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New push-pull fluorophores on the basis of 6-alkoxy-2,2'-bipyridines: rational synthetic approach and photophysical properties

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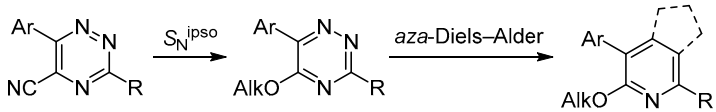


Alk = Me, Et, Pr, CH_2CF_3, $C_{14}H_{29}$; R = 2-Py, Ar^1

We propose an effective synthetic approach to 2(6)-alkoxy(bi)pyridines as new push-pull fluorophores *via* their 1,2,4-triazine analogs. The photophysical properties of the new compounds were studied and the first study of their structure-property relationship was performed.

Keywords: 6-alkoxy-2,2'-bipyridines, 2-alkoxypyridines, 1,2,4-triazines, autoclave, aza-Diels–Alder reactions, luminescence, nucleophilic *ipso* substitution.

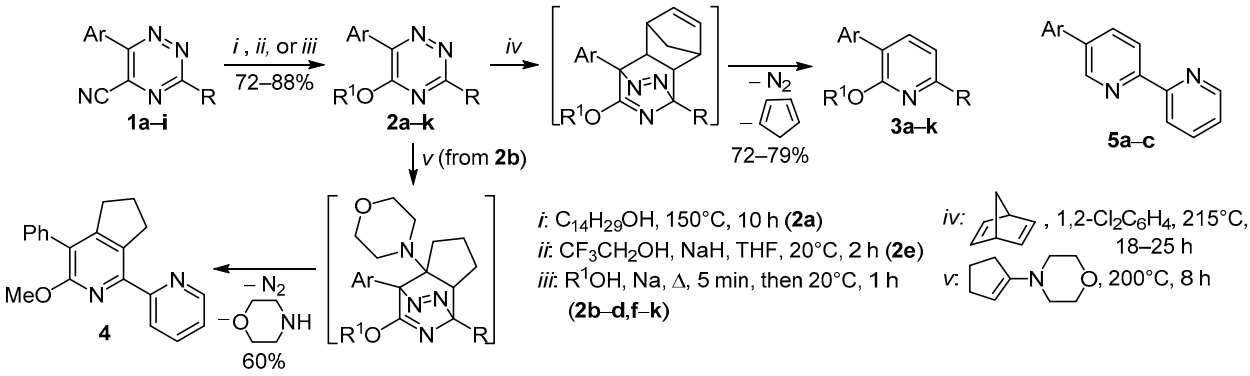
The interest toward 2,2'-bipyridines is motivated by their promising luminescent properties, including when used in metal complexes. Significant attention has been devoted to the functionalization of 2,2'-bipyridines as a tool for the fine tuning of their practically useful properties. For example, an improvement in the photophysical and electroluminescent properties of copper complexes containing 2,2'-bipyridine ligands was observed upon the introduction of methoxy groups in their molecules.[1,2] Promising luminescent properties were found in the case of lanthanide complexes containing 2,2'-bipyridine or its fused analog in the role of chromophore,[3] while iridium complexes of alkoxy-2,2'-bipyridines can be employed as catalysts for the hydrogenation of CO_2.[4] Interesting results have also been obtained by studying the photophysical properties of free 2,2'-bipyridine ligands.[5]

Many methods are currently known for the synthesis of 6-alkoxy-2,2'-bipyridines and their analogs. For example, various procedures for the assembly of 2,2'-bipyridine framework are widely used that involve cross-coupling reactions[6–8] between two pyridine-containing synthons, as well as some homo-coupling processes.[9] Another approach relies on *ipso* substitution of halogen atoms in 2,2'-bipyridines, for example, the Williamson reaction[1,10] and other methods.[11–17]

In addition, it should be noted that such compounds can be obtained through their 1,2,4-triazine analogs, providing an effective methodology in many cases.[18] In particular, this method was proposed for the preparation of 6,6'-dimethoxy-2,2'-bipyridine on the basis of bis-1,2,4-triazine precursor that can be obtained by direct dimerization of 3-methoxy-1,2,4-triazine. In that particular example, an aza-Diels–Alder reaction with 2,5-norbornadiene served as the final step.[19]

Besides that, it is also necessary to mention a strategy for the preparation of functionalized bipyridines using a sequence of steps: nucleophilic substitution of hydrogen or good leaving groups in 1,2,4-triazine and aza-Diels–Alder reactions. This strategy was used, for example, for the preparation of bipyridines containing various moieties: carboranes,[20] acetylenes,[21] C–H-active compounds,[22]

Table 1. The synthesis and structure of compounds **2**, **3 a**–**k** and photophysical properties of 2,6-diarylpyridines **3a**–**k**, **5a**–**c***



