Electronic Supplementary Material (ESI) for RSC Advances.

Highly Efficient Construction of an Oxa-[3.2.1]octane-embedded 5-7-6 Tricyclic Carbon Skeleton and Ring-opening of the Bridged Ring via C-O Bond Cleavage

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Contents

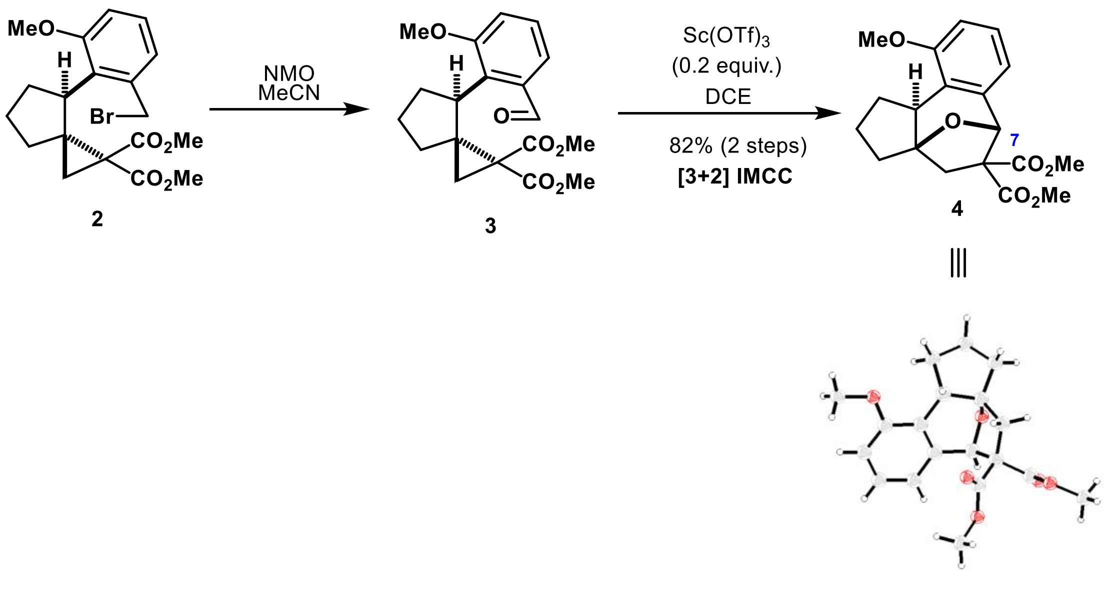
1. General Information

All reactions were carried out under an argon atmosphere with dry solvents under anhydrous conditions, unless otherwise noted. All solvents were obtained from commercial sources and were purified according to standard procedures, and stored under an argon atmosphere. All reagents were purchased from the commercial sources and used without further purification, unless otherwise stated. Reactions were monitored by thin layer chromatography (TLC) carried out on S-2 0.25 mm E. Merck silica gel plates (GF-254) and visualized by UV-light (254 and 365 nm)/$KMnO_4$/I_2/*p*-anisaldehyde. NMR spectra were recorded in $CDCl_3$, at 400 MHz (Bruker). Chemical shifts were reported in parts per million (δ) using the residual solvent signals ($CDCl_3$: δH 7.26, δC 77.10) as the internal standards for the ^{1}H and ^{13}C NMR spectra and coupling constants (*J*) in Hz. The following abbreviations are used to designate multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, quint = quintet, br = broad. Melting points were obtained on a Yanaco-241 apparatus and are uncorrected. HRMS were recorded on Varian 7.0T FTMS (Fouier transform ion cyclotron resonance mass spertrometer, ESI) and Agilent 6520 Q-TOF LC/MS (ESI). Single crystal XRD was recorded on Rigaku 007 Saturn 70 (*Mo*).

2. Experimental Procedures and Spectral Data

Preparation of the compound 4.

Benzyl bromide **2** was prepared from the known compound **1** according to the previous literature.[1]



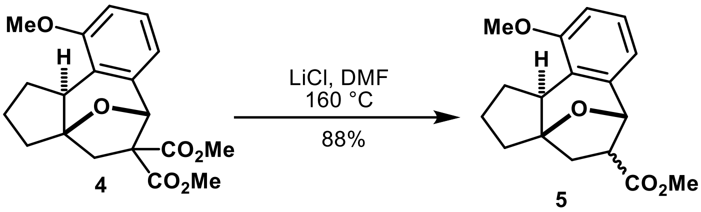
ORTEP of **4**

NMO (2.86 g, 24.4 mmol, 10.0 equiv) was added to a solution of the benzyl bromide **2** (1 g, 2.44 mmol, 1.0 equiv) in CH_3CN (25 mL, 0.1 M) under Ar. The solution was stirred at room temperature overnight until the reaction was deemed to be completed by TLC analysis. The solvent was concentrated under reduced pressure, water was added and the mixture was then diluted with DCM. The organic layer was then separated and the aqueous layer was extracted twice with DCM. The combined organic layers were washed once with water and once with brine, dried over Na_2SO_4, and then filtered and concentrated to give aldehyde cyclopropanediester **3**, which was used directly in the next step without further purification.

The brown oil obtained above was dissolved in 25 mL DCE (0.1 M), then $Sc(OTf)_3$ (240 mg, 0.48 mmol, 0.2 equiv) was added. The mixture was stirred at room temperature overnight. After filtration through Celite, the filtrate was concentrated under reduced pressure and the residue was purified by column chromatography on silica gel (EtOAc/hexane = 1/100-2/100) to obtain **4** (693 mg, 82% over two steps) as a white solid. The structure of compound **4** was confirmed by XRD.

4: mp:153-154 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.05 (t, *J* = 7.9 Hz, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.57 (d, *J* = 7.5 Hz, 1H), 5.63 (s, 1H), 3.79 (s, 3H), 3.78 (s, 3H), 3.42 (s, 3H), 2.87 (d, *J* = 13.7 Hz, 1H), 2.76 (dd, *J* = 10.2, 8.5 Hz, 1H), 2.47–2.27 (m, 2H), 2.06–1.95 (m, 2H), 1.85 (dt, *J* = 7.6, 4.7 Hz, 1H), 1.74 (dq, *J* = 14.0, 4.5, 3.9 Hz, 1H), 1.57 (dtd, *J* = 12.6, 10.0, 8.0 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 168.1, 157.7, 134.8, 126.1, 126.1, 117.8, 109.9, 90.5, 80.8, 68.6, 55.1, 53.0, 52.5, 45.7, 40.6, 36.1, 31.9, 22.7; ^{13}C DEPT 90 NMR (101 MHz, CDCl3) δ 126.1, 117.8, 109.9, 80.8, 45.7. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{19}H_{23}O_6^+$ 347.1495; found 347.1498.

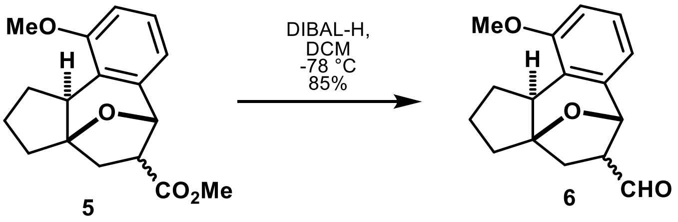
Preparation of the compound 5.



To a stirred solution of **4** (204 mg, 0.589 mmol, 1.0 equiv) in 12 mL DMF (commercial DMF was used directly without prior drying, 0.05 M), LiCl (250 mg, 5.89 mmol, 10.0 equiv) was added at room temperature under Ar. The mixture was put into a 160 °C preheated oil bath and stirred at that temperature for 2 h before it was cooled to room temperature and quenched by water, the mixture was extracted by EtOAc and washed with brine for several times, then dried over anhydrous Na_2SO_4. After the solvent was evaporated under vacuum, the residue was purified by flash chromatography (EtOAc/hexane = 4/100) to give **5** (150 mg, 88% yield) as a yellow oil.

5: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (t, *J* = 7.9 Hz, 1H), 7.04 (t, *J* = 7.9 Hz, 1H), 6.78–6.66 (m, 3H), 6.53 (d, *J* = 7.5 Hz, 1H), 5.26 (s, 1H), 5.19 (d, *J* = 7.1 Hz, 1H), 3.81 (s, 3H), 3.79 (s, 3H), 3.75 (s, 3H), 3.60–3.51 (m, 1H), 3.45 (s, 3H), 3.02 (dd, *J* = 9.5, 4.1 Hz, 1H), 2.71 (ddd, *J* = 35.2, 10.4, 8.3 Hz, 2H), 2.49–2.28 (m, 4H), 2.19–2.02 (m, 4H), 2.00 (d, *J* = 7.8 Hz, 2H), 1.94–1.72 (m, 4H), 1.65–1.49 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.2, 171.4, 158.1, 157.8, 139.9, 136.3, 126.9, 126.1, 125.6, 124.8, 117.4, 116.2, 109.6, 109.2, 90.6, 90.4, 80.0, 78.7, 55.2, 55.1, 52.9, 52.4, 52.3, 51.5, 46.6, 45.8, 36.1, 35.7, 35.4, 34.2, 32.0, 31.9, 22.9, 22.7. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{17}H_{22}O_4^+$ 289.1440; found 289.1442.

