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TRITERPENOIDS FROM *Pogostemon benghalense*

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ABSTRACT

A new natural triterpenoid Lanost-8(9), 25-dien-3-one has been isolated from aerial parts of Pogostemon benghalense in addition to β-Sitosterol and Lupeol acetate.

KEYWORDS : Pogostemon Benghalense, Labiatae, Triterpenes, β-Sitosterol, Lupeol Acetate, Lanost-8(9), 25-dien-3-one (A_2)

Pogostemon benghalense (Labiatae) is an aromatic pubescent shrub about 1-1.3m high, leaves petiolate ovate, 5-10cm long, doubly serrate. Flower in dense bracteate whorls very close and crowded in short cylindric, hoary-pubescent spikes(Raizada and Sexena, 1926). The fresh bruised leaves are applied as a cataplasm in order to clean wounds and promote healthy granulation. Juice is given in colic and fever. The root is a reputed remedy for haemorrhage, especially uterine haemorrhage.

The plant in combination with other drugs is prescribed as an antidote to snake and scorpion venoms. During the course of investigation stemonolone, a sesquiterpenoid has been reported from this plant (Phadnis et al., 1984). An essential oil has been obtained from the leaves of P. benghalense. Much work has been done on the essential oil found from this plant (Thapa et al. and Gupta,1971).

It was of interest to work on the aerial part as no systematic effort has been made to chemically examine the aerial parts of this plant. This report describes the characterization of a new tetracyclic triterpenoid.

EXPERIMENTAL

General

Mps: uncorrected; FTIR:KBr; ^{1}HNMR: Bruker DRX-300 (300MHz ^{1}HNMR) instrument in $CDCl_3$ with TMS as internal reference; TLC : silica gel G; CC: silica gel (Merk 60-120mesh). Spots were detected by exposure to I_2 vapour. The homogeneity of the isolate was checked on TLC in at least two solvent systems.

Plant Material

Pogostemon benghalense were collected from Jaunpur and identified in our Botany Department where a voucher specimen has been maintained.

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Extraction and Isolation

Dried and chopped aerial part (5kg) with hot MeOH (3x5L) for 30 hrs. The extract was filtered and concentrated by distillation of the solvent to give a dark brownish semi solid mass (130g).The semi solid mass so obtained was fractionated into hexane soluble (16g), ethyl acetate soluble (43g) and ethyl acetate insoluble (70g) fractions. A portion (16g) of the hexane fraction was chromatographed over silica gel (800g) eluting with varying proportions of hexane, EtOAc to provide compound A_1 (50mg), A_2 (90mg), and A_3 (50mg). The fractions collected were 200mL each and monitored by TLC.

β-Sitosterol(A_1)

Hexane-ethylacetate (4:1) eluate gave solid mass which on recrystallization from methanol yielded colourless crystals m.p.135-136^{0}C. IR V_{max}^{KB}(cm^{-1}) 3240, 2940, 2825, 1640, 1540, 1370, 1060, 970 and 780; It gave a positive L-B, Nolller's and TNM Test (Acetate m.p. 131-132^{0}C).

Lanost - 8(9), 25-dien-3-one (A_2)

Hexane-ethyleacetate (9:1) eluate gave solid mass, on repeated crystallization from methanol, it afforted white crystals, m.p.80-81^{0}C. IR V_{max}^{KB}(cm^{-1}) 2950, 2919, 1707, 1649, 1460, 1379, 1305, 1260, 1021 and 889; ^{1}HNMR: δ0.925(6H,s 18-H_3 and 29-H_3), 0.950(3H, S,19-H_3), 1.016(3H, s, 30-H_3), 1.076(3H,s, 28- H_3), 1.084(3H,d,J=6.8Hz, 21-H_3), 1.690 (3H, s, 27- H_3), 4.639 (1H, brs,s,26-H), 4.717 (1H, brs,s, 26-H); MS m/z (rel.int.) : M^+ 424(94%) for ($C_{30}H_{48}O_2$). 409(86), 407(94), 397(86), 383(31), 369(17),355(11),341(14),313(25),298(8), 271(36),256(11),255(33),2189(33),217(42),203(80),191(39),154(83),151(19),137(19),111(17).

