

Bioassay-guided Purification of Sesquiterpsnoids from the Fruiting Bodies of *Fomitopsis pinicola* and Their Bioactivity

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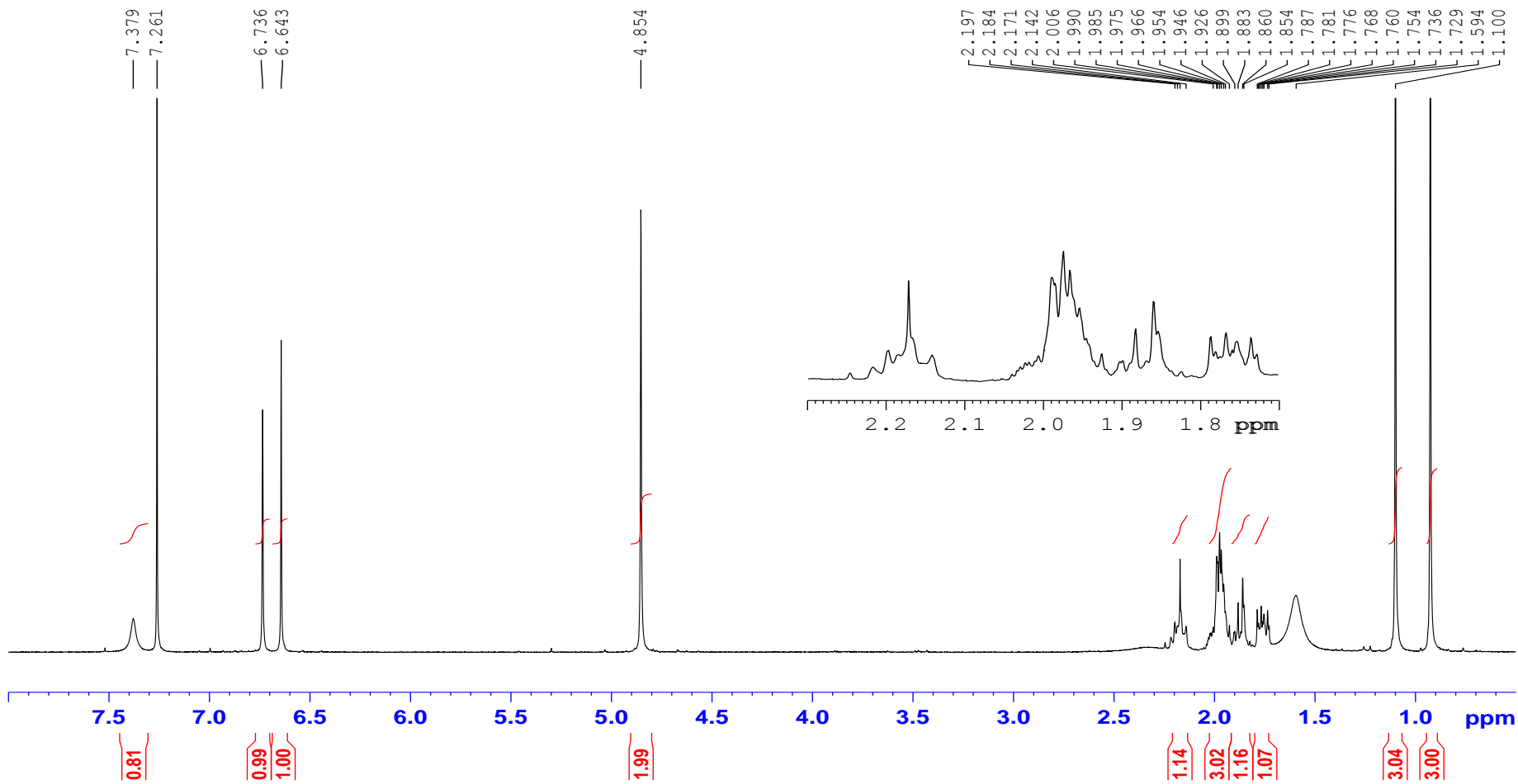
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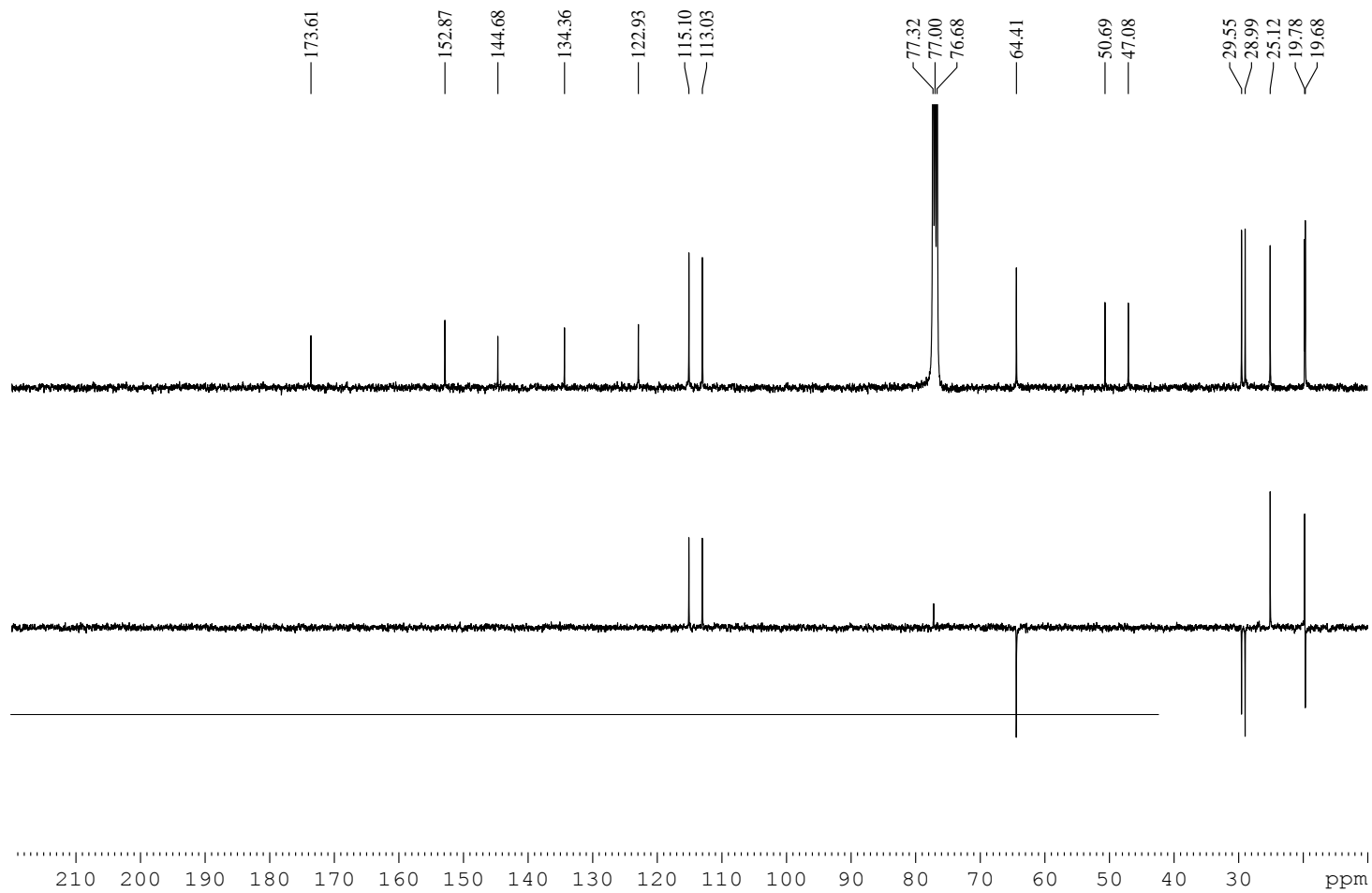
S 1 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-A (1).

FP-5-v9 CDCl₃ 102/09/13 AVVIII400 (H1)



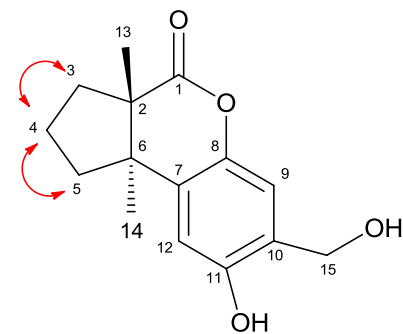
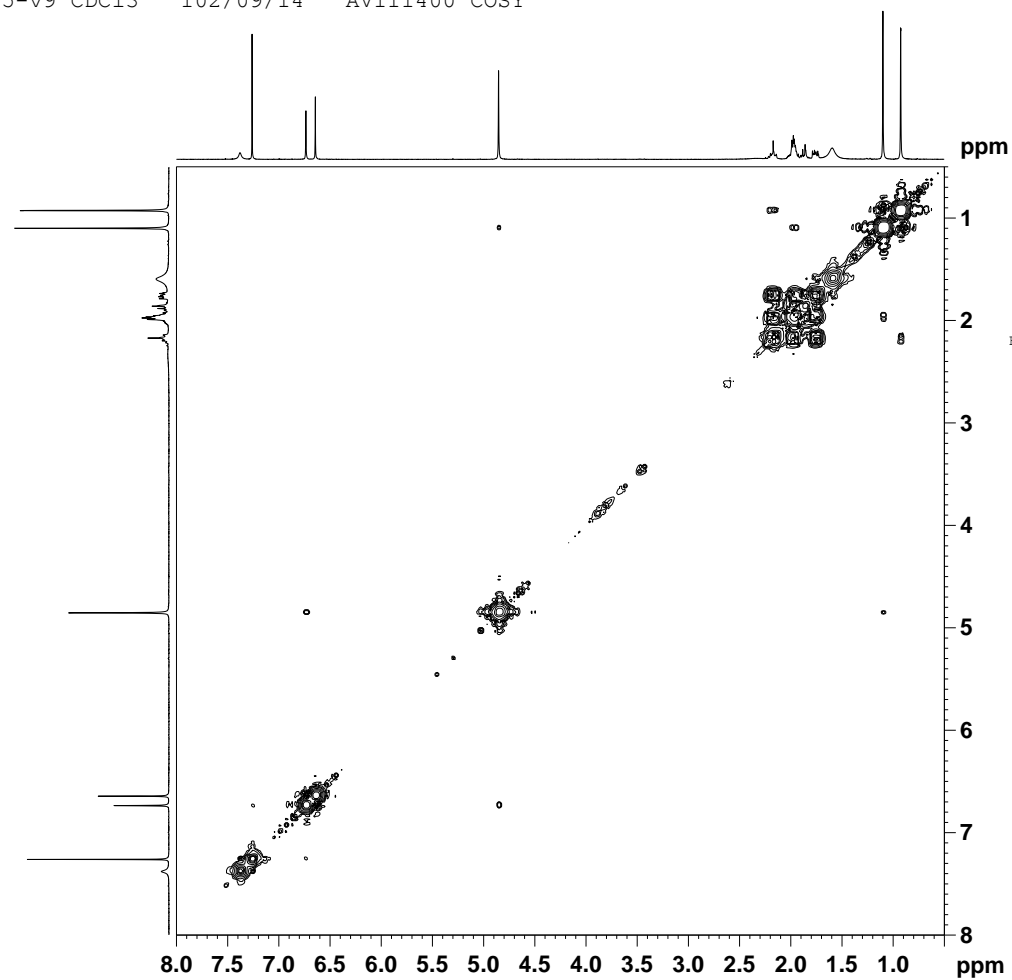
S 2 ¹³C NMR (100 MHz, CDCl₃) spectrum of fomitopin-A (1).

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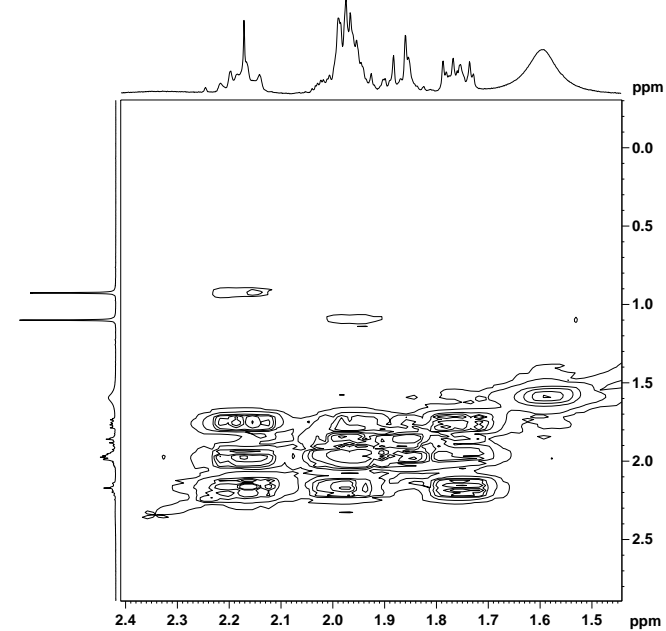


S 3 COSY spectrum of fomitopin-A (1).

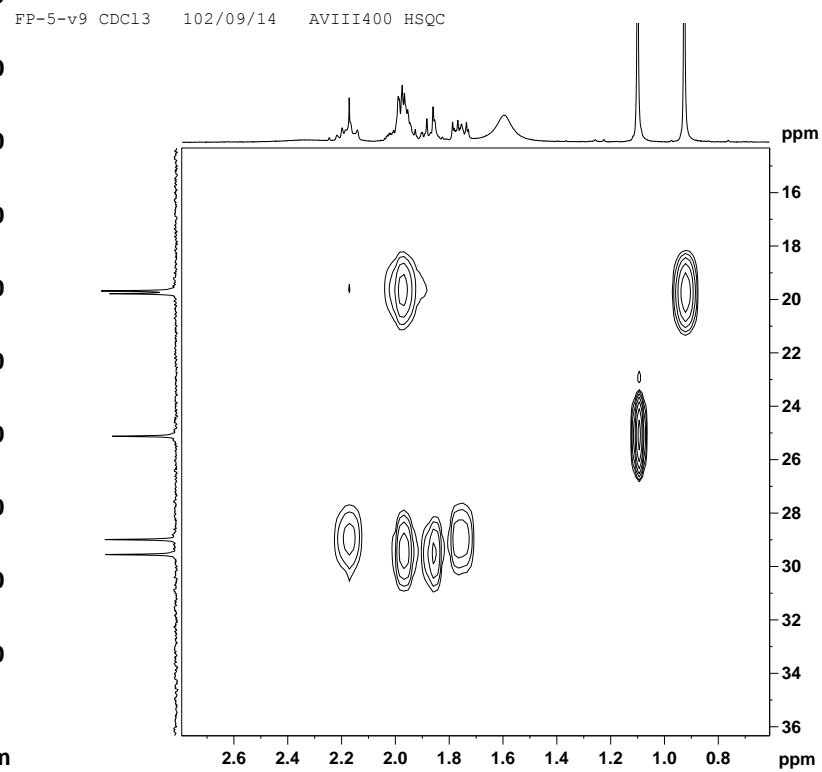
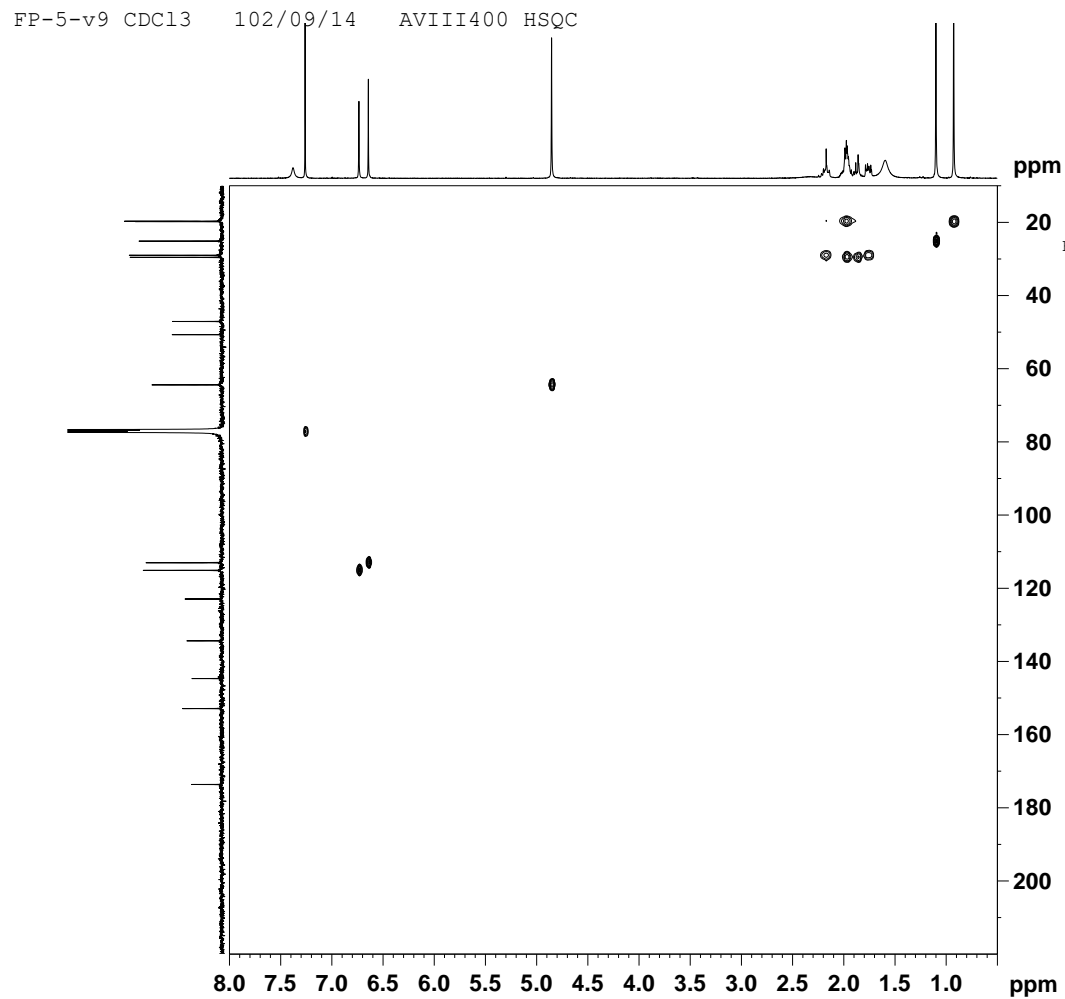
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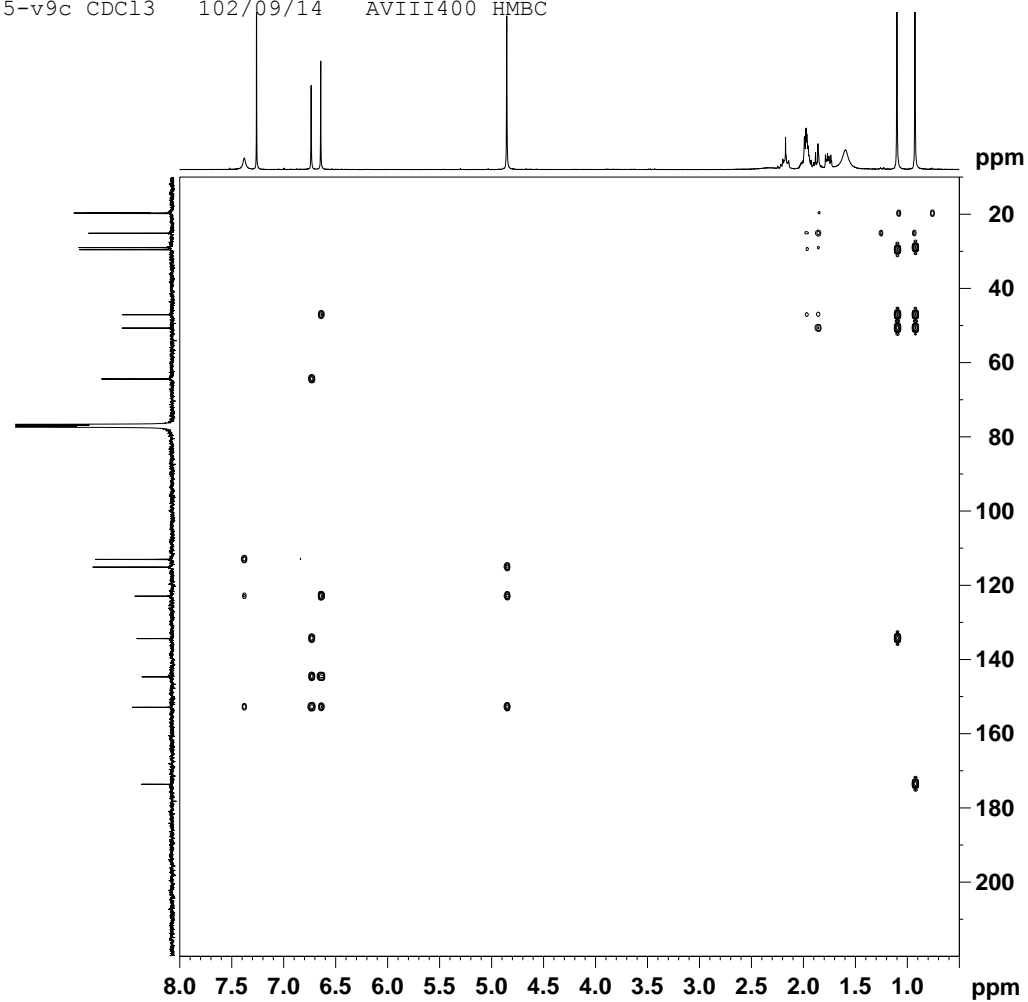


S 4 HSQC spectrum and key of HSQC spectrum of fomitopin-A (1).

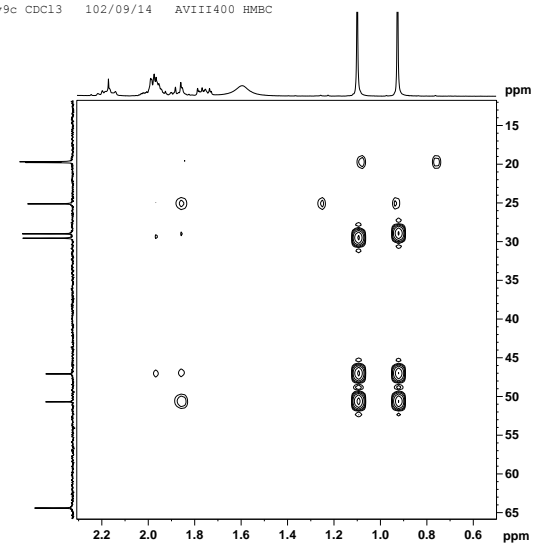


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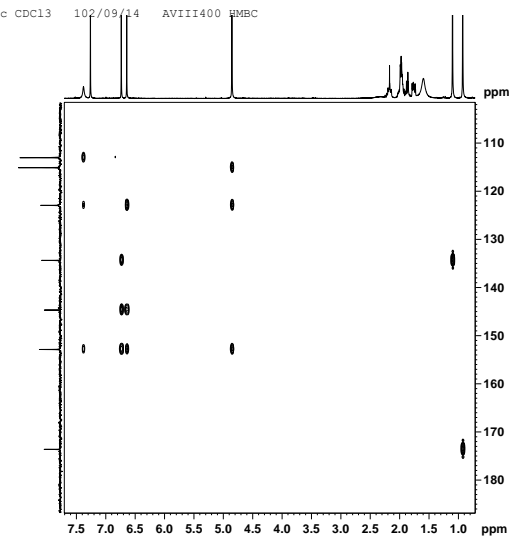
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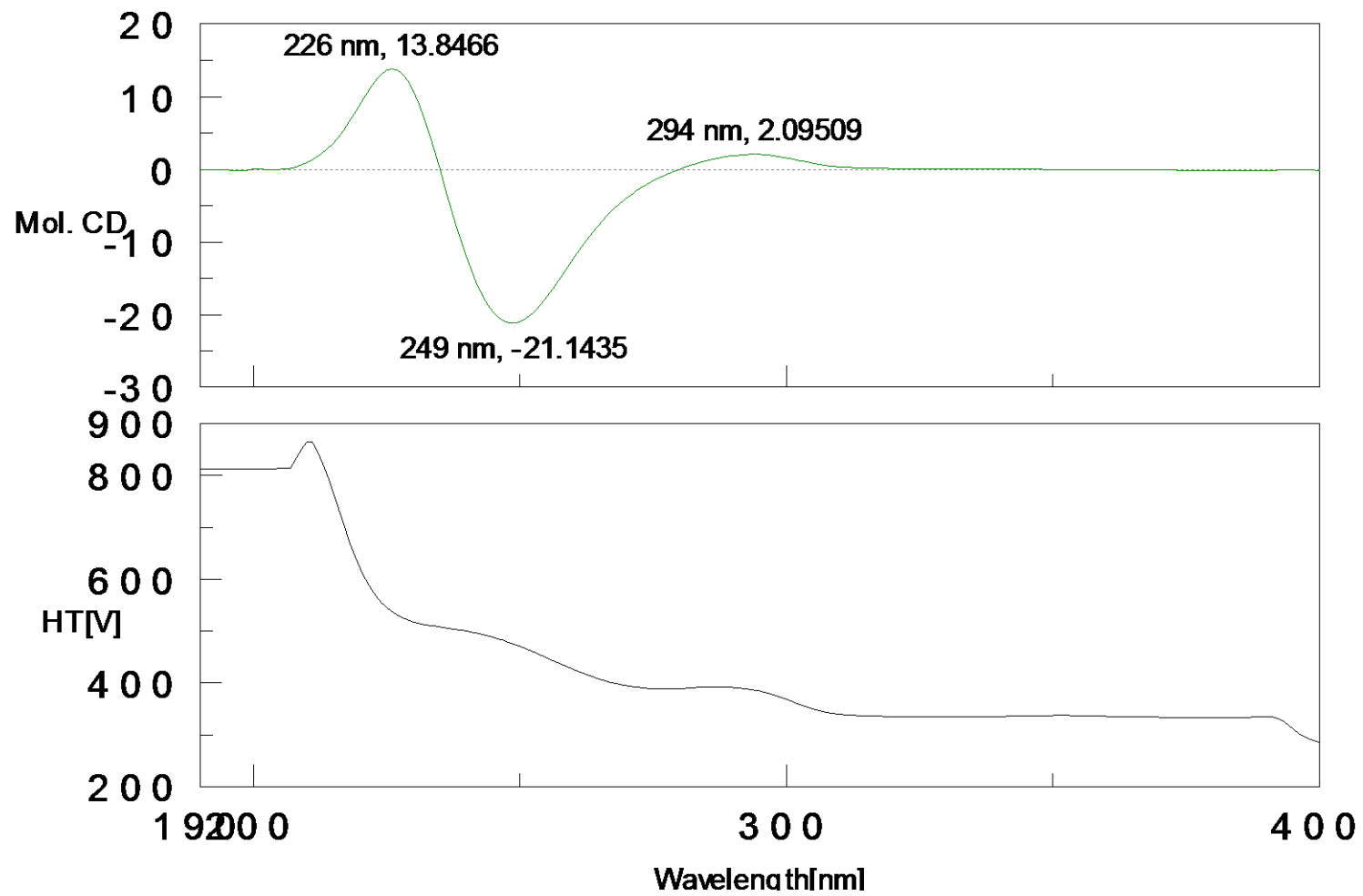
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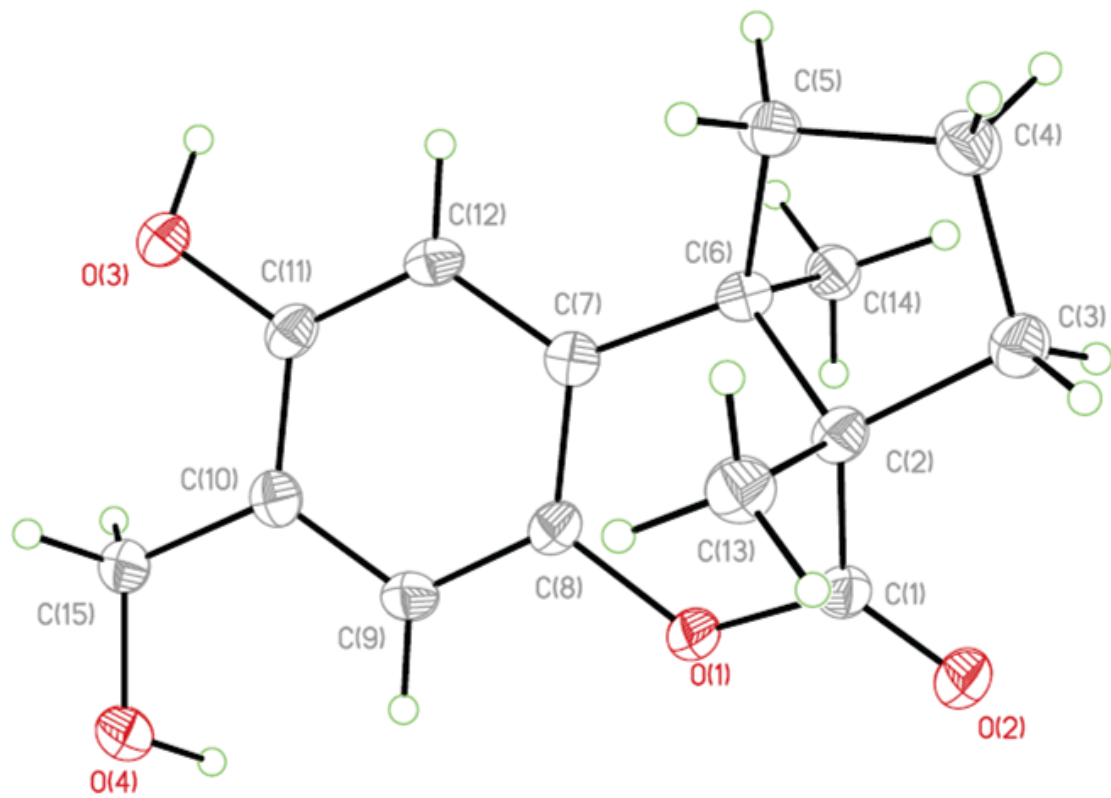
FP-5-v9c CDCl3 102/09/14 AVIII400 HMBC



S 6 ECD spectrum of fomitopin-A (1).

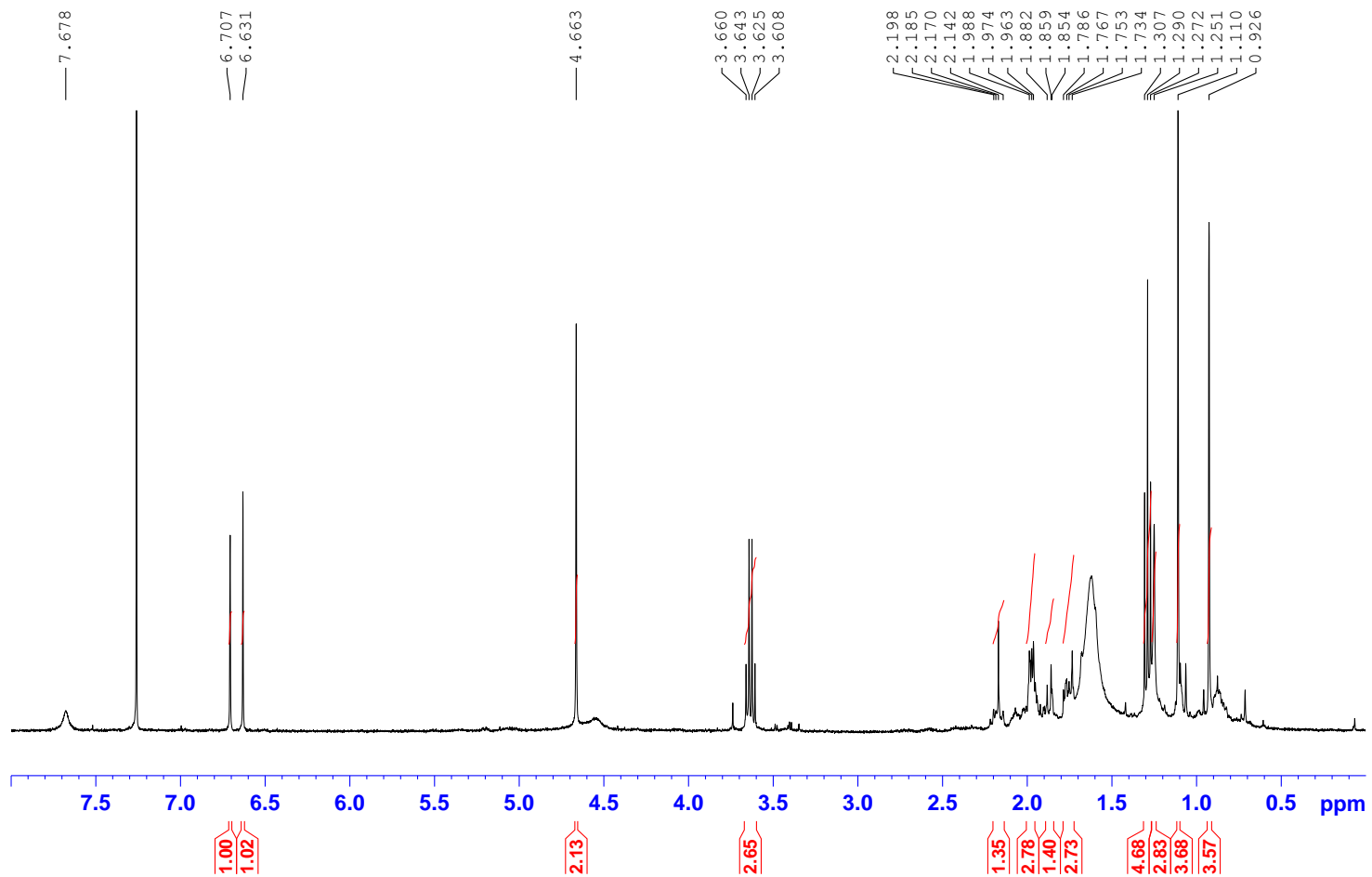


S 7 ORTEP drawing of fomitopin-A (1).



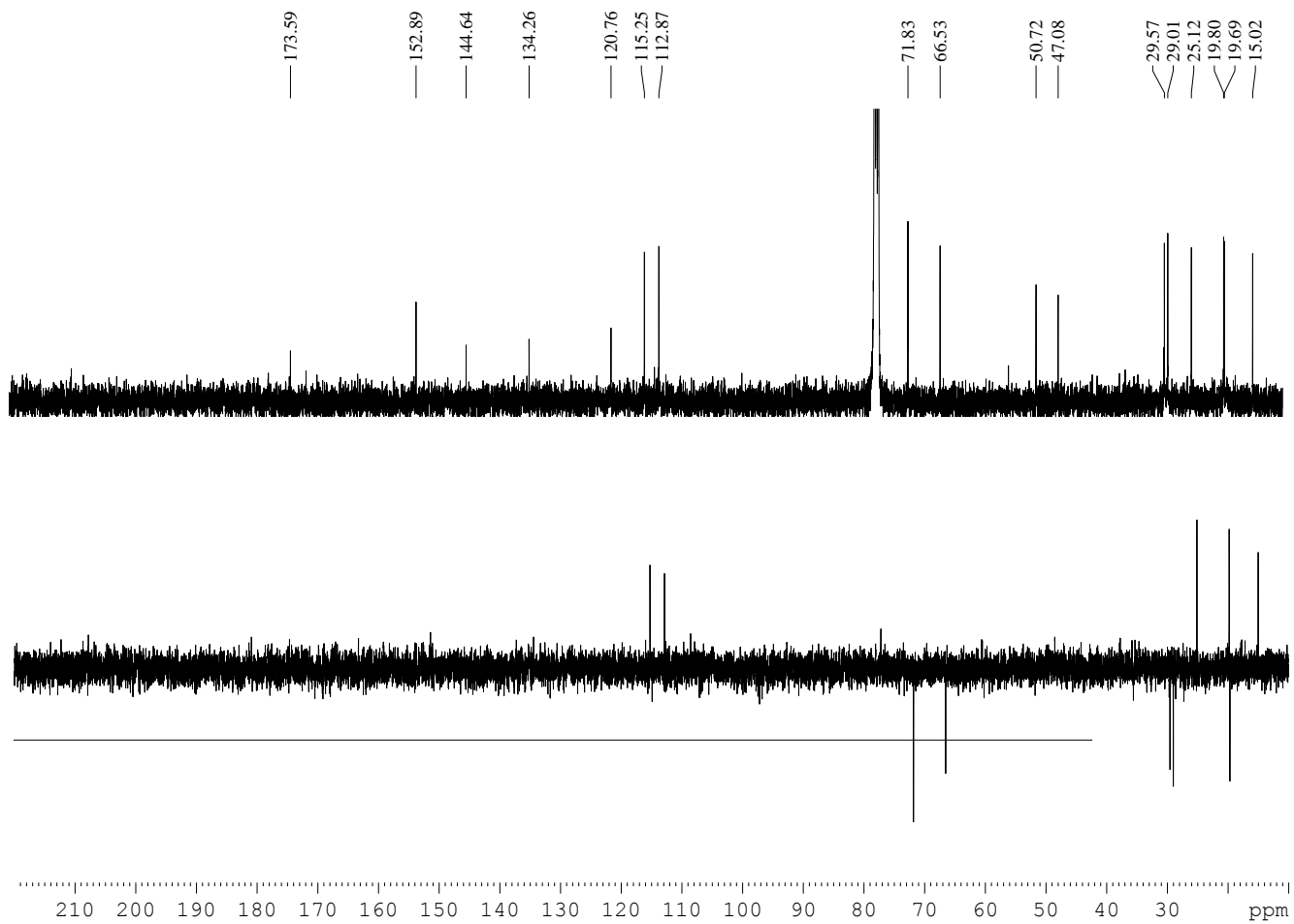
S 8 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-B (2).

FP-6252-t7 CDCl₃ 103/08/02 AVIII400 (H1)



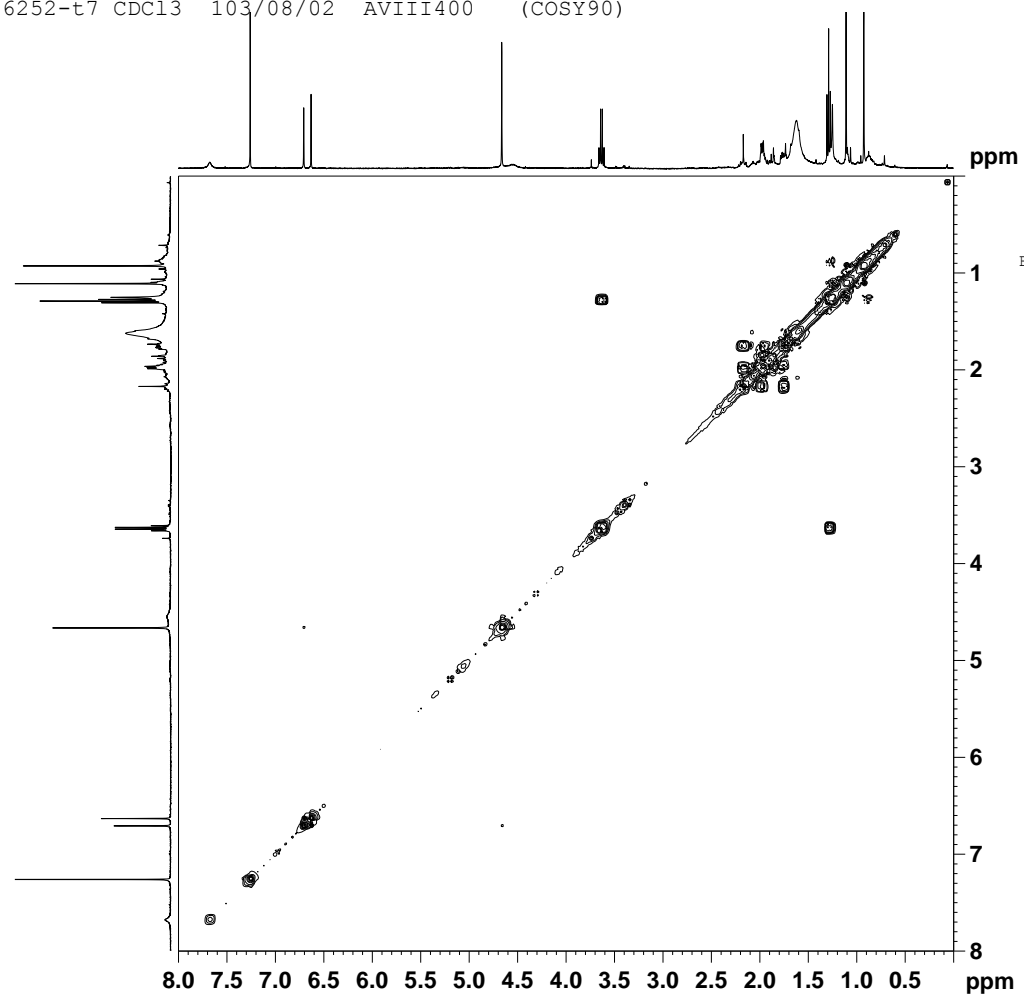
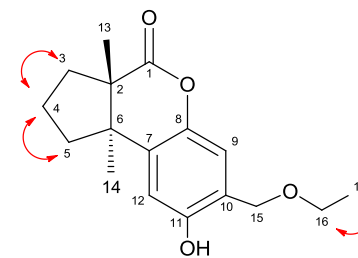
S 9 ^{13}C NMR (100 MHz, CDCl_3) spectrum of fomitopin-B (2).

FP-6252-t7 CDCl_3 103/08/02 AVIII400 (C13)

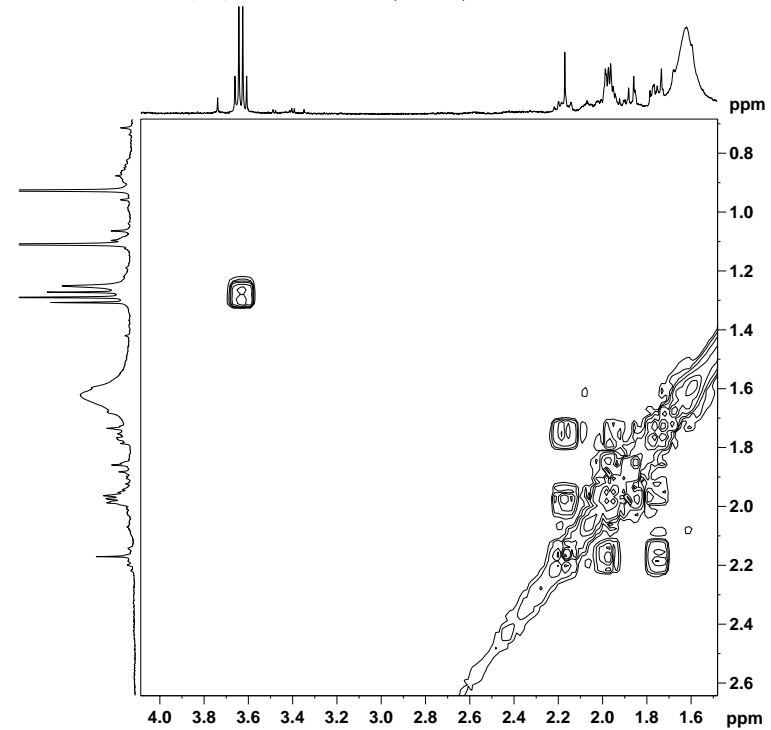


S 10 COSY spectrum of fomitopin-B (2).

