Tao Fu, Jin-Xia Ma, Qing-Lin Li, Ping Wei, Ji-Xing Zhao and Li Zhao*

Crystal structure of (*E*)-1-(4-(((*E*)-3,5-dichloro-2-hydroxybenzylidene)amino)phenyl)ethan-1-one *O*-methyl oxime, C₁₆H₁₄Cl₂N₂O₂

<https://doi.org/10.1515/ncrs-2017-0405>

Received July 19, 2018; accepted August 3, 2018; available online August 20, 2018

Abstract

C₁₆H₁₄Cl₂N₂O₂, monoclinic, *P*₂/c (no. 14), *a* = 7.9157(6) Å, *b* = 13.4533(8) Å, *c* = 14.8459(10) Å, β = 103.651(2)°, *V* = 1536.31(18) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0410, *wR*_{ref}(*F*²) = 0.1162, *T* = 296(2) K.

CCDC no.: 1860089

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 solvents and other reagents were of analytical grade and the title compound was prepared by modification of the reported method [4, 5]. 3,5-Dichlorosalicylaldehyde (191.1 mg, 1 mmol) dissolved in ethanol solution (5 mL) was added to 1-(4-aminophenyl)ethanone *O*-methyl oxime (164.2 mg, 1 mmol) dissolved in ethanol solution (5 mL). The solution had been stirred at 328 K for 10 h, and the mixture was filtered at room temperature. Some yellow solid was obtained (yield 66.3%, m.p. 377–384 K). Crystals were obtained by slow evaporation in a quiet environment at room temperature. Anal. Calcd. for C₁₆H₁₄Cl₂N₂O₂: C, 56.99%; H, 4.18%; N, 8.31%; Found: C, 56.87%; H, 4.25%; N, 8.27%.

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

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

Table 1: Crystal collection and handling.

Crystal:	Block, red
Size:	0.22 × 0.18 × 0.17 mm
Wavelength:	Mo <i>K</i> radiation (λ = 0.71073 Å)
μ:	0.43 mm ^{−1}
Diffractometer, scan mode:	Bruker SMART CCD, φ and ω-scan
2θ _{max} , completeness:	52.99°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	11991, 3168, 0.0627
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2σ(<i>I</i> _{obs}), 2757
<i>N</i> (<i>param</i>) _{refined} :	202
Programs:	Bruker [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	0.24332(7)	0.22160(3)	0.14590(3)	0.03503(16)
Cl2	0.14421(7)	−0.16966(3)	0.07978(3)	0.03444(16)
O1	0.49137(19)	0.15795(8)	0.31249(9)	0.0287(3)
H1	0.561644	0.136953	0.358252	0.043
O2	1.32073(19)	0.02804(10)	0.89959(9)	0.0337(3)
N1	0.6488(2)	0.02689(10)	0.43001(9)	0.0230(3)
N2	1.1974(2)	0.04531(12)	0.81615(10)	0.0287(4)
C1	0.4138(2)	0.08144(11)	0.26176(11)	0.0207(3)
C2	0.2930(2)	0.09937(12)	0.17812(11)	0.0225(4)
C3	0.2099(2)	0.02362(12)	0.12210(11)	0.0231(4)
H3	0.129761	0.037462	0.066820	0.028
C4	0.2489(2)	−0.07352(12)	0.15030(11)	0.0235(4)
C5	0.3653(2)	−0.09510(12)	0.23222(11)	0.0240(4)
H5	0.388583	−0.160929	0.249979	0.029
C6	0.4491(2)	−0.01814(12)	0.28910(11)	0.0214(4)
C7	0.5685(2)	−0.04118(12)	0.37640(11)	0.0239(4)
H7	0.587403	−0.107330	0.394091	0.029
C8	0.7659(2)	0.00524(12)	0.51566(11)	0.0223(4)
C9	0.8022(3)	−0.08943(13)	0.55294(12)	0.0310(4)
H9	0.746378	−0.144315	0.521144	0.037
C10	0.9209(3)	−0.10260(13)	0.63699(12)	0.0299(4)
H10	0.943869	−0.166495	0.660637	0.036
C11	1.0063(2)	−0.02266(13)	0.68669(11)	0.0234(4)
C12	0.9653(3)	0.07256(13)	0.64981(12)	0.0302(4)
H12	1.017812	0.127764	0.682527	0.036
C13	0.8484(3)	0.08568(13)	0.56588(12)	0.0291(4)
H13	0.824381	0.149581	0.542454	0.035
C14	1.1361(2)	−0.03553(13)	0.77589(11)	0.0246(4)
C15	1.1900(3)	−0.13686(15)	0.81399(13)	0.0342(4)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H15A	1.300134	−0.132507	0.858026	0.051
H15B	1.200152	−0.180422	0.764317	0.051
H15C	1.104296	−0.162554	0.844025	0.051
C16	1.3795(3)	0.12249(15)	0.93907(13)	0.0366(5)
H16A	1.424305	0.160614	0.895228	0.055
H16B	1.469484	0.112788	0.994317	0.055
H16C	1.284071	0.157453	0.954152	0.055

