J Chem Crystallogr (2011) 41:82–84
DOI 10.1007/s10870-010-9920-6

COMMUNICATION

Microwave Synthesis and Crystal Structure of 2-Hydroxy-3-iodo-benzaldehyde-copper (II)

Suo-Ping Xu · Xiao-Liang Wang · Jian-Feng Tang · Ban-Feng Ruan · Hai-Liang Zhu

Received: 21 March 2010 / Accepted: 28 November 2010 / Published online: 14 December 2010

Abstract One new complex, 2-hydroxy-3-iodo-benzaldehyde-copper (II) has been designed and microwave synthesized. The structure was determined by UV, IR and single X-ray crystallography study. The title complex $C_{14}H_8CuI_2O_4$ crystallizes in the orthorhombic space group $Pna2_1$ with the cell parameters $a = 12.7897(12)$ Å, $b = 6.1132(8)$ Å, $c = 19.5114(18)$ Å, $V = 1525.5(3)$ Å^3 and $Z = 4$. The central copper (II) is four-coordinated by four oxygen atoms from two 3-iodosalicylaldehyde. The complex is linked into rhombic crystals by weak intermolecular interactions.

Keywords Crystal structure · 3-iodosalicylaldehyde · Microwave synthesis · Copper (II)

Introduction

Copper is an important life-required element. Many copper (II) complexes were prepared and structurally determined by single X-ray analysis [1–4]. Copper element exists in some native enzymes and is required by creatures. Due to their important relevant biological behaviors, many copper complexes with Schiff base have been reported in recent years [5–8]. However, to our knowledge, there are no structure reports about copper (II) complexes with 2-hydroxy-3-iodo-benzaldehyde. In this paper, the complex, 2-hydroxy-3-iodo-benzaldehyde-copper (II), is obtained from 2-hydroxy-3-iodo-benzaldehyde and $Cu(CH_3COO)_2 \cdot H_2O$ by microwave synthesis. The study will report crystal structure of 2-hydroxy-3-iodo-benzaldehyde-copper (II) (Scheme 1).

S.-P. Xu · X.-L. Wang · J.-F. Tang · B.-F. Ruan · H.-L. Zhu (✉)
State Key Laboratory of Pharmaceutical Biotechnology, Nanjing University, Nanjing 210093, China
e-mail: zhuhl@nju.edu.cn

S.-P. Xu
School of Chemistry and Chemical Engineering, Xuzhou Normal University, Xuzhou 221116, China

Experimental

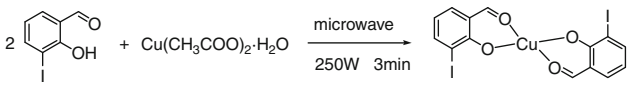
The 2-hydroxy-3-iodo-benzaldehyde was synthesized with salicylaldehyde, KI and KIO_3 [9]. The other chemicals (reagent grade) used were commercially available. UV spectra were recorded on a U-3000 Spectrophotometer. IR spectra were recorded on a Nexus 870 FT-IR. The crystal structure of complex was determined using SMART 1000 CCD diffractometer instrument. Melting points were measured on a Boetius micro melting point apparatus.

Synthesis of the Complex

Copper (II) acetate and two equivalent molar of 2-hydroxy-3-iodo-benzaldehyde were mixed together and microwave radiated 3 min in 250 W. Then, the breen powder was dissolved in DMF and stood still in air for 2 weeks. Large rhombic crystals were precipitated. They were filtered and washed with ethanol and dried in a vacuum desiccator containing anhydrous $CaCl_2$. The yield is 75%, m.p: 348–350 °C. UV (λ nm): 256, 410. Selected IR data (cm^{-1}, KBr): 3443 (w), 1591 (s), 1501 (m), 1422 (s), 1403 (s), 1329 (m), 1216 (m), 750 (m), 674 (m).

