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The authors congratulate Academician of the Russian Academy of Sciences I.L. Eremenko on his 70th jubilee

Linear Metal-Organic Frameworks Based on Bis(1-Benzotriazolyl)methane and Zinc and Copper Nitrates

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Abstract—Complexes $\{[(Zn(Bbtm)(H_2O)_4](NO_3)_2\}_n$ (**I**) and $[Cu(Bbtm)(NO_3)_2]_n$ (**II**) are formed due to the reactions of solutions of zinc and copper(II) nitrates with the bis(1,1'-1,2,3-benzotriazolyl)methane ligand (Bbtm). Their crystal structures are determined by X-ray diffraction analysis (CIF files CCDC nos. 1963126 (**I**) and 1963127 (**II**)). Complex **I** is a linear metal-organic framework (1D-MOF) in which the octahedral coordination of the central atom is provided by four water molecules and two nitrogen atoms of two Bbtm molecules in the *trans* position. In the structure of complex **II**, the coordination sphere of copper contains two nitrogen atoms of the Bbtm ligands and four oxygen atoms of two nitrate anions, one of which is bridging like the Bbtm ligand. This makes it possible to describe the structure of complex **II** as 3D-MOF. The luminescence spectra are recorded for earlier undescribed compound **I**. The emission maximum is observed at 363 nm. Compound **I** is also tested as a catalyst for the cycloaddition of CO_2 to epoxides. The synthesized MOF efficiently catalyzes the cycloaddition reactions for both monosubstituted and disubstituted epoxides.

Keywords: zinc, copper, complexes, benzotriazole, catalysis, X-ray diffraction analysis, luminescence

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INTRODUCTION

Metal-organic frameworks (MOF) are used in the storage and separation of gases and liquids, sensorics, and catalysis [1, 2]. Zinc and copper cations are components of many MOF, and these materials are often used as catalysts [3, 4]. Double-charge zinc and copper cations are intermediate according to Pearson's scale of absolute hardness and, hence, can be coordinated by both oxygen- and nitrogen-containing donor ligands.

Various zinc complexes with 1,2,3-benzotriazole have been described at present [5–8]. These compounds represent coordination polymers of various dimensionalities, and each benzotriazole ring coordinates the zinc atom at the N^1 and N^3 atoms. The zinc and cadmium complexes of the ligands based on 1,2,3-benzotriazole are able to exhibit ligand-centered fluorescence [5, 6, 8, 9], the excitation usually occurs in a range of 250–290 nm, and the emission is observed at 350–400 nm.

The bis(1*H*-benzo[1,2,3]triazol-1-yl)methane ligand has first been synthesized [10] by the reaction of 1,2,3-benzotriazole with formaldehyde in the presence of dimethylamine in glacial acetic acid. A more convenient method of the synthesis is the reaction of 1,2,3-benzotriazole with dichloromethane [11]. Several Bbtm complexes, for example, the derivatives of copper $[Cu(Bbtm)(NO_3)_2]_n$, $[Cu(Bbtm)(H_2O)\text{-}(NO_3)_2]_n$, $[Cu_3(Bbtm)_4(H_2O)_8]A_6$ (A = BF_4^-, ClO_4^-) [12], $[Cu(Bbtm)Cl_2]_n$ [13], and $[Cu_2(Bbtm)_2Br_4]_n$ [14], cadmium $[Cd_2(Bbtm)(H_2O)\text{-}(C_2H_5OH)Cl_4]_n \cdot 1.5nH_2O$ [9], and some platinum metals [13], have been described to the present time. In these compounds, the coordination polyhedra and dimensionalities of the formed polymers differ substantially. No zinc complexes with Bbtm were described to the moment.

The MOF based on transition metals are widely used in the cycloaddition of carbon dioxide to epoxides [15–17]. The zinc-based MOF are among the most efficient compounds in this reaction [18–26], since they make it possible to selectively synthesize cyclic carbonates in high yields [18, 23–26] and to conduct the reactions under mild conditions [18, 23–25] or with a small amount of the catalyst [19]. The disubstituted substrates can be carboxylated in the presence of the heterobimetallic gold–zinc MOF under mild conditions [27]. It should be mentioned that the polynuclear cluster acted as the metal center in these Zn-MOF in the most part of cases.

