

SYNTHESIS OF 2-p-NITROBENZYL-5-PHENYL-1, 3, 4-OXADIAZOLE USING GREEN SOLVENT

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ABSTRACT

p-nitro-phenylacetic acid hydrazide was reacted with benzoyl chloride to produce diacyl hydrazide and simultaneously subjected to cyclization to 2,5-disubstituted oxadiazole using dichloromethane in place of earlier used carbon tetrachloride.

Key words: 2-p-nitrobenzyl-5-phenyl-1, 3, 4-oxadiazole, Green solvent.

INTRODUCTION

The present work deals with the synthesis of 2-p-nitrobenzyl-5-phenyl-1,3,4-oxadiazole using some green solvent instead of the solvents used such as carbon tetrachloride, pyridine solution etc.

EXPERIMENTAL

p-nitrophenylacetic acid (III) required for this synthesis was prepared by nitrating benzylcyanide (I) according to standard literature procedure and then hydrolyzing the nitrated benzylcyanide (II). The acid produced was esterified and the ester treated with hydrazine to give p-nitrophenylacetic acid hydrazide (V). This was then reacted with benzoyl chloride under forcing conditions to produce the diacylhydrazide and simultaneously to bring about its cyclization to the 2, 5-disubstituted oxadiazole (VI). The required cyclization was affected by heating the reactants with P_2O_5 in solvent (CCl₄) for 6 hours.

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The progress of the reaction was routinely checked by T.L.C from time to time, and the yields of the cyclized products were fairly good. The evolution of HCl in the reaction between the acid hydrazide and the acid chloride, evidently aids cyclodehydration in the above case.

$$\begin{array}{c} \text{CH}_2\text{CN} & \text{HNO}_3/\text{H}_2\text{SO}_4 \\ \text{O}_2\text{N} & \text{(II)} \\ \\ \text{O}_2\text{N} & \text{(III)} \\ \\ \text{CH}_2\text{COOH} \\ \\ \text{O}_2\text{N} & \text{(IV)} \\ \\ \text{O}_2\text{N} & \text{(IV)} \\ \\ \text{PhCONHNH}_2 & \text{CH}_2\text{COEt} \\ \\ \text{O}_2\text{N} & \text{(V)} \\ \\ \text{PhCOCI} \\ \\ \text{O}_2\text{N} & \text{(VI)} \\ \end{array}$$

RESULTS AND DISCUSSION

2-p-nitrobenzyl-1,3,4-oxadiazole had sharp melting point and was shown to be a pure compound. It analyzed correctly for the elements. Besides this, Its IR spectrum showed a sharp absorption at 1585 cm⁻¹ which was assigned to C=N stretching vibration, a strong absorption at 1510 cm⁻¹ characteristic of aromatic –NO₂ group, a band of medium intensity at 2900 cm⁻¹ due to C-H stretching and bands in the region 1010-1030 cm⁻¹ considered due to C-O bending vibration. These evidences were in agreement with the oxadiazole structure (VI).

Further corroboration of structure of the products was done by carrying out the synthesis alternatively as follows. p-nitrophenylacetic acid was first converted into its acid chloride and then reacted with benzhydrazide under cyclizing conditions as in the previous case. The product from this reaction was identical with that obtained earlier as shown by T.L.C, mixed melting point and the complete identity of their IR spectra.

Thus, the compound obtained in the above synthesis was undoubtedly 2-p-nitrobenzyl-5-phenyl-1,3,4-oxadiazole (V) prepared using the green solvents.

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