Supplementary Information of

Nine-Step Total Synthesis of (–)-Strychnofoline

Qingzhen Yu [a,†], Pan Guo [a,†], Jie Jian [a,†], Yuye Chen [a,b] and Jing Xu [a]

a. Department of Chemistry, Southern University of Science and Technology, Shenzhen, Guangdong Province, China; and SUSTech, Grubbs Institute, Shenzhen, Guangdong Province, China.
b. State Key Laboratory of Quality Research in Chinese Medicine, Institute of Chinese Medical Sciences, University of Macau, Macau, China.

† These authors contributed equally to this work.

Table of Contents

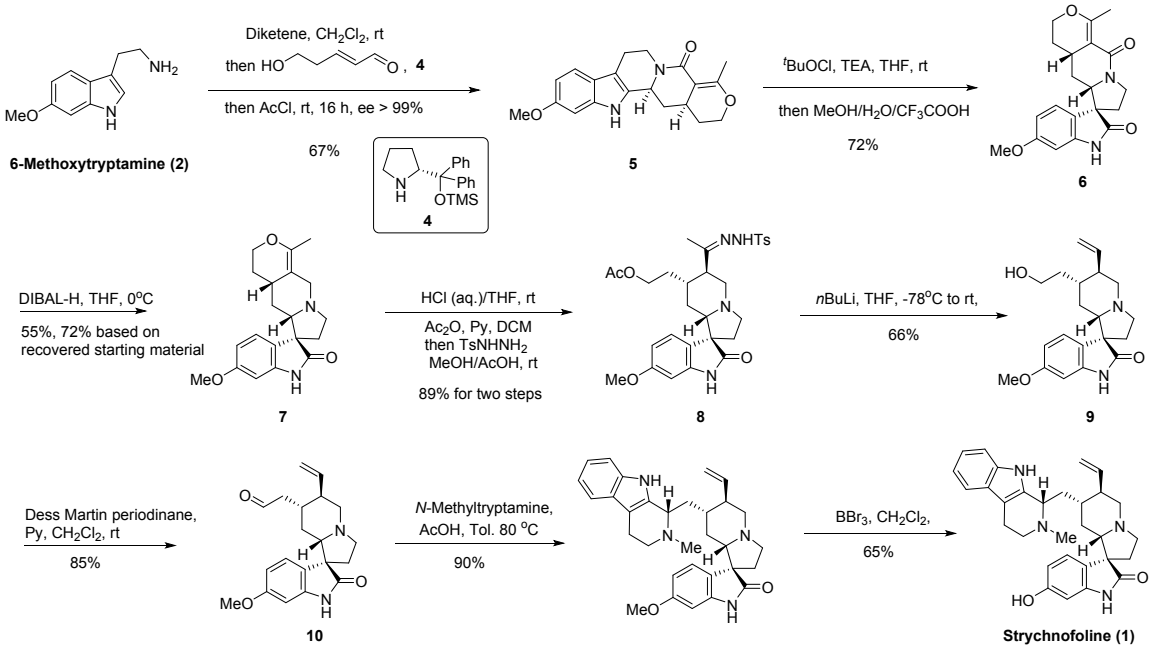
I Experimental Procedures and Spectroscopic Data of Compounds

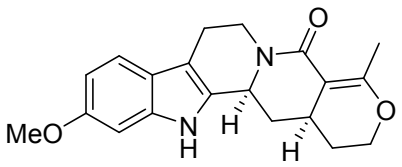
General Procedures

Unless indicated, all commercial available reagents and anhydrous solvents were purchased at the highest commercial quality and were used as received without further purification. All non-aqueous reactions were carried out under argon atmosphere using dry glassware that had been flame-dried under a stream of argon unless otherwise noted. Flash column chromatography was performed on silica gel (Qingdao Haiyang Chemical Co., Ltd., 200-300 meshes) using hexane-EtOAc mixtures of increasing polarity.

The progress of all the reactions was monitored by thin-layer chromatography (TLC) using UV light as a visualizing agent and aqueous ammonium cerium nitrate/ammonium molybdate or basic aqueous potassium permanganate as a developing agent. ^{1}H NMR and ^{13}C NMR spectra were recorded on either 400 MHz/500 MHz Bruker instruments. Chemical shifts (δ) are quoted in parts per million (ppm) referenced to the appropriate residual solvent peak ($CDCl_3$ or d_6-acetone), with the abbreviations s, d, dd, dt, t, td, q, and m denoting singlet, doublet, double doublet, triplet, triple doublet, quartet and multiplet respectively. J = coupling constants given in Hertz (Hz). High resolution Mass spectra (HRMS) were recorded on a Thermo ScientificTM Q-exactive hybrid quadrupole-orbitrap mass spectrometer. Optical rotation data were collected on an Autopol automatic polarimeter (Rudolph Research Analytical) using HPLC grade anhydrous $CHCl_3$. Chiral HPLC analyses were performed on an Agilent 1260 Series using a Daicel Chiralpak (AD-H, OD-H) column with hexanes/iPrOH as the eluent. Melting points (m.p.) were uncorrected and were recorded on a SGW X-4 apparatus.

Full Reaction Sequence for the Total Synthesis of (-)-Strychnofoline

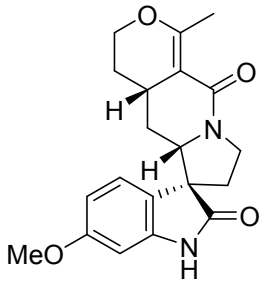




5

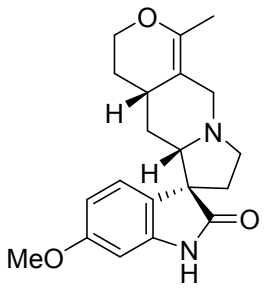
Preparation of cyclization product 5. To a solution of 6-methoxytryptamine (10.0 g, 52.6 mmol, 1.0 equiv) in DCM (c = 0.2 M) was added diketene (4.9 g, 57.8 mmol, 1.1 equiv) dropwise, and the mixture was stirring for 0.5 h at 0 °C (monitored by TLC). After the consumption of the 6-methoxytryptamine, the catalyst (*R*)-2-(diphenyl((trimethylsilyl)oxy)methyl)pyrrolidine (**4**, 5.1 g, 15.8 mmol, 30 mol%) and (*E*)-5-hydroxypent-2-enal (5.8 g, 57.9 mmol, 1.1 equiv) was added at 0 °C. The mixture was left at room temperature until the ketoamide was completely consumed. To this mixture was added H_2O (4.7 mL, 262.8 mmol, 5.0 equiv) and AcCl (37.2 mL, 526 mmol, 10.0 equiv) at 0 °C, then stirred at room temperature for 0.5 h. Aqueous sat. $NaHCO_3$ was added to quench the reaction and the water phase was extracted with DCM. The combined organic phases were dried (Na_2SO_4), filtered and concentrated under reduced pressure to give the crude product. The resulting dark brown solid was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate, 2:1) to afford **5** (11.5 g, 67%, 99% ee) as a light-yellow solid. $[\alpha]_D^{23} = -240.0°$ (c = 0.20, $CHCl_3$); m.p.: 234 °C (decompose); ^1H NMR (400 MHz, $CDCl_3$) δ 7.67 (s, 1H), 7.37 (d, *J* = 8.6 Hz, 1H), 6.83 (d, *J* = 2.1 Hz, 1H), 6.78 (dd, *J* = 8.6, 2.2 Hz, 1H), 5.25 – 5.11 (m, 1H), 4.85 – 4.73 (m, 1H), 4.24 (ddd, *J* = 10.6, 3.7, 2.3 Hz, 1H), 3.99 – 3.89 (m, 1H), 3.84 (s, 3H), 2.92 – 2.75 (m, 2H), 2.76 – 2.57 (m, 2H), 2.52 –

2.42 (m, 1H), 2.32 (s, 3H), 2.09 – 1.93 (m, 1H), 1.60 – 1.48 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.9, 162.2, 156.5, 137.0, 132.4, 121.4, 119.0, 109.8, 109.1, 103.7, 95.1, 65.55, 55.8, 53.5, 40.2, 35.5, 29.7, 29.1, 21.3, 20.5. HRMS (ESI) m/e 339.1703 $[M+H]^+$ calcd for $C_{20}H_{23}N_2O_3^+$: 339.1709. The enantiomers were separated by HPLC on Chiralcel OD-H column (250x4.6 mm) with Hexane / *i*PrOH as the eluent (85:15), flow rate: 1.0 mL/min): Rt (min): 17.3 (minor); 35.6 (major).



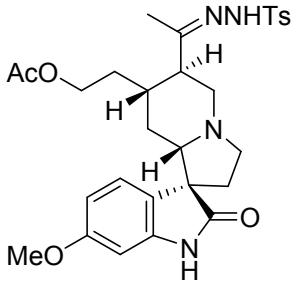
6

Preparation of rearrangement product 6. To a stirring solution of compound **5** (10 g, 29.6 mmol, 1.0 equiv) in DCM (200 mL), Et_3N (12.3 mL, 88.9 mmol, 3.0 equiv) and *t*BuOCl (10.6 mL, 88.9 mmol, 3.0 equiv) were added dropwise successively at 0 °C. The mixture was stirred at room temperature for 0.5 h, then to the mixture were added MeOH, H_2O and TFA (6:3:1, 0.2 M). The resulting mixture was stirred for 1 h, quenched with sat. $NaHCO_3$, extracted with DCM. The combined organic phases were dried (Na_2SO_4), filtered and concentrated under reduced pressure to give the crude product. It was then purified by flash column chromatography on silica gel (petroleum ether/ ethyl acetate, 1.5:1) to afford **6** (7.5 g, 72%) as a yellow solid. $[\alpha]_D^{23}$ = –118.4º (c = 0.20, $CHCl_3$); m.p.: 252 °C (decompose); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (s, 1H), 6.88 (d, *J* = 8.7 Hz, 1H), 6.59 – 6.53 (m, 2H), 4.25 – 4.15 (m, 1H), 4.08 – 3.97 (m, 2H), 3.92 – 3.80 (m, 2H), 3.82 (s, 3H), 3.56 – 3.46 (m, 2H), 2.40 (s, 3H), 2.07 – 1.99 (m, 1H), 1.83 (dd, *J* = 13.4, 5.4 Hz, 1H), 1.67 – 1.62 (m, 1H), 1.50 – 1.39 (m, 1H), 0.76 (dd, *J* = 12.2, 12.2 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.2, 165.3, 162.1, 160.2, 141.1, 124.9, 121.9, 107.2, 102.6, 97.7, 65.9, 63.9, 56.8, 55.5, 44.4, 33.6, 31.3, 30.3, 29.0, 20.2. HRMS (ESI) m/e 355.1662 $[M+H]^+$ calculated for $C_{20}H_{23}N_2O_4^+$: 355.1658.



