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Scientific paper

Synthesis, Characterization and X-Ray Crystal Structures of Schiff Base Zinc(II) Complexes with Antibacterial Activity

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Abstract

Two mononuclear zinc(II) complexes [$ZnCl_2$(HL)] (**1**) and [$ZnBr_2$(HL)] (**2**), and a polymeric zinc(II) complex [Zn(dca)L] (**3**), where HL is the zwitterionic form of the Schiff base 2,4-difluoro-6-(((2-pyrrolidin-1-yl)ethylimino)methyl) phenol (HL), L is the monoanionic form of HL, and dca is dicyanoamide, were prepared and characterized by elemental analysis and infrared spectroscopy, as well as X-ray single crystal determination. Complexes **1** and **2** are isostructural, in which the Zn atoms are in tetrahedral coordination. The Schiff base ligands in both complexes are coordinate to the Zn atoms through phenolate oxygen and imino nitrogen. The zinc atoms in complex **3** are in tetrahedral coordination and are bridged by dicyanoamide ligands. The Schiff base ligand in this complex is coordinates to the zinc atom through phenolate oxygen, imino nitrogen and pyrrolidine nitrogen. Molecules of the three complexes are stabilized by hydrogen bonds. The biological assay indicates that the complexes have good antimicrobial activities on the bacteria strains *Escherichia coli*, *Pseudomonas aeruginosa*, *Salmonella typhi* and *Staphylococcus aureus*.

Keywords: Schiff base; Zinc complex; X-Ray crystal structure; Antibacterial activity.

1. Introduction

In recent years, zinc complexes have received much attention for their interesting biological activities such as antibacterial, antifungi and antitumor.[1] Schiff base compounds derived from the condensation reaction of aldehydes with various amines are much attractive for their extensive biological applications and coordination capability to metal atoms.[2] In literature, a number of zinc complexes with Schiff base ligands have been reported to show effective antimicrobial activities.[3] Halide and pseudohalide anions are versatile ligands in coordination chemistry. They play either terminal or bridging mode in the formation of metal complexes.[4] Fluoro- and chloro-substituted compounds are reported to have effective antibacterial activities.[5] Recently, our research group has reported some Schiff base complexes with biological activities.[6] In continuation of the work, the present paper reports three new zinc(II) complexes, [$ZnCl_2$(HL)] (**1**), [$ZnBr_2$(HL)] (**2**) and [Zn(dca)L] (**3**), where HL is the zwitterionic form of the Schiff base 2,4-difluoro-6-[(2-pyrrolidin-1-ylethylimino) methyl]phenol (HL), L is the monoanionic form of HL, and dca is dicyanoamide.

2. Experimental

2. 1. Materials and Measurements

Commercially available 3,5-difluorosalicylaldehyde and *N*-(2-aminoethyl)pyrrolidine were purchased from Sigma-Aldrich and used without further purification. Other solvents and reagents were made in China and used as received. The Schiff base HL was prepared according to the literature method.[7] C, H and N elemental analyses were performed with a Perkin-Elmer elemental analyzer. Infrared spectra were recorded on a Nicolet AVATAR 360 spectrometer as KBr pellets in the 4000–400 cm^{-1} region.

2. 2. Synthesis of [$ZnCl_2$(HL)] (1)

A methanolic solution (10 mL) of zinc chloride (0.10 mmol, 13.6 mg) was added to a methanolic solution (10 mL) of HL (0.10 mmol, 25.4 mg) with stirring. The mixture was stirred for 30 min at room temperature to give a colorless solution. The resulting solution was allowed to stand in air for a few days. Colorless block-shaped crystals suitable for X-ray single crystal diffraction were formed at the bottom of the vessel. The isolated products were

washed three times with cold ethanol and dried in air. Yield: 27 mg (69%). Analysis: Found: C 39.83%, H 4.20%, N 7.26%. Calculated for $C_{13}H_{16}Cl_2F_2N_2OZn$: C 39.98%, H 4.13%, N 7.17%. IR data (KBr, cm^{-1}): *ν* 3139 w (NH), 1623 s (C=N).

