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11-R-Dibenzo[*b*,*e*][1,4]diazepin-1-ones, the Chemosensors for Transition Metal Cations

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Abstract—A series of 11-R-dibenzo[*b*,*e*][1,4]diazepin-1-ones was synthesized. The luminescence, complex formation, and redox properties of these compounds were investigated. The 11-(anthracen-9-yl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1*H*-dibenzo[*b*,*e*][1,4]diazepin-1-one was found to be an effective and selective fluorescent chemosensor for Cd^{2+} cations.

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The polycyclic heteroatomic systems containing 1,4-diazepine structure exhibit a wide spectrum of biological activity, like inhibition of hepatitis C (HCV) and human immunodeficiency (HIV-1) viruses [1]. They have been used to create the drugs affecting the central nervous system, including antidepressants, stimulants, anticonvulsants, analgesics, etc. [2–4]. The main method of synthesis of the dibenzo[*b*,*e*][1,4]-diazepin-1-one derivatives is the reaction of substituted 3-[(2-aminophenyl)amino]cyclohex-2-en-1-ones and their hetero-analogs with carbonyl derivatives [5–7].

We have previously shown that the presence of *ortho*-phenylenediamine fragment in the structure of a compound and the possibility of further modification open a way to use such systems for creation of efficient and selective fluorescent chemosensors [8–10].

To study the complexing properties of such compounds, we synthesized 2,3,4,5,10,11-hexahydro-1*H*-dibenzo[*b*,*e*][1,4]diazepin-1-ones **VII** and **VIII** containing a fluorescent fragment, anthracene or naphtho[2,3-*b*]furan, in 11-position (Scheme 1). The main method of synthesis of these compounds is the reaction of the amine **I** [5] with the corresponding aldehydes under acid catalysis. To study the effect of substituents on the chemosensory properties, we synthesized also compounds **IX–XI** containing 4-methylphenyl, 2-hydroxy-4-methylphenyl, and 2-hydroxy-1-naphthyl groups.

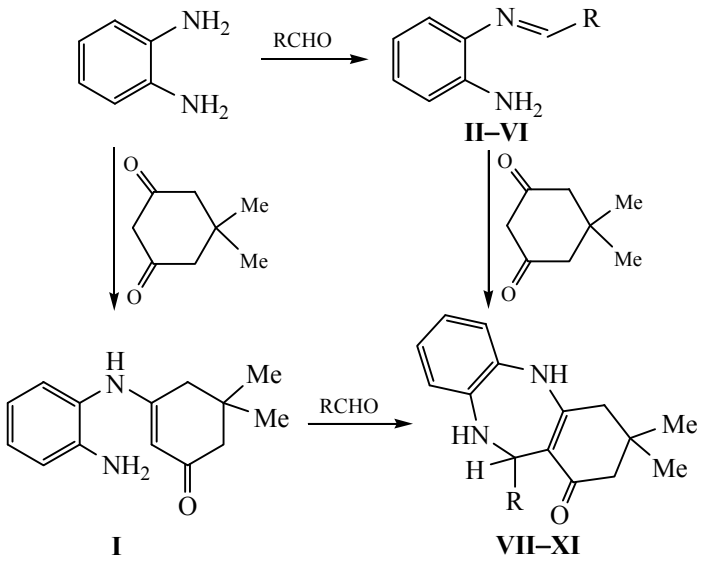
The ^{1}H NMR spectra of obtained derivatives **VII–XI** contain two typical singlet signals of the protons of two CH_3 groups in the region 1.03–1.20 ppm, and a singlet of the CH group at 5.91–6.52 ppm, which clearly indicates the formation of the diazepine ring.

Diazepines **VII–XI** were obtained also in the TosOH or $BF_3 \cdot Et_2O$ catalyzed reaction of monoimines **II–VI** with dimedone.

To study the intramolecular electron transfer from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO) it is necessary to evaluate the energies of these orbitals. They are known to be proportional to the oxidation and reduction electrochemical potentials, respectively, of the compound [11]. For the occurrence of fluorescent properties the value of the LUMO–HOMO energy gap is very significant, which in turn is associated with ΔE, the difference of the potentials of the first stages of oxidation and reduction [12].

