Supplementary data

Isolation, structural elucidation, and bioactivity of novel cholestane derivatives from *Ypsilandra thibetica*

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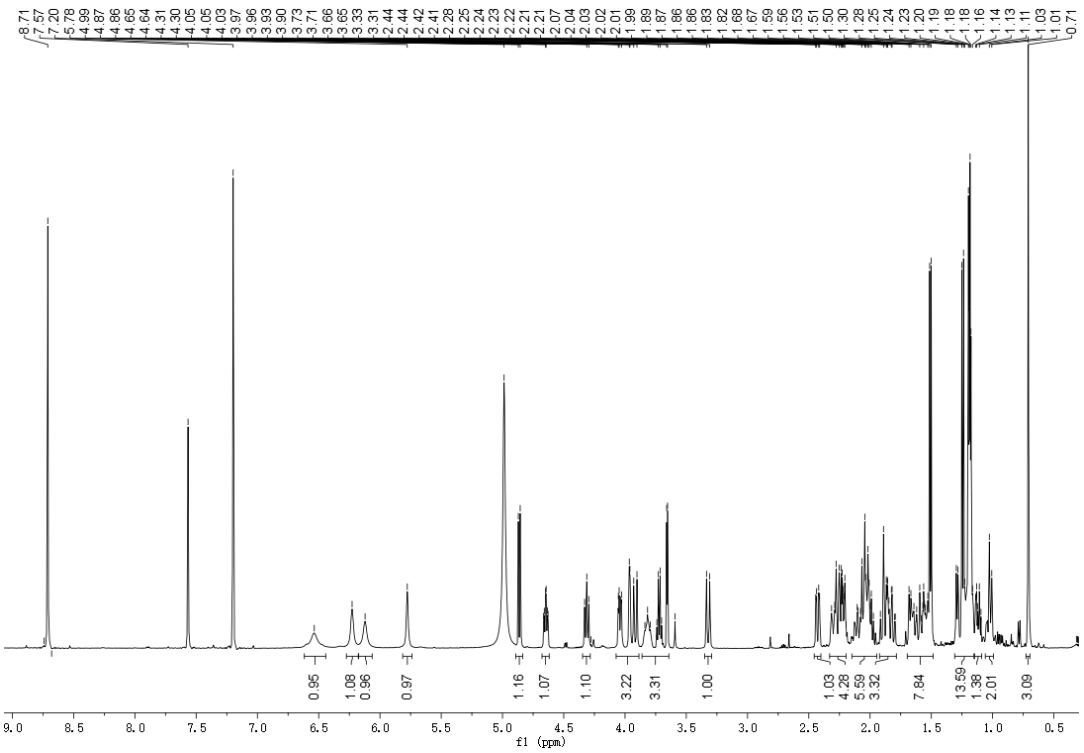


Fig. S1. ^{1}H NMR spectrum of compound **1** in pyridine-d_5.

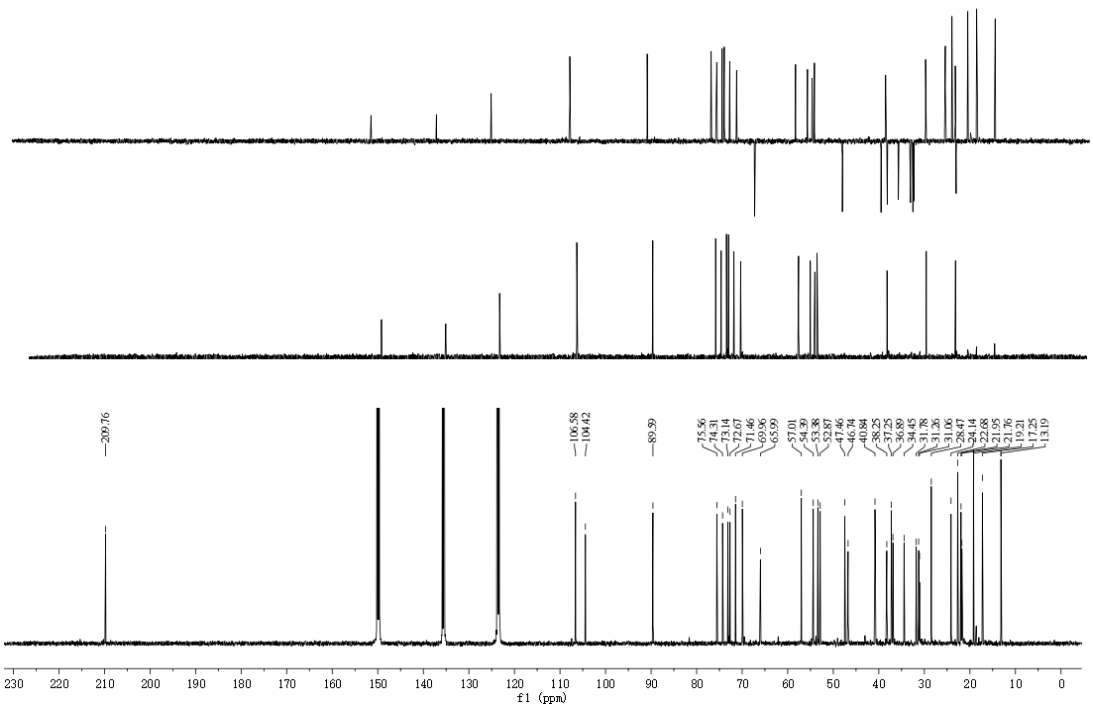


Fig. S2. ^{13}C NMR spectrum of compound **1** in pyridine-d_5.

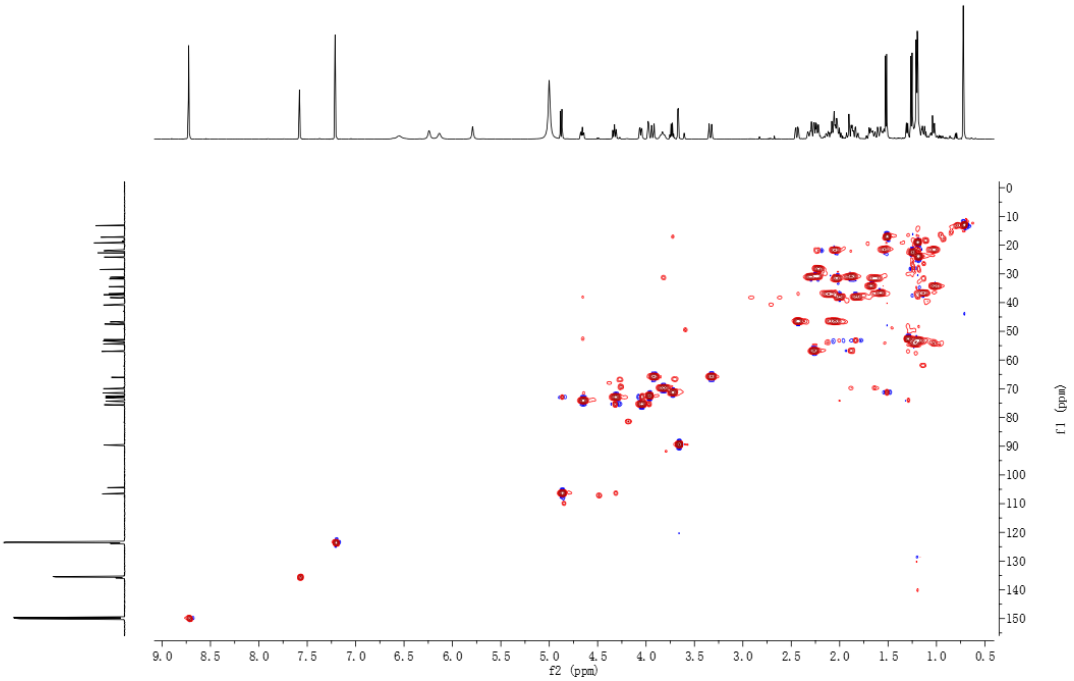


Fig. S3. HSQC spectrum of compound **1.**

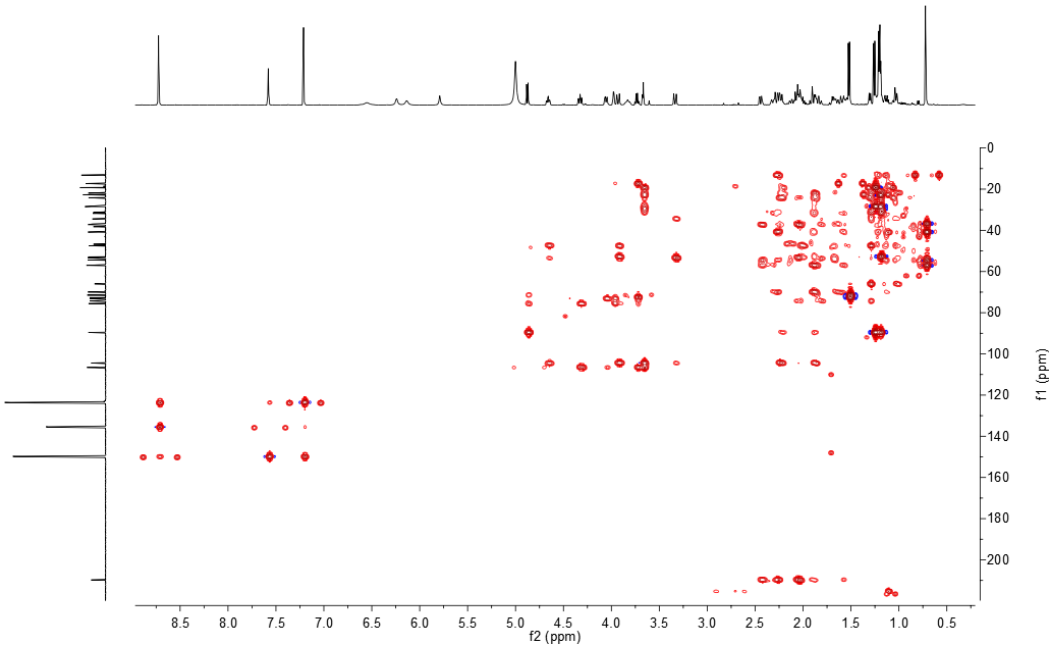


Fig. S4. HMBC spectrum of compound **1**.

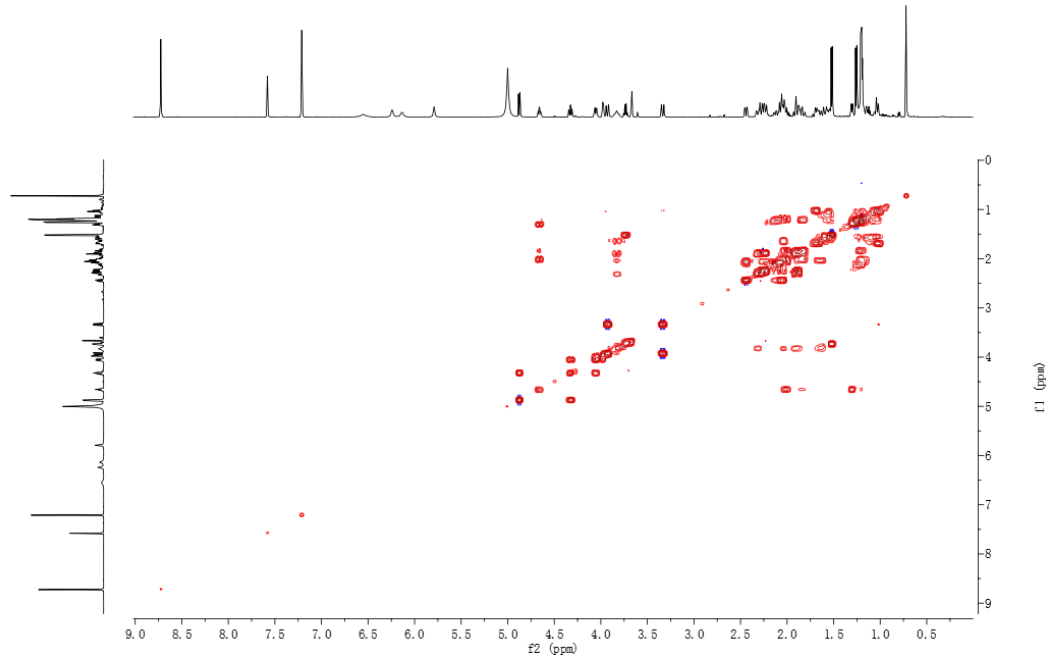


Fig. S5. $^{1}H–^{1}H$ COSY spectrum of compound **1**.

