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SYNTHESIS AND HYDROXYLATION OF 1-ALKYL- AND 7-ALKYL-1,3,7-TRIAZAPYRENIUM SALTS

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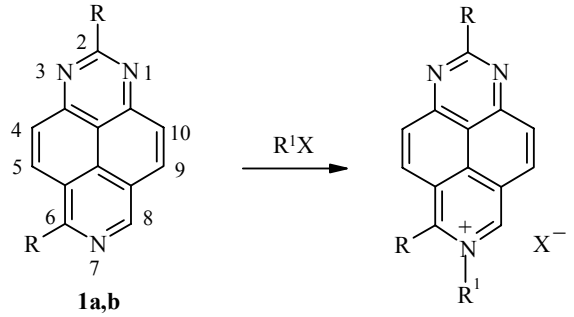
The regioselectivity of quaternization of 1,3,5-triazapyrenes by alkyl halides has been studied. Oxidative hydroxylation of 1-alkyl- and 7-alkyl-1,3,7-triazapyrenium salts gives 1-alkyl-1,2-dihydro-1,3,7-triazapyren-2-ones or 7-alkyl-6,7-dihydro-1,3,7-triazapyren-6-ones respectively. In the absence of an oxidant the hydroxylation of the 1-alkyl-1,3,7-triazapyrenium salts leads to a hydrolytic cleavage of the heterocycle.

Keywords: 1,3,7-triazapyrenes, 1,3,7-triazapyrenium salts, hydroxylation, quaternization.

Virtually nothing was known up to this time regarding the preparation and properties of 1,3,7-triazapyrenes. We have recently developed a series of synthetic methods for these compounds from perimidines, including the parent of the series [1-4]. The aim of our work was to study the regioselectivity of the quaternization process for 1,3,7-triazapyrenes and to investigate hydroxylation reactions for the 1,3,7-triazapyrenium salts obtained.



1a,b **2a–d**

1 a R = H, **b** R=Ph; **2 a** R = H, R^1 = Me, X = I; **b** R = H, R^1 = Et, X = I; **c** R = H, R^1 = $PhCOCH_2$, X = Br; **d** R = Ph, R^1 = Me, X = I

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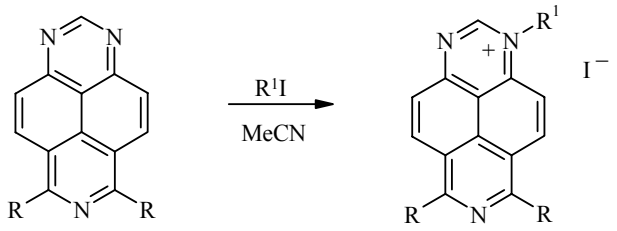
A priori it can be proposed that the nucleophilicity (and basicity) of the N-7 atom of the 1,3,7-triazapyrene (**1a**) will be greater than the N-1 and N-3 atoms due to their acceptor properties. Indeed, quaternization of the unsubstituted compound **1a** and of 2,6-diphenyl-1,3,7-triazapyrene (**1b**) using an excess of alkyl halides in acetonitrile occurs readily to give the 7-alkyl-1,3,7-triazapyrenium salts **2a-d**.

However, in the case of the 6,8-dimethyl- (**1c**) and 6,8-diphenyl-1,3,7-triazapyrene (**1d**) the regioselectivity of the reaction changes and only the 1-alkyl-1,3,7-triazapyrenium salts **3a-c** are formed.

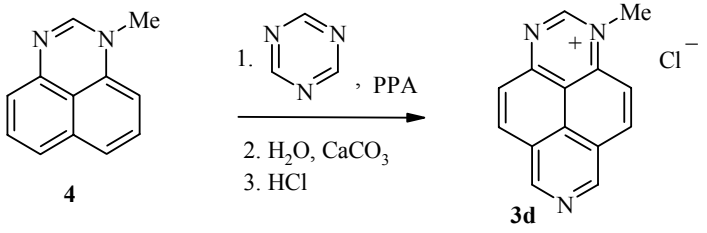


1c,d **3a–c**

1c R = Me, **d** R = Ph; **3 a** R = R^1 = Me; **b** R = Ph, R^1 = Me; **c** R = Ph, R^1 = Et

Evidently, the change in reaction route is due to steric hindrance from the substituents in positions 6 and 8.