Cyano-triazine	Alkoxy-triazine	Pyridine	Ar	R	R^1	λ_{abs}, nm (MeCN)	λ_{em}, nm (MeCN)	Φ, % (MeCN)
–	–	**5a**[32]	Ph	2-Py	–	298	357	3.2
1a	**2a**	**3a**	Ph	2-Py	$C_{14}H_{29}$	318	369	57.3
1a	**2b**	**3b**	Ph	2-Py	Me	308	369	1.7
1a	**2b**	**4**	Ph	2-Py	Me	314	368	26.4
1b	**2c**	**3c**	Ph	2-FC_6H_4	Pr	222 sh, 263, 319	366	17.6
–	–	**5b**[32]	*p*-Tol	2-Py	–	302	360	17
1c	**2d**	**3d**	*p*-Tol	2-Py	Me	234, 319	374	73.8
1d	**2e**	**3e**	*p*-Tol	Ph	CH_2CF_3	262, 311	353 sh, 365	8.7
1d	**2f**	**3f**	*p*-Tol	Ph	Me	263, 315	370	15.6
–	–	**5c**[32]	4-$MeOC_6H_4$	2-Py	–	309	399	89
1e	**2g**	**3g**	4-$MeOC_6H_4$	2-Py	Me	323	396	95.4
1f	**2h**	**3h**	4-$MeOC_6H_4$	2-Thienyl	Me	281, 333	397	33.6
1g	**2i**	**3i**	4-$MeOC_6H_4$	*p*-Tol	Me	228, 240 sh, 263 sh, 272, 319	383	33.7
1h	**2j**	**3j**	4-FC_6H_4	2-Py	Me	316	356 sh, 368	57.3
1i	**2k**	**3k**	4-FC_6H_4	4-FC_6H_4	Et	260, 315	364	5.3

* λ_{abs} and λ_{em} – the wavelengths of absorption and emission maxima, respectively, Φ – luminescence quantum yield.

coumarins,[23] cyano groups,[24] polycyclic aromatic hydrocarbons,[25] and others. We have recently demonstrated[26] the possibility for the synthesis of 6-methoxy-5-phenyl-2,2'-bipyridine by using an analogous methodology on the basis of its mono-1,2,4-triazine precursor, where the reaction with 2,5-norbornadiene was performed under high pressure.[27]

The method described above was applied in the current study to develop a convenient approach for the synthesis of 2,2'-bipyridine fluorophores containing various alkoxy groups at the α-position. We selected the previously described[24,28] 5-cyano-1,2,4-triazines **1a**–**i** as starting materials (Table 1). Nucleophilic *ipso* substitution of cyano group with an alkoxy group was achieved by two methods: by treatment with sodium alkoxide solution in the respective alcohol[29,30] or by our previously proposed solvent-free procedure in the case of higher alcohols (tetradecanol).[31] The further reaction of triazines **2a**–**k** with 2,5-norbornadiene proceeded according to a previously described procedure[26] and was performed at elevated temperature and pressure, while a more conventional procedure relying on refluxing in high-boiling organic solvents was not successful. When the C-3 position of 1,2,4-triazine contained a group that was different from the electron-withdrawing 2-pyridyl substituent, even under these conditions a longer time was necessary to complete the reaction. As a result, 6-alkoxy(bi)pyridines **3a**–**k** were obtained in good yields.

When reacting with an excess of 1-morpholinocyclopentene under solvent-free conditions at 200°C according to a literature procedure,[5,32] 5-methoxy-1,2,4-triazine **2a** was transformed into 2,2'-bipyridine **4** that contained a fused cyclopentene ring. It was also observed that complete conversion required a reaction time as long as 8 h, unlike the previously used reaction duration of 3 h. The obtained 6-alkoxy-2,2'-bipyridines **3** and **4** were subsequently purified by flash chromatography.

The photophysical properties of products **3a**–**k**, **4** are listed in Table 1. For comparison, the photophysical properties of some 6-unsubstituted 5-aryl-2,2'-bipyridines **5a**–**c** are given according to the literature.[32] When the obtained data are considered in the context of earlier reports, it can be concluded that the introduction of an alkoxy group at the position C-6 of 5-phenyl-2,2'-bipyridine resulted in a bathochromic shift of the absorption maxima (by as much as 20 nm) and emission maxima (by as much as 12 nm). Besides that, we should note the substantial increase in the luminescence quantum yield

from 3.2 to 57.3% when a group containing 14 carbon atoms was introduced into the molecule (compound **3a**). The fusion of cyclopentene ring with pyridine ring influenced the luminescence intensity somewhat less (compound **4**, quantum yield 26.4%). In the case of 4-tolyl-bipyridine **3d**, a similar situation was observed, namely, the introduction of a methoxy group led to a bathochromic shift of the emission maximum by 14 nm compared to compound **5b**. The luminescence quantum yield for compound **3d** reached 73.8%. When a 4-methoxyphenyl substituent was present at the position C-6 (compound **3g**), the introduction of alkoxy groups led to a slight hypsochromic shift of the emission maximum (by 3 nm) compared to compound **5c**, but the highest quantum yield of luminescence (95.4%) was thus achieved for the studied series of luminophores. The introduction of a fluorine atom in the aromatic substituent of bipyridine (compound **3j**) gave a substantial improvement of the quantum yield of luminescence from 1.7 to 57.3%.