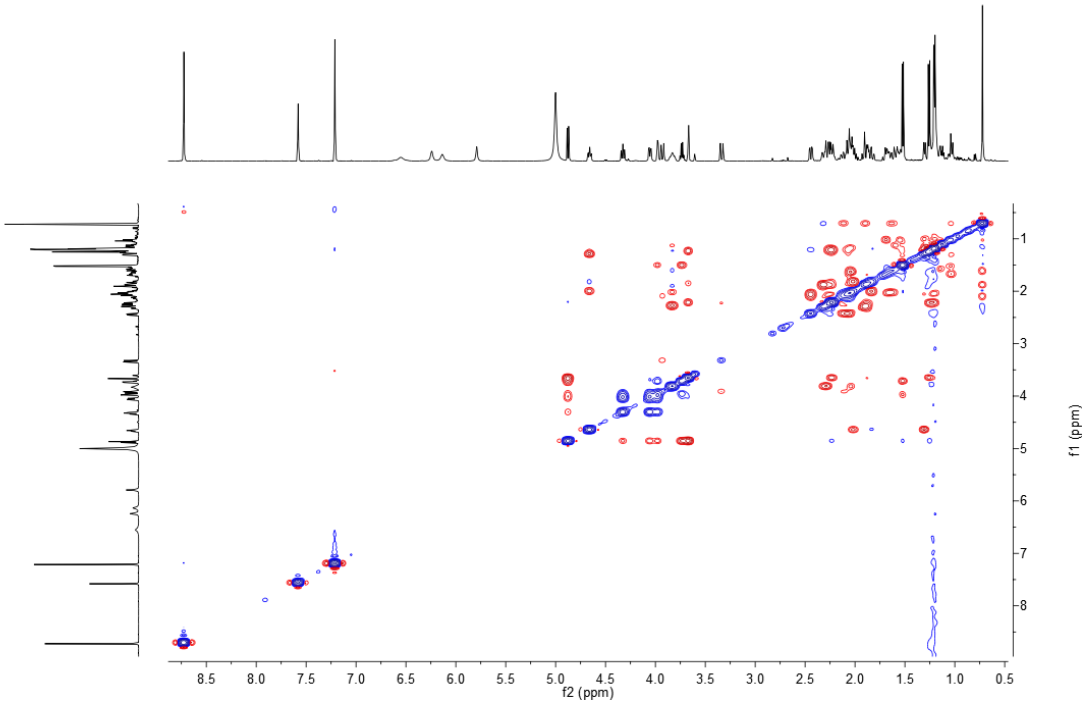
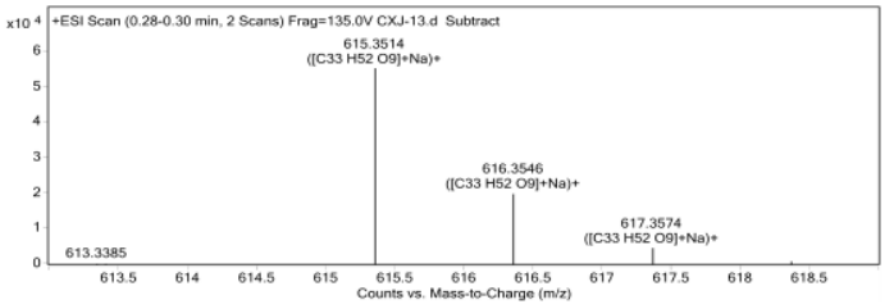


Fig. S6. ROESY spectrum of compound **1**.

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
135	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
429.3007	1	54649.6		
430.3041	1	15937.83		
610.3961	1	26778		
611.3995	1	9846.02		
615.3514	1	55217.32	C33 H52 O9	(M+Na)+
616.3546	1	20038.19	C33 H52 O9	(M+Na)+
617.3574	1	4630.79	C33 H52 O9	(M+Na)+
631.3257	1	12735.54		
1207.7134	1	11989.66		
1208.7162	1	9617.28		

Formula Calculator Element Limits

Element	Min	Max
C	3	60
H	0	120
O	0	30

Formula Calculator Results

Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C33 H52 O9	592.3611	615.3504	615.3514	-1.00	-1.63	8.0000

Fig. S7. HRESIMS spectrum of compound **1**.

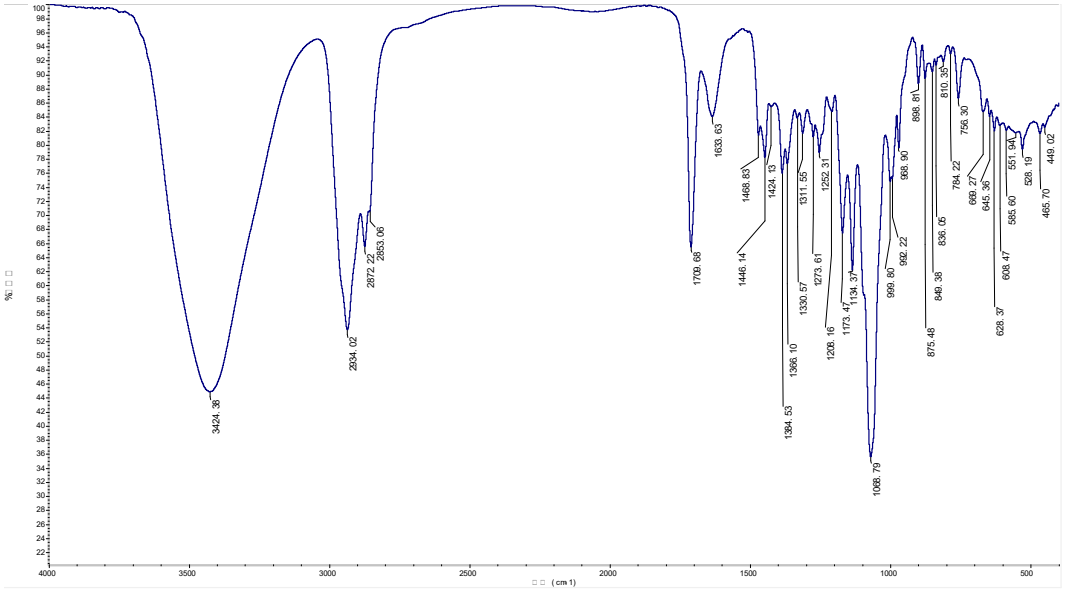


Fig. S8. IR spectrum of compound **1**.

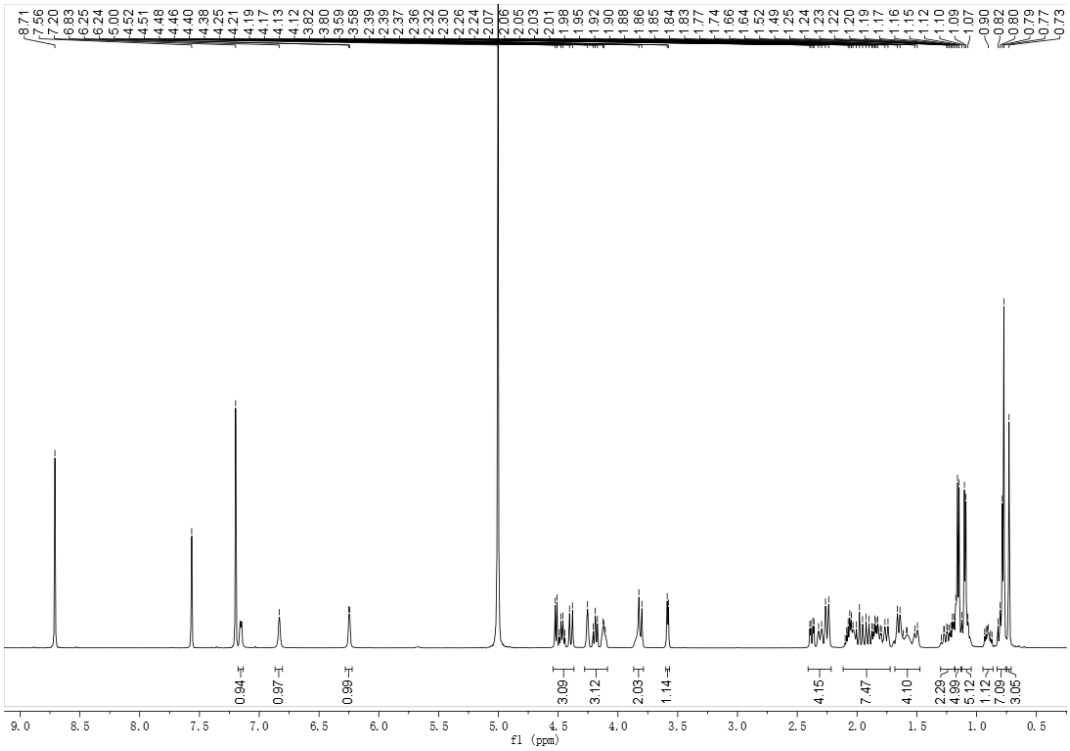


Fig. S9. ^{1}H NMR spectrum of compound **2** in pyridine-d_5.

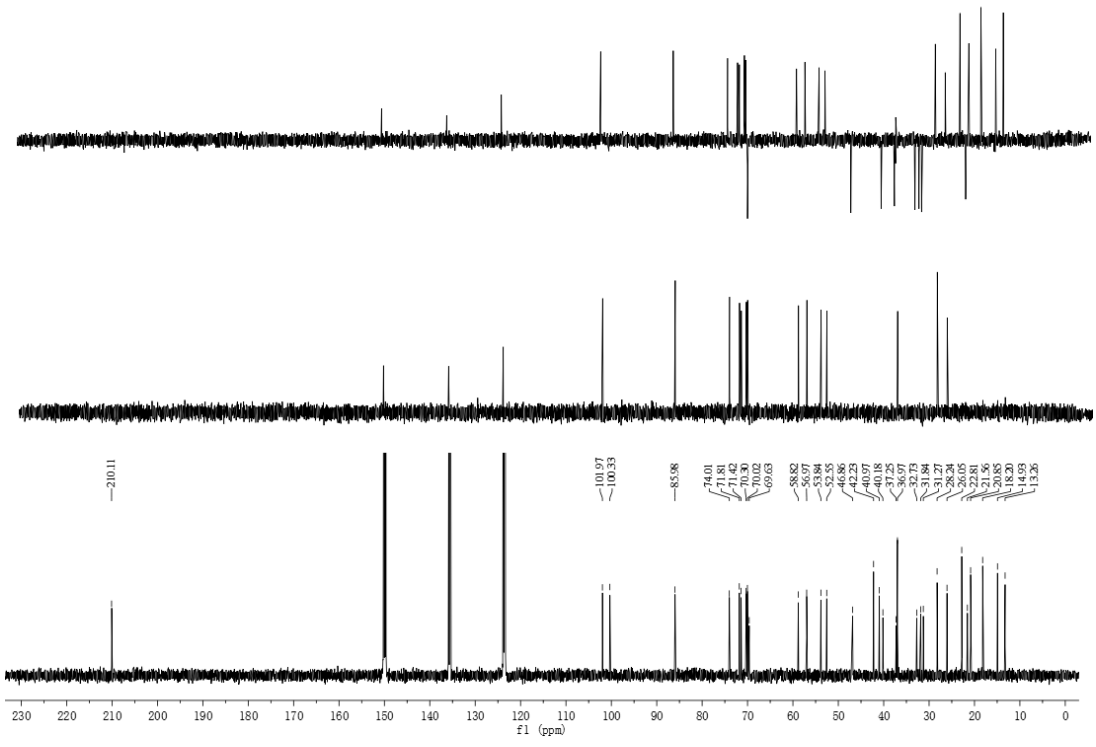


Fig. S10. ^{13}C NMR spectrum of compound **2** in pyridine-d_5.

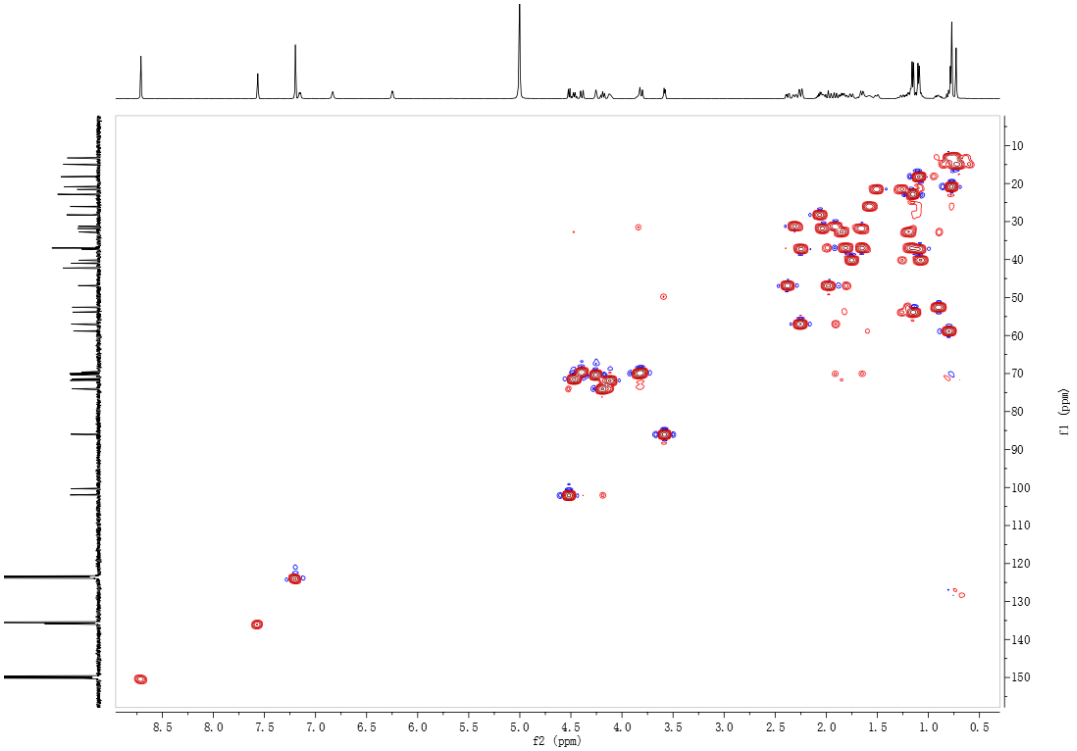


Fig. S11. HSQC spectrum of compound **2**.

