

EXPERIMENTAL

Two dimensional chromatograms on Whatman 3MM paper were developed first in TBA (t-BuOH-HOAc-H₂O, 3 1 1), and then in 15% HOAc, NMR spectra of the trimethylsilyl ethers were recorded in CCl₄ and benzene-*d*₆ using TMS as an internal standard. All UV spectra were obtained using standard procedures.¹

Air-dried ground leaf material of *Eschscholzia mexicana* (collected near Monterrey, Mexico*) was extracted with 85% MeOH. A yellow amorphous material (100 mg) precipitated from the extract, the precipitate was purified over polyamide.¹ Color test: purple (UV) to yellow (UV/NH₃), *R_f*'s: TBA 0.27, HOAc 0.71, UV, λ_{\max} (nm): MeOH, 355, 270 sh, 257, NaOMe, 402, 270, 250 sh, AlCl₃, 400, 368, 300 sh, 270, AlCl₃-HCl, 400, 360 sh, 281, NaOAc, 417, 264, NaOAc-H₃BO₃, 360, 270 sh, 258.

Mass spectral data for PDM-I *m/e* at 533, 516, 368, 367, 350 (base peak), 322, 321, 230 (PDM-glucose moiety), 196, 184, 183 (PDM-arabinose fragment), 149 and 107. PDM of isorhamnetin 3-*O*-glucoside 7-*O*-rhamnoside: 547, 530, 368, 367, 350 (base peak), 321, 230 (PDM-glucose ion), 199, 198, 197 (PDM-rhamnose ion), 145, 127, 121 and 107.

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COUMARINS AND ALKALOIDS OF *AEGLE MARMELOS**

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The ubiquitous usage^{1, 2} of *Aegle marmelos* Corr. in the indigenous system of Indian medicine and the observed hypoglycaemic activity of the crude alcoholic extract of its root in rats prompted us to undertake the present investigation. A number of alkaloids,³⁻⁷ coumarins,^{5, 7-9} sterols^{6, 8, 9} and essential oils¹⁰ have previously been isolated from this plant.

The EtOAc soluble fraction of the alcoholic extract of the root on column chromatography over silica gel afforded the constituents outlined below.

Psoralen, 37 mg (C₁₁H₆O₃), m.p. and m.m.p. 169-170°, eluted with C₆H₆. UV $\lambda_{\max}^{\text{EtOH}}$ 240.

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(4.4), 290 (4.0), 342 (3.9) nm IR (KBr) ν_{\max} 1720 (C=O), 1640, 1575 (C=C), 880 cm^{-1} (furan), indicative of a furanocoumarin structure. The linear fusion of the furan moiety followed the downfield shift of C₄, C₅, C₆ and C₇ protons at δ 7.78, 7.67, 6.82 and 7.68 respectively in its NMR spectrum (60 MHz, CDCl₃). Its MS (M⁺ at *m/e* 186 as the base peak) also showed the linear fusion in contrast to the angular one where the M-28 is more intense than M⁺.

Xanthotoxin, 45 mg (C₁₂H₈O₄), m p and m m p 145–146°, M⁺ 216 eluted with C₆H₆. UV $\lambda_{\max}^{\text{EtOH}}$ 219 (4.48), 245 sh (4.44), 249 (4.45), 262 sh (4.23), 301 (4.16) nm IR (KBr) ν_{\max} 1705 (C=O), 1620, 1580 (C=C), 875 cm^{-1} (furan). It had a MeO group which was placed at C₉ due to upfield shift of the shielded C₅ proton (δ 7.33) in its NMR spectrum.

6,7-Dimethoxycoumarin, 65 mg (C₁₁H₁₀O₄), m p 145°, M⁺ 206 (base peak) eluted with 15% EtOAc in C₆H₆. Its IR and UV spectra were suggestive of a coumarin chromophore. NMR (CDCl₃) δ 3.94, 3.98 (*s*, 3H each, two Ar CH₃O), 6.32, 7.68 (*d*, 1H, each, *J* 10 Hz, *AB* pattern), 6.90, 6.94 (*s*, 1H each). Methylation of scopoletin gave an identical compound.

Scopoletin, 150 mg (C₁₀H₈O₄), m p and m m p 204°, eluted with 40% EtOAc in C₆H₆. Its IR and UV were suggestive of a phenolic coumarin. NMR (CDCl₃) δ 6.23, 7.58 (*d*, 1H each, *J* 9.5 Hz), 6.83, 6.90 (*s*, 1H each), 3.93 (*s*, 3H, CH₃O), OH confirmed by D₂O shake. MS. An intense M-15 fragment indicative of scopoletin.¹¹

Tembamide,^{12–15} 100 mg (C₁₆H₁₇NO₃), m p 156–157°, eluted with 40% EtOAc in C₆H₆, optically inactive. UV $\lambda_{\max}^{\text{EtOH}}$ 230 (4.34), 275 (3.41), 282 (3.22) nm IR (KBr) ν_{\max} 3495 (OH), 3390 (NH), 1625 cm^{-1} (CONH). NMR (CDCl₃-DMSO-*d*₆, 50%) δ 7.80–8.00 (*m*, 5H, unsubstituted phenyl), 6.91, 7.73 (*d*, 2H each, *J* 9 Hz, *AB* pattern), 4.52 (*q*, 1H benzylic), 4.03 (*t*, 2H), 3.78 (*s*, 3H, CH₃O), 5.33, 2.92 (NH, OH respectively, eliminated by D₂O).

Contrary to an earlier observation,¹³ a conspicuous M⁺ signal (23%) was found at *m/e* 271 in the MS.

The BuOH soluble fraction yielded a glycoside (Feigl Test), 263 mg, m p 208–209°. UV $\lambda_{\max}^{\text{EtOH}}$ 217 (4.09), 325 (4.15) nm NMR (DMSO-*d*₆) δ 6.42, 8.08 (*d*, 1H each, *J* 9 Hz), 7.00–7.90 (*m*, 3H, Ar). MS. M⁺ at *m/e* 324, 162 (base peak, M-sugar residue), 134, 105, 78.

On hydrolysis with 4% H₂SO₄ at 100°, it gave umbelliferone (IR, UV, NMR, MS, m p identical with an authentic sample) and D-glucose (PC, TLC) suggesting its structure as skimmmin.

Other constituents isolated and characterized were umbelliferone (265 mg), marmesin (150 mg), marmmin (4.3 g) and skimmianine (250 mg).

EXPERIMENTAL

Extraction Milled roots (20 kg) was exhaustively percolated with EtOH, the concentrate diluted with 2% tartaric acid, and extracted with hexane followed by EtOAc. On removal of the solvent, the EtOAc soluble residue (23 g) was chromatographed over silica gel (1 kg) and eluted with solvents mentioned in the text to yield the coumarins and alkaloids. The aq. phase was extracted with *n*-BuOH and the soluble residue (15 g) was chromatographed over silica gel (600 g). Elution was effected with 6% MeOH in EtOAc to furnish skimmmin.

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