7

Preparation of reduction product 7. To a solution of compound **6** (6 g, 16.9 mmol, 1.0 equiv) in THF(200 mL) at 0°C was added DIBAL-H (85 mL, 1.0 M in toluene, 5.0 equiv) over 15 min . After the addition of DIBAL-H, the reaction was immediately quenched with 2.0 M NaOH aqueous solution (100 mL). The reaction mixture was extracted with EA three times and washed with brine. The organic phases were concentrated in vacuo and purified by flash column chromatography on silica gel (petroleum ether/ ethyl acetate, 1:1) to afford **7** (3.2 g, 55%, 72% based on recovered starting material) as a colorless oil. $[\alpha]_D^{24} = -56.3^{\circ}$ (c = 0.20, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.84 (s, 1H), 7.25 (d, *J* = 8.2 Hz, 1H), 6.55 (dd, *J* = 8.2, 2.3 Hz, 1H), 6.52 (d, *J* = 2.3 Hz, 1H), 3.92 (dt, *J* = 10.4, 3.9 Hz, 1H), 3.81 (s, 3H), 3.76 (td, *J* = 10.6, 2.2 Hz, 1H), 3.38 – 3.28 (m, 1H), 2.59 (d, *J* = 12.1 Hz, 1H), 2.52 – 2.43 (m, 2H), 2.42 – 2.37 (m, 1H), 2.08 – 2.01 (m, 3H), 1.81 (s, 3H), 1.81 – 1.73 (m, 1H), 1.50 – 1.36 (m, 1H), 1.27 (ddd, *J* = 12.0, 4.4, 2.4 Hz, 1H), 0.75 (q, *J* = 11.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 183.2, 159.6, 145.1, 141.4, 125.8, 125.6, 107.0, 106.1, 96.9, 71.8, 64.7, 56.2, 55.5, 54.5, 54.1, 35.5, 33.7, 31.5, 30.6, 16.4. HRMS (ESI) m/e 341.1858 $[M+H]^+$ calculated for $C_{20}H_{25}N_2O_3^+$: 341.1860.

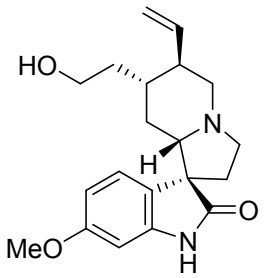


8

Preparation of hydrazone 8. Compound **7** (2.0 g, 5.9 mmol, 1.0 equiv) was dissolved in 2.0 M HCl (aq.) /THF (1:3, c= 0.2 M) at 0°C and stirred at room temperature overnight. The reaction mixture was quenched with Na_2CO_3 (aq.) (2.0 M) and the water phase was extracted with DCM. The combined organic phases were dried (Na_2SO_4), filtered and concentrated under reduced pressure to give the crude hydrolyzed product (2.1 g, 99%) without further purification.

The crude hydrolyzed product was dissolved in DCM (30 mL) and cooled down to 0 °C. DMAP (72.1 mg, 0.59 mmol, 10 mol%), pyridine (1.4 mL, 17.7 mmol, 3.0 equiv.) and acetic anhydride (1.7 mL, 17.7 mmol, 3.0 equiv, dropwise addition) were added in this order and the reaction was left at room temperature for 4 h. MeOH (4.8 mL, 20 equiv) was added to quench the reaction. The resulting solution was stirred at rt for 15 min and concentrated under reduced pressure to afford a yellow oil, which was used directly for next operation without further purification.

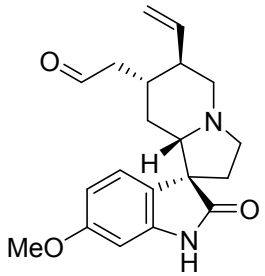
To a stirred solution of the aforementioned oil in MeOH/AcOH (3:1, 40 mL) was added tosylhydrazide (2.2 g, 11.8 mmol, 2.0 equiv). The mixture was stirred at room temperature overnight. $NaHCO_3$ (sat. aq.) was added to quench this reaction and the water phase was extracted with DCM. The combined organic phases were dried (Na_2SO_4), filtered and concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ ethyl acetate, 1:1) to afford **8** (3.0 g, 89% over two steps) as a light-yellow solid. $[\alpha]_D^{24}$ = 17.4º (c = 0.20, $CHCl_3$); m.p.: 116–117 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (s, 1H), 7.83 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 8.2 Hz, 1H), 6.56 (dd, *J* = 8.3, 2.3 Hz, 1H), 6.50 (d, *J* = 2.2 Hz, 1H), 3.81 (s, 3H), 3.80 – 3.70 (m, 1H), 3.67 – 3.61 (m, 1H), 3.24 (t, *J* = 7.7 Hz, 1H), 3.09 (dd, *J* = 10.6, 3.6 Hz, 1H), 2.50 – 2.32 (m, 3H), 2.43 (s, 3H), 2.25 (td, *J* = 11.1, 3.6 Hz, 1H), 2.08 – 1.94 (m, 2H), 1.92 (s, 3H), 1.87 (s, 1H), 1.76 (s, 3H), 1.64 – 1.52 (m, 1H), 1.37 – 1.25 (m, 2H), 1.07 – 1.00 (m, 1H), 0.66 (q, *J* = 11.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 182.0, 171.0, 159.7, 157.5, 144.0, 141.1, 135.4, 129.5, 128.0, 125.6, 125.4, 107.1, 96.9, 71.2, 61.8, 56.3, 56.2, 55.5, 53.5, 50.8, 35.2, 34.3, 31.8, 30.8, 21.6, 20.9, 14.2; HRMS (ESI) m/e 569.2420 $[M+H]^+$ calcd for $C_{29}H_{37}N_4O_6S^+$: 569.2428.



9

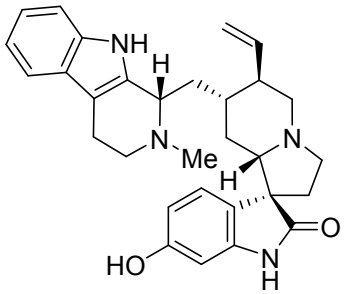
Preparation of alcohol 9. The hydrazone **8** (500 mg, 0.88 mmol, 1.0 equiv) was dissolved in anhydrous THF (c = 0.1 M) under argon atmosphere and cooled down to -78 °C. *n*-BuLi (5.5 mL, 1.6 M in THF, 10 equiv.) was added dropwise to this solution. During the addition, the reaction mixture turned from yellow to brown and resulting solution was allowed to slowly reach room temperature and stirred overnight. Brine (5 mL) was added to quench the reaction and the water phase was extracted with DCM. The combined organic phases were dried (Na_2SO_4), filtered and concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel

(petroleum ether/ethyl acetate, 1:2) to afford **9** (200 mg, 66%) as a colorless oil. $[\alpha]_D^{24} = -20.5^\circ$ (c = 0.20, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.30 – 7.26 (m, 1H), 6.58 (dd, *J* = 8.2, 2.3 Hz, 1H), 6.50 (d, *J* = 2.3 Hz, 1H), 5.81 – 5.54 (m, 1H), 5.12 – 5.07 (m, 2H), 3.83 (s, 3H), 3.57 – 3.53 (m, 1H), 3.50 – 3.44 (m, 1H), 3.29 (td, *J* = 8.5, 1.9 Hz, 1H), 3.13 (d, *J* = 7.1 Hz, 1H), 2.51 – 2.37 (m, 3H), 2.07 – 1.96 (m, 3H), 1.84 – 1.79 (m, 1H), 1.36 – 1.27 (m, 3H), 1.16 – 1.11 (m, 1H), 0.68 (dd, *J* = 11.7, 11.7 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 182.9, 159.6, 141.4, 139.9, 125.7, 125.6, 116.7, 107.1, 97.0, 71.7, 60.3, 58.9, 56.5, 55.5, 53.8, 47.1, 36.3, 35.2, 31.2; HRMS (ESI) m/e 343.2198 $[M+H]^+$ calcd for $C_{20}H_{27}N_2O_3^+$: 343.2202.



10

Preparation of aldehyde 10. To a solution of alcohol **9** (150 mg, 0.44 mmol, 1.0 equiv) in DCM (5 mL) was added pyridine (0.1 mL, 1.3 mmol, 3.0 equiv) followed by Dess-Martin periodinane (373 mg, 0.88 mmol, 2.0 equiv) at room temperature and the reaction was stirred at room temperature for 2 h. NaOH (5% aq., 10 drops) was added to quench the reaction. The mixture was stirred for 0.5 h at room temperature before it was diluted with water. The aqueous phase was extracted with DCM and the combined organic phases were dried (Na_2SO_4), filtered and concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ ethyl acetate, 1:1) to afford **10** (128 mg, 85%) as a light-yellow solid. $[\alpha]_D^{24} = 16.5^\circ$ (c = 0.20, $CHCl_3$); m.p.: 122–123 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 9.62 (d, *J* = 1.3 Hz, 1H), 8.24 (s, 1H), 7.26 (d, *J* = 8.2 Hz, 1H), 6.56 (dd, *J* = 8.2, 2.3 Hz, 1H), 6.50 (d, *J* = 2.2 Hz, 1H), 5.56 – 5.50 (m, 1H), 5.14 – 5.10 (m, 2H), 3.82 (s, 3H), 3.30 (t, *J* = 8.5 Hz, 1H), 3.16 (d, *J* = 7.2 Hz, 1H), 2.58 – 2.48 (m, 3H), 2.42 – 2.37 (m, 1H), 2.07 – 1.98 (m, 3H), 1.85 – 1.78 (m, 1H), 1.36 – 1.27 (m, 2H), 0.76 (dd, *J* = 11.8, 11.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.3, 182.7, 159.7, 141.5, 139.1, 125.5, 177.8, 107.3, 97.0, 77.3, 71.3, 58.4, 56.4, 55.5, 53.7, 48.2, 46.8, 35.3, 34.7, 32.0; HRMS (ESI) m/e 341.1866 $[M+H]^+$ calcd for $C_{20}H_{25}N_2O_3^+$: 341.1865.



