

Supplementary Material

Artemongolides A–F, undescribed sesquiterpenoid dimers from *Artemisia mongolica* and their antihepatic fibrosis activities

Chong Shang^{a,b}, Xiao-Yan Huang^a, Yuan Wang^a, Wei Dong^a, Xiao-Feng He^a, Tian-Ze Li^a, Ji-Jun Chen^{a, b*}

^a *State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences, Kunming 650201, People's Republic of China*

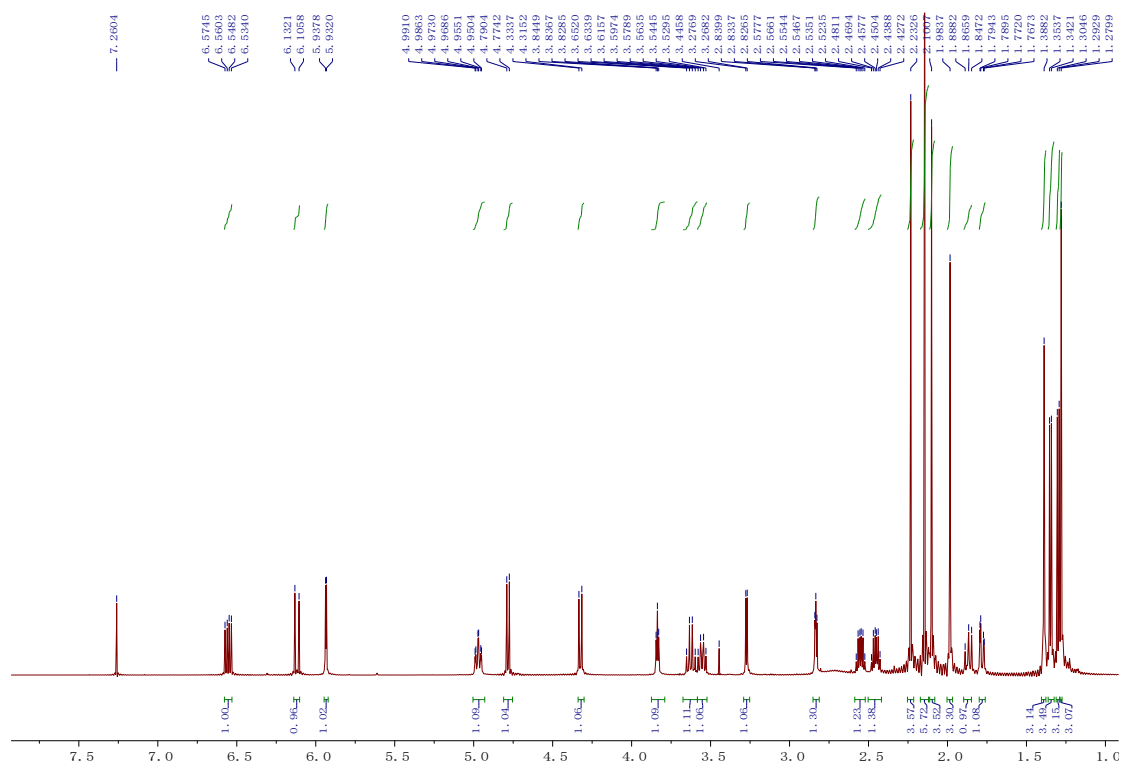
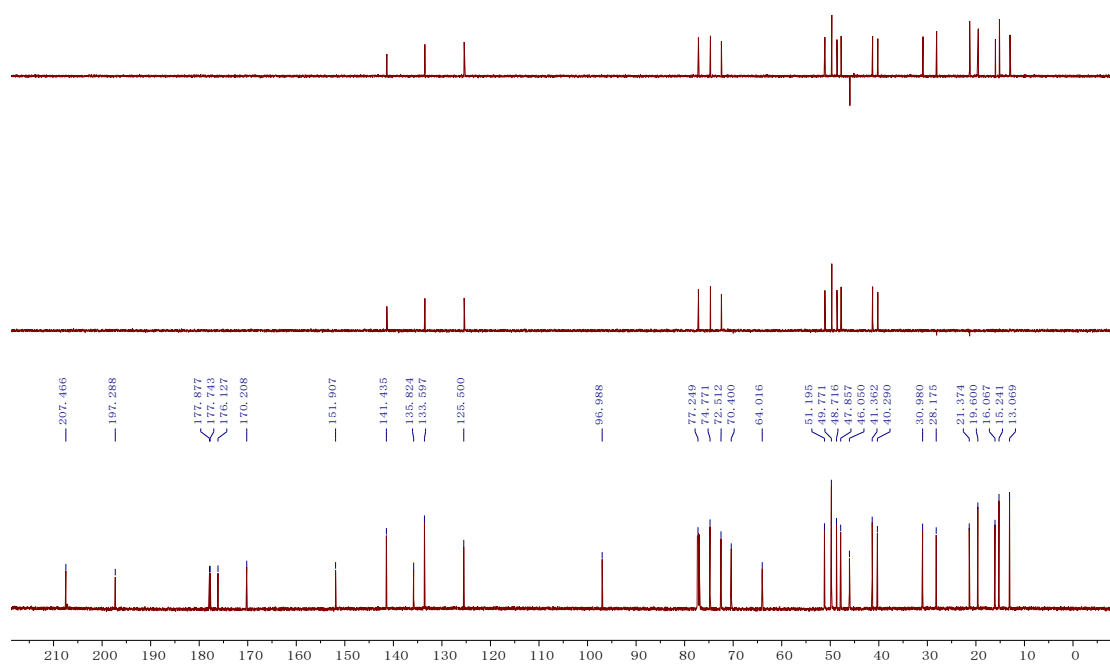
^b *University of Chinese Academy of Sciences, Beijing 100049, People's Republic of China*

Corresponding Author

* Tel: + 86 871 65223265. Fax: + 86 871 65227197. E-mail address: chenjj@mail.kib.ac.cn

目录

1. NMR, MS, IR, $[\alpha]_{\text{D}}$ and CD spectra of compound 1	3
2. NMR, MS, IR, $[\alpha]_{\text{D}}$ and CD spectra of compound 2	8
3. NMR, MS, IR, $[\alpha]_{\text{D}}$ and CD spectra of compound 3	13
4. NMR, MS, IR, $[\alpha]_{\text{D}}$ and CD spectra of compound 4	18
5. NMR, MS, IR, $[\alpha]_{\text{D}}$ and CD spectra of compound 5	23
6. NMR, MS, IR, $[\alpha]_{\text{D}}$ and CD spectra of compound 6	28
7. General experimental procedures.....	33
8. ECD calculations for compounds 2–5	34
9. Cytotoxicity assay	35
10. Col I, HA, and HL secretion assay.....	36
11. Crystal data for compound 1	37
12. Crystal data for compound 6	39

1. NMR, MS, IR, $[\alpha]$ and CD spectra of compound 1Figure. S1 ^1H NMR spectrum (600 MHz) of artemongolide A (1) in CDCl_3 Figure. S2 ^{13}C NMR spectrum (150 MHz) of artemongolide A (1) in CDCl_3

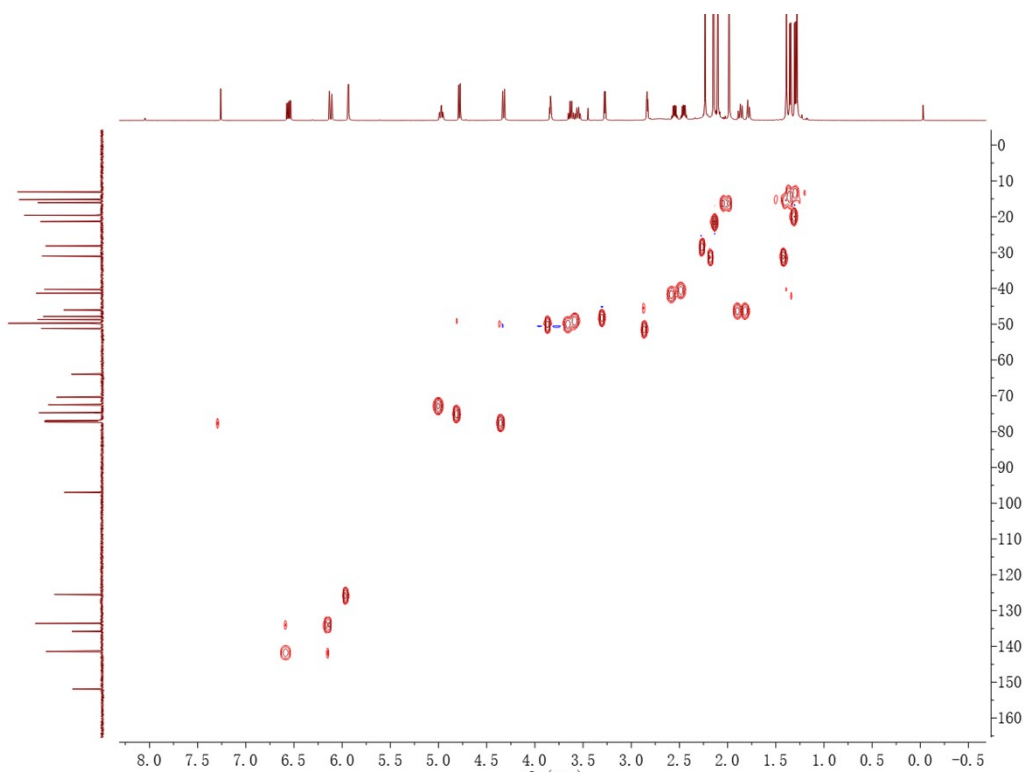


Figure. S3 HSQC spectrum (600 MHz) of artemongolide A (1) in CDCl₃

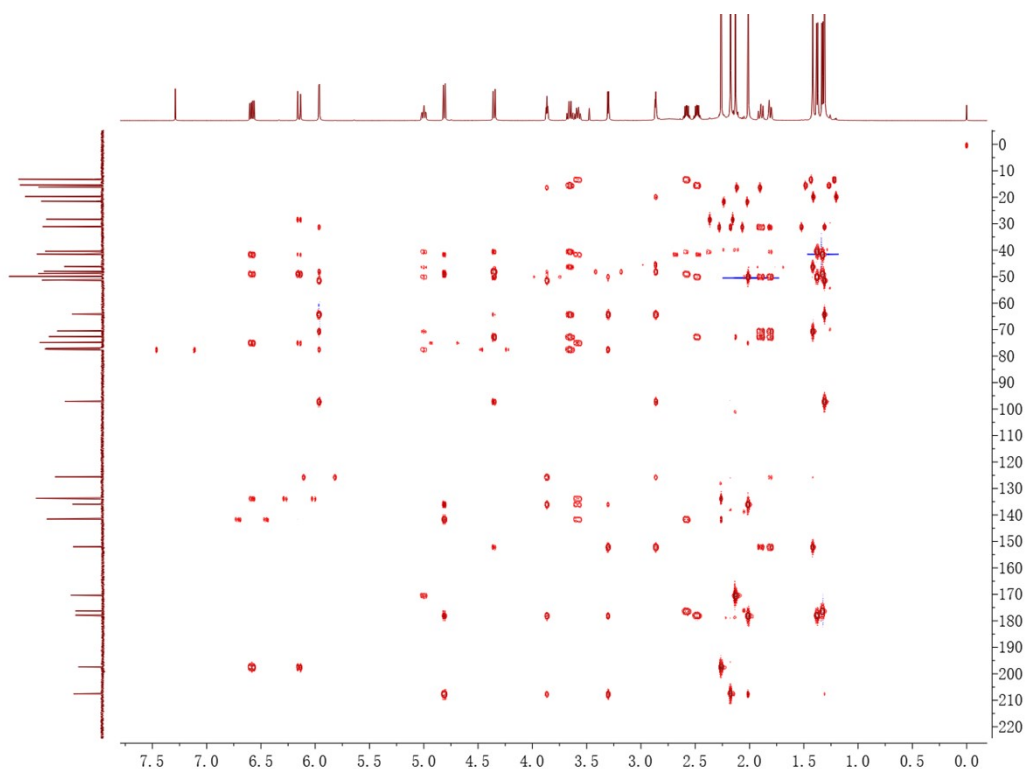


Figure. S4 HMBC spectrum (600 MHz) of artemongolide A (1) in CDCl₃

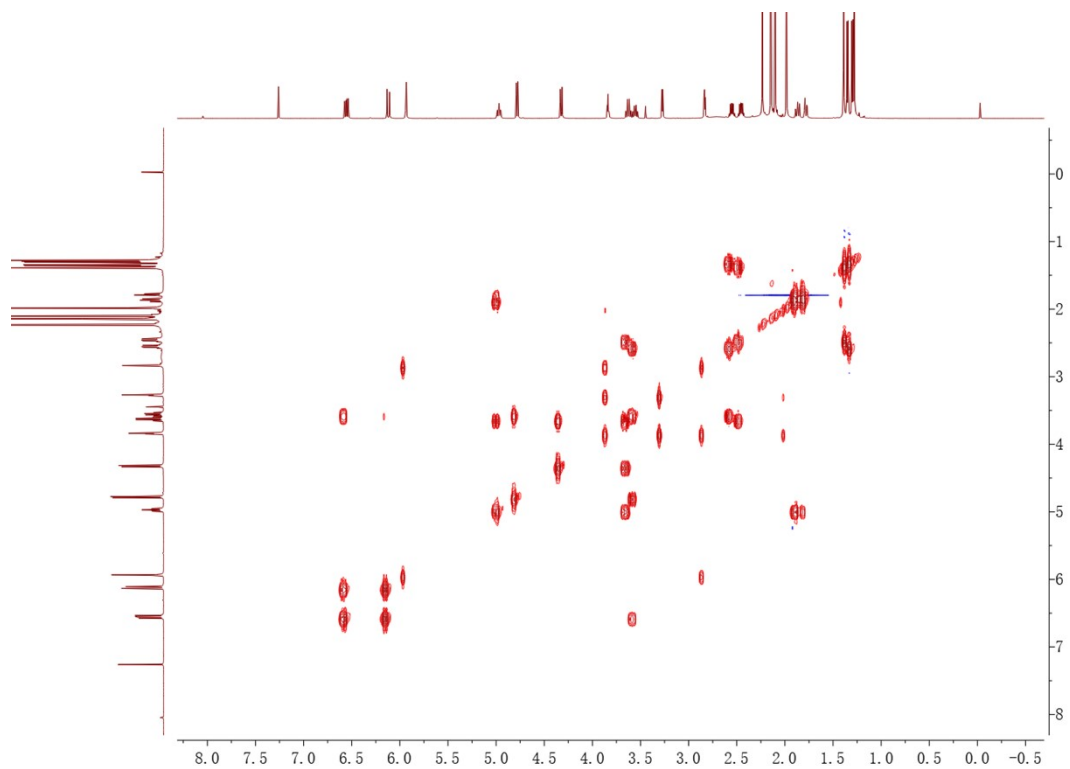


Figure. S5 ^1H - ^1H COSY spectrum (600 MHz) of artemongolide A (1) in CDCl_3

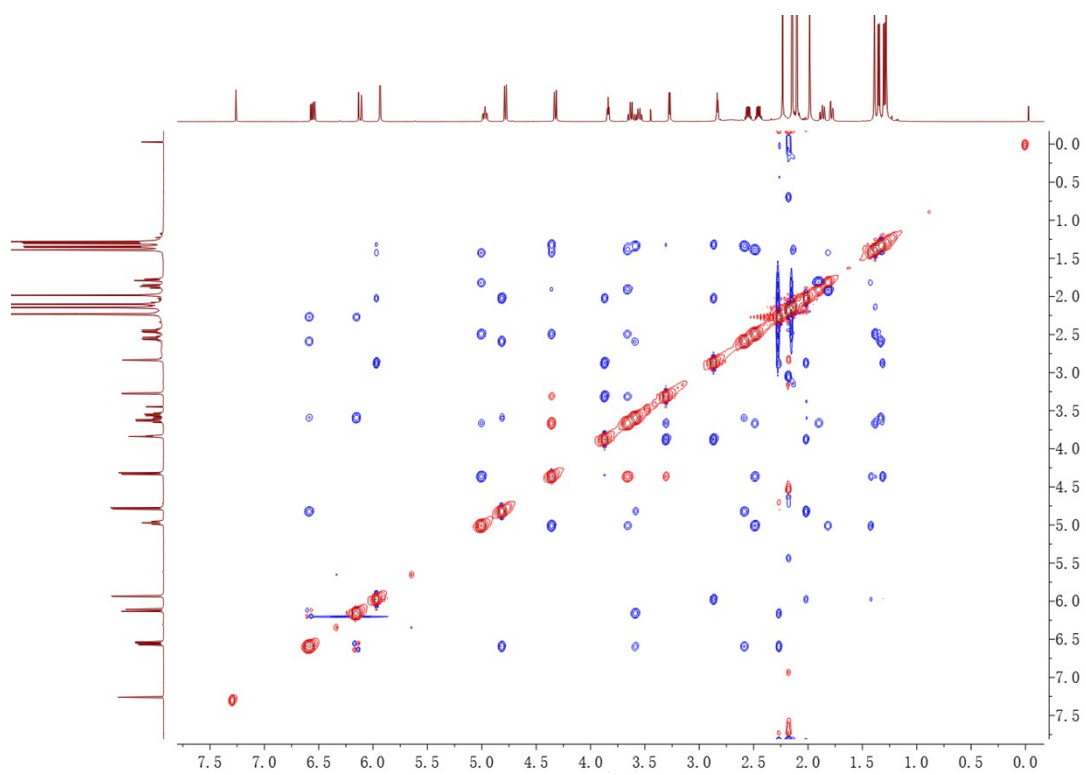


Figure. S6 ROESY spectrum (600 MHz) of artemongolide A (1) in CDCl_3

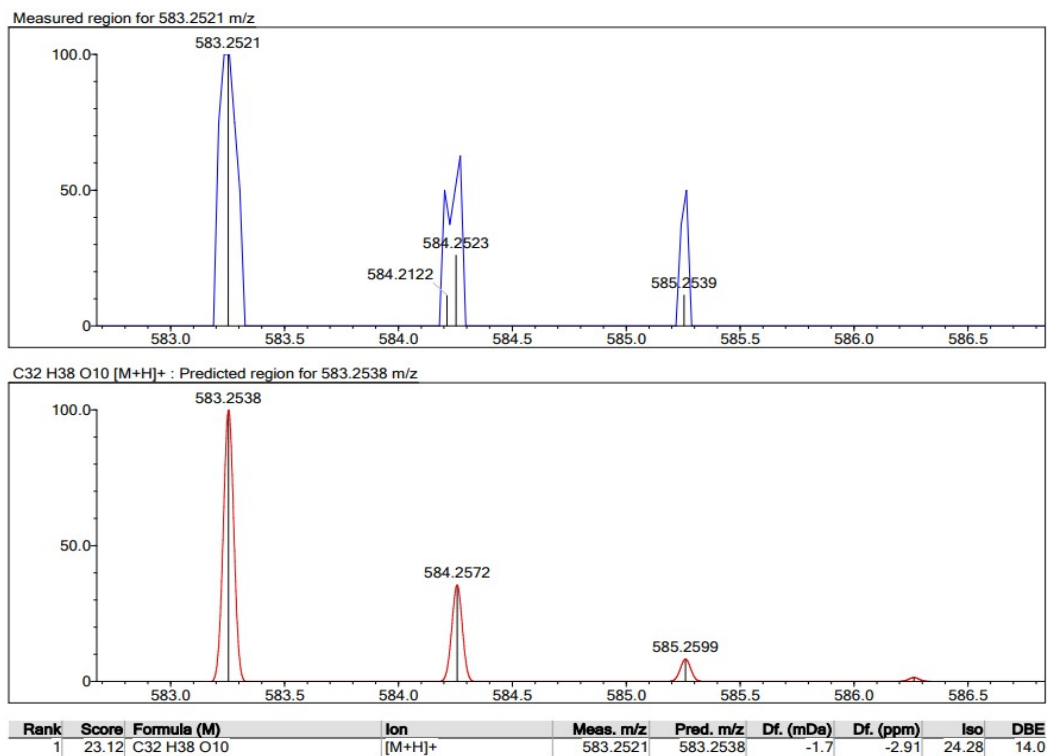


Figure. S7 HRESIMS spectrum of artemongolide A (1)

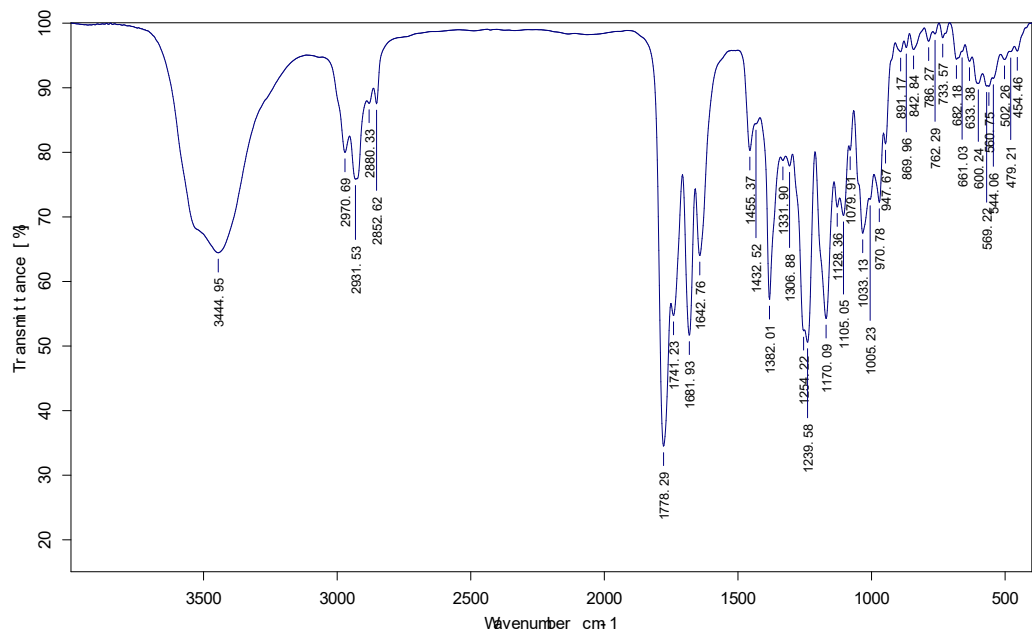


Figure. S8 IR spectrum of artemongolide A (1)

Rudolph Research Analytical

This sample was measured on an Autopol VI, Serial #91058
Manufactured by Rudolph Research Analytical, Hackettstown, NJ, USA.