We have also studied another possible method for the synthesis of the salts **3** i.e. by reaction of 1-R-perimidines with *sym*-triazines in PPA*. As in the case of the unsubstituted perimidine [1], the reaction of the 1-methylperimidine (**4**) with 1,3,5-triazine results in formation of a salt which could be separated as the chloride **3d**. However, the yield of the 1-methyl-1,3,7-triazapyrenium chloride (**3d)** was only 45% due to problems in separation.



4 **3d**

Quaternization of 6-phenyl-1,3,7-triazapyrene (**1e**) with methyl iodide resulted in the formation of a mixture of the three possible salts **2e, 3e, 3f** in the ratio 4: 3:3 (from ^{1}H NMR spectroscopic data).

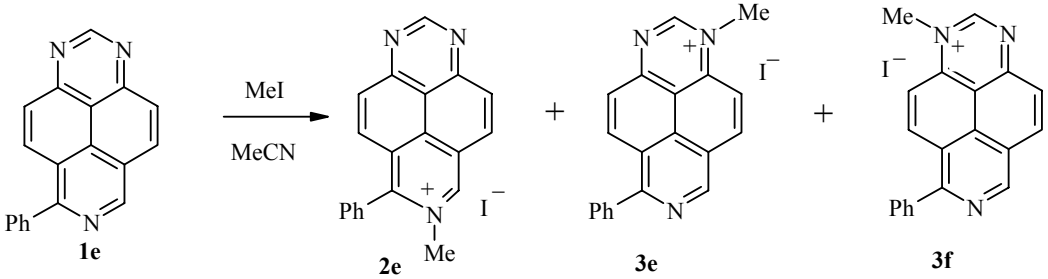
2,6,8-Trimethyl- and 2,6,8-tripenyl-1,3,7-triazapyrenes did not take part in the quaternization reaction, even upon prolonged refluxing in acetonitrile with a large excess of methyl iodide or dimethyl sulfate.

Thus substituents in the 6 and 8 positions block S_N^2-quaternization at atom N-7 and substituents at position 2 at atoms N-1 and N-3 . It should be noted in particular that, in contrast to 4,9- [6] and 2,7-diazapyrenes [7, 8], the products of a double quaternization of the 1,3,7-triazapyrenes **1a-e** were not observed.

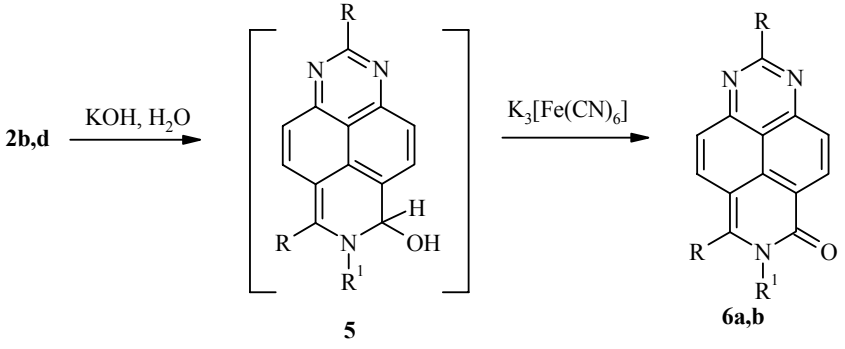
The ^{1}H NMR spectra of the salts **2** and **3** show a series of features. In particular, when compared with the starting triazapyrenes, the signals of all of the protons are shifted to low field, in greatest degree relating to the protons in positions 2 and 6(8). The signals for the phenyl groups in positions 2, 6, and 8 in the triazapyrenes appear as two multiplets since the *ortho* protons fall in the deshielding area of the pyridine ring atoms and are shifted to low field, although by a markedly different degree. Thus for 2,6,8-triphenyl-1,3,7-triazapyrene the signal at 7.95 corresponds to the *o*-H of the 6(8)-Ph and that at 8.84 ppm to the *o*-H of the 2-Ph [1]. In contrast