2. 3. Synthesis of [$ZnBr_2$(HL)] (2)

This complex crystallized as colorless block like single crystals was prepared by the same method as described for complex **1**, with zinc chloride replaced by zinc bromide (0.10 mmol, 22.5 mg). Yield: 35 mg (73%). Analysis: Found: C 32.37%, H 3.43%, N 5.92%. Calculated for $C_{13}H_{16}Br_2F_2N_2OZn$: C 32.56%, H 3.36%, N 5.84%. IR data (KBr, cm^{-1}): *ν* 3127 w (NH), 1623 s (C=N).

2. 4. Synthesis of [Zn(dca)L] (3)

A methanolic solution (10 mL) of zinc nitrate hexahydrate (0.10 mmol, 29.7 mg) was added to a methanolic solution (10 mL) of HL (0.10 mmol, 25.4 mg) with stirring. Then, sodium dicyanoamide (0.20 mmol, 17.8 mg) was added to the mixture. The final mixture was stirred for 30 min at room temperature to give a colorless solution. The resulting solution was allowed to stand in air for a few days. Colorless block-shaped crystals suitable for X-ray single crystal diffraction were formed at the bottom of the vessel. The isolated products were washed three times with cold ethanol and dried in air. Yield: 30 mg (79%). Analysis: Found: C 46.95%, H 3.86%, N 18.07%. Calculated for $C_{15}H_{15}F_2N_5OZn$: C 46.83%, H 3.93%, N 18.20%. IR data (KBr, cm^{-1}): *ν* 2322 w, 2257 w, 2193 s (dca), 1617 sc(C=N).

2. 5. X-ray Crystallography

Diffraction intensities for the complexes were collected at 298(2) K using a Bruker D8 VENTURE PHOTON diffractometer with Mo*Ka* radiation (l = 0.71073 Å). The collected data were reduced using the SAINT program,[8] and multi-scan absorption corrections were performed using the SADABS program.[9] The structures were solved by direct method and refined against F^2 by full-matrix least-squares method using the SHELXTL.[10] All of the non-hydrogen atoms were refined anisotropically. The H atoms attached to the N atoms of pyrrolidine groups in complexes **1** and **2** were located from difference Fourier maps and refined isotropically, with N–H distances restrained to 0.90(1) Å. All other hydrogen atoms were placed in idealized positions and constrained to ride on their parent atoms. The crystallographic data for the complexes are summarized in Table 1. Selected bond lengths and angles are given in Table 2. Hydrogen bonding information is listed in Table 3.

3. Results and Discussion

The three complexes were facile prepared by reaction of the Schiff base HL and zinc salts in methanol in the presence of halide or pseudohalide anions (Scheme 1).

Table 1. Crystallographic data and refinement parameters for the complexes

	1	**2**	**3**
Chemical formula	$C_{13}H_{16}Cl_2F_2N_2OZn$	$C_{13}H_{16}Br_2F_2N_2OZn$	$C_{15}H_{15}F_2N_5OZn$
Mr	390.55	479.47	384.69
Crystal color, habit	Colorless, block	Colorless, block	Colorless, block
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$	$P2_1/n$
a (Å)	14.2038(16)	14.3819(11)	7.2966(4)
b (Å)	15.0934(12)	15.2563(11)	29.393(2)
c (Å)	14.6834(16)	14.8598(11)	7.5410(5)
α (°)	90	90	90
β (°)	101.804(1)	100.321(2)	94.478(1)
γ (°)	90	90	90
V (Å^3)	3081.3(5)	3207.7(4)	1612.36(18)
Z	8	8	4
D_{calc} (g cm^{-3})	1.684	1.986	1.585
μ (mm^{-1})	1.960	6.533	1.557
F(000)	1584	1872	784
Number of unique data	5734	5959	2995
Number of observed data [$I > 2\sigma(I)$]	4566	3747	2383
Number of parameters	385	385	217
Number of restraints	2	2	0
R_1, wR_2 [$I > 2\sigma(I)$]	0.0355, 0.0889	0.0461, 0.1277	0.0499, 0.1040
R_1, wR_2 (all data)	0.0487, 0.0966	0.0839, 0.1466	0.0641, 0.1122
Goodness of fit on F^2	1.029	0.956	1.061

