Supporting Information

Kalshiolin A, new lignan from *Kalimeris shimadai*

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Qualitative Analysis Report

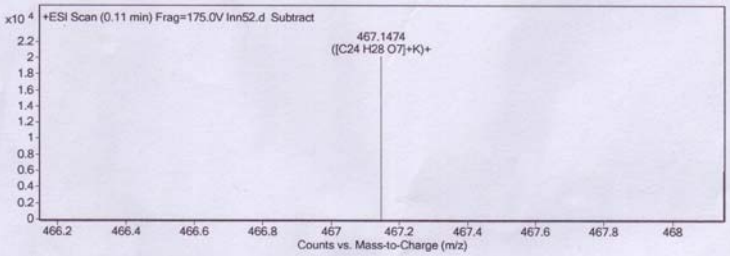
Data Filename	Inn52.d	**Sample Name**	Inn52
Sample Type	Sample	**Position**	P1-A5
Instrument Name	Instrument 1	**User Name**	
Acq Method	SIBU.m	**Acquired Time**	7/10/2017 10:59:45 AM
IRM Calibration Status	Success	**DA Method**	Default.m
Comment			

Sample Group **Info.**

Acquisition SW Version 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
175	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
323.1267	1	2655.59		
381.0801		2046.47		
451.173	1	9990.57		
452.1754	1	2331.2		
467.1474	1	20216.04	C24 H28 O7	(M+K)+
468.15	1	6015.62	C24 H28 O7	(M+K)+
469.1487	1	2115.34	C24 H28 O7	(M+K)+
474.2489	1	3581.32		
488.2444	1	3078.19		
735.2455	1	1932.15		

