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Crystal structure of (*E*)-2,4-dichloro-6-(((4-methoxy-2-nitrophenyl)imino)methyl)phenol, $C_{14}H_{10}Cl_2N_2O_4$

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Abstract

$C_{14}H_{10}Cl_2N_2O_4$, monoclinic, $P2_1/c$, $a = 14.2599(10)$ Å, $b = 6.9970(4)$ Å, $c = 15.0779(10)$ Å, $\beta = 108.245(2)^\circ$, $V = 1428.79(16)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0355$, $wR_{ref}(F^2) = 0.0860$, $T = 150(2)$ 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

4-Methoxy-2-nitroaniline (1 mmol, 0.168 g) and 3,5-dichlorosalicylaldehyde (1 mmol, 0.191 g) were added to the solution of ethanol (10 mL), then the mixture was refluxed for 4 h. After filtering, the residue was dissolved in dichloromethane and the solution was evaporated slowly at room temperature to obtain colorless prismatic crystals suitable for X-ray structure determination.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.32 \times 0.25 \times 0.14$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.47 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	26.4° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	22119, 2929, 0.058
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2282
$N(\text{param})_{\text{refined}}$:	201
Programs:	Bruker programs [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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	1.41836(15)	0.4060(3)	0.58235(14)	0.0321(5)
H1A	1.402735	0.274375	0.594897	0.048*
H1B	1.385194	0.494935	0.612962	0.048*
H1C	1.489909	0.425800	0.606692	0.048*
C2	1.28649(13)	0.4313(3)	0.43827(13)	0.0190(4)
C3	1.21563(13)	0.3810(3)	0.48005(12)	0.0180(4)
H3	1.233811	0.352960	0.544873	0.022*
C4	1.11790(13)	0.3732(2)	0.42432(12)	0.0147(4)
C5	1.08512(13)	0.4212(2)	0.32948(12)	0.0150(4)
C6	1.15869(13)	0.4740(3)	0.29075(13)	0.0186(4)
H6	1.140404	0.509574	0.226797	0.022*
C7	1.25684(14)	0.4753(3)	0.34328(13)	0.0212(4)
H7	1.305228	0.506745	0.314391	0.025*
C8	0.95190(13)	0.3997(2)	0.19261(12)	0.0167(4)
H8	0.997362	0.358814	0.162008	0.020*
C9	0.84837(13)	0.4211(2)	0.13888(12)	0.0160(4)
C10	0.77691(13)	0.4702(3)	0.18207(12)	0.0170(4)
C11	0.67799(13)	0.4808(3)	0.12602(13)	0.0202(4)
C12	0.65013(13)	0.4468(3)	0.03101(12)	0.0208(4)
H12	0.582603	0.453188	−0.005785	0.025*
C13	0.72209(13)	0.4032(3)	−0.00981(12)	0.0192(4)
C14	0.81985(13)	0.3880(3)	0.04265(12)	0.0179(4)
H14	0.867901	0.355075	0.013586	0.021*
Cl1	0.58894(4)	0.53953(9)	0.17759(3)	0.03328(15)
Cl2	0.68764(4)	0.36970(8)	−0.13031(3)	0.03304(15)
N1	1.04752(11)	0.2997(2)	0.46899(10)	0.0172(3)
N2	0.98336(11)	0.4350(2)	0.28064(10)	0.0164(3)
O1	1.38497(9)	0.4397(2)	0.48332(9)	0.0276(3)
O2	1.06310(10)	0.3325(2)	0.55236(9)	0.0273(3)
O3	0.97773(9)	0.20356(19)	0.42167(9)	0.0235(3)
O4	0.79912(9)	0.5047(2)	0.27367(8)	0.0226(3)
H4	0.860203	0.491931	0.299387	0.034*

Experimental details

The H atoms were positioned geometrically with $d(\text{C-H}) = 0.93\text{--}0.98\text{ \AA}$ and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{carrier})$ or $1.5 U_{\text{eq}}(\text{methyl})$. Solving, refining and finishing of the crystal structure were finished by SHELXS [2] and OLEX2 [3].

Discussion

Schiff bases of salicylaldehyde with amines(anils) comprise a chemical system undergoing hydrogen-atom tautomerism between enol and keto forms and show the phenomena of solid state photochromism and thermochromism [4].

Similar to the crystal structure of the related Schiff base [5, 6], the title compound with electron-withdrawing chloro-substituents (cf. the figure) clearly shows the enol form. The molecule displays a *E*-configuration at the central C8=N2 bond. The dihedral angle between the two substituted phenyl moieties (C2—C3—C4—C5—C6—C7 and C9—C10—C11—C12—C13—C14) is 30.5°. There is an intramolecular O—H...N hydrogen bond in the molecular structure of the title compound. All bond lengths and angles are in the expected ranges.

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