

**NOVEL THREE-COMPONENT REACTION OF PERIMIDINES
WITH 1,3,5-TRIAZINES AND CARBONYL COMPOUNDS IN
POLYPHOSPHORIC ACID. AN EFFICIENT METHOD FOR
peri-ANNELATION OF A CARBOCYCLIC AND PYRIDINE RING**

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Methods have been developed for the synthesis of 1,3-diazapyrenes, 8(6)-aryl-6(8)-oxo-1,6,7,8-tetrahydro-1,3-diazapyrenes, and 1,3,7-triazapyrenes based on a three-component reaction of perimidines with 1,3,5-triazines and carbonyl compounds, benzonitrile, or vinyl butyl ether in polyphosphoric acid.

Keywords: aldehydes, 8(6)-aryl-6(8)-oxo-1,6,7,8-tetrahydro-1,3-diazapyrene, 1,3-diazapyrenes, ketones, nitriles, perimidines, polyphosphoric acid, 1,3,7-triazapyrenes, 1,3,5-triazines, *peri*-annelation.

Polynuclear aromatic and heteroaromatic compounds, including derivatives of pyrene and their heterocyclic analogs, have considerable practical value. Many organic luminophores and dyes [1-4] and efficient medications [5-10] have been created on their basis. Recently, interest has arisen in such structures, principally as luminescent intercalators [11-15] and also in connection with the nanotechnologies development as potential "structural units" for building different nanostructures [16].

Despite the many possible structures for azapyrenes (about 300), only a few representatives have been synthesized at this time and generally without functional groups [17, 18]. This is mostly due to the absence of convenient methods for the *peri*-annelation of carbocyclic and heterocyclic rings to azaphenalenes, including perimidine derivatives.

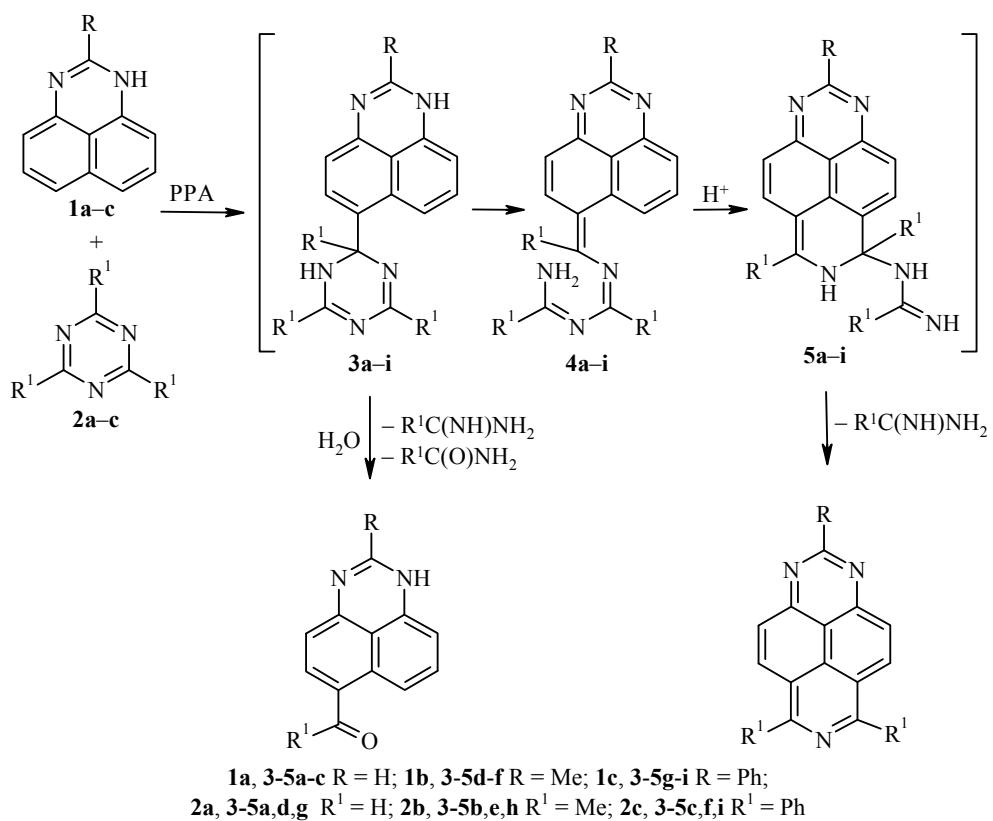
In our work, we propose novel methods for *peri*-annelation of carbocyclic and [c,d]pyridine rings to the perimidine.