* PPA with an 86% content of P_2O_5 was used as obtained in the method [5].

to the salts **3b,c,e,f**, where rotation of the 6(8)-phenyl substituent is not hindered, the main conformations of the benzene rings in the 6-phenyl-substituted salts **2d** and **2e** are placed perpendicularly to the triazapyrene ring plane and so their protons appear as a sharp multiplet without separation of the *ortho* protons to low field. The signals for the methyl group and the H-5 proton fall in the shielding region of the benzene ring and are shifted by 0.4 and 0.53 ppm to high field respectively when compared with 7-methyl-1,3,7-triazapyrenium iodide (**2a**). These features permit an unambiguous assignment of the signals for the protons in the mixture of salts **2e, 3e** and **3f**.



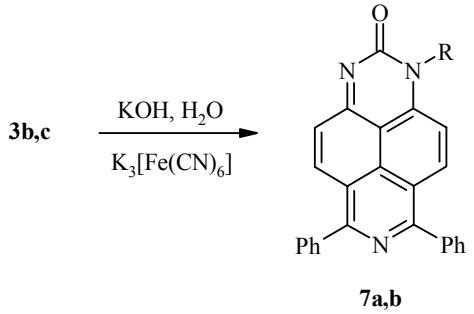
An increased π-deficiency of salts **2** and **3** suggested a ready reaction with nucleophilic reagents. In fact, treatment of salts **2b,d** with aqueous alkali in the presence of $K_3[Fe(CN)_6]$ gave the products of oxidative hydroxylation, i.e. 7-ethyl-6,7-dihydro-1,3,7-triazapyren-6-one (**6a**) and 7-methyl-2,8-diphenyl-6,7-dihydro-1,3,7-triazapyren-6-one (**6b**) in 59 and 53% yields respectively.



6 a R = H, R^1 = Et; **b** R = Ph, R^1 = Me

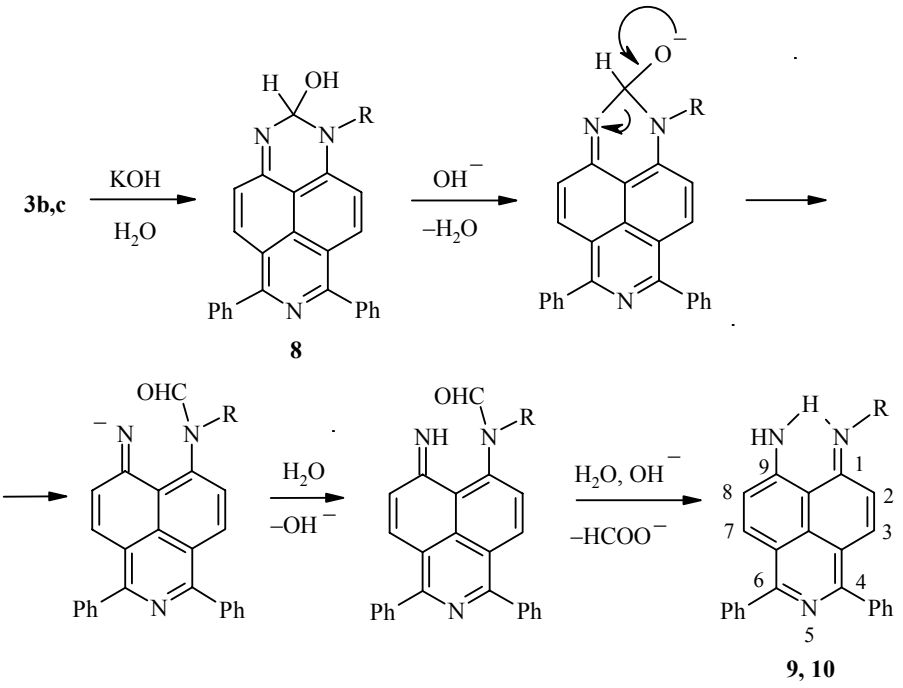
The reaction takes place *via* a stage of formation of the pseudobase **5** and its subsequent oxidation.

