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The synthesis of *N*-derivatives of 3-aminoperylene and their absorption and fluorescence properties

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ABSTRACT

N-Acetyl and *N*-triazinyl 3-aminoperylenes were prepared. *N*-(4,6-Dichloro-1,3,5-triazin-2-yl)-3-aminoperylene was synthesized by the condensation of 3-aminoperylene with cyanuric chloride; other *N*-triazinyl derivatives were prepared by the successive substitution of chlorine atoms with methoxy or aniline groups. The structure and purity of the compounds were confirmed by elemental analysis, NMR spectroscopy and mass spectrometry. The UV/vis absorption, fluorescence and excitation spectra as well as the fluorescence quantum yields for the compounds were measured in dibutyl ether, 1,4-dioxane, ethyl acetate and acetonitrile; fluorescence lifetimes were measured in ethyl acetate and dimethyl sulfoxide. The influences of both the character of the *N*-substituent and the solvent polarity upon the spectra and quantum yields are discussed.

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1. Introduction

In connection with the study of excitation energy transfer (EET) and relationships between structure and fluorescence characteristics of organic dyes, we are interested in spectral and photophysical properties of bi- and trichromophoric compounds with 1,3,5-triazine (T) as a spacer. Earlier we described [1] the spectral and photophysical properties of bichromophoric compounds of D–T–A type with 1-aminopyrene as the donor (D) of EET and 3-aminobenzanthrone as the acceptor (A) of EET.

A better understanding of EET in this system needed the investigation of the role of photophysical properties of 3-aminobenzanthrone and 1-aminopyrene triazinyl derivatives in such a system. In previous papers [2,3] we reported on the synthesis and solvent effect on absorption and fluorescence spectra, fluorescence lifetime and fluorescence quantum yield of *N*-triazinyl derivatives of 3-aminobenzanthrone. Recently, we have described spectral and photophysical characteristics of *N*-triazinyl derivatives of 1- and 2-aminopyrenes [4]. Presently we are studying the properties of the bichromophore of the D–T–A type with 3-aminoperylene as the EET acceptor. In this paper, we present the synthesis and the absorption and fluorescence spectra and fluorescence quantum yields (q_F) of some *N*-triazinyl derivatives of 3-aminoperylene.

The syntheses of 3-nitroperylene [5–9] and 3-aminoperylene [10] are described in the literature to date; the synthesis of *N*-derivatives of 3-aminopyrene has not been described yet.

2. Exprimental

2.1. Synthesis

Cyanuric chloride, perylene and aniline (Fluka products) were used without purification. The course of the reactions and purity of the substances were checked by TLC, HPLC, and by comparison of fluorescence excitation spectra with absorption spectra of the final substances. Chemical structures were confirmed using MS and ^{1}H and ^{13}C NMR spectra and elemental analysis. The relevant scheme for the synthesis of the compounds is shown in Fig. 1.

2.1.1. 3-Nitroperylene (**1**)

Compound **1** was prepared by the nitration of perylene with fuming nitric acid in 1,4-dioxan according to the procedure described by Dugarova et al. [5]; m.p. 208–210 °C (210–212 °C) [5].

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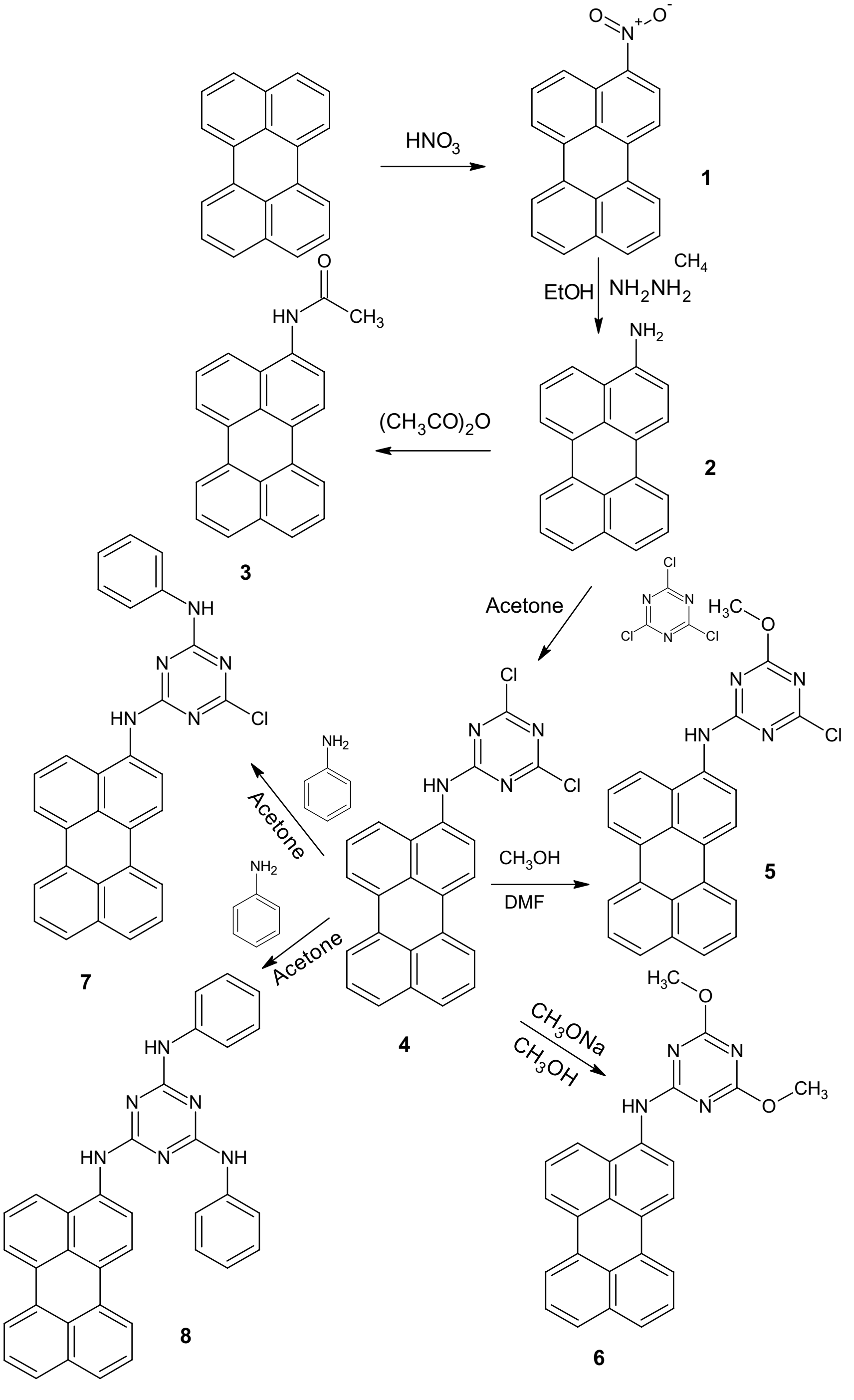

