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# (BENZO[*h*])QUINOLINYL-SUBSTITUTED MONOAZATRIPHENYLENES: SYNTHESIS AND PHOTOPHYSICAL PROPERTIES

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*We propose a method for the synthesis of quinolinyl- and benzo[h]quinolinylmonoazatriphenylenes through 1,2,4-triazine intermediates with subsequent transformations in aza-Diels–Alder reaction. The photophysical properties of these new compounds were examined, and the effects due to additional fused aromatic rings were explored.*

**Keywords:** monoazatriphenylene, phenanthrenequinone, aza-Diels–Alder reaction, heterocyclization, luminescence.

Azatriphenylene derivatives attract significant interest due to their promising photophysical properties, in particular the prolonged lifetime of fluorescent excited state, bathochromic shift of the absorption and emission maxima compared to triphenylene, and the chemical reactivity with respect to the formation of coordination compounds. Azatriphenylenes are widely used for the preparation of photoluminescent *N*,*N*'-metallated [1] and cyclometallated [2] complexes. From biochemical point of view, azatriphenylenes represent interest as neutral intercalating ligands for the structural study of DNA [3, 4]. The presence of 2-azatriphenylene fragments in certain natural compounds [5-7] motivate the search for such compounds with biological activity, as potential anti-inflammatory, antitumor, etc. agents. In the field of materials science, azatriphenylenes offer promise for the design of discotic liquid crystals [8] and supramolecular architectures [9], as well as chemosensors for visual (photoluminescent) detection of aliphatic and aromatic nitro compounds, such as explosives [10].

A particular interest has been attracted by pyridyl-substituted azatriphenylenes, which are 2,2'-bipyridine ligands having annelated polyarene rings, with a wide range of possibilities for further functionalization, but only one method is currently known for the synthesis of these compounds [11]. The possibility of tuning the photophysical properties (the bathochromic shift of absorption and luminescence maxima, and the quantum yield of luminescence) was previously demonstrated in the series of 2,2'-bipyridines by introducing various

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aromatic substituents in the pyridine ring, including the possibility of annelation with additional aromatic rings [12-15], that increased the practical potential of such bipyridines. Besides, the expansion of the conjugated system increases the intermolecular π–π interactions, which are necessary for a range of processes (for example, intermolecular migration of exciton after photoexcitation), while the change of geometry of the key pyridine ligand enables selective binding of certain metal cations (for example, preferential complex formation with $Zn^{2+}$ and $Ca^{2+}$ cations in the presence of $Cd^{2+}$ or selective binding of $Am^{3+}$ cations from solutions of lantanoid ions [16]). In this article, we propose a method for the synthesis of 2,2'-bipyridine type ligands of monoazatriphenylene series with extended conjugated systems involving pyridine rings, namely, containing 2-quinoline and 2-benzo[*h*]quinoline fragments.

In order to obtain pyridylmonoazatriphenylenes, we previously used a method based on the preparation of their 1,2,4-triazine analogs and subsequent aza-Diels–Alder reaction with enamines. This is a well-known method for the synthesis of various pyridine derivatives [17-19], but rarely applied to quinolinylpyridines [20], and no such examples of benzo[*h*]quinolinylpyridine preparation are known. Previous approaches to the synthesis of 3-(quinolin-2-yl)-1,2,4-triazines include the cyclization of isonitrosoacetophenone hydrazones with quinoline-2-carbaldehyde [20], the interaction of quinoline-2-carboxylic acid hydrazide with 1,2-diones followed by reaction with ammonia [21], as well as the interaction of 1,2-diones with quinoline-2-carboxyamidrazone [22]. Besides, the preparation of 3-(quinolin-2-yl)-1,2,4-triazin-5-ones by cyclization of quinoline-2-carboxyamidrazone and glyoxylic acid derivatives is also known [23]. No 3-(benzo[*h*]quinolin-2-yl)-1,2,4-triazines have been described in the literature, and only few methods are known for the preparation of 2-(benzo[*h*]quinolin-2-yl)pyridines. For example, the Friedlander condensation [24], Krönke method [25], as well as the condensation of α-naphthisatin with acetylpyridine derivatives, have been proposed for this purpose [26, 27].

