1. Introduction

Terpyridines have attracted strong research interest due to their potential applications in coordination chemistry [1], asymmetric catalysis [2], chemo-therapeutics [3] and supramolecular chemistry [4]. Their distinct photophysical characteristics create the possibility to design new functional materials such as light-emitting devices [5]. In particular 4'-aryl-substituted terpyridines are known to possess interesting fluorescent properties and can be used in oligonucleotide derivatization [6], construction of photo- and redox-active complexes [7] and in synthesis of fluorescent-labeled proteins and peptides [8]. Frequently, a fine-tuning of terpyridines fluorescence color induced by chemical modification is required for a better performance of photofunctional systems [9]. On the other hand, tuning fluorophores to desirable photophysical properties is still a difficult task and structural alteration can easily modify their emission properties. Anyhow, the introduction of different aromatic substituents at 4'-position of the terpyridine unit can lead to quite dramatic effects on the fluorescence intensities and, in some cases, the wavelength of emission [10]. Variation of the emissive properties can also be achieved by symmetrical substitution at positions 6,6” of the terpyridine ring but, to the best of our knowledge, no extensive investigation of photophysical properties for these type of compounds has been done so far. In addition, maintaining the same aromatic unit in the position 4’ and inserting either electron withdrawing groups (F, Cl, Br) or electron donating groups (NH2, CH3, OCH3, and N(CH3)2) at positions 6,6” of the terpyridine ring can modify absorption and emission properties. We therefore became interested in examining the photophysical properties of symmetrically disubstituted terpyridines and, in this contribution, we present the synthesis and UV-Vis and fluorescence properties of a series of known and new 6,6”-symmetrically disubstituted 4'-aryl-terpyridines I (Fig. 1). A study of their absorption and emission properties is detailed herein.

2. Experimental Procedure

Chemicals and solvents of commercial grade were used without further purification. 1H, and 13C NMR spectra were recorded in CDCl3 and DMSO-d6, at room temperature on Bruker Avance300 and Avance400 spectrometer (δ in ppm, J in Hz) at 1H operating frequencies of 300.13 MHz and 400.13 MHz (75 MHz and 100 MHz for 13C); spectra were referenced using the solvent signal as internal standard. The mass spectra (MALDI+) were recorded from CHCl3 solutions on a MALDI-TOF Microflex (Bruker) spectrometer (DCTB as matrix); APCI mass spectra were...
recorded on an Agilent 6320 ion trap mass spectrometer in positive mode. Elemental analyses were carried out on a Perkin-Elmer 2400-B microanalyser. The UV–Vis absorption spectra (230–500 nm) were measured with a Perkin Elmer Lambda 35 UV–Vis spectrophotometer and the luminescence spectra (285–750 nm) were obtained with a Perkin Elmer LS 55 Luminescence apparatus at 20°C. Relative quantum yields were measured with a Kleinfeld melting point apparatus and samples were degassed prior to use. Melting points were performed using PharmPrep 60 CC (40–63 µm) silica gel purchased from Merck. Preparative column chromatography was conducted on silica gel 60 F254 TLC plates purchased from Merck. All solvents used for spectrophotometric measurements were purchased from Merck or Acros and were used without further purification.

Syntheses of 3a [12], 4 [12], 5 [12] and 6a [6] are described elsewhere.

6,6”-dibromo-4’-[4”-(methoxymethyl)phenyl]-2,2’,6’,2”-terpyridine (3b)

