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Heteroleptic Zn(II) Halide Complexes with Iodine-Substituted Benzonitriles: Peculiarities of the Halogen Bond in the Solid State

M. A. Vershinin[a], A. S. Novikov[b], M. N. Sokolov[a], and S. A. Adonin[a, c, *]

[a] *Nikolaev Institute of Inorganic Chemistry, Siberian Branch, Russian Academy of Sciences, Novosibirsk, Russia*

[b] *St. Petersburg State University, St. Petersburg, Russia*

[c] *Favorsky Irkutsk Institute of Chemistry, Siberian Branch, Russian Academy of Sciences, Irkutsk, Russia*

**e-mail: adonin@niic.nsc.ru*

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Abstract—The reactions of zinc(II) bromide with 3- and 4-iodobenzonitriles (3-I-BzCN and 4-I-Bz-CN) afford heteroligand complexes $[L_2ZnBr_2]$ (L = 3-I-BzCN (**I**) and 4-I-BzCN (**II**)), whose structures are determined by X-ray diffraction (XRD) (CIF files CCDC nos. 2253175 (**I**) and 2253176 (**II**)). Both crystal structures contain halogen bonds I···Br linking the $[ZnBr_2L_2]$ fragments into supramolecular layers (**I**) or chains (**II**). The energies of these noncovalent interactions are estimated by quantum-chemical calculations.

Keywords: zinc complexes, N-donor ligands, halogen bonds, crystal structure, quantum-chemical calculations

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INTRODUCTION

Halogen bond (HB) represents a specific type of noncovalent interactions involving halogen atoms acting as electrophiles [1]. The study of this trend has attracted much attention of specialists in the field of supramolecular chemistry during recent several years [2–10].

The search for new "building blocks" capable of forming HB is an interesting problem in the context of HB studies. Many works devoted to the application of perfluorinated iodo- and bromoarenes for these purposes have been published nowadays [11–17]. Supramolecular ensembles involving haloalkanes [18–20], polyhalides [21–25], high-valent iodine derivatives [26–29], etc. can also be mentioned. In some cases, HB was manifested in neutral complexes of the $[M^{II}L_2X_2]$ type, where L is the monodentate halogen-substituted N-donor ligand (halogenated pyridines, quinolines, etc.; X = Cl, Br, or I). Similar compounds were synthesized for the majority of *d* elements [30–33], and especially many examples are known for Cu(II) [34–37].

It can be assumed that the $[M^{II}L_2X_2]$ complexes can be synthesized by analogy to the use of halogen-containing nitriles as ligands. Nevertheless, these examples are not numerous. Considering only chloro-, bromo-, and iodosubstituted benzonitriles, we can say that several their structurally characterized complexes are presently known: for Ag(I) [38–40], Pt(II) [41, 42], Ti(IV) [43], Au(I) [44], and Fe(II) [45].

In this work, we synthesized two new Zn(II) complexes with iodosubstituted benzonitriles: $[L_2ZnBr_2]$ (L = 3-I-BzCN (**I**) and 4-I-BzCN (**II**)). Their structures were determined by XRD. Both structures contain halogen bonds I···Br linking the $[ZnBr_2L_2]$ fragments into layers (**I**) or chains (**II**). The noncovalent interaction energies were estimated by quantum-chemical calculations.

EXPERIMENTAL

The synthesis was carried out in air. The initial reagents were purchased from commercial sources.

Synthesis of complex I. A mixture of anhydrous zinc bromide (84.1 mg, 0.37 mmol) and 3-iodobenzonitrile (173 mg, 0.75 mmol) was dissolved in nitromethane (5 mL) on stirring. The slow evaporation of the solution leads to the formation of colorless crystals of complex **I** suitable for XRD. The yield was 93%.

Synthesis of complex II. A mixture of anhydrous zinc bromide (52.3 mg, 0.23 mmol) and 4-iodobenzonitrile (105.8 mg, 0.46 mmol) was dissolved in nitromethane (5 mL) on stirring. The slow evaporation of the solution leads to the formation of colorless crystals of complex **II** suitable for XRD. The yield was 91%.

