ISSN 0036-0236, Russian Journal of Inorganic Chemistry, 2020, Vol. 65, No. 9, pp. 1351–1359. © Pleiades Publishing, Ltd., 2020.

COORDINATION COMPOUNDS

Synthesis, Characterization, and Antibacterial Activity of Cd(II) Complexes with 3-/4-Fluorobenzoates and 3-Hydroxypiridine as Co-Ligands

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Received January 13, 2020; revised February 10, 2020; accepted March 3, 2020

Abstract—In this study, two new complexes of Cd(II) 4-fluorobenzoate (4-FB)/3-fluorobenzoate (3-FB) with 3-hydroxypyridine (3-HPY) have been synthesized and structural characterizations have been performed by using elemental analysis, FT-IR spectroscopy, and single-crystal X-ray diffraction methods. In both complexes, the metal atom is chelated by two carboxylate groups from two 4- or 3-fluorobenzoate anions and coordinated by two 3-hydroxypyridine (HPY) molecules. In both complexes, an oxygen atom of carboxylate from the adjacent anion bridges to the Cd atom, completing the distorted seven-coordination geometry. The only difference between complexes is the positions of fluorine atoms in fluorobenzoic acids (4-fluorobenzoic acid (complex **1**) and 3-fluorobenzoic acid (complex **2**)). Antibacterial resistance of two new complexes to some bacteria has been investigated.

Keywords: cadmium(II) complex, 4-fluorobenzoic acid, 3-fluorobenzoic acid, 3-hydroxypyridine, antibacterial, X-ray diffraction

DOI: 10.1134/S0036023620090168

INTRODUCTION

In recent years, the synthesis of new complexes with striking properties that can be used in various application areas has gained importance. These complexes are composed of metal atom/atoms at the center of symmetry and anions or molecules attached to the metal atom. These complexes stand out both with the variety of their structure and with their physical, chemical and biological properties. The structure and application-oriented properties of these compounds, which are part of material chemistry, are influenced by metal cations, ligands, and intra- and intermolecular interactions [1–7].

Carboxylic acids and ligands containing N-, O-, and S-donor atoms in their structure have many biological activities such as anti-inflammatory, antibacterial, antitumor, and antifungal [8–11]. Recent studies on antimicrobial activity of non-steroidal anti-inflammatory drugs have shown the increased antimicrobial activity of transition metals complexes with various carboxylic acid and nitrogen-containing ligands [12]. Therefore, it is very important to learn about the structure and binding relationships of complexes in the preparation of effective antimicrobial species [12]. Heterocyclic compounds are known to play an important role in many biological systems, as components of various vitamins and drugs [13–15].

Antibiotics are the basic life-saving drugs that revolutionized the pharmaceutical world with the discovery of penicillin in 1928. Recently, bacterial infections have caused deadly diseases and widespread outbreaks in humans, increasing at an alarming rate. As a result of the erroneous and widespread use of antibiotics, the antibiotic resistance, which is defined as the ability of microorganisms to resist the effects of drugs, emerges. It has also been noted that there are mortal cases due to infection from antibiotic-resistant bacteria. When this situation is evaluated, the choice of the most appropriate antibiotic against infection is of vital importance. The increase in mortality associated with infectious diseases is directly related to bacteria that are resistant to antibiotics. The development of new antimicrobial agents with new and stronger mechanisms of action is certainly an urgent medical need [16–23].

In this study, two new complexes of Cd(II) 4-/3-fluorobenzoates with 3-hydroxypyridine, $[Cd_2(4\text{-}FB)_4(3\text{-}HPY)_4]$ (**1**) and $[Cd_2(3\text{-}FB)_4(3\text{-}HPY)_4]$ (**2**), were firstly synthesized and their structures were characterized by elemental analysis, FT-IR spectroscopy and single crystal X-ray diffraction methods and anti-