The purpose of this work is to study the coordination possibilities of the Bbtm ligand in the formation of the zinc and copper(II) complexes with the cations of intermediate Pearson's hardness: {[(Zn(Bbtm)-$(H_2O)_4](NO_3)_2\}_n$ (**I**) and $[Cu(Bbtm)(NO_3)_2]_n$ (**II**). In addition, the luminescence properties of the zinc complex and its catalytic activity in the carboxylation of mono- and disubstituted epoxides were studied.

EXPERIMENTAL

Commercially available reagents and solvents were used, in particular, zinc and copper(II) nitrates (reagent grade, Reakhim). Ligand Bbtm was synthesized using a known procedure [11] and characterized by ^{1}H NMR spectroscopy.

IR spectra were recorded on a FTIR Spectrum One Perkin-Elmer spectrometer in KBr pellets in a range of 400–4000 cm^{-1} with a resolution of 0.5 cm^{-1}.

The fluorescence spectra of powdered samples were measured on a Varian Cary Eclipse spectrofluorimeter. All luminescence and excitation spectra were measured with the applied instrumental function correction. The measurements were carried out in 5-mm quartz cells at room temperature.

Thermal analysis (TG) was conducted on a NETZSCHSTA 409 PC/PG instrument in air with a heating rate of 10°C/min.

Elemental analyses were carried out using a Fision Instruments 1108 microanalyzer.

Synthesis of complex I. A solution of zinc nitrate hexahydrate (0.1487 g, 0.5 mmol) in water (5 mL) was placed in a tube, after which a water–acetonitrile (1 : 1) mixture (1 mL) and then a layer of a solution of Bbtm (0.1251 g, 0.5 mmol) in acetonitrile (2 mL) were poured into the tube avoiding layer mixing. The tube was hermetically closed, and white crystals of complex **I** formed in a week were filtered off and dried in a desiccator over $CaCl_2$.

IR (KBr), ν, cm^{-1}: 3650–3100 s, 3098 w, 3020 w, 2968 vw, 1972 vw, 1937 vw, 1845 vw, 1811 vw, 1655 s, 1615 w, 1592 w, 1494 vw, 1457 w, 1387 vs, 1333 vw, 1292 w, 1212 m, 1165 w, 1003 w, 987 vw, 934 w, 830 w, 778 w, 755 m, 433 w.

For $C_{13}H_{18}N_8O_{10}Zn$

Anal. calcd., %	C, 30.51	H, 3.55	N, 21.90
Found, %	C, 30.91	H, 3.72	N, 22.05

Synthesis of complex II was carried out according to a procedure similar to that for complex **I** using $Cu(NO_3)_2 \cdot 3H_2O$.

IR (KBr), ν, cm^{-1}: 3022 vw, 2965 w, 1972 m, 1932 w, 1841 vw, 1615 m, 1490 s, 1377 vs, 1334 m, 1212 m, 1168 w, 1111 m, 1000 w, 987 vw, 933 vw, 831 w, 877 w, 778 w, 751 m.

For $C_{13}H_{10}N_8O_6Cu$

Anal. calcd., %	C, 35.66	H, 2.30	N, 25.59
Found, %	C, 36.12	H, 2.40	N, 25.80

X-ray diffraction analysis (XRD). Single crystals of complexes **I** and **II** were obtained by the slow evaporation of acetonitrile solutions. Experimental data were collected on an Xcalibur Sapphire 3 four-circle automated diffractometer at 120 K (MoK_α radiation, λ = 0.71073 Å, graphite monochromator) in the κ scan mode. An absorption correction was applied by measurements of equivalent reflection intensities [28]. The structures were solved by a direct method and refined by full-matrix anisotropic least squares for F^2 for all non-hydrogen atoms [29]. Hydrogen atoms were placed in the calculated positions and refined using the riding model. The crystallographic data and the experimental and structure refinement details are presented in Table 1.

The full tables of atomic coordinates, bond lengths, and bond angles in the structures of compounds **I** and **II** were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 1963126 and 1963127, respectively; http://www.ccdc.cam.ac.uk).

