

2-Substituted 5-Nitrobenzimidazole 3-Oxides from Aldehydes

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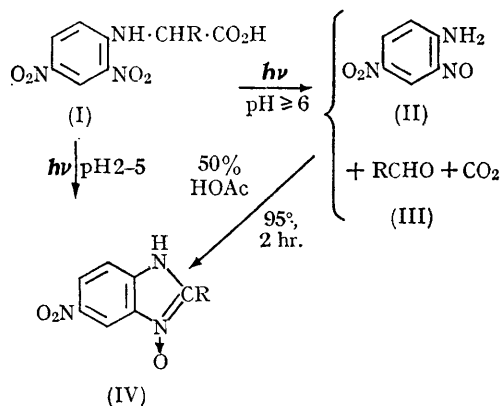
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PHOTOLYSIS of *N*-2,4-dinitrophenyl derivatives (I) of α -amino-acids in solution at pH 6 or higher yields 4-nitro-2-nitrosoaniline (II) and the corresponding aldehyde (III).^{1,2} At pH 2–5 the 2-substituted 5-nitrobenzimidazole 3-oxide (IV) is produced,³ this affording a convenient route to (IV) when the corresponding amino-acid is readily available. In other cases, in particular when R = aryl, the amino-acid must be prepared, usually from the corresponding aldehyde. A synthetic method which produced (IV) directly from the aldehyde would therefore effect a useful economy.

Without making any assumption as to the mechanism of the photochemical reaction leading to (IV), it seemed possible that 4-nitro-2-nitrosoaniline (II) might condense directly with aldehydes to yield the required compounds. When (II) was treated with 1.1 mol. of benzaldehyde under the conditions shown, 5-nitro-2-phenylbenzimidazole 3-oxide (IV; R = Ph), separating from the hot solution, was obtained in 46% yield after two crystallizations. It was identical with the product obtained by photolysing the *C*-phenylglycine derivative (I; R = Ph). The analogous compounds

(IV; R = *p*-chlorophenyl or *p*-cyanophenyl) were similarly prepared.

Since 4-nitro-2-nitrosoaniline (II) is readily accessible,¹ the technique affords a convenient general route to (IV), particularly for R = aryl. Although these compounds have high melting points, the reaction may find use for characterizing aldehydes.



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¹ D. W. Russell, *J. Chem. Soc.*, 1963, 894.

² D. W. Russell, *Biochem. J.*, 1963, 87, 1.

³ R. J. Pollitt, *Chem. Comm.*, 1965, 262.