

Synthesis, Structure, Spectral-Luminescent Properties, and Biological Activity of Chlorine-Substituted Azomethines and Their Zinc(II) Complexes

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Abstract—A number of chlorine-substituted azomethines of 2-hydroxybenzaldehydes and their zinc(II) complexes were synthesized. Structure of azomethines and zinc complexes was established by elemental analysis, IR, ¹H NMR, and X-ray spectroscopy data. Complexes ZnL₂ have a tetrahedral structure. The complexes exhibit weak photoluminescent properties in methylene chloride, whereas the photoluminescence quantum yields of the solid complexes are 100 times higher. Biological activity of the azomethines and zinc complexes was studied.

Keywords: azomethines, zinc complexes, photoluminescence, biological activity

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Chelating azomethine compounds and metal complexes based on them attract constant attention of researchers [1–12]. This is caused by their relative synthetic availability, great variability of structures, and a number of practically important properties. Azomethines and their metal complexes have a wide spectrum of biological activity [13–18], including antifungal [19, 20], antibacterial [21, 22], antimalarial [23–25], anticancer [26–29], and antiviral [30, 31], which makes them popular in medicine, veterinary medicine, and agriculture.

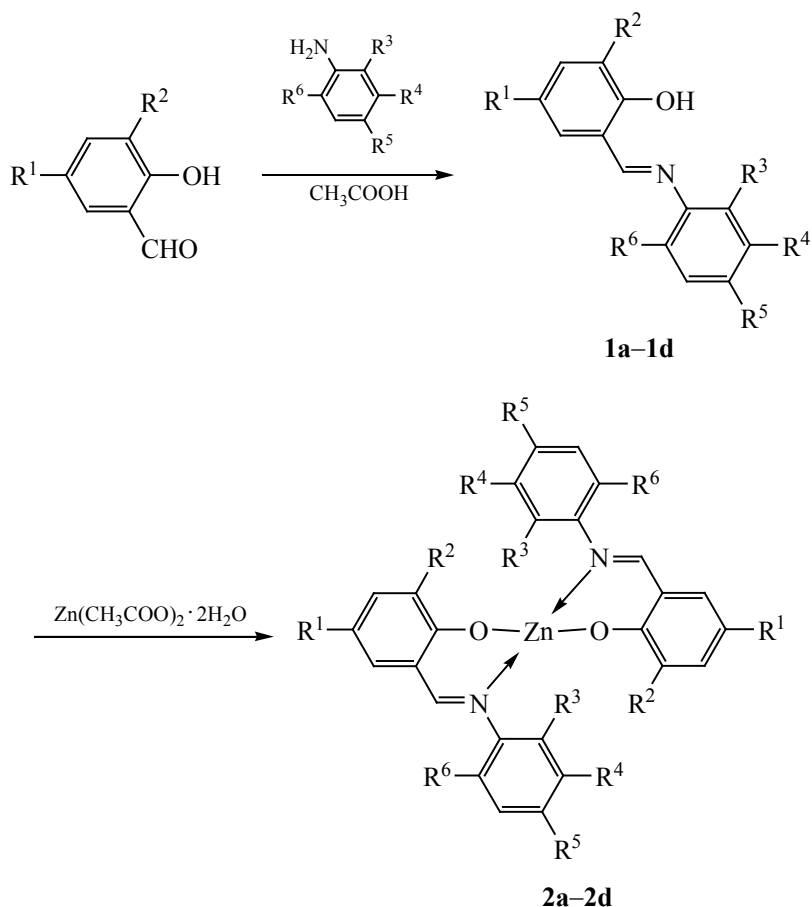
Complexes of metals with Schiff bases are used in homogeneous and heterogeneous catalysis. For example, copper complexes with *N*-(2-hydroxybenzylidene)-aryl(alkyl)amines are used as catalysts for epoxidation and oxidation of olefins [32–34]. Zinc, cadmium, beryllium, etc. complexes with azomethine ligands exhibiting photoluminescent properties can be used in manufacturing electroluminescent devices, for example, as OLED emission layers [35, 36–38]. They have thermal stability, high electron transport properties, and easily sublime during the formation of films. The most in

demand are luminescent compounds emitting in the 400–450 nm range—the main components of blue, red, and white emitters in optoelectronics [39–41]. OLED devices of various configurations were made on the basis of zinc complexes with *N*-(2-hydroxybenzylidene)arylamines, which demonstrated changes in brightness characteristics depending on the structure of the complexes [35, 36–38].

Earlier, a series of photoluminescent zinc complexes, λ_{fl} 465–541 nm, with bidentate azomethine ligands, *N*-[2-hydroxy-3-methoxy(methyl)benzylidene]-2,4,6-trimethylimines, was obtained [42]. On their basis, three different OLED devices were made, one of which had a maximum brightness of about 8000 cd/m² at 17 V, and for the other two, the brightness was 2500 and 3000 cd/m², respectively. The synthesis of new azomethine zinc complexes with photo- and electroluminescent properties is still relevant.