Table 1. Crystal and structure refinement data of complexes **1** and **2**

Crystal data	Complex **1**	Complex **2**
Chemical formula	$C_{48}H_{36}Cd_2F_4N_4O_{12}$	$C_{48}H_{36}Cd_2F_4N_4O_{12}$
FW	1161.61	1161.61
Crystal system, space group	Triclinic, *P*	Triclinic, *P*
Temperature, K	296	296
a, *b*, *c*, Å	8.7635 (3), 11.8088 (4), 12.1645 (4)	10.2492 (2), 10.2769 (2), 12.0497 (3)
α, β, γ, deg	93.866 (2), 110.079 (2), 98.280 (2)	66.183 (2), 86.824 (3), 87.985 (3)
V, Å^3	1160.74 (7)	1159.21 (4)
Z	1	1
Radiation type	MoK_α	MoK_α
μ, mm^{-1}	1.00	1.00
Crystal size, mm	0.17 × 0.12 × 0.09	0.12 × 0.10 × 0.09
Data collection		
Diffractometer	Bruker APEX-II CCD	Bruker APEX-II CCD
Absorption correction	Multi-scan SADABS; Bruker, 2012	Multi-scan SADABS; Bruker, 2012
T_{min}, T_{max}	0.568, 0.745	0.879, 0.905
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17479, 4694, 4279	14282, 4582, 3795
R_{int}	0.034	0.053
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.626	0.627
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.060, 1.16	0.085, 0.184, 1.39
No. of reflections	4694	4582
No. of parameters	317	316
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 11.2792P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.41, −0.42	1.49, −0.83

Computer programs: APEX2 [43], SAINT [43], SAINT, SHELXS97 [44], SHELXL97 [44], ORTEP-3 for Windows [45], WinGX publication routines [45] and PLATON [46].

bacterial properties of the obtained complexes were investigated by agar well diffusion method against *Pseudomonas aeruginosa* (*P. aeruginosa*), *Klebsiella pneumonia* (*K. pneumonia*), *Escherichia coli* (*E. coli*), and *Staphylococcus aureus* (*S. aureus*) bacteria.

EXPERIMENTAL

Materials and methods. Chemicals used in the study; 4-fluorobenzoic acid, 3-fluorobenzoic acid (Fluka), 3-hydroxypyridine (Merck), sodium bicarbonate (Merck) and cadmium(II) sulfate 8.3 hydrate $CdSO_4 \cdot 8.3H_2O$ (Merck) were taken and used without any purification. Elemental Analysis was measured with LECO CHNS-932 elemental analyzer. FT-IR Spectra were recorded by a PerkinElmer Frontier™ FT-IR spectrometer using a Diamond ATR accessory in the range of 4000–600 cm^{-1} from solid samples. Crystal structures were determined by Bruker SMART BREEZE CCD diffractometer. The experimental details related to the crystal structures of complexes were given in Table 1.

Synthesis of the complexes. Bis(μ-4-fluorobenzoato)-κ^3*O,O'*:*O*;κ^3*O*:*O,O'*-bis[(4-fluorobenzoato-κ^2*O,O'*) bis(3-hydroxypyridine-κ*N*^{1})cadmium] (complex 1). To

obtain the sodium 4-fluorobenzoate, 0.84 g (0.01 mol) of 4-fluorobenzoic acid and 1.40 g (0.01 mol) of sodium bicarbonate were mixed in 100 mL distilled water and continuously heated and stirred at 60°C until CO_2 gas was removed. Then 3.84 g (0.005 mol) of $CdSO_4 \cdot 8.3H_2O$ dissolved in 50 mL of distilled water. In a separate beaker, 1.75 g (0.01 mol) of 3-hydroxypyridine solution was prepared and added on cadmium sulfate solution, and the sodium salt of 4-fluorobenzoic acid was added by filtration, then left to crystallize. After 5–6 days, cream colored crystals formed. For complex **1**, anal. calcd. (%): $[Cd_2(4\text{-}FB)_4(3\text{-}HPy)_4]$ (MW = 1161.61) C, 49.46; H, 3.46; N, 4.81. Found (%): C, 49.6; H, 3.12; N, 4.82. Selected IR bands (cm^{-1}): $\nu(OH)_{H_2O}$ 3290, $\nu(C{-}N)_{py}$, 1089, $\nu COO^-)_{as}$ 1540, $\nu(COO^-)_s$ 1392, $\nu(C{=}C)_{ar}$ 1600, $\nu(M{-}O)$ 641 cm^{-1}.

Bis(μ-3-fluorobenzoato)-κ^3*O*,*O*':*O*;κ^3*O*:*O*,*O*'-bis[(3-fluorobenzoato-κ^2*O*,*O*') bis(3-hydroxypyridine-κ*N*^{1})cadmium] (complex 2). The synthesis of complex **1** was repeated using 3-fluorobenzoic acid instead of 4-fluorobenzoic acid. After 5–6 days, cream colored crystals formed. For complex **2**, anal. calcd. (%): $[Cd_2(3\text{-}FB)_4(3\text{-}HPy)_4]$ (MW = 1161.61) C, 49.46; H, 3.46; N, 4.81. Found (%): C, 49.20; H, 3.12; N, 4.82; Selected IR bands (cm^{-1}): $\nu(OH)_{H_2O}$ 3149 $\nu(C{-}N)_{py.}$ 1050, $\nu COO^-)_{as}$ 1537, $\nu(COO^-)_s$ 1386, $\nu(C{=}C)_{ar}$ 1601, $\nu(M{-}O)$ 640 cm^{-1}.