2-acyethyl-6-bromopyridine (0.58 g, 2.92 mmol) was added to 50 mL MeOH solution of 4-(hydroxydimethyl) benzaldehyde (0.20 g, 1.46 mmol), NaOH (0.05 g, 1.46 mmol) and 10 mL aq. NH₄OH 25%. The reaction mixture was refluxed for 2 days and then let to reach room temperature. The formed precipitate was filtered and washed with water and cold methanol and the crude product was purified by column chromatography on silica gel (CH₂Cl₂:Petroleum Ether=2:1, Rf = 0.7) to afford 3c. (Yield 0.37 g, 52%) Appearance: white solid, mp. 251–252°C. 1H NMR (300 MHz, CDCl₃) δ ppm: 8.69 (s, 2H, H3’, H5’), 8.58 (dd, 2H, H3, H3”, J = 7.8 Hz, J = 0.6 Hz), 7.86 (d, 2H, H4, H4”, J = 8.1 Hz), 7.71 (t, 2H, H5, H5”, J = 7.8 Hz), 7.51 (overlapped signals, 4H, H1, H2, H1”, H2”), 4.56 (s, 2H, -CH₂-O-), 3.43 (s, 3H, -O-CH₃). 13C NMR (75 MHz, CDCl₃) δ ppm: 156.45, 154.32, 150.38, 141.57, 139.40, 139.11, 137.34, 128.18, 128.15, 127.38, 119.95, 119.67, 74.17, 58.14. MS (MALDI+/DCTB): [M+H]⁺ m/z: 509.9, 511.9, 513.9; [M+Na]⁺ m/z: 531.8, 533.8, 535.8; [M+K]⁺ m/z: 547.8, 549.8, 551.8. Anal. Calcd. for C₂₂H₁₅Br₂N₃O: C, 54.04; H, 3.35; Br, 32.14; N, 8.45; found: C, 53.99; H, 3.31; Br, 31.33; N, 8.25.

4’-(4”-bromophenyl)-2,2’:6’,2”-terpyridine-6,6”-dicarboxylic acid (6b)

A solution of dinitrile 5 (0.70 g, 1.59 mmol) in acetic acid (50 mL) and conc. HCl (20 mL) was refluxed for 16 h. The precipitate formed upon cooling was collected by filtration, washed with water and cold methanol and the crude product was purified by column chromatography on silica gel (AcOEt:Petroleum Ether=1:1, Rf = 0.4) to afford 3b. (Yield 0.37 g, 52%) Appearance: white solid, mp. 251-252°C. 1H NMR (300 MHz, CDCl₃) δ ppm: 8.69 (s, 2H, H3’, H5’), 8.59 (d, 2H, H3, H3”, J = 7.8 Hz), 7.88 (d, 2H, H4, H4”, J = 8.4 Hz), 7.72 (t, 2H, H5, H5”, J = 7.8 Hz), 7.54 (overlapped signals, 4H, H1, H2, H1”, H2”), 4.81 (s, 2H, -CH₂-OH). 13C NMR (75 MHz, CDCl₃) δ ppm: 153.68, 149.74, 142.56, 140.84, 138.55, 136.03, 127.50, 126.66, 126.52, 119.33, 118.83. 63.41. MS (MALDI+/DCTB): [M+H]⁺ m/z: 496.20, 498.20, 500.20; [M+Na]⁺ m/z: 518.20, 520.20, 522.20; [M+K]⁺ m/z: 534.10, 536.10, 538.10. Anal. Calcd. for C₂₀H₁₄Br₃N₅O: C, 53.15; H, 3.04; Br, 32.14; N, 8.45; found: C, 53.19; H, 3.13; Br, 32.05; N, 8.43.

6,6”-dibromo-4’-[4”-(methoxymethyl)phenyl]-2,2’,6’,2”-terpyridine (3c)

