

THE SYNTHETIC USE OF 2-(4'-ACETYLBIPHENYL-4-YL)-5-PHENYL-1,3,4-OXADIAZOLE FOR PREPARATION OF NEW HETEROCYCLIC LUMINOPHORES

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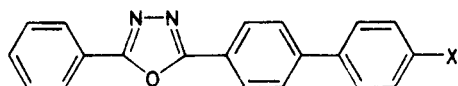
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Dedicated to Professor Miroslav Ferles on the occasion of his 70th birthday.

2-(4'-Acetylbiphenyl-4-yl)-5-phenyl-1,3,4-oxadiazole (*I*) was converted into α,β -unsaturated ketone *II* and pyridinium salt *IV*, which were used in preparation of triarylpyridines *XIII*–*XIX* by Kröhnke's method. Also the diketones *V*–*VIII* were obtained from derivative *I* and served as precursors for the synthesis of pyrazoles *IX*–*XII*. The discussion of ^1H NMR spectra and luminescent properties of prepared bifluorophoric heterocyclic systems is given.

The regioselective electrophilic 4'-substitution¹ of wellknown luminophore 2-(biphenyl-4-yl)-5-phenyl-1,3,4-oxadiazole (PBD, ref.²) opened the new ways to the syntheses of compounds with interesting optical properties. One possibility is the preparation of heterocyclic bifluorophores³ containing PBD substituent. In connec-



PBD, X = H

I, X = CO-CH₃

II, X = CO-CH=CH-Ph

III, X = CO-CH₂-Br

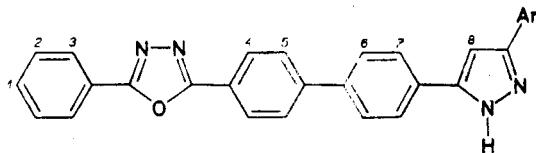
IV, X = CO-CH₂-N⁽⁺⁾(C₆H₅) Br⁽⁻⁾

V, X = CO-CH₂-CO-Ph

VI, X = CO-CH₂-CO-2-pyridyl

VII, X = CO-CH₂-CO-3-pyridyl

VIII, X = CO-CH₂-CO-4-pyridyl



IX, Ar = phenyl

X, Ar = 2-pyridyl

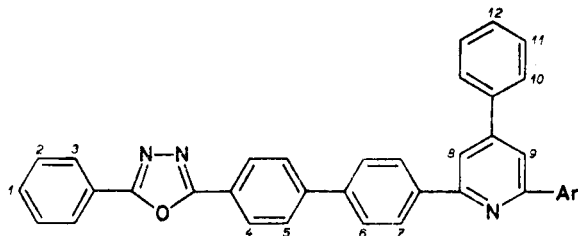
XI, Ar = 3-pyridyl

XII, Ar = 4-pyridyl

tion with our previous synthesis⁴ of fluorescent molecules we have checked the use of acetyl derivative *I* for the synthesis of bifluorophoric compounds composed of PBD and 2,4,6-triarylpyridine moieties.

2-(4'-Acetylbiphenyl-4-yl)-5-phenyl-1,3,4-oxadiazole (*I*) was prepared by acylation¹ of PBD with acetyl chloride in the presence of AlCl_3 . Acetyl derivative *I* yielded with benzaldehyde in the presence of a base chalcone *II*. Bromoacetyl derivative *III* was prepared both by direct bromoacetylation of PBD with bromoacetyl bromide¹, and by bromination of compound *I* in dioxane. Compound *III* yielded pyridinium salt *IV* by heating with pyridine.

1,3-Diaryl-1,3-propanediones *V*–*VIII* were obtained by the reaction of acetyl derivative *I* with ethylesters of corresponding acids and potassium tert-butoxide. These diketones were transformed to pyrazoles *IX*–*XII* by heating with hydrazine hydrate. Yields and the physical and analytical properties of new prepared compounds are given in Table I.



