RESEARCH PAPER

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Characterization of the Chemical Constituents of *Datura metel* Linn

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One steroidal constituent, daturasterol, and a tricyclic diterpene, daturabietatriene, have been isolated for the first time from the stem bark of *Datura metel* Linn. along with β-sitosterol and atropine. The structures of the new compounds have been elucidated as 24β-methylcholest-4-ene-22-one-3α-ol and 15, 18- dihydroxyabietatriene, respectively, on the basis of the spectral data analyses and chemical ractions.

DATURA metel Linn. (Solanaceae) has an important place in the traditional systems of medicine as a narcotic, anodyne and antispasmodic drug similar to Belladonna and Stramonium[1,2]. Withanolides[3-8], tropane alkaloids[9] and steroidal lactones[10] have been reported from *D. metel*. This communication describes the isolation and characterization of two new non-alkaloidal constituents from this plant.

EXPERIMENTAL

Extraction

Air-dried and powdered stem bark (2.65 Kg), collected from the campus of Jamia Hamdard, was extracted with with ethanol (95 %) in a Soxhlet. The extract was concentrated in vaccum to yield a dark brown mass (170 g). It was fractionated into dil. HCl- insoluble and soluble portions to separate alkaloidal component. The HCl-insoluble non-alkaloidal part was dried and chromatographed over silica gel column. The column was eluted with petroleum ether, chloroform and methanol in the order of increasing polarity to isolate the following compounds:

Compound 1 : Elution of the column with $CHCl_3$ gave compound 1, crystallized from $CHCl_3$ - MeOH (1:1), positive to Liebermann-Burchard test, 0.25 g, m.p. 139-140°, $[\alpha]_{D25}$ ^{-36}o(c 1.5, $CHCl_3$), identified as β-sitosterol.

Compound 2: Fractions eluted with $CHCl_3$ - MeOH (9:1) on crystallisation with $CHCl_3$-MeOH (1:1) furnished compound 2, 0.60 g (0.022%), m.p. 175-176°, $[\alpha]_D$ ^{25} - 23° (c. 0.2, MeOH), UV λmax (MeOH) 212 nm (log ε 3.2), IR υmax 3460 (OH), 2936, 1700 (CO), 1620 (C=C), 1440, 1375, 1255, 1026 cm^{-1}; ^{1}H NMR (100 MHz), $CDCl_3$) δ 5.10 (1H, m, H-4), 3.10 (1 H, m, w 1/2 = 9.0 Hz, H-3β), 2.10 (1 H, brs, H- 20β) 1.97 (2H, m, H_2 - 23), 1.00 (3 H, brs, Me-19), 0.96 (3 H, d, J = 6.0 Hz, Me-21), 0.90 (3 H, d, J=6.0 Hz, Me-28) 0.80 (6 H, brs, Me-26, Me-27), 0.66 (3 H, br s, Me-18), EIMS m/z (rel.int.) 414 $[M]^+$ ($C_{28}H_{46}O_2$) (6.4), 399 (11.2), 381 (15.0), 329 (2.0), 304 (3.4), 273 (4.2), 255 (11.1), 222 (2.1), 205 (3.5), 192 (4.0), 191 (5.4), 178 (4.9), 177 (8.6), 161 (16.6),113 (10.1), 110 (24.8) 95 (100), 85 (8.3), 83 (30.5), 71 (41.3), 55 (93.3), 43 (89.5); monoacetyl derivative m.p. 141-142°, υmax 1725 cm^{-1}.

Compound 3 : Elution of the column with $CHCl_3$-MeOH (1:1) afforded colourless compound 3, crystallized from MeOH, 0.52 g (0.02 %), m.p. 239-240°, $[\alpha]_D$ ^{25} - 250° (C0.2, MeOH), UV λmax (MeOH) 209, 260 nm (log ε 8.2, 1.2), IR υmax (KBr) 3350 (OH), 2950, 1640 (C=C), 1460 (C=C), 1400, 1385, 1169,

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1115, 1055, 898 (C=C), 795 cm^{-1}. ^1H NMR ($CDCl_3$+DMSO-d_6) δ 7.50 (1 H, d, J = 3.0 Hz, H-14), 7.00 (1 H, d, J = 9.0 Hz, o,o- coupled, H-11), 6.93 (1 H, m, o,o- and o,m-coupled, H-12), 3.53 (1H, br s, H_2-15a), 3.40 (1 H, br s, H_2-15b), 2.50 (2 H, m, H_2-7), 2.26 (1H, br s, H-5), 2.16 (2 H, m, H_2-1), 1.86 (2 H, m, H_2-2) 1.76 (2 H, m, H_2-3), 1.70 (2 H, m, H_2-6), 1.23 (6 H, br s, Me-19, Me-20), 1.20 (6H, br s, Me-16, Me-17). EIMS m/z (rel. int.) 302 $[M]^+$ ($C_{20}H_{30}O_2$) (1.2), 227 (1.9), 212 (2.9), 203 (6.7), 202 (6.6), 168 (4.1), 162 (1.3), 158 (2.2), 154 (4.0), 148 (2.7), 144 (5.1), 143 (3.4), 140 (3.2), 134 (5.0), 130 (6.6), 123 (16.7), 113 (5.6), 103 (10.3), 100 (4.1), 89 (9.6), 82 (10.1), 75 (6.1), 71 (100), 70 (67.2), 69 (33.4) 59 (12.0), 54 (67.2), 51 (88.2); monoacetyl product m.p. 202-203°, IR νmax 3300, 1725 cm^{-1}.