In preliminary experiments with different rates of the variation of the polarizing voltage (0.5, 0.2, 0.1, 0.05, and 0.02 V s^{-1}) we found that the slope of the plot of logarithm of the maximum current vs. the

Scheme 1.

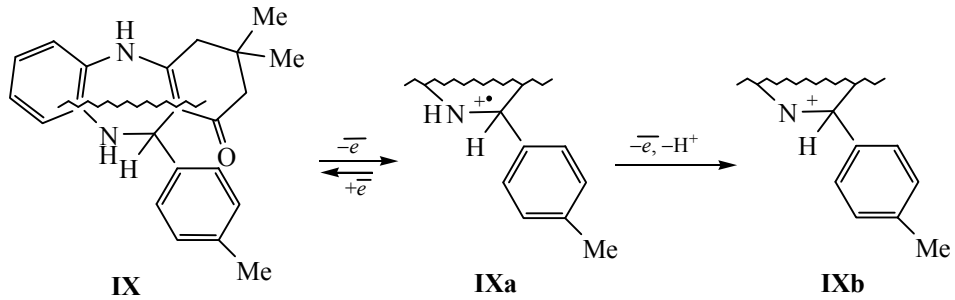


II–VI **I** **VII–XI**

II, **VII** R = anthracen-9-yl, **III**, **VIII** R = 2-methyl-9-(5-methylfuran-2-yl)naphtho[2,3-*b*]furan-4-yl, **IV**,**IX** R = *p*-tolyl, **V**, **X** R = 2-hydroxy-4-methylphenyl, **VI**, **XI** R = 2-hydroxynaphthalen-1-yl.

logarithm of the rate falls for the studied compounds in the range 0.39–0.42. These values, combined with a directly proportional dependence between the value of the maximum current and the diazepine concentration allow us to consider the oxidation processes as diffusion and irreversible [13]. Therefore we could estimate the number of transferred electrons in each stage of the electro-oxidation from the value of the peak current, comparing it with the authentic processes of one-electron oxidation of ferrocene and anthracene under the same conditions [14]. As follows from the form of cyclic voltammograms of compound **IX** containing tolyl substituent, the diazepine fragment, which is a cyclic diamine, is oxidized in two one-electron steps, therewith in the first step an unstable radical cation is formed (Scheme 2, **IXa**). This follows from the existence of the reverse signal on the cathodic branch of the curve (Fig. 1a).

Scheme 2.

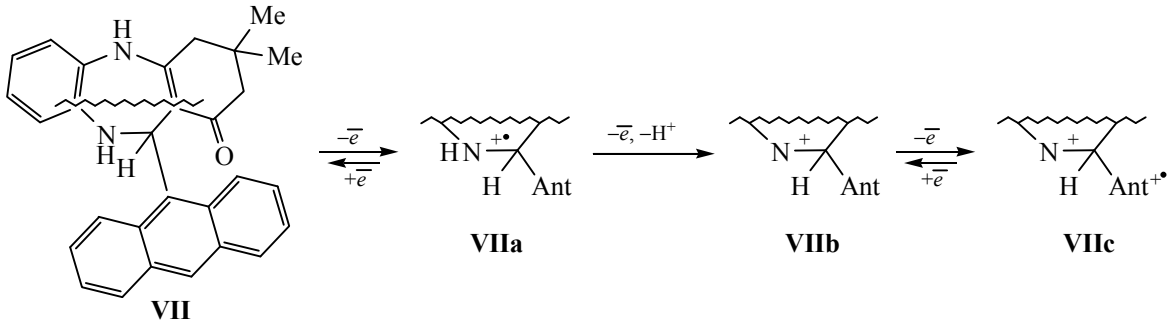


IX **IXa** **IXb**

In the second step of oxidation an unstable dication is formed, which decays with the removal of a proton to produce structure **IXb**, the peak of which is observed at the potential of 0.05 V. The presence of protons was confirmed by an increase in the signal at 0.05 V at the adding 0.002 M perchloric acid. The oxidation proceeds by a scheme similar to that of the oxidation of aliphatic amines [15].

