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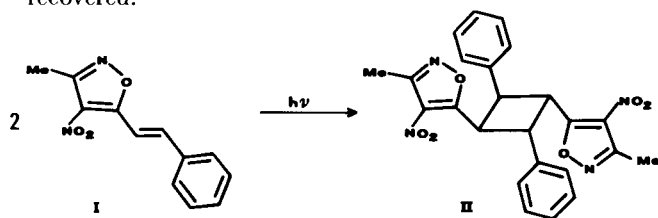
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The photoreactivity of 3-methyl-4-nitro-5-styrylisoxazole in the solid state was studied and the centrosymmetric product identified.

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Sir:

During a study of the reactivity of 3,5-dimethyl-4-nitroisoxazole it was noted that 3-methyl-4-nitro-5-styrylisoxazole (I), obtained as yellow needles (m.p. 153°) turned to a colourless compound (II) (m.p. 201-202°) when exposed to sunlight. The molecular weight and the elemental analysis of II were found to be in agreement with the values required for a dimer of I (1). We have now reinvestigated the photoreactivity of I in the solid state in order to clarify the molecular structure of the photoproduct obtained. Compound I irradiated with the light of a medium pressure mercury lamp (250 W) filtered with a glass filter, gave II as the main product. Unreacted monomer and other minor products were also recovered.



The best yield of II (90%) was obtained after 90 minutes irradiation of I deposited ( $\cong 0.30$  mg./cm<sup>2</sup>) on glass plates by evaporation from a benzene solution.

The uv spectrum of II in methanol [maxima at 219 nm ( $\log \epsilon = 4.18$ ) and at 269 nm ( $\log \epsilon = 4.06$ )] and the ir spectrum were in agreement for a cyclobutane type dimer but not determinant for the stereochemical assignment.

Chemical and nmr analysis proved the *trans* configuration of I. In fact alkaline hydrolysis of I followed by acidification gave *trans*-cinnamic acid. In addition the coupling constant of the ethylenic protons ( $J_{AB} = 16.6$  cps) of I deuterated in the phenyl ring is in agreement for a *trans* configuration.

The mass spectrum of II allows a distinction to be made between the possible truxinic or truxillic structures; in fact the presence of fragments at m/e (relative intensity) 460 (0.012, molecular ion) and 230 (100, monomer) together with the absence of signals at m/e 180

( $C_6H_5-CH=CH-C_6H_5$ )<sup>+</sup> is consistent only with a truxillic structure of II.

Accurate nmr analysis (2) of the multiplet AA'BB' type spectrum gave the following coupling constants ( $J_{AB} = 10.5$ ;  $J_{AB'} = 7.6$ ;  $J_{AA'} = J_{BB'} = 0$ ). The results are in agreement with a  $\alpha$ -truxillic type structure for II which was confirmed by the results of hydrolysis in alkaline aqueous solution. Compound II refluxed for 19 hours in 2% sodium hydroxide solution gave after acidification, a compound which was identified as  $\alpha$ -truxillic acid [m.p. and ir spectrum are the same as those of an authentic sample prepared by photodimerization of  $\alpha$ -*trans*-cinnamic acid (3)]. Alkaline ethanolysis of II gave a product (III) (m.p. 144-145°) identified as the diethyl ester of  $\alpha$ -truxillic acid.

Recently the importance of truxillic and truxinic acid derivatives as intermediates for polymers with photochemical properties was studied (4). In this work the reduction of the nitro groups of the two isoxazole rings was undertaken in order to prepare the diamino derivative of II. This reaction was achieved by refluxing II with stannous chloride and concentrated hydrochloric acid in alcohol (1). Evaporation of the alcohol separated a compound identified as II (40%) which was filtered off. By adding concentrated sodium hydroxide solution dropwise the expected diamino derivative (45%) precipitated. The elemental analysis was in agreement for the values required for C<sub>24</sub>H<sub>24</sub>N<sub>4</sub>O<sub>2</sub>; m.p. 199-200° dec. from alcohol. The uv spectrum in methanol shows  $\lambda_{max} = 213$  nm ( $\log \epsilon = 3.25$ ) and  $\lambda_{max} = 258$  nm ( $\log \epsilon = 4.05$ ).

#### REFERENCES AND NOTES

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