2-acyethyl-6-bromopyridine (1.60 g, 8.02 mmol) was added to 120 mL MeOH solution of 4-(bromomethyl) benzaldehyde (0.80 g, 4.01 mmol), NaOH (0.16 g, 4.01 mmol) and 30 mL aq. NH₄OH 25%. The reaction mixture was refluxed for 2 days and then let to reach room temperature. The formed precipitate was filtered and washed with water and cold methanol and the crude product was purified by column chromatography on silica gel (CH₂Cl₂:Petroleum Ether=2:1, Rf = 0.7) to afford 3c. (Yield 1.12 g, 54%) Appearance: white solid, mp. 155°C. 1H NMR (300 MHz, CDCl₃) δ ppm: 8.68 (s, 2H, H3’, H5’), 8.58 (dd, 2H, H3, H3”, J = 7.8 Hz, J = 0.6 Hz), 7.86 (d, 2H, H4, H4”, J = 8.1 Hz), 7.71 (t, 2H, H5, H5”, J = 7.8 Hz), 7.51 (overlapped signals, 4H, H1, H2, H1”, H2”), 4.56 (s, 2H, -CH₂-O-), 3.43 (s, 3H, -O-CH₃). 13C NMR (75 MHz, CDCl₃) δ ppm: 157.16, 154.32, 150.38, 141.57, 139.40, 139.11, 137.34, 128.18, 128.15, 127.38, 119.95, 119.67, 74.17, 58.14. MS (MALDI+/DCTB): [M+H]⁺ m/z: 509.9, 511.9, 513.9; [M+Na]⁺ m/z: 531.8, 533.8, 535.8; [M+K]⁺ m/z: 547.8, 549.8, 551.8. Anal. Calcd. for C₂₂H₁₅Br₂N₃O: C, 54.04; H, 3.35; Br, 31.26; N, 8.82; found: C, 53.99; H, 3.31; Br, 31.33; N, 8.25.

4’-(4”-bromophenyl)-2,2’:6’,2”-terpyridine-6,6”-dicarboxylic acid dimethyl ester (6c)

Thionyl chloride (1.00 mL, 13.76 mmol) was added dropwise to cold methanol (50 mL). After the mixture was stirred for 15 min at room temperature, terpyridine derivative 6b (1.00 g, 2.09 mmol) was added and the mixture refluxed for 5 h. After cooling, the white
precipitate formed was filtered and washed several times with cold ethanol and dried to afford compound 6c (Yield 0.81 g, 78%). Appearance: white solid, mp. 268-269°C. 1H NMR (300 MHz, CDCl₃) δ ppm: 8.84 (d, 2H, H₅, H₅”, 3J = 7.8 Hz), 8.78 (s, 2H, H₃, H₃”), 8.19 (d, 2H, H₃, H₃”, 3J = 7.8 Hz), 7.77 (d, 2H, H₈, H₈”, 3J = 8.7 Hz), 7.67 (d, 2H, H₆, H₆”), 3J = 8.7 Hz), 4.06 (s, 6H, -O-CH₃). 13C NMR (75 MHz, CDCl₃) δ ppm: 165.79, 156.19, 155.23, 149.69, 147.57, 137.92, 137.24, 132.96, 132.13, 129.05, 125.28, 124.62, 119.71, 52.92. MS (MALDI+/DCTB) [M+H]+ m/z: 504.10, 506.10; [M+Na]+ m/z: 526.10, 528.10; [M+K]+ m/z: 542.10, 544.10. Anal. Calcd. for C₂₅H₁₈BrN₃O₄: C, 63.57; H, 3.84; Br, 16.92; N, 8.90; found: C, 63.59; H, 3.84; Br, 16.81; N, 8.99.