XIII, Ar = phenyl

XIV, Ar = biphenyl-4-yl

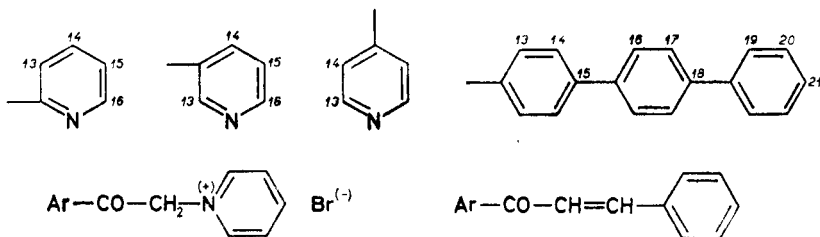
XV, Ar = *p*-terphenyl-4-yl

XVI, Ar = PBD

XVII, Ar = 2-pyridyl

XVIII, Ar = 3-pyridyl

XIX, Ar = 4-pyridyl



XXa, Ar = phenyl

XXb, Ar = biphenyl-4-yl

XXc, Ar = *p*-terphenyl-4-yl

XXI a, Ar = phenyl

XXI b, Ar = biphenyl-4-yl

XXI c, Ar = *p*-terphenyl-4-yl

XXI d, Ar = 2-pyridyl

XXI e, Ar = 3-pyridyl

XXI f, Ar = 4-pyridyl

Cinnamoyl derivative *II* and pyridinium bromide *IV* were used together with chalcones *XXI* and pyridinium salts *XX* for the Kröhnke's synthesis^{1,5} of 2,4,6-triarylpyridines *XIII–XIX*. The combination of starting materials, along with the yields of reactions, the physical and analytical characteristics of synthesized pyridines are given in Table II.

The structures of prepared compounds are in agreement with the results of elemental analyses and their spectral properties. ¹H NMR spectra were interpreted with the help of previous study of analogous 1,3,4-oxadiazoles¹ and 2,4,6-triarylpyridines⁴. The NMR data of carbonyl compounds *II*, *IV–VIII* are collected in Table III. The diketones *V–VIII* were found to exist only as enols in DMSO solution at 20°C.

TABLE I
Analytical data for compounds *II*, *IV–XII*

| Compound | Formula M.w. | Yield, % M.p., °C | IR spectrum cm ⁻¹ | Calculated/Found | | |
|-------------|---|------------------------|---------------------------------|------------------|------|-------------------|
| | | | | % C | % H | % N |
| <i>II</i> | C ₂₉ H ₂₀ N ₂ O ₂ | 83 | 1 695 s, 1 610 s | 81.28 | 4.71 | 6.54 |
| | 428.5 | 218–219 ^a | | 81.28 | 4.79 | 6.69 |
| <i>IV</i> | C ₂₇ H ₂₀ BrN ₃ O ₂ | 93 | 1 680 s, 1 602 s | 65.06 | 4.05 | 8.43 ^b |
| | 498.4 | 260–263.5 ^c | | 65.37 | 4.16 | 8.26 |
| <i>V</i> | C ₂₉ H ₂₀ N ₂ O ₃ | 38 | 3 450 s, 1 678 s | 78.35 | 4.54 | 6.30 |
| | 444.5 | 238–240 | 1 608 s, 1 546 s | 78.22 | 4.49 | 6.42 |
| <i>VI</i> | C ₂₈ H ₁₉ N ₃ O ₃ | 30 | 3 450 s, 1 610 s | 75.48 | 4.31 | 9.43 |
| | 445.5 | 225–226 | 1 550 s | 75.39 | 4.40 | 9.35 |
| <i>VII</i> | C ₂₈ H ₁₉ N ₃ O ₃ | 20 | 3 450 s, 1 680 sh | 75.48 | 4.31 | 9.43 |
| | 445.5 | 218–220 | 1 608 s, 1 548 s | 75.28 | 4.52 | 9.16 |
| <i>VIII</i> | C ₂₈ H ₁₉ N ₃ O ₃ | 20 | 3 450 s, 1 680 sh | 75.48 | 4.31 | 9.43 |
| | 445.5 | 239–241 | 1 605 s, 1 548 s | 75.56 | 4.42 | 9.28 |
| <i>IX</i> | C ₂₉ H ₂₁ N ₄ O | 67 | 1 610 s, 1 550 s | 79.06 | 4.59 | 12.72 |
| | 440.5 | 176–180 | | 79.77 | 4.36 | 12.38 |
| <i>X</i> | C ₂₈ H ₁₉ N ₅ O | 67 | 1 610 s, 1 548 s | 76.16 | 4.35 | 15.78 |
| | 441.5 | 284–286 | | 76.45 | 4.24 | 15.50 |
| <i>XI</i> | C ₂₈ H ₁₉ N ₅ O | 50 | 1 610 s, 1 545 s | 76.16 | 4.35 | 15.78 |
| | 441.5 | 262–265 | | 67.58 | 4.20 | 15.99 |
| <i>XII</i> | C ₂₈ H ₁₉ N ₅ O | 67 | 1 607 s, 1 548 s | 76.16 | 4.35 | 15.78 |
| | 441.5 | 321–323 | | 76.61 | 4.05 | 15.40 |

^a Dioxane-methanol; ^b % Br calculated/found: 16.03/15.78; ^c methanol.

