Crystal structure of bis(3,5-dichloro-*N*-*n*-propylsalicylaldiminato-*N*,*O*)zinc(II), $Zn(C_{10}H_{10}Cl_2NO)_2$

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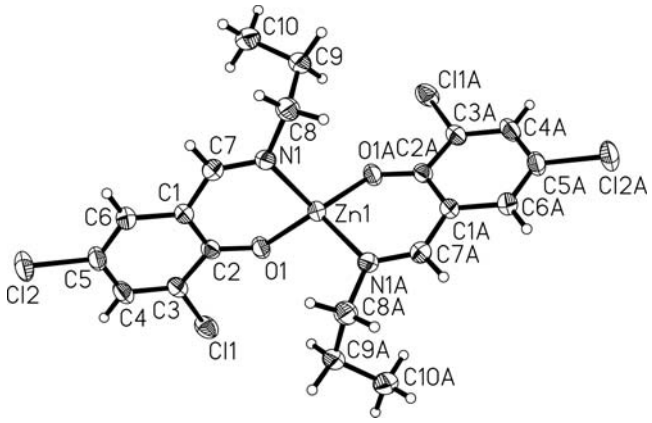
Received December 6, 2010, accepted and available on-line January 25, 2011; CCDC no. 1267/3318



Abstract

$C_{20}H_{20}Cl_4N_2O_2Zn$, monoclinic, *C*12/*c*1 (no. 15), a = 24.204(1) Å, b = 4.7284(3) Å, c = 21.384(1) Å, β = 114.845(3)°, V = 2220.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.085, T = 291 K.

Source of material

3,5-Dichlorosalicylaldehyde (382 mg, 2 mmol) and *n*-propylamine (118 mg, 2 mmol) were dissolved in an aqueous ethanol solution (25 mL). The mixture was stirred at room temperature for 2 h to give a clear yellow solution, which was added to a methanol solution (10 mL) of $Zn(NO_3)_2 \cdot 6H_2O$ (595 mg, 2 mmol). The mixture was stirred for another 10 min at room temperature to give a yellow solution and then filtered. The yellow single crystals suitable for X-ray analysis were obtained by slowly evaporating the above filtrate at room temperature. The crystals were isolated and dried in a vacuum desiccator containing anhydrous $CaCl_2$ (yield 68 %). Elemental analysis — found: C, 45.41 %; H, 3.90 %; N, 5.11 %; calculated for $C_{20}H_{20}ZnCl_4N_2O_2$: C, 45.53 %; H, 3.82 %; N, 5.31 %. IR data are available in the CIF.

Experimental details

All the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with d(C—H) = 0.93 - 0.97 Å, and with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C).

Discussion

Transition metal complexes of Schiff bases have been extensively investigated for many years due to their novel structures and potential applications in many fields [1-4]. These compounds play an important role in the development of coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architecture. Zinc(II) chelate complexes were studied as models for the active site of carbonic anhydrase and other hydrolytically active enzymes [5,6]. The role of zinc(II) structural properties in protein folding is also extensively studied [7-10]. The title complex is a mononuclear zinc(II) complex with a Schiff-base ligand. Zn(II) atom is four-coordinated by two N atoms and two O atoms from two Schiff-base ligands in a distorted tetrahedral arrangement. The dihedral angle between the coordination planes (N1/Zn1/O1 and N1A/Zn1A/O1A) is 84.23(5)°, and the dihedral angles between the plane six-membered ring (C1/C2/C3/C4/C5/C6) and the plane N1/Zn1/O1 is 17.91(8)° (symmetry codes A: $-x,y,½-z$). The bond angles ∠O1–Zn1–O1 and ∠N1–Zn1–N1 are 122.4(1)° and 119.0(1)°, respectively. The other angles subtended at the Zn(II) ion are in the range of 95.12(7)° - 113.57(7)°. The average bond lengths of Zn—O and Zn—N are 1.923(2) and 2.002(2) Å, respectively.

Table 1. Data collection and handling.

