

NOTES

Gossypetin-7-0- β -D-Glucopyranoside, A Novel Flavonoid Glycoside from *Impatiens Scabrida* D.C.

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Phytochemical examination of *Impatiens scabrida* resulted in the isolation and identification of a novel flavonoid glycoside Gossypetin-7-0- β -D-glucopyranoside.

*Impatiens scabrida*¹ D.C. (N.O. Balsaminaceae) is distributed throughout India. It is an annual herb with sessile and narrow leaves. The Ayurvedic system of medicines describes the oil of the plant to be used as a semidrying oil. The present paper deals the isolation and structural elucidation of a novel flavonoid glycoside.

The methanolic extract of the water soluble part of the 95% concentrated ethanolic extract of dried and powdered plant, when worked up by C. C. gave crude glycoside (0.48%). Methanolic extract on TLC examination showed two spots which was separated by column chromatography on Si-gel G. One of the compd. eluted with ethylacetate : methanol (1 : 3) gave a brown amorphous compound m.pt. 208°C, molecular formula C₂₁H₂₀O₁₃, M⁺480 (R_f = 0.64). It gave positive Molisch test and responded to all the colour tests for flavonoidal glycoside^{2,3} [1], the study of another compd is in progress.

[1] On acid hydrolysis with 7% ethanolic H₂SO₄ gave an aglycone [2] and the sugar moiety, which was indentified as D-glucose (by Co-Pc and Co-TLC).

The aglycone C₁₅H₁₀O₈, m.pt. 308°, M⁺318, m/e = 290, 288, 169, 168, 140, 143, 132, crystallised in yellow needle, UV. $\lambda_{\max}^{\text{MeOH}}$ 256, 270, 375 nm, $\lambda_{\max}^{\text{AlCl}_3}$ 257, 272, 446 nm, $\lambda_{\max}^{\text{NaOAc}+\text{H}_3\text{BO}_3}$ 266, 395 nm, IR ν_{\max}^{KBr} 3388, 2916, 1680, 1600, 1280, 1114, 810 cm⁻¹, ¹HNMR spectrum of hexa-acetyl derivative of the aglycone showed signals at δ = 6.48 (d, J = 2, C₆-1H),

δ = 7.53 (d, J = 2.5, C₂, -¹H), δ = 6.88 (d, J = 8.5, C₅, -¹H), δ = 7.63 (d, d, J = 2.5, 9, C₆, -¹H), δ = 2.34 (S, 3H, C₃, -OAC), δ = 2.36 (S, 3H, C₄, -OAC), δ = 2.48 (S, 3H, C₃-OAC), δ = 2.45 (S, 3H, C₅-OAC), δ = 2.41 (S, 3H, C₇-OAC), δ = 2.39 (S, 3H, C₈-OAC).

A peak at 3388 cm⁻¹ in the IR spectrum of the [2] showed the presence of OH groups in it. The number of OH groups were estimated by the acetylation with AC₂O/pyridine yielded hexa acetyl derivative, thereby confirming the presence of six hydroxyl groups in it.

Formation of 4 : 5 dihydroxy-benzoic acid (protocate cheuc acid) on alkaline degradation of the [2] confirmed the presence of OH groups at C-3' and C-4' in the [2].

A bathochromic shift of 38 nm in the band I and hypsochromic shift of

34 nm upon addition of AlCl_3 and H_3BO_3 in the UV Spectrum of [2] further confirmed the presence of OH groups at C_3 , and $\text{C}-4'$ respectively⁴.

Formation of phloroglucinol on alkaline degradation of the [2] showed the presence of two $-\text{OH}$ groups at $\text{C}-5$ and $\text{C}-7$ respectively.

Spectral shift⁵ with AlCl_3 (bathochromic shift 24 nm) and CH_3COOH (bathochromic shift 34 nm) indicated the presence of $-\text{OH}$ groups at $\text{C}-5$ and $\text{C}-7$ respectively.

Yellow fluorescence of the [2] in UV light⁶ and the spectral shift with AlCl_3 in presence of HCl relative to band (I) in MeOH indicating a free 3-OH group in the aglycone.

A characteristic colour reaction with Zn/Hg and zirconium oxychloride in citric acid⁷ further suggested the presence of $-\text{OH}$ group at $\text{C}-3$.

The [2] gave a red complex with zirconium nitrate solution soluble in HCl showing the presence of a hydroxyl group at position C_8 ⁸, which was further confirmed by a signal at $\delta = 2.39$ in the ^1H NMR spectrum of acetylated aglycone.

Formation of above degradation products established the identity of the [2] as 3 : 5 : 7 : 8 : 3' : 4'—hexahydroxy flavone (Gossypetin).

The aqueous hydrolysate on chromatographic study revealed the presence of only D-glucose (by CO-PC and CO-TLC). The sodium metaperiodate oxidation⁹ of the [1] consumed 2.01 mole of periodate and liberated 1.07 mole of formic acid indicating the presence of a monosaccharide and also that it was in the pyranose form.

[1] on permethylation¹⁰ followed by hydrolysis yielded 2 : 3 : 4 : 6-tetra-O-methyl-D-glucose (by CO-PC and CO-TLC) confirmed that the sugar was attached via its $\text{C}_1\text{-OH}$ to the $\text{C}_7\text{-OH}$ of the aglycone.

[1] on hydrolysis with emulsion¹¹ indicated β -linkage between the sugar and the aglycone thereby confirming that the glycoside was Gossypetin-7-O- β -D-glucopyranoside which was further supported by its spectral analysis, UV $\lambda_{\text{max}}^{\text{MeOH}}$ 254, 272, 363 nm, $\lambda_{\text{max}}^{\text{AlCl}_3}$ 295, 364 nm, $\lambda_{\text{max}}^{\text{H}_3\text{BO}_3}$ 224, 270, 362 nm, IR $\nu_{\text{max}}^{\text{KBr}}$ 3400, 2910, 1675, 1598, 1112, 1275, 815 cm^{-1} , ^1H NMR $\delta = 6.46$ (d, $J = 2$, $\text{C}_6\text{-1H}$).

$\delta = 7.54$ (d, $J = 2.5$, C_2 , $-\text{1H}$), $\delta = 6.86$ (d, $J = 8.5$, C_5 , $-\text{1H}$), $\delta = 7.61$ (d, $J = 2.5$, C_6 , $-\text{1H}$), $\delta = 2.31$ (s, 3H, C_3 , $-\text{OAC}$), $\delta = 2.35$ (s, 3H, C_4 , $-\text{OAC}$), $\delta = 2.47$ (s, 3H, $\text{C}_3\text{-OAC}$), $\delta = 2.44$ (s, 3H, $\text{C}_5\text{-OAC}$), $\delta = 2.40$ (s, 3H, $\text{C}_8\text{-OAC}$), $\delta = 5.7$ (d, $J = 7$, $\text{C}_1\text{-anomericproton}$), $\delta = 5.46$ (m, 6-protons of sugar residue), $\delta = 2.06$ (s, 3H, $\text{C}_2\text{-OAC}$), $\delta = 2.10$ (s, 3H, $\text{C}_3\text{-OAC}$), $\delta = 2.02$ (s, 3H, $\text{C}_4\text{-OAC}$), $\delta = 3.92$ (s, 3H, $\text{C}_6\text{-OAC}$), $\text{M}^+\text{480}$, $\text{m/e} = 318, 290, 288, 169, 168, 140, 134, 132$.

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