

Supplementary Materials

A New Dihydrochromone Dimer and Other Secondary Metabolites from Cultures of the Marine Sponge-Associated Fungi *Neosartorya fennelliae* KUFA 0811 and *Neosartorya tsunodae* KUFC 9213

Decha Kumla ^{a, b, ||}, Tin Shine Aung ^{a, b, ||}, Suradet Buttachon ^{a, b}, Tida Dethoup ^c, Luís Gales ^{a, d}, José A. Pereira ^{a, b}, Ângela Inácio ^b, Paulo M. Costa ^{a, b}, Michael Lee ^e, Nazim Sekeroglu ^f, Artur M. S. Silva ^g, Madalena M. M. Pinto ^{b, h}, Anake Kijjoa ^{a, b*}

^a ICBAS-Instituto de Ciências Biomédicas Abel Salazar, Rua de Jorge Viterbo Ferreira, 228, 4050-313 Porto, Portugal. E-mail: decha1987@hotmail.com (DK), tinshineaung@gmail.com (TSA), jpereira@icbas.up.pt (JAP), pmcosta@icbas.up.pt (PMC).

^b Interdisciplinary Centre of Marine and Environmental Research (CIIMAR), Terminal de Cruzeiros do Porto de Leixões, Av. General Norton de Matos s/n, 4450-208, Matosinhos, Portugal. E-mail: nokrari_209@hotmail.com (SB), angelainacio@gmail.com (AI).

^c Department of Plant Pathology, Faculty of Agriculture, Kasetsart University, Bangkok 10240, Thailand. E-mail: tdethoup@yahoo.com.

^d Instituto de Biologia Molecular e Celular (i3S-IBMC), Universidade do Porto, Rua de Jorge Viterbo Ferreira, 228, 4050-313 Porto, Portugal. E-mail: lgales@ibmc.up.pt.

^e Department of Chemistry, University of Leicester, University Road, Leicester LE 7 RH, UK. E-mail: m134@leicester.ac.uk.

^f Medicinal and Aromatic Plant Programme, Plant and Animal Sciences Department, Vocational School, Kilis 7 Aralık University, 79000, Kilis, Turkey. E-mail: nsekeroglu@gmail.com.

^g Departamento de Química & QOPNA, Universidade de Aveiro, 3810-193 Aveiro, Portugal. E-mail: artur.silva@ua.pt.

^h Laboratório de Química Orgânica, Departamento de Ciências Químicas, Faculdade de Farmácia, Universidade do Porto, Rua de Jorge Viterbo Ferreira, 228, 4050-3 13 Porto, Portugal. E-mail: madalena@ff.up.pt

^{||} These authors contributed equally to this work.

*Corresponding author.

E-mail address: ankijjoa@icbas.up.pt (A. Kijjoa)

Comentado [M1]: Changed § to this symbol, please confirm.
AK: It is OK.

Comentado [M2]: Changed § to this symbol, please confirm.
AK: It is OK.

Comentado [M3]: Changed § to this symbol, please confirm.
AK: It is OK.

Figure S1. Structures of metabolites isolated from *Neosartorya tsunodae* KUFC 9231 and *N. fennelliae* KUFA 0811.

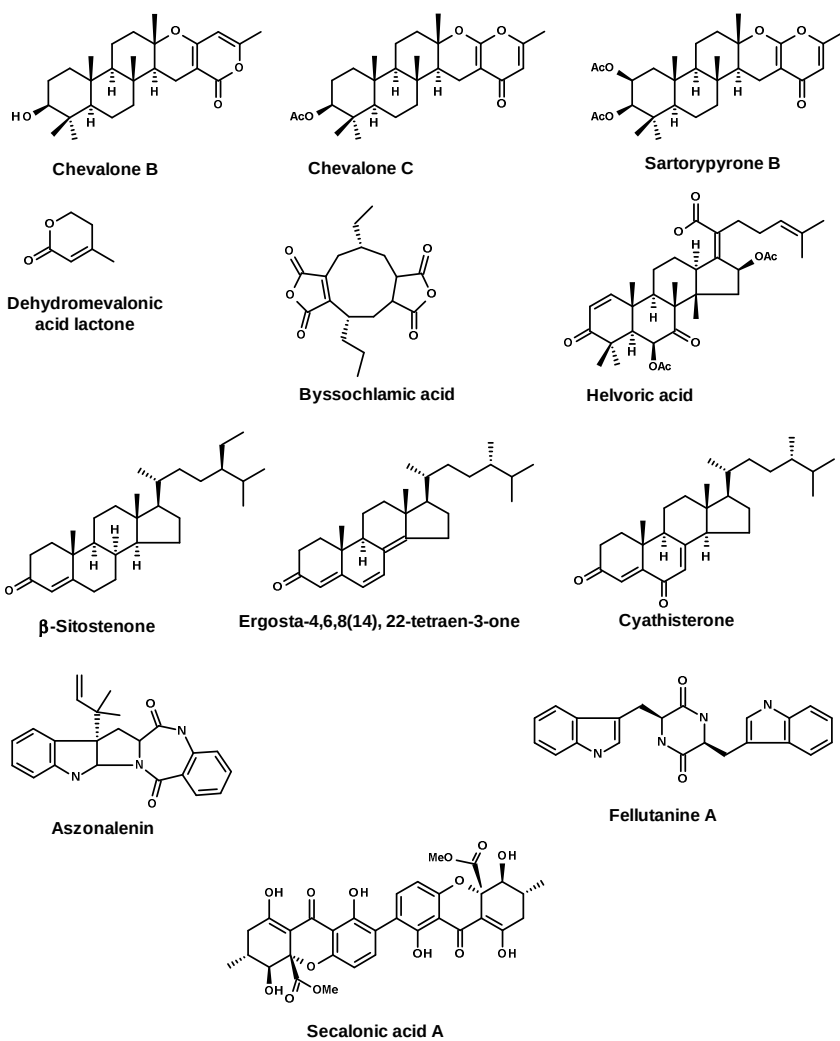


Figure S2. ^1H NMR spectrum of byssochlamic acid (CDCl_3 , 300.13 MHz).

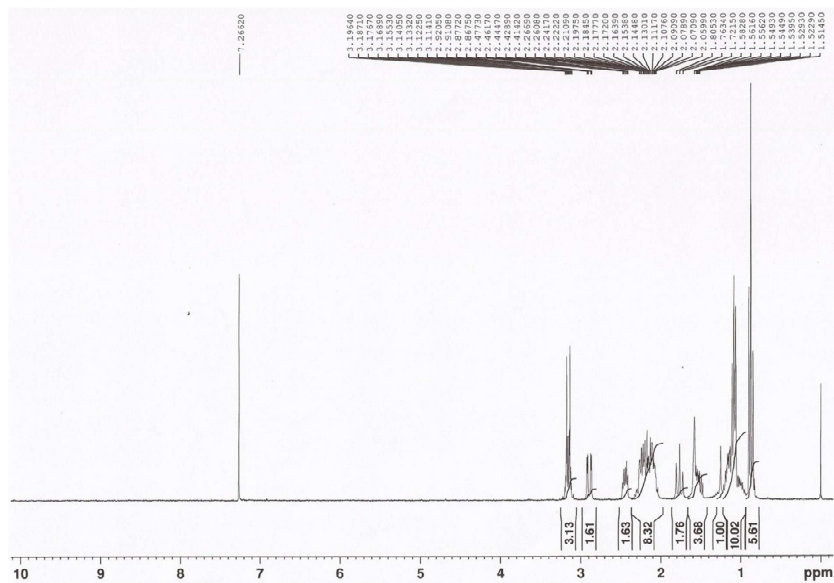
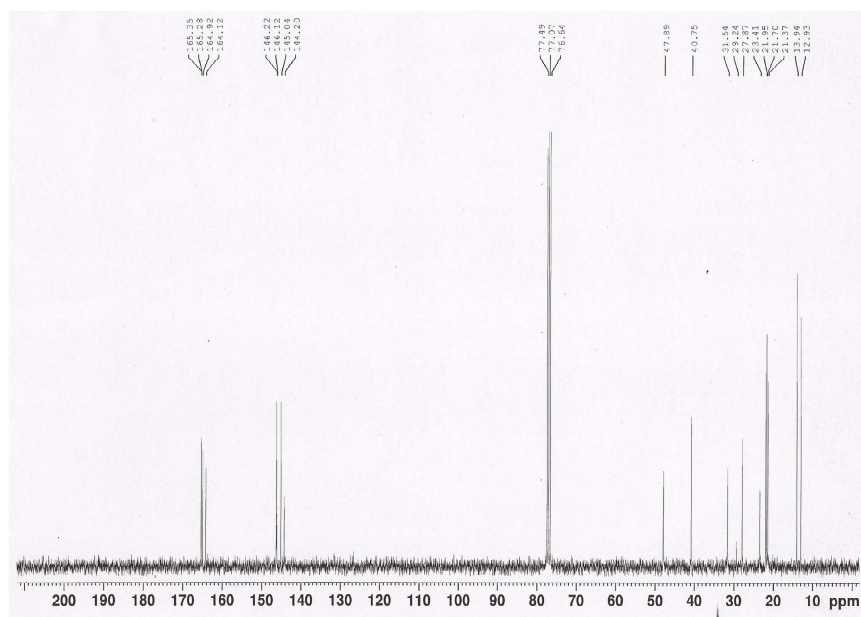


Figure S3. ^{13}C NMR spectrum of byssochlamic acid (CDCl_3 , 75.4 MHz).



[illegible]

Chemical shifts (ppm) labeled on the right side of the spectrum:

- 79.02
- 78.93
- 77.62
- 76.78
- 72.88
- 55.89
- 51.09
- 49.08
- 48.08
- 44.76
- 41.70
- 38.84
- 38.68
- 34.40
- 32.26
- 29.73
- 28.73
- 27.38
- 24.01
- 21.92
- 18.38
- 17.00
- 16.16

Figure S6. ^1H NMR spectrum of chevalone B (CDCl_3 , 300.13 MHz).

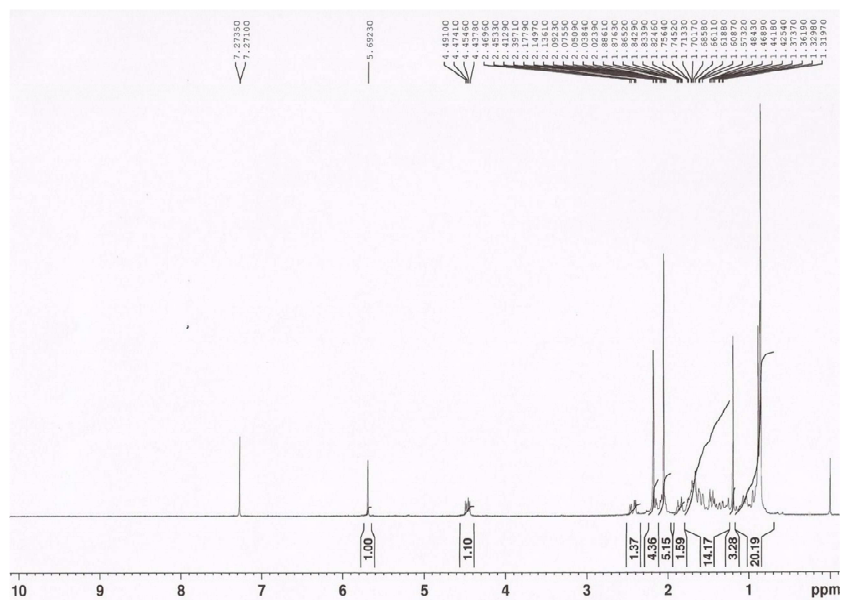


Figure S7. ^{13}C NMR spectrum of chevalone B (CDCl_3 , 75.4 MHz).

