Electronic Supplementary Information (ESI)

Enantioselective Total Synthesis of Colomitides and their absolute configuration determination and Structural Revision

Hongguang Yang,[a] Xiaoyu Liu,[a] Xiaoyu Li,[a] Xiang Shi,[a] Feilong Yang,[a] Xiaozhen Jiao,[a] Ping Xie,[a]**

[a] State Key Laboratory of Bioactive Substances and Functions of Natural Medicines, Institute of Materia Medica, Beijing Key Laboratory of Active Substances Discovery and Druggability Evaluation , Peking Union Medical College and Chinese Academy of Medical Sciences, Beijing 100050, PR China.
E-mail: xp@imm.ac.cn

Table of contents

Experimental.....2

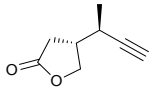
^{1}H and ^{13}C NMR Spectra of Reported Compounds 5

^{1}H NMR analysis of the ratio of 2/1c epimers 24

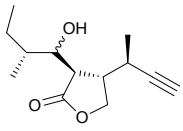
X-Ray Crystallographic Data.....25

NMR Comparison Table of natural colomitide B and synthetic 2.....27

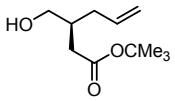
Experimental



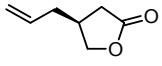
(*R*)-4-((*R*)-but-3-yn-2-yl)dihydrofuran-2(3H)-one (**9a**). **9a** (490 mg) was prepared from **15** (700 mg, 5.0 mmol) following the procedure described for **9b** in 70% yield. $[\alpha]_D^{18.5}$ = -56.8 (*c* 0.38, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.46 (dd, *J* = 9.2, 7.1 Hz, 1H), 4.23 (dd, *J* = 9.2, 6.7 Hz, 1H), 2.66–2.57 (m, 3H), 2.44 (dd, *J* = 11.8, 8.9 Hz, 1H), 2.17 (d, *J* = 2.0 Hz, 1H), 1.23 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (100 MHz, $CHCl_3$): δ 176.4, 84.1, 71.2, 70.6, 40.1, 32.6, 28.5, 18.7. IR (film)ν_{max}/cm^{-1} 3283, 2978, 2937, 1778, 1177, 1037, 1004, 656. HRMS (ESI): Calcd for $C_8H_{11}O_2$ $[M+H]^+$ 139.0753, found 139.0748.



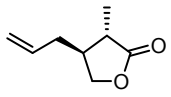
(3R,4S)-4-((R)-but-3-yn-2-yl)-3-((R)-1-hydroxy-2-methylbutyl)dihydrofuran-2(3H)-one (**7a**). To a solution of **9a** (150 mg, 1.1 mmol) in CH_2Cl_2 (5 mL) was added Bu_2BOTf (1 M in CH_2Cl_2, 1.63 mL, 1.6 mmol) at -78 °C under argon. After stirred for 15 min, EtN^iPr_2 (0.29 mL, 1.7 mmol) was added and the solution was stirred for 1 hour at -78 °C. A solution of **8a** (187 mg, 2.2 mmol) in CH_2Cl_2 was added within 1 hour by syringe pump. After the system stirred for 4 hours at -78 °C, the reaction was quenched by addition of aqueous phosphate buffer (pH 7.4, 4mL), MeOH (2 mL), 30% H_2O_2 (1 mL). The mixture was warmed to rt and stirred for 1 hour, CH_2Cl_2 (6 mL) was added to the mixture, the organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (5 mL x 3). The combined organic layers were washed with saturated aqueous Na_2SO_3 solution and dried over Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel (CH_2Cl_2 / petroleum ether = 3:2) to recycle **9a** (100 mg) and then eluent with CH_2Cl_2 / MeOH = 50:1 to give **7a** (60 mg, 25% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.40 (t, *J* = 8.7 Hz, 1H), 4.23 (m, 1H), 3.59 (dd, *J* = 6.4, 5.1 Hz, 0.4H), 3.44 (dd, *J* = 7.5, 4.0 Hz, 0.5H), 2.93 – 2.83 (m, 1H), 2.79 (m, 1H), 2.63 (m, 0.7H), 2.48 (m, 0.5H), 2.22 (dd, *J* = 5.7, 2.4 Hz, 1H), 1.94 – 1.87 (m, 0.5H), 1.72 – 1.63 (m, 1H), 1.60 – 1.50 (m, 0.5H), 1.38 – 1.30 (m, 0.5H), 1.30 – 1.24 (m, 0.6H), 1.22 (dd, *J* = 7.1, 2.6 Hz, 3H), 0.99 (dd, *J* = 6.7, 3.5 Hz, 3H), 0.94 (td, *J* = 7.4, 1.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.0, 177.7, 83.5, 83.4, 75.4, 74.3, 72.7, 72.5, 68.2, 67.9, 45.9, 45.5, 42.8, 42.6, 37.4, 27.3, 27.1, 26.4, 25.6, 24.2, 18.6, 18.5, 15.8, 13.4, 11.4, 10.9. HRMS (ESI): Calcd for $C_{13}H_{21}O_3$ $[M+H]^+$ 225.1485, found 225.2583.



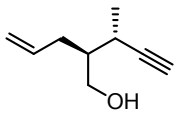
(*S*)-tert-butyl 3-(hydroxymethyl)hex-5-enoate (*ent*-**13**) *ent*-**13** (10 g) was prepared from **Evans' auxiliary** (22 g 0.059 mol) following the procedure described for **13** in 85% yield. $[\alpha]_D^{20.6}$ = -6.3 (*c* 1.20, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.84–5.73 (m, 1H), 5.11–5.02 (m, 2H), 3.64 (dd, *J* = 11.0, 4.7 Hz, 1H), 3.52 (dd, *J* = 11.0, 6.0 Hz, 1H), 2.29 (dd, *J* = 6.4, 1.2 Hz, 2H), 2.19–2.03 (m, 3H), 1.45 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 172.9, 136.0, 116.9, 80.6, 65.4, 37.5, 37.5, 35.6, 28.0. IR (film)ν_{max}/cm^{-1} 3431, 2978, 2930, 1728, 1368, 1155, 1037, 915. HRMS (ESI): Calcd for $C_{12}H_{20}O_3Na$ $[M+Na]^+$ 223.1304, found 223.1289.



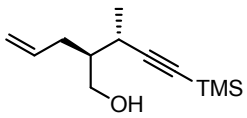
(*S*)- 4-allyldihydrofuran-2(3H)-one (*ent*-**14**) *ent*-**14** (5.86 g) was prepared from *ent*-**13** (10 g 0.050 mol) following the procedure described for **14** in 93% yield. $[\alpha]_D^{20.6}$ = -26.5 (*c* 1.10, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.79–5.67 (m, 1H), 5.16–5.07 (m, 2H), 4.40 (dd, *J* = 9.1, 7.4 Hz, 1H), 4.00 (dd, *J* = 9.1, 5.9 Hz, 1H), 2.68–2.58 (m, 2H), 2.29–2.20 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 176.9, 134.2, 117.8, 72.6, 37.1, 34.7, 33.8. IR (film)ν_{max}/cm^{-1} 2978, 2910, 1779, 1172, 1014, 920. HRMS (ESI): Calcd for $C_7H_{10}O_2Na$ $[M+Na]^+$ 149.0573, found 149.0223.



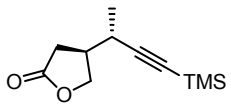
(3*S*,4*S*)-4-allyl-3-methyldihydrofuran-2(3H)-one (*ent*-**10**) *ent*-**10** (4.7 g) was prepared from *ent*-**14** (5.85 g 0.046 mol) following the procedure described for **10** in 72% yield. $[\alpha]_D^{20.7}$ = -58.6 (*c* 1.05, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): δ 5.81–5.69 (m, 1H), 5.16–5.08 (m, 2H), 4.37 (dd, *J* = 9.1, 7.0 Hz, 1H), 3.82 (t, *J* = 9.1 Hz, 1H), 2.45–2.36 (m, 1H), 2.29–2.12 (m, 3H), 1.27 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 179.59, 134.1, 117.7, 70.8, 42.9, 39.8, 35.9, 13.9. IR (film)ν_{max}/cm^{-1} 2976, 2905, 2340, 1776, 1642, 1385, 1177, 1014, 921. HRMS (ESI): Calcd for $C_8H_{13}O_2$ $[M+H]^+$ 141.0910, found 141.0899.



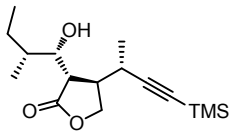
(*S*) -2-((S)-but-3-yn-2-yl)pent-4-en-1-ol (*ent*-**15**) *ent*-**15** (1.7 g) was prepared from *ent*-**10** (2.5 g 17.83 mmol) following the procedure described for **15** in 70% yield. $[\alpha]_D^{20.9}$ = +43.6 (*c* 0.99, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): δ 5.81 (m, 1H), 5.15–5.04 (m, 2H), 3.80 (dd, *J* = 11.3, 5.3 Hz, 1H), 3.72 (dd, *J* = 11.3, 5.3 Hz, 1H), 2.68–2.60 (m, 1H), 2.33–2.18 (m, 2H), 2.11 (d, *J* = 2.5 Hz, 1H), 1.70 (s, 1H), 1.66–1.57 (m, 1H), 1.24 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.5, 116.8, 87.3, 69.9, 63.3, 44.8, 33.9, 26.6, 18.6. IR (film)ν_{max}/cm^{-1} 3303, 3077, 2976, 2935, 2110, 1640, 1442, 1041, 916. 636. HRMS (ESI): Calcd for $C_9H_{15}O$ $[M+H]^+$ 139.1117, found 139.1115.



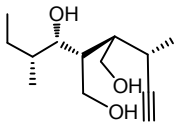
(*S*)-2-((*S*)-4-(trimethylsilyl)but-3-yn-2-yl)pent-4-en-1-ol (*ent*-**16**) *ent*-**16** (2.2 g) was prepared from *ent*-**15** (1.5 g 10.85 mmol) following the procedure described for **16** in quant yield. $[\alpha]_D^{20.9}$ = +40.0 (*c* 0.98, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): δ 5.81 (m, 1H), 5.15–5.01 (m, 2H), 3.81 (dd, *J* = 11.4, 4.7 Hz, 1H), 3.70 (dd, *J* = 11.4, 5.3 Hz, 1H), 2.61 (dd, *J* = 7.1, 5.5 Hz, 1H), 2.24 (q, *J* = 7.1 Hz, 2H), 1.85 (brs, 1H), 1.58 (q, 6.7 Hz, 1H), 1.22 (d, *J* = 7.1 Hz, 3H), 0.15 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.5, 116.7, 110.2, 86.4, 63.5, 45.1, 34.2, 27.9, 18.9. IR (film)ν_{max}/cm^{-1} 3332, 3078, 2960, 2898, 2165, 1640, 1443, 1250, 1041, 916, 844, 760. HRMS (ESI): Calcd for $C_{12}H_{23}OSi$ $[M+H]^+$ 211.1512, found 211.1499.



(*S*)-4-((*S*)-4-(trimethylsilyl)but-3-yn-2-yl)dihydrofuran-2(3H)-one (*ent*-**9b**) *ent*-**9b** (1.55 g) was prepared from *ent*-**16** (2.2 g 10.46 mmol) following the procedure described for **9b** in 71% yield. $[\alpha]_D^{21}$ = +65.2 (*c* 0.46, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): δ 4.43 (dd, *J* = 9.2, 7.0 Hz, 1H), 4.22 (dd, *J* = 9.1, 6.8 Hz, 1H), 2.66–2.55 (m, 3H), 2.48–2.39 (m, 1H), 1.20 (d, *J* = 6.4 Hz, 3H), 0.15 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 176.6, 106.3, 87.7, 70.7, 40.2, 32.7, 29.7, 18.8. 0.03. IR (film)ν_{max}/cm^{-1} 2962, 2170, 1796, 1247, 1174, 916, 842, 759. HRMS (ESI): Calcd for $C_{11}H_{19}O_2Si$ $[M+H]^+$ 211.1148, found 211.1144.

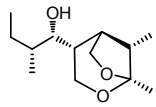


(3*S*,4*R*)-3-((1*S*,2*R*)-1-hydroxy-2-methylbutyl)-4-((*S*)-4-(trimethylsilyl)but-3-yn-2-yl)dihydrofuran-2(3H)-one (*ent*-**17**) *ent*-**17** (635 mg) was prepared from *ent*-**9b** (550 mg 2.6 mmol) following the procedure described for **7b** in 82% yield. $[\alpha]_D^{21.1}$ = +118.8 (*c* 0.7, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): δ 4.37 (t, *J* = 8.9 Hz 1H), 4.21 (t, *J* = 8.9 Hz, 1H), 3.56 (t, *J* = 6.0 Hz 1H), 2.86 (dd, *J* = 9.5, 6.2 Hz, 1H), 2.82 (dd, *J* = 7.1, 4.3 Hz, 1H), 2.48 (m, 1H), 1.72–1.66 (m, 1H), 1.58–1.51 (m, 1H), 1.37–1.30 (m, 1H), 1.18 (d, *J* = 7.2 Hz, 3H), 0.99 (d, *J* = 6.7 Hz, 3H), 0.93 (t, *J* = 7.5 Hz, 3H), 0.15 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 179.0, 105.6, 89.3, 68.2, 45.5, 42.7, 37.5, 28.2, 26.4, 18.5, 13.4, 11.3. 0.02. IR (film)ν_{max}/cm^{-1} 3470, 2965, 2170, 1740, 1250, 1196, 843, 759. HRMS (ESI): Calcd for $C_{16}H_{29}O_3Si$ $[M+H]^+$ 297.1880, found 297.1872.



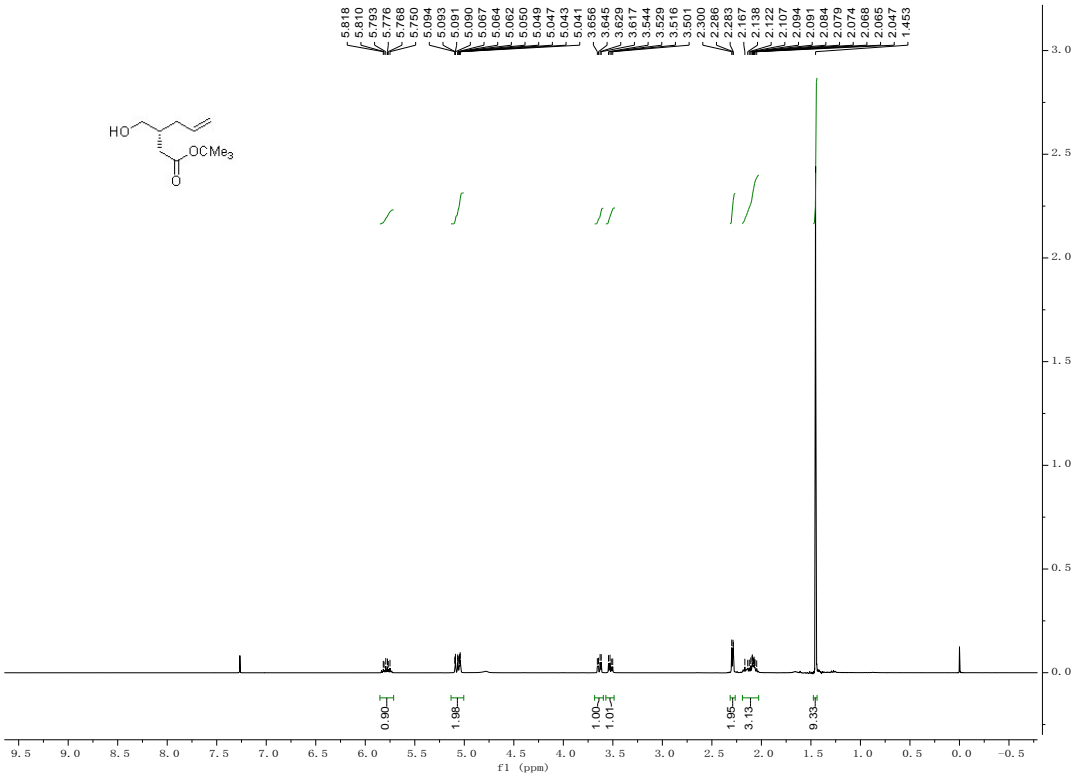
(2*R*,3*R*,4*S*,5*R*)-2-((*S*)-but-3-yn-2-yl)-3-(hydroxymethyl)-5-methylheptane-1,4-diol (*ent*-**18**) *ent*-**18** (250 mg) was prepared from *ent*-**17** (600 mg 2.0 mmol) following the procedure described for **6** in 60% yield over two steps. $[\alpha]_D^{21.1}$ = +16.6 (*c* 0.65, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.04–3.93 (m, 2H), 3.84 (td, *J* = 12.6, 11.8, 2.7 Hz, 2H), 3.63 (t, *J* = 5.6 Hz, 1H), 2.77 (m, 1H), 2.09 (d, *J* = 2.5 Hz, 1H), 1.88–1.84 (m, 1H), 1.81–1.76 (m, 1H), 1.67–1.57 (m, 1H), 1.49–1.39 (m, 1H), 1.29 (d, *J* = 7.0 Hz, 3H), 1.24–1.15 (m, 1H), 0.97 (d, *J* = 6.7 Hz, 3H), 0.92 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 86.8, 75.8, 70.0, 60.7, 59.8, 44.5, 41.3, 37.3, 26.0, 25.7, 19.4, 13.9, 11.1. IR (film)ν_{max}/cm^{-1} 3305, 2964, 2933, 2109, 1461, 1034, 635. HRMS (ESI): Calcd for $C_{13}H_{24}O_3Na$ $[M+Na]^+$ 251.1617, found 251.1609.

