

SUPPLEMENTARY MATERIAL

Xylastriasan A, a new cytochalasan from the fungus *Xylaria striata*

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Abstract

Xylastriasan A (**1**), a new cytochalasan alkaloid with a rare 5/6/6/5/6 pentacyclic skeleton, and ergosterol (**2**) were isolated from the ethanol extract of fruiting bodies of the fungus *Xylaria striata*. Their structures were determined by analysis of their spectroscopic data. Compound **1** exhibited cytotoxic activity against HEPG2, B16 and A549 cell lines with IC₅₀ values of 93.61, 85.61 and 91.58 μ M, respectively. Ergosterol (**2**) potentiated pentobarbital-induced sleep by not only increasing the number of falling asleep and prolonging sleeping time but also reducing sleep latency at a dosage of 5 mg/kg.

Keywords: *Xylaria striata*; cytochalasan; ergosterol; cytotoxic; hypnotic activity

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Figure S1. The structure of Xylastriasan A (1).

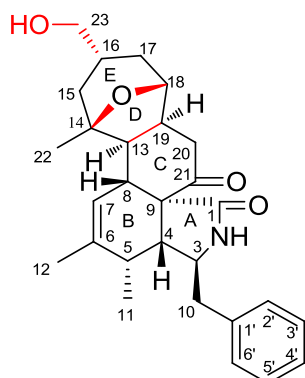
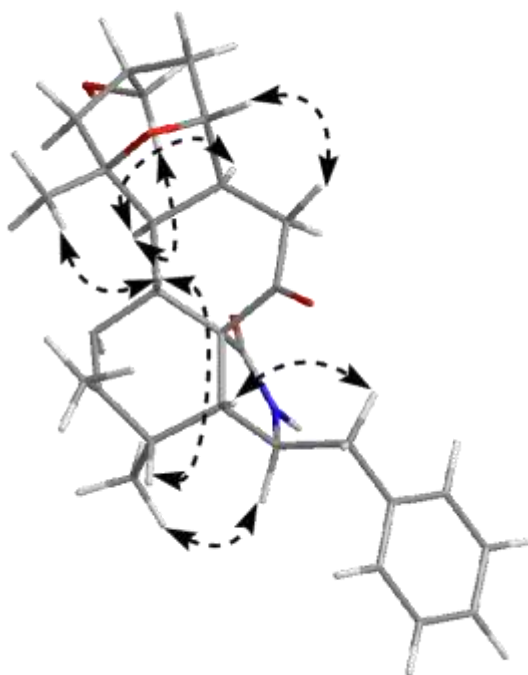
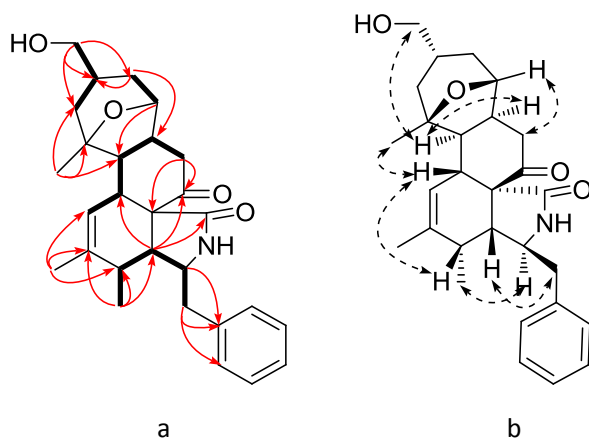


Figure S2. Main ^1H - ^1H COSY (thick lines), HMBC correlations (red arrows, from ^1H to ^{13}C) and NOE correlations (black arrows, from ^1H to ^1H) of compound 1.



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a: Main ^1H - ^1H COSY and HMBC correlations. b: Key NOE correlations. c: Chem3D model of key NOE correlations.

Figure S3. The HR-ESI spectrum of compound **1** in MeOD.

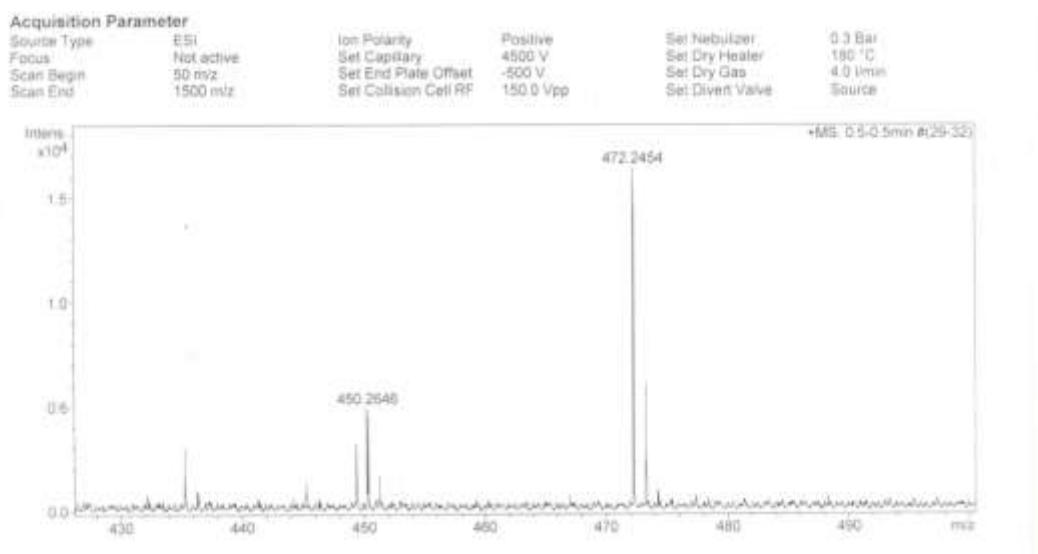


Figure S4. The IR spectrum of compound **1**.