We used the interaction of 9,10-phenanthrenequinone (**1**) and the corresponding amidrazones for the preparation of the necessary 1,2,4-triazine precursors of the target structures. The starting materials for the synthesis of the necessary amidrazones were quinoline *N*-oxide (**2a**) and benzo[*h*]quinoline *N*-oxide (**2b**) [28].

The cyanation reaction of the *N*-oxides **2a**,**b** was performed by a modified literature procedure [29] using trimethylsilyl cyanide and dimethylcarbamoyl chloride. We substituted the dichloromethane solvent reported in the literature [29] with 1,2-dichloroethane. This change of solvent was found to result in a complete conversion of the starting *N*-oxides **2a**,**b** into the nitriles **3a**,**b**, unlike in the previously proposed example.

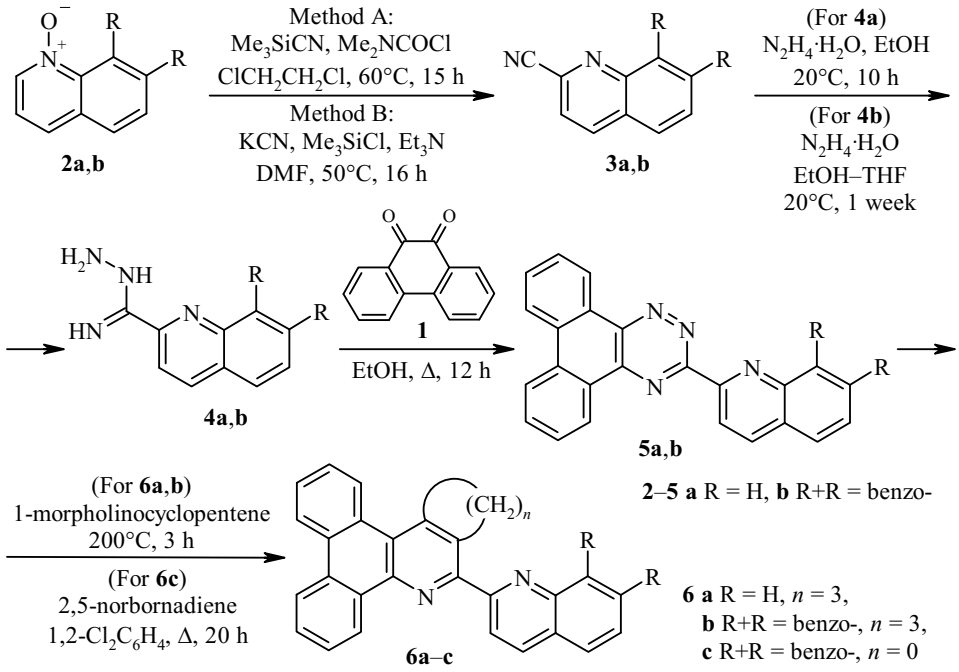
Besides that, we investigated an alternative approach to the preparation of cyano derivatives **3a**,**b**, by using KCN and trimethylsilyl chloride in DMF, with triethylamine (*in situ* preparation of $Me_3SiCN$). It should be noted that these reagents are considerably more affordable than trimethylsilyl cyanide and dimethylcarbamoyl chloride, while the yields of compounds **3a**,**b** were comparable in both cases.

Quinoline-2-carboxyamidrazone **4a** was obtained according to a published method [22]. The corresponding benzo[*h*]quinoline amidrazone **4b** has not been previously described. The method for its synthesis is significantly different from that for 2-cyanopyridine or 2-cyanoquinoline, because the exceedingly slow progress of this reaction requires treatment of 2-cyanobenzo[*h*]quinoline with excess hydrazine hydrate for many days at room temperature. The reaction was performed in a 1:1 mixture of ethanol and THF due to the low solubility of the starting material in ethanol.