We have obtained a series of chlorine-substituted azomethines and their zinc(II) complexes and studied the photoluminescent properties and biological activity of these compounds. Azomethines **1a–1d** were obtained

Scheme 1.



$R^1 = R^5 = \text{Cl}$, $R^2 = R^3 = R^4 = R^6 = \text{H}$ (**a**); $R^1 = R^4 = R^5 = \text{Cl}$, $R^2 = R^3 = R^6 = \text{H}$ (**b**); $R^1 = R^2 = R^5 = \text{Cl}$, $R^3 = R^4 = R^6 = \text{H}$ (**c**); $R^1 = R^2 = R^4 = R^5 = \text{Cl}$, $R^3 = R^6 = \text{H}$ (**d**).

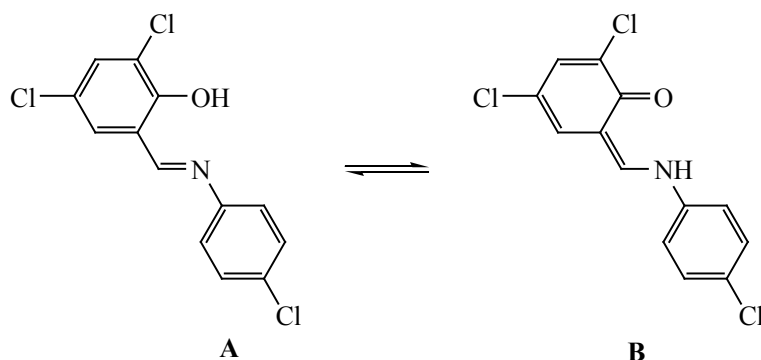
by condensation of substituted 2-hydroxybenzaldehydes and amines in glacial acetic acid (Scheme 1).

Azomethines **1a–1d** are fine-crystalline substances, from light yellow to orange in color, with mp from 122 to 172°C. Their structure was established by elemental analysis and IR, ^1H NMR spectroscopy. The IR spectra of compounds **1a–1d** contain absorption bands $\nu(\text{CH}=\text{N})$ in the range of 1615–1620 cm^{-1} and $\nu(\text{Ph}-\text{O})$ in the range of 1277–1278 cm^{-1} . The ^1H NMR spectra of azomethines **1a**, **1b**, and **1d** contain signals of protons corresponding to their structures. The signals of the protons of the phenolic OH groups appear as a singlet in the range 12.36–13.57 ppm, and the signals of the protons of the $\text{CH}=\text{N}$ groups in the range 8.52–8.92 ppm. In the ^1H NMR spectrum of azomethine **1b**, along with the signal of the proton of the OH group at 12.70 ppm and the signal of

the $\text{CH}=\text{N}$ group proton at 8.91 ppm, there are the proton signal of the quinoid form of the NH group at 14.11 ppm and the signal at 9.00 ppm of the proton at the carbon atom in the $\text{CH}-\text{NH}$ group. As is known [43, 44], for *N*-(2-hydroxybenzylidene)arylimine benzoid-quinoid tautomerism is possible (Scheme 2), due to the proton transfer from the oxygen atom to the nitrogen atom. Thus, the appearance in the ^1H NMR spectrum of the proton signals of the OH, $\text{CH}=\text{N}$, and $\text{CH}-\text{NH}$ groups and the analysis of their integral intensities suggest that a mixture of benzoic (**A**) and quinoid (**B**) tautomers of azomethine **1c** exists in a solution in DMSO in the ratio 1: 1.

In order to confirm this assumption and determine the relative stability in DMSO of two tautomeric forms **A** and **B** of azomethine **1c**, quantum-chemical calculations by the density functional method were carried out. These

Scheme 2.



calculations showed that the energy of the quinoid form of the tautomer **B** is only 0.66 kcal/mol lower than the energy of the benzoic form **A**, which points to the existence of the tautomeric equilibrium of complex **1c** in DMSO.

Bischelate zinc complexes **2a–2d** were synthesized by refluxing a mixture of azomethine **1a–1d** with methanol–chloroform (1 : 1) and a methanolic solution of zinc acetate dihydrate in a 2 : 1 molar ratio (Scheme 1). Complexes **2a–2d** are yellow fine-crystalline substances with high melting points from 262 to >290°C. According to the elemental analysis, zinc complexes **2a–2d** have the composition ZnL_2 . In the IR spectra of the zinc complexes, absorption bands $\nu(C=N)$ are observed at 1599–1606 cm^{-1} , which are shifted to the low-frequency region by 9–16 cm^{-1} in comparison with the initial azomethines **1a–1d**, whereas the absorption bands $\nu(Ph-O)$ are shifted to the high-frequency region by 25–42 cm^{-1} up to 1301–1319 cm^{-1} .

The signals of OH groups protons of azomethines **1a–1d** disappear from the 1H NMR spectra of complexes **2a–2d**, whereas the signals of the CH=N groups protons are slightly shifted by 0.06–0.48 ppm in a strong field in comparison with azomethines and appear at 8.46–8.53 ppm. Changes observed in the spectra of complexes **2a–2d** in comparison with the initial azomethines are characteristic of the formation of chelate structures [19, 20, 42, 45, 46].