A replacement of 2-pyridyl ring with other (hetero)-aromatic fragments usually degrades the photophysical properties, which is reflected by the hypsochromic shift of emission and absorption maxima and a decrease in the quantum yield of luminescence.

The previously described 4-methylsulfanyl derivatives of 2,2'-bipyridines containing a methoxy group at the position C-6, on the other hand, showed only very weak fluorescence.[14]

Thus, we have proposed a rational approach to the preparation of new bipyridine fluorophores containing various alkoxy groups at the α-position. A wide range of possibilities was demonstrated for variation of substituents in the pyridine ring, as well as the alkoxy groups. The photophysical properties of the new compounds were studied and in some cases improvements were observed that resulted from the introduction of an alkoxy group.

Experimental

UV-visible absorption spectra were recorded on a Shimadzu UV-1800 spectrometer, while luminescence spectra and absolute quantum yields were obtained using a HORIBA Scientific FluoroMax-4 spectrofluorometer according to a published method.[33] ^{1}H, ^{13}C, and ^{19}F NMR spectra were acquired on a Bruker Avance II spectrometer (400, 100, and 376 MHz, respectively), using TMS (for ^{1}H and ^{13}C nuclei) or $CFCl_3$ (for ^{19}F nuclei) as internal standards. Mass spectra were recorded on a Bruker Daltonics micrOTOF-Q II instrument, using electrospray ionization. Elemental analyses were performed on a PerkinElmer 2400 Series II CHN-analyzer. The reaction progress and purity of the obtained products were controlled by TLC on Sigma-Aldrich 91835 plates. The products were isolated by chromatography on silica gel from Sigma-Aldrich (230–400 mesh).

The starting 5-cyano-1,2,4-triazines **1a**–**i**[24,28] and 3-phenyl-6-(*p*-tolyl)-5-(2,2,2-trifluoroethoxy)-1,2,4-triazine (**2e**)[30] were obtained according to literature procedures.

6-Phenyl-3-(2-pyridyl)-5-tetradecyloxy-1,2,4-triazine (2a). A mixture of 1,2,4-triazine-5-carbonitrile **1a** (100 mg, 0.39 mmol) and tetradecan-1-ol (0.1 ml, 0.39 mmol) was stirred at 150°C under argon atmosphere for 10 h. The product was isolated by column chromatography (eluent AcOEt, R_f 0.4) and used in the next step without additional purification. An analytically pure sample was obtained by recrystallization from MeCN. Yield 152 mg (88%), light-yellow crystals, mp 98–100°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 0.87 (3H, t, *J* = 6.8, CH_3); 1.18–1.43 (20H, m, $(C\underline{H}_2)_{10}CH_3$); 1.43–1.55 (2H, m, $O(CH_2)_2C\underline{H}_2$); 1.83–1.94 (2H, m, $OCH_2C\underline{H}_2$); 4.67 (2H, t, *J* = 6.8, OCH_2); 7.47–7.56 (4H, m, H Ph, H-5 Py); 7.96 (1H, td, *J* = 7.6, *J* = 1.0, H-4 Py); 8.08–8.12 (2H, m, H Ph); 8.50 (1H, d, *J* = 8.0, H-3 Py); 8.78 (1H, d, *J* = 4.8, H-6'). Mass spectrum, *m*/*z* (I_{rel}, %): 447 $[M+H]^+$ (100). Found, %: C 75.10; H 8.17; N 12.38. $C_{28}H_{38}N_4O$. Calculated, %: C 75.30; H 8.58; N 12.54.

Preparation of 5-alkoxy-1,2,4-triazines 2b–d,f–k (General method). 1,2,4-Triazine-5-carbonitrile **1** (1.0 mmol) was added to a solution of sodium alkoxide, obtained from metallic sodium (10 mg) and the appropriate alcohol (25 ml). The mixture was stirred and refluxed for 5 min, then maintained at room temperature for 1 h. The solvent was removed at reduced pressure. The products were isolated using column chromatography (eluent AcOEt, R_f 0.4) and used in the next step without additional purification. Analytically pure samples were obtained by recrystallization from MeCN.

