

Synthesis of unsymmetric 6,6'-diaryl-2,2'-bipyridines using a 1,2,4-triazine methodology*

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New unsymmetric 6,6'-diaryl-2,2'-bipyridines were synthesized in high yields using a "1,2,4-triazine" methodology. Their photophysical properties were studied.

Key words: 1,2,4-triazine, Diels–Alder reaction, 6,6'-diaryl-2,2'-bipyridines.

α,α' -Diaryl-substituted 2,2'-bipyridines and their annulated analogs 1,10-phenanthrolines are of practical interest as ligands for transition metal cations,¹ catalysts,² as well as components for organic light-emission diodes (OLED).^{3,4} In supramolecular chemistry, such compounds are used for obtaining macrocyclic systems.⁵ Besides, there are examples of application of diaryl-substituted bipyridines as photoluminescent sensors for explosives.⁶

Analysis of approaches to the synthesis of such structures showed that earlier they were obtained by nucleophilic substitution of hydrogen at α -positions of 2,2'-bipyridine or 1,10-phenanthroline in the reactions with aromatic nucleophiles under conditions of oxidative aromatization,^{7,8} as well as by various cross-coupling reactions.^{9,10} There are selected examples of obtaining such compounds by the Krenke heterocyclization reaction of pyridine rings,^{11,12} as well as by the metal-catalyzed dimerization of pyridine derivatives^{13,14} and noncatalytic dimerization of *N*-oxides.¹⁵ Most often all these approaches deal with the synthesis of symmetric representatives of 6,6'-diaryl-2,2'-bipyridines. Unsymmetric compounds can be obtained by the introduction of different aromatic moieties at α -positions of two pyridine rings as a result of S_NH -reaction^{16,17} or cross-coupling.¹⁸ A selective modification of one of the substituents in a symmetric bipyridine was also described.¹⁹

In this paper, we suggest a synthetic approach to unsymmetric 6,6'-diaryl-2,2'-bipyridines using a "1,2,4-triazine" methodology of obtaining of substituted pyrid-

ines.^{20,21} Earlier, this methodology was successfully used for the synthesis of symmetric α,α' -diaryl-substituted 2,2'-bipyridines resulting from the Diels–Alder aza-reaction with 3,3'-(5,5')-bis-1,2,4-triazines.^{22,23} The starting triazines were synthesized by heterocyclization based on diacetamide dihydrazone,^{24,25} by dimerization of 3(5)-unsubstituted triazine in the presence of cyanides,^{26,27} as well as in the reaction of phenylmagnesium bromide with 3-chloro-5,6-diphenyl-1,2,4-triazine (a side product).²⁸ Only separate publications are devoted to the preparation of nonsymmetrically functionalized bis-1,2,4-triazines,^{29,30} while the examples of their transformation to the corresponding bipyridines are absent at all.

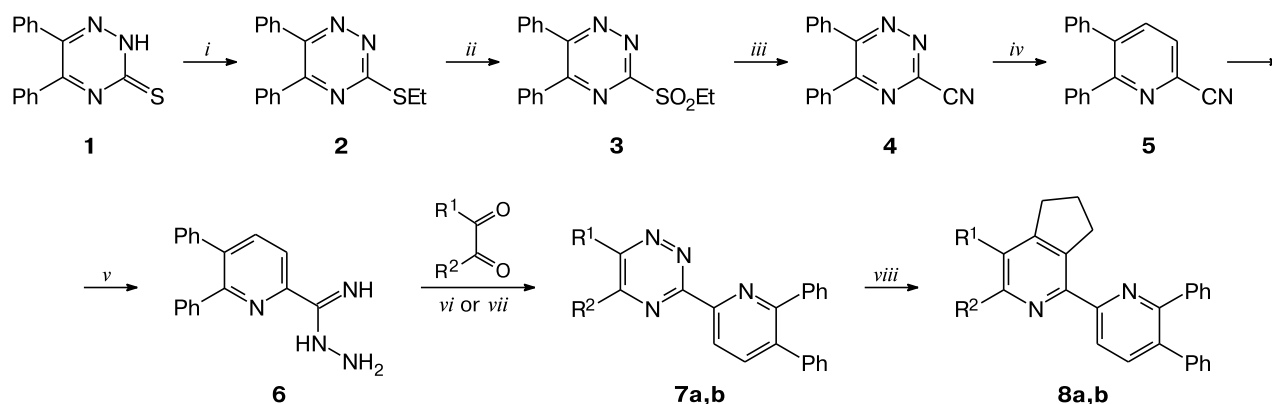
In the present work, we suggest the use of the "1,2,4-triazine" methodology for obtaining substituted pyridines in order to selectively assemble unsymmetrically functionalized 2,2'-bipyridines.