Chemical shifts of protons in PBD moiety of pyrazoles are collected in Table IV. The signals for N—H group of pyrazole ring were not found under experimental conditions.

¹H NMR spectra of triarylpyridines *XIII*–*XIX* are formed by AA'BB' or AA'XX' systems. Owing to the free rotation of *p*-phenylene rings their signals were simply interpreted as doublets. The chemical shift is dependent on the distance from pyridine and/or oxadiazole ring. The most downfield shifted signals are the doublets of *ortho* positions H-7, H-4 and H-13. With exception of compounds *XV* and *XVI* the signals of H-8 and H-9 are visible as two singlets. The unusual downfield shift of proton H-9 in compound *XVII* is caused by the proximity of 2-pyridyl nitrogen atom. The most upfield signals are the multiplets of *meta* and *para* protons H-1, H-2, H-11, H-12 which are actually independent on the substitution. The full assignment of ¹H NMR signals of compounds *XIII*–*XIX* is given in Table V.

The luminiscent characteristics of pyridines *XIII*–*XIX* are given in Table VI. The absorption maxima are independent (with exception of compound *XIII*) on

TABLE II
Analytical data for triarylpyridines *XIII*–*XIX*

| Compound | Formula M.w. | Yield, % M.p., °C | IR spectrum cm ⁻¹ | Calculated/Found | | |
|--------------|--|------------------------|---------------------------------|------------------|------|-------------------|
| | | | | % C | % H | % N |
| <i>XIII</i> | C ₃₇ H ₂₅ N ₃ O 527.6 | 37 | 1 610 s, 1 595 s | 84.22 | 4.78 | 7.97 |
| | | 204–205 ^a | 1 548 s | 85.41 | 4.91 | 7.94 |
| <i>XIV</i> | C ₄₃ H ₂₉ N ₃ O 603.7 | 70 | 1 610 s, 1 595 s | 85.54 | 4.85 | 6.96 |
| | | 240–243 ^a | 1 542 s | 85.71 | 5.02 | 6.85 |
| <i>XV</i> | C ₄₉ H ₃₃ N ₃ O 679.8 | 25 | 1 612 s, 1 596 s | 86.56 | 4.90 | 6.18 |
| | | 316–317 ^a | 1 547 s | 86.77 | 5.08 | 6.22 |
| <i>XVI</i> | C ₃₁ H ₃₃ N ₃ O ₂ 747.9 | 10 | 1 610 s, 1 596 s | 81.90 | 4.47 | 9.37 ^c |
| | | 285–288 ^b | 1 548 s | | | |
| <i>XVII</i> | C ₃₆ H ₂₄ N ₄ O 528.6 | 12 | 1 604 s, 1 588 s | 81.79 | 4.59 | 10.60 |
| | | 212–212.5 ^d | 1 548 s | 81.90 | 4.80 | 10.54 |
| <i>XVIII</i> | C ₃₆ H ₂₄ N ₄ O 528.6 | 41 | 1 605 m, 1 595 s | 81.79 | 4.59 | 10.60 |
| | | 220–223 ^d | 1 548 s | 81.94 | 4.92 | 10.40 |
| <i>XIX</i> | C ₃₆ H ₂₄ N ₄ O 528.6 | 33 | 1 605 s, 1 595 s | 81.79 | 4.59 | 10.60 |
| | | 242.5–244 ^e | 1 548 s | 81.50 | 4.83 | 10.70 |

^a Benzene–methanol; ^b CHCl₃–benzene; ^c mass spectrum, *m/z*: 746 (M-1); ^d methanol–CHCl₃; ^e acetone–ethanol.

substitution. The same we could say about fluorescent maxima and quantum yields of fluorescence.