Salts **3b,c** react similarly under these conditions to give the 1-methyl- (**7a**) and 1-ethyl-6,8-diphenyl-1,2-dihydro-1,3,7-triazapyren-2-ones (**7b**) in 60 and 58% yields respectively.

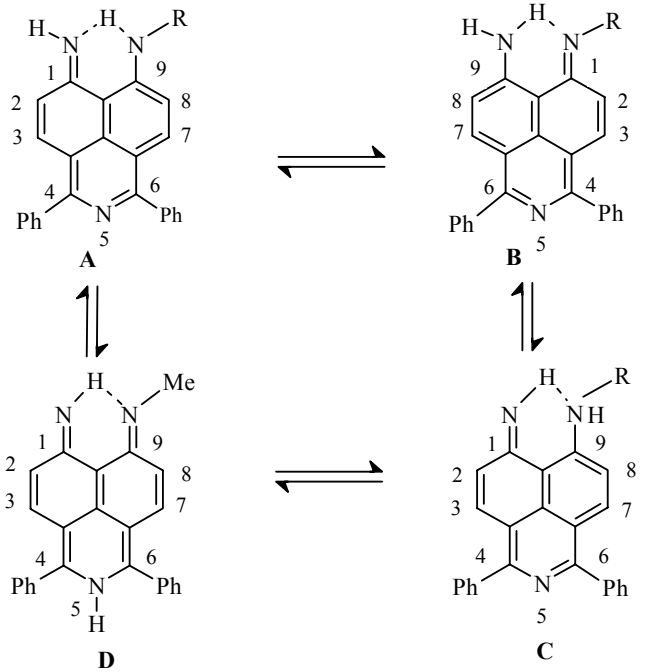


7 a R = Me; **b** R = Et

In the absence of oxidant, however, the reaction of salts **3b,c** with aqueous alkali occurs differently. The pseudobase **8** formed in the first step undergoes hydrolytic cleavage of the heterocycle and the corresponding N-methyl- (**9**) and N-ethyl-9-amino-4,6-diphenyl-5-azaphenalenone-1-imine (**10**) can be separated.



9 R = Me; **10** R = Et



9 R = Me; **10** R = Et

In principle, compounds **9** and **10** can exist in $CDCl_3$ solution as an equilibrium mixture of four non-degenerate tautomers **A-D**. In our opinion the main tautomers are **A** and **B** since transfer between them involves migration of a hydrogen bridge and rebuilding of the π-electron system while formation of **C** has to include cleavage of a hydrogen bond and synchronous inversion of a nitrogen atom. The possibility of the further tautomer **D** seems unlikely due to disturbance to the aromaticity of the system.

The ^{1}H NMR spectra of the amino imines **9** and **10** in $CDCl_3$ show two NH proton signals, one intramolecularly hydrogen bonded at about 12.8 and one unbonded at about 7.9 ppm and these are lost on addition of D_2O. Characteristic features of the spectra of **9** and **10** are their non-symmetry, not only with respect to their chemical shifts but also to their spin-spin couplings for the aromatic protons at positions 2,3 and 7,8 (9.50 and 9.80 Hz). In our view, this indicates that they exist mainly in a single tautomeric form. Since an unambiguous choice of this form is difficult the recorded spectra of compounds **9** and **10** are reported arbitrarily as the **B** tautomers (see Experimental).

