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Terpenoids and steroids from *Lappula anocarpa*

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A novel nor-terpenoid, named lappulanocarpine A and a known terpenoid derivative, nine known steroids and three known long chine esters were isolated from the alcoholic extract of the whole plant of *Lappula anocarpa*. The novel structure was characterized by means of spectral methods including 1D-, 2D-NMR and HR-ESIMS and the known compounds were identified on the basis of comparing their NMR data with those of the corresponding compounds in the literature.

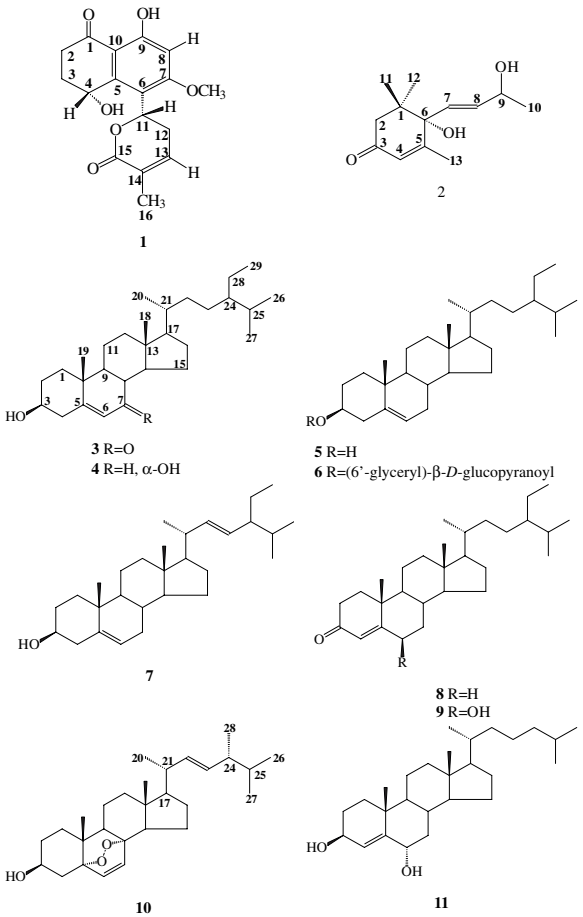
1. Introduction

The genus *Lappula* (Boraginaceae) consists of about 61 species distributed throughout the world, particularly, in the dry and desert zone. Among them, *L. myosotis* and *L. heteracantha* have been used as an important folk medicine for anti-inflammatory and anti-bacteria agents (Wu et al. 1999; Ma et al. 2003). In order to find bioactive principles, the chemical constituents of *Lappula anocarpa* C. J. Wang were investigated for the first time, and thirteen known compounds and a new compound were obtained from the alcoholic extract of this species. In this paper, we report the isolation and structural elucidation of a new and a known terpenoid derivative, along with 9 known steroids and 3 long chine esters.

2. Investigations, results and discussion

From the alcoholic extract of the whole plant of *Lappula anocarpa* C. J. Wang, a novel terpenoid, named lappulanocarpine A (**1**), together with a known terpenoid derivative, blumenol A (**2**) (Bhakuni et al. 1974; Çahş et al. 2002; Galbraith et al. 1973; Gonzáles et al. 1994; Weiss et al. 1973), nine known steroids, 7-oxositosterol (**3**) (Greca et al. 1990), 7α-hydroxysitosterol (**4**) (Greca et al. 1990; Yoshiyasu et al. 1988), β-sitosterol (**5**) (Greca et al. 1990; Yoshiyasu et al. 1988), β-sitosterol-3-(6′-glyceryl)-D-glucopyranoside (**6**) (Greca et al. 1990; Shi et al. 1997), stigmasterol (**7**), enone (**8**) (Greca et al. 1990; Jamaluddin et al. 1995), 6β-hydroxyenone (**9**) (Greca et al. 1990), 5α, 8α-epidioxy-methylcholesta-6, 22-dien-3β-ol (**10**) (Gunatilaka et al. 1981), 3β, 6α-dihydroxy-4-ene-cholestane (**11**) (You et al. 1993), as well as three known long chine esters, *n*-hexadecanol (**12**) (Sadtler 1969; Sadtler 1977; Zhang et al. 2003), glyceryl palmitate (**13**) (Du et al. 2002), glyceryl linolentate (**14**) (Shi et al. 1997) were isolated. The structures of the known compounds were confirmed by comparing their properties (melting point, MS, IR, ^{1}H NMR and ^{13}C NMR) with the reported values in the literature.

