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2D Coordination Polymers of Zn(II) with Diethylmalonic Acid Dianions and 4,4'-bipyridine: Synthesis and Structure

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Abstract—Two new coordination compounds of zinc(II) with diethylmalonic acid anions (Et_2mal^{2-}) and 4,4'-bipyridine (4,4'-bipy) are synthesized: $\{[Zn(H_2O)(4,4'\text{-bipy})(Et_2mal)]\cdot 0.5C_2H_5OH\cdot 1.5H_2O\}_n$ (**I**) and $\{[Zn_4(H_2O)_2(4,4'\text{-bipy})_3(Et_2mal)_4]\cdot 6H_2O\}_n$ (**II**). According to the XRD data (CIF files CCDC nos. 2323336 (**I**) and 2323337 (**II**)), both compounds are 2D polymers with the **sql** and **bey** topology, respectively. The choice of the initial zinc salt and solvent predetermines the compositions and structures of the polymers under similar synthesis conditions.

Keywords: coordination polymers, diethylmalonic acid, zinc(II), substituted malonates, dicarboxylates, 4,4'-bipyridine

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INTRODUCTION

The class of coordination 3*d*-metal polymers attracts rapt attention of researchers owing to the variety of their properties and a broad range of application fields. These compounds can possess catalytic [1, 2], magnetic [3–5], and luminescence [6, 7] properties. A possibility of using them for the separation of industrial gases and liquid mixtures [8–10] is of special interest. Various dicarboxylate ligands are widely applied for the synthesis of coordination polymers, since the presence of two carboxylate groups favors their bridging coordination [11, 12].

The heteroligand coordination polymers of zinc(II) containing anions of various dicarboxylic acids and N-donor ligands are actively studied as sorption and luminescent materials [13–16] and serve as a basis for studying photochemical reactions [17–19]. The zinc malonate coordination polymers with additional N-donor ligands are presented to a higher extent by the compounds with anions of unsubstituted malonic acid [20–34]. However, the use of its substituted analogs makes it possible to prepare complexes with different structure. In addition, the physicochemical properties of these molecules can be varied depending on the substituent nature and, correspondingly, variants of packing in the crystal [35–38]. The chemistry of zinc diethyl malonate coordination polymers with additional N-donor ligands is presently restricted by single examples [39], and some heteroligand copper(II) and manganese(II) diethyl malonates were described [40, 41].

In this work, we demonstrate two methods for the synthesis of the zinc(II) coordination polymers with diethylmalonic acid (H_2Et_2mal) and 4,4'-bipyridine (4,4'-bipy) as the bridging N-donor ligand giving $\{[Zn(H_2O)(4,4'\text{-bipy})(Et_2mal)]\cdot 0.5C_2H_5OH\cdot 1.5H_2O\}_n$ (**I**) and $\{[Zn_4(H_2O)_2(4,4'\text{-bipy})_3(Et_2mal)_4]\cdot 6H_2O\}_n$ (**II**), respectively. The structural and topological features of the synthesized compounds were studied. This system was found to provide a diversity of products when using different initial salts and solvents during crystallization.

EXPERIMENTAL

All compounds were synthesized in air using distilled water. The following commercially available reagents were used: $Zn(NO_3)_2\cdot 6H_2O$ (high-purity grade, Khimmed, Russia), $ZnSO_4\cdot 7H_2O$ (high-purity grade, REAKHIM, Russia), diethylmalonic acid (H_2Et_2Mal, 98%, Sigma Aldrich), KOH (reagent grade, Khimmed), $Ba(OH)_2\cdot H_2O$ (98%, Sigma Aldrich), 4,4'-bipyridine (4,4'-bipy, 98%, Alfa Aesar), ethanol (EtOH, 96%), and acetonitrile (reagent grade, Khimmed). IR spectra were recorded on a Spectrum 65 (Perkin Elmer) FT-IR spectrophotometer using the attenuated total internal reflectance (ATR) method in a frequency range of 4000–400 cm^{-1}.

