Supplementary Information

Penicimenolides A-F, Resorcylic Acid Lactones from *Penicillium* sp., isolated from the Rhizosphere Soil of *Panax notoginseng*

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[+]These authors contributed equally to this work.

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List of Supporting Information

1. Acetylation of 2 and 12.

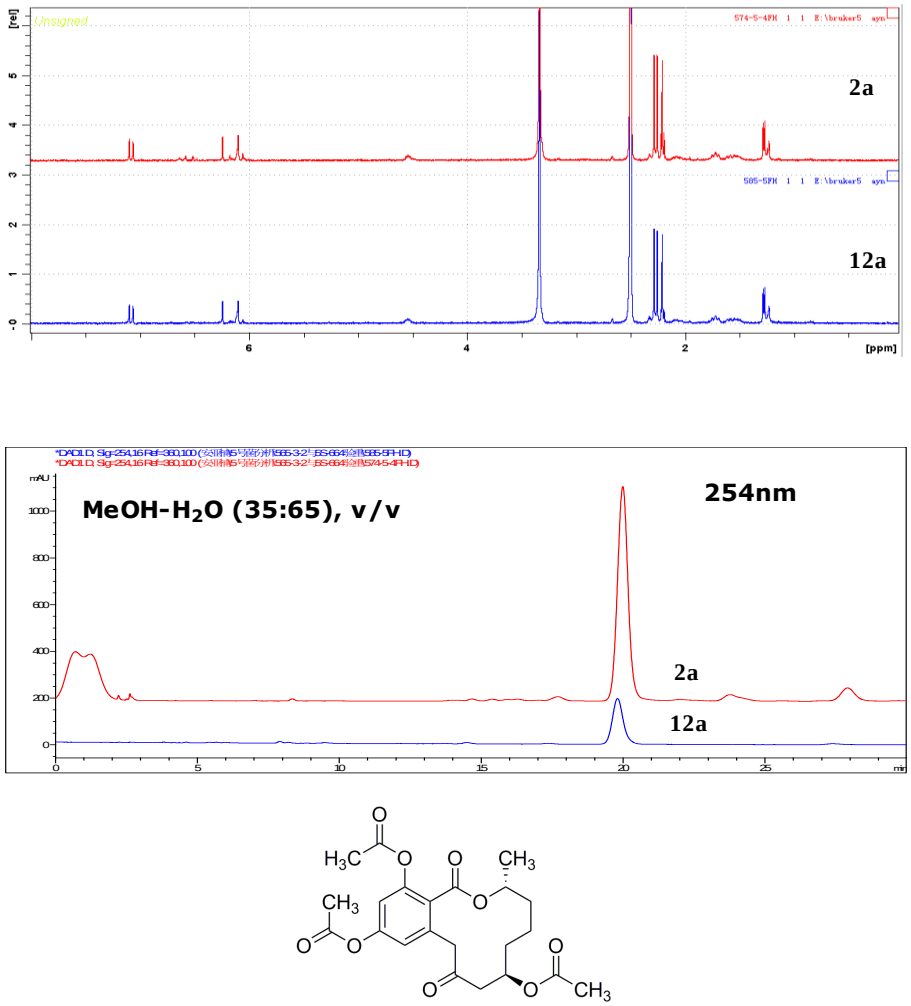

Figure S1 The ^{1}H-NMR spectra, HPLC analysis and structures of **2a** and **12a**

2.Alkaline hydrolysis of 3.

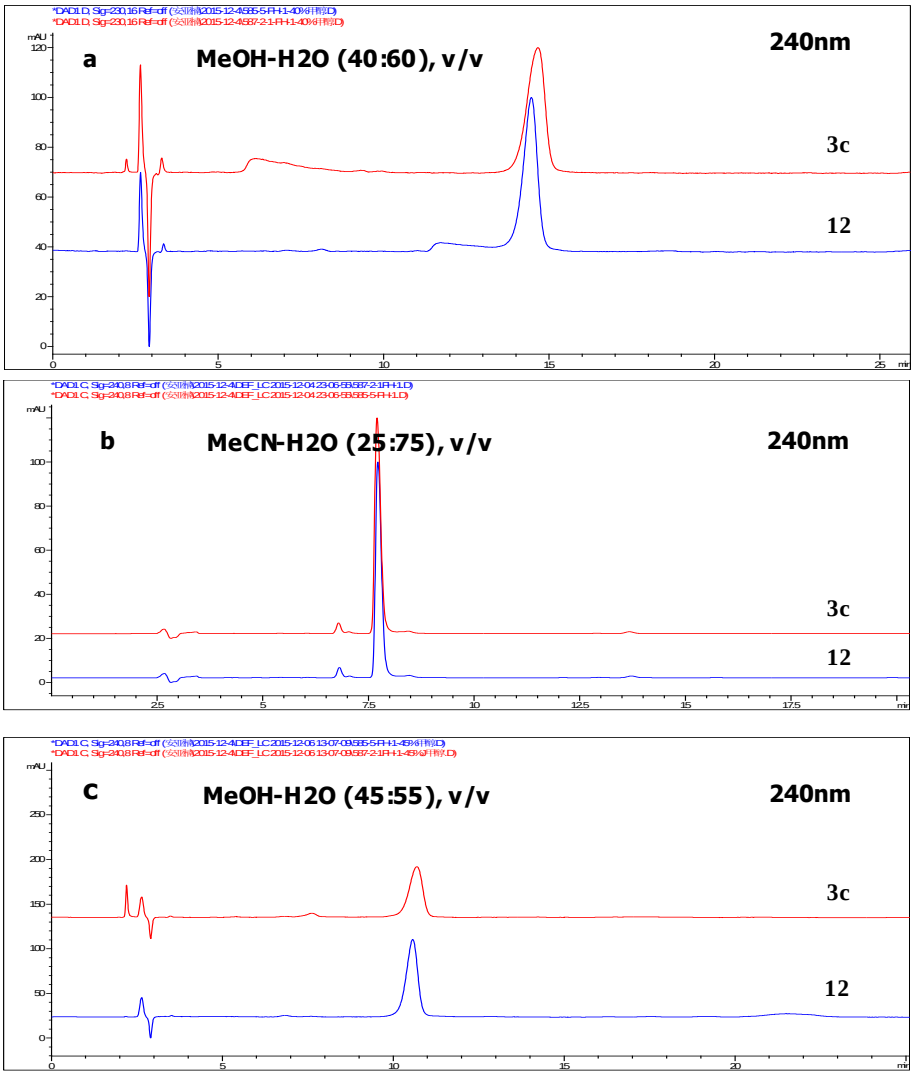

Figure S2 The alkaline hydrolysis product **3c** was compared with **12** by HPLC with three eluting systems (a: MeOH–H_2O (40:60, v/v), at a flow rate of 1 mL/min; b: MeOH–H_2O (45:55, v/v), at a flow rate of 1 mL/min; c: MeCN–H_2O (25:75, v/v), at a flow rate of 1 mL/min)

3. The spectra of compounds 1-6.

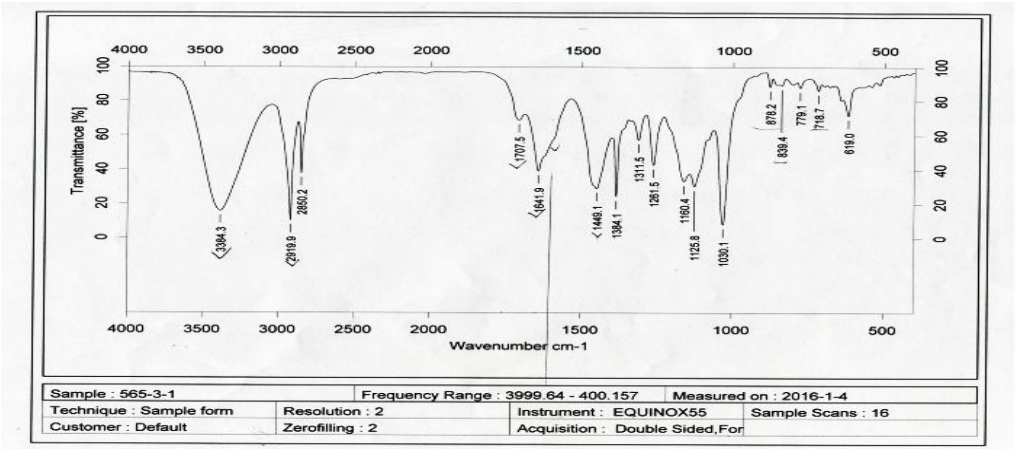

Figure S3 IR (KBr disc) spectrum of penicimenolide A (**1**)

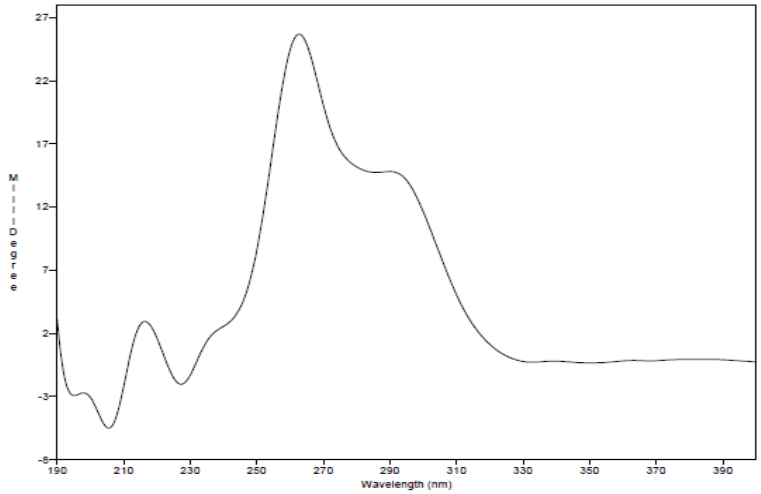

Figure S4 CD spectrum of penicimenolide A (**1**) in MeOH

Spectrum Peak Pick Report

FIELD FIELD TEXT

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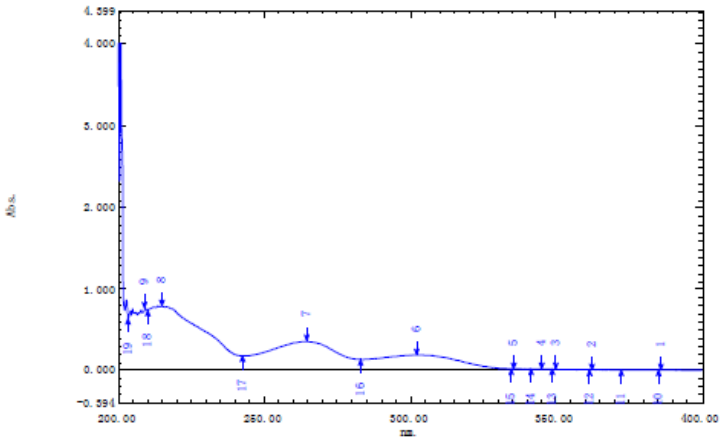

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采样间隔:	0.2
自动采样间隔:	停用
扫描模式:	单一的
试样准备属性	
重量:	0.5
体积 :	10
稀释:	
光程长:	407
附加信息:	
仪器属性	
仪器类型:	UV-1700
测定方式:	吸收值
狭缝宽:	1.0 nm
光源改变波长:	340.8 nm
D/R 转换:	标准
附件属性	
附件:	无

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1		386.00	.008	
2		362.40	.013	
3		350.00	.018	
4		345.00	.018	
5		335.40	.024	
6		302.20	.193	
7		264.60	.359	
8		214.80	.792	
9		209.00	.758	
10		385.20	.007	
11		372.40	.008	
12		361.60	.012	
13		348.60	.015	
14		341.60	.016	
15		334.60	.018	
16		283.00	.137	
17		242.60	.181	
18		210.00	.738	
19		203.20	.640	

Figure S5 UV spectrum of penicimenolide A (**1**) in MeOH

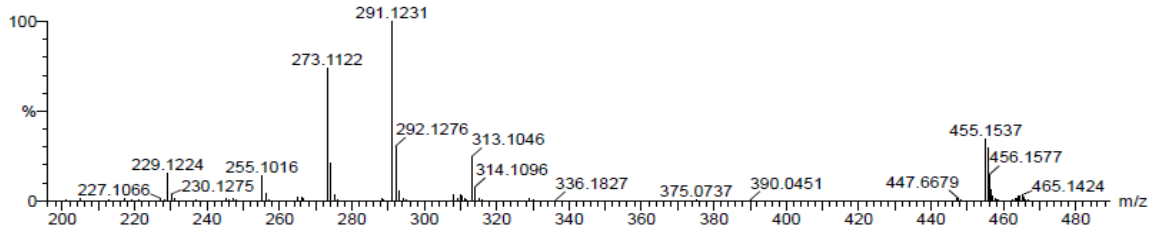
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 30.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
276 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 0-100 H: 0-200 N: 0-2 O: 0-100 Na: 0-1

565-3-1
2015090410 146 (1.182)

1: TOF MS ES+
1.92e+005



Minimum:				-1.5			
Maximum:		5.0	5.0	30.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
291.1231	291.1232	-0.1	-0.3	7.5	339.2	n/a	n/a	C16 H19 O5

Figure S6 HR-ESI-MS spectrum of penicimenolide A (**1**)

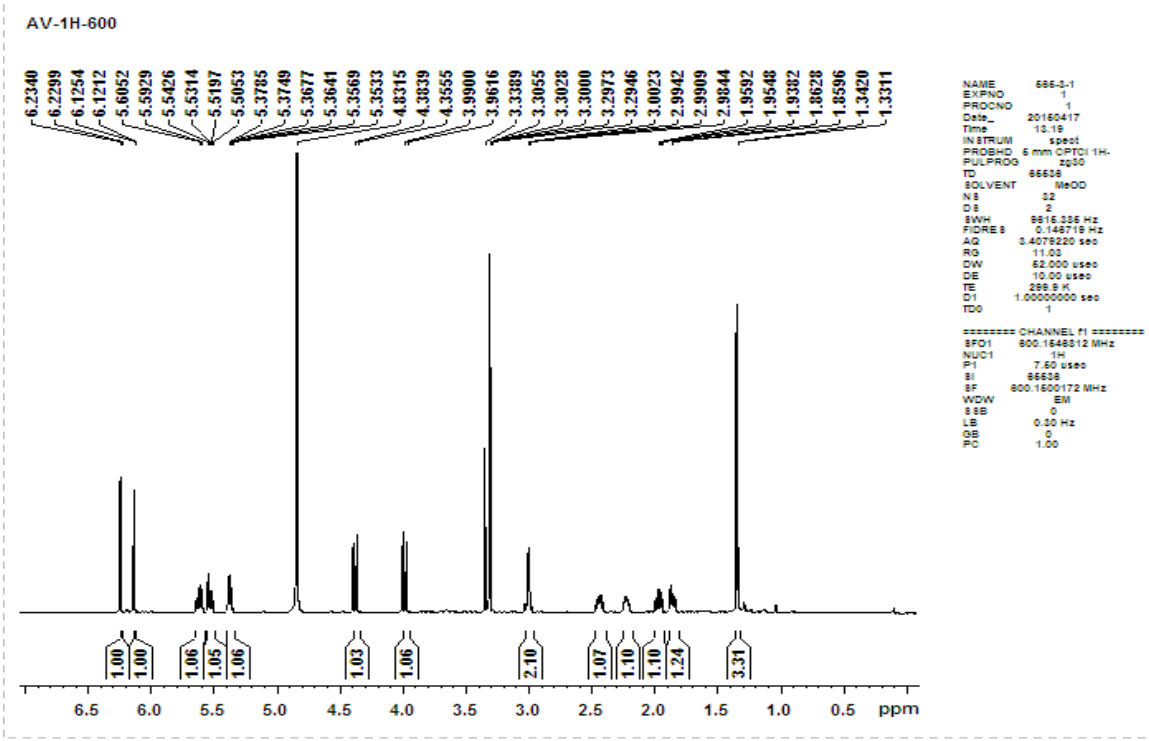


Figure S7 ^{1}H-NMR spectrum of **1** (600MHz, in CD_3OD)

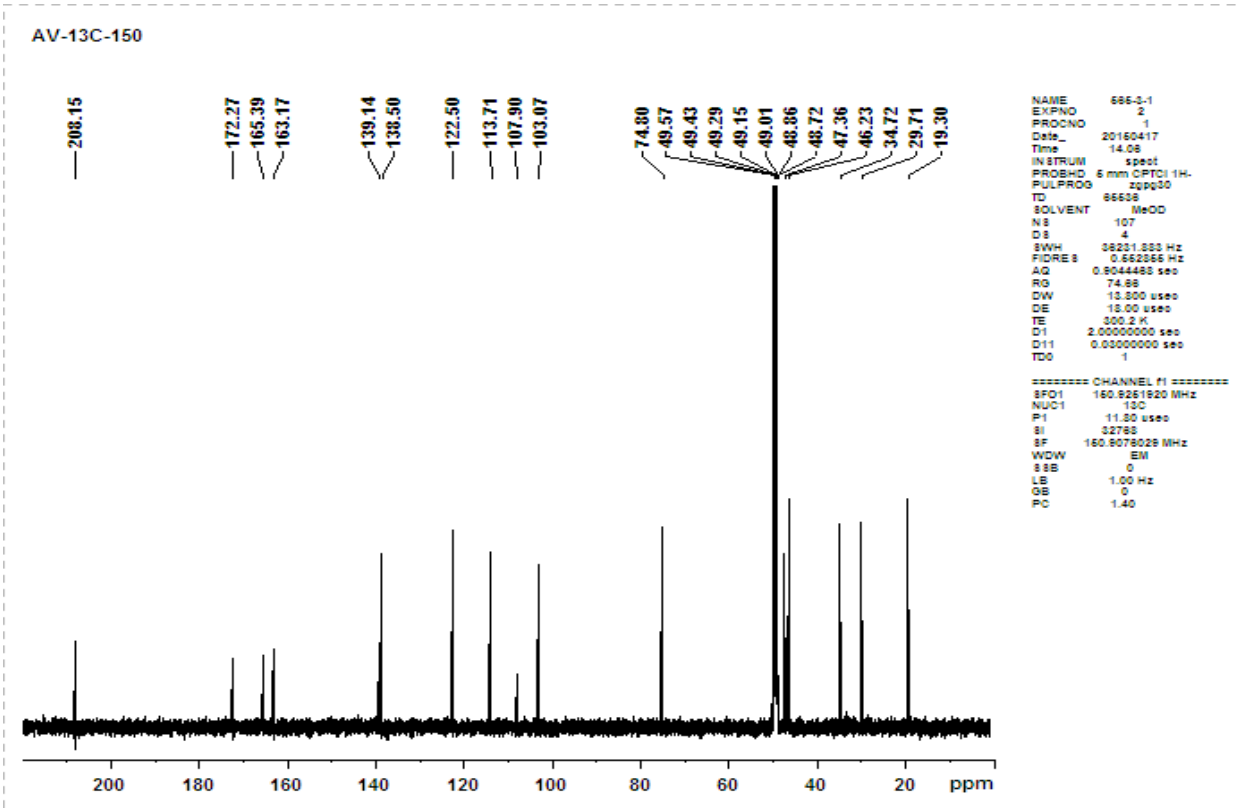


Figure S8 ^{13}C-NMR spectrum of **1** (150MHz, in CD_3OD)

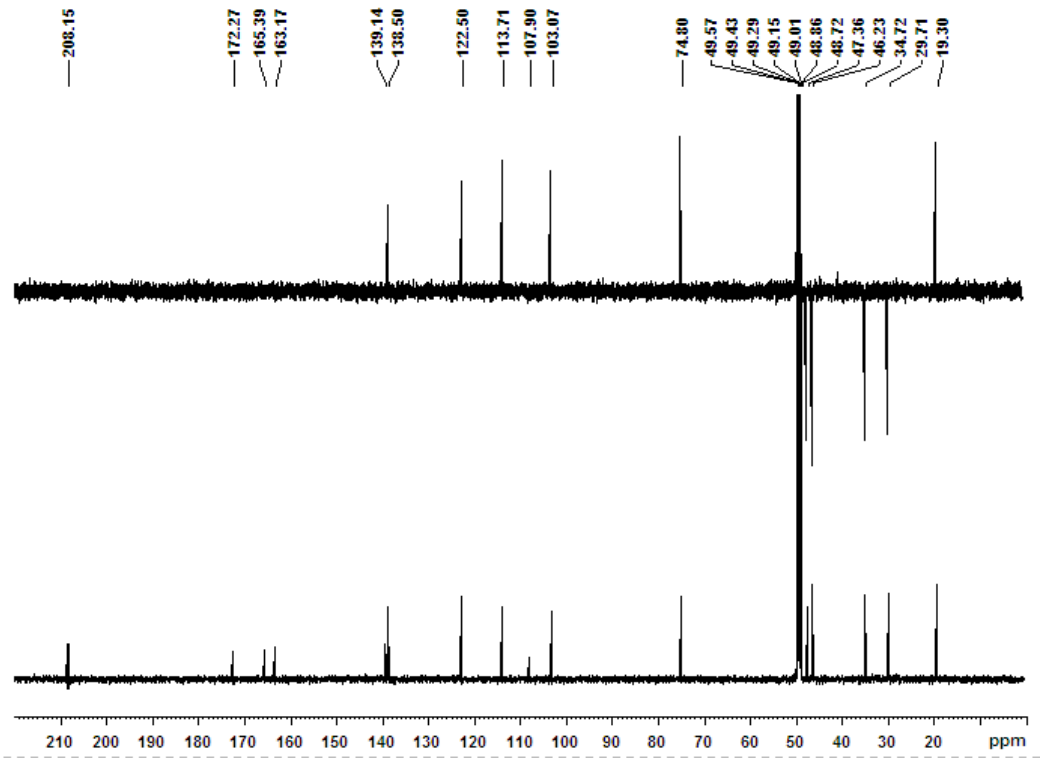


Figure S9 ^{13}C-NMR and DEPT 135 spectra of **1** (150MHz, in CD_3OD)