Determination of antimicrobial activity of complexes. Antibacterial effects of complexes were investigated using agar well diffusion method. The bacteria used were Gram-negative *P. aeruginosa* (ATCC 27853), *K. pneumoniae* (ATCC 4352), and *E. coli* (ATCC 25922), Gram-positive *S. aureus* (ATCC) 6538). Microorganisms obtained from company of Microbiological Environmental Protection Laboratories were grown in research laboratories of Kafkas University Engineering and Architecture Faculty. Mueller Hinton Agar (MHA) was used as medium. First, the activation of bacteria from stocks in Mueller Hinton Broth (MHB) was performed for 24 hours during the 37°C incubation. Bacteria which were standardized with 0.5 McFarland standard were planted in sterile prepared petri dishes. 0.05 g of the synthesized complexes and standard antibiotics (Ampicillin X3261, Neomycin X3385, Steptomycin X3385) were dissolved in 5 mL of DMSO and homogeneous solutions were prepared and injected 50 μL from the stocks into wells drilled 4 mm in diameter using an automated pipette. Petri dishes were at for incubated at 37 ± 1°C for (18–24) ± 2 hours in order to determine inhibition zone diameters [24–28]. All inhibition zones were measured as mm.

RESULTS AND DISCUSSION

Elemental analysis, FT-IR spectroscopy, and X-ray structure analysis results are in harmony for the two new complexes synthesized.

FT-IR spectroscopy. Some important stretching bands of FT-IR spectra of synthesized complexes are as follows. The peaks of the O–H group of the complexes were recorded in the range of 3600–3300 cm^{-1}. The O–H stretching vibrations of the complexes were observed at 3290 cm^{-1} (**1**), 3149 cm^{-1} (**2**) [29]. The carbonyl group COO^- asymmetric and symmetric vibrations were observed at 1540–1392 cm^{-1} (**1**), 1537–1386 cm^{-1} (**2**) The vibrations related to the Me-O bond were recorded at 641 cm^{-1} (**1**) and 640 cm^{-1} (**2**) [29, 30]. The C–N absorption bands of pyridine ring were showed at 1089 cm^{-1} (**1**) and 1050 cm^{-1} (**2**) [31] (Figs. S1 and S2).

Descriptions of the crystal structures. The metal atoms are chelated by four oxygen atoms of two carboxylate groups from two 4-fluorobenzoate anions (for complex **1**) and two 3-fluorobenzoate anions (for complex **2**), and coordinated by two 3-hydroxypyridine molecule; In both complexes, an O atom of carboxylate from the neighboring anion bridges to the Cd atom, creating the uneven seven-coordination environment (Figs. 1 and 2). The Cd(1)–Cd(1)^i (Symmetry code: (i) $-x$, $-y$, $-z$) distances are 3.812(8) Å (**1**) and 3.838(8) Å (**2**). Bond length of the average Cd–O (Table 2) is 2.4064(19) Å (complex **1**) and 2.431(6) Å (**2**). The Cd atom is displaced out of the least-squares planes of the carboxylate groups (O(1)/C(1)/O(2)) and (O(3)/C(8)/O(4)) by 0.0124(2) and 0.0627(2) Å (**1**) and 0.0433(7) and −0.0327(7) Å (**2**), respectively. The OCdO angles are 52.78(6)° and 53.81(6)° (for **1**) and 53.5(2)° and 52.0(2)° (for **2**), respectively. In the previous researches, the OMO (where M is transition metal) angles were found 55.71(5)° and 117.52(4)° in $[Cd_2(4\text{-methylaminobenzoate})_4(nicotinamide)_2(H_2O)_2]$ [32], 55.96(4)° and 53.78(4)° in $[Cd_2(4\text{-dimethylaminobenzoate})_4(nicotinamide)_2(H_2O)_2]$ [33], 52.91(4)° and 53.96(4)° in $[Cd(4\text{-formylbenzoate})_2(isonicotinamide)_2(H_2O)] \cdot H_2O$ [34], 60.70(4)° in $[Co(4\text{-dimethylaminobenzoate})_2(isonicotinamide)(H_2O)_2]$ [35], 58.45(9)° in $[Mn(4\text{-dimethylaminobenzoate})_2(isonicotinamide)(H_2O)_2]$ [36], 60.03(6)° in $[Zn(4\text{-methylaminobenzoate})_2(isonicotinamide)_2] \cdot H_2O$ [37], 58.3(3)° in $[Zn_2(N,N\text{-diethylnicotinamide})_2(4\text{-hydroxybenzoate})_4] \cdot 2H_2O$ [38] and 55.2(1)° in $[Cu(acetylsalicylate)_2(pyridine)_2]$ [39]. The dihedral angles between the planar carboxylate groups (O(1)/O(2)/C(1)), (O(3)/O(4)/C(8)) and the neighboring benzene rings A (C(2)–C(7)), B (C(9)–C(14)) are 1.5(2)° and 3.8(2)° (**1**) and 3.5(3)° and 1.5(8)° (**2**), respectively, while that between rings A and B is A/B = 11.8(1)° (**1**) and 3.7(4)° (**2**). The pyridine rings of 3-HPY C (N(1)/C(15)–C(19)) and D (N(2)/C(20)—C(24))