Formula Calculator Element Limits

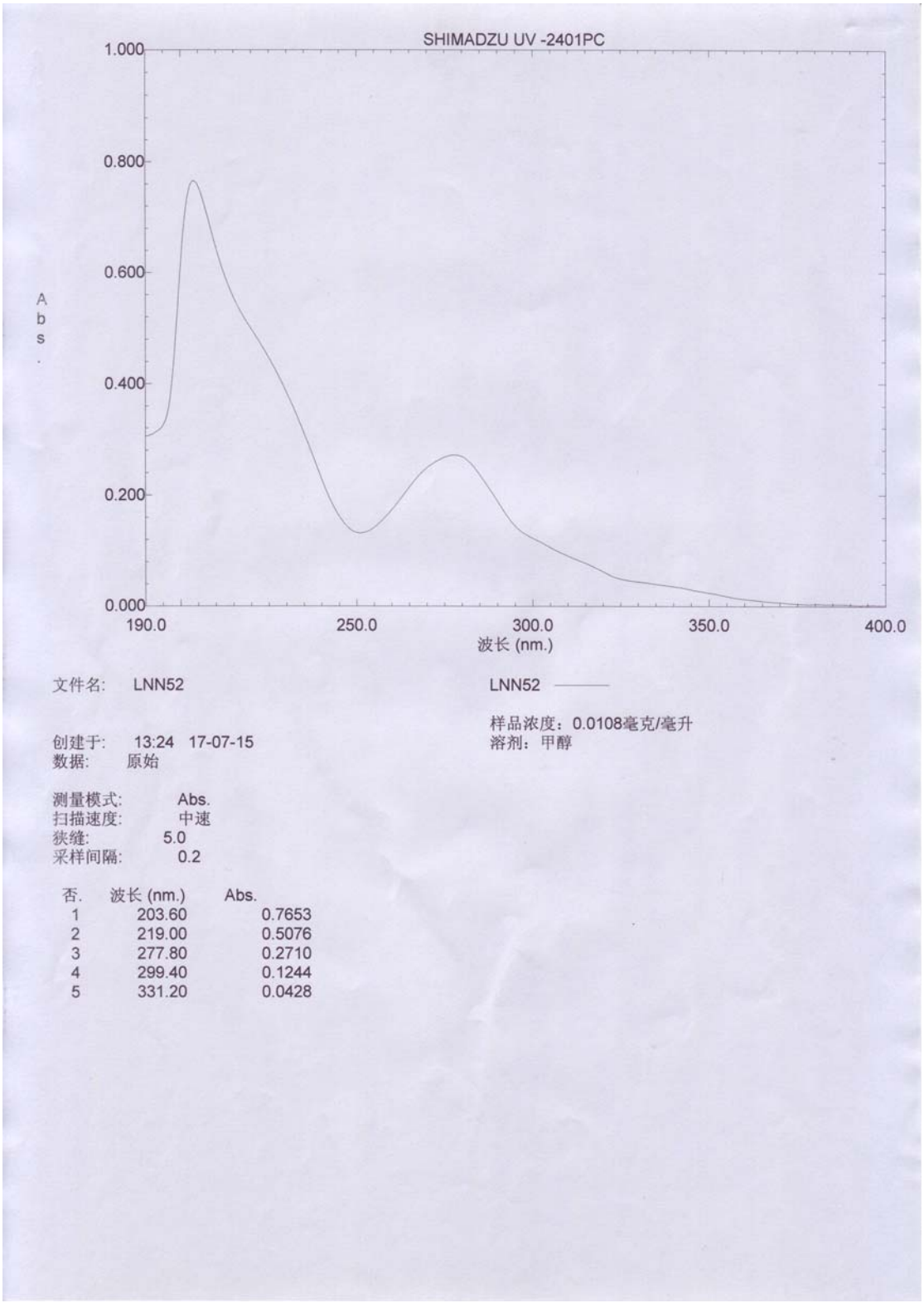
Element	Min	Max
C	3	60
H	0	120
O	0	30

Formula Calculator Results

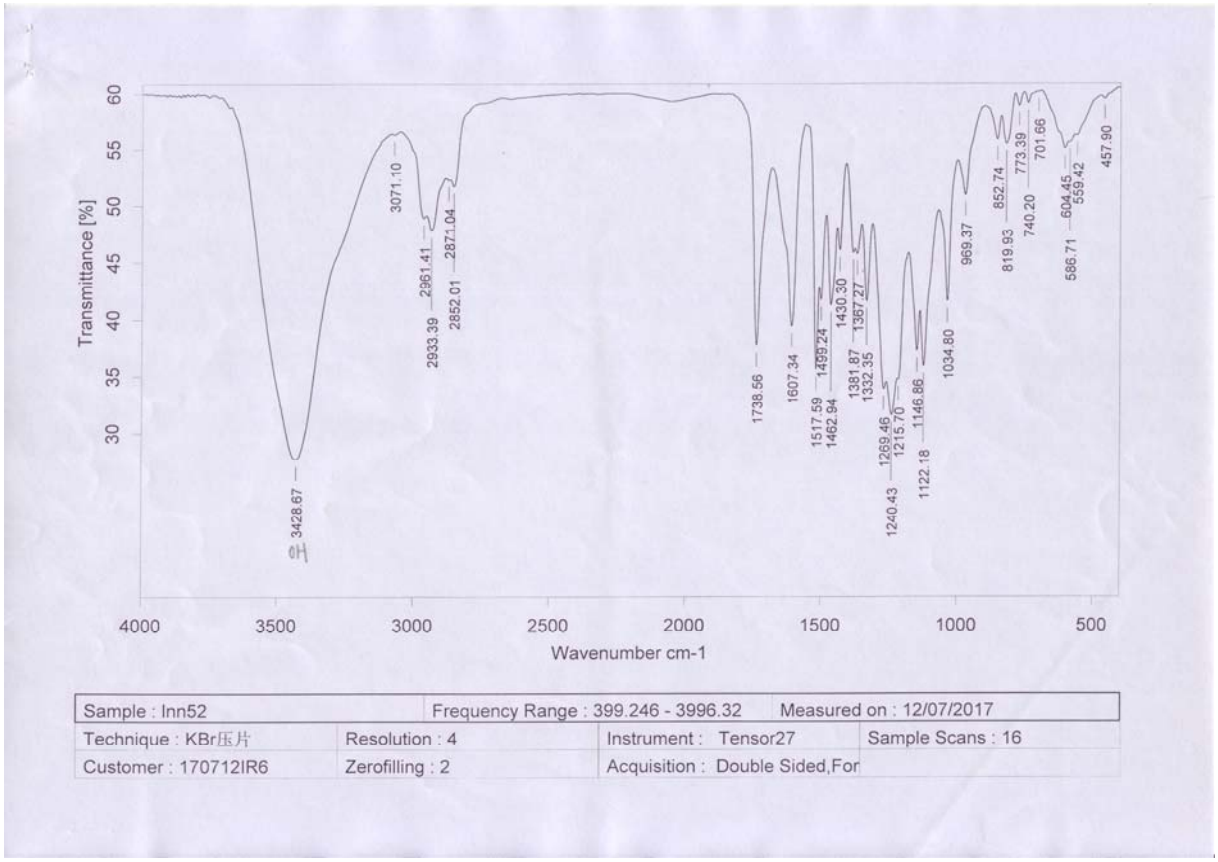
Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C24 H28 O7	428.1835	467.1467	467.1474	-0.6	-1.4	11.0000