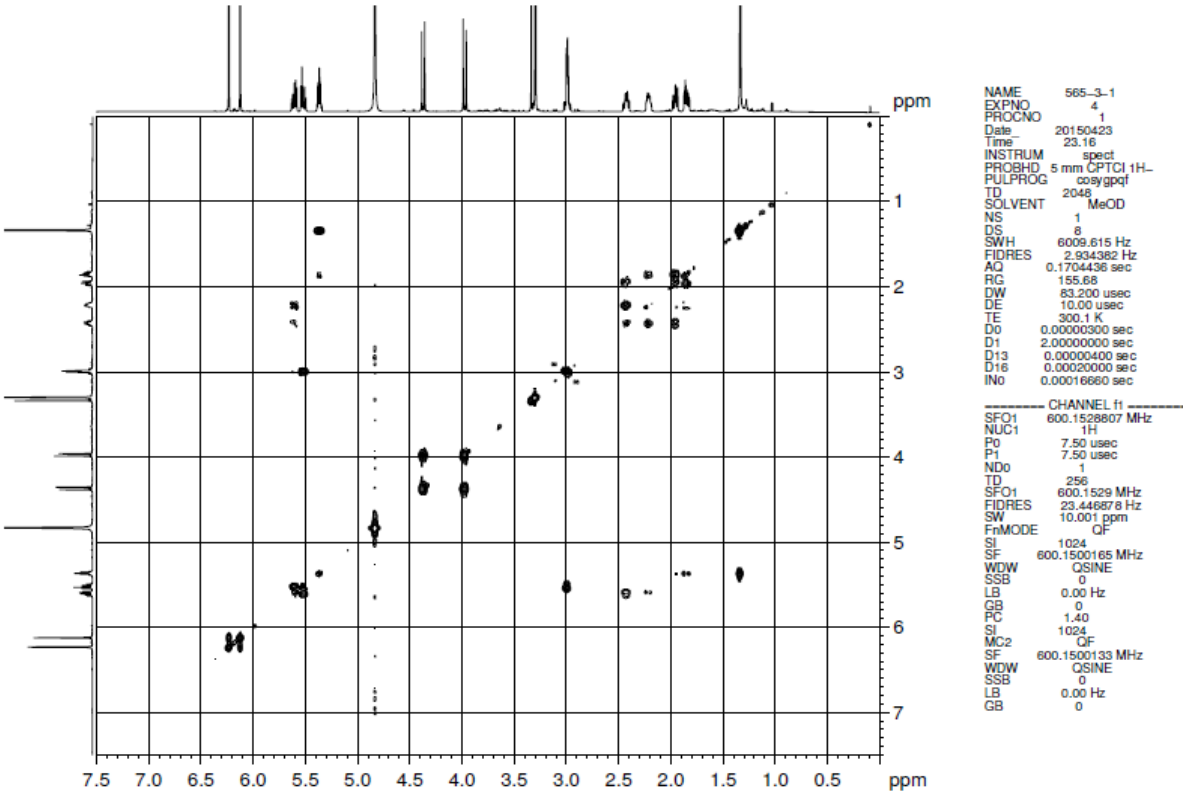


Figure S10 ^{1}H-^{1}H COSY spectrum of **1** (600MHz, in CD_3OD)

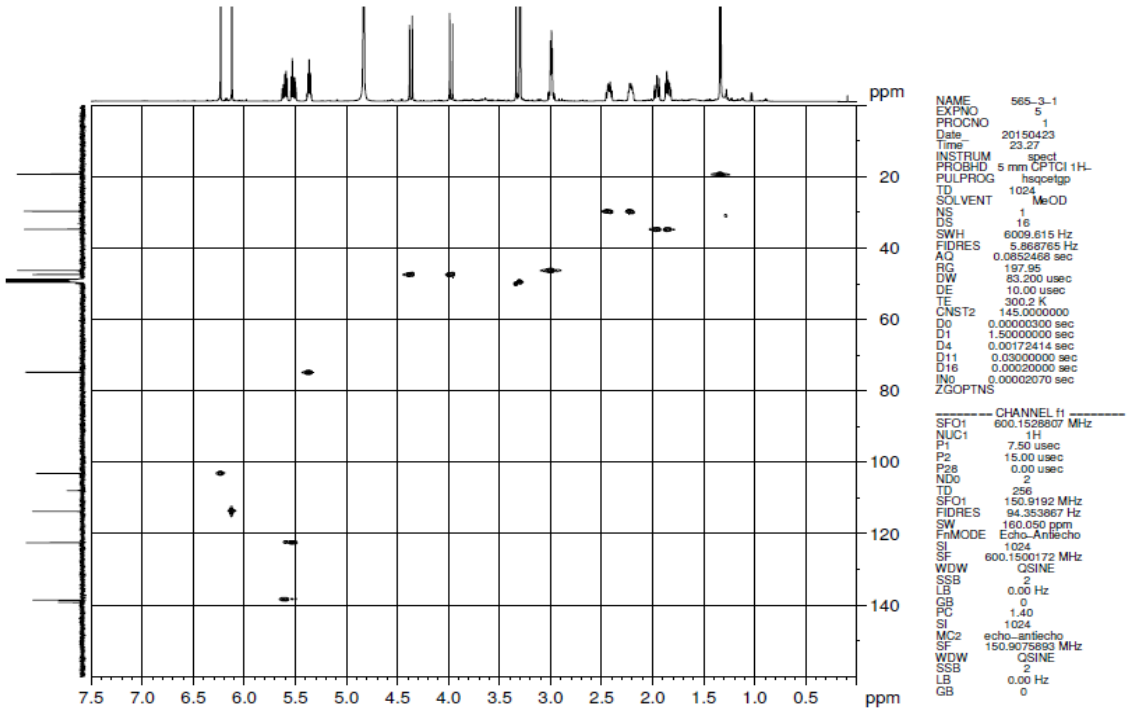


Figure S11 HSQC spectrum of **1** (600MHz, in CD_3OD)

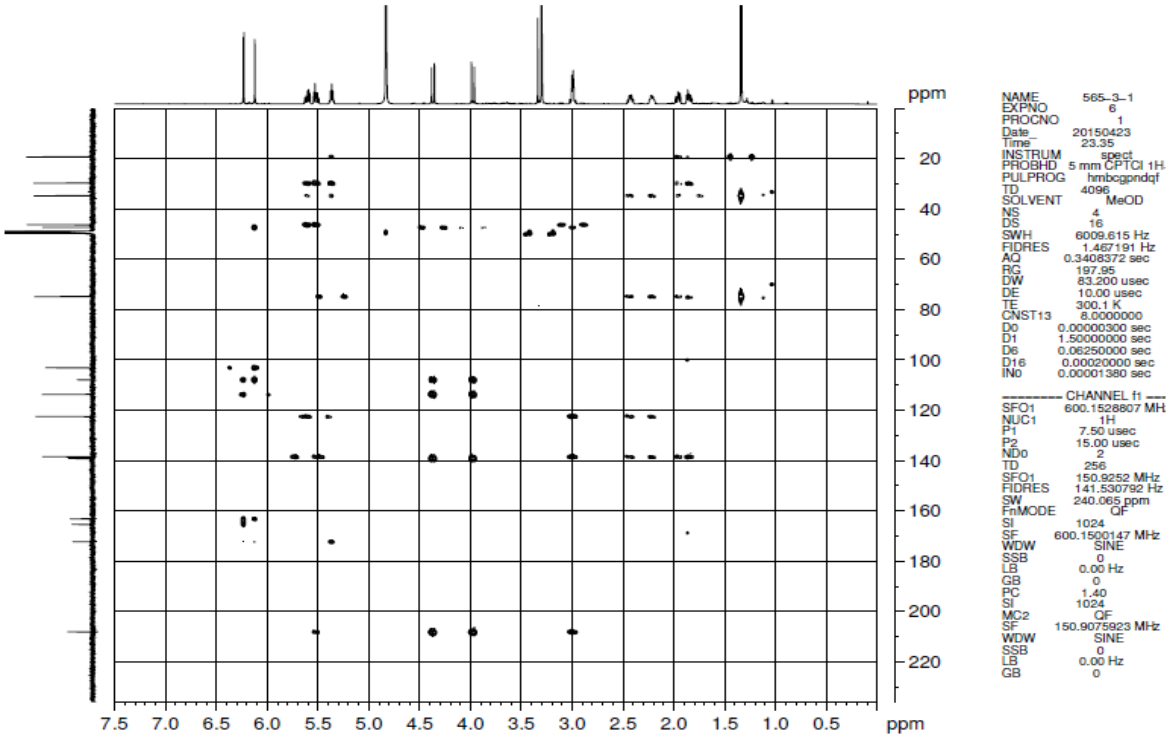


Figure S12 HMBC spectrum of **1** (600MHz, in CD_3OD)

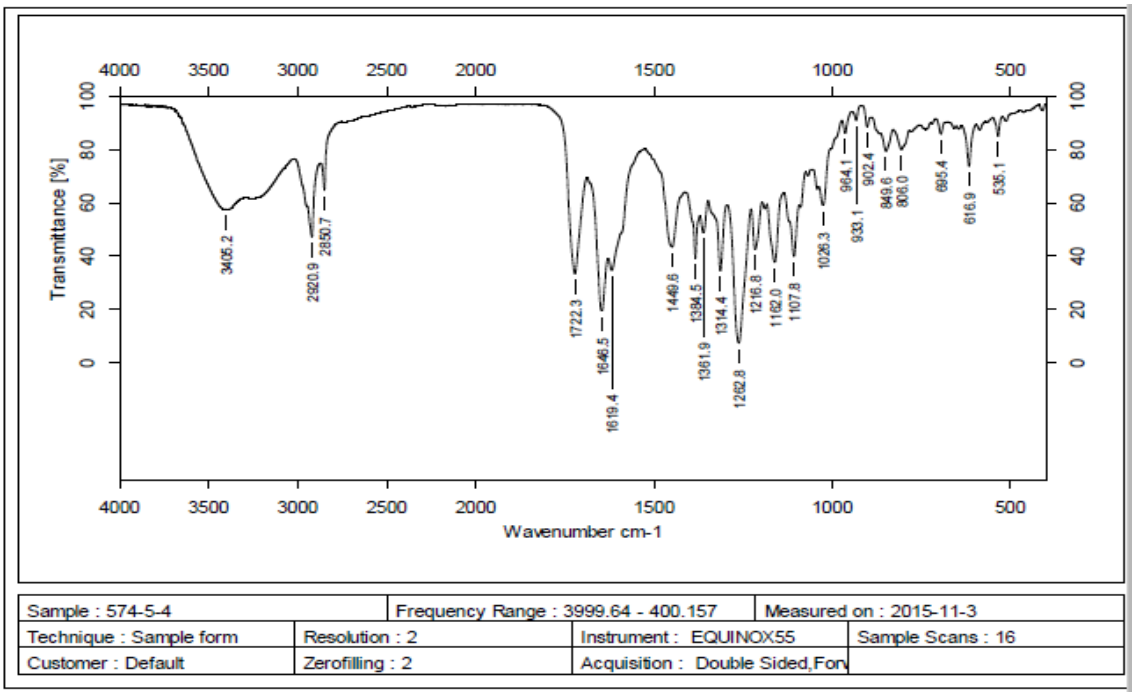


Figure S13 IR (KBr disc) spectrum of penicimenolide B (**2**)

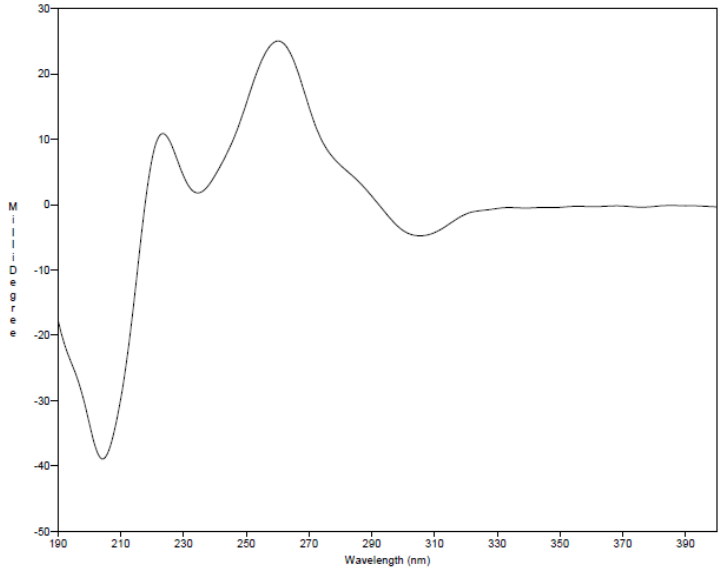


Figure S14 CD spectrum of penicimenolide B (**2**) in MeOH

Spectrum Peak Pick Report

FIELD FIELD TEXT

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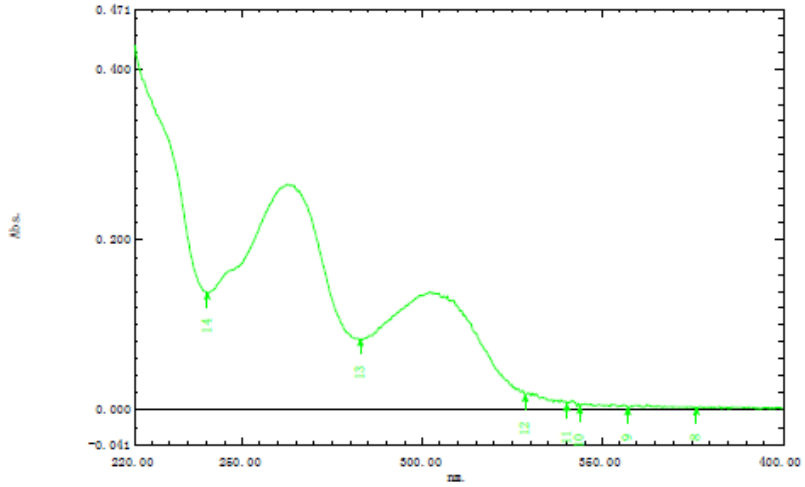


测定属性	
波长范围 (nm.):	220.00到400.00
扫描速度:	高速
采样间隔:	0.2
自动采样间隔:	启用
扫描模式:	单一的
试样准备属性	
重量:	0.5
体积 :	10
稀释:	
光程长:	407
附加信息:	
仪器属性	
仪器类型:	UV-1700
测定方式:	吸收值
狭缝宽:	1.0 nm
光源改变波长:	340.8 nm
S/R 转换:	标准

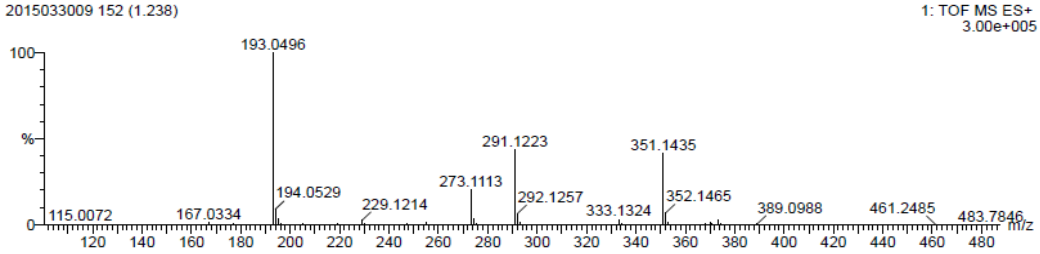
No.	P/V	Wavelength	Abs.	描述
1	P	382.80	.004	
2	P	362.80	.006	
3	P	352.60	.008	
4	P	341.80	.011	
5	P	329.40	.020	
6	P	302.00	.139	
7	P	262.40	.266	
8	V	376.20	.003	
9	V	357.20	.003	
10	V	343.80	.004	
11	V	340.20	.007	
12	V	328.60	.018	
13	V	282.60	.083	
14	V	240.20	.137	

Figure S15 UV spectrum of penicimenolide B (**2**) in MeOH

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
72 formula(e) evaluated with 2 results within limits (up to 10 best isotopic matches for each mass)
Elements Used:
C: 0-500 H: 0-1000 O: 0-200
574-5-4
2015033009 152 (1.238)



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
351.1435	351.1444	-0.9	-2.6	7.5	173.2	0.006	99.44	C18 H23 O7
	351.1385	5.0	14.2	16.5	178.4	5.187	0.56	C25 H19 O2

Figure S16 HR-ESI-MS spectrum of penicimenolide B (**2**)

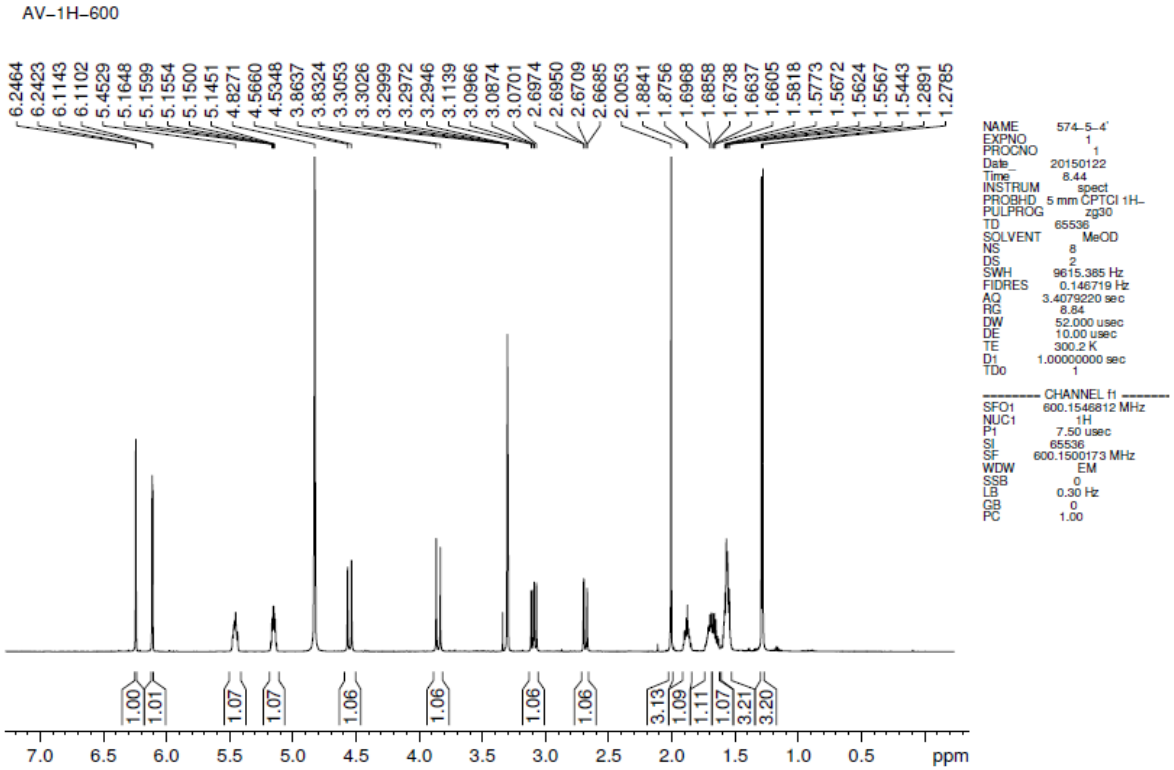


Figure S17 ^{1}H-NMR spectrum of **2** (600 MHz, in CD_3OD)

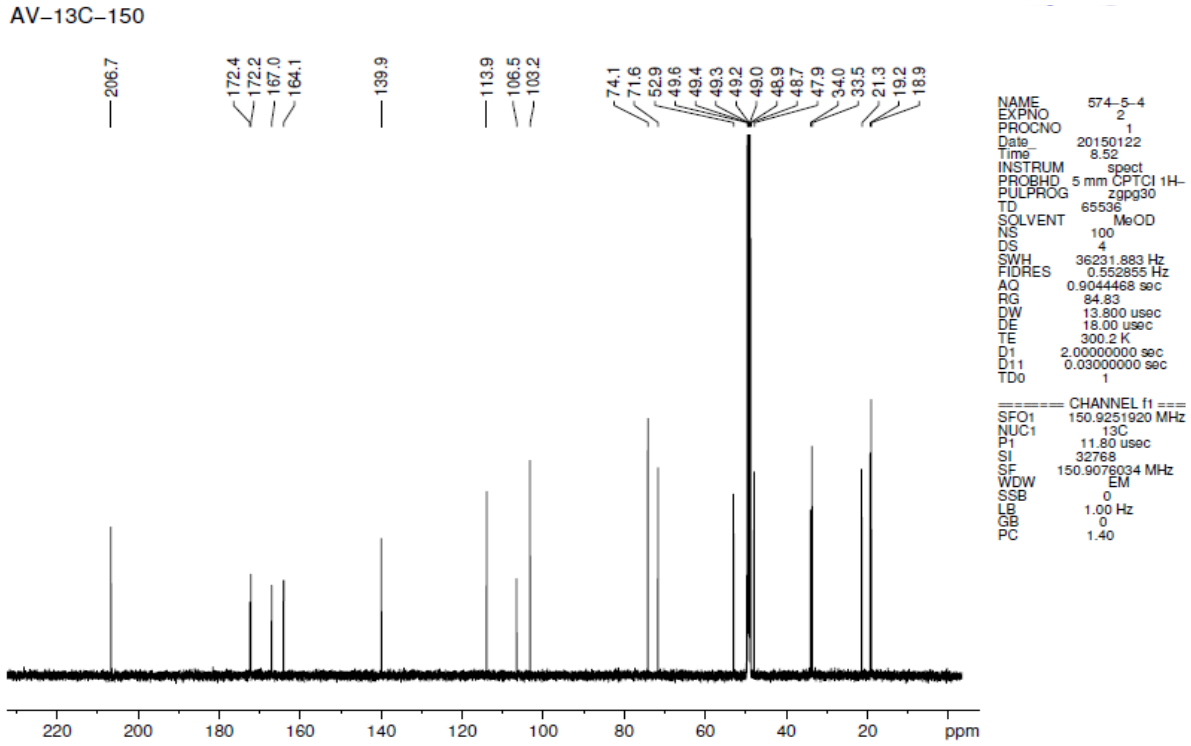


Figure S18 ^{13}C-NMR spectrum of **2** (150MHz, in CD_3OD)

