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RUBONE, A NEW CHALCONE FROM *DERRIS ROBUSTA* SEED SHELLS

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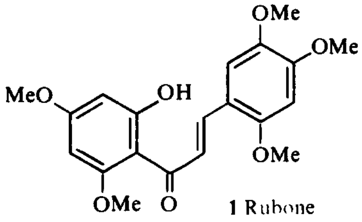
Key Word Index—*Derris robusta*; Leguminosae; chalcone; rubone; 2′-hydroxy-4′,6′,2,4,5-pentamethoxychalcone.

In continuation [1] of our work on the seed shells of *D. robusta*, we wish to report the isolation of a new chalcone rubone from its petrol extract. It analysed for $C_{20}H_{22}O_7$ (M^+ 374). That rubone is a chalcone containing a chelated hydroxyl was shown by absorptions at 3450 and 1618 cm^{-1} in IR; $\lambda_{\max}^{\text{MeOH}}$ 265, 325, 400 nm in UV and a singlet at δ 14.54 (chelated hydroxyl), two doublets each with $J = 16.5$ Hz at 8.1 and 7.8 (H_β and H_α, respectively) in ^{1}H NMR. The location of a chelated hydroxyl at the 2′-position is shown by a bathochromic shift of 52 nm with $AlCl_3$ in UV. Absence of any bathochromic shift [2] with NaOAc in the UV spectrum indicated the absence of a free hydroxyl at the 4′- and 4-positions. ^{1}H NMR showed three singlets at δ 3.93, 3.87 and 3.81 integrating for 3, 9 and 3 protons, respectively, due to five methoxyls. The compound gave a positive Gibb's test and two doublets ($J = 2.5$ Hz) centred at δ 6.07 and 5.92 corresponding to H-5′ and H-3′, respectively, thereby suggesting 2′-hydroxy-4′,6′-dimethoxy substitution for ring A. The remaining three methoxyls are therefore located in ring B. Sharp singlets at δ 7.09 and 6.49, integrating for one proton each, were attributed to H-6 and H-3 protons, respectively. Any other arrangement of three methoxyl groups in ring B would instead give rise to either *meta*- or *ortho*-coupled doublets. Presence of only one hydroxyl in rubone is supported by the ^{1}H NMR of its monoacetate and monomethyl ether. The mass spectrum of the compound is also in agreement [3, 4] with the above assignments. Hence rubone is 2′-hydroxy-4′,6′,2,4,5-pentamethoxychalcone (**1**).



1 Rubone

Isolation of rubone is of biogenetic interest because three isoflavones, namely robustigenin, robustigenin-5-*O*-methyl ether and derrugenin, have earlier been reported [1] from this source.

EXPERIMENTAL

^{1}H NMR spectra were taken at 90 MHz in $CDCl_3$ and chemical shifts are given in δ (ppm) scale relative to TMS; UV spectra were obtained in MeOH and IR spectra as KBr discs.

