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> ORGANIC SYNTHESIS AND INDUSTRIAL ORGANIC CHEMISTRY

# Synthesis, Absorption Spectra, and Luminescence Properties of Dihydrobenzoacridinone Derivatives

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**Abstract**—9,9-Dimethyl-12-[(5-aryl-2-furyl) (or 5-aryl-2-thienyl, or 5-aryl-1-methyl-1*H*-pyrrolyl)]-7,8,9,10,11,12-hexahydrobenzo[*a*]acridin-11-ones were synthesized, and their absorption and luminescence spectrum characteristics in ethanol at room temperature and at 77 K were studied. The spectra suggest the existence of these compounds in a liquid solution as mixtures of conformers, each of which is characterized by its own absorption and fluorescence spectra.

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Proceeding with studies on synthesis of organic luminophores containing carbonyl group [1], we synthesized new compounds with the carbonyl group incorporated in the dihydrobenzoacridine core, following the procedure that we developed (Scheme 1).

Refluxing equimolar amounts of 2-naphthylamine I, appropriate aldehyde IIa–IIf, and dimedone IV in an alcoholic solution led to selective formation of 9,9-dimethyl-12-[(5-aryl-2-furyl) (or 5-aryl-2-thienyl, or 5-aryl-1methyl-1*H*-pyrrolyl)]-7,8,9,10,11,12-hexahydrobenzo[a] acridin-11-ones Va–Vf.

Dihydrobenzoacridinones **Va–Vd** can be prepared both by the reaction of azomethines **IIIa–IIId** with 2-naphthylamine and by the ternary condensation of 2-naphthylamine, aldehydes **IIa–IId**, and dimedone **IV**. Dihydrobenzoacridinones **Ve** and **Vf** can be prepared only by cascade condensation of 2-naphthylamine, aldehydes **IIe** and **IIf**, and dimedone, because in reaction of 2-naphthylamine with *N*-methyl-5-arylpyrrole-2-carbaldehydes **IIe** and **IIf** we failed to isolate the pure azomethines from the reaction mixture. Azomethine **IIIa–IIId**, according to the suggested reaction scheme, adds a dimedone molecule to form unstable intermediate **A**, which then decomposes into arylidene diketone **B** and 2-naphthylamine. In 2-naphthylamine, the electron density is the highest at the  $\alpha$ -carbon atom relative to the amino group, and it undergoes addition via this atom to the double bond of arylidene diketone **B** to form compound **C** whose heterocyclization with water elimination yields the final products, 9,9-dimethyl-12-[(5-aryl-2-furyl) (or 5-aryl-2-thienyl, or 5-aryl-1-methyl-*IH*-pyrrolyl)]-7,8,9,10,11,12-hexahydrobenzo[*a*]acridin-11-ones **Va–Vf**. The possibility of the occurrence of such transformations in the course of ternary condensation of aromatic aldehydes, 1,3-diketones, and 2-naphthylamine was reported in [2, 3].

The physicochemical constants of the synthesized dihydrobenzoacridinones **Va–Vf** are given in Table 1.

The <sup>1</sup>H NMR spectra of dihydrobenzoacridinones **Va–Vf** in all the cases are superpositions of the spectra of different fragments and consist of three fragments: (1) seven-spin system of aromatic protons belonging to



Scheme 1. Scheme of the synthesis of dihydrobenzoacridine derivatives



ÇH<sub>3</sub>

R

 $X = O, R = R = 4 - NO_2$  (a), 2-NO<sub>2</sub>-4-OCH<sub>3</sub> (b);  $X = S, R = 4 - NO_2$  (c), 3-NO<sub>2</sub> (d);  $X = N - CH_3, R = 4 - Br$  (e), 2-Br (f).

the naphthalene core, AB system of the heteroaromatic ring, and four-spin system AA'BB' or ABCD depending on the substituent position in the aromatic ring; (2) two AB systems of the dimedone moiety in the aliphatic region; and (3) singlet from H<sup>12</sup> proton at 5.45–6.11 ppm. The positions and multiplicities of the proton signals are typical of the <sup>1</sup>H NMR spectra of related compounds [4].

