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WAMPETIN, A FUROCOUMARIN FROM *CLAUSENA WAMPI*

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Key Word Index—*Clausena wampi* (Syn. *Clausena lansium*); Rutaceae; wampetin; furocoumarin; ^{13}C NMR data.

Abstract—A new furocoumarin wampetin has been isolated from *Clausena wampi* (syn. *Clausena lansium*). The structure was established from ^1H NMR, ^{13}C NMR, MS and chemical data.

The aerial and underground parts of *Clausena* species have been studied extensively for the presence of coumarins [1, 2] and carbazoles [3]. Here we report the isolation and characterization of a new furocoumarin (**1**), wampetin, from the root bark of *Clausena wampi* Blanco (Syn. *C. lansium*)†.

Wampetin (**1**), mp 78°, analysed for $C_{21}H_{18}O_6$ ($[M]^+$ 366). It showed IR bands at 1755 cm^{-1} and 1710 cm^{-1} indicative of the presence of an α,β-unsaturated-γ-lactone and α,β-unsaturated-δ-lactone groups. Its cleavage with concentrated sulphuric acid afforded Xanthotoxol [4] indicating that wampetin is a C-8 ether of xanthotoxol. The 200 MHz ^1H NMR spectrum of **1** integrated for 18 protons and the assignments of their chemical shift values are given in Table 1. The proposed structure was supported by decoupling experiments (NMDR technique) and ^{13}C NMR data.

EXPERIMENTAL

The EtOAc extract of powdered and dried root bark of *C. wampi* on CC followed by prep. TLC afforded wampetin (**1**) from EtOAc–Et_2O, mp 78° (uncorr.). MS *m/z* (rel. int.): 366 (3.2), 202

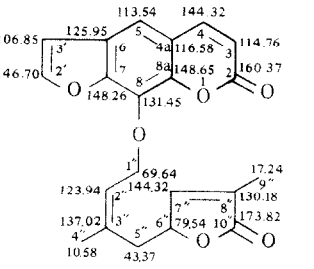

Table 1. ^1H NMR spectral data of compound **1** (200 MHz, $CDCl_3$, TMS as int. stand.)

H	δ	H	δ
2′	7.71 (1H, *d*)	2″	5.72 (1H, *tm*)
3′	6.83 (1H, *d*)	4″	1.79 (3H, *s br*)
3	6.35 (1H, *d*)	5″	2.36 (2H, *m*)
4	7.79 (1H, *d*)	6″	4.92 (1H, *tm*)
5	7.39 (1H, *s*)	7″	6.93 (1H, *dq*)
1″	5.09 (2H, *d*)	9″	1.88 (3H, *t*)

J-values in Hz: $J_{H\text{-}2',H\text{-}3'} = 2.30$; $J_{H\text{-}3,H\text{-}4} = 9.52$; $J_{H\text{-}1'',H\text{-}2''} = 6.6$; $J_{H\text{-}2'',H\text{-}4''} = 0.98$; $J_{H\text{-}5'',H\text{-}6''} = 6.5$; $J_{H\text{-}6'',H\text{-}7''} = 1.6$; $J_{H\text{-}7'',H\text{-}9''} = 1.71$; $J_{H\text{-}6'',H\text{-}9''} = 1.95$.

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†Collected from Forest Research Institute, Dehradun and identified by Mr. Kunwar Naresh Bahadur, Botany Division.

), 201 (7.5), 174 (10), 173 (3.3), 147 (3.2), 146 (2.1), 145 (4.2), (1), 118 (1), 117 (1), 97 (17), 89 (5.5), 69 (4.2), 63 (3.2), 41 (7.3), 2.6) and 18 (8.4). IR ν_{max}^{KBr} cm^{-1}: 2920, 1755, 1710, 1615, 1590,), 1400, 1325, 1290, 1210, 1180, 1150 and 1025. ^1H NMR) MHz, $CDCl_3$): Table 1. ^{13}C NMR [5] (50 MHz, $CDCl_3$, ɔable assignments, OFR): singlets 173.82 (C-10″), 160.37 (C-48.65 (C-8a), 148.26 (C-7), 137.02 (C-3″), 131.45 (C-8), 130.18 ;″), 125.95 (C-6), 116.58 (C-4a); doublets 146.70 (C-2′), 144.32 ł, C-7″), 123.94 (C-2″), 114.76 (C-3), 113.54 (C-5), 106.83 (C-79.54 (C-6″); triplets 69.64 (C-1″), 43.37 (C-5″); quartets 17.24 ″), 10.58 (C-4″).