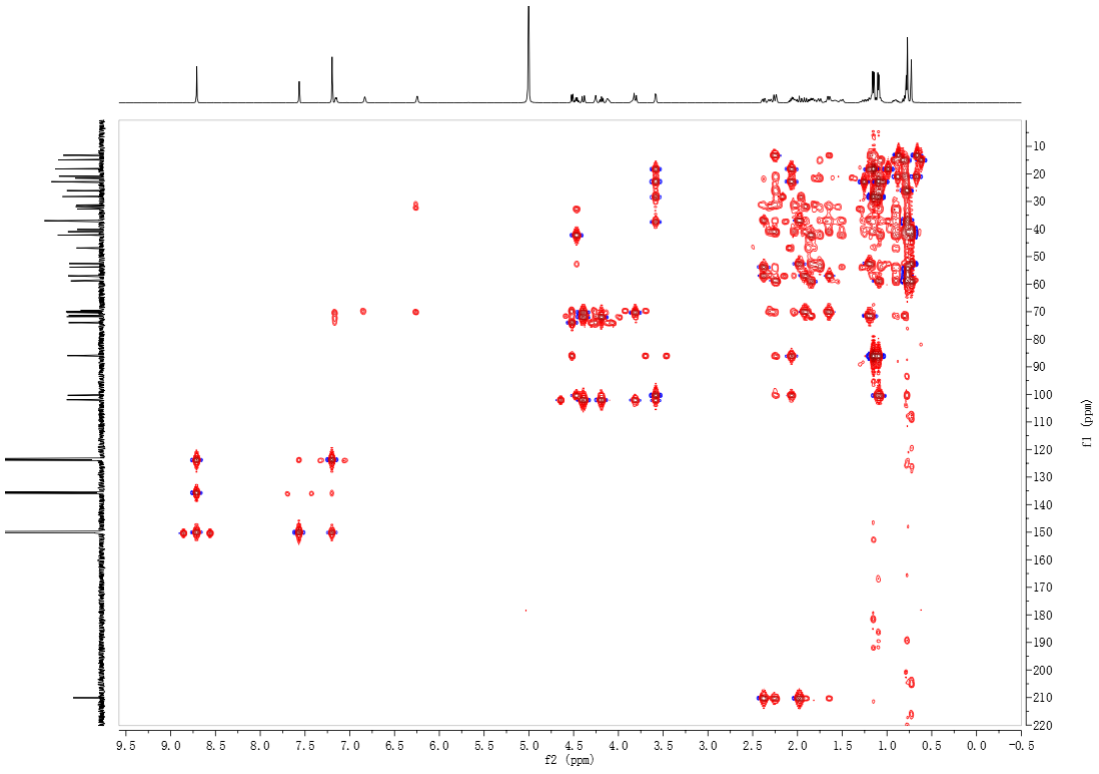


Fig. S12. HMBC spectrum of compound **2**.

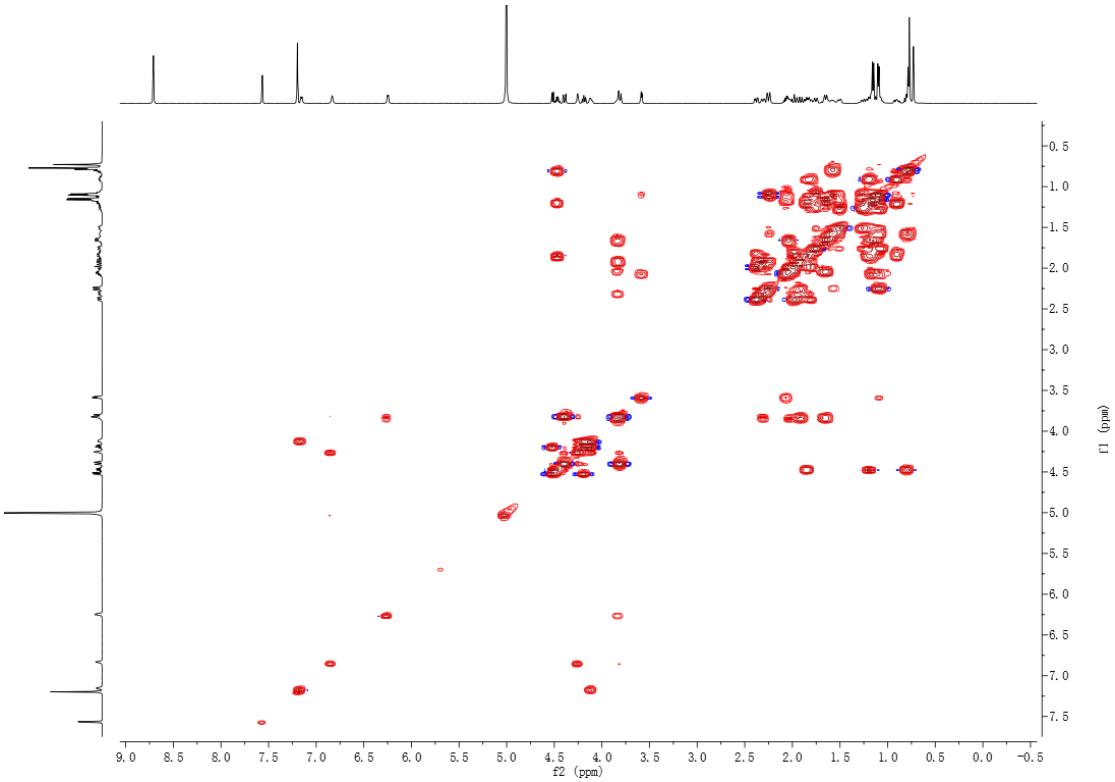


Fig. S13. $^{1}H–^{1}H$ COSY spectrum of compound **2**.

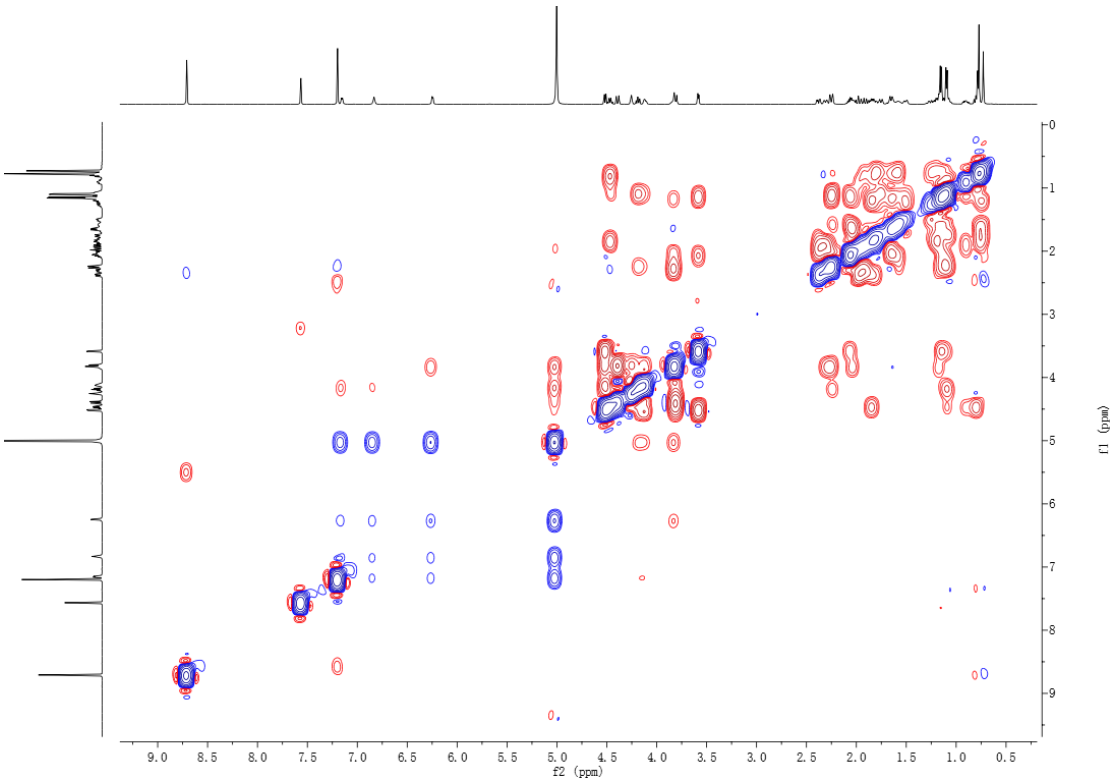
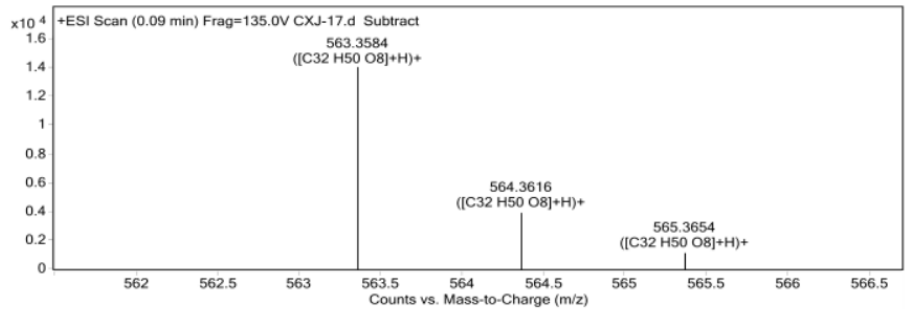


Fig. S14. ROESY spectrum of compound **2**.

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
135	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
61.0083		995.78		
89.5071	2	1549.17		
102.0544		767.07		
122.0807		785.94		
124.0867		2967.83		
413.3028	1	918.51		
563.3584	1	14097	C32 H50 O8	(M+H)+
564.3616	1	4000.51	C32 H50 O8	(M+H)+
565.3654	1	1201.51	C32 H50 O8	(M+H)+
585.3418		719.16		

Formula Calculator Element Limits

Element	Min	Max
C	3	60
H	0	120
O	0	30

Formula Calculator Results

Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C32 H50 O8	562.3506	563.3578	563.3584	-0.60	-1.07	8.0000

Fig. S15. HRESIMS spectrum of compound **2**.

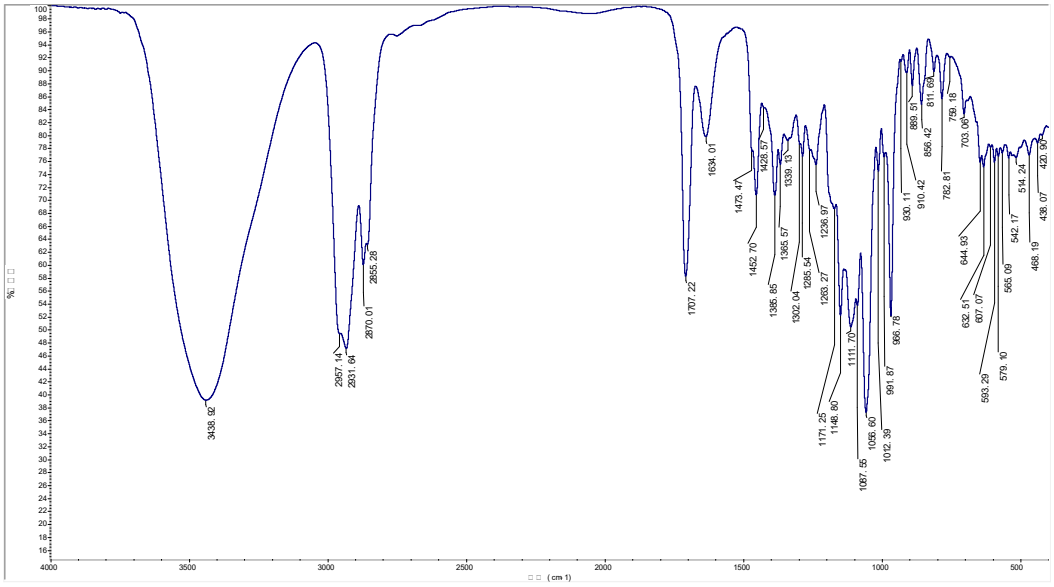


Fig. S16. IR spectrum of compound **2**.