EXPERIMENTAL

The melting points were determined on a Boetius block and are uncorrected. IR spectra (ν , cm^{-1}) were measured in KBr on a Perkin-Elmer 325. ^1H NMR spectra were recorded on a Bruker AM 400 (400 MHz) with tetramethylsilane as internal standard. Fluorescent spectra were determined on a Perkin-Elmer MPF 44B. For the quantum yield determinations solutions of absorbance not higher than 0.01 were prepared. The spectra were corrected manually with regard to the instrument parameters. *p*-Terphenyl was selected as standard with reported⁶ quantum yield ϕ 0.88 (cyclohexane) and ϕ 0.93 (toluene). The course of reaction was checked by TLC on Silufol foils (Kavalier, Votice).

The starting chalcones *XXIa*–*XXIj* were obtained by known methods^{4,7–9} from acetyl derivatives. Pyridinium salts *XXa*–*XXc* were prepared from corresponding bromoacetyl derivatives^{4,10,11}.

TABLE III

^1H NMR spectra (δ , ppm; J , Hz; CDCl_3 , 20°C) of compounds *II*, *IV*–*VIII*

| Compound | H-1, H-2 | H-3 | H-4 | H-5 | H-6 | H-7 |
|--------------------------|----------|--------|----------------------|----------------------|----------------------|----------------------|
| <i>II</i> ^a | 7.56 m | 8.17 m | 8.25 d $J = 8.4$ | 7.82 d $J = 8.5$ | 7.79 m $J = 8.4$ | 8.16 d $J = 8.2$ |
| <i>IV</i> ^b | 7.71 m | 8.25 m | 8.32 d $J = 8.40$ | 8.15 d $J = 8.37$ | 8.16 d $J = 8.47$ | 8.37 d $J = 8.45$ |
| <i>V</i> ^c | 7.57 m | 8.18 m | 8.26 d $J = 8.66$ | 7.83 d $J = 8.68$ | 7.79 d $J = 8.59$ | 8.12 d $J = 8.55$ |
| <i>VI</i> ^d | 7.56 m | 8.16 m | 8.22 d $J = 8.19$ | 7.80 d $J = 8.24$ | 7.75 d $J = 8.23$ | 8.16 m |
| <i>VII</i> ^e | 7.56 m | 8.16 m | 8.24 d $J = 8.22$ | 7.80 m | 7.80 m | 8.11 d $J = 8.22$ |
| <i>VIII</i> ^f | 7.57 m | 8.18 m | 8.27 d $J = 8.35$ | 7.83 m | 7.83 m | 8.14 d $J = 8.27$ |

^a 7.59 d, $J = 15.6$ (COCH=), 7.87 d, $J = 15.7$ (=CH), 7.67 dd, $J = 6.5$ (o), 7.44 m, (*m*, *p*);

^b in $(\text{CD}_3)_2\text{SO}$, 6.94 s (COCH₂N), 9.40 d, $J = 5.63$ (pyr. H-2), 8.48 m (pyr. H-3), 8.94 t, $J = 7.84$ (pyr. H-4); ^c 6.92 s (COCH=C), 8.02 d, $J = 8.35$ (o), 7.52 m, (*m*, *p*), 16.95 s (C=COH); ^d 7.62 s (COCH=C), 7.87 t (pyr. H-3), 8.16 m (pyr. H-4), 7.46 m (pyr. H-5), 8.74 d (pyr. H-6), 16.51 s (C=COH); ^e 6.90 s (COCH=C), 9.22 s (pyr. H-2), 8.28 m (pyr. H-4), 7.44 m (pyr. H-5), 8.78 d (pyr. H-6), 16.65 s (C=COH); ^f 6.95 s (COCH=C), 8.83 d, $J = 5.52$ (pyr. H-2), 7.83 m (pyr. H-3), 16.57 s (C=COH).

2-(4'-Cinnamoylbiphenyl-4-yl)-5-phenyl-1,3,4-oxadiazole (*II*)

Methanolic solution of KOH (10 ml, 25%) was dropped to the solution of 4 g acetyl derivative *I* and 16 ml of benzaldehyde in 200 ml dioxane under reflux. After 4 h boiling the reaction mixture was cooled to 20°C, poured into 200 ml methanol and precipitate was collected by suction. After crystallization from dioxane it was obtained 4.17 g of product *II* (83%).

2-(4'-Bromoacetylbiphenyl-4-yl)-5-phenyl-1,3,4-oxadiazole (*III*)

Bromine (1.41 g) was added to the solution of 3 g compound *II* in 200 ml dioxane and reaction mixture was then stirred at 20°C for 2 h. After pouring into 300 ml water the solid matter was filtered and crystallized from the mixture acetone-toluene to yield 3.70 g (98%) of product *III*.