(1*S*,2*R*)-1-((1*R*,4*R*,5*S*,8*S*)-1,8-dimethyl-2,7-dioxabicyclo[3.2.1]octan-4-yl)-2-methylbutan-1-ol (*ent*-**19**) *ent*-**19**

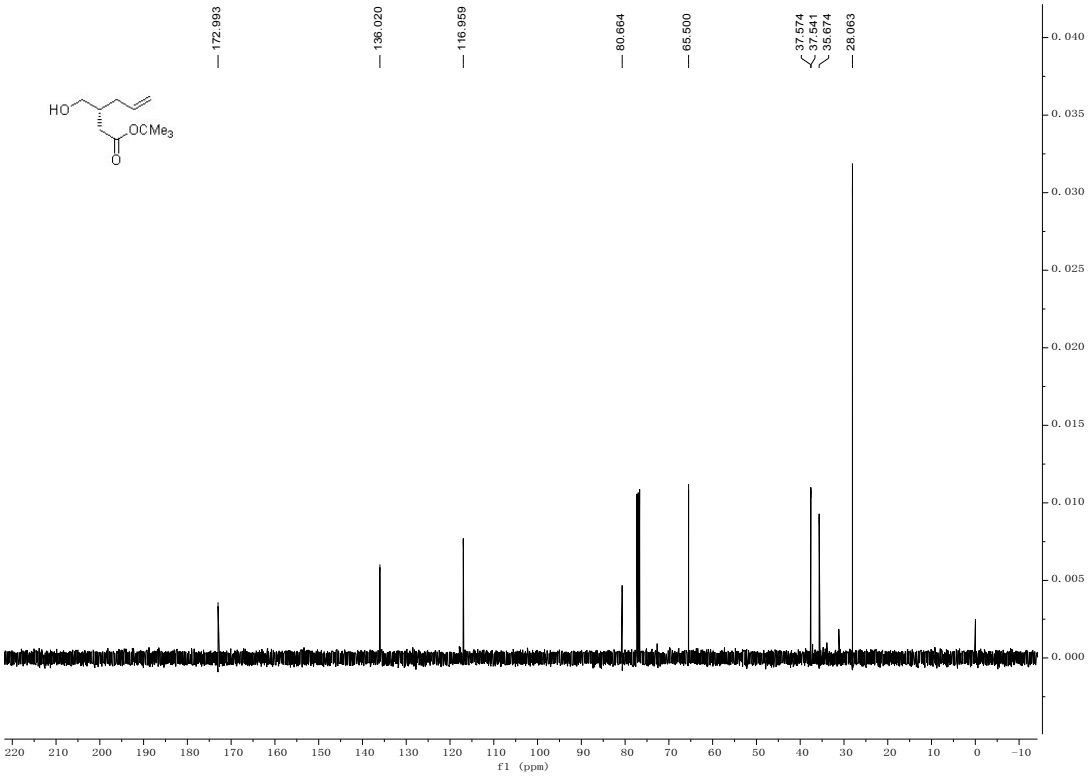


(150 mg) was prepared from *ent*-**18** (220 mg 0.96 mmol) following the procedure described for **5** in 70% yield. $[\alpha]_D^{21.1}$ = +37.5 (*c* 0.16, acetone). ^{1}H NMR (400 MHz, Acetone-d_6): δ 3.97 (d, *J* = 8.5 Hz, 1H), 3.91 (ddd, *J* = 11.5, 5.9, 1.1 Hz, 1H), 3.84 (ddt, *J* = 8.5, 4.4, 0.8 Hz, 1H), 3.60 (t, *J* = 11.3 Hz, 1H), 3.45–3.40 (m, 1H), 2.13 (d, *J* = 4.1 Hz, 1H), 2.04–1.95 (m, 1H), 1.83 (q, *J* = 7.0 Hz, 1H), 1.52–1.38 (m, 2H), 1.33–1.28 (m, 1H), 1.25 (s, 3H), 0.93–0.88 (m, 6H), 0.85 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, Acetone-d_6): δ 108.0, 73.7, 67.9, 64.9, 48.1, 44.0, 43.4, 37.2, 37.2, 27.5, 20.3, 14.6, 12.5, 12.2. IR (film)ν_{max}/cm^{-1} 3463, 2963, 2936, 2880, 1460, 1384, 1121, 1043, 842. HRMS (ESI): Calcd for $C_{13}H_{25}O_3$ $[M+H]^+$ 229.1798, found 229.1792.

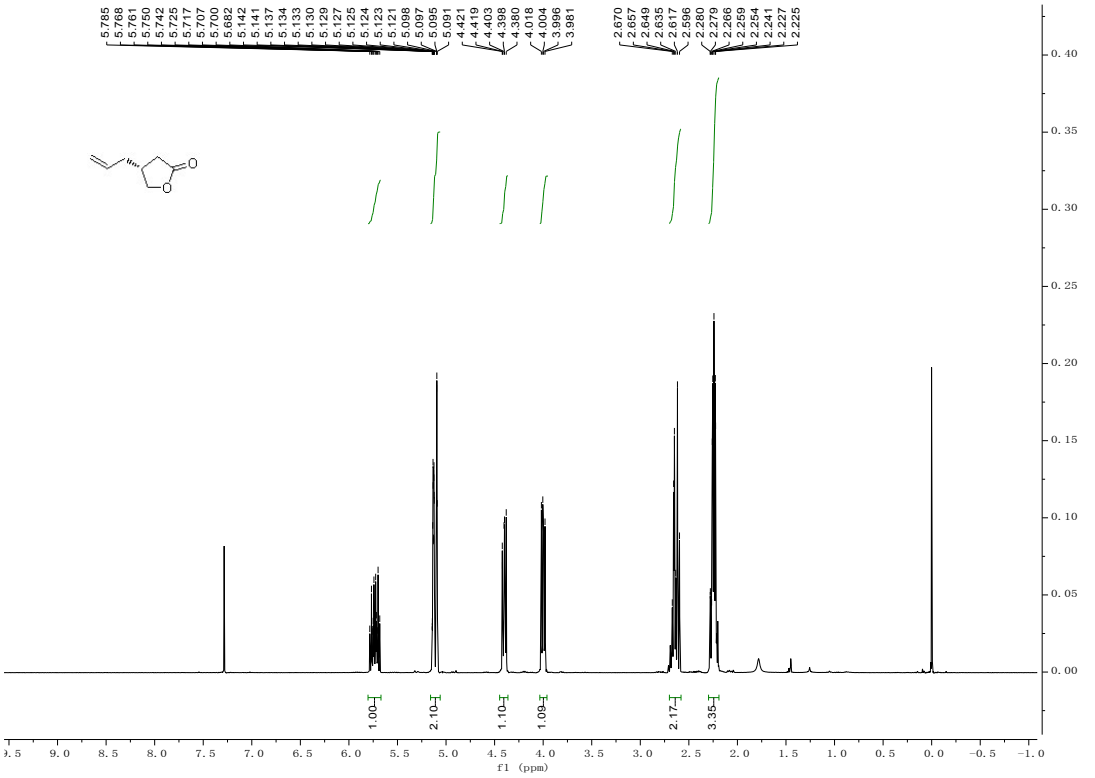
^{1}H and ^{13}C NMR Spectra of Reported Compounds



