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Triumfettoside, a new alkaloidal steroid glycoside and triumfettosterol, a new sterol from *Triumfetta flavescens*

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The aerial parts of *Triumfetta flavescens* H. (N. O. Tiliaceae) afforded a new alkaloidal sterioid glycoside, characterized as stigma 5(6)-ene-7,22-dione-25-methylamino-3β,23β-diol-3-*O*-β-D-glucoside and designated as triumfettoside (**1**); and a new sterol identified as stigma 5(6)-ene-7,22-dione-3β,23β-diol, designated as triumfettosterol (**2**). Their structures were elucidated on the basis of chemical and spectral analysis.

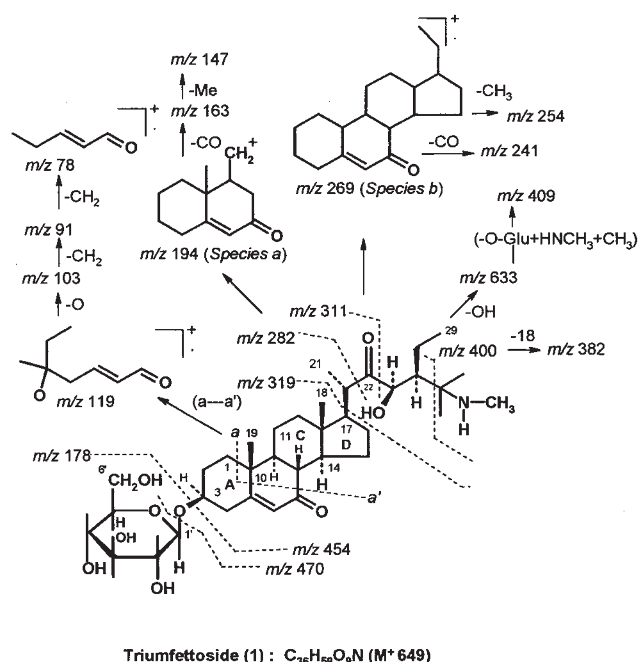
1. Introduction

Triumfetta flavescens H. (N. O. Tiliaceae) commonly as known as Rasha is wildly distributed in Saudi Arabia specially in Najd region [1] and has been reported to possess sedative and cardiotonic activity as the alcoholic extract produced a moderate sedation accompanied by slow and shallow respiration in experimental animals [2], and moderate stimulation on isolated rabbit heart causing the increase in force of contraction and heart rate [3]. The literature survey of the plant has revealed the presence of flavonoids and volatile oils so far [1]. We now report herein the isolation of a new alkaloidal steroid glycoside, characterized as stigma 5(6)-ene-7,22-dione-25-methylamino-3,23β-diol-3-*O*-β-D-glucoside and designated as triumfettoside (**1**); and a new sterol identified as stigma 5(6)-ene-7,22-dione-3β,23β-diol, designated as triumfettsterol (**2**), from the aerial parts of the plant. Their structures were elucidated on the basis of chemical and spectral analysis.