The signals of the OH and NH groups protons of ligand **1c** disappear from the 1H NMR spectrum of zinc complex **2c**, and the coordination of the zinc atom, as in other complexes **2a**, **2b**, and **2d**, occurs with the benzoid form, as evidenced by the shape and nature of the 1H NMR and IR spectra.

The local atomic structure of the nearest atomic environment of zinc ions in complexes **2a–2d** was established by the X-ray spectroscopy from the analysis of XANES (X-Ray Absorption Near Edge Structure) and EXAFS (Extended X-Ray Absorption Fine Structure) of Zn absorption *K*-edges. The normalized XANES spectra and the corresponding EXAFS Fourier transform modules (MFT) for the compounds obtained are shown in Fig. 1. It can be noted that the position and shape of the Zn absorption *K*-edges of complexes **2a–2d** are very close, which points to the same environment of zinc ions in these compounds. In the XANES spectra (Fig. 1a) of complexes **2a–2d**, there is no pre-edge peak A due to the filled 3*d*-orbital of Zn(II). The presence of several maxima B, C, and D usually indicates a mixed composition of the nearest environment of zinc ions (in our case, oxygen and nitrogen atoms).

The quantitative characteristics of the coordination polyhedron in complexes **2a–2d** were obtained from the EXAFS analysis of Zn absorption *K*-edges. The MFT EXAFS of these compounds are shown in Fig. 1b. All MFT have a main peak at *r* 1.51–1.53 Å, which corresponds to the scattering of a photoelectron wave by the nearest coordination sphere of nitrogen and oxygen atoms of the ligands. The MFT peaks at large *r* values are associated with subsequent coordination spheres, including various ligand atoms, mainly carbon atoms. As a result of calculations of model EXAFS spectra, it was found that the nearest environment of zinc ions in complexes **2a–2d** is the same and consists of two nitrogen atoms and two oxygen atoms with average Zn···O distances about 1.92 Å and Zn···N distances about 2.01 Å (Table 1). The obtained Debye–Waller factors of

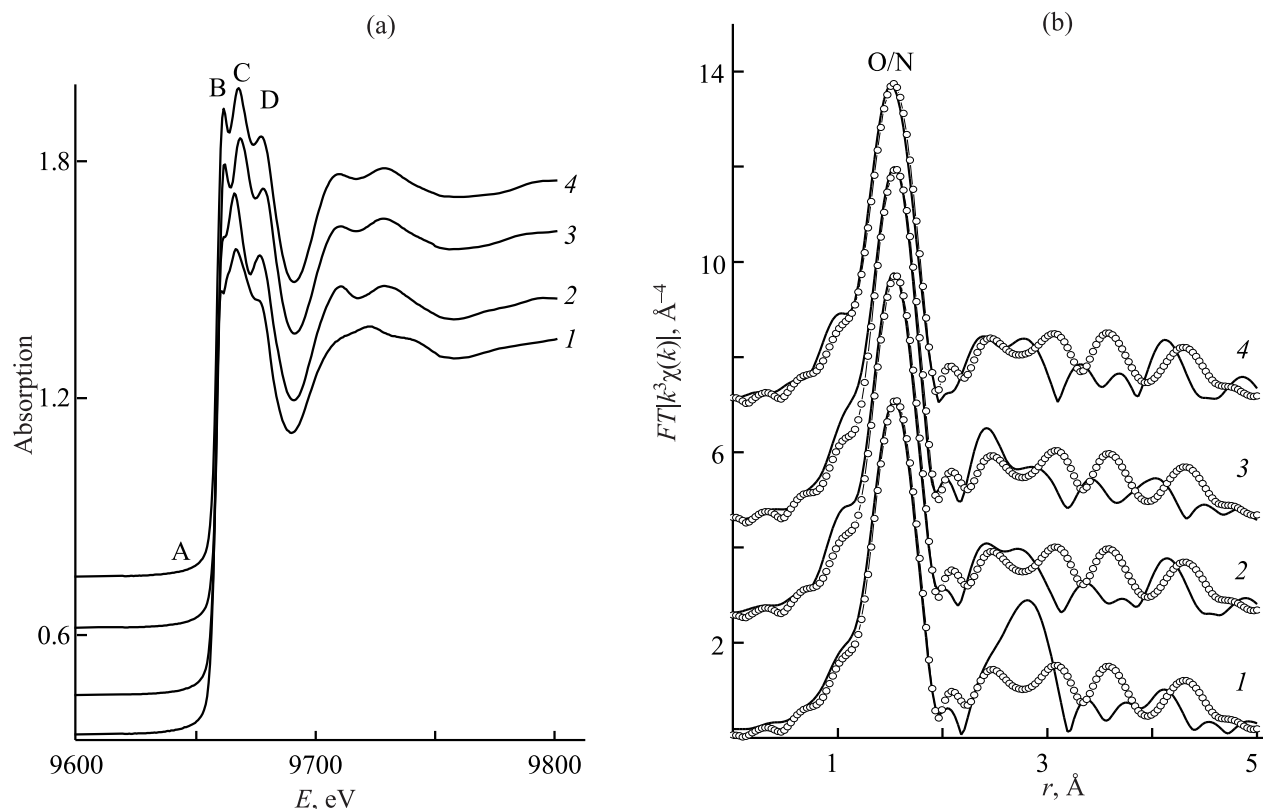


Fig. 1. Normalized (a) XANES and (b) MFT EXAFS spectra of Zn absorption K-edges of complexes (1) **2a**, (2) **2b**, (3) **2c**, (4) **2d**, (solid line) experimental data, (open circles) theory.

