Heterocyclic analogs of pleiadiene.
69.* Synthesis and oxidative hydroxylation of 1-alkyl-1,3-diazapyrenium salts

I. V. Borovlev,[a] O. P. Demidov,[a] A. V. Chernyshev,[b] and A. F. Pozharskii [c]*

[a]Department of Biology and Chemistry, Stavropol State University,
1a ul. Pushkina, 355009 Stavropol, Russian Federation.
Fax:+7 (8652) 35 40 33. E-mail: biochem-org@stavsu.ru
[b]Scientific Research Institute of Physical and Organic Chemistry of Rostov State University,
194/2 ul. Stachka, 344090 Rostov-on-Don, Russian Federation.
Fax: +7 (8632) 28 56 67. E-mail: photo@ipoc.rsu.ru
[c]Department of Chemistry, Rostov State University,
7 ul. Zorge, 344090 Rostov-on-Don, Russian Federation.
Fax:+7 (8632) 22 39 58. E-mail: apozharskii@chimfak.rsu.ru

1-Alkyl-1,3-diazapyrenium salts were prepared according to two procedures. Oxidative hydroxylation of these salts afforded 1-alkyl-1,3-diazapyren-2-ones. The spectral characteristics of the resulting compounds are discussed.

Key words: 1,3-diazapyrene, quaternization, oxidative hydroxylation, luminescence.

Considerable recent attention has been given to 2,7-diazapyrenium salts[2—6] because they combine the properties of efficient luminophores, redox agents, photographic agents, and intercalators. This interest is partly extended to derivatives of other diazapyrenes.[7]

Until recently, data on the synthesis and properties of 1,3-diazapyrenes were virtually lacking. Earlier, we have developed[8] a convenient method for the preparation of these compounds, including the parent compound.** The present study was aimed at preparing 1,3-diazapyrenium salts, examining their luminescence characteristics, and investigating a number of their reactions.

Results and Discussion

Quaternization of symmetrical 1,3-diazapyrenes **1a—c** with an excess of alkyl halides in MeCN proceeded readily to give 1-alkyl-1,3-diazapyrenium salts (**2**) (Scheme 1).*** Unlike virtually colorless starting amines, salts **2** were obtained as yellow or red crystalline compounds. The appearance of a deep color results, probably, from efficient delocalization of the positive charge throughout the polynuclear π system with a high contribution of canonical structures **2″** and **2‴** to the resonance hybrid. The former structure is a combination of 1-R-perimidine and the phenalenium cation.

We studied another possible procedure for the synthesis of salts **2** based on the reactions of 1-R-perimidines with α,β-unsaturated carbonyl compounds in a medium of polyphosphoric acid (PPA). In the absence of an oxidizing agent, the reaction of 1-methylperimidine (**3**) with benzalacetophenone (chalcone) would be expected to afford intermediates of type **4** or **5** (Scheme 2). However, as in the case of unsubstituted perimidine,[8] these intermediates were not detected. At 70—75 °C, the reaction in PPA gave rise to a salt, which we isolated as perchlorate **2h** (the yield was 73%). Salt **2h** was also independently prepared by the reaction of **2c** with $HClO_4$.

Since solutions of compounds **1** and **2** exhibit fluorescence, we recorded their absorption and fluorescence spectra and measured the quantum yields of fluorescence (Table 1).

On going from bases **1** to salts **2**, the bathochromic shifts of the long-wavelength absorption maxima and the bathofloric shifts of the fluorescence maxima are observed due, apparently, to the lowering of the energy of the lowest unoccupied molecular orbital (LUMO, see Table 1). As in other analogous cases,[10] small structural changes have a pronounced effect on luminescence properties. Thus, the extension of the conjugated chain due to the presence of two phenyl groups (on going from **1a** to **1b**) leads to a visually noticeable sharp increase in the fluores-

* For Part 68, see Ref. 1.