2. Investigations, results and discussion

Compound **1** named triumfettoside, obtained as colorless amorphous powder had the molecular composition $C_{36}H_{59}O_9N$ established on the basis of positive HRFABMS showing the molecular ion peak at m/z 650.4185 $[M + H]^+$, elemental analysis, ^{13}C NMR and DEPT spectra. It gave a positive Liebermann-Burchard test for steroid and Molisch's test for glycoside indicating it to be a steroidal glycoside. The IR spectrum indicated the presence of a hydroxyl group (3450–3550 due to OH and 1020–1070 due to C–O alcoholic), a carbonyl function (1750), a double bond (1600–1610) and a secondary amino nitrogen (1220–1260, 600–650 cm^{-1}). The ^{13}C NMR and DEPT spectra [4] showed 36 carbon atoms for the molecule consisting of seven methyls, nine methylenes, fourteen methines, four quaternary, and tow carbonyl carbon atoms (in total $C_{36}H_{53}$). The six carbon atoms comprising of one methylene, four methine and one-anomeric carbon atoms were assigned to one sugar moiety, and the remaining 30 carbon signals to the steroid skelton (Table 1). The sequential assignments of proton and carbon atoms were made with the help of COSY and HMQC experiments starting with the easily distinguishable carbinyl and olefinic protons at δ 3.116 and 5.161 respectively and further correlated with HMBC experiment (Fig.) (Table 1). The presence of an olefinic proton at δ 5.161 (d, J = 8.5 Hz) in its ^1H NMR spectrum was supported further by the resonance of sp^2 carbons deduced at C-5 (quaternary carbon) at δ 138.5 and C-6 (tertiary carbon) at 127.5 ppm in its ^{13}C NMR and DEPT spectra. It was, therefore, attributed at $\Delta^{5\,(6)}$ on the basis long range coupling in HMBC spectrum exhibiting correlations of C-5 with H-4 and H-9, and C-6 with H-8 and vice versa (Fig.). It was further confirmed by fragment ions in its mass spectrum at m/z 119, 103, 91 and 78 orginated by the rupture of ring A and B (Scheme).

Scheme



Triumfettoside (1) : $C_{36}H_{59}O_9N$ (M^+ 649)

The six methyl functionalities were found attached to saturated carbon atoms and could be placed with the help of COSY and long range coupling in HMBC spectra at positions 18 (δ_H 0.0680, s; δ_C 16.79), 19 (1.225, s; 24.09), 21 (1.248, d, J = 11.5; 25.11), 26 (1.091, s; 26.77), 27 (0.819, s; 15.77), and 29 (0.797, d, J = 11.0; 16.94); whereas the seventh methyl group was attached to secondary nitrogen atom exhibiting a sharp singlet at δ_H 2.51 and δ_C 40.70 in ^1H- and ^{13}C NMR spectra of the compound, which could be placed at C-25 on the basis of HMQC correlations as C-25 was appeared down field at δ_C 72.02 (quaternary carbon) rather then up field and exhibited a long range coupling (HMBC) with methyl groups at positions 26, 27 and $N-CH_3$ (Fig.).

Table 1: 1D and 2D-NMR Data for triumfettoside (1) (500 MHz)