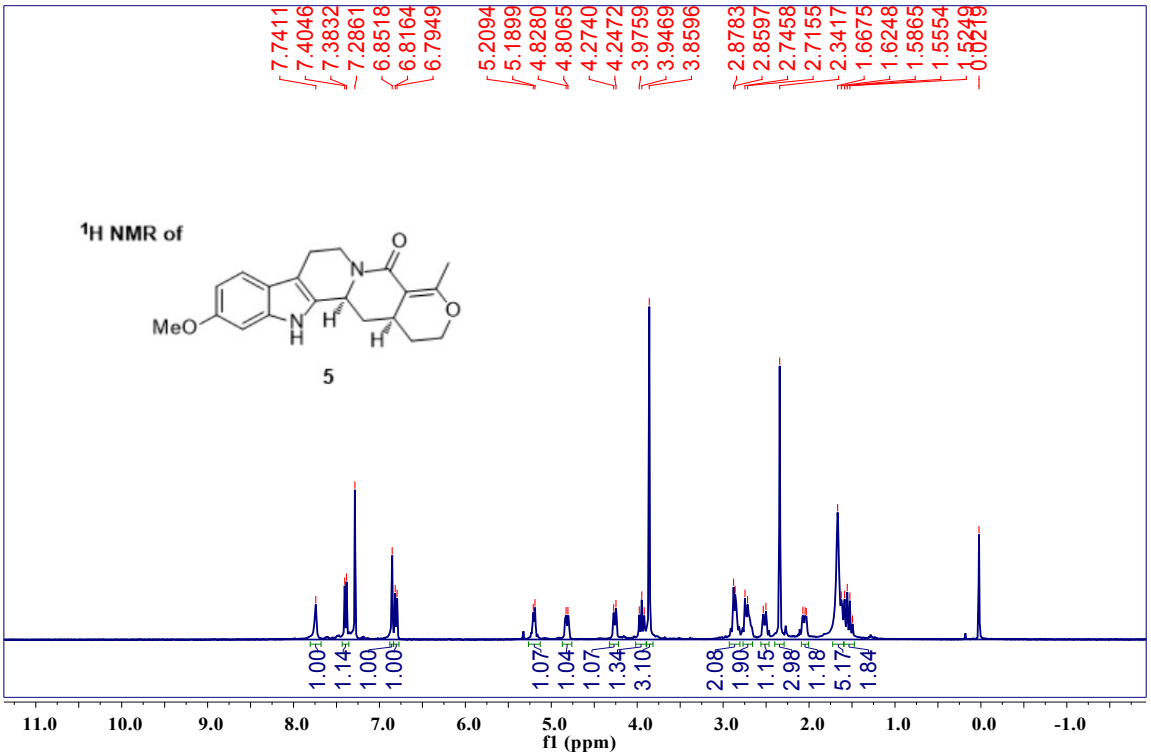
Strychnofoline (1)

Preparation of (–)-Strychnofoline (1). AcOH (0.5 mL) was added to a solution of compound **10** (100 mg, 0.29 mmol, 1.0 equiv) and *N*-methyl-tryptamine (51 mg, 0.29 mmol, 1.0 equiv) in toluene (5 mL). It was heated at 80 °C for 2 h and then diluted with ether and water. The organic layer was separated and washed with saturated aq. Na_2CO_3 and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give two inseparable epimers (130 mg, 90%, mixture, d.r. = ca. 1: 1.5) as a yellow oil. It was used directly to next step without further purification.

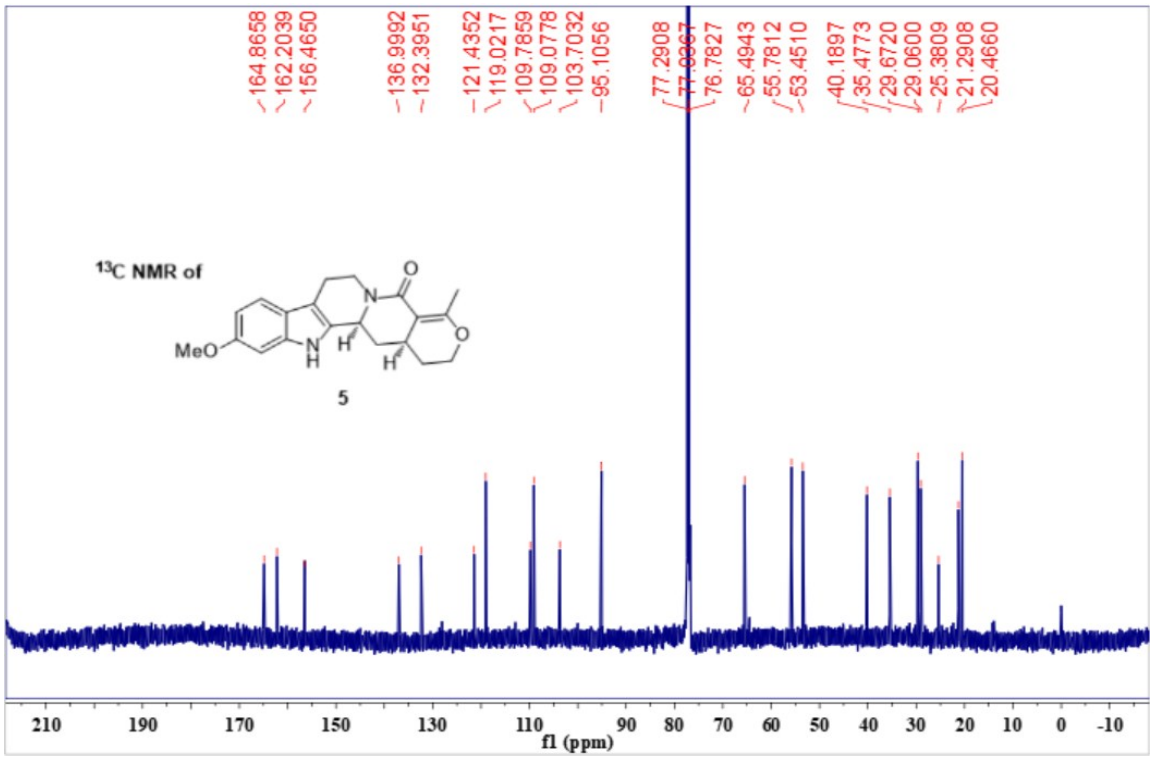
To a solution of the crude material obtained above (50 mg, 0.1 mmol, 1.0 equiv) in DCM (0.1 M) at -20 ^oC, BBr_3 (1.0 mL, 10.0 equiv, 1.0 M in DCM) was added dropwise. After stirring at -20 ^oC for 0.5 h, the mixture warmed to room temperature and stirred for 1 h. The reaction was quenched with sat $NaHCO_3$, extracted with DCM. The collected organic layer washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (petroleum ether/ ethyl acetate, 1:1) to give **1** (31 mg, 65%) as a pale-yellow gel. $[\alpha]_D^{24}$ = –46.2º (c = 0.20, $CHCl_3$); ^1H NMR (400 MHz, d_6-acetone) δ 9.71 (s, 1H), 9.29 (s, 1H), 8.02 (s, 1H), 7.36 (d, *J* = 8.2 Hz, 1H), 7.23 (d, *J* = 8.2 Hz, 1H), 7.20 (t, *J* = 8.5 Hz, 1H), 7.00 – 6.95 (m, 2H), 6.53 – 6.50 (m, 2H), 5.67 – 5.53 (m, 1H), 5.12 – 5.00(m, 2H), 3.57 – 3.54 (m, 1H), 3.29 – 3.26 (m, 1H), 3.06 – 3.00 (m, 2H), 2.89 – 2.78 (m, 2H), 2.54 – 2.49 (m, 1H), 2.32 (s, 3H), 2.40 – 2.17 (m, 3H), 2.00 – 1.87 (m, 4H), 1.72 – 1.64 (m, 1H), 1.61 – 1.55 (m, 1H), 1.25 – 1.20 (m, 1H), 0.82 – 0.76 (m, 1H); ^{13}C NMR (101 MHz, d_6-acetone) δ 181.5, 158.2, 143.5, 141.5, 137.1, 136.7, 128.3, 126.4, 125.4, 121.4, 119.2, 118.3, 116.7, 111.5, 108.9, 106.6, 98.4, 72.2, 59.7, 56.9, 56.3, 54.2, 48.2, 45.7, 41.2, 38.9, 36.6, 36.0, 31.4, 16.8. HRMS (ESI) m/e 483.2757 $[M+H]^+$ calcd for $C_{30}H_{35}N_4O_2^+$: 483.2760.