Compound **VII** containing anthryl substituent behaves differently. Here, in the first stage an unstable radical cation also appears (Fig. 1b). However, judging from the maximum current, the second stage of oxidation is a partially reversible two-electron process. By the magnitude of the peak of the oxidation potential this stage is close to the second stage of the diazepine **VII** oxidation and to the process of oxidation of anthracene (Scheme 3).

Scheme 3.



Ant = 9-anthryl.

All the compounds are reduced difficultly and irreversibly, probably due to the presence of keto groups, and in the case of compounds **VII**, **VIII** the reduction process involves the anthracen-9-yl and naphthafuryl substituents. Data on the electrochemical properties of the diazepines are listed in the table.

As follows from the tabulated data, the compound **IX** behaves at the oxidation like aliphatic amines. The reduction runs like that of aliphatic ketones [14]. By their electrochemical properties, the diazepines **VII** and **VIII** are actually anthracene and naphthofuran with diazepine as a substituent. In both oxidation and reduction of these compounds there is mutual influence of the composing fragments on the correspond-ing potentials (see the table).

While examining compounds **X** and **XI** we revealed four oxidation steps. The first and the third steps, judging from the closeness of their potentials to those

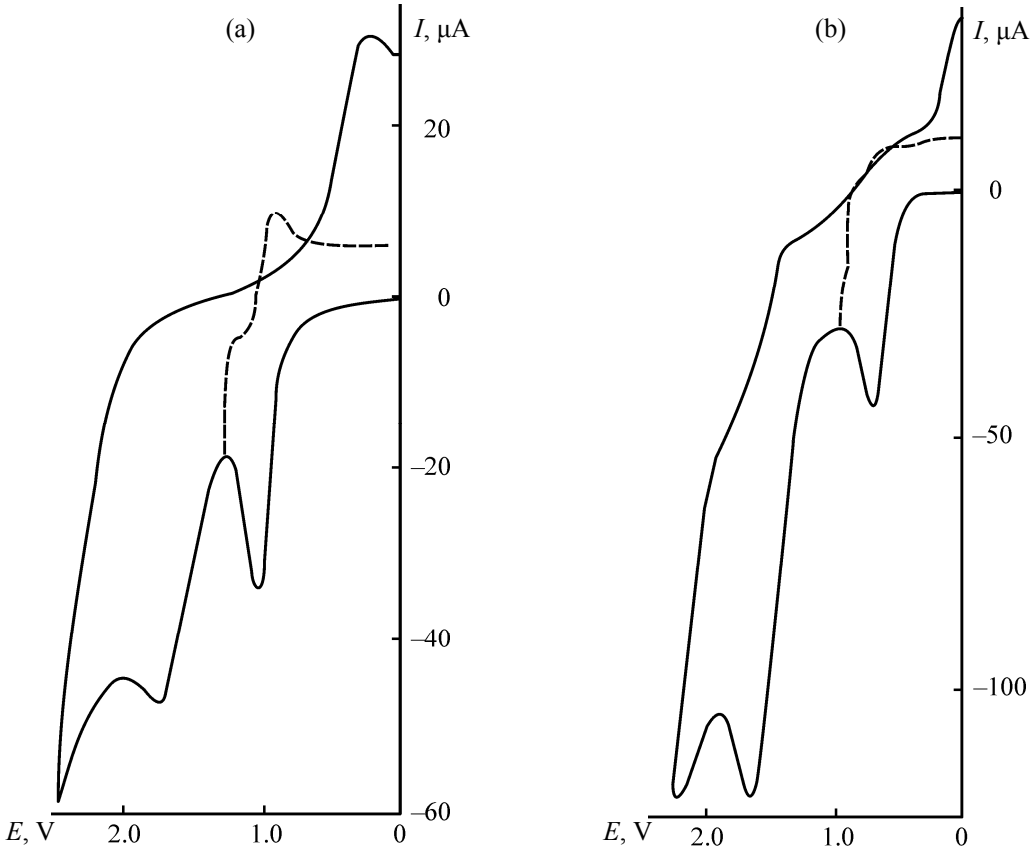


Fig. 1. Cyclic voltammograms of oxidation of compounds I**X** (a) and **VII**) (b).The dotted line shows the reversible signal indicating the formation of ion radical.