Compound **1** was obtained as a brown gum, $[\alpha]_D^{25}$ −158° (C, 0.25, $CHCl_3$). The molecular formula was assigned as $C_{17}H_{18}O_6$ by HR-ESIMS spectral data ($[M + NH_4]^+$ at m/z 336.1454; Calcd. 336.1442), ^{13}C NMR and DEPT NMR experiments showed 2 × CH_3, 3 × CH_2, 4 × CH and



The molecular structures of compound **1**–**11**

Table 1: ^{1}H-(400.13 MHz) and ^{13}CNMR (100.62 MHz) data, ^{1}H-^{1}H COSY and HMBC for compound 1

No.	δ_H	δ_C	COSY	HMBC
1	–	205.42 s	–	H_2-2, H_2-3
2α	3.07, ddd (17.6, 11.6, 4.8)	33.48 t	H-2β, H_2-3	H-3, H-4
2β	2.56, dt_(17.6, 4.4)		H-2α, H_2-3	
3α	2.56, dt (17.6, 4.4)	28.90 t	H_2-2, H-3β	H_2-2
3β	2.26, m		H_2-2, H-3α, H-4	
4β	5.26, t (4.4)	61.61 d	H-3β	H_2-2, H_2-3
5	–	115.25 s	–	H-3α, H-4β, HO-9
6	–	130.90 s	–	H-4, H-8, H-12α
7	–	146.95 s	–	H-8, CH_3O-7
8	7.43, s	117.26 d	CH_3O-7, HO-9, H-11	–
9	–	152.13 s	–	H-8, HO-9
10	–	127.96 s	–	H-8, HO-9
11β	5.78, dd (12.4, 3.6)	74.01 d	H-8, H_2-12	H-8, H-13
12α	2.35, m	30.44 t	H-11β, H-12β, H-13	H-13
12β	2.82, ddd (18.4, 5.6, 4.0)		H-11β, H-12α, H-13	
13	6.67 brd (6.4)	139.52 d	H_2-12, H_3-16	H_2-12, H_3-16
14	–	128.45 s	–	H_2-12, H_3-16
15	–	165.91 s	–	H-13, H_3-16
16	1.98, brs	17.00 q	–	H-13
7-OCH_3	3.89, s	56.57 q	H-8	–
9-OH	12.24, s	–	H-8	–

Assignments were aided by spin splitting patterns, DEPT, HMQC, HMBC experiments, and chemical shift values (δ). The δ values are in ppm and are referenced to either the residual $CHCl_3$ (7.26 ppm) or $CDCl_3$ (77.0 ppm) signals

8 × C. Its UV spectrum showed bands at 210 nm (log ε 4.65) and 368 nm (log ε 3.99). The IR spectrum (film) indicated the presence of hydroxyl (3452 cm^{-1}) and carbonyl (1722 cm^{-1}) and phenyl groups and double bond (1643, 1594 cm^{-1}). The ^{1}H NMR spectrum (Table 1) indicated the presence of a methyl group (δ 1.98, brs, 3 H) linking to double bond, a methoxyl group (δ 3.89, s, 3 H), a phenolic hydroxyl group (δ 12.24, s, 1 H) and two proton signals for double bond (δ 7.43, s, 1 H; 6.67, brd, 1 H, J = 6.4 Hz), as well as two proton signals due to oxymethine (δ 5.26, t, 1 H, J = 4.4 Hz and 5.78, dd, 1 H, J = 12.4, 3.6 Hz). In the ^{13}C NMR spectrum, there were a ketone carbonyl (δ 205.42), an α, β-unsaturated δ-lactone carbonyl (δ 165.91) groups, a typical methoxyl group and eight signals due to one penta-substituted phenyl ring and one olefinic bond (Table 1).