Study of the catalytic activity. Epoxide **1** (1 mmol), tetrabutylammonium bromide (0.016 mmol), and the catalyst (10 mg) were placed in an RLP15ML low-pressure glass reactor (15 mL) equipped with a gas supply system, a manometer, and a magnetic stirrer. The reaction was carried out without a solvent in a mixture of the substrates. The reactor was filled with CO_2 three times, and then the gas pressure was maintained at 5 atm (in the case of **1a**–**1c**) or 8 atm (for **1d** and **1e**). The reaction mixture was stirred at 80°C (in the case of **1a**–**1d**) or 120°C (for **1e**) for 20 h. After this, the reaction mixture was cooled to room temperature, the catalyst was filtered off, and the mixture was analyzed by NMR spectroscopy. Product **2** was purified by column chromatography on silica gel using petroleum ether as an eluent. The isolated product was dried in vacuo.

RESULTS AND DISCUSSION

The syntheses of the complexes, under the same mild conditions, gave basically different structures: linear polymeric hydrate $\{[Zn(Bbtm)(H_2O)_4](NO_3)_2\}_n$ (**I**) and anhydrous 2D polymer $\{[Cu(Bbtm)(NO_3)_2]_n\}$ (**II**). The central atom in complex **I** exists in the high-symmetry octahedral environment, whereas in polymer **II** the symmetry of the environment is substan-

Table 1. Crystallographic parameters, structure refinement details, and detection conditions for compounds **I** and **II**

Parameter	Value	
	I	**II**
Crystal system	Monoclinic	Orthorhombic
Space group	$C2/c$	$Pna2_1$
a, Å	18.7162(8)	9.1109(6)
b, Å	9.9145(3)	15.1737(10)
c, Å	13.9023(6)	11.2731(7)
β, deg	114.6680(10)	90
V, Å^3	2107.86(15)	1558.46
ρ_{calc}, g/cm^3	0.971	1.124
Z	4	2
Scan range over θ, deg	2.395–31.387	2.251–28.699
Range of indices	$-15 \le h \le 26$, $-12 \le k \le 11$, $-20 \le l \le 17$	$-11 \le h \le 12$, $-20 \le k \le 20$, $-15 \le l \le 15$
Number of measured/independent reflections (R_{int})	6839/3320 (0.0231)	22563/4029 (0.0693)
Reflections with $I > 2\sigma(I)$	2711	3118
Number of refined parameters	144	254
GOOF for F^2	1.040	1.013
R_1 and wR_2, $I > 2\sigma(I)$	$R_1 = 0.0568$, $wR_2 = 0.1445$	$R_1 = 0.0363$, $wR_2 = 0.0680$
$\Delta\rho_{max}$, $\Delta\rho_{min}$, e Å^{-3}	1.988, –0.666	0.384, –0.643

tially distorted, which is induced by the Jahn–Teller effect characteristic of Cu^{2+}. The crystal structure of compound **II** has been described earlier [12] and, therefore, we concentrated our attention on the zinc complex.

The IR spectra of both compounds contain two bands corresponding to the vibrations of the nitrate ion: 1377–1387 and 830–831 cm^{-1}. The bands characteristic of the vibrations of the N–CH_2 bond of the 1,1'-Bbtm ligand are also observed: 1610, 1490, and 950 cm^{-1}.

According to the differential thermal analysis (DTA) data (Fig. 1), complex **I** decomposes in four stages. At first, four water molecules of the nearest coordination sphere of zinc undergo the endothermic removal from the complex at 80–180°C. On heating to 240°C, the partial oxidative decomposition of the Bbtm ligand occurs with the elimination of CO_2, N_2, and H_2O (for example, unsubstituted benzotriazole undergoes self-ignition at 210°C), and the partial decomposition to ZnO finishes above 600°C.