** The detailed data will be published in *Chemistry of Heterocyclic Compounds* in 2002.

*** Diquaternization of compounds **1a—c** did not proceed under the action of alkylating agents used in the present study. We plan to prepare the corresponding dications according to other procedures in the future (*cf.* the lit. data[2,9]).

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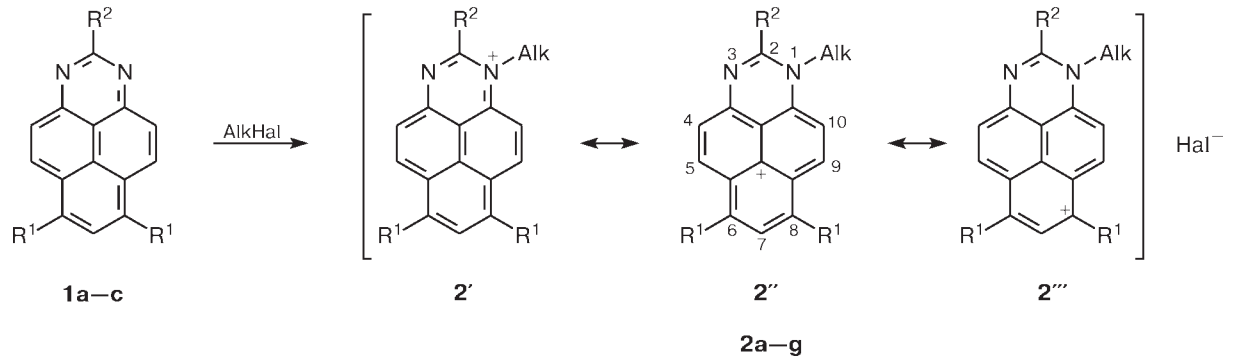
Scheme 1



	R^1	R^2	Alk	Hal		R^1	R^2	Alk	Hal
1a	H	H	—	—	**2c**	Ph	H	Me	I
1b	Ph	H	—	—	**2d**	Ph	H	Et	I
1c	Ph	Me	—	—	**2e**	Ph	H	Bn	Cl
2a	H	H	Me	I	**2f**	Ph	H	All	Br
2b	H	H	Et	I	**2g**	Ph	Me	Me	I

cence intensity under UV light. Analogous changes accompanied by substantial bathochromic and bathofloric shifts are observed when comparing the properties of salts **2a,b** and **2c—g**. In the latter case, noteworthy are high quantum yields. For all compounds, the Stokes shifts are observed in a standard range. However, the Stokes shifts for bases **1b** and salts **2c—g** (45—51 nm) are essentially larger than the corresponding values for **2a,b** (24—28 nm) and depend only weakly on the solvent used. The nature both of the *N*-alkyl radical and the counterion affects only slightly the values of λ_{max} of the long-wavelength absorption and fluorescence of compounds **2**.