Readily available diphenyl-1,2,4-triazinethione **1**³¹ was used as the starting compound (Scheme 1). Then, we used procedures suggested earlier for another substrates to *S*-alkylate³² and oxidize³³ ethylthio derivative **2** with the formation of sulfonyl derivative **3**. Further *ipso*-substitution for the sulfonyl group with a cyano group carried out according to a modified procedure³⁴ allowed us to obtain 3-cyano-5,6-diphenyl-1,2,4-triazine **4**, which was converted to the corresponding cyanopyridine **5** by the aza-Diels–Alder reaction with 2,5-norbornadiene. To assemble the second triazine ring, the cyano group in compound **5** was transformed. The subsequent reaction of amidrazone **6** with 1,2-diones gave rise to the aza-analogs of the target unsymmetrically functionalized bipyridines **7**.

It is necessary to note that pyridines during their synthesis by the "1,2,4-triazine" methodology can be addi-

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Scheme 1



$R^1, R^2 = \text{biphenyl-2,2'-diyl}$ (a); $R^1 = \text{H}, R^2 = \text{Ph}$ (b)

Reagents and conditions: *i.* KOH, EtOH, 25 °C, 30 min; then EtI, 25 °C, 4 h; *ii.* KMnO₄, AcOH—acetone (1 : 10), 5 °C, then 25 °C, 4 h; *iii.* KCN, DMF, 25 °C, 1.5 h; *iv.* 2,5-norbornadiene, toluene, 110 °C, 8 h; *v.* hydrazine hydrate, EtOH—THF (1 : 1), 25 °C, 3 days; *vi.* ethanol—THF (1 : 1), reflux, 10 h; *vii.* EtOH, 78 °C, 10 h; *viii.* 1-morpholinocyclopentene, 200 °C, 3 h.

tionally functionalized by using various dienophiles, for example, 1-morpholinocyclopentene. The reaction resulted in the obtaining of the target ligands **8**. Note that the preparation of functionalized cyclopenta[*c*]pyridines by other synthetic approaches seems very problematic, while the introduction of a fused cyclopentene fragment frequently makes it possible to improve properties of compounds, in particular, their solubility.³⁵

The photophysical properties of new ligands **8** studied in this work are given in Table 1, their luminescence spectra are shown in Fig. 1.

According to the data obtained, the introduction of two additional phenyl rings in the pyridylmonoazatriphenylene system considerably increased the quantum yield of luminescence (from 21.3% to 43.2%) with the shift of the emission maximum from 381 nm to 474 nm (see Ref. 37), while the introduction of two phenyl substituents at different positions of the monoazatriphenylene part of the molecule³⁸ led to a decrease in the quantum

yield of luminescence. Thus, the changes in the structure of the pyridylmonoazatriphenylene system performed in this work are more promising from the point of view of tuning photophysical properties. As to bipyridine **8b**, in this case we observed a hypsochromic shift of the absorption and emission maxima as compared to compound **8a** and a higher quantum yield of photoluminescence.

In conclusion, in the present work we suggested an efficient synthetic approach to the unsymmetrically aryl-functionalized 2,2'-bipyridines, which are poorly available by other methods of synthesis. Photophysical properties of compounds obtained were studied.

Experimental

¹H and ¹³C NMR spectra were recorded on a Bruker Avance-400 spectrometer (400 MHz and 100 MHz, respectively), using

Table 1. Photophysical properties of compounds **8a,b**

Compound	λ/nm		Φ^c
	A_{max}^a	I_{max}^b	
8a	210, 252, 263 sh, 307 sh, 389	474	0.432
8b	213, 240, 292 пл, 353	459	0.576

^a A are the absorption maxima in acetonitrile.

^b I are the luminescence maxima in acetonitrile.