According to the XRD data, the structure of complex **I** (Fig. 2a) is a zigzag linear polymer. The adjacent polymeric chains are linked by the nitrate ions to thus form a planar layer consisting of chains remote at a distance of 3.13–4.26 Å. The nitrate ions retain the chains together by the formation of hydrogen bonds (from 2.810 to 2.977 Å in length) with the water molecules from the coordination environment of the zinc atoms. The zinc atom is arranged in the poorly distorted octahedron (Fig. 3) formed by two nitrogen atoms of the ligand (Zn–O distances 2.156 Å) and four oxygen atoms of the water molecules (the Zn–O(H_2O) distances in pairs are equal to 2.088 and 2.103 Å). The $N_{Bbtm}ZnO_{H2O}$ angles in pairs are equal to 87.74° and 92.26°, which results in a slight distortion of the coordination polyhedron. The ligand molecules bound to the zinc atom are arranged in the trans position to each other in such a way that the inflections of the polymeric chain are induced only by the nonlinear character of the ligand molecules. The $NC_{CH_2}N$ angle in the ligand (109.21 Å) is smaller than the same angle in the uncoordinated ligand (111.87 Å).

As in the case of the zinc complex, the structure of compound **II** (Fig. 2b) represent bent chains bound into a planar layer, but the distance between the adjacent chains is longer: 4.20–5.18 Å. In the copper complex, the adjacent polymers are linked by the nitrate ions that coordinate (by the oxygen atoms) the copper atoms arranged in different polymeric chains. The copper atom exists in a polyhedron formed of two nitrogen atoms of the ligand (lengths 1.993 and

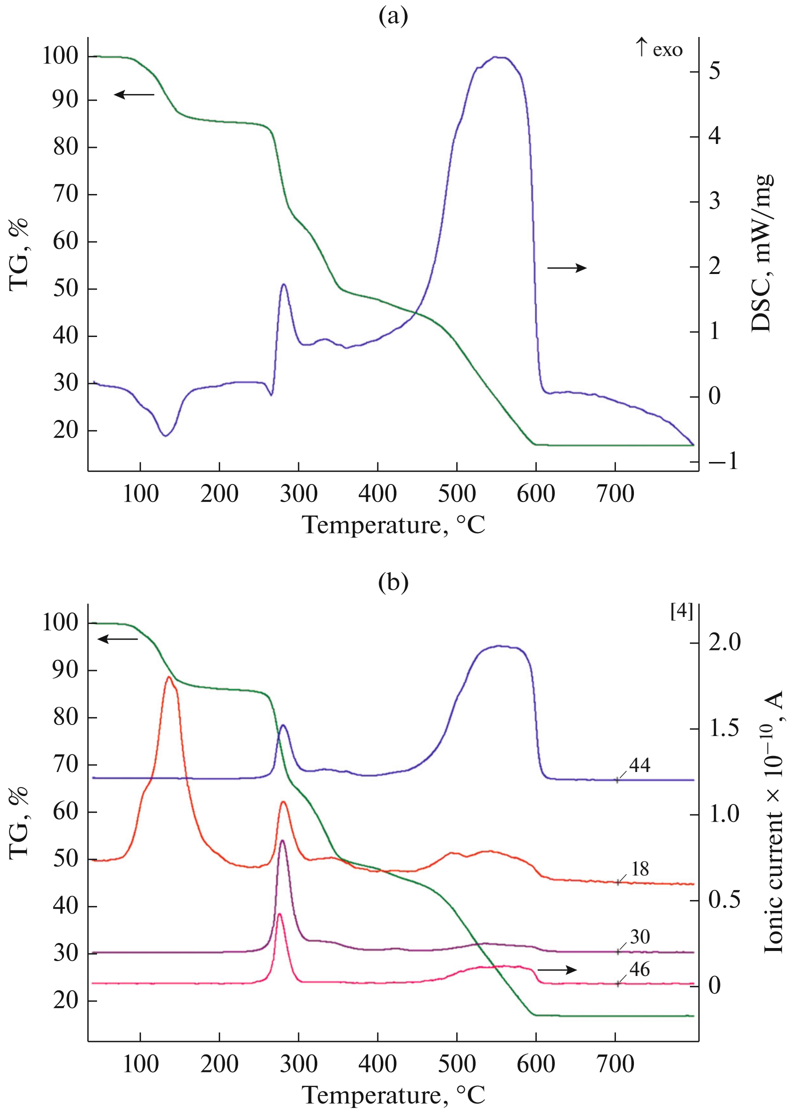


Fig. 1. (a) Mass loss (TG) and DTA curves and (b) signals from the mass spectrometric detector of the thermal decomposition products for complex **I**.