Experimental details

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

Discussion

Oximes are compounds which carry a carbon-nitrogen double bond formed by the reactions of aldehydes or ketones with hydroxylamines [6–9]. Various properties of oxime-type compounds along with their applications in analytical chemistry increased interest [10, 11]. As so far, such oxime-type compounds are used as analytical reagents for the detection and determination of some metal ions [12, 13]. Further more some compounds represent nonlinear optical materials and others show supramolecular architectures [14–17].

The asymmetric unit (*cf.* the figure) is built up by C₁₆H₁₄Cl₂N₂O₂ molecules. The crystal structure of the title compound was determined by X-ray crystallography. All bond lengths are in normal ranges. There is a strong intramolecular O1–H1···N1 hydrogen bond interaction (*d*(N1···H1) = 1.86 Å, *d*(O1–H1) = 0.82 Å and *d*(O1···N1) = 2.584 Å) in the title compound.

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. Crystallogr.* **42** (2009) 339–341.
4. Ma, J. X.; Li, Q. L.; Li, P. P.; Zhao, J. X.; Zhao, L.: Synthesis and crystal structure of (E)-1-(4-(((E)-5-chloro-2-hydroxybenzylidene)amino)phenyl)ethan-1-one O-methyl oxime, C₁₆H₁₅ClN₂O₂. *Z. Kristallogr. NCS* **233** (2018) 463–464.
5. 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.
6. Li, P. P.; Zhao, L.; Liu, G. H.; Zhao, J. X.; Zhu, Z. B.: Synthesis and crystal structure of 1-{4-[(2-hydroxy-benzylidene)amino]phenyl}ethanone oxime, C₁₅H₁₄N₂O₂. *Z. Kristallogr. NCS* **232** (2017) 1005–1006.
7. Li, Q. L.; Li, P. P.; Ma, J. X.; Zhao, J. X.; Zhao, L.: Crystal structure of bis{2-((E)-((4-((E)-1-(methoxyimino)ethyl)phenyl)imino)methyl)phenolato-κ²N,O}zinc(II), C₃₂H₃₀N₄O₄Zn. *Z. Kristallogr. NCS* **233** (2018) 637–639.
8. 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,N)copper(II), C₃₆H₃₈CuN₄O₆. *Z. Kristallogr. NCS* **232** (2016) 595–597.
9. Li, P. P.; Ma, J. X.; Li, Q. L.; Zhao, J. X.; Zhao, L.: Crystal structure of (E)-1-(4-(((E)-2-hydroxy-5-methylbenzylidene)amino)phenyl)ethan-1-one O-methyl oxime, C₁₇H₁₈N₂O₂. *Z. Kristallogr. NCS* **233** (2018) 533–534.
10. 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.
11. 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.
12. Dong, W. K.; Li, X. L.; Wang, L.; Zhang, Y.; Ding, Y. J.: A new application of Salamo-type bisoximes: as a relay-sensor for Zn²⁺/Cu²⁺ and its novel complexes for successive sensing of H⁺/OH[−]. *Sens. Actuators B* **229** (2016) 370–378.
13. Dong, W. K.; Sunday, F. A.; Zhang, Y.; Sun, Y. X.; Dong, X. Y.: A reversible “turn-on” fluorescent sensor for selective detection of Zn²⁺. *Sens. Actuators B* **238** (2017) 723–734.
14. 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.
15. 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.
16. 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.
17. 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.