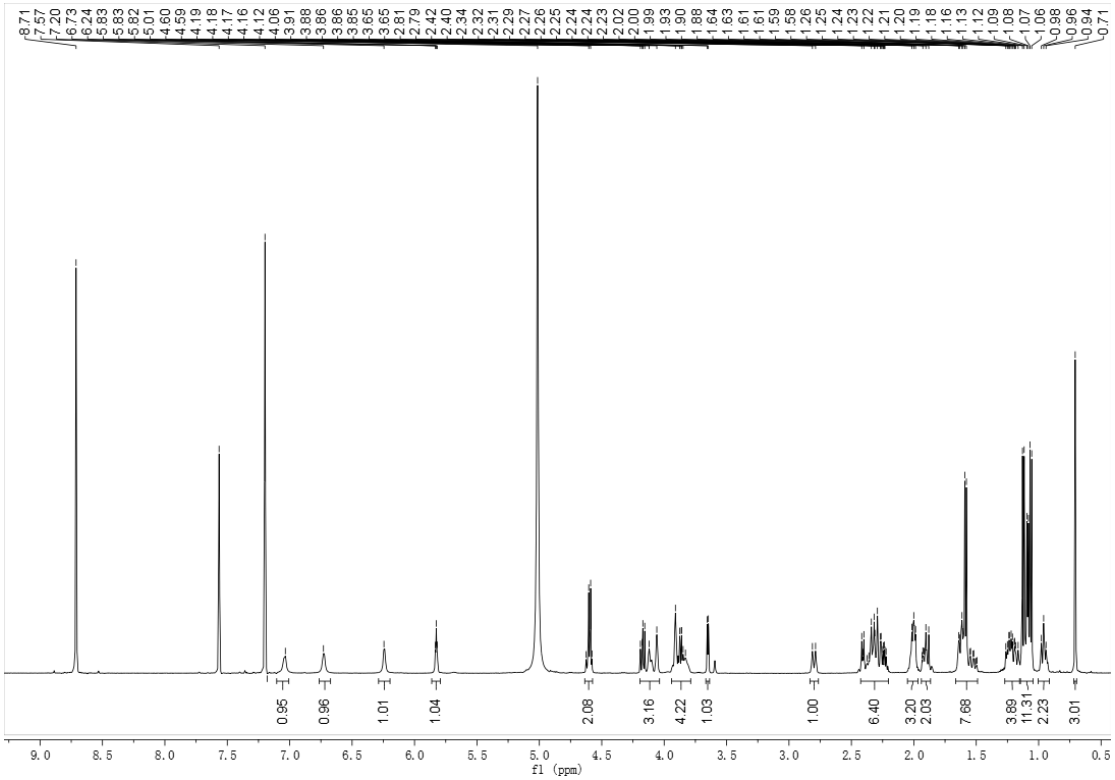


Fig. S17. ^{1}H NMR spectrum of compound **3** in pyridine-d_5.

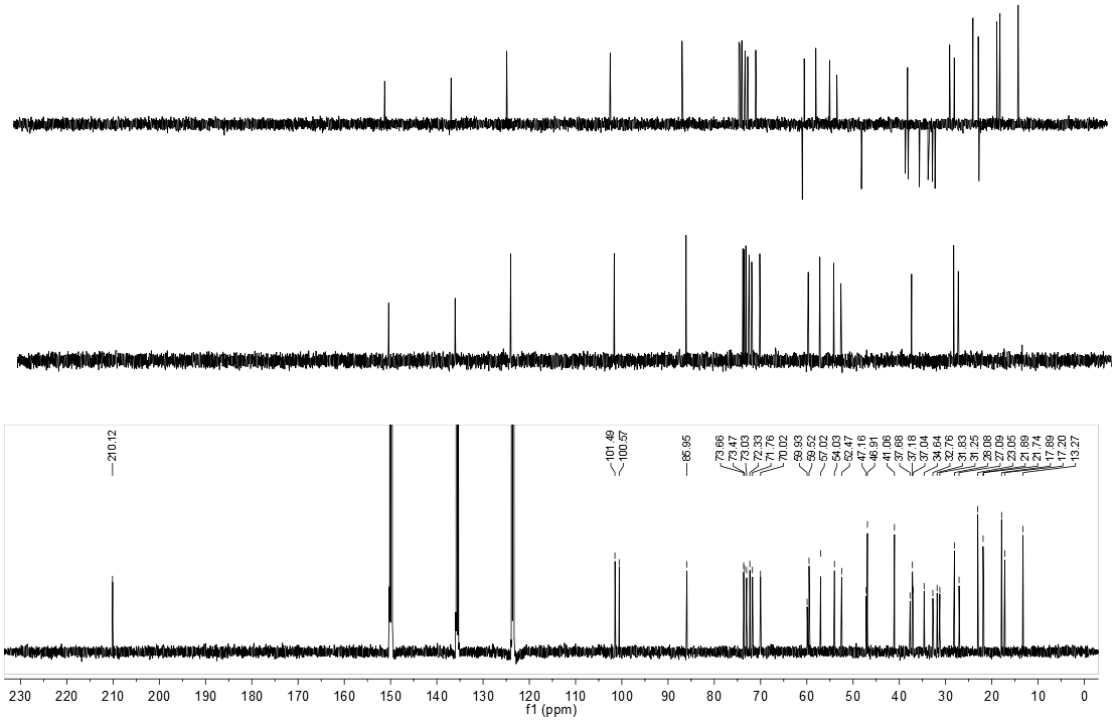


Fig. S18. ^{13}C NMR spectrum of compound **3** in pyridine-d_5.

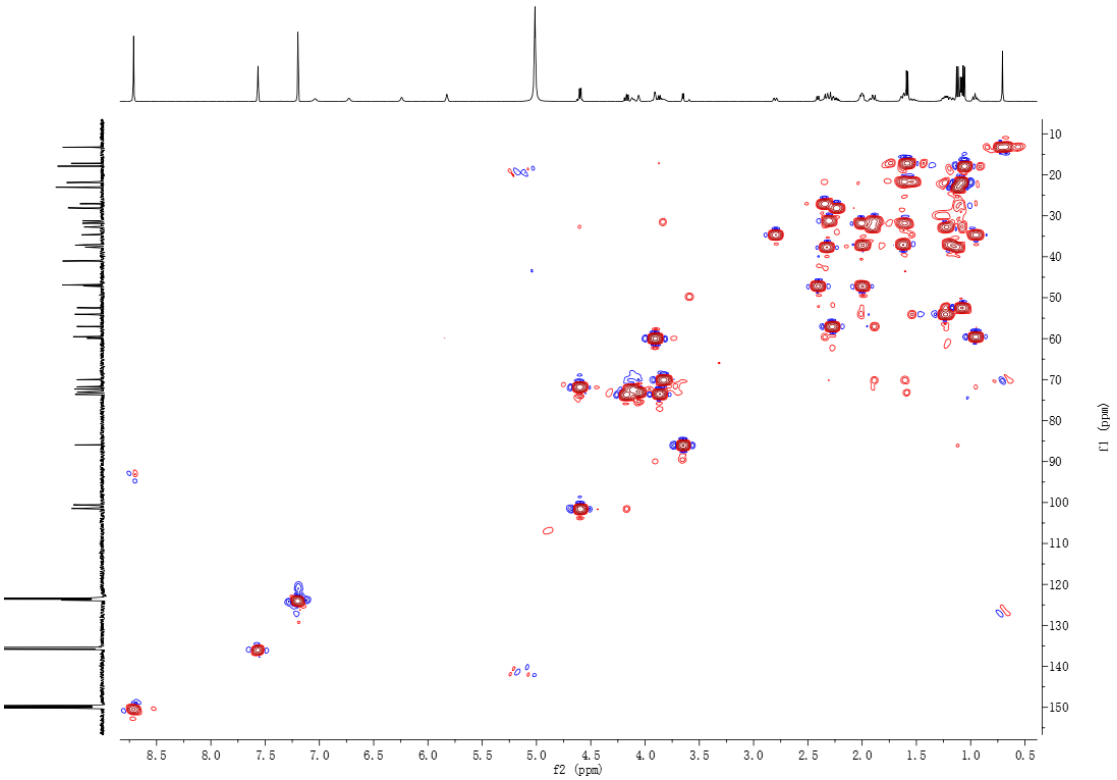


Fig. S19. HSQC spectrum of compound **3.**

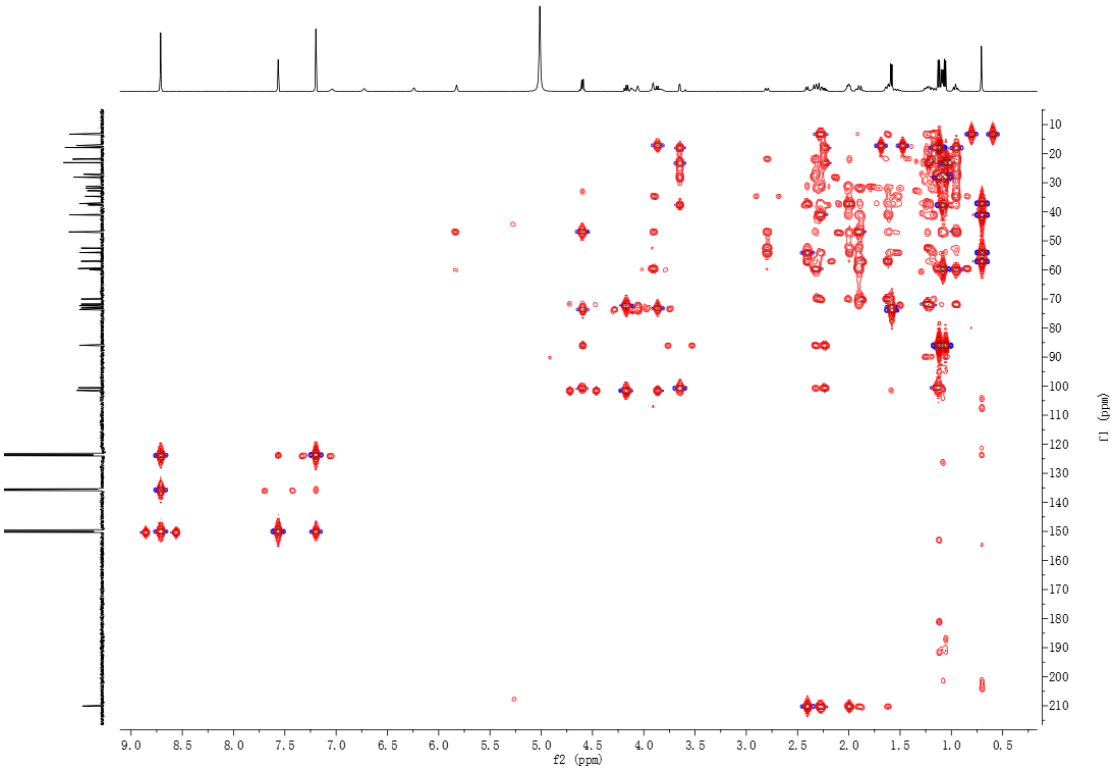


Fig. S20. HMBC spectrum of compound **3**.

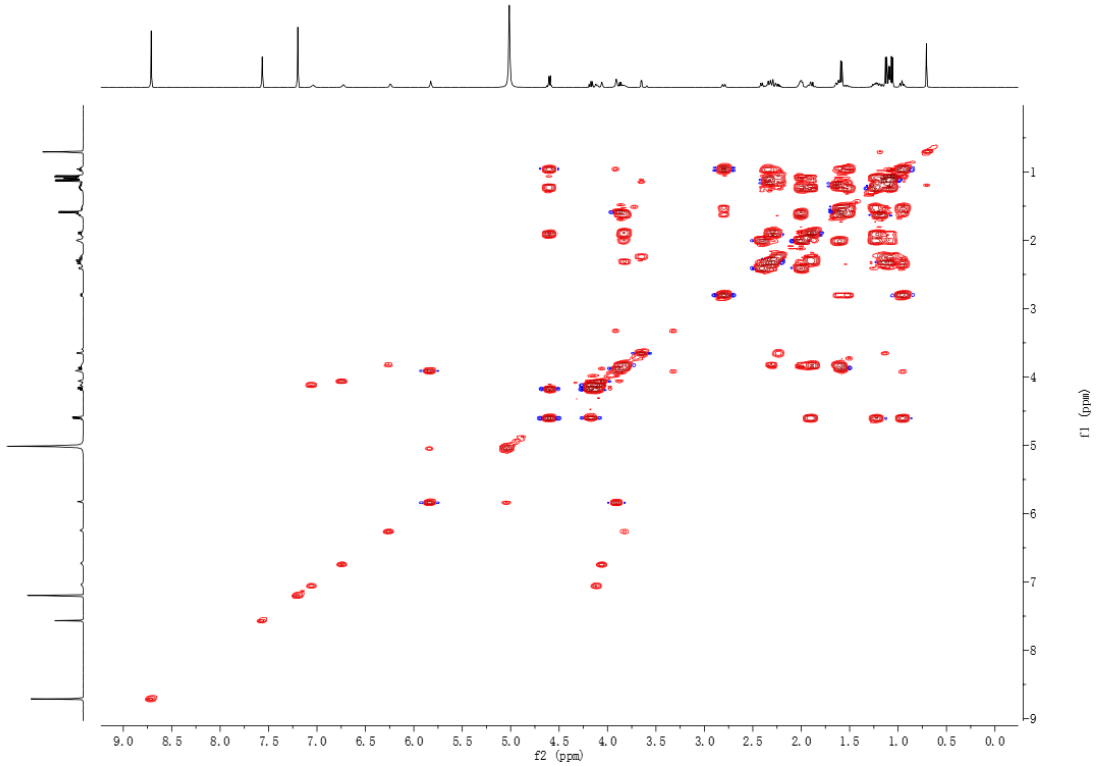


Fig. S21. $^{1}H–^{1}H$ COSY spectrum of compound **3**.