Synthesis of $\{[Zn(H_2O)(4,4'\text{-bipy})(Et_2mal)]\cdot 0.5C_2H_5OH\cdot 1.5H_2O\}_n$ (I). The synthesis was carried

out by the slow mixing method. Weighed samples of diethylmalonic acid (0.016 g, 0.1 mmol) and KOH (0.011 g, 0.2 mmol) were dissolved in distilled water (10 mL). A weighed sample of $Zn(NO_3)_2 \cdot 6H_2O$ (0.030 g, 0.1 mmol) was added to the resulting solution, and the mixture was stirred until complete dissolution. The obtained mixture was placed on the bottom of a tube. Distilled water (2 mL) and then EtOH (5 mL) were carefully layered on the mixture. A weighed sample of 4,4'-bipy (0.031 g, 0.2 mmol) was dissolved in EtOH (10 mL), and the formed light yellow solution was carefully layered on the top. Colorless crystals suitable for XRD were formed in 3 weeks. The crystals of compound **I** were filtered off, washed with cold MeCN ($T = 5°C$), and dried in air. The yield was 0.016 g (36% based on $Zn(NO_3)_2 \cdot 6H_2O$).

IR (ATR; ν, cm^{-1}): 3520 w, 3182 w br, 2968 w, 2877 w, 1661 w, 1597 s, 1518 m, 1465 m, 1411 m, 1369 m, 1254 w, 1222 w, 1183 w, 1152 w, 1067 w, 1046 w, 1010 w, 945 w, 864 w, 817 s, 770 w, 723 m, 689 m, 633 m, 585 m, 527 w, 470 m, 425 w.

Synthesis of $\{[Zn_4(H_2O)_2(4,4'\text{-bipy})_3(Et_2mal)_4] \cdot 6H_2O\}_n$ (II). The synthesis was conducted by the slow mixing method. Weighed samples of diethylmalonic acid (0.016 g, 0.1 mmol), $Ba(OH)_2 \cdot H_2O$ (0.020 g, 0.1 mmol), and $ZnSO_4 \cdot 7H_2O$ (0.030 g, 0.1 mmol) were dissolved in distilled water (10 mL) with continuous stirring on a hot plate at 75°C for 30 min. The formation of a white precipitate of $BaSO_4$ was observed. The precipitate was filtered off, and the mother liquor was placed on the tube bottom. Distilled water (3 mL) and then MeCN (5 mL) were layered on the solution. A weighed sample of 4,4'-bipy (0.031 g, 0.2 mmol) was dissolved in MeCN (10 mL), and the resulting light yellow solution was carefully layered on the top. Colorless crystals suitable for XRD were formed in 3 weeks. The crystals of compound **II** were filtered off, washed with cold MeCN ($T = 5°C$), and dried in air. The yield was 0.019 g (49% based on $ZnSO_4 \cdot 7H_2O$).

IR (ATR; ν, cm^{-1}): 3270 m br, 2963 w, 2935 w, 2835 w, 2360 w, 1527 s, 1442 m, 1413 m, 1385 m, 1315 m, 1296 m, 1219 w, 1178 w, 1071 w, 1005 w, 987 w, 935 w, 811 s, 772 m, 733 m, 694 s, 656 s, 625 s, 570 s, 510 s.

XRD. The crystals of compounds **I** and **II** were isolated from the reaction mixtures. The XRD experiment was conducted on a Bruker Apex 2 CCD automated four-circle diffractometer with a two-coordinate detector. The unit cell parameters were refined over the whole data set. The structures were solved by the adjoint space approach implemented in the SHELXT program [42] and refined by the SHELXL-2018/3 full-matrix least squares [43] for F^2 over all data using the OLEX2 program [44]. One of the water molecules and ethanol molecule in compound **I** and one of the water molecules in compound **II** are disordered over two positions and refined with a halved population. Hydrogen atoms were placed in geometrically calculated positions and refined by the riding model with the isotropic thermal parameters $U_{iso} = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$, where $U_{eq}(X)$ are the equivalent isotropic thermal parameters of the atoms to which the hydrogen atom is attached.

The crystallographic data and experimental and structure refinement parameters are given in Table 1. The topology of the basis networks of the coordination polymers was determined using the ToposPro software [45], as described earlier [46].

The coordinates of atoms and temperature parameters were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 2323336 (**I**) and 2323337 (**II**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/structures).