Scheme 2

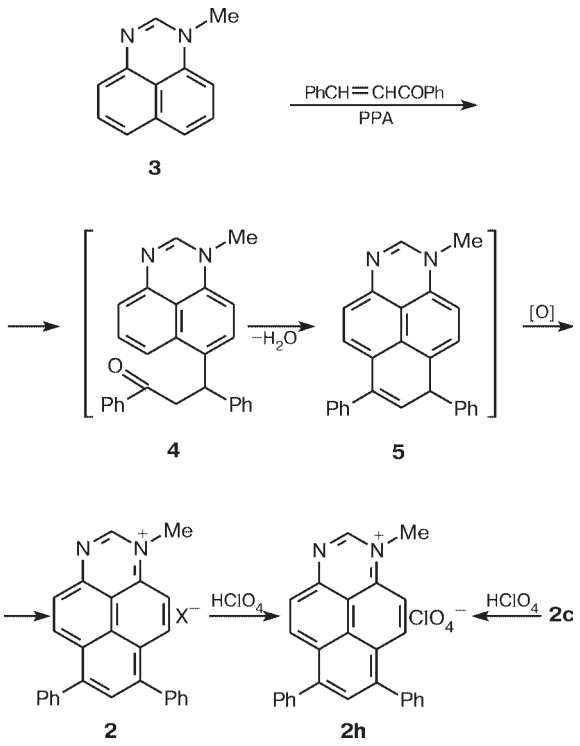


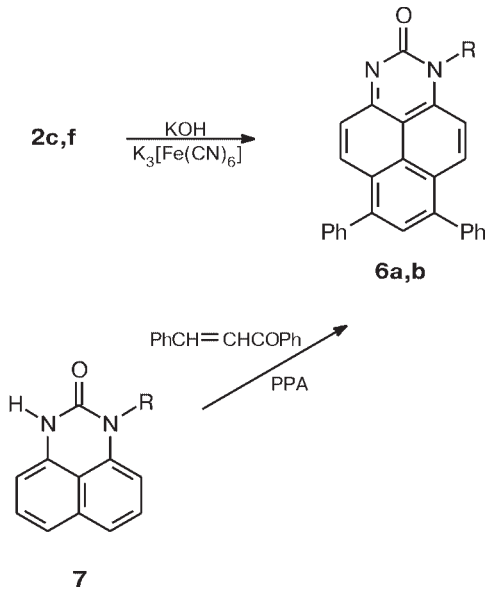
Table 1. UV and fluorescence spectra of compounds **1** and **2**

Compound	UV spectrum (EtOH), λ_{max}/nm (lgε)	Fluorescence (EtOH), λ_{max}/nm (Φ_{fl})	UV spectrum ($CHCl_3$), λ_{max}/nm (lgε)	Fluorescence ($CHCl_3$), λ_{max}/nm (Φ_{fl})
1a	239 (4.53) 266 (4.22) 339 (4.17) 366 (3.61)	— (—)	267 (4.26) 336 (4.20) 367 (3.60)	— (—)
1b	243 (4.52) 278 (4.46) 375 (4.44)	426 (0.87)	281 (4.43) 374 (4.40)	415 (0.38)
2a	243 (—) 286 (—)** 387 (—)	415* (0.01)	287** 394	— (—)
2b	241 (4.36) 285 (4.00) 388 (4.26)	412* (0.016)	287 (—)** 393	— (—)
2c	260 (shoulder) 427 (4.46)	477 (0.98)	434 (4.51)	478 (0.14)
2d	263 (4.38) 429 (4.50)	477 (0.97)	270 (shoulder) 435 (4.46)	477 (0.12)
2e	268 (4.32) 435 (4.42)	480 (0.88)	270 (4.34) 438 (4.41)	475 (0.13)
2f	268 (4.33) 431 (4.39)	480 (0.84)	270 (4.40) 439 (4.48)	478 (0.15)
2g	427 (4.40)	474 (0.98)	433 (4.47)	477 (0.15)

* Low-intensity fluorescence.
** The compound is poorly soluble.

Taking into account high π-deficiency of salts **2**, the latter would be expected to readily react with nucleophilic agents. Actually, treatment of compounds **2c,f** with aqueous alkali in the presence of $K_3[Fe(CN)_6]$ afforded products of oxidative hydroxylation at position 2, *viz.*, 1-methyl- and 1-allyl-6,8-diphenyl-1,3-diazapyren-2-ones (**6a,b**) in 85 and 32% yields, respectively (Scheme 3).

Scheme 3



We prepared compound **6a** also by the reaction of chalcone with 1-methylperimidone (**7**) in PPA. Undoubtedly, the latter reaction proceeded analogously to that in the case of perimidine. Hence, the proposed procedure for the fusion of the *peri*-ring to perimidines has a rather general character.

