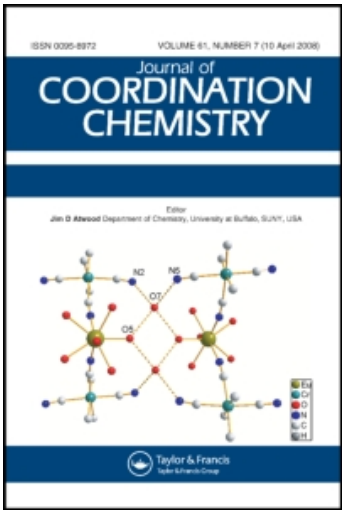

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Spectroscopic studies and crystal structure of a dimeric Zn(II) complex with diethyl (pyridin-2-ylmethyl)phosphate

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Spectroscopic studies and crystal structure of a dimeric Zn(II) complex with diethyl (pyridin-2-ylmethyl)phosphate

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The crystal structure of $[Zn_2(2\text{-pmOpe})_2Cl_4]$ (2-pmOpe = diethyl (pyridin-2-ylmethyl)phosphate) was determined by X-ray-diffraction method. The compound was also characterized by IR, far-IR, 1H, and ^{31}P NMR spectroscopy. In this compound, 2-pmOpe is a bidentate N,O-bridging ligand and Zn(II) are slightly distorted tetrahedral $ZnNOCl_2$. Zn(II) ions are doubly bridged by the 2-pmOpe ligands, resulting in a dinuclear species. The structure is stabilized by intermolecular C–H···O and C–H···Cl hydrogen bonds. The spectral properties are in agreement with the structural data.

Keywords: Zn(II); Phosphoric acid ester; N,O-donor ligand; Crystal structure; Spectroscopic properties

1. Introduction

Phosphonate and phosphate derivatives with heterocyclic pendants have broad spectrum biological properties [1–3]. In particular, biological activities of their Pd(II) and Pt(II) complexes as well as of their complexes with biologically relevant Ca^{2+}, Mg^{2+}, Na^+, and K^+ were described [4–22]. Recently, we demonstrated the reactivity of *N*-heterocyclic phosphonate [23–31] and phosphate ligands [32, 33] toward transition metal ions. These compounds have been examined by structural, spectroscopic, and magnetic studies. The N,O-donor phosphonate ligands which combine a pyridine or quinoline residue with phosphonate are of special interest because of a variety of ways in which they are bonded to metal ions. Their donor properties clearly depend on the kind of *N*-heterocyclic ring and anionic functionalities.

The investigations presented in this article are part of systematic studies of organophosphorus derivatives of pyridine and a continuation of our investigation on the interaction of phosphate ligands bearing *N*-heterocyclic nitrogen of pyridine, i.e., diethyl (pyridyn-2-ylmethyl)phosphate (2-pmOpe) with perchlorate metal salts.

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For $[Co(2\text{-pmOpe})_2(H_2O)_2](ClO_4)_2$, the crystal structure determined by X-ray diffraction has been presented [32]. Its structure reveals that in this compound 2-pmOpe is a bidentate N,O-chelating ligand. In the previous studies, the synthesis and spectroscopy of 2-pmOpe (figure 1) and its crystal structure of palladium(II) complex [19] have been presented. In *trans*-$[Pd(2\text{-pmOpe})_2Cl_2]$ (square-planar), 2-pmOpe is a *N*-monodentate ligand.

Here we are interested in the interaction of Zn(II) chloride with 2-pmOpe and the stoichiometry, geometrical preferences of the resulting compound, and coordination properties of the ligand. Zinc is one of the most important trace elements, playing a versatile role in biological systems due to its structural and catalytic roles in enzymes [34–40]. These studies provide a chemical basis for the biological activity of the ligand.

Further biological tests will be facilitated by a detailed understanding of the spectroscopic properties and X-ray structure of zinc complex with 2-pmOpe, and therefore we have now studied the new complex of empirical formula $[Zn(2\text{-pmOpe})Cl_2]$.

2. Experimental

2.1. *Reagents and physical measurements*

The starting materials and solvents for the synthesis were obtained commercially and used as received. Elemental analyses were carried out using a Perkin Elmer elemental analyzer 2400 CHN. Infrared spectra (100–4000 cm^{-1}) were recorded on a Bruker IFS 113v spectrophotometer using KBr pellets. ^1H and ^{31}P NMR spectra were recorded on a Varian Mercury − 300 spectrometer operating at 300 MHz. Chemical shifts were reported using the standard (δ) notation in ppm with respect to TMS (1%) as internal standard and H_3PO_4 (85%) as external standard.