In the ^{1}H-^{1}H COSY spectrum of **1**, there were some significant cross-peaks (Table 1), suggesting the presence of two partial structures (see Fig. 1). These substituents and partial structures could be put together by key relative peaks in HMBC (Table 1), such as C-6 (δ 130.90) with H-4 (δ 5.26), H-8 (δ 7.43), and H-12α (δ 2.82); C-7 (δ 146.95) with H-8 and 7-OCH_3 (δ 3.89); C-9 (δ 152.13) with H-8 and 9-OH (δ 12.24); C-15 (δ 165.91) with H-13 (δ 6.67) and H_3-16 (δ 1.98) (see Fig. 2). The relative configuration of two chiral carbons (C-4 and C-11) in the molecule **1** can also be deduced by couple constants: $J_{4\beta,\,3\alpha} = J_{4\beta,\,3\beta} = 4.4$ Hz, $J_{11\beta,\,12\alpha} = 12.4$ Hz and $J_{11\beta,\,12\beta} = 3.6$ Hz in the ^{1}H NMR, suggesting the 4-OH for α-oriented and 11-H for β-oriented (see Fig. 3). Thus, the structure of compound **1** with a nor-carbon terpene skeleton was elucidated as shown and named as lappulanocarpine A after its resource (*Lappula anocarpa*).

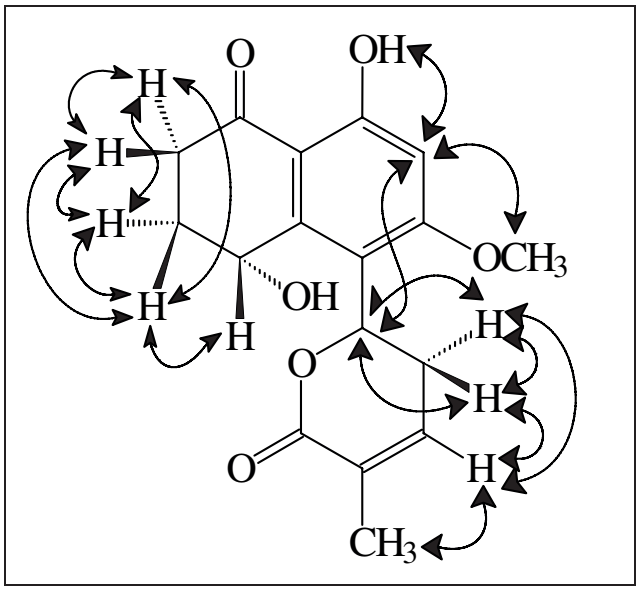


Fig. 1: Cross peaks in COSY of **1** (H and H)