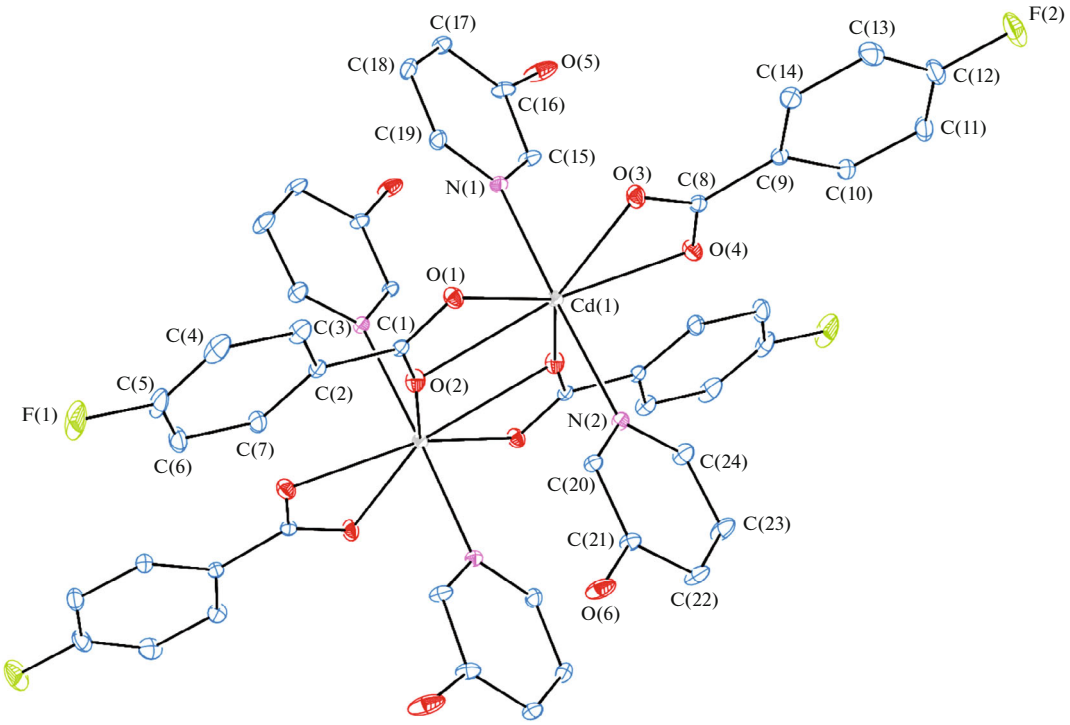

Fig. 1. The molecular structure of the dinuclear complex **1** with the atom numbering scheme. Thermal ellipsoids are created at the 30% probability level. Hydrogen atoms have been removed for clarity.