XRD of complexes **I** and **II** were carried out on a Bruker D8 Venture diffractometer (MoK_α, λ = 0.71073 Å) at 150 K. Reflection intensities were measured in the ω and φ scan modes of narrow (0.5°) frames. An absorption correction was applied empiri-

Table 1. Crystallographic data and structure refinement details for complexes **I** and **II**

Parameter	Value	
	I	**II**
Empirical formula	$C_{14}H_8N_2Br_2I_2Zn$	$C_{14}H_8N_2Br_2I_2Zn$
FW	683.21	683.21
Crystal system	Monoclinic	Orthorhombic
Space group	*C*2/*c*	*Pnma*
a, Å	23.6038(5)	15.4013(3)
b, Å	5.5376(1)	16.2266(4)
c, Å	16.2083(3)	7.4660(2)
β, deg	121.314(1)	90
V, Å^3	1809.95 (6)	865.83 (8)
Z	4	4
μ, mm^{-1}	9.18	8.90
T_{min}, T_{max}	0.646, 0.747	0.562, 0.746
Number of reflections measured/independent	35112/3468	20342/1819
Number of reflections with ($I > 2\sigma(I)$)	3202	1724
R_{int}	0.039	0.029
Scan range over θ, deg	33.2–2.0	25.7–2.5
$(\sin\theta/\lambda)_{max}$, Å^{-1}	0.770	0.610
Index ranges *h*, *k*, *l*	$-36 \le h \le 36$, $-8 \le k \le 8$, $-24 \le l \le 24$	$-18 \le h \le 18$, $-19 \le k \le 19$, $-9 \le l \le 8$
$R(F^2 > 2\sigma(F^2))$, $wR(F^2)$, *S*	0.015, 0.035, 1.05	0.016, 0.033, 0.95
Residual electron density (max/min), e Å^{-3})	0.49/–0.63	0.82/–0.75

cally using the SADABS program. The structures were solved by SHELXT [46] and refined by full-matrix least squares in the anisotropic (for non-hydrogen atoms) approximation by the SHELXL 2017\1 algorithm [47] in the ShelXle program [48]. The crystallographic data for complexes **I** and **II** are listed in Table 1.

The coordinates of atoms and other XRD experimental parameters were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 2253175 (**I**) and 2253176 (**II**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

It is most likely that the most serious problem in the synthesis of the benzonitrile complexes is the choice of the solvent. The majority of the most popular polar solvents has a higher donor number and, hence, they occupy sites in the coordination sphere of the metal, because of which the synthesis of the target compound becomes impossible. This is the reason why we used nitromethane combining the high polarity and incapability of coordinating. The crystals of complexes **I** and **II** were isolated directly from the reaction mixture during its slow evaporation.

In both compounds, Zn(II) demonstrates the characteristic tetrahedral coordination (Zn–N 2.035 and 2.054 Å in complexes **I** and **II**, respectively). The Zn–Br bond lengths are 2.344 and 2.315–2.345 Å, respectively, which is well consistent with the published data for similar heteroleptic complexes [49–51].

The key peculiarity of both structures is the presence of specific interactions I···Br linking the I atoms of the benzonitrile fragments and bromide ligands of adjacent $[ZnL_2Br_2]$. These contacts can be assumed to exist by a comparison of the corresponding distances with the sum of van der Waals radii (S_W) for the corresponding atoms (3.81 Å, Bondi [52, 53]). In the case of complex **I** (I–Br 3.498 Å), the system of these interactions leads to the formation of supramolecular layers (Fig. 1), and chains are formed (Fig. 2) in the case of complex **II** (I–Br 3.672 Å).

In order to understand the nature and to estimate the energy of noncovalent interactions I···Br in the crystals of complexes **I** and **II** (these short contacts can be classified as typical halogen bonds [54]), we per-

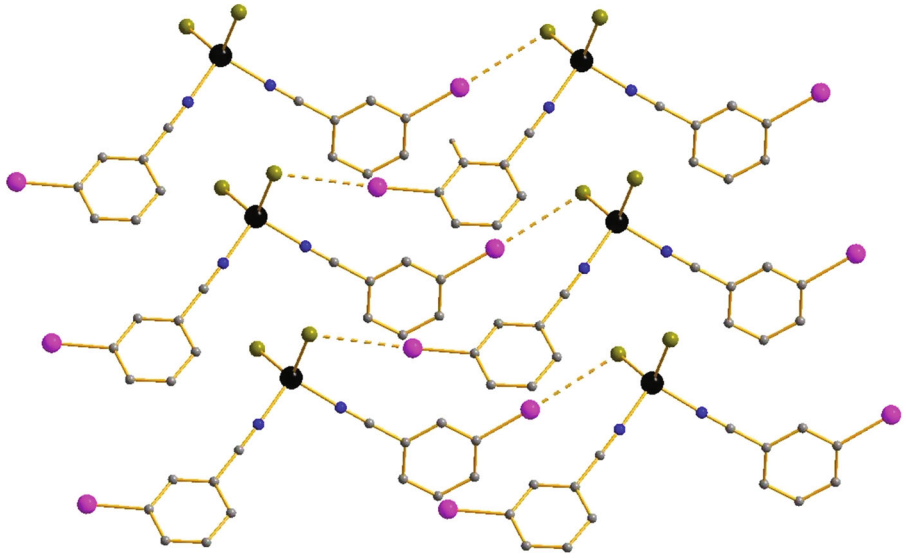

Fig. 1. Supramolecular layers in the structure of compound **I**. Hereinafter: Zn is black, I is violet, Br is olive-green, N is blue, and C is gray; halogen bonds are shown by dash, and hydrogen atoms are omitted.