Compared to the signals for the protons of the diazapyrene ring in the ^{1}H NMR spectra of bases, the corresponding signals for salts **2** are shifted downfield even more substantially and are observed at δ > 8.5, the largest shift being characteristic of the H(2) proton (δ 10.2—10.4). The protons at positions 4, 5, 9, and 10 are manifested as two pairs of doublets, whereas the H(6) and H(8) protons of the peripheral *peri*-ring of compounds **2a,b** give one two-proton doublet. In the spectra of compounds **2c—g**, the phenyl groups located in the same positions give two multiplet signals with the intensity ratio of 2 : 3, *i.e.*, the signals for the *ortho* protons are slightly shifted downfield due, apparently, to anisotropy of the diazapyrene ring. The ^{1}H NMR spectra of compounds **6a,b** are characterized by the large spin-spin coupling constants for the *ortho* substituents ($J_{4,5} = J_{9,10}$ = 9.4 Hz for both compounds). The mass spectrum of compound **6a** has a pronounced medium-intensity molecular ion peak [M] and a peak [M + 1] corresponding to the $^{13}C_1$-isotopomer. The main initial direction of fragmentation of the molecular ion involves elimination of the $H_2C{=}NH$ group (the intense signal [M − 29]), whereas elimination of the methyl group at this stage does not take place at all (the signal [M − 15] is absent).

To summarize, 1-alkyl-1,3-diazapyrenium salts can be prepared both by quaternization of 1,3-diazapyrenes with alkyl halides and by the reactions of 1-alkylperimidines with chalcones in a medium of polyphosphoric acid. Their oxidative hydroxylation affords the corresponding 1-alkyl-1,3-diazapyren-2-ones. Some of the compounds synthesized have high quantum yields of fluorescence.

Experimental

The ^{1}H NMR spectra were recorded on a Bruker WP-200 instrument with Me_4Si as the internal standard. The assignment of the signals was made using selective homodecoupling. The mass spectra were measured on an MX-1321A instrument (70 eV, EI). The electronic absorption spectra were recorded on a Specord M-40 spectrophotometer. The fluorescence spectra were measured on a Shimadzu RF 5001 PC spectrofluorometer. The quantum yields of fluorescence were determined by Parker—Rees's[11] method with the use of quinine bisulfate in a 1 *M* solution of H_2SO_4 (Φ_{fl} = 0.51, λ_{ex} = 365 nm[12]) and 3-methoxybenzanthrone in toluene (Φ_{fl} = 0.1, λ_{ex} = 365 nm[10]) as the standard luminophores. The course of the reactions and the purities of the compounds were monitored on Silufol UV-254 plates. Column chromatography was carried out on silica gel Chemapol L (40—100 μm).

Acetononitrile, dioxane, benzalacetophenone, and ethyl acetate of chemical purity grade were used. Polyphosphoric acid was prepared according to a known procedure.[13] 1-Methylperimidine and 1-methylperimidone were synthesized according to procedures reported previously.[14,15]

Synthesis of salts 2 (general procedure). A solution of compound **1a—c** (1 mmol) and the corresponding alkyl halide (3 mmol) in MeCN (20 mL) was refluxed for 4 h. Then the reaction mixture was concentrated to 5 mL and benzene (15 mL) was added. The precipitate that formed was filtered off, washed with benzene and light petroleum, and dried.

1-Methyl-1,3-diazapyrenium iodide (2a). The yield was 56%. Yellow crystals, m.p. 312—314 °C. ^{1}H NMR (DMSO-d_6), δ: 4.76 (s, 3 H, Me); 8.60 and 9.37 (both d, 1 H each, H(4), H(5), $J_1 = J_2$ = 9.4 Hz); 8.67 (t, 1 H, H(7), J = 7.7 Hz); 8.80 and 9.50 (both d, 1 H each, H(10), H(9), $J_1 = J_2$ = 9.4 Hz); 9.20 (d, 2 H, H(6), H(8), $J_{6(8),7}$ = 7.7 Hz); 10.11 (s, 1 H, H(2)). Found (%): C, 52.24; H, 3.32; N, 8.15. $C_{15}H_{11}IN_2$. Calculated (%): C, 52.05; H, 3.20; N, 8.09.