The heterocyclization reactions were performed by refluxing a mixture of 9,10-phenanthrenequinone (**1**) and the amidrazones **4a**,**b** in ethanol. Precipitation of compounds **5a**,**b** was observed upon cooling of the reaction mixture, which provided for a convenient isolation of these products.

The aza-Diels–Alder reaction using 1-morpholinocyclopentene as dienophile allowed to prepare the 2,2'-bipyridine type ligands **6a**,**b**, annelated with a cyclopentane ring. The reaction was performed according to an efficient previously described method (heating of 1,2,4-triazine with enamine at 200°C without solvent [30]). Treatment of the reaction mixture with acetonitrile led to the precipitation of compounds **6a**,**b**, which obviated the need for purification by column chromatography. The preparation of monoazatriphenylenes lacking an annelated cyclopentane ring was possible by using 2,5-norbornadiene as dienophile (involving *in situ* formation of aza-Diels–Alder product and subsequent retro process with the elimination of a nitrogen molecule), and was accomplished by

refluxing for many hours in 1,2-dichlorobenzene as a high-boiling solvent. When using the lower-boiling *o*-xylene, the yield of compound **6c** did not exceed 5% even after prolonged refluxing.




Thus, the application of 1-morpholinocyclopentene as a dienophile is preferred, in particular because monoazatriphenylenes with annelated cycloalkane rings typically have a better solubility compared to compounds lacking such a fragment, and therefore have more potential for practical applications.

The structures of the obtained compounds **5a**,**b**, **6a-c** were confirmed by $^{1}$H and $^{13}$C NMR data, mass spectrometry, and elemental analysis.

Within the scope of this project we compared the photophysical characteristics of new monoazatriphenylenes to our previously described 10-(pyridin-2-yl)-12,13-dihydro-11*H*-dibenzo[*f*,*h*]cyclopenta[*c*]quinoline (**6d**) [11]. The luminescence spectra of compounds **6a-d** are presented in Figure 1, and the

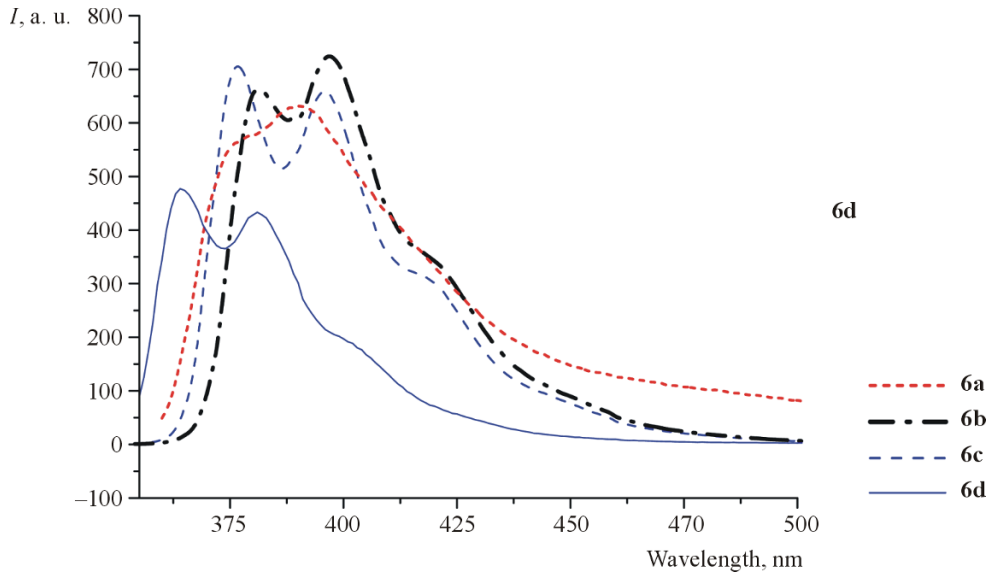



Fig. 1. The fluorescence spectra of monoazatriphenylenes **6a-d**.