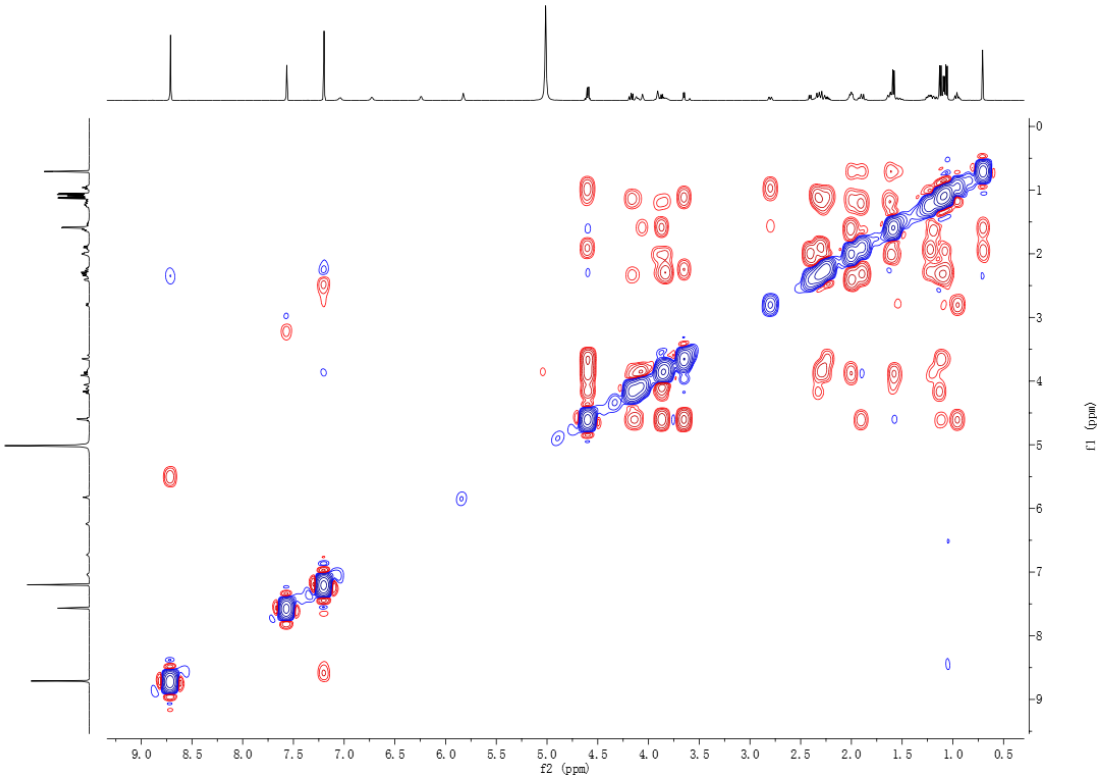
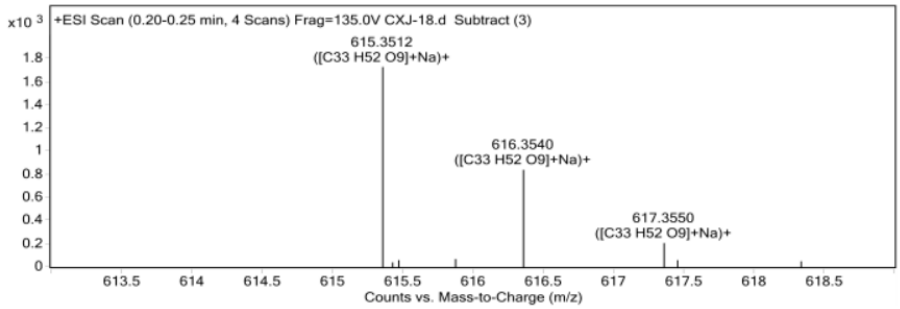


Fig. S22. ROESY spectrum of compound **3**.

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
135	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
102.1281	1	8644.77		
336.3264	1	1838.55		
338.3425	1	49782.18		
339.3457	1	11630.62		
340.3539	1	2370.91		
357.1887	2	1420.8		
360.3245	1	4225.5		
429.3005	1	14012.18		
430.3038	1	3817.69		
615.3512	1	1729.51	C33 H52 O9	(M+Na)+

Formula Calculator Element Limits

Element	Min	Max
C	3	60
H	0	120
O	0	30

Formula Calculator Results

Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C33 H52 O9	592.3611	615.3504	615.3512	-0.80	-1.30	8.0000

Fig. S23. HRESIMS spectrum of compound **3**.

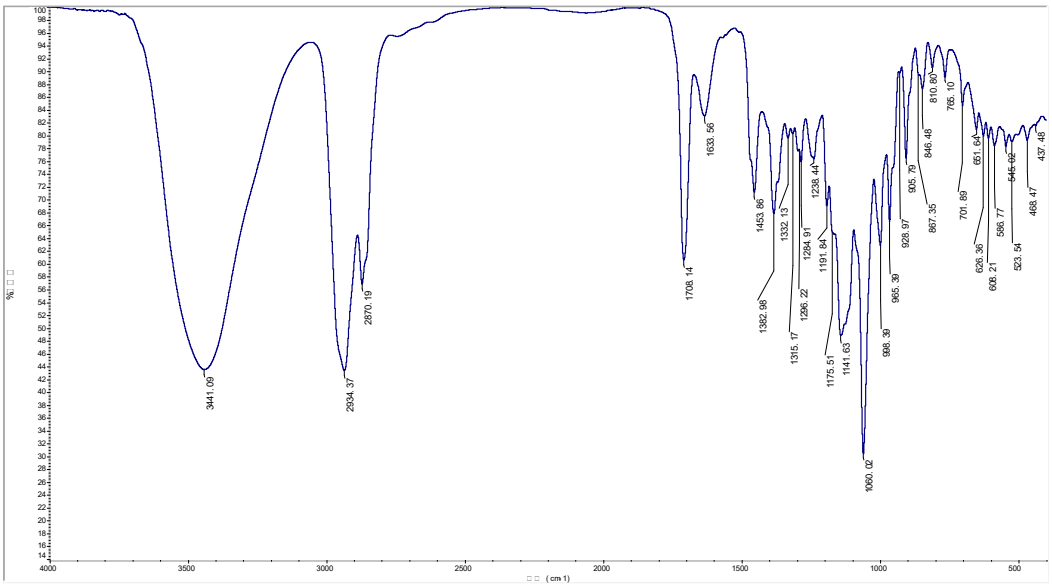


Fig. S24. IR spectrum of compound **3**.

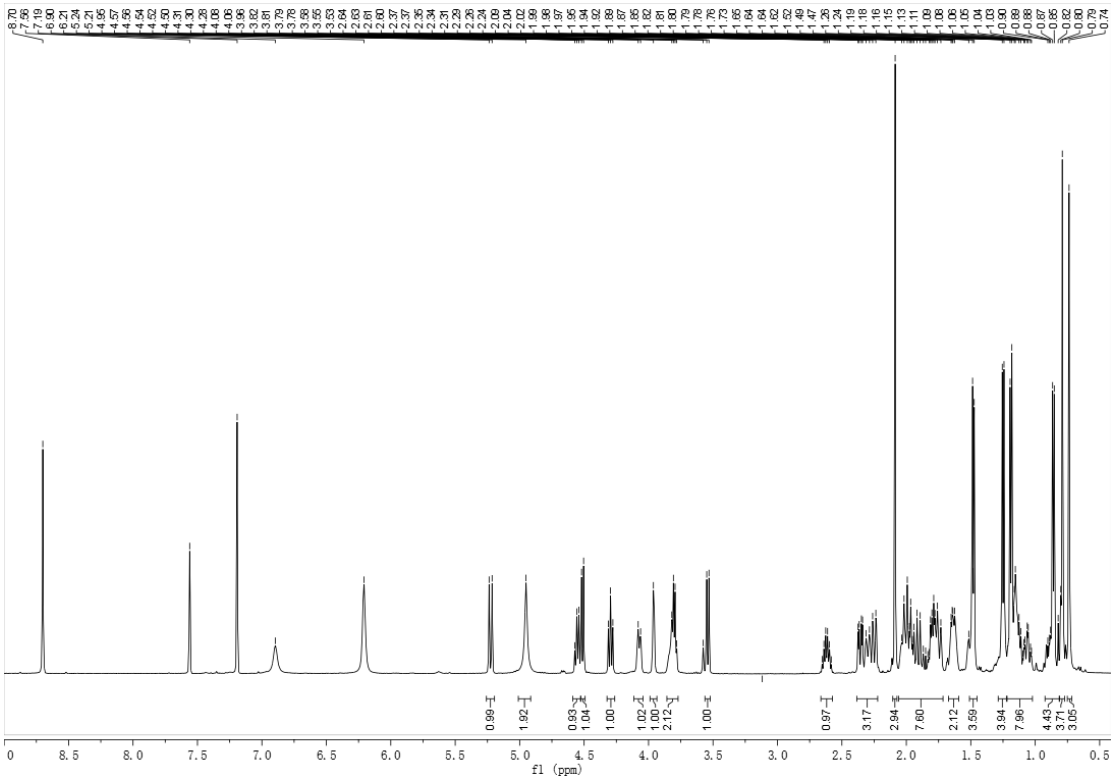


Fig. S25. ^{1}H NMR spectrum of compound **4** in pyridine-d_5.

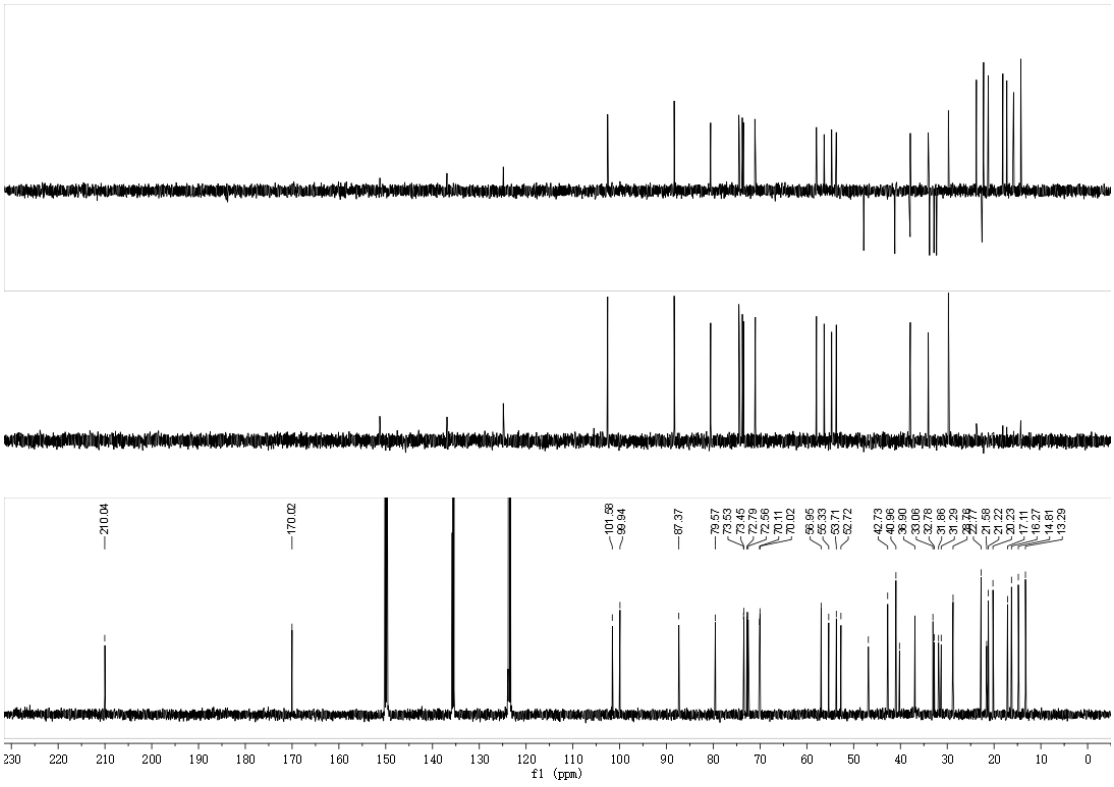


Fig. S26. ^{13}C NMR spectrum of compound **4** in pyridine-d_5.

