Jin-Xia Ma, Qing-Lin Li, Peng-Peng Li, Ji-Xing Zhao and Li Zhao*

Synthesis and crystal structure of (*E*)-1-(4-(((*E*)-5-chloro-2-hydroxybenzylidene)amino)phenyl)ethan-1-one *O*-methyl oxime, C₁₆H₁₅ClN₂O₂

<https://doi.org/10.1515/ncrs-2017-0355>

Received November 15, 2017; accepted March 7, 2018; available online March 16, 2018

Abstract

C₁₆H₁₅ClN₂O₂, monoclinic, *P*₂₁/*c* (no. 14), *a* = 33.491(11) Å, *b* = 7.124(2) Å, *c* = 6.1977(19) Å, β = 92.212(6)°, *V* = 1477.6(8) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0418, *wR*_{ref}(*F*²) = 0.1193, *T* = 296(2) K.

CCDC no.: 1828200

Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.27 × 0.25 × 0.22 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.26 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ _{max} , completeness:	25°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	7801, 2604, 0.028
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2012
<i>N</i> (<i>param</i>) _{refined} :	193
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cl1	0.03656(2)	0.57169(11)	0.32995(11)	0.0708(3)
N1	0.21967(5)	0.5103(2)	0.1187(3)	0.0417(4)
N2	0.40580(5)	0.5223(3)	0.3165(3)	0.0525(5)
O1	0.17301(5)	0.4278(2)	−0.2133(2)	0.0596(4)
H1A	0.1942	0.4483	−0.1466	0.089*
O2	0.44547(4)	0.5162(3)	0.4015(3)	0.0651(5)
C1	0.11554(6)	0.5600(3)	0.2553(3)	0.0423(5)
H1	0.1193	0.6039	0.3959	0.051*
C2	0.07743(6)	0.5313(3)	0.1709(3)	0.0473(5)
C3	0.07137(6)	0.4667(3)	−0.0392(4)	0.0511(6)
H3	0.0455	0.4464	−0.0949	0.061*
C4	0.10350(6)	0.4328(3)	−0.1649(3)	0.0502(5)
H4	0.0992	0.3903	−0.3058	0.060*
C5	0.14230(6)	0.4612(3)	−0.0838(3)	0.0432(5)
C6	0.14857(6)	0.5236(3)	0.1311(3)	0.0384(5)
C7	0.18861(6)	0.5407(3)	0.2278(3)	0.0405(5)
H7	0.1918	0.5745	0.3724	0.049*
C8	0.25859(6)	0.5099(3)	0.2157(3)	0.0373(4)
C9	0.28942(6)	0.5813(3)	0.0976(3)	0.0394(5)
H9	0.2838	0.6313	−0.0388	0.047*
C10	0.32829(6)	0.5792(3)	0.1793(3)	0.0390(5)
H10	0.3484	0.6308	0.0987	0.047*
C11	0.33799(5)	0.5007(2)	0.3820(3)	0.0354(4)
C12	0.30699(6)	0.4271(3)	0.4992(3)	0.0411(5)
H12	0.3127	0.3745	0.6343	0.049*
C13	0.26782(6)	0.4308(3)	0.4180(3)	0.0412(5)
H13	0.2476	0.3804	0.4986	0.049*
C14	0.38040(6)	0.4955(3)	0.4628(3)	0.0398(5)
C15	0.39156(7)	0.4617(4)	0.6951(4)	0.0589(6)
H15A	0.4100	0.3589	0.7070	0.088*
H15B	0.3680	0.4320	0.7718	0.088*
H15C	0.4038	0.5725	0.7560	0.088*
C16	0.47112(7)	0.5373(5)	0.2244(5)	0.0871(9)
H16A	0.4670	0.4346	0.1258	0.131*
H16B	0.4985	0.5381	0.2771	0.131*
H16C	0.4652	0.6533	0.1513	0.131*