Measurement Date : Friday, 12-AUG-2022

Set Temperature : OFF

Time Delay : Disabled

Delay between Measurement : Disabled

<u>n</u>	<u>Average</u>	<u>Std.Dev.</u>	<u>% RSD</u>	<u>Maximum</u>	<u>Minimum</u>					
5	-54.33	0.17	-0.31	-54.17	-54.50					
<u>S.No</u>	<u>Sample ID</u>	<u>Time</u>	<u>Result</u>	<u>Scale</u>	<u>OR °Arc</u>	<u>WLG.nm</u>	<u>Lq.mm</u>	<u>Conc.g/100ml</u>	<u>Temp.</u>	
1	JSC-6	02:20:20 PM	-54.17	SR	-0.0325	589	100.00	0.060	24.6	
2	JSC-6	02:20:29 PM	-54.50	SR	-0.0327	589	100.00	0.060	24.5	
3	JSC-6	02:20:37 PM	-54.50	SR	-0.0327	589	100.00	0.060	24.6	
4	JSC-6	02:20:45 PM	-54.17	SR	-0.0325	589	100.00	0.060	24.6	
5	JSC-6	02:20:53 PM	-54.33	SR	-0.0326	589	100.00	0.060	24.6	

Figure. S9 Optical rotation spectrum of artemongolide A (1)

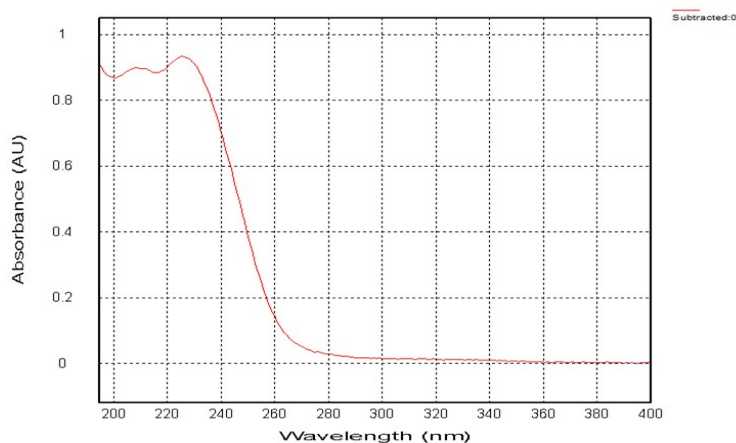
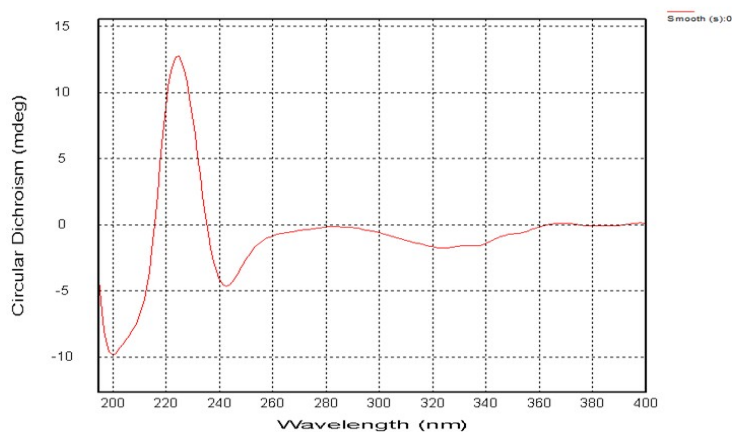


Figure. S10 CD (top) and UV (bottom) spectrums of artemongolide A (1)