Fig. 1. Synthesis of 3-aminoperylene and its derivatives.

2.1.2. *3-Aminoperylene (**2**)*

Compound **1** (5 g, 0.0168 mol), $FeCl_3$ (0.17 g, 0.001 mol) and activated charcoal (0.33 g) were added to a mixture of toluene (25 mL) and absolute ethanol (73 mL). The reaction mixture was heated with stirring to boiling and hydrazine hydrate (10 mL, 0.2 mol) was added dropwise. Then the reaction mixture was refluxed for 3 h. After the completion of the reaction, charcoal was filtered off. Removal of the solvent under reduced pressure gave **2**, which was crystallized from ethyl acetate. Yield 1.4 g (32%); m.p. 228–232 °C (220–230 °C) [10]. MS: ($APCI^+$): m/z 268 $[M + H]^+$ (M.W. 267).

^1H chemical shifts: 8.27 (1H, d), 8.20 (1H, d), 8.12 (1H, d), 8.02 (2H, d), 7.67 (1H, d), 7.54 (1H, d), 7.42 (3H, m), 6.83 (1H, d), 6.19 (2H, br s, NH_2).

2.1.3. N*-Acetyl-3-aminoperylene (**3**)*

Compound **2** (1 g, 0.0037 mol) was dissolved in DMSO (10 mL), and acetic anhydride (1.25 mL) was added thereto. The reaction mixture was heated in a boiling water bath for 5 h, whereupon the mixture was diluted with cold water (60 mL). The precipitated solid was collected by filtration and crystallized from xylene, yielding 1.1 g (90.6%) pure **3**; m.p. 295–300 °C. MS: ($APCI^-$): m/z 308

$[M-H]^-$ (M.W. 309). ^1H chemical shifts: 9.99 (1H, br s, CONH), 8.36–8.46 (4H, m), 8.01 (1H, d), 7.80–7.86 (3H, m), 7.64 (1H, t), 7.55 (1H, d), 7.54 (1H, t), 2.25 (3H, s, CH_3). Anal. calcd. for $C_{22}H_{15}NO$: C 85.41%, H 4.89%, N 4.53%; found: C 85.35%, H 4.76%, N 4.61%.

2.1.4. N-*(4,6-Dichloro-1,3,5-triazin-2-yl)-3-aminoperylene (**4**)*

Compound **2** (5 g, 0.019 mol) was dissolved in acetone (150 mL). The solution was cooled to −5 °C in an ice bath and cyanuric chloride (3.5 g, 0.019 mol) and $NaHCO_3$ (2.4 g) were added. The reaction mixture was stirred at 0–10 °C, and the reaction course was monitored by TLC. After 3 h, the product was isolated by filtration, washed with water, dried and crystallized from toluene; yield 6.5 g (82%); m.p. >360 °C. MS: ($APCI^-$): *m/z* 413 $[M-H]^-$ (M.W. 414). ^1H chemical shifts: 11.26 (1H, br s, NH), 8.36–8.44 (4H, m), 7.83–7.88 (3H, m), 7.67 (1H, d), 7.62 (1H, t), 7.56–7.60 (2H, m). ^{13}C chemical shifts: 169.8, 169.2, 165.9, 134.3, 131.7, 131.0, 130.4, 130.2, 130.1, 130.0, 129.8, 128.6, 127.7 (all C), 128.3, 128.2, 127.4, 127.0 (2C), 124.9, 122.9, 121.2 (2C), 121.1, 120.5 (all CH). Anal. calcd. for $C_{23}H_{12}Cl_2N_4$: C 66.52%, H 2.91%, Cl 17.07%, N 13.49%; found: C 66.41%, H 2.85%, Cl 17.15%, N 13.66%.