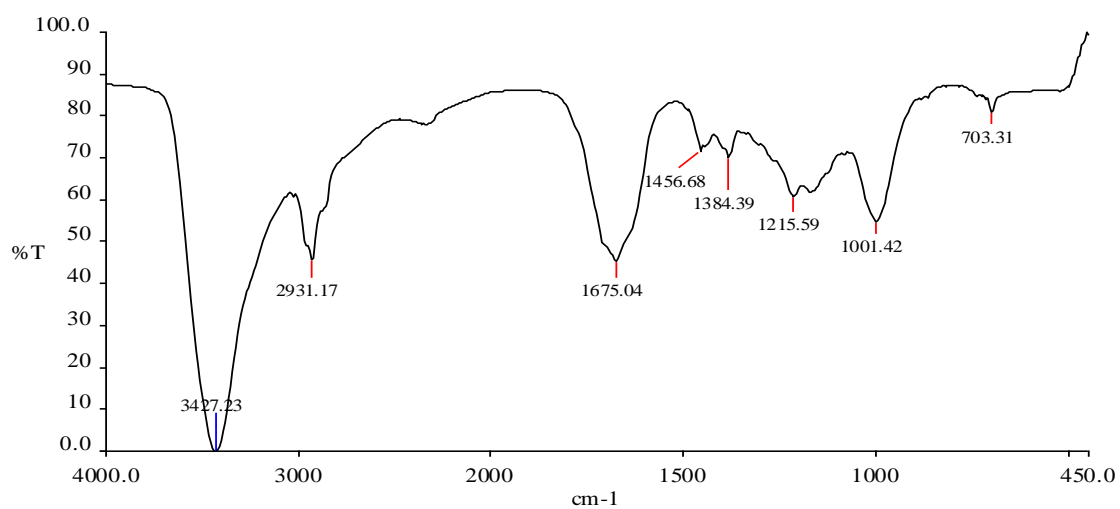


Figure S5. ^1H NMR spectrum of compound **1** in CD_3OD (400 MHz).

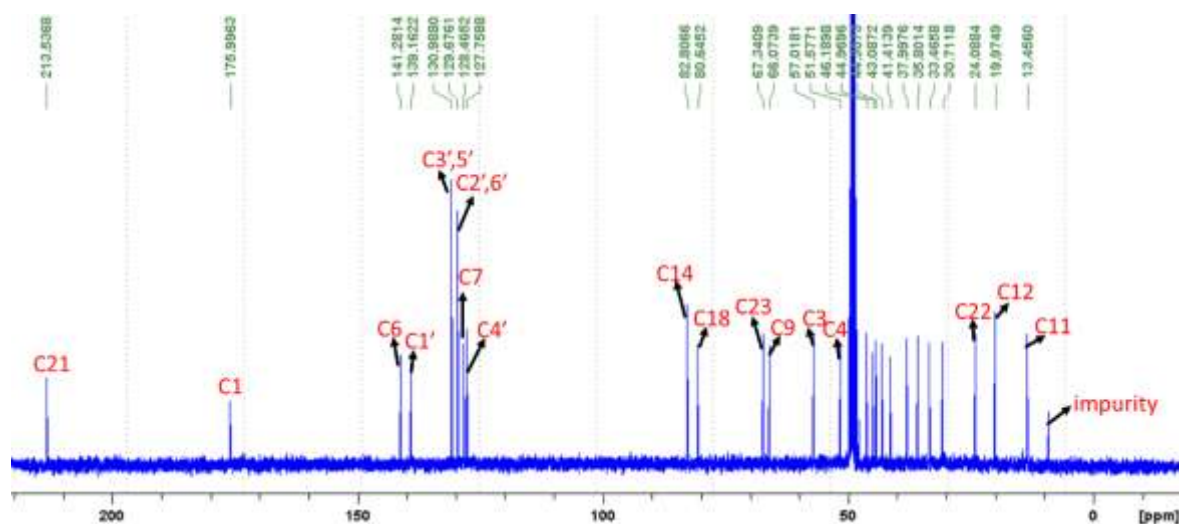


Figure S9. ^{13}C NMR spectrum (0–50 ppm) of compound **1** in CD_3OD (100 MHz).

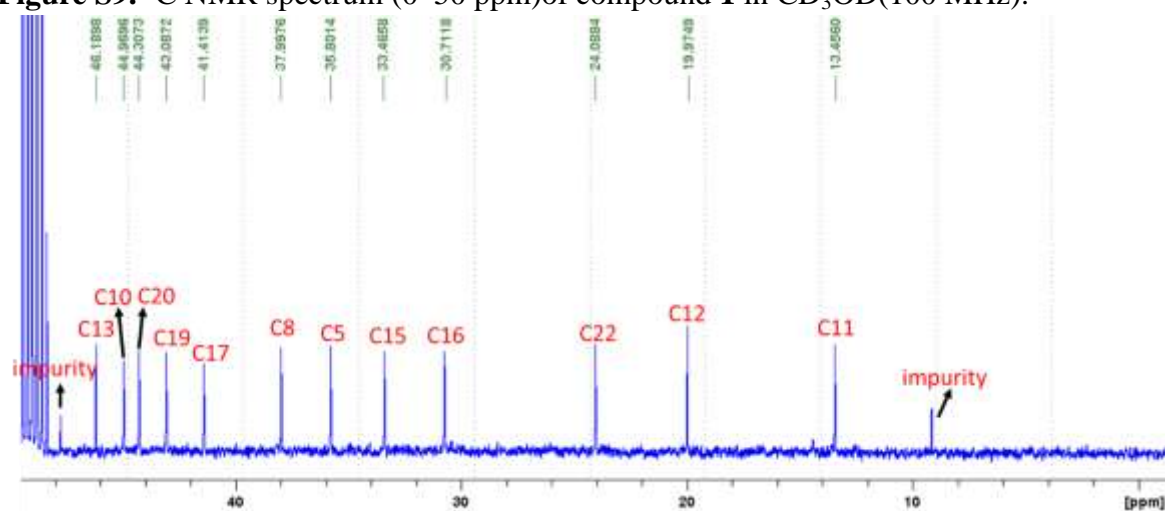


Figure S10. ^{13}C and DEPT NMR spectrum of compound **1** in CD_3OD (100 MHz).