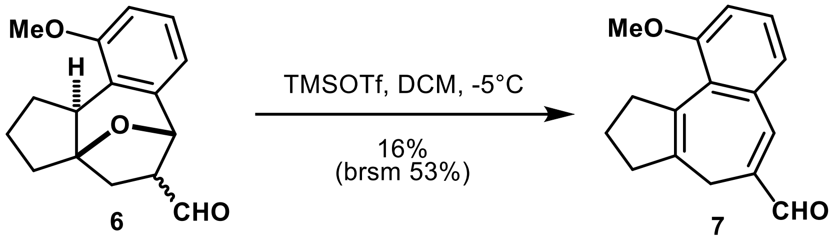
Preparation of the compound 6.



To a solution of compound **5** (210 mg, 0.728 mmol, 1.0 equiv) in dry DCM (15 mL, 0.05 M), DIBAL-H (1.5 M, 2.43 mL, 3.64 mmol, 5.0 equiv) was added dropwise at -78 °C under Ar atmosphere, and the resulting mixture was stirred at the same temperature for 15 mins. The reaction was quenched by adding MeOH dropwise. The reaction was stirred for continuous 20 mins at -78 °C, then warmed to room temperature. The solution was poured into 1 M HCl aqueous solution and stirred vigorously until the solution became clear. The mixture was extracted with DCM for 3 times. The organic layers were combined and washed with brine, dried over Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel (EtOAc/hexane = 6/100) to afford **6** (160 mg, 85%) as a colorless oil.

6: ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.72 (d, *J* = 2.0 Hz, 1H), 9.03 (d, *J* = 3.3 Hz, 1H), 7.17–7.05 (m, 2H), 6.78–6.72 (m, 2H), 6.70 (d, *J* = 7.5 Hz, 1H), 6.56 (d, *J* = 7.5 Hz, 1H), 5.31–5.06 (m, 2H), 3.81 (d, *J* = 3.8 Hz, 6H), 3.38 (dtd, *J* = 10.2, 6.9, 3.4 Hz, 1H), 2.94 (d, *J* = 8.7 Hz, 1H), 2.73 (ddd, *J* = 13.0, 10.4, 8.4 Hz, 2H), 2.47–2.40 (m, 2H), 2.36 (dd, *J* = 13.2, 3.9 Hz, 1H), 2.21 (dd, *J* = 8.5, 3.8 Hz, 2H), 2.08 (d, *J* = 11.6 Hz, 2H), 2.03 (s, 1H), 1.84 (dddd, *J* = 31.5, 16.1, 9.5, 3.3 Hz, 5H), 1.61 (dq, *J* = 20.7, 9.7, 8.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.2, 201.1, 158.1, 158.3, 139.1, 136.1, 127.1, 127.0, 125.2, 124.9, 116.7, 116.0, 109.8, 109.4, 90.9, 90.4, 78.1, 77.6, 60.5, 58.9, 55.3, 55.2, 46.7, 46.1, 35.9, 35.7, 33.2, 32.7, 32.0, 31.9, 22.8, 22.8. HRMS (ESI) m/z: [M+H]$^+$ calcd for $C_{16}H_{20}O_3^+$ 259.1334; found 259.1337.

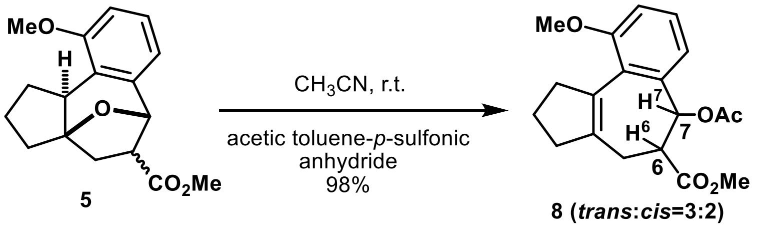
Preparation of the compound 7.



To a solution of compound **6** (87.1 mg, 0.337mmol, 1.0 equiv) in dry DCM (13.5 mL, 0.025 M), TMSOTf (609 µL, 3.37 mmol, 10.0 equiv) was added dropwise at -5 °C under Ar atmosphere, and the resulting mixture was stirred at the same temperature for 1 h. The reaction was quenched by aqueous $NaHCO_3$ solution. The mixture was extracted with DCM for 3 times. The organic layers were combined and washed with brine, dried over Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel (EtOAc/hexane = 4/100-10/100) to afford **7** (13.4 mg, 16%) as a colorless oil and 59.7 mg **6** had been recycled.

7: ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.52 (s, 1H), 7.23 (d, *J* = 8.1 Hz, 1H), 7.05 (d, *J* = 7.7 Hz, 1H), 6.91 (d, *J* = 8.1 Hz, 1H), 3.84 (s, 3H), 2.79 (t, *J* = 7.3 Hz, 2H), 2.75 (s, 2H), 2.52 (t, *J* = 7.3 Hz, 2H), 1.92–1.78 (m, 2H).; ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.0, 157.9, 148.1, 142.2, 141.8, 135.1, 133.1, 128.1, 126.6, 123.5, 111.4, 55.6, 37.1, 36.1, 24.4, 22.2. HRMS (ESI) m/z: [M+H]$^+$ calcd for $C_{16}H_{17}O_2^+$ 241.1229; found 241.1225.

Preparation of the compound 8.



To a stirred mixture of the compound **5** (58.2 mg, 0.2 mmol, 1.0 equiv) and acetonitrile (2 ml) was added acetic toluene-p-sulphonic anhydride (85.7 mg, 0.4 mmol, 2.0 equiv) and stirring was continued for 3 hours. The mixture was quenched by aqueous sodium hydrogen carbonate. The mixture was extracted with EtOAc for 3 times. The organic layers were combined and washed with brine, dried over Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel (EtOAc/hexane = 4/100) to afford **8** (64.9 mg, 98%, *trans*: *cis* = 3: 2) as a colorless oil.

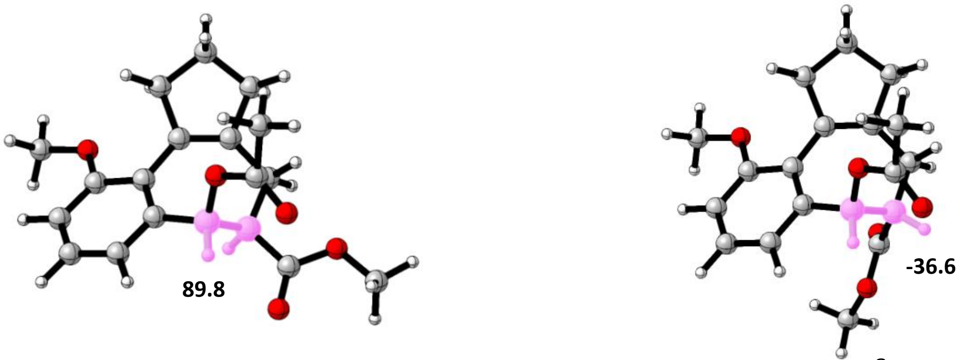
8: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (q, *J* = 8.2 Hz, 1H), 7.07 (d, *J* = 7.7 Hz, 0.4H), 6.97 (d, *J* = 7.7 Hz, 0.6H), 6.89–6.81 (m, 1H), 5.99 (d, *J* = 9.9 Hz, 0.6H), 5.86 (d, *J* = 6.7 Hz, 0.4H), 3.80 (s, 3H), 3.73 (s, 1.7H), 3.66 (s, 1.3H), 3.22 (td, *J* = 9.4, 8.4, 2.7 Hz, 0.6H), 3.11 (tt, *J* = 14.7, 7.2 Hz, 1H), 2.70–2.20 (m, 5.4H), 2.11 (s, 1.6H), 2.10 (s, 1.4H), 2.05–1.94 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.7, 172.5, 169.6, 169.3, 157.0, 156.9, 140. 8, 140.3, 138.5, 137.34, 135.7, 135.5, 127.6, 127.4, 125.0, 118.4, 116.4, 110.2, 110.2, 73.6, 73.0, 56.5, 55.4, 54.2, 52.1, 51.6, 37.2, 36.9, 35.7, 35.3, 27.2, 27.0, 23.2, 23.1, 21.1, 21.0; ^{13}C DEPT-90 NMR (101 MHz, $CDCl_3$) δ 127.6, 127.4, 118.4, 116.4, 110.2, 110.2, 73.6, 73.0, 56.5, 54.2; ^{13}C DEPT-135 NMR (101 MHz, $CDCl_3$) δ 127.6, 127.4, 118.4, 116.4, 110.2, 110.2, 73.6, 73.0, 56.5, 55.4, 54.2, 52.0, 51.6, 37.2, 36.9, 35.7, 35.3, 27.2, 27.0, 23.2, 23.1, 21.1, 21.0. HRMS (ESI) m/z: [M+Na]$^+$ calcd for $C_{19}H_{22}NaO_5^+$ 353.1365; found 353.1362.

According to the DFT Calculations, the dihedral angle of H7-C7-C6-H6 was -36.64° in *trans*-**8** and was 89.85° in *cis*-**8**, which indicated that the coupling constant of H7 (hydrogen in benzyl) in *trans*-**8** was bigger than that in *cis*-**8**. Considering of the practical coupling constant of H7 at 5.99 ppm and 5.86 ppm in ^{1}H-NMR, we got a conclusion that the ratio of *trans*-/*cis*-isomers was 3:2.