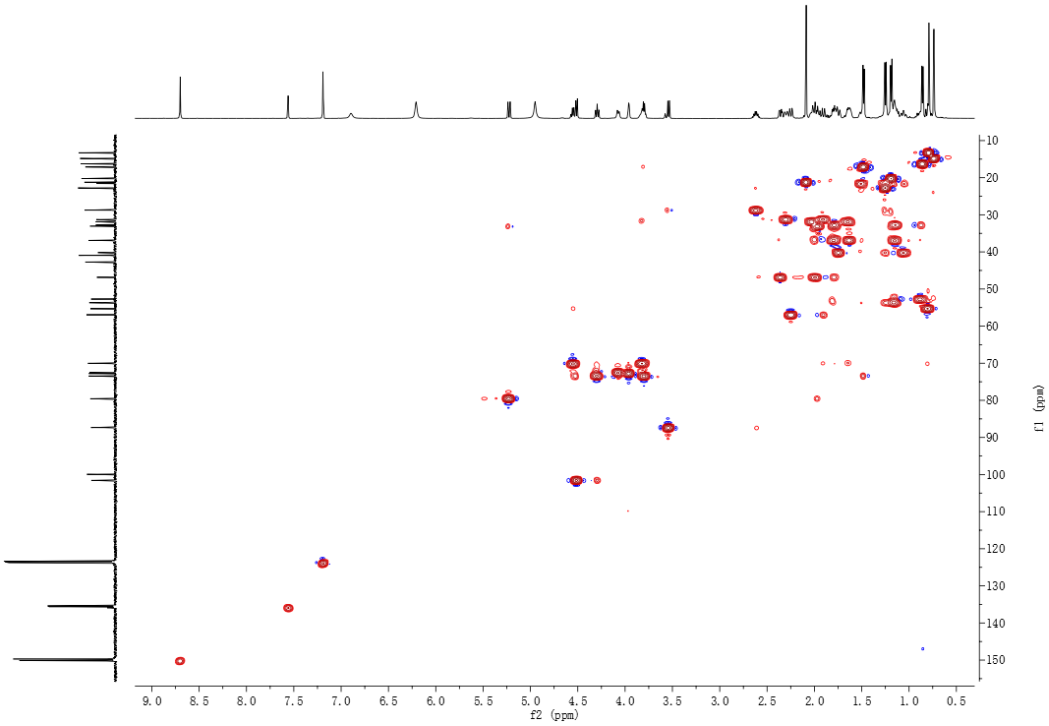


Fig. S27. HSQC spectrum of compound **4**.

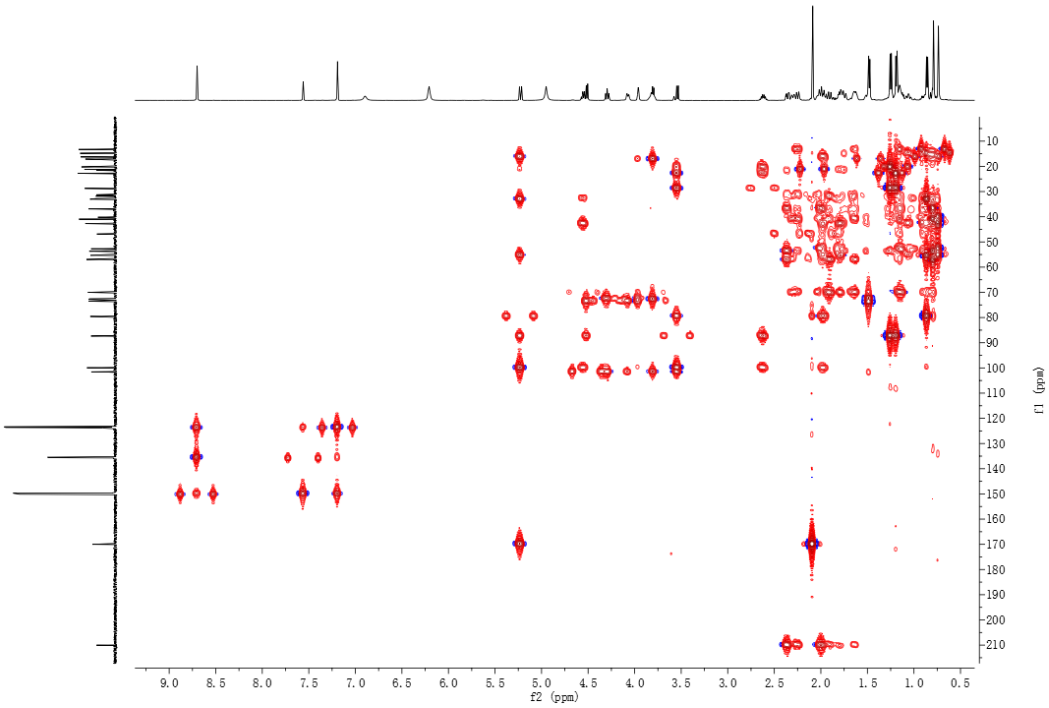


Fig. S28. HMBC spectrum of compound **4**.

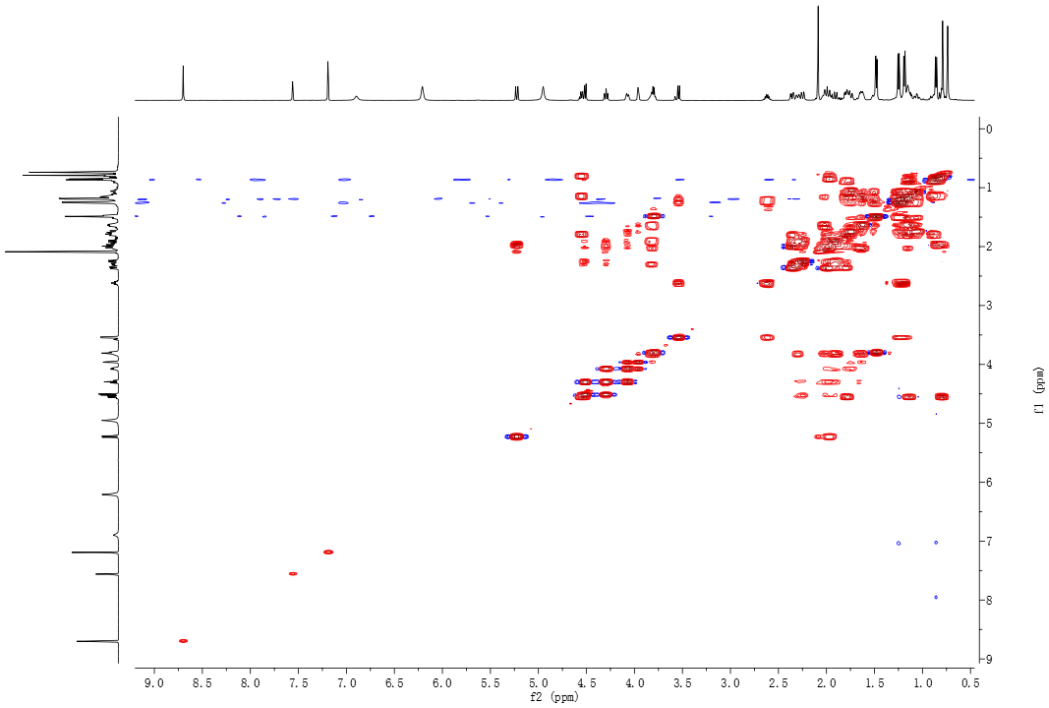


Fig.S29. $^{1}H–^{1}H$ COSY spectrum of compound **4.**

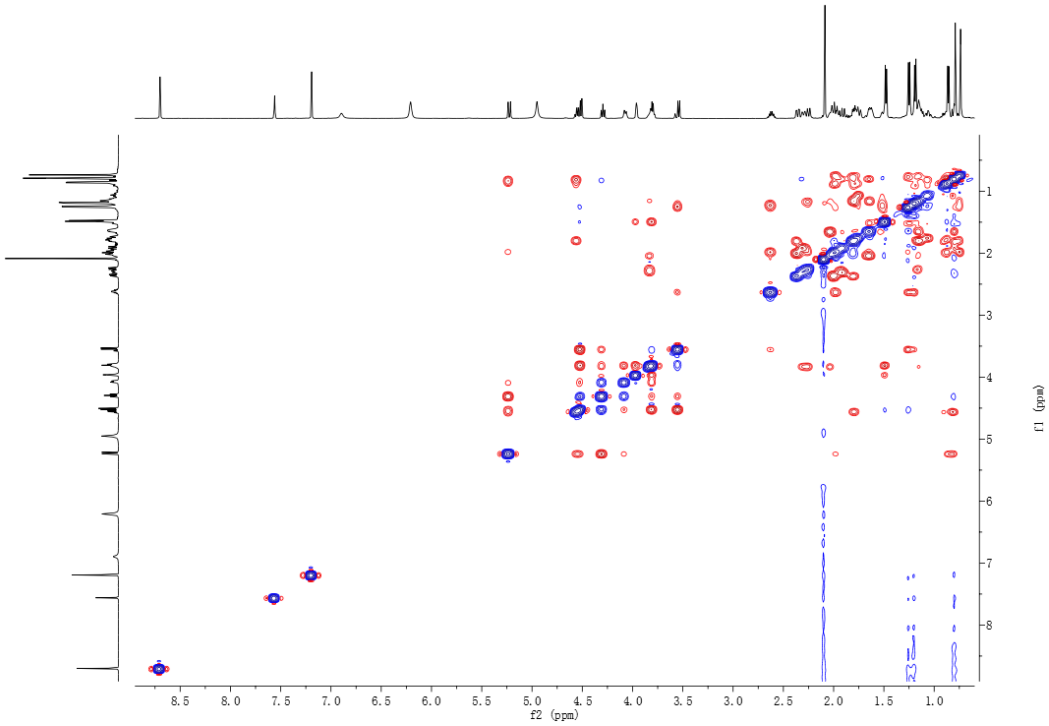
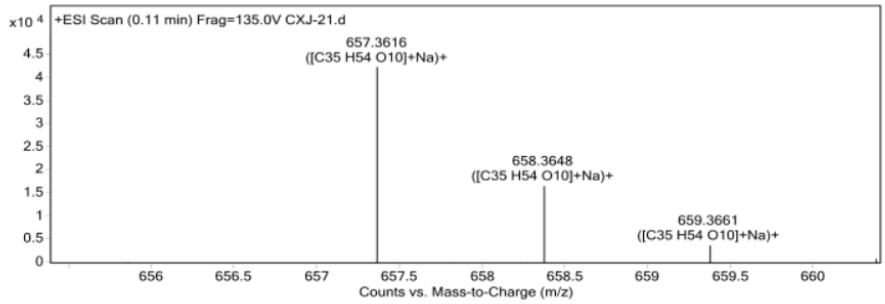


Fig. S30. ROESY spectrum of compound **4**.

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
135	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
110.0205	2	14621.05		
121.0509		16316.68		
429.3006	1	31904.4		
430.3031	1	9780.1		
471.3108	1	17232.18		
635.3792	1	16721.52		
657.3616	1	42411.7	C35 H54 O10	(M+Na)+
658.3648	1	16763.3	C35 H54 O10	(M+Na)+
922.0098	1	24569.45		
1291.732	1	12588.57		

Formula Calculator Element Limits

Element	Min	Max
C	3	60
H	0	120
O	0	30

Formula Calculator Results

Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C35 H54 O10	634.3717	657.3609	657.3616	-0.70	-1.06	9.0000

Fig. S31. HRESIMS spectrum of compound **4**.