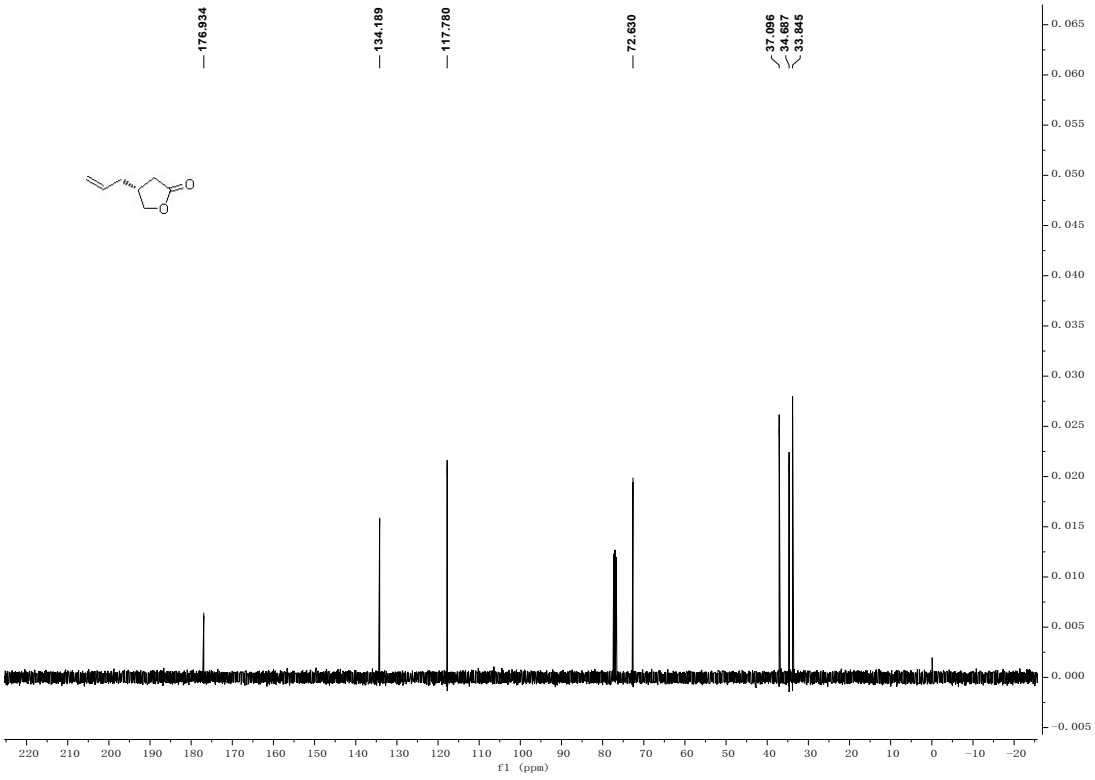
^{1}H NMR spectra of compound **13**



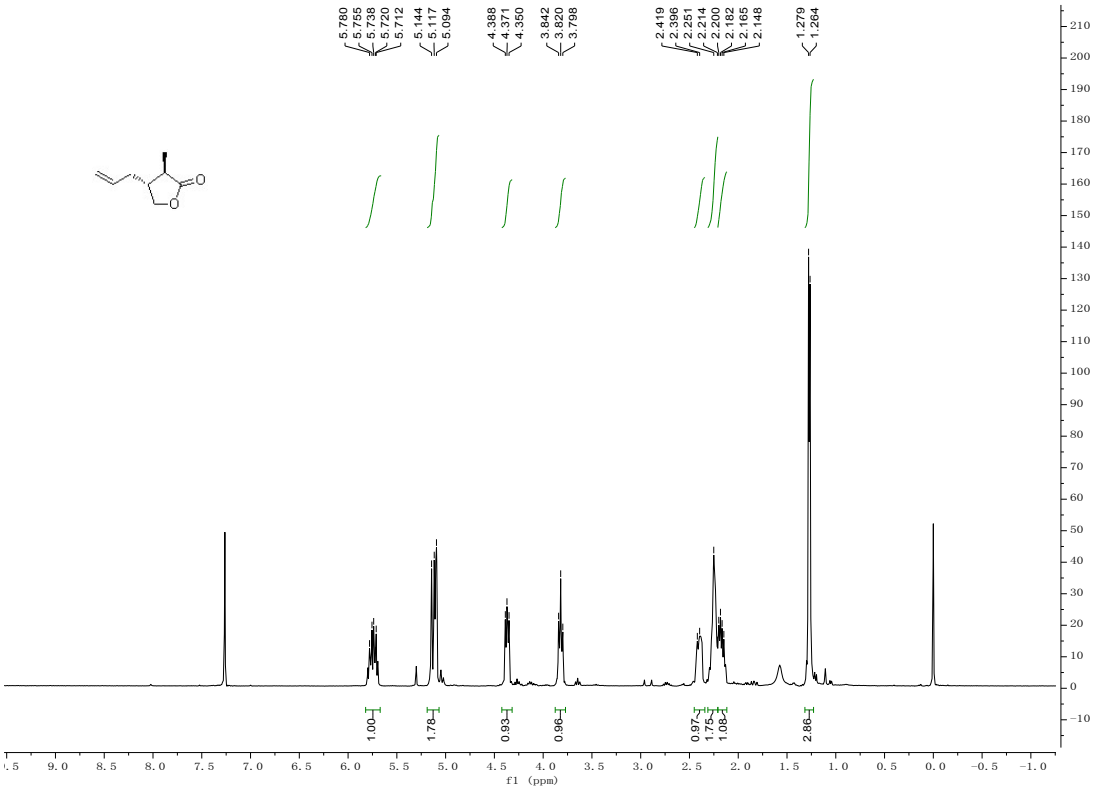
^{13}C NMR spectra of compound **13**



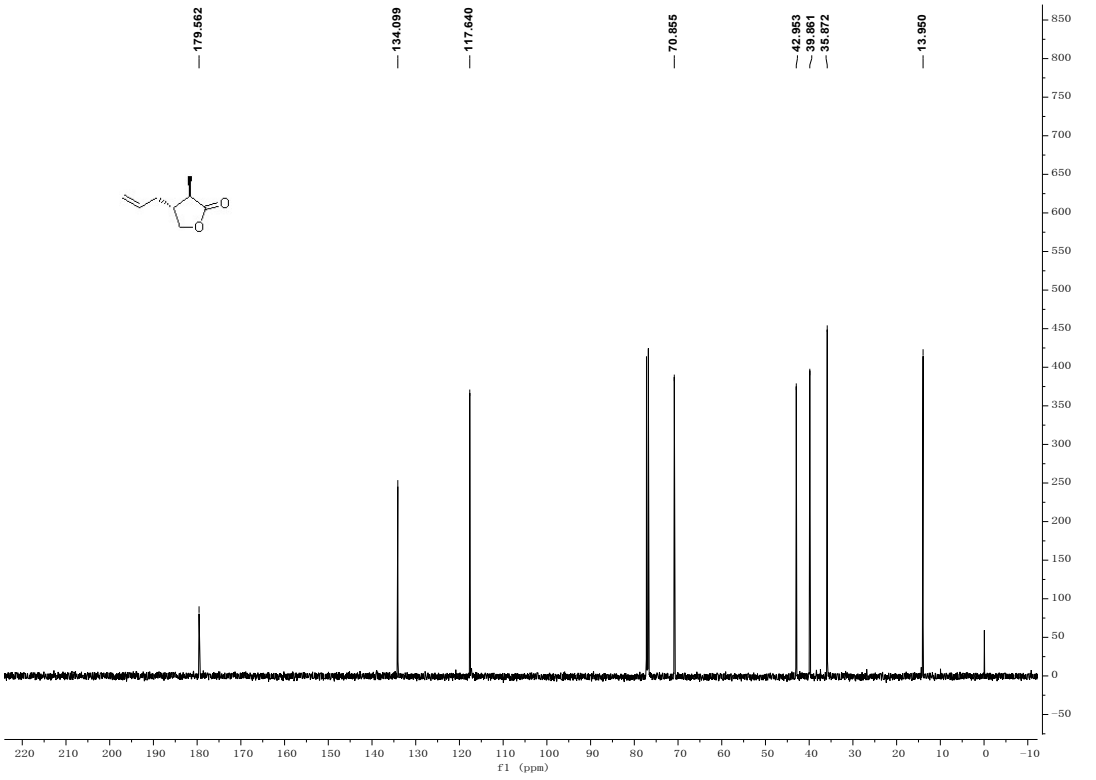
^{1}H NMR spectra of compound **14**



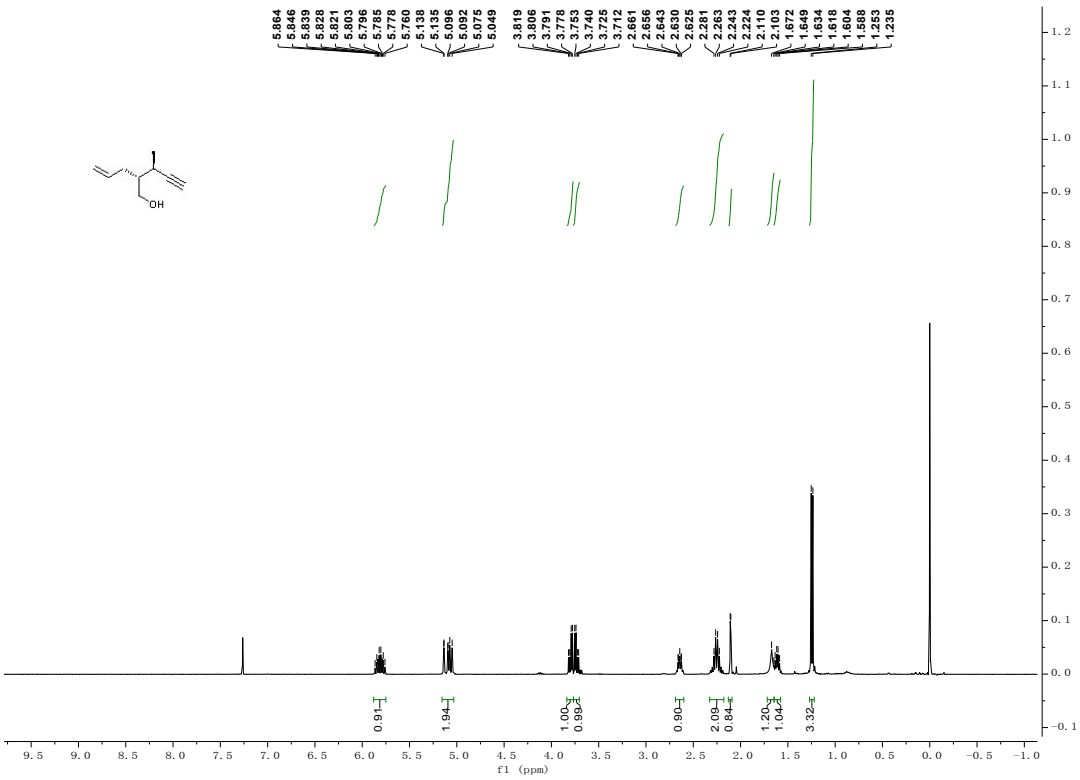
^{13}C NMR spectra of compound **14**



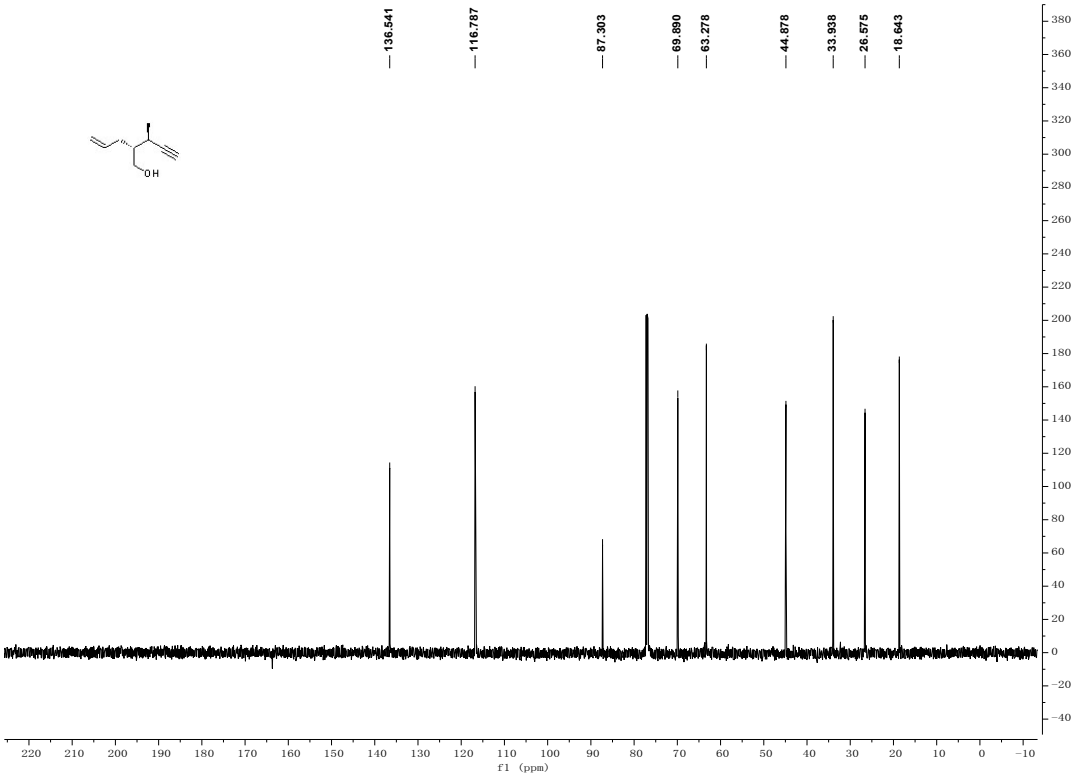
^{1}H NMR spectra of compound **10**



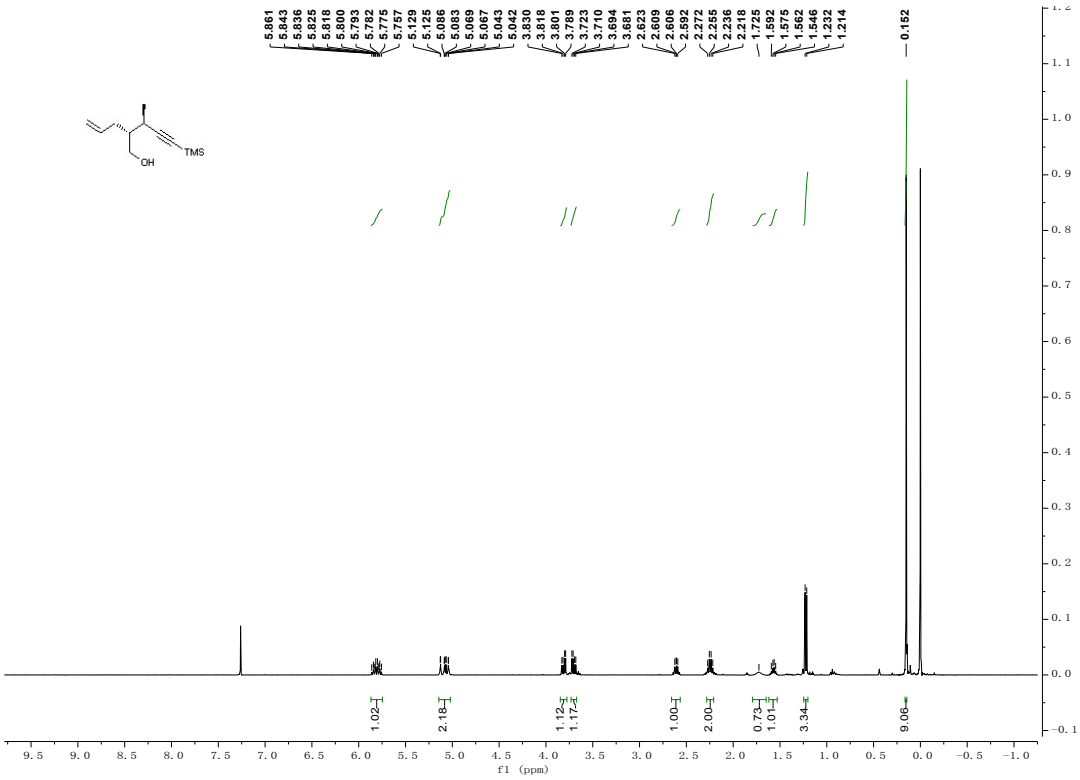
^{13}C NMR spectra of compound **10**



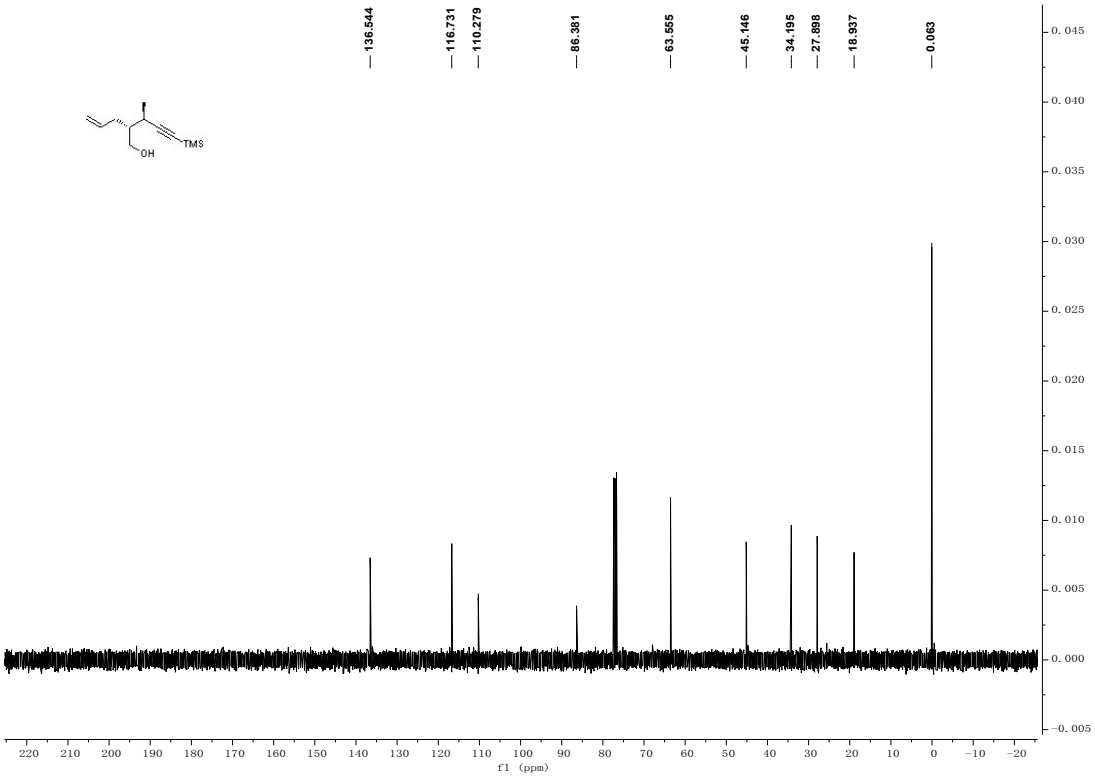
^{1}H NMR spectra of compound **15**



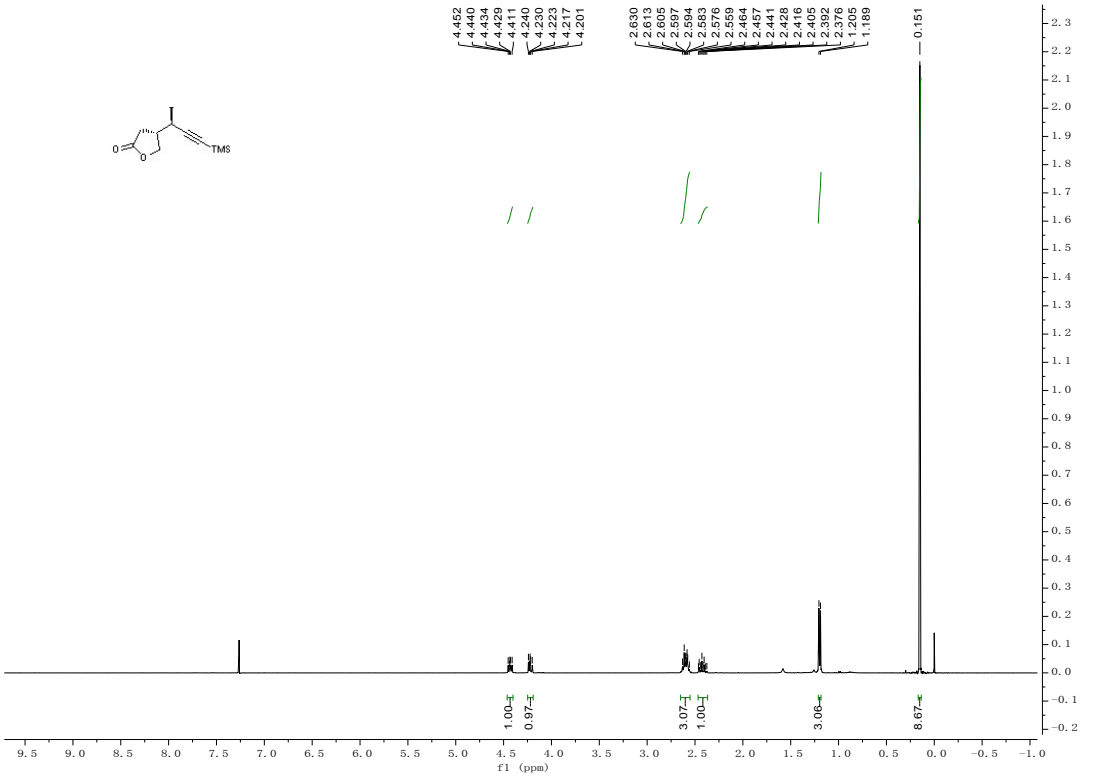
^{13}C NMR spectra of compound **15**



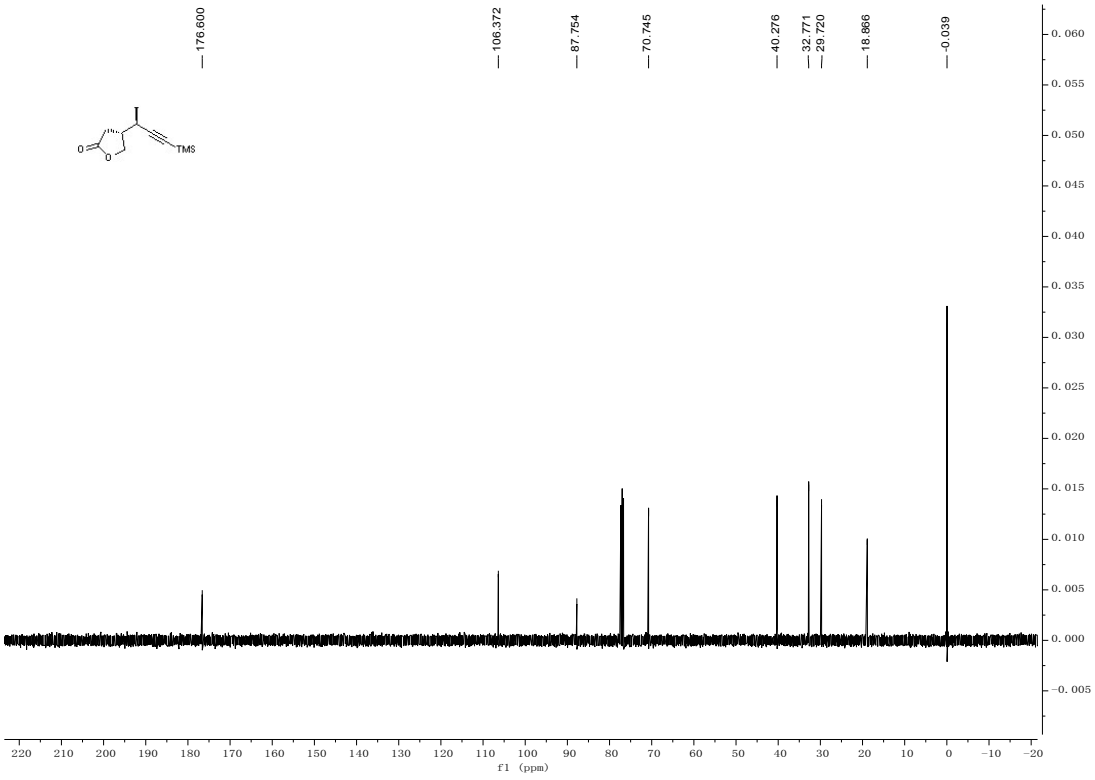
^{1}H NMR spectra of compound **16**



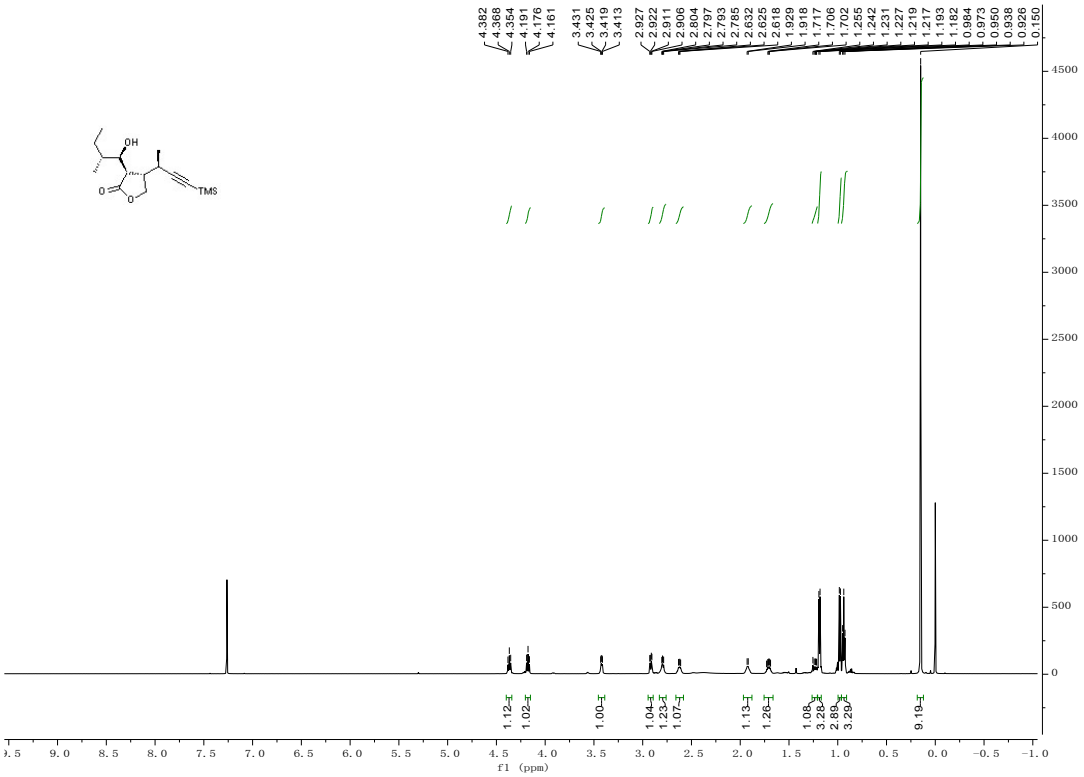
^{13}C NMR spectra of compound **16**



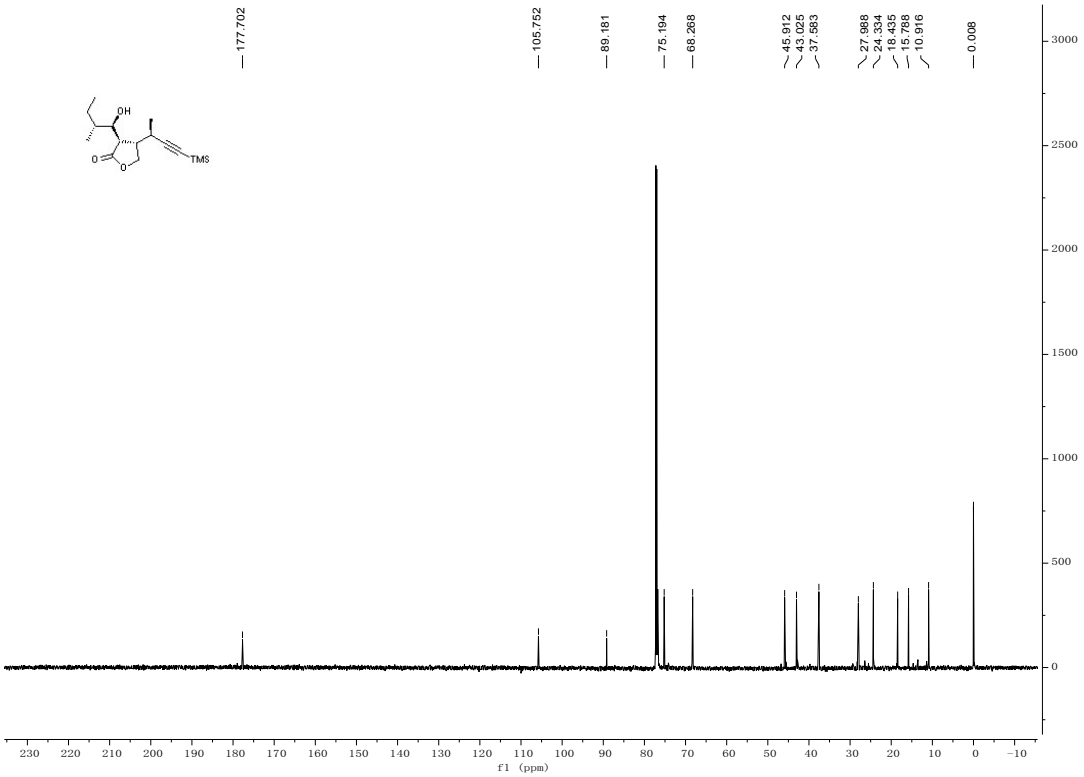
^{1}H NMR spectra of compound **9b**



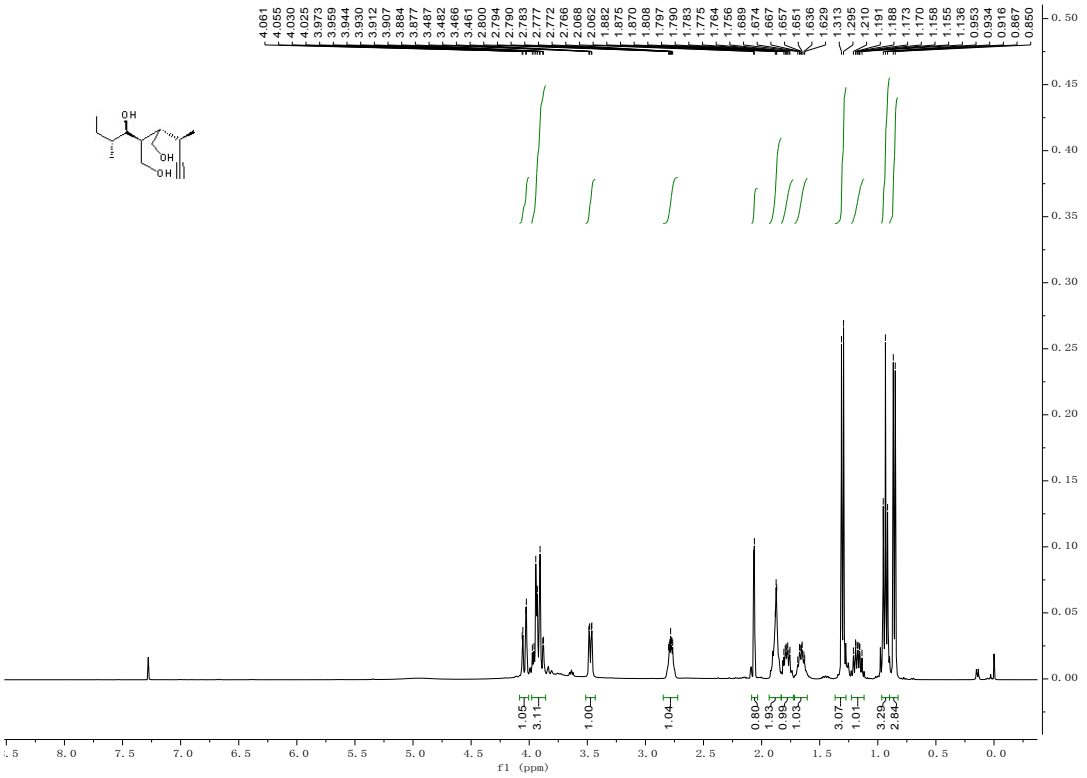
^{13}C NMR spectra of compound **9b**



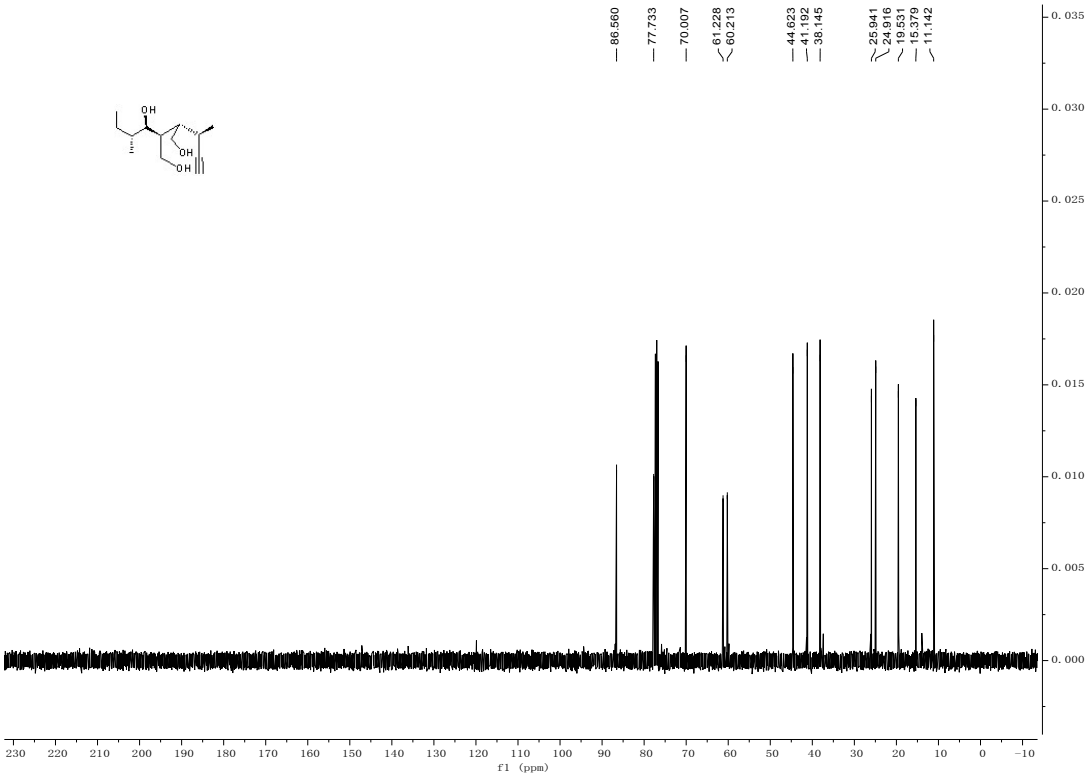