2.1.5. N-*(4-Chloro-6-methoxy-1,3,5-triazin-2-yl)-3-aminoperylene (**5**)*

Compound **4** (1 g, 0.0024 mol) and $NaHCO_3$ (0.3 g) were added to a mixture of methyl alcohol (10 mL) and DMF (5 mL) at room temperature. The solution was heated to 40–45 °C for 7 h, whereupon it was diluted with water (50 mL) and filtered; yield 0.7 g of **5** (71% of theoretical yield); m.p. 222–226 °C. MS: ($APCI^-$): *m/z* 409 $[M-H]^-$ (M.W. 410). Anal. calcd. for $C_{24}H_{15}ClN_4O$: C 70.16%, H 3.68%, Cl 8.63%, N 13.64%; found: C 70.05%, H 3.72%, Cl 8.74%, N 13.73%.

2.1.6. N-*(4,6-Dimethoxy-1,3,5-triazin-2-yl)-3-aminoperylene (**6**)*

Compound **4** (1 g, 0.0024 mol) was dissolved in methyl alcohol (70 mL) and sodium methoxide solution (10 mL, 0.02 mol) was added. The reaction mixture was refluxed for 4 h, diluted with water (60 mL); the precipitated solid was collected by filtration and dried to give 0.8 g (82%) **6**; m.p. 235–239 °C. MS: ($APCI^-$): *m/z* 405 $[M-H]^-$ (M.W. 406). ^1H chemical shifts: 10.16 (2H, br s, NH), 8.38–8.46 (4H, m), 7.89 (1H, d), 7.84 (2H, m), 7.72 (1H, d), 7.57–7.62 (3H, m), 3.88 (OCH_3). ^{13}C chemical shifts: 172.2 (2C), 167.9, 134.4, 133.7, 130.8, 130.5, 130.4, 130.1, 128.7, 128.4, 127.7 (all C), 128.1, 127.8, 127.1, 127.0, 126.8, 124.4, 123.4, 121.0, 120.9, 120.7, 120.5 (all CH), 54.4 (OCH_3). Anal. calcd. for $C_{25}H_{18}N_4O_2$: C 0.73.88%, H 4.46%, N 13.78%; found: C 74.09%, H 4.57%, N 13.88%.

2.1.7. N-*(4-Chloro-6-anilino-1,3,5-triazin-2-yl)-3-aminoperylene (**7**)*

Compound **4** (1 g, 0.0024 mol) was added to acetone (75 mL). The suspension was cooled to −5 °C in an ice bath, whereupon aniline (0.22 mL, 0.024 mol) was added dropwise with stirring. The reaction mixture was stirred for 16 h at the temperature of about 5 °C, and then diluted with water (30 mL) and filtered. Yield 1 g (88%) **7**; m.p. 230–232 °C. MS: ($APCI^-$): *m/z* 470 $[M-H]^-$ (M.W. 471). Anal. calcd. for $C_{29}H_{18}ClN_5$: C 73.80%, H 3.84%, Cl 7.51%, N 14.84%; found: C 74.01%, H 3.95%, Cl 7.58%, N 14.93%.

2.1.8. N-*(4,6-Dianilino-1,3,5-triazin-2-yl)-3-aminoperylene (**8**)*

Compound **4** (1 g, 0.0024 mol) and aniline (0.44 mL, 0.0048) were added to acetone (90 mL). The reaction mixture was refluxed with stirring for 4.5 h, whereupon it was diluted with water (50 mL) and filtered. Yield 1.2 g (94%) **8**; m.p. 168–170 °C. MS: ($APCI^-$): *m/z* 527 $[M-H]^-$ (M.W. 528). ^1H chemical shifts: major form: 10.36 (2H, br s, NH), 8.43–8.52 (4H, m), 7.86 (2H, m), 7.72 (1H, d), 7.59–7.67 (3H, m), 7.31 (3H, br s), minor form: 10.50 (2H, br s, NH), 8.01 (1H, d), 7.77 (1H, m), 7.10 (br s), other signals were overlapped. Anal. calcd. for $C_{35}H_{24}N_6$: C 79.53%, H 4.58%, N 15.90%; found: C 79.61%, H 4.65%, N 15.99%.

2.2. *Spectra*

The mass spectra were measured on an LC/MS system LC-MSD Trap XCT Plus (Agilent Technologies) using a direct infusion measurement. Negative and positive-ion APCI mass spectra were recorded in the mass range of 50–1500 Da. The ion trap analyzer was tuned to obtain an optimal response in the range of expected *m/z* values. The other APCI ion source parameters were as follows: drying gas flow rate 7 L/min, nebulizer gas pressure 60 psi, drying gas temperature 350 °C. The samples were dissolved in acetonitrile for HPLC (Sigma–Aldrich) in concentrations appropriate for MS detection.

The ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance 500 instrument operating at 500.13 MHz for ^1H and 125.76 MHz for ^{13}C in hexadeuteriodimethyl sulfoxide and measured at room temperature. The ^1H and ^{13}C chemical shifts were referenced to the central signal of the solvent ($\delta = 2.55$ (^1H) and 39.6 (^{13}C)). The positive values of chemical shifts denote shifts of signals to higher frequencies with respect to the standard.

The absorption spectra were measured on a Perkin–Elmer Lambda 35 UV/vis spectrophotometer. Typical concentrations were of the order of 10^{-5} mol/L, which yielded optical density of solutions 0.4–0.8 at the main absorption maximum in a 1-cm cell.

The steady-state fluorescence spectra were measured using a Hitachi Perkin–Elmer LS 55 spectrophotometer. The instrument provides corrected excitation spectra directly; the fluorescence emission spectra were corrected for the characteristics of the emission monochromator and for the detection photomultiplier response. For fluorescence measurements, very weakly absorbing solutions (optical density ~0.05 at the exciting wavelength in 1-cm cell) were used. The fluorescence quantum yields (q_{Fl}) were measured using quinine sulfate ($q_{Fl} = 0.54$ in 0.5 mol/L H_2SO_4) [11] as the standard. Deaeration of the samples by bubbling through N_2 or Ar did not make any difference in the spectra and quantum yield recorded; therefore, the data reported here correspond to aerated solutions.

The samples for absorption spectra and fluorescence measurements were prepared by preparative TLC on Silufol UV 254 plates. The purity of measured substances was confirmed by coincidence of absorption and excitation spectra. All the solvents used were of spectroscopic grade and were checked for their own fluorescence under relevant conditions.

The fluorescence kinetics was recorded on Edinburgh Analytical Instruments FS/FL spectrophotometer that employs time-correlated single photon counting (TCSPC) detection. Pulse diodes IBH Nanoled-07 (405 nm p laser diode) (for *N*-derivatives) and IBH Nanole-05A (455 nm n LED) (for **2**) were used as the excitation sources. The instrument response function FWHM was <150 ps for 405 nm laser diode and <1.5 ns for 455 nm diode. The fluorescence lifetimes were obtained by multiexponential reconvolution fitting process.

3. Results and discussion

3.1. *Synthesis*

The starting **1** was prepared by nitration of perylene according to the literature procedure [5]. The reduction of **1** to **2** was performed according the method successfully used for reduction of 1-nitropyrene to 1-aminopyrene [12]. The obtained **2** was of high purity; the melting point was in accordance with the literature [10].

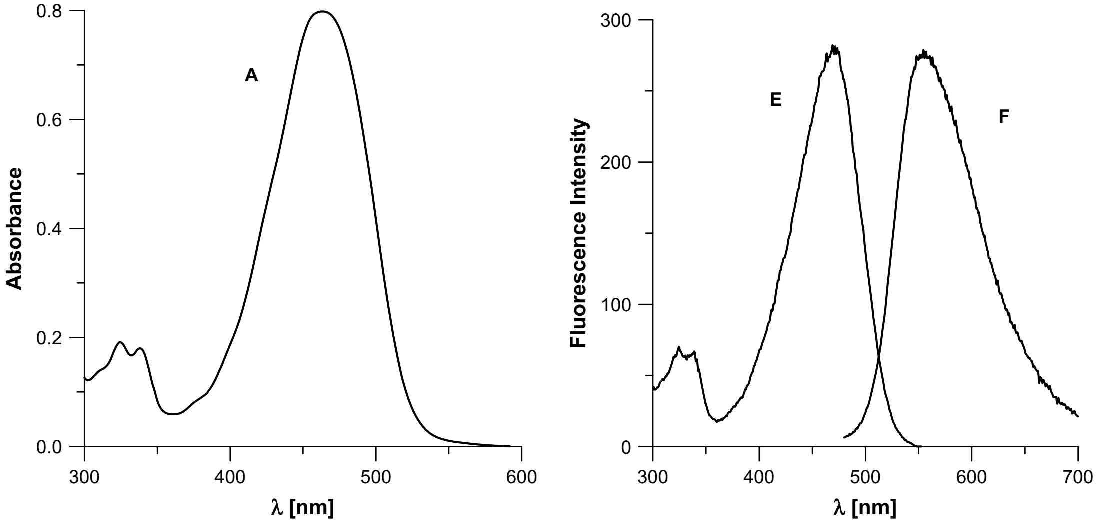

Fig. 2. Absorption (A), fluorescence (F, exc. 463 nm) and fluorescence excitation (E, em. 555 nm) spectra of **2** in ethyl acetate.

Table 1
The absorption (λ_A) and fluorescence (λ_F) maxima [nm].