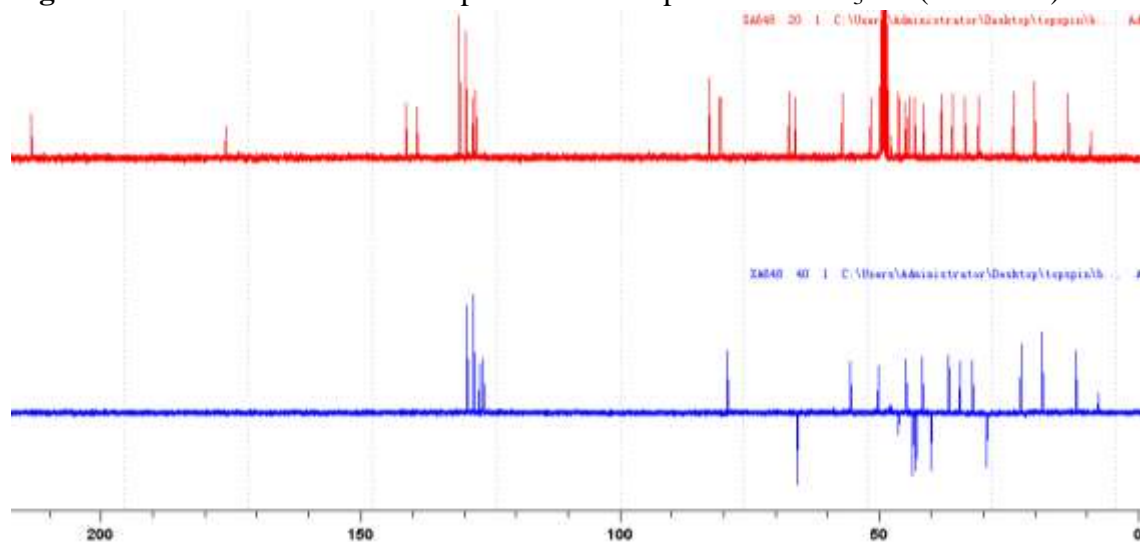


Figure S11. HSQC spectrum (δ_{H} 0–8.0 ppm and δ_{C} 0–160 ppm) of compound **1**.

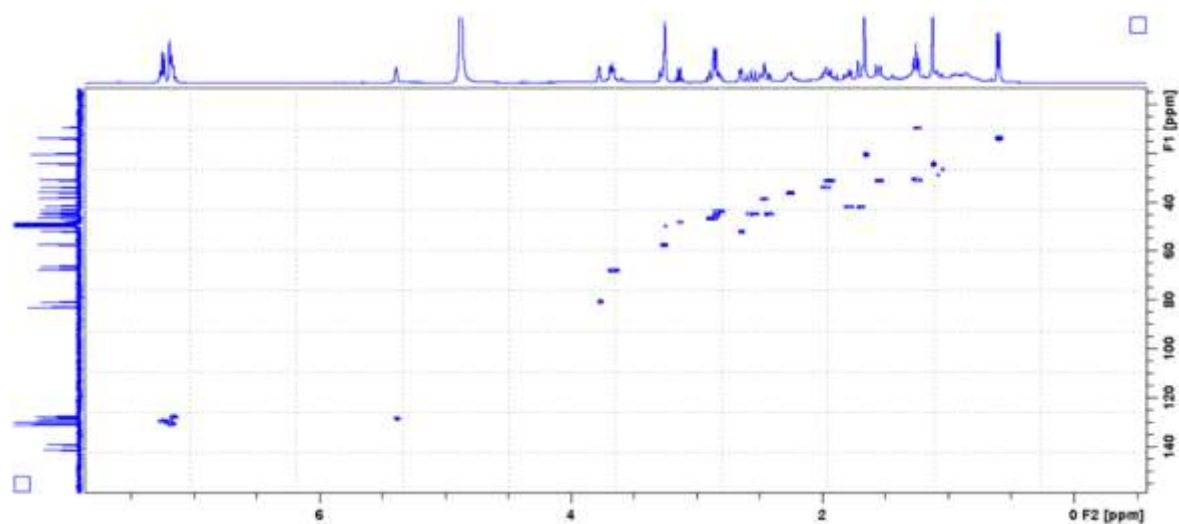


Figure S12. HSQC spectrum (δ_{H} 0.5–2.7 ppm and δ_{C} 0–60 ppm) of compound **1**.

