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# Coordination Compounds of Ambidentate 1-(H)Alkyl-2-(2-pyridyl)benzimidazoles. Synthesis and Crystal Structure

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**Abstract**—Mono- and binuclear complexes of 2-(2-pyridyl)benzimidazole and its 1-alkyl-substituted derivatives with zinc dichloride were synthesized and structurally characterized. In terms of the competitive coordination problem, it was shown that pyridylbenzimidazole is an *N*,*N*-chelating ligand. The binuclear complex is formed through a dichloride bridge.

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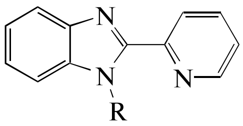
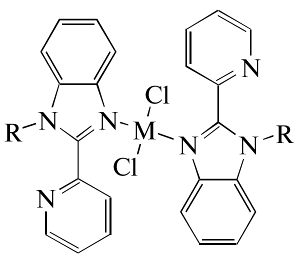
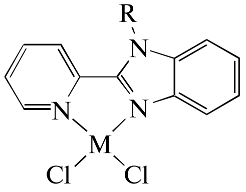
The metal complexes based on 2-(2-pyridyl)benzimidazoles (**I**) occupy an important place among the coordination compounds of bis-heterocyclic ligands [1–6].



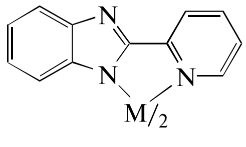
(**I**): R = H, Alk, Ar



(**II**)



**(III)**



**(IV)**

These potentially tridentate ligand systems form complexes with different types of metal binding: molecular adducts where they function as mono- (**II**) [7] or bidentate (**III**) ligands [5–12] or bis-chelates (**IV**) [2, 3, 13] formed upon deprotonation of the imidazole N–H group. Both mono- [1–13] and oligonuclear [6, 11, 14–18] complexes have been isolated. These are of interest as polyfunctional materials (for example, luminescence materials [8, 9, 19], catalysts [7, 10, 13, 20, 21], and other practically valuable compounds [22, 23]).

As a continuation of this subject matter, we synthesized complexes of ligands **I** with zinc chloride and studied them in view of the potential luminescence activities [24].

## EXPERIMENTAL

2-(2-Pyridyl)benzimidazole (**Ia**, R = H) was prepared by a reported procedure [25].

**Synthesis of 1-alkyl-2-(2-pyridyl)benzimidazoles (R = $CH_3$ (Ib), $C_4H_9$ (Ic), $CH_2$-CH=$CH_2$ (Id), $CH_2C_6H_5$ (Ie)).** 2-(2-Pyridyl)benzimidazole (5.86 g, 0.03 mol)) and acetone (30 mL) were added to 40% aqueous KOH (12 mL). To the resulting mixture, alkyl halide (for **Ib**, $CH_3I$; for **Ic**, $C_4H_9Br$; for **Id**, $CH_2CHCH_3Br$; and for **Ie**, $CH_2C_6H_5Cl$) (0.033 mol) was added dropwise with stirring over a period of 10 min. The mixture was refluxed for 2 h. The acetone layer was separated and treated with water (50 mL). The precipitate that formed was filtered off, washed with water, and dried. The product was dissolved in 15% HCl (30 mL) and refluxed with activated carbon, and pH was brought to 7 by adding a 22%solution of ammonia. The precipitate was filtered off, washed with water, and dried. Then the product was purified by chromatography on a column with $Al_2O_3$ (chloroform as the eluent), chloroform was distilled off from the eluate to give **I** (R = Alk) as colorless crystals (or colorless oil in the case of **Ic**).

The elemental analysis data, the yields, and the melting points of compounds **Ib–Ie** are summarized in Table 1.