5-Methoxy-3-(2-pyridyl)-6-phenyl-1,2,4-triazine (2b). Yield 212 mg (80%), yellow crystals, mp 214–216°C (mp 214–216°C[26]). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 4.25 (3H, s, OCH_3); 7.52–7.59 (4H, m, H Ph, H-5 Py); 8.00 (1H, td, *J* = 7.8, *J* = 1.8, H-4 Py); 8.09–8.11 (2H, m, H Ph); 8.53 (1H, dd, *J* = 7.8, *J* = 0.8, H-3 Py); 8.81 (1H, dd, *J* = 4.8, *J* = 1.8, H-6 Py). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 54.4 (OCH_3); 123.8; 125.6; 128.4; 129.1; 130.3; 132.4; 137.3; 148.9; 150.0; 152.4; 160.4; 161.0. Mass spectrum, *m*/*z* (I_{rel}, %): 265 $[M+H]^+$ (100). Found, %: C 68.01; H 4.43; N 20.98. $C_{15}H_{12}N_4O$. Calculated, %: C 68.17; H 4.58; N 21.20.

3-(2-Fluorophenyl)-6-phenyl-5-propoxy-1,2,4-triazine (2c). Yield 238 mg (72%), light-yellow crystals, mp 166–168°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.07 (3H, t, *J* = 8.0, $(CH_2)_2C\underline{H}_3$); 1.91 (2H, q, *J* = 8.0, $C\underline{H}_2CH_3$); 4.56 (2H, t, *J* = 8.0, OCH_2); 7.27–7.37 (2H, m, H Ar); 7.51–7.58 (4H, m, H Ar); 8.08–8.18 (3H, m, H Ar). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 10.7; 21.8; 69.2; 117.1 (d, *J* = 22.6); 123.7 (d, *J* = 9.5); 124.2 (d, *J* = 3.8); 128.4; 129.2; 130.3; 131.6 (d, *J* = 1.6); 132.5 (2C); 132.7; 147.9; 160.2 (2C); 160.3; 161.6 (d, *J* = 258.6). Mass spectrum, *m*/*z* (I_{rel}, %): 310 $[M+H]^+$ (100). Found, %: C 69.80; H 5.15; N 13.49. $C_{18}H_{16}FN_3O$. Calculated, %: C 69.89; H 5.21; N 13.58.

5-Methoxy-3-(2-pyridyl)-6-(*p*-tolyl)-1,2,4-triazine (2d). Yield 220 mg (79%), light-yellow crystals, mp 210–212°C (mp 209–211°C[34]). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 2.47 (3H, s, CH_3); 4.26 (3H, s, OCH_3); 7.34–7.39 (2H, m, H Ar); 7.55–7.60 (1H, m, H-5 Py); 7.99–8.06 (3H, m, H-4 Py, H Ar); 8.52 (1H, dd, *J* = 7.8, *J* = 0.8, H-3 Py); 8.82 (1H, dd, *J* = 4.8, *J* = 1.8, H-6 Py). Mass spectrum, *m*/*z*

(I_{rel}, %): 279 $[M+H]^+$ (100). Found, %: C 69.15; H 5.19; N 20.02. $C_{16}H_{14}N_4O$. Calculated, %: C 69.05; H 5.07; N 20.13.

5-Methoxy-3-phenyl-6-(*p*-tolyl)-1,2,4-triazine (2f). Yield 208 mg (75%), light-yellow crystals, mp 202–204°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 2.44 (3H, s, CH_3); 4.22 (3H, s, OCH_3); 7.29–7.32 (2H, m, H Ar); 7.52–7.55 (3H, m, H Ar); 7.97–7.99 (2H, m, H Ar); 8.46–8.49 (2H, m, H Ar). Mass spectrum, *m*/*z* (I_{rel}, %): 278 $[M+H]^+$ (100). Found, %: C 73.55; H 5.35; N 15.09. $C_{17}H_{15}FN_3O$. Calculated, %: C 73.63; H 5.45; N 15.15.

5-Methoxy-6-(4-methoxyphenyl)-3-(2-pyridyl)-1,2,4-triazine (2g). Yield 215 mg (73%), yellow crystals, mp 222–224°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 3.88 (3H, s, OCH_3); 4.26 (3H, s, OCH_3); 7.00–7.06 (2H, m, H Ar); 7.48–7.53 (1H, m, H-5 Py); 7.95 (1H, td, *J* = 7.8, *J* = 1.8, H-4 Py); 8.10–8.15 (2H, m, H Ar); 8.50 (1H, dd, *J* = 7.8, *J* = 0.8, H-3 Py); 8.77 (1H, dd, *J* = 4.8, *J* = 1.8, H-6 Py). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 53.2; 54.4; 113.0; 122.9; 123.9; 124.1; 129.9; 135.8; 147.6; 149.2; 152.1; 159.0; 160.0; 160.6. Mass spectrum, *m*/*z* (I_{rel}, %): 295 $[M+H]^+$ (100). Found, %: C 65.43; H 4.66; N 19.19. $C_{16}H_{14}N_4O_2$. Calculated, %: C 65.30; H 4.79; N 19.04.