Compound 4 : The HCl-insoluble portion was worked-up as usual[11] to get total basic mass responding positively to alkaloidal tests. It was subjected to alumina basic column chromatography. Elution of the column with $CHCl_3$ yielded compound 4, 0.04 g (0.002 %), m.p. 116-117°, picrate m.p. 175-176°, identified as atropine.

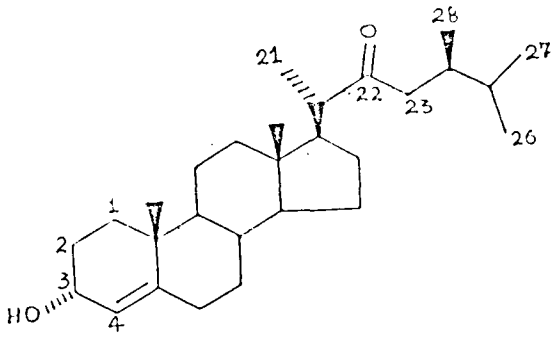
RESULTS AND DISCUSSION

Compound 2, named daturasterol, $C_{28}H_{46}O_2$ ([M] + m/z 414), responded positively to LB test. Its IR spectrum indicated the presence of hydroxyl, carbonyl groups and unsaturation. The EI mass spectrum of 2 exhibited diagnostically important peaks at m/z 399 $[M\text{-}Me]^+$, 396 $[M\text{-}H_2O]^+$, 381 $[396\text{-}Me]^+$, 273 [M- $C_9H_{17}O$, side chain SC]$^+$, 257 $[273\text{-}Me]^+$, 255 $[273\text{-}H_2O]^+$, and 212 [255-ring D cleavage]$^+$ which suggested that it was a C_{28} sterol possessing C_9-saturated side chain[12,13]. The prominent ion peaks at m/z 304, 110 [$C_{5,6}$-$C_{9,10}$ fission]$^+$, 55 [$C_{2,3}$-$C_{5,10}$-$C_{5,6}$ fission]$^+$, 161 [304- SC]$^+$, and 95 $[110\text{-}Me]^+$ indicated the location of C- 4(5) olefinic linkage and the hydroxyl group in ring A which was placed at C-3 on the basis of biogenetic considerations.[14] The saturated nature of ring C was concluded [15] from the peaks appearing at m/z 164, 178, 222, 192, 205, 191 and 177 generated due to cleavage of rings C and D. The ion peaks at m/z 85 [C_{22}-C_{23} fission, $C_6H_{13}]^+$, 113 [C_{20}-C_{22} fission]$^+$, and 329 $[M\text{-}85]^+$ indicated the presence of the carbonyl group at C-22.

The ^1H NMR spectrum of 2 displayed a one-proton downfield multiplet at δ5.10 assigned to H-4. A carbinol signal at δ3.10 was assigned to 3-position and its half-width of 9.0 Hz reflected β-orientation of the 3-proton[16]. The spectrum showed signals for two tertiary methyls (δ1.00, Me-19; 0.66, Me-18) and four secondary methyls (δ0.80, Me- 26, Me-27; 0.96, Me-21; 0.90, Me-28). These values were compared with 24-methyl steroids[17]. Treatment of 2 with Ac_2O-pyridine yielded a monoacetyl product, νmax 1725 cm^{-1}. On the basis of these accumulative data the structure of the 2 has been formulated as 24 β-methyl cholest-4-ene-22-one-3α-ol. A Δ^4-sterol, lawsaritol, possessing 3β-ol-4-ene system, has been recently isolated from ***Lawsonia inermis***[14].

Compound 3, designated as daturabietatriene, showed IR absorption bands for hydroxyl group and aromatic ring (1640, 1460, 898 cm^{-1}). It had a molecular ion peak at m/z 302 in its mass spectrum corresponding to a diterpenic formula, $C_{20}H_{30}O_2$ which indicated six degrees of unsaturation. The spectrum exhibited important ion fragments at m/z 202, 100 [C_1,10-$C_{4,5}$ fission]$^+$, 85 [100- Me]$^+$, 69 [100-$CH_2OH]^+$, 143 [202 - $(CH_3)_2COH$, 59]$^+$ and 71 [$C_{3,4}$- $C_{5,10}$-$C_{5,6}$ fission]$^+$. indicating the presence of hydroxymethylene group in ring A. The saturated nature of ring B was deduced from the fragments appearing at m/z 140, 162, 154, 148, 168 and 134 arose due to cleavage of the ring at different bonds. Elimination of mass unit 59 and water from various ions generated ion peaks at m/z 184, 103, 144, 89, 130, 75, 114, 203, and 113. From these data it was concluded that the compound 3 was an abietatriene-type diterpene.

The ^1H NMR spectrum of 3 displayed [18] one-proton each **meta**-coupled downfield doublet at δ7.50



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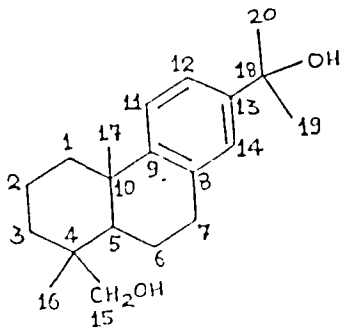

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(J= 3.0 Hz) assigned to H-14, ortho-coupled doublet at δ7.00 (J=9.0 Hz) due to H-11 and a multiplet at δ6.93 ascribed to H-12. The spectrum showed two hydroxylmethylene signals (δ3.53, 3.40) and two methyl signals (δ1.23, Me-19, Me-20; and 1.20, Me-16, Me-17). Based on these data the structure of the new diterpene 3 has been established as 15,18-dihydroxyabietriene.

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