Crystal structure of dichloro-bis[*N*-(4-cyanophenyl)salicylaldiminato]-dizinc(II), $Zn_2Cl_2(C_{14}H_9N_2O)_2$

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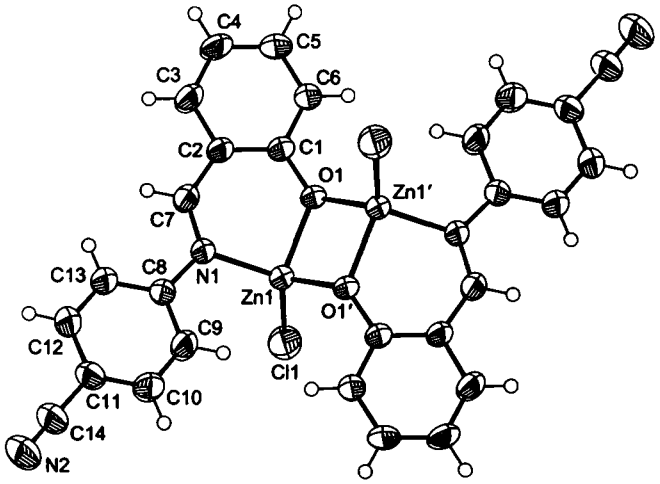

Abstract

$C_{28}H_{18}Cl_2N_4O_2Zn_2$, monoclinic, $P12_1/c1$ (no. 14), $a = 11.550(2)$ Å, $b = 13.987(2)$ Å, $c = 8.580(2)$ Å, $\beta = 110.36(1)°$, $V = 1299.5$ Å^3, $Z = 2$, $R_{gt}(F) = 0.035$, $wR_{ref}(F^2) = 0.115$, $T = 293$ K.

Source of material

A mixture of salicylaldehyde and 4-amino-benzonitrile in a 1:1 molar ratio was heated and the following recrystallization with ethanol gave an orange precipitate. To a solution of the previous compound (5 mmol) and piperidine (5 mmol) in ethanol (120 ml), a solution of $ZnCl_2$ (5 mmol) in ethanol (30 ml) was gradually added. After the mixture was stirred for 0.5 h while being heated at reflux, and stirred again for 24 h at room temperature, a yellow precipitate was produced. The crude product was collected by filtration, washed with ethanol, and finally dried under an infrared lamp. A single crystal suitable for X-ray crystallography was obtained by gradient-temperature vacuum sublimation.

Discussion

Zinc is able to adopt coordination numbers of 4, 5 or 6 in its complexes and changes its coordination geometry relatively easy. Therefore zinc complexes can display a variety of structural types, such as monomeric, dimeric and polymeric molecular structures [1-3].

Comparable with [2] the complex structure of the title compound is a dimer, whereas two zinc atoms are equivalent in one molecule. The crystal structure shows that the zinc ion is four-coordinate to the Schiff base ligands and the Cl atom. The bond angles O1–Zn1–O1′ and O1–Zn1–N1 are much less than 109.5°, therefore the coordination polyhedron around the Zn(II) ion is a distorted tetrahedron. The tension of the Zn1–O1–C1–C2–C7–N1 hexatomic ring results from the longer Zn1—O1′ and Zn1—N1 coordination bonds (d(Zn1—O1′) = 2.003(3) Å, d(Zn1—N1) = 2.005(3) Å opp. d(Zn1—O1) = 1.982(3) Å).