B

2. NMR, MS, IR, $[\alpha]$ and CD spectra of compound 2

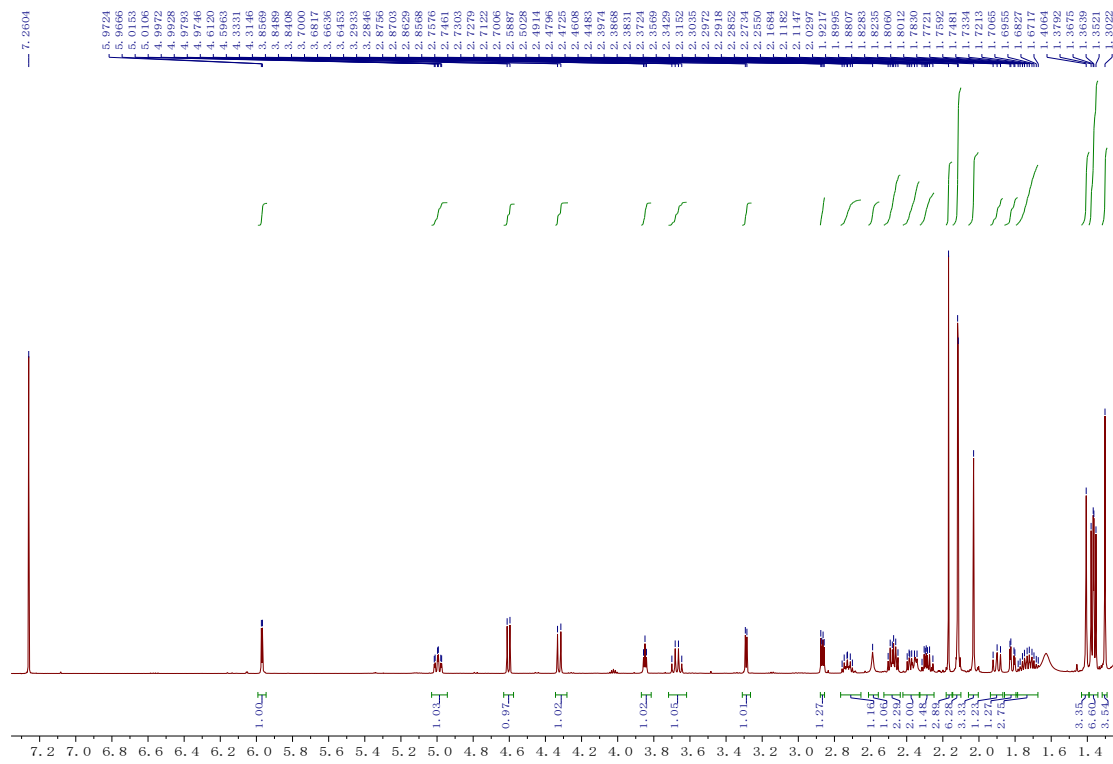


Figure. S11 ^1H NMR spectrum (600 MHz) of artemongolide B (2) in CDCl_3

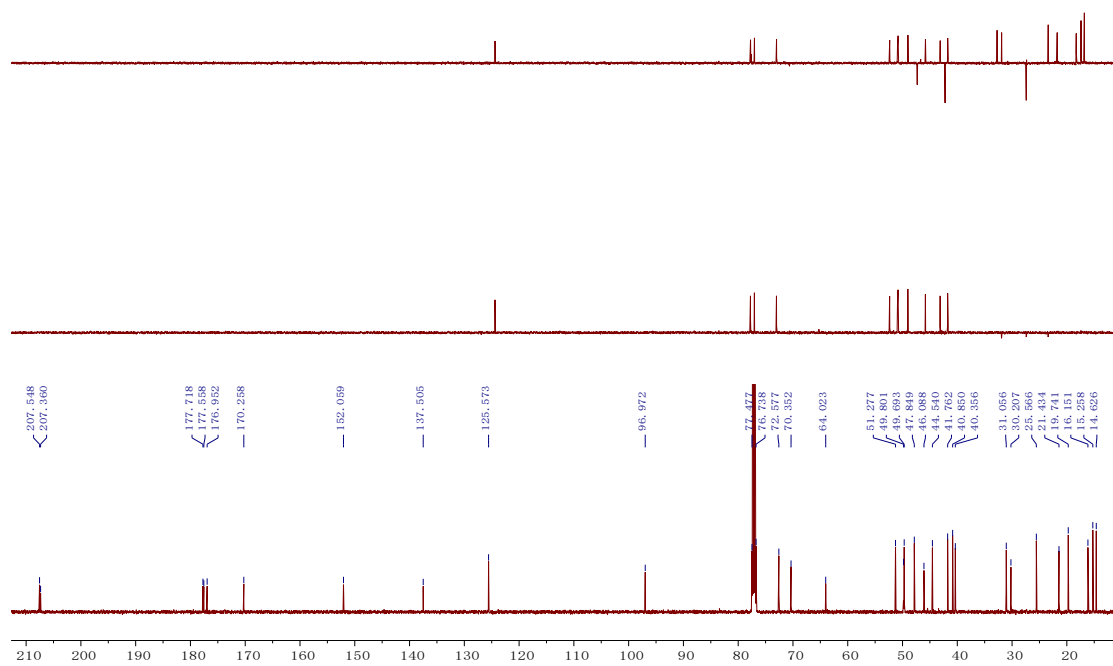


Figure. S12 ^{13}C NMR spectrum (150 MHz) of artemongolide B (2) in CDCl_3

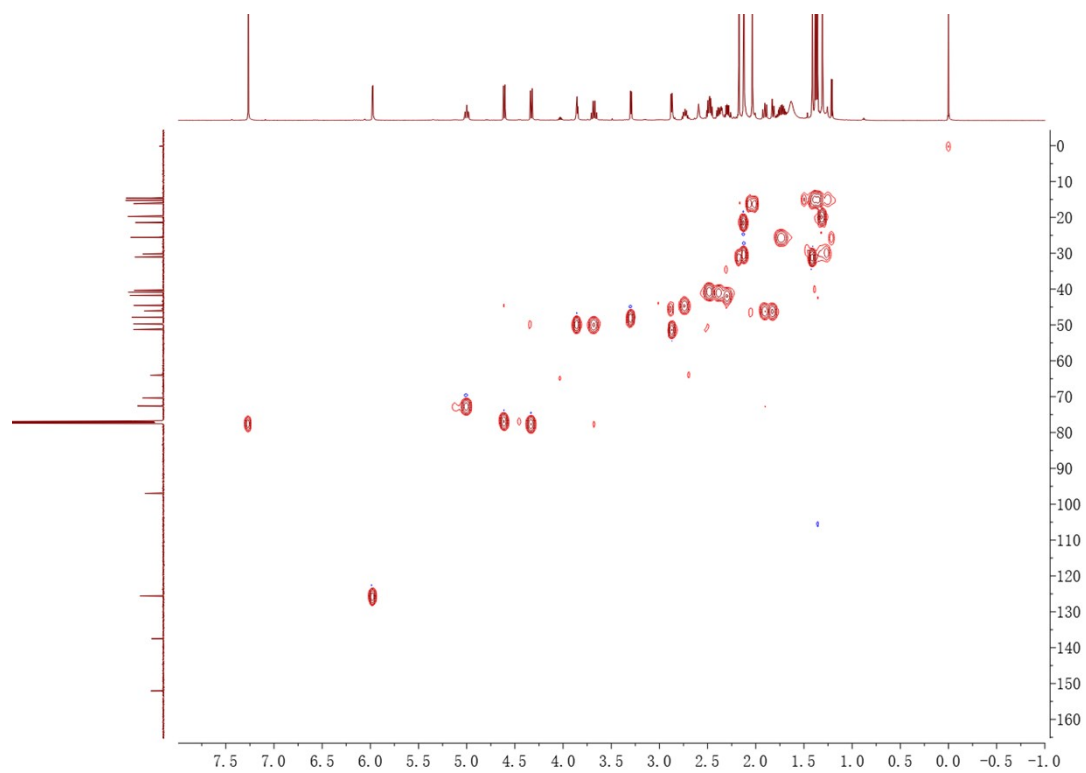


Figure. S13 HSQC spectrum (600 MHz) of artemongolide B (2) in CDCl_3

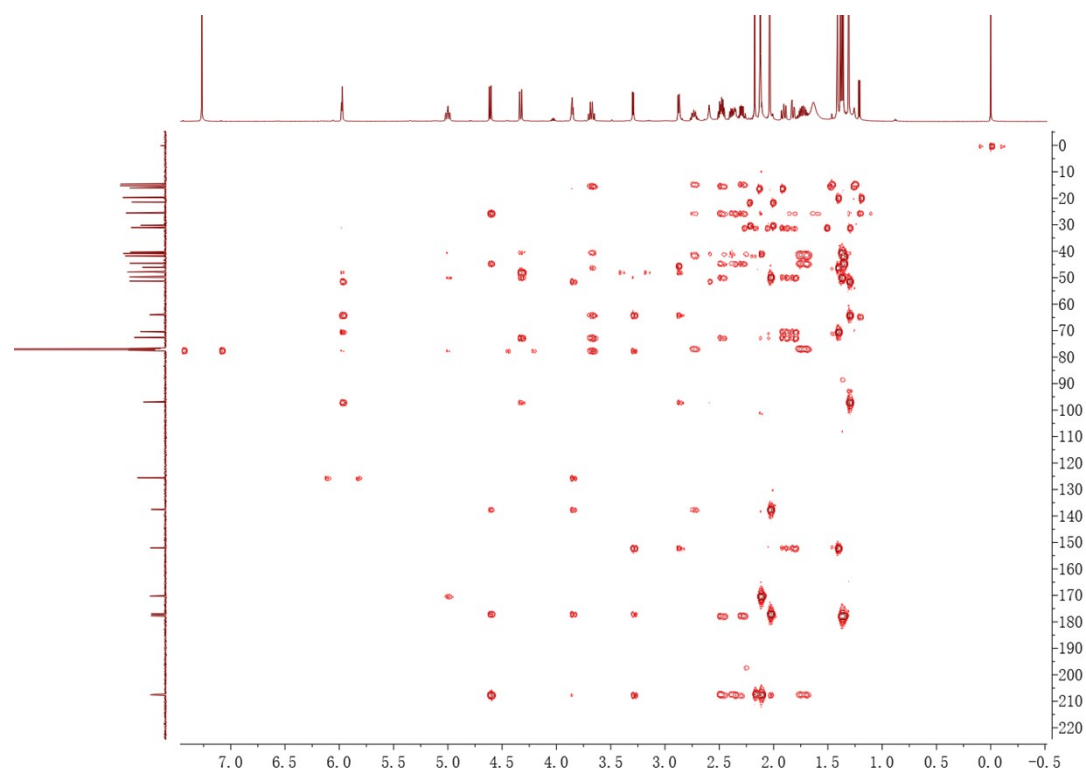


Figure. S14 HMBC spectrum (600 MHz) of artemongolide B (2) in CDCl_3

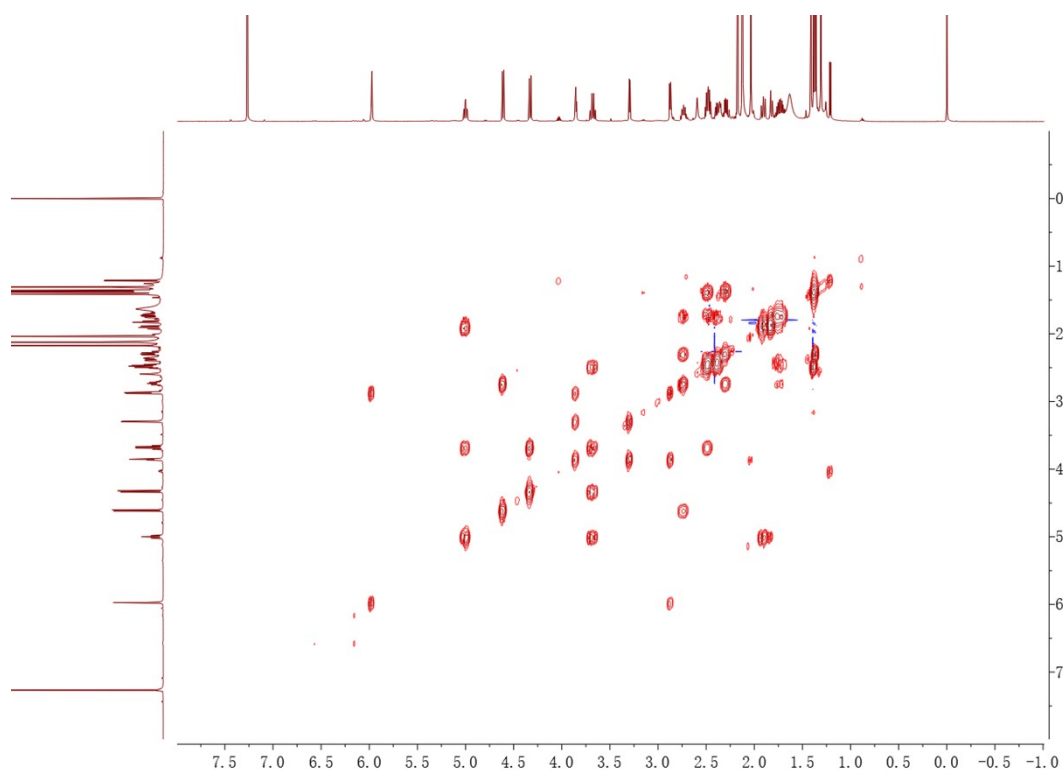


Figure. S15 ^1H - ^1H COSY spectrum (600 MHz) of artemongolide B (2) in CDCl_3

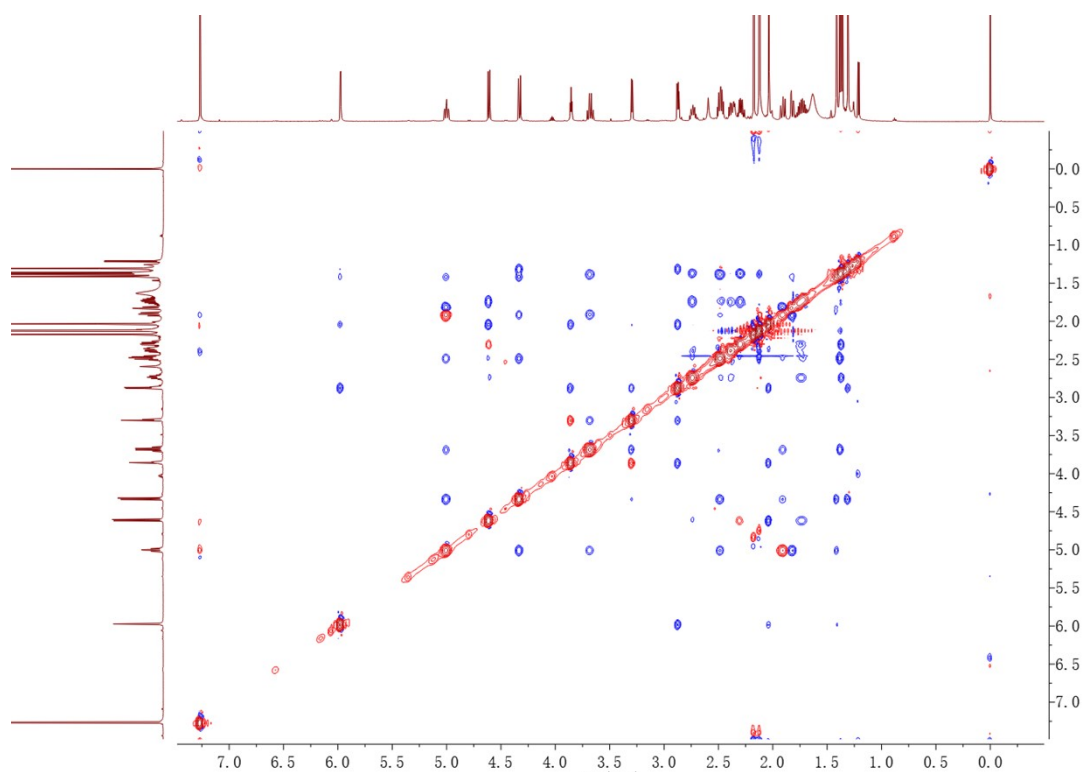


Figure. S16 ROESY spectrum (600 MHz) of artemongolide B (2) in CDCl_3

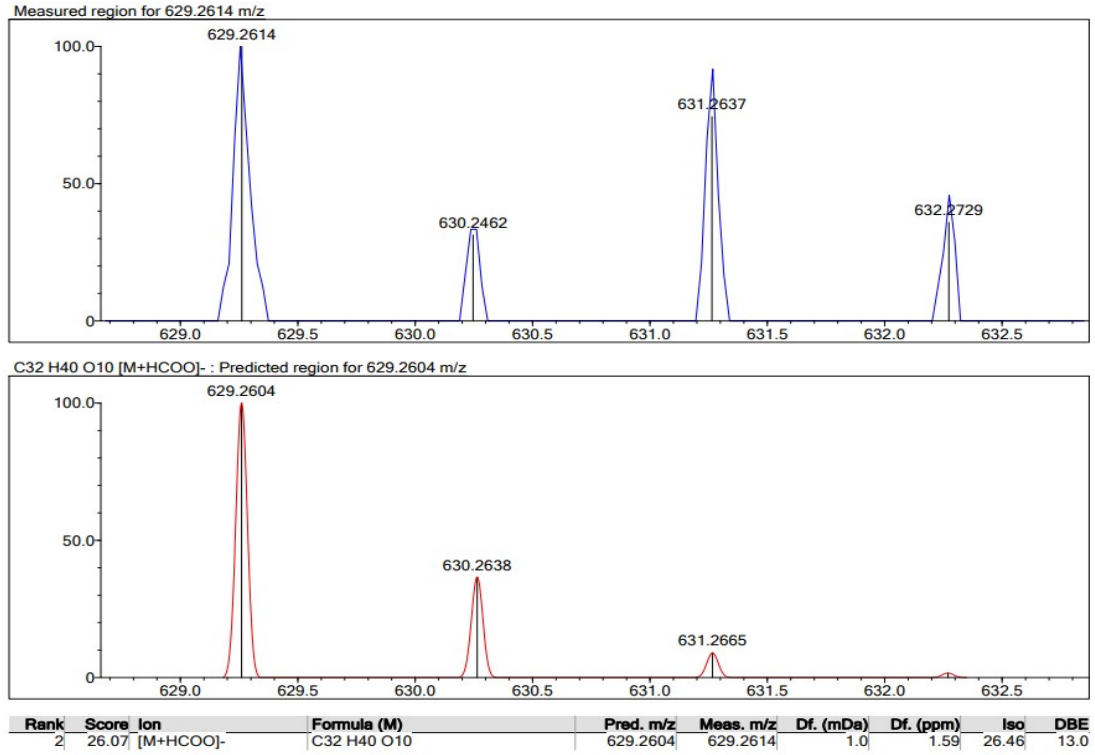


Figure. S17 HRESIMS spectrum of artemongolide B (2)

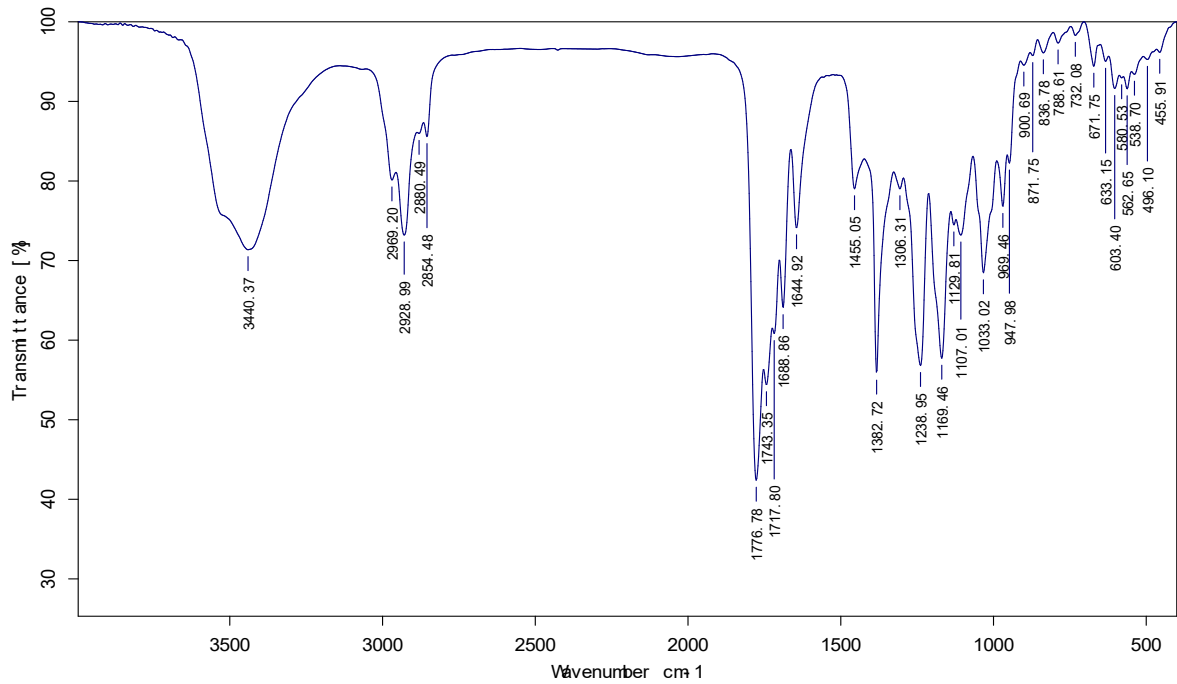


Figure. S18 IR spectrum of artemongolide B (2)

Rudolph Research Analytical

This sample was measured on an Autopol VI, Serial #91058
Manufactured by Rudolph Research Analytical, Hackettstown, NJ, USA.

Measurement Date : Friday, 12-AUG-2022

Set Temperature : OFF

Time Delay : Disabled

Delay between Measurement : Disabled

<u>n</u>	<u>Average</u>	<u>Std.Dev.</u>	<u>% RSD</u>	<u>Maximum</u>	<u>Minimum</u>					
5	-85.07	0.45	-0.52	-84.50	-85.67					
<u>S.No</u>	<u>Sample ID</u>	<u>Time</u>	<u>Result</u>	<u>Scale</u>	<u>OR °Arc</u>	<u>WLG.nm</u>	<u>Lg.mm</u>	<u>Conc.g/100ml</u>	<u>Temp.</u>	
1	JSC-5	02:14:20 PM	-85.33	SR	-0.0512	589	100.00	0.060	24.5	
2	JSC-5	02:14:29 PM	-85.00	SR	-0.0510	589	100.00	0.060	24.5	
3	JSC-5	02:14:37 PM	-84.50	SR	-0.0507	589	100.00	0.060	24.4	
4	JSC-5	02:14:45 PM	-84.83	SR	-0.0509	589	100.00	0.060	24.4	
5	JSC-5	02:14:53 PM	-85.67	SR	-0.0514	589	100.00	0.060	24.4	

Figure. S19 Optical rotation spectrum of artemongolide B (2)

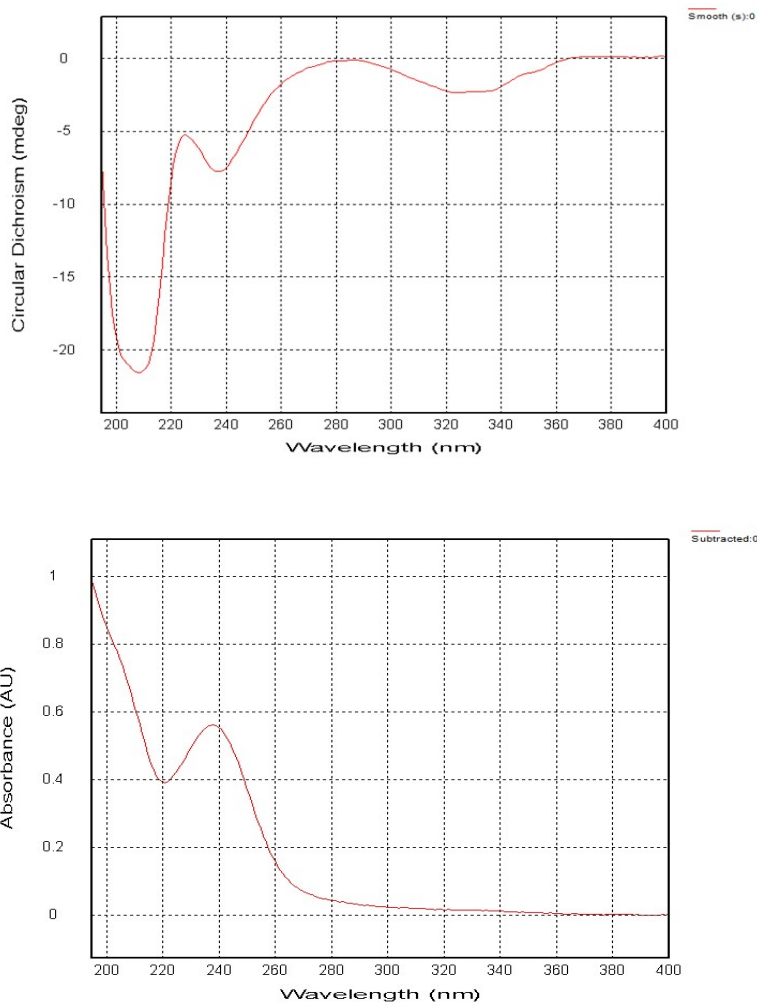
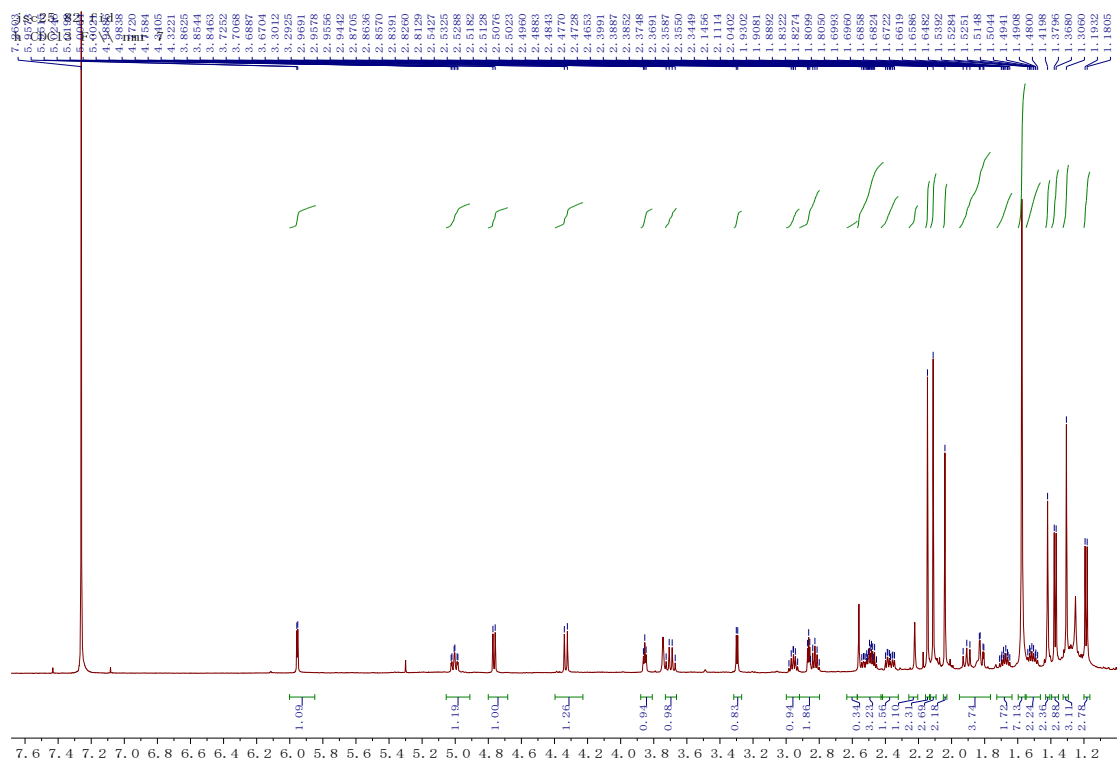
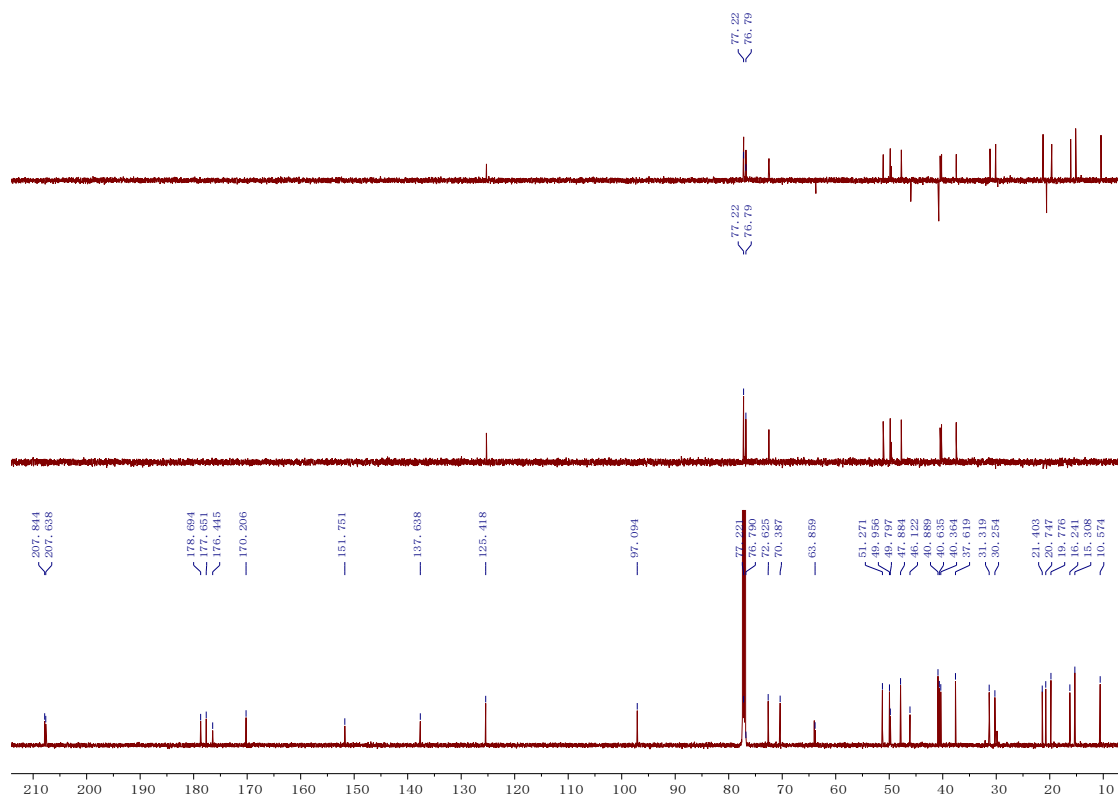


Figure. S20 CD (top) and UV (bottom) spectrums of artemongolide B (2)

3. NMR, MS, IR, $[\alpha]$ and CD spectra of compound 3Figure. S21 ^1H NMR spectrum (600 MHz) of artemongolide C (3) in CDCl_3 Figure. S22 ^{13}C NMR spectrum (150 MHz) of artemongolide C (3) in CDCl_3