A series of methods have been reported in the literature for the *peri*-annelation of carbocyclic ring to naphthalenes and phenalenes which allow the synthesis of phenalenes and pyrenes (including 1,3-diazapyrenes). These methods include the reaction of naphthalenes or phenalenes with α,β -unsaturated carbonyl compounds or the condensation of two carbonyl compounds and subsequent ring closure [17-27].

We have previously developed methods for the acylation of perimidines **1a-c** with 1,3,5-triazines **2a-c** [18, 28, 29] and *peri*-annelation of a pyridine ring to phenalenes and azaphenalenes using these reagents [18, 30, 31]. The following scheme has been proposed for these reactions:

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Based on this scheme, we have proposed that the reaction course can be directed to annelation of a carbocyclic ring by the addition to the reaction mixture of a carbonyl compound **6a-d** in the presence of polyphosphoric acid (PPA) to give derivatives **7a-d**. The latter can then react with intermediates **4a-i** [32].

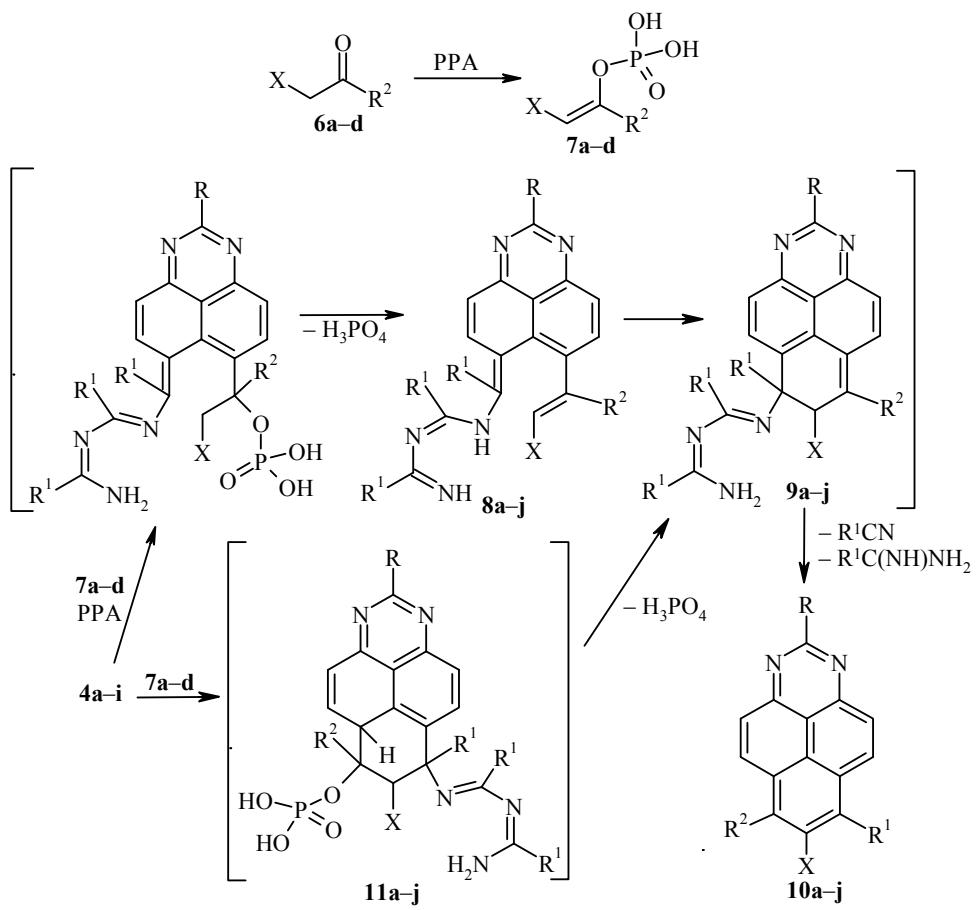
The reaction of compounds **7a-d** with intermediates **4a-i** can occur either as a result of an electrophilic substitution to form the intermediates **8a-j** with subsequent ring closure *via* an electrocyclic reaction or as a result of cycloaddition to form compounds **11a-j**. In either case the intermediates **9a-j** are formed which undergo further transformation to the diazapyrenes **10a-j**.