II NMR Spectra of Compounds

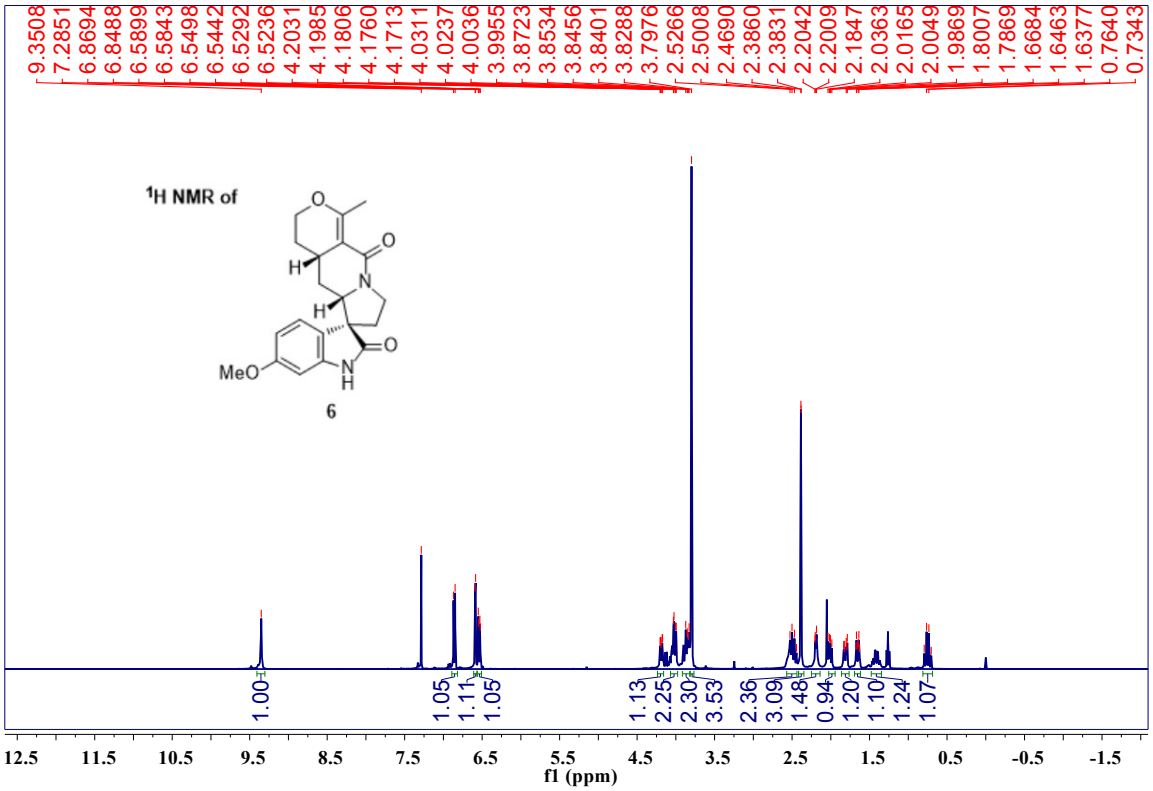
Supplementary Figure 1. ^{1}H spectrum of 5 (400 MHz, $CDCl_3$)



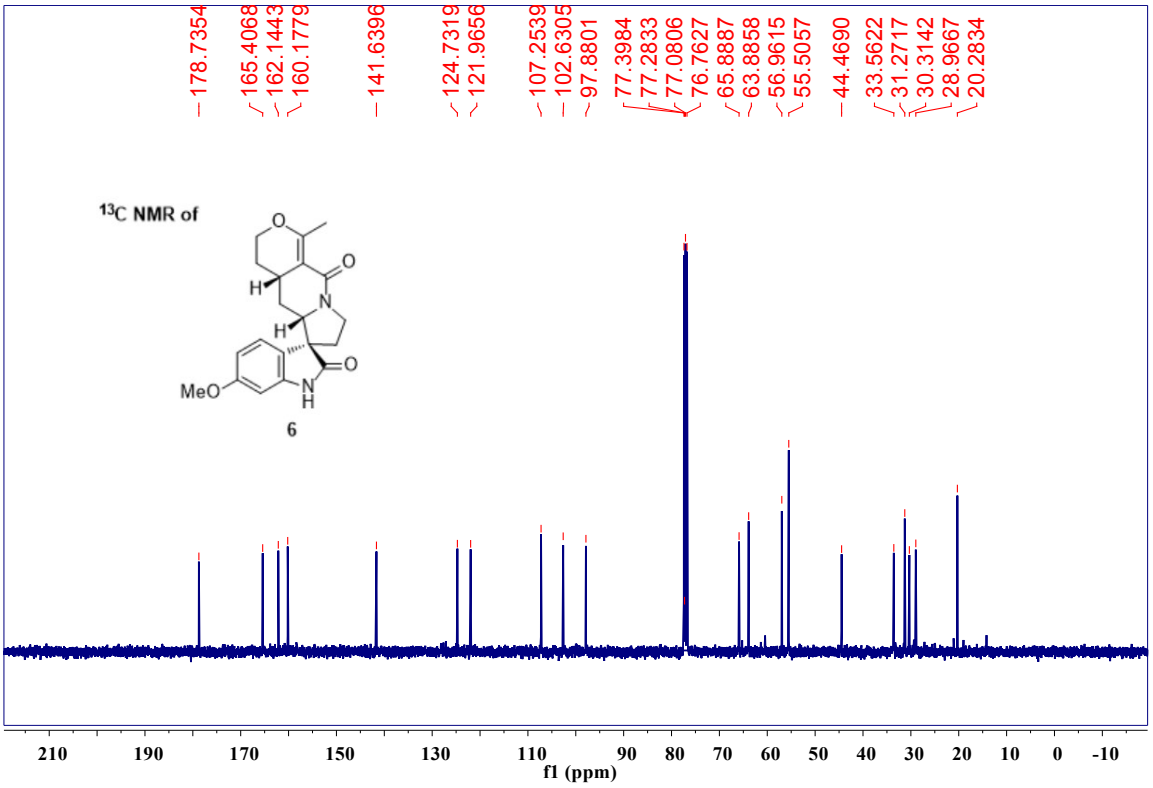
Supplementary Figure 2. ^{13}C NMR spectrum of 5 (126 MHz, $CDCl_3$)



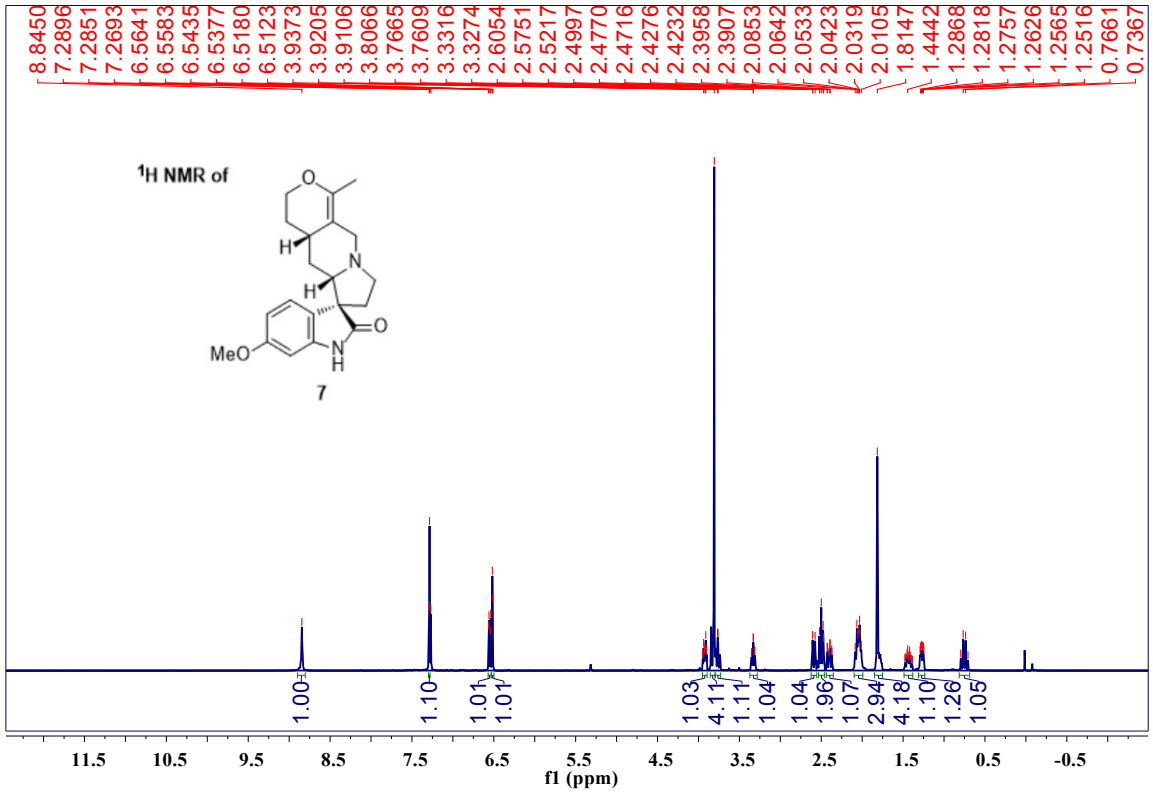
Supplementary Figure 3. ^{1}H spectrum of 6 (400 MHz, $CDCl_3$)



