Electronic Supplementary Material (ESI) for RSC Advances
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Supporting Information for

Room Temperature Synthesis of Isoquino[2,1-*a*][3,1]oxazine and Isoquino[2,1-*a*]pyrimidine Derivatives *via* Visible Light Photoredox Catalysis

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1. General Information

Unless otherwise noted, materials were purchased from commercial suppliers and used without further purification. All the solvents were treated according to general methods. Flash column chromatography was performed using 200-300 mesh silica gel. ^{1}H NMR spectra were recorded on 400 MHz spectrophotometers. Chemical shifts (δ) are reported in ppm from the solvent resonance as the internal standard ($CDCl_3$: 7.26 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet), coupling constants (Hz) and integration. ^{13}C NMR spectra were recorded on 100 MHz with complete proton decoupling spectrophotometers ($CDCl_3$: 77.0 ppm). Mass spectra were measured on a MS spectrometer. Elemental analysis was taken on an elementary analysis instrument.

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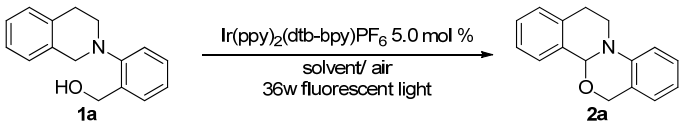
2. The Optimization of Reaction Conditions

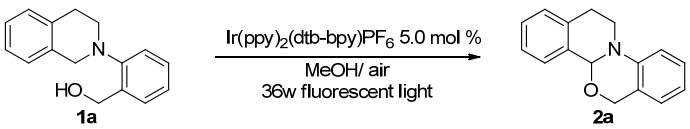
SI-Table 1. Solvent effects on the model reaction.[a]



entry	solvent	time (h)	yield (%)[b]
1	DMF	8	16
2	DMSO	3	37
3	CH_3CN	8	20
4	DCM	48	16
5	Acetone	10	38
6	Toluene	48	15
7	THF	4	19
8	MeOH	7	62
9	EtOH	24	58
10	^{i}PrOH	48	trace
11	^{i}BuOH	48	trace
12	BnOH	24	31
13	Glycol	48	trace

[a] Reaction conditions: **1a** (0.3 mmol) and $Ir(ppy)_2(dtb\text{-}bpy)PF_6$ (0.05 equiv) were dissolved in 3.0 mL indicated solvent, then stirred open to air at room temperature under irradiation of 36 w fluorescent light. [b] Isolated yield after flash chromatography.

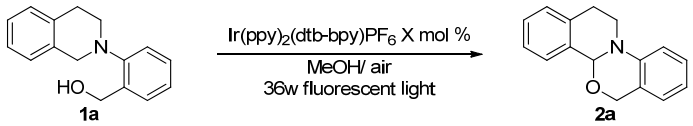
SI-Table 2. Effects of concentration on the model reaction.[a]



entry	concentration	time (h)	yield (%)[b]
1	0.05 M	7	56
2	0.1 M	7	65
3	0.15 M	12	69
4	0.3 M	12	73
5	0.5 M	12	62

[a] Reaction conditions: **1a** (0.3 mmol) and $Ir(ppy)_2(dtb\text{-}bpy)PF_6$ (0.05 equiv) were dissolved in MeOH (X mL), then stirred open to air at room temperature under irradiation of 36 w fluorescent light. [b] Isolated yield after flash chromatography.

SI-Table 3. Effects of catalyst loading on the model reaction.[a]



entry	X (mol %)	time (h)	yield (%)[b]
1	5.0	12	73
2	2.5	13	67
3	1.0	13	69
4	0.5	13	72

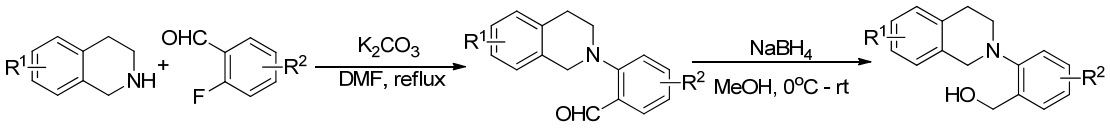
[a] Reaction conditions: **1a** (0.3 mmol) and $Ir(ppy)_2(dtb\text{-}bpy)PF_6$ (X mol%) were dissolved in MeOH (1.0 mL), then stirred open to air at room temperature under irradiation of 36 w fluorescent light. [b] Isolated yield after flash chromatography.

3. Preparation and Spectral Data of Substrates

3.1 Preparation of Substrates

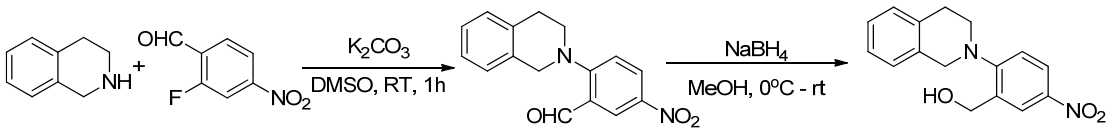
Substrates **1a-1f** and **1h** were prepared by following the procedures Route **I**.

*Route **I***



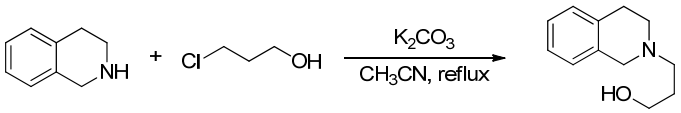
Substrate **1g** was prepared by following the Route **II**.

*Route **II***



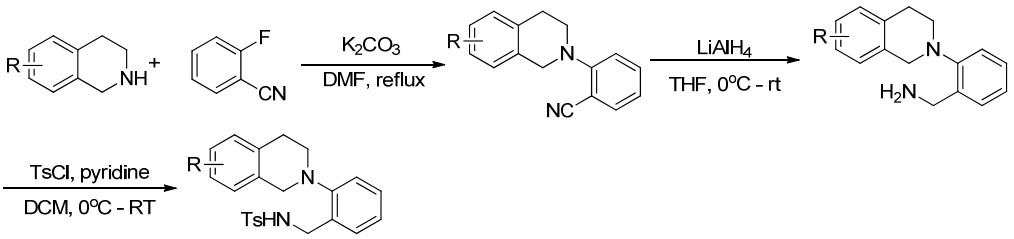
Substrate **1i** was prepared by following the Route **III**.

*Route **III***



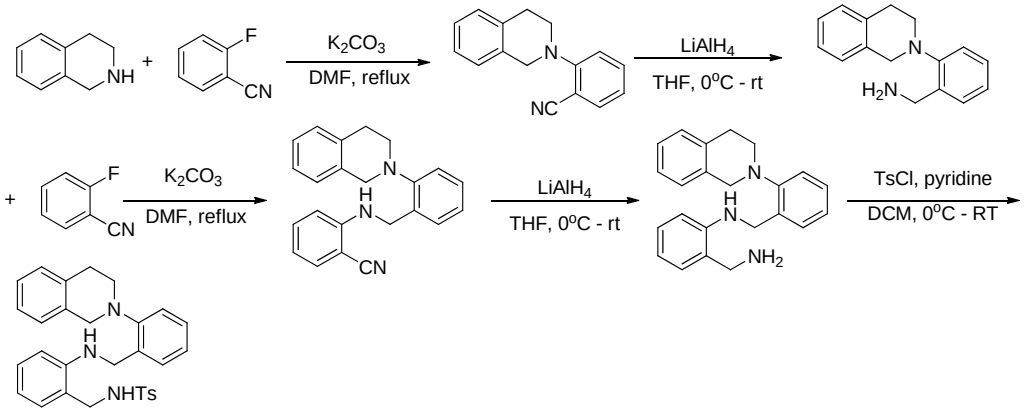
Substrates **1j – 1m** were prepared by following the Route **IV**.

*Route **IV***



Substrate **1n** was prepared by following the Route **V**.

*Route **V***

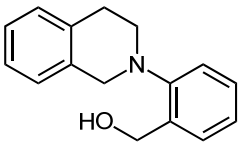


References:

1. S. P. Stanforth, *Tetrahedron*, 2000, **56**, 461.
2. D. J. Sall and G. L. Grunewald, *J. Med. Chem.*, 1987, **30**, 2208.
3. R. H. Mach, Y. Huang, R. A. Freeman, L. Wu, S. Vangveravonga and R. R. Luedtkec, *Bioorg. Med. Chem. Lett.*, 2004, **14**, 195.
4. G. Kumaraswamy, A. N. Murthy and A. Pitchaiah, *J. Org. Chem.*, 2010, **75**, 3916.

3.2 Spectral Data of Substrates

(2-(3,4-dihydroisoquinolin-2(1*H*)-yl)phenyl)methanol (1a)



White solid, **mp:** 61~64°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.38 – 7.21 (m, 3H), 7.21 – 7.11 (m, 4H), 7.06 (d, *J* = 6.9 Hz, 1H), 5.07 (s, 1H), 4.83 (s, 2H), 4.16 (s, 2H), 3.29 (t, *J* = 5.8 Hz, 2H), 3.05 (t, *J* = 5.7 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 150.75, 135.81, 134.42, 133.68, 128.90, 128.63, 128.37, 126.46, 126.34, 125.86, 124.92, 120.96, 64.50, 54.91, 50.70, 29.53. **MS:** m/z = 239.20 (M^+).

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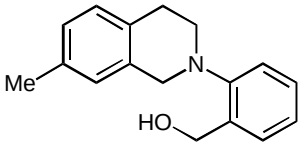
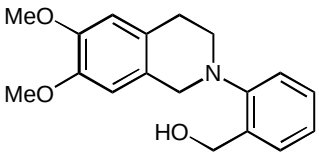
(2-(7-methyl-3,4-dihydroisoquinolin-2(1*H*)-yl)phenyl)methanol (1b)



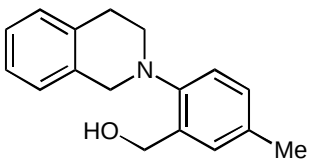
Yellow oil, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.33 – 7.19 (m, 3H), 7.14 (t, *J* = 7.2 Hz, 1H), 7.06 (d, *J* = 7.7 Hz, 1H), 7.01 (d, *J* = 7.8 Hz, 1H), 6.88 (s, 1H), 5.12 (s, 1H), 4.82 (s, 2H), 4.12 (s, 2H), 3.28 (t, *J* = 5.8 Hz, 2H), 2.99 (t, *J* = 5.6 Hz, 2H), 2.32 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 150.78, 135.77, 135.35, 134.22, 130.58, 128.74, 128.58, 128.31, 127.31, 126.79, 124.83, 120.94, 64.44, 54.88, 50.79, 29.07, 20.96. **MS:** m/z = 253.19 (M^+).

(2-(6,7-dimethoxy-3,4-dihydroisoquinolin-2(1*H*)-yl)phenyl)methanol (1c)



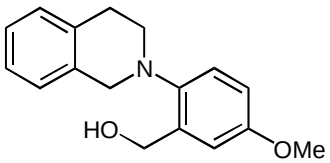
White solid, **mp:** 128~132°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.33 – 7.19 (m, 3H), 7.14 (t, J = 7.1 Hz, 1H), 6.65 (s, 1H), 6.55 (s, 1H), 5.19 (s, 1H), 4.83 (s, 2H), 4.09 (s, 2H), 3.87 (s, 3H), 3.85 (s, 3H), 3.28 (t, *J* = 5.8 Hz, 2H), 2.95 (t, *J* = 5.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 150.70, 147.68, 147.33, 135.74, 128.56, 128.28, 126.25, 125.60, 124.82, 120.96, 111.52, 109.10 , 64.47, 55.87, 54.44, 50.64, 28.98. **MS:** m/z = 299.17(M^+).

(2-(3,4-dihydroisoquinolin-2(1*H*)-yl)-5-methylphenyl)methanol (1d)



Yellow oil, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.17 (d, *J* = 7.7 Hz, 4H), 7.10 (d, *J* = 8.1 Hz, 1H), 7.08 – 6.95 (m, 2H), 5.25 (s, 1H), 4.79 (s, 2H), 4.13 (s, 2H), 3.27 (t, *J* = 5.7 Hz, 2H), 3.04 (t, *J* = 5.4 Hz, 2H), 2.33 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 148.26, 135.59, 134.58, 134.51, 133.67, 129.23, 128.87, 128.78, 126.40, 126.33, 125.81, 120.92, 64.57, 55.09, 50.79, 29.58, 20.78. **MS:** m/z = 253.19(M^+).

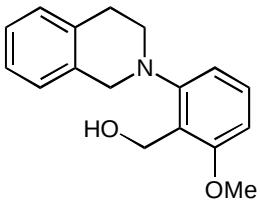
(2-(3,4-dihydroisoquinolin-2(1*H*)-yl)-5-methoxyphenyl)methanol (1e)



White solid, **mp:** 77~80°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ

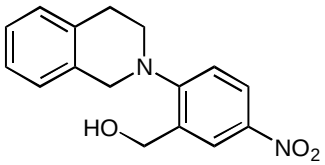
(ppm) 7.25 – 7.17 (m, 4H), 7.05 (d, *J* = 7.0 Hz, 1H), 6.88 – 6.79 (m, 1H), 6.76 (s, 1H), 5.39 (s, 1H), 4.79 (s, 2H), 4.11 (s, 2H), 3.80 (s, 3H), 3.24 (t, *J* = 5.8 Hz, 2H), 3.03 (t, *J* = 5.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 156.85, 143.90, 137.43, 134.56, 133.67, 128.90, 126.43, 126.35, 125.84, 122.41, 113.73, 113.19, 64.75, 55.52, 55.43, 51.05, 29.69. **MS:** m/z = 269.25(M^+).

(2-(3,4-dihydroisoquinolin-2(1*H*)-yl)-6-methoxyphenyl)methanol (1f)



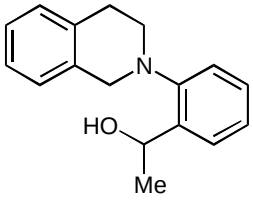
Yellow oil, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.31 – 7.23 (m, 1H), 7.17 (s, 3H), 7.06 (d, J = 7.0 Hz, 1H), 6.85 (d, J = 8.1 Hz, 1H), 6.71 (d, J = 8.3 Hz, 1H), 4.90 (s, 2H), 4.65 (s, 1H), 4.16 (s, 2H), 3.85 (s, 3H), 3.31 (t, J = 5.8 Hz, 2H), 3.04 (t, J = 5.6 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 157.93, 152.22, 134.57, 133.92, 128.82, 128.67, 126.30, 125.73, 123.60, 112.87, 106.68, 57.59, 55.54, 54.90, 51.18, 29.37. **MS:** m/z = 269.18(M^+).

(2-(3,4-dihydroisoquinolin-2(1*H*)-yl)-5-nitrophenyl)methanol (1g)[1]



Yellow solid, **mp:** 79~81°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 8.33 (s, 1H), 8.14 (d, *J* = 6.3 Hz, 1H), 7.20 (dd, *J* = 11.1, 6.0 Hz, 4H), 7.11 (d, *J* = 5.3 Hz, 1H), 4.83 (s, 2H), 4.28 (s, 2H), 3.40 (t, *J* = 5.8 Hz, 2H), 3.04 (t, *J* = 5.8 Hz, 2H), 2.99 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 155.97, 142.73, 135.07, 133.62, 133.49, 128.93, 126.73, 126.26, 126.15, 124.40, 123.83, 119.04, 61.57, 53.59, 50.55, 28.93. **MS:** m/z = 284.21(M^+).

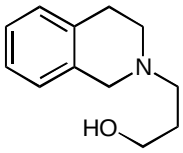
1-(2-(3,4-dihydroisoquinolin-2(1*H*)-yl)phenyl)ethanol (1h)



Yellow oil, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.32 – 7.24 (m, 3H), 7.22 – 7.13 (m, 4H), 7.04 (d, *J* = 6.8 Hz, 1H), 6.10 (s, 1H), 5.12 (q, *J* = 6.5 Hz, 1H), 4.19 (d, *J* = 15.1 Hz, 1H), 4.11 (d, *J* = 15.0 Hz, 1H), 3.28 (dd, *J* = 9.3, 5.6 Hz, 2H), 3.06 (s, 2H), 1.53 (t,

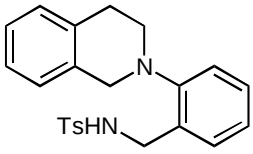
J = 5.5 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 150.36, 140.72, 134.34, 133.55, 128.83, 127.94, 126.86, 126.39, 126.28, 125.76, 125.66, 122.28, 68.45, 55.96, 51.37, 29.59, 24.29. **MS:** m/z = 253.18(M^+).

3-(3,4-dihydroisoquinolin-2(1*H*)-yl)propan-1-ol (1i)[2]



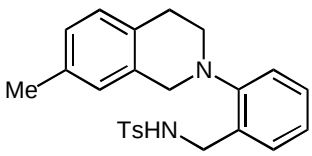
Yellow oil, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.12 (dd, *J* = 8.6, 5.3 Hz, 3H), 7.02 (d, *J* = 5.5 Hz, 1H), 3.82 (dd, *J* = 11.3, 6.1 Hz, 2H), 3.70 (s, 2H), 2.94 – 2.86 (m, 2H), 2.86 – 2.70 (m, 4H), 1.81 (dd, *J* = 10.7, 5.4 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 133.91, 133.77, 128.36, 126.28, 126.05, 125.48, 63.61, 57.79, 56.12, 50.49, 28.68, 27.53. **MS:** m/z = 191.25(M^+).

***N*-(2-(3,4-dihydroisoquinolin-2(1*H*)-yl)benzyl)-4-methylbenzenesulfonamide (1j)**



White solid, **mp:** 124~126°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.48 (d, *J* = 8.2 Hz, 2H), 7.28 – 7.21 (m, 2H), 7.20 – 7.10 (m, 4H), 7.09 – 6.98 (m, 3H), 6.94 (d, *J* = 7.4 Hz, 1H), 6.29 (t, *J* = 5.6 Hz, 1H), 4.17 (d, *J* = 5.9 Hz, 2H), 3.91 (s, 2H), 3.14 (t, *J* = 5.8 Hz, 2H), 2.92 (t, *J* = 5.6 Hz, 2H), 2.34 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 151.01, 142.86, 136.23, 134.25, 133.62, 131.07, 130.17, 129.35, 128.94, 126.75, 126.45, 126.23, 125.80, 124.85, 121.01, 55.09, 50.44, 45.65, 29.45, 21.38. **MS:** m/z = 392.24(M^+).

4-methyl-*N*-(2-(7-methyl-3,4-dihydroisoquinolin-2(1*H*)-yl)benzyl)benzenesulfonamide (1k)



White solid, **mp:** 102~105°C, **^{1}H NMR** (600 MHz, $CDCl_3$) δ (ppm) 7.48 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 7.9 Hz, 1H), 7.18 (d, *J* = 7.3 Hz, 1H), 7.12 (d, *J* = 7.7 Hz, 1H), 7.09 – 6.98 (m, 5H), 6.75 (s, 1H), 6.38 (s, 1H), 4.16 (d, *J* = 5.7 Hz, 2H), 3.87 (s, 2H), 3.13 (t, *J* = 5.7 Hz, 2H), 2.88 (s, 2H), 2.34 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 150.96, 142.73, 136.06, 135.15, 134.00, 130.92, 130.48, 130.09, 129.26, 128.88,

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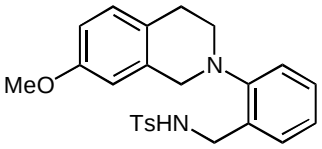
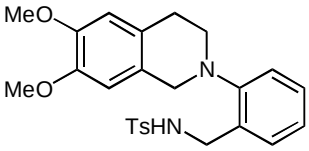
128.75, 127.26, 126.71, 124.75, 120.96, 55.02, 50.42, 45.68, 28.98, 21.35, 20.96. **MS:** m/z = 406.06(M^+).

***N*-(2-(7-methoxy-3,4-dihydroisoquinolin-2(1*H*)-yl)benzyl)-4-methylbenzenesulfonamide (1l)**



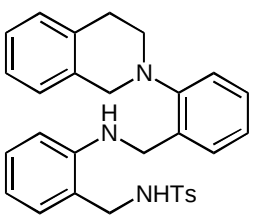
White solid, **mp:** 121~123°C, **^{1}H NMR** (600 MHz, $CDCl_3$) δ (ppm) 7.48 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 7.4 Hz, 1H), 7.12 – 7.03 (m, 5H), 6.81 (d, *J* = 8.3 Hz, 1H), 6.45 (s, 1H), 6.39 (s, 1H), 4.16 (d, *J* = 5.6 Hz, 2H), 3.86 (s, 2H), 3.80 (s, 3H), 3.13 (t, *J* = 5.6 Hz, 2H), 2.86 (t, *J* = 5.1 Hz, 2H), 2.34 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 157.59, 150.89, 142.84, 136.06, 135.23, 130.92, 130.18, 129.84, 129.32, 128.94, 126.74, 125.63, 124.84, 121.02, 112.70, 110.86, 55.13, 50.51, 45.81, 29.62, 28.56, 21.32. **MS:** m/z = 422.24(M^+).

***N*-(2-(6,7-dimethoxy-3,4-dihydroisoquinolin-2(1*H*)-yl)benzyl)-4-methylbenzenesulfonamide (1m)**



White solid, **mp:** 146~148°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.47 (d, *J* = 8.2 Hz, 2H), 7.30 – 7.22 (m, 1H), 7.15 (dd, *J* = 16.2, 7.3 Hz, 2H), 7.06 (t, *J* = 8.3 Hz, 3H), 6.68 (s, 1H), 6.56 (s, 1H), 6.41 (s, 1H), 4.16 (d, *J* = 5.3 Hz, 2H), 3.92 (s, 3H), 3.84 (s, 3H), 3.83 (s, 2H), 3.15 (t, *J* = 5.7 Hz, 2H), 2.88 (t, *J* = 5.5 Hz, 2H), 2.35 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 150.98, 147.70, 147.29, 142.82, 136.28, 131.00, 130.17, 129.33, 128.96, 126.85, 126.07, 125.56, 124.87, 121.11, 111.51, 108.96, 55.92, 55.81, 54.80, 50.25, 45.92, 29.03, 21.36. **MS:** m/z = 452.17(M^+).

***N*-(2-((2-(3,4-dihydroisoquinolin-2(1*H*)-yl)benzyl)amino)benzyl)-4-methylbenzenesulfonamide (1n)**

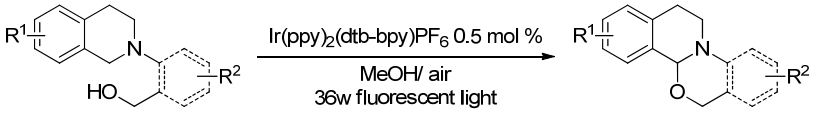


White solid, **mp:** 114~116°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.71 (d, *J* = 8.2 Hz, 2H), 7.42 (d, *J* = 7.5 Hz, 1H), 7.28 –

7.24 (m, 3H), 7.19 (dd, *J* = 18.9, 6.8 Hz, 4H), 7.11 – 7.06 (m, 3H), 6.86 (d, *J* = 7.3 Hz, 1H), 6.55 (dd, *J* = 16.3, 8.0 Hz, 2H), 4.98 (s, 1H), 4.48 (s, 1H), 4.45 (s, 2H), 4.17 (s, 2H), 3.98 (d, *J* = 6.0 Hz, 2H), 3.26 (t, *J* = 5.7 Hz, 2H), 3.02 (d, *J* = 5.3 Hz, 2H), 2.41 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 150.96, 146.73, 143.59, 135.97, 135.14, 134.33, 133.71, 130.00, 129.72, 129.56, 128.87, 128.64, 127.80, 127.14, 127.00, 126.33, 126.21, 125.69, 124.19, 124.05, 119.88, 119.00, 116.41, 111.26, 54.83, 51.08, 45.58, 43.30, 29.58, 21.47. **MS:** m/z = 497.47(M^+). **Anal. calcd for ($C_{30}H_{31}N_3O_2S$):** C, 72.40; H, 6.28; N, 8.44; S, 6.44 Found: C, 72.32; H, 6.31; N, 8.34; S, 6.44.

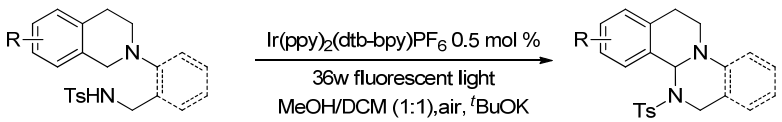
4. General Procedure and Spectral Data of Products

4.1 General procedure



1a-1i **2a-2i**

To a 5.0 mL flask equipped with a magnetic stir bar was added substrates **1** (0.3 mmol), 0.5 mol % $Ir(ppy)_2(dtb\text{-}bpy)PF_6$ (1.37 mg, 0.0015 mmol) and MeOH (1.0 mL). After, this solution was stirred open to air at a distance of ~5 cm from a 36w fluorescent lamp at room temperature. The reaction was stopped at the indicated time and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (silica: 200~300; eluant: petroleum ether/ ethyl acetate (10:1~5:1)) to provide pure product **2**.



1j-1n **2j-2o**

To a 5.0 mL flask equipped with a magnetic stir bar was added substrates **1** (0.2 mmol), 0.5 mol % $Ir(ppy)_2(dtb\text{-}bpy)PF_6$ (0.913 mg, 0.001 mmol), ^{t}BuOK (44.8 mg, 0.4 mmol) and solvent (2.0 mL). After, this solution was stirred open to air at a distance of ~5 cm from a 36w fluorescent lamp at room temperature. Upon the completion of reaction monitored by TLC, the solvent was removed under reduced

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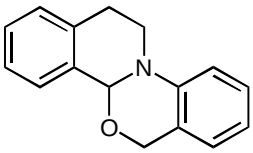
pressure. The crude product was purified by flash chromatography on silica gel (silica: 200~300; eluant: petroleum ether/ ethyl acetate (10:1~5:1)) to provide pure product **2**.

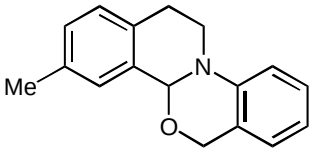
4.2 Spectral Data of Products

4b,6,12,13-tetrahydrobenzo[4,5][1,3]oxazino[2,3-*a*]isoquinoline (2a)



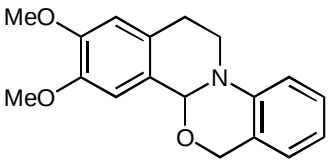
Yield: 72%, white solid, **mp**: 135~138°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.50 – 7.42 (m, 1H), 7.29 – 7.25 (m, 2H), 7.24 – 7.15 (m, 2H), 7.12 (d, *J* = 8.1 Hz, 1H), 7.02 – 6.96 (m, 2H), 5.41 (s, 1H), 5.23 (d, *J* = 14.7 Hz, 1H), 4.97 (d, *J* = 14.7 Hz, 1H), 3.61 – 3.51 (m, 1H), 3.50 – 3.42 (m, 1H), 3.11 – 3.19 (m, 1H), 2.88 – 2.94 (m, 1H).; **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 145.83, 135.18, 133.30, 128.59, 128.20, 128.18, 127.20, 126.30, 126.16, 124.85, 121.54, 121.11, 84.25, 68.17, 46.04, 29.67.; **Anal. calcd for ($C_{16}H_{15}NO$):** C, 80.98; H, 6.37; N, 5.90. Found: C, 80.72; H, 6.53; N, 5.63.

3-methyl-4b,6,12,13-tetrahydrobenzo[4,5][1,3]oxazino[2,3-*a*]isoquinoline (2b)



Yield: 65%, white solid, **mp**: 101~104°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) (dd, *J* = 17.8, 7.6 Hz, 2H), 7.12 – 7.07 (m, 3H), 7.02 – 6.95 (m, 2H), 5.38 (s, 1H), 5.22 (d, *J* = 14.7 Hz, 1H), 4.97 (d, *J* = 14.7 Hz, 1H), 3.57 – 3.40 (m, 2H), 3.14 – 3.06 (m, 1H), 2.90 – 2.84 (m, 1H), 2.35 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 145.92, 135.83, 133.06, 132.14, 129.18, 128.94, 128.07, 127.19, 126.16, 124.85, 121.50, 121.13, 84.34, 68.20, 46.21, 29.32, 21.01.; **Anal. calcd for ($C_{17}H_{17}NO$):** C, 81.24; H, 6.82; N, 5.57. Found: C, 81.44; H, 6.63; N, 5.31.

2,3-dimethoxy-4b,6,12,13-tetrahydrobenzo[4,5][1,3]oxazino[2,3-*a*]isoquinoline (2c)



Yield: 52%, white solid, **mp**: 124~127°C, ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.28 – 7.20 (m, 1H), 7.12 (d, *J* = 8.1 Hz, 1H), 7.03 – 6.96 (m, 2H), 6.92 (s, 1H), 6.66 (s, 1H), 5.34 (s, 1H), 5.21 (d, *J* = 14.7 Hz, 1H), 4.97 (d, *J* = 14.7 Hz, 1H), 3.91 (s, 3H), 3.89 (s, 3H), 3.57 – 3.40 (m, 2H), 3.12 – 3.04 (m, 1H), 2.85 – 2.79 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 148.97, 147.67, 145.83, 127.72, 127.16, 126.09, 125.23, 124.81, 121.49, 121.09, 110.75, 110.40, 84.13, 68.02, 55.86, 55.78, 46.14, 29.32.; **Anal. calcd for ($C_{18}H_{19}NO_3$):** C, 72.71; H, 6.44; N, 4.71. Found: C, 72.82; H, 6.73; N, 4.52.