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Bruker-200 instrument (200 MHz) using DMSO-d_6 (compounds **2a-c**, **3a-f**, and **6a,b**) or $CDCl_3$ (compounds **7a,b**, **9**, and **10**) with TMS as internal standard. Mass spectra were taken on an MX-1321A instrument with direct introduction of the sample into the ionizing chamber at 50-100ºC and with an ionization intensity of 70 eV.

Quaternization of 1,3,7-Triazapyrenes (General Method). A solution of the corresponding 1,3,7-triazapyrene (3 mmol) and the alkyl halide (9 mmol) in acetonitrile (20 ml) was refluxed for 4 h, the solution was evaporated to 3 ml, and benzene (10 ml) was added. The precipitate was filtered off, washed with benzene and petroleum ether, and dried. Further purification was not carried out.

7-Methyl-1,3,7-triazapyrenium Iodide (2a). Yield 1.1 g (96%). Orange crystals, mp 285-287ºC. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.87 (3H, s, CH_3); 8.65 (2H, d, *J* = 9.5, H-4,10); 9.06 (2H, d, *J* = 9.5, H-5,9); 10.15 (1H, s, H-2); 10.27 (2H, s, H-6,8). Found, %: C 48.12; H 3.06; N 12.23. $C_{14}H_{10}IN_3$. Calculated, %: C 48.44; H 2.90. N 12.10.

7-Ethyl-1,3,7-triazapyrenium Iodide (2b). Yield 0.92 g (85%). Cherry-red crystals, mp 260-262ºC. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.88 (3H, t, *J* = 7.3, CH_3); 5.22 (2H, q, *J* = 7.3, CH_2); 8.57 (2H, d, *J* = 9.3, H-4,10); 9.08 (2H, d, *J* = 9.3, H-5,9); 10.04 (1H, s, H-2); 10.52 (2H, s, H-6,8). Found, %: C 50.11; H 3.40; N 11.32. $C_{15}H_{12}IN_3$. Calculated, %: C 49.88; H 3.35; N 11.63.

7-Phenacyl-1,3,7-triazapyrenium Bromide (2c). Yield 1.47 g (82%). Dark-brown crystals, mp 282-284ºC. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.13 (2H, s, CH_2); 7.72 (5H, m, Ph); 8.60 (2H, d, *J* = 9.3, H-4,10); 9.13 (2H, d, *J* = 9.3, H-5,9); 10.06 (1H, s, H-2); 10.43 (2H, s, H-6,8). Found, %: C 62.07; H 3.55; N 10.23. $C_{21}H_{14}BrN_3O$. Calculated, %: C 62.39; H 3.49; N 10.39.

7-Methyl-2,6-diphenyl-1,3,7-triazapyrenium Iodide (2d). Yield 1.16 g (78%). Pale-yellow crystals, mp 256-258ºC. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.44 (3H, s, CH_3), 7.68 (3H, d, 2-Ph, H-*m* and -*p*); 7.84 (5H, d, 6-Ph); 8.18 (1H, d, *J* = 9.5, H-4); 8.54 (1H, d, *J* = 9.5, H-5); 8.69 (1H, d, *J* = 9.5, H-10); 8.81 (2H, m, 2-Ph, H-*o*); 9.05 (1H, d, *J* = 9.5, H-9); 10.35 (1H, s, 8-H). Found, %: C 62.29; H 3.72; N 8.60. $C_{26}H_{18}IN_3$. Calculated, %: C 62.54; H 3.63; N 8.41.

7-Methyl-6-phenyl-1,3,7-triazapyrenium Iodide (2e) was prepared as a mixture with isomers **3e** and **3f**: overall yield 81%. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.48 (3H, s, CH_3); 7.84 (5H, m, Ph); 8.21 (1H, d, *J* = 9.5, H-4); 8.53 (1H, d, *J* = 9.5, H-5); 8.70 (1H, d, *J* = 9.5, H-10); 9.11 (1H, d, *J* = 9.5, H-9); 10.15 (1H, s, H-2); 10.50 (1H, s, H-8).