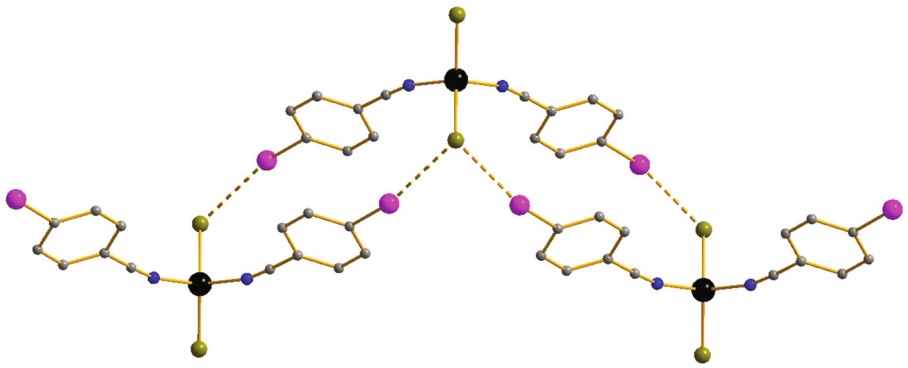

Fig. 2. Supramolecular chains in the structure of compound **II**.

formed quantum-chemical calculations in terms of the density functional theory (ωB97XD/DZP-DKH) [55–57] using the Gaussian-09 software and topological analysis of the electron density distribution by the QTAIM method [58] using the Multiwfn program (version 3.7) [59]. This approach has successfully been applied previously for studying the properties of various noncovalent interactions [60, 61] and coordination bonds [62, 63] in the transition metal complexes. The results of the estimation of the parameters corresponding to the noncovalent interactions are presented in Table 2. The diagrams of contour lines of the electron density Laplacian distribution, bond paths, and zero flow surfaces corresponding to the noncovalent interactions in the crystals of complexes **I** and **II** are shown in Figs. 3 and 4, respectively. An analysis makes it possible to reveal both the aforementioned I···Br interactions and Br···Br interactions, although the corresponding distances substantially exceed S_W (3.66 Å).

The values of the electron density, electron density Laplacian, total energy density, potential energy density, and kinetic energy Lagrangian at the bond critical points (3, –1) corresponding to the I···Br and Br···Br noncovalent interactions in the crystals of complexes **I** and **II** are quite typical of this type supramolecular contacts involving halogen atoms. The estimate values of the energy of these contacts vary from 1.1 to 2.9 kcal/mol. The ratio of the potential energy density and kinetic energy Lagrangian at the bond critical points (3, –1) indicates the absence of a substantial fraction of the covalent component in these supramolecular contacts. The visualization of the I···Br and Br···Br noncovalent interactions in complexes **I** and **II** in the framework of the formalism of noncovalent interaction analysis (NCI analysis [65]) is shown in Fig. 5.

Table 2. Electron density ρ(**r**), electron density Laplacian $\nabla^2\rho(\mathbf{r})$, total energy density H_b, potential energy density $V(\mathbf{r})$, and kinetic energy Lagrangian $G(\mathbf{r})$ (atomic units) at the bond critical points (3, −1) corresponding to the noncovalent interactions I···Br and Br···Br in compounds **I** and **II**, lengths of these constants l (Å), and their energies E (kcal/mol)

Contact	ρ(**r**)	$\nabla^2\rho(\mathbf{r})$	H_b	$V(\mathbf{r})$	$G(\mathbf{r})$	l	E*
1 I···Br	0.013	0.034	0.000	−0.008	*0.008*	3.498	2.9
2 I···Br	0.005	0.017	0.000	−0.003	*0.003*	4.011	1.1
2 I···Br	0.010	0.027	0.000	−0.006	*0.006*	3.672	2.1
2 Br···Br	0.007	0.018	0.000	−0.004	*0.004*	3.749	1.4

* $E = 0.57G(\mathbf{r})$ (correlation was developed specially for the estimation of the energy of the noncovalent interactions involving bromine atoms) [64].