3. DFT Calculations

3.1 Computational Methods

All calculations were carried out using Gaussian 09[2]. Geometry optimizations were performed using the B3LYP-D3 functional[3-6] and 6-31G (d, p) basis set. Vibrational frequencies were calculated at the same level of theory to obtain the thermal correction and confirm the optimized geometries as minima (no imaginary frequencies) at the same level of theory. Molecular structure figures were prepared using CYLview20[7].



cis-**8**   *trans*-**8**

Figure S1. Comparison of *cis*-8 and *trans*-8 (Calculated at B3LYP-D3/6-31G**; Gas Phase)

Cartesian Coordinates for Optimized Structures

Compound **8** (*cis*)

C	-3.33816200	-2.13504800	0.25857200
C	-2.35978900	-2.99538300	0.74761800
C	-1.04444700	-2.55749800	0.83988400
C	-0.69942300	-1.25317200	0.47442300
C	-1.67193100	-0.33824800	0.00562200
C	-2.99950900	-0.83186100	-0.12108300
C	0.76845100	-0.90504300	0.51157700
C	1.27340100	-0.46481400	-0.89083500
C	1.10569400	1.03564900	-1.12173400
C	-0.21324100	1.63426700	-0.73586600
C	-1.36605500	1.08217600	-0.29132200
C	-0.32937500	3.14474700	-0.82813300
C	-1.53600400	3.45756900	0.07077600

C	-2.39629800	2.18776900	-0.03634300
C	2.68301600	-0.99978800	-1.12420000
O	2.97163900	-2.17048600	-0.99887100
O	3.55439500	-0.05019600	-1.51428800
C	4.91851100	-0.49180300	-1.61952900
O	-3.91493100	0.02520500	-0.66540300
C	-5.27148400	-0.37556900	-0.75933400
O	0.95363000	0.12865900	1.51904800
O	3.19869500	-0.16802100	1.47796400
C	2.21427200	0.38653500	1.92091900
C	2.21796400	1.45954600	2.98255300
H	-4.35720500	-2.48687300	0.16129700
H	-2.62587400	-4.00612000	1.04115000
H	-0.26982800	-3.23011600	1.19483100
H	1.34559000	-1.77982300	0.81760300
H	0.65294200	-1.01717600	-1.60500900
H	1.89139900	1.56778500	-0.57090900
H	1.31636100	1.25898200	-2.17609000
H	-0.50990000	3.44984200	-1.87104200
H	0.59179400	3.65200100	-0.51422000
H	-1.19151300	3.58249900	1.10415000
H	-2.07218100	4.36778000	-0.21523000
H	-3.09607500	2.25857100	-0.87588300
H	-3.00090700	1.99573400	0.85532600
H	5.48477000	0.37669400	-1.95614300
H	5.27144800	-0.82874900	-0.64178900
H	5.00650000	-1.31273800	-2.33554600
H	-5.80463700	0.47758400	-1.18184200
H	-5.39612300	-1.24101700	-1.42328200
H	-5.69584300	-0.61428100	0.22469400
H	3.24242700	1.66267700	3.29205300
H	1.75506800	2.36832600	2.58683200
H	1.62082000	1.13694800	3.84006600

Compound **8** (*trans*)

C	-2.96182400	0.81690800	-1.47053000
C	-2.11592800	1.78500200	-2.00628300
C	-0.75598600	1.73368200	-1.73271000
C	-0.22134700	0.70744000	-0.94520000
C	-1.04517900	-0.30812200	-0.40786900
C	-2.43910200	-0.20669100	-0.67474700
C	1.24905500	0.81194900	-0.61288600
C	1.50679200	0.91872600	0.91612900

C	1.53728000	-0.44191200	1.61499300
C	0.56649100	-1.49591300	1.17537500
C	-0.51015600	-1.45516400	0.35956500
C	0.78372800	-2.89475300	1.72200800
C	-0.01137500	-3.77775000	0.74737400
C	-1.13876900	-2.84855200	0.26643100
C	0.48532200	1.86224600	1.53035400
O	-0.27128400	1.60120600	2.43854500
O	0.53201100	3.06753600	0.91652200
C	-0.48286800	4.00117700	1.32229100
O	-3.23636500	-1.14488200	-0.08329400
C	-4.63161400	-1.12610000	-0.33081800
O	1.93890000	-0.33509900	-1.17461900
O	3.92999100	0.71656400	-0.92032100
C	3.28336300	-0.24795600	-1.27230800
C	3.84765800	-1.51464500	-1.86852000
H	-4.02534100	0.87175200	-1.66443300
H	-2.52464500	2.57837100	-2.62454800
H	-0.09312900	2.50038400	-2.12071900
H	1.65956000	1.70706800	-1.08258400
H	2.48587100	1.39364700	1.03545600
H	1.41600400	-0.27691500	2.69357700
H	2.54512800	-0.86048500	1.49286400
H	0.38966100	-2.96571800	2.74798900
H	1.84718400	-3.16001200	1.78117100
H	0.63066800	-4.04764800	-0.09951200
H	-0.37932500	-4.70533000	1.19676000
H	-2.01001900	-2.91877400	0.92608200
H	-1.49343400	-3.08055100	-0.74277700
H	-0.27756800	4.91873000	0.77110500
H	-1.47256200	3.61365400	1.06526000
H	-0.43773000	4.17894100	2.39939600
H	-5.04286900	-1.97102600	0.22366000
H	-5.09681800	-0.19942100	0.02999400
H	-4.85908700	-1.25054300	-1.39784200
H	4.93210700	-1.43551200	-1.93401800
H	3.56694800	-2.36942800	-1.24617900
H	3.42012900	-1.68121500	-2.86126700