about 0.0032 \AA^2 are typical for such $\text{Zn}\cdots\text{O/N}$ distances in structurally similar coordination compounds [47].

A comparative study of the electronic spectra of azomethines **1a–1d** and zinc complexes **2a–2d**, recorded at room temperature in methylene chloride and in the

solid state, has been carried out. Electronic absorption spectra (EAS) of solutions of zinc complexes **2a–2d** are shown in Fig. 2, and the spectral and photoluminescent characteristics of the studied compounds are given in Table 2. In the spectral range from 300 to 400 nm, the electronic absorption spectra of azomethines **1a–1d** are

Table 1. Parameters^a of the local atomic environment in zinc(II) complexes^b **2a–2d** obtained from the EXAFS analysis of ZnK absorption edges

Complex	Bond	$r, \text{ \AA}$	$\sigma^2, \text{ \AA}^2$	$Q, \%$
2a	Zn–O	1.90	0.0031	3.0
	Zn–N	2.02	0.0031	
2b	Zn–O	1.92	0.0032	1.9
	Zn–N	2.01	0.0032	
2c	Zn–O	1.92	0.0032	1.1
	Zn–N	2.01	0.0032	
2d	Zn–O	1.92	0.0033	6.1
	Zn–N	2.01	0.0033	

^a r —interatomic distances, σ^2 —Debye–Waller factor, Q —fit quality function. The region of approximation in space $\Delta r = 1.00\text{--}1.9 \text{ \AA}$.

^b Coordination number $N = 4$.

Table 2. Parameters of the electronic absorption and photoluminescence spectra of azomethines **1a–2d** and complexes **2a–2d** in methylene chloride and in solid state at 293 K

Compound	λ_{\max} , nm ($\epsilon \times 10^4$, M ⁻¹ cm ⁻¹)	Photoluminescence		
		excitation λ_{\max} , nm	emission λ_{\max} , nm	Φ_{fl}
1a	311 (11.53), 326 (11.74), 354 (12.84)			
2a	268 (2.68), 309 (2.65), 353 (1.97), 412 (1.09)	410/390 ^a	504/543 ^a	0.002/0.147 ^a
1b	310 (10.16), 327 (9.94), 358 (10.96)			
2b	269 sh (2.50), 310 (2.41), 358 (1.70), 416 (0.99)	416/390 ^a	514/552 ^a	0.002/0.191 ^a
1c	314 (12.58), 328 (12.42), 355 (12.20)			
2c	274 (2.62), 314 (2.75), 354 (2.28), 413 sh (0.49)	412/390 ^a	511/560 ^a	0.008/0.108 ^a
1d	315 (11.92), 331 (11.74), 359 (10.36)			
2d	278 (2.55), 315 (2.76), 355 sh (1.82), 415 sh (0.70)	418/390 ^a	521/572 ^a	0.003/0.077 ^a

^a Data for solid complexes.

characterized by three absorption bands similar in shape, position, and intensity (Table 2).

In the EAS of zinc complexes **2a–2d** in methylene chloride, four absorption bands are observed in the region of 309–418 nm. The longest-wavelength absorption bands in the spectra of zinc complexes **2a–2d** are shifted bathochromically compared to azomethines **1a–1d** by 58–60 nm and are observed at 412 (**2a**), 418 (**2b**), 413 (**2c**), and 417 nm (**2d**), respectively.

Azomethines **1a–1d** do not luminesce. Complexes **2a–2d** in methylene chloride exhibit weak photoluminescence with insignificant photoluminescence quantum yields, ϕ

0.002–0.008. The photoluminescence bands of zinc complexes, in comparison with azomethines **1a–1d**, undergo a bathochromic shift. The Stokes shifts (the difference between the maxima of the long-wavelength absorption spectra and fluorescence bands) were 4431 (**2a**), 4584 (**2b**), 4643 (**2c**), and 4903 cm⁻¹ (**2d**) in methylene chloride and 4923 (**2a**), 5923 (**2b**), 6626 (**2c**), and 6634 cm⁻¹ (**2d**) for solid complexes.

