Supporting Information

For

One-Pot Enantioselective Construction of Indoloquinolizidine Derivatives Bearing Five Contiguous Stereocenters Using Aliphatic Aldehydes, Nitroethylenes, and Tryptamine **

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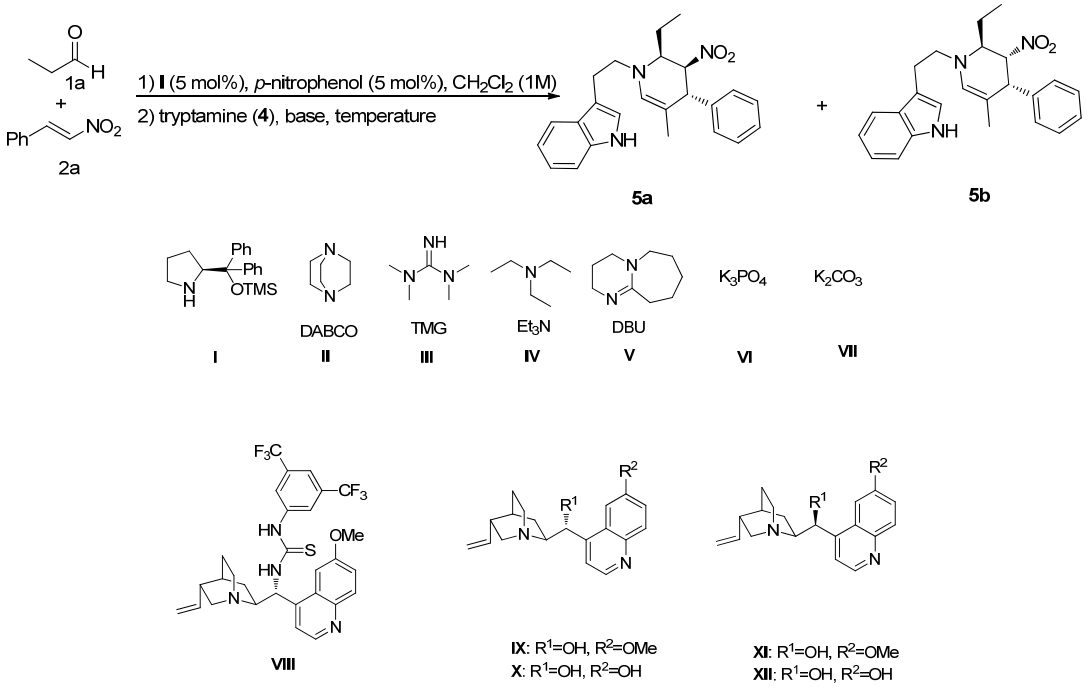
Table of Contents

1. General Experimental Methods:

NMR spectra were all recorded on a Bruke (400MHz) spectrometer. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal TMS on the δ scale. Data for ^{1}H NMR are reported as follows: chemical shift (δppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), integration, coupling constant (Hz) and assignment. Data for ^{13}C NMR are reported in terms of chemical shift and no special nomenclature is used for equivalent carbons. Flash column chromatography was performed using silica gel (300–400 mesh). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 300–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Commercial reagents and solvents were used as received. Catalyst **I** was commercial from Aldrich. Enantioselectivities were determined by high-performance liquid chromatography (HPLC) with a Jasco uv-2075 plus intelligent uv/ivs detector (λ = 254 nm orλ = 214 nm) andPhenomenex Lux5u Amylose-2 column. Optical rotations were measured in $CHCl_3$ on a Jasco P-1030 polarimeter. Dichloromethane was fractionally distilled.

2. Optimization ofMichael/aza-Henry/hemiaminalization/dehydrationsequence[a]

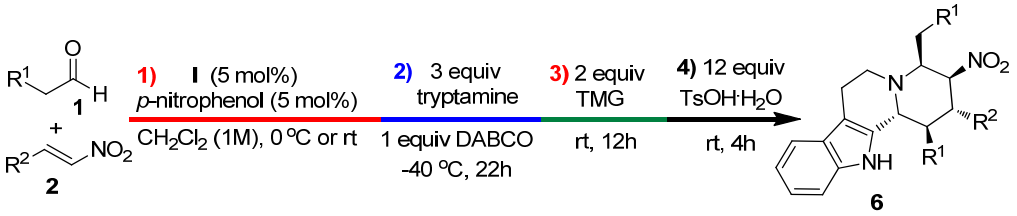


Entry	Cat.	Tempera ture(°C)	Time (h)	Yield of **5a**(%)[b]	**5a**:**5b**[c]	ee of **5a** (%)[d]
1	1 eq **II**	25	22	57	16 : 1	94
2	1 eq **III**	25	22	39	18 : 1	94
3	1 eq **IV**	25	22	45	16 : 1	94
4	1 eq **V**	25	22	42	> 20 : 1	94

5	1eq **VI**	25	22	22	1 : 1.8	94
6	1eq **VII**	25	22	33	1.1 : 1	94
7	0.1eq **VIII**	25	22	44	> 20 : 1	94
8	0.2eq **IX**	25	22	34	2.5 : 1	94
9	0.2eq **X**	25	22	42	3.6 : 1	94
10	0.2eq **XI**	25	22	38	15 : 1	94
11	0.2eq **XII**	25	22	41	7 : 1	94
12	1eq **II**	0	22	12	1 : 4.6	94
13[e]	1eq **II**	0	22	66	> 20 : 1	94
14[e]	1eq **II**	-20	22	70	> 20 : 1	95
15[e]	1eq **II**	-40	22	80	> 20 : 1	95
16[e]	1eq **II**	-60	22	38	> 20 : 1	95
17[e]	1eq **II**	-40	48	68	> 20 : 1	95
18[e]	1eq **II**	-60	48	46	> 20 : 1	95
19[e]	2eq **II**	-40	22	64	> 20 : 1	95
20[e]	0.5eq **II**	-40	22	61	> 20 : 1	95
21[e]	0.5eq **II**	-40	48	64	> 20 : 1	95
22[ef]	1eq **II**	-40	22	75	> 20 : 1	97
23[eg]	1eq **II**	-40	22	46	> 20 : 1	97

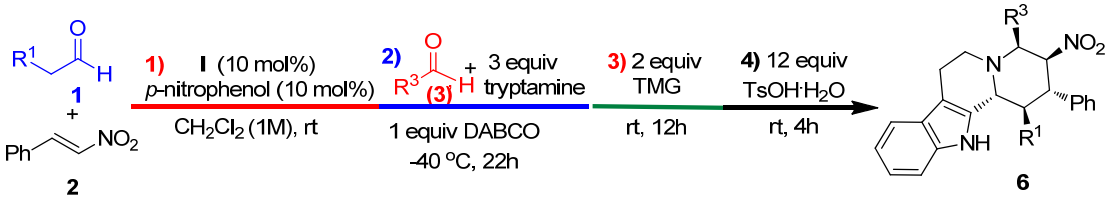
[a] The reaction was performed using **1a** (0.75 mmol), **2a** (0.25 mmol, 1M), *p*-nitrophenol (0.0125 mmol), **I** (0.0125 mmol) at rt. After **2a** consumed, **4** (0.75 mmol), base and 4 Å molecular sieves (MS) (50mg) was added to the mixture. [b] Yield of isolated product **5a**. [c] Determined by ^{1}H NMR of the crude product. [d] Determined by chiral HPLC analysis. [e] 4 mL CH_2Cl_2 and TMG (0.5 mmol) was added to stir for another 12h at rt. [f] The first step was performed at 0 ℃. [g] The first step was performed at -20 ℃.

3. General procedure for the synthesis ofindoloquinolizidine derivatives 6a-6m



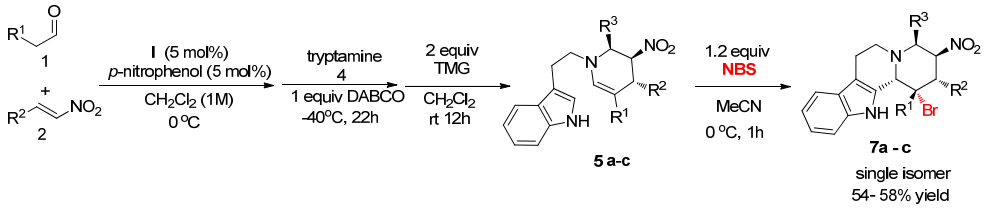
A solution of nitroalkene nitroethylenes **2** (0.25 mmol), *p*-nitrophenol (0.125 mmol) and catalyst **I** (0.0125mmol) in CH_2Cl_2 (0.25 mL) was added with aldehyade **1** (0.75 mmol). The result mixture was stirred at 0 °C (**1** = propanal) or rt until all the nitroalkene was consumed, then cooled to -40 °C, added with DABCO(0.25mmol), tryptamine **4**(0.75 mmol) and 4Å MS (50 mg). The mixture was kept at -40°C for 22 hours then treated with 4mL $CH_2Cl_2$and TMG (0.5 mmol) at rt for further 12h. Finally, $TsOH{\cdot}H_2O$ (3 mmol) was added to the reaction mixture to stir for 4h at rt before filtered through a short pad of celite. The filtrate was concentrated and purified on silica gel to afford the desired products.

4. General procedure for the synthesis ofindoloquinolizidine derivatives 6n-6q



A solution of nitroalkene **2** (0.25 mmol), *p*-nitrophenol (0.025 mmol) and catalyst **I** (0,025 mmol) in CH_2Cl_2 (0.25 mL) was added with aldehyade **1** (0.30 mmol). The result mixture was stirred at room temperature until all the nitroalkene was consumed, then cooled to -40℃, added with DABCO (0.25mmol), aldehyade **3** (0.75 mmol), tryptamine **4** (0.75 mmol) and 4Å MS (50 mg). The mixture was kept at -40°C for 22 hours then treated with 4mL $CH_2Cl_2$and TMG (0.5 mmol) at rt for further 12h. Finally, $TsOH{\cdot}H_2O$ (3 mmol) was added to the reaction mixture to stir for 4h at rt before filtered through a short pad of celite. The filtrate was concentrated and purified on silica gel to afford the desired products.

5. Procedure for the synthesis of indoloquinolizidine derivative 7

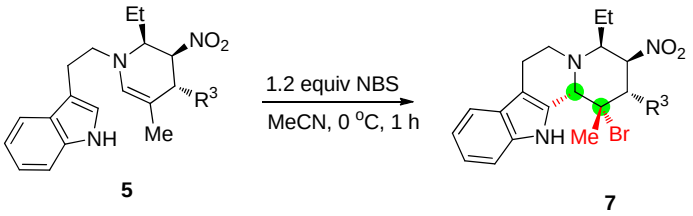


A solution of nitroalkene **2** (0.25 mmol), *p*-nitrophenol (0.0125 mmol) and catalyst **I** (0.0125 mmol) in CH_2Cl_2 (0.25 mL) was added with aldehyade **1** (0.75 mmol). The result mixture was stirred at 0 °C until all the nitroalkene was consumed, then cooled to -40 °C, added with DABCO(0.25 mmol), tryptamine **4** (0.75 mmol) and 4Å MS (50 mg). The mixture was kept at -40°C for 22 hours then treated with 4mL CH_2Cl_2 and TMG (0.5mmol) at rt for further 12h before filtered through a short pad of celite.The filtrate was concentrated and purified on silica gel to afford products **5a-c**.

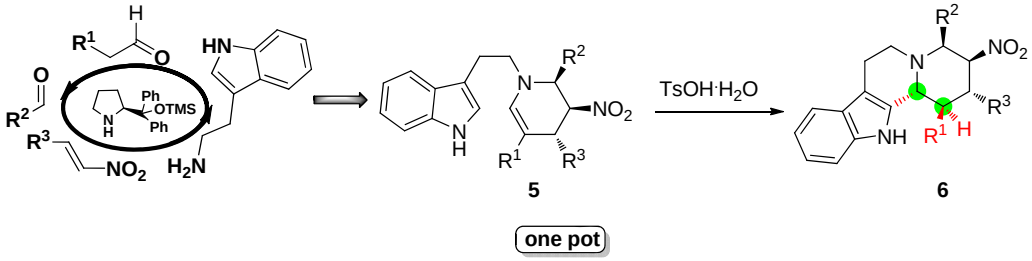
A solution of **5** (0.10 mmol) in MeCN(2 mL) was cooled to 0 °C, then added with NBS (0.12 mmol). After 1 hour, the result mixture was purified by flash chromatography on a short silica gel directly to afford the desired product**7a-c**.

6. Explanations of the determinations of compounds 5a and 6a's absolute configurations.

The absolute configurations shown in scheme 3 and 4 in manuscript were determined as follows: First, the absolute configuration of the bromide-containing indoloquinolizidines **7** was unambiguously determined by X-ray crystallography of **7a** (Fig. 1). Indoloquinolizidines **7** were obtained from treating products **5** with bromosuccinimide (NBS), and have two more new-generated stereocenters. The absolute configuration of stereocenters in product **5** should be the same as those in products **7** except these new-generated stereocenters (eq. 1). Therefore, the absolute configuration of products **5** can be assigned reasonably and scientifically according to X-ray crystallography of **7a**. On the other hand, the relative configuration of the products **6** can be surely determined through X-ray crystallography of **6c** (Fig. 2). After the absolute configuration of products **5** confirmed, and with the relative configuration of the products **6** in hand, the absolute configuration of products **6** can be assigned in a similar way without arbitrariness (eq. 2).



5 **7** **eq. 1**



5 **6** **eq. 2**

one pot

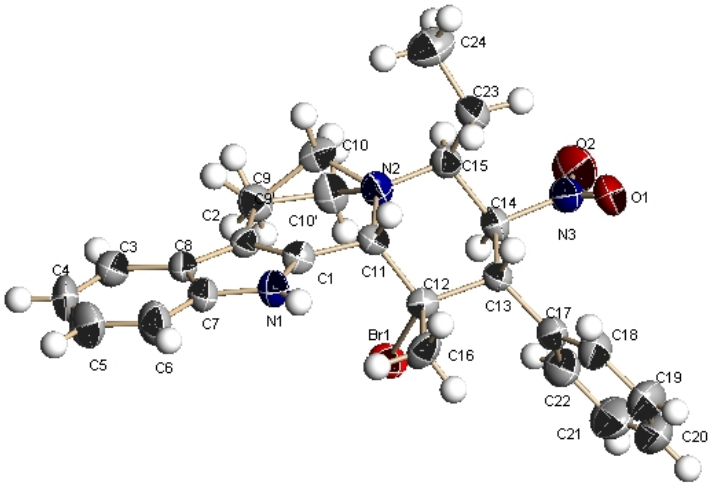


Figure 1. X-ray crystallography of **7a**

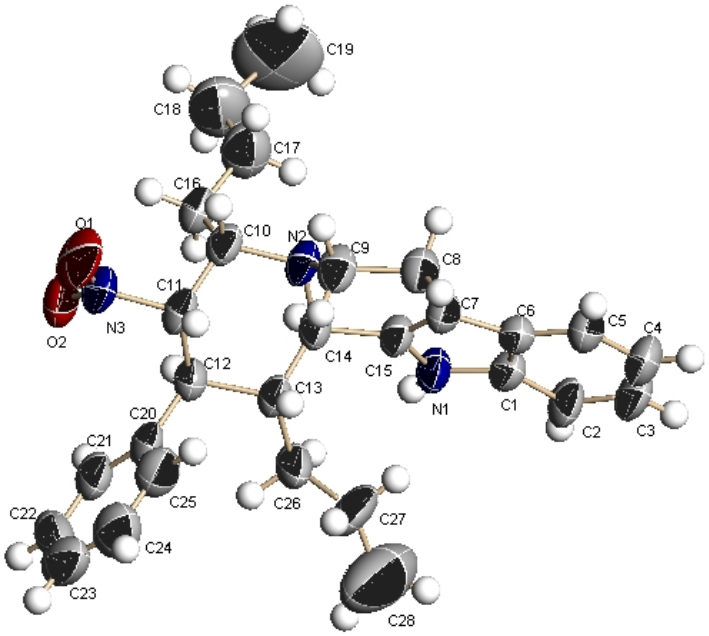
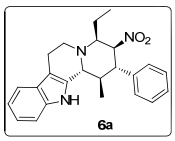


Figure 2. X-ray crystallography of **6c**

7. Characterization data of indoloquinolizidine derivatives



(1*R*,2*S*,3*S*,4*S*,12b*R*)-4-ethyl-1-methyl-3-nitro-2-phenyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6a)

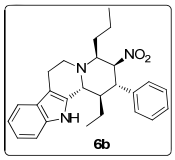
Purified by flash chromatography (PE:EtOAc = 20:1). 50% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH 90/10);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 9.6 min, τ_{major} = 21.6 min, 97% ee.

$[\alpha]_D^{34}$ = - 11.4 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.30 – 7.27 (m, 3H), 7.23 – 7.19 (m, 3H), 7.16 (t, *J* = 6.8 Hz, 1H), 7.10 (t, *J* = 7.2 Hz, 1H), 5.38 (dd, *J* = 12.0, 5.2 Hz, 1H), 3.83 (d, *J* = 10.4 Hz, 1H), 3.70 (td, *J* = 10.8, 4.4 Hz, 1H), 3.52 – 3.47 (m, 1H), 3.29 (t, *J* = 12.0 Hz, 1H), 3.11 – 3.07(m, 1H), 3.02 – 2.94 (m, 1H), 2.86 – 2.81 (m, 1H), 2.33 – 2.26 (m, 1H), 1.99 – 1.91 (m, 1H), 1.45– 1.39 (m, 1H), 0.94 – 0.91 (m, 6H).^{13}C NMR (100 MHz, $CDCl_3$) δ 139.2, 135.8, 133.7, 128.7, 128.3, 127.4, 126.6, 121.9, 119.5, 118.2, 110.7, 109.2, 87.4, 66.1, 54.3, 47.9, 47.7, 36.7, 22.6, 18.5, 16.0, 11.0.

IR (KBr) *v* (cm^{-1})3430, 2955, 3063, 3031, 2923, 2851,1540, 1467, 1366, 1347, 1163, 1086，742, 698.

HRMS (ESI)*m/z* calcd for $C_{24}H_{27}N_3O_2$ $[M+H]^+$390.2176, found: 390.2181



(1*R*,2*S*,3*S*,4*S*,12b*R*)-4-ethyl-1-methyl-3-nitro-2-phenyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6b)

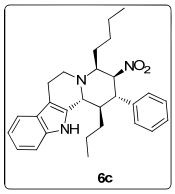
Purified by flash chromatography (PE:EtOAc = 20:1). 38% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 90/10);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 6.7min, τ_{major} = 18.0 min, 95% *ee*.

$[\alpha]_D^{34}$ = - 12.6(c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (s, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.31 – 7.26 (m, 5H), 7.23 – 7.21 (m, 1H), 7.16 (t, *J* = 7.2 Hz, 1H), 7.10 (t, *J* = 6.8 Hz, 1H), 5.37 (dd, *J* = 12.0, 5.6 Hz, 1H), 4.16 (d, *J* = 10.8 Hz, 1H), 3.72 (td, *J* = 10.8, 4.4 Hz, 1H), 3.63 (t, *J* = 12.0 Hz, 1H), 3.58 – 3.54 (m, 1H), 3.11 – 3.06 (m, 1H), 3.01 – 2.93 (m, 1H), 2.85 – 2.81 (m, 1H), 2.39 – 2.33 (m, 1H), 1.99 – 1.90 (m, 1H), 1.55 – 1.48 (m, 2H), 1.31 – 1.22 (m, 4H), 1.01 (t, *J* = 7.6 Hz, 3H), 0.88 (t, *J* = 7.2 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 138.9, 135.9, 133.7, 128.7, 127.4, 126.7, 121.9, 119.5, 118.2, 110.6, 109.5, 87.8, 64.1, 49.7, 47.6, 43.4, 40.6, 27.5, 22.6, 20.2, 19.6, 13.7, 8.4.

IR (KBr) *v* (cm^{-1})3433，3056，3028，2961，2870，1540，1461，1369，1350，1166，1086，742, 701.

HRMS (ESI)*m/z* calcd for $C_{26}H_{31}N_3O_2$ $[M+H]^+$418.2489, found: 418.2484



6c

(1*R*,2*S*,3*S*,4*S*,12b*R*)-4-butyl-3-nitro-2-phenyl-1-propyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6c)

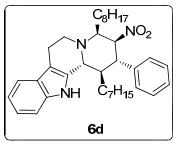
Purified by flash chromatography (PE:EtOAc = 20:1). 42% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 90/10);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 6.1min, τ_{major} = 13.8 min, 97% *ee*.

$[\alpha]_D^{34}$ = - 10.0(c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.31 – 7.20 (m, 6H), 7.17 (td, *J* = 7.2 Hz, 1.2Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 1H), 5.37 (dd, *J* = 12.0, 5.6 Hz, 1H), 4.12 (d, *J* = 10.8 Hz, 1H), 3.73 (td, *J* = 11.2, 4.8 Hz, 1H), 3.62 – 3.53 (m, 2H), 3.11 – 3.07 (m, 1H), 3.02 – 2.94 (m, 1H), 2.86 – 2.82 (m, 1H), 2.36 – 2.29 (m, 1H), 1.97 – 1.87 (m, 1H), 1.48 – 1.13 (m, 9H), 0.87 – 0.80 (6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.0, 135.9, 133.8, 128.7, 127.4, 126.8, 121.9, 119.6, 118.2, 110.6, 109.5, 87.7, 64.4, 50.7, 47.6, 44.4, 40.6, 30.4, 28.6, 25.2, 22.6, 22.4, 17.8, 14.6, 14.0.

IR (KBr) *v* (cm^{-1})3417，3063，3025，2952，2927，2860，1549，1530，1496，1461，1366，1343, 1166, 1087, 751, 701, 571, 485.

HRMS (ESI)*m/z* calcd for $C_{28}H_{35}N_3O_2$ $[M+H]^+$446.2802, found: 446.2810



6d

(1*R*,2*S*,3*S*,4*S*,12b*R*)-1-heptyl-3-nitro-4-octyl-2-phenyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6d)

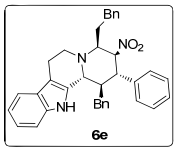
Purified by flash chromatography (PE:EtOAc = 100:1). 34% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 90/10);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 7.0min, τ_{major} = 19.6 min, 97% *ee*.

$[\alpha]_D^{34}$ = - 21.0 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.31 – 7.20 (m, 6H), 7.17 (t, *J* = 6.8 Hz, 1H), 7.11 (t, *J* = 6.8 Hz, 1H), 5.37 (dd, *J* = 12.0, 5.6 Hz, 1H), 4.12 (d, *J* = 11.2 Hz, 1H), 3.73 (td, *J* = 11.2, 4.8 Hz, 1H), 3.62 – 3.53 (m, 2H), 3.11 – 3.07 (m, 1H), 3.02 – 2.94 (m, 1H), 2.86 – 2.82 (m, 1H), 2.36 – 2.29 (m, 1H), 1.97 – 1.87 (m, 1H), 1.48 – 1.13 (m, 9H), 0.87 – 0.80 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.0, 135.9, 133.8, 128.7, 127.4, 126.8, 121.9, 119.6, 118.2, 110.6, 109.5, 87.7, 64.4, 50.7, 47.6, 44.4, 40.6, 30.4, 28.6, 25.2, 22.6, 22.4, 17.8, 14.6, 14.0.

IR (KBr) *v* (cm^{-1})3433, 3060, 3031, 2923, 2857, 1540, 1458, 1369, 1344, 1166, 1102, 739, 698, 565.

HRMS (ESI)*m/z* calcd for $C_{37}H_{55}N_3O_2$ $[M+H]^+$558.4054, found: 558.4064



(1*R*,2*S*,3*S*,4*S*,12b*R*)-1-benzyl-3-nitro-4-phenethyl-2-phenyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6e)

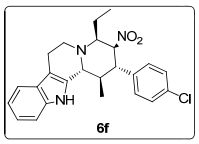
Purified by flash chromatography (PE:EtOAc = 20:1). 35% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 90/10);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 10.0min, τ_{major} = 22.5 min, 91% *ee*.

$[\alpha]_D^{34}$ = - 40.0 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.52 – 7.50 (m, 1H), 7.24 –7.15 (m, 8H), 7.13 – 7.08 (m, 8H), 6.83 – 6.81 (m, 2H), 5.34 (dd, *J* = 11.6, 5.2 Hz, 1H), 4.28 (d, *J* = 10.4 Hz, 1H), 3.76 – 3.70 (m, 1H), 3.62 (t, *J* = 11.6 Hz, 1H), 3.53 – 3.47 (m, 1H), 3.08 – 3.00 (m, 2H), 2.90 – 2.84 (m, 2H), 2.81 – 2.71 (m, 3H), 2.62 – 2.54 (m, 1H), 2.28 – 2.19 (m, 1H), 1.70 – 1.61 (m, 1H).^{13}C NMR (100 MHz, $CDCl_3$) δ 141.2, 138.8, 138.5, 135.9, 133.2, 128.7, 128.5, 128.5, 128.3, 127.6, 126.5, 126.3, 125.9, 121.9, 119.5, 118.1, 110.7, 109.6, 87.8, 62.8, 52.0, 47.5, 46.0, 41.7, 35.9, 31.7, 27.0, 22.6.

IR (KBr) *v* (cm^{-1})3424, 3056, 3025, 2945, 2914, 2844, 1603, 1543, 1492, 1451, 1362, 1340, 1163, 1078, 745, 698.

HRMS (ESI)*m/z* calcd for $C_{37}H_{39}N_3O_2$ $[M+H]^+$542.2802, found: 542.2808



(1*R*,2*S*,3*S*,4*S*,12b*R*)-2-(4-chlorophenyl)-4-ethyl-1-methyl-3-nitro-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6f)

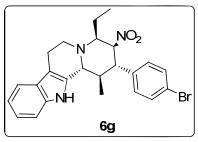
Purified by flash chromatography (PE:EtOAc = 20:1). 45% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 5.6min, τ_{major} = 10.8 min, 96% *ee*.

$[\alpha]_D^{34}$ = - 6.0 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (s, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.30 – 7.23 (m, 3H), 7.19 – 7.14 (m, 3H), 7.11 (t, *J* = 8.0 Hz, 1H), 5.31 (dd, *J* = 12.0, 5.6 Hz, 1H), 3.81 (d, *J* = 10.4 Hz, 1H), 3.67 (td, *J* = 11.2, 4.8 Hz, 1H), 3.51 – 3.46 (m, 1H), 3.28 (t, *J* = 11.6 Hz, 1H), 3.10 – 3.06 (m, 1H), 3.02 – 2.94 (m, 1H), 2.86 – 2.81 (m, 1H), 2.28 – 2.21 (m, 1H), 1.97 – 1.89 (m, 1H), 1.43 – 1.36 (m, 1H), 0.94 – 0.90 (m, 6H).^{13}C NMR (100 MHz, $CDCl_3$) δ 137.7, 135.8, 133.5, 133.2, 129.6, 128.9, 126.6, 122.0, 119.5, 118.3, 110.7, 109.3, 87.2, 66.1, 54.2, 47.9, 47.2, 36.6, 22.5, 18.5, 15.9, 11.0.

IR (KBr) *v* (cm^{-1})3427, 2961, 2920, 2851, 1543, 1489, 1458, 1372, 1340, 1166, 1090, 1011, 818, 742.

HRMS (ESI)*m/z* calcd for $C_{25}H_{30}ClN_3O_2$$[M+H]^+$424.1786, found: 424.1768



(1*R*,2*S*,3*S*,4*S*,12b*R*)-2-(4-bromophenyl)-4-ethyl-1-methyl-3-nitro-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6g)

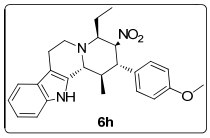
Purified by flash chromatography (PE:EtOAc = 20:1). 40% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 6.0 min, τ_{major} = 11.2 min, 94% *ee*.

$[\alpha]_D^{34}$ = - 41.2 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.59 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.31 – 7.27 (m, 2H), 7.20 – 7.05 (m, 4H), 5.41 (dd, *J* = 12.0, 5.6 Hz, 1H), 4.15 (t, *J* = 12.0 Hz, 1H), 3.91 (d, *J* = 10.8 Hz, 1H), 3.69 (td, *J* = 10.8, 4.4 Hz, 1H), 3.58 – 3.53 (m, 1H), 3.14 – 3.10 (m, 1H), 3.03 – 2.95 (m, 1H), 2.85 – 2.82 (m, 1H), 2.27 – 2.16 (m, 1H), 2.08 – 1.96 (m, 1H), 1.48 – 1.39 (m, 1H), 1.00 – 0.92 (m, 6H).^{13}C NMR (100 MHz, $CDCl_3$) δ 139.0, 135.8, 133.4, 133.3, 128.6, 128.2, 127.4, 126.6, 121.9, 119.5, 118.2, 110.7, 109.4, 86.7, 66.4, 54.5, 48.0, 45.0, 38.4, 22.6, 18.5, 15.0, 11.0.

IR (KBr) *v* (cm^{-1})3430, 2958, 2927, 1628, 1540, 1458, 1369, 1337, 1163, 1074, 1008, 748.

HRMS (ESI)*m/z* calcd for $C_{24}H_{26}BrN_3O_2[M+H]^+$468.1281, found: 468.1275



(1*R*,2*S*,3*S*,4*S*,12b*R*)-4-ethyl-2-(4-methoxyphenyl)-1-methyl-3-nitro-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6h)

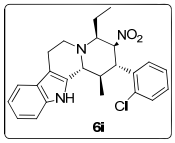
Purified by flash chromatography (PE:EtOAc = 10:1). 30% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 7.5min, τ_{major} = 19.3 min, 97% *ee*.

$[\alpha]_D^{34}$ = - 16.2 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.29 (d, *J* = 7.6 Hz, 1H), 7.18 – 7.08 (m, 4H), 6.82 (d, *J* = 8.4 Hz, 2H), 5.33 (dd, *J* = 12.0, 5.2 Hz, 1H), 3.82 (d, *J* = 10.4 Hz, 1H), 3.76 (s, 3H), 3.70 (td, *J* = 10.8, 4.4 Hz, 1H), 3.50 – 3.45 (m, 1H), 3.24 (t, *J* = 12.0 Hz, 1H), 3.11 – 3.07 (m, 1H), 3.02 – 2.94 (m, 1H), 2.86 – 2.81 (m, 1H), 2.31 – 2.21 (m, 1H), 2.00 – 1.89 (m, 1H), 1.45–1.37 (m, 1H), 0.95 – 0.90 (m, 6H).^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7,135.8, 133.8, 131.1, 129.2, 126.7, 121.9, 119.48, 118.2, 114.1, 110.7, 109.2, 87.7, 66.1, 55.2, 54.4, 47.9, 46.9, 36.8, 22.6, 18.5, 16.0, 11.0.

IR (KBr) *v* (cm^{-1})3427, 2958, 2927, 1616, 1540, 1511, 1458, 1366, 1340, 1248, 1172, 1036, 745.

HRMS (ESI)*m/z* calcd for $C_{26}H_{33}N_3O_3[M+H]^+$420.2282, found: 420.2285



(1*R*,2*S*,3*S*,4*S*,12b*R*)-2-(2-chlorophenyl)-4-ethyl-1-methyl-3-nitro-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6i)

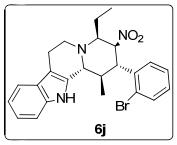
Purified by flash chromatography (PE:EtOAc = 20:1). 55% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 5.6min, τ_{major} = 10.4 min, 96% *ee*.

$[\alpha]_D^{34}$ = - 9.6(c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (s, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.30– 7.23 (m, 3H), 7.19 – 7.09 (m, 4H), 5.31 (dd, *J* = 12.0, 5.2 Hz, 1H), 3.82 (d, *J* = 10.4 Hz, 1H), 3.68 (td, *J* = 10.8, 4.4 Hz, 1H), 3.59 – 3.42 (m, 1H), 3.28 (t, *J* = 11.6 Hz, 1H), 3.15 – 3.04 (m, 1H), 3.03 – 2.93 (m, 1H), 2.90 – 2.71 (m, 1H), 2.36 – 2.15 (m, 1H), 1.99 – 1.87 (m, 1H), 1.45 – 1.35 (m, 1H), 0.94 – 0.90 (m, 6H).^{13}C NMR (100 MHz, $CDCl_3$) δ137.7, 135.8, 133.5, 133.2, 129.6, 128.9, 126.6, 122.0, 119.5, 118.3, 110.7, 109.3, 87.2, 66.1, 54.2, 47.9,47.2, 36.6, 22.5, 18.5, 15.9, 10.1.

IR (KBr) *v* (cm^{-1})3424, 2920, 2847, 1543, 1489, 1461, 1372, 1337, 1163, 1087, 821, 742.

HRMS (ESI)*m/z* calcd for $C_{25}H_{30}ClN_3O_2[M+H]^+$424.1786, found: 424.1763



(1*R*,2*S*,3*S*,4*S*,12b*R*)-2-(2-bromophenyl)-4-ethyl-1-methyl-3-nitro-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6j)

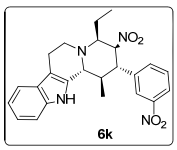
Purified by flash chromatography (PE:EtOAc = 20:1). 37% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 19.9min, τ_{major} = 21.4 min, 96% *ee*.

$[\alpha]_D^{34}$ = - 37.0 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.59 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.31 – 7.24 (m, 2H), 7.20 – 7.05 (m, 4H), 5.41 (dd, *J* = 12.0, 5.2 Hz, 1H), 4.15 (t, *J* = 11.6 Hz, 1H), 3.91 (d, *J* = 10.4 Hz, 1H), 3.69 (td, *J* = 10.8, 4.4 Hz, 1H), 3.57 – 3.52 (m, 1H), 3.13 – 3.09 (m, 1H), 3.03 – 2.95 (m, 1H), 2.86 – 2.81 (m, 1H), 2.24 – 2.17 (m, 1H), 2.06 – 1.98 (m, 1H), 1.48 – 1.41 (m, 1H), 0.99 (d, *J* = 6.4 Hz, 3H), 0.95 (t, *J* = 7.2 Hz, 4H).^{13}C NMR (100 MHz, $CDCl_3$) δ 139.0, 135.8, 133.3, 128.6, 128.2, 127.4, 126.6, 121.9, 119.5, 118.2, 110.7, 109.3, 86.7, 66.4, 54.5, 48.0, 44.9, 38.4, 22.6, 18.5, 15.0, 11.0.

IR (KBr) *v* (cm^{-1})3430, 2965, 2927, 2876, 2844, 1546, 1461, 1366, 1340, 1163, 1084, 1017, 742.

HRMS (ESI)*m/z* calcd for $C_{24}H_{26}BrN_3O_2[M+H]^+$468.1281, found: 468.1270



(1*R*,2*S*,3*S*,4*S*,12b*R*)-4-ethyl-1-methyl-3-nitro-2-(3-nitrophenyl)-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6k)

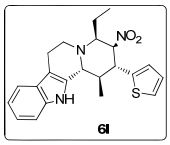
Purified by flash chromatography (PE:EtOAc = 10:1). 44% yield, yellow solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 98/2);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 19.9min, τ_{major} = 21.4 min, 98% *ee*.

$[\alpha]_D^{34}$ = - 7.8(c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (s, 1H), 8.10 (d, *J* = 8.0 Hz, 1H), 7.77 (s, 1H), 7.59 (d, *J* = 7.6 Hz, 1H), 7.53 – 7.46 (m, 2H), 7.30 (d, *J* = 7.6 Hz, 1H), 7.17 (t, *J* = 6.8 Hz, 1H), 7.12 (d, *J* = 7.2 Hz, 1H), 5.38 (dd, *J* = 11.6, 5.2 Hz, 1H), 3.86 (d, *J* = 10.4 Hz, 1H), 3.71 (td, *J* = 11.2, 4.4 Hz, 1H), 3.57 – 3.52 (m, 1H), 3.47 (t, *J* = 11.6 Hz, 1H), 3.14 – 3.10 (m, 1H), 3.04 – 2.96 (m, 1H), 2.88 – 2.84 (m, 1H), 2.36 – 2.36 (m, 1H), 1.99 – 1.91 (m, 1H), 1.43 – 1.36 (m, 1H), 0.96 – 0.92 (m, 6H).^{13}C NMR (100 MHz, $CDCl_3$) δ 148.4, 141.6, 135.9, 133.1, 129.8, 126.6, 122.7, 122.0, 119.6, 118.3, 110.7, 109.4, 86.8, 66.1, 54.1, 47.9, 47.6, 36.6, 22.5, 18.4, 15.9, 10.9.

IR (KBr) ν (cm-1)3429, 2963, 2927, 2873, 2844, 1616, 1542, 1530, 1461, 1350, 1166, 1087, 739, 691.

HRMS (ESI)*m/z* calcd for $C_{25}H_{30}N_4O_4[M+H]^+$435.2027, found: 435.2017



(1*R*,2*R*,3*S*,4*S*,12b*R*)-4-ethyl-1-methyl-3-nitro-2-(thiophen-2-yl)-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6l)

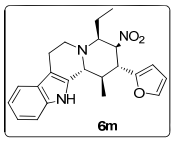
Purified by flash chromatography (PE:EtOAc = 20:1). 44% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 5.8min, τ_{major} = 10.4 min, 96% *ee*.