Characteristics of cyclic voltammograms of diazepines and model compounds[a]

Compound	Reduction				Oxidation				ΔE, V	I_0
	E_{pc}	I_{pc}	E_{pa}	$I_{па}$	E_{pa}	I_{pa}	E_{pc}	I_{pc}		
VII	–2.17	72	–2.05	62	0.68	47	0.54	12	2.80	1.80
	–2.42	82	–2.30	11	1.43	84	1.44	15		
VIII	–2.10	81	–	–	0.80	41	1.10	14	2.90	8.19
					1.24	43	1.70	24		
					1.85	71				
IX	–2.24	77	–	–	0.70	54	0.65	10	3.04	0.18
					1.25	38				
					1.70	43				
					2.10	55				
							0.00	56		
X	–2.10	73	–	–	0.80	56	0.70	10	2.90	4.39
					1.65	60	–	52		
							0.05			
XI	–2.15	74	–	–	0.65	53	0.55		3.0	2.63
					1.20	40				
					1.65	39				
					2.00	55				
9-Furylnaphtho[2,3-*b*]furan	–2.00	82	–	–	1.13				3.13	2300
Anthracene	–2.03	78	–1.90	71	1.30	50	1.20		3.33	2000
	–2.30	81	–2.20	11						
Ferrocene					0.45	68	0.40	64		

[a] E_{pc} is cathode peak potential, V, I_{pc} is the catode peak current, μA, E_{pa} is anode peak potential, V, I_{pa} is anode peak current, μA, ΔE is the difference between the potentials of the first oxidation and first reduction peaks, V, I_0 is relative fluorescence intensity.

in the oxidation of diazepine **IX**, correspond to the oxidation of the amine moiety, while the second and fourth correspond to hydroxy substituents.

Note that fluorescence of the diazepines **VII** and **VIII** is reduced considerably compared with the reference compounds, anthracene and 9-furylnaphtho-[2,3-*b*]furan, primarily due to the presence of nitrogen donor center and the fluorescence quenching through the PET-effect. This allows us to investigate the diazepines obtained for the presence of chemosensory properties [16].

Study of chemosensory ability of the compounds **VII**–**XI** toward the cations H^+, Zn^{2+}, Cd^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Pb^{2+}, and Hg^{2+} in acetonitrile was carried out on the basis of the data of corresponding fluorescence spectra (Fig. 2).

The spectral studies showed that in this series of cyclic derivatives only the diazepine **VII** is fairly selective. Only in the presence of Cd^{2+} ions occurs a 9-fold flare up of its fluorescence. Adding trifluoroacetic acid to acetonitrile solutions of the studied compounds, despite the presence of two amine centers, does not lead to a significant change in the fluorescence intensity. However, except for a tricycle **VII**, the protonation of the diazepines **VIII**–**XI** results in a significant blue shift of the fluorescence maxima (λ_{max} at 492, 515, 540, and 504 nm of compounds **VIII**–**XI**, respectively) by 75, 95, 37, and 122 nm, respectively . In the case of compound **X** all the examined cations cause a similar effect (shift by 25–40 nm.

Thus, the oxidation of the diazepines proceeds, like that of cyclic amines, in two stages, resulting in the first step in the formation of an unstable radical cation. The second step is completely irreversible and proceeds with the removal of a proton. The presence of a fluorescent substituent in the structures of the diazepines **VII** and **VIII** allows to use these compounds as fluorescent chemosensors that significantly expands the scope of application of such systems.

EXPERIMENTAL

^{1}H NMR spectra were obtained on a Varian Unity 300 spectrometer (300 MHz) in $CDCl_3$ or DMSO-d_6. As the internal reference the residual signals of $CHCl_3$ (δ 7.25 ppm) and $(CH_3)_2SO$ (δ 2.50 ppm) were used. Electron absorption spectra were recorded on a Varian Cary 100 spectrophotometer, the luminescence spectra

