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RUTACEAE

ALKALOIDS OF THE STEM BARK OF *HESPERETHUSA CRENULATA*

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Abstract—From the petroleum extract of the title plant was isolated the weak base 4-methoxy-1-methyl-2-quinolone(I).

Hesperethusa crenulata (Roxb.) M. Rome is widely distributed in India. The different parts of the plant are reported to have medicinal properties and in Burma and Thailand the powdered bark of the wood mixed with sandalwood is used as a cosmetic.[1] Mohammad and Asif[2] isolated the coumarin luvangetin from the leaves.

We now report our preliminary findings on the alkaloid of the stem bark. The petroleum extract of the powdered bark on chromatography on neutral alumina, gave an alkaloidal fraction which on rechromatography and recrystallization from petrol–benzene yielded a colourless crystalline compound m.p. 100–100·5°. On exposure to air it slowly absorbed moisture and melted at 68–69°.

The base analysed for $C_{11}H_{11}NO_2 . H_2O$ (M^+ *m/e* 189). The IR spectrum in Nujol showed the presence of an OH group (3200 cm^{-1}), conjugated C=O (1640 cm^{-1}), aromatic bands (1598 cm^{-1}, 1500 cm^{-1}) and C—O—C asymmetric stretching (1250 cm^{-1}). The UV spectrum of the base in ethanol with λ_{max} 225 (log ϵ 4·46), 268 (log ϵ 3·95), 279 (log ϵ 3·970), 318 (log ϵ 3·951), 333 nm(sh) (log ϵ 3·862) and λ_{min} at 253 (log ϵ 3·759) 273 (log ϵ 3·889), 286 (log ϵ 3·595) and 329 nm (log ϵ 3·89), coupled with the fact that the spectrum was unaffected by the addition of acid suggested its nature as a 2-quinolone as distinct from a 4-quinolone.[3] The NMR spectrum* of the alkaloid in $CDCl_3$ using TMS as internal reference showed four hydrogens in the aromatic region. The signals centred at δ7·35 (2H) could be ascribed to H_6 and H_7 while H_5 and H_8 appeared as two quartets centred at δ8·0 and 7·55 (J = 2c/s (meta coupling) and J = 9 c/s (ortho coupling)). A signal at δ6·01 (1H singlet) has been assigned to H_3. The two three proton singlets at δ3·99 and 3·70 are due to —OCH_3 and —NCH_3 though an unequivocal assignment between the two is rather difficult. This accounts for all the hydrogens in the parent alkaloid and on the basis of the above spectral data the compound was identified as 4-methoxy-1-methyl-2-quinolone(I), a compound previously synthesized by Fritz *et al.*[4] who had reported a m.p. of 102° and 68° respectively for the anhydrous and hydrated compound.

In addition to the NMR signals already mentioned there is a one proton signal at δ2·2 (singlet, removed by D_2O exchange) which needs an explanation. This signal is explained

* 60 MHZ spectrum.

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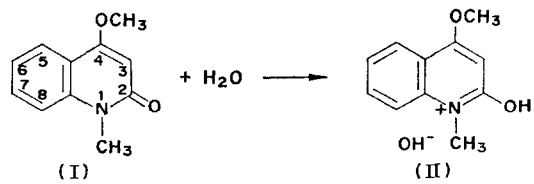

(I) (II)

on the basis that the alkaloid in solution reacts with the water of hydration to exist as a 2-hydroxyquinoline(II), rather than as the 2-quinolone(I). The signal at $\delta 2{\cdot}2$ is assigned to the hydroxyl group at position 2 of the hydroxyquinoline. The hydroxyl ion was not located in the NMR spectrum.

More detailed investigations are being undertaken to study the structural changes of the molecule in solution as well as in the solid state. To the best of our knowledge the compound does not appear to have isolated from a natural source before and constitutes an example of previously synthesized compound being isolated from a natural source.

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SALICACEAE

PHENOLIC EXTRACTIVES OF THE LEAVES OF *POPULUS BALSAMIFERA* AND OF *P. TRICHOCARPA**

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Abstract—Fresh June leaves of *Populus balsamifera* and *P. trichocarpa* were extracted with ethanol, and the hot water-soluble portions were fractionated by ethyl acetate extraction and polyamide chromatography and step gradient elution with water and dilutions of ethanol. Although generally similar, several important differences were noted in the components of the leaves of the two related species.

IN AN earlier study on the leaves of *Populus balsamifera*[1] the similarity of the components isolated from the leaves of *P. balsamifera* and those obtained in the past from the bark of *P. trichocarpa*[2,3] and the bark of *P. balsamifera*[4] was noted, and it was suggested that the

* Part XVI in the series "Studies on the Leaves of the Family Salicaceae".

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