8-methyl-4b,6,12,13-tetrahydrobenzo[4,5][1,3]oxazino[2,3-*a*]isoquinoline (2d)

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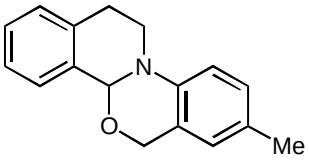
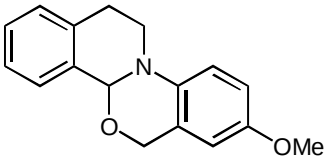


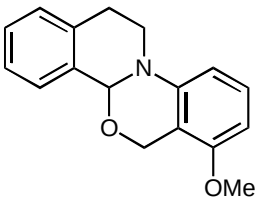
Yield: 55%, white solid, **mp**: 98~101°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.43 (d, *J* = 3.4 Hz, 1H), 7.26 (dd, *J* = 8.3, 4.7 Hz, 2H), 7.19 (d, *J* = 4.0 Hz, 1H), 7.08 – 6.98 (m, 2H), 6.83 (s, 1H), 5.37 (s, 1H), 5.18 (d, *J* = 14.7 Hz, 1H), 4.93 (d, *J* = 14.7 Hz, 1H), 3.56 – 3.34 (m, 2H), 3.20 – 3.12 (m, 1H), 2.88 (d, *J* = 16.1 Hz, 1H), 2.30 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 143.50, 135.19, 133.35, 131.41, 128.75, 128.22, 128.04, 126.34, 126.27, 125.17, 121.81, 84.42, 68.15, 46.51, 29.77, 20.73.; **Anal. calcd for ($C_{17}H_{17}NO$):** C, 81.24; H, 6.82; N, 5.57. Found: C, 81.38; H, 7.00; N, 5.54.

8-methoxy-4b,6,12,13-tetrahydrobenzo[4,5][1,3]oxazino[2,3-*a*]isoquinoline (2e)



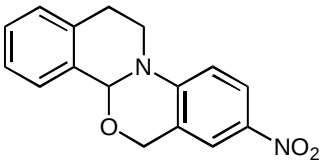
Yield: 63%, white solid, **mp**: 111~114°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.48 – 7.35 (m, 1H), 7.27 (dd, *J* = 7.9, 3.6 Hz, 2H), 7.23 – 7.15 (m, 1H), 7.08 (d, *J* = 8.8 Hz, 1H), 6.83 (dd, *J* = 8.8, 2.7 Hz, 1H), 6.55 (d, *J* = 2.5 Hz, 1H), 5.35 (s, 1H), 5.19 (d, *J* = 14.9 Hz, 1H), 4.94 (d, *J* = 14.9 Hz, 1H), 3.79 (s, 3H), 3.49 – 3.42 (m, 1H), 3.38 – 3.34 (m, 1H), 3.26 – 3.13 (m, 1H), 2.86 (d, *J* = 16.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) 155.01, 139.43, 135.13, 133.28, 128.87, 128.25, 127.79, 126.23, 123.98, 114.00, 108.84, 84.54, 68.09, 55.43, 47.04, 29.84.; **Anal. calcd for ($C_{17}H_{17}NO_2$):** C, 76.38; H, 6.41; N, 5.24 Found: C, 76.22; H, 6.69; N, 5.15.

7-methoxy-4b,6,12,13-tetrahydrobenzo[4,5][1,3]oxazino[2,3-*a*]isoquinoline (2f)



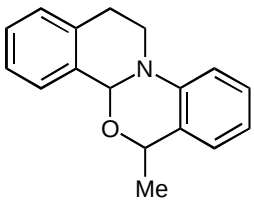
Yield: 61%, white solid, **mp**: 82~84°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.53 – 7.41 (m, 1H), 7.30 – 7.23 (m, 2), 7.20 (t, *J* = 8.0 Hz, 2H), 6.76 (d, *J* = 8.2 Hz, 1H), 6.49 (d, *J* = 8.1 Hz, 1H), 5.36 (s, 1H), 5.14 – 4.92 (m, 2H), 3.82 (s, 3H), 3.57 – 3.44 (m, 2H), 3.18 – 3.10 (m, 1H), 2.96 – 2.85 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 155.90, 147.00, 135.21, 133.36, 128.71, 128.15, 127.44, 126.29, 115.05, 113.50, 102.68, 83.80, 65.24, 55.15, 45.95, 29.72.; **Anal. calcd for ($C_{17}H_{17}NO_2$):** C, 76.38; H, 6.41; N, 5.24 Found: C, 76.36; H, 6.69; N, 5.06.

8-nitro-4b,6,12,13-tetrahydrobenzo[4,5][1,3]oxazino[2,3-*a*]isoquinoline (2g) [1]



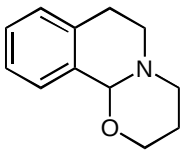
Yield: 26%, yellow solid, **mp**: 175~177°C (lit. 179~180 °C), **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) δ 8.09 (dd, *J* = 9.1, 2.3 Hz, 1H), 7.88 (s, 1H), 7.61 – 7.51 (m, 1H), 7.36 – 7.28 (m, 2H), 7.24 – 7.17 (m, 1H), 6.87 (d, *J* = 9.2 Hz, 1H), 5.73 (s, 1H), 5.18 (d, *J* = 14.5 Hz, 1H), 5.00 (d, *J* = 14.5 Hz, 1H), 3.79 – 3.73 (m, 1H), 3.67 – 3.56 (m, 1H), 3.15 – 3.05 (m, 1H), 3.02 – 2.93 (m, 1H).; **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 149.24, 138.36, 134.75, 133.08, 128.60, 127.85, 126.94, 126.67, 124.62, 121.42, 121.11, 112.28, 83.76, 67.72, 43.12, 28.41.; **Anal. calcd for ($C_{16}H_{14}N_2O_3$):** C, 68.07; H, 5.00; N, 9.92 Found: C, 68.04; H, 5.27; N, 9.77.

6-methyl-4b,6,12,13-tetrahydrobenzo[4,5][1,3]oxazino[2,3-*a*]isoquinoline (2h)



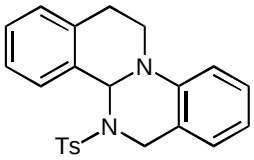
Yield: 41%, white solid, **mp**: 74~77°C, **diastereomer ratio**: 2:1. **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.51 (dd, *J* = 12.7, 8.0 Hz, 1.5H), 7.37 – 7.23 (m, 6H), 7.15 (d, *J* = 19.1 Hz, 3H), 7.05 (dd, *J* = 14.6, 7.3 Hz, 1.5H), 5.57 (s, 0.5H), 5.54 (s, 1H), 5.43 – 5.39 (m, 1H), 5.19 5.14 (m, 0.5H), 3.65 – 3.59 (m, 1.5H), 3.50 (d, *J* = 11.7 Hz, 1.5H), 3.31 – 3.12 (m, 1.5H), 2.98 (d, *J* = 16.0 Hz, 1.5H), 1.79 (d, *J* = 6.6 Hz, 1.5H), 1.67 (d, *J* = 6.4 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 145.93, 145.48, 135.12, 133.49, 131.66, 130.65, 128.71, 128.45, 128.23, 128.19, 128.02, 127.28, 127.22, 126.35, 126.09, 125.01, 121.80, 121.51, 121.29, 120.72, 83.84, 77.97, 73.81, 71.03, 46.38, 45.59, 29.78, 29.54, 22.58, 21.97.; **Anal. calcd for ($C_{17}H_{17}NO$):** C, 81.24; H, 6.82; N, 5.57. Found: C, 81.01; H, 7.11; N, 5.35.

2,3,4,6,7,11b-hexahydro-[1,3]oxazino[2,3-*a*]isoquinoline (2i)[2]



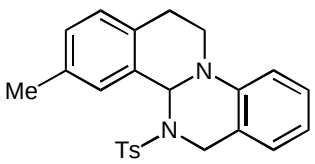
Yield: 50%, colorless oil, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.40 – 7.28 (m, 1H), 7.21 – 7.16 (m, 2H), 7.09 (d, *J* = 7.7 Hz, 1H), 4.90 (s, 1H), 4.21 (dd, *J* = 11.1, 4.6 Hz, 1H), 3.99 – 3.80 (m, 1H), 3.33 – 3.21 (m, 1H), 3.19 3.09 (m, 1H), 3.04 – 2.92 (m, 2H), 2.90 – 2.80 (m, 1H), 2.69 – 2.63 (m, 1H), 2.26 – 2.05 (m, 1H), 1.35 (d, J = 13.2 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 134.70, 134.60, 128.28, 127.79, 127.20, 125.91, 89.78, 68.31, 53.09, 46.03, 28.94, 22.81.

5-tosyl-5,6,12,13-tetrahydro-4b*H*-isoquinolino[2,1-*a*]quinazoline (2j)



Yield: 70%, white solid, **mp**: 177~179°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.68 (d, *J* = 8.2 Hz, 2H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.30 – 7.22 (m, 1H), 7.18 (t, *J* = 7.4 Hz, 1H), 7.01 (dd, *J* = 12.9, 7.9 Hz, 3H), 6.94 (t, *J* = 7.7 Hz, 1H), 6.70 – 6.64 (m, 2H), 6.54 (t, *J* = 7.3 Hz, 1H), 6.40 (s, 1H), 4.52 (d, *J* = 17.4 Hz, 1H), 4.25 (d, *J* = 17.5 Hz, 1H), 4.11 (dd, *J* = 14.6, 5.4 Hz, 1H), 3.52 – 3.37 (m, 1H), 3.10 – 2.92 (m, 1H), 2.49 (dd, *J* = 16.8, 3.5 Hz, 1H), 2.27 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 143.19, 141.68, 136.29, 136.09, 134.83, 128.96, 128.73, 127.89, 127.64, 127.59, 127.11, 126.02, 125.48, 118.67, 118.12, 113.53, 69.19, 44.97, 42.40, 24.16, 21.35.; **Anal. calcd for ($C_{23}H_{22}N_2O_2S$):** C, 70.74; H, 5.68; N, 7.17; S, 8.21. Found: C, 70.56; H, 5.89; N, 7.13; S, 8.18.

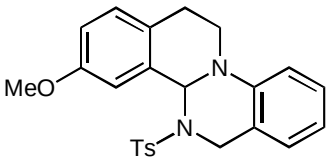
3-methyl-5-tosyl-5,6,12,13-tetrahydro-4b*H*-isoquinolino[2,1-*a*]quinazoline (2k)



Yield: 86%, white solid, **mp**: 163~165°C, **^{1}H NMR** (600 MHz, $CDCl_3$) δ (ppm) 7.67 (d, *J* = 8.2 Hz, 2H), 7.39 (s, 1H), 7.02 (d, *J* = 8.0 Hz, 2H), 6.99 (d, *J* = 7.5 Hz, 1H), 6.95 – 6.85 (m, 2H), 6.65 (dd, *J* = 11.4, 8.1 Hz, 2H), 6.53 (t, *J* = 7.4 Hz, 1H), 6.36 (s, 1H), 4.53 (d, *J* = 17.2 Hz, 1H), 4.27 (d, *J* = 17.4 Hz, 1H), 4.09 (dd, *J* = 14.7, 5.2 Hz, 1H), 3.46 – 3.32 (m, 1H), 2.95 (t, *J* = 12.0 Hz, 1H), 2.44 (d, J =

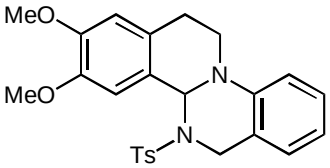
13.6 Hz, *1*H), 2.32 (s, 3H), 2.27 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 143.11, 141.61, 136.69, 135.94, 134.43, 133.10, 128.78, 128.62, 127.50, 125.92, 125.56, 118.51, 117.96, 113.46, 69.15, 44.96, 42.40, 23.72, 21.30, 21.10.; **Anal. calcd for ($C_{24}H_{24}N_2O_2S$):** C, 71.26; H, 5.98; N, 6.93; S, 7.93. Found: C, 71.14; H, 6.11; N, 6.65; S, 7.96.

3-methoxy-5-tosyl-5,6,12,13-tetrahydro-4b*H*-isoquinolino[2,1-*a*]quinazoline (2l)



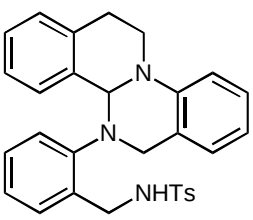
Yield: 80%, white solid, **mp**: 172~175°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.67 (d, *J* = 8.2 Hz, 2H), 7.11 (s, 1H), 7.03 (d, *J* = 8.1 Hz, 2H), 6.92 (t, *J* = 8.1 Hz, 2H), 6.78 – 6.71 (m, 1H), 6.70 – 6.61 (m, 2H), 6.54 (t, *J* = 7.4 Hz, 1H), 6.35 (s, 1H), 4.54 (d, *J* = 17.6 Hz, 1H), 4.28 (d, *J* = 17.4 Hz, 1H), 4.09 (dd, *J* = 14.7, 5.1 Hz, 1H), 3.80 (s, 3H), 3.45 – 3.31 (m, 1H), 2.99 – 2.84 (m, 1H), 2.50 – 2.37 (m, 1H), 2.27 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 158.58, 143.22, 141.59, 135.93, 135.83, 130.03, 128.68, 128.13, 127.60, 125.97, 118.59, 118.08, 114.61, 113.54, 109.36, 69.24, 55.38, 45.08, 42.49, 23.33, 21.36.; **Anal. calcd for ($C_{24}H_{24}N_2O_3S$):** C, 68.55; H, 5.75; N, 6.66; S, 7.63. Found: C, 68.35; H, 5.81; N, 6.37; S, 7.70.

2,3-dimethoxy-5-tosyl-5,6,12,13-tetrahydro-4b*H*-isoquinolino[2,1-*a*]quinazoline (2m)



Yield: 75%, white solid, **mp**: 210~212°C, **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm) 7.66 (d, *J* = 8.2 Hz, 2H), 7.02 (d, *J* = 7.6 Hz, 3H), 6.93 (t, *J* = 7.7 Hz, 1H), 6.71 – 6.61 (m, 2H), 6.55 (t, *J* = 7.3 Hz, 1H), 6.47 (s, 1H), 6.33 (s, 1H), 4.54 (d, *J* = 17.4 Hz, 1H), 4.25 (d, *J* = 17.3 Hz, 1H), 4.09 (dd, *J* = 14.6, 5.1 Hz, 1H), 3.88 (s, 3H), 3.79 (s, 3H), 3.42 – 3.38 (m, 1H), 3.01 – 2.82 (m, 1H), 2.37 (dd, *J* = 16.4, 3.3 Hz, 1H), 2.27 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 148.61, 148.12, 143.17, 141.68, 136.05, 128.65, 128.49, 127.58, 126.48, 125.96, 118.74, 118.09, 113.67, 111.39, 107.62 , 69.03, 56.06, 55.76, 45.14, 42.47, 23.77, 21.32.; **Anal. calcd for ($C_{25}H_{26}N_2O_4S$):** C, 66.64; H, 5.82; N, 6.22; S, 7.12. Found: C, 66.57; H, 6.08; N, 6.01; S, 7.28.

N-(2-(12,13-dihydro-4b*H*-isoquinolino[2,1-*a*]quinazolin-5(6*H*)-yl)benzyl)-4-methylbenzenesulfonamide (2o)



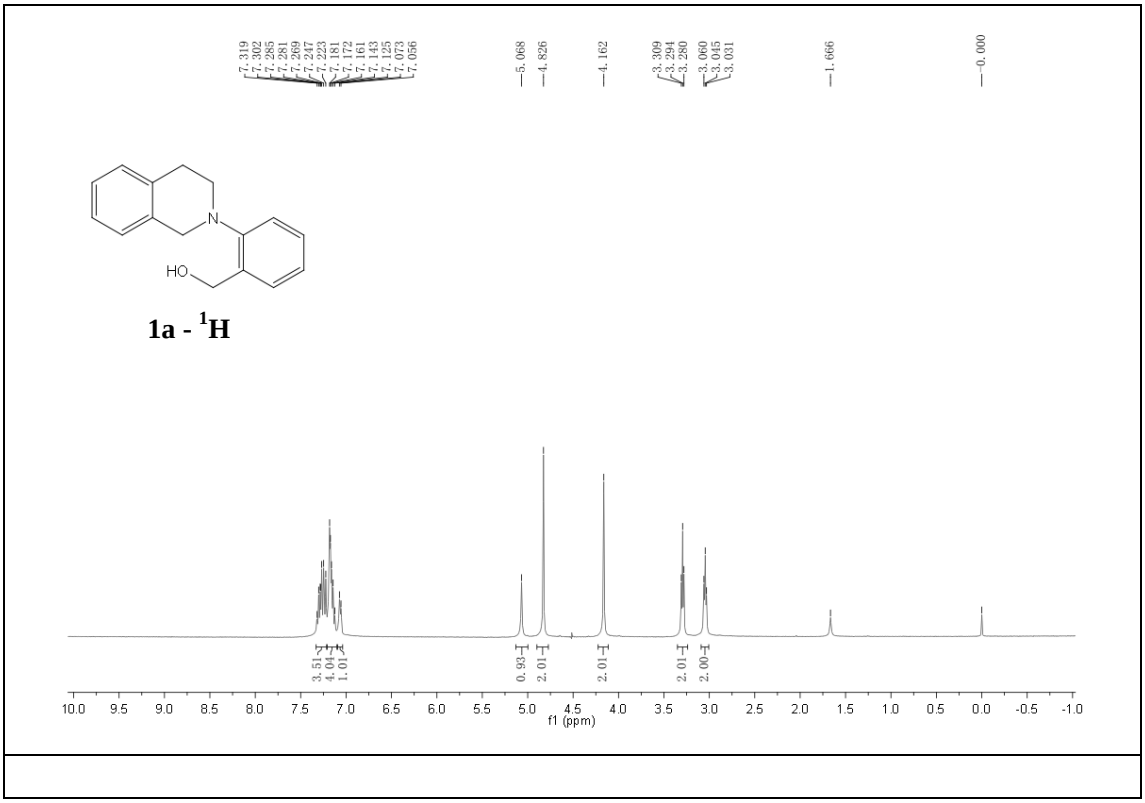
Yield: 56%, white solid, **mp**: 171~173°C, **^{1}H NMR** (600 MHz, $CDCl_3$) δ (ppm) 7.73 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 7.4 Hz, 1H), 7.33 (d, *J* = 7.7 Hz, 1H), 7.25 (d, *J* = 6.8 Hz, 3H), 7.19 – 7.07 (m, 4H), 7.00 (dd, *J* = 15.1, 7.9 Hz, 3H), 6.76 (d, *J* = 7.3 Hz, 1H), 6.67 (t, *J* = 7.3 Hz, 1H), 5.34 (s, 1H), 5.00 (s, 1H), 4.42 (dd, *J* = 13.8, 5.8 Hz, 1H), 4.36 – 4.25 (m, 2H), 4.20 (dd, *J* = 14.0, 5.2 Hz, 1H), 3.80 (d, *J* = 16.6 Hz, 1H), 3.39 – 3.28 (m, 1H), 3.10 (t, *J* = 11.6 Hz, 1H),

2.52 (d, *J* = 14.0 Hz, 1H), 2.41 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ (ppm) 149.72, 143.54, 143.27, 136.65, 136.53, 136.42, 131.00, 129.61, 129.03, 128.93, 128.68, 127.54, 127.42, 127.09, 126.77, 126.06, 125.76, 124.66, 123.58, 121.48, 118.07, 113.28, 74.63, 44.72, 43.58, 29.64, 24.87, 21.52.; **Anal. calcd for ($C_{30}H_{29}N_3O_2S$):** C, 72.70; H, 5.90; N, 8.48; S, 6.47 Found: C, 72.72; H, 6.15; N, 8.19; S, 6.34.

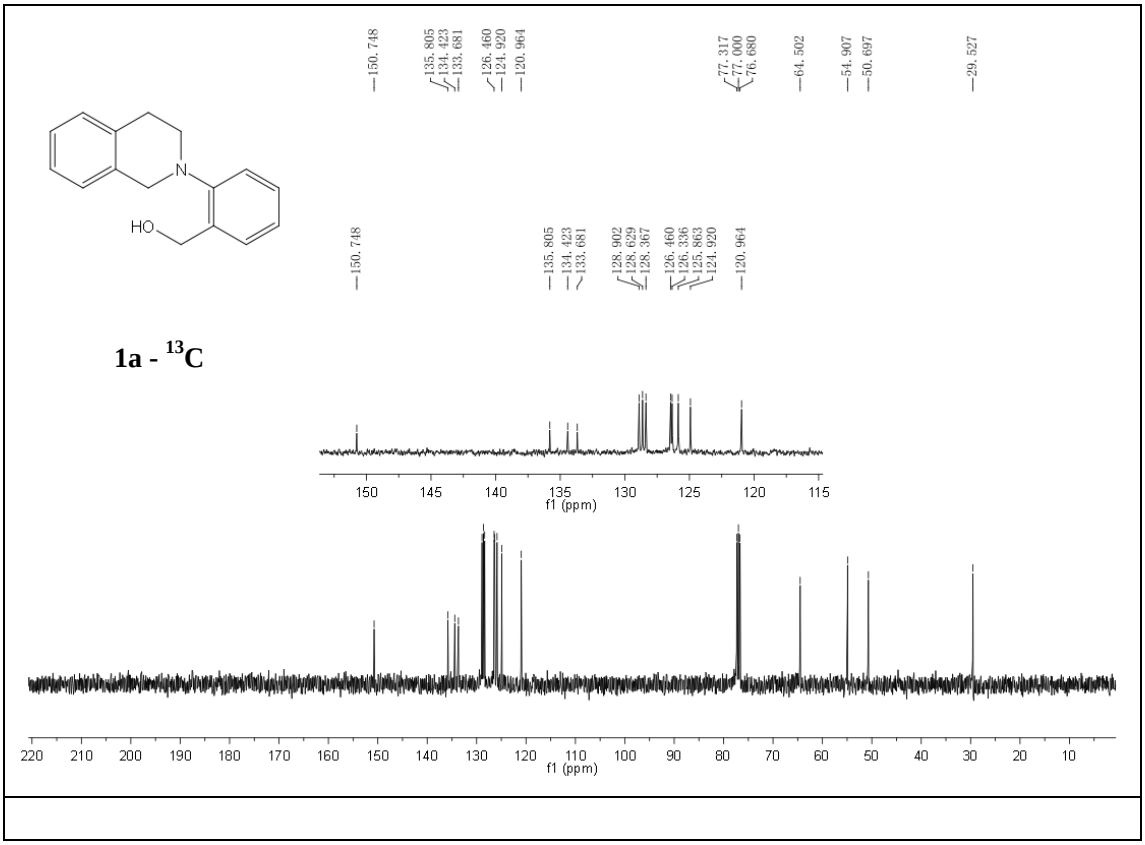
References:

1. S. P. Stanforth, *Tetrahedron*, 2000, **56**, 461.
2. G. Kumaraswamy, A. N. Murthy and A. Pitchaiah, *J. Org. Chem.*, 2010, **75**, 3916.

5. Copies of ^{1}H NMR, ^{13}C NMR Spectrums



1a - ^{1}H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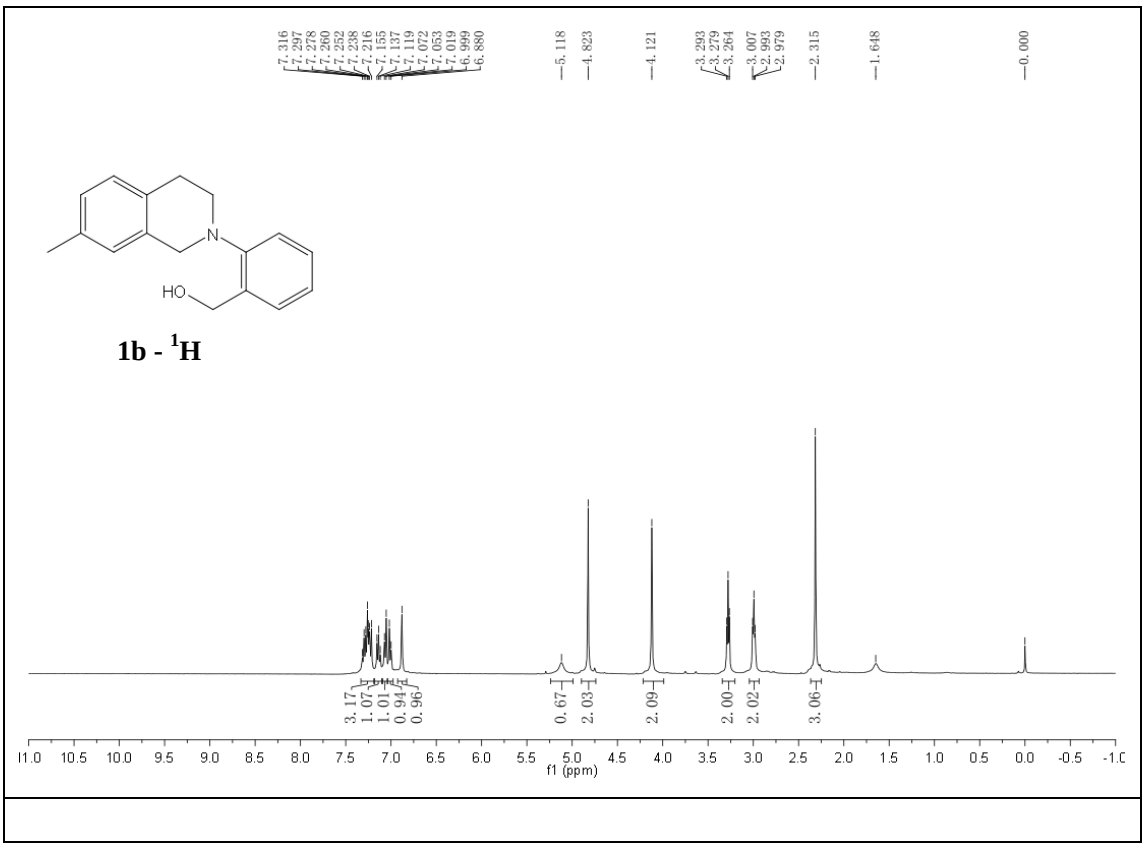
135.805
134.423
133.681
126.460
124.920
120.964
77.317
77.000
76.680
64.502
54.907
50.697
29.527
HO
N
f1 (ppm)

1a - ^{13}C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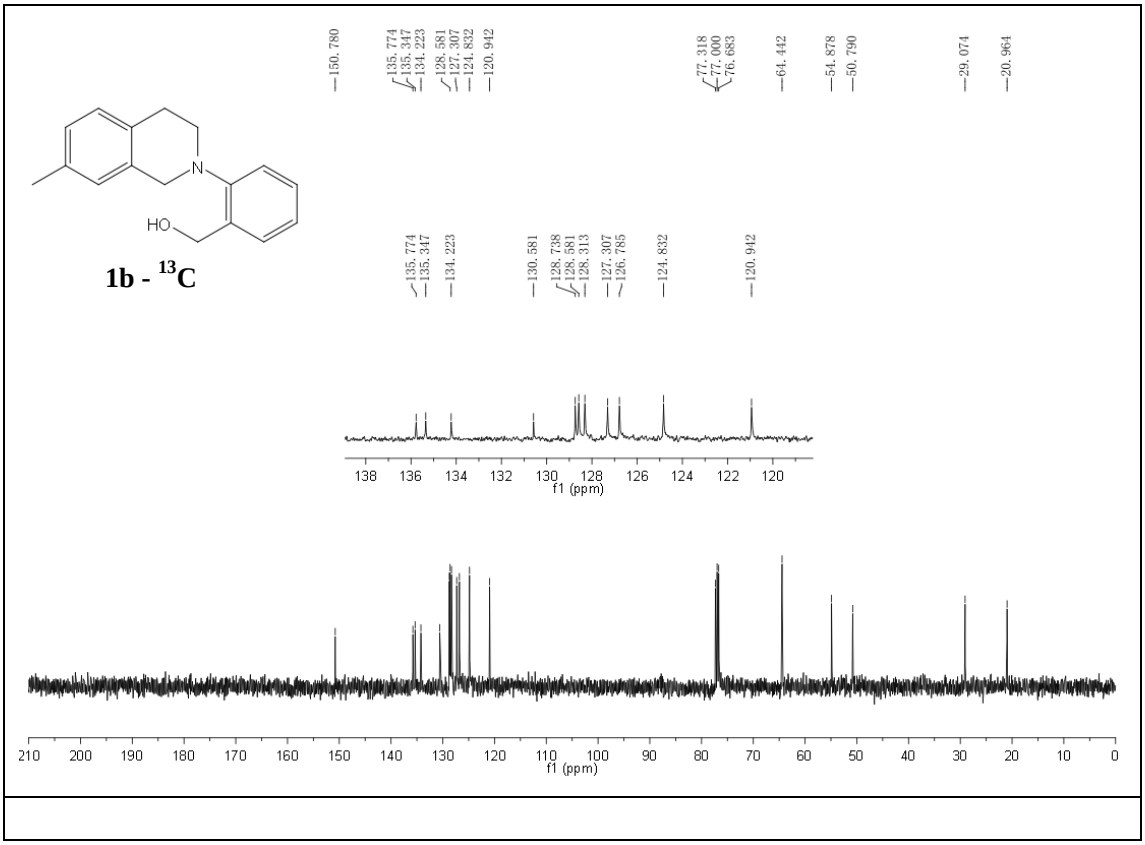
7.297
7.278
7.260
7.252
7.238
7.216
7.155
7.137
7.119
7.072
7.053
7.019
6.999
6.880
5.118
4.823
4.121
3.293
3.279
3.264
3.007
2.993
2.979
2.315
1.648
0.000
HO
N
3.17
1.07
1.01
0.94
0.96
0.67
2.03
2.09
2.00
2.02
3.06
f1 (ppm)