FP-6252-t7 CDC13 103/08/02 AVIII400 (COSY90)

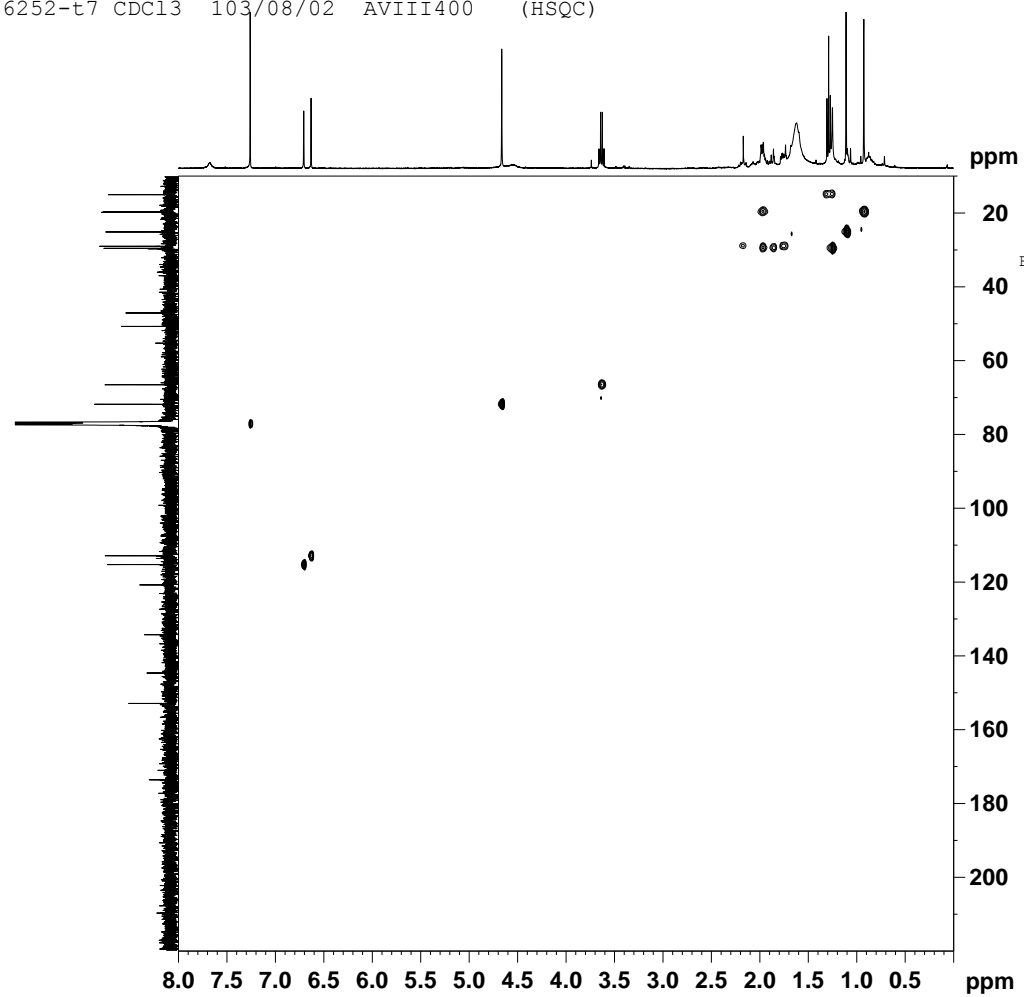


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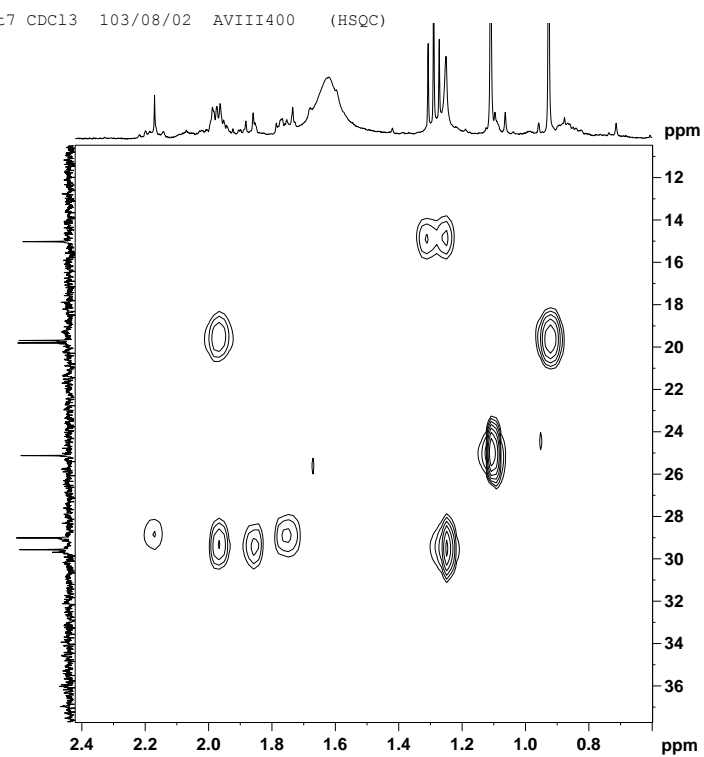


S 11 HSQC spectrum and key of HSQC spectrum of fomitopin-B (2).

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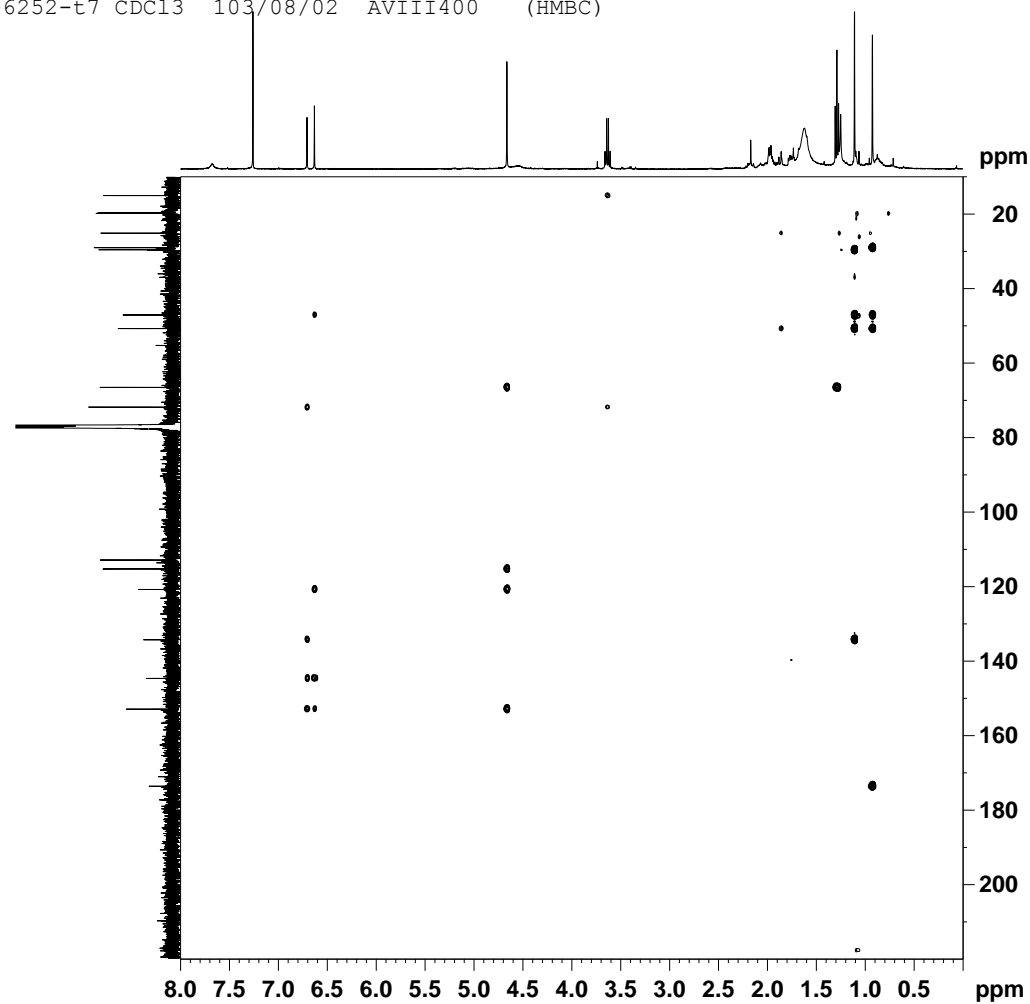


FP-6252-t7 CDC13 103/08/02 AVIII400 (HSQC)

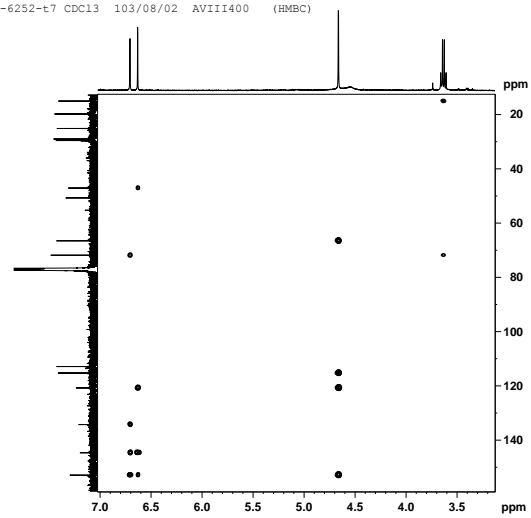


S 12 HMBC spectrum of fomitopin-B (2).

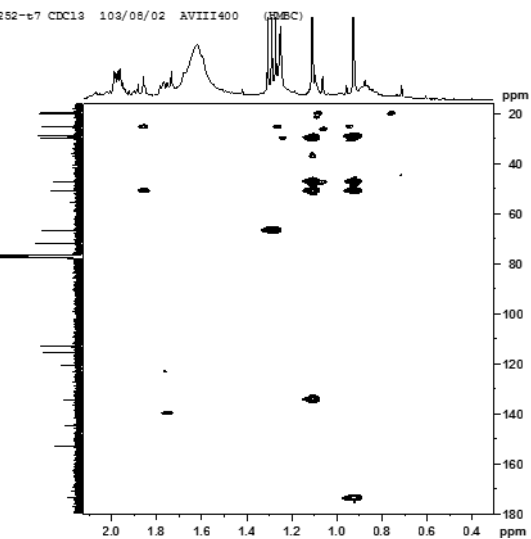
FP-6252-t7 CDCl3 103/08/02 AVIII400 (HMBC)



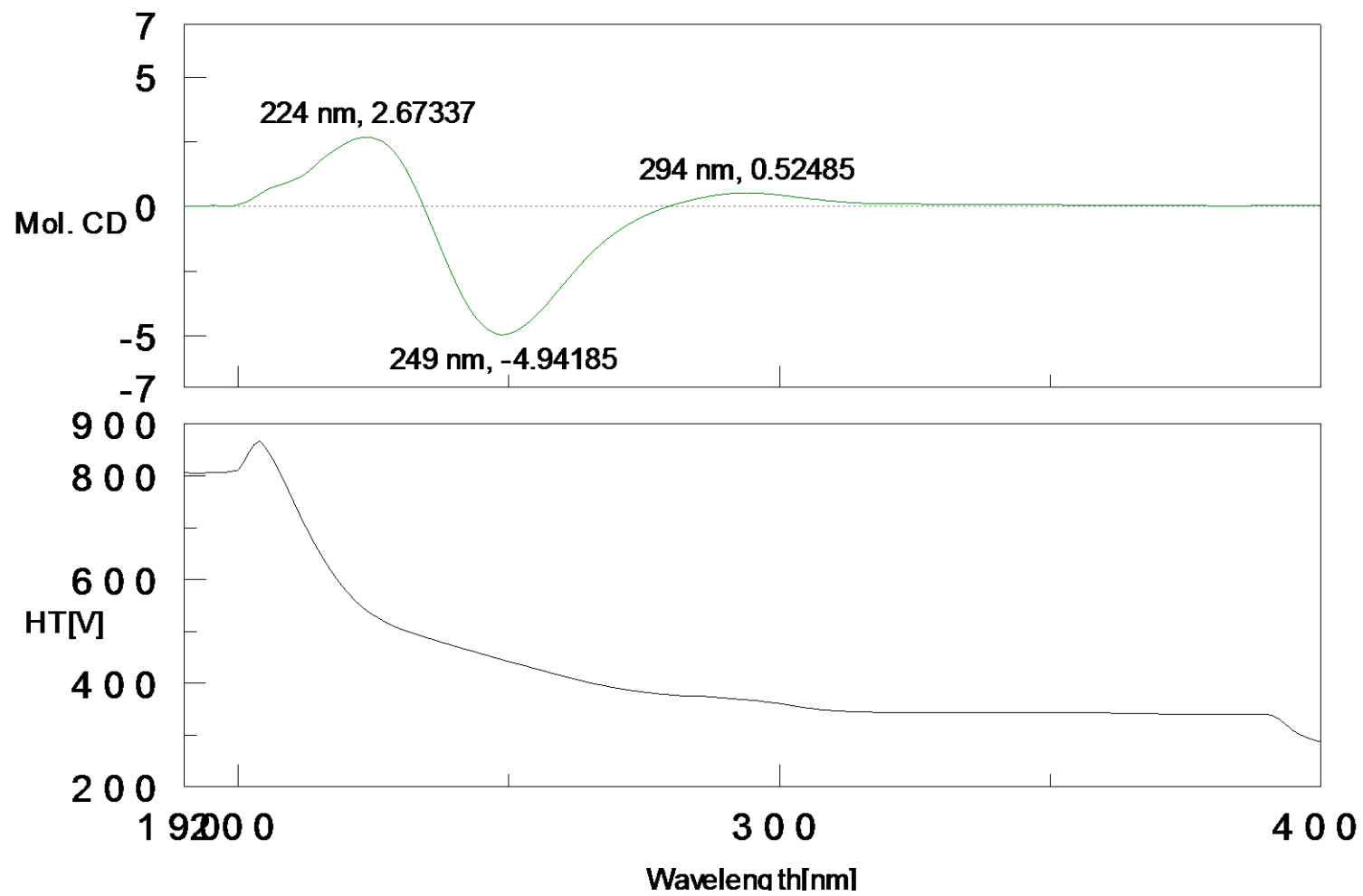
FP-6252-t7 CDCl3 103/08/02 AVIII400 (HMBC)



FP-6252-t7 CDCl3 108/08/02 AVIII400 (HMBC)

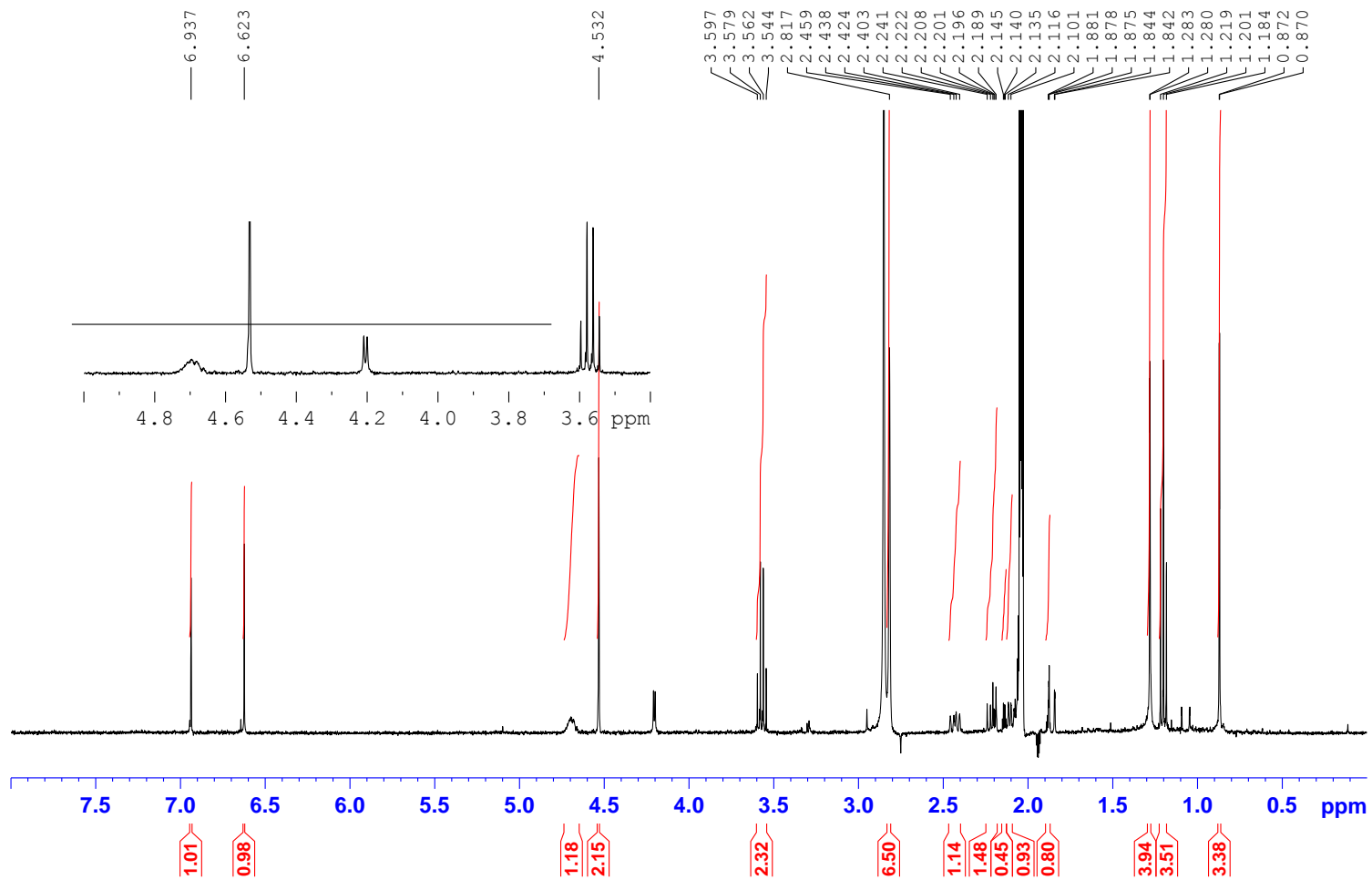


S 13 ECD spectrum of fomitopin-B (2).



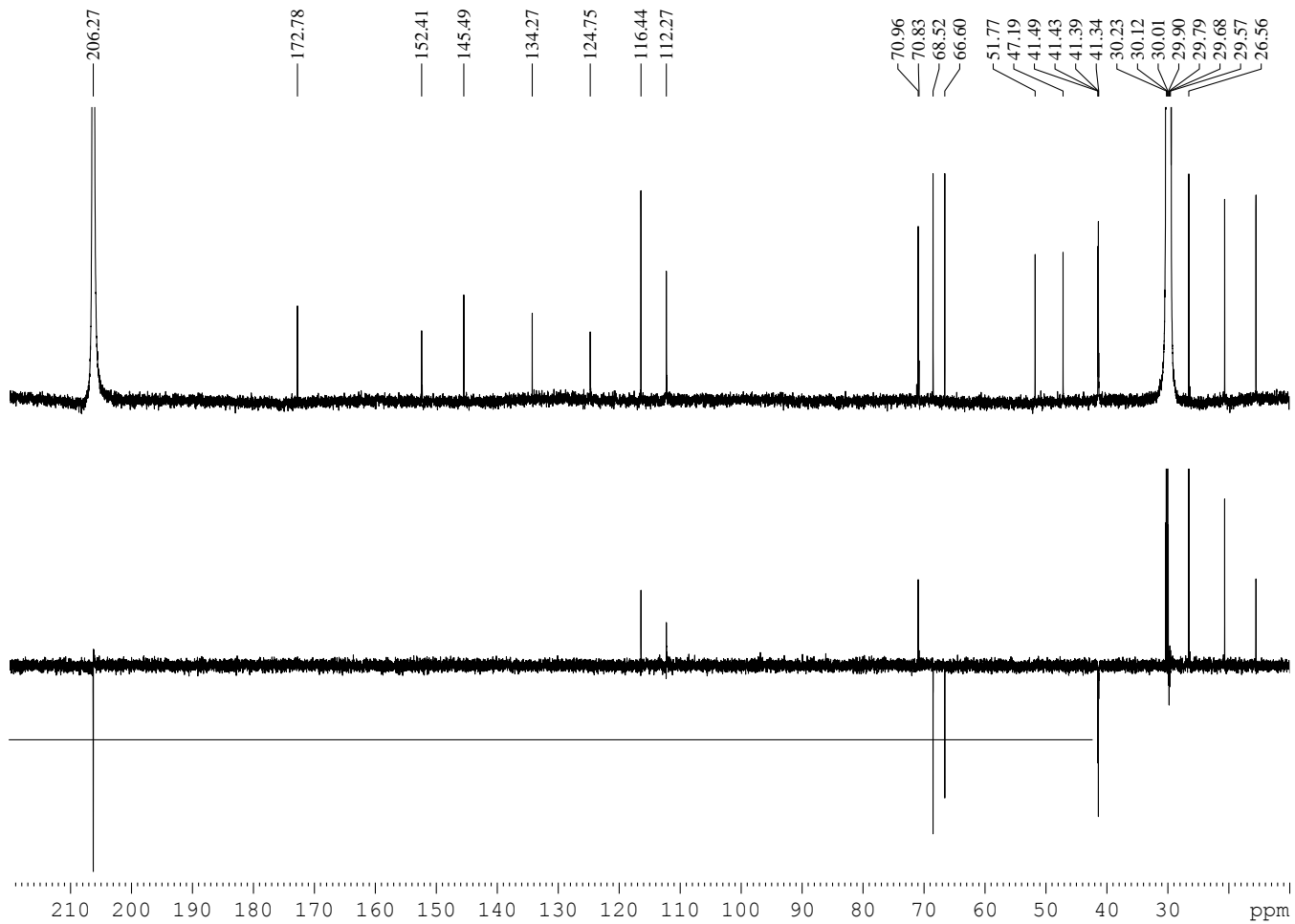
S 14 ¹H NMR (400 MHz, Acetone-d₆) spectrum of fomitopin-C (3).

FP-5-7-5C (HPLC) Acetone-d₆ 103/1/28 AVIII400 (H1)



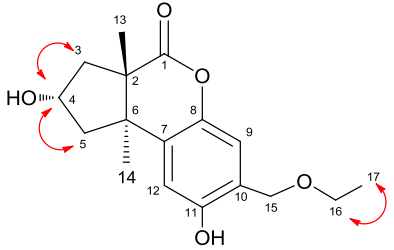
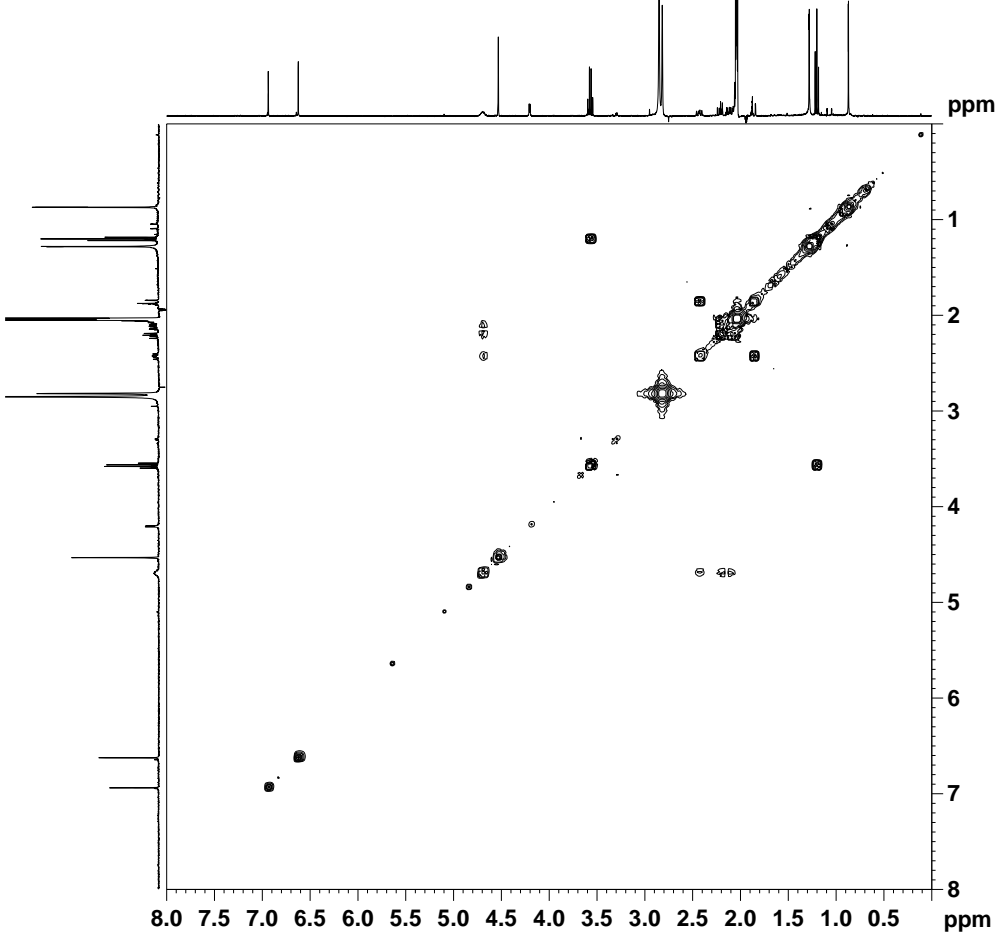
S 15 ¹³C NMR (100 MHz, Acetone-d₆) spectrum of fomitopin- C (3).

FP5-7-5C (HPLC) Acetone-d₆ 2014/01/28 AV700 C13



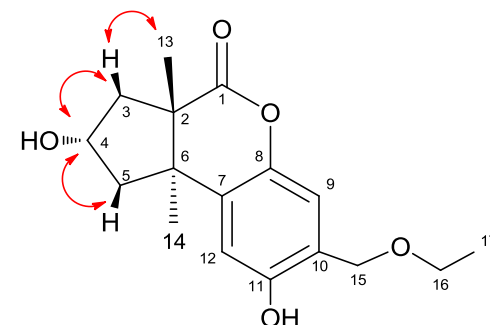
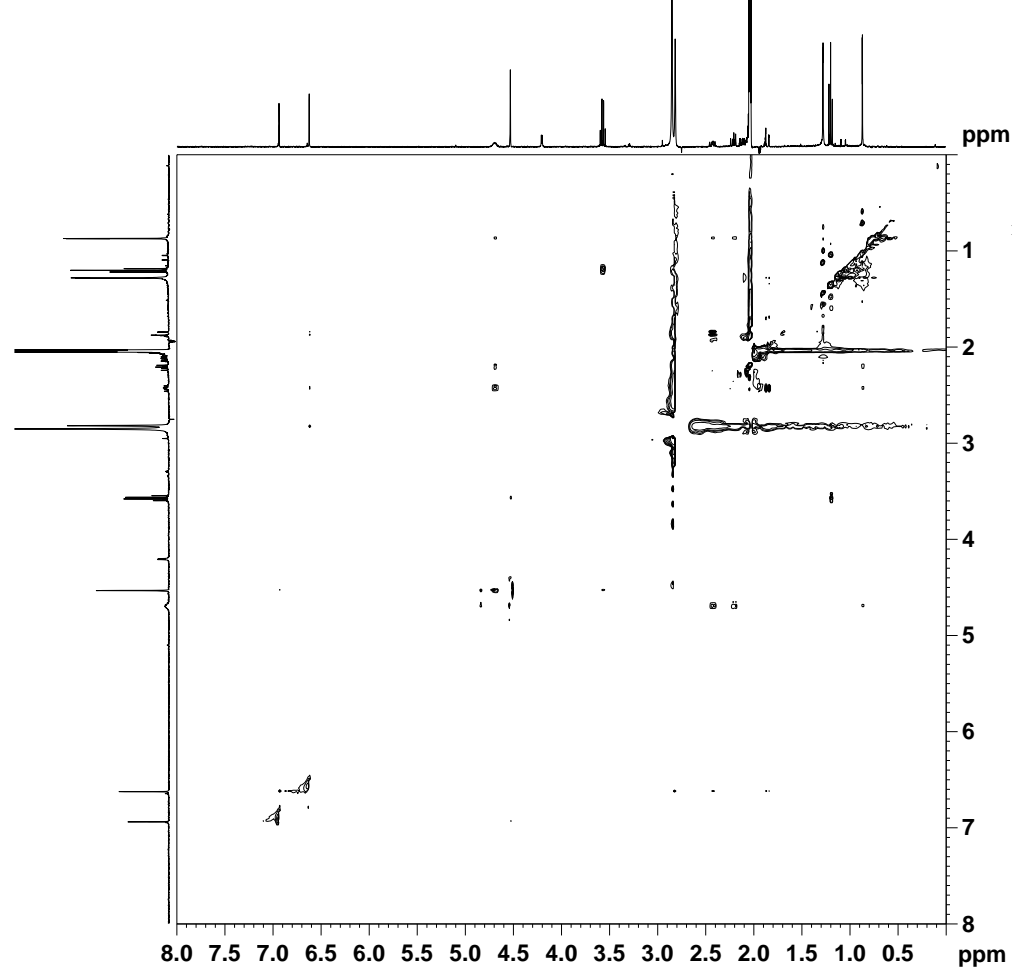
S 16 COSY spectrum of fomitopin-C (3).

FP-5-7-5C Acetone-d6 103/7/31 AVIII400 (COSY90)

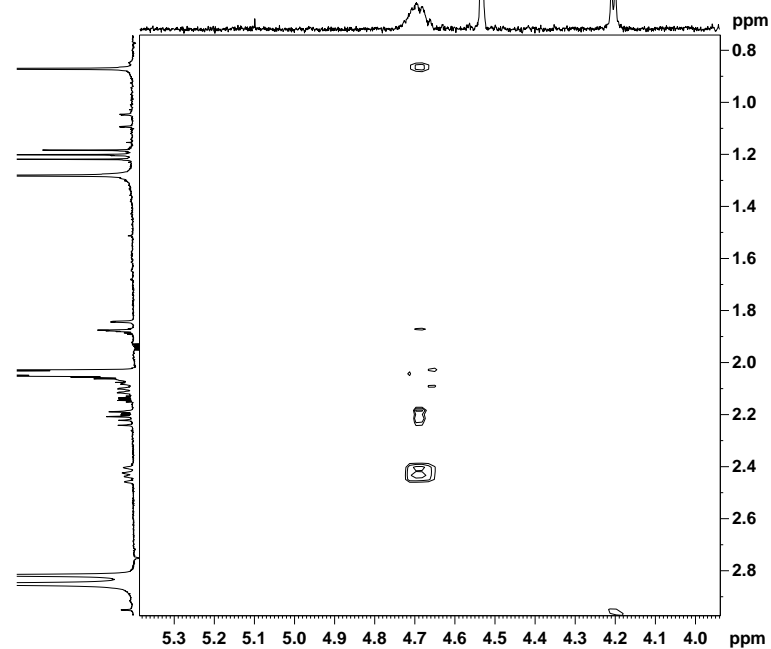


S 17 NOESY spectrum of fomitopin-C (3).

FP-5-7-5C Acetone-d6 103/7/31 AVIII400 (NOESY)

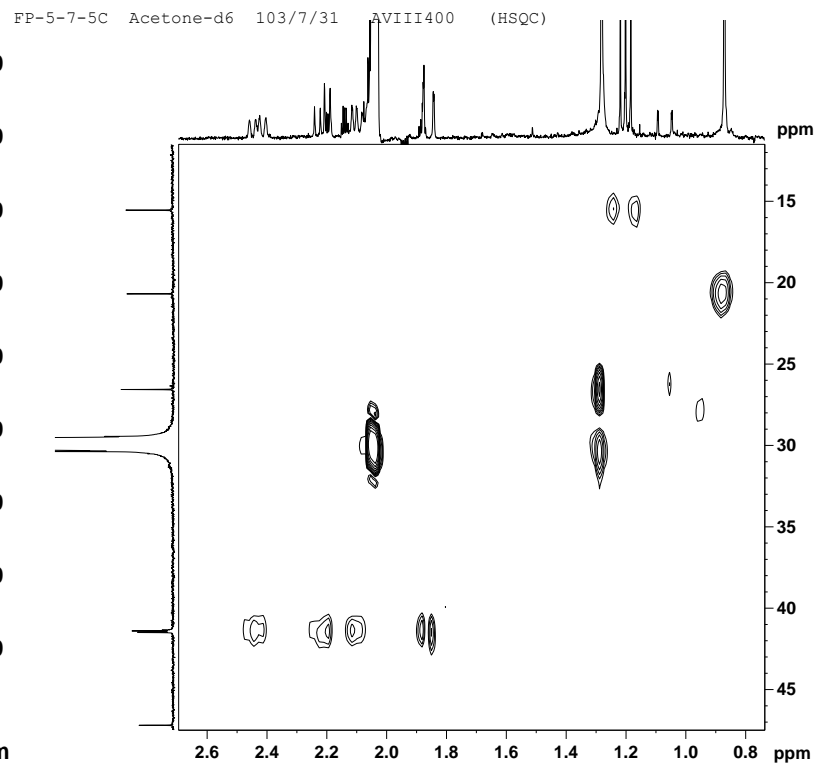
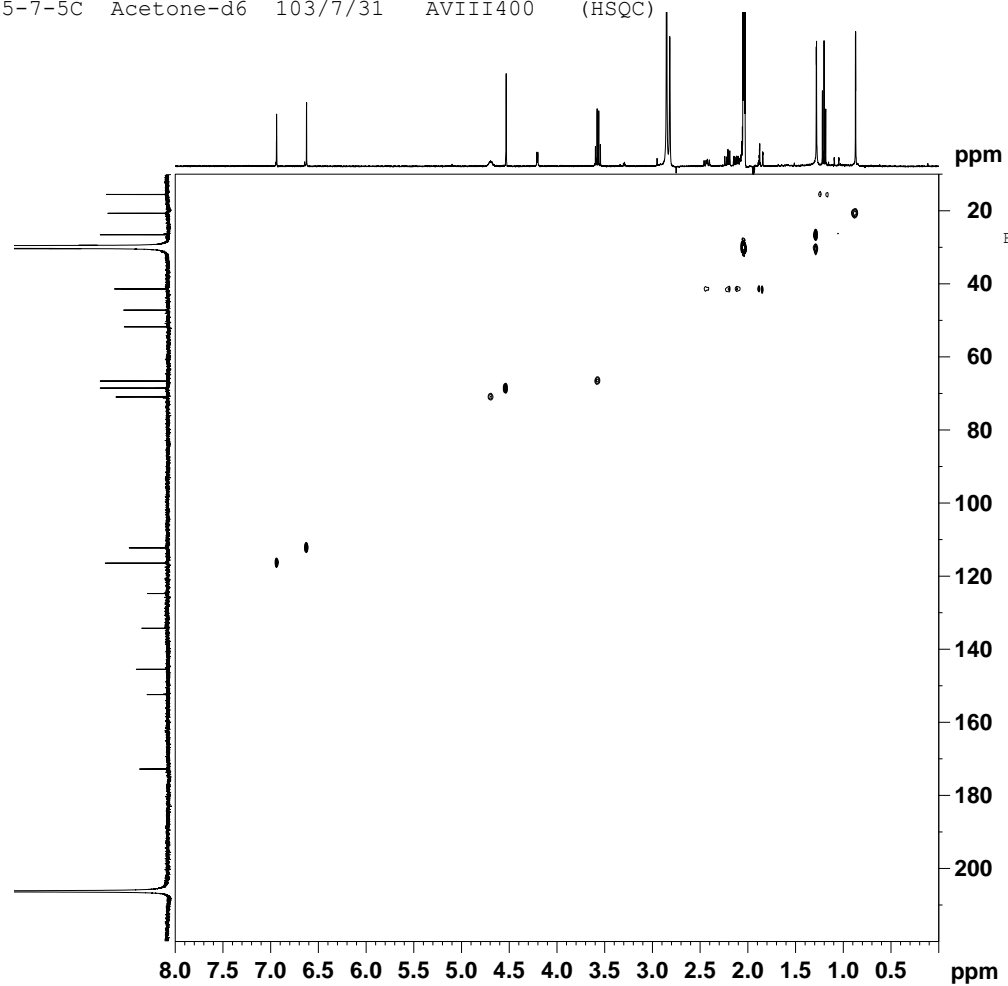


FP-5-7-5C Acetone-d6 103/7/31 AVIII400 (NOESY)

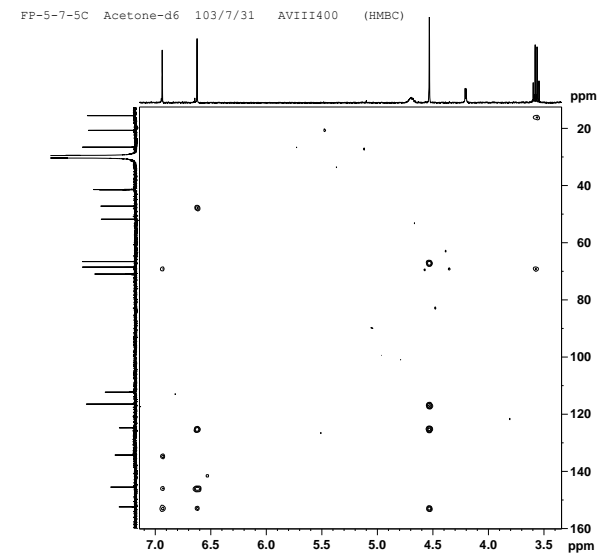
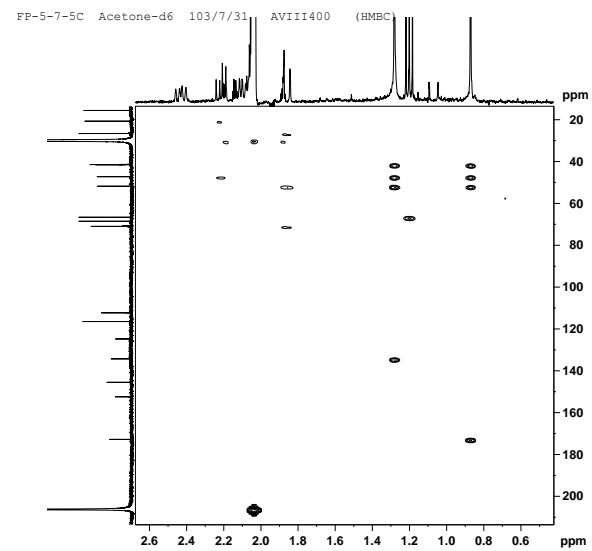
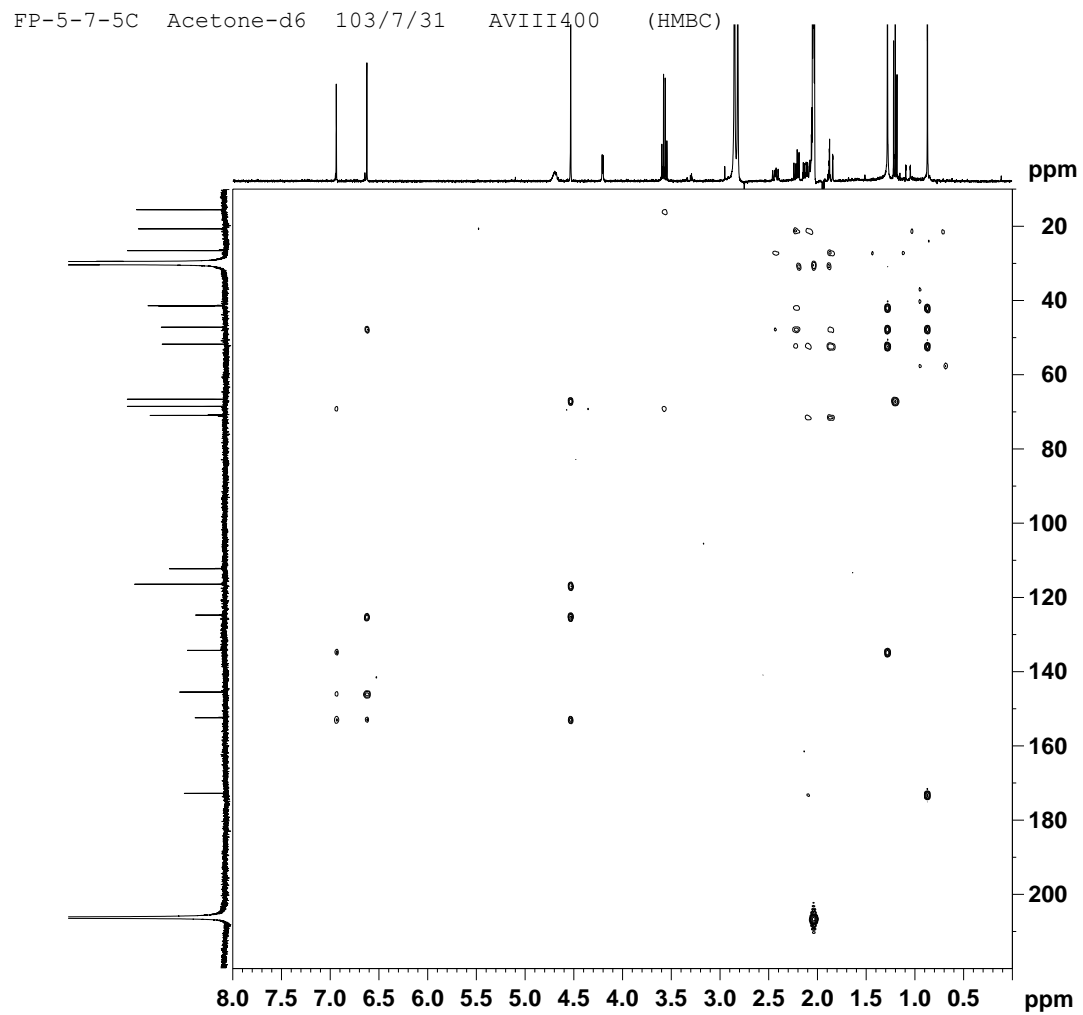


S 18 HSQC spectrum and key of HSQC spectrum of fomitopin-C (3).

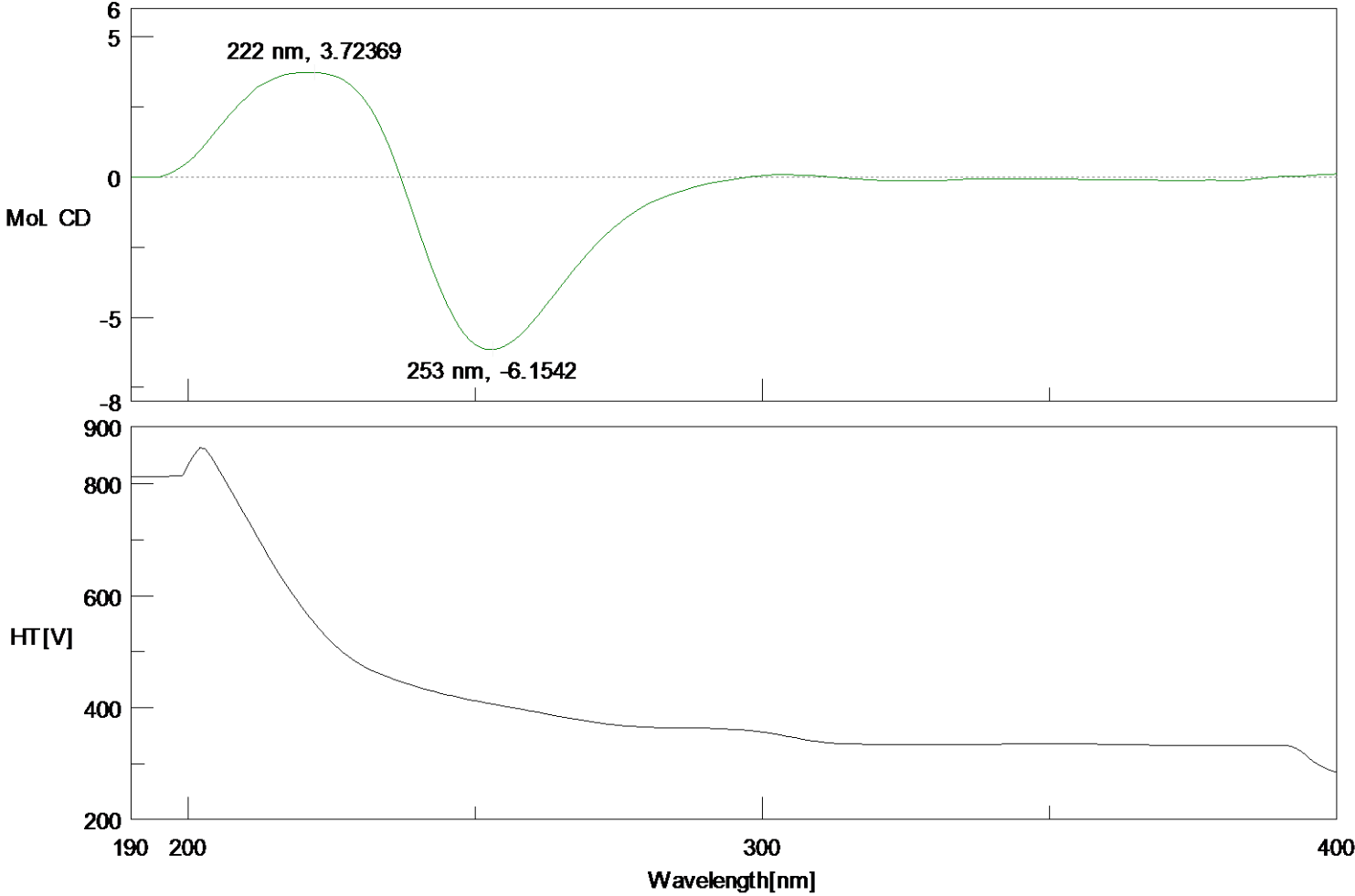
FP-5-7-5C Acetone-d6 103/7/31 AVIII400 (HSQC)



S 19 HMBC spectrum of fomitopin-C (3).

