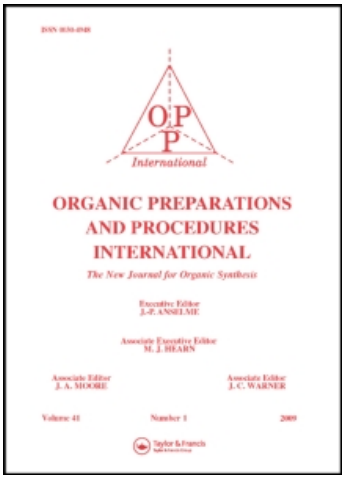

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A CONVENIENT PREPARATION OF 3-CARBETHOXY-2, 4-DIMETHYLQUINOLINE HYDROCHLORIDE

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A CONVENIENT PREPARATION OF 3-CARBETHOXY-2,4-DIMETHYLQUINOLINE HYDROCHLORIDE

Submitted by (02/11/87) L. A. M. Bastiaansen*, J. A. M. v. Schijndel and H. M. Buck

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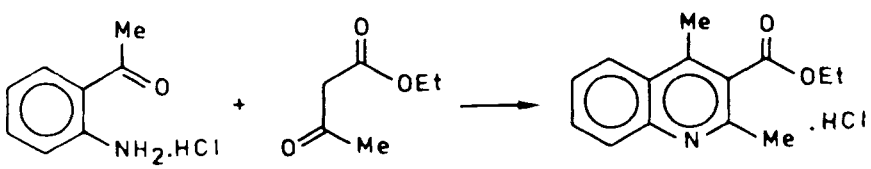
During the study of the stereochemistry of the hydride uptake of substituted 2,4-dimethyl-3-carbamoylpyridinium cations we observed both 1,6 and 1,4 reductions.[1] To exclude the former we focussed our attention on the corresponding quinolinium compounds, which have been introduced[2] and recently applied[3] as NAD^+ model systems. The present procedure for the title compound is an improvement of the well documented[4] Friedländer synthesis. The use of acidified isopropanol as a reaction medium in the Kempter modification effects a rapid conversion and a simple isolation of the product, directly in the form of its salt.



EXPERIMENTAL SECTION

3-Carbethoxy-2,4-dimethylquinoline Hydrochloride.- Finely powdered[5] o-aminoacetophenone hydrochloride (0.5 g, 2.9 mmoles) was suspended in a solution of i-PrOH (5 ml), i-PrOH/HCl[6] (0.5 ml) and ethyl acetoacetate (0.76 g, 5.8 mmoles). The mixture was rapidly heated to its boiling point, which resulted in a clear solution. After maintaining this temper-

ature for a few minutes a precipitate separated. Cooling, addition of dry ether (20 ml), filtration and washing with dry ether yielded 0.76 g (98%, mp. 190-192°) of 3-carbethoxy-2,4-dimethylquinoline hydrochloride.

^{1}H-NMR ($CDCl_3$): δ 1.47 (t,3H,J = 6Hz,$CH_2\underline{CH}_3$), 2.88(s,3H,4-CH_3), 3.12(s,3H,2-CH_3), 4.52(q,2H,J = 6Hz,$C\underline{H}_2CH_3$), 7.73-9.03(m,4H,Ar-$\underline{H}$)

An analytical sample was obtained from MeOH acidified with i-PrOH/HCl/ether.[6]

Anal. Calcd. for $C_{14}H_{15}NO_2HCl$: C, 63.27; H, 6.07; N, 5.27

Found: C, 63.30; H, 6.16; N, 5.27

Basification of an aqueous solution of this hydrochloride followed by extraction with CH_2Cl_2, drying and evaporation yielded 3-carbethoxy-2,4-dimethylquinoline as an oil. ^{1}H-NMR data were identical to those described in the literature.[3]

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5. In order to insure rapid dissolution.
6. A saturated solution of dry HCl in isopropanol. Omission of this acidic solution lowers the yield.