Crystal:	yellow needle, size 0.13 × 0.23 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	16.06 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5764, 2166
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1744
$N(param)_{refined}$:	133
Program:	SHELXTL [11]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(4)	8*f*	0.1340	0.4731	0.0677	0.066
H(6)	8*f*	0.1964	0.9950	0.2299	0.063
H(7)	8*f*	0.1503	1.0509	0.3032	0.057
H(8A)	8*f*	0.0481	1.1639	0.3582	0.068
H(8B)	8*f*	0.1165	1.2168	0.3727	0.068
H(9A)	8*f*	0.0781	0.7199	0.4162	0.072
H(9B)	8*f*	0.1036	0.9815	0.4650	0.072
H(10A)	8*f*	0.1718	0.6508	0.4131	0.110
H(10B)	8*f*	0.1816	0.6593	0.4904	0.110
H(10C)	8*f*	0.1976	0.9244	0.4572	0.110

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn(1)	4*e*	0	0.69557(9)	¼	0.0391(2)	0.0619(3)	0.0408(2)	0	0.0208(2)	0
Cl(1)	8*f*	0.03118(3)	0.2243(2)	0.07097(3)	0.0809(5)	0.0640(5)	0.0530(4)	–0.0103(3)	0.0356(4)	–0.0137(3)
Cl(2)	8*f*	0.23037(3)	0.8570(2)	0.12387(4)	0.0640(4)	0.1219(7)	0.0874(5)	0.0001(4)	0.0534(4)	0.0199(5)
N(1)	8*f*	0.07714(8)	0.9105(4)	0.30188(9)	0.043(1)	0.053(1)	0.038(1)	0.0041(9)	0.0165(8)	–0.0008(9)
O(1)	8*f*	0.02965(7)	0.4996(4)	0.19141(8)	0.0498(9)	0.060(1)	0.0485(9)	–0.0111(8)	0.0297(8)	–0.0101(8)
C(1)	8*f*	0.1195(1)	0.7855(5)	0.2197(1)	0.041(1)	0.053(1)	0.038(1)	0.001(1)	0.019(1)	0.003(1)
C(2)	8*f*	0.0754(1)	0.5844(5)	0.1795(1)	0.043(1)	0.046(1)	0.038(1)	0.005(1)	0.020(1)	0.006(1)
C(3)	8*f*	0.0837(1)	0.4710(5)	0.1223(1)	0.054(1)	0.049(2)	0.042(1)	0.004(1)	0.025(1)	0.002(1)
C(4)	8*f*	0.1304(1)	0.5508(6)	0.1058(1)	0.064(2)	0.064(2)	0.050(1)	0.013(1)	0.038(1)	0.008(1)
C(5)	8*f*	0.1720(1)	0.7472(6)	0.1463(1)	0.047(1)	0.072(2)	0.057(2)	0.007(1)	0.033(1)	0.015(1)
C(6)	8*f*	0.1676(1)	0.8641(6)	0.2029(1)	0.045(1)	0.067(2)	0.050(1)	–0.003(1)	0.023(1)	0.006(1)
C(7)	8*f*	0.1179(1)	0.9305(5)	0.2792(1)	0.044(1)	0.052(2)	0.045(1)	–0.005(1)	0.018(1)	–0.003(1)
C(8)	8*f*	0.0860(1)	1.0715(6)	0.3647(1)	0.056(1)	0.062(2)	0.053(1)	0.005(1)	0.025(1)	–0.012(1)
C(9)	8*f*	0.1060(1)	0.8788(7)	0.4269(1)	0.052(1)	0.085(2)	0.043(1)	0.006(1)	0.020(1)	–0.011(1)
C(10)	8*f*	0.1701(1)	0.7682(7)	0.4489(2)	0.054(2)	0.107(3)	0.051(2)	0.011(2)	0.015(1)	–0.001(2)

Acknowledgments. This work was financially supported by the Hubei National Science Foundation (grant no. 2009CDA022), and the Education Office of Hubei Province (grant no. D20091704).

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