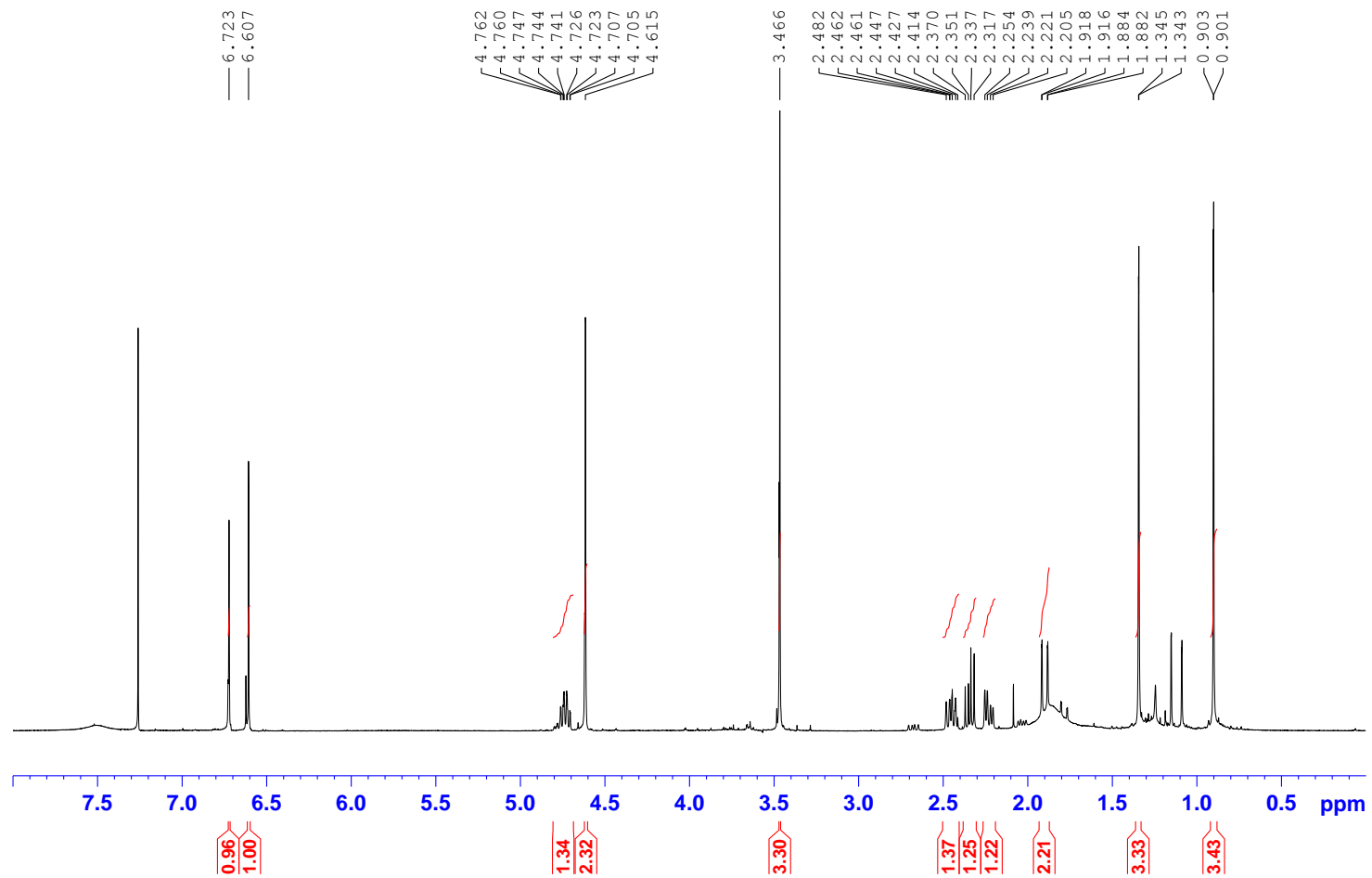


S 20 ECD spectrum of fomitopin-C (3).



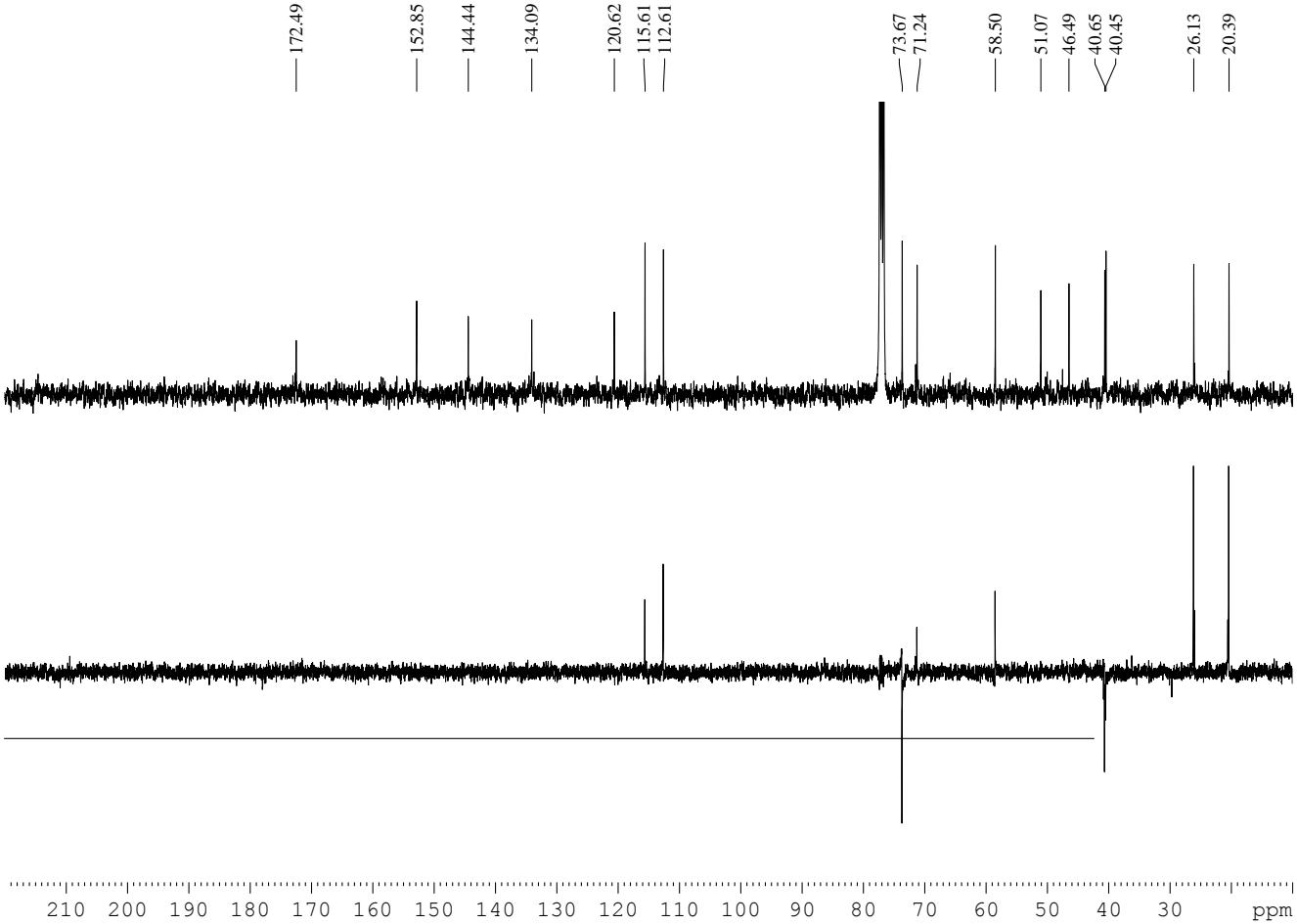
S 21 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-D (4).

FP-4-2+3-t3 CDCl₃ 104/05/21 AVIII400 (H1)



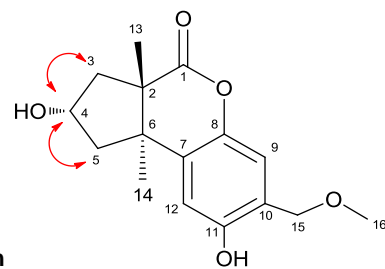
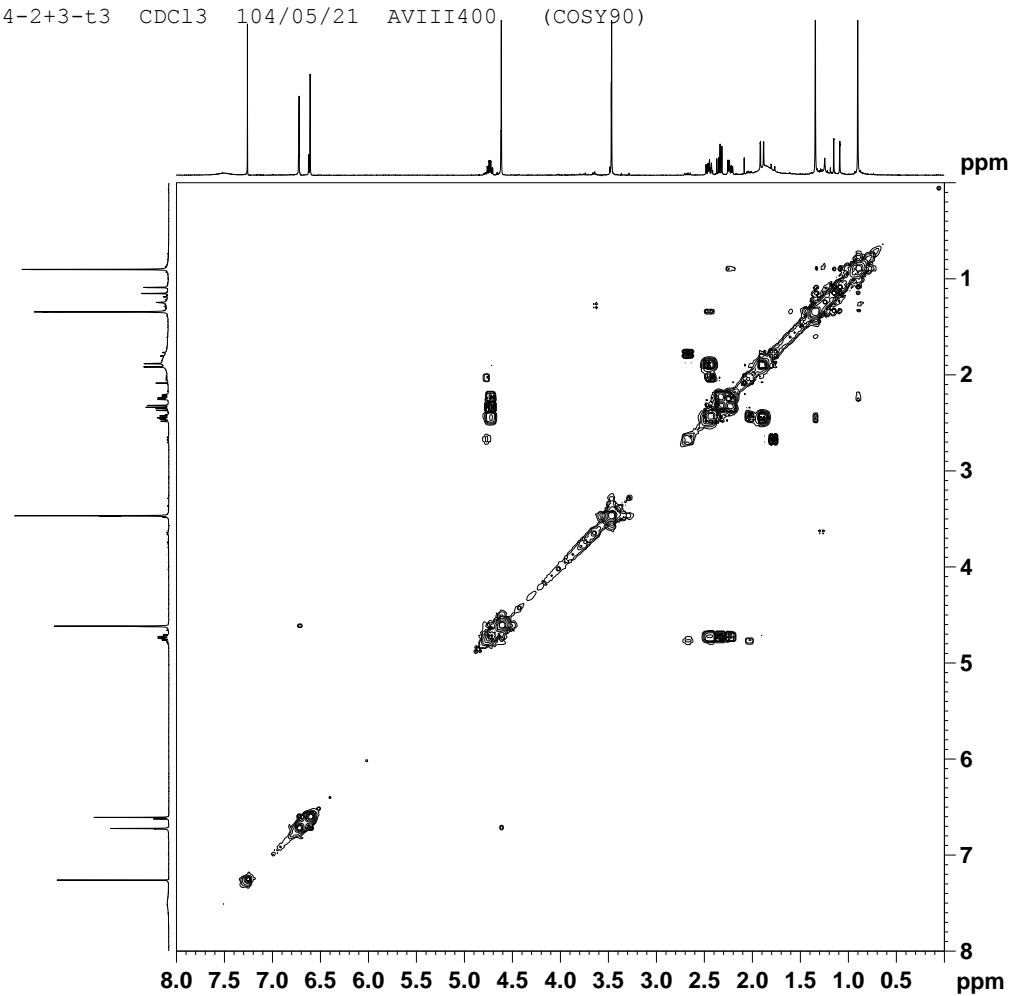
S 22 ¹³C NMR (100 MHz, CDCl₃) spectrum of fomitopin-D (4).

FP-4-2+3-t3 CDC13 104/05/21 AVIII400 (C13)



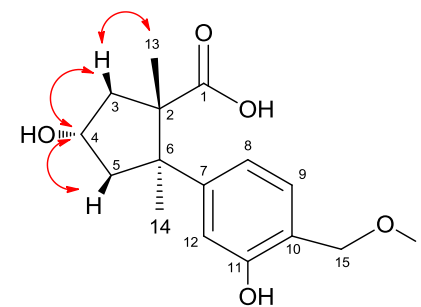
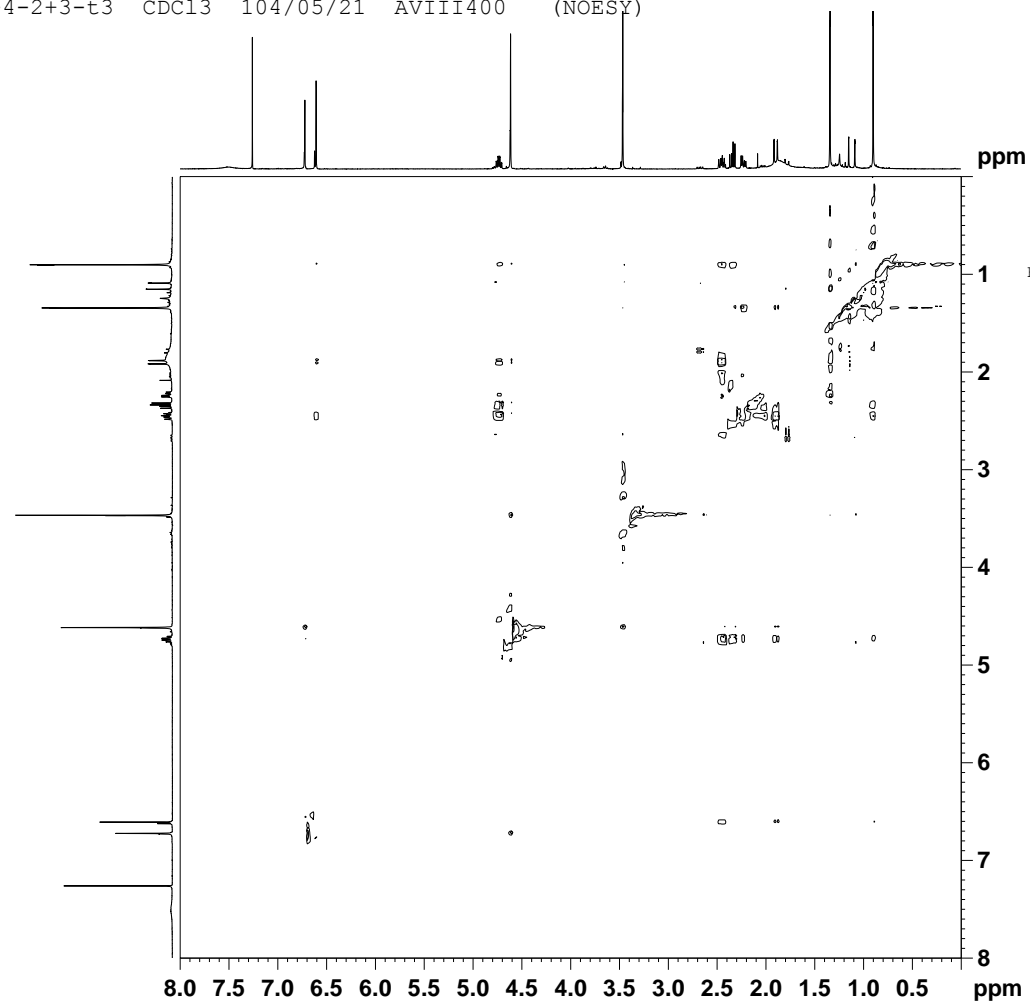
S 23 COSY spectrum of fomitopin-D (4).

FP-4-2+3-t3 CDC13 104/05/21 AVIII400 (COSY90)

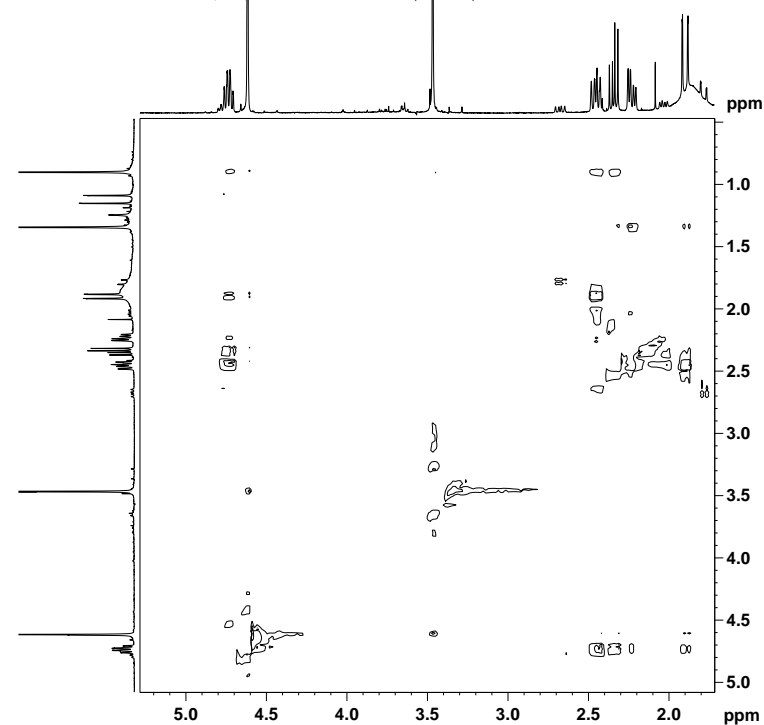


S 24 NOESY spectrum and key of NOESY spectrum of fomitopin-D (4).

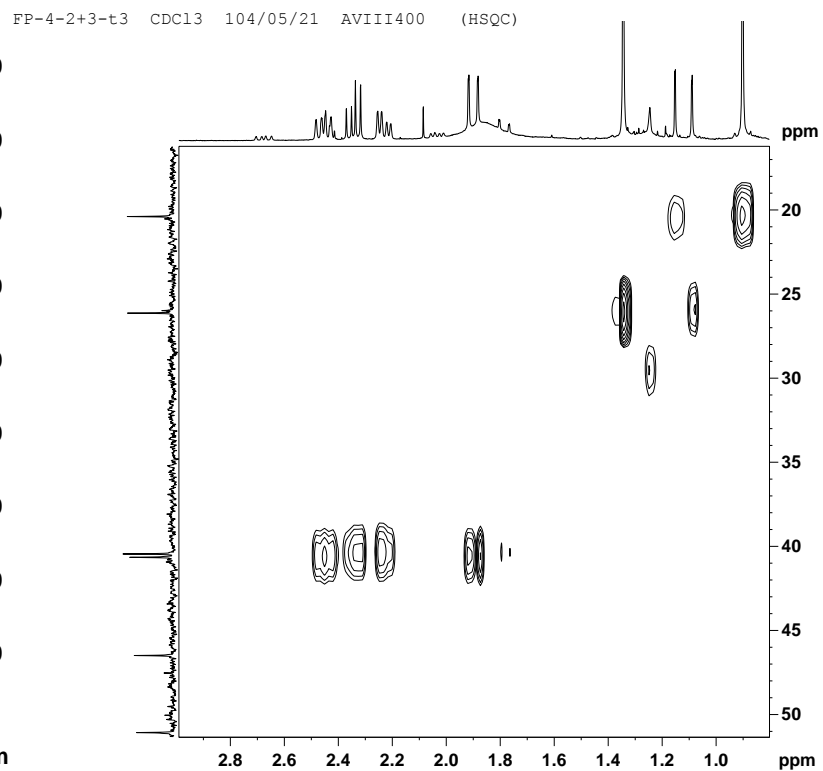
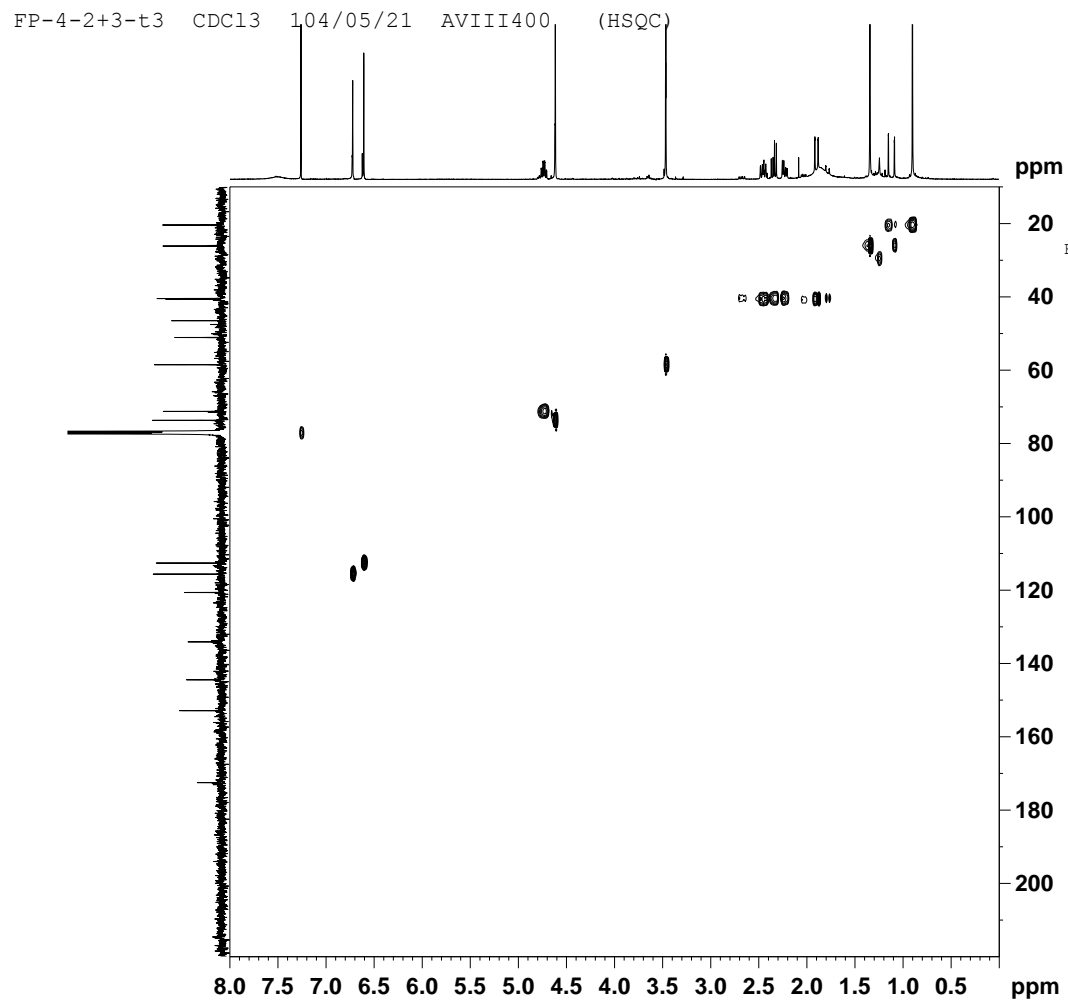
FP-4-2+3-t3 CDC13 104/05/21 AVIII400 (NOESY)



FP-4-2+3-t3 CDC13 104/05/21 AVIII400 (NOESY)

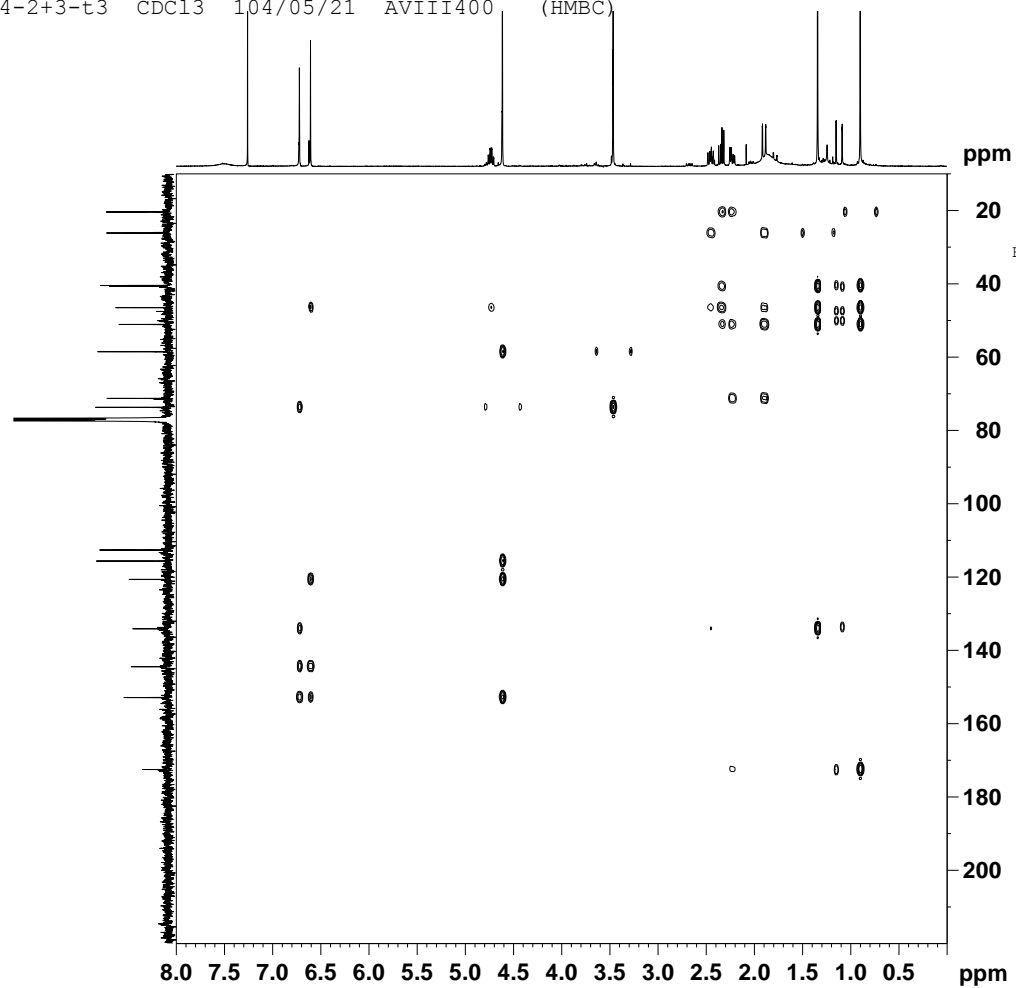


S 25 HSQC spectrum and key of HSQC spectrum of fomitopin-D (4).

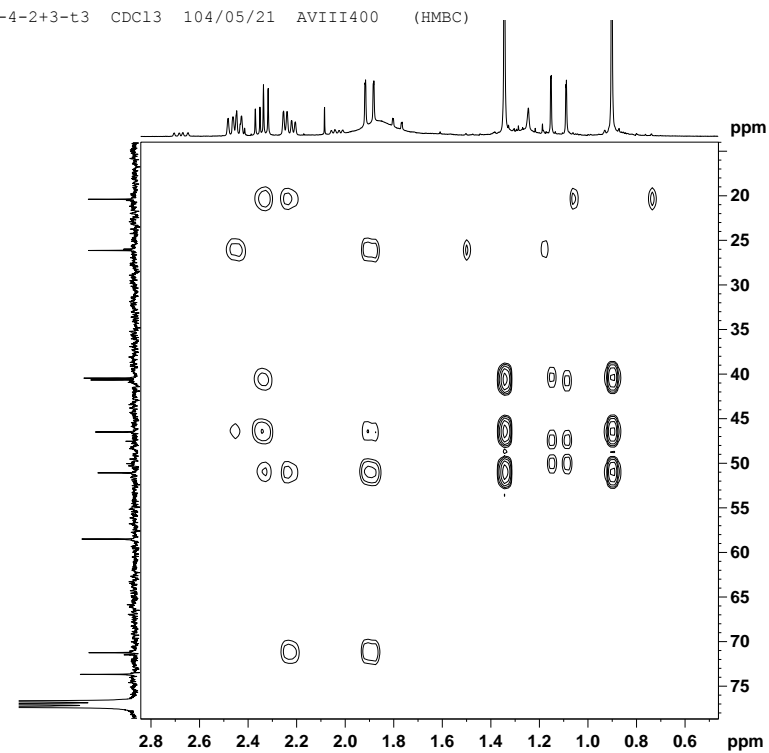


S 26 HMBC spectrum of fomitopin-D (4).

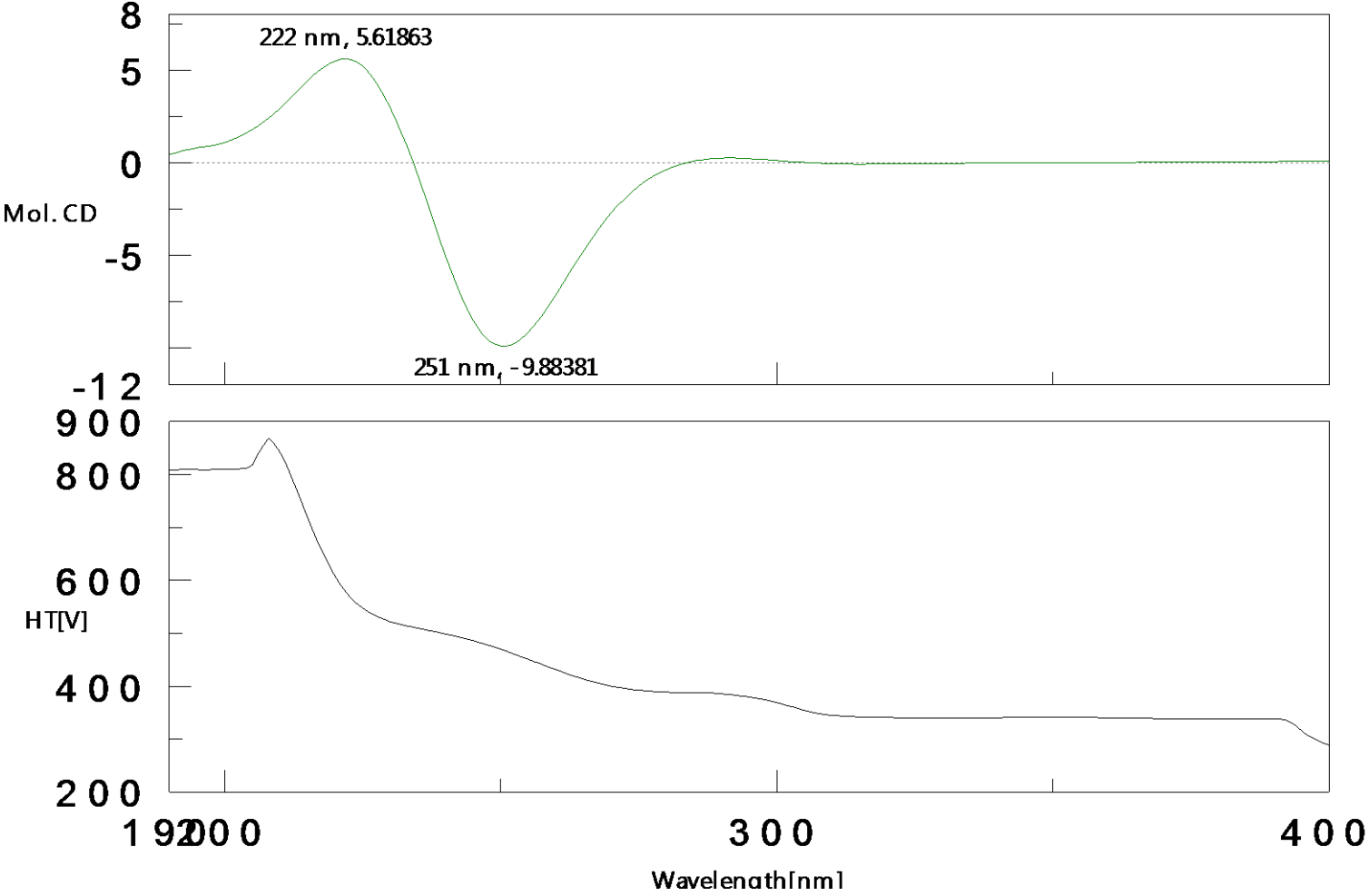
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FP-4-2+3-t3 CDC13 104/05/21 AVIII400 (HMBC)

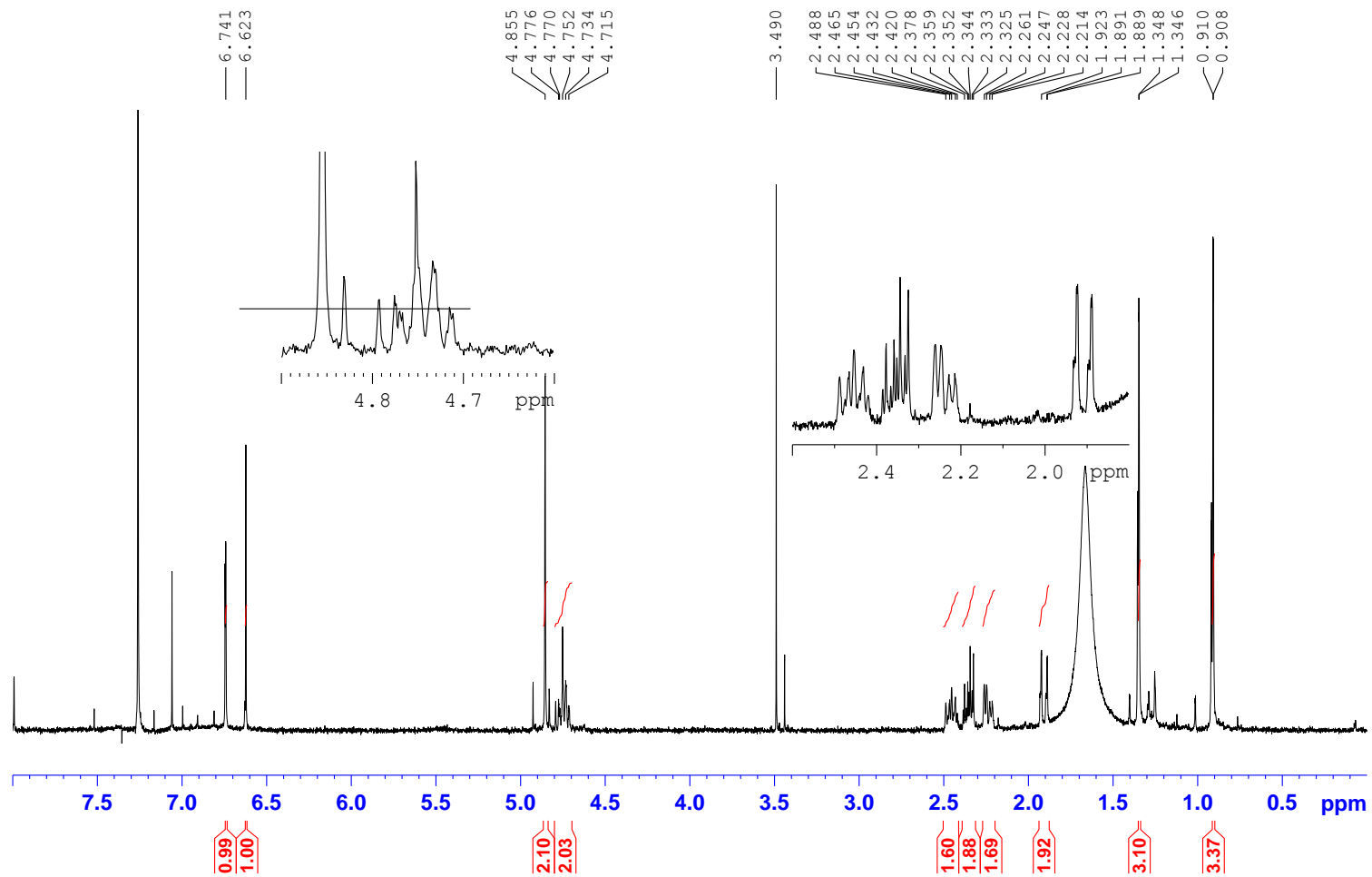


S 27 ECD spectrum of fomitopin-D (4).



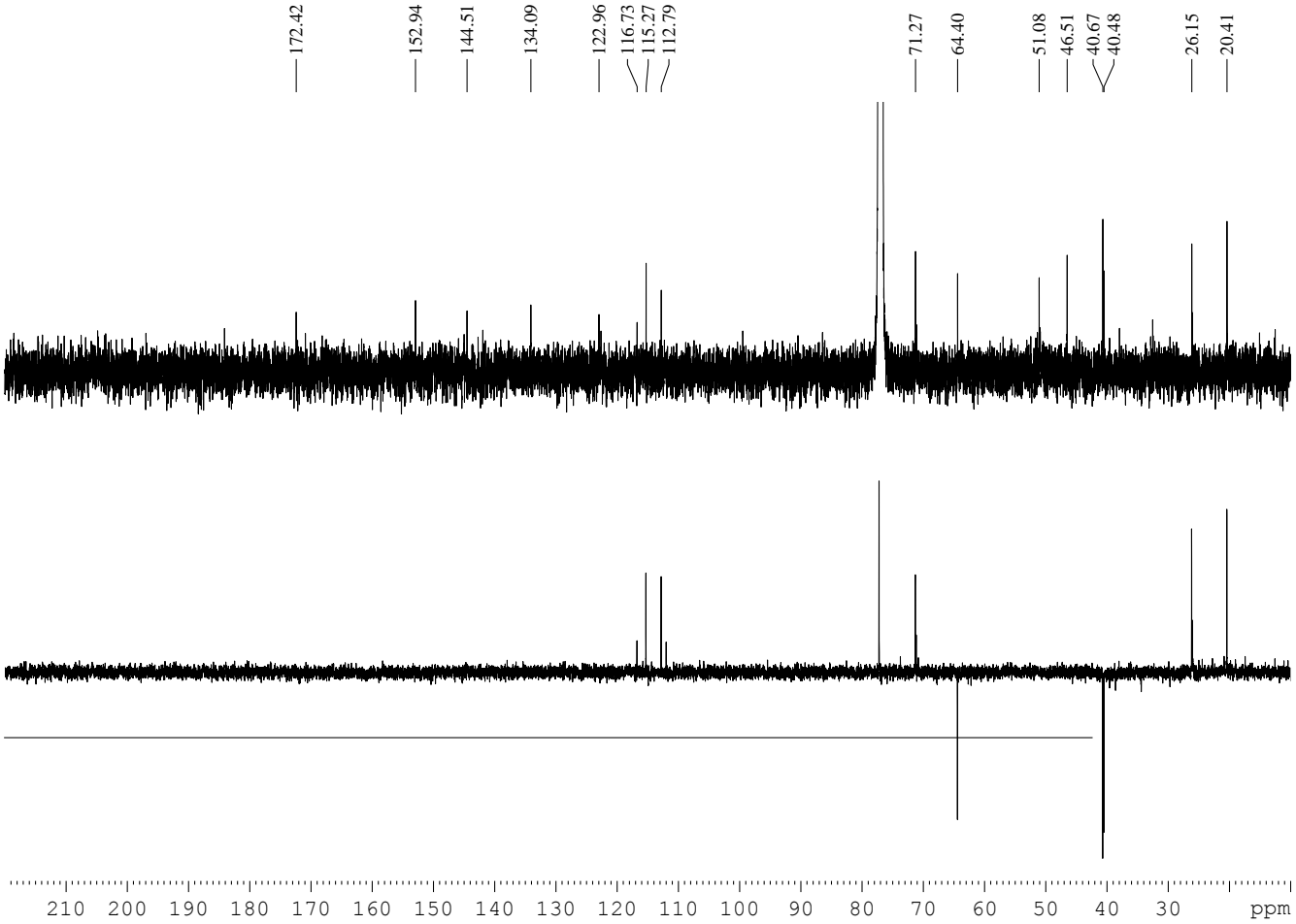
S 28 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-E (5).

FP-4-4+5-3-h1 CDCl₃ 104/05/26 AVIII400 (H1)



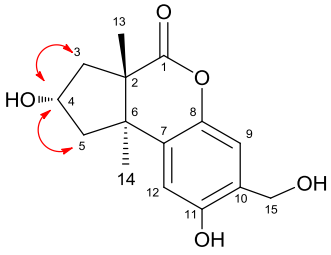
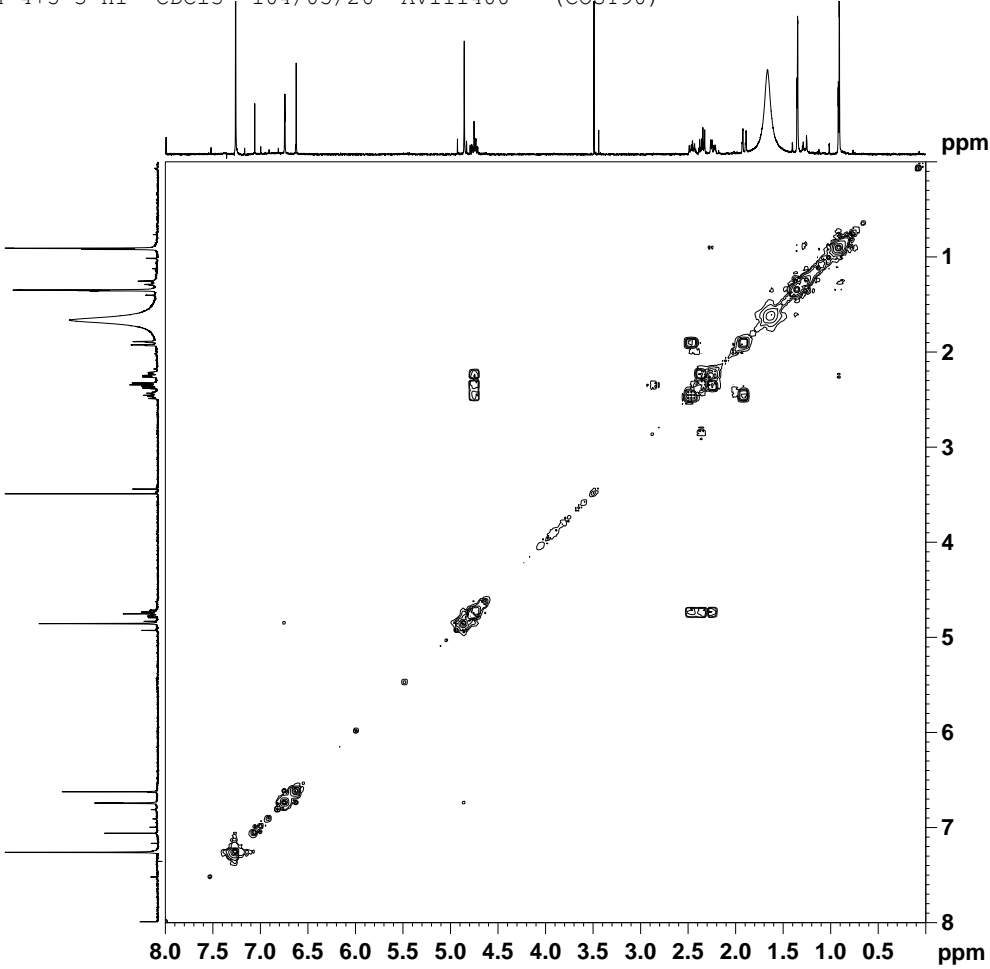
S 29 ¹³C NMR (100 MHz, CDCl₃) spectrum of fomitopin-E (5).