**Table 1.** Elemental analysis data, yields, and melting points of compounds **Ib–Ie*** and **IIIa–IIIe**

| Compound | Molecular formula | Content (found/calculated), % | | | | | $T_{mp}$, °C | Yield, % |
|---|---|---|---|---|---|---|---|---|
| | | C | H | N | Zn | Cl | | |
| Ib | $C_{13}H_{11}N_3$ | 74.64/74.62 | 5.21/5.30 | 20.01/20.08 | | | 62–63 | 49 |
| Ic | $C_{16}H_{17}N_3$ | 72.43/72.48 | 6.46/6.54 | 21.11/21.17 | | | oil | 57 |
| Id** | $C_{15}H_{13}N_3$ | 76.57/76.61 | 5.57/5.60 | 17.86/17.77 | | | 60–61 | 62 |
| Ie | $C_{19}H_{15}N_3$ | 79.89/79.98 | 5.28/5.30 | 14.47/14.73 | | | 116–118 | 66 |
| IIIa | $C_{12}H_9Cl_2N_3Zn$ | 43.50/43.48 | 2.71/2.74 | 12.82/12.67 | 19.83/19.72 | 21.42/21.39 | >250 | 41 |
| IIIb | $C_{26}H_{22}Cl_4N_6Zn_2$ | 45.19/45.21 | 3.21/3.18 | 12.16/12.26 | 18.92/19.01 | 20.46/20.32 | >250 | 45 |
| IIIc | $C_{16}H_{17}Cl_2N_3Zn$ | 47.85/47.89 | 4.27/4.38 | 13.95/13.89 | 16.28/16.31 | 17.66/17.58 | >250 | 32 |
| IIId | $C_{15}H_{13}Cl_2N_3Zn$ | 48.49/48.51 | 3.53/5.48 | 11.71/11.28 | 17.60/17.62 | 19.08/19.17 | >250 | 38 |
| IIIe | $C_{19}H_{15}Cl_2N_3Zn$ | 54.12/54.18 | 3.59/3.61 | 9.97/9.84 | 15.51/15.45 | 16.82/16.94 | >250 | 46 |

Notes: * For **Ib**: yield 49%, $T_{mp}$ = 62–63°C (higher than published value [26]), for **Ie**: yield 66%, $T_{mp}$ = 116–118° (corresponds to the published value [26]).

** Recrystallized from hexane.

**Ib**: IR (ν, cm$^{-1}$): 1588 w, 1564 w (benzimidazole ring). $^1$H NMR (DMSO-d$_6$; δ, ppm): 4.30 (s 3H, $CH_3$), 7.21–7.51 (m, 4H, $C_{Ar}$–H), 7.65–7.95 (m, 2H, $C_{Ar}$–H), 8.36 (d, 1H, $J$ = 8.1 Hz, $C_{Ar}$–H), 8.69 (d, 1H, $J$ = 4.8 Hz, $C_{Ar}$–H).

**Ic**: IR (ν, cm$^{-1}$): 1587 m, 1563 w (C=N). $^1$H NMR (DMSO-d$_6$; δ, ppm): 0.94 (t, 3H, $J$ = 7.4 Hz, $CH_3$), 1.40 (q, 2H, $J$ = 7.8 Hz, $CH_2\underline{CH_2}CH_2CH_3$), 1.83 (2H, q, $J$ = 7.7 Hz, $CH_2CH_2\underline{CH_2}CH_3$), 4.86 (t, 2H, $J$ = 7.4 Hz, $\underline{CH_2}CH_2CH_2CH_3$), 7.20–7.48 (m, 4H, $C_{Ar}$–H), 7.64–7.92 (m, 2H, $C_{Ar}$–H), 8.38 (d, 1H, $J$ = 8.1 Hz, $C_{Ar}$–H), 8.68 (d, 1H, $J$ = 4.2 Hz, $C_{Ar}$–H).

**Id**: IR (ν, cm$^{-1}$): 1589 m, 1563 w (C=N). $^1$H NMR (DMSO-d$_6$; δ, ppm): 4.95 (d, 1H, $J$ = 17.1 Hz, $CH_2$), 5.08 (d, 1H, $J$ = 10.2 Hz, $CH_2$), 5.54 (d, 2H, $J$ = 5.1 Hz, $CH_2$), 6.00–6.09 (m, 1H, CH), 7.25–7.54 (m, 4H, $C_{Ar}$–H), 7.61 (d, 1H, $J$= 7.5 Hz, $C_{Ar}$–H), 7.74 (d, 1H, $J$= 6.9 Hz, $C_{Ar}$–H), 7.97–8.03 (m, 1H, CAr–H), 8.33 (d, 1H, $J$= 7.8 Hz, $C_{Ar}$–H), 8.73 (d, 1H, $J$ = 4.2 Hz, $C_{Ar}$–H).