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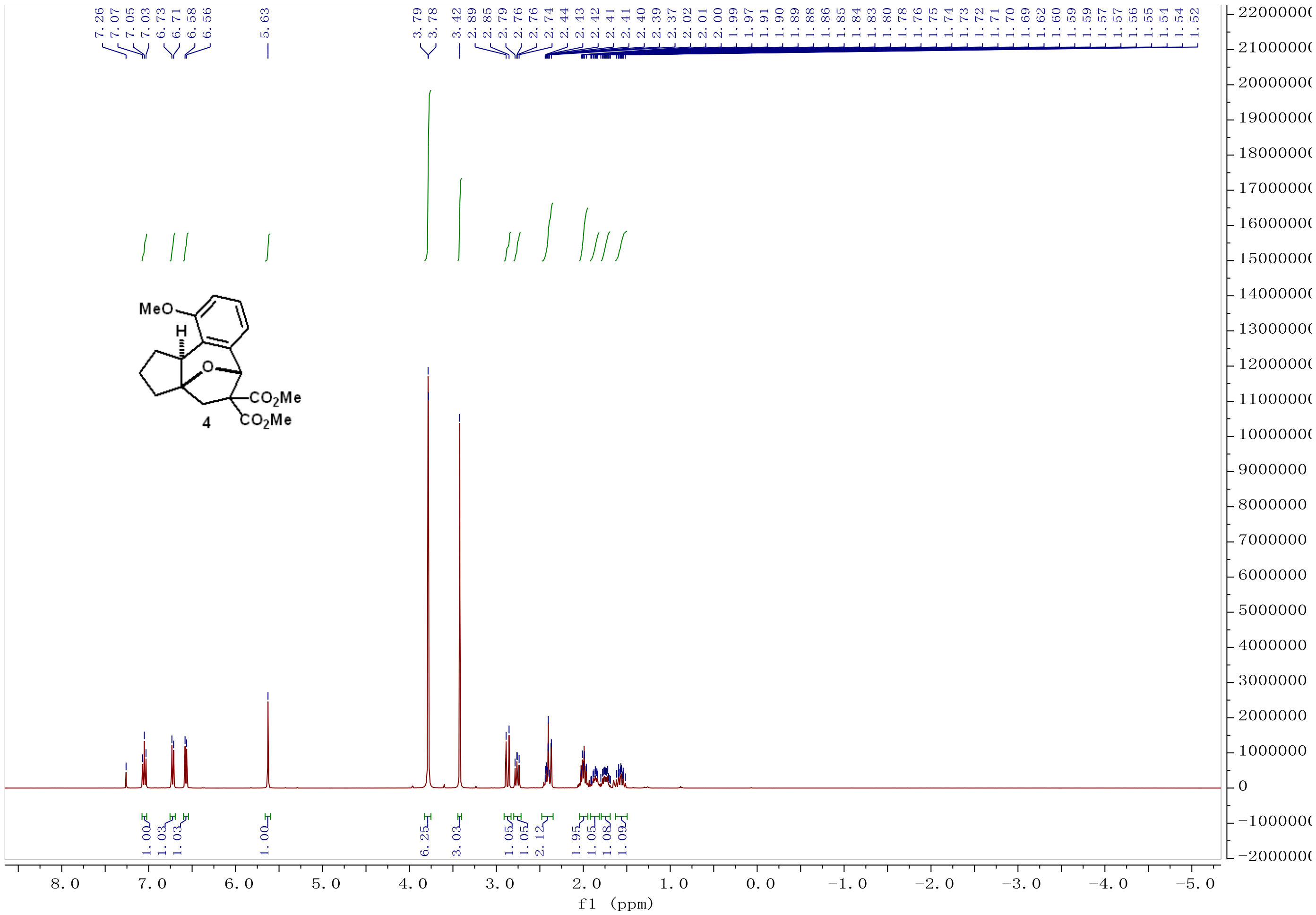
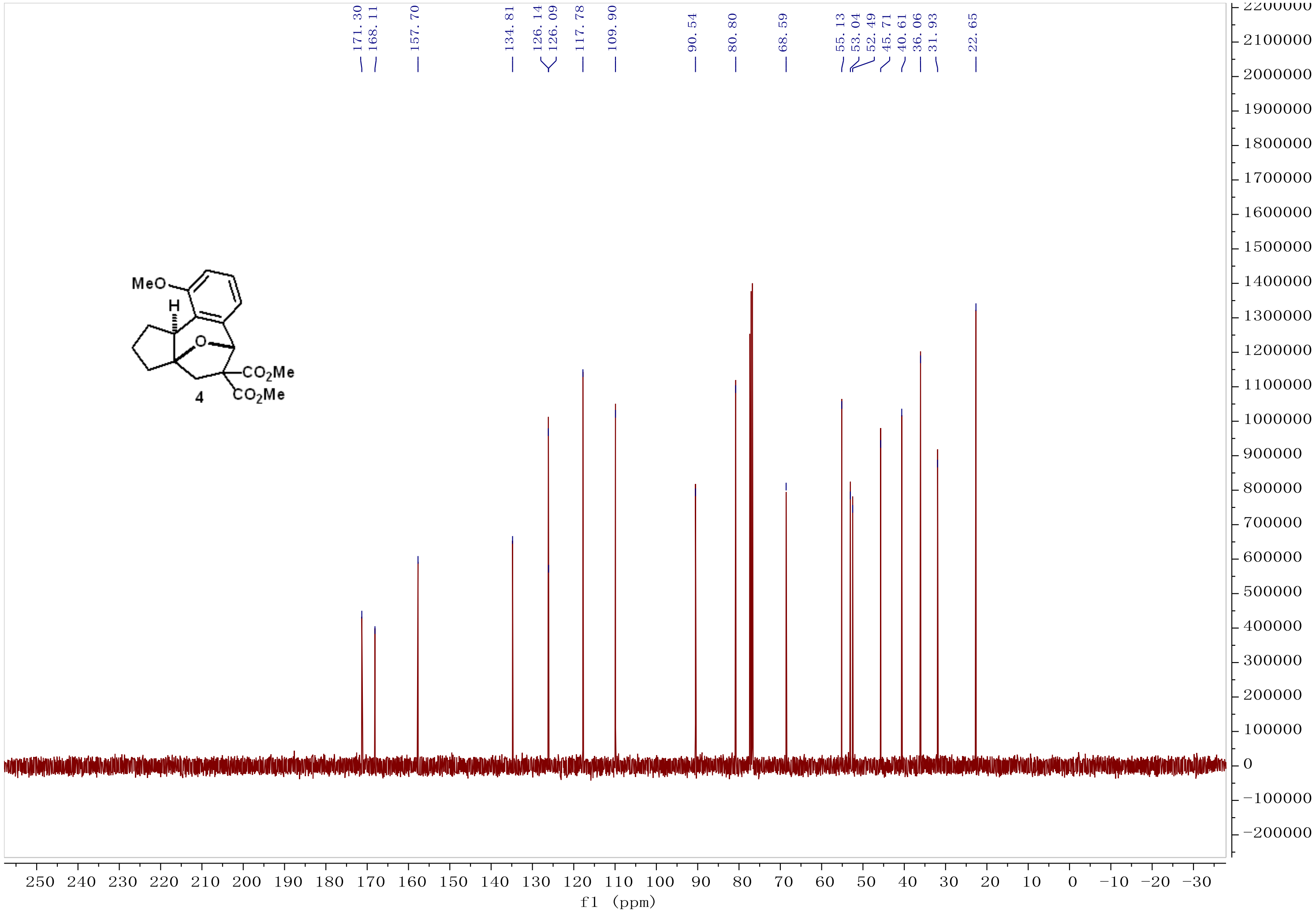
(7) CYLview20; Legault, C. Y., Université de Sherbrooke, 2020 (http://www.cylview.org)