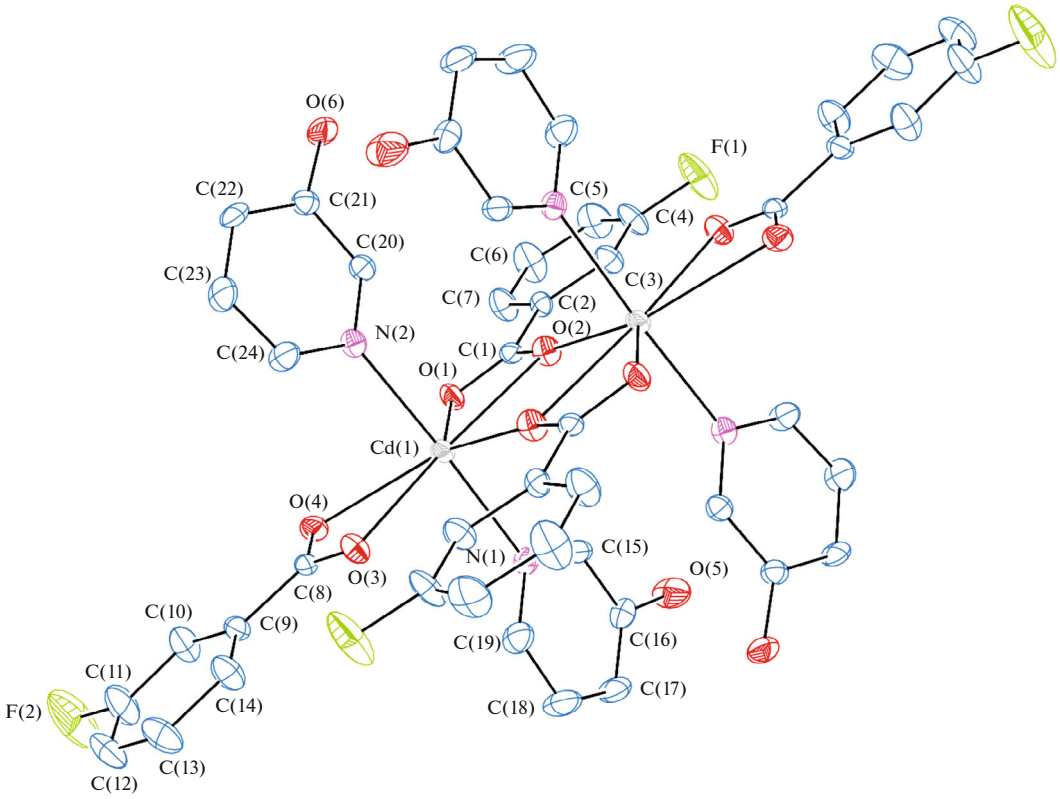

Fig. 2. The molecular structure of the dinuclear complex **2** with the atom numbering scheme. Thermal ellipsoids are created at the 30% probability level. Hydrogen atoms have been removed for clarity.

Table 2. Selected bond lengths (Å) and angles (°) for complexes

Complex **1**		Complex **2**	
Bond lengths			
Cd(1)–O(1)	2.3027(18)	Cd(1)–N(2)	2.313(7)
Cd(1)–O(2)^{i}	2.3254(18)	Cd(1)–N(1)	2.316(7)
Cd(1)–N(2)	2.327(2)	Cd(1)–O(3)	2.323(6)
Cd(1)–N(1)	2.328(2)	Cd(1)–O(1)	2.350(6)
Cd(1)–O(4)	2.3625(19)	Cd(1)–O(2)^{i}	2.362(6)
Cd(1)–O(3)	2.461(2)	Cd(1)–O(2)	2.483(6)
Cd(1)–O(2)	2.5802(19)	Cd(1)–O(4)	2.637(6)
O(1)–C(1)	1.251(3)	O(1)–C(1)	1.241(10)
O(2)–C(1)	1.257(3)	O(2)–C(1)	1.288(10)
O(3)–C(8)	1.258(3)	O(3)–C8	1.241(10)
O(4)–C(8)	1.254(3)	O(4)–C8	1.276(11)
Bond angles			
O(1)Cd(1)O(2)^{i}	130.85(7)	N(2)Cd(1)N(1)	177.0(3)
O(1)Cd(1)N(2)	92.49(7)	N(2)Cd(1)O(3)	89.5(2)
O(2)^{i}Cd(1)N(2)	87.04(7)	N(1)Cd(1)O(3)	87.5(2)
O(1)Cd(1)N(1)	93.36(7)	N(2)Cd(1)O(1)	90.9(2)
O(2)^{i}Cd(1)N(1)	87.42(7)	N(1)Cd(1)O(1)	91.4(2)
N(2)Cd(1)N(1)	173.73(7)	O(3)Cd(1)O(1)	136.2(2)
O(1)Cd(1)O(4)	138.45(7)	N(2)Cd(1)O(2)^{i}	90.5(2)
O(2)^{i}Cd(1)O(4)	90.55(7)	N(1)Cd(1)O(2)^{i}	89.5(2)
N(2)Cd(1)O(4)	85.36(7)	O(3)Cd(1)O(2)^{i}	95.0(2)
N(1)Cd(1)O(4)	91.76(7)	N(2)Cd(1)O(2)	91.3(2)
O(1)Cd(1)O(3)	85.32(6)	N(1)Cd(1)O(2)	91.6(2)
O(2)^{i}Cd(1)O(3)	143.67(6)	O(1)Cd(1)O(2)	53.5(2)
N(2)Cd(1)O(3)	95.74(7)	N(2)Cd(1)O(4)	88.2(2)
N(1)Cd(1)O(3)	87.01(7)	N(1)Cd(1)O(4)	90.1(2)
O(4)Cd(1)O(3)	53.81(6)	O(3)Cd(1)O(4)	52.0(2)
O(1)Cd(1)O(2)	52.78(6)	O(1)C(1)O(2)	119.0(7)
N(2)Cd(1)O(2)	87.36(7)	O(3)C(8)O(4)	121.0(8)
N(1)Cd(1)O(2)	94.37(7)		
O(4)Cd(1)O(2)	166.84(6)		
O(3)Cd(1)O(2)	138.10(6)		

Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.