RESULTS AND DISCUSSION

The 2D polymers $\{[Zn(H_2O)(4,4'\text{-bipy})(Et_2mal)] \cdot 0.5C_2H_5OH \cdot 1.5H_2O\}_n$ (**I**) or $\{[Zn_4(H_2O)_2(4,4'\text{-bipy})_3(Et_2mal)_4] \cdot 6H_2O\}_n$ (**II**) are formed by the reaction of zinc nitrate with potassium diethyl malonate or zinc diethyl malonate with 4,4'-bipy, respectively, under slow mixing conditions. The synthesis conditions for compounds **I** and **II** differ in the choice of the initial zinc(II) salt and solvent (ethanol or acetonitrile, respectively). We have previously described two zinc(II) diethyl malonates with 4,4'-bipy: $\{[Zn(4,4'\text{-bipy})_{1.5}(Et_2mal)] \cdot H_2O\}_n$ and $\{[Zn(H_2O)_4(4,4'\text{-bipy})](HEt_2mal)_2 \cdot 4,4'\text{-bipy} \cdot 2H_2O)\}_n$. In this case, no coordination of the acid anions to the zinc atoms was observed when using zinc(II) nitrate as the initial salt in the absence of base: they are present in the crystal as anions [39].

The IR spectra of the synthesized compounds exhibit bands of stretching asymmetric and symmetric vibrations of the carboxyl groups of the diethylmalonic acid anions at 1597, 1369 and 1527, 1385 cm^{-1} for compounds **I** and **II**, respectively. The broad bands in ranges of 3270 and 3182 cm^{-1} for complexes **I** and **II** are observed, since both compounds contain coordinated and solvate water molecules. The strong bands at 817 and 811 cm^{-1} for compounds **I** and **II** correspond to bending vibrations of the C–H groups of the heteroaromatic fragments in 4,4'-bipy.

The crystal and molecular structures of complexes **I** and **II** were determined by XRD. The independent parts of their unit cells are shown in Fig. 1. The structure of complex **I** contains zinc atoms of two types, one anion, 4,4'-bipy molecule coordinated by the Zn(2) atom, water molecule, two molecules of solvate water (one with the full and another with halved population), and halved populated ethanol molecule. The dicarboxylate anion is the chelate-bridging ligand coordinated through two oxygen atoms to the Zn(1)

Table 1. Crystallographic data and experimental and structure refinement parameters for compounds **I** and **II**

Parameter	Value	
	I	**II**
Empirical formula	$C_{18}H_{26}N_2O_7Zn$	$C_{58}H_{80}N_6O_{24}Zn_4$
Molecular weight	447.78	1506.76
T, K	120.0(2)	120.0(2)
Crystal system	Monoclinic	Triclinic
Space group	$C2/c$	$P\bar{1}$
Z	8	1
a, Å	18.3417(18)	7.7240(15)
b, Å	17.5199(17)	10.511(2)
c, Å	12.9134(12)	20.330(4)
α, deg	90	93.12(3)
β, deg	100.475(2)	92.53(3)
γ, deg	90	91.34(3)
V, Å^3	4080.5(7)	1646.0(6)
ρ_{calc}, g cm^{-3}	1.458	1.520
μ, mm^{-1}	1.245	1.522
F(000)	1872	782
Number of measured reflections	35146	29533
Number of independent reflections (R_{int})	8737 (0.062)	10060 (0.123)
Number of reflections with $I > 2\sigma(I)$	5045	5917
Number of refined parameters	253	418
R_1	0.068	0.087
wR_2	0.163	0.187
GOOF	1.06	1.07
Residual electron density (min/max), e Å^{-3}	1.87/−2.13	1.40/−1.60

Table 2. Coordination bond lengths (Å) in complexes **I** and **II**

Complex	Central atom	Bond		
		Zn–N(4,4'-bipy)	Zn–O(H_2O)	Zn–O(Et_2mal)
I	Zn(1)	2.224(2)		2.026(2)–2.030(2)
	Zn(2)	2.155(2)	2.151(2)	2.087(2)
II	Zn(1)	2.061(5)		1.954(4)–2.038(4)
	Zn(2)	2.132(5)–2.173(5)	2.060(4)	2.022(4)–2.357(4)

atom and through the third O atom to the Zn(2) atom. 4,4'-bipyridine and water in the complex act as the bridging and terminal ligands, respectively. As a result, the complex has the [Zn(H_2O)(4,4'-bipy)(Et_2mal)] composition (Fig. 2a). The zinc atoms form coordination polyhedra ZnO_4N_2 as distorted octahedra, whose axial bonds are formed by the nitrogen atoms of the bipyridine molecules, and the equatorial plane is formed by four oxygen atoms from two chelate Et_2mal anions (Zn(1)) or two water molecules and two oxygen