X-ray Crystal Structure Determinations

A single crystal of the title complex with dimensions 0.17 × 0.15 × 0.15 mm was chosen for X-ray diffraction



Scheme 1 Synthesis of the complex

Table 1 Experimental crystallographic data

Empirical formula	$C_{14}H_8CuI_2O_4$
Formula weight	557.54
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	$Pna2_1$
Cell dimensions	$a = 12.7897(12)$ Å
	$b = 6.1132(8)$ Å
	$c = 19.5114(18)$ Å
Volume	$V = 1525.5(3)$ Å^3
Z	4
Density(calculated)	2.428 Mg/m^3
Absorption coefficient	5.490 mm^{-1}
F_{000}	1036
Crystal size	0.17 × 0.15 × 0.15 mm
Theta range for data collection	2.09–27.00°
Index ranges	$-16 \le h \le 15$, $-3 \le k \le 7$, $-24 \le l \le 23$
Reflections collected	7855
Independent reflections	3252 [R(int) = 0.0464]
Data/restraints/parameters	3252/1/190
Goodness-of-fit on F^2	1.031
Final R indices [$I > 2\ \sigma(I)$]	$R1 = 0.0532$, $wR2 = 0.1269$
R indices(all data)	$R1 = 0.0855$, $wR2 = 0.1506$
Extinction correction	None
Largest diff. peak and hole	1.625 and −1.049 e Å^{-3}
Deposition number	CCDC

study. The data were collected with graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) using $\omega-2\theta$ scan technique. The structure was solved using direct methods and refined by full-matrix least-squares techniques. All non-hydrogen atoms were assigned anisotropic displacement parameters in the refinement. All hydrogen atoms were added at calculated positions and refined using a riding model. The structures were refined on F^2 using SHELXTL-97 10]. The crystal used for the diffraction study showed no decomposition during data collection. The crystal data and refinement data are listed in Table 1. Selected bond lengths and bond angles are given in Table 2.

Table 2 Selected bond lengths (Å) and angles (°)

Atoms	Length	Atoms	Length
Cu–O4	1.891(9)	Cu–O2	1.896(10)
Cu–O1	1.946(9)	Cu–O3	1.953(9)
I1–C4	2.110(14)	I2–C11	2.110(14)
O1–C1	1.268(18)	O2–C3	1.307(16)
O3–C8	1.237(15)	O4–C10	1.288(15)
Atoms	**Angles**	**Atoms**	**Angles**
O4–Cu–O2	179.5(5)	O4–Cu–O1	86.8(4)
O2–Cu–O1	92.7(4)	O4–Cu–O3	93.0(4)
O2–Cu–O3	87.5(4)	O1–Cu–O3	179.5(5)
C1–O1–Cu	126.0(9)	C3–O2–Cu	128.4(9)
C8–O3–Cu	125.6(8)	C10–O4–Cu	126.3(8)

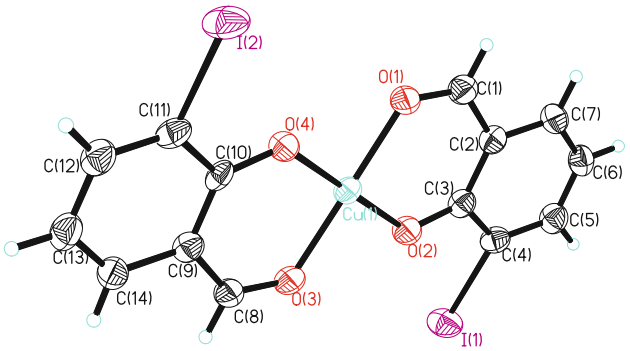


Fig. 1 A view of the title complex, showing 30% probability displacement ellipsoids (*arbitrary spheres* for the H atoms)