ÇH<sub>3</sub>

10

I

In the IR spectra of dihydrobenzoacridinones Va-Vf, there are absorption bands of free (3440-3400 cm<sup>-1</sup>) and associated (3275-3257 cm<sup>-1</sup>) amino groups. Such pattern is observed at incomplete association of amines.

The electronic absorption spectra of the synthesized compounds in ethanol are characterized by a set of vibronic bands in the UV range (Fig. 1, Table 2), which can be subdivided into three groups. The first group includes bands in the range 200-250 nm with high molar extinction coefficient  $\varepsilon$  (30000–80000 L mol<sup>-1</sup> cm<sup>-1</sup>); the second group, bands at 250-320 nm (10000- $38\,000 \text{ L} \text{ mol}^{-1} \text{ cm}^{-1}$ ; and the third group, bands in the range 320-450 nm (11 000-25 000 L mol<sup>-1</sup> cm<sup>-1</sup>).

Each of these groups can be assigned to electronic transitions  $S_n \leftarrow S_0$  (n = 1-3) characterized by the cor-

Compound	Yield, %	T <sub>m</sub> , °C	Found, %			Formula	Calculated, %		
			С	Н	N	Formula	С	Н	N
Va	70	310-312	75.00	5.26	5.97	$C_{29}H_{24}N_2O_4$	74.98	5.21	6.03
Vb	62	288–290	80.21	6.00	3.16	C <sub>30</sub> H <sub>27</sub> NO <sub>5</sub>	80.15	6.05	3.12
Vc	60	279–280	72.52	5.07	5.78 S 6.70	$C_{29}H_{24}N_2O_3S$	72.48	5.03	5.83 S 6.67
Vd	47	210–212	72.42	5.00	5.81 S 6.67	$C_{29}H_{24}N_2O_3S$	72.48	5.03	5.83 S 6.67
Ve	50	>320	70.20	5.47	5.33 Br 15.38	C <sub>30</sub> H <sub>27</sub> BrN <sub>2</sub> O	70.15	5.32	5.48 Br 15.62
Vf	44	289–290	69.88	5.46	5.32 Br 15.49	C <sub>30</sub> H <sub>27</sub> BrN <sub>2</sub> O	70.15	5.32	5.48 Br 15.62

**Table 1.** Yields, melting points, and elemental analyses of 9,9-dimethyl-12-[(5-aryl-2-furyl) (or 5-aryl-2-thienyl, or 5-aryl-1-methyl-1*H*-pyrrolyl)]-7,8,9,10,11,12-hexahydrobenzo[a]acridin-11-ones **Va–Vf** 

responding oscillator strength f. The quantity f was calculated by the formula [5]

$$f = \frac{4.39 \times 10^{-9}}{n} \int \varepsilon(\tilde{\nu}) d\tilde{\nu}, \qquad (1)$$

where *n* is the refractive index of the solvent, and  $\tilde{v}$  is the wavenumber.

The values of *f* for the long-wave absorption band at 320–450 nm (Table 2) vary in the range 0.21–0.33, with  $\lambda_{abs}^{max}$  varying insignificantly (363–370 nm).

As seen from Table 2, the oscillator strength f of the long-wave transition  $S_1 \leftarrow S_0$  is high and proportional to the rate constant  $k_{\rm fl}^{\rm calc}$  of the radiative transition  $S_1 \rightarrow S_0$ , calculated from the absorption and fluorescence spectra [6] by the formula

$$k = (2.88 \times 10^{-9}) n^2 \int \frac{\varepsilon(\widetilde{v}) (2\widetilde{v}_0 - \widetilde{v})^3}{\widetilde{v}} \, \mathrm{d}\widetilde{v}, \qquad (2)$$

where  $\tilde{v}_0$  is the wavenumber of the 0–0 transition, determined from intersection of the absorption and fluorescence spectra.