2.2. *Synthesis of ligand and complex*

The diethyl (pyridyl-2-ylmethyl)phosphate (2-pmOpe) ligand was prepared according to procedure described in detail elsewhere [19]. The chloride complex was prepared by dissolving $ZnCl_2$ (1 mmol) in EtOH (10 mL) and adding dropwise under stirring to a solution of the ligand (1 mmol) in EtOH (20 mL). The crystalline product was obtained by slow evaporation of the solvent at room temperature. Anal. Calcd for $C_{10}H_{16}PO_4NCl_2Zn$ (%): C, 31.48; H, 4.23; N, 3.67. Found: C, 31.19; H, 4.45; N, 3.75.

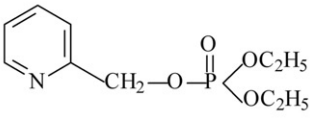

Figure 1. Schematic representation of 2-pmOpe.

2.3. *NMR spectral data of 2-pmOpe*

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.34 (t, 6H, $2CH_3$, $^3J_{HH} = 6.9$ Hz), 4.15 (dq, 4H, $2POCH_2$, $^3J_{HH} = 6.9$ Hz), 5.17 (d, 2H, py-CH_2OP, $^3J_{HP} = 7.8$ Hz), 7.23–7.27 (m, 1H, (py)H(5)), 7.51 (d, 1H, (py)H(3), $^3J_{H(4)H(3)} = 8.0$ Hz), 7.75 (t, 1H, (py)H(4)), 8.57 (d, 1H, (py)H(6), $^3J_{H(6)H(5)} = 4.8$ Hz); ^{31}P NMR (121 MHz, $CDCl_3$): $\delta + 0.027$.

2.4. *NMR spectral data of [Zn(2-pmOpe)Cl$_2$]*

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.34 (t, 6H, $2CH_3$, $^3J_{HH} = 7.1$ Hz), 4.25 (dq, 4H, $2POCH_2$, $^3J_{HH} = 7.1$ Hz), 5.55 (d, 2H, py-CH_2OP, $^3J_{HP} = 14.1$ Hz), 7.65–7.69 (m, 2H, (py)H(3) and (py)H(5)), 8.10 (t, 1H, (py)H(4)), and 9.16 (d, 1H, (py)H(6), $^3J_{H(6)H(5)} = 4.8$ Hz); ^{31}P NMR (121 MHz, $CDCl_3$): $\delta + 1.839$.

2.5. *Crystal structure determination*

X-ray data were collected on a Kuma KM4CCD diffractometer (Mo-Kα radiation; $\lambda = 0.71073$ Å). X-ray data were collected at 100 K using an Oxford Cryosystem device. Data reduction and analysis were carried out with the CrysAlice "RED" program [41]. The space group was determined using the XPREP program. Structure was solved by direct methods using the XS program and refined using all F^2 data, as implemented by the XL program [42]. Non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogens were placed at calculated positions. Before the last cycle of refinement, all hydrogens were fixed and allowed to ride on their parent atoms.

3. Results and discussion

3.1. *Description of the crystal structure*

The crystallographic parameters are summarized in table 1 and selected bond lengths and angles are listed in table 2. The arrangement of the components of the crystal structure showing the local environment and labeling scheme is depicted in figure 2.

Two chlorides, Cl(1) and Cl(2), with N(1) and O(2) $(-x+1, -y+1, -z+2)$ of 2-pmOpe form a distorted tetrahedral environment around Zn($ZnONCl_2$), with Zn–O, Zn–N, and Zn–Cl distances listed in table 2. The 2-pmOpe are N,O-bridging through pyridyl N(1) and phosphate O(2), resulting in the centrosymmetric dimer with the Zn$\cdots$Zn $(-z+1, -y+1, -z+2)$ distance of 6.854(2) Å.

The geometry of the phosphates deviate significantly from an ideal tetrahedron (table 2), reflected mainly in the formal double P(1) = O(2) and P(1)–O(1) bond lengths (being the shortest and the longest, respectively), and the values of phosphate O(1)–P(1)–O(2) and O(2)–P(1)–O(3) angles, in which the O(1) and O(2) atoms are involved.

The centrosymmetric dimer is additionally stabilized by C(7)–H(7A)$\cdots$O(2) $(-x+1, -y+1, -z+2)$ hydrogen interactions, the geometrical parameters of which are listed in table 3.

