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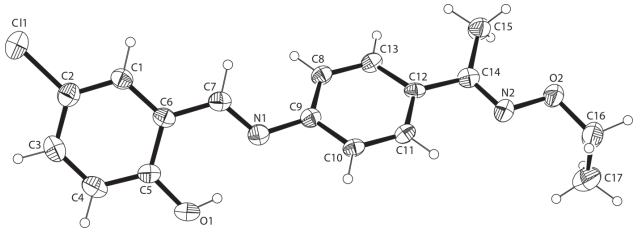
Crystal structure of 1-{4-[(5-Chloro-2-hydroxybenzylidene)amino]phenyl} ethanone O-ethyloxime, $C_{17}H_{17}ClN_2O_2$



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Abstract

$C_{17}H_{17}ClN_2O_2$, monoclinic, *Pc* (no. 7), $a = 17.789(2)$ Å, $b = 7.1127(7)$ Å, $c = 6.2885(6)$ Å, $\beta = 97.513(10)°$, $Z = 2$, $V = 788.83(15)$ Å^3, $R_{gt}(F) = 0.0600$, $wR_{ref}(F^2) = 0.1651$, $T = 290.83(10)$ K.

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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

The title compound was prepared by modification of the reported method [4–6]. To an ethanol solution (8 mL) of 5-chlorosalicylaldehyde (172.3 mg, 1.1 mmol) was added an ethanol solution (6 mL) of 4-aminophenylethanone ethoxy oxime (316.6 mg, 1 mmol). After the solution had been stirred at 328 K for 14 h, the mixture was filtered. The residue was washed successively with ethanol and n-hexane, respectively. The product was dried under vacuum and a yellow solid was obtained (yield 70.1%, m.p. 419–425 K). The single crystals were obtained by slow evaporation from chloroform/ethanol

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Table 1: Data collection and handling.

Crystal:	Block, clear light brown
Size:	0.21 × 0.17 × 0.14 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.25 mm^{-1}
Diffractometer, scan mode:	SuperNova ω-scans
$2\theta_{max}$, completeness:	52°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	2947, 2300, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1661
$N(param)_{refined}$:	202
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}
Cl1	−0.82711(13)	−0.1839(4)	−0.7205(3)	0.0864(8)
O1	−0.5622(3)	−0.3175(9)	−0.0823(7)	0.0669(15)
H1	−0.5231	−0.2913	−0.1319	0.100*
O2	−0.0555(3)	−0.2137(8)	−0.5366(8)	0.0654(14)
N1	−0.4772(3)	−0.2358(7)	−0.3776(8)	0.0450(14)
N2	−0.1278(3)	−0.2071(9)	−0.4723(9)	0.0570(16)
C1	−0.6781(4)	−0.1910(9)	−0.5906(11)	0.0467(16)
H1A	−0.6734	−0.1505	−0.7288	0.056*
C2	−0.7477(4)	−0.2185(11)	−0.5323(11)	0.0531(18)
C3	−0.7571(5)	−0.2837(12)	−0.3302(13)	0.062(2)
H3	−0.8054	−0.3063	−0.2950	0.075*
C4	−0.6952(5)	−0.3146(11)	−0.1832(11)	0.059(2)
H4	−0.7020	−0.3561	−0.0468	0.070*
C5	−0.6224(4)	−0.2858(10)	−0.2316(10)	0.0491(17)
C6	−0.6126(4)	−0.2232(9)	−0.4433(10)	0.0427(15)
C7	−0.5382(4)	−0.2083(10)	−0.5060(10)	0.0466(16)
H7	−0.5340	−0.1770	−0.6476	0.056*
C8	−0.3912(4)	−0.3172(9)	−0.6404(9)	0.0442(16)
H8	−0.4312	−0.3671	−0.7332	0.053*
C9	−0.4056(4)	−0.2380(9)	−0.4472(10)	0.0418(15)
C10	−0.3444(3)	−0.1689(9)	−0.3122(9)	0.0415(15)
H10	−0.3520	−0.1200	−0.1795	0.050*
C11	−0.2730(4)	−0.1707(9)	−0.3691(9)	0.0439(15)
H11	−0.2334	−0.1187	−0.2766	0.053*
C12	−0.2578(3)	−0.2491(8)	−0.5636(8)	0.0378(16)
C13	−0.3194(4)	−0.3232(9)	−0.6967(11)	0.0434(15)
H13	−0.3117	−0.3778	−0.8264	0.052*
C14	−0.1801(4)	−0.2528(8)	−0.6189(11)	0.0443(16)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C15	−0.1650(4)	−0.3049(12)	−0.8397(11)	0.062(2)
H15A	−0.2122	−0.3251	−0.9295	0.074*
H15B	−0.1353	−0.4179	−0.8333	0.074*
H15C	−0.1377	−0.2050	−0.8981	0.074*
C16	0.0007(4)	−0.1607(13)	−0.3611(13)	0.072(2)
H16A	−0.0172	−0.0526	−0.2882	0.086*
H16B	0.0470	−0.1248	−0.4165	0.086*
C17	0.0168(6)	−0.3156(15)	−0.2075(16)	0.096(3)
H17A	0.0247	−0.4294	−0.2836	0.144*
H17B	−0.0253	−0.3317	−0.1280	0.144*
H17C	0.0616	−0.2869	−0.1104	0.144*

(3:1) at room temperature. Elemantal analysis. Anal. calcd. For $C_{17}H_{17}ClN_2O_2$: C, 64.60%; H, 5.45%; N, 8.78%; Found: C, 64.62%; H, 5.46%; N, 8.75%.

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. The oxime ligands form stable complexes with transition metals through the nitrogen and oxygen atoms [7–10]. There is an interest on such complexes, for their applications [10–13]. For example: they have been used to identify metal ions [14, 15], have been used in the field of fluorescence [16, 17] and supramolecular architectures [18] etc.

The crystal structure of the title compound is only built up by the $C_{17}H_{17}ClN_2O_2$ molecules. The bond lengths and angles are in normal ranges. In the title compound, there is a strong intramolecular O1—H1···N1 hydrogen bond [d(N1···H1) = 1.881 Å, d(O1—H1) = 0.820 Å, d(O1···N1) = 2.609 Å].

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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.; Dong, X. T.; Zhang, S. T.; Chen, Q.: Crystal structure 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.
5. 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.
6. Zhao, L.; Dong, X. T.; Zhang, J.; Cheng, Q.; Zhao, J. X.: Crystal structure of 1-{4-[(*E*)-2,4-ichlorobenzylideneamino] phenyl}ethanone oxime, $C_{15}H_{12}Cl_2N_2O$. Z. Kristallogr. NCS **227** (2012) 185–186.
7. 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.
8. Nomura, M.; Nakamura, T.; Kashimura, Y.; Sugiyama, T.; Kajitani, M.: Syntheses and structural diversity of salicylideneaniline derivatives with cobaltadithiolene backbone. J. Mol. Struct. **1008** (2012) 77–82.
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. Dong, W. K.; Zhang, J. T.; Dong, Y. J.; Zhang, Y.; Wang, Z. K.: Construction of mononuclear copper(II) and trinuclear cobalt(II) complexes based on asymmetric Salamo-type ligands. Z. Anorg. Allg. Chem. **642** (2016) 189–196.
11. 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.
12. 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.
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. 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.
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. 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.

17. 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.

18. 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.