Compound	Solvent							
	Dibutylether		Dioxane		Ethyl acetate		Acetonitrile	
	λ_A	λ_F	λ_A	λ_F	λ_A	λ_F	λ_A	λ_F
2	455	540	464	549	463	555	468	564
3	453	471	447	471	451	471	441	471
4	443	475	446	472	442	469	443	475
5	443	477	443	479	442	477	444	482
6	442	477	443	478	441	477	444	479
7	443	476	444	478	442	475	442	477
8	444	486	444	487	443	488	444	492

Compound **3** was synthesized by treatment of **2** with acetic anhydride according to a literature procedure for synthesis of *N*-acetyl-2-aminopyrene with a slight modification [4]. The product was obtained in a high yield and purity.

Compound **4** was obtained by the condensation of equimolar amounts of cyanuric chloride and **2** in acetone and in the presence of sodium hydrogen carbonate as a base at the temperature of 0–10 °C. The condensation started at 0°, and then the temperature was gradually increased to 10 °C. A precipitate gradually formed during the course of reaction and this was isolated giving the first portion of product of high purity. The second portion of product was obtained by dilution of the mother liquor with water. This product was contaminated with cyanuric chloride which was removed by crystallization from toluene.

The situation becomes somewhat more complicated with the selective substitution of second chlorine atom on the triazine ring of **4** by anilino group. The problem consists in the substitution of both chlorine atoms even under mild conditions. However, starting the reaction at the temperature of −5 °C and gradually increasing the temperature up to 5 °C in the course of the reaction led to a selective substitution of one chlorine atom. The substitution of both chlorine atoms by two aniline or two methoxy groups in boiling acetone or in boiling methanol, respectively, led to **8** and **6** of high purity.

3.2. Absorption and fluorescence properties

The first absorption band of **2** corresponds to the transition from electronic ground state (S_o) to S_1 ($\pi\pi^*$) excited state with a main

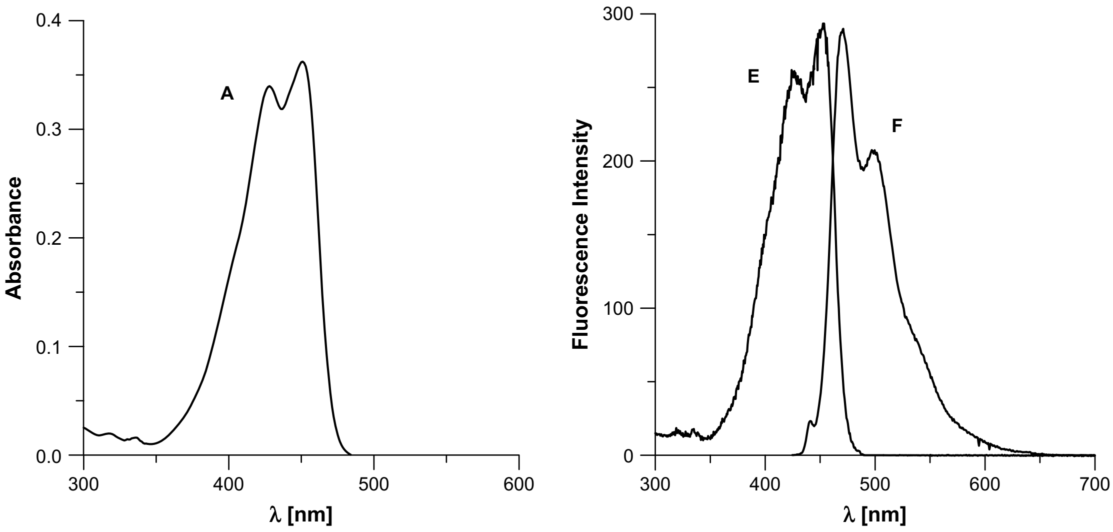

Fig. 3. Absorption (A), fluorescence (F, exc. 451 nm) and fluorescence excitation (E, em. 471 nm) spectra of **3** in ethyl acetate.

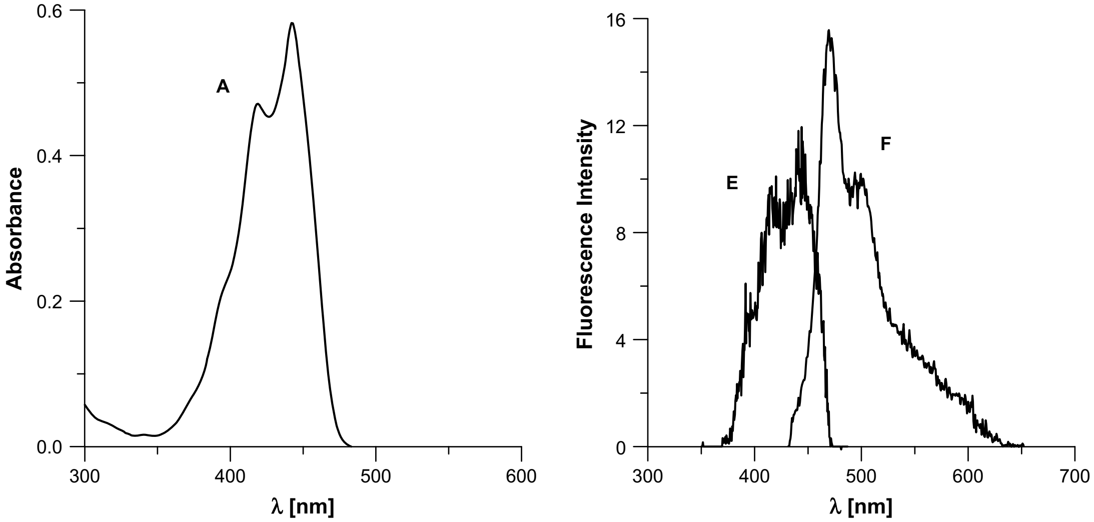

Fig. 4. Absorption (A), fluorescence (F, exc. 442 nm) and fluorescence excitation (E, em. 500 nm) spectra of **4** in ethyl acetate.