The maxima of the photoluminescence bands λ_{fl} of complexes **2a–2d** in solid state are even more bathochromically shifted compared to the spectra of their solutions (by 38–41 nm, Fig. 3). The photoluminescence

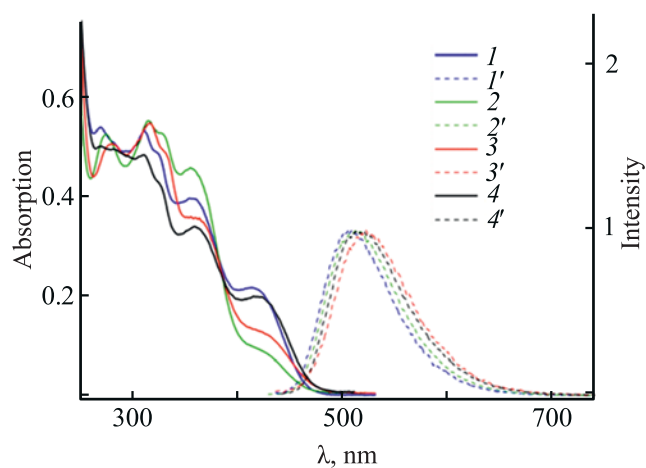


Fig. 2. (1–4) Electronic absorption and (1'–4') photoluminescence (λ_{ex} 410 nm) spectra of complexes **2a–2d** in methylene chloride at room temperature.

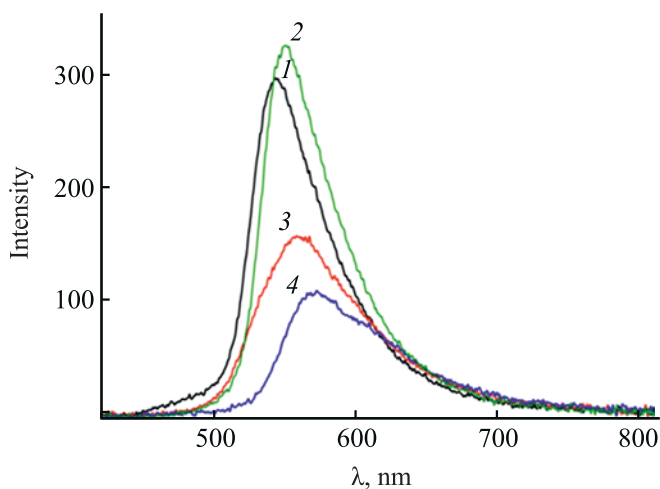


Fig. 3. Photoluminescence spectra (λ_{ex} 390 nm) of complexes (1) **2a**, (2) **2b**, (3) **2c**, and (4) **2d** in solid state at room temperature.

Table 3. Protistocidal, fungistatic, and antibacterial activities of azomethines **1a–1d** and zinc complexes **2a–2d**

Compound	MIC ^a , µg/mL	Inhibition zone diameter, mm		
	<i>Colpoda steinii</i>	<i>Penicillium italicum</i>	<i>Staphylococcus aureus</i> 6538 P	<i>Escherichia coli</i> F 50
1a	>500	0	0	0
1b	>500	0	9	8
1c	250	0	0	0
1d	500	0	0	0
2a	>500	0	0	0
2b	500	0	10	8
2c	>500	0	0	0
2d	500	0	0	0
Baycox (toltrazuril)	62.5	–	–	–
Fundazol	–	40	–	–
Furazolidone	–	–	20	18

^a MIC—the minimum inhibitory concentration.

quantum yields of complexes in solid state are much higher (almost 100 times) than the quantum yields of their solutions in methylene chloride (Table 2). Complexes **2a** and **2b** have the highest quantum yields (ϕ 0.147 and 0.191, respectively).

Azomethines **1a–1d** and zinc complexes **2a–2d** were tested for antibacterial, protistocidal, and fungistatic activity (Table 3). Azomethines **1a–1d** and zinc complexes **2a–2d** did not show fungistatic activity against *Penicillium italicum*. Azomethine **1b** had antibacterial activity against *Staphylococcus aureus*, but its activity is 2 times weaker compared to the reference drug furazolidone. Azomethine **1b** was moderately active against *Escherichia coli* (its activity is 2.2 times weaker than that of furazolidone). Complexes **2** did not show bacteriostatic activity against *Staphylococcus aureus* and *Escherichia coli*, except for complex **2b**, the activity of which against these bacteria was 2 and 2.2 times weaker compares to the reference drug furazolidone, respectively.

When studying protistocidal properties (Table 3), it was found that among azomethines, azomethine **1c** is most active against *Colpoda steinii*, the activity of which is 4 times weaker than that of the reference drug, whereas the activity of azomethine **1d** is 8 times less than that of baycox. Azomethines **1a** and **1b** did not show protistocidal activity. Zinc complexes **2b** and **2d** had the highest activity against *Colpoda steinii*, but their activity was 8 times weaker compared to the baycox preparation. Complexes **2a** and **2c** had no protistocidal activity.

