

Table 2

Spectroscopic Data of 3*H*-Phenoxazin-3-one Derivatives

Compound No.	Mass	Infrared Spectrum cm ⁻¹	¹ H NMR Spectrum (deuteriochloroform) ppm
3a	211	1650 (C=O); 1618; 1584; 1518; 1464; 1164; 906; 757	2.13 (s, 3H, CH ₃), 6.23 (m, 1H, aromatic), 7.01-7.46 (m, 4H, aromatic), 7.58-7.83 (m, 1H, aromatic)
3b	225	1650 (C=O); 1610; 1582; 1514; 1458; 1160; 878; 745	1.28 (t, 3H, CH ₃), 2.54 (q, 2H, CH ₂), 6.22 (s, 1H, aromatic), 6.90-7.47 (m, 4H, aromatic), 7.53-7.83 (m, 1H, aromatic)
4a	211	1648 (C=O); 1618; 1577; 1516; 1462; 1176; 837; 752	2.08 (s, 1H, CH ₃), 6.64-7.51 (m, 5H, aromatic), 7.56-7.73 (m, 1H, aromatic),
5a	225	1651 (C=O); 1614; 1586; 1509; 1459; 1187; 874; 748	2.06 (s, 3H, CH ₃), 2.17 (s, 3H, CH ₃), 6.98-7.41 (m, 4H, aromatic), 7.57-7.76 (m, 1H, aromatic)
5b	253	1646 (C=O); 1608; 1584; 1512; 1458; 1178; 878; 754	1.13 (t, 6H, CH ₃), 2.61 (q, 4H, CH ₂), 6.93-7.42 (m, 4H, aromatic), 7.48-7.83 (m, 1H, aromatic)
5c	239	1649 (C=O); 1618; 1586; 1514; 1457; 1187; 897; 757	1.11 (t, 3H, CH ₃), 2.16 (s, 1H, CH ₃), 2.63 (q, 2H, CH ₂), 7.03-7.43 (m, 4H, aromatic), 7.48-7.84 (m, 1H, aromatic)

The structures of the compounds **3-5** were determined by elemental analysis and from spectroscopic data. In particular, the nmr spectra (deuteriochloroform) of **1** exhibited a characteristic singlet at 6.30 ppm due to the olefinic proton but those of **4** and **5** did not show any evidence that could be assigned to this type of proton. Therefore, the addition of alkyl radicals did occur at the 4-position of **1**. On the other hand, the nmr spectrum of **3** exhibited a characteristic singlet at 6.30 ppm and compound **3a** thus obtained was identical in every respect with a sample prepared by the previously reported method (12). Consequently, compounds **3** were identified as 2-alkyl-3*H*-phenoxazin-3-ones. Furthermore, compound **5a** obtained by the reaction of **1** with **2a** was identical in every respect with a sample prepared by the reaction of **3a** with **2a**.

The presently available method for the preparation of 2-alkyl- and 2,4-dialkyl-3*H*-phenoxazin-3-ones has the disadvantage of the very poor overall yield of the multi-step procedures (12,13). Therefore, the present note provides a new synthetic route to iminoquinone derivatives.

EXPERIMENTAL

Melting points were determined on a Yanagimoto micromelting apparatus and are uncorrected. The infrared spectra were recorded on a Jasco DS 701G spectrometer. Absorption frequencies are reported in reciprocal centimeters. Nuclear magnetic resonance spectra were determined on a Hitachi R-20B spectrometer using tetramethylsilane as an internal reference. Chemical shifts are reported in parts per million (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). Mass spectra were

determined on a Hitachi M-52 spectrometer.

General Procedure for the Alkylation of 3*H*-Phenoxazin-3-one (**1**) and 2-Methyl-3*H*-phenoxazin-3-one (**3a**).

To a vigorously stirred suspension of 3*H*-phenoxazin-3-one (**1**) (0.2 g, 1 mmole) or 2-methyl-3*H*-phenoxazin-3-one (**3a**), the carboxylic acid (**2**) (10 mmoles) and silver nitrate (0.17 g, 1 mmole) in acetonitrile (50 ml) and water (20 ml) was added a solution of ammonium peroxydisulfate (0.45 g, 2 mmoles) in water (25 ml) during a period of 45 minutes at 70-75°. The mixture was stirred and heated for an additional 15 minutes. After removal of the organic solvent under reduced pressure, the precipitate was collected, washed well with water and chromatographed over an aluminium oxide column and eluted with benzene. The solid obtained on concentration of the eluate was recrystallized from benzene.

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