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Structural Studies of a New Flavone Glycoside from the Seeds of *Cassia nodosa*

R. D. TIWARI and K. S. SINHA

Department of Chemistry, Allahabad University, Allahabad-211 002

IN an earlier communication[1] isolation of anthraquinone and flavone glycosides from the seeds of *Cassia nodosa* and the structural studies of the anthraquinone glycosides had been reported. The present report deals with the structural studies of the flavone glycoside.

The flavone glycoside, $C_{23}H_{24}O_{10}$, when hydrolysed with ethanolic hydrochloric acid, gave rhamnose (PC, osazone) and an aglycone $C_{17}H_{14}O_6$. On the basis of standard colour reactions[2] and spectral data[3,4] and chemical degradation studies, the aglycone has been identified as 5,4'-dihydroxy 7,3'-dimethoxy flavone (velutin).

The aglycone showed bathochromic shift with aluminium chloride but the glycoside did not, indicating thereby that the rhamnose molecule is attached to the hydroxyl group at position 5. On periodate oxidation two moles of periodate per mole of the glycoside were consumed producing one mole of formic acid which showed that the sugar rhamnose is in the pyranose form. The hydrolysability of the glycoside by Taka diastase showed the presence of α-linkage.

The aglycone, velutin, had been reported earlier from some plant sources[5] but the glycoside is a new one, being reported for the first time.

Experimental

Isolation and purification of the glycoside has been described in an earlier communication[1]. Glycoside, m.p. 138°; Found C, 60.02; H, 5.99. Calcd. for $C_{23}H_{24}O_{10}$; C, 60.00; H, 6.00%. UV λ_{max}^{EtOH} 265, 345; +$AlCl_3$ 280, 350; +NaOEt 290, 398 nm; ir ν_{max}^{KBr} 3440, 2950, 2880, 1185, 1605, 847, 730, 720 cm^{-1}; nmr (DMSO) δ : 3.90 (s, 6H, OCH_3); 4.20 (s, 1H, H-1″ rhamnosyl); 3.50-4.00 (br, 4H, sugar protons); 6.38 (d, J=2.5 Hz, 1H, C-6); 6.75 (d, J=2.5 Hz, 1H, C-8); 6.88 (s, 1H, C-8); 7.02 (d, J=8.5 Hz, 1H, C-5'); 7.60 (dd, J=2.5, 8.5 Hz, 2H, C-2', C-6').

Hydrolysis: The glycoside (200 mg) was hydrolysed using 7% ethanolic hydrochloric acid (25 ml) under reflux for 6 hr, ethanol distilled off and the aglycone crystallised from ethyl acetate, m.p. 225° (C, 64.70; H, 4.70, Calcd. for $C_{17}H_{14}O_6$: C, 65.00; H=4.50%). Acetate: (Ac_2O/Py) m.p. 207°, acetyl percentage, found: 20; calcd. for $C_{17}H_{12}O_6(COCH_3)_2$; 21.60. Methyl ether: ($Me_2SO_4/K_2CO_3$) m.p. 192°, methoxyl percentage, found 32.92; calcd. for $C_{15}H_6O_2(OCH_3)_4$; 36.26). UV λ_{max}^{EtOH}: 268, 348; +$AlCl_3$ 268, 383; +NaOEt 293, 398 nm; ir ν_{max}^{KBr}: 3440, 2950, 2850, 1180, 1660, 1605, 845 cm^{-1}; nmr(DMSO) δ : 3.85 (s, 3H, OCH_3); 3.92 (s, 3H, OCH_3); 6.35 (d, J=2.5, 1H, C-6); 6.75 (d, J=2.5 Hz, 1H, C-8); 6.90 (s, 1H, C-3); 7.01 (d, J=8.5 Hz, 1H, C-5); 7.60 (dd, J=2.5 Hz and 8.5 Hz, C-2', C-6').

Periodate oxidation: The glycoside (20 mg) was dissolved in ethanol (25 ml) and distilled water (25 ml), treated with 0.1 *M* sodium *meta*-periodate (25 ml) and the reaction mixture allowed to stand for 48 hr. The amounts of periodate consumed and formic acid liberated were estimated by titrimetric method of Hirst and Jones[6]. For each mole of the glycoside, 1.8 mole of periodate was consumed and 1.2 mole of formic acid liberated.

Enzymic hydrolysis: The glycoside was hydrolysed using Taka diastase at 25-30° for three days when L(−)-rhamnose was found in the hydrolysate (PC).

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