Positions	^{1}H NMR[a]	^{1}H NMR[a] (Acetate)	^{13}C NMR	DEPT[b]	^{1}H-^{1}H-COSY	HMQC[c]	HMBC[d]	
							$^2J_{CH}$	$^3J_{CH}$
1a	1.501 ddd (11.0,9.7, 5.2)	1.555 ddd (11.9, 9.7, 5.2)	25.49	CH_2	H-1b, H-2a	25.49 t	–	–
1b	1.865 ddd (12.5, 5.8, 3.4)	1.855 ddd (12.5, 5.8, 3.4)	–	–	H-1a, H-2b	–	–	–
2a	1.835 dd (12.5, 5.5)	1.882 dd (12.5, 5.5)	23.82	CH_2	H-1b, H1a, H-2b	23.82 t	–	–
2b	1.355 m	1.376 m	–	–	H-2a, H-1a	–	–	–
3	3.116 dddd (5.0, 4.5, 9.0, 8.5, α-H)	4.253 dddd (5.0, 4.5, 9.0, 8.5, α-H)	69.98	CH	H-4a	69.98	–	C-1′
4a	1.860 dd (12.5, 5.5)	1.908 dd (12.5, 5.5)	47.98	CH_2	H-4b	47.98 t	C-5	–
4b	1.479 dd (11.0, 10.5)	1.474 dd (11.0, 10.5)	–	–	H-4a	–	–	–
5	–	–	138.5	C	–	138.5 s	–	–
6	5.161 d (8.5)	5.790 d (8.5)	127.5	CH	–	127.5 s	C-7	C-8
7	–	–	176.02	C	–	176.02 s	–	–
8	2.375 m	2.385 m	53.64	CH	–	53.64 d	C-7, C-9	C-6, C-10
9	1.274 m	1.335 m	41.58	CH	H-11a	41.58 d	C-10	C-1, C-14
10	–	–	49.02	C	–	49.02 s	–	–
11a	1.86 m	1.845 m	20.47	CH_2	H-11b, H-12a	20.47 t	–	–
11b	1.542 m	1.573 m	–	–	H-11a	–	–	–
12a	1.479 ddd (12.8, 12.8, 3.2)	1.451 ddd (12.8, 12.8, 3.2)	36.95	CH_2	H-11a, H-12b	36.95 t	–	C-18
12b	1.86 ddd (12.8, 3.1, 3.1)	1.855 ddd (12.8, 3.1, 3.1)	–	–	H-12a, H-11b	–	C-13	–
13	–	–	38.25	C	–	38.25 s	–	–
14	0.819 m	0.905 m	56.25	CH	H-15a	56.25 d	–	C-18
15a	1.479 m	1.465 m	28.15	CH_2	H-15b, H-16a	28.15 t	C-16	–
15b	1.145 m	1.141 m	–	–	H-15a	–	–	–
16a	1.656 m	1.678 m	33.25	CH_2	H-15b	33.25 t	C-15	–
16b	1.274 m	1.275 m	–	–	–	–	–	–
17	2.488 d (11.0)	2.532 d (11.0)	56.13	CH	H-16b	56.13 d	C-16	C-18, C-15
18	0.680 s	0.718 s	16.79	CH_3	H-14	16.79 q	C-13	–
19	1.225 s	1.250 s	24.09	CH_3	–	24.09 q	–	C-1, C-5, C-9
20	1.14 m (β-H)	1.141 m (β-H)	29.91	CH	H-17	29.91 d	C-22	–
21	1.248 d (8.5)	1.281 d (8.5)	25.11	CH_3	–	25.11 q	–	C-17, C-22
22	–	–	179.69	C	–	179.69 s	–	–
23	2.656 d (9.0, α-H)	3.933 d (9.0, α-H)	83.07	CH	–	83.07 d	C-22, C-24	C-28
24	1.504 d (9.0, α-H)	1.573 d (9.0, α-H)	46.37	CH	H-28a	46.37 d	–	C-29
25	–	–	72.02	C	–	72.02 s	–	–
26	1.091 s	1.059 s	26.77	CH_3	–	26.77 q	C-25	C-27
27	0.819 s	0.948 s	15.77	CH_3	–	15.77 q	C-25	C-26
28a	1.15 m	1.165 m	26.15	CH_2	H-28b, H-24	26.15 t	–	–
28b	1.835 m	1.908 m	–	–	H-28a	–	–	–
29	0.797 d (11.0)	0.9478 d (11.0)	16.94	CH_3	H-28b	16.94 q	C-28	C-24
NH–	1.633 s	1.690 s	–	–NH–	–	–	–	–
NH–CH_3	2.51 s	2.55 s	40.70	–NH–CH_3	–	40.70 q	–	C-25
23-OH	3.778 brs	2.113 s (3 H, $COCH_3$)	–	–	–	–	–	–
1′	5.178 d (8.5)	5.522 d (8.5)	94.46	CH	H-2′	94.46 d	–	H-3′
2′	3.098 d (9.5, 9.0)	5.129 dd (9.5, 9.0)	72.58	CH	H-3′	72.58 d	H-3′, H-1′	–
3′	3.203 dd (9.5, 9.0)	5.265 dd (9.5, 9.0)	77.09	CH	H-4′	77.09 d		H-4′
4′	3.126 dd (9.5, 9.0)	5.181 dd (9.5, 9.0)	77.93	CH	H-5′	77.93 d	H-3′	–
5′	3.879 brm	4.867 brm	67.66	CH	H-4′	67.66 d	–	–
6a′	3.457 dd (11.5, 5.6)	3.781 dd (11.5, 5.6)	61.06	CH_2	H-6′b	61.06 t	–	H-4′
6b′	3.606 dd (11.5, 5.6)	4.063 dd (11.5, 5.6)	–	–	H-6′a	–	–	–
4 × O	–	2.009, 2.017, 2.025, 2.066	–	–	–	–	–	–
Ac		(3 H each, 4 × s, sugar acetoxyls)	–	–	–	–	–	–

