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4-[(2,4-Dichlorobenzylidene)amino]-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.094
Data-to-parameter ratio = 13.4

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

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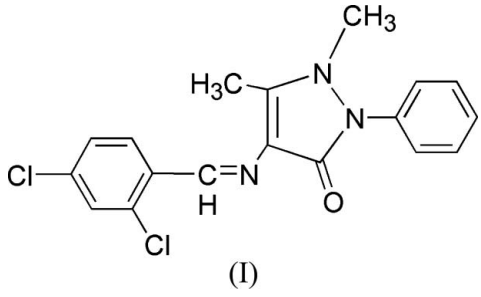
The crystal structure of the title compound, $C_{18}H_{15}Cl_2N_3O$, shows that the Cl atoms take part in intermolecular C—H···Cl interactions.

Comment

The synthesis and crystal structures of Schiff base ligands derived from 4-aminoantipyrine, such as thenoyltrifluoroacetone and 4-hydroxy-3-methoxybenzaldehyde, have been reported (Yu *et al.*, 2002; Diao *et al.*, 2005). In the present study, we report the synthesis and structure of the title compound, (I).



(I)

In (I) (Fig. 1), the central system (C7–C10/N1–N3/O1) is planar, with an r.m.s. deviation of fitted atoms of 0.0776 Å, and the dihedral angle with the phenyl ring (C13–C18) is 55.98 (6)°. The 2,4-dichlorobenzene group (C1–C7/Cl1/Cl2) is planar, with an r.m.s. deviation of fitted atoms of 0.0192 Å, and the dihedral angle with the central system is 24.25 (5)°. The Cl atoms participate in C—H···Cl interactions (Table 2).

Experimental

An anhydrous ethanol solution of 2,4-dichlorobenzaldehyde (1.75 g, 10 mmol) was added to an anhydrous ethanol solution of 4-amino-

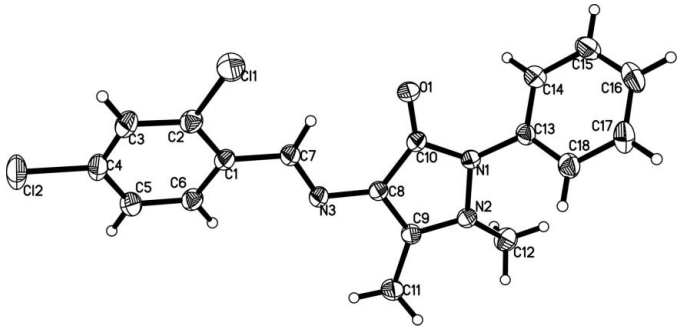


Figure 1
A view of the title compound, with 30% probability displacement ellipsoids.

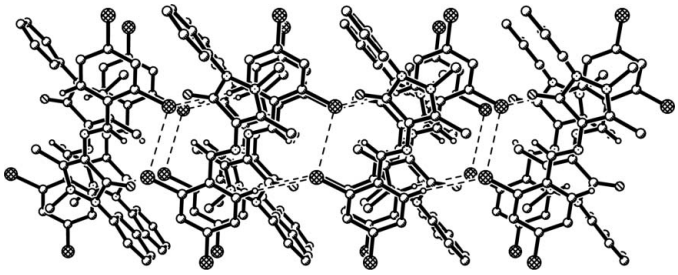


Figure 2
Intermolecular hydrogen-bonding interactions (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

1,5-dimethyl-2-phenylpyrazolidin-3-one (2.03 g, 10 mmol), and the mixture was stirred at 350 K for 5 h under nitrogen. A yellow precipitate appeared. The product was isolated, recrystallized from ethanol and then dried *in vacuo* to give pure compound (I) in 78% yield. Bright-yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution of (I).

Crystal