1b - ^{1}H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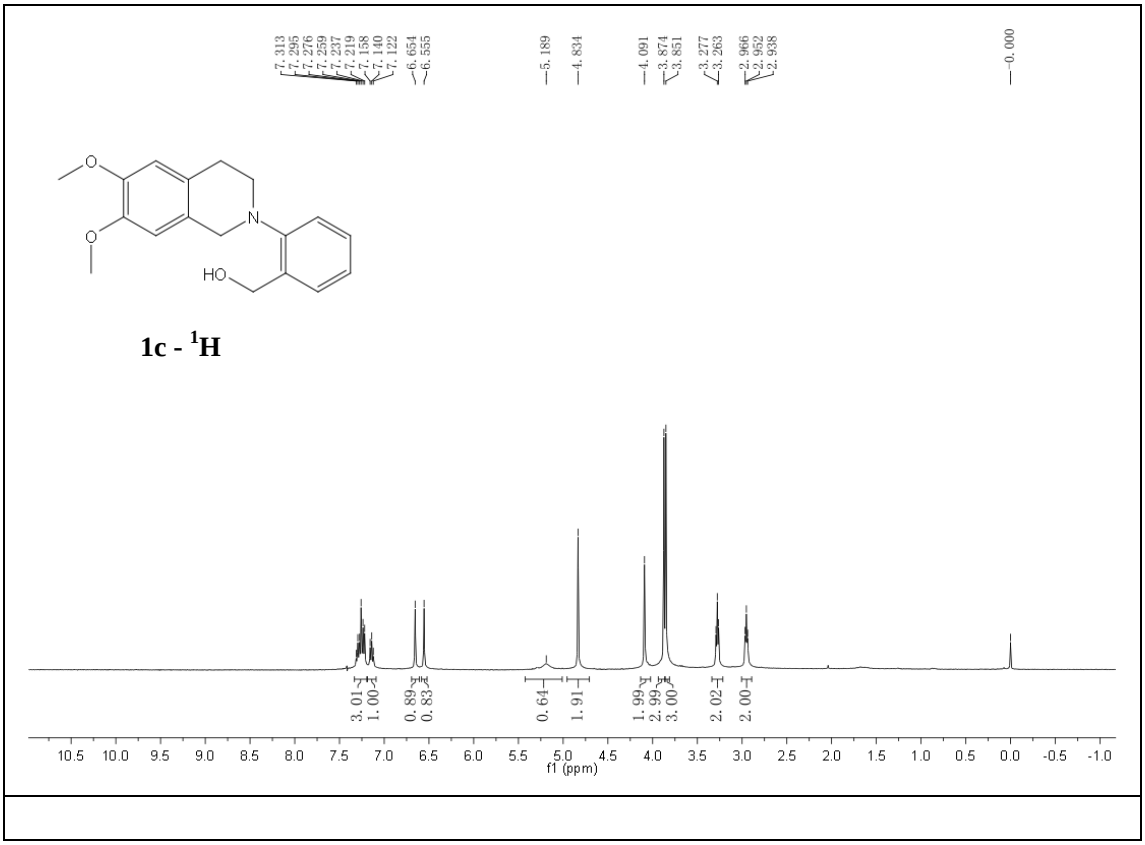
135.774
135.347
134.223
128.581
127.307
124.832
120.942
77.318
77.000
76.683
64.442
54.878
50.790
29.074
20.964
HO
1b - ^{13}C
f1 (ppm)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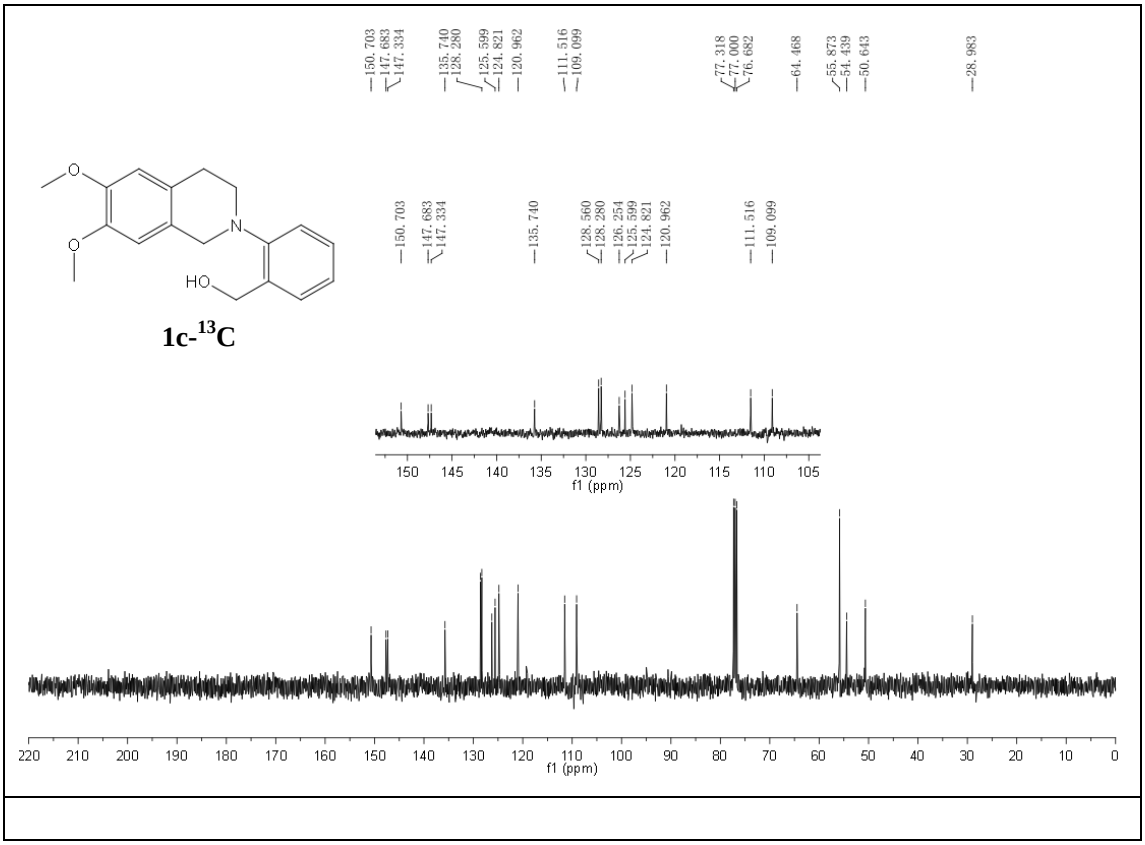
7.296
7.276
7.259
7.237
7.219
7.158
7.140
7.122
6.654
6.555
5.189
4.834
4.091
3.874
3.851
3.277
3.263
2.966
2.952
2.938
0.000
O
O
N
HO
1c - ^{1}H
3.01
1.00
0.89
0.83
0.64
1.91
1.99
2.99
3.00
2.02
2.00
f1 (ppm)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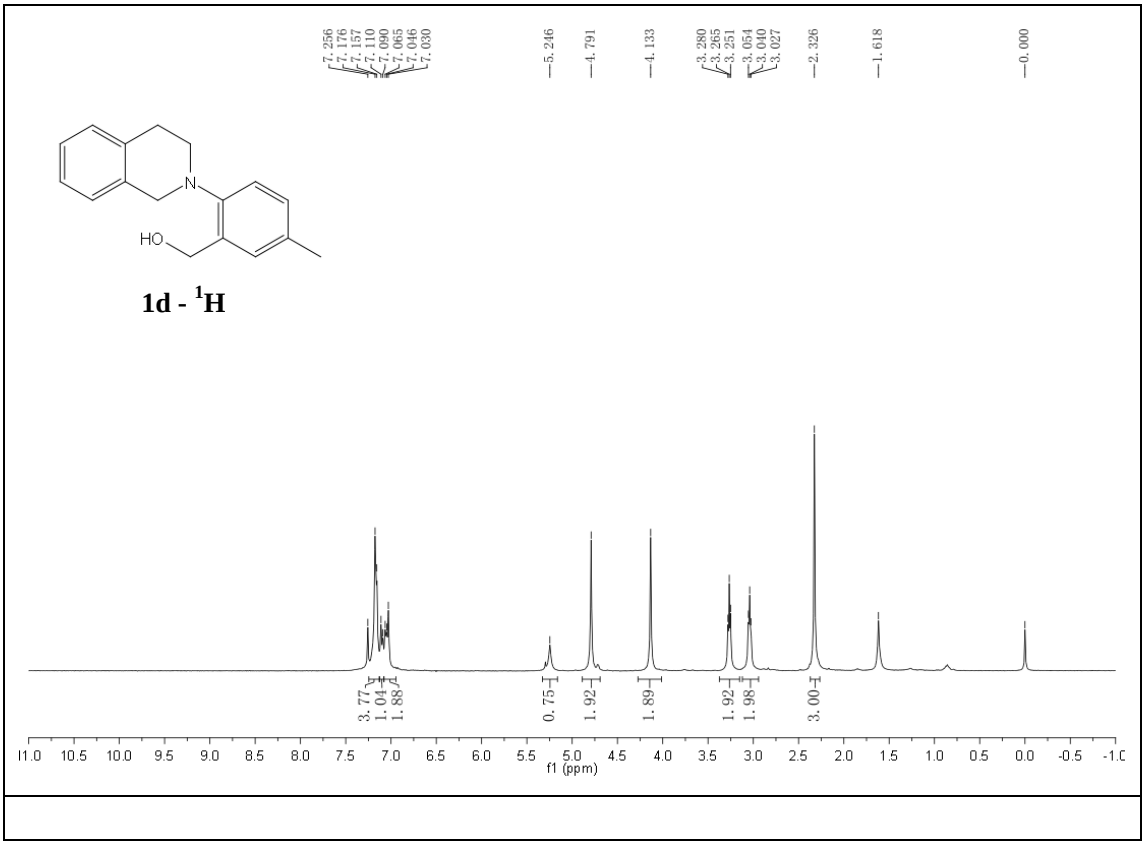
147.683
147.334
135.740
128.280
125.599
124.821
120.962
111.516
109.099
77.318
77.000
76.682
64.468
55.873
54.439
50.643
28.983
O
O
N
HO
f1 (ppm)

1c-^{13}C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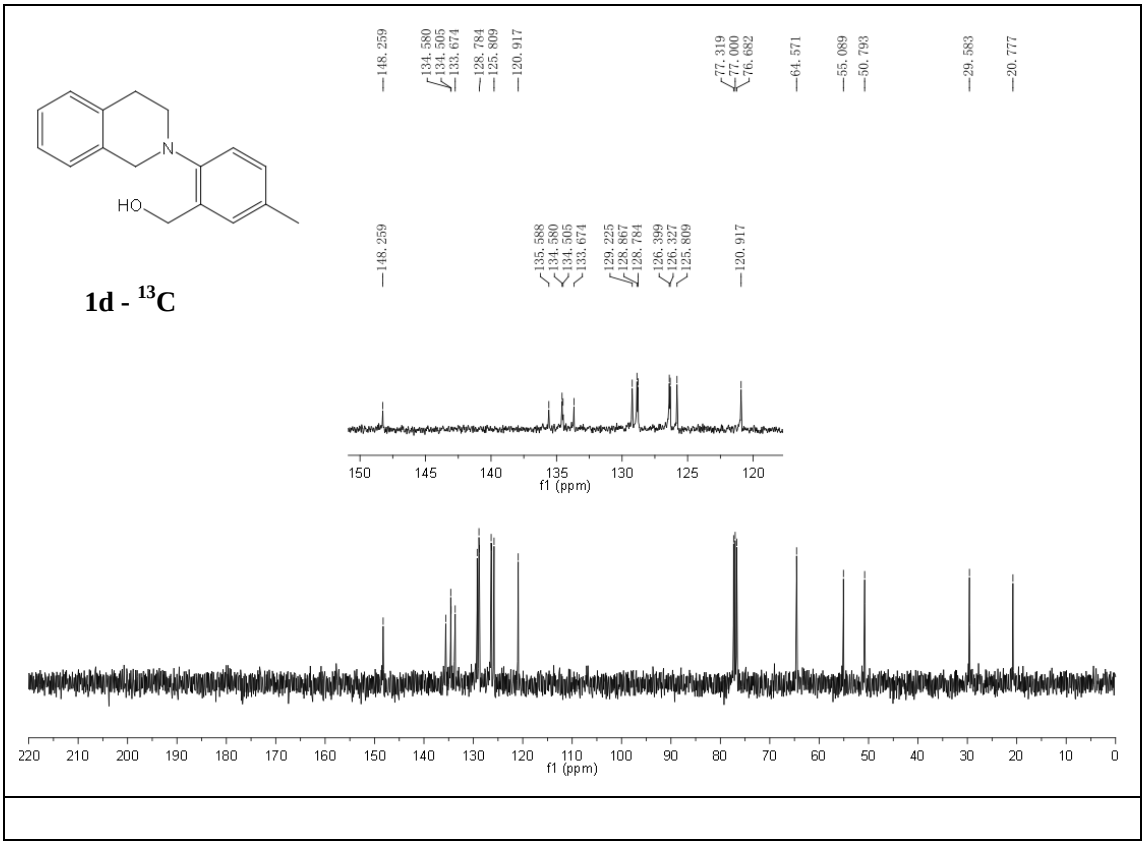
7.176
7.157
7.110
7.090
7.065
7.046
7.030
5.246
4.791
4.133
3.280
3.265
3.251
3.054
3.040
3.027
2.326
1.618
0.000
N
HO
3.77
1.04
1.88
0.75
1.92
1.89
1.92
1.98
3.00
f1 (ppm)

1d - ^{1}H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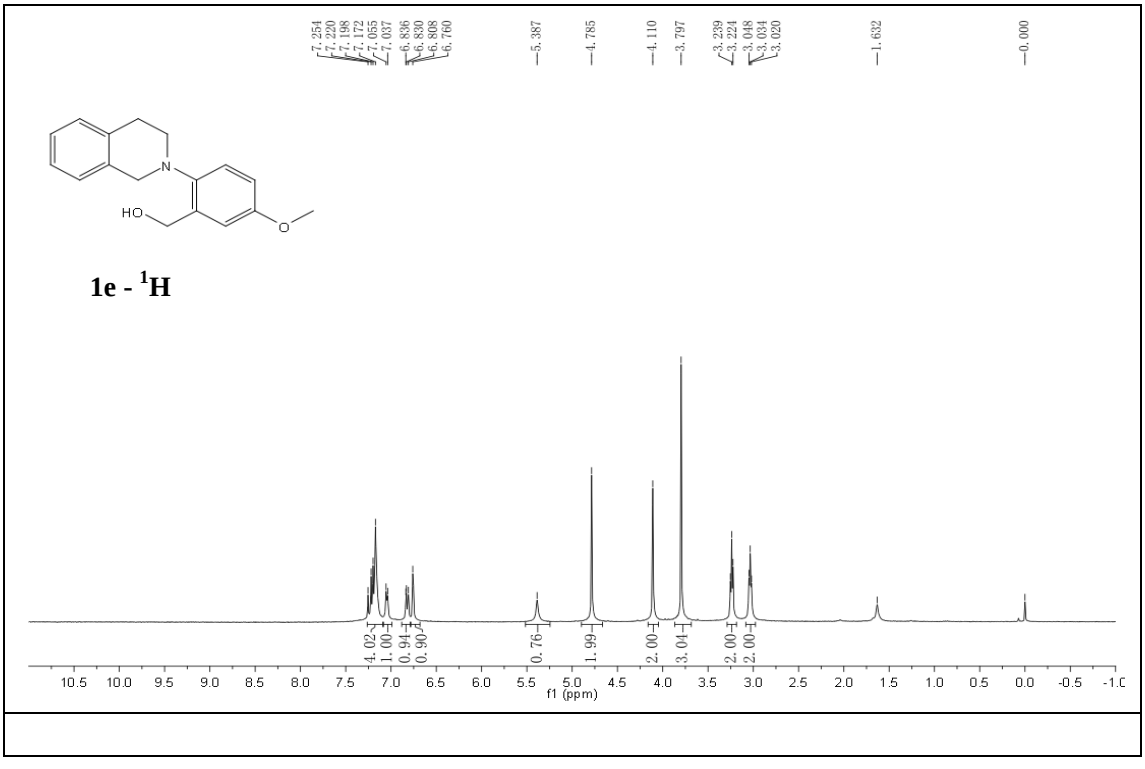
134.505
133.674
128.784
125.809
120.917
77.319
77.000
76.682
64.571
55.089
50.793
29.583
20.777
HO
N
1d - 13C
135.588
134.580
134.505
133.674
129.225
128.867
128.784
126.399
126.327
125.809
f1 (ppm)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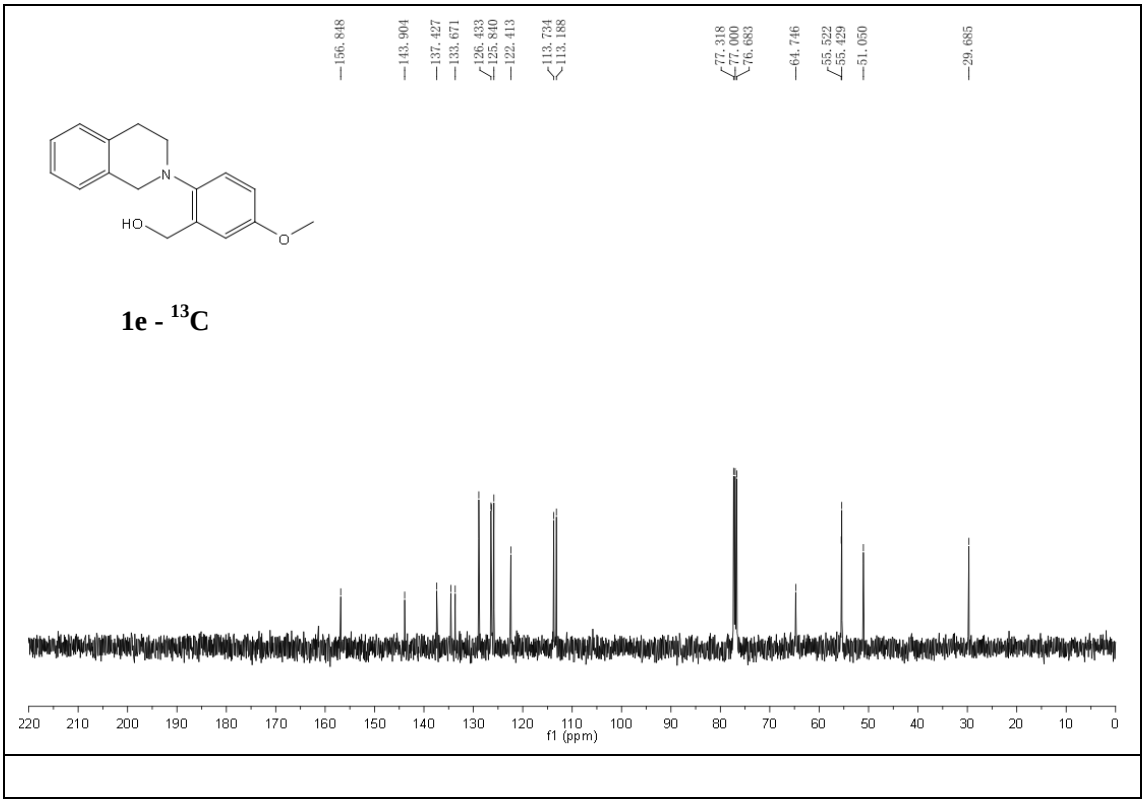
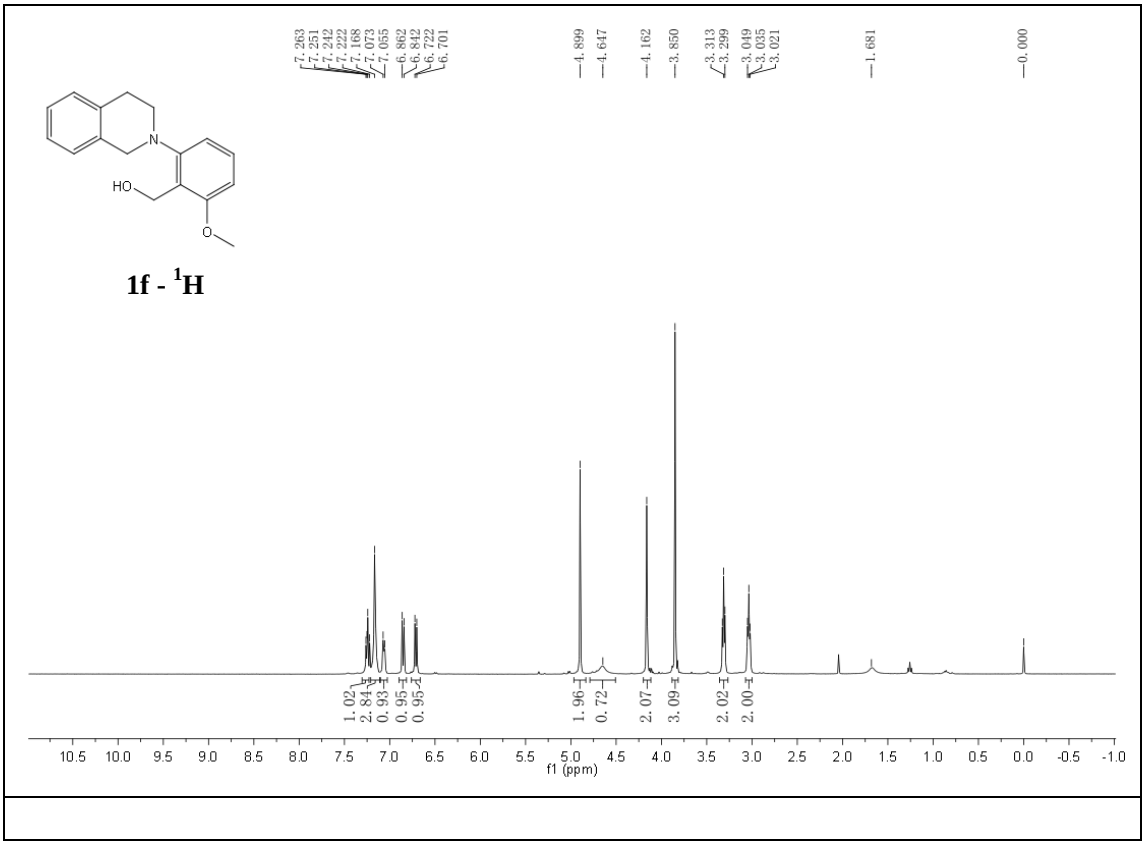
7.220
7.198
7.172
7.055
7.037
6.836
6.830
6.808
6.760
5.387
4.785
4.110
3.797
3.239
3.224
3.048
3.034
3.020
1.632
0.000
HO
O
N
1e - 1H
4.02
1.00
0.94
0.90
0.76
1.99
2.00
3.04
2.00
2.00
f1 (ppm)


156.848
143.904
137.427
133.671
126.433
125.840
122.413
113.734
113.188
77.318
77.000
76.683
64.746
55.522
55.429
51.050
29.685
HO
O
N
f1 (ppm)

1e - ^{13}C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6.842
6.722
6.701
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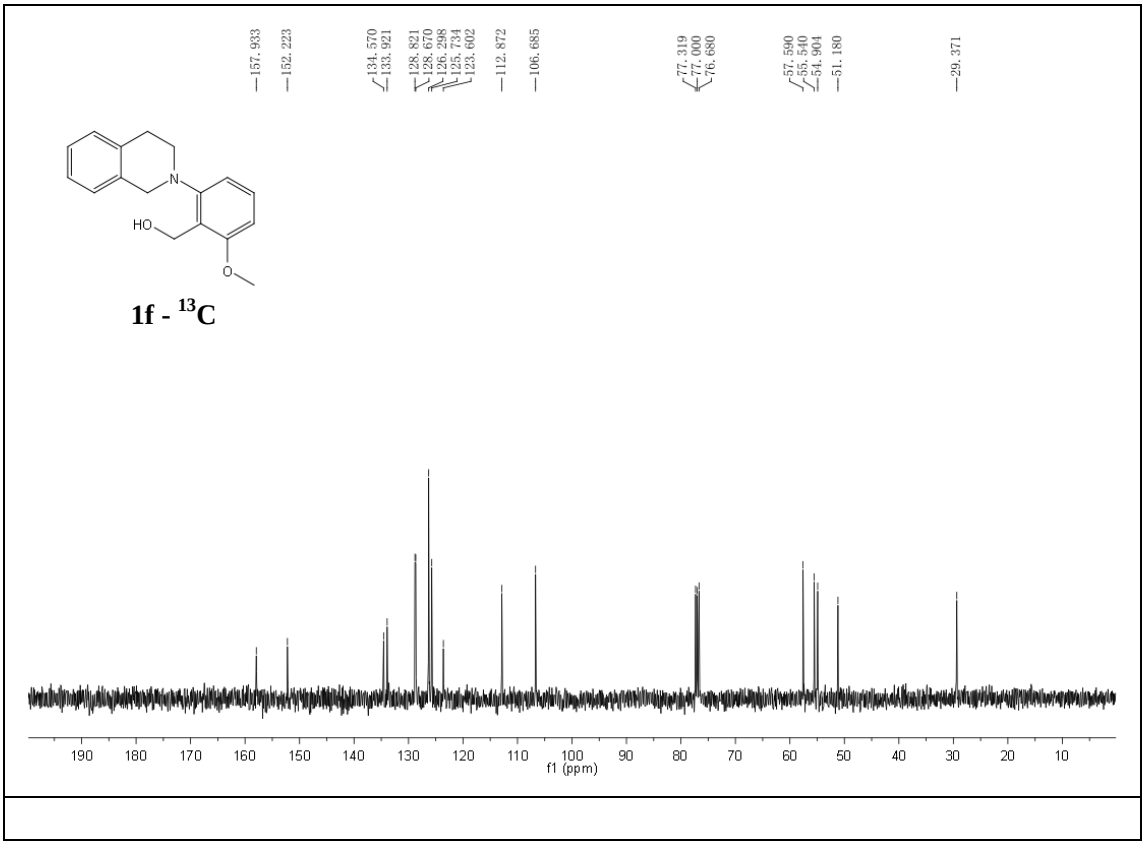
4.162
3.850
3.313
3.299
3.049
3.035
3.021
1.681
0.000
HO
O
N
1.02
2.84
0.93
0.95
0.95
1.96
0.72
2.07
3.09
2.02
2.00
f1 (ppm)

1f - ^{1}H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128.670
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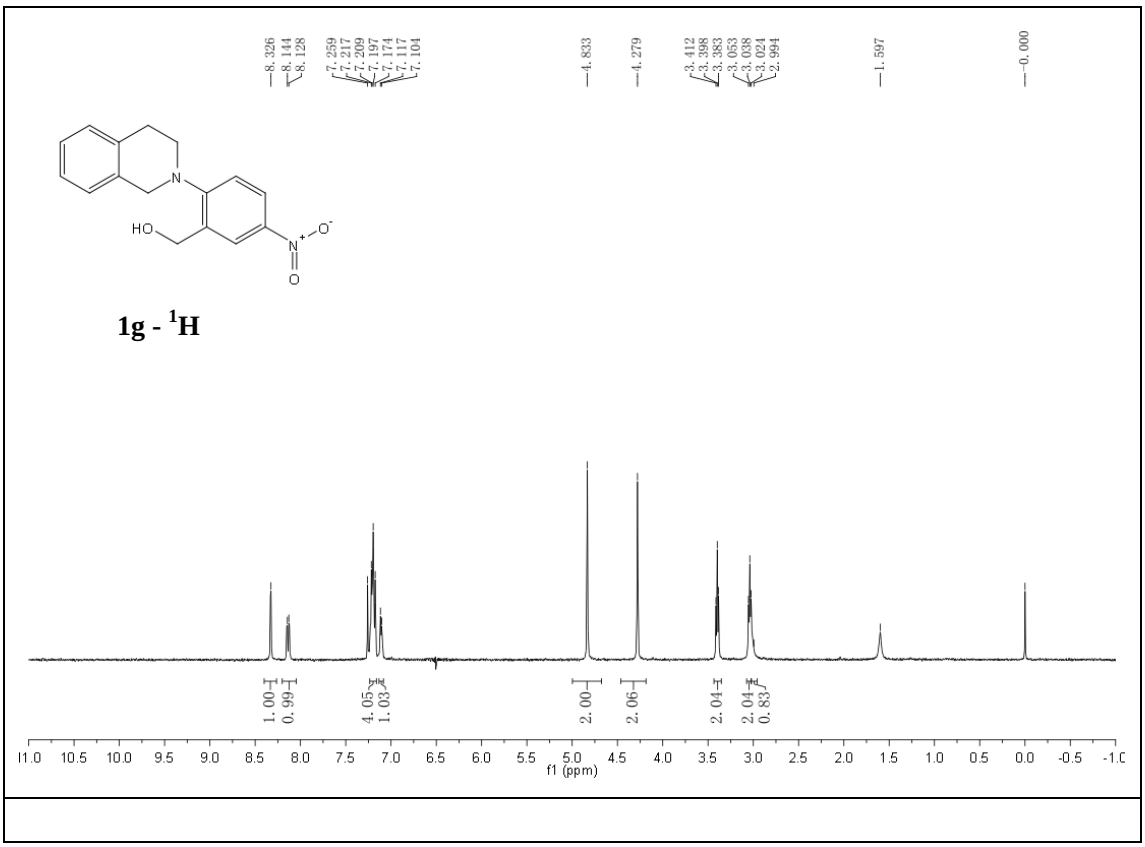
125.734
123.602
112.872
106.685
77.319
77.000
76.680
57.590
55.540
54.904
51.180
29.371
HO
N
O
f1 (ppm)