--- End Of Report ---

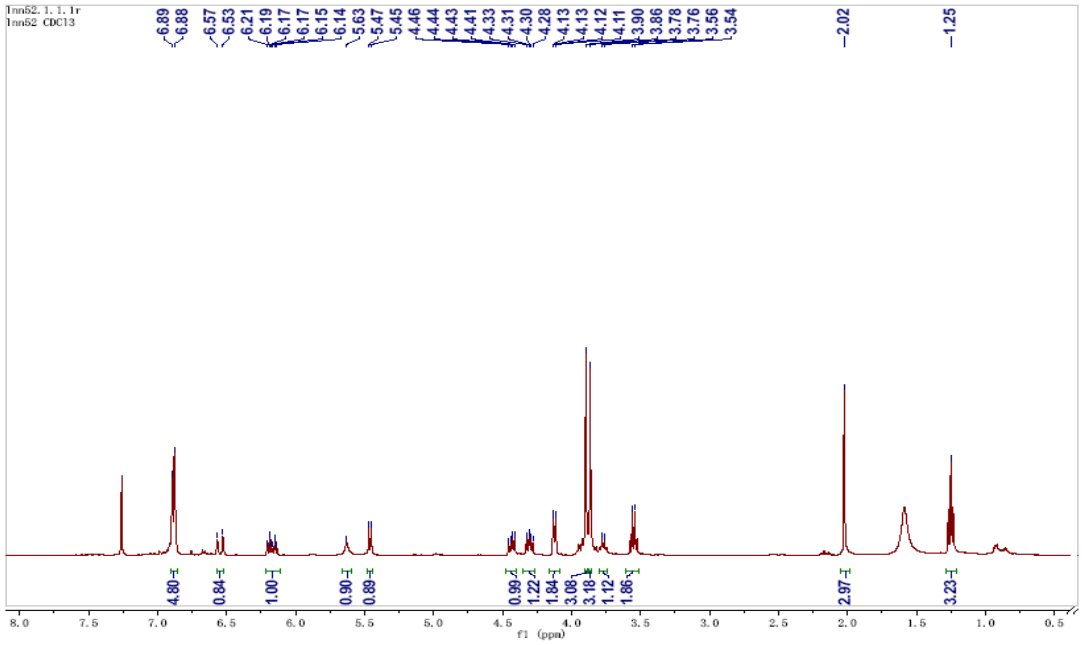
S1. HR-ESI-MS spectrum of compound **1**



