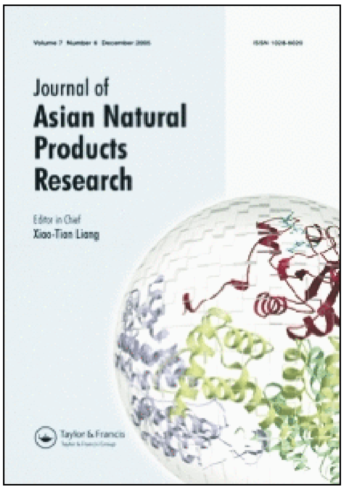

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Note

A new chromone from *Ficus lyrata*

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A new chromone, named 5,6-dihydroxy-2-methylchromone (FL-2), along with seven known flavonoids, 5-hydroxy-7,3,3′,4′-tetramethoxyflavone (FL-3), 5,4′-dihydroxy-6,7,8-trimethoxyflavone (FL-4), 5,4′-dihydroxy-7,8-dimethoxyflavone (FL-5), 4-methoxychalcone (FL-6), 7,4′-dimethoxyapigenin (FL-7), 5,7,4′-trihydroxy-2′,3′,6′-trimethoxyisoflavone (FL-8 a rare flavonoid), acacetin-7-*O*-glucoside (FL-9) and acacetin-7-*O*-neohesperidoside (FL-10), and β-sitosterol-D-glucoside (FL-1) have been isolated from the leaves of *Ficus lyrata*. Their structures have been established on the basis of chemical and spectral evidence (IR, UV, ^{1}H NMR, ^{13}C NMR and mass spectra).

Keywords: *Ficus lyrata*; Moraceae; Chromone; 5,6-Dihydroxy-2-methylchromone; Flavonoids; Terpenoidic glycoside

1. Introduction

Ficus is a large genus of trees, shrubs and often climbers, with milky juice, widely distributed throughout the tropics of both hemispheres but particularly abundant in South-East Asia and Polynesia. About 65 species are found in India. All species of *Ficus* yield latex-containing caoutchouc [1]. Many species of *Ficus* are medicinally important [2]; *Ficus lyrata* is used to treat hypothermic, diuretic and CNS activities [3]. The medicinal importance and scanty work carried out on *Ficus lyrata* accelerated our comprehensive investigation of it. The present paper deals with the isolation and characterization of a new chromone, 5,6-dihydroxy-2-methylchromone (FL-2), along with the nine known compounds β-sitosterol-D-glucoside (FL-1) [4,5], 5-hydroxy-7,3,3′,4′-tetramethoxyflavone (FL-3) [6], 5,4′-dihydroxy-6,7,8-trimethoxyflavone (FL-4) [7], 5,4′-dihydroxy-7,8-dimethoxyflavone (FL-5) [8–10], 4-methoxychalcone (FL-6) [11], 7,4′-dimethoxyapigenin (FL-7) [12], 5,7,4′-trihydroxy-2′,3′,6′-trimethoxy isoflavone (FL-8 a rare flavanoid) [13], acacetine-7-*O*-glucoside (FL-9) [14] and acacetin-7-*O*-neohesperidoside (FL-10) [15].

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2. Results and discussion

Leaves of *Ficus lyrata* (2.5 kg) were dried under shade and powdered. After being defatted with light petroleum (60–80°C), they were then extracted exhaustively with chloroform to yield fraction A and finally with methanol to give fraction B. The chloroform extract (fraction A) responded positively for the colour reaction of flavonoids [16]. Repeated column chromatography of the appropriate fractions followed by fractional crystallization afforded TLC homogenous substances, labelled FL-1-8. These compounds have been identified as β-sitosterol-D-glucoside (FL-1), 5,6-dihydroxy-2-methylchromone (FL-2) a new compound, 5-hydroxy-7,3,3′,4′-tetramethoxyflavone (FL-3), 5,4′-dihydroxy-6,7,8-trimethoxyflavone (FL-4), 5,4′-dihydroxy-7,8-dimethoxyflavone (FL-5), 4-methoxychalcone (FL-6), 7,4′-dimethoxyapigenin (FL-7) and 5,7,4′-trihydroxy-2′,3′,6′-trimethoxyisoflavone (FL-8). The methanol extract (fraction B) also gave a positive test for flavonoids [16]. Repeated column chromatography over silica gel and fractional crystallization afforded two crystalline compounds, labelled FL-9 and FL-10, that have been identified as acacetine-7-*O*-glucoside (FL-9) and acacetin-7-*O*-neohesperidoside (FL-10). The characterization of FL-2 is discussed below.