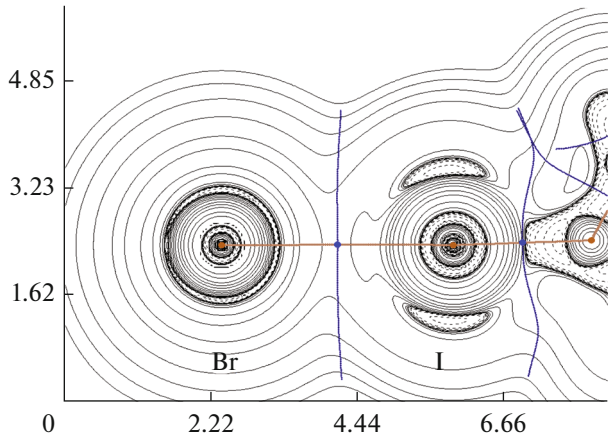

Fig. 3. Diagram of contour lines of the electron density Laplacian distribution $\nabla^2\rho(\mathbf{r})$ and bond paths and zero flow surfaces corresponding to the I···Br noncovalent interactions in the crystal of compound **I**. Bond critical points (3, −1) are blue-colored, and nuclear critical points (3, −3) are shown by pale brown. Units of measure of length are Å.

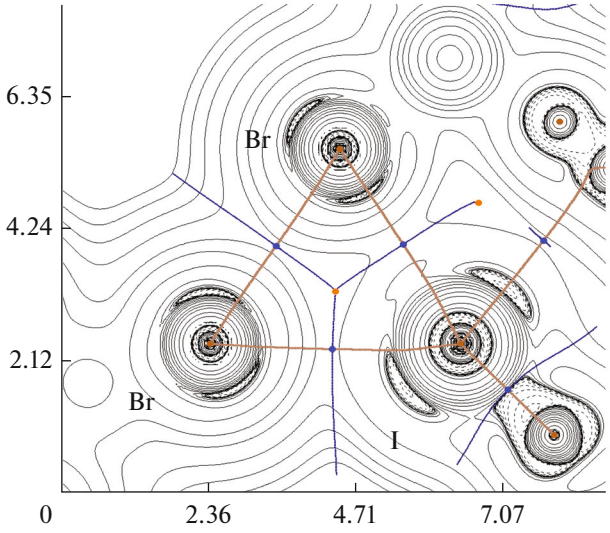

Fig. 4. Diagram of contour lines of the electron density Laplacian distribution $\nabla^2\rho(\mathbf{r})$ and bond paths and zero flow surfaces corresponding to the I···Br noncovalent interactions in the crystal of compound **II**. Bond critical points (3, −1) are blue-colored, and nuclear critical points (3, −3) are shown by pale brown. Units of measure of length are Å.

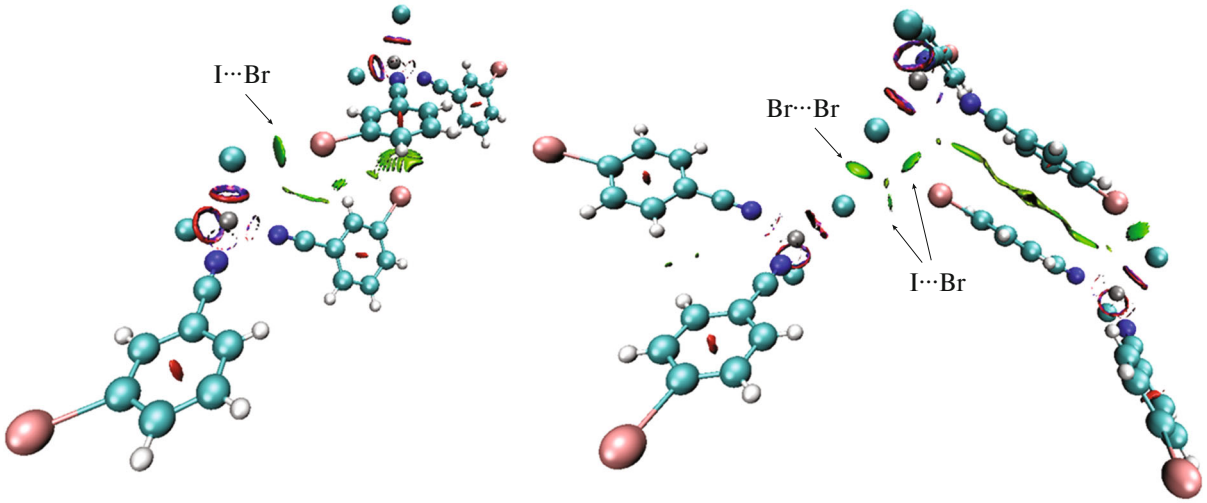

Fig. 5. Visualization of the I···Br and Br···Br noncovalent interactions in the crystals of compound **I** (left) and **II** (right) in the framework of the formalism of noncovalent interaction analysis (NCI analysis).

Thus, halogenated benzonitriles can act as building blocks for the formation of HB-based supramolecular ensembles. The use of nitromethane makes it possible to surmount problems related to the competitive coordination of the solvent molecules.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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