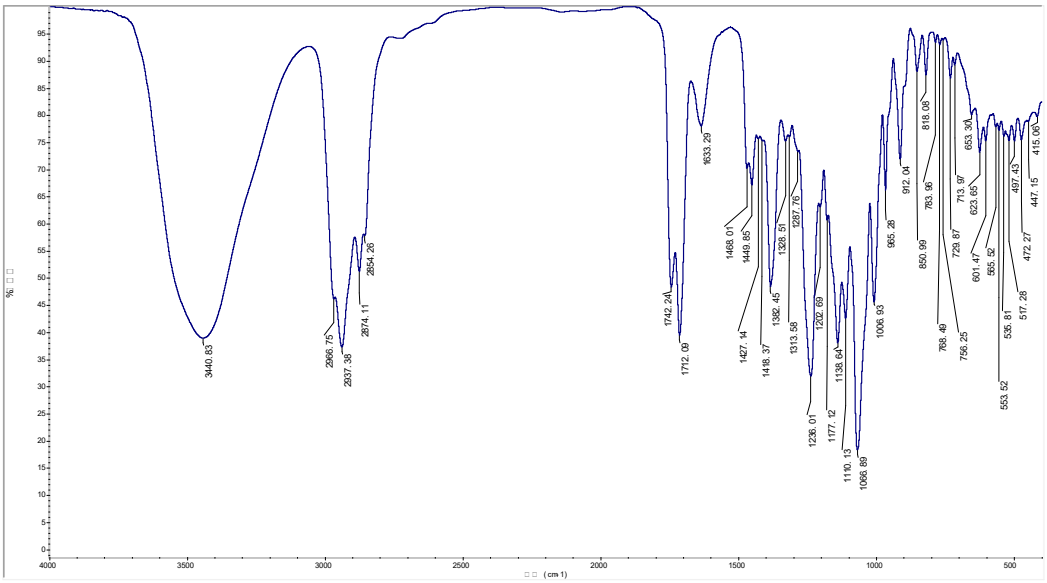


Fig. S32. IR spectrum of compound **4**.

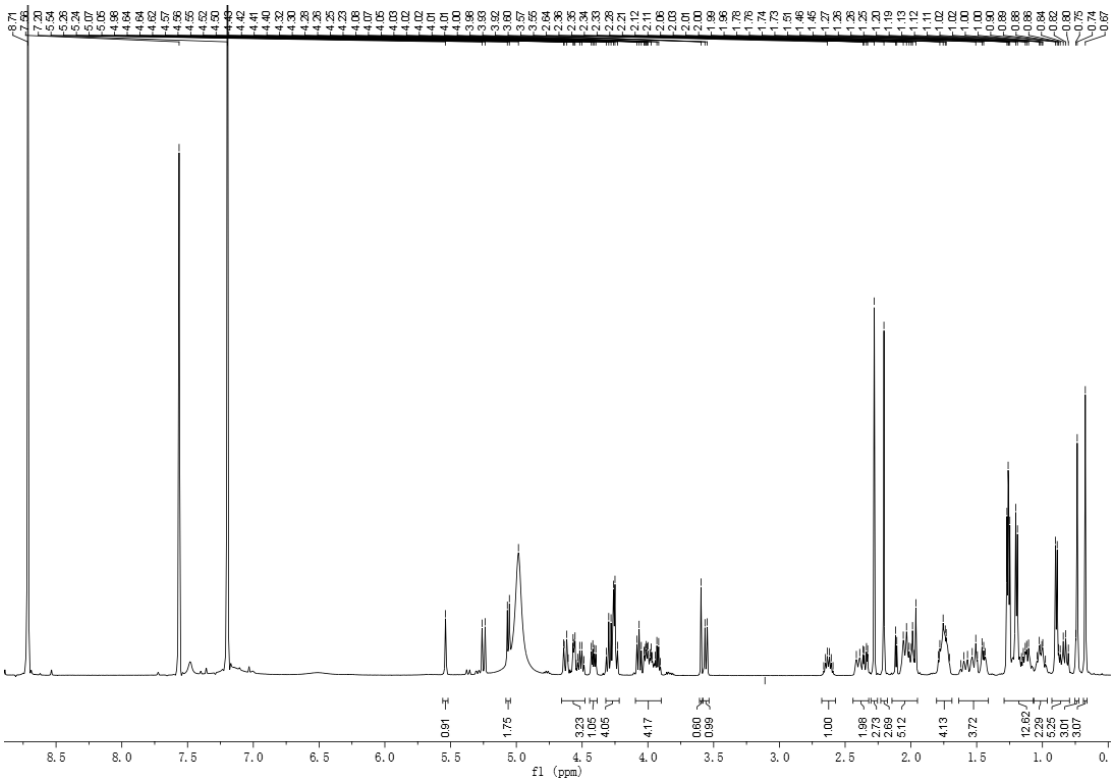


Fig. S33. ^{1}H NMR spectrum of compound **5** in pyridine-d_5.

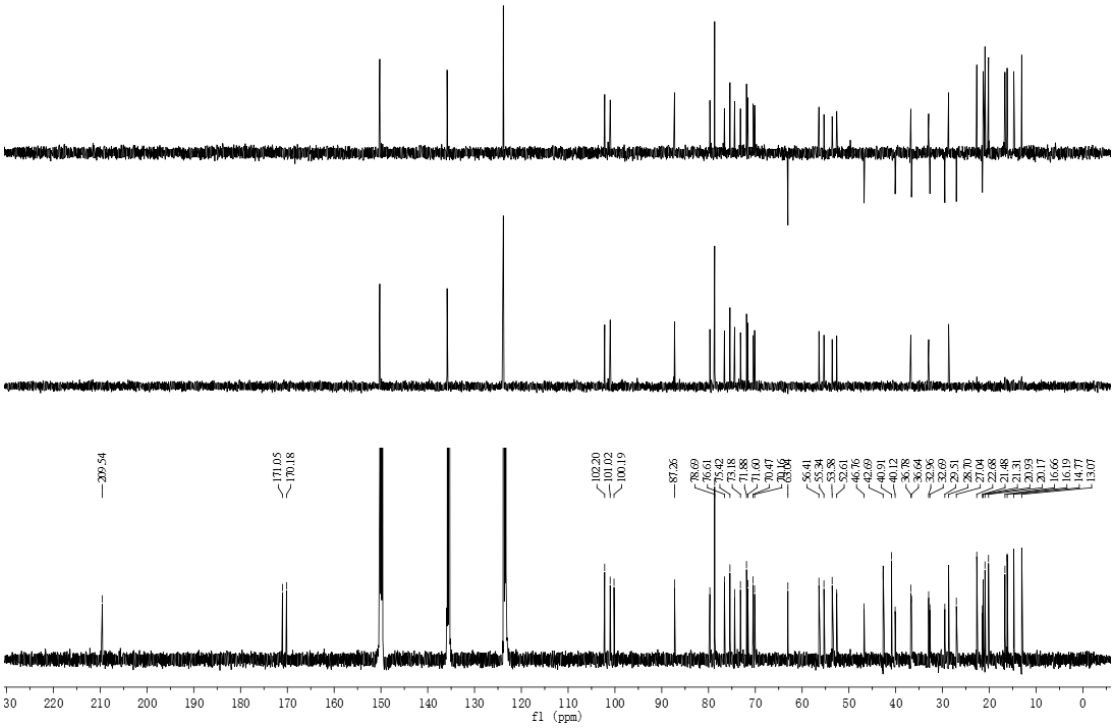


Fig. S34. ^{13}C NMR spectrum of compound **5** in pyridine-d_5.

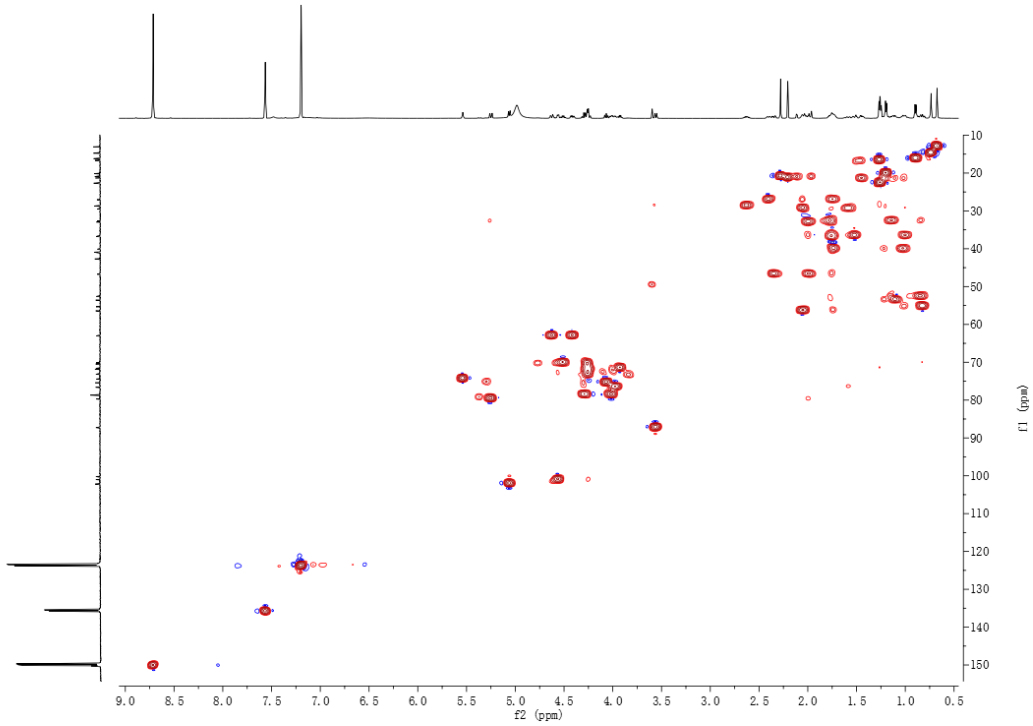


Fig. S35. HSQC spectrum of compound **5**.

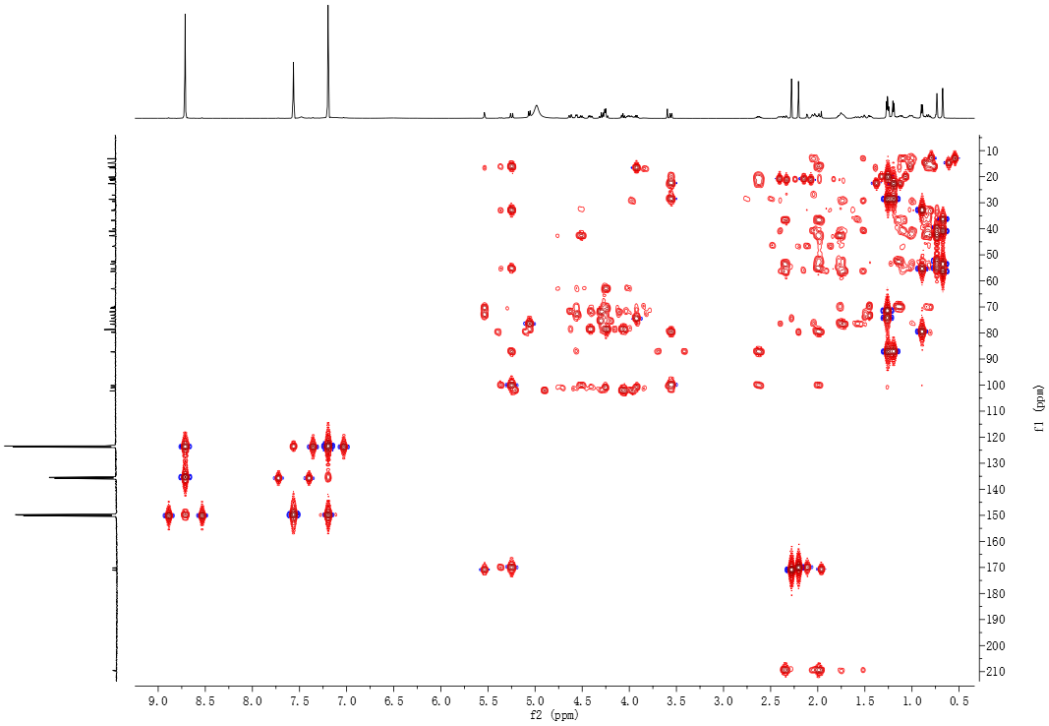


Fig. S36. HMBC spectrum of compound **5**.

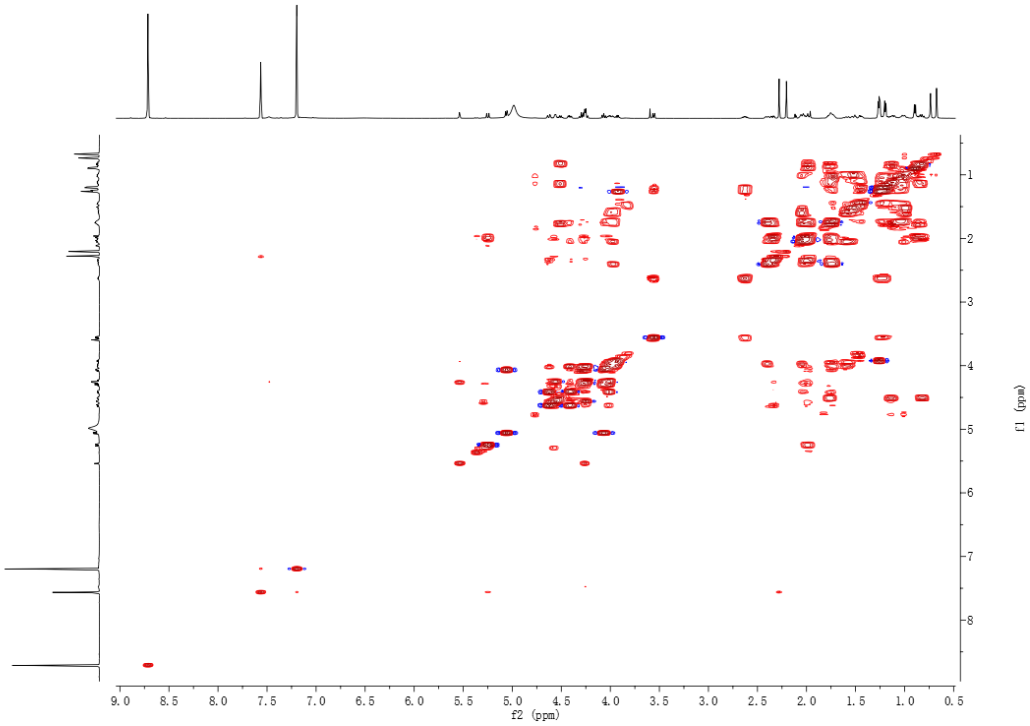


Fig. S37. $^{1}H–^{1}H$ COSY spectrum of compound **5**.