FL-2 was obtained by eluting the column with benzene-ethyl acetate (10:1); $C_{10}H_8O_4$, mp 172–74°C. It was crystallized from chloroform–methanol as light cream needle-shaped crystals (65 mg). It gave a greenish brown colour with alcoholic ferric chloride, indicating a phenolic hydroxyl group. It responded negatively to Shinoda's test [16], which supported the absence of the flavone nucleus. Elemental analysis along with the molecular ion peak at *m*/*z* 192 agreed with the molecular formula $C_{10}H_8O_4$. The characteristic IR showed a chelated OH group at 3244 cm^{-1}, and a carbonyl group at 1648 cm^{-1}. Its UV spectrum displayed absorption maxima at 220, 261 and 320 nm, corresponding to a chromone nucleus [17–19]. A bathochromic shift of 20 nm with $AlCl_3$ indicates a hydroxyl group at the 5-position, while the hypsochromic shift with $AlCl_3$/HCl points to an *ortho*-dihydroxyl group. Thus OH groups are clearly indicated at the 5 and 6-positions of the chromone nucleus.

Its ^{1}H NMR spectrum (table 1) showed a sharp singlet of three protons at δ 2.35, assigned to methyl group, while a pair of ortho-coupled doublets indicative of one proton each at δ 6.79 ($J = \mathbf{9.0\,Hz}$) and δ 7.0 ($J = \mathbf{9.0\,Hz}$) were ascribed to H-7 and H-8, respectively. The remaining singlet of one proton at δ 6.12 may be assigned to C-2 or C-3 protons. The possibility of an H-2 proton is ruled out as it appears at upfield [19]. Thus the methyl group can only be placed at C-2. This was further supported by the ^{13}C NMR spectrum. The assignments of corresponding carbons are given in the table 2. The above-assigned structure was also confirmed by the mass spectrum (scheme 1), which

Table 1. ^{1}H NMR spectral data of FL-2*.

Assignment	δ *(ppm)*
CH_3	2.35 (s)
H-3	6.12 (s)
H-7	6.79 (d, $J = 9.0$ Hz)
H-8	7.10 (d, $J = 9.0$ Hz)
5-OH	11.0 (s)
6-OH	9.57 (br s)

*Spectrum recorded in DMSO-d_6 at 300 MHz, using TMS as internal standard.

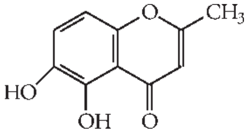
Table 2. ^{13}C NMR spectral data of FL-2*.

Position	*δ (ppm)*
C-2	160.26
C-3	112.14
C-4	171.10
C-5	153.98
C-6	149.44
C-7	132.19
C-8	110.19
C-9	143.46
C-10	115.51
CH_3	18.26

*Spectrum recorded in DMSO-d_6 at 300 MHz, using TMS as internal standard.

shows the molecular ion peak at *m*/*z* 192. The fragment ions [20] are rationalized in scheme 1.

On the basis of the above evidences, FL-2 was identified as 5,6-dihydroxy-2-methylchromone (**1**), which is reported for the first time.