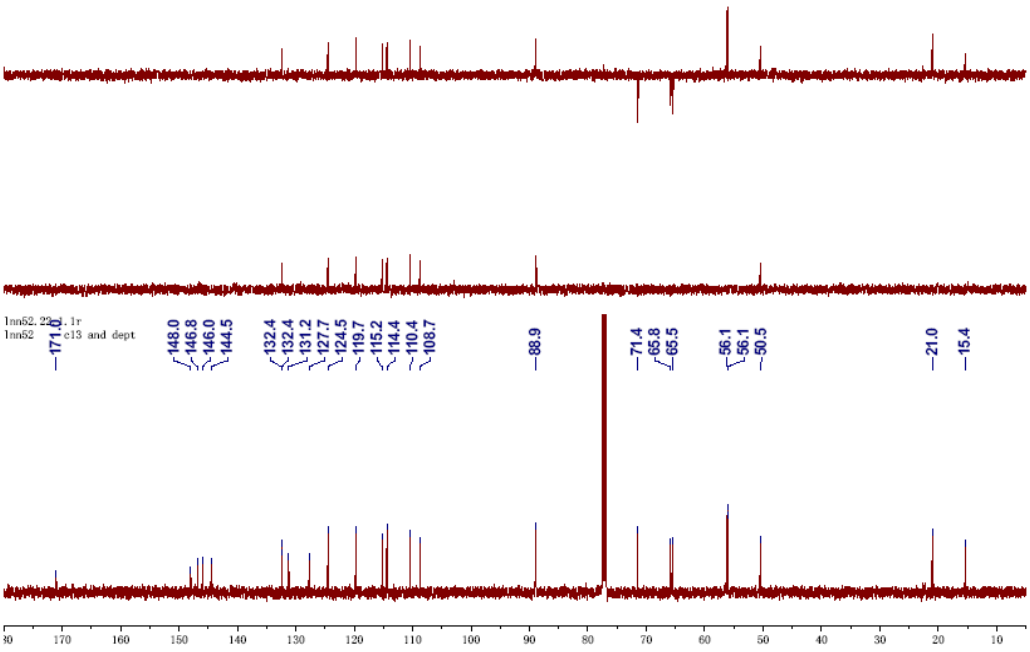
S2. UV spectrum of compound **1**



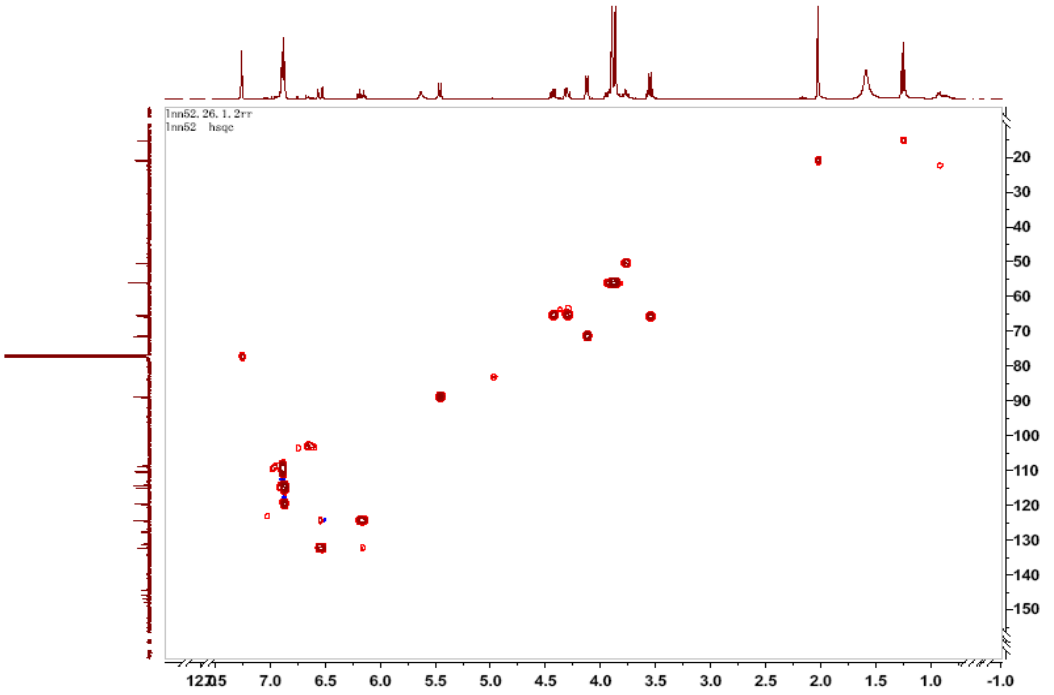
S3. IR spectrum of compound **1**



