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A NEW FLAVANONE GLYCOSIDE FROM *Clausena pentaphylla*

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From the ethyl acetate extract of the roots of Clausena pentaphylla, a *flavanone glycoside, 5,7-dihydroxy-3′,4′-dimethoxyflavanone 6*-C-*[α-rhamnopyranosyl-(1→6)]-β-glucopyranoside (**1**), was isolated. The isolated compound was characterized by UV, IR, and NMR (^{1}H, ^{13}C) studies.*

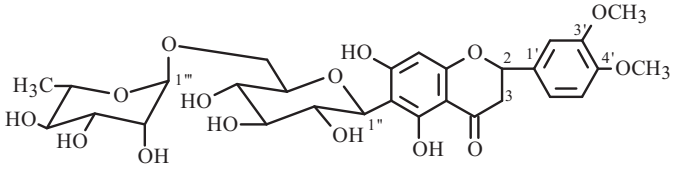
Keywords: Rutaceae, *Clausena pentaphylla,* flavanone glycoside.

Clausena pentaphylla DC. belongs to the family Rutaceae, which is extensively cultivated throughout India and Bangladesh. It is widely used as a folk medicine for the treatment of diarrhea, renal colic, vesical pain, renal calculi, and pain of hepatitis. It is also used to treat uterine pain and colic and in other disorders. A compound, clausmarin, isolated from *C. pentaphylla* showed spasmolytic activity and also reduces blood pressure [1–3]. As part of our investigations on the chemical constituents of medicinal plants of the Rutaceae family found in Shahjahanpur [4–6] District, this paper deals with the isolation and characterization of a new flavanone glycoside from the roots of *Clausena pentaphylla*.

Compound **1** was isolated from the ethyl acetate extract, eluting with chloroform–ethyl acetate (3:2), as a yellow powder; in its mass spectra, the molecular ion peak obtained at *m/z* 624 corresponds to the molecular formula $C_{29}H_{36}O_{15}$. This compound gave characteristic colors with Mg-HCl, Na-Hg/HCl, NaOMe, H_2SO_4, $NaBH_4$, 2,4-dinitrophenylhydrazine, and $FeCl_3$, and a positive Molisch and Tollen′s reagent test [7, 8]. In the UV spectrum a major absorption band appeared at 210, 281, and 325 (sh) nm, indicating that the compound was a flavanone glycoside [7–9]. The IR spectrum of this compound exhibited absorption bands at 1652 and 3448 cm^{-1}, which were indicative of the presence of a carbonyl group and hydroxyl groups.

In the ^{1}H NMR studies, the most characteristic peaks at δ 2.80 (dd, J = 2.8 and 17.2 Hz), 3.12 (dd, J = 13.2 and 17.2 Hz), and 5.38 (dd, J = 2.8 and 13.2 Hz), which were assigned to two H-3 protons and one H-2 proton, respectively, confirmed the presence of an ABX type signal for the flavanone moiety [7–11]. The downfield proton at δ 12.01 was assigned to C_5-OH chelated to C-4 carbonyl [7–11]. In the ^{1}H NMR of the flavonoids, the signals resonating at δ 7.5–7.6 and δ 6.5–6.9 were assigned to H-6 and H-8, respectively [7–11]. In the ^{1}H NMR, the signal of an isolated aromatic proton appeared at δ 6.53 (1H, s). Thus, the A-ring aromatic proton signal at δ 6.53 (1H, s) was assigned to H-8.

The singlet signal of six protons at δ 3.80 indicated the presence of two methoxyl groups substituted on an aromatic ring. The three ABC type aromatic proton signals at δ 7.03 (1H, dd, J = 1.8, 8.5 Hz), 7.01 (1H, d, J = 1.8 Hz), and 6.98 (1H, d, J = 8.5 Hz) were characteristic of H-6′, H-2′, and H-5′, respectively, indicating that the B-ring is 3′,4′-methoxylated [7–11].



1

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In the ^{13}C NMR spectrum, the signals at δ 79.2 and 45.3 were assigned to C-2 and C-3 of the dihydropyrone moiety, whereas the signals of two methoxy carbons observed at lower field, δ 58.6 and 58.3, suggested that these two methoxy groups have substituents at both *ortho* positions [11, 12]. The NMR further indicated the presence of two sugar moieties. In the NMR spectra, anomeric proton signals appeared at δ_H 4.56 (d, J = 9.5 Hz)/ δ_C 75.09 (H-1″) and δ_H 4.52 (br.s)/ δ_C 101.52 (H-1‴). A signal appeared for the methyl protons of α-rhamnose at δ_H 1.06 (d, J = 6.0 Hz) [10–12]. The HMBC correlation of H-1″ to C-5, C-6, and C-7 clearly indicated the presence of a 6-*C*-β-glucopyranoside. The downfield shift of C-6″ of the glucose unit (δ_C 68.34) and the cross peak of H-1‴ to C-6″ was assigned to the (1→6)-*C*-glucosidic bond. On the basis of the above evidence, the compound was identified as 5,7-dihydroxy-3′,4′-dimethoxyflavanone 6-*C*-[α-rhamnopyranosyl-(1→6)]-β-glucopyranoside (**1**).

EXPERIMENTAL

General Procedures. Ultraviolet absorption spectrum was recorded on a Perkin–Elmer Lambda Bio 20 UV spectrometer. IR spectroscopy was performed using the KBr disc method on a Perkin–Elmer 1710 infrared Fourier transform spectrometer. NMR spectra were recorded on Bruker AVANCE DRX-300 (300, 100 MHz). Chemical shifts are shown as δ values (ppm) with tetramethylsilane (TMS) as internal reference. FAB-MS was recorded on a JEOLSX 1021/DA-6000 mass spectrometer. Column chromatography was carried out using silica gel (60–120 mesh). Chemicals of analytical-reagent grade were purchased from E-Merck (India).

Plant Material. The roots of *Clausena pentaphylla* were collected from the rural areas of Shahjahanpur District in September. The plant was authenticated by comparison with data of the herbarium specimen deposited in the Herbarium of the Faculty of Botany, G. F. College (Rohilkhand University), Shahjahanpur (Herbarium No. GFCB 1361).

Extraction and Isolation. Dried and pulverized bark and leaves (1.5 kg) of the plant were first defatted with hexane (3 L × 5 times) and then Soxhlet-extracted with chloroform, ethyl acetate, and methanol (3 L × 5 times each). The ethyl acetate extract was then evaporated under vacuum on a rotary evaporator below 50°C to yield a brownish mass (35 g). The mass was then subjected to column chromatography. A well-stirred suspension of silica gel (100–150 g in petroleum ether at 60–80°) was poured into the column (150 cm long and 50 mm in diameter). When the absorbent was well settled, the excess of petroleum ether was allowed to pass through the column. The column was eluted with petroleum ether, chloroform, ethyl acetate, and methanol, and their mixtures with increasing polarity. Elution with chloroform–ethyl acetate (3:2) afforded the compound.

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