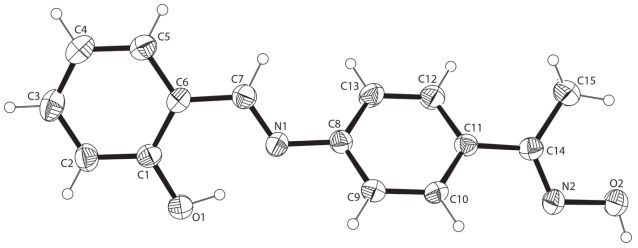
Peng-Peng Li, Li Zhao*, Guo-Hua Liu, Ji-Xing Zhao and Zhao-Bin Zhu

Synthesis and crystal structure of 1-{4-[(2-hydroxy-benzylidene)amino]phenyl}ethanone oxime, $C_{15}H_{14}N_2O_2$



https://doi.org/10.1515/ncrs-2017-0134
Received May 1, 2017; accepted September 29, 2017; available online October 16, 2017

Abstract

$C_{15}H_{14}N_2O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 13.5809(12)$ Å, $b = 9.7794(14)$ Å, $c = 9.8883(9)$ Å, $\beta = 97.972(9)°$, $Z = 4$, $V = 1300.6(3)$ Å^3, $R_{gt}(F) = 0.0635$, $wR_{ref}(F^2) = 0.1758$, $T = 291(2)$ K.

CCDC no.: 1577291

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.

Table 1: Data collection and handling.

Crystal:	Block, clear light orange
Size:	0.21 × 0.17 × 0.14 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω-scans
$2\theta_{max}$, completeness:	52°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4966, 2551, 0.042
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1248
$N(param)_{refined}$:	178
Programs:	CrysAlis^{PRO} [1], SHELX [2], OLEX2 [3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	−0.60627(16)	0.0413(3)	0.0040(2)	0.0615(7)
H1	−0.562(2)	0.052(4)	0.082(3)	0.081(12)*
O2	0.00342(14)	0.0658(3)	0.6523(2)	0.0658(7)
H2	0.0486	0.0370	0.6130	0.099*
N1	−0.52562(17)	0.0860(3)	0.2524(2)	0.0502(7)
N2	−0.08717(18)	0.0587(3)	0.5634(2)	0.0543(7)
C1	−0.6937(2)	0.0739(3)	0.0458(3)	0.0478(8)
C2	−0.7794(2)	0.0669(4)	−0.0466(3)	0.0599(10)
H2A	−0.7760	0.0392	−0.1358	0.072*
C3	−0.8700(2)	0.1003(4)	−0.0082(3)	0.0681(11)
H3	−0.9273	0.0940	−0.0714	0.082*
C4	−0.8769(2)	0.1432(4)	0.1233(4)	0.0680(10)
H4	−0.9381	0.1663	0.1488	0.082*
C5	−0.7913(2)	0.1512(3)	0.2163(3)	0.0574(9)
H5	−0.7955	0.1808	0.3048	0.069*
C6	−0.6988(2)	0.1160(3)	0.1807(3)	0.0466(8)
C7	−0.6110(2)	0.1218(3)	0.2818(3)	0.0505(8)
H7	−0.6166	0.1519	0.3696	0.061*
C8	−0.4381(2)	0.0941(3)	0.3487(3)	0.0466(8)
C9	−0.3655(2)	−0.0028(3)	0.3420(3)	0.0522(9)
H9	−0.3766	−0.0732	0.2785	0.063*
C10	−0.2764(2)	0.0031(3)	0.4280(3)	0.0497(8)
H10	−0.2290	−0.0648	0.4237	0.060*
C11	−0.2565(2)	0.1109(3)	0.5223(3)	0.0451(8)
C12	−0.3308(2)	0.2057(4)	0.5288(3)	0.0592(9)
H12	−0.3205	0.2760	0.5925	0.071*
C13	−0.4200(2)	0.1990(4)	0.4433(3)	0.0601(9)
H13	−0.4683	0.2652	0.4491	0.072*
C14	−0.1591(2)	0.1206(3)	0.6089(3)	0.0489(8)
C15	−0.1479(2)	0.1989(4)	0.7415(3)	0.0672(10)
H15A	−0.0912	0.1652	0.8009	0.101*
H15B	−0.2066	0.1872	0.7843	0.101*
H15C	−0.1388	0.2942	0.7235	0.101*

Source of material

The title compound was prepared by modification of the reported method [4–6]. The solution of salicylaldehyde (122 mg, 1 mmol) in ethanol (10 mL) was slowly added to the solution of 4-amino-acetophenone oxime (150.17 mg, 1 mmol) in ethanol (10 mL). The solution had been stirred at 328 K for 9h. The mixture was cooled down to room temperature and filtered. The residue was washed successively with ethanol and

***Corresponding author: Li Zhao,** School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, People's Republic of China, e-mail: zhaoli_72@163.com
Peng-Peng Li, Guo-Hua Liu, Ji-Xing Zhao and Zhao-Bin Zhu: School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, People's Republic of China

n-hexane, respectively. The product was dried under vacuum and obtained orange solid (yield 76.5%, m.p. 413–415 K). The single crystals were obtained by slow evaporation from a mixture of chloroform/ethanol (2:1) solution at room temperature. Elemental analysis – Anal. Calcd. for $C_{15}H_{14}N_2O_2$: C, 70.85%; H, 5.55%; N, 11.02%; Found: C, 70.87%; H, 5.57%; N, 10.98%.