Preparation of Pyridinium Salt *IV*

A mixture of 1 g bromoacetyl derivative *III* and 5 ml pyridine was refluxed for 3 h. Precipitate was filtered, washed with ether and crystallized from methanol to give 1.10 g (93%) of product *IV*.

1-(4'-(5-Phenyl-1,3,4-oxadiazol-2-yl)-biphenyl-4-yl)-3-aryl-1,3-propanedione (*V*–*VIII*)

Potassium tert-butoxide (0.66 g, 5.9 mmol) was added to the solution of 2 g compound *I* (5.9 mmol) and 5.9 mmol of corresponding acid ethylester in 90 ml dry dioxane and reaction mixture was refluxed for 2–20 h. The disappearance of compound *I* was followed by TLC. After decomposition with 150 ml water the solution was adjusted to pH 5 with 50% acetic acid. Precipitate was filtered off, washed twice with 30 ml methanol and 30 ml ether. The residue was chromatographed on SiO₂ column with eluent CHCl₃-acetone 20:1. After evaporation of solvent the solid was twice crystallized from toluene-dioxane mixture (Table I).

TABLE IV

¹H NMR spectra (δ , ppm; *J*, Hz; (CD₃)₂NCDO 20°C) of compounds *IX*–*XII*

| Compound | H-1, H-2 | H-3 | H-4 | H-5 | H-6 | H-7 | H-8 |
|--------------------------|----------|--------|---------------------------|---------------------------|---------------------------|---------------------------|--------|
| <i>IX</i> ^{a,b} | 7.55 m | 8.18 m | 8.25 d <i>J</i> = 8.11 | 7.77 m | 7.77 m | 8.18 m | |
| <i>X</i> ^c | 7.65 m | 8.20 m | 8.27 d <i>J</i> = 8.37 | 8.02 d <i>J</i> = 8.42 | 7.90 d <i>J</i> = 8.18 | 8.07 d <i>J</i> = 8.15 | 7.38 s |
| <i>XI</i> ^{d,e} | 7.65 m | 8.15 d | 8.22 d <i>J</i> = 7.99 | 7.98 m | 7.94 d <i>J</i> = 7.89 | 7.98 m | 7.31 s |
| <i>XII</i> ^f | 7.66 m | 8.20 m | 8.27 d <i>J</i> = 8.36 | 8.02 d <i>J</i> = 8.48 | 7.92 d <i>J</i> = 8.22 | 8.06 d <i>J</i> = 8.22 | 7.40 s |

^a Other signals were not recognized due to the strong signal of solvent (low solubility); ^b CDCl₃;

^c 7.86 m (pyr. H-3), 8.02 m (pyr. H-4), 7.32 m (pyr. H-5), 8.64 d, *J* = 4.66 (pyr. H-6); ^d 9.08 s (pyr. H-2), 8.22 m (pyr. H-4), 7.47 m (pyr. H-5), 8.52 d, *J* = 3.64 (pyr. H-6); ^e (CD₃)₂SO, 80°C; ^f 8.64 d, *J* = 5.31 (pyr. H-2), 7.86 d, *J* = 5.58 (pyr. H-3).

3(5)-Aryl-5(3)-(4'-(5-phenyl-1,3,4-oxadiazol-2-yl)-biphenyl-4-yl)pyrazoles (IX—XII)

A solution of 67.0 mmol diketone V—VIII and 0.5 ml 80% hydrazine hydrate in 30 ml dioxane was refluxed for 10 h and then 20 ml dioxane were distilled off. After cooling to the room temperature separated solid was collected by filtration and three times crystallized from toluene-dioxane-mixture (Table I).

Preparation of 2,4,6-Triarylpyridines XII—XIX

A mixture of 1.0 mmol chalcone XXI and 1.5 mmol corresponding pyridinium salt XX was refluxed in a mixture DMF-acetic acid in the presence of 2 g ammonium acetate. After the