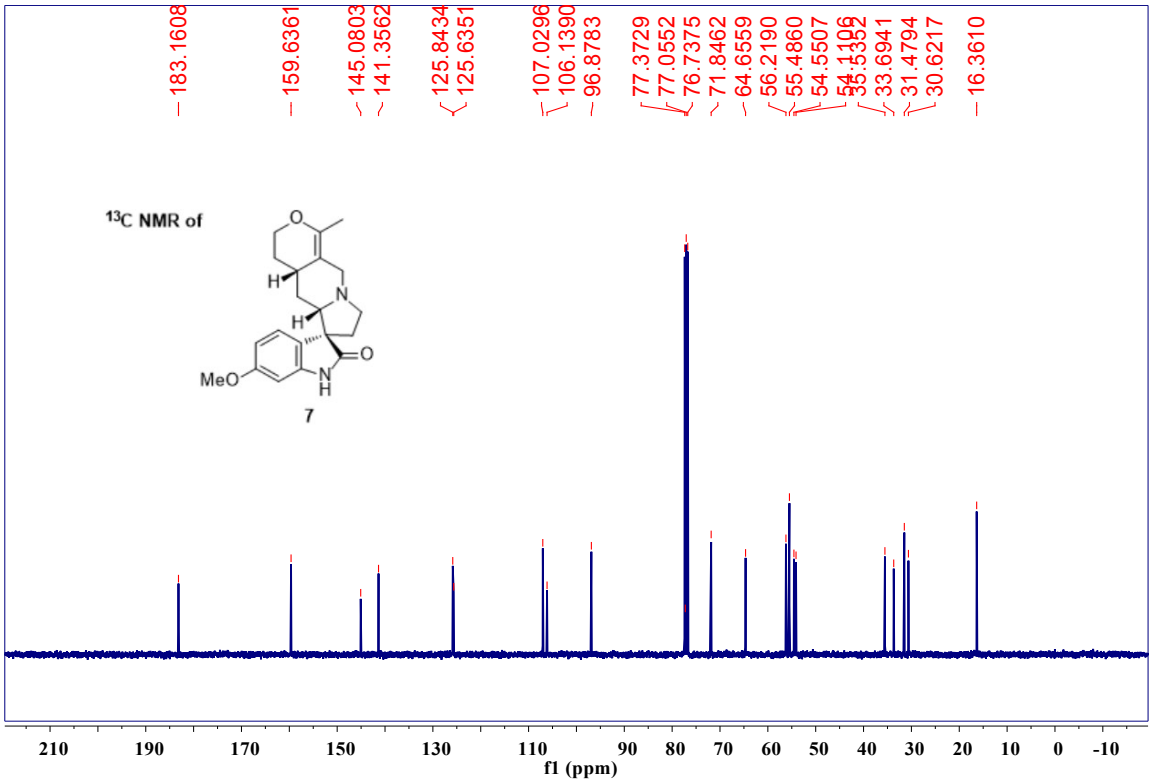
Supplementary Figure 4. ^{13}C NMR spectrum of 6 (101 MHz, $CDCl_3$)



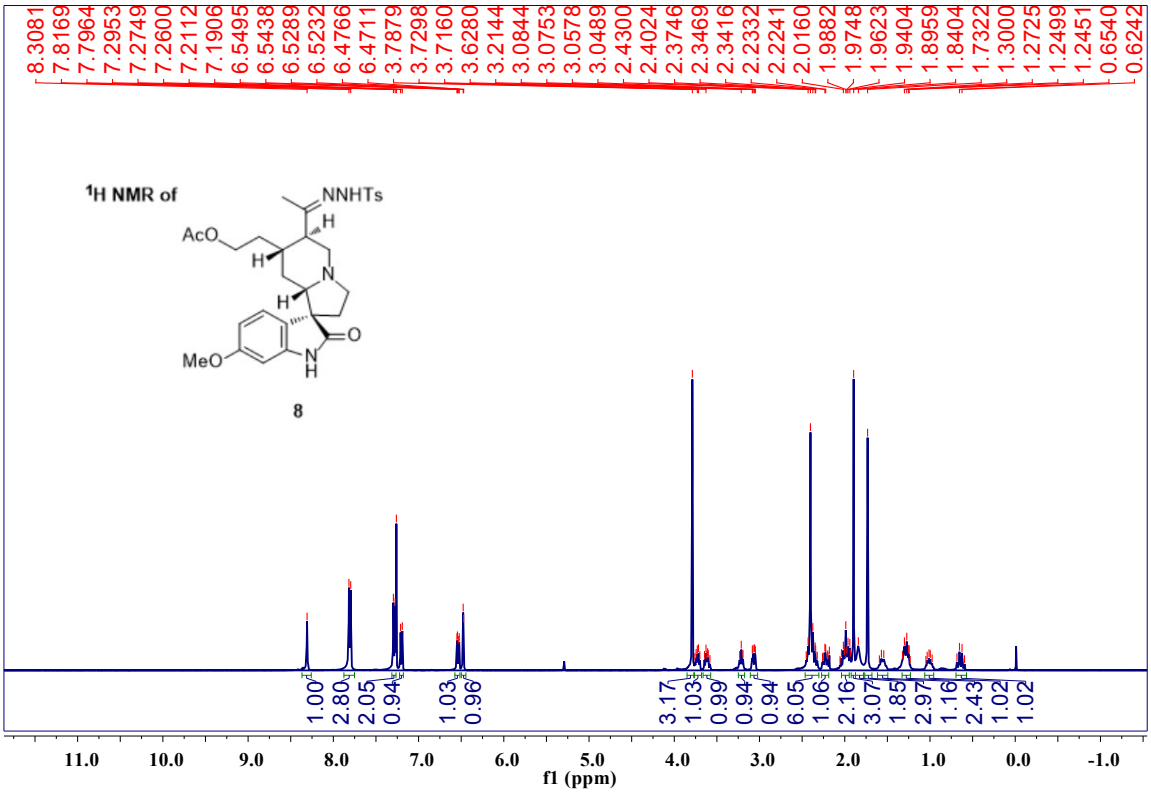
Supplementary Figure 5. ^{1}H sp spectrum ectra of 7 (400 MHz, $CDCl_3$)



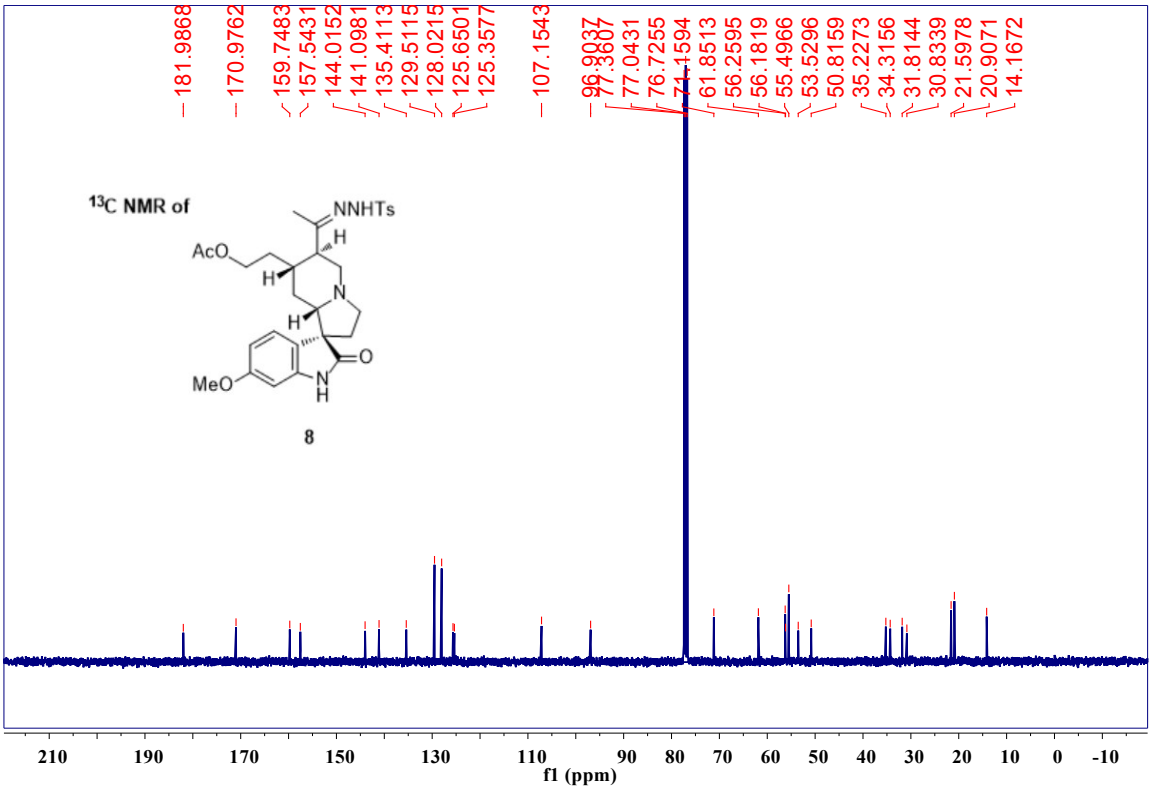
Supplementary Figure 6. ^{13}C NMR spectrum of 7 (101 MHz, $CDCl_3$)



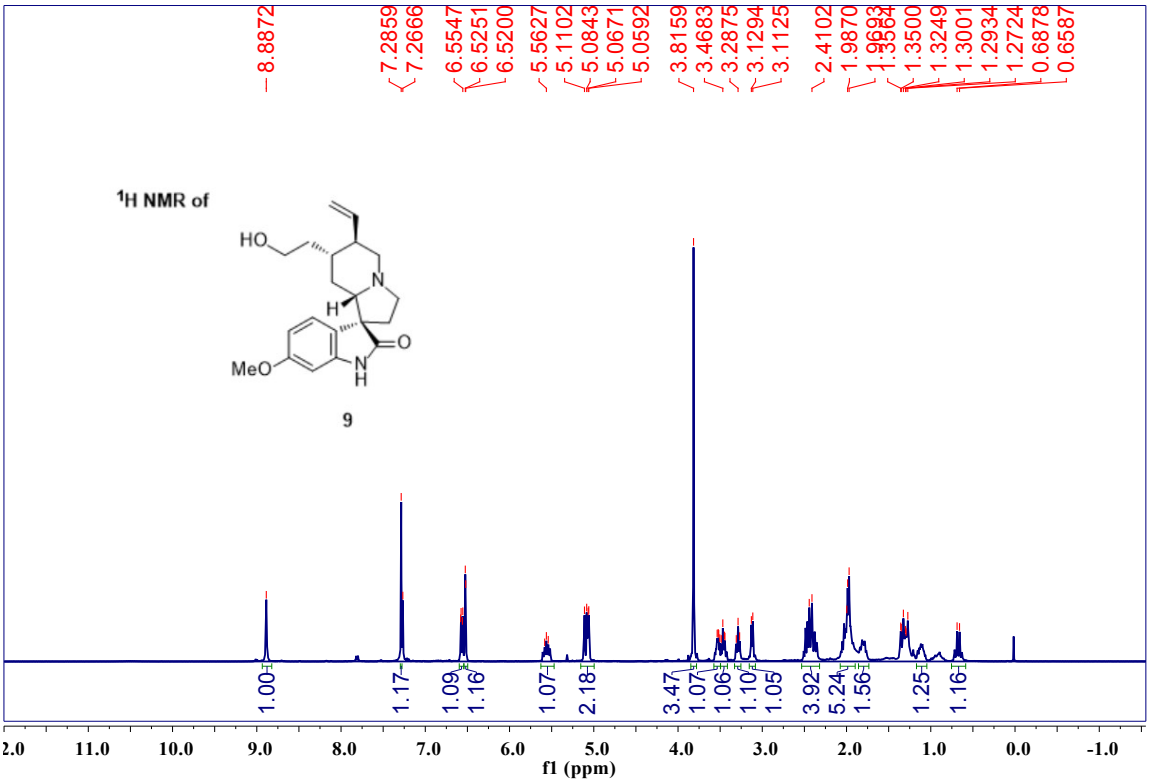
Supplementary Figure 7. ^{1}H spectrum of 8 (400 MHz, $CDCl_3$)