Table 3. Hydrogen-bond geometry (Å, deg)

D–H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
		Complex **1**		
O(5)–H(5)···O(6)^{ii}	0.82	1.92	2.727(3)	168
O(6)–H(6)···O(3)^{iii}	0.82	1.78	2.577(3)	163
C(7)–H(7)···O(4)^{i}	0.93	2.39	3.314(5)	172
C(3)–H(3)···Cg(4)^{iv}	0.93	2.96	3.888(3)	172
		Complex **2**		
O(5)–H(5)*A*···O(4)^{ii}	0.82	2.00	2.715(10)	146
O(6)–H(6)*A*···O(4)^{iii}	0.82	2.00	2.720(11)	147
C(3)–H(3)···O(3)^{i}	0.93	2.34	3.265(12)	171
C(6)–H(6)···F(2)^{iv}	0.93	2.54	3.224(18)	131

Symmetry codes (**1**): (i) $-x+1, -y+1, -z+1$; (ii) $x+1, y+1, z$; (iii) $-x, -y+1, -z+1$; (iv) $-x+2, -y+1, -z+1$. Cg4 is the centroid or ring D (N2/C20—C24).
Symmetry codes (**2**): (i) $-x+1, -y+1, -z+1$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, -y, -z+1$; (iv) $-x+2, -y, -z+1$.

are positioned at a dihedral angle of C/D = 3.2(1)° (Complex **1**) and 13.0(3)° (**2**), while they are oriented with respect to the benzene rings at dihedral angles of A/C = 83.6(1)°, A/D = 83.8(1)°, B/C = 85.8(1)° and B/D = 85.4(1)° (**1**) and A/C = 88.9(3)°, A/D = 80.1(2)°, B/C = 87.1(3)° and B/D = 81.5(3)° (**2**). In the crystal structure, the bifurcated O–H···O hydrogen bonds (Table 3) connect the molecules, via the