FP-4-4+5-3-h1 CDC13 104/05/26 AVIII400 (C13)



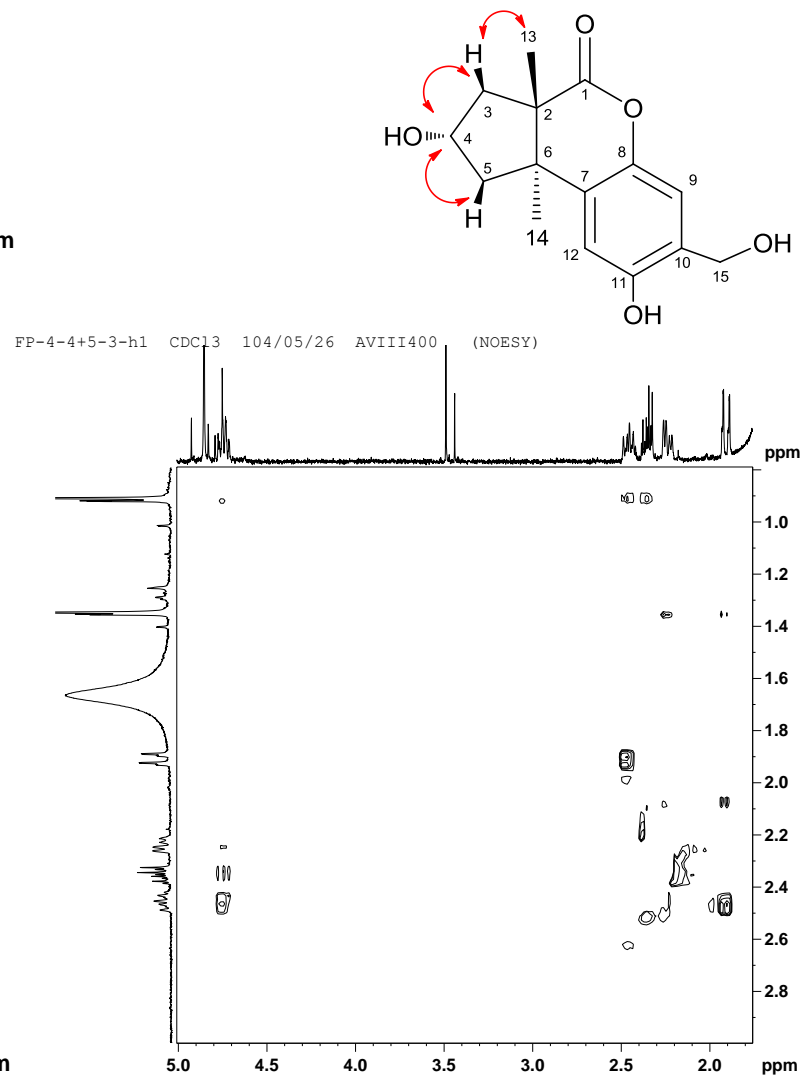
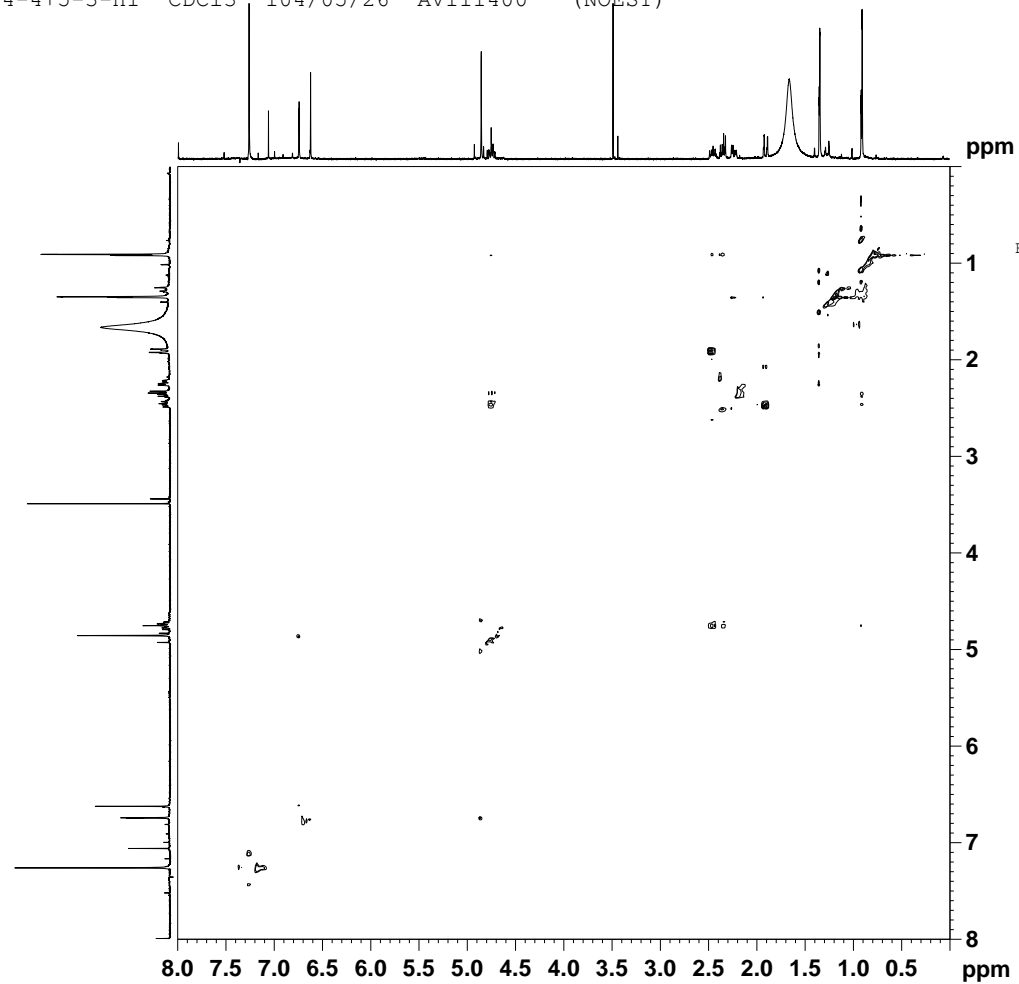
S 30 COSY spectrum of fomitopin-E (5).

FP-4-4+5-3-h1 CDC13 104/05/26 AVIII400 (COSY90)



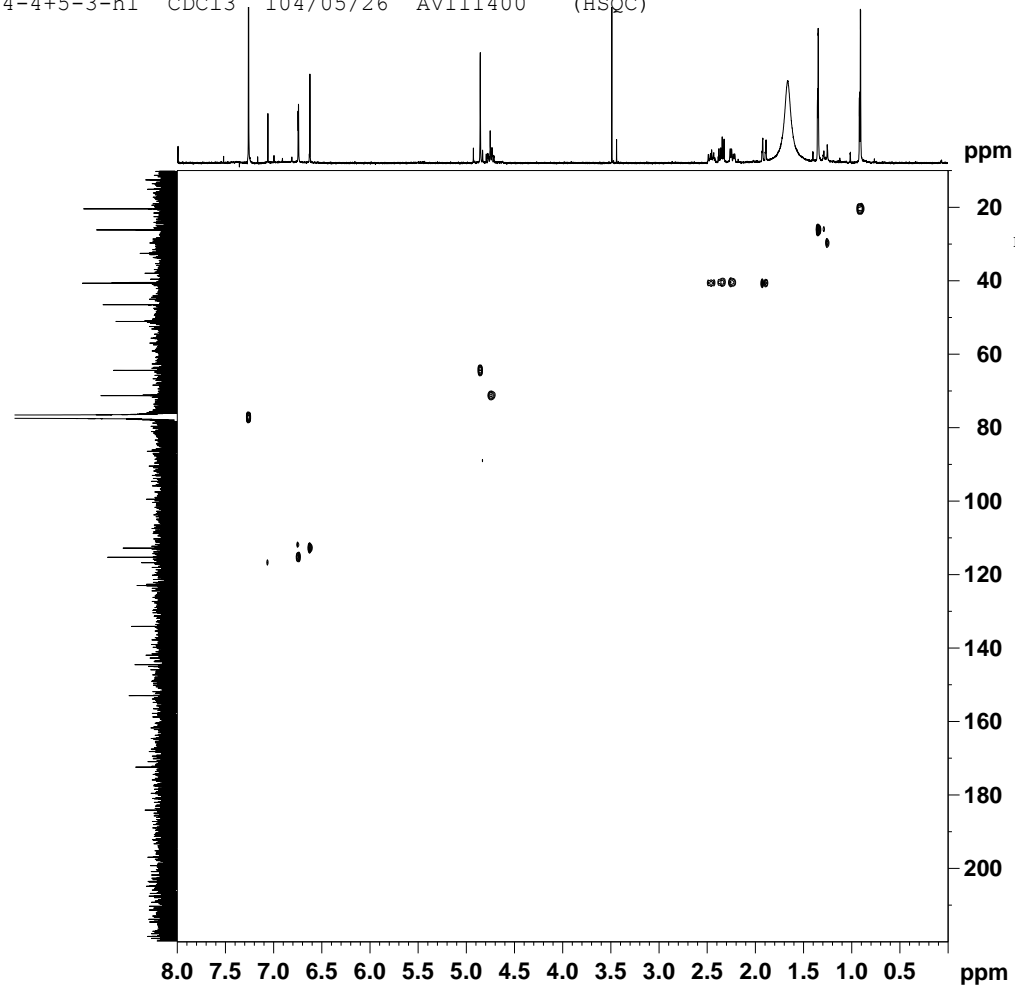
S 31 NOESY spectrum and key of NOESY spectrum of fomitopin-E (5).

FP-4-4+5-3-h1 CDCl3 104/05/26 AVIII400 (NOESY)

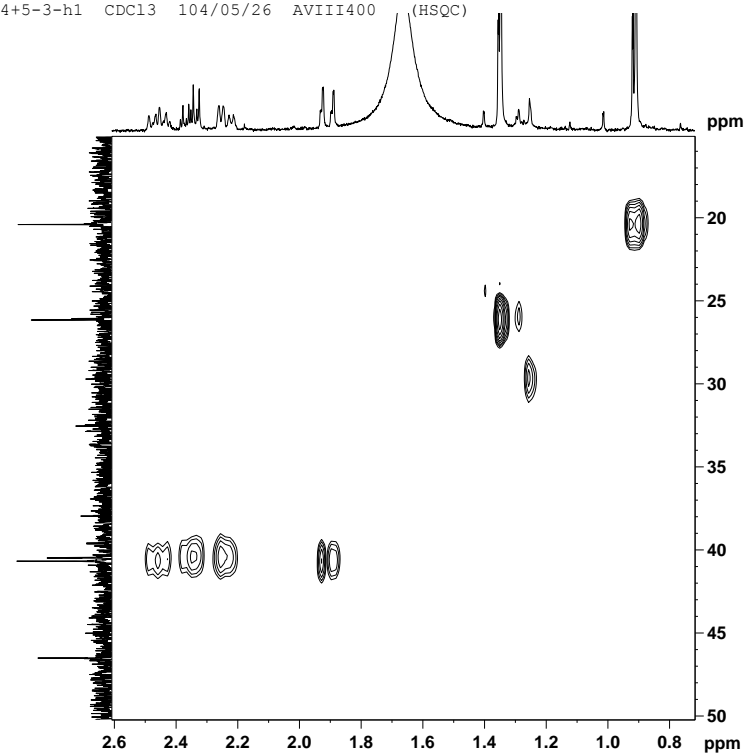


S 32 HSQC spectrum and key of HSQC spectrum of fomitopin-E (5).

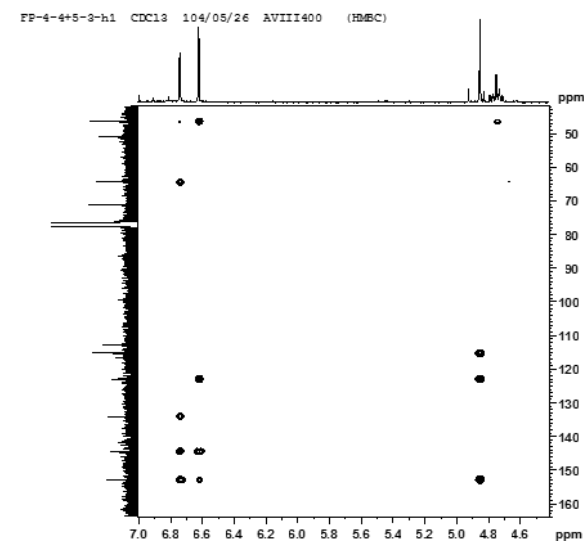
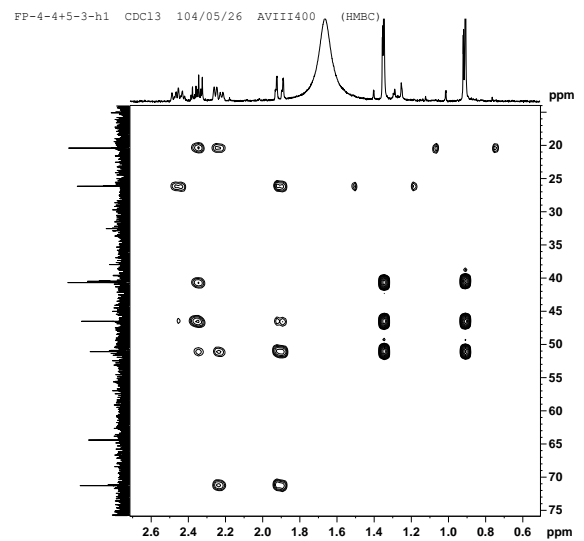
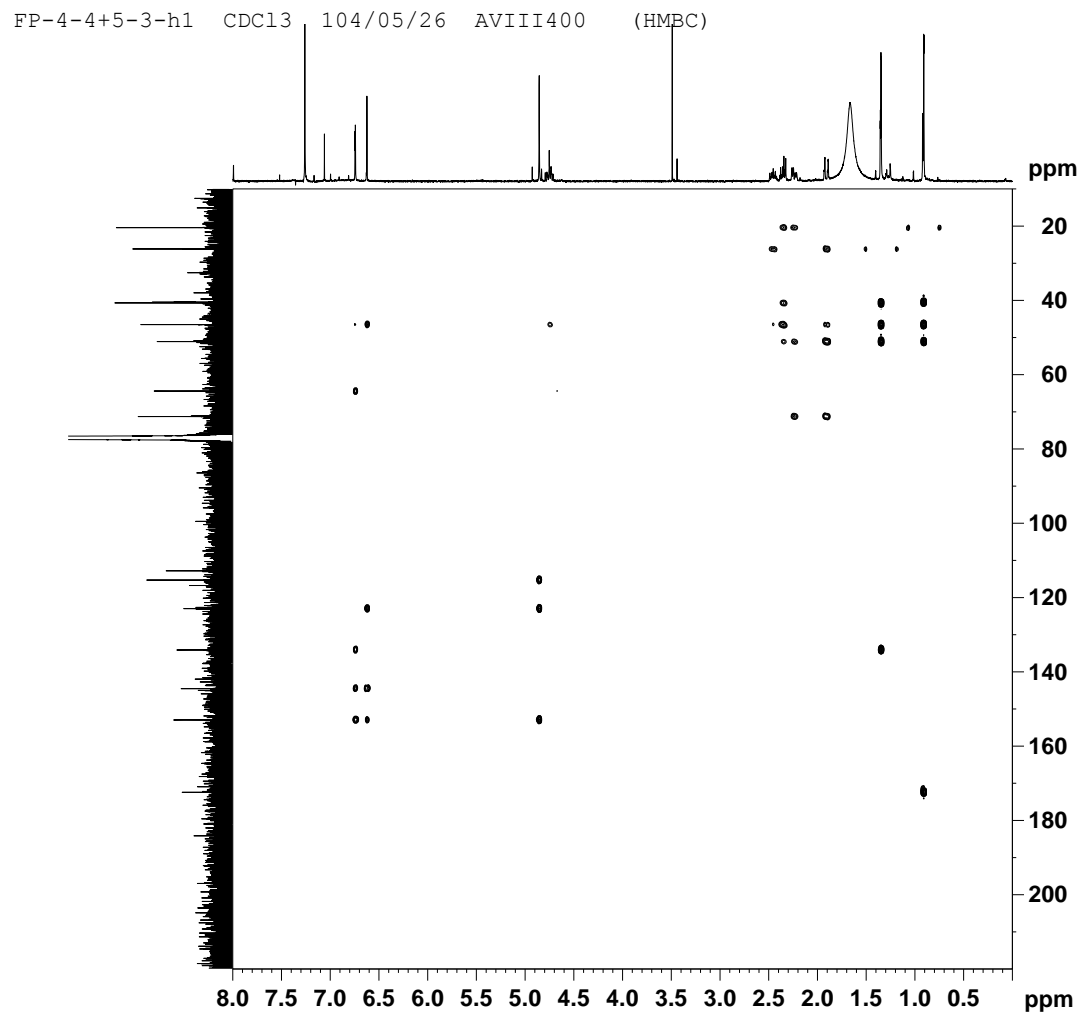
FP-4-4+5-3-h1 CDCl3 104/05/26 AVIII400 (HSQC)



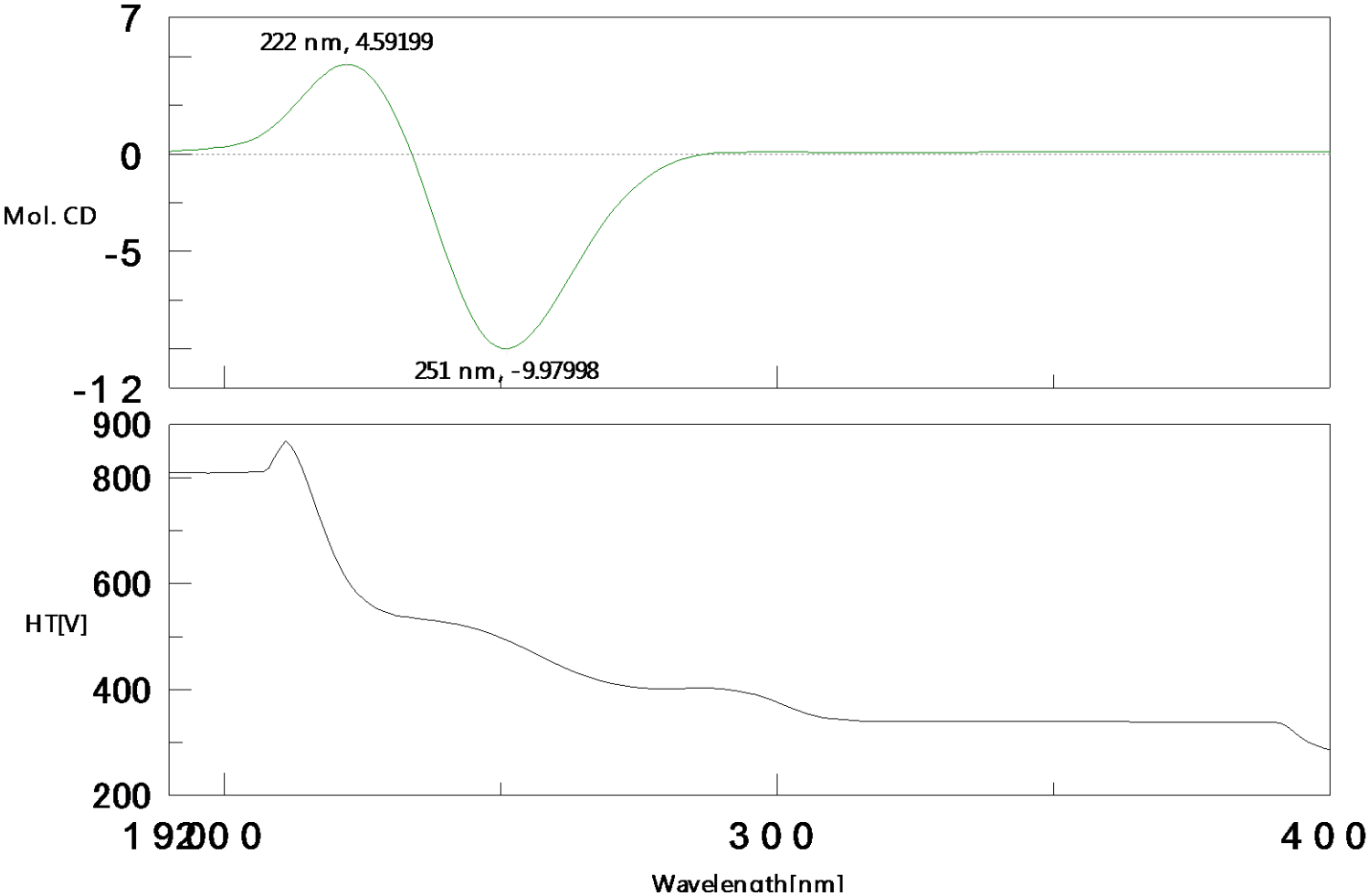
FP-4-4+5-3-h1 CDCl3 104/05/26 AVIII400 (HSQC)



S 33 HMBC spectrum of fomitopin-E (5).

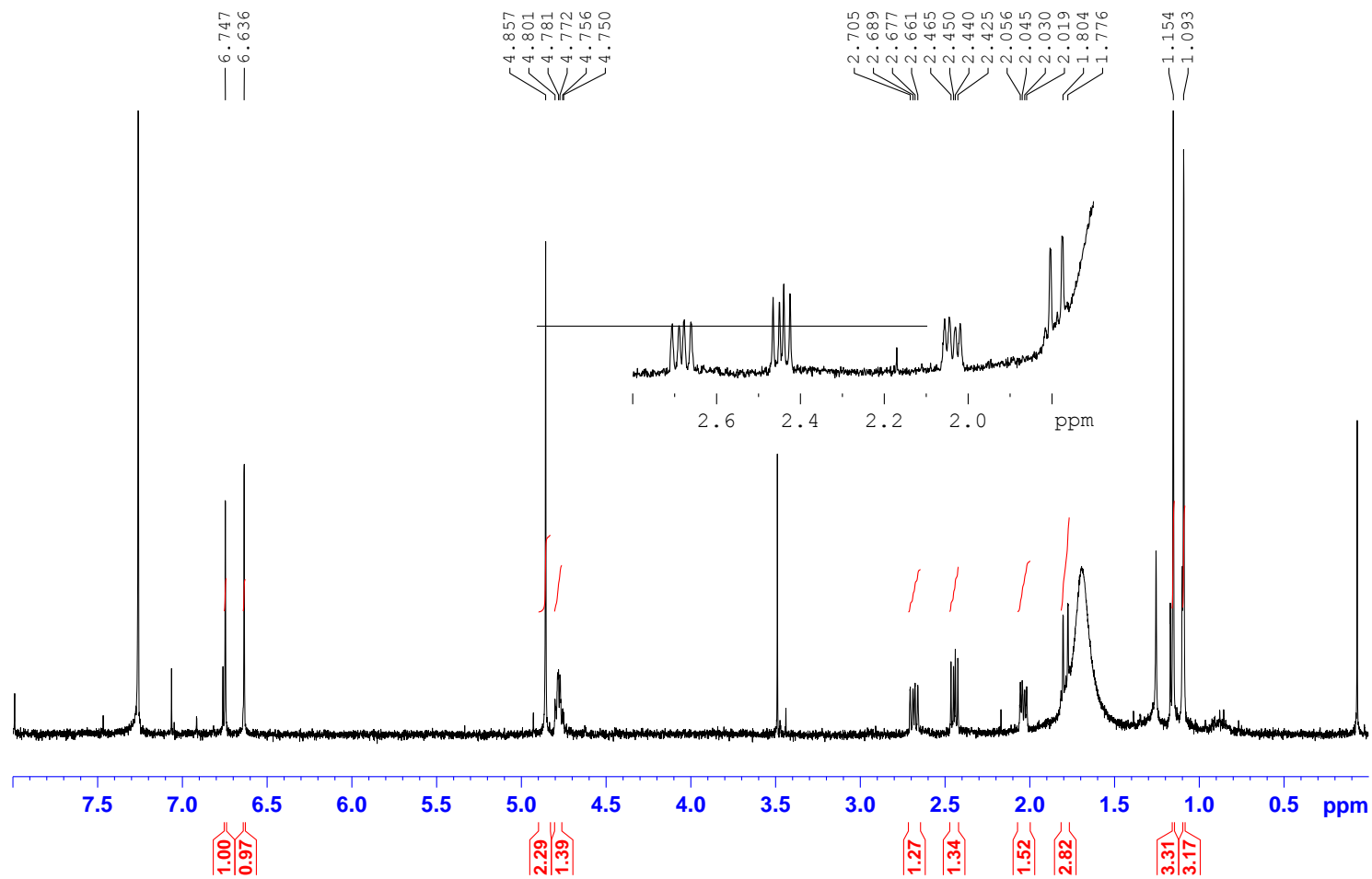


S 34 ECD spectrum of fomitopin-E (5).



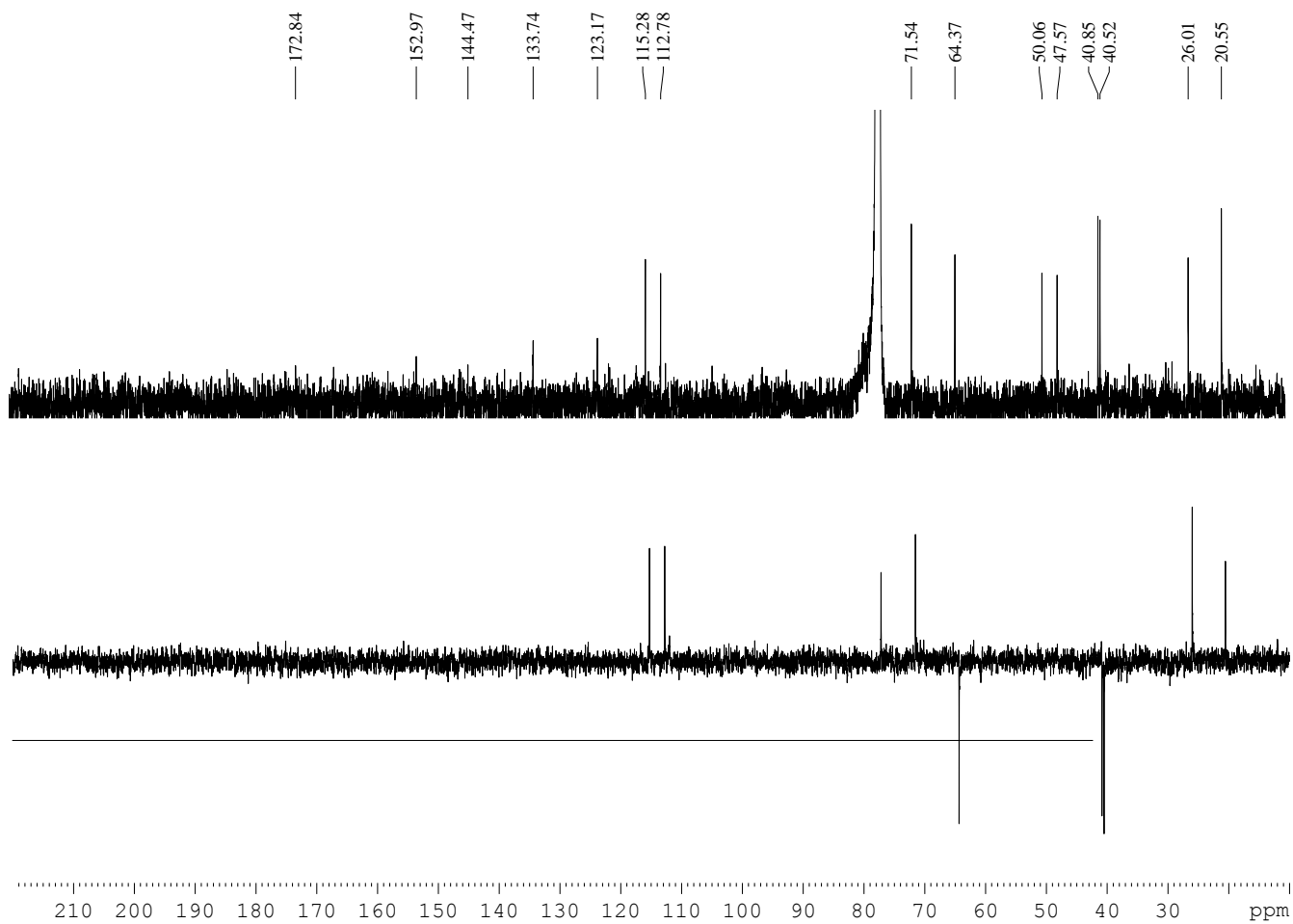
S 35 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-F (6).

FP-4-4+5-3-h2 CDCl₃ 2015/06/03 AV500



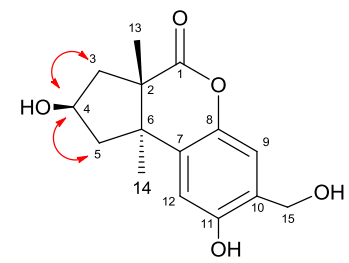
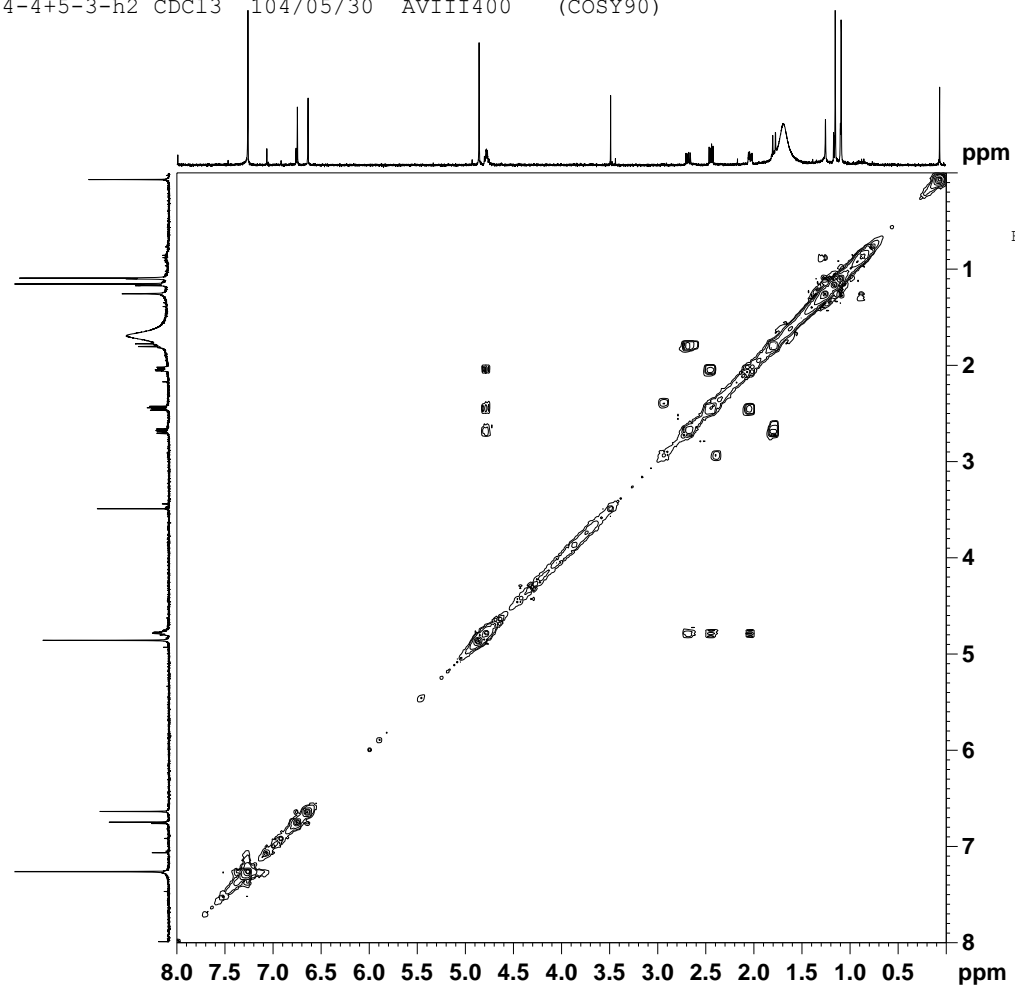
S 36 ¹³C NMR (100 MHz, CDCl₃) spectrum of fomitopin-F (6).

FP-4-4+5-3-h2 CDC13 2015/06/03 AV500 C13

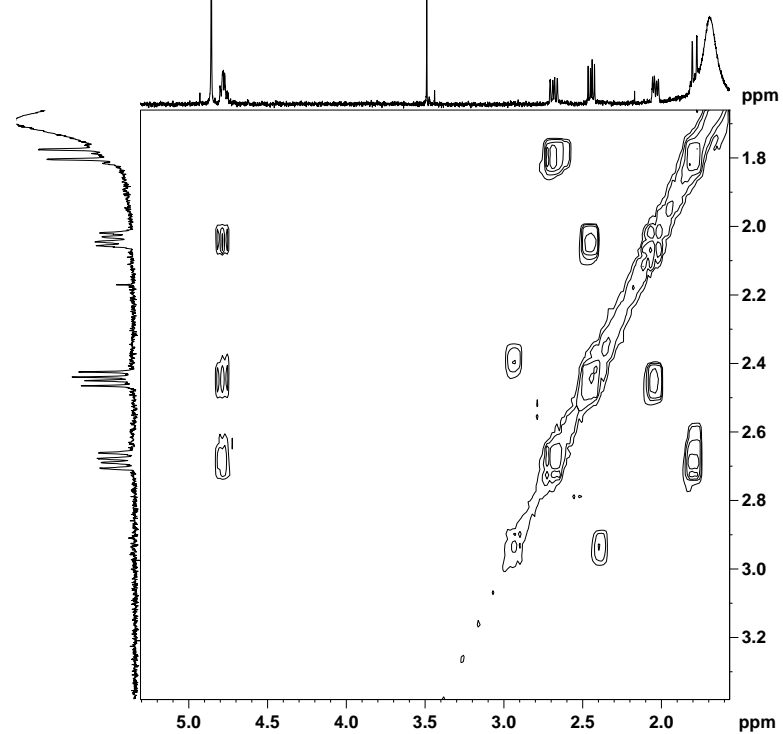


S 37 COSY spectrum of fomitopin-F (6).

FP-4-4+5-3-h2 CDCl₃ 104/05/30 AVIII400 (COSY90)

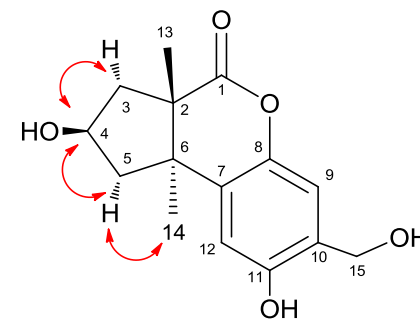
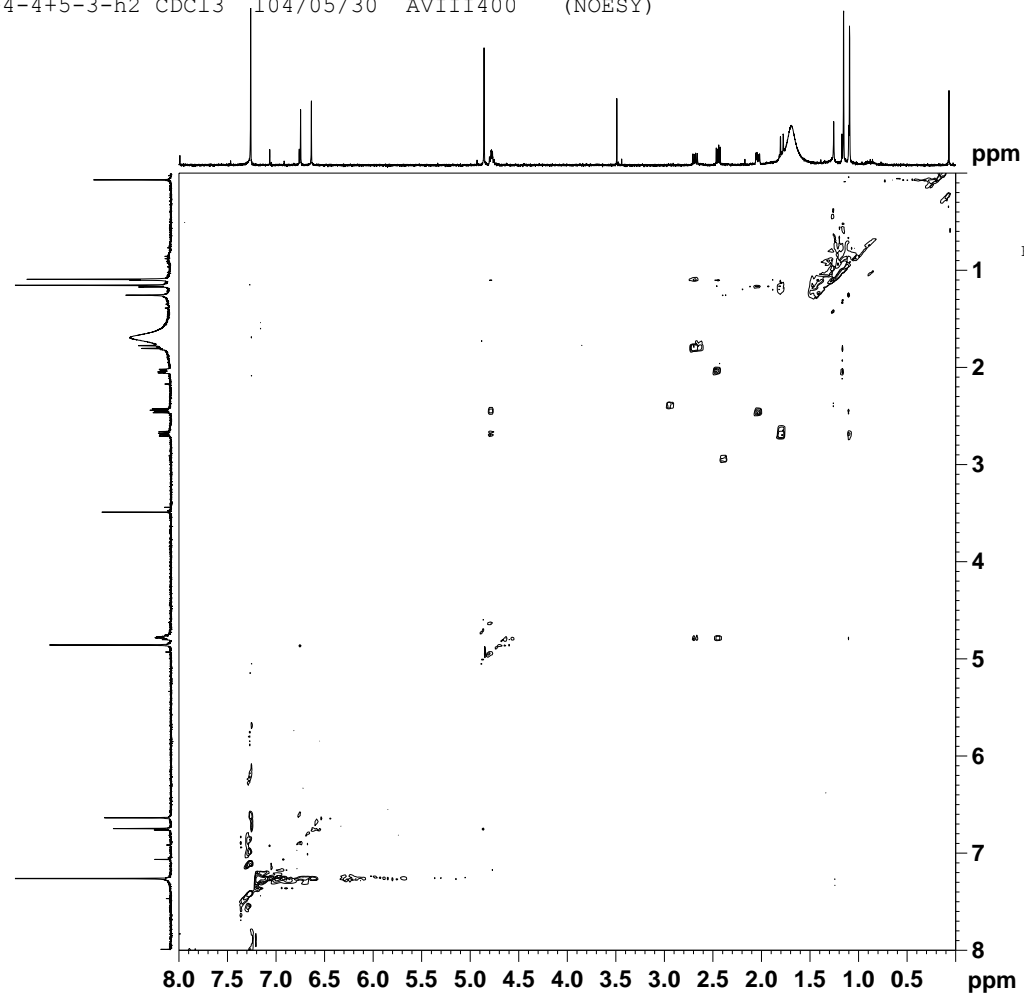


FP-4-4+5-3-h2 CDCl₃ 104/05/30 AVIII400 (COSY90)

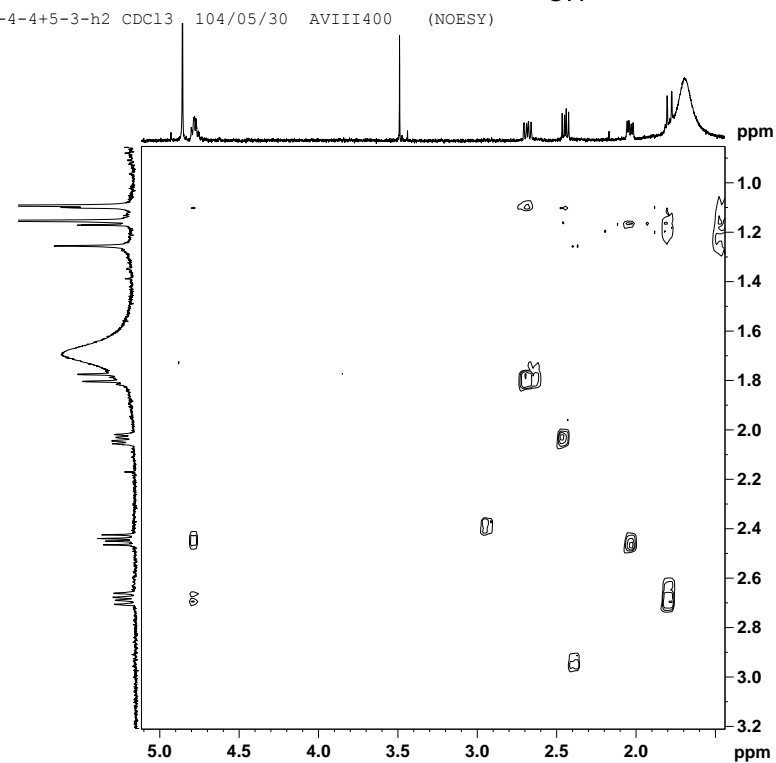


S 38 NOESY spectrum of fomitopin-F (6).

FP-4-4+5-3-h2 CDC13 104/05/30 AVIII400 (NOESY)

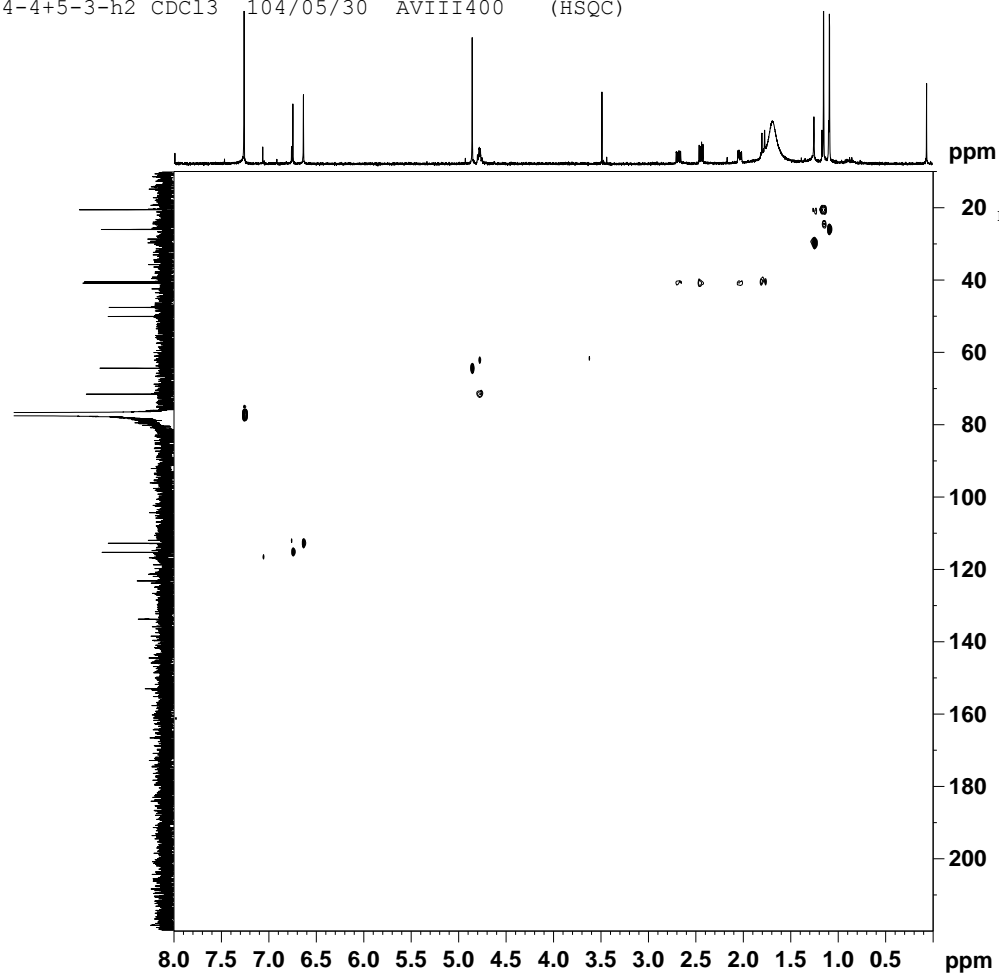


FP-4-4+5-3-h2 CDC13 104/05/30 AVIII400 (NOESY)

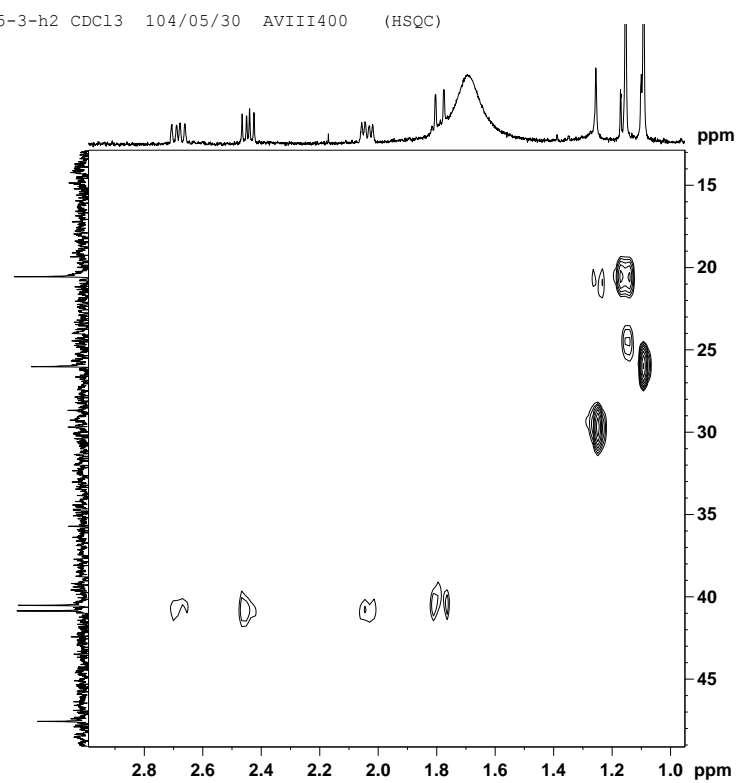


S 39 HSQC spectrum and key of HSQC spectrum of fomitopin-F (6).