1-Ethyl-1,3-diazapyrenium iodide (2b). The yield was 48%. Yellow crystals, m.p. 265—267 °C. ^{1}H NMR (DMSO-d_6), δ: 1.73 (t, 3 H, Me, J = 7.2 Hz); 5.24 (q, 2 H, CH_2, J = 7.2 Hz); 8.60 and 9.36 (both d, 1 H each, H(4), H(5), $J_1 = J_2$ = 9.4 Hz); 8.67 (t, 1 H, H(7), J = 7.7 Hz); 8.80 and 9.49 (both d, 1 H each, H(10), H(9), $J_1 = J_2$ = 9.4 Hz); 9.20 (d, 2 H, H(6), H(8), $J_{6(8),7}$ = 7.7 Hz); 10.16 (s, 1 H, H(2)). Found (%): C, 53.45; H, 3.33; N, 7.58. $C_{16}H_{13}IN_2$. Calculated (%): C, 53.35; H, 3.64; N, 7.78.

1-Methyl-6,8-diphenyl-1,3-diazapyrenium iodide (2c). The yield was 92%. Cherry-red crystals, m.p. 236—237 °C. ^{1}H NMR (DMSO-d_6), δ: 5.00 (s, 3 H, Me); 7.75 (m, 6 H, *m*- and *p*-H Ph); 7.89 (m, 4 H, *o*-H Ph); 8.64 and 9.30 (both d, 1 H each, H(4), H(5), $J_1 = J_2 = 9.4$ Hz); 8.65 (s, 1 H, H(7)); 8.96 and 9.42 (both d, 1 H each, H(10), H(9), $J_1 = J_2 = 9.4$ Hz); 10.23 (s, 1 H, H(2)). Found (%): C, 65.24; H, 4.01; N, 5.48. $C_{27}H_{19}IN_2$. Calculated (%): C, 65.07; H, 3.84; N, 5.62.

1-Ethyl-6,8-diphenyl-1,3-diazapyrenium iodide (2d). The yield was 90%. Red crystals, m.p. 273—274 °C. ^{1}H NMR (DMSO-d_6), δ: 1.71 (t, 3 H, Me, $J = 7.1$ Hz); 5.20 (q, 2 H, CH_2, $J = 7.1$ Hz); 7.65 (m, 6 H, *m*- and *p*-H Ph); 7.82 (m, 4 H, *o*-H Ph); 8.58 (s, 1 H, H(7)); 8.60 and 9.14 (both d, 1 H each, H(4), H(5), $J_1 = J_2 = 9.4$ Hz); 8.87 and 9.19 (both d, 1 H each, H(10), H(9), $J_1 = J_2 = 9.7$ Hz); 10.20 (s, 1 H, H(2)). Found (%): C, 65.54; H, 4.17; N, 5.38. $C_{28}H_{21}IN_2$. Calculated (%): C, 65.64; H, 4.13; N, 5.47.

1-Benzyl-6,8-diphenyl-1,3-diazapyrenium chloride (2e). The yield was 34%. Yellow crystals, m.p. 192—194 °C. ^{1}H NMR (DMSO-d_6), δ: 6.45 (s, 2 H, CH_2); 7.40—7.49 (m, 5 H, $C_6\underline{H}_5CH_2$); 7.70—7.84 (m, 10 H, 2 Ph); 8.56 (s, 1 H, H(7)); 8.65 and 9.18 (both d, 1 H each, H(10), H(9), $J_1 = J_2 = 9.4$ Hz); 8.71 and 9.15 (both d, 1 H each, H(4), H(5), $J_1 = J_2 = 9.4$ Hz); 10.36 (s, 1 H, H(2)). Found (%): C, 82.12; H, 4.73; N, 5.87. $C_{33}H_{23}ClN_2$. Calculated (%): C, 82.06; H, 4.80; N, 5.80.