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3. Experimental

3.1 General experimental procedure

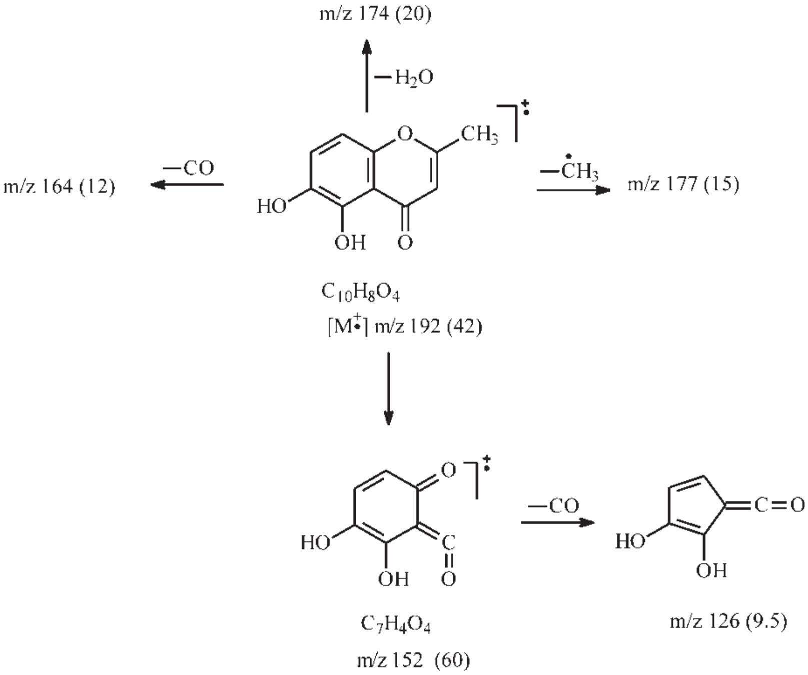
Melting points were recorded on a Kofler block and are uncorrected. IR spectra were taken on a Shimadzu IR-408 Perkin Elmer 1800 (FTIR). The MS and ^{1}H NMR spectra were obtained from different institutes, both in and outside the country. MS spectra were mostly measured in E.I. mode at 70 eV with a JEOL D-300, while the NMR spectra were usually recorded on a JEOL 4H-100 MHz, Perkin Elmer R-32 (90 MHz), Bruker dpx 200 MHz and DRX 300 MHz in $CDCl_3$ using TMS as internal standard. Silica gel G (Merck, 60–120 mesh) was used for column chromatography (cc). Pre-coated silica gel plates (Merck) were used for analytical TLC.

3.2 Plant material

Leaves of *Ficus lyrata* were collected from A.M.U. Campus, Aligarh, India, and identified by Professor Wazahat Hussain, Department of Botany, A.M.U., Aligarh, India. A voucher specimen has been deposited at the Department of Botany, A.M.U., Aligarh.

3.3 Extraction and isolation

Leaves of *Ficus lyrata* (2.5 kg) were dried under shade and powdered. The powdered leaves were then defatted with light petroleum (60–80°C) and extracted thoroughly twice with hot



Scheme 1.

chloroform to yield fraction A. Finally, the leaves were extracted twice with hot methanol to give fraction B.

Fraction A, a gummy greenish mass, gave a positive test for flavonoids. TLC examination in different solvent systems [benzene–pyridine–formic acid (36:9:5) and toluene–ethyl formate–formic acid (5:4:1)] showed that it was a complex mixture. Therefore, it was chromatographed over silica gel column. The column was eluted successively with light petroleum, light petroleum–benzene mixtures, benzene, benzene–ethyl acetate mixtures, ethyl acetate and methanol respectively. Repeated column chromatography of the fractions followed by fractional crystallization afforded TLC homogeneous substances, labelled as FL-1-8. Fractions showing similar behaviour on TLC examination and the same IR spectra were combined.

3.4 *FL-2*

FL-2 was obtained from the column with benzene–ethyl acetate (10:1). It was crystallized from chloroform as light cream needle-shaped crystals (65 mg), mp 172–74°C; it gave a greenish brown solution with alcoholic ferric chloride. (Elemental analysis: found (%) C, 62.48; H, 4.14; calcd. for $C_{10}H_8O_4$, C, 62.5; H, 4.16.) IR (KBr) ν_{max} (cm^{-1}): 3244 (OH), 1648 (C=O), 1600, 1512, 1384, 1307. UV (MeOH) λ_{max} (nm): 220, 261, 320, 323 (sh); (+NaOAc) 220, 262, 319, 323 (sh); (+NaOAc/H_3BO_3) 219, 269, 327; (+$AlCl_3$) 220, 270, 340, 362 (sh); (+$AlCl_3$/HCl) 219, 259, 318, 321 (sh); (+NaOMe) 219, 262, 320, 322 (sh). ^1H NMR (300 MHz, DMSO-d_6): see table 1. ^{13}C NMR (300 MHz, DMSO-d_6): see table 2.

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