hot 95% EtOH. The combined extracts were filtered, concd to a small vol. *in vacuo* and re-filtered. The two pigments, C_1 and C_2, were purified by prep PC in BAW, 5% HOAc and BEW. R_f data for C_1 and C_2 are: BAW 0.64, 0.64; 5% HOAc 0.06, 0.16 and BEW 0.66, 0.66. Two other yellow pigments were isolated but were not present in sufficient amount for analysis. Their UV spectra suggested that they may be chalcones. When paper strips of C_2 bands were kept for one to two days before elution with EtOH the eluate showed a complete absence of C_2 and contained instead the corresponding flavanone (R_f 0.65 in BAW; 0.66 in BEW; 0.54 in H_2O; 0.59 in 5% HOAc; 0.64 in 30% HOAc).

Hydrolysis of the pigments. Controlled acid hydrolysis was carried with 10% aq. HOAc (3.5 hr under reflux); total acid hydrolysis was carried with 2 N HCl (2 hr at 100°). Naringenin was identified by CO-PC with an authentic sample (5 solvents) and UV spectral analysis with usual shift reagents. D-Glucose was identified by the use of glucose oxidase, CO-PC (4 solvents) and GLC of its TMS ether [5]. The aglycone: sugar ratio was determined according to ref. [6].

Methylation of F. The flavanone was methylated (Me_2SO_4–K_2CO_3–Me_2CO) and hydrolysed as in ref. [3]. Alkaline degradation of the partially methylated aglycone was achieved as in ref. [7]; *p*-coumaric acid was identified by PC (3 solvents), TLC on Si gel (C_6H_6–HOAc–H_2O, 2:1:1, upper phase) and paper electrophoresis at pH 4.5 and 8.7; di-*O*-methylphloroglucinol was identified by comparison with an authentic sample (TLC on Si gel; 3 solvents); 2,3,4,6-tetra-*O*-methyl-D-glucose was identified by TLC on Si gel ($CHCl_3$–EtOAc. 1:1) and PC [8].

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A NEW CHALCONE AS A NATURAL MOLLUSCICIDE FROM *POLYGONUM SENEGALENSE*

ASAFU MARADUFU* and JOHN H. OUMA†

* The International Centre of Insect Physiology and Ecology, P.O. Box 30772, Nairobi, Kenya; † National Health Laboratory, Nairobi, Kenya

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Key Word Index—*Polygonum senegalense*; Polygonaceae; molluscicide; 2′,4′-dihydroxy-3′,6′-dimethoxychalcone.

There is a pressing need in the tropical world for cheap and simple molluscicides to control vector snails of parasites responsible for schistosomiasis and other snail-borne diseases. Of the numerous molluscicides available, few [1] are from botanical sources. We now report the structure of a molluscicide from an East African plant.

Crude aqueous extracts of seeds and leaves of *Polygonum senegalense* were found [2] to have molluscidal activity against *Biomphalaria pfeifferi* and *B. sudanica* snails. One of the active principles is the chalcone **1**, (2′,4′-dihydroxy-3′,6′-dimethoxychalcone). At 40 ppm concentration, **1** is 100% lethal to *B. pfeifferi* and *B. sudanica* in less than six hours.

Hydrophilic and hydrophobic fractions of a methanolic extract of seeds and leaves of the plant both contain molluscicides. A hydrophobic fraction in benzene gives, after chromatography on silica gel, the chalcone **1** as orange crystals mp 124–125° (CH_2Cl_2–*n*-hexane) M^+ 300, $C_{17}H_{16}O_5$. The UV spectrum of **1** in MeOH has bands at λ_{max} 211 nm (ε 31 500) and 348 nm (ε 27 000). Addition of NaOMe, $AlCl_3$, $AlCl_3$–HCl, NaOAc, NaOAc–H_3BO_3 to **1** in MeOH, cause bathochromic shifts of 44, 36, 32, 44 and 48 nm resp. of the long wavelength band with no increase in intensity. These shifts suggest that **1** is a flavonoid [3–5] with hydroxyl groups located at C-2′ and C-4′. 2′-Hydroxy-4′,6′-dimethoxychalcone [6] which is also present in *P. senagalense* extracts does not give a bathochromic shift with NaOAc but gives one of 38 nm with $AlCl_3$. That **1** has phenolic hydroxyl groups is confirmed by the presence of a sharp band at ν ($CHCl_3$) 3455 cm^{-1} in the IR spectrum and the appearance of acetoxyl singlets at δ 2.23, and 2.4 in the PMR spectrum (recorded in $CDCl_3$) of the acetylated product of **1**. The α,β-unsaturated C=O group absorbs at 1628 cm^{-1}.