1-Allyl-6,8-diphenyl-1,3-diazapyrenium bromide (2f). The yield was 58%. Yellow crystals, m.p. 204—206 °C. ^{1}H NMR ($CDCl_3$), δ: 5.41 and 5.45 (both br.d, 1 H each, CH_2—CH=C$\underline{H}_2$, $J_{cis} = 9.8$ Hz, $J_{trans} = 16.2$ Hz); 6.35 (m, 1 H, CH_2—C$\underline{H}$=CH_2); 6.43 (m, 2 H, C$\underline{H}_2$—CH=CH_2); 7.65—7.68 (m, 10 H, 2 Ph); 8.47 (s, 1 H, H(7)); 8.49 and 9.12 (both d, 1 H each, H(4), H(5), $J_1 = J_2 = 9.4$ Hz); 8.98 and 9.28 (both d, 1 H each, H(10), H(9), $J_1 = J_2 = 9.4$ Hz); 10.29 (s, 1 H, H(2)). Found (%): C, 72.82; H, 4.31; N, 5.67. $C_{29}H_{21}BrN_2$. Calculated (%): C, 72.96; H, 4.43; N, 5.87.

1,2-Dimethyl-6,8-diphenyl-1,3-diazapyrenium iodide (2g). The yield was 50%. Yellow-brown crystals, m.p. 254—256 °C. ^{1}H NMR (DMSO-d_6), δ: 3.34 (s, 3 H, C(2)Me); 4.60 (s, 3 H, NMe); 7.71—7.84 (m, 10 H, 2 Ph); 8.47 and 9.07 (both d, 1 H each, H(4), H(5), $J_1 = J_2 = 9.0$ Hz); 8.51 (s, 1 H, H(7)); 8.85 and 9.14 (both d, 1 H each, H(10), H(9), $J_1 = J_2 = 9.8$ Hz). Found (%): C, 65.74; H, 4.31; N, 5.58. $C_{28}H_{21}IN_2$. Calculated (%): C, 65.64; H, 4.13; N, 5.47.

1-Methyl-6,8-diphenyl-1,3-diazapyrenium perchlorate (2h). ***A.*** Benzalacetophenone (0.30 g, 1.4 mmol) was added portionwise with stirring to a mixture of 1-methylperimidine (**3**) (0.20 g, 1.1 mmol) and PPA (5 g), which was heated to 75 °C, during 30 min. Then the reaction mixture was stirred at the same temperature for 1.5 h and poured into cold water (30 mL). The precipitate that formed was half crystalline and half amorphous. It was solidified in 30 min. Then the precipitate was separated by filtration, dissolved in 60% $HClO_4$ (20 mL) with heating, and poured into cold water (50 mL). The precipitate of the salt that formed was filtered off and washed with water until washings became neutral. The product was dried and then refluxed with AcOEt (20 mL) for 5 min to purify from soluble impurities. After filtration, the yield was 0.36 g (73%), m.p. 286—288 °C.

B. Salt **2c** (0.20 g) was dissolved in 60% $HClO_4$ (10 mL) with heating and the reaction mixture was poured into cold water (30 mL). The precipitate that formed was filtered off, washed with water until washings became neutral, and dried. The product was purified as described above. M.p. 287—288 °C. The specimens of the salts prepared according to procedures *A* and *B* did not give a melting point depression.

1-Methyl-6,8-diphenyl-1,3-diazapyren-2-one (6a). ***A.*** An aqueous solution of KOH (0.13 g, 2.4 mmol) and $K_3[Fe(CN)_6]$ (0.40 g, 1.2 mmol) was added dropwise with stirring to a suspension of 1-methyl-6,8-diphenyl-1,3-diazapyrenium iodide (**2c**) (0.30 g, 0.6 mmol) in dioxane (20 mL) with heating to 80 °C for 10 min. The reaction mixture was stirred at this temperature for 1 h. Then water (30 mL) was added. After 30 min, the precipitate that formed was filtered off. The dry product was placed in a Soxlet apparatus and extracted with AcOEt for 2 h. The solution was concentrated until it became turbid. The precipitate that formed upon cooling was filtered off and dried. The yield of yellow-brown crystals was 0.20 g (85%), m.p. 262—264 °C (from a mixture of benzene and light petroleum).