^c Φ is the quantum yield of luminescence. Quantum yields for all the compounds were measured relative to quinine sulfate ($\Phi = 0.546$ in 0.1 *N* aqueous solution of H₂SO₄).³⁶

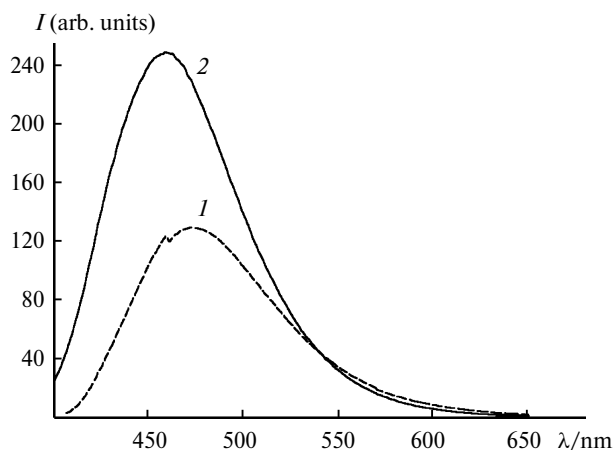


Fig. 1. Luminescence spectra of bipyridines **8a** (I) and **8b** (2).

SiMe₄ as an internal standard. Melting points were measured on a Boetius apparatus. Electrospray ionization mass spectra were recorded on a Bruker Daltonics MicrOTOF-Q II instrument (Bremen, Germany). Elemental analysis was performed on a Perkin Elmer PE 2400, series II CHN-analyzer. Absorption spectra were recorded on a Shimadzu UV-2401PC spectrophotometer. Luminescence spectra were recorded on a Varian Cary Eclipse fluorimeter. 5,6-Diphenyl-1,2,4-triazine-3-thione (**1**)³¹ and phenylglyoxal³⁹ were obtained according to the described procedure.

5,6-Diphenyl-3-ethylthio-1,2,4-triazine (2). 5,6-Diphenyl-1,2,4-triazine-3-thione **1** (3.5 g, 13.2 mmol) was added to a solution of potassium hydroxide (0.74 g, 13.2 mmol) in ethanol (120 mL) cooled to room temperature, the mixture obtained was stirred for 30 min. Then, ethyl iodide (2.42 mL, 31.02 mmol) was added dropwise, the resulting mixture was stirred for 4 h at room temperature and quenched with water (100 mL). The product was extracted with dichloromethane (3×70 mL), the extract was dried with anhydrous sodium sulfate, the solvents were evaporated at reduced pressure. Butan-1-ol (20 mL) was added to the residue, the mixture was allowed to stand for 4 h at -18 °C. A precipitate formed was filtered off and dried. The compound was used in the next step without additional purification. The yield was 2.71 g (70%), m.p. 67–69 °C. ¹H NMR (DMSO-d₆), δ: 1.48 (t, 3 H, CH₃, *J* = 7.2 Hz); 3.31 (q, 2 H, CH₂, *J* = 7.2 Hz); 7.31–7.51 (m, 10 H, Ph). MS (ESI), *m/z* (%): 294.11 [M + H]⁺ (100).

3-Ethylsulfonyl-5,6-diphenyl-1,2,4-triazine (3). A solution of triazine **2** (2.71 g, 9.24 mmol) in a mixture of glacial acetic acid (9 mL) and acetone (90 mL) was cooled in an ice–water bath for 10 min, followed by the addition of potassium permanganate (2.92 g, 18.48 mmol) and stirring for 4 h at room temperature. After quenching with a saturated aqueous solution of sodium sulfite (40 mL), the mixture was poured into water (800 mL). A precipitate formed was filtered off, washed with water (150 mL), and dried. Compound **3** was used in the next step without additional purification. The yield was 2.61 g (87%), m.p. 105–107 °C. ¹H NMR (DMSO-d₆) δ: 1.43 (t, 3 H, CH₃, *J* = 7.2 Hz); 3.73 (q, 2 H, CH₂, *J* = 7.2 Hz); 7.34–7.61 (m, 10 H, Ph). MS (ESI), *m/z* (%): 326.10 [M + H]⁺ (100).

3-Cyano-5,6-diphenyl-1,2,4-triazine (4). Potassium cyanide (1.05 g, 16.08 mmol) was added to a solution of sulfone **3** (2.61 g, 8.04 mmol) in dry DMF (20 mL) and the mixture obtained was stirred for 1.5 h at room temperature, followed by quenching with water (50 mL) and extraction with dichloromethane (3×50 mL). The extract was washed with water and dried with anhydrous sodium sulfate, the solvents were evaporated at reduced pressure. The residue was treated with butan-1-ol, a precipitate formed was filtered off and dried. The product was used in the next step without additional purification. The yield was 1.24 g (60%). ¹H NMR (DMSO-d₆), δ: 7.35–7.60 (m, 10 H, Ph). MS (ESI), *m/z* (%): 259.10 [M + H]⁺ (100).

