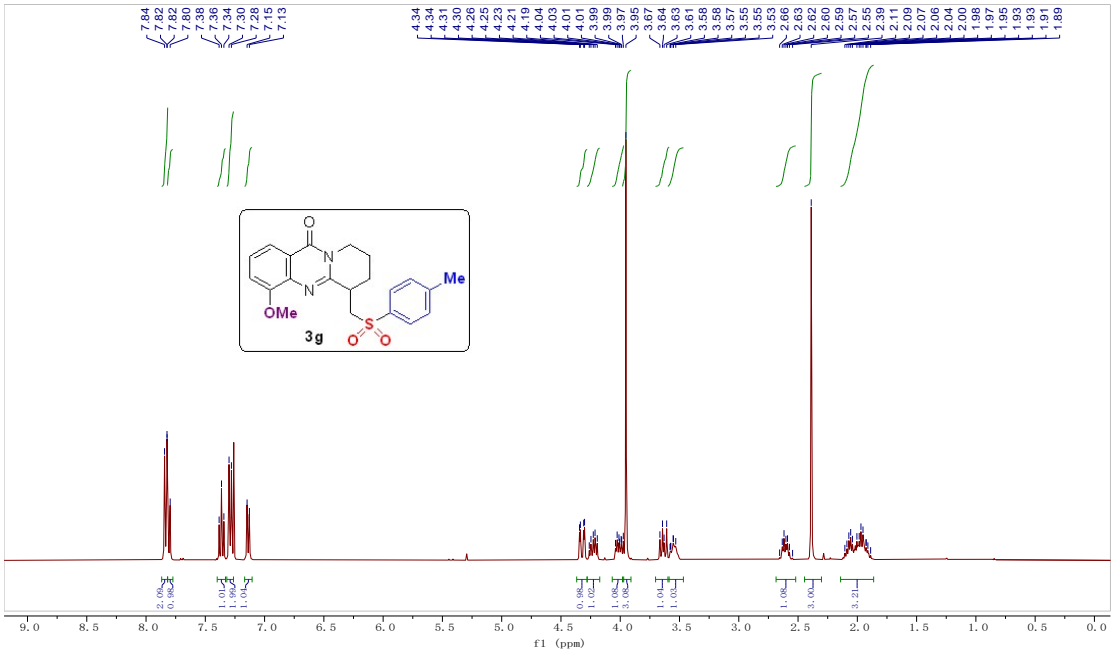
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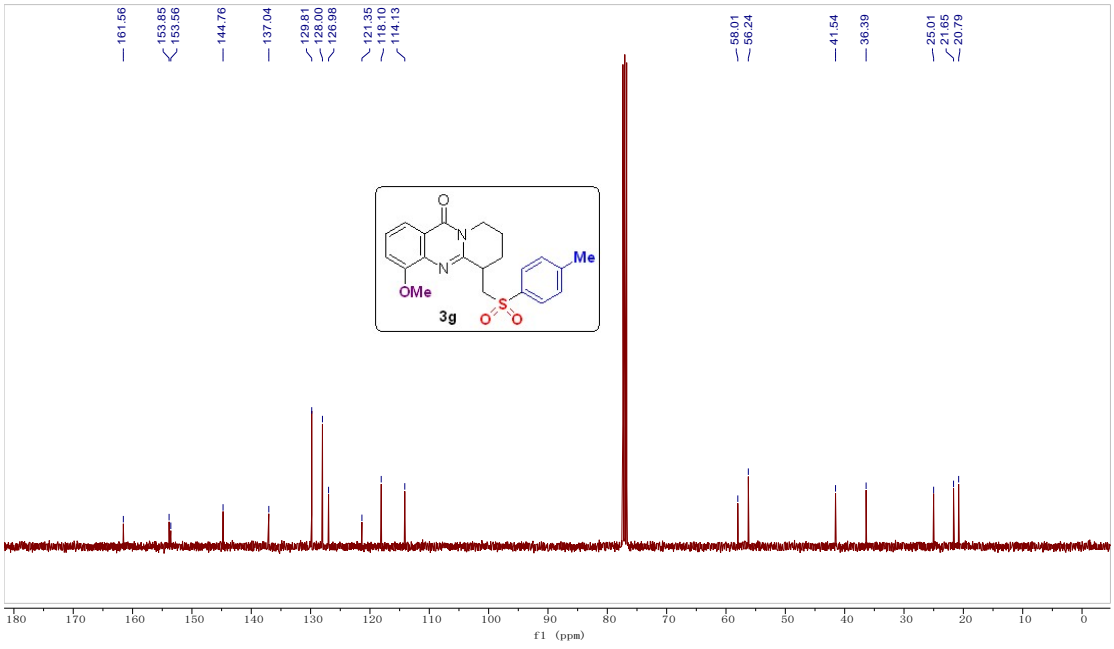
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S
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O
3g
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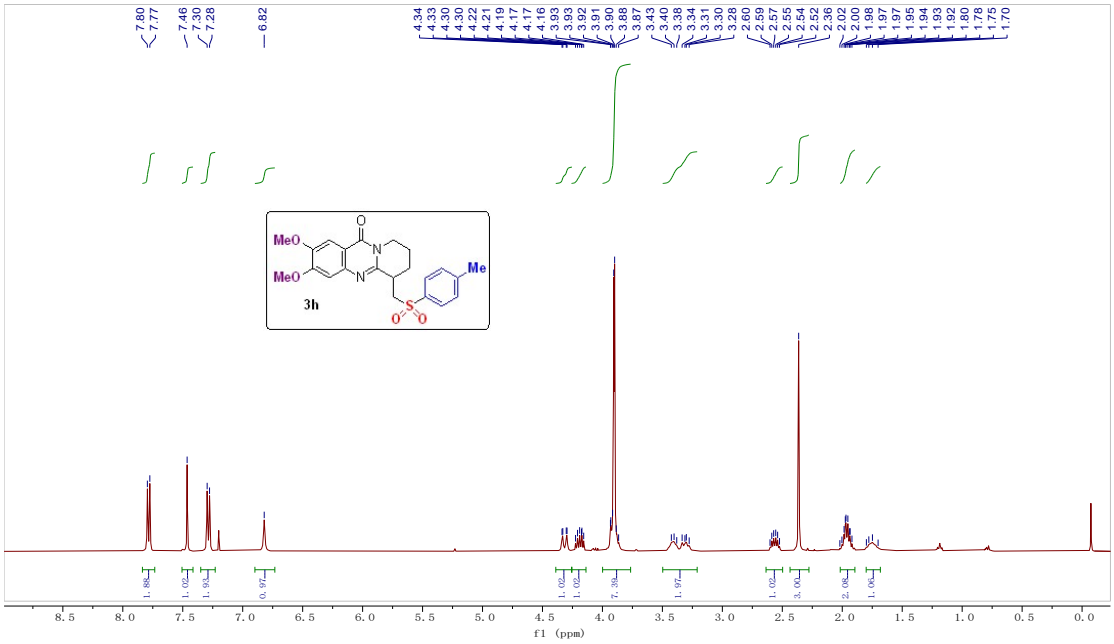
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MeO
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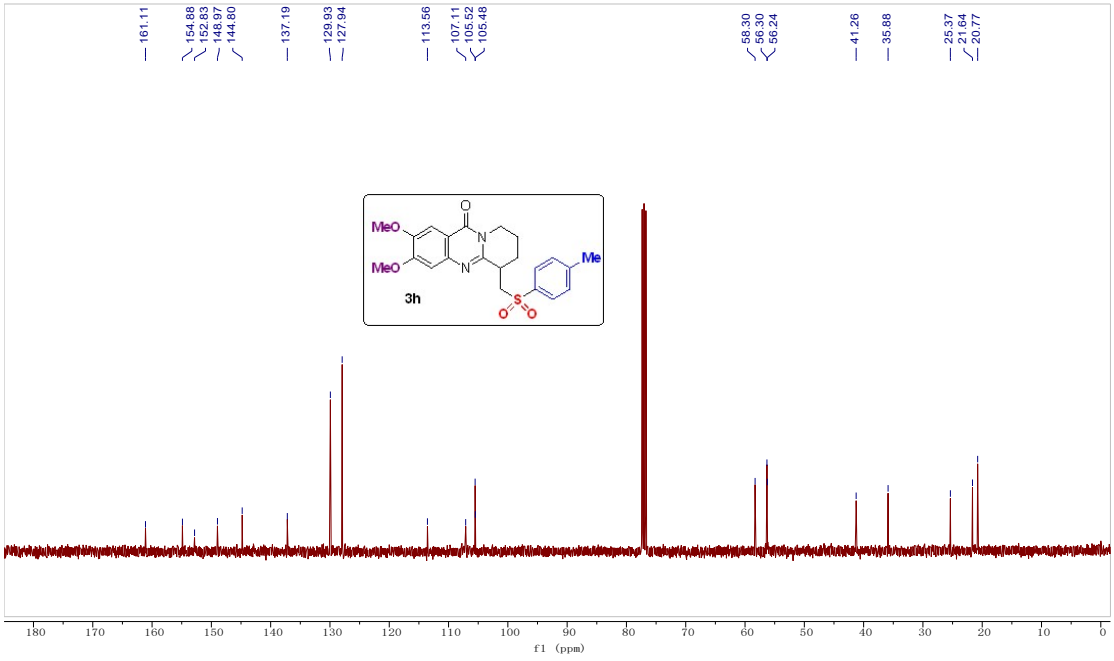
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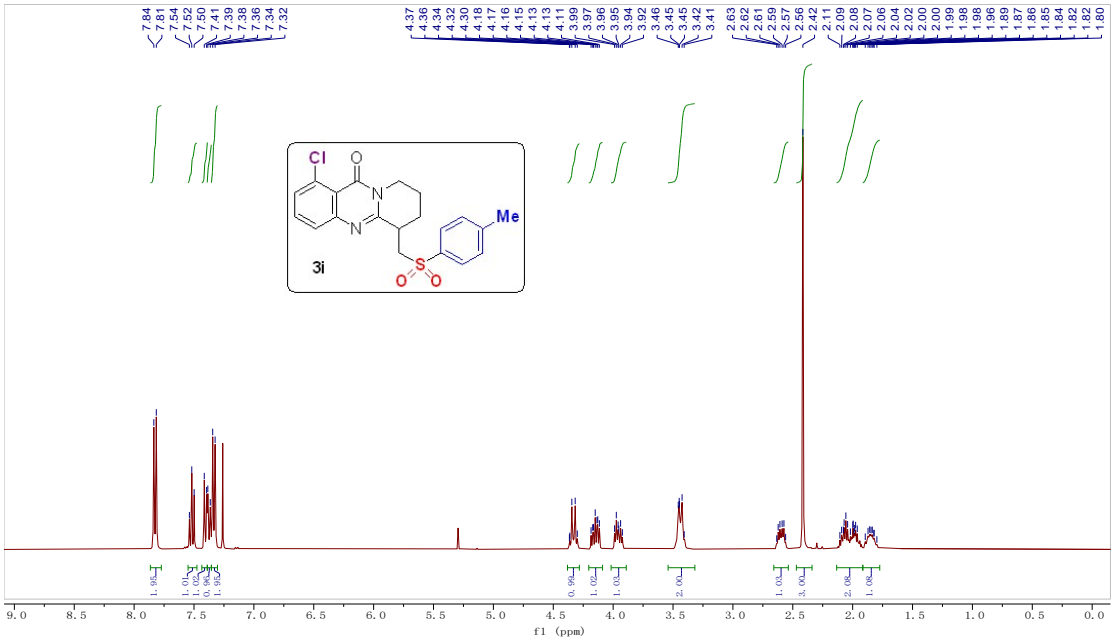
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Cl
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N
Me
S
O
O
3i
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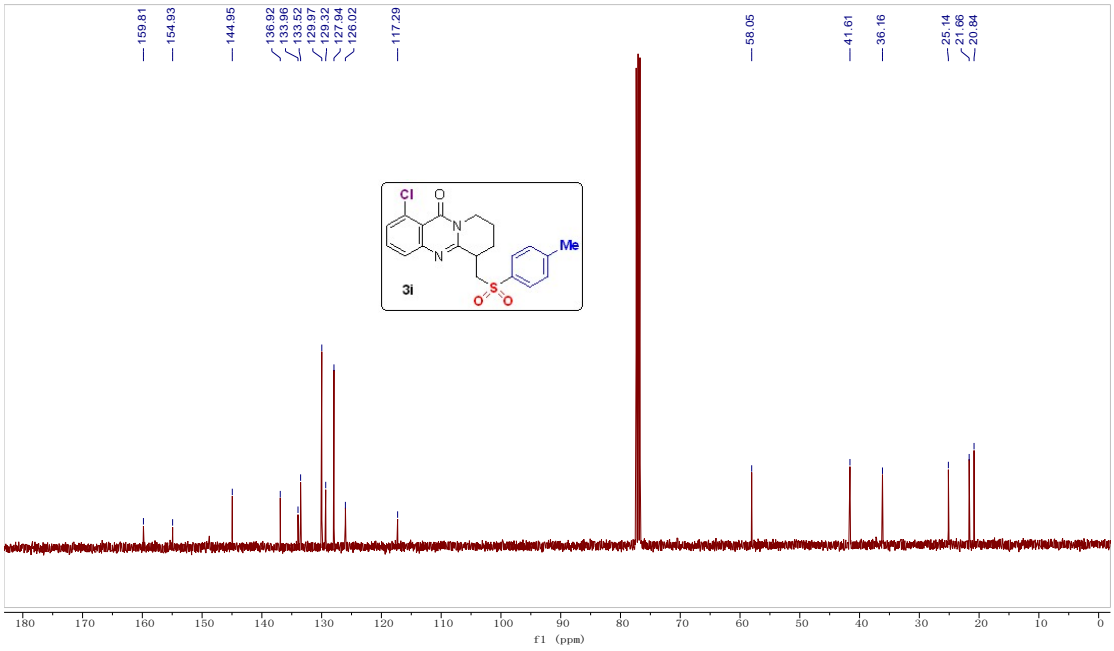
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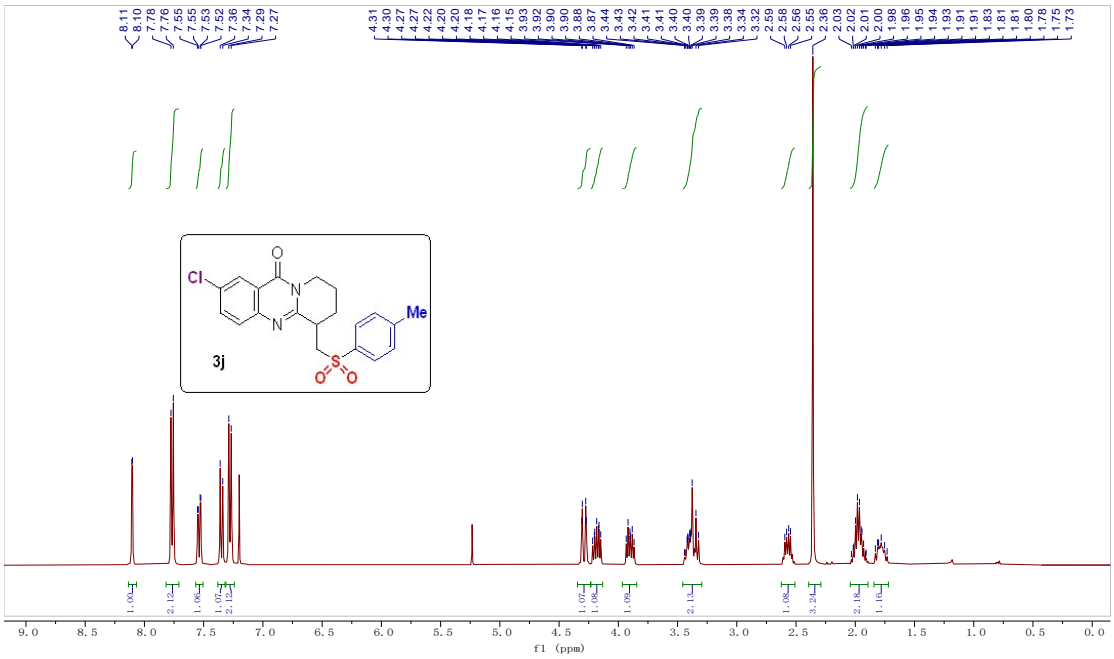