'ydrolysis of wampetin. Wampetin (**1**) (30 mg) was dissolved in l HOAc to which 2 drops of conc H_2SO_4 were added. The tion mixture was heated for 30 min, cooled, diluted with ice) and extracted with EtOAc. The EtOAc layer was washed ı H_2O, dried (Na_2SO_4) and the solvent removed to afford le product (20 mg) which on purification by prep. TLC on a gel yielded xanthotoxol (6 mg) mp 240–243° (uncorr.), $_{max}^{KBr}$ cm^{-1}: 3400 (–OH), 1730, 1700 (coumarin carbonyl).

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)LUBOLIN, A 4-PHENYL-2H-1-BENZOPYRAN-2-ONE FROM *DALBERGIA VOLUBILIS*

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Key Word Index—*Dalbergia volubilis*; Leguminosae; young branches; 7-hydroxy-4-methyl coumarin; dalbergin; biochanin-A; 7-hydroxy-4-(3-hydroxy-4-methoxyphenyl)-2H-1-benzopyran-2-one.

stract—From the ether soluble portion of a methanolic extract of young non-green branches of *Dalbergia volubilis*, ɔsterol, 7-hydroxy-4-methyl-2H-1-benzopyran-2-one, dalbergin, *p*-hydroxy cinnamic acid, biochanin-A and a new 4-ɔnylcoumarin, volubolin, have been isolated. The structure of volubolin as 7-hydroxy-4-(3-hydroxy-4-methoxy ɔnyl)-2H-1-benzopyran-2-one has been established on the basis of spectral and chemical evidence. Cooccurrence of ıethyl- and 4-phenyl-coumarins with isoflavones is of biogenetic interest.

ıwers and stem bark of *Dalbergia volubilis* have been rked out earlier [1–5], but no work has been done on young non-green branches. We have now isolated five ɔwn compounds: dalbergin, biochanin-A, sitosterol, *p*-lroxycinnamic acid, 7-hydroxy-4-methyl-2H-1-benzo-ɔan-2-one and a new 4-phenyl-2H-1-benzopyran-2-one ich we name volubolin.

The new compound, volubolin, analysing for $_6H_{12}O_5$, showed a deep blue fluorescence under UV ıt and λ_{max} at 224, 284 and 340 nm. It gave a red colour h alcoholic ferric chloride and showed strong absorp-n bands at 1715 and 3300 cm^{-1} indicating the presence a lactone carbonyl and a hydroxylic function in it. The ɜence of strong absorption bands at 1624, 1607, 1580 l 1546 cm^{-1} in its IR spectrum indicated that the positions 5 and 8 of the coumarin nucleus were free [6]. Since it gave a pink colour with Mg–HCl which changed from blue to brown, the compound was considered to be a 4-phenylcoumarin [7]. In its ^1H NMR spectrum it showed a one proton singlet at δ 5.93 which could be assigned to a proton at position 3 of the 4-phenyl-coumarin. Two one proton singlets at δ 9.85 and 10.3, exchangeable with deuterium, could be assigned to two phenolic protons while a three proton singlet at δ 3.8 could be attributed to three methoxyl protons. It showed a prominent molecular ion at m/z 284 followed by a loss of CO to give a strong $[M-28]^+$ peak at m/z 256 which further lost a molecule of CO to yield a peak at m/z 228 $[M-56]^+$. Other prominent peaks at m/z 269 $[M-15]^+$ and m/z 241 $[M-43]^+$ could be accounted for