^{1}H NMR spectra of compound **7b**



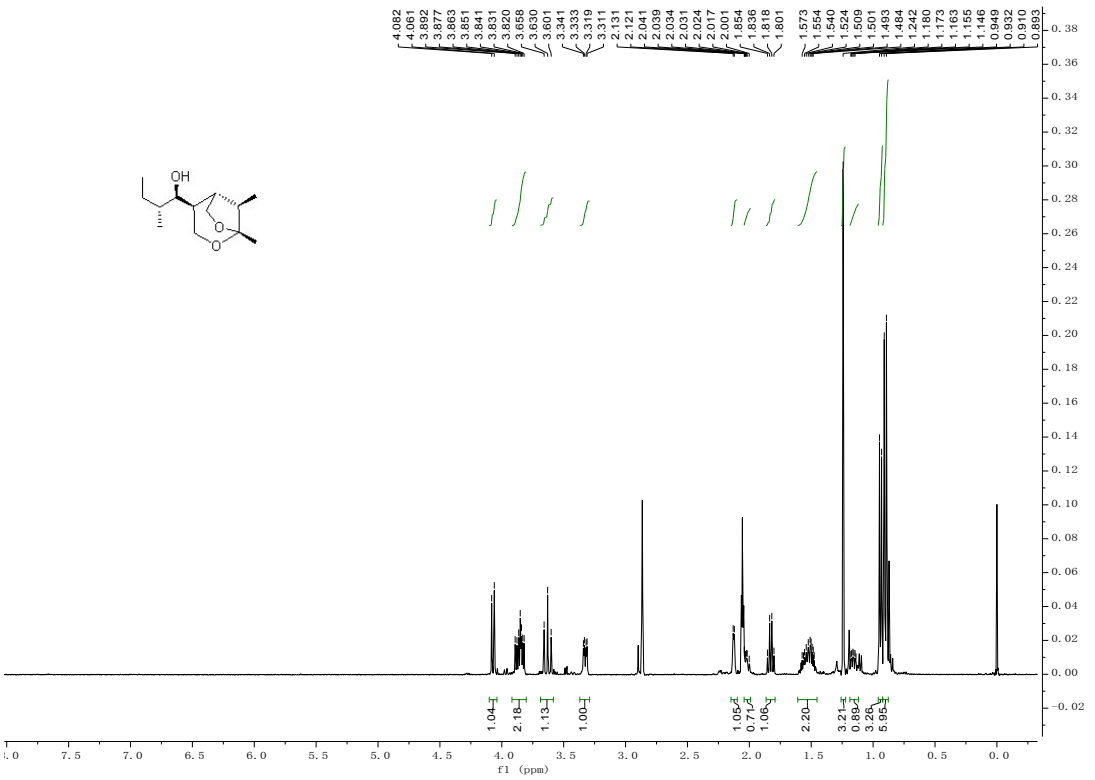
^{13}C NMR spectra of compound **7b**



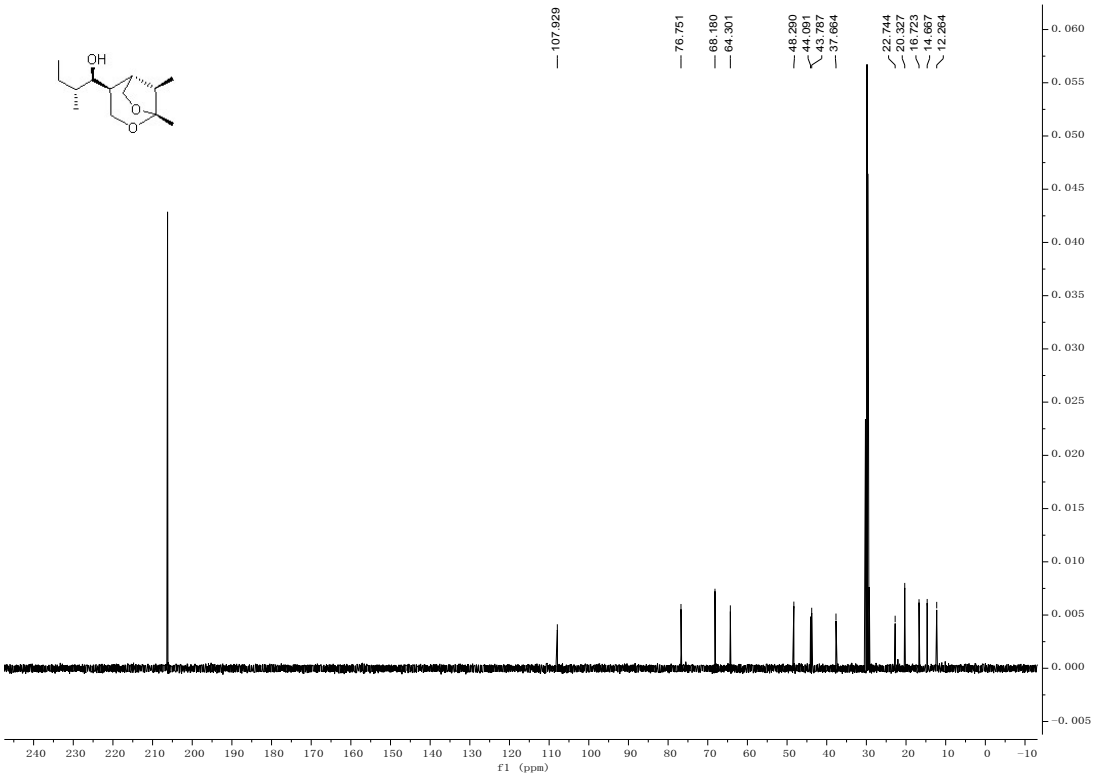
^{1}H NMR spectra of compound **6**



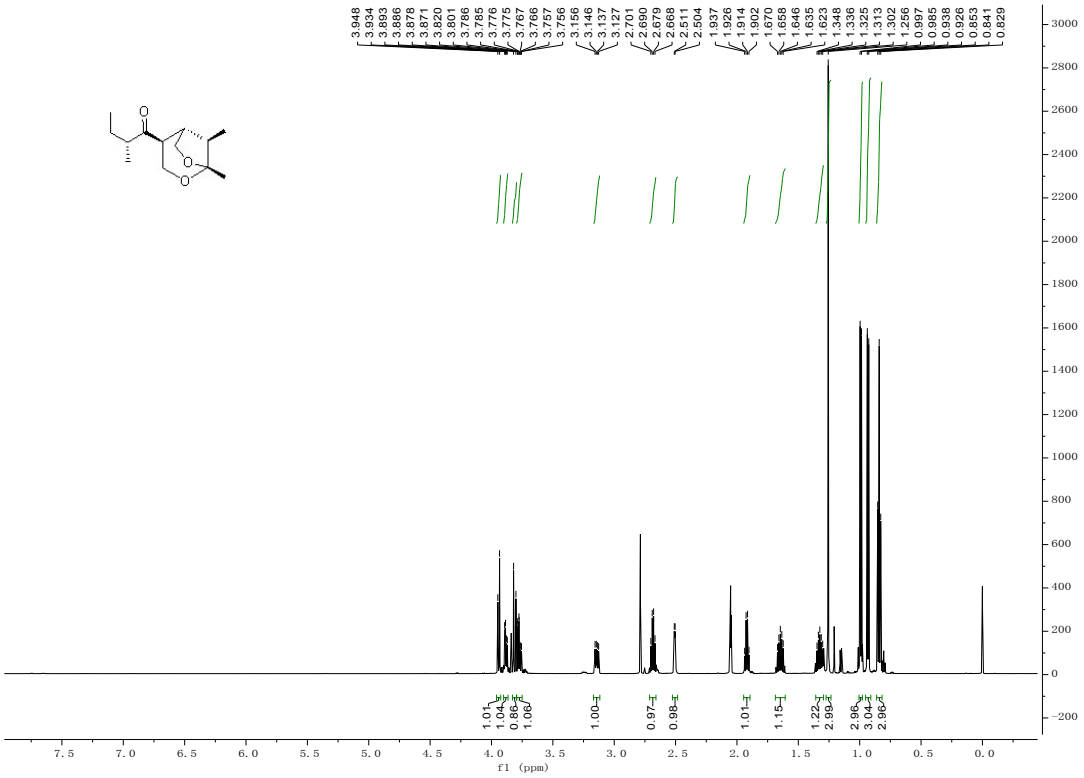
^{13}C NMR spectra of compound **6**



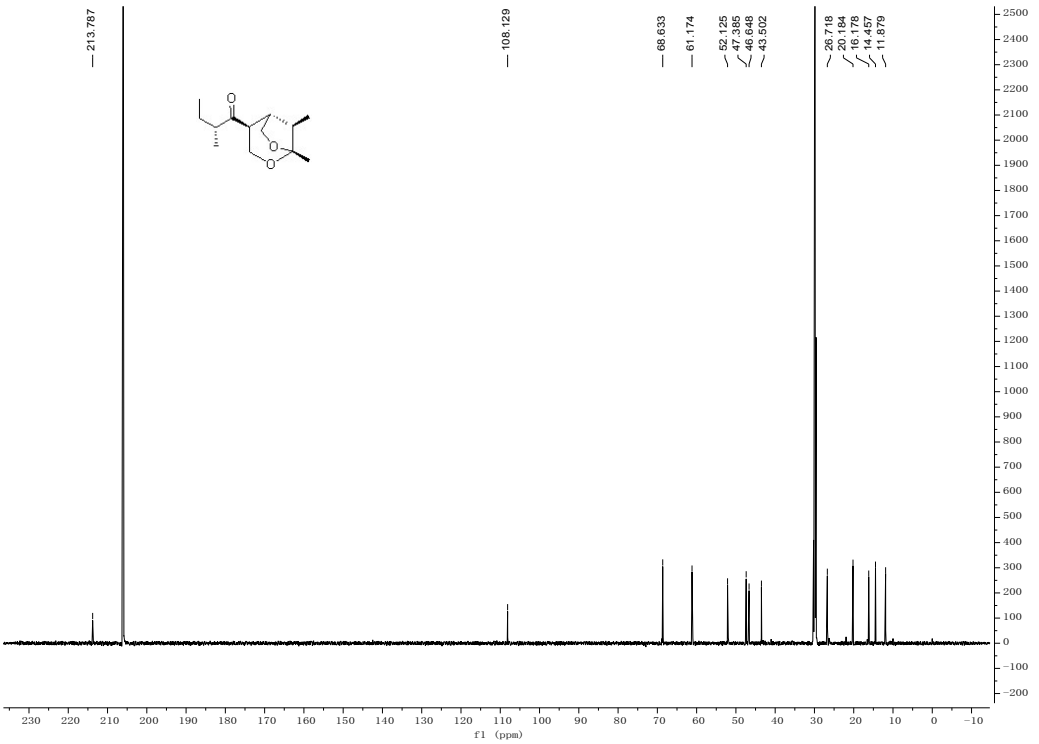
^{1}H NMR spectra of compound **5**



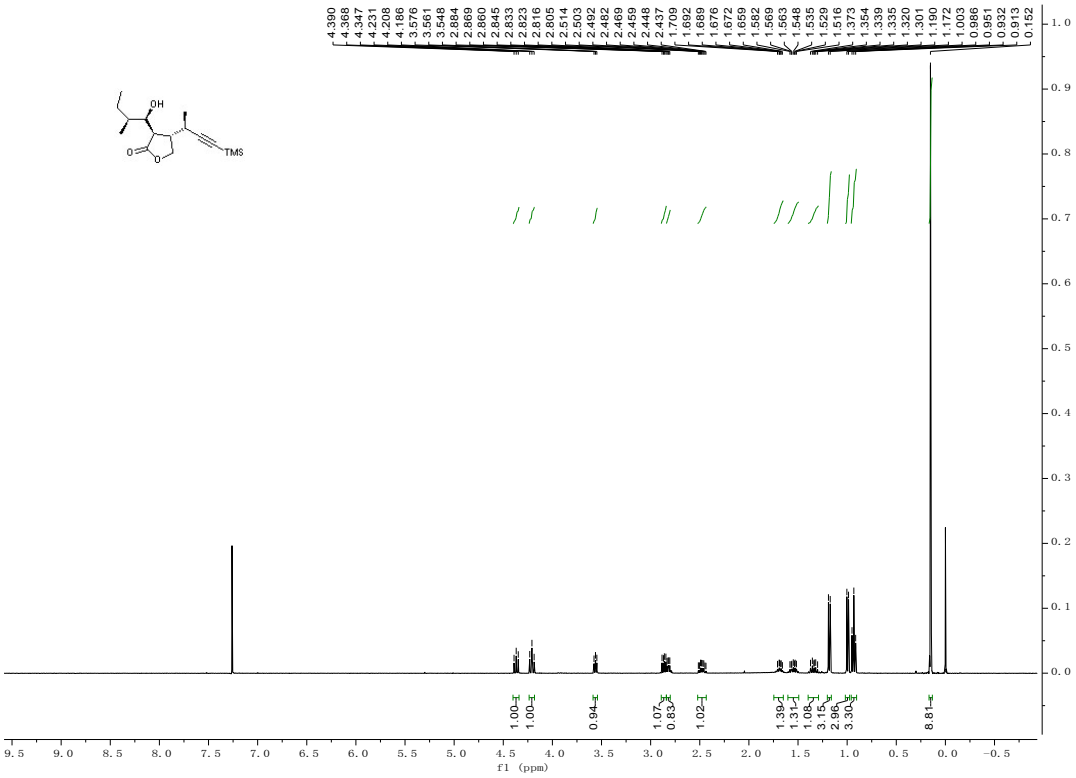
^{13}C NMR spectra of compound **5**



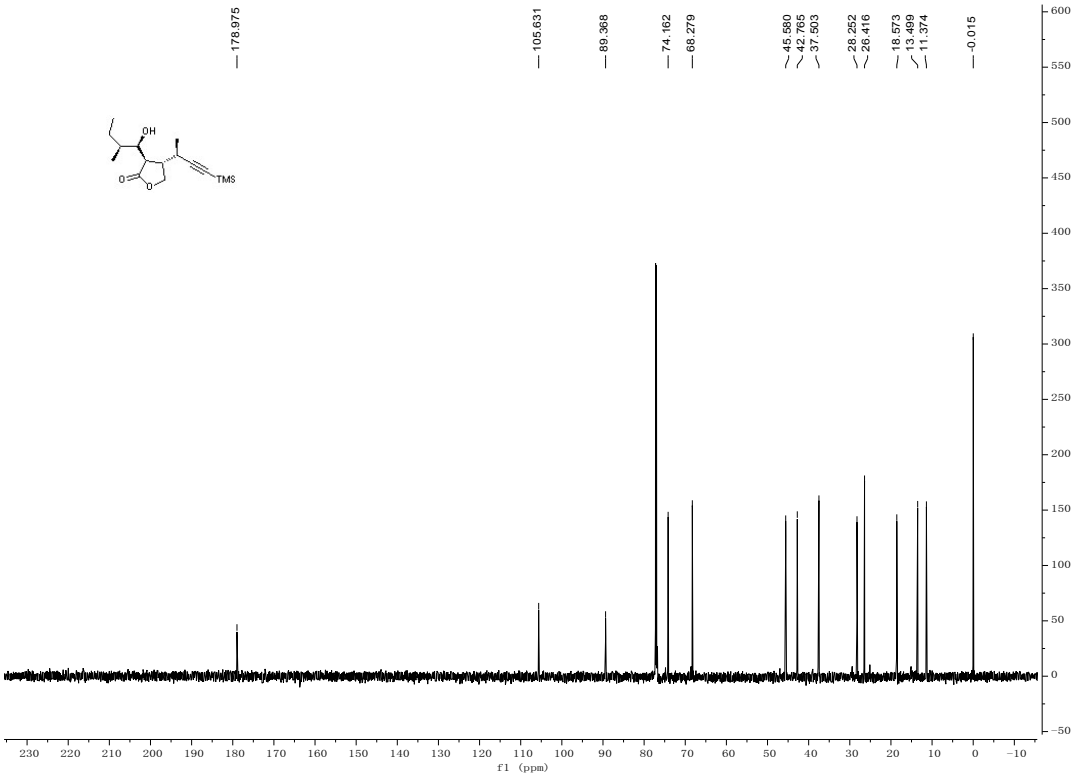
^{1}H NMR spectra of compound **1a**



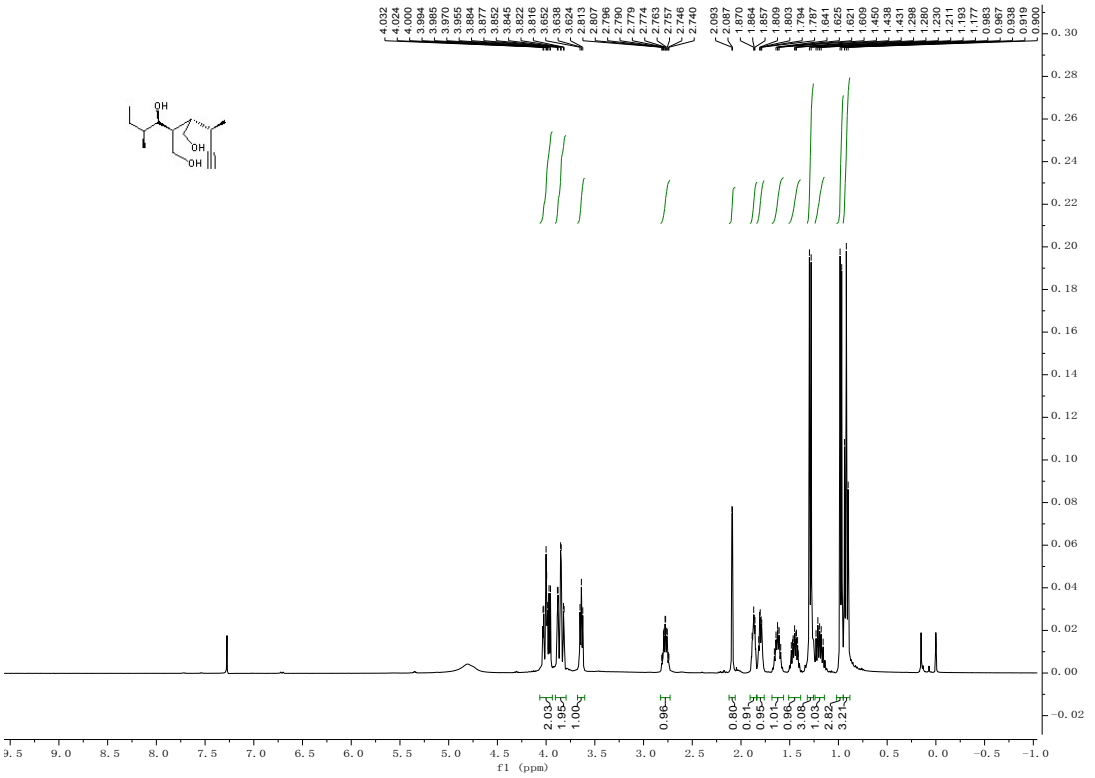
^{13}C NMR spectra of compound **1a**



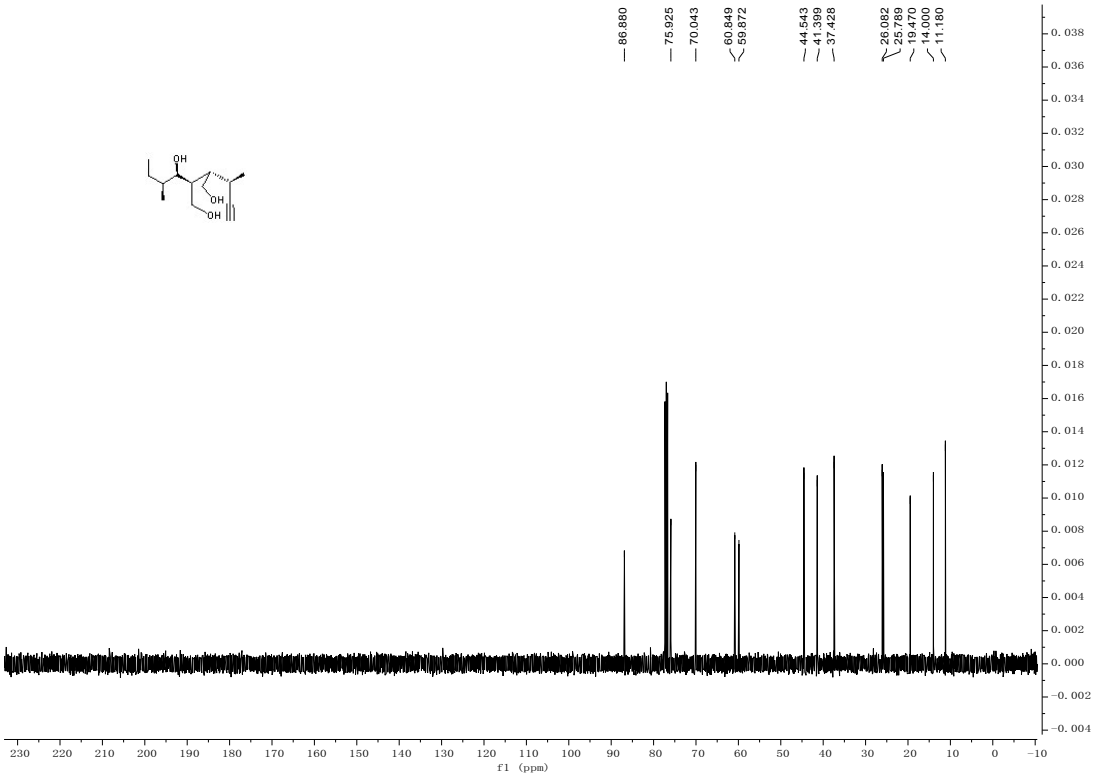
^{1}H NMR spectra of compound **17**



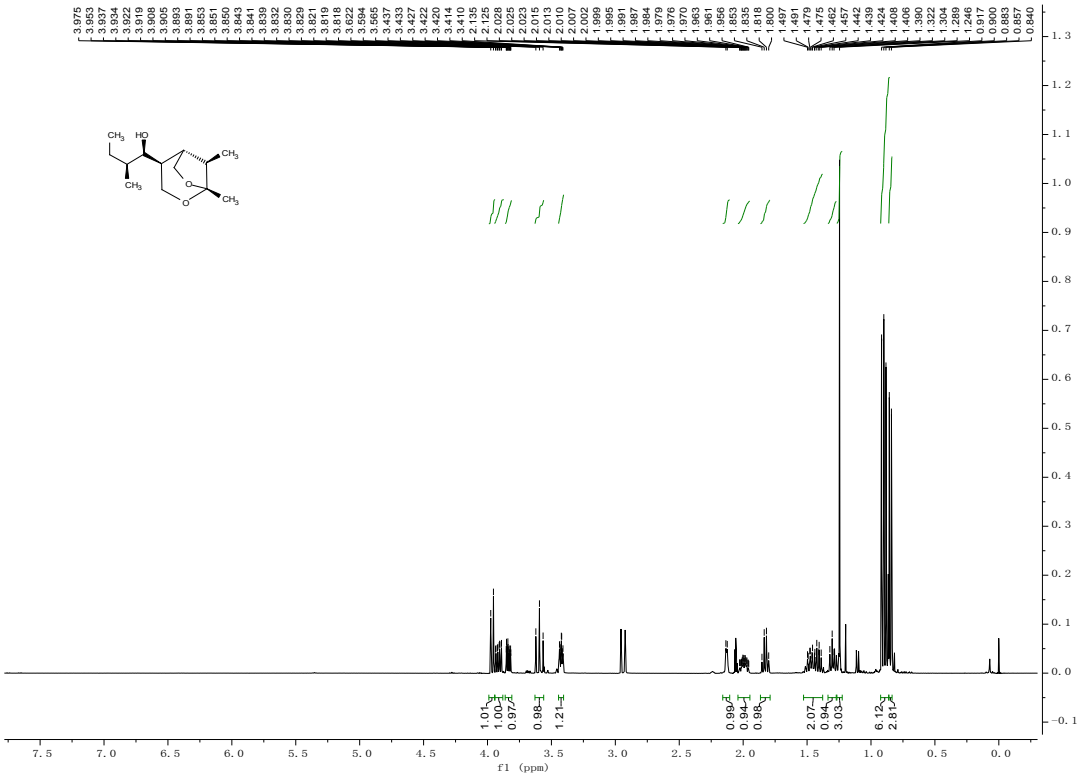
^{13}C NMR spectra of compound **17**



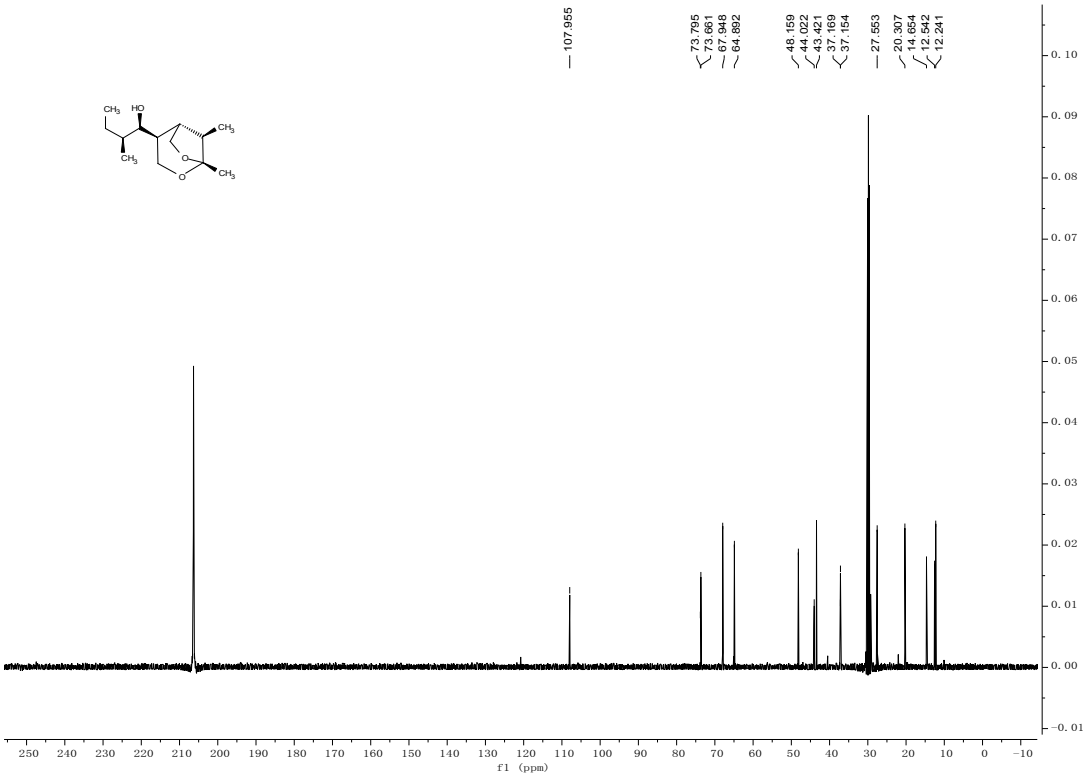
^{1}H NMR spectra of compound **18**



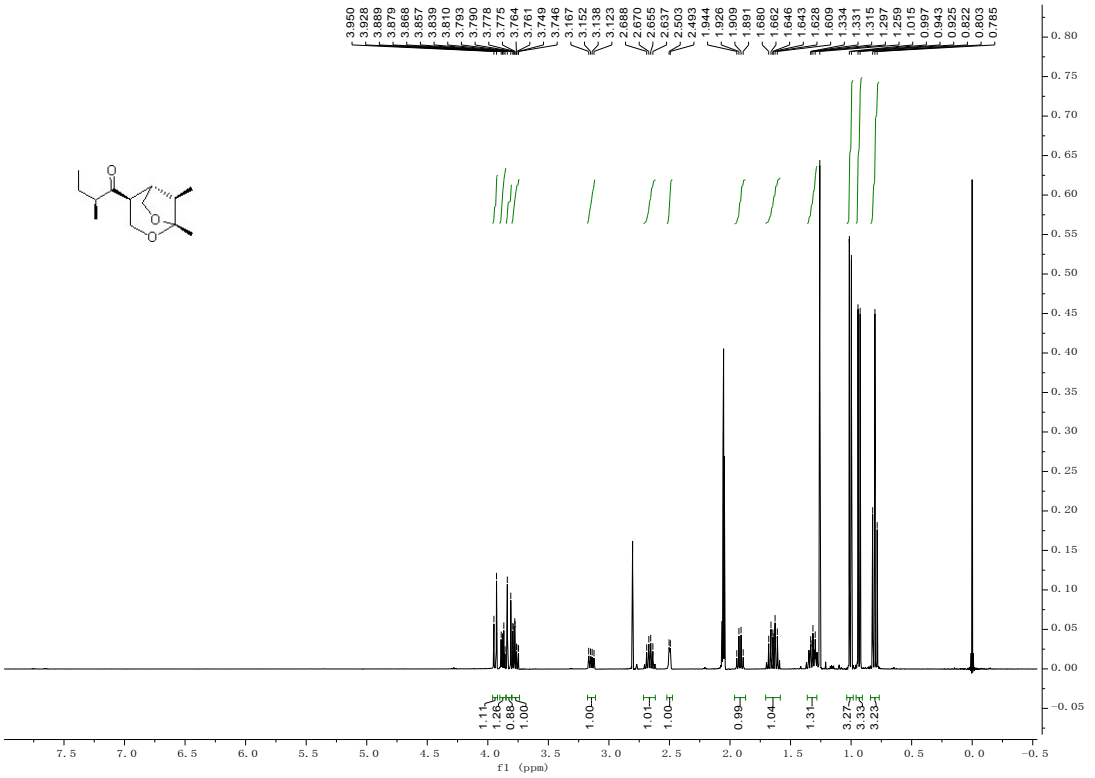
^{13}C NMR spectra of compound **18**



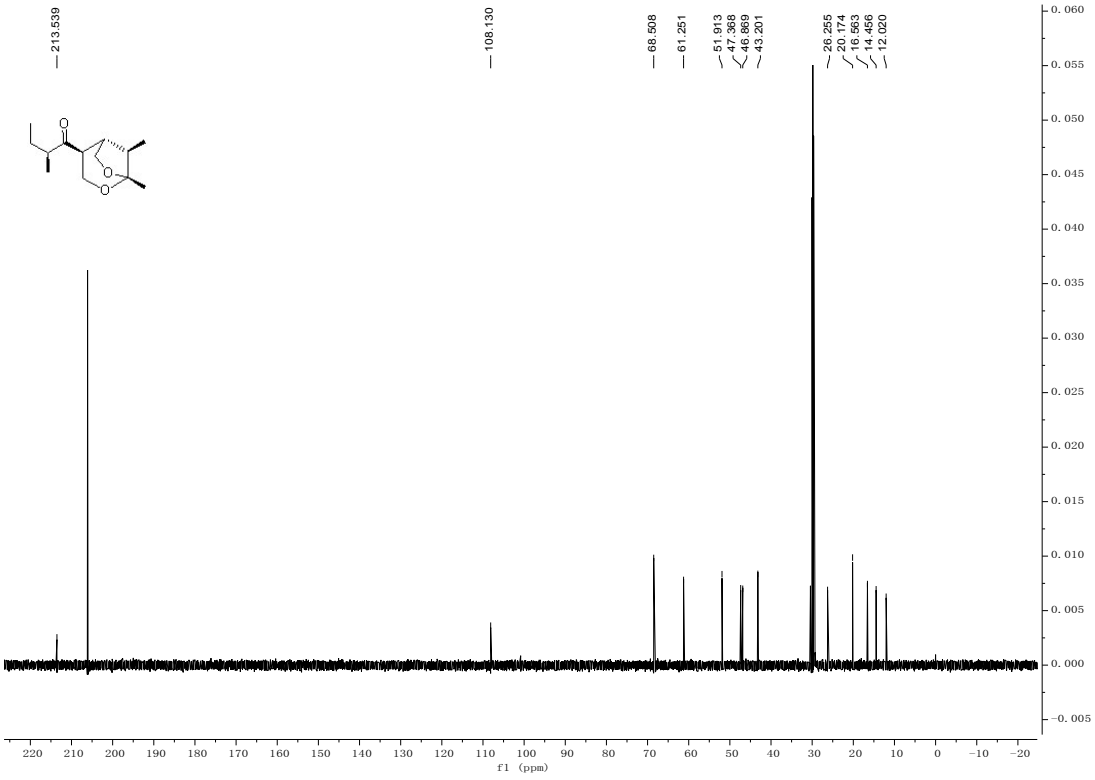
^{1}H NMR spectra of compound **19**