TABLE 1. Photophysical Characteristics of Compounds **6a-d**

| Com-pound | Absorption maxima in acetonitrile, nm | Luminescence maximum in acetonitrile, nm | Quantum yield of luminescence* |
|---|---|---|---|
| **6a** | 263, 330, 343, 361 | 389 | 0.031 |
| **6b** | 253, 329, 348, 368 | 381, 397, 421 (sh) | 0.523 |
| **6c** | 251, 332, 348, 368 | 377, 396, 418 (sh) | 0.390 |
| **6d** | 263, 313, 339, 357 | 364, 381, 403 (sh) | 0.213 |

*The quantum yields were determined relative to quinine sulfate ($\Phi$ = 0.546 in 0.1 N aquous $H_2SO_4$ solution [28]) for compounds **6a**,**b**,**d** and 2-aminopyridine ($\Phi$ = 0.60±0.005 in 1 N aqueous $H_2SO_4$ solution [32]) for compound **6c**.

results are listed in Table 1. The annelation of additional aromatic rings to the pyridine fragment led in all cases to a bathochromic shift of absorption and emission maxima. At the same time, the quantum yield of luminescence for compounds **6b**,**c** was significantly increased, compared to the azatriphenylene **6d**, especially in the case of product **6b**. The low quantum yield of compound **6a** was apparently linked to a significant contribution from the non-radiative $n\rightarrow\pi^*$ transition in the excited state, which resulted in a low intensity of photoluminescence [31].

Thus, in the current work we propose convenient methods for the preparation of previously unknown, potentially useful 2,2'-bipyridine type ligands – monoazatriphenylenes, containing quinoline and benzo[*h*]-quinoline fragments at position 2. The extended conjugated system of these compounds may allow the tuning of their properties.

## EXPERIMENTAL

Absorption spectra were recorded on a Shimadzu UV-2401PC spectrometer in acetonitrile. Luminescence spectra were recorded on a Varian Cary Eclipse fluorimeter in acetonitrile. $^1$H and $^{13}$C NMR spectra were acquired on a Bruker Avance II spectrometer (400 and 100 MHz, respectively) in $CDCl_3$, the internal standard was TMS. Mass spectra were recorded on a Bruker Daltonics MicrOTOF-Q II mass spectrometer with electrospray ionization. Elemental analysis was performed with a PerkinElmer PE 2400 II CHN-analyzer. Melting points were determined with a Boetius apparatus. Merck silica gel 60F254 plates were used for TLC, with visualization under UV light.

Quinoline *N*-oxide (**2a**) was purchased from Sigma-Aldrich. Benzo[*h*]quinoline *N*-oxide (**2b**) [28] and quinoline-2-carboxamide hydrazone **4a** [22] were obtained by literature methods.

**Preparation of the Nitriles 3a,b (General Method)**. A. The *N*-oxide **2a**,**b** (7.0 mmol) was dissolved in 1,2-dichloroethane (50 ml). Trimethylsilyl cyanide (0.96 ml, 7.7 mmol) and dimethylcarbamoyl chloride (0.65 ml, 7.0 mmol) were added, and the mixture was stirred for 15 h at 60°C. The reaction mixture was cooled, washed with $Na_2CO_3$ solution, then with $H_2O$, and extracted with $CH_2Cl_2$ (3×50 ml). The extract was dried over anhydrous $Na_2SO_4$, the solvents were removed under vacuum. The residue was treated with $Et_2O$, the precipitate was filtered off and dried. The obtained nitriles **3a**,**b** were used in the next stage without additional purification.