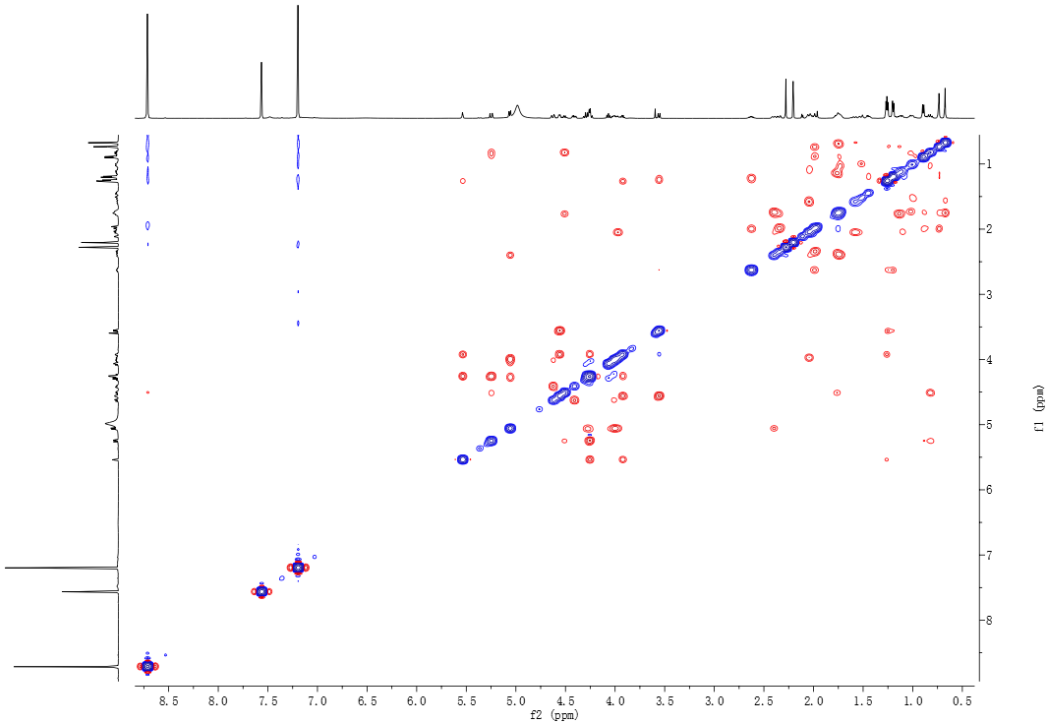
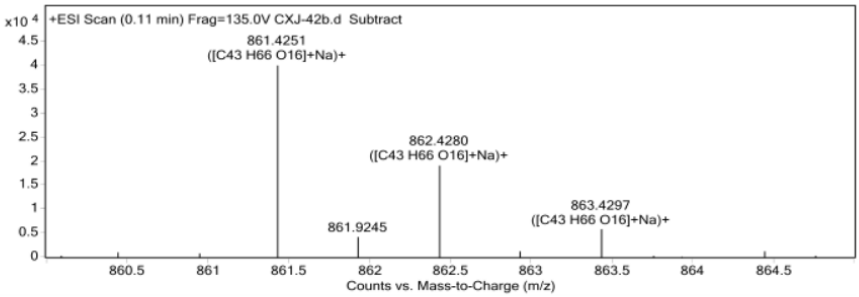


Fig. S38. ROESY spectrum of compound **5**.

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
135	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
231.0865	1	33109.54		
429.3005	1	91842.64		
430.3037	1	27346.28		
471.3112	1	27208.01		
591.3533	1	45325.41		
633.364	1	29771.33		
635.3787	1	29928.3		
677.3898	1	43686.63		
839.4434	1	59195.44		
861.4251	1	40074.09	C43 H66 O16	(M+Na)+

Formula Calculator Element Limits

Element	Min	Max
C	3	60
H	0	120
O	0	30

Formula Calculator Results

Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C43 H66 O16	838.4351	861.4243	861.4251	-0.80	-0.93	11.0000

Fig. S39. HRESIMS spectrum of compound **5**.

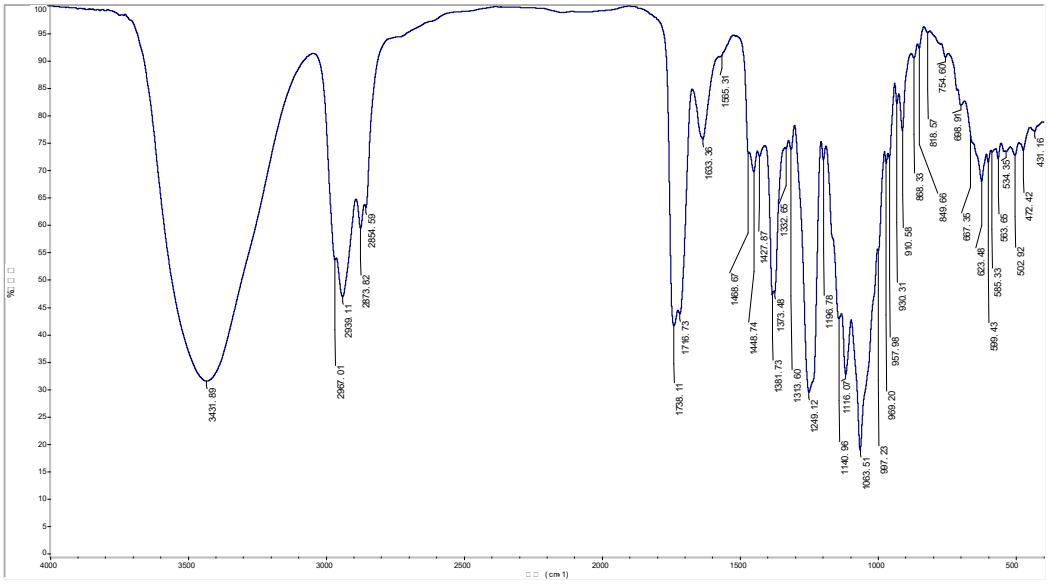


Fig. S40. IR spectrum of compound **5**.

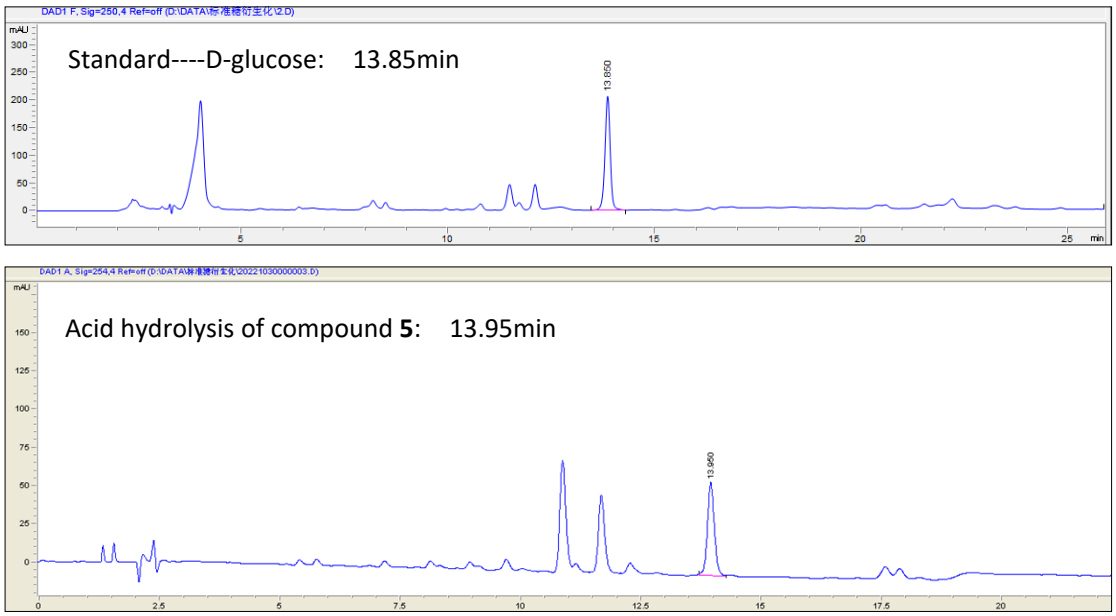


Fig. S41. Acid hydrolysis of compound **5.**

Table S1. X-ray crystallographic data for **1**

Identification code	global	
Empirical formula	C34 H56 O10	
Formula weight	624.78	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 1 21 1	
Unit cell dimensions	a = 12.2949(3) Å	α= 90°.
	b = 9.7000(2) Å	β= 110.8410(10)°.
	c = 14.6302(3) Å	γ = 90°.
Volume	1630.65(6) $Å^3$	
Z	2	
Density (calculated)	1.272 Mg/m^3	
Absorption coefficient	0.752 mm^{-1}	
F(000)	680	
Crystal size	0.480 x 0.450 x 0.190 mm^3	
Theta range for data collection	3.23 to 72.47°.	
Index ranges	-15<=h<=15, -11<=k<=11, -17<=l<=18	
Reflections collected	30722	
Independent reflections	6401 [R(int) = 0.0363]	
Completeness to theta = 72.47°	99.5 %	
Absorption correction	Semi-empirical from equivalents	

Max. and min. transmission	0.87 and 0.71
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6401 / 1 / 412
Goodness-of-fit on F^2	1.046
Final R indices [I>2sigma(I)]	R1 = 0.0279, wR2 = 0.0702
R indices (all data)	R1 = 0.0280, wR2 = 0.0703
Absolute structure parameter	-0.02(3)
Largest diff. peak and hole	0.217 and -0.181 e.Å^{-3}

Table S2. X-ray crystallographic data for **2**

Identification code	global	
Empirical formula	C32 H52 O9	
Formula weight	580.73	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 6.70730(10) Å	α= 90°.
	b = 11.6828(3) Å	β= 90°.
	c = 38.5757(8) Å	γ = 90°.
Volume	3022.79(11) Å^3	
Z	4	
Density (calculated)	1.276 Mg/m^3	
Absorption coefficient	0.747 mm^{-1}	
F(000)	1264	
Crystal size	0.540 x 0.030 x 0.010 mm^3	
Theta range for data collection	2.29 to 72.38°.	
Index ranges	-8<=h<=6, -14<=k<=14, -47<=l<=47	
Reflections collected	27775	
Independent reflections	5969 [R(int) = 0.0969]	
Completeness to theta = 72.38°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.99 and 0.88	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5969 / 0 / 381	
Goodness-of-fit on F^2	1.041	
Final R indices [I>2sigma(I)]	R1 = 0.0372, wR2 = 0.0845	
R indices (all data)	R1 = 0.0479, wR2 = 0.0898	

Absolute structure parameter	0.14(9)
Largest diff. peak and hole	0.267 and -0.238 e.Å^{-3}

Table S3. X-ray crystallographic data for **3**

Identification code	global	
Empirical formula	C34 H58 O11	
Formula weight	642.80	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 1 21 1	
Unit cell dimensions	a = 12.0479(3) Å	α= 90°.
	b = 7.6358(2) Å	β= 94.9550(10)°.
	c = 17.9880(5) Å	γ = 90°.
Volume	1648.63(8) Å^3	
Z	2	
Density (calculated)	1.295 Mg/m^3	
Absorption coefficient	0.781 mm^{-1}	
F(000)	700	
Crystal size	0.450 x 0.220 x 0.130 mm^3	
Theta range for data collection	3.68 to 72.36°.	
Index ranges	-14<=h<=14, -9<=k<=9, -21<=l<=16	
Reflections collected	19289	
Independent reflections	6224 [R(int) = 0.0359]	
Completeness to theta = 72.36°	99.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.91 and 0.68	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6224 / 1 / 417	
Goodness-of-fit on F^2	1.056	
Final R indices [I>2sigma(I)]	R1 = 0.0285, wR2 = 0.0726	
R indices (all data)	R1 = 0.0289, wR2 = 0.0731	
Absolute structure parameter	0.03(4)	
Largest diff. peak and hole	0.245 and -0.207 e.Å^{-3}	