

SHORT COMMUNICATIONS

Reaction of Vinylpyridines and Vinylquinolines with 1,3,5-Triazine in Polyphosphoric Acid

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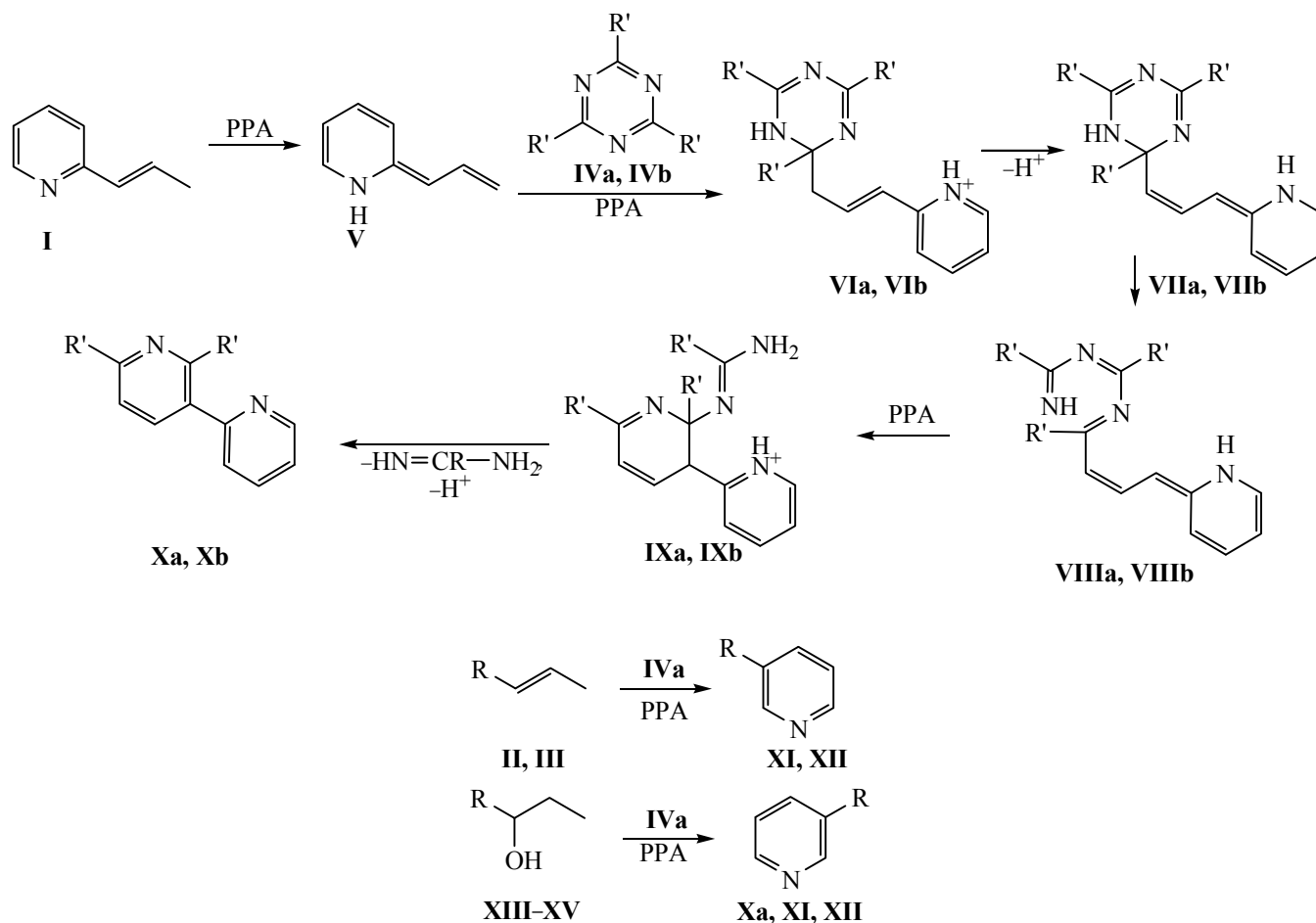
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We showed formerly that the system 1,3,5-triazine–polyphosphoric acid (PPA) was an efficient reagent for *peri*-fusion of a pyridine ring to azaphenalenenes [1, 2] and

1,8-naphthylenediamine [3]. Here we report on reactions of 2-propenylpyridine (**I**), 4-propenylpyridine (**II**), and 2-propenylquinoline (**III**) with 1,3,5-triazines



R = 2-pyridyl (**I**, **X**, **XIII**), 4-pyridyl (**II**, **XI**, **XIV**), 2-quinolyl (**III**, **XII**, **XV**); R' = H (a), Me (b).

IVa and **IVb** in PPA^a.

We showed that at heating compound **I** with triazines **IVa** and **IVb** in PPA formed 2,3'-bipyridyls **Xa** and **Xb** in 37 and 34% yield respectively. Similarly from azine **II** formed bipyridyl **XI**, from reagent **III**, compound **XII**.