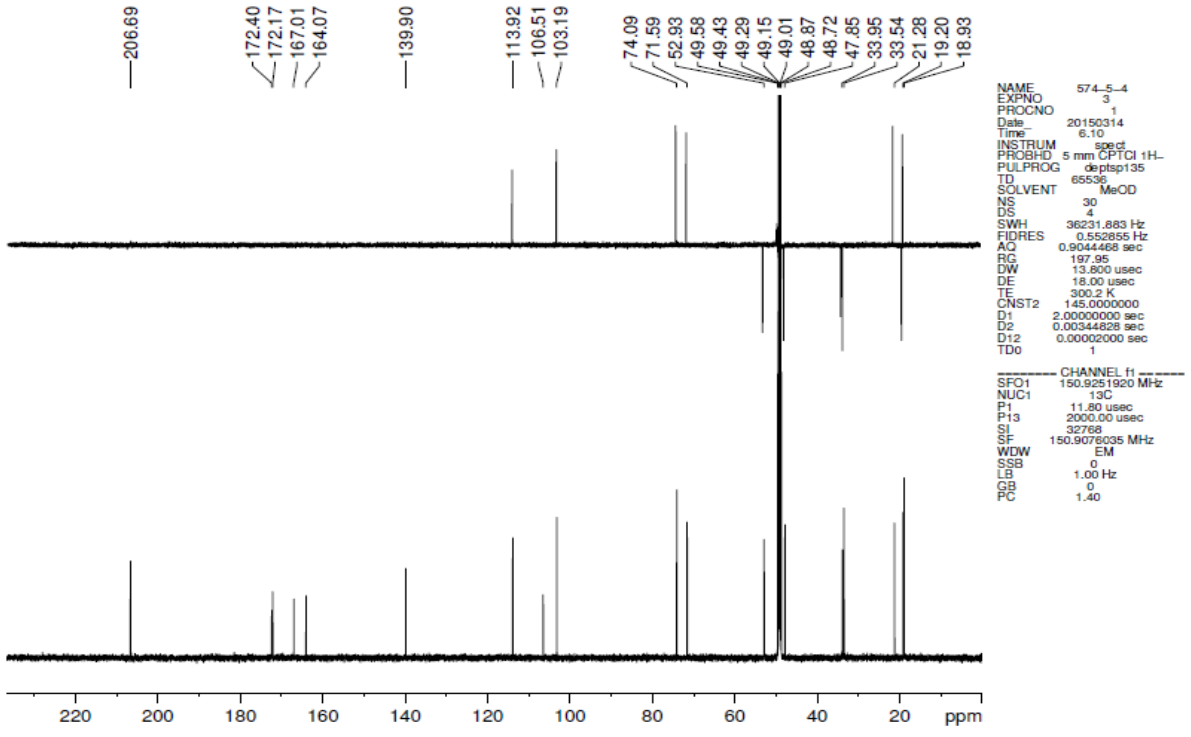


Figure S19 ^{13}C-NMR and DEPT 135 spectra of **2** (150MHz, in CD_3OD)

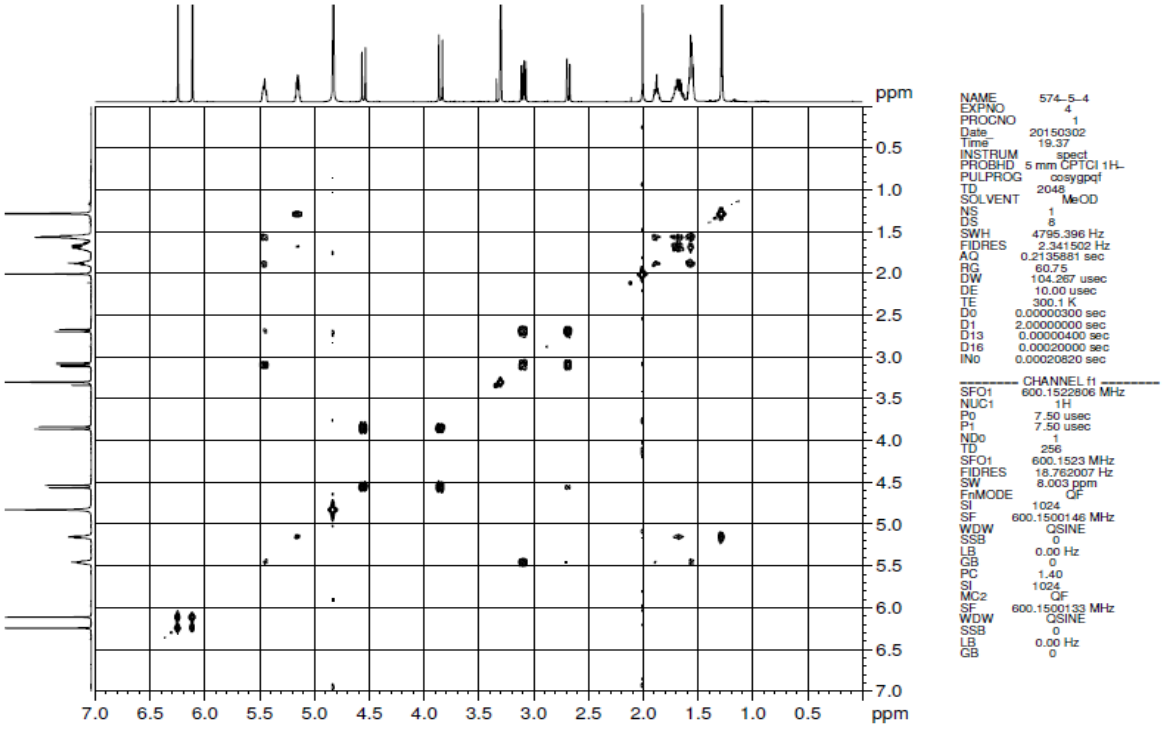


Figure S20 ^{1}H-^{1}H COSY spectrum of **2** (600MHz, in CD_3OD)

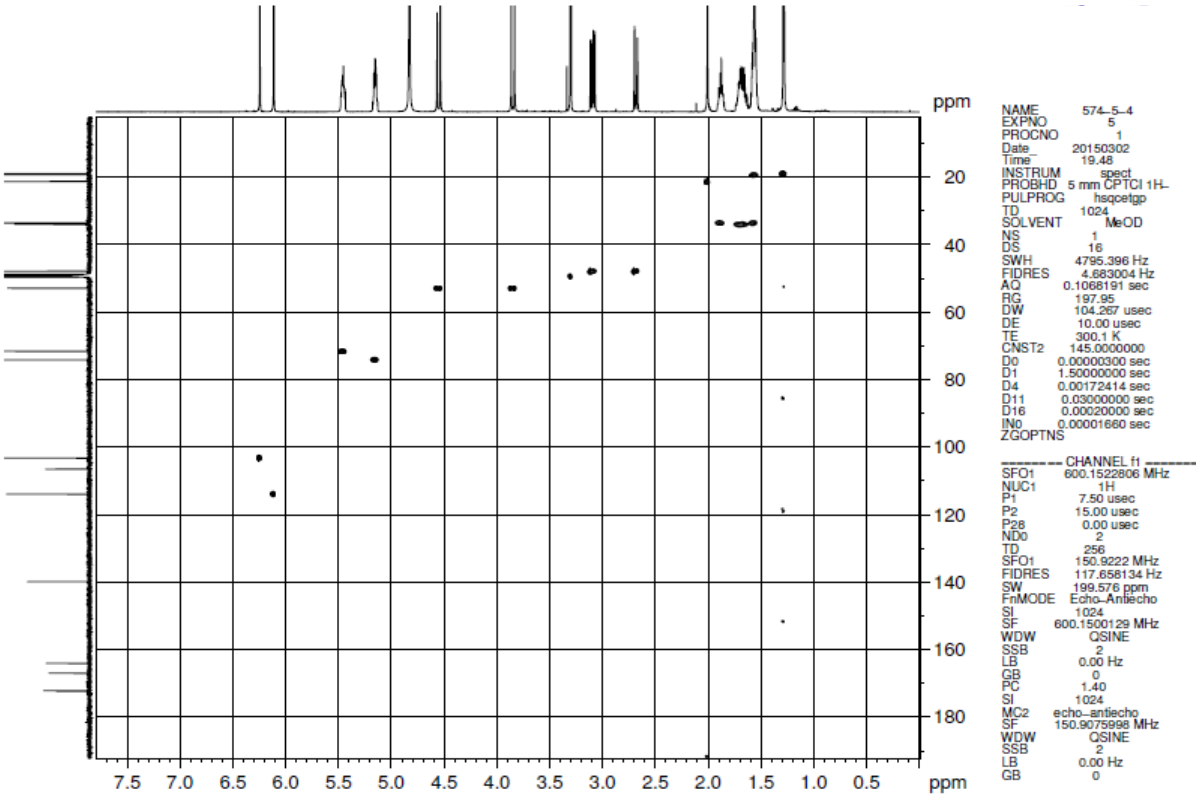


Figure S21 HSQC spectrum of **2** (600MHz, in CD_3OD)

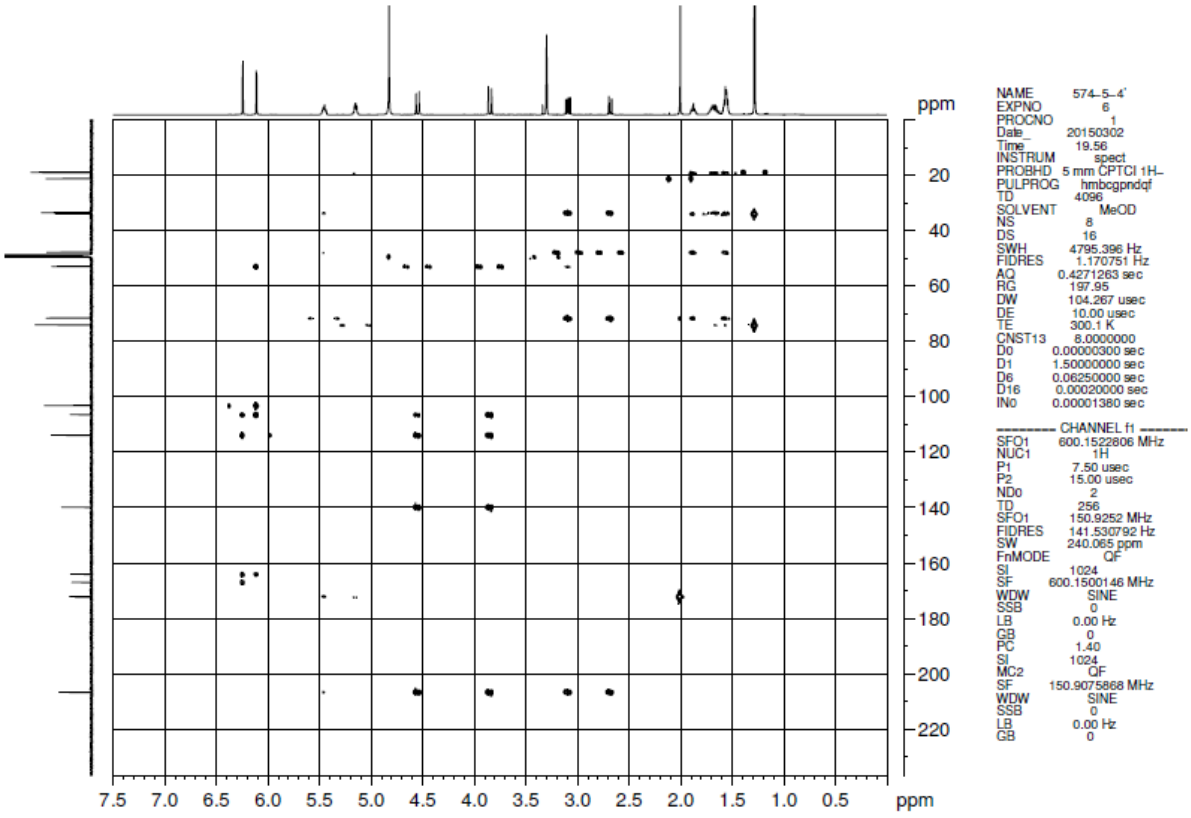


Figure S22 HMBC spectrum of **2** (600MHz, in CD)

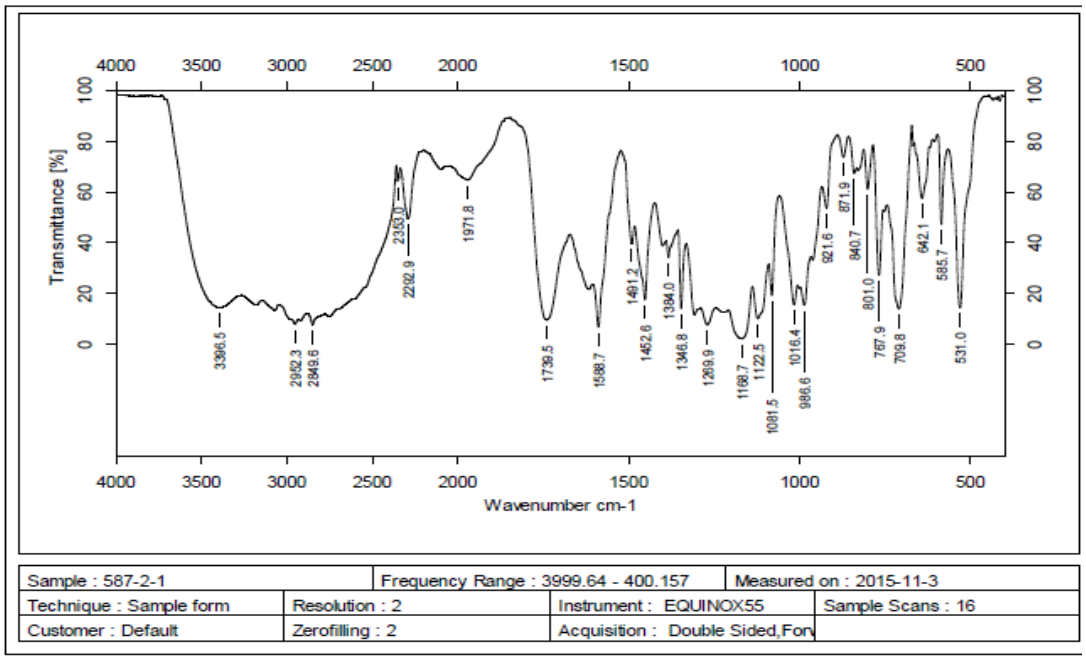


Figure S23 IR (KBr disc) spectrum of penicimenolide C (**3**)

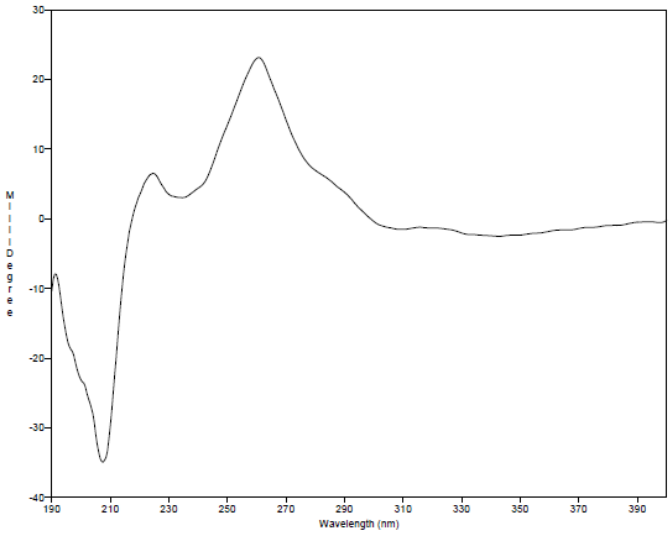


Figure S24 CD spectrum of penicimenolide C (**3**) in MeOH

Spectrum Peak Pick Report

FIELD FIELD TEXT

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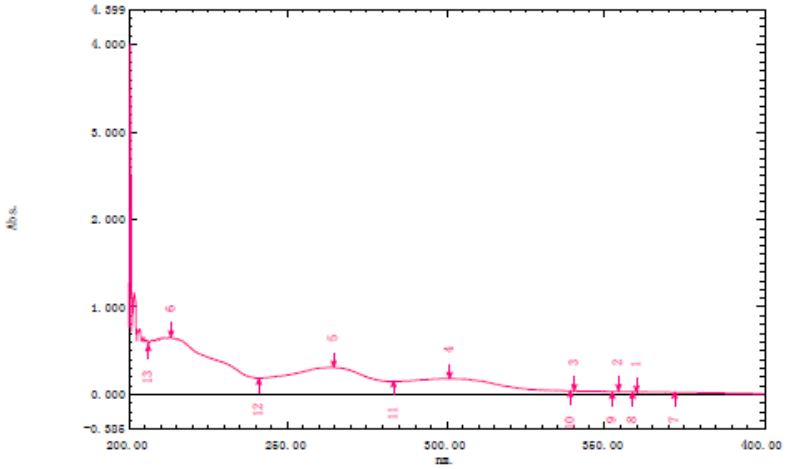

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2	P	354.40	.037	
3	P	340.40	.045	
4	P	301.00	.186	
5	P	264.60	.314	
6	P	213.20	.658	
7	V	372.00	.026	
8	V	358.80	.033	
9	V	352.20	.034	
10	V	339.20	.042	
11	V	283.20	.152	
12	V	241.00	.189	
13	V	206.20	.577	

测定属性
波长范围（nm.）: 200.00到400.00
扫描速度: 高速
采样间隔: 0.2
自动采样间隔: 停用
扫描模式: 单一的

试样准备属性
重量: 0.5
体积 : 10
稀释:
光程长: 407
附加信息:

仪器属性
仪器类型: UV-1700
测定方式: 吸收值
狭缝宽: 1.0 nm
光源改变波长: 340.8 nm
S/R 转换: 标准

附件属性
附件: 无

Figure S25 UV spectrum of penicimenolide C (**3**) in MeOH

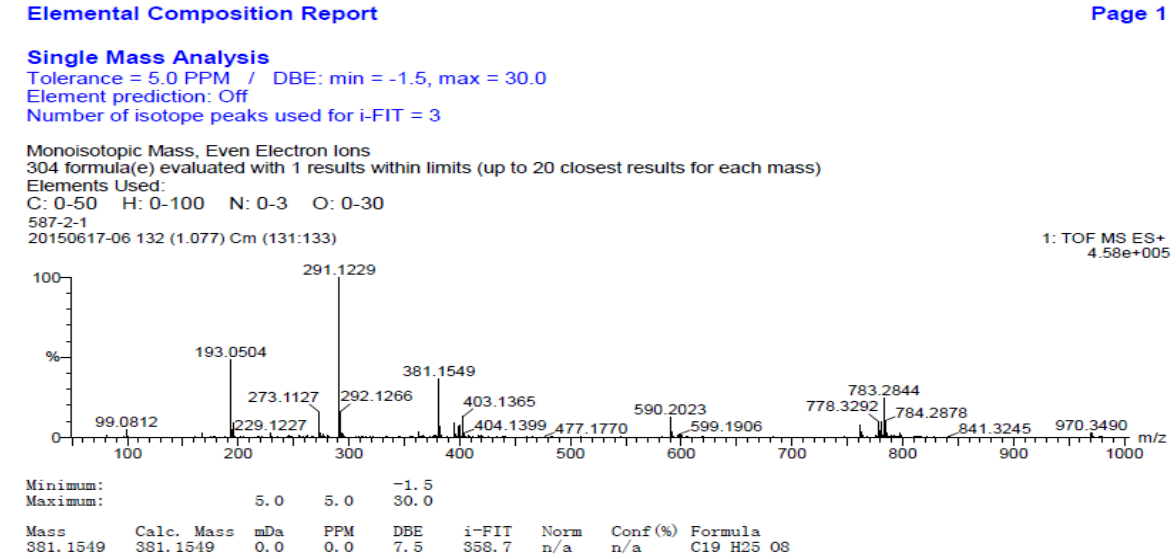


Figure S26 HR-ESI-MS spectrum of penicimenolide C (**3**)

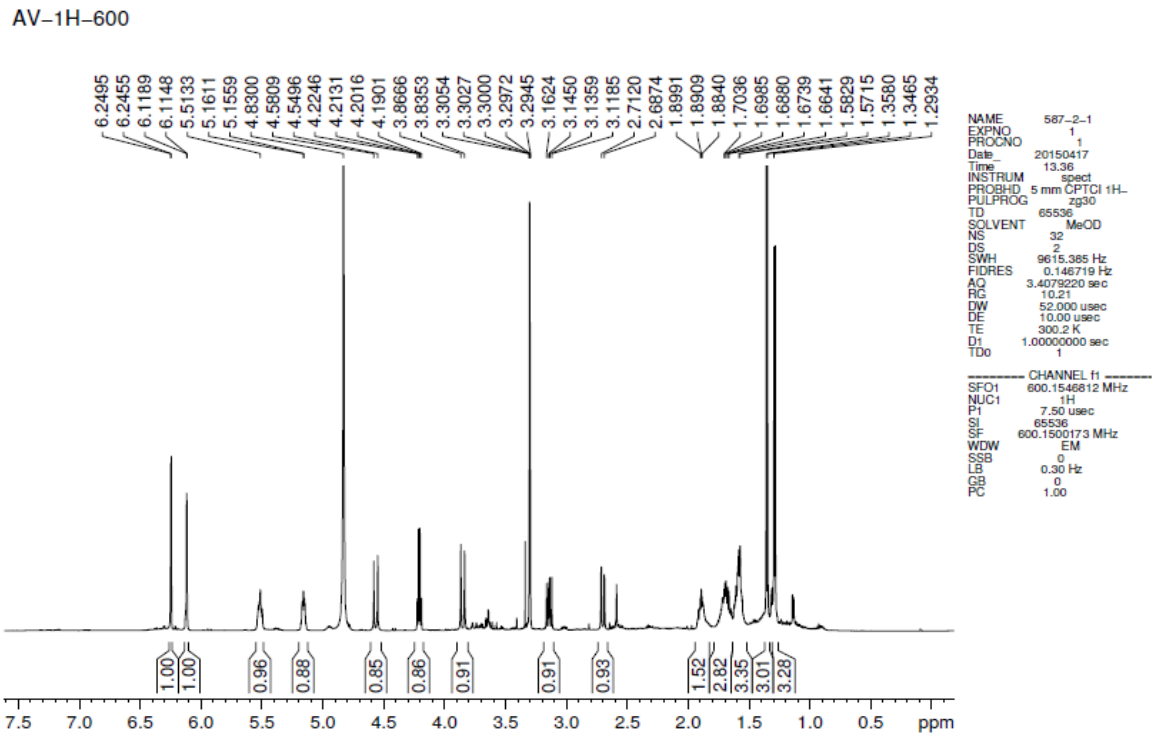


Figure S27 ^{1}H-NMR spectrum of **3** (600 MHz, in CD_3OD)