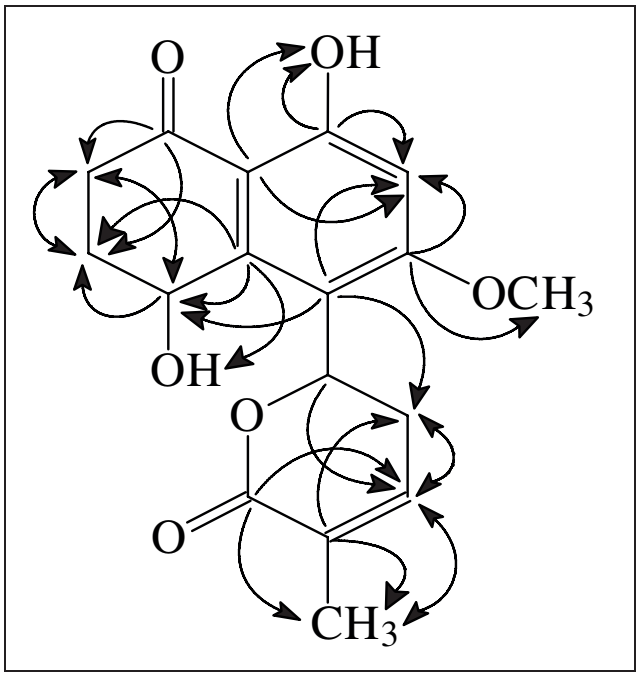


Fig. 2: Major relative peaks in HMBC of **1** (from C to H)

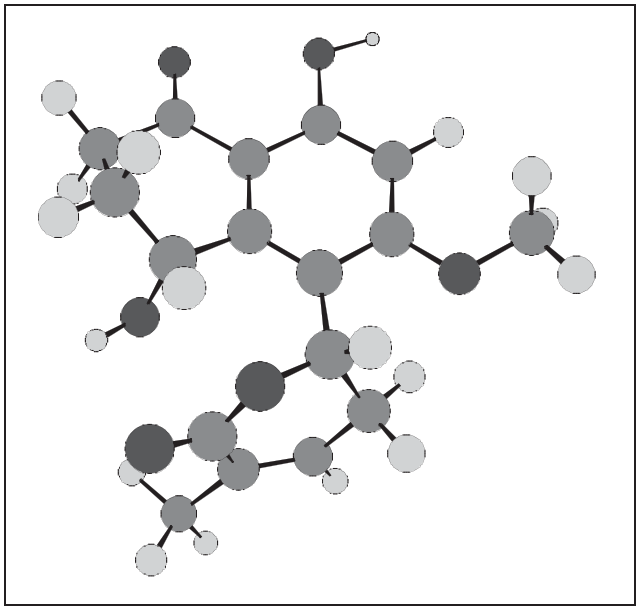


Fig. 3: The 3D structure shows the configuration of two chiral carbons (C-4 and C-11) in **1**

3. Experimental

3.1. Equipment

Melting points: X-4 Digital Display Micro-melting point apparatus, uncorr. Optical rotation: polarimeter 241 (Perkin Elmer), solvent $CHCl_3$. UV-spectra were measured on a Spect 50-UV/Vis spectrophotometer in MeOH solution (Analytic Jena AG). IR-spectra were recorded on an FTS 165-IR spectrophotometer (Bio-Rad, USA). ^{1}H NMR (400.13 MHz), ^{13}C NMR (100.62 MHz) and 2D NMR were recorded on a Varian INOVA-400 FT-NMR spectrometer (USA) in $CDCl_3$ with TMS as int. standard. HR-ESIMS (High resolution electro-spray ionization mass spectrometry) was determined on a Bruker APEX II. Separation and purification were performed by chromatographic column (CC) over silica gel. Silica gel (200–300 mesh) used for CC and silica gel GF_{254} for TLC were obtained from the Qingdao Marine Chemical Factory, Qindao, P. R. China. Spots were detected on the TLC under UV light or by heating over 110 °C after spraying with 98% H_2SO_4-EtOH (v/v = 5 : 95).

3.2. Plant material

The plant material (No. 2001-03) was collected from Weiyuan county, Gansu province of P. R. China and was identified by adjunct Prof. Ji MA, Faculty of Pharmacy, First Military Medical University of PLA, Gangzhou, P. R. of China. A specimen has been deposited at our Lab.