**Ie**: IR (ν, cm$^{-1}$)): 1585 m, 1565 w (benzimidazole ring). $^1$H NMR (DMSO-d$_6$; δ, ppm): 6.22 (s, 2H, $CH_2$), 7.11–7.28 (m, 7H, m, $C_{Ar}$–H), 7.48–7.58 (m, 2H, $C_{Ar}$–H), 7.74–7.77 (m, 1H, $C_{Ar}$–H), 7.97–8.03 (m, 1H, $C_{Ar}$–H), 8.36 (d, 1H, $J$ = 8.1 Hz, $C_{Ar}$–H), 8.69 (d, 1H, $J$ = 4.8 Hz, $C_{Ar}$–H).

**Synthesis of complexes III.** Zinc chloride (0.01 mol) was added to a solution of specified 1-alkyl(H)-2-(2-pyridyl)benzimidazole (0.01 mol) in absolute methanol (10 mL). The mixture was refluxed for 1 h. The precipitated crystals of the complexes were filtered off, washed with methanol (2 × 2 mL), and dried in a vacuum drying chamber. The crystals for X-ray diffraction analaysis were grown from a methanol–chloroform mixture (2 : 1).

**IIIa**: IR (ν, cm$^{-1}$): 3256 br (NH), 1601 m (benzimidazole ring). $^1$H NMR (DMSO-d$_6$; δ ppm): 7.25 (s 2H, $C_{Ar}$–H), 7.56–7.63 (m, 3H, $C_{Ar}$–H), 8.08–8.10 (m, 1H, $C_{Ar}$–H), 8.40 (d, 1H, $J$ = 7.8 Hz, $C_{Ar}$–H), 8.64 (s, 1H, $C_{Ar}$–H), 13.58 (br.s. 1H, NH).

**IIIb**: IR (ν, cm$^{-1}$): 1595 m, 1585 w (benzimidazole ring). $^1$H NMR (DMSO-d$_6$; δ ppm): 4.26 (s, 3H, $CH_3$), 7.27–7.38 (m, 2H, $C_{Ar}$–H), 7.57–7.59 (m, 1H, $C_{Ar}$–H), 7.69 (d, 1H, $J$ = 7.8 Hz, $C_{Ar}$–H), 7.77 (d, 1H, $J$ = 7.8 Hz, $C_{Ar}$–H), 8.05 (t, 1H, $J$ = 7.8 Hz, $C_{Ar}$–H), 8.34 (d, 1H, $J$ = 8.1 Hz, $C_{Ar}$–H), 8.76 (d, 1H, $J$ = 4.5 Hz, $C_{Ar}$–H).

**IIIc**: IR (ν, cm$^{-1}$): 1597 m, 1587 w (benzimidazole ring). $^1$H NMR (DMSO-d$_6$; δ, ppm): 0.83 (t, 3H, $J$ = 7.3 Hz, $CH_3$), 1.26 (q, 2H, $J$= 7.5 Hz, $CH_2CH_2CH_2CH_3$), 1.74 (q, 2H, $J$= 7.4 Hz, $CH_2CH_2CH{=}CH_3$), 4.83 (t, 2H, $J$ = 7.3 Hz, $-CH_2CH_2CH_2CH_3$), 7.24–7.36 (m, 2H, $C_{Ar}$–H), 7.52–7.56 (m, 1H, $C_{Ar}$–H), 7.68–7.76 (m, 2H, $C_{Ar}$–H), 7.99–8.05 (m, 1H, $C_{Ar}$–H), 8.31 (d, 1H, $J$ = 8.1 Hz, $C_{Ar}$–H), 8.74 (d, 1H, $J$ = 4.1 Hz, $C_{Ar}$–H).

**IIId**: IR (ν, cm$^{-1}$): 1598 m, 1588 m (benzimidazole ring). $^1$H NMR (DMSO-d$_6$; δ, ppm): 5.01 (d, 1H, $J$ = 17.1 Hz, CH), 5.10 (d, 2H, $J$ = 10.3 Hz, $CH_2$), 5.55 (d, 2H, $J$ = 4.4 Hz, $CH_2$), 6.00–6.12 (m, 1H, CH), 7.19–7.28 (m, 2H, $C_{Ar}$–H), 7.42–7.49 (m, 2H, $C_{Ar}$–H), 7.69–7.72 (m, 1H, $C_{Ar}$–H), 7.91–7.97 (m,

