

This article was downloaded by:

On: 27 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

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

L. A. M. Bastlaansen<sup>a</sup>; J. A. M. v. Schijndel<sup>a</sup>; H. M. Buck<sup>a</sup>

<sup>a</sup> Department of Organic Chemistry, Eindhoven University of Technology, Eindhoven, THE NETHERLANDS

**To cite this Article** Bastlaansen, L. A. M. , Schijndel, J. A. M. v. and Buck, H. M.(1988) 'A CONVENIENT PREPARATION OF 3-CARBETHOXY-2, 4-DIMETHYLQUINOLINE HYDROCHLORIDE', *Organic Preparations and Procedures International*, 20: 1, 102 – 103

**To link to this Article:** DOI: 10.1080/00304948809355876

**URL:** <http://dx.doi.org/10.1080/00304948809355876>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

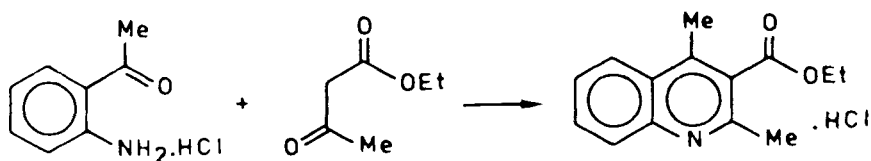
## A CONVENIENT PREPARATION OF

## 3-CARBETHOXY-2,4-DIMETHYLQUINOLINE HYDROCHLORIDE

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

Department of Organic Chemistry, Eindhoven University of  
Technology, Eindhoven, THE NETHERLANDS

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.<sup>1</sup> To exclude the former we focussed our attention on the corresponding quinolinium compounds, which have been introduced<sup>2</sup> and recently applied<sup>3</sup> as NAD<sup>+</sup> model systems. The present procedure for the title compound is an improvement of the well documented<sup>4</sup> 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<sup>5</sup> o-aminoacetophenone hydrochloride (0.5 g, 2.9 mmoles) was suspended in a solution of i-PrOH (5 ml), i-PrOH/HCl<sup>6</sup> (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.

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  1.47 (t, 3H,  $J = 6\text{Hz}$ ,  $\text{CH}_2\text{CH}_3$ ), 2.88 (s, 3H, 4- $\text{CH}_3$ ), 3.12 (s, 3H, 2- $\text{CH}_3$ ), 4.52 (q, 2H,  $J = 6\text{Hz}$ ,  $\text{CH}_2\text{CH}_3$ ), 7.73-9.03 (m, 4H, Ar-H)

An analytical sample was obtained from MeOH acidified with *i*-PrOH/HCl/ether.<sup>6</sup>

Anal. Calcd. for  $\text{C}_{14}\text{H}_{15}\text{NO}_2\text{HCl}$ : 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  $\text{CH}_2\text{Cl}_2$ , drying and evaporation yielded 3-carbethoxy-2,4-dimethylquinoline as an oil.  $^1\text{H-NMR}$  data were identical to those described in the literature.<sup>3</sup>

#### REFERENCES

1. P. M. T. de Kok, M. C. A. Donkersloot, P. M. van Lier, G. H. W. M. Meulendijks, L. A. M. Bastiaansen, H. J. G. van Hooff, J. A. Kanters and H. M. Buck, *Tetrahedron*, **42**, 941 (1986).
2. S. Shinkai, H. Hamada, Y. Kusano and O. Manabe, *J. Chem. Soc., Perkin II*, 699 (1979).
3. A. Ohno, M. Kashiwagi, Y. Ishihara, S. Ushida and S. Oka, *Tetrahedron*, **42**, 961 (1986).
4. C.-C. Cheng and S.-Y. Yan, *Org. React.*, **28**, 37 (1982).
5. In order to insure rapid dissolution.
6. A saturated solution of dry HCl in isopropanol. Omission of this acidic solution lowers the yield.