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NOVEL METHOD FOR THE *peri*-ANNELATION OF PYRROLE RING TO PERIMIDINES

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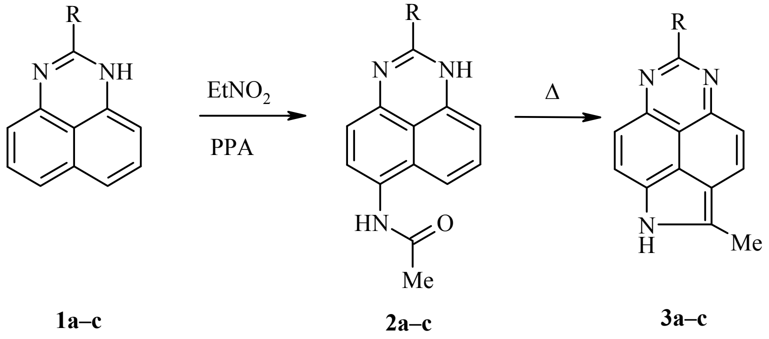
We have previously developed a series of methods for the *peri*-annelation of a pyrrole ring to a perimidine [1-3]. The drawback of these methods is the need for initially functionalizing the perimidines. In this work we propose a method for removing this drawback based on our recently reported system of reagents for acetamidation of aromatic compounds, *viz.* nitroethane and polyphosphoric acid (PPA) [4, 5]. It was found that reaction of 1 mmol of perimidines **1a-c** with nitroethane (0.1 g, 1.33 mmol) in PPA** (2-3 g) at 95-105ºC for 3 h (TLC monitoring) followed by 4 h at 150-160ºC (again TLC monitoring) gave the 1H-1,5,7-triazacyclopenta[*c,d*]phenalenes **3a-c** in 46-54% yields.

The reaction includes an intermediate formation of acetamides **2a-c** [4] which give compounds **3a-c** *via* thermal cyclization.



1–3 a R = H, **b** R = Me, **c** R = Ph

^{1}H NMR spectra were recorded on a Bruker WP-200 instrument (200 MHz) using TMS as internal standard. Monitoring of the reaction course and the purity of the compounds synthesized was carried out on Silufol UV-254 plates in the solvent system ethyl acetate–alcohol (1:1).

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** PPA with an 86% P_2O_5 content was prepared by method [6].

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The reaction mixture was treated with water (50 ml) and basified with a solution of ammonia to pH 8-9. The precipitate formed was filtered off. The mother liquor was extracted with hot benzene (5×50 ml). The solvent was evaporated and the residue was combined with the precipitate. The compounds prepared were purified by recrystallization from benzene.

2-Methyl-1H-1,5,7-triazacyclopenta[*c,d*]phenalene (3a). Yield 0.095 g (46%); mp 259-260ºC (benzene) (mp 259-260ºC [1]). The ^{1}H NMR spectrum was similar to that reported in [1].

2,6-Dimethyl-1H-1,5,7-triazacyclopenta[*c,d*]phenalene (3b). Yield 0.106 g (48%); mp 271-272ºC (benzene) (mp 271-272ºC [1]). The ^{1}H NMR spectrum was similar to that reported in [1].

2-Methyl-6-phenyl-1H-1,5,7-triazacyclopenta[*c,d*]phenalene (3c). Yield 0.158 g (54%); mp 245-246ºC (benzene with petrolium ether) (mp 245-246ºC [2]). The ^{1}H NMR spectrum was similar to that reported in [2].

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