Lupeol acetate(A_3)

Hexane-ethylacetate (3:1) eluate gave solid mass which on repeated crystallization from chloroform-hexane (1:1) yielded colourless needless, m.p.214-2150C. IR V_{max}^{KB} (cm^{-1}) 3050, 2900, 1735, 1640, 1450, 1380, 1370, 1250, 1020,1010, 890; ^{1}HNMR: δ0.78-1.10 (18H,six angular methyl group),1.68(3H,s,=C- CH_3), 2.02(3H,s,CH_3COO-), 4.40(1H,m,-CHOAc), 4.50(2H,m,>C=CH_2).

RESULTS AND DISCUSSION

The first compound was shown to be β-Sitosterol by mp, IR and colour test (Noller et al., 1942 and Heiborn, Bunbury, 1953). This was confirmed by CO-TLC with an authentic sample. Similarly the second compound proved to be lupeol acetate by comparing the data with the literature value (Upadhyay et al., 1982). The third compound, mp 80-81^{0}C, gave colour reactions which indicated that it is unsaturated triterpenoid. An $[M]^+$ at m/z 424 suggested the molecular formula as $C_{30}H_{48}O$. Its IR spectrum bands corresponded to carbonyl group (1707cm^{-1}), unsaturation (1649 cm^{-1}), and presence of terminal methylene group (2919, 889 cm^{-1}) (Silverstein et al., 1967).

^{1}HNMR spectrum exhibited signals due to five tertiary methyl groups at δ0.925 (6H,s), δ0.950 (3H,s), δ1.016(3H,s), δ1.076 (3H,s) and secondary methyl group δ1.084 (3H,d,J=6.8Hz). The appearance of a signal at δ 1.690 (3H,s), was indicative that the methyl group is linked to the unsaturated carbon(Wang et al., 2003). All these indicated the presence of tetracyclic triterpene skeleton (Deng et al., 2000). Signals at δ 4.639 and δ 4.717 as singlet (one proton each) were assigned to two protons of an exomethylene group or terminal methylene group. All these indicated that it was a dienone with lanostane skeleton (Yoshikawa et al., 2002).

Examination of the mass spectrum revealed the presence of fragment ions at m/z 409, 383, 381, 355, 341, 313, 298, 271, 256, 255, 218, 217, 191, 190 and 111. Peaks at m/z 313 and 111 are formed by the cleavage of C_{17}-C_{20} bond from the M^+ ion, suggested the presence of a monosaturated side chain. Formation of ion peak at m/z 298 by the loss of a methyl group at C-13 from the ion at m/z 313, ruled out the possibility of carbonyl group in the side chain. The formation of fragment ion at m/z 409 due to loss of metyl group at C-14 in M^+. This ion undergoes cleavage of C_{13}-C_{17} and C_{15}-C_{16} bonds simultaneously to produce a fragment ion appeared at m/z 271. The loss of a -CH_3 and then H from this ion resulted the formation of ions at m/z 256 and 255. The appearance of these ion peaks indicated the possible position of the carbonyl group and another double bond in the tetracyclic system. Fragment ion at m/z 218 and 191 are formed by the cleavage of C_{12}-C_{13} and C_8-C_{14} bonds. The loss of H and CH_2=CH_2 from the fragment ion at m/z 218 resulted into the formation of ions at m/z 217 and 190. The appearance of fragment ion at m/z 218 and 217 in which ring A and B are intact, further confirmed the position of carbonyl group at C-3 and double bond at 8(9) in the tetracyclic system.

Formation of fragment ion at m/z 341, 383, 381, 369, and 355 strongly supported the presence of one methyl group at C-20 which resonates as doublet at δ1.084 in ^{1}HNMR spectrum. Thus, on the basis of above discussion compound A_2 is characterized as Lanost-8(9),25-dien-3-one.

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