$[\alpha]_D^{34}$ = - 15.6 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.19 – 7.15 (m, 2H), 7.11 (t, *J* = 7.2 Hz, 1H), 6.94 – 6.89 (m, 2H), 5.27 (dd, *J* = 11.6, 5.2 Hz, 1H), 3.82 (d, *J* = 10.8 Hz, 1H), 3.68 – 3.62 (m, 2H), 3.49 – 3.44 (m, 1H), 3.08 – 3.04 (m, 2H), 2.86 – 2.81 (m, 1H), 2.34 –2.23 (m, 1H), 1.96 – 1.84 (m, 1H), 1.46 –1.36 (m, 1H), 1.06 (d, *J* = 6.8 Hz, 3H), 0.91 (t, *J* = 7.2 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 142.6, 135.8, 133.5, 126.8, 126.7, 126.6, 124.3, 122.0, 119.5, 118.3, 110.7, 109.4, 88.5, 66.1, 54.3, 47.8, 43.3, 38.1, 22.5, 18.6, 16.2, 10.9.

IR (KBr) *v* (cm^{-1})3421, 2961, 2920, 2847, 1622, 1546, 1461, 1369, 1340, 1166, 1084, 739, 701.

HRMS (ESI)*m/z* calcd for $C_{22}H_{25}N_3O_2S[M+H]^+$396.1740, found: 396.1739



(1*R*,2*R*,3*S*,4*S*,12b*R*)-4-ethyl-2-(furan-2-yl)-1-methyl-3-nitro-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6m)

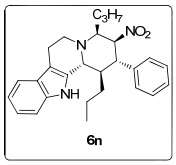
Purified by flash chromatography (PE:EtOAc = 20:1). 47% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 5.3min, τ_{major} = 9.5 min, 96% *ee*.

$[\alpha]_D^{34}$ = - 20.6(c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.50 (d, *J* = 7.6 Hz, 1H), 7.31 – 7.28 (m, 2H), 7.17 (t, *J* = 7.2 Hz, 1H), 7.10 (t, *J* = 6.8 Hz, 1H), 6.27– 6.26(m, 1H), 6.19 (d, *J* = 2.8 Hz, 1H), 5.37 (dd, *J* = 12.0, 5.6 Hz, 1H), 3.78 (d, *J* = 10.8 Hz, 1H), 3.65 (td, *J* = 11.2, 4.8 Hz, 1H), 3.51 – 3.44 (m, 2H), 3.07 – 2.93 (m, 2H), 2.84 – 2.79 (m, 1H), 2.45 – 2.39 (m, 1H), 1.92 – 1.81 (m, 1H), 1.42 – 1.30 (m, 1H), 1.03 (d, *J* = 6.4 Hz, 3H), 0.91 (t, *J* = 7.2 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 152.0, 141.8, 135.8, 133.5, 126.7, 121.9, 119.5, 118.3, 110.7, 110.2, 109.3, 108.5, 84.9, 65.8, 54.0, 47.7, 41.6, 34.7, 22.5, 18.4, 16.2, 10.9.

IR (KBr) *v* (cm^{-1})3446, 2968, 2930, 2901, 1628, 1540, 1461, 1369, 1340, 732.

HRMS (ESI)*m/z* calcd for $C_{23}H_{29}N_3O_3[M+H]^+$380.1969, found: 380.1944



(1*R*,2*S*,3*S*,4*S*,12b*R*)-1-butyl-3-nitro-2-phenyl-4-propyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6n)

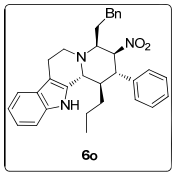
Purified by flash chromatography (PE:EtOAc = 20:1). 42% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 4.6min, τ_{major} = 9.4 min, 98% *ee*.

$[\alpha]_D^{34}$ = - 14.8(c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.31 – 7.20 (m, 6H), 7.16 (t, *J* = 7.2 Hz, 1H), 7.10 (t, *J* = 6.8 Hz, 1H), 5.37 (dd, *J* = 12.0, 5.6 Hz, 1H), 4.11 (d, *J* = 11.2 Hz, 1H), 3.72 (td, *J* = 11.2, 4.8 Hz, 1H), 3.62 – 3.54 (m, 2H), 3.10 – 3.06 (m, 1H), 3.01 – 2.93 (m, 1H), 2.86 – 2.81 (m, 1H), 2.34 – 2.29 (m, 1H), 1.98 – 1.88 (m, 1H), 1.52 – 1.12 (m, 9H), 0.88 (t, *J* = 7.2 Hz, 3H), 0.80 (t, *J* = 6.4 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 139.0, 135.9, 133.7, 128.7, 127.4, 126.7, 121.9, 119.5, 118.2, 110.6, 109.5, 87.7, 64.1, 50.8, 47.5, 44.4, 40.5, 30.4, 27.5, 22.6,19.6,17.8, 14.5, 13.7.

IR (KBr) *v* (cm^{-1})3417, 3063, 3037, 2955, 2927, 2863, 1550, 1546, 1530, 1496, 1464, 1366, 1347, 1166, 1084, 748, 701, 571, 485.

HRMS (ESI)*m/z* calcd for $C_{28}H_{35}N_3O_2[M+H]^+$432.2646, found: 432.2641



6o

(1*R*,2*S*,3*S*,4*S*,12b*R*)-1-butyl-3-nitro-4-phenethyl-2-phenyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6o)

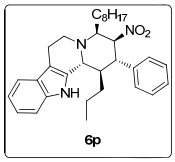
Purified by flash chromatography (PE:EtOAc = 20:1). 48% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 5.6min, τ_{major} = 11.5 min, 94% *ee*.

$[\alpha]_D^{34}$ = - 31.6 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (s, 1H), 7.53 (d, *J* = 7.6 Hz, 1H), 7.31 – 7.10 (m, 13H), 5.34 (dd, *J* = 12.0, 5.2 Hz, 1H), 4.12 (d, *J* = 11.2 Hz, 1H), 3.71 – 3.66 (m, 1H), 3.58 (t, *J* = 12.0 Hz, 1H), 3.51 – 3.46 (m, 1H), 3.08 – 2.96 (m, 2H), 2.87 – 2.82 (m, 1H), 2.79 – 2.73 (m, 1H), 2.62 – 2.55 (m, 1H), 2.34 – 2.24 (m, 2H), 1.70 – 1.61 (m, 1H), 1.41 – 1.38(m, 2H), 1.26 – 1.13 (m, 3H), 0.88 – 0.83 (m, 1H), 0.77 (t, *J* = 6.0 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 141.2, 138.8, 135.9, 133.6, 128.7, 128.5, 128.3, 127.4, 126.7, 125.9, 121.9, 119.5, 118.2, 110.7, 109.4, 87.5, 62.9, 50.7, 47.2, 44.5, 40.4, 31.8, 30.3, 27.1, 22.5, 17.8, 14.5.

IR (KBr) *v* (cm^{-1})3424, 3060, 3028, 2952, 2866, 2837, 1603, 1540, 1492, 1454, 1366, 1347, 1163, 1078, 907, 742, 704, 568.

HRMS (ESI)*m/z* calcd for $C_{33}H_{39}N_3O_2[M+H]^+$494.2802, found: 494.2779



6p

(1*R*,2*S*,3*S*,4*S*,12b*R*)-1-butyl-3-nitro-4-octyl-2-phenyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6p)

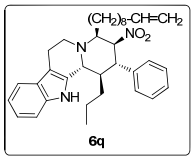
Purified by flash chromatography (PE:EtOAc = 20:1). 37% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 4.0min, τ_{major} = 6.8 min, 91% *ee*.

$[\alpha]_D^{34}$ = - 17.6 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.30 – 7.21 (m, 7H), 7.16 (t, *J* = 7.2 Hz, 1H), 7.10 (t, *J* = 7.2 Hz, 1H), 5.37 (dd, *J* = 12.0, 5.6 Hz, 1H), 4.10 (d, *J* = 10.8 Hz, 1H), 3.71 (td, *J* = 10.8, 4.4 Hz, 1H), 3.61 – 3.52 (m, 2H), 3.09 – 3.05 (m, 1H), 3.01 –2.93 (m, 1H), 2.85 – 2.81 (m, 1H), 2.30 (t, *J* = 11.6 Hz, 1H), 1.97 – 1.88 (m, 1H), 1.42 – 1.15 (m, 18H), 0.85 (t, *J* = 6.8 Hz, 3H), 0.79 (t, *J* = 6.4 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 139.0, 135.9, 133.8, 128.7, 127.4, 126.7, 121.9, 119.5, 118.2, 110.6, 109.4, 87.7, 64.4, 50.7, 47.5, 44.4, 40.5, 31.8, 30.4, 29.4, 29.3, 29.2, 26.4, 25.4, 22.6, 17.8, 14.5, 14.0.

IR (KBr) *v* (cm^{-1})3427, 3060, 3025, 2955, 2927, 2847, 1540, 1458, 1366, 1350, 1163, 742, 698.

HRMS (ESI)*m/z* calcd for $C_{33}H_{47}N_3O_2[M+H]^+$502.3428, found: 502.3402



6q

(1*R*,2*S*,3*S*,4*S*,12b*R*)-1-butyl-4-(dec-9-en-1-yl)-3-nitro-2-phenyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (6q)

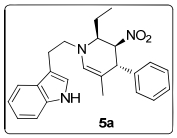
Purified by flash chromatography (PE:EtOAc = 20:1). 48% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 80/20);flow rate 1 mL/min; λ = 254 nm; τ_{minor} = 4.0min, τ_{major} = 7.0 min, 93% *ee*.

$[\alpha]_D^{34}$ = -15.2 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.30 – 7.19 (m, 6H), 7.16 (t, *J* = 7.2 Hz, 1H), 7.10 (t, *J* = 6.8 Hz, 1H), 5.83 – 5.73 (m, 1H), 5.37 (dd, *J* = 12.0, 5.6 Hz, 1H), 5.00 – 4.89 (m, 2H), 4.10 (d, *J* = 10.8 Hz, 1H), 3.71 (td, *J* = 10.8, 4.4 Hz, 1H), 3.61 – 3.52 (m, 2H), 3.09 – 3.05 (m, 1H), 3.01 – 2.93 (m, 1H), 2.85 – 2.81 (m, 1H), 2.31 (t, *J* = 11.6 Hz, 1H), 2.00 (q, *J* = 6.8Hz, 2H), 1.94 – 1.88 (m, 1H), 1.42 – 1.11 (m, 18H), 0.79 (t, *J* = 6.4 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 139.2, 139.0, 135.9, 133.8, 128.7, 127.4, 126.7, 121.9, 119.5, 118.2, 114.1, 110.6, 109.4, 87.7, 64.4, 50.8, 47.5, 44.4, 40.5, 33.7, 30.4, 29.4, 29.2, 29.0, 28.8, 26.3, 25.4, 22.6, 17.8, 14.5.

IR (KBr) *v* (cm^{-1})3421, 3059, 3028, 2923, 2854, 1639, 1549, 1534, 1458, 1366, 1340, 1166, 1087, 907, 755, 701, 571, 485.

HRMS (ESI)*m/z* calcd for $C_{35}H_{49}N_3O_2[M+H]^+$528.3585, found: 528.3588



5a

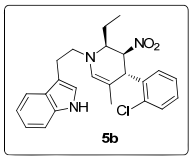
3-(2-((2*S*,3*S*,4*R*)-2-ethyl-5-methyl-3-nitro-4-phenyl-3,4-dihydropyridin-1(2*H*)-yl)ethyl)-1*H*-indole (5a)

Purified by flash chromatography (PE:EtOAc = 20:1). 75% yield, white solid. The *ee* was determined by chiralHPLC using a Phenomenex Lux 5u Amylose-2 column (hexane/*i*-PrOH = 98/2);flow rate 1 mL/min; λ = 214 nm; τ_{minor} = 27.2min, τ_{major} = 29.0 min, 97% *ee*.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.26 – 7.18 (m, 4H), 7.17 – 7.13 (m, 1H), 7.08 (d, *J* = 7.2 Hz, 2H), 7.04 (d, *J* = 2.0 Hz, 1H), 5.87 (s, 1H), 4.65 (dd, *J* = 10.4, 4.0 Hz, 1H), 3.85 (d, *J* = 10.8 Hz, 1H), 3.50 – 3.31 (m, 3H), 3.04 (t, *J* = 7.2 Hz, 2H), 1.62 – 1.52 (m, 1H), 1.32 – 1.26 (m, 4H), 0.93 (t, *J* = 7.6 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 136.3, 128.7, 128.5, 127.3, 127.1, 122.2, 121.7, 119.4, 118.6, 113.2, 111.3, 105.1, 90. 8, 60.7, 55.5, 43.7, 25.2, 20.2, 17.8, 11.0.

IR (KBr) *v* (cm^{-1})3422, 2958, 2920, 1658, 1541, 1455, 1360, 1109, 746, 702.

HRMS (ESI)*m/z* calcd for $C_{25}H_{31}N_3O_2[M+Na]^+$412.1995, found: 412.1955

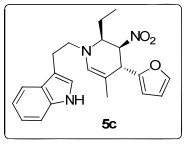


3-(2-((2*S*,3*S*,4*S*)-4-(2-chlorophenyl)-2-ethyl-5-methyl-3-nitro-3,4-dihydropyridin-1(2*H*)-yl)ethyl)-1*H*-indole (5b)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.24 – 7.14 (m, 4H), 7.06 (d, *J* = 1.6 Hz, 1H), 6.94 (d, *J* = 7.6 Hz, 2H), 5.86 (s, 1H), 4.49 (dd, *J* = 10.8, 4.4 Hz, 1H), 3.81 (d, *J* = 10.8 Hz, 1H), 3.50 – 3.44 (m, 2H), 3.40 – 3.33 (m, 1H), 3.04 (t, *J* = 6.8 Hz, 2H), 1.56 – 1.51 (m, 1H), 1.29 – 1.24 (s, 4H), 0.93 (t, *J* = 7.2 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 138.4, 136.3, 132.8, 130.0, 129.0, 128.7, 127.3, 122.2, 121.7, 119.5, 118.6, 113.1, 111.3, 103.9, 90.6, 60.8, 55.5, 43.0, 25.3, 20.3, 17.7, 11.0.

IR (KBr) *v* (cm^{-1})3422, 2961, 2920, 2872, 1658, 1541, 1490, 1458, 1363, 1232, 1096, 1010, 826, 743.

HRMS (ESI)*m/z* calcd for $C_{24}H_{26}ClN_3O_2$[M+Na]$^+$446.1606, found: 446.1569

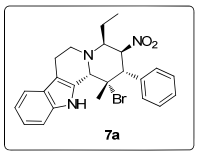


3-(2-((2*S*,3*S*,4*R*)-2-ethyl-4-(furan-2-yl)-5-methyl-3-nitro-3,4-dihydropyridin-1(2*H*)-yl)ethyl)-1*H*-indole (5c)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.38 – 7.33 (m, 2H), 7.24 – 7.19 (m, 1H), 7.14 (t, *J* = 7.2 Hz, 1H), 7.05 (s, 1H), 6.27 (s, 1H), 6.19 (d, *J* = 2.4 Hz, 1H), 5.84 (s, 1H), 4.89 (dd, *J* = 10.4, 4.0 Hz, 1H), 4.05 (d, *J* = 10.4 Hz, 1H), 3.48 – 3.46 (m, 1H), 3.38 – 3.32 (m, 2H), 3.04 – 2.99 (m, 2H), 1.62 – 1.49 (m, 2H), 1.43 (s, 3H), 0.92 (t, *J* = 7.2 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 152.3, 142.0, 136.3, 128.7, 127.2, 122.1, 121.8, 119.4, 118.6, 113.2, 111.3, 110.2, 108.8, 102.9, 86.3, 60.3, 55.4, 37.7, 25.2, 20.1, 17.7, 10.9.

IR (KBr) *v* (cm^{-1})3422, 2964, 2920, 2875, 1658, 1544, 1458, 1360, 1229, 1144, 1102, 1010, 743.

HRMS (ESI)*m/z* calcd for $C_{23}H_{29}N_3O_3$[M+Na]$^+$402.1788, found: 402.1770



(1*S*,2*S*,3*S*,4*S*,12b*S*)-1-bromo-4-ethyl-1-methyl-3-nitro-2-phenyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (7a)

Purified by FC (PE:EtOAc = 5:1). 54% yield, white solid, a single isomer.

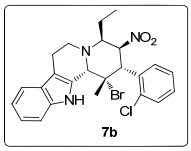
$[\alpha]_D^{26}$ = +28.0 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.55 (d, *J* = 7.8 Hz, 2H), 7.34 – 7.32 (m, 5H), 7.19 (t, *J* = 7.0 Hz, 1H), 7.13 (t, *J* = 7.0 Hz, 1H), 5.75 (dd, *J* = 12.0, 5.4 Hz, 1H), 4.00 (s, 1H), 3.85 – 3.79(m,

1H), 3.75 – 3.75 (m, 1H), 3.49 (d, *J* = 12.1 Hz, 1H), 3.15 – 3.09 (m, 1H), 3.03 – 2.97 (m, 1H), 2.91 – 2.85 (dt, *J* = 10.6, 5.1 Hz, 1H), 1.91 (s, 3H), 1.62 – 1.55 (m, 2H), 0.92 (t, *J* = 7.4 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 136.1, 135.0, 132.9, 130.6, 128.2, 126.4, 122.4, 119.7, 118.5, 113.2, 110.8, 85.9, 72.3, 65.5, 60.2, 52.0,50.0, 28.5, 21.8, 17.7, 12.6.

IR (KBr) *v* (cm^{-1})3403, 2951, 2891, 1719, 1639, 1553, 1452, 1375, 1340, 1188, 1064, 959, 927, 848, 759, 708.

HRMS (ESI)*m/z* calcd for $C_{24}H_{26}BrN_3O_2[M+H]^+$468.1281, found: 468.1280



7b

(1*S*,2*S*,3*S*,4*S*,12b*S*)-1-bromo-2-(2-chlorophenyl)-4-ethyl-1-methyl-3-nitro-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (7b)

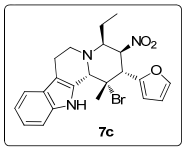
Purified by FC (PE:EtOAc = 5:1). 58% yield, white solid, a single isomer.

$[\alpha]_D^{24}$ = + 8.2 (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.47 (d, *J* = 7.6 Hz, 2H), 7.27 – 7.14 (m, 5H), 7.11 (t, *J* = 7.2 Hz, 1H), 7.05 (t, *J* = 7.2 Hz, 2H), 5.60 (dd, *J* = 12.0, 5.2 Hz, 1H), 3.89 (s, 1H), 3.73 – 3.68 (m, 1H), 3.65 – 3.61 (m, 1H), 3.38 (d, *J* = 12.0 Hz, 1H), 3.05 – 2.99 (m, 1H), 2.94 – 2.88 (m, 1H), 2.82 – 2.76 (m, 1H), 1.80 (s, 3H), 1.49 – 1.43 (m, 1H), 0.83 (t, *J* = 7.6 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 136.2, 134.2, 133.6, 130.4, 128.2, 126.4, 122.4, 119.7, 118.5, 113.2, 110.9, 85.8, 72.2, 65.4, 60.2, 51.5, 50.0, 28.4, 21.8, 17.7, 12.5.

IR (KBr) *v* (cm^{-1})3431, 2967, 2929, 1620, 1544, 1490, 1455, 1369, 1331, 1140, 1090, 1010, 819, 737.

HRMS (ESI)*m/z* calcd for $C_{24}H_{25}BrClN_3O_2$ $[M+H]^+$502.0891, found: 502.0889



7c

(1*S*,2*R*,3*S*,4*S*,12b*S*)-1-bromo-4-ethyl-2-(furan-2-yl)-1-methyl-3-nitro-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine (7c)

Purified by FC (PE:EtOAc = 5:1). 56% yield, white solid, a single isomer.

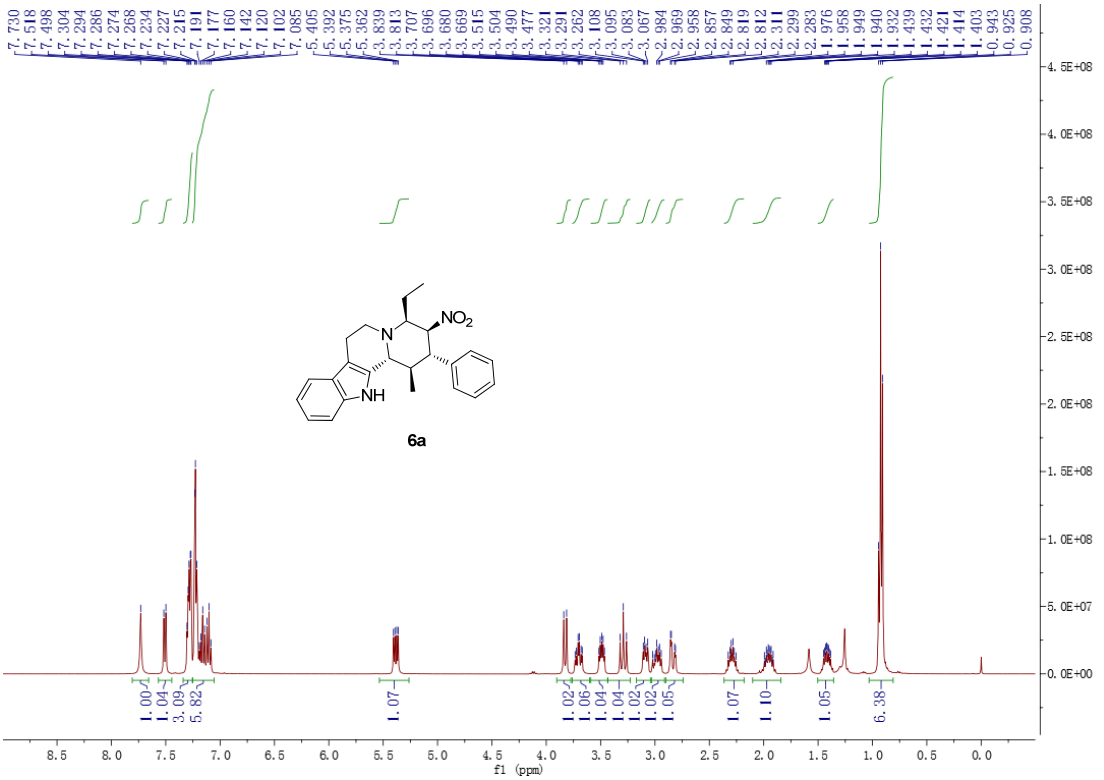
$[\alpha]_D^{24}$ = + 11.0 (c = 1.0, $CHCl_3$).

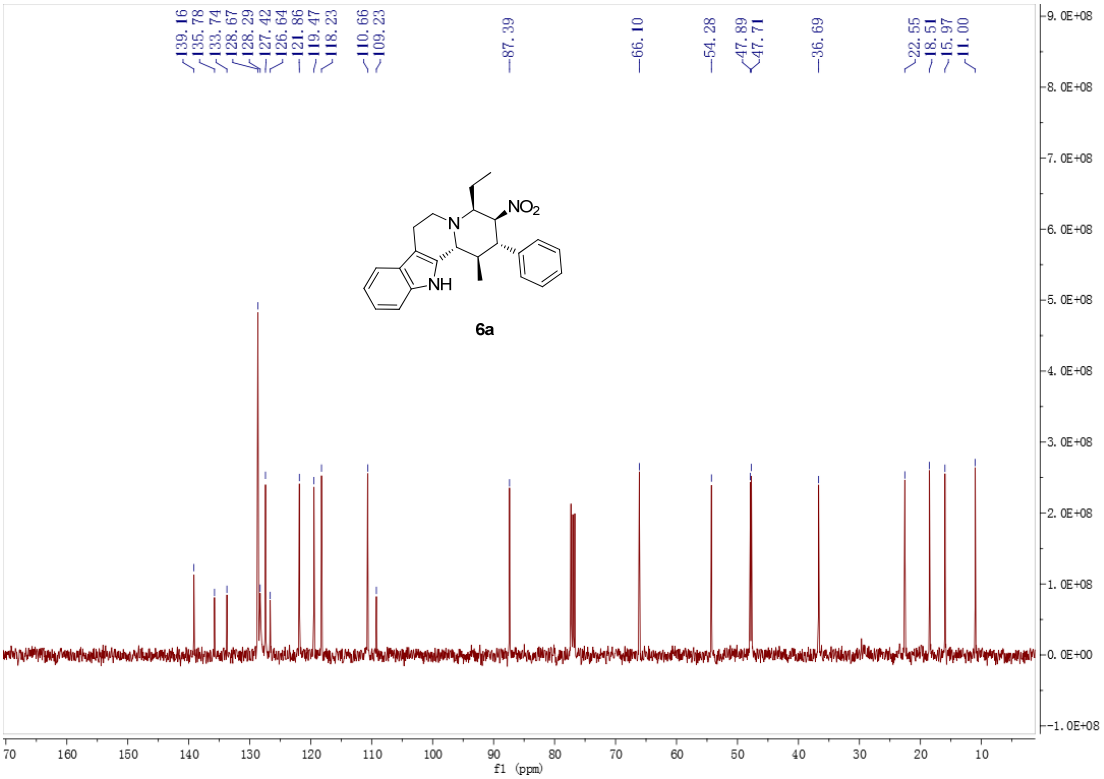
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.47 (d, *J* = 7.6 Hz, 1H), 7.31 – 7.26 (m, 2H), 7.12 (t, *J* = 7.2 Hz, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 6.28 (s, 2H), 5.50 (dd, *J* = 12.0, 5.6 Hz, 1H), 3.87 (s, 1H), 3.74 (dt, *J* = 10.8, 5.2 Hz, 1H), 3.68 – 3.60 (m, 2H), 3.05 – 2.99 (m, 1H), 2.93 – 2.89 (m, 1H), 2.82 – 2.76 (m, 1H), 1.93 (s, 3H), 1.80 – 1.73 (m, 1H), 1.45 – 1.38 (m, 1H), 0.84 (t, *J* = 7.6 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 150.3, 142.0, 136.1, 130.5, 126.4, 122.4, 119.7, 118.5, 113.0, 110.9, 110.5, 109.1, 85.0, 71.6, 65.2, 59.5, 49.8, 46.7, 28.9, 21.8, 17.6, 12.4.

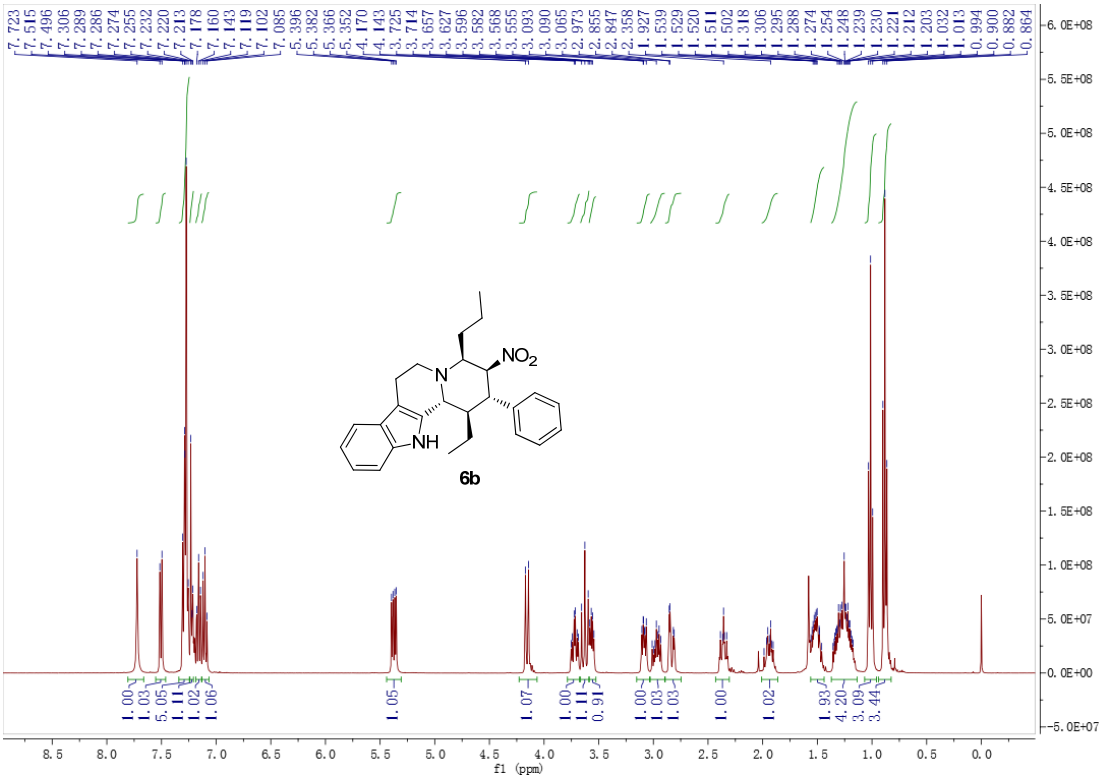
IR (KBr) *v* (cm^{-1})3425, 2967, 2923, 1617, 1547, 1455, 1369, 1334, 1144, 1010, 737.

HRMS (ESI)*m/z* calcd for $C_{22}H_{24}BrN_3O_3$ $[M+H]^+$458.1074, found: 458.1040

8. ^{1}H and ^{13}C NMR Spectra



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6b
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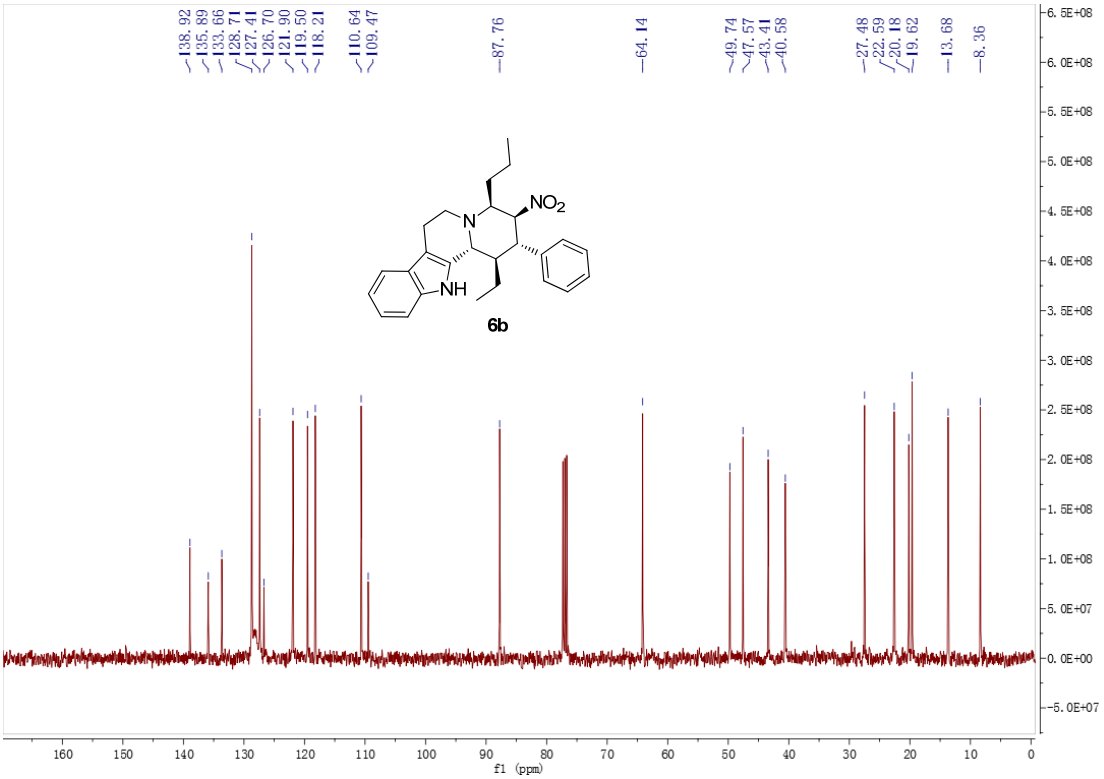
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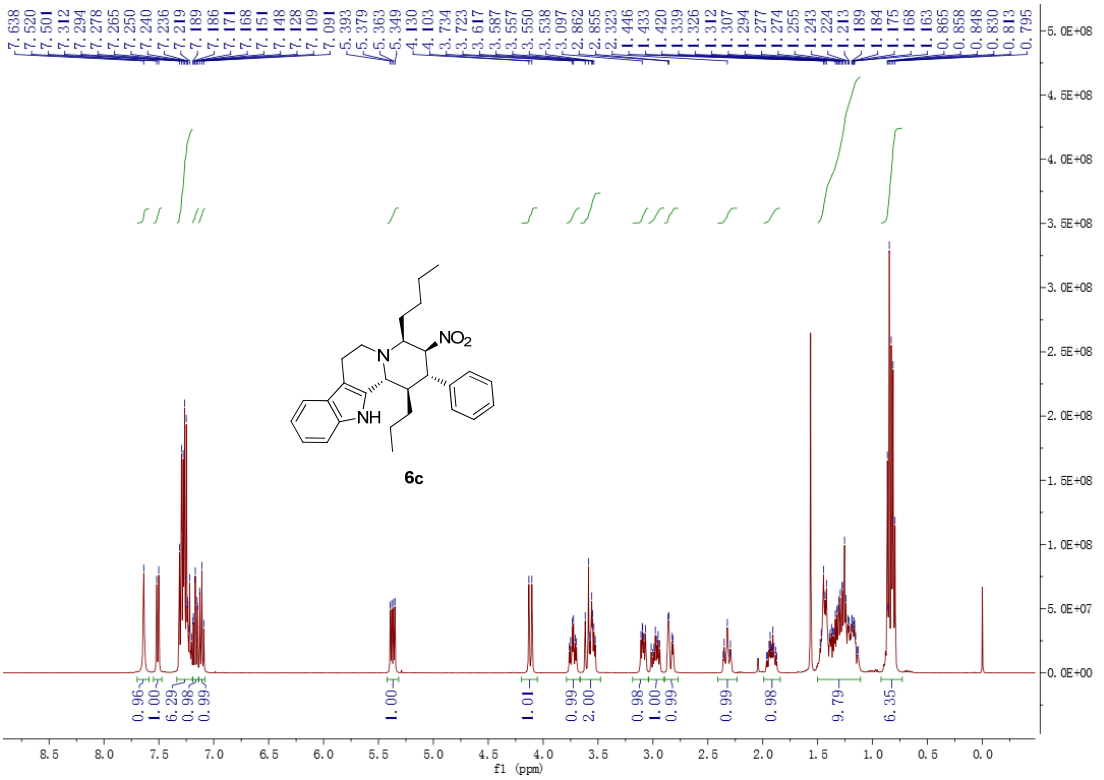
N
NH
6b
f1 (ppm)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NH
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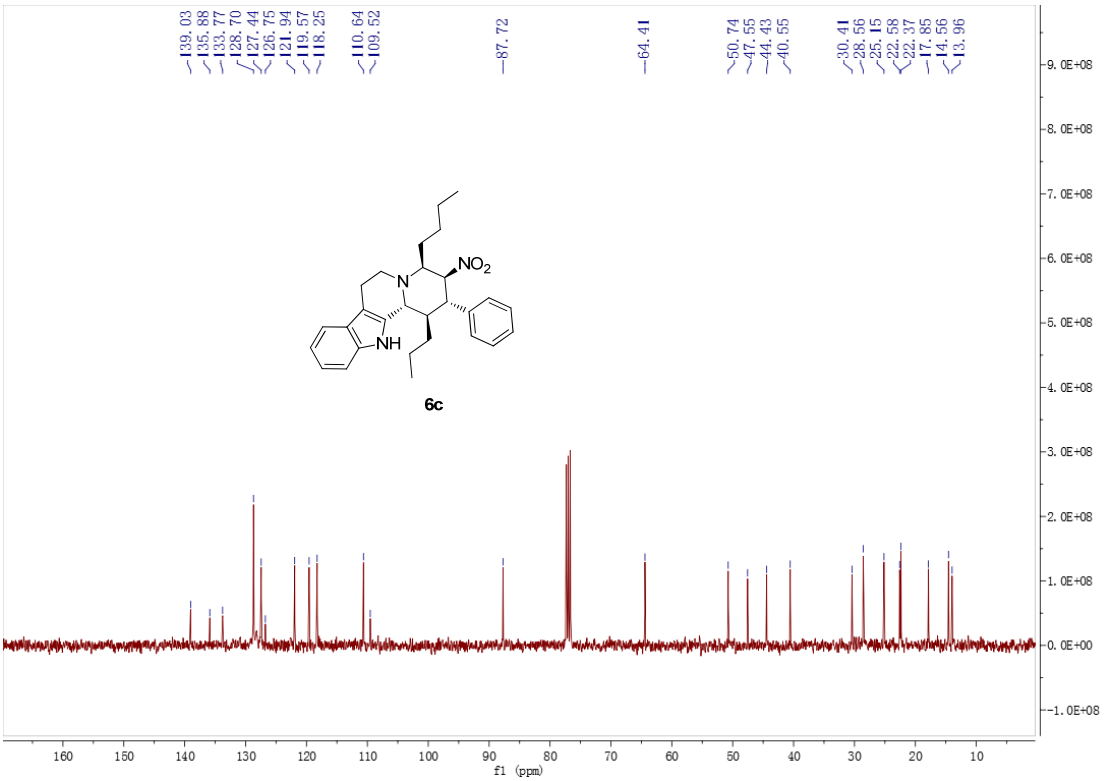
f1 (ppm)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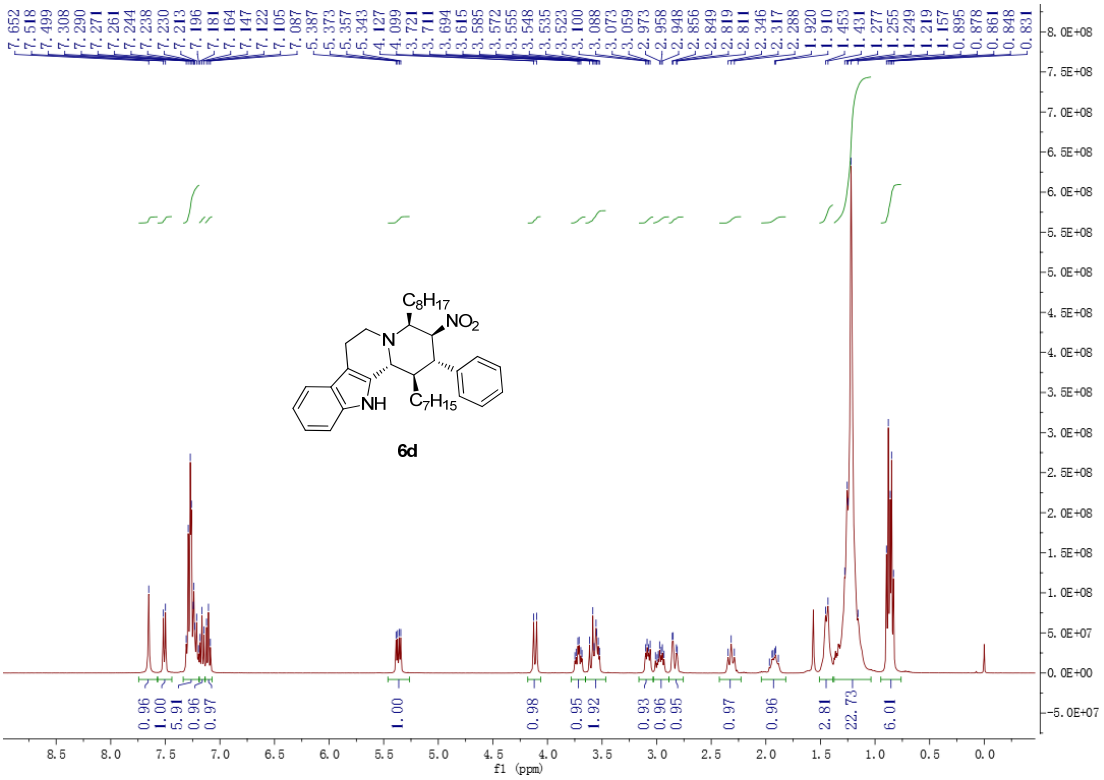
NO2
N
NH
6c
f1 (ppm)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C8H17
NO2
N
NH
C7H15
6d
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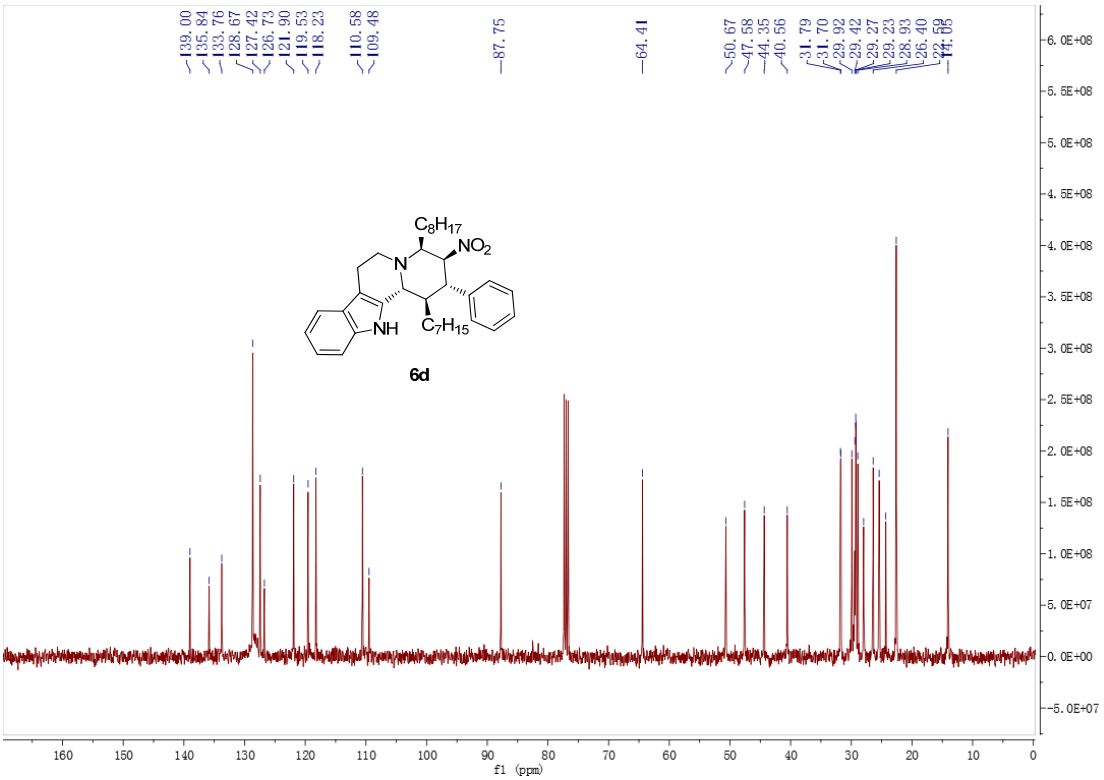
f1 (ppm)