[a] Assignments were based on ^{1}H-^{1}H- and ^{1}H-^{13}C-COSY, and HMQC experiments; coupling constants in Hertz are given in parentheses; s: singlet, d: doublet, m: multiplet, t: triplet. Methylene diasterotropic protons exhibited different δ_H in ^{1}H-NMR and same δ_C, but different signals in HMQC
[b] DEPT chemical shifts are presented at $\theta = 3\pi/4$ when methylene groups reaches negative maximum
[c] C-multiplicities were established by DEPT experiment
[d] The correlations in HMBC have been shown from protons to carbons

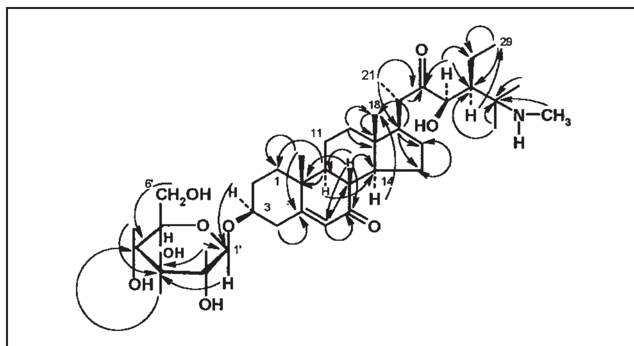


Fig.: Significant heteronuclear multiple bond correlations (HMBC) for triumfettoside (1). Arrows point from proton to carbon

The ^{13}C NMR spectrum aslo showed two signals at 176.69 due to two cabonyl groups. The peak at 176.02 exhibited long range couplings in HMBC with methine proton of positions-6 and 8 and thus could be placed at positions-7, which was further substantiated by prominent peaks at m/z 119, 103, 91 and 78 in its mass spectrum (Scheme). The other ketonic group showed long-range corrlations with protons at positions 20, 21 and 23, which could place it at position-22. It was further supported by the signifacant peaks at m/z 311 and 282 originated by the fragmentation of side chain (Scheme).

The ^{1}H- and ^{13}C NMR spectra of the compound exhibited signal at δ_H 3.116 (1 H, dddd, J = 9.0, 8.5, 5.0, 4.5 Hz, $^1/_2$ w = 13.5) and δ_C 69.98 due to one carbinyl proton, which could be placed at position-3 biogenetically, and on the basis of mass fragmentation pattern displaying prominent peaks at m/z 119, 103, 91 and 78 in its mass spectrum (Scheme). The coupling interactions indicated the α-orientation of the methine proton and β-orientation of the hydroxyl oxygen at C-3 [5]. The H-3 also showed long-range coupling in the HMBC spectrum with an anomeric carbon at C-1′ (δ_C 94.46) of the sugar moiety indicating the linkage of the hydroxyl group at position-3 with C-1′ of sugar unit. The ^{1}H- and ^{13}C NMR spectra also showed the presence of another hydroxyl group exhibiting a signal at δ_H 2.656 (1 H, d, J = 9.0 Hz) and δ_C 83.07, which could be placed at position-23 as it displayed long range correlations with C-22, C-24 and C-28. The coupling interaction (d, J = 9.0 Hz) indicated the axial-axial coupling of H-23 with H-24 justifying the α-orientation of the methine proton and β-orientation of the hydroxyl group.