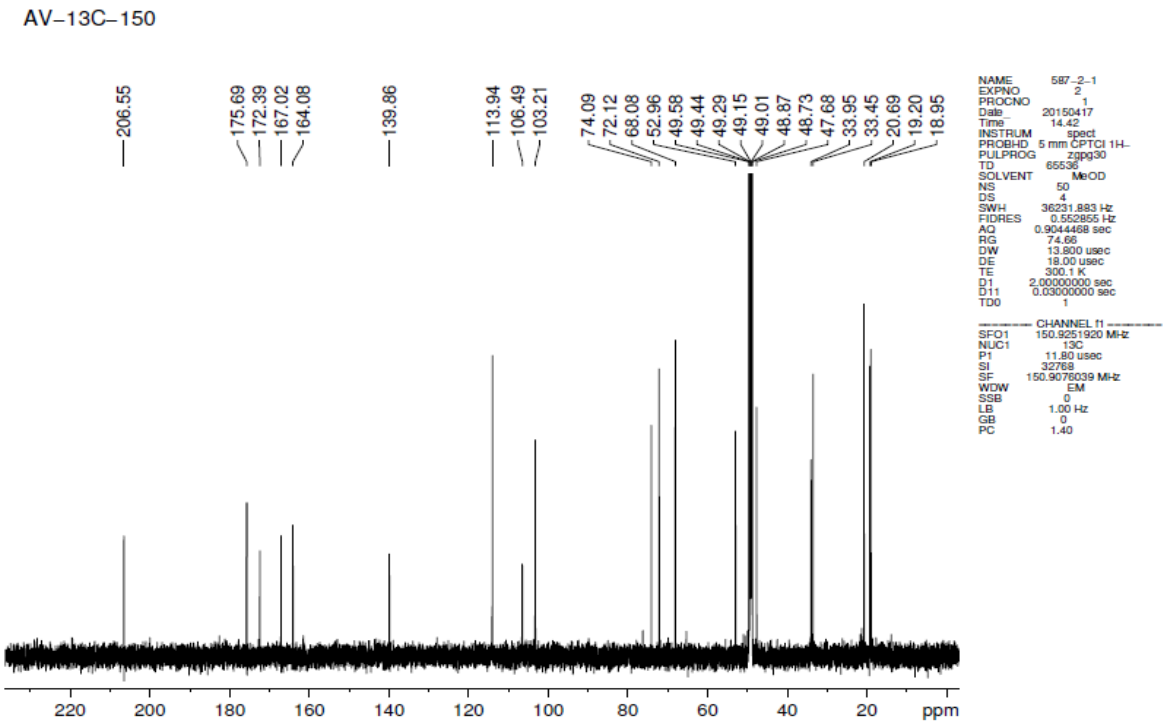


Figure S28 ^{13}C-NMR spectrum of **3** (150MHz, in CD_3OD)

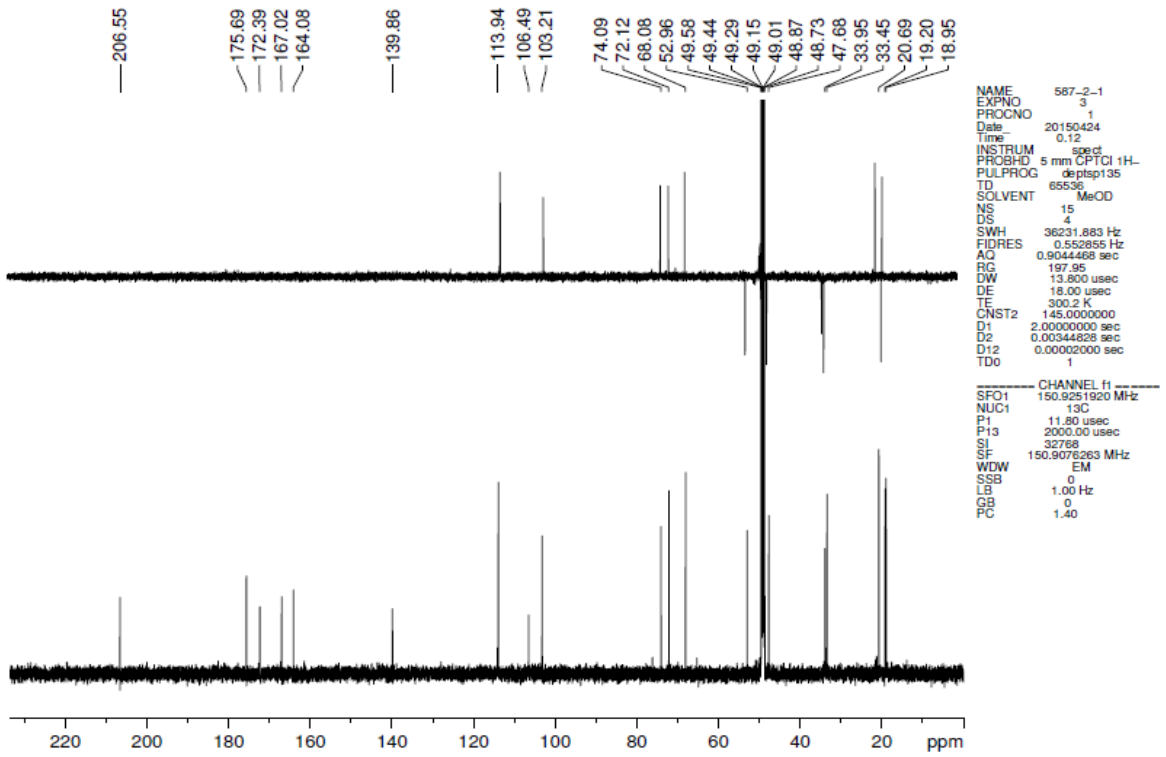


Figure S29 ^{13}C-NMR and DEPT 135 spectra of **3** (150MHz, in CD_3OD)

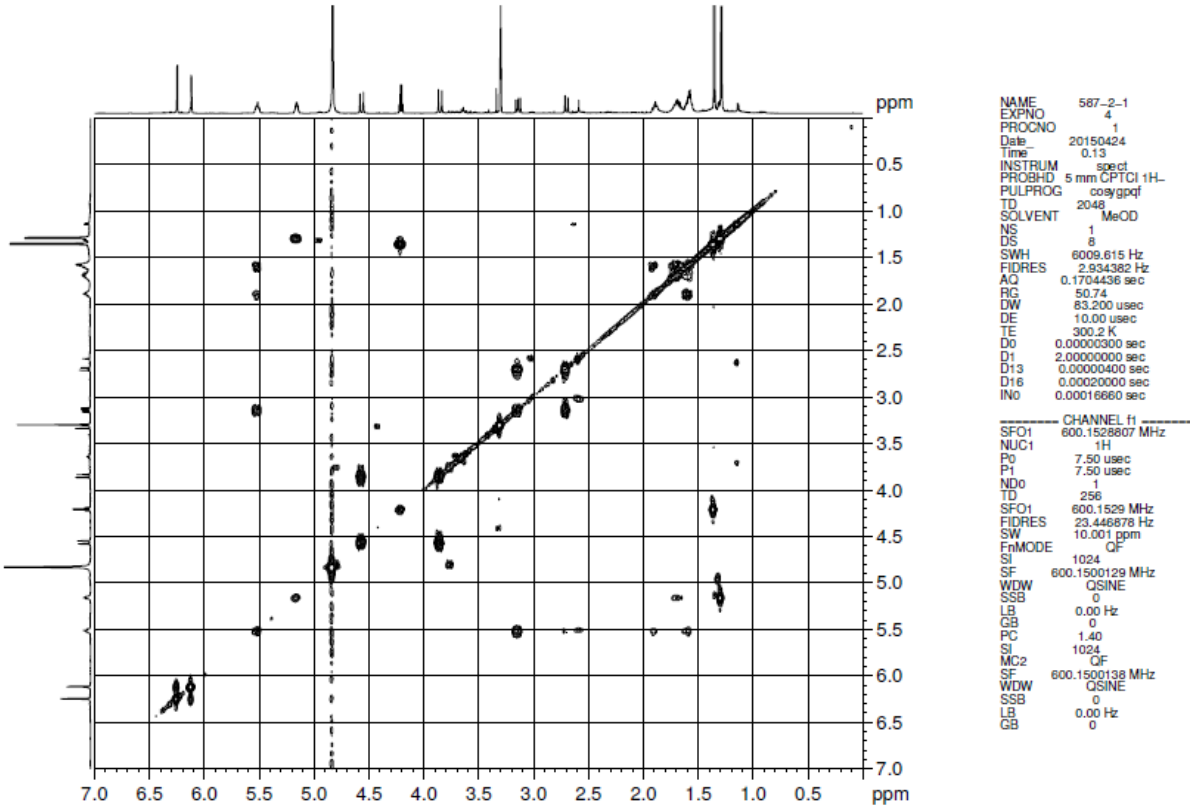


Figure S30 ^{1}H-^{1}H COSY spectrum of **3** (600MHz, in CD_3OD)

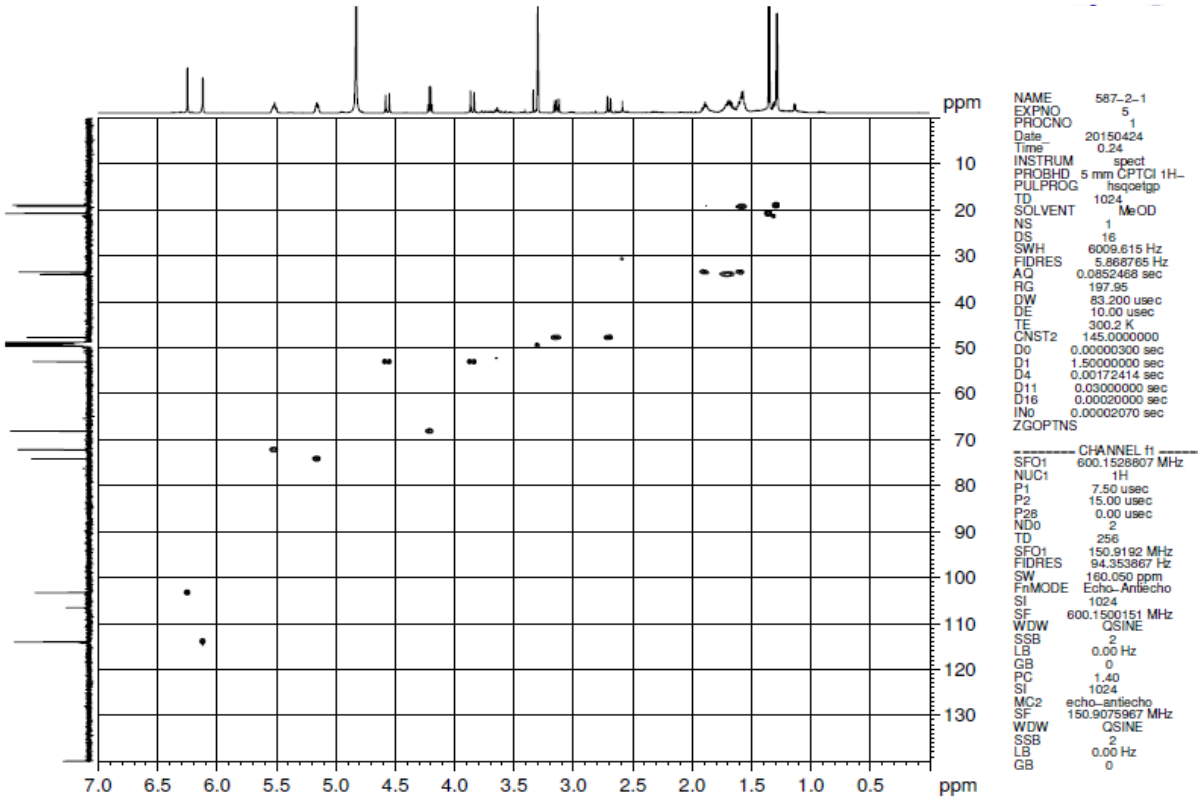


Figure S31 HSQC spectrum of **3** (600MHz, in CD_3OD)

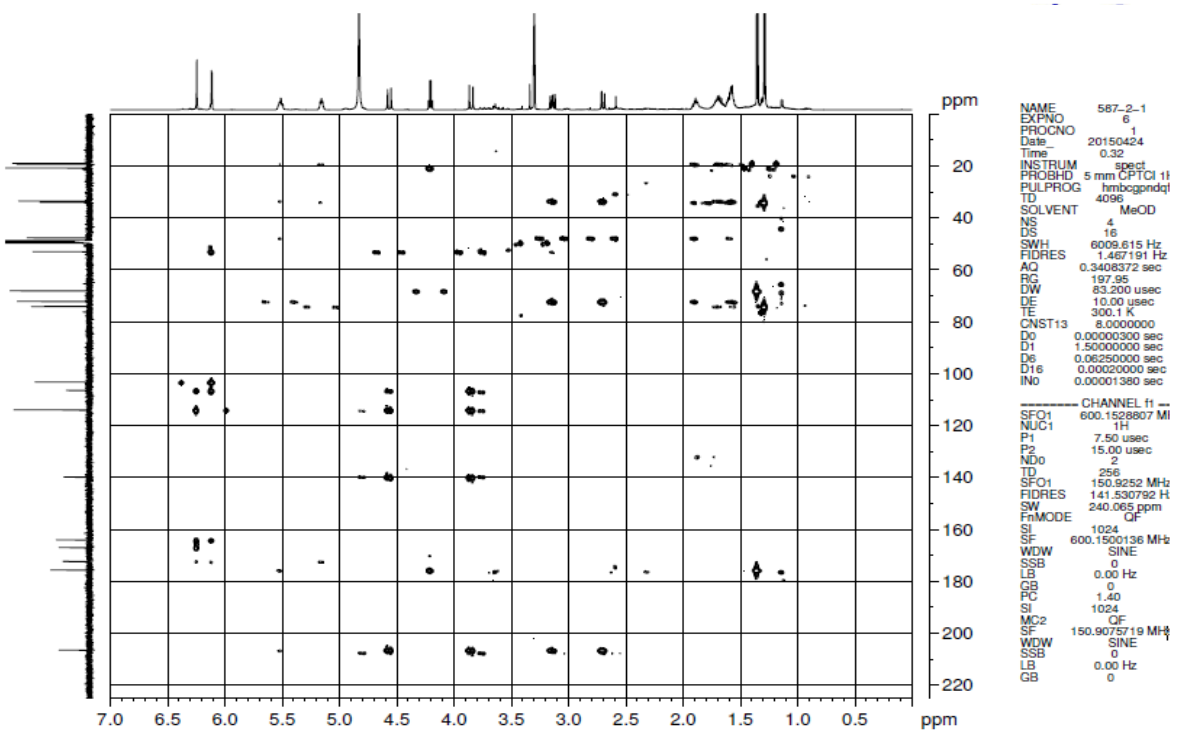


Figure S32 HMBC spectrum of **3** (600MHz, in CD_3OD)

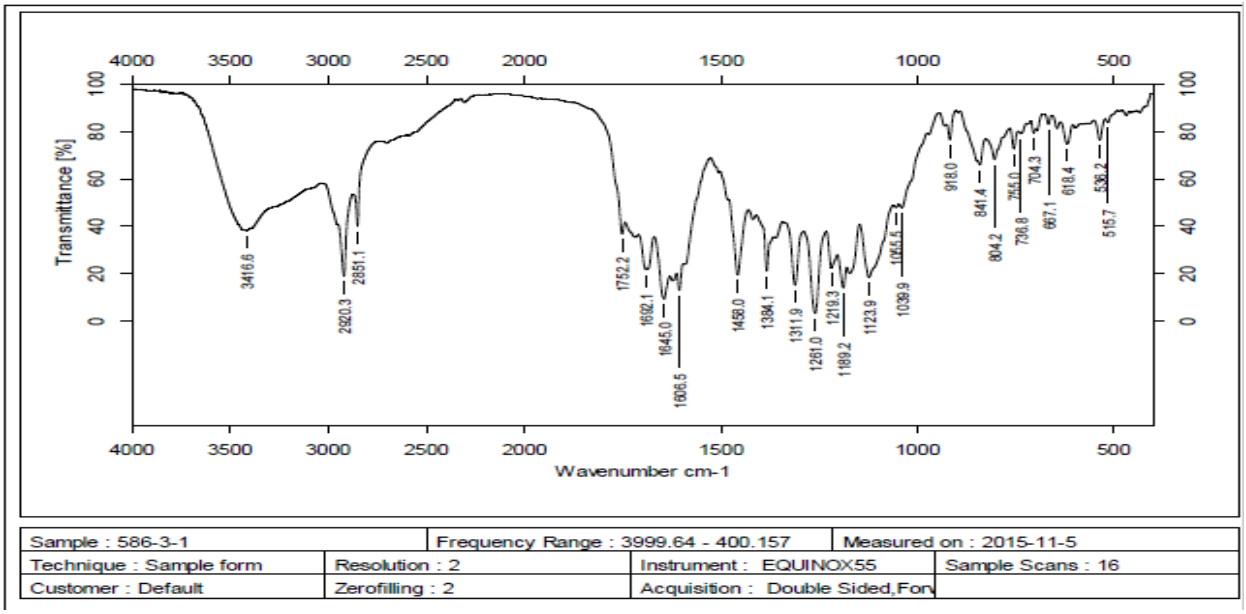


Figure S33 IR (KBr disc) spectrum of penicimenolide D (**4**)

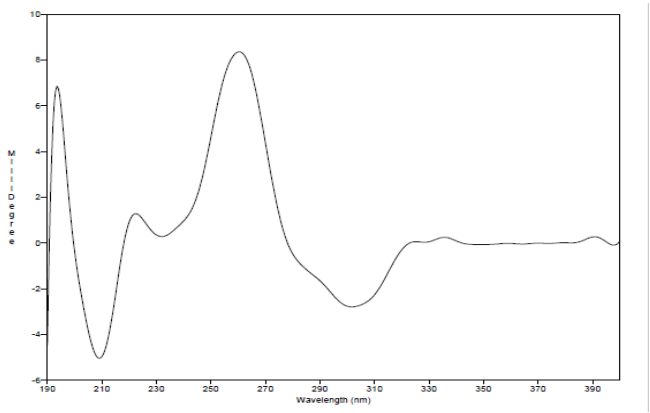


Figure S34 CD spectrum of penicimenolide D (**4**) in MeOH

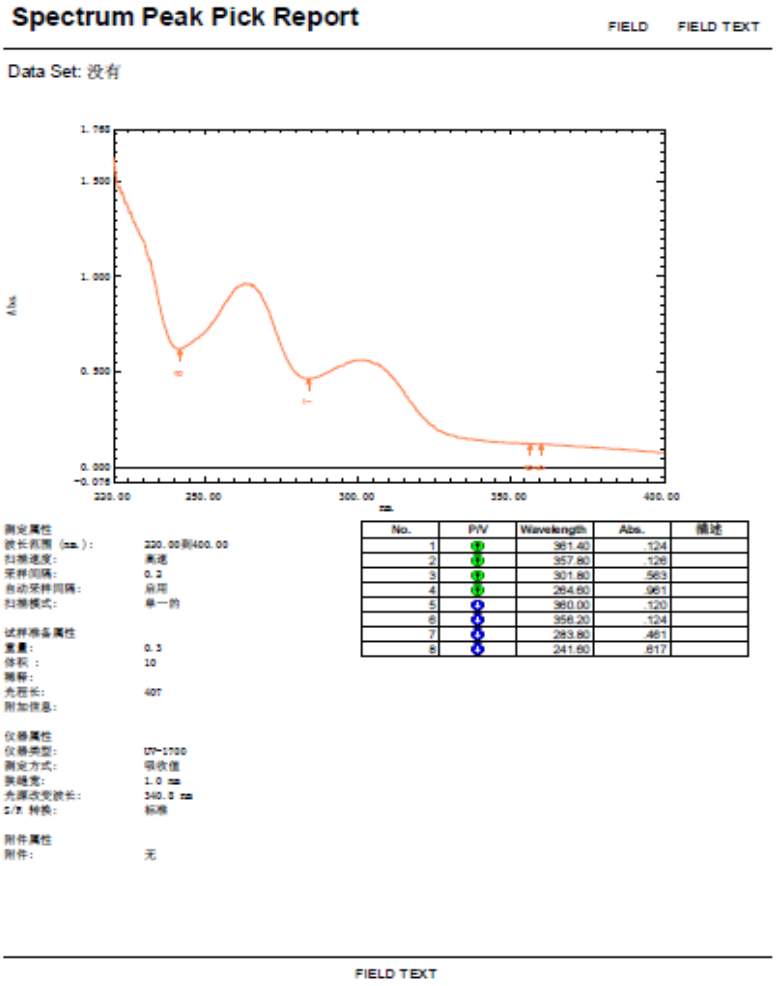


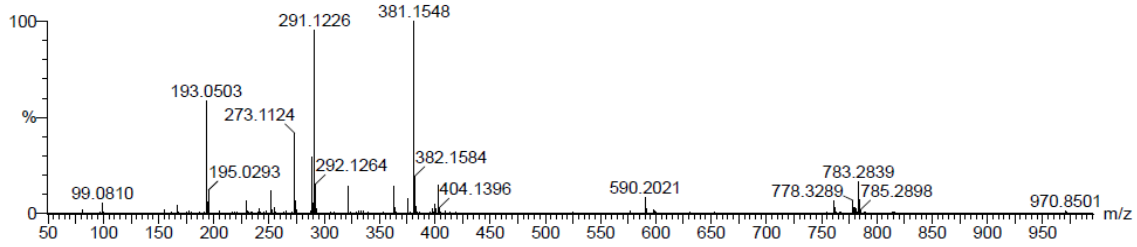
Figure S35 UV spectrum of penicimenolide D (**4**) in MeOH

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 30.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
304 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 0-50 H: 0-100 N: 0-3 O: 0-30

586-3-1
20150617-08 121 (0.984) Cm (120:124)

1: TOF MS ES+
7.19e+005



Minimum:				-1.5				
Maximum:		5.0	5.0	30.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
381.1548	381.1549	-0.1	-0.3	7.5	619.6	n/a	n/a	C19 H25 O8

Figure S36 HR-ESI-MS spectrum of penicimenolide D (**4**)