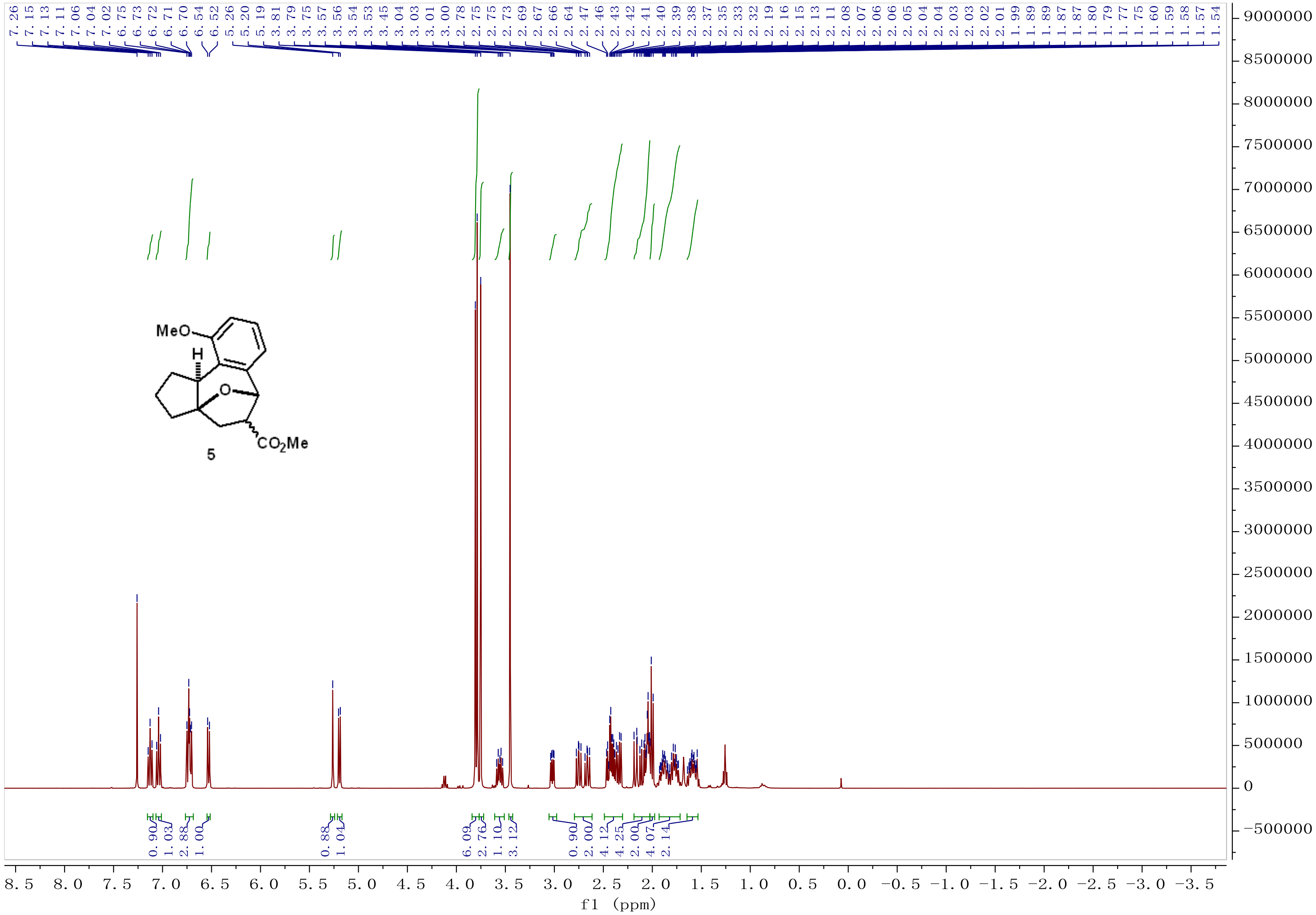
5. NMR Spectra



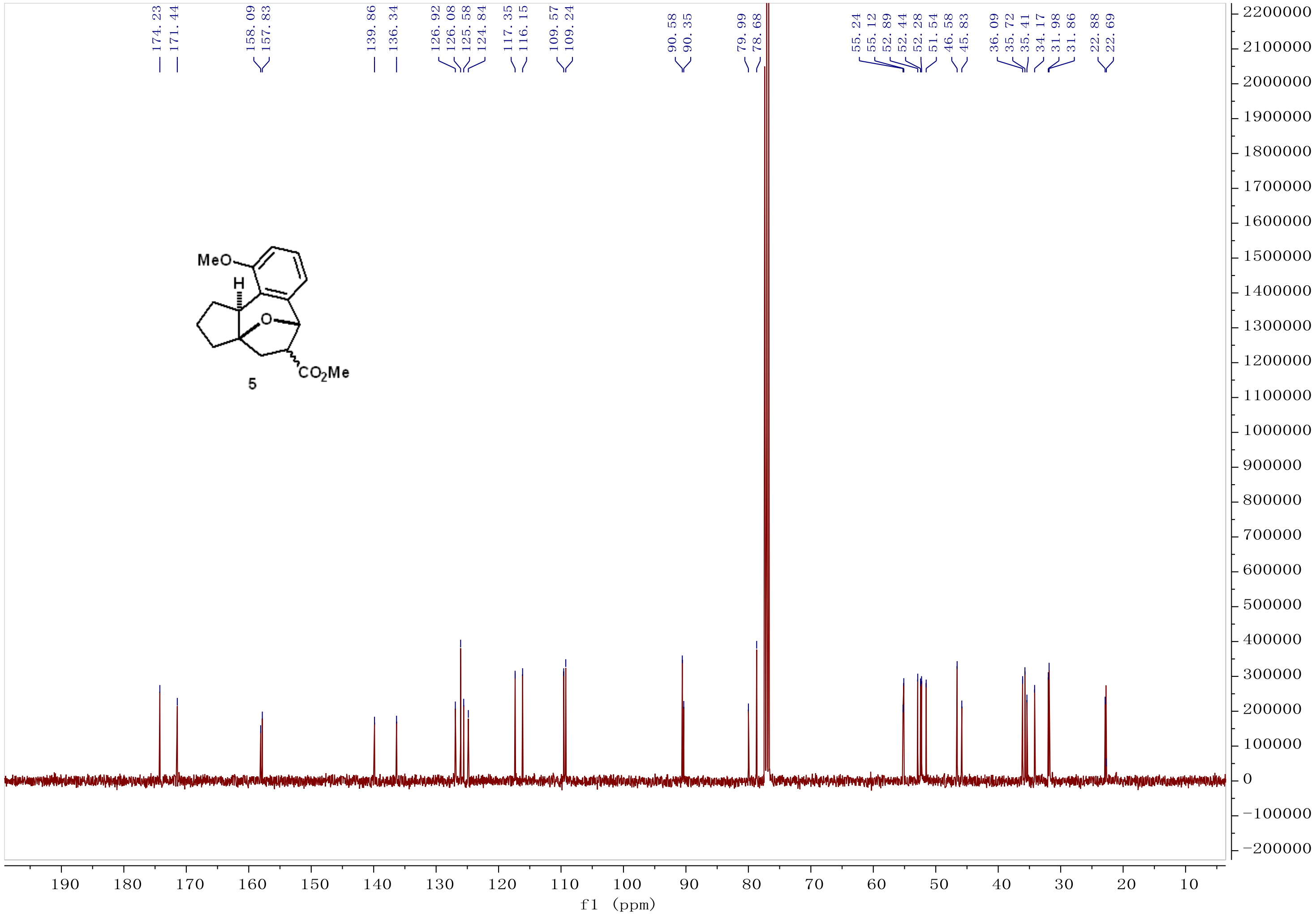
^{1}H NMR spectrum for compound **4** ($CDCl_3$, 400 MHz)



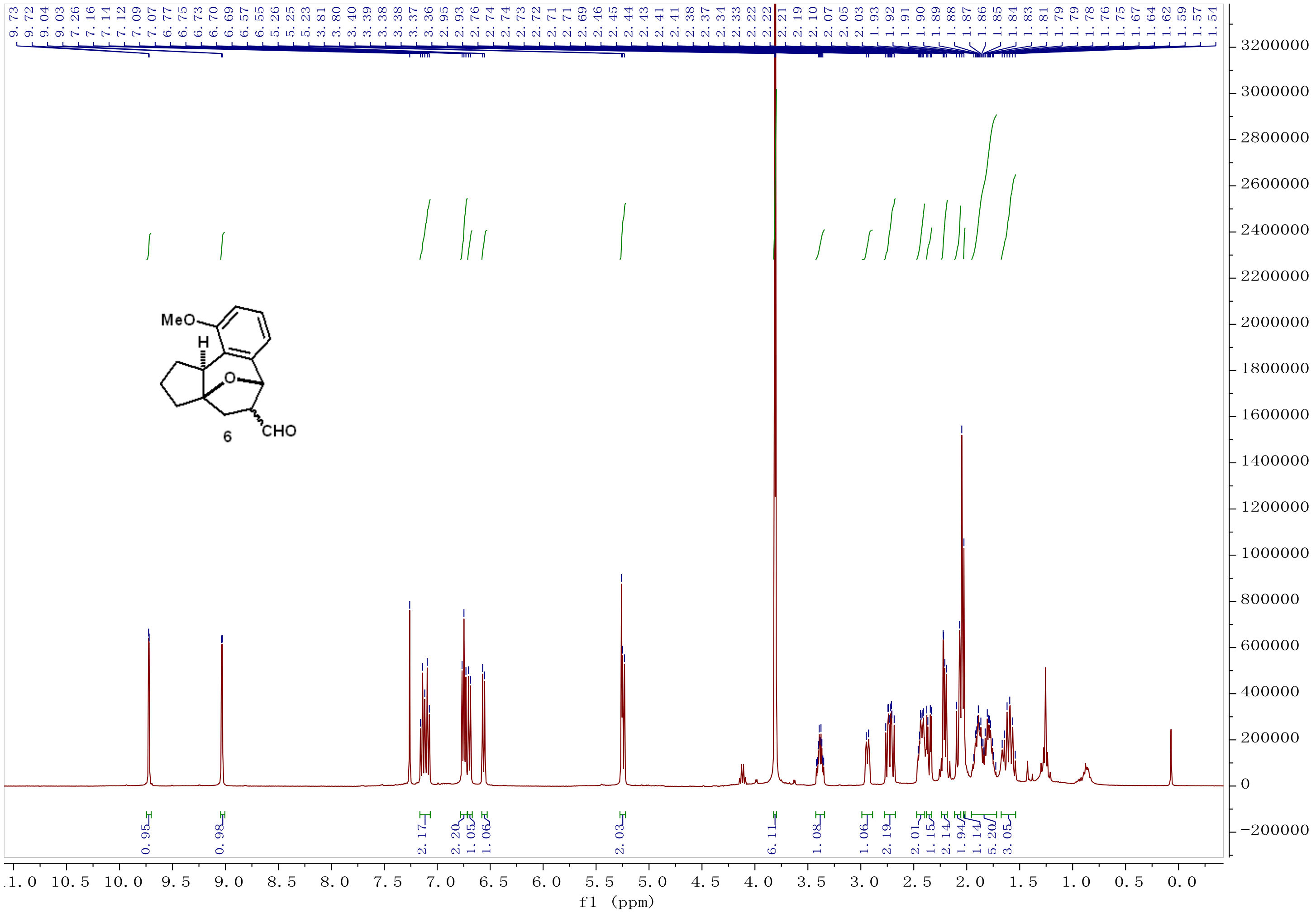
^{13}C NMR spectrum for compound **4** ($CDCl_3$, 101 MHz)



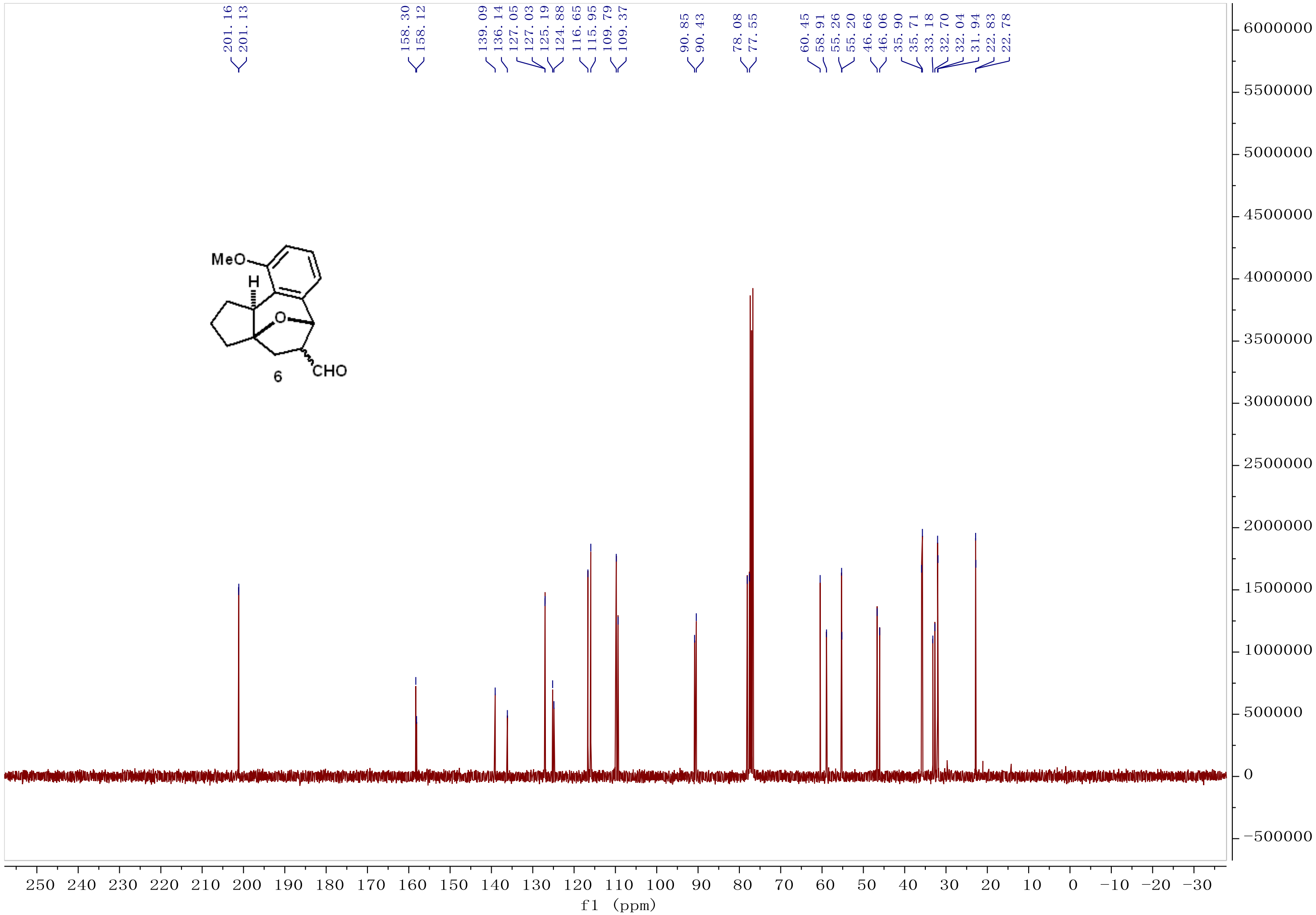
^{1}H NMR spectrum for compound **5** ($CDCl_3$, 400 MHz)



