Electronic Supplementary Material (ESI) for ChemComm.

# Supplementary Information

# Stereoselective total synthesis of (±)-vindeburnol and (±)-16-*epi*-vindeburnol

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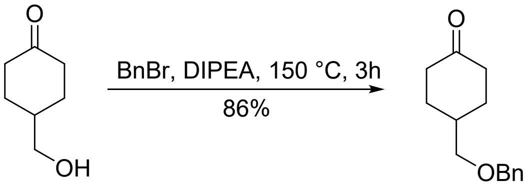
## 1. General Information

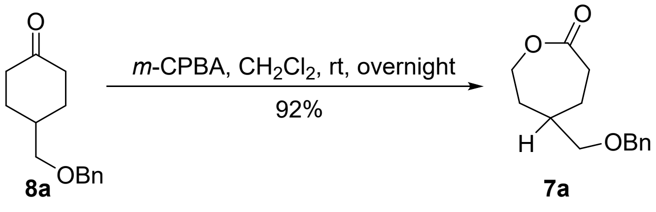
All commercially available reagents were used without further purification. Tetrahydrofuran was distilled from sodium/benzophenone ketyl. Dichloromethane was distilled from calcium hydride. DMSO was dried from the activated molecular sieve. Chromatography was conducted by using 200−300 mesh silica gel. All new compounds gave satisfactory spectroscopic analyses (IR, $^1$H NMR, $^{13}$C NMR, HRMS). NMR spectra were recorded on a 400 MHz NMR spectrometer. Chemical shifts are reported relative to the residue peaks of the solvent ($CDCl_3$: 7.26 ppm for $^1$H and 77.0 ppm for $^{13}$C) (DMSO-$d_6$: 2.50 ppm for $^1$H and 39.5 ppm for $^{13}$C). Coupling constants (J) are given in Hz and are uncorrected and multiplicities for coupled signals were denoted as: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br. = broad, apt. = apparent and dd = double doublet etc. Infrared (IR) spectra were recorded on a Perkin Elmer Spectrum Two FT-IR spectrometer. High-resolution mass spectra (HRMS) were recorded on a Bruker TOF Premier, by the ESI method. Melting points (m.p.) were recorded on an SRS-optic melting point apparatus. X-ray diffraction analysis was carried out by Dr. Meng Yang (Sichuan University). The data collections were done on a Bruker Xcalibur E using MoKα-radiation. HPLC analysis was performed with Daicel Chiralpak OD column (4.6 × 250 mm).

## 2. Experimental Procedures



**9** **8a**

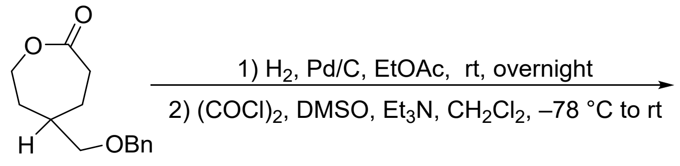
Commercially available 4-(hydroxymethyl)cyclohexan-1-one **9** (0.60 g, 4.68 mmol) was added to a solution of BnBr (0.67 mL, 5.61 mmol) in DIPEA (1.6 mL, 9.4 mmol). The resulting mixture was refluxed at 150 °C in sealed tube for 3 h and quenched by adding water at room temperature, extracted by EtOAc (3 × 30 mL). The combined organic layer was washed with brine, dried over anhydrous $Na_2SO_4$, filtered, and concentrated in vacuo. The residue was purified by silica gel flash column chromatography [petroleum ether/EtOAc = 10:1, $R_f$ (**8a**) = 0.4] to give **8a** (0.88 g, 86%) as a pale yellow oil. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.18 (m, 5H), 4.44 (s, 2H), 3.31 (d, *J* = 6.0 Hz, 2H), 2.34 – 2.21 (m, 4H), 2.09 – 1.95 (m, 3H), 1.43 – 1.32 (m, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 211.9, 138.4, 128.5, 127.7, 127.6, 74.3, 73.2, 40.5, 36.6, 29.6. IR (neat) 1711, 1452, 1245, 1170, 1099, 919, 737, 698, 503 cm$^{-1}$. HRMS (ESI) m/z calcd for $C_{14}H_{19}O_2$ $[M + H]^+$: 219.1380, found: 219.1386.



**8a** **7a**

To a stirred solution of compound **8a** (0.90 g, 4.12 mmol) in $CH_2Cl_2$ (20.0 mL) was added *m*-CPBA (0.92 g, 4.53 mmol). The reaction mixtures were stirred for overnight at room temperature. The saturated $K_2CO_3$

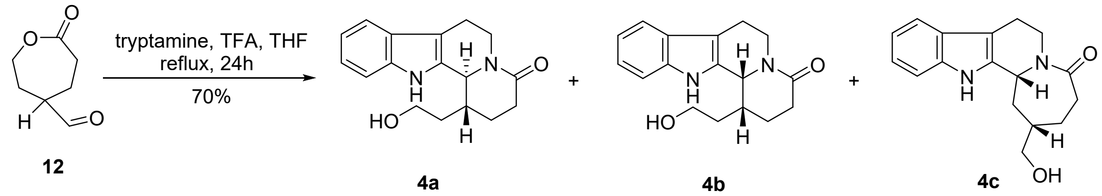
aqueous solution (50 mL) was added and extracted with $CH_2Cl_2$ (3 x 50 mL).Then the organic layer was concentrated in vacuo and the crude products were purified directly by column chromatography on silica gel [petroleum ether/EtOAc = 3:1, $R_f$ (**7a**) = 0.2] to afford the desired products **7a** (0.89 g, 92%) as a pale yellow oil. $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.20 (m, 5H), 4.43 (s, 2H), 4.27 – 4.20 (m, 1H), 4.14 – 4.07 (m, 1H), 3.31 – 3.22 (m, 2H), 2.67 – 2.50 (m, 2H), 2.05 – 1.84 (m, 3H), 1.54 – 1.43 (m, 1H), 1.38 – 1.27 (m, 1H). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 175.7, 138.1, 128.3, 127.6, 127.4, 74.2, 73.0, 67.8, 40.6, 32.8, 32.3, 25.7. IR (neat) 1723, 1475, 1392, 1169, 1102, 1070, 1008, 736, 698, 586 $cm^{-1}$. HRMS (ESI) m/z calcd for $C_{14}H_{18}NaO_3$ [M + Na]$^+$: 257.1148, found: 257.1153.



**7a** **12**

A stirred solution of **7a** (0.89 g, 3.80 mmol) in EtOAc (12.0 mL) was treated with 5% Pd/C (0.18 g). After stirring at room temperature under $H_2$ for overnight, the reaction mixture was filtrated through a pad of celite. The solid was washed with EtOAc (3 × 30 mL) and concentrated in vacuo to give the alcohol intermediate as a colorless oil, which was directly used in next step without further purification.

A round bottom flask equipped with stirring bar was charged with oxalyl chloride (0.60 mL, 7.60 mmol) in dry $CH_2Cl_2$ (15.0 mL) and cooled to -78 °C. Dry dimethylsulfoxide (1.11 mL, 15.6 mmol) was diluted into 10 mL dry $CH_2Cl_2$, which was added slowly for 10 minutes. After stirring for 30 min at -78 °C, the alcohol intermediate was dissolved in 5.0 mL dry $CH_2Cl_2$, which was added into the reaction mixture over a period of 10 minutes. After stirring for additional 30 min at -78 °C, triethylamine (2.80 mL) was added and the reaction allowed to warm to room temperature. The reaction mixture was diluted with 20 mL $H_2O$ and extracted with $CH_2Cl_2$ (3 x 30 mL). The combined organic phase was dried over $Na_2SO_4$ and concentrated in vacuum. The residue was purified directly by column chromatography on silica gel [petroleum ether/EtOAc = 3:1, $R_f$ (**12**) = 0.3] to afford the corresponding product **12** (0.49 g, 90%) as a pale yellow oil. $^1H$ NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 4.39 – 4.35 (m, 1H), 4.03 – 3.98 (m, 1H), 2.66 – 2.49 (m, 5H), 2.13 – 2.04 (m, 1H), 1.63 – 1.53 (m, 1H). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 199.4, 170.9, 72.4, 45.6, 28.8, 27.3, 25.3. IR (neat) 2933, 1706, 1260, 1168, 971, 914, 803, 733 $cm^{-1}$. HRMS (ESI) m/z calcd for $C_7H_{11}O_3$ [M + H]$^+$: 143.0703, found: 143.0707.



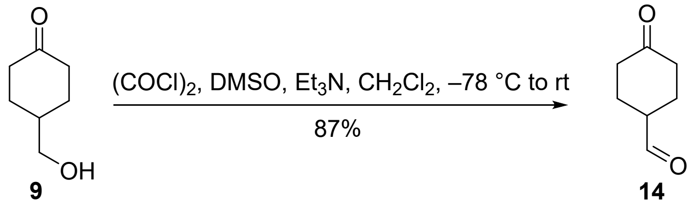
**12** **4a** **4b** **4c**

To a solution of **12** (0.47 g, 3.31 mmol) in THF (33.0 mL) was added tryptamine (0.69 g, 4.30 mmol) and TFA (0.30 mL). After refluxing for 24 h, water (10.0 mL) was added to the cooled reaction mixture and the pH was adjusted to 7.0 at 0 °C with saturated $Na_2CO_3$ solution. The layers were separated and the aqueous layer was extracted with $CH_2Cl_2$ (3 × 30 mL). The combined organic layers were washed with brine, dried over $Na_2SO_4$, filtered, and evaporated in vacuo. The residue was purified by silica gel column chromatography [$CH_2Cl_2$/Acetone = 2:1, $R_f$ (**4a**) = 0.5, $R_f$ (**4b**) = 0.3 , $R_f$ (**4c**) = 0.4] to afford **4a** (0.44 g, 47%) as a white solid, **4b** (0.11 g, 12%) as a white solid, **4c** (0.10 g,11%) as a white solid.

**4a**: mp = 189.8–192.5 ºC; $^1$H NMR (400 MHz, DMSO-$d_6$) δ 10.83 (s, 1H), 7.35 (dd, *J* = 12.4, 8.0 Hz, 2H), 7.05 (td, *J* = 8.0, 1.2 Hz, 1H), 6.96 (td, *J* = 8.0, 1.2 Hz, 1H), 4.74 – 4.70 (m, 3H), 3.70 – 3.60 (m, 2H), 2.88 (td, *J* = 12.0, 4 Hz, 1H), 2.82 – 2.75 (m, 1H), 2.60 – 2.53 (m, 2H), 2.39 – 2.30 (m, 1H), 2.12 – 2.05 (m, 1H), 1.94 – 1.85 (m, 1H), 1.78 – 1.70 (m, 1H), 1.66 – 1.56 (m, 2H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 169.0, 135.8, 135.1, 126.8, 120.9, 118.7, 117.5, 111.4, 108.4, 59.2, 58.9, 42.1, 34.4, 31.3, 28.8, 22.7, 20.5. IR (KBr) 3258, 2931, 2866, 1610, 1471, 1409, 1355, 1303, 1234, 1056, 1028, 736, 677, 511 cm$^{-1}$. HRMS (ESI) m/z calcd for $C_{17}H_{20}N_2NaO_2$ [M + Na]$^+$: 307.1417, found: 307.1424;

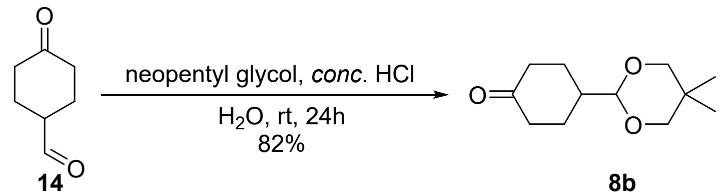
**4b**: mp = 203.9–204.8 ºC; $^1$H NMR (400 MHz, DMSO-$d_6$) δ 10.87 (s, 1H), 7.42 (d, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.06 (t, *J* = 8.0 Hz, 1H), 6.97 (t, *J* = 7.6 Hz, 1H), 4.94 – 4.91 (m, 2H), 4.31 (t, *J* = 4.8 Hz, 1H), 3.31 – 3.23 (m, 2H), 2.70 (td, *J* = 14.8, 3.2 Hz, 2H), 2.60 – 2.53 (m, 2H), 2.38 – 2.29 (m, 2H), 1.98 – 1.87 (m, 2H), 1.28 – 1.18 (m, 1H), 1.00 – 0.92 (m, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 168.3, 136.4, 132.8, 126.3, 120.9, 118.5, 117.6, 111.1, 108.8, 58.9, 57.7, 38.6, 32.3, 27.7, 27.3, 22.2, 20.7. IR (KBr) 3190, 2931, 2850, 1612, 1491, 1435, 1294, 1226, 1080, 1209, 935, 871, 748, 688 cm$^{-1}$. HRMS (ESI) m/z calcd for $C_{17}H_{20}N_2NaO_2$ [M + Na]$^+$: 307.1417, found: 307.1424;

**4c**: mp = 228.6–230.3 ºC; $^1$H NMR (400 MHz, DMSO-$d_6$) δ 10.99 (s, 1H), 7.41 (d, *J* = 7.6 Hz, 1H), 7.33 (d, *J* = 8 Hz, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 6.96 (t, *J* = 7.6 Hz, 1H), 5.11 (d, *J* = 9.6 Hz, 1H), 4.71 (dd, *J* = 12.8, 4.8 Hz, 1H), 4.61 (t, *J* = 5.6 Hz, 1H), 3.29 – 3.24 (m, 2H), 2.95 (t, *J* = 13.6 Hz, 1H), 2.85 (td, *J* = 12.4, 3.6 Hz, 1H), 2.77 (td, *J* = 16.0, 4 Hz, 1H), 2.58 – 2.52 (m, 1H), 2.36 (q, *J* = 6.8 Hz, 1H), 2.25 – 2.21 (m, 1H), 2.04 – 1.92 (m, 2H), 1.36 (q, *J* = 11.2 Hz, 1H), 1.13 (q, *J* = 13.2 Hz, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 174.5, 136.3, 134.2, 125.9, 120.9, 118.4, 117.7, 111.0, 107.7, 66.0, 52.5, 43.2, 36.8, 36.1, 34.8, 26.4, 20.6. IR (KBr) 3325, 2964, 2901, 1620, 1502, 1429, 1354, 1307, 1172, 1068, 1004, 933, 883, 852 cm$^{-1}$. HRMS (ESI) m/z calcd for $C_{17}H_{20}N_2NaO_2$ [M + Na]$^+$: 307.1417, found: 307.1425.

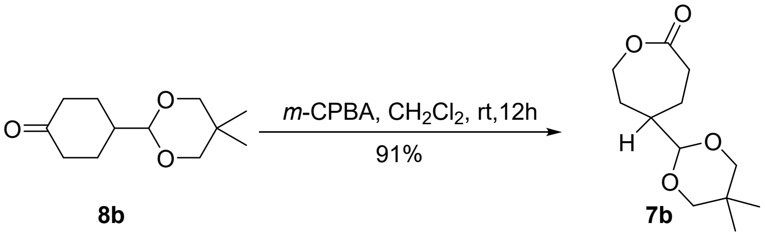


**9** **14**

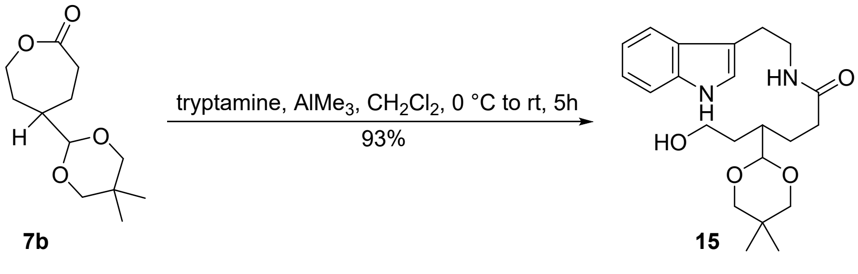
A round bottom flask equipped with stirring bar was charged with (1.30 mL, 15.60 mmol) oxalyl chloride in dry $CH_2Cl_2$ (25.0 mL) and cooled to -78 ºC. Dry dimethylsulfoxide (2.26 mL, 32.0 mmol) diluted with 10 mL dry $CH_2Cl_2$, was added slowly for 10 min. After stirring for another 30 min at -78 ºC , **9** (1.0 g, 7.80 mmol) dissolved in 15 mL dry $CH_2Cl_2$, was added over a period of 20 minutes. After stirring for additional 30 min at -78 ºC, trimethylamine (5.70 mL) was added and the reaction allowed to warm to room temperature. The reaction mixture was diluted with 30 mL $H_2O$ and extracted with $CH_2Cl_2$ (3 x 50 mL). The combined organic phases were dried over $Na_2SO_4$ and concentrated in vacuum to give the crude product. The residue was purified by silica gel flash column chromatography [petroleum ether/EtOAc = 3:1, $R_f$ (**14**) = 0.3] to give **14** (0.86 g, 87%) as a colorless oil. $^1$H NMR (400 MHz, $CDCl_3$) δ 9.71 (s, 1H), 2.67 – 2.59 (m, 1H), 2.45 – 2.28 (m, 4H), 2.21 – 2.13 (m, 2H), 1.97 – 1.87 (m, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 209.7, 202.5, 47.3, 39.5, 25.5. IR (neat) 2856, 1731, 1339, 1272, 1088, 905, 803, 701, 646 cm$^{-1}$. HRMS (ESI) m/z calcd for $C_7H_{11}O_2$ [M + H]$^+$: 127.0754, found: 127.0758.



To a stirred solution of compound **14** (0.87 g, 6.90 mmol) in $H_2O$ (14.0 mL) was added neopentyl glycol (0.72 g, 6.90 mmol) and *con*. HCl (4.0 mL). After at room temperature for 24 h, the precipitate was filtered, washed with ice-cold slightly alkaline water and $Et_2O$. The aqueous layers were extracted with $CH_2Cl_2$ (3 x 50 mL). The combined organic phases were dried over $Na_2SO_4$ and concentrated in vacuum to give the crude product. The solid was dried under vacuum to give the residue as a pale yellow solid. The residue was purified by silica gel flash column chromatography [petroleum ether/EtOAc = 8:1, $R_f$ (**8b**) = 0.4] to give **8b** (1.20 g, 82%) as a white soild. mp = 75.1–75.7 ºC; $^1$H NMR (400 MHz, $CDCl_3$) δ 4.29 (d, *J* = 4.8 Hz, 1H), 3.60 (t, *J* = 1.6 Hz, 1H), 3.57 (d, *J* = 1.6 Hz, 1H), 3.41 (s, 1H), 3.38 (s, 1H), 2.43 – 2.35 (m, 2H), 2.30 (td, *J* = 13.2, 6.0 Hz, 2H), 2.15 – 2.10 (m, 2H), 2.01 – 1.92 (m, 1H), 1.65 – 1.54 (m, 2H), 1.15 (s, 3H), 0.70 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 211.9, 103.7, 77.3, 40.6, 40.4, 30.3, 27.0, 22.9, 21.9. IR (neat) 2953, 2845, 1711, 1649, 1394, 1384, 1105, 925, 828, 793, 757, 656 cm$^{-1}$. HRMS (ESI) m/z calcd for $C_{12}H_{21}O_3$ [M + H]$^+$: 213.1485, found: 213.1489.



To a stirred solution of compound **8b** (0.50 g, 2.36 mmol) in $CH_2Cl_2$ (12.0 mL) was added *m*-CPBA (0.57 g, 2.83 mmol). After the reaction mixtures were stirred for 12 h at room temperature, the saturated $K_2CO_3$ solution (30 mL) was added and the suspension was extracted with $CH_2Cl_2$ (3 x 50 mL).Then the combine organic layers were concentrated in vacuo and the crude products were purified directly by column chromatography on silica gel (petroleum ether/EtOAc = 3:1) to afford the desired product **7b** (0.49 g, 91%) as a white soild. mp = 122.9–125.6 ºC; $^1$H NMR (400 MHz, $CDCl_3$) δ 4.32 (dd, *J* = 13.2, 6 Hz, 1H), 4.23 (d, *J* = 4.4 Hz, 1H), 4.12 (t, *J* = 10.4 Hz, 1H), 3.57 (d, *J* = 10.8 Hz, 2H), 3.37 (d, *J* = 10.8 Hz, 2H), 2.70 (dd, *J* = 14, 7.6 Hz, 1H), 2.54 (t, *J* = 13.6 Hz, 1H), 2.11 – 2.08 (m, 1H), 2.05 – 2.00 (m, 1H), 1.86 – 1.79 (m, 1H), 1.73 – 1.63 (m, 1H), 1.53 (q, *J* = 12.8 Hz, 1H), 1.11 (s, 3H), 0.68 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 175.9, 103.2, 77.2, 67.9, 44.8, 32.9, 30.2, 29.9, 23.4, 22.8, 21.8. IR (neat) 2958, 2853, 1729, 1472, 1393, 1311, 1137, 1078,1018, 957, 929, 792, 646 cm$^{-1}$. HRMS (ESI) m/z calcd for $C_{12}H_{20}KO_4$ [M + K]$^+$: 267.0993, found: 267.0996.



To a stirred mixture of tryptamine (0.38 g, 2.41 mmol) and **7b** (0.50 g, 2.19 mmol) in dry $CH_2Cl_2$ (22.0 mL) was added 2.0 M $AlMe_3$-hexane (1.70 mL, 3.30 mmol) at 0 °C, and then the reaction mixture was stirred for 5 h at room temperture. The reaction was carefully quenched with $H_2O$ at 0 °C and the mixture was extracted with $CH_2Cl_2$ (3 x 30 mL). The organic extract was washed with brine, dried over $Na_2SO_4$, and evaporated to give a residue, which was chromatographed on silica gel [$CH_2Cl_2$/MeOH = 20:1, $R_f$ (**15**) = 0.5] to afford the desired product **15** (0.79 g, 93%) as a gel product. $^1H$ NMR (400 MHz, $CDCl_3$) δ 8.73 (s, 1H), 7.58 (d, *J* = 8 Hz, 1H), 7.37 (d, *J* = 8.4 Hz, 1H), 7.17 (t, *J* = 7.2 Hz, 1H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.99 (s, 1H), 5.96 (s, 1H), 4.29 (d, *J* = 2.8 Hz, 1H), 3.69 – 3.61 (m, 2H), 3.58 – 3.49 (m, 4H), 3.36 – 3.29 (m, 2H), 2.93 (t, *J* = 6.8 Hz, 2H), 2.29 – 2.13 (m, 2H), 1.84 – 1.71 (m, 3H), 1.66 – 1.57 (m, 1H), 1.51 – 1.473 (m, 1H), 1.12 (s, 3H), 0.67 (s, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 173.6, 136.6, 127.4, 122.5, 122.0, 119.3, 118.7, 112.7, 111.4, 104.1, 77.3, 77.2, 60.8, 39.8, 39.2, 34.5, 32.1, 30.2, 25.4, 25.3, 23.1, 21.8. IR (neat) 3286, 2950, 2893, 1636, 1534, 1456, 1361, 1100, 972, 925, 739, 701, 568 $cm^{-1}$. HRMS (ESI) m/z calcd for $C_{22}H_{32}N_2NaO_4$ $[M + Na]^+$: 411.2254, found: 411.2262.