1,6,8-Trimethyl-1,3,7-triazapyrenium Iodide (3a). Yield 1.4 g (80%). Red crystals, mp above 350ºC. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.38 (3H, s, 6-CH_3); 3.40 (3H, s, 8-CH_3); 4.70 (3H, s, NCH_3); 8.43 (1H, d,

J = 9.3, H-9); 8.68 (1H, d, *J* = 9.5, H-10); 9.46 (1H, d, *J* = 9.3, H-5); 9.58 (1H, d, *J* = 9.5, H-9); 10.10 (1H, s, H-2). Found, %: C 51.34; H 3.66; N 11.44. $C_{16}H_{14}IN_3$. Calculated, %: C 51.22; H 3.76; N 11.20.

1-Methyl-6,8-diphenyl-1,3,7-triazapyrenium Iodide (3b). Yield 1.06 g (71%). Red crystals, mp 286-288ºC. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.69 (3H, s, NCH_3); 7.75 (6H, m, 6- and 8-Ph, H-*m* and -*p*); 8.05 (4H, m, 6- and 8-Ph, H-*o*); 8.61 (1H, d, *J* = 9.5, H-4); 8.76 (1H, d, *J* = 9.5, H-10); 9.26 (1H, d, *J* = 9.5, H-5); 9.34 (1H, d, *J* = 9.5, H-9); 10.13 (1H, s, H-2). Found, %: C 62.88; H 3.49; N 8.31. $C_{26}H_{18}IN_3$. Calculated, %: C 62.54; H 3.63; N 8.41.

1-Ethyl-6,8-diphenyl-1,3,7-triazapyrenium Iodide (3c). Yield 1.07 g (69%). Red crystals, mp 293-295ºC. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.77 (3H, t, *J* = 7.2, CH_3); 5.28 (2H, q, *J* = 7.2, CH_2); 7.76 (6H, m, 6- and 8-Ph, H-*m* and -*p*); 8.04 (4H, d, 6- and 8-Ph, H-*o*); 8.56 (1H, d, *J* = 9.3, H-4); 8.94 (1H, d, *J* = 9.3, H-10); 9.24 (1H, d, *J* = 9.3; H-5); 9.33 (1H, d, *J* = 9.3, H-9); 10.27 (1H, s, H-2). Found, %: C 62.99; H 4.07; N 7.98. $C_{27}H_{20}IN_3$. Calculated, %: C 63.17; H 3.93; N 8.18.

1-Methyl-6-phenyl-1,3,7-triazapyrenium Iodide (3e) was obtained as a mixture with isomers **2e** and **3f**. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.73 (3H, s, CH_3); 7.76 (3H, m, Ph, H-*m* and -*p*); 8.01 (2H, m, Ph, H-*o*); 8.77 (1H, d, *J* = 9.8, H-4); 8.87 (1H, d, *J* = 9.1, H-10); 9.31 (1H, d, *J* = 9.8, H-5); 9.59 (1H, d, *J* = 9.1, H-9); 10.19 (1H, s, H-2); 10.32 (1H, s, H-8).

1-Methyl-8-phenyl-1,3,7-triazapyrenium Iodide (3f) was obtained as a mixture with isomers **2e** and **3e**. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.70 (3H, s, CH_3); 7.76 (3H, m, Ph, H-*m* and -*p*); 8.01 (2H, m, Ph, H-*o*); 8.61 (1H, d, *J* = 9.3, H-4); 8.66 (1H, d, *J* = 9.1, H-10); 9.24 (1H, d, *J* = 9.3, H-5); 9.46 (1H, d, *J* = 9.1,m H-9); 10.19 (1H, s, H-2); 10.32 (1H, s, H-6).