2.015 Å) and four oxygen atoms of the nitrate ions. One of the ions acts as the chelating agent, and another ion is bridging. The coordination of the nitrate ions to the copper atoms of the polymeric chains results in the elongation of the bond to ~2.5 Å, inducing a strong distortion of the coordination polyhedron of copper, which is intermediate between an octahedron and a trigonal prism. In the first case, the polyhedron can be described as an octahedron with the almost square base (O(1), N(3), O(4), O(8)) and two O(6) oxygen atoms in the remote axial positions. Both O(6) atoms of various nitrate groups are shifted from the octahedron center to the O(4) vertex. When the polyhedron is described as a distorted trigonal prism, the O(1)–N(6)–O(8) and N(3)–O(4)–O(6) bases strongly unfolded relative to each other can be distinguished. An analysis of the structure using the Shape program package shows that the routes of minimum distortion for the octahedron and trigonal prism are 5.403 and 12.071%, respectively. The Cu–O(NO_3) bond lengths with the O(4) and O(6) oxygen atoms of the chelating nitrate ion are 1.992 and 2.533 Å, that with the O(6) atom and copper atom of the adjacent fragment is 2.501 Å, and the bond length with the O(1) atom of the nonchelating nitrate ion is 1.956 Å. The angle between the oxygen atoms of one nitrate ion is 55.92°, and the angles $N(1)_{NO3}CuN(2)_{NO3}$, $N(2)_{NO3}CuN(3)_{NO3}$, and $N(3)_{NO3}CuN(1)_{NO3}$ are 116.14°, 104.21°, and 83.10°, respectively. The $NC(CH_2)N$ angle in the Bbtm ligand is 110.53°. Undoubtedly, the reason for such a serious distortion of the octahedral geometry is related to the manifestation of the Jahn–Teller effect characteristic of the hexacoordinated Cu^{2+} derivatives.

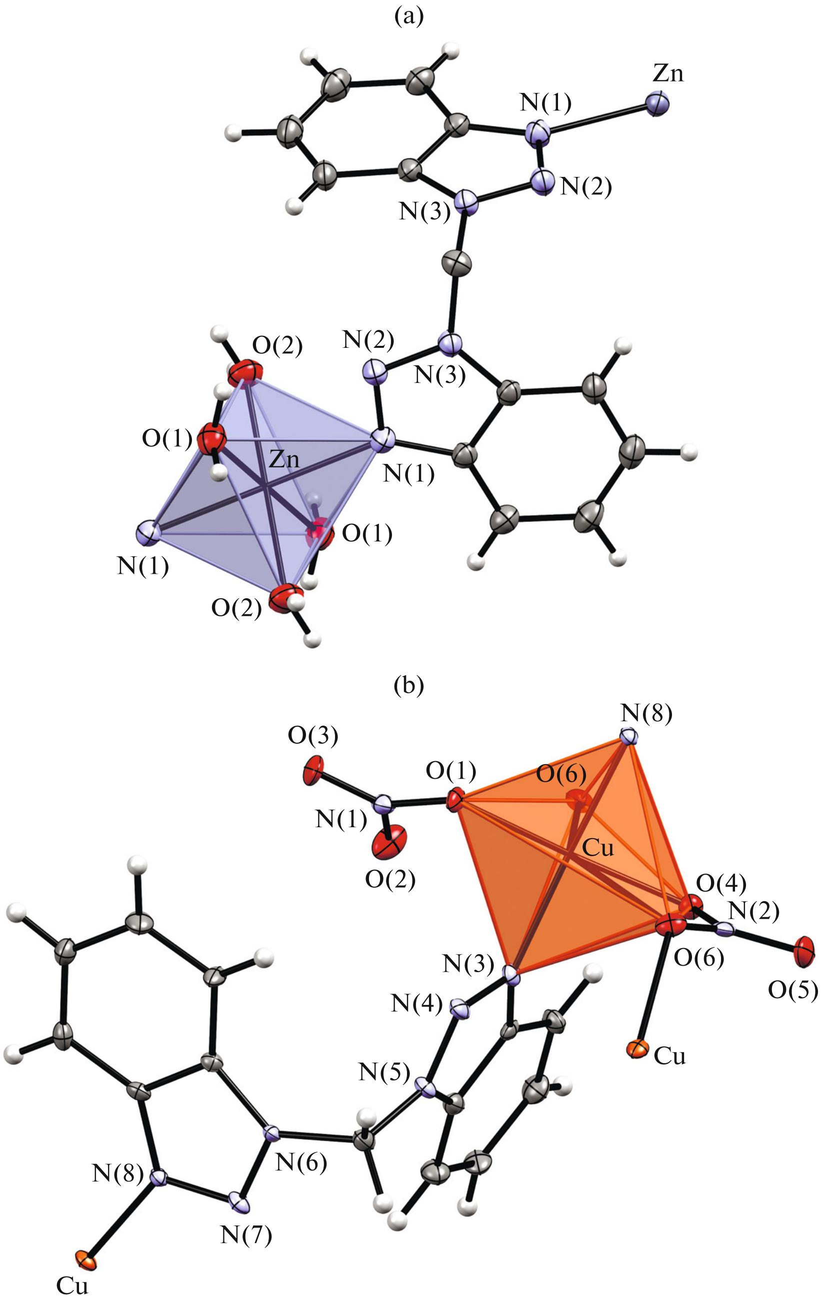
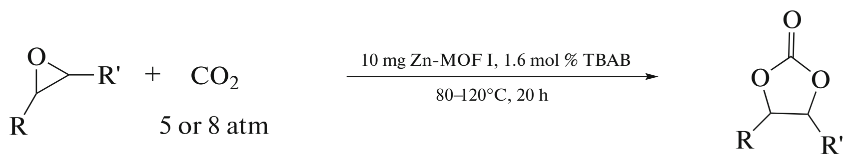