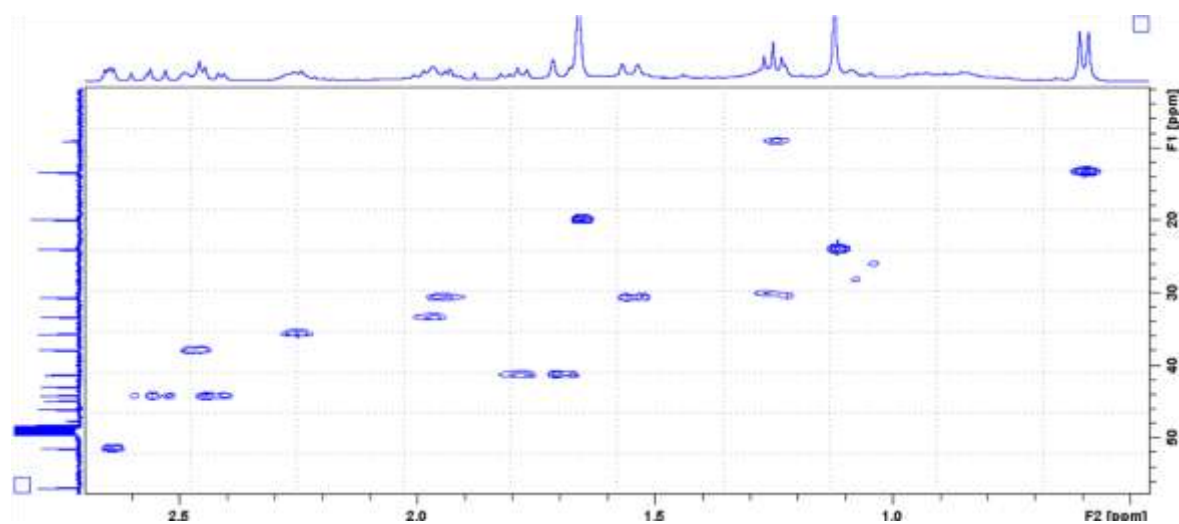


Figure S13. HSQC spectrum (δ_{H} 2.7–4.0 ppm and δ_{C} 30–85 ppm) of compound **1**.

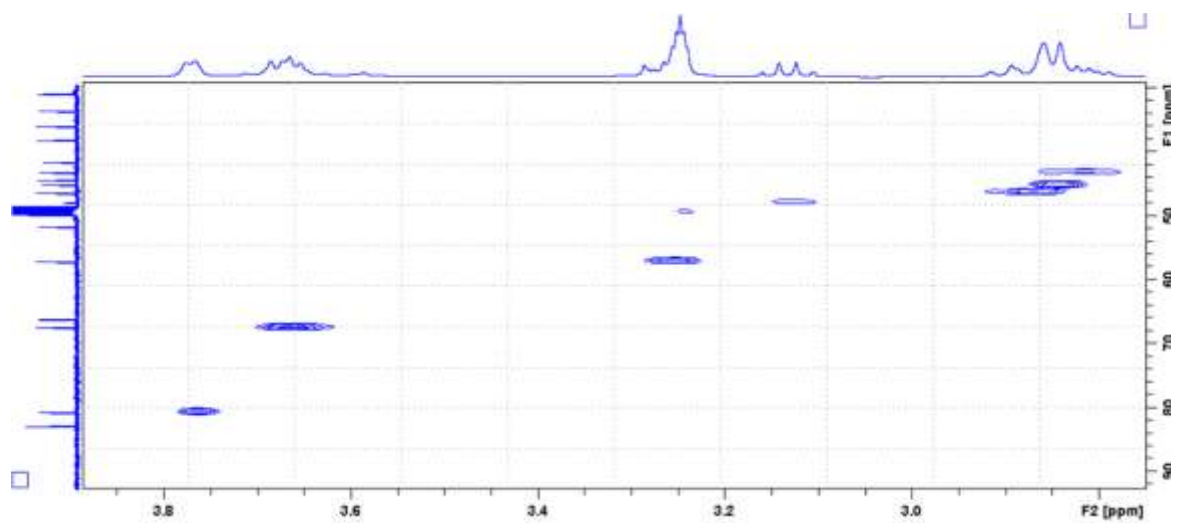


Figure S14. HSQC spectrum (δ_{H} 5.0–8.0 ppm and δ_{C} 100–150 ppm) of compound **1**.

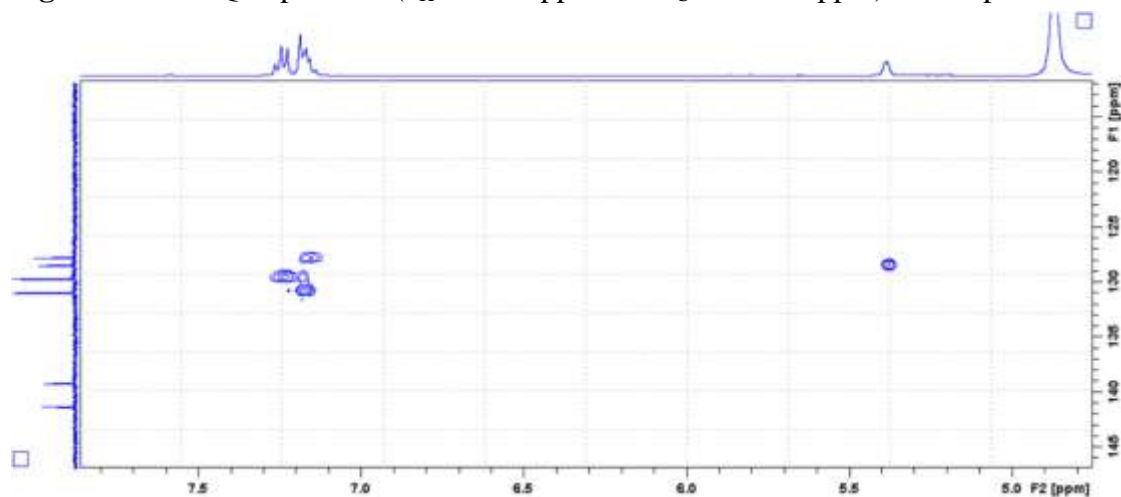


Figure S15. HMBC spectrum (δ_{H} 0–8.0 ppm and δ_{C} 0–210 ppm) of compound **1**.

