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LETTERS TO THE EDITOR

FORMATION OF A FUROXAN RING IN REACTION OF 3,5-DINITRO-4-HYDROXYLAMINOPYRIDINE WITH PICRYL CHLORIDE

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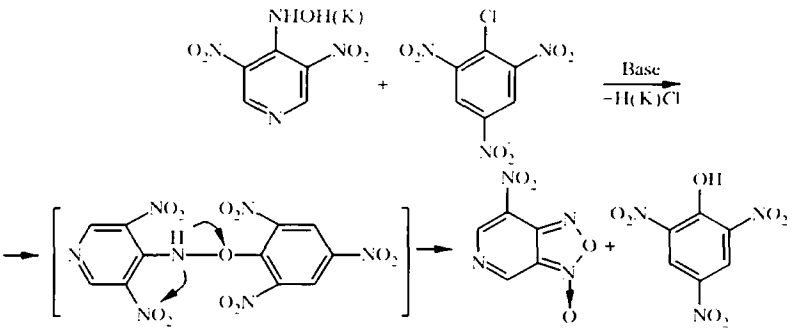
In a continuation of work on establishing the mechanism of the furoxan ring formation in the Nietsky–Dische reaction [1] for the example of pyrido derivatives, we have established that reaction of 3,5-dinitro-4-hydroxylaminopyridine with picryl chloride in the presence of base leads to 3-nitro-4,5-pyridofuroxan and picric acid. By an alternate synthesis (reaction of 3,5-dinitro-4-hydroxylaminopyridine potassium salt with picryl chloride), we obtained analogous compounds according to the scheme:



We explain the presence of a furoxan ring, as in the examples described earlier [1], by intermediate formation of an unstable hydroxylamine ester, the concerted decomposition of which leads to 3-nitro-4,5-pyridofuroxan and picric acid. Thus the given reactions indicate that the furoxan ring formation mechanisms are identical for both the arene and pyrido derivatives.

3-Nitro-4,5-pyridofuroxan was obtained by stirring of 3,5-dinitro-4-hydroxylaminopyridine (4.0 g, 0.02 mol) with picryl chloride (4.95 g, 0.02 mol) in methanol (30 ml) in the presence of base (sodium bicarbonate or acetate) for 15 min at 20-25°C. The reaction mass was diluted with water; the precipitate was filtered out, washed with chloroform and then water, and dried. Yield 3.2 g (88%); mp 134-135°C.

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