Neighboring dimers are linked to each other by C(3)–H(3)$\cdots$Cl(2) $(x, y-1, z)$, C(6)–H(6)$\cdots$Cl(1) $(-x, -y+1, -z+1)$, and C(4)–H(4)$\cdots$O(4) $(-x, -y, -z+2)$

Table 1. Crystal data and structure refinement for $[Zn_2(2\text{-pmOpe})_2Cl_4]$.

Empirical formula	$C_{20}H_{32}Cl_4N_2O_8P_2Zn_2$
Formula weight	762.96
Temperature (K)	100(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions (Å, °)	
a	8.331(2)
b	8.556(2)
c	11.028(3)
α	92.23(3)
β	93.19(3)
γ	101.21(5)
Volume (Å^3), Z	768.9(4), 1
Calculated density (Mg m^{-3})	1.648
Absorption coefficient (mm^{-1})	2.055
Crystal size (mm^3)	$0.46 \times 0.19 \times 0.05$
θ range for data collection (°)	3.12–36.61
Limiting indices	$-10 \le h \le 10$; $-10 \le k \le 10$; $-14 \le l \le 13$
Reflections collected	9013
Independent reflections	$R(\text{int}) = 3348$
Completeness to $2\theta = 54.00$	0.997
Absorption correction	Analytical
Data/parameters	3348/182
Goodness-of-fit on F^2	1.082
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0294$, $wR_2 = 0.0642$
Largest difference peak and hole (e Å^{-3})	0.412 and −0.354

Table 2. Bond lengths (Å) and angles (°) for $[Zn_2(2\text{-pmOpe})_2Cl_4]$.

Zn–O(2)^i	2.0212(16)	O(2)^i–Zn–Cl(1)	105.07(6)
Zn–N(1)	2.0564(19)	N(1)–Zn–Cl(1)	108.74(6)
Zn–Cl(2)	2.2124(9)	Cl(2)–Zn–Cl(1)	121.55(3)
Zn–Cl(1)	2.2235(9)	O(2)–P–O(4)	112.67(9)
P–O(1)	1.5607(16)	O(2)–P–O(3)	116.44(10)
P–O(2)	1.4841(17)	O(4)–P–O(3)	105.05(9)
P–O(3)	1.5494(16)	O(2)–P–O(1)	113.45(9)
P–O(4)	1.5492(17)	O(4)–P–O(1)	104.31(9)
O(2)^i–Zn–N(1)	100.90(8)	O(3)–P–O(1)	103.72(9)
O(2)^i–Zn–Cl(2)	105.57(6)	P–O(2)–Zn^i	137.96(10)
N(1)–Zn–Cl(2)	112.62(6)		

Symmetry transformations used to generate equivalent atoms: ^{i} $-x+1$, $-y+1$, $-z+2$.

hydrogen bonds resulting in a 3-D structure (table 3). The crystal packing showing 3-D arrangement in the crystal lattice is depicted in figure 3.

3.2. *NMR spectra of the ligand and $[Zn_2(2\text{-pmOpe})_2Cl_4]$*

The coordination mode of the dinuclear complex $[Zn_2(2\text{-pmOpe})_2Cl_4]$ was determined using ^{1}H and ^{31}P NMR. The comparison of spectra of $[Zn_2(2\text{-pmOpe})_2Cl_4]$ complex and free ligand [19] is presented in figure 4. The chemical shifts (ppm) and coupling constants (Hz) for the complex and the free ligand [19] are presented in Section 2.

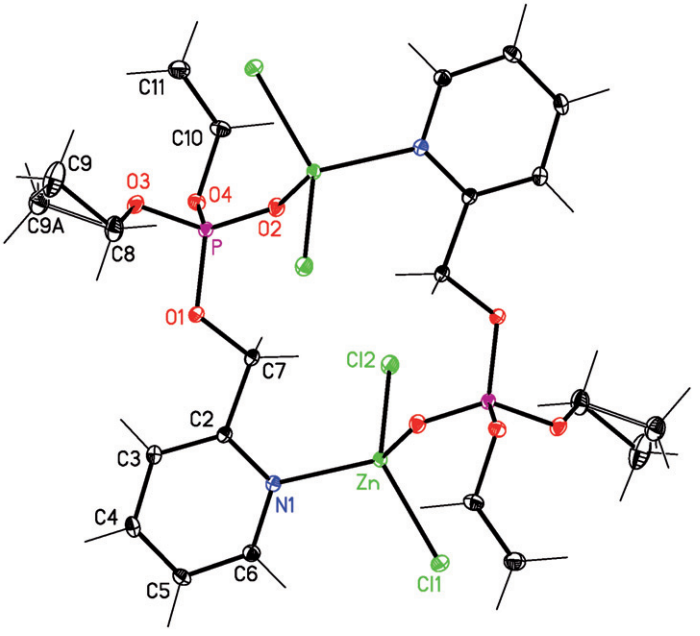

Figure 2. Molecular structure of $[Zn_2(2\text{-pmOpe})_2Cl_4]$ showing the atomic numbering.