3.3. Extraction and isolation

Air-dried and grounded whole plants of *Luppula anocarpa* (2.5 kg) were extracted seven times with 95% EtOH at room temperature (7 × 24 h), and the solvent was removed under reduced pressure to give a residue (110 g). The residue was suspended in water (1.0 L), then extracted with petroleum ether (60–90 °C) (1.5 L × 8), ethyl acetate (1.5 L × 6) and n-butanol (1.5 L × 8). The ethyl acetate fraction (8 g) was subjected to chromatography on a silica gel column with dichloromethane-acetone as gradient eluent to give four fractions (Fr. 1-Fr. 4). Fr. 1 (v/v, from 40 : 1 to 20 : 1) gave compound **5** (33 mg) after CC on a silica gel column eluting with cyclohexane-acetone (v/v, 10 : 1). Fr. 2 (v/v, from 20 : 1 to 15 : 1) was chromatographed using petroleum-acetone (v/v, 2 : 1) as eluent to obtain crude compound **1** (20 mg), which was further chromatographed on silica gel column using ether-ethyl acetate (v/v, 4 : 1) as eluent to yield pure terpene **1** (12 mg). Fr. 3 (v/v, from 10 : 1 to 5 : 1) was repeatedly purified by CC over silica gel with chloroform-methanol (v/v, 20 : 1) as eluent to give compound **2** (14 mg). The petroleum ether fraction (60 g) was subjected to silica gel chromatography and eluted with petroleum-ethyl acetate (v/v, from 40 : 1 to 1 : 1) to give nine fractions (Fr. A-I). Fr. B was chromatographed on silica gel column with petroleum-ethyl acetate (v/v, 8 : 1) as eluent to produce the mixture of compounds **8** and **12**, which was isolated by CC on a silica gel column, eluting with petroleum-acetone (v/v, 100 : 1) to afford pure compound **8** (7 mg) and **12** (5 mg). Fr. D was subjected to silica gel column using petroleum-ethyl acetate (v/v, 10 : 1) as eluent to produce compound **7** (500 mg). Fr. E was repeatedly chromatographed on silica gel with chloroform-acetone (v/v, 10 : 1) as eluent to obtain crude compound **3** (20 mg), which was further purified by silica gel column using ethyl acetate-chloroform (v/v, 1 : 7) as eluent to yield purity of **3** (9 mg). Fr. I was chromatographed on silica gel column, eluting with petroleum-acetone (4 : 1) to give compound **4** (5 mg), crude **11** (25 mg) and impure **6** (30 mg). The crude **11** was further purified by CC over silica gel with chloroform-acetone (v/v 12 : 1), and the impure **6** was also purified by CC silica gel with chloroform-acetone (4 : 1). Fr. H was chromatographed on silica gel column, with petroleum-acetone (v/v, 5 : 1) to produce compound **14** (33 mg). Fr. F was further separated into four sub-fractions (Fr. F1-Fr4) by CC using chloroform-acetone (v/v, 15 : 1) as eluent. In which Fr. F2 was the mixture of compounds **10** and **9**. The mixture was subjected to repeatedly silica gel chromatographic column with petroleum-ethyl acetate (v/v, 10 : 1) as eluent to give compound **10** (14 mg), and with benzene-acetone (5 : 1) as eluent to give compound **9** (7 mg). Fr. F4 was tested as a pure compound **13** (5 mg).

3.3.1. Lappulanocarpine A (**1**)

Brown gum, $[\alpha]_D^{25}$ −158° (C 0.25, $CHCl_3$). HR-ESIMS ($[M + NH_4]^+$, found: 336.1454; Calcd.: 336.1442). IR (KBr, cm^{-1}): 3451, 2926, 2854, 1722, 1642, 1594, 1455, 1432, 1365, 1336, 1233, 1131, 1047, 944, 847, 762. UV (λ_{max}, nm): 210 (loge 4.65), 368 (loge 3.99). ^{1}H NMR ($CDCl_3$, TMS, 400.13 MHz) and ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz): see Table 1.