**Table 1** Exploration and optimization studies for synthesis of *trans*-fused indoloquinolizidine core **4a**.[a]

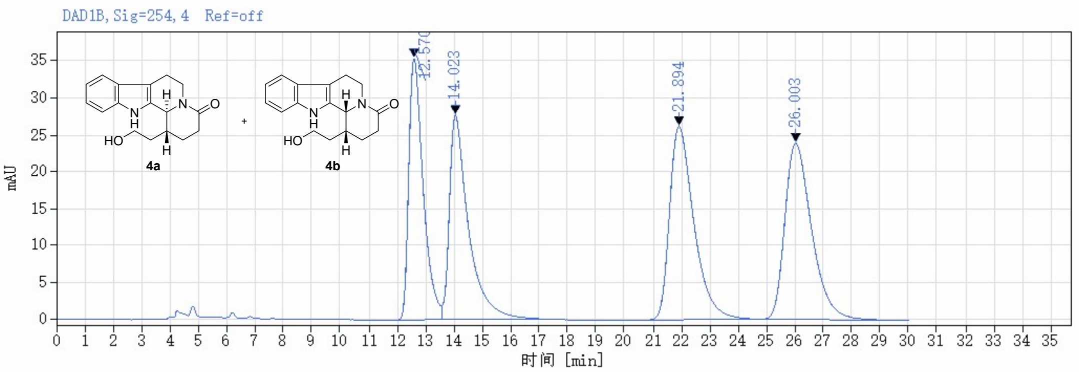
| Entry | Acid | Sol. | Time (h) | Yield(%) [b] | *dr* [c] |
|---|---|---|---|---|---|
| 1 | TFA | DCM | 16 | 46 | 5.5:1 |
| 2 | TFA | MeCN | 18 | 45 | 2.8:1 |
| 3 | TFA | PhMe | 24 | 54 | 4.6:1 |
| 4 | TFA | THF | 72 | / | / |
| 5 | TFA | EtOH | 72 | / | / |
| 6 | TFA | Acetone | 8 | 58 | 7.2:1 |
| 7 | TsOH | Acetone | 6 | 65 | 8.7:1 |
| 8 | CSA | Acetone | 12 | 81 | 12.5:1 |
| 9 | MsOH | Acetone | 24 | 60 | 10.0:1 |
| 10 | AcOH | Acetone | 48 | / | / |
| 11 | Dichloroacetic acid | Acetone | 36 | 83 | 16.5:1 |

[a]Unless noted otherwise, reactions were performed on scale of 100 mg of **15** at 25 °C. [b]Isolated yields. [c]Determined by HPLC analysis.

**Procedure (entries 1 to 11)**

Acid (10.0 equiv.) was added into a solution (4.90 mL) of **15** (0.10 g, 0.26 mmol).[1] After stirring at room temperature for various time, the pH was adjusted to 7.0 at 0 °C by adding saturated $Na_2CO_3$ aqueous solution. The aqueous layer was extracted with DCM. The combined organic layers were washed with brine, dried over $Na_2SO_4$, filtered, and evaporated in vacuo. The residue was purified by silica gel flash column chromatography [($CH_2Cl_2$/MeOH = 20:1, $R_f$ (**4a**) = 0.4, $R_f$ (**4b**) = 0.3)] to afford the product as a mixture of **4a** and **4b** compounds. Chiral HPLC conditions: Daicel Chiralcel OD column, 4.6 × 250 mm, 85:15 *n*-hexane/*i*-PrOH, 0.75 mL/min, UV detector at 254 nm. $t_{R1}$ = 12.5 min, $t_{R2}$ = 14.0 min, $t_{R3}$ = 21.9 min, $t_{R4}$ = 26.0 min. *dr* = 16.5:1 (**4a**:**4b**) (Table 1). In addition, **4a** and **4b** could be separated via column chromatography ($CH_2Cl_2$/MeOH = 30:1) to give **4a** as a white solid.

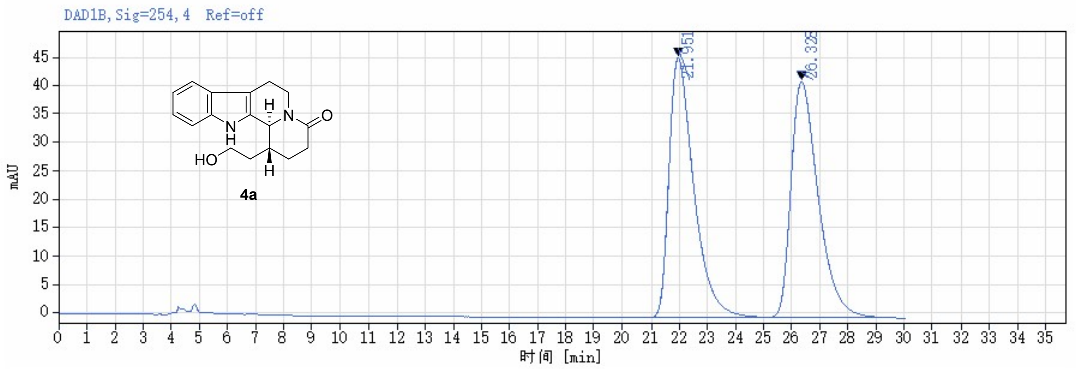
**Table 2.** HPLC Spectrum of mixture of **4a** and **4b**



| RetTime [min] | Type | Width [min] | Area [mAU * s] | Height [mAU] | Area% |
|---|---|---|---|---|---|
| 12.570 | MM m | 0.53 | 1250.48 | 35.48 | 22.03 |
| 14.023 | MM m | 0.67 | 1302.87 | 27.75 | 22.95 |
| 21.894 | MM m | 0.89 | 1557.91 | 26.23 | 27.44 |
| 26.003 | MM m | 0.95 | 1565.41 | 23.94 | 27.58 |
| | | Totals | 5676.67 | | |

Chiral HPLC conditions: Daicel Chiralcel OD column, 4.6 × 250 mm, 85:15 *n*-hexane/*i*-PrOH, 0.75 mL/min, UV detector at 254 nm. $t_{R1}$ = 12.5 min, $t_{R2}$ = 14.0 min, $t_{R3}$ = 21.9 min, $t_{R4}$ = 26.0 min

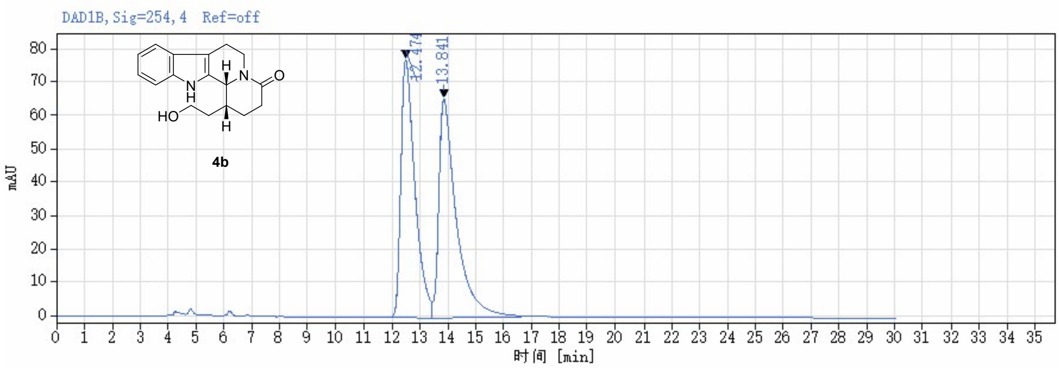
**Table 3.** HPLC Spectrum of **4a**



| RetTime [min] | Type | Width [min] | Area [mAU * s] | Height [mAU] | Area% |
|---|---|---|---|---|---|
| 21.951 | MM m | 0.90 | 2786.11 | 45.80 | 50.02 |
| 26.328 | MM m | 1.00 | 2783.82 | 41.56 | 49.98 |
| | | Totals | 5569.92 | | |

Chiral HPLC conditions: Daicel Chiralcel OD column, 4.6 × 250 mm, 85:15 *n*-hexane/*i*-PrOH, 0.75 mL/min, UV detector at 254 nm. $t_{R3}$ (**4a**) = 21.9 min, $t_{R4}$ (**4a**) = 26.3 min.

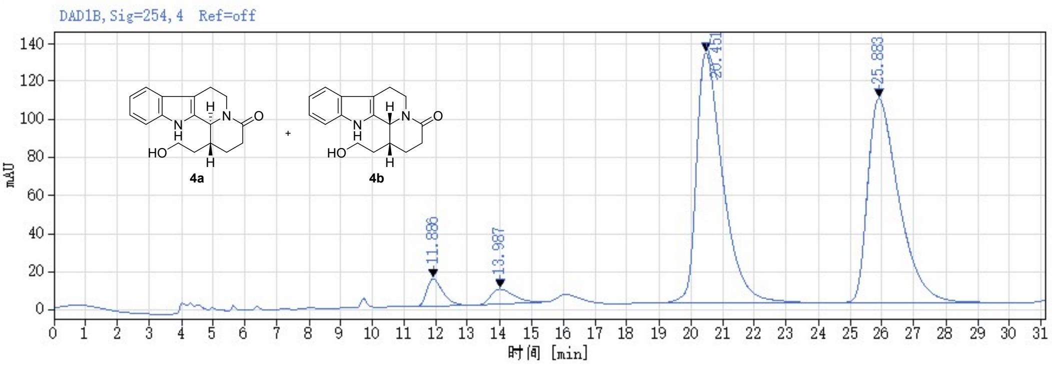
**Table 4.** HPLC Spectrum of **4b**



| RetTime [min] | Type | Width [min] | Area [mAU * s] | Height [mAU] | Area% |
|---|---|---|---|---|---|
| 12.474 | MM m | 0.52 | 2699.01 | 77.42 | 48.49 |
| 13.841 | MM m | 0.62 | 2867.46 | 65.73 | 51.51 |
| | | Totals | 5566.47 | | |

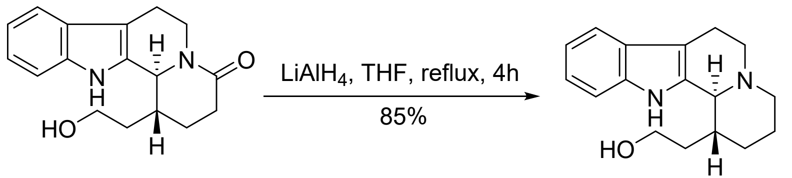
Chiral HPLC conditions: Daicel Chiralcel OD column, 4.6 × 250 mm, 85:15 *n*-hexane/*i*-PrOH, 0.75 mL/min, UV detector at 254 nm. $t_{R1}$ (**4b**) = 12.4 min, $t_{R2}$ (**4b**) = 13.8 min.

**Table 5.** HPLC Spectrum of the products of acyl-Pictet-Spengler cascade reaction from **15** (entry 11)



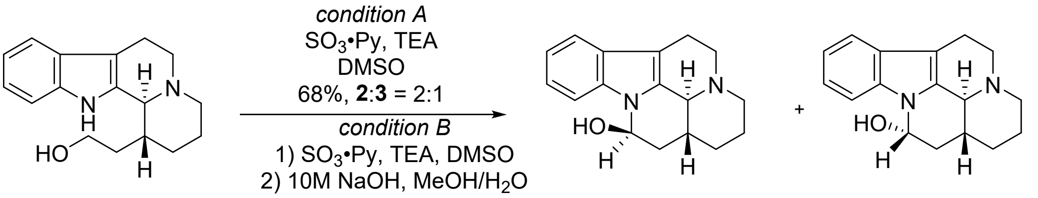
| RetTime [min] | Type | Width [min] | Area [mAU * s] | Height [mAU] | Area% |
|---|---|---|---|---|---|
| 11.886 | MM m | 1.89 | 505.83 | 14.42 | 3.23 |
| 13.987 | MM m | 2.15 | 393.80 | 7.82 | 2.51 |
| 20.451 | MM m | 4.94 | 7521.93 | 131.45 | 47.98 |
| 25.883 | MM m | 5.71 | 7526.991 | 10717 | 46.29 |
| | | Totals | 15678.55 | | |

Chiral HPLC conditions: Daicel Chiralcel OD column, 4.6 × 250 mm, 85:15 *n*-hexane/*i*-PrOH, 0.75 mL/min, UV detector at 254 nm. $t_{R1}$ (**4b**) = 11.9min, $t_{R2}$ (**4b**) = 13.9 min, $t_R$ (**4a**) = 20.4 min, $t_R$ (**4a**) = 25.8 min.



**4a** **16**

To a stirred solution of compound **4a** (0.30 g, 1.05 mmol) in dry THF (20.0 mL) was added $LiAlH_4$ (0.20 g, 5.27 mmol). After the reaction mixture was refluxed for 4 h, the saturated $Na_2SO_4$ solution (30 mL) was added and extracted with $CH_2Cl_2$ (3 x 30 mL).Then the combined organic layers were concentrated in vacuo and the crude products were purified directly by column chromatography on silica gel [$CH_2Cl_2$/MeOH = 20:1, $R_f$ (**16**) = 0.2] to afford the desired product **16** (0.24 g, 85%) as a white soild. mp = 216.3 - 220.3 °C; $^1H$ NMR (400 MHz, DMSO-*d₆*) δ 10.58 (s, 1H), 7.33 (q, *J* = 7.6 Hz, 2H), 7.00 (t, *J* = 7.2 Hz, 1H), 6.93 (t, *J* = 7.6 Hz, 1H), 4.51 (t, *J* = 4.8 Hz, 1H), 3.75 (d, *J* = 5.2 Hz, 1H), 3.60 – 3.47 (m, 2H), 3.20 – 3.13 (m, 1H), 2.88 – 2.83 (m, 1H), 2.81 – 2.72 (m, 2H), 2.63 – 2.57 (m,1H), 2.54 – 2.50 (m,1H), 2.25 – 2.19 (m, 1H), 1.99 – 1.88 (m, 1H), 1.68 – 1.60 (m, 1H), 1.56 – 1.43 (m, 3H), 1.37 – 1.28 (m, 1H). $^{13}C$ NMR (100 MHz, DMSO-$d_6$) δ 135.7, 134.9, 126.9, 120.2, 118.1, 117.3, 111.0, 106.5, 59.5, 58.9, 49.6, 49.2, 34.9, 31.8, 26.8, 20.6, 18.9. IR (KBr) 3188, 3059, 2920, 1454, 1361, 1321, 1255, 1107, 1056, 858, 736 $cm^{-1}$. HRMS (ESI) m/z calcd for $C_{17}H_{23}N_2O$ $[M + H]^+$: 271.1805, found: 271.1810.



**16** (±)-vindeburnol (**2**) (±)-16-*epi*-vindeburnol (**3**)

**Condition A**

To a stirred solution of compound **16** (0.15 g, 0.55 mmol) in dry DMSO (5.50 mL) was added $SO_3$·Py (0.35 g, 2.20 mmol) and triethylamine (0.60 mL) . After at room temperature for 4h, the mixture was diluted by $CH_2Cl_2$ (20 mL), and washed with $H_2O$ (3 × 10 mL). The aqueous layer was extracted with $CH_2Cl_2$ (3 × 30 mL). The combined organic layers were washed with brine, dried over $Na_2SO_4$, filtered, and evaporated in vacuo to give a brown soild as crude product. The residue was purified by column chromatography [$CH_2Cl_2$/Acetone/MeOH = 25:4:1, $R_f$ (**2**) = 0.3, $R_f$ (**3**) = 0.5] to afford (±)-vindeburnol (**2**) (67.1 mg, 45%) as a brown solid, (±)-16-*epi*-vindeburnol (**3**) (34.2 mg, 23%) as a brown solid.

(±)-vindeburnol (**2**): mp = 178.9–180.9 °C; $^1H$ NMR (400 MHz, DMSO-$d_6$) δ 7.44 (d, *J* = 8 Hz, 1H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 8 Hz, 1H), 6.99 (t, *J* = 7.6 Hz, 1H), 6.17 (d, *J* = 6.8 Hz, 1H), 5.91 (d, *J* = 6.8 Hz, 1H), 3.05 (dd, *J* = 11.2, 6.0 Hz, 1H), 2.96 (d, *J* = 10.8 Hz, 1H), 2.81 – 2.72 (m, 1H), 2.66 – 2.61 (m, 2H), 2.41 (td, *J* = 11.2, 4.4 Hz, 1H), 2.16 (td, *J* = 11.2, 2.8 Hz, 1H), 1.98 – 1.92 (m, 3H), 1.76 – 1.62 (m, 3H), 1.19 – 1.10 (m, 1H). $^{13}C$ NMR (100 MHz, DMSO-$d_6$) δ 136.0, 134.3, 128.2, 120.1, 119.2, 117.6, 111.3, 103.7, 73.8, 63.7, 54.6, 52.6, 37.4, 30.8, 29.5, 25.4, 21.2. IR (KBr) 2924, 2852, 1627, 1444, 1377, 1263, 1201, 1176, 1105, 1062, 902, 875, 854, 856 $cm^{-1}$.HRMS (ESI) m/z calcd for $C_{17}H_{21}N_2O$ $[M + H]^+$: 269.1648, found: 269.1652.

(±)-16-*epi*-vindeburnol (**3**): mp = 170.1–171.9 °C; $^1H$ NMR (400 MHz, DMSO-$d_6$) δ 7.65 (d, *J* = 8 Hz, 1H), 7.37 (d, *J* = 8 Hz, 1H), 7.07 – 6.99 (m, 2H), 6.46 (d, *J* = 9.2 Hz, 1H), 5.54 – 5.48 (m, 1H), 3.02 (dd, *J* = 11.6, 6.0 Hz, 1H), 2.93 (d, *J* =10.4 Hz, 1H), 2.78 – 2.61 (m, 3H), 2.44 (dd, *J* = 11.6, 4.8 Hz, 1H), 2.25 – 2.17 (m, 2H), 1.77 – 1.58 (m, 4H), 1.49 – 1.40 (m, 1H), 1.18 (dd, *J* = 12.0, 4 Hz, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 137.4, 135.4, 127.9, 120.4, 119.3, 117.6, 112.0, 104.0, 77.9, 63.2, 54.3, 52.0, 38.8, 36.0, 29.6, 25.2, 21.3. IR (KBr) 2924, 2854, 2808, 1732, 1458, 1321, 1258, 1186, 1112, 1082, 966, 740 cm$^{-1}$. HRMS (ESI) m/z calcd for $C_{17}H_{21}N_2O$ [M + H]$^+$: 269.1648, found: 269.1654.

**Condition B**

The residue from the Parikh-Doering oxidation, which was directly used in next step without further purification, was added to the solution of 10M NaOH (MeOH/$H_2O$ = 1:1) for 36 h at room temperature. The mixture was diluted by $CH_2Cl_2$, and washed with $H_2O$. The aqueous layer was extracted with $CH_2Cl_2$. The combined organic layers were washed with brine, dried over $Na_2SO_4$, filtered, and evaporated in vacuo to give a brown soild as crude product. The crude products were purified directly by flash column chromatography to afford the mixture soild [(±)-vindeburnol (**2**): (±)-16-*epi*-vindeburnol (**3**) = 13:1, according to $^1$H NMR].

$^1$H NMR (400 MHz, DMSO-$d_6$) δ 7.65 (d, *J* = 8 Hz, 0.09H), 7.44 (d, *J* = 8 Hz, 1.01H), 7.36 (d, *J* = 7.2 Hz, 1.07H), 7.07 – 6.97 (m, 2.35H), 6.48 (d, *J* = 9.2 Hz, 0.08H), 6.18 (d, *J* = 6.8 Hz, 1.00H), 5.91 (d, *J* = 6.4 Hz, 1.01H), 5.54 – 5.48 (m, 0.09H), 3.06 (dd, *J* = 11.2, 6 Hz, 1.03H), 2.97 – 2.93 (m, 1.06H), 2.81 – 2.72 (m, 1.16H), 2.66 – 2.61 (m, 2.09H), 2.42 (qd, *J* = 11.2, 4.8 Hz, 1.01H), 2.22 – 2.13 (m, 1.11H), 1.95 – 1.88 (m, 2.91H), 1.76 – 1.65 (m, 3.78H), 1.16 – 1.11 (m, 1.07H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 137.3, 136.0, 135.7, 134.2, 128.1, 127.9, 120.4, 120.0, 119.3, 119.1, 117.6, 117.3, 111.9, 111.3, 103.9, 103.6, 77.8, 73.8, 63.6, 63.1, 54.5, 54.2, 52.6, 52.0, 37.3, 35.9, 31.2, 30.8, 29.6, 29.4, 25.3, 25.2, 21.3, 21.1.