^{1}H NMR ($CDCl_3$), δ: 4.03 (s, 3 H, Me); 7.60 (m, 10 H, 2 Ph); 7.63 and 8.32 (both d, 1 H each, H(10), H(9), $J_1 = J_2 = 9.4$ Hz); 7.73 and 8.60 (both d, 1 H each, H(4), H(5), $J_1 = J_2 = 9.4$ Hz); 7.88 (s, 1 H, H(7)). MS, m/z (I_{rel} (%)): 387 [M + 1] (7), 386 [M] (26), 357 [M – H_2C=NH] (44). Found (%): C, 83.82; H, 4.81; N, 7.37. $C_{27}H_{18}N_2O$. Calculated (%): C, 83.92; H, 4.69; N, 7.25.

B. Benzalacetophenone (0.27 g, 1.3 mmol) was added portionwise with stirring to a mixture of 1-methylperimidone (**7**) (0.20 g, 1 mmol) and PPA (5 g), which was heated to 65 °C, during 15 min. Then the reaction mixture was stirred at 65—70 °C for 1.5 h, poured into cold water (50 mL), made alkaline (pH ≈ 8) with a solution of NH_3, and extracted with AcOEt (3S30 mL). The solution was dried with Na_2SO_4, filtered, concentrated until the it became turbid, and cooled. The precipitate that formed was separated by filtration and dried. The yield was 0.12 g (30%), m.p. 262—264 °C (from a mixture of benzene and light petroleum). The specimens of **6a** prepared according to procedures *A* and *B* did not give a melting point depression.

1-Allyl-6,8-diphenyl-1,3-diazapyren-2-one (6b). An aqueous solution of KOH (0.14 g, 2.4 mmol) and $K_3[Fe(CN)_6]$ (0.40 g, 1.2 mmol) was added dropwise with stirring to a solution of 1-allyl-6,8-diphenyl-1,3-diazapyrenium bromide (**2f**) (0.27 g, 0.6 mmol) in dioxane (10 mL) with heating to 80 °C during 10 min. Then the reaction mixture was stirred at the same temperature for 1 h and extracted with AcOEt (3S30 mL). The solution was concentrated to 20 mL and transferred to a chromatographic column with silica gel. The yellow fraction was eluted with AcOEt. Brown crystals were obtained in a yield of 0.07 g (32%), m.p. 178—180 °C (from a mixture of benzene and light petroleum).

^{1}H NMR ($CDCl_3$), δ: 5.17 and 5.29 (both dd, 1 H each, CH_2—CH=C$\underline{H}_2$, both $J_{trans} = 15.7$ Hz, both $J_{cis} = 10.3$ Hz, both $J_{gem} = 2.8$ Hz); 5.23 (d, 2 H, C$\underline{H}_2$—CH=CH_2, $J = 5.3$ Hz); 6.10 (m, 1 H, CH_2—C$\underline{H}$=CH_2); 7.58—7.60 (m, 10 H, 2 Ph); 7.64 and 8.34 (both d, 1 H each, H(10), H(9), $J_1 = J_2 = 9.4$ Hz); 7.69 and 8.57 (both d, 1 H each, H(4), H(5), $J_1 = J_2 = 9.4$ Hz); 7.87 (s, 1 H, H(7)).

^{1}H NMR (acetone-d_6), δ: 5.16—5.27 (m, 4 H, C$\underline{H}_2$—CH=C$\underline{H}_2$); 6.13 (m, 1 H, CH_2—C$\underline{H}$=CH_2); 7.51 and 8.58 (both d, 1 H each, H(10), H(9), $J_1 = J_2 = 9.8$ Hz); 7.60—7.75 (m, 10 H, 2 Ph); 7.87 (s, 1 H, H(7)); 7.98 and 8.31 (both d, 1 H each, H(4), H(5), $J_1 = J_2 = 9.4$ Hz). Found (%): C, 84.62; H, 4.81; N, 6.47. $C_{29}H_{20}N_2O$. Calculated (%): C, 84.44; H, 4.89; N, 6.79.

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