5-Methoxy-6-(4-methoxyphenyl)-3-(2-thienyl)-1,2,4-triazine (2h). Yield 234 mg (78%), light-yellow crystals, mp 219–222°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.87 (3H, s, OCH_3); 4.18 (3H, s, OCH_3); 7.00–7.02 (2H, m, H Ar); 7.19–7.21 (1H, m, H thiophene); 7.68–7.69 (1H, m, H thiophene); 8.02–8.05 (3H, m, H thiophene, H Ar). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 54.0; 55.4; 113.9; 125.1; 128.2; 129.6; 130.6 (2C); 139.8; 147.2; 158.0; 160.3; 161.3. Mass spectrum, *m*/*z* (I_{rel}, %): 300 $[M+H]^+$ (100). Found, %: C 60.09; H 4.29; N 13.98. $C_{15}H_{13}N_3O_2S$. Calculated, %: C 60.19; H 4.38; N 14.04.

5-Methoxy-6-(4-methoxyphenyl)-3-(*p*-tolyl)-1,2,4-triazine (2i). Yield 246 mg (80%), light-yellow crystals, mp 202–204°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.45 (3H, s, CH_3); 3.87 (3H, s, OCH_3); 4.21 (3H, s, OCH_3); 7.01–7.03 (2H, m, H Ar); 7.31–7.33 (2H, m, H Ar); 8.05–8.07 (2H, m, H Ar); 8.33–8.35 (2H, m, H Ar). Mass spectrum, *m*/*z* (I_{rel}, %): 308 $[M+H]^+$ (100). Found, %: C 70.24; H 5.51; N 13.59. $C_{18}H_{17}N_3O_2$. Calculated, %: C 70.34; H 5.58; N 13.67.

6-(4-Fluorophenyl)-5-methoxy-3-(2-pyridyl)-1,2,4-triazine (2j). Yield 215 mg (76%), yellow crystals, mp 199–201°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 4.26 (3H, s, OCH_3); 7.24–7.31 (2H, m, H Ar); 7.50–7.55 (1H, m, H-5 Py); 7.97 (1H, td, *J* = 7.8, *J* = 1.8, H-4 Py), 8.17–8.22 (2H, m, H Ar); 8.52 (1H, dd, *J* = 7.8, *J* = 0.8, H-3 Py); 8.78 (1H, dd, *J* = 4.8, *J* = 1.8, H-6 Py). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 54.3; 115.6 (d, *J* = 22.3); 124.1; 125.3; 128.6 (d, *J* = 2.9); 131.4; 131.5; 136.9; 148.2; 150.3; 152.9; 161.2; 164.3 (d, *J* = 251.3). Mass spectrum, *m*/*z* (I_{rel}, %): 283 $[M+H]^+$ (100). Found, %: C 63.70; H 3.82; N 19.72. $C_{15}H_{11}FN_4O$. Calculated, %: C 63.83; H 3.93; N 19.85.

5-Ethoxy-3,6-bis(4-fluorophenyl)-1,2,4-triazine (2k). Yield 238 mg (76%), light-yellow crystals, mp 188–190°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.53 (3H, t, *J* = 8.0, $CH_2C\underline{H}_3$); 4.71 (2H, q, *J* = 8.0, $C\underline{H}_2CH_3$); 7.24–7.30 (4H, m, H Ar); 8.12–8.17 (2H, m, H Ar); 8.49–8.53 (2H, m, H Ar). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 14.1; 63.4; 115.5 (d, *J* = 21.3); 115.8 (d, *J* = 22.0); 128.8 (d, *J* = 3.5); 130.3; 130.4; 131.0 (d, *J* = 3.5); 131.1; 131.2; 146.9; 160.2 (2C); 164.0 (d, *J* = 251.5); 165.1 (d, *J* = 252.5). Mass spectrum, *m*/*z* (I_{rel}, %): 314 $[M+H]^+$ (100). Found, %: C 65.08; H 4.10; N 13.35. $C_{17}H_{13}F_2N_3O$. Calculated, %: C 65.17; H 4.18; N 13.41.

Preparation of bipyridines 3a–k (General method). The appropriate 1,2,4-triazine **2a**–**k** (1.0 mmol) was suspended in 1,2-dichlorobenzene (25 ml), then 2,5-norbornadiene (0.82 ml, 8.0 mmol) was added. The obtained mixture was stirred in autoclave at 215°C for 18 h (in the case of products **3c**,**e**,**f**,**h**,**i**,**k** – for 25 h). The solvent was removed by evaporation at reduced pressure. The products were isolated using column chromatography (eluent CH_2Cl_2, R_f 0.5). Analytically pure samples were obtained by recrystallization from MeCN.