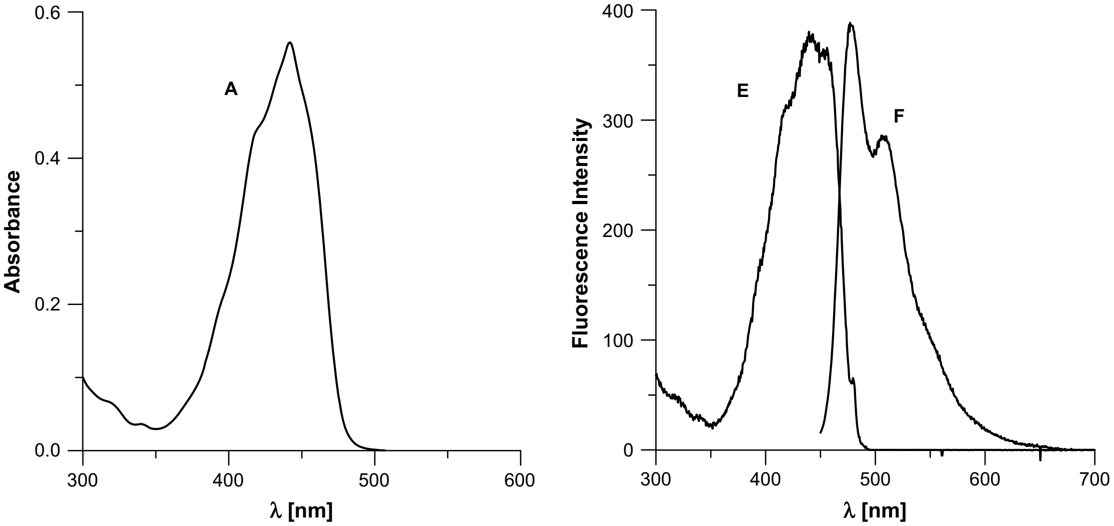

Fig. 5. Absorption (A), fluorescence (F, exc. 442 nm) and fluorescence excitation (E, em. 477 nm) spectra of **5** in ethyl acetate.

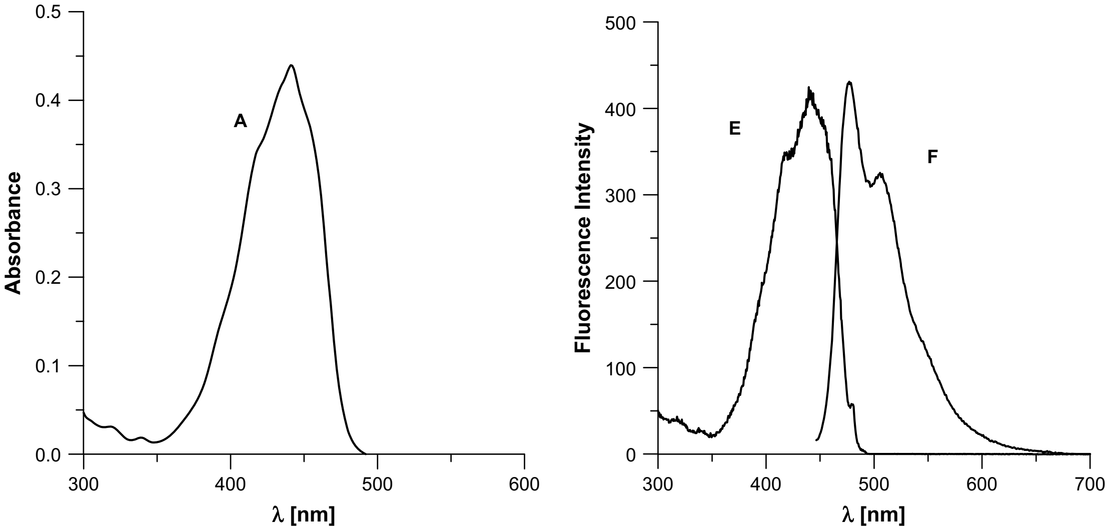

Fig. 6. Absorption (A), fluorescence (F, exc. 441 nm) and fluorescence excitation (E, em. 477 nm) spectra of **6** in ethyl acetate.