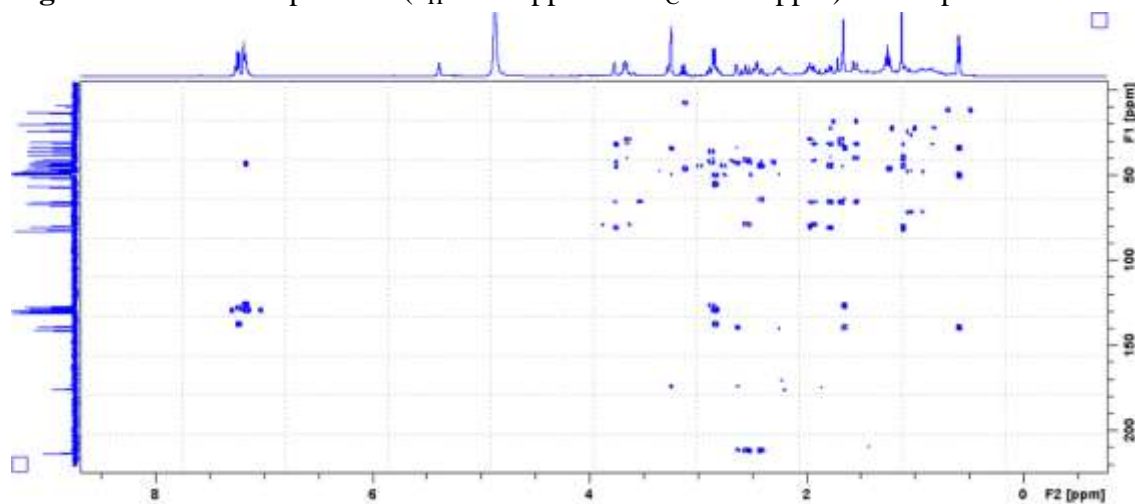


Figure S16. HMBC spectrum (δ_{H} 0–2.4 ppm and δ_{C} 0–100 ppm) of compound **1**.

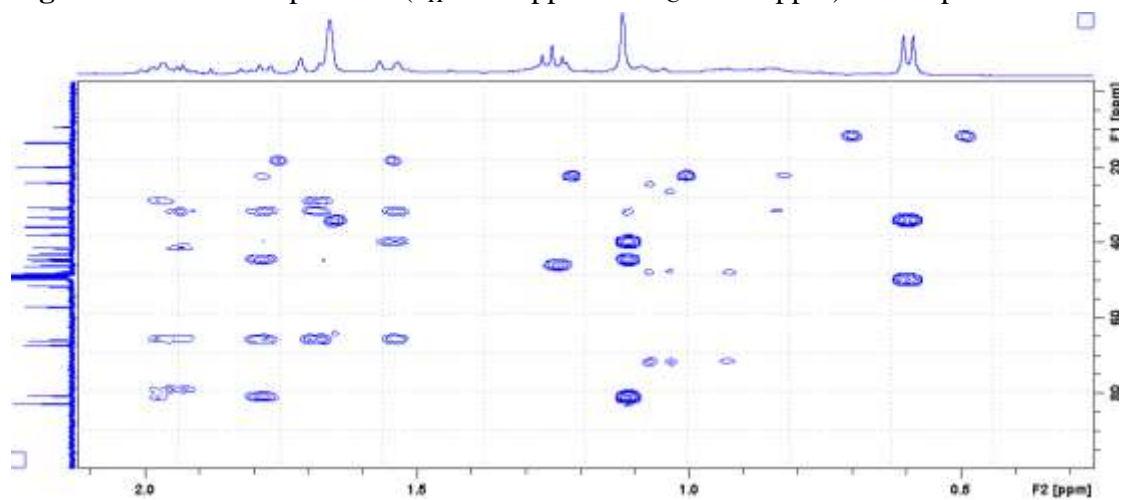


Figure S17. HMBC spectrum (δ_{H} 2.0–4.0 ppm and δ_{C} 0–100 ppm) of compound **1**.

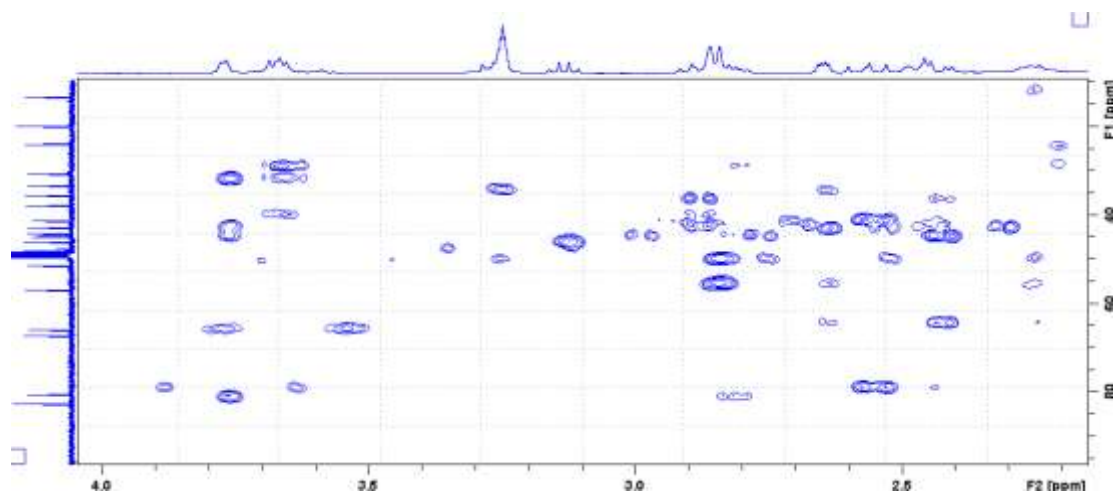


Figure S18. HMBC spectrum (δ_{H} 6.5–8.0 ppm and δ_{C} 30–190 ppm) of compound **1**.