139.00 135.84 133.76 128.67 127.42 126.73 121.90 119.53 118.23 110.58 109.48 87.75 64.41 50.67 47.58 44.35 40.56 31.79 31.70 29.92 29.42 29.27 29.23 28.93 26.40 22.59 14.05
C8H17
NO2
N
NH
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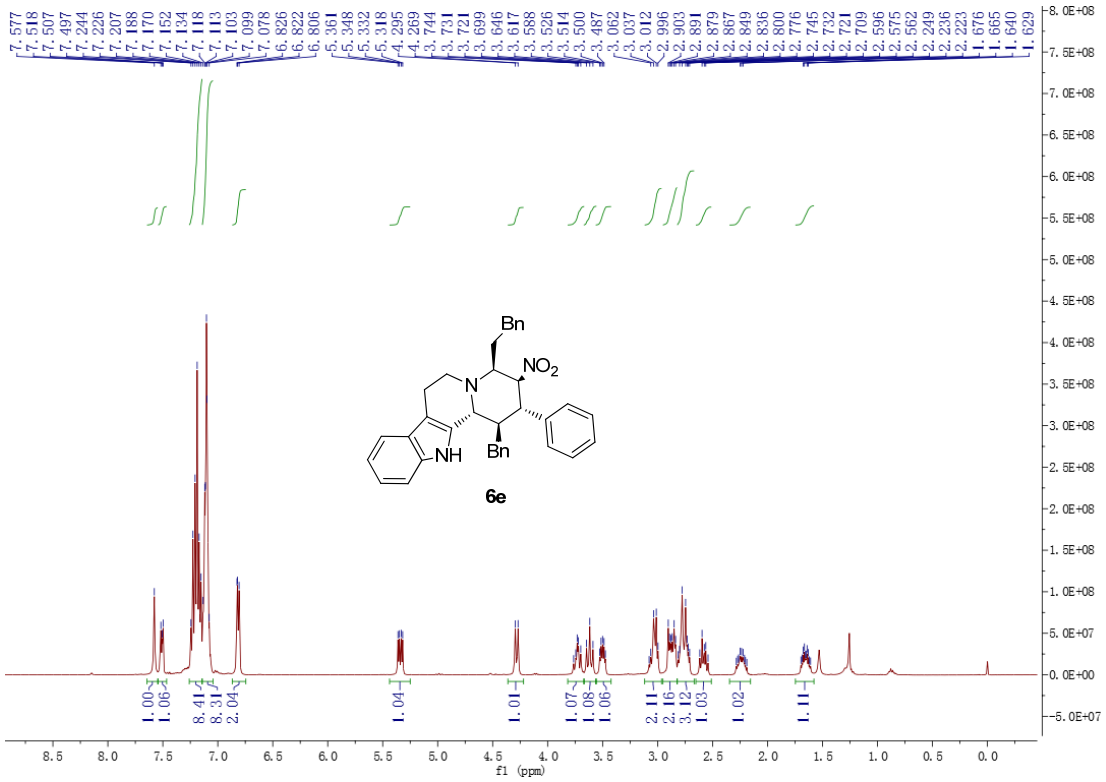
6d
f1 (ppm)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NO2
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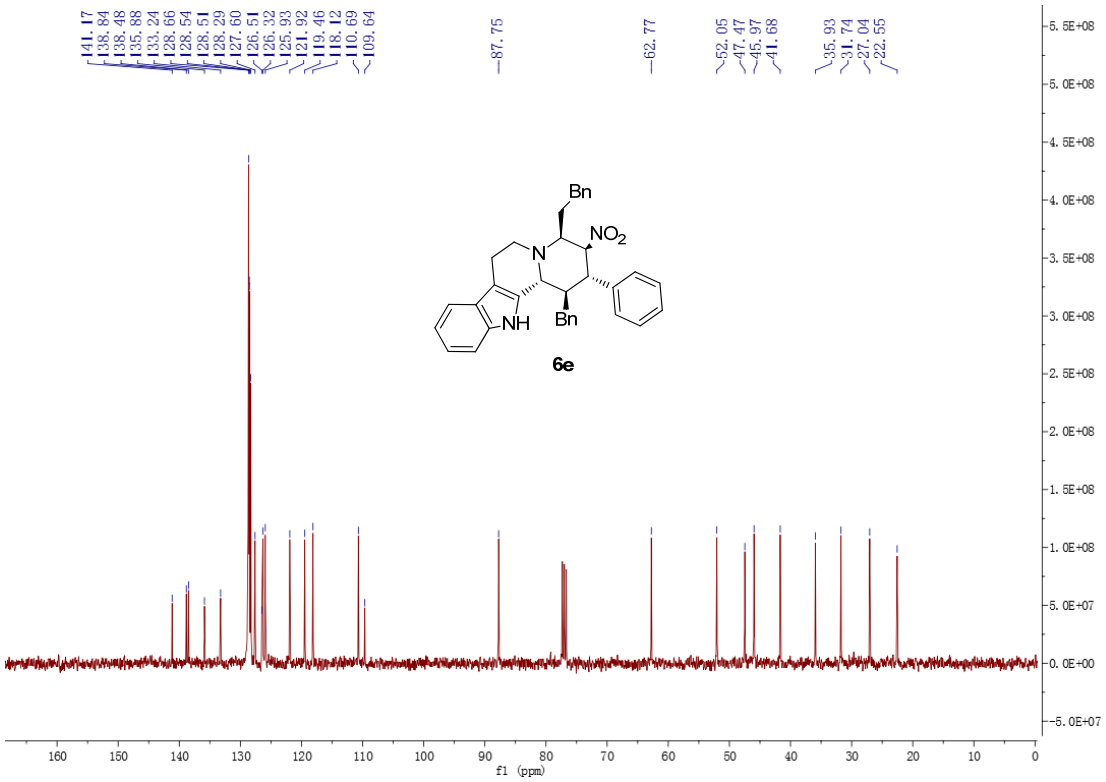
f1 (ppm)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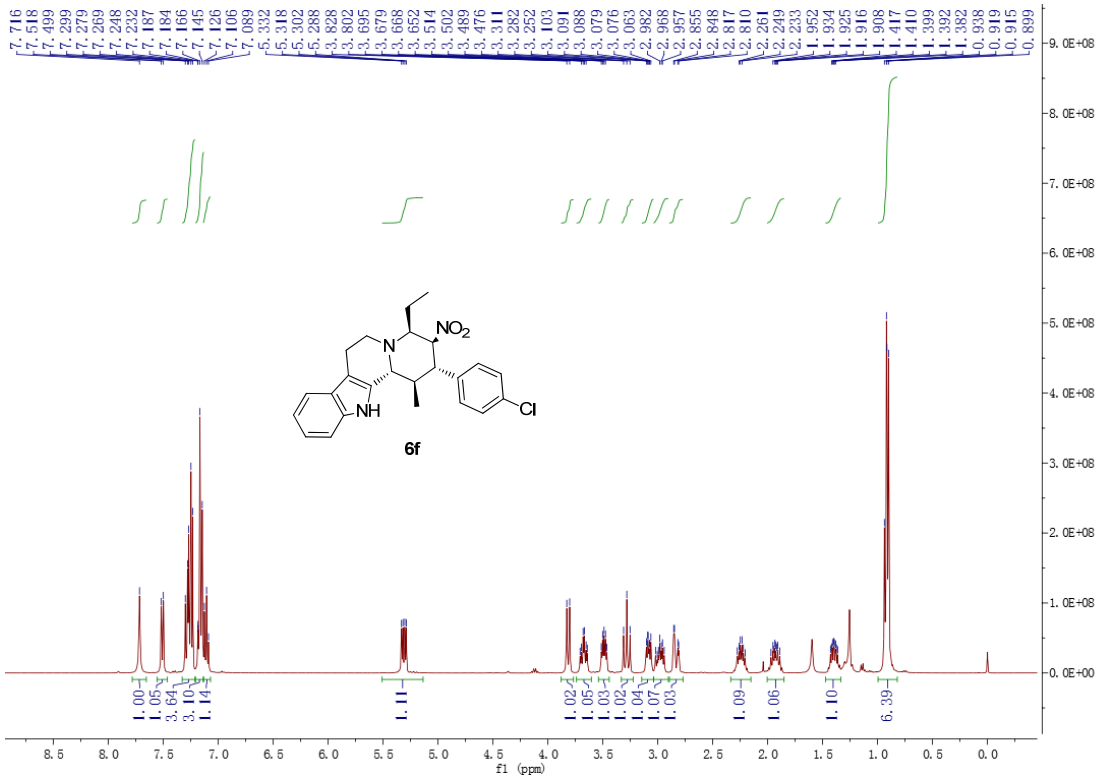
f1 (ppm)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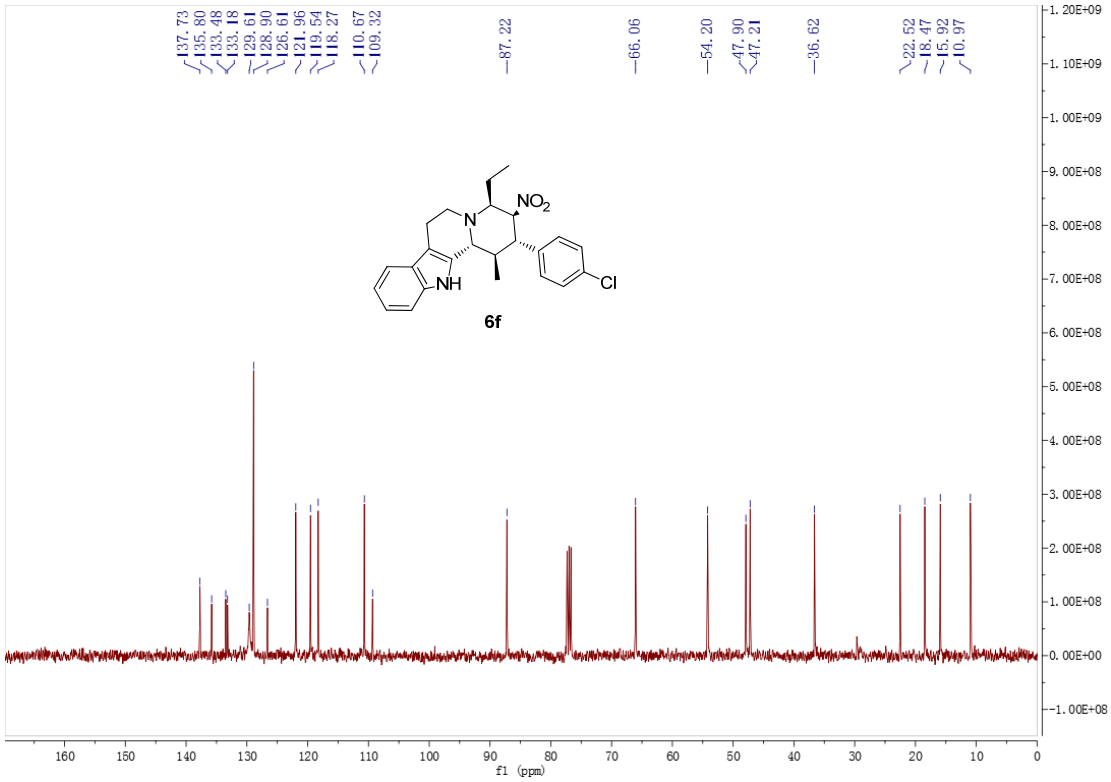
Cl
6f
f1 (ppm)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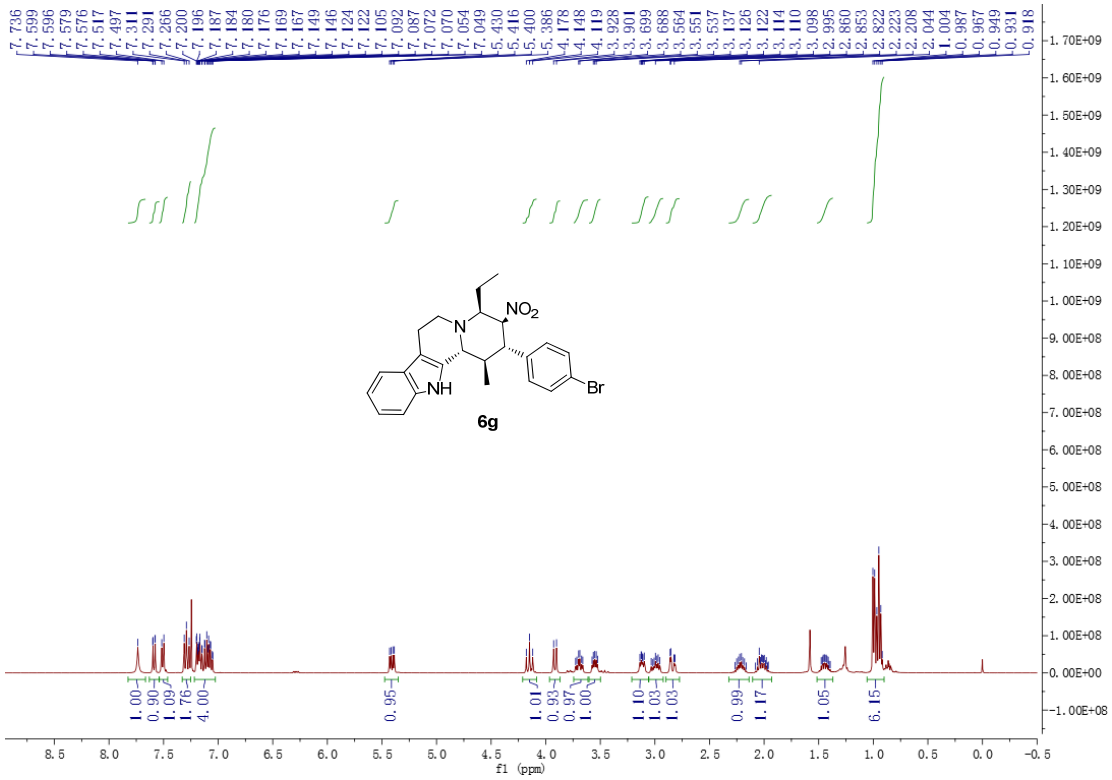
6f
f1 (ppm)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7.196
7.187
7.184
7.180
7.176
7.169
7.167
7.149
7.146
7.124
7.122
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7.092
7.087
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7.049
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5.400
5.386
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4.119
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3.901
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3.688
3.564
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3.537
3.137
3.126
3.122
3.114
3.110
3.098
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2.860
2.853
2.822
2.223
2.208
2.044
1.004
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0.967
0.949
0.931
0.918
NO2
N
NH
Br
6g
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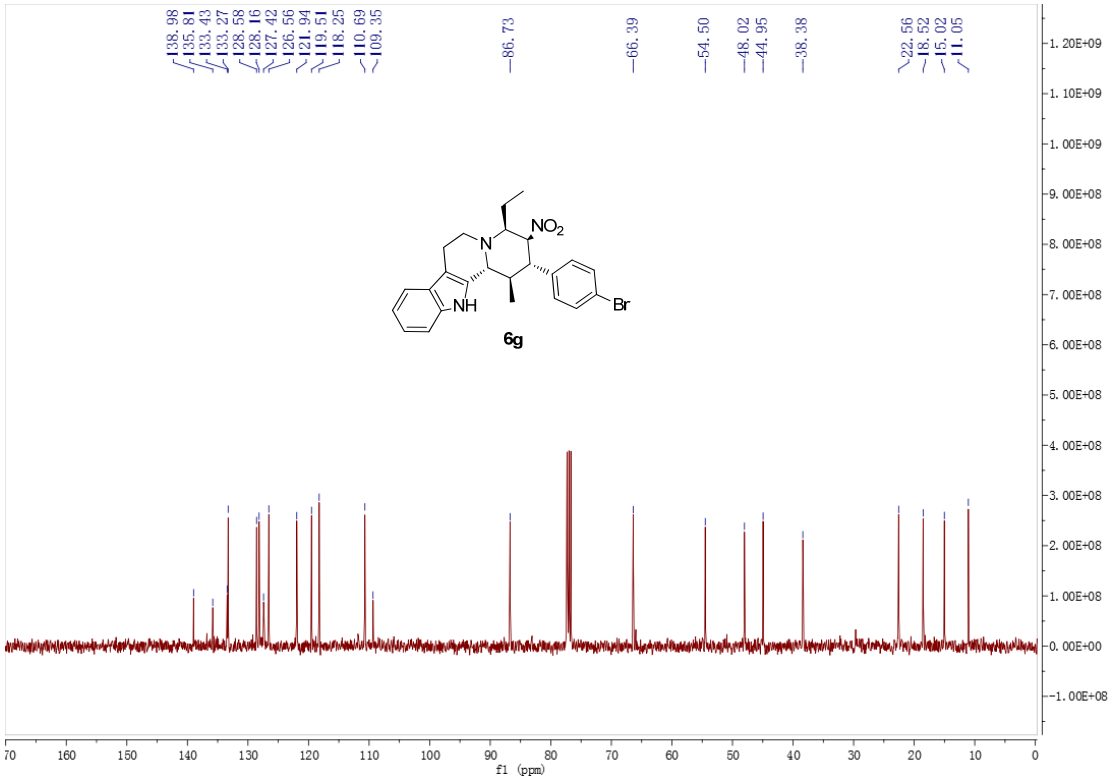
0.90
1.09
1.76
4.00
0.95
1.01
0.93
0.97
1.00
1.10
1.03
1.03
0.99
1.17
1.05
6.15
f1 (ppm)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118.25
110.69
109.35
86.73
66.39
54.50
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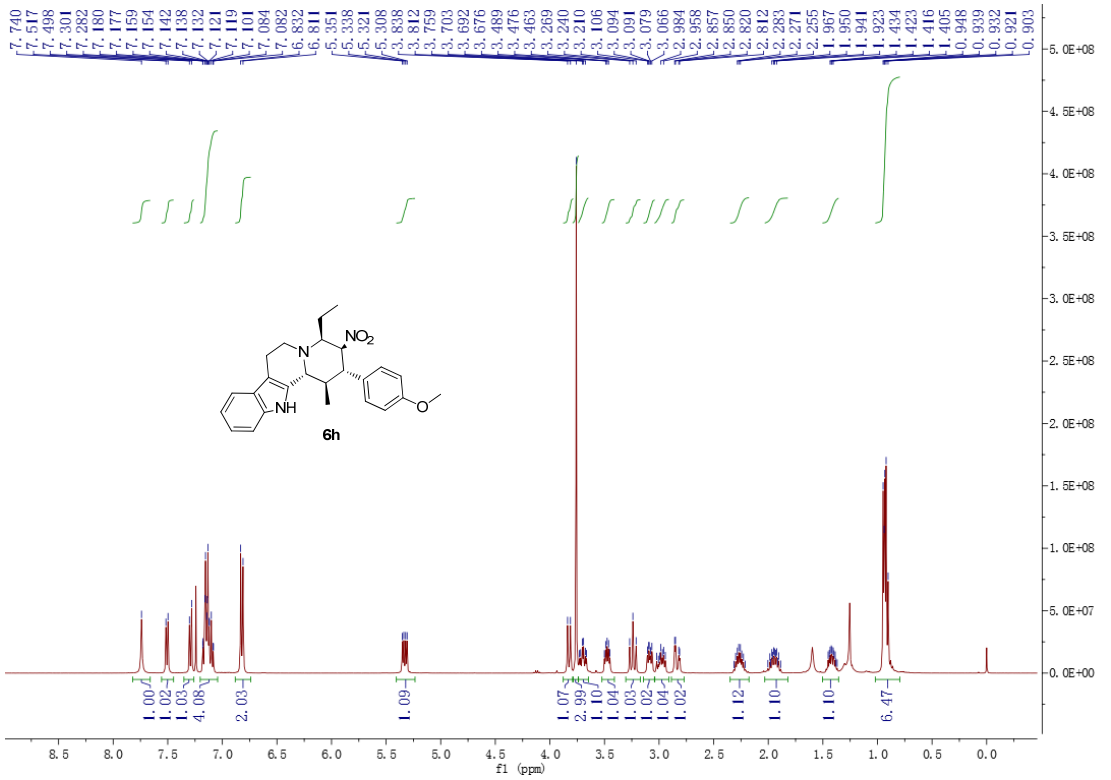
22.56
18.52
15.02
11.05
NO2
N
NH
Br
6g
f1 (ppm)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7.132
7.121
7.119
7.101
7.084
7.082
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6.811
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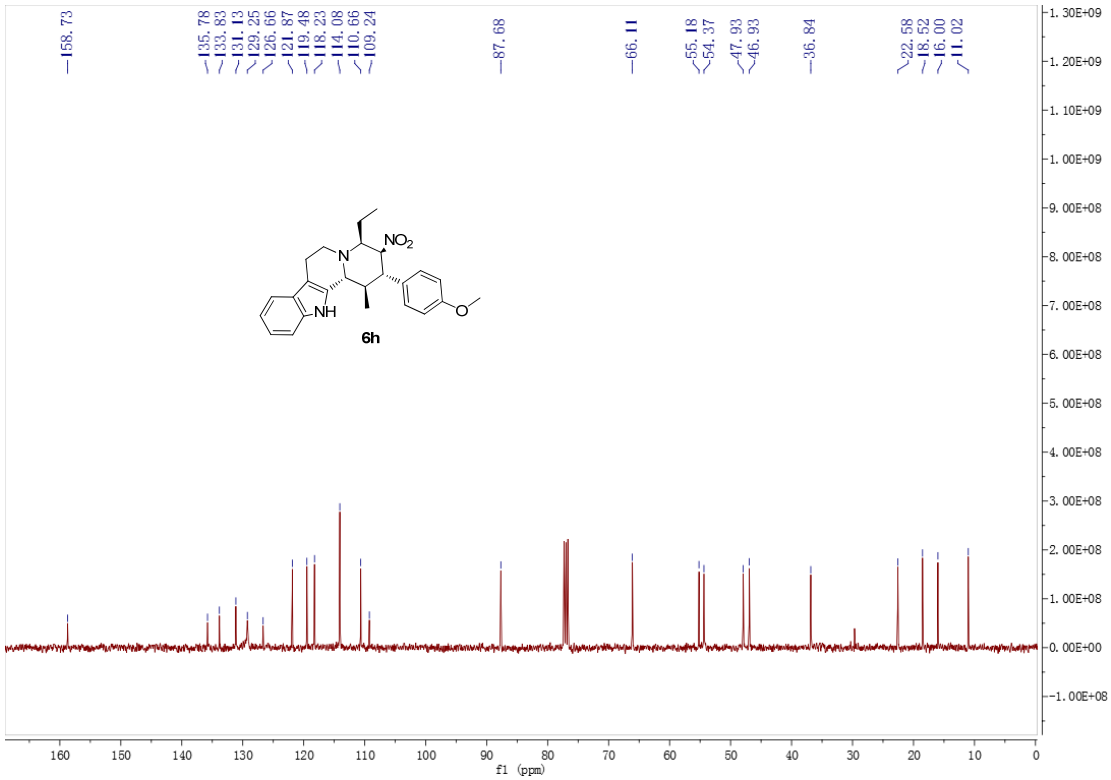
5.338
5.321
5.308
3.838
3.812
3.759
3.703
3.692
3.676
3.489
3.476
3.463
3.269
3.240
3.210
3.106
3.094
3.091
3.079
3.066
2.984
2.958
2.857
2.850
2.820
2.812
2.283
2.271
2.255
1.967
1.950
1.941
1.923
1.434
1.423
1.416
1.405
0.948
0.939
0.932
0.921
0.903
NO2
N
NH
O
6h
1.00
1.02
1.03
4.08
2.03
1.09
1.07
2.99
1.10
1.04
1.03
1.02
1.04
1.02
1.12
1.10
1.10
6.47
f1 (ppm)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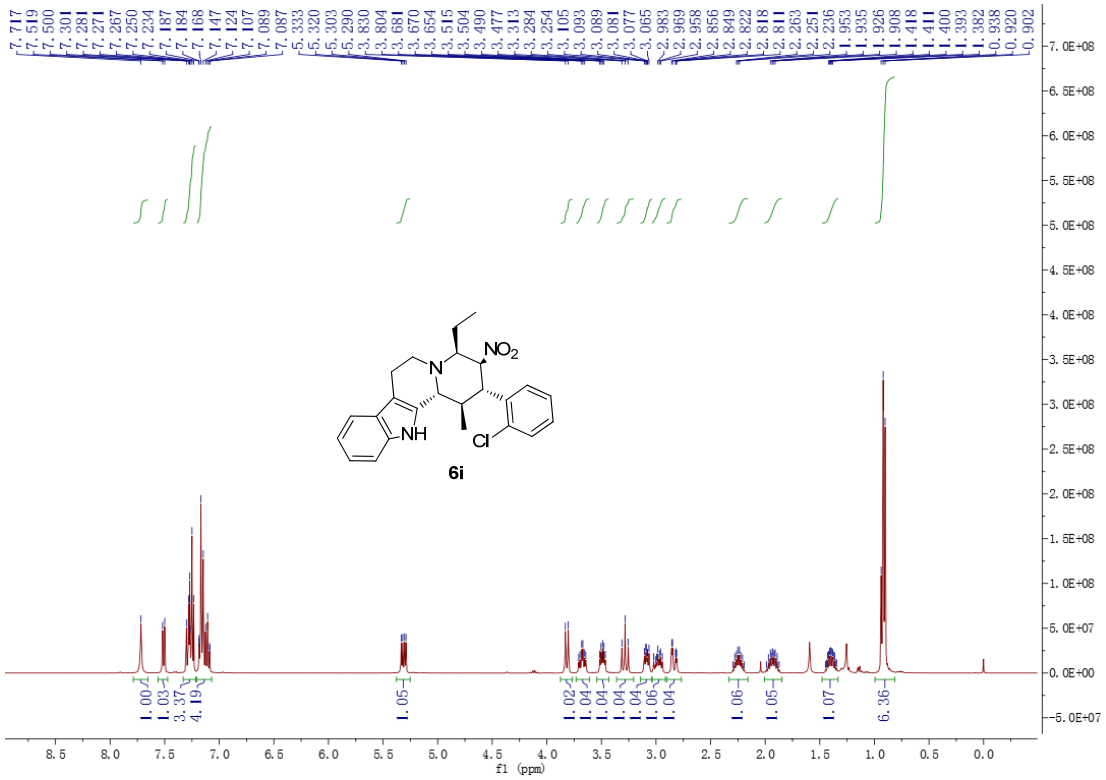
129.25
126.66
121.87
119.48
118.23
114.08
110.66
109.24
87.68
66.11
55.18
54.37
47.93
46.93
36.84
22.58
18.52
16.00
11.02
NO2
N
NH
O
6h
f1 (ppm)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7.271
7.267
7.250
7.234
7.187
7.184
7.168
7.147
7.124
7.107
7.089
7.087
5.333
5.320
5.303
5.290
3.830
3.804
3.681
3.670
3.654
3.515
3.504
3.490
3.477
3.313
3.284
3.254
3.105
3.093
3.089
3.081
3.077
3.065
2.983
2.969
2.958
2.856
2.849
2.822
2.818
2.811
2.263
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2.236
1.953
1.935
1.926
1.908
1.418
1.411
1.400
1.393
1.382
0.938
0.920
0.902
NO2
N
NH
Cl
6i
1.00
1.03
3.37
4.19
1.05
1.02
1.04
1.04
1.04
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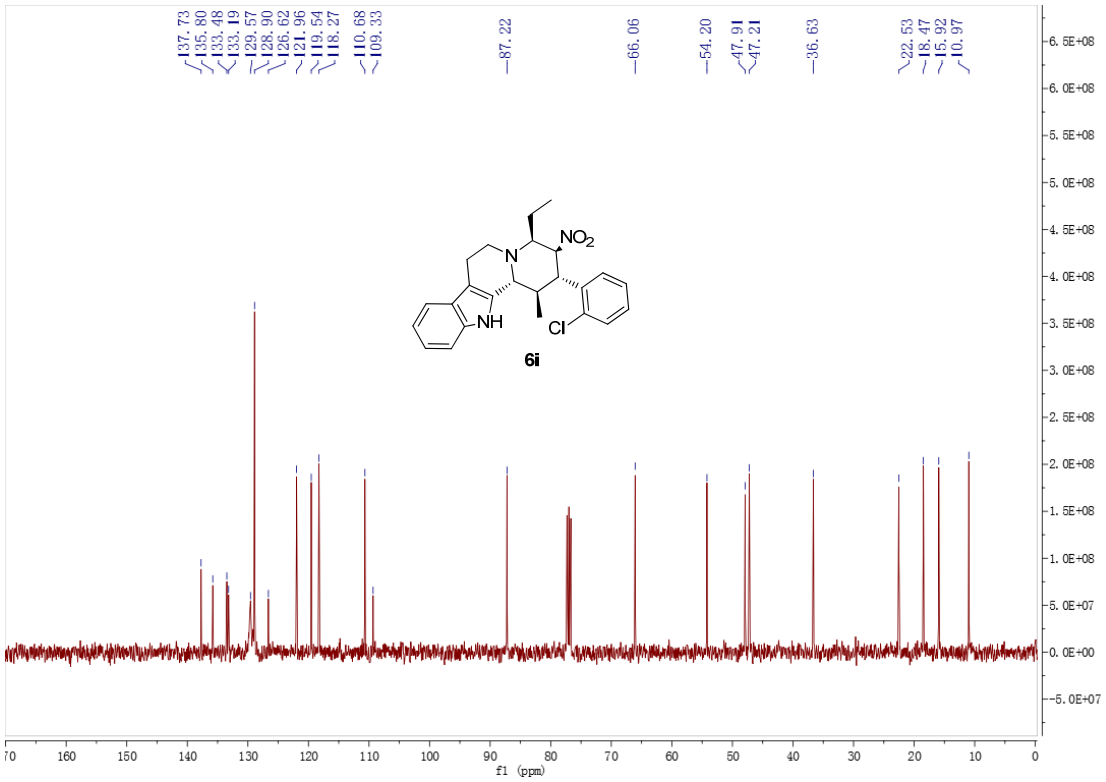
1.06
1.05
1.07
6.36
f1 (ppm)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128.90
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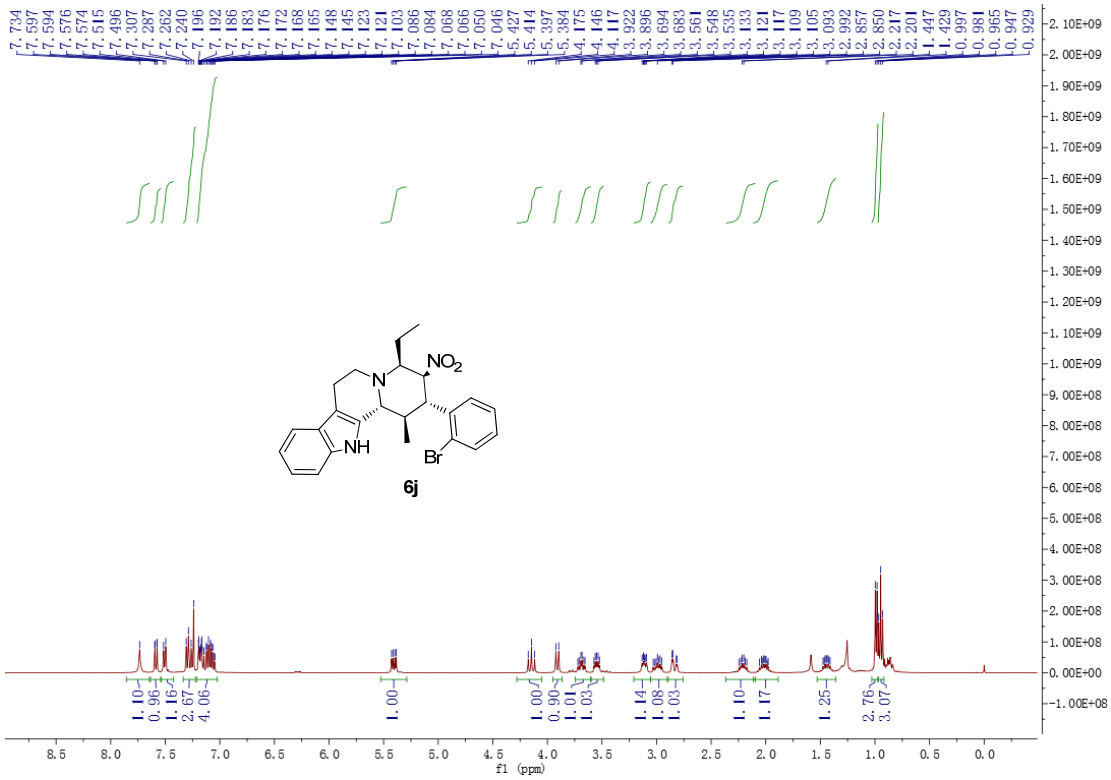
121.96
119.54
118.27
110.68
109.33
87.22
66.06
54.20
47.91
47.21
36.63
22.53
18.47
15.92
10.97
NO2
N
NH
Cl
6i
f1 (ppm)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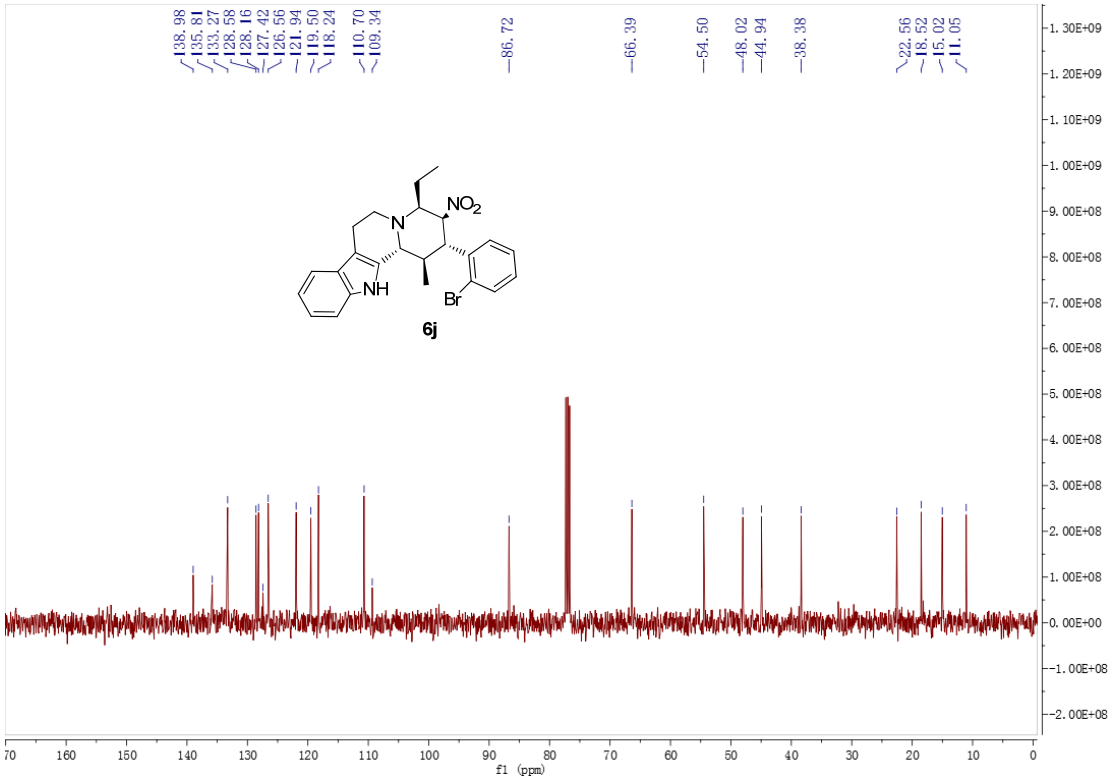
NH
Br
6j
f1 (ppm)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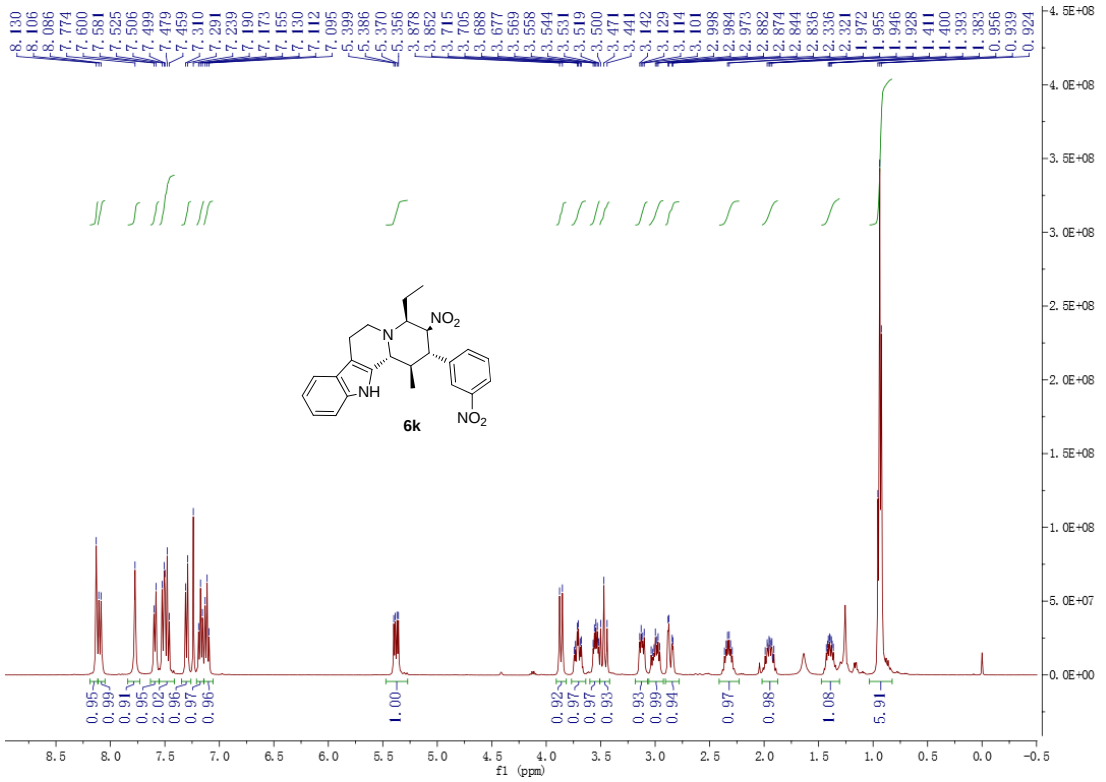
NH
Br
6j
f1 (ppm)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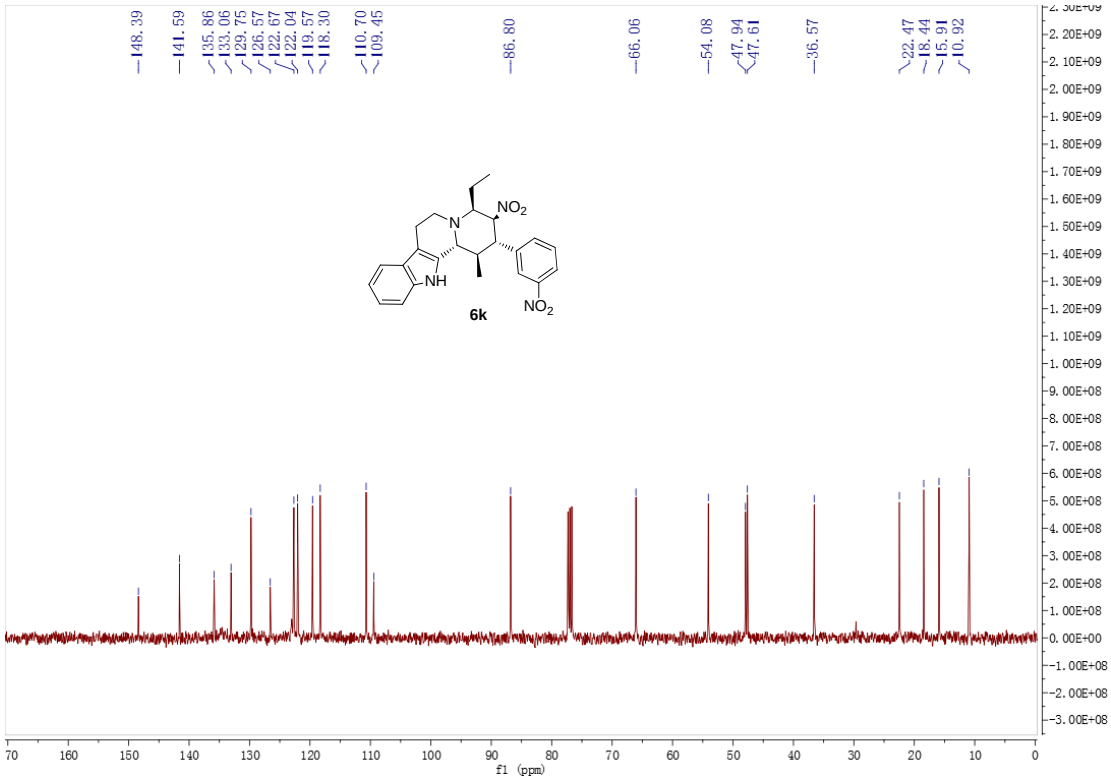
7.600
7.581
7.525
7.506
7.499
7.479
7.459
7.310
7.291
7.239
7.190
7.173
7.155
7.130
7.112
7.095
5.399
5.386
5.370
5.356
3.878
3.852
3.715
3.705
3.688
3.677
3.569
3.558
3.544
3.531
3.519
3.500
3.471
3.441
3.142
3.129
3.114
3.101
2.998
2.984
2.973
2.882
2.874
2.844
2.836
2.336
2.321
1.972
1.955
1.946
1.928
1.411
1.400
1.393
1.383
0.956
0.939
0.924
NO2
NH
N
NO2
6k
0.95
0.99
0.91
0.95
2.02
0.96
0.97
0.96
1.00
0.92
0.97
0.97
0.93
0.93
0.99
0.94
0.97
0.98
1.08
5.91
f1 (ppm)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126.57
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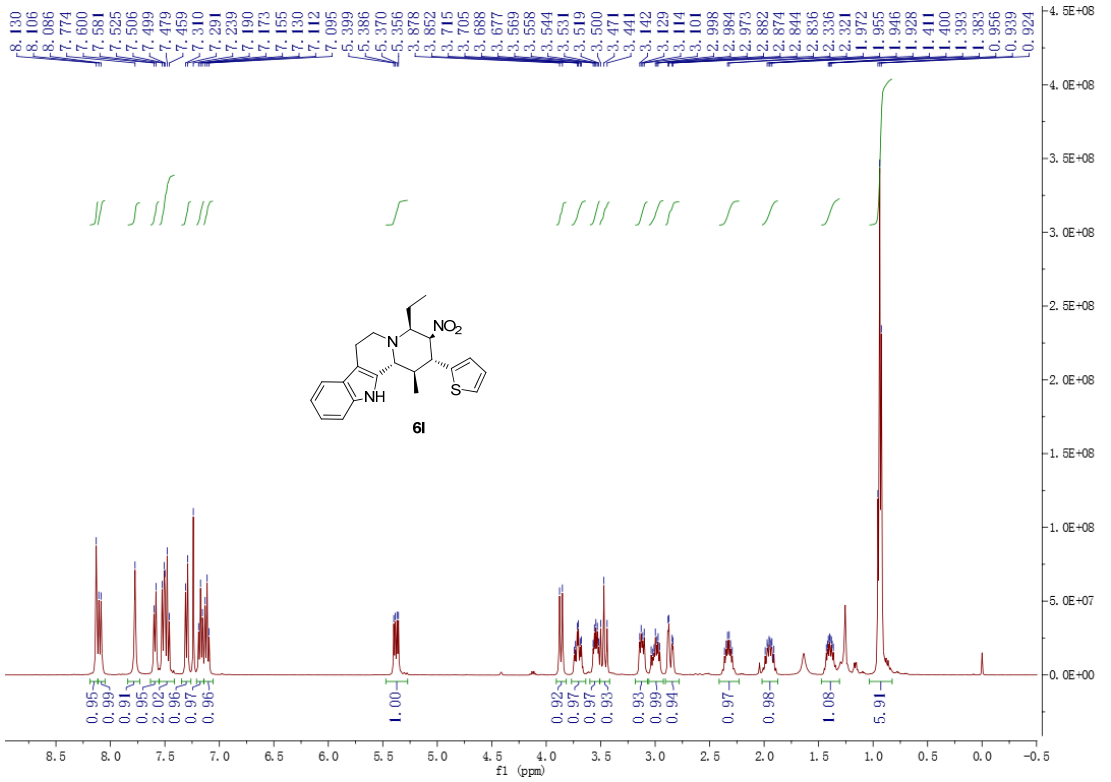
122.04
119.57
118.30
110.70
109.45
86.80
66.06
54.08
47.94
47.61
36.57
22.47
18.44
15.91
10.92
NO2
NH
N
NO2
6k
f1 (ppm)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8.106
8.086
7.774
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7.581
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7.506
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7.479
7.459
7.310
7.291
7.239
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7.173
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7.130
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3.500
3.471
3.441
3.142
3.129
3.114
3.101
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2.882
2.874
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2.836
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2.321
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1.955
1.946
1.928
1.411
1.400
1.393
1.383
0.956
0.939
0.924
NO2
N
NH
S
6l
0.95
0.99
0.91
0.95
2.02
0.96
0.97
0.96
1.00
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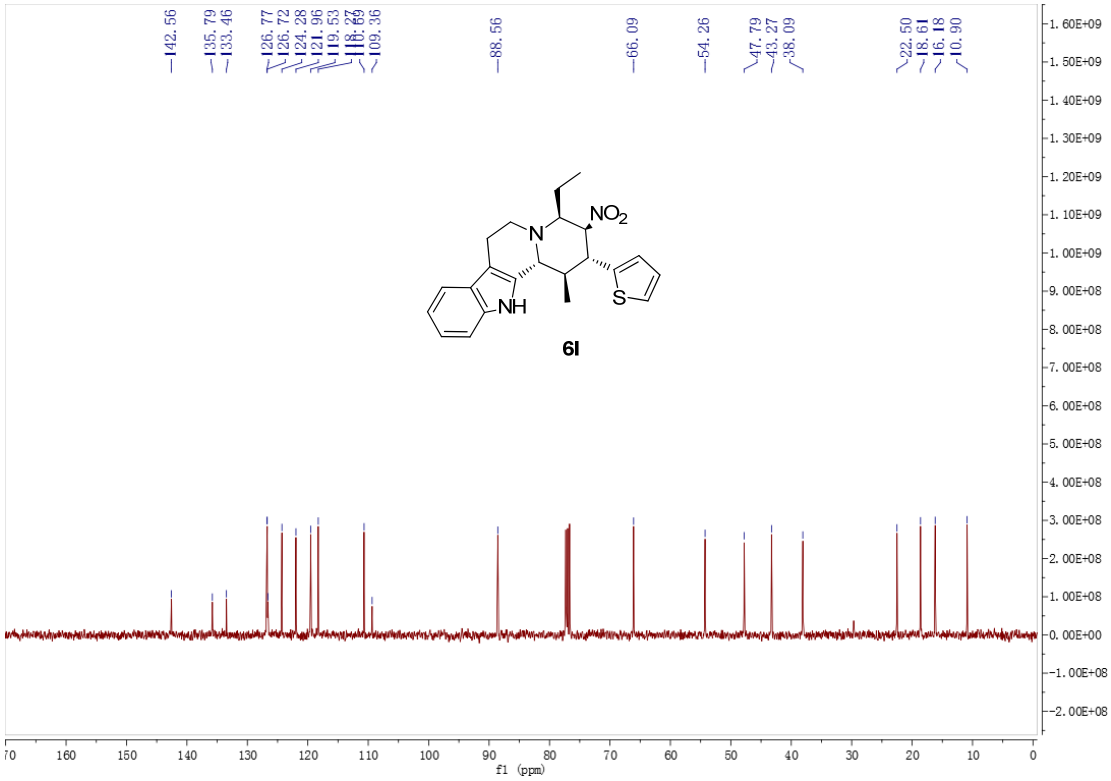
0.97
0.97
0.93
0.93
0.99
0.94
0.97
0.98
1.08
5.91
f1 (ppm)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124.28
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119.53
109.36
88.56
66.09
54.26
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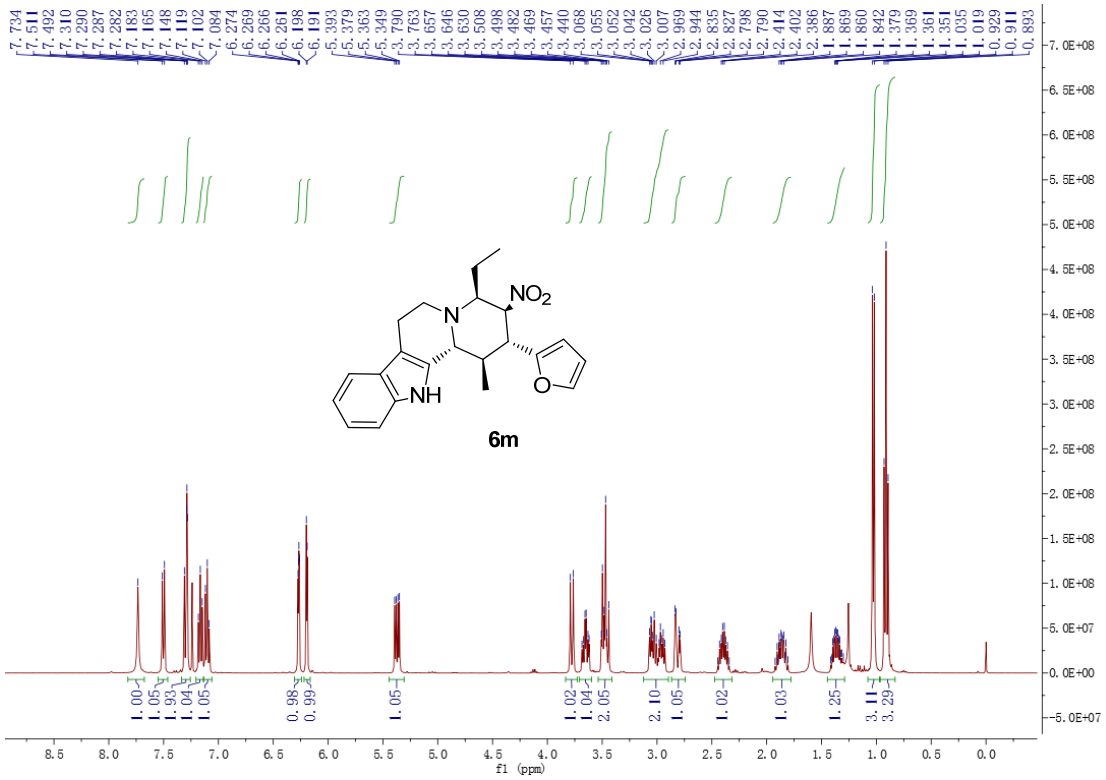
38.09
22.50
18.61
16.18
10.90
NO2
N
NH
S
6l
f1 (ppm)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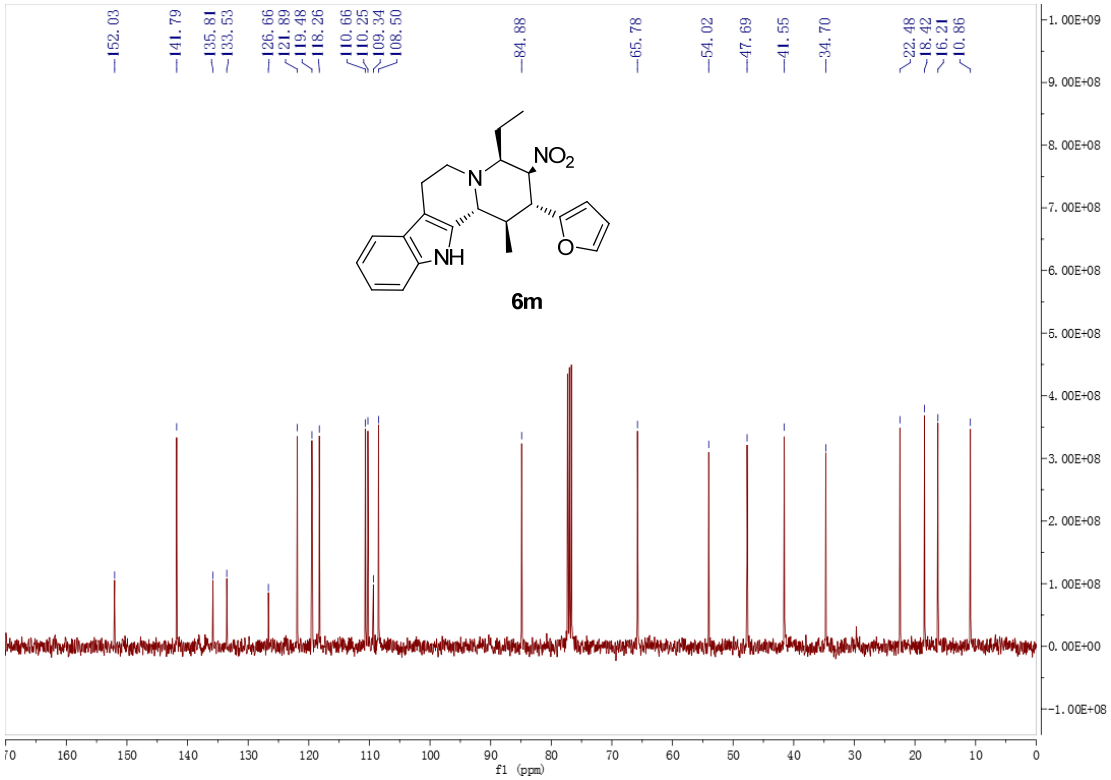
NH
O
6m
f1 (ppm)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NH
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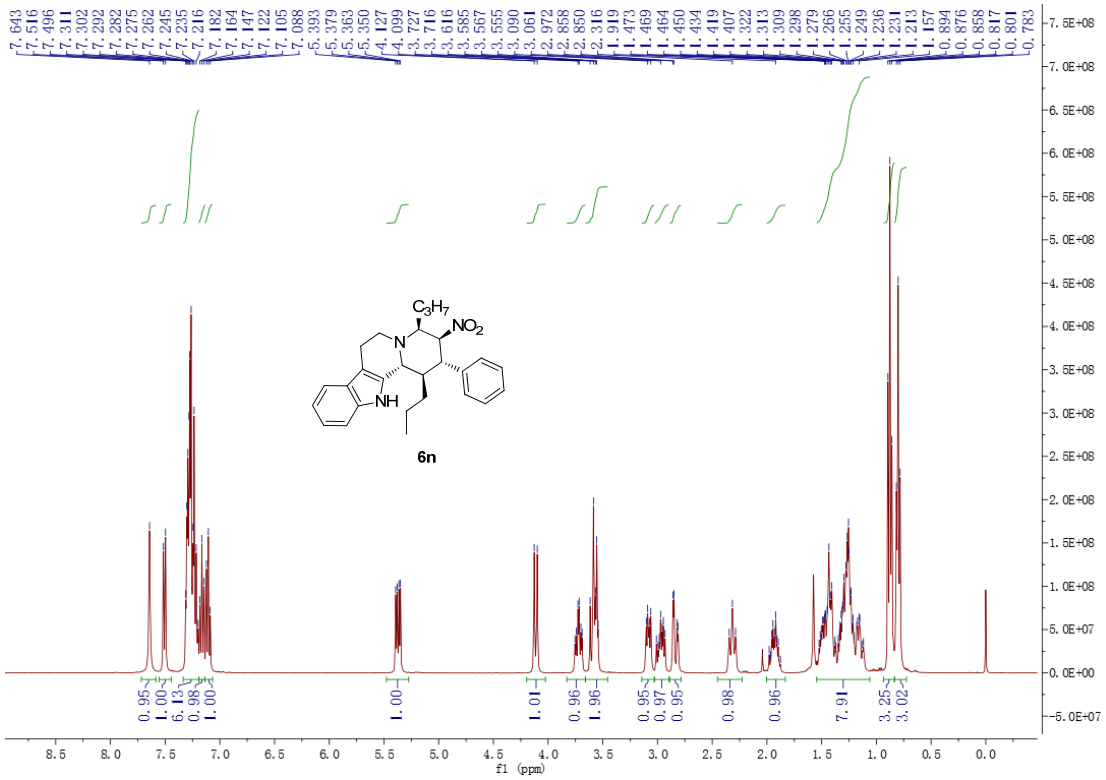
f1 (ppm)