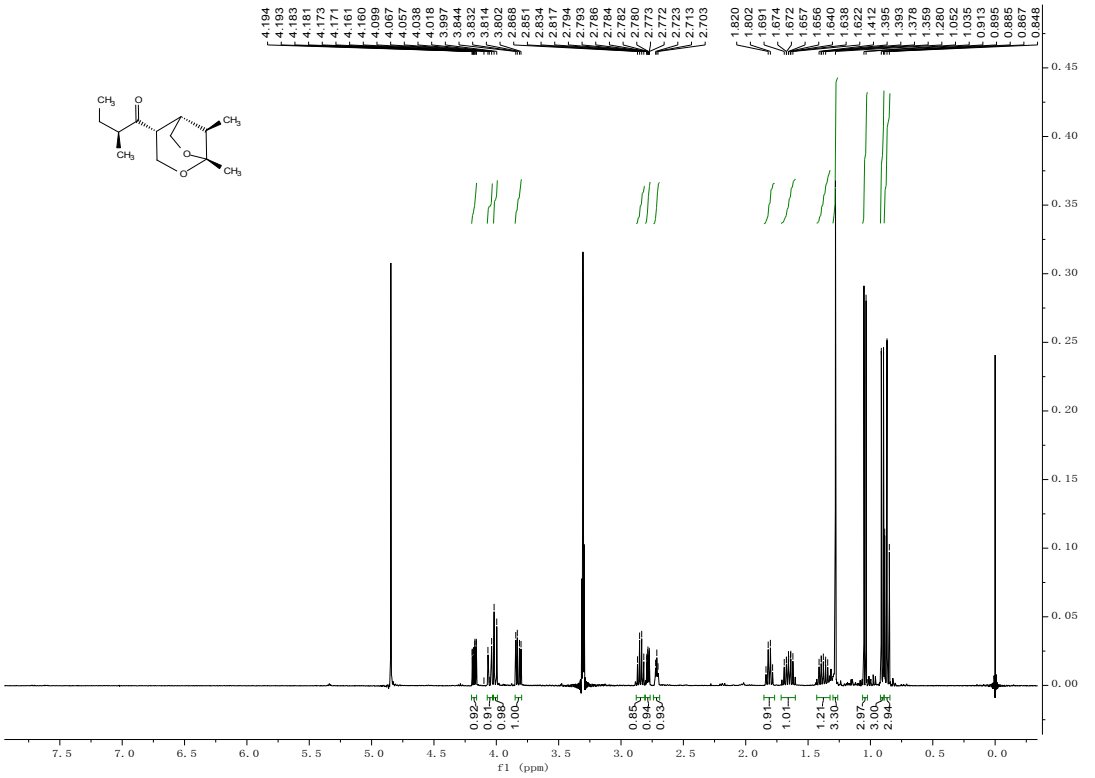
^{13}C NMR spectra of compound **19**



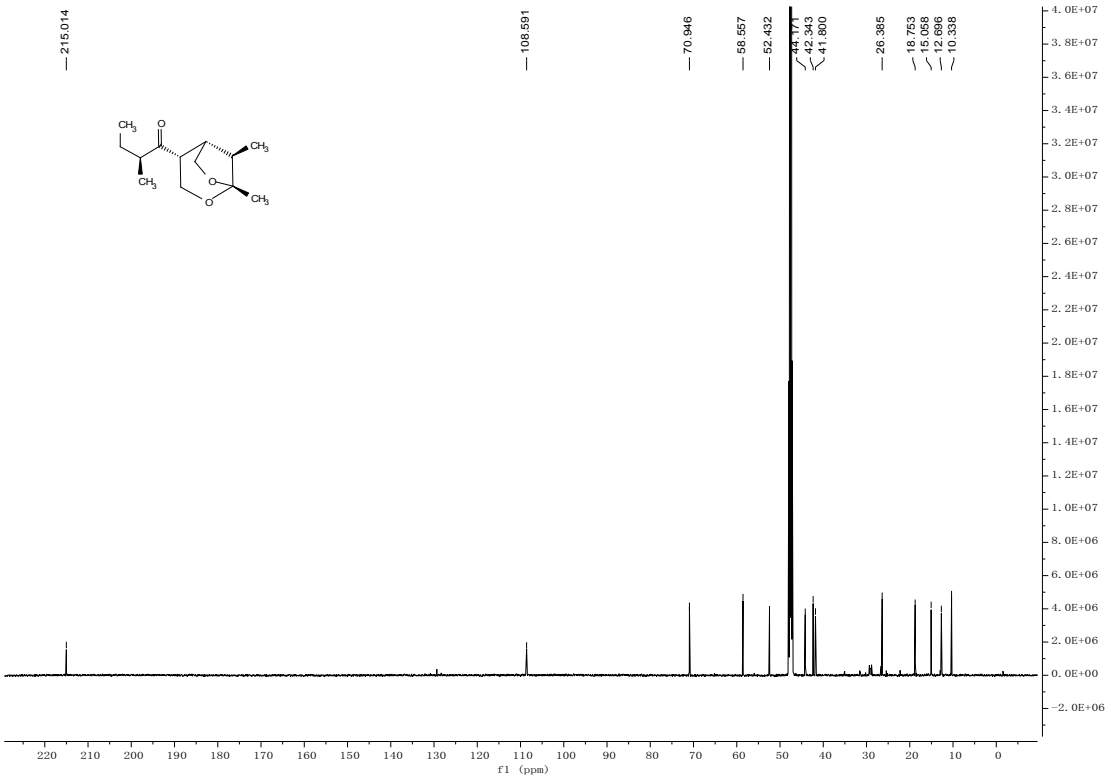
^{1}H NMR spectra of compound **1b**



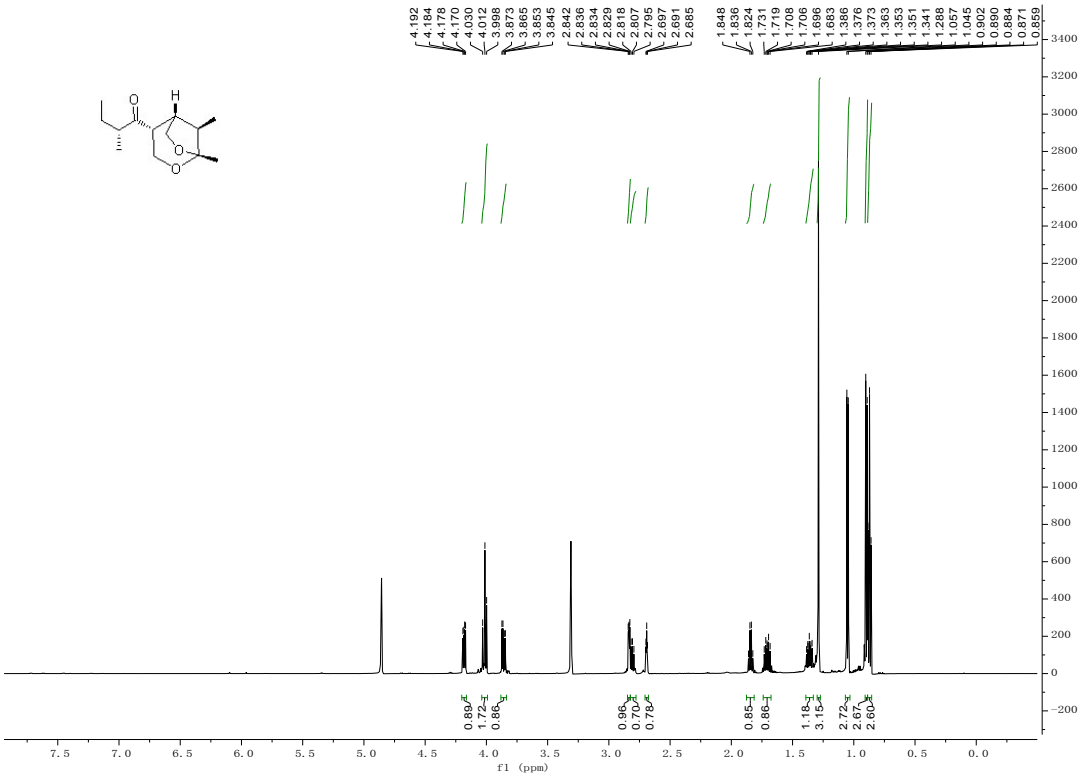
^{13}C NMR spectra of compound **1b**



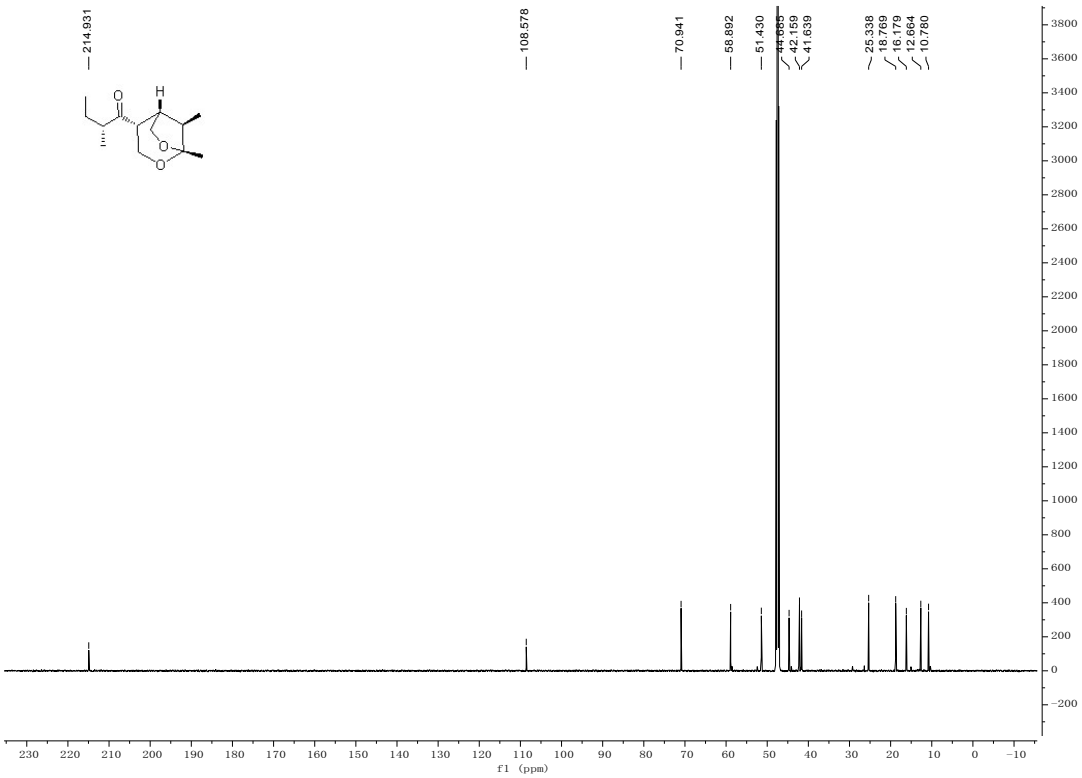
^{1}H NMR spectra of compound **3**



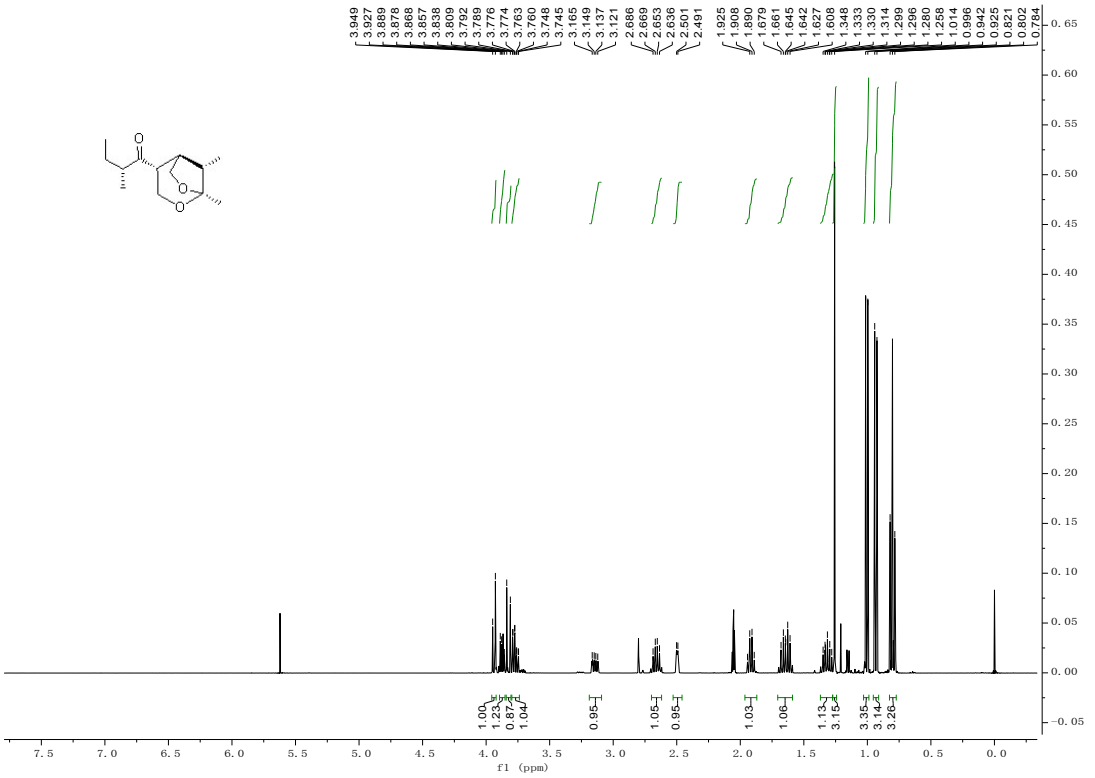
^{13}C NMR spectra of compound **3**



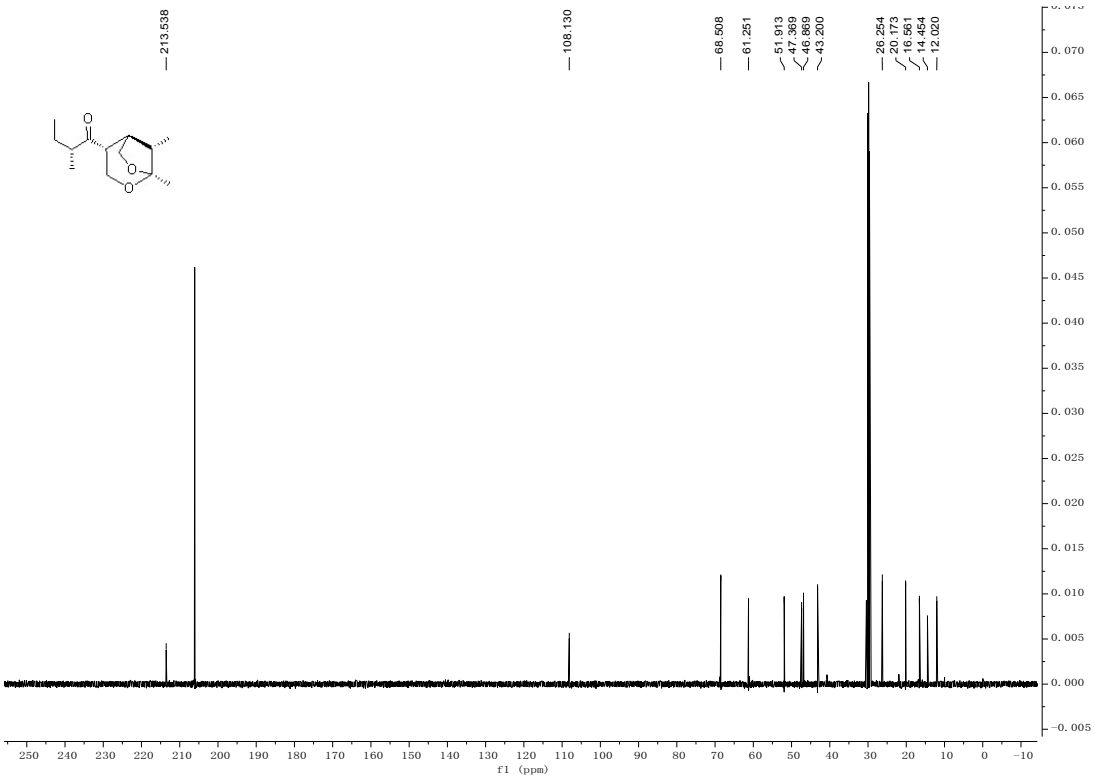
^{1}H NMR spectra of compound **20**



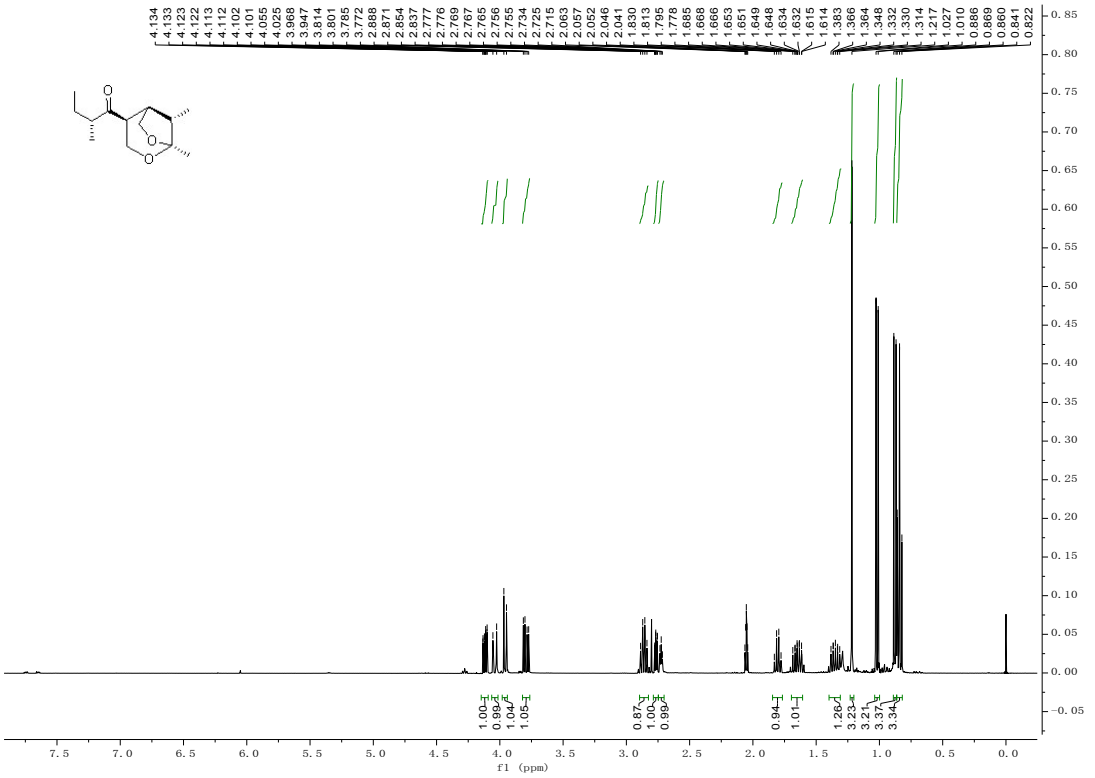
^{13}C NMR spectra of compound **20**



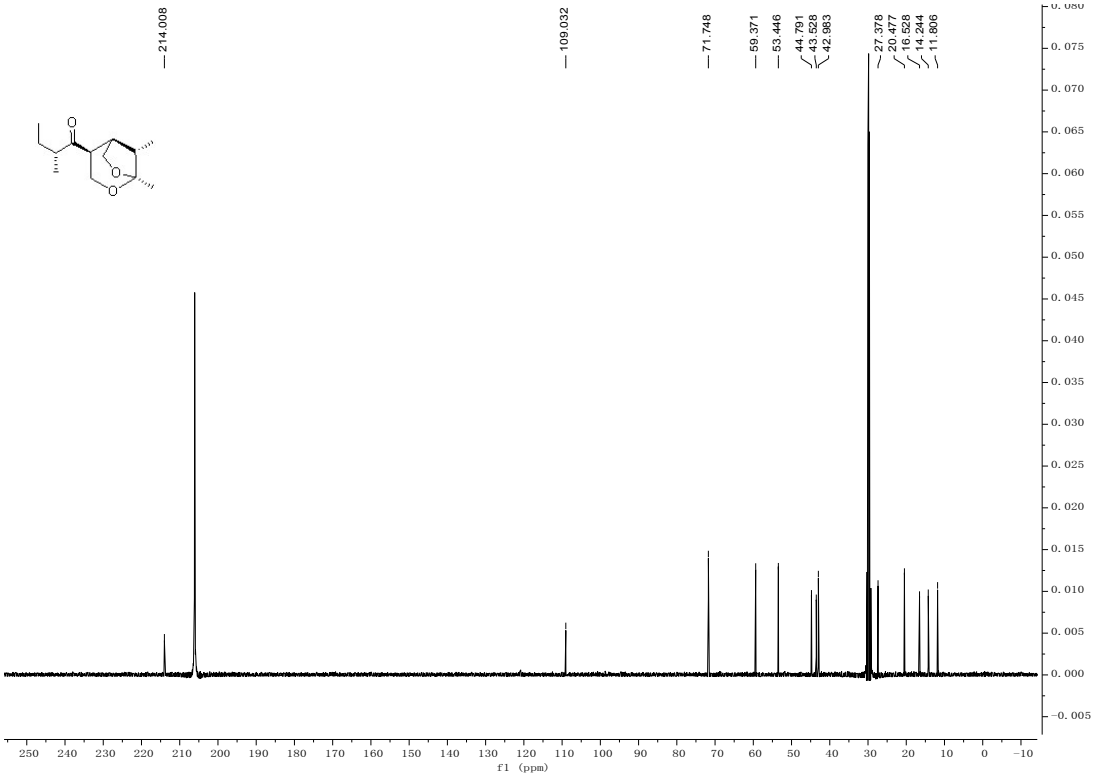
^{1}H NMR spectra of compound **1c**



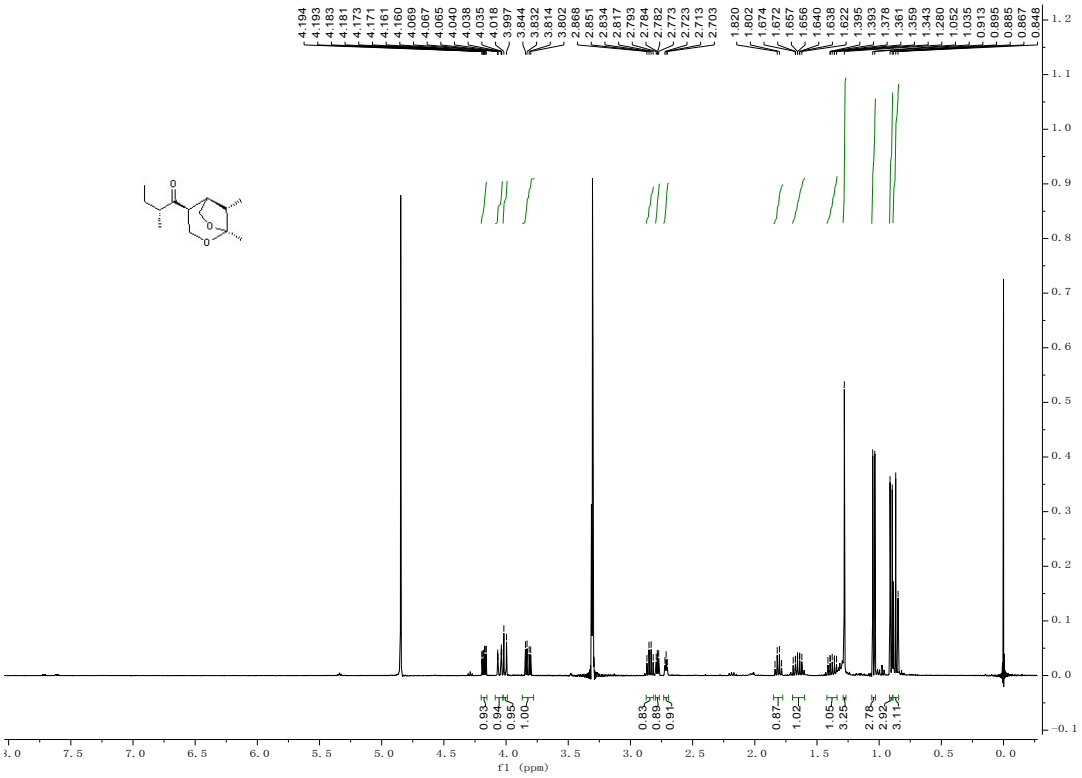
^{13}C NMR spectra of compound **1c**



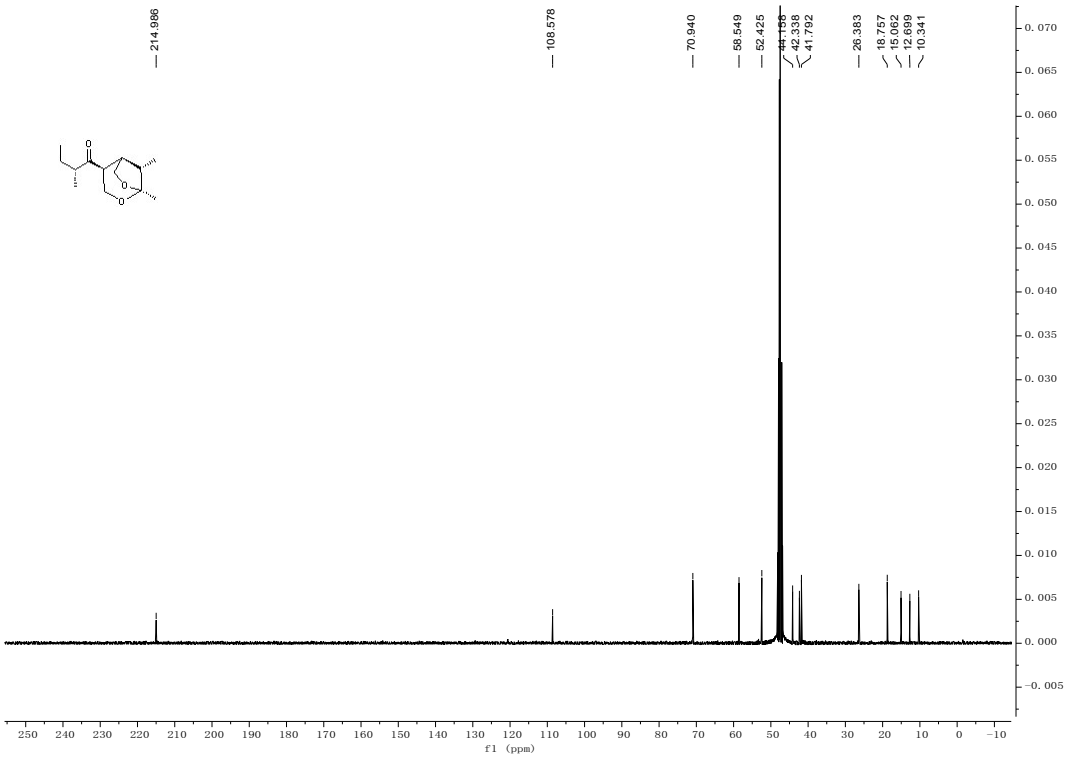
^{1}H NMR spectra of compound **2** in $(CD_3)CO$



^{13}C NMR spectra of compound **2** in $(CD_3)CO$

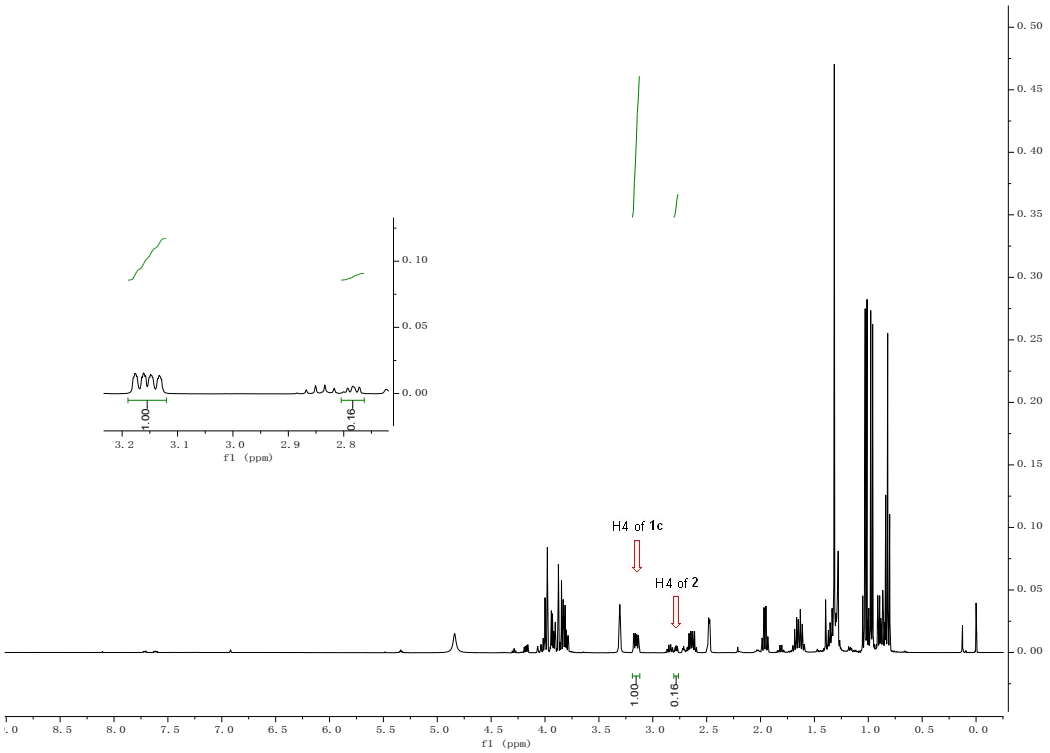


^{1}H NMR spectra of compound **2** in CD_3OD

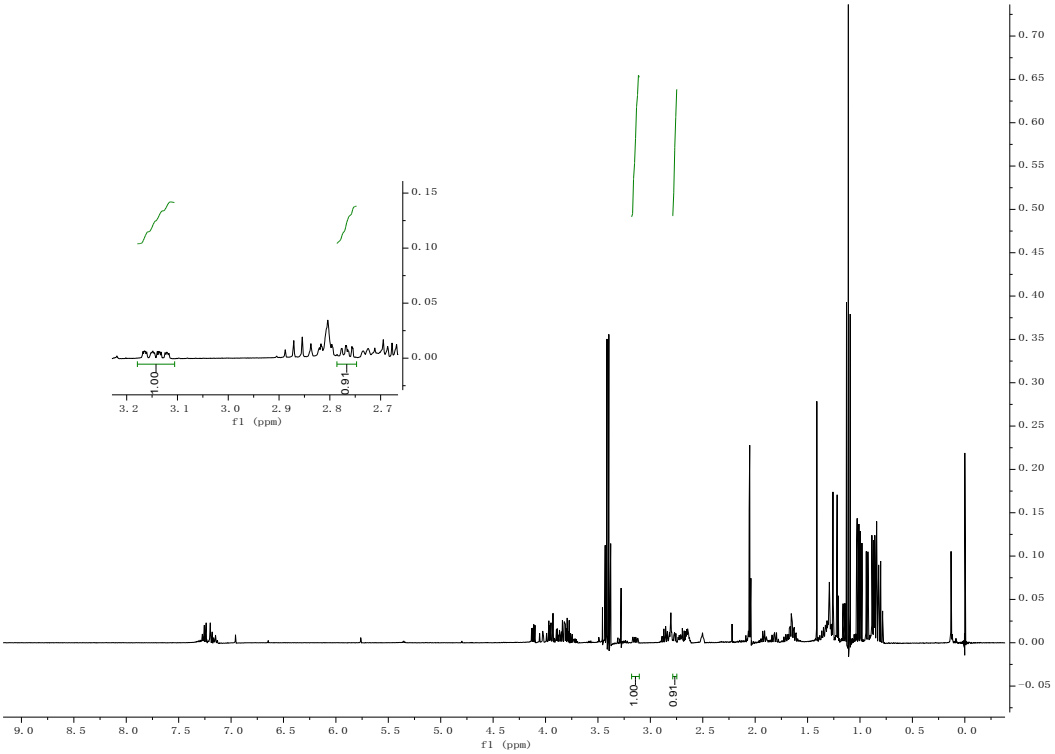