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

		1	
Zn1–O1	1.931(2)	Zn1–N1	2.035(2)
Zn1–Cl1	2.2235(10)	Zn1–Cl2	2.2379(9)
O1–Zn1–N1	94.72(9)	O1–Zn1–Cl1	108.78(7)
N1–Zn1–Cl1	116.69(7)	O1–Zn1–Cl2	115.00(7)
N1–Zn1–Cl2	109.58(7)	Cl1–Zn1–Cl2	110.49(4)
		2	
Zn1–O1	1.926(5)	Zn1–N1	2.046(5)
Zn1–Br1	2.3788(11)	Zn1–Br2	2.3567(11)
O1–Zn1–N1	95.1(2)	O1–Zn1–Br2	108.86(14)
N1–Zn1–Br2	116.92(15)	O1–Zn1–Br1	116.51(15)
N1–Zn1–Br1	108.59(15)	Br2–Zn1–Br1	110.36(4)
		3	
Zn1–O1	1.986(3)	Zn1–N1	2.019(3)
Zn1–N3	2.008(3)	Zn1–N5A	2.039(3)
Zn1–N2	2.422(3)		
O1–Zn1–N3	96.27(12)	O1–Zn1–N1	91.42(11)
N3–Zn1–N1	128.72(11)	O1–Zn1–N5A	94.10(13)
N3–Zn1–N5A	106.06(12)	N1–Zn1–N5A	123.88(12)
O1–Zn1–N2	169.17(11)	N3–Zn1–N2	91.87(12)
N1–Zn1–N2	77.90(11)	N5A–Zn1–N2	90.48(12)

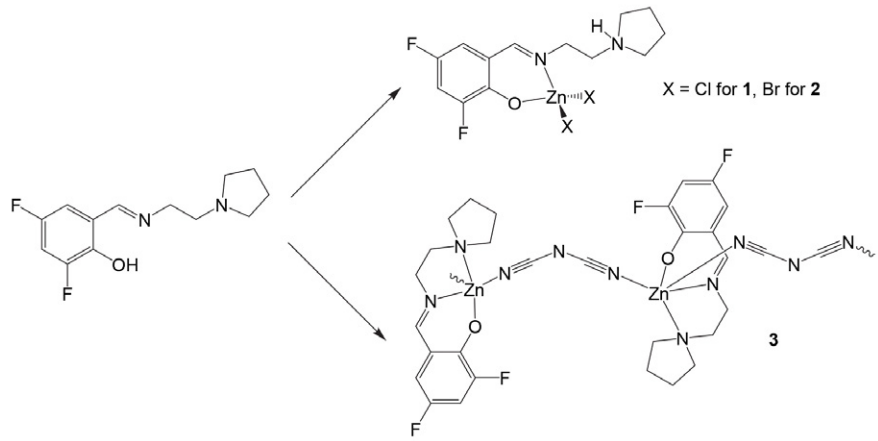
Symmetry code for A: *x*, *y*, 1 + *z*.

