and the residue subjected to chromatography on a silica gel column (3 × 40 cm). Elution with chloroform-methanol (4:1) furnished a small quantity of ecdysterone in fractions 7–9 (each fraction 100 ml), as indicated by thin-layer chromatography, developing with chloroform-ethanol-acetone, 6:2:1. Spraying the plate with 50% sulfuric acid and heating revealed ecdysterone as a green spot, Rf 0.44. The fractions were pooled and concentrated, and the residue (127 mg) recrystallized from ethyl acetate-ethanol, 1:1, to give crystals of ecdysterone of mp 240–241°, characterized further by UV-, NMR- and mass-spectrum.

Résumé. L'ecdystérone, hormone de mue des insectes a été extraite de l'*Achyranthes aspera* et identifiée par des méthodes chromatographiques et spectroscopiques.

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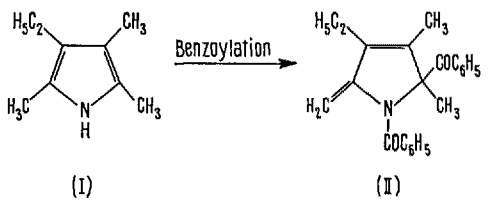
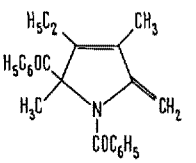
Benzoylation Studies in Pyrroles: Benzoylation of Phyllopyrrole

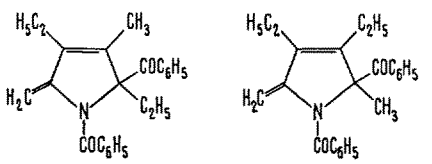
We have reported[1,2] the benzoylation of 2,3,4,5-tetramethyl pyrrole. The present report deals with the benzoylation of an unsymmetrically substituted pyrrole viz 2,3,5-trimethyl-4-ethylpyrrole (I) (Phyllopyrrole)



(I) (II)



(III)



(IV) (V)

which can be expected to give 2 different products (II) and (III).

However benzoylation of phyllopyrrole gave only one product, mp 156–157°, colourless needles, 80% yield, analyzed for $C_{23}H_{23}NO_2$ (Found C, 79.49; H, 8.10; N, 5.57. Calcd. C, 79.63; H, 7.94; N, 5.80). The IR-spectrum did not exhibit O–H or N–H absorption but showed carbonyl absorption bands at 1690 and 1645 cm^{-1}. Structure (II) was assigned to the product on the bases of its nuclear magnetic resonance spectrum and the comparison of its chemical shift with the benzoylated product of 2,4-dimethyl-3,5-diethyl pyrrole[3] (IV) and 2,5-dimethyl-3,4-diethyl pyrrole[4] (V). The nuclear magnetic resonance (NMR) spectral data of the benzoylation product of (II), (IV) and (V) are presented in the Table. The NMR-spectrum of the product was taken in $CDCl_3$ (TMS as internal standard) and has been recorded in varian A-60 machine.

The ethyl groups in position 4 of (II), (IV) and (V) have a common triplet around 1.11–1.15 ppm and quartet at 2.29 ppm. Two methylene protons in these compounds show doublets around 3.41 and 4.29 ppm. The methyl group in position 3 of compound (II) shows a singlet at 1.79 which is very similar to the singlet at 1.78 ppm for the methyl in position 3 of (IV). The methyl group at position 2 in compound (II) and (V) both show a singlet at 1.84 ppm. It is worth mentioning that the benzoyl groups in all these compounds show multiplet in the low field around 6.9–7.8 ppm. The probable explanation of

Chemical shifts of different group protons[a] in ppm in $CDCl_3$

Compound	2	3	4	5
II	1.84 (s)	1.79 (s)	1.11 (t) 2.29 (q)	3.41 (d) 4.29 (d)
IV	0.75 (t) 2.63 (q)	1.78 (s)	1.12 (t) 2.36 (q)	3.4 (d) 4.28 (d)
V	1.84 (s)	1.0 (t) 2.28 (q)	1.15 (t) 2.32 (q)	3.42 (d) 4.32 (d)

[a] d, doublet; q, quartet; t, triplet and s, singlet.

the preferential benzoylation at position 2 in preference to position 5 may be due to the steric effect of the ethyl group at position 4 of the phyllopyrrole[5].

Zusammenfassung. Vergleichende NMR-Untersuchungen zeigten, dass die Benzoylierung von Phyllopyrrole die Verbindung II und nicht I liefert.

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Organisch-Chemisches Institut der Technischen Hochschule, München (Germany), 16 November 1970.

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