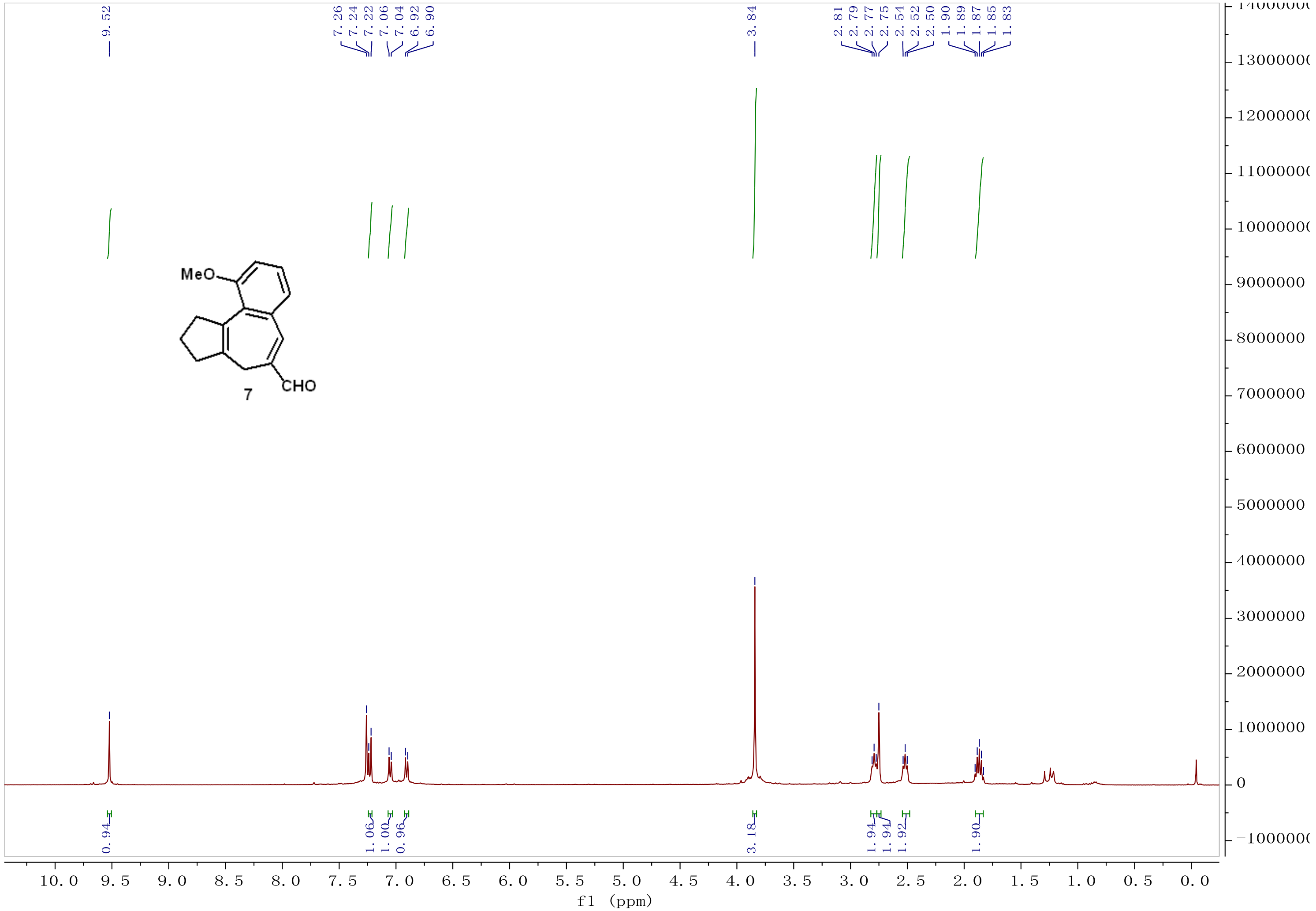
^{13}C NMR spectrum for compound **5** ($CDCl_3$, 101 MHz)



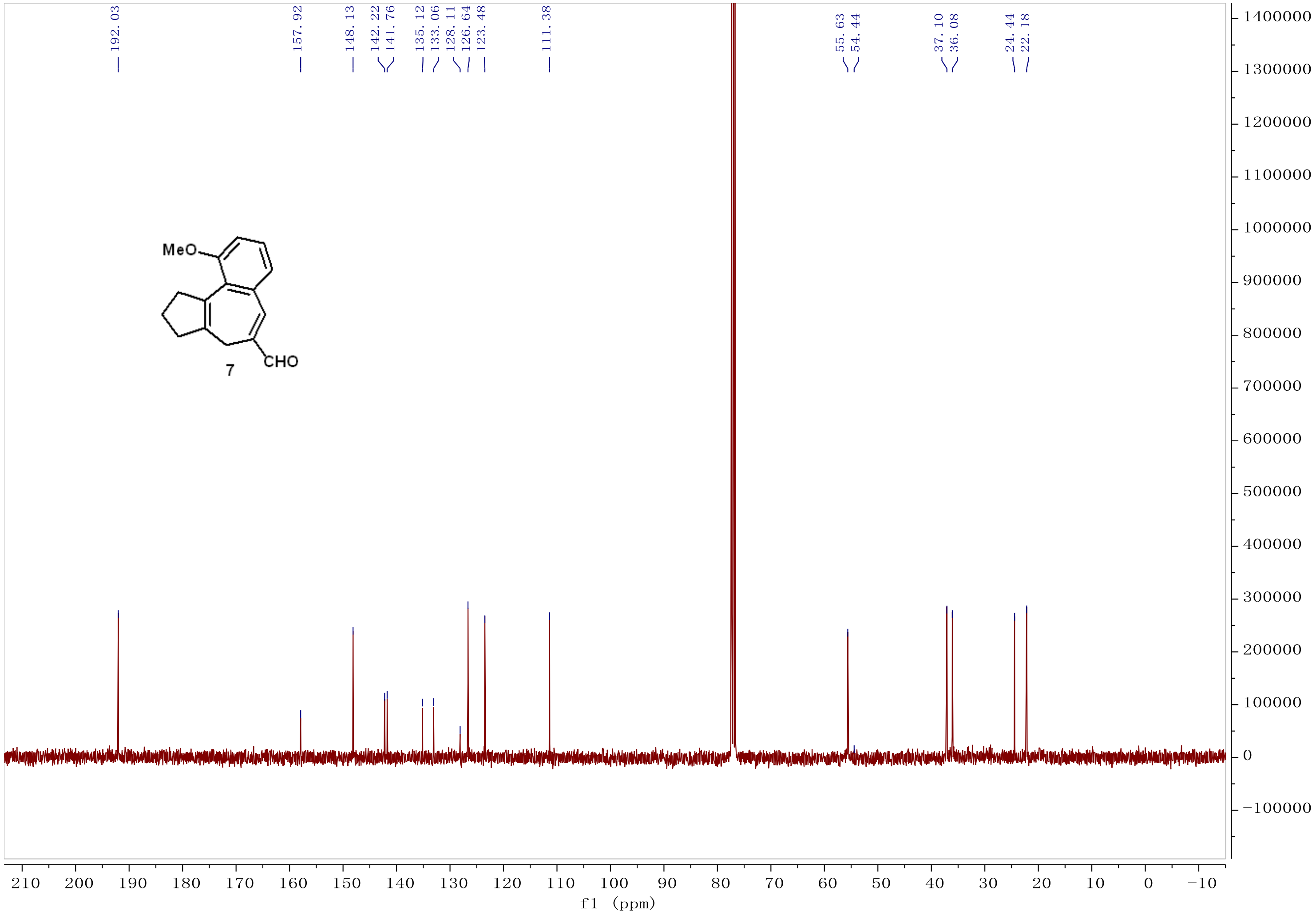
^{1}H NMR spectrum for compound **6** (CDCl$_3$, 400 MHz)