Table 3. Hydrogen bond distances (Å) and bond angles (°) for the complexes

D*–H···*A	***d*(*D*–H), Å**	***d*(H···*A*), Å**	***d*(*D*···*A*), Å**	**Angle (*D*–H···*A*), °**
		1		
N2–H2···F3$^{\#1}$	0.90(1)	2.40(3)	3.032(3)	128(3)
N2–H2···O2$^{\#1}$	0.90(1)	2.32(2)	3.129(3)	149(3)
N4–H4···Cl4	0.90(1)	2.55(2)	3.257(2)	136(3)
C10–H10A···F3$^{\#1}$	0.97	2.47	2.992(3)	113
C10–H10B···O1$^{\#2}$	0.97	2.36	3.315(4)	169
C13–H13A···Cl3$^{\#1}$	0.97	2.80	3.760(3)	171
C25–H25B···F2	0.97	2.44	3.048(4)	122
		2		
N2–H2···F4	0.90(1)	2.30(5)	3.020(6)	137(6)
N2–H2···O2	0.90(1)	2.40(4)	3.193(7)	147(6)
N4–H4···Br4	0.90(1)	2.63(5)	3.385(5)	142(6)
C10–H10B···O1$^{\#3}$	0.97	2.37	3.333(9)	174
C24–H24A···F2$^{\#4}$	0.97	2.45	3.144(9)	128
		3		
C10–H10B···N5$^{\#5}$	0.97	2.61	3.220(6)	122
C7–H7···F1$^{\#6}$	0.93	2.34	3.208(4)	155

Symmetry codes: #1: *x*, ½ – *y*, –½ + *z*; #2: –*x*, ½ + *y*, ½ – *z*; #3: 2 – *x*, ½ + *y*, 1½ – *z*; #4: *x*, ½ – *y*, ½ + *z*; #5: 1 – *x*, 1 – *y*, 1 – *z*; #6: *x*, *y*, 1 + *z*.

Zinc chloride and zinc bromide were used in the synthesis of complexes **1** and **2**, respectively. The Cl and Br anions act as terminal ligands in both complexes. Complex **3** was synthesized from zinc nitrate and sodium dicyanoamide. As a result, the dicyanoamide anion participates in coordination and is a bridging ligand. The Schiff base ligands coordinate to the metal atoms in zwitterionic form for complexes **1** and **2**. While for complex **3**, it adopts deprotonated monoanionic form. All the complexes are soluble in DMF, DMSO, methanol, ethanol and acetoni-



Scheme 1. The synthetic procedures for the complexes

trile, insoluble in water, chloroform and dichloromethane. Molar conductance of the complexes at concentration of 10^{-3} mol L^{-1} is 23–45 Ω^{-1} cm^2 mol^{-1}, indicating they are non-electrolytes.[11]

3. 1. Crystal Structure Description of Complexes 1 and 2

The molecular structures and atom numbering schemes of complexes **1** and **2** are shown in Figures 1 and 2, respectively. The asymmetric units of **1** and **2** possess two crystallographically independent molecules. Complexes **1** and **2** are isostructural mononuclear zinc(II) compounds. The Zn atom in each complex is in tetrahedral coordination and is coordinated by one phenolate oxygen and one imino nitrogen of a zwitterionic Schiff base ligand, and two halide anions (Cl for **1**, Br for **2**). The coordinate bond angles are in the ranges 94.72(9)–116.69(7)° for **1** and 95.1(2)–116.51(15)° for **2**, which are similar to those observed in similar Schiff base zinc complexes.[12] The Zn–O, Zn–N, Zn–Cl and Zn–Br bond lengths are comparable to those observed in the zinc complexes.[12]

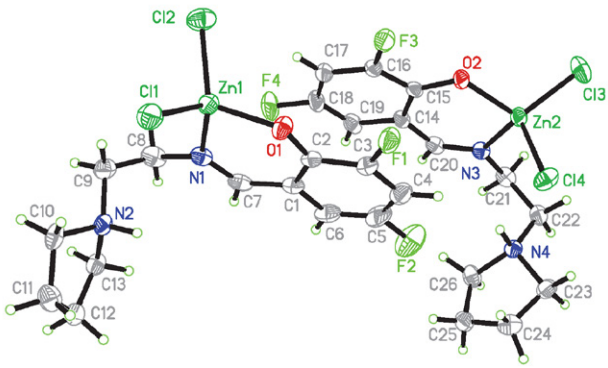


Figure 1. ORTEP plot of the crystal structure of complex **1**. Displacement ellipsoids of non-hydrogen atoms are drawn at the 30% probability level.