**Table 6.** Comparison of $^1$H NMR data of (±)-vindeburnol (**2**) with literature

| Lit.[2] data (400 MHz, DMSO-*d*) δ/ppm | Our data (400 MHz, DMSO-*d*) δ/ppm | Error Δ δ/ppm |
|---|---|---|
| 7.43 (dt, *J* = 7.8, 0.8 Hz, 1H) | 7.44 (d, *J* = 8 Hz, 1H) | 0.01 |
| 7.35 (d, *J* = 7.5 Hz, 1H) | 7.36 (q, *J* = 7.6 Hz, 1H) | 0.01 |
| 7.05 (br t, *J* = 7.6 Hz, 1H) | 7.06 (t, *J* = 8 Hz, 1H) | 0.01 |
| 7.00 (br t, *J* = 7.4 Hz, 1H) | 6.99 (t, *J* = 7.6 Hz, 1H) | -0.01 |
| 6.14 (d, *J* = 6.9 Hz, 1H) | 6.17 (d, *J* = 6.8 Hz, 1H) | 0.03 |
| 5.9 (m, 1H) | 5.91 (d, *J* = 6.8 Hz, 1H) | 0 |
| 3.04 (dd, *J* = 11.3, 5.8 Hz, 1H) | 3.05 (dd, *J* = 11.2, 6.0 Hz, 1H) | 0.01 |
| 2.95 (m, 1H) | 2.96 (d, *J* = 10.8 Hz, 1H) | 0 |
| 2.77 (m, 1H) | 2.78 – 2.72 (m, 1H) | 0 |
| 2.64 (m, 2H) | 2.66 – 2.61 (m, 2H) | 0 |
| 2.41 (td, *J* = 11.4, 4.4 Hz, 1H) | 2.41 (td, *J* = 11.2, 4.4 Hz, 1H) | 0 |
| 2.16 (td, *J* = 11.1, 2.6 Hz, 1H) | 2.16 (td, *J* = 11.2, 2.8 Hz, 1H) | 0 |
| 1.98 – 1.89 (m, 3H) | 1.98 – 1.92 (m, 3H) | 0 |
| 1.78 – 1.61 (m, 3H) | 1.76 – 1.62 (m, 3H) | 0 |
| 1.15 (m,1H) | 1.19 – 1.10 (m, 1H) | 0 |

**Table 7.** Comparison of $^{13}$C NMR data of (±)-vindeburnol (**2**) with literature

| Lit.[2] data (125 MHz, DMSO-*d*) δ/ppm | Our data (100 MHz, DMSO-*d*) δ/ppm | Error Δ δ/ppm |
|---|---|---|
| 136.0 | 136.0 | 0 |
| 134.3 | 134.3 | 0 |
| 128.2 | 128.2 | 0 |
| 120.0 | 120.1 | 0.1 |
| 119.2 | 119.2 | 0 |

| | | |
|---|---|---|
| 117.6 | 117.6 | 0 |
| 111.3 | 111.3 | 0 |
| 103.7 | 103.7 | 0 |
| 73.8 | 73.8 | 0 |
| 63.7 | 63.7 | 0 |
| 54.6 | 54.6 | 0 |
| 52.6 | 52.6 | 0 |
| 37.4 | 37.4 | 0 |
| 30.8 | 30.8 | 0 |
| 29.5 | 29.5 | 0 |
| 25.4 | 25.4 | 0 |
| 21.2 | 21.2 | 0 |

**Table 8.** Comparison of $^1$H NMR data of (±)-16-epi-vindeburnol (**3**) with literature

| Lit.[2] data (400 MHz, DMSO-*d*) δ/ppm | Our data (400 MHz, DMSO-*d*) δ/ppm | Error Δ δ/ppm |
|---|---|---|
| 7.65 (m, 1H) | 7.65 (d, *J* = 8 Hz, 1H) | 0 |
| 7.37 (m, 1H) | 7.37 (q, *J* = 8 Hz, 1H) | 0 |
| 7.03 (m, 2H) | 7.07 – 6.99 (m, 2H) | 0 |
| 6.44 (d, *J* = 9.0 Hz, 1H) | 6.46 (d, *J* = 9.2 Hz, 1H) | 0.02 |
| 5.5 (td, *J* = 9.0, 5.7 Hz, 1H) | 5.54 – 5.48 (m, 1H) | 0 |
| 3.02 (dd, *J* = 11.2, 5.6 Hz, 1H) | 3.02 (dd, *J* = 11.6, 6.0 Hz, 1H) | 0 |
| 2.92 (dt, *J* = 11.0, 2.7 Hz, 1H) | 2.93 (d, *J* = 10.4 Hz, 1H) | 0.01 |
| 2.79 – 2.61 (m, 3H) | 2.78 – 2.61 (m, 3H) | 0 |
| 2.45 (td, *J* = 11.2, 4.4 Hz, 1H) | 2.44 (dd, *J* = 11.2, 4.4 Hz, 1H) | -0.01 |
| 2.20 (m, 2H) | 2.25 – 2.17 (m, 2H) | 0 |
| 1.77 – 1.55 (m, 4H) | 1.77 – 1.58 (m, 4H) | 0 |
| 1.44 (m,1H) | 1.49 – 1.40 (m, 1H) | 0 |
| 1.17 (m,1H) | 1.17 (dd, *J* = 12, 4 Hz, 1H) | 0 |

**Table 9.** Comparison of $^{13}$C NMR data of (±)-16-epi-vindeburnol (**3**) with literature

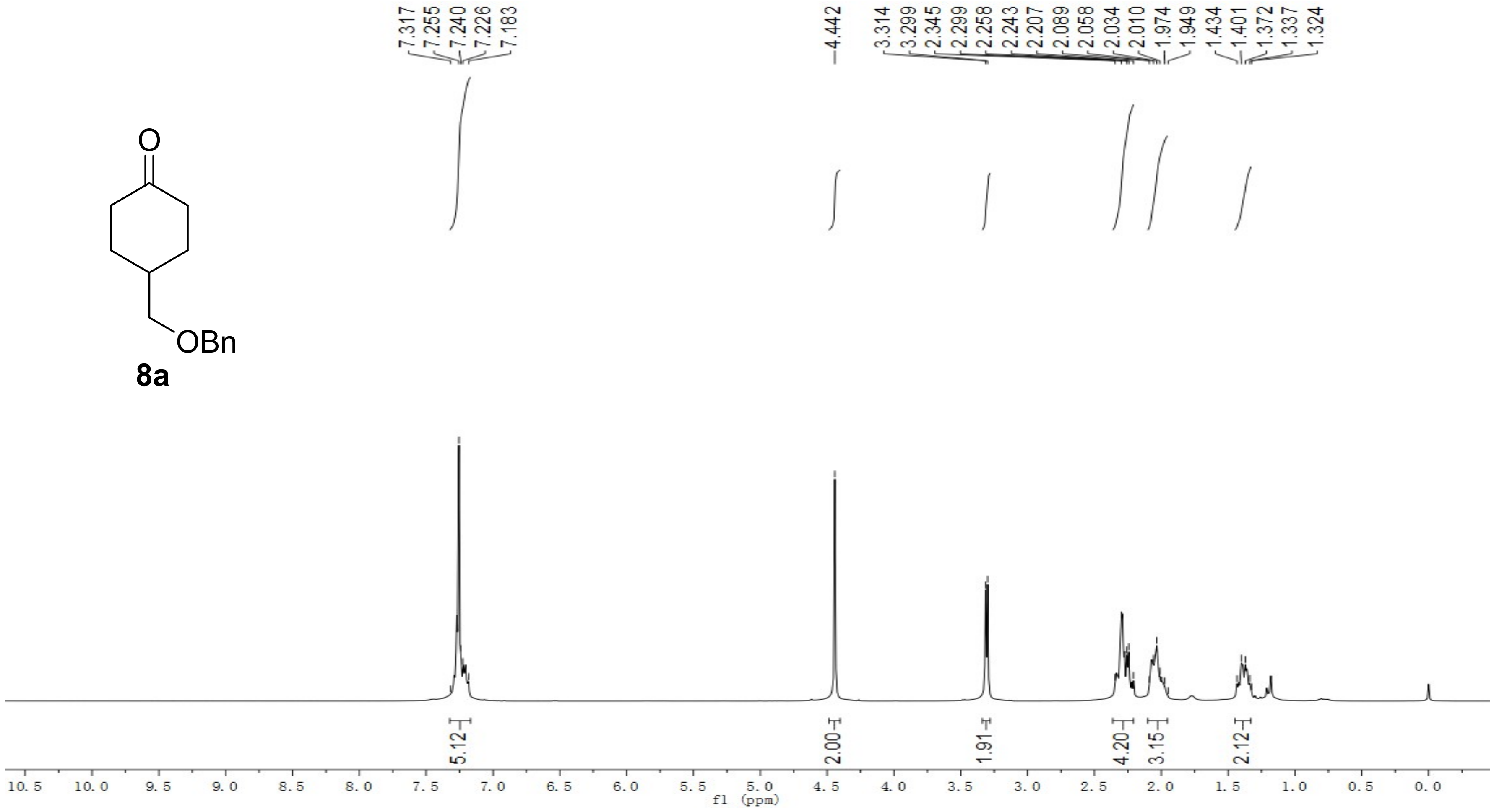
| Lit.[2] data (125 MHz, DMSO-*d*) δ/ppm | Our data (100 MHz, DMSO-*d*) δ/ppm | Error Δ δ/ppm |
|---|---|---|
| 137.4 | 137.4 | 0 |
| 135.4 | 135.4 | 0 |
| 127.9 | 127.9 | 0 |
| 120.3 | 120.4 | 0.1 |
| 119.3 | 119.3 | 0 |
| 117.6 | 117.6 | 0 |
| 111.9 | 112.0 | 0.1 |
| 103.9 | 104.0 | 0.1 |
| 77.8 | 77.9 | 0.1 |
| 63.2 | 63.2 | 0 |
| 54.3 | 54.3 | 0 |
| 52.0 | 52.0 | 0 |
| 38.8 | 38.8 | 0 |
| 36.0 | 36.0 | 0 |
| 29.6 | 29.6 | 0 |
| 25.2 | 25.2 | 0 |
| 21.3 | 21.3 | 0 |

# 3. References

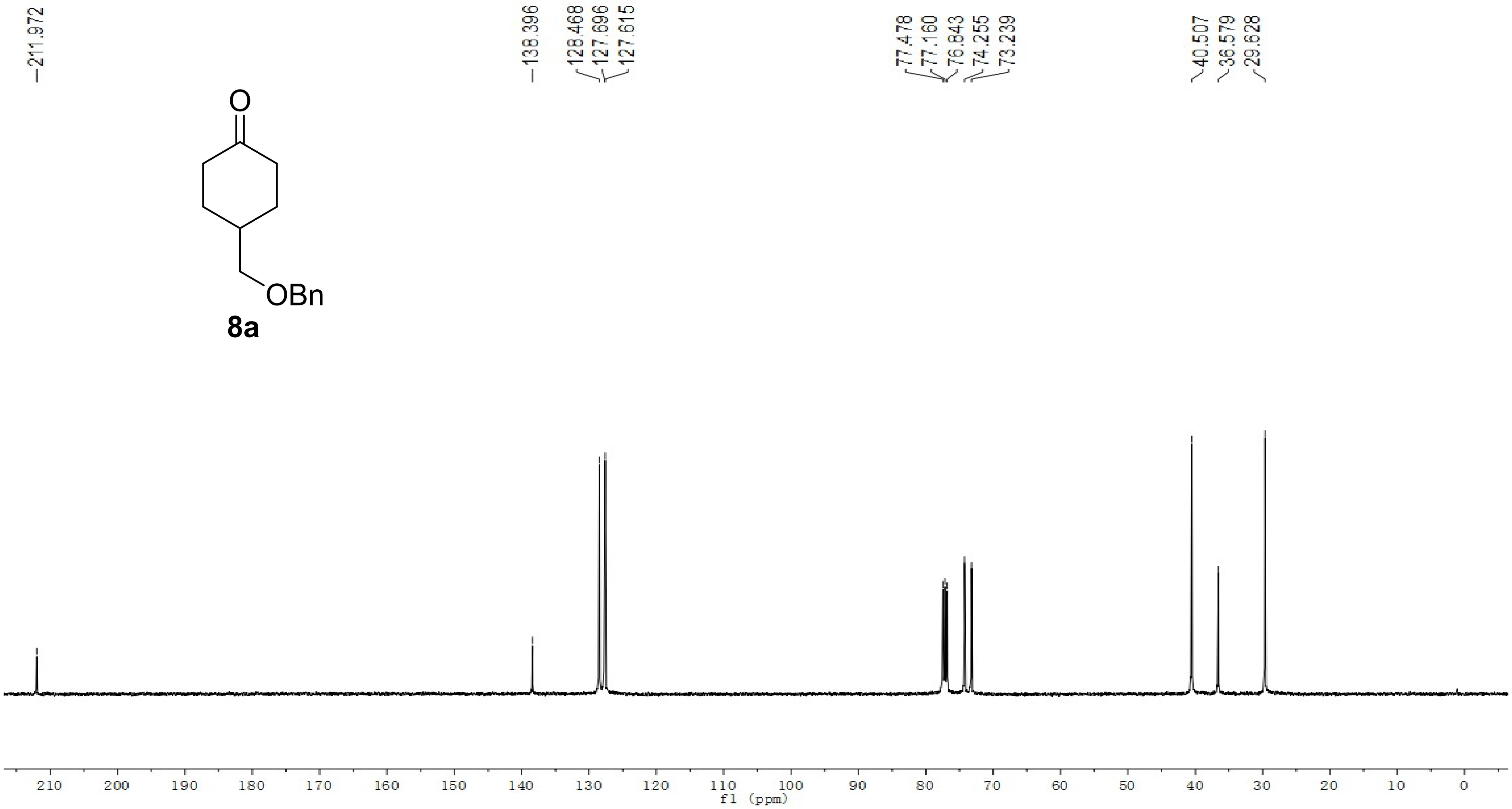
1 T. Yamashita, N. Kawai, H. Tokuyama and T. Fukuyama, *J. Am. Chem. Soc.*, 2005, **127**, 15038.