5-Phenyl-6-tetradecyloxy-2,2'-bipyridine (3a). Yield 350 mg (79%), colorless crystals, mp 80–82°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 0.87 (3H, t, *J* = 6.8, CH_3); 1.16–1.40 (20H, m, $(C\underline{H}_2)_{10}CH_3$); 1.40–1.50 (2H, m, $O(CH_2)_2C\underline{H}_2$); 1.74–1.84 (2H, m, $OCH_2C\underline{H}_2$); 4.47 (2H, t, *J* = 6.8, OCH_2); 7.30–7.43 (4H, m, H Ph, H-5'); 7.60–7.62 (2H, m, H Ph); 7.82 (1H, d, *J* = 7.6, H-3); 7.86 (1H, td, *J* = 7.6, *J* = 1.6, H-4'); 8.07 (1H, d, *J* = 7.6, H-4); 8.35 (1H, d, *J* = 8.0, H-3'); 8.62 (1H, dd, *J* = 4.8, *J* = 2.0, H-6'). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 14.2; 22.8; 26.3; 29.0; 29.4; 29.5; 29.7 (3C); 29.8 (2C); 32.0; 66.1; 68.0; 113.9; 121.0; 123.4; 124.7; 127.5; 128.2; 129.3; 136.8 (2C); 139.5; 149.2; 152.2; 156.1; 160.0. Mass spectrum, *m*/*z* (I_{rel}, %): 445 $[M+H]^+$ (100). Found, %: C 81.16; H 9.17; N 6.48. $C_{30}H_{40}N_2O$. Calculated, %: C 81.03; H 9.07; N 6.30.

6-Methoxy-5-phenyl-2,2'-bipyridine (3b). Yield 205 mg (78%), colorless crystals, mp 155–157°C (mp 155–157°C[26]). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 4.09 (3H, s, OCH_3); 7.26–7.31 (1H, m, H-5'); 7.33–7.38 (1H, m, H Ph); 7.41–7.47 (2H, m, H Ph); 7.61–7.65 (2H, m, H Ph); 7.76 (1H, d, *J* = 7.6, H-3); 7.81 (1H, td, *J* = 7.6, *J* = 1.6, H-4'); 8.09–8.13 (1H, d, *J* = 7.6, H-4); 8.45 (1H, d, *J* = 8.0, H-3'); 8.68 (1H, d, *J* = 4.8, H-6'). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 53.4 (OCH_3); 114.2; 121.0; 123.5; 124.8; 127.6; 128.3; 129.2; 136.7; 136.8; 139.5; 149.2; 152.2; 156.0; 160.2. Mass spectrum, *m*/*z* (I_{rel}, %): 263 $[M+H]^+$ (100). Found, %: C 77.76; H 5.30; N 10.50. $C_{17}H_{14}N_2O$. Calculated, %: C 77.84; H 5.38; N 10.68.

6-(2-Fluorophenyl)-3-phenyl-2-propoxypyridine (3c). Yield 224 mg (73%), colorless crystals, mp 94–96°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.12 (3H, t, *J* = 8.0, $(CH_2)_2C\underline{H}_3$); 1.92 (2H, q, *J* = 8.0, $C\underline{H}_2CH_3$); 4.53 (2H, t, *J* = 8.0, OCH_2); 7.21–7.53 (6H, m, H Ar); 7.62–7.65 (1H, m, H-5); 7.73–7.78 (3H, m, H-4, H Ar); 8.24–8.28 (1H, m, H Ar). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 11.0; 22.4; 67.7; 116.3 (d, *J* = 23.4); 117.5 (d, *J* = 11.6); 123.3; 124.4 (d, *J* = 3.2); 126.9; 127.0; 127.5; 128.2; 129.3; 130.0; 130.1; 130.8 (d, *J* = 3.2); 136.7; 139.1; 149.0 (2C); 160.2; 161.0 (d, *J* = 250.4). Mass spectrum, *m*/*z* (I_{rel}, %): 308 $[M+H]^+$ (100). Found, %:

C 78.11; H 5.81; N 4.48. $C_{20}H_{18}FNO$. Calculated, %: C 78.15; H 5.90; N 4.56.

6-Methoxy-5-(*p*-tolyl)-2,2'-bipyridine (3d). Yield 210 mg (76%), colorless crystals, mp 151–153°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 2.31 (3H, s, CH_3); 4.00 (3H, s, OCH_3); 7.14–7.20 (2H, m, H Ar); 7.32–7.38 (1H, m, H-5'); 7.42–7.47 (2H, m, H Ar); 7.67 (1H, d, *J* = 7.9, H-3); 7.72 (1H, td, *J* = 7.6, *J* = 1.6, H-4'); 8.01 (1H, d, *J* = 7.6, H-4); 8.36 (1H, d, *J* = 8.0, H-3'); 8.59 (1H, d, *J* = 4.8, H-6'). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 21.3; 53.4; 114.2; 121.0; 123.4; 124.8; 129.0; 129.1; 133.7; 136.8; 137.5; 139.3; 149.2; 151.9; 156.0; 160.2. Mass spectrum, *m*/*z* (I_{rel}, %): 277 $[M+H]^+$ (100). Found, %: C 78.08; H 5.71; N 10.29. $C_{18}H_{16}N_2O$. Calculated, %: C 78.24; H 5.84; N 10.14.