**Table 2.** Crystallographic data and X-ray experiment details for compounds **IIIb–IIIe**

| Parameter | Value | | | |
|---|---|---|---|---|
| | **IIIb** | **IIIc** | **IIId** | **IIIe** |
| $M$ | 691.04 | 387.60 | 371.55 | 421.61 |
| System | Triclinic | Triclinic | Monoclinic | Monoclinic |
| Space group | $P\bar{1}$ | $P\bar{1}$ | $P2_1/n$ | $P2_1/c$ |
| $a$, Å | 8.962(3) | 9.380(5) | 9.792(4) | 9.698(4) |
| $b$, Å | 9.2651(3) | 10.261(5) | 14.294(3) | 13.965(4) |
| $c$, Å | 9.351(3) | 10.427(5) | 11.105(4) | 14.148(5) |
| $\alpha$, deg | 105.79(3) | 61.67(3) | 90 | 90 |
| $\beta$, deg | 107.25(4) | 85.33(2) | 101.25(3) | 108.25(3) |
| $\gamma$, deg | 106.41(4) | 74.82(2) | 90 | 90 |
| $V$, Å$^3$ | 655.47(11) | 853.2(4) | 1524.6(2) | 1819.8(3) |
| Z | 1 | 2 | 4 | 4 |
| $\rho$(calcd), g/cm$^3$ | 1.751 | 1.509 | 1.619 | 1.539 |
| $F$(000) | 348 | 396 | 752 | 856 |
| $\mu_{Mo}$, mm$^{-1}$ | 2.267 | 1.751 | 1.956 | 1.649 |
| $\theta$ range, deg | 2.48–29.97 | 2.22–29.97 | 2.35–29.98 | 2.10–25.72 |
| Range of indices | $-11 \le h \le 11, 0 \le k \le 11, -11 \le l \le 11$ | $-12 \le h \le 12, 0 \le k \le 13, -12 \le l \le 13$ | $-12 \le h \le 8, 0 \le k \le 18, -14 \le l \le 14$ | $-10 \le h \le 0, 0 \le k \le 15, -15 \le l \le 15$ |
| The number of independent reflections | 3087 | 4862 | 4324 | 3162 |
| The number of reflections ($I \ge 2\sigma(I)$) | 1289 | 2112 | 1306 | 1515 |
| The number of refined parameters | 216 | 196 | 190 | 286 |
| GOOF | 1.028 | 1.028 | 1.036 | 1.032 |
| $R_1/wR_2$ ($I \ge 2\sigma(I)$) | 0.0555/0.1331 | 0.0431/0.0970 | 0.0539/0.0772 | 0.0424/0.1105 |
| $\Delta\rho_{min}/\Delta\rho_{max}$, $e$ Å$^{-3}$ | –1.641/1.299 | –0.478/0.575 | –0.498/0.658 | –1.804/0.124 |

1H, $C_{Ar}$–H), 8.36 (d, 1H, $J$ = 8.0 Hz, $C_{Ar}$–H), 8.67 (d, 1H, $J$ = 4.8 Hz, $C_{Ar}$–H).

**IIIe**: IR (ν, cm$^{-1}$): 1599 m, 1585 w (benzimidazole ring). $_1$H NMR (DMSO-$d_6$; δ, ppm): 6.22 (s, 2H, $CH_2$), 7.11–7.29 (m, 7H, $C_{Ar}$–H), 7.49–7.60 (m, 2H, $C_{Ar}$–H), 7.75–7.78 (m, 1H, $C_{Ar}$–H), 7.98–8.04 (m, 1H, $C_{Ar}$–H), 8.36 (d, 1H, $J$= 7.8 Hz, $C_{Ar}$–H), 8.70 (d, 1H, $J$ = 4.8 Hz, $C_{Ar}$–H).

The results of elemental analysis, the yields, and melting points of compounds **IIIa–IIIe** are summarized in Table 1.

The $^1$H NMR spectra were recorded on a Varian Unity 300 (300 MHz) instrument in the internal stabilization mode of $^2$H resonance line in DMSO-$d_6$. The IR spectra were recorded for solid samples of ligands and complexes on a Varian-Excalibur 3100 FT-IR instrument in the frustrated total internal reflection mode for powders. IR spectra were recorded on a Varian Cary 100 spectrophotometer.