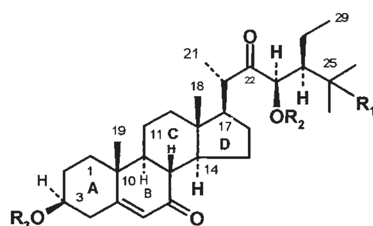
The ^{1}H- and ^{13}C NMR spectra of the compound exhibited resonances for an anomeric proton at δ_H 5.178 (d, J = 8.5 Hz) and a carbon at δ_C 94.46 along with another sugar proton and carbon atoms (Table 1) indicating only one sugar moiety in the molecule. The chemical shift of proton and carbon atoms were compared with the literature [6, 7] indicating it to be a β-D-glucose, which was further substanitated by co-paper chromatography of the sugar obtained on hydrolysis of **1** with an authentic sample of glucose using aniline-phthalate as visualizing reagent. The acetate **1a** exhibited five singlets of three protons each in the range of δ 2.009–2.113 due to four acetoxyls of sugar and one acetoxyl at position-23, thereby indicating only one sugar moiety in the molecule. Multiplet pattern and coupling constant of the anomeric proton (d, J = 8.5 Hz) confirmed the β-configuration of the sugar [6, 8]. The sequential assignments of the resonances for the glucose residue were made with COSY and HMQC experiments (Table 1) starting with the easily distinguishable anomeric proton at δ 5.178 and further correlated with an HMBC experiment (Fig.), whereupon the HMBC spectrum showed the correlation of H-3 of the aglycone with C-1′ of glucose unit indicating it to be linked with the hydroxyl group at position-3. Other correlations were also clearly indicative of the proposed structure.

The mass spectrum of the compound also supported the proposed structure, which exhibited prominent peaks characteristic of a stigmastane skeleton at m/z 193 (species a) due to a fragment originated by the rupture of ring-C; m/z 269 (species b) due to an ion produced by elimination of the side chain at position-20; m/z 119, 103, 91 and 78 due to ions produced by the rupture of rings-A and B; m/z 454 and 470 due to elimination of glucosoyl and glucosyl fragments, and m/z 409 due to elimination of a sugar unit, $NH{-}CH_3$, and methyl groups from the molecule. Other peaks in the spectrum were also supportive of the proposed structured of the compound (Scheme).

Thus, on the basis of the spectral and chemical studies the structure of compound **1** was elucidated as stigma 5(6)-ene-7,22-dione-25-methylamino-3β,23β-diol-3-*O*-β-D-glucoside and has been designated as triumfettoside (**1**).

Compound **2** named triumfettosterol, obtained as colorless needless had the molecular composition $C_{29}H_{46}O_4$ as established on the basis of HRMS showing the molecular ion peak at m/z 459.3393 $[M + H]^+$, elemental analysis, ^{13}C NMR and DEPT spectra. It gave a positive Liebermann-Burchard test indicating it to be a steroid. The IR spectrum indicated the presence of a hydroxyl group (3300–3500, 1040–1050), a carbonyl function (1750) and a double bond (1540–1560). The ^{13}C NMR and DEPT spectra [4] showed 29 carbon atoms for the molecule consisting of six methyls, eight methylenes, ten methines, three quaternary and two carbonyl carbon atoms. The sequential assignments of proton and carbon atoms were made in the same way as in the case of compound **1** with the help of COSY and HMQC experiments starting with the easily distinguishable carbinyl and olefinic protons and further correlated with HMBC experiment (Table 2). The spectral data of compound **2** were close to those of the aglycone obtained on hydrolysis of compound **1**, except for the absence of the methylamino group at position-25. Its acetate **2a** exhibited two singlets at δ_H 2.104 and 2.001 due to two acetoxyls, which indicated two hydroxyl groups assignable at positions-3 and 23 with the help of the HMBC spectrum. The coupling interactions indicated the α-orientation of the methine protons and the β-orientation of the hydroxyl groups at C-3 and C-23 [5] (Table 2). The two-carbonyl functions could be assigned at positions-7 and 22 with the help of HMBC and MS fragmentation pattern.