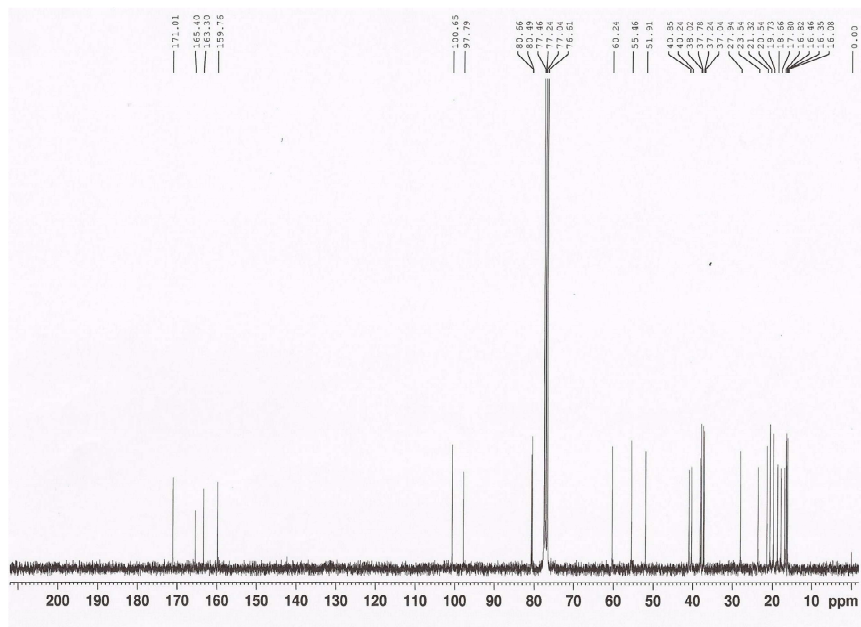


Figure S8. ^1H NMR spectrum of chevalone C (CDCl_3 , 300.13 MHz).

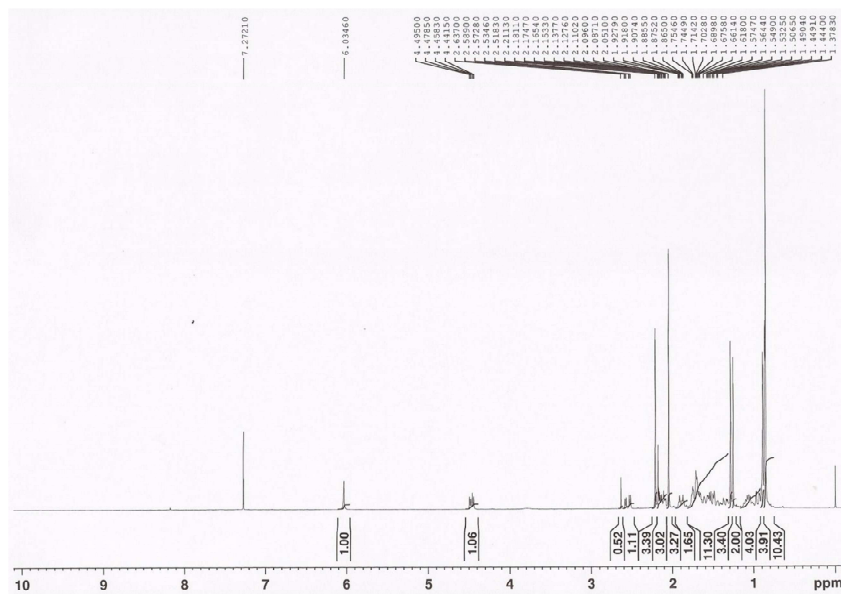


Figure S9. ^{13}C NMR spectrum of chevalone C (CDCl_3 , 75.4 MHz).

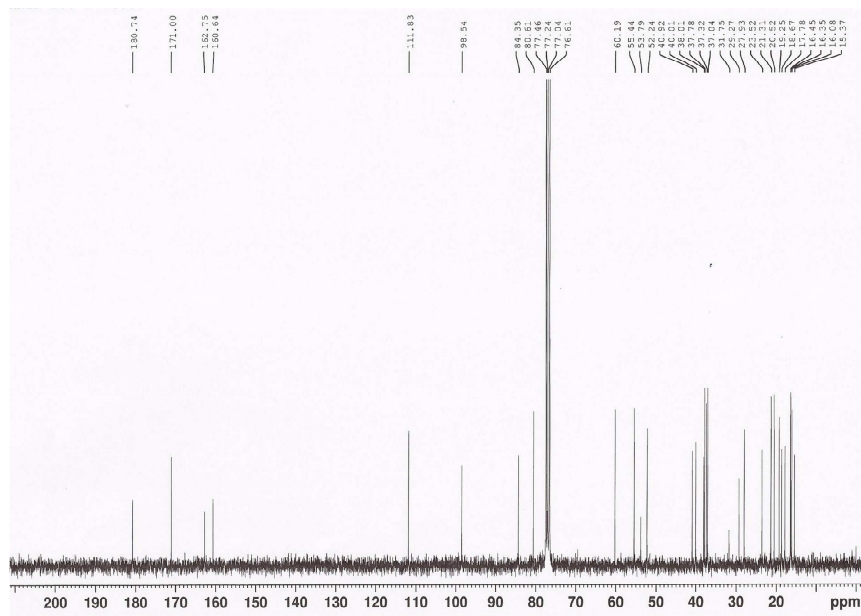


Figure S10. ^1H NMR spectrum of sartorypyrone B (CDCl_3 , 300.13 MHz).

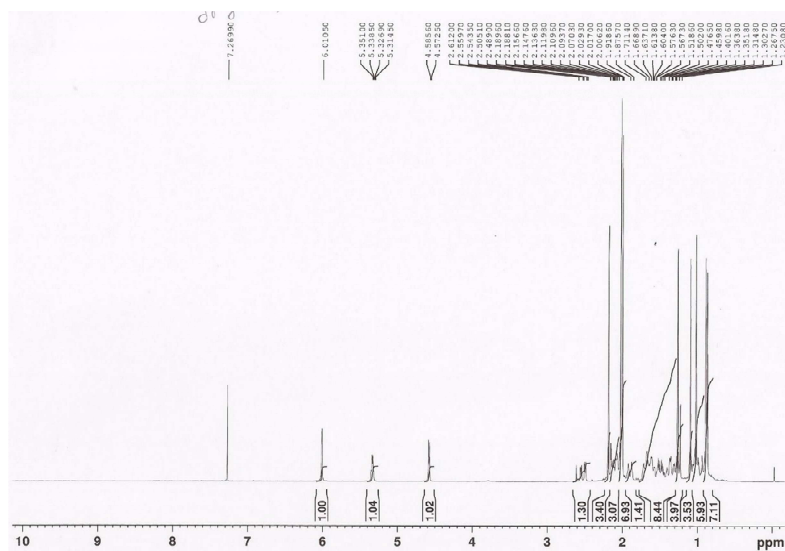


Figure S11. ^{13}C NMR spectrum of sartorypyrone B (CDCl_3 , 75.4 MHz).

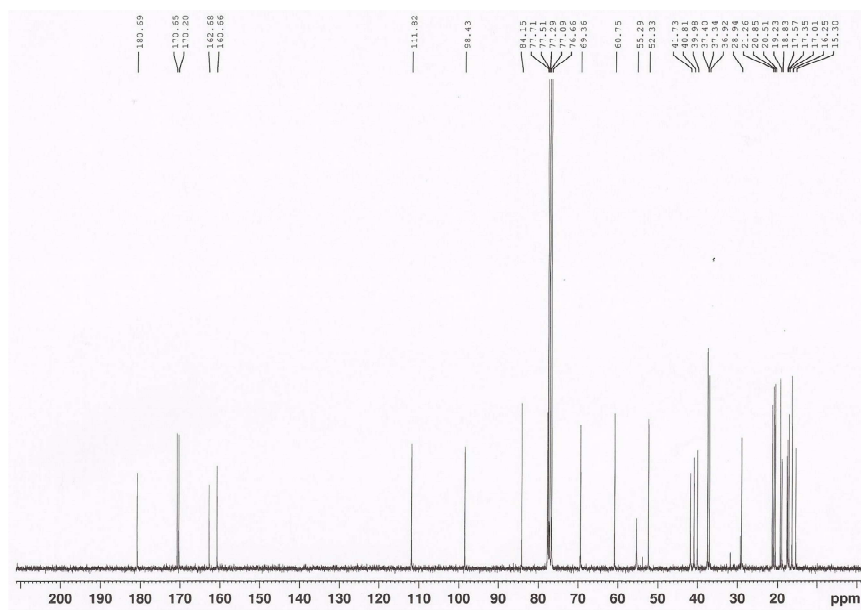


Figure S12. ^1H NMR spectrum of helvolic acid (CDCl_3 , 300.13 MHz).

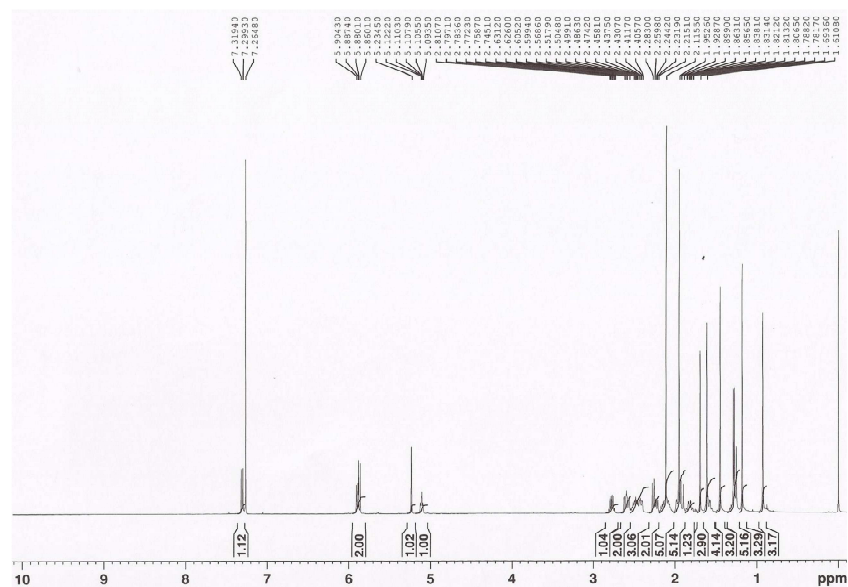


Figure S13. ^{13}C NMR spectrum of helvolic acid (CDCl_3 , 75.4 MHz).