1f - ^{13}C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7.209
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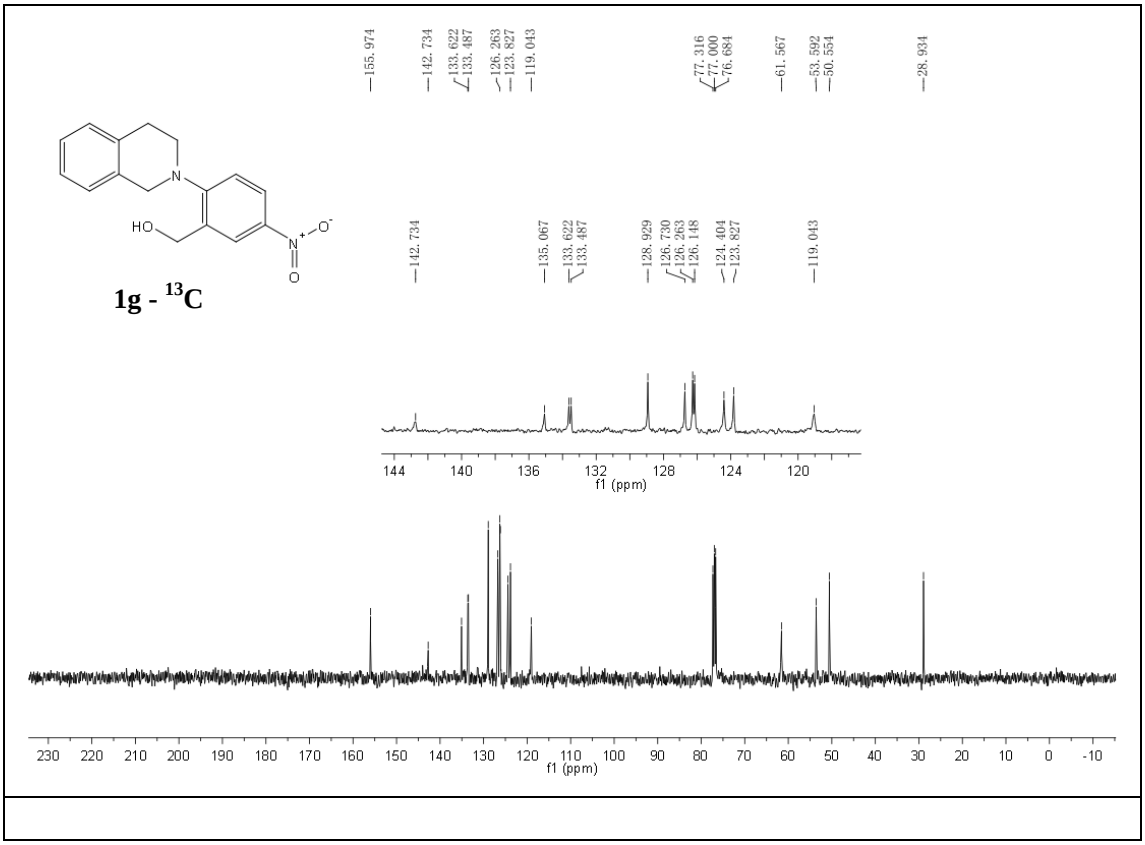
7.174
7.117
7.104
4.833
4.279
3.412
3.398
3.383
3.053
3.038
3.024
2.994
1.597
0.000
HO
N
O
1.00
0.99
4.05
1.03
2.00
2.06
2.04
2.04
0.83
f1 (ppm)

1g - ^{1}H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133.622
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126.263
123.827
119.043
77.316
77.000
76.684
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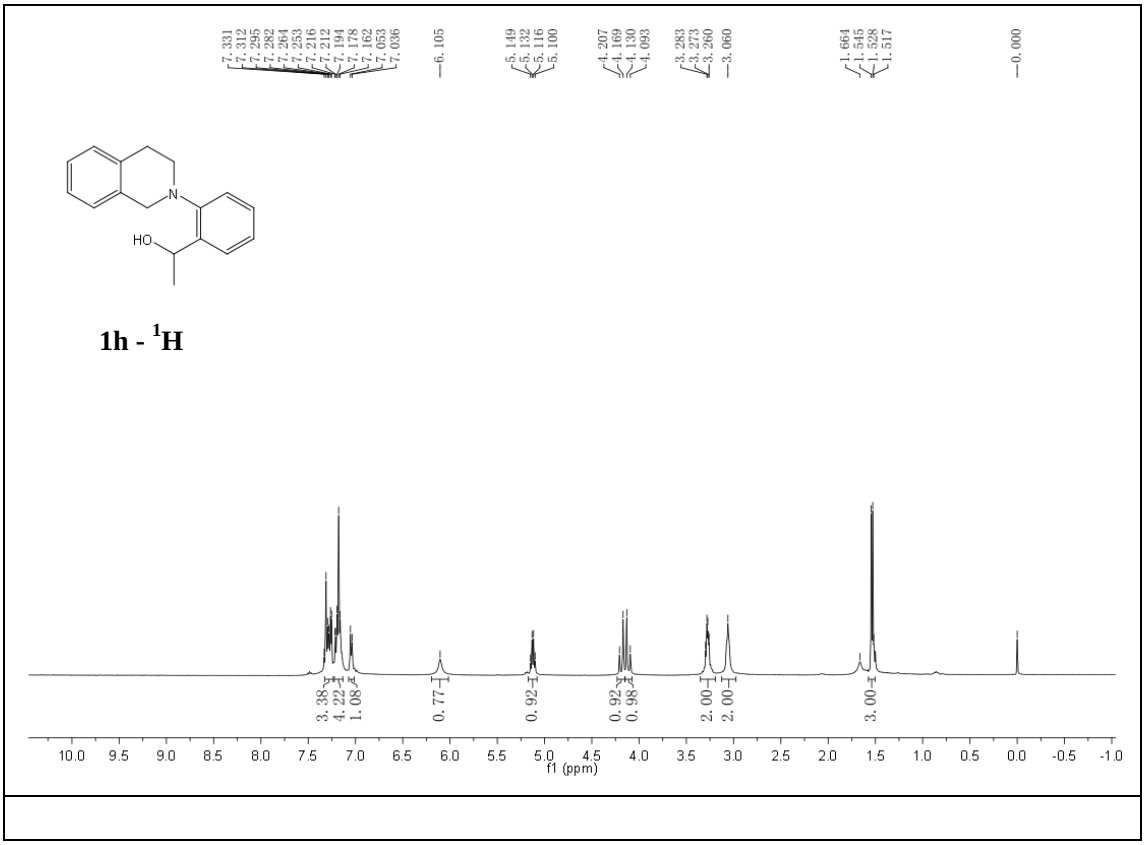
53.592
50.554
28.934
HO
N
O
O
142.734
135.067
133.622
133.487
128.929
126.730
126.263
126.148
124.404
123.827
119.043
144
140
136
132
128
124
120
f1 (ppm)
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

1g - ^{13}C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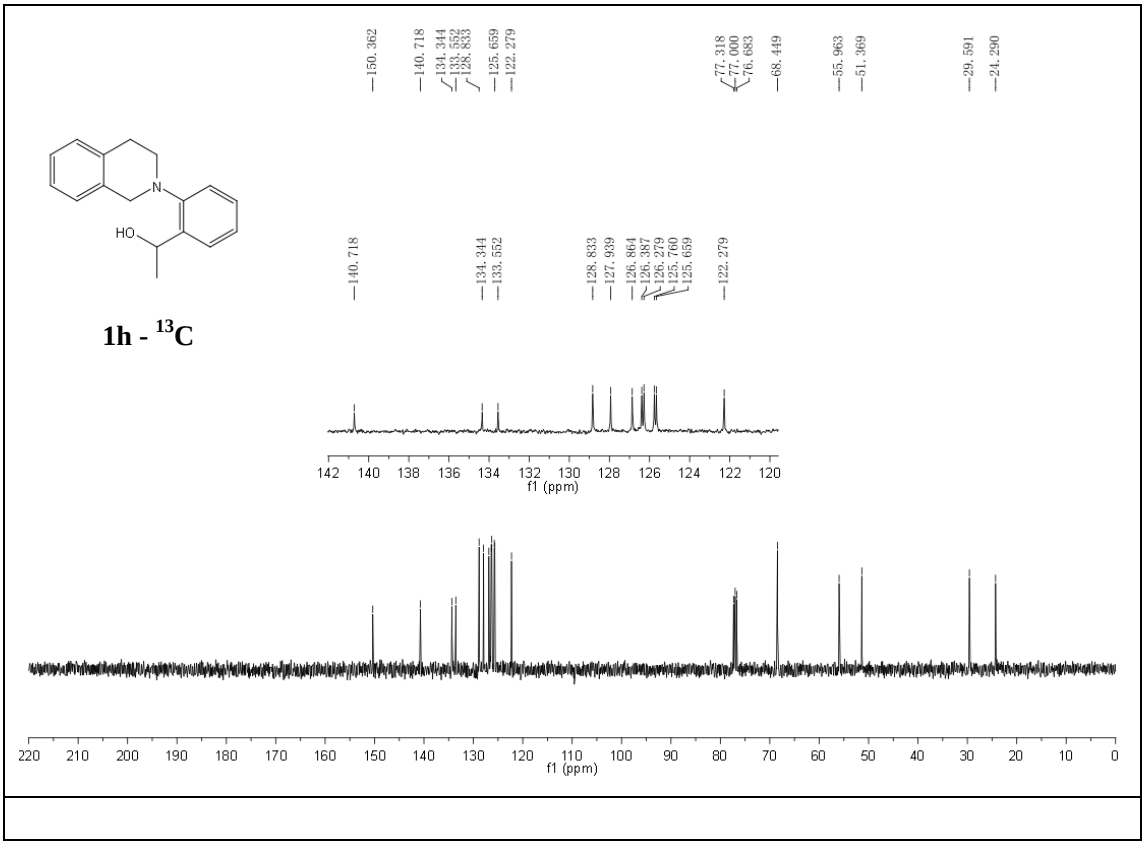
7.282
7.264
7.253
7.216
7.212
7.194
7.178
7.162
7.053
7.036
6.105
5.149
5.132
5.116
5.100
4.207
4.169
4.130
4.093
3.283
3.273
3.260
3.060
1.664
1.545
1.528
1.517
0.000
HO
N
3.38
4.22
1.08
0.77
0.92
0.92
0.98
2.00
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

1h - ^{1}H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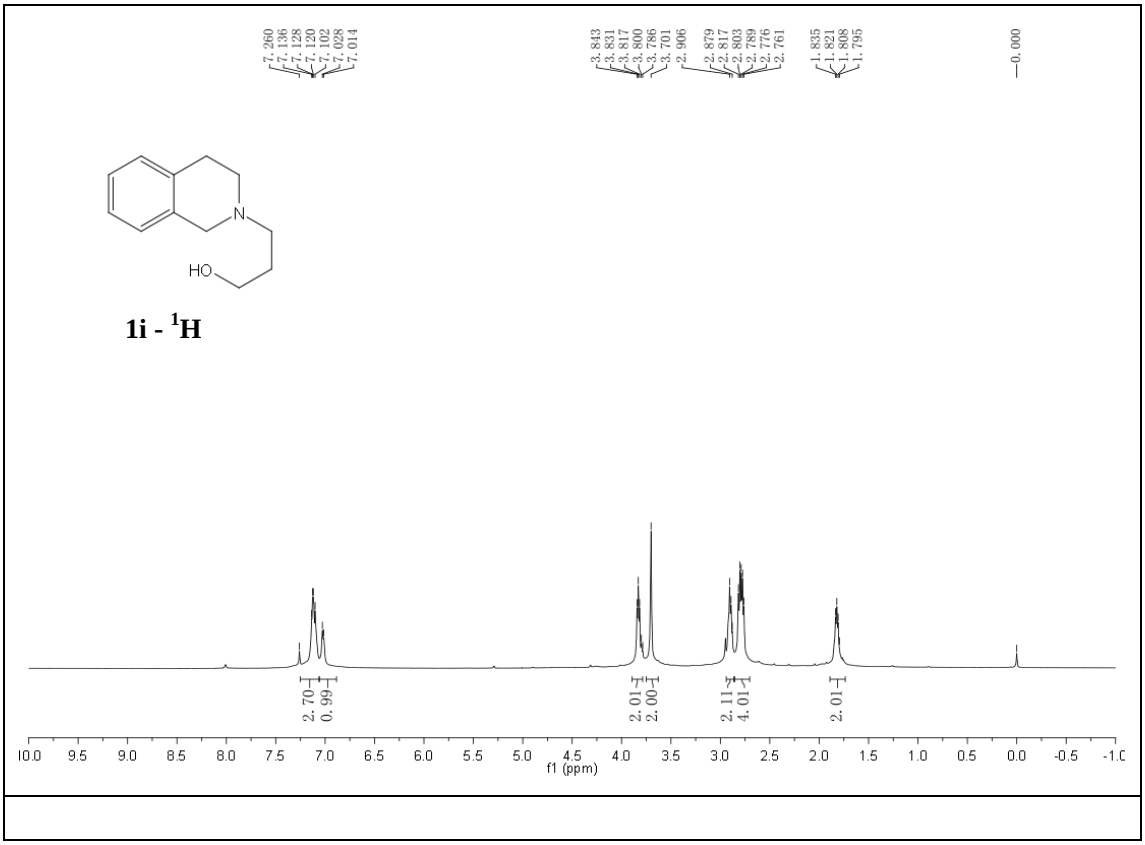
134.344
133.552
128.833
125.659
122.279
77.318
77.000
76.683
68.449
55.963
51.369
29.591
24.290
HO
N
140.718
134.344
133.552
128.833
127.939
126.864
126.387
126.279
125.760
125.659
122.279
142 140 138 136 134 132 130 128 126 124 122 120
f1 (ppm)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

1h - ^{13}C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7.136
7.128
7.120
7.102
7.028
7.014
3.843
3.831
3.817
3.800
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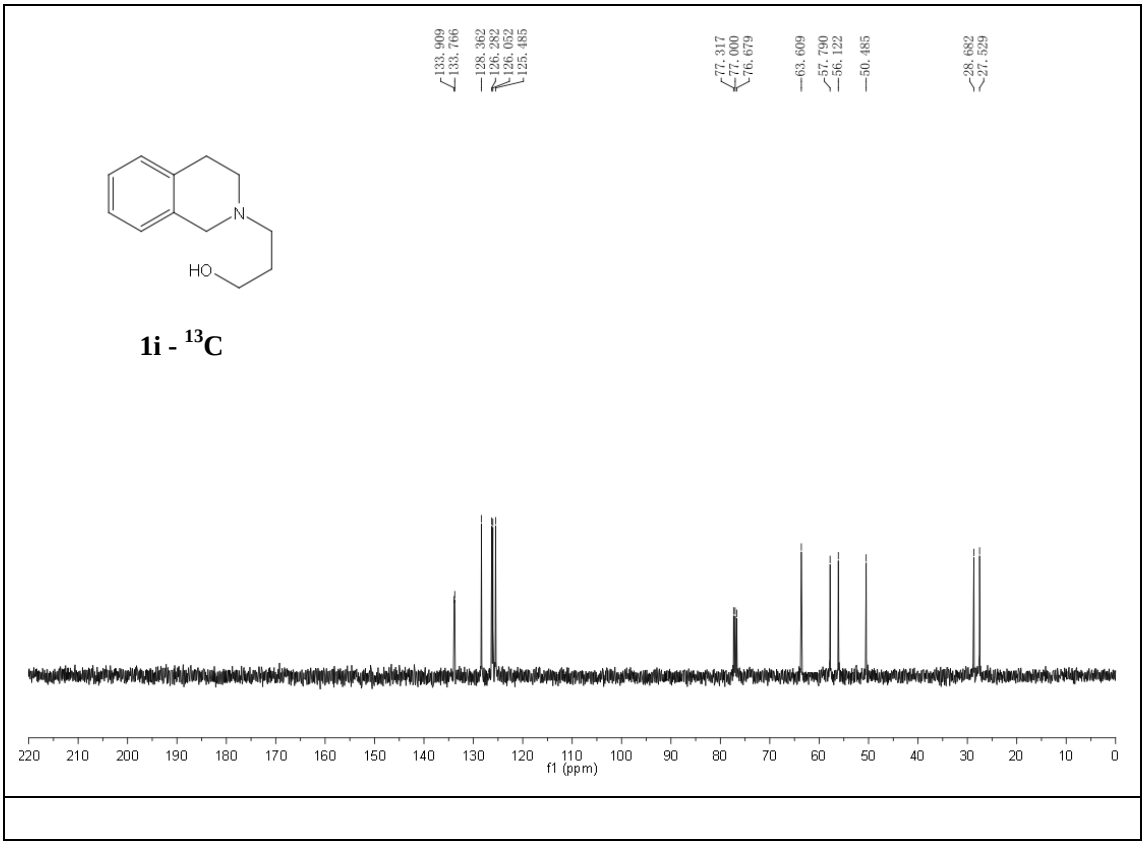
3.701
2.906
2.879
2.817
2.803
2.789
2.776
2.761
1.835
1.821
1.808
1.795
0.000
HO
N
2.70
0.99
2.01
2.00
2.11
4.01
2.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

1i - ^{1}H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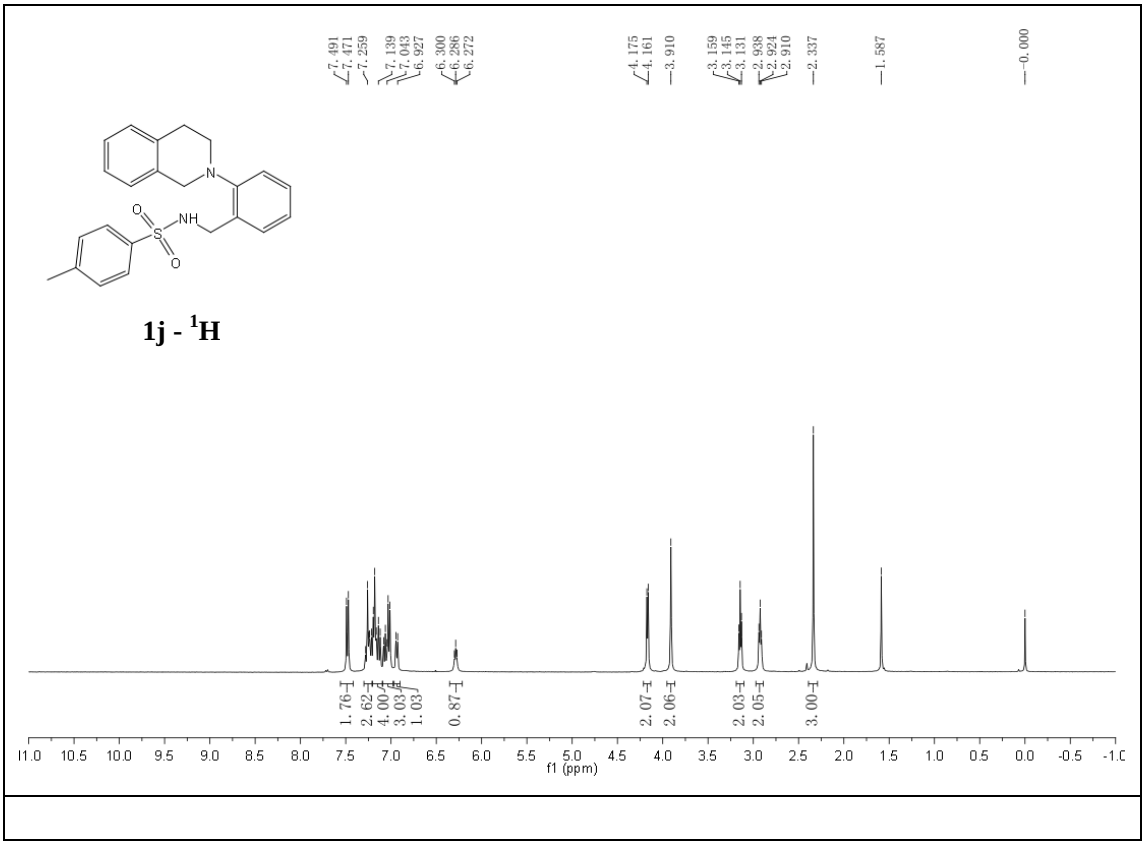
133.766
128.362
126.282
126.052
125.485
77.317
77.000
76.679
63.609
57.790
56.122
50.485
28.682
27.529
N
HO
f1 (ppm)

1i - ^{13}C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6.286
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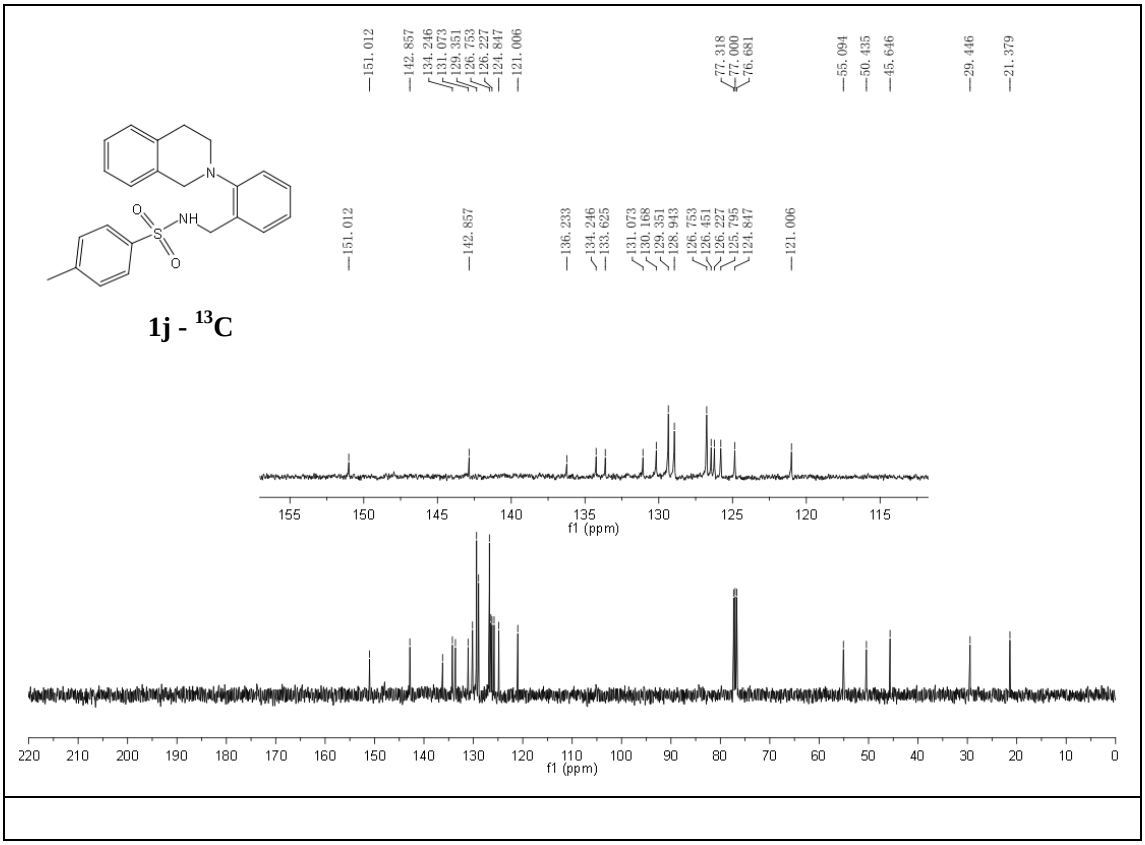
3.910
3.159
3.145
3.131
2.938
2.924
2.910
2.337
1.587
0.000
N
NH
O
S
O
1.76
2.62
4.00
3.03
1.03
0.87
2.07
2.06
2.03
2.05
3.00
f1 (ppm)

1j - ^{1}H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126.753
126.227
124.847
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77.318
77.000
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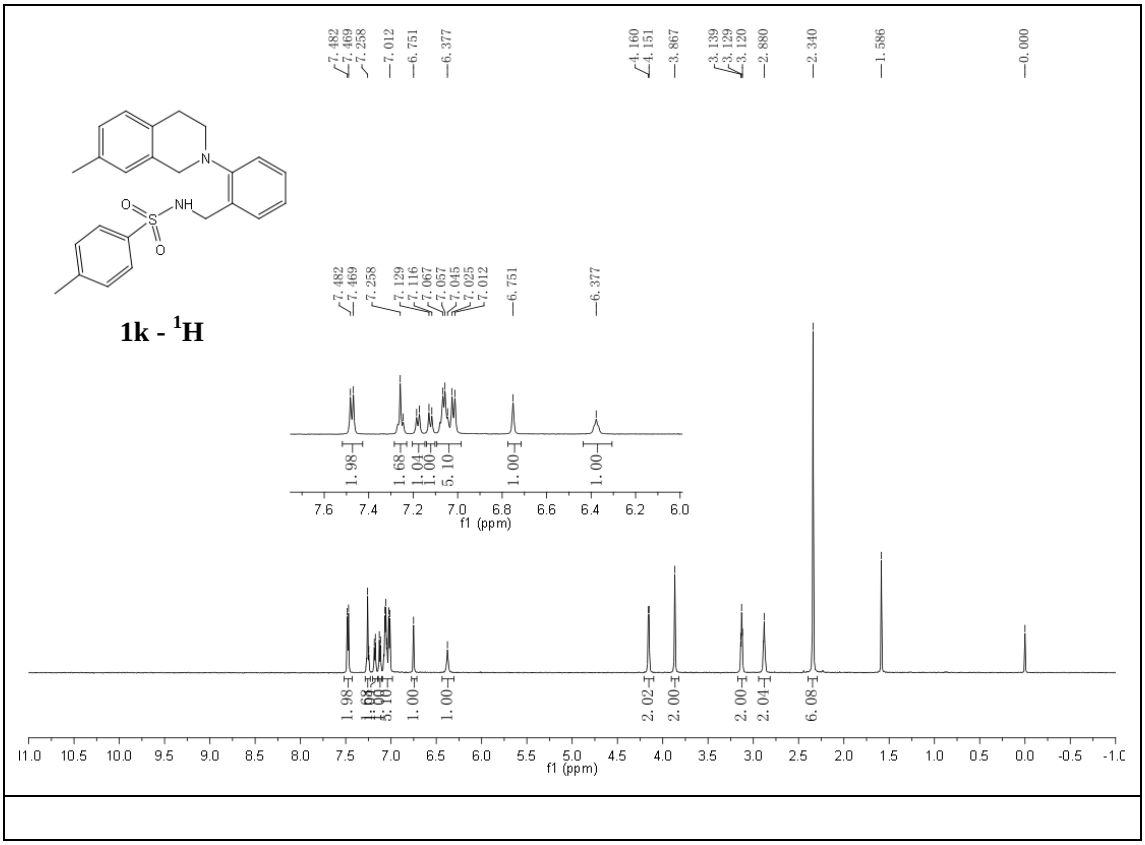
55.094
50.435
45.646
29.446
21.379
136.233
134.246
133.625
131.073
130.168
129.351
128.943
126.753
126.451
126.227
125.795
124.847
121.006
f1 (ppm)