FP-4-4+5-3-h2 CDCl3 104/05/30 AVIII400 (HSQC)

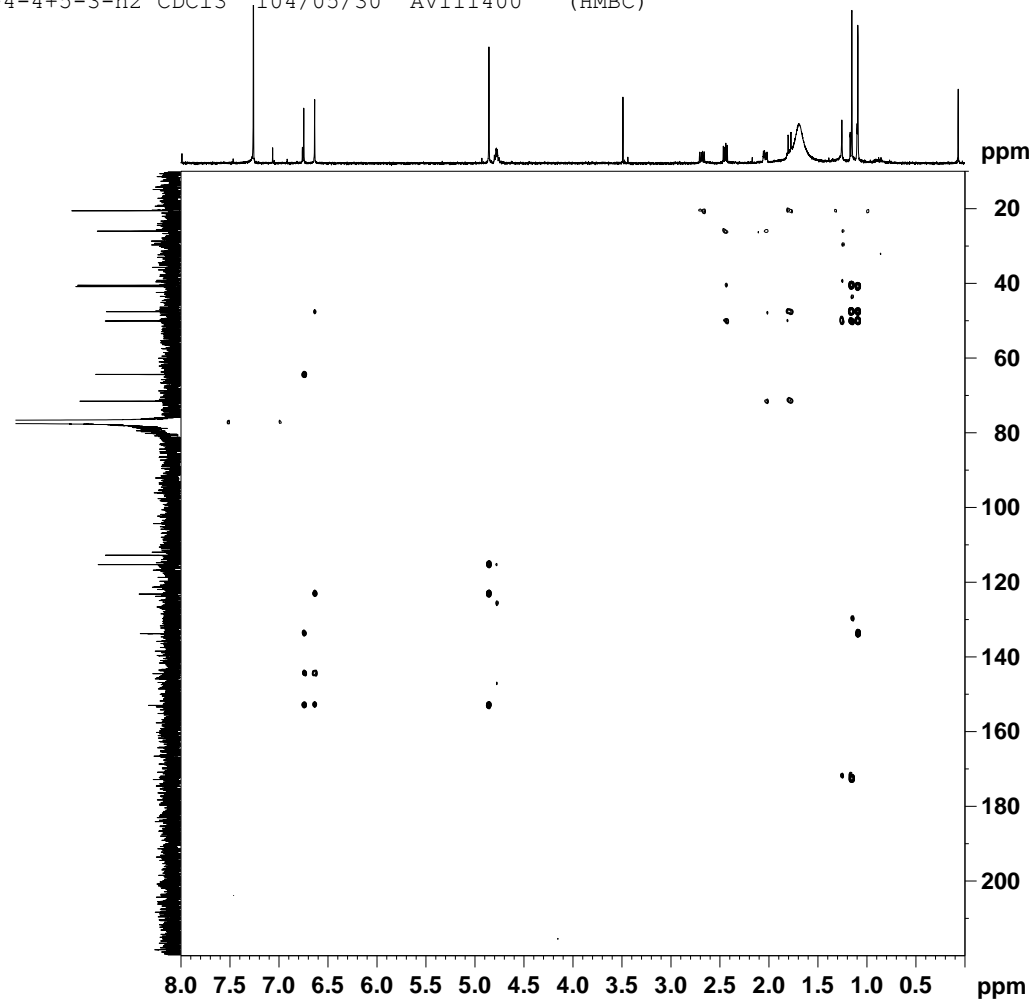


FP-4-4+5-3-h2 CDCl3 104/05/30 AVIII400 (HSQC)

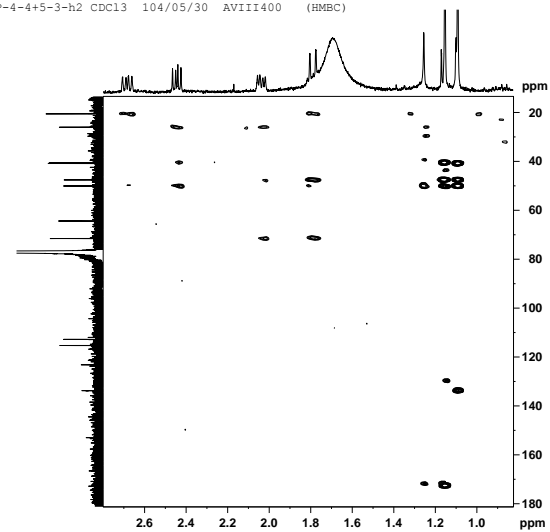


S 40 HMBC spectrum of fomitopin-F (6).

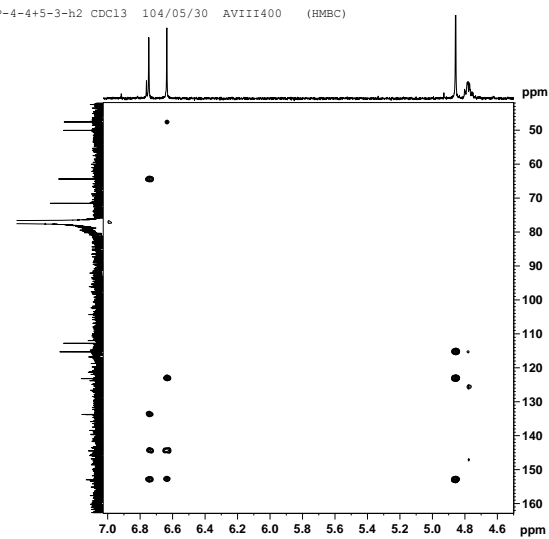
FP-4-4+5-3-h2 CDCl3 104/05/30 AVIII400 (HMBC)



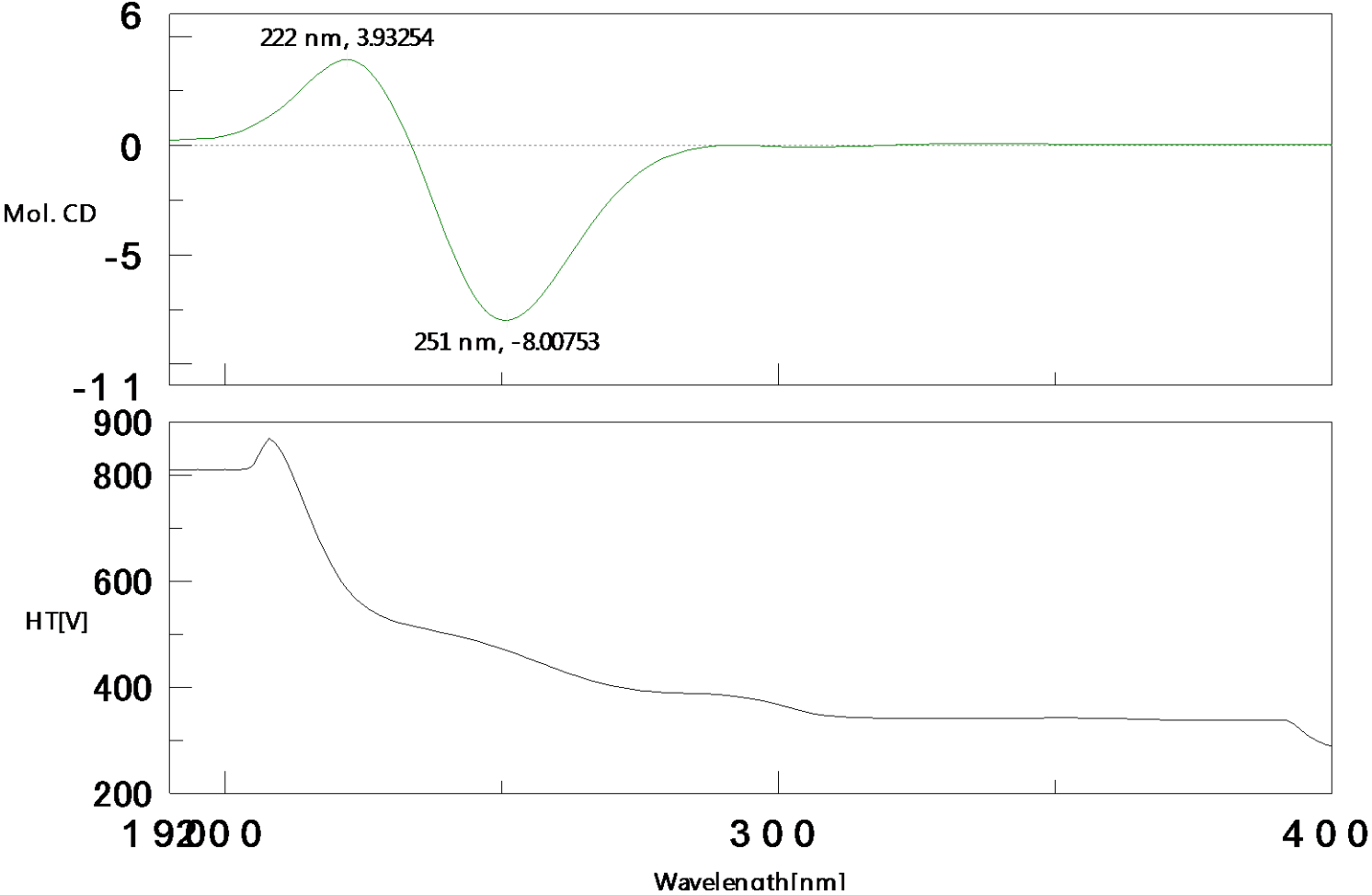
FP-4-4+5-3-h2 CDCl3 104/05/30 AVIII400 (HMBC)



FP-4-4+5-3-h2 CDCl3 104/05/30 AVIII400 (HMBC)

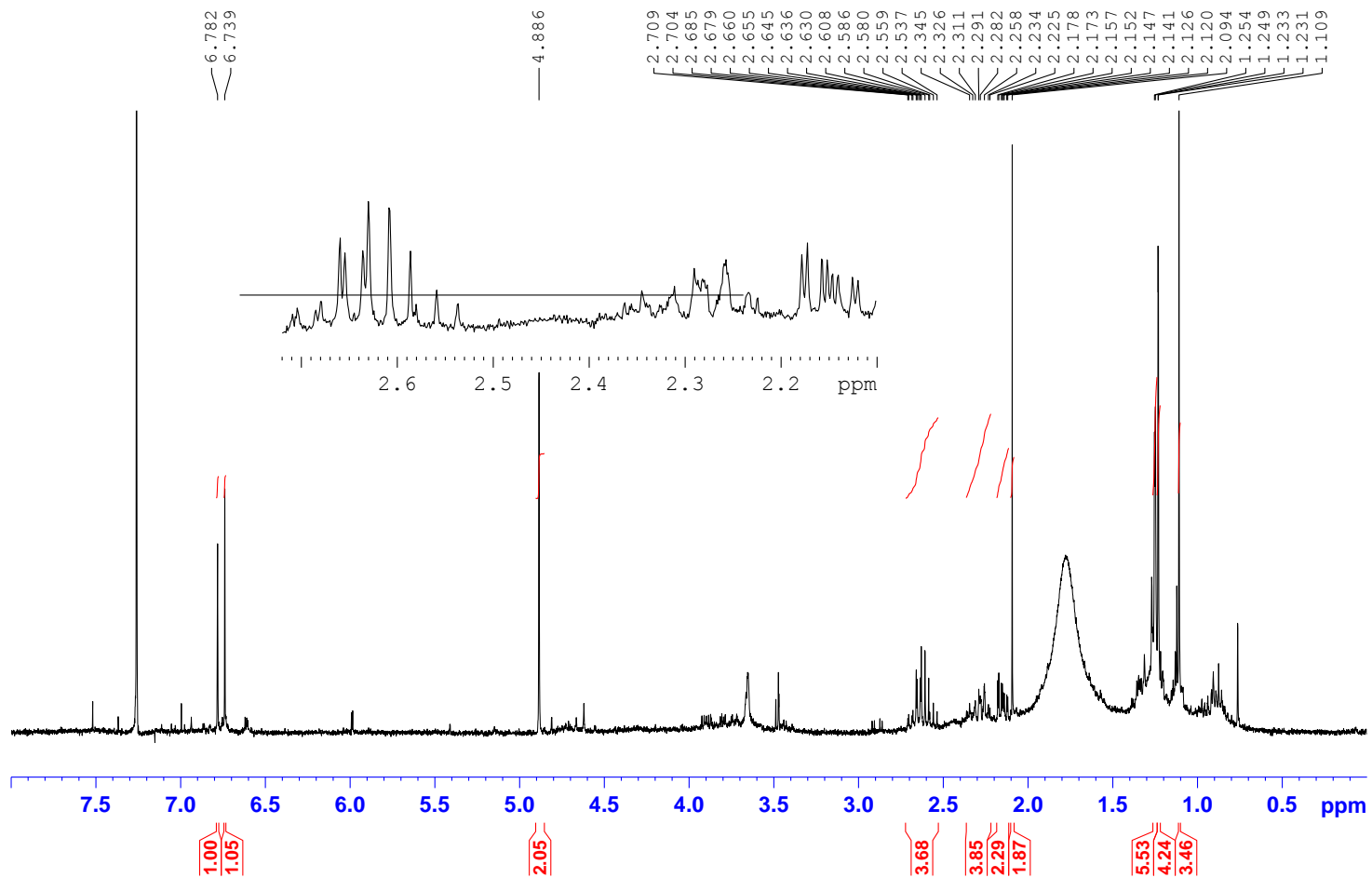


S 41 ECD spectrum of fomitopin-F (6).



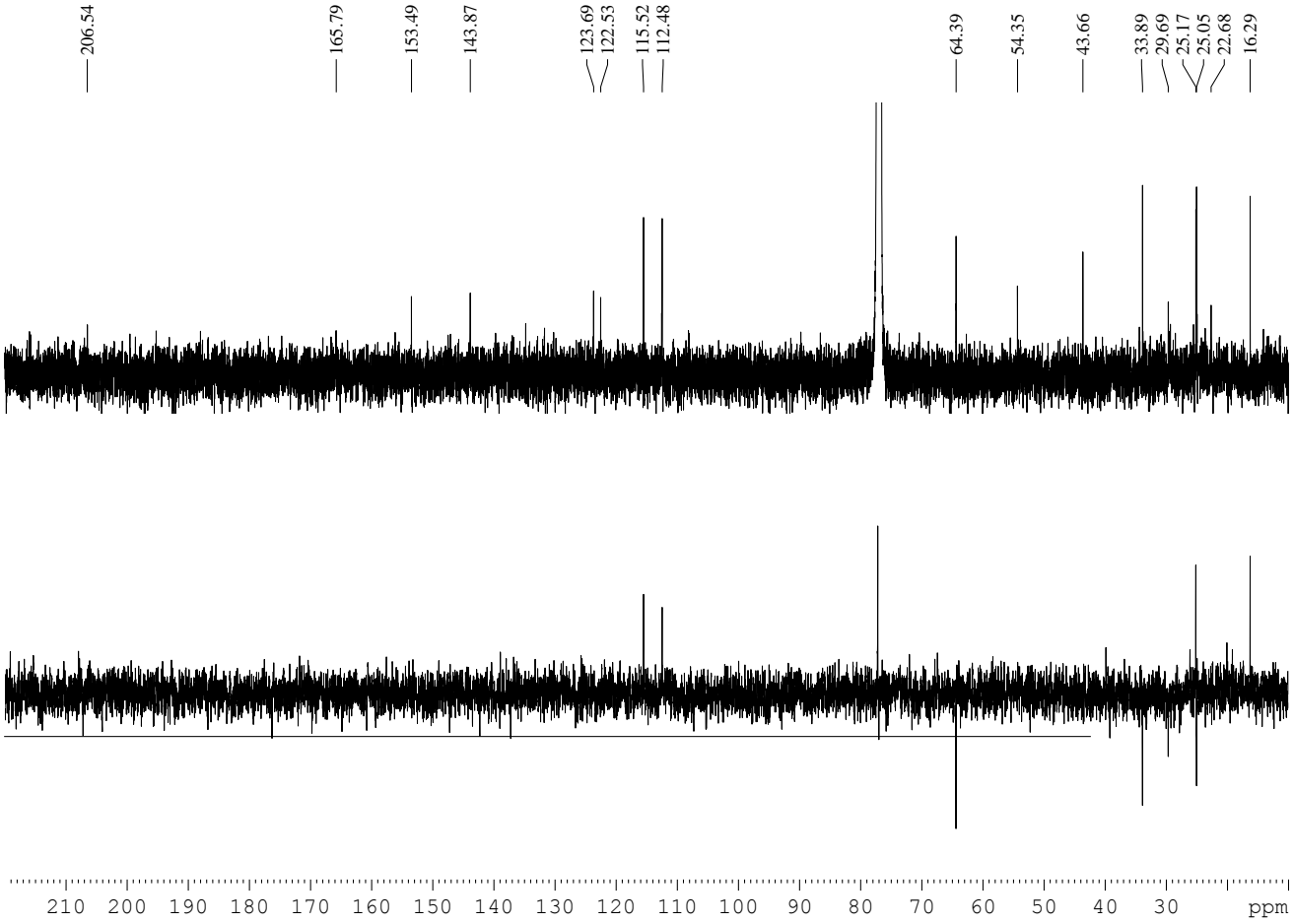
S 42 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-G (7).

FP-4-2+3-t4 CDCl₃ 104/05/21 AVIII400 (H1)



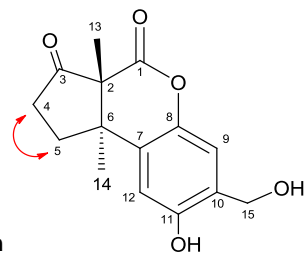
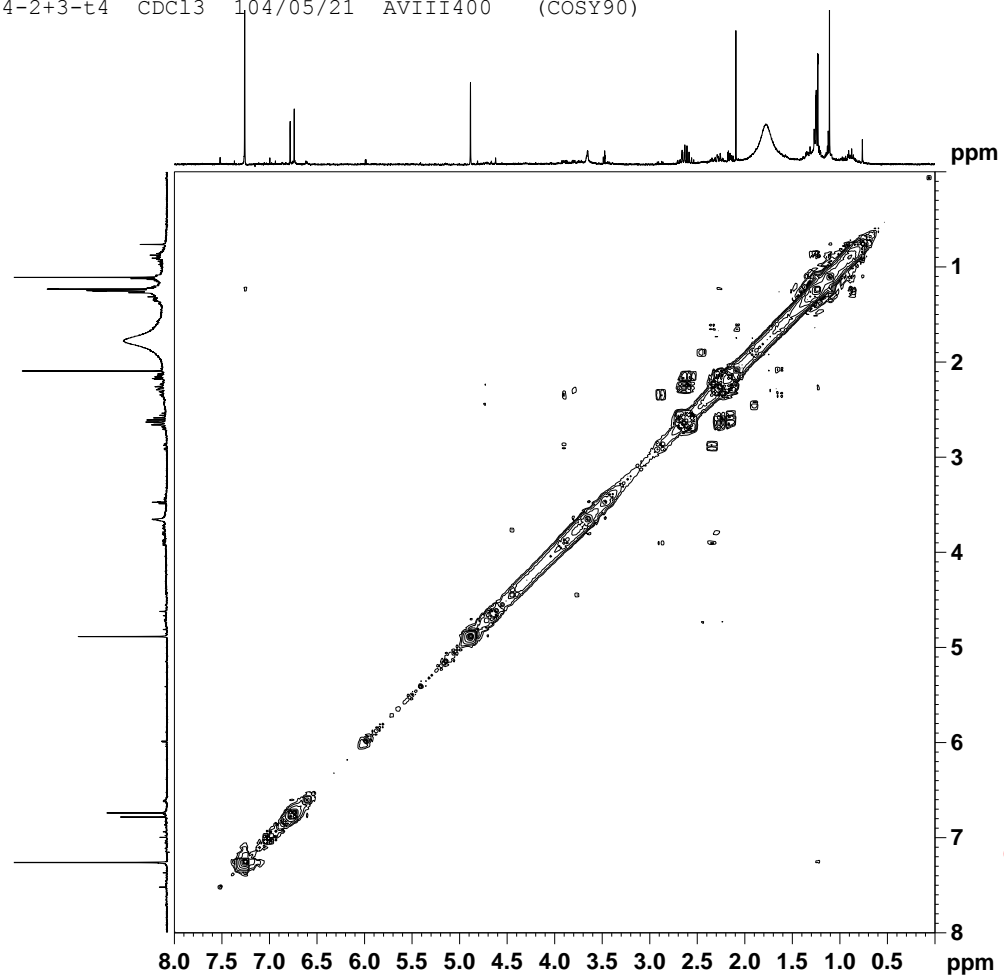
S 43 ¹³C NMR (100 MHz, CDCl₃) spectrum of fomitopin-G (7).

FP-4-2+3-t4 CDC13 104/05/21 AVIII400 (C13)



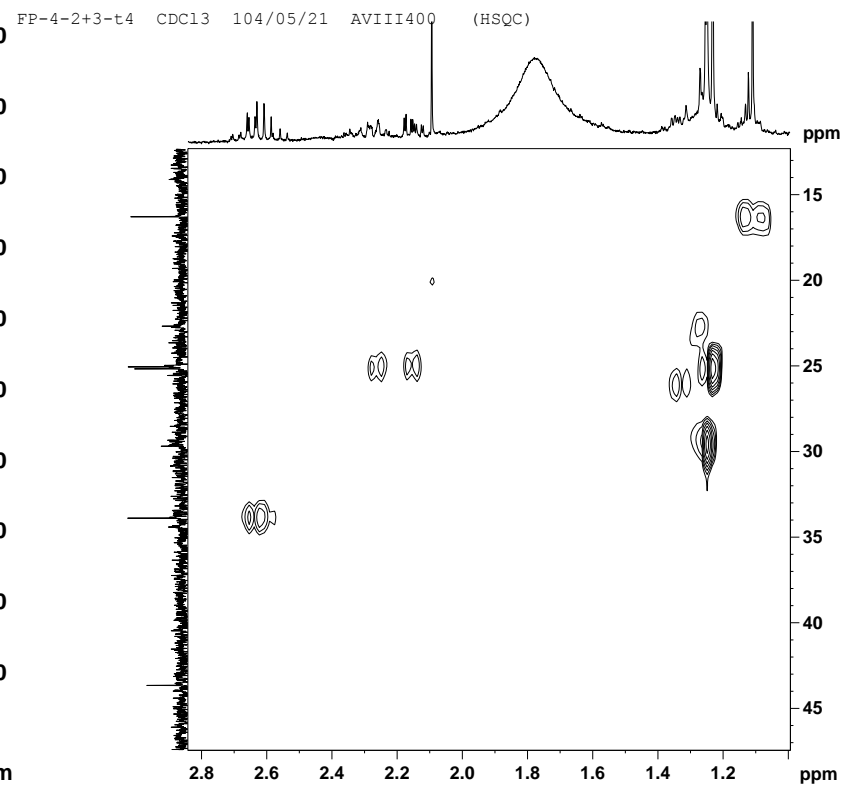
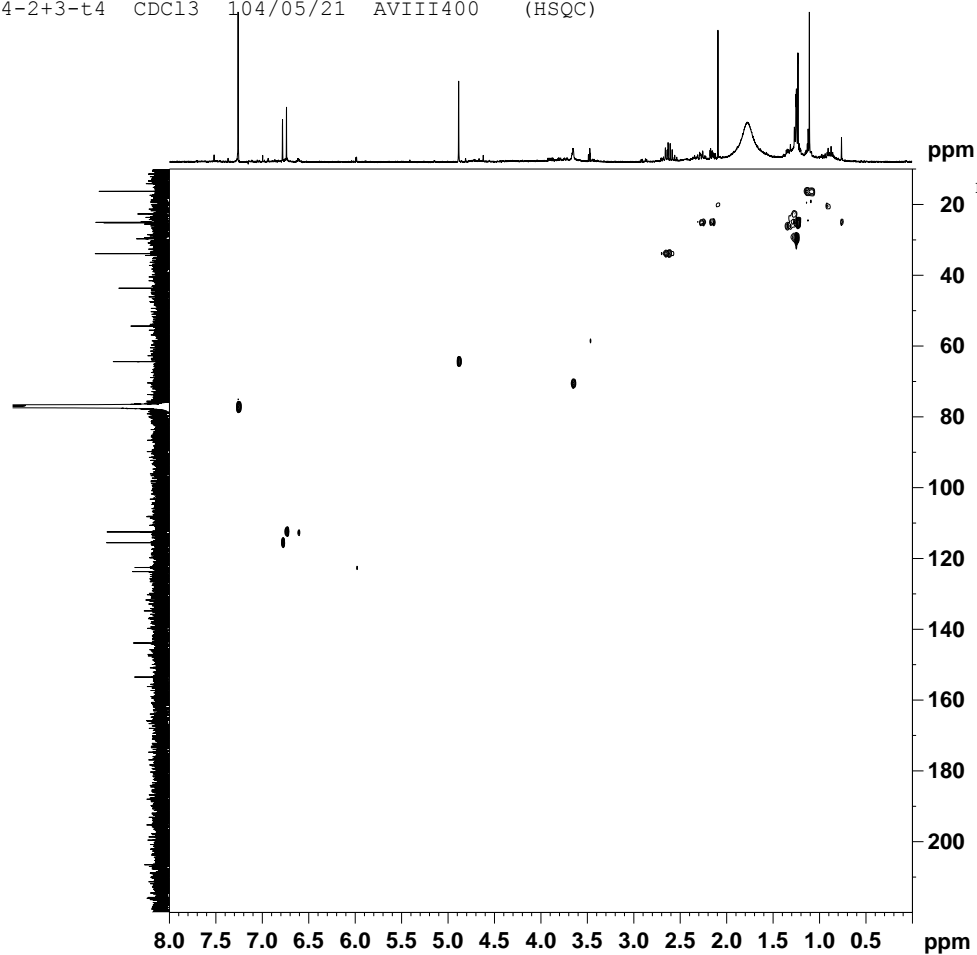
S 44 COSY spectrum of fomitopin-G (7).

FP-4-2+3-t4 CDC13 104/05/21 AVIII400 (COSY90)



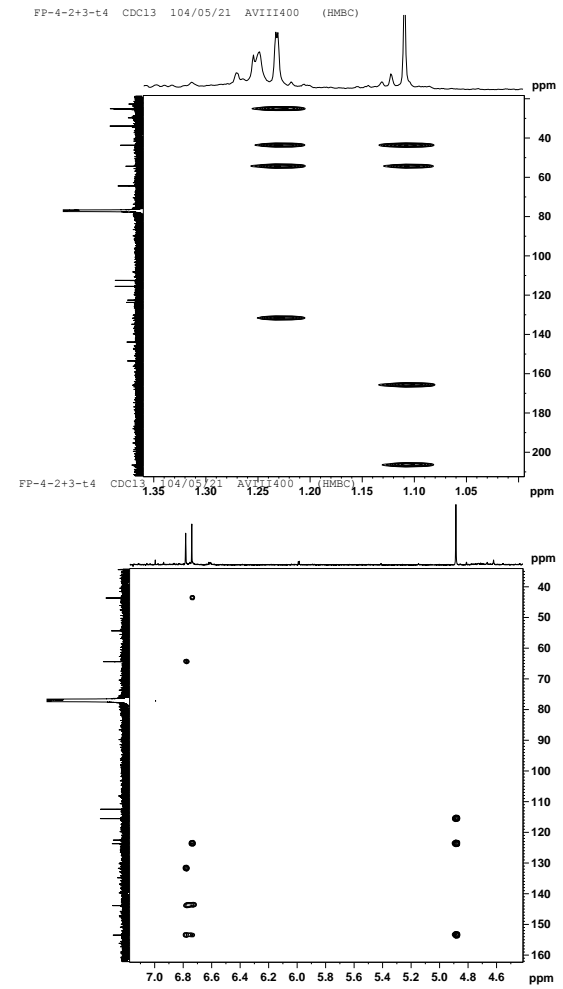
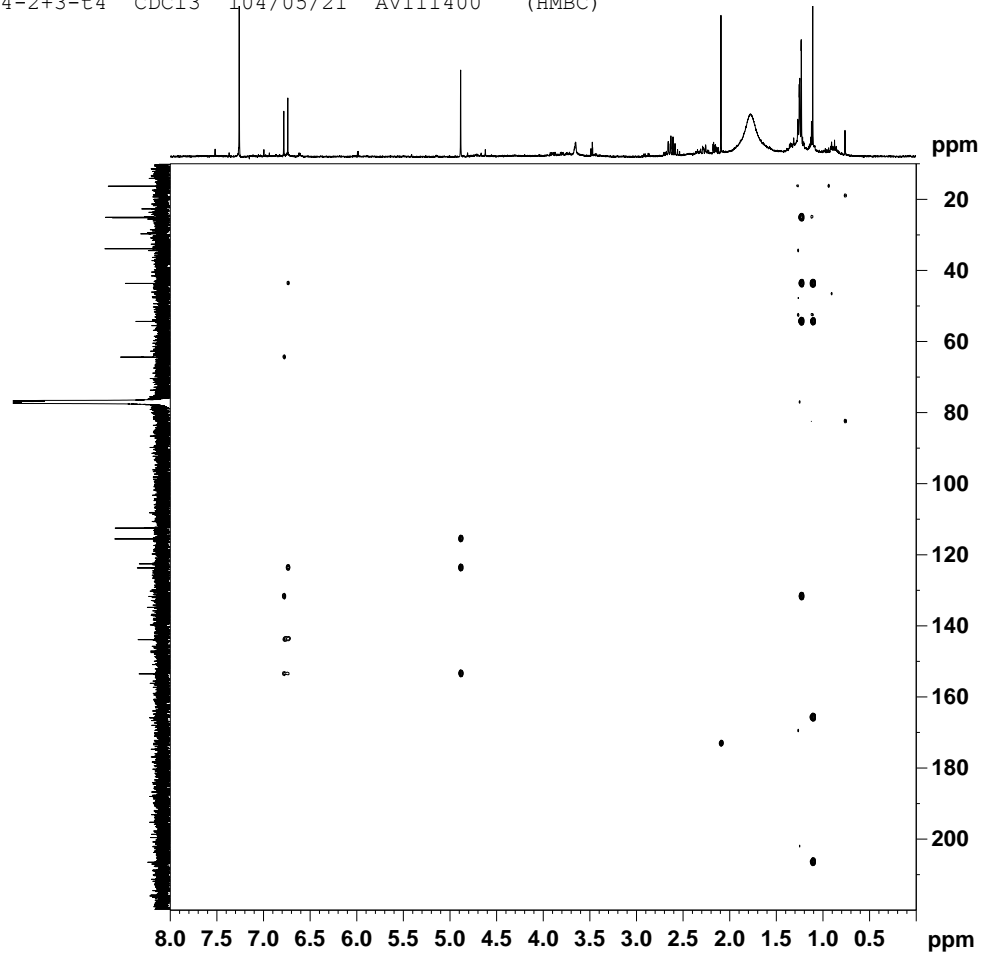
S 45 HSQC spectrum and key of HSQC spectrum of fomitopin-G (7).

FP-4-2+3-t4 CDCl3 104/05/21 AVIII400 (HSQC)

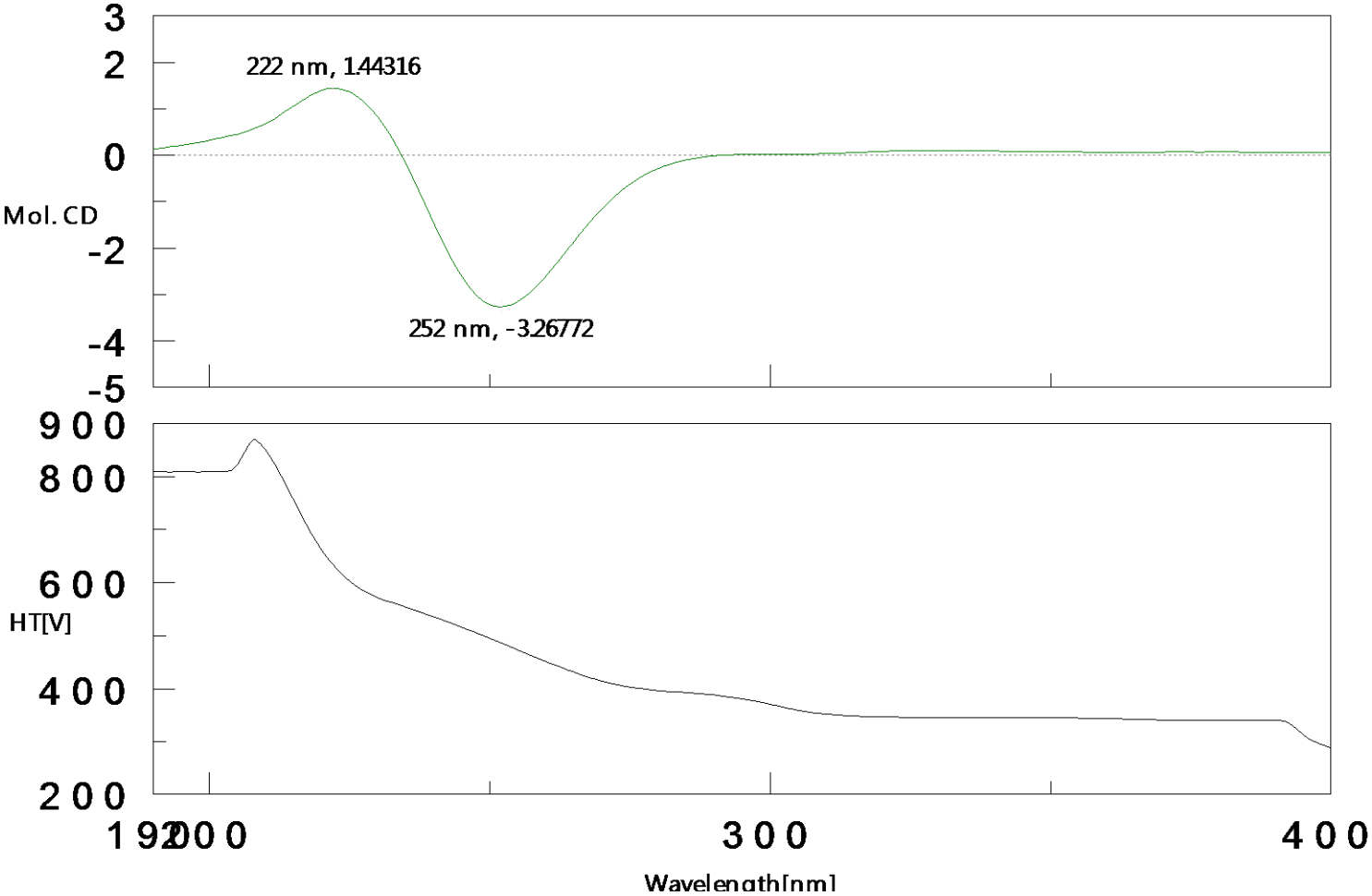


S 46 HMBC spectrum of fomitopin-G (7).

FP-4-2+3-t4 CDCl3 104/05/21 AVIII400 (HMBC)

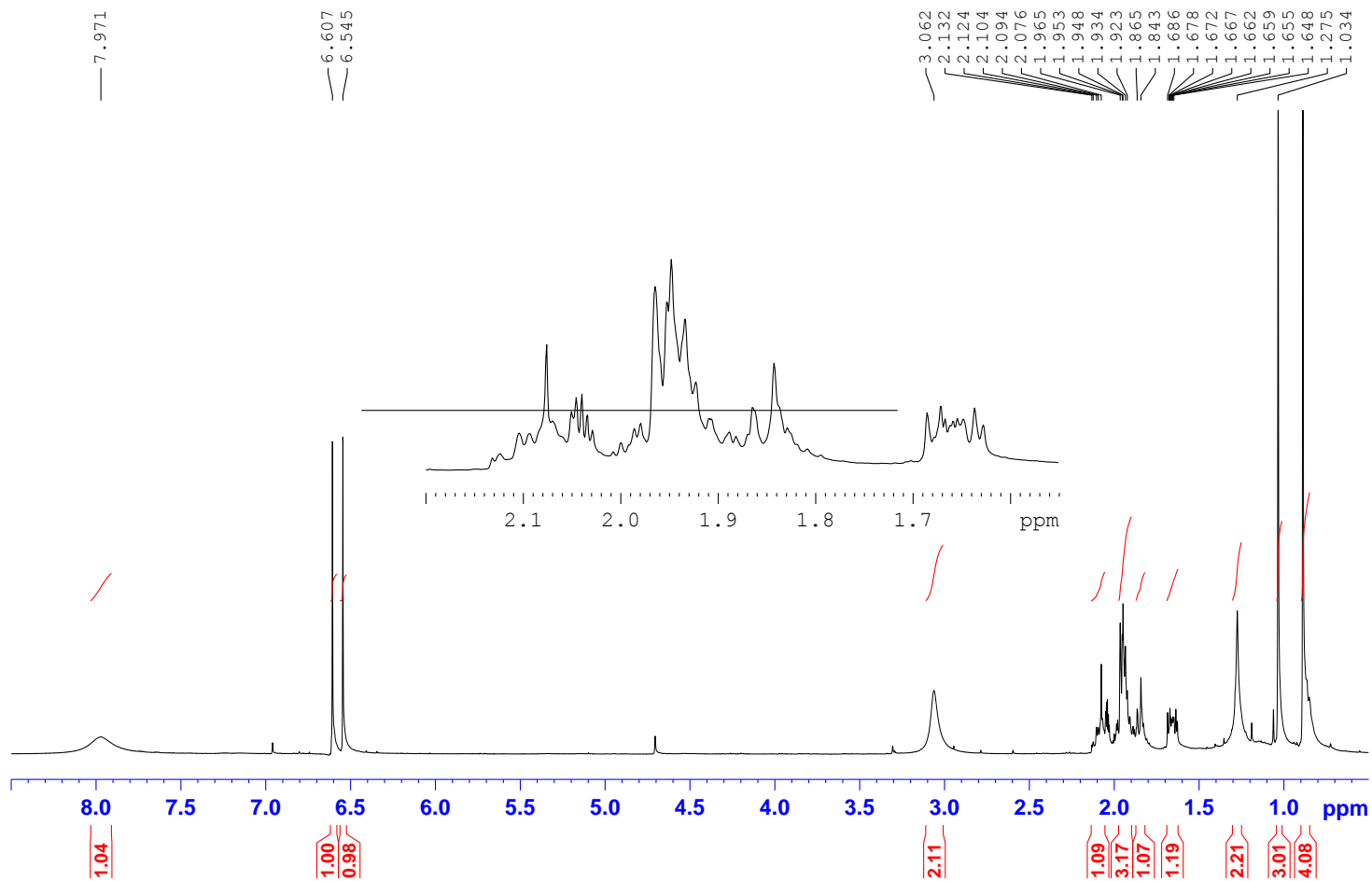


S 47 ECD spectrum of fomitopin-G (7).



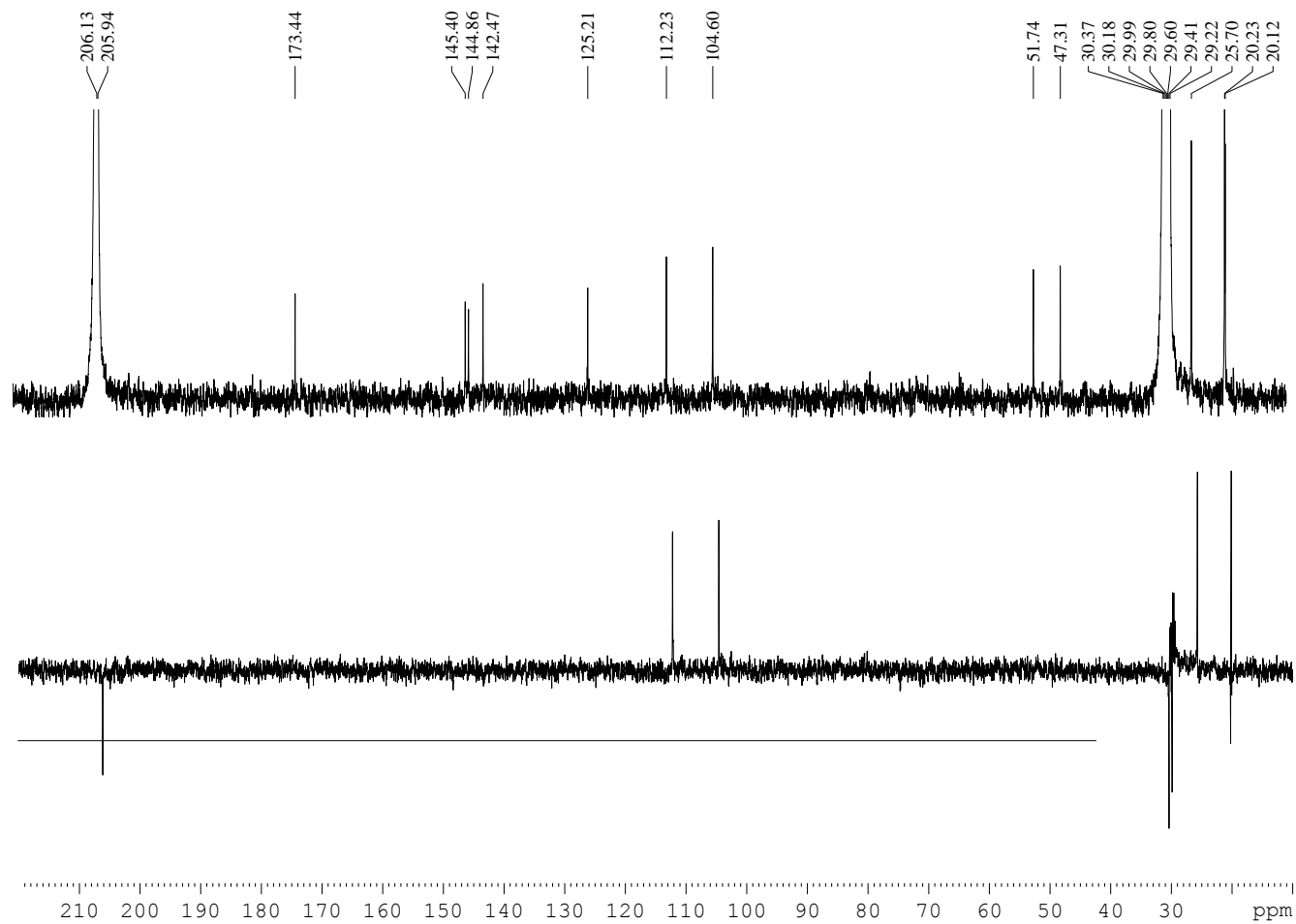
S 48 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-H (8).