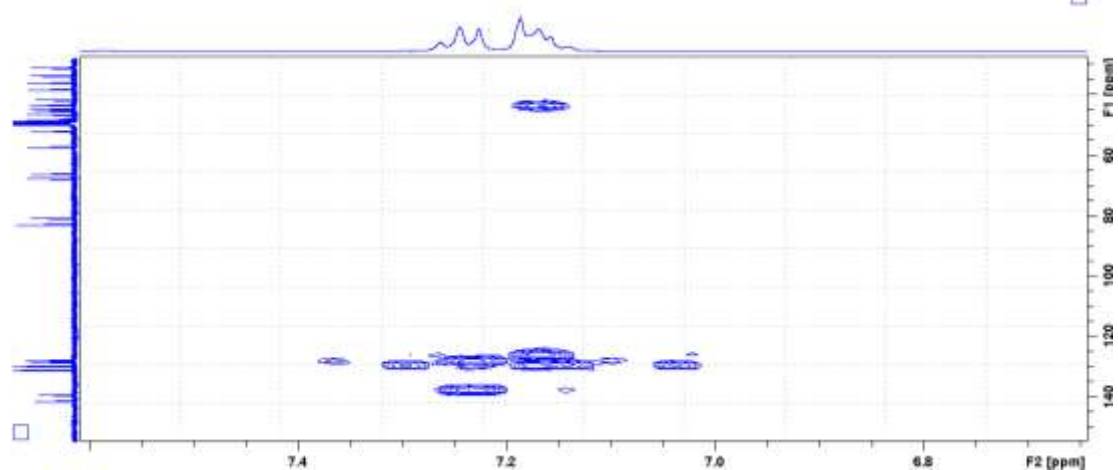


Figure S19. ^1H - ^1H COSY spectrum (0–8.0 ppm) of compound **1**.

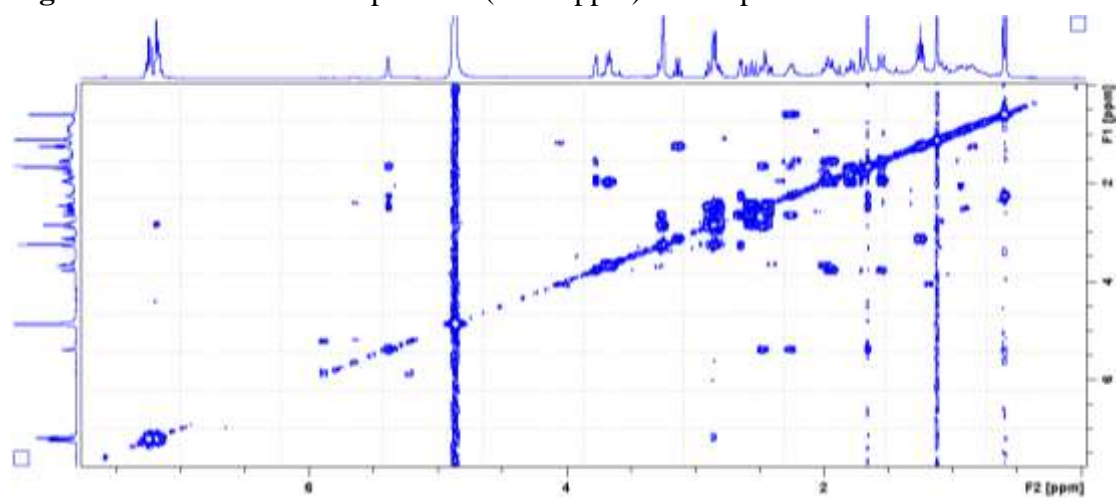


Figure S20. ^1H - ^1H COSY spectrum (δ_{H} 0–3.5 ppm) of compound **1**.

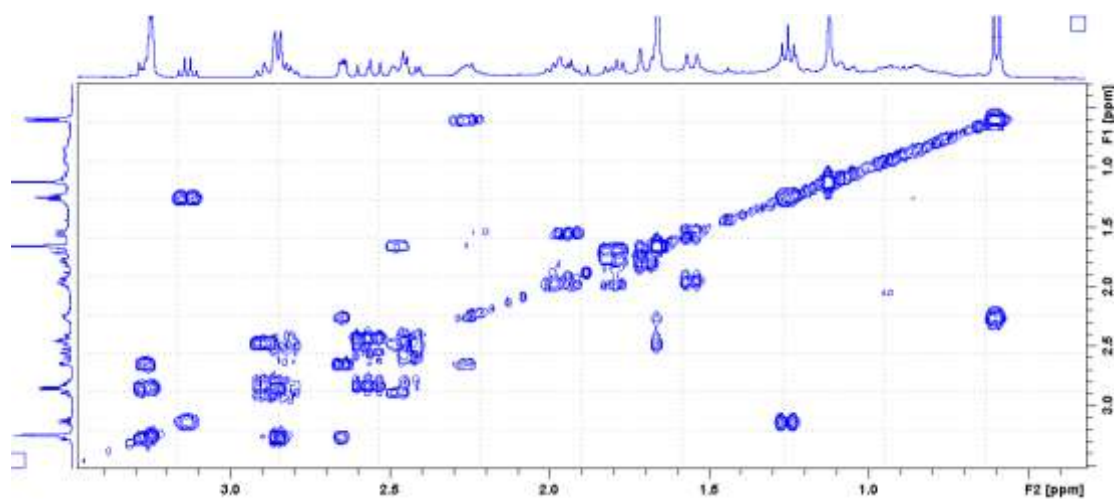


Figure S21. NOESY spectrum (0–8.0 ppm) of compound **1**.