7.643 7.516 7.496 7.311 7.302 7.292 7.282 7.275 7.262 7.245 7.235 7.216 7.182 7.164 7.147 7.122 7.105 7.088 5.393 5.379 5.363 5.350 4.127 4.099 3.727 3.716 3.616 3.585 3.567 3.555 3.090 3.061 2.972 2.858 2.850 2.316 1.919 1.473 1.469 1.464 1.450 1.434 1.419 1.407 1.322 1.313 1.309 1.298 1.279 1.266 1.255 1.249 1.236 1.231 1.213 1.157 0.894 0.876 0.858 0.817 0.801 0.783
C3H7
NO2
N
NH
6n
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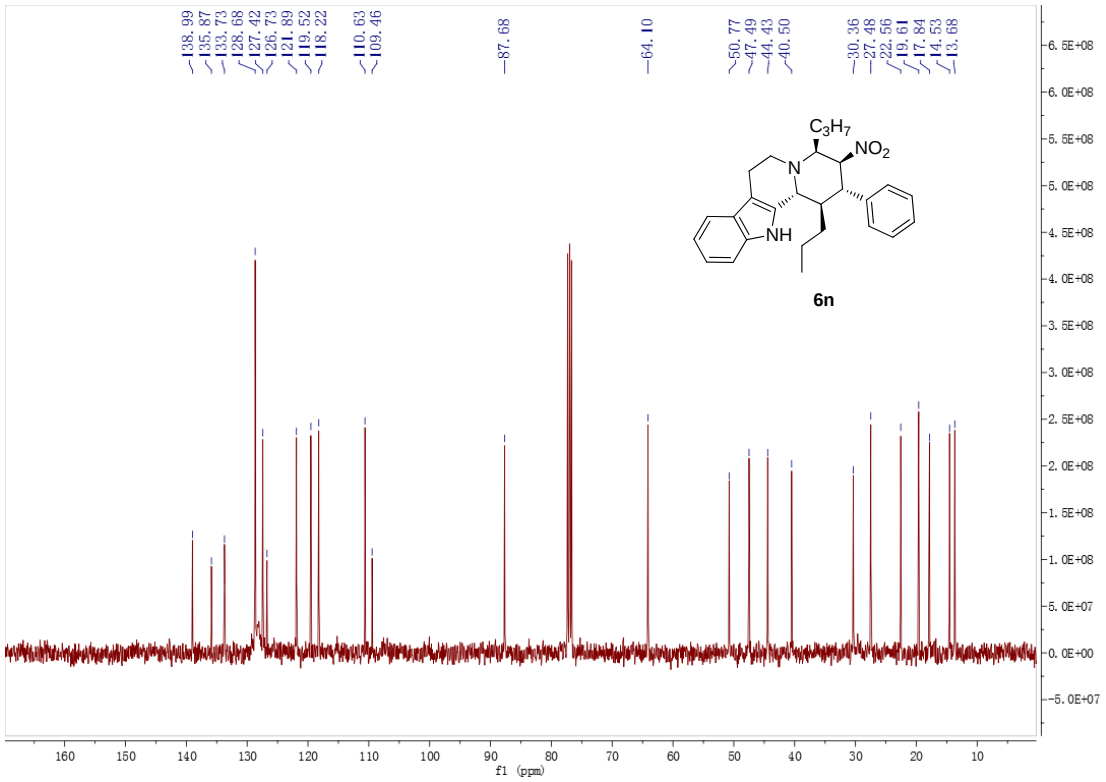
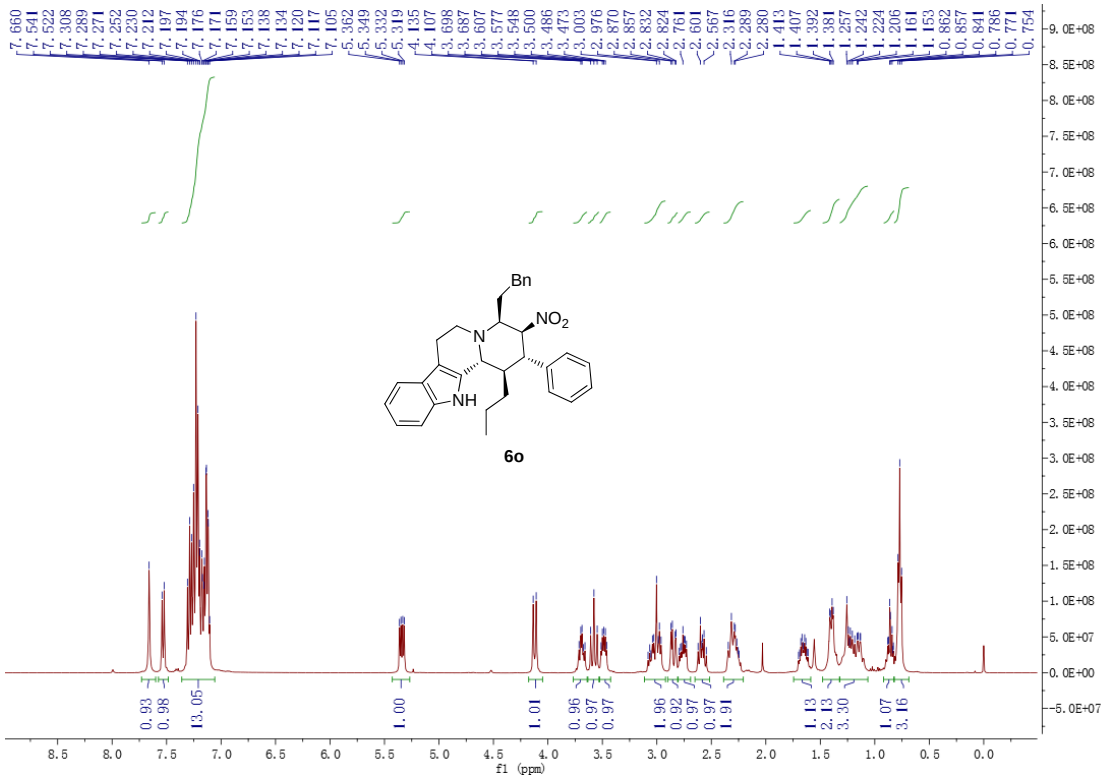
f1 (ppm)


138.99 135.87 133.73 128.68 127.42 126.73 121.89 119.52 118.22 110.63 109.46 87.68 64.10 50.77 47.49 44.43 40.50 30.36 27.48 22.56 19.61 17.84 14.53 13.68
C3H7
NO2
N
NH
6n
f1 (ppm)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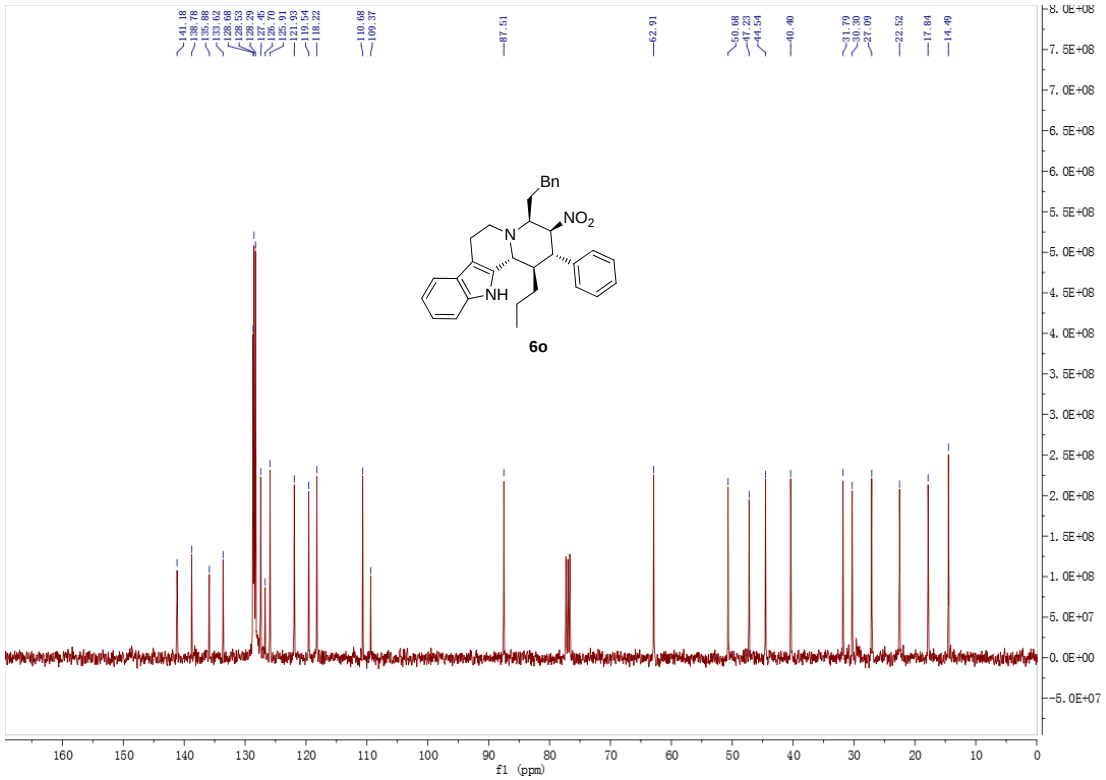
NH
6o
f1 (ppm)