2 L. Salacz, C. Charpentier and N. Girard, *J. Org. Chem.* 2017, **82**, 2257.

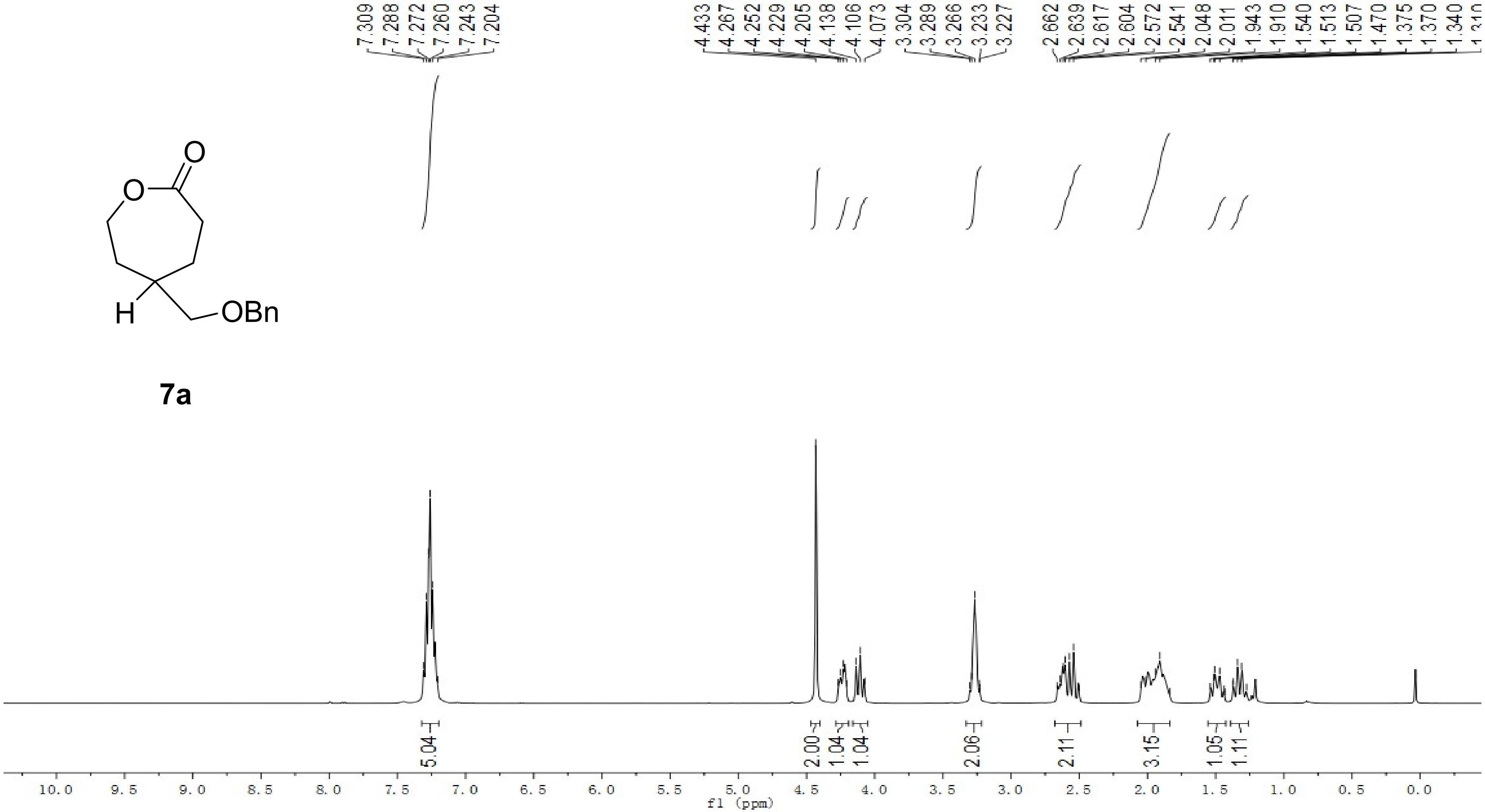
## 4. NMR Spectra



$^1$H-NMR Spectrum of 8a

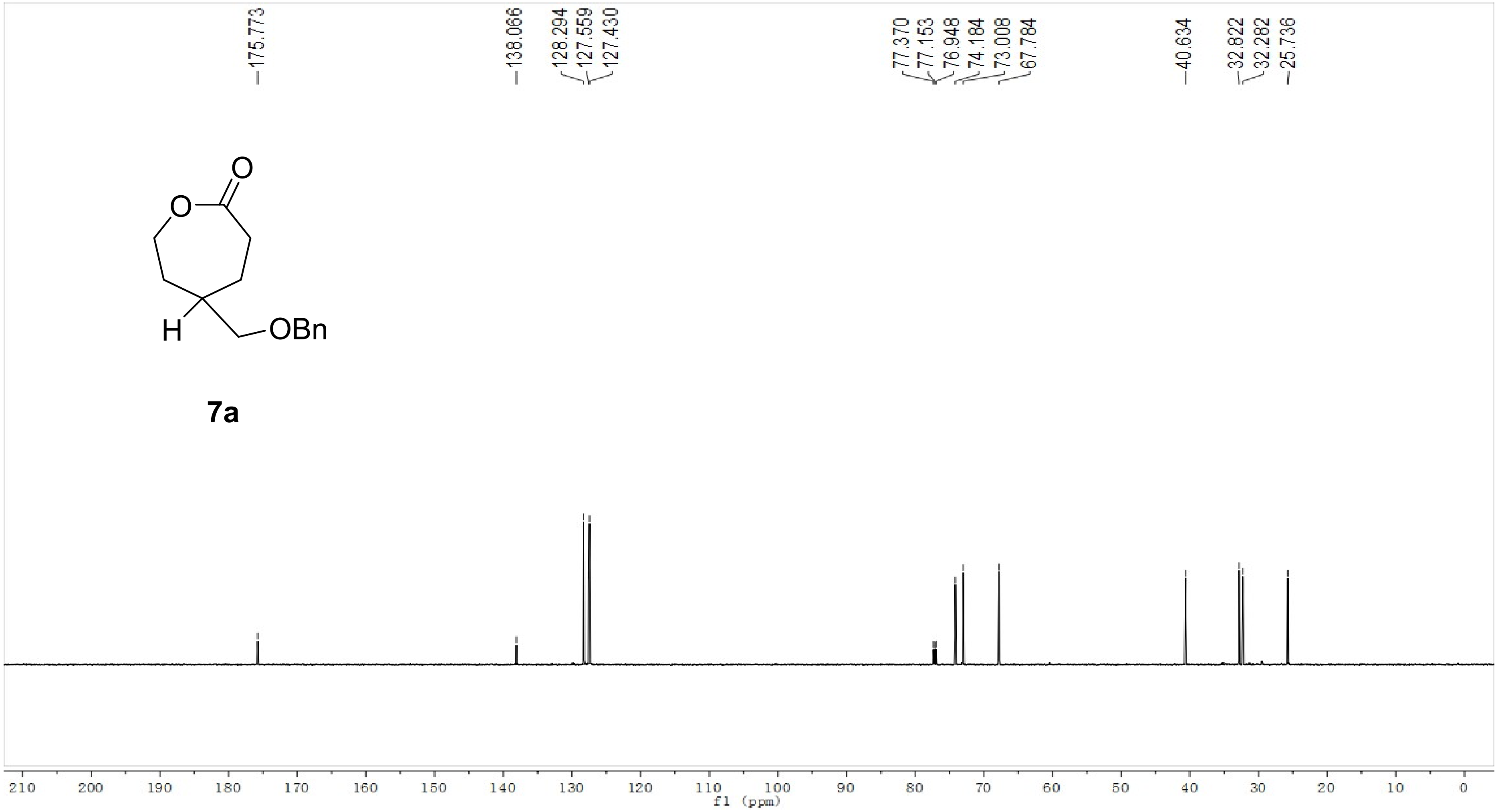


$^{13}$C-NMR Spectrum of 8a

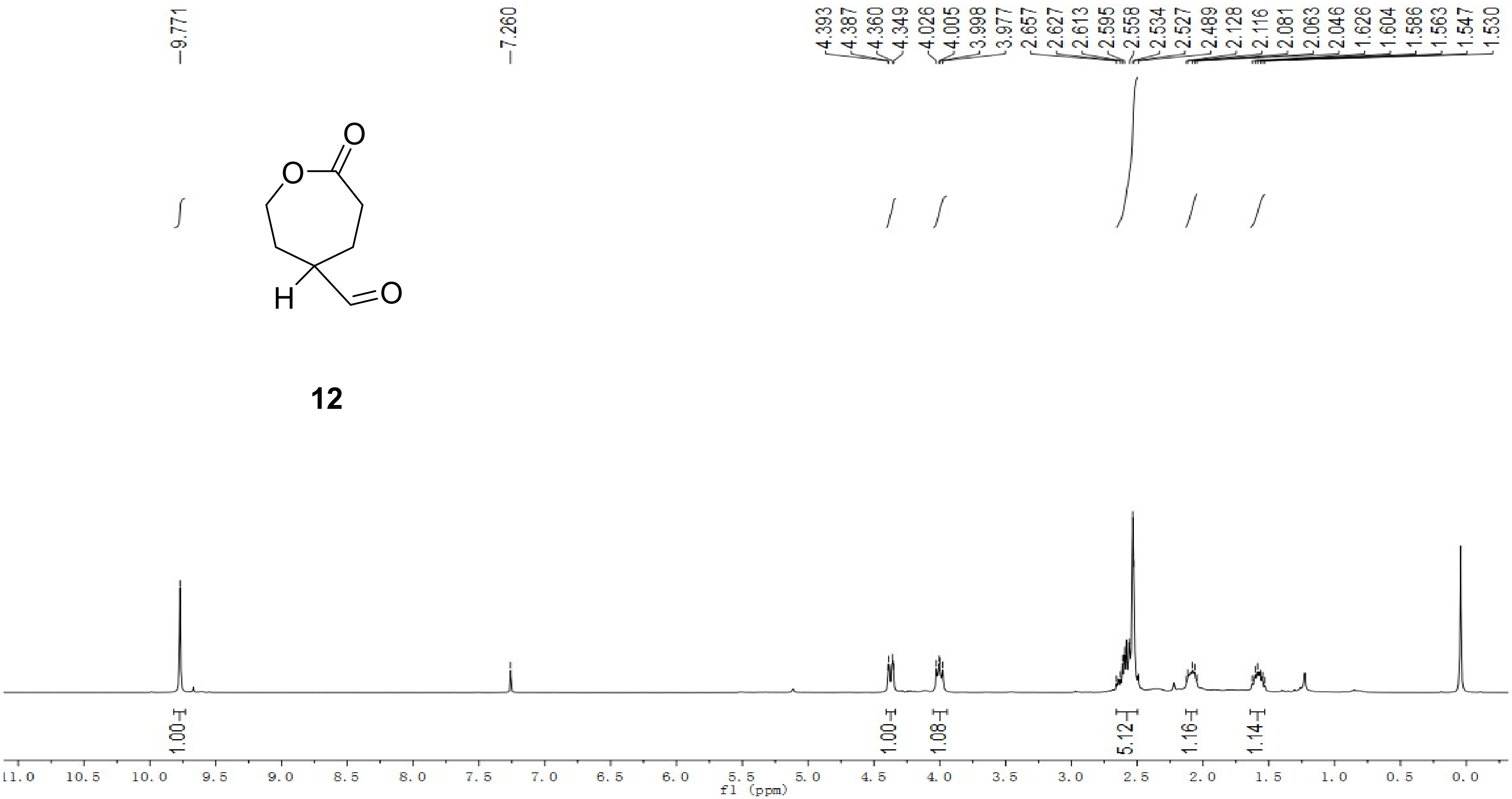


**7a**

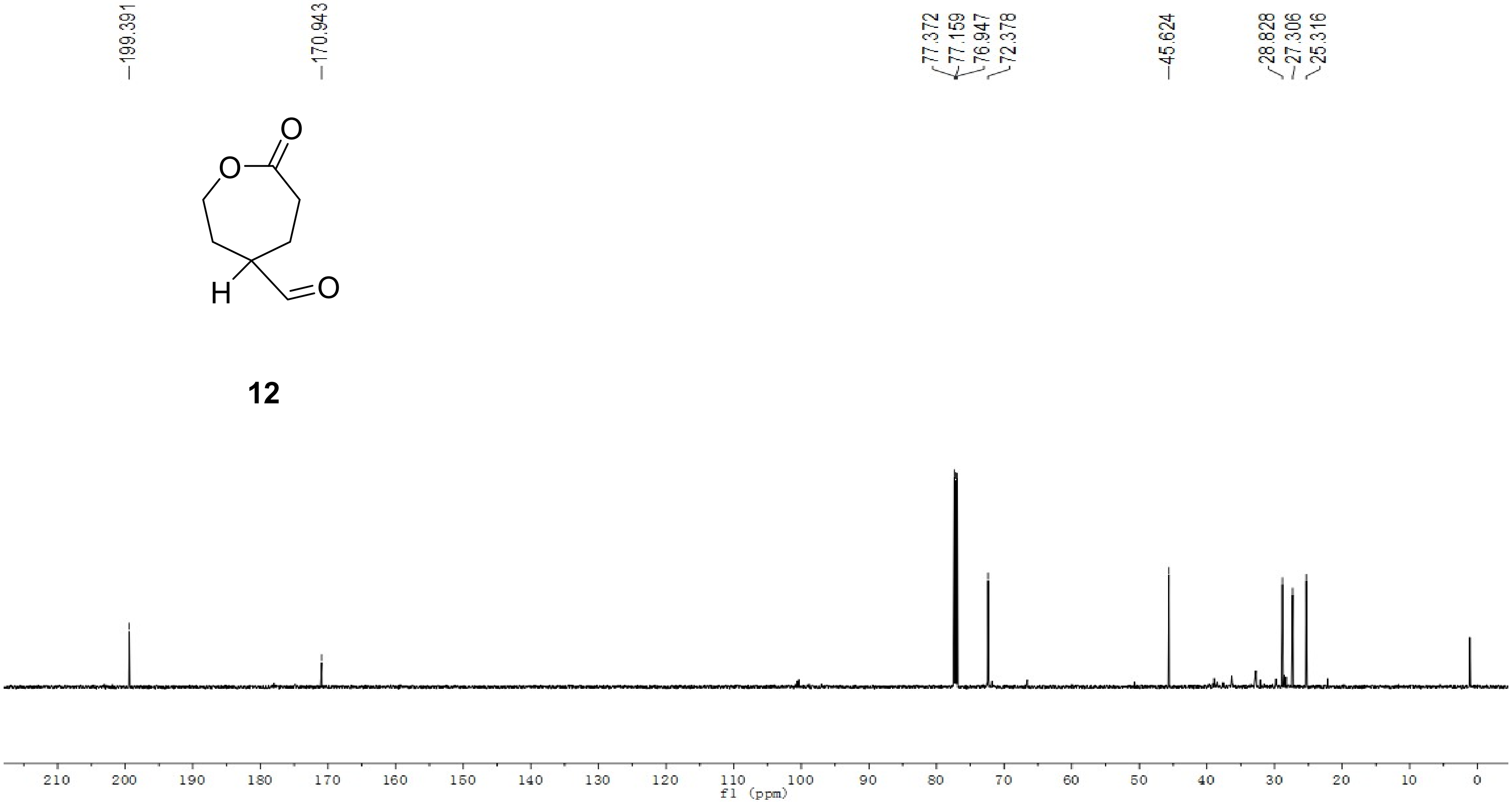
$^1$H-NMR Spectrum of 7a



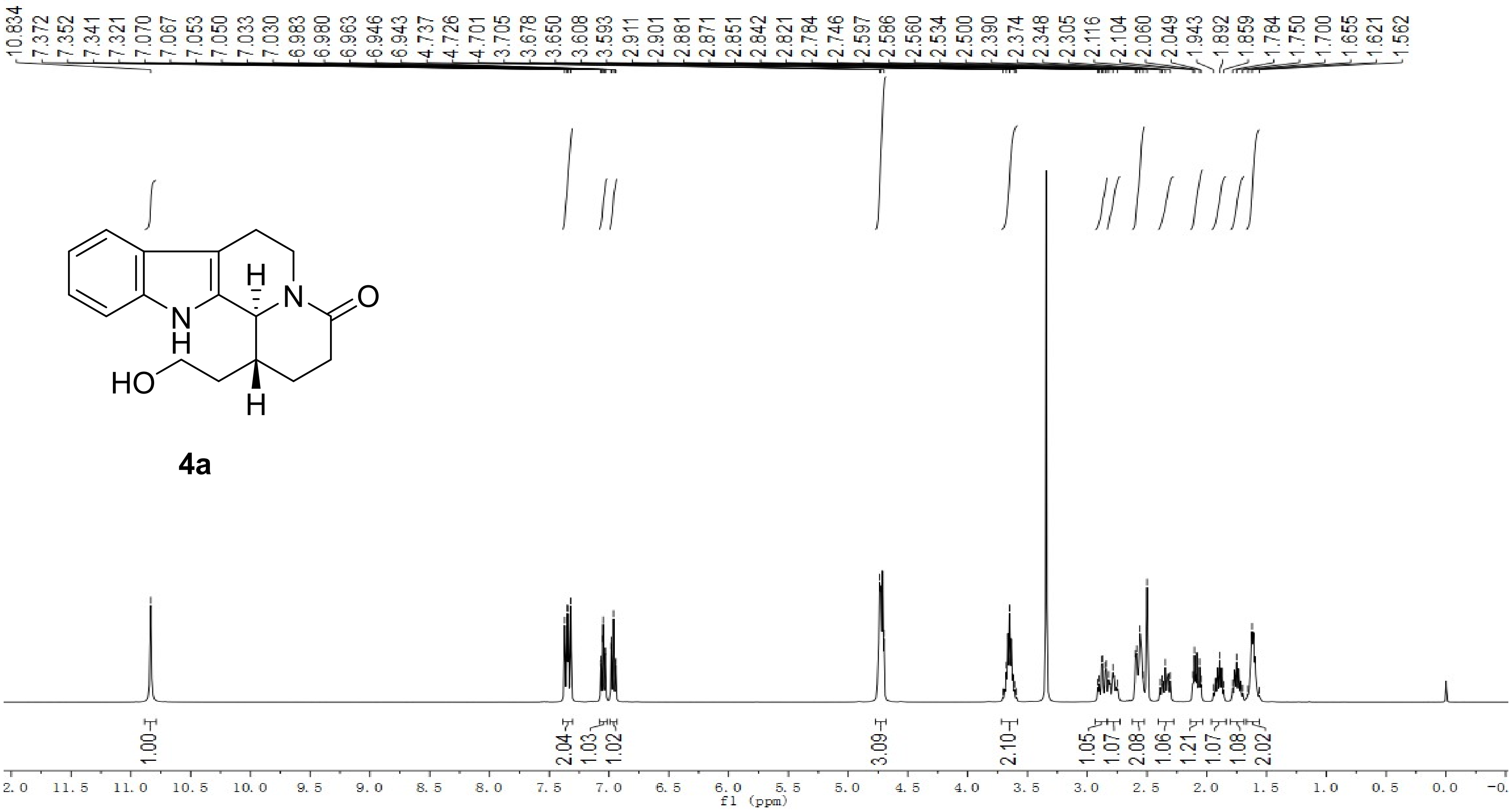
**$^{13}$C-NMR Spectrum of 7a**



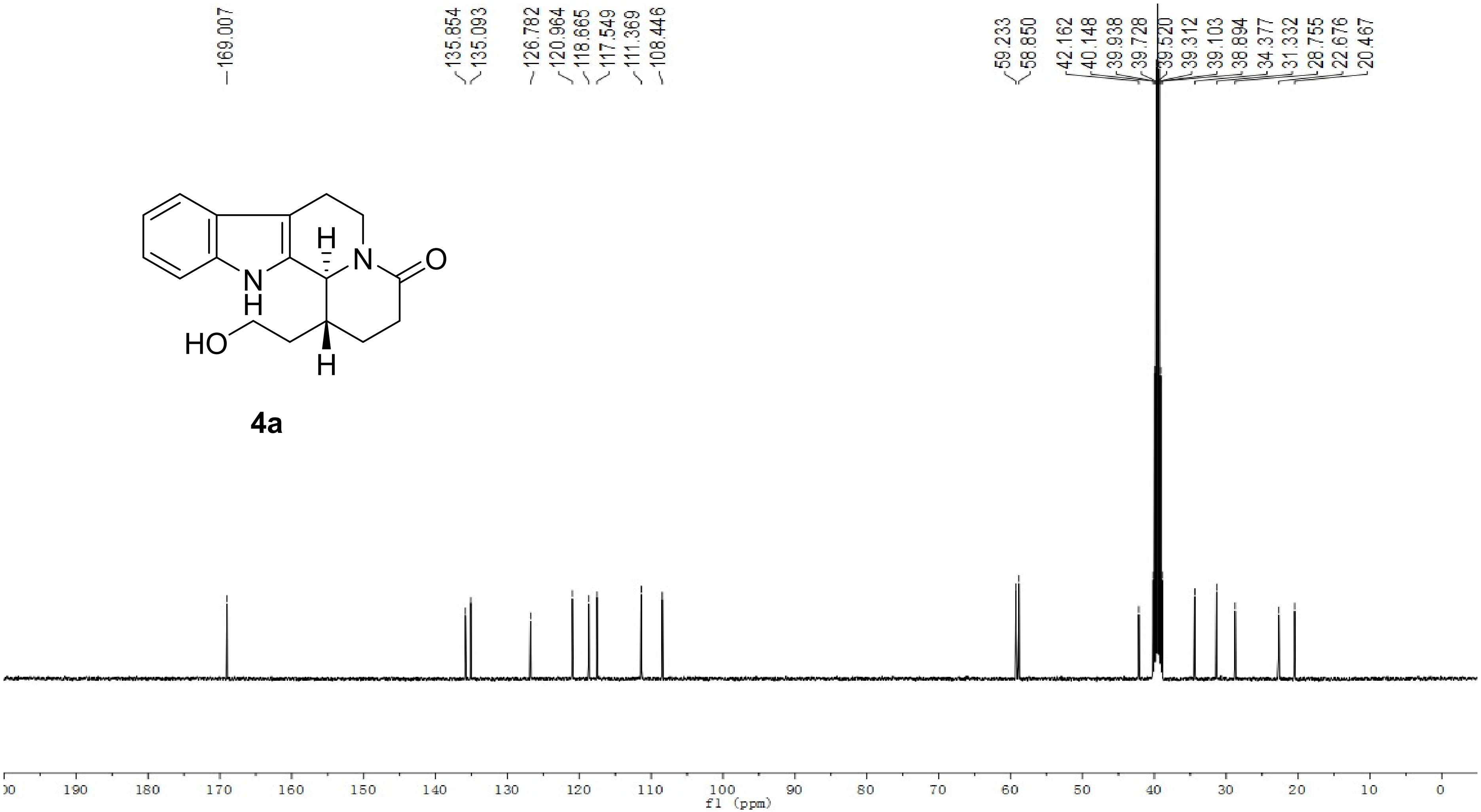
$^1$H-NMR Spectrum of 12



$^{13}$C-NMR Spectrum of 12

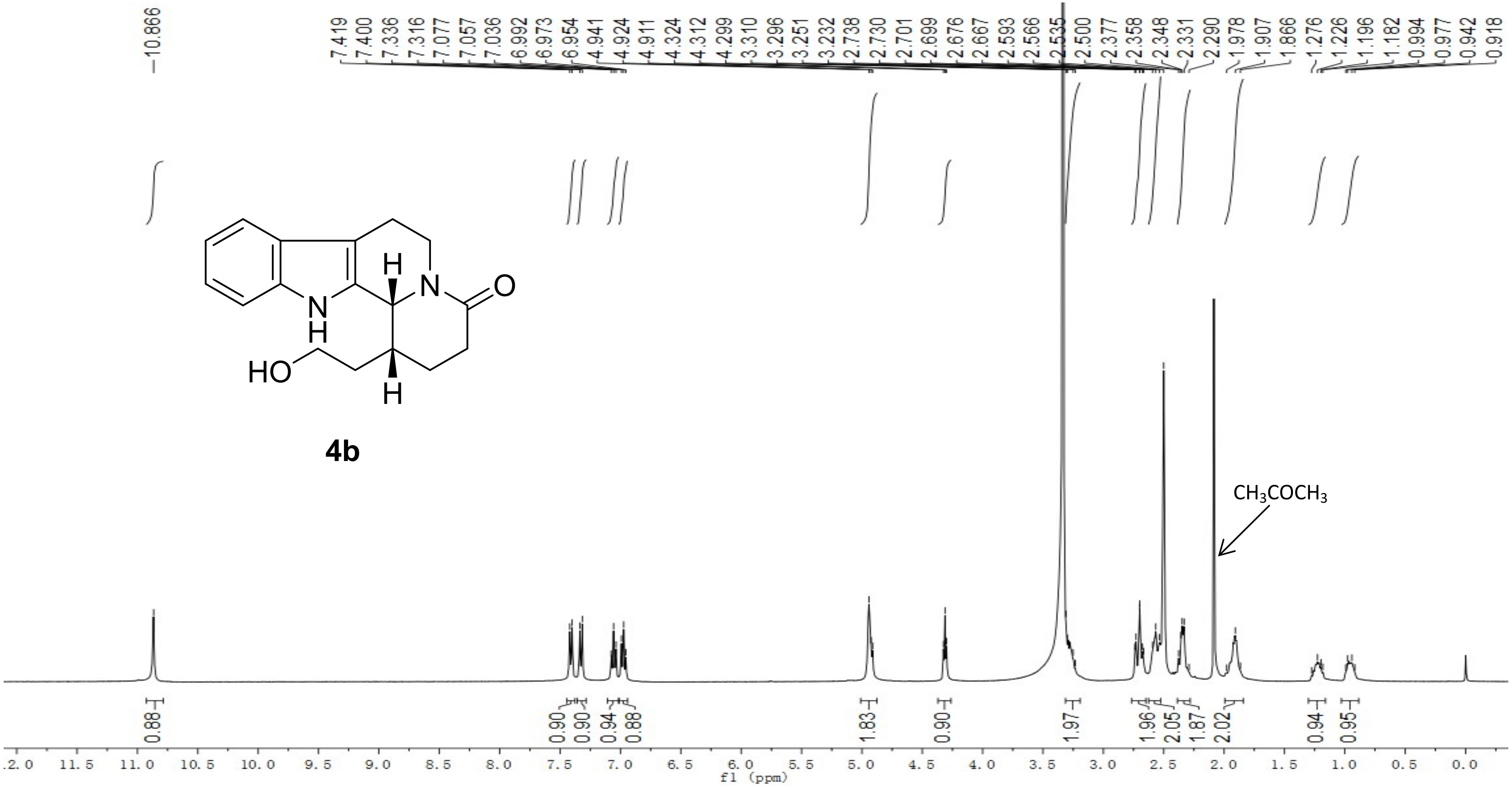


**$^1$H-NMR Spectrum of 4a**



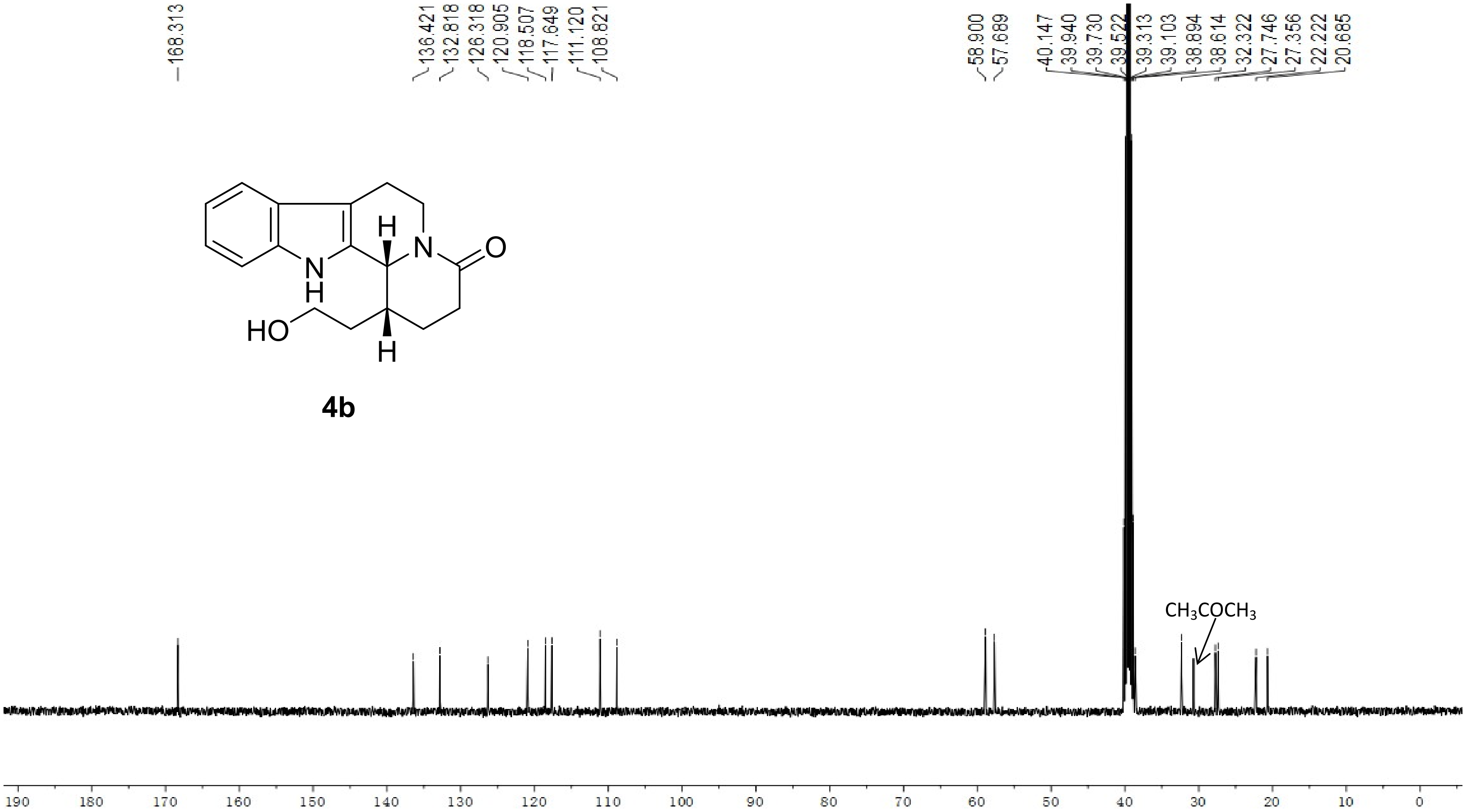