2-Cyano-5,6-diphenylpyridine (5). 2,5-Norbornadiene (1.47 mL, 14.46 mmol) was added to a suspension of triazine **4** (1.24 g, 4.82 mmol) in toluene (40 mL) and the mixture obtained was refluxed for 8 h. The solvent was evaporated at reduced pressure, the residue was treated with ethanol, a precipitate was filtered off, washed with ethanol, and dried. An analytical sample was recrystallized from ethanol. The yield was 0.93 g (75%), m.p. 126–128 °C. Found (%): C, 84.24; H, 4.59; N, 10.74. C₁₈H₁₂N₂. Calculated (%): C, 84.35; H, 4.72; N, 10.93. ¹H NMR (DMSO-d₆), δ: 7.16–7.21 (m, 2 H, Ph); 7.23–7.33 (m, 8 H,

Ph); 7.99 (d, 1 H, pyridine, *J* = 7.8 Hz); 8.02 (d, 1 H, pyridine, *J* = 7.8 Hz). MS (ESI), *m/z* (%): 257.11 [M + H]⁺ (100).

5,6-Diphenylpyridine-2-carboxamide hydrazone (6). Hydrazone hydrate (190 μL, 3.9 mmol) was added to a solution of diphenylpyridine **5** (0.5 g, 1.95 mmol) in a mixture of ethanol–THF (1 : 1, 50 mL). The mixture obtained was allowed to stand for 3 days at room temperature. The solvents were evaporated at reduced pressure, the residue was treated with diethyl ether, a precipitate was filtered off and dried. The compound was used in the next step without additional purification. The yield was 450 mg (80%). ¹H NMR (DMSO-d₆), δ: 5.35 (br.s, 2 H, NH₂); 7.15–7.20 (m, 3 H, Ph); 7.23–7.30 (m, 4 H, Ph); 7.36–7.41 (m, 3 H, Ph); 7.74 (d, 1 H, pyridine, *J* = 8.3 Hz); 8.03 (d, 1 H, pyridine, *J* = 8.3 Hz). MS (ESI), *m/z* (%): 289.15 [M + H]⁺ (100).

3-(5,6-Diphenylpyridin-2-yl)phenanthro[9,10-*e*]-1,2,4-triazine (7a). 9,10-Phenanthrenequinone (150 mg, 0.72 mmol) was dissolved in a mixture of ethanol–THF (1 : 1, 100 mL) with heating, followed by the addition of amidrazone **6** (210 mg, 0.72 mmol) and reflux for 10 h. Then, the reaction mixture was cooled to room temperature, a precipitate formed was filtered off, washed with ethanol, and dried. An analytical sample was recrystallized from ethanol. The yield was 150 mg (45%), m.p. 244–246 °C. Found (%): C, 83.36; H, 4.33; N, 12.23. C₃₂H₂₀N₄. Calculated (%): C, 83.46; H, 4.38; N, 12.17. ¹H NMR (DMSO-d₆), δ: 7.28–7.38 (m, 8 H, Ph); 7.49–7.53 (m, 2 H, Ph); 7.86–7.93 (m, 2 H); 7.97 (m, 1 H); 8.02 (m, 1 H); 8.11 (d, 1 H, pyridine, *J* = 8.2 Hz); 8.81 (d, 1 H, pyridine, *J* = 8.2 Hz); 8.85 (m, 2 H); 9.41 (dd, 1 H, H(5), *J* = 7.0 Hz, *J* = 1.0 Hz); 9.45 (dd, 1 H, H(12), *J* = 7.0 Hz, *J* = 1.0 Hz). MS (ESI), *m/z* (%): 461.18 [M + H]⁺ (100).

3-(5,6-Diphenylpyridine-2-yl)-5-phenyl-1,2,4-triazine (7b). Amidrazone **6** (285 mg, 0.99 mmol) was added to a solution of phenylglyoxal hydrate (150 mg, 0.99 mmol) in ethanol (80 mL) and the mixture obtained was refluxed for 10 h. The solvent was evaporated at reduced pressure, the residue was recrystallized from ethanol. The yield was 155 mg (0.40 mmol, 40%), m.p. 142–144 °C. Found (%): C, 80.74; H, 4.53; N, 14.61. C₂₆H₁₈N₄. Calculated (%): C, 80.81; H, 4.69; N, 14.50. ¹H NMR (DMSO-d₆), δ: 7.25–7.35 (m, 8 H, Ph); 7.43–7.47 (m, 2 H, Ph); 7.60–7.68 (m, 3 H, Ph (triazine)); 8.04 (d, 1 H, pyridine, *J* = 8.1 Hz); 8.45–8.49 (m, 2 H, Ph (triazine)); 8.58 (d, 1 H, pyridine, *J* = 8.1 Hz); 10.09 (s, 1 H, H(6)). MS (ESI), *m/z* (%): 387.16 [M + H]⁺ (100).