Thus, on the basis of the spectral and chemical studies the structure of compound **2** was elucidated at stigma 5(6)-ene-7,22-dione-3β-23β-diol and has been designated as triumfettosterol (**2**).



(1) Triumfettoside: $R_1 = NH{-}CH_3$, R_2 = β-D-Glucopyranoside.
(1a) Triumfettoside penta acetate: $R_1 = NH{-}CH_3$, $R_2 = COCH_3$, R_3 = β-D-Glucopyranoside (4 × $COCH_3$).
(2) Triumfettosterol: $R_1, R_2, R_3 = H$
(2a) Triumfettosterol diacetate: $R_1 = H_1$, $R_2, R_3 = COCH_3$

Table 2: 1D and 2D-NMR Data of triumfettosterol (2) (500 MHz)

Positions	^{1}H NMR[a]	^{1}H NMR[a]	^{13}C NMR	DEPT[b]	^{1}H-^{1}H-COSY	HMQC[c]	HMBC[d]	
							$^{2}J_{CH}$	$^{3}J_{CH}$
1a	1.50 ddd	1.555 ddd	25.63	CH_2	H-1b, H-2a	25.63 t	–	C-19
1b	1.852 ddd (12.5, 5.5, 3.5)	1.855 ddd (12.5, 5.5, 3.5)	–	–	H-1a, H-2b	–	–	–
2a	1.855 dd (12.5, 5.5)	1.882 dd (12.5, 5.5)	23.80	CH_2	H1a, H-2b	23.80 t	–	–
2b	1.376 m	1.376 m	–	–	H-2a, H-1a	–	–	–
3	3.933 brm ($W_{1/2}$ = 13.5, α-H)	4.331 brm ($W_{1/2}$ = 13.5, α-H)	72.01	CH	H-4a, H-2b	72.01 d	–	–
4a	1.861 dd (12.5, 5.5)	1.915 dd (12.5, 5.5)	47.35	CH_2	H-4b	47.35 t	C-5	–
4b	1.50 dd (11.0, 10.5)	1.471 dd (11.0, 10.5)	–	–	H-4a	–	–	–
5	–	–	139.06	C	–	139.06 s	–	–
6	5.168 d (8.5)	5.361 d (8.5)	127.31	CH	–	127.31 s	C-7	C-8
7	–	–	179.49	C	–	179.49 s	–	–
8	2.382 m	2.385 m	53.67	CH	–	53.67 d	C-7, C-9, C-14	C-6, C-10
9	1.374 m	1.410 m	41.61	CH	H-11a	41.61 d	C-10	C-1, C-19, C-14
10	–	–	48.87	C	–	48.87 s	–	–
11a	1.86 m	1.843 m	20.58	CH_2	H-11b, H-12a	20.58 t	–	–
11b	1.681 m	1.564 m	–	–	H-11a	–	–	–
12a	1.50 ddd (12.8, 12.8, 3.2)	1.448 ddd (12.8, 12.8, 3.2)	35.26	CH_2	H-11a, H-12b	35.26 t	–	C-18
12b	1.86 ddd (12.8, 3.1, 3.1)	1.915 ddd (12.8, 3.1, 3.1)	–	–	H-12a, H-11b	–	C-13	–
13	–	–	38.29	C	–	38.29 s	–	–
14	0.820 m	0.978 m	56.57	CH	H-15a	56.57 d	–	C-18
15a	1.589 m	1.490 m	28.46	CH_2	H-15b, H-16a	28.46 t	C-16	–
15b	1.135 m	1.140 m	–	–	H-15a	–	–	–
16a	1.681 m	1.645 m	33.52	CH_2	H-15b	33.52 t	C-15	–
16b	1.378 m	1.275 m	–	–	–	–	–	–
17	2.505 d (11.0)	2.532 d (11.0)	56.43	CH	H-16b	56.43 d	C-16	C-18
18	0.722 s	0.722 s	16.65	CH_3	–	16.65 q	C-13	–
19	1.285 s	1.308 s	24.18	CH_3	–	24.18 q	–	C-1, C-5, C-9
20	1.150 m (β-H)	1.145 m (β-H)	29.34	CH	H-17	29.34 d	C-22	–
21	1.232 d (8.5)	1.280 d (8.5)	26.80	CH_3	–	26.80 q	–	C-17, C-22
22	–	–	183.01	C	–	183.01 s	–	–
23	2.616 d (9.0, α-H)	4.875 d (9.0, α-H)	83.44	CH	–	83.44 d	C-22	C-28
24	1.584 d (9.0, α-H)	1.591 d (9.0, α-H)	46.24	CH	H-28a, H-25	46.24 d	–	C-29
25	–	–	26.33	CH	H-24	26.33 d	C-26, C-27	–
26	1.080 d (6.5)	1.227 d (6.5)	25.77	CH_3	–	25.77 q	C-25	C-27
27	0.846 d (6.5)	0.965 d (6.5)	15.58	CH_3	–	15.58 q	C-25	C-26
28a	1.15 m	1.168 m	26.80	CH_2	H-28b, H-24	26.80 t	–	–
28b	1.835 m	1.973 m	–	–	H-28a	–	–	–
29	0.901 d (11.0)	0.978 d (11.0)	17.01	CH_3	H-28b	17.01 q	C-28	C-24
3-OH	3.768 brs	2.104 s (3 H, $COCH_3$)	–	–	–	–	–	–
23-OH	4.312 brs	2.002 s (3 H, $COCH_3$)	–	–	–	–	–	–

