Indian Journal of Chemistry
Vol. 45B, September 2006, pp. 2154-2156

Note

10-Methyl-*n*-heptacosane and diglucosyldirhamnoside from the stem bark of *Balanites aegyptiaca* Delile

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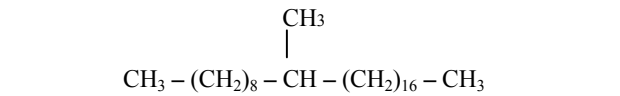
Received 21 August 2005; accepted (revised) 2 May 2006

A new long chain aliphatic compound, 10-methyl-*n*-heptacosane and a new sugar, diglucosyldirhamnoside have been isolated from the ethanolic extract of the stem bark of *Balanites aegyptiaca.* The structures of these compounds were elucidated by a combination of spectral methods (lR, MS, ^{1}H and ^{13}C NMR).

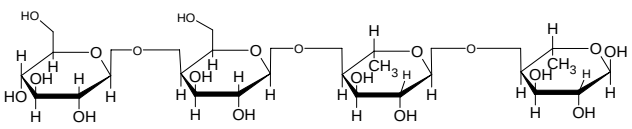
Keywords: *Balanites aegyptiaca*, 10-methyl-*n*-heptacosane, diglucosyldirhamnoside, jaundice, stem bark

IPC Code: Int.Cl.[8] A612K

Balanites aegyptiaca Delile a multibranched, spiny small tree up to 10 meter tall, is a native of India and widely distributed in Arabia, Egypt, Eritrea and tropical Africa[1]. The stem bark of this plant is used traditionally in the jaundice and syphilis[2-4] and fruits are used as an antidiabetic drug in the Egyptian folk medicine[5]. The plant has previously been reported to produce coumarins[6], flavanoids[7], alkaloids[8] and saponins and steroids[9-15]. In this paper, we report the isolation and identification of a new long chain aliphatic compound, 10-methyl-*n*-heptacosane **1** and a sugar, diglucosyldirhamnoside **2** from the stem bark of this plant.



1—10-methyl-*n*-heptacosane ($C_{28}H_{58}$)



$C_{24}H_{42}O_{19}$

2—Diglucosyldirhamnoside

Results and Discussion

Compound **1** was obtained from petroleum ether eluents (fractions 1-30) as colourless amorphous powder. It did not respond to TNM and bromine water tests suggesting saturated nature of the molecule. Its IR spectrum showed absorption peak at 721 cm^{-1} for long chain aliphatic compound. Its +ve FAB mass spectrum shows molecular ion peak at m/z 394 corresponding to molecular formula of a saturated aliphatic compound $C_{28}H_{58}$. The mass spectrum showed ion peaks related to C_nH_{2n+2}, C_nH_{2n-1}, and CnH_2n and the intensity and abundance of the peaks decreasing with increasing molecular weight of the fragments. The presence of C_nH_{2n+1} ion in higher abundance in comparison to C_nH_{2n-1} suggested the saturated and long chain aliphatic nature of the molecule. The fragment ion peaks at m/z 155 [C_{10-11} fission]$^+$ and 127 [C_{9-10} fission]$^+$ supported the presence of methyl group at C-10. This also supported by its ^{1}H NMR spectrum, which displayed a three-proton doublet at δ 0.88 (J = 7.2 Hz) assigned to C-28 secondary methyl located at C-10. The terminal primary methyl protons appeared as two three-proton triplets (J = 8.7 Hz at δ 0.85 and 0.82). The ^{1}H NMR exhibited a one proton ($w^{1/2}$ = 17.55 Hz) ascribed to methine proton. Two two-proton multiples at δ 1.58 and 1.43 were ascribed to C-9 and C-11 methylene protons, respectively adjusted to methine C-I0. A broad signal integrating for 30 methylene protons appeared at δ 1.25.

The ^{13}C NMR displayed signals at δ 022.32, 21.13 and 14.31 for C-27, C-28 and C-l methyl carbons, respectively. An intense carbon signal at δ 29.71 was associated with the methylene carbons. Methine carbon appeared at δ 32.81. The absence of any signal beyond δ 2.32 in the ^{1}H NMR spectrum and beyond δ 32.81 in the ^{13}C NMR spectrum supported the saturated nature of the compound. On the basis of above evidences, the structure of compound **1** has been characterized as 10-methyl-*n*-heptacosane.

Compound **2** designated as diglucosyldirhamnoside was obtained from MeOH eluents. Its IR spectrum showed absorption band for hydroxyl group at 3399 cm^{-1}. Its +ve FAB mass spectrum showed molecular ion peak at m/z 634 corresponding to molecular

formula $C_{24}H_{42}O_{19}$ which was supported by its ^{13}C NMR spectrum (**Table I**).

The ^{1}H NMR of diglucosyldirhamnoside displayed four one-proton doublets at δ 5.2 *(J* =8.4Hz), and 5.17 (*J* = 3.3 Hz), 5.04 (*J* = 9.0 Hz) and 4.66 (*J* = 6.6 Hz) assigned to the anomeric protons H-l, H-l′, H-′′ and H-l′′′. Two sets of three two-proton each multiplet at δ 3.54, 3.18, 4.45 and 3.42, 3.14, 4.24 were ascribed to hydroxyl methine protons H-3, H-3′, H-4, H-4′, H-5, H-5′ and H-3′′, H-3′′′, H-4′′, H-4′′′, H-5′′, H-5′′′, respectively. Four one-proton multiplet for H-2, H-2′, H-2′′ and H-2′′′ appeared correspondingly at δ 3.96, 3.78, 3.75, and 3.62. Two two-proton multiplets at δ 3.11 and 3.03 were attributed to oxygenated methylene proton respectively. Two three-proton doublets at δ 1.04 *(J* = 5.7 Hz) and δ 0.87 *(J* = 4.2 Hz) were accounted to terminal methyl protons C-6′′ and C-6′′′, respectively.