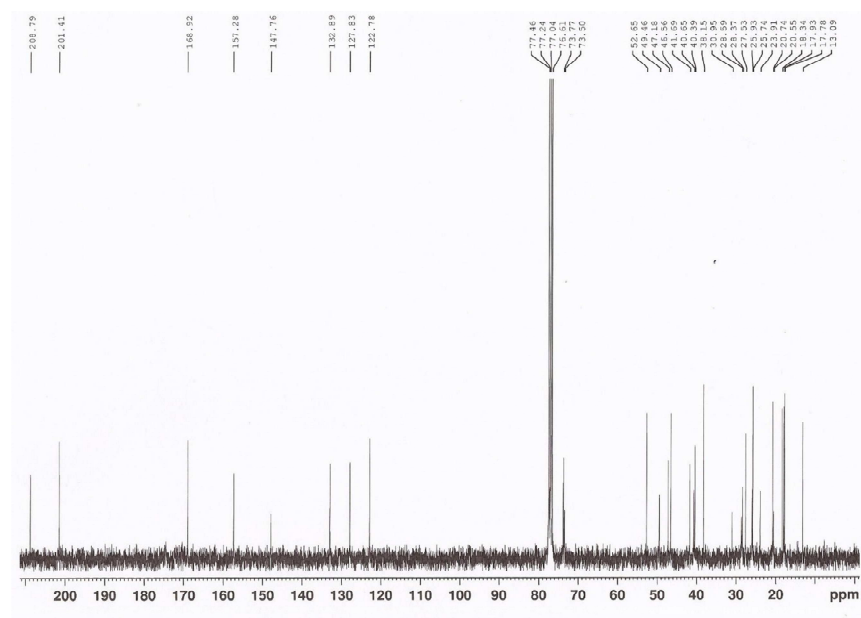


Figure S14. ^1H NMR spectrum of lumichrome (DMSO, 300.13 MHz).

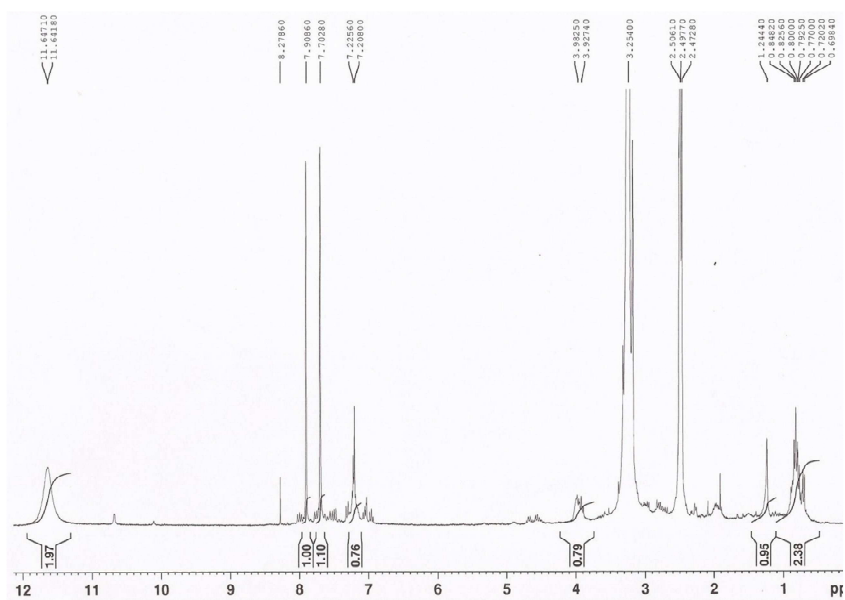


Figure S15. ^{13}C NMR spectrum of lumichrome (DMSO, 75.4 MHz).

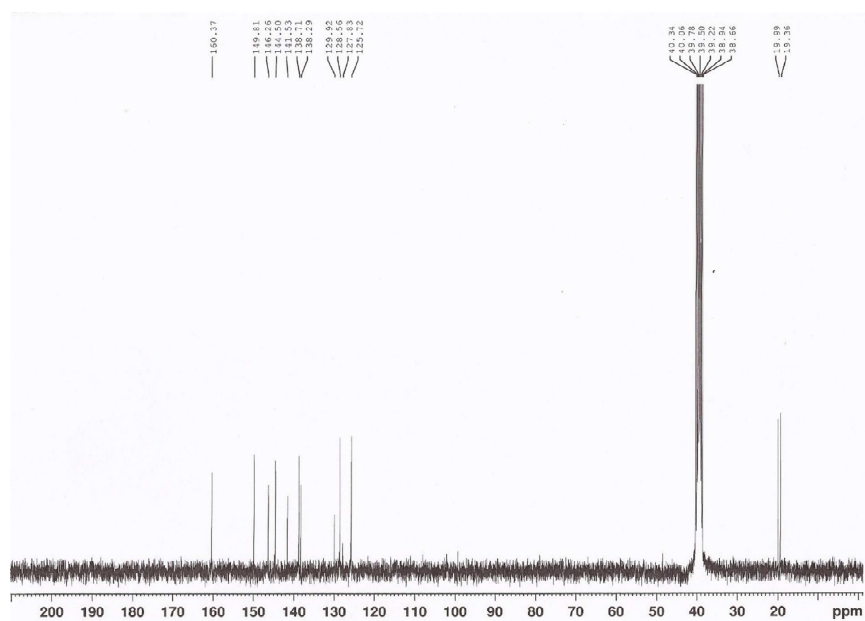


Figure S16. ^1H NMR spectrum of harmane (DMSO, 500.13 MHz).

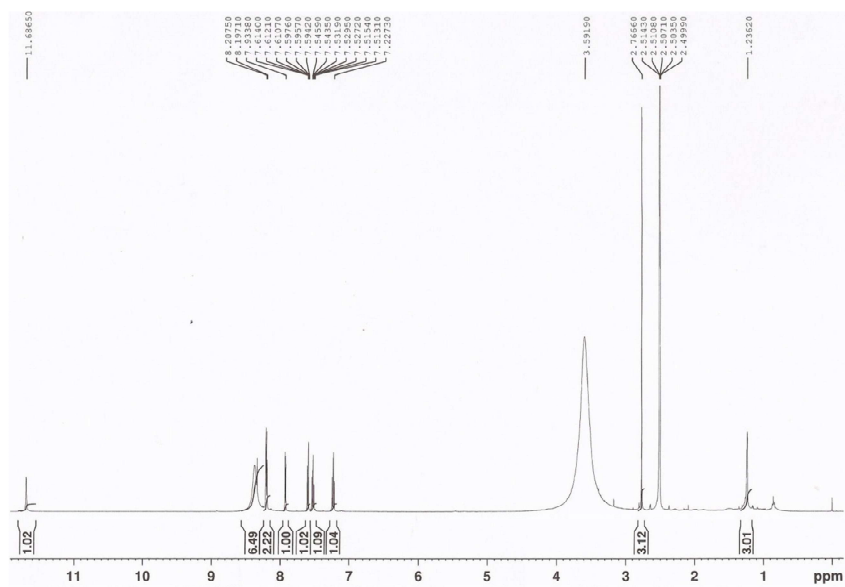


Figure S18. ^1H NMR spectrum of β -sitostenone (CDCl_3 , 300.13 MHz).

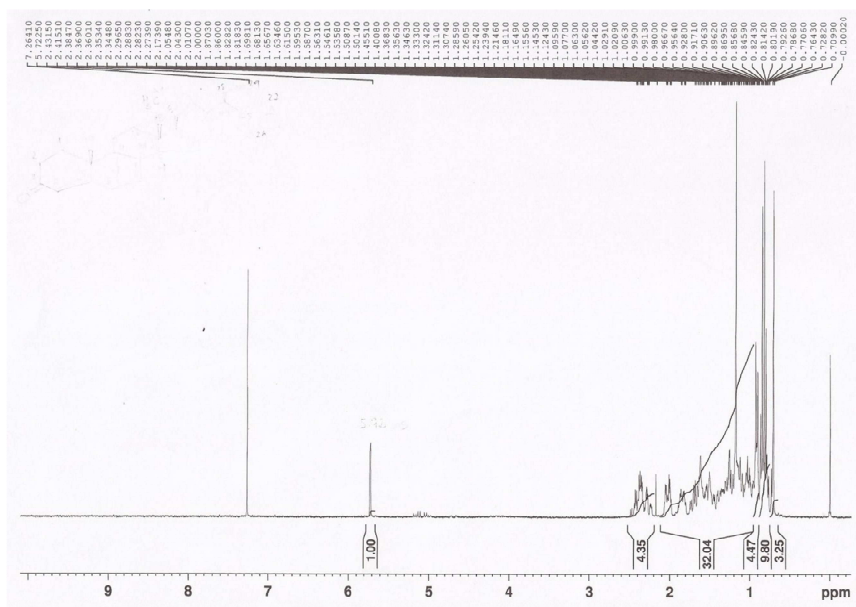


Figure S19. ^{13}C NMR spectrum of β -sitostenone (CDCl_3 , 75.4 MHz).

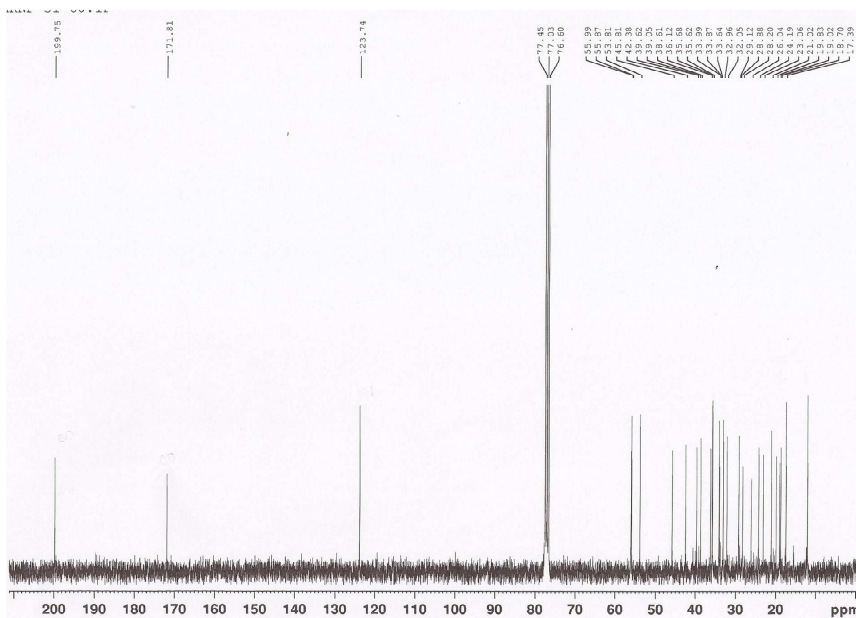


Figure S20. ^1H NMR spectrum of ergosta-4,6,8 (14), 22-tetraen-3-one (CDCl_3 , 300.13 MHz).