3.3.2. Blumenol A (**2**)

Colorless needle crystals from EtOAc. M.p. 114–115 °C. IR (KBr, cm^{-1}): 3360, 1660, 975. UV (λ_{max}, nm): 237 (log ε 4.05) and 316 (log ε 2.55). ^{1}H NMR ($CDCl_3$, TMS, 400.13 MHz): δ 2.45 (1 H, d, J = 16.8 Hz), 2.25 (1 H, d, J = 16.8 Hz), 5.91 (1 H, brs), 5.79 (1 H, d, J = 15.7 Hz), 5.87 (1 H, dd, J = 15.7, 5.1 Hz), 4.42 (1 H, m), 1.30 (3H, d, J = 6.3 Hz), 1.02 (3 H, s), 1.11 (3 H, s), 1.90 (3 H, brs). ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz): δ 41.14 (C-1), 49.68 (C-2), 198.08 (C-3), 126.80 (C-4), 162.96 (C-5), 79.01 (C-6), 135.74 (C-7), 128.96 (C-8), 67.96 (C-9), 23.71 (C-10), 22.88 (C-11), 24.01 (C-12), 18.89 (C-13).

3.3.3. 7-Oxositosterol (3β-hydroxystigmast-5-en-7-one) (**3**)

Colourless needle crystals from Me_2CO. M.p. 108–110 °C. IR (KBr cm^{-1}): 3533, 3344, 2958, 2938, 2870, 1673, 1464, 1382, 1295, 1184, 1067, 1017, 948, 846. UV (MeOH, λ_{max}, nm): 202. EI-MS m/z (%) (rel. int.): 428 ($[M]^+$, 14). ^{1}H NMR ($CDCl_3$, TMS, 400.13 MHz): see Table 2, ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz): see Table 3.

3.3.4. 7α-Hydroxysitosterol (Stigmast-5-ene-3β, 7β-diol) (**4**)

Colorless needle crystals from Me_2CO. M. p. 202–204°C. IR (KBr, cm^{-1}): 3605, 3400, 2950, 2935, 2860, 1665, 1464, 1380, 1228, 1192, 1111, 1057, 1010, 952, 928, 892, 866. EI-MS m/z (%) (rel. int.): 430 ($[M]^+$, 3), 412 (M-18, 100). ^{1}H NMR ($CDCl_3$, TMS, 400.13 MHz): see Table 2, ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz): see Table 3.

3.3.5. β-Sitosterol (**5**)

White needle crystals from Me_2CO. M.p. 139–140 °C. TLC and IR spectrum were identical with those of an author's sample.

3.3.6. β-Sitosterol-3-(6′-glyceryl)-D-glucopyranoside (**6**)

Colourless wax. IR (KBr, cm^{-1}): 3417, 2927, 2854, 1740, 1466, 1379, 1174, 1083, 1021, 723. ^{1}H NMR ($CDCl_3$, TMS, 400 MHz) and ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) spectral data were identical to the reported values published by Greca et al. (1990).

3.3.7. Stigmasterol (**7**)

Colorless needles. M.p. 149–151 °C. TLC and IR spectrum were identical with those of an author's sample.

Table 2: ^{1}H NMR (400.13 MHz) data for compounds 3, 4, 8, 9

Proton	3	4	8	9
H-3	3.69, m	3.59, m		
H-4			5.74, d (2.2)	5.82, s
H-6	5.70, d (1.7)	5.62, d (4.8)		4.34
H-7		3.68, m		
H-18	0.69, s	0.69, s	0.72, s	0.75, s
H-19	1.20, s	0.99, s	1.19, s	1.38, s
H-21	0.93, d (6.5)	0.93, d (6.6)	0.93, d (6.6)	0.93, d (6.5)
H-26	0.84, d (6.5)	0.83, d (6.7)	0.84, d (6.8)	0.84, d (6.1)
H-27	0.82, d (6.7)	0.82, d (6.7)	0.82, d (6.8)	0.82, d (6.1)
H-29	0.85, t (7.1)	0.85, t (7.2)	0.85, t (7.2)	0.85, t (6.7)

(δ, in $CDCl_3$, TMS as int. standard, the coupling constants were in the parentheses)

