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4-{[(1*E*)-(3,5-Dichloro-2-hydroxyphenyl)-methylene]amino}-1,5-dimethyl-2-phenyl-3*H*-pyrazol-3(2*H*)-one

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The crystal structure of the title compound, $C_{18}H_{15}Cl_2N_3O_2$, shows a strong intramolecular O—H···N hydrogen bond [N···O = 2.598 (3) Å, O—H = 0.81 (3) Å, H···N = 1.86 (3) Å and O—H···N = 152 (3)°], which leads to the existence of a phenol–imine tautomer.

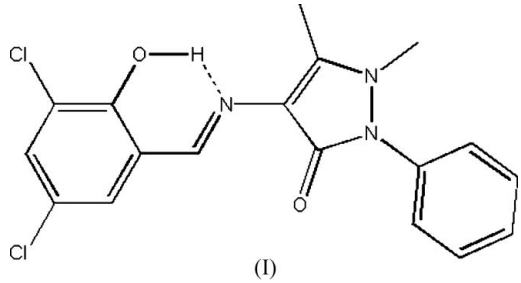
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.135
Data-to-parameter ratio = 12.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Great interest has been devoted to the preparation and study of the Schiff bases derived from salicylaldehyde due to their tautomeric structure (Salman *et al.*, 1991), fluorescent (Morishige *et al.*, 1980), and thermo- and photochromic properties (Barbara *et al.*, 1980; Cohen *et al.*, 1964). In a search for new analytical reagents, we have synthesized some compounds of substituted salicylaldehyde with 4-aminoantipyrine (Huang *et al.*, 2005). We report here the synthesis and crystal structure of the title compound, (I).



(I)

All the bond distances and angles are normal and agree with the corresponding values found in a similar compound, *viz*. 4-[(2-hydroxy-3-methoxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Diao *et al.*, 2005). There is an intramolecular O—H···N hydrogen bond (Table 2); the compound is in the phenol–imine form, as in 4-{[(1*E*)-(2-hydroxyphenyl)methylidene]amino}-1,5-dimethyl-2-phenyl-2,3-dihydro-1*H*-pyrazol-3-one [N1···O1 = 2.607 (3) Å, O1—H1 = 0.97 (3) Å, H1···N1 = 1.71 (3) Å and O1—H1···N1 = 153 (2)°; Hökelek *et al.*, 2001].

Experimental

3,5-Dichlorosalicylaldehyde was prepared according to the method of Sukuzi & Takashi (1983). Ethanol solutions of 3,5-dichlorosalicylaldehyde (10 mmol, 1.70 g) and 4-aminoantipyrine (10 mmol, 2.03 g) were mixed and refluxed on a water bath for 2 h. After cooling, the separated precipate was filtered off, washed and recrystallized from methanol (yield: 83%; m.p. 498.6–499.1 K). IR (KBr, cm^{-1}): ν_{max} 3430.5, 1664.5, 1592.1, 1452.3, 1356.8, 1290.3, 1136.0, 766.7. ^{1}H NMR (200 MHz, $CDCl_3$): δ 14.25 (1H), 9.72 (1H), 7.18–7.59 (7H), 3.21 (3H), 2.42 (3H).

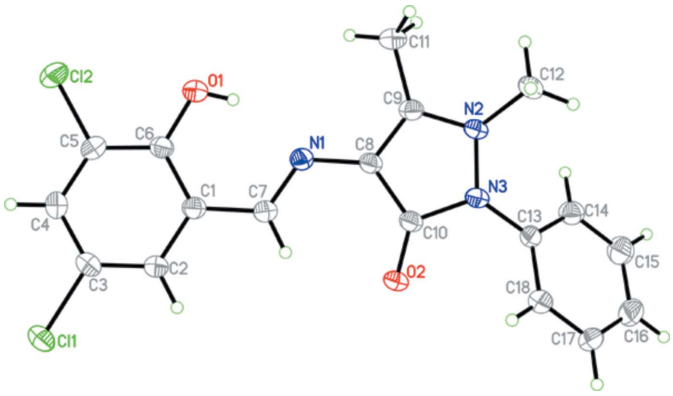


Figure 1
The structure of compound (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Crystal data

$C_{18}H_{15}Cl_2N_3O_2$
M_r = 376.23
Monoclinic, $P2_1/n$
a = 7.0146 (6) Å
b = 8.0466 (7) Å
c = 30.510 (3) Å
β = 90.921 (2)°
V = 1721.9 (3) Å^3
Z = 4
D_x = 1.451 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3109 reflections
θ = 1.3–25.3°
μ = 0.39 mm^{-1}
T = 293 (2) K
Block, orange
0.22 × 0.17 × 0.16 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.923, T_{max} = 0.939
8818 measured reflections
3109 independent reflections
2510 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.3°
$h = -6 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -36 \rightarrow 35$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
S = 1.14
3109 reflections
255 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.3379P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0028 (13)

Table 1
Selected geometric parameters (Å, °).

Cl1—C3	1.744 (3)	N1—C8	1.395 (3)
Cl2—C5	1.736 (3)	N2—N3	1.412 (3)
O1—C6	1.340 (3)	N3—C10	1.403 (3)
O2—C10	1.233 (3)		
C7—N1—C8	120.7 (2)	O1—C6—C5	120.4 (2)
C2—C3—C4	121.3 (2)	O1—C6—C1	121.3 (2)

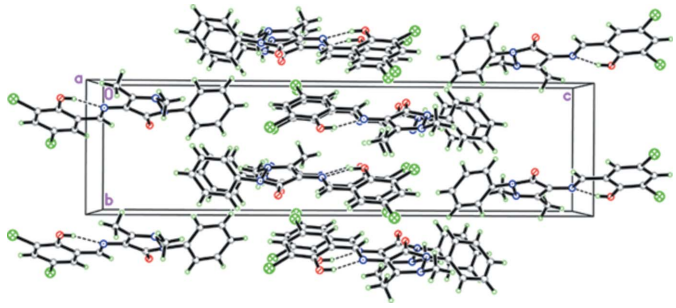


Figure 2
The packing diagram of (I), viewed down the a axis. Dashed lines indicate hydrogen bonds.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1	0.81 (3)	1.86 (3)	2.598 (3)	152 (3)

The hydroxy H atom (H1A) and the methyl H atom were positioned from a difference map, refined several cycles then fixed at a distance of 0.80 Å; the methyl H atoms on C11 and C12 were located in a Fourier synthesis and refined freely. The remaining H atoms were were positioned geometrically and treated as riding, at distances of 0.93 (CH) and 0.96 Å (CH_3) and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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