Thus, a number of chlorine-substituted azomethines of 2-hydroxybenzaldehydes and their zinc(II) complexes

have been obtained. The structure of the complexes was established by X-ray absorption spectroscopy. In all the complexes obtained, a tetrahedral configuration of two oxygen atoms and two nitrogen atoms is realized around zinc(II) ions. The zinc complexes in methylene chloride exhibit weak photoluminescent properties; however, in solid state, the photoluminescence quantum yields for these complexes are about 100 times higher.

The biological activity of azomethines and zinc complexes has been studied. Azomethine, which has two chlorine atoms in positions 4 and 6 of the aldehyde fragment and one chlorine atom in position 4 of the amine fragment, exhibited the highest protistocidal activity among the studied azomethines and zinc complexes. The highest antibacterial activity was shown by 2-[(*E*)-(3,4-dichlorophenyl)iminomethyl]-4-chlorophenol and its complex with zinc, but their activity was 2–2.2 times weaker than that of the reference drug furazolidone. The results of studying biological activity make it possible to consider the search for antiprotozoal drugs among chlorine-substituted azomethines of 2-hydroxybenzaldehyde and metal complexes based on them as promising.

EXPERIMENTAL

We used commercially available solvents, zinc acetate dihydrate (CAS no. 5970-45-6), 2-hydroxybenzaldehyde (CAS no. 90-02-8), 2-hydroxy-5-chlorobenzaldehyde (CAS no. 635-93-8), 2-hydroxy-3,5-dichlorobenzaldehyde (CAS no. 90-60-8), 2-hydroxy-3,4-dichlorobenzaldehyde, acetic acid (CAS no. 64-19-7), sodium hydroxide (CAS

no. 1310-73- 2), 4-chloroaniline (CAS no. 106-47-8), aniline (CAS no. 62-53-3), and 3,4-dichloroaniline (CAS no. 95-76-1) (Alfa Aesar) without preliminary purification.

The C, H, N elemental analysis was carried out on a Carlo Erba Instruments TCM 480 analyzer. The amount of the metal was determined by the gravimetric method. Melting points were measured on a Kofler table. The IR spectra of the samples were recorded on a Varian 3100-FTIR Excalibur instrument in the range 4000–400 cm^{-1} by the method of disturbed total internal reflection. The ^1H NMR spectra were recorded on a Varian Unity-300 instrument (300 MHz) in $\text{DMSO}-d_6$ and CDCl_3 . The chemical shifts of the ^1H nuclei are given relative to the residual signals of the deuterated solvent. Electronic absorption spectra for 2.0×10^{-5} M solutions were obtained on an Agilent 8453 spectrophotometer. Photoluminescence spectra of 5.0×10^{-6} M solutions were recorded on a Varian Cary Eclipse fluorescence spectrophotometer. All spectra were recorded in dichloromethane (spectroscopy grade, Acros Organics) solutions at room temperature. The fluorescence quantum yields were determined relative to 3-methoxybenzanthrone in toluene as a standard (ϕ_{fl} 0.1, excitation at 365 nm) [48]. The photoluminescence spectra of the compounds in the solid state were recorded using a Hamamatsu C11347-01 absolute quantum yield spectrometer. The photoluminescence absolute quantum yields were determined using an integrating sphere of a Hamamatsu C11347-01 spectrometer (excitation at 390 nm).

The X-ray Zn absorption K -edges of zinc complexes were obtained at the “Structural Materials Science” station of the Kurchatov Synchrotron Center (Moscow) [49]. The energy of the electron beam, which was used as a source of X-ray synchrotron radiation, was 2.5 GeV at an average current of 100–120 mA. The X-ray absorption spectra were processed by standard procedures for background isolation, normalization to the value of the K -edge jump, and isolation of atomic absorption μ_0 , after which the Fourier transformation of the extracted EXAFS (χ)-spectrum was performed in the range of photoelectron wave vectors k from 2.5 to 12–13 \AA^{-1} with k^3 weight function. The exact parameters of the zinc ion nearest environment in the studied compounds were determined by nonlinear fitting of parameters of the corresponding coordination spheres by comparing the calculated EXAFS patterns and those extracted from the full absorption spectrum by the Fourier filtration

method. This procedure was performed using the IFFEFIT software package [50]. The phases and amplitudes of the photoelectron wave scattering required for constructing the model spectrum were calculated using the FEFF7 program [51]. As the initial atomic coordinates required for calculating the phases and amplitudes of scattering and further fitting, we used X-ray structural data for single crystals of metal complexes with a similar molecular structure from the Cambridge Database. The function of the Q fit quality, minimization of which was carried out while finding the parameters of the nearest environment structure, was calculated by formula (1).

$$Q^2 = \frac{\sum_{i=1}^m w(k_i) [k_i \chi_{\text{exp}}(k_i) - k_i \chi_{\text{th}}(k_i)]^2}{\sum_{i=1}^m w(k_i) [k_i \chi_{\text{exp}}(k_i)]^2}. \quad (1)$$

Here $w(k_i)$ is the weight function, m is the number of experimental points, $\chi_{\text{exp}}(R_i)$ and $\chi_{\text{th}}(R_i)$ are EXAFS functions in r -space.