4’-(4”’-bromophenyl)-6,6”-bis(hydroxymethyl)-2,2’:6’,2”-terpyridine (6d)
NaBH₄ (0.20 g, 5.28 mmol) was added to a suspension of diester 6c (0.30 g, 0.59 mmol) in absolute ethanol (25 mL) and the mixture was stirred at room temperature for 3 h and then it was refluxed for 1h. After cooling, the solvent was evaporated, saturated aqueous NaHCO₃ (30 mL) was added and the solution was heated to boiling. The cold mixture was filtered off and washed with water to afford 6d (Yield 0.22 g, 88%). Appearance: white solid, mp. 295°C. 1H NMR (300 MHz, DMSO-d₆) δ ppm: 8.67 (s, 2H, H₃’, H₅’), 8.52 (d, 2H, H₃, H₃”, 3J = 5.7 Hz), 8.03 (t, 2H, H₄, H₄”, 3J = 5.7 Hz), 7.87 (d, 2H, H₃”, H₅”, 3J = 6.6 Hz), 7.80 (d, 2H, H₈, H₈”, 3J = 6.6 Hz), 7.59 (d, 2H, H₇, H₇”, 3J = 5.7 Hz), 5.57 (s, 2H, -OH), 4.71 (s, 4H, -CH₂-OH). 13C NMR (75 MHz, DMSO-d₆) δ ppm: 161.64, 155.66, 153.73, 148.11, 137.90, 136.71, 132.15, 128.93, 123.48, 120.52, 119.03, 34.11. MS (MALDI+/DCTB): [M+H]+ m/z: 448.30, 450.30; [M+Na]+ m/z: 470.30, 472.30; [M+K]+ m/z: 486.20, 488.20. Anal. Calcd. for C₂₃H₁₈BrN₃O₂: C, 61.62; H, 4.05; Br, 17.82; N, 9.37; found: C, 61.57; H, 4.13; Br, 17.72; N, 9.33.

6,6”-bis(bromomethyl)-4’-(4”’-bromophenyl)-2,2’:6’,2”-terpyridine (6e)
A mixture of dry DMF (40 mL) and PBr₃ (0.42 mL, 4.44 mmol) was stirred for 15 min at room temperature. The diol 6d (0.49 g, 1.11 mmol) was added and the mixture was heated at 60°C for 1 h and then stirred at room temperature overnight. After neutralization with aqueous NaHCO₃ (saturated, 20 mL), the precipitate was filtered and washed with cold water and acetonitrile. The solid was purified by column chromatography on silica gel (AcOEt:Petroleum Ether=1:3, Rf = 0.66) to afford 6e (Yield 0.42 g, 68%). Appearance: white solid, mp. 211-213°C. 1H NMR (400 MHz, CDCl₃) δ ppm: 8.71 (s, 2H, H₃’, H₅’), 8.58 (dd, 2H, H₃, H₃”, 3J = 7.8 Hz, 4J = 0.9 Hz), 7.88 (t, 2H, H₄, H₄”, 3J = 7.8 Hz); 7.77 (d, 2H, H₇, H₇”, 3J = 5.7 Hz), 5.52 (dd, 2H, H₈, H₈”, 3J = 5.7 Hz, 4J = 0.9 Hz), 4.68 (s, 4H, -CH₂-Br). 13C NMR (100 MHz, CDCl₃) δ ppm: 156.32, 155.71, 155.61, 149.18, 137.90, 137.58, 132.20, 128.93, 123.67, 123.48, 120.52, 119.03, 34.11. MS (MALDI+/DCTB): [M+H]+ m/z: 571.90, 573.80, 575.90, 577.9; [M+Na]+ m/z: 593.80, 595.80, 597.80, 599.80. Anal. Calcd. for C₂₃H₁₆Br₃N₃: C, 48.12; H, 2.81; Br, 41.75; N, 7.32; found: C, 48.31; H, 2.87; Br, 41.67; N, 7.31.

3. Results and Discussions
Our initial approach focused on the synthesis of different 6,6” symmetrically substituted 4’-aryl-2,2’:6’,2”-terpyridine substrates. Terpyridines 3a-c were easily obtained, in fair to good yields, using typical procedures [11] (Scheme 1). Condensation of aromatic aldehyde 1a with 2-acetylpyridine 2a and aldehyde 1b with 2-acetyl-6-bromopyridine 2b afforded 3a and 3b, respectively. When 1c was treated with 2b in the presence of methanol and NaOH, methoxymethyl substituted terpyridine 3c was obtained. Attaching different functional groups, like Br (3a), CH₂OH (3b) and CH₂OCH₃ (3c), to the phenyl group at position 4’ can not only open many pathways toward the construction of advanced supramolecular
structures, but may also affect the emission properties of these compounds.