As seen from the values of  $k_{\rm fl}^{\rm calc}$  in Table 2, their variation well correlates with the variation of f for the long-wave transition  $S_1 \leftarrow S_0$ . High values of f and  $k_{\rm fl}^{\rm calc}$  are due to low symmetry of the molecular structure of

**Va–Vf** and, correspondingly, to high extent of allowance of the  $S_1 \rightleftharpoons S_0$  transition.

The oscillator strength determined by the integral extinction coefficient in accordance with Eq. (1) is the measure of the probability of the electronic transition. To estimate the probability of the  $S_1 \leftarrow S_0$  transition in Va-Vf, let us perform comparative analysis of the oscillator strengths given in Table 2 with those for benzene and naphthalene (naphthalene in the form of the 2-amino derivative participated in the synthesis of Va-Vf). The oscillator strengths for benzene and naphthalene were determined in ethanol using the molar extinction coefficients for benzene, 204 L mol<sup>-1</sup> cm<sup>-1</sup> at  $\lambda_{max} = 254$  nm, and naphthalene, 350 L mol<sup>-1</sup> cm<sup>-1</sup> at  $\lambda_{max} = 275$  nm [7]. The oscillator strengths f for benzene and naphthalene in ethanol, calculated from Eq. (1), appeared to be 0.002 and 0.0045. Comparison of these values with those obtained for Va–Vf shows that the oscillator strength for the  $S_1 \leftarrow S_0$  transition in **Va–Vf** increases by one–two orders of magnitude. Such a strong increase in the probability of the  $S_1 \leftarrow S_0$  transition is largely associated with changes in the symmetry of the molecular structure. Indeed, introduction of substituents into a benzene molecule (i.e., a decrease in the molecular symmetry) leads to an increase in the oscillator strength by an order of magnitude and more [8].

The absorption spectra of compounds Va–Vf are mainly formed on the basis of the absorption spectrum of the

Compound	$\lambda_{abs}^{max}$ , nm (log $\epsilon$ [L mol <sup>-1</sup> cm <sup>-1</sup> ])	f	$k_{\rm fl}^{\rm calc} \times 10^{-8},  {\rm s}^{-1}$	$\lambda_{fl}^{max}$ , nm	$\Phi^{\rm eff}_{\rm fl}  imes 10^3$
Va	230 (4.5), 280 (4.0), 370 (4.2)	0.27	2.2	440	0.52
Vb	235 (4.8), 290 (4.4), 363 (4.1)	0.24	2.1	470	8.4
Vc	235 (4.3), 285 (3.9), 370 (4.1)	0.23	2.1	465	0.4
Vd	235 (4.9), 290 (4.5), 368 (4.05)	0.21	2.0	410	5.0
Ve	240 (4.9), 290 (4.5), 363 (4.4)	0.33	2.5	445	1.6
Vf	240 (4.8), 280 (4.5), 363 (4.2)	0.28	2.3	450	2.0

Table 2. Absorption and luminescence spectrum characteristics of Va-Vf in ethanol solution at room temperature

naphthalene fragment (bands in the range 200–320 nm) of the dihydrobenzoacridine moiety and also contain a charge-transfer band. The band in the range 320-450 nm is due to interaction of the dihydrobenzoacridine core and hetaryl fragment bearing p-nitrophenyl or p-bromophenyl substituents [9]. This interaction leads to the charge transfer; hence, the band in the range 320-450 nm is the charge-transfer band and can be assigned to the electronic transition  $S_1 \leftarrow S_0$ . Depending on the structure of the hetaryl moiety, the shape of the charge-transfer band varies from broad and smeared (Va, Vc) to structured, consisting of several maxima (Vb, Vd–Vf). Their intensity relative to the band at 200-320 nm varies. The charge-transfer bands are characterized by high molar extinction coefficients (Table 2), comparable or exceeding (in the case of Va and Vc) those of the bands at 250–320 nm. For



**Fig. 1.** Absorption spectra of (1) Va, (2) Vb, (3) Vf, and (4) Vd in ethanol at 293 K. ( $\epsilon$ ) Extinction coefficient and ( $\lambda$ ) wavelength.