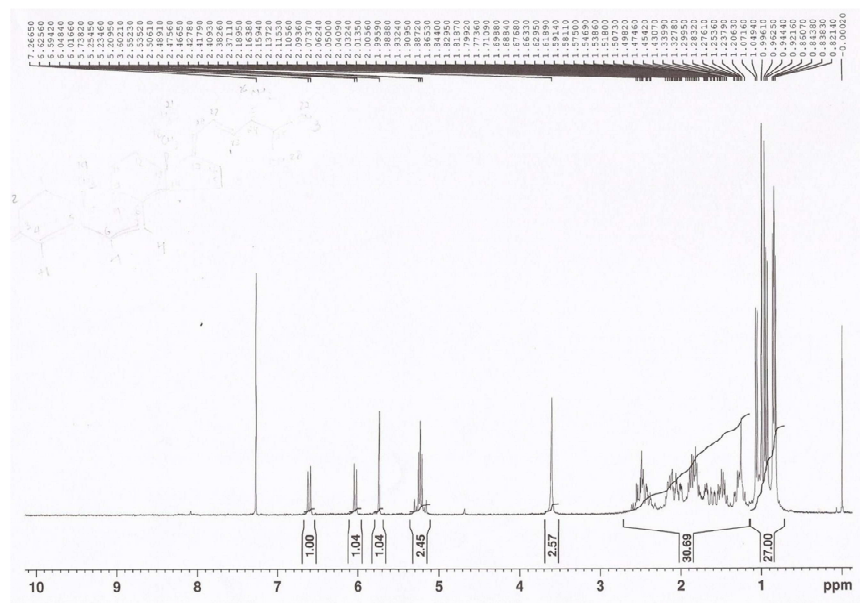


Figure S21. ^{13}C NMR spectrum of ergosta-4,6,8 (14), 22-tetraen-3-one (CDCl_3 , 75.4 MHz).

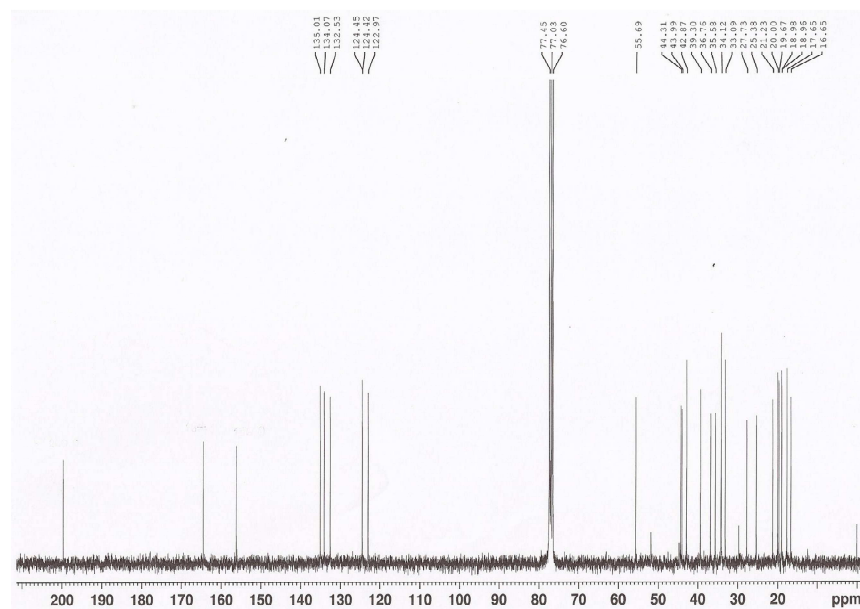


Figure S22. ^1H NMR spectrum of cyathisterone (CDCl_3 , 300.13 MHz).

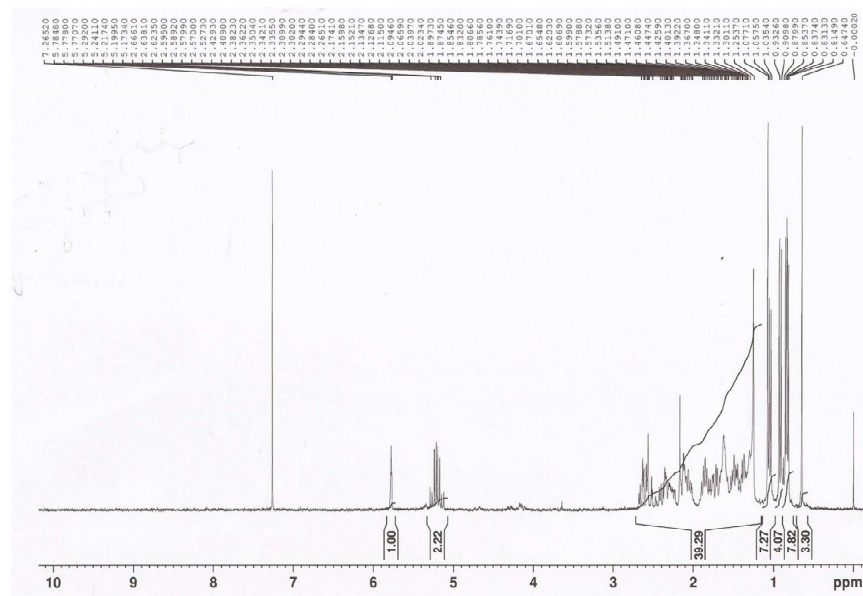


Figure S23. ^{13}C NMR spectrum of cyathisterone (CDCl_3 , 75.4 MHz).

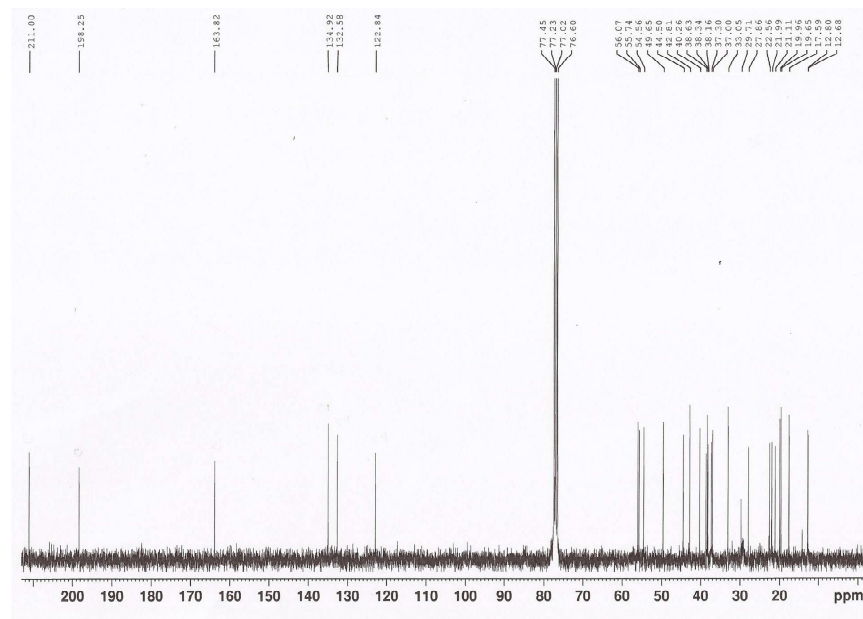


Figure S24. ^1H NMR spectrum of dehydromevalonic acid lactone (CDCl_3 , 300.13 MHz).

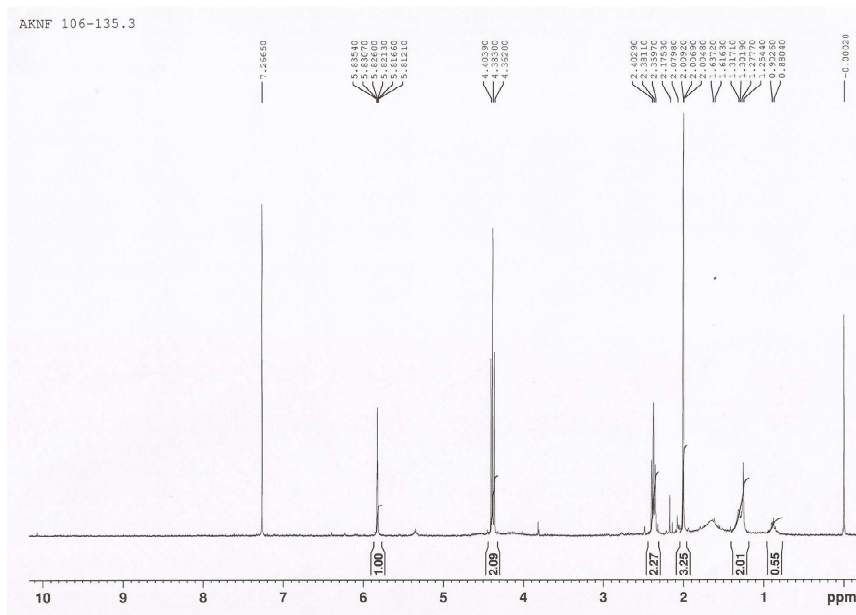


Figure S26. ^1H NMR spectrum of aszonalenin (CDCl_3 , 300.13 MHz).

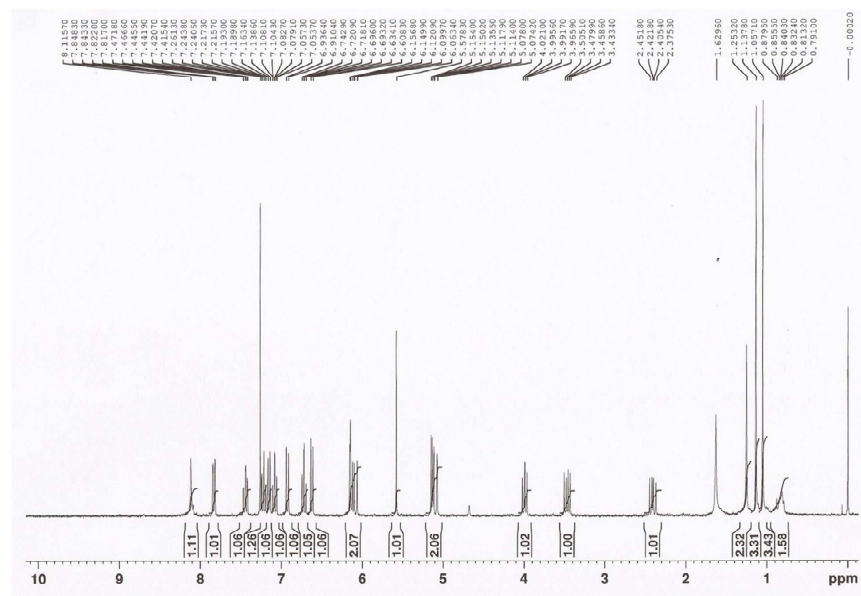
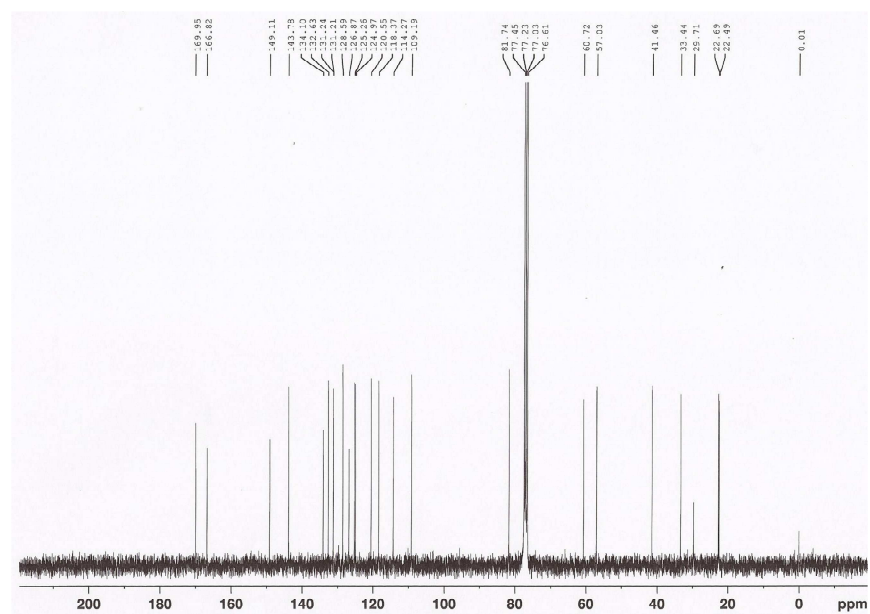


Figure S27. ^{13}C NMR spectrum of aszonalenin (CDCl_3 , 75.4 MHz).