**4a**

**$^{13}$C-NMR Spectrum of 4a**

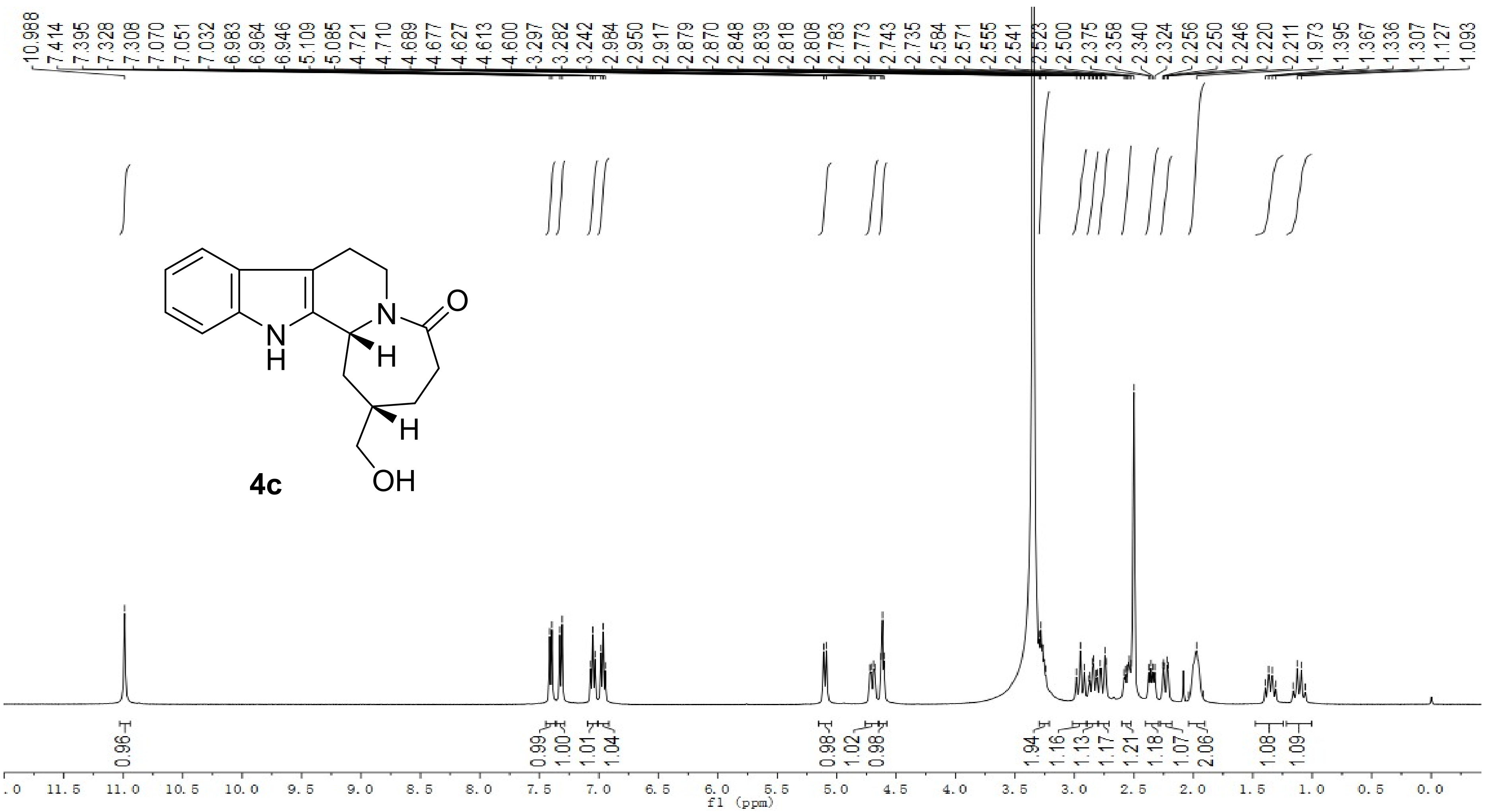


**4b**

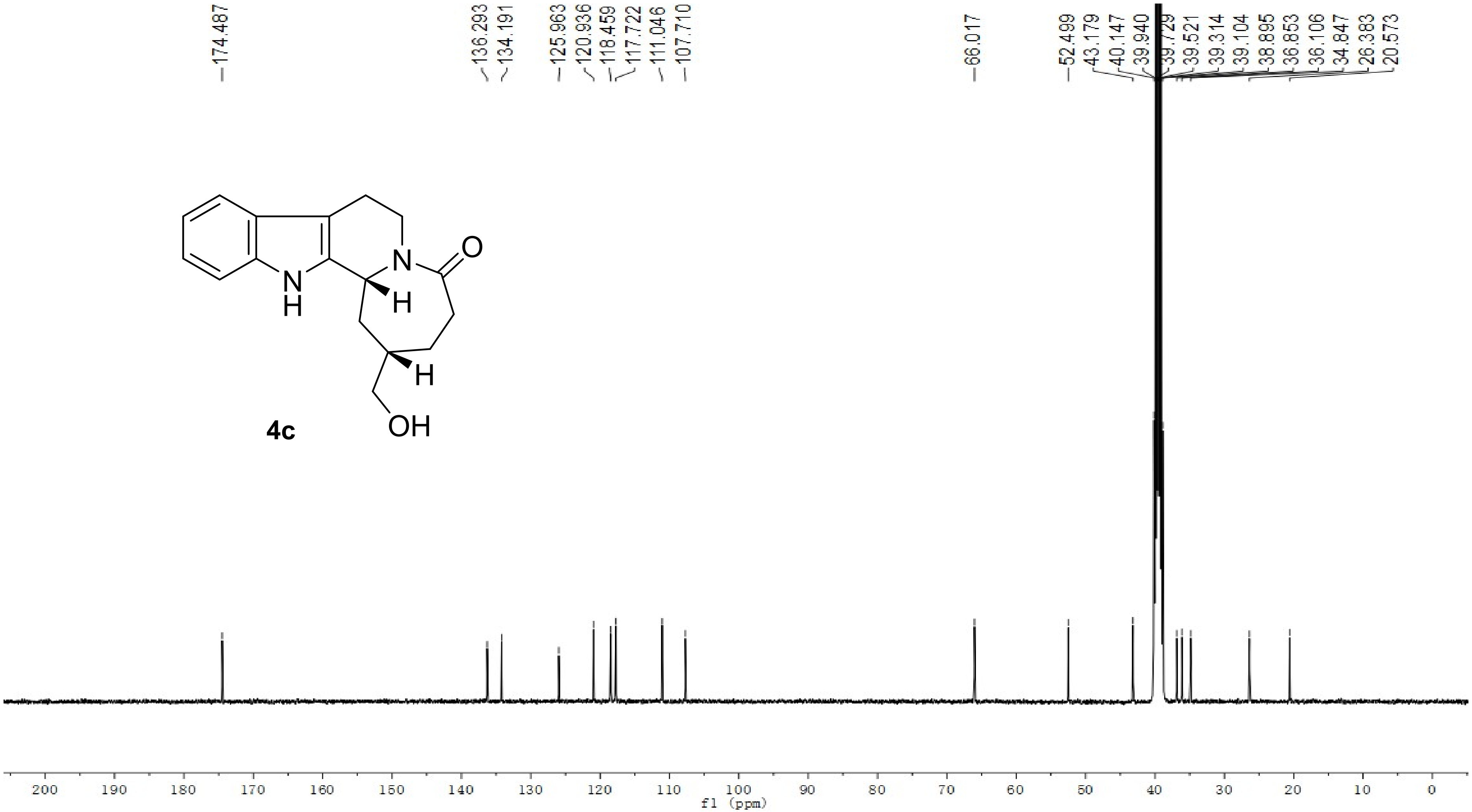
**$^1$H-NMR Spectrum of 4b**



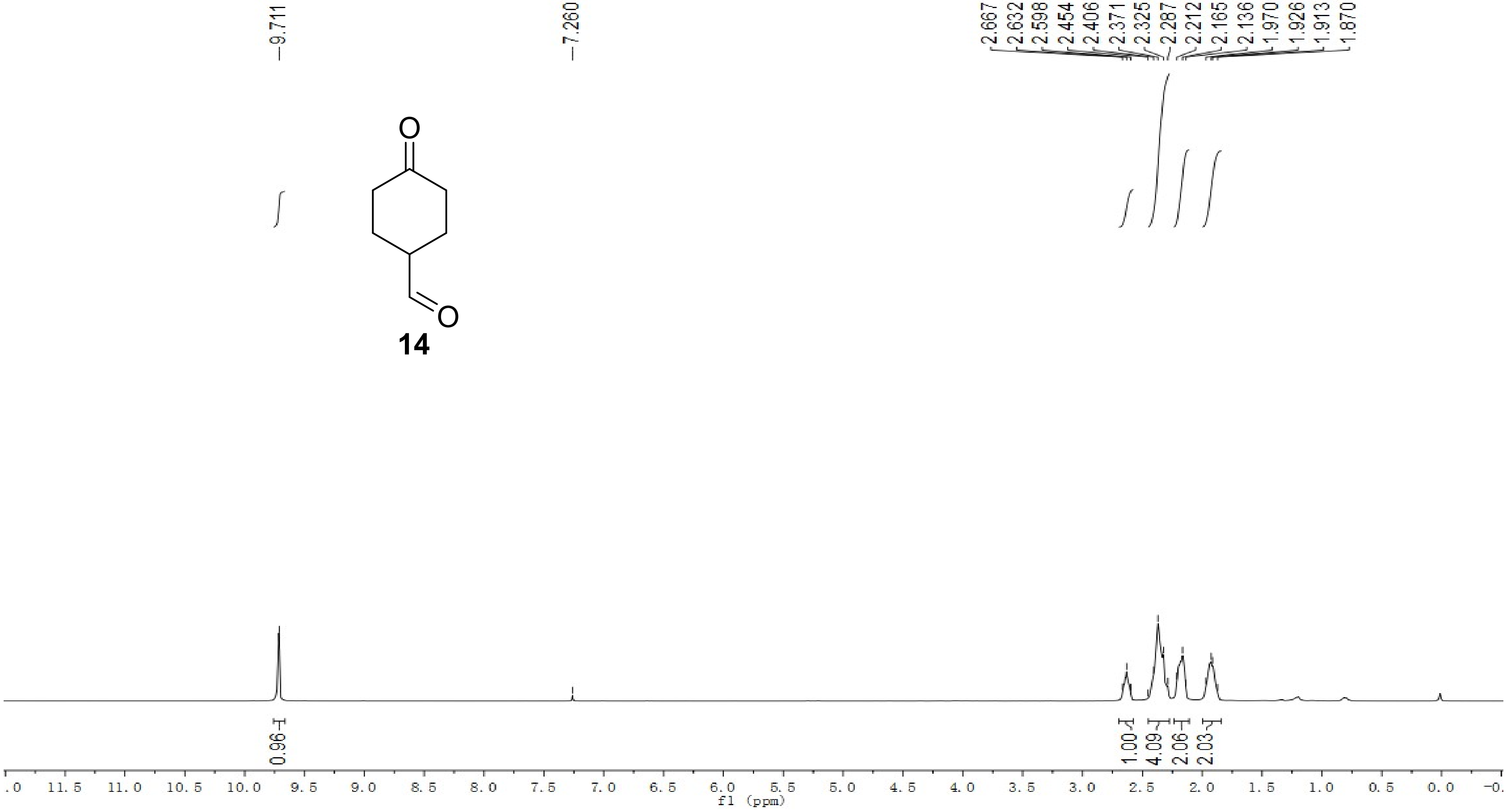
**$^{13}$C-NMR Spectrum of 4b**



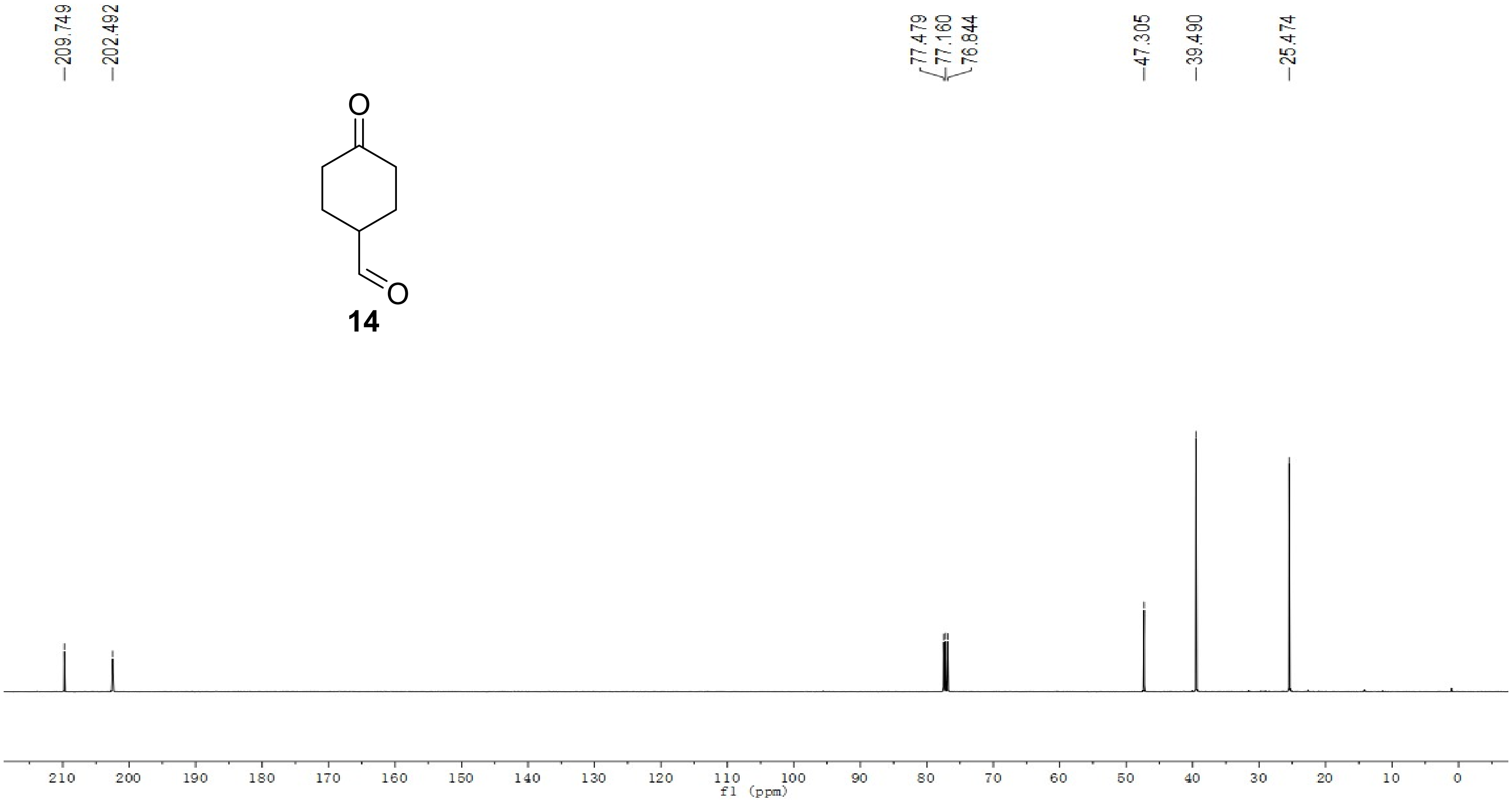
$^1$H-NMR Spectrum of 4c



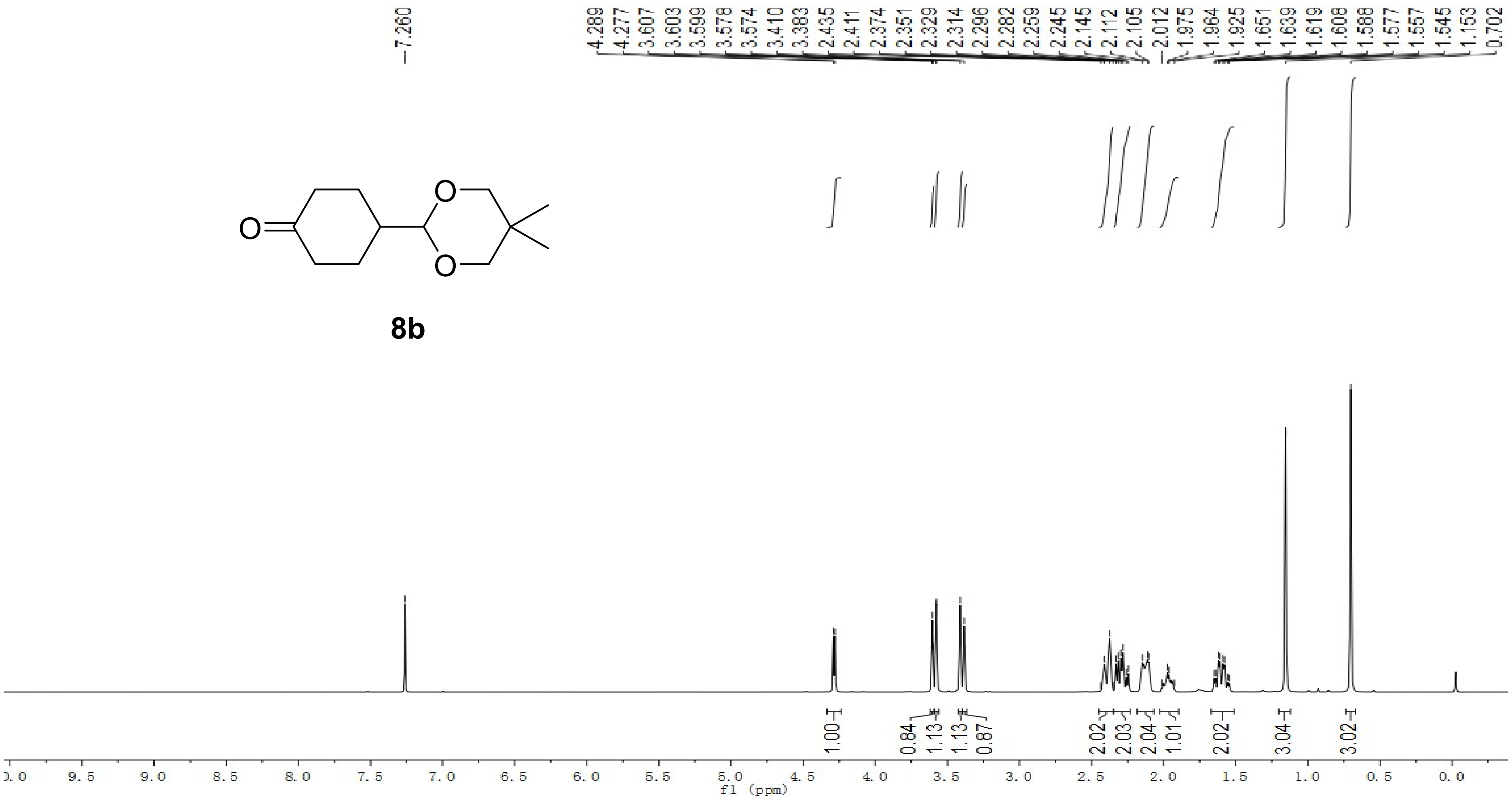
$^{13}$C-NMR Spectrum of 4c



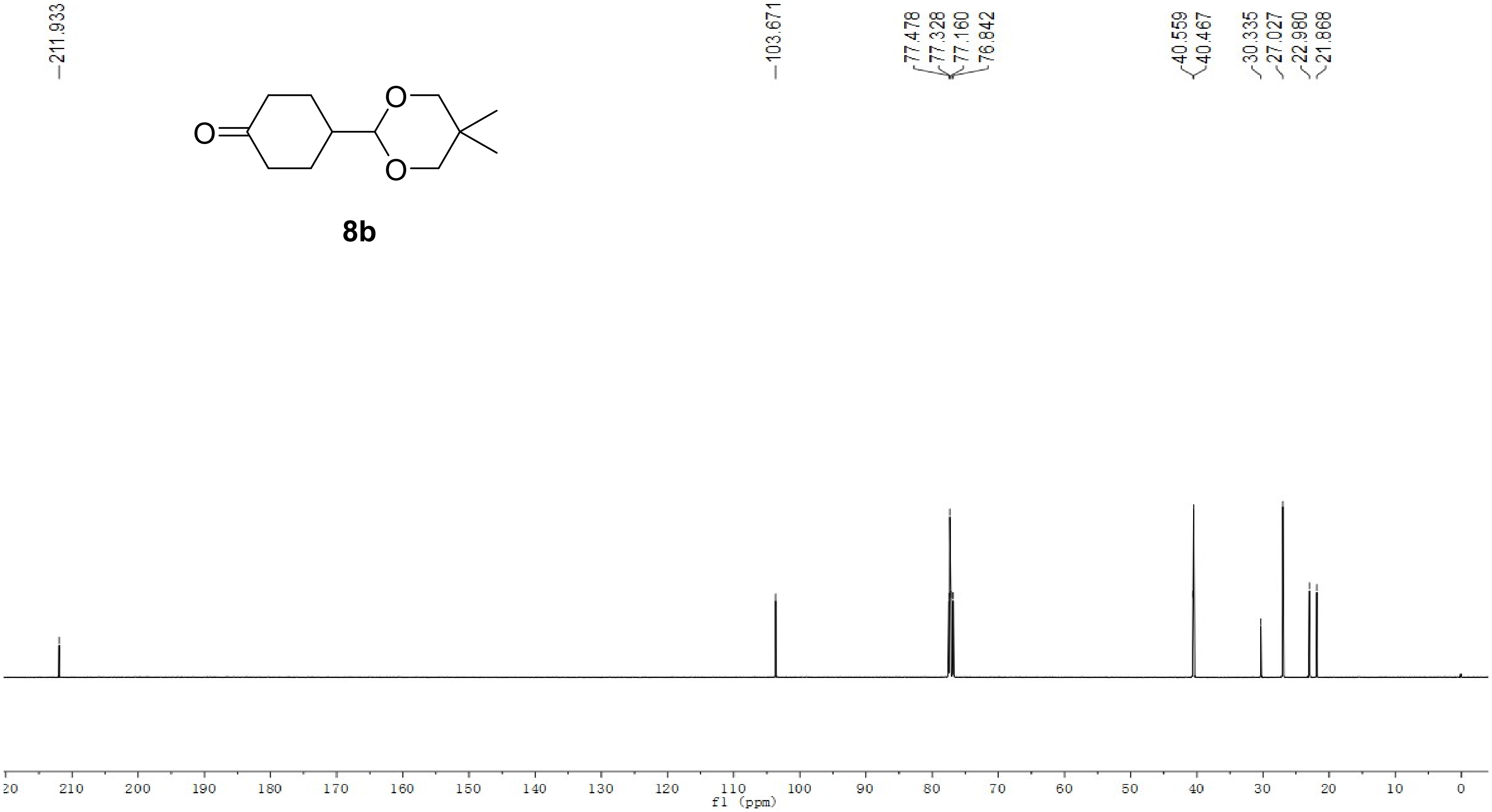
**$^1$H-NMR Spectrum of 14**



$^{13}$C-NMR Spectrum of 14

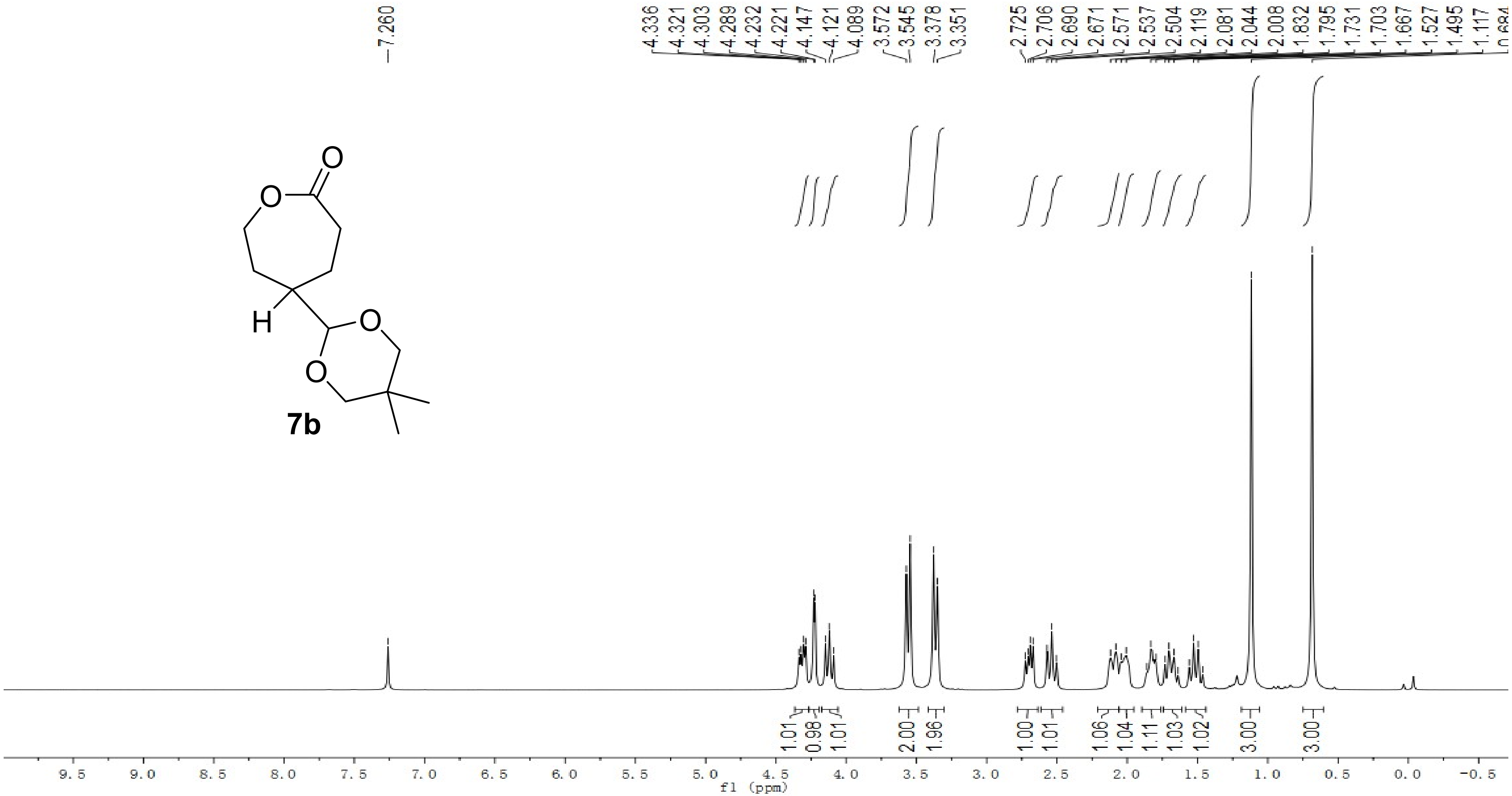


**$^1$H-NMR Spectrum of 8b**

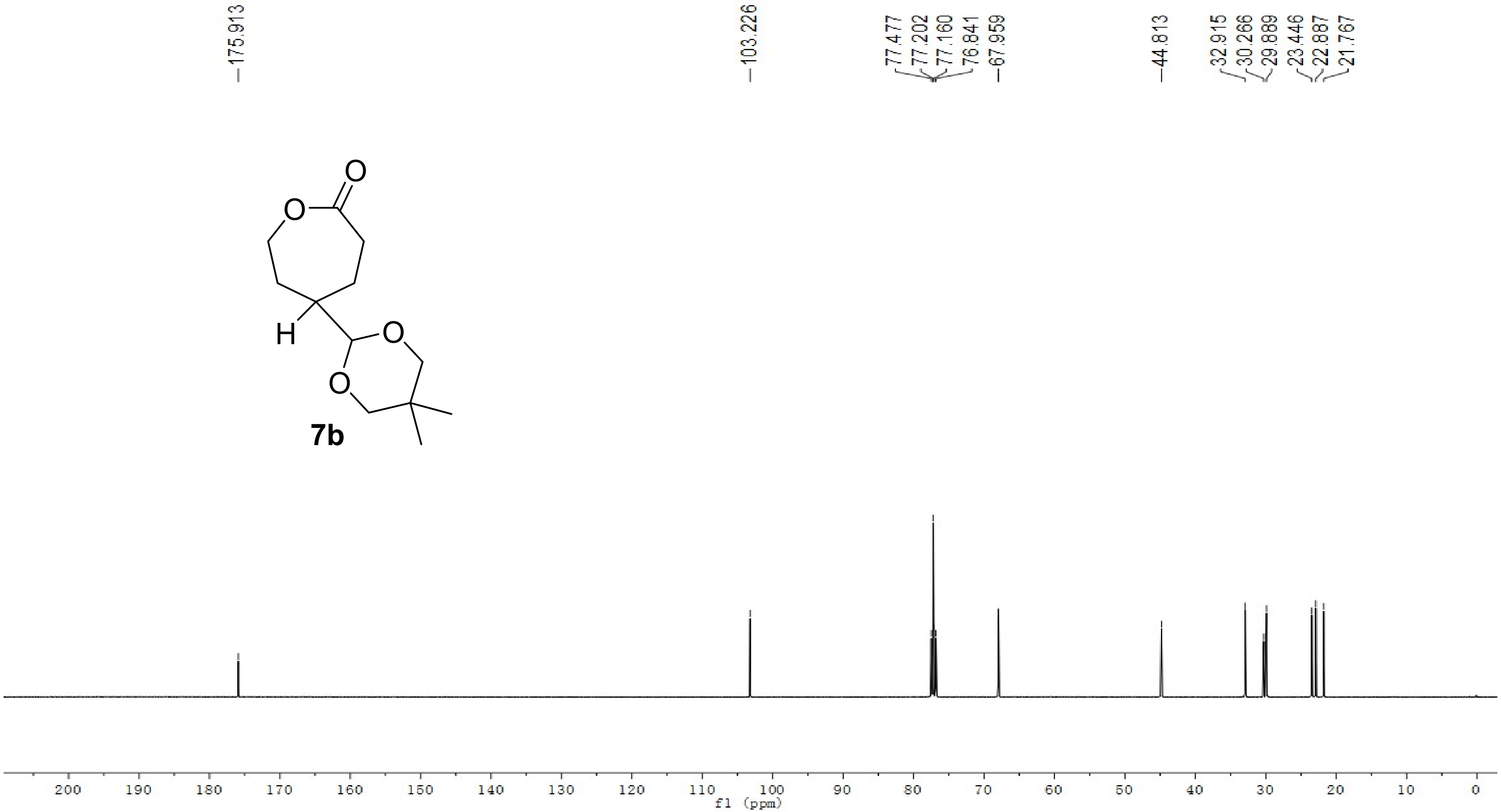


$^{13}$C-NMR Spectrum of 8b

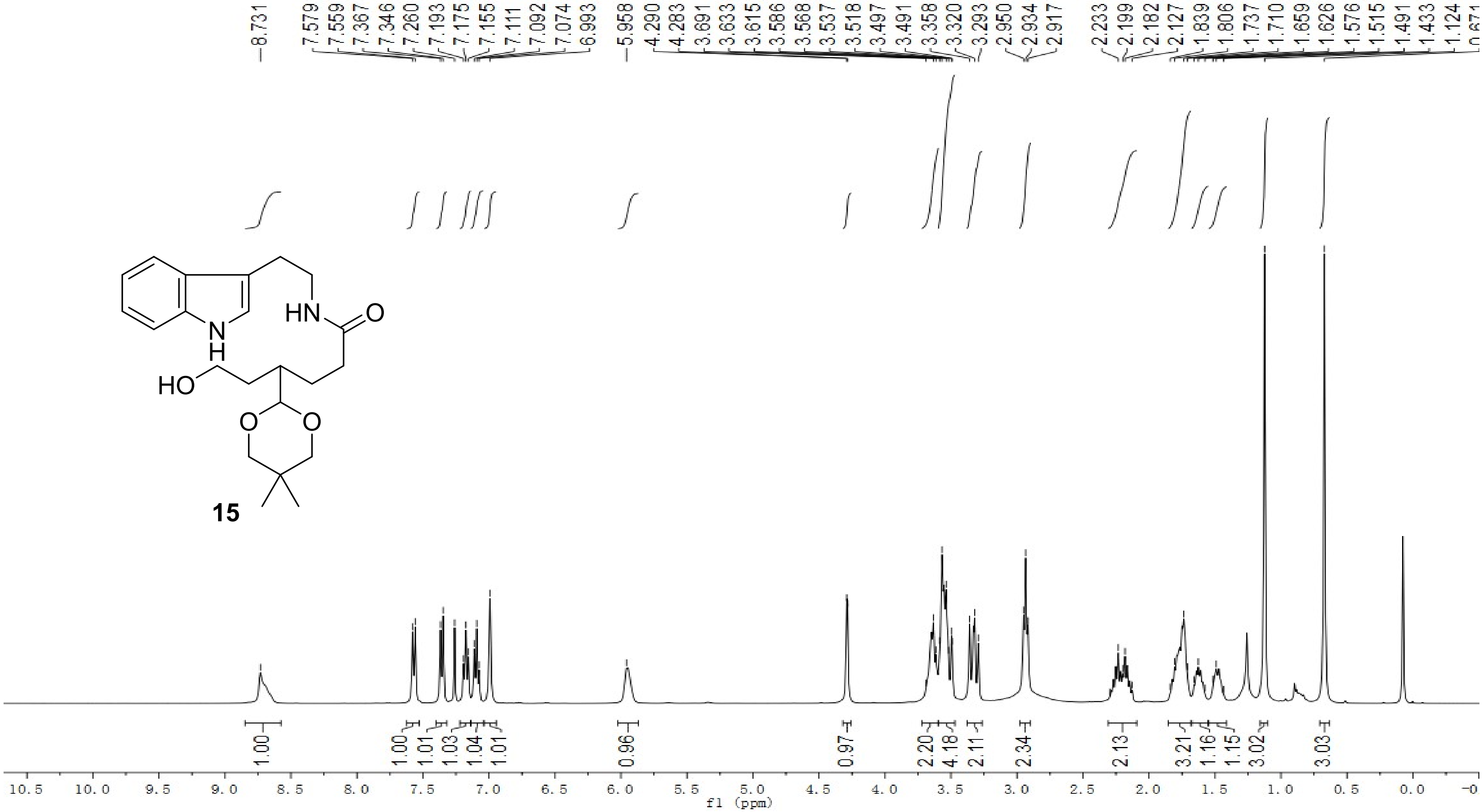