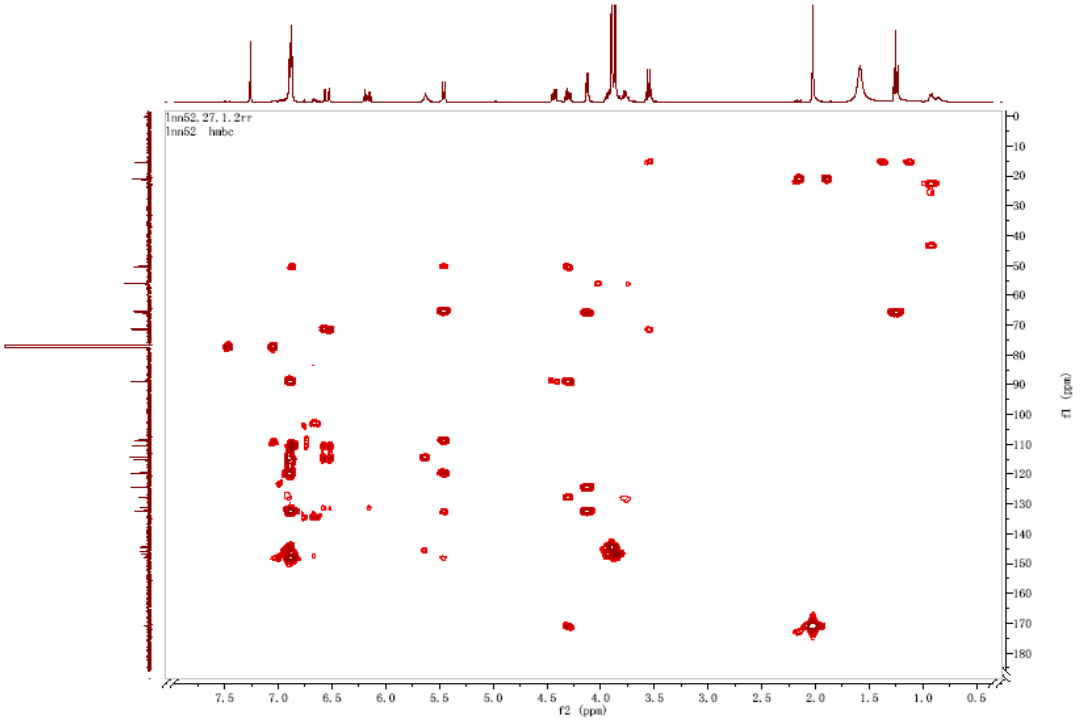
S4. ^{1}H NMR spectrum of compound **1** in CD_3OD



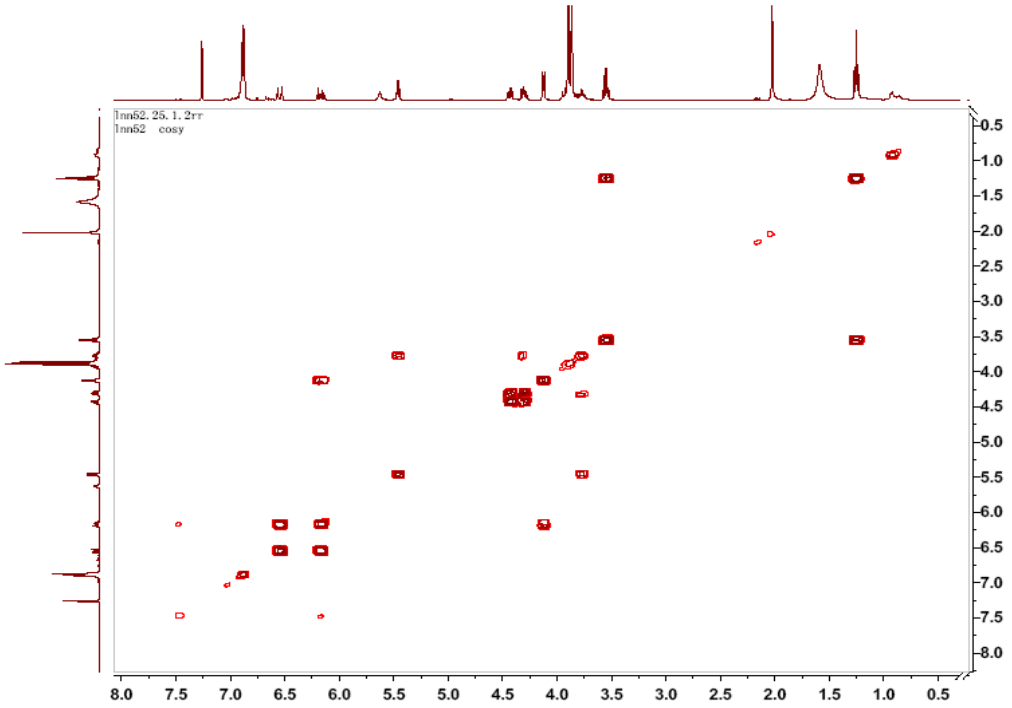
S5. ^{13}C NMR spectrum of compound **1** in CD_3OD