Table 3. Hydrogen bonds for $[Zn_2(2\text{-pmOpe})_2Cl_4]$ (Å and °).

	d(D–H)	d(H···A)	d(D···A)	∠(DHA)
C(3)–H(3)···Cl(2)^{ii}	0.95	2.79	3.592(2)	143
C(6)–H(6)···Cl(1)^{iii}	0.95	2.94	3.622(2)	129
C(4)–H(4)···O(4)^{iv}	0.95	2.55	3.416(3)	152
C(7)–H(7A)···O(2)^{v}	0.99	2.52	3.340(3)	140

Symmetry transformations used to generate equivalent atoms: ^{ii} $x, y-1, z$; ^{iii} $-x, -y+1, -z+1$; ^{iv} $-x, -y, -z+2$; and ^{v} $-x+1, -y+1, -z+2$.

The resonance of the 2-substituted pyridine coordinated to zinc is noticeably different from that of non-coordinating ligand. Coupling constants for the complex are very similar to those of the free ligand.

After complexation, all signals of the pyridine protons and methylene group in the vicinity of pyridine ring are shifted to higher frequencies compared to the free ligand. H(6), H(5), H(4), and H(3) signals of 2-substituted pyridine ring for the zinc complex are shifted by 0.59, 0.42, 0.35, and 0.16 ppm, respectively. The methylene protons, closely adjacent to nitrogen atom of pyridine, are shifted by 0.38 ppm. These shifts confirm the coordination of the ligand to zinc ion through nitrogen of the pyridine ring.

3.3. *Infrared spectrum*

In the IR spectrum of the complex, bands due to the stretching modes of pyridine ν(C=C) and ν(C=N) observed at 1600–1500 cm^{-1} are not shifted appreciably (1592 and

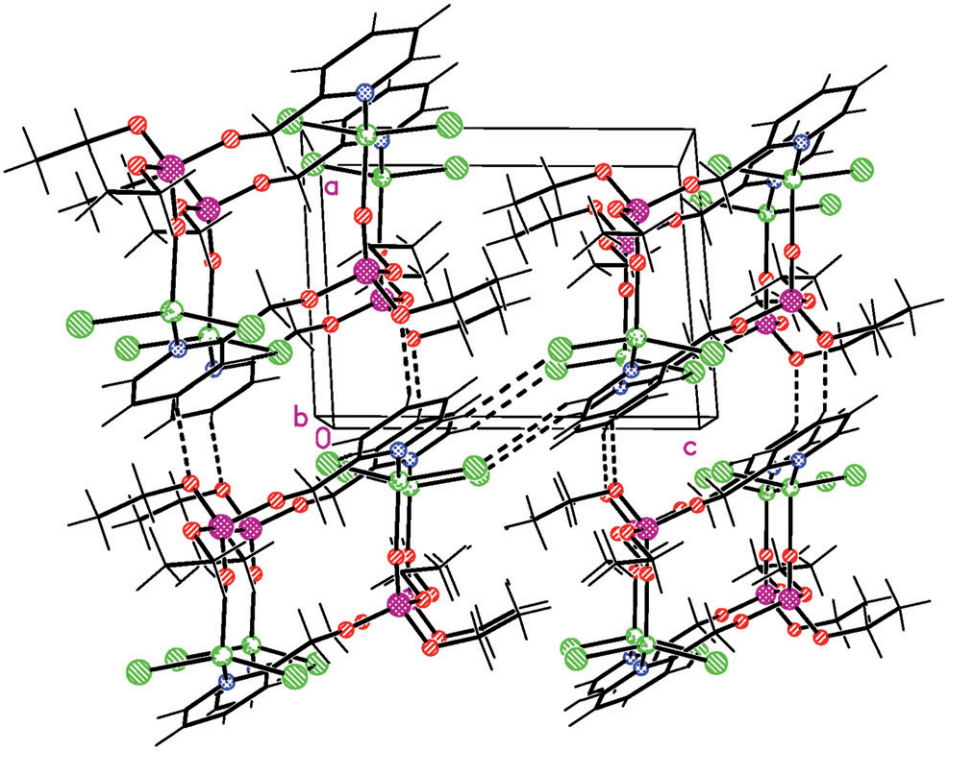

Figure 3. View of the packing of $[Zn_2(2\text{-pmOpe})_2Cl_4]$ along the *b*-axis.