Terpyridine 3a was further functionalized (Scheme 2) to symmetrically substituted derivatives by oxidation with m-CPBA to give N,N'-dioxide intermediate 4 [6]. Reduction of dinitrile 5 with diborane, afforded the bis(aminomethyl) derivative 6a, isolated as its hydrochloride salt [6]. Hydrolysis of 5 to carboxylic derivative 6b was performed in acetic acid in a very good 92% yield. Esterification of acid 6b with methanol allowed the preparation of the methyl ester 6c in 78% yield, which was further reduced with NaBH4 in dry ethanol to afford 6d in 88% yield. Bromomethyl substituted phenyl terpyridine 6e was obtained in 68% yield by treatment of 6d with PBr3 in anhydrous DMF.

With a diverse class of terpyridines in hand, we next investigated their photophysical properties. The absorption spectra of the phenyl substituted terpyridines, recorded in acetonitrile and dichloromethane, are shown in Fig. 2. Absorption spectra suggest some differences in the electronic band structures of 3a-6e in acetonitrile and dichloromethane. In the spectra recorded in acetonitrile, intense broad absorption bands are present in the areas 252-262 nm and 275-289 nm, respectively, and they are associated with π-π* transitions of the terpyridines. Generally, when compared to the reference compound 3a, the maximum absorption bands of 3b-6e are slightly shifted by appending either electron donating or electron withdrawing groups at the peripheral pyridines. The longest wavelengths of absorption in the UV-Vis spectrum of 3b (λmax = 255 and 289 nm) are broad and comparable in intensity (log ε = 4.41 and 4.51, respectively) to that of the parent compound 3a (Table 1); a bathochromic shift of 13 nm is observed. The introduction of CH2OCH3 group in 3c decreases the molar absorptivity (log ε = 4.40 and 4.47) with absorption maxima similar to those observed for 3b and not much different than those of 3a. Interesting changes are noted when the absorption spectrum of 3a is compared with that of its N-oxide 4. Absorption maxima in these spectra show a slight hypsochromic shift (1-2 nm) and a decrease of the molecular extinction coefficient (e.g., for the longest wavelength absorption band at 275 nm from 4.54 in 3a to 4.14 in 4) is observed. Further insertion of CN groups in 5 caused only minor changes of the molecular extinction coefficients, as well as a slight bathochromic shift for the longest wavelength of absorption.

The absorption maximum of the right shoulder of 6a was slightly red-shifted along with a significant decrease of the log ε down to 3.29, probably due to

Scheme 2. Reagents and conditions: i) m-CPBA, CH2Cl2, r.t.; ii) (CH3)3SiCN, CH3COCl, CH2Cl2, r.t.; iii) BH3 • THF, THF, HCl, 16h, (6a); iv) SOCl2, MeOH, Δ, 5h, (6b); v) NaBH4, EtOH, (6d); vi) DMF, PBr3, (6e).

Table 1. Absorption and emission details of terpyridines 3a-6e in acetonitrile and dichloromethane.

| Compound | λmax (nm) (log10 ε) ACN | λem (nm)Φ ACNa | λmax (nm) (log10 ε) DCM | λem (nm)Φ DCM |
|----------|------------------------|---------------|------------------------|---------------|
| 3a       | 254 (4.50), 276 (4.54) | 356 0.17      | 255 (4.52), 278 (4.59) | 358 0.24      |
| 3b       | 255 (4.41), 289 (4.51) | 355 0.15      | 256 (3.48), 290 (3.57) | 357 0.20      |
| 3c       | 254 (4.40), 288 (4.47) | 356 0.11      | 256 (4.35), 290 (4.44) | 357 0.15      |
| 4        | 252 (4.21), 275 (4.14) | 353 0.02      | 255 (4.53), 278 (4.60) | 357 0.06      |
| 5        | 259 (4.54), 283 (4.45) | 353 0.16      | 262 (4.51), 285 (4.44) | 354 0.22      |
| 6a       | 263 (3.29), 282 (3.32) | 354 0.08      | 258 (3.59), 284 (3.63) | 359 0.08      |
| 6b       | 260 (4.20), 282 (4.09) | 353 0.10      | 262 (4.47), 284 (4.40) | 361 0.16      |
| 6c       | 259 (4.63), 280 (4.51) | 355 0.19      | 262 (4.62), 284 (4.49) | 362 0.43      |
| 6d       | 254 (4.30), 282 (4.33) | 358 0.26      | 255 (4.34), 284 (4.44) | 361 0.23      |
| 6e       | 262 (4.67), 281 (4.61) | 357 0.04      | 260 (3.62), 285 (3.58) | 358 0.09      |

