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NOTE

Crystal Structure of *bis*-[5-Chloro-2-hydroxybenzaldehyde]copper(II)

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A new Schiff base complex CuL_2 [HL = 5-chloro-2-hydroxybenzaldehyde] is synthesized and characterized using single crystal X-ray diffraction analysis. The crystal belongs to the monoclinic system, space group P2(1)/c, with a = 13.79(2), b = 3.818(6) (10), c = 12.51(2) Å, β = 102.49(2)º, V = 643.5(19) Å^3, Z = 2, Dc = 1.934 g/cm^3, R(int) = 0.0622. The geometry around copper(II) is a distorted square planar coordination geometry. The units of the complex are linked *via* the weak intermolecular Cu...O contracts, leading to the formation of one-dimension (1D) chains along the c axis.

Key Words: Copper(II), Complexes, Single crystal, X-ray analysis.

Copper(II) complexes have a wide range of biological activity and some of these complexes have been known to be antitumour, antiviral and antiinflammatory[1,2].

Thus, it is quite important to have a good understanding of the structure of such metal complexes. In this paper, the complex, *bis*-[5-chloro-2-hydroxybenzaldehyde] copper(II), is obtained from the reaction of 5-chloro-2-hydroxybenzaldehyde and $CuCl_2{\cdot}2H_2O$. Single crystal X-ray analyses of the complex is undertaken to elucidate the conformation and structure of the complex.

Preparation: To the solution of 5-chloro-2-hydroxybenzaldehyde (0.002 mol) in methanol (10 mL) and $CuCl_2{\cdot}H_2O$ (0.001 mol) in methnol (10 mL) was added. The mixture was stirring for 0.5 h and then filtered, the filtrate was left to stand undisturbed at room temperature. After a week brown block crystals suitable for X-ray analysis were obtained in 60 % yield.

Crystal structure determination: The crystallographic data, experimental details and parameters of the structure solution and refinement for the complex are summarized in Table-1. All X-ray diffraction measurements were performed at 153(2) K according to the standard procedure on a Bruker Smart 1000 CCD diffractometer equipped with a graphite-monochromated MoK_α radiation (λ = 0.71073Å). The correction for absorption was applied Semi-empirically with the SADABS program. The structure was solved by direct methods and refined using the full matrix least squares method in the anisotropic approximation for the non-hydrogen atoms with the SHELXLT program package[3,4]. All the non-hydrogen atoms were refined anisotropically and hydrogen atoms were located at their idealized positions.

The coordinates and thermal parameters of the atoms in the structure of *bis*-[5-chloro-2-hydroxybenzaldehyde] copper(II) have been deposited with the Cambridge structural database (CCDC no. 839325). The selected bond lengths and angles in the crystal structure of the complex are presented in Table-2.

The compound has a crystallographic two-fold axis passing through Cu1. The Cu atom is coordinated by four oxygen atoms in a distorted square planar coordination geometry (Fig. 1), where the dihedral angle between the two coordination planes defined by O1Cu1O2 and O1ACu1O2A is 0.0º and the phenyl ring plane (C1/C2/C3/C4/C5/C6) and the chelate ring (O1/Cu1/O2/C7/C6/C1) are nearly coplanar with a dihedral angle of 4.8(3)º. Bond angles also show that the coordination geometry about the copper atom in the title complex is a slightly distorted square planar structure, with O1Cu1O2, O1ACu1O2A and O1Cu1O1A angles of 93.19(16)º, 86.81(16)º and 180.00(15)º, respectively. [Symmetry code A: -x, - y + 0.5, - z + 1.5]. The Cu1O1 and Cu1O2 distances are 1.918(4) (4) Å and 1.932(5) Å, respectively. The distances are approach to the values found in other two coordinate copper complexes with similar ligands[5,6]. The phenyl rings of the ligands are nearly coplanar with the chelating rings. This is probably a result of the coordinate of the O atoms with copper(II).

A view of the crystal cell of the complex is shown in Fig. 2. The 1D infinite chain structure is formed with the weak Cu…O intermolecular contacts linkages along the c axis.