6-Phenyl-3-(*p*-tolyl)-2-(2,2,2-trifluoroethoxy)pyridine (3e). Yield 248 mg (72%), colorless crystals, mp 135–137°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 2.39–2.50 (3H, s, CH_3); 5.00–5.07 (2H, t, *J* = 13.6, OCH_2); 7.22–7.49 (7H, m, H Ar); 7.65 (1H, d, *J* = 7.9, H-5); 7.84 (1H, d, *J* = 7.9, H-4); 8.05–8.11 (2H, m, H Ar). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 21.3; 62.1 (q, *J* = 36.0, $\underline{C}H_2CF_3$); 114.8; 123.0; 124.1 (q, *J* = 278.0, $CH_2\underline{C}F_3$); 126.7; 128.8; 129.0; 129.1; 129.2; 132.7; 137.7; 138.2; 140.0; 152.9; 157.8. ^{19}F NMR spectrum (DMSO-d_6), δ, ppm: –72.61 (3F, m). Mass spectrum, *m*/*z* (I_{rel}, %): 344 $[M+H]^+$ (100). Found, %: C 69.79; H 4.63; N 4.57. $C_{20}H_{16}F_3NO$. Calculated, %: C 69.96; H 4.70; N 4.08.

2-Methoxy-6-phenyl-3-(*p*-tolyl)pyridine (3f). Yield 194 mg (70%), colorless crystals, mp 149–151°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 2.40 (3H, s, CH_3); 4.08 (3H, s, OCH_3); 7.24–7.25 (2H, m, H Ar); 7.39–7.53 (6H, m, H-5, H Ar); 7.67 (1H, d, *J* = 7.9, H-4); 8.08–8.11 (2H, m, H Ar). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 21.3; 53.4; 113.1; 123.0; 126.6; 128.7; 128.8; 129.1 (2C); 133.6; 137.3; 138.9; 139.1; 153.1; 160.4. Mass spectrum, *m*/*z* (I_{rel}, %): 276 $[M+H]^+$ (100). Found, %: C 82.79; H 6.12; N 4.99. $C_{19}H_{17}NO$. Calculated, %: C 82.88; H 6.22; N 5.09.

6-Methoxy-5-(4-methoxyphenyl)-2,2'-bipyridine (3g). Yield 222 mg (76%), colorless crystals, mp 161–163°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 3.83 (3H, s, OCH_3); 4.06 (3H, s, OCH_3); 6.92–6.94 (2H, m, H Ar); 7.31–7.34 (1H, m, H-5'); 7.52–7.54 (2H, m, H Ar); 7.82–7.86 (2H, m, H-3,4'); 8.07 (1H, d, *J* = 7.4, H-4); 8.37 (1H, d, *J* = 8.0, H-3'); 8.60 (1H, d, *J* = 4.8, H-6'). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 53.5; 55.4; 113.8; 114.2; 121.0; 123.4; 124.5; 128.9; 130.4; 136.8; 139.0; 149.2; 151.7; 156.1; 159.2; 160.1. Mass spectrum, *m*/*z* (I_{rel}, %): 293 $[M+H]^+$ (100). Found, %: C 73.81; H 5.36; N 9.51. $C_{18}H_{16}N_2O_2$. Calculated, %: C 73.96; H 5.52; N 9.58.

2-Methoxy-3-(4-methoxyphenyl)-6-(2-thienyl)pyridine (3h). Yield 232 mg (78%), colorless crystals, mp 131–133°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 3.88 (3H, s, OCH_3); 4.07 (3H, s, OCH_3); 6.98–6.99 (2H, m, H Ar); 7.11–7.13 (1H, m, H-5); 7.26–7.33 (1H, m, H Ar); 7.37 (1H, d, *J* = 7.9, H-4); 7.55–7.61 (4H, m, H thiophene, H Ar). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 53.6; 55.4; 111.8; 113.8; 122.4; 124.1; 127.0; 128.0; 129.0; 130.2; 138.7; 145.1; 148.4; 159.1; 160.1. Mass spectrum, *m*/*z* (I_{rel}, %): 298 $[M+H]^+$ (100). Found, %: C 68.67; H 4.99; N 4.61. $C_{17}H_{15}NO_2S$. Calculated, %: C 68.66; H 5.08; N 4.71.