TABLE V
¹H NMR spectra (δ , ppm; J , Hz; (CD₃)₂SO 20°C) of triarylpyridines XIII—XIX

| Position | XIII | XIV ^a | XV ^b | XVI | XVII ^c | XVIII ^c | XIX ^c |
|----------|----------------------|----------------------|---------------------|---------------------|----------------------|----------------------|----------------------|
| 1, 2 | 7.66 m | 7.63 m | 7.64 m | 7.64 m | 7.55 m | 7.55 m | 7.56 m |
| 3 | 8.17 m | 8.13 m | 8.15 m | 8.14 m | 8.16 m | 8.17 m | 8.17 m |
| 4 | 8.28 d $J = 7.91$ | 8.23 d $J = 8.24$ | 8.25 d $J = 8.3$ | 8.24 d $J = 8.4$ | 8.23 d $J = 8.35$ | 8.24 d $J = 8.41$ | 8.24 d $J = 8.59$ |
| 5 | 7.98 d $J = 8.36$ | 7.94 d $J = 8.44$ | 7.97 d $J = 8.3$ | 7.96 d $J = 8.1$ | 7.83 d $J = 8.30$ | 7.85 d $J = 8.50$ | 7.83 m |
| 6 | 8.06 d $J = 8.42$ | 8.01 m | 8.03 d $J = 8.4$ | 8.02 d $J = 8.2$ | 7.81 d $J = 8.40$ | 7.82 d $J = 8.44$ | 7.83 m |
| 7 | 8.50 d $J = 8.41$ | 8.45 d $J = 8.16$ | 8.48 d $J = 8.4$ | 8.46 d $J = 8.3$ | 8.32 d $J = 8.31$ | 8.32 d $J = 8.38$ | 8.32 d $J = 8.51$ |
| 8 | 8.23 s | 8.187 s ^d | 8.22 s | 8.22 s | 8.03 s | 7.93 s ^d | 7.97 s ^d |
| 9 | 8.25 s | 8.194 s ^d | 8.22 s | 8.22 s | 8.69 s | 8.00 s ^d | 8.03 s ^d |
| 10 | 8.08 d $J = 7.17$ | 8.01 m | 8.03 d $J = 7.1$ | 8.02 d $J = 7.1$ | 7.78 d $J = 8.34$ | 7.78 d $J = 7.68$ | 7.77 d $J = 6.81$ |
| 11, 12 | 7.51 m | 7.57 m | 7.49 m | 7.56 m | 7.55 m | 7.55 m | 7.56 m |
| 13 | 8.36 d $J = 7.32$ | 8.40 d $J = 8.46$ | 8.44 d $J = 8.3$ | — | 8.69 d $J = 7.30$ | 9.42 s | 8.79 d $J = 5.54$ |
| 14 | 7.58 m | 7.83 s $J = 8.42$ | 7.91 d $J = 8.3$ | — | 7.79 m | 8.54 d $J = 7.99$ | 8.11 d $J = 5.35$ |
| 15 | 7.58 m | — | — | — | 7.36 m | 7.47 m | — |
| 16 | — | 7.75 d $J = 7.59$ | 7.87 d $J = 8.4$ | — | 8.73 m | 8.71 d $J = 3.52$ | — |

^a 7.50 t, $J = 8.0$ (H-17), 7.38 t, $J = 7.3$ (H-18); ^b 7.79 d, $J = 8.4$ (H-17), 7.72 d, $J = 8.2$ (H-19), 7.58 m (H-20), 7.37 m (H-21); ^c CDCl₃; ^d signals could be interchanged.

TABLE VI
Luminiscence characteristics of compounds XIII–XIX

| Compound | Absorption ^a | Fluorescence ^a | | |
|--------------------|-------------------------|---------------------------|-----------------------|---------------|
| | | Emission | Emission ^b | Quantum yield |
| XIII ^c | 331 | 386 | 423 | 0.66 |
| XIV ^c | 313 | 372, 385 | 510 | 0.62 |
| XV ^c | 314 | 372 | 441 | 0.63 |
| XVI ^c | 319 | 372, 385 | 479 | 0.60 |
| XVII ^d | 317 | 363, 382 | — | 0.73 |
| XVIII ^d | 320 | 363, 381 | — | 0.69 |
| XIX ^d | 319 | 362, 391 | — | 0.64 |

^a λ_{\max} , nm; ^b in solid state; ^c in toluene; ^d in cyclohexane.

end of reaction (TLC), the reaction mixture was poured into cold water, the precipitate was filtered, washed with water and dried over phosphorus pentoxide in a desiccator. The solid was dissolved in a small amount of CHCl_3 and chromatographed on SiO_2 column with eluent CHCl_3 –benzene. Fractions with the highest R_f values and strong fluorescence (UV light) were collected. For yields, crystallization solvent and melting points see Table II.

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