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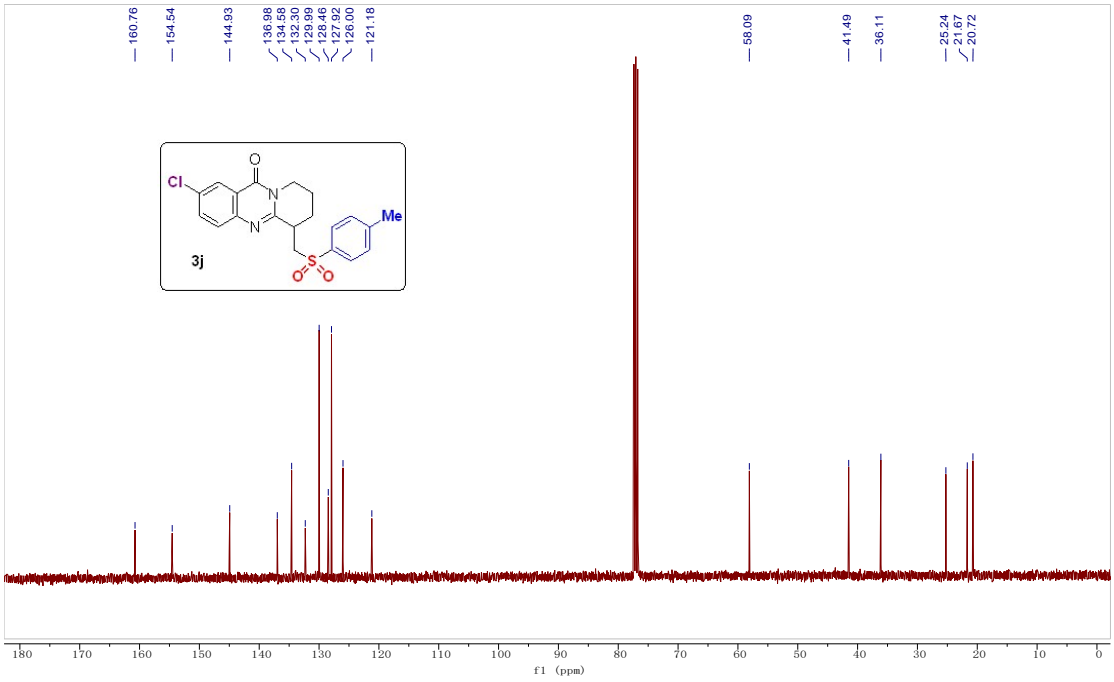
f1 (ppm)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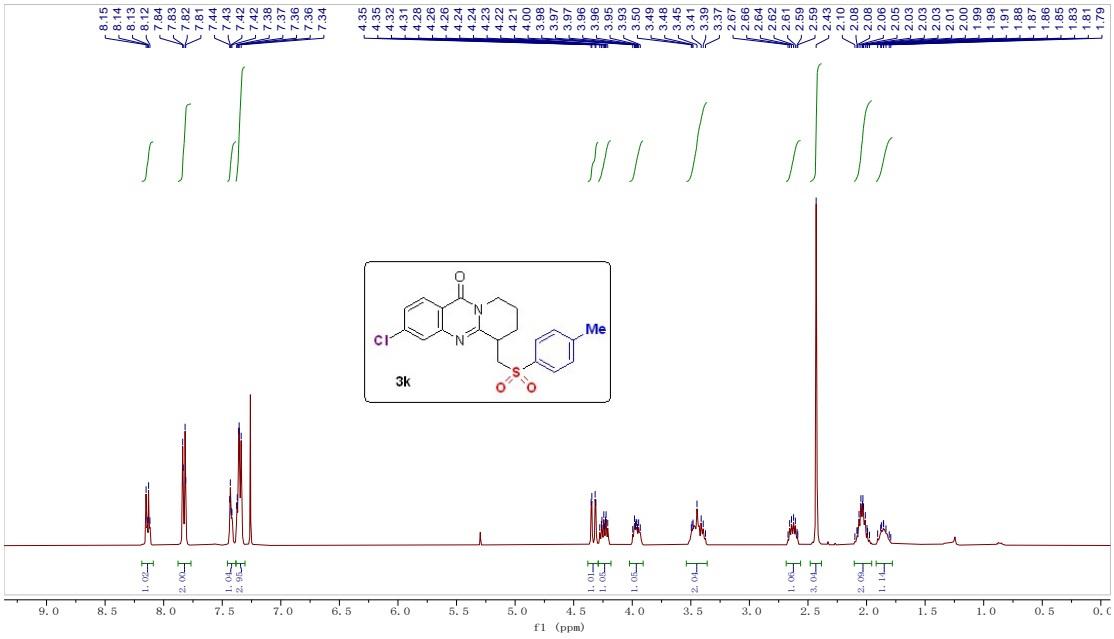
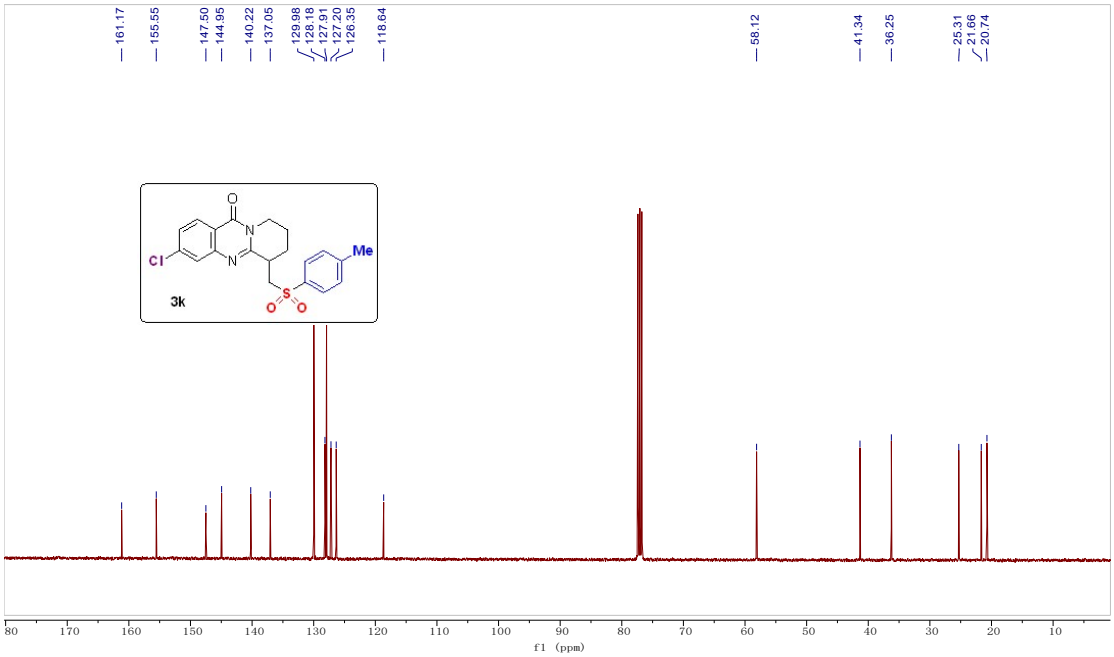
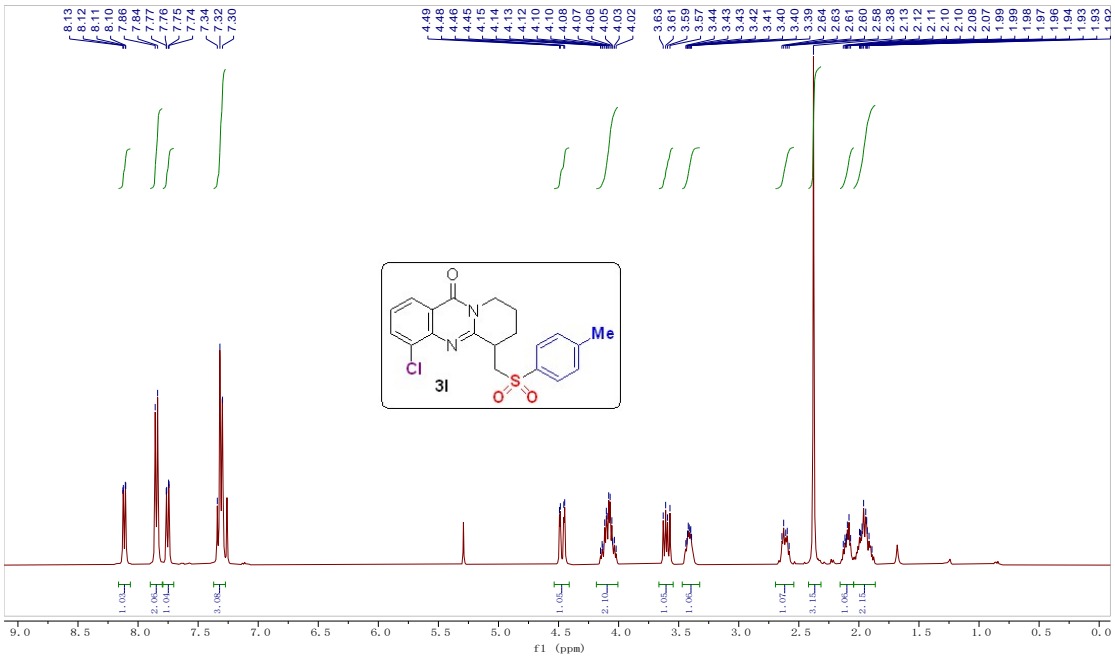
25.24
21.67
20.72
Cl
O
N
N
Me
O=S=O
3j
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.15 8.14 8.13 8.12 7.84 7.83 7.82 7.81 7.44 7.43 7.42 7.42 7.38 7.37 7.36 7.36 7.34
4.35 4.35 4.32 4.31 4.28 4.26 4.26 4.24 4.24 4.23 4.22 4.21 4.00 3.98 3.97 3.97 3.96 3.96 3.95 3.93 3.50 3.49 3.48 3.45 3.41 3.39 3.37 2.67 2.66 2.64 2.62 2.61 2.59 2.43 2.10 2.08 2.08 2.06 2.05 2.03 2.03 2.01 2.00 1.99 1.98 1.91 1.88 1.87 1.86 1.85 1.83 1.81 1.79
Cl
N
O
N
Me
S
O
O
3k
1.02 2.00 1.01 2.05 1.01 1.02 1.02 2.04 1.00 3.04 2.00 1.14
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