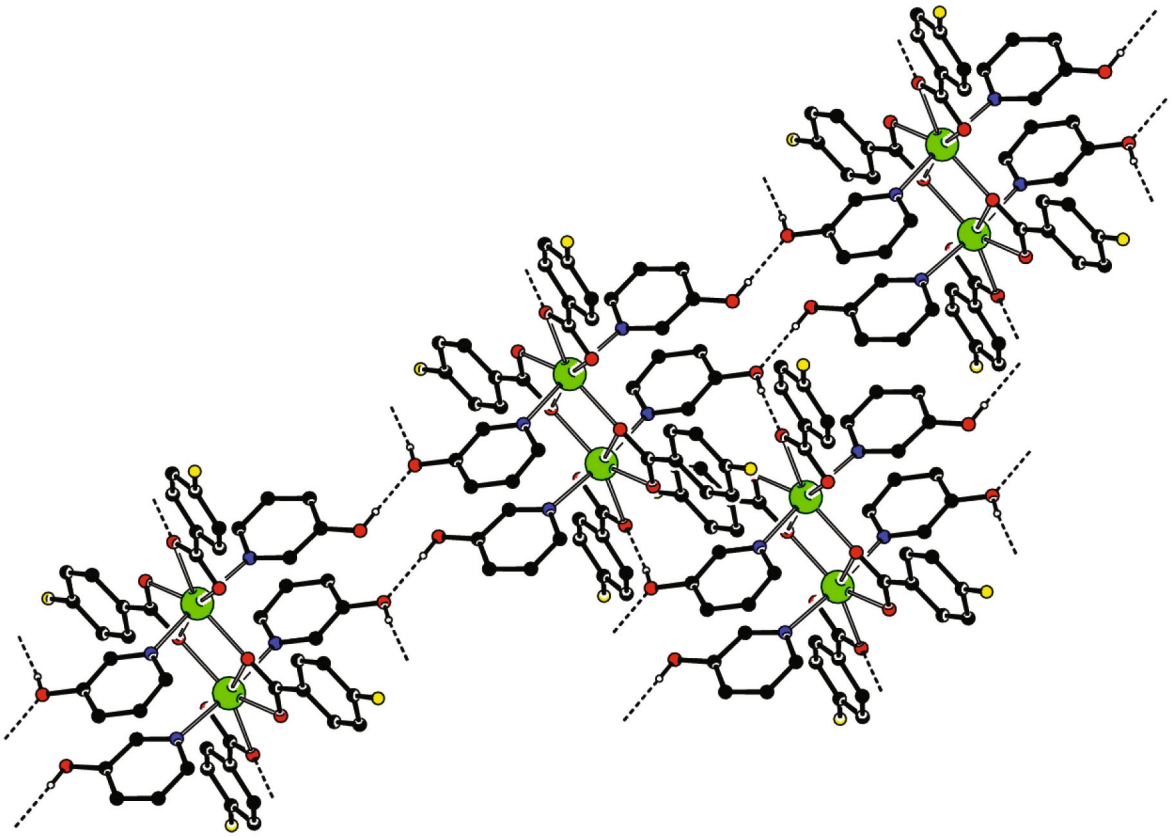

Fig. 3. A partial packing diagram of complex **1**. Only the O–H···O hydrogen bonds are shown as dashed lines.