B. The *N*-oxide **2a**,**b** (7.0 mmol) was dissolved in anhydrous DMF (50 ml). Triethylamine (1.07 ml, 7.7 mmol) and KCN (1.37 g, 21.0 mmol) were added, followed by dropwise addition of $Me_3SiCl$ (3.55 ml, 28.0 mmol). The obtained mixture was stirred for 8 h at 50°C. Another portion of KCN (0.91 g, 14.0 mmol) was added, followed by dropwise addition of $Me_3SiCl$ (2.37 ml, 18.7 mmol), the obtained mixture was stirred for another 8 h at 50°C, cooled to room temperature, treated with water (100 ml), and stirred for 1 h. The precipitate formed was filtered off, washed with water, and dried. The obtained nitriles **3a**,**b** were used in the next stage without additional purification.

**Quinoline-2-carbonitrile (3a)**. Yield 0.98 g (91%, method A), 0.82 g (76%, method B), colorless crystals, mp 91-93°C (mp 88-91°C [33]). The spectral data matched the data from the literature [33].

**Benzo[*h*]quinoline-2-carbonitrile (3b)**. Yield 1.21 g (85%, method A), 1.01 g (71%, method B), colorless crystals, mp 162-164°C. $^1$H NMR spectrum, δ, ppm (*J*, Hz): 7.72 (1H, d, $^3J$ = 8.4, H-6); 7.78-7.82 (2H, m); 7.85 (1H, d, $^3J$ = 8.4, H-5); 7.92-7.97 (2H, m); 8.29 (1H, d, $^3J$ = 7.9, H-3); 9.28-9.32 (1H, m, H-4). Mass spectrum, *m*/*z* ($I_{rel}$, %): 205 $[M+H]^+$ (100). Found, %: C 82.16; H 3.87; N 13.55. $C_{14}H_8N_2$. Calculated, %: C 82.34; H 3.95; N 13.72.

**Benzo[*h*]quinoline-2-carboxamide Hydrazone (4b)**. The nitrile **3b** (0.50 g, 2.45 mmol) was dissolved in a 1:1 mixture of EtOH and THF (100 ml). Hydrazine hydrate (1.19 ml, 24.5 mmol) was added, and the obtained mixture was maintained for 1 week at room temperature. The solvents were removed under vacuum, the residue was treated with ether, and the precipitate was filtered off. The compound was used in the next stage without additional purification. Yield 0.52 g (90%). $^1$H NMR spectrum, δ, ppm (*J*, Hz): 5.54 (2H, br. s, $NH_2$); 7.68-7.78 (3H, m); 7.83 (1H, d, $^3J$ = 8.8); 7.91-7.95 (1H, m); 8.18 (1H, d, $^3J$ = 8.4, H-5); 8.27 (1H, d, $^3J$ = 8.4, H-6); 9.29-9.31 (1H, m, H-4). Mass spectrum, *m*/*z* ($I_{rel}$, %): 237 $[M+H]^+$ (100). Found, %: C 70.92; H 4.89; N 23.49. $C_{14}H_{12}N_4$. Calculated, %: C 71.17; H 5.12; N 23.71.

**Preparation of Triazatriphenylenes 5a,b (General Method)**. 9,10-Phenanthrenequinone (**1**) (0.25 g, 1.2 mmol) was dissolved in EtOH (50 ml). A solution of the amidrazone **4a**,**b** (1.44 mmol) in EtOH (30 ml) was added, the mixture was stirred at reflux for 12 h and cooled to room temperature. The precipitate was filtered off, washed with EtOH, and dried.