161.17 155.55 147.50 144.95 140.22 137.05 129.98 128.18 127.91 127.20 126.35 118.64 58.12 41.34 36.25 25.31 21.66 20.74
Cl
N
O
N
Me
S
O
O
3k
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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8.12
8.11
8.10
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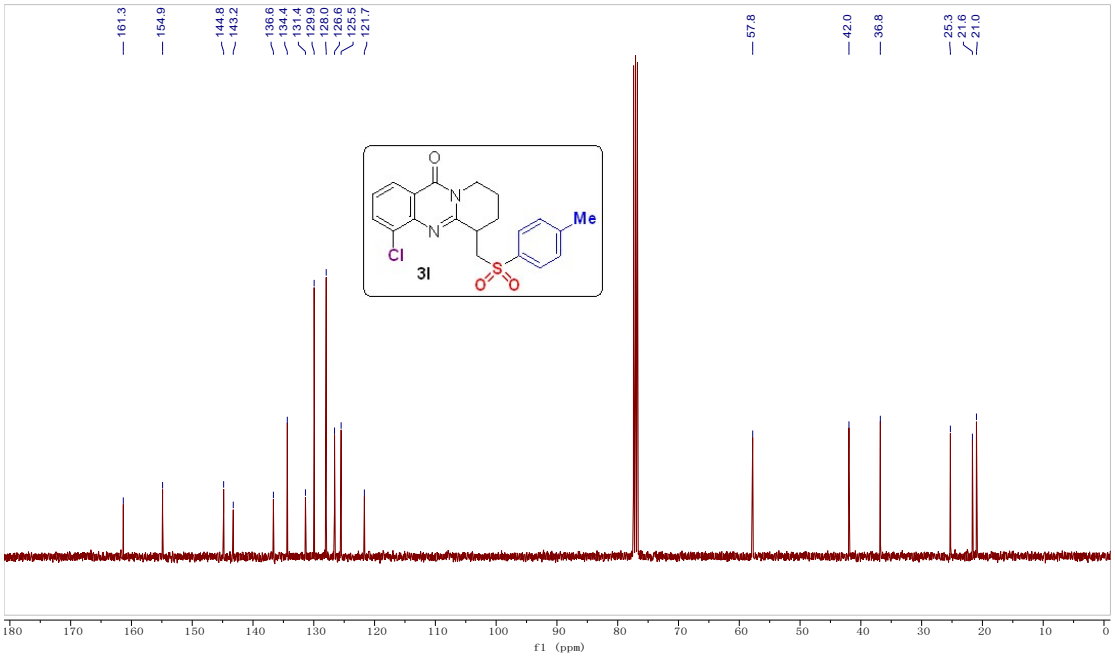
7.84
7.77
7.76
7.75
7.74
7.34
7.32
7.30
4.49
4.48
4.46
4.45
4.15
4.14
4.13
4.12
4.10
4.10
4.08
4.07
4.06
4.05
4.03
4.02
3.63
3.61
3.59
3.57
3.44
3.43
3.43
3.42
3.41
3.40
3.40
3.39
2.64
2.63
2.61
2.60
2.58
2.38
2.13
2.12
2.11
2.10
2.10
2.08
2.07
1.99
1.99
1.98
1.97
1.96
1.94
1.93
1.93
1.92
1.03
2.06
1.01
3.08
1.03
2.10
1.03
1.06
1.07
3.15
1.06
2.15
O
N
N
Cl
Me
S
O
O
3l
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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154.9
144.8
143.2
136.6
134.4
131.4
129.9
128.0
127.6
125.5
121.7
57.8
42.0
36.8
25.3
21.6
21.0
O
N
N
Cl
Me
S
O
O
3l
180
170
160
150
140
130
120
110
100
90
80
70
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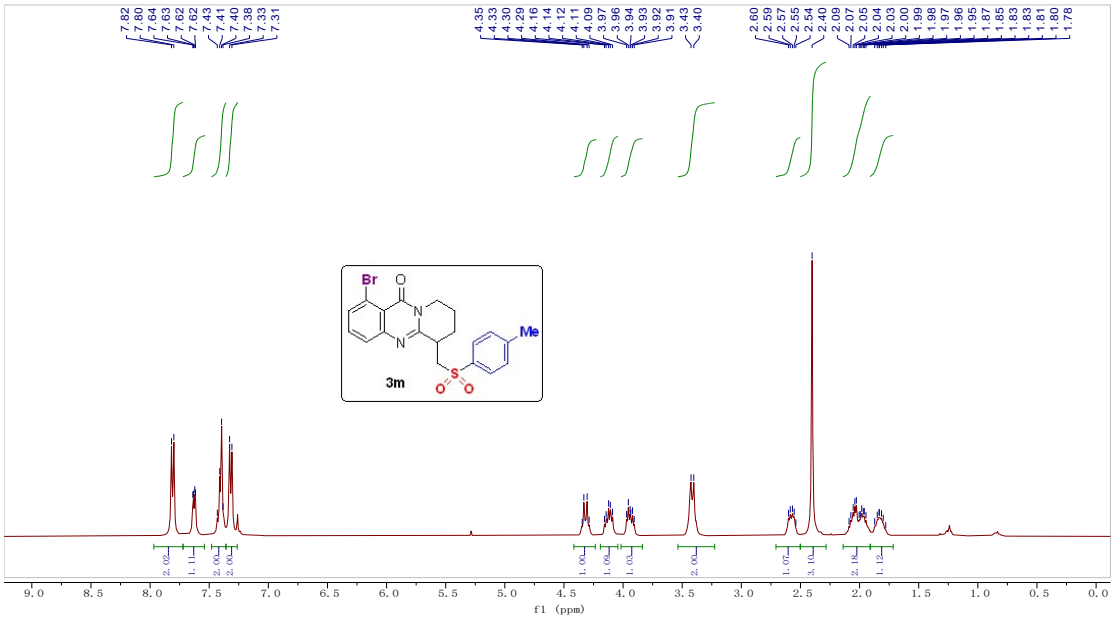
50
40
30
20
10
0
f1 (ppm)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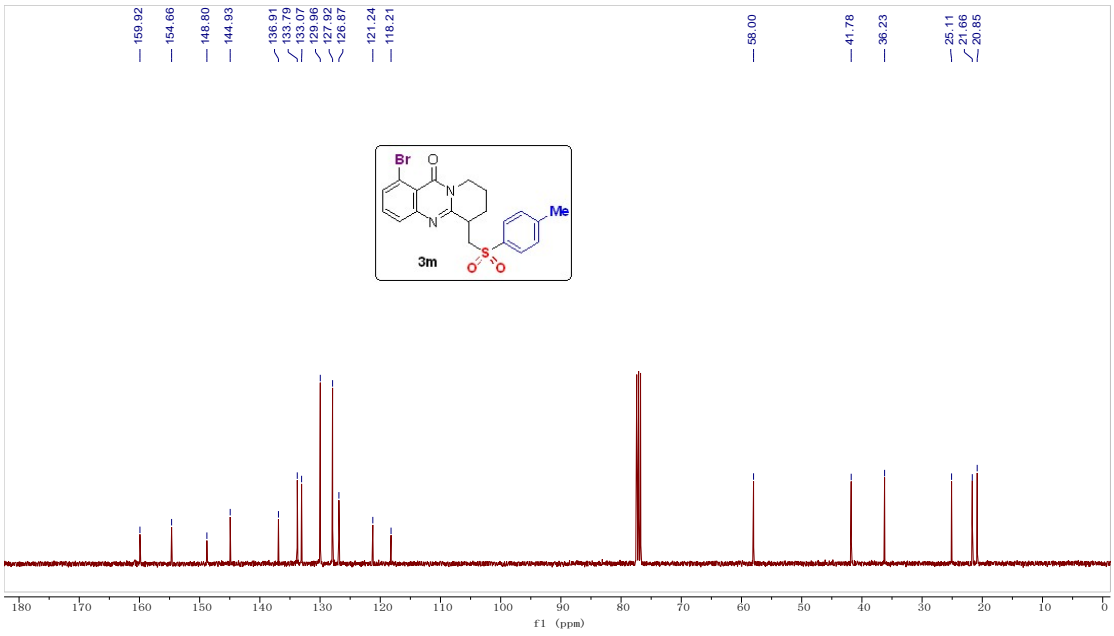
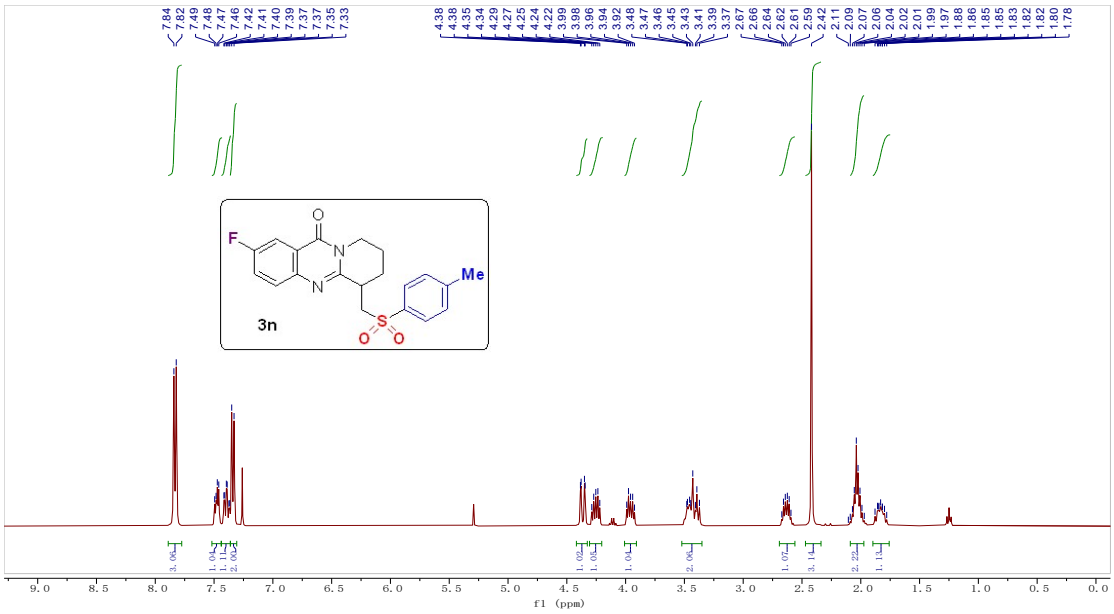
7.80
7.64
7.63
7.62
7.62
7.43
7.41
7.40
7.38
7.33
7.31
4.35
4.33
4.30
4.29
4.16
4.14
4.12
4.11
4.09
3.97
3.96
3.94
3.93
3.92
3.91
3.43
3.40
2.60
2.59
2.57
2.55
2.54
2.40
2.09
2.07
2.05
2.04
2.03
2.00
1.99
1.98
1.97
1.96
1.95
1.87
1.85
1.83
1.83
1.81
1.80
1.78
Br
O
N
N
Me
S
O
O
3m
2.02
1.11
2.00
2.00
1.00
1.00
1.03
2.00
1.07
3.10
2.18
1.12
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