**7b**



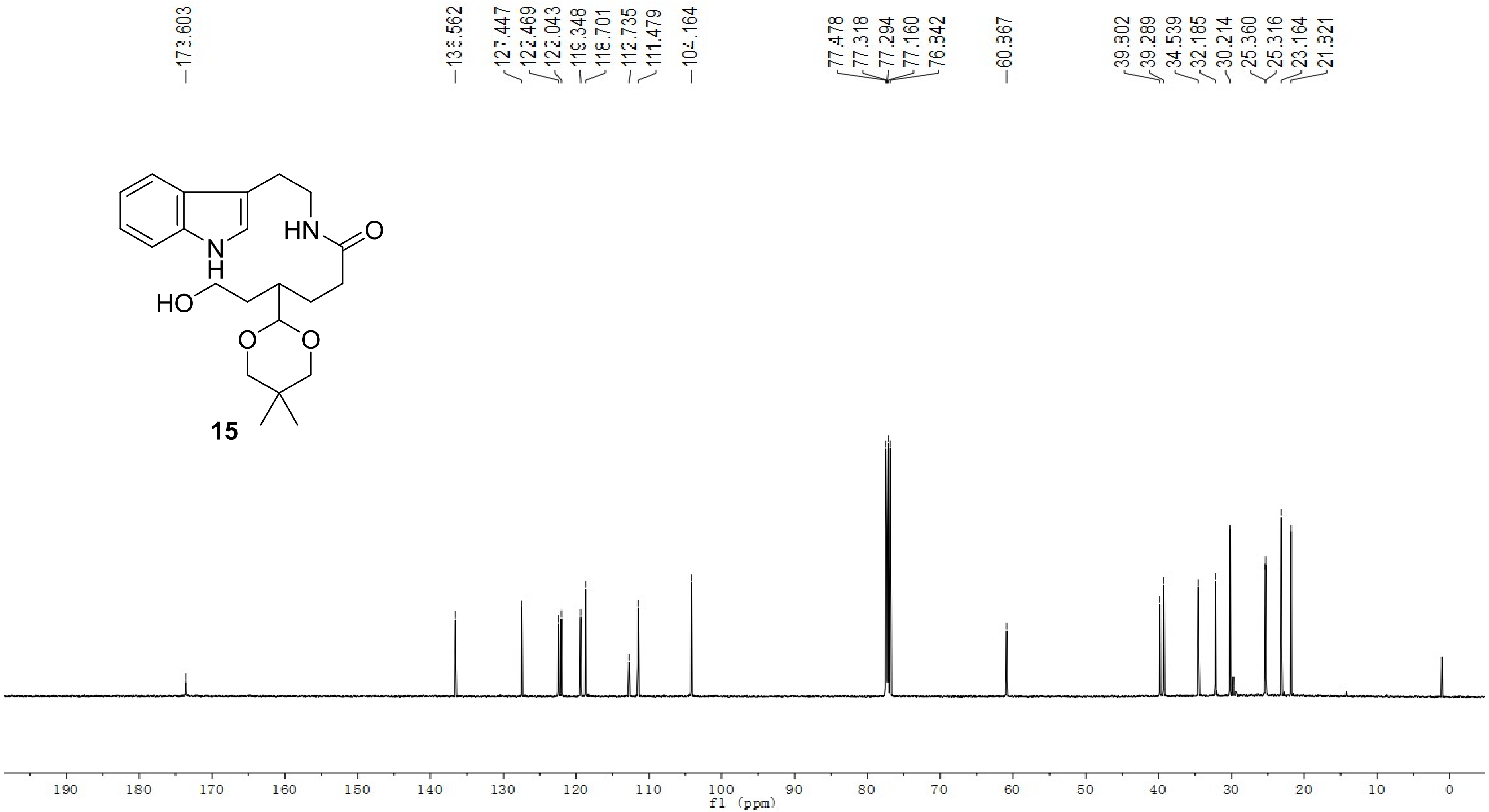
**$^1$H-NMR Spectrum of 7b**



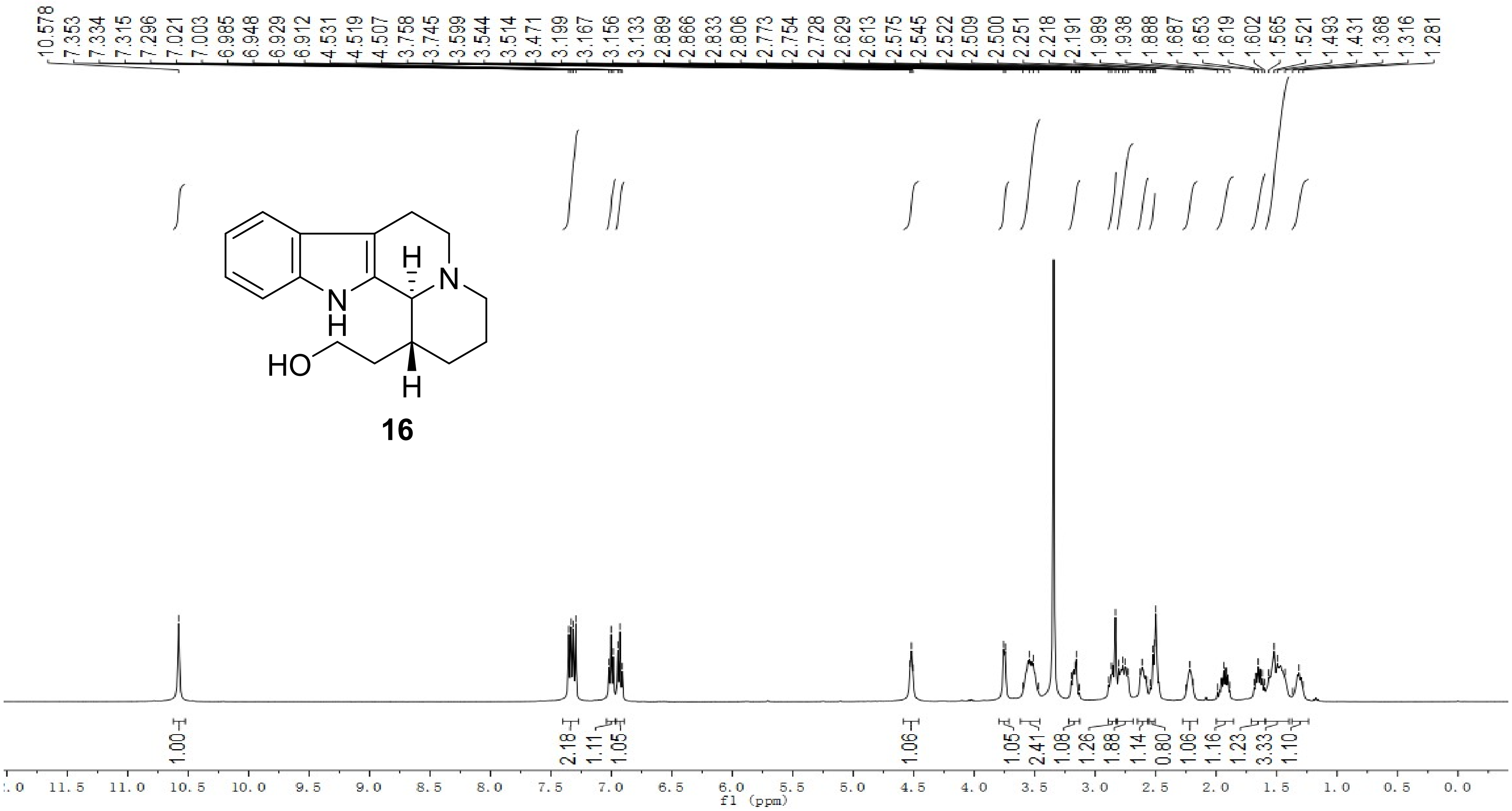
$^{13}$C-NMR Spectrum of 7b



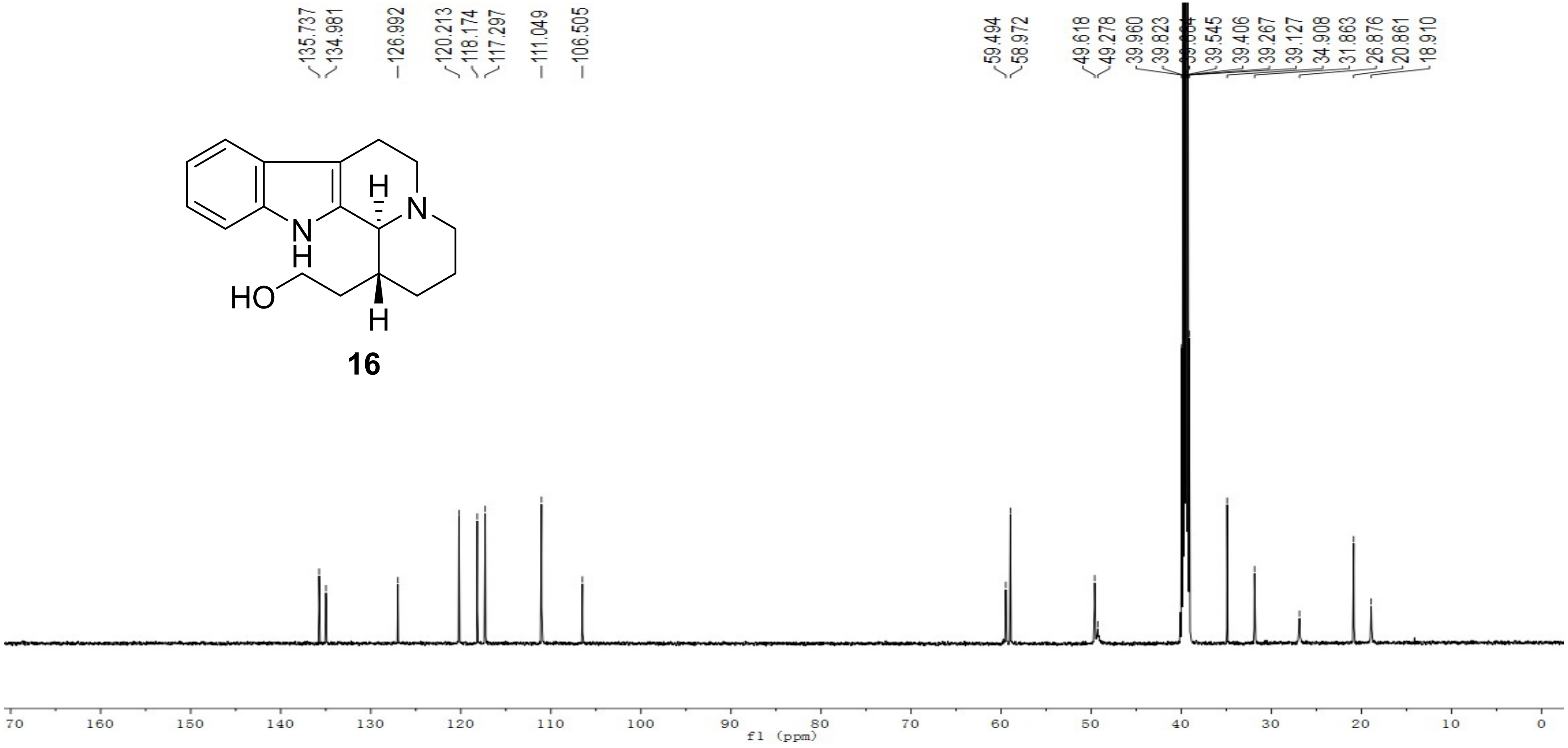
**$^1$H-NMR Spectrum of 15**



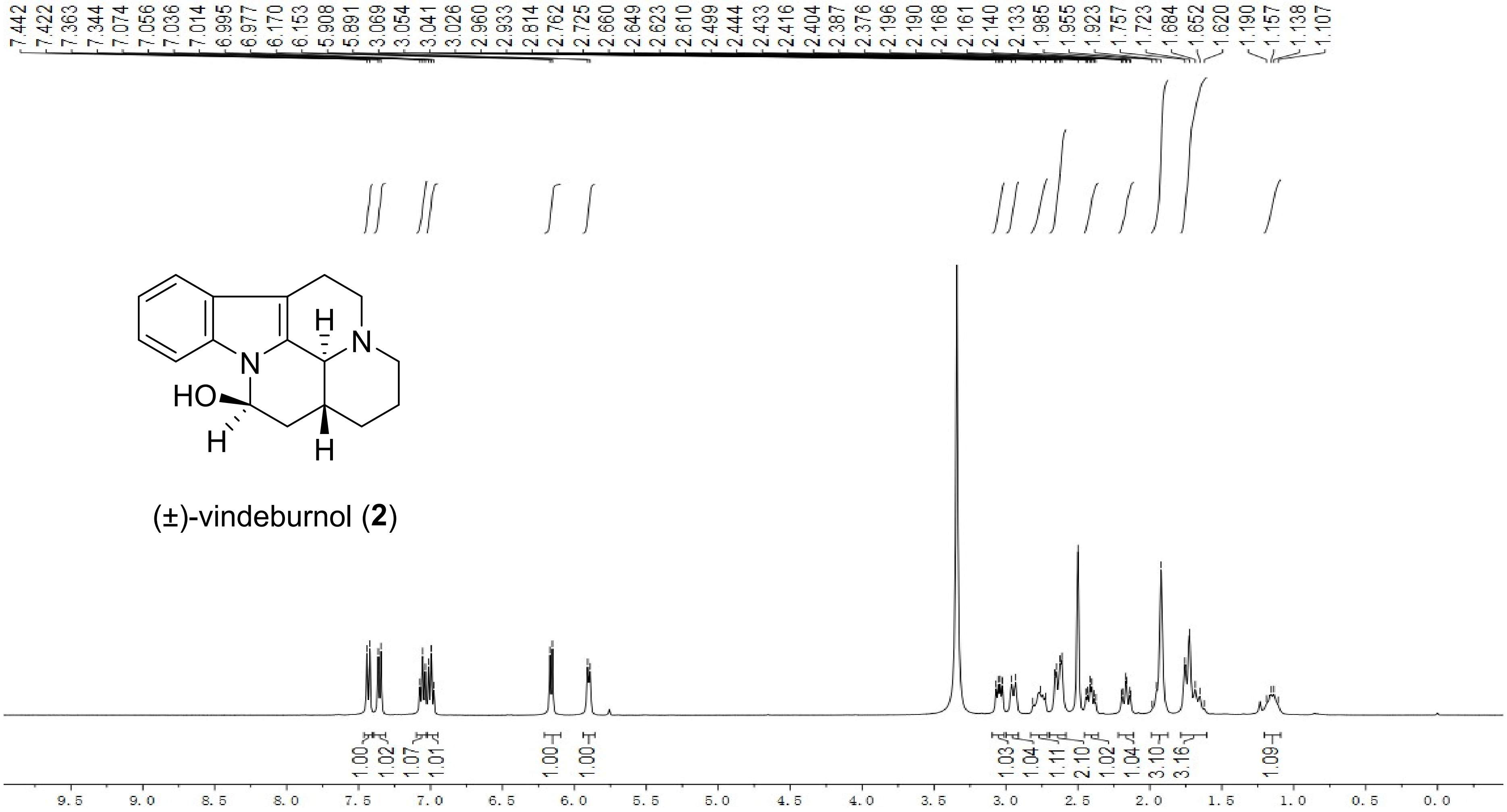
**$^{13}$C-NMR Spectrum of 15**



**$^1$H-NMR Spectrum of 16**

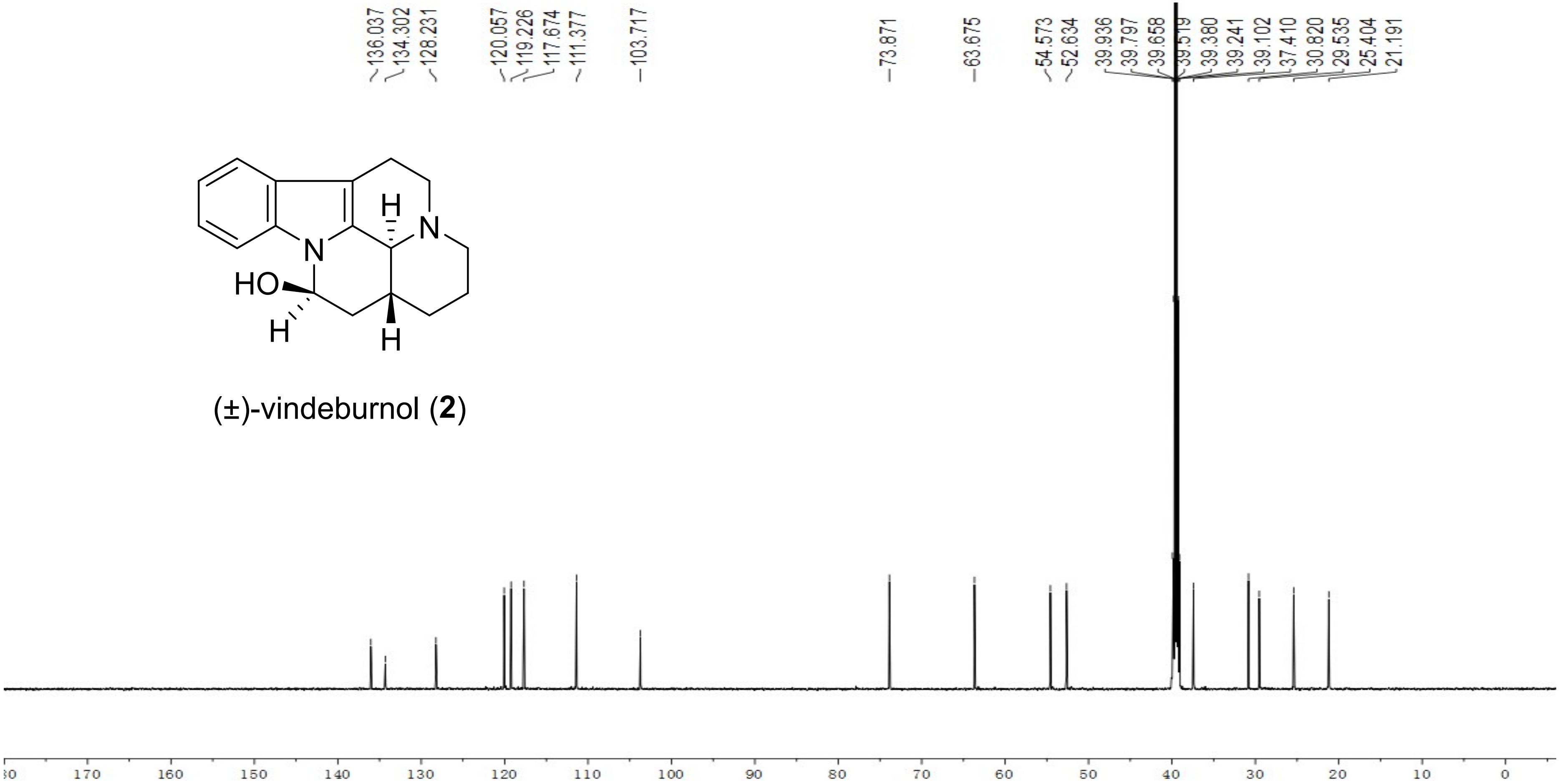


$^{13}$C-NMR Spectrum of 16

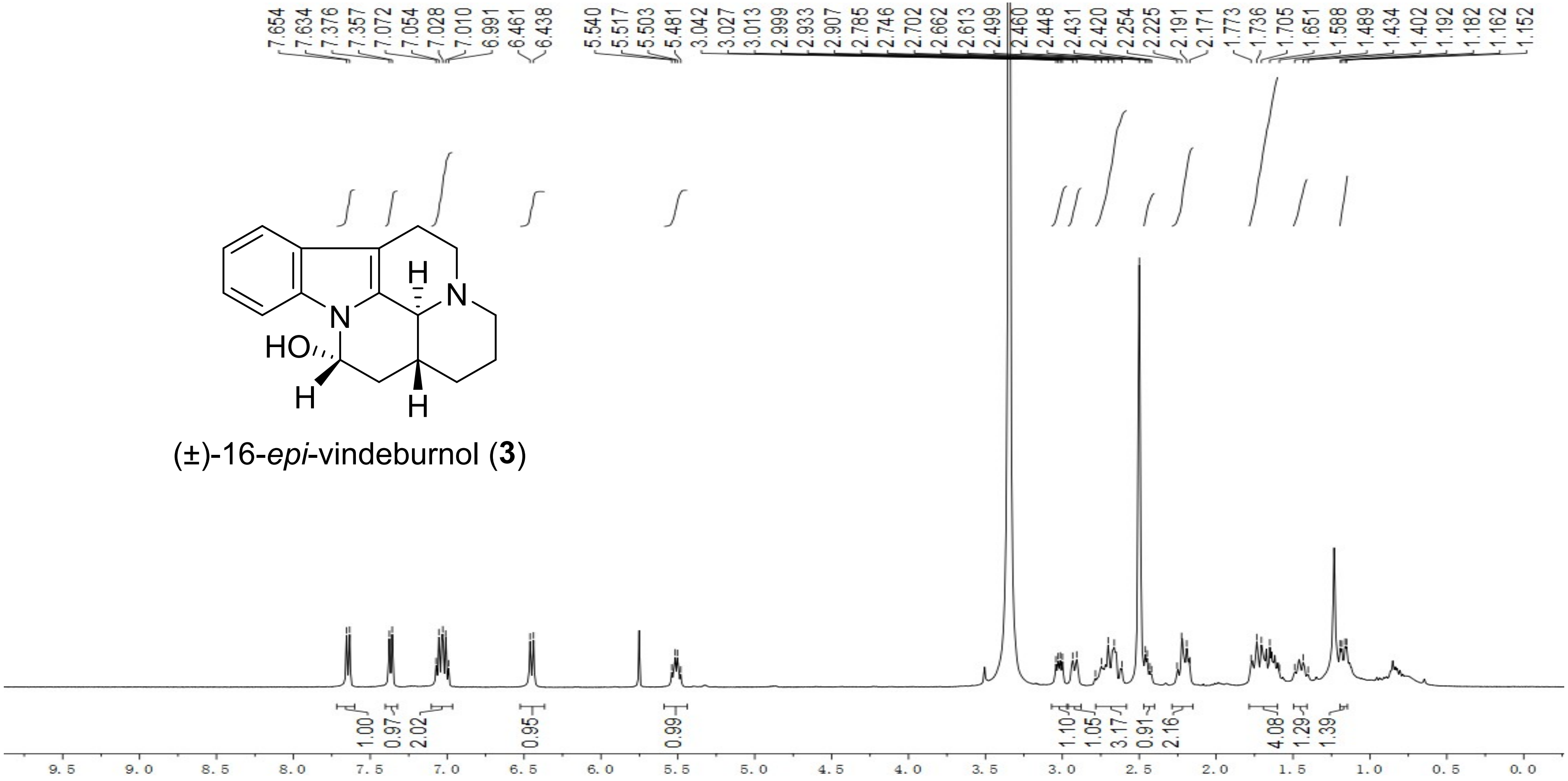


(±)-vindeburnol (**2**)

**$^1$H-NMR Spectrum of (±)-vindeburnol (2)**

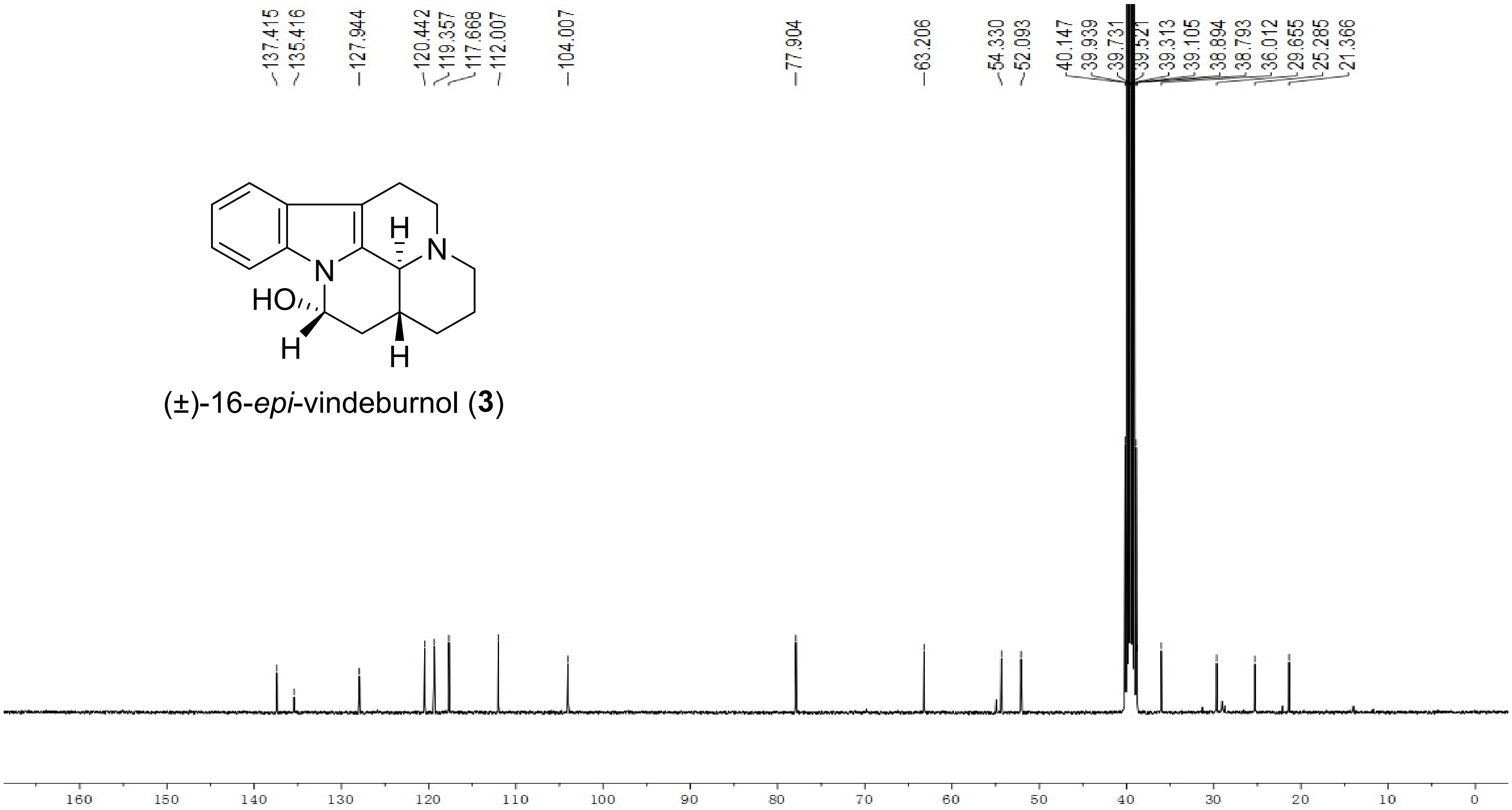


**$^{13}$C-NMR Spectrum of (±)-vindeburnol (2)**

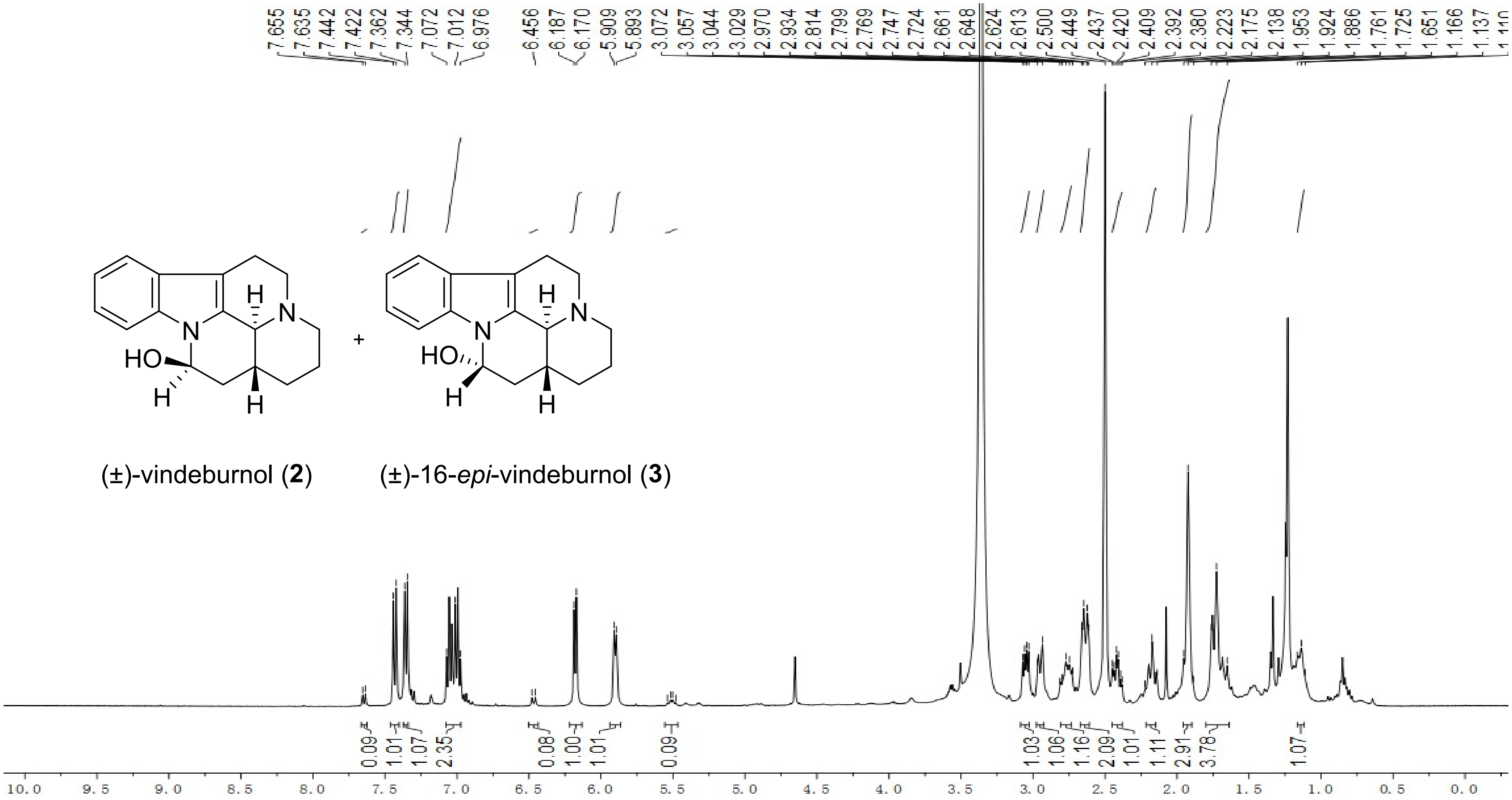