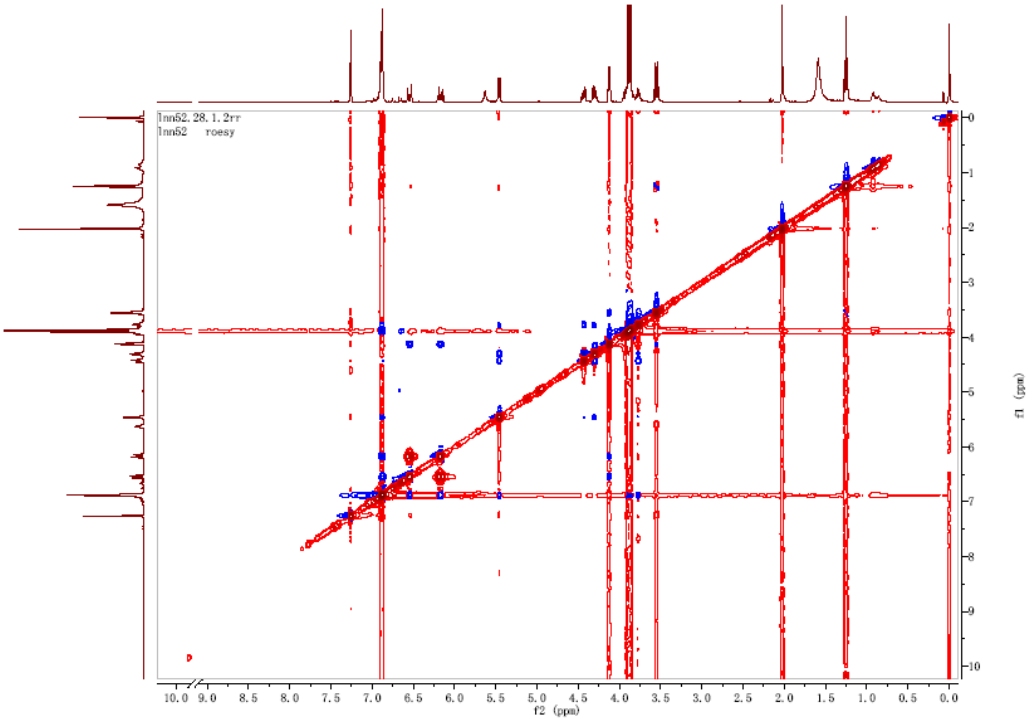
S6. HSQC spectrum of compound **1** in CD_3OD



S7. HMBC spectrum of compound **1** in CD_3OD



S8. ^{1}H-^{1}H COSY spectrum of compound **1** in CD_3OD



S9. ROESY spectrum of compound **1** in CD_3OD

Optical rotation measurement

Model : P-1020 (A060460638)

No.	Sample	Mode	Data	Monitor Blank	Temp. Cell Temp Point	Date Comment Sample Name	Light Filter Operator	Cycle Time Integ Time
No.1	35 (1/3)	Sp.Rot	-8.7500	-0.0021 0.0000	23.7 10.00 Cell	Wed Jul 12 21:25:06 2017 0.00240g/mL MeOH LNN52	Na 589nm	2 sec 2 sec
No.2	35 (2/3)	Sp.Rot	-8.3330	-0.0020 0.0000	23.7 10.00 Cell	Wed Jul 12 21:25:11 2017 0.00240g/mL MeOH LNN52	Na 589nm	2 sec 2 sec
No.3	35 (3/3)	Sp.Rot	-7.9170	-0.0019 0.0000	23.7 10.00 Cell	Wed Jul 12 21:25:16 2017 0.00240g/mL MeOH LNN52	Na 589nm	2 sec 2 sec

S10. Optical rotation of compound **1**

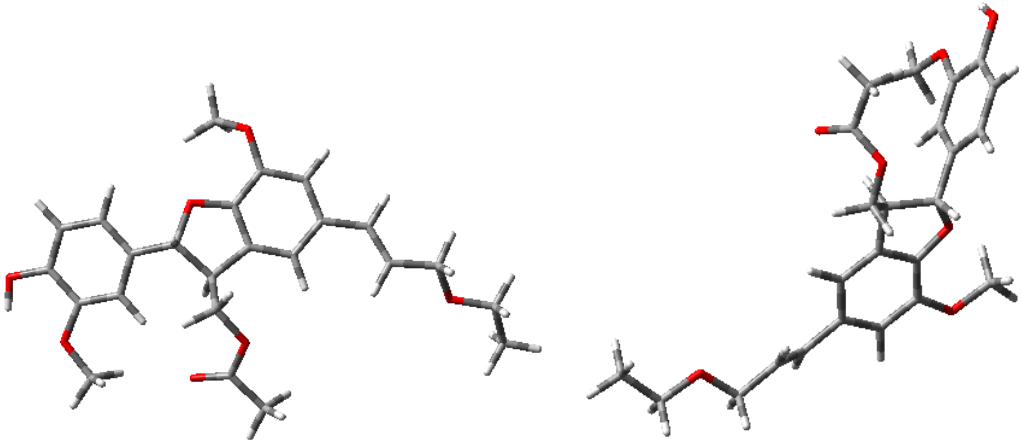
Compound	Cytotoxic activity, IC_{50} (μM)				
	A549	MDA-MB-231	MCF7	KB	KB-VIN
Kalshiolin A	38.84±2.41	33.99±0.63	33.61±1.56	35.91±1.01	43.29±1.09
Paclitaxel (nM)	6.38±0.21	9.21±0.29	14.50±0.13	6.44±0.14	2338.59±68.14

