

any explanation to this anomalous behaviour of the EL cell. A detailed analysis will be published elsewhere.

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#### AN ALTERNATIVE SYNTHESIS OF 7-O-METHYL GLABRANINE

GLABRANINE m.p. 154–55° was isolated from *Glycyrrhiza glabra*<sup>1</sup> and has been assigned the structure 8-C-prenyl-5, 7-dihydroxy flavanone from its spectral studies and synthesis<sup>2</sup>. Synthesis of 5-hydroxy-7-methoxy flavanone was accomplished *via* isomerisation of 2'-hydroxy-4'-methoxy-6'-benzyloxy chalcone and its nuclear prenylation with 2-methyl-but 3-en-2-ol and boron trifluoride etherate in dry dioxan medium gave 7-O-methyl glabranine.

Phloroacetophenone was converted into its ditosyl ester and the remaining hydroxyl was benzylated using benzyl chloride. The resulting product was saponified and the monobenzylphloroacetophenone was obtained as a buff coloured solid m.p. 233–35° (Found: C, 69.6; H, 5.20, C<sub>15</sub>H<sub>14</sub>O<sub>4</sub> requires C, 69.77; H, 5.42%). It was then subjected to methylation in the 4-position to yield 4-O-methyl-6-O-benzylphloroacetophenone m.p. 110–13° (Found: C, 70.30; H, 5.62 C<sub>16</sub>H<sub>16</sub>O<sub>4</sub> requires C, 70.58; H, 5.58%). On condensation with benzaldehyde in alcoholic potassium hydroxide, it gave the 2'-hydroxy-4'-methoxy-6'-benzyloxychalcone m.p. 126–27°;  $\nu_{\text{max}}^{\text{KBr}}$  1618, 1582, 1563, 1389, 1337, 1282, 1212, 1105, 1031, 816, 746 and 690 cm<sup>-1</sup>;  $\lambda_{\text{max}}^{\text{MeOH}}$  337; + AlCl<sub>3</sub> 367 nm. NMR ( $\delta$ , CDCl<sub>3</sub>): 3.67 (s, 3H, -OCH<sub>3</sub>), 4.88 (s, 2H, -OCH<sub>2</sub>-C<sub>6</sub>H<sub>5</sub>), 5.92, 6.0 (d, J = 2 Cps, 3', 5'-protons), 7.13 (m, 1OH, -OCH<sub>2</sub>-C<sub>6</sub>H<sub>5</sub>) and ring A protons), 7.50 (d, J = 15 Cps, 1H), 7.80 (d, J = 15 Cps, 1H)  $\alpha$  and  $\beta$  Protons, 14.70 (s, 1H,

-OH) (Found: C, 77.0; H, 5.9; C<sub>23</sub>H<sub>20</sub>O<sub>4</sub> requires C, 76.7, H, 5.6%). Its isomerisation to 5-hydroxy-7-methoxyflavanone was carried out by refluxing with 4% H<sub>2</sub>SO<sub>4</sub>. The flavanone was crystallised from the mixture by petroleum ether as colourless needles m.p. 104–5°;  $\nu_{\text{max}}^{\text{KBr}}$  2941, 1626 (b), 1575, 1379, 1351, 1299, 1259, 1208, 1093, 995, 962, 891, 794, 743, 698 and 683 cm<sup>-1</sup>. NMR ( $\delta$ , CDCl<sub>3</sub>), 2.80 (m, 2H, C<sub>3</sub>-H<sub>2</sub>), 3.70 (s, 3H, -OCH<sub>3</sub>), 5.20 (m, 1H, C<sub>2</sub>-H), 5.90 (s, 2H, 6, 8-H), 7.31 (s, 5H, side phenyl protons), 11.90 (s, 1H, -OH). (Found: C, 70.95; H, 5.08; C<sub>16</sub>H<sub>14</sub>O<sub>4</sub> requires C, 71.11; H, 5.18%). Nuclear prenylation of 5-hydroxy-7-methoxy flavanone with 2-methyl-but-3-en-2-ol in the presence of boron trifluoride-etherate in dry dioxan medium gave a mixture of two compounds which were separated by column chromatography over silica gel and designated as A and B. Compound A crystallised from petroleum ether as colourless needles m.p. 108–110°,  $\nu_{\text{max}}^{\text{KBr}}$  3378, 1640, 1626 cm<sup>-1</sup>;  $\lambda_{\text{max}}^{\text{MeOH}}$  288, 322; + AlCl<sub>3</sub> 310, 370; + AlCl<sub>3</sub> + HCl 308, 370 nm; NMR ( $\delta$ , CDCl<sub>3</sub>): 1.60 [s, 6H, (CH<sub>3</sub>)<sub>2</sub>C = C<], 2.75–2.95 (m, 2H, C<sub>3</sub>-H<sub>2</sub>), 3.15 (d, J = 7 Cps, -CH<sub>2</sub>-Ar), 3.80 (s, 3H, -OCH<sub>3</sub>), 5.20 (m, 1H, C<sub>2</sub>-H), 5.40 (m, 1H, vinylic proton), 5.90 (s, 1H, 6-H), 7.25 (s, 5H; side phenyl protons), 12.00 (s, 1H, -OH). That the prenyl unit had entered into one of the two available nuclear positions 6 or 8 in the ring A was shown by its NMR spectrum which had the expected resonance signals of one C-prenyl unit at  $\delta$  1.60, 3.15 and 5.40 and signals at 5.90 and 7.25 integrating for one and five protons respectively. No change in R<sub>f</sub> value and other properties was observed when compound A was treated with formic acid for ½ hr. Thus the C-prenyl unit is at 8-position. Hence the structure of compound A is 8-C-prenyl-5-hydroxy-7-methoxy flavanone, which resembled in all respects with that reported by Khanna *et al*<sup>3</sup> (Found: C, 74.40; H, 6.80; C<sub>21</sub>H<sub>22</sub>O<sub>4</sub> requires C, 74.5; H, 6.6%).

Compound B was found to be the starting material by direct comparison.

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