Bn
NO2
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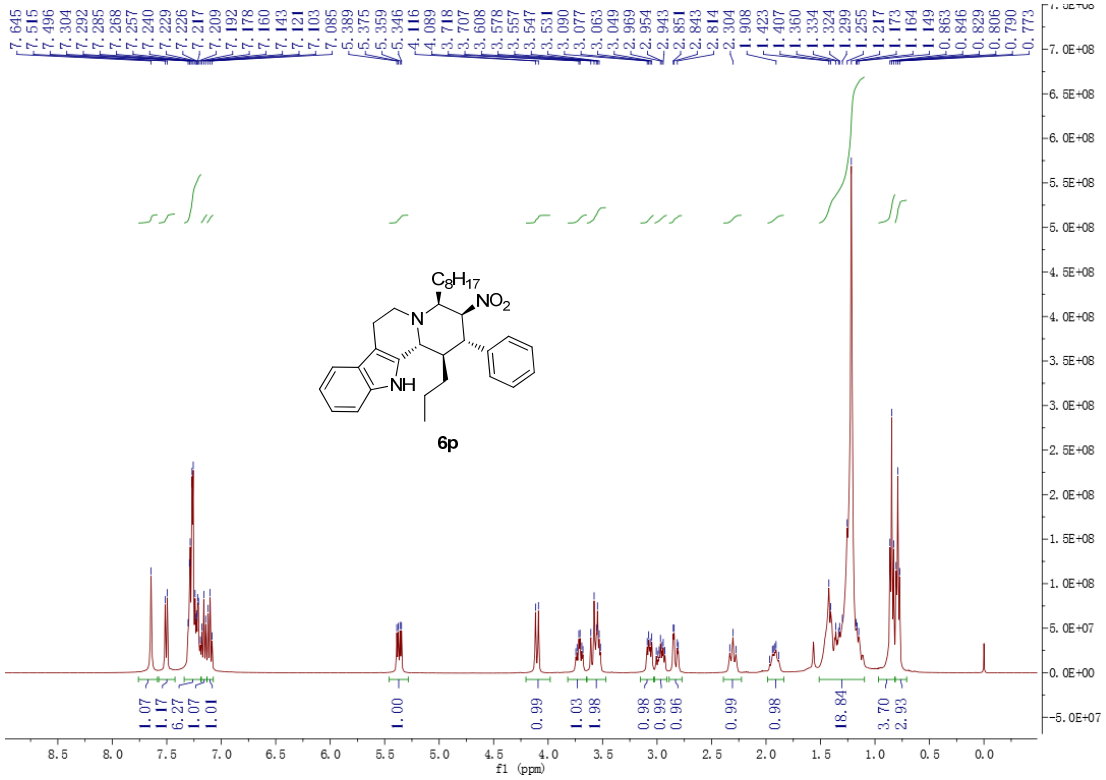
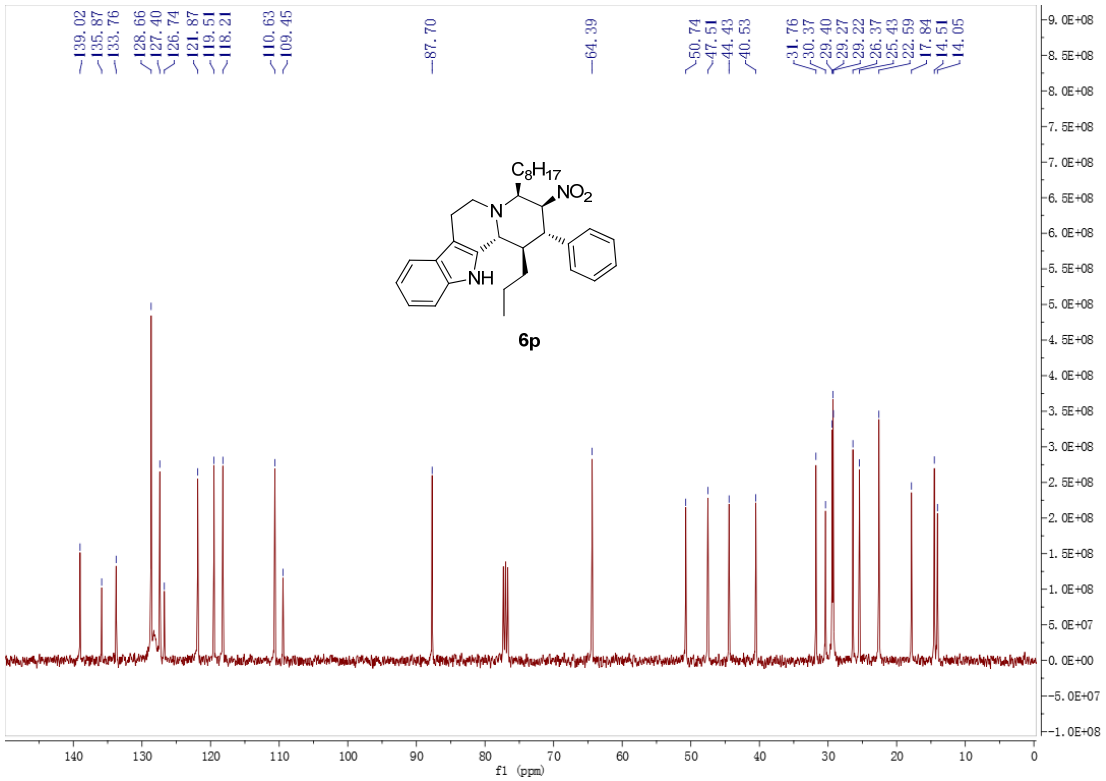
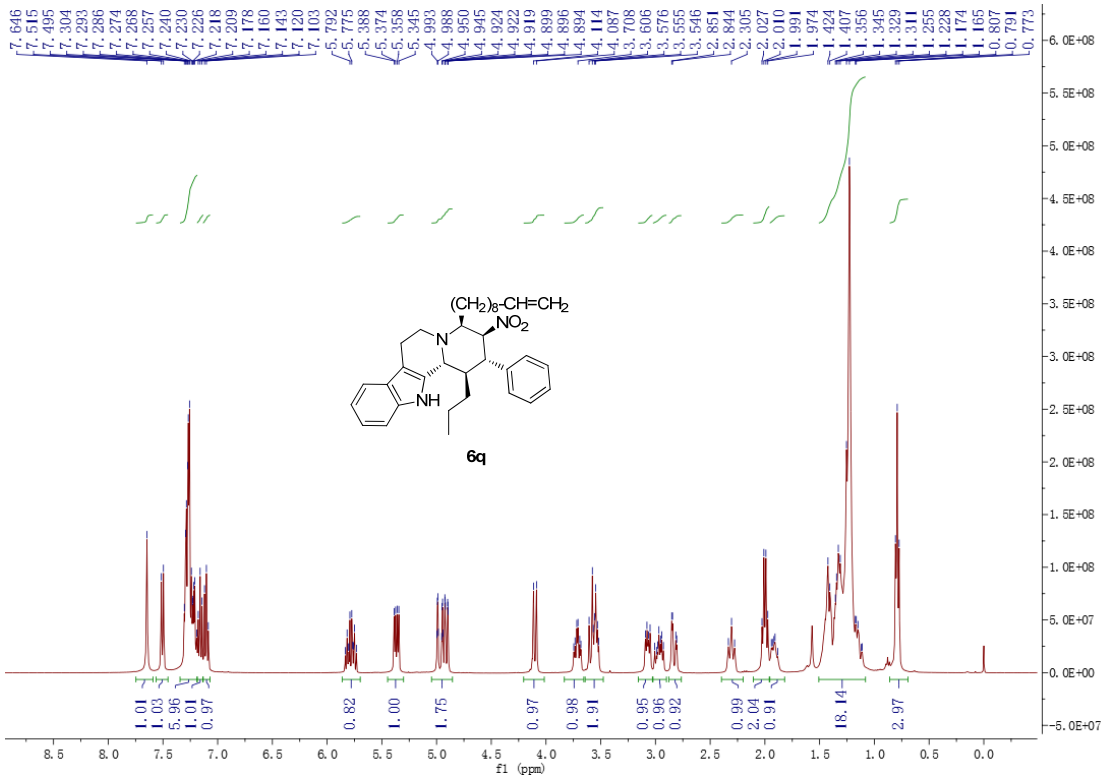
NH
6o
f1 (ppm)


7.645 7.515 7.496 7.304 7.292 7.285 7.268 7.257 7.240 7.229 7.226 7.217 7.209 7.192 7.178 7.160 7.143 7.121 7.103 7.085 5.389 5.375 5.359 5.346 4.116 4.089 3.718 3.707 3.608 3.578 3.557 3.547 3.531 3.090 3.077 3.063 3.049 2.969 2.954 2.943 2.851 2.843 2.814 2.304 1.908 1.423 1.407 1.360 1.334 1.324 1.299 1.255 1.217 1.173 1.164 1.149 0.863 0.846 0.829 0.806 0.790 0.773
C8H17
NO2
N
NH
6p
1.07 1.17 6.27 1.07 1.01 1.00 0.99 1.03 1.98 0.98 0.99 0.96 0.99 0.98 18.84 3.70 2.93
f1 (ppm)


139.02 135.87 133.76 128.66 127.40 126.74 121.87 119.51 118.21 110.63 109.45 87.70 64.39 50.74 47.51 44.43 40.53 31.76 30.37 29.40 29.27 29.22 26.37 25.43 22.59 17.84 14.51 14.05
C8H17
NO2
N
NH
6p
f1 (ppm)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7.240
7.230
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7.218
7.209
7.178
7.160
7.143
7.120
7.103
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5.388
5.374
5.358
5.345
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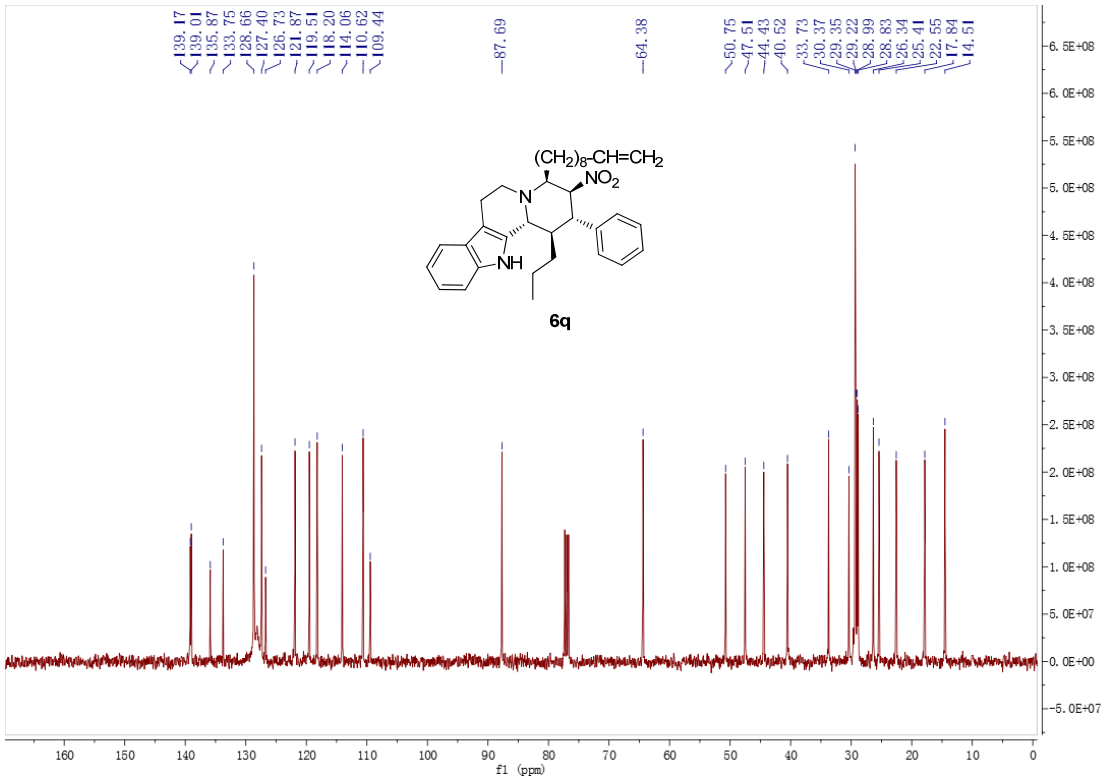
4.988
4.950
4.945
4.924
4.922
4.919
4.899
4.896
4.894
4.114
4.087
3.708
3.606
3.576
3.555
3.546
2.851
2.844
2.305
2.027
2.010
1.991
1.974
1.424
1.407
1.356
1.345
1.329
1.311
1.255
1.228
1.174
1.165
0.807
0.791
0.773
(CH2)8-CH=CH2
NO2
N
NH
6q
1.01
1.03
5.96
1.01
0.97
0.82
1.00
1.75
0.97
0.98
1.91
0.95
0.96
0.92
0.99
2.04
0.91
18.14
2.97
f1 (ppm)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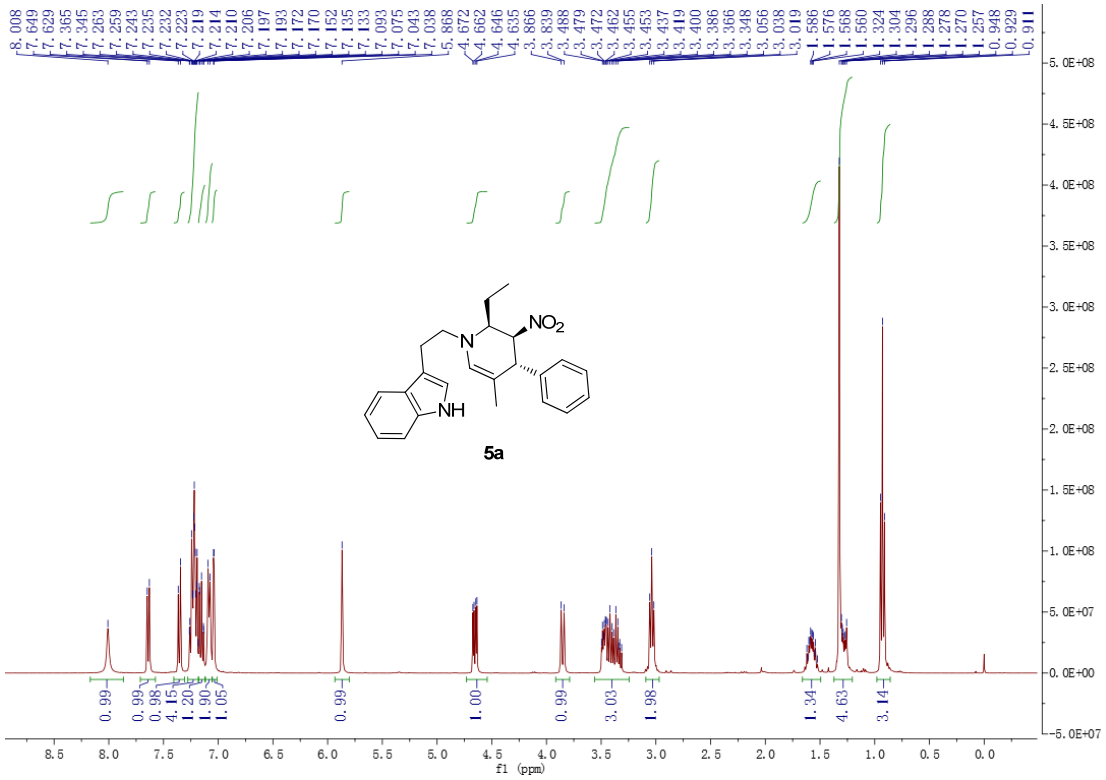
139.01
135.87
133.75
128.66
127.40
126.73
121.87
119.51
118.20
114.06
110.62
109.44
87.69
64.38
50.75
47.51
44.43
40.52
33.73
30.37
29.35
29.22
28.99
28.83
26.34
25.41
22.55
17.84
14.51
(CH2)8-CH=CH2
NO2
N
NH
6q
f1 (ppm)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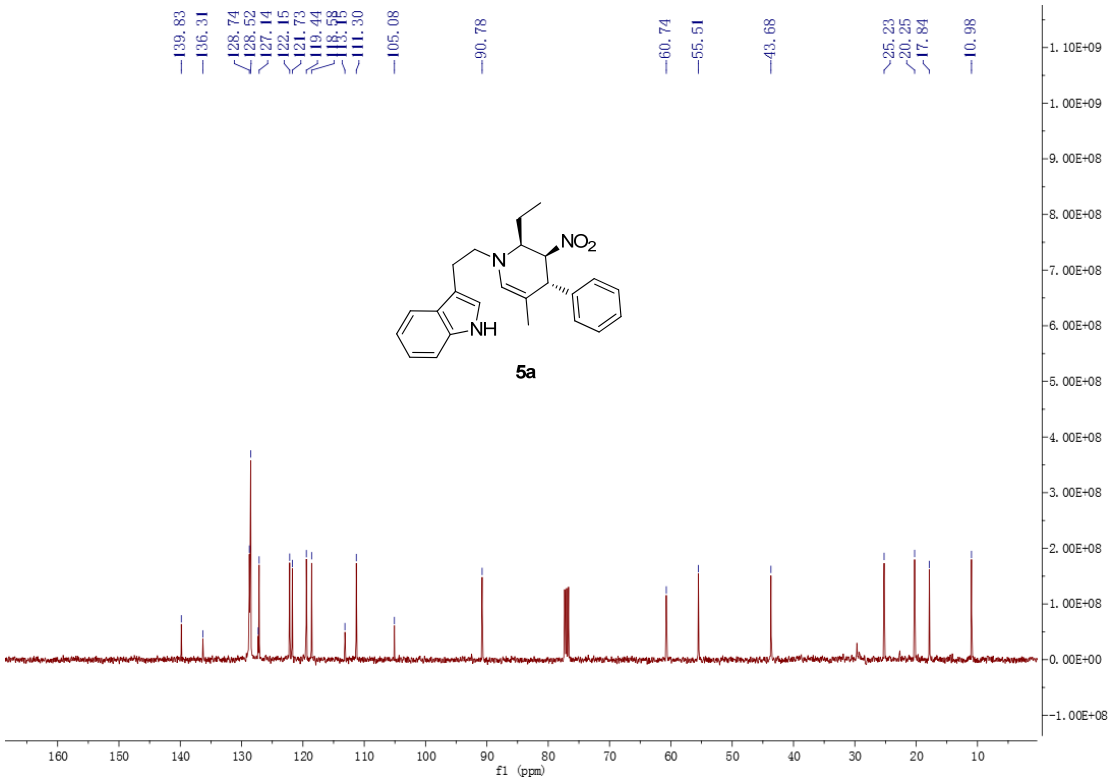
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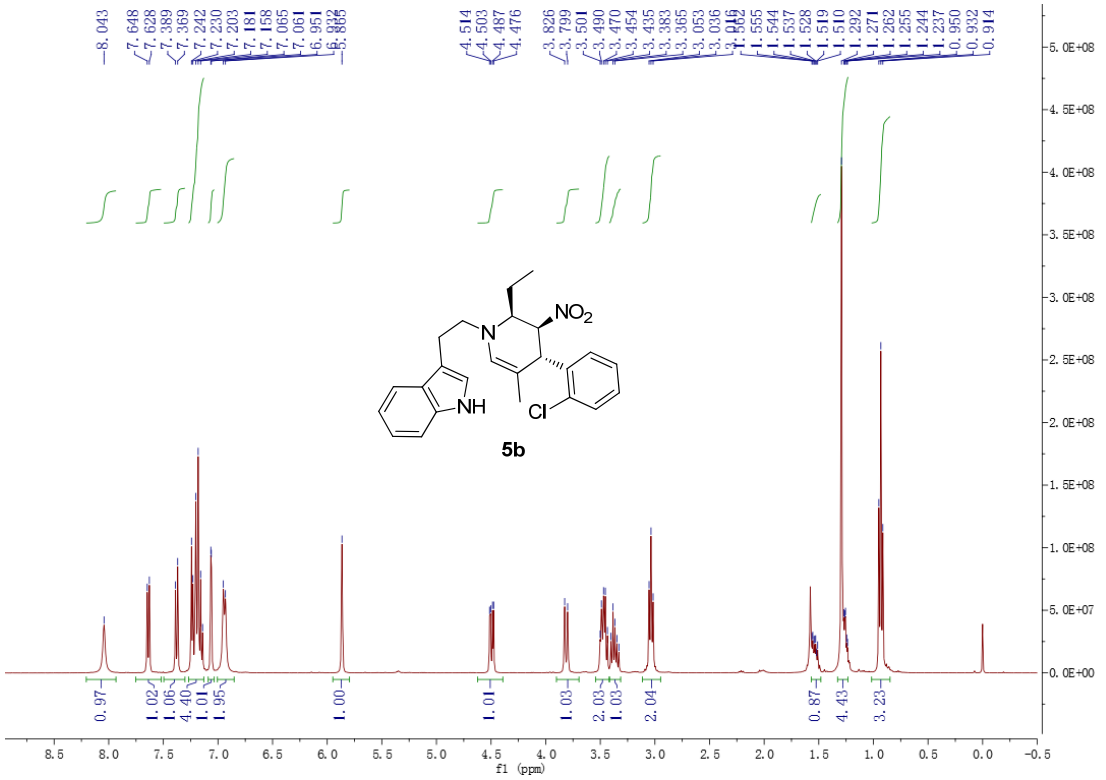
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6.932
6.885
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4.503
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4.476
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3.383
3.365
3.053
3.036
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1.544
1.537
1.528
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1.244
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Cl
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1.95
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1.01
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2.03
1.03
2.04
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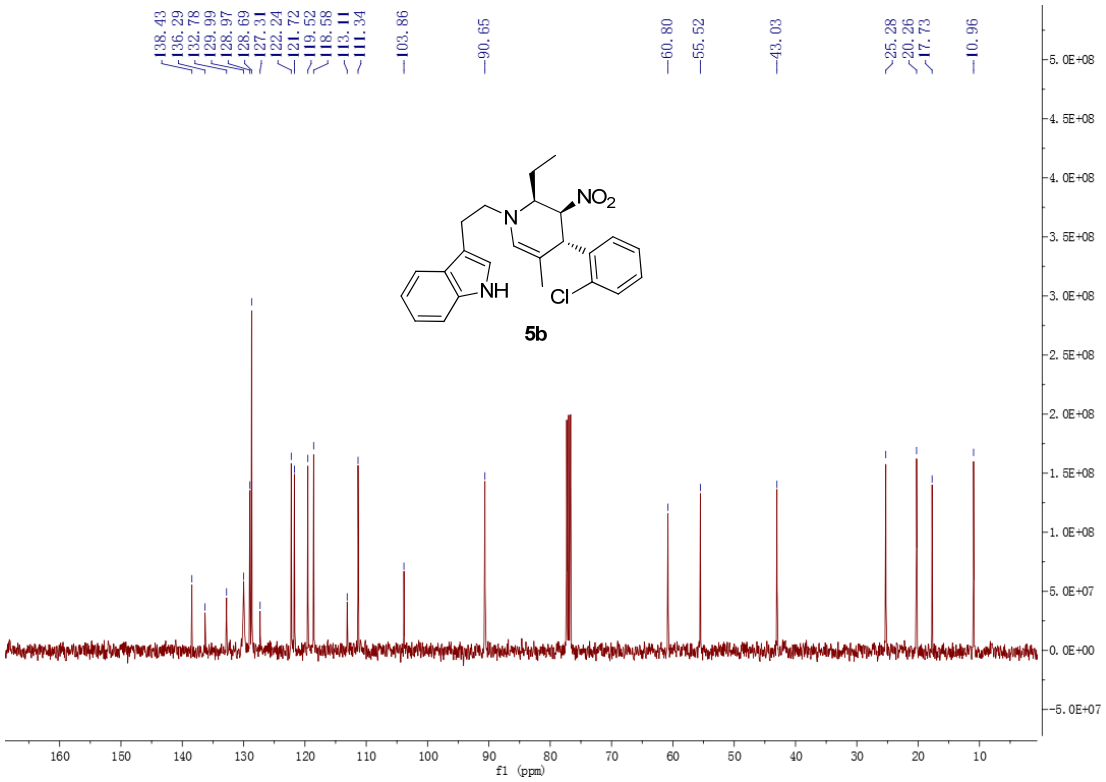
f1 (ppm)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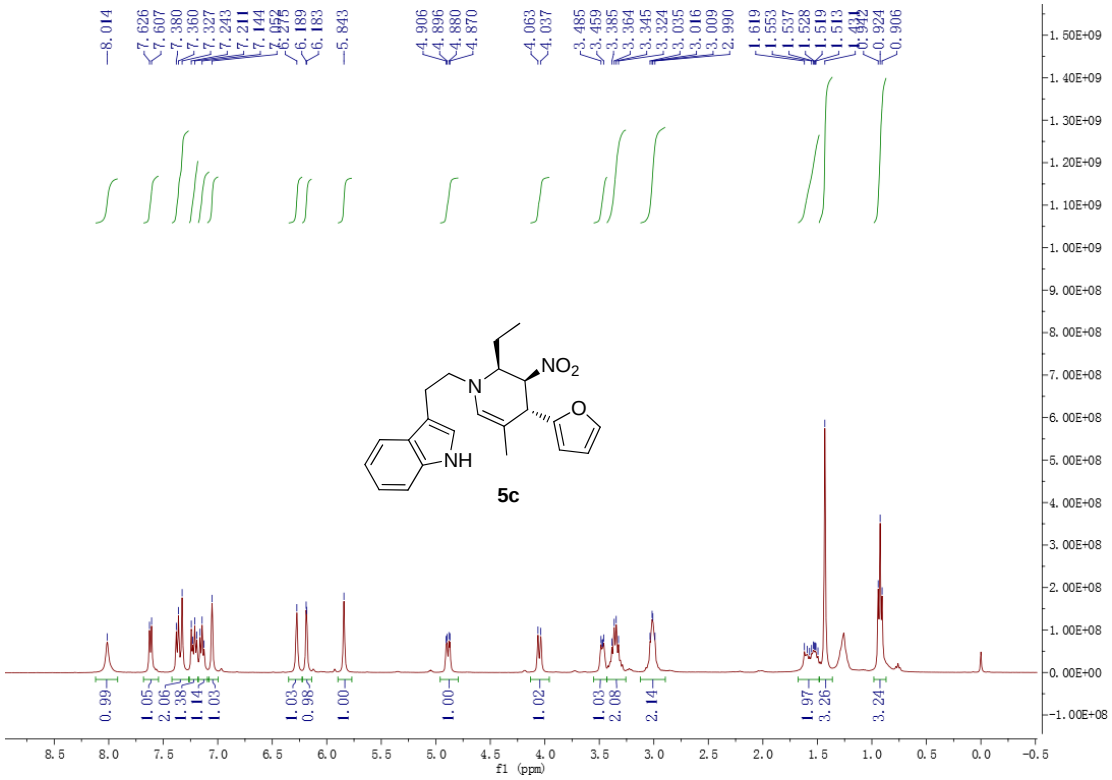
f1 (ppm)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4.880
4.870
4.063
4.037
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3.459
3.385
3.364
3.345
3.324
3.035
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1.619
1.553
1.537
1.528
1.519
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0.924
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NH
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1.14
1.03
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1.03
2.08
2.14
1.97
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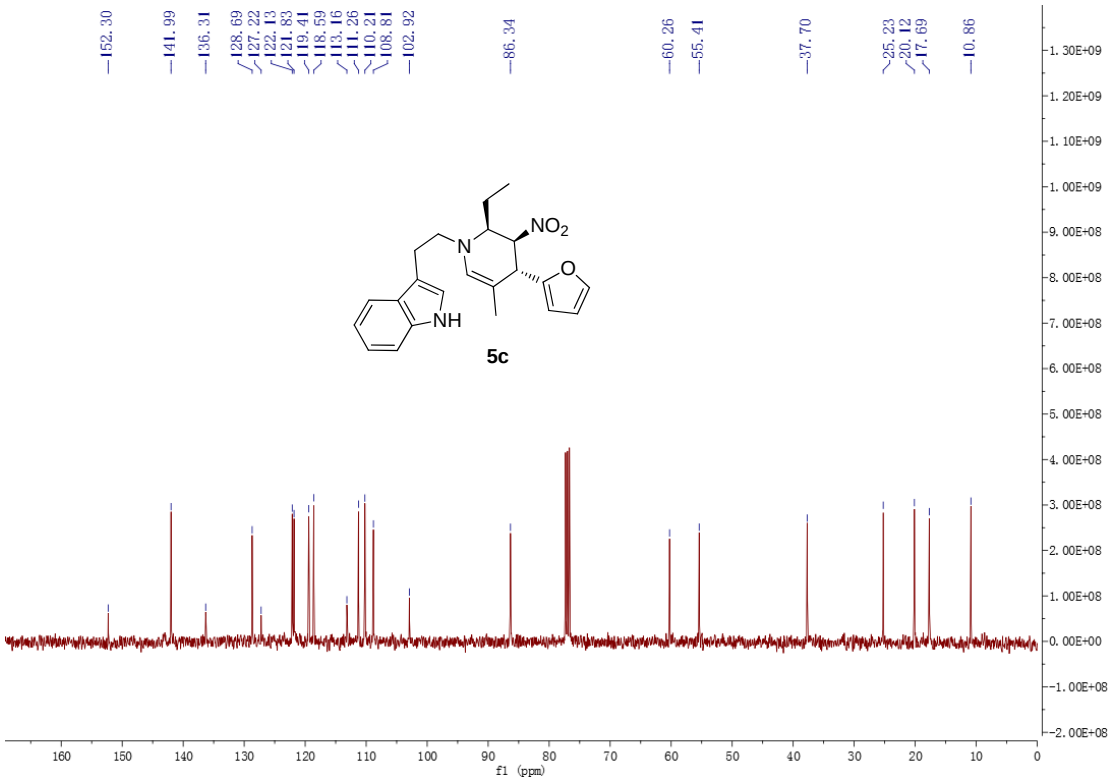
3.24
f1 (ppm)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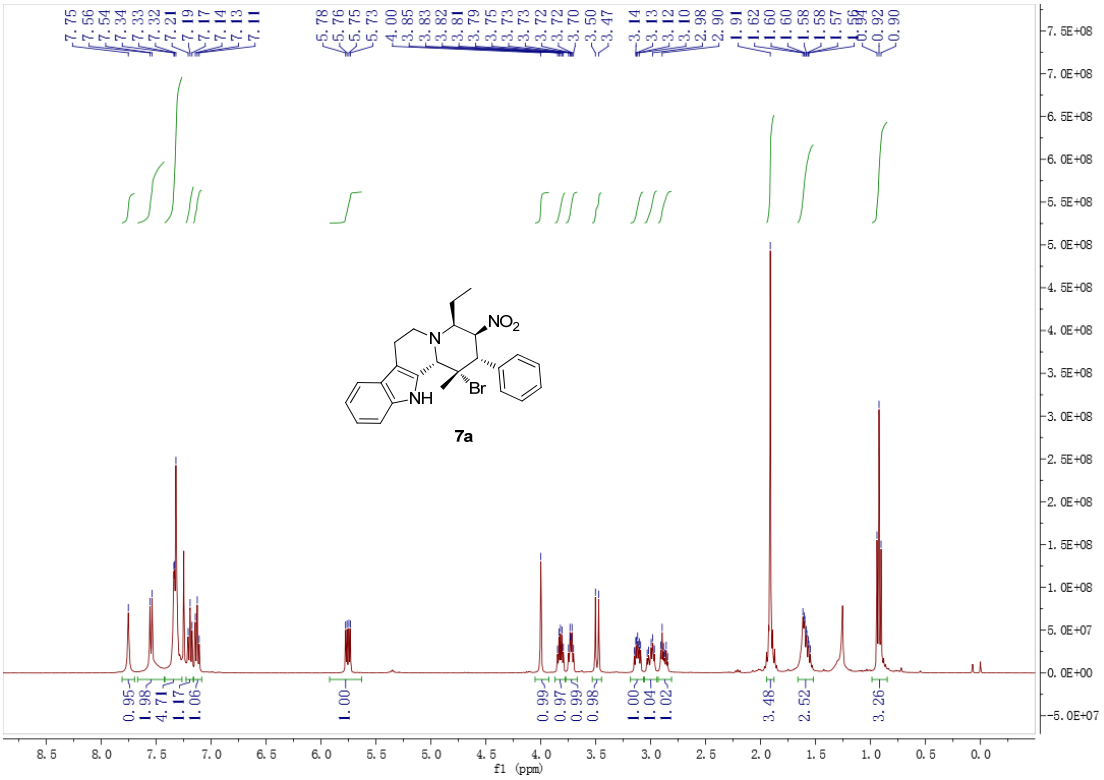
f1 (ppm)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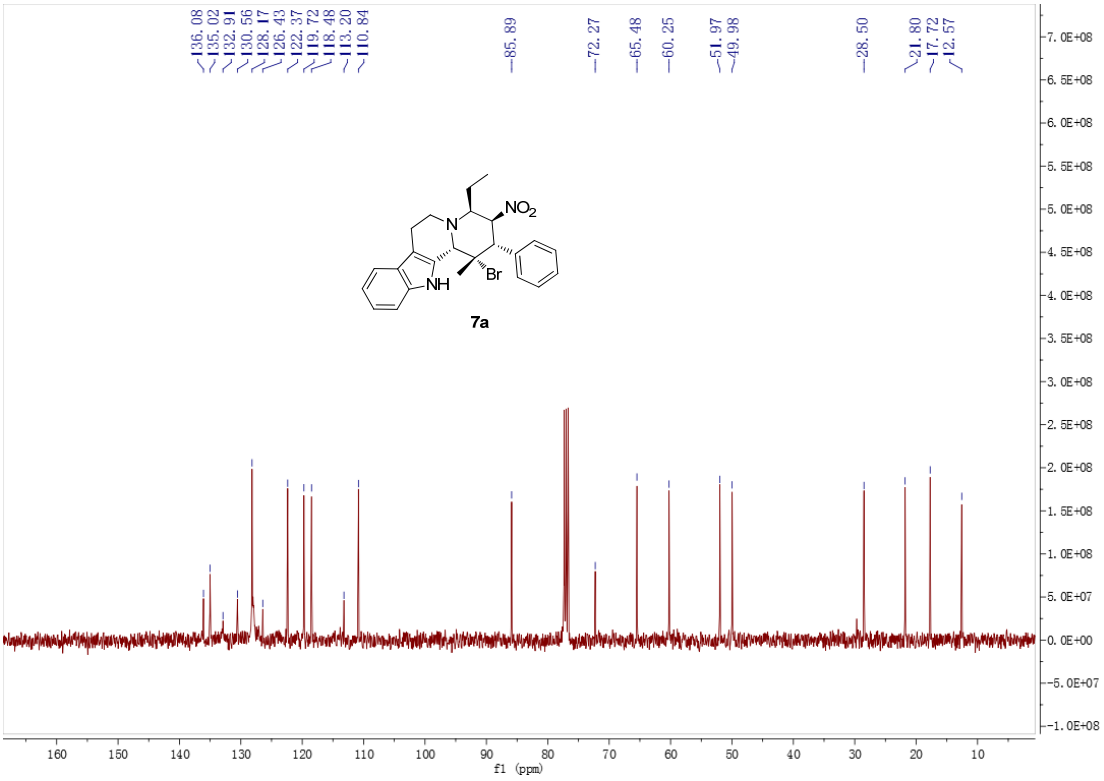
5.78 5.76 5.75 5.73
4.00 3.85 3.83 3.82 3.81 3.79 3.75 3.73 3.73 3.72 3.72 3.70 3.50 3.47
3.14 3.13 3.12 3.10 2.98 2.90
1.91 1.62 1.60 1.60 1.58 1.58 1.57
0.92 0.90
NO2
N
NH
Br
7a
0.95 1.98 4.71 1.17 1.06
1.00
0.99 0.97 0.99 0.98
1.00 1.04 1.02
3.48 2.52 3.26
f1 (ppm)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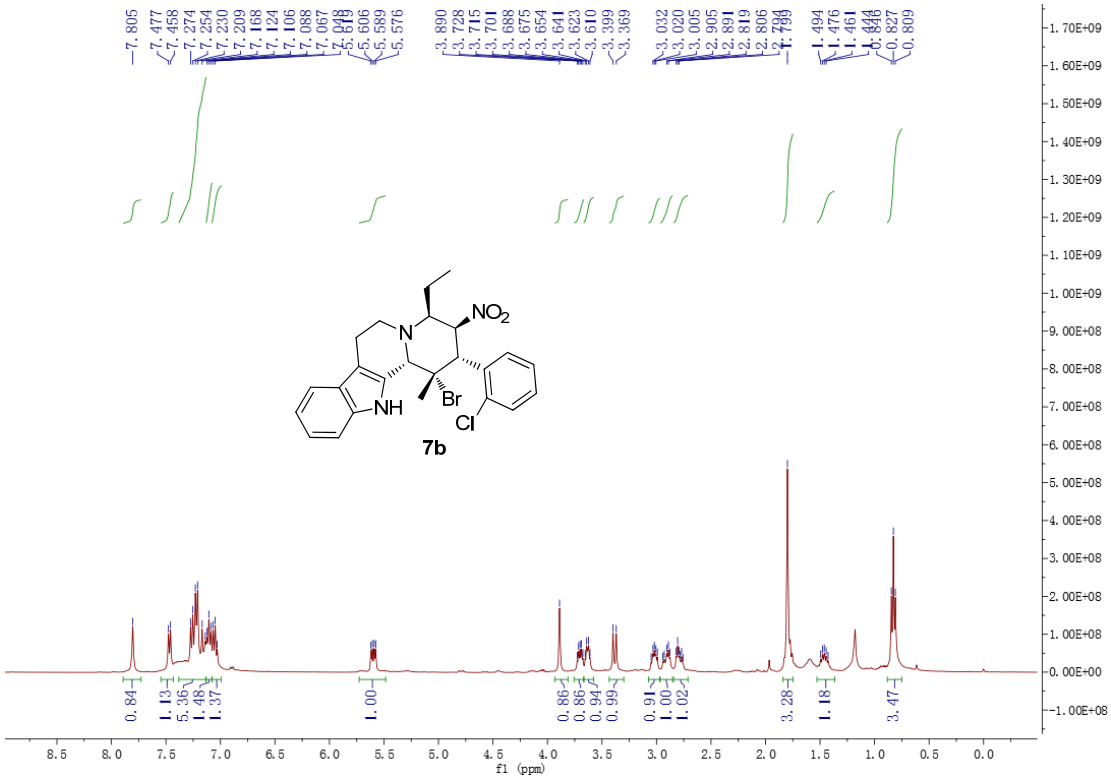
85.89 72.27 65.48 60.25 51.97 49.98 28.50 21.80 17.72 12.57
NO2
N
NH
Br
7a
f1 (ppm)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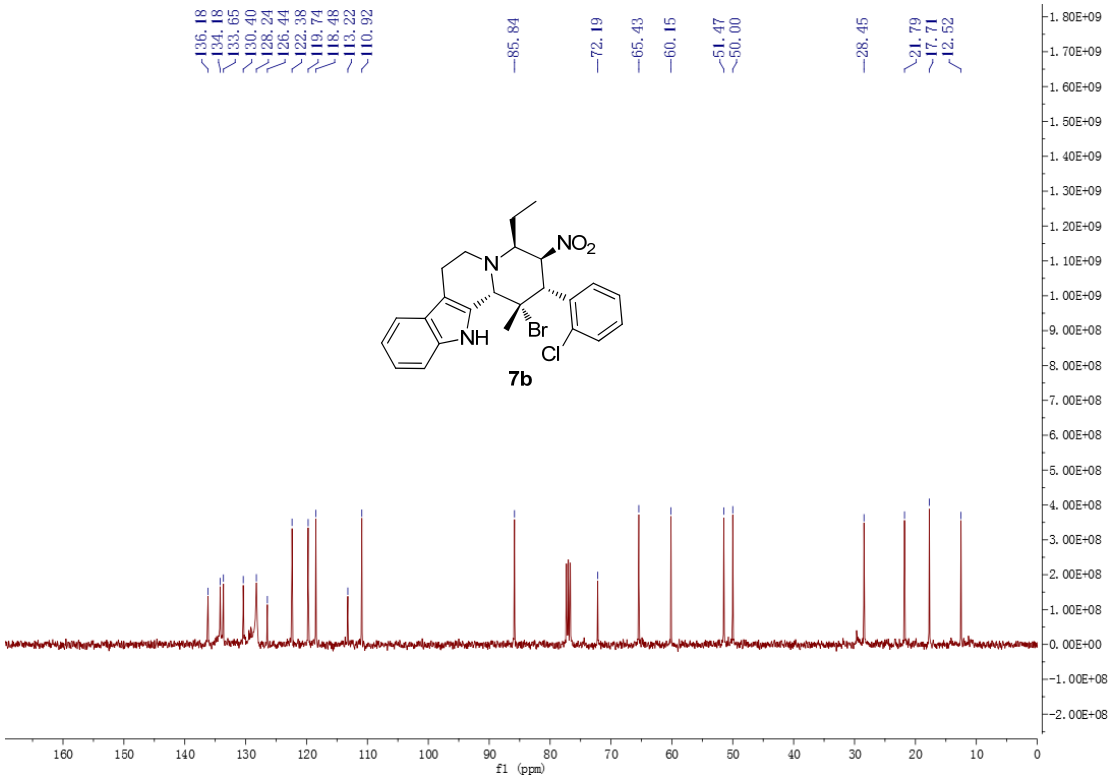
f1 (ppm)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65.43
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28.45
21.79
17.71
12.52
NO2
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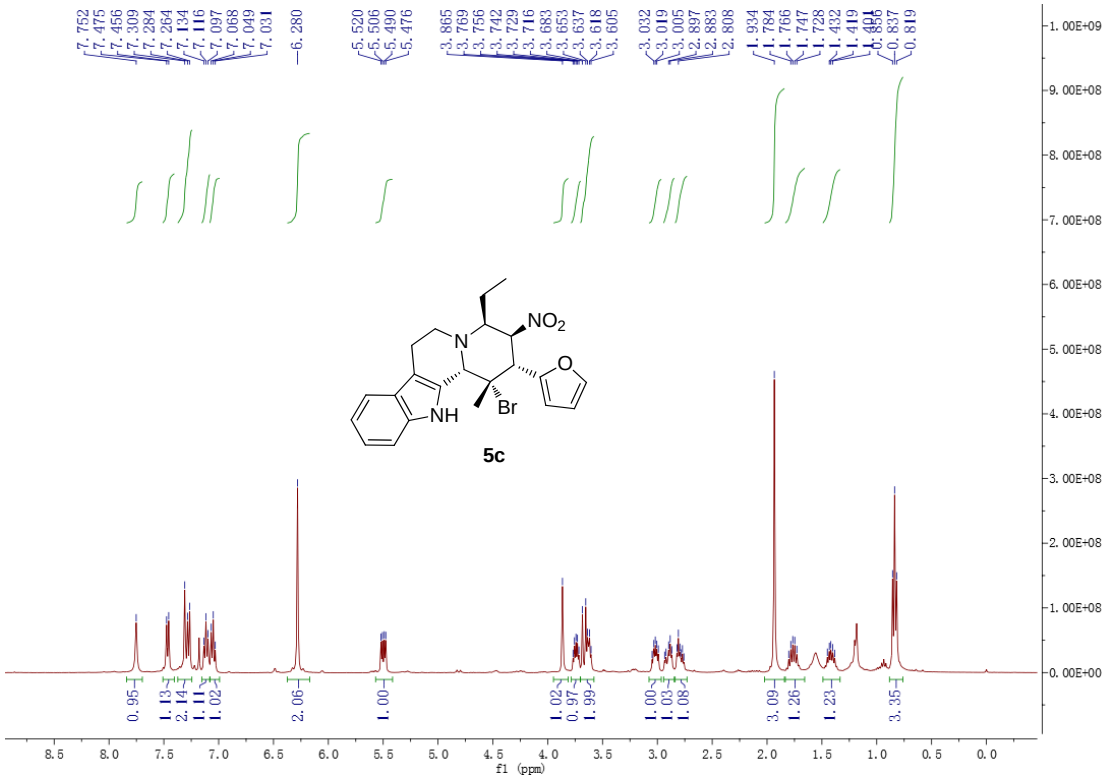
NH
Br
Cl
7b
f1 (ppm)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5.506
5.490
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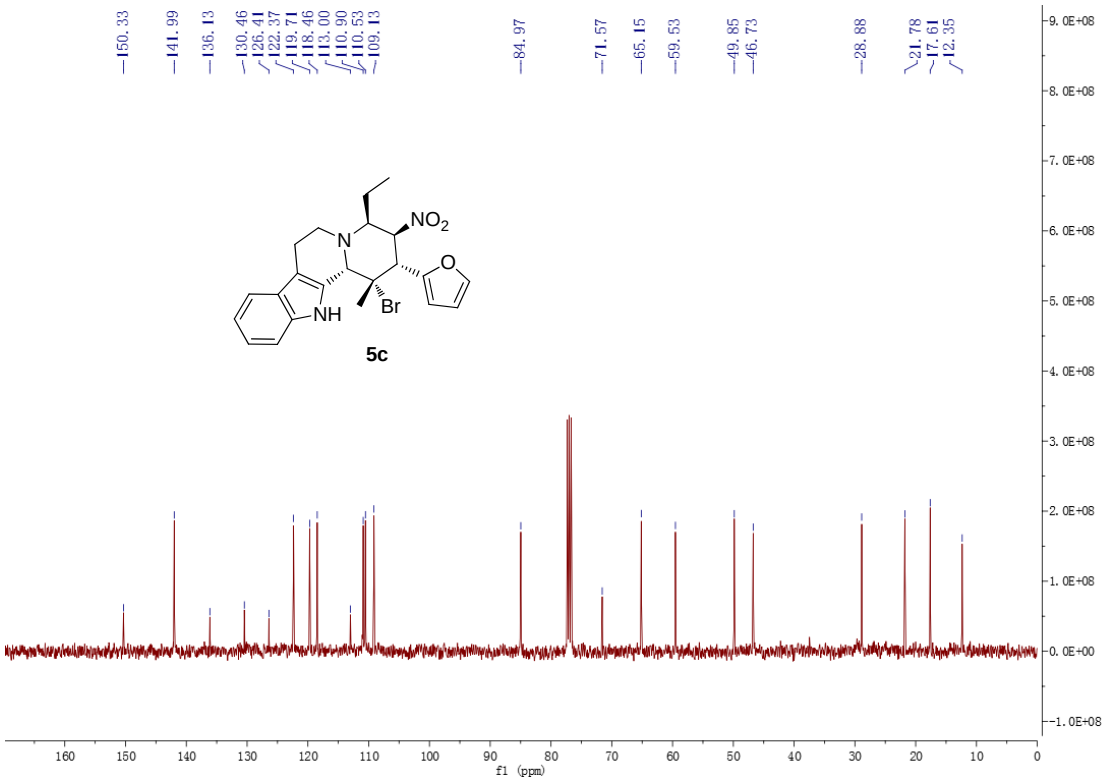
f1 (ppm)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59.53
49.85
46.73
28.88
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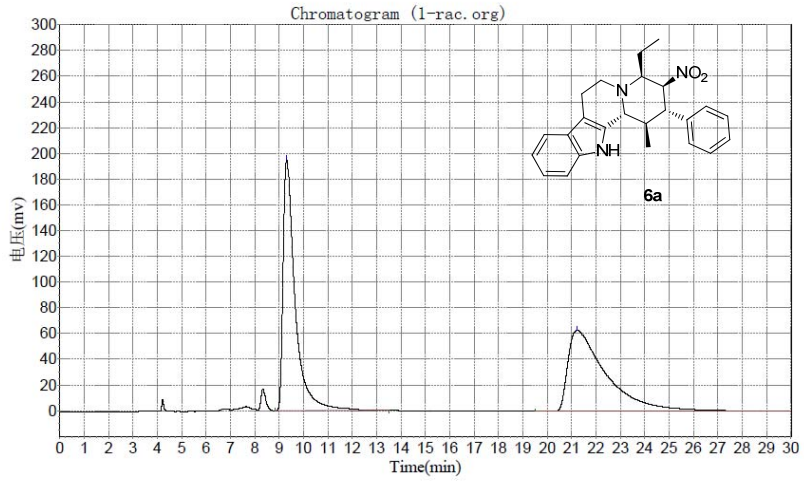
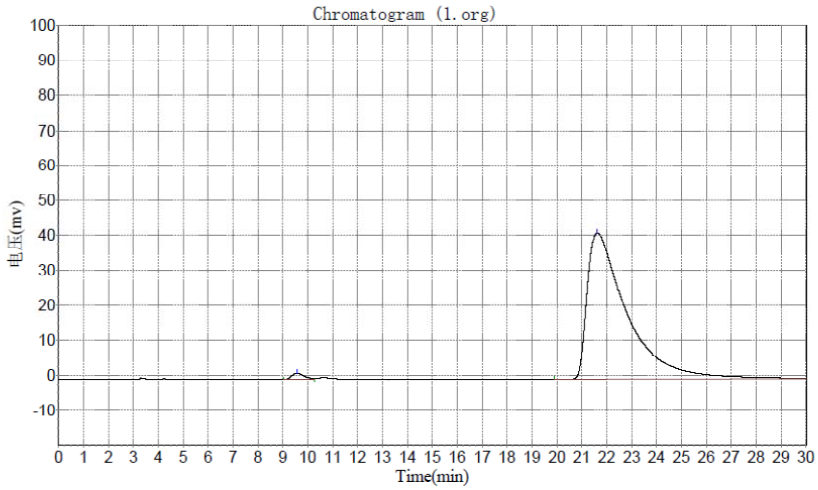
17.61
12.35
NO2
N
O
NH
Br
5c
f1 (ppm)