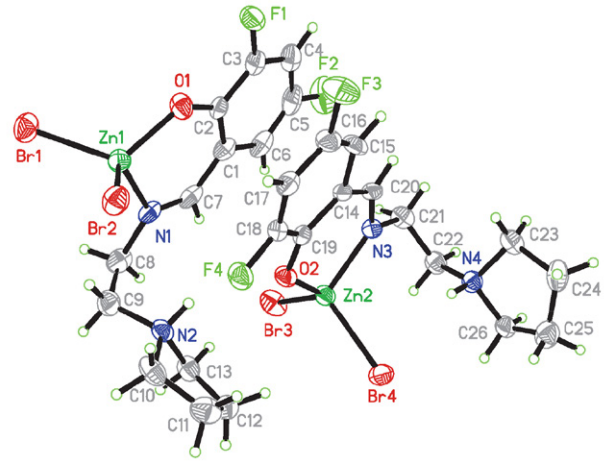


Figure 2. ORTEP plot of the crystal structure of complex **2**. Displacement ellipsoids of non-hydrogen atoms are drawn at the 30% probability level.

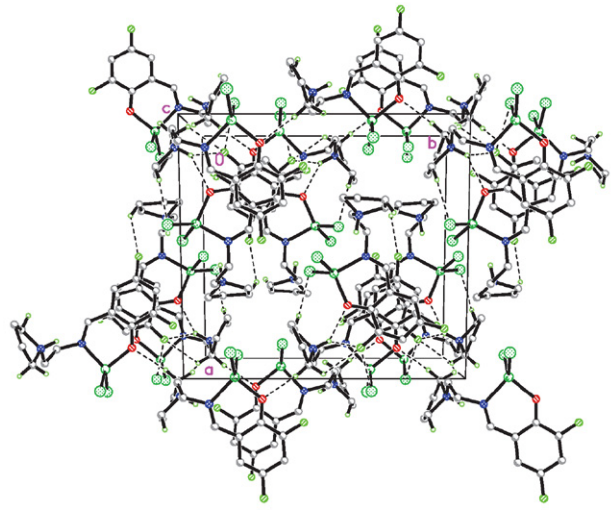


Figure 3. Molecular packing diagram of complex **1**. Viewed along the *c* axis. Hydrogen atoms not related to hydrogen bonding are omitted. Hydrogen bonds are shown as dashed lines.

In the crystal structures of the complexes, molecules are linked through intermolecular hydrogen bonds of types N–H···O, N–H···F, N–H···Cl/Br, C–H···O, C–H···F and C–H···Cl/Br (Table 3), to form three dimensional networks (Figures 3 and 4).

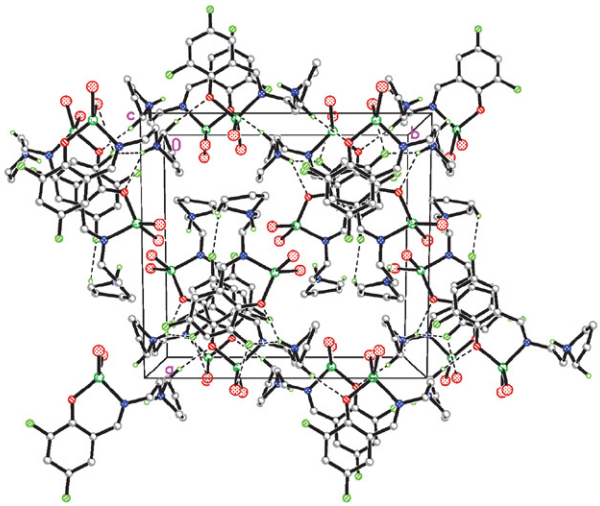


Figure 4. Molecular packing diagram of complex **2**. Viewed along the *c* axis. Hydrogen atoms not related to hydrogen bonding are omitted. Hydrogen bonds are shown as dashed lines.