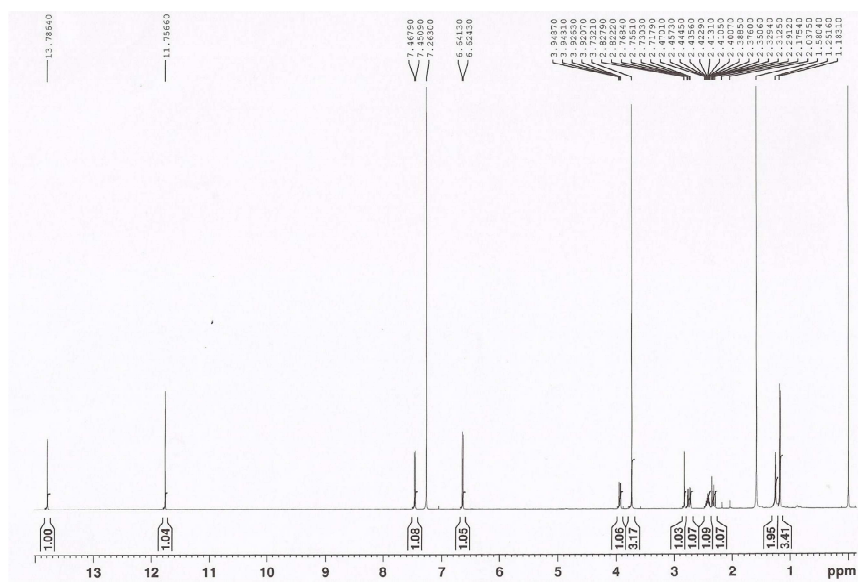


Figure S29. ^{13}C NMR spectrum of secalonic acid A (CDCl_3 , 75.4 MHz).

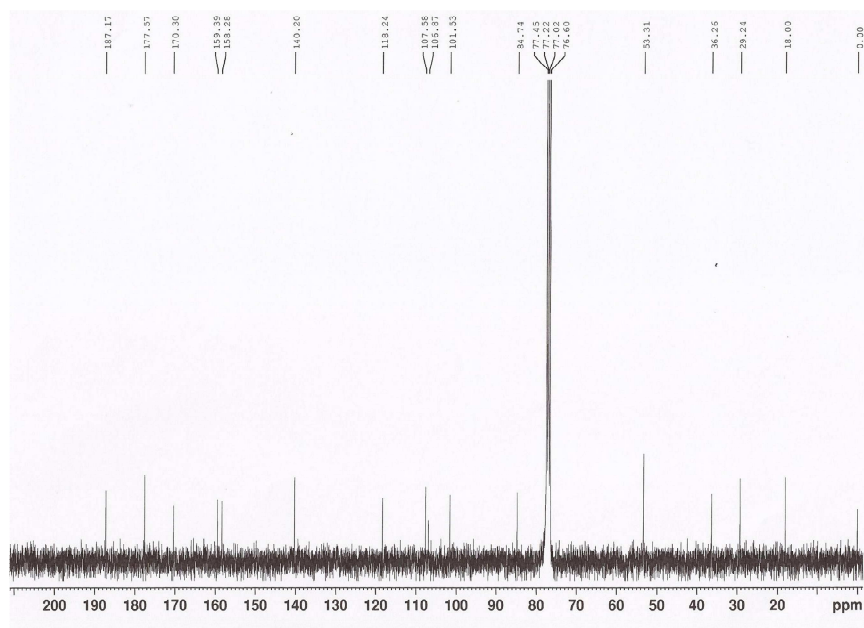


Figure S30. ^1H NMR spectrum of fellutanine A (DMSO, 300.13 MHz).

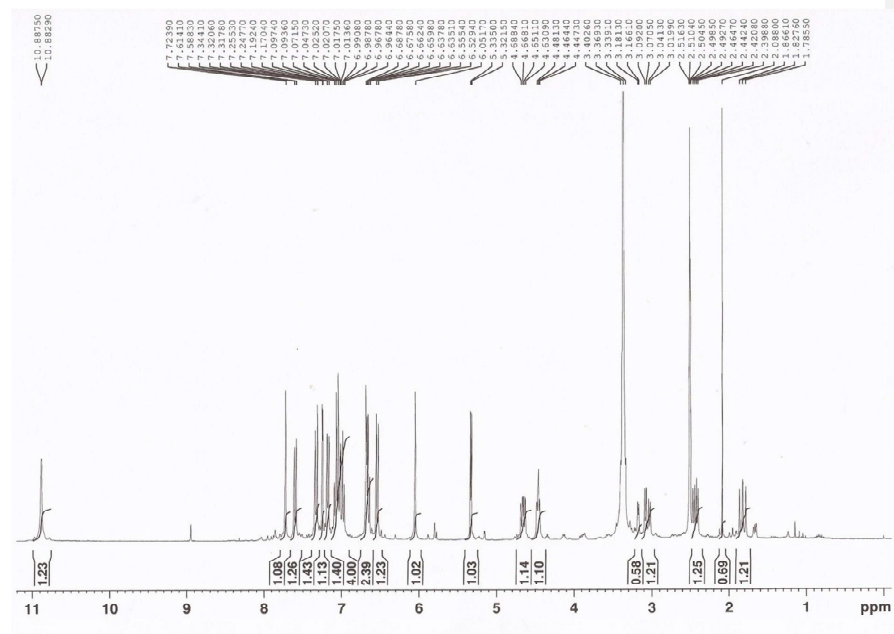


Figure S31. ^{13}C NMR spectrum of fellutanine A (DMSO, 75.4 MHz).

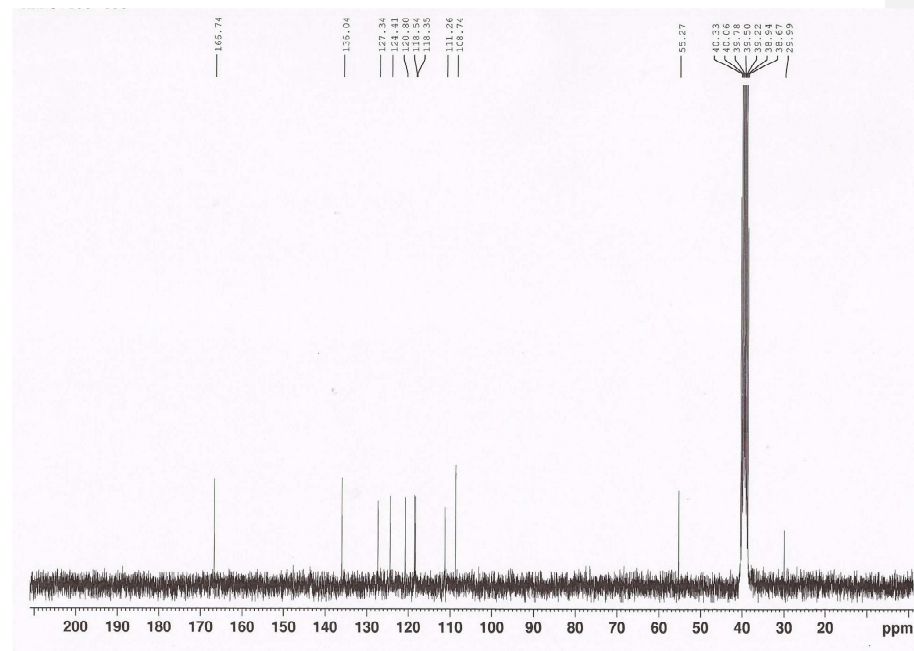


Figure S32. ^1H NMR spectrum of **1** (DMSO, 500.13 MHz).

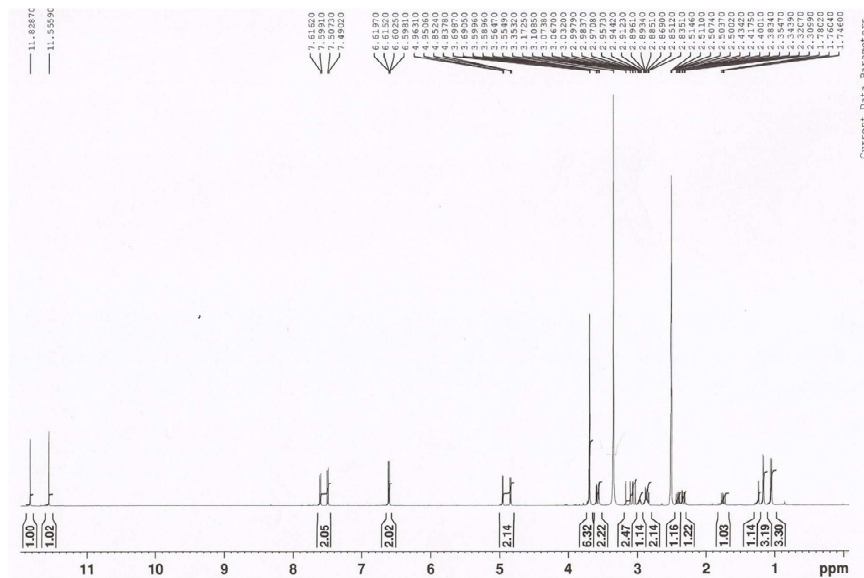


Figure S33. ^{13}C NMR spectrum of **1** (DMSO, 125.8 MHz).

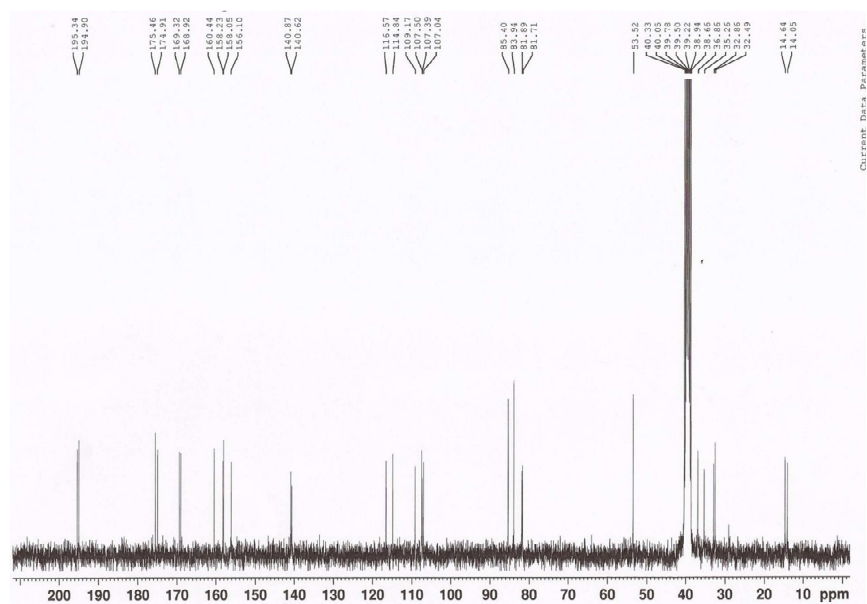


Figure S34. COSY spectrum of **1** (DMSO, 500.13 MHz).