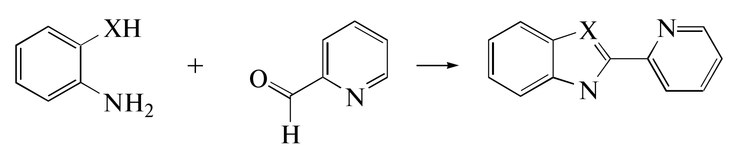
**X-Ray diffraction analysis**. The experimental data for crystal structure determination of $ZnCl_2$ with pyridylbenzimidazole containing different substituents R (Alk, Ar) at the imidazole nitrogen (type **III**) were collected on a CAD4 Enraf-Nonius diffractometer (Mo$K_\alpha$ radiation, graphite monochromator, $\theta/2\theta$ scan mode).

Crystallographic characteristics and X-ray experiment details are summarized in Table 2 and deposited with the Cambridge Crystallographic Data Centre (nos. 3770420 ($C_{19}H_{15}Cl_2N_3Zn$), 770421 ($C_{16}H_{17}Cl_2N_3Zn$), 770422 ($C_{15}H_{13}Cl_2N_3Zn$), 771 080 ($C_{26}H_{22}Cl_4N_6Zn_2$); deposit@icdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

The structures of compounds **Ia–IIIe** were solved by the direct method using SHELXS-86 program package [27] and refined by the SHELXL-97 program package [28]. Non-hydrogen atoms were refined anisotropically, the hydrogen atoms were partly located from difference Fourier syntheses and refined with the isotropic thermal parameter $U_H = 0.08$ Å$^2$, the lacking ones were specified geometrically and refined by the riding model with fixed $U_H = 0.08$ Å$^2$.

## RESULTS AND DISCUSSION

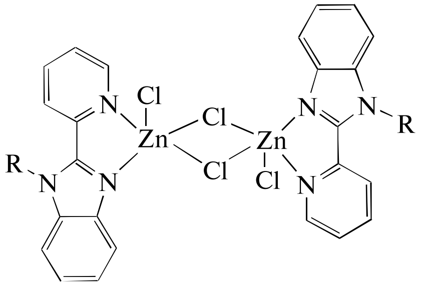
2-(2-Pyridyl)benzimidazole was synthesized by the procedure commonly used to prepare 2-(2-pyridyl)benzazoles based on the reaction of anilines containing a proton-donor group in the *ortho*-position (**V**) with pyridine-2-carbaldehyde (**VI**) [10] to give (**VII**):



(**V**): X = NR, O, S (**VI**) (**VII**)

N-substituted **VII** were prepared by alkylation of 2-(2-pyridyl)benzimidazole.

Complex compounds of type **III** (M = Zn, R = $C_4H_9$, $CH_2$–CH=$CH_2$, $CH_2C_6H_5$) were isolated upon the reaction of **I** with zinc dichloride in methanol. The binulear complex **IIIb** (R = $CH_3$) was prepared in a similar way.



**(IIIb)**

The complex formation induces an increase in the IR stretching frequencies of the benzimidazole ring at 1590–1600 cm$^{-1}$, which is consistent with published data for molecular complexes [29].

According to X-ray diffraction data, 2-(2-pyridyl)benzimidazoles **I** and analogs **VII** exist most often as *trans*-isomers as regards the arrangement of pyridine type nitrogen atoms of the azole and azine substituents relative to the C(2)–C(2)' bond. [25]. Only one example is known where 2-(2-pyridyl)benzimidazole containing a bulky dioxyribose substituent in position 1 exists as the *cis*-isomer [30].

The higher stability of *trans*-isomers and the chelating effect determine the possibility of existence of structures **II–IV**. These structures are important for development of the competitive coordination problem. To our knowledge, complexes of nitrogen bisheterocycles have not been considered previously from this standpoint [3, 31].

The X-ray diffraction data indicate that the compounds have type **III** chelate structure with zinc coordination to both $sp^2$ hybridized nitrogen atoms of the imidazole and pyridine fragments. As a result, irrespective of the nature (Alk, Ar) and the length of substituent R, a fairly planar system of four conjugated rings including the metal ring is formed (Figs. 1–4). The metal rings of all four compounds are slightly folded at the N(1)–N(3) line to form a dihedral angle of 2°–9° between the corresponding planes. In the (pyridyl)benzimidazole fragment, the bond length distribution order typical of free molecule **I** is retained.

