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FLAVONOID AND TERPENOID CONSTITUENTS OF *EUPATORIUM ODORATUM*

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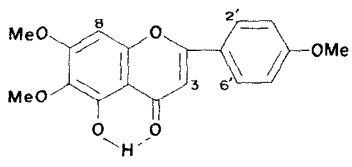
Eupatorium odoratum (Compositae, Subfam Corymbiferoe), distributed in temperate Himalayas, belongs to the tribe Eupatorieae [1] The plant is used in the country as fish poison [2] Isolation of a number of flavonoid compounds[3] from several *Eupatorium* species,[4] and a host of sesquiterpenoids from different Compositae plants prompted us to investigate the constituents of *Eupatorium odoratum* Earlier workers reported a sesquiterpene alcohol eupatol,[5] in its essential oil, and a trihydric alcohol, $C_{25}H_{34}O_5$ (m p 278–280) along with anisic acid [6] The present study has revealed the occurrence of two common triterpene alcohols, lupeol and β-amyrin, and a rarely occurring flavone, salvigenin (**1**) (first natural occurrence in Compositae) reported recently from *Salvia triloba*[7] (Labiatae) and *Alnus japonica*[8] (Betulaceae)



(1)

The cold light petrol (b p 60–80) extract of the whole plant (3 kg) upon concentration yielded an oily residue (free from basic component) The latter was chromatographed over silica gel (660 g) Elution with $CHCl_3$ gave a solid crystallizing from $CHCl_3$–light petrol in pale yellow needles (yield 0 01%), m p 185–186°, R_f 0 4 (TLC on silica gel, $CHCl_3$), analyzed for $C_{18}H_{16}O_6$ (M^+ 328) A pink coloration in Shinoda test and the UV spectrum [λ_{max}^{EtOH} 274 nm (ε 18 700) and 330 nm (ε 26 500)] are characteristic of a flavonoid The compound gave an intense green colour with $FeCl_3$ but no IR band for OH indicating the probable presence of a 5-OH This was supported by the appropriately deshielded phenolic proton (1H, δ 12 76 ppm exchangeable with D_2O) and IR bands at 1635

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[4] KUPCHAN, S M SIGEL, C W HEMINGWAY R J KNOX J R and UDAYAMURTHI M S (1969) *Tetrahedron* **25,** 1603 WAGNER H, IYENGER M A HORHAMMER L and HERZ W (1972) *Phytochemistry* **10,** 1504
[5] CHOPRA R N CHOPRA I C and VARMA B S (1969) *Supplement to the Glossary of Indian Medicinal Plants* p 28 Publications and Information Directorate CSIR New Delhi
[6] AHMAD M (1969) *Sci Res (Dacca Pak)* **6,** 37
[7] ULUBELEN A OZTURK S and ISILDATICI S (1968) *J Pharm Sci* **57,** 1037
[8] WOLLENWEBER E and WASSUM M (1972) *Tetrahedron Letters* 797

(chelated CO) and 830 cm^{-1} (*p*-substituted phenyl ring) The 100 MHz PMR spectrum ($CDCl_3$, δ) displayed the following signals in addition to the signal at 12 76 ppm which fit in well with structure (**1**) 3 90, 3 94, 3 98 (3H, *s* each, three OMe's), 6 54 and 6 58 (1H, *s* each, H-3 and H-8), 7 84* (2H, *d*, H-2′ and H-6′), 7 02* ppm (2H, *d*, H-3′ and H-5′, $J_{2',3'}$ or $J_{5',6'}$ 9 Hz) All the above data suggested its identity with salvigenin (**1**), which was confirmed by the preparation of the corresponding acetate, (Ac_2O–Py, R_t 24 hr), m p 166–167° (lit [8] 169–172°) and the fully methylated product (MeI–K_2CO_3 in refluxing acetone, 6 hr), m p 159–160° (lit [9] 162–163°) The MS measured in a Hitachi Perkin Elmer Spectrometer at 70 eV [*m/e* 328 (M^+), 313, 299, 285, 282, 269, 253, 214, 181, 167, 153, 135, 133, 132] is in full agreement with the assigned structure and is similar† to that reported earlier [8]

From the main chromatogram, C_6H_6 eluted in poor yield (0 005%) a solid containing mainly lupeol (R_f 0 38) mixed with small amount (estimated to be 3–5% from relative intensities of the spots) of β-amyrin (R_f 0 42) as evident from TLC comparison on silica gel plates impregnated with 12 5% $AgNO_3$ (C_6H_6–50% $CHCl_3$, spots developed by spraying with EtOH–5% Ac_2O–5% conc H_2SO_4 followed by heating at 120° for 15 min) with authentic materials and their respective acetates (lupeol acetate 0 35, β-amyrin acetate 0 54–light petrol–50% C_6H_6) and benzoates (lupeol benzoate 0 86, β-amyrin benzoate 0 92–light petrol–50% C_6H_6) Several successive chromatographies of the crude acetate over Brockmann alumina afforded pure lupeol acetate, m p 215°, [hydrolysis product, m p 213° ($CHCl_3$–MeOH), $[\alpha]_D$ +26°, identified (m m p , IR) as lupeol] The other fractions contained lupeol acetate enriched with β-amyrin acetate which, however, could not be isolated in pure state due to paucity of material

The cold $CHCl_3$ extract of the marc, when processed in a similar way, yielded the same compounds in low yields, (salvigenin 0 006%, lupeol and β-amyrin mixture 0 0014%) A voucher specimen No T/E o /3/72 has been preserved in this laboratory

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* Ulubelen *et al* [7] erroneously assigned H-2′, H-3′, H-5 and H-6′ as singlets at 7 93, 7 1, 6 93 and 7 68 ppm respectively

† The intensities of the peaks at *m/e* 167 (4 4%) and 135 (8 8%) were much less than those reported earlier (51 and 19% respectively)

[9] GEISSMAN, T A (1962) *The Chemistry of Flavonoid Compounds*, p 421, Pergamon Press, Oxford