Table 3: ^{13}C NMR (100 MHz) data for compounds 3, 4, 8, 9

Position	3	4	8	9		3	4	8	9
C-1	36.34	36.96	35.68	37.10	C-16	28.53	29.24	28.17	28.17
C-2	31.18	31.33	33.88	34.26	C-17	54.71	55.66	56.05	56.07
C-3	70.52	71.35	199.59	200.43	C-18	11.96	11.62	11.93	12.01
C-4	41.80	41.96	123.72	126.32	C-19	17.29	18.98	17.37	19.50
C-5	165.15	146.28	171.66	168.49	C-20	36.07	36.07	36.10	36.11
C-6	126.10	123.83	32.93	73.29	C-21	18.91	18.21	18.80	18.84
C-7	202.36	65.36	32.04	38.57	C-22	33.95	33.86	33.96	33.91
C-8	45.41	37.47	35.82	29.70	C-23	26.10	29.05	26.08	26.12
C-9	49.95	42.11	53.81	53.63	C-24	45.41	45.77	45.82	45.84
C-10	38.27	37.38	38.59	37.99	C-25	29.14	29.22	29.15	29.15
C-11	21.21	20.67	21.03	20.97	C-26	19.78	18.77	19.79	19.81
C-12	39.05	39.13	39.51	39.60	C-27	19.03	19.78	19.01	19.03
C-13	41.80	42.22	42.38	42.51	C-28	23.05	23.01	23.06	23.08
C-14	49.95	49.39	55.94	55.88	C-29	12.18	11.96	11.10	11.97
C-15	26.32	25.90	24.16	24.15					

(δ, in $CDCl_3$, TMS as int. standard, types of carbons were assigned by DEPT experiments)

3.3.8. Enone (Stigmast-4-en-3-one) (**8**)

White needle crystals from n-hexane. M.p. 87–89 °C; EI-MS m/z (%) (rel. int.): 412($[M]^+$, 100), 370 (8), 289 (12), 271 (10), 229 (31), 147 (29) and 124 (93). IR (KBr, cm^{-1}): 2926, 2856, 1677, 1621, 1469, 1392. UV ($CHCl_3$, λ_{max}, nm): 246 ($\log_\varepsilon$ 4.20). ^1H NMR ($CDCl_3$, TMS, 400.13 MHz): see Table 2, ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz): see Table 3.

3.3.9. 6β-Hydroxyenone (Stigmast-4-en-6β-ol-3-one) (**9**)

Solids (Me_2CO). M.p. 192–194 °C. IR (KBr, cm^{-1}): 3500, 3403, 2985, 2869, 1681, 1466, 1384, 1232, 1194, 1039, 1018, 971, 879. UV (MeOH, λ_{max}, nm): 218, 233, 249 (log ε 4.32, 4.28, 3.84 respectively). EI-MS m/z (%) (rel. int.): 428 ($[M]^+$, 75). ^1H NMR ($CDCl_3$, TMS, 400.13 MHz): see Table 2, ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz): see Table 3.

3.3.10. 5α,8α-Epidioxy-methylcholesta-6, 22-dien-3β-ol (**10**)