9. Chiral HPLC Traces



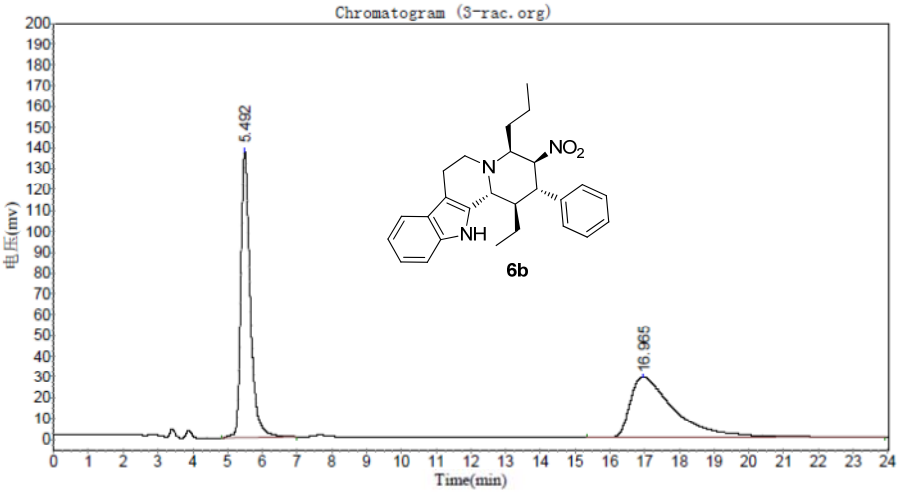
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.315	195437.734	6796518.000	49.4173
2		21.248	63000.684	6956804.000	50.5827
Total			258438.418	13753322.000	100.0000



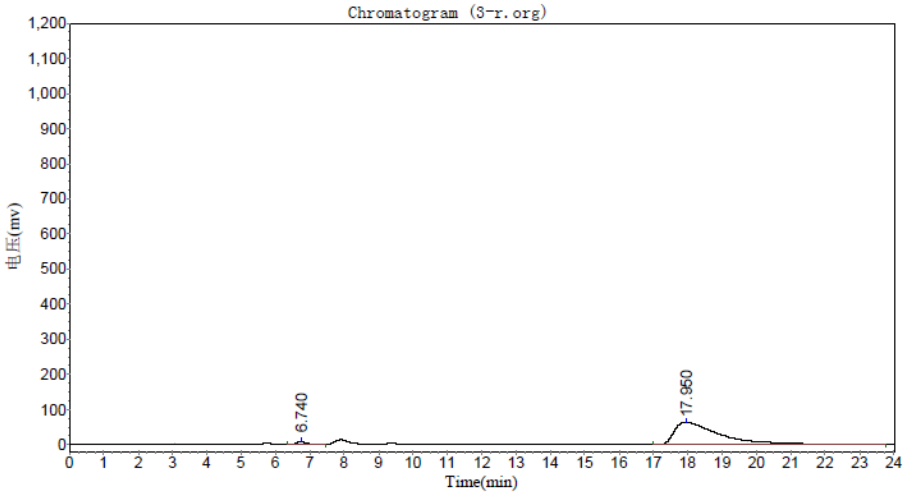
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.560	1861.781	63572.539	1.3616
2		21.622	41658.137	4605395.500	98.6384
Total			43519.917	4668968.039	100.0000



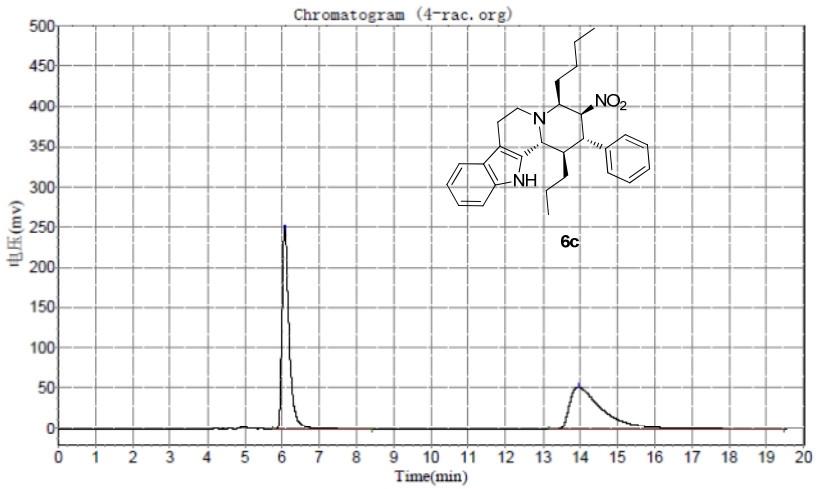
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.492	137133.453	2596633.750	50.4534
2		16.965	28900.949	2549960.500	49.5466
Total			166034.402	5146594.250	100.0000



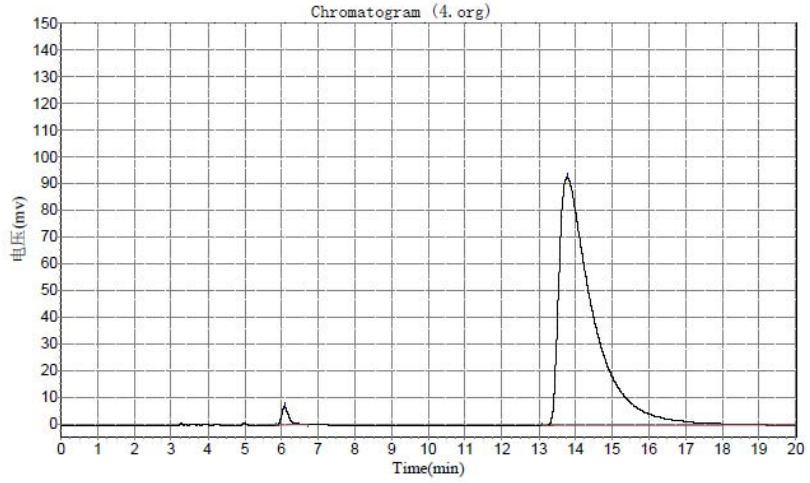
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.740	6657.645	132007.641	2.3524
2		17.950	62770.215	5479726.500	97.6477
Total			69427.859	5611734.141	100.0000



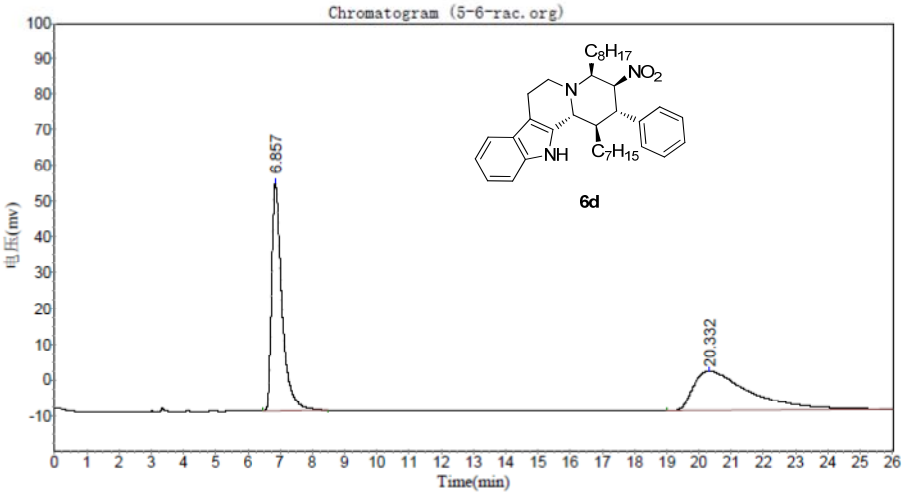
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.090	250976.688	3339507.500	50.4583
2		13.957	51720.270	3278843.000	49.5417
Total			302696.957	6618350.500	100.0000



Results

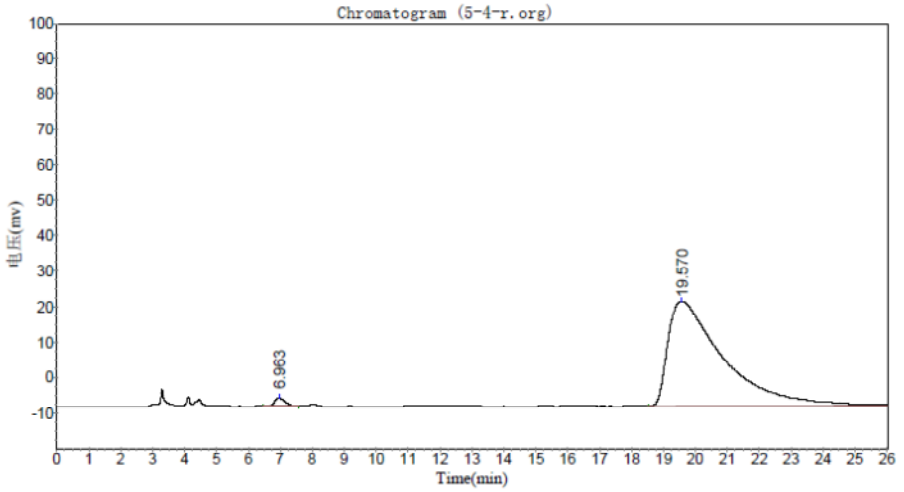
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.090	6859.786	91623.227	1.5147
2		13.790	92994.008	5957475.000	98.4853
Total			99853.793	6049098.227	100.0000



6d

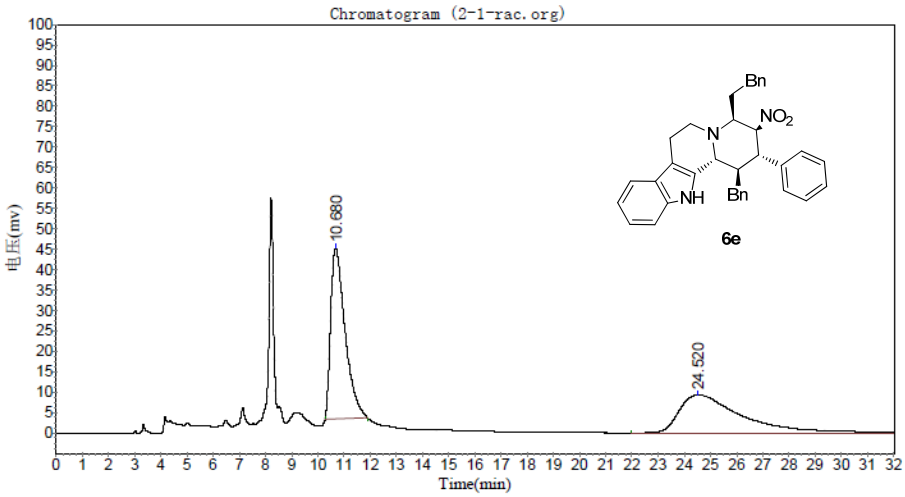
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.857	63869.441	1419488.500	51.2164
2		20.332	11034.188	1352062.000	48.7836
Total			74903.630	2771550.500	100.0000



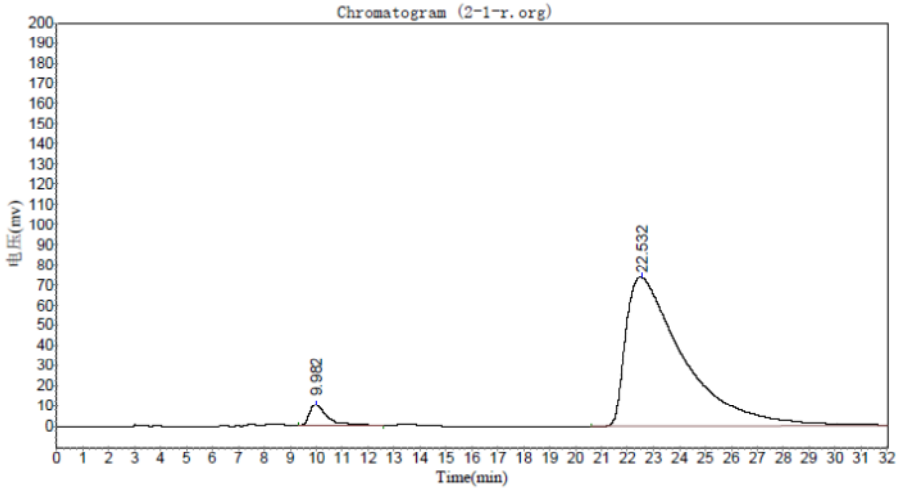
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.963	2318.823	54809.543	1.5027
2		19.570	29584.627	3592693.500	98.4973
Total			31903.450	3647503.043	100.0000



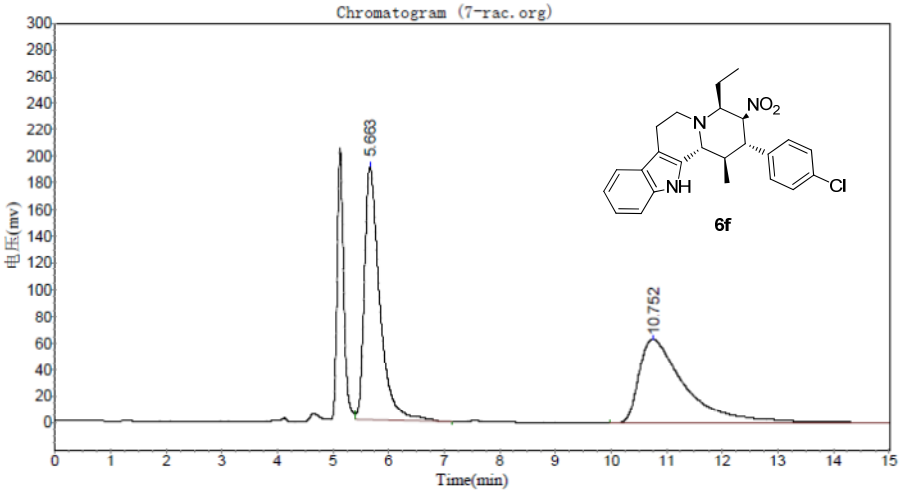
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		10.680	41882.863	1592529.500	50.9090
2		24.520	9319.682	1535656.125	49.0910
Total			51202.545	3128185.625	100.0000



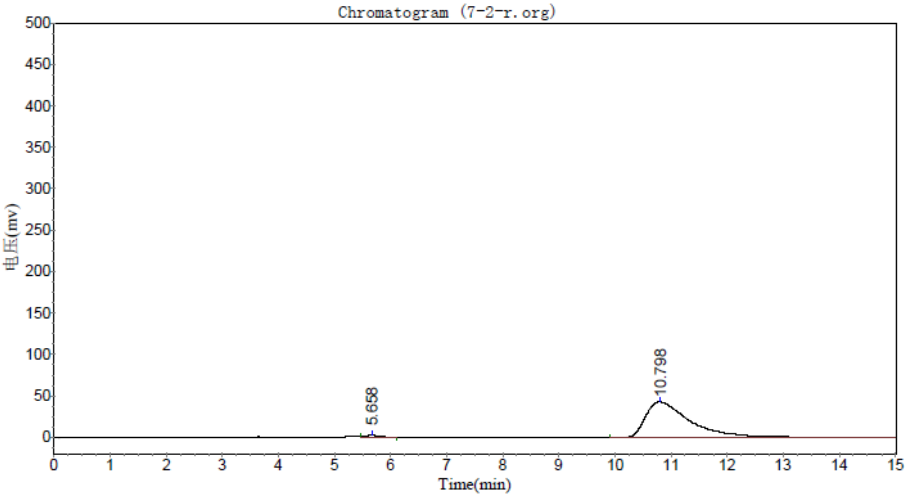
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.982	10308.383	553892.500	4.5947
2		22.532	73669.250	11501081.000	95.4053
Total			83977.633	12054973.500	100.0000



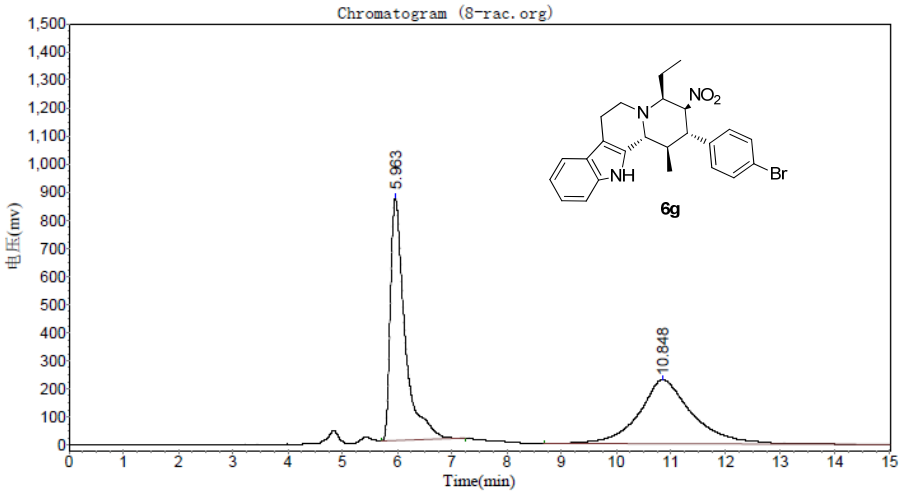
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.663	190413.953	3642722.000	49.2693
2		10.752	63406.887	3750771.250	50.7307
Total			253820.840	7393493.250	100.0000



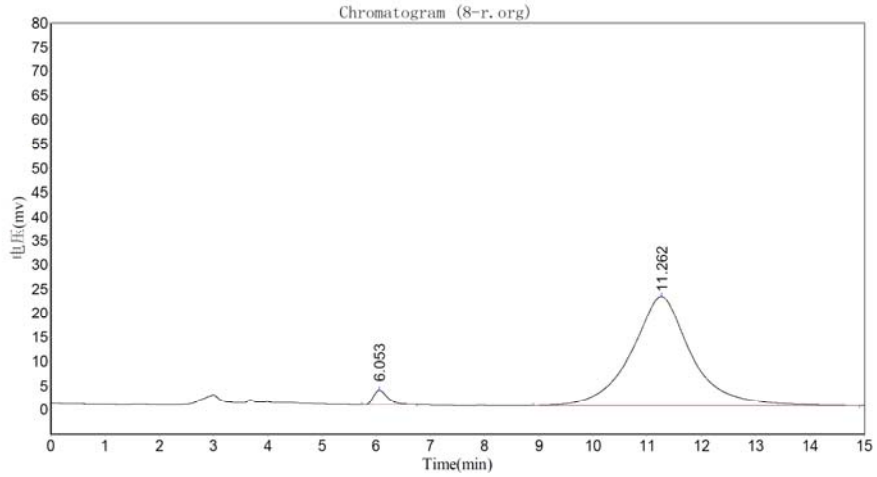
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.658	2837.276	55694.375	2.2102
2		10.798	43436.688	2464194.500	97.7898
Total			46273.964	2519888.875	100.0000



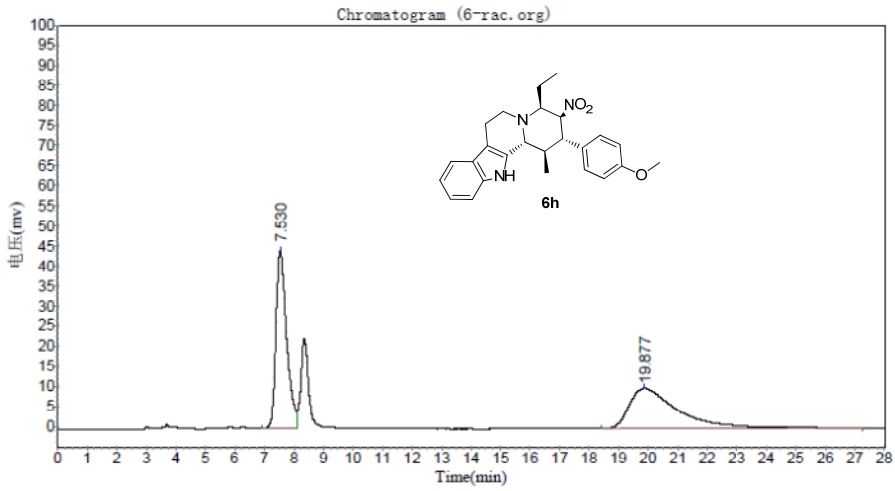
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.963	863316.375	15961526.000	50.6801
2		10.848	227710.328	15533145.000	49.3199
Total			1091026.703	31494671.000	100.0000



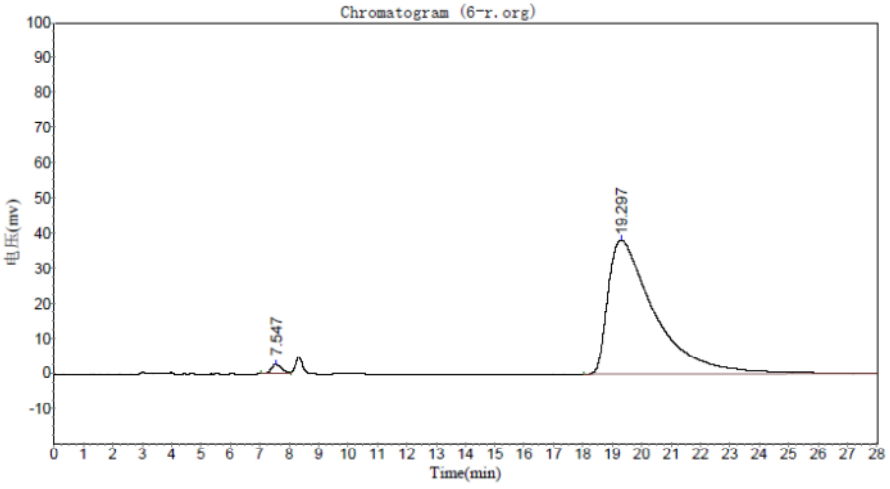
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.053	3049.134	55684.348	3.1082
2		11.262	22540.713	1735851.375	96.8918
Total			25589.847	1791535.723	100.0000



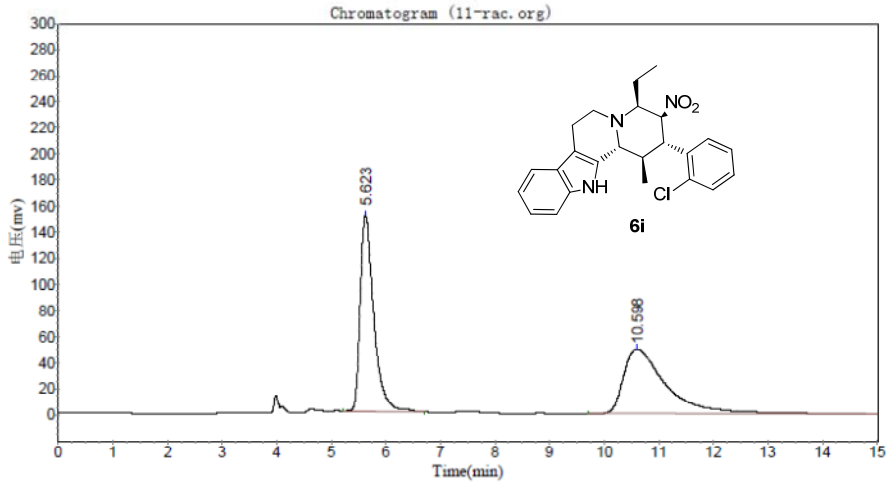
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.530	44304.313	1117800.250	49.4951
2		19.877	9930.500	1140606.500	50.5049
Total			54234.813	2258406.750	100.0000



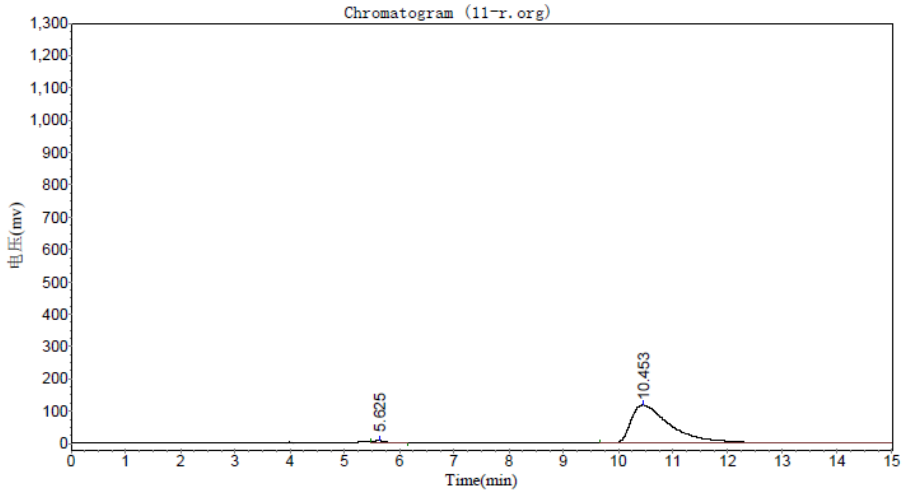
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.547	2631.492	66187.750	1.5306
2		19.297	38409.422	4258162.000	98.4694
Total			41040.914	4324349.750	100.0000



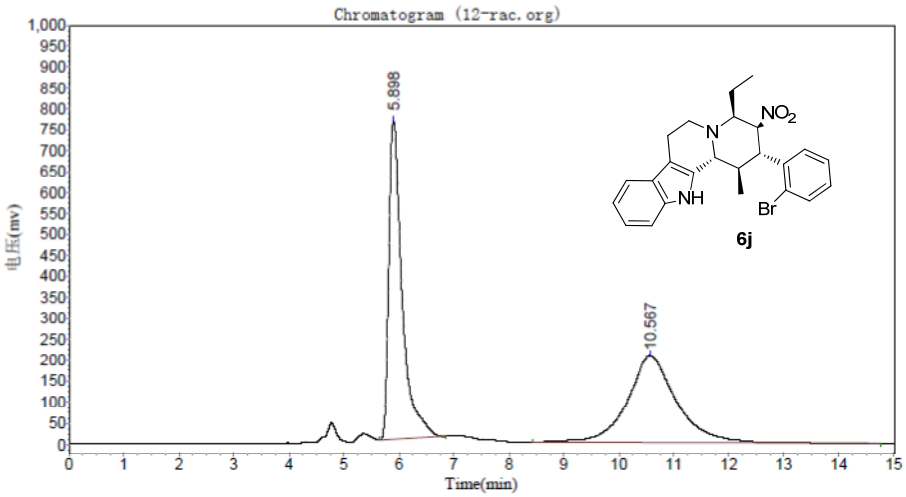
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.623	151192.172	2612889.500	49.3019
2		10.598	49949.570	2686882.500	50.6981
Total			201141.742	5299772.000	100.0000