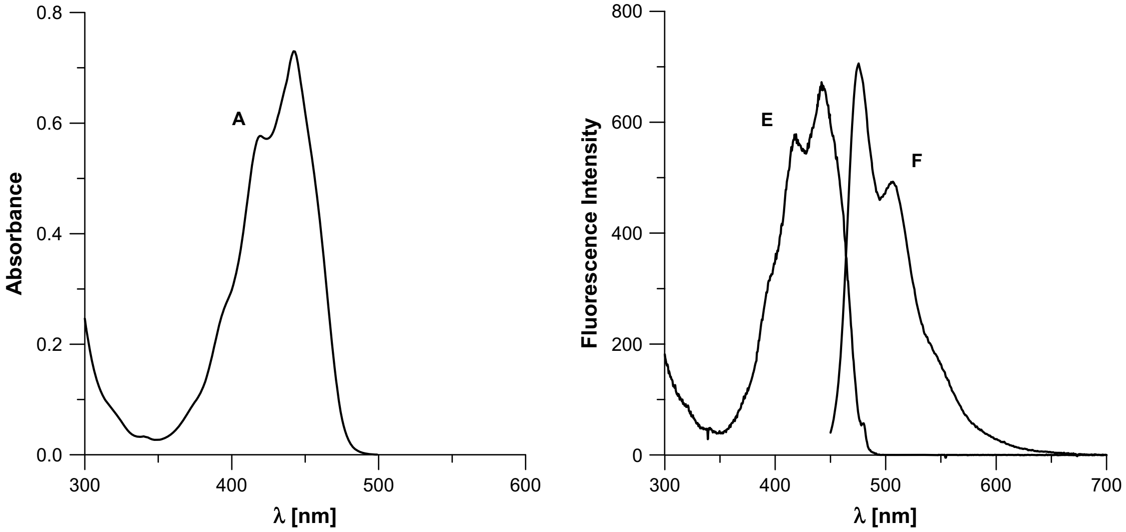

Fig. 7. Absorption (A), fluorescence (F, exc. 442 nm) and fluorescence excitation (E, em. 475 nm) spectra of **7** in ethyl acetate.

monoexcited configuration 1,1′ (HOMOnbsp;→ LUMO) and with a high oscillator strength [13]. In contrast to perylene, this band is broad, structureless and bathochromically shifted (Fig. 2); the shift is not large and increases with solvent polarity (Table 1). 1,4-Dioxane (DO) behaves like a more polar solvent even though its dielectric constant (2.22) is smaller than that of dibutyl ether (DBE) (3.08); it could be caused by quadrupole moments that contribute significantly to solvation (similar behaviour was observed for toluene and chlorbenzene) [3].

Owing to a decrease in conjugation of the amino 2 p_π electrons with the perylene π-system caused by a substitution on the amino group with an electron-withdrawing acetyl or triazinyl group, a hypsochromic shift of the absorption bands with a fairly well defined vibronic structure was recorded (Figs. 3–8). The position of absorption maxima of *N*-derivatives is not influenced by solvent polarity or by a substituent on triazinyl ring (Table 1).

In comparison with *N*-derivatives, **2** shows a larger Stokes shift in all solvents used. Contrary to the observation noted for the position of the absorption bands, the position of fluorescence maxima of *N*-derivatives is more sensitive to a type of *N*-substituent (Table 1); a feature which may be connected with different changes of geometry and polarity of emitting states.

The fluorescence spectra of presented compounds are approximate mirror images of the first absorption bands, suggesting that the state formed upon excitation into the absorption band is the originating state for the fluorescence. The fluorescence excitation spectra are almost identical with corresponding absorption spectra suggesting that the absorbing and emitting species are identical; furthermore, this feature supports the high purity of the presented compounds.

The fluorescence kinetics was recorded near the center of the fluorescence emission band. The decay of the fluorescence of the studied compounds is monoexponential with the fluorescence lifetimes ranging between 3.4 and 5.1 ns in ethyl acetate and 3.9–5.6 ns in dimethyl sulfoxide (DMSO) (Table 3). The fluorescence lifetime of **4** could not be determined because of the low fluorescence quantum yield. The results show that the fluorescence lifetimes, similar to the position of the fluorescence maxima, are almost independent on the type of *N*-substituent. The order of magnitude of the radiation lifetime can be estimated from the