For this multicomponent reaction to proceed efficiently it is necessary to implement a specific sequence and rate ratio of its stages. This can be achieved by varying the temperature or the ratio of reagents. As noted before [18, 28, 29], realization of the perimidines acylation and prevention of *peri*-annelation is possible by reaction of perimidines **1a-c** with triazines **2a-c** at a temperature below 70°C and a ratio of reagents of 1:2 (the formation stage of the intermediate compounds **3a-i** is intermolecular hence its rate can be increased by raising the concentration of the corresponding triazine **2a-c**). It was found that the optimum ratio of reagents **1a-c** to **6a-d** is 3:1. To a marked extent, this ratio helps to avoid side reactions of diacylation of perimidines and their *peri*-annelation. The rate of perimidines **1a-c** reaction with ketones **6a-d** is lower than with triazines **2a-c**.

The reaction of perimidines **1a-c** (1 mmol) with triazines **2a-c** (2 mmol) and carbonyl compound **6a-d** (3 mmol) in PPA gives diazapyrenes **10a-j** in 43-75% yield.

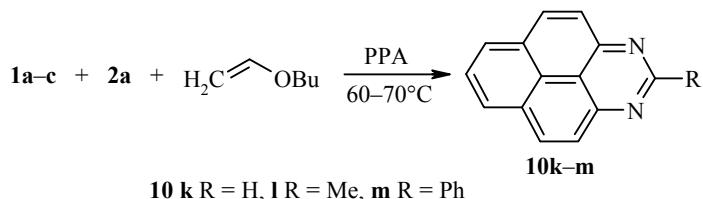
Vinyl butyl ether can be used in place of carbonyl compounds in the reaction to give compounds **10k-m** but with significantly lower yield (18-23%) in this case. An excess of the vinyl butyl ether is needed due to its polymerization in PPA.

The synthetic methods for the 1,3,7-triazapyrenes given in [30, 33, 34] have a series of drawbacks. The *peri*-annelation using 1,3,5-triazines or nitriles [30, 33] only leads to triazapyrenes with identical substituents in positions 6 and 8. The method based on the use of carbonyl perimidine derivatives needs the preliminary



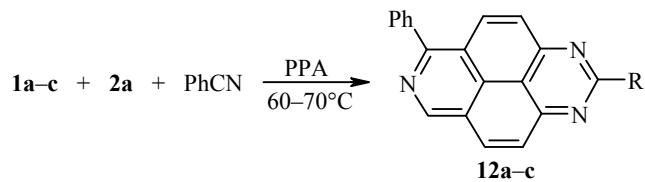
$\begin{array}{l} \mathbf{6a}, \mathbf{7a}, \mathbf{8-11a,b} \text{ X} = \text{H}, \text{R}^2 = \text{Me}; \mathbf{6b}, \mathbf{7b}, \mathbf{8-11c,f,g} \text{ X} = \text{H}, \text{R}^2 = \text{Ph}; \\ \mathbf{6c}, \mathbf{7c}, \mathbf{8-11d,j} \text{ X} = \text{COMe}, \text{R}^2 = \text{Me}; \mathbf{6d}, \mathbf{7d}, \mathbf{8-11e,h} \text{ X} = \text{CO}_2\text{Et}, \text{R}^2 = \text{Me}; \\ \mathbf{8-11a,c,d,e} \text{ R} = \text{R}^1 = \text{H}; \mathbf{b,f,j} \text{ R} = \text{H}, \text{R}^1 = \text{Me}; \mathbf{g} \text{ R} = \text{H}, \text{R}^1 = \text{Ph}; \\ \mathbf{h} \text{ R} = \text{Me}, \text{R}^1 = \text{H}; \mathbf{i} \text{ R} = \text{R}^1 = \text{Ph} \end{array}$