S11. Cytotoxicity data of compound **1**

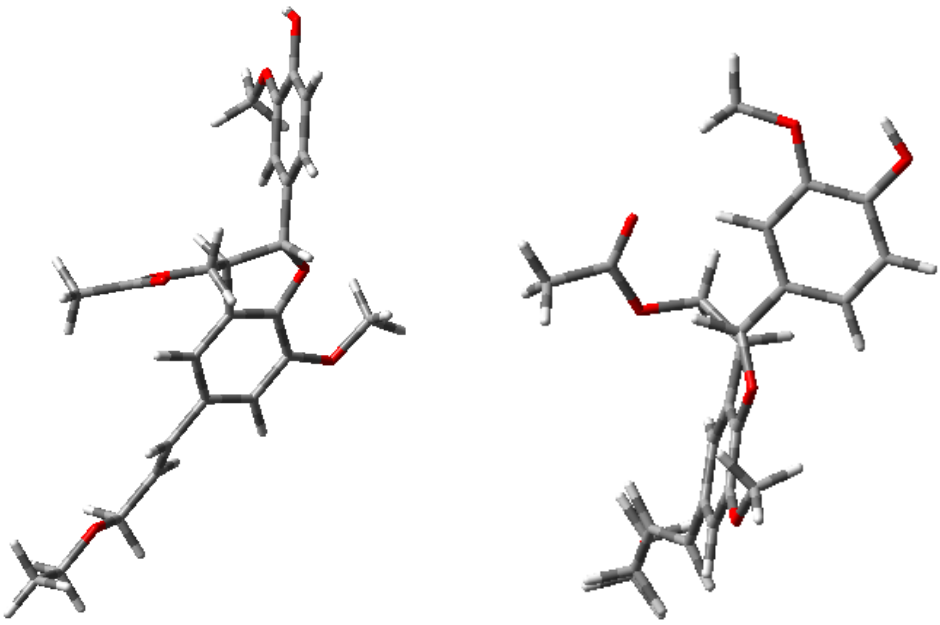
S12 Computational details of 1

A conformation search based on molecular mechanics with MMFF94S force fields was performed for **1**, which gave five stable conformers.[1,2] Two selected conformers with distributions higher than 5% were further optimized by density functional theory method at the B3LYP/6-31G(d,p) level in Gaussian 09 program package,[3] leading to two minimum geometries, which were further checked by frequency calculation and resulted in no imaginary frequencies. The ECD was calculated using TDDFT-B3LYP/6-31G(d,p) of theory on B3LYP/6-311G(2d,p) optimized geometry. The calculated ECD curve and weighted ECD were generated using SpecDis 1.64 with σ = 0.2 ev, and UV shift 3 nm.[4]

Four optimized conformers of **1**



12.25 % 37.14 %



23.26 %　　27.24 %

References:

[1] Goto, H.; Osawa, E.; *J. Am. Chem. Soc.***1989**, *111*, 8950–8951.

[2] Goto, H.; Osawa, E.; *J. Chem. Soc., Perkin Trans. 2*, **1993**, 187–198.

[3] Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, J. A.; Jr., Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Keith, T.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, J. M.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, O.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; and Fox, D. J.; Gaussian *09*, Revision B.01, Gaussian, Inc., Wallingford CT, 2010.

[4] SpecDis: Quantifying the Comparison of Calculated and Experimental Electronic Circular Dichroism Spectra**,** T. Bruhn, A. Schaumlöffel, Y. Hemberger, G. Bringmann, *Chirality* **2013**, *25*, 243–249.