Fig. 2. Molecular structures of complexes (a) **I** and (b) **II**.

Under the same conditions, zinc and copper nitrate react with the ligand in different ways to form compounds with different compositions. This is likely related to the affinity to copper(II) ions to the formation of complexes with a distorted octahedral environment and a comparatively low flexibility of the ligand based on the conjugated benzotriazole system.

The UV-irradiated (at 254 nm) zinc complex demonstrates the blue fluorescence, which was studied by the excitation and emission spectra recorded for this purpose (Fig. 4). The maximum in the excitation spectrum is observed at 265 nm, and that in the emission spectrum is at 365 nm with a shoulder at 395 nm. The positions and shapes of the bands correspond, on the whole, to the luminescence spectra of the zinc complexes with 1,2,3-benzotriazole [5, 6, 8].

To study the catalytic activity of the synthesized MOF, we chose a series of monosubstited oxiranes **1a–1c**, disubstituted isobutylene oxide **1d**, and cyclohexene oxide **1e**. The reaction of epoxides with CO_2 is presented in Scheme 1.



1a–1e
1 mmol

R = H, R' = Me (**a**), Ph (**b**), CH_2Cl (**c**), Me_2 (**d**);
R,R' = $-(CH_2)_4-$ (**e**)

2a–2e

Scheme 1.

The reaction was carried out under the conditions optimized and used previously for epoxide carboxylation in the presence of the heterogeneous zinc catalyst [30]: 60–80°C, $P_{CO_2} = 5$ atm, and 1.6 mol % tetrabutylammonium bromide as the nucleophilic additive. The necessity of a nucleophilic additive was shown in many works on the cycloaddition of CO_2 to epoxides [31], and this additive is sufficient, in many cases, for the reaction to occur in the absence of a metal-containing catalyst [32]. Although several examples of the successful synthesis without this cocatalyst are available [20, 33], the most part of the MOF-based catalytic systems requires the addition of the catalyst [18, 19, 21, 22, 24–26]. As can be seen from the data in Table 2, monosubstituted cyclic carbonates **1a–1e** were synthesized in high yields (87–91%). An increase in the pressure to 8 atm was required for disubstituted epoxides, but the yields of compounds **1d** and **1e** remain moderate. We succeeded to carboxylate 1,1-dimethyloxirane **1d** in a yield of 59%, whereas the corresponding carbonate **2e** was formed only in 42% yield even with the temperature increase to 120°C in the case of cyclohexene oxide **1e**. However, this result exceeds the efficiency of some zinc-based MOF under both mild [22, 24, 25] and comparable [26] conditions.