[a] Assignments were based on ^{1}H-^{1}H- and ^{1}H-^{13}C-COSY, and HMQC experiments; coupling constants in Hertz are given in parentheses; s: singlet, d: doublet, m: multiplet, t: triplet. Methylene diastereotopic protons exhibited different δ_H in ^{1}H-NMR and same δ_C, but different signals in HMQC
[b] DEPT chemical shifts are presented at $\theta = 3\pi/4$ when methylene groups reaches negative maximum
[c] C-multiplicities were established by DEPT experiment
[d] The correlations in HMBC have been shown from protons to carbons

3. Experimental

3.1. General

Melting points were determined on a Metler 9100 Electro thermal apparatus by open capillary method and are uncorrected. The IR spectra were recorded as KBr pellets on a PYE UNICAM Spectrophotometer, mass spectra on a Finnegan MAT 300 mass spectrometer, ^{1}H (500 MHz) and ^{13}C & DEPT 90 and 135 NMR (125 MHz) and 2D NMR (COSY, HMBC & HMQC) on a Bruker DRX 500 spectrometer in DMSO-d_6 using TMS as internal standard reference; chemical shift in δ (ppm) and coupling constants (J values) are in Hz. CC was performed using silica gel (0.04–0.063 nm, 230–400 mesh) as an adsorbent. TLC was performed on silica gel 60 F254 Merck plates and sprayed with vanillin-H_2SO_4 for visualization of the spots.

3.2. Plant material

The aerial parts of *Triumfetta flavescens* were collected on 10th June 2000 from Aqabah, and Tanumah region of southern Saudi Arabia and identified by a taxonomist of the center. A voucher specimen no. 141415 has been deposited in the herbarium of the center for future reference.

3.3. Extraction and isolation

Air-dried aerial parts (1.5 kg) were crushed to coarse powder and extracted thrice with 95% alcohol. The alcoholic extract was concentrated and dried under reduced pressure to get a viscous mass (120 g). It was then defatted with acetonitrile, the filtrate after removal of the fat was concentrated to dryness and subsequently fractionated into petroleum ether (20 g), chloroform (10 g) and methanol (10 g) soluble portions. The methanolic fraction was chromatographed on a column of silica gel, which afforded compound **2** (24 mg) on elution with chloroform-methanol (3 : 2), further elution (7 : 3) gave compound **1** (170 mg).