1-Methyl-1,3,7-triazapyrenium Chloride (3d). A mixture of 1-methylperimidine (1 mmol), 1,3,5-triazine (1.5 mmol), and PPA (86%, 3 g) was stirred for 4 h at 100-105ºC, cooled to 50ºC, poured into water (20 ml), neutralized with potassium carbonate, and filtered. The mother liquor was evaporated to dryness *in vacuo* and the residue was dissolved in methanol. The insoluble admixture was filtered off and the solution was evaporated to dryness *in vacuo*. The residue was treated with a solution of hydrochloric acid and the solution was evaporated to dryness *in vacuo*, and washed with ethyl acetate. Yield 0.11 g (45%). Brown crystals with mp 344-346ºC (decomp. with flash). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.76 (3H, s, NCH_3); 8.59 (1H, d, *J* = 9.1, H-4); 8.86 (1H, d, *J* = 9.3, H-10); 9.41 (1H, d, *J* = 9.1, H-5); 9.55 (1H, d, *J* = 9.3, H-9); 10.20 (2H, s, H-6,8); 10.21 (1H, s, H-2). Found, %: C 65.66; H 3.78; N 16.50. $C_{14}H_{10}ClN_3$. Calculated, %: C 65.76; H 3.94; N 16.43.

Oxidative Hydroxylation of the 7-Alkyl- and 1-Alkyl-1,3,7-triazapyrenium Salts (General Method). An aqueous solution containing KOH (0.216 g, 4 mmol) and $K_3[Fe(CN)_5]$ (0.67 g, 2 mmol) was added dropwise with stirring and heating to 80ºC over 10 min to a solution of the corresponding 1,3,7-triaza-pyrenium salt (1 mmol) in water (20 ml). The mixture was stirred at the same temperature for 1 h, water (30 ml) was added, and the precipitate formed was filtered off after 30 min.

7-Ethyl-6,7-dihydro-1,3,7-triazapyren-6-one (6a). Yield 0.146 g (59%). Dark-orange crystals, mp 338-340ºC (ethyl acetate). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.58 (3H, t, *J* = 7.0, CH_3); 4.42 (2H, q, *J* = 7.0, CH_2); 7.61 (1H, d, *J* = 9.2, H-9); 7.97 (1H, d, *J* = 9.2, H-10); 8.14 (1H, d, *J* = 8.9, H-4); 8.31 (1H, s, H-8); 8.92 (1H, d, *J* = 8.9, H-5); 9.49 (1H, s, H-2). Found, %: C 72.09; H 4.67; N 16.52. $C_{15}H_{11}N_3O$. Calculated, %: C 72.28; H 4.45; N 16.86.

7-Methyl-2,8-diphenyl-6,7-dihydro-1,3,7-triazapyren-6-one (6b). Yield 0.205 g (53%). Yellow-green crystals, mp 275-277ºC (ethyl acetate). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.63 (3H, s, CH_3); 7.42 (2H, br. s, H-9,10); 7.47 (3H, m, 8-Ph, H-*m* and -*p*); 7.54 (3H, m, 2-Ph, H-*m* and -*p*); 7.67 (2H, m, 8-Ph, H-*o*); 8.14 (1H, d, *J* = 9.2, H-4); 8.72 (2H, m, 2-Ph, H-*o*); 8.90 (1H, d, *J* = 9.2, H-5). Found, %: C 80.48; H 4.56; N 10.49. $C_{26}H_{17}N_3O$. Calculated, %: C 80.60; H 4.42; N 10.85.

1-Methyl-6,8-diphenyl-1,2-dihydro-1,3,7-triazapyren-2-one (7a). Yield 0.233 g (60%). Yellow-green crystals, mp above 350ºC (DMSO). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.04 (3H, s, CH_3); 7.62 (6H, m, 6- and

8-Ph, H-*m* and -*p*); 7.66 (1H, d, *J* = 9.6, H-9); 7.76 (1H, d, *J* = 9.3, H-5); 7.88 (4H, m, 6- and 8-Ph, H-*o*); 8.43 (1H, d, *J* = 9.6, H-10); 8.74 (1H, d, *J* = 9.3, H-4). Found, %: C 80.76; H 4.33; N 10.90. $C_{26}H_{17}N_3O$. Calculated, %: C 80.60; H 4.42; N 10.85.