TABLE-1
CRYSTAL DATA AND STRUCTURE
REFINEMENT PARAMETERS

Properties	Complex
Empirical formula	$C_{14}H_8O_4Br_2Cl_2Cu$
Formula weight	374.64
Temperature (K)	153 (2)
Radiation(MoK_α), λ (Å)	0.71073
Crystal shape/colour	Prism/gray
Crystal size (mm^3)	0.41 × 0.11 × 0.07 mm
Crystal system	Monoclinic
Space group	P2(1)/c
a (Å)	13.79(2)
b (Å)	3.818(6)
c (Å)	12.51(2)
β (º)	90.00
V ($Å^3$)	643.5(19)
Z	2
D_c (g/cm^3)	1.730
μ (mm^{-1})	2.123
F (000)	374
θ range (º)	3.026/27.481
Index range (h, k, l)	-16/17, -4/4, -16/13
Measured reflections	3926
Observed reflections [I ≥ 2σ (I)]	1010
Data/restraints/parameters	1432/0/97
Goodness-of-fit on F^2	0.999
R_1, wR_2 [I ≥ 2σ (I)]	0.0622/0.1500
R_1, wR_2 (all data)	0.0782/0.1572
Large diff. peak and hole (e $Å^{-3}$)	0.178 and -1.151

Note: $R_1 = \Sigma||F_o|-|F_c||/|F_o|$, $wR_2 = [\Sigma w(Fo^2-Fc^2)^2/\Sigma w(Fo^2)]^{1/2}$, where w = $1/[\sigma^2(Fo^2) + (0.0685P)^2 + 2.3600P$, $P = (Fo^2 + 2Fc^2)/3$

TABLE-2
SELECTED BOND LENGTHS (Å) AND BOND ANGLES (º)

Bond	Å	Angle	º
Cu(1)-O(1)	1.914(4)	O(1)-Cu(1)-O(1A)	180.0
Cu(1)-O(1A)	1.914(4)	O(1A)-Cu(1)-O(2A)	93.19(17)
Cu(1)-O(2)	1.932(5)	O(1)-Cu(1)-O(2A)	86.80(17)
Cu(1)-O(2A)	1.932(5)		
Cl(1)-C(4)	1.744(6)		

Symmetry code: A, -*x*, -*y*+0.5, -z +1.5

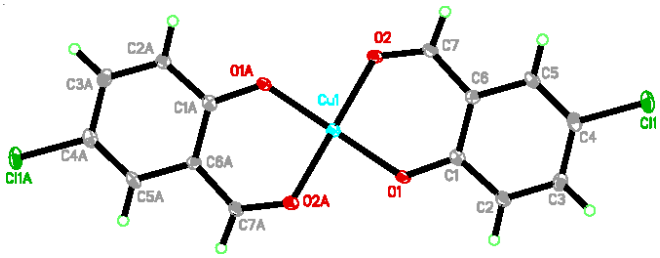

Fig. 1. A view of the complex, showing 30 % probability displacement ellipsoids (Symmetry code: A -x, y, 1/2-z)

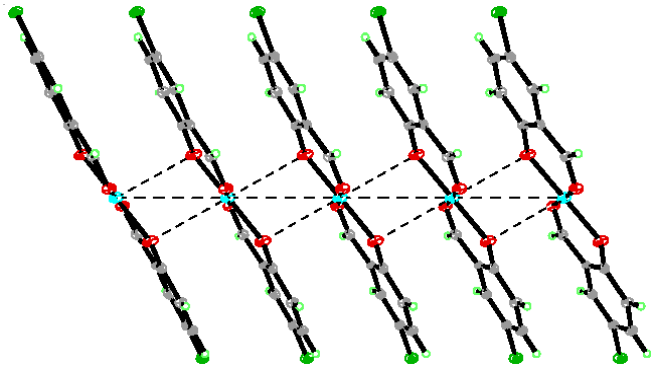

Fig. 2. 1D chain of complex along the c axis. (The dash lines represent the weak intermolecular Cu…O interactions)

Conclusion

In summary, we have designed and synthesized a 1D chain compound, which is constructed from 5-chloro-2-hydroxybenzaldehyde and Cu(II). In this compound, it possesses extensive weak Cu…O intermolecular contacts which connect the single molecules to form the 1D chain and stabilize the structure.

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