1j - ^{13}C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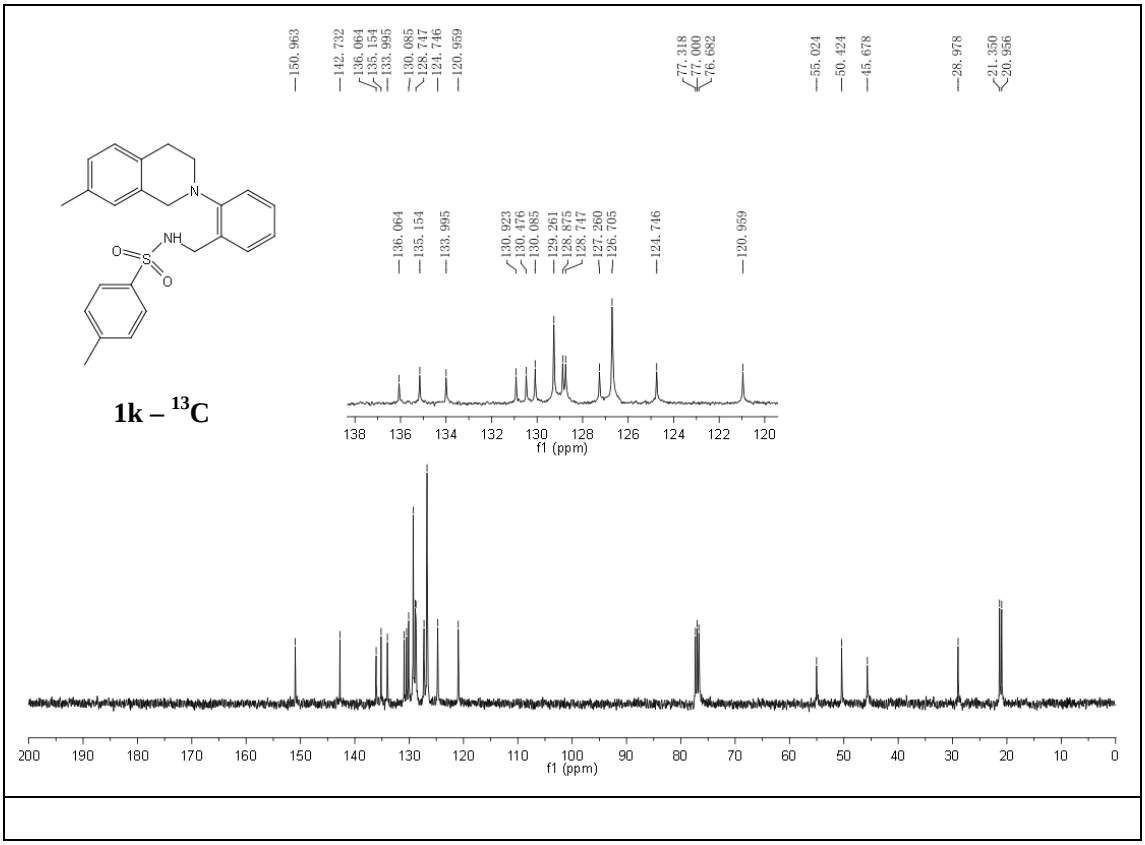
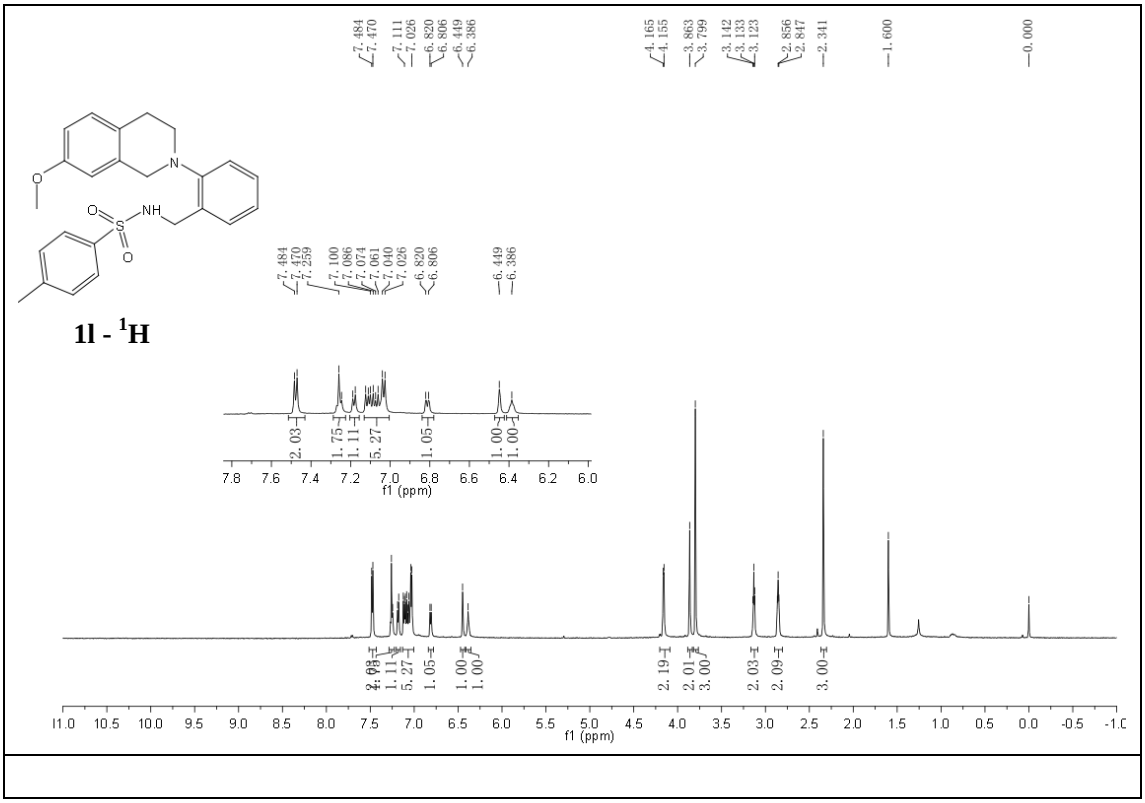
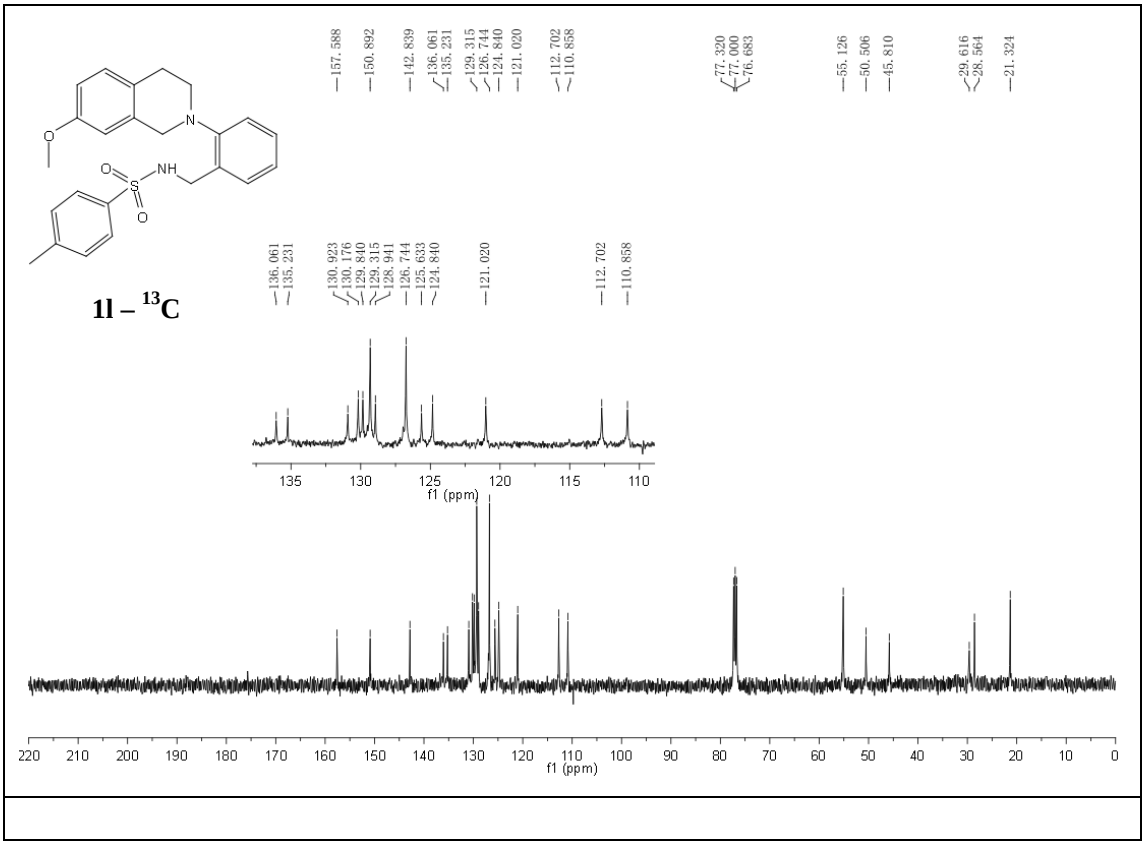
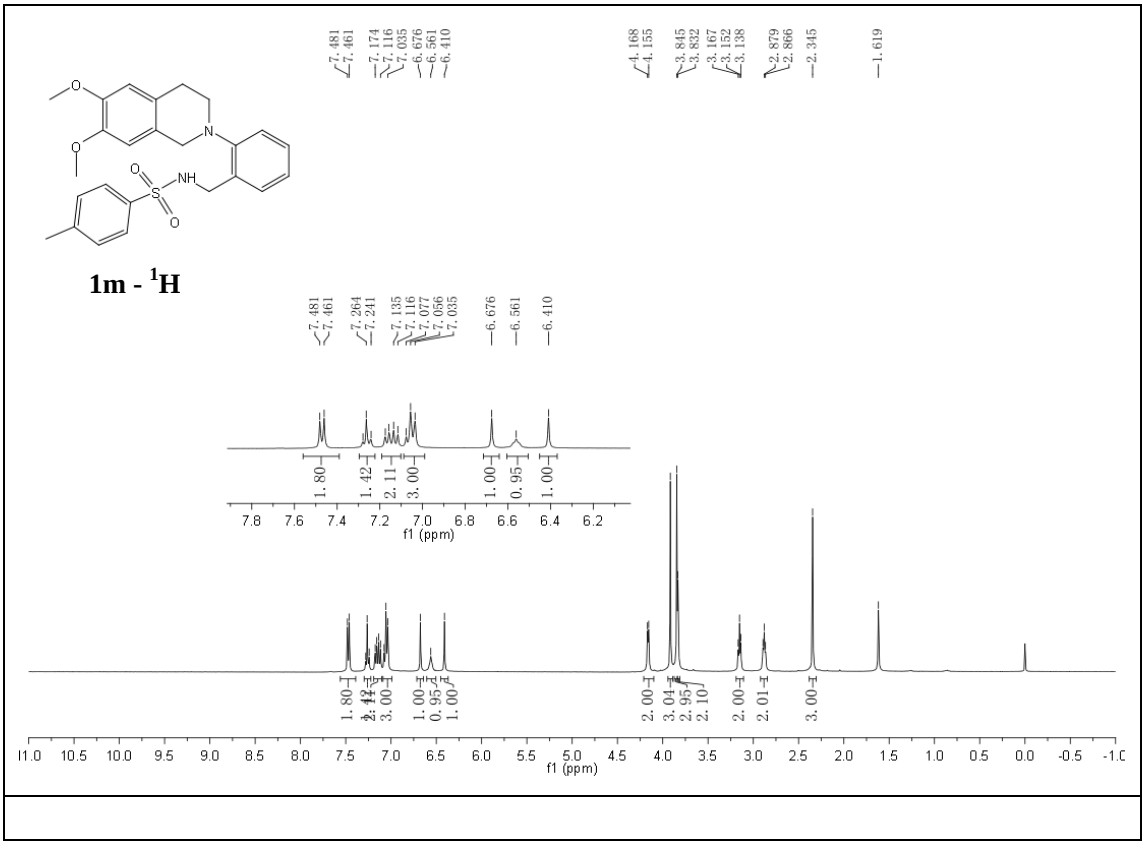
6.751
6.377
4.160
4.151
3.867
3.139
3.129
3.120
2.880
2.340
1.586
0.000
7.129
7.116
7.067
7.057
7.045
7.025
7.012
1.98
1.68
1.04
1.00
5.10
1.00
1.00
2.02
2.00
2.00
2.04
6.08
f1 (ppm)

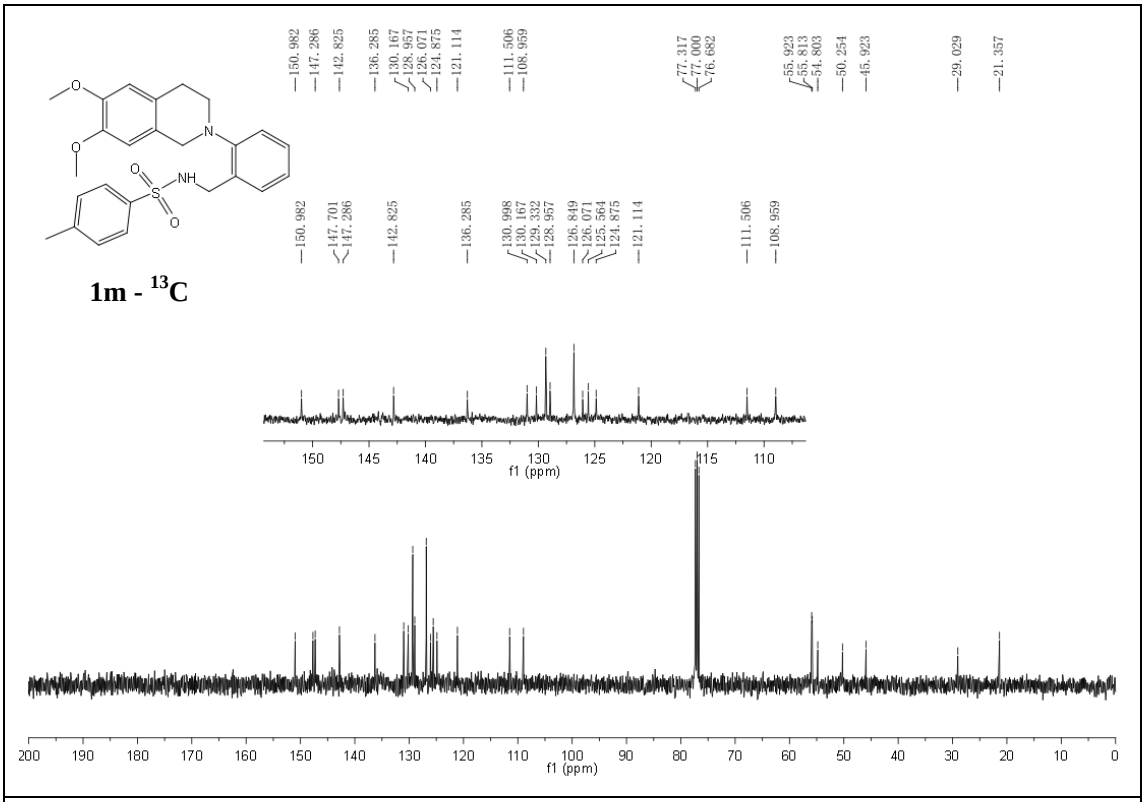
1k - ^{1}H

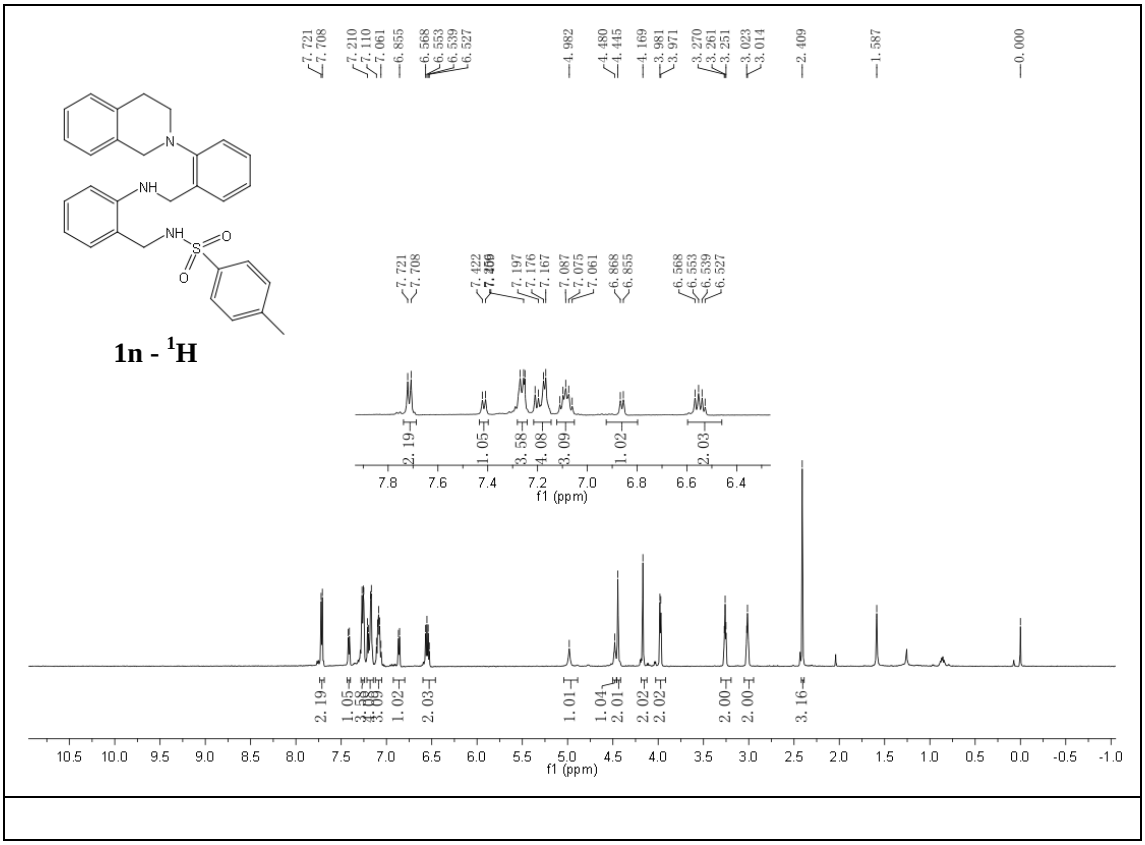

150.963
142.732
136.064
135.154
133.995
130.085
128.747
124.746
120.959
77.318
77.000
76.682
55.024
50.424
45.678
28.978
21.350
20.956
NH
O
S
O
1k – 13C
f1 (ppm)
f1 (ppm)

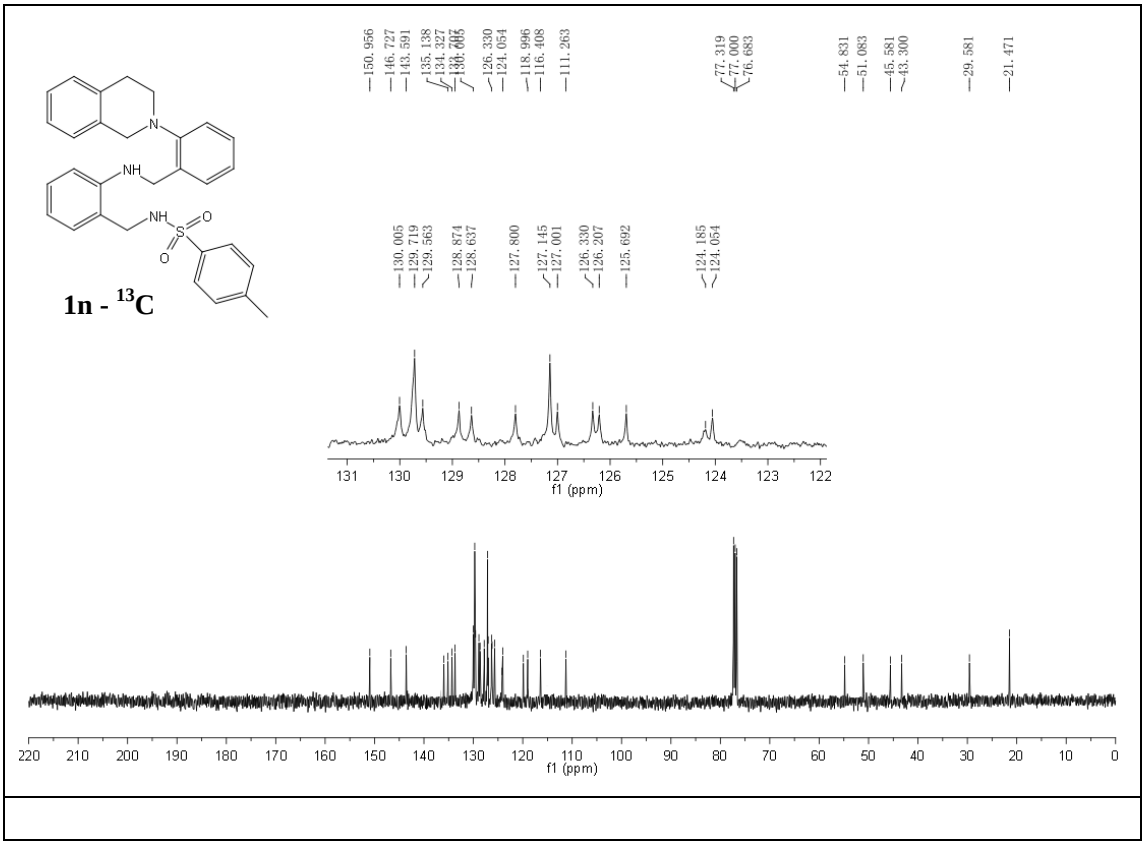

7.484
7.470
7.111
7.026
6.820
6.806
6.449
6.386
4.165
4.155
3.863
3.799
3.142
3.133
3.123
2.856
2.847
2.341
1.600
0.000
O
NH
O
S
O
1l - 1H
f1 (ppm)
f1 (ppm)

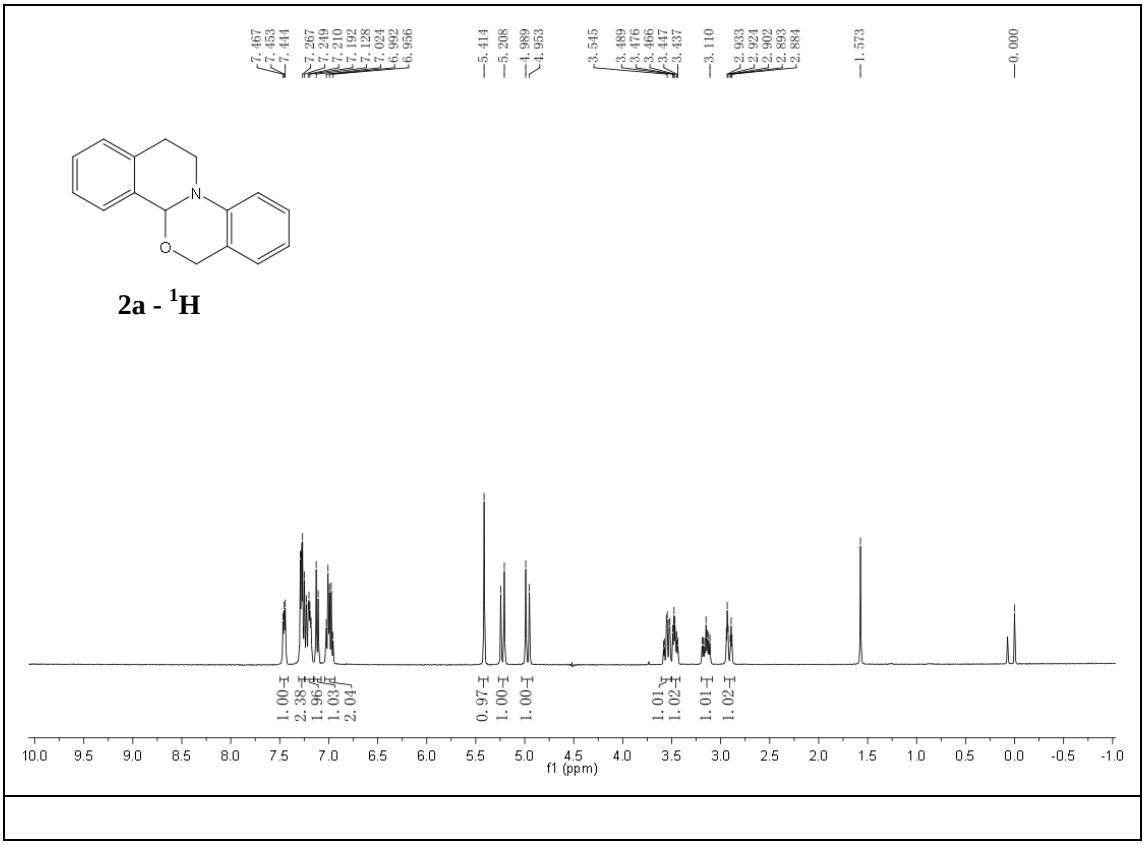

1l – 13C
157.588
150.892
142.839
136.061
135.231
129.315
126.744
124.840
121.020
112.702
110.858
77.320
77.000
76.683
55.126
50.506
45.810
29.616
28.564
21.324
f1 (ppm)


1m - 1H
7.481
7.461
7.264
7.241
7.135
7.116
7.077
7.056
7.035
6.676
6.561
6.410
4.168
4.155
3.845
3.832
3.167
3.152
3.138
2.879
2.866
2.345
1.619
1.80
1.42
2.11
3.00
1.00
0.95
1.00
2.00
3.04
2.95
2.10
2.00
2.01
3.00
f1 (ppm)


1m - 13C
f1 (ppm)


1n - 1H
f1 (ppm)


150.956
146.727
143.591
135.138
134.327
126.330
124.054
118.996
116.408
111.263
77.319
77.000
76.683
54.831
51.083
45.581
43.300
29.581
21.471
130.005
129.719
129.563
128.874
128.637
127.800
127.145
127.001
126.330
126.207
125.692
124.185
124.054
NH
NH
S
O
O
1n - 13C
f1 (ppm)
f1 (ppm)


7.467
7.453
7.444
7.267
7.249
7.210
7.192
7.128
7.024
6.992
6.956
5.414
5.208
4.989
4.953
3.545
3.489
3.476
3.466
3.447
3.437
3.110
2.933
2.924
2.902
2.893
2.884
1.573
0.000
N
O
2a - 1H
1.00
2.38
1.96
1.03
2.04
0.97
1.00
1.00
1.01
1.02
1.01
1.02
f1 (ppm)