159.92
154.66
148.80
144.93
136.91
133.79
133.07
129.96
127.92
126.87
121.24
118.21
58.00
41.78
36.23
25.11
21.66
20.85
Br
O
N
N
Me
S
O
O
3m
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.49
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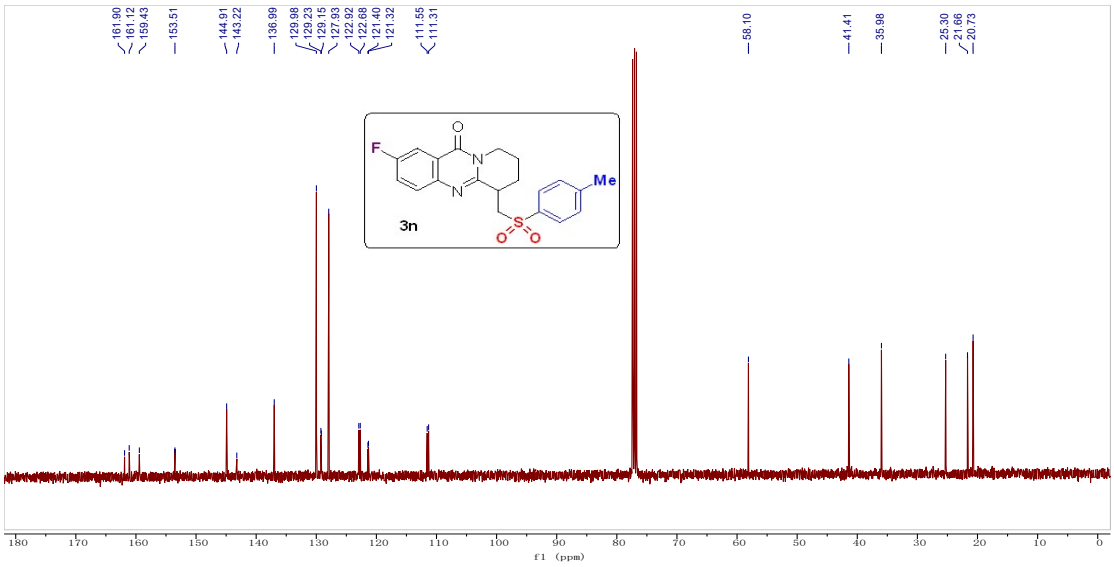
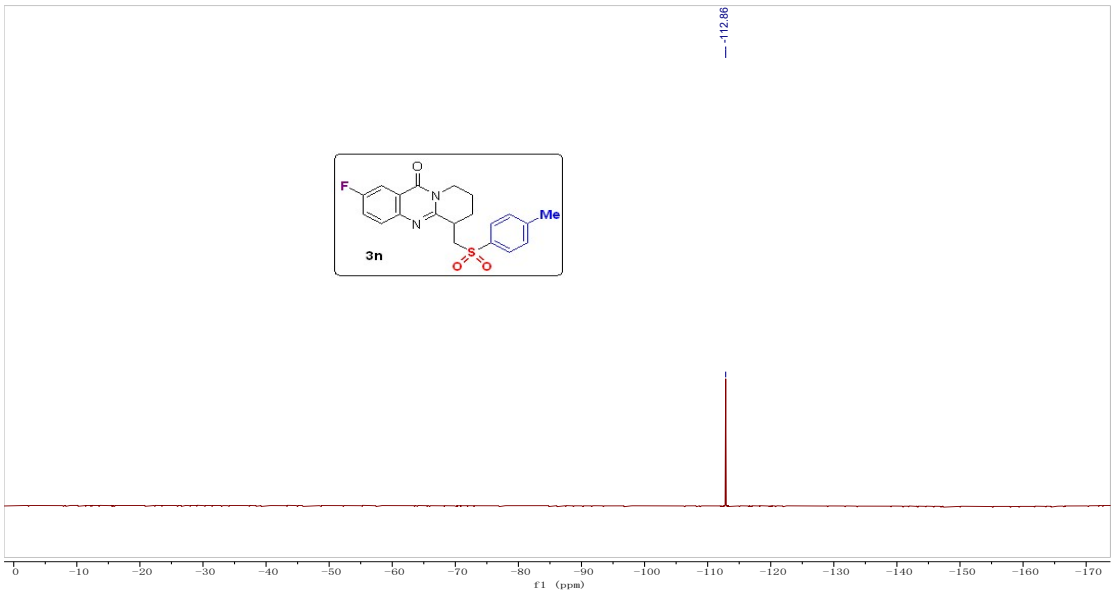
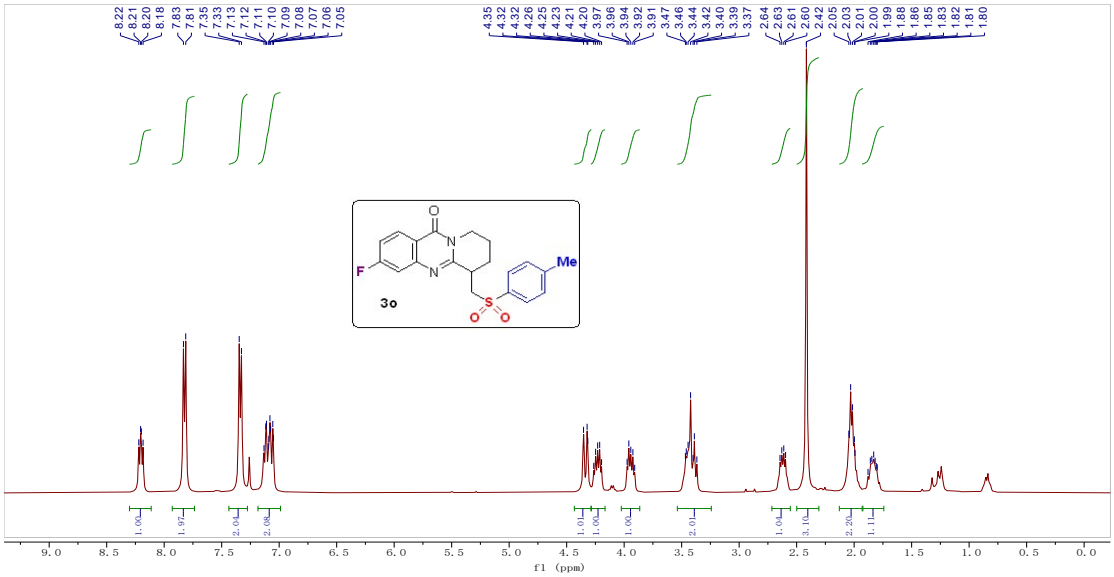
7.47
7.46
7.42
7.41
7.40
7.39
7.37
7.37
7.35
7.33
4.38
4.38
4.35
4.34
4.29
4.27
4.25
4.24
4.22
3.99
3.98
3.96
3.94
3.92
3.48
3.47
3.46
3.45
3.43
3.41
3.39
3.37
2.67
2.66
2.64
2.62
2.61
2.59
2.42
2.11
2.09
2.07
2.06
2.04
2.02
2.01
1.99
1.97
1.88
1.86
1.85
1.85
1.83
1.82
1.82
1.80
1.78
F
O
N
N
Me
S
O
O
3n
3.06
1.04
1.11
2.00
1.02
1.03
1.04
2.08
1.07
3.14
2.22
1.13
f1 (ppm)


161.90
161.12
159.43
153.51
144.91
143.22
136.99
129.98
129.23
129.15
127.93
122.92
122.68
121.40
121.32
111.55
111.31
58.10
41.41
35.98
25.30
21.66
20.73
F
O
N
N
Me
S
O
O
3n
f1 (ppm)


-112.86
F
O
N
N
Me
S
O
O
3n
f1 (ppm)


