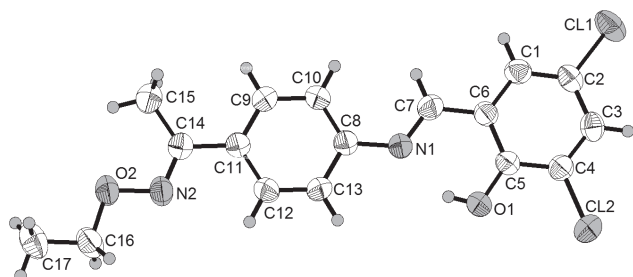


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Crystal structure of (*E*)-1-(4-(((*E*)-3,5-dichloro-2-hydroxybenzylidene)amino)phenyl)ethan-1-one O-ethyl oxime, C₁₇H₁₆Cl₂N₂O₂



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Abstract

C₁₇H₁₆Cl₂N₂O₂, monoclinic, *P*₂/c (no. 14), *a* = 9.1238(5) Å, *b* = 13.0575(4) Å, *c* = 14.3740(5) Å, β = 104.616(4)°, *V* = 1657.02(11) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0460, *wR*_{ref}(*F*²) = 0.1120, *T* = 293(2) K.

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

Source of material

To an ethanol solution (5 mL) of 4-amino acetophenone ethoxy oxime (178.0 mg, 1 mmol) was added an ethanol solution (5 mL) of 3,5-dichlorosalicyde (279.9 mg, 1 mmol). After the solution had been stirred at 328 K for 10 h, the mixture was filtered, washed successively with ethanol and ethanol/hexane (1:4), respectively. The isolated compound was dried under reduced pressure and purified with recrystallization from ethanol solution to yield 300.7 mg of red

Table 1: Data collection and handling.

Crystal:	orange blocks
Wavelength:	size 0.21 × 0.15 × 0.12 mm
μ:	Mo K _α radiation (0.71073 Å)
Diffractometer, scan mode:	4.0 cm ^{−1}
2θ _{max} , completeness:	SuperNova, Eos diffractometer, ω
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	55.5°, >99%
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	7227, 3254, 0.029
<i>N</i> (<i>param</i>) _{refined} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2253
Programs:	211
	SHELX [11], CrysAlis ^{PRO} [12]

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	0.67356(9)	1.17732(5)	0.06238(5)	0.0708(3)
Cl2	0.67331(8)	0.78706(5)	0.18033(5)	0.0638(2)
O1	0.9414(2)	0.84690(12)	0.32597(12)	0.0565(5)
H1	1.0102	0.8683	0.3700	0.085*
O2	1.7720(2)	0.94665(13)	0.88606(12)	0.0619(5)
N1	1.1301(2)	0.97613(15)	0.42158(13)	0.0464(5)
N2	1.6472(2)	0.93650(16)	0.80591(14)	0.0546(6)
C1	0.8820(3)	1.10028(18)	0.21426(17)	0.0480(6)
H1A	0.9248	1.1654	0.2205	0.058*
C2	0.7557(3)	1.08055(19)	0.14210(16)	0.0479(6)
C3	0.6892(3)	0.98502(19)	0.13113(16)	0.0479(6)
H3	0.6020	0.9729	0.0826	0.057*
C4	0.7542(3)	0.90786(18)	0.19333(16)	0.0449(6)
C5	0.8827(3)	0.92437(17)	0.26794(16)	0.0418(6)
C6	0.9470(3)	1.02282(17)	0.27871(16)	0.0426(6)
C7	1.0755(3)	1.04473(19)	0.35885(17)	0.0480(6)
H7	1.1190	1.1096	0.3647	0.058*
C8	1.2518(3)	0.99340(17)	0.50345(16)	0.0427(6)
C9	1.4524(3)	1.09077(19)	0.60495(18)	0.0565(7)
H9	1.5072	1.1515	0.6169	0.068*
C10	1.3353(3)	1.08257(19)	0.52277(18)	0.0564(7)
H10	1.3126	1.1374	0.4803	0.068*
C11	1.4900(3)	1.01102(17)	0.66973(16)	0.0421(6)
C12	1.4046(3)	0.92197(18)	0.64997(17)	0.0475(6)
H12	1.4270	0.8672	0.6926	0.057*
C13	1.2878(3)	0.91325(17)	0.56873(16)	0.0466(6)
H13	1.2320	0.8529	0.5573	0.056*
C14	1.6184(3)	1.01900(19)	0.75661(17)	0.0455(6)

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C15	1.7056(3)	1.1162(2)	0.78211(19)	0.0654(8)
H15A	1.7827	1.1067	0.8408	0.098*
H15B	1.6383	1.1699	0.7906	0.098*
H15C	1.7519	1.1344	0.7314	0.098*
C16	1.7931(3)	0.8503(2)	0.93561(18)	0.0626(8)
H16A	1.7931	0.7950	0.8906	0.075*
H16B	1.7116	0.8385	0.9666	0.075*
C17	1.9421(3)	0.8542(2)	1.0093(2)	0.0728(9)
H17A	2.0226	0.8604	0.9776	0.109*
H17B	1.9555	0.7926	1.0469	0.109*
H17C	1.9436	0.9122	1.0506	0.109*

crystalline solid (yield 85.6%, m.p. 419–421 K). The single crystals were obtained by slow evaporation from a mixture of chloroform/DMSO/methanol solution at room temperature. Elemental analysis: Anal. calcd for C₁₇H₁₆Cl₂N₂O₂: C, 58.13%; H, 4.59%; N, 7.98%; Found: C, 58.12%; H, 4.56%; N, 7.92%.

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with *U*_{iso}(H) set to 1.2*U*_{eq}(C). The H atoms of the methyl group were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density (HFIX 137 in the SHELX program suite [11]), with *U*_{iso}(H) set to 1.5*U*_{eq}(C). The H atom of the hydroxyl group was allowed to rotate with a fixed angle around the C–O bond to best fit the experimental electron density (HFIX 147 [11]), with *U*_{iso}(H) set to 1.5*U*_{eq}(O).

Discussion

Oximes are often synthesized by aldehydes or ketones with hydroxylamine through condensation reaction, with a general formula of R¹R²C=N–OR³. As a potential chelating agent, these compounds excellently coordinates metal atoms [1–2]. Particular attention has recently been paid to the synthesis and application of oximes and their complexes. Some of them or their metal complexes are used in various organic reaction processes as catalysts [3], models of reaction centers of metalloenzymes [4], have magnetic properties [5] and are nonlinear optical materials [6]. They can also be used as models for biomolecules and biological processes [7, 8].

In the crystal structure of the title compound, the title compound is only built up by the C₁₇H₁₆Cl₂N₂O₂ molecules, in which all bond lengths are in normal ranges. The planes

of the two rings are nearly parallel to each other with a dihedral angle of 8.51(3)°. An intramolecular O1–H1···N1 hydrogen bond is present between the hydroxy group and the nitrogen atoms of Schiff base group (*cf.* the figure). The O1–N1 distances between the hydroxyl groups and the nitrogen atoms of Schiff base group are about 2.551(3) Å, indicating a strong hydrogen bond. The results indicate that the Schiff base-OH form is more favorable in the crystalline state (*d*(O1–H1) = 0.820 Å, *d*(H1–N1) = 1.822 Å, *d*(O1–N1) = 2.551(3) Å, O1–H1···N1 = 147.32(4)°) [9, 10].

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