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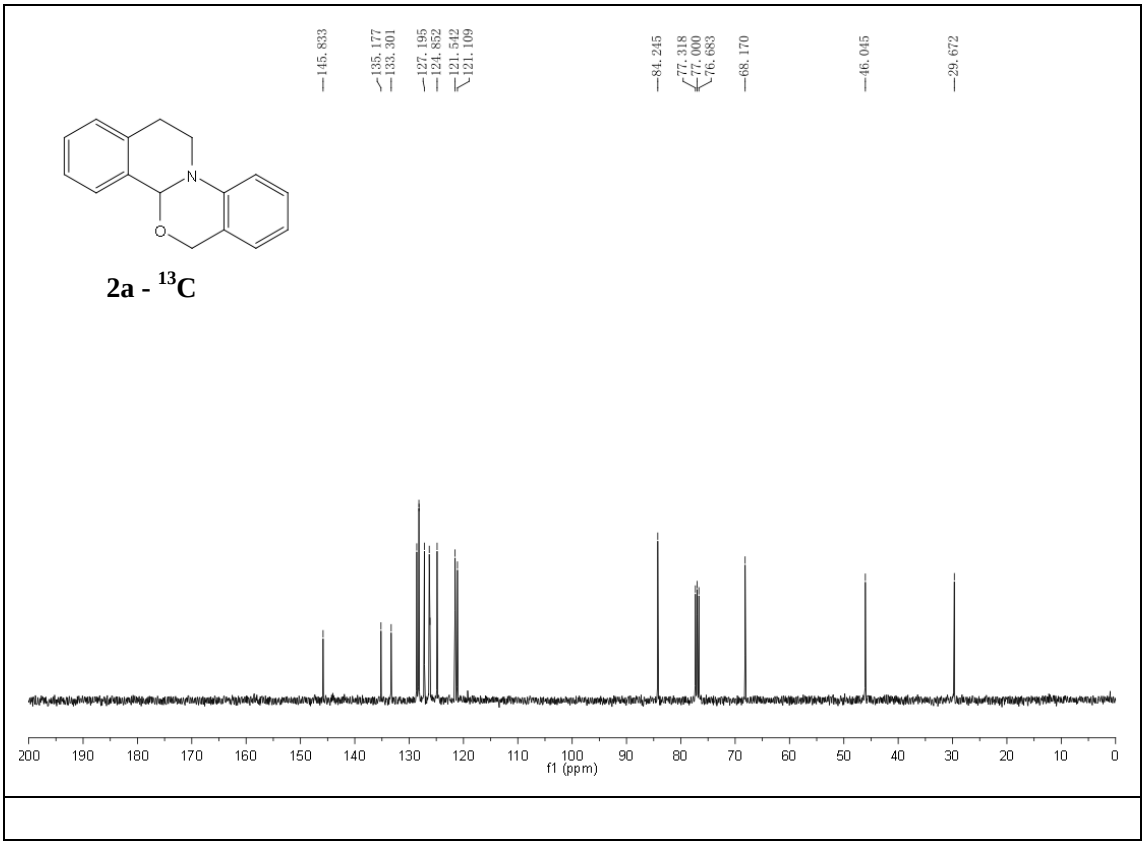
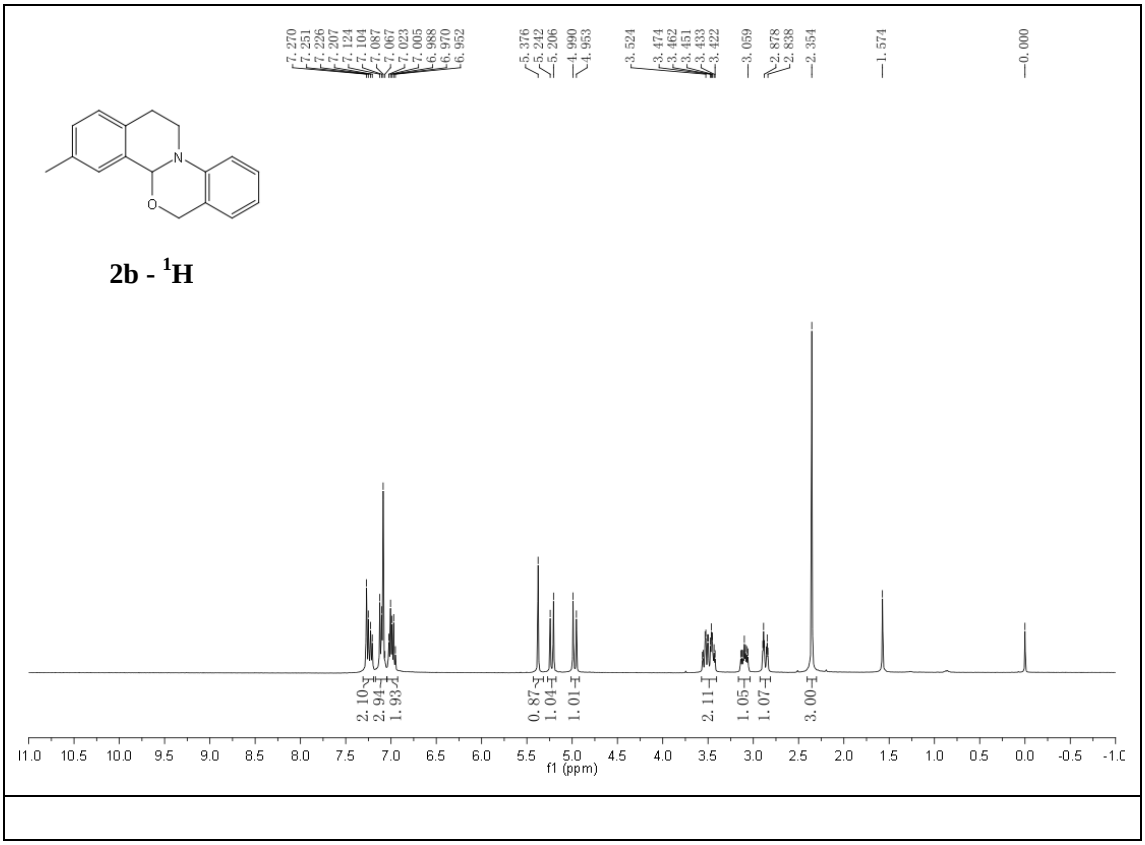
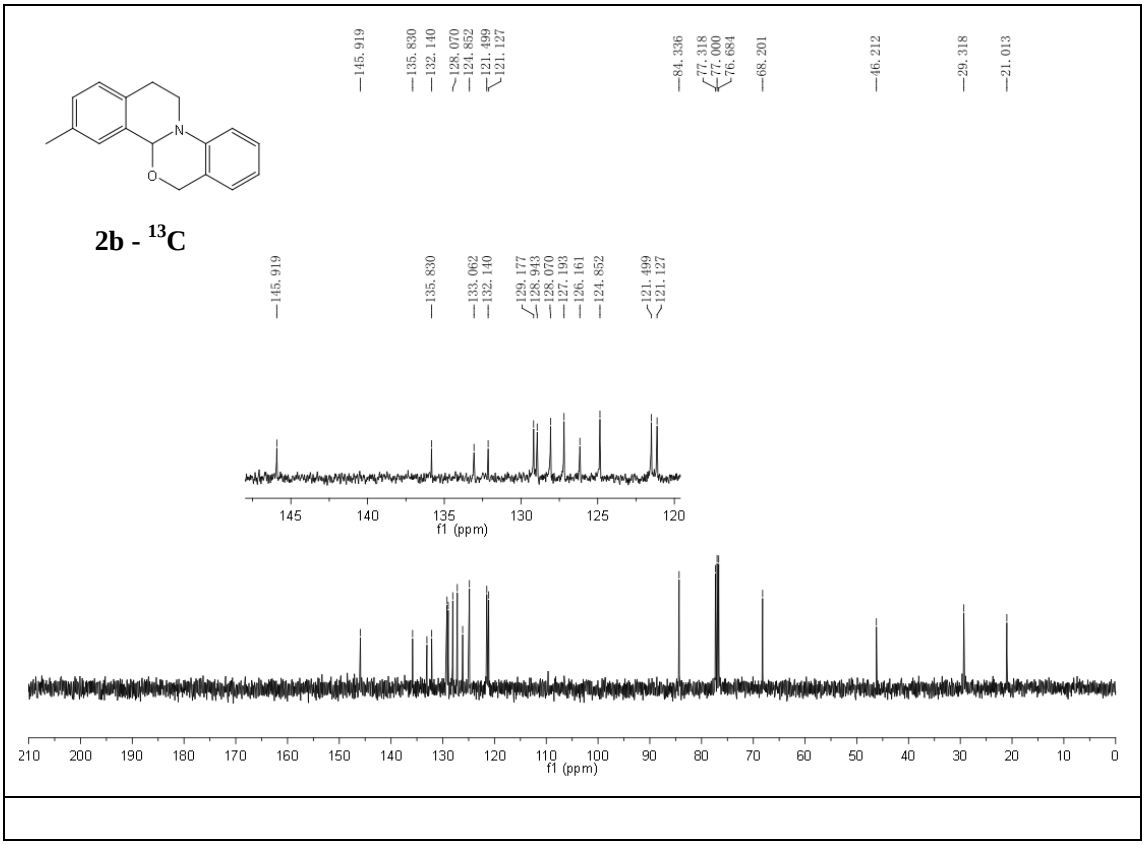
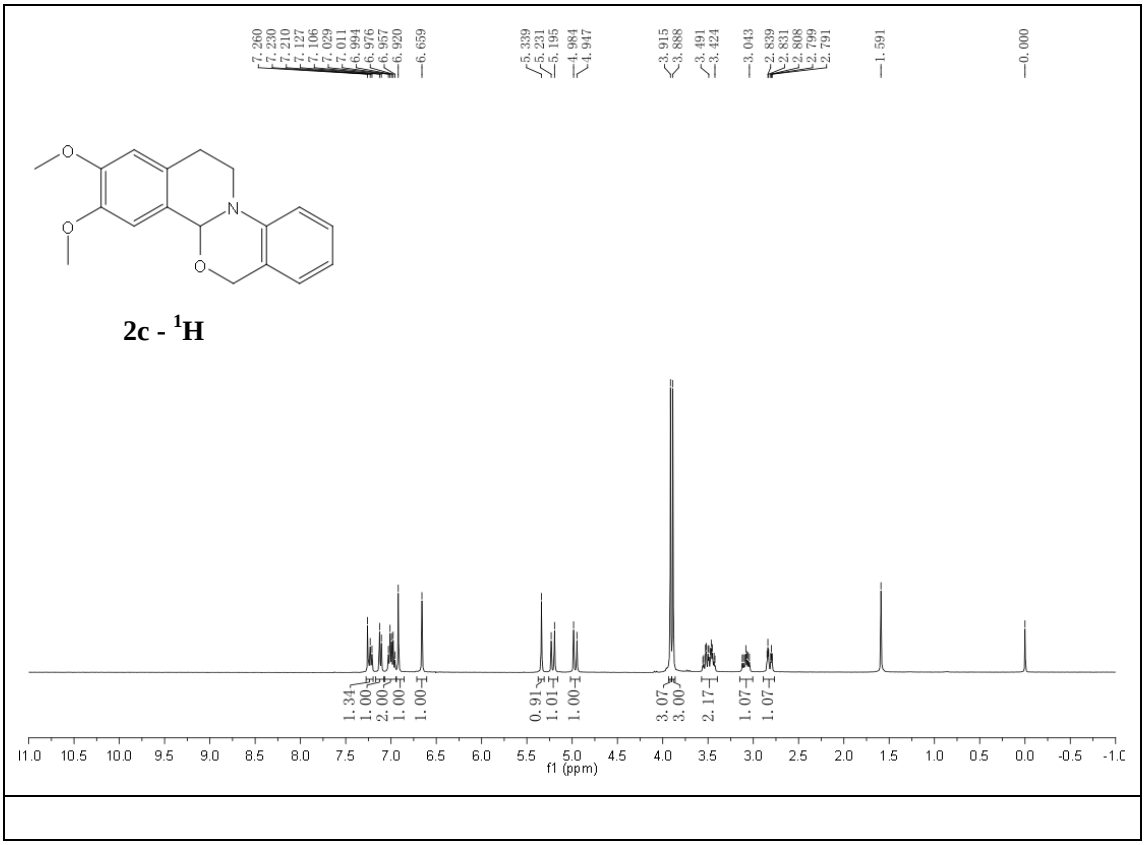


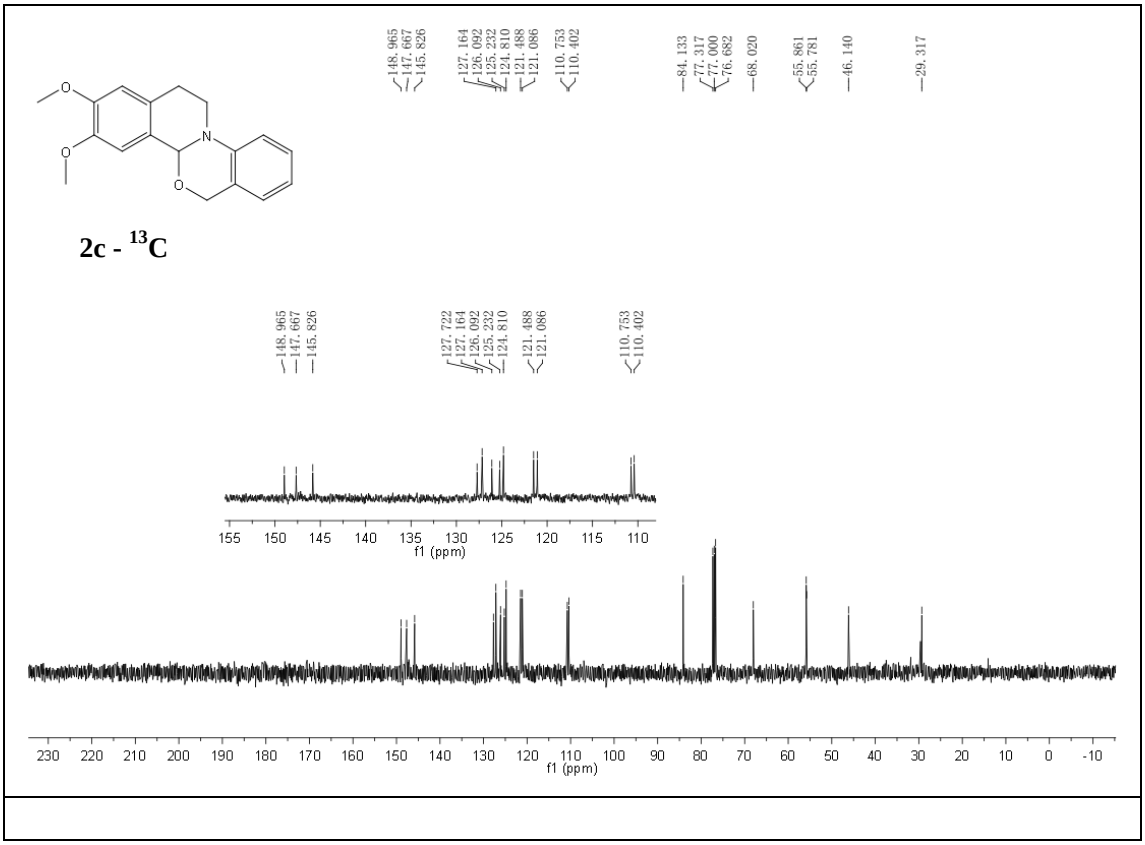
2a - ^{13}C



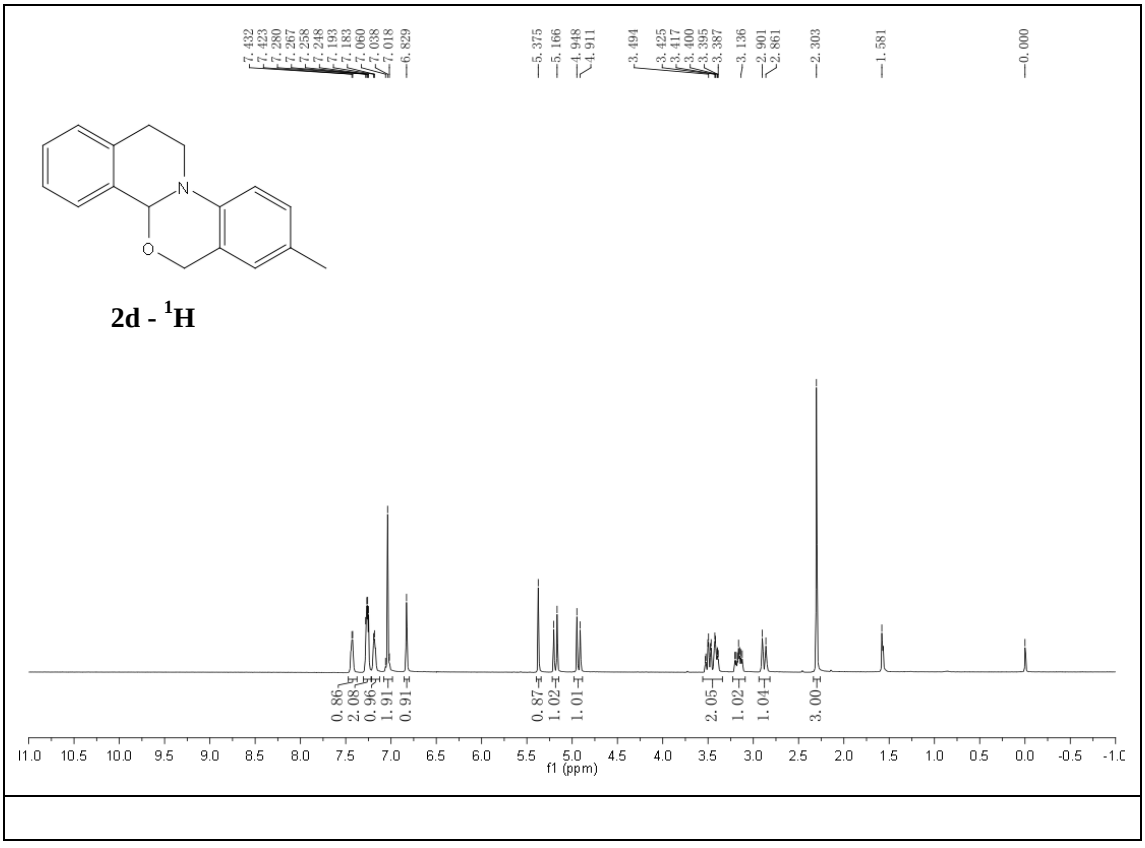
2b - ^{1}H


145.919
135.830
132.140
128.070
124.852
121.499
121.127
84.336
77.318
77.000
76.684
68.201
46.212
29.318
21.013
2b - 13C
145.919
135.830
133.062
132.140
129.177
128.943
128.070
127.193
126.161
124.852
121.499
121.127
f1 (ppm)
f1 (ppm)

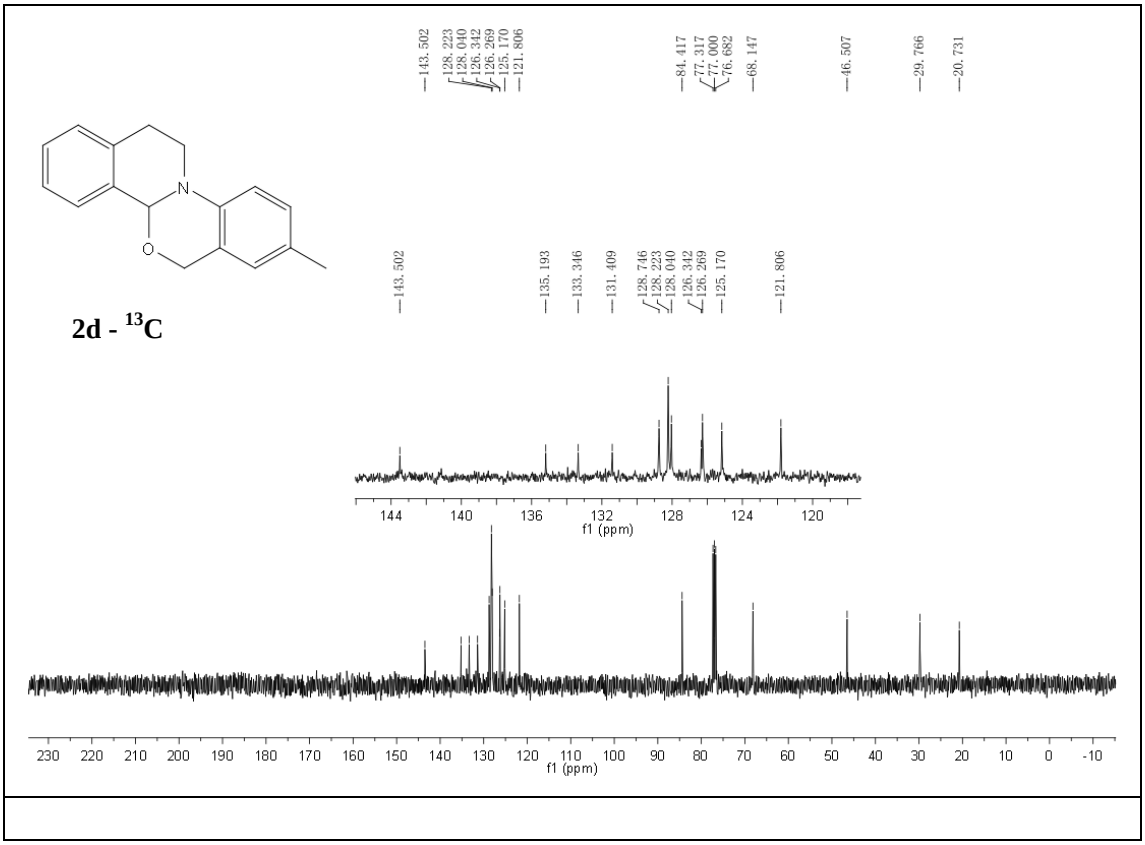

7.260
7.230
7.210
7.127
7.106
7.029
7.011
6.994
6.976
6.957
6.920
6.659
5.339
5.231
5.195
4.984
4.947
3.915
3.888
3.491
3.424
3.043
2.839
2.831
2.808
2.799
2.791
1.591
0.000
2c - 1H
1.34
1.00
2.00
1.00
1.00
0.91
1.01
1.00
3.07
3.00
2.17
1.07
1.07
f1 (ppm)


148.965
147.667
145.826
127.164
126.092
125.232
124.810
121.488
121.086
110.753
110.402
84.133
77.317
77.000
76.682
68.020
55.861
55.781
46.140
29.317
f1 (ppm)

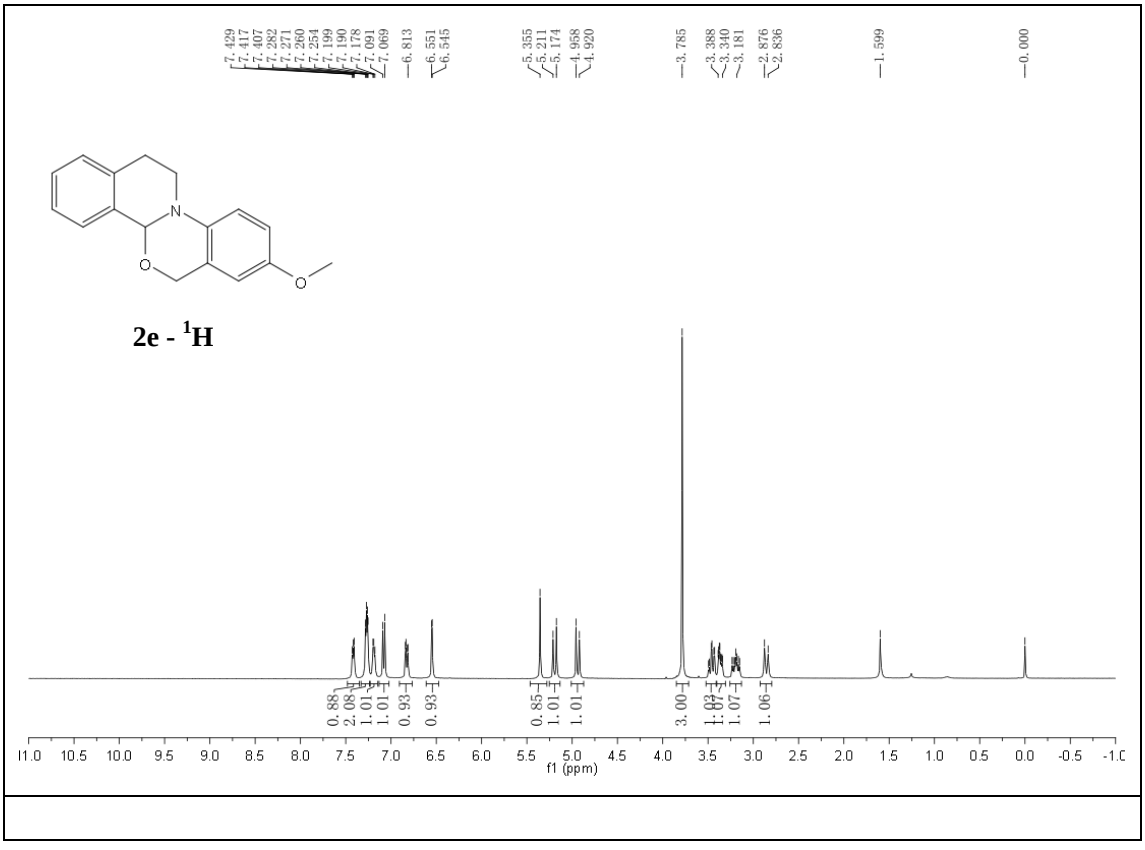
2c - ^{13}C


7.432
7.423
7.280
7.267
7.258
7.248
7.193
7.183
7.060
7.038
7.018
6.829
5.375
5.166
4.948
4.911
3.494
3.425
3.417
3.400
3.395
3.387
3.136
2.901
2.861
2.303
1.581
0.000
0.86
2.08
0.96
1.91
0.91
0.87
1.02
1.01
2.05
1.02
1.04
3.00
f1 (ppm)

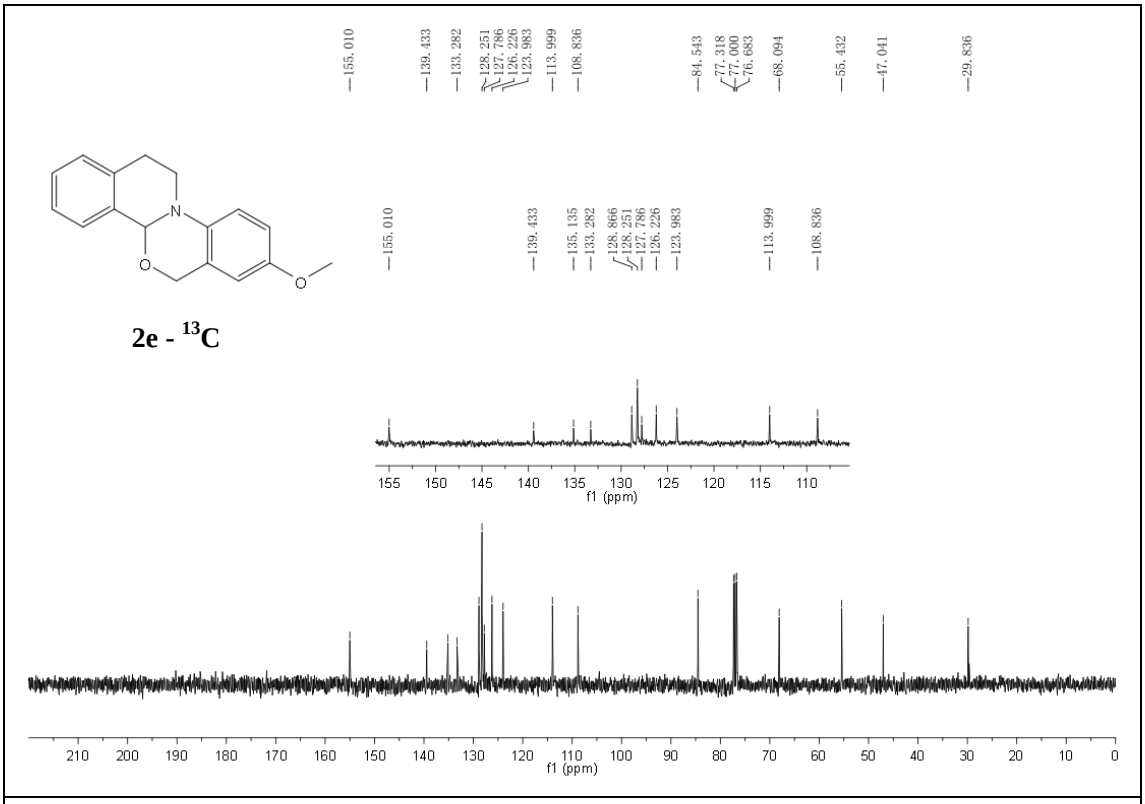
2d - ^{1}H

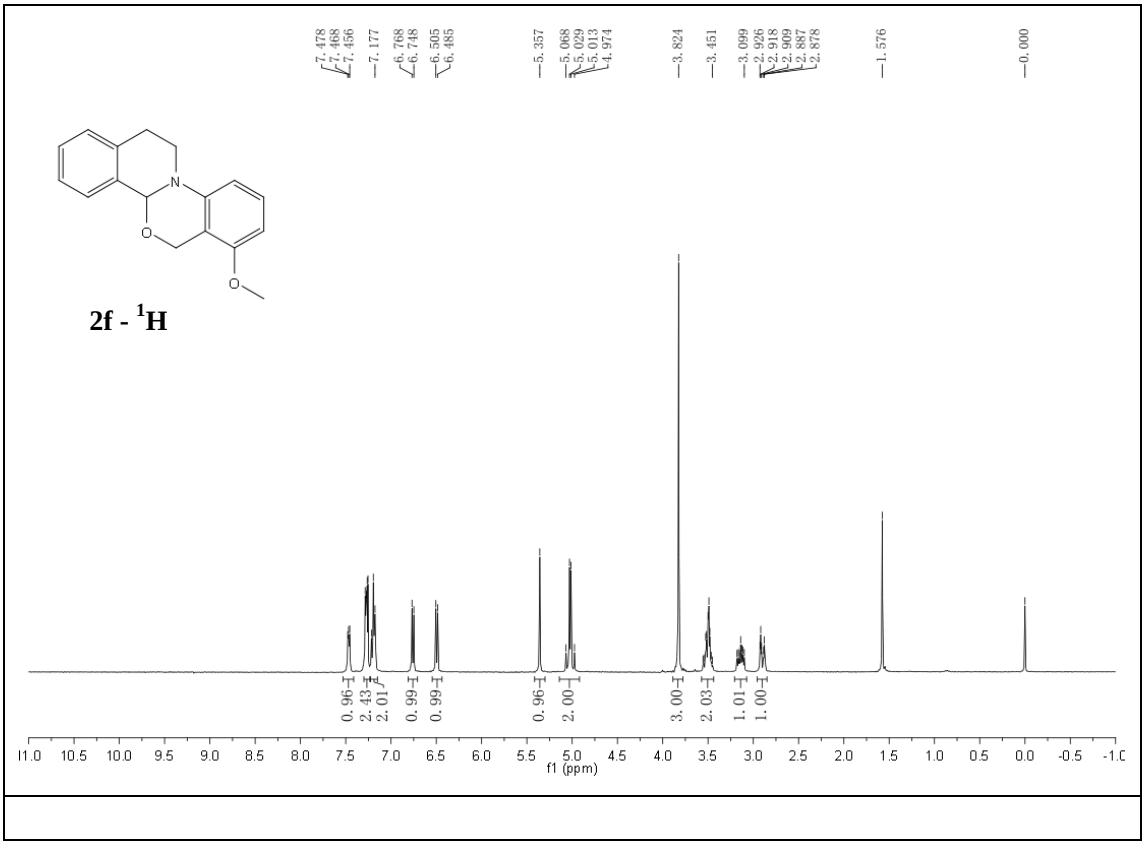

143.502
128.223
128.040
126.342
126.269
125.170
121.806
84.417
77.317
77.000
76.682
68.147
46.507
29.766
20.731
N
O
143.502
135.193
133.346
131.409
128.746
128.223
128.040
126.342
126.269
125.170
121.806
f1 (ppm)

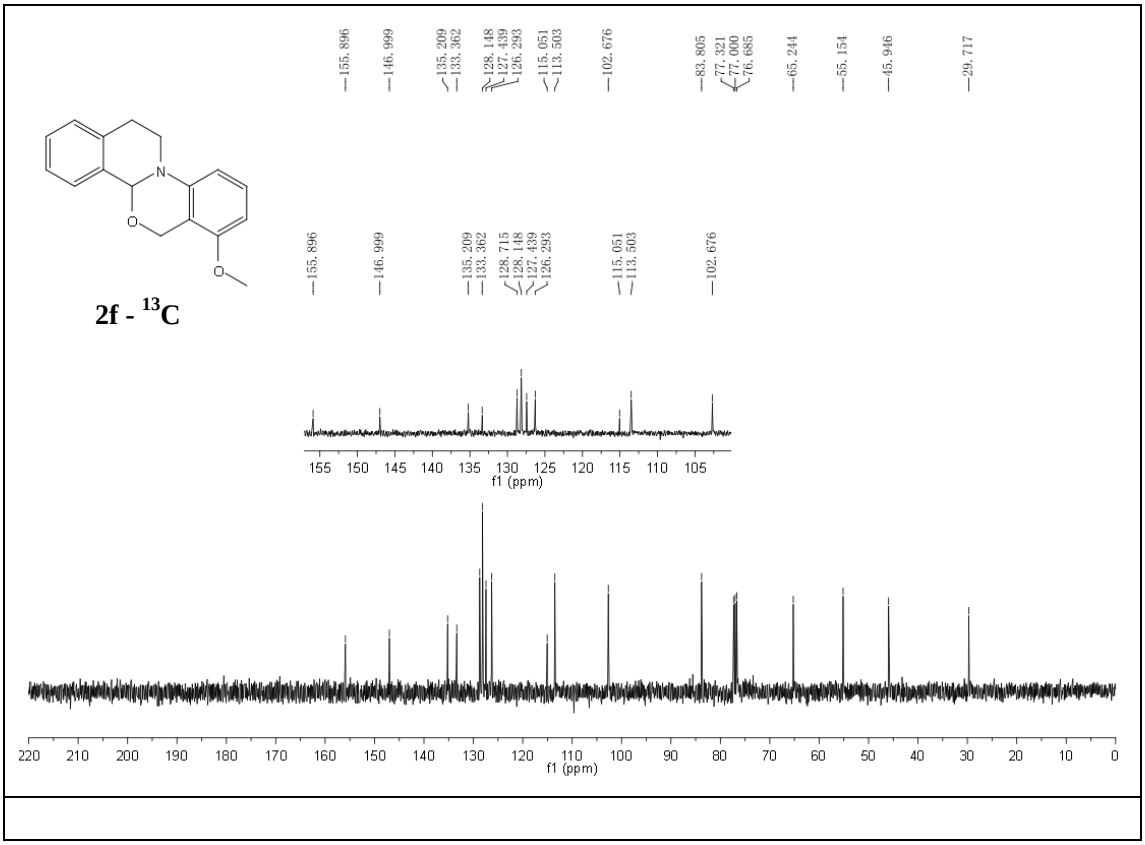
2d - ^{13}C


7.429
7.417
7.407
7.282
7.271
7.260
7.254
7.199
7.190
7.178
7.091
7.069
6.813
6.551
6.545
5.355
5.211
5.174
4.958
4.920
3.785
3.388
3.340
3.181
2.876
2.836
1.599
0.000
N
O
O
0.88
2.08
1.01
1.01
0.93
0.93
0.85
1.01
1.01
3.00
1.03
1.07
1.06
f1 (ppm)