8.22
8.21
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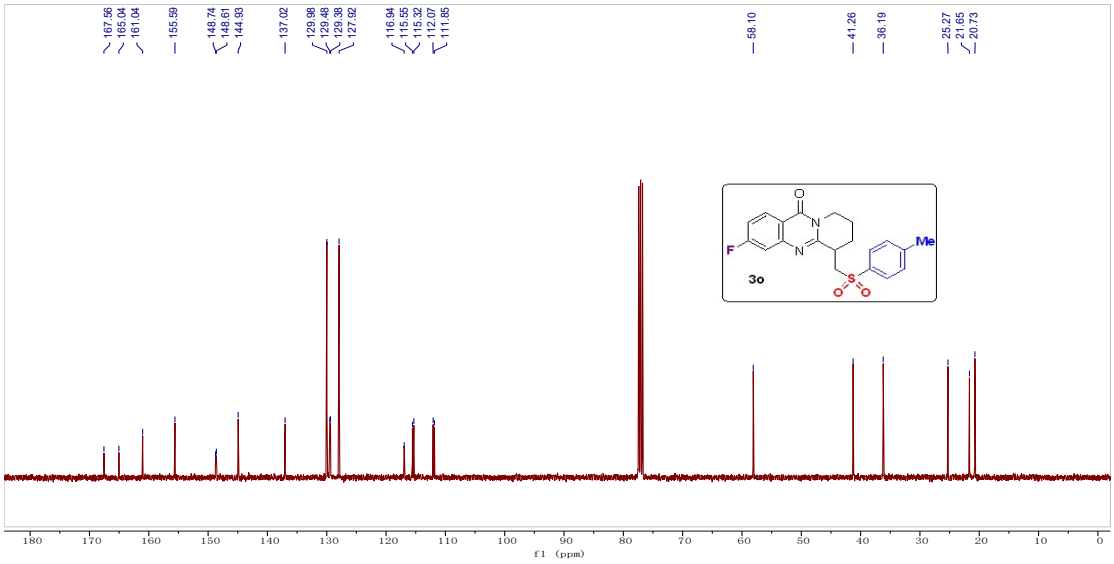
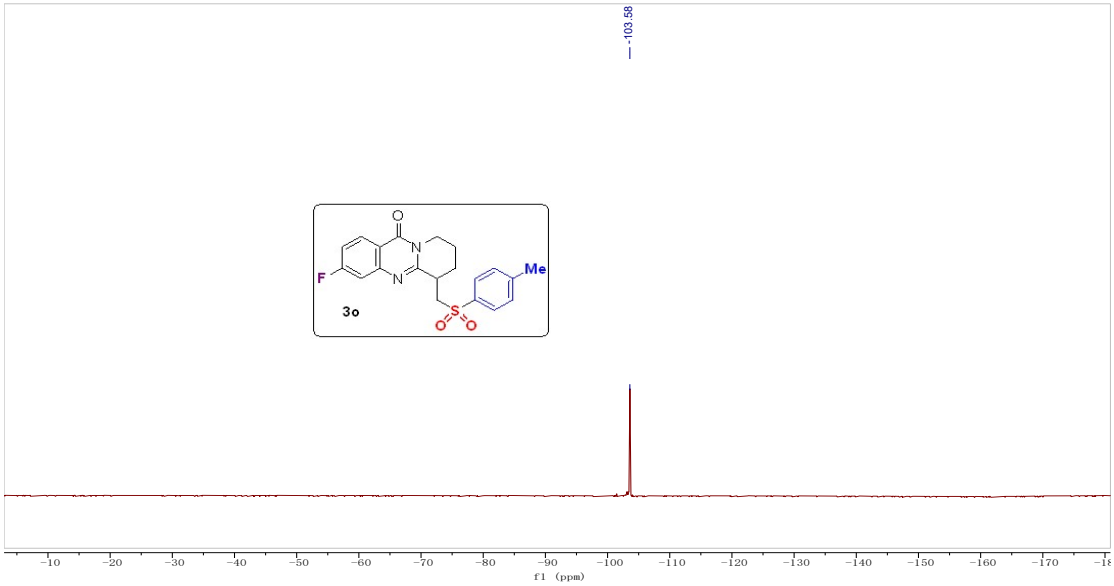
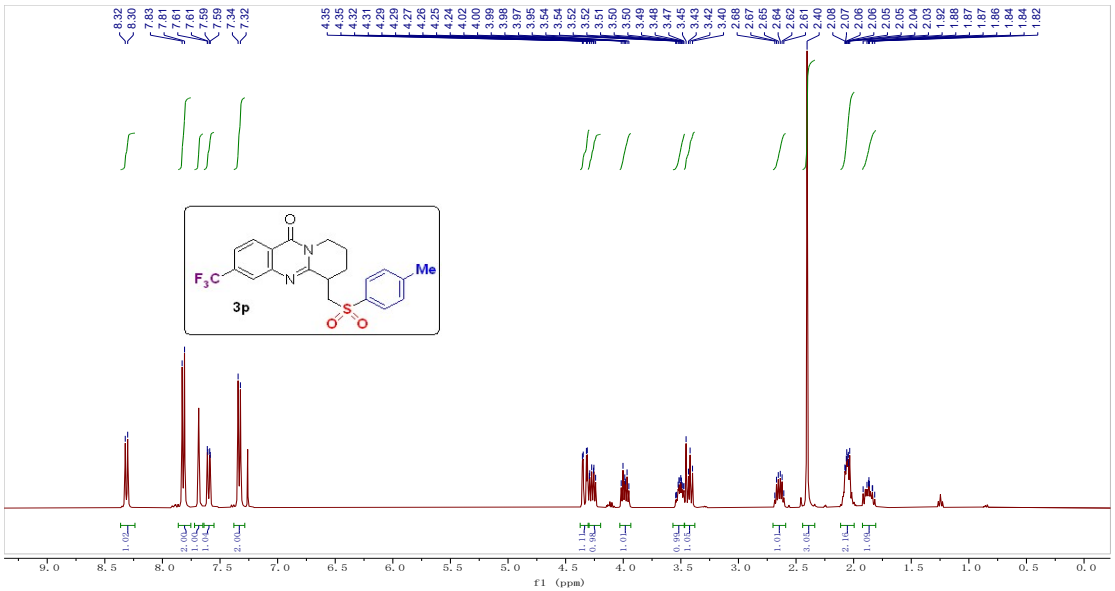
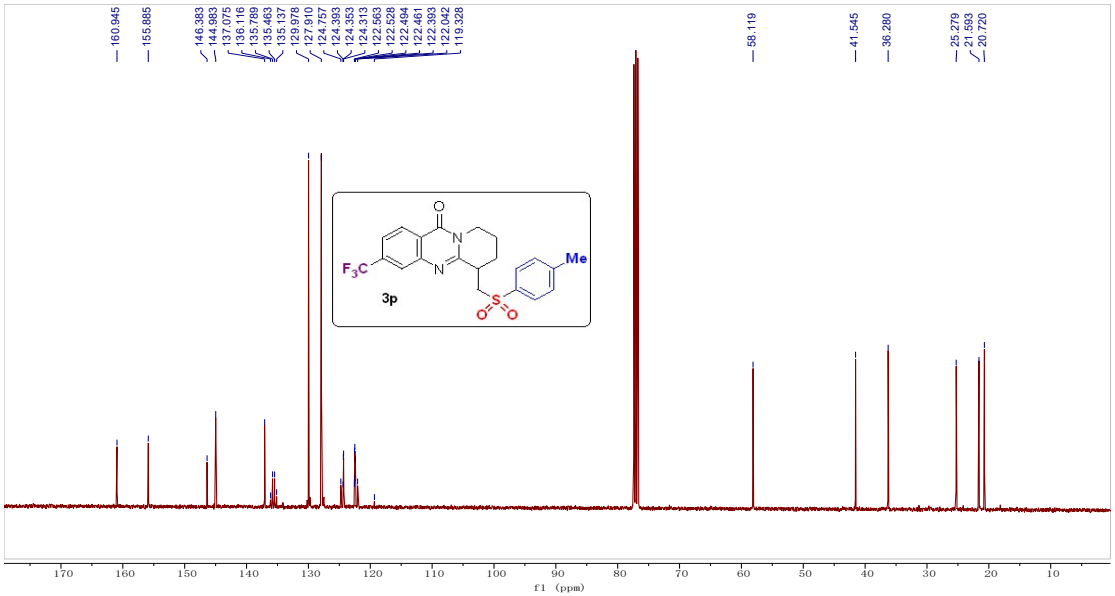
8.18
7.83
7.81
7.35
7.33
7.13
7.12
7.11
7.10
7.09
7.08
7.07
7.06
7.05
4.35
4.32
4.32
4.26
4.25
4.23
4.21
4.20
3.97
3.96
3.94
3.92
3.91
3.47
3.46
3.44
3.42
3.40
3.39
3.37
2.64
2.63
2.61
2.60
2.42
2.05
2.03
2.01
2.00
1.99
1.88
1.86
1.85
1.83
1.82
1.81
1.80
1.00
1.97
2.04
2.08
1.01
1.06
1.00
2.01
1.04
3.10
2.20
1.11
F
N
N
O
Me
S
O
O
3o
f1 (ppm)


167.56
165.04
161.04
155.59
148.74
148.61
144.93
137.02
129.98
129.48
129.38
127.92
116.94
115.55
115.32
112.07
111.85
58.10
41.26
36.19
25.27
21.65
20.73
F
N
N
O
Me
S
O
O
3o
f1 (ppm)


-103.58
F
N
N
O
Me
S
O
O
3o
f1 (ppm)


8.32
8.30
7.83
7.81
7.61
7.61
7.59
7.59
7.34
7.32
4.35
4.35
4.32
4.31
4.29
4.29
4.27
4.26
4.25
4.24
4.02
4.00
3.99
3.98
3.97
3.95
3.54
3.52
3.52
3.51
3.50
3.50
3.49
3.48
3.47
3.45
3.43
3.42
3.40
2.68
2.67
2.65
2.64
2.62
2.61
2.40
2.08
2.07
2.06
2.06
2.05
2.05
2.04
2.03
1.92
1.88
1.87
1.87
1.86
1.84
1.84
1.82
1.02
2.00
1.04
1.04
2.00
1.11
0.98
1.01
0.98
1.05
1.01
3.00
2.10
1.09
F3C
Me
3p
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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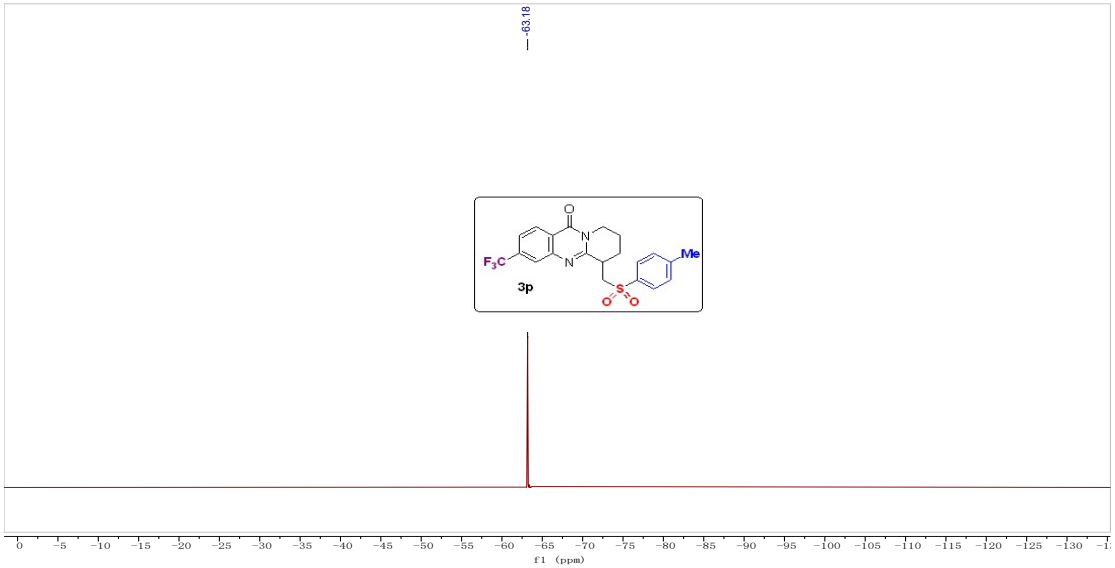
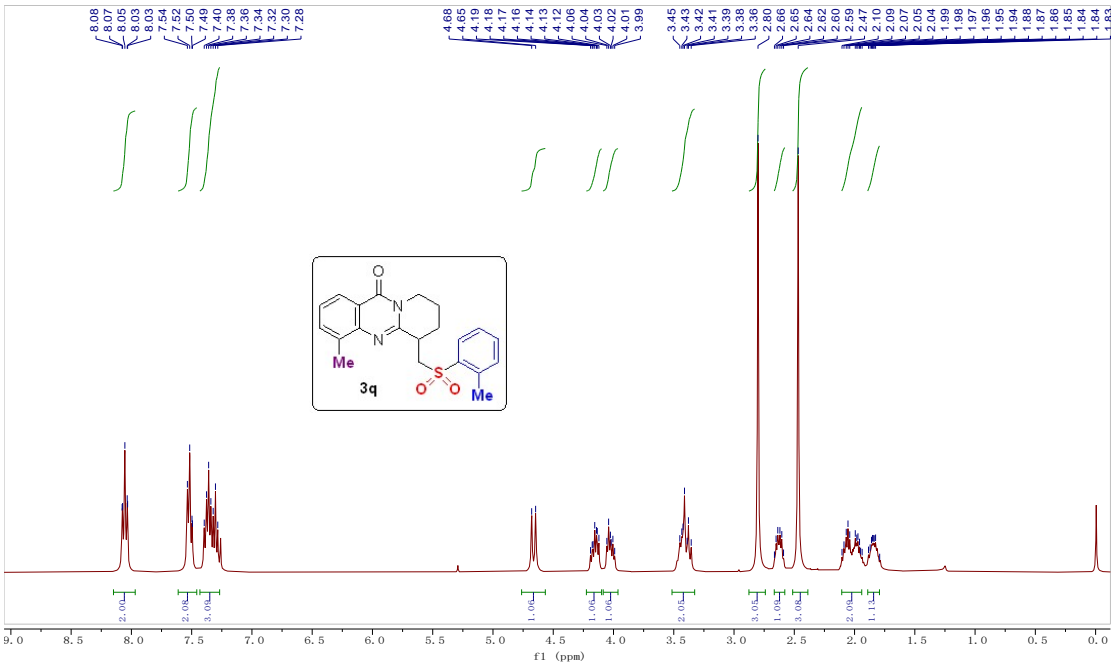
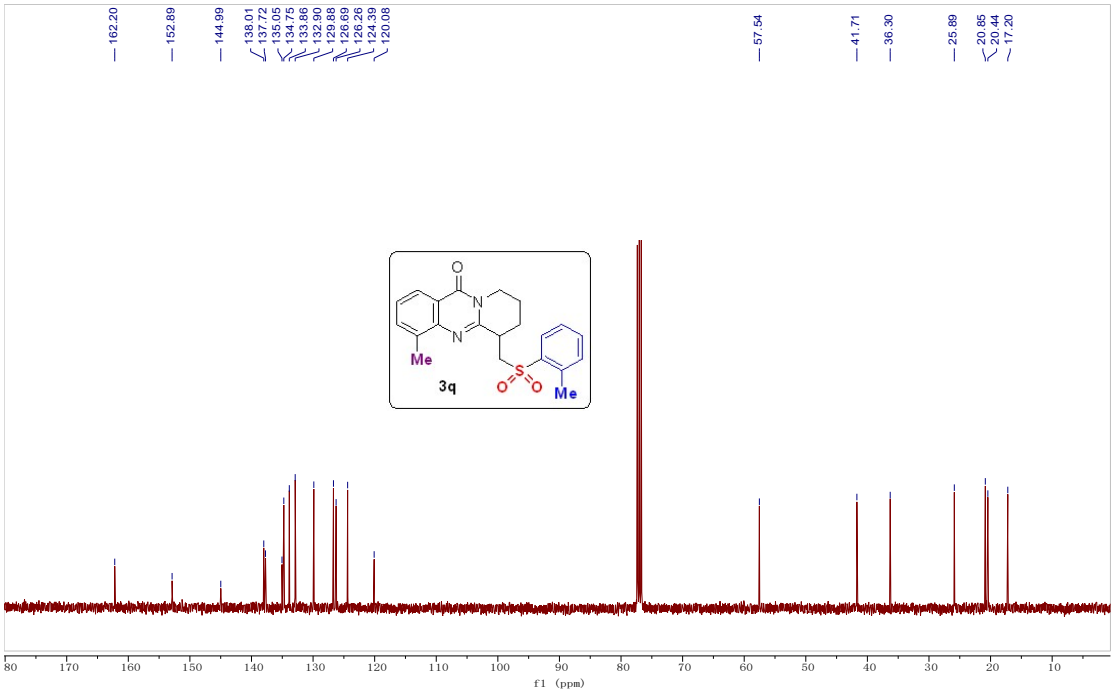
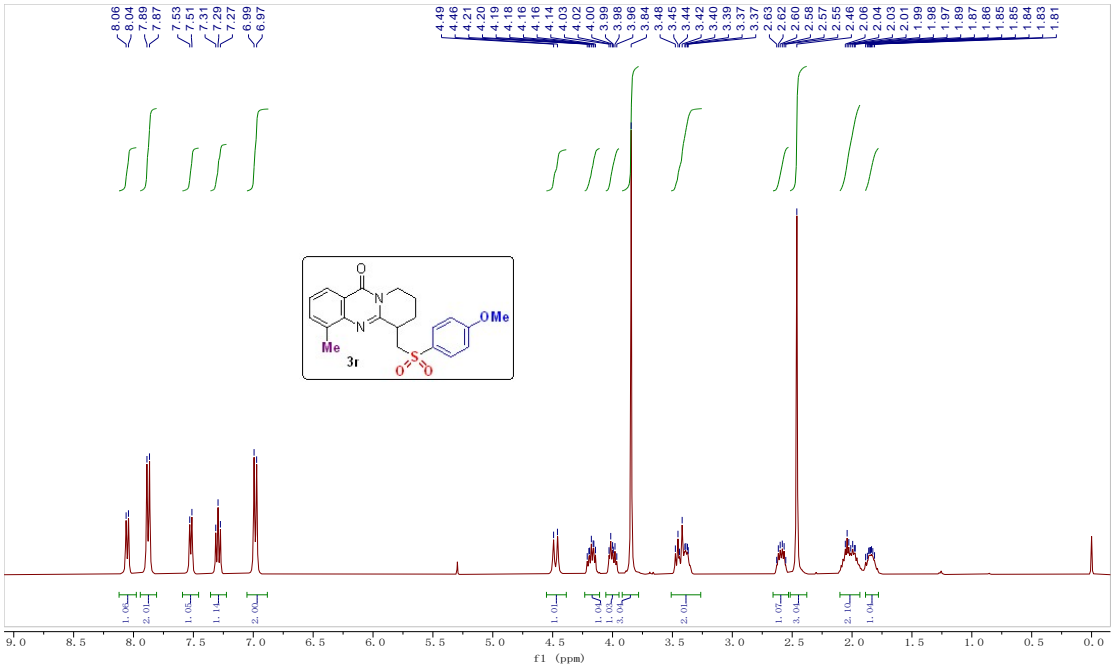
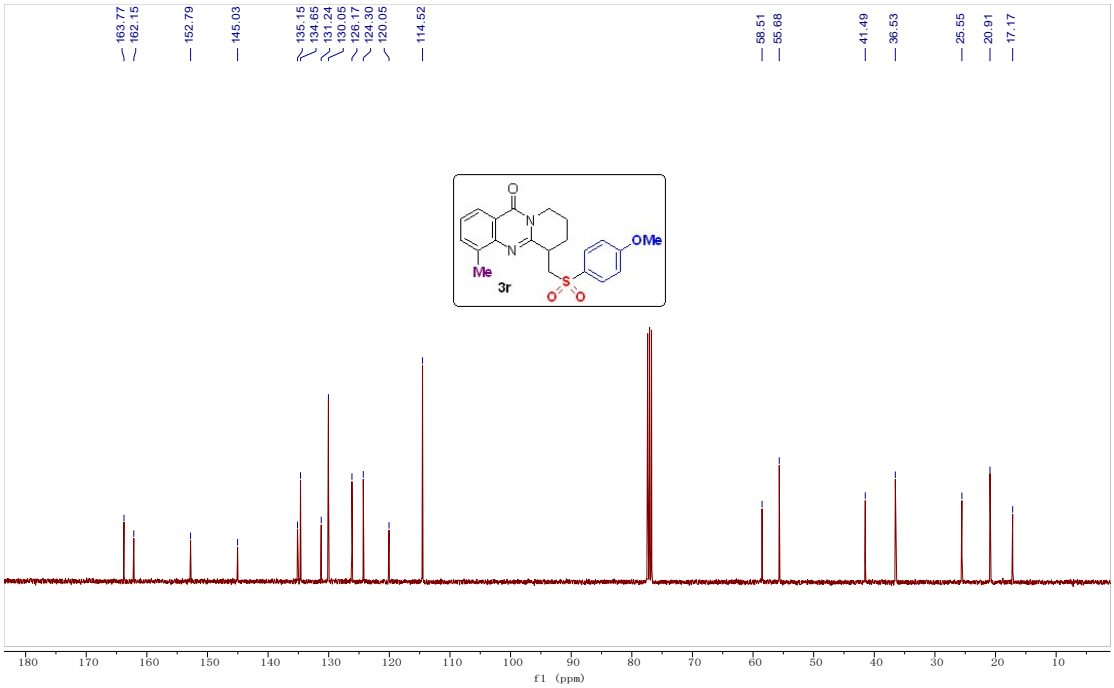
146.383
144.983
137.075
136.116
135.789
135.463
135.137
129.978
127.910
124.757
124.393
124.353
124.313
122.563
122.528
122.494
122.461
122.393
122.042
119.328
58.119
41.545
36.280
25.279
21.583
20.720
F3C
Me
3p
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