Quantum-chemical calculations were carried out within the framework of the density functional theory using the hybrid exchange-correlation functional B3LYP [52, 53] and the 6-311++G(d,p) valence-split basis of Gaussian functions extended with polarization d -functions on heavy atoms [54]. We used the Gaussian'03 program [55]. The geometry of the molecules was optimized without restriction on symmetry, the minima of the potential energy surface being characterized by the absence of imaginary frequencies of the calculated normal vibrations. The influence of the medium was taken into account within the continuous polarizable medium model [56] using the parameters for the solvent (DMSO).

The antibacterial activity was estimated using the *Staphylococcus aureus* 6538 P and *Escherichia coli* F 50 strains (field isolates from the collection of the Rostov Regional Veterinary Laboratory) by the agar diffusion method [20, 57]. Furazolidone was used as a reference. The level of antibacterial activity was determined by the size of the growth inhibition zones.

The study of the fungistatic activity of new substances was carried out on a culture of fungi of the *Penicillium* genus and *Penicillium italicum* Wehmer (1894) species (field isolate) from the collection of micromycetes of the mycotoxicology laboratory of the North Caucasian Zonal

Research Veterinary Institute according to the method [20]. Fundazol served as a reference drug.

The protistocidal activity was studied on the protozoa of the *Colpoda steinii* species (field isolate) from the collection of the parasitology laboratory of SKZNIVI. Protistocidal activity was studied by the method of serial dilutions according to the procedure [20, 57, 58] on the species of *Colpoda steinii* protozoa culture. The reference drug was baycox (2.5% solution of toltrazuril) in the form of aqueous solutions with the same concentrations as the test compounds.

General procedure for the synthesis of azomethines 1a–1d. To a hot solution of 10 mmol of chlorine-substituted aniline in 5 mL of glacial acetic acid, a hot solution of 10 mmol of chlorine-substituted 2-hydroxybenzaldehyde in 5 mL of glacial acetic acid was added. The reaction mixture was stirred for 1 h at 100°C, then cooled to room temperature, and 10 mL of ethanol was added. The precipitate was filtered off, washed with ethanol, dried in a vacuum oven at 100°C, recrystallized from glacial acetic acid, and washed with ethanol.

4-Chloro-2-[(E)-(4-chlorophenyl)iminomethyl]-phenol (1a) was obtained from 1.56 g of 2-hydroxy-5-chlorobenzaldehyde and 1.27 g of 4-chloroaniline. Yield 2.07 g (78%), yellow crystals, mp 151–152°C (AcOH). IR spectrum, ν , cm^{-1} : 1615 m (CH=N), 1276 s (Ph–O). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 6.99 d (1H_{Ar}, 3J 8.7 Hz), 7.41–7.53 m (5H_{Ar}), 7.74 d (1H_{Ar}, 4J 2.7 Hz), 8.91 s (1H, CH=N), 12.70 s (1H, OH). Found, %: C 58.63; H 3.47; N 5.23. C₁₃H₉Cl₂NO. Calculated, %: C 58.67; H 3.41; N 5.26.

2-[(E)-(3,4-Dichlorophenyl)iminomethyl]4-chlorophenol (1b) was obtained from 1.56 g of 2-hydroxy-5-chlorobenzaldehyde and 1.62 g of 3,4-dichloroaniline. Yield 2.4 g (80%), orange powder, mp 137–138°C (AcOH). IR spectrum, ν , cm^{-1} : 1620 s (CH=N), 1278 s (Ph–O). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 6.99 d (1H_{Ar}, 3J 9.0 Hz), 7.38–7.47 m (2H_{Ar}), 7.68 s (1H_{Ar}), 7.71–7.73 m (2H_{Ar}), 8.92 s (1H, CH=N), 12.36 s (1H, OH). Found, %: C 51.93; H 2.65; N 4.62. C₁₃H₈Cl₃NO. Calculated, %: C 51.95; H 2.68; N 4.66.

2,4-Dichloro-6-[(E)-(4-chlorophenyl)iminomethyl]-phenol (1c) was obtained from 1.91 g of 2-hydroxy-3,5-dichlorobenzaldehyde and 1.27 g of 4-chloroaniline. Yield 2.52 g (84%), orange powder, mp 121–122°C (AcOH). IR spectrum, ν , cm^{-1} : 1615 m (CH=N), 1277 m (Ph–O). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 7.41–7.46 m (1.5H_{Ar}), 7.49–7.57 m (3H_{Ar}), 7.71–7.74 m

(1.5H_{Ar}), 8.91 s (0.5H, CH=N), 9.00 s (0.5H, CH–NH), 12.70 s (0.5H, OH), 14.11 s (0.5H, NH). Found, %: C 51.98; H 2.63; N 4.69. C₁₃H₈Cl₃NO. Calculated, %: C 51.95; H 2.68; N 4.66.

