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A One Pot Synthesis of 5,7-Diphenyl-2,3-dihydro-1H-pyrrolizine

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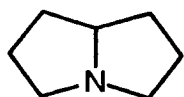
A ONE POT SYNTHESIS OF 5,7-DIPHENYL-2,3-DIHYDRO-1H-PYRROLIZINE

R.W. Soeder*, K. Bowers, L.D. Pegram and C.P. Cartaya-Marin*

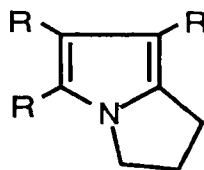
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Abstract: 5,7-Diphenyl-2,3-dihydro-1H-pyrrolizidine was synthesized from dibenzoylmethane and pyrrolidine in one step in 60% yield.

The pyrrolizine skeleton, an azabicyclo[3.3.0]octane ring system (1), is an essential part of the biologically active pyrrolizidine alkaloids¹, mitomycin antibiotics² and ant venom alkaloids³. The biological properties of these alkaloids include antitumor, hypotensive, anti-inflammatory, carcinogenic and hepatotoxic activity⁴. 2,3-Dihydro-1H-pyrrolizine derivatives (2) are potent anti-neoplastic and anti-inflammatory agents^{5,6}.



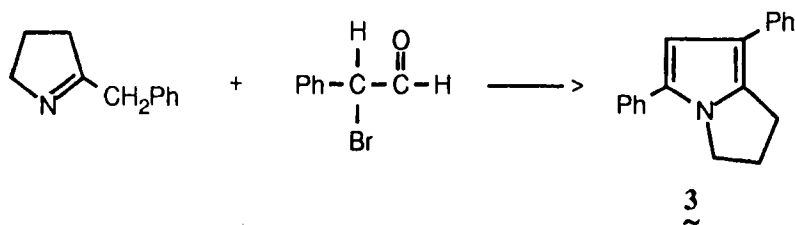
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* To whom correspondence should be addressed

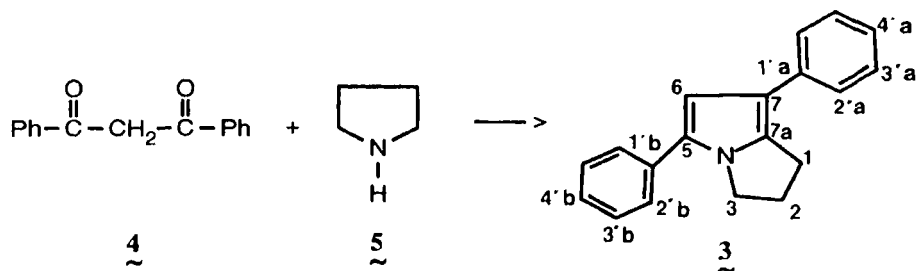
Equation 1



The synthesis of 5,7-diphenyl-2,3-dihydro-1H-pyrrolizine (**3**) has been reported by Dannhardt⁷ in 15% overall yield by the reaction of 2-benzyl-Δ¹-pyrrolidine with α-bromophenylacetaldehyde (equation 1) in ethanol for two days at room temperature.

A one pot synthesis of 5,7-diphenyl-2,3-dihydro-1H-pyrrolizine (**3**) from readily available starting materials has been developed in this laboratory. The reaction of 1,3-diphenyl-1,3-propanedione (dibenzoylmethane) (**4**), 1.7 equivalents of pyrrolidine (**5**) and molecular sieves in a limited amount of benzene for 1 hour at high temperature using a Dean-Stark trap for the removal of water produced compound **3** in 60% yield after recrystallization (equation 2). Reaction of dibenzoylmethane (**4**), and pyrrolidine (**5**) with benzene as solvent, under milder conditions, such as lower temperatures, higher dilution or ethanol as solvent gave high yields of the corresponding enaminone. The products from these reactions were characterized ¹H NMR, ¹³C NMR and gas chromatography/mass spectrometry (GC/MS).⁸

Equation 2



The scope and limits of this reaction is currently under investigation.

Acknowledgment: We would like to thank the Department of Chemistry of Appalachian State University for the generous financial support and Professors Huw Davies and Mark Welker of Wake Forest University for the use of their 200 MHz NMR spectrometer.

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8. Physical data: MP=149-150°(uncor.) from ethanol.
¹H NMR(Varian XL-200) δ =2.51-2.68(2H,m), 3.15(2H,t, J=7.3Hz), 4.18(2H,t,J=7.0Hz), 6.78(1H,s), 7.14-7.33(2H,m) 7.35-7.47(4H,m), 7.51-7.63(4H,m).
¹³C NMR(decoupled)(Varian XL-200) with assignment,[]=rel. intensity δ =25.22[4.0] C₂, 27.79[4.1] C₁, 46.53[4.2] C₃, 108.52[4.7] C₆, 116.01[1.1] C₇, 124.56[5.1] & 125.82[5.2] C_{4'a} & C_{4'b}, 125.05[9.8], 125.64[9.6], 128.48[9.9] & 128.57[9.9] C_{2'a}, C_{3'a}, C_{2'b}, & C_{3'b}, 129.18[1.0] C_{7a}, 133.25[1.2] C₅, 135.84[1.2] & 136.25[1.2] C_{1'a} & C_{1'b}.
MS (Finnigan-MAT ITD) Molecular mass(C₁₉H₁₇N)= 259; 260(M+1)(12), 259(M)(100), 258(M-1)(51), 230(M-C₂H₂,H)(6), 191(M-C₄H₈N)(5), 182(M-C₆H₈)(19), 128(10),115(9), 102(6), 91(6), 77(22).

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