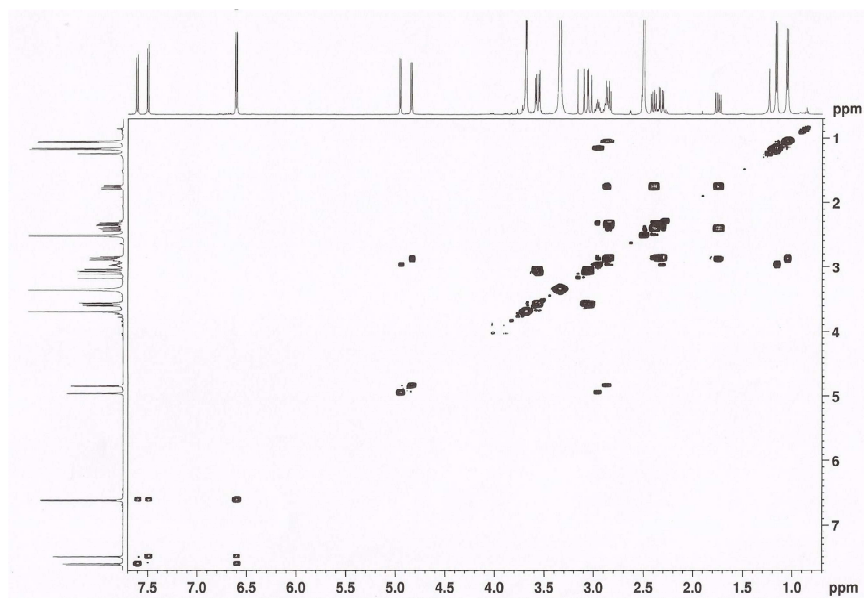
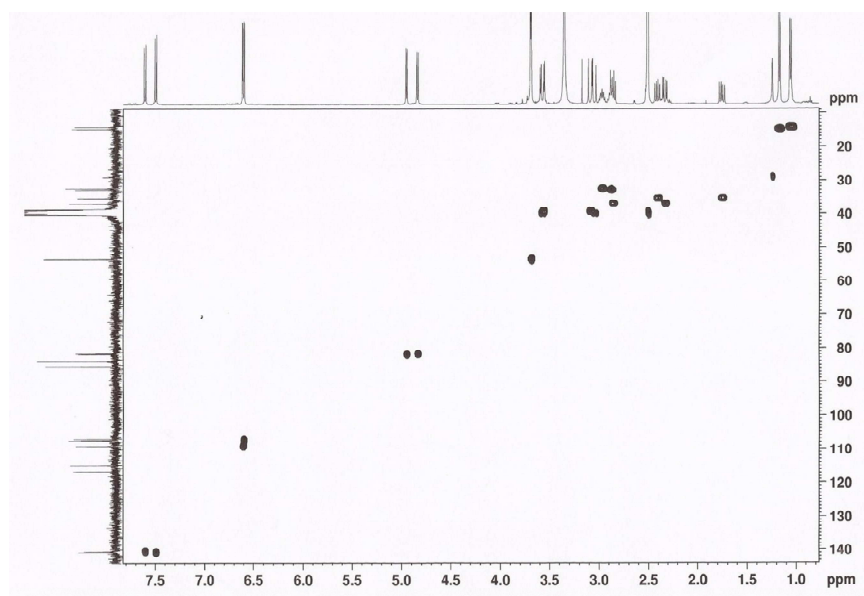


Figure S35. HSQC spectrum of **1** (DMSO, 500.13 MHz).



Chemical shifts (ppm) listed on the right side of the spectrum:

- 7.27256
- 6.35440
- 5.27450
- 5.26840
- 5.26440
- 5.26040
- 5.25640
- 5.25240
- 5.24840
- 5.24440
- 5.24040
- 5.23640
- 5.23240
- 5.22840
- 5.22440
- 5.22040
- 5.21640
- 5.21240
- 5.20840
- 5.20440
- 5.20040
- 5.19640
- 5.19240
- 5.18840
- 5.18440
- 5.18040
- 5.17640
- 5.17240
- 5.16840
- 5.16440
- 5.16040
- 5.15640
- 5.15240
- 5.14840
- 5.14440
- 5.14040
- 5.13640
- 5.13240
- 5.12840
- 5.12440
- 5.12040
- 5.11640
- 5.11240
- 5.10840
- 5.10440
- 5.10040
- 5.09640
- 5.09240
- 5.08840
- 5.08440
- 5.08040
- 5.07640
- 5.07240
- 5.06840
- 5.06440
- 5.06040
- 5.05640
- 5.05240
- 5.04840
- 5.04440
- 5.04040
- 5.03640
- 5.03240
- 5.02840
- 5.02440
- 5.02040
- 5.01640
- 5.01240
- 5.00840
- 5.00440
- 5.00040
- 4.99640
- 4.99240
- 4.98840
- 4.98440
- 4.98040
- 4.97640
- 4.97240
- 4.96840
- 4.96440
- 4.96040
- 4.95640
- 4.95240
- 4.94840
- 4.94440
- 4.94040
- 4.93640
- 4.93240
- 4.92840
- 4.92440
- 4.92040
- 4.91640
- 4.91240
- 4.90840
- 4.90440
- 4.90040
- 4.89640
- 4.89240
- 4.88840
- 4.88440
- 4.88040
- 4.87640
- 4.87240
- 4.86840
- 4.86440
- 4.86040
- 4.85640
- 4.85240
- 4.84840
- 4.84440
- 4.84040
- 4.83640
- 4.83240
- 4.82840
- 4.82440
- 4.82040
- 4.81640
- 4.81240
- 4.80840
- 4.80440
- 4.80040
- 4.79640
- 4.79240
- 4.78840
- 4.78440
- 4.78040
- 4.77640
- 4.77240
- 4.76840
- 4.76440
- 4.76040
- 4.75640
- 4.75240
- 4.74840
- 4.74440
- 4.74040
- 4.73640
- 4.73240
- 4.72840
- 4.72440
- 4.72040
- 4.71640
- 4.71240
- 4.70840
- 4.70440
- 4.70040
- 4.69640
- 4.69240
- 4.68840
- 4.68440
- 4.68040
- 4.67640
- 4.67240
- 4.66840
- 4.66440
- 4.66040
- 4.65640
- 4.65240
- 4.64840
- 4.64440
- 4.64040
- 4.63640
- 4.63240
- 4.62840
- 4.62440
- 4.62040
- 4.61640
- 4.61240
- 4.60840
- 4.60440
- 4.60040
- 4.59640
- 4.59240
- 4.58840
- 4.58440
- 4.58040
- 4.57640
- 4.57240
- 4.56840
- 4.56440
- 4.56040
- 4.55640
- 4.55240
- 4.54840
- 4.54440
- 4.54040
- 4.53640
- 4.53240
- 4.52840
- 4.52440
- 4.52040
- 4.51640
- 4.51240
- 4.50840
- 4.50440
- 4.50040
- 4.49640
- 4.49240
- 4.48840
- 4.48440
- 4.48040
- 4.47640
- 4.47240
- 4.46840
- 4.46440
- 4.46040
- 4.45640
- 4.45240
- 4.44840
- 4.44440
- 4.44040
- 4.43640
- 4.43240
- 4.42840
- 4.42440
- 4.42040
- 4.41640
- 4.41240
- 4.40840
- 4.40440
- 4.40040
- 4.39640
- 4.39240
- 4.38840
- 4.38440
- 4.38040
- 4.37640
- 4.37240
- 4.36840
- 4.36440
- 4.36040
- 4.35640
- 4.35240
- 4.34840
- 4.34440
- 4.34040
- 4.33640
- 4.33240
- 4.32840
- 4.32440
- 4.32040
- 4.31640
- 4.31240
- 4.30840
- 4.30440
- 4.30040
- 4.29640
- 4.29240
- 4.28840
- 4.28440
- 4.28040
- 4.27640
- 4.27240
- 4.26840
- 4.26440
- 4.26040
- 4.25640
- 4.25240
- 4.24840
- 4.24440
- 4.24040
- 4.23640
- 4.23240
- 4.22840
- 4.22440
- 4.22040
- 4.21640
- 4.21240
- 4.20840
- 4.20440
- 4.20040
- 4.19640
- 4.19240
- 4.18840
- 4.18440
- 4.18040
- 4.17640
- 4.17240
- 4.16840
- 4.16440
- 4.16040
- 4.15640
- 4.15240
- 4.14840
- 4.14440
- 4.14040
- 4.13640
- 4.13240
- 4.12840
- 4.12440
- 4.12040
- 4.11640
- 4.11240
- 4.10840
- 4.10440
- 4.10040
- 4.09640
- 4.0

Figure S38. ^{13}C NMR spectrum of **2** (CDCl_3 , 125.8 MHz).

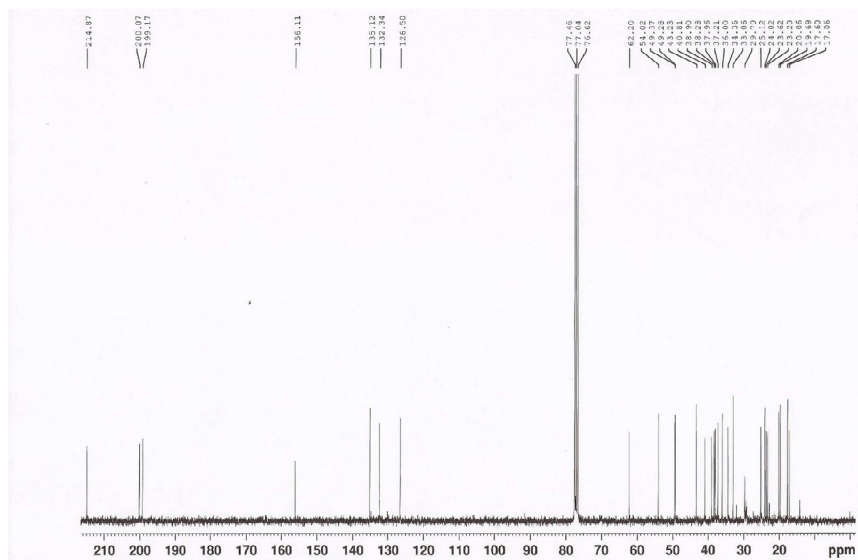


Figure S39. HSQC spectrum of **2** (CDCl_3 , 125.8 MHz).