*Corresponding author: Li Zhao, Lanzhou Jiaotong University, School of Chemical and Biological Engineering, Lanzhou 730070, P.R. China, e-mail: zhaoli_72@163.com

Jin-Xia Ma, Qing-Lin Li, Peng-Peng Li and Ji-Xing Zhao: Lanzhou Jiaotong University, School of Chemical and Biological Engineering, Lanzhou 730070, P.R. China

Source of material

All solvents and other reagents were of analytical grade and our methods of synthesis for the title compound has been improved [4–6]. An ethanol (5 mL) solution of 1-(4-aminophenyl)ethanone *O*-methyl-oxime (146.2 mg, 1 mmol)

was added to an ethanol solution (5 mL) of 5-chlorine-salicylaldehyde (156.5 mg, 1 mmol). The reaction mixture was stirred under refluxed conditions at 328 K for 11 h, cooling to room temperature. The mixture was filtered and washed, successively. The product was dried under vacuum and a yellow solid was obtained (yield 86.5%). The crystals were obtained by slow evaporation from chloroform/ethanol (2:1) at room temperature. Elemental analysis Anal. calcd. for C₁₆H₁₅ClN₂O₂: C, 63.47%; H, 4.99%; N, 9.25%; Found: C, 63.52%; H, 4.93%; N, 9.21%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Transition metal complexes with oxime-type ligands play important roles in present research [7, 8]. And they have better coordination abilities with metal ions. In addition, this kind of complex has potential application on photophysical properties materials [9–12] and supramolecular architectures [13–17] and so on. So we pay more attention to their synthesis [18–20].

The title oxime-type compound was determined by X-ray crystallography. All bond lengths are in normal ranges. In the crystal structure, there is a strong intramolecular O1–H1A···N1 hydrogen bond interaction involving the O1 and N1 atoms ($d(\text{O1}–\text{H1A}) = 0.82$, $d(\text{H1A} \cdots \text{N1}) = 1.88$, $d(\text{O1} \cdots \text{N1}) = 2.603$, $\text{O1}–\text{H1A} \cdots \text{N1} = 147.02^\circ$).