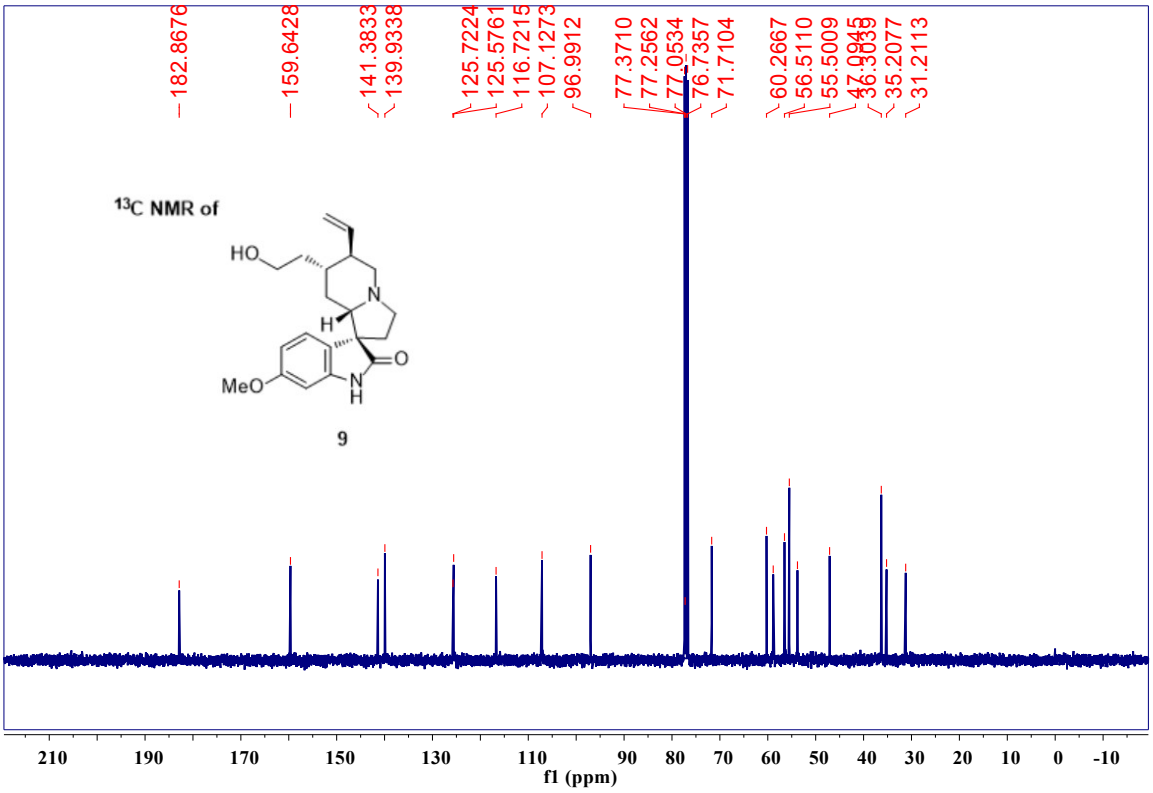
Supplementary Figure 8. ^{13}C NMR spectrum of 8 (101 MHz, $CDCl_3$)



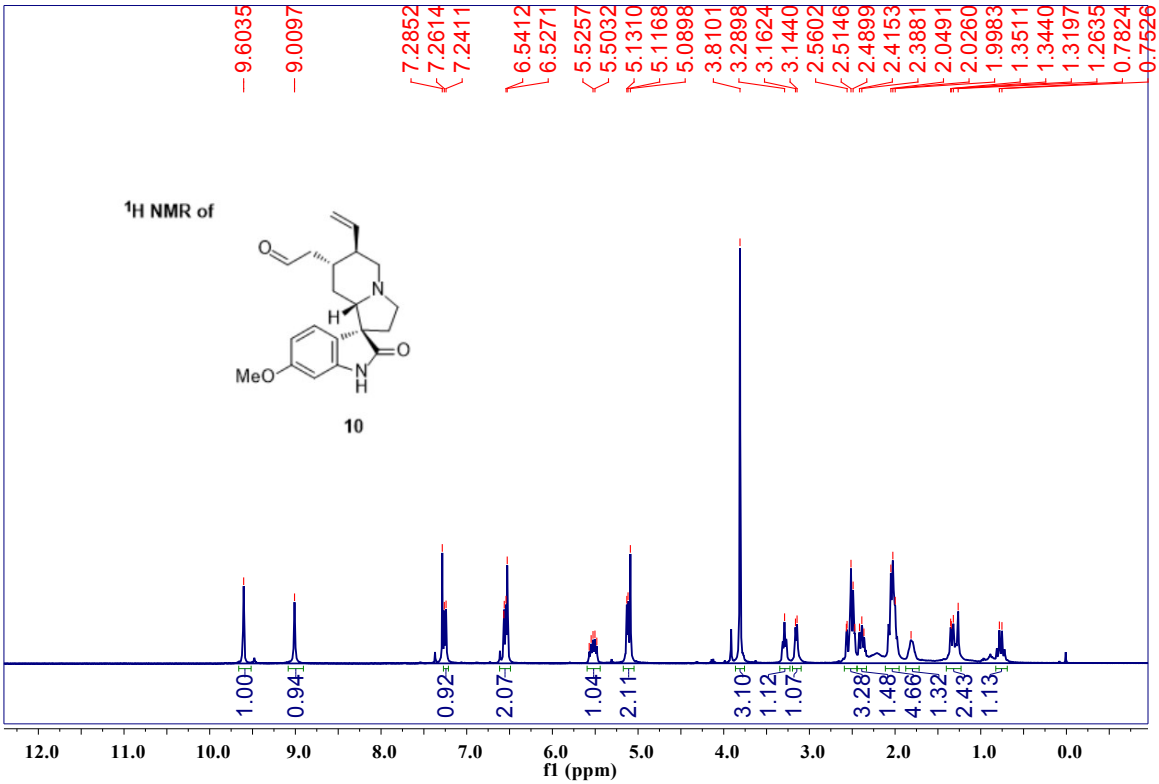
Supplementary Figure 9. ^{1}H spectrum of 9 (400 MHz, $CDCl_3$)



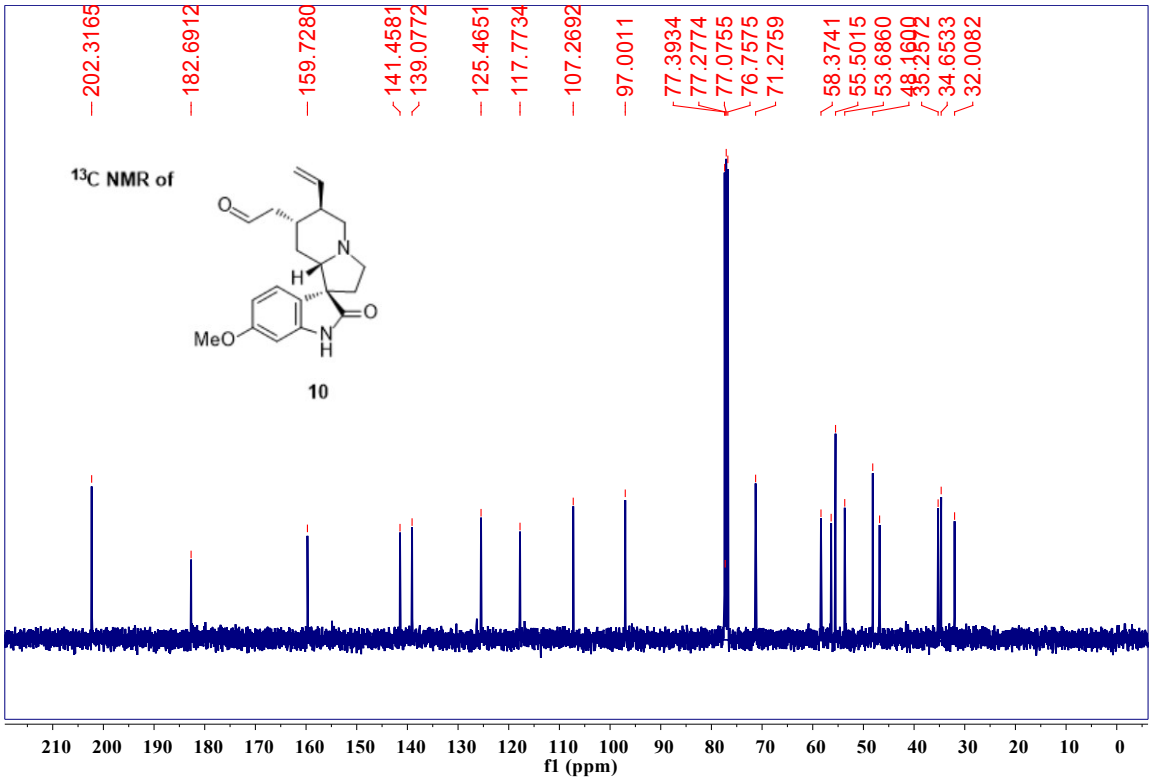
Supplementary Figure 10. ^{13}C NMR spectrum of 9 (101 MHz, $CDCl_3$)