introduction of a carbonyl group into one of the *peri*-positions [34]. Development of the three-component reaction allows to create methods lacking these drawbacks.

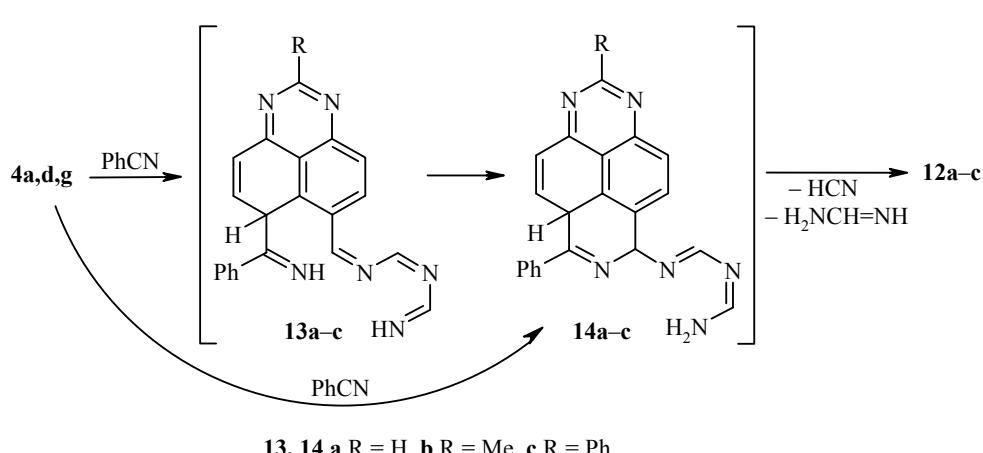


Our studies have shown that aromatic nitriles are, on one hand, suitable reagents for a second acylation but, on the other, less reactive than triazines **2a-c** [33]. This ensures the necessary regioselectivity since the perimidines **1a-c** will react firstly with triazines **2a-c** and a subsequent reaction with nitriles can be achieved. Therefore the latter can be used in a three-component reaction.

We have shown that the reaction of perimidines **1a-c** (1 mmol) with triazine **2a** (2 mmol) and benzonitrile (5 mmol) in PPA, really leads to formation of triazaperylenes **12a-c** in 71-75% yields.



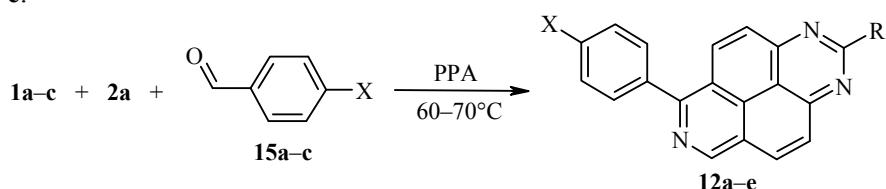
As in the case of 1,3-diazapyrène synthesis, the intermediates **4a,d,g** are formed in this reaction. They subsequently react with benzonitrile to give derivatives **14a-c** either stepwise with intermediate formation of compounds **13a-c** or by direct cycloaddition. Compounds **14a-c** then react to give the triazapyrènes **12a-c**.