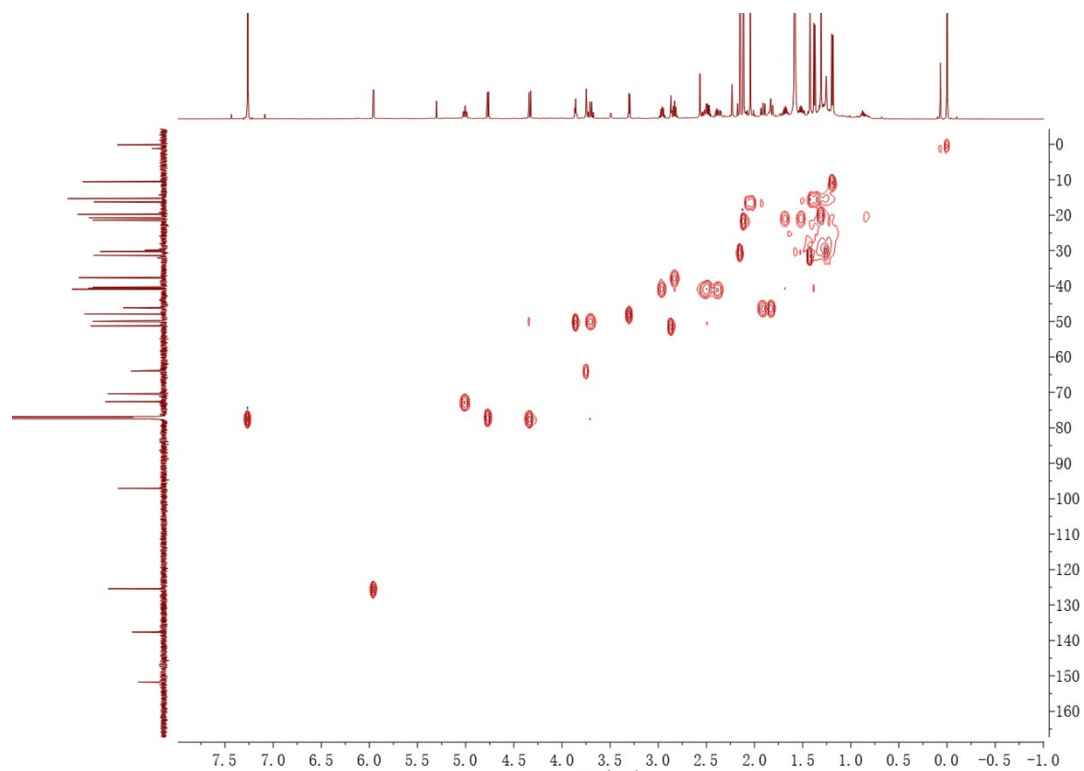


Figure. S23 HSQC spectrum (600 MHz) of artemongolide C (3) in CDCl₃

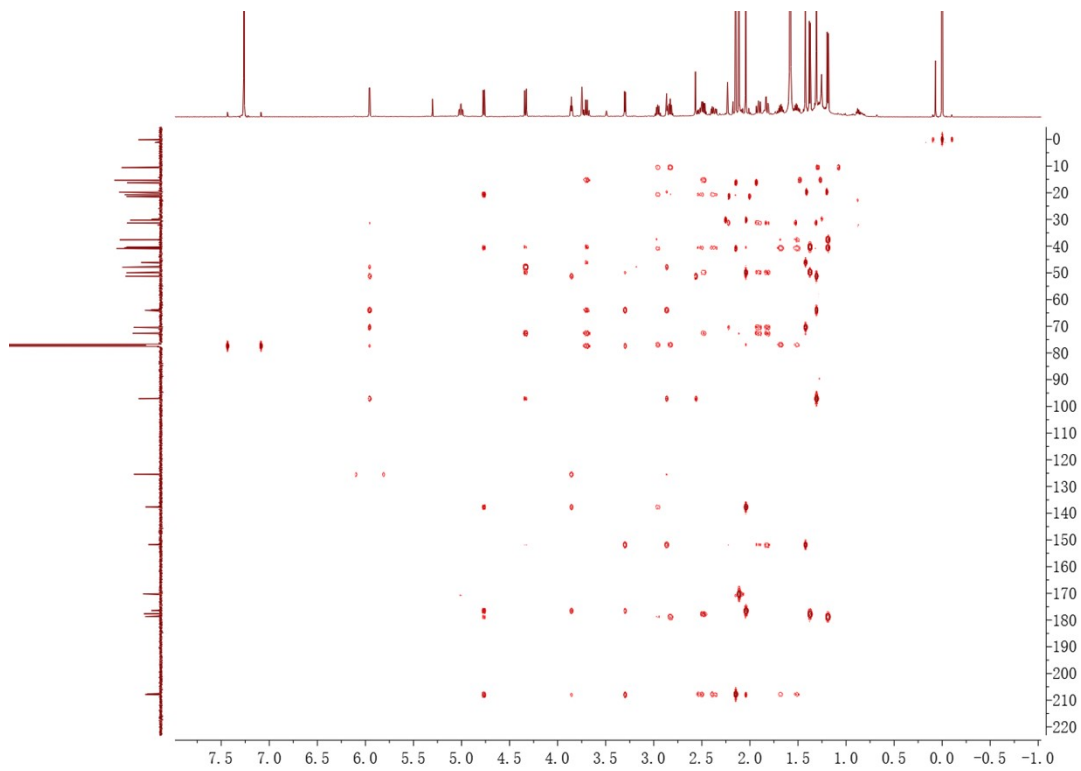


Figure. S24 HMBC spectrum (600 MHz) of artemongolide C (3) in CDCl₃

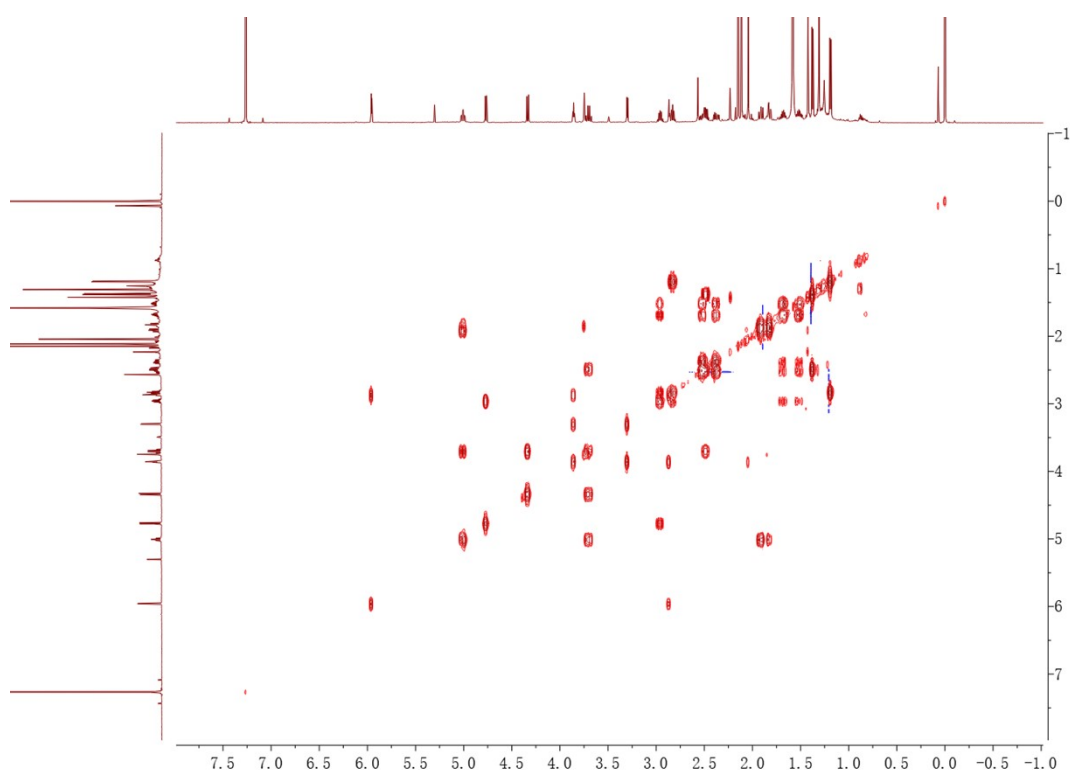


Figure. S25 ^1H - ^1H COSY spectrum (600 MHz) of artemongolide C (3) in CDCl_3

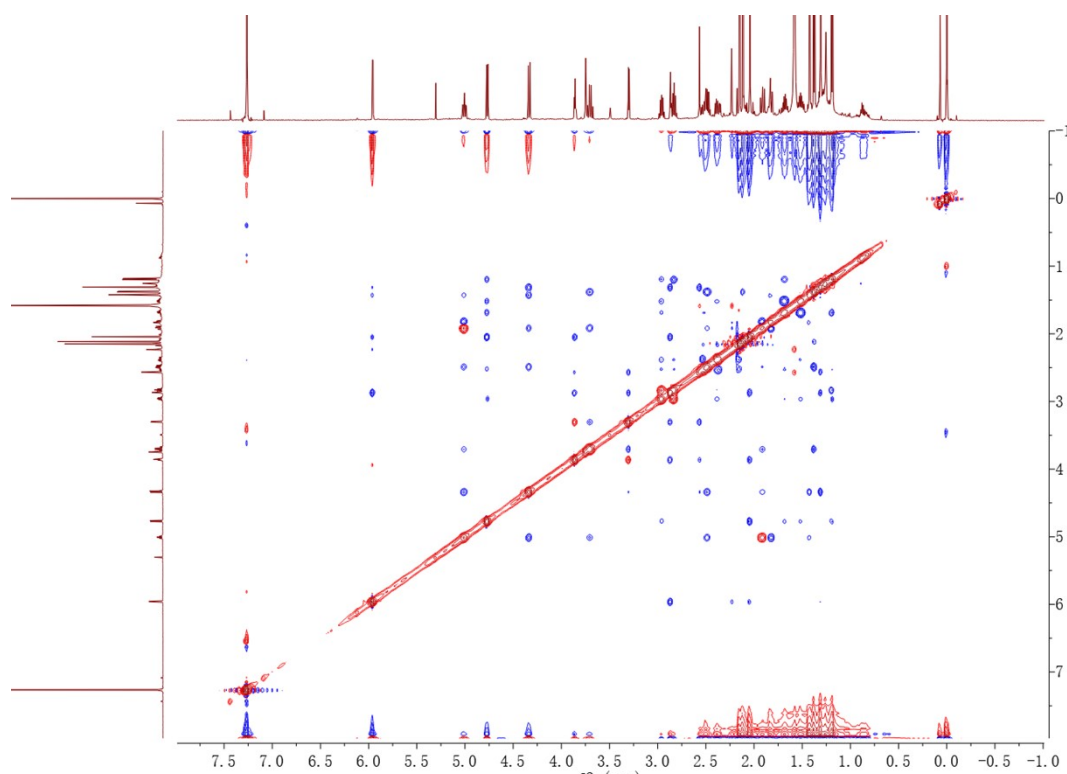


Figure. S26 ROESY spectrum (600 MHz) of artemongolide C (3) in CDCl_3

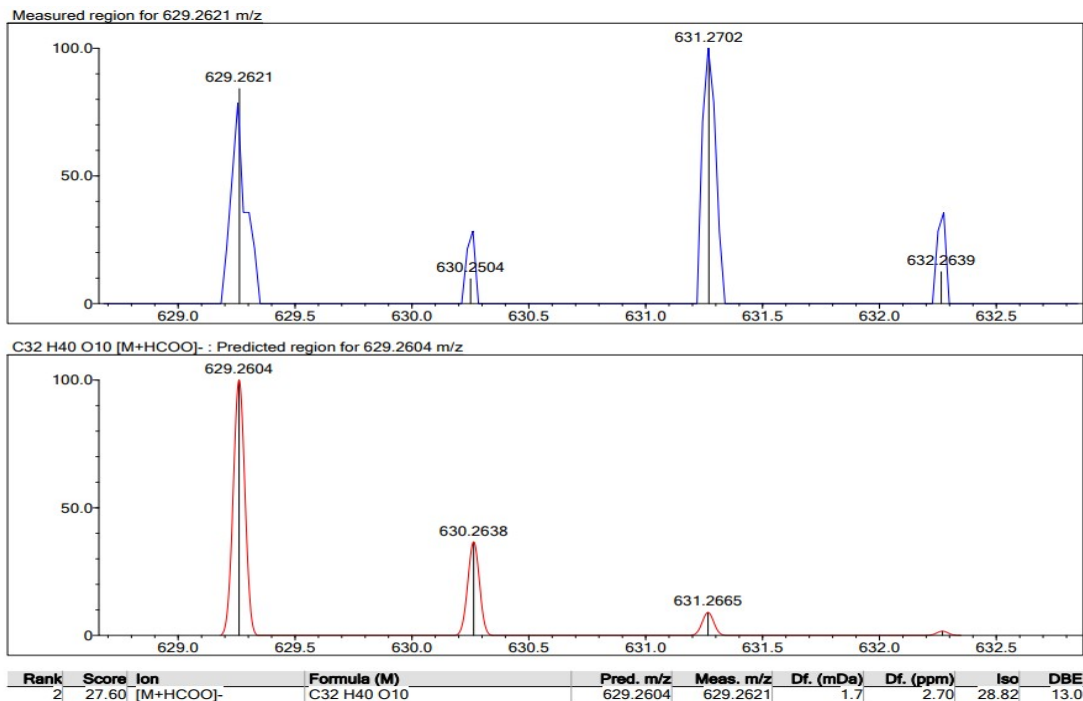


Figure. S27 HRESIMS spectrum of artemongolide C (3)

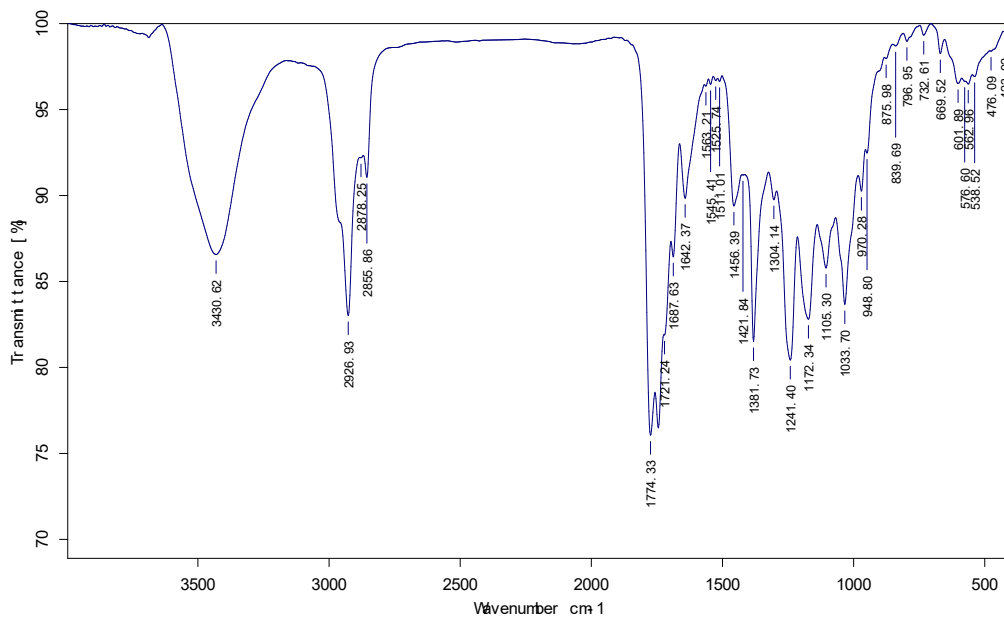


Figure. S28 IR spectrum of artemongolide C (3)

Rudolph Research Analytical

This sample was measured on an Autopol VI, Serial #91058
Manufactured by Rudolph Research Analytical, Hackettstown, NJ, USA.

Measurement Date : Friday, 12-AUG-2022

Set Temperature : OFF

Time Delay : Disabled

Delay between Measurement : Disabled

<u>n</u>	<u>Average</u>	<u>Std.Dev.</u>	<u>% RSD</u>	<u>Maximum</u>	<u>Minimum</u>					
5	-47.07	0.80	-1.69	-45.83	-47.83					
<u>S.No</u>	<u>Sample ID</u>	<u>Time</u>	<u>Result</u>	<u>Scale</u>	<u>OR °Arc</u>	<u>WLG.nm</u>	<u>Lg.mm</u>	<u>Conc.g/100ml</u>	<u>Temp.</u>	
1	JSC-25	02:47:04 PM	-45.83	SR	-0.0275	589	100.00	0.060	25.0	
2	JSC-25	02:47:12 PM	-47.17	SR	-0.0283	589	100.00	0.060	24.9	
3	JSC-25	02:47:20 PM	-47.67	SR	-0.0286	589	100.00	0.060	24.9	
4	JSC-25	02:47:29 PM	-47.83	SR	-0.0287	589	100.00	0.060	24.9	
5	JSC-25	02:47:37 PM	-46.83	SR	-0.0281	589	100.00	0.060	24.9	

Figure. S29 Optical rotation spectrum of artemongolide C (3)

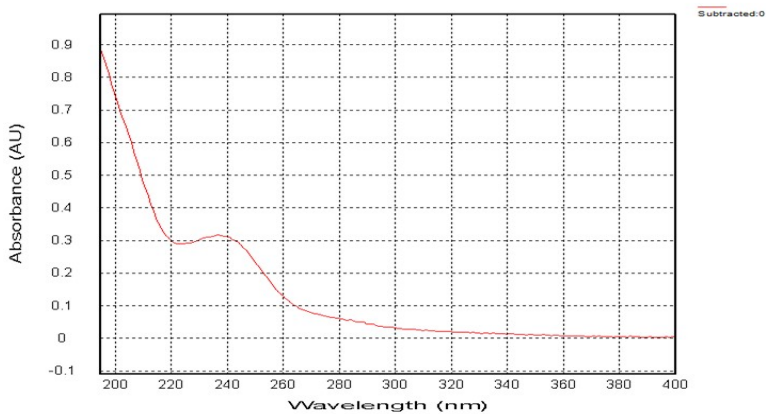
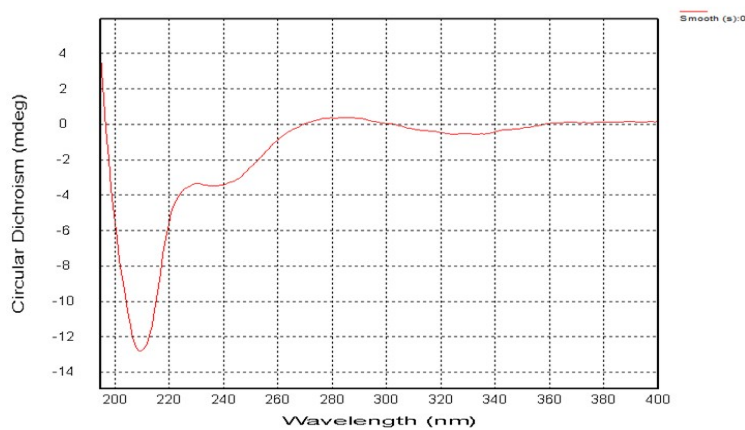
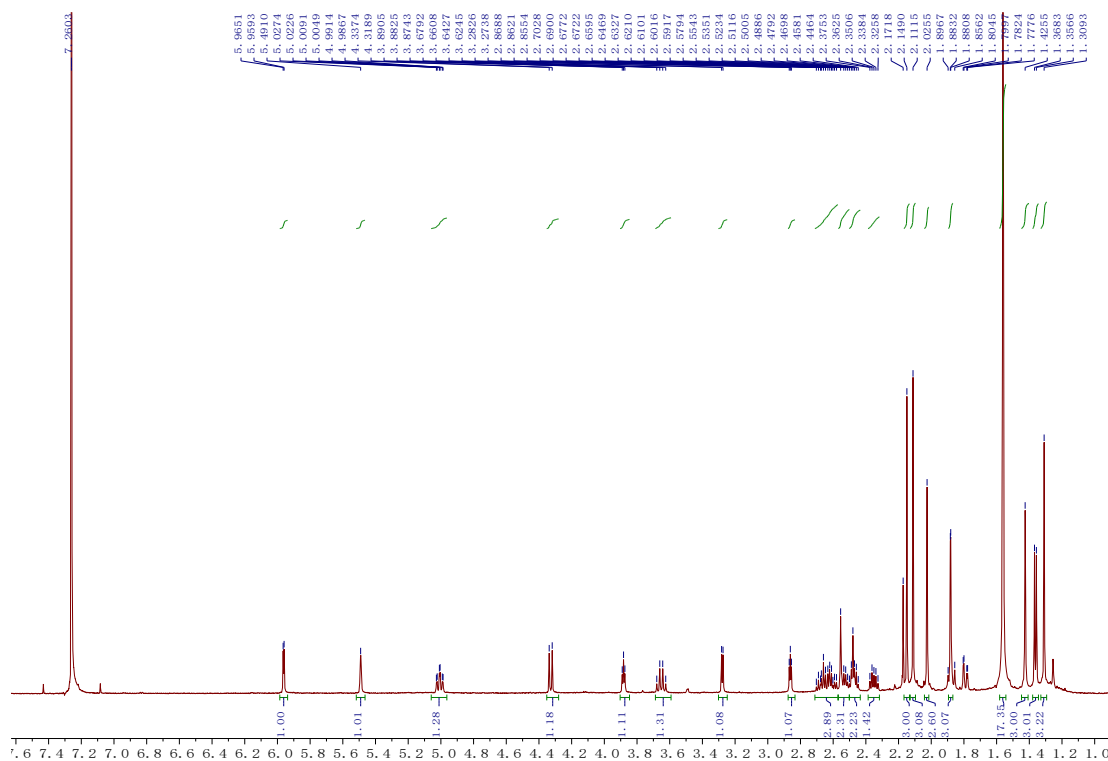
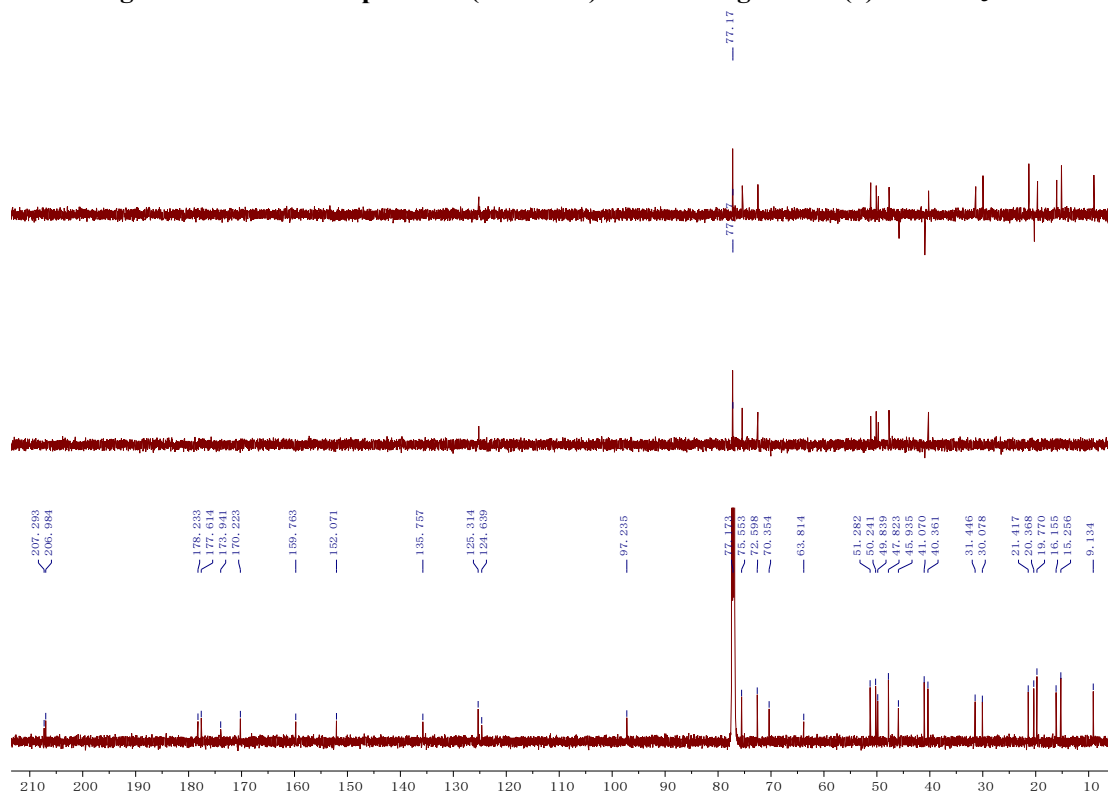


Figure. S30 CD (top) and UV (bottom) spectrums of artemongolide C (3)

4. NMR, MS, IR, $[\alpha]$ and CD spectra of compound 4Figure. S31 ^1H NMR spectrum (600 MHz) of artemongolide D (4) in CDCl_3 Figure. S32 ^{13}C NMR spectrum (150 MHz) of artemongolide D (4) in CDCl_3

