Fig. 2: UV Spectrum (MeOH) of TC-1
Fig. 3: IR Spectrum (KBr) of TC-1
Fig. 4: $^1$H NMR Spectrum (400 MHz, Me$_2$CO-$d_6$) of TC-1
shift of 20 nm in band II absorption maximum with aluminium chloride and aluminium chloride-hydrochloric acid, suggested the presence of a chelated hydroxyl at C-5. The IR spectrum (Fig. 3) showed two strong absorption bands attributable to hydroxyl (3444 cm\(^{-1}\)) and conjugated carbonyl group (1637 cm\(^{-1}\)), respectively.

The \(^1\)H NMR spectrum (Fig. 4) of TC-1 showed a D\(_2\)O exchangeable downfield signal at \(\delta\) 12.16 (1H, s) assignable to a C-5 chelated hydroxyl group. The presence of three sets of doublet of doublets at \(\delta\) 5.42 (1H, \(J = 13.0, 3.0\) Hz), 3.05 (1H, \(J = 17.2, 13.0\) Hz) and 2.86 (1H, \(J = 17.2, 3.0\) Hz) were typical of H-2, H-3\(_a\) and H-3\(_{eq}\), respectively of a flavanone moiety. The signals at \(\delta\) 5.16 (1H, t, \(J = 8.0\) Hz), 3.23 (2H, d, \(J = 8.0\) Hz) and 1.64 (6H, s), were assigned to methine, methylene and \textit{gem}-dimethyl protons, respectively, revealed a C-prenyl residue. A three-proton singlet at \(\delta\) 3.88 was attributed to a methoxyl group and was placed at C-7, as TC-1 didn’t show any bathochromic shift with sodium acetate in its UV spectrum. An aromatic proton singlet at \(\delta\) 6.10 was placed at C-6 on the basis of a negative Gibb’s test, which fixes the attachment of the C-prenyl residue at C-8. It also displayed a multiplet at \(\delta\) 7.42 (5H) indicating the unsubstituted nature of the ring B. The appearance of a significant \textit{retro} Diels-Alder fragment at \(m/z\) 234 in its El mass spectrum further supported the presence of a hydroxyl, a methoxyl and a prenyl group in ring A.

Thus, the structure of TC-1 was elucidated as 5-hydroxy-7-methoxy-8-C-prenylflavanone (or 7-O-methylglabranin) (48) as its physical and spectral data were in good agreement with the literature values.

![Chemical Structure](image)
Examination of Acetone Extract

The acetone extract was concentrated under reduced pressure to give a brownish gummy mass (120 g). It was purified over a silica gel column using n-hexane-ethyl acetate step gradient mixtures. The n-hexane-ethyl acetate 6:4 eluate on evaporation afforded a white solid and it was designated as TC-2.

TC-2

(Tephrowatsin C)

TC-2 was obtained as colorless needles (28 mg) from chloroform, mp 72-74°C. \([\alpha]_D^{21} = 51^\circ (c 0.16, \text{MeOH})\). It gave a green colour with alcoholic ferric chloride and an orange red colour with both magnesium-hydrochloric acid, and sodium borohydride-hydrochloric acid. It was analyzed for C\(_{21}\)H\(_{24}\)O\(_5\) consistent with the presence of [M+H]\(^+\) ion at \(m/z\) 357 in its FAB mass spectrum (Fig. 5). This was corroborated by \(^1\)C NMR spectrum (Fig. 6) which showed signals for all the twenty one carbons present in the molecule. The UV (Fig. 7) absorption maxima at 289 and 340 (sh) nm suggested TC-2 to be a flavanone derivative\(^8\). Addition of sodium acetate didn’t cause any change in band II absorption maximum indicating the absence of a free hydroxyl at C-7. A bathochromic shift of 22 nm in band II absorption maximum with aluminium chloride and aluminium chloride-hydrochloric acid suggested the presence of a chelated hydroxyl at C-5\(^8\). The IR spectrum (Fig. 8) showed two strong absorption bands at 3437 and 1636 cm\(^{-1}\) due to hydroxyl and carbonyl functions, respectively.

The \(^1\)H NMR spectrum (Fig. 9) of TC-2 showed a D\(_2\)O exchangeable downfield signal at \(\delta\) 12.08 indicating the presence of a chelated hydroxyl at C-5. The presence of three characteristic signals for H-2, H-3\(_{ax}\) and H-3\(_{eq}\) at \(\delta\) 5.39 (1H, dd, \(J = 12.6, 3.1\) Hz), 5.02 (1H, dd, \(J = 17.1, 12.6\) Hz) and 2.85 (1H, dd, \(J = 17.1, 3.1\) Hz), respectively indicated that TC-2 had a flavanone skeleton\(^8\). A sharp one-proton singlet at \(\delta\) 6.10 correlating with the carbon at 92.5 ppm in the HSQC spectrum (Fig. 10) was assigned to H-6 as it showed \(^2\)J correlations with C-5 (162.5 ppm) and C-7 (165.6 ppm), and \(^3\)J correlations with C-8 (109.2 ppm) and C-10 (102.9 ppm) in its HMBC spectrum (Fig. 11). The presence of a multiplet at \(\delta\) 7.42 integrating for five protons indicated the unsubstituted nature of ring B. A three-proton singlet