We have revealed the obvious drawback of this method in that only benzonitrile can be used as the second component. Experiments have shown that the rate of reaction of intermediates **4a,d,g** with other nitriles is lower than the rate of their intramolecular cyclization to form the 1,3,7-triazapyrènes unsubstituted in the 6 and 8 positions. This applied to both 4-nitrobenzonitrile and 4-methoxybenzonitrile. Hence we decided to develop a more generally applicable three-component reaction.

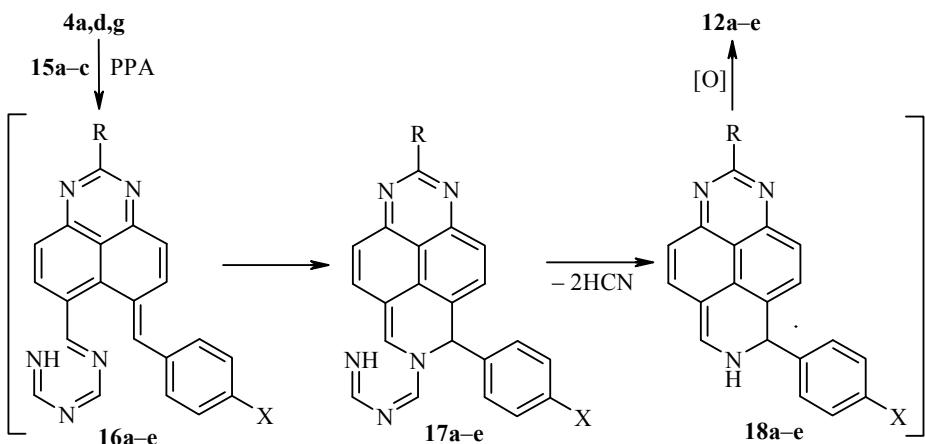
In order to resolve this problem it was necessary to use compounds more reactive than nitriles such as the aldehydes **15a-c**. It was also of interest to examine how the reaction we have discovered occurs with non-enolizable carbonyl compounds. Hence the reaction of perimidines **1a-c** with triazine **2a** in the presence of the aromatic aldehydes **15a-c** was studied.

It was found that the reaction of perimidines **1a-c** with 1,3,5-triazine (**2a**) in the presence of benzaldehydes **15a-c** occurs more readily than with benzonitrile and gives high yields (83-91%) of triazapyrènes **12a-e**.



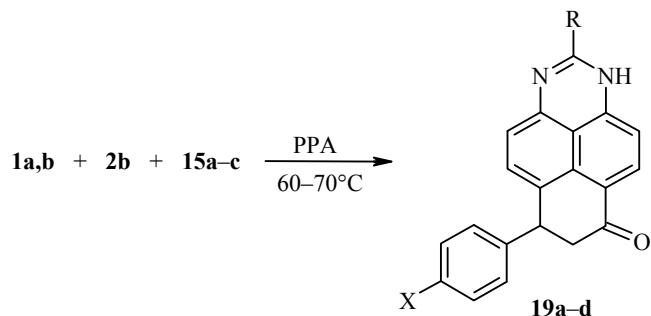
12a,d,e R = H, **12b** R = Me, **12c** R = Ph; **12a,b,c, 15a** X = H; **12d, 15b** X = Br; **12e, 15c** X = NO₂

The mechanism of the reaction is likely similar to that given for benzonitrile and includes condensation of intermediates **4a,d,g** with aldehydes **15a-c** to form compounds **16a-e**, their heterocyclization to compounds **17a-e**, loss of HCN from the latter, and oxidation of the dihydro derivatives **18a-e** by atmospheric oxygen:

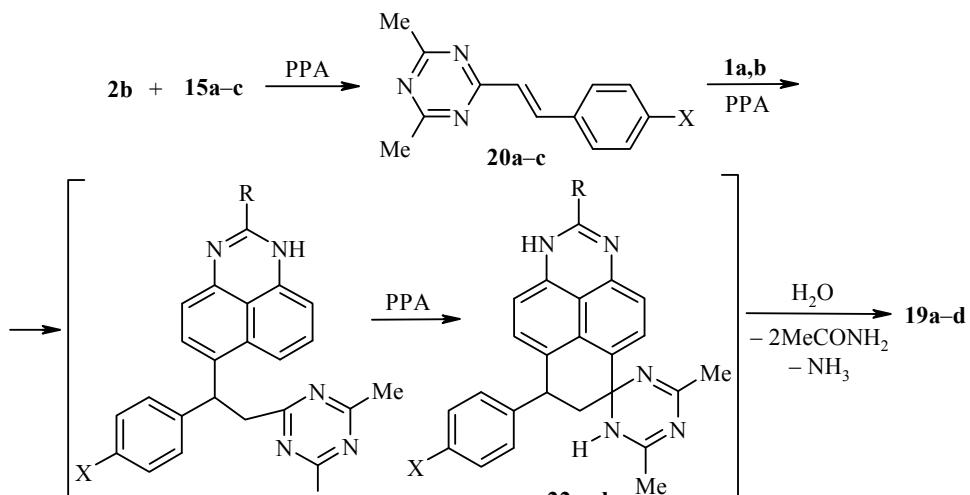


16–18 a R = X = H; b R = Me, X = H; c R = Ph, X = H; d R = H, X = Br; e R = H, X = NO₂

The reaction of perimidines **1a,b** with 2,4,6-trimethyl-1,3,5-triazine (**2b**) in the presence of aromatic aldehydes **15a-c** occurs differently to the 1,3,5-triazine (**2a**). In this case 8(6)-aryl-6(8)-oxo-1,6,7,8-tetrahydro-1,3-diazapyrenes **19a-d** were formed in 76–84% yield.



19 a R = X = H; b R = Me, X = H; c R = H, X = Br; d R = H, X = NO₂



20a, 21–22a,b X = H; 20b, 21–22c X = Br; 20c, 21–22d X = NO₂;
 21–22 a R = H, b R = Me, c R = H, d R = H

The results obtained can be explained in the following way: aldehydes **15a-c** react more rapidly with triazine **2b** than the latter reacts with perimidines **1a,b**. This results in the formation of triazines **20a-c** which can alkylate compounds **1a,b** to give the intermediate compounds **21a-d**. Further cyclization gives the spiro compounds **22a-d** which hydrolyze to give the 1,3-diazapyrene derivatives **19a-d**.

Hence the three-component reaction we have discovered for perimidines with triazines in the presence of carbonyl compounds in PPA depending on the structure of the triazine and carbonyl compound allows to obtain 1,3-diazapyrenes, 8(6)-aryl-6(8)-oxo-1,6,7,8-tetrahydro-1,3-diazapyrenes, and 1,3,7-triazapyrenes.

EXPERIMENTAL

¹H and ¹³C NMR spectra were recorded on a Bruker AM-300 instrument (300 and 75 Hz respectively) with TMS as internal standard. Monitoring of the reaction course and the purity of the compounds synthesized was carried out on Silufol UV-254 plates with ethyl acetate as solvent. Column chromatography was performed on L 40/100 silica gel with ethyl acetate as eluent. PPA with 86% P₂O₅ was obtained by method [35]. 1,3,5-Triazine (**2a**) and 2,4,6-triphenyl-1,3,5-triazine (**2c**) were commercial reagents from Aldrich and the 2,4,6-trimethyl-1,3,5-triazine (**2b**) was prepared by method [36].

1,3-Diazapyrenes 10a-m (General Method). A mixture of the perimidine **1a-c** (1 mmol), the 1,3,5-triazine **2a-c** (2 mmol), and the carbonyl compound (3 mmol, 10 mmol in the case of vinyl butyl ether) in PPA (3-4 g) was vigorously stirred at 60-70°C for 9 h. The reaction mixture was poured into water (30 ml), basified using ammonia solution to pH 7-8, and the precipitated crystals or the oil formed on cooling were extracted with ethyl acetate (3×50 ml). The solvent was evaporated and the residue was chromatographed. The melting points of compounds **10f,g,i** were identical to those reported in [26], of compounds **10a-e,h,j** in [32], and compounds **10k-m** in [37]. The IR spectra of compounds **10d,e,h,j** agreed with those given in [32]. The ¹H NMR spectra of compounds **10a-j** were identical to those published in [32] and of compounds **10k-m** in [37]. The ¹³C NMR spectra for compounds **10a-e,h,j** were identical to those published in [32]. Samples of compounds **10f,g,i,k-m** did not show a depression of melting point when mixed with a known sample.