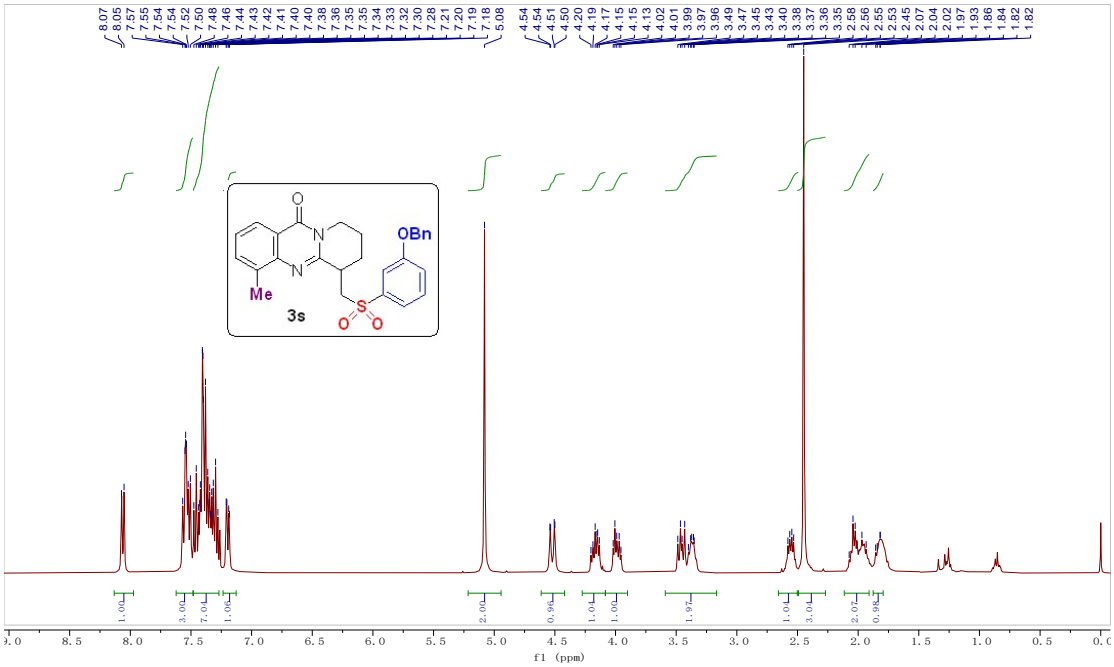

-63.18
F3C
Me
3p
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
f1 (ppm)

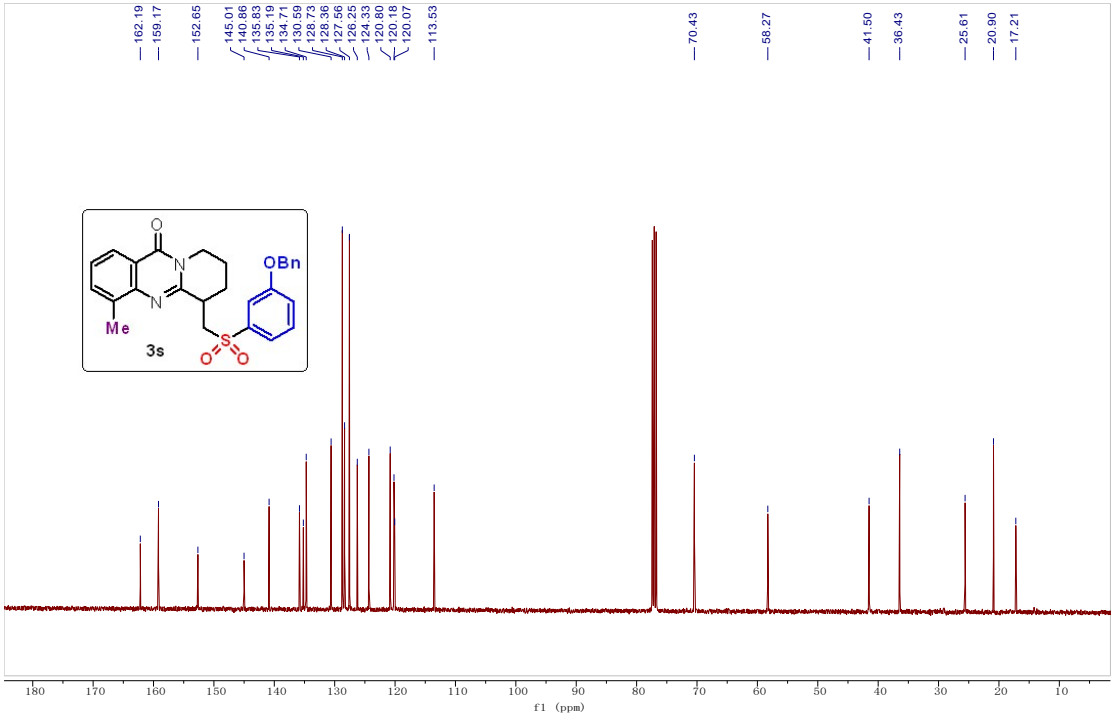

8.08 8.07 8.05 8.03 8.03 7.54 7.52 7.50 7.49 7.40 7.38 7.36 7.34 7.32 7.30 7.28
4.68 4.65 4.19 4.18 4.17 4.16 4.14 4.13 4.12 4.06 4.04 4.03 4.02 4.01 3.99
3.45 3.43 3.42 3.41 3.39 3.38 3.36 2.80 2.66 2.65 2.64 2.62 2.60 2.59 2.47 2.10 2.09 2.07 2.05 2.04 1.99 1.98 1.97 1.96 1.95 1.94 1.88 1.87 1.86 1.85 1.84 1.84 1.83
Me
3q
Me
2.00 2.08 3.09 1.00 1.00 1.00 2.05 3.05 1.00 3.08 2.09 1.13
f1 (ppm)

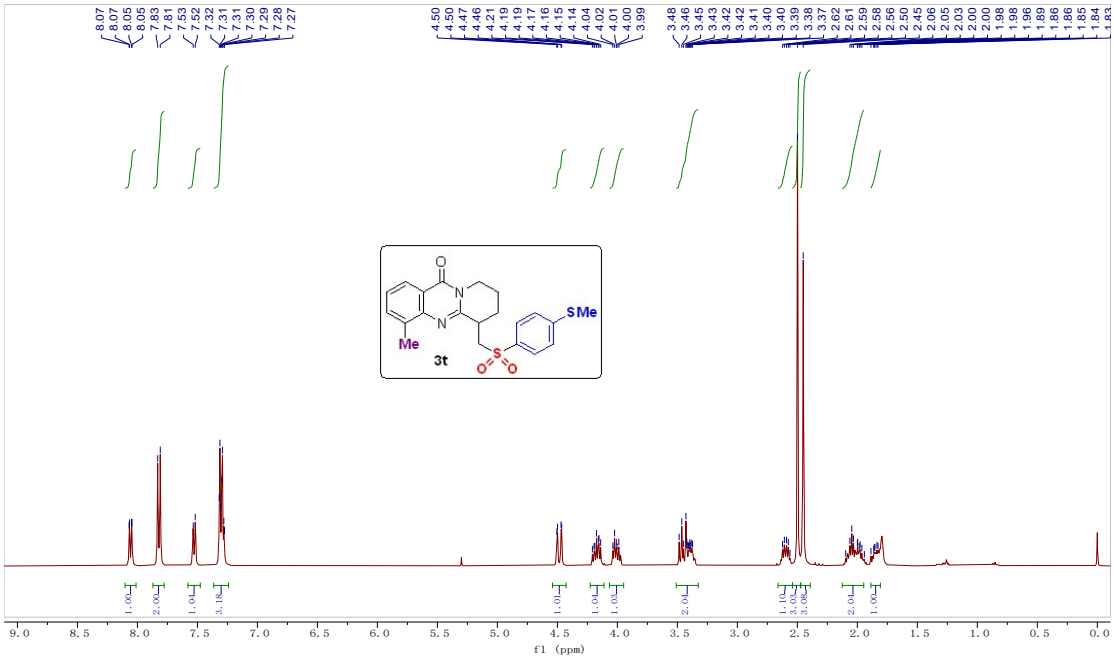

162.20 152.89 144.99 138.01 137.72 135.05 134.75 133.86 132.90 129.88 126.69 126.26 124.39 120.08
57.54 41.71 36.30 25.89 20.85 20.44 17.20
Me
3q
Me
f1 (ppm)