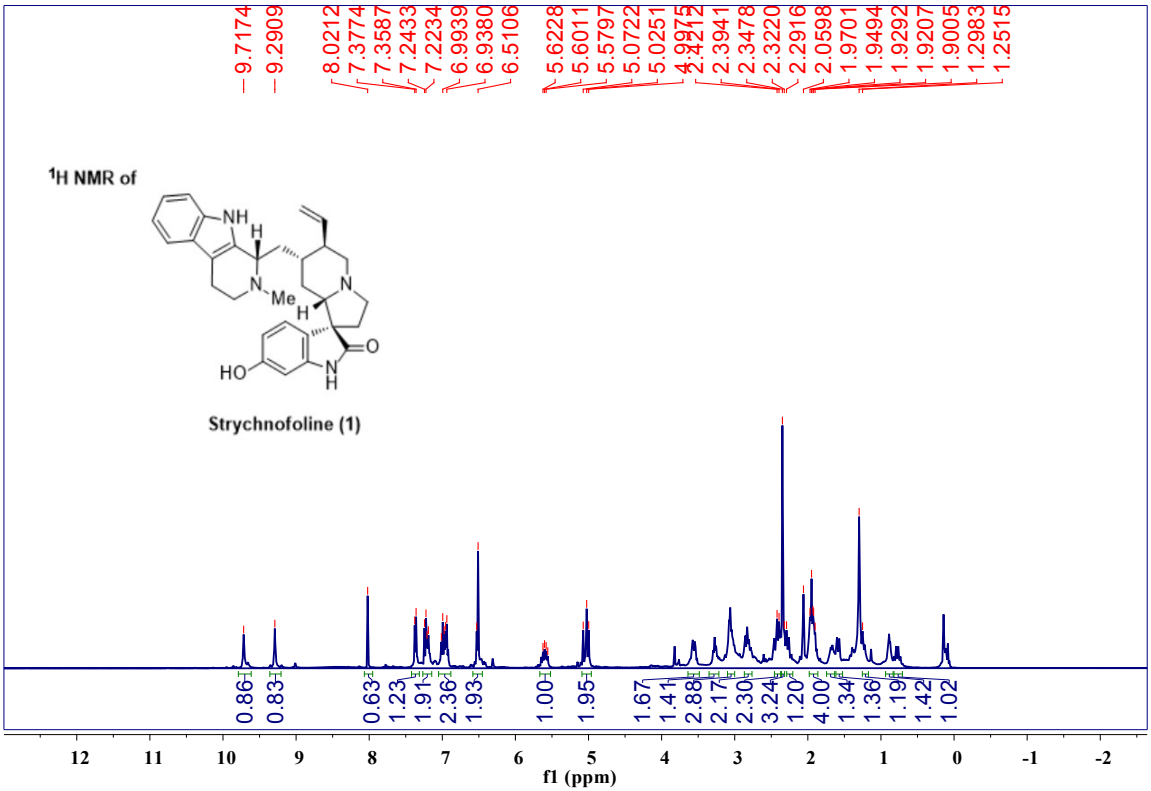
Supplementary Figure 11. ^{1}H spectrum of 10 (400 MHz, $CDCl_3$)



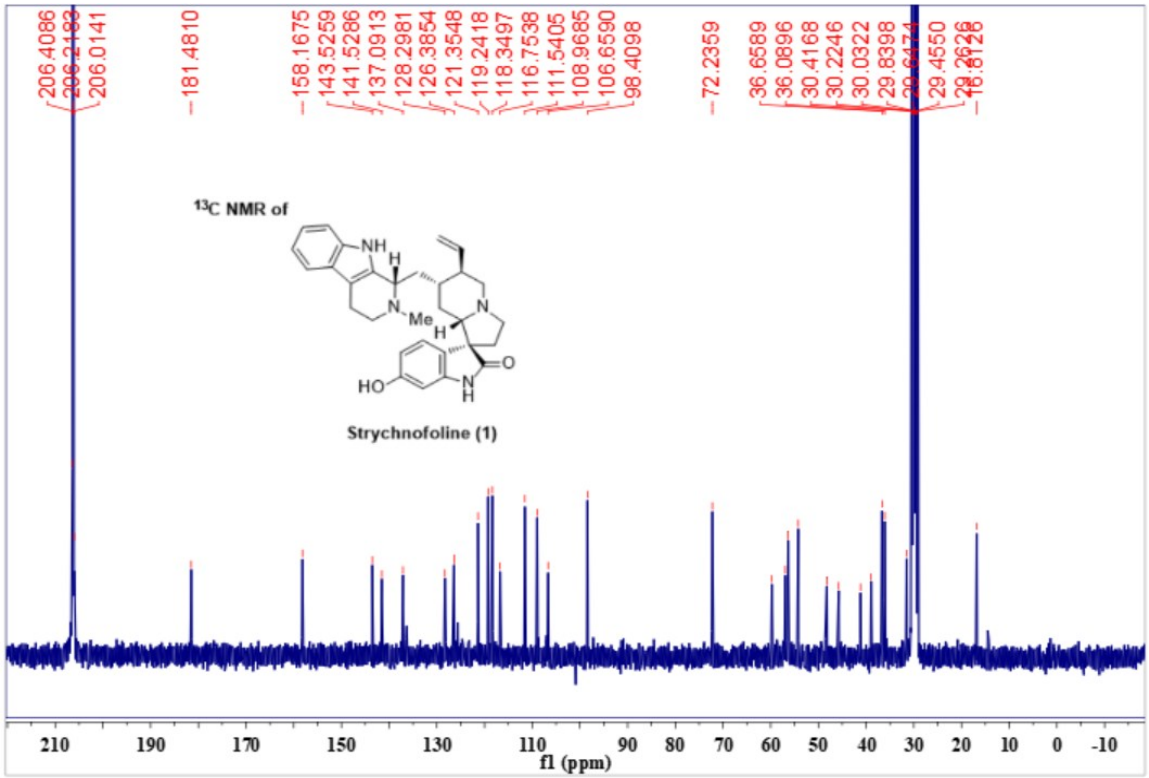
Supplementary Figure 12. ^{13}C NMR spectrum of 10 (101 MHz, $CDCl_3$)



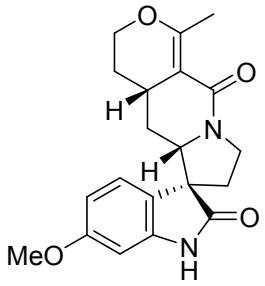
Supplementary Figure 13. ^{1}H spectrum of 1 (400 MHz, d_6-acetone)



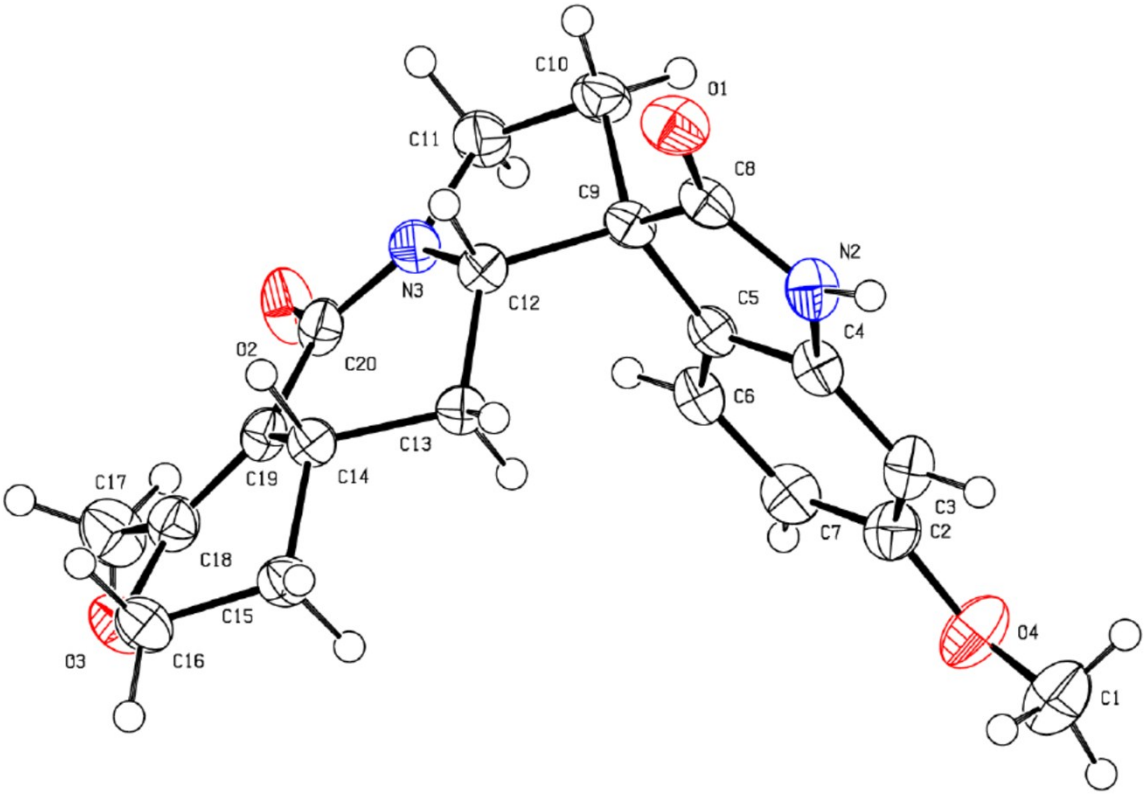
Supplementary Figure 14. ^{13}C NMR spectrum of 1 (101 MHz, d_6-acetone)