(±)-16-*epi*-vindeburnol (**3**)

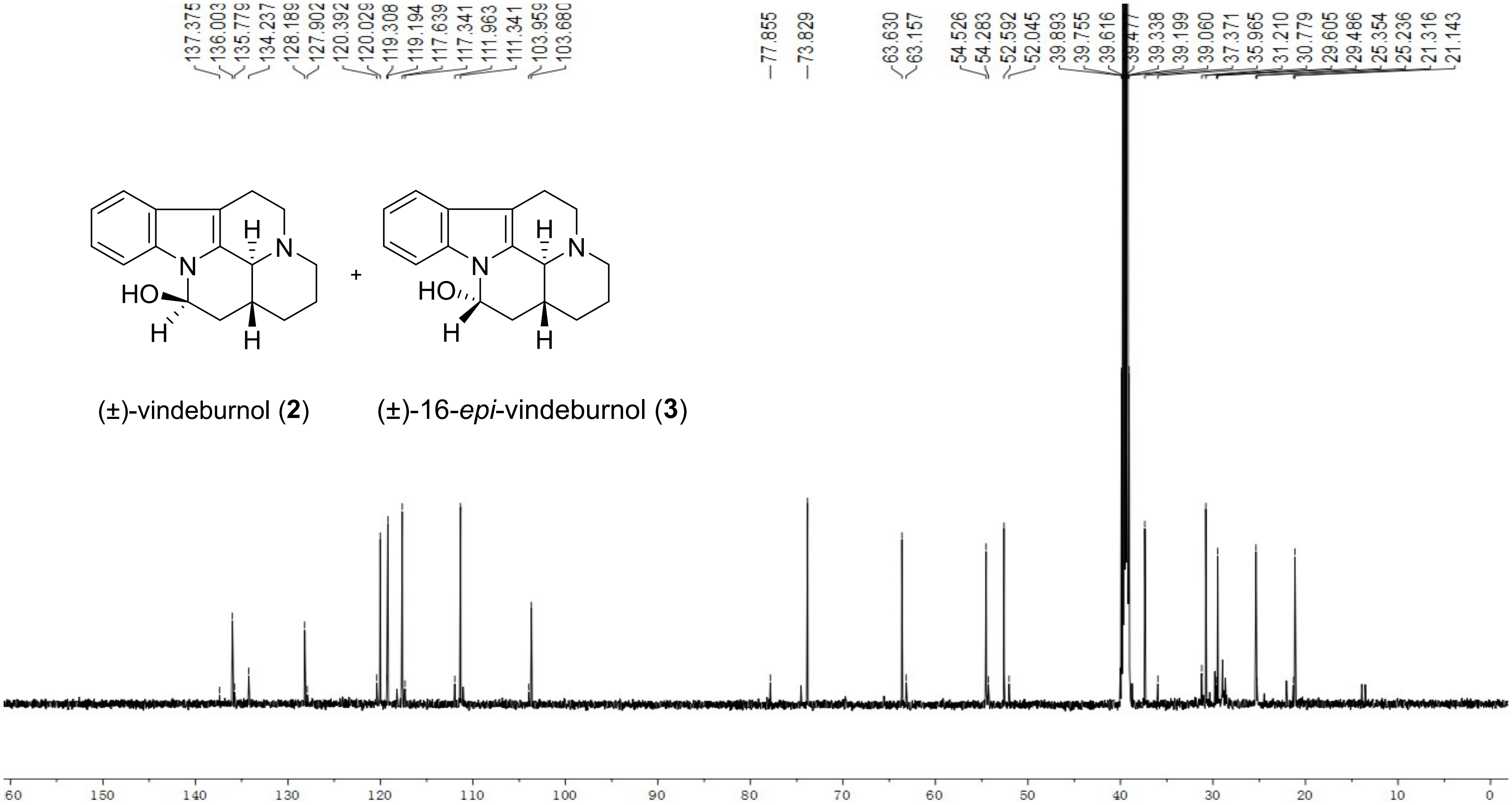
**$^1$H-NMR Spectrum of Compound 16-*epi*-(±)-vindeburnol (3)**



**$^{13}$C-NMR Spectrum of Compound 16-*epi*-(±)-vindeburnol (3)**



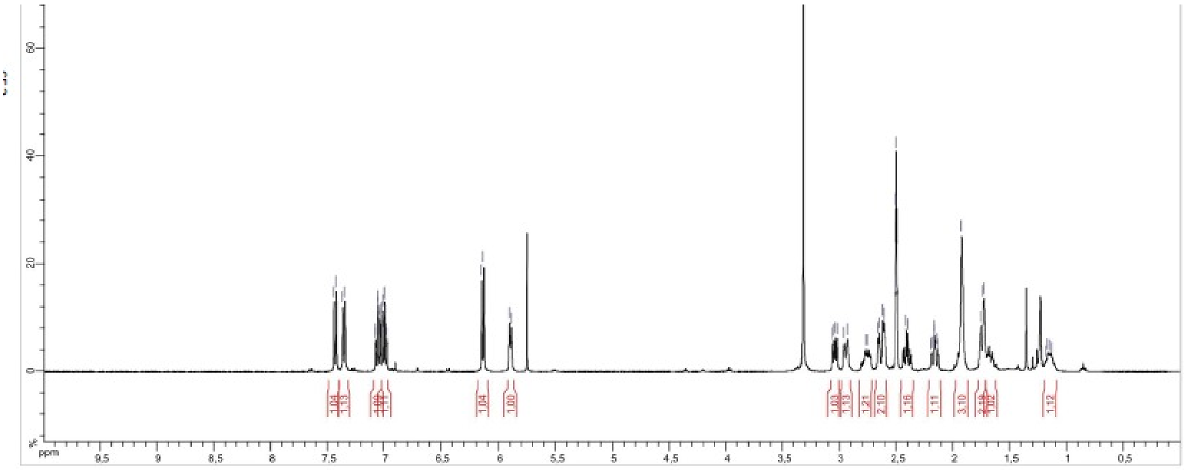
**$^1$H-NMR Spectrum of Mixture of Compound (±)-vindeburnol (2)/16-*epi*-(±)-vindeburnol (3) (2:3 = 13:1)**



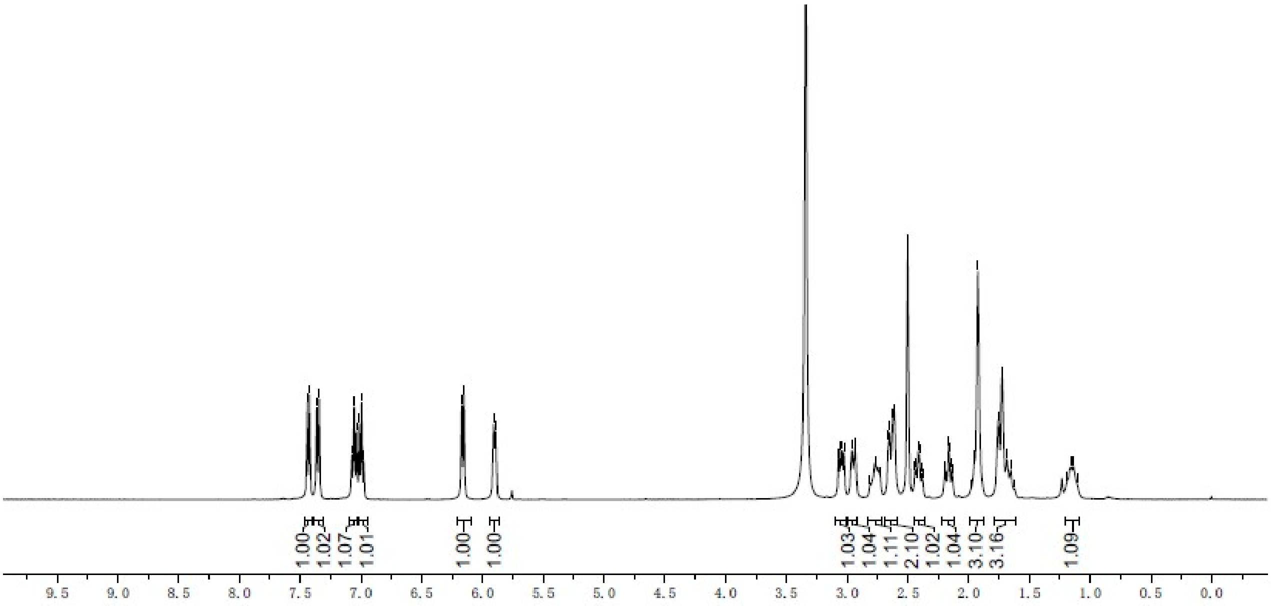
**$^{13}$C-NMR Spectrum of Mixture of Compound (±)-vindeburnol (2) and16-*epi*-(±)-vindeburnol (3) (2:3 = 13:1)**

## Comparison of $^1$H spectrums between the synthesized (±)-vindeburnol (2) by Prof. Girard and the synthesized 2 by Prof. Chen.