^{13}C NMR spectra of compound **2** in CD_3OD

^{1}H NMR analysis of the ratio of 2/1c epimers



^{1}H NMR spectra of compounds **2** and **1c** epimers (entry 2 in **Table 2**)



^{1}H NMR spectra of compounds **2** and **1c** epimers (entry 6 in **Table 2**)

X-Ray Crystallographic Data

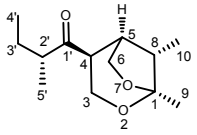
Crystal data and structure refinement for Data CCDC 1533934

Empirical formula	$C_{16}H_{28}O_3Si$
Formula weight	296.47
Temperature / K	104.6
Crystal system	monoclinic
Space group	$P2_1$
a / Å, b / Å, c / Å	9.9108(3), 9.8191(3), 18.3013(6)
α/°, β/°, γ/°	90, 91.679(3), 90
Volume / $Å^3$	1780.22(10)
Z	4
ρ_{calc} / mg mm^{-3}	1.106
μ / mm^{-1}	1.200
F(000)	648
Crystal size / mm^3	0.200 × 0.130 × 0.100
2Θ range for data collection	9.67 to 142.256°
Index ranges	$-12 \le h \le 11$, $-11 \le k \le 11$, $-22 \le l \le 21$
Reflections collected	13060
Independent reflections	6632[R(int) = 0.0299 (inf-0.9Å)]
Data/restraints/parameters	6632/1/375
Goodness-of-fit on F^2	1.099