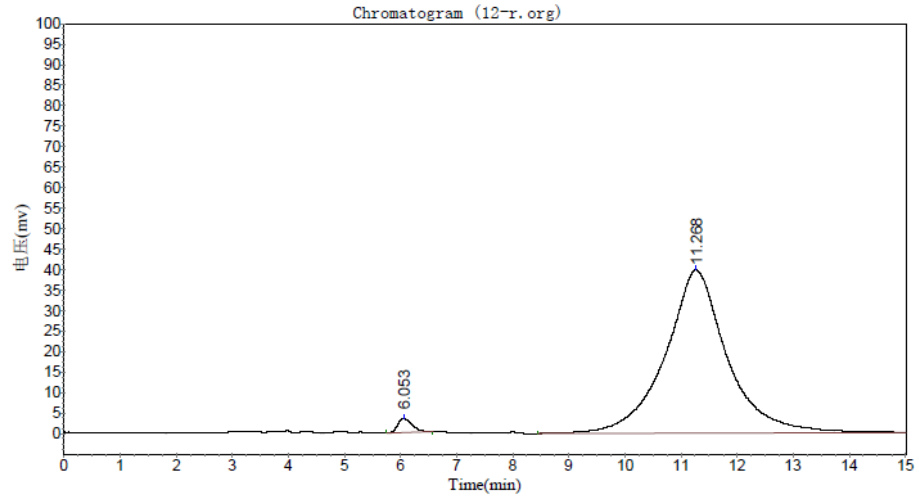
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.625	7004.274	131641.438	2.0892
2		10.453	118640.875	6169362.500	97.9108
Total			125645.149	6301003.938	100.0000



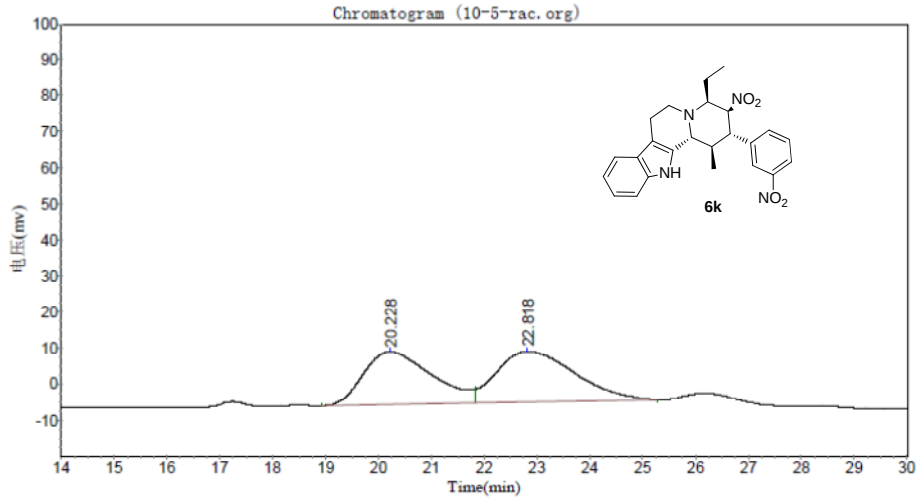
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.898	761007.125	12700940.000	49.7524
2		10.567	206638.625	12827363.000	50.2476
Total			967645.750	25528303.000	100.0000



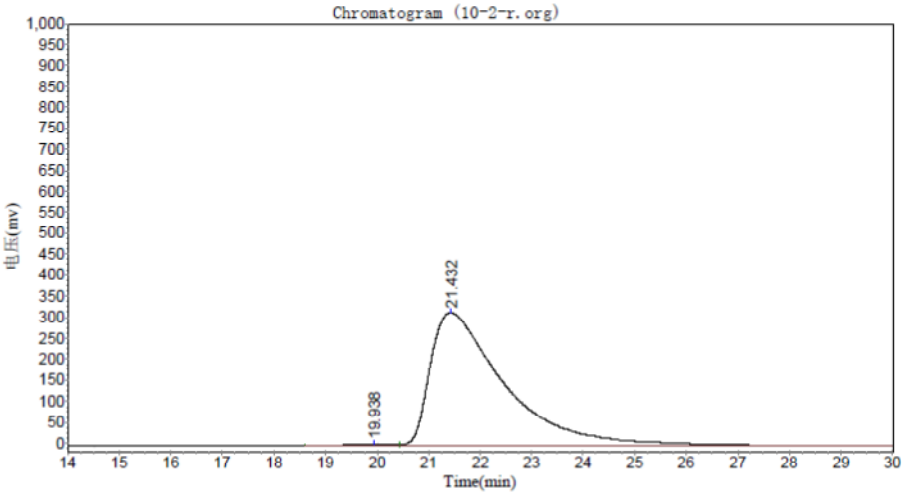
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		6.053	3427.636	59496.547	1.8995
2		11.268	39772.281	3072679.750	98.1005
Total			43199.918	3132176.297	100.0000



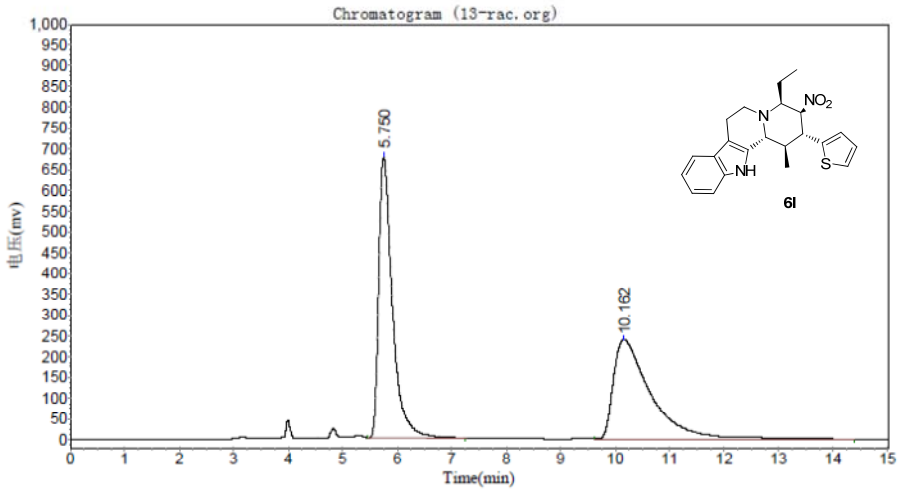
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		20.228	14582.641	1309304.750	47.6610
2		22.818	13959.867	1437817.750	52.3390
Total			28542.508	2747122.500	100.0000



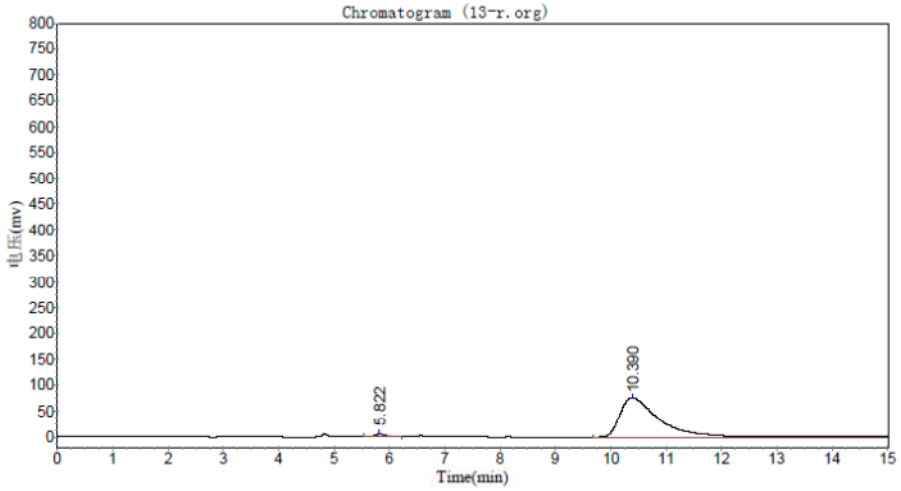
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		19.938	5264.724	342719.250	1.0431
2		21.432	317947.031	32514244.000	98.9569
Total			323211.755	32856963.250	100.0000



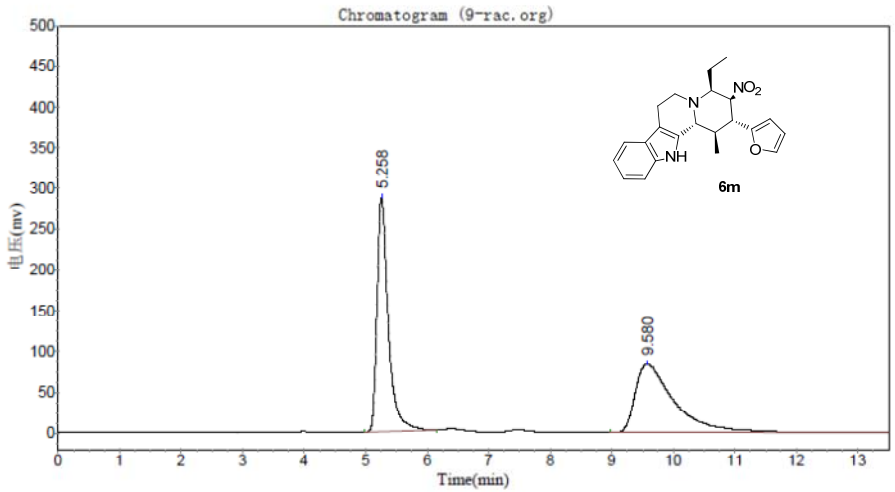
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.750	677332.125	11835445.000	50.8635
2		10.162	239888.500	11433610.000	49.1365
Total			917220.625	23269055.000	100.0000



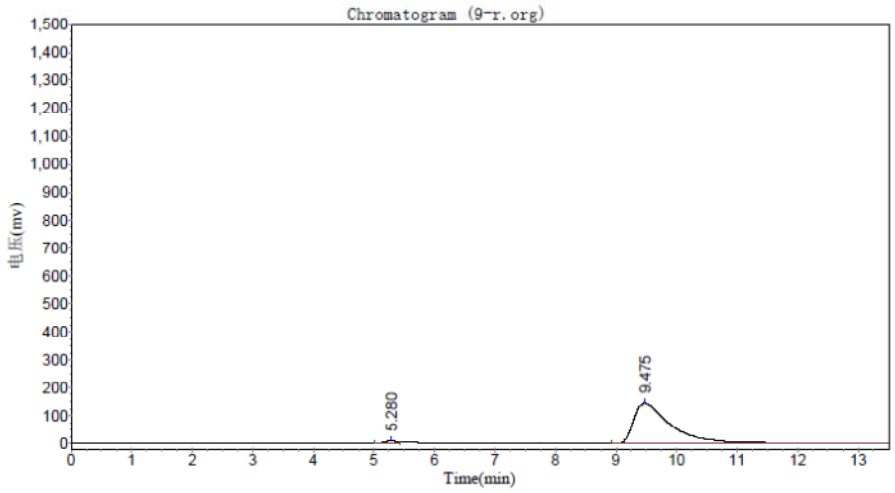
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.822	4729.024	75380.047	1.9571
2		10.390	75349.828	3776189.000	98.0429
Total			80078.853	3851569.047	100.0000



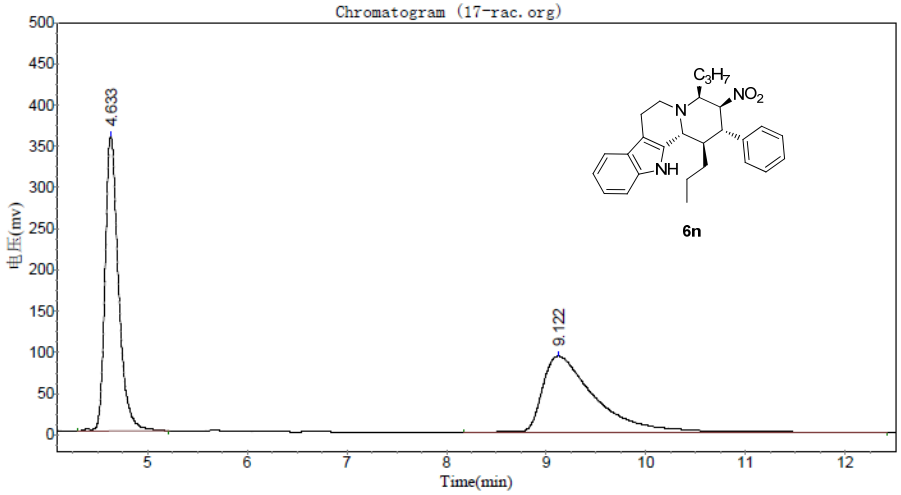
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.258	286134.906	3649790.000	49.6636
2		9.580	82760.602	3699237.500	50.3364
Total			368895.508	7349027.500	100.0000



Results

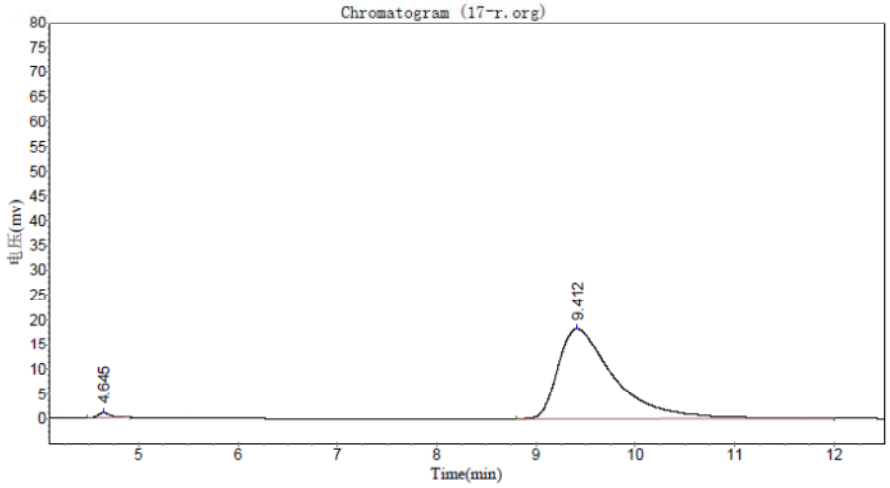
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.280	10473.578	127499.531	1.9914
2		9.475	143229.469	6275135.000	98.0086
Total			153703.047	6402634.531	100.0000



6n

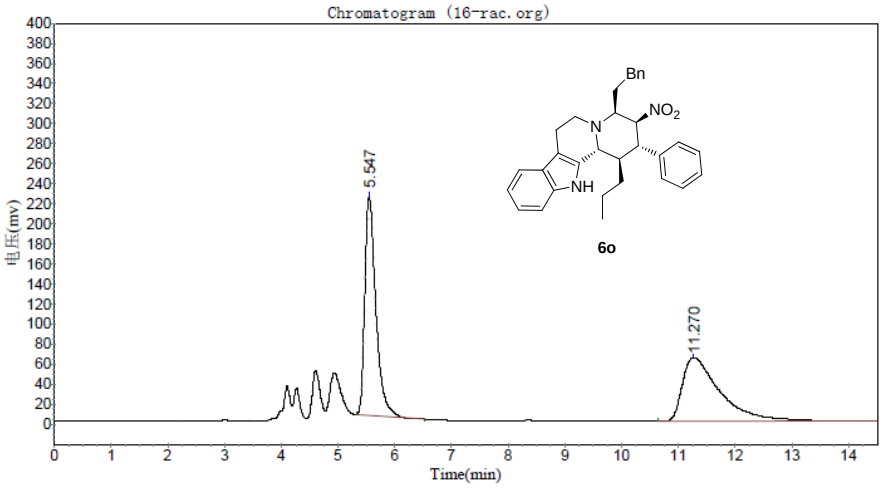
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.633	357931.563	3345966.750	49.7023
2		9.122	92393.180	3386052.250	50.2977
Total			450324.742	6732019.000	100.0000



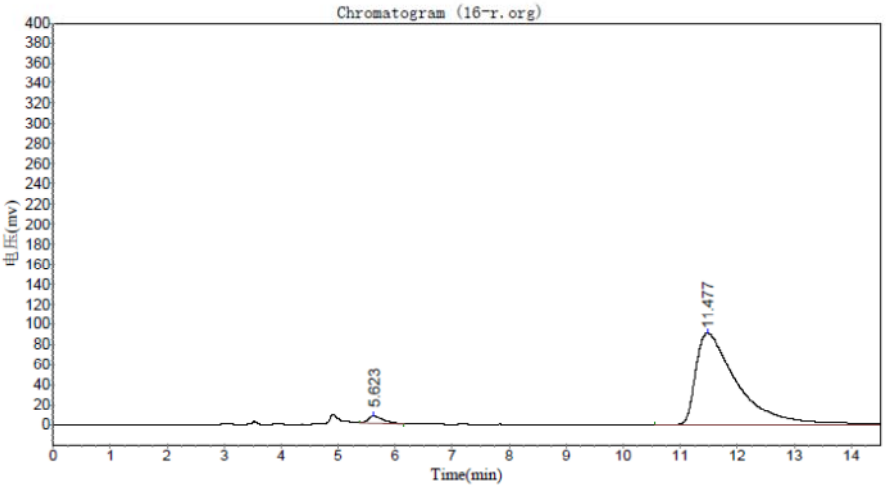
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.645	902.938	8280.750	1.1487
2		9.412	18132.932	712604.063	98.8513
Total			19035.870	720884.813	100.0000



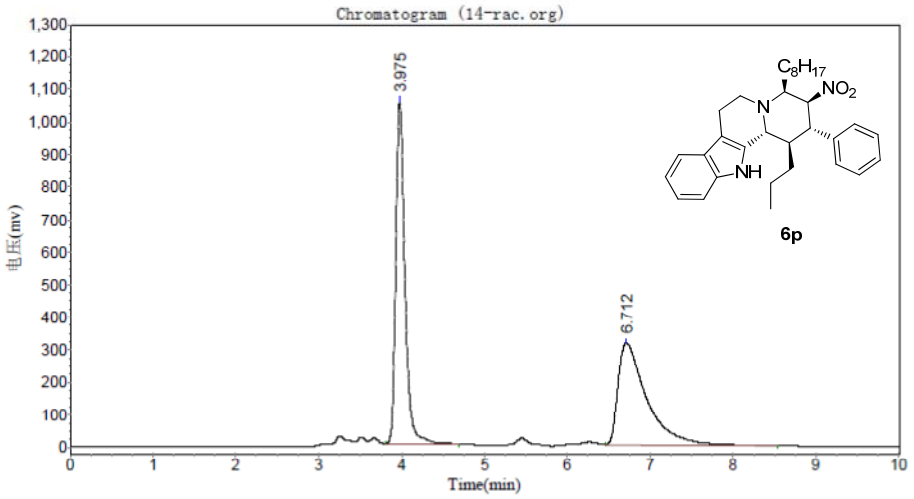
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.547	218308.500	2945389.250	50.2485
2		11.270	63200.637	2916253.000	49.7515
Total			281509.137	5861642.250	100.0000



Results

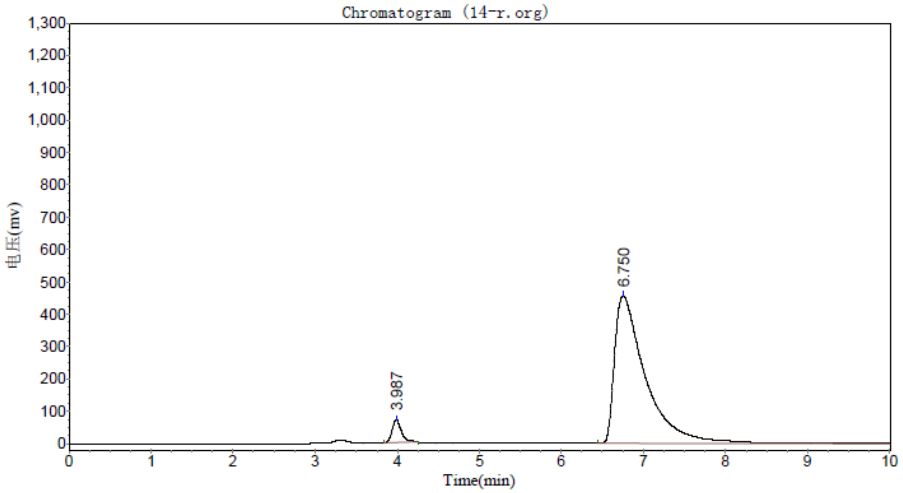
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		5.623	7454.828	127117.148	2.6824
2		11.477	91077.336	4611744.000	97.3176
Total			98532.164	4738861.148	100.0000



6p

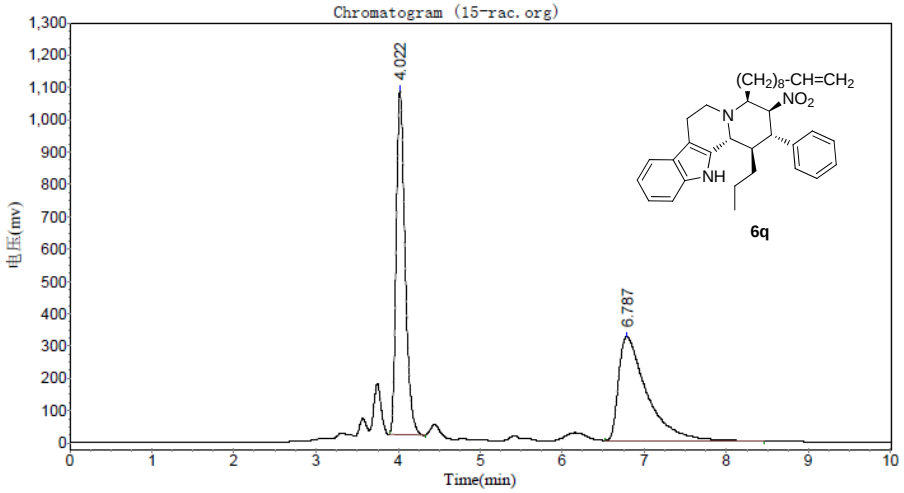
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.975	1052811.875	8029675.500	51.3764
2		6.712	314577.219	7599441.500	48.6236
Total			1367389.094	15629117.000	100.0000



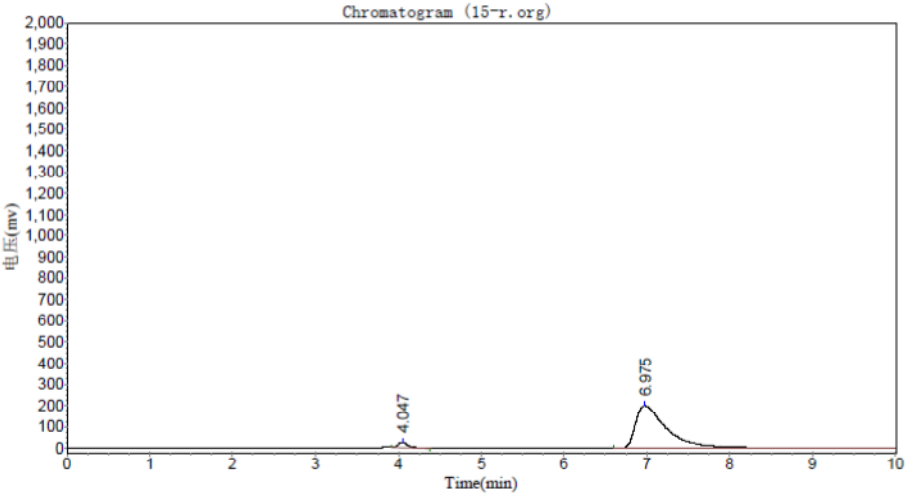
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		3.987	70511.430	551150.750	4.3533
2		6.750	456168.688	12109508.000	95.6467
Total			526680.117	12660658.750	100.0000



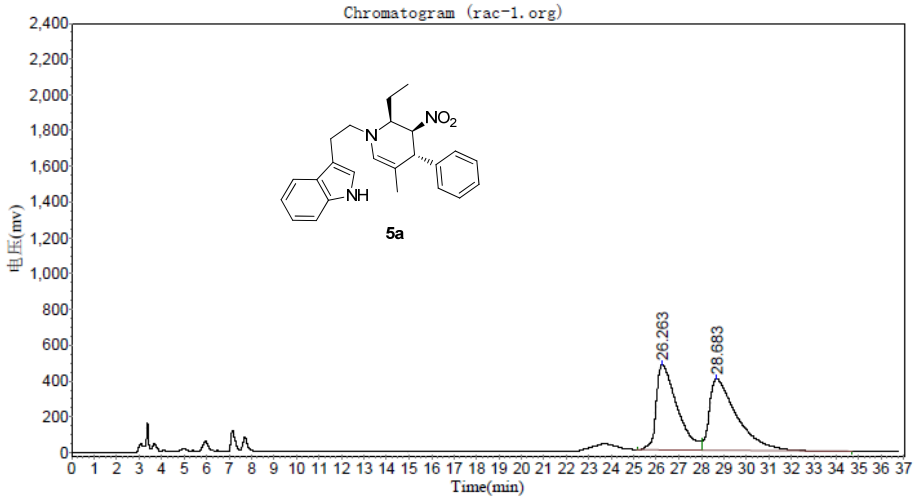
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.022	1065608.625	7995031.500	50.4292
2		6.787	324515.844	7858929.000	49.5708
Total			1390124.469	15853960.500	100.0000



Results

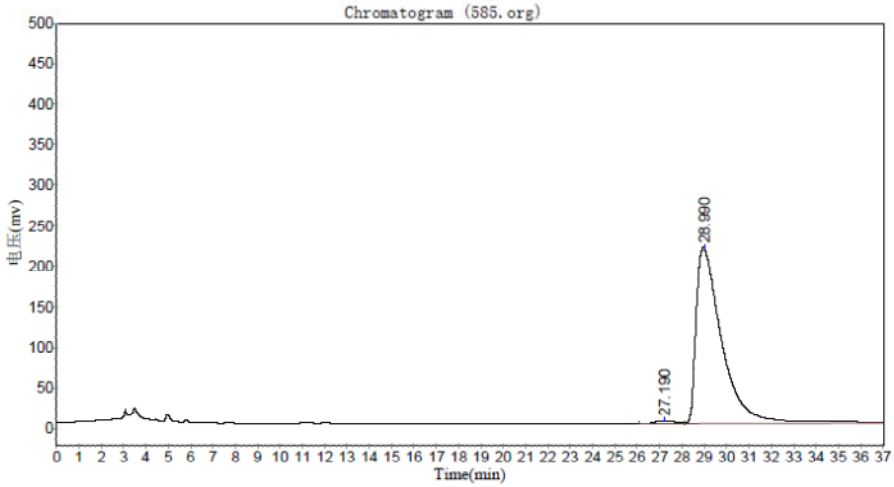
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		4.047	23626.779	184095.359	3.3175
2		6.975	198322.906	5365181.000	96.6825
Total			221949.686	5549276.359	100.0000



5a

Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		26.263	476215.500	30669642.000	47.5966
2		28.683	397463.281	33767008.000	52.4034
Total			873678.781	64436650.000	100.0000



Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		27.190	3834.935	235334.266	1.3266
2		28.990	216363.797	17503946.000	98.6734
Total			220198.731	17739280.266	100.0000

9. X-ray Crystallography Data of 6c

Crystals of **6c** were grown from PE:EA=5:1 to give white crystals that were submitted to X-Ray crystallography

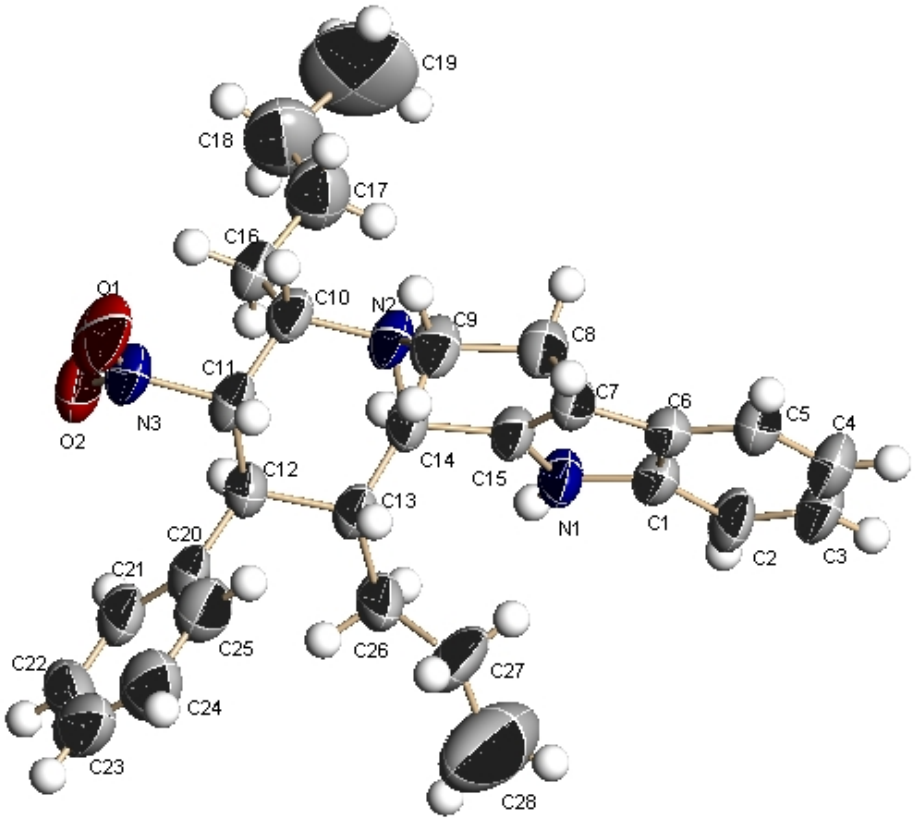


Table 1. Crystal data and structure refinement for a30711a.

Identification code	a30711a
Empirical formula	C56 H70 N6 O4
Formula weight	891.18
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P 21

Unit cell dimensions	a = 9.784(5) A alpha = 90 deg. b = 8.872(4) A beta = 90.000(7) deg. c = 14.497(7) A gamma = 90 deg.
Volume	1258.4(10) A^3
Z, Calculated density	1, 1.176 Mg/m^3
Absorption coefficient	0.074 mm^-1
F(000)	480
Crystal size	0.220 x 0.060 x 0.020 mm
Theta range for data collection	1.405 to 25.994 deg.
Limiting indices	-10<=h<=12, -10<=k<=10, -17<=l<=15
Reflections collected / unique	5689 / 4523 [R(int) = 0.0770]
Completeness to theta = 25.242	99.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.000 and 0.223
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4523 / 16 / 304
Goodness-of-fit on F^2	0.854
Final R indices [I>2sigma(I)]	R1 = 0.0629, wR2 = 0.1604
R indices (all data)	R1 = 0.1097, wR2 = 0.1794
Absolute structure parameter	0.5(10)
Extinction coefficient	0.017(5)
Largest diff. peak and hole	0.323 and -0.180 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for a30711a.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
N(1)	4260(5)	8487(6)	4906(3)	66(1)
N(2)	4091(4)	10215(5)	2623(3)	60(1)
N(3)	3767(6)	8459(7)	257(3)	77(2)
O(1)	3302(5)	9347(7)	-295(3)	113(2)
O(2)	4532(5)	7446(6)	62(3)	96(2)
C(1)	3843(6)	9253(7)	5690(3)	67(2)
C(2)	3936(6)	8841(8)	6612(3)	78(2)
C(3)	3388(7)	9835(10)	7238(4)	93(2)
C(4)	2743(7)	11141(10)	6977(4)	91(2)
C(5)	2647(6)	11541(7)	6069(4)	75(2)
C(6)	3198(5)	10587(6)	5406(3)	60(1)
C(7)	3234(5)	10582(6)	4403(3)	60(1)
C(8)	2754(6)	11740(7)	3729(3)	68(2)
C(9)	2880(6)	11134(7)	2756(4)	71(2)
C(10)	4413(6)	9908(7)	1648(3)	63(2)
C(11)	3410(6)	8738(7)	1263(3)	66(2)
C(12)	3356(5)	7261(6)	1797(3)	60(1)
C(13)	3016(6)	7669(7)	2825(3)	61(1)
C(14)	4064(6)	8805(6)	3171(3)	58(1)
C(15)	3874(5)	9311(6)	4144(3)	56(1)
C(16)	5893(6)	9513(8)	1515(3)	73(2)
C(17)	6851(7)	10589(11)	1976(5)	106(2)
C(18)	8292(9)	10443(15)	1766(7)	153(4)
C(19)	9161(10)	11460(20)	2376(9)	231(7)
C(20)	2330(6)	6175(7)	1391(3)	68(2)
C(21)	2768(7)	4782(8)	1072(3)	79(2)
C(22)	1825(10)	3788(8)	706(4)	98(2)
C(23)	473(10)	4165(13)	647(5)	110(3)
C(24)	51(8)	5509(13)	968(5)	105(2)
C(25)	958(7)	6524(9)	1337(4)	83(2)
C(26)	2848(7)	6300(7)	3438(4)	85(2)
C(27)	1598(10)	6504(10)	4103(5)	148(4)
C(28)	1320(18)	5291(15)	4746(10)	262(8)

Table 3. Bond lengths [A] and angles [deg] for a30711a.