AV-1H-600

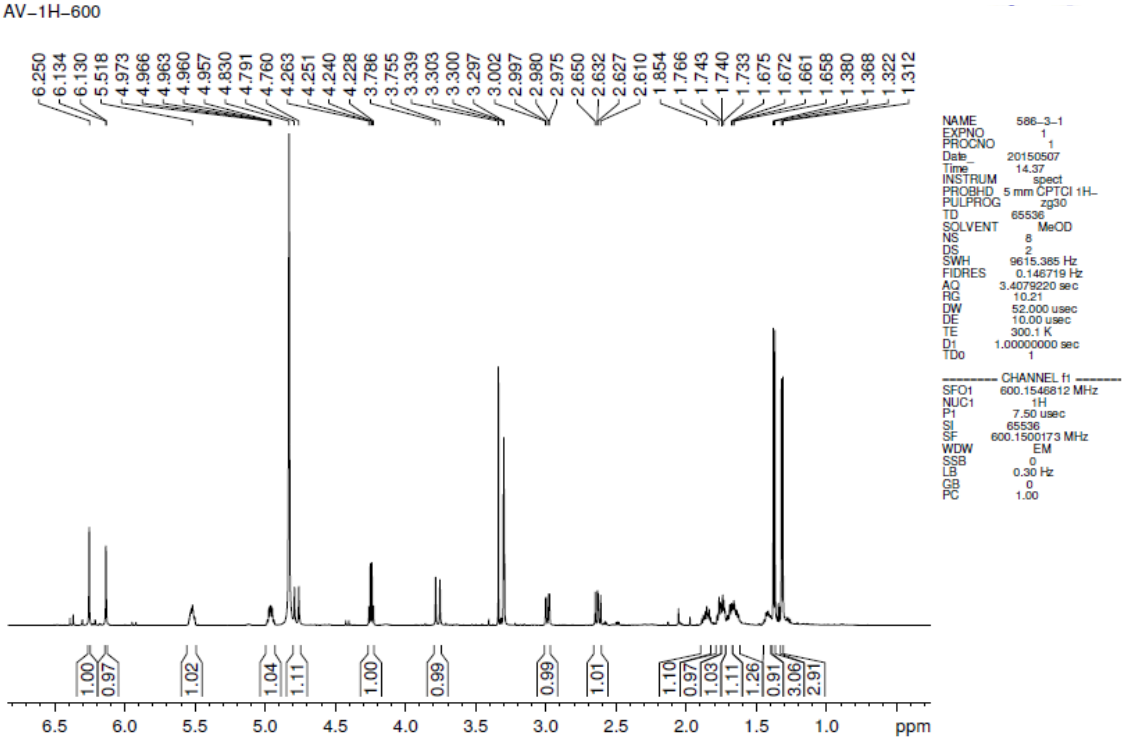


Figure S37 ^{1}H-NMR spectrum of **4** (600 MHz, in CD_3OD)

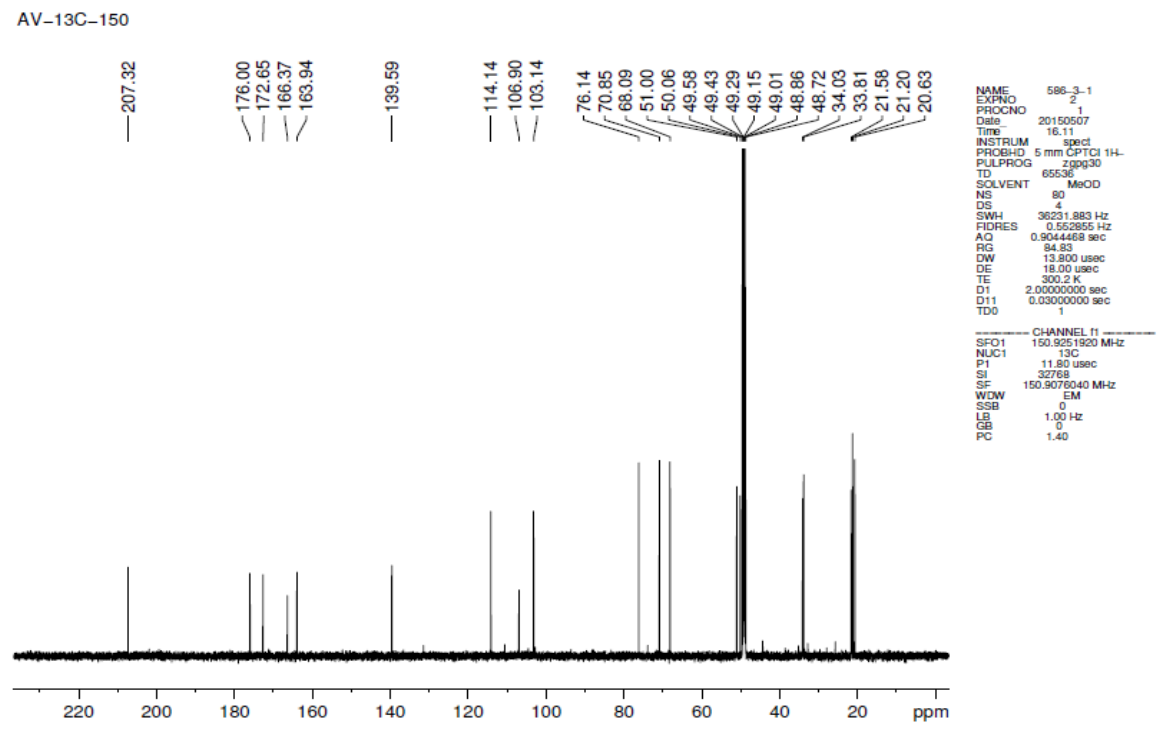


Figure S38 ^{13}C-NMR spectrum of **4** (150MHz, in CD_3OD)

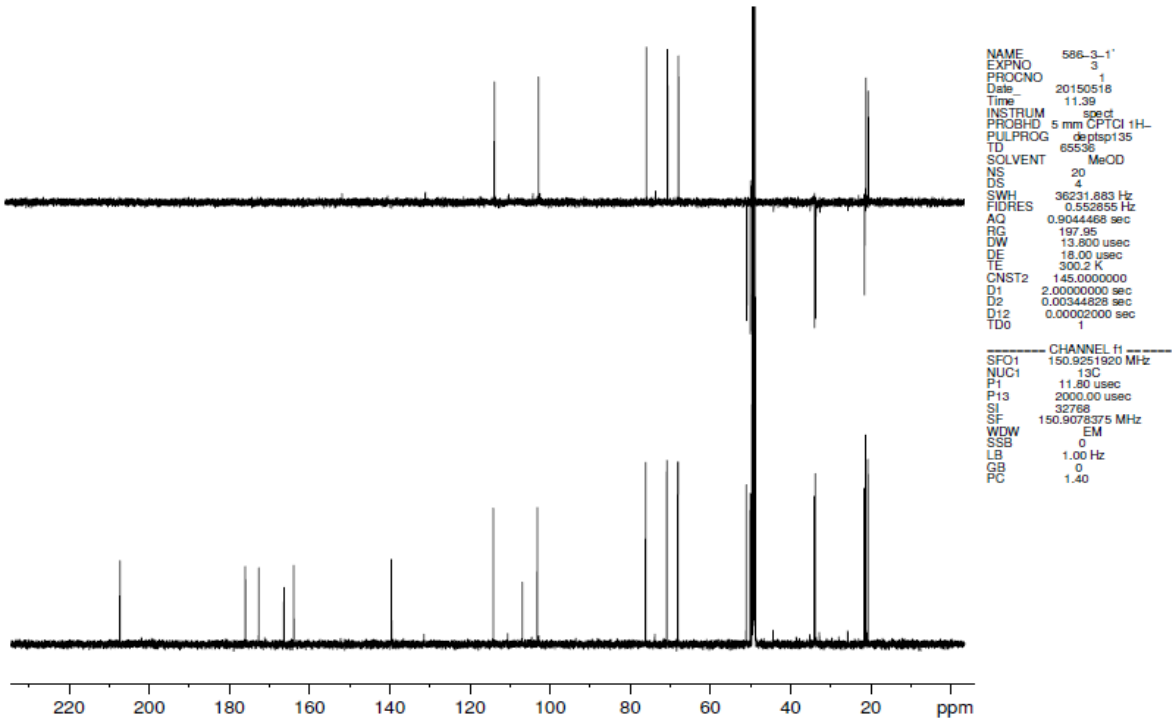


Figure S39 ^{13}C-NMR and DEPT 135 spectra of **4** (150MHz, in CD_3OD)

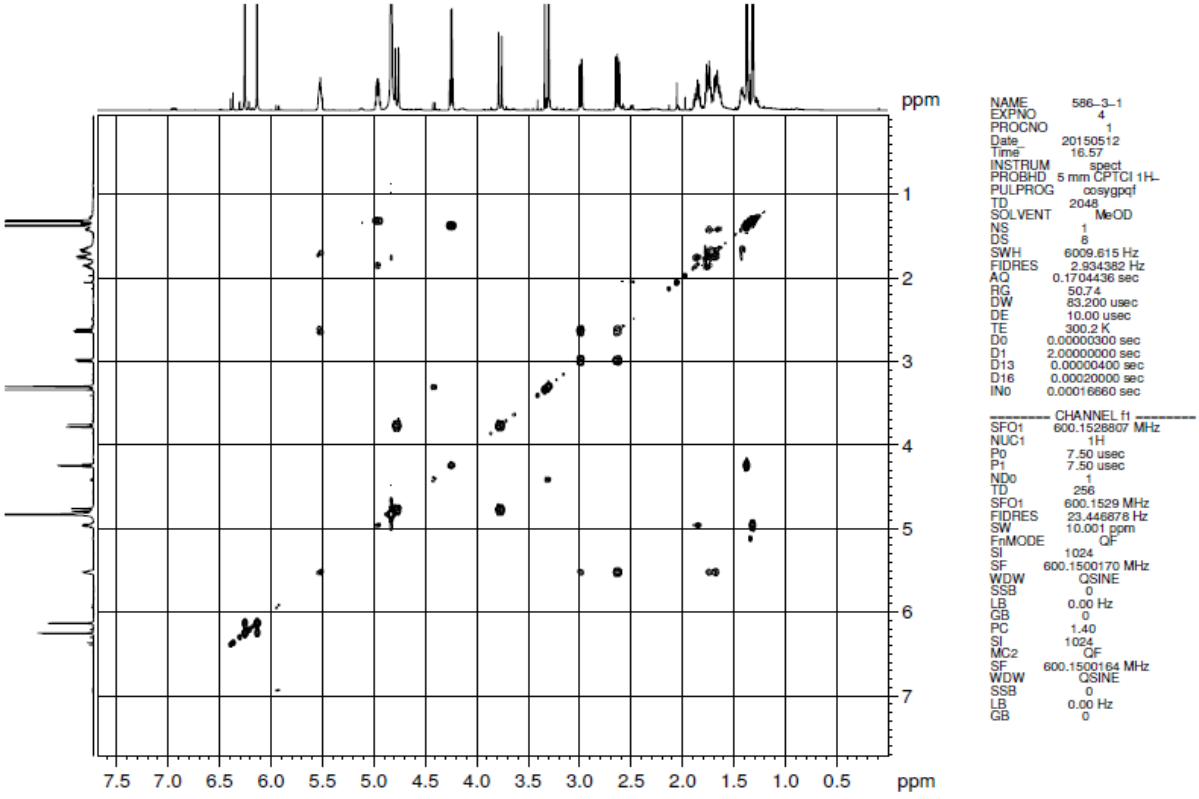


Figure S40 ^{1}H-^{1}H COSY spectrum of **4** (600MHz, in CD_3OD)

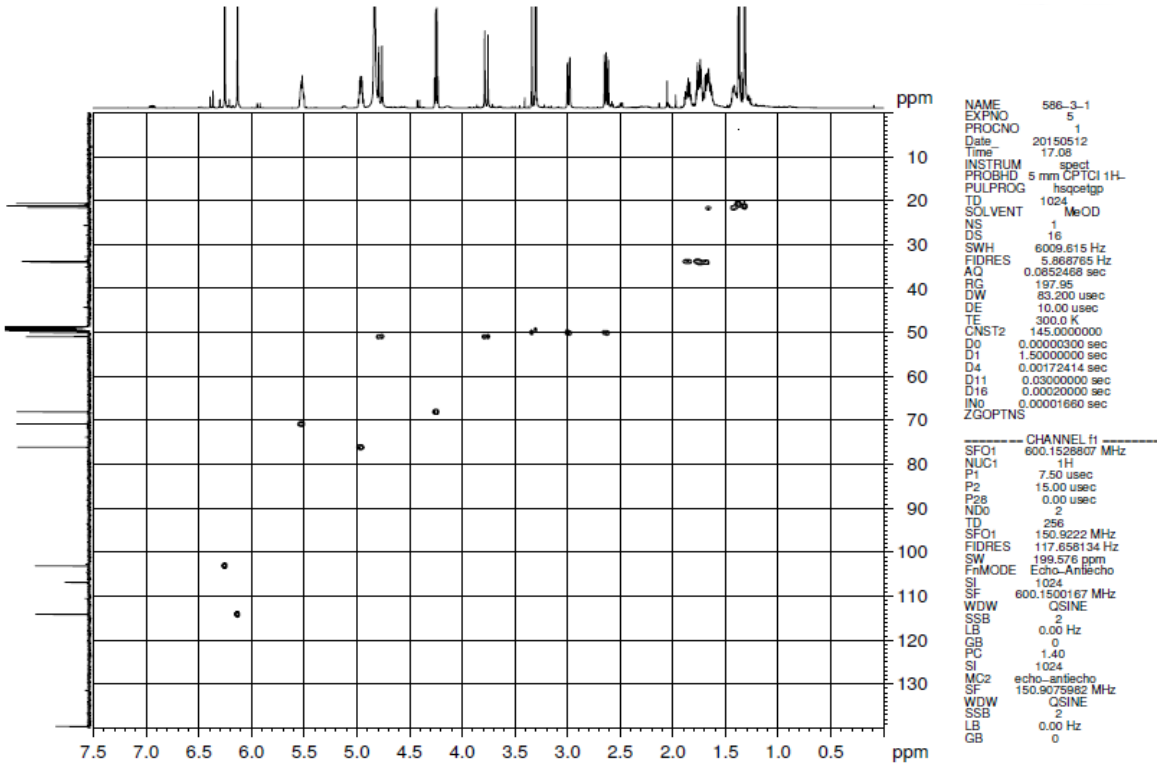


Figure S41 HSQC spectrum of **4** (600MHz, in CD_3OD)

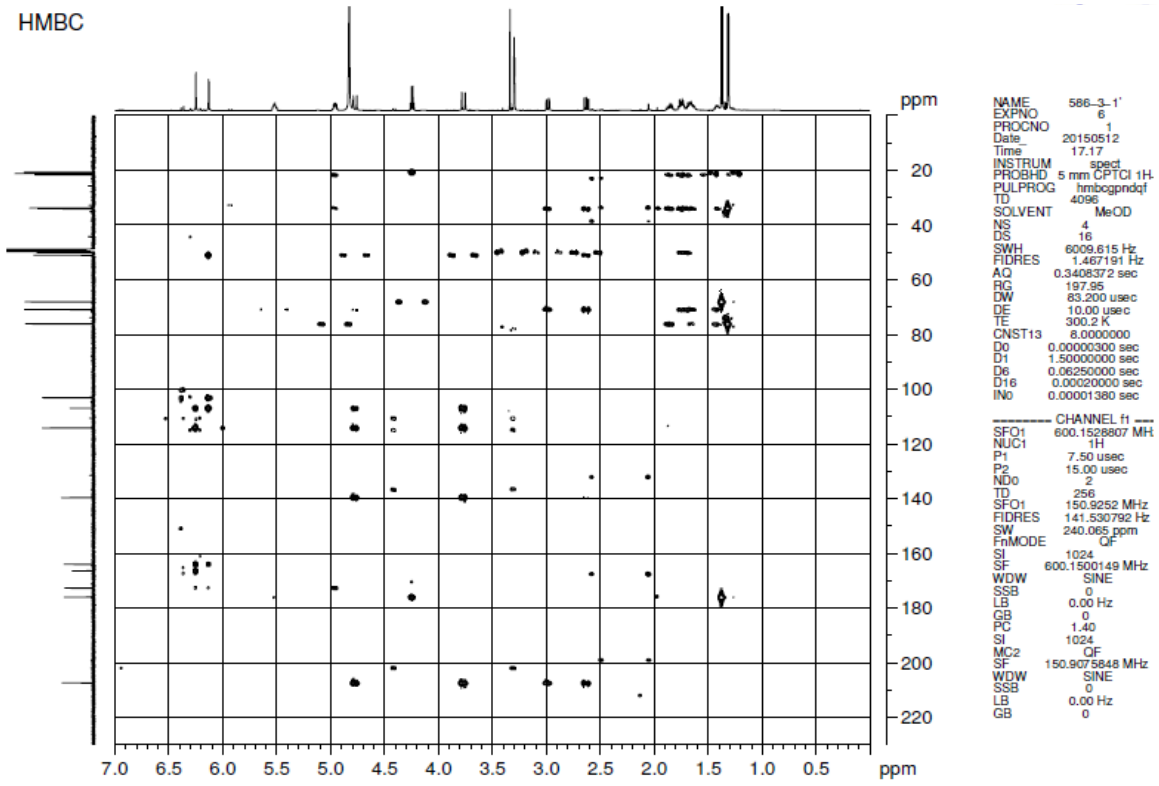


Figure S42 HMBC spectrum of **4** (600MHz, in CD_3OD)

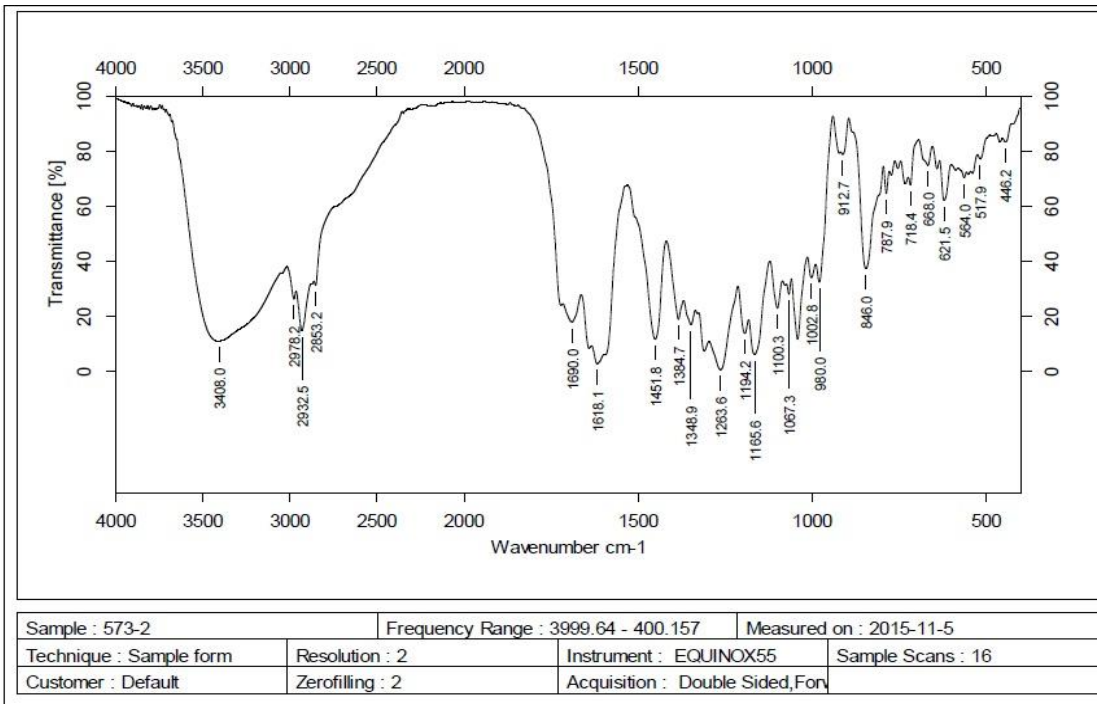


Figure S43 IR (KBr disc) spectrum of penicimenolide E (**5**)

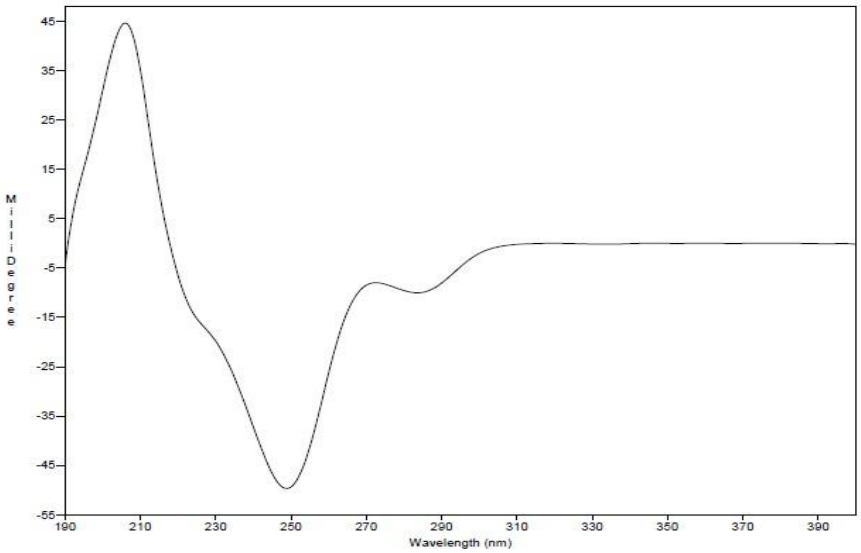


Figure S44 CD spectrum of penicimenolide E (**5**) in MeOH

Spectrum Peak Pick Report

FIELD FIELD TEXT

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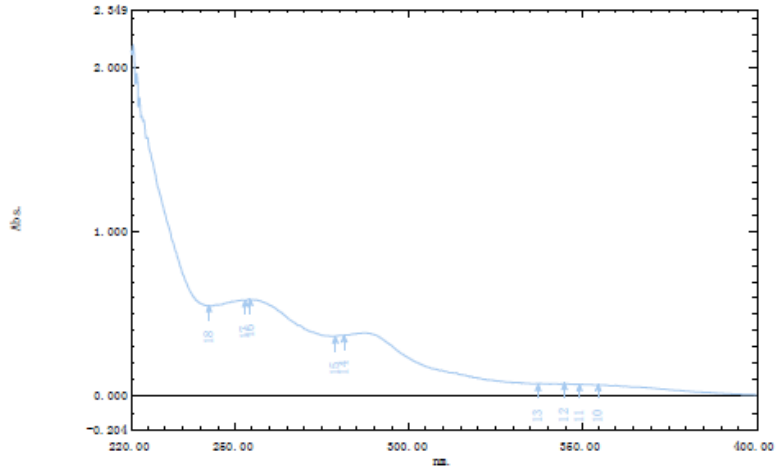

测定属性	
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扫描速度:	高速
采样间隔:	0.2
自动采样间隔:	启用
扫描模式:	单一的
试样准备属性	
重量:	0.5
体积 :	10
稀释:	
光程长:	407
附加信息:	
仪器属性	
仪器类型:	UV-1700
测定方式:	吸收值
狭缝宽:	1.0 nm
光源改变波长:	340.8 nm
S/N 转换:	标准

No.	P/V	Wavelength	Abs.	描述
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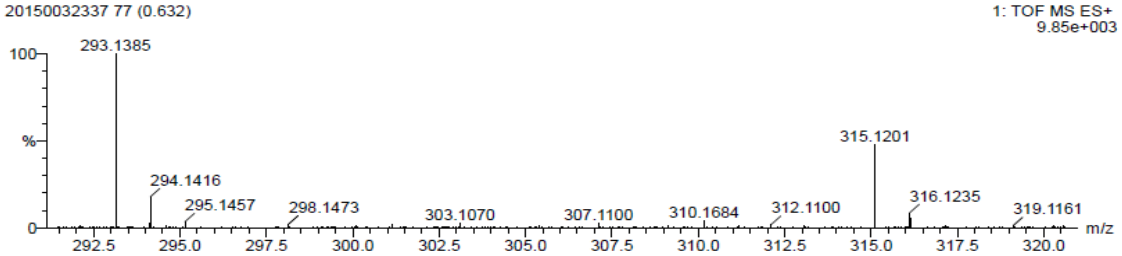
2	P	350.00	.073	
3	P	345.60	.076	
4	P	339.00	.077	
5	P	287.60	.386	
6	P	280.40	.369	
7	P	255.20	.587	
8	P	253.60	.588	
9	P	252.00	.585	
10	V	354.40	.067	
11	V	349.20	.072	
12	V	344.80	.074	
13	V	337.40	.074	
14	V	281.20	.368	
15	V	278.80	.364	
16	V	254.40	.585	
17	V	252.80	.582	
18	V	242.40	.548	

Figure S45 UV spectrum of penicimenolide E (**5**) in MeOH

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
436 formula(e) evaluated with 6 results within limits (up to 10 best isotopic matches for each mass)
Elements Used:
C: 0-500 H: 0-1000 N: 0-10 O: 0-200
573-2
20150032337 77 (0.632)



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
293.1385	293.1389	-0.4	-1.4	6.5	187.0	0.078	92.48	C16 H21 O5
	293.1402	-1.7	-5.8	11.5	189.6	2.702	6.70	C17 H17 N4 O
	293.1362	2.3	7.8	7.5	192.1	5.198	0.55	C12 H17 N6 O3
	293.1349	3.6	12.3	2.5	192.9	5.993	0.25	C11 H21 N2 O7
	293.1421	-3.6	-12.3	-1.5	196.7	9.741	0.01	C5 H21 N6 O8
	293.1434	-4.9	-16.7	3.5	196.9	10.005	0.00	C6 H17 N10 O4

Figure S46 HR-ESI-MS spectrum of penicimenolide E (**5**)

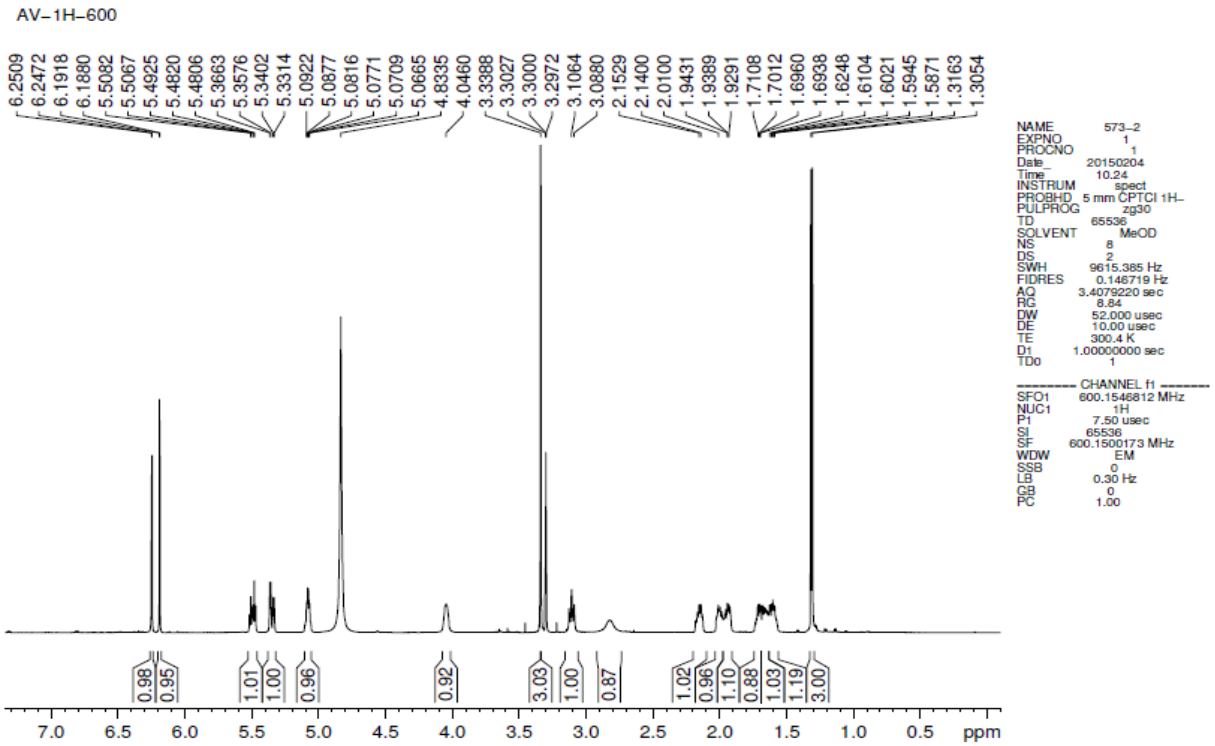


Figure S47 ^{1}H-NMR spectrum of **5** (600 MHz, in CD_3OD)

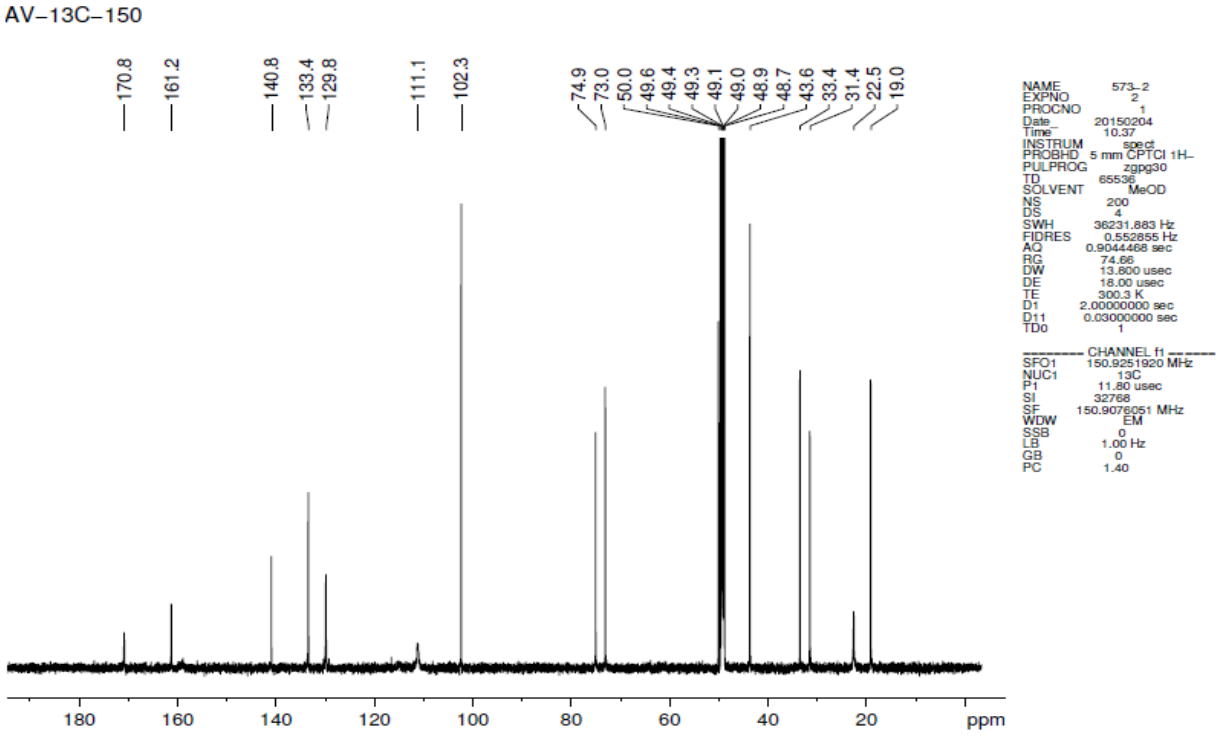


Figure S48 ^{13}C-NMR spectrum of **5** (150MHz, in CD_3OD)

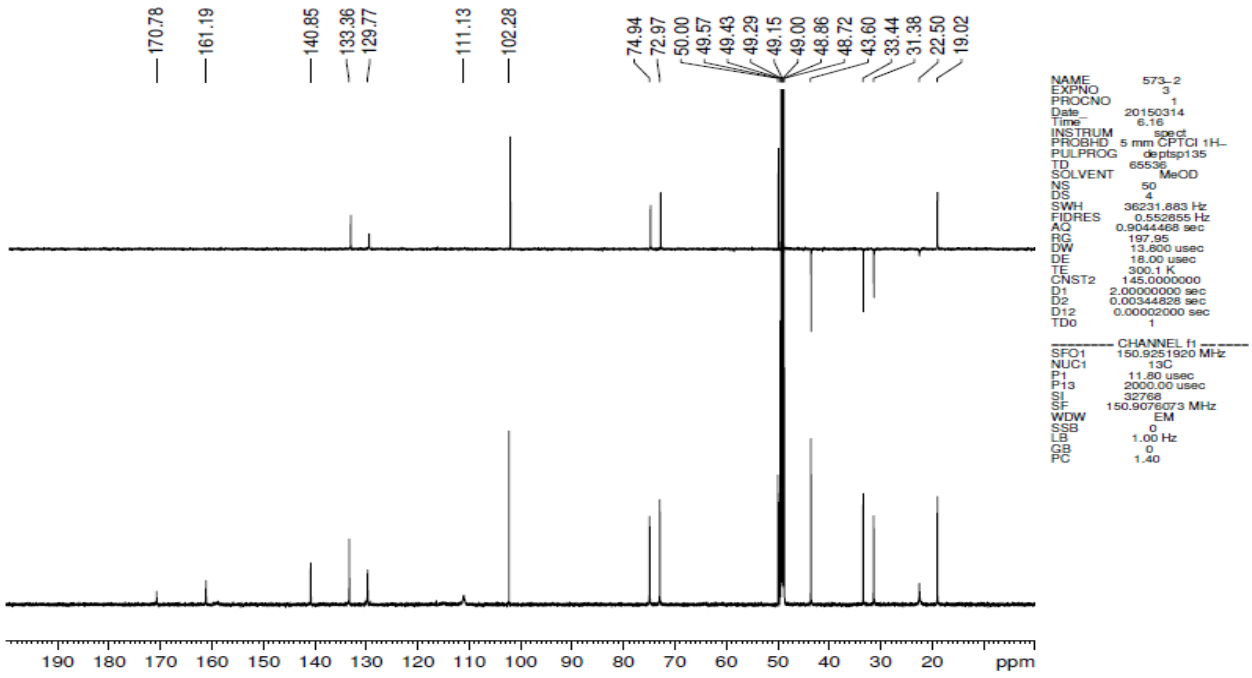


Figure S49 ^{13}C-NMR and DEPT 135 spectra of **5** (150MHz, in CD_3OD)

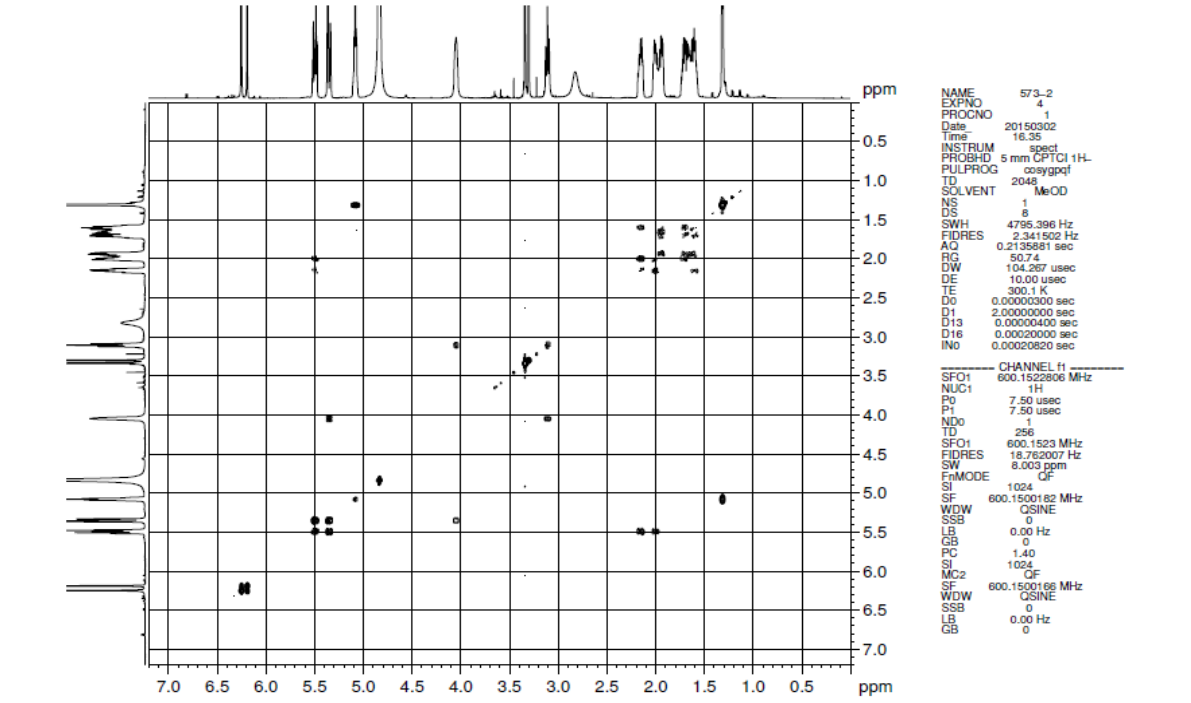


Figure S50 ^{1}H-^{1}H COSY spectrum of **5** (600MHz, in CD_3OD)

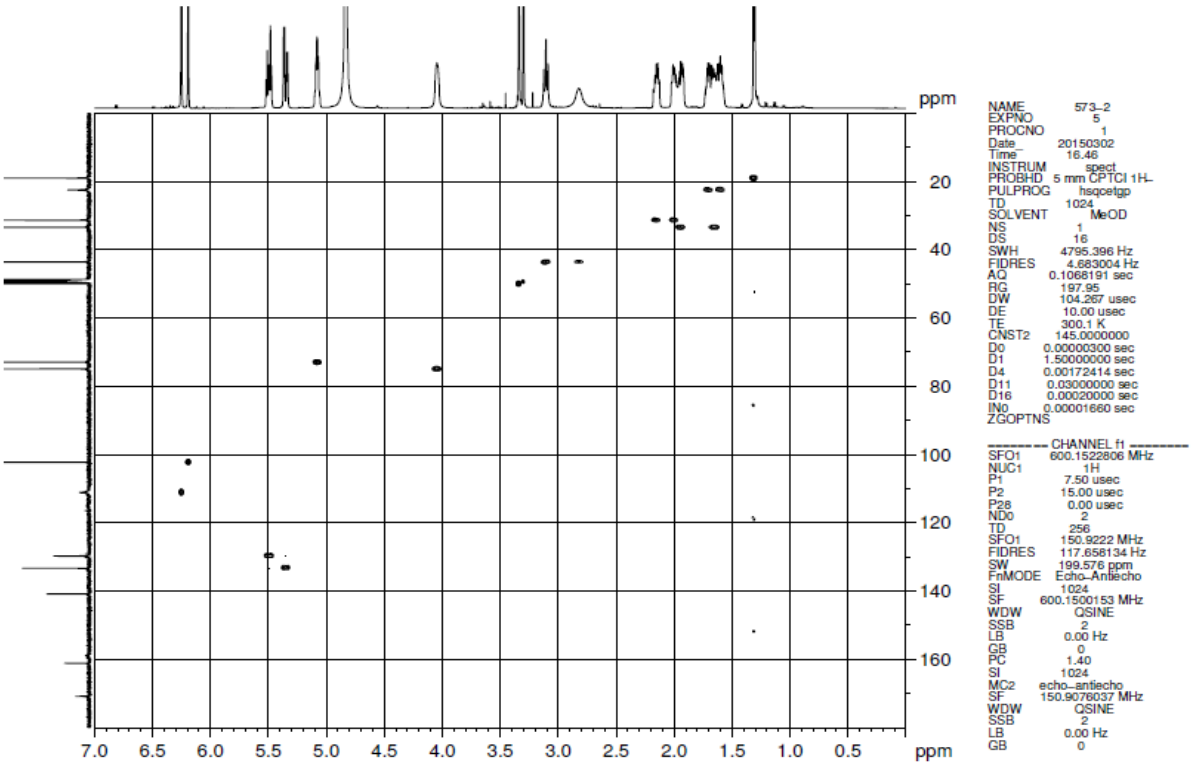


Figure S51 HSQC spectrum of **5** (600MHz, in CD_3OD)

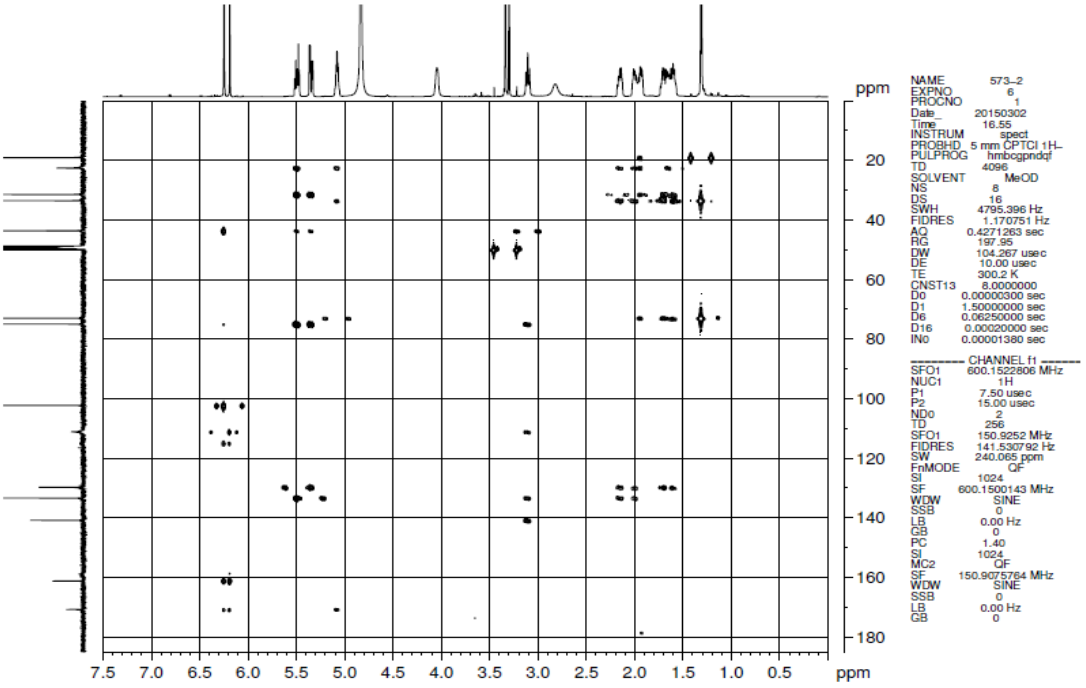


Figure S52 HMBC spectrum of **5** (600MHz, in CD_3OD)

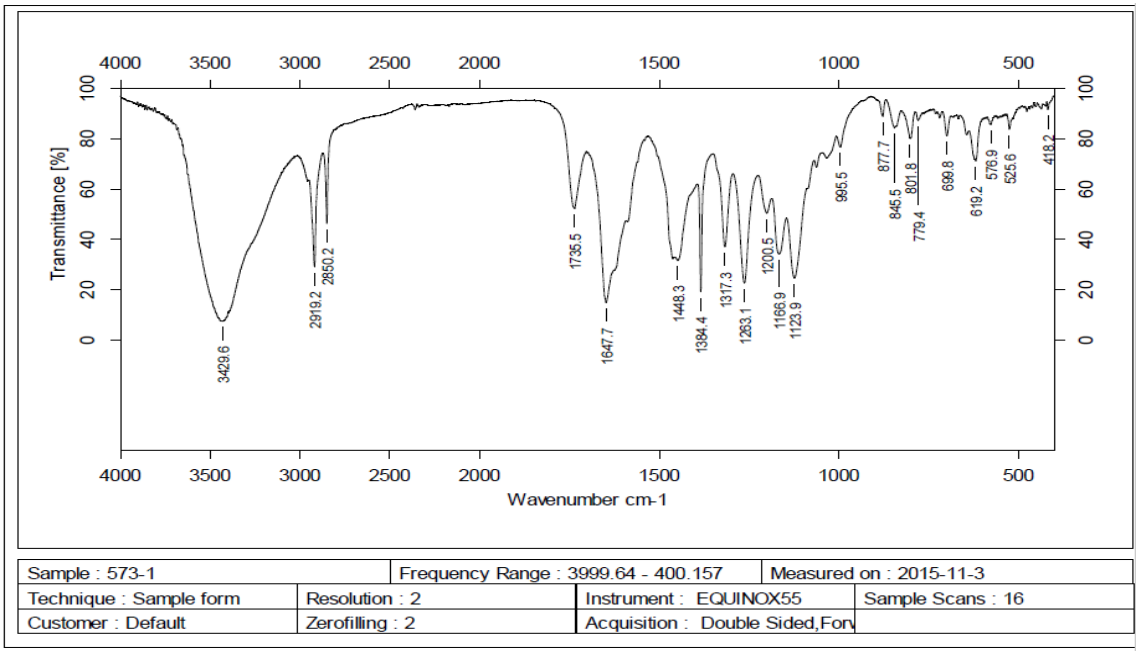


Figure S53 IR (KBr disc) spectrum of penicimenolide F (**6**)

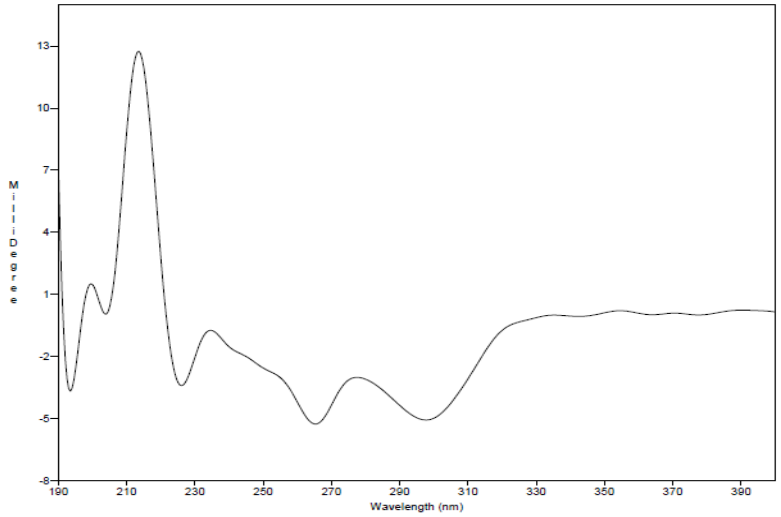
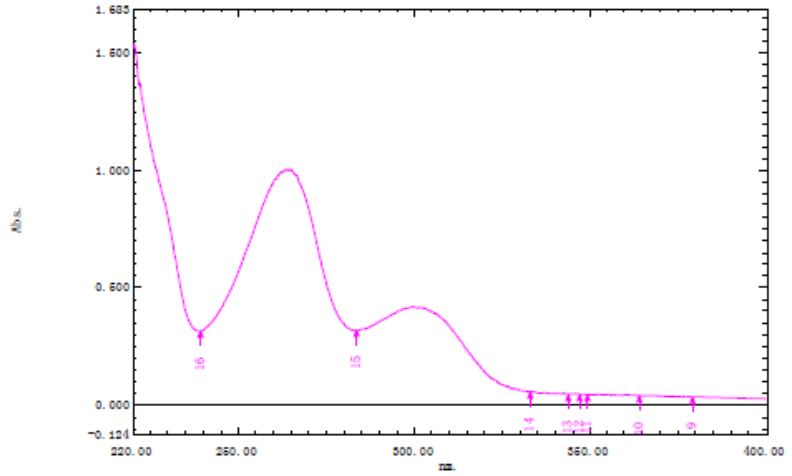


Figure S54 CD spectrum of penicimenolide F (**6**) in MeOH

Spectrum Peak Pick Report

FIELD FIELD TEXT

Data Set: 没有



测定属性	
波长范围 (nm.):	220.00到400.00
扫描速度:	高速
采样间隔:	0.2
自动采样间隔:	启用
扫描模式:	单一的
试样准备属性	
重量:	0.5
体积 :	10
稀释:	
光程长:	407
附加信息:	
仪器属性	
仪器类型:	UV-1700
测定方式:	吸收值
狭缝宽:	1.0 nm
光源改变波长:	340.0 nm
S/R 转换:	标准

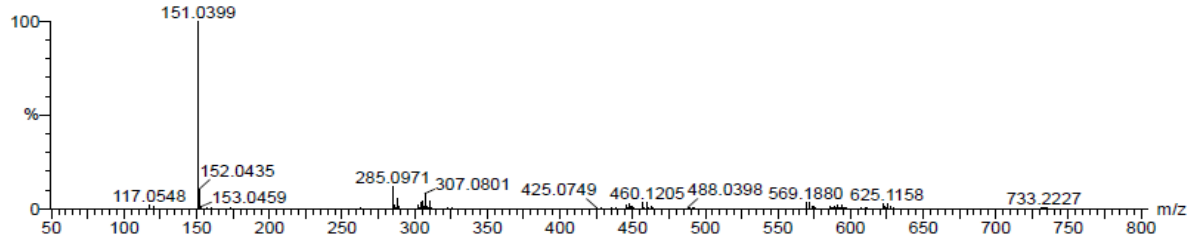
No.	P/V	Wavelength	Abs.	描述
1	↑	380.60	.035	
2	↑	364.60	.040	
3	↑	355.20	.044	
4	↑	348.00	.046	
5	↑	345.60	.048	
6	↑	333.80	.057	
7	↑	299.40	.418	
8	↑	263.80	1.003	
9	↓	379.00	.034	
10	↓	364.00	.040	
11	↓	349.00	.041	
12	↓	347.00	.044	
13	↓	343.80	.045	
14	↓	333.00	.052	
15	↓	283.60	.316	
16	↓	239.20	.314	

Figure S55 UV spectrum of penicimenolide F (**6**) in MeOH

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 30.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
186 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 0-50 H: 0-100 N: 0-3 O: 0-30
573-1
20150617-05 65 (0.531) Cm (63:65) 1: TOF MS ES+ 6.61e+005



Minimum:				-1.5				
Maximum:		5.0	5.0	30.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
285.0971	285.0974	-0.3	-1.1	5.5	288.6	n/a	n/a	C13 H17 O7

Figure S56 HR-ESI-MS spectrum of penicimenolide F (**6**)

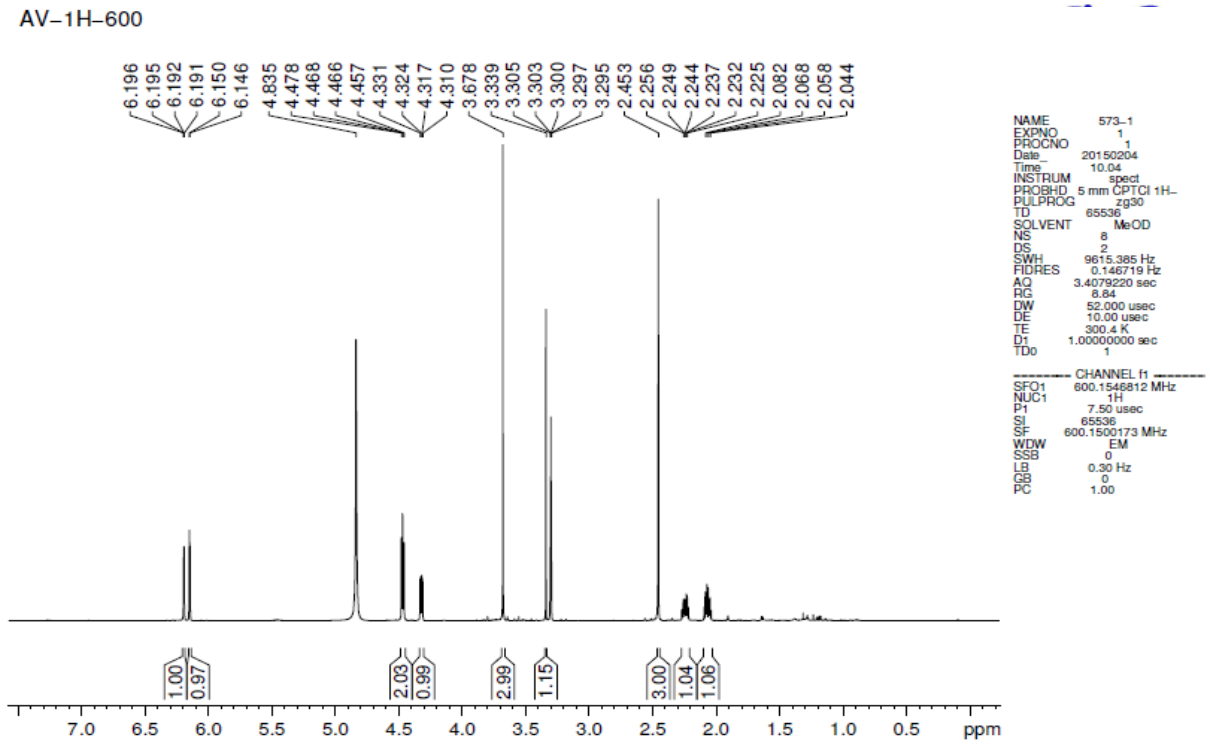


Figure S57 ^{1}H-NMR spectrum of **6** (600 MHz, in CD_3OD)

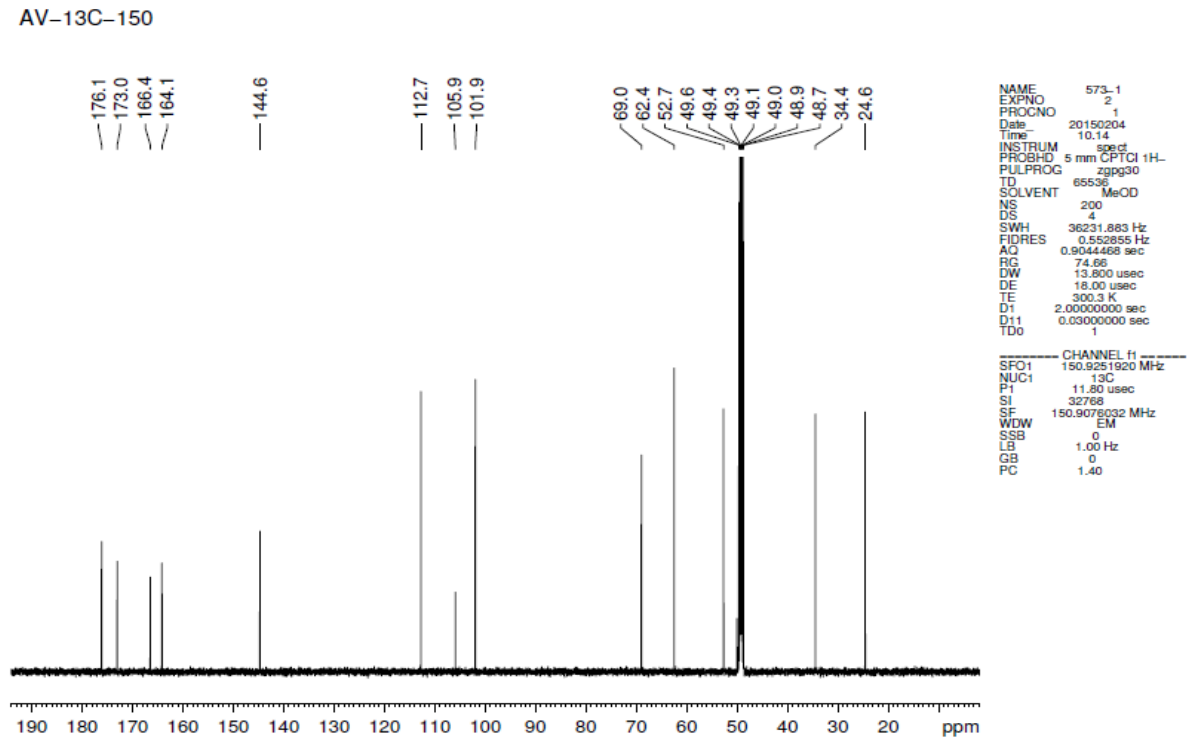


Figure S58 ^{13}C-NMR spectrum of **6** (150MHz, in CD_3OD)

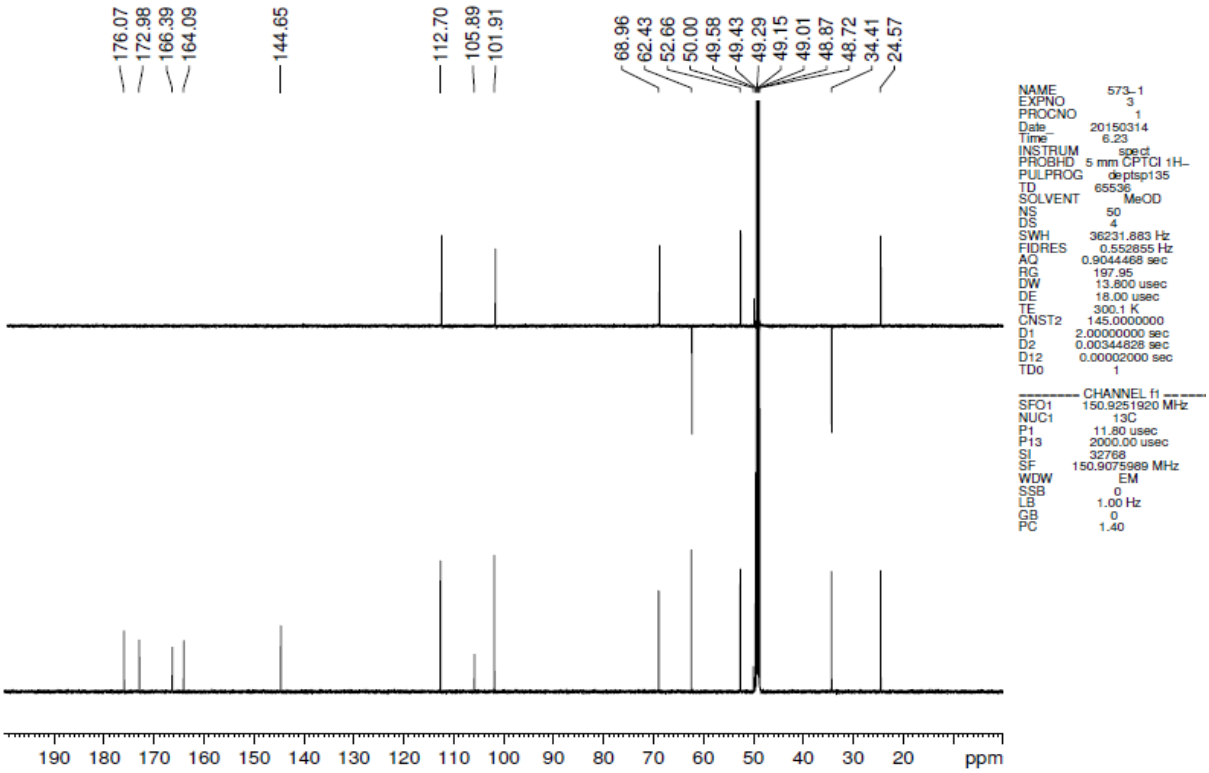


Figure S59 ^{13}C-NMR and DEPT 135 spectra of **6** (150MHz, in CD_3OD)

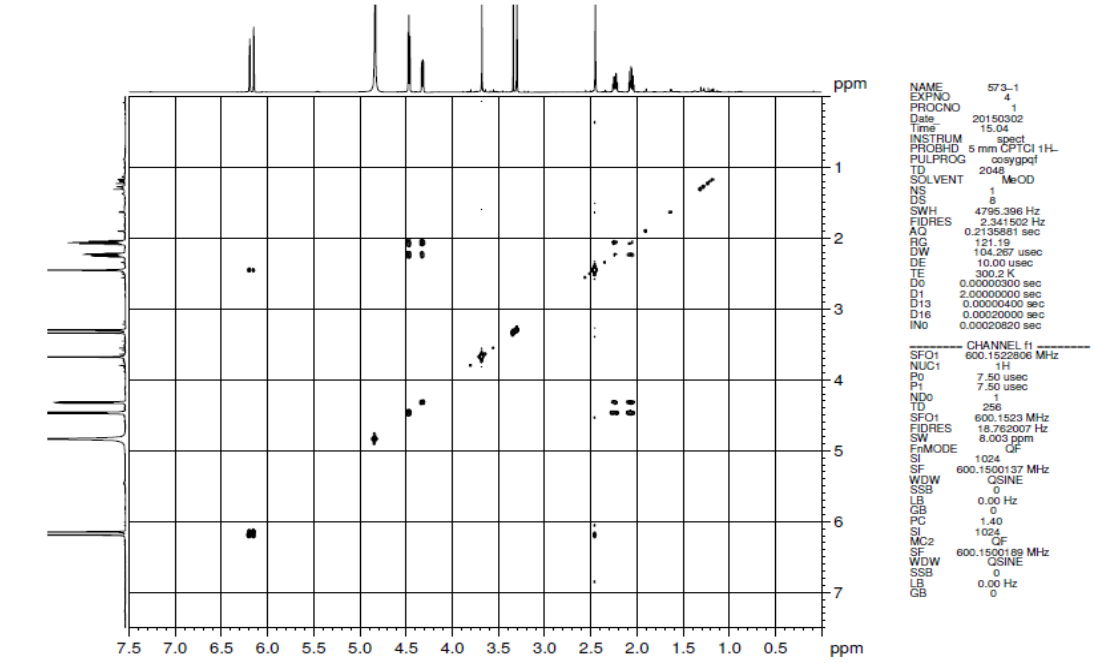


Figure S60 ^{1}H-^{1}H COSY spectrum of **6** (600MHz, in CD_3OD)

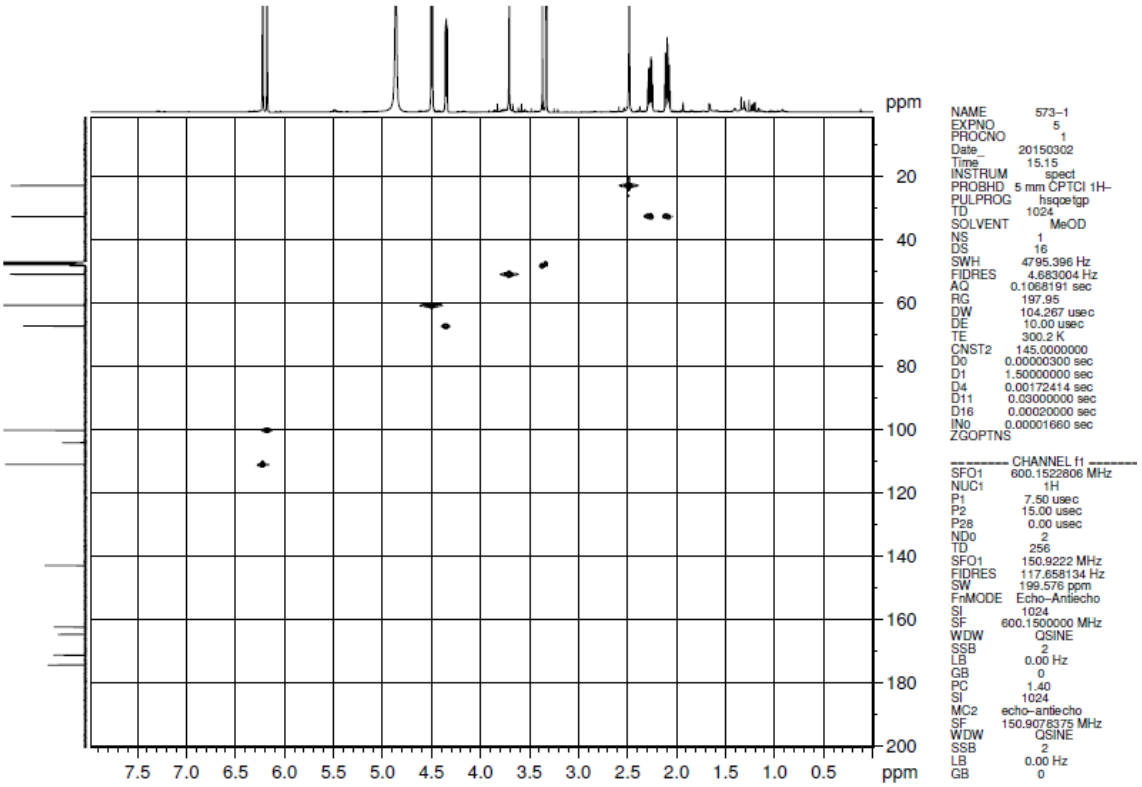


Figure S61 HSQC spectrum of **6** (600MHz, in CD_3OD)

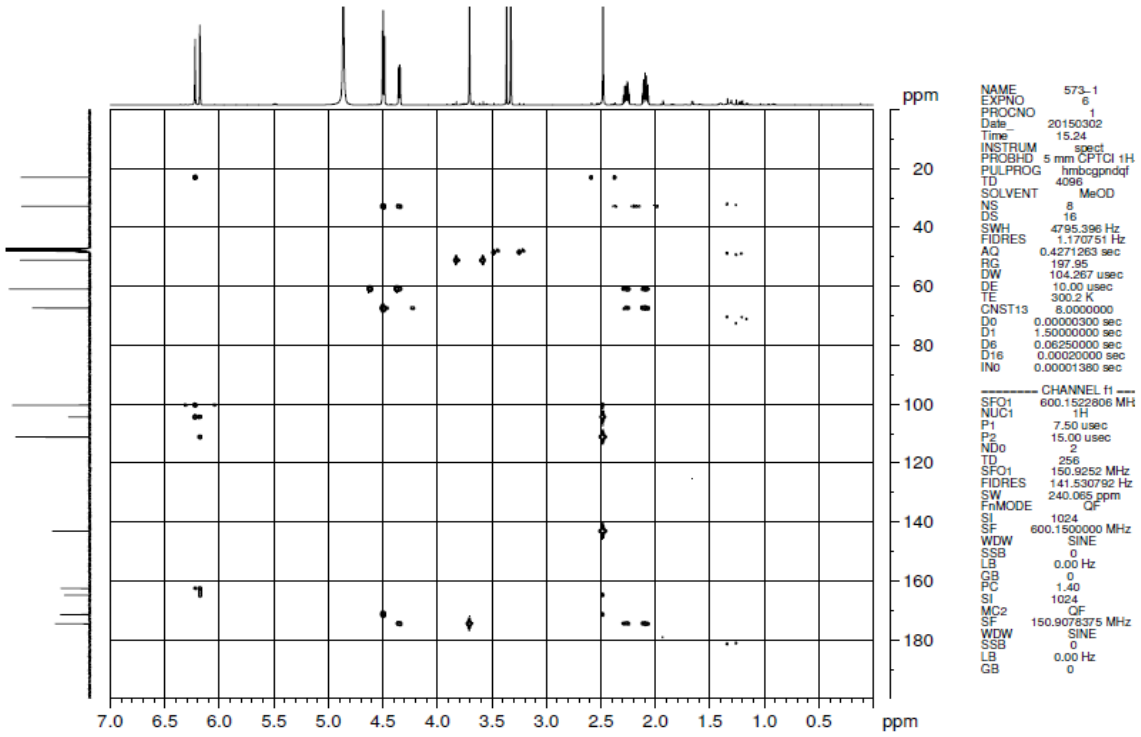


Figure S62 HMBC spectrum of **6** (600MHz, in CD_3OD)

4. NMR data of compound 8

Table S1 NMR data of compound **8** in CD_3OD

position	δ_C	δ_H (J in Hz)
1	172.6	
3	74.7	5.10, m
4	32.9	1.62, m
		1.64, m
5	22.3	1.46, m
		1.47, m
6	28.3	1.47, m
		1.49, m
7	22.3	2.00, m
		1.79, m
8	42.7	2.67, m
		2.33, m
9	211.8	
10	51.8	4.68, d (18.7)
		3.78, d (18.5)
11	140.4	
12	113.9	6.09, d (2.3)
13	163.9	
14	103.0	6.24, d (2.3)
15	166.7	
16	106.8	
17	19.6	1.28, d (6.2)

5. ^{1}H-NMR chemical shift differences for the MTPA esters of 3, 5 and 6

Table S2 ^{1}H-NMR chemical shift differences between MTPA esters of **3a** and **3b** (Pyridine-d_5)

position	**3**	**3a**	**3b**	**Δδ**
	δ_H	δ_H	δ_H	
7	5.928	5.761	5.778	-0.017
3´	1.627	1.581	1.511	0.069

Table S3 ^{1}H-NMR chemical shift differences between MTPA esters of **5a** and **5b** (Pyridine-d_5)

position	**5**	**5a**	**5b**	**Δδ**
	δ_H	δ_H	δ_H	
4	2.052	1.705	1.629	0.076
	-	-	-	-
5	1.768	1.507	1.466	0.041
	-	-	-	-
6	2.247	2.025	1.937	0.05
	2.120	1.860	1.747	0.05
7	4.754	-	-	-
8	5.598	5.537	5.406	0.131
9	3.876	-	-	-
10	3.461	3.364	3.409	-0.306
17	1.408	1.225	1.073	0.152

Table S4 ^{1}H-NMR chemical shift differences between MTPA esters of **6a** and **6b** (Pyridine-d_5)

position	**6**	**6a**	**6b**	**Δδ**
	δ_H	δ_H	δ_H	
1´	4.792	4.555	4.440	0.115
	4.756	4.518	4.394	0.120
2´	2.496	2.538	2.476	0.06
	2.358	2.456	2.423	0.03
5´	3.69	3.709	3.775	-0.07

6. Single crystal X-ray diffraction analysis of compound 10

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(compiled 2015.09.30 svn.r3233 for OlexSys, GUI svn.r5103)

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Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H.

(2009), J. Appl. Cryst. 42, 339-341.

Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

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 'H' 'H' 0.0000 0.0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O' 'O' 0.0492 0.0322 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
_space_group_crystal_system       'monoclinic'
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_space_group_name_Hall            'P 2yb'
loop_
  _space_group_symop_id
  _space_group_symop_operation_xyz
 1 'x, y, z'
 2 '-x, y+1/2, -z'
_cell_length_a                    5.05804(7)
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_exptl_absorpt_correction_type multi-scan

_exptl_absorpt_process_details

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CrysAlisPro, Agilent Technologies,

Version 1.171.36.32 (release 02-08-2013 CrysAlis171 .NET)

(compiled Aug 2 2013,16:46:58)

Empirical absorption correction using spherical harmonics,

implemented in SCALE3 ABSPACK scaling algorithm.

;

_exptl_crystal_colour ''

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_exptl_crystal_density_method 'not measured'

_exptl_crystal_description ''

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omega____ theta____ kappa____ phi_____ frames
    -      -39.3998  -57.0000   30.0000 80
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omega____ theta____ kappa____ phi_____ frames
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omega____ theta____ kappa____ phi_____ frames
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omega____ theta____ kappa____ phi_____ frames
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5 omega 17.00 43.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 39.3998 57.0000 -90.0000 26

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6 omega -27.00 56.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 39.3998 -38.0000 -180.0000 83

#__ type_ start__ end____ width___ exp.time_

7 omega 81.00 178.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 30.0000 60.0000 97

#__ type_ start__ end____ width___ exp.time_

8 omega 120.00 151.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 45.0000 120.0000 31

#__ type_ start__ end____ width___ exp.time_

9 omega 82.00 177.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 30.0000 -30.0000 95

#__ type_ start__ end____ width___ exp.time_

10 omega 83.00 108.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 -94.0000 -90.0000 25

#__ type_ start__ end____ width___ exp.time_

11 omega 30.00 102.00 1.0000 1.0000

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- 111.3657 -94.0000 120.0000 72

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12 omega 53.00 95.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 -94.0000 0.0000 42

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13 omega 35.00 62.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 -94.0000 -90.0000 27

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14 omega 30.00 111.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 -98.0000 -142.0000 81

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15 omega 81.00 175.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 61.0000 -150.0000 94

#__ type_ start__ end____ width___ exp.time_

16 omega 33.00 89.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 -30.0000 150.0000 56

#__ type_ start__ end____ width___ exp.time_

17 omega 34.00 81.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 -30.0000 -90.0000 47

#__ type_ start__ end____ width___ exp.time_

18 omega 88.00 132.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 45.0000 90.0000 44

#__ type_ start__ end____ width___ exp.time_

19 omega 78.00 109.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 111.3657 -94.0000 30.0000 31

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omega____ theta____ kappa____ phi______ frames
   -      111.3657  111.0000   60.0000 63
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_diffrn_standards_interval_time   ?
_diffrn_standards_number          ?
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_reflns_odcompleteness_theta      68.13
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CrysAlisPro, Agilent Technologies,
Version 1.171.36.32 (release 02-08-2013 CrysAlis171 .NET)
(compiled Aug  2 2013,16:46:58)
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Version 1.171.36.32 (release 02-08-2013 CrysAlis171 .NET)
(compiled Aug  2 2013,16:46:58)
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(compiled Aug  2 2013,16:46:58)
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_computing_structure_solution     'XS (Sheldrick, 2008)'
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_refine_ls_extinction_method      none
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_refine_ls_hydrogen_treatment constr

_refine_ls_matrix_type full

_refine_ls_number_parameters 212

_refine_ls_number_reflns 3141

_refine_ls_number_restraints 1

_refine_ls_R_factor_all 0.0336

_refine_ls_R_factor_gt 0.0333

_refine_ls_restrained_S_all 0.971

_refine_ls_shift/su_max 0.000

_refine_ls_shift/su_mean 0.000

_refine_ls_structure_factor_coef Fsqd

_refine_ls_weighting_details

'calc w=1/[\s^2^(Fo^2^)+(0.1000P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'

_refine_ls_weighting_scheme calc

_refine_ls_wR_factor_gt 0.1000

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_refine_special_details

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Refinement of F^2^ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2^, conventional R-factors R are based on F, with F set to zero for negative F^2^. The threshold expression of F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2^ are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

;

_olex2_refinement_description

;

1. Fixed Uiso

At 1.2 times of:

All C(H) groups, All C(H,H) groups

At 1.5 times of:

All C(H,H,H) groups, All O(H) groups

2.a Ternary CH refined with riding coordinates:

C7(H7), C3(H3)

2.b Secondary CH2 refined with riding coordinates:

C8(H8A,H8B), C10(H10A,H10B), C5(H5A,H5B), C4(H4A,H4B), C6(H6A,H6B)

2.c Aromatic/amide H refined with riding coordinates:

C14(H14), C12(H12)

2.d Idealised Me refined as rotating group:

C18(H18A,H18B,H18C), C17(H17A,H17B,H17C)

2.e Idealised tetrahedral OH refined as rotating group:

O1(H1), O6(H6)

;

_atom_sites_solution_hydrogens geom

_atom_sites_solution_primary direct

_atom_sites_solution_secondary difmap

loop_

_atom_site_label

_atom_site_type_symbol

_atom_site_fract_x

_atom_site_fract_y

_atom_site_fract_z

_atom_site_U_iso_or_equiv

_atom_site_adp_type

_atom_site_occupancy

_atom_site_calc_flag

_atom_site_disorder_assembly

_atom_site_disorder_group

_atom_site_refinement_flags_posn

O5 O 0.09976(16) 0.24159(8) 0.14651(7) 0.0257(2) Uani 1 d . . .

O1 O -0.03984(19) 0.60821(9) 0.33759(8) 0.0343(2) Uani 1 d . . .

H1 H -0.0778 0.5522 0.3704 0.051 Uiso 1 calc . . GR

O6 O 0.4603(2) 0.70790(8) 0.09151(7) 0.0342(2) Uani 1 d . . .

H6 H 0.4091 0.7733 0.1045 0.051 Uiso 1 calc . . GR

O3 O 0.29093(18) 0.28453(8) 0.36167(6) 0.0267(2) Uani 1 d . . .

O4 O 0.27681(18) -0.07692(7) 0.10716(6) 0.0260(2) Uani 1 d . . .

O2 O 0.0163(2) 0.41098(10) 0.41253(8) 0.0377(3) Uani 1 d . . .

C11 C 0.4129(2) 0.43710(10) 0.21309(8) 0.0200(2) Uani 1 d . . .

C16 C 0.2390(2) 0.46321(10) 0.28110(8) 0.0209(2) Uani 1 d . . .

C1 C 0.1699(2) 0.38533(11) 0.35630(9) 0.0239(2) Uani 1 d . . .

C9 C 0.3413(2) 0.22805(10) 0.16438(7) 0.0178(2) Uani 1 d . . .

C14 C 0.1935(3) 0.65669(10) 0.21330(10) 0.0277(3) Uani 1 d . . .

H14 H 0.1136 0.7300 0.2111 0.033 Uiso 1 calc . . R

C8 C 0.4613(2) 0.11390(9) 0.14335(8) 0.0210(2) Uani 1 d . . .

H8A H 0.6518 0.1115 0.1762 0.025 Uiso 1 calc . . R

H8B H 0.4562 0.1066 0.0715 0.025 Uiso 1 calc . . R

C12 C 0.4767(2) 0.52030(10) 0.15095(9) 0.0221(2) Uani 1 d . . .

H12 H 0.5927 0.5022 0.1060 0.026 Uiso 1 calc . . R

C7 C 0.3119(2) 0.01291(9) 0.17901(8) 0.0205(2) Uani 1 d . . .

H7 H 0.1303 0.0396 0.1883 0.025 Uiso 1 calc . . R

C10 C 0.5363(2) 0.32141(9) 0.20361(8) 0.0198(2) Uani 1 d . . .

H10A H 0.6701 0.3285 0.1595 0.024 Uiso 1 calc . . R

H10B H 0.6338 0.2982 0.2693 0.024 Uiso 1 calc . . R

C3 C 0.2421(2) 0.20521(12) 0.43870(8) 0.0266(3) Uani 1 d . . .

H3 H 0.0633 0.2220 0.4569 0.032 Uiso 1 calc . . R

C5 C 0.4987(2) 0.05022(11) 0.36038(8) 0.0250(2) Uani 1 d . . .

H5A H 0.5892 0.1182 0.3392 0.030 Uiso 1 calc . . R

H5B H 0.6190 0.0168 0.4178 0.030 Uiso 1 calc . . R

C18 C 0.0440(3) -0.05978(12) 0.03270(9) 0.0316(3) Uani 1 d . . .

H18A H 0.0435 -0.1154 -0.0203 0.047 Uiso 1 calc . . GR

H18B H 0.0474 0.0173 0.0058 0.047 Uiso 1 calc . . GR

H18C H -0.1182 -0.0695 0.0616 0.047 Uiso 1 calc . . GR

C4 C 0.2360(2) 0.08723(12) 0.39228(8) 0.0268(3) Uani 1 d . . .

H4A H 0.0906 0.0854 0.3338 0.032 Uiso 1 calc . . R

H4B H 0.1905 0.0309 0.4402 0.032 Uiso 1 calc . . R

C15 C 0.1290(2) 0.57493(10) 0.27746(9) 0.0247(3) Uani 1 d . . .

C6 C 0.4560(2) -0.03674(11) 0.27614(9) 0.0265(3) Uani 1 d . . .

H6A H 0.6328 -0.0666 0.2668 0.032 Uiso 1 calc . . R

H6B H 0.3499 -0.1014 0.2948 0.032 Uiso 1 calc . . R

C17 C 0.4610(3) 0.22269(14) 0.52803(9) 0.0348(3) Uani 1 d . . .

H17A H 0.6370 0.2104 0.5093 0.052 Uiso 1 calc . . GR

H17B H 0.4364 0.1685 0.5797 0.052 Uiso 1 calc . . GR

H17C H 0.4512 0.3005 0.5527 0.052 Uiso 1 calc . . GR

C13 C 0.3749(3) 0.63107(10) 0.15233(9) 0.0254(3) Uani 1 d . . .

loop_

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_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

O5 0.0199(4) 0.0212(4) 0.0345(4) 0.0013(3) 0.0009(3) 0.0034(3)

O1 0.0311(5) 0.0341(5) 0.0406(5) -0.0127(4) 0.0147(4) 0.0052(4)

O6 0.0520(6) 0.0168(4) 0.0366(5) 0.0020(4) 0.0155(4) 0.0056(4)

O3 0.0305(5) 0.0301(5) 0.0223(4) 0.0044(4) 0.0119(3) 0.0046(4)

O4 0.0328(4) 0.0171(4) 0.0268(4) -0.0031(3) 0.0015(3) 0.0022(3)

O2 0.0416(5) 0.0390(5) 0.0387(5) -0.0023(4) 0.0240(4) 0.0051(4)

C11 0.0182(5) 0.0182(5) 0.0233(5) -0.0034(4) 0.0027(4) -0.0004(4)

C16 0.0188(5) 0.0212(5) 0.0226(5) -0.0058(4) 0.0034(4) -0.0004(4)

C1 0.0208(5) 0.0287(6) 0.0229(5) -0.0057(4) 0.0055(4) -0.0016(5)

C9 0.0213(5) 0.0172(5) 0.0157(5) 0.0027(4) 0.0055(3) 0.0024(4)

C14 0.0287(5) 0.0202(5) 0.0327(6) -0.0088(5) 0.0011(4) 0.0054(5)

C8 0.0231(5) 0.0168(5) 0.0238(5) -0.0006(4) 0.0057(4) 0.0028(4)

C12 0.0245(5) 0.0177(5) 0.0246(5) -0.0035(4) 0.0061(4) 0.0018(4)

C7 0.0234(5) 0.0151(5) 0.0224(5) -0.0009(4) 0.0027(4) 0.0012(4)

C10 0.0190(5) 0.0175(5) 0.0242(5) -0.0001(4) 0.0073(4) 0.0031(4)

C3 0.0273(5) 0.0360(7) 0.0179(5) 0.0011(5) 0.0079(4) -0.0041(5)

C5 0.0256(5) 0.0264(6) 0.0215(5) 0.0020(5) -0.0004(4) -0.0007(4)

C18 0.0323(6) 0.0323(6) 0.0281(6) -0.0077(5) -0.0009(5) -0.0024(5)

C4 0.0263(5) 0.0323(6) 0.0221(5) 0.0010(5) 0.0047(4) -0.0073(5)

C15 0.0215(5) 0.0251(6) 0.0269(5) -0.0100(5) 0.0026(4) 0.0018(5)

C6 0.0305(6) 0.0222(6) 0.0249(6) 0.0047(4) -0.0003(4) 0.0021(5)

C17 0.0361(7) 0.0482(9) 0.0198(5) -0.0012(5) 0.0039(4) -0.0096(6)

C13 0.0317(6) 0.0185(5) 0.0246(5) -0.0023(4) 0.0013(4) 0.0015(4)

_geom_special_details

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All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken

into account individually in the estimation of esds in distances, angles

and torsion angles; correlations between esds in cell parameters are only

used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.

;

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O5 C9 1.2133(14) . ?

O1 C15 1.3485(15) . ?

O6 C13 1.3535(16) . ?

O3 C1 1.3290(17) . ?

O3 C3 1.4658(15) . ?

O4 C7 1.4362(13) . ?

O4 C18 1.4326(14) . ?

O2 C1 1.2282(15) . ?

C11 C16 1.4273(16) . ?

C11 C12 1.3749(17) . ?

C11 C10 1.5108(15) . ?

C16 C1 1.4699(17) . ?

C16 C15 1.4230(16) . ?

C9 C8 1.5207(14) . ?

C9 C10 1.5096(15) . ?

C14 C15 1.3830(19) . ?

C14 C13 1.3821(19) . ?

C8 C7 1.5333(16) . ?

C12 C13 1.4009(16) . ?

C7 C6 1.5223(15) . ?

C3 C4 1.5245(19) . ?

C3 C17 1.5169(17) . ?

C5 C4 1.5342(18) . ?

C5 C6 1.5325(16) . ?

loop_

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_geom_angle_atom_site_label_3

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_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

C1 O3 C3 118.15(9) . . ?

C18 O4 C7 112.08(9) . . ?

C16 C11 C10 124.20(10) . . ?

C12 C11 C16 119.74(10) . . ?

C12 C11 C10 116.07(10) . . ?

C11 C16 C1 125.44(11) . . ?

C15 C16 C11 117.23(11) . . ?

C15 C16 C1 117.30(10) . . ?

O3 C1 C16 115.42(9) . . ?

O2 C1 O3 121.34(12) . . ?

O2 C1 C16 123.22(12) . . ?

O5 C9 C8 119.90(10) . . ?

O5 C9 C10 123.22(10) . . ?

C10 C9 C8 116.84(9) . . ?

C13 C14 C15 119.63(11) . . ?

C9 C8 C7 112.66(9) . . ?

C11 C12 C13 121.63(11) . . ?

O4 C7 C8 110.82(9) . . ?

O4 C7 C6 107.81(9) . . ?

C6 C7 C8 113.06(9) . . ?

C9 C10 C11 115.52(9) . . ?

O3 C3 C4 105.52(9) . . ?

O3 C3 C17 108.00(11) . . ?

C17 C3 C4 114.90(12) . . ?

C6 C5 C4 113.13(9) . . ?

C3 C4 C5 115.07(10) . . ?

O1 C15 C16 122.14(12) . . ?

O1 C15 C14 115.98(11) . . ?

C14 C15 C16 121.86(11) . . ?

C7 C6 C5 113.07(10) . . ?

O6 C13 C14 123.37(11) . . ?

O6 C13 C12 116.91(11) . . ?

C14 C13 C12 119.71(12) . . ?

_olex2_submission_special_instructions 'No special instructions were received'

7. CheckCIFPLATON report of compound 10

You have not supplied any structure factors. As a result the full set of tests cannot be run.

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 574-4-4

```
Bond precision:  C-C = 0.0017 A          Wavelength=1.54184

Cell:          a=5.05804(7)   b=11.74745(14)       c=13.77837(18)
               alpha=90       beta=100.1922(13)    gamma=90
Temperature:   150 K

                Calculated                Reported
Volume          805.779(18)               805.778(18)
Space group     P 21                      P 1 21 1
Hall group      P 2yb                     P 2yb
Moiety formula  C17 H22 O6                C17 H22 O6
Sum formula     C17 H22 O6                C17 H22 O6
Mr              322.35                    322.35
Dx,g cm-3       1.329                     1.329
Z               2                         2
Mu (mm-1)       0.836                     0.836
F000            344.0                     344.0
F000'           345.17
h,k,lmax        6,14,17                   6,14,17
Nref            3359[ 1766]               3141
Tmin,Tmax                                 0.547,1.000
Tmin'

Correction method= # Reported T Limits: Tmin=0.547 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.78/0.94       Theta(max)= 75.460

R(reflections)= 0.0333( 3123)      wR2(reflections)= 0.1007( 3141)


S = 0.971                  Npar= 212
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
CRYSC01_ALERT_1_C No recognised colour has been given for crystal colour.
PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full Low .......      0.971 Note
PLAT053_ALERT_1_C Minimum Crystal Dimension Missing (or Error) ...     Please Check
PLAT054_ALERT_1_C Medium  Crystal Dimension Missing (or Error) ...     Please Check
PLAT055_ALERT_1_C Maximum Crystal Dimension Missing (or Error) ...     Please Check
```

Alert level G

```
PLAT005_ALERT_5_G No Embedded Refinement Details found in the CIF      Please Do !
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          2 Report
PLAT791_ALERT_4_G The Model has Chirality at C3      (Chiral SPGR)          R Verify
PLAT791_ALERT_4_G The Model has Chirality at C7      (Chiral SPGR)          S Verify
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   5 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   4 ALERT level G = General information/check it is not something unexpected

   4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   0 ALERT type 2 Indicator that the structure model may be wrong or deficient
   1 ALERT type 3 Indicator that the structure quality may be low
   2 ALERT type 4 Improvement, methodology, query or suggestion
   2 ALERT type 5 Informative message, check
```

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_CRYSC01_574-4-4
;
PROBLEM: No recognised colour has been given for crystal colour.
RESPONSE: ...
;
_vrf_PLAT029_574-4-4
;
PROBLEM: _diffrn_measured_fraction_theta_full Low .......      0.971 Note
RESPONSE: ...
;
_vrf_PLAT053_574-4-4
;
PROBLEM: Minimum Crystal Dimension Missing (or Error) ...     Please Check
RESPONSE: ...
;
_vrf_PLAT054_574-4-4
;
PROBLEM: Medium  Crystal Dimension Missing (or Error) ...     Please Check
RESPONSE: ...
;
_vrf_PLAT055_574-4-4
;
PROBLEM: Maximum Crystal Dimension Missing (or Error) ...     Please Check
RESPONSE: ...
;
# end Validation Reply Form
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 19/11/2015; check.def file version of 17/11/2015

Datablock 574-4-4 - ellipsoid plot