Final R indexes [I>2σ (I) i.e. F_o>4σ (F_o)]	R_1 = 0.0579, wR_2 = 0.1552
Final R indexes [all data]	R_1 = 0.0597, wR_2 = 0.1572
Largest diff. peak/hole / e $Å^{-3}$	0.941/-0.237
Flack Parameters	0.03(2)
Completeness	0.999

Crystal data and structure refinement for Data CCDC 1533935

Empirical formula	$C_{16}H_{28}O_3Si$
Formula weight	296.47
Temperature / K	105.3
Crystal system	monoclinic
Space group	$P2_1$
a / Å, b / Å, c / Å	12.0052(10), 9.6664(6), 15.8157(12)
α/°, β/°, γ/°	90.00, 96.318(8), 90.00
Volume / Å^3	1824.2(2)
Z	4
ρ_{calc} / mg mm^{-3}	1.079
μ / mm^{-1}	1.171
F(000)	648
Crystal size / mm^3	0.42 × 0.17 × 0.05
2Θ range for data collection	8.8 to 142.72°
Index ranges	-14 ≤ h ≤ 14, -11 ≤ k ≤ 11, -19 ≤ l ≤ 19
Reflections collected	12860
Independent reflections	6840[R(int) = 0.0300 (inf-0.9Å)]
Data/restraints/parameters	6840/1/375
Goodness-of-fit on F^2	1.036
Final R indexes [I>2σ (I) i.e. F_o>4σ (F_o)]	R_1 = 0.0404, wR_2 = 0.1070
Final R indexes [all data]	R_1 = 0.0456, wR_2 = 0.1142
