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A NEW ISOFLAVONE FROM *IRIS GERMANICA*

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Key Word Index—*Iris germanica*; Iridaceae; isoflavone; irilone.

Recently the presence of irisolone,[1] a known isoflavone has been reported from *Iris germanica* Linn. In this communication we report the isolation and structure elucidation of a new isoflavone, irilone.

Chloroform extract of defatted rhizomes (white flowered variety) gave a yellow crystalline substance which on re-crystallization with EtOAc–light petrol. gave yellow crystals of irilone, m.p. 231°. It gave a green colour with $FeCl_3$ and responded to the Labat test for methylenedioxy group. Elemental analysis and MS (M^+ 298) of irilone established the molecular formula as $C_{16}H_{10}O_6$. The UV spectrum exhibits λ_{max} at 269 and 332 (inflection) nm shifting to 282 nm on the addition of alluminium chloride solution, indicating an isoflavone with a 5-OH group. The IR spectrum exhibits the principal peaks at 1680 (>C=O), 3440, 3460 (two–OH groups) and 926 cm^{-1} (methylenedioxy group).

Due to the difficulty in obtaining substantial quantities of the compound in pure state by the above procedure the dried crude MeOH extract was acetylated and the acetate mixture subjected to column chromatography over silica gel. A crystalline compound, m.p. 213°, M^+ 382, giving no colouration with $FeCl_3$ solution was obtained, identical with the diacetate prepared from irilone in m.p., m.m.p. and TLC. The IR spectrum of the diacetate shows the principal peaks at 1654 (>C=O), 928 (methylenedioxy group), 1748 and 1782 cm^{-1} (two–OAc groups). The NMR (100 MHz, $CDCl_3$) of the diacetate gave bands at (δ): 7·87*s* due to 2-H; 6·84*s*, 8-H; 6·15*s*, $-O-CH_2-O-$; 7·55d (J_{AB}=9 Hz), $H_{2',6'}$; 7·17d (J_{AB} = 9 Hz), $H_{3',5'}$; two acetate proton signals at 2·41*s* (5'–OAc) and 2·29*s* (4'–OAc).

On methylation, irilone gave a compound $C_{18}H_{14}O_6$, M^+ 326, m.p. 184° identified as irisolone methyl ether[2,3] by its direct comparison with the authentic sample.

Irisolone could be converted into irilone by selective demethylation by anhydrous $AlCl_3$. The above evidence shows that the new isoflavone has structure 5,4'-dihydroxy-6,7-methylenedioxyisoflavone.

EXPERIMENTAL

Isolation. $CHCl_3$ extract of air dried, coarsely powdered and defatted rhizomes (white flowered variety) on subjecting to repeated column chromatography over silica gel using EtOAc–light petrol. (1:1) as eluent gave a yellow crystalline substance from one of the fractions. The solid on re-crystallization with EtOAc–light petrol. gave pale yellow crystals of irilone, m.p. 231° (Calc. for $C_{16}H_{10}O_6$: C, 64·42; H, 3·35. Found: C, 64·1; H, 3·41%). Diacetate (Ac_2O–pyridine) colourless crystals m.p. 213°, M^+ 382. Dimethyl ether (Me_2SO_4–K_2CO_3), from benzene–light petrol. as colourless shining crystals, m.p. 184°, M^+ 326.

Acetylation of crude product. The dried crude MeOH extract of defatted rhizomes was acetylated (Ac_2O–pyridine) and the acetate mixture was separated on a silica gel column. Elution with benzene gave irilone diacetate, m.p. 213°, M^+382 (Calc. for $C_{20}H_{14}O_8$: C, 62·82; H, 3·66. Found: C, 62·3; H, 3·29%).

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Partial synthesis of irilone. Irisolone was refluxed with anh. $AlCl_3$ in Et_2O. The product was poured in ice–H_2O and the brownish yellow solid obtained was refluxed conc. HCl–HOAc (1:1). On dilution with H_2O and extraction with Et_2O irilone, identical with the natural product, was obtained in m.p., m.m.p., TLC and identical diacetates.

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