1-Ethyl-6,8-diphenyl-1,2-dihydro-1,3,7-triazapyren-2-one (7b). Yield 0.233 g (58%). Yellow crystals, mp 316-318ºC (DMSO). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.55 (3H, t, *J* = 7.0, CH_3); 4.62 (2H, q, *J* = 7.0, CH_2); 7.60 (6H, m, 6- and 8-Ph, H-*m* and -*p*); 7.69 (1H, d, *J* = 9.5, H-9); 7.76 (1H, d, *J* = 9.2, H-5); 7.87 (4H, m, 6- and 8-Ph, H-*o*); 8.43 (1H, d, *J* = 9.5, H-10); 8.73 (1H, d, *J* = 9.2, H-4). Found, %: C 80.98; H 4.96; N 10.22. $C_{27}H_{19}N_3O$. Calculated, %: C 80.78; H 4.77; N 10.47.

Hydroxylation of Salts 3b,c without Oxidant (General Method). A mixture of potassium hydroxide (4 mmol) and the corresponding 1,3,7-triazapyrenium salt (1 mmol) was dissolved in ethanol (96%, 30 ml) and heated at 80ºC for 40 min. The product was diluted threefold with water and the precipitate formed was filtered off, washed with water, and dried.

N-Methyl-9-amino-4,6-diphenyl-5-azaphenalenone-1-imine (9, tautomer **B**). Yield 0.342 g (95%). Yellow crystals, mp 173-175ºC (ethyl acetate and petroleum ether). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.34 (3H, s, CH_3); 6.85 (1H, d, *J* = 9.5, H-8); 7.20 (1H, d, *J* = 9.8, H-3); 7.49 (6H, m, 4- and 6-Ph, H-*m* and -*p*); 7.67 (1H, d, *J* = 9.5, H-7); 7.73 (4H, m, 4- and 6-Ph, H-*o*); 7.90 (1H, br. s, NH); 8.07 (1H, d, *J* = 9.8, H-2); 12.80 (1H, br. s, NH···N). The signals at 7.90 and 12.80 ppm disappeared upon addition of D_2O. Mass spectrum, *m/z* (I_{rel}, %): 361 $[M]^+$ (100), 362 $[M^+ + 1]$ (28), 343 (93), 329 (35), 207 (22), 44 (45). Found, %: C 82.82; H 5.26; N 11.43. $C_{25}H_{19}N_3$. Calculated, %: C 83.08; H 5.30; N 11.63.

N-Ethyl-9-amino-4,6-diphenyl-5-azaphenalenone-1-imine (10, tautomer **B**). Yield 0.348 g (93%). Yellow crystals, mp 149-151ºC (ethyl acetate and petroleum ether). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.46 (3H, t, *J* = 7.2, CH_3); 3.63 (2H, q, *J* = 7.2, CH_2); 6.72 (1H, d, *J* = 9.6, H-8); 7.18 (1H, d, *J* = 9.6, H-3); 7.50 (6H, m, 4- and 6-Ph, H-*m* and -*p*); 7.62 (1H, d, *J* = 9.3, H-7); 7.73 (4H, m, 4- and 6-Ph, H-*o*); 7.92 (1H, br. s, NH); 8.02 (1H, d, *J* = 9.3, H-2); 12.75 (1H, br. s, NH···N). The signals at 7.92 and 12.75 ppm disappeared upon addition of D_2O. Found, %: C 83.02; H 5.76; N 11.31. $C_{26}H_{21}N_3$. Calculated, %: C 83.17; H 5.64; N 11.19.

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