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Va and Vc ( $\lambda_{max}$  370 nm for both), log  $\varepsilon$  is 4.2 and 4.1, compared to log  $\varepsilon$  = 4.0 and 3.9 at  $\lambda_{max}$  280 and 285 nm for Va and Vc, respectively.

The charge-transfer bands are also revealed in the spectra of the starting compounds from which compounds **Va–Vf** are synthesized. For example, 2-naphthylamine **I** in ethanol exhibits a weak charge-transfer band with a maximum at 340 nm, and 5-(4-nitrophenyl)furan-2-carbaldehyde **IIa** has a strong charge-transfer band at 345 nm. In compound **Va** synthesized from these components, the charge-transfer band is red-shifted and is located at 370 nm.

The fluorescence spectra of the compounds studied, recorded in ethanol at room temperature, consist of broad structureless bands with the maxima  $\lambda_{fl}^{max}$  in the range 400–500 nm (Table 2). A specific feature of these



**Fig. 2.** (1, 2) Absorption and (3–6) fluorescence spectra of (1, 3, 4) Va and (2, 5, 6) Vf in ethanol at 293 K.  $\lambda_{exc}$ , nm: (3, 5) 320 and (4, 6) 360. (1) Intensity and ( $\lambda$ ) wavelength; the same for Figs. 3 and 4.



**Fig. 3.** (1) Absorption, (3, 4) fluorescence + phosphorescence, (5) phosphorescence, and (2) luminescence excitation spectra of Vf in ethanol at 77 K.  $\lambda_{exc}$ , nm: (3) 320 and (4) 360; (2)  $\lambda_{reg}$  500 nm. Phosphorescence spectrum 5 was recorded with a phosphoroscope.

spectra is the dependence of  $\lambda_{fl}^{max}$  on the excitation wavelength  $\lambda_{exc}$ . The fluorescence spectra of Va and Vf, shown in Fig. 2, are characterized, on the one hand, by a change in the spectrum shape (for Vf) and, on the other hand, by a bathochromic shift (Va) on changing  $\lambda_{exc}$  from 320 to 360 nm. The corresponding values of  $\lambda_{fl}^{max}$  are estimated at 430 and 450 nm. For Vb, on the contrary, the fluorescence spectra undergo hypsochromic shift with increasing  $\lambda_{exc}$ . For example, at  $\lambda_{exc} = 320$  nm, the fluorescence maximum is observed at 550 nm, whereas at  $\lambda_{exc} = 360 \text{ nm} \lambda_{fl}^{max} =$ 450 nm. Therefore, in Table 2 we present the  $\lambda_{fl}^{max}$  values averaged over three values of maxima at  $\lambda_{exc} = 320, 340,$ and 360 nm. The averaged fluorescence spectrum was also used in calculation of  $\lambda_{fl}^{calc}$ . It should be noted that the fluorescence excitation spectra do not coincide with the absorption spectra. This fact suggests differences in the sites that absorb radiation in the ground state and are responsible for the emission. In other words, different molecular structures of Va-Vf in the ground state coexist in equilibrium. These structures are characterized by slightly different absorption spectra and the corresponding fluorescence spectra, which is also different.

Presumably, such molecular structures are conformers differing in the turn of the hetaryl moiety around the single bond. Rotation around this bond leads to a change in the efficiency of the charge transfer from the dihydrobenzoacridine core. Because there is no common conjugation in the molecule, the charge transfer occurs by the inductive mechanism [9] and is controlled by mutual arrangement of the donor and acceptor. Thus, the charge transfer efficiency depends of the angle of the turn around the single bond, determining the existence of several conformers. The rotation of the molecular fragments around a single bond should depend on the viscosity of the medium. Therefore, to study this effect, we performed the absorption and luminescence measurements at 77 K in ethanol, when the molecules occur in solid glassy solutions.