III Crystallographic Data of Compounds 6



6



Crystal data and structure refinement

Empirical formula	$C_{20}H_{22}N_2O_4$
Formula weight	354.39
Temperature/K	170.0
Crystal system	orthorhombic
a/Å	6.4100(4)
b/Å	12.0057(9)
c/Å	22.2391(15)
α/°	90

β/°	90
γ/°	90
Volume/Å^3	1711.4(2)
Z	4
ρ_{calc}g/cm^3	1.375
μ/mm^{-1}	0.789
F(000)	752.0
Crystal size/mm^3	0.3 × 0.3 × 0.3
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	14.04 to 136.4
Index ranges	-7 ≤ h ≤ 7, -12 ≤ k ≤ 14, -26 ≤ l ≤ 23
Reflections collected	10789
Independent reflections	3106 [R_{int} = 0.0246, R_{sigma} = 0.0218]
Data/restraints/parameters	3106/0/237
Goodness-of-fit on F^2	1.089
Final R indexes [I>=2σ (I)]	R_1 = 0.0311, wR_2 = 0.0817
Final R indexes [all data]	R_1 = 0.0313, wR_2 = 0.0819
Largest diff. peak/hole / e Å^{-3}	0.18/-0.16
Flack parameter	0.03(5)

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

Atom	***x***	***y***	***z***	**U(eq)**
O1	9583(2)	7666.4(13)	7517.0(7)	38.3(4)
O2	3687(3)	4278.2(11)	5994.3(6)	38.6(4)
O3	-154(2)	6962.9(12)	5396.4(7)	36.8(3)
O4	825(3)	5794.6(14)	9211.2(7)	46.9(4)
N2	7050(3)	7400.7(13)	8236.5(7)	30.3(4)
N3	5691(3)	5497.2(13)	6492.9(7)	27.6(4)
C1	509(5)	6703(2)	9600.7(11)	51.3(6)
C2	2351(3)	5893.1(17)	8780.2(9)	34.0(4)
C3	3923(4)	6700.2(17)	8795.7(8)	32.8(4)
C4	5340(3)	6684.1(15)	8326.7(8)	26.7(4)
C5	5224(3)	5931.0(15)	7852.1(8)	24.2(4)
C6	3657(3)	5144.6(15)	7852.5(8)	27.9(4)
C7	2242(3)	5118.8(17)	8322.6(9)	33.2(4)
C8	8077(3)	7160.6(16)	7716.6(9)	27.8(4)
C9	6946(3)	6189.0(14)	7412.1(8)	25.0(4)
C10	8311(3)	5191.0(17)	7225.2(9)	32.6(4)

C11	7023(3)	4608.9(17)	6740.1(9)	34.3(5)
C12	6152(3)	6569.6(15)	6784.9(8)	24.6(4)
C13	4298(3)	7336.6(14)	6763.2(8)	23.9(4)
C14	3476(3)	7387.4(15)	6122.2(8)	24.0(4)
C15	1675(3)	8207.3(16)	6076.7(8)	28.6(4)
C16	593(3)	8089.2(17)	5481.6(9)	32.5(4)
C17	100(4)	5045.7(19)	5349.0(11)	43.1(5)
C18	1049(3)	6098.3(17)	5589.6(8)	30.2(4)
C19	2774(3)	6230.7(15)	5928.6(8)	24.8(4)
C20	4038(3)	5269.4(16)	6134.1(8)	27.4(4)

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

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
O1	28.4(7)	37.4(8)	49.1(9)	5.3(6)	0.1(6)	-10.0(6)
O2	63.7(10)	20.6(7)	31.5(7)	-3.2(5)	-13.9(7)	3.0(7)
O3	35.3(7)	32.7(8)	42.3(8)	1.0(6)	-11.2(6)	3.5(6)
O4	56.1(10)	41.6(9)	42.9(8)	-2.4(7)	20.1(8)	-8.9(8)
N2	34.9(8)	25.1(8)	30.8(8)	-3.4(6)	-4.3(7)	-9.1(7)
N3	34.3(8)	21.7(8)	26.8(8)	-1.9(6)	-1.6(7)	7.0(7)
C1	65.0(16)	44.8(13)	44.0(12)	-2.8(10)	14.7(12)	3.0(12)
C2	36.4(10)	33.3(10)	32.5(10)	10.9(8)	5.5(8)	-1.4(9)
C3	44.4(11)	28.6(9)	25.5(9)	-0.1(7)	1.1(9)	-1.4(8)
C4	30.6(9)	21.7(8)	28.0(9)	3.5(7)	-4.2(8)	-3.9(7)
C5	26.9(9)	20.0(8)	25.9(9)	3.9(6)	-3.8(7)	0.1(7)
C6	32.1(10)	23.3(9)	28.4(9)	2.6(7)	-5.8(8)	-3.1(8)
C7	32.9(10)	28.9(10)	37.7(11)	6.3(8)	-1.6(9)	-7.8(8)
C8	25.2(9)	24.6(9)	33.6(10)	4.7(7)	-5.7(7)	-1.3(7)
C9	23.5(8)	22.6(9)	28.8(9)	1.2(7)	-2.8(7)	1.0(7)
C10	30.2(10)	29.4(10)	38.2(10)	0.1(8)	-3.8(8)	9.2(8)
C11	41.3(11)	26.8(9)	34.7(10)	-2.5(8)	-4.8(9)	12.9(9)
C12	25.9(9)	22.2(8)	25.6(8)	0.3(7)	0.5(7)	-0.2(7)
C13	27.2(9)	19.0(8)	25.7(8)	-0.7(7)	0.0(7)	1.0(7)
C14	26.6(9)	20.1(8)	25.2(8)	2.6(7)	0.9(7)	1.1(7)
C15	30.3(10)	23.3(9)	32.3(10)	2.4(7)	1.4(8)	5.0(8)
C16	33(1)	28.6(9)	35.8(10)	4.6(8)	-4.5(8)	4.2(8)
C17	44.5(13)	36.5(12)	48.4(12)	-7.3(10)	-11.4(11)	-4.7(9)
C18	34.3(10)	28.4(10)	27.9(9)	0.0(8)	0.8(8)	1.5(8)
C19	31.7(9)	21.9(9)	20.8(8)	0.6(6)	2.5(7)	0.4(7)

C20	38.6(10)	23.7(9)	20.0(8)	-0.5(7)	1.1(8)	3.9(8)

Bond Lengths

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C8	1.224(2)	C5	C6	1.379(3)
O2	C20	1.250(2)	C5	C9	1.507(3)
O3	C16	1.447(3)	C6	C7	1.384(3)
O3	C18	1.363(2)	C8	C9	1.531(3)
O4	C1	1.407(3)	C9	C10	1.541(3)
O4	C2	1.375(2)	C9	C12	1.553(2)
N2	C4	1.408(2)	C10	C11	1.528(3)
N2	C8	1.361(3)	C12	C13	1.504(2)
N3	C11	1.473(2)	C13	C14	1.521(2)
N3	C12	1.472(2)	C14	C15	1.520(2)
N3	C20	1.354(3)	C14	C19	1.522(2)
C2	C3	1.399(3)	C15	C16	1.501(3)
C2	C7	1.380(3)	C17	C18	1.501(3)
C3	C4	1.383(3)	C18	C19	1.347(3)
C4	C5	1.392(3)	C19	C20	1.482(3)

Bond Angles

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C18	O3	C16	118.89(15)	C8	C9	C10	116.28(16)
C2	O4	C1	117.72(18)	C8	C9	C12	109.15(14)
C8	N2	C4	111.60(15)	C10	C9	C12	99.94(15)
C12	N3	C11	110.63(15)	C11	C10	C9	103.85(15)
C20	N3	C11	121.83(16)	N3	C11	C10	104.22(15)
C20	N3	C12	126.41(15)	N3	C12	C9	101.80(14)
O4	C2	C3	123.72(19)	N3	C12	C13	111.28(15)
O4	C2	C7	114.86(18)	C13	C12	C9	117.89(15)
C7	C2	C3	121.42(18)	C12	C13	C14	109.17(14)
C4	C3	C2	116.42(18)	C13	C14	C19	109.33(14)
C3	C4	N2	127.61(18)	C15	C14	C13	110.57(15)
C3	C4	C5	123.08(18)	C15	C14	C19	110.32(15)
C5	C4	N2	109.30(16)	C16	C15	C14	110.38(16)
C4	C5	C9	108.64(16)	O3	C16	C15	110.90(15)
C6	C5	C4	118.91(17)	O3	C18	C17	107.43(17)

C6	C5	C9	132.44(16)	C19	C18	O3	123.43(18)
C5	C6	C7	119.55(17)	C19	C18	C17	129.13(19)
C2	C7	C6	120.58(18)	C18	C19	C14	120.57(17)
O1	C8	N2	125.77(19)	C18	C19	C20	121.95(17)
O1	C8	C9	126.22(18)	C20	C19	C14	117.48(16)
N2	C8	C9	107.94(15)	O2	C20	N3	118.65(17)
C5	C9	C8	102.48(15)	O2	C20	C19	124.46(18)
C5	C9	C10	115.54(16)	N3	C20	C19	116.88(16)
C5	C9	C12	113.79(15)				

Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³)

Atom	***x***	***y***	***z***	**U(eq)**
H2	7410	7937	8485	36
H1A	297	7382	9364	77
H1B	1734	6795	9860	77
H1C	-725	6565	9850	77
H3	4013	7231	9111	39
H6	3548	4624	7532	34
H7	1187	4563	8330	40
H10A	9668	5445	7063	39
H10B	8565	4689	7571	39
H11A	7936	4292	6425	41
H11B	6167	4004	6915	41
H12	7334	6931	6566	29
H13A	4711	8091	6898	29
H13B	3194	7059	7036	29
H14	4625	7640	5850	29
H15A	669	8067	6406	34
H15B	2209	8977	6121	34
H16A	-597	8613	5463	39
H16B	1574	8283	5154	39
H17A	-1412	5061	5412	65
H17B	698	4404	5560	65
H17C	396	4985	4918	65