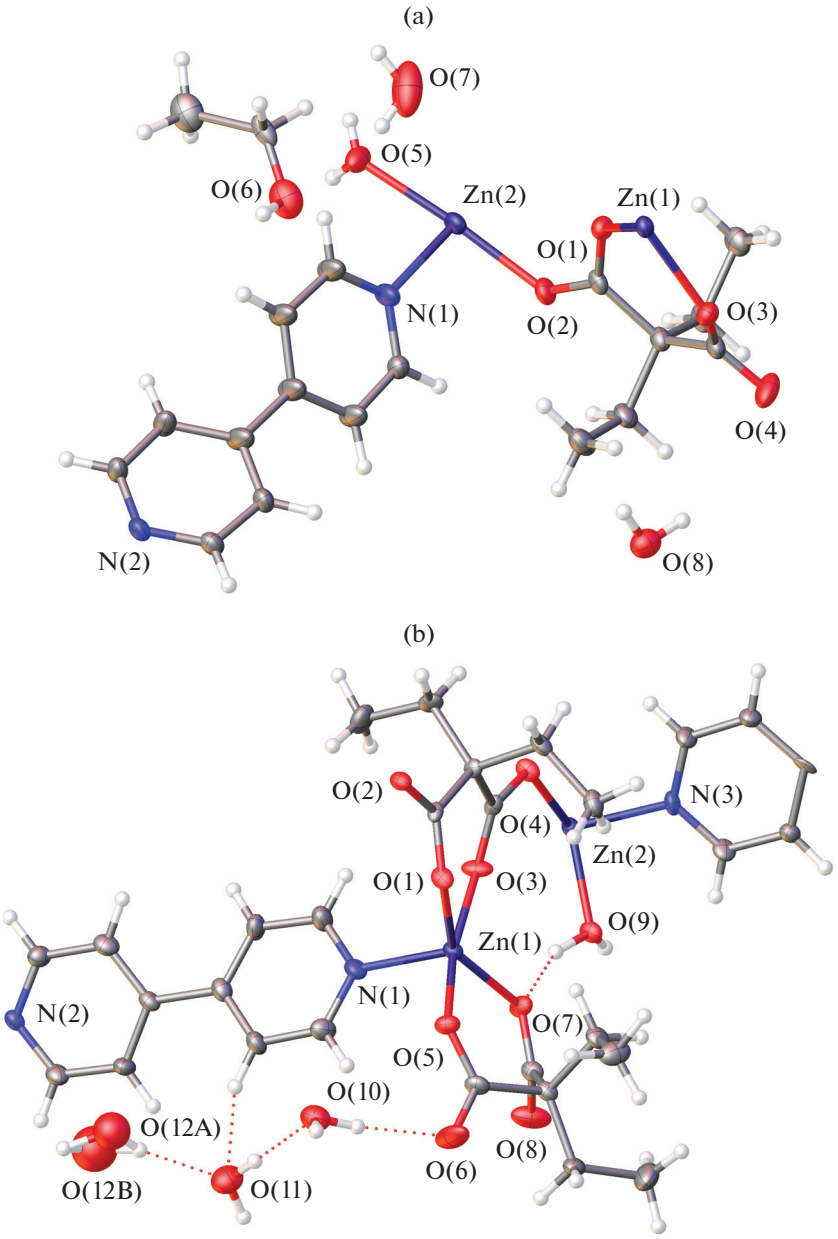

Fig. 1. Independent part of the unit cell in complexes (a) **I** and (b) **II** in the representation of atoms by thermal ellipsoids (shown with the probability $p = 50\%$).

atoms from the Et_2mal anions (Zn(2)). The Zn–N bonds are longer than the Zn–O bonds, and the shortest of the latter are with the chelate-coordinated Et_2mal anions (Table 2).