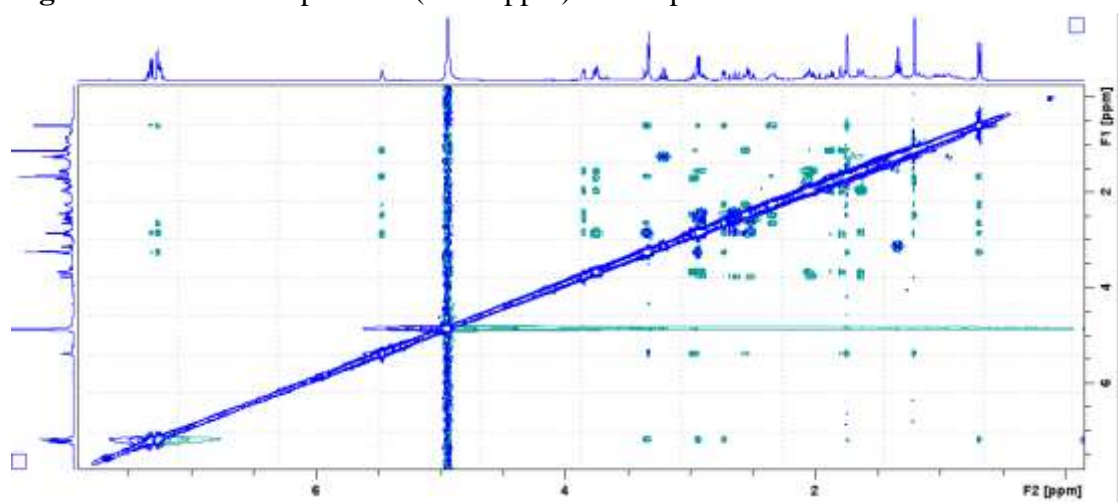


Figure S22. NOESY spectrum (0–4.0 ppm) of compound **1**.

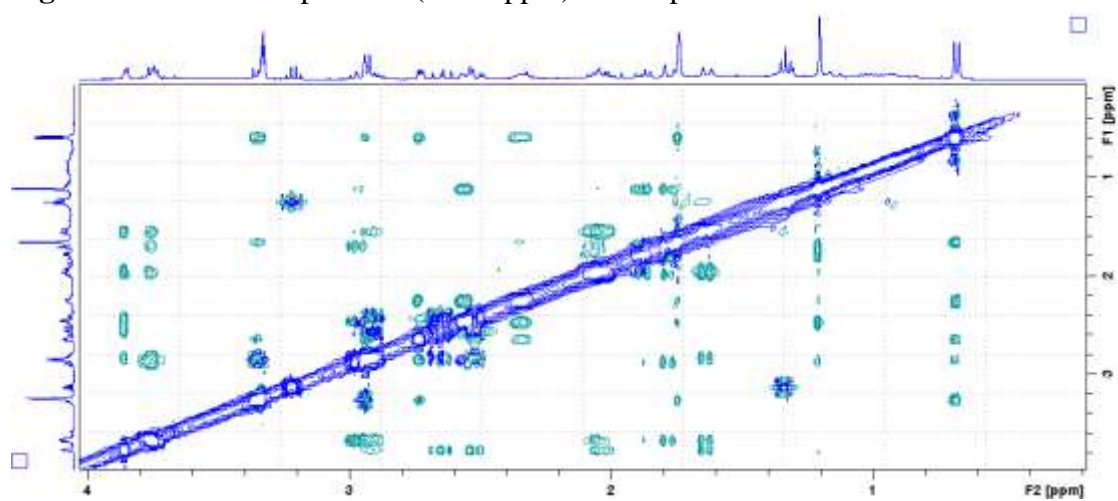


Figure S23. ^1H NMR spectrum of compound **2** in CD_3Cl (400 MHz).

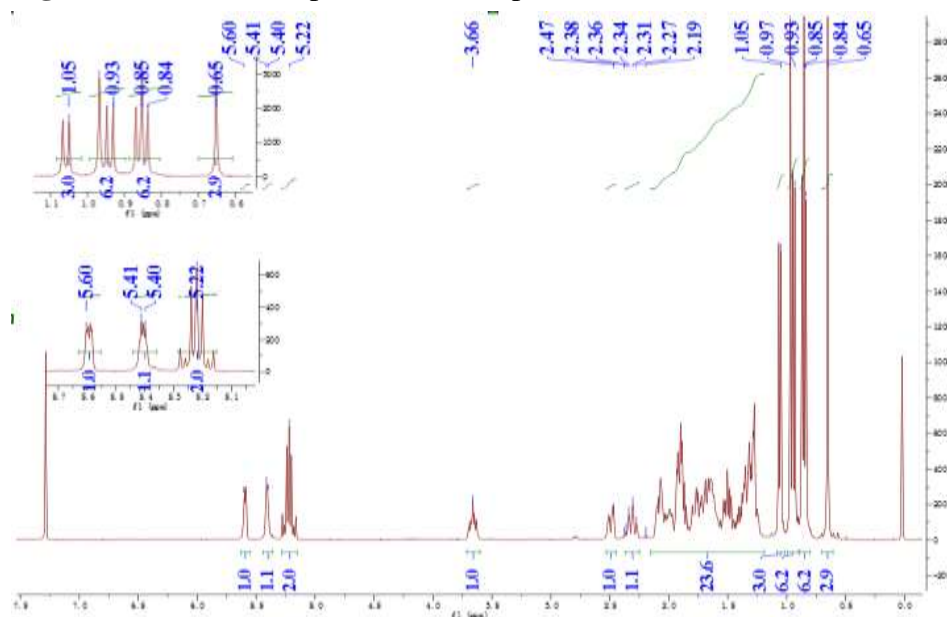
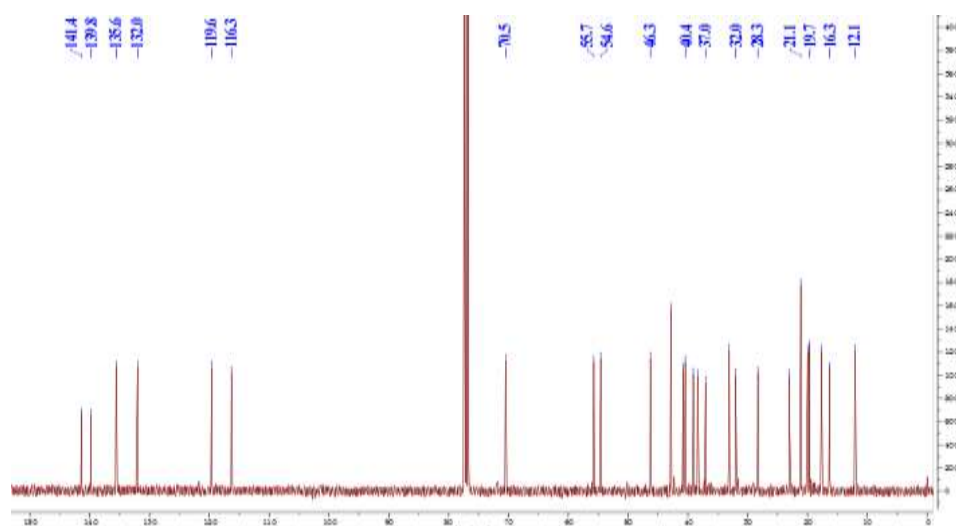