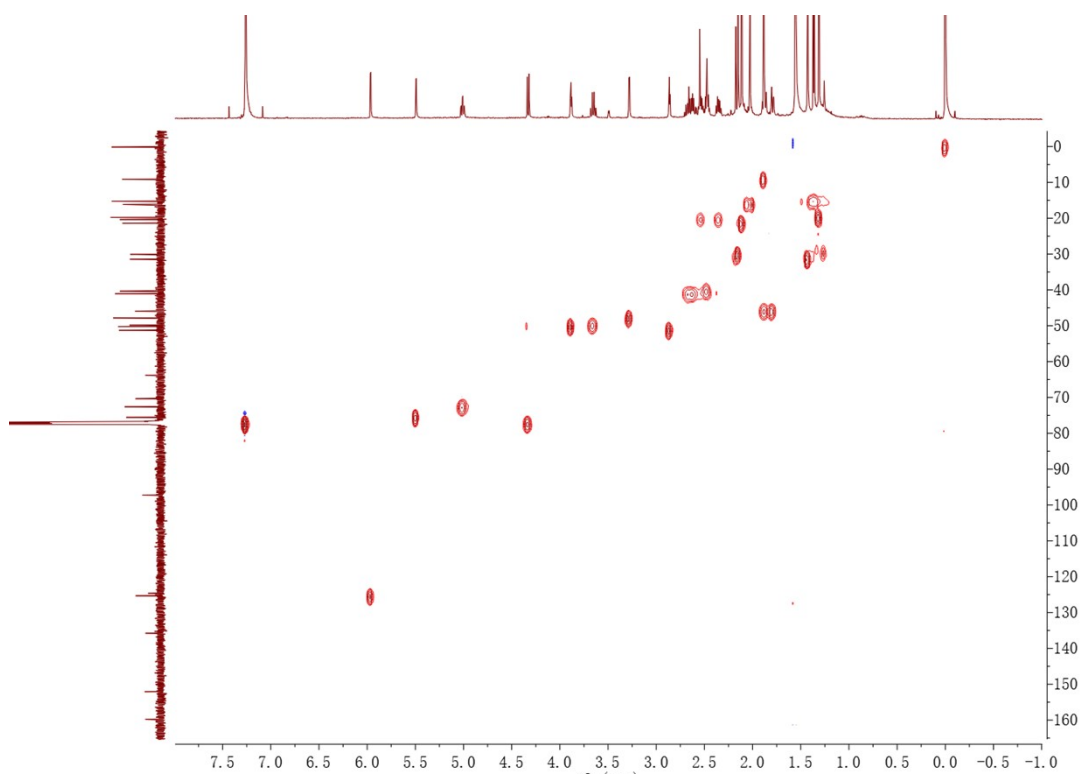


Figure. S33 HSQC NMR spectrum (600 MHz) of artemongolide D (4) in CDCl₃

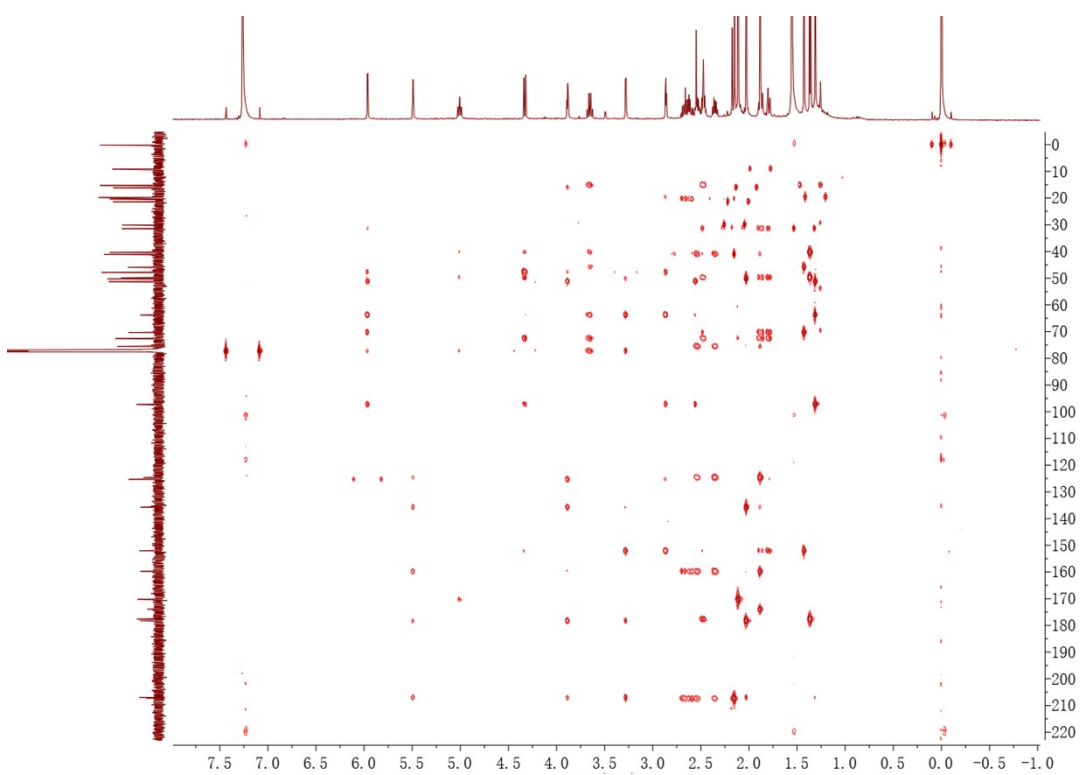


Figure. S34 HMBC spectrum (600 MHz) of artemongolide D (4) in CDCl₃

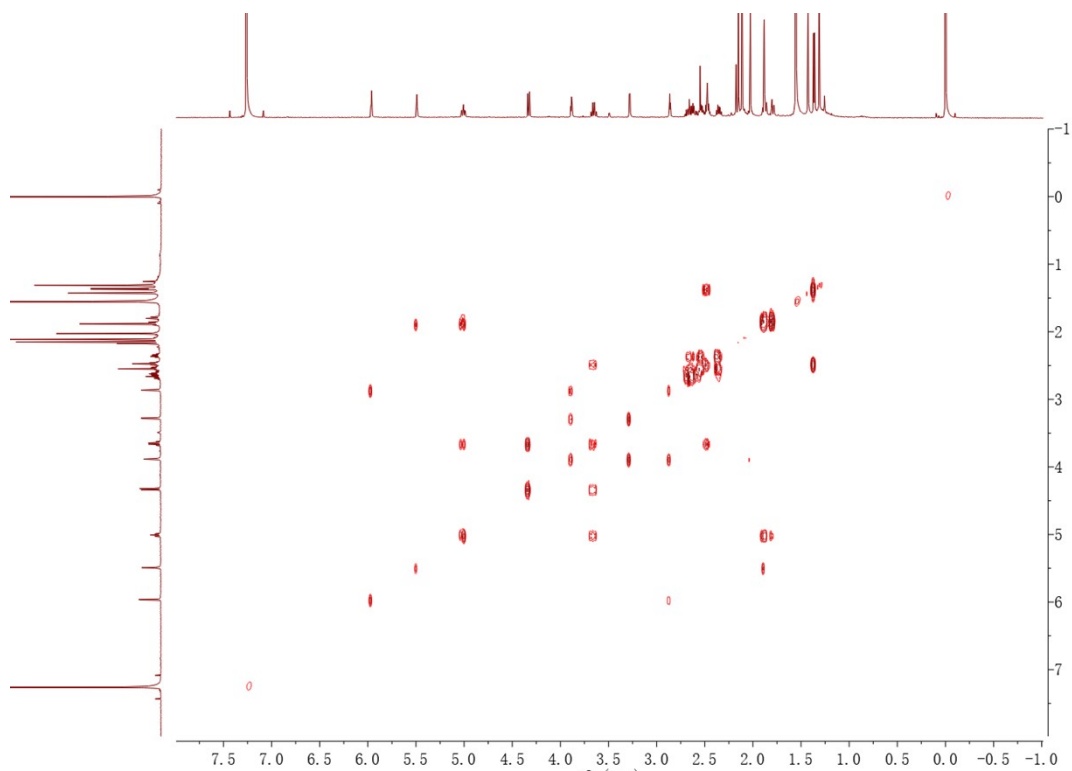


Figure. S35 ^1H - ^1H COSY spectrum (600 MHz) of artemongolide D (4) in CDCl_3

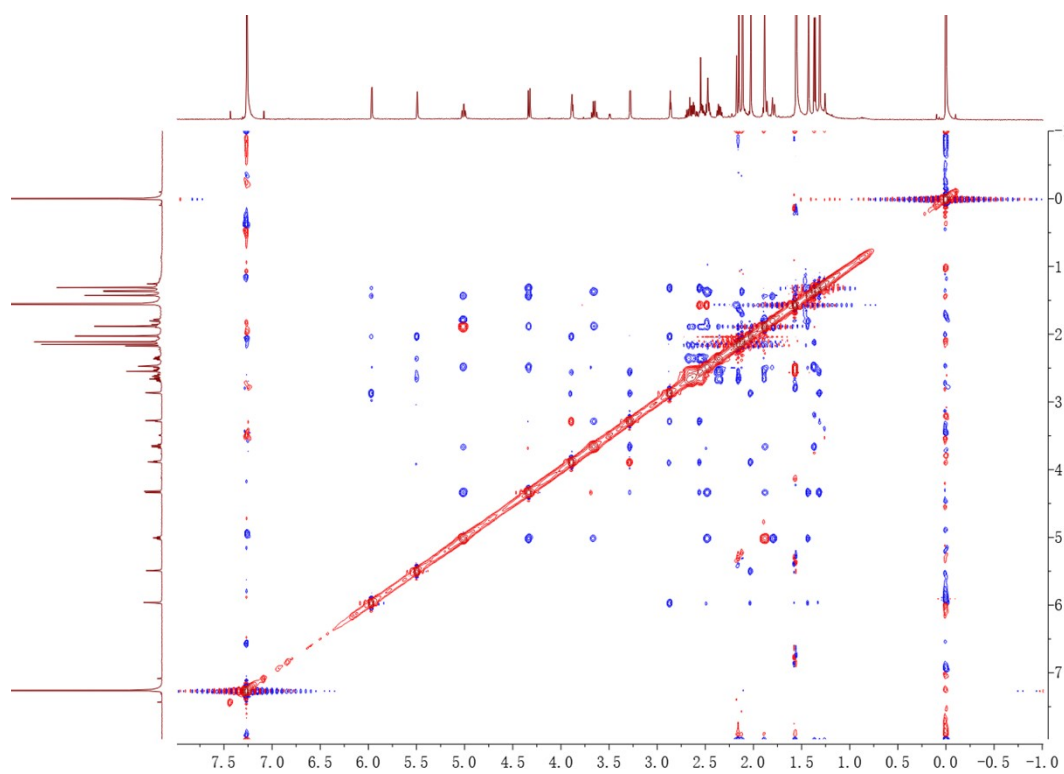


Figure. S36 ROESY spectrum (600 MHz) of artemongolide D (4) in CDCl_3

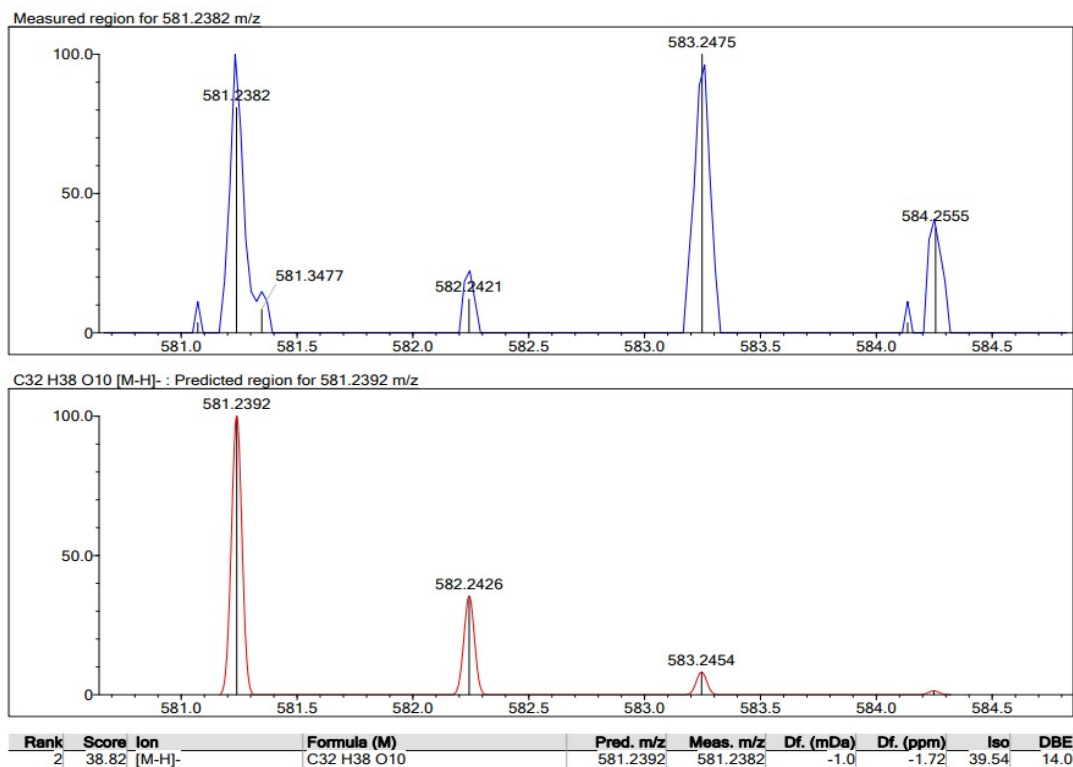


Figure. S37 HRESIMS spectrum of artemongolide D (4)

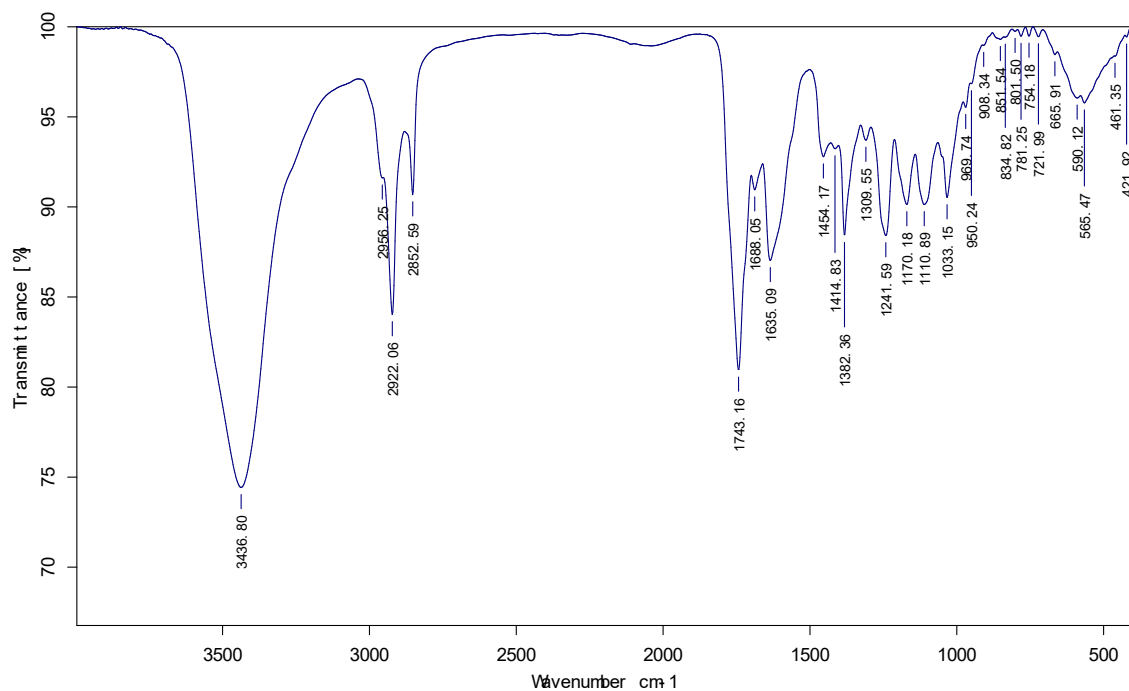


Figure. S38 IR spectrum of artemongolide D (4)

Rudolph Research Analytical

This sample was measured on an Autopol VI, Serial #91058
Manufactured by Rudolph Research Analytical, Hackettstown, NJ, USA.

Measurement Date : Friday, 12-AUG-2022

Set Temperature : OFF

Time Delay : Disabled

Delay between Measurement : Disabled

<u>n</u>	<u>Average</u>	<u>Std.Dev.</u>	<u>% RSD</u>	<u>Maximum</u>	<u>Minimum</u>					
5	-51.00	0.26	-0.50	-50.67	-51.33					
<u>S.No</u>	<u>Sample ID</u>	<u>Time</u>	<u>Result</u>	<u>Scale</u>	<u>OR °Arc</u>	<u>WLG.nm</u>	<u>Lq.mm</u>	<u>Conc.g/100ml</u>	<u>Temp.</u>	
1	JSC-30	02:53:50 PM	-50.83	SR	-0.0305	589	100.00	0.060	25.0	
2	JSC-30	02:53:59 PM	-51.33	SR	-0.0308	589	100.00	0.060	25.0	
3	JSC-30	02:54:07 PM	-50.67	SR	-0.0304	589	100.00	0.060	25.0	
4	JSC-30	02:54:16 PM	-51.17	SR	-0.0307	589	100.00	0.060	25.0	
5	JSC-30	02:54:23 PM	-51.00	SR	-0.0306	589	100.00	0.060	25.0	

Figure. S39 Optical rotation spectrum of artemongolide D (4)