FP-5-9-3-1 Acetone 102/10/23 AVIII400 (H1)



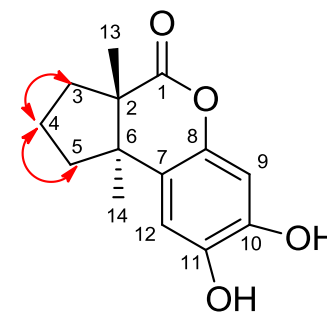
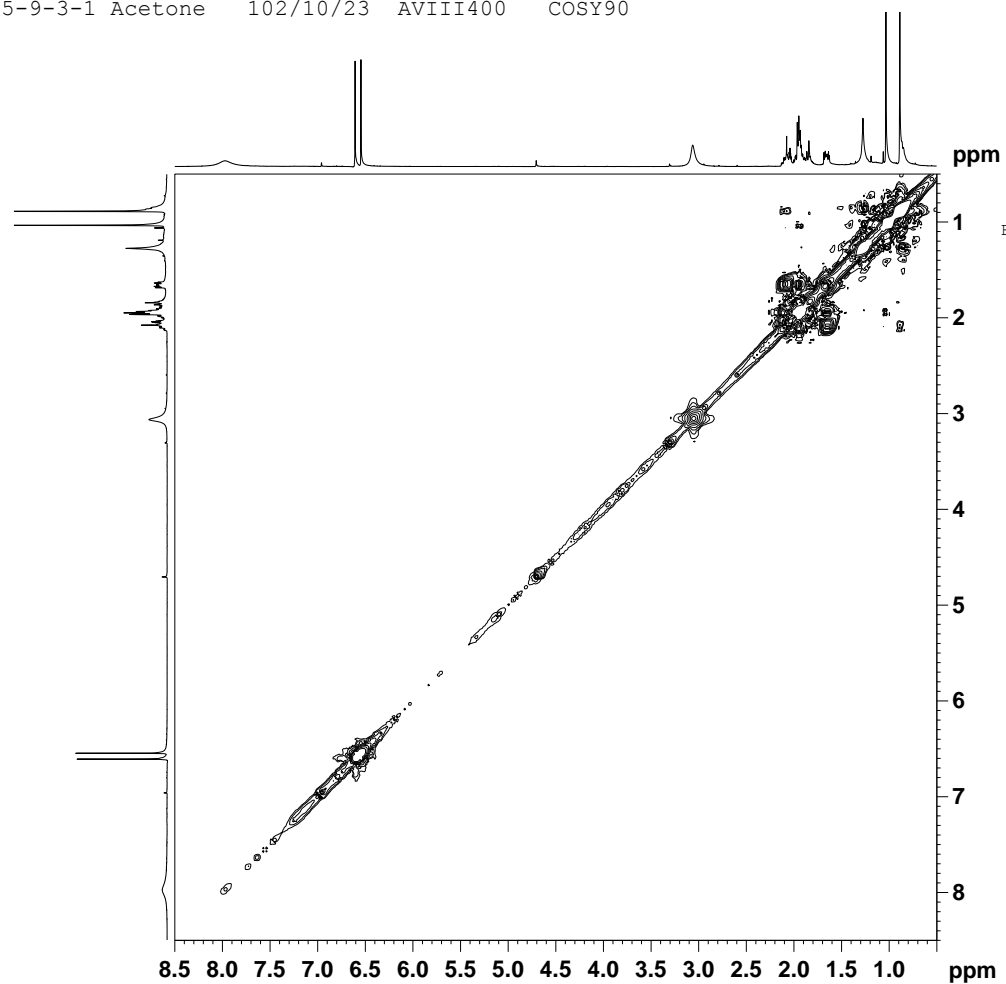
S 49 ¹³C NMR (100 MHz, CDCl₃) spectrum of fomitopin-H (8).

FP-5-9-3-1 Acetone-D6 102/10/9 AVIII400 (H1)

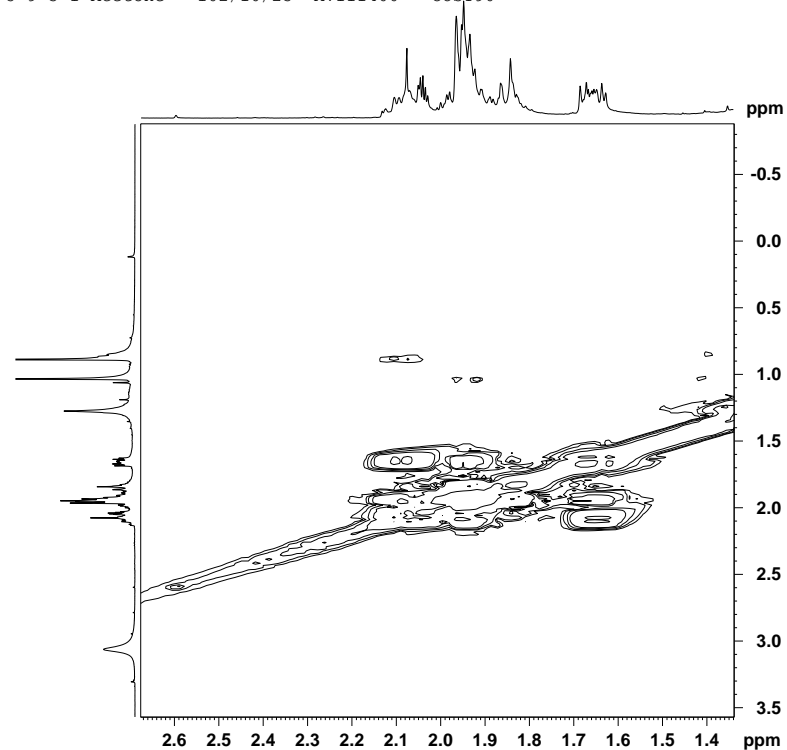


S 50 COSY spectrum of fomitopin-H (8).

FP-5-9-3-1 Acetone 102/10/23 AVIII400 COSY90

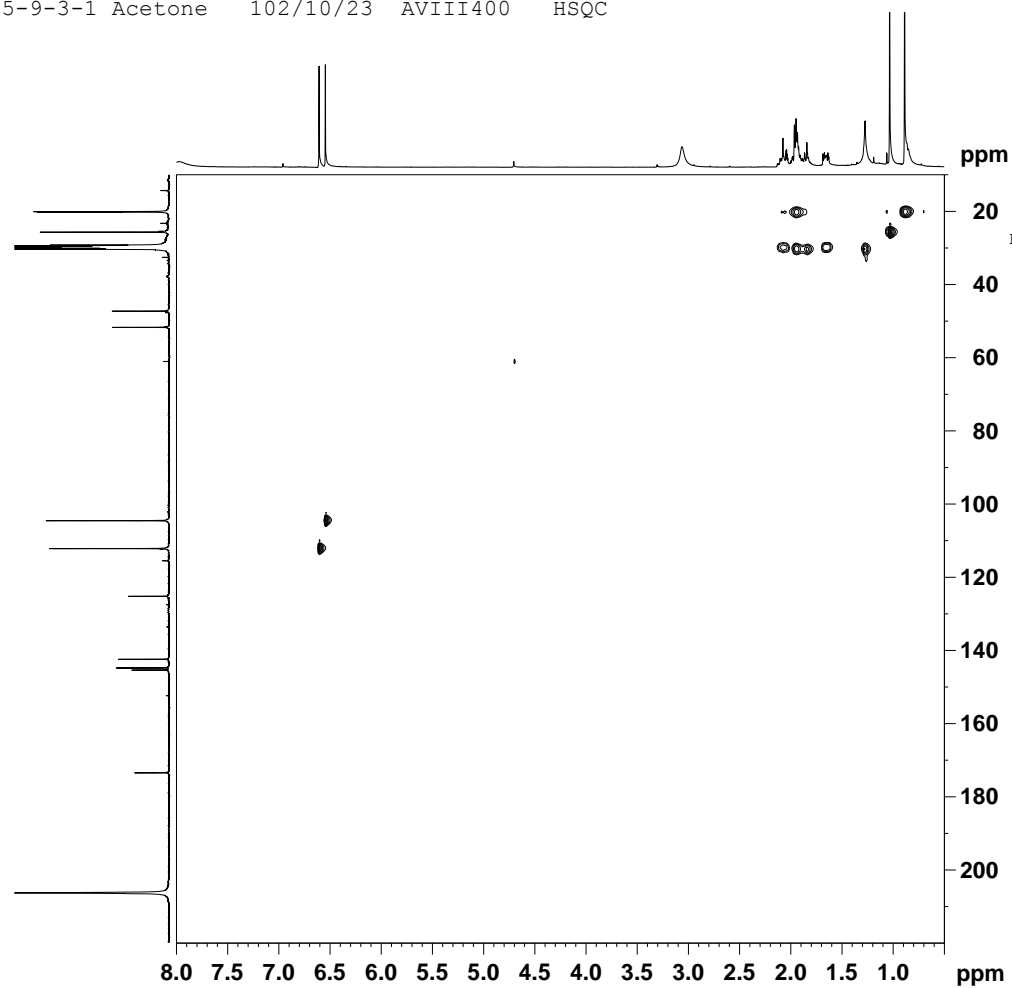


FP-5-9-3-1 Acetone 102/10/23 AVIII400 COSY90

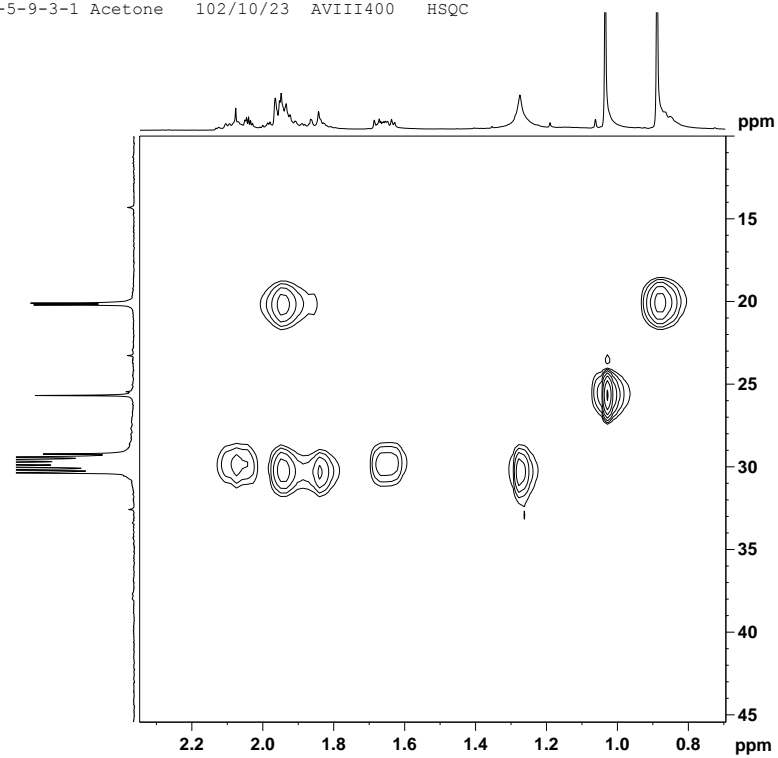


S 51 HSQC spectrum and key of HSQC spectrum of fomitopin-H (8).

FP-5-9-3-1 Acetone 102/10/23 AVIII400 HSQC

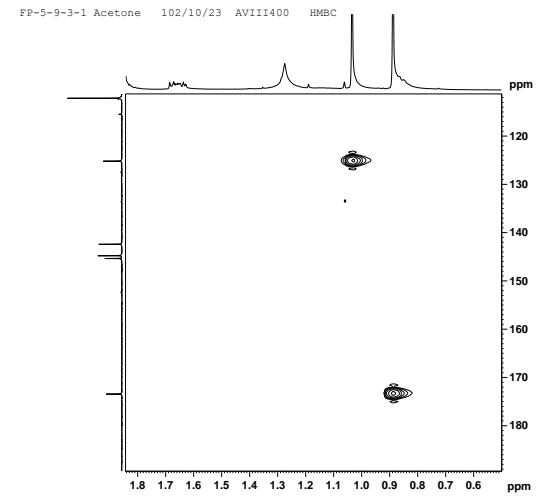
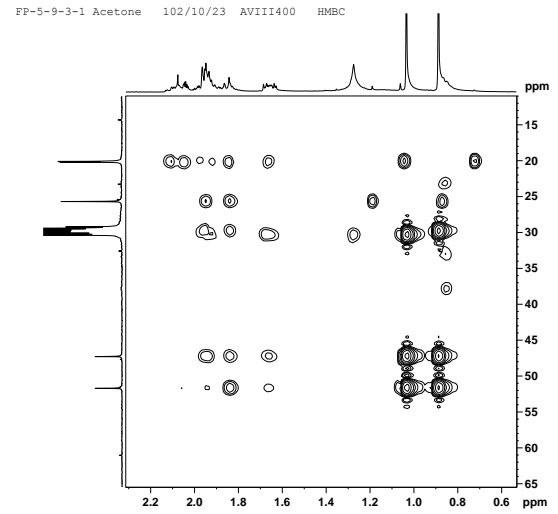
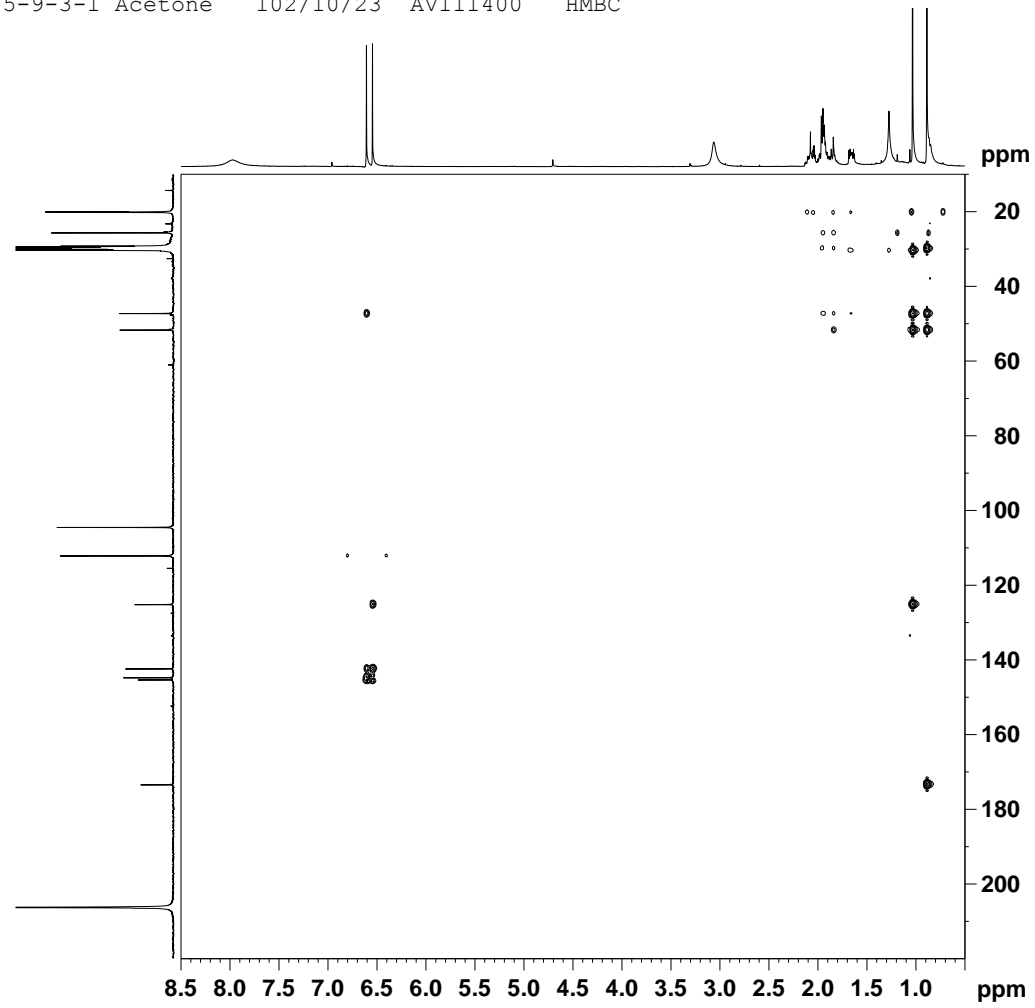


FP-5-9-3-1 Acetone 102/10/23 AVIII400 HSQC

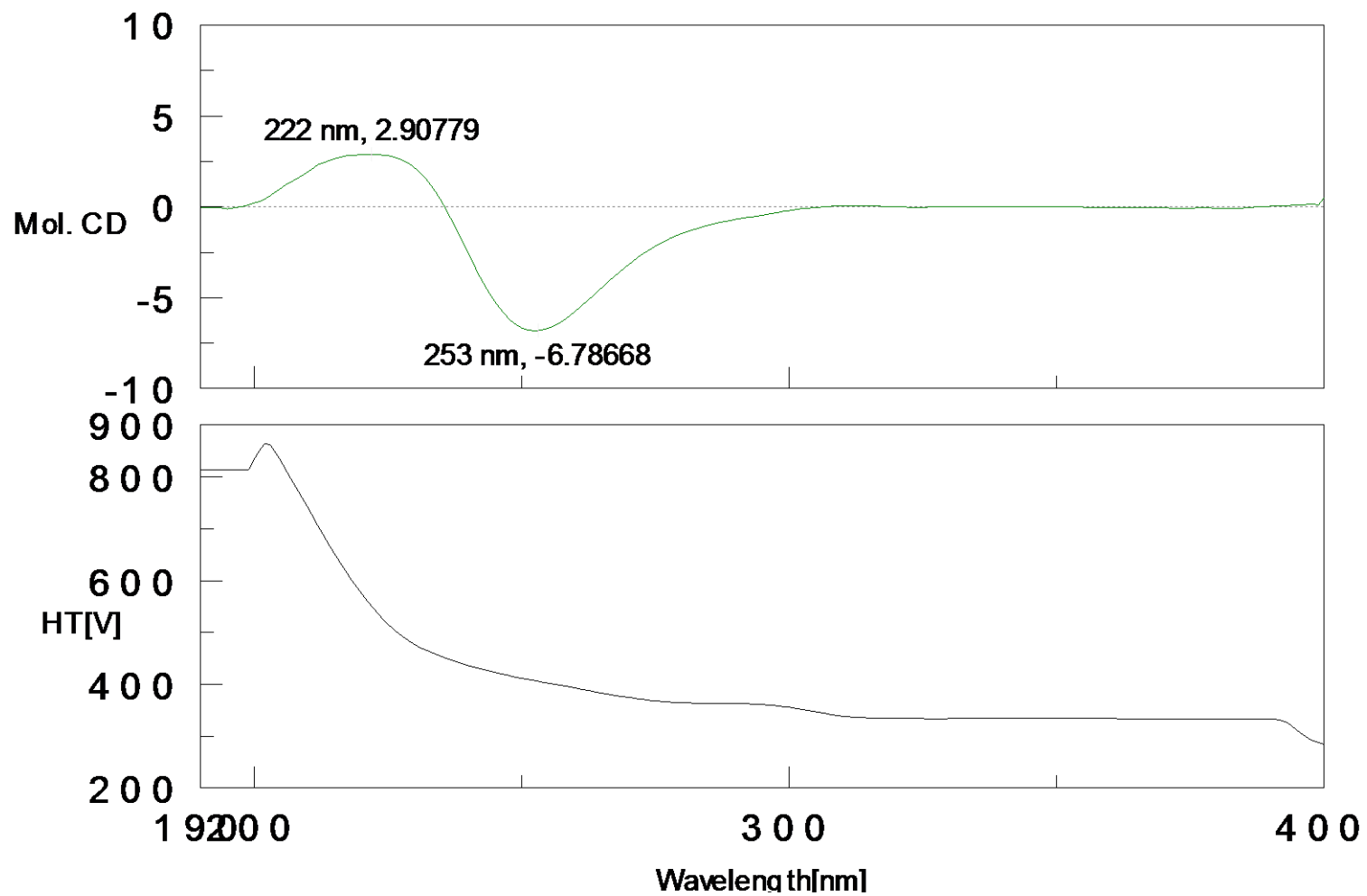


S 52 HMBC spectrum of fomitopin-H (8).

FP-5-9-3-1 Acetone 102/10/23 AVIII400 HMBC

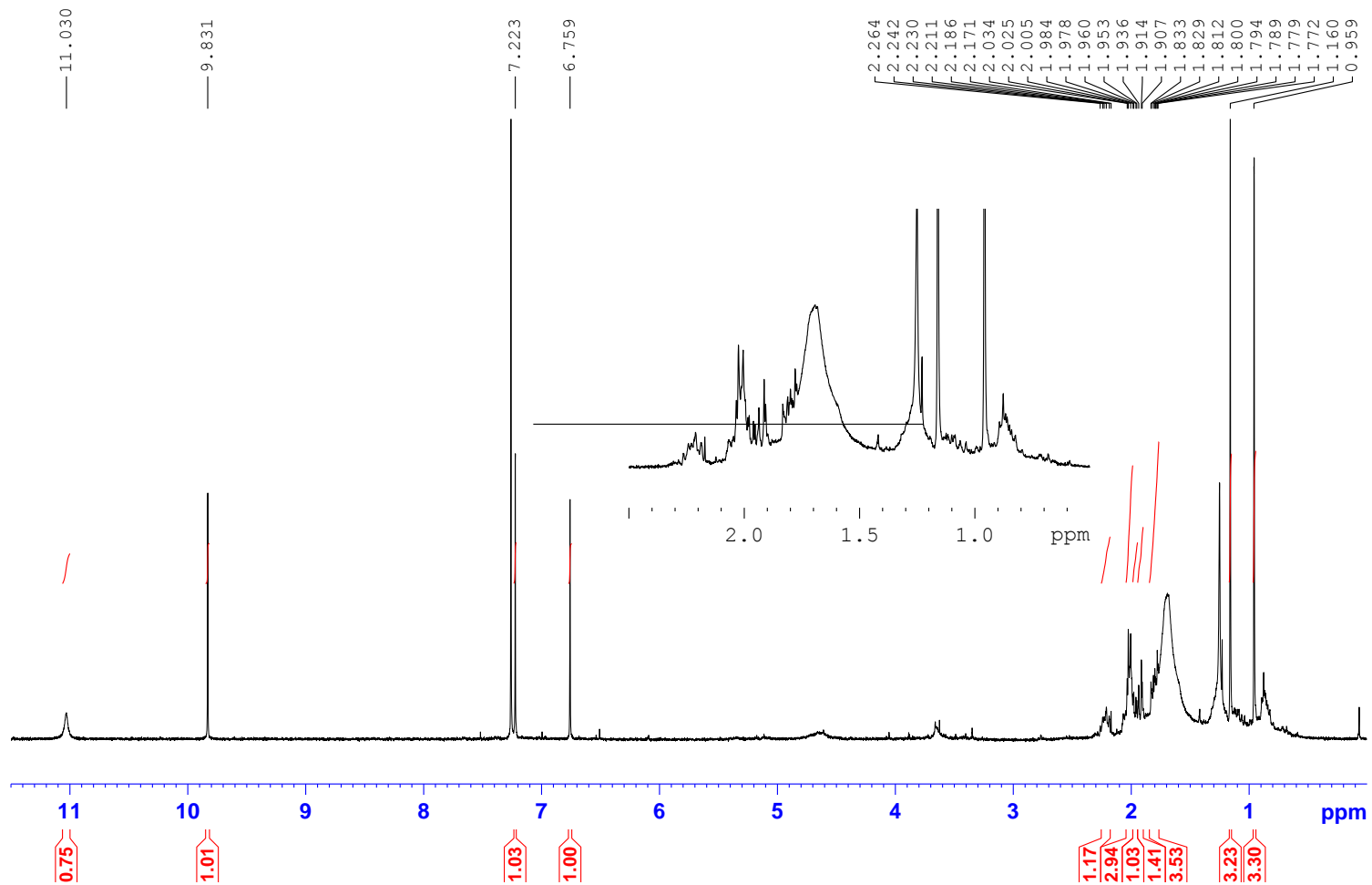


S 53 ECD spectrum of fomitopin-H (8).



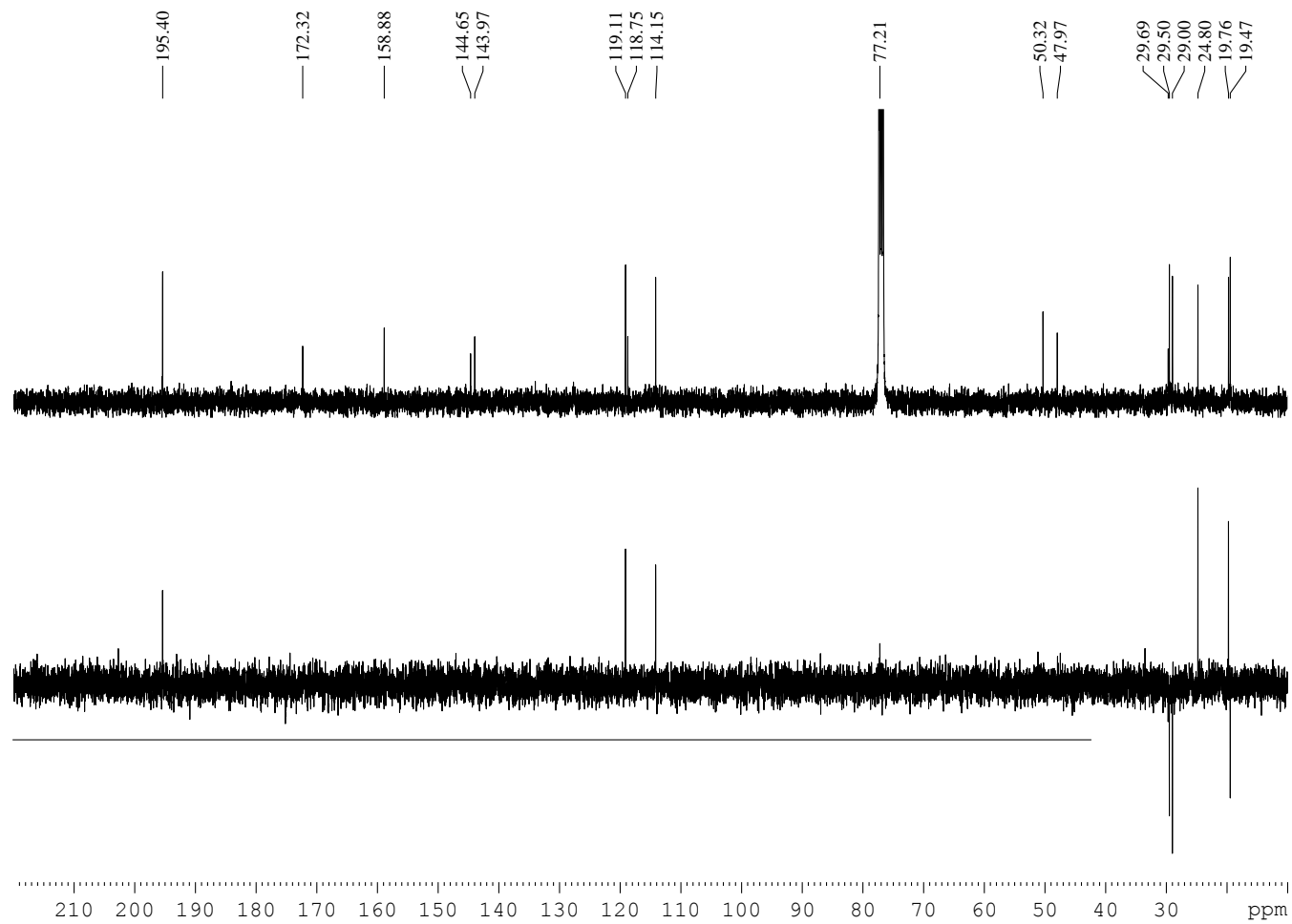
S 54 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-I (9).

FP-6252-t5 CDCl₃ 103/08/02 AVIII400 (H1)



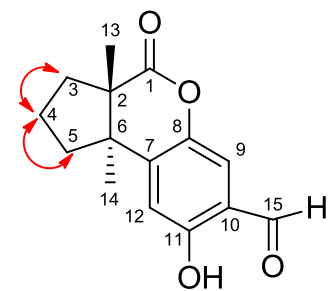
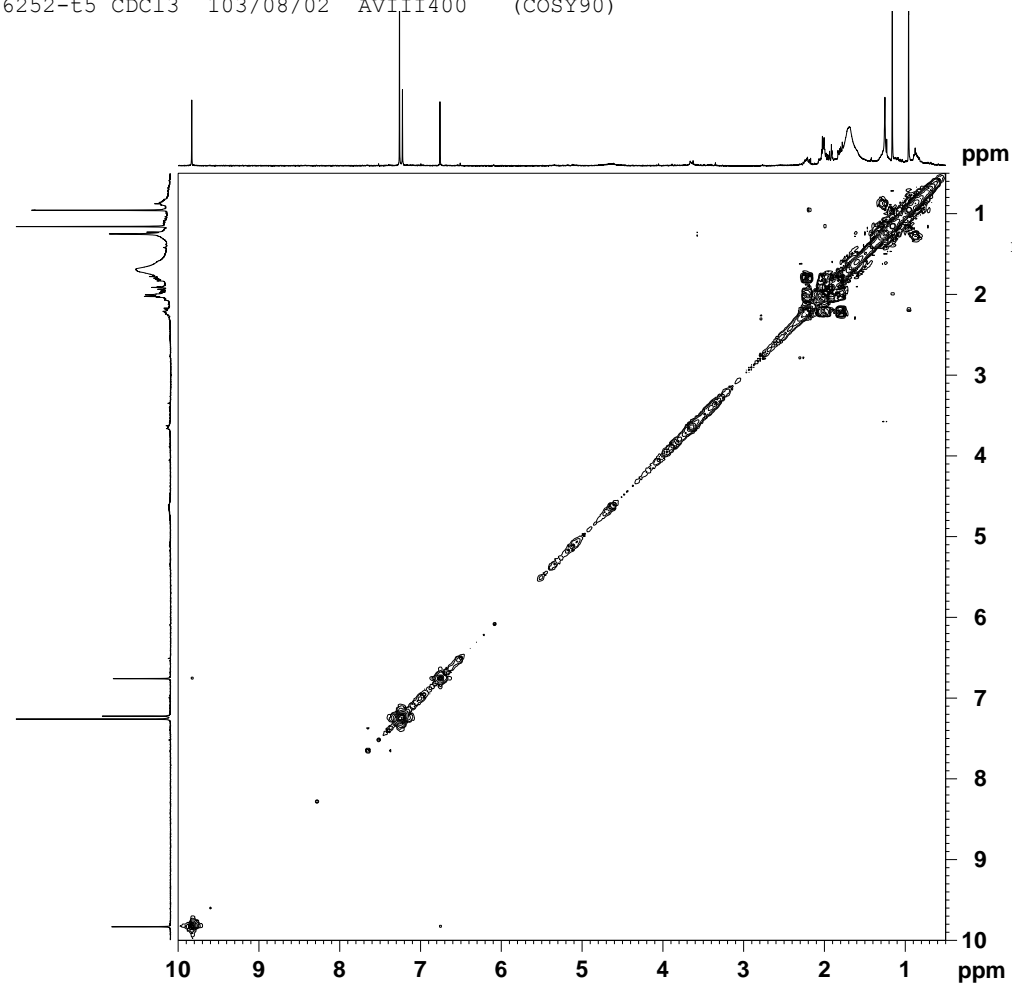
S 55 ¹³C NMR (100 MHz, CDCl₃) spectrum of fomitopin-I (9).

FP-6252-t5 CDCl₃ 103/08/02 AVIII400 (DEPT135)

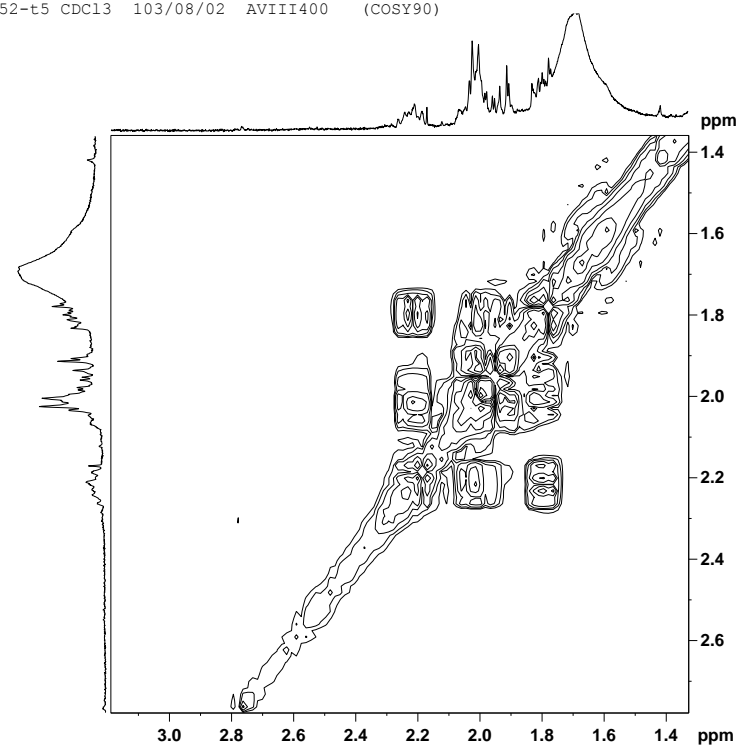


S 56 COSY spectrum of fomitopin-I (9).

FP-6252-t5 CDC13 103/08/02 AVIII400 (COSY90)

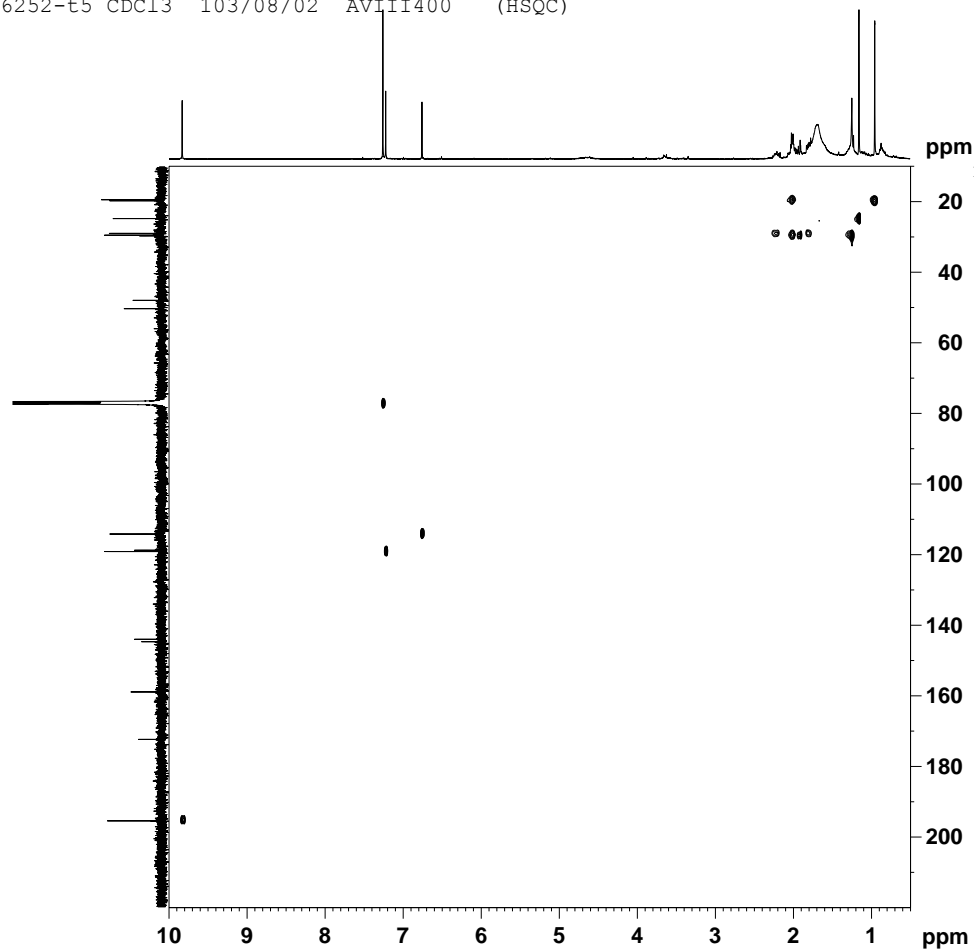


FP-6252-t5 CDC13 103/08/02 AVIII400 (COSY90)

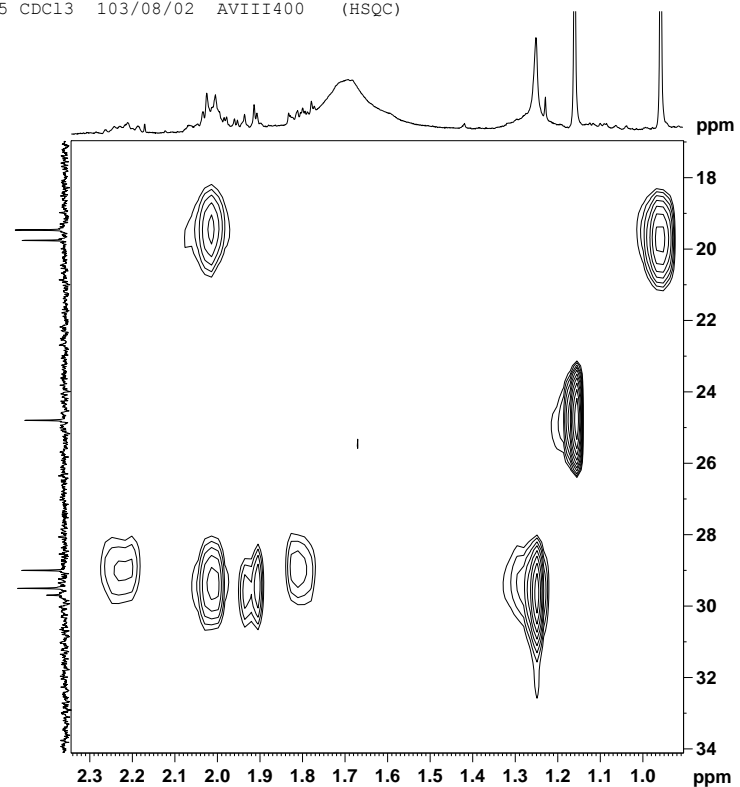


S 57 HSQC spectrum and key of HSQC spectrum of fomitopin-I (9).

FP-6252-t5 CDC13 103/08/02 AVIII400 (HSQC)

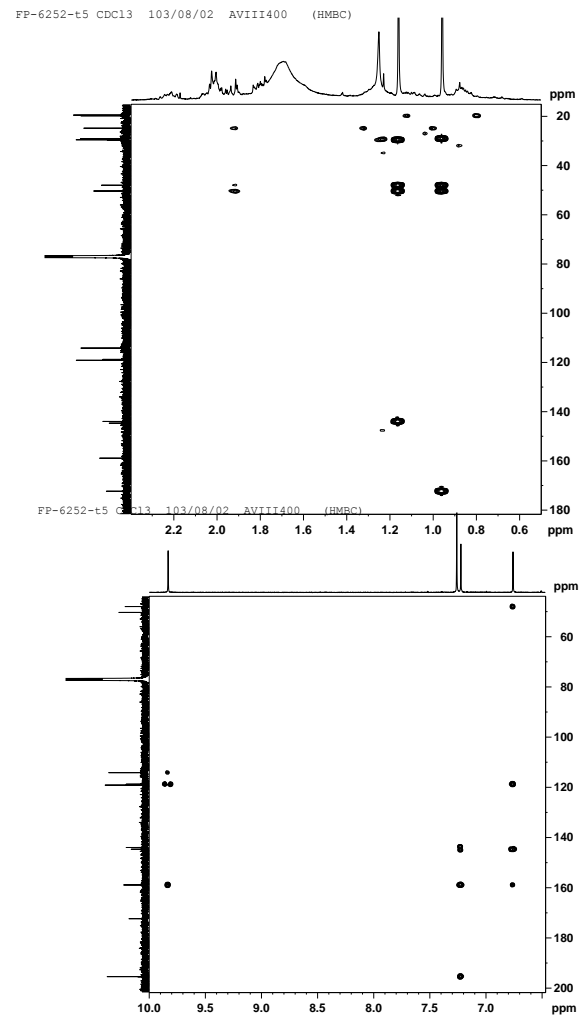
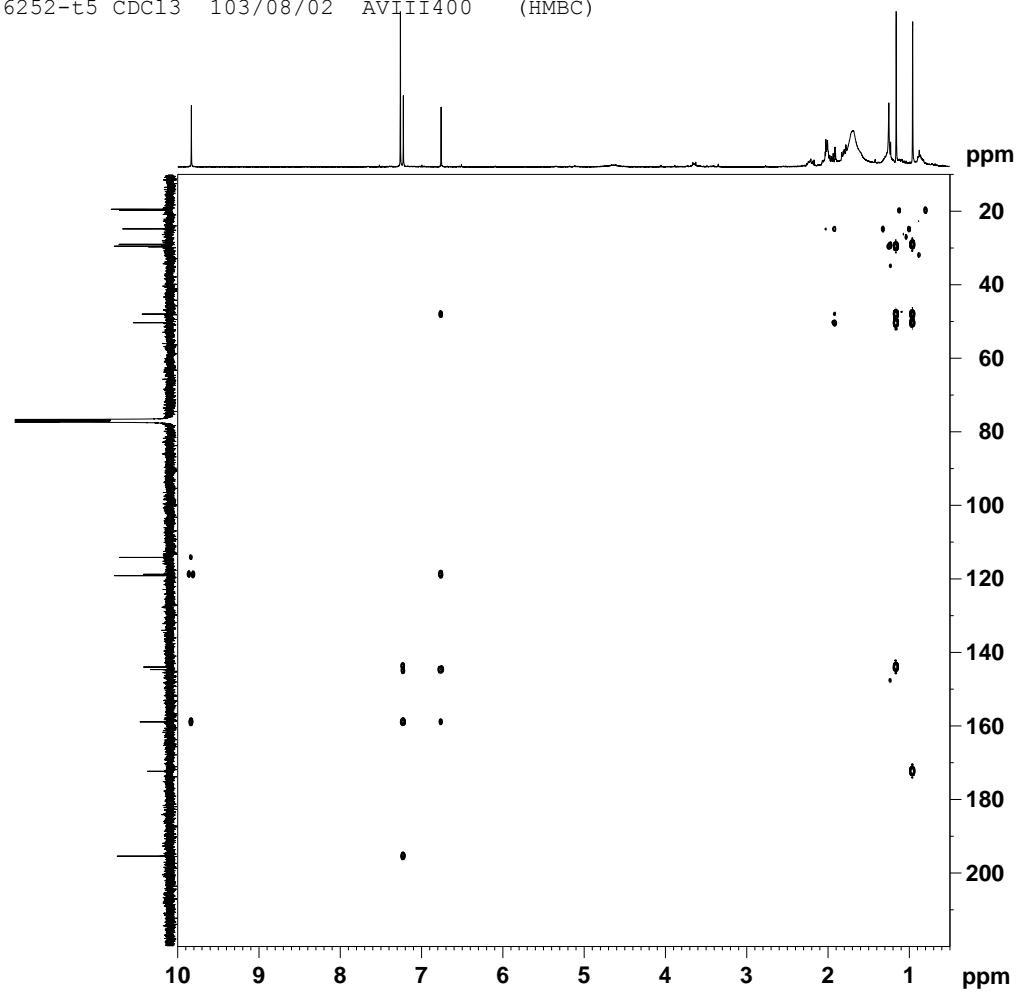


FP-6252-t5 CDC13 103/08/02 AVIII400 (HSQC)

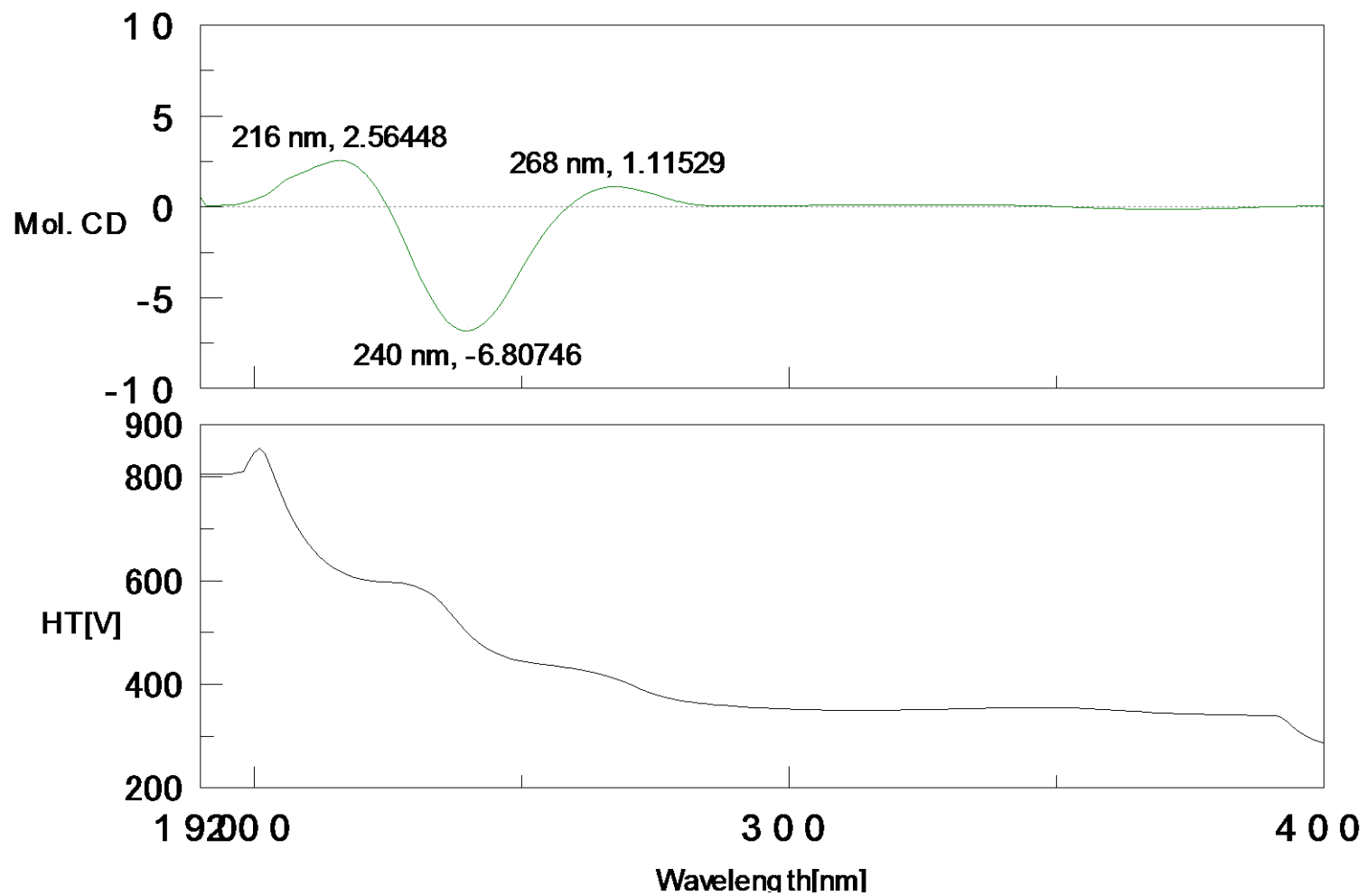


S 58 HMBC spectrum of fomitopin-I (9).