Experimental details

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

Discussion

The term "oxime" is formed by the combination of the words oxy and imine, and oximes are amphoteric compounds: weak acid due to hydroxyl group (OH) and weak base due to the azomethine group (C=N) [7–9]. Oxime-type compounds can form stable complexes with transition metal ions [10–12]. Particular attention has been paid to the synthesis and application of oxime-type compounds and their complexes. For example: it has been used in the chemistry of materials [13–16], and also been used to supramolecular architectures [17–19] etc.

The title compound, built up by the $C_{15}H_{14}N_2O_2$ molecules, has been synthesized. The single crystal structure verifies that all bond lengths are in normal ranges. In the title compound, there is a strong intramolecular O1—H1···N1 hydrogen bond [d(N1···H1) = 1.721 Å, d(O1—H1) = 0.92(3) Å, d(O1···N1) = 2.584 Å]; *cf*. the figure.

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

References

1. Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.37.35, Agilent Technologies UK Ltd, Oxford, UK (2011).
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (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.; 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, $C_{17}H_{16}Cl_2N_2O_2$. Z. Kristallogr. NCS **231** (2016) 1045–1046.
5. Zhao, L.; Dong, X. T.; Zhang, S. T.; Chen, Q.: Crystal struc ture of 1-(4-{[(*E*)-5-bromo-2-hydroxybenzylidene] amino}phenyl)ethanone oxime, $C_{15}H_{13}BrN_2O_2$. Z. Kristallogr. NCS **227** (2012) 101–102.
6. 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 $C_{17}H_{16}Br_2N_2O_2$. Z. Kristallogr. NCS **231** (2016) 1053–1054.
7. Chakravorty, A.: Structural chemistry of transition metal complexes of oximes. Coord. Chem. Rev. **13** (1974) 1–46.
8. Kurtoglu, M.: Synthesis, complexation, spectral, antibacterial and antifungal activity of 2,4-dihydroxy-5-[(*E*)-phenyldiazenyl]benzaldehyde oxime, J. Serbian Chem. Soc. **75** (2010) 1231–1239.
9. Dong, W. K.; Feng, J. H.; Yang, X. Q.: Crystal structure of 4,4′-dibromo-2,2′-((1,3-propylene)dioxybis(nitrilomethylidyne)) diphenol, $C_{17}H_{16}Br_2N_2O_4$. Z. Kristallogr. **221** (2006) 447–448.
10. 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.
11. 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.
12. 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.
13. 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 Zn^{2+}/Cu^{2+} and its novel complexes for successive sensing of H^+/OH^-. Sens. Actuators B **229** (2016) 370–378.
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. Dong, W. K.; Sunday, F. A.; Zhang, Y.; Sun, Y. X.; Dong, X. Y.: A reversible "turn-on" fluorescent sensor for selective detection of Zn^{2+}. Sens. Actuators B **238** (2017) 723–734.
16. Dong, W. K.; Zhang, X. Y.; Sun, Y. X.; Dong, X. Y.; Li, G.; Wang, J.: A 2D supramolecular copper(II) complex with an asymmetric salamo-type ligand: synthesis, crystal structure, and fluorescent property. Synth. React. Inorg. Met.-Org. Nano-Met. Chem. **45** (2015) 956–962.
17. Razik, B. M. A.; Osman, H.; Basiri, A.; Salhin, A.; Kia, Y.; Ezzat, M. O.; Murugaiyah, V.: Ionic liquid mediated synthesis and molecular docking study of novel aromatic embedded schiff bases as potent cholinesterase inhibitors. Bioorg. Chem. **57** (2014) 162–168.
18. Song, P. S.; Chen, X. T.; Xiang, Y.; Huang, L.; Zhou, Z. J.; Wei, R. R.; Tong, A. J.: A ratiometric fluorescent pH probe based on aggregation-induced emission enhancement and its application in live-cell imaging. J. Mater. Chem. **21** (2011) 13470–13475.
19. Zaltariov, M. F.; Cazacu, M.; Vornicu, N.; Shova, S.; Racles, C.; Balan, M.; Turta, C.: A new diamine containing disiloxane moiety and some derived Schiff bases: synthesis, structural characterisation and antimicrobial activity. Supramol. Chem. **25** (2013) 490–502.