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

Crystal structure of (*E*)-1-(4-(((*E*)-2-hydroxy-3-methoxybenzylidene)amino)phenyl)ethan-1-one oxime, $C_{16}H_{16}N_2O_3$

<https://doi.org/10.1515/ncrs-2017-0385>

Received February 17, 2018; accepted May 23, 2018; available online June 15, 2018

Abstract

$C_{16}H_{16}N_2O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 12.929(13)$ Å, $b = 11.966(12)$ Å, $c = 9.742(9)$ Å, $\beta = 107.449(19)^\circ$, $Z = 4$, $V = 1438(2)$ Å³, $R_{gt}(F) = 0.0515$, $wR_{ref}(F^2) = 0.1767$, $T = 296(2)$ K.

CCDC no.: 1844851

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

Source of material

All chemicals were used without further purification. 1-(4-Aminophenyl)ethanone oxime was prepared by a method reported earlier [1, 2]. The solution of 2-hydroxy-3-methoxy benzaldehyde (304.2 mg, 2 mmol) in ethanol (5 mL) was slowly added to the solution of 4-amino-acetophenone oxime (301.1 mg, 2 mmol) in ethanol (5 mL). The solution had been stirred at 328 K for 15 h. The mixture was cooled to room temperature and filtered. The product was dried under vacuum and a red solid of title compound was obtained (yield 87.2%,

Table 1: Data collection and handling.

Crystal:	Red block
Size:	$0.27 \times 0.25 \times 0.22$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$\theta_{\max}$, completeness:	25.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5368, 2506, 0.029
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1776
$N(\text{param})_{\text{refined}}$:	194
Programs:	Bruker programs [12], SHELX [13]

m.p. 390–396 K). Crystals were obtained by slow evaporation from chloroform/ethanol (2:3) solution in a quiet environment at room temperature. Elemental analysis: Anal. calcd. for $C_{16}H_{16}N_2O_3$: C, 67.59%; H, 5.67%; N, 9.85%; Found: C, 67.61%; H, 5.62%; N, 9.81%.

Experimental details

Molecular structure was refined using the SHELX-14/7 package. Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

The structures of many complexes with oxime-type ligands have been reported [3, 4]. And we believe that the oxime-type ligands and their complexes opens up a new route for the development of new properties [5, 6]. At the same time the interaction of transition metal ions with oxime-type ligands have gained much more attention due to their great importance in the design of their extensive applications fields [7, 8]. For example: it has been used to nonlinear optical materials field [9] and supramolecular architectures [10] etc. Recently, we have focused our interests on the oxime-type complexes [11].

The single crystal structure of the title compound was determined by X-ray crystallographic methods all bond lengths are in normal ranges. There is one intramolecular $O2-H2 \cdots N1$ hydrogen bond ($d(O2 \cdots N1) = 2.619$ Å,