The Zn coordination unit in **IIIc–IIIe** (Figs. 2–4) is a somewhat distorted tetrahedron. The dihedral angles between the N(1)Zn(1)N(3) and Cl(1)Zn(1)Cl(2) planes are 96.3°, 93.2°, and 91.7°, respectively (the ideal value is 90°). The linear and angular parameters in the coordination polyhedron of these three compounds are almost identical (Table 3). The Zn–N bond with the pyridine nitrogen atom is 0.1 Å longer than that for the imidazole atom. The Zn–Cl bonds are equal (average length 2.203 Å).

Unlike the previous compounds in which the effect of substituent R on the structure of the complex was minor, in compounds like **IIIb** (Fig 1), the practically monoatomic substituent does not create hindrance that would prevent approach to the neighboring mole-

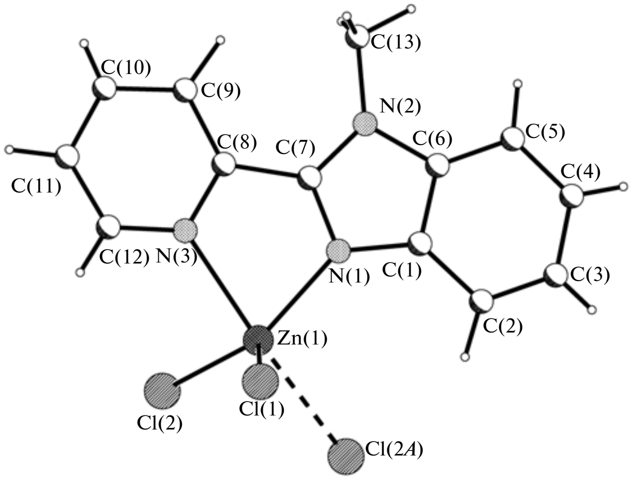


**Fig. 1.** Molecular structure of binuclear complex **IIIb**.

cule to a distance sufficient for interaction. The molecule is dimerized through sharing a chlorine atom of the neighboring molecule to form a four-membered $Zn_2Cl_2$ ring and an additional elongated Zn–Cl bond (2.646 Å). The main Zn–Cl bonds and the Zn–N(3) bonds are also elongated (Table 3).

The insertion of an additional chlorine atom to the coordination sphere of the central atom results in a pronounced rearrangement of the polyhedron, which can rather be classified as a trigonal bipyramid with elongated axial Zn–Cl(2*A*) and Zn–N(3) bonds and shorter equatorial Zn–N(3), Zn–Cl(1), and Zn–Cl(2) bonds (Table 3).

In the considered compound **IIIb**, the small substituent plays rather a passive role in structure formation, while in compounds with longer or more bulky groups, these groups play a key role in the choice of the packing pattern of structural units; as a consequence, compounds with nearly the same structure of the complex form different crystal packings.

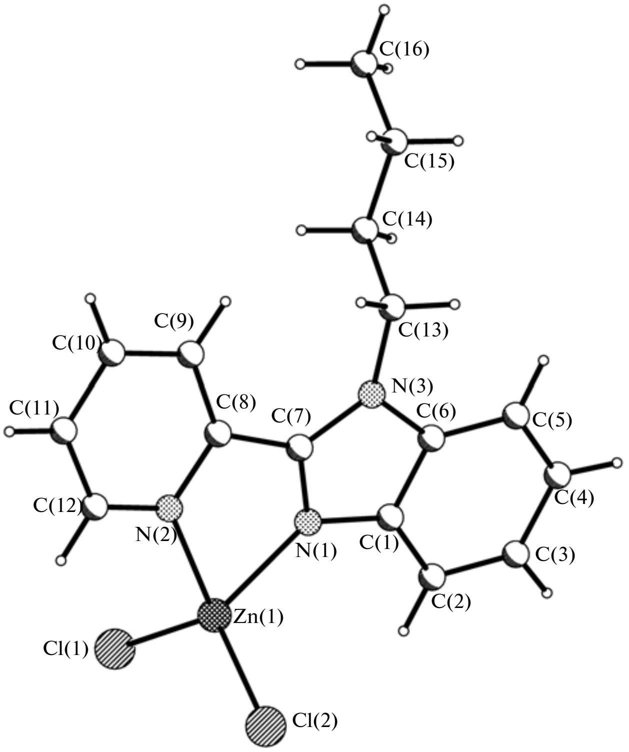


**Fig. 2.** Molecular structure of complex **IIIc**.

The structures with dichloride bridges have not been known previously among 2-(2-pyridyl)benzazole