Figure S24. ^{13}C NMR spectrum of compound **2** in CD_3Cl (100 MHz).



^1H and ^{13}C NMR data of Ergosterol (**2**)

White needle crystal; ^1H NMR (400 MHz, CDCl_3) δ : 5.60 (1H, dd, $J = 5.5, 2.4$ Hz, H-6), 5.41 (1H, ddd, $J = 5.5, 2.8, 2.4$ Hz, H-7), 5.16-5.28 (2H, overlap, H-22 and H-23), 3.66 (1H, m, H-3 α), 2.47 (1H, ddd, $J = 14.3, 4.8, 3.2$ Hz, H-4 α), 2.31 (1H, t, $J = 12.2$ Hz, H-4 β), 1.05 (3H, d, $J = 6.6$ Hz, H-21), 0.97 (3H, s, H-19), 0.95 (3H, d, $J = 8.1$ Hz, H-28), 0.86 (3H, d, $J = 6.6$ Hz, H-27), 0.85 (3H, d, $J = 6.6$ Hz, H-26), 0.65 (3H, s, H-18). ^{13}C NMR (100 MHz, CDCl_3) δ : 141.4 (C-8), 139.8 (C-5), 135.6 (C-22), 132.0 (C-23), 119.6 (C-6), 116.3 (C-7), 70.5 (C-3), 55.74 (C-17), 54.6 (C-14), 46.3 (C-9), 42.8 (C-24), 40.8 (C-4), 40.4 (C-20), 39.1 (C-12), 38.4 (C-1), 37.0 (C-10), 33.1 (C-25), 32.0 (C-2), 28.3 (C-16), 23.0 (C-15), 21.1 (C-21), 20.0 (C-26), 19.7 (C-27), 17.6 (C-28), 16.3 (C-19), 12.1 (C-18).

Table S1. ^1H and ^{13}C NMR, HMBC, NOE data (^1H NMR 400 MHz; ^{13}C NMR 100 MHz, in methanol-*d*4) for compound **1**.

Position	δ_{H} mult. <i>J</i> (Hz)	δ_{C} (type)	HMBC	NOE
1		176.0 C		
3	3.28 m	57.0 CH	C-1, C-5	H-11
4	2.65 dd (5.6, 2.5)	51.6 CH	C-1, C-3, C-5, C-6, C-10, C-21	H-10
5	2.26 m	35.8 CH	C-3, C-4, C-6, C-11	H-8
6		141.3 C		
7	5.38 s	128.5 CH		
8	2.48 d (11.1)	38.0 CH		H-5, H-22
9		66.1 C		
10	2.85 d (7.6)	45.0 CH ₂	C-3, C-4, C-1', C-2', C-6'	H-4
11	0.59 d (6.9)	13.5 CH ₃	C-4, C-5, C-6	H-3
12	1.66 s	20.0 CH ₃	C-5, C-6, C-7	
13	2.90 dd (11.1, 8.8)	46.2 CH	C-7, C-8, C-20	H-19, H-23
14		82.8 C		
15Ha	1.55 d (12.4)	30.7 CH ₂	C-16, C-17, C-23	
Hb	1.95 m			
16	1.97 m	33.5 CH	C-14, C-15, C-17, C-23	
17Ha	1.69 d (14.1)	41.4 CH ₂	C-15, C-16, C-18, C-23	
Hb	1.79 dd (14.1, 7.3)			
18	3.77 d (3.6)	80.6 CH	C-13, C-14, C-16, C-20	H-20a, H-20b
19	2.81 m	43.1 CH	C-14	H-13
20Ha	2.43 dd (16.3, 5.0)	44.3 CH ₂	C-9, C-13, C-18, C-19,	H-18

Hb	2.56 dd (16.3, 12.6)		C-21	H-18
21		213.5 C		
22	1.12 s	24.1 CH ₃	C-13, C-14, C-15, C-16	H-8
23	3.67 m	67.3 CH ₂	C-15, C-16, C-17, C-18	H-13
1'		139.2 C		
2', 6'	7.30 overlap	129.7 CH		
3', 5'	7.24 overlap	131.0 CH		
4'	7.20 overlap	127.8 CH		

Table S2. Comparison of the ¹³C NMR data of rings A and B of **1** (¹³C NMR 100 MHz, in methanol-*d*₄) and cytochalasa-6,13,19-triene-1,21-dione-1g-hydroxy-16,18-dimethyl-10-phenyl (the compound compared, ¹³C NMR 100 MHz, in CDCl₃).

Position	Xylastriasan A δ_C (type)	The compound compared δ_C (type)
1	176.0 C	174.4 C
3	57.0 CH	54.8 CH
4	51.6 CH	48.6 CH
5	35.8 CH	34.8 CH
6	141.3 C	140.1 C
7	128.5 CH	125.6 CH
9	66.1 C	68.8 C
10	45.0 CH ₂	45.1 CH ₂
11	13.5 CH ₃	13.4 CH ₃
12	20.0 CH ₃	19.9 CH ₃