CRYSTAL STRUCTURE OF BIS(2-PYRIDYMETHYL)AMINO-3-PROPIONATOZINC PERCHLORATE HEMIETHANOL SOLVATE

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Figure 1. ORTEP plot of the bis(2-pyridylmethyl)amino-3-propionatozinc cation depicting the octahedral geometry of the zinc atom. Selected bond distances and angles: Zn1-N1 2.071(4), Zn1-N2 2.180(4), Zn1-N3 2.076(4), Zn1-O1 2.006(4), Zn1-O2i 2.139(4), Zn-O1i 2.282(3) Å; N1-Zn1-N2 80.0(2), N1-Zn1-N3 153.9(2), N1-Zn1-O1 94.8(2), N1-Zn1-O1i 86.4(1), N1-Zn1-O2i 106.4(2), N2-Zn1-N3 78.3(2), N2-Zn1-O1 94.7(1), N2-Zn1-O1i 117.5(1), N2-Zn1-O2i 171.1(1), N3-Zn1-O1 101.4(2), N3-Zn1-O1i 90.8(2), N3-Zn1-O2i 93.8(2), O1-Zn1-O1i 147.3(1), O1-Zn1-O2 57.8(1), O1-Zn1-O2i 90.9(1) °. Symmetry transformation: $i = 1/2 - x, 1/2 + y, 1/2 - z$.

Comment

In the preceding paper, we mentioned our interest in the structural chemistry of the *N*-heterocyclic complexes of zinc(II) [1]. In continuing the studies, we have designed a potentially tetradentate ligand, bis(pyridyl-2-methyl)amino-3-propionic acid, $[\text{C}_5\text{H}_4\text{N}-2\text{-CH}_2]_2\text{N-CH}_2\text{CH}_2\text{C(O)OH}$,

whose deprotonated form can chelate to zinc through the nitrogen and oxygen atoms. In the title complex, the three amino atoms coordinate to the metal atom as expected. Interestingly, the carboxyl $-\text{CO}_2$ entity interacts with an adjacent zinc atom, the chelating interaction giving rise to the formation of a helical polycationic chain running along the *b*-axis of the unit cell (by the two-fold screw axial translation). The geometry is described as distorted octahedral, the distortion arising from the small bite of the carboxyl

group. The intermolecular zinc-oxygen_{carboxyl} bond (shown as a dash line) is only marginally longer than the corresponding zinc-oxygen_{carbonyl} distance.

Experimental

Synthesis of bis(pyridyl-2-methyl)amino-3-propionic acid. A solution of 2-picoly chloride hydrochloride (1.64 g, 1 mmol) was added to an aqueous solution of β -alanine (0.45 g, 5.0 mmol) and sodium hydroxide (0.64 g, 16 mmol). After being stirred overnight, the mixture was extracted with chloroform. The removal of the solvent yielded the crude white product, which was then purified by recrystallization from a 1:1 water-ethanol mixture.

To a solution of bis(pyridyl-2-methyl)amino-3-propionic acid (0.28 g, 1 mmol) in ethanol (10 mL) was added a solution of zinc nitrate hexahydrate (0.29 g, 1.0 mmol) and sodium perchlorate (0.14 g, 1 mmol) in ethanol (10 mL). The mixture yielded colorless crystals that were suitable for X-ray diffraction when it was set aside for a week. Calc. for $C_{16}H_{19}ClN_3O_6 \cdot 5Zn$: C, 41.94; H, 4.18; N, 9.17%. Found: C, 41.86; H, 4.25; N, 8.87%. The diffraction intensities were corrected for absorption effects [2].

Table 1. Crystal data for bis(2-pyridylmethyl)amino-3-propionatozinc perchlorate hemiethanol solvate

Empirical formula	$C_{16}H_{19}ClN_3O_6 \cdot 5Zn$	Formula weight	458.16
Space group	$C2/c$	Diffractometer	Siemens R3m
Unit cell dimensions	a 25.92(2) b 7.722(2) β 117.76(5)° c 20.91(1) Å	Volume, Å ³	3703(4)
μ (Mo-K α), mm ⁻¹	1.513	D_{calc} , g cm ⁻³	1.644
θ range, °	2.1 – 26.0	$F(000)$	1880
Reflections collected	3715	Temperature, °C	25
Independent reflections	3635 ($R_{int} = 0.025$)	Completeness of data	99.5%
No. parameters refined	244	Reflections with $I > 2\sigma(I)$	2406
$[I > 2\sigma(I)]$	0.055	R (all data)	0.093 Final R
w	$[\sigma^2 + (0.0818P)^2 + 6.6907P]^{-1}$	wR (all data)	0.157
Diff. hole and peak, eÅ ⁻³	-0.48 – 0.63	Goodness-of-fit on F^2	1.03
Programs	<i>SHELXS-97</i> , <i>SHELXL-97</i> [3,4]	CCDC deposition no.	165483

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