The independent part of the unit cell in the structure of complex **II** contains two zinc cations, two Et_2mal anions, 1.5 molecules of 4,4'-bipy, one coordinated water molecule, and three uncoordinated water molecules. One of the Et_2mal anions is chelate and another anion is bridging-chelate, which are used for coordination by two and four its O atoms, respectively. 4,4'-bipyridine and water in the complex are also bridging and terminal ligands, respectively. The composition of the complex ignoring the solvate water molecules is $[Zn_4(H_2O)_2(4,4'\text{-bipy})_3(Et_2mal)_4]$, and the structure is layered (Fig. 2c). The Zn(1) atom has the ZnO_4N coordination polyhedron as a distorted square pyramid with the N atom at the vertex and the metal atom shifting by 0.414(2) Å from the plane formed by the oxygen atoms. In spite of different in nature atoms forming this pentagon and a high variation of the bond lengths (Table 2), the total deviation

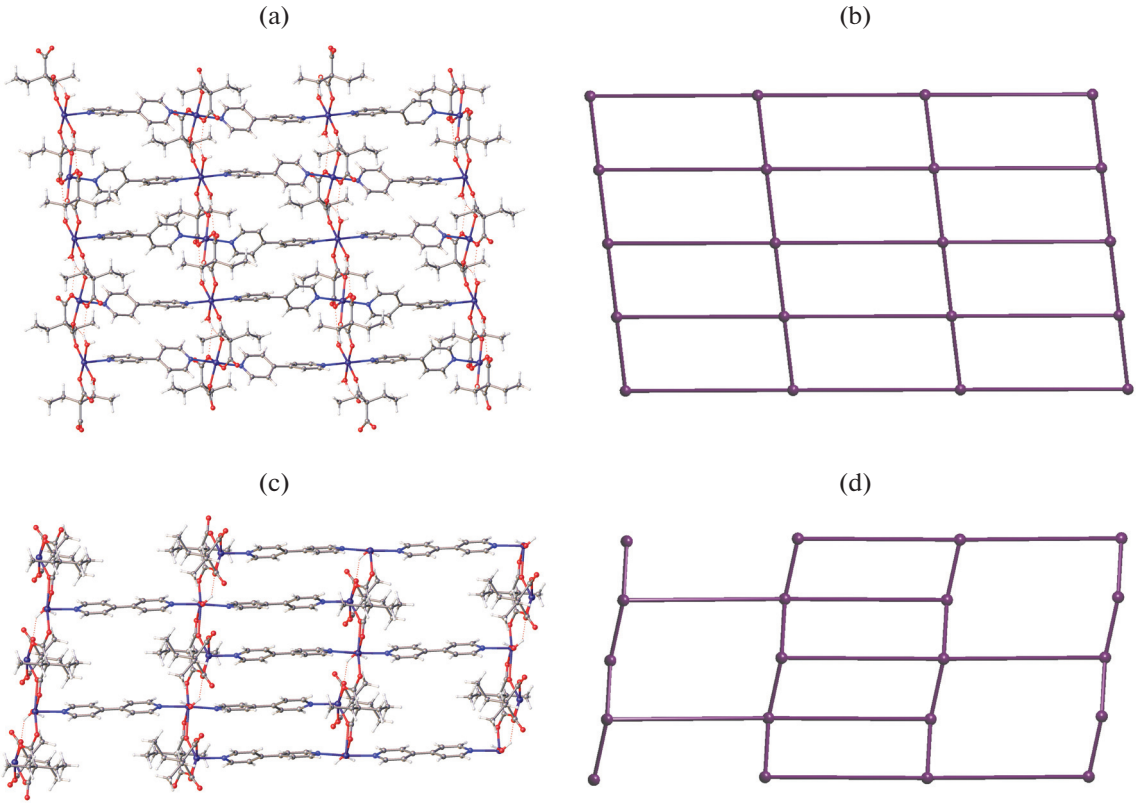

Fig. 2. Fragments of the $[Zn(H_2O)(4,4'\text{-bipy})(Et_2mal)]$ and $[Zn_4(H_2O)_2(4,4'\text{-bipy})_3(Et_2mal)_4]$ layers in complexes (a) **I** and (c) **II** and the lattices obtained by the simplification of these structures with the (b) **sql** and (d) **bey** topology, respectively.

of the atoms estimated by the described algorithm [47] is only 0.838 Å. The coordination polyhedron of the Zn(2) atom is the ZnO_4N_2 distorted octahedron containing in the equatorial plane one water molecule and two and one O atoms from two different Et_2mal anions. The corresponding coordination bonds for the pentacoordinate zinc atom are appreciably shorter than those for the hexacoordinate atom.