**3-(Quinolin-2-yl)phenanthro[9,10-*e*][1,2,4]triazine (5a)**. Yield 0.25 g (58%), light-yellow crystals, mp >250°C. $^1$H NMR spectrum, δ, ppm (*J*, Hz): 7.65-7.69 (1H, m); 7.81-7.99 (6H, m); 8.46-8.50 (2H, m); 8.64-8.68 (2H, m); 8.99 (1H, d, $^3J$ = 8.8, H-4 quinoline); 9.58-9.62 (2H, m, H-1,9). $^{13}$C NMR spectrum, δ, ppm: 121.1; 123.1; 125.4; 127.1; 127.2; 127.6; 127.9; 128.2; 128.7; 128.8; 129.1; 130.0; 130.6; 131.0; 131.1; 131.5; 132.5; 132.6; 134.0; 137.2; 148.6; 153.6; 160.7; 161.2. Mass spectrum, *m*/*z* ($I_{rel}$, %): 359 $[M+H]^+$ (100). Found, %: C 80.22; H 3.83; N 15.29. $C_{24}H_{14}N_4$. Calculated, %: C 80.43; H 3.94; N 15.63.

**3-(Benzo[*h*]quinolin-2-yl)phenanthro[9,10-*e*][1,2,4]triazine (5b)**. Yield 0.32 g (66%), light-yellow crystals, mp 255-257°C. $^1$H NMR spectrum, δ, ppm (*J*, Hz): 7.74-7.98 (9H, m); 8.44 (1H, d, $^3J$ = 8.4, H-6 benzoquinoline); 8.61-8.65 (2H, m); 9.10 (1H, d, $^3J$ = 8.4, H-5 benzoquinoline); 9.58-9.62 (2H, m, H-1,8); 9.73 (1H, d, $^3J$ = 8.0, H-4 benzoquinoline). $^{13}$C NMR spectrum, δ, ppm: 122.1; 123.2; 123.3; 125.2; 125.4; 125.5; 127.2; 127.5; 127.6; 127.7; 128.0; 128.2; 128.4; 128.8; 128.9; 129.5; 131.2; 131.6; 132.1; 132.7; 134.0; 134.1; 137.0; 143.6; 145.6; 147.1; 152.0; 161.2. Mass spectrum, *m*/*z* ($I_{rel}$, %): 409 $[M+H]^+$ (100). Found, %: C 82.11; H 3.81; N 13.49. $C_{28}H_{16}N_4$. Calculated, %: C 82.34; H 3.95; N 13.72.

**Preparation of Monoazatriphenylenes 6a,b (General Method)**. A mixture of triazatriphenylene **5a**,**b** (0.70 mmol) and 1-morpholinocyclopentene (0.56 ml, 3.50 mmol) was stirred for 2 h at 200°C under argon atmosphere. Another portion of 1-morpholinocyclopentene (0.28 ml, 1.75 mmol) was then added, and stirred for 1 h under the same conditions. The reaction mixture was cooled to room temperature and acetonitrile (15 ml) was added. The obtained mixture was refluxed for 10 min, the precipitate formed was filtered off, washed with acetonitrile, and dried. A sample for analysis was prepared by recrystallization from acetonitrile.

**10-(Quinolin-2-yl)-12,13-dihydro-11*H*-dibenzo[*f*,*h*]cyclopenta[*c*]quinoline (6a)**. Yield 0.24 g (87%), light-colored crystals, mp 190-192°C. $^1$H NMR spectrum, δ, ppm (*J*, Hz): 2.28-2.32 (2H, m, 12-$CH_2$); 3.78 (2H, t, $^3J$ = 7.2, 11-$CH_2$); 3.85 (2H, t, $^3J$ = 7.2, 13-$CH_2$); 7.88-7.92 (1H, m); 8.19-8.23 (1H, m); 8.34-8.38 (1H, m); 8.60-8.64 (1H, m); 8.67-8.75 (2H, m); 8.89 (1H, d, $^3J$ = 8.4, H-4 quinoline); 9.56-9.60 (1H, m, H-8). $^{13}$C NMR spectrum, δ, ppm: 26.0; 33.6; 37.4; 121.3; 122.4; 123.3; 123.4; 126.1; 126.6; 126.7; 127.2; 127.3; 127.6; 127.7; 127.8; 128.3; 129.2; 129.9; 130.1; 130.8; 131.1; 131.8; 136.0; 139.7; 144.9; 147.7; 150.1; 152.0; 158.6. Mass spectrum, *m*/*z* ($I_{rel}$, %): 397 $[M+H]^+$ (100). Found, %: C 87.81; H 4.94; N 6.87. $C_{29}H_{20}N_2$. Calculated, %: C 87.85; H 5.08; N 7.07.