2,4-Dichloro-6-[(E)-(3,4-dichlorophenyl)iminomethyl]phenol (1d) was obtained from 1.91 g of 2-hydroxy-3,5-dichlorobenzaldehyde and 1.62 g of 3,4-dichloroaniline. Yield 2.58 g (77%), orange powder, mp 171–172°C (AcOH). IR spectrum, ν , cm^{-1} : 1615 m (CH=N), 1278 m (Ph–O). ^1H NMR spectrum (CDCl₃), δ , ppm: 7.13 d. d (1H_{Ar}, 3J 8.7, 4J 2.4 Hz), 7.3 d (1H_{Ar}, 4J 2.7 Hz), 7.38 d (1H_{Ar}, 4J 2.4 Hz), 7.47 d (1H_{Ar}, 4J 2.4 Hz), 7.5 d (1H_{Ar}, 3J 8.7 Hz), 8.52 s (1H, CH=N), 13.57 s (1H, OH). Found, %: C 46.54; H 2.15; N 4.12. C₁₃H₇Cl₄NO. Calculated, %: C 46.61; H 2.11; N 4.18.

General procedure for the synthesis of complexes 2a–2d. A solution of zinc acetate dihydrate (0.22 g, 1 mmol) in 5 mL of methanol was added to a boiling solution of 2 mmol of azomethine 1a–1d in 30 mL of a mixture of methanol and chloroform (1 : 1). Then, a solution of 0.08 g (2 mmol) of sodium hydroxide in 5 mL of methanol was added dropwise. The reaction mixture was boiled for 2 h, the precipitate was filtered off, washed with methanol, and dried in a vacuum oven at 100°C.

Bis{4-chloro-2-[(E)-(4-chlorophenyl)iminomethyl]-phenoxy}zinc (2a) was obtained from 0.53 g (2 mmol) of azomethine 1a. Yield 0.42 g (70%), yellow powder, mp 267–268°C. IR spectrum, ν , cm^{-1} : 1600 m (CH=N), 1301 m (Ph–O). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 6.67 d (2H_{Ar}, 3J 9.0 Hz), 7.27 d. d (2H_{Ar}, 3J 9.3, 4J 2.7 Hz), 7.28 d (4H_{Ar}, 3J 9.0 Hz), 7.33 d (4H_{Ar}, 3J 9.0 Hz), 7.49 d (2H_{Ar}, 4J 2.7 Hz), 8.53 s (2H, CH=N). Found, %: C 52.47; H 2.76; N 4.73; Zn 10.95. C₂₆H₁₆Cl₄N₂O₂Zn. Calculated, %: C 52.43; H 2.71; N 4.70; Zn 10.98.

Bis{2-[(E)-(3,4-dichlorophenyl)iminomethyl]4-chlorophenoxy}zinc (2b) was obtained from 0.6 g (2 mmol) of azomethine 1b. Yield 0.49 g (73%), yellow powder, mp 261–262°C. IR spectrum, ν , cm^{-1} : 1604 m (CH=N), 1312 m (Ph–O). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 6.62 d (2H_{Ar}, 3J 9.0 Hz), 7.24 d (2H_{Ar}, 3J 8.4 Hz), 7.34 d. d (2H_{Ar}, 3J 8.4, 4J 2.1 Hz), 7.45 s (2H_{Ar}), 7.6 d (2H_{Ar}, 3J 8.4 Hz), 7.69 s (2H_{Ar}), 8.45 s (2H, CH=N). Found, %: C 46.94; H 2.16; N 4.25; Zn 9.85. C₂₆H₁₄Cl₆N₂O₂Zn. Calculated, %: C 46.99; H 2.12; N 4.22; Zn 9.84.

Bis{2,4-dichloro-6-[(E)-(4-chlorophenyl)iminomethyl]phenoxy}zinc (2c) was obtained from 0.6 g

(2 mmol) of azomethine **1c**. Yield 0.51 g (76%), yellow powder, mp > 290°C. IR spectrum, ν , cm^{-1} : 1599 s (CH=N), 1319 w (Ph-O). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 7.24 d (2H_{Ar}, 8.1 Hz), 7.35–7.52 m (10H_{Ar}), 8.47 s (2H, CH=N). Found, %: C 46.95; H 2.16; N 4.27; Zn 9.80. C₂₆H₁₄Cl₆N₂O₂Zn. Calculated, %: C 46.99; H 2.12; N 4.22; Zn 9.84.

Bis[2,4-dichloro-6-[(E)-(3,4-dichlorophenyl)imino-methyl]phenoxy]zinc (2d) was obtained from 0.67 g (2 mmol) of azomethine **1d**. Yield 0.52 g (71%), yellow powder, mp > 290°C. IR spectrum, ν , cm^{-1} : 1606 s (CH=N), 1318 w (Ph-O). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 7.46–7.49 m (6H_{Ar}), 7.61 d (2H_{Ar}, 3J 7.2 Hz), 7.93 br. s (2H_{Ar}), 8.46 s (2H, CH=N). Found, %: C 42.53; H 1.69; N 3.86; Zn 8.95. C₂₆H₁₂Cl₈N₂O₂Zn. Calculated, %: C 42.58; H 1.65; N 3.82; Zn 8.91.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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