3.4. Compound characteristics

3.4.1. Triumfettoside (1)

White amorphous powder, m. p. > 300 °C; R_f 0.32 ($CHCl_3$-MeOH = 3 : 2); IR ν_{max} cm^{-1} (KBr): 3450–3550 (OH), 2935 (CH_3), 2860 (CH_2), 1750 (C=O), 1600–1610 (C=C), 1450, 1360, 1220–1260 (>N–H, sec. amine)), 1020–1070 (C–O, alcoholic), 960, 600–650 (>N–H, sec. amine); 1 D and 2D-NMR: Table 1; EIMS m/z (rel. int.): 649 (1.5) (M^+, $C_{36}H_{59}O_9N$), 633 (1.5), 470 (4.5), 454 (5), 409 (3.5), 390 (5), 382 (1.5),

378 (4), 360 (5), 350 (5), 319 (4), 311 (2.5), 282 (3), 277 (5), 269 (4), 254 (10), 241 (4), 208 (6), 194 (15), 178 (8>), 163 (7), 147 (45), 128 (12), 119 (10), 103 (18), 91 (45), 78 (90), 63 (68), 44 (100); Elemental analysis, found: C 66.51%, H 9.16%, N 2.16%, required: C 66.52%, H 9.15%, N 2.15%; HRFABMS: $[M^+]$ m/z 649.4185 (cald for $C_{36}H_{59}O_9N$, 649.4189).

*3.4.2. Preparation of acetate of compound **1***

Compound **1** (50 mg) was treated with Ac_2O-pyridine (1 : 1), which on usal work up afforded acetate **1a** (40 mg); IR ν_{max} cm^{-1} (KBr): 2930 (CH_3), 2850 (CH_2), 1750, 1740 (C=O), 1610–1650 (C=C), 1455, 1366, 1245 (C–O, ester), 1223–1258 (>N–H, sec. amine), 1020–1070 (C–O, alcoholic), 960, 600–650 (>N–H, sec. amine); ^{1}H NMR: Table 1.

*3.4.3. Acid hydrolysis of compound **1***

Compound **1** (20 mg) was refluxed with 10% HCl in aqueous alcohol (5 ml) at 100 °C for 2 h, cooled and filtered, the filtrate was paper chromatographed with an authentic sample of glucose (R_f 0.24) using *n*-butanol-benzene-pyridine-water (5 : 1 : 3 : 3).

3.4.4. Triumfettosterol (**2**)

Colourless neddles, m.p. 276–277 °C; R_f0.65 ($CHCl_3$-MeOH = 3 : 2); IR ν_{max} cm^{-1} (KBr): 3300–3500 (OH), 2920 (CH_3), 2860 (CH_2), 1750 (C=O), 1540–1560 (C=C), 1450, 1400, 1040–1050) (C–O, alcoholic), 600; 1D and 2D NMR: Table 2; EIMS m/z (rel. int.): 458 (3) (M^+, $C_{29}H_{46}O_4$), 442 (4), 430 (2), 422 (3), 416 (6), 402 (3), 380 (50), 361 (19), 287 (8), 273 (10), 269 (7), 245 (9), 189 (6), 216 (5), 119 (40), 103 (20), 91 (30), 81 (100), 78 (80), 65 (25).

*3.4.5. Preparation of acetate of compound **2***

Compound **2** (10 mg) was treated with Ac_2-O-pyridine (1 : 1), which on usal work up afforded acetate **2a** (8 mg); IR ν_{max} cm^{-1} (KBr): 2920 (CH_3), 2850 (CH_2), 1750, 1740, (C=O), 1540–1560 (C=C), 1455, 1410, 1245 (C–O, ester), 1040–1050 (C–O, alcoholic), 600; ^{1}H NMR: Table 2.

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