Colourless needles from Me_2CO. M.p. 152–154 °C. IR (KBr, cm^{-1}): 3526, 3305, 2935, 2857, 2837, 1660, 1460, 1387, 1076, 1047, 968, 935, 858. EI-MS m/z (%) (rel. int.): 410 ($[M]^+$, 4). ^1H NMR ($CDCl_3$, TMS, 400.13 MHz): δ 6.51 (1 H, d, J = 8.4 Hz, H-7), 6.25 (1 H, d, J = 8.4 Hz, H-6), 5.18 (2 H, m, H-22, 23), 3.97 (1 H, m, H-3), 1.04 (3H, d, J = 6.4 Hz, H-21), 0.91 (3 H, d, J = 6.4 Hz, H-28), 0.89 (3 H, s, H-19), 0.84 (3 H, d, J = 6.8 Hz, H-27), 0.82 (3 H, d, J = 6.8 Hz, H-26), 0.81 (3 H, s, H-18); ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz): δ 34.7 (C-1), 30.1 (C-2), 66.5 (C-3), 37.0 (C-4), 82.1 (C-5), 135.4 (C-6), 130.7 (C-7), 79.4 (C-8), 51.1 (C-9), 36.9 (C-10), 20.6 (C-11), 39.4 (C-12), 44.6 (C-13), 51.7 (C-14), 23.4 (C-15), 28.6 (C-16), 56.3 (C-17), 12.9 (C-18), 18.1 (C-19), 39.7 (C-20), 20.9 (C-21), 135.2 (C-22), 132.3 (C-23), 42.8 (C-24), 33.0 (C-25), 19.9 (C-26), 19.6 (C-27), 17.6 (C-28).

3.3.11. 3β,6α-Dihydroxy-4-ene-cholestane (**11**)

Colourless crystals. ^1H NMR ($CDCl_3$, TMS, 400.13 MHz): δ 5.55 (1 H, brs, H-4), 4.24 (1 H, brs, H-3), 4.19 (1 H, m, H-6). ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz): δ 36.9 (C-1), 29.3 (C-2), 74.3 (C-3), 128.7 (C-4), 147.7 (C-5), 68.0 (C-6), 39.2 (C-7), 29.2 (C-8), 54.3 (C-9), 36.9 (C-10), 21.0 (C-11), 39.8 (C-12), 42.6 (C-13), 56.2 (C-14), 24.2 (C-15), 28.2 (C-16), 56.2 (C-17), 12.0 (C-18), 19.0 (C-19), 36.1 (C-20), 18.7 (C-21), 36.1 (C-22), 24.2 (C-23), 39.8 (C-24), 28.8 (C-25), 21.5 (C-26), 21.0 (C-27).

3.3.12. n-Hexadecanol (**12**)

White amorphous powder. M.p. 74.5–76.5 °C. IR (KBr, cm^{-1}): 3300, 2956, 2917, 2849, 1473, 1463, 1061, 730, 719. ^1H NMR ($CDCl_3$, TMS, 400 MHz): δ 0.88 (3 H, t, J = 6.0 Hz, H-16), 1.54 (2H, m, H-2), 3.64 (2 H, t, J = 6.8 Hz, H-1).

3.3.13. Glyceryl palmitate (**13**)

Colorless oil. ^1H NMR ($CDCl_3$, TMS, 400.13 MHz) and ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz) spectral data were identical to the data published by Du et al 2002.

3.3.14. Glyceryl linolentate (**14**)

Colourless gum. ^1H NMR ($CDCl_3$, TMS, 400.13 MHz): δ 5.36 (H-9, 10, 12, 13, 15, 16, m), 4.18 (H-1′, m), 3.94 (H-2′, m), 3.70 (H-3′ a, dd, J = 4.0, 12.0 Hz), 3.60 (H-3′ b, dd, J = 6.0, 12.0 Hz), 2.81 (H-8, m), 2.35 (H-2, t, J = 7.6 Hz), 2.06 (H-17, m), 1.63 (H-11, 14, t, J = 7.2 Hz), 1.33–1.25 (H-3–7, m), 0.88 (H-18, t, J = 4.4 Hz). ^{13}C NMR ($CDCl_3$, TMS, 100.62 MHz): δ 174.28 (C-1), 34.07 (C-2), 24.82 (C-3), 29.10 (C-4, 5, 6), 29.50 (C-7), 27.13 (C-8), 130.17 (C-9), 127.63 (C-10), 25.57 (C-11), 128.20 (C-12), 128.49 (C-13), 25.48 (C-14), 126.97 (C-15), 131.90 (C-16), 22.50 (C-17), 14.20 (C-18), 65.07 (C-1′), 70.20 (C-2′), 63.33 (C-3′).

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