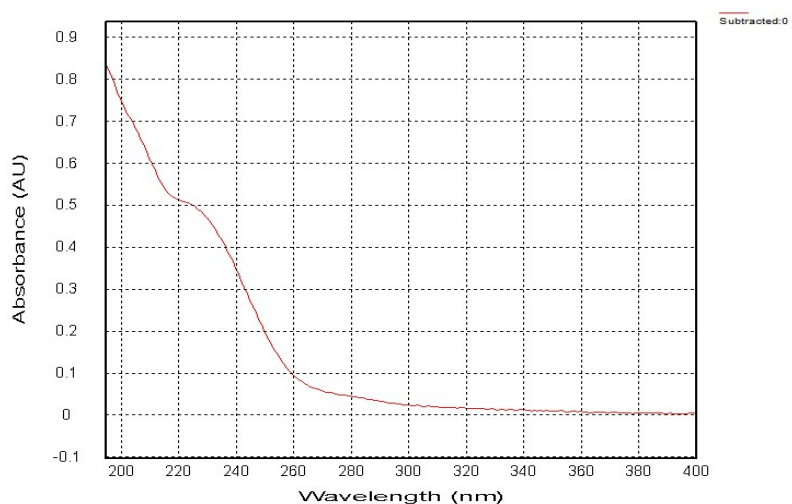
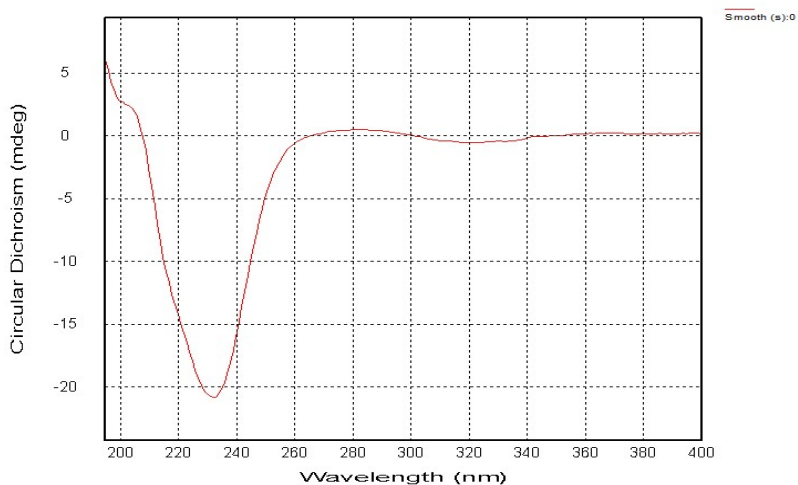
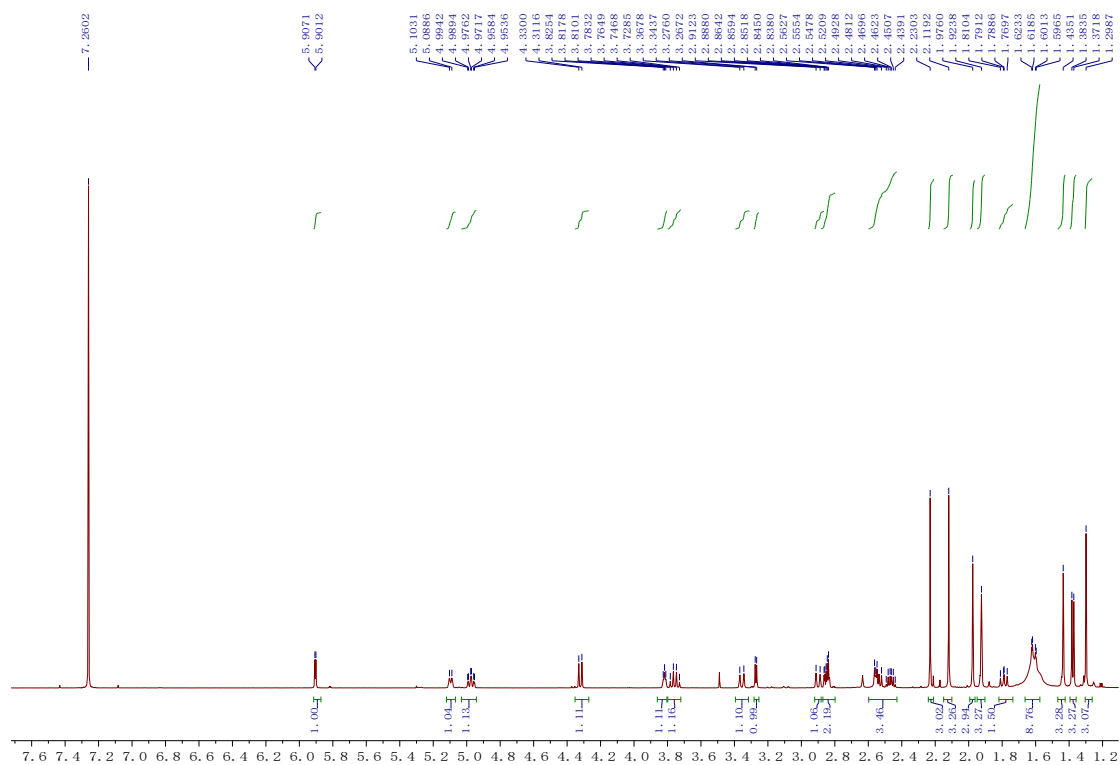
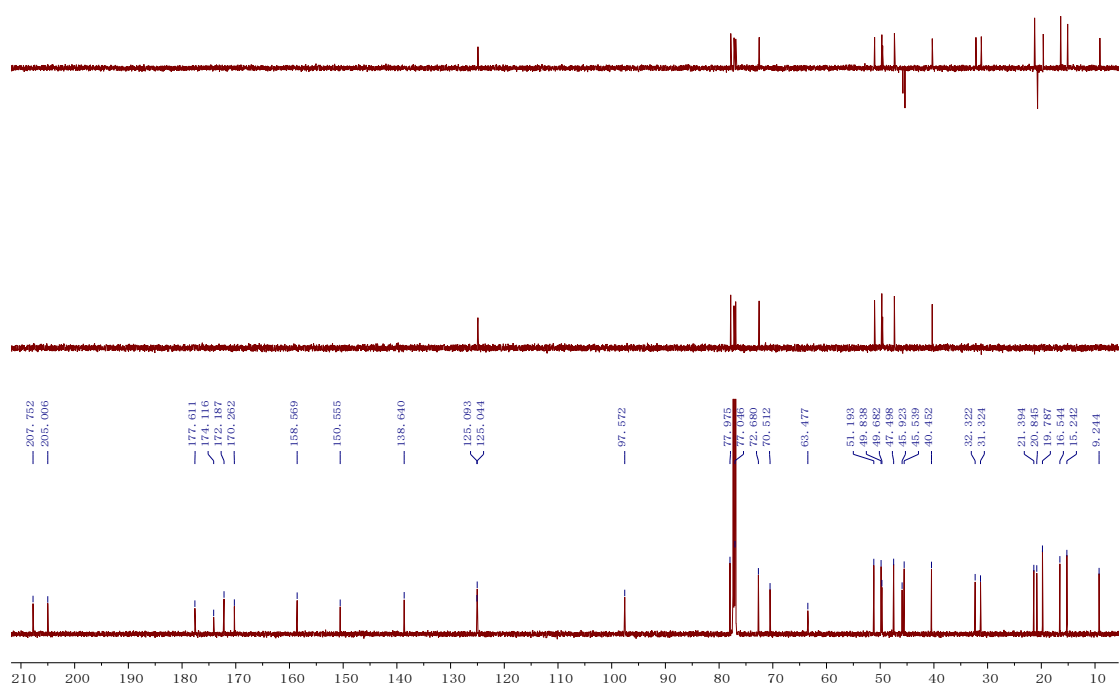


Figure. S40 CD (top) and UV (bottom) spectrums of artemongolide D (4)

5. NMR, MS, IR, $[\alpha]$ and CD spectra of compound 5Figure. S41 ^1H NMR spectrum (600 MHz) of artemongolide E (5) in CDCl_3 Figure. S42 ^{13}C NMR spectrum (150 MHz) of artemongolide E (5) in CDCl_3

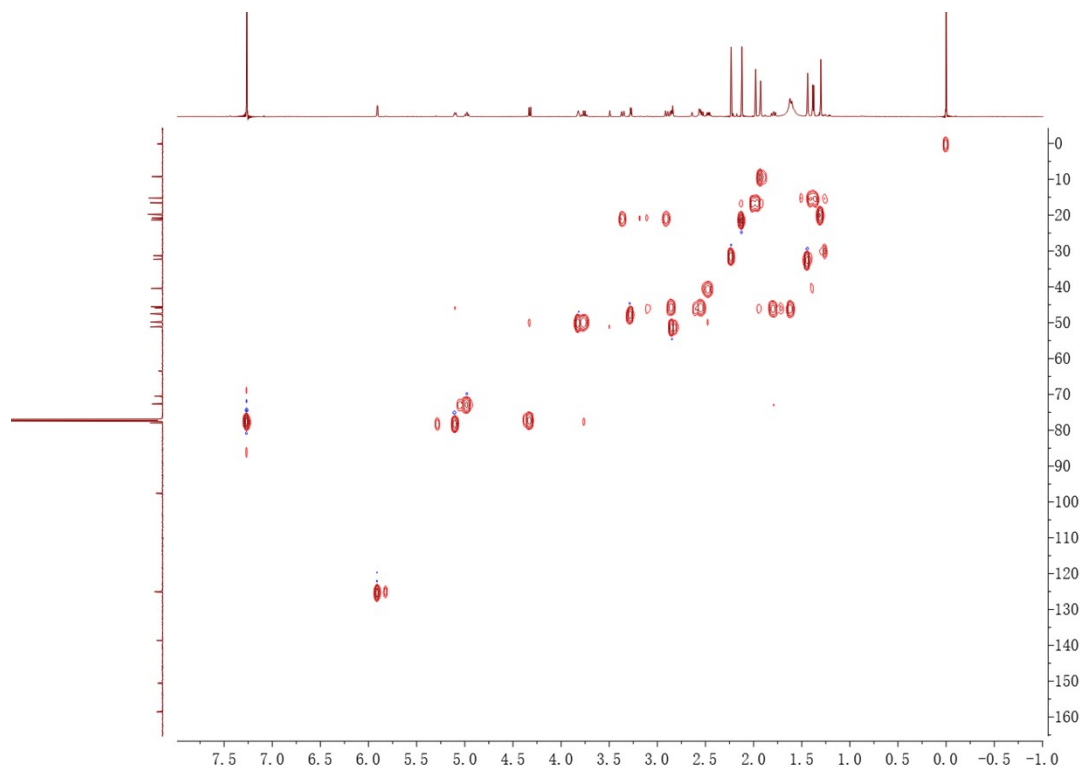


Figure. S43 HSQC spectrum (600 MHz) of artemongolide E (5) in CDCl_3

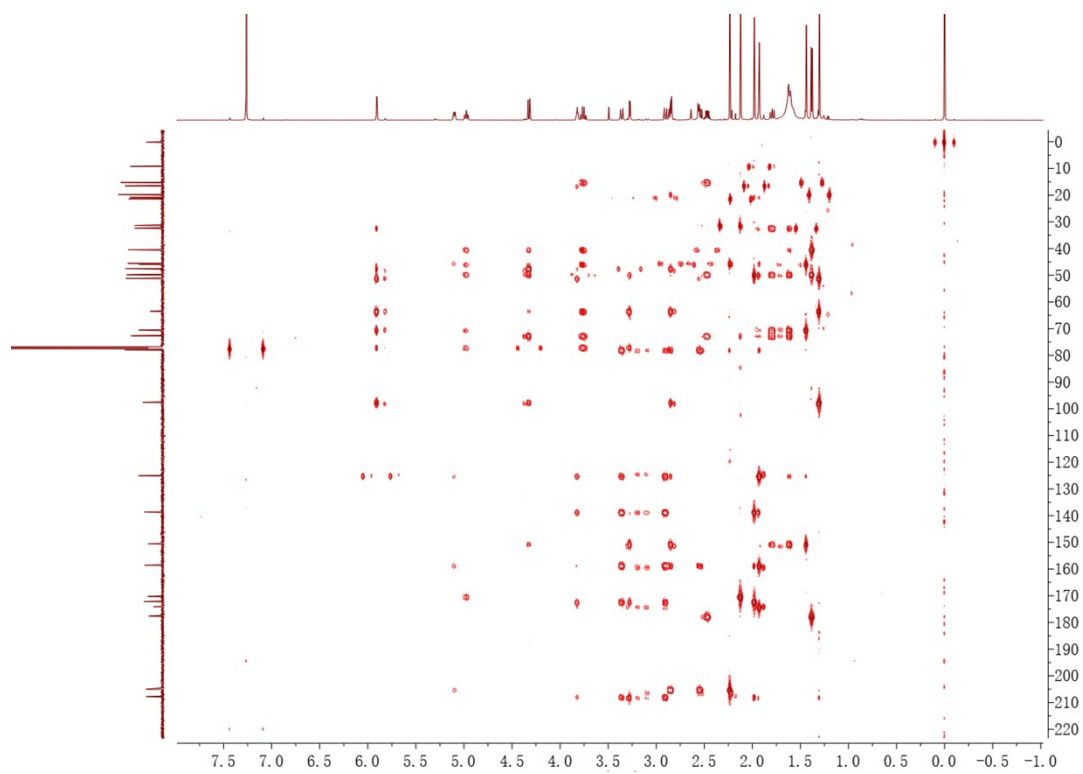


Figure. S44 HMBC spectrum (600 MHz) of artemongolide E (5) in CDCl_3

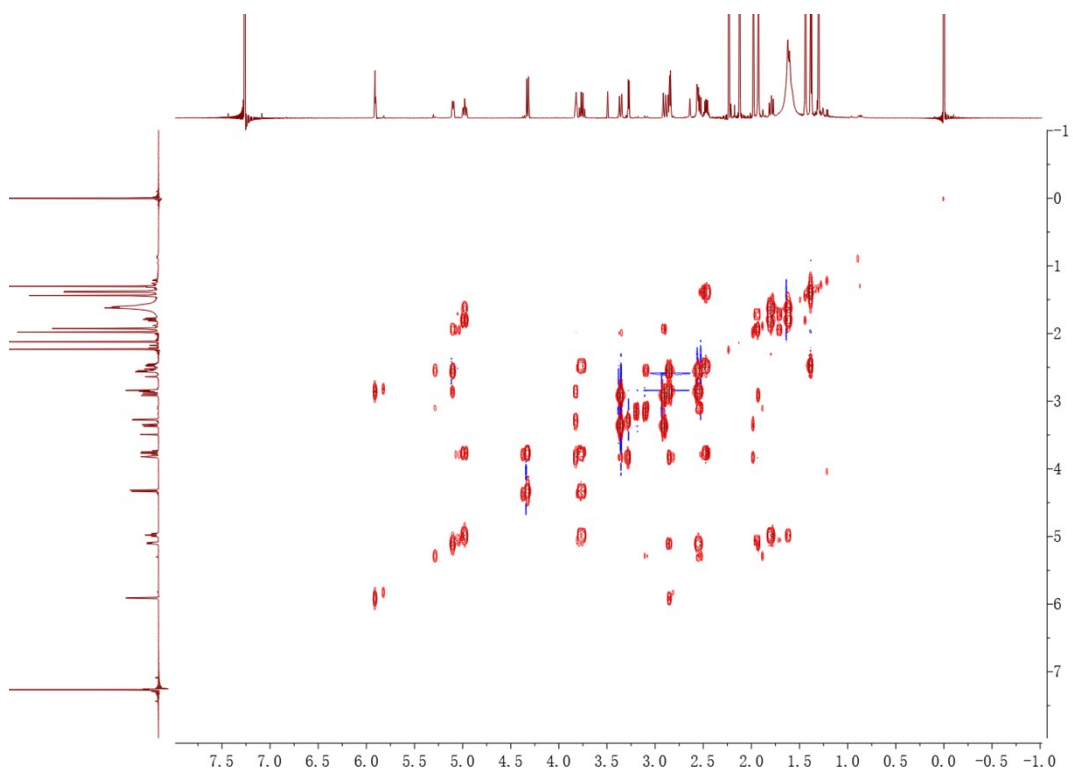


Figure. S45 ^1H - ^1H COSY spectrum (600 MHz) of artemongolide E (5) in CDCl_3

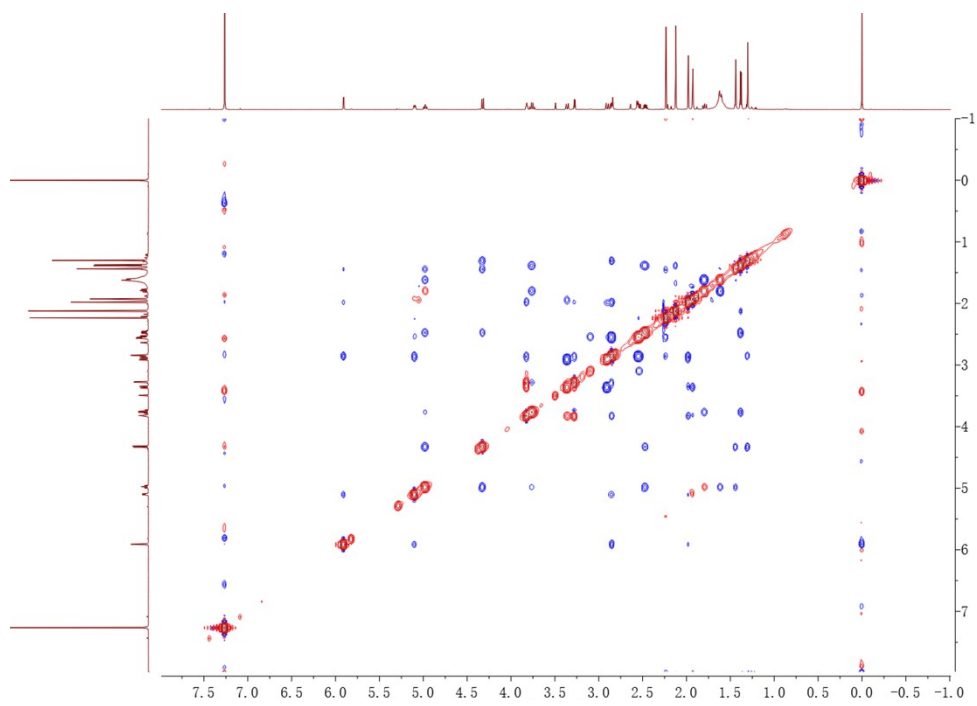


Figure. S46 ROESY spectrum (600 MHz) of artemongolide E (5) in CDCl_3

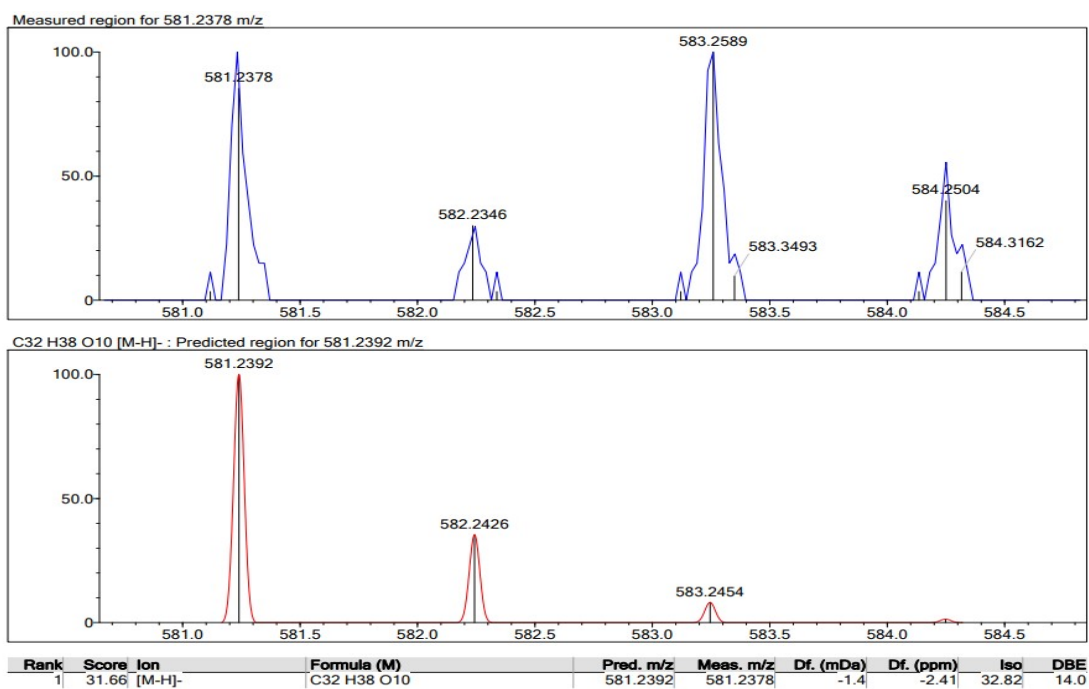


Figure. S47 HRESIMS spectrum of artemongolide E (5)

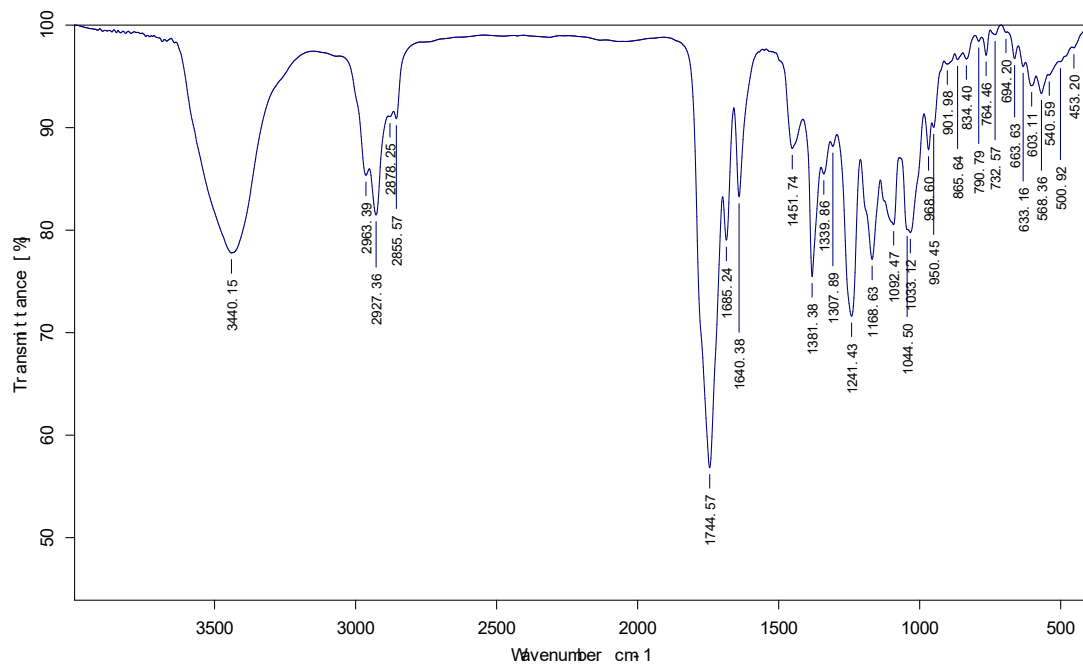


Figure. S48 IR spectrum of artemongolide E (5)

Rudolph Research Analytical

This sample was measured on an Autopol VI, Serial #91058
Manufactured by Rudolph Research Analytical, Hackettstown, NJ, USA.