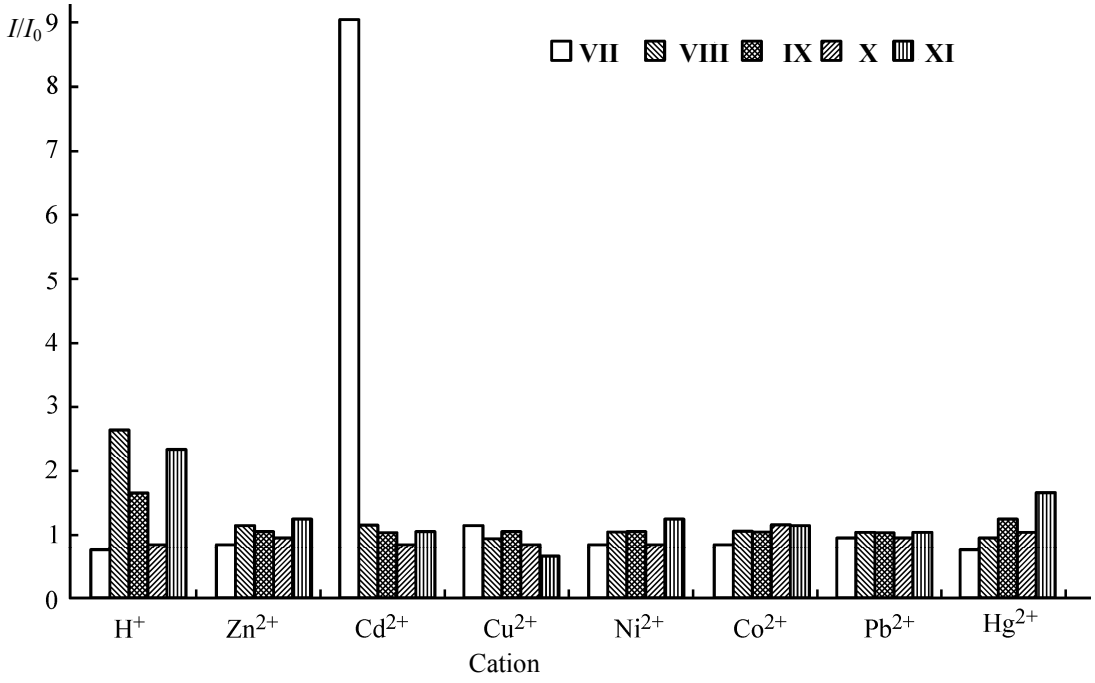


Fig. 2. The relative change in fluorescence intensity of diazepines **VII–XI** (c 5.0×10^{-6} M) in acetonitrile at adding various cations (c 2.5×10^{-5} M).

were measured on a Hitachi 650-60 and Varian Eclipse spectrofluorimeters. The vibrational spectra were recorded on a Specord IR75 instrument. Melting points were determined in glass capillaries on a PTP (M) device. The completeness of the reactions and the individuality of the compounds obtained were monitored by TLC (plates Silufol U254, eluent chloroform, development by iodine vapor in a moist chamber).

Cyclic and differential pulse voltammograms of the compounds were obtained by standard methods [17, 18] in acetonitrile with a water content $\leq 10^{-3}$ M using a platinum disk electrode of 2 mm diameter, a saturated calomel electrode as a reference electrode and platinum wire as an auxiliary electrode against the 0.1 M tetraethylammonium perchlorate as a background solution. The concentration of the compounds was 5×10^{-3} M, the concentration of added cations 5×10^{-2} M. The electrochemical measurements were carried out in an argon atmosphere. Ferrocene was used as an internal reference.

The voltammograms were recorded on a PA-2 polarograph equipped with an XY recorder, the rate of potential sweep for the cyclic voltammetry was 0.5 V s^{-1}, for the differential pulse voltammetry, 0.005 V s^{-1} with the pulse amplitude 12.5 mV.

3-[(2-Aminophenyl)amino]-5,5-dimethylcyclohex-2-en-1-one (**I**) was obtained according to the previously described technique [5].

