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UNEXPECTED RESULT OF THE REACTION OF 2-ETHOXYNAPHTHALENE WITH 2,4,6-TRIMETHYL-1,3,5-TRIAZINE

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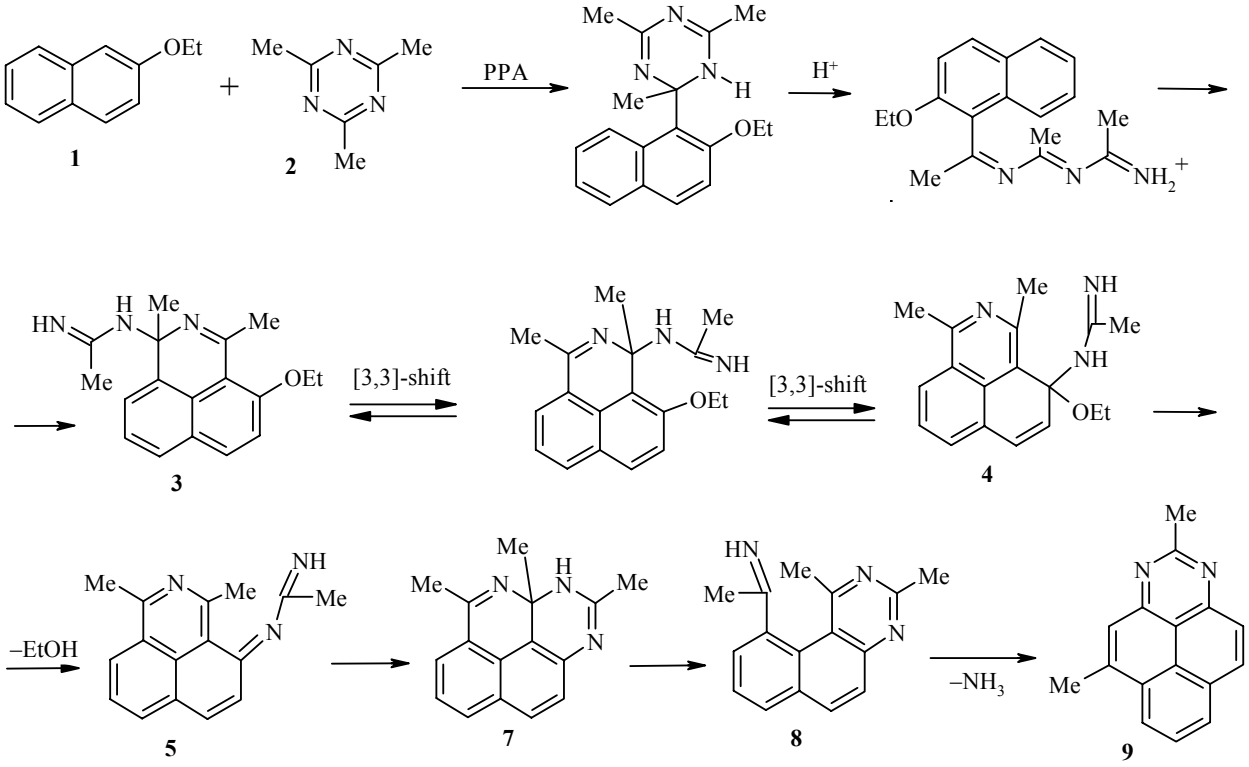
We have previously developed a method for the acylation (formylation) perimidines [1] and diacylation of 1-naphthol [2] based on their reactions with 1,3,5-triazine in PPA. It has been shown unexpectedly that the use of 2-ethoxynaphthalene (**1**) as the substrate in the reaction with 3,4,6-trimethyl-1,3,5-triazine (**2**) at an increase in temperature to 160-170°C led to a change in the direction of the reaction. Thus we observed that



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heating 1mmol of compound **1** with 1.8 mmol of triazine **2** in 3-4 g of PPA* initially for 1.5 h at 60-70°C and then for 6 h at 160-170°C (work up was normal for similar reactions) led to the previously unknown compound 2,5-dimethyl-1,3-diazapyrene **9** in 42% yield.

It is probable that the reaction proceeds through the following sequences of stages. As in all preceding reactions of naphthalene derivatives with 1,3,5-triazines [2, 4-6], a substituted dihydrotriazine is formed , the ring is opened and intermediate **3** is formed by intramolecular cyclization, which at higher temperature underwent two successive [3,3]-sigmatropic rearrangements to give intermediate **4.** Loss of ethanol led to intermediate **5** which cyclized into the tetracyclic derivative **7** ring opening of which leads to the imine **8** which rearranges via intramolecular condensation to the diazapyrene **9**.

^{1}H NMR spectra of DMSO-d_6 solutions containing TMS as internal standard were recorded on a Bruker -200 (200 MHz) instrument.

2,5-Dimethyl-1,3-diazapyrene (9). Yield 0.098 g (42%); mp 162-164°C (ethyl acetate). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.29 (3H, s, CH_3); 2.63 (3H, s, CH_3); 8.03 (1H, s, H-4); 8.21 (1H, d, *J* = 9.1, H-9); 8.24 (1H, t, *J* = 7.7, H-7); 8.48 (1H, d, *J* = 9.1, H-10); 8.67 (1H, d, *J* = 7.7, H-6); 8.74 (1H, d, *J* = 7.7, H-8). Found, %: C 82.86; H 5.15;N 11.99. $C_{16}H_{12}N_2$. Calculated, %: C 82.73; H 5.21; N 12.06.

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* The PPA used contained 86% P_2O_5 and was made by method [3].