**10-(Benzo[*h*]quinolin-2-yl)-12,13-dihydro-11*H*-dibenzo[*f*,*h*]cyclopenta[*c*]quinoline (6b)**. Yield 0.27 g (85%), light-colored crystals, mp 235-237°C. $^1$H NMR spectrum, δ, ppm (*J*, Hz): 2.36-2.40 (2H, m, 12-$CH_2$);

3.81 (2H, t, $^3J$ = 7.2, 11-$CH_2$); 4.05 (2H, t, $^3J$ = 7.2, 13-$CH_2$); 7.65-7.88 (8H, m); 7.93-7.97 (1H, m); 8.38 (1H, d, $^3J$ = 8.6, H-6 benzoquinoline); 8.61-8.65 (1H, m); 8.69-8.73 (2H, m); 9.12 (1H, d, $^3J$ = 8.6, H-5 benzoquinoline); 9.44 (1H, d, $^3J$ = 8.0, H-4 benzoquinoline); 9.60-9.62 (1H, m, H-8). $^{13}$C NMR spectrum, δ, ppm: 26.3; 34.4; 37.4; 121.8; 122.4; 123.2; 123.3; 124.9; 125.4; 125.8; 126.2; 126.6; 127.0; 127.2; 127.4; 127.8; 127.9; 128.0; 128.3; 130.2; 130.9; 131.2; 131.9; 132.2; 133.9; 136.0; 139.4; 144.9; 145.7; 147.3; 150.1; 152.1; 157.1. Mass spectrum, *m*/*z* ($I_{rel}$, %): 447 $[M+H]^+$ (100). Found, %: C 88.55; H 4.79; N 6.03. $C_{33}H_{22}N_2$. Calculated, %: C 88.76; H 4.97; N 6.27.

**2-(Benzo[*h*]quinolin-2-yl)dibenzo[*f*,*h*]quinoline (6c)**. The triazatriphenylene **5a** (0.13 g, 0.31 mmol) was suspended in 1,2-dichlorobenzene (30 ml). 2,5-Norbornadiene (0.16 ml, 1.53 mmol) was added, and the mixture was refluxed for 20 h with the addition of 2,5-norbornadiene in portions (0.16 ml, 1.53 mmol) every 5 h. The reaction mixture was cooled to room temperature, the precipitate formed was filtered off, washed with EtOH, and dried. A sample for analysis was obtained by recrystallization from *o*-xylene. Yield 0.27 g (85%), light-colored crystals, mp 273-275°C. $^1$H NMR spectrum, δ, ppm (*J*, Hz): 7.71-7.90 (8H, m); 7.94-7.98 (1H, m); 8.40 (1H, d, $^3J$ = 8.4, H-6 benzoquinoline); 8.65-8.74 (3H, m); 9.10 (1H, d, $^3J$ = 8.8, H-3); 9.17 (1H, d, $^3J$ = 8.4, H-5 benzoquinoline); 9.23 (1H, d, $^3J$ = 8.8, H-4); 9.60 (1H, d, $^3J$ = 8.0, H-4 benzoquinoline); 9.62-9.66 (1H, m, H-12). Mass spectrum, *m*/*z* ($I_{rel}$, %): 407 $[M+H]^+$ (100). Found, %: C 88.52; H 4.28; N 6.71. $C_{30}H_{18}N_2$. Calculated, %: C 88.65; H 4.46; N 6.89.

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