Instead of compounds **I–III** their precursors, alcohols **XIII–XV**, can be employed.

General procedure. A mixture of 1 mmol of vinyltetraene **I–III** or alcohol **XIII–XV** and 3–4 g of PPA was vigorously stirred for 15 min at 60–65°C, 1.5 mmol of an appropriate 1,3,5-triazine was added, and the reaction mixture was stirred for 2 h at 70–75°C, then 5 h at 120–130°C. The reaction mixture was cooled, poured at stirring into 30 ml of cold water, alkalized with 25% aqueous ammonia, saturated with potassium carbonate, and extracted with ethyl acetate (5×50 ml). The solvent was evaporated, the residue was subjected to column chromatography. Eluent ethyl acetate–petroleum ether, 1:1.

2,3'-Bipyridyl (Xa). Yield 0.058 g (37%) ([from compound **I**), 0.056 g (from compound **XIII**). Yellow oily substance. Picrate, mp 153–154°C (mp 153–155°C [5]). ¹H NMR spectrum, δ, ppm: 7.27 d.d (1H, H^{5'}, *J*_{4',5'} 7.9, *J*_{5',6'} 4.9 Hz), 7.39 d.d (1H, H⁵, *J*_{4,5} 7.9, *J*_{5,6} 4.9 Hz), 7.74 d (1H, H³, *J*_{3,4} 7.9 Hz), 7.78 t (1H, H⁴ *J* 7.9 Hz), 8.31 d.d (1H, H^{4'}, *J*_{4',5'} 7.9, *J*_{2',4'} 2.1 Hz), 8.64 d (1H, H⁶, *J*_{5,6} 4.9 Hz), 8.72 d (1H, H^{6'}, *J*_{5',6'} 4.9 Hz), 9.18 d (1H, H^{2'}, *J*_{2',4'} 2.1 Hz). Found, %: C 77.08; H 5.11; N 17.81. C₁₀H₈N₂. Calculated, %: C 76.90; H 5.16; N 17.94.

2',6'-Dimethyl-2,3'-bipyridyl (Xb). Yield 0.063 g (34%). Yellow oily substance. Picrate, mp 136–138°C ¹H NMR spectrum, δ, ppm: 2.42 s (3H, Me), 2.46 s (3H, Me), 7.27 d (1H, H^{5'}, *J*_{4',5'} 7.9 Hz), 7.39 d.d (1H, H⁵, *J*_{4,5} 7.9, *J*_{5,6} 4.9 Hz), 7.54 d (1H, H³, *J*_{3,4} 7.9 Hz), 7.59 t (1H, H⁴, *J* 7.9 Hz), 8.21 d (1H, H^{4'}, *J*_{4',5'} 7.9 Hz), 8.89 d (1H, H⁶, *J*_{5,6} 4.9 Hz). Found, %: C 78.39; H 6.49; N 15.12.

C₁₂H₁₂N₂. Calculated, %: C 78.23; H 6.57; N 15.20.

3,4'-Bipyridyl (XI). Yield 0.064 g (41%) (from compound **II**), 0.055 g (35%) (from compound **XIV**). Yellow oily substance. Picrate, mp 205–206°C (mp 204–205°C [5]). ¹H NMR spectrum, δ, ppm: 7.41 d.d (1H, H⁵, *J*_{4,5} 7.9, *J*_{5',6'} 4.9 Hz), 7.49 d (2H, H^{3'}, H^{5'}, *J* 4.9 Hz), 7.74 d (1H, H³, *J*_{3,4} 7.9 Hz), 7.91 d.d (1H, H⁴, *J*_{4,5} 7.9, *J*_{2,4} 2.1 Hz), 8.67 d (1H, H⁶, *J*_{5,6} 4.9 Hz), 8.70 d (2H, H^{2'}, H^{6'}, *J* 4.9 Hz), 8.88 d (1H, H^{2'}, *J*_{2',4'} 2.1 Hz). Found, %: C 77.06; H 5.09; N 17.85. C₁₀H₈N₂. Calculated, %: C 76.90; H 5.16; N 17.94.

2-(3-Pyridyl)quinoline (XII). Yield 0.116 g (56%) (from compound **III**), 0.117 g (57%) (from compound **XV**), mp 69–71°C (from petroleum ether) (mp 70–71°C [5]). ¹H NMR spectrum corresponds to the published one [6]. Found, %: C 81.28; H 4.71; N 13.53. C₁₄H₁₀N₂. Calculated, %: C 81.53; H 4.89; N 13.58.

NMR spectra were registered on a spectrometer Bruker WP-200 (200 MHz) in CDCl₃, internal reference TMS. The reactions progress was monitored and the homogeneity of compounds obtained was checked by TLC on Silufol UV-254 plates, eluent ethyl acetate–petroleum ether, 1:1.

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^a PPA used contained 86% of P₂O₅ and was prepared by procedure [4].