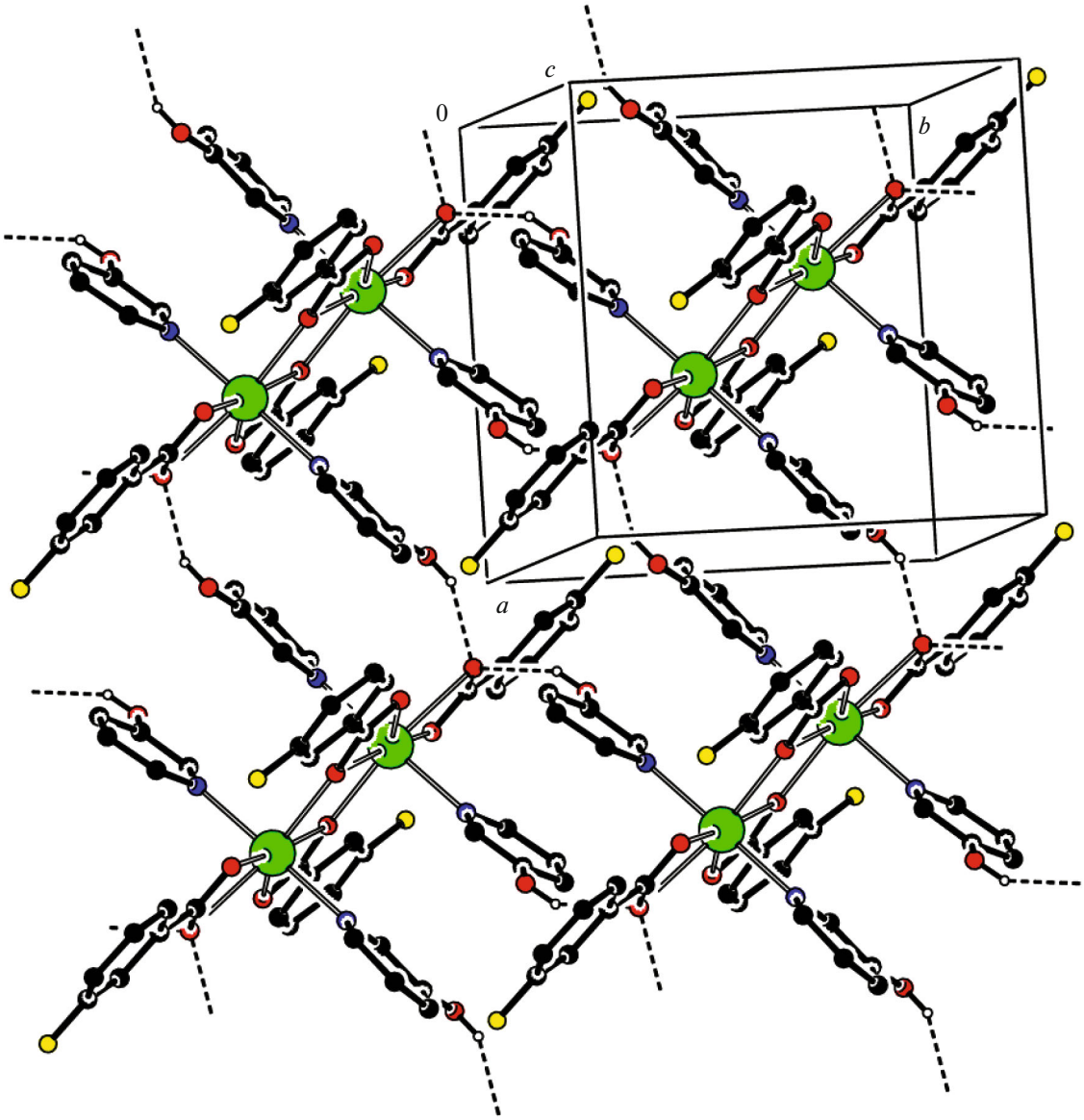

Fig. 4. A partial packing diagram viewed down the *c*-axis of complex **2**. Only the bifurcated O–H···O hydrogen bonds are shown as dashed lines. Remaining hydrogen atoms have been removed for clarity.

R22(14) and R22(24) (**1**) and R22(14) and R22(28) (**2**) ring motifs [40], into a network (Figs. 3 and 4), they are then connected by the C–H—O hydrogen bonds (Table 3) into a 3D structure, in which they could be instrumental in the stabilization of the structure. For complex **1**, a weak C–H-π interaction (Table 3) was also observed. For complex **2**, the π–π contacts between the pyridine rings, Cg3–Cg3i and Cg4–

Table 4. Antibacterial zone diameters of complexes (mm)

Complex	*P. aeruginosa*	*K. pneumoniae*	*E. coli*	*S. aureus*
Complex **1**	30	26	28	31
Complex **2**	30	30	26	30
Ampicillin X3261	36	35	34	37
Neomycin X3385	17	16	16	13
Steptomycin X3385	12	11	10	21

Cg4ii, and benzene rings, Cg2–Cg2iii [symmetry codes: (i) $2-x$, $1-y$, $1-z$, (ii) $1-x$, $-y$, $1-z$, (iii) $2-x$, $-y$, $2-z$, where Cg2, Cg3 and Cg4 are the centroids of the rings B (C(9)–C(14)), C (N(1)/C(15)–C(19)) and D (N(2)/C(20)–C(24)), respectively]. It was suggested that the structure could be stabilized, along with centroid–centroid distances of 3.904(6), 3.725(6), and 3.742(7) Å, respectively.

Antimicrobial activity of complexes. The antimicrobial effects of complex **1** and complex **2** were examined and zone diameters are given in Table 4. When we look at the zone diameters, both complexes are effective [41, 42] against *P. aeruginosa*, *K. pneumoniae*, *E. coli* and *S. aureus* bacteria and Ampicillin X3261, Neomycin X3385, Steptomycin X3385 as standart.

CONCLUSIONS

In this study, the structure of two novel synthesized complexes were determined by single crystal X-ray diffraction method and supported by elemental analysis and FT-IR spectroscopy. The only difference between two complexes with the same metal center is the position of fluorine in the fluorobenzoic acid used as the ligand. The antimicrobial activity of the complexes were assessed against *P. aeruginosa* (ATCC 27853), *K. pneumoniae* (ATCC 4352), and *E. coli* (ATCC 25922), Gram-positive *S. aureus* (ATCC) 6538. Complexes **1** and **2** have dinuclear and exhibit a similar coordination. Antibiotics such as Ampicillin X3261, Neomycin X3385, Steptomycin X3385 were used as standard. It was observed that the synthesized complexes had higher activity than neomycin and streptomycin and were close to ampicillin. Complexes **1** and **2** show excellent antibacterial properties against *P. aeruginosa*, *K. pneumoniae*, and *E. coli* and *S. aureus*. The increased bacterial resistance to antibiotics forces the scientific world to constantly produce new generation antibiotics. It can be evaluated in advanced clinical trials due both complexes are effective against *P. aeruginosa*, *K. pneumoniae*, *E. coli*, and *S. aureus* bacteria.

ACKNOWLEDGMENTS

We would like to thank Tuncer HÖKELEK, Hacali NECEFOĞLU and F.Elif ÖZTÜRKKAN for the X-ray analysis and helpful discussions. This article was obtained from the master thesis of Murat Durman.

CONFLICT OF INTEREST

The authors declare no conflict of interests.

SUPPLEMENTARY MATERIALS

Crystallographic data for complexes **1** and **2** reported in this article have been deposited with the Cambridge Crystallographic Data Center as Supplementary Publication CCDC nos. 1952575 (**1**) and 1952576 (**2**). Copies of these data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (+44) 1223 336033, or online via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk.

Supporting information includes FT-IR (Figs. S1, S2), and cif data (Figs. S3, S4) for compounds **1** and **2** synthesized in this work.

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