

## 7-Acetyl-4,5,5a,6,6a,7,8,9-octahydroindeno[7,1-fg]quinolin-9-one

Ralph E. Bowman

Welch School of Pharmacy, University of Wales, Institute of Science and Technology,  
Cardiff CF1 3NU, United Kingdom

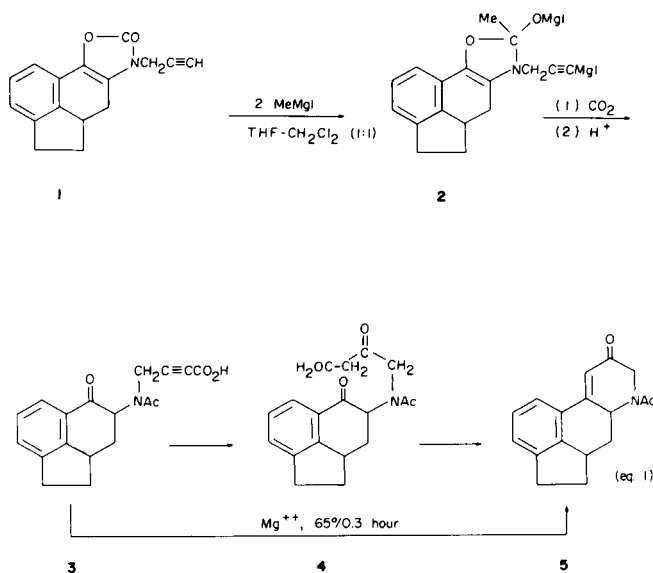
Received February 18, 1982

Treatment of the acetylenic acid **3** with magnesium carbonate in water at 60° yielded the tetracyclic ketone **5**.

*J. Heterocyclic Chem.*, **19**, 703 (1982).

N-Prop-2-ynyl-oxazolinones such as **1** offer a potential route to lysergic acid and related isosteres (equation 1).

On the first occasion, work-up of **3** was complicated by emulsion problems and the crude product (*ca.* 150% yield) containing inorganic contaminants, was treated with 0.5*M* sodium hydrogencarbonate to isolate the required acid.



To our surprise, the clear extract on warming to 60° became hazy and over 0.25 hour deposited the crystalline ketone **5**, (**2**) in 12% overall yield. Despite many attempts this totally unexpected 'one-pot' reaction could never be repeated. Improved conditions for the isolation of the acetylenic acid **3** furnished it as a yellow glass (**3**) which, unlike the first crude product, could not be converted into the ketone **5** with aqueous sodium hydrogencarbonate. Subsequently it was found that a similar treatment of **3** with magnesium carbonate (1 equivalent) did furnish **5** and that this treatment could be repeated at least twice on the recovered acetylenic acid to give the tetracyclic ketone **5** in overall yield from **1** of 29%, a not unsatisfactory result considering that seven steps could well have been involved in the overall process.

The small conversion, limited data and curious reaction conditions make difficult a rational explanation of these results. It would appear that magnesium ion was essential since many other metallic hydroxides were examined without success, and that possibly, under its influence a small conversion to the magnesium salt of the  $\beta$ -ketoacid **4** in its enolic form **6** took place with subsequent cyclisation and decarboxylation to give **5** (equation 2).

**Acknowledgement.**

This work was carried out during the tenure of an Allen and Hanbury Research Fellowship.

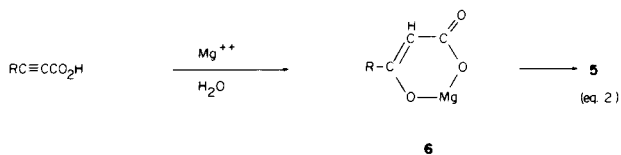
## REFERENCES AND NOTES

(1) R. E. Bowman, *J. Chem. Soc., Perkin Trans. I*, 2126 (1980).

(2) Colourless plates from ethanol, mp 184-185°; ir:  $\nu$  max 1662 (C=C-CO), 1640 (amide I), 1610 (C=C), 1587 cm<sup>-1</sup>; nmr (deuteriochloroform): 90 MHz,  $\delta$  1.4-3.5 (7H, m, 5a-H and 4,5,6-H<sub>2</sub>), 2.2 (3H, s, COMe), 3.9-4.5 (2H, m, 8-H<sub>2</sub>), 5.1-5.3 (1H, m, 6a-H), 6.5 (1H, s, 10-H), 7.17-7.3 (2H, m, 2,3-H) and 7.37-7.4 (1H, m, 1-H).

*Anal.* Calcd. for C<sub>17</sub>H<sub>17</sub>N<sub>2</sub>: C, 76.4; H, 6.4; N, 5.25; M<sup>+</sup> 267.1259. Found: C, 76.4; H, 6.3; N, 5.4; M<sup>+</sup> 267.1260.

(3) Isolated as a light-stable glass [ir:  $\nu$  max 2240 s (C=C-CO<sub>2</sub>), 1690 (C=O) and 1650 (amide I) cm<sup>-1</sup>] by extraction with 0.5*M* sodium acetate [ $\alpha,\beta$ -acetylenic acids are considerably stronger acids than acetic acid (R. A. Raphael, "Acetylenic Compounds in Organic Synthesis", Butterworths, London, 1955, p 81)].



where R =