Isolation. Air-dried and coarsely powdered seed shells (750 g) of *D. robusta* were extracted with hot petrol (bp 60–80°). This extract was concd under red. pres. and subjected to CC over Si gel. On elution with C_6H_6–petrol (1:1) it gave a yellow crystalline compound (90 mg), rubone (**1**). TLC: R_f 0.35 (C_6H_6–EtOAc, 9.5:0.5), 0.45 ($CHCl_3$) and 0.70 (C_6H_6–EtOAc, 9:1), mp 184–86°. $\lambda_{\max}^{\text{MeOH}}$ nm: 265, 325, 400; +NaOAc 262, 328, 400; +$AlCl_3$ 320, 360, 375, 400; +NaOMe 255, 305, 335, 448. $\nu_{\max}^{\text{KBr}}$ cm^{-1}: 3450, 1618, 1608, 1442, 1035 and 828. ^{1}H NMR ($CDCl_3$): δ 3.81 (3H, *s*, $OC\underline{H}_3$), 3.87 (9H, *s*, 3 × $OC\underline{H}_3$), 3.93 (3H, *s*, $OC\underline{H}_3$), 5.92 (1H, *d*, $J = 2.5$ Hz, Ar $\underline{H}_{3'}$), 6.07 (1H, *d*, $J = 2.5$ Hz, Ar $\underline{H}_{5'}$), 6.49 (1H, *s*, Ar $\underline{H}_3$), 7.09 (1H, *s*, Ar $\underline{H}_6$), 7.80 (1H, *d*, $J = 16.5$ Hz, $\underline{H}_\alpha$) and 8.1 (1H, *d*, $J = 16.5$ Hz, $\underline{H}_\beta$), 14.54 (1H, *s*, chelated hydroxyl). MS *m/e* (rel. int.): 374 (M^+, 100), 344(55), 343(95), 221(13), 207(30), 194(75), 181(80), 180(18), 179(60) and 151(60). Acetylation (Ac_2O–Py) gave the monoacetate as yellow needles, R_f 0.15 ($CHCl_3$), 0.40 (C_6H_6–EtOAc, 9:1), mp 143–45°. $\nu_{\max}^{\text{KBr}}$ cm^{-1}: 1756, 1650, 1602, 1045, 1015 and 840. ^{1}H NMR ($CDCl_3$): δ 2.15 (3H, *s*, $OCOC\underline{H}_3$), 3.77 (3H, *s*, $OC\underline{H}_3$), 3.84 (9H, *s*, 3 × $OC\underline{H}_3$), 3.90 (3H, *s*, $OC\underline{H}_3$), 6.30 (1H, *d*, $J = 2.5$ Hz, Ar $\underline{H}_{3'}$), 6.40 (1H, *d*, $J = 2.5$ Hz, Ar $\underline{H}_{5'}$), 6.47 (1H, *s*, Ar $\underline{H}_3$), 6.93 (1H, *d*, $J = 16.5$ Hz, $\underline{H}_\alpha$), 7.20 (1H, *s*, Ar $\underline{H}_6$) and 7.72 (1H, *d*, $J = 16.5$ Hz, $\underline{H}_\beta$). Methylation (Me_2SO_4, K_2CO_3, Me_2CO) gave the monomethyl ether as yellow needles, R_f 0.2 ($CHCl_3$), 0.45 (C_6H_6–EtOAc, 9:1), mp 132–34°. $\nu_{\max}^{\text{KBr}}$ cm^{-1}: 1630, 1594, 1578, 1200, 1038 and 850. ^{1}H NMR ($CDCl_3$): δ 3.73 (3H, *s*, $OC\underline{H}_3$), 3.77 (3H, *s*, $OC\underline{H}_3$), 3.80 (9H, *s*, 3 × $OC\underline{H}_3$), 3.87 (3H, *s*, $OC\underline{H}_3$), 6.12 (2H, *s*, Ar $\underline{H}_{3'}$ and Ar $\underline{H}_{5'}$), 6.43 (1H, *s*, Ar $\underline{H}_3$), 6.83 (1H, *d*, $J = 16.5$ Hz, $\underline{H}_\alpha$), 7.00 (1H, *s*, Ar $\underline{H}_6$) and 7.63 (1H, *d*, $J = 16.5$ Hz, $\underline{H}_\beta$).

REFERENCES

1. Chibber, S. S. and Sharma, R. P. (1979) *Phytochemistry* (in press) and refs. cited therein.
2. Mabry, T. J., Markham, K. R. and Thomas, M. B. (1970) *The Systematic Identification of Flavonoids*, p. 228. Springer, Berlin.
3. Itagaki, Y., Kurosawa, T., Sasaki, S., Chang, C. T. and Chen, F. C. (1966) *Bull Chem. Soc. Jpn* **39**, 538.
4. Van de Sande, C., Serum, J. W. and Vandewalle, M. (1972) *Org. Mass Spectrom.* **6**, 1333.