FP-6252-t5 CDC13 103/08/02 AVIII400 (HMBC)

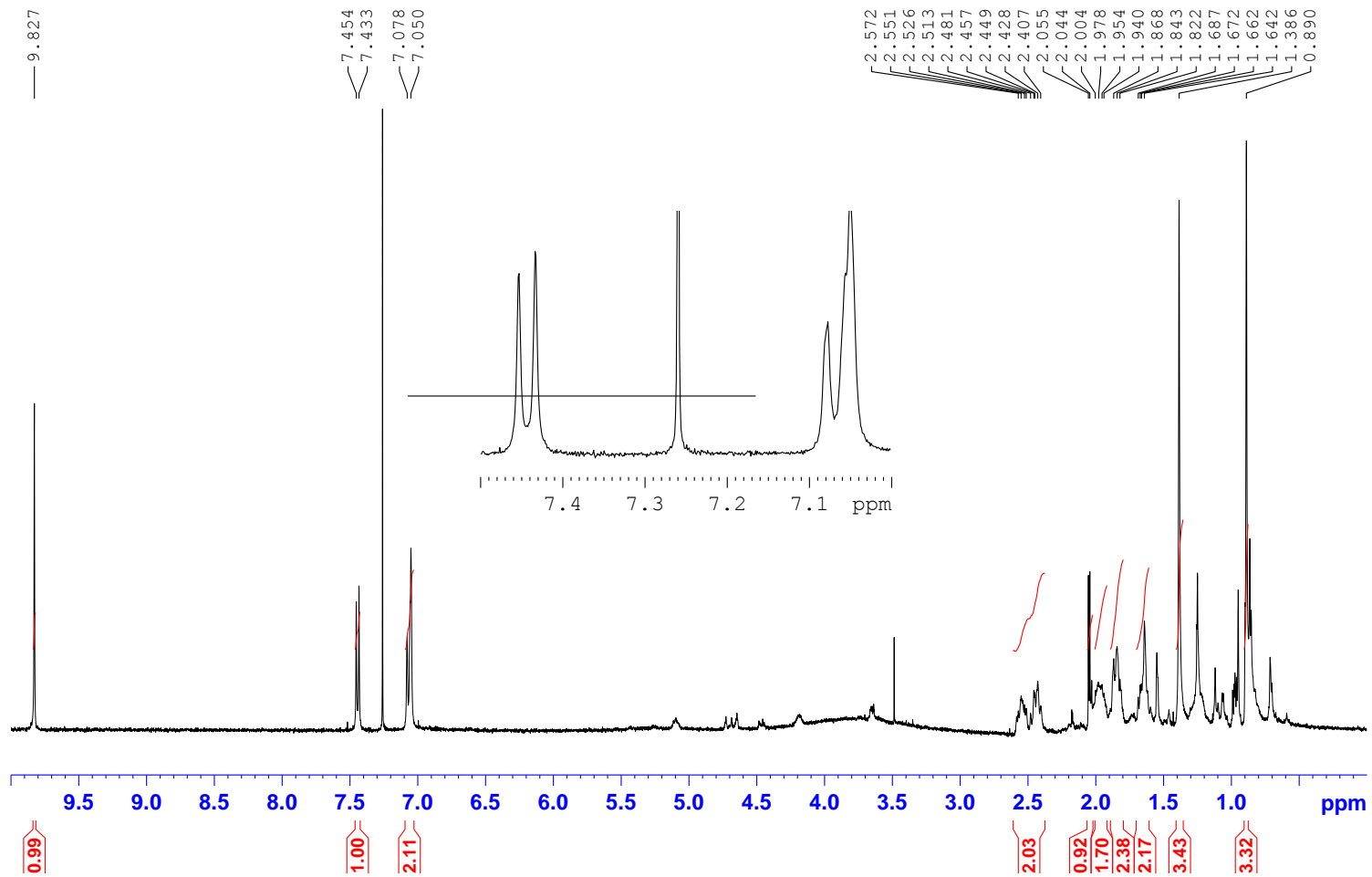


S 59 ECD spectrum of fomitopin-I (9).



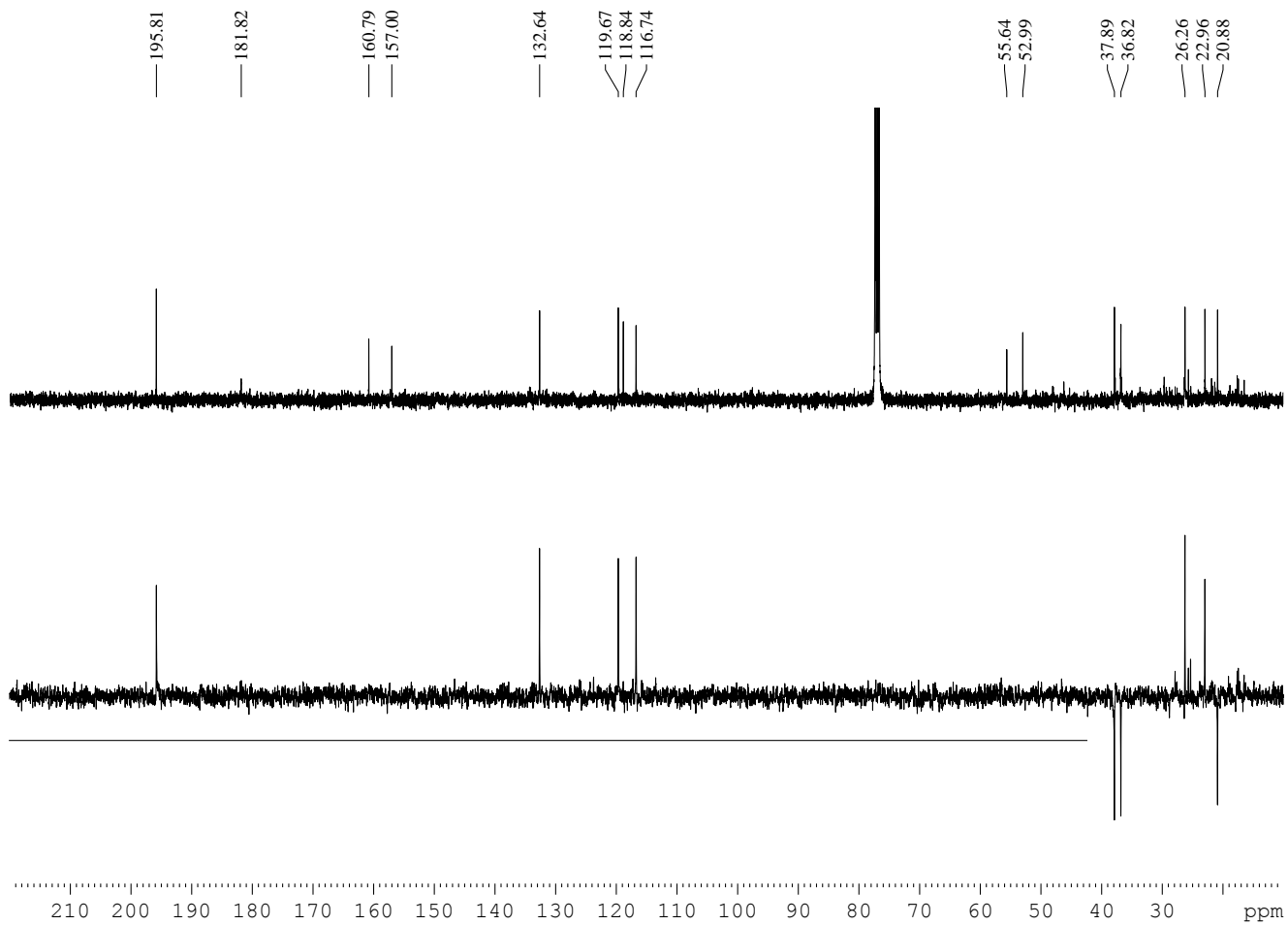
S 60 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-J (10).

FP-62-10-5-5-t1 CDCl₃ 103/09/20 AVIII400 (H1)



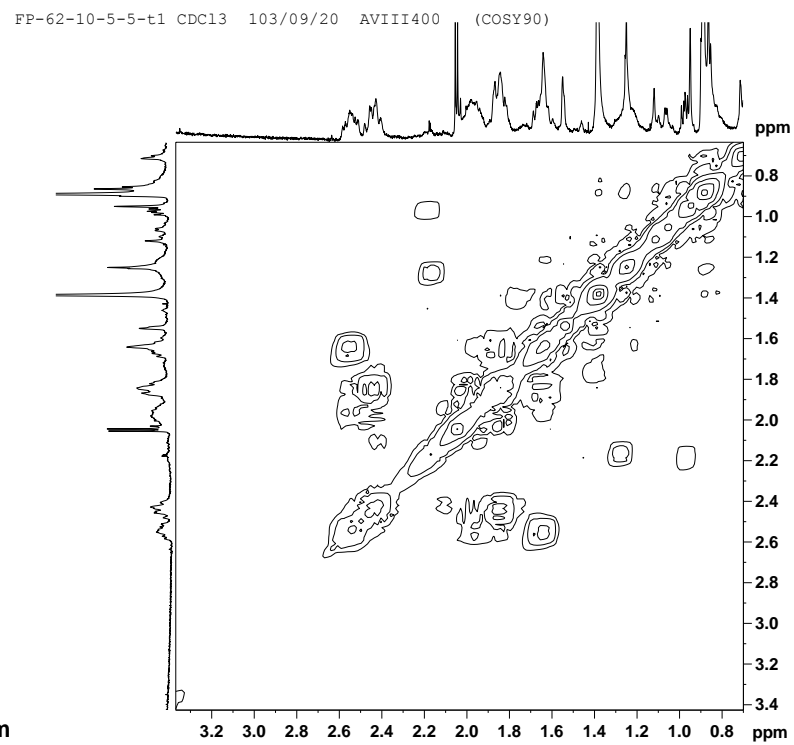
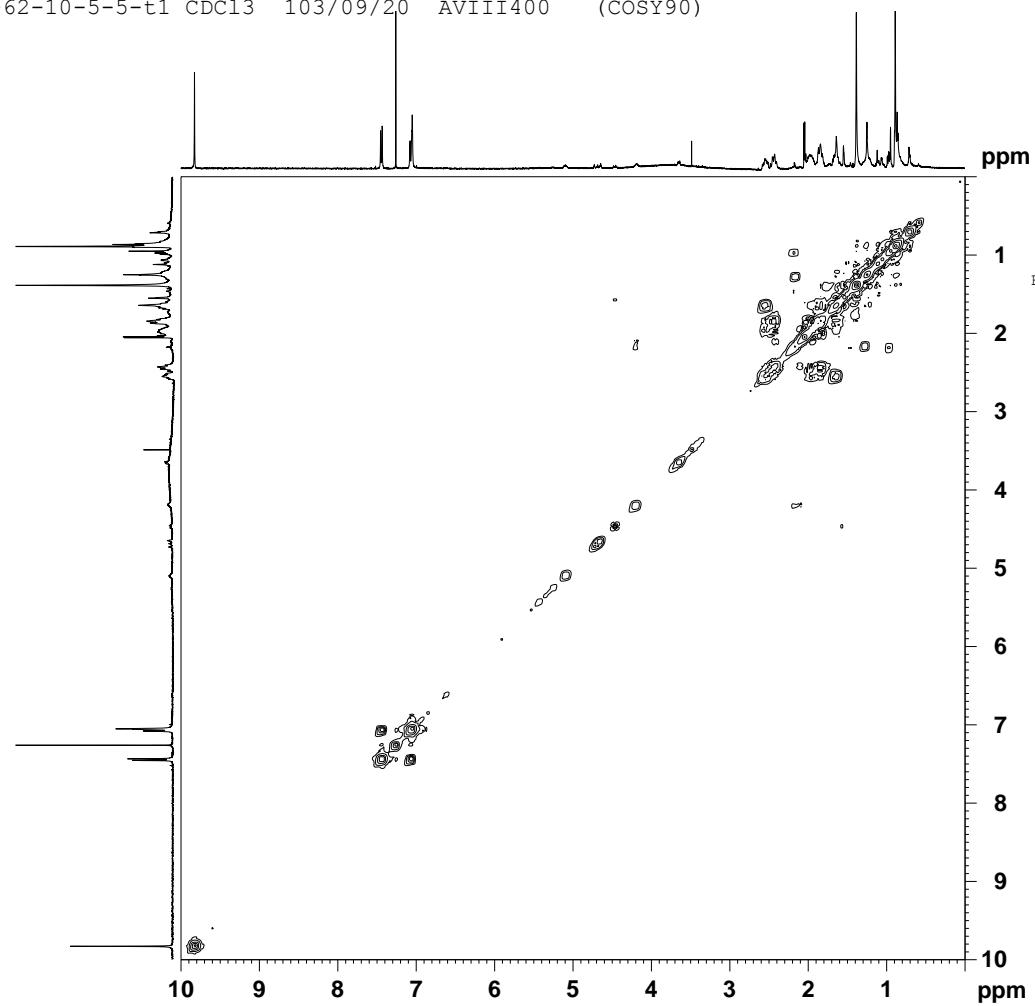
S 61 ¹³C NMR (100 MHz, CDCl₃) spectrum of fomitopin-J (10).

FP-62-10-5-5-t1 CDCl₃ 103/09/20 AVIII400 (DEPT135)



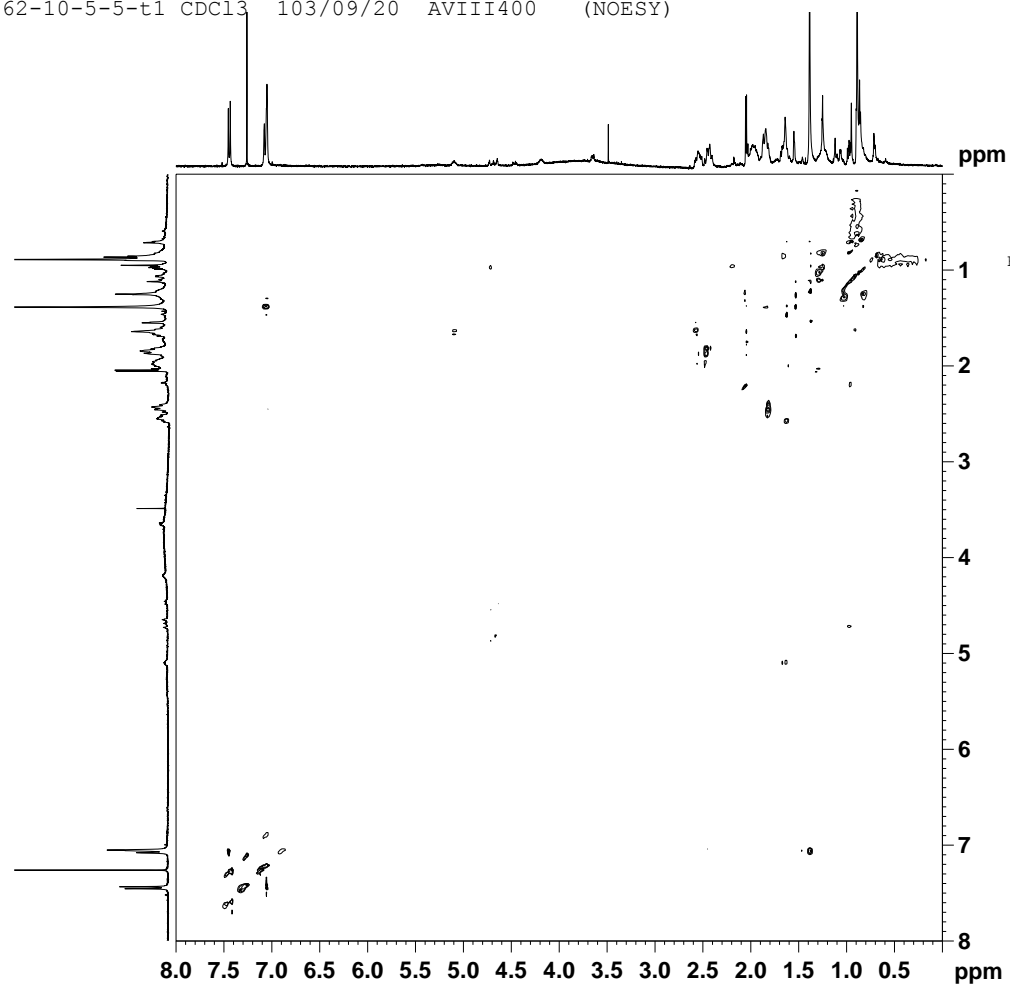
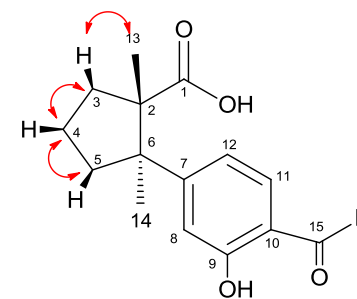
S 62 COSY spectrum of fomitopin-J (10).

FP-62-10-5-5-t1 CDC13 103/09/20 AVIII400 (COSY90)

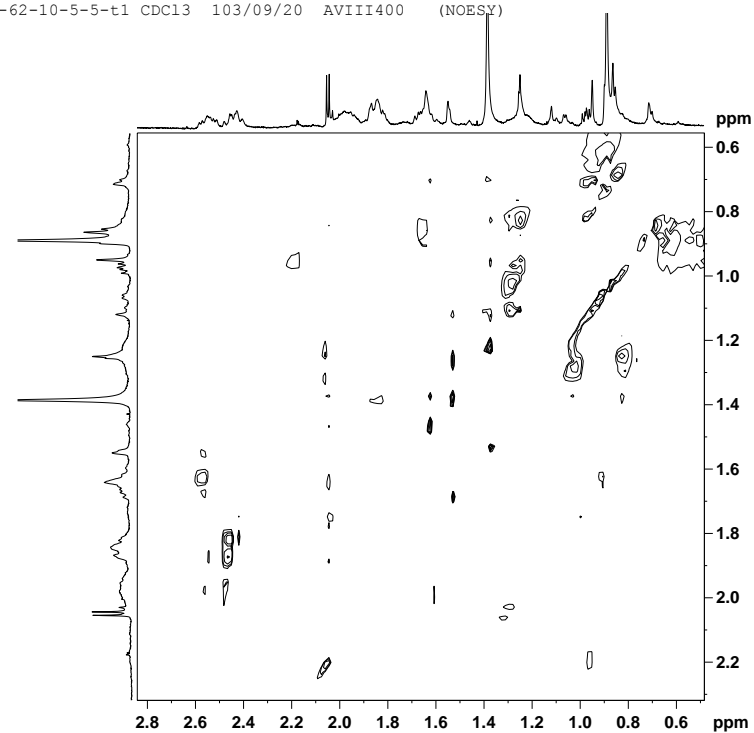


S 63 NOESY spectrum and key of NOESY spectrum of fomitopin-J (10).

FP-62-10-5-5-t1 CDC13 103/09/20 AVIII400 (NOESY)

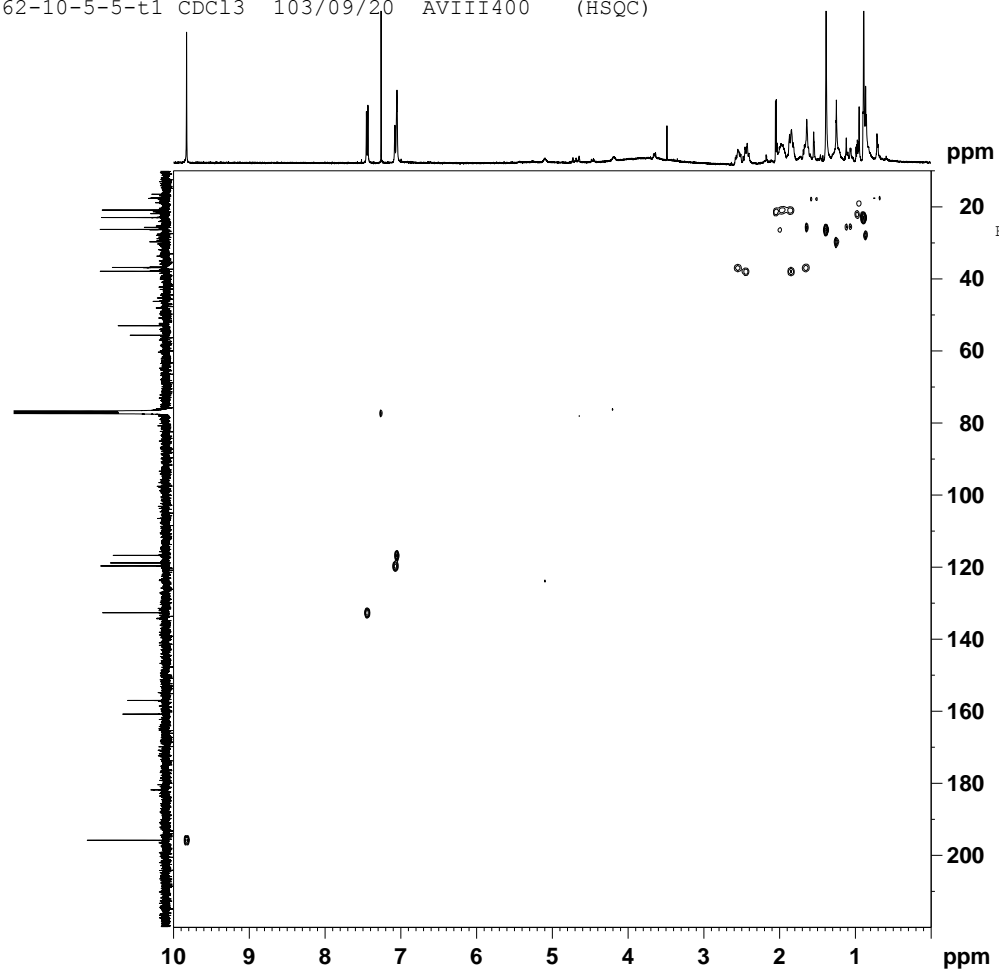


FP-62-10-5-5-t1 CDC13 103/09/20 AVIII400 (NOESY)

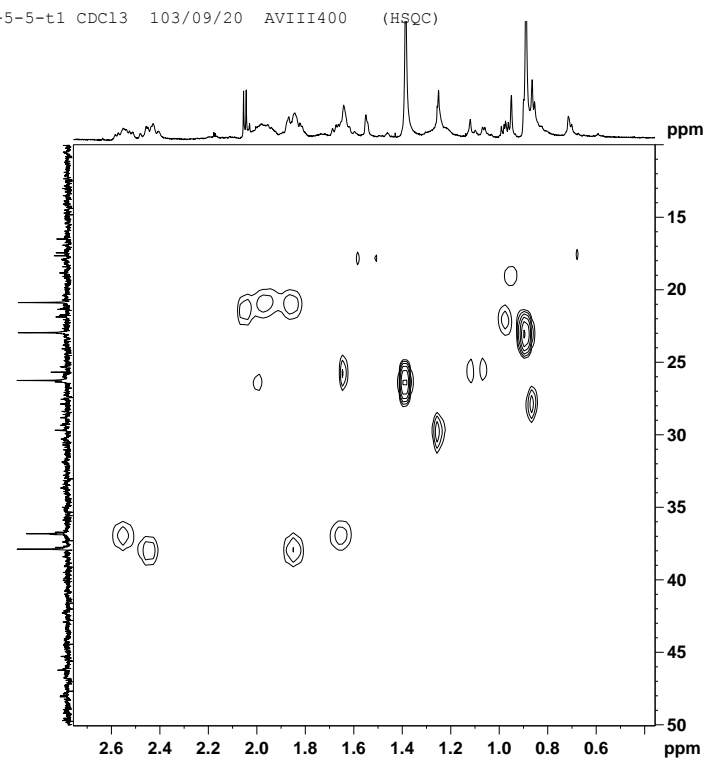


S 64 HSQC spectrum and key of HSQC spectrum of fomitopin-J (10).

FP-62-10-5-5-t1 CDC13 103/09/20 AVIII400 (HSQC)

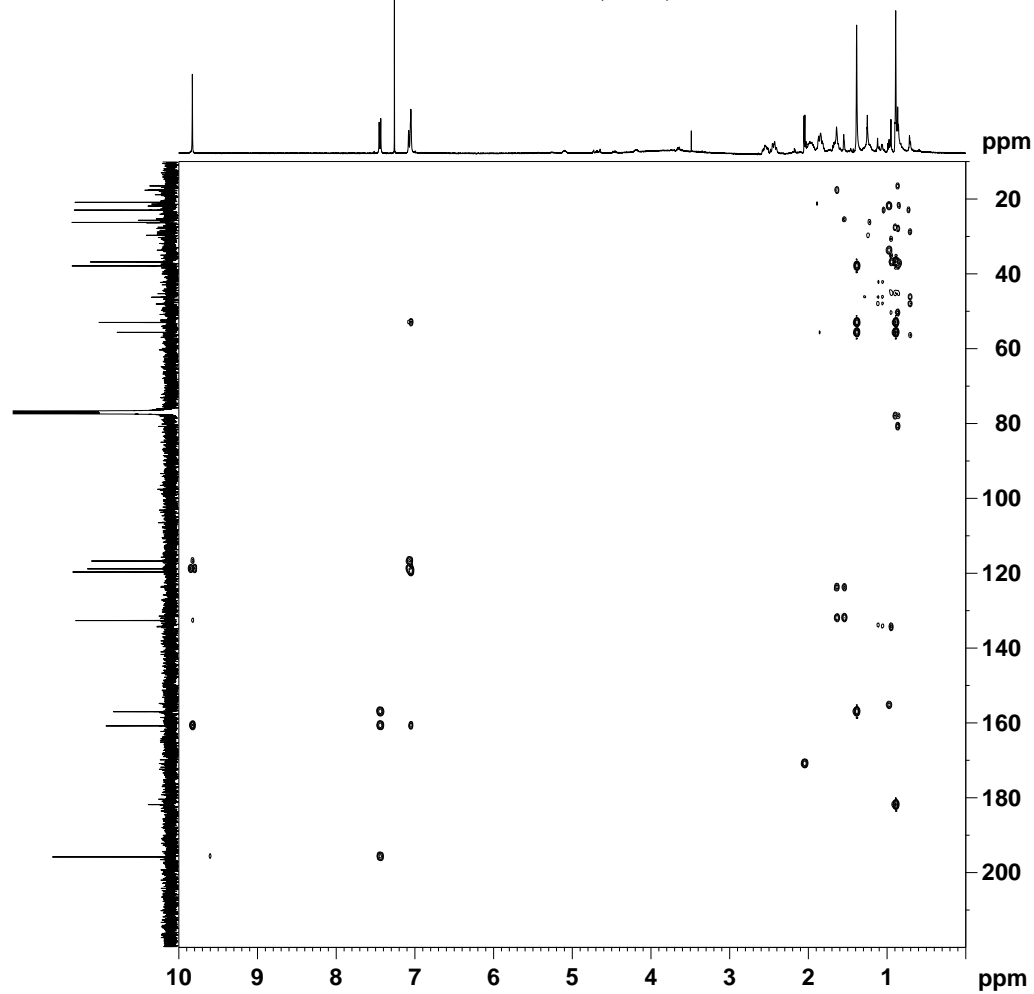


FP-62-10-5-5-t1 CDC13 103/09/20 AVIII400 (HSQC)

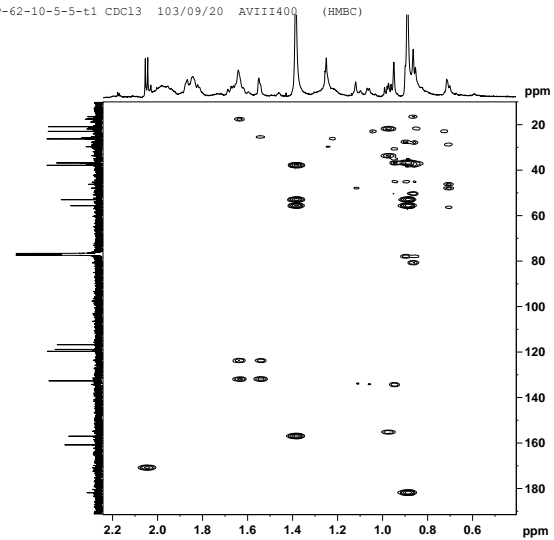


S 65 HMBC spectrum of fomitopin-J (10).

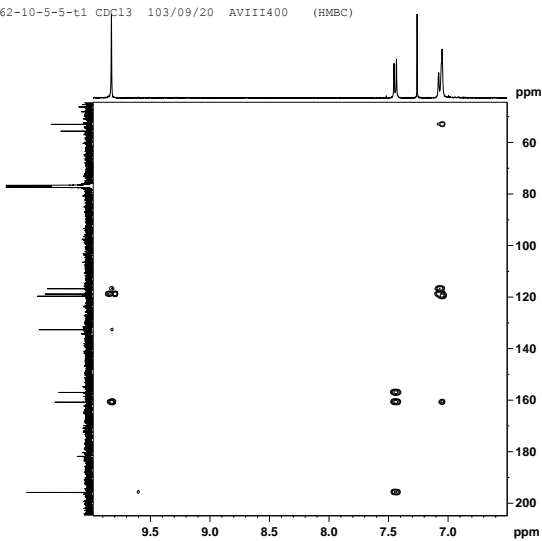
FP-62-10-5-5-t1 CDC13 103/09/20 AVIII400 (HMBC)



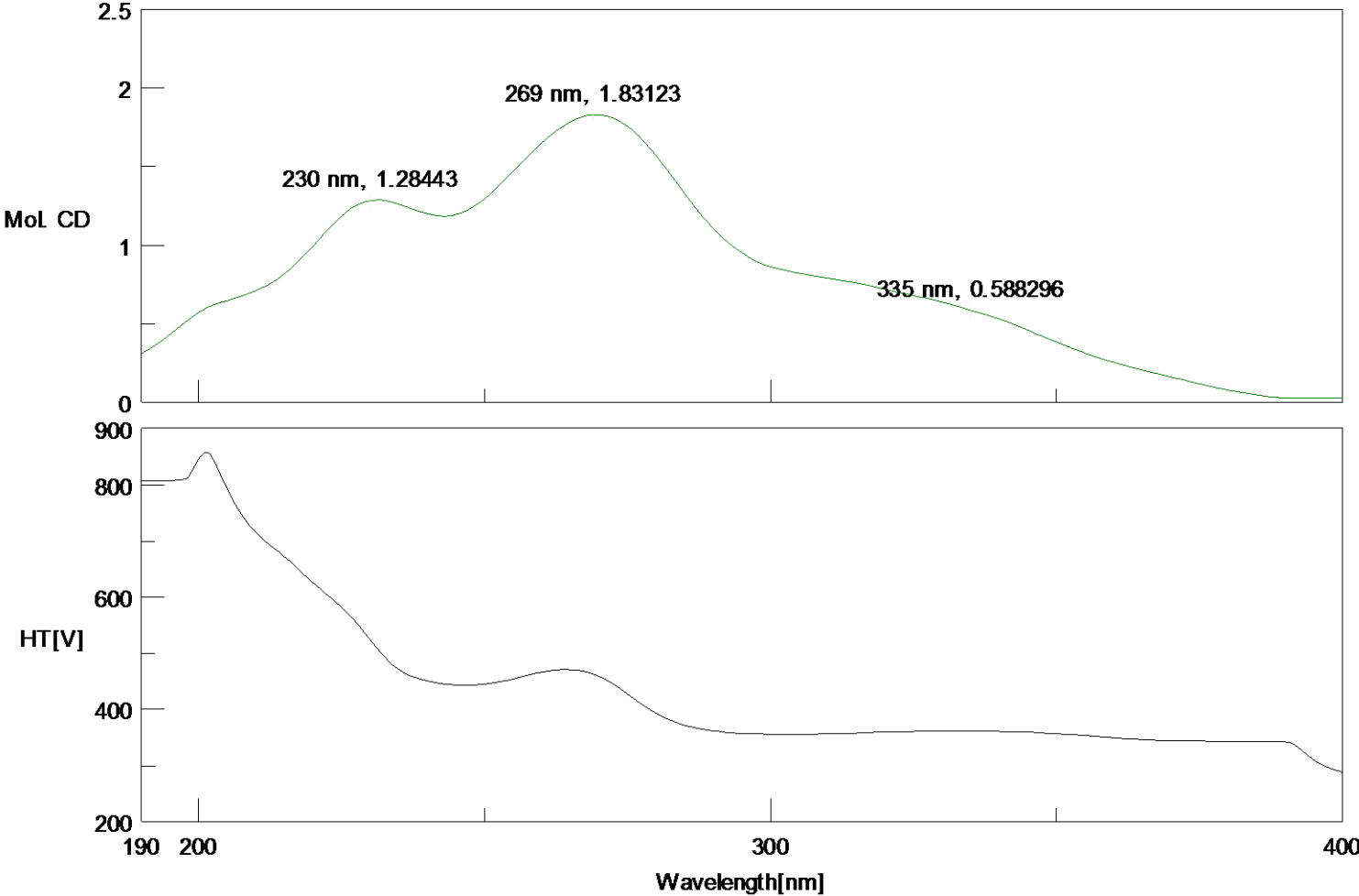
FP-62-10-5-5-t1 CDC13 103/09/20 AVIII400 (HMBC)



FP-62-10-5-5-t1 CDC13 103/09/20 AVIII400 (HMBC)

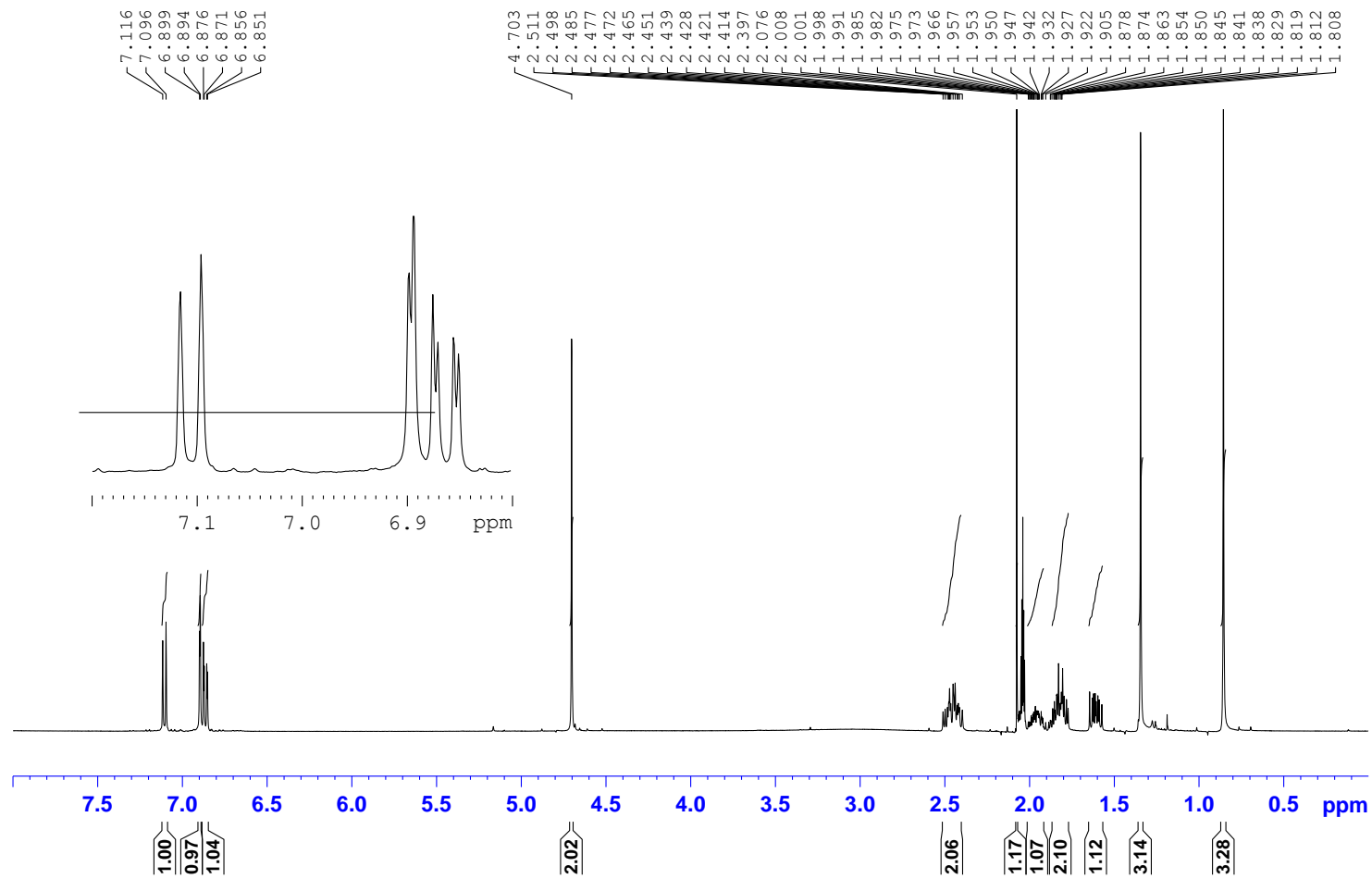


S 66 ECD spectrum of fomitopin-J (10).



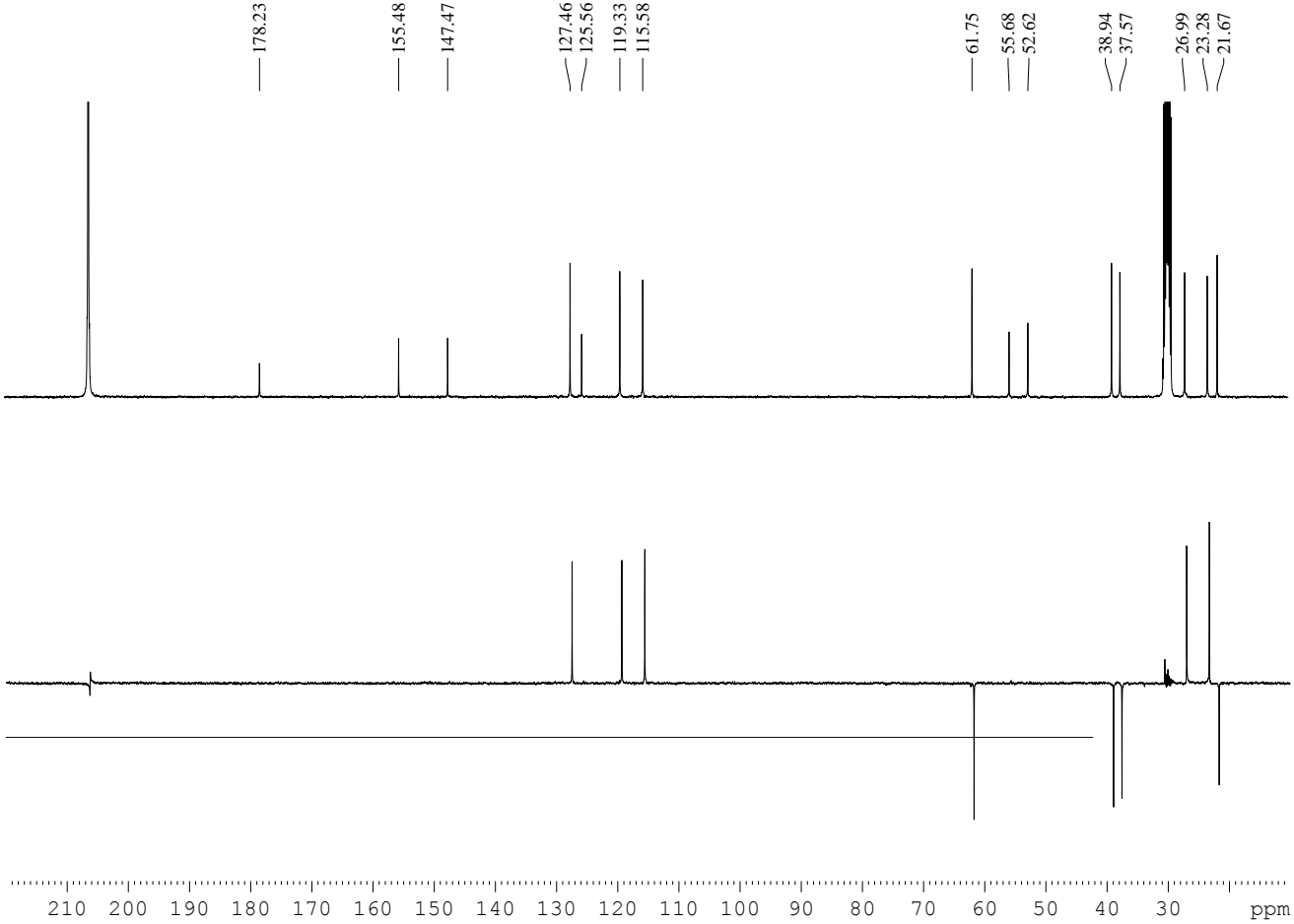
S 67 ¹H NMR (400 MHz, Acetone-d₆) spectrum of fomitopin-K (11).

FP-5-13-8-2C Acetone 102/12/15 AVIII400 (H1)



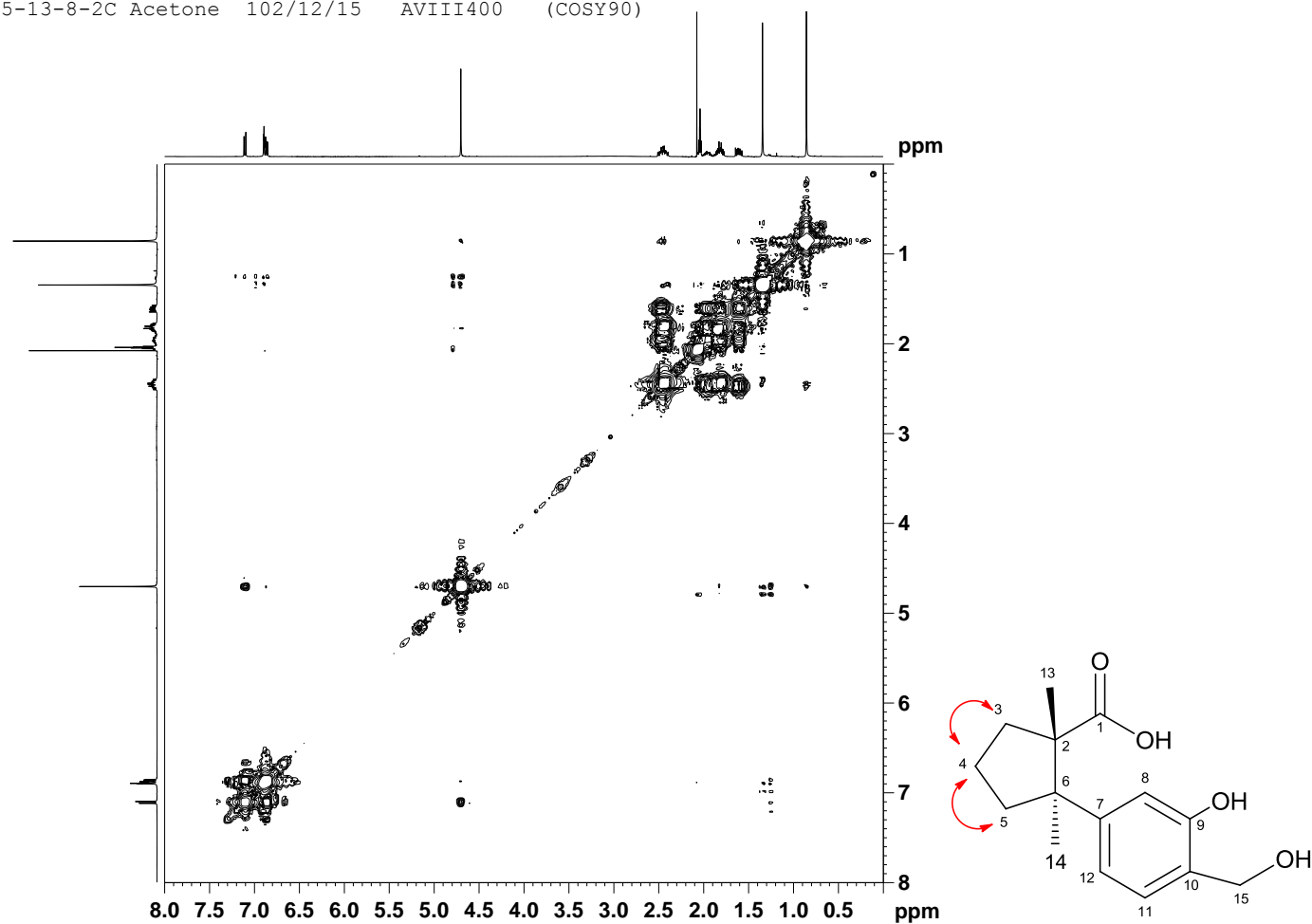
S 68 ¹³C NMR (100 MHz, Acetone-d₆) spectrum of fomitopin-K (11).