Largest diff. peak/hole / e Å^{-3}	0.454/-0.205
Flack Parameters	0.03(2)
Completeness	0.986

NMR Comparison Table of natural colomitide B and synthetic 2



Colomitide B (**2**)

Table S1 The comparison of ^{1}H and ^{13}C NMR data of reported natural colomitide B and synthetic 2 in $(CD_3)CO$

Position	δ H (600 MHz *J* in Hz)		δ C (150MHz)	
	Natural colomitide B	**Synthetic 2**	**Natural colomitide B**	**Synthetic 2**
1			109.1	109
3	A 4.04 (d, 11.7) B 3.79 (dd, 11.8, 5.1)	A 4.04 (d, 11.7) B 3.79 (dd, 11.7, 5.1)	59.4	59.3
4	2.76 (t, 8.1, 3.5)	2.77 m	53.5	53.4
5	2.71 (d, 3.5)	2.72 m	43.0	42.9
6	A 4.11 (dd, 8.3, 4.5) B 3.95 (d, 8.4)	A 4.12 (dd, 8.4, 4.5) B 3.96 (d, 8.4)	71.8	71.7
8	1.79 (dd, 14.0, 7.0)	1.80 (dd, 14.0, 7.0)	43.6	43.5
9	1.21 (s)	1.22 (s)	20.5	20.4
10	0.87 (d, 7.0)	0.88 (d, 7.1)	14.3	14.2
1'			214	214
2'	2.86 (dd, 13.4, 6.7)	2.86 (dd, 13.4, 6.7)	44.8	44.7
3'	A 1.62-1.57 (m) B 1.34-1.29 (m)	A 1.68–1.61 (m) B 1.38–1.31 (m)	27.4	27.3
4'	0.84 (t, 7.4)	0.84 (t, 7.4)	11.9	11.8
5'	1.01 (d, 6.7)	1.02 (d, 6.8)	16.6	16.5