Synthesis of unsymmetric 2,2'-bipyridines 8 (general procedure). A mixture of the corresponding triazine (0.25 mmol) and 1-morpholinocyclopentene (0.2 mL, 1.25 mmol) was stirred for 2 h at 200 °C under argon. Then, 1-morpholinocyclopentene (0.1 mL, 0.625 mmol) was added and the stirring was continued for another 1 h under the same conditions. Then, the reaction mixture was cooled to room temperature, a corresponding solvent was added, brought to boiling, and cooled to room temperature. A precipitate formed was filtered off, washed with the same solvent, and dried. An analytical sample was obtained by recrystallization from the same solvent.

10-(5,6-Diphenylpyridin-2-yl)-12,13-dihydro-11*H*-dibenzo[*f,h*]cyclopenta[*c*]quinoline (8a). Acetonitrile was used as the solvent. The yield was 95 mg (76%), m.p. 215–217 °C. Found (%): C, 89.05; H, 5.13; N, 5.39. C₃₇H₂₆N₂. Calculated (%): C, 89.13; H, 5.26; N, 5.62. ¹H NMR (CDCl₃), δ: 2.24–2.33 (m, 2 H, C(12)H₂); 3.71–3.80 (m, 4 H, C(11)H₂, C(13)H₂); 7.27–7.35 (m, 8 H, Ph); 7.49–7.54 (m, 2 H, Ph); 7.63–7.77 (m, 4 H); 7.97 (d, 1 H, pyridine, *J* = 8.2 Hz); 8.59–8.63 (m, 1 H); 8.65–8.69

(m, 1 H); 8.70–8.74 (m, 1 H); 8.78 (d, 1 H, pyridine, $J = 8.2$ Hz); 9.54–9.59 (m, 1 H, H(8)). ^{13}C NMR (CDCl_3), δ : 26.0, 33.7, 37.3, 121.7, 122.3, 123.2, 123.3, 126.2, 126.5, 127.1, 127.2, 127.3, 127.6, 127.7, 128.2, 128.3, 129.7, 130.2, 130.8, 131.2, 131.9, 135.1, 139.0, 139.1, 140.4, 140.7, 145.0, 149.7, 151.8, 155.6, 157.3. MS (ESI), m/z (%): 499.22 $[\text{M} + \text{H}]^+$ (100).

1-(5,6-Diphenylpyridin-2-yl)-3-phenyl-6,7-dihydro-5H-cyclopenta[c]pyridine (8b). Ethanol was used as the solvent. The yield was 75 mg (72%), m.p. 152–154 °C. Found (%): C, 87.54; H, 5.61; N, 6.47. $\text{C}_{31}\text{H}_{24}\text{N}_2$. Calculated (%): C, 87.70; H, 5.70; N, 6.60. ^1H NMR (CDCl_3), δ : 2.12–2.22 (m, 2 H, $\text{CH}_2(6)$); 3.03 (t, 2 H, $\text{CH}_2(7)$, $J = 7.6$ Hz); 3.61 (t, 2 H, $\text{CH}_2(5)$, $J = 7.6$ Hz); 7.23–7.32 (m, 8 H, Ph); 7.38–7.44 (m, 1 H, Ph); 7.45–7.53 (m, 4 H, Ph); 7.69 (s, 1 H, H(4)); 7.88 (d, 1 H, pyridine, $J = 7.7$ Hz); 8.13–8.17 (m, 2 H, Ph); 8.57 (d, 1 H, pyridine, $J = 7.7$ Hz). ^{13}C NMR (CDCl_3), δ : 25.1, 32.8, 33.6, 116.5, 121.3, 126.9, 127.2, 127.6, 127.8, 128.4, 128.5, 128.6, 129.7, 130.2, 134.9, 138.2, 139.2, 140.0, 140.3, 140.6, 151.0, 154.4, 155.5, 156.9, 157.2. MS (ESI), m/z (%): 425.20 $[\text{M} + \text{H}]^+$ (100).

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