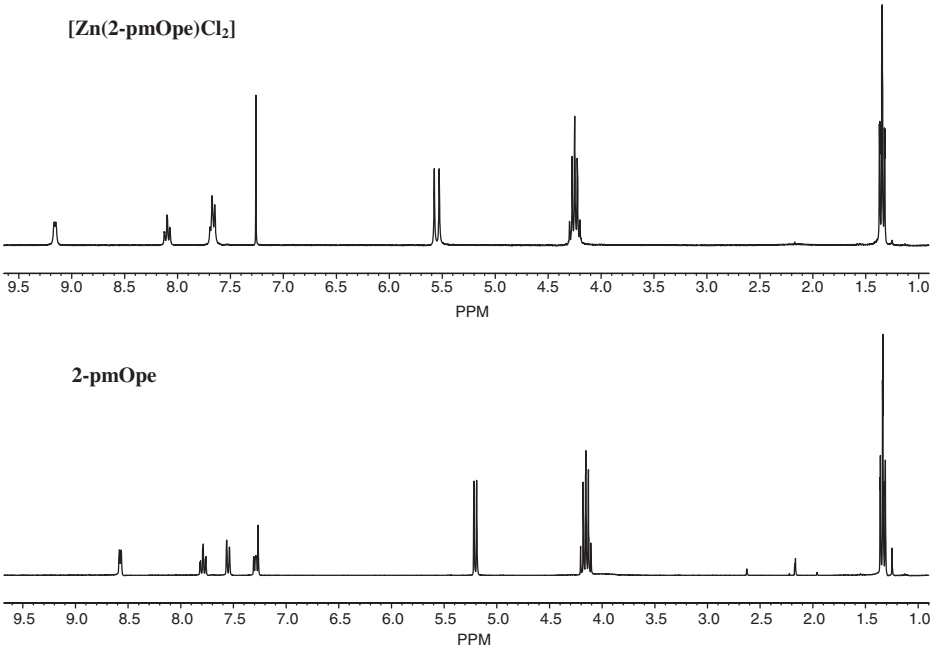

Figure 4. NMR spectra of 2-pmOpe and $[Zn_2(2\text{-pmOpe})_2Cl_4]$.

1573 cm^{-1} for free ligand, and 1612 and 1573 cm^{-1} in the complex), whereas the characteristic out-of-plane and in-plane deformation bands (605 and 400 cm^{-1}, respectively) of the 2-substituted pyridine ring are shifted to higher frequencies (613 and 420 cm^{-1}, respectively), suggesting the coordination of the pyridyl nitrogen. The band at 964 cm^{-1} is associated with a pyridine ring breathing mode and is characteristically shifted to higher energy on coordination. Thus, the band observed at 980 cm^{-1} indicates the coordination of pyridine. The very strong band at 1273 cm^{-1}, which corresponds to the P=O stretching frequencies of the free ligand in the spectrum of the complex, is shifted to lower frequency (1198 cm^{-1}), indicating the coordination of the phosphoryl oxygen to zinc. Other bands characteristic for the phosphate, δ(PO–C) at 1101 cm^{-1} and ν(P–OC) at 1034 cm^{-1}, do not show significant shifts upon Zn(II) complex formation (1106 and 1048 cm^{-1}, respectively). The far-IR region shows one band (250 cm^{-1}) attributed to ν(Zn–N). The ν(Zn–Cl) symmetric and asymmetric frequencies (304 and 339 cm^{-1}) in the compound studied are consistent with a pseudotetrahedral environment [43].

4. Conclusions

$ZnCl_2$ reacts with 2-pmOpe ligand in 1 : 1 molar metal/ligand ratio. In this study, [Zn(2-pmOpe)Cl_2] was isolated and the crystal structure was determined. In [Zn(2-pmOpe)Cl_2], the Zn(II) ions adopt tetrahedral coordination (most common for zinc). This compound is dimeric of formula [Zn_2(2-pmOpe)$_2Cl_4$], in which 2-pmOpe is a N,O-bridging ligand. Our earlier studies of perchlorate transition-metal complexes with 2-pmOpe ligand indicate that the ligand is able to act as N,O-chelating agent involving a seven-membered chelate ring.

Supplementary material

Crystallographic data for the structure have been deposited with the Cambridge Crystallographic Data Centre, CCDC-695271. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: int. code +(1223)336-033; Email for inquiry: fileserv@ccdc.cam.ac.uk).

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