FP-5-13-8-2C Acetone 102/12/15 AVIII400 (C13)



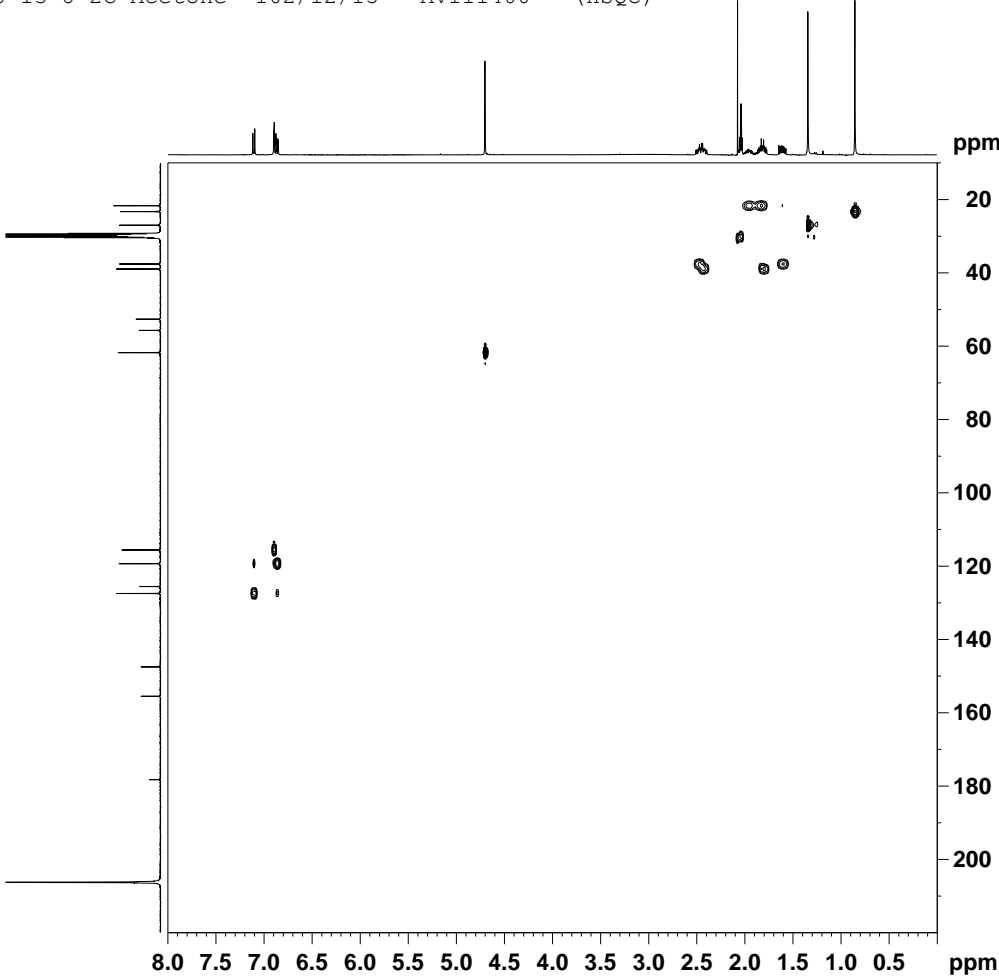
S 69 COSY spectrum of fomitopin-K (11).

FP-5-13-8-2C Acetone 102/12/15 AVIII400 (COSY90)



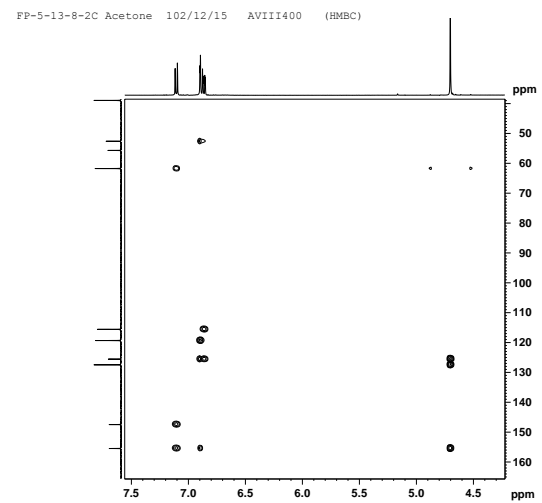
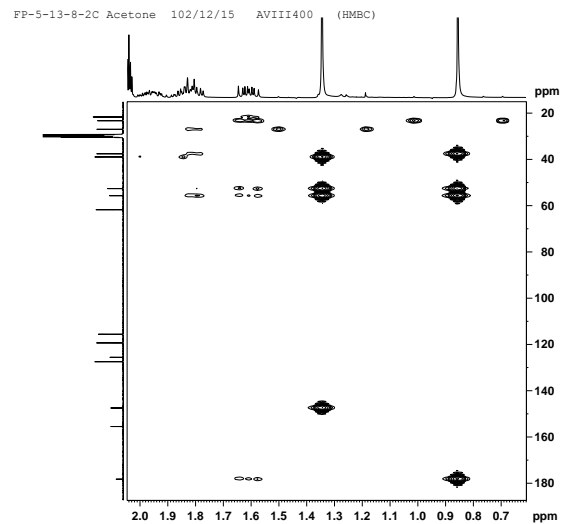
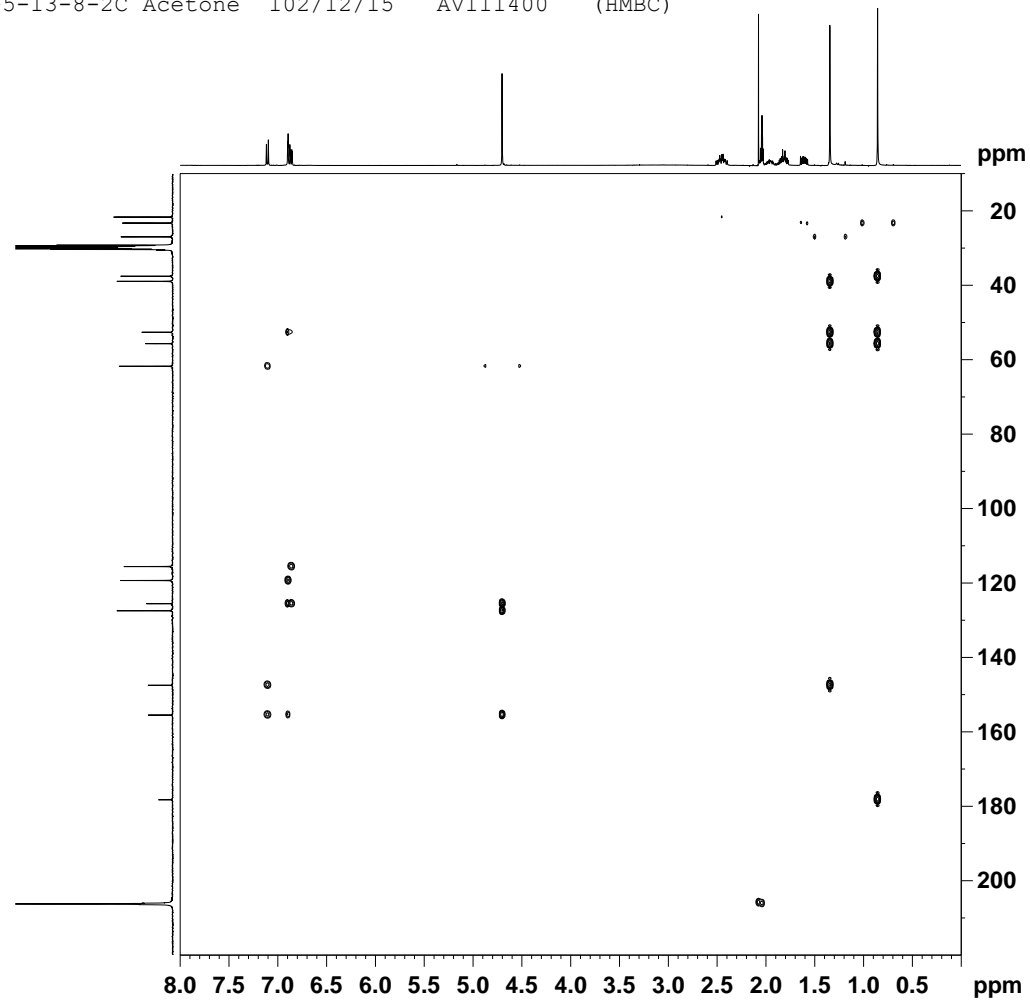
S 70 HSQC spectrum and key of HSQC spectrum of fomitopin-K (11).

FP-5-13-8-2C Acetone 102/12/15 AVIII400 (HSQC)

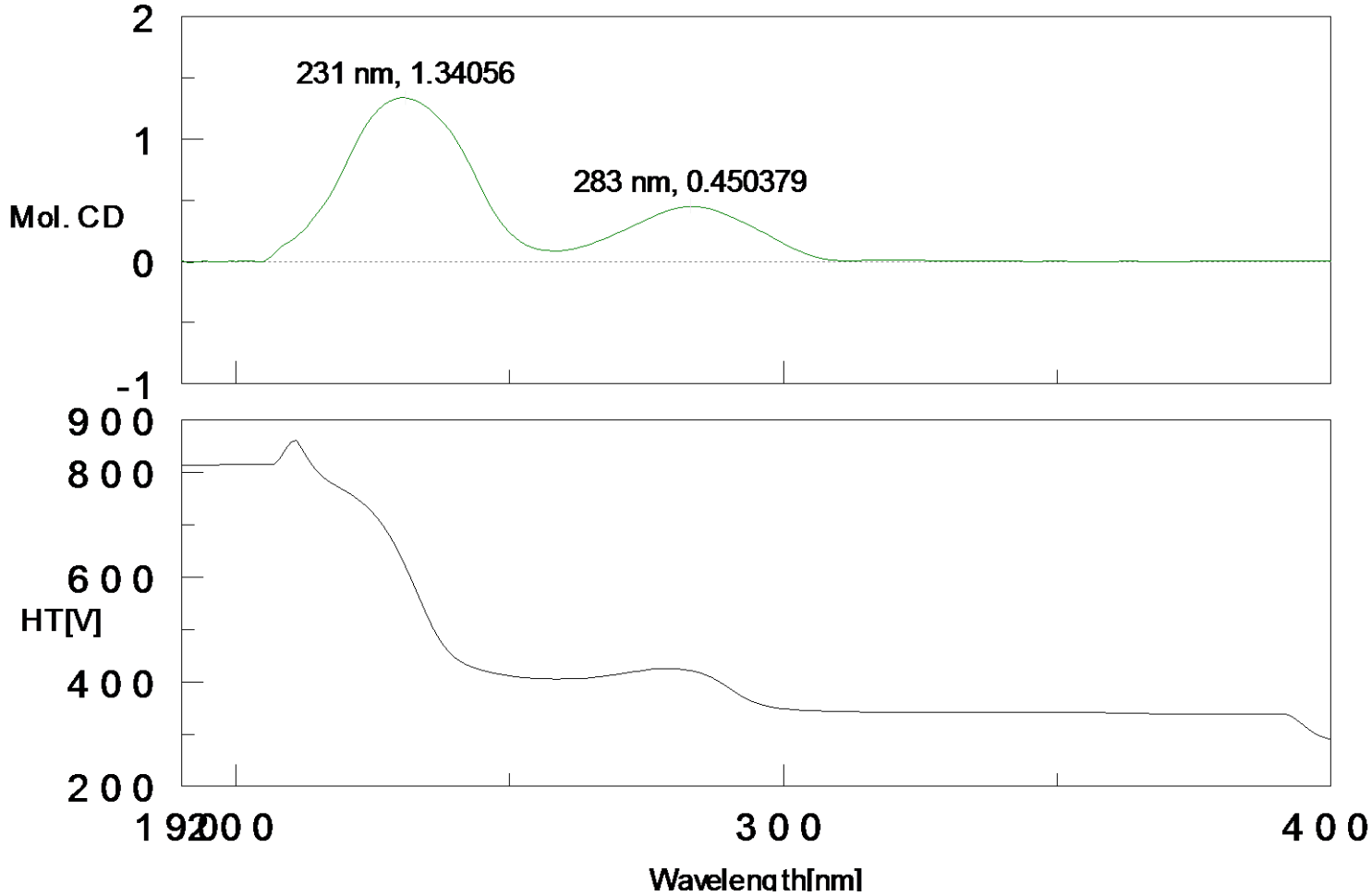


S 71 HMBC spectrum of fomitopin-K (11).

FP-5-13-8-2C Acetone 102/12/15 AVIII400 (HMBC)

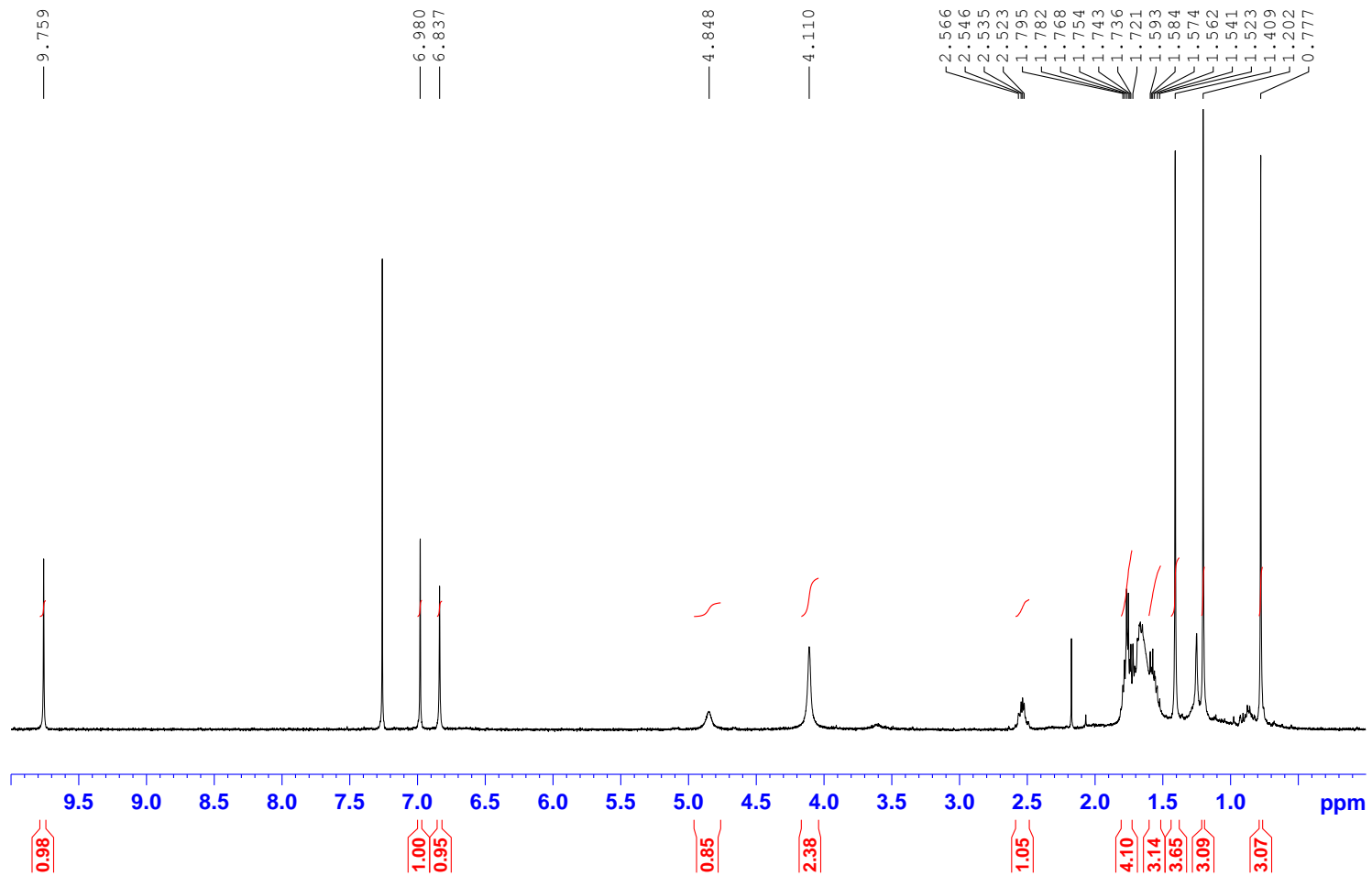


S 72 ECD spectrum of fomitopin-K (11).



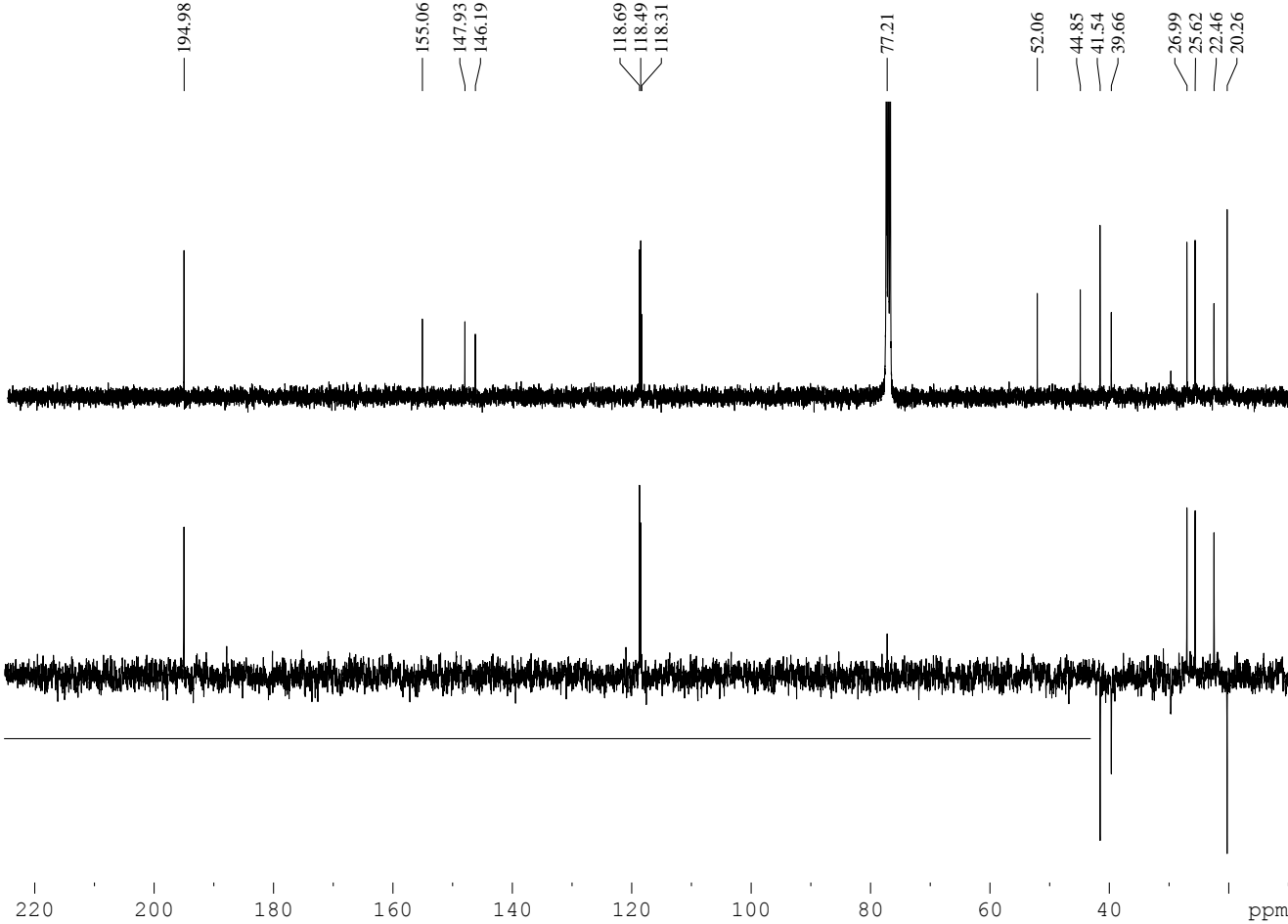
S 69 ¹H NMR (400 MHz, CDCl₃) spectrum of fomitopin-L (12).

FP-6256-t1-t2 CDCl₃ 103/08/18 AVIII400 (H1)



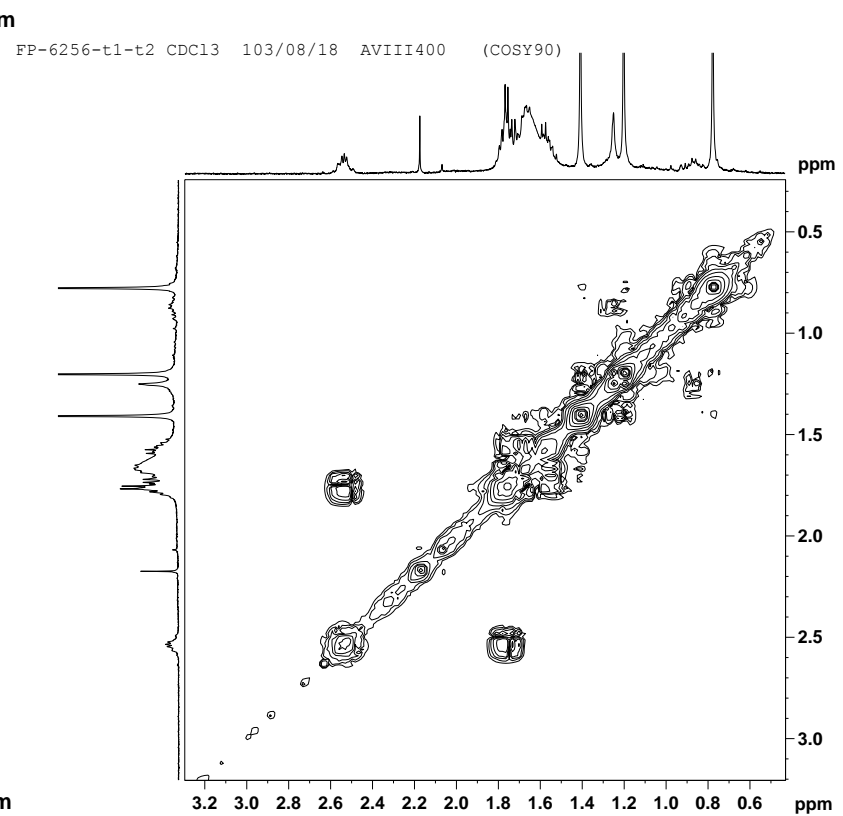
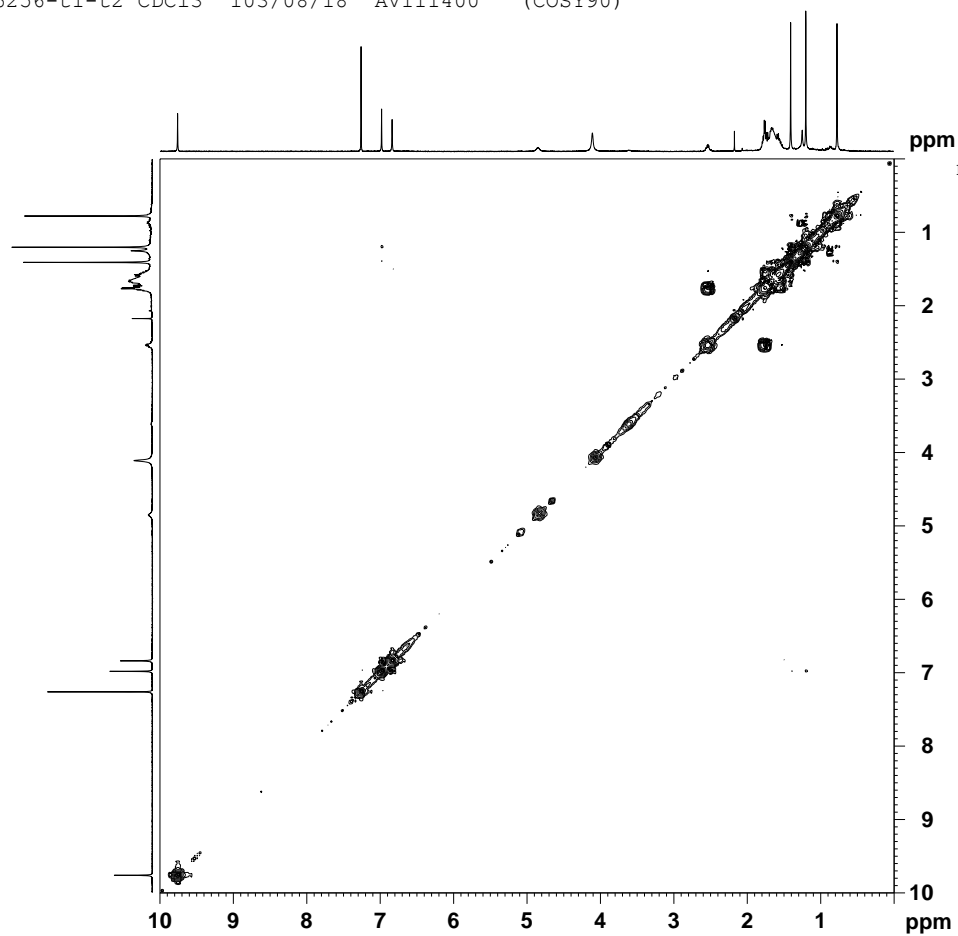
S 70 ¹³C NMR (100 MHz, CDCl₃) spectrum of fomitopin-L (12).

FP-6256-t1-t2 CDC13 103/08/18 AVIII400 (C13)



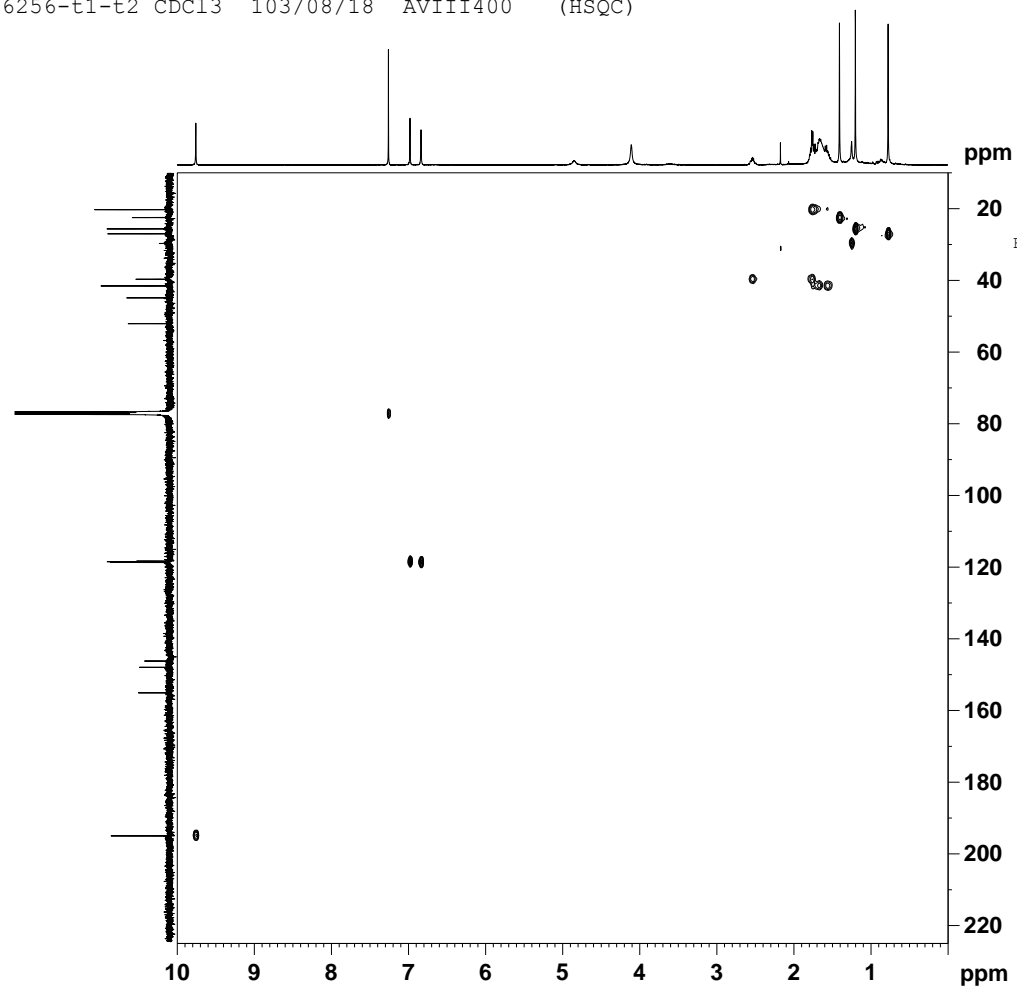
S 71 COSY spectrum of fomitopin-L (12).

FP-6256-t1-t2 CDC13 103/08/18 AVIII400 (COSY90)

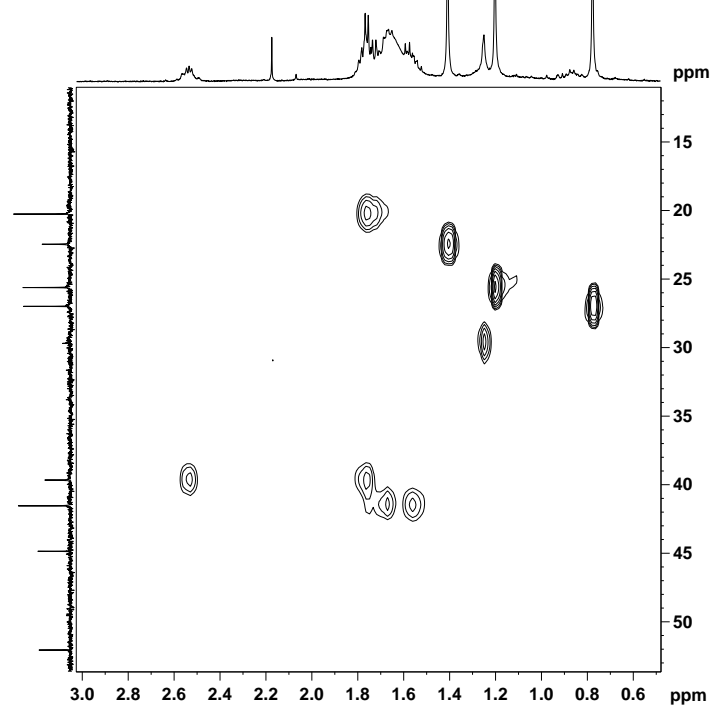


S 72 HSQC spectrum and key of HSQC spectrum of fomitopin-L (12).

FP-6256-t1-t2 CDC13 103/08/18 AVIII400 (HSQC)

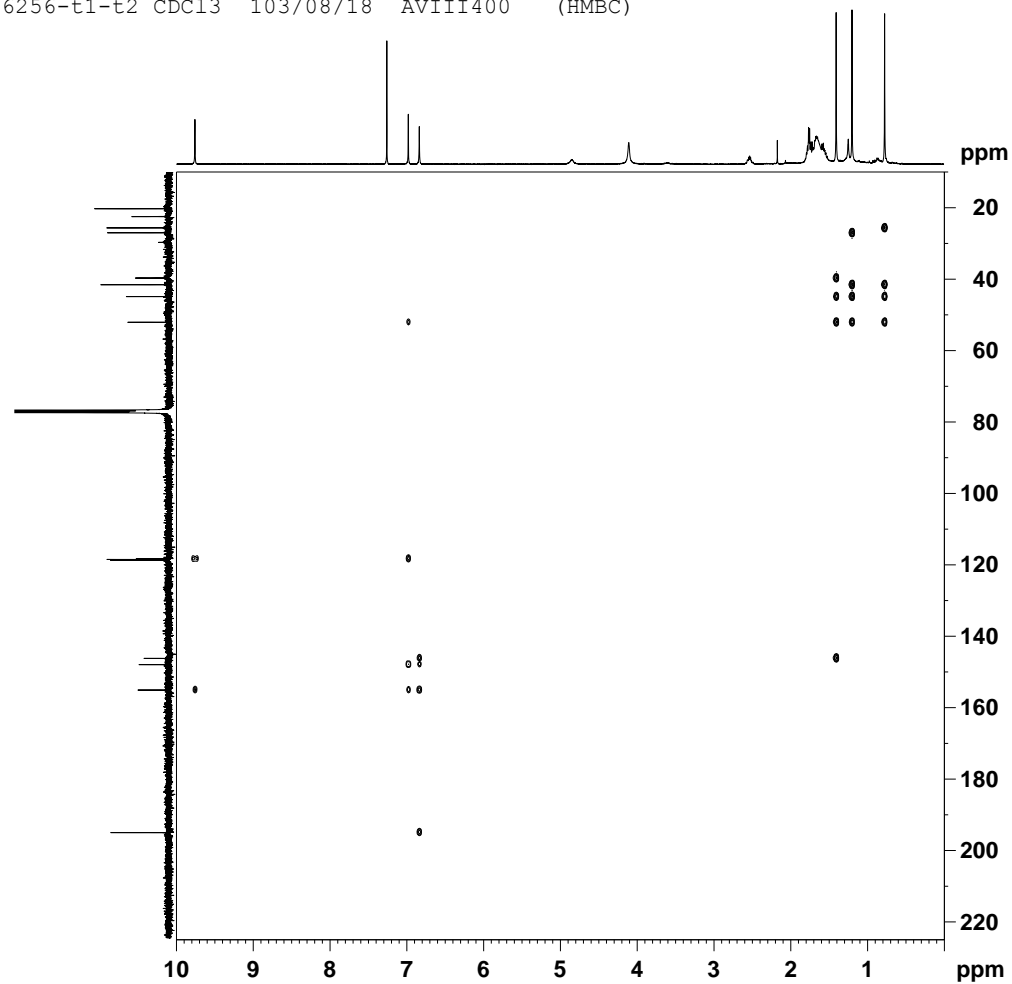


FP-6256-t1-t2 CDC13 103/08/18 AVIII400 (HSQC)

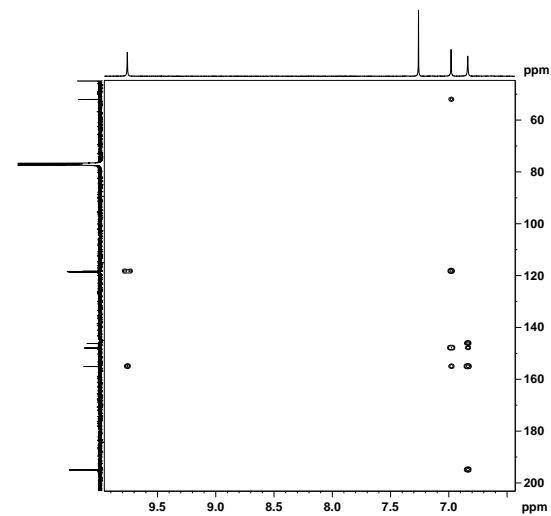


S 73 HMBC spectrum of fomitopin-L (12).

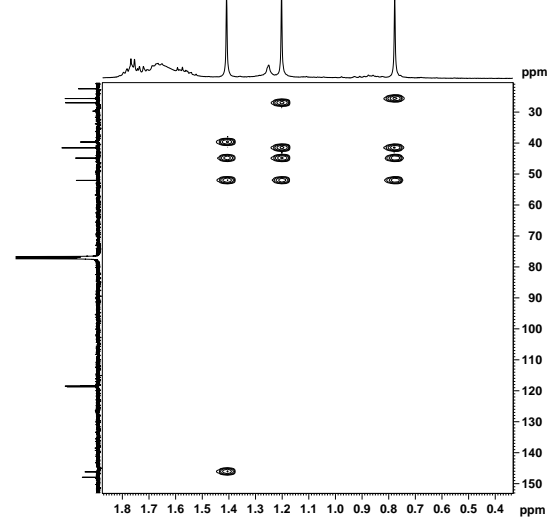
FP-6256-t1-t2 CDCl3 103/08/18 AVIII400 (HMBC)



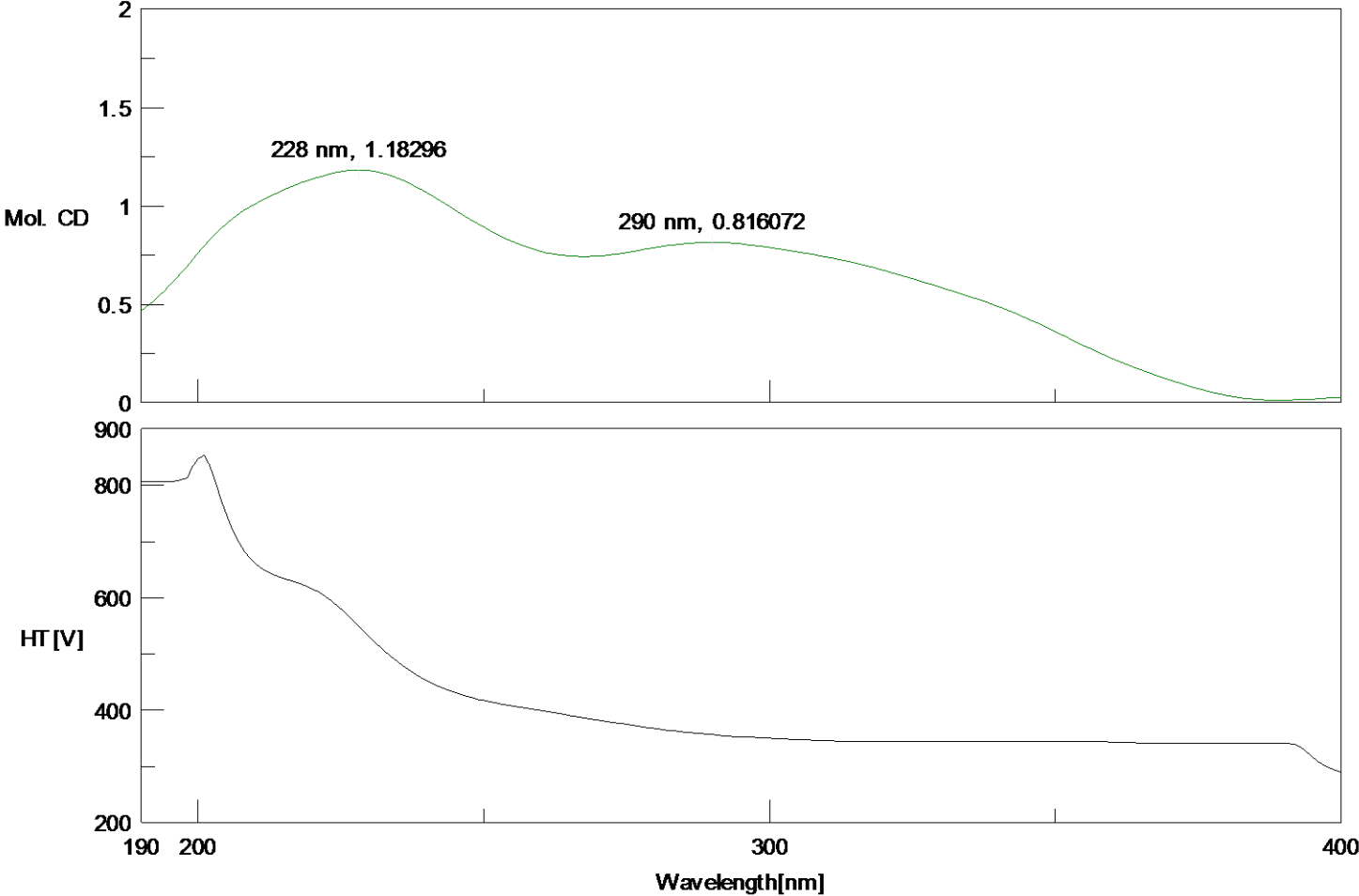
FP-6256-t1-t2 CDCl3 103/08/18 AVIII400 (HMBC)



FP-6256-t1-t2 CDCl3 103/08/18 AVIII400 (HMBC)



S 74 ECD spectrum of fomitopin-L (12).



S 75 Anti-inflammatory activity of *Fomitopsis pinicola* fractions.

Fraction	Superoxide anion			Fraction	Elastase release		
	IC ₅₀ (μM) ^a	Inh %			IC ₅₀ (μM) ^a	Inh %	
FP 1	>10	16.45 ± 1.75	***	FP 1	>10	16.10 ± 5.06	*
FP 2	>10	19.68 ± 1.91	***	FP 2	>10	17.05 ± 3.73	*
FP 3	2.52 ± 0.24	68.55 ± 1.61	***	FP 3	>10	17.59 ± 3.48	**
FP 4	2.22 ± 0.17	68.73 ± 1.20	***	FP 4	1.88 ± 0.26	94.52 ± 5.55	***
FP 5	1.64 ± 0.01	90.26 ± 0.68	***	FP 5	2.85 ± 0.66	95.39 ± 6.53	***
FP 6	4.06 ± 0.24	84.46 ± 2.07	***	FP 6	3.38 ± 0.89	89.15 ± 8.91	***

Percentage of inhibition (Inh %) at 10 μM concentration.

* $P < 0.05$, ** $P < 0.01$ compared with the control value.

^aConcentration necessary for 50 % inhibition(IC₅₀)

Table 1. Crystal data and structure refinement for Fomitopin A.

Identification code	Fomitopin A
Empirical formula	C ₁₅ H ₁₈ O ₄
Formula weight	262.29
Temperature	150(2) K
Wavelength	1.54178 Å
Crystal system	Orthorhombic
Space group	P 21 21 21
Unit cell dimensions	a = 6.6564(2) Å a = 90°. b = 10.3704(3) Å b = 90°. c = 17.9622(6) Å g = 90°.
Volume	1239.92(7) Å ³
Z	4
Density (calculated)	1.405 Mg/m ³
Absorption coefficient	0.831 mm ⁻¹
F(000)	560
Crystal size	0.45 x 0.36 x 0.32 mm ³
Theta range for data collection	4.92 to 72.19°.
Index ranges	-7<=h<=5, -12<=k<=12, -21<=l<=21
Reflections collected	4664
Independent reflections	2382 [R(int) = 0.0183]
Completeness to theta = 72.19°	98.3 %
Absorption correction	Semi-empirical from equivalents

Max. and min. transmission	1.00000 and 0.96185
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2382 / 0 / 180
Goodness-of-fit on F ²	1.022
Final R indices [I>2sigma(I)]	R1 = 0.0375, wR2 = 0.0975
R indices (all data)	R1 = 0.0407, wR2 = 0.1000
Absolute structure parameter	0.0(2)
Largest diff. peak and hole	0.216 and -0.192 e.Å ⁻³