***N*-(Anthracen-9-yl-methylidene)-1,2-diaminobenzene (II)**. In a three-neck 100 ml flask equipped with a mechanical stirrer and a reflux condenser was dissolved 2.4 g (22 mmol) of benzene-1,2-diamine in 40 ml of toluene and 0.2 ml of CH_3COOH, and while stirring and heating at 60–70ºC within 10 min was added a solution of 4.1 g (20 mmol) of anthracen-9-aldehyde in 20 ml of toluene. The reaction mixture was refluxed for 2 h, and then the solvent was removed on a rotary evaporator, the residue was crystallized from 1-butanol. Yield 94%, mp > 250ºC (decomp., 1-butanol). IR spectrum, ν, cm^{-1}: 3200, 1500, 1465. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 4.25 br.s (2H, NH); 6.80–6.90 m (2H, H_{Ar}); 7.12–7.60 m (6H, H_{Ar}), 8.04 d (2H, J 7.0 Hz, H_{Ar}); 8.53 s (1H, H_{Ar}); 8.74 d (2H, J 7.6 Hz, H_{Ar}); 9.75 s (1H, CH). The fluorescence spectrum in acetonitrile, λ_{max}, nm (5×10^{-5} M): 415. Found, %: C 85.09, H 5.39; N 9.52. $C_{21}H_{16}N_2$. Calculated, %: C 85.11, H 5.44; N 9.45.

General methods for preparing compounds (VIII–XI). *a*. 2 mmol of 3-[(2-aminophenyl)amino]-5,5-dimethylcyclohex-2-en-1-one (**I**) was dissolved in

10 ml of *i*-PrOH, AcOH was added as a catalyst, and to the hot mixture a solution was added of 2 mmol of the corresponding aldehyde in 10 ml of *i*-PrOH. The reaction solution was refluxed for 1 h. The solvent was removed on a rotary evaporator, the residue was crystallized from a suitable solvent and dried in air.

b. A solution of 2 mmol of a *N*-[1-R-methylidene]-1,2-diaminobenzene (**II**–**VI**), 2 mmol of dimedone and catalytic amount of TosOH (or $BF_3 \cdot Et_2O$) in 5–10 ml of 1-butanol was refluxed for 4–6 h. The mixture was evaporated under reduced pressure, the residue was crystallized.

11-(Anthracen-9-yl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1*H*-dibenzo[*b*,*e*][1,4]diazepin-1-one (VII). *a*. From amine **I** and anthracene-9-aldehyde. Yield 86%, mp > 270ºC (decomp., 1-butanol). IR spectrum, ν, cm^{-1}: 3320, 1586, 1520, 1467, 1370. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.03 s (3H, CH_3), 8.1 s (3H, CH_3), q 1.81 (2H, CH_2); 2.66 s (2H, CH_2); br.s 4.96 (1H, NH); 6.52 s (1H, CH), 6.58–6.80 m (3H, H_{Ar}), 7.13 d (1H, *J* 7.8 Hz, H_{Ar}), 7.20–7.48 m (4H, H_{Ar}), 7.92 d (2H, *J* 7.6 Hz, H_{Ar}), 8.30 s (1H, H_{Ar}), 8.51 d (2H, *J* 7.8 Hz, H_{Ar}), 8.84 s (1H, NH). The fluorescence spectrum in acetonitrile, λ_{max}, nm (5×10^{-5} M): 419. Found, %: C 83.15, H 6.30; N 6.74. $C_{29}H_{26}N_2O$. Calculated, %: C 83.22, H 6.26; N 6.69.

b. From imine **II** and dimedone. Yield 66%, mp > 270ºC (decomp., 1-butanol). The spectral data coincide with those described in *a*.

11-[2-Methyl-9-(5-methyl-furan-2-yl)naphtho[2,3-*b*]-furan-4-yl]-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1*H*-dibenzo[*b*,*e*][1,4]diazepin-1-one (VIII). *a.* Fom amine **I** and 2-methyl-9-(5-methylfuran-2-yl)naphtho-[2,3-*b*]furan-4-aldehyde. Yield 75%, mp 185–186ºC (decomp., 1-butanol). IR spectrum, ν, cm^{-1}: 3300, 1580, 1510, 1480. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.14 with (3H, CH_3), 1.20 s (3H, CH_3), 2.22 d (2H, *J* 4.1 Hz, CH_2), 2.30 s (3H, CH_3), 2.45 s (3H, CH_3), 2.64 s (2H, CH_2); 4.42 br.s (1H, NH); 6.11 d (2H, *J* 7.2 Hz, H_{Ar}), 6.22 s (1H, CH), 6.38–6.93 m (6H, H_{Ar}, CH, NH); 7.40–7.60 m (2H, H_{Ar}); 8.44 d (2H, *J* 8.3 Hz, H_{Ar}). The fluorescence spectrum in acetonitrile, λ_{max}, nm (5×10^{-5} M): 492. Found, %: C 78.92, H 5.97; N 5.51. $C_{33}H_{30}N_2O_3$. Calculated, %: C 78.86, H 6.02; N 5.57.