**Table 3.** Parameters of Zn coordination polyhedron in the complexes **IIIb–IIIe**

| Compound | Bond, *d*, Å | | | | Angle, ω, deg | | |
|---|---|---|---|---|---|---|---|
| | Zn–Cl(1) | Zn–Cl(2)<br>Zn–Cl(2*A*) | Zn–N(1) | Zn–N(3) | Cl(1)ZnCl(2) | N(1)ZnN(3) | Cl(1)ZnCl(2*A*) |
| **IIIb** | 2.239(2) | 2.337(2)<br>2.646(3) | 2.044(5) | 2.216(6) | 97.63(7) | 76.4(2) | 119.83(8) |
| **IIIc** | 2.208(2) | 2.193(1) | 2.036(5) | 2.106(4) | 116.4(7) | 79.3(2) | |
| **IIId** | 2.214(2) | 2.210(2) | 2.016(5) | 2.102(5) | 114.0(8) | 80.2(3) | |
| **IIIe** | 2.194(2) | 2.202(2) | 2.030(4) | 2.098(4) | 119.70(7) | 79.2(2) | |

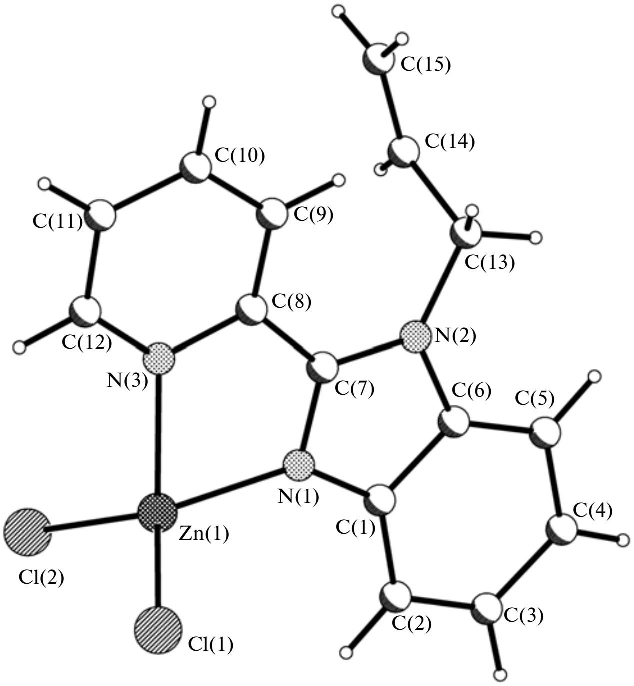


**Fig. 3.** Molecular structure of complex **IIId**.

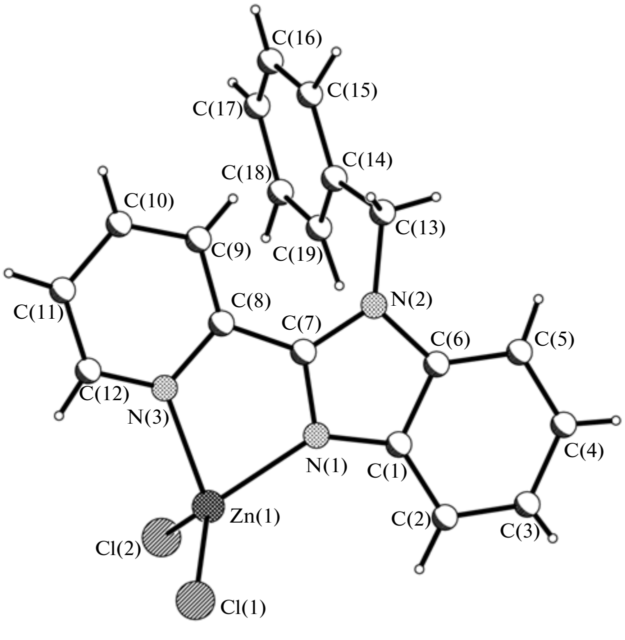


**Fig. 4.** Molecular structure of complex **IIIe**.

zinc complexes, although a bridge of this type occurs in the nickel complex of 1-ethyl-2-(2-phenanthronyl)benzimidazole [16]. Other examples of oligonuclear 2-(2-pyridyl)benzimidazole complexes include structurally characterized chelates obtained from more bulky ligands containing pyridylbenzimidazole fragments [11, 15,17, 18,32-36].

## ACKNOWLEDGMENTS

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