### The synthesized (±)-vindeburnol by Prof. Girard

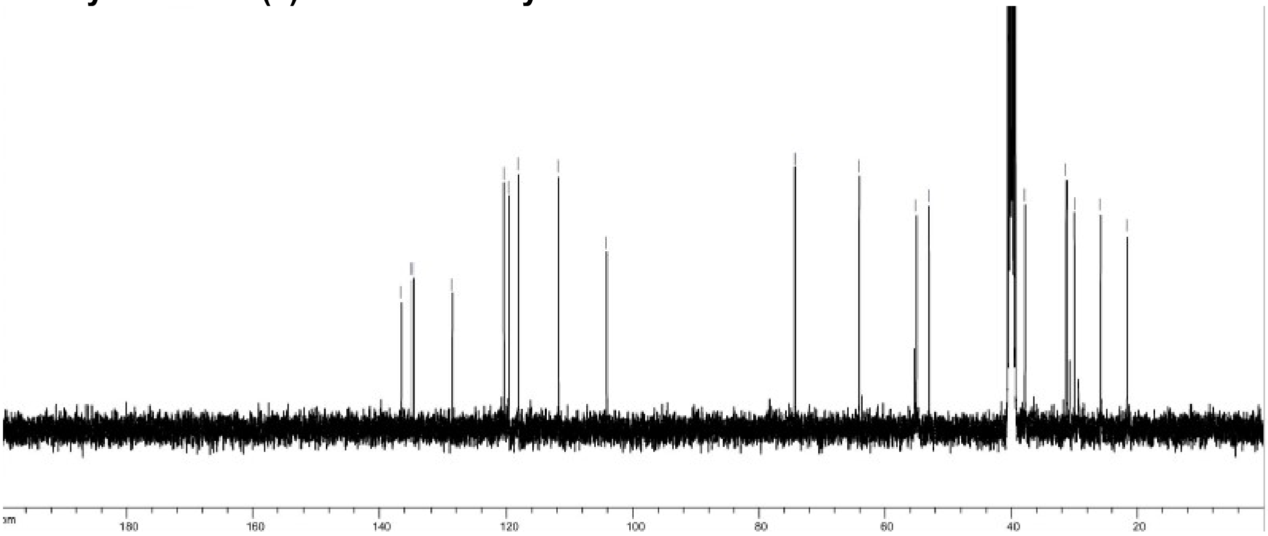


### The synthesized (±)-vindeburnol by Prof. Chen

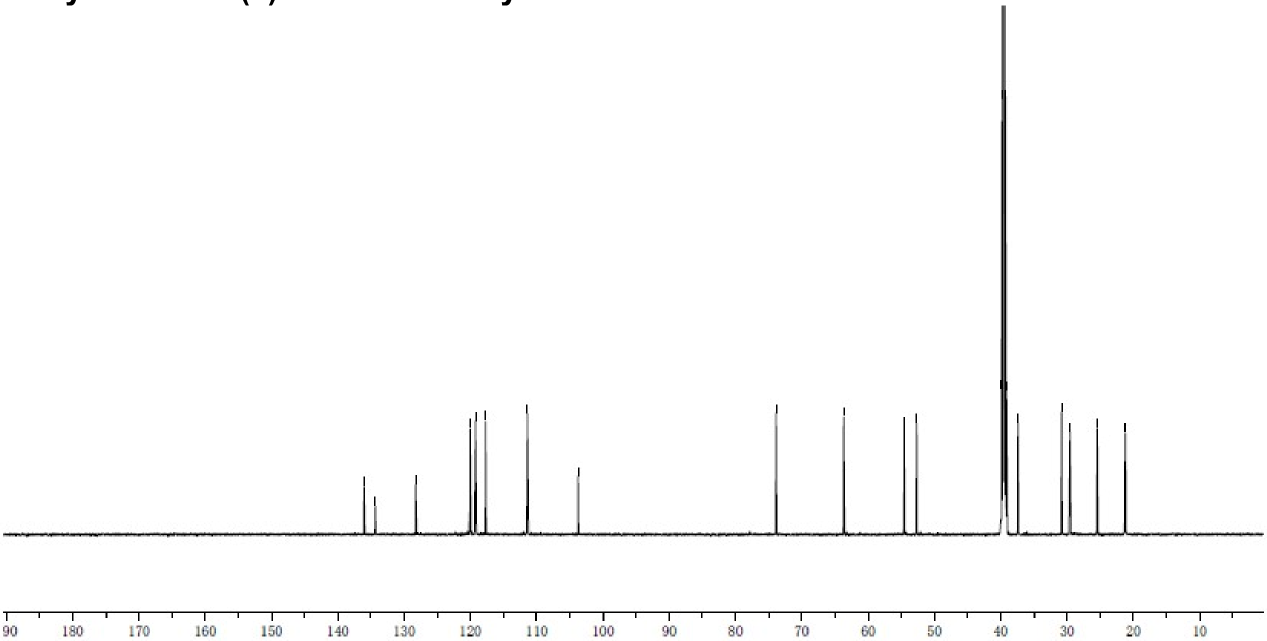


## Comparison of $^{13}$C spectrums between the synthesized (±)-vindeburnol (2) by Prof. Girard and the synthesized 2 by Prof. Chen.

### The synthesized (±)-vindeburnol by Prof. Girard

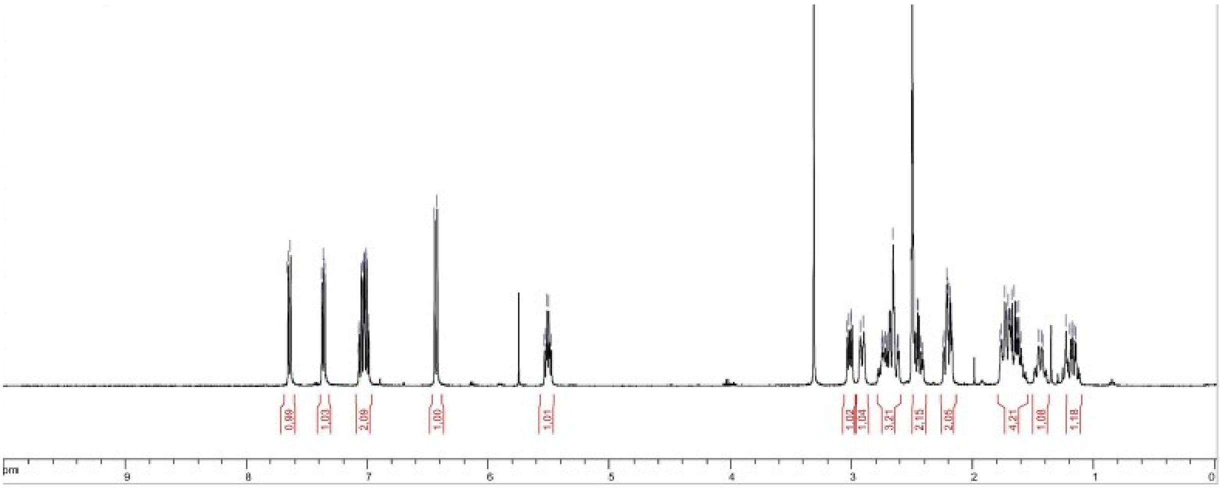


### The synthesized (±)-vindeburnol by Prof. Chen

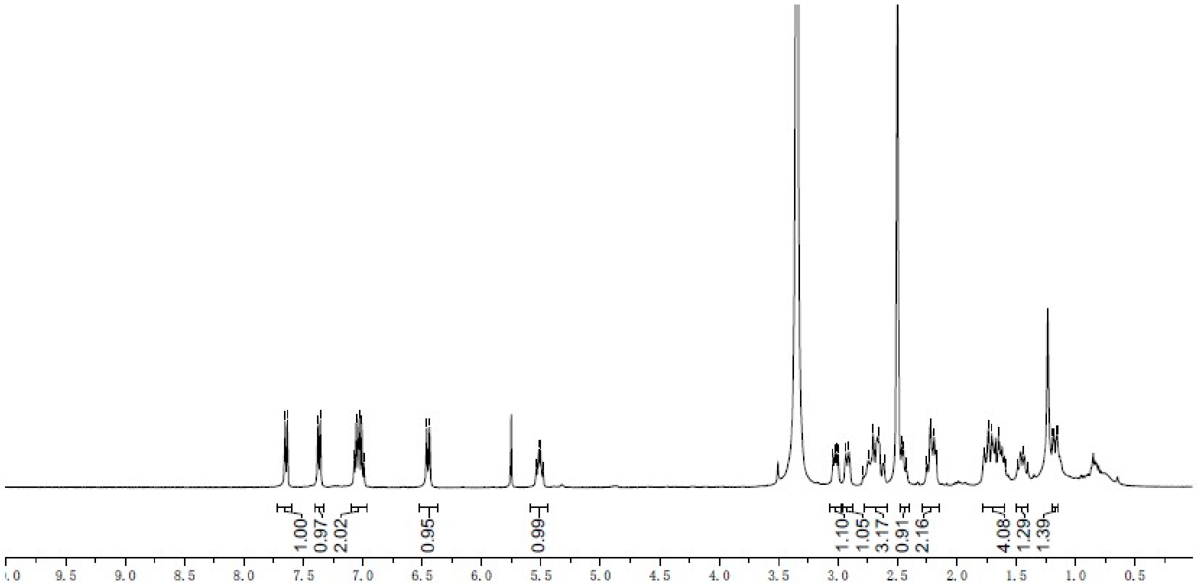


## Comparison of $^1$H spectrums between the synthesized 16-*epi*-(±)-vindeburnol (3) by Prof. Girard and the synthesized 3 by Prof. Chen.

### The synthesized 16-*epi*-(±)-vindeburnol (3) by Prof. Girard

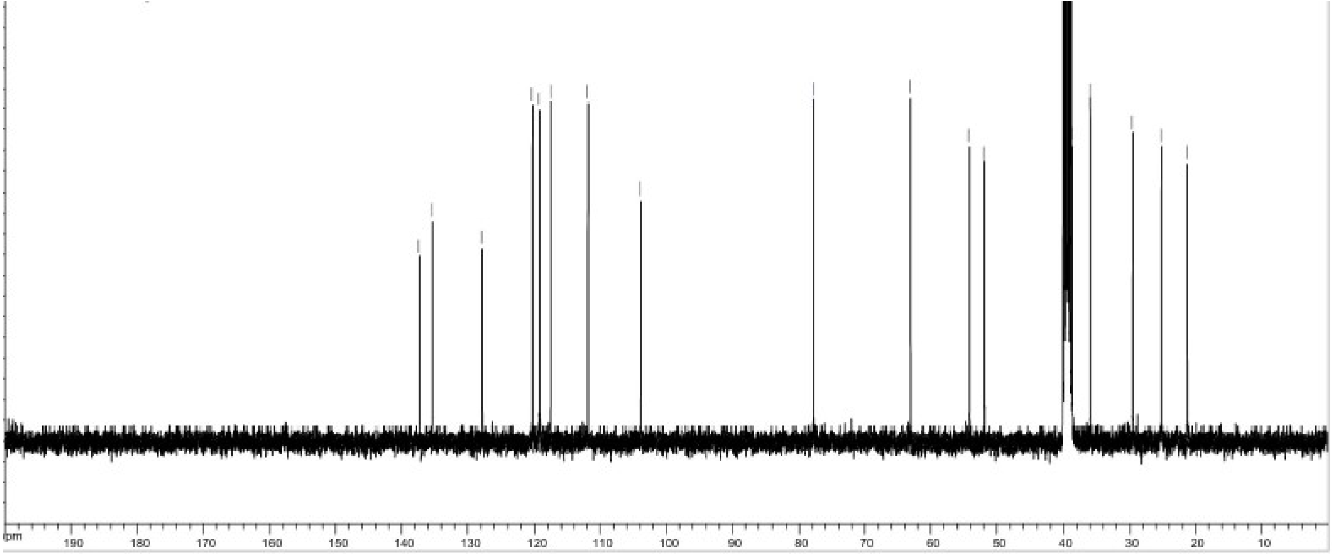


### The synthesized 16-*epi*-(±)-vindeburnol (3) by Prof. Chen

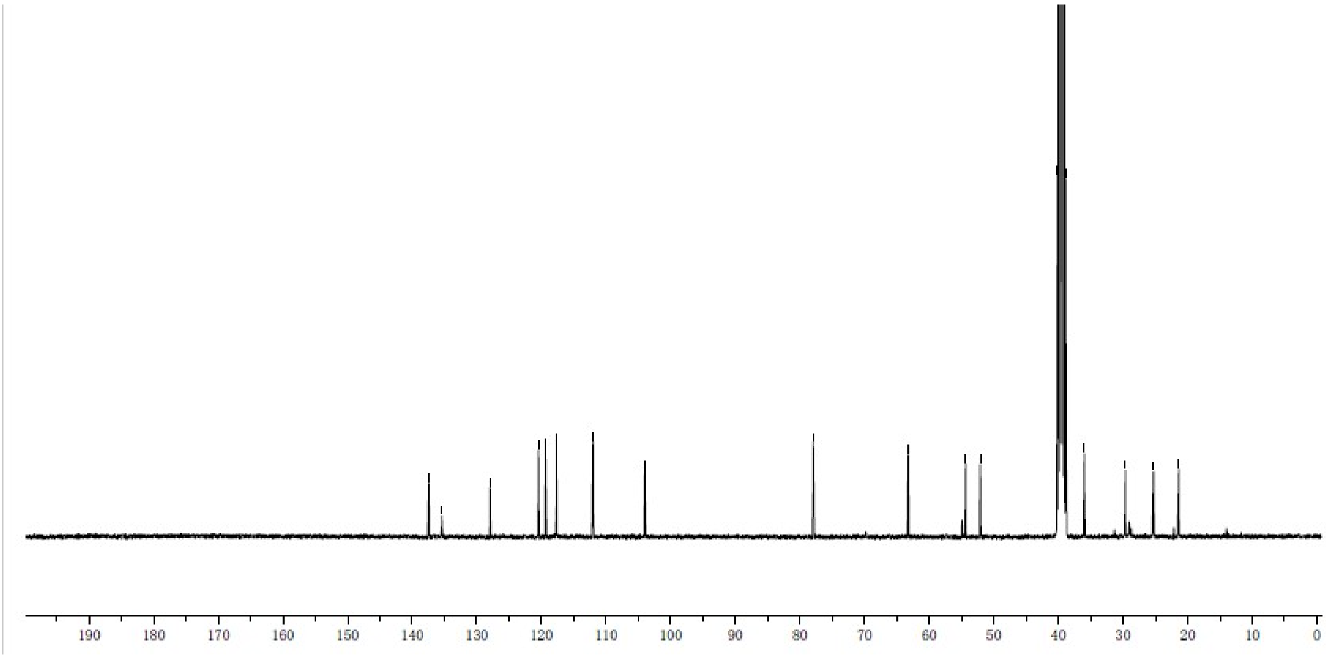


## Comparison of $^{13}$C spectrums between the synthesized 16-*epi*-(±)-vindeburnol (3) by Prof. Girard and the synthesized 3 by Prof. Chen.

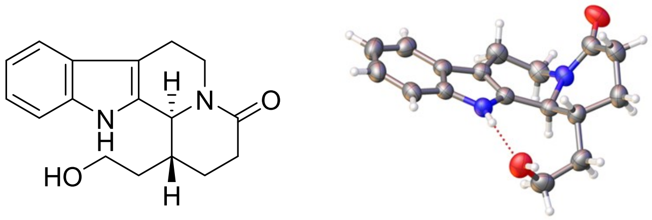
### The synthesized 16-*epi*-(±)-vindeburnol (3) by Prof. Girard



### The synthesized 16-*epi*-(±)-vindeburnol (3) by Prof. Chen



# Crystallographic Data



4a

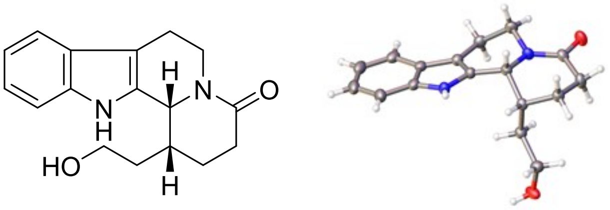
X-Ray of **4a**

**Figure 1**. ORTEP of the molecular structure of **4a**

CCDC 2103051 contains the supplementary crystallographic data for compound **4a**

| | |
|---|---|
| Empirical formula | $C_{17}H_{20}N_2O_2.H_2O$ |
| Formula weight | 284.35 |
| Temperature/K | 297.1(5) |
| Crystal system | monoclinic |
| Space group | $P2_1/n$ |
| a/Å | 10.1352(3) |
| b/Å | 18.7726(5) |
| c/Å | 15.8738(5) |
| α/° | 90 |
| β/° | 103.950(3) |
| γ/° | 90 |
| Volume/Å$^3$ | 2931.14(15) |
| Z | 8 |
| $\rho_{calc}$g/cm$^3$ | 1.289 |
| μ/mm$^{-1}$ | 0.681 |
| F(000) | 1216.0 |
| Crystal size/mm$^3$ | 0.5 × 0.45 × 0.2 |
| Radiation | CuKα (λ = 1.54184) |
| 2θ range for data collection/° | 7.424 to 142.708 |
| Index ranges | $-12 \le h \le 12, -22 \le k \le 13, -19 \le l \le 19$ |
| Reflections collected | 13940 |
| Independent reflections | 5563 [$R_{int}$ = 0.0410, $R_{sigma}$ = 0.0357] |
| Data/restraints/parameters | 5563/0/381 |
| Goodness-of-fit on F$^2$ | 1.028 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0565, $wR_2$ = 0.1488 |
| Final R indexes [all data] | $R_1$ = 0.0654, $wR_2$ = 0.1611 |
| Largest diff. peak/hole / e Å$^{-3}$ | 0.24/-0.27 |

These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



4b

X-Ray of **4b**

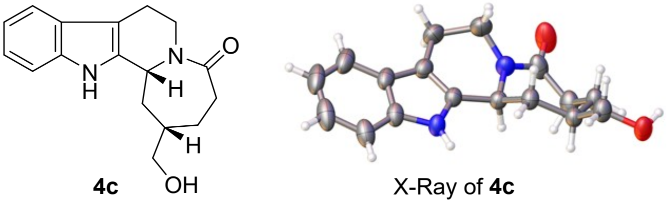
**Figure 2**. ORTEP of the molecular structure of **4b**

CCDC 2103479 contains the supplementary crystallographic data for compound **4b**.

| | |
|---|---|
| Empirical formula | $C_{17}H_{20}N_2O_2$ |
| Formula weight | 284.35 |
| Temperature/K | 150.00(10) |
| Crystal system | monoclinic |
| Space group | $P2_1/c$ |

| | |
|---|---|
| a/Å | 6.52889(15) |
| b/Å | 26.9565(6) |
| c/Å | 9.50589(19) |
| α/° | 90 |
| β/° | 104.117(2) |
| γ/° | 90 |
| Volume/Å$^3$ | 1622.47(6) |
| Z | 4 |
| $\rho_{calc}$g/cm$^3$ | 1.164 |
| μ/mm$^{-1}$ | 0.615 |
| F(000) | 608.0 |
| Crystal size/mm$^3$ | 0.5 × 0.3 × 0.05 |
| Radiation | CuKα (λ = 1.54184) |
| 2θ range for data collection/° | 10.14 to 142.726 |
| Index ranges | $-7 \le h \le 8$, $-32 \le k \le 28$, $-10 \le l \le 11$ |
| Reflections collected | 9153 |
| Independent reflections | 3096 [$R_{int}$ = 0.0461, $R_{sigma}$ = 0.0396] |
| Data/restraints/parameters | 3096/0/195 |
| Goodness-of-fit on F$^2$ | 1.062 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0533, $wR_2$ = 0.1467 |
| Final R indexes [all data] | $R_1$ = 0.0585, $wR_2$ = 0.1525 |
| Largest diff. peak/hole / e Å$^{-3}$ | 0.24/-0.26 |

These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



**4c**

X-Ray of **4c**

**Figure 3**. ORTEP of the molecular structure of **4c**

CCDC 2103481 contains the supplementary crystallographic data for compound **4c**

| | |
|---|---|
| Empirical formula | $C_{17}H_{20}N_2O_2$ |
| Formula weight | 284.35 |
| Temperature/K | 297.4(2) |
| Crystal system | monoclinic |
| Space group | P2$_1$/n |
| a/Å | 10.0470(2) |
| b/Å | 11.20580(19) |
| c/Å | 13.1505(3) |
| α/° | 90 |
| β/° | 101.241(2) |
| γ/° | 90 |
| Volume/Å$^3$ | 1452.15(6) |
| Z | 4 |
| $\rho_{calc}$g/cm$^3$ | 1.301 |
| μ/mm$^{-1}$ | 0.687 |
| F(000) | 608.0 |
| Crystal size/mm$^3$ | 0.4 × 0.3 × 0.3 |
| Radiation | CuKα (λ = 1.54184) |
| 2θ range for data collection/° | 10.178 to 142.792 |
| Index ranges | $-11 \le h \le 12$, $-13 \le k \le 8$, $-14 \le l \le 15$ |
| Reflections collected | 7040 |
| Independent reflections | 2768 [$R_{int}$ = 0.0349, $R_{sigma}$ = 0.0338] |

| | |
|---|---|
| Data/restraints/parameters | 2768/0/191 |
| Goodness-of-fit on $F^2$ | 1.060 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0648, $wR_2$ = 0.1612 |
| Final R indexes [all data] | $R_1$ = 0.0711, $wR_2$ = 0.1710 |
| Largest diff. peak/hole / e Å$^{-3}$ | 0.26/-0.42 |

These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif