

Supplementary data

**Pentasaccharide resin glycosides with multidrug resistance reversal activities
from the seeds of *Pharbitis nil***

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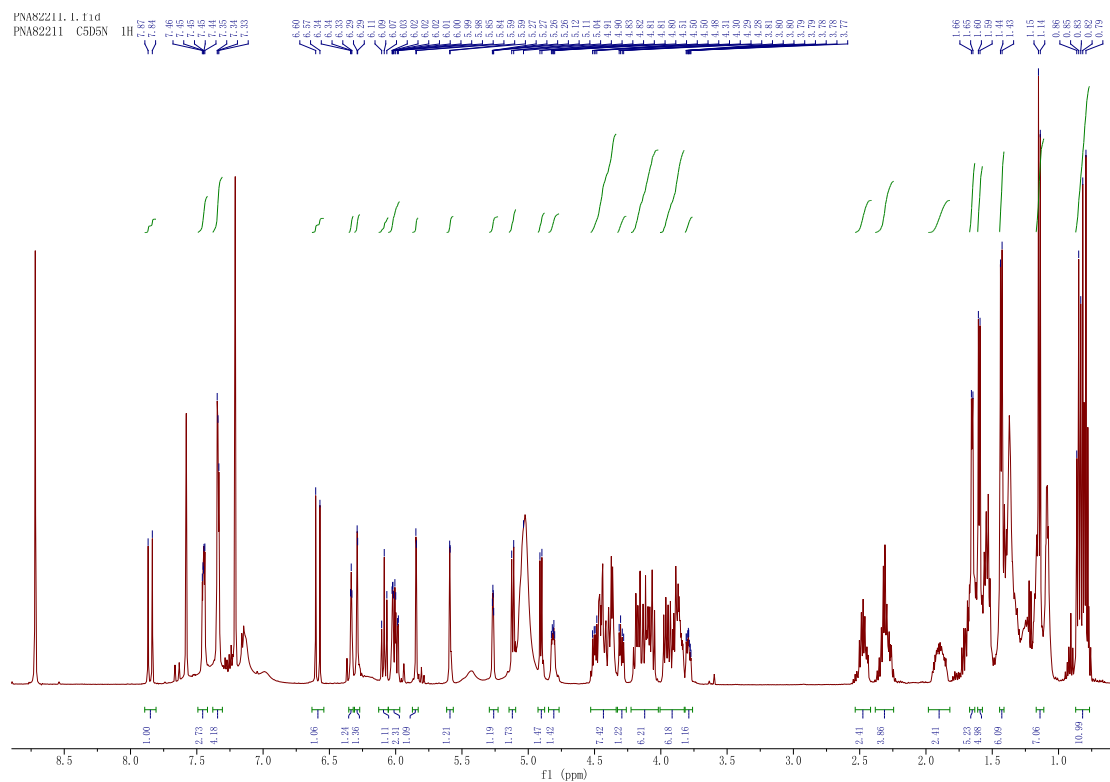


Figure S1. ¹H NMR (500MHz, pyridine-*d*₅) spectrum of pharbitin A (1)

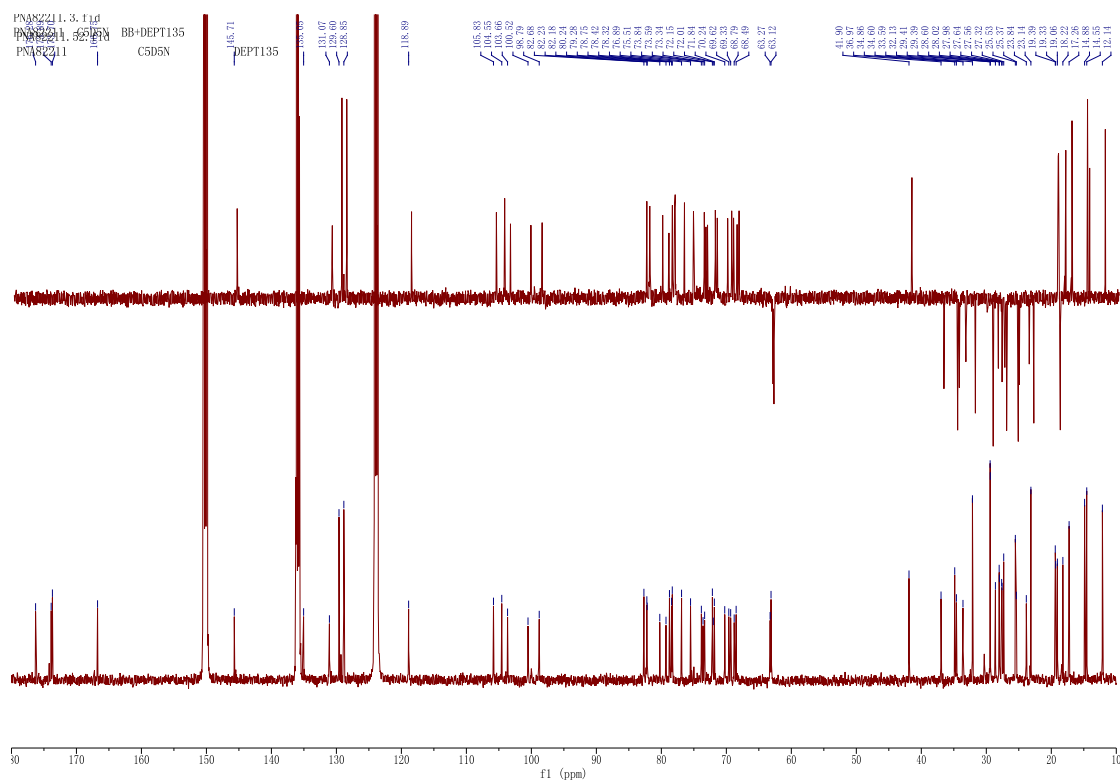


Figure S2. ¹³C NMR (125MHz, pyridine-*d*₅) spectrum of pharbitin A (1)

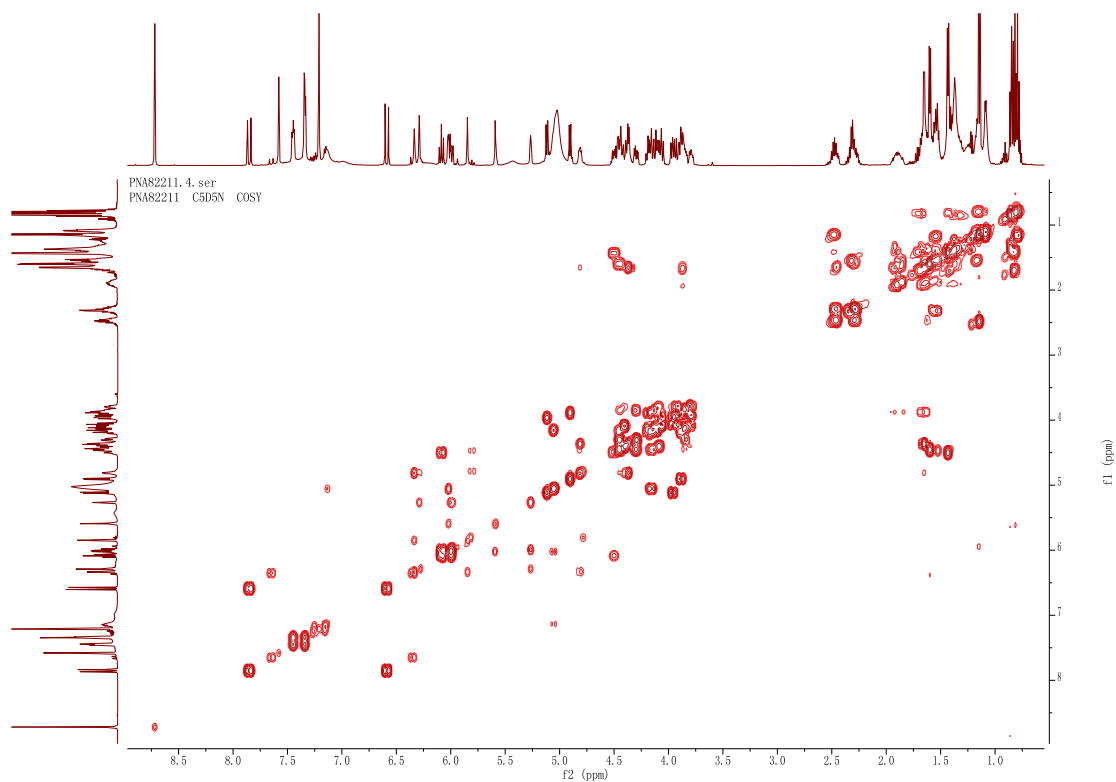


Figure S3. ^1H - ^1H COSY (500MHz, pyridine- d_5) spectrum of pharbitin A (1)

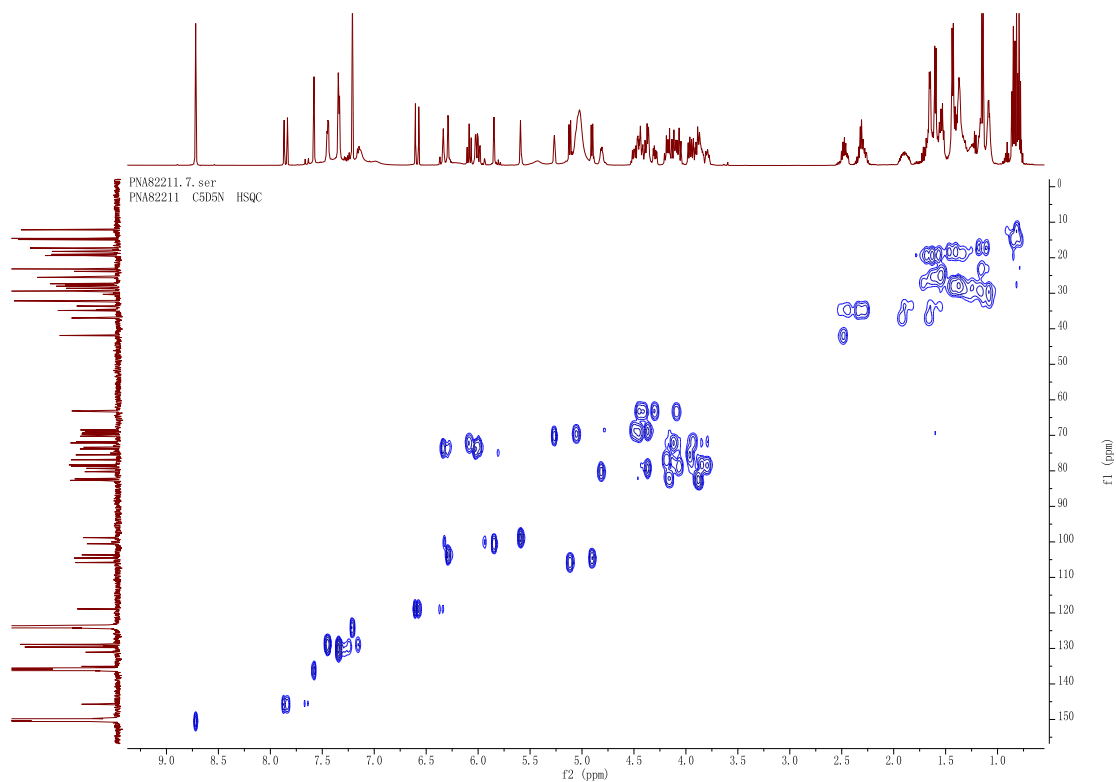


Figure S4. HSQC (500MHz, pyridine- d_5) spectrum of pharbitin A (1)

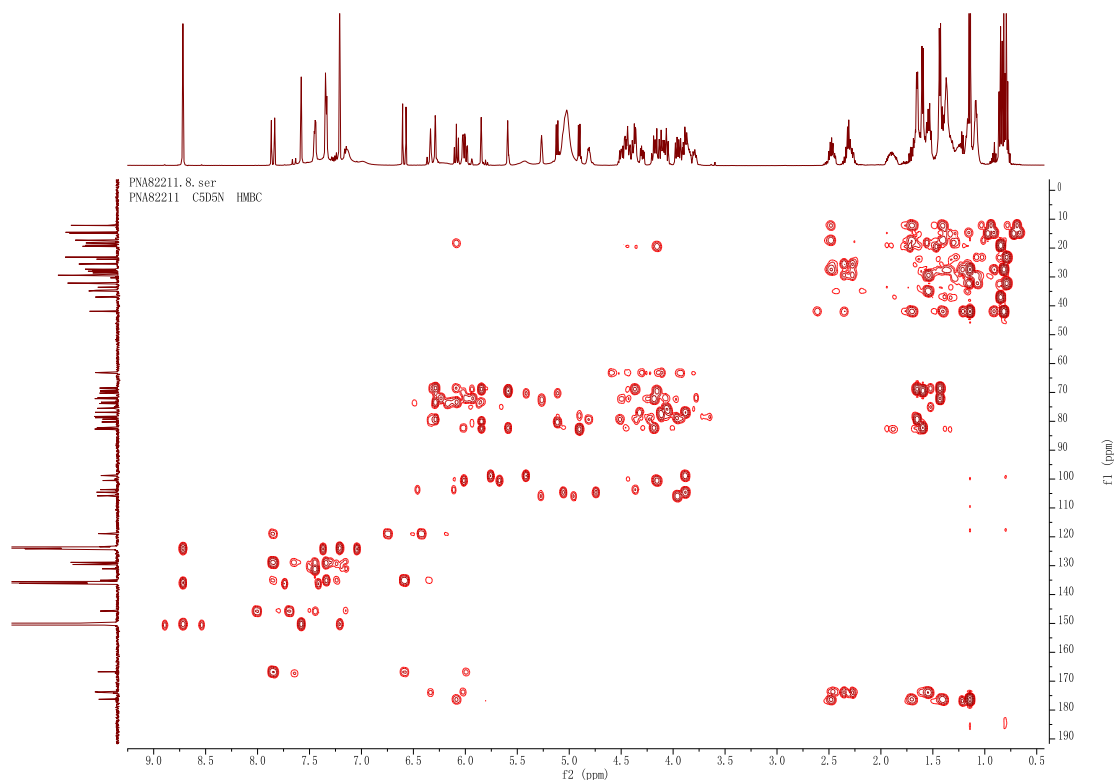


Figure S5. HMBC (500MHz, pyridine- d_5) spectrum of pharbitin A (**1**)

User Spectra

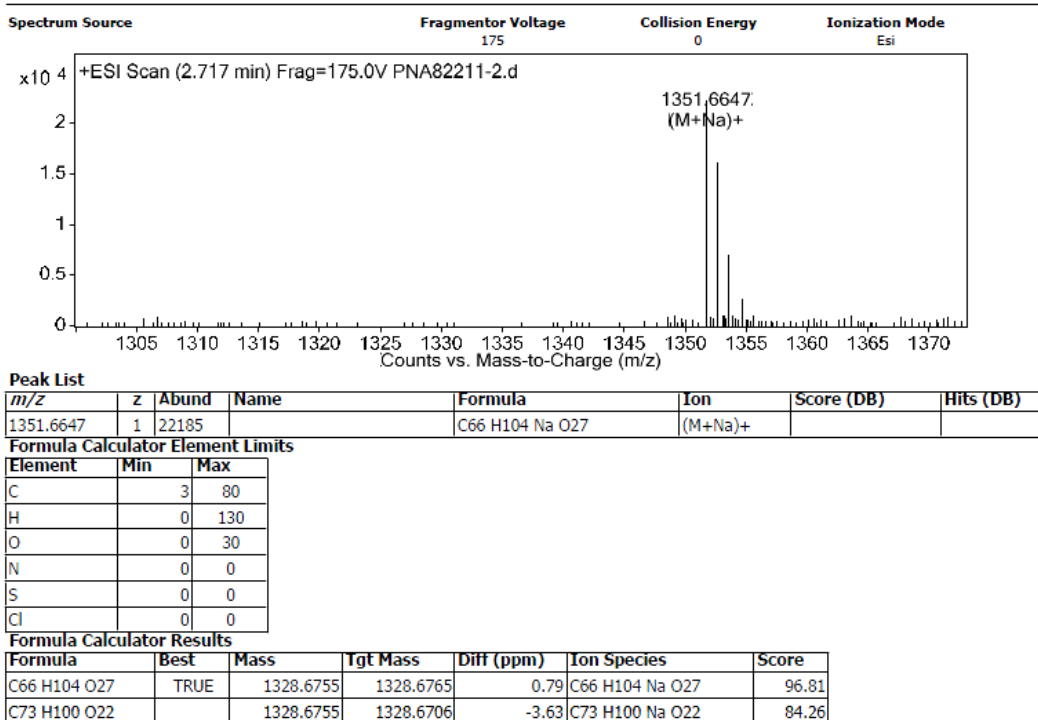


Figure S6. HRESIMS spectrum of pharbitin A (**1**)

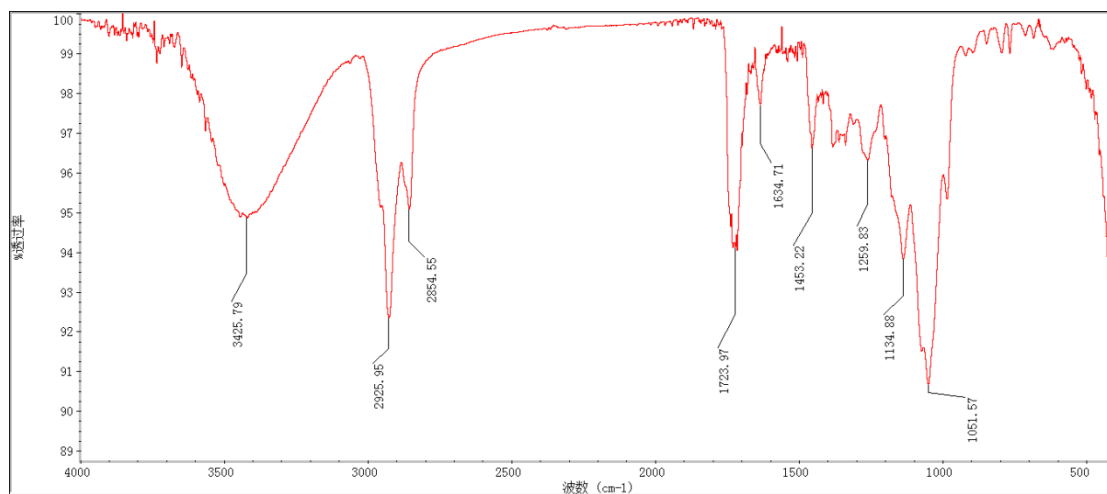


Figure S7. IR (KBr disc) spectrum of pharbitin A (1)

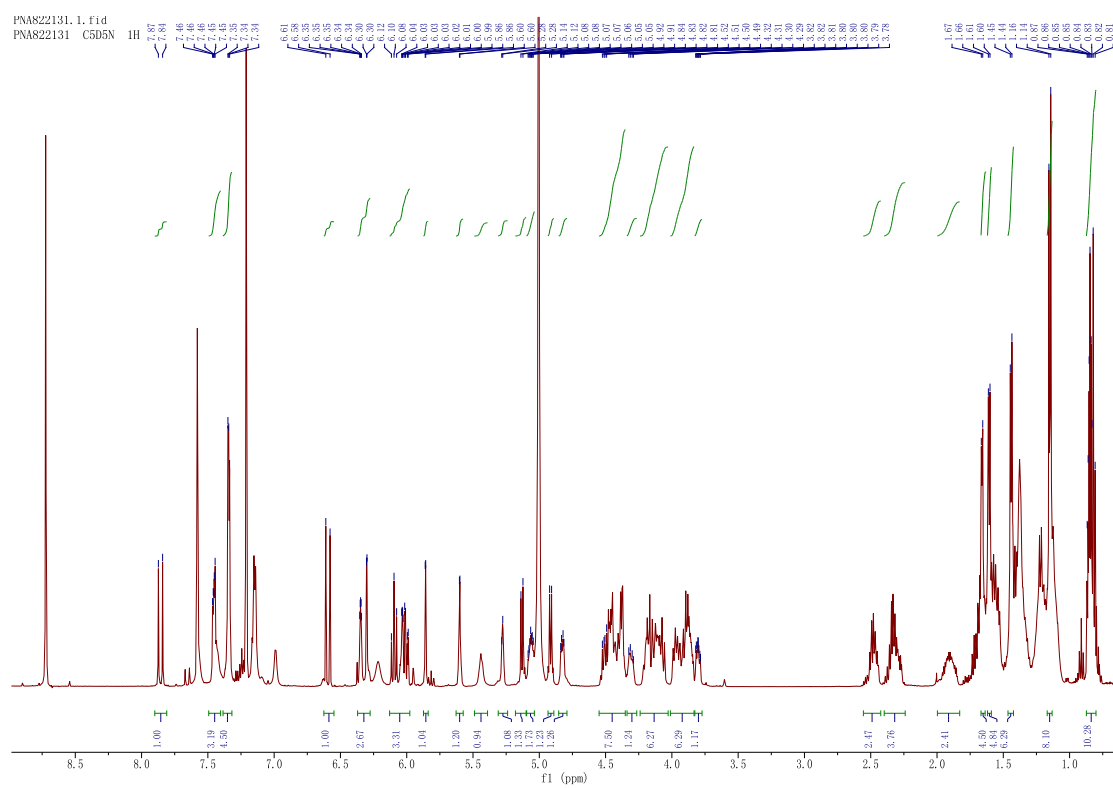


Figure S8. ^1H NMR (500MHz, pyridine- d_5) spectrum of pharbitin B (2)

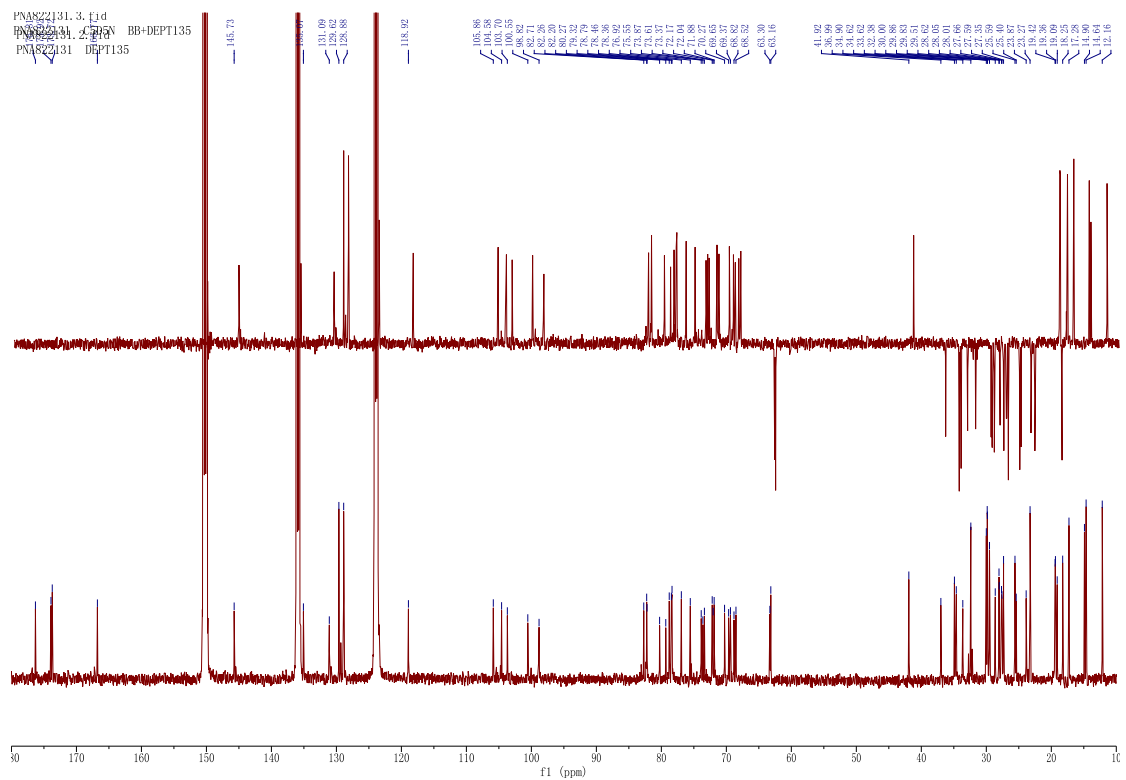


Figure S9. ^{13}C NMR (125MHz, pyridine- d_5) spectrum of pharbitin B (2)

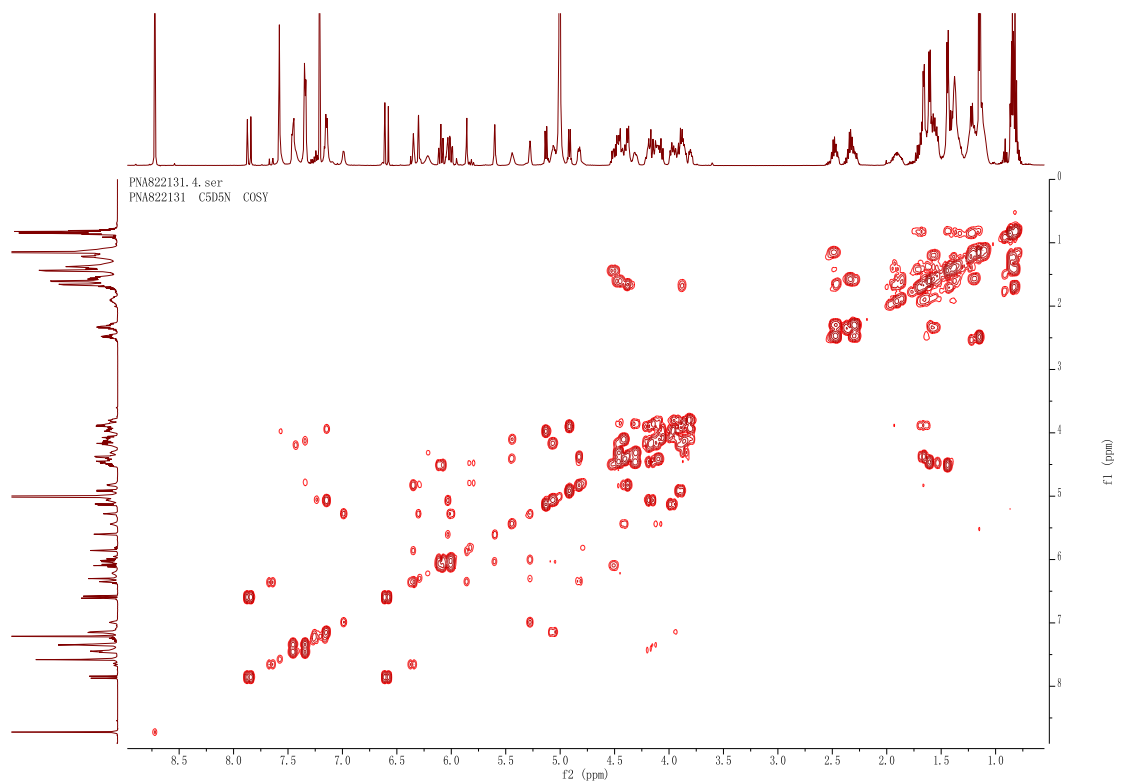


Figure S10. ^1H - ^1H COSY (500MHz, pyridine- d_5) spectrum of pharbitin B (2)

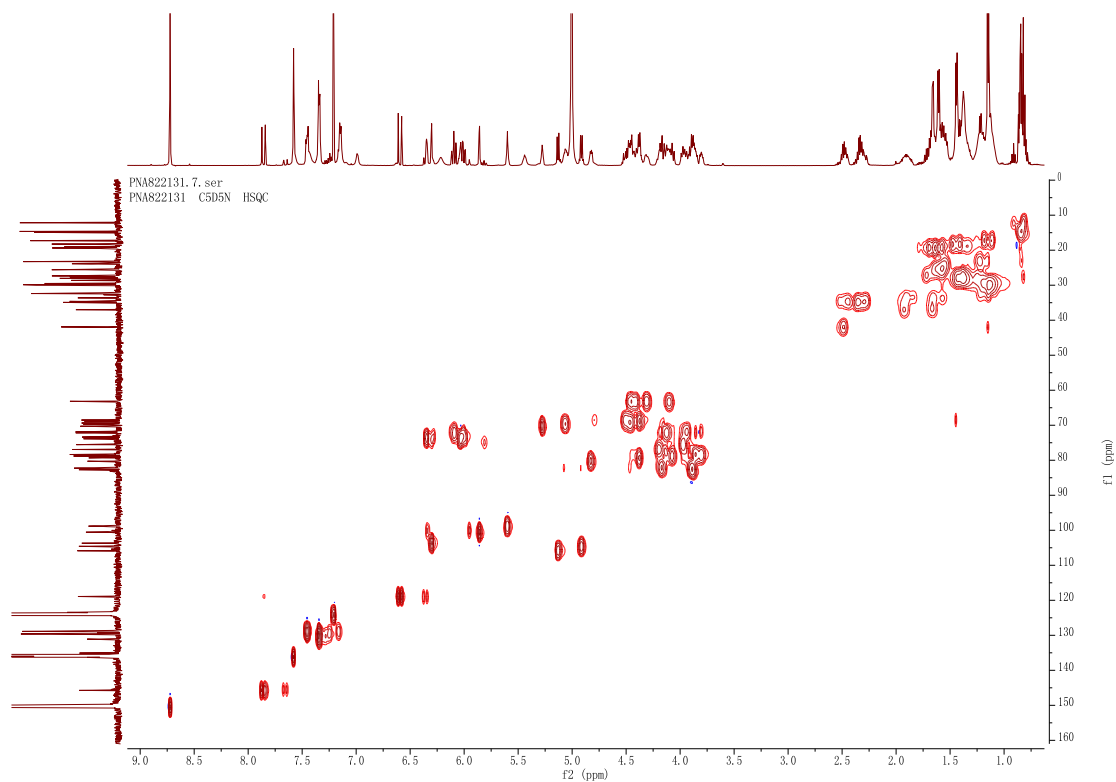


Figure S11. HSQC (500MHz, pyridine-*d*₅) spectrum of pharbitin B (2)

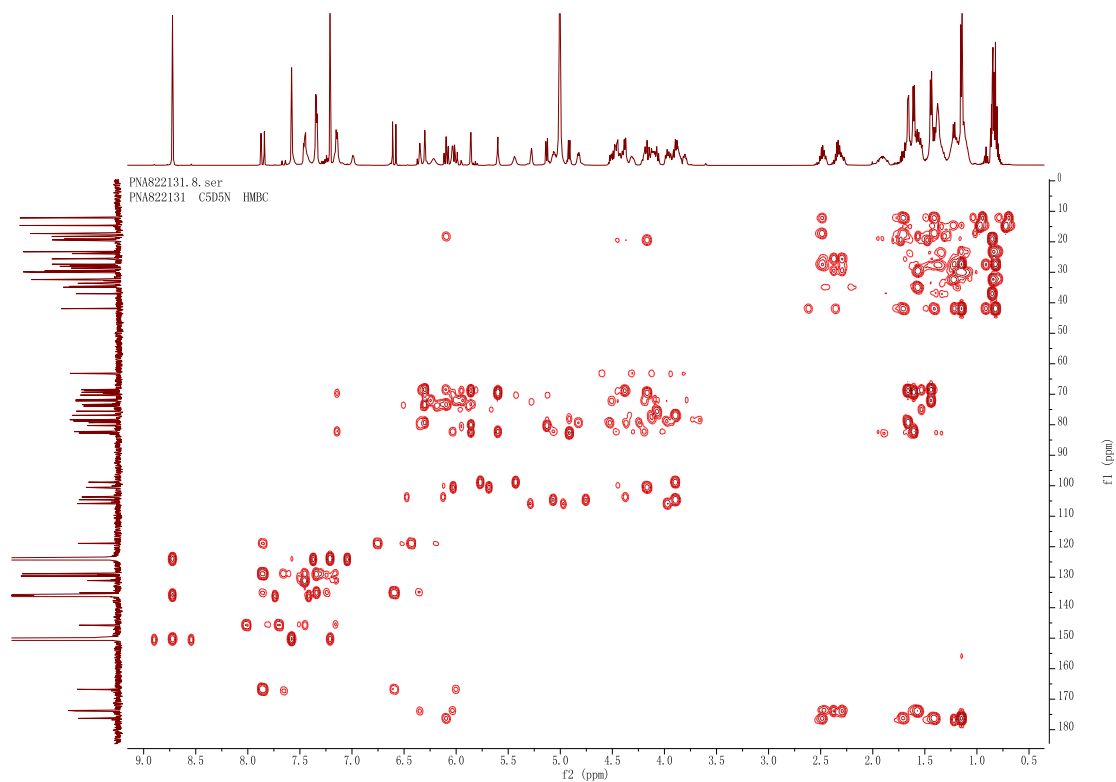


Figure S12. HMBC (500MHz, pyridine-*d*₅) spectrum of pharbitin B (2)

User Spectra

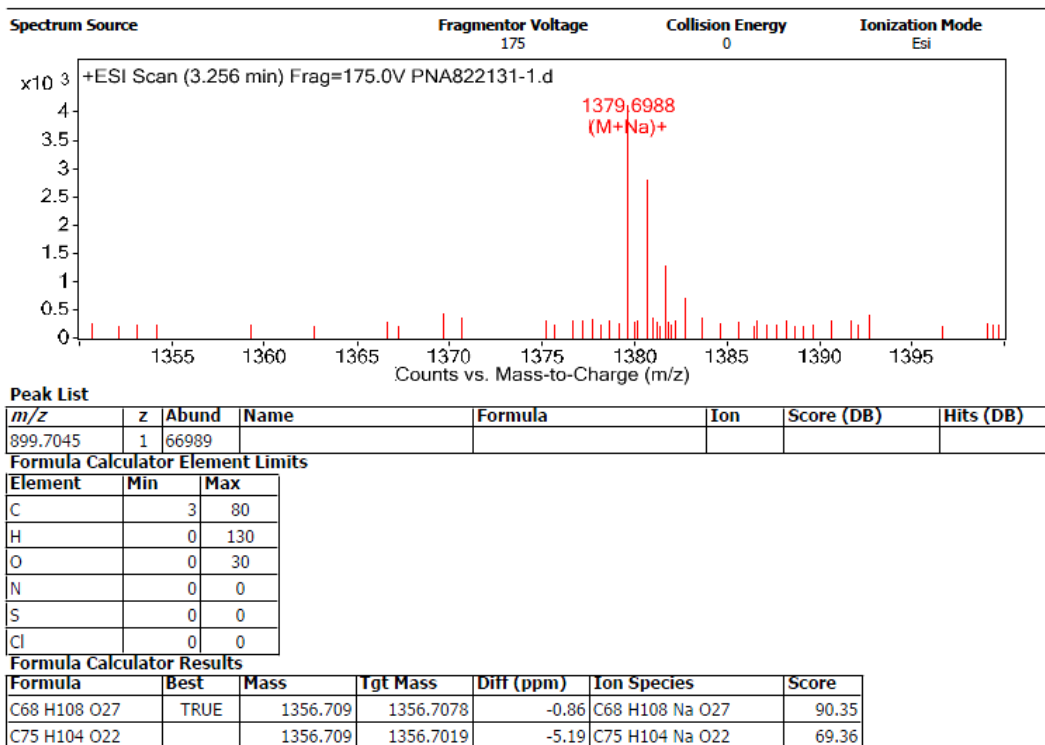


Figure S13. HRESIMS spectrum of pharbitin B (2)

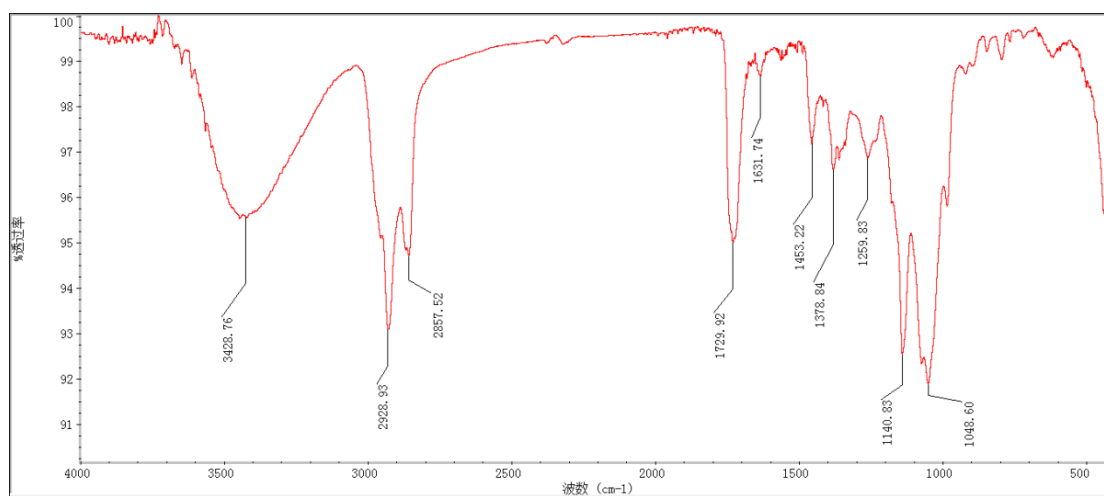


Figure S14. IR (KBr disc) spectrum of pharbitin B (2)

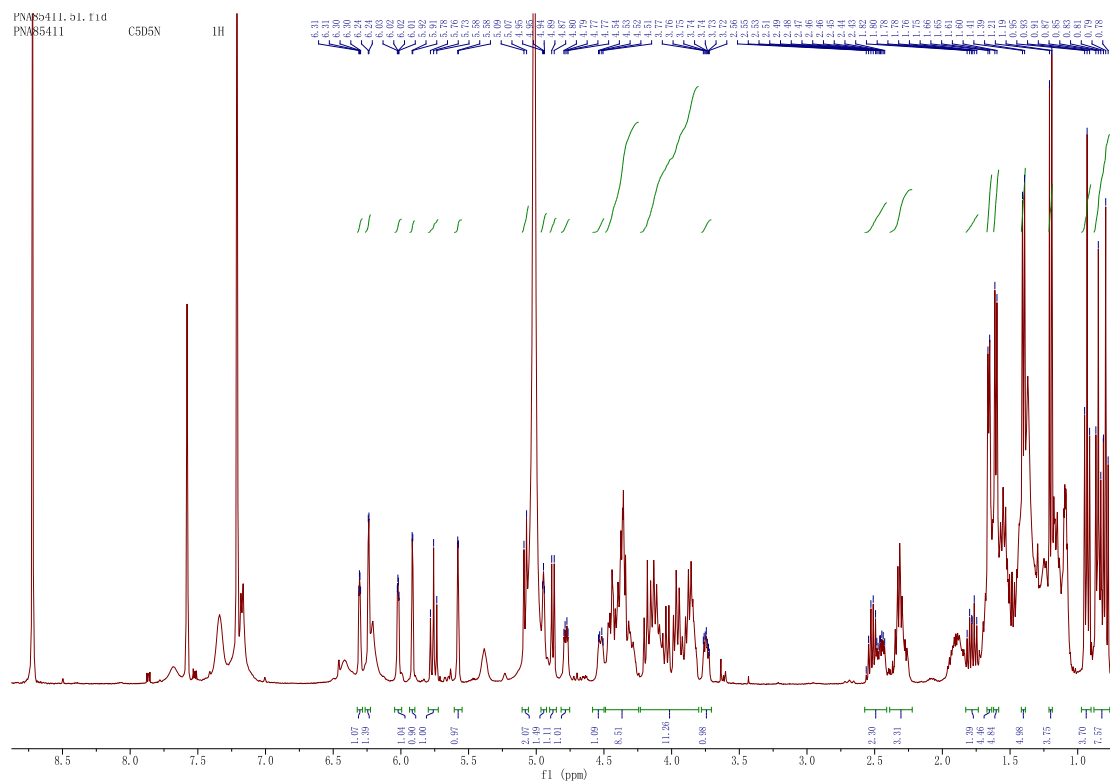


Figure S15. ^1H NMR (500MHz, pyridine- d_5) spectrum of pharbitin C (**3**)

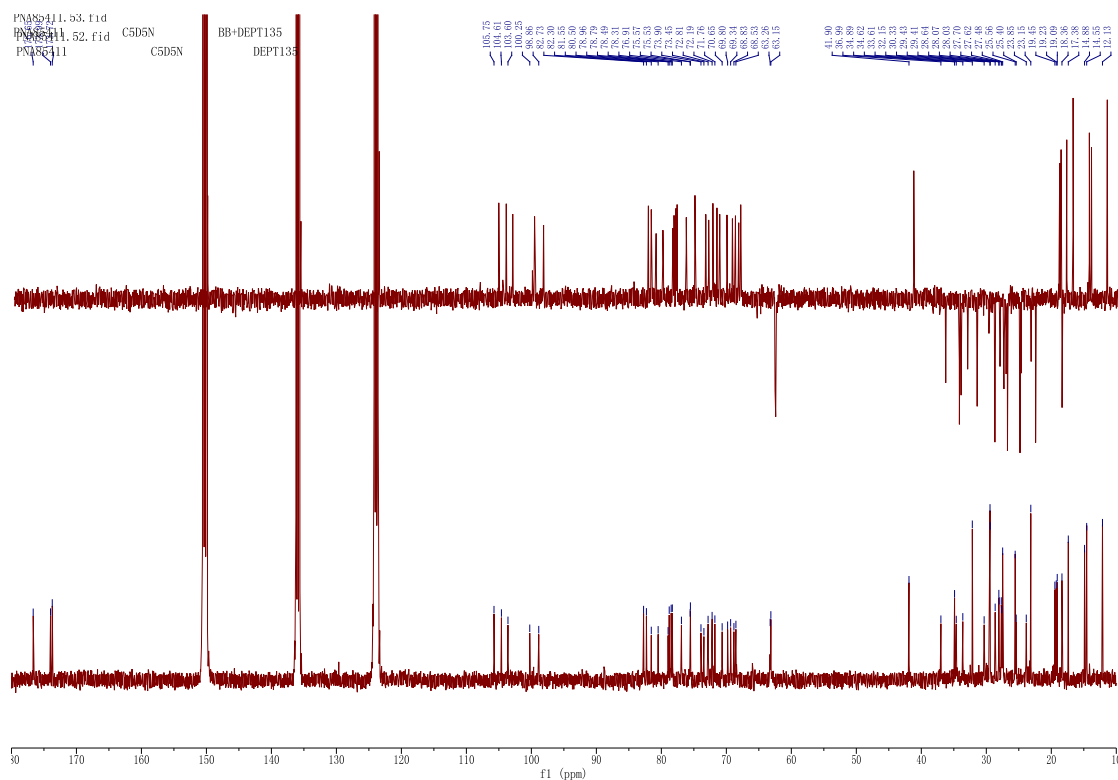


Figure S16. ^{13}C NMR (125MHz, pyridine- d_5) spectrum of pharbitin C (**3**)

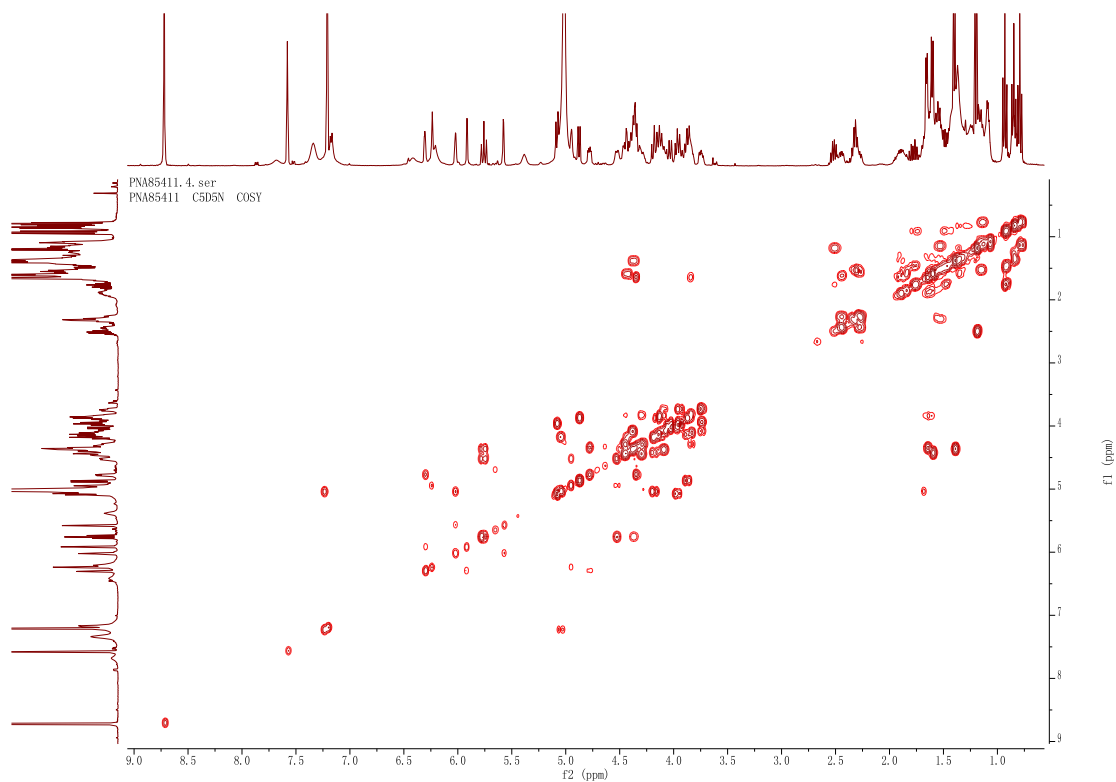


Figure S17. ^1H - ^1H COSY (500MHz, pyridine- d_5) spectrum of pharbitin C (3)

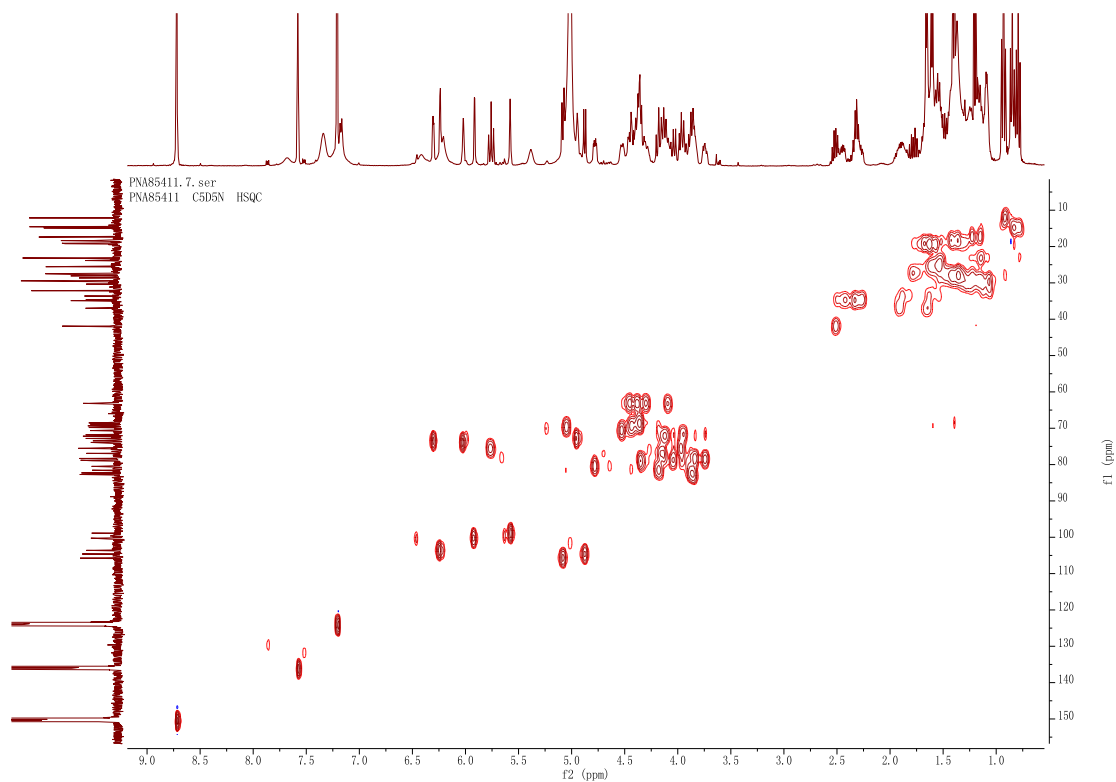


Figure S18. HSQC (500MHz, pyridine- d_5) spectrum of pharbitin C (3)

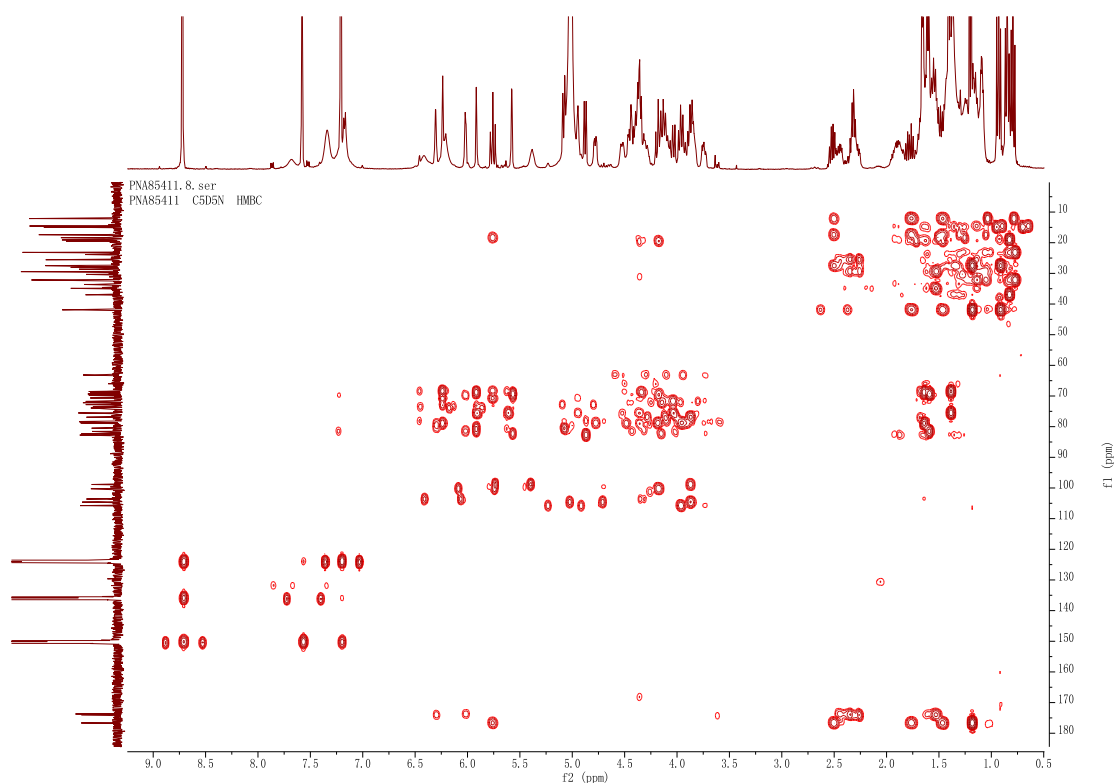
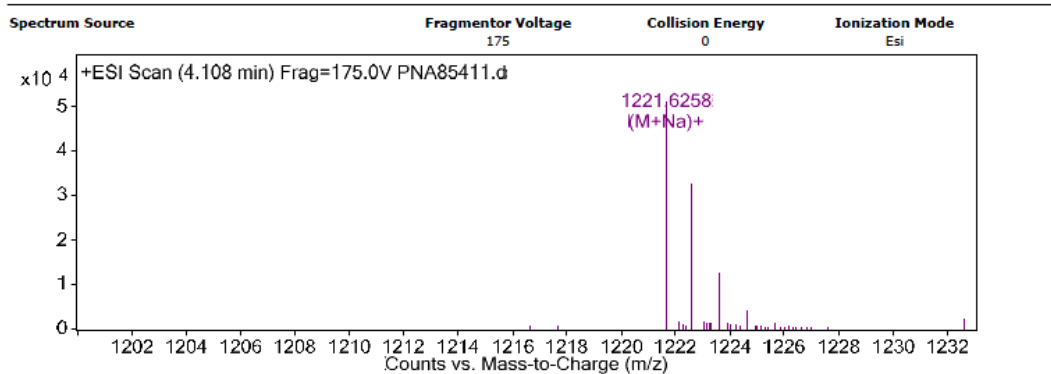


Figure S19. HMBC (500MHz, pyridine- d_5) spectrum of pharbitin C (3)

User Spectra



Peak List

m/z	z	Abund	Name	Formula	Ion	Score (DB)	Hits (DB)
1221.6258	1	51018		C57 H98 Na O26	(M+Na)+		

Formula Calculator Element Limits

Element	Min	Max
C	3	80
H	0	130
O	0	30
N	0	0
S	0	0
Cl	0	0

Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C57 H98 O26	TRUE	1198.6366	1198.6346	-1.6	C57 H98 Na O26	97.41
C64 H94 O21		1198.6366	1198.6288	-6.5	C64 H94 Na O21	71.88
C68 H94 O18		1198.6366	1198.644	6.23	C68 H94 Na O18	71.05

Figure S20. HRESIMS spectrum of pharbitin C (3)

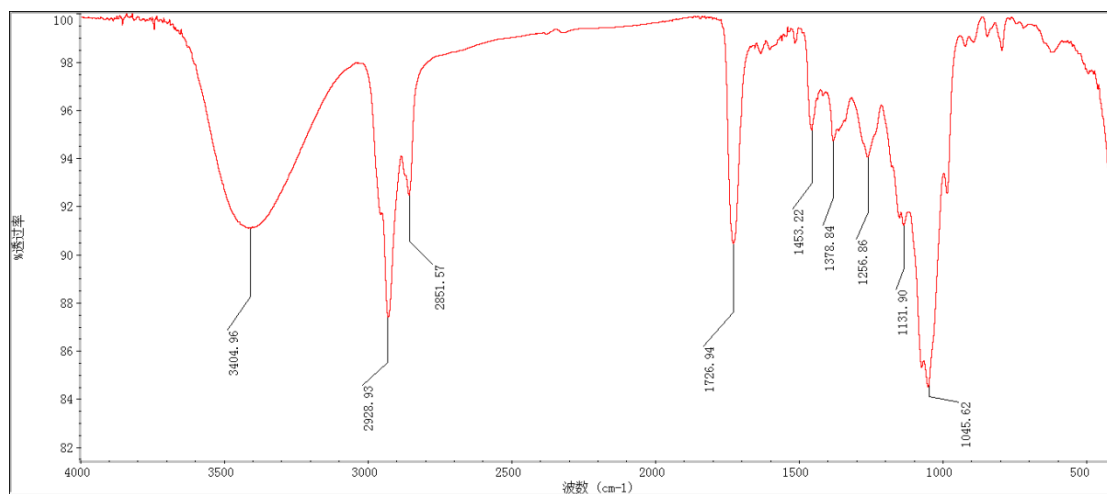


Figure S21. IR (KBr disc) spectrum of pharbitin C (3)

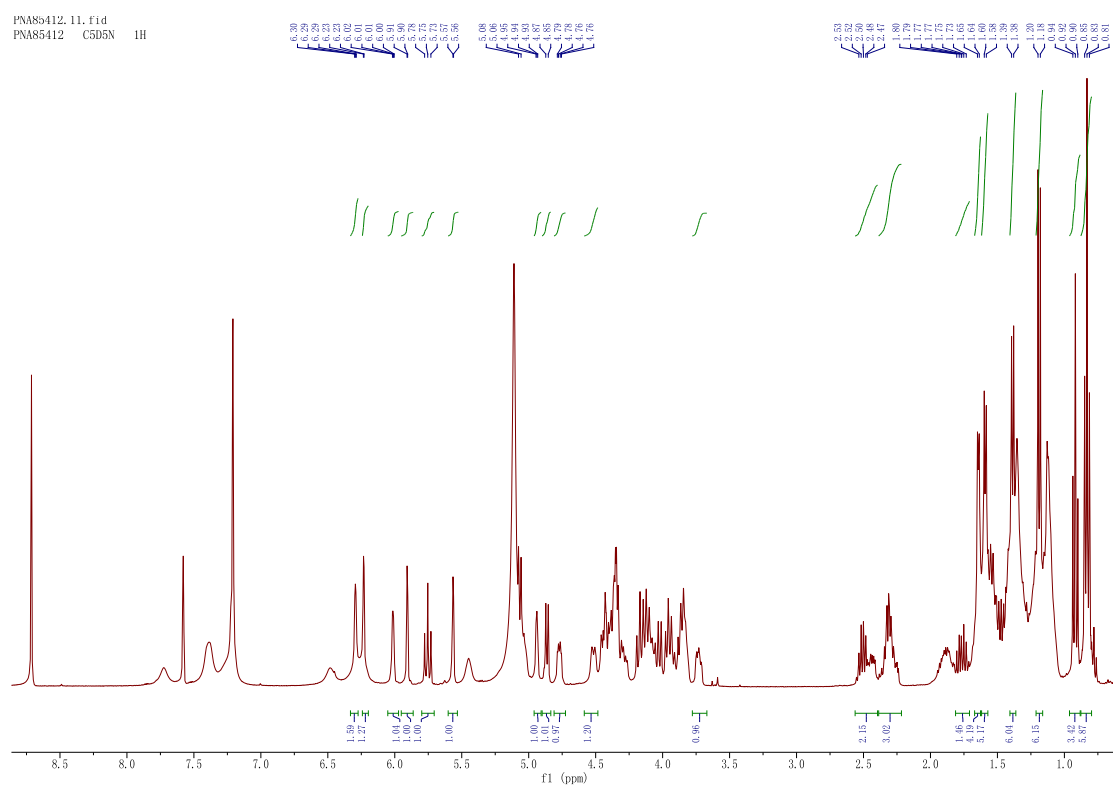


Figure S22. ^1H NMR (500MHz, pyridine- d_5) spectrum of pharbitin D (4)

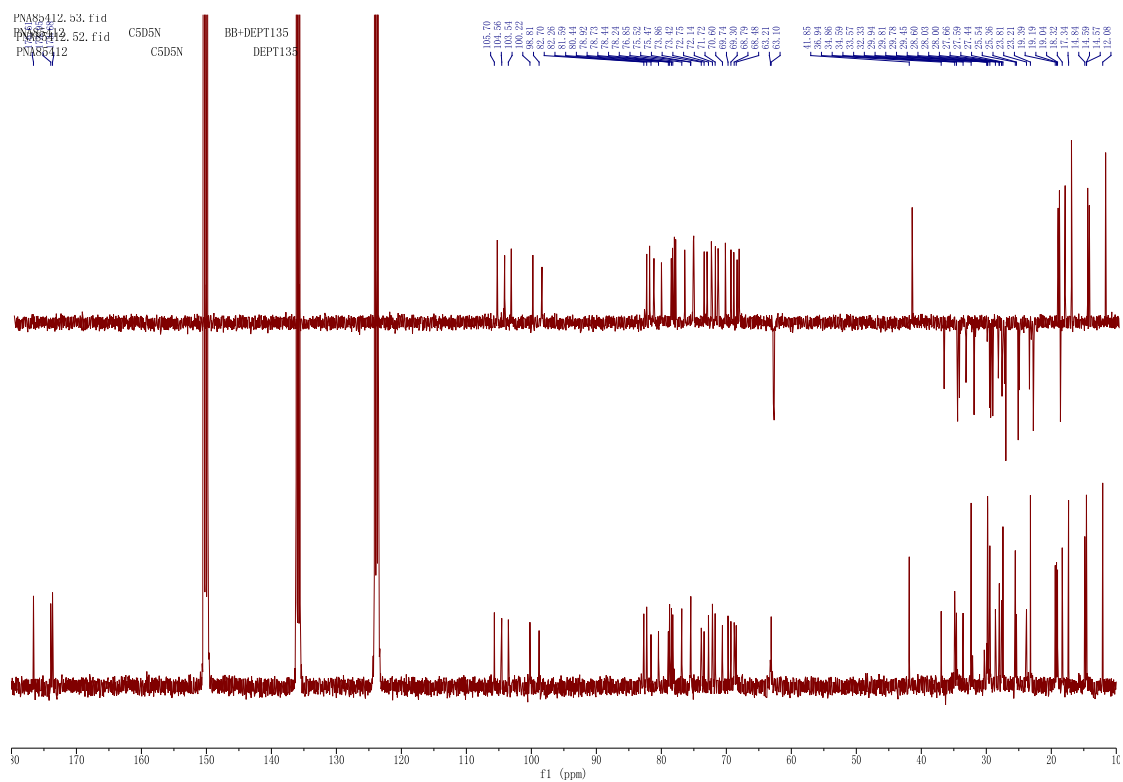


Figure S23. ^{13}C NMR (125MHz, pyridine- d_5) spectrum of pharbitin D (4)

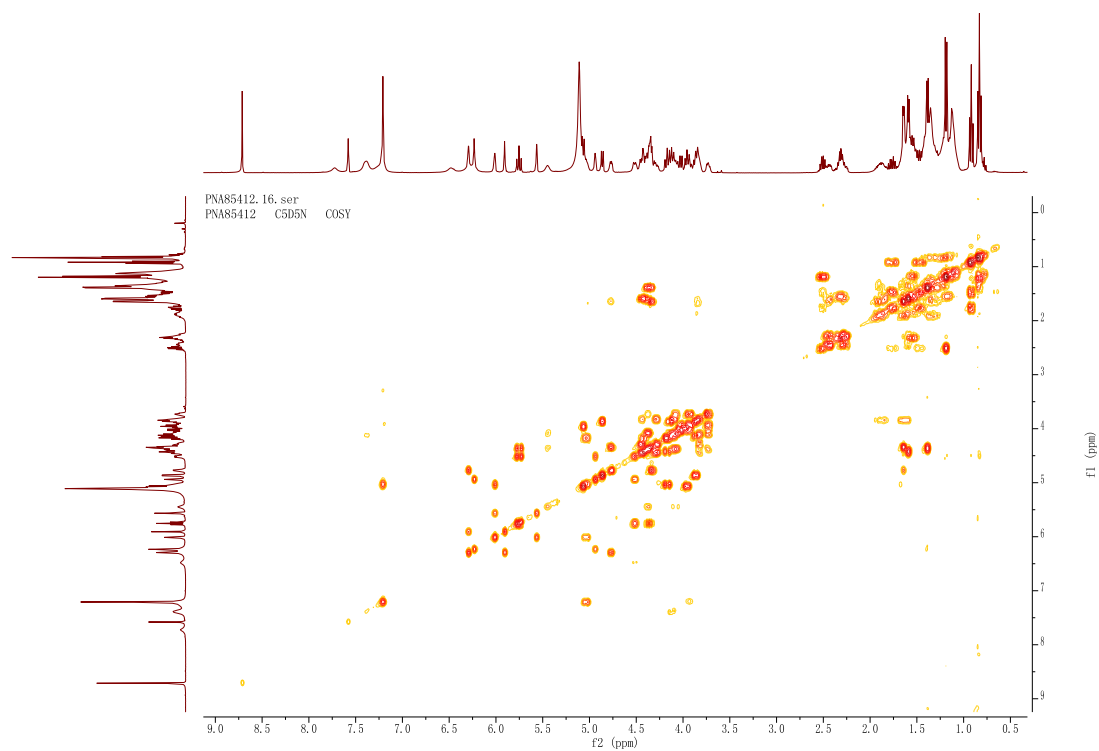


Figure S24. ^1H - ^1H COSY (500MHz, pyridine- d_5) spectrum of pharbitin D (4)

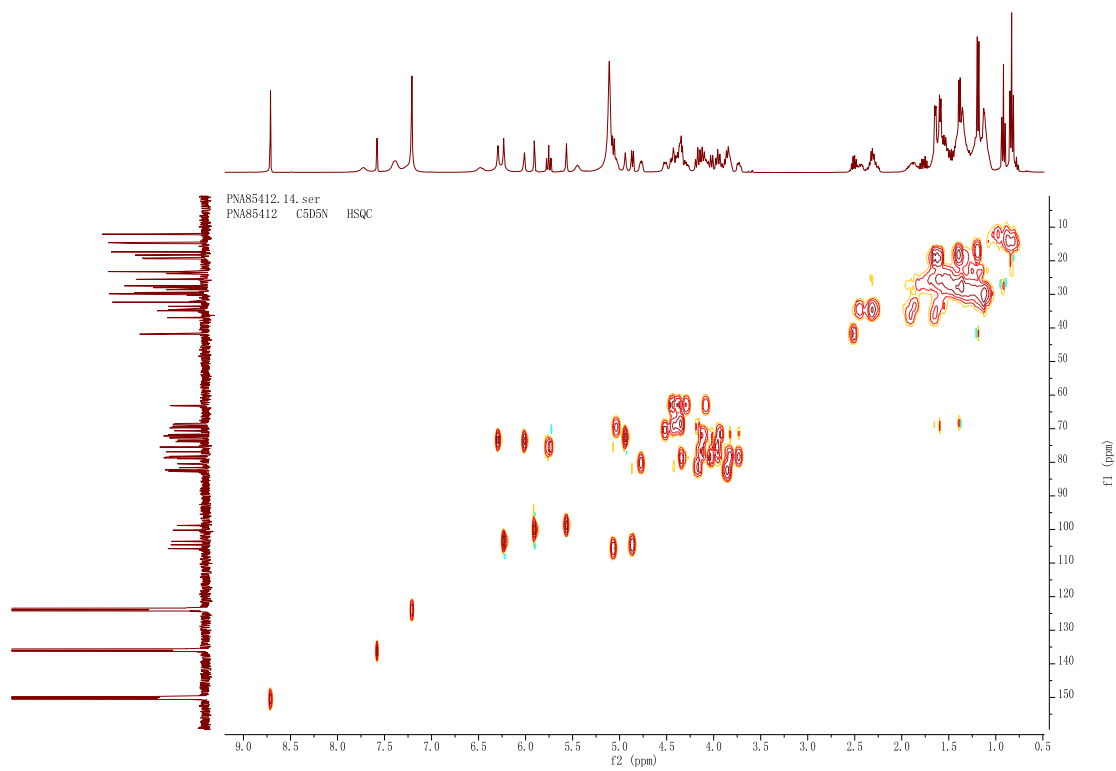


Figure S25. HSQC (500MHz, pyridine-*d*₅) spectrum of pharbitin D (4)

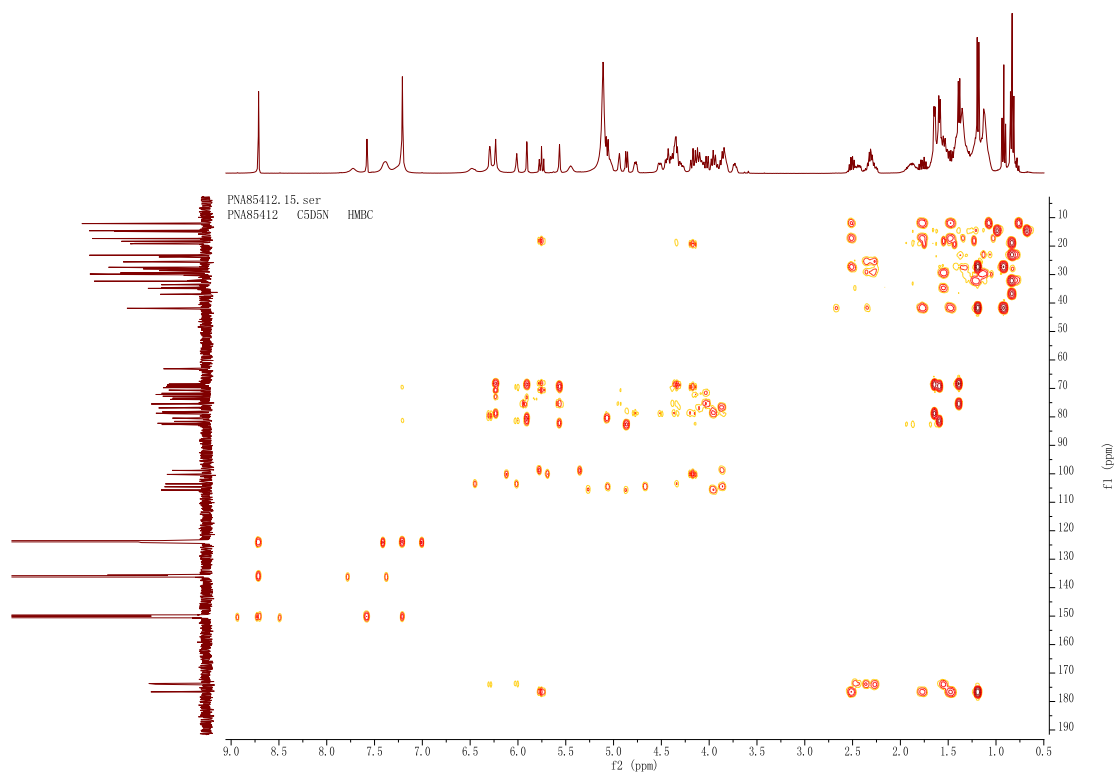
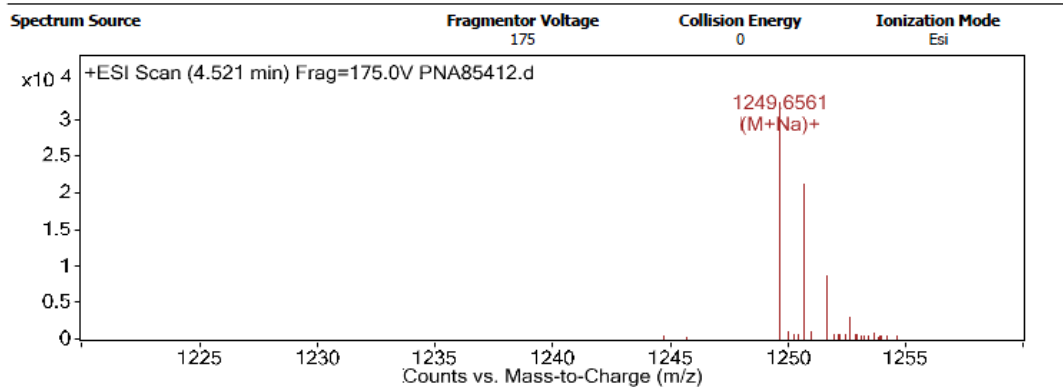


Figure S26. HMBC (500MHz, pyridine-*d*₅) spectrum of pharbitin D (4)

User Spectra



Peak List

m/z	z	Abund	Name	Formula	Ion	Score (DB)	Hits (DB)
393.2989	1	97080					

Formula Calculator Element Limits

Element	Min	Max
C	3	80
H	0	130
O	0	30
N	0	0
S	0	0
Cl	0	0

Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C59 H102 O26	TRUE	1226.6671	1226.6659	-0.96	C59 H102 Na O26	98.77
C66 H98 O21		1226.6671	1226.6601	-5.75	C66 H98 Na O21	75.89
C70 H98 O18		1226.6671	1226.6753	6.69	C70 H98 Na O18	68.73

Figure S27. HRESIMS spectrum of pharbitin D (4)

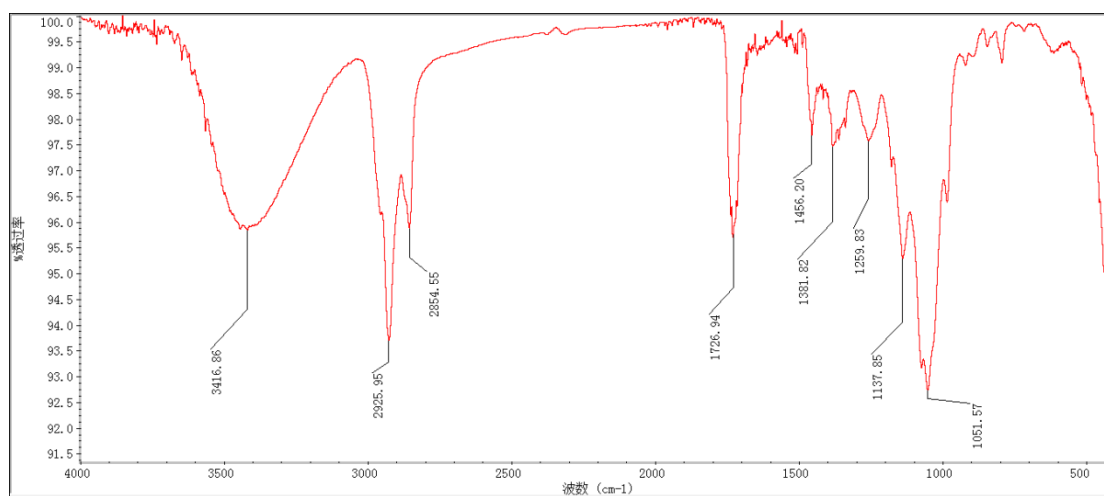


Figure S28. IR (KBr disc) spectrum of pharbitin D (4)

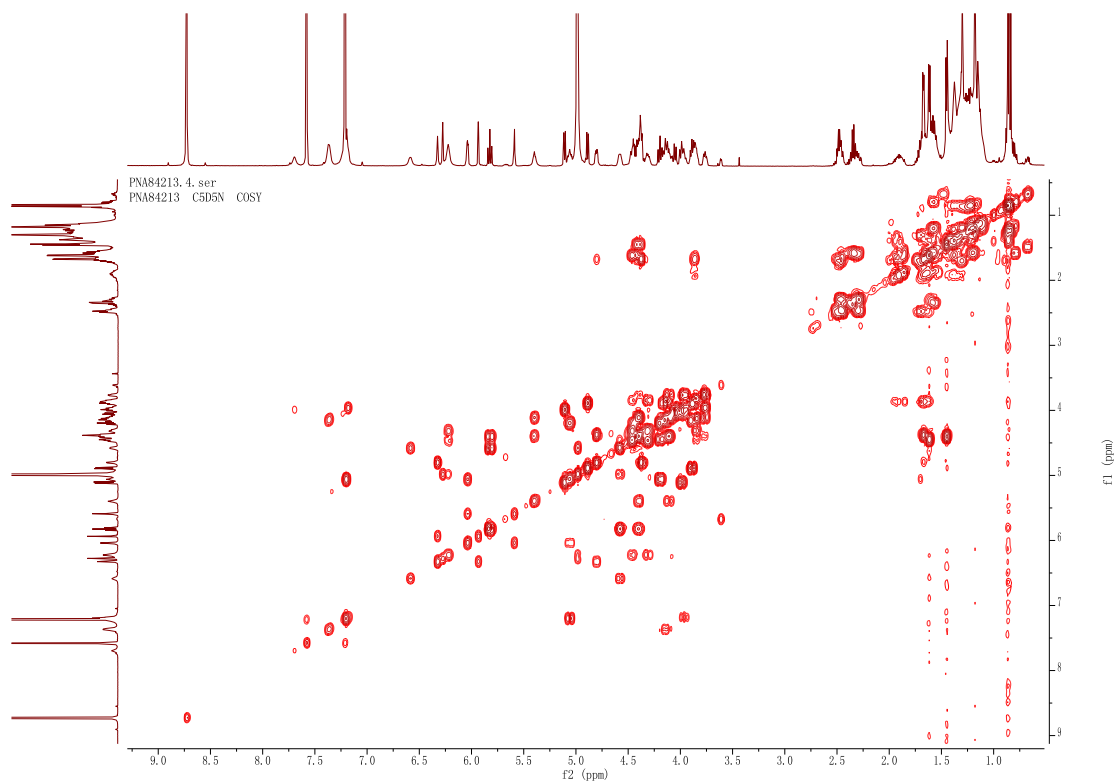


Figure S31. ^1H - ^1H COSY (500MHz, pyridine- d_5) spectrum of pharbitin E (5)

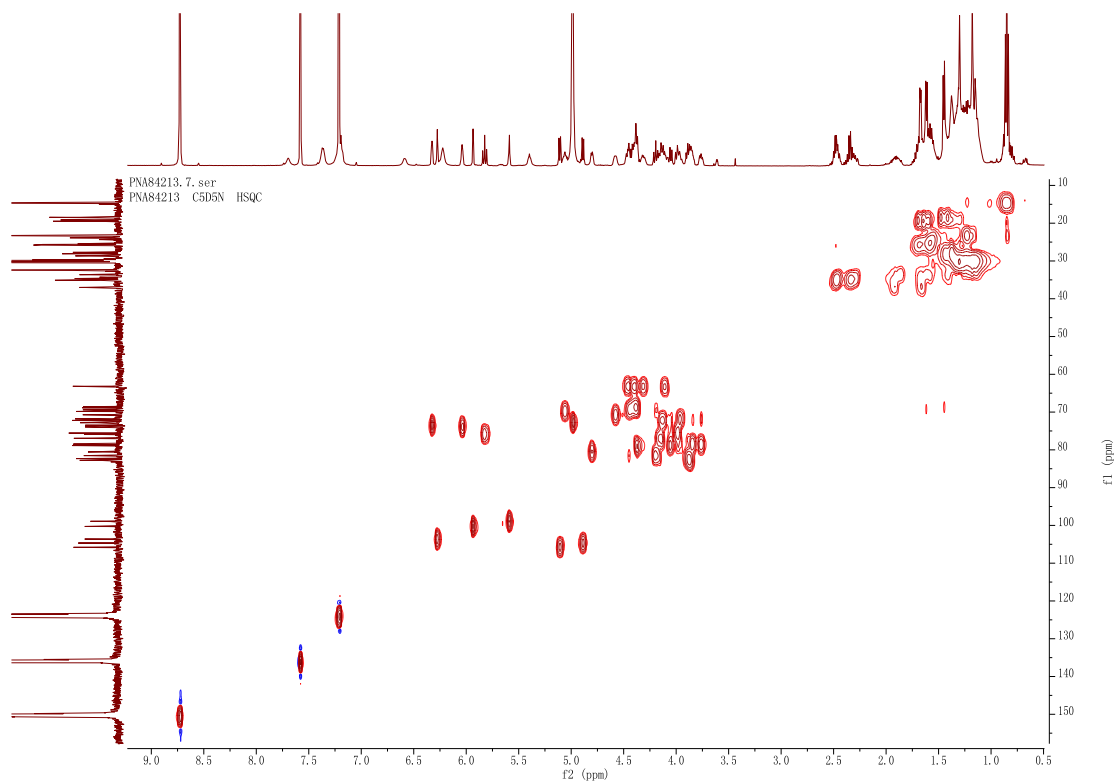


Figure S32. HSQC (500MHz, pyridine- d_5) spectrum of pharbitin E (5)

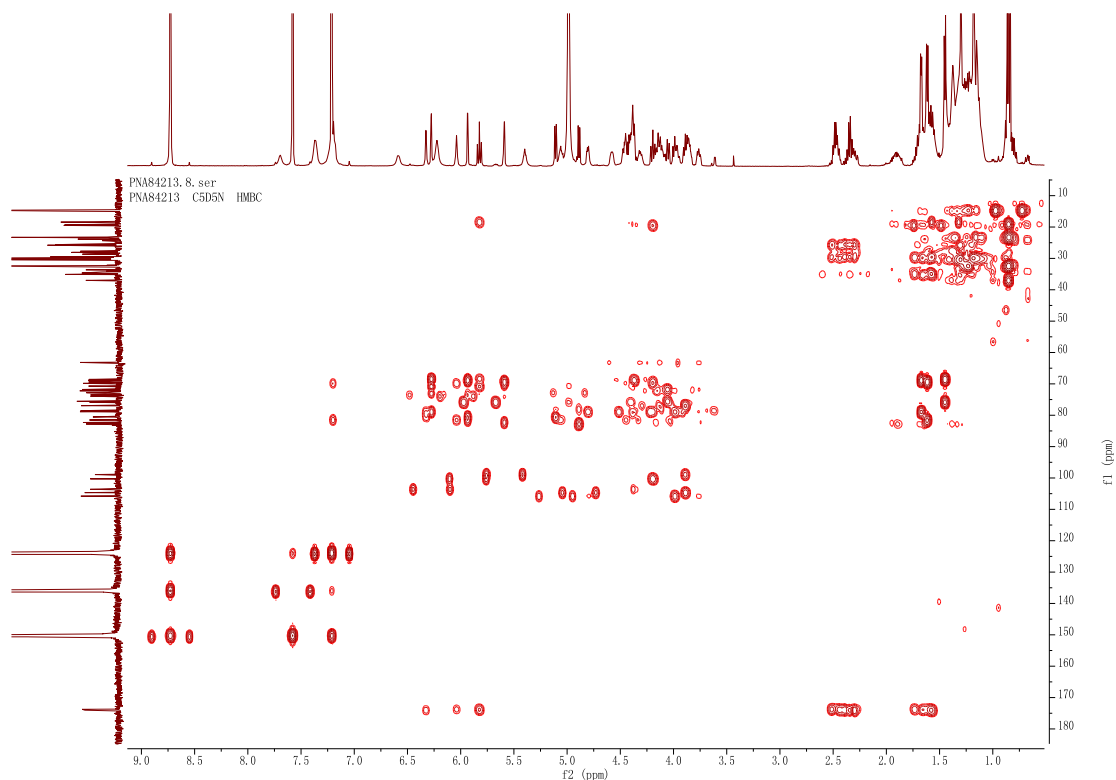
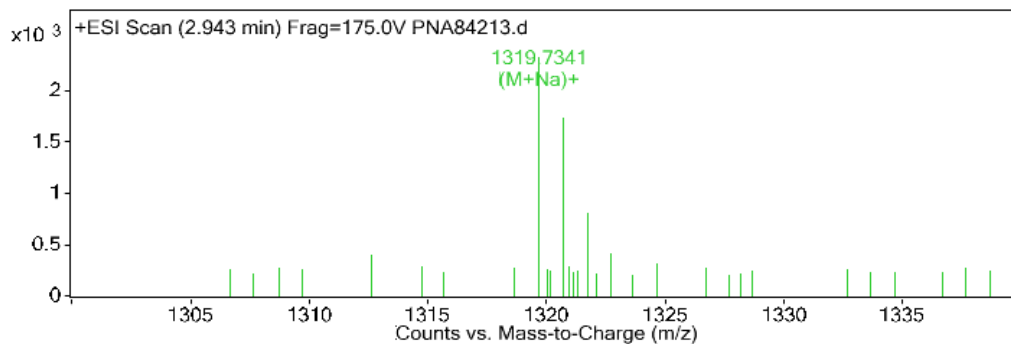


Figure S33. HMBC (500MHz, pyridine- d_5) spectrum of pharbitin E (5)

User Spectra

Spectrum Source Fragmentor Voltage Collision Energy Ionization Mode



Peak List

m/z	z	Abund	Name	Formula	Ion	Score (DB)	Hits (DB)
901.7278	1	29289					

Formula Calculator Element Limits

Element	Min	Max
C	3	80
H	0	130
O	0	30
N	0	0
S	0	0
Cl	0	0

Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C64 H112 O26	TRUE	1296.7448	1296.7442	-0.47	C64 H112 Na O26	90.87
C71 H108 O21		1296.7448	1296.7383	-5	C71 H108 Na O21	73.16
C75 H108 O18		1296.7448	1296.7536	6.76	C75 H108 Na O18	62.82

Figure S34. HRESIMS spectrum of pharbitin E (5)

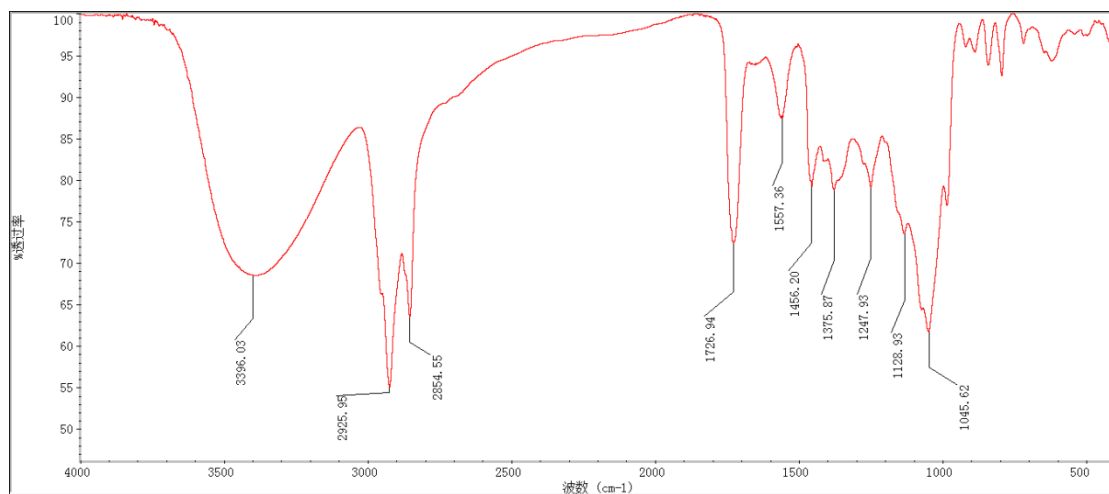


Figure S35. IR (KBr disc) spectrum of pharbitin E (5)

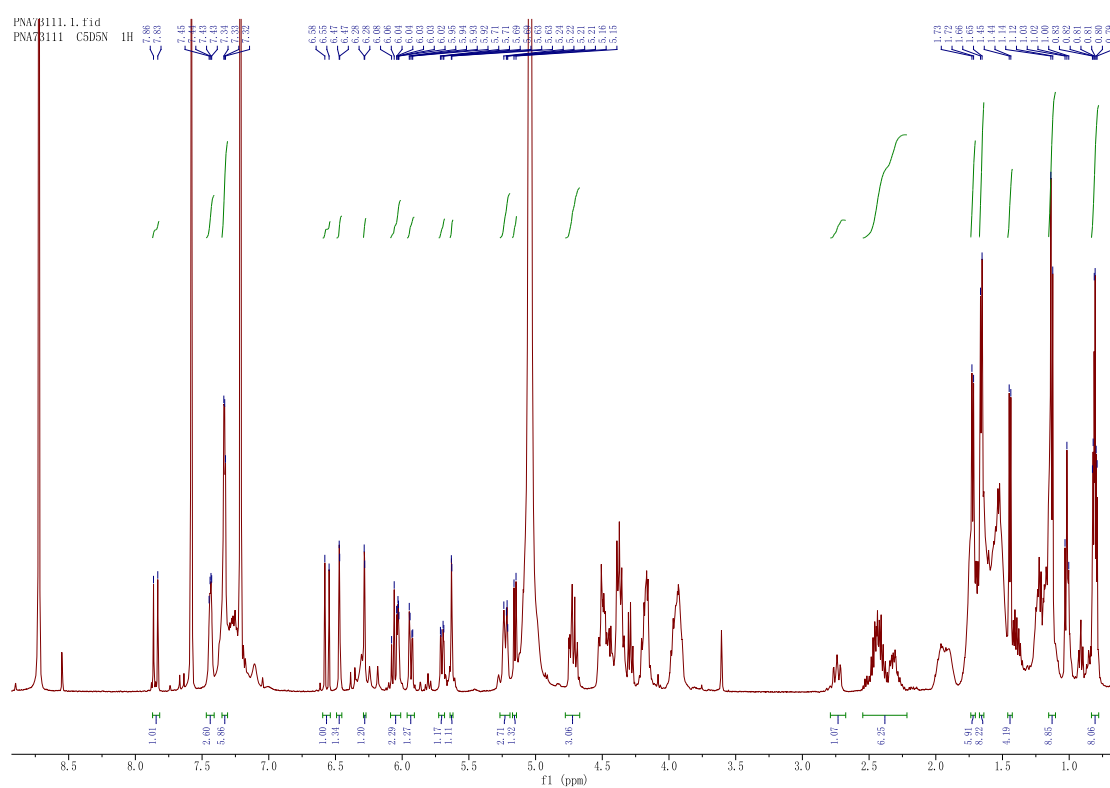


Figure S36. ^1H NMR (500MHz, pyridine- d_5) spectrum of pharbitin F (6)

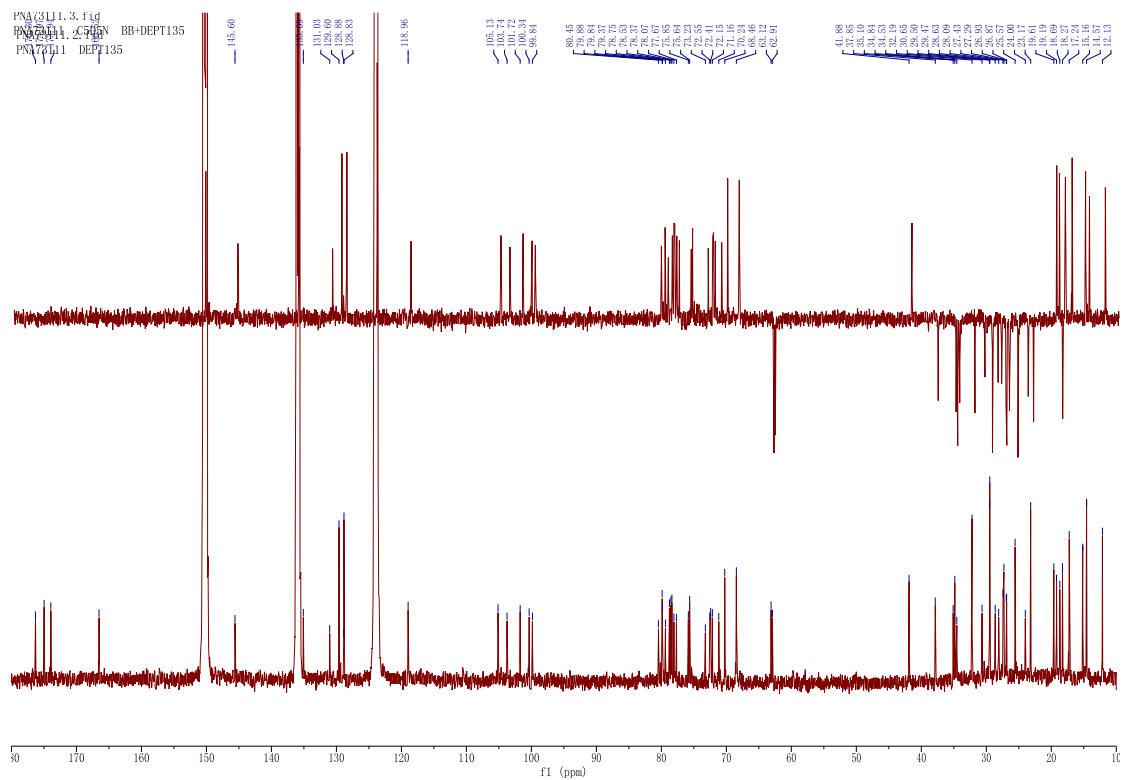


Figure S37. ^{13}C NMR (125MHz, pyridine- d_5) spectrum of pharbitin F (6)

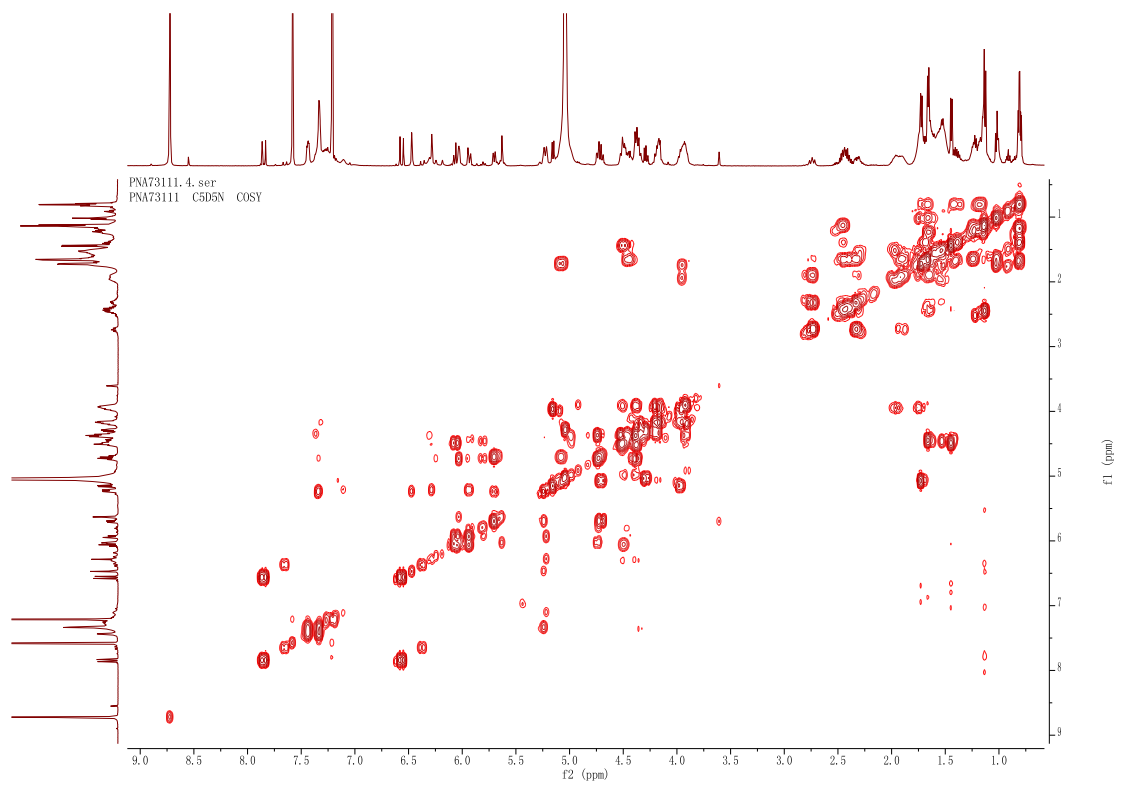


Figure S38. ^1H - ^1H COSY (500MHz, pyridine- d_5) spectrum of pharbitin F (6)

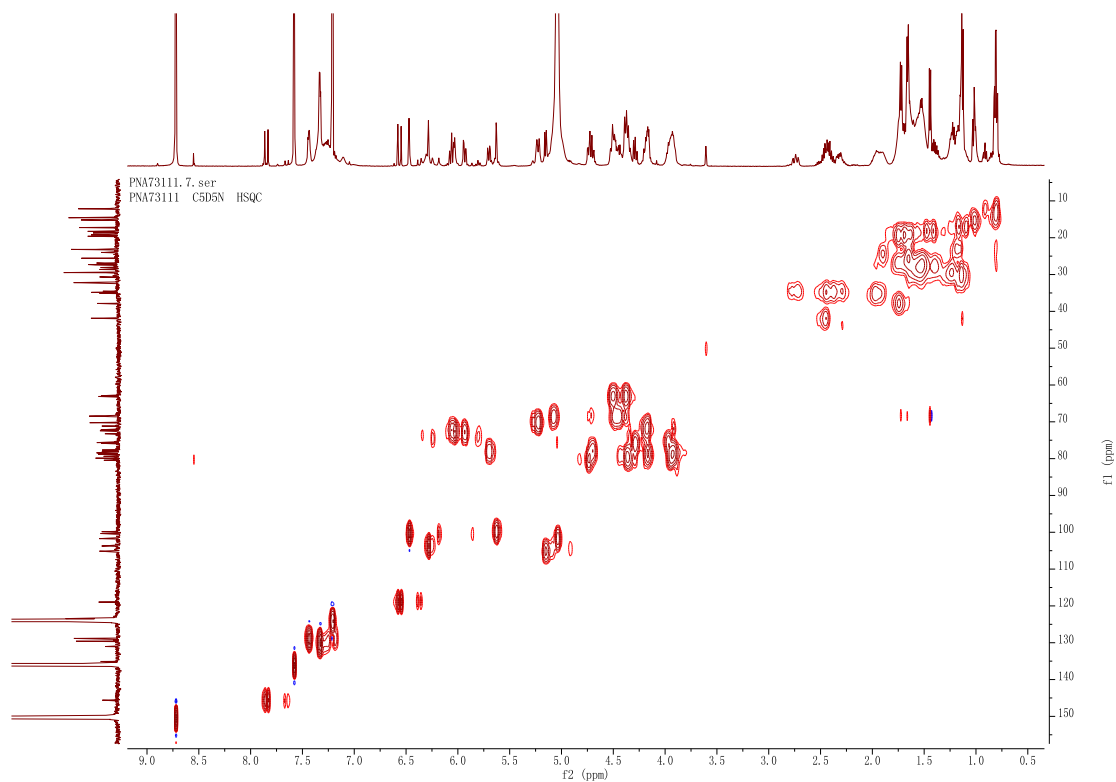


Figure S39. HSQC (500MHz, pyridine- d_5) spectrum of pharbitin F (6)

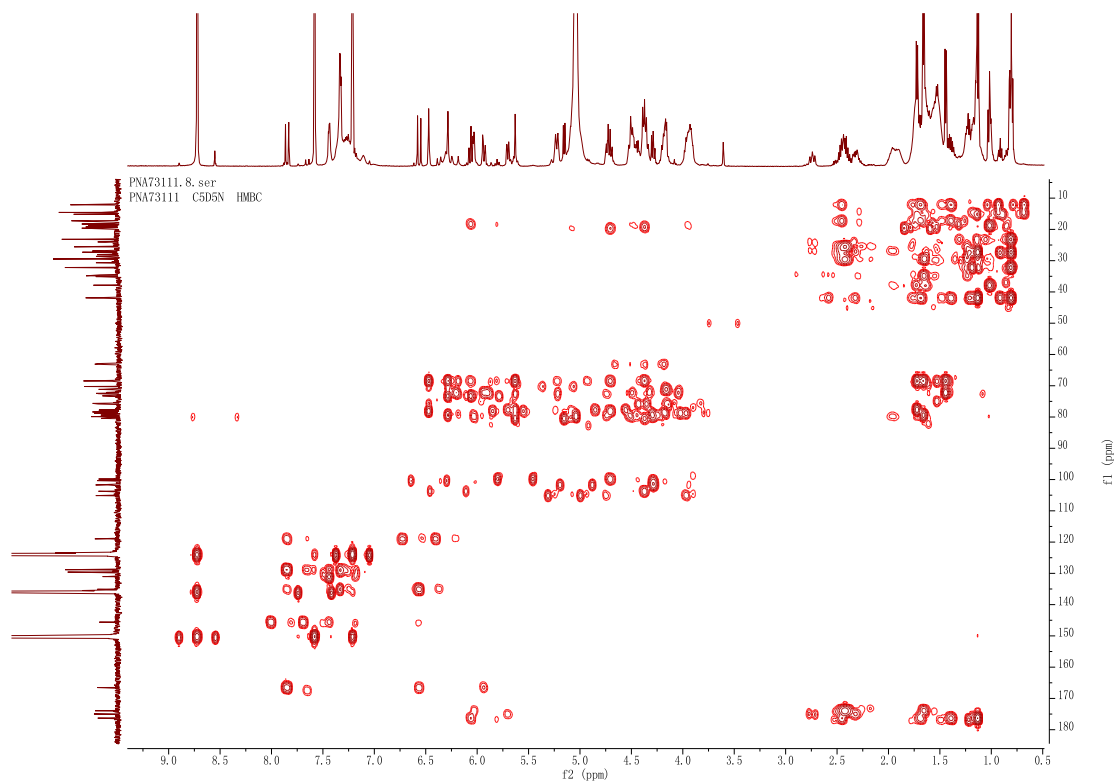
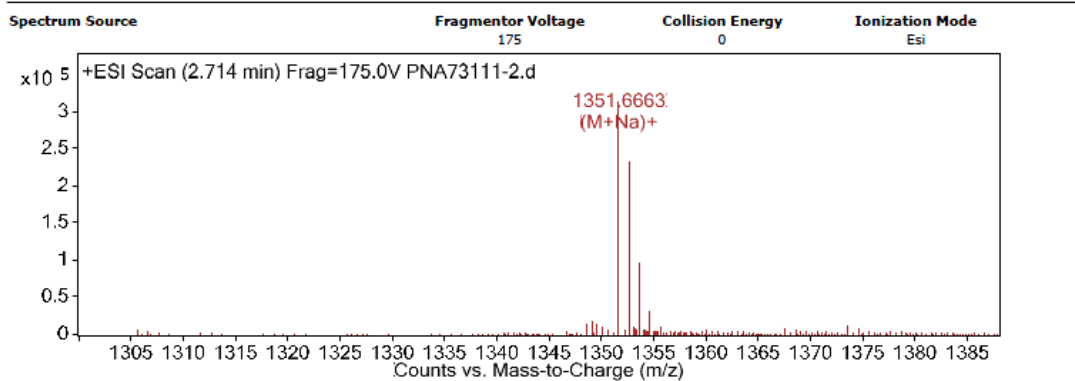


Figure S40. HMBC (500MHz, pyridine- d_5) spectrum of pharbitin F (6)

User Spectra



Peak List

m/z	z	Abund	Name	Formula	Ion	Score (DB)	Hits (DB)
1351.6663	1	312813		C66 H104 Na O27	(M+Na)+		

Formula Calculator Element Limits

Element	Min	Max
C	3	80
H	0	130
O	0	30
N	0	0
S	0	0
Cl	0	0

Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C66 H104 O27	TRUE	1328.6769	1328.6765	-0.34	C66 H104 Na O27	99.34
C73 H100 O22		1328.6769	1328.6706	-4.76	C73 H100 Na O22	80.98
C77 H100 O19		1328.6769	1328.6859	6.73	C77 H100 Na O19	68.25

Figure S41. HRESIMS spectrum of pharbitin F (6)

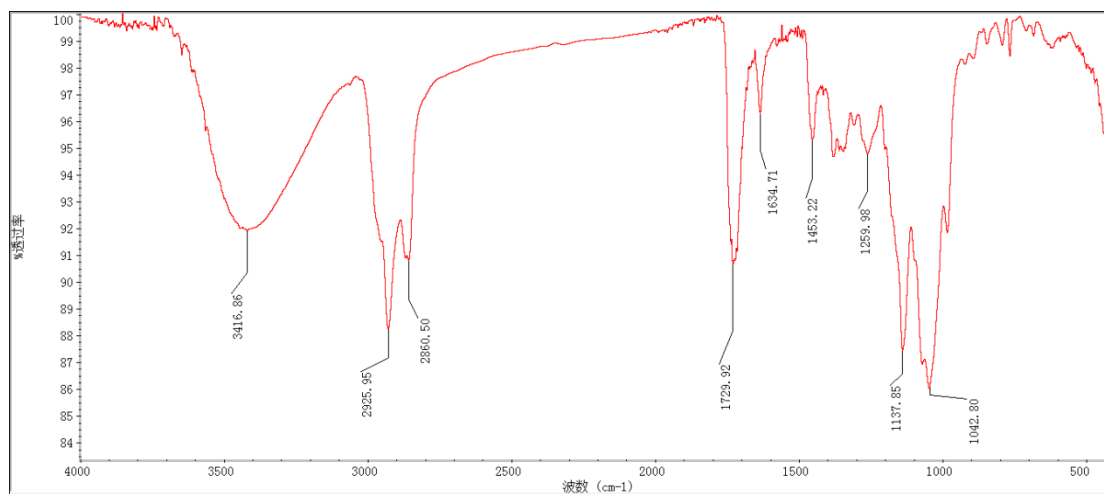


Figure S42. IR (KBr disc) spectrum of pharbitin F (6)

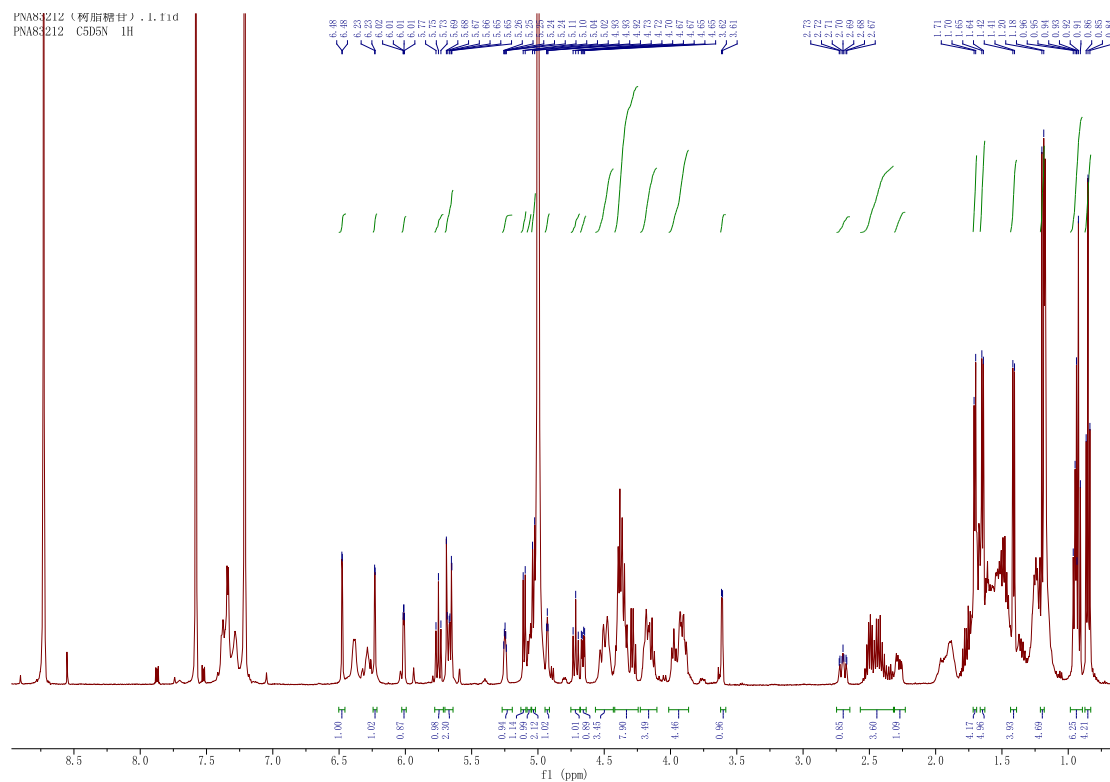


Figure S43. ^1H NMR (500MHz, pyridine- d_5) spectrum of pharbitin G (7)

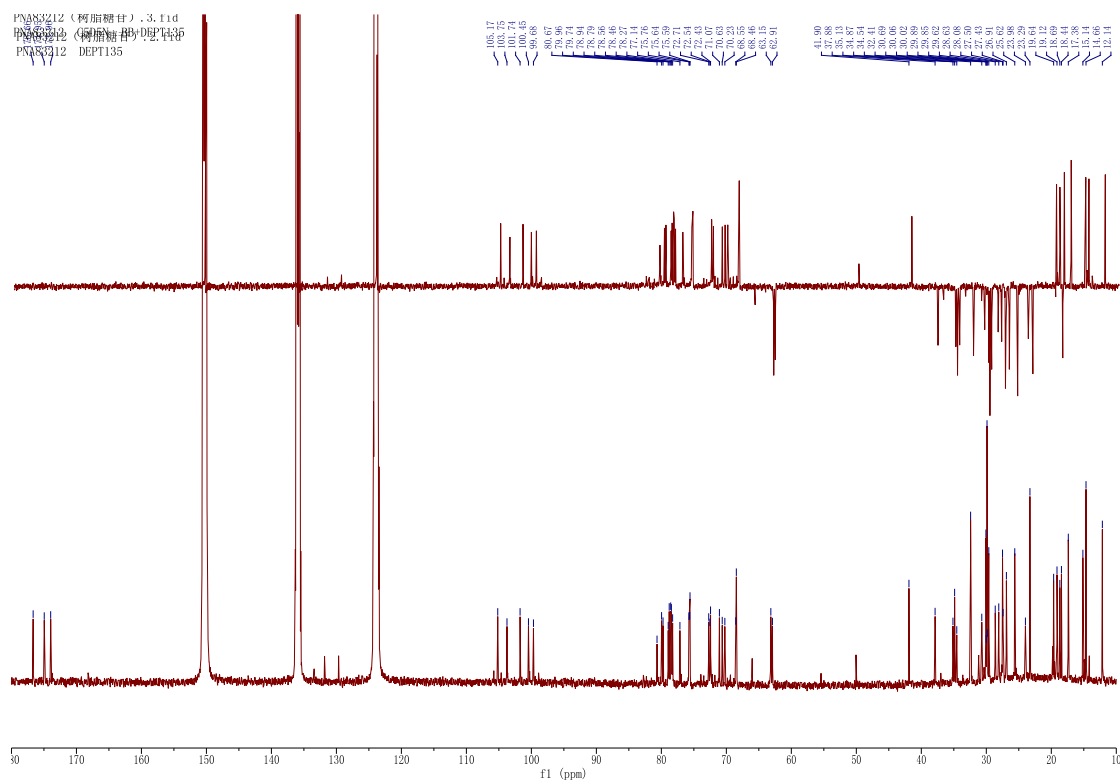


Figure S44. ^{13}C NMR (125MHz, pyridine- d_5) spectrum of pharbitin G (7)

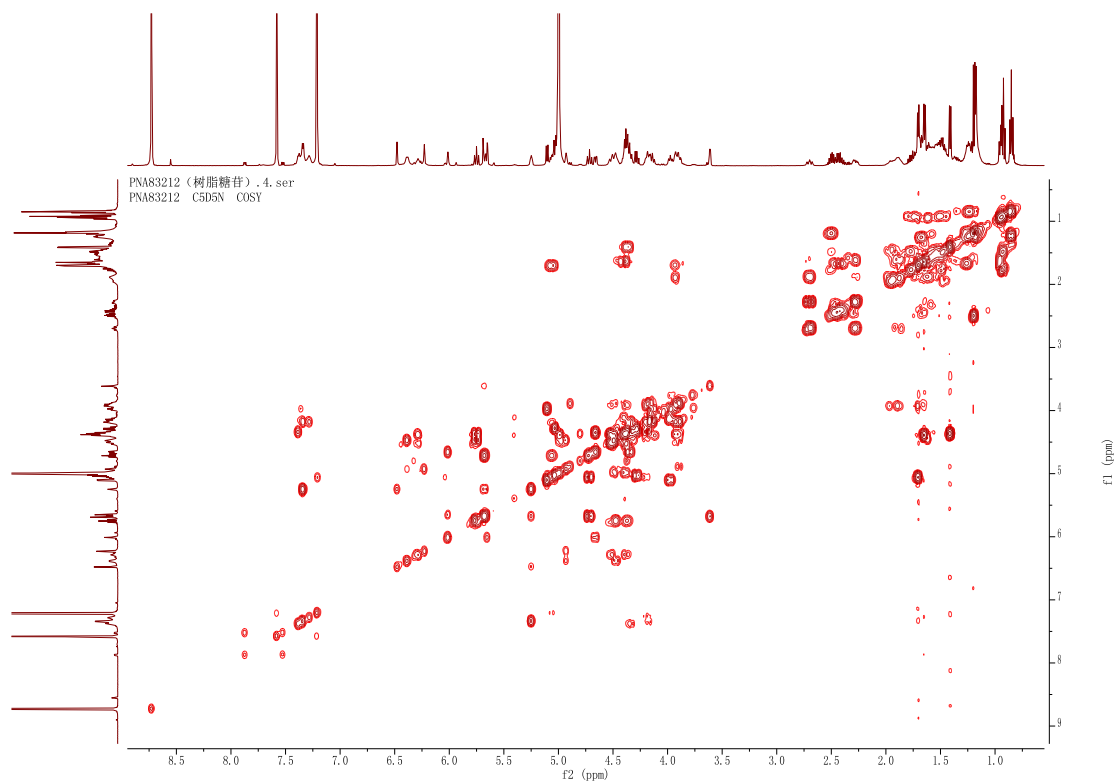


Figure S45. ^1H - ^1H COSY (500MHz, pyridine- d_5) spectrum of pharbitin G (7)

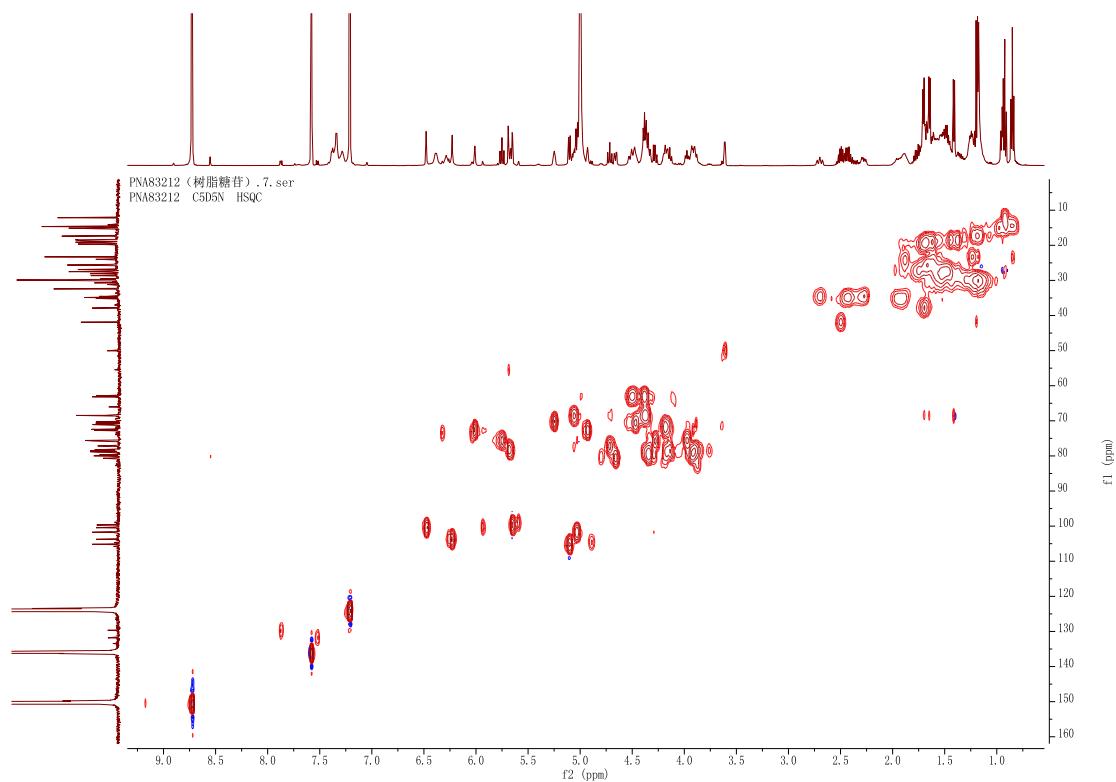


Figure S46. HSQC (500MHz, pyridine- d_5) spectrum of pharbitin G (7)

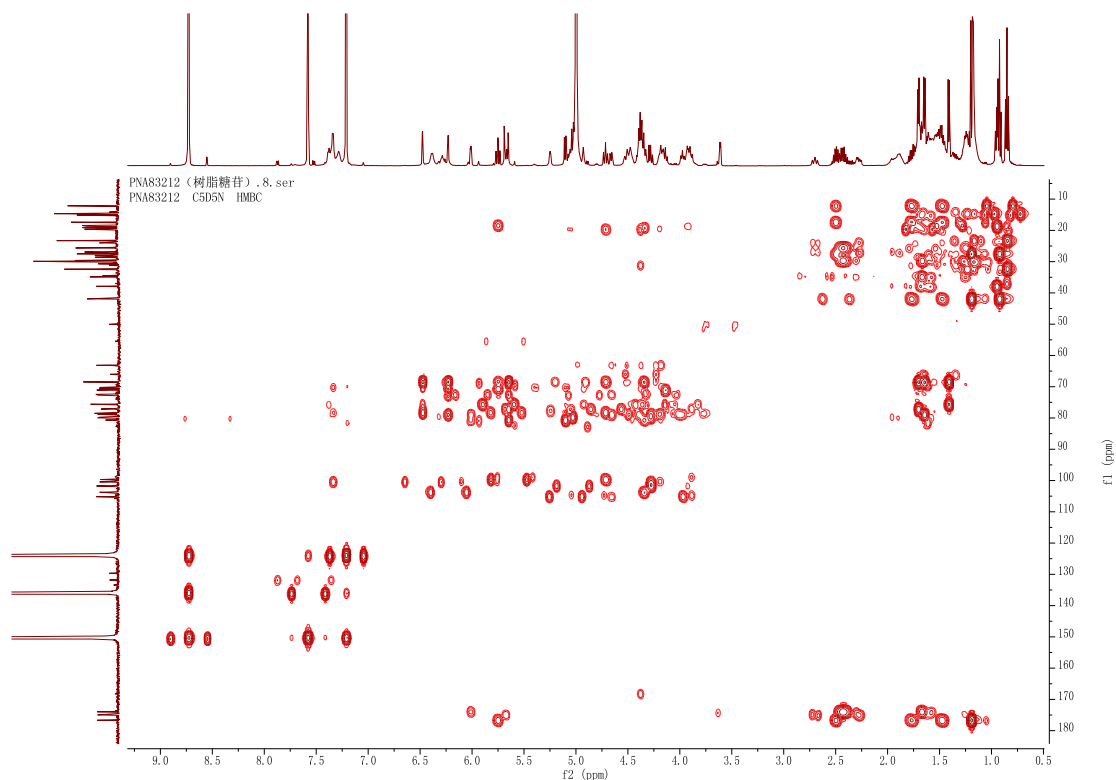
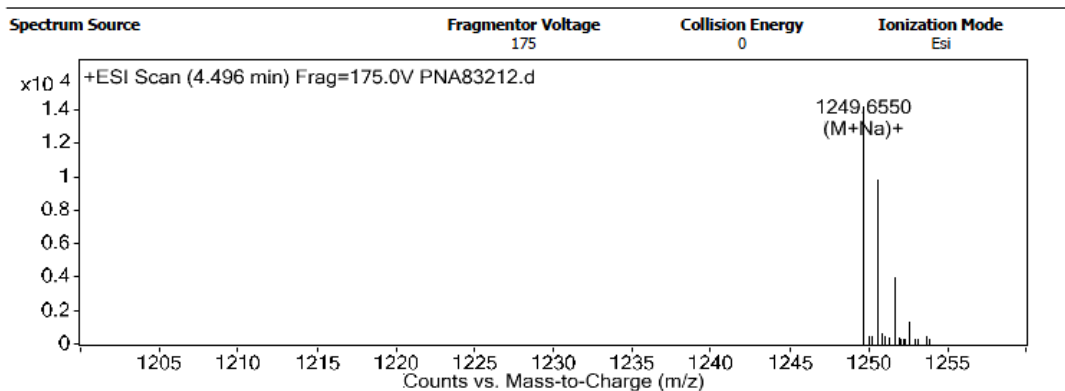


Figure S47. HMBC (500MHz, pyridine- d_5) spectrum of pharbitin G (7)

User Spectra



Element	Min	Max
C	3	80
H	0	130
O	0	30
N	0	0
S	0	0
Cl	0	0

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C59 H102 O26	TRUE	1226.6659	1226.6659	0.05	C59 H102 Na O26	99.23
C77 H94 O13		1226.6659	1226.6694	2.91	C77 H94 Na O13	86.58
C66 H98 O21		1226.6659	1226.6601	-4.74	C66 H98 Na O21	82.62

Figure S48. HRESIMS spectrum of pharbitin G (7)

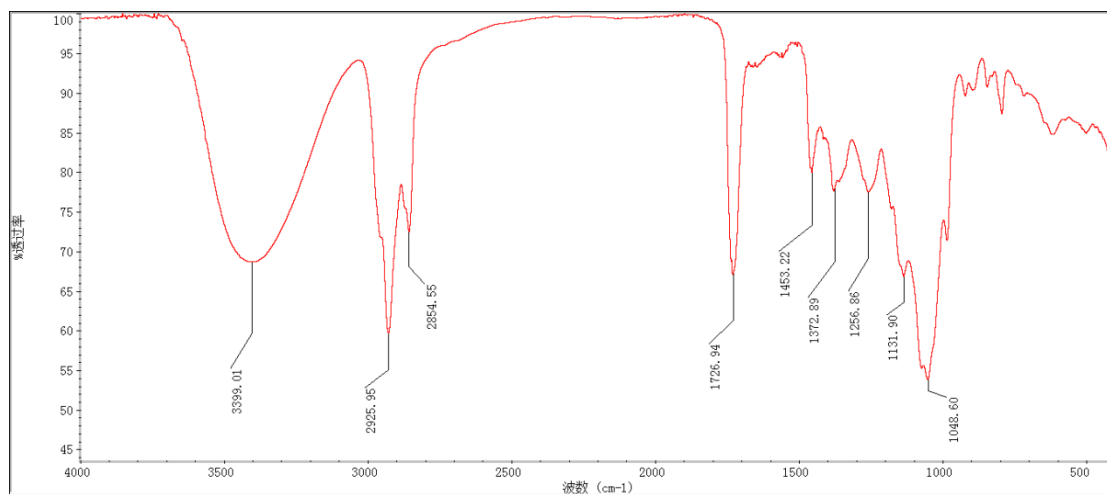


Figure S49. IR (KBr disc) spectrum of pharbitin G (7)

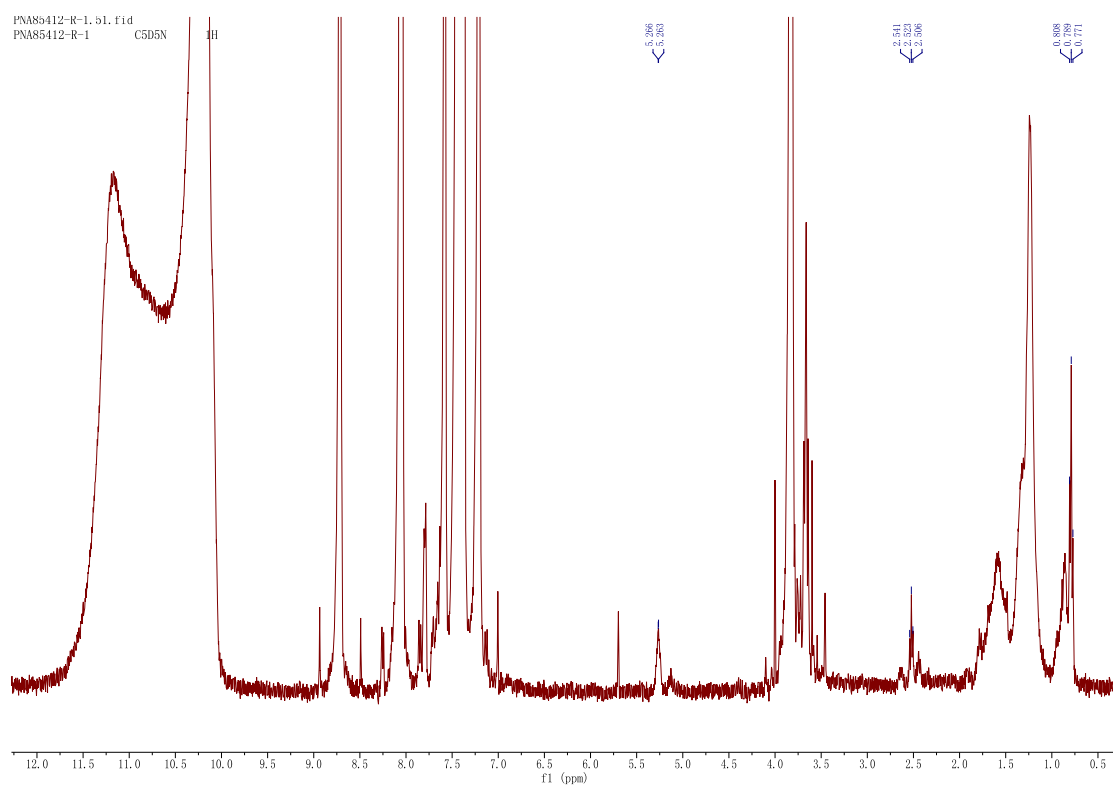


Figure S50 ^1H NMR (400MHz, pyridine- d_5) data of 11-(*S*-MPTA)-hydroxytetradecanoic acid

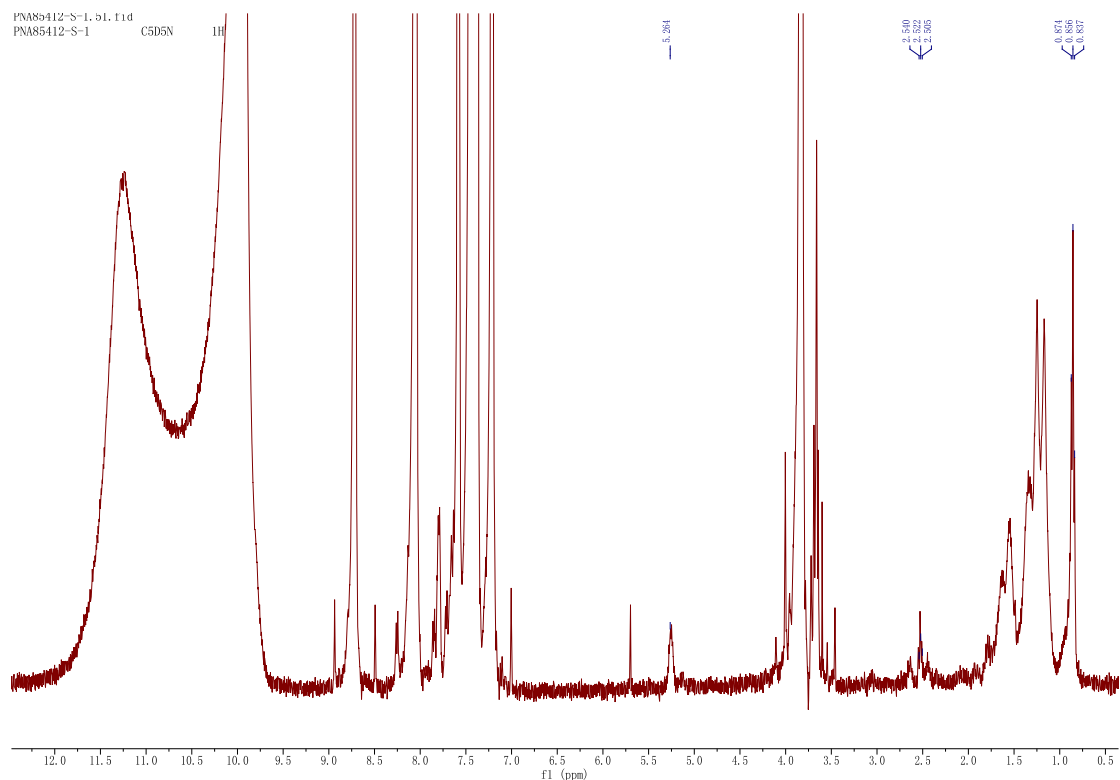
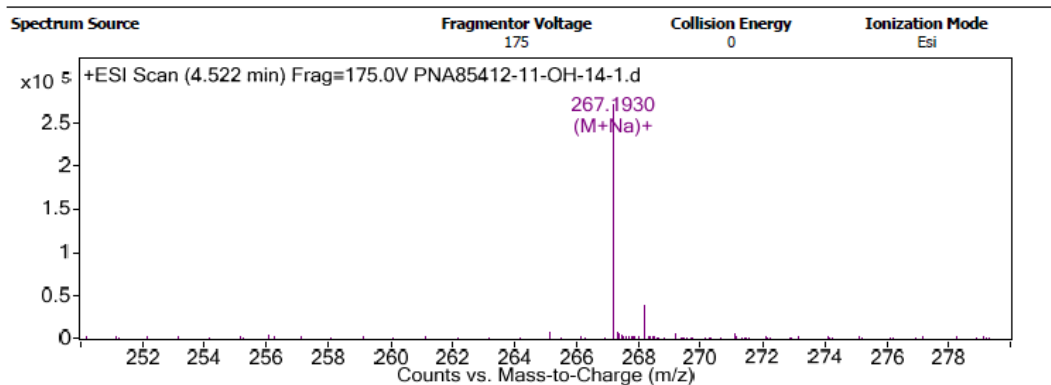


Figure S51 ^1H NMR (400MHz, pyridine- d_5) data of 11-(*R*-MPTA)-hydroxytetradecanoic acid

User Spectra



Peak List

m/z	z	Abund	Name	Formula	Ion	Score (DB)	Hits (DB)
267.193	1	269870		C14 H28 Na O3	(M+Na)+		

Formula Calculator Element Limits

Element	Min	Max
C	3	20
H	0	100
O	0	10
N	0	0
S	0	0
Cl	0	0

Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C14 H28 O3	TRUE	244.2038	244.2038	0.29	C14 H28 Na O3	98.84

Figure S52 HRESIMS of 11-hydroxytetradecanoic acid

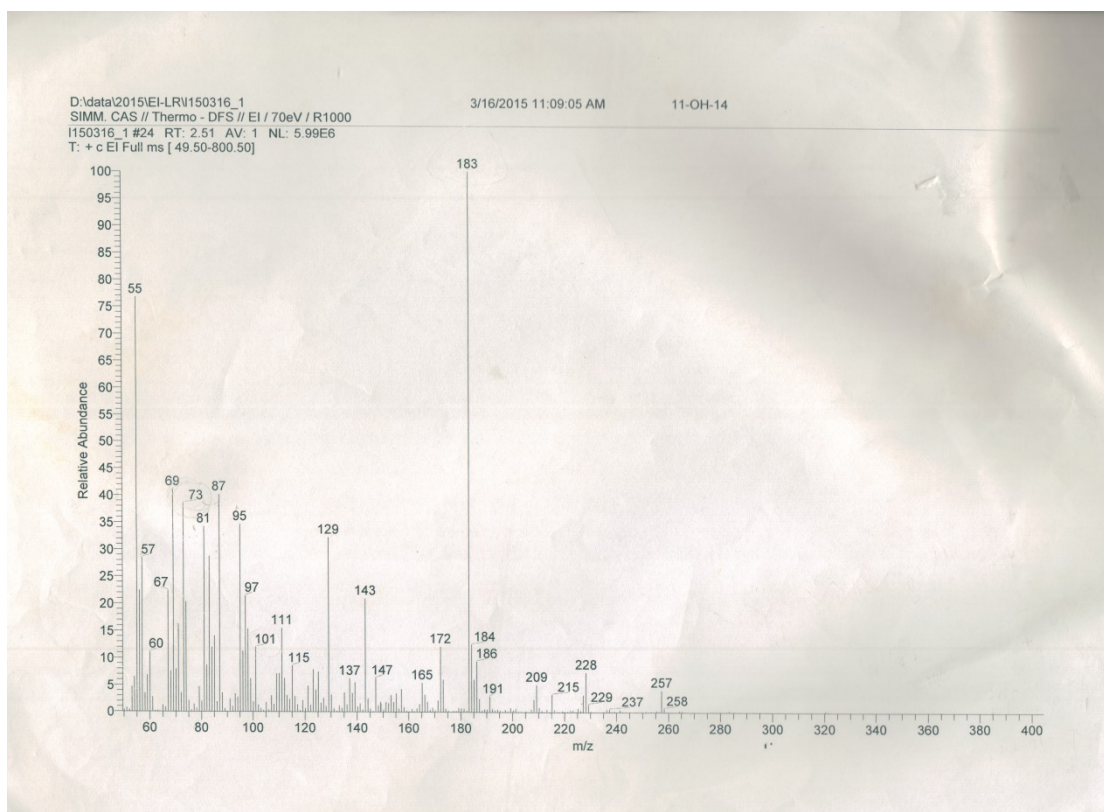
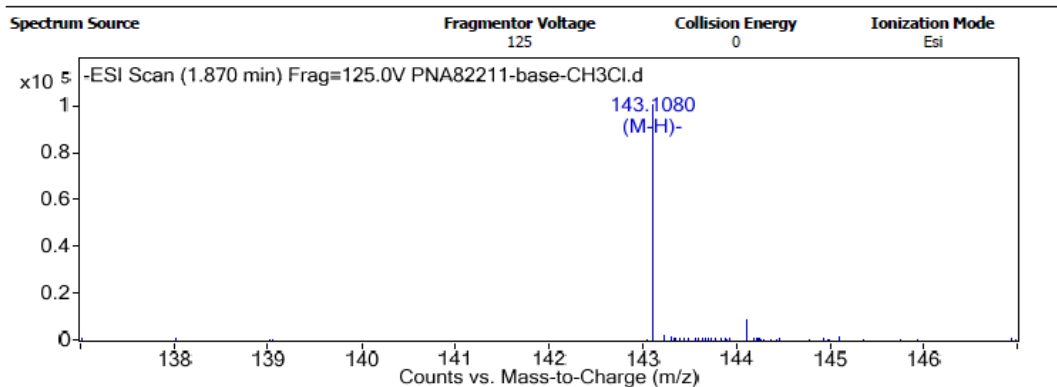


Figure S53 EIMS of 11-hydroxytetradeconoic acid

User Spectra



Peak List

m/z	z	Abund	Name	Formula	Ion	Score (DB)	Hits (DB)
243.1982	1	207610					

Formula Calculator Element Limits

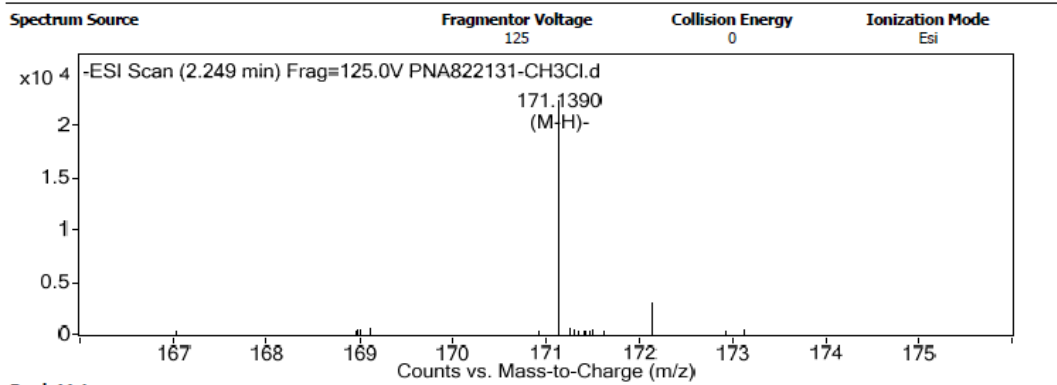
Element	Min	Max
C	3	20
H	0	100
O	0	10
N	0	0
S	0	0
Cl	0	0

Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C8 H16 O2	TRUE	144.1153	144.115	-1.6	C8 H15 O2	98.4

Figure S54 HRESIMS of *n*-octanoic acid

User Spectra



Peak List

m/z	z	Abund	Name	Formula	Ion	Score (DB)	Hits (DB)
339.2357	1	210640					

Formula Calculator Element Limits

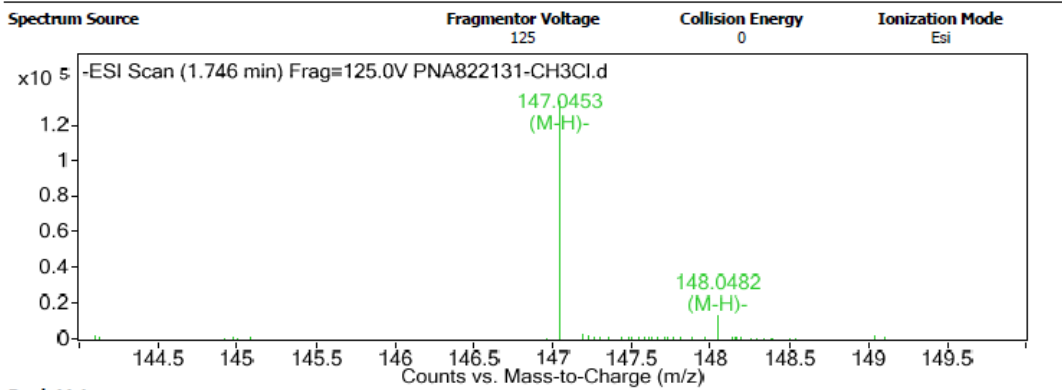
Element	Min	Max
C	3	80
H	0	130
O	0	30
N	0	0
S	0	0
Cl	0	0

Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C10 H20 O2	TRUE	172.1463	172.1463	0.13	C10 H19 O2	89.22

Figure S55 HRESIMS of *n*-decanoic acid

User Spectra



Peak List

m/z	z	Abund	Name	Formula	Ion	Score (DB)	Hits (DB)
112.9856		167451					

Formula Calculator Element Limits

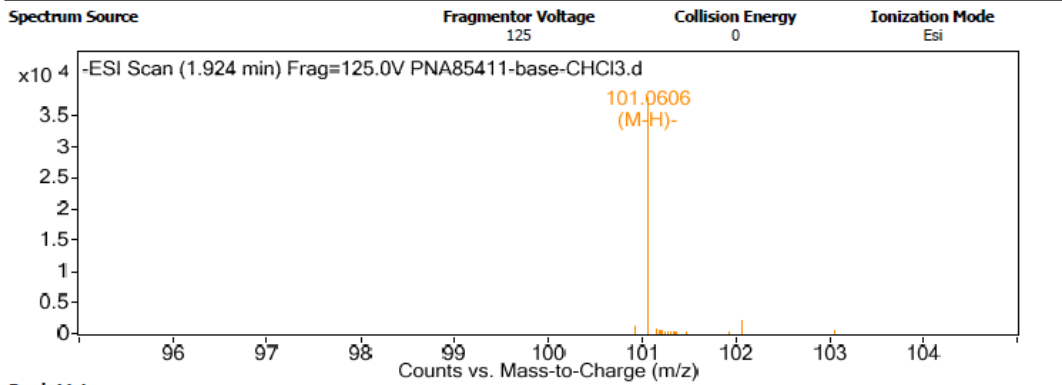
Element	Min	Max
C	3	80
H	0	130
O	0	30
N	0	0
S	0	0
Cl	0	0

Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C9 H8 O2	TRUE	148.0526	148.0524	-1.22	C9 H7 O2	98.95

Figure S56 HRESIMS of *trans*-cinnamic acid

User Spectra



Peak List

m/z	z	Abund	Name	Formula	Ion	Score (DB)	Hits (DB)
143.1096	1	491314					

Formula Calculator Element Limits

Element	Min	Max
C	3	20
H	0	100
O	0	10
N	0	0
S	0	0
Cl	0	0

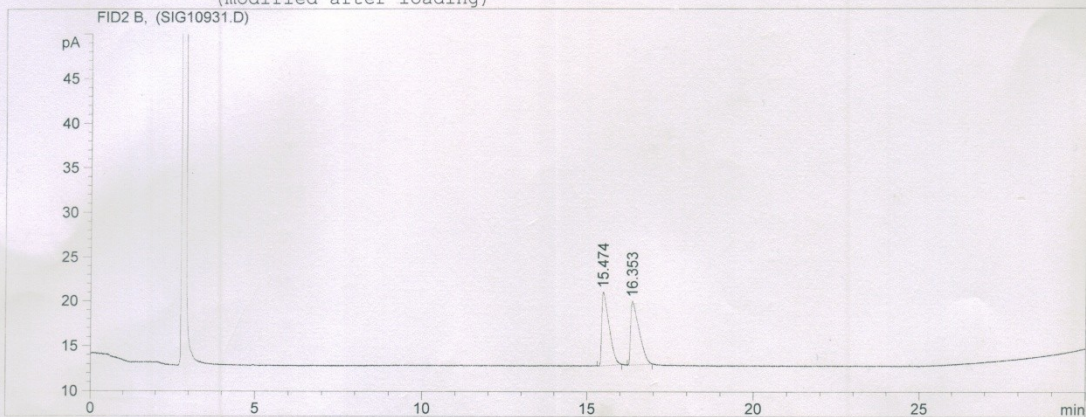
Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C5 H10 O2	TRUE	102.0679	102.0681	1.81	C5 H9 O2	97.03

Figure S57 HRESIMS of 2-methylbutanoic acid

(reemic)2-methylbutyric-acid

=====
Injection Date : 6/30/2017 6:01:00 AM
Sample Name : (reemic) Location : Vial 1
Acq. Operator : LXL Inj : 1
Inj Volume : Manually
Acq. Method : C:\HPCHEM\1\METHODS\DEF_GC1.M
Last changed : 6/30/2017 6:00:39 AM by LXL
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\DEF_GC1.M
Last changed : 6/30/2017 6:44:02 AM by LXL
(modified after loading)
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	15.474	BB	0.1928	124.69380	8.17989	50.14840
2	16.353	BB	0.2242	123.95580	7.08276	49.85160

Totals : 248.64960 15.26265

Results obtained with enhanced integrator!

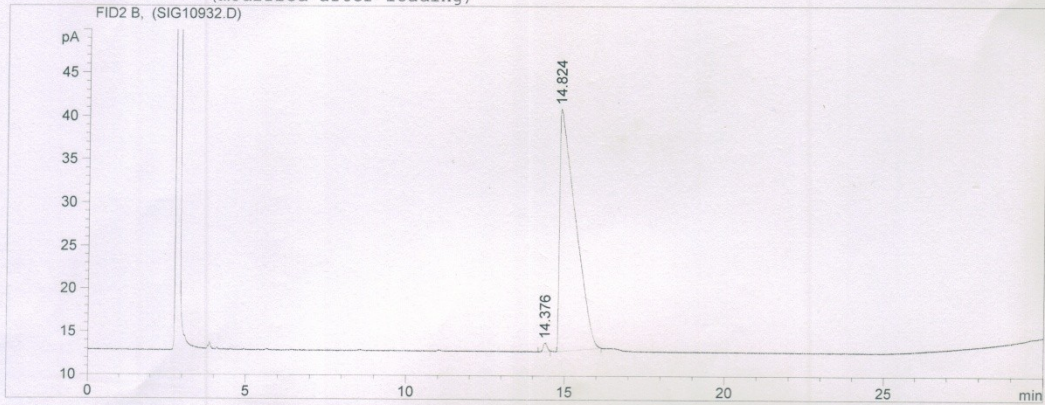
=====
*** End of Report ***
=====

Figure S58 Chiral GC analysis of (R/S)-2-methylbutanoic acid

s-2-methylbutyric-acid

```
=====
Injection Date   : 6/30/2017 6:45:23 AM
Sample Name     : s-2-methylbutyri
Acq. Operator   : LXL
Location        : Vial 1
Inj             : 1
Inj Volume      : Manually

Acq. Method     : C:\HPCHEM\1\METHODS\DEF_GCL.M
Last changed    : 6/30/2017 6:00:39 AM by LXL
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\DEF_GCL.M
Last changed    : 6/30/2017 1:07:28 PM by LXL
                  (modified after loading)
=====
```



=====
Area Percent Report
=====

```
Sorted By       : Signal
Multiplier      : 1.0000
Dilution        : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	14.376	BB	0.1052	8.92875	1.01902	0.91810
2	14.824	BB	0.4111	963.59937	28.16752	99.08190

Totals : 972.52812 29.18654

Results obtained with enhanced integrator!

=====
*** End of Report ***

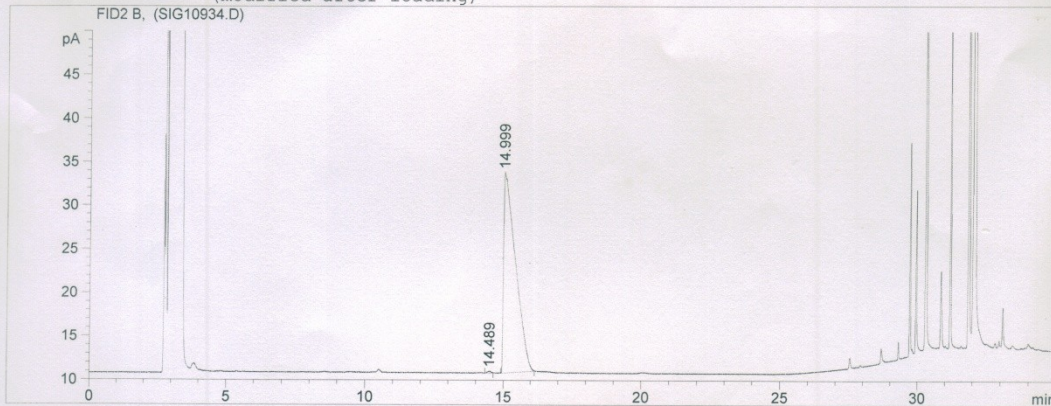
Figure S59 Chiral GC analysis of (S)-2-methylbutanoic acid


```

=====
Injection Date   : 6/30/2017 1:33:30 PM
Sample Name     : PNA85412-mba
Acq. Operator   : LXL
Location       : Vial 1
Inj           : 1
Inj Volume    : Manually

Acq. Method    : C:\HPCHEM\1\METHODS\DEF_GC1.M
Last changed   : 6/30/2017 1:33:06 PM by LXL
                (modified after loading)
Analysis Method: C:\HPCHEM\1\METHODS\DEF_GC1.M
Last changed   : 6/30/2017 2:41:01 PM by LXL
                (modified after loading)

```



```

=====
                          Area Percent Report
=====

```

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: FID2 B,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	14.489	PB	0.1111	2.03377	2.32461e-1	0.28957
2	14.999	BB	0.3678	700.30249	23.02859	99.71043

```
Totals :                702.33626  23.26105
```

Results obtained with enhanced integrator!

```

=====
*** End of Report ***

```

Figure S60 Chiral GC analysis of alkali hydrolysate of parbitin D (4)

Table S1. Results of Modulating MDR^a Activities in A549/T Cells of Compound **1-4** and **6**

Sample	Inhibition ratio % (5µM)	taxel + sample ^b	
		IC ₅₀ value (µM)	RF ^c value
3	-0.7	4.8	1.2
4	1.4	4.9	1.2
6	11.8	3.7	1.6
taxel		6.0	

Sample	Inhibition ratio % (2.5µM)	taxel + sample ^d	
		IC ₅₀ value (µM)	RF ^e value
1	33.7	2.8	2.2
2	44.0	1.6	3.7
taxel		6.0	

^aMDR: multidrug resistance.

^bSerial dilutions ranging from 0.6 to 20µM of taxel in the presence or absence of 5 µM sample.

^cRF: IC₅₀ of taxel alone/IC₅₀ of taxel in presence of 5 µM sample.

^dSerial dilutions ranging from 0.6 to 20µM of taxel in the presence or absence of 2.5 µM sample.

^eRF: IC₅₀ of taxel alone/IC₅₀ of taxel in presence of 2.5 µM sample.

Table S2. Results of Modulating MDR^a Activities in K562/ADR Cells of Compound **1-4** and **6**

Sample	Inhibition ratio % (5µM)	adriamycin + sample ^b	
		IC ₅₀ value (µM)	RF ^c value
1	-6.6	16.0	1.3
2	0.1	19.0	1.1
3	-5.2	18.4	1.1
4	-12.1	17.8	1.1
6	-7.2	18.5	1.1
adriamycin		20.1	

^aMDR: multidrug resistance.

^bSerial dilutions ranging from 3.1 to 100µM of adriamycin in the presence or absence of 5 µM sample.

^cRF: IC₅₀ of adriamycin alone/IC₅₀ of adriamycin in presence of 5 µM sample.