11-(4-Methylphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1*H*-dibenzo[*b*,*e*][1,4]diazepin-1-one (IX). *a*. From amine **I** and 4-methylbenzaldehyde. Yield 70%, mp 157–158ºC (decomp., 1-butanol). IR spectrum, ν, cm^{-1}: 3315, 1580, 1520, 1470. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.07 s (3H, CH_3), 1.15 s (3H, CH_3), 2.19 s (3H, CH_3), 2.23–2.72 m (4H, $2CH_2$); 4.44 br.s (1H, NH); 5.91 s (1H, CH); 6.31 br.s (1H, NH); 6.40–6.50 m (1H, H_{Ar}), 6.67–6.78 m (3H, H_{Ar}), 6.86–6.99 m (4H, H_{Ar}). The fluorescence spectrum in acetonitrile, λ_{max}, nm (5×10^{-5} M): 515. Found, %: C 79.43, H 7.34; N 8.39. $C_{22}H_{24}N_2O$. Calculated, %: C 79.48, H 7.28; N 8.43.

11-(2-Hydroxy-4-methylphenyl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1*H*-dibenzo[*b*,*e*][1,4]diazepin-1-one (X). *a*. Compound **X** was obtained from amine **I** and 2-hydroxy-4-methylbenzaldehyde. Yield 78%, mp 184–185ºC (decomp., 1-butanol). IR spectrum, ν, cm^{-1}: 3350, 3200, 1570, 1500, 1467. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.14 s (3H, CH_3), 1.20 s (3H, CH_3), 1.93 s (3H, CH_3), 2.20–2.63 m (4H, $2CH_2$); 5.04 br.s (1H, NH); 6.08 s (1H, CH); 6.12–6.80 m (8H, H_{Ar}, NH); 9.12 br.s (1H, OH). The fluorescence spectrum in acetonitrile, λ_{max}, nm (5×10^{-5} M): 540. Found, %: C 75.78, H 7.00; N 8.10. $C_{22}H_{24}N_2O_2$. Calculated, %: C 75.83, H 6.94; N 8.04.

b. From *N*-(2-hydroxy-4-methylbenziliden)-1,2-diaminobenzene (**V**) and dimedone. Yield 64%, mp 182–183ºC (decomp., 1-butanol). Spectral characteristics match the data for a substance obtained by method *a*.

11-(2-Hydroxynaphthalen-1-yl)-3,3-dimethyl-2,3,4,5,10,11-hexahydro-1*H*-dibenzo[*b*,*e*][1,4]diazepin-1-one (XI). *a*. From the amine **I** and 2-hydroxynaphthalene-1-aldehyde. Yield 81%, mp 201–202ºC (decomp., 1-butanol). IR spectrum, ν, cm^{-1}: 3300, 3250, 1580, 1515, 1475. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.16 s (6H, $2CH_3$), 2.22 s (2H, CH_2); 2.43 s (2H, CH_2), 5.46 br.s (1H, NH); 6.14 s (1H, CH), 6.90–8.23 m (10H, H_{Ar}, NH); 9.42 d (1H, *J* 14.0 Hz, H_{Ar}); 15.00 (1H, br.s, OH). The fluorescence spectrum in acetonitrile, λ_{max}, nm (5×10^{-5} M): 504. Found, %: C 78.16, H 6.23; N 7.35. $C_{25}H_{24}N_2O_2$. Calculated, %: C 78.10, H 6.29; N 7.29.

b. From *N*-(2-hydroxynaphthalen-1-ylmethylene)-1,2-diaminobenzene (**VI**) and dimedone. Yield 67%, mp 200–201ºC (decomp., 1-butanol). Spectral characteristics match the data for a substance obtained by method *a*.

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