Measurement Date : Thursday, 22-SEP-2022

Set Temperature : OFF

Time Delay : Disabled

Delay between Measurement : Disabled

<u>n</u>	<u>Average</u>	<u>Std.Dev.</u>	<u>% RSD</u>	<u>Maximum</u>	<u>Minimum</u>				
5	-66.67	0.39	-0.58	-66.33	-67.17				
<u>S.No</u>	<u>Sample ID</u>	<u>Time</u>	<u>Result</u>	<u>Scale</u>	<u>OR °Arc</u>	<u>WLG.nm</u>	<u>Lq.mm</u>	<u>Conc.g/100ml</u>	<u>Temp.</u>
1	JSC-22	04:26:08 PM	-66.33	SR	-0.0398	589	100.00	0.060	25.2
2	JSC-22	04:26:16 PM	-67.00	SR	-0.0402	589	100.00	0.060	25.2
3	JSC-22	04:26:24 PM	-67.17	SR	-0.0403	589	100.00	0.060	25.2
4	JSC-22	04:26:33 PM	-66.33	SR	-0.0398	589	100.00	0.060	25.2
5	JSC-22	04:26:41 PM	-66.50	SR	-0.0399	589	100.00	0.060	25.2

Figure. S49 Optical rotation spectrum of artemongolide E (5)

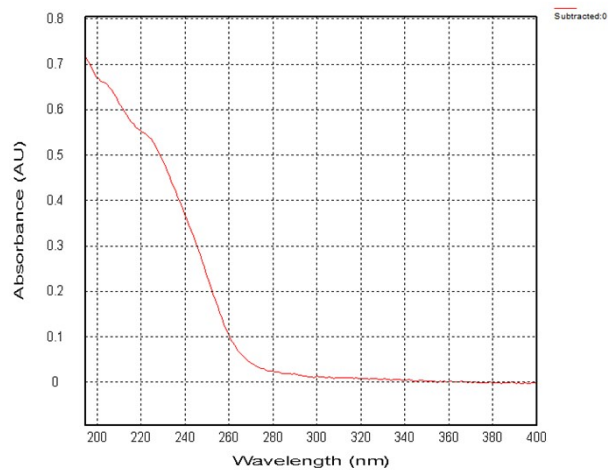
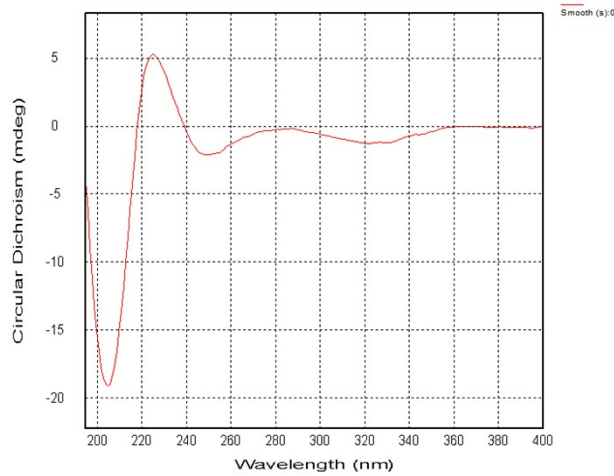
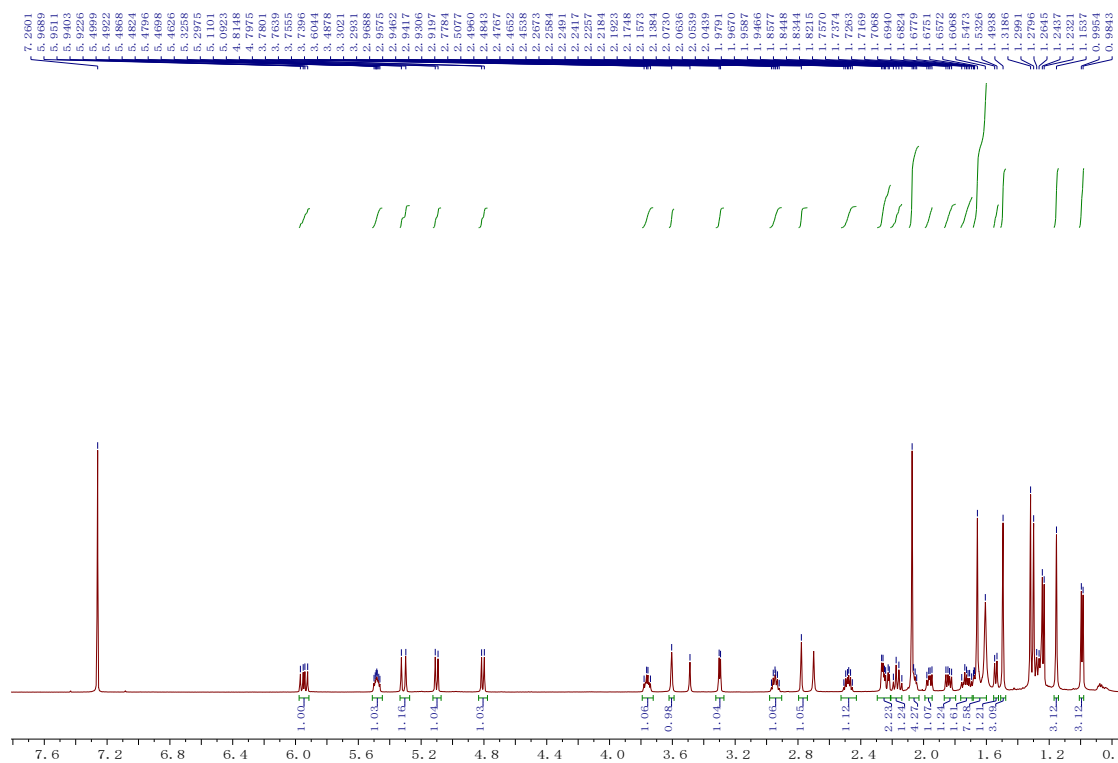
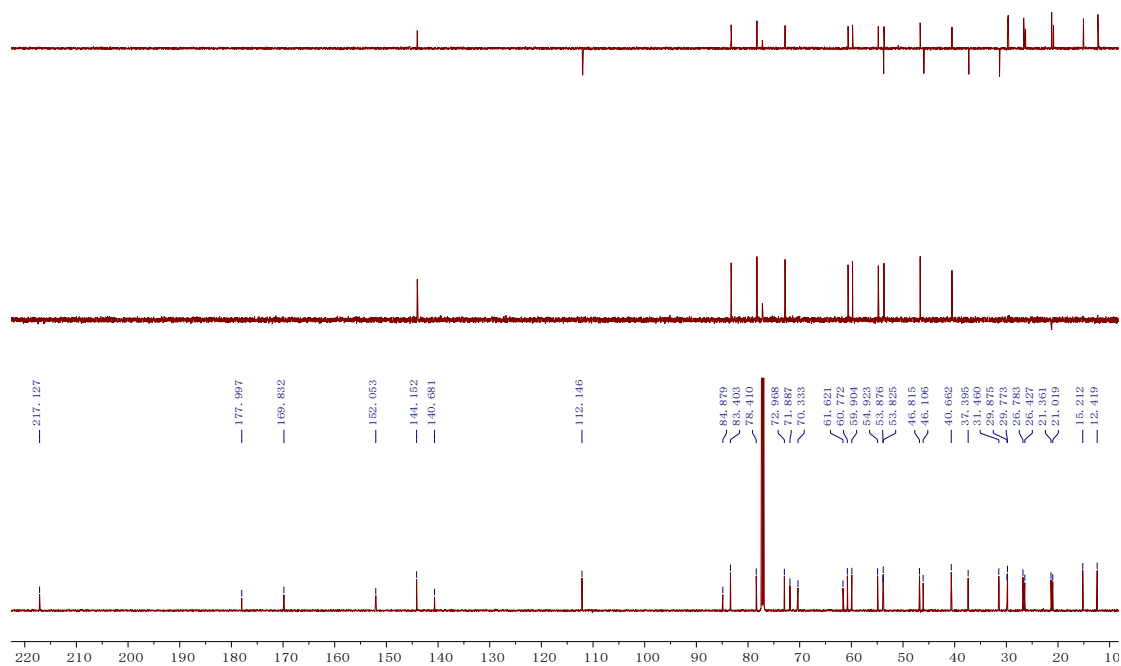


Figure. S50 CD (top) and UV (bottom) spectrums of artemongolide E (5)

6. NMR, MS, IR, $[\alpha]$ and CD spectra of compound 6Figure. S51 ^1H NMR spectrum (600 MHz) of artemongolide F (6) in CDCl_3 Figure. S52 ^{13}C NMR spectrum (150 MHz) of artemongolide F (6) in CDCl_3

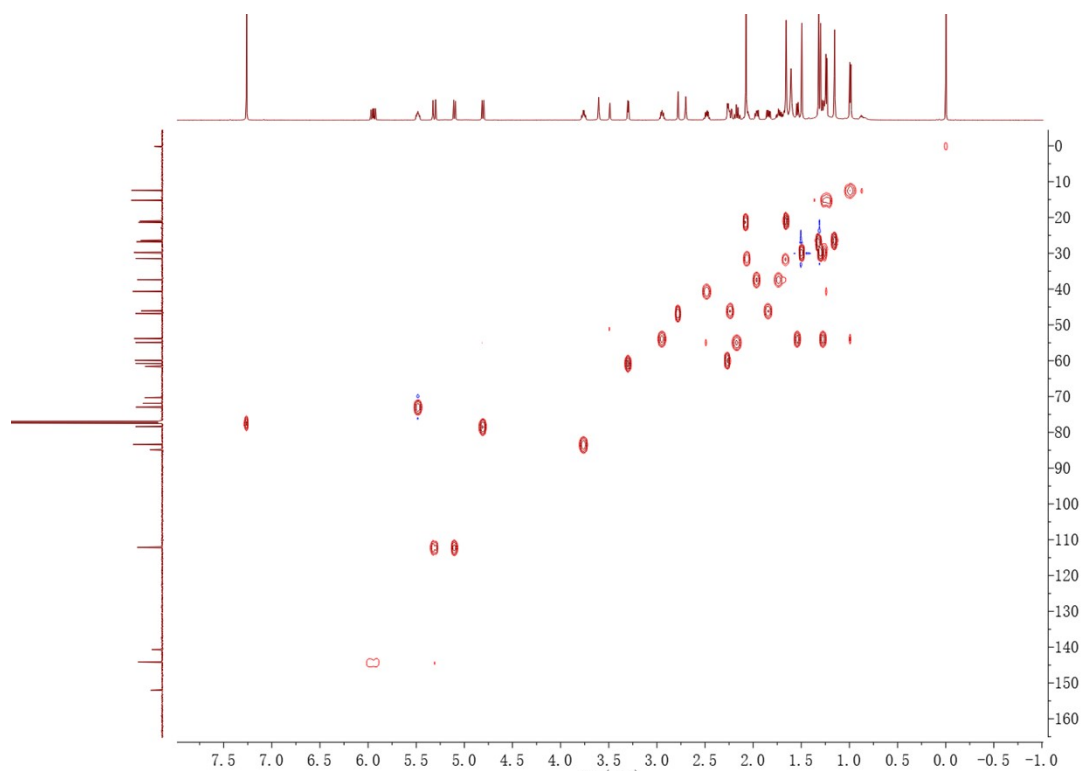


Figure. S53 HSQC spectrum (600 MHz) of artemongolide F (6) in CDCl₃

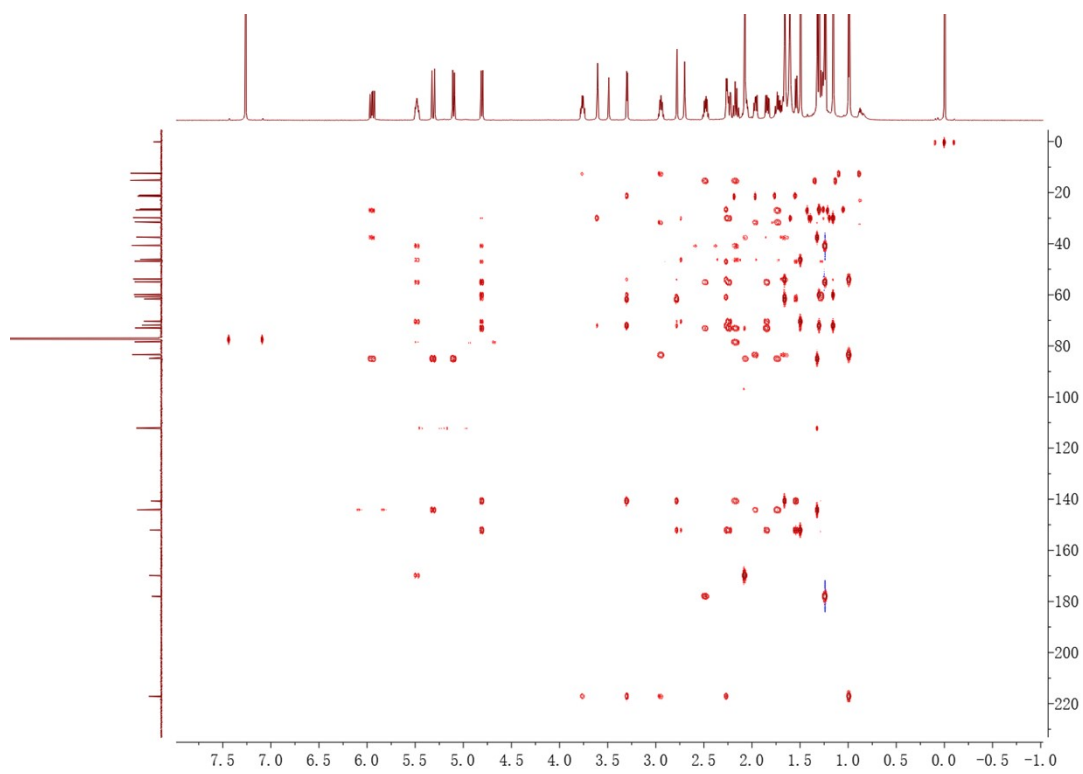


Figure. S54 HMBC spectrum (600 MHz) of artemongolide F (6) in CDCl₃

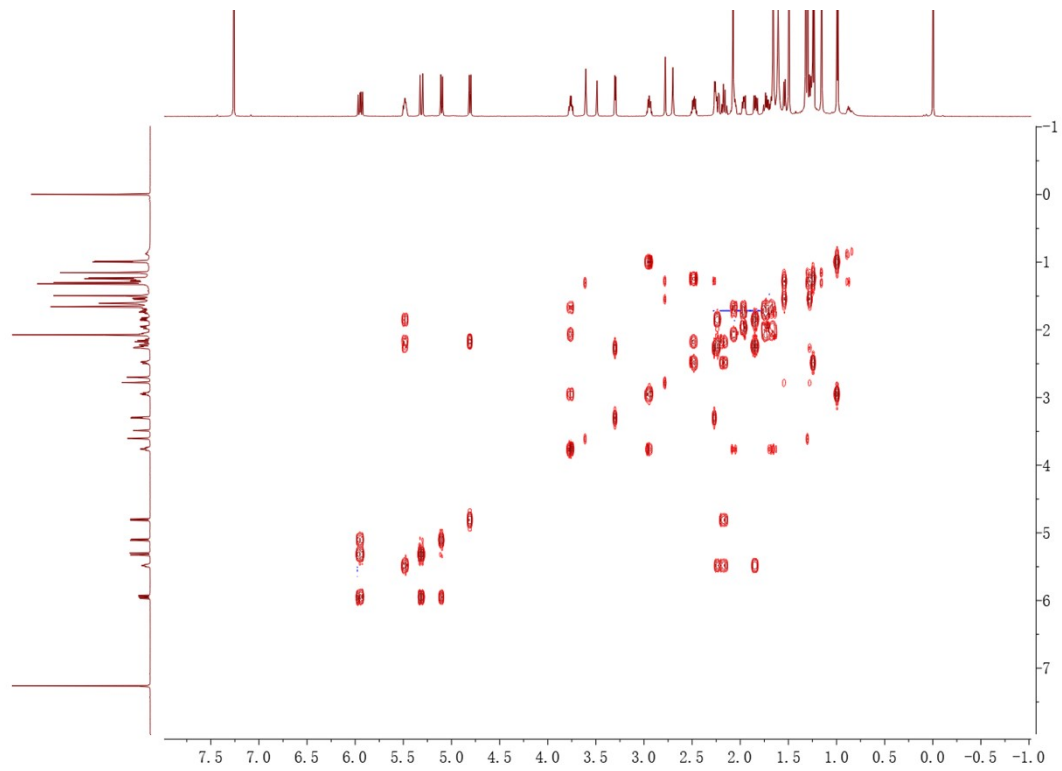


Figure. S55 ^1H - ^1H COSY spectrum (600 MHz) of artemongolide F (6) in

CDCl_3

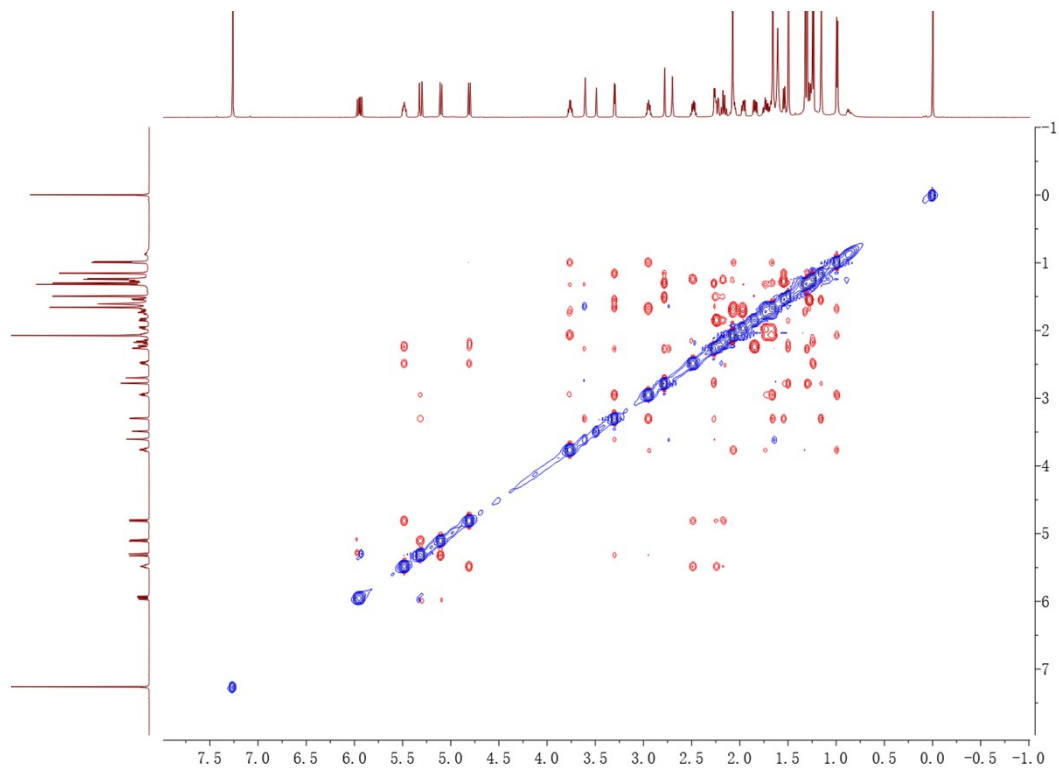


Figure. S56 ROESY spectrum (600 MHz) of artemongolide F (6) in CDCl_3

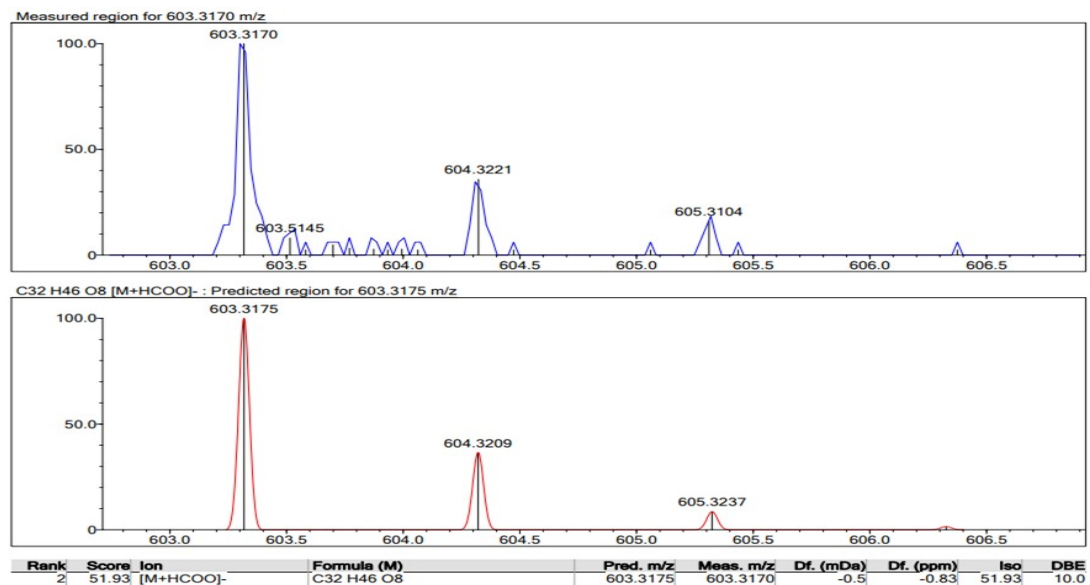


Figure. S57 HRESIMS spectrum of artemongolide F (6)