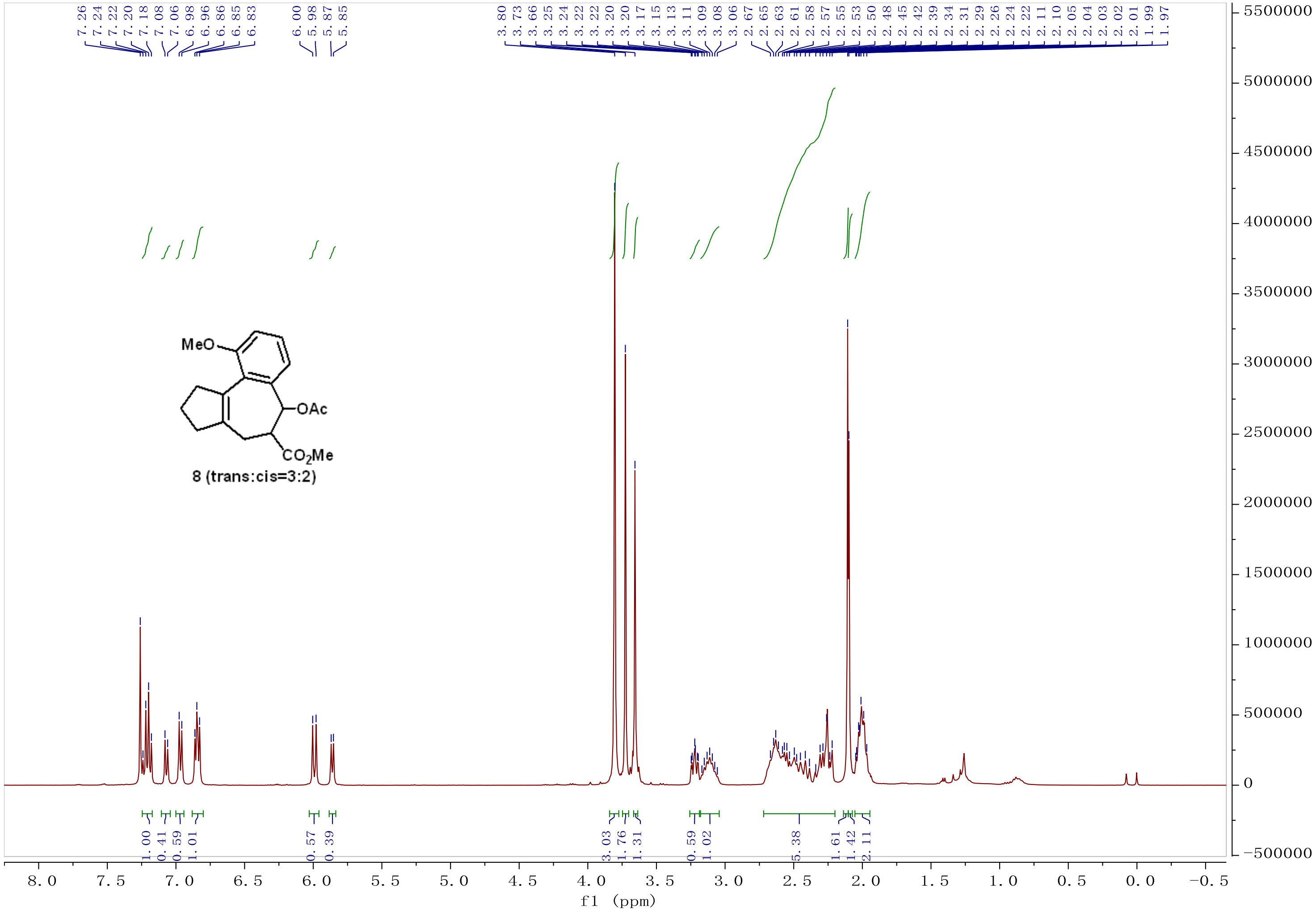
^{13}C NMR spectrum for compound **6** ($CDCl_3$, 101 MHz)



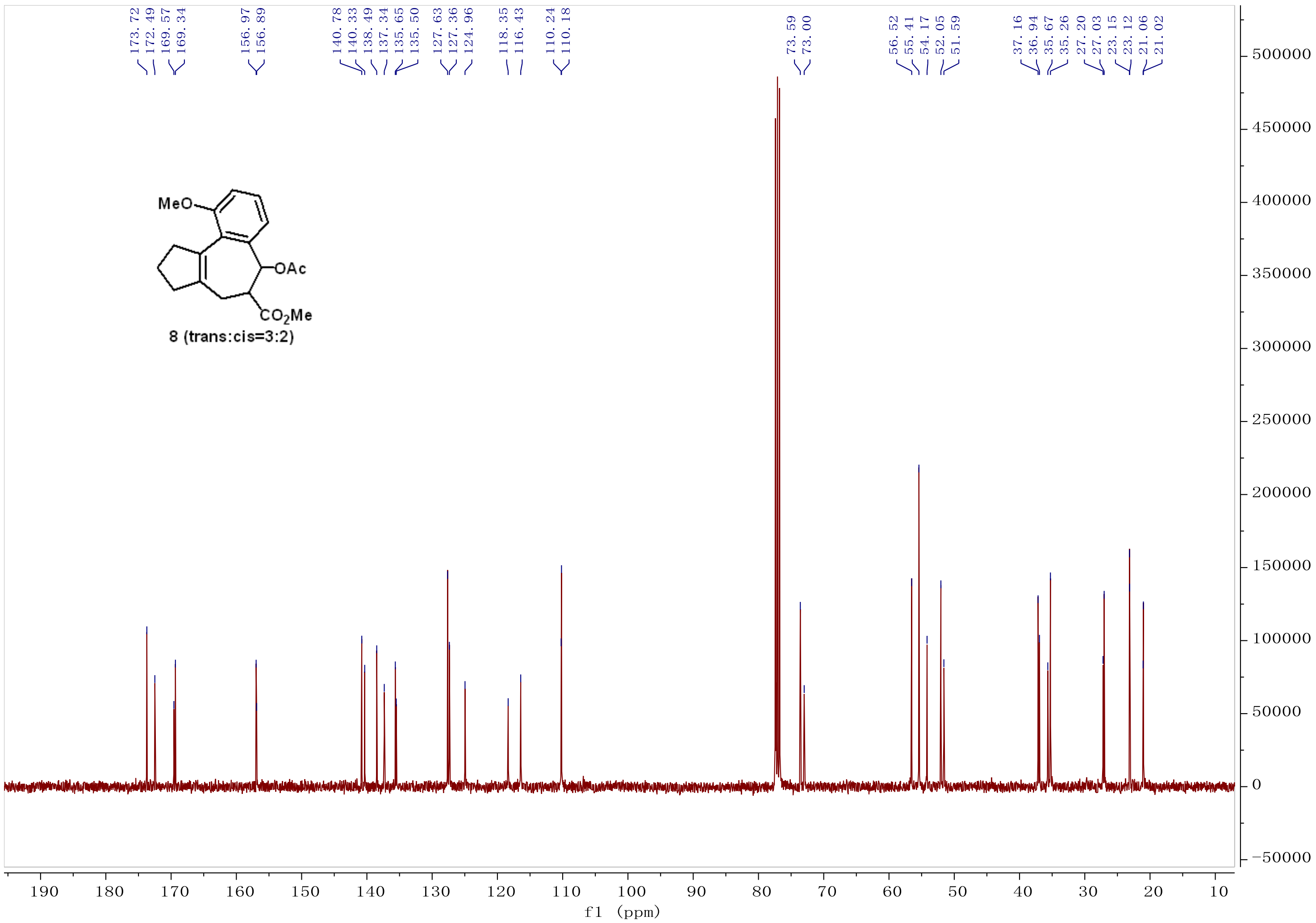
^{1}H NMR spectrum for compound **7** (CDCl$_3$, 400 MHz)



