STRUCTURE OF THE PRODUCT OF CONDENSATION OF DIETHYL OXALATE WITH β , β '-IMINODIPROPIONITRILE

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Treibs and Lange have reported [1] the preparation of 1-aza-3,6-dicyanocycloheptane-4,5-dione (III) by condensation of β , β -iminodipropionitrile (I) with diethyl oxalate (II):



However, in addition to a broad singlet centered at 9.0 ppm (1H, OH), only two triplets at 3.71 and 2.73 ppm (J = 6.5 Hz) and a singlet at 4.09 with relative intensities of two protons each were obtained in the PMR spectrum of the condensation product (which we synthesized by the Treibs – Lange method and was character-ized by the same physicochemical constants as those presented in [1] for III); these signals constitute unambiguous evidence for the presence of three methylene groups, two of which are directly bonded to one another. Consequently, the conclusion of Treibs and Lange regarding the formation of III as a result of Dieckmann condensation of I and II is erroneous, inasmuch as in this case the PMR spectrum should contain signals of the protons of two isolated methylene groups, an imino group, and a methylidyne group. In fact, dimethyl oxalate acylates only one methylene group and the imino group in I to give N-(β -cyanoethyl)-3-hydroxy-4-cyano-3-pyrrolin-2-one (IV), the structure of which is in complete agreement with the spectroscopic data.

LITERATURE CITED

1. W. Treibs and A. Lange, J. Prakt. Chem., 14, 208 (1961).

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