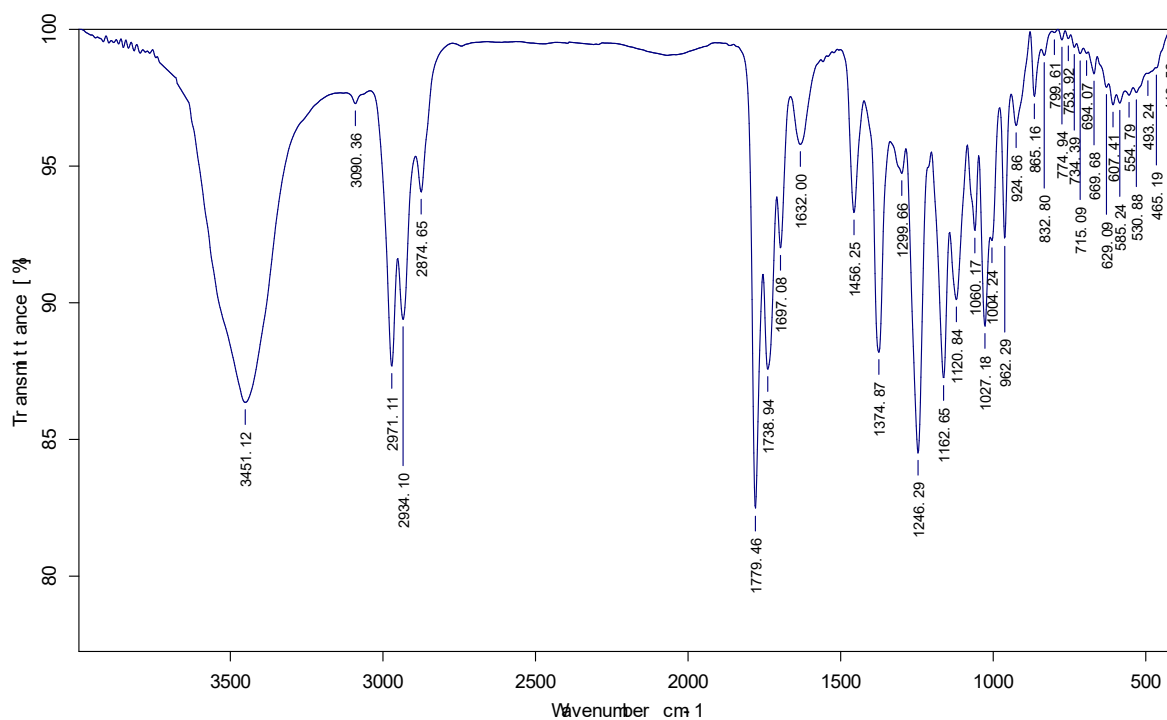


Figure. S58 IR spectrum of artemongolide F (6)

Rudolph Research Analytical

This sample was measured on an Autopol VI, Serial #91058
Manufactured by Rudolph Research Analytical, Hackettstown, NJ, USA.

Measurement Date : Thursday, 22-SEP-2022

Set Temperature : OFF

Time Delay : Disabled

Delay between Measurement : Disabled

<u>n</u>	<u>Average</u>	<u>Std.Dev.</u>	<u>% RSD</u>	<u>Maximum</u>	<u>Minimum</u>					
5	58.04	0.46	0.79	58.60	57.60					
<u>S.No</u>	<u>Sample ID</u>	<u>Time</u>	<u>Result</u>	<u>Scale</u>	<u>OR °Arc</u>	<u>WLG.nm</u>	<u>Lg.mm</u>	<u>Conc.g/100ml</u>	<u>Temp.</u>	
1	JSC-57	04:33:07 PM	58.60	SR	0.0293	589	100.00	0.050	25.1	
2	JSC-57	04:33:15 PM	57.60	SR	0.0288	589	100.00	0.050	25.1	
3	JSC-57	04:33:24 PM	57.60	SR	0.0288	589	100.00	0.050	25.0	
4	JSC-57	04:33:32 PM	58.00	SR	0.0290	589	100.00	0.050	25.0	
5	JSC-57	04:33:40 PM	58.40	SR	0.0292	589	100.00	0.050	25.0	

Figure. S59 Optical rotation spectrum of artemongolide F (6)

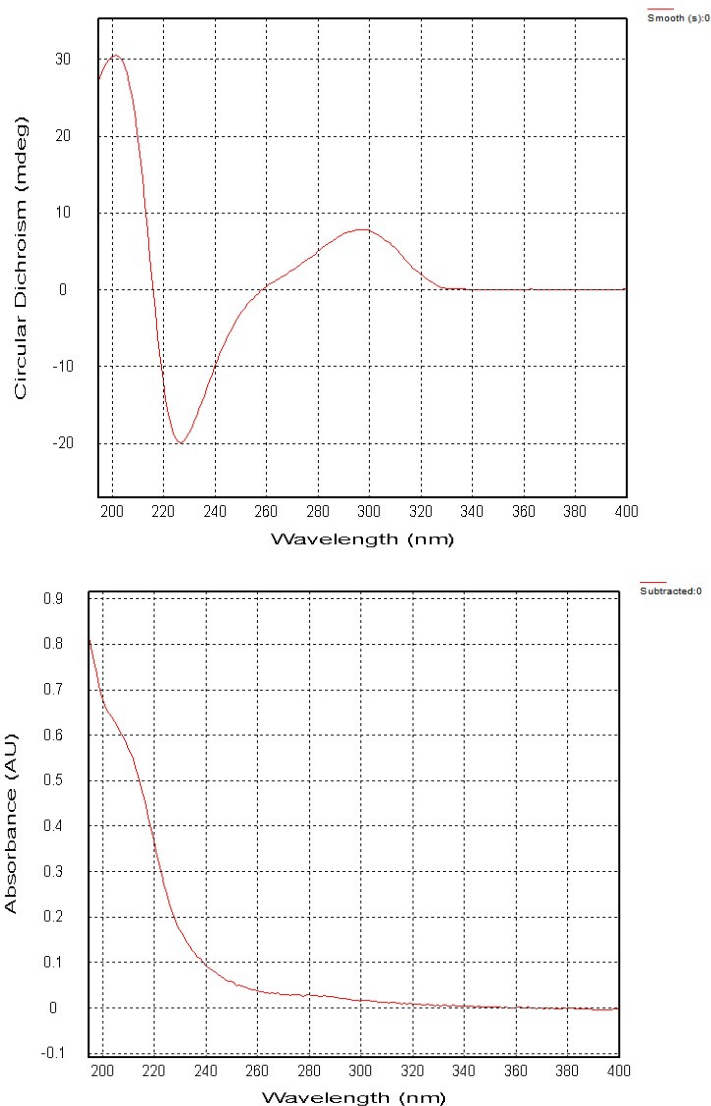


Figure. S60 CD (top) and UV (bottom) spectrums of artemongolide F (6)

7. General experimental procedures

Optical rotations were measured on Autopol VI polarimeter (Rudolph Research Analytical, Hackettstown, NJ, USA). IR (KBr) spectra were collected on a Nicolet iS10 spectrometer (Thermo Fisher Scientific, Madison, WI, USA). LC-MS-IT-TOF mass spectrometer was used for high-resolution mass spectra (Shimadzu, Kyoto, Japan). The ECD spectra were recorded on a Chirascan CD spectrometer (Applied Photophysics Ltd., Leatherhead, UK). NMR data was obtained on Avance III 600 (Bruker, Faellanden, Switzerland). TLC analyses were performed on silica gel HSGF254 plates (Yantai Xinnuo Silica Gel Development Co., Ltd., Yantai, China). Column chromatography was performed with silica gel (200–300 mesh, Yantai xinnuo Co., Ltd., Yantai, China), CHP 20P MCI gel (75–150 μm , Mitsubishi Chemical Corporation, Tokyo, Japan), RP-C18 silica gel (75–150 μm , Fuji Silysia Chemical Ltd, USA), and Sephadex LH-20 (GE Healthcare Amersham Biosciences, Uppsala, Sweden). Medium pressure liquid chromatography (MPLC) separations were carried out on a LC3050N apparatus (Beijing Tong Heng Innovation Technology Co., Ltd, Beijing, China). Semipreparative HPLC purification was performed by Shimadzu LC-CBM-20 liquid chromatography (Shimadzu, Kyoto, Japan) with an Agilent Eclipse XDB-C18 column (5 μm , 9.4 \times 250 mm, Agilent Technologies, Santa Clara, CA, USA).

8. ECD calculations for compounds 2–5

The relative configurations of compounds 2–5 were established according to their ROESY experiments and optimized by DFT calculation at the B3LYP/6-31G(d,p) level in the gas phase. To exclude imaginary frequencies, frequency calculations were performed at the same level. ECD calculations were performed using the TDDFT methodology at the B3LYP/6-311 + g(d,p) level with the consideration of solvent effects. ECD calculations were performed using the Gaussian 09 program package. The ECD curves were drawn using the Origin Pro 9 program (OriginLab Corporation, Northampton, MA, USA).

9. Cytotoxicity assay

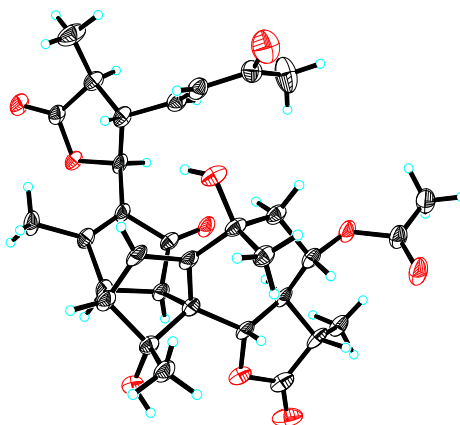
The cytotoxicity assay of compounds was conducted by referring to our previous papers. Briefly, the cytotoxicity of fractions and compounds were measured using the MTT method. HSC-LX2 were cultured in the RPMI 1640 medium containing 10 % fetal bovine serum at 37 °C with 5 % CO₂. Cells were cultured in 96-well plates (1×10^4 cells/well) for 24 h. Fractions or compounds with different concentrations were added to the cells and incubated for 48 h. After that, 100 μ L of MTT reagent (1 mg/mL) was added into each well and co-incubated for 4 h at 37 °C. Then the solution was removed and 100 μ L of DMSO was added to dissolve the formazan crystals. The absorbance was tested using a microplate reader at 490 nm. All the experiments were performed in triplicate. The inhibitory ratios were calculated as $[A(\text{control}) - A(\text{sample})] / A(\text{control}) \times 100 \%$, and IC₅₀ values were calculated by GraphPad Prism 5 (GraphPad Software, San Diego, CA, USA).

10. Col I, HA, and HL secretion assay

HSC-LX2 were seeded into 24-well plates at a density of 8×10^4 cells/well and cultured for 24 h, then the cells were treated with the compounds at different concentrations. After 72 h incubation with 5% CO₂ at 37 °C, the cell conditioned medium was collected and centrifuged for 15 min at $1000 \times g$ and 4 °C. The supernatant was obtained to detect the secretion of Col I, HA and HL using the commercial ELISA kits according to the manufacturer's protocol.

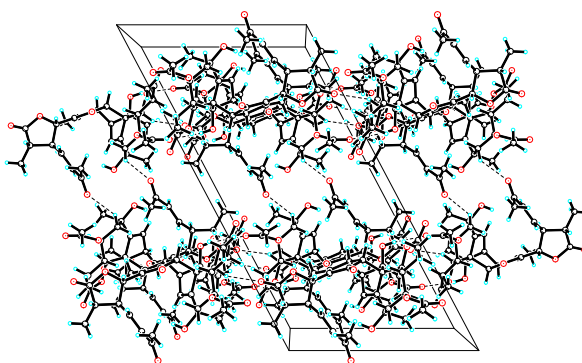
11. Crystal data for compound **1**

Crystal data for **1**: $C_{32}H_{38}O_{10}$, $M = 582.62$, $a = 23.9849(14)$ Å, $b = 14.4810(8)$ Å, $c = 12.3512(6)$ Å, $\alpha = 90^\circ$, $\beta = 117.884(3)^\circ$, $\gamma = 90^\circ$, $V = 3791.8(4)$ Å³, $T = 100.(2)$ K, space group $C121$, $Z = 4$, $\mu(\text{Cu K}\alpha) = 0.627$ mm⁻¹, 31598 reflections measured, 7097 independent reflections ($R_{int} = 0.1026$). The final R_I values were 0.0728 ($I > 2\sigma(I)$). The final $wR(F^2)$ values were 0.2117 ($I > 2\sigma(I)$). The final R_I values were 0.0898 (all data). The final $wR(F^2)$ values were 0.2296 (all data). The goodness of fit on F^2 was 1.072. Flack parameter = 0.03(11).



View of a molecule of **1** with the atom-labelling scheme.

Displacement ellipsoids are drawn at the 30% probability level.



View of the pack drawing of **1**.

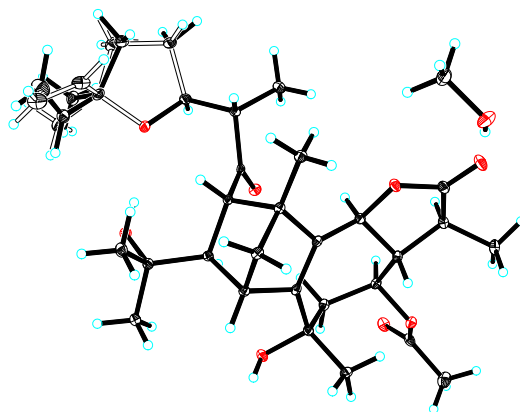
Hydrogen-bonds are shown as dashed lines.

Table 1. Crystal data and structure refinement for **1**

Identification code	global
Empirical formula	C ₃₂ H ₃₈ O ₁₀
Formula weight	582.62
Temperature	100(2) K
Wavelength	1.54178 Å
Crystal system	Monoclinic
Space group	C 1 2 1
Unit cell dimensions	a = 23.9849(14) Å $\alpha = 90^\circ$ b = 14.4810(8) Å $\beta = 117.884(3)^\circ$ c = 12.3512(6) Å $\gamma = 90^\circ$.
Volume	3791.8(4) Å ³
Z	4
Density (calculated)	1.021 Mg/m ³
Absorption coefficient	0.627 mm ⁻¹
F (000)	1240
Crystal size	0.540 x 0.200 x 0.080 mm ³
Theta range for data collection	3.70 to 70.38°
Index ranges	-29 ≤ h ≤ 27, -17 ≤ k ≤ 17, -12 ≤ l ≤ 14
Reflections collected	31598
Independent reflections	7097 [R(int) = 0.1026]
Completeness to theta = 70.38°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.95 and 0.75
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	7097 / 1 / 388
Goodness-of-fit on F ²	1.072
Final R indices [I > 2σ(I)]	R1 = 0.0728, wR2 = 0.2117
R indices (all data)	R1 = 0.0898, wR2 = 0.2296
Absolute structure parameter	0.03(11)
Largest diff. peak and hole	0.667 and -0.288 e.Å ⁻³

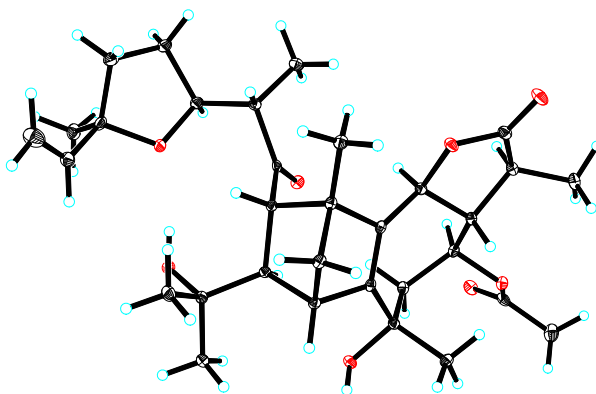
12. Crystal data for compound **6**

Crystal data for **6**: $C_{32}H_{46}O_8 \cdot CH_4O$, $M = 590.73$, $a = 9.7168(2) \text{ \AA}$, $b = 13.2364(3) \text{ \AA}$, $c = 24.3055(5) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 90^\circ$, $\gamma = 90^\circ$, $V = 3126.06(12) \text{ \AA}^3$, $T = 100.(2) \text{ K}$, space group $P212121$, $Z = 4$, $\mu(\text{Cu K}\alpha) = 0.734 \text{ mm}^{-1}$, 49783 reflections measured, 5918 independent reflections ($R_{int} = 0.0413$). The final R_I values were 0.0255 ($I > 2\sigma(I)$). The final $wR(F^2)$ values were 0.0651 ($I > 2\sigma(I)$). The final R_I values were 0.0257 (all data). The final $wR(F^2)$ values were 0.0652 (all data). The goodness of fit on F^2 was 1.035. Flack parameter = 0.00(3).



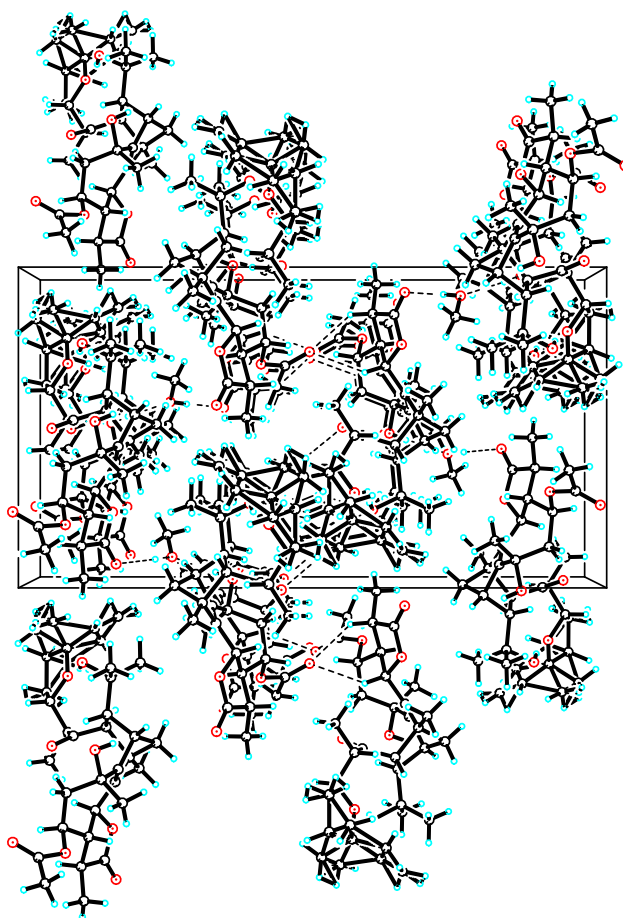
View of the molecules in an asymmetric unit.

Displacement ellipsoids are drawn at the 30% probability level.



View of a molecule of **6** with the atom-labelling scheme.

Displacement ellipsoids are drawn at the 30% probability level.



View of the pack drawing of **6**.

Hydrogen-bonds are shown as dashed lines.

Table 2. Crystal data and structure refinement for **6**.

Identification code	global
Empirical formula	C ₃₃ H ₅₀ O ₉
Formula weight	590.73
Temperature	100(2) K
Wavelength	1.54178 Å
Crystal system	Orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁
Unit cell dimensions	a = 9.7168(2) Å α = 90°. b = 13.2364(3) Å β = 90°. c = 24.3055(5) Å γ = 90°..
Volume	3126.06(12) Å ³
Z	4
Density (calculated)	1.255 Mg/m ³
Absorption coefficient	0.734 mm ⁻¹
F (000)	1280
Crystal size	0.350 x 0.180 x 0.150 mm ³
Theta range for data collection	3.64 to 70.15°.
Index ranges	-11 ≤ h ≤ 11, -16 ≤ k ≤ 16, -29 ≤ l ≤ 29
Reflections collected	49783
Independent reflections	5918 [R(int) = 0.0413]
Completeness to theta = 70.38°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.90 and 0.75
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	5918 / 10 / 429
Goodness-of-fit on F ²	1.035
Final R indices [I > 2σ(I)]	R1 = 0.0255, wR2 = 0.0651
R indices (all data)	R1 = 0.0257, wR2 = 0.0652
Absolute structure parameter	0.00(3)
Largest diff. peak and hole	0.228 and -0.153 e.Å ⁻³