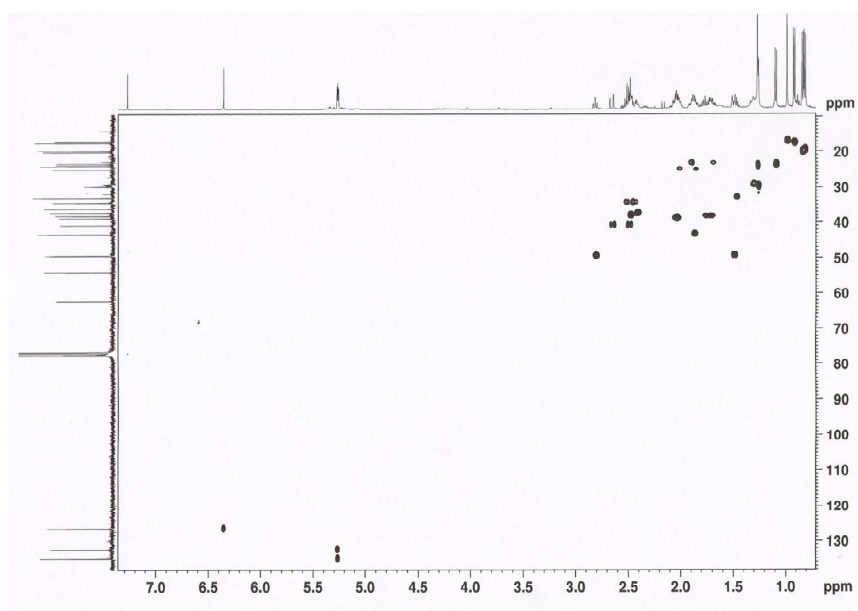


Figure S40. HMBC spectrum of **2** (CDCl₃, 125.8 MHz).

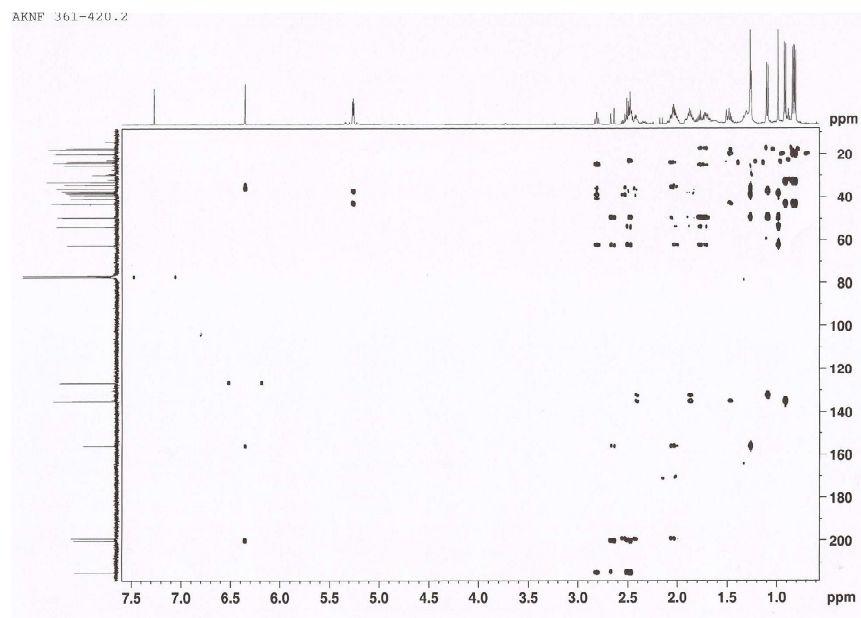


Figure S41. ¹H NMR spectrum of **3** (CDCl₃, 300.13 MHz).

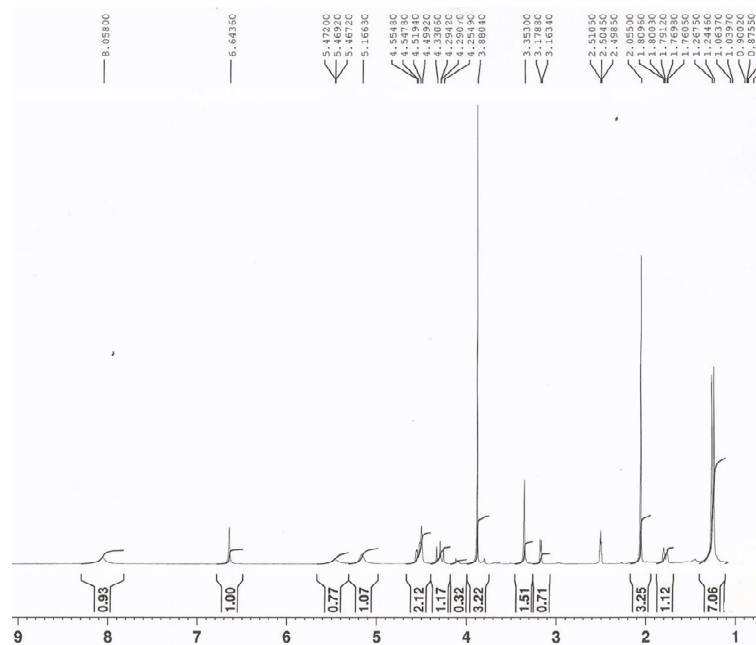


Figure S42. ^{13}C NMR spectrum of **3** (CDCl_3 , 75.4 MHz).

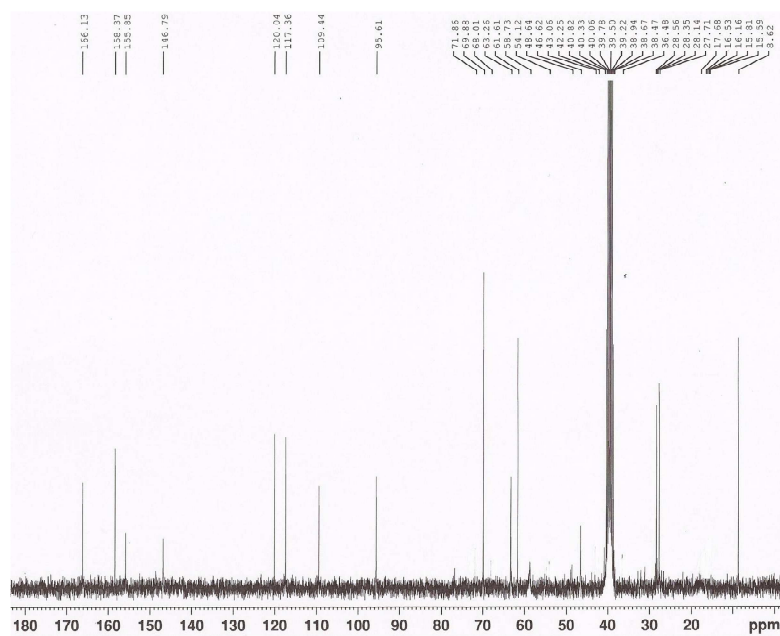


Figure S43. COSY spectrum of **3** (CDCl_3 , 300.13 MHz).

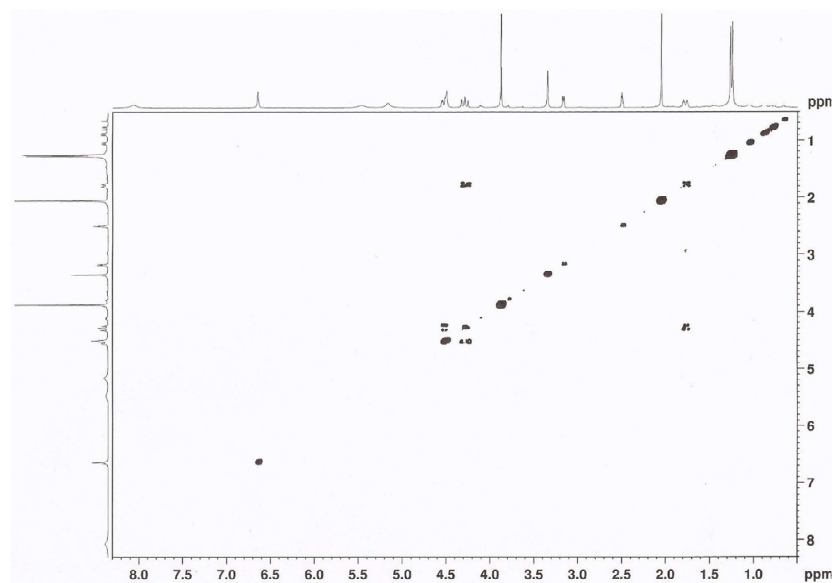


Figure S44. HSQC spectrum of **3** (CDCl₃, 300.13 MHz).

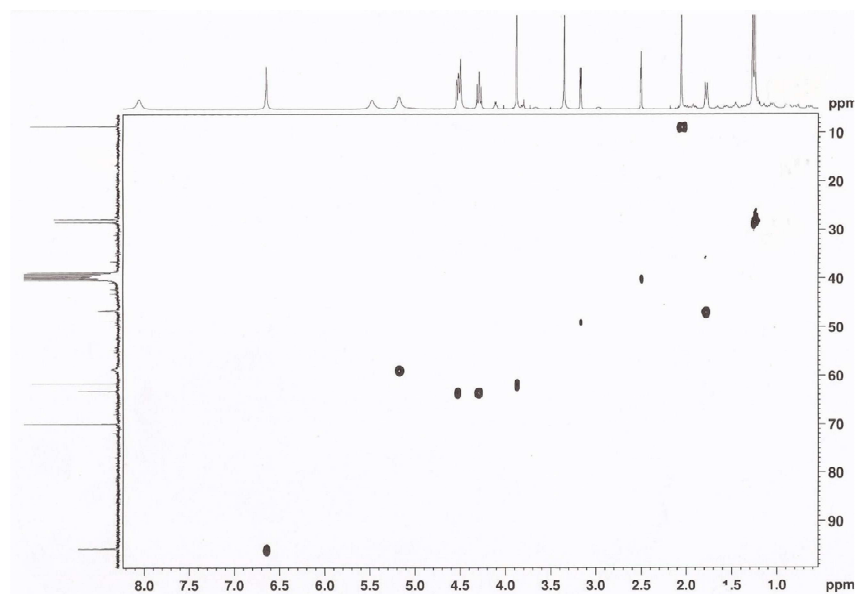


Figure S45. HMBC spectrum of **3** (CDCl₃, 300.13 MHz).

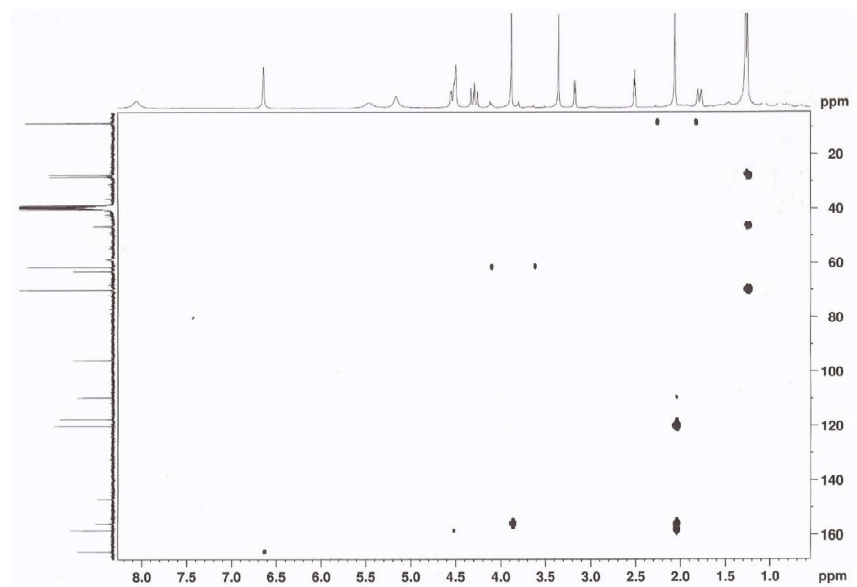


Figure S46. NOESY spectrum of **3** (CDCl₃, 300.13 MHz).