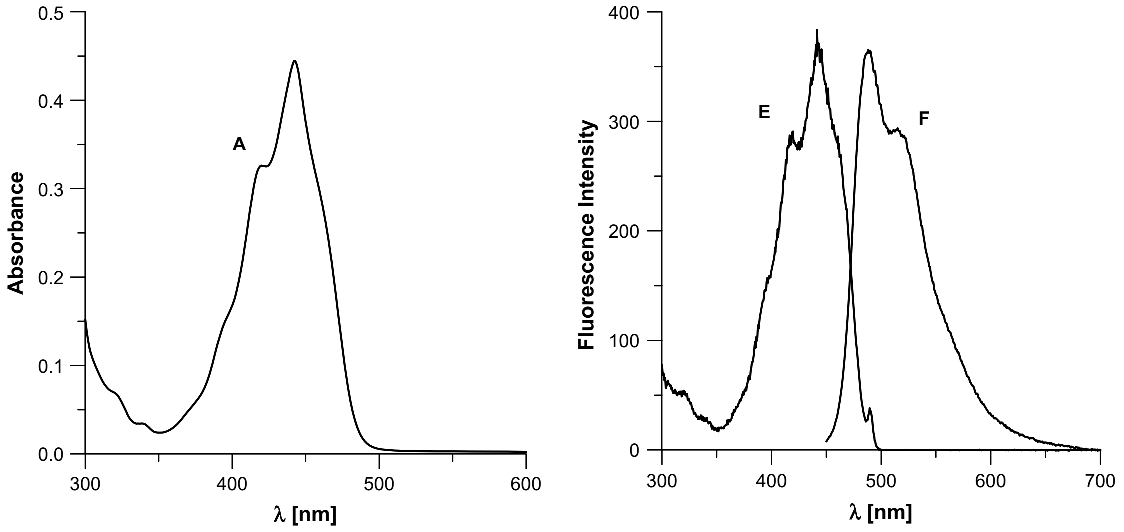

Fig. 8. Absorption (A), fluorescence (F, exc. 447 nm) and fluorescence excitation (E, em. 488 nm) spectra of **8** in ethyl acetate.

Table 2
Fluorescence quantum yields (q_{Fl}) of the prepared compounds.

Solvent	**2**	**3**	**4**	**5**	**6**	**7**	**8**
DBE	0.24	0.46	0.12	0.37	0.46	0.49	0.50
DO	0.28	0.56	0.068	0.40	0.51	0.53	0.52
EtAc	0.20	0.50	0.014	0.33	0.47	0.43	0.44
MeCN	0.11	0.45	0.011	0.32	0.43	0.45	0.42

Table 3
Fluorescence lifetimes of **2** and its *N*-substituted derivatives.

Compound	Fluorescence lifetime [ns]		Compound	Fluorescence lifetime [ns]	
	EtAc	DMSO		EtAc	DMSO
2	5.1	5.6	**6**	3.5	4.0
3	3.8	4.5	**7**	3.4	3.9
5	3.5	4.0	**8**	3.6	4.3

relationship $\tau_o \sim 10^{-4}/\varepsilon_{max}$ for absorption bands in the near UV. Thus allowed transitions (strong absorption bands, $\varepsilon \sim 10^5$) will give rise to excited states having an approximate lifetime of $\sim 10^{-9}$ s (ns). The presented lifetimes show (Table 3), that all studied compounds fluoresce from strong absorption band corresponding to the allowed $\pi\pi^*$ transition.

The q_{Fl} of the studied compounds is also influenced by the character of *N*-substituents and by solvent polarity (Table 2):

- the q_{Fl} of **2** is reduced about 2.5 times in acetonitrile (MeCN);
- N-acetylation causes an increase of q_{Fl} in all the solvents;
- a substitution on amino group by cyanuric chloride causes a dramatic drop of q_{Fl} of **4** in all the solvents; a fall of q_{Fl} is more expressive in acetonitrile (dielectric constant 35.94); a polar character of 1,4-dioxane (2.22), caused by qauadrupole moments, is revealed by comparing the q_{Fl} of **4** in this solvent with that in dibutyl ether (3.08);
- a substitution of one or both chlorine atoms on triazinyl ring by anilino or methoxy groups results in high q_{Fl} of all the derivatives in all the solvents.

The number of chlorine atoms on triazinyl ring is the determining factor for q_{Fl} of *N*-triazinyl derivatives. This phenomenon is similar to that for *N*-triazinyl derivatives of aminopyrenes [4]. The strong fluorescence quenching of dichlorotriazinyl derivatives is the most remarkable feature. The presence of chlorine atoms could enhance the spin–orbit coupling, and so it could increase the S–T transition probability. However, 1-(dichloro-1,3,5-triazinyl)pyrene exhibits a high q_{Fl} in non-polar and polar solvents [14]. This fact rules out an S–T transition as an efficient deactivation nonradiative process. As the chlorine atoms enhance the electron-withdrawing character of the triazinyl ring, an excited state connected with π-electron transfer from the aminoperylene (aminopyrene) moiety to the triazinyl ring could exist (CT state). Due to a solvent relaxation, the energy of such a state could decrease to the vicinity or even below that of a fluorescent state in polar solvents. Hence, a new efficient deactivation channel could be opened. For an explanation of relationships between structure and q_{Fl} of the described compounds, quantum chemical calculations are now in progress in our laboratory.

4. Conclusions

The aim of this work was the syntheses and the investigation of fluorescence characteristics of the hitherto unknown *N*-acetyl and *N*-triazinyl derivatives of 3-aminoperylene.

The structure of the prepared compounds was confirmed by elemental analysis, MS and NMR spectra.

The UV/vis absorption and fluorescence spectra, fluorescence quantum yields and lifetimes were measured in several solvents. It has been found that the character of *N*-substituent does not influence the position and the shape of absorption and fluorescence spectra; a small effect on fluorescence lifetimes was also observed. However, a strong effect of character of *N*-substituent on fluorescence quantum yield of compounds has been found; the presence of chlorine atoms on the triazine ring is most important for fluorescence quenching.

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