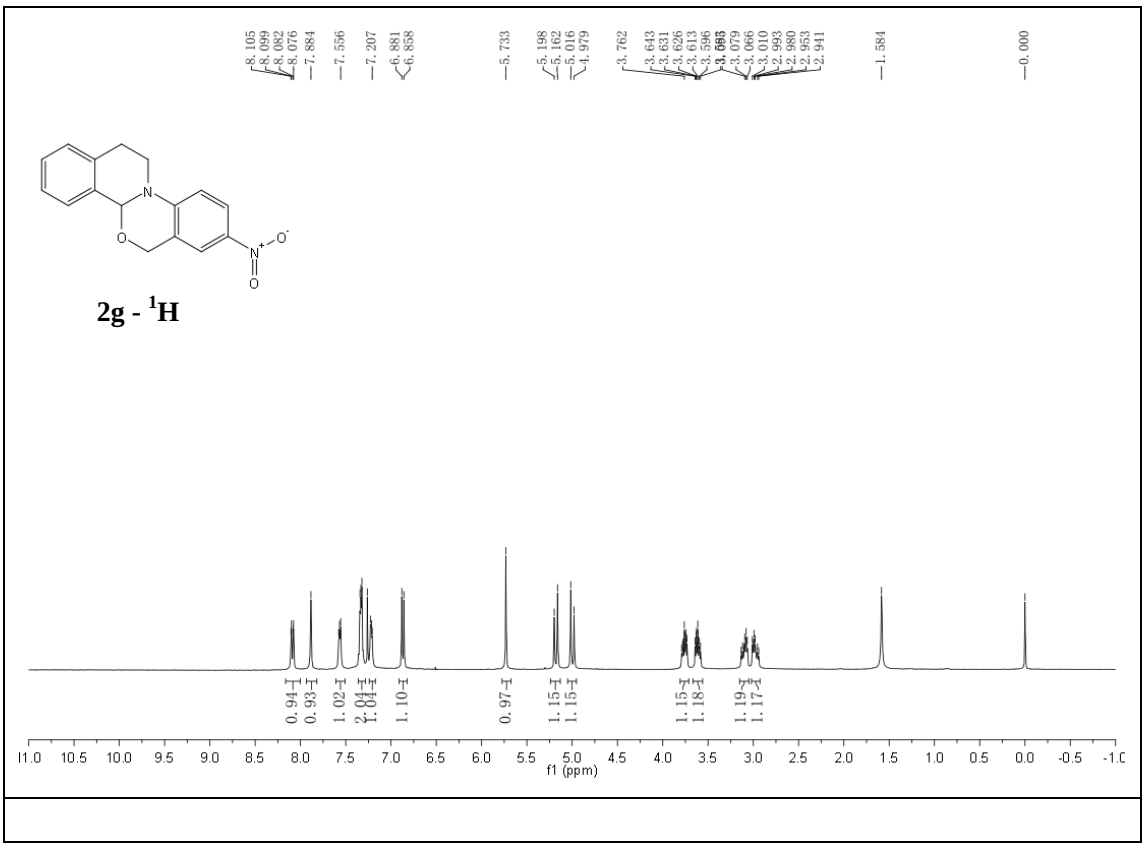
2e - ^{1}H


155.010
139.433
133.282
128.251
127.786
126.226
123.983
113.999
108.836
84.543
77.318
77.000
76.683
68.094
55.432
47.041
29.836
O
N
O
2e - ^{13}C
f1 (ppm)

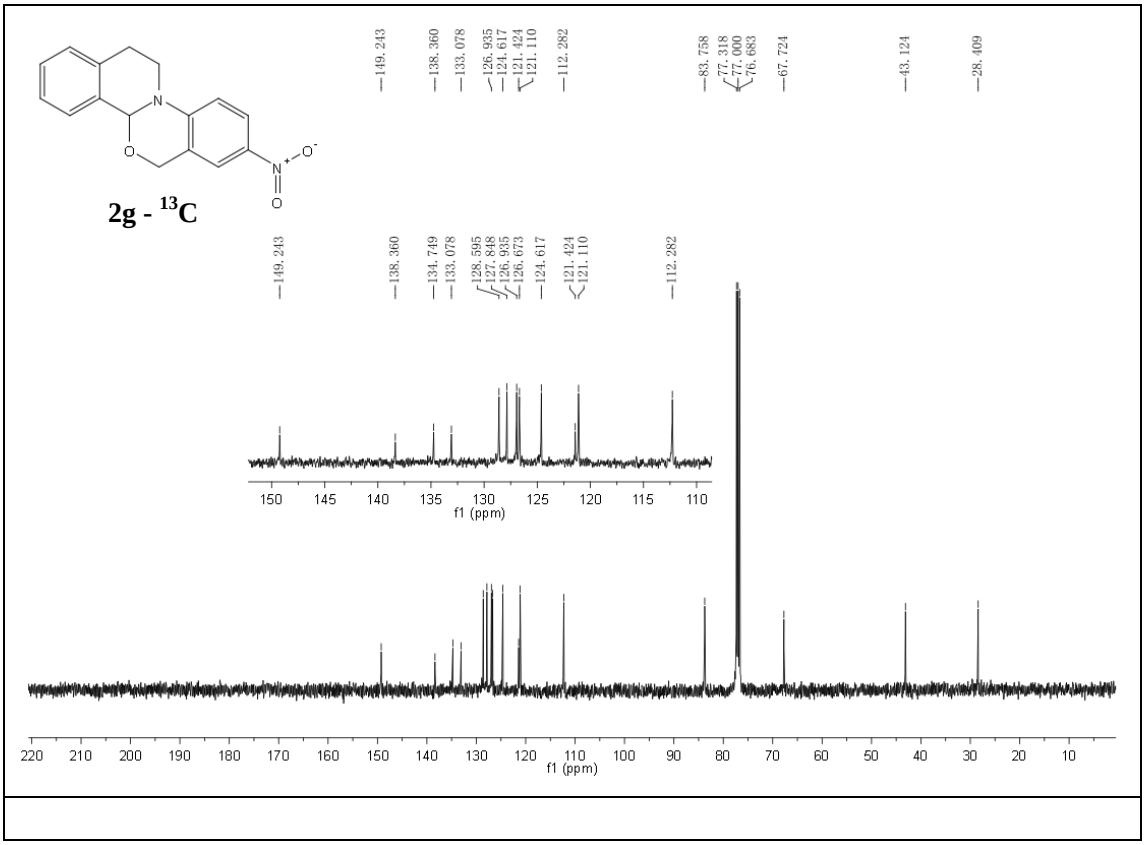

7.478
7.468
7.456
7.177
6.768
6.748
6.505
6.485
5.357
5.068
5.029
5.013
4.974
3.824
3.451
3.099
2.926
2.918
2.909
2.887
2.878
1.576
0.000
O
N
O
2f - ^{1}H
0.96
2.43
2.01
0.99
0.99
0.96
2.00
3.00
2.03
1.01
1.00
f1 (ppm)


155.896
146.999
135.209
133.362
128.148
127.439
126.293
115.051
113.503
102.676
83.805
77.321
77.000
76.685
65.244
55.154
45.946
29.717
f1 (ppm)

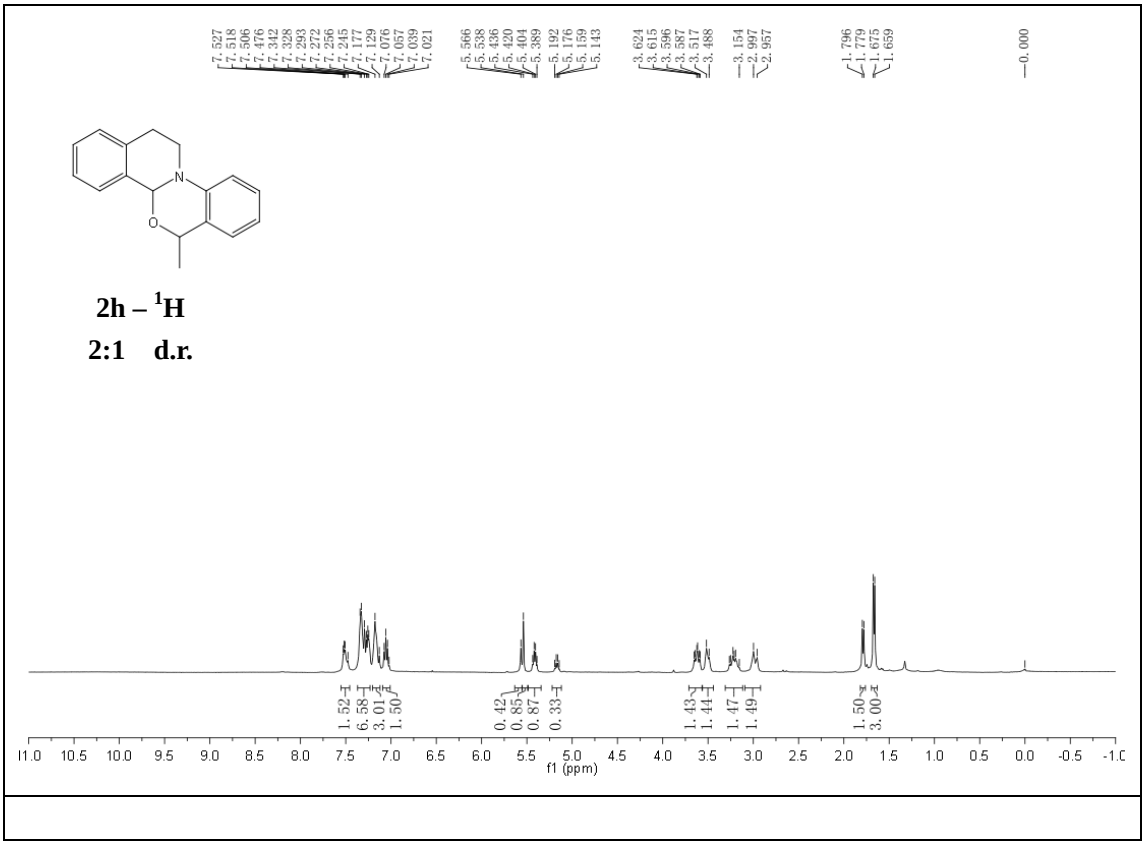
2f - ^{13}C


8.105
8.099
8.082
8.076
7.884
7.556
7.207
6.881
6.858
5.733
5.198
5.162
5.016
4.979
3.762
3.643
3.631
3.626
3.613
3.596
3.079
3.066
3.010
2.993
2.980
2.953
2.941
1.584
0.000
0.94
0.93
1.02
2.04
1.04
1.10
0.97
1.15
1.15
1.15
1.18
1.19
1.17
f1 (ppm)

2g - ^{1}H

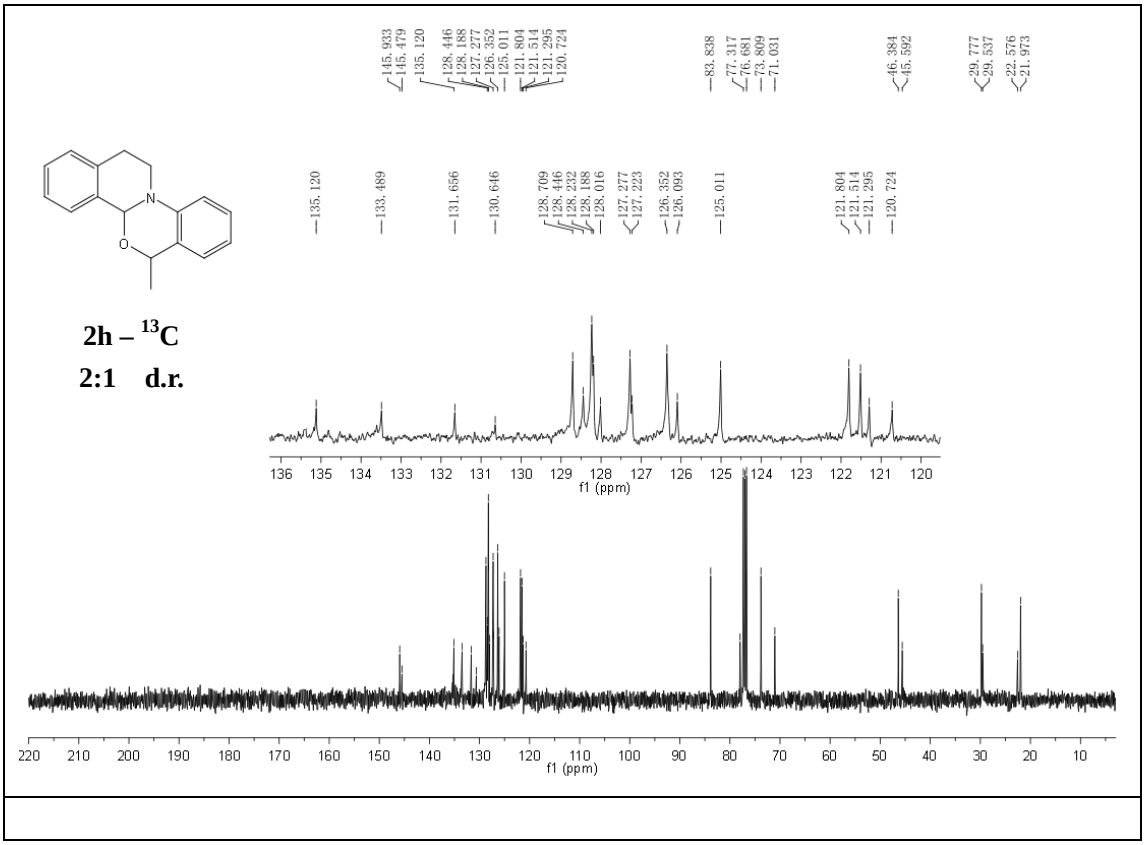


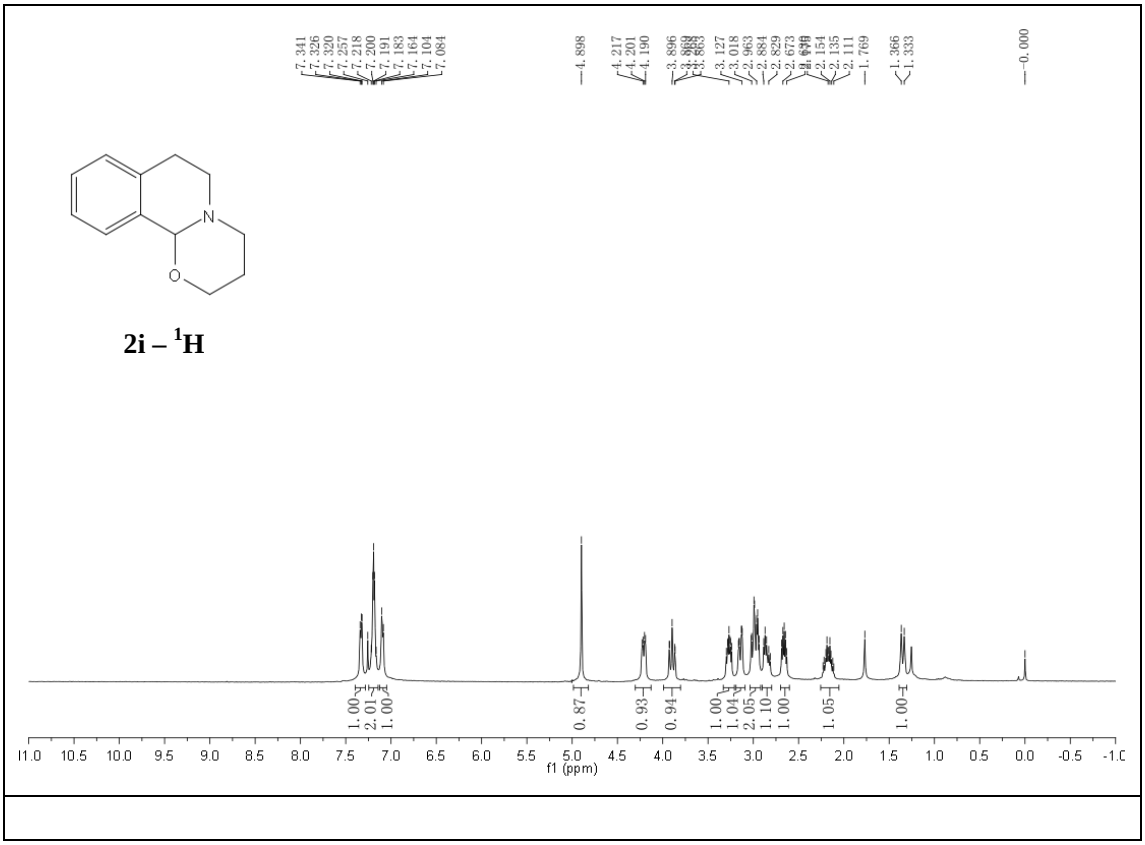
2g - ^{13}C

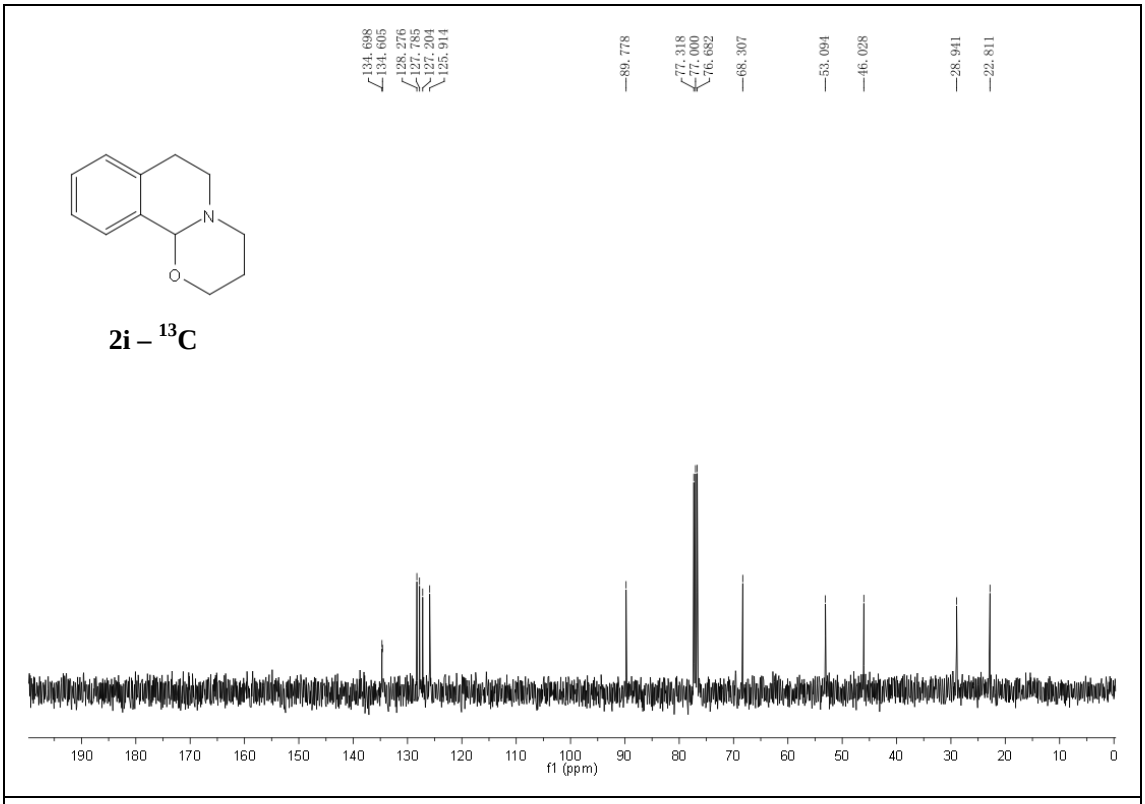


2h – ^{1}H

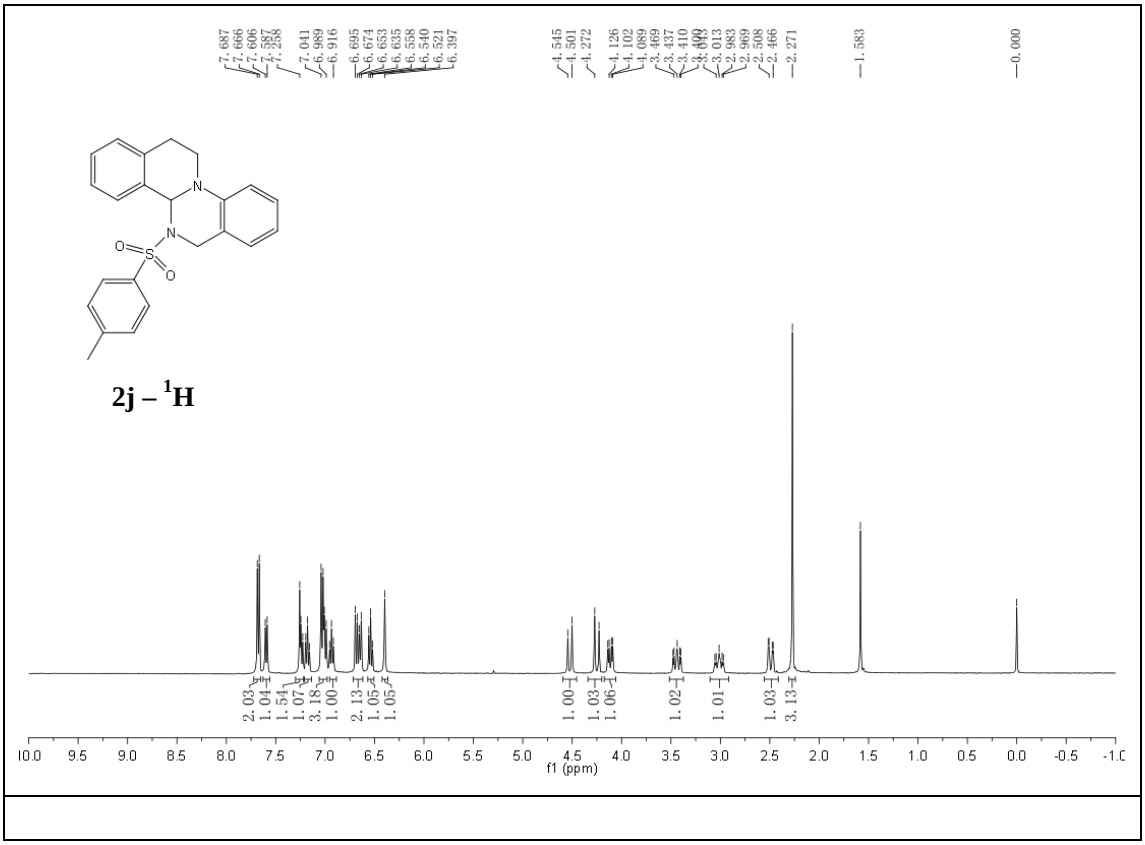
2:1 d.r.


2h – 13C
2:1 d.r.
f1 (ppm)

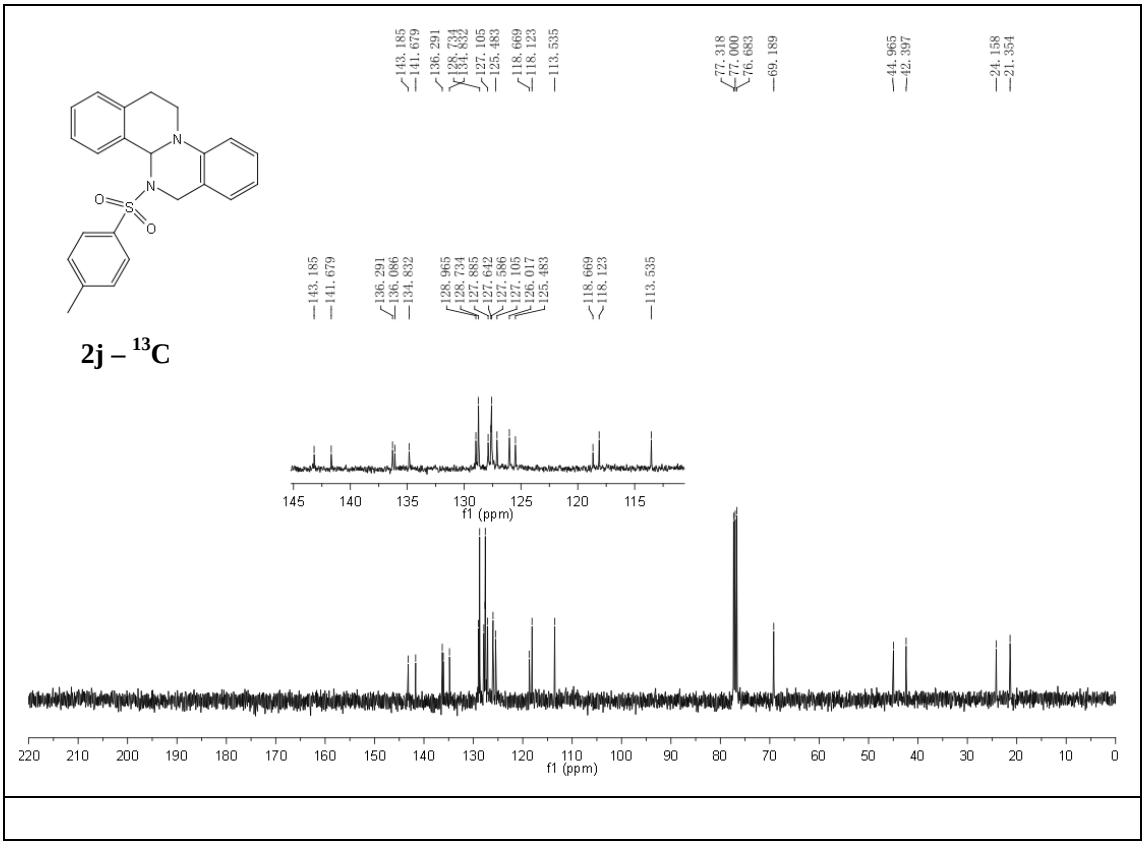

2i – 1H
f1 (ppm)

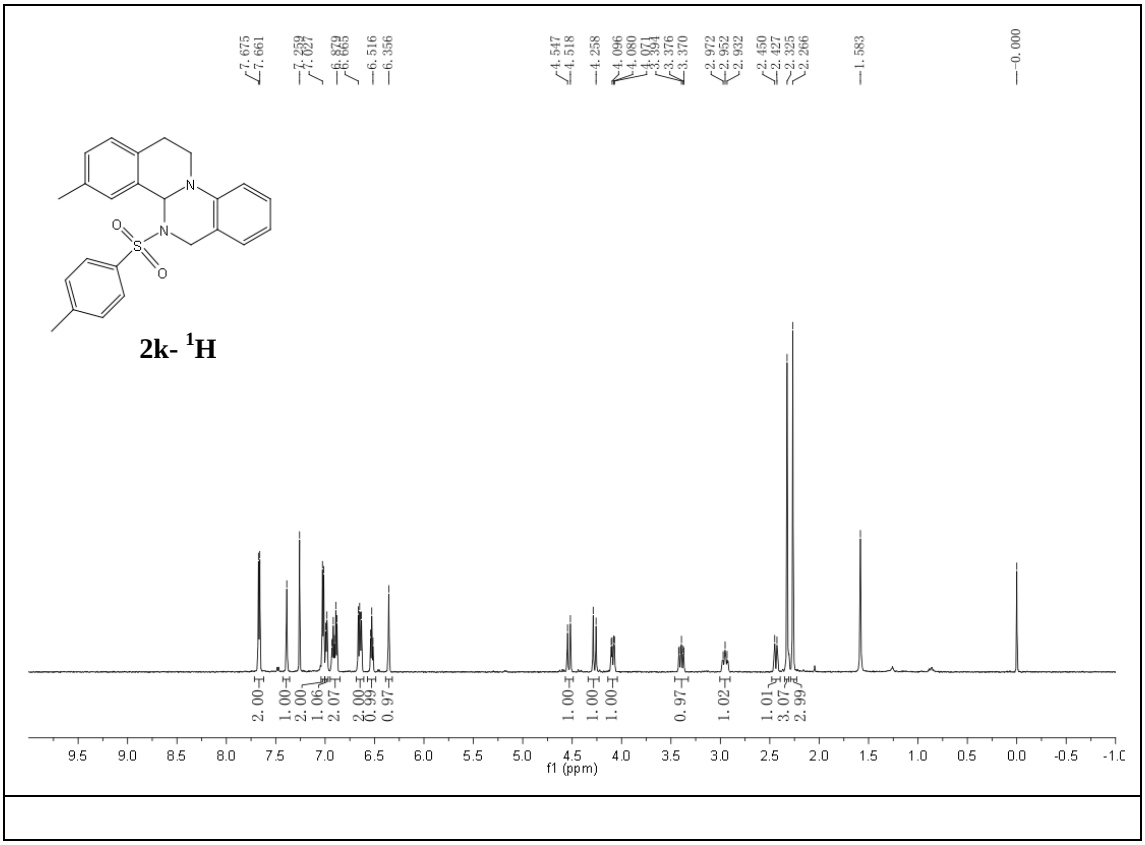

134.698
134.605
128.276
127.785
127.204
125.914
89.778
77.318
77.000
76.682
68.307
53.094
46.028
28.941
22.811
N
O
f1 (ppm)