N(1)-C(15)	1.378(6)
N(1)-C(1)	1.386(7)
N(1)-H(1)	0.88(5)
N(2)-C(9)	1.451(7)
N(2)-C(10)	1.474(6)
N(2)-C(14)	1.482(7)
N(3)-O(2)	1.204(7)
N(3)-O(1)	1.212(7)
N(3)-C(11)	1.519(7)
C(1)-C(2)	1.388(7)
C(1)-C(6)	1.404(8)
C(2)-C(3)	1.374(9)
C(2)-H(2)	0.9300
C(3)-C(4)	1.372(10)
C(3)-H(3)	0.9300
C(4)-C(5)	1.367(8)
C(4)-H(4)	0.9300
C(5)-C(6)	1.390(8)
C(5)-H(5)	0.9300
C(6)-C(7)	1.454(7)
C(7)-C(15)	1.344(8)
C(7)-C(8)	1.493(8)
C(8)-C(9)	1.515(7)
C(8)-H(8A)	0.9700
C(8)-H(8B)	0.9700
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-C(16)	1.502(7)
C(10)-C(11)	1.533(8)
C(10)-H(10)	0.9800
C(11)-C(12)	1.523(8)
C(11)-H(11)	0.9800
C(12)-C(20)	1.511(8)
C(12)-C(13)	1.570(6)
C(12)-H(12)	0.9800
C(13)-C(26)	1.514(8)
C(13)-C(14)	1.522(8)
C(13)-H(13)	0.9800
C(14)-C(15)	1.492(7)

C(14)-H(14)	0.9800
C(16)-C(17)	1.495(9)
C(16)-H(16A)	0.9700
C(16)-H(16B)	0.9700
C(17)-C(18)	1.449(10)
C(17)-H(17A)	0.9700
C(17)-H(17B)	0.9700
C(18)-C(19)	1.521(17)
C(18)-H(18A)	0.9700
C(18)-H(18B)	0.9700
C(19)-H(19A)	0.9600
C(19)-H(19B)	0.9600
C(19)-H(19C)	0.9600
C(20)-C(25)	1.379(8)
C(20)-C(21)	1.387(9)
C(21)-C(22)	1.382(9)
C(21)-H(21)	0.9300
C(22)-C(23)	1.367(10)
C(22)-H(22)	0.9300
C(23)-C(24)	1.345(13)
C(23)-H(23)	0.9300
C(24)-C(25)	1.372(10)
C(24)-H(24)	0.9300
C(25)-H(25)	0.9300
C(26)-C(27)	1.568(8)
C(26)-H(26A)	0.9700
C(26)-H(26B)	0.9700
C(27)-C(28)	1.450(10)
C(27)-H(27A)	0.9700
C(27)-H(27B)	0.9700
C(28)-H(28A)	0.9600
C(28)-H(28B)	0.9600
C(28)-H(28C)	0.9600

C(15)-N(1)-C(1)	108.5(5)
C(15)-N(1)-H(1)	123(4)
C(1)-N(1)-H(1)	128(4)
C(9)-N(2)-C(10)	114.0(4)
C(9)-N(2)-C(14)	112.9(4)
C(10)-N(2)-C(14)	111.2(4)
O(2)-N(3)-O(1)	124.3(5)
O(2)-N(3)-C(11)	119.3(5)
O(1)-N(3)-C(11)	116.2(6)
N(1)-C(1)-C(2)	129.9(6)

N(1)-C(1)-C(6)	107.8(4)
C(2)-C(1)-C(6)	122.3(6)
C(3)-C(2)-C(1)	116.2(6)
C(3)-C(2)-H(2)	121.9
C(1)-C(2)-H(2)	121.9
C(4)-C(3)-C(2)	122.6(6)
C(4)-C(3)-H(3)	118.7
C(2)-C(3)-H(3)	118.7
C(5)-C(4)-C(3)	121.1(6)
C(5)-C(4)-H(4)	119.5
C(3)-C(4)-H(4)	119.5
C(4)-C(5)-C(6)	118.8(6)
C(4)-C(5)-H(5)	120.6
C(6)-C(5)-H(5)	120.6
C(5)-C(6)-C(1)	119.0(5)
C(5)-C(6)-C(7)	134.7(6)
C(1)-C(6)-C(7)	106.3(5)
C(15)-C(7)-C(6)	107.0(5)
C(15)-C(7)-C(8)	122.8(4)
C(6)-C(7)-C(8)	130.1(5)
C(7)-C(8)-C(9)	109.9(5)
C(7)-C(8)-H(8A)	109.7
C(9)-C(8)-H(8A)	109.7
C(7)-C(8)-H(8B)	109.7
C(9)-C(8)-H(8B)	109.7
H(8A)-C(8)-H(8B)	108.2
N(2)-C(9)-C(8)	113.0(4)
N(2)-C(9)-H(9A)	109.0
C(8)-C(9)-H(9A)	109.0
N(2)-C(9)-H(9B)	109.0
C(8)-C(9)-H(9B)	109.0
H(9A)-C(9)-H(9B)	107.8
N(2)-C(10)-C(16)	111.8(4)
N(2)-C(10)-C(11)	109.7(4)
C(16)-C(10)-C(11)	114.4(5)
N(2)-C(10)-H(10)	106.8
C(16)-C(10)-H(10)	106.8
C(11)-C(10)-H(10)	106.8
N(3)-C(11)-C(12)	110.8(5)
N(3)-C(11)-C(10)	108.2(4)
C(12)-C(11)-C(10)	114.8(4)
N(3)-C(11)-H(11)	107.6
C(12)-C(11)-H(11)	107.6
C(10)-C(11)-H(11)	107.6

C(20)-C(12)-C(11)	112.0(4)
C(20)-C(12)-C(13)	112.1(4)
C(11)-C(12)-C(13)	106.9(4)
C(20)-C(12)-H(12)	108.6
C(11)-C(12)-H(12)	108.6
C(13)-C(12)-H(12)	108.6
C(26)-C(13)-C(14)	114.3(4)
C(26)-C(13)-C(12)	113.3(5)
C(14)-C(13)-C(12)	108.8(4)
C(26)-C(13)-H(13)	106.6
C(14)-C(13)-H(13)	106.6
C(12)-C(13)-H(13)	106.6
N(2)-C(14)-C(15)	104.8(4)
N(2)-C(14)-C(13)	113.2(4)
C(15)-C(14)-C(13)	115.2(4)
N(2)-C(14)-H(14)	107.8
C(15)-C(14)-H(14)	107.8
C(13)-C(14)-H(14)	107.8
C(7)-C(15)-N(1)	110.4(4)
C(7)-C(15)-C(14)	125.1(5)
N(1)-C(15)-C(14)	124.4(5)
C(17)-C(16)-C(10)	113.5(5)
C(17)-C(16)-H(16A)	108.9
C(10)-C(16)-H(16A)	108.9
C(17)-C(16)-H(16B)	108.9
C(10)-C(16)-H(16B)	108.9
H(16A)-C(16)-H(16B)	107.7
C(18)-C(17)-C(16)	117.3(7)
C(18)-C(17)-H(17A)	108.0
C(16)-C(17)-H(17A)	108.0
C(18)-C(17)-H(17B)	108.0
C(16)-C(17)-H(17B)	108.0
H(17A)-C(17)-H(17B)	107.2
C(17)-C(18)-C(19)	111.6(9)
C(17)-C(18)-H(18A)	109.3
C(19)-C(18)-H(18A)	109.3
C(17)-C(18)-H(18B)	109.3
C(19)-C(18)-H(18B)	109.3
H(18A)-C(18)-H(18B)	108.0
C(18)-C(19)-H(19A)	109.5
C(18)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
C(18)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5

H(19B)-C(19)-H(19C)	109.5
C(25)-C(20)-C(21)	118.8(6)
C(25)-C(20)-C(12)	121.7(6)
C(21)-C(20)-C(12)	119.5(6)
C(22)-C(21)-C(20)	119.4(7)
C(22)-C(21)-H(21)	120.3
C(20)-C(21)-H(21)	120.3
C(23)-C(22)-C(21)	120.9(8)
C(23)-C(22)-H(22)	119.6
C(21)-C(22)-H(22)	119.6
C(24)-C(23)-C(22)	119.5(8)
C(24)-C(23)-H(23)	120.3
C(22)-C(23)-H(23)	120.3
C(23)-C(24)-C(25)	121.2(8)
C(23)-C(24)-H(24)	119.4
C(25)-C(24)-H(24)	119.4
C(24)-C(25)-C(20)	120.2(7)
C(24)-C(25)-H(25)	119.9
C(20)-C(25)-H(25)	119.9
C(13)-C(26)-C(27)	110.6(6)
C(13)-C(26)-H(26A)	109.5
C(27)-C(26)-H(26A)	109.5
C(13)-C(26)-H(26B)	109.5
C(27)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	108.1
C(28)-C(27)-C(26)	117.1(9)
C(28)-C(27)-H(27A)	108.0
C(26)-C(27)-H(27A)	108.0
C(28)-C(27)-H(27B)	108.0
C(26)-C(27)-H(27B)	108.0
H(27A)-C(27)-H(27B)	107.3
C(27)-C(28)-H(28A)	109.5
C(27)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
C(27)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for a30711a.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
N(1)	92(3)	65(3)	41(2)	-1(2)	4(2)	6(3)
N(2)	78(3)	67(3)	34(2)	7(2)	5(2)	14(3)
N(3)	101(4)	95(4)	36(3)	0(3)	3(2)	-11(3)
O(1)	155(4)	145(5)	41(2)	21(3)	-7(2)	5(4)
O(2)	127(4)	102(4)	58(3)	-18(3)	26(2)	3(3)
C(1)	81(4)	78(5)	43(3)	0(3)	6(2)	-15(3)
C(2)	108(4)	93(5)	33(3)	5(3)	2(3)	-1(4)
C(3)	122(6)	120(7)	36(3)	3(4)	9(3)	-21(5)
C(4)	113(5)	111(6)	48(4)	-7(4)	22(3)	-11(5)
C(5)	92(4)	79(5)	53(3)	-9(3)	16(3)	-6(4)
C(6)	68(3)	64(4)	47(3)	-3(3)	3(2)	-12(3)
C(7)	72(3)	62(4)	45(3)	-3(3)	11(2)	0(3)
C(8)	82(4)	68(4)	55(3)	-1(3)	7(3)	8(3)
C(9)	95(4)	68(4)	51(3)	4(3)	4(3)	18(3)
C(10)	81(4)	66(4)	40(3)	8(3)	6(2)	2(3)
C(11)	78(4)	84(4)	35(3)	6(3)	5(2)	2(3)
C(12)	73(3)	66(4)	41(3)	-5(3)	5(2)	-2(3)
C(13)	78(4)	66(4)	38(3)	0(3)	8(2)	-1(3)
C(14)	78(3)	59(3)	36(2)	0(3)	4(2)	5(3)
C(15)	68(3)	65(4)	37(3)	1(3)	0(2)	2(3)
C(16)	84(4)	86(4)	50(3)	-1(3)	14(3)	-2(4)
C(17)	82(5)	142(7)	93(5)	-14(5)	5(4)	-12(5)
C(18)	113(7)	193(11)	154(8)	-35(8)	26(6)	-43(7)
C(19)	105(7)	320(20)	267(15)	10(14)	-39(8)	-72(11)
C(20)	93(5)	80(4)	31(3)	4(3)	7(3)	-10(4)
C(21)	112(5)	83(5)	42(3)	3(3)	8(3)	-4(4)
C(22)	163(8)	75(5)	57(4)	-8(4)	-6(4)	-23(6)
C(23)	122(7)	145(9)	64(4)	3(5)	-3(4)	-50(7)
C(24)	102(5)	138(7)	76(5)	-7(5)	-7(4)	-20(6)
C(25)	75(4)	104(6)	69(4)	-2(4)	4(3)	-1(4)
C(26)	140(5)	69(4)	45(3)	-2(3)	5(3)	-17(4)
C(27)	241(10)	126(7)	76(5)	15(5)	72(6)	-40(7)
C(28)	360(19)	165(12)	260(15)	13(13)	184(14)	-1(14)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for a30711a.

	x	y	z	U(eq)
H(1)	4680(60)	7620(70)	4870(50)	99
H(2)	4345	7944	6795	94
H(3)	3458	9614	7863	111
H(4)	2366	11763	7426	109
H(5)	2221	12436	5898	90
H(8A)	3298	12648	3792	82
H(8B)	1808	11995	3856	82
H(9A)	2077	10537	2614	85
H(9B)	2907	11975	2328	85
H(10)	4251	10847	1309	75
H(11)	2494	9184	1281	79
H(12)	4262	6792	1776	72
H(13)	2132	8188	2819	73
H(14)	4969	8337	3125	69
H(16A)	6053	8509	1757	88
H(16B)	6092	9493	860	88
H(17A)	6570	11604	1816	127
H(17B)	6739	10481	2637	127
H(18A)	8445	10704	1124	184
H(18B)	8570	9403	1852	184
H(19A)	8953	12492	2247	347
H(19B)	10111	11271	2254	347
H(19C)	8968	11244	3012	347
H(21)	3687	4519	1105	95
H(22)	2115	2851	496	118
H(23)	-151	3497	388	132
H(24)	-872	5756	940	127
H(25)	647	7450	1551	99
H(26A)	3673	6151	3798	102
H(26B)	2710	5412	3059	102
H(27A)	1740	7420	4456	177
H(27B)	788	6656	3728	177
H(28A)	805	4517	4441	392
H(28B)	804	5674	5258	392
H(28C)	2168	4879	4965	392

Table 6. Torsion angles [deg] for a30711a.

C(15)-N(1)-C(1)-C(2)	176.3(6)
C(15)-N(1)-C(1)-C(6)	-0.6(6)
N(1)-C(1)-C(2)-C(3)	-178.2(6)
C(6)-C(1)-C(2)-C(3)	-1.6(8)
C(1)-C(2)-C(3)-C(4)	2.1(10)
C(2)-C(3)-C(4)-C(5)	-1.9(11)
C(3)-C(4)-C(5)-C(6)	1.0(10)
C(4)-C(5)-C(6)-C(1)	-0.5(8)
C(4)-C(5)-C(6)-C(7)	176.2(6)
N(1)-C(1)-C(6)-C(5)	178.1(5)
C(2)-C(1)-C(6)-C(5)	0.9(8)
N(1)-C(1)-C(6)-C(7)	0.5(6)
C(2)-C(1)-C(6)-C(7)	-176.7(5)
C(5)-C(6)-C(7)-C(15)	-177.3(6)
C(1)-C(6)-C(7)-C(15)	-0.3(6)
C(5)-C(6)-C(7)-C(8)	5.9(10)
C(1)-C(6)-C(7)-C(8)	-177.2(5)
C(15)-C(7)-C(8)-C(9)	9.5(8)
C(6)-C(7)-C(8)-C(9)	-174.0(5)
C(10)-N(2)-C(9)-C(8)	-166.5(5)
C(14)-N(2)-C(9)-C(8)	65.4(6)
C(7)-C(8)-C(9)-N(2)	-37.3(7)
C(9)-N(2)-C(10)-C(16)	157.5(5)
C(14)-N(2)-C(10)-C(16)	-73.5(6)
C(9)-N(2)-C(10)-C(11)	-74.5(6)
C(14)-N(2)-C(10)-C(11)	54.5(5)
O(2)-N(3)-C(11)-C(12)	-34.0(7)
O(1)-N(3)-C(11)-C(12)	149.9(5)
O(2)-N(3)-C(11)-C(10)	92.7(6)
O(1)-N(3)-C(11)-C(10)	-83.4(6)
N(2)-C(10)-C(11)-N(3)	179.6(5)
C(16)-C(10)-C(11)-N(3)	-53.8(6)
N(2)-C(10)-C(11)-C(12)	-56.0(6)
C(16)-C(10)-C(11)-C(12)	70.6(5)
N(3)-C(11)-C(12)-C(20)	-58.1(6)
C(10)-C(11)-C(12)-C(20)	178.9(5)
N(3)-C(11)-C(12)-C(13)	178.7(4)
C(10)-C(11)-C(12)-C(13)	55.7(6)
C(20)-C(12)-C(13)-C(26)	53.7(6)

C(11)-C(12)-C(13)-C(26)	176.7(5)
C(20)-C(12)-C(13)-C(14)	-178.0(5)
C(11)-C(12)-C(13)-C(14)	-55.0(5)
C(9)-N(2)-C(14)-C(15)	-56.2(5)
C(10)-N(2)-C(14)-C(15)	174.3(4)
C(9)-N(2)-C(14)-C(13)	70.2(5)
C(10)-N(2)-C(14)-C(13)	-59.3(5)
C(26)-C(13)-C(14)-N(2)	-173.1(4)
C(12)-C(13)-C(14)-N(2)	59.2(6)
C(26)-C(13)-C(14)-C(15)	-52.5(7)
C(12)-C(13)-C(14)-C(15)	179.8(5)
C(6)-C(7)-C(15)-N(1)	-0.1(6)
C(8)-C(7)-C(15)-N(1)	177.1(5)
C(6)-C(7)-C(15)-C(14)	176.2(5)
C(8)-C(7)-C(15)-C(14)	-6.6(8)
C(1)-N(1)-C(15)-C(7)	0.4(6)
C(1)-N(1)-C(15)-C(14)	-175.9(5)
N(2)-C(14)-C(15)-C(7)	28.2(7)
C(13)-C(14)-C(15)-C(7)	-96.9(7)
N(2)-C(14)-C(15)-N(1)	-156.0(5)
C(13)-C(14)-C(15)-N(1)	78.9(7)
N(2)-C(10)-C(16)-C(17)	-48.8(7)
C(11)-C(10)-C(16)-C(17)	-174.3(5)
C(10)-C(16)-C(17)-C(18)	-170.4(8)
C(16)-C(17)-C(18)-C(19)	-172.2(10)
C(11)-C(12)-C(20)-C(25)	-60.8(6)
C(13)-C(12)-C(20)-C(25)	59.4(6)
C(11)-C(12)-C(20)-C(21)	119.9(5)
C(13)-C(12)-C(20)-C(21)	-119.9(5)
C(25)-C(20)-C(21)-C(22)	0.4(8)
C(12)-C(20)-C(21)-C(22)	179.7(5)
C(20)-C(21)-C(22)-C(23)	0.5(9)
C(21)-C(22)-C(23)-C(24)	-1.3(11)
C(22)-C(23)-C(24)-C(25)	1.3(12)
C(23)-C(24)-C(25)-C(20)	-0.4(10)
C(21)-C(20)-C(25)-C(24)	-0.4(8)
C(12)-C(20)-C(25)-C(24)	-179.7(5)
C(14)-C(13)-C(26)-C(27)	96.8(7)
C(12)-C(13)-C(26)-C(27)	-137.8(6)
C(13)-C(26)-C(27)-C(28)	-179.0(11)

Symmetry transformations used to generate equivalent atoms:

10. X-ray Crystallography Data of 7a

Crystals of **7a** were grown from PE:EA:THF=10:2:1 to give white crystals that were submitted to X-Ray crystallography

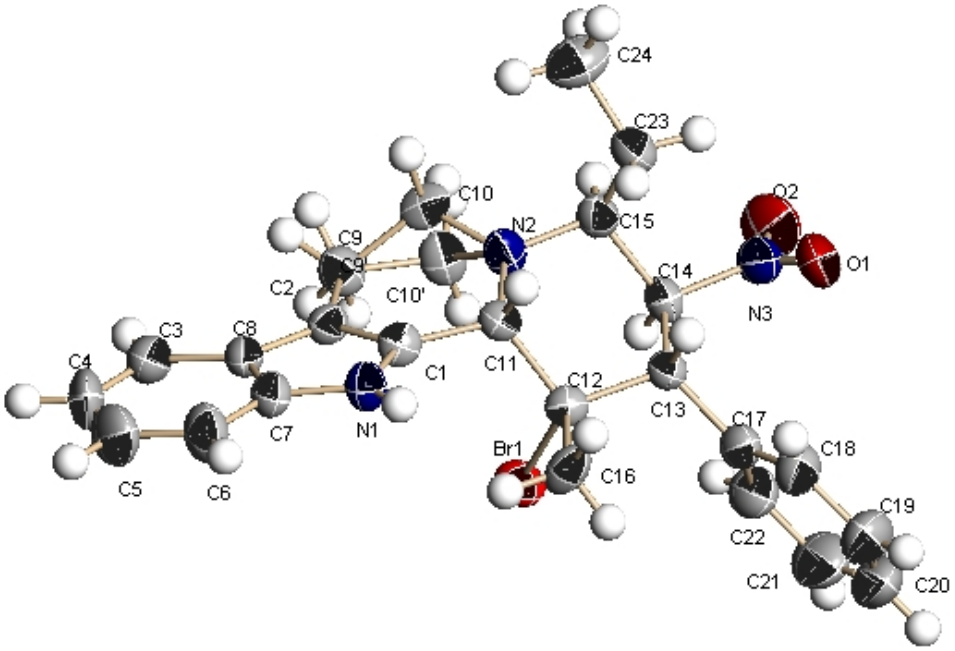


Table 1. Crystal data and structure refinement for a30916b.

Identification code	a30916b	
Empirical formula	C24 H26 Br N3 O2	
Formula weight	468.39	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 10.780(4) Å	α= 90°.
	b = 13.453(6) Å	β= 90°.
	c = 15.304(6) Å	γ = 90°.
Volume	2219.6(16) Å^3	

Z	4
Density (calculated)	1.402 Mg/m^3
Absorption coefficient	1.877 mm^{-1}
F(000)	968
Crystal size	0.160 x 0.160 x 0.140 mm^3
Theta range for data collection	2.016 to 25.592°.
Index ranges	-12<=h<=13, -16<=k<=16, -11<=l<=18
Reflections collected	9621
Independent reflections	4143 [R(int) = 0.0444]
Completeness to theta = 25.242°	99.9 %
Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4143 / 16 / 281
Goodness-of-fit on F^2	1.000
Final R indices [I>2sigma(I)]	R1 = 0.0335, wR2 = 0.0429
R indices (all data)	R1 = 0.0465, wR2 = 0.0439
Absolute structure parameter	0.004(5)
Extinction coefficient	n/a
Largest diff. peak and hole	0.639 and -0.416 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for a30916b. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3228(3)	7575(3)	10453(2)	38(1)
C(2)	3712(3)	6659(3)	10370(2)	38(1)
C(3)	3868(4)	5224(3)	11513(3)	62(1)
C(4)	3563(5)	4992(3)	12357(3)	83(2)
C(5)	2935(5)	5663(4)	12879(3)	84(2)
C(6)	2581(4)	6573(3)	12583(3)	66(1)
C(7)	2905(4)	6819(3)	11731(3)	46(1)
C(8)	3533(3)	6156(3)	11181(2)	44(1)
C(9)	4223(4)	6297(3)	9527(2)	51(1)
C(10)	3540(20)	6815(12)	8790(10)	43(6)
C(9')	4223(4)	6297(3)	9527(2)	51(1)
C(10')	4317(10)	7108(5)	8864(4)	54(3)
C(11)	3168(3)	8347(2)	9757(2)	35(1)
C(12)	3942(3)	9302(2)	9935(2)	38(1)
C(13)	3843(3)	10005(3)	9135(2)	36(1)
C(14)	4135(4)	9435(2)	8301(2)	42(1)
C(15)	3297(3)	8515(2)	8172(2)	43(1)
C(16)	3541(4)	9833(3)	10758(2)	52(1)
C(17)	4561(4)	10975(3)	9218(2)	47(1)
C(18)	3939(4)	11815(3)	9463(3)	61(1)
C(19)	4544(5)	12726(3)	9534(3)	83(2)
C(20)	5780(7)	12773(4)	9359(3)	97(2)
C(21)	6412(5)	11948(4)	9114(3)	86(2)
C(22)	5813(4)	11053(3)	9031(2)	62(1)
C(23)	1942(3)	8779(3)	7967(3)	55(1)
C(24)	1181(4)	7895(3)	7700(3)	73(1)
N(1)	2724(3)	7680(2)	11277(2)	44(1)
N(2)	3432(3)	7875(2)	8921(2)	47(1)
N(3)	4009(4)	10084(3)	7509(2)	57(1)
O(1)	3324(3)	10802(2)	7542(2)	74(1)
O(2)	4542(3)	9815(2)	6853(2)	84(1)
Br(1)	5687(1)	8906(1)	10125(1)	54(1)

Table 3. Bond lengths [Å] and angles [°] for a30916b.

C(1)-C(2)	1.343(4)
C(1)-N(1)	1.380(4)
C(1)-C(11)	1.490(4)
C(2)-C(8)	1.427(4)
C(2)-C(9)	1.485(5)
C(3)-C(4)	1.369(5)
C(3)-C(8)	1.400(5)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.382(6)
C(4)-H(4A)	0.9300
C(5)-C(6)	1.360(5)
C(5)-H(5A)	0.9300
C(6)-C(7)	1.390(5)
C(6)-H(6A)	0.9300
C(7)-N(1)	1.365(5)
C(7)-C(8)	1.401(5)
C(9)-C(10)	1.516(16)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-N(2)	1.444(16)
C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700
C(10')-N(2)	1.408(6)
C(10')-H(10C)	0.9700
C(10')-H(10D)	0.9700
C(11)-N(2)	1.457(4)
C(11)-C(12)	1.556(4)
C(11)-H(11A)	0.9800
C(12)-C(16)	1.512(4)
C(12)-C(13)	1.551(4)
C(12)-Br(1)	1.977(3)
C(13)-C(17)	1.522(5)
C(13)-C(14)	1.522(4)

C(13)-H(13A)	0.9800
C(14)-N(3)	1.500(4)
C(14)-C(15)	1.544(4)
C(14)-H(14A)	0.9800
C(15)-N(2)	1.442(4)
C(15)-C(23)	1.536(4)
C(15)-H(15A)	0.9800
C(16)-H(16A)	0.9600
C(16)-H(16B)	0.9600
C(16)-H(16C)	0.9600
C(17)-C(18)	1.366(5)
C(17)-C(22)	1.384(5)
C(18)-C(19)	1.393(5)
C(18)-H(18A)	0.9300
C(19)-C(20)	1.361(7)
C(19)-H(19A)	0.9300
C(20)-C(21)	1.355(6)
C(20)-H(20A)	0.9300
C(21)-C(22)	1.373(5)
C(21)-H(21A)	0.9300
C(22)-H(22A)	0.9300
C(23)-C(24)	1.502(5)
C(23)-H(23A)	0.9700
C(23)-H(23B)	0.9700
C(24)-H(24A)	0.9600
C(24)-H(24B)	0.9600
C(24)-H(24C)	0.9600
N(1)-H(1A)	0.8600
N(3)-O(2)	1.212(4)
N(3)-O(1)	1.217(4)

C(2)-C(1)-N(1)	109.5(3)
C(2)-C(1)-C(11)	126.1(4)
N(1)-C(1)-C(11)	124.3(3)
C(1)-C(2)-C(8)	107.5(3)
C(1)-C(2)-C(9)	121.8(3)
C(8)-C(2)-C(9)	130.5(3)
C(4)-C(3)-C(8)	118.9(4)

C(4)-C(3)-H(3A)	120.5
C(8)-C(3)-H(3A)	120.5
C(3)-C(4)-C(5)	121.0(4)
C(3)-C(4)-H(4A)	119.5
C(5)-C(4)-H(4A)	119.5
C(6)-C(5)-C(4)	122.1(5)
C(6)-C(5)-H(5A)	118.9
C(4)-C(5)-H(5A)	118.9
C(5)-C(6)-C(7)	117.2(5)
C(5)-C(6)-H(6A)	121.4
C(7)-C(6)-H(6A)	121.4
N(1)-C(7)-C(6)	130.1(4)
N(1)-C(7)-C(8)	107.7(4)
C(6)-C(7)-C(8)	122.2(4)
C(3)-C(8)-C(7)	118.5(4)
C(3)-C(8)-C(2)	134.9(4)
C(7)-C(8)-C(2)	106.6(3)
C(2)-C(9)-C(10)	108.4(7)
C(2)-C(9)-H(9A)	110.0
C(10)-C(9)-H(9A)	110.0
C(2)-C(9)-H(9B)	110.0
C(10)-C(9)-H(9B)	110.0
H(9A)-C(9)-H(9B)	108.4
N(2)-C(10)-C(9)	112.9(13)
N(2)-C(10)-H(10A)	109.0
C(9)-C(10)-H(10A)	109.0
N(2)-C(10)-H(10B)	109.0
C(9)-C(10)-H(10B)	109.0
H(10A)-C(10)-H(10B)	107.8
N(2)-C(10')-H(10C)	108.2
N(2)-C(10')-H(10D)	108.2
H(10C)-C(10')-H(10D)	107.3
N(2)-C(11)-C(1)	108.3(3)
N(2)-C(11)-C(12)	114.1(3)
C(1)-C(11)-C(12)	115.3(3)
N(2)-C(11)-H(11A)	106.1
C(1)-C(11)-H(11A)	106.1
C(12)-C(11)-H(11A)	106.1

C(16)-C(12)-C(13)	110.5(2)
C(16)-C(12)-C(11)	112.5(3)
C(13)-C(12)-C(11)	109.2(3)
C(16)-C(12)-Br(1)	106.1(2)
C(13)-C(12)-Br(1)	110.3(2)
C(11)-C(12)-Br(1)	108.28(19)
C(17)-C(13)-C(14)	113.4(3)
C(17)-C(13)-C(12)	115.0(3)
C(14)-C(13)-C(12)	109.9(3)
C(17)-C(13)-H(13A)	106.0
C(14)-C(13)-H(13A)	106.0
C(12)-C(13)-H(13A)	106.0
N(3)-C(14)-C(13)	111.5(3)
N(3)-C(14)-C(15)	108.0(3)
C(13)-C(14)-C(15)	113.0(3)
N(3)-C(14)-H(14A)	108.1
C(13)-C(14)-H(14A)	108.1
C(15)-C(14)-H(14A)	108.1
N(2)-C(15)-C(23)	113.4(3)
N(2)-C(15)-C(14)	108.5(3)
C(23)-C(15)-C(14)	113.4(3)
N(2)-C(15)-H(15A)	107.0
C(23)-C(15)-H(15A)	107.0
C(14)-C(15)-H(15A)	107.0
C(12)-C(16)-H(16A)	109.5
C(12)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(12)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(18)-C(17)-C(22)	118.2(4)
C(18)-C(17)-C(13)	118.8(4)
C(22)-C(17)-C(13)	123.0(4)
C(17)-C(18)-C(19)	121.3(4)
C(17)-C(18)-H(18A)	119.4
C(19)-C(18)-H(18A)	119.4
C(20)-C(19)-C(18)	119.0(5)
C(20)-C(19)-H(19A)	120.5

C(18)-C(19)-H(19A)	120.5
C(21)-C(20)-C(19)	120.6(5)
C(21)-C(20)-H(20A)	119.7
C(19)-C(20)-H(20A)	119.7
C(20)-C(21)-C(22)	120.5(5)
C(20)-C(21)-H(21A)	119.7
C(22)-C(21)-H(21A)	119.7
C(21)-C(22)-C(17)	120.4(5)
C(21)-C(22)-H(22A)	119.8
C(17)-C(22)-H(22A)	119.8
C(24)-C(23)-C(15)	113.1(3)
C(24)-C(23)-H(23A)	109.0
C(15)-C(23)-H(23A)	109.0
C(24)-C(23)-H(23B)	109.0
C(15)-C(23)-H(23B)	109.0
H(23A)-C(23)-H(23B)	107.8
C(23)-C(24)-H(24A)	109.5
C(23)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
C(23)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
C(7)-N(1)-C(1)	108.7(3)
C(7)-N(1)-H(1A)	125.6
C(1)-N(1)-H(1A)	125.6
C(10')-N(2)-C(15)	117.2(4)
C(15)-N(2)-C(10)	119.1(7)
C(10')-N(2)-C(11)	120.5(4)
C(15)-N(2)-C(11)	114.7(3)
C(10)-N(2)-C(11)	124.7(7)
O(2)-N(3)-O(1)	124.0(4)
O(2)-N(3)-C(14)	116.9(4)
O(1)-N(3)-C(14)	118.9(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for a30916b. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	36(2)	40(2)	39(2)	-3(2)	7(2)	4(2)
C(2)	41(2)	33(2)	41(3)	-3(2)	-1(2)	1(2)
C(3)	75(3)	45(3)	66(3)	4(3)	-16(3)	-5(2)
C(4)	123(5)	47(3)	78(4)	32(3)	-23(3)	-11(3)
C(5)	115(5)	82(4)	56(4)	29(3)	-2(3)	-25(4)
C(6)	84(4)	60(3)	53(3)	16(3)	7(3)	-3(3)
C(7)	50(3)	42(3)	47(3)	9(2)	-1(2)	-6(2)
C(8)	46(2)	37(2)	49(2)	7(3)	-11(2)	-4(2)
C(9)	54(2)	47(3)	52(2)	-2(2)	-1(2)	13(2)
C(10)	33(11)	56(12)	42(10)	-4(9)	20(9)	-13(9)
C(9')	54(2)	47(3)	52(2)	-2(2)	-1(2)	13(2)
C(10')	66(6)	51(4)	43(4)	-3(3)	11(4)	15(4)
C(11)	32(2)	41(2)	32(2)	-1(2)	3(2)	3(2)
C(12)	34(2)	40(2)	39(2)	-2(2)	2(2)	2(1)
C(13)	36(2)	37(2)	36(2)	2(2)	-2(2)	-1(2)
C(14)	47(2)	43(2)	36(2)	9(2)	1(2)	3(2)
C(15)	55(3)	40(2)	34(2)	-1(2)	0(2)	2(2)
C(16)	74(3)	38(3)	45(2)	-9(2)	15(2)	-12(2)
C(17)	59(3)	47(3)	36(2)	7(2)	-1(2)	-10(2)
C(18)	75(3)	46(3)	61(3)	3(2)	-3(2)	-5(2)
C(19)	117(4)	49(3)	82(4)	-3(3)	-20(4)	-8(3)
C(20)	133(6)	71(4)	86(4)	22(3)	-35(4)	-48(4)
C(21)	78(4)	91(4)	89(4)	19(4)	-7(3)	-38(4)
C(22)	61(3)	67(3)	60(3)	12(3)	0(2)	-16(3)
C(23)	55(3)	58(3)	51(3)	9(3)	-14(2)	-4(2)
C(24)	56(3)	72(3)	91(4)	-16(3)	3(3)	-9(2)
N(1)	50(2)	40(2)	41(2)	4(2)	8(2)	6(2)
N(2)	65(2)	38(2)	39(2)	-4(2)	0(2)	11(2)
N(3)	68(3)	59(3)	43(2)	9(2)	-3(2)	-17(2)
O(1)	98(3)	59(2)	66(2)	20(2)	-26(2)	-2(2)
O(2)	113(3)	98(2)	42(2)	9(2)	19(2)	-15(2)
Br(1)	42(1)	64(1)	55(1)	5(1)	-8(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for a30916b.

	x	y	z	U(eq)
H(3A)	4290	4769	11165	75
H(4A)	3781	4375	12582	99
H(5A)	2748	5485	13451	101
H(6A)	2142	7012	12936	79
H(9A)	4113	5584	9481	61
H(9B)	5103	6443	9493	61
H(10A)	3978	6694	8246	52
H(10B)	2717	6532	8736	52
H(9'1)	3693	5774	9301	61
H(9'2)	5039	6017	9626	61
H(10C)	5136	7403	8910	64
H(10D)	4255	6810	8288	64
H(11A)	2300	8561	9730	42
H(13A)	2967	10193	9094	44
H(14A)	4997	9207	8335	50
H(15A)	3621	8151	7666	51
H(16A)	2695	10046	10698	78
H(16B)	4063	10401	10852	78
H(16C)	3610	9389	11247	78
H(18A)	3095	11777	9584	73
H(19A)	4110	13293	9699	99
H(20A)	6195	13377	9409	116
H(21A)	7258	11989	9000	103
H(22A)	6252	10495	8848	75
H(23A)	1569	9080	8479	66
H(23B)	1924	9266	7500	66
H(24A)	343	8101	7589	110
H(24B)	1188	7411	8161	110
H(24C)	1525	7607	7179	110
H(1A)	2356	8203	11472	52

Table 6. Torsion angles [°] for a30916b.

N(1)-C(1)-C(2)-C(8)	-1.1(4)
C(11)-C(1)-C(2)-C(8)	-177.8(3)
N(1)-C(1)-C(2)-C(9)	174.5(3)
C(11)-C(1)-C(2)-C(9)	-2.1(6)
C(8)-C(3)-C(4)-C(5)	0.0(7)
C(3)-C(4)-C(5)-C(6)	0.6(8)
C(4)-C(5)-C(6)-C(7)	-1.5(7)
C(5)-C(6)-C(7)-N(1)	-177.0(4)
C(5)-C(6)-C(7)-C(8)	2.0(6)
C(4)-C(3)-C(8)-C(7)	0.5(6)
C(4)-C(3)-C(8)-C(2)	178.0(4)
N(1)-C(7)-C(8)-C(3)	177.7(3)
C(6)-C(7)-C(8)-C(3)	-1.5(6)
N(1)-C(7)-C(8)-C(2)	-0.5(4)
C(6)-C(7)-C(8)-C(2)	-179.7(4)
C(1)-C(2)-C(8)-C(3)	-176.8(4)
C(9)-C(2)-C(8)-C(3)	8.1(7)
C(1)-C(2)-C(8)-C(7)	1.0(4)
C(9)-C(2)-C(8)-C(7)	-174.2(4)
C(1)-C(2)-C(9)-C(10)	-29.0(11)
C(8)-C(2)-C(9)-C(10)	145.6(10)
C(2)-C(9)-C(10)-N(2)	46.8(16)
C(2)-C(1)-C(11)-N(2)	13.9(5)
N(1)-C(1)-C(11)-N(2)	-162.3(3)
C(2)-C(1)-C(11)-C(12)	-115.4(4)
N(1)-C(1)-C(11)-C(12)	68.4(4)
N(2)-C(11)-C(12)-C(16)	173.5(3)
C(1)-C(11)-C(12)-C(16)	-60.0(4)
N(2)-C(11)-C(12)-C(13)	50.5(3)
C(1)-C(11)-C(12)-C(13)	177.0(3)
N(2)-C(11)-C(12)-Br(1)	-69.6(3)
C(1)-C(11)-C(12)-Br(1)	56.9(3)
C(16)-C(12)-C(13)-C(17)	55.9(4)
C(11)-C(12)-C(13)-C(17)	-179.9(3)
Br(1)-C(12)-C(13)-C(17)	-61.0(3)
C(16)-C(12)-C(13)-C(14)	-174.8(3)

C(11)-C(12)-C(13)-C(14)	-50.6(3)
Br(1)-C(12)-C(13)-C(14)	68.3(3)
C(17)-C(13)-C(14)-N(3)	-51.6(4)
C(12)-C(13)-C(14)-N(3)	178.2(3)
C(17)-C(13)-C(14)-C(15)	-173.5(3)
C(12)-C(13)-C(14)-C(15)	56.3(4)
N(3)-C(14)-C(15)-N(2)	178.7(3)
C(13)-C(14)-C(15)-N(2)	-57.5(4)
N(3)-C(14)-C(15)-C(23)	-54.4(4)
C(13)-C(14)-C(15)-C(23)	69.4(4)
C(14)-C(13)-C(17)-C(18)	133.8(4)
C(12)-C(13)-C(17)-C(18)	-98.6(4)
C(14)-C(13)-C(17)-C(22)	-44.2(5)
C(12)-C(13)-C(17)-C(22)	83.4(4)
C(22)-C(17)-C(18)-C(19)	-0.8(6)
C(13)-C(17)-C(18)-C(19)	-178.9(4)
C(17)-C(18)-C(19)-C(20)	-0.3(7)
C(18)-C(19)-C(20)-C(21)	0.4(8)
C(19)-C(20)-C(21)-C(22)	0.5(8)
C(20)-C(21)-C(22)-C(17)	-1.6(7)
C(18)-C(17)-C(22)-C(21)	1.7(6)
C(13)-C(17)-C(22)-C(21)	179.7(4)
N(2)-C(15)-C(23)-C(24)	-64.9(4)
C(14)-C(15)-C(23)-C(24)	170.7(3)
C(6)-C(7)-N(1)-C(1)	178.9(4)
C(8)-C(7)-N(1)-C(1)	-0.2(4)
C(2)-C(1)-N(1)-C(7)	0.8(4)
C(11)-C(1)-N(1)-C(7)	177.6(3)
C(23)-C(15)-N(2)-C(10')	139.4(6)
C(14)-C(15)-N(2)-C(10')	-93.7(6)
C(23)-C(15)-N(2)-C(10)	96.0(11)
C(14)-C(15)-N(2)-C(10)	-137.0(11)
C(23)-C(15)-N(2)-C(11)	-70.9(4)
C(14)-C(15)-N(2)-C(11)	56.1(4)
C(9)-C(10)-N(2)-C(10')	56.7(13)
C(9)-C(10)-N(2)-C(15)	154.5(9)
C(9)-C(10)-N(2)-C(11)	-40.1(19)
C(1)-C(11)-N(2)-C(10')	-36.7(6)

C(12)-C(11)-N(2)-C(10')	93.3(6)
C(1)-C(11)-N(2)-C(15)	174.6(3)
C(12)-C(11)-N(2)-C(15)	-55.4(4)
C(1)-C(11)-N(2)-C(10)	8.6(12)
C(12)-C(11)-N(2)-C(10)	138.6(12)
C(13)-C(14)-N(3)-O(2)	159.5(4)
C(15)-C(14)-N(3)-O(2)	-75.8(4)
C(13)-C(14)-N(3)-O(1)	-25.2(5)
C(15)-C(14)-N(3)-O(1)	99.5(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for a30916b [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)