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.2 × 0.3 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	20.86 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3016, 2287
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2106
$N(param)_{refined}$:	172
Program:	SHELXTL [4]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	4e	0.7154	0.1717	0.7896	0.064
H(4A)	4e	0.5900	0.0592	0.6198	0.069
H(5A)	4e	0.4303	0.1075	0.3845	0.072
H(6A)	4e	0.3986	0.2666	0.3177	0.065
H(7A)	4e	0.7629	0.3223	0.8812	0.055
H(9A)	4e	0.7373	0.6234	0.8315	0.077
H(10A)	4e	0.8558	0.7130	1.0549	0.079
H(12A)	4e	0.9984	0.4722	1.2968	0.068
H(13A)	4e	0.8822	0.3839	1.0708	0.063

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)	4e	0.63719(4)	0.51465(3)	0.56844(5)	0.0421(3)	0.0302(3)	0.0417(3)	–0.0023(2)	0.0032(2)	0.0033(2)
Cl(1)	4e	0.7569(1)	0.5832(1)	0.4550(2)	0.0624(7)	0.0732(8)	0.0649(7)	–0.0098(6)	0.0199(5)	0.0169(6)
O(1)	4e	0.5180(2)	0.4106(2)	0.4684(3)	0.046(1)	0.025(1)	0.049(2)	–0.000(1)	0.002(1)	–0.001(1)
N(1)	4e	0.7189(3)	0.4449(2)	0.7830(4)	0.041(2)	0.031(2)	0.039(2)	0.001(1)	0.003(1)	–0.001(1)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(2)	4*e*	1.0744(4)	0.7011(4)	1.4470(6)	0.081(3)	0.069(3)	0.080(3)	–0.012(2)	–0.009(2)	–0.021(3)
C(1)	4*e*	0.5382(4)	0.3182(3)	0.5129(5)	0.044(2)	0.028(2)	0.044(2)	0.002(2)	0.011(2)	–0.002(2)
C(2)	4*e*	0.6344(4)	0.2900(3)	0.6591(5)	0.049(2)	0.028(2)	0.042(2)	0.002(2)	0.009(2)	–0.001(2)
C(3)	4*e*	0.6515(4)	0.1912(3)	0.6947(5)	0.067(3)	0.032(2)	0.053(2)	0.009(2)	0.009(2)	0.005(2)
C(4)	4*e*	0.5770(4)	0.1238(3)	0.5940(6)	0.078(3)	0.025(2)	0.064(3)	0.002(2)	0.016(2)	–0.001(2)
C(5)	4*e*	0.4821(4)	0.1529(3)	0.4533(6)	0.067(3)	0.029(2)	0.071(3)	–0.005(2)	0.008(2)	–0.008(2)
C(6)	4*e*	0.4630(4)	0.2486(3)	0.4133(6)	0.054(2)	0.031(2)	0.060(3)	–0.000(2)	–0.003(2)	–0.003(2)
C(7)	4*e*	0.7114(4)	0.3522(3)	0.7852(5)	0.053(2)	0.035(2)	0.041(2)	0.004(2)	0.004(2)	0.002(2)
C(8)	4*e*	0.7967(4)	0.4943(3)	0.9283(5)	0.042(2)	0.034(2)	0.039(2)	–0.000(2)	0.007(2)	–0.003(2)
C(9)	4*e*	0.7904(5)	0.5928(3)	0.9254(6)	0.079(3)	0.035(2)	0.052(2)	0.007(2)	–0.010(2)	–0.002(2)
C(10)	4*e*	0.8612(5)	0.6467(3)	1.0588(6)	0.081(3)	0.034(2)	0.063(3)	0.003(2)	0.001(2)	–0.008(2)
C(11)	4*e*	0.9394(4)	0.6021(3)	1.1969(5)	0.046(2)	0.044(2)	0.050(2)	–0.005(2)	0.008(2)	–0.009(2)
C(12)	4*e*	0.9463(4)	0.5028(3)	1.2022(6)	0.058(3)	0.048(2)	0.047(2)	–0.001(2)	–0.003(2)	0.001(2)
C(13)	4*e*	0.8761(4)	0.4502(3)	1.0676(5)	0.059(3)	0.035(2)	0.049(2)	–0.001(2)	–0.001(2)	0.000(2)
C(14)	4*e*	1.0147(4)	0.6568(3)	1.3375(6)	0.057(3)	0.053(3)	0.060(3)	–0.007(2)	0.005(2)	–0.012(2)

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