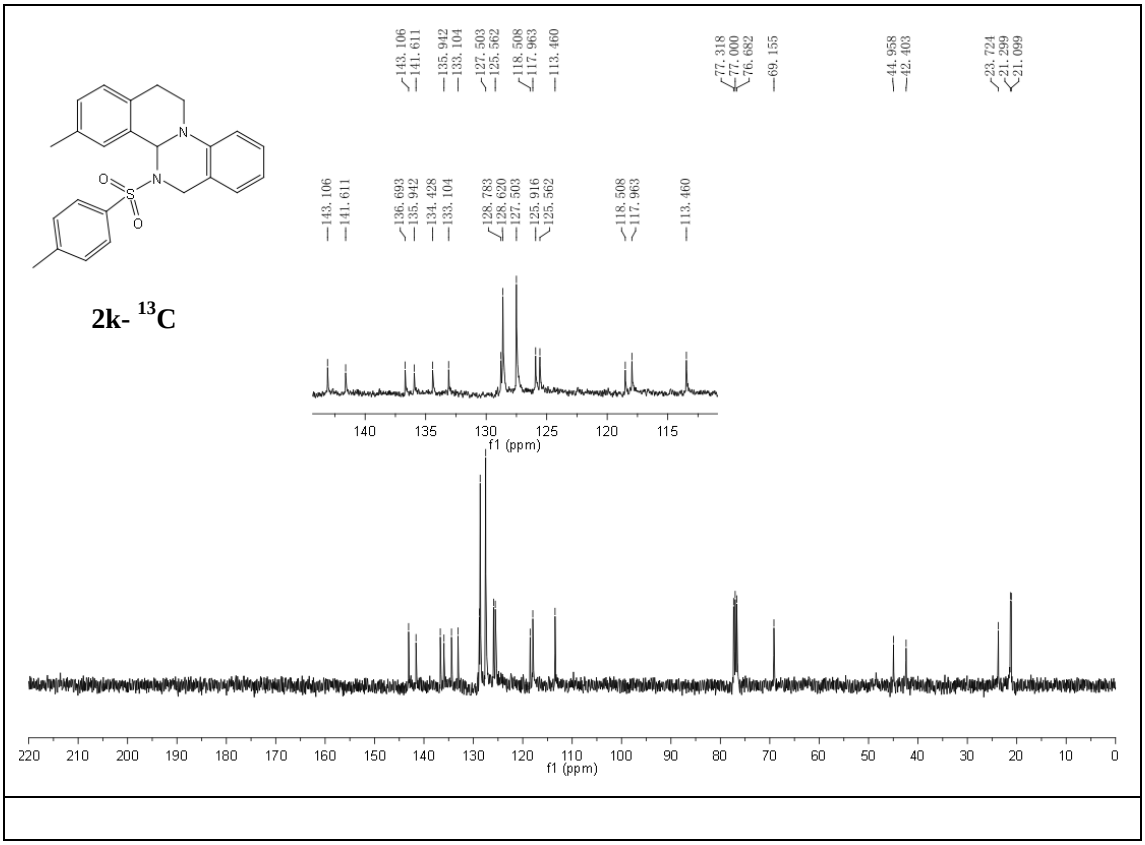
2i – ^{13}C


7.687
7.666
7.606
7.587
7.258
7.041
6.989
6.916
6.695
6.674
6.653
6.635
6.558
6.540
6.521
6.397
4.545
4.501
4.272
4.126
4.102
4.089
3.469
3.437
3.410
3.013
2.983
2.969
2.508
2.466
2.271
1.583
0.000
N
N
O
S
O
2.03
1.04
1.54
1.07
3.18
1.00
2.13
1.05
1.05
1.00
1.03
1.06
1.02
1.01
1.03
3.13
f1 (ppm)

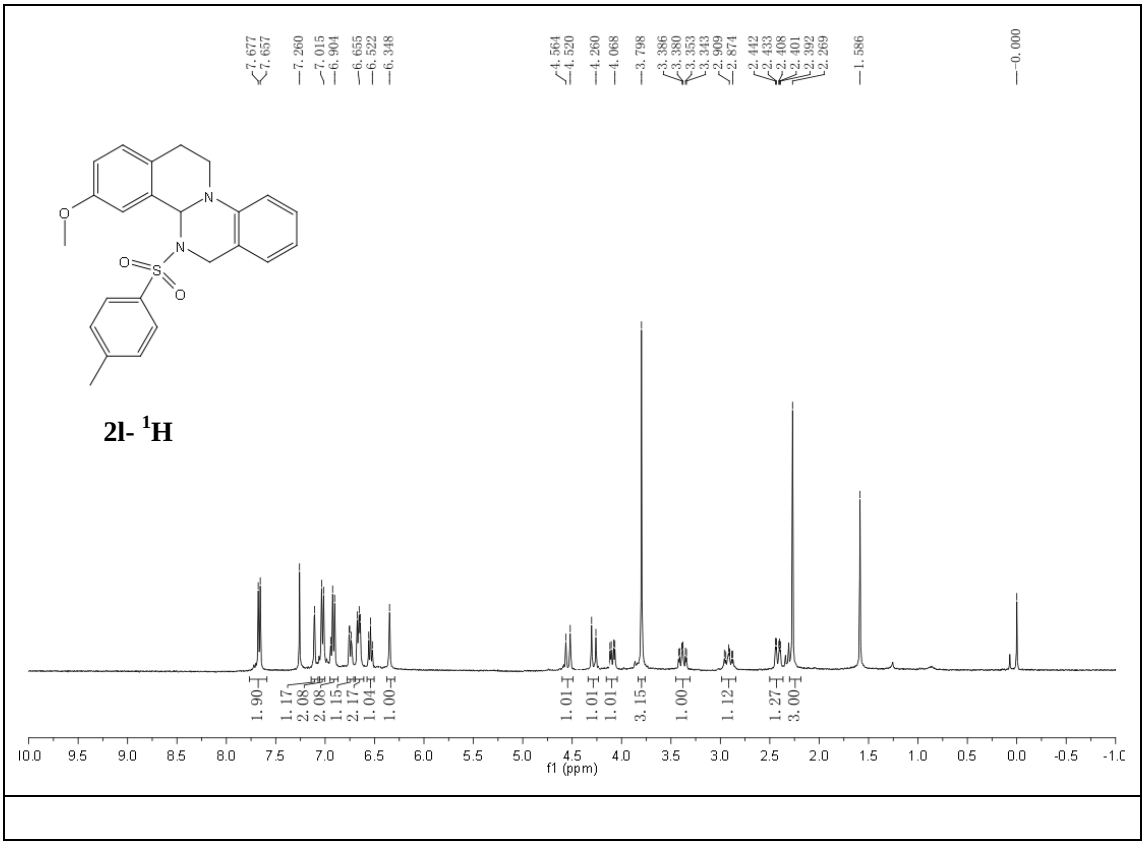
2j – ^{1}H


143.185
141.679
136.291
136.086
134.832
128.965
128.734
127.885
127.642
127.586
127.105
126.017
125.483
118.669
118.123
113.535
77.318
77.000
76.683
69.189
44.965
42.397
24.158
21.354
2j – 13C
f1 (ppm)

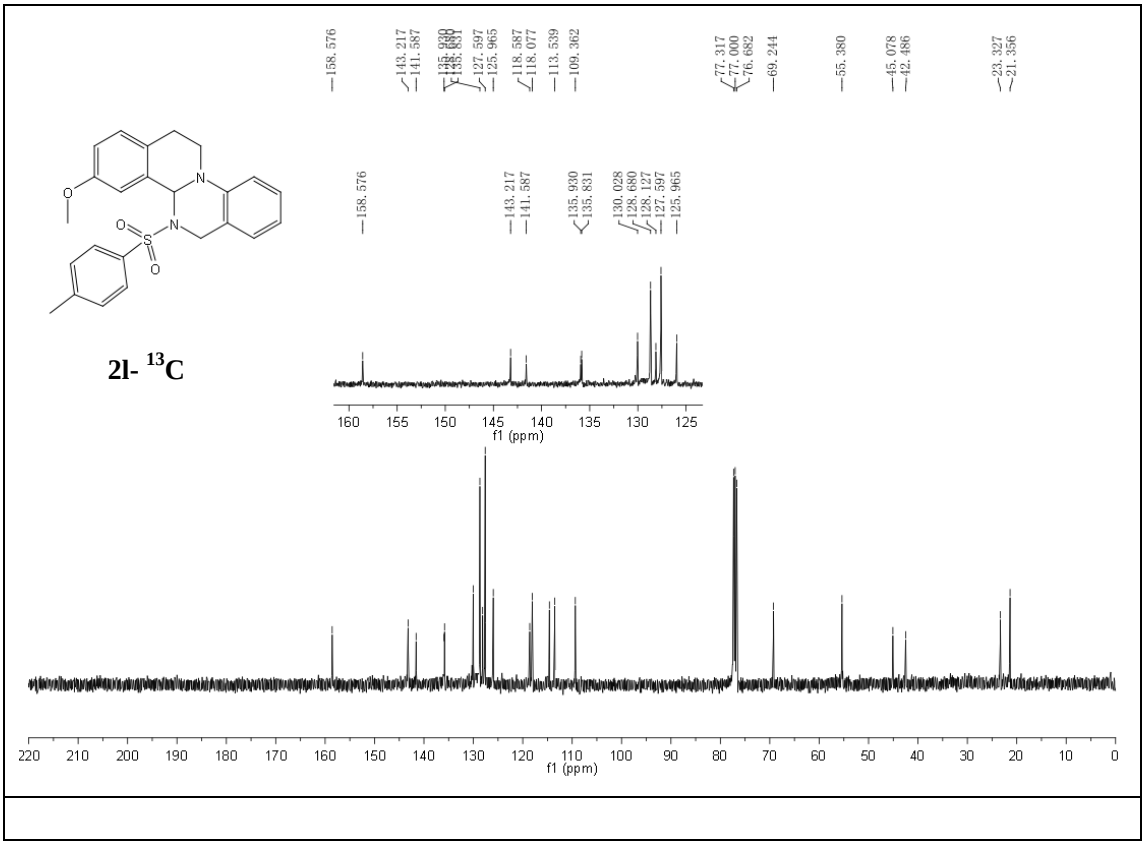

7.675
7.661
7.659
7.627
6.879
6.665
6.516
6.356
4.547
4.518
4.258
4.096
4.080
4.071
4.054
3.394
3.376
3.370
2.972
2.952
2.932
2.460
2.427
2.325
2.266
1.583
0.000
2k- 1H
f1 (ppm)


143.106
141.611
135.942
133.104
127.503
125.562
118.508
117.963
113.460
77.318
77.000
76.682
69.155
44.958
42.403
23.724
21.299
21.099
136.693
128.783
128.620
127.503
125.916
f1 (ppm)

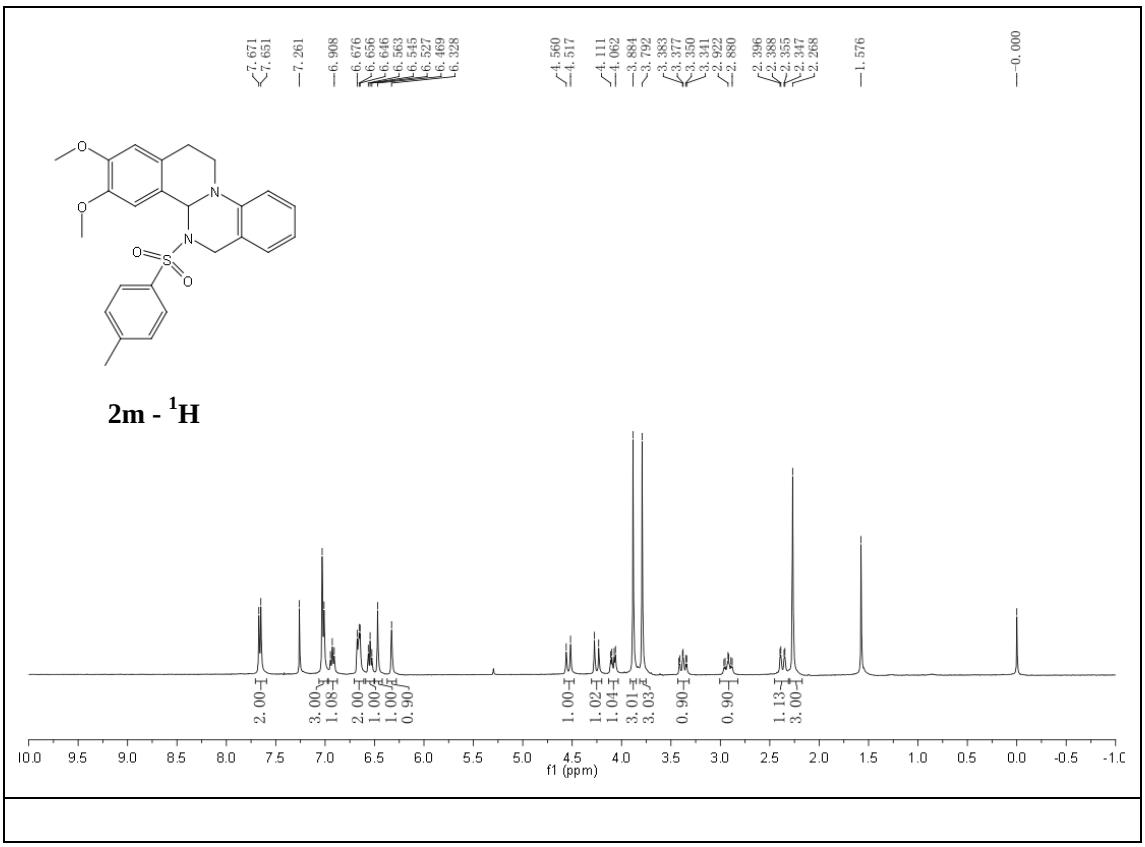
2k- ^{13}C


7.677
7.657
7.260
7.015
6.904
6.655
6.522
6.348
4.564
4.520
4.260
4.068
3.798
3.386
3.380
3.353
3.343
2.909
2.874
2.442
2.433
2.408
2.401
2.392
2.269
1.586
0.000
1.90
1.17
2.08
1.15
2.17
1.04
1.00
1.01
1.01
1.01
3.15
1.00
1.12
1.27
3.00
f1 (ppm)

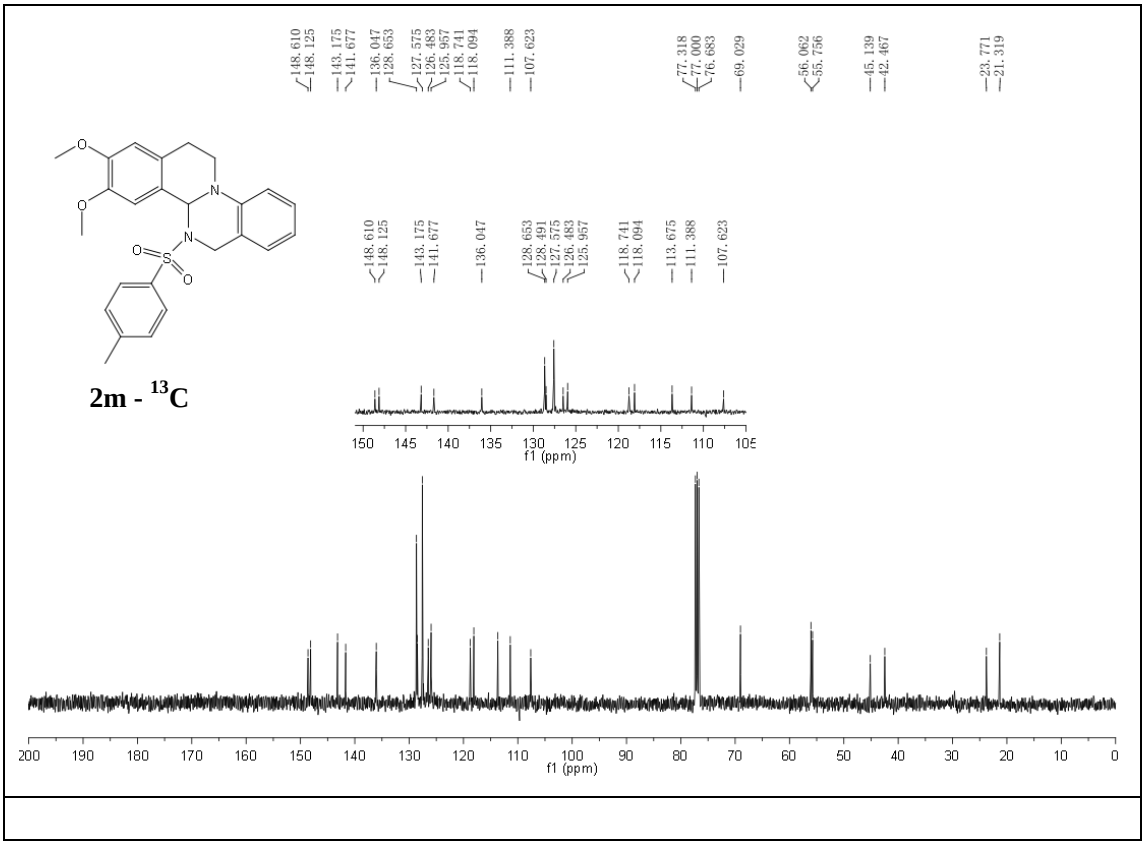
2l- ^{1}H

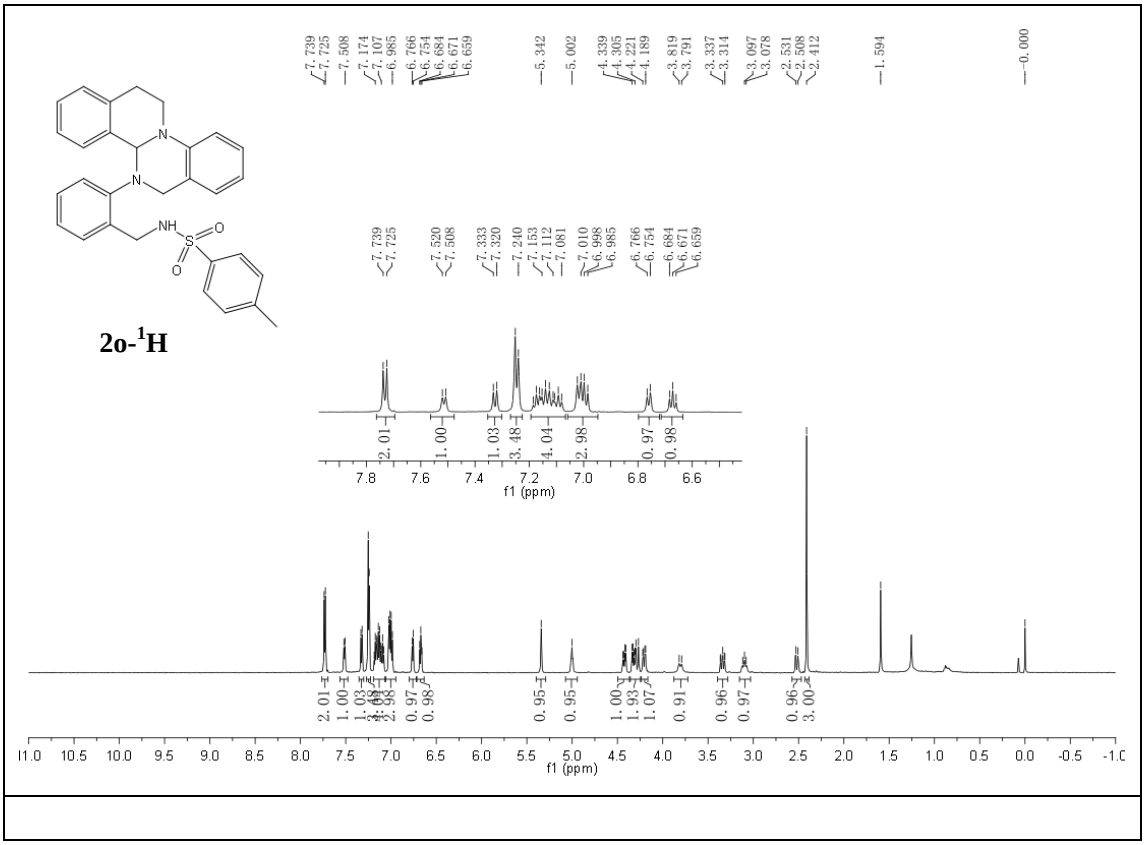

158.576
143.217
141.587
135.930
135.831
130.028
128.680
128.127
127.597
125.965
118.587
118.077
113.539
109.362
77.317
77.000
76.682
69.244
55.380
45.078
42.486
23.327
21.356
f1 (ppm)

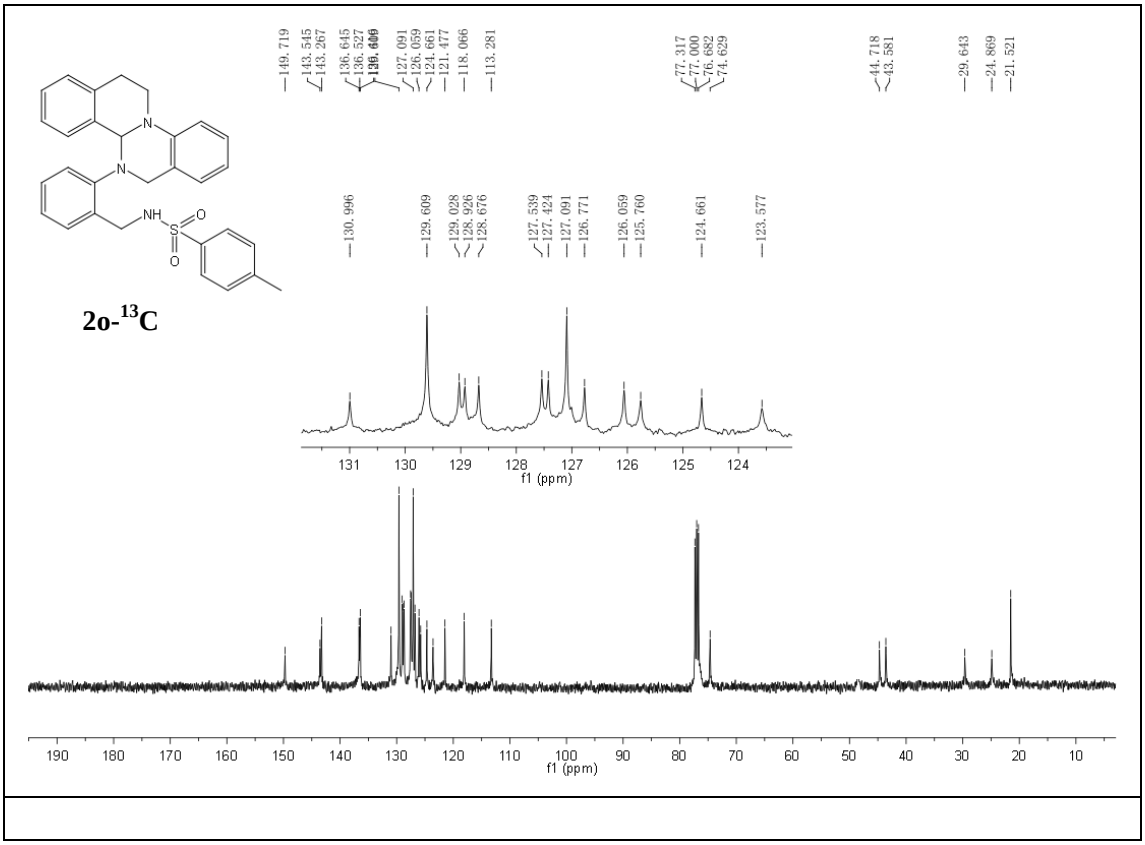
2l- ^{13}C


7.671
7.651
7.261
6.908
6.676
6.656
6.646
6.563
6.545
6.527
6.469
6.328
4.560
4.517
4.111
4.062
3.884
3.792
3.383
3.377
3.350
3.341
2.922
2.880
2.396
2.388
2.355
2.347
2.268
1.576
0.000
2.00
3.00
1.08
2.00
1.00
1.00
0.90
1.00
1.02
1.04
3.01
3.03
0.90
0.90
1.13
3.00
f1 (ppm)

2m - ^{1}H


148.610
148.125
143.175
141.677
136.047
128.653
127.575
126.483
125.957
118.741
118.094
111.388
107.623
77.318
77.000
76.683
69.029
56.062
55.756
45.139
42.467
23.771
21.319
2m - 13C
f1 (ppm)
f1 (ppm)

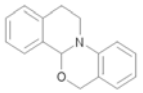

7.739
7.725
7.508
7.174
7.107
6.985
6.766
6.754
6.684
6.671
6.659
5.342
5.002
4.339
4.305
4.221
4.189
3.819
3.791
3.337
3.314
3.097
3.078
2.531
2.508
2.412
1.594
0.000
2o-1H
2.01
1.00
1.03
3.48
4.04
2.98
0.97
0.98
f1 (ppm)
f1 (ppm)


149.719
143.545
143.267
136.645
136.527
127.091
126.059
124.661
121.477
118.066
113.281
77.317
77.000
76.682
74.629
44.718
43.581
29.643
24.869
21.521
130.996
129.609
129.028
128.926
128.676
127.539
127.424
127.091
126.771
126.059
125.760
124.661
123.577
2o-13C
f1 (ppm)
f1 (ppm)

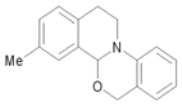
6. Copies of Elementary Analysis Date

华中师范大学分析测试中心 12.01.12
VarioEL III CHNS
serial number 11033032

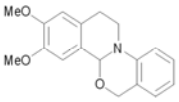
No.	Name	Weight [mg]	Content [%]
37	XJ616	1.3840	N: 5.626 C: 80.72 S: 0.000 H: 6.533
38	XJ766	1.5820	N: 5.310 C: 81.44 S: 0.000 H: 6.624
39	XJ727	1.3630	N: 4.520 C: 72.82 S: 0.000 H: 6.730
40	XJ770	1.3580	N: 5.539 C: 81.38 S: 0.000 H: 6.999
41	XJ729	1.3690	N: 5.152 C: 76.22 S: 0.000 H: 6.690
42	XJ772	1.1910	N: 5.059 C: 76.36 S: 0.000 H: 6.686
43	XJ730	1.3040	N: 9.770 C: 68.04 S: 0.000 H: 5.274
44	XJ773	1.3200	N: 5.345 C: 81.01 S: 0.000 H: 7.108
45	XJ775	1.4130	N: 7.133 C: 70.56 S: 8.184 H: 5.886



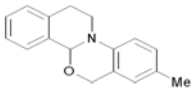
XJ-616 $C_{16}H_{15}NO$
Elemental Analysis: N, 5.90
C, 80.98
H, 6.37



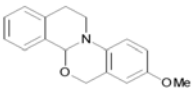
XJ-766 $C_{17}H_{17}NO$
Elemental Analysis: N, 5.57
C, 81.24
H, 6.82;



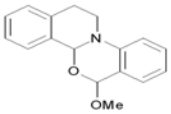
XJ-727 $C_{18}H_{19}NO_3$
Elemental Analysis: N, 4.71
C, 72.71
H, 6.44



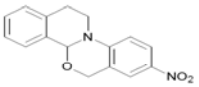
XJ-770 $C_{17}H_{17}NO$
Elemental Analysis: N, 5.57
C, 81.24
H, 6.82



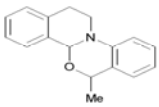
XJ-729 $C_{17}H_{17}NO_2$
Elemental Analysis: N, 5.24
C, 76.38
H, 6.41



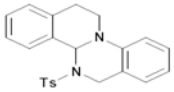
XJ-772 $C_{17}H_{17}NO_2$
Elemental Analysis: N, 5.24
C, 76.38
H, 6.41



XJ730 $C_{16}H_{14}N_2O_3$
Elemental Analysis: N, 9.92
C, 68.07
H, 5.00



XJ773 $C_{17}H_{17}NO$
Elemental Analysis: N, 5.57
C, 81.24
H, 6.82



XJ775 $C_{23}H_{22}N_2O_2S$
Elemental Analysis: N, 7.17
C, 70.74
S, 8.21
H, 5.68

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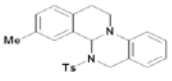
Document: F:\20101117\20111108-10-CHNS-OK.dat

华中师范大学分析测试中心 12.01.12

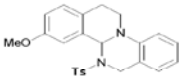
VarioEL III CHNS

serial number 11033032

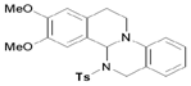
No.	Name	Weight [mg]	Content [%]
46	XJ826	1.2530	N: 6.648 C: 71.14 S: 7.961 H: 6.111
47	XJ833	1.4100	N: 6.373 C: 68.35 S: 7.699 H: 5.812
48	XJ801	1.3460	N: 6.012 C: 66.57 S: 7.278 H: 6.079
50	XJ827	1.3180	N: 8.190 C: 72.72 S: 6.337 H: 6.146
64	XJ812	1.4060	N: 8.337 C: 72.32 S: 6.438 H: 6.311



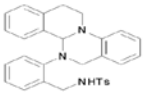
XJ-826 $C_{24}H_{24}N_2O_2S$
Elemental Analysis: N, 6.93
C, 71.26
S, 7.93
H, 5.98



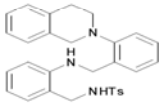
XJ-833 $C_{24}H_{24}N_2O_3S$
Elemental Analysis: N, 6.66
C, 68.55
S, 7.63
H, 5.75



XJ-801 $C_{26}H_{26}N_2O_4S$
Elemental Analysis: N, 6.22
C, 66.64
S, 7.12
H, 5.82



XJ-827 $C_{30}H_{29}N_3O_2S$
Elemental Analysis: N, 8.48
C, 72.70
S, 6.47
H, 5.90



XJ-812 $C_{30}H_{31}N_3O_2S$
Elemental Analysis: N, 8.44
C, 72.40
S, 6.44
H, 6.28

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Document: F:\20101117\20111108-10-CHNS-OK.dat