6-Methyl-1,3-diazapyrene (10a). Yield 0.102 g (47%).

6,8-Dimethyl-1,3-diazapyrene (10b). Yield 0.104 g (45%).

6-Phenyl-1,3-diazapyrene (10c). Yield 0.21 g (75%).

1-(6-Methyl-1,3-diazapyren-7-yl)ethanone (10d). Yield 0.148 g (57%).

Ethyl 6-Methyl-1,3-diazapyrene-7-carboxylate (10e). Yield 0.125 g (43%).

6-Methyl-8-phenyl-1,3-diazapyrene (10f). Yield 0.215 g (73%).

6,8-Diphenyl-1,3-diazapyrene (10g). Yield 0.153 g (43%).

Ethyl 2,6-Dimethyl-1,3-diazapyrene-7-carboxylate (10h). Yield 0.173 g (57%).

2,6,8-Triphenyl-1,3-diazapyrene (10i). Yield 0.22 g (51%).

7-Acetyl-6,8-dimethyl-1,3-diazapyrene (10j). Yield 0.101 g (37%).

1,3-Diazapyrene (10k). Yield 0.037 g (18%).

2-Methyl-1,3-diazapyrene (10l). Yield 0.048 g (22%).

2-Phenyl-1,3-diazapyrene (10m). Yield 0.064 g (23%).

1,3,7-Triazapyrenes (12a-e) (General Method). A mixture of perimidine **1a-c** (1 mmol), 1,3,5-triazine (**2a**) (0.162 g, 2 mmol), and the carbonyl compound (2 mmol) or benzonitrile (0.515 g, 5 mmol) in PPA (3-4 g) was vigorously stirred over 9 h at 60-70°C. The reaction mixture was poured into water (30 ml), basified using ammonia solution to pH 7-8, and the precipitated crystals formed after cooling were filtered off, dried, and purified by recrystallization. The melting points and the ¹H and ¹³C NMR spectra for compounds **12a-c** were identical to those published in [32].

6-Phenyl-1,3,7-triazapyrene (12a). Yield 0.211 g (75%) in the reaction with benzonitrile and 0.256 g (91%) in the reaction with benzaldehyde.

2-Methyl-6-phenyl-1,3,7-triazapyprene (12b). Yield 0.209 g (71%) in the reaction with benzonitrile and 0.257 g (87%) in the reaction with benzaldehyde.

2,6-Diphenyl-1,3,7-triazapyprene (12c). Yield 0.264 g (74%) in the reaction with benzonitrile and 0.314 g (88%) in the reaction with benzaldehyde.

6-(4-Bromophenyl)-1,3,7-triazapyprene (12d). Yield 0.320 g (89%); mp 242-244°C (EtOAc). ^1H NMR spectrum (CDCl_3), δ , ppm (J , Hz): 7.61 (2H, d, J = 8.8, H-3,5 Ar); 7.95 (2H, d, J = 8.8, H-2,6 Ar); 8.30 (1H, d, J = 9.5, H-9); 8.34 (1H, d, J = 9.2, H-5); 8.74 (1H, d, J = 9.5, H-10); 8.87 (1H, d, J = 9.2, H-4); 9.69 (1H, s, H-8); 9.89 (1H, s, H-2). Found, %: C 63.48; H 2.73; N 11.71. $\text{C}_{19}\text{H}_{10}\text{BrN}_3$. Calculated, %: C 63.35; H 2.80; N 11.67.

