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SYNTHESIS OF 3-ACETOXY-1-METHYL AND 1-ETHYLINDOLES

Roy W. Daisley^a; Zaha A. Elagbar^a

^a Department of Pharmacy, Brighton Polytechnic, Sussex, England

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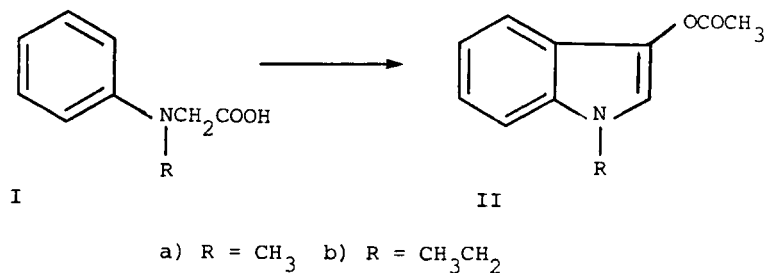
2. For example, chromatography on silica gel using a mixture of hexane and benzene (1:1) as eluent, gave diphenyldisulfide and 2,4,6-tri-(*t*-butyl)phenol in 63% and 69% yields respectively.
3. O. Hinsberg, Ber., 36, 107 (1903).

SYNTHESIS OF 3-ACETOXY-1-METHYL AND 1-ETHYLINDOLES

Submitted by Roy W. Daisley* and Zaha A. Elagbar
(12/14/83)

Department of Pharmacy, Brighton Polytechnic
Lewes Road
Brighton, Sussex, BN2 4GJ, ENGLAND

Indol-3(2H)-ones (indoxyls) (II) may be synthesized by the cyclization of the appropriate phenylglycines (I) or the appropriate 2-carboxyphenylglycine. Cyclizing agents for the former include sodium or potassium hydroxide¹ at 260° or soda-mide with cyanide moderators;² however, the indoxyl once formed has a great tendency to oxidize to indigo unless air is rigorously excluded from the reaction and work-up procedures and yields tend to be low. Although cyclization of 2-carboxyphenylglycines proceeds smoothly using acetic anhydride and sodium acetate at reflux temperatures,^{3,4} the corresponding N-substituted 2-carboxyphenylglycines are not readily available and



require extra synthetic stages for their preparation. The titled compounds were required for further studies and we have developed the present procedure for their easy production.

EXPERIMENTAL SECTION

Mps are uncorrected. IR and PMR spectra (TMS as internal standard) were recorded using Perkin-Elmer Model 157G and R32 spectrometers.

General Procedure.- The appropriate N-substituted-N-phenylglycine (I) as its hydrochloride⁵ (0.033 mol), anhydrous sodium acetate (5.5 g, 0.067 mol), acetic anhydride (10 ml, 0.10 mol) and dimethylformamide (30 ml) was boiled under reflux for 3 hrs and then evaporated to dryness. The residue was treated with water (40 ml) and extracted with dichloromethane (2 x 40 ml). The extract was washed with sodium bicarbonate solution (2 x 10 ml) and water (2 x 10 ml). Evaporation of the dried extract yielded the appropriate crude product. Recrystallization from aqueous ethanol yielded IIa; IIb was purified by vacuum distillation. IIa, 89% yield, mp. 56.5^o, lit.⁴ mp. 56.5^o; IR(KBr): 1740 (CO), 1615 (C=C) cm⁻¹; PMR (DMSO-d₆): δ 2.32 (s, 3H, OCOCH₃), 3.72 (s, 3H, N-CH₃), 7.38 (s, 1H, CH), 6.99-7.50 (m, 4H, arom. H). IIb, 63% yield, pale green semi-solid, bp. 120^o/3mm Hg; IR(KBr): 1735 (CO), 1610 (C=C) cm⁻¹; PMR (DMSO-d₆): δ 1.13 (t, 3H, J = 7.5 Hz, CH₂CH₃), 2.33 (s, 3H, OCOCH₃), 4.08 (q, 2H, J = 7.5 Hz, CH₂CH₃), 7.34 (s, 1H, CH), 6.97-7.50 (m, 4H, arom. H).

Anal. Calcd for C₁₂H₁₃NO₂: C, 70.91; H, 6.45; N, 6.89

Found: C, 70.86; H, 6.41; N, 6.82.

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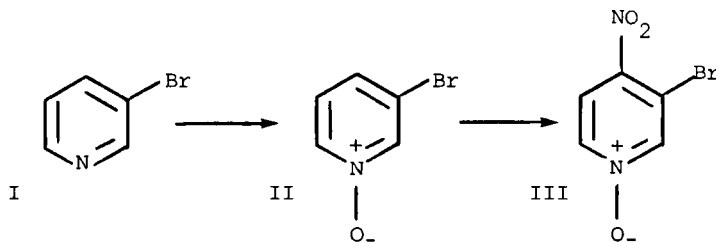
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PREPARATION OF 3-BROMO-4-NITROPYRIDINE 1-OXIDE

Submitted by Roy W. Daisley* and Jafar R. Hanbali
(1/12/83)

Department of Pharmacy, Brighton Polytechnic
Lewes Road, Brighton, Sussex BN2 4GJ, ENGLAND

The preparation of 3-bromo-4-nitropyridine 1-oxide (III) from 3-bromopyridine (I) has been described by several workers.



In the initial synthesis,¹ the intermediate 3-bromopyridine 1-