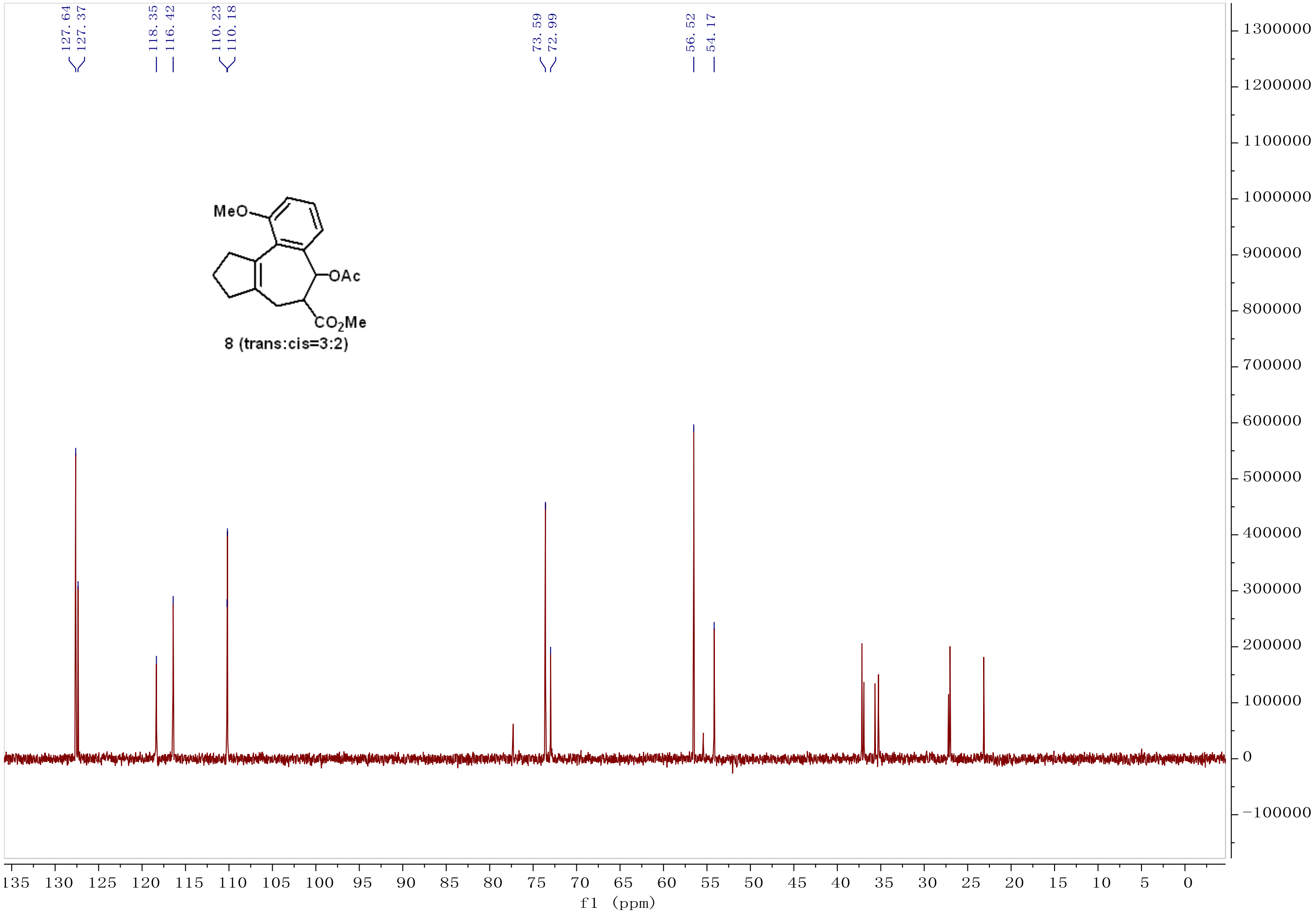
^{13}C NMR spectrum for compound **7** (CDCl$_3$, 101 MHz)



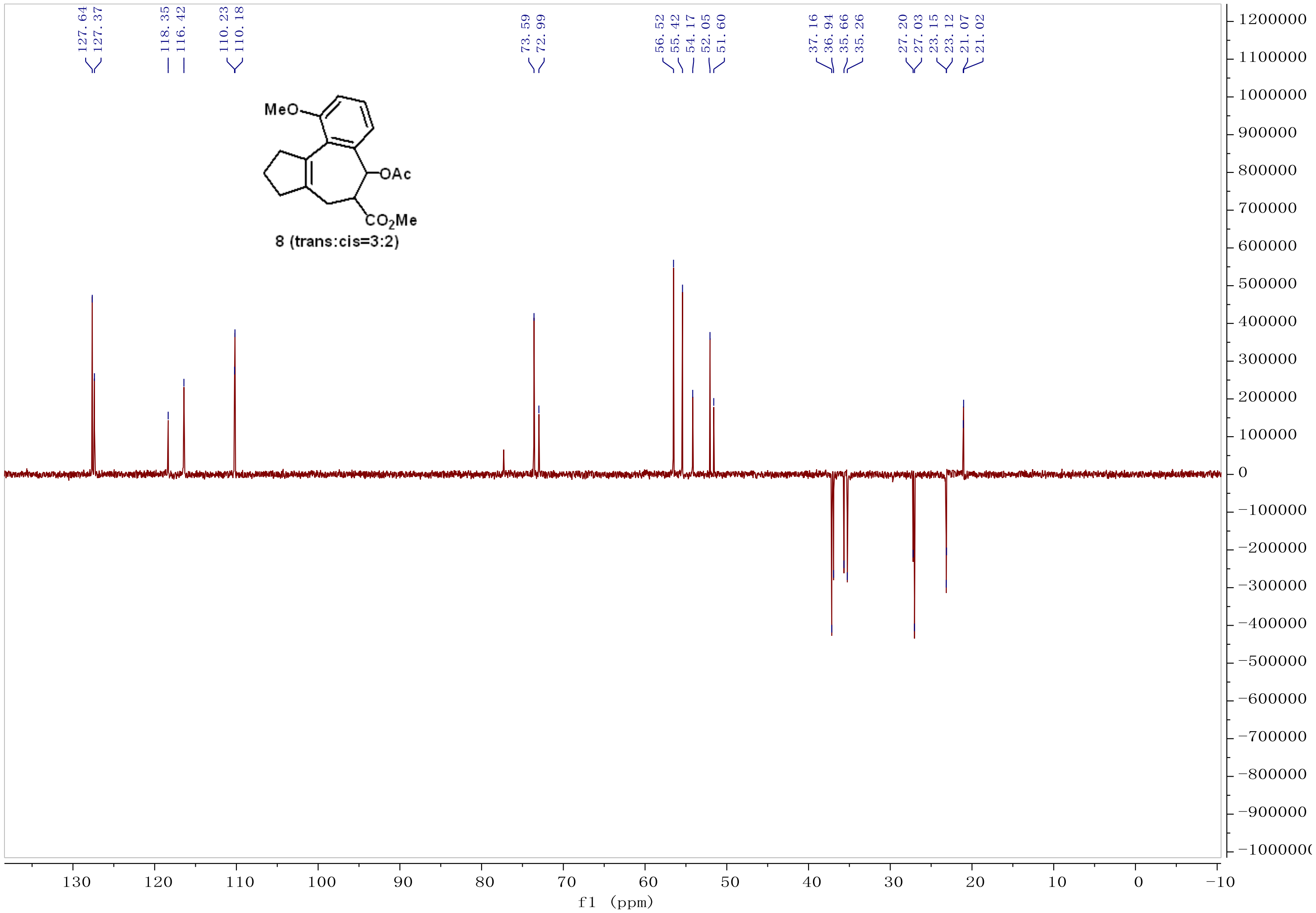
^1H NMR spectrum for compound **8** ($CDCl_3$, 400 MHz)



^{13}C NMR spectrum for compound **8** ($CDCl_3$, 101 MHz)



^{13}C DEPT-90 NMR spectrum for compound **8** ($CDCl_3$, 101 MHz)



^{13}C DEPT-135 NMR spectrum for compound **8** ($CDCl_3$, 101 MHz)

6. X-ray Crystal Structure and Data

The preparation of crystal 4: A colorless stick-shaped crystal of **4** for X-ray diffraction was obtained by slowly volatilizing a saturated solution of **4** in a 20 mL sample bottle with the mixed solvent of dichloromethane / hexane (1:1) at room temperature. The X-ray intensity data was measured on a Rigaku 007 Saturn 70 single crystal diffractometer (Mo).

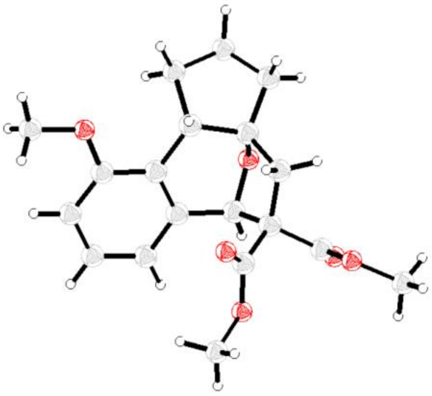


Figure S2. ORTEP drawing of compound 4. Thermal ellipsoids are shown at the 50% probability level and the hydrogen atoms are not shown.

Table S3. Crystal data and structure refinement for compound 4

Identification code	2110705
Empirical formula	$C_{19}H_{22}O_6$
Formula weight	346.36
Temperature/K	113.15
Crystal system	monoclinic
Space group	C2/c
a/Å	27.2898(10)
b/Å	7.6810(3)
c/Å	16.4376(6)
α/°	90
β/°	98.219(4)
γ/°	90
Volume/Å^3	3410.1(2)
Z	8
ρcalcg/cm^3	1.349
μ/mm^{-1}	0.100
F(000)	1472.0
Crystal size/mm^3	0.24 × 0.22 × 0.2
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.464 to 67.238
Index ranges	$-40 \leq h \leq 41, -11 \leq k \leq 11, -24 \leq l \leq 24$
Reflections collected	24108
Independent reflections	6323 [R_{int} = 0.0517, R_{sigma} = 0.0463]
Data/restraints/parameters	6323/0/229

Goodness-of-fit on F2	1.025
Final R indexes [I>=2σ (I)]	R_1 = 0.0497, wR_2 = 0.1145
Final R indexes [all data]	R_1 = 0.0680, wR_2 = 0.1273
Largest diff. peak/hole / e Å^{-3}	0.40/-0.23