Both complexes, $[Zn(H_2O)(4,4'\text{-bipy})(Et_2mal)]$ and $[Zn_4(H_2O)_2(4,4'\text{-bipy})_3(Et_2mal)_4]$, are layered, which is not characteristic of the zinc(II) complexes with substituted malonic acids anions and 4,4'-bipyridine analogs [39]. Moreover, they correspond to the basis network topology **sql** and its derivative **bey** topology (Figs. 2b and 2d), respectively, in designations of the Reticular Chemistry Structure Resource (RCSR) database [48]. Among the zinc(II) complexes, only one coordination polymer with the planar square lattice **sql** topology, $[Zn(4,4'\text{-bipy})(Ph(CH_2)_3mal)(H_2O)]\cdot 4H_2O$, was found [49], and the **bey** topology was not observed. By contrast, among the copper(II) complexes, the coordination polymers of both types have been synthesized previously [50]. Infinite layers $[Zn(H_2O)(4,4'\text{-bipy})(Et_2mal)]$ are linked with each other by hydrogen bonds involving coordinated and uncoordinated solvent molecules, whose parameters are listed in Table 3. In the case of $[Zn_4(H_2O)_2(4,4'\text{-bipy})_3(Et_2mal)_4]$, the solvate water molecules maintain the layer structure due to the hydrogen bonds (Table 3), whereas interlayer nonvalent interactions are presented by predominantly hydrophobic H...H contacts.

Two new coordination polymers of zinc(II) with diethylmalonic acid anions and 4,4'-bipy as the bridging N-donor ligand were synthesized: $\{[Zn(H_2O)(4,4'\text{-bipy})(Et_2mal)]\cdot 0.5C_2H_5OH\cdot 1.5H_2O\}_n$ (**I**) and $\{[Zn_4(H_2O)_2(4,4'\text{-bipy})_3(Et_2mal)_4]\cdot 6H_2O\}_n$ (**II**). The formation of layered structures, which are not characteristic of the zinc(II) malonate polymers, with the **sql** and **bey** topologies for complexes **I** and **II**, respectively, was shown by the topological analysis data. Note that the **bey** topology, which has not been observed previously for the coordination zinc polymers, was successfully obtained in the present work replacing the H_2O : EtOH crystallization mixture of solvents by H_2O : MeCN and using zinc sulfate as the initial salt in the method of slow solvent mixing. These results emphasize importance of selecting synthesis conditions to control the structures of the coordination polymers and provide prospects for further studies in the field of design of new molecular materials with specified properties.

Table 3. Geometric parameters of hydrogen bonds in the structures of complexes **I** and **II***

Complex	*D*–H···*A*	Distance, Å			Angle *DHA*, deg
		D—H	H···*A*	*D*···*A*	
I	O(5)–H(5A)...O(1)^{i}	0.86	1.939	2.683(3)	143.4
	O(5)–(H5B)...O(7)^{i}	0.86	1.999	2.716(4)	167.6
	O(6)–(H6)...O(4)^{ii}	0.84	1.767	2.599(7)	170.3
	O(7)–(H7E)...O(6)	0.85	2.243	3.061(8)	161.4
	O(7)–(H7D)...O(8)^{ii}	0.85	1.973	2.617(6)	131.8
II	O(9)–(H9A)...O(7)	0.88	1.962	2.747(6)	148.1
	O(9)–(H9B)...O(5)^{iii}	0.88	1.815	2.694(5)	178.9
	O(10)–(H10A)...O(8)^{iv}	0.87	1.880	2.725(6)	163.2
	O(10)–(H10B)...O(6)	0.87	1.875	2.736(7)	170.3
	O(11)–(H11C)...O(2)^{v}	0.87	2.060	2.894(6)	160.2
	O(11)–(H11D)...O(10)	0.88	1.800	2.676(7)	173.3
	O(12A)–H12D)...O(11)	0.77	2.053	2.809(14)	166.8

* Symmetry procedures: ^{i} $1-x, y, 3/2-z$; ^{ii} $3/2-x, -1/2+y, 3/2-z$; ^{iii} $-1+x, y, z$; ^{iv} $1+x, y, z$; ^{v} $x, -1+y, z$.

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CONFLICT OF INTEREST

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

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