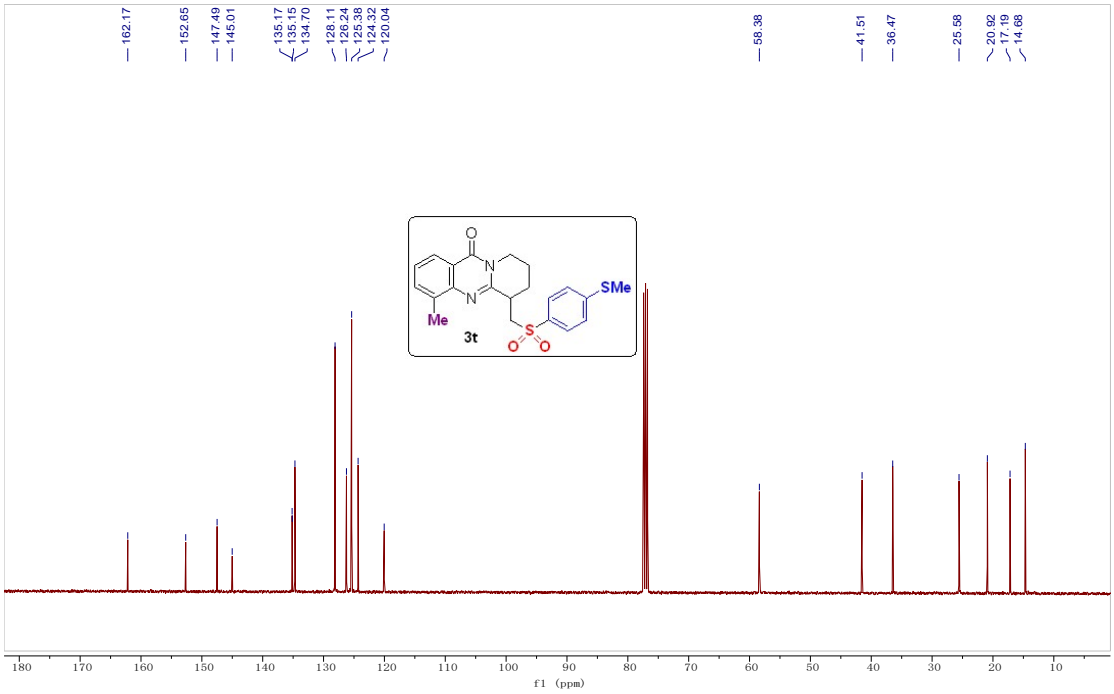

8.06
8.04
7.89
7.87
7.53
7.51
7.31
7.29
7.27
6.99
6.97
4.49
4.46
4.21
4.20
4.19
4.18
4.16
4.16
4.14
4.03
4.02
4.00
3.99
3.98
3.96
3.84
3.48
3.45
3.44
3.42
3.40
3.39
3.37
3.37
2.63
2.62
2.60
2.58
2.57
2.55
2.46
2.06
2.04
2.03
2.01
1.99
1.98
1.97
1.89
1.87
1.86
1.85
1.85
1.84
1.83
1.81
OMe
Me
3r
f1 (ppm)

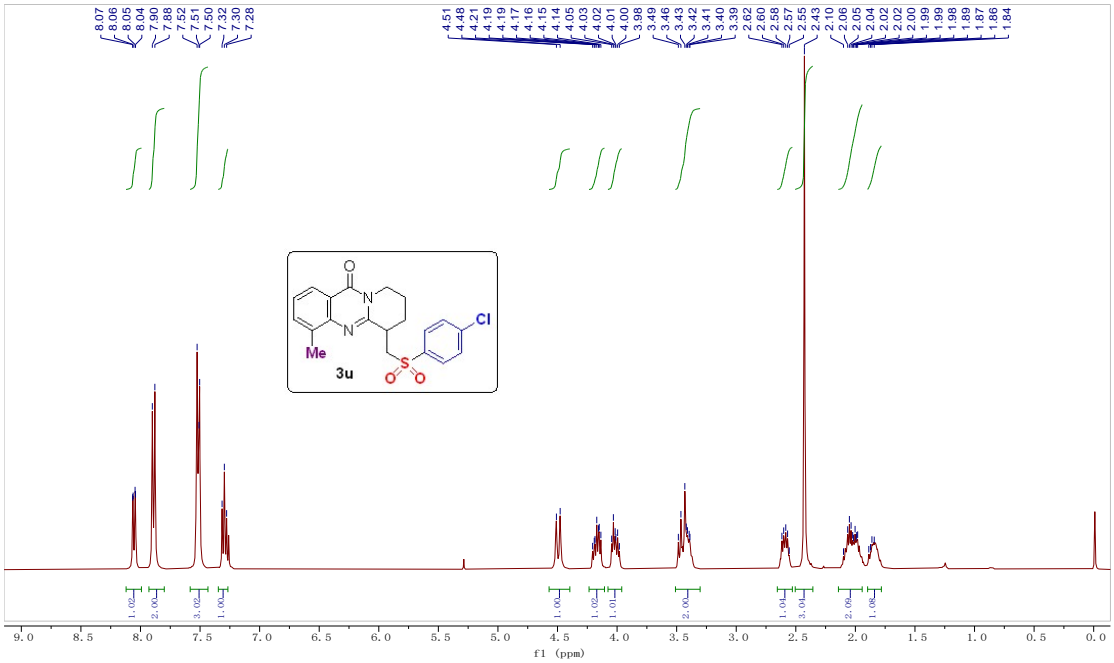

163.77
162.15
152.79
145.03
135.15
134.65
131.24
130.05
126.17
124.30
120.05
114.52
58.51
55.68
41.49
36.53
25.55
20.91
17.17
OMe
Me
3r
f1 (ppm)


8.07 8.05 7.57 7.55 7.54 7.54 7.52 7.50 7.48 7.46 7.44 7.43 7.42 7.41 7.40 7.40 7.38 7.36 7.35 7.35 7.34 7.33 7.32 7.30 7.28 7.21 7.20 7.19 7.18 5.08
4.54 4.54 4.51 4.50 4.20 4.19 4.17 4.15 4.15 4.13 4.02 4.01 3.99 3.97 3.96 3.49 3.47 3.45 3.43 3.40 3.38 3.37 3.36 3.35 2.58 2.56 2.55 2.53 2.45 2.07 2.04 2.02 1.97 1.93 1.86 1.84 1.82 1.82
OBn
Me
3s
1.00 3.00 7.01 1.00 2.00 0.96 1.01 1.00 1.97 1.01 3.01 2.07 0.98
f1 (ppm)


162.19 159.17 152.65 145.01 140.86 135.83 135.19 134.71 130.59 128.73 128.36 127.56 124.25 124.33 120.80 120.18 120.07 113.53
70.43 58.27 41.50 36.43 25.61 20.90 17.21
OBn
Me
3s
f1 (ppm)


8.07 8.07 8.05 8.05 7.83 7.81 7.53 7.52 7.32 7.31 7.31 7.30 7.29 7.28 7.27
4.50 4.50 4.47 4.46 4.21 4.19 4.19 4.17 4.16 4.15 4.14 4.04 4.02 4.01 4.00 3.99
3.48 3.46 3.45 3.43 3.42 3.42 3.41 3.40 3.40 3.39 3.38 3.37 2.62 2.61 2.59 2.58 2.56 2.50 2.45 2.06 2.05 2.03 2.00 2.00 1.98 1.98 1.96 1.89 1.86 1.86 1.85 1.84 1.83
1.00 2.00 1.04 3.18 1.01 1.04 1.03 2.04 1.10 3.02 3.08 2.04 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
O
N
N
Me
SMe
O
S
O
3t


162.17 152.65 147.49 145.01 135.17 135.15 134.70 128.11 126.24 125.38 124.32 120.04
58.38 41.51 36.47 25.58 20.92 17.19 14.68
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)
O
N
N
Me
SMe
O
S
O
3t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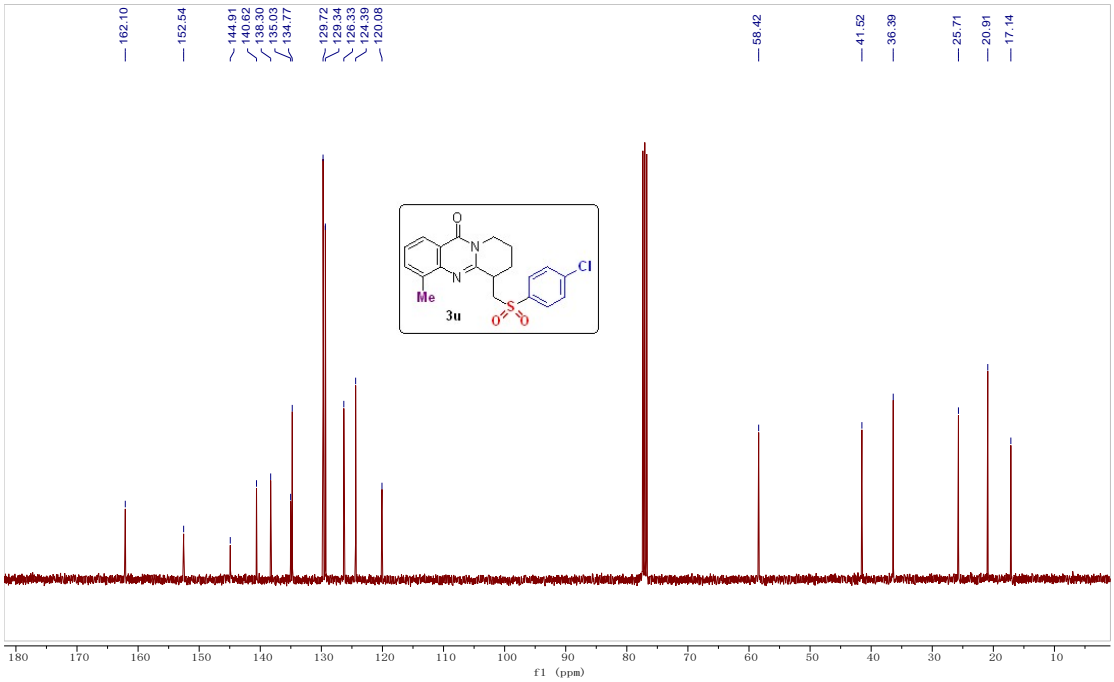
8.06
8.05
8.04
7.90
7.88
7.52
7.51
7.50
7.32
7.30
7.28
4.51
4.48
4.21
4.19
4.19
4.17
4.16
4.15
4.14
4.05
4.03
4.02
4.01
4.00
3.98
3.49
3.46
3.43
3.42
3.41
3.40
3.39
2.62
2.60
2.58
2.57
2.55
2.43
2.10
2.06
2.05
2.04
2.02
2.00
1.99
1.99
1.98
1.89
1.87
1.86
1.84
O
N
N
Me
S
O
O
Cl
3u
1.02
2.00
3.02
1.00
1.00
1.02
1.01
2.00
1.04
3.01
2.09
1.08
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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152.54
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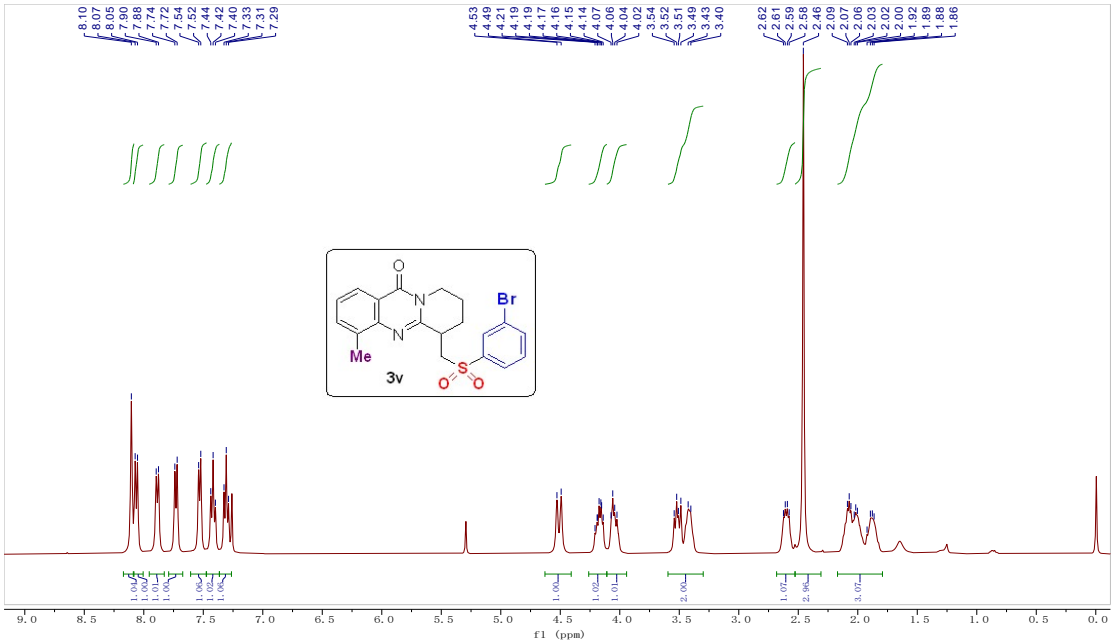
140.62
138.30
135.03
134.77
129.72
129.34
126.33
124.39
120.08
58.42
41.52
36.39
25.71
20.91
17.14
O
N
N
Me
S
O
O
Cl
3u
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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8.07
8.05
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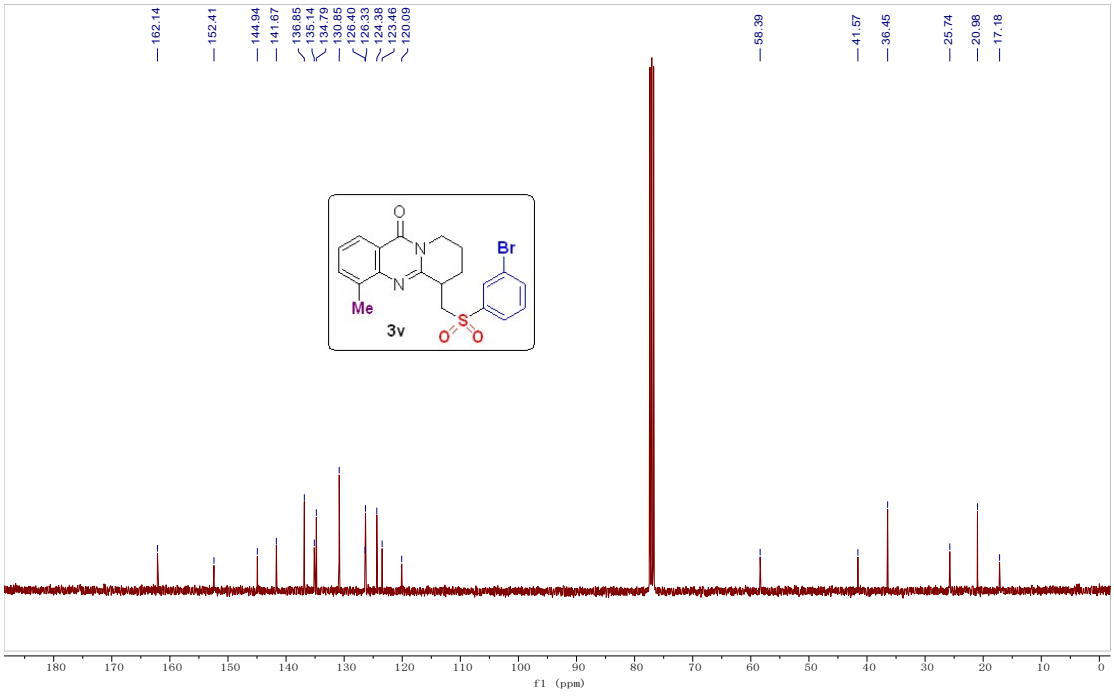
7.74
7.72
7.54
7.52
7.44
7.42
7.40
7.33
7.31
7.29
4.53
4.49
4.21
4.19
4.19
4.17
4.16
4.15
4.14
4.07
4.06
4.04
4.02
3.54
3.52
3.51
3.49
3.43
3.40
2.62
2.61
2.59
2.58
2.46
2.09
2.07
2.06
2.03
2.02
2.00
1.92
1.89
1.88
1.86
Br
Me
3v
f1 (ppm)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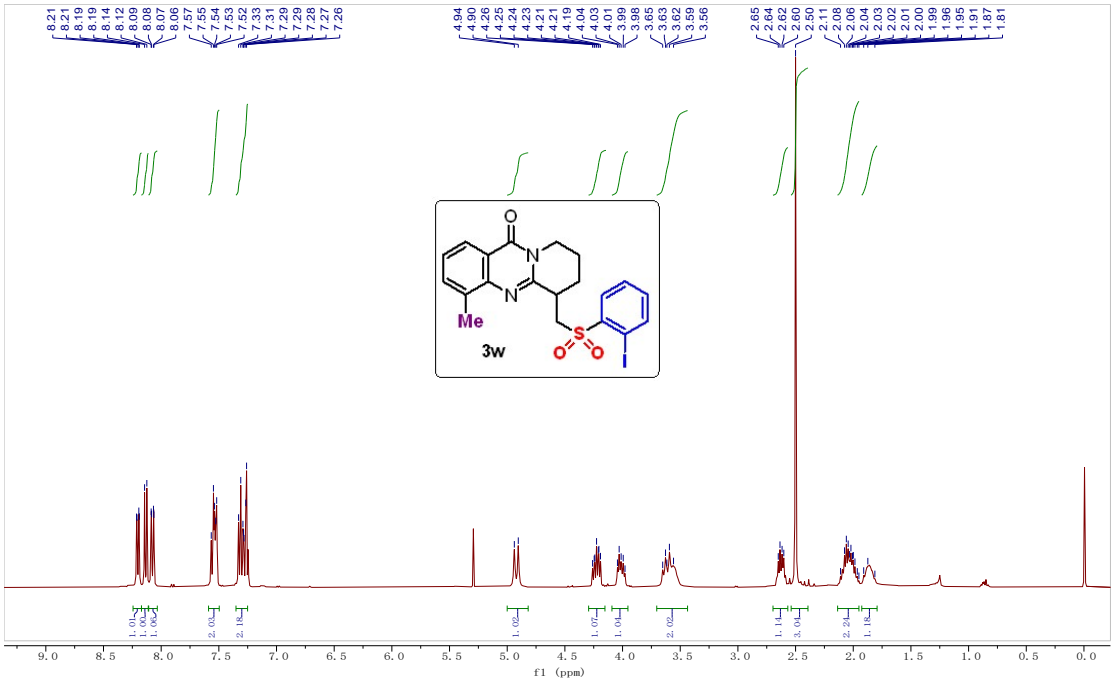
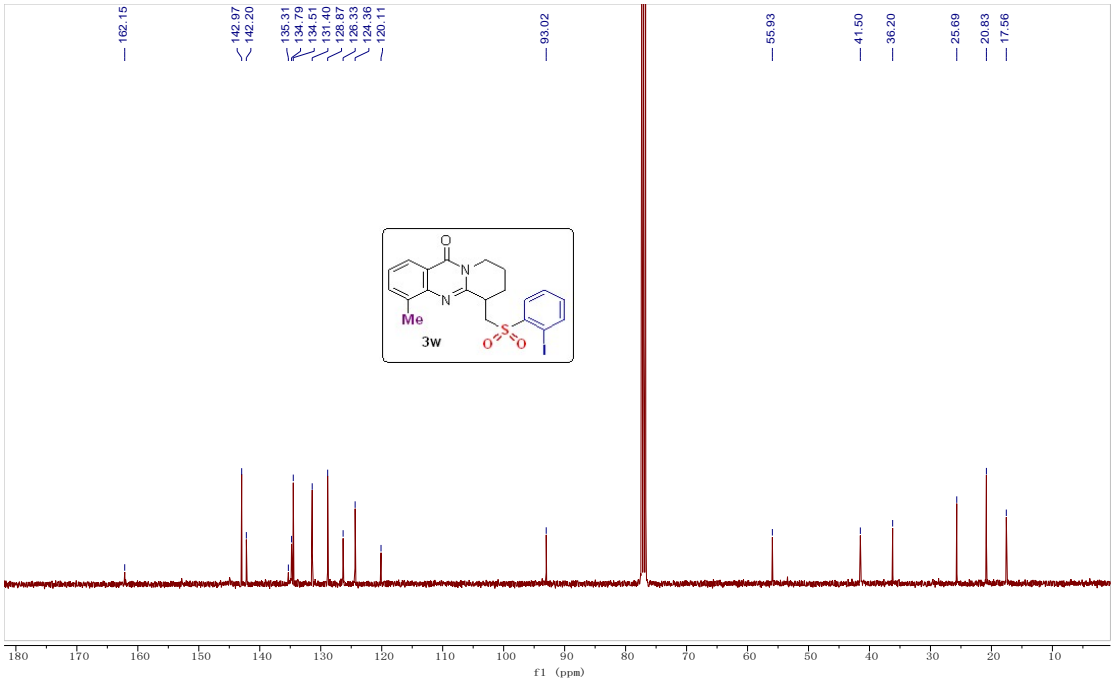
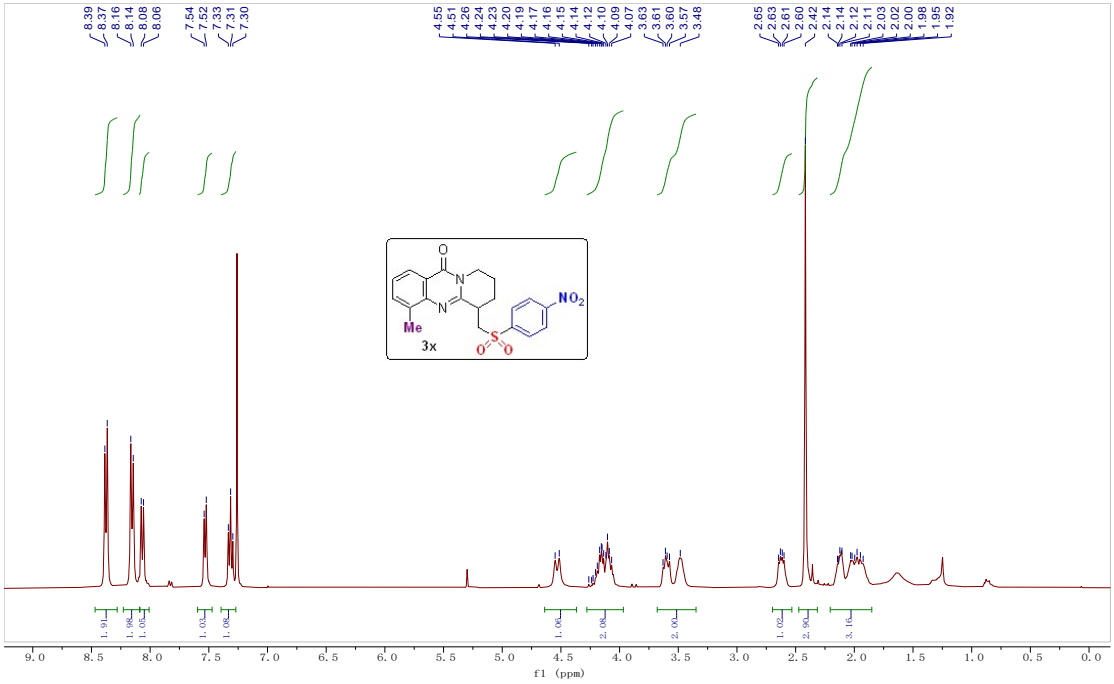
152.41
144.94
141.67
136.85
135.14
134.78
130.85
126.40
126.33
124.38
123.46
120.09
58.39
41.57
36.45
25.74
20.98
17.18
Br
Me
3v
f1 (ppm)