References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2012).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: A complete structure solution, refinement and analysis program. *J. Appl. Cryst.* **42** (2009) 339–341.
4. Zhao, L.; Zhao, J. X.; An, Q. Q.; Wang, F.: Crystal structure of (E)-1-(4-(((E)-3,5-dibromo-2-hydroxybenzylidene)amino)phenyl)ethan-1-one O-methyl oxime C₁₇H₁₆Br₂N₂O₂. *Z. Kristallogr. NCS* **231** (2016) 1053–1054.
5. Zhao, L.; Ng, S. W.: 1-(4-(((E)-4-Methylbenzylidene)amino)phenyl)ethanone oxime. *Acta Crystallogr. E* **66** (2010) o2473.
6. Zhao, L.; Dong, X. T.; Zhang, S. T.; Chen, Q.: Crystal structure of 1-(4-(((E)-5-bromo-2-hydroxybenzylidene)amino)phenyl)ethanone oxime, C₁₅H₁₃BrN₂O₂. *Z. Kristallogr. NCS* **227** (2012) 101–102.
7. Sun, Y. X.; Li, C. Y.; Yang, C. J.; Zhao, Y. Y.; Guo, J. Q.; Yu, B.: Two Cu(II) complexes with Schiff base ligands: synthesis, crystal structure, spectroscopic properties, and substituent effect. *Chin. J. Inorg. Chem.* **32** (2016) 327–335.
8. Dong, W. K.; Gong, S. S.; Tong, J. F.; Sun, Y. X.; Wu, J. C.: Syntheses and structure of two copper(II) complexes with salicyl mono-oxime ligands. *Chinese J. Inorg. Chem.* **26** (2010) 1868–1874.
9. Zhao, L.; Guo, H. X.; Sun, Y. X.; Meng, W. S.; Yang, C. J.; Zhang, X. Y.: Synthesis and supramolecular structure of 1-(4-(((E)-3,5-dibromo-2-hydroxy benzylidene)amino)phenyl)ethanone O-benzylloxime. *Asian J. Chem.* **25** (2013) 6366–6368.
10. Sun, Y. X.; Li, C. Y.; Yang, C. J.; Zhao, Y. Y.; Guo, J. Q.; Yu, B.: Two Cu(II) complexes with Schiff base ligands: Synthesis, crystal structure, spectroscopic properties, and substituent effect. *Chinese J. Inorg. Chem.* **32** (2016) 327–335.
11. Chen, L.; Dong, W. K.; Zhang, H.; Zhang, Y.; Sun, Y. X.: Structural variation and luminescence properties of tri- and dinuclear Cu^{II} and Zn^{II} complexes constructed from a naphthalenediol-based bis(Salamo)-type ligand. *Cryst. Growth Des.* **17** (2017) 3636–3648.
12. Ma, J. C.; Dong, X. Y.; Dong, W. K.; Zhang, Y.; Zhu, L. C.; Zhang, J. T.: An unexpected dinuclear Cu(II) complex with a bis(Salamo) chelating ligand: Synthesis, crystal structure, and photophysical properties. *J. Coord. Chem.* **69** (2016) 149–159.
13. Zhao, L.; Dong, X. T.; Sun, Y. X.; Cheng, Q.; Dong, X. Y.; Wang, L.: Synthesis, crystal structure and thermal property of a 1D chain-like copper(II) complex. *Chinese J. Inorg. Chem.* **28** (2012) 2413–2418.
14. Wang, L.; Wu, J. J.; Zhang, Y.; Li, G.; Li, X.: Synthesis and crystal structure of 3,3'-dimethoxy-1,1'-[butane-1,4-diylidioxybis(nitrilomethylidyne)] dibenzene. *Asian J. Chem.* **25** (2013) 4115–4116.
15. Sun, Y. X.; Zhao, Y. Y.; Li, C. Y.; Yu, B.; Guo, J. Q.; Li, J.: Supramolecular cobalt(II) and copper(II) complexes with Schiff base ligand: Syntheses characterizations and crystal structures. *Chin. J. Inorg. Chem.* **32** (2016) 913–920.
16. Zhao, L.; Wang, L.; Sun, Y. X.; Dong, W. K.; Tang, L.; Gao, X. H.: A supramolecular copper(II) complex bearing Salen-type bisoxime ligand: Synthesis, structural characterization, and thermal property. *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.* **42** (2012) 1303–1308.
17. Sun, Y. X.; Lu, R. E.; Li, X. R.; Zhao, Y. Y.; Li, C. Y.: A Schiff base ligand containing oxime group and its Cu(II) complex: Syntheses and supramolecular structures. *Chin. J. Inorg. Chem.* **31** (2015) 1055–1062.
18. Zhao, J. X.; Zhao, L.; Li, P. P.; Wang, F.; An, Q. Q.: Crystal structure of (E)-1-(4-(((E)-5-bromo-2-hydroxybenzylidene)amino)phenyl)ethan-1-one O-methyl oxime, C₁₆H₁₅BrN₂O₂. *Z. Kristallogr. NCS* **232** (2017) 731–732.
19. Zhao, L.; An, Q. Q.; Zhao, J. X.; Li, P. P.; Wang, F.: Crystal structure of bis(2-(((E)-((4-((E)-1-(ethoxyimino)ethyl)phenyl)imino)methyl)-5-methoxyphenolato-κ²O,M)copper(II), C₃₆H₃₈CuN₄O₆. *Z. Kristallogr. NCS* **232** (2017) 595–597.
20. Zhao, L.; Wang, F.; An, Q. Q.; Zhao, J. X.: Crystal structure of (E)-1-(4-(((E)-3,5-dichloro-2-hydroxybenzylidene)amino)phenyl)ethan-1-one O-ethyl oxime, C₁₇H₁₆Cl₂N₂O₂. *Z. Kristallogr. NCS* **231** (2016) 1045–1046.