Results and Discussion

The title crystal structure consists of mononuclear complex (Fig. 1). The central copper (II) atom is four-coordinated by four oxygen atoms from two 3-iodosalicylaldehyde. The 3-iodosalicylaldehyde is bidentate ligand. The copper atom is in a distorted tetrahedral environment. The Cu–O bonds with the lengths of 1.891(9), 1.896(10), 1.946(9) and 1.953(9) Å are in normal range [11–13]. In the crystal structure of complex, the atoms of phenyl ring plane A (C2/C3/C4/C5/C6/C7) and plane B (O2/Cu/O1/C1/C2/C3), display an almost coplanar configuration with the dihedral angle being 3.8(4)°. The plane B (O2/Cu/O1/C1/C2/C3) and plane C (O4/Cu/O3/C8/C9/C10) display coplanar configuration with the dihedral angle being 0.2(1)°. The plane C (O4/Cu/O3/C8/C9/C10) and plane D (C9/C10/C11/C12/C13/C14) display an almost coplanar configuration with the dihedral angle being 5.3(2)°. In the title crystal structure indicated by the torsion angles separately being O4–Cu–O1–C1 = 174.69(3)°, O2–Cu–O1–C1 = −5.4(6)° and O3–Cu–O1–C1 = −117.0(4)°. X-ray

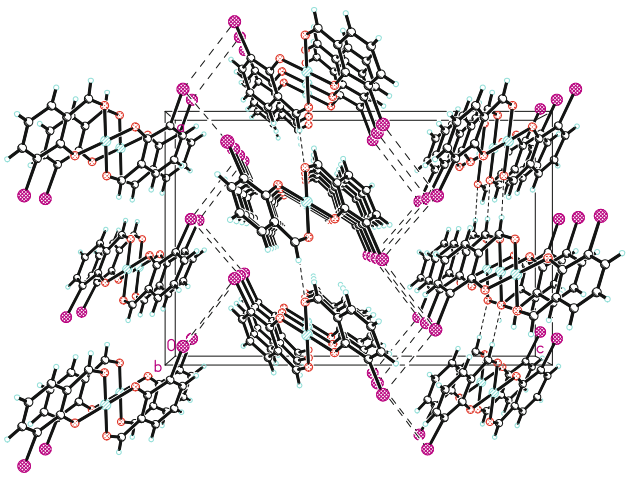


Fig. 2 The packing structure of complex along the b-axis, showing the formation of column by weak I···I intermolecular interactions

analysis reveals that intermolecular hydrogen bond C(1)-H(1)···O(3) (the bond length is 3.493(2) Å and the bond angle is 171.0(7)°) and molecules participate to form weak intermolecular interactions, which join the constituents into a three-dimensional network structure (Fig. 2).

Acknowledgments The work was co-financed by grants (Projects 30772627) from National Natural Science Foundation of China and (Projects 09KJD150005) from Natural Science Foundation of Jiangsu Province, China.

References

1. You ZL, Zhu HL (2004) Z Kristallogr 630(15):2754–2760
2. Roth A, Spielberg ET, Plass W (2007) Inorg Chem 46(11): 4362–4364
3. Xu S.-P, Shi L, Lv P.-C, Fang R.-Q, Zhu H.-L (2009) J Coord Chem 62(12):2048–2057
4. Liu J, Zhang T.-X, Lu T.-B, Qu L.-H, Zhou H, Zhang Q.-L, Ji L.-N (2002) J Inorg Biochem 91(1):269–276
5. Li C.-H, Wang Q, Xu Y.-Q, Hu C.-W (2008) Chin J Struct Chem 27(2):187–190
6. Shi L, Ge H.-M, Tan S.-H, Li H.-Q, Song Y.-C, Zhu H.-L, Tan R.-X (2007) Eur J Med Chem 42:558–564
7. Peng L.-L, Ye J.-C (2004) J Hubei Norm Univ (Nat Sci) 24(4): 86–88
8. Xu S.-P, Shi L, Lv P.-C, Fang R.-Q, Zhu H.-L (2009) J Coord Chem 62(12):2048–2057
9. Xu S.-P, Zhu G.-Z, Fang R.-Q, Li X.-L, Zhu H.-L (2009) Chin J Struct Chem 28(1):87–93
10. Sheldrick GM (2008) Acta Cryst A 64:112–122
11. Akitsu T, Einaga Y (2004) Acta Crystallogr C60:m640–m642
12. Akitsu T, Einaga Y (2004) Acta Crystallogr E60:m1605–m1607
13. Akitsu T, Einaga Y (2004) Acta Crystallogr E60:m1552–m1554