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

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

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}
C1	0.16893(19)	0.1185(2)	−0.1412(2)	0.0621(7)
H1A	0.1096	0.1581	−0.1237	0.093 [*]
H1B	0.1477	0.0427	−0.1674	0.093 [*]
H1C	0.1887	0.1541	−0.2181	0.093 [*]
C2	0.29219(16)	0.22255(19)	0.0454(2)	0.0433(6)
C3	0.25064(18)	0.3238(2)	−0.0134(2)	0.0510(6)
H3A	0.1961	0.3258	−0.1010	0.061 [*]
C4	0.28995(18)	0.4233(2)	0.0578(3)	0.0572(7)
H4	0.2614	0.4912	0.0171	0.069 [*]
C5	0.37000(17)	0.4221(2)	0.1868(3)	0.0516(6)
H5	0.3953	0.4890	0.2334	0.062 [*]
C6	0.41391(15)	0.32053(18)	0.2488(2)	0.0398(5)
C7	0.37559(16)	0.22008(18)	0.1770(2)	0.0403(5)
C8	0.49905(17)	0.32059(19)	0.3860(2)	0.0444(5)
H8	0.5219	0.3885	0.4315	0.053 [*]
C9	0.62933(15)	0.23146(18)	0.5774(2)	0.0384(5)
C10	0.64999(16)	0.13241(17)	0.6564(2)	0.0410(5)
H10	0.6057	0.0707	0.6239	0.049 [*]
C11	0.73474(16)	0.12439(17)	0.7818(2)	0.0410(5)
H11	0.7470	0.0576	0.8329	0.049 [*]
C12	0.80212(15)	0.21580(18)	0.8323(2)	0.0395(5)
C13	0.78036(18)	0.31539(19)	0.7548(2)	0.0483(6)
H13	0.8236	0.3776	0.7888	0.058 [*]
C14	0.69560(18)	0.32356(19)	0.6282(2)	0.0472(6)
H14	0.6830	0.3904	0.5773	0.057 [*]
C15	0.89851(16)	0.2071(2)	0.9624(2)	0.0456(6)
C16	0.9427(2)	0.3090(2)	1.0525(3)	0.0633(7)
H16A	0.9655	0.2887	1.1525	0.095 [*]
H16B	0.8872	0.3651	1.0360	0.095 [*]
H16C	1.0034	0.3379	1.0264	0.095 [*]
N1	0.54369(13)	0.23024(15)	0.44645(17)	0.0422(5)
N2	0.94108(15)	0.10979(17)	0.98800(18)	0.0518(6)
O1	0.25934(12)	0.11947(13)	−0.01420(16)	0.0570(5)
O2	0.41605(13)	0.11876(12)	0.23013(16)	0.0547(5)
H2	0.4618	0.1271	0.3086	0.082 [*]
O3	1.03123(13)	0.10724(16)	1.11399(17)	0.0674(6)
H3	1.0612	0.0462	1.1206	0.101 [*]

$d(\text{O2} \cdots \text{H2}) = 0.82 \text{ \AA}$ and $d(\text{H2} \cdots \text{N1}) = 1.896 \text{ \AA}$ in the title compound.

Acknowledgements: This work was supported by the Science Foundation of Gansu Education Department of China (2017A–023).

References

1. 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.
2. 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.
3. Li, P. P.; Zhao, L.; Zhao, J. X.; Zhu, Z. B.; Wang, F.; An, Q. Q.: Synthesis and crystal structure of bis{1-(((4-(1-(hydroxyimino)ethyl)phenyl)imino)methyl)naphthalene-2-olato-κ²O,N}}copper(II), C₃₈H₃₀CuN₄O₄. Z. Kristallogr. NCS **232** (2017) 889–890.
4. Zhao, J. X.; Zhao, L.; Li, P. P.; Zhu, Z. B.: Crystal structure of bis{5-methoxy-2-(((*E*)-((4-((*E*)-1-(methoxyimino)ethyl)phenyl)imino)methyl)phenolato-κ²N,O)}copper(II), C₃₄H₃₄CuN₄O₆. Z. Kristallogr. NCS **232** (2017) 863–865.
5. 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.
6. 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.
7. 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. Chin. J. Inorg. Chem. **28** (2012) 2413–2418.
8. Zhao, L.; Dong, X. T.; Cheng, Q.; Zhao, J. X.; Wang, L.: Synthesis, crystal structure and spectral properties of a 2D supramolecular copper(II) complex with 1-(4-(((*E*)-3-ethoxyl-2-hydroxybenzylidene)amino)phenyl)ethanone oxime. Synth. React. Inorg. Met.-Org. Nano-Met. Chem. **43** (2013) 1241–1246.
9. 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.
10. 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.
11. Zhao, J. X.; Zhao, L.; Liu, G. H.; Li, P. P.; Zhu, Z. B.: Crystal structure of 1-(4-[(5-chloro-2-hydroxybenzylidene)amino]phenyl)ethanone *O*-ethyl oxime, C₁₇H₁₇ClN₂O₂. Z. Kristallogr. NCS **232** (2017) 999–1001.
12. Bruker. SAINT, APEX2 and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
13. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.