Thus, under equivalent conditions, copper and zinc nitrates with Bbtm form complexes **I** and **II** differed substantially in composition and structure. Both compounds are MOF, but the structure of copper nitrate is transformed into the three-dimensional polymer due to the bridging nitrate ions along with chain formation due to the bridging molecules of the Bbtm ligand. In the structure of complex **I**, the adjacent chains interact only due to hydrogen bonds of the inner-sphere water molecules and nitrate ions. Compound **I** exhibits the ligand-centered fluorescence in the near-UV range. In addition, this linear MOF was tested as a catalyst for the cycloaddition of CO_2 to epoxides and showed a high efficiency compared to other zinc-containing MOF.

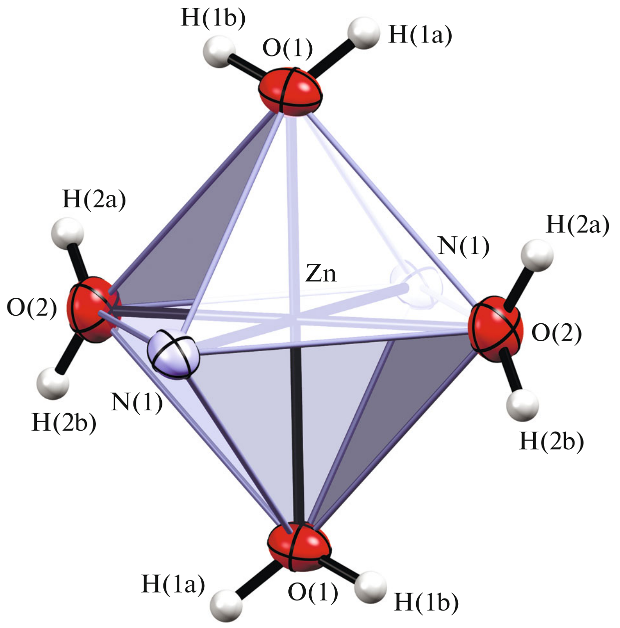


Fig. 3. Coordination polyhedron of zinc in complex **I**.

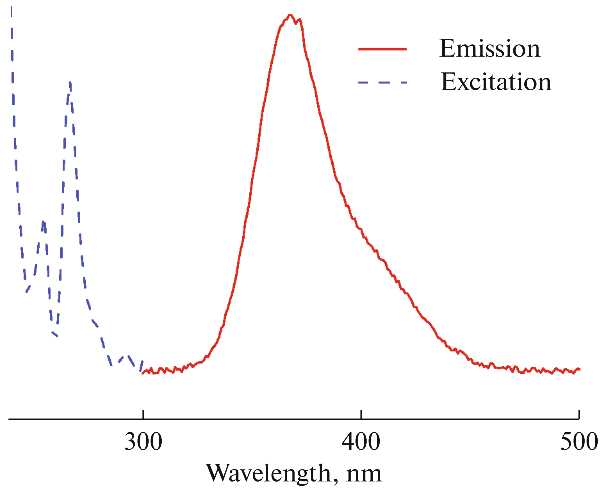


Fig. 4. Excitation and luminescence spectra of complex **I**.

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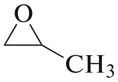
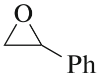
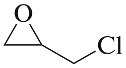
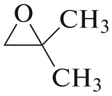
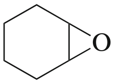
The X-ray diffraction studies were carried out on the equipment of the Center for Collective Use at the Kurnakov Institute of General and Inorganic Chemistry (Russian Academy of Sciences) in terms of the state assignment of the

Table 2. Results of the study of the catalytic activity of complex **I**

Substrate	Temperature, °C	Yield, %*
 **(1a)**	80	91
 **(1b)**	80	89
 **(1c)**	80	87
 **(1d)**	80	59**
 **(1e)**	120	42**

* 1 mmol epoxide, 1.6 mol % Bu_4NBr, 10 mg Zn-MOF **I**, P_{CO_2} = 5 atm, without solvent.

** P_{CO_2} = 8 atm.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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