In contrast to room temperature when only weak fluorescence was detected, at 77 K, along with short-wave fluorescence at 380-480 nm, a phosphorescence spectrum was observed at 480-600 nm (Fig. 3), and the fluorescence and phosphorescence excitation spectra coincide with the absorption spectra. The structured phosphorescence band is observed in the spectra of compounds Ve and Vf whose molecules contain a heavy bromine atom. Comparison of the phosphorescence spectra of Ve and Vf with the published spectrum of 1-bromonaphthalene in alcohol-ether mixture at 77 K [10] shows that these spectra fully coincide. Thus, the phosphorescence in the compounds studied is due to the naphthalene fragment of the dihydrobenzoacridinone molecule. The energy of the  $T_1$  state, estimated from the 0–0 transition at 483 nm, for Ve and Vf is 20 700 cm<sup>-1</sup>. The frequency of the 0–0 transition between the lower excited  $(S_1)$  and ground  $(S_0)$  states of these compounds appeared to be equal to 26 300 cm<sup>-1</sup>.

Phosphorescence was also detected for the other four compounds, but the spectrum was not always so clear and structured as for Vf. For example, for Va, the phosphorescence spectrum consists of a broad weakly structured band with a maximum at 490 nm (Fig. 4). Apparently, in this case it is necessary to take into account stronger influence exerted on the hetaryl moiety by the electron-withdrawing nitro group.

As seen from Fig. 3, the fluorescence and phosphorescence spectra of Vf at 77 K, recorded at two essentially different  $\lambda_{exc}$  values (320 and 360 nm), as well as the absorption and fluorescence and phosphorescence excitation spectra ( $\lambda_{reg}$  420 and 500 nm), coincide almost fully. The same coincidence of the spectral pattern under different conditions of excitation and registration of fluorescence and phosphorescence was also observed for other compounds. These facts unambiguously indicate that in a solid solution, when the turn around a single bond is impossible, there is only one conformer with its own spectral characteristics.

The compounds studied exhibit low emission efficiency at room temperature. Because the absorption spectrum consisted of the spectra of separate conformers, we were able to estimate only the effective quantum yield of fluorescence,  $\Phi_{\rm fl}^{\rm eff}$ .

The  $\Phi_{fl}^{eff}$  values given in Table 2 do not exceed 1%, which is due to strong fluorescence quenching as a result of charge transfer. The strongest quenching effect was observed for compounds Va and Vc (Table 2), in which the strong electron-withdrawing nitro group is in the p-position of the phenyl ring in the hetaryl moiety. For these compounds,  $\Phi_{fl}^{eff}$  is lower by an order of magnitude than for compounds Vb and Vd containing the NO<sub>2</sub> group in the o- or m-position. The nitro group in Va and Vc also strongly affects the electronic absorption spectra (Fig. 1) at 293 K and the luminescence spectra at 77 K (Fig. 4).

Indeed, the absorption spectra of Va and Vc in ethanol are characterized by the most pronounced charge-transfer band (320-450 nm) whose molar extinction coefficient becomes comparable with that of the most intense band in the spectra of the other compounds, 200-250 nm. Also, strong differences between the luminescence spectra of Va, on the one hand, and Vb and Vf, on the other hand, are observed at 77 K (Fig. 4). In the spectrum of Va, the short-wave fluorescence band is virtually absent, and the phosphorescence spectrum consists of a smeared structureless band. Thus, the nitro group in the *p*-position of the phenyl ring of the hetaryl moiety, as in the case of Va and Vc, exerts the strongest effect on the spectral and photophysical characteristics, expressed in more efficient fluorescence quenching at 293 K and in virtually full disappearance of the fluorescence and of vibronic structure in the phosphorescence spectra at 77 K.