6-(4-Nitrophenyl)-1,3,7-triazapyprene (12e). Yield 0.271 g (83%); mp 178-180°C (EtOAc). ^1H NMR spectrum (CDCl_3), δ , ppm (J , Hz): 7.96 (2H, d, J = 8.7, H-2,6 Ar); 8.31 (1H, d, J = 9.5, H-9); 8.35 (1H, d, J = 9.2, H-5); 8.42 (2H, d, J = 8.7, H-3,5 Ar); 8.77 (1H, d, J = 9.5, H-10); 8.89 (1H, d, J = 9.2, H-4); 9.78 (1H, s, H-8); 9.91 (1H, s, H-2). Found, %: C 70.06; H 3.01; N 17.24. $\text{C}_{19}\text{H}_{10}\text{N}_4\text{O}_2$. Calculated, %: C 69.94; H 3.09; N 17.17.

8(6)-Aryl-6(8)-oxo-1,6,7,8-tetrahydro-1,3-diazapypenes 19a-d (General Method). A mixture of perimidine **1a,b** (1.0 mmol), 2,4,6-trimethyl-1,3,5-triazine (**2b**) (0.185 g, 1.5 mmol), and the aromatic aldehyde (1.5 mmol) in PPA (3-4 g) was vigorously stirred for 8 h at 60-70°C. The reaction mixture was poured into water (30 ml), refluxed for 5 min, cooled, and basified using ammonia solution to pH 7-8. The crystals formed after cooling were filtered off, dried, and recrystallized from EtOAc. The melting points and ^1H NMR spectra of compounds **19a,c** were identical to those published in [27]. A sample mixed with a known sample did not show a depression in melting point.

6(8)-Oxo-8(6)-phenyl-1,6,7,8-tetrahydro-1,3-diazapyprene (19a). Yield 0.250 g (84%).

2-Methyl-6(8)-oxo-8(6)-phenyl-1,6,7,8-tetrahydro-1,3-diazapyprene (19b). Yield 0.243 g (78%); mp 151-152°C (EtOAc). ^1H NMR spectrum (DMSO-d_6), δ , ppm (J , Hz): 2.18 (3H, s, CH_3); 2.95-3.01 (2H, m, CH_2); 4.48 (1H, br. dd, J = 5.9, J = 6.4, CHPh); 6.52 (1H, br. d, J = 8.1, H-10); 6.61 (1H, br. d, J = 7.8, H-4); 7.04 (1H, d, J = 7.8, H-5); 7.15-7.30 (5H, m, H Ph); 7.77 (1H, d, J = 8.1, H-9); 11.37 (1H, br. s, NH). Found, %: C 80.92; H 5.11; N 8.89. $\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}$. Calculated, %: C 80.75; H 5.16; N 8.97.

8(6)-(4-Bromophenyl)-6(8)-oxo-1,6,7,8-tetrahydro-1,3-diazapyprene (19c). Yield 0.286 g (76%).

8(6)-(4-Nitrophenyl)-6(8)-oxo-1,6,7,8-tetrahydro-1,3-diazapyprene (19d). Yield 0.278 g (81%); mp 197-198°C (EtOAc). ^1H NMR spectrum (DMSO-d_6), δ , ppm (J , Hz): 3.01 (1H, dd, J = 11.3, J = 6.9) and 3.24 (1H, dd, J = 11.3, J = 7.0, CH_2); 4.70 (1H, br. dd, J = 6.9, J = 7.0, CH); 6.53 (1H, br. d, J = 7.7, H-10); 6.67 (1H, br. d, J = 7.7, H-4); 7.06 (1H, d, J = 7.7, H-5); 7.45 (2H, d, J = 8.4, H-2,6 Ar); 7.80 (1H, d, J = 7.7, H-9); 7.60 (1H, s, H-2); 8.14 (2H, d, J = 8.4, H-3,5 Ar); 11.37 (1H, br. s, NH). Found, %: C 70.11; H 3.75; N 12.16. $\text{C}_{20}\text{H}_{13}\text{N}_3\text{O}_3$. Calculated, %: C 69.97; H 3.82; N 12.24.

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