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Syntheses of 2-(1-Methyl-4-Nitro-2-Pyrrolyl)-5-Substituted-1,3,4-Thiadiazoles and 1,3,4-Oxadiazoles

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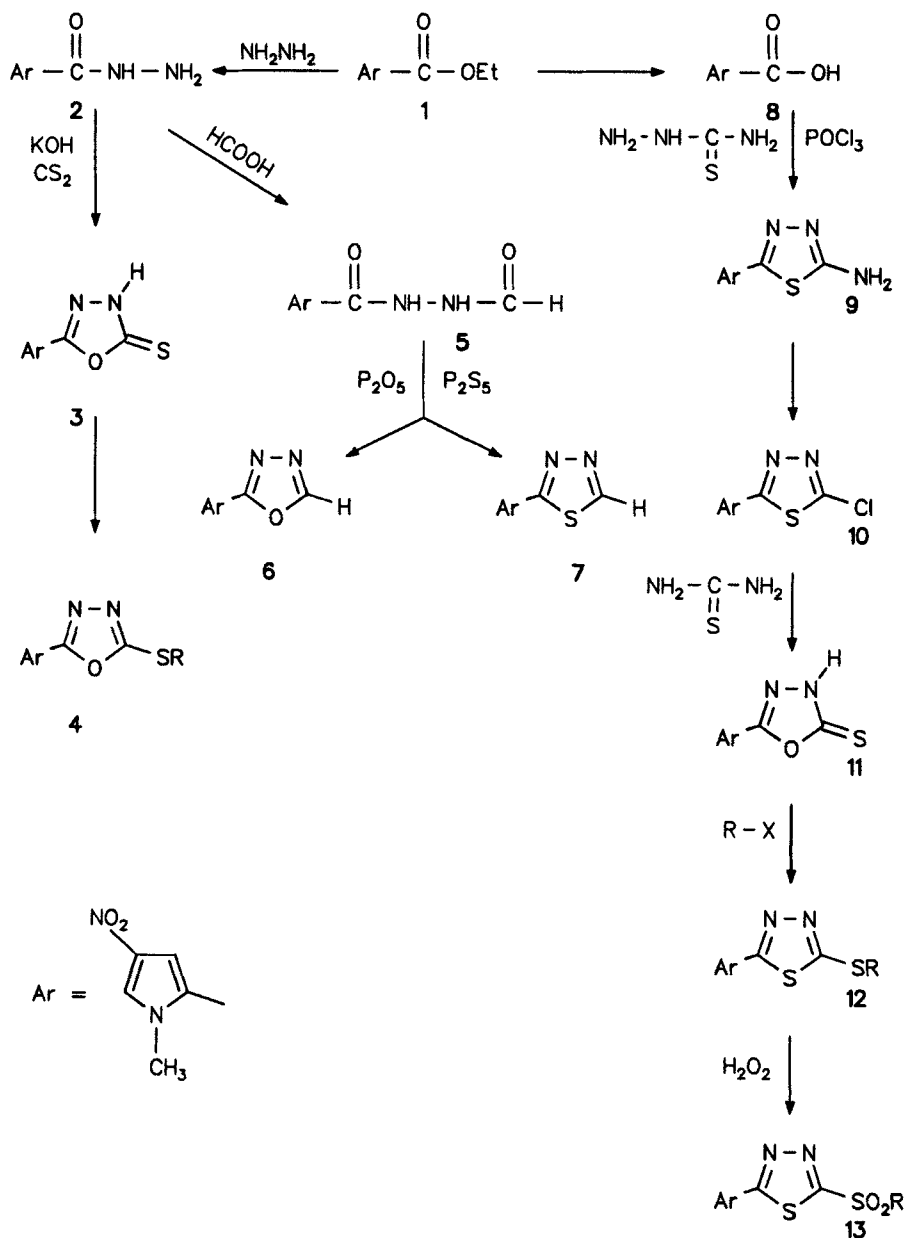
SYNTHESES OF 2-(1-METHYL-4-NITRO-2-PYRROLYL)-5-SUBSTITUTED-1,3,4-THIADIAZOLES AND 1,3,4-OXADIAZOLES

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Starting from readily available ethyl 1-methyl-4-nitropyrrole-2-carboxylate (1), the title compounds were prepared. Reaction of compound 1 with hydrazine hydrate afforded the corresponding hydrazide 2. The reaction of 2 with formic acid yielded 1-(1-methyl-4-nitropyrrole-2-carboxyl)-2-(formyl)hydrazine (5). Refluxing of the latter with phosphorus pentasulfide in xylene yielded compound 7 in 40% yield. Reaction of compound 5 with phosphorus pentoxide afforded compound 7.

Compound 3 could be obtained through the reaction of compound 2 with carbon disulfide in basic medium. Alkylation of compound 3 afforded the corresponding alkylthio derivative 4. Reaction of 1-methyl-4-nitropyrrole-2-carboxylic acid (8) with thiosemicarbazide and phosphorus oxychloride gave 2-amino-5-(1-methyl-4-nitro-2-pyrrolyl)1,3,4-thiadiazole (9). Sandmeyer reaction of compound 9 yielded 2-chloro-5-(1-methyl-4-nitro-2-pyrrolyl)1,3,4-thiadiazole (10). Refluxing of the latter with thiourea afforded 2-(1-methyl-4-nitro-2-pyrrolyl)1,3,4-thiadiazoline-4(H)-5-thione (11). Alkylation of compound 11 gave the corresponding alkylthio derivative 12. Oxidation of the latter with hydrogen peroxide in acetic acid yielded 2-(1-methyl-4-nitro-2-pyrrolyl)-5-methylsulfonyl-1,3,4-thiadiazole (13) (Scheme 1).

The structure of all compounds were confirmed by spectroscopic methods (UV, IR, NMR and MS).



Scheme 1