3. 2. Crystal Structure Description of Complex 3

The molecular structure and atom numbering scheme of complex **3** is shown in Figure 5. The asymmetric unit of the complex contains [Zn(dca)L] unit. The [ZnL] moieties are linked through dicyanoamide ligands, forming a polymeric structure. The Zn atom is in trigonal bipyramidal coordination, with the basal plane defined by the imino nitrogen (N1) of the Schiff base ligand L, and two N atoms from two dca ligands. The axial positions are occupied by one phenolate oxygen (O1) and one pyrollidine nitrogen (N2). The Zn atom deviates from the least squares plane defined by the three basal donor atoms by 0.037(2) Å. The angular distortion in the trigonal bipyramidal environment comes from the five- and six-membered chelate rings taken by the Schiff base ligand, with angles of 77.90(11)° and 91.42(11)°. Distortion of the trigonal bipyramidal coordination can be observed from the coordinate bond angles, ranging from 106.06(12) to 128.72(11)° for the basal angles, and 169.17(11) ° for the diagonal angle. The trigonal bipyramidal coordination can be calculated from the structural index value τ of 0.67.[13] The Zn–O and Zn–N bonds are comparable to those observed in similar Schiff base zinc complexes.[14]

In the crystal structure of the complex, the [CuL] moieties are linked by dca ligands to form chain structure along the *c* axis. The chains are further linked by intermolecular hydrogen bonds of types C–H···N and C–H···F (Table 3), to form a three-dimensional network (Figure 6).

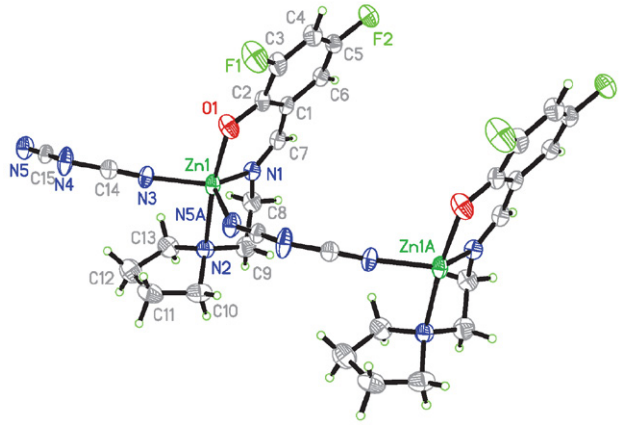


Figure 5. ORTEP plot of the crystal structure of complex **3**. Displacement ellipsoids of non-hydrogen atoms are drawn at the 30% probability level. Atoms labelled with the suffix A are related to the symmetry position *x*, *y*, 1 + *z*.

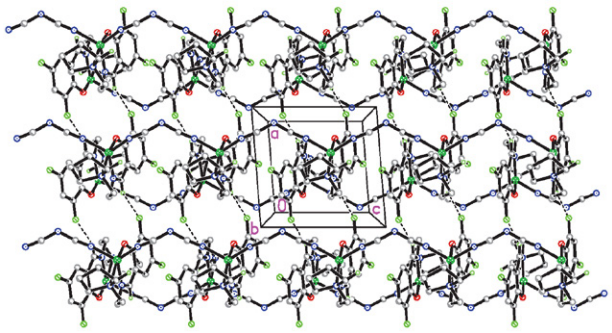


Figure 6. Molecular packing diagram of complex **3**. Viewed along the *b* axis. Hydrogen atoms not related to hydrogen bonding are omitted. Hydrogen bonds are shown as dashed lines.