In the 60 MHz PMR spectrum of **1** in $CDCl_3$ methoxyl signals appear at δ 4.04 and 4.06. A singlet appears at δ 6.17 (1 H). Aromatic, α, and β, protons (7 H) resonate between 7.38 and 8.12 ppm. Hydroxyl protons resonate at 14.42 ($O\underline{H}$-2′ [6] and 6.57 ppm ($O\underline{H}$-4′). The α and β-protons of **1** are well resolved in the spectrum of the di-*O*-trimethylsilyl derivative in CCl_4 and C_6D_6; thus in CCl_4 the α-proton resonates at 6.86 ppm $J_{\alpha,\beta} = 17$ Hz while in C_6D_6 the β-proton signal is at 7.74 ppm. $J_{\beta,\alpha} = 17$ Hz. In the MS of **1** (see below) ring B is unsubstituted; therefore the singlet at δ 6.17 arises from Ar.$\underline{H}$ of ring A and is typical of H-3′ or H-5′ signals in 2′, 4′, 6′-trioxygenated chalcones and other similarly constituted flavonoids [4, 6]. Furthermore, H-5′, moves downfield by 0.51 ppm in the spectrum of the diacetate of **1** in $CDCl_3$ and suggests [7] that in this derivative H-5′ is *ortho* or alternatively *para* to an acetoxyl group. Again, in the PMR spectrum of the di-*O*-trimethylsilyl derivative of **1** in CCl_4–C_6D_6 a benzene-induced shift of 0.5 ppm takes place for only one methoxyl group which implies [8, 9], that **1** has a methoxyl group *ortho* to an aromatic ring proton.

The ^{13}C NMR spectrum signals of **1** in DMSO-d_6 occur at similar resonances as those recently reported for chalcones [10, 11]: δ 193.4 (C=O); δ 160.0, 159.2, 158.5 1 $\underline{C}$—Me, 2 $\underline{C}$—OH); δ 143.1 (C-β); δ 136.2 (C-1); δ 131.2 (C-4); δ 130.2 (C-2, C-6, C-3′); δ 129.6 (C-3, C-5); δ 128.7 C-α); δ 106.6 (C-1′); δ 92.8 (C-5′); δ 61.1, 57.2 (2 $O\underline{C}H_3$). In the ^{13}C-^1H coupled spectrum the C-5′ signal is a doublet of 162.6 Hz and is unaffected when the spectrum is recorded after D_2O exchange. This implies that C-5′ experiences no long range coupling with $O\underline{\underline{H}}$-2′ and is therefore *para* to C-2′ [12]. All other A ring carbons show a coupling of 2–5 Hz due to OH-2′ hydrogen bonded proton [12]. A *meta* coupling of 5 Hz between C-1′ and H-5′ is also present.

Fragmentation of **1** in the mass spectrometer follows fragmentation patterns similar to those described for chalcones [13]. Significant peaks appear at *m/e* 223 (M-77), 197 (M-103), 196 (M-104), 131, 104. 103, and 77. The rest of the spectrum is dominated by peaks at *m/e* 181, 167, 153, 139, 84, 69, and 49, all of which result from cleavage of ring A.

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STRUCTURE OF TWO *C*-DIGLYCOSYLFLAVONES FROM *CANNABIS SATIVA**

Alvin B. Segelman*, Florence P. Segelman*, Aura E. Star*, Hildebert Wagner† and Otto Seligmann†

*Department of Pharmacognosy, College of Pharmacy, Rutgers—The State University, New Brunswick, NJ 08903, U.S.A.; †Institut für pharmazeutische Arzneimittellehre der Universität München, Karlstraße 29, D-8000 München 2, BRD

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From a biologically active [11] aqueous extract of the aerial parts of *C. sativa* after pre-extraction with petroleum and MeOH, a flavonoid-rich butanol fraction has been obtained. Subsequent fractionation and purification by Sephadex and polyamide chromatography resulted in the isolation of three flavone *C*-glycosides A, B and C. Glycoside A with the lowest R_f (0.25) on cellulose TLC with *t*-BuOH–HOAc–H_2O(3:1:1) and the mp 266–267° was identified as orientin by comparison of the physical data with those of an authentic sample. The MS of the permethylated product (M^+ 564) was in accordance with the reported values [2]. The second flavonoid (glycoside B) had mp 218–230° and R_f 0.72. The third flavonoid (glycoside C) differed only slightly from the latter (mp 213.5–214) but was well separated by TLC (R_f 0.61). Compounds B and C could each be hydrolyzed by HCl compound B to a mixture of vitexin/isovitexin and compound C to orientin/isoorientin. In each case the 8-*C*-glucoside was a second product of hydrolysis, it was assumed that the compounds B and C were *O*-glucosides of vitexin and orientin respectively. Similar findings but without the presentation of any detailed physical and

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*Part 8 in the series '*Cannabis sativa* L. (marijuana)'. For Part 7 see Segelman, A. B. and Segelman, F. P. (1976) *J. Chromatogr.* **123**, 9. Part 15 in the series flavone-*C*-glycosides. Presented at the 17th Annual Meeting of the American Society of Pharmacognosy, Cable, Wisconsin, 14 July 1976.