2-Methoxy-3-(4-methoxyphenyl)-6-(*p*-tolyl)pyridine (3i). Yield 230 mg (75%), colorless crystals, mp 141–143°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 2.40 (3H, s, CH_3); 3.82 (3H, s, OCH_3); 4.03 (3H, s, OCH_3); 7.91–7.93 (2H, m, H Ar); 7.22–7.24 (2H, m, H Ar); 7.46–7.51 (3H, m, H-5, H Ar); 7.67 (1H, d, *J* = 7.9, H-4); 7.94–7.96 (2H, m, H Ar). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 21.4; 53.4; 55.4; 112.8; 113.8; 122.3; 126.5; 129.2; 129.4; 130.3; 136.2; 138.8 (2C); 152.9; 159.1; 160.2. Mass spectrum, *m*/*z* (I_{rel}, %): 306 $[M+H]^+$ (100). Found, %: C 78.59; H 6.18; N 4.50. $C_{20}H_{19}NO_2$. Calculated, %: C 78.66; H 6.27; N 4.59.

5-(4-Fluorophenyl)-6-methoxy-2,2'-bipyridine (3j). Yield 205 mg (73%), colorless crystals, mp 157–159°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 4.07 (3H, s, OCH_3); 7.12–7.16 (2H, m, H-5', H Ar); 7.32–7.35 (1H, m, H Ar); 7.59–7.63 (2H, m, H Ar); 7.78–7.87 (2H, m, H-3,4'); 8.09 (1H, d, *J* = 7.4, H-4); 8.39 (1H, d, *J* = 8.0, H-3'); 8.61 (1H, d, *J* = 4.8, H-6'). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 53.8; 114.4; 115.6 (d, *J* = 22.4); 121.0; 123.5; 124.6; 131.5; 131.6; 132.7 (d, *J* = 2.2); 137.8; 140.2; 149.8; 152.2 (2C); 160.0; 160.2 (d, *J* = 243.4). Mass spectrum, *m*/*z* (I_{rel}, %): 281 $[M+H]^+$ (100). Found, %: C 72.77; H 4.49; N 9.86. $C_{17}H_{13}FN_2O$. Calculated, %: C 72.85; H 4.67; N 9.99.

2-Ethoxy-3,6-bis(4-fluorophenyl)pyridine (3k). Yield 240 mg (77%), colorless crystals, mp 126–128°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.42 (3H, t, *J* = 8.0, $CH_2C\underline{H}_3$); 4.53 (2H, q, *J* = 8.0, $C\underline{H}_2CH_3$); 7.12–7.20 (4H, m, H Ar); 7.50 (1H, d, *J* = 7.9, H-5); 7.60–7.63 (2H, m, H Ar); 7.73 (1H, d, *J* = 7.9, H-4); 8.08–8.12 (2H, m, H Ar). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 14.9; 61.8; 113.5; 115.5 (d, *J* = 21.4); 116.0 (d, *J* = 21.4); 121.5; 128.9 (2C); 131.3; 131.4; 132.8 (d, *J* = 3.4); 134.9 (d, *J* = 3.4); 140.1; 151.9; 159.6; 161.9 (d, *J* = 186.2); 163.5 (d, *J* = 188.5). Mass spectrum, *m*/*z* (I_{rel}, %): 312 $[M+H]^+$ (100). Found, %: C 73.25; H 4.77; N 4.41. $C_{19}H_{15}F_2NO$. Calculated, %: C 73.30; H 4.86; N 4.50.

3-Methoxy-4-phenyl-1-(2-pyridyl)-6,7-dihydro-5*H*-cyclopenta[*c*]pyridine (4). A mixture of triazine **2b** (266 mg, 1 mmol) and 1-morpholinocyclopentene (0.79 ml, 5 mmol) was stirred at 200°C under argon atmosphere for 8 h. The product was isolated from the obtained mixture by column chromatography (eluent CH_2Cl_2, R_f 0.5). The solvent was removed from product fractions by evaporation at reduced pressure and the residue was treated with EtOH. The precipitate that formed was filtered off and washed with EtOH. Analytically pure sample was obtained by recrystallization from MeCN. Yield 184 mg (60%), colorless crystals, mp 134–136°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.96–2.06 (2H, m, 6-CH_2); 2.77 (2H, t, *J* = 7.6, 7-CH_2); 3.42 (2H, t, *J* = 7.6, 5-CH_2); 3.95 (3H, s, OCH_3); 7.22–7.42 (6H, m, H Ph, H-5 Ph); 7.82 (1H, td, *J* = 7.6, *J* = 1.6, H-4 Py); 8.34 (1H, d, *J* = 8.0, H-3 Py); 8.62 (1H, d, *J* = 4.0, H-6 Py). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 26.2; 32.4; 32.9; 53.7; 121.1; 122.4; 122.8; 127.3; 128.1; 129.8; 132.9; 135.8; 136.3; 146.0; 148.5; 157.3; 158.3; 159.0. Mass spectrum, *m*/*z* (I_{rel}, %):

303 $[M+H]^+$ (100). Found, %: C 79.32; H 6.11; N 9.34. $C_{20}H_{18}N_2O$. Calculated, %: C 79.44; H 6.00; N 9.26.

Supplementary information file containing absorption and emission spectra for luminophores **3** and **4** is available at the journal website at http://link.springer.com/journal/10593.

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