The ^{13}C NMR spectrum of diglucosyldirhamnoside showed signals for four anomeric carbons at 103.61, 102.70, 97.6 and 92.01 assigned to C-I, C-I′, C-I′′ and C-1′′′. Two terminal methyl carbons for C-6′′ and C-6′′′ appeared correspondingly at δ 19.01 and 17.77.

On the basis of these evidences the structure of diglucosyldirhamnoside has been elucidated as 1,4-glucopyranosyl-l′, 4′-glucopyranosyl-l′′, 4′′-rhamnopyranosyl-l′′′, 4′′′-rhamnopyranoside.

Extraction and isolation

Stem bark of *B. aegyptiaca* was washed, chopped, dried in air and finally in an oven at a temperature below 45°C, and then powdered coarsely. The dried and powdered stem bark (2.5 kg) was extracted with ethanol in Soxhlet apparatus. The extract was concentrated under reduced pressure to get a dark brown viscous semi-solid mass (200 g). It was analyzed chemically for the determination of the presence of different chemical constituents. The concentrated mass was then dissolved in minimum amount of methanol (MeOH) and absorbed on silica gel to form slurry. The slurry was air dried and subjected to silica gel column chromatography prepared in petroleum ether. The column was eluted with petroleum ether, chloroform and methanol in order of increasing polarity to isolate the 10-methyl-*n*-heptacosane and diglucosyldirhamnoside. Elution of the column with petroleum ether (fraction 1-30) furnished colourless amorphous powder of 10-methyl-heptacosane recrystallized from $CHCl_3$-MeOH (1:1), 300 mg (0.021% yield) and elution of the column with MeOH (fraction 31-52) furnished colourless crystals of diglucosyldirhamnoside, recrystallized from ethanol, 420 mg (0.016% yield).

Compound 1: Colourless amorphous powder; m.p. 62-63°C; specific rotation, 68.9°; IR (KBr): 2919, 2850, 1463, 1379, 1371, 805, 721 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.32 (lH,m, w ½ = 17.55 Hz, H-10 α), 1.58 (2H, m, H_2-9), 1.43 (2H, brs, H_2-11), 1.25 (30 H, brs, 15 × CH_2), 0.88 (3H, d, *J* = 7.20 Hz, Me-28), 0.85 (3H, t, *J* = 8.7 Hz, Me-1), 0.82 (3H, t, *J* = 8.7 Hz, Me-27); ^{13}C NMR ($CDCl_3$): δ 32.81 (C-10), 29.71 (24 × CH_2), 22.32 (C-27), 21.13 (C-28), 14.31 (C-1); +ve FAB MS m/z (rel. int.): 394 $[Mt]^+(C_{28}H_{58})$ 8.7, 155 (68.9), 127 (27.3).

Compound 2: Colourless crystals; m.p. 251-52°C; specific rotation, 97.6°; IR(KBr): 399, 2937, 1631, 1417, 1379, 1051 cm^{-1}; ^{1}H NMR (DMSO-d_6): 0 5.20 (IH, d, *J*= 8.4 Hz, H-l), 5.17 (lH, d, *J*= 3.3 Hz, H-l′), 5.04 (lH, d, *J*= 9.0 Hz, H-l′′), 4.66 (lH, d, *J*= 6.6 Hz, H-l′′′), 4.45 (2H, m, H-5, H-5′), 4.24 (2H, m, H-5′′, H-5′′′), 3.96 (lH, m, H-2), 3.78 (1 H, m, H-2′), 3.75 (lH, m, H-2′′), 3.62 (lH, m, H-2′′′), 3.54 (2H, m, H-3, H-3′), 3.42 (2H, m, H-3′′, H-3′′′), 3.18 (2H, m, H-4, H-4′), 3.14 (2H, m, H-4′′, H-4′′′), 3.11 (2H, m, H_2-6), 3.03 (2H, m, H2-6′), 1.06 (3H, d, *J* = 5.7 Hz, Me-6′′), 0.87 (3H, d, *J* = 4.2 Hz, Me-6′′′).^{I3}C NMR ($CDCl_3$): **Table I**, +ve FAB MS m/z (rel. int.): 634 $[M]^+$ ($C_{24}H_{42}O_{19}$) (5.6).

Table I—^{13}C NMR spectral data of diglucosyldirhamnoside

C	δ_C	C	δ_C	C	δ_C	C	δ_C
I	103.61	1′	102.70	1′′	97.60	I′′′	92.10
2	74.69	2′	74.35	2′′	74.21	2′′′	74.11
3	72.91	3′	72.65	3′′	72.45	3′′′	72.00
4	70.98	4′	70.63	4′′	70.14	4′′′	69.92
5	83.81	5′	76.84	5′′	76.23	5′′′	75.06
6	62.17	6′	60.98	6′′	19.01	6′′′	17.77

Acknowledgments

The first two authors thank Prof M Iqbal for extending facilities for the completion of the research work. The authors also thank Dr M P Sharma, Department of Botany, Jamia Hamdard, New Delhi for the identification of the plant material.

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