3. 3. Antibacterial Activity

The antibacterial assay was performed according to the literature method.[15] Penicillin G was used as a standard drug. DMSO was used as solvent and the solutions were further diluted by distilled water. The DMSO at the tested concentration has no activity on the bacteria. The zone of inhibition for the 5000 μg mL^{-1} test solutions on the four bacteria *Escherichia coli*, *Pseudomonas aeruginosa*, *Salmonella typhi* and *Staphylococcus aureus* is given in Table 4. The MIC values are given in Table 5. The results indicated that the metal complexes have from medium to strong activities against the bacteria, and stronger than the free Schiff base. The three zinc complexes have similar activities against all the bacterial strains, indicating the Cl, Br and dca ligands have similar effects on antibacterial activities. The complexes show strong activity on *E. coli* and *P. aeruginosa*, and medium activity on *B. subtilis* and *S. aureus*. The three metal complexes have better activities against *E. coli*, *P. aeruginosa* and *S. aureus* than the reference drug penicillin G.

Table 4 Antibacterial screening results

Compound	Zone of inhibition (mm)			
	E. coli	*P. aeruginosa*	*B. subtilis*	*S. aureus*
HL	8.2 ± 1.5	6.7 ± 1.6	6.1 ± 1.2	5.5 ± 1.3
1	23 ± 1.6	25 ± 1.3	16 ± 1.5	18 ± 1.7
2	22 ± 1.8	23 ± 2.0	15 ± 1.7	17 ± 1.4
3	20 ± 2.2	21 ± 2.0	15 ± 1.8	16 ± 1.5
Penicillin G	30 ± 2.8	26 ± 3.1	30 ± 3.2	24 ± 2.9

Table 5 Antibacterial activities as MIC values (μg mL^{-1})

Compound	*E. coli*	*P. aeruginosa*	*B. subtilis*	*S. aureus*
HL	6.25	12.5	12.5	12.5
1	1.56	1.56	6.25	3.13
2	1.56	1.56	6.25	3.13
3	1.56	1.56	6.25	3.13
Penicillin G	3.13	6.25	1.56	6.25

4. Supplementary Material

CCDC 2376703–2376705 for the three complexes contains the supplementary crystallographic data for this paper. These data can be obtained free of charge at http://www.ccdc.cam.ac.uk/const/retrieving.html or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk.

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Povzetek

Sintetizirali smo dva enojedrna cinkova(II) kompleksa $[ZnCl_2(HL)]$ (**1**) in $[ZnBr_2(HL)]$ (**2**) ter polimerni cinkov(II) kompleks [Zn(dca)L] (**3**), kjer je HL zwitterionska oblika Schiffove baze 2,4-difluoro-6-(((2-pirolidin-1-il)etilimino) metil)fenol (HL), L je monoanionska oblika HL, dca pa je dicianoamid. Spojine smo okarakterizirali z elementno analizo in infrardečo spektroskopijo ter jim določili kristalne strukture z rentgensko monokristalno analizo. Kompleksa **1** in **2** sta izostrukturna, Zn centralni atomi imajo tetraedrično koordinacijo. Ligandi Schiffove baze so v obeh kompleksih koordinirani na Zn atome preko fenolatnega kisika in imino dušika. Cinkovi atomi v kompleksu **3** so tetraedrično koordinirani in jih povezujejo dicianoamidni mostnovni ligandi. Ligand Schiffove baze je v tem kompleksu koordiniran na cinkov atom preko fenolatnega kisika, imino dušika in pirolidinskega dušika. Molekule vseh treh kompleksov so stabilizirane z vodikovimi vezmi. Biološki testi kažejo, da imajo kompleksi dobro protimikrobno delovanje na bakterijske seve *Escherichia coli*, *Pseudomonas aeruginosa*, *Salmonella typhi* in *Staphylococcus aureus*.