*Relative quantum yields were determined by using 2-aminopyridine (Φ = 0.37, excitation at 285 nm, in ethanol) as standard compound.
The fluorescence properties of the synthesized terpyridines were also investigated in different solvents; details of emission spectra, recorded at various concentrations, are given in Fig. 2 and details of the emission maxima and fluorescence quantum yields ($\Phi$) are shown in Table 1.

The maximum emission band of compounds 3a-6e was located at about 353-358 nm when spectra were taken in acetonitrile, their fluorescence quantum yield being affected by the nature of the functional groups at the peripheral pyridine, while the emission profile was similar upon excitation at 285 nm. As it can be seen from the data collected in Table 1, the absorption maximum for each compound varies little in both CH$_2$CN and CH$_2$Cl$_2$. Thus, the emission spectrum of 3a consists of one band with a maximum at 356 nm, red-shifted in comparison to 2,2':6',2"-terpyridine, which is known to have a fluorescence maximum at 338 nm [13]. Introduction of CH$_2$OH (3b) or CH$_2$OCH$_3$ (3c) group on the phenyl ring decreases the emission intensity when compared to 3a, with values of the $\Phi$ = 0.15 and 0.11 in acetonitrile. As expected, N-oxide 4 has reduced emission intensity down to 0.02. Dinitrile 5 and compound 6c show a similar $\Phi$ to that of 3a indicating that the photophysical properties are less affected when CN or COOCH$_3$ groups are inserted, the intensity of emission of aminomethyl, carboxy and bromomethyl substituted terpyridines 6a, 6b and 6e is significantly reduced, while the alcohol 6d has an increased quantum yield when compared to 3a. As can be seen from the data collected in Table 1, when the emission spectra were recorded in dichloromethane the quantum yield of the terpyridine 3a increased to 0.24. Moreover, compounds which are modified by appending either electron-donating or withdrawing groups (3b, 3c, 4, 5, 6b and 6e) also show higher efficiencies in comparison to the fluorescence quantum yields registered in acetonitrile. The values observed for 6a and 6d remain practically unchanged. In addition, the fluorescence intensity of compound 6c increased twice in comparison to the value measured in acetonitrile ($\Phi=0.43$).

Given the moderate value for the fluorescence quantum yield of 6c in dichloromethane, we have tried to find other solvents to obtain higher fluorescence quantum yields for this compound, but fluorescence spectra registered in the low-solubilizing non-polar cyclohexane and the protic polar ethanol used as solvents showed a poorer fluorescence quantum yield for compound 6c ($\Phi=0.28$ and $\Phi=0.16$, respectively).

The excitation spectra of compounds 3a-6e were similar to the corresponding absorption spectra, showing that the emitting state is the lowest excited state.

![Figure 2](image-url)
4. Conclusions

New 6,6" symmetrically substituted 4'-aryl-2,2':6',2"-terpyridine substrates have been prepared and their UV-Vis and fluorescence spectra have been analyzed. We have noticed that the introduction of either electron donating or electron withdrawing groups modified the molar absorptivity as compared to terpyridine 3a, and that most compounds showed red-shifted and differently shaped absorption spectra in comparison with the parent compound 3a. Our results show also that the emission maximum wavelength of this class of compounds is only slightly modified by chemical change. Measurement of fluorescence quantum yield in acetonitrile and dichloromethane lead to low values ($\Phi=0.02-0.26$) with the exception of the moderate value obtained for compound 6c ($\Phi=0.43$) in dichloromethane.

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