8.21 8.21 8.19 8.19 8.14 8.12 8.09 8.08 8.07 8.06 7.57 7.55 7.54 7.53 7.52 7.33 7.31 7.29 7.29 7.28 7.27 7.26
4.94 4.90 4.26 4.25 4.24 4.23 4.21 4.21 4.19 4.04 4.03 4.01 3.99 3.98 3.65 3.63 3.62 3.59 3.56
2.65 2.64 2.62 2.60 2.50 2.11 2.08 2.06 2.04 2.03 2.02 2.01 2.00 1.99 1.96 1.95 1.91 1.87 1.81
Me
3w
f1 (ppm)


162.15
142.97 142.20 135.31 134.79 134.51 131.40 128.87 126.33 124.36 120.11
93.02
55.93
41.50 36.20
25.69 20.83 17.56
Me
3w
f1 (ppm)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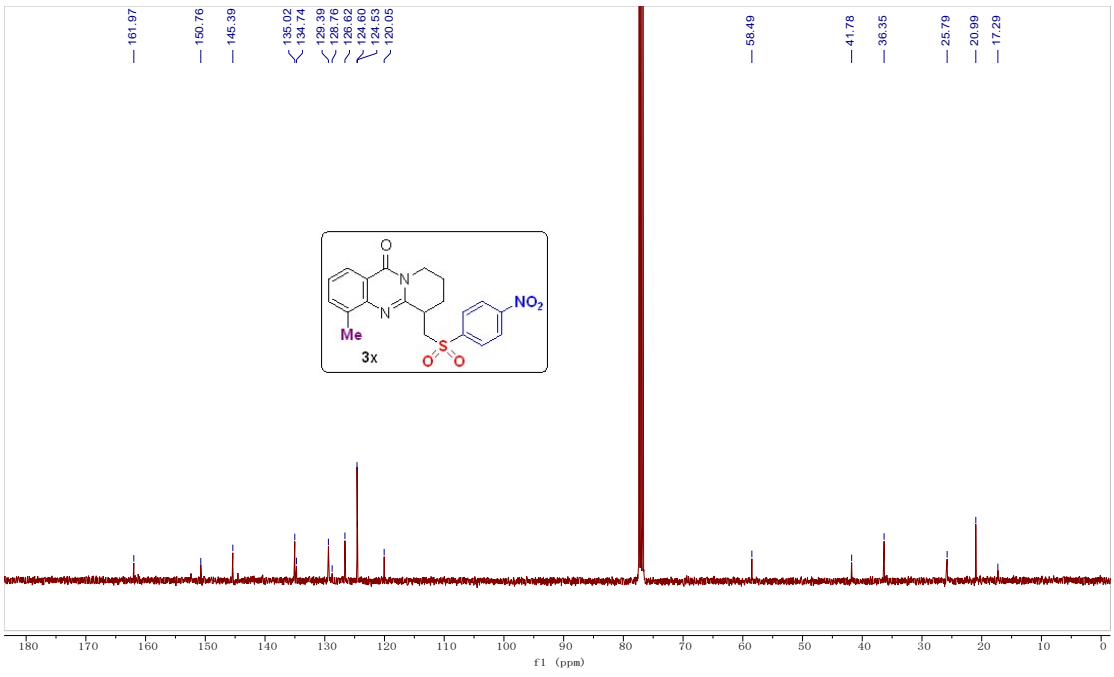
8.37
8.16
8.14
8.08
8.06
7.54
7.52
7.33
7.31
7.30
4.55
4.51
4.26
4.24
4.23
4.20
4.19
4.17
4.16
4.15
4.14
4.12
4.10
4.09
4.07
3.63
3.61
3.60
3.57
3.48
2.65
2.63
2.61
2.60
2.42
2.14
2.12
2.11
2.03
2.02
2.00
1.98
1.95
1.92
O
N
N
Me
NO2
S
O
O
3x
1.91
1.98
1.00
1.03
1.08
1.00
2.08
2.00
1.02
2.90
3.16
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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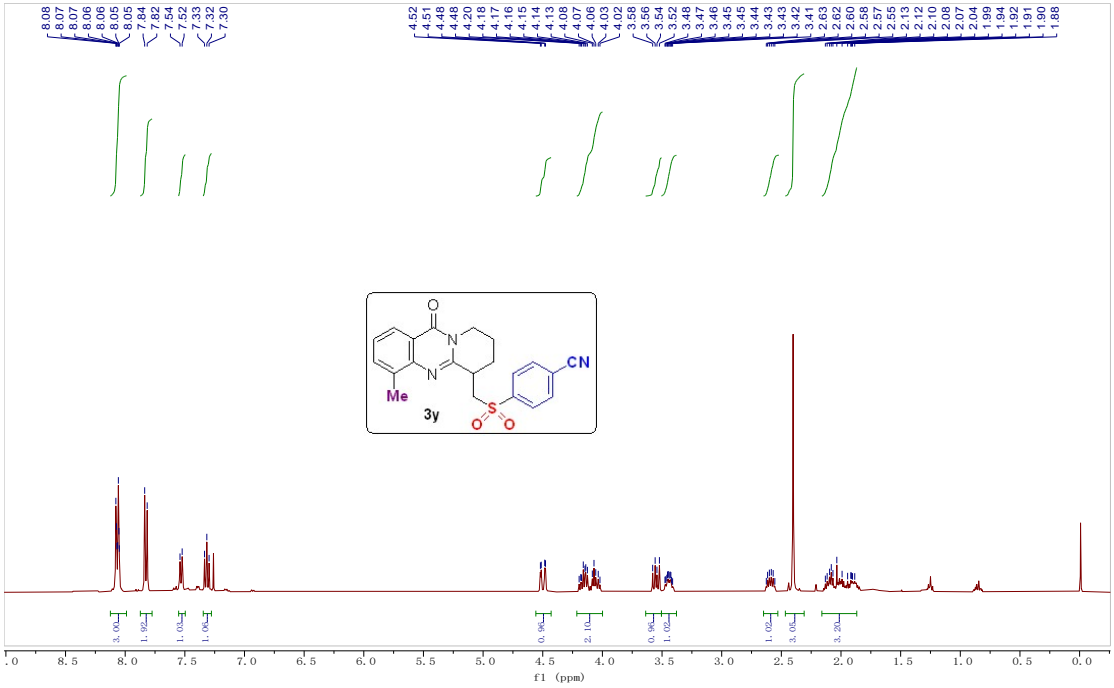
150.76
145.39
135.02
134.74
129.39
128.76
126.62
124.60
124.53
120.05
58.49
41.78
36.35
25.79
20.99
17.29
O
N
N
Me
NO2
S
O
O
3x
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.07
8.07
8.06
8.06
8.06
8.05
8.05
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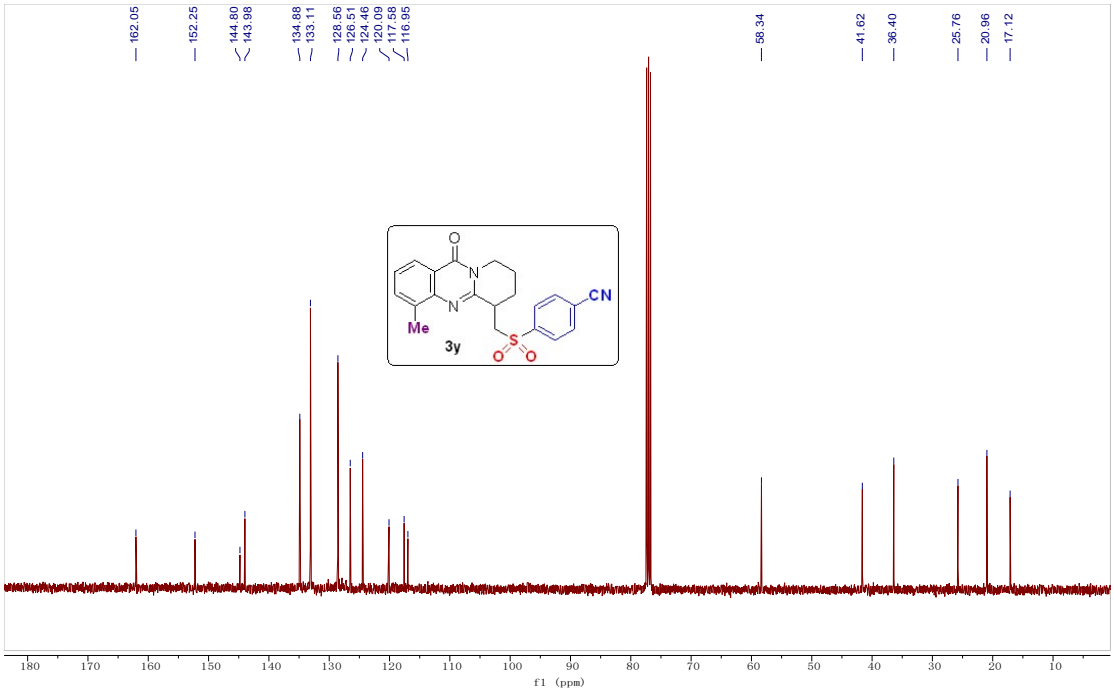
7.82
7.54
7.52
7.33
7.32
7.30
4.52
4.51
4.48
4.48
4.20
4.18
4.17
4.16
4.15
4.14
4.13
4.08
4.07
4.06
4.03
4.02
3.58
3.56
3.54
3.52
3.48
3.47
3.46
3.45
3.45
3.44
3.43
3.43
3.42
3.41
2.63
2.62
2.60
2.58
2.57
2.55
2.13
2.12
2.10
2.08
2.07
2.04
1.99
1.94
1.92
1.91
1.90
1.88
O
N
N
Me
S
O
O
CN
3y
3.00
1.95
1.03
1.06
0.96
2.10
0.96
1.05
1.02
3.06
3.20
f1 (ppm)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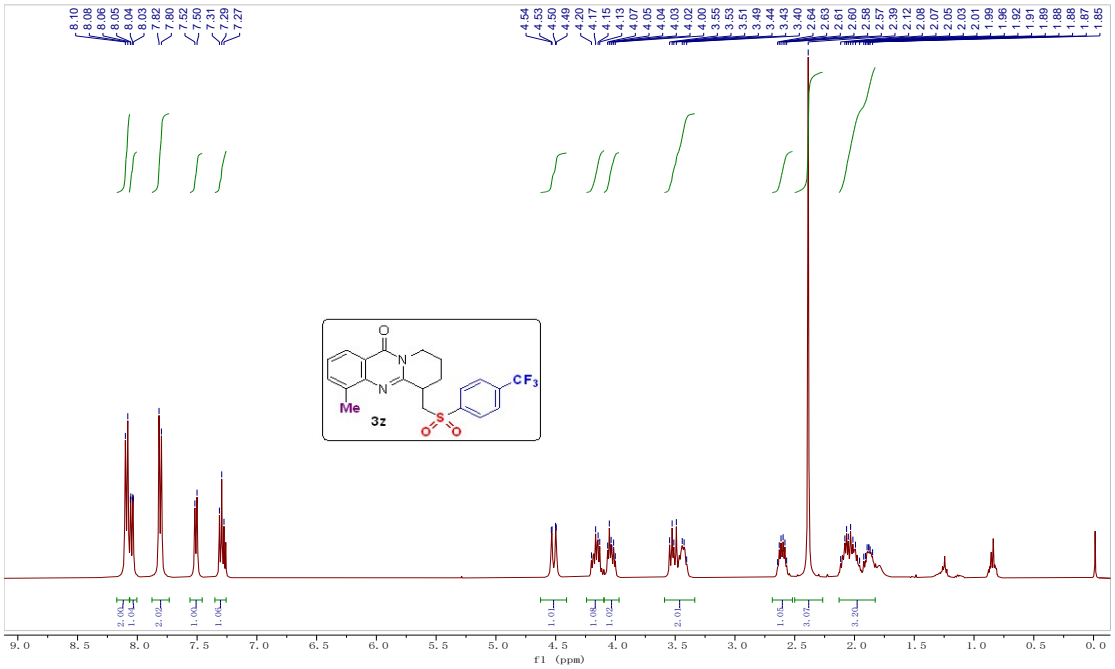
144.80
143.98
134.88
133.11
128.56
126.51
124.46
120.09
117.58
116.95
58.34
41.62
36.40
25.76
20.96
17.12
O
N
N
Me
S
O
O
CN
3y
f1 (ppm)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8.08
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8.05
8.04
8.03
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7.80
7.52
7.50
7.31
7.29
7.27
4.54
4.53
4.50
4.49
4.20
4.17
4.15
4.13
4.07
4.05
4.04
4.03
4.02
4.00
3.55
3.53
3.51
3.49
3.44
3.43
3.40
2.64
2.63
2.61
2.60
2.58
2.57
2.39
2.12
2.08
2.07
2.05
2.03
2.01
1.99
1.96
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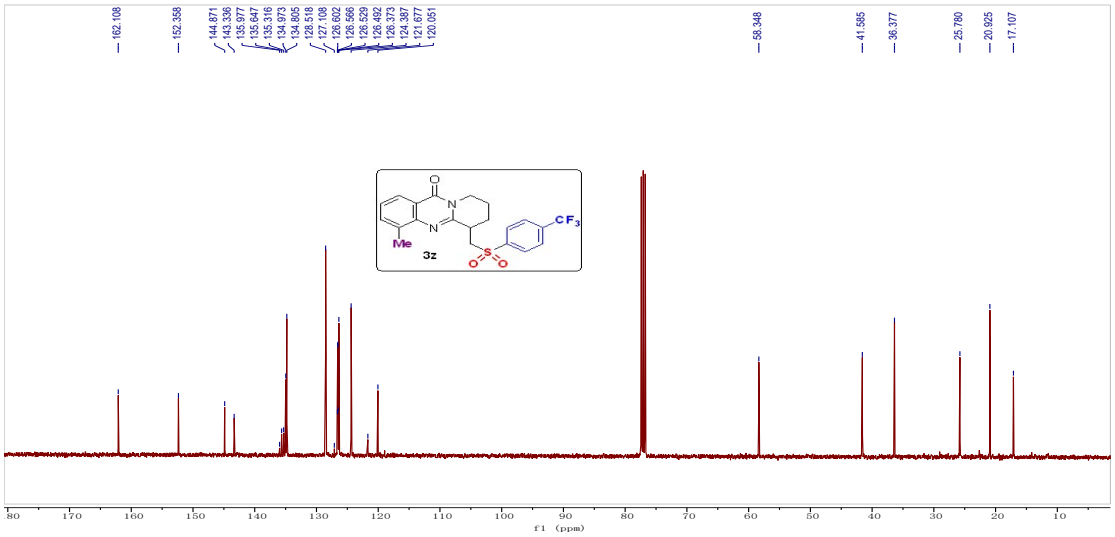
1.91
1.89
1.88
1.88
1.87
1.85
Me
3z
CF3
f1 (ppm)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126.602
126.566
126.529
126.492
126.373
124.387
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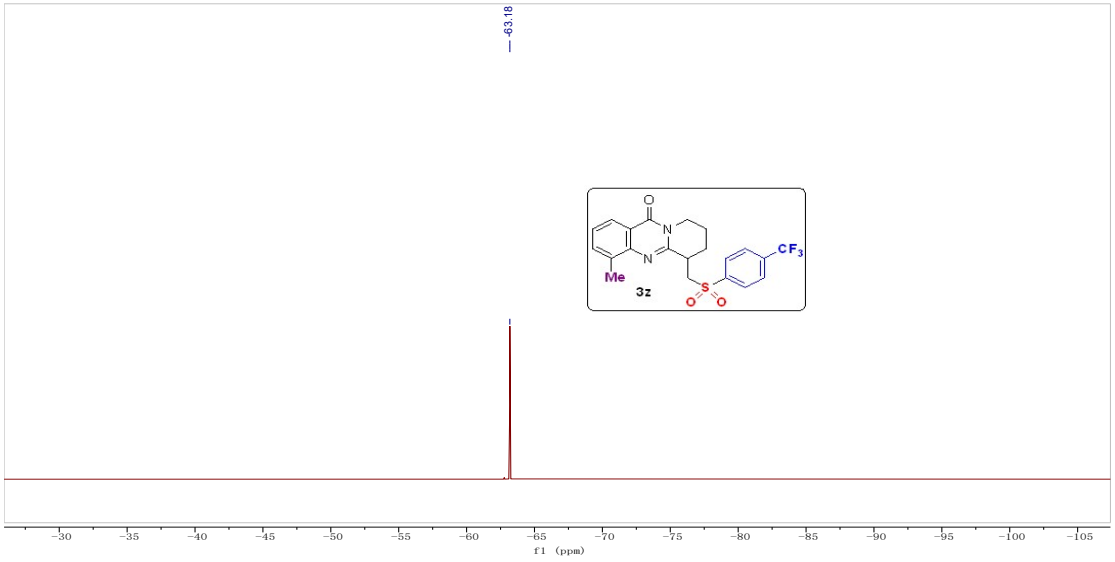
58.348
41.885
36.377
25.780
20.925
17.107
Me
3z
CF3
f1 (ppm)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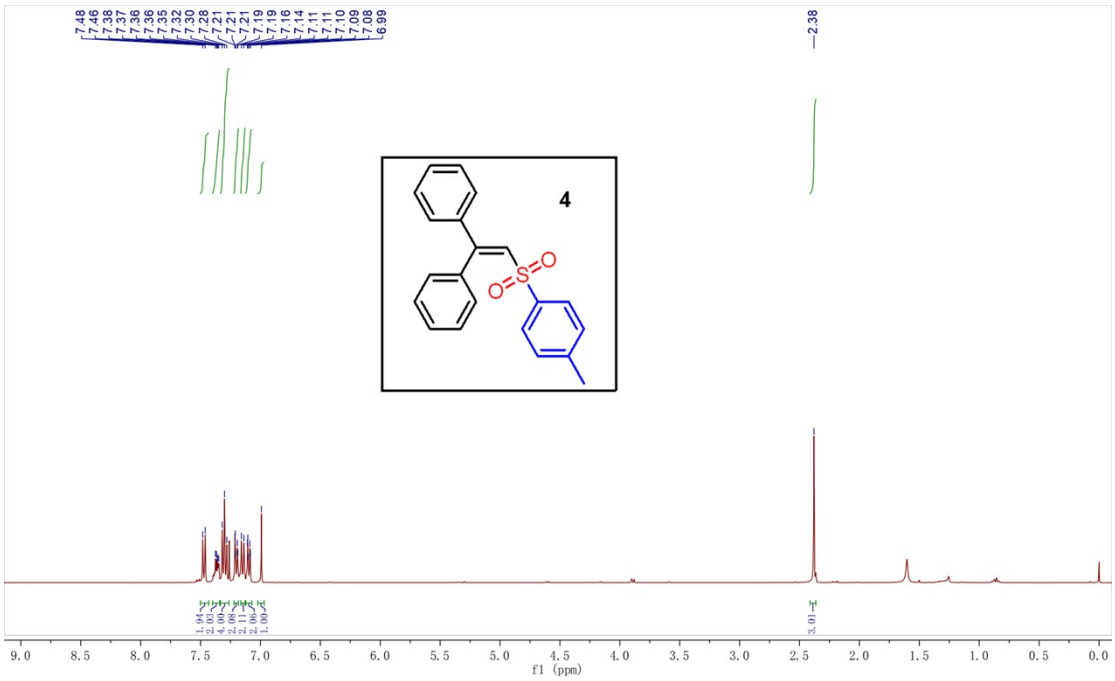
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7.36
7.35
7.32
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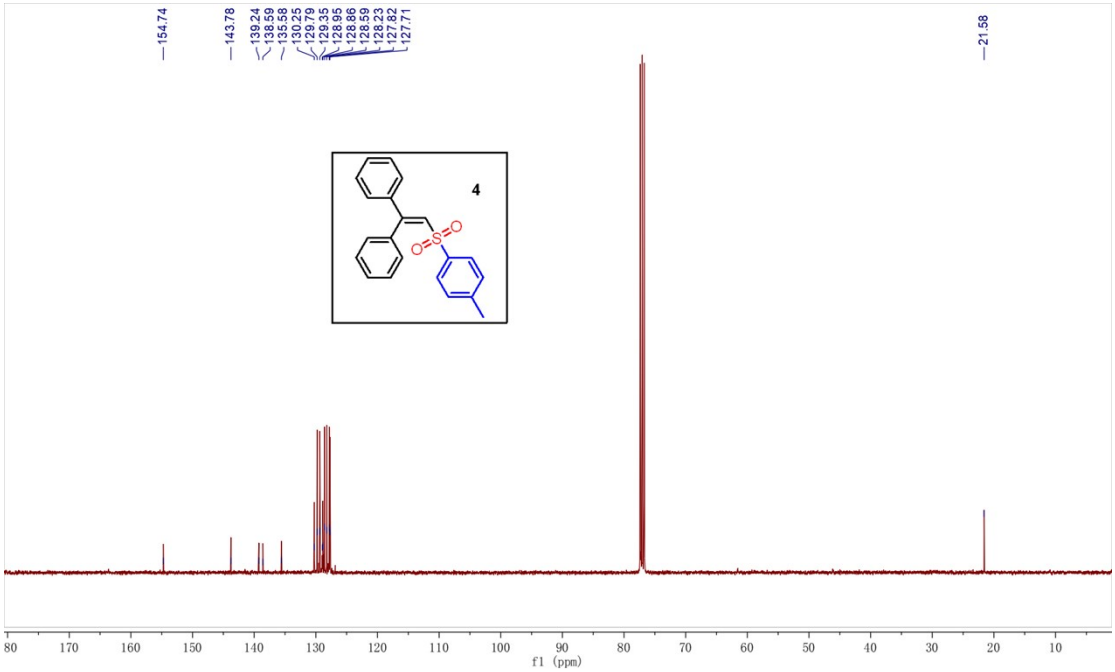
7.28
7.21
7.21
7.21
7.19
7.19
7.16
7.14
7.11
7.11
7.10
7.09
7.08
6.99
2.38
4
O=S=O
1.04
2.05
4.00
2.08
2.11
1.08
1.00
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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136.59
135.58
130.25
129.79
129.35
128.95
128.86
128.59
128.23
127.82
127.71
21.58
4
O=S=O
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

4 References

1. Mu, B.; Zhang, L.; Lv, G.; Chen, K.; Wang, T.; Chen, J.; Huang, T.; Guo, L.; Yang, Z.; Wu, Y. *J. Org. Chem.* **2022**, *87*, 10146.
2. Runemark, A.; Sundén, H. *J. Org. Chem.* **2022**, *87*, 1457.