The efficiency of the charge transfer depends both on substituent positions in the phenyl ring of furan- (or N-methylpyrrole-, or thiophene)carbaldehyde and on the angle of turn of furan (or thiophene, or N-methylpyrrole) rings around the single bond. As a result of such turns, the compounds exist in a liquid solution at room temperature as mixtures of conformers, each of which is characterized by its own absorption and fluorescence spectra. The assumption that the compounds exist as mixtures of conformers was confirmed by the absorption and luminescence spectra recorded at 77 K, when only one conformer remained in the solid solution owing to "freezing" of the rotation of the hetaryl fragment around the single bond. This is confirmed by coincidence of the luminescence



spectra recorded at different  $\lambda_{exc}$  with each other and of the fluorescence and phosphorescence excitation spectra with the absorption spectra.

## **EXPERIMENTAL**

The IR spectra were recorded on a Nicolet Protege-460 Fourier spectrometer from thin films or KBr pellets. The <sup>1</sup>H NMR spectra were taken with an Avance-500 spectrometer (Bruker-Biospin) operating at 500 MHz. The compounds were taken as 2-5% solutions in DMSO- $d_6$ ; the chemical shifts were determined relative to TMS as internal reference. Elemental analysis was performed with a VarioMICRO superuser CHNS analyzer, ser. no. 15106057, except the halogenated compounds whose elemental composition was determined by classical microanalysis.

The absorption and fluorescence spectra of the solutions were recorded with a Cary-500 spectrophotometer (Varian, USA) and on an installation described in [11]. The substance concentration in solutions in these measurements did not exceed  $2 \times 10^{-5}$  M. The effective quantum yields of fluorescence,  $\Phi^{\text{eff}}_{\text{fl}}$  , were measured by the relative method using quinine sulfate in  $1 \text{ N H}_2\text{SO}_4$ as reference ( $F_{\rm fl} = 0.55$  [12]). Low-temperature measurements were performed in a quartz Dewar vessel for ethanol solutions freezing at 77 K to form a transparent glass. The measurement uncertainty was 20% for the molar extinction coefficients  $\varepsilon$  and ~15% for the effective quantum yields of fluorescence.



**9,9-Dimethyl-12-[(5-aryl-2-furyl) (or 5-aryl-2-thienyl, or 5-aryl-1-methyl-1***H***-pyrrolyl)]-7,8,9,10,11,12hexahydrobenzo[***a***]acridin-11-ones Va–Vf. Procedure a: To a solution of 0.01 mol of azomethine IIIa–IIId in 30 mL of ethanol, 1.40 g (0.01 mol) of dimedone was added, and the mixture was refluxed for 2–3 h. The precipitate formed after cooling was filtered off, washed with ether, and recrystallized with an alcohol–benzene mixture (1 : 2) or washed with the same mixture.** 

Procedure b: A solution of 1.43 g (0.01 mol) of 2-naphthylamine I, 0.01 mol of appropriate aldehyde IIa–IIf, and 1.40 g (0.01 mol) of dimedone in 30 mL of ethanol was refluxed for 3–4 h. The precipitate formed after cooling was worked up similarly to procedure a.

## CONCLUSIONS

(1) The broad structureless band in the long-wave region of the absorption spectrum (320–450 nm) is due to charge transfer from the dihydrobenzoacridinone core to the hetaryl fragment of the molecule.

(2) The charge transfer processes play an important role in photophysics and spectroscopy of the compounds studied. This role is manifested most strongly, on the one hand, in efficient fluorescence quenching at 293 K for compounds **Va** and **Vc** containing the nitro group in the *p*-position of the phenyl ring of the hetaryl moiety and, on the other hand, in virtually full quenching of the fluorescence and disappearance of the vibronic structure in the phosphorescence spectra at 77 K.

(3) In the ground and excited electronic states at room temperature, the molecules of the compounds occur as mixtures of conformers differing in the angle of turn of furan (or thiophene, or *N*-methylpyrrole) rings around single C–C bond. At 77 K, the rotation around this bond is "frozen," and only one conformer remains with its specific absorption and luminescence spectra.

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