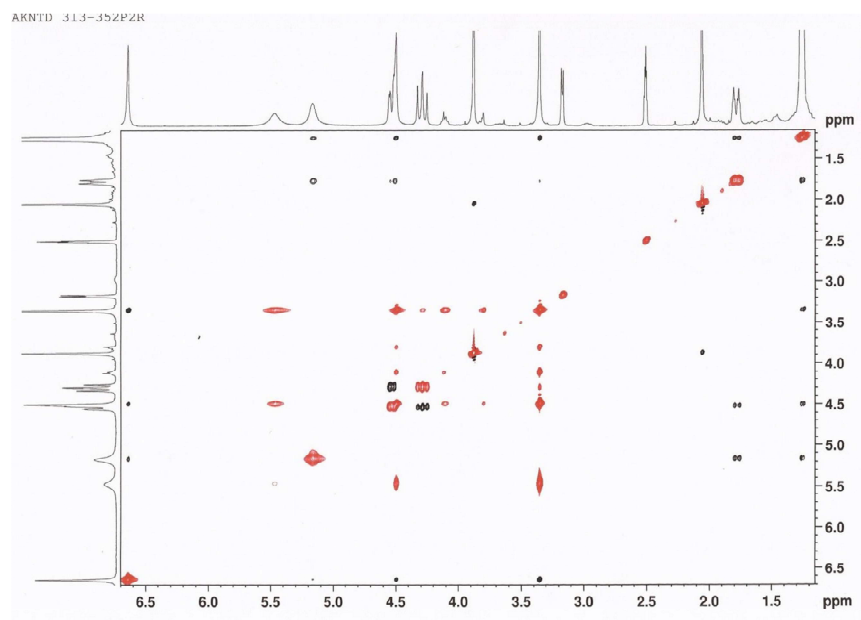


Figure S47. ¹H NMR spectrum of **4** (CDCl₃, 500.13 MHz).

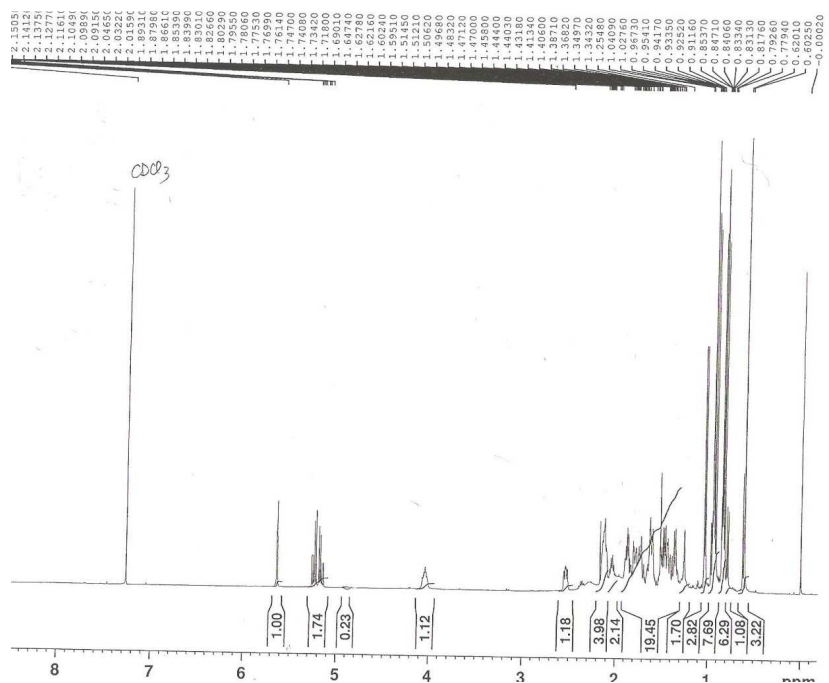


Figure 48. ^{13}C NMR spectrum of **4** (CDCl_3 , 125.4 MHz).

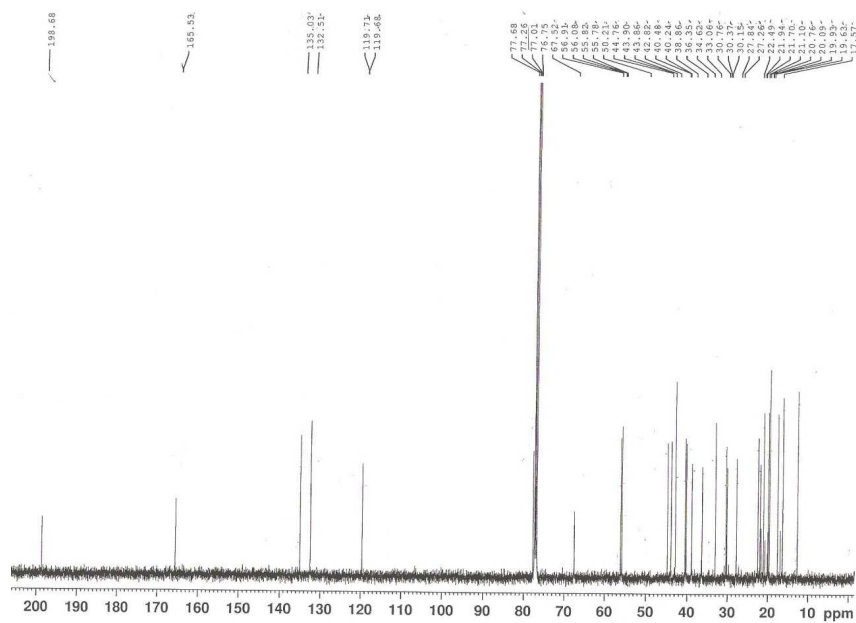


Figure S49. Ortep view of dankasterone A (**2**)

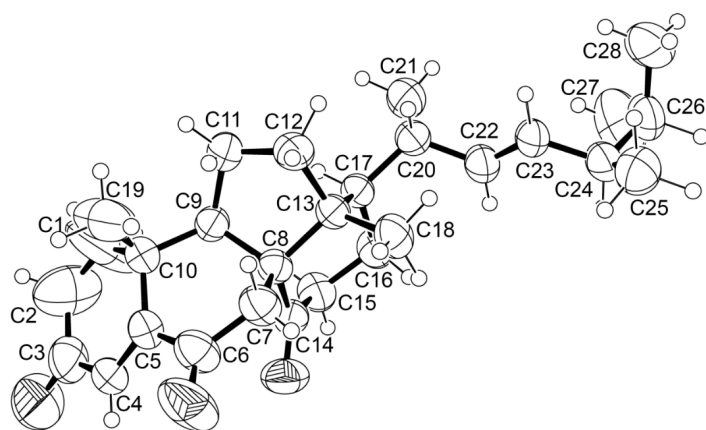


Table S1¹H and ¹³C NMR (CDCl₃, 500 MHz and 125 MHz) and HMBC assignment for **2**

Position	δ _C , type	δ _H , (J in Hz)	COSY	HMBC
1	38.9, CH ₂	2.05, m	H-2	C-3, 5
2	34.4, CH ₂	2.46, m	H-1	
3	199.2, CO	-		
4	126.5, CH	6.36, s	-	C-2, 5, 6, 10
5	156.1, C	-		
6	200.1, CO	-		
7α	40.8, CH ₂	2.49, d (16.9)	H-7β	C-6, 8, 13
β		2.66, dd (16.9, 1.4)	H-7α	C-5, 6, 8, 9
8	62.2, C	-		
9	49.3, CH	2.81, brt (9.0)	H-11	C-1, 7, 8, 10, 14, 19
10	36.0, C	-		
11	23.2, CH ₂	1.68, m	H-9, 12	
		1.88, m	H-9, 12	
12	38.3, CH ₂	1.71, m	H-11	
		1.76, m	H-11	
13	54.0, C	-		
14	214.9, CO	-		
15	38.0, CH ₂	2.47, m	H-16	C-14, 17
16	25.1 CH ₂	1.85, m	H-15, 17	
		2.05, m	H-15, 17	
17	49.4, CH	1.48, m	H-16, 20	
18	17.1, CH ₃	0.98, s	-	C-8, 12, 13,
19	24.0, CH ₃	1.26, s	-	C-1, 5, 9, 10
20	37.2, CH	2.41, m	H-17, 21, 22	C-22, 23
21	23.6, CH ₃	1.09, d (7.0)	H-20	C-17, 20, 23
22	135.1, CH	5.27, m	H-20, 23	C-24
23	132.3, CH	5.27, m	H-22, 24	C-20
24	43.2, CH	1.87, m	H-23, 26	
25	33.1, CH	1.46, m	H-24, 27, 28	
26	17.6, CH ₃	0.91, d (6.9)	H-24	C-22, 24, 25
27	20.1, CH ₃	0.83, d (6.8)	H-25	C-24, 25, 28
28	19.7, CH ₃	0.81, d (6.8)	H-25	C-24, 25, 27

Table S2¹H and ¹³C NMR (CDCl₃, 500 MHz and 125 MHz) of **4**.

Position	δ _C , type	δ _H , (J in Hz)	HMBC
----------	-----------------------	----------------------------	------

1	27.8, CH ₂	1.35, m	
		1.77, m	
2	30.4, CH ₂	1.61, m	
		1.79, m	
3	67.5, CH	4.04, q (6.4)	
4	36.4, CH ₂	1.61, m	
		1.79, m	
5	77.7, C	-	
6	198.7, CO	-	
7	119.7, CH	5.63, brs	C-5, 9, 14
8	165.5, C	-	
9	43.9, CH	2.53, ddd (11.8, 7.9, 2.2)	C-7, 8, 10, 11, 19
10	40.5, C	-	
11	21.9, CH ₂	1.61, m	
		1.72, m	
12	38.9, CH ₂	1.43, m	
		2.10, m	
13	44.8, C	-	
14	55.8, CH	2.13, m	
15	22-5, CH ₂	1.35, m	
		1.77, m	
16	30.2, CH ₂	1.42, m	
		1.87, m	
17	56.1, CH	1.35, m	
18	12.7, CH ₃	0.60, s	C-12, 13, 17
19	16.4, CH ₃	0.94, s	C-1, 5, 7
20	40.2, CH	2.03, m	
21	21.1, CH ₃	1.03, d (6.6)	C-17, 20
22	135.0, CH	5.16, dd (15.3, .6)	C-17, 20, 21, 24
23	132.5, CH	5.24, dd (15.3, 7.6)	C-20, 24, 25, 26
24	42.8, CH	2.03, m	
25	33.1, CH	1.47, m	
26	17.6, CH ₃	0.92, d (6.8)	C-23, 24, 25
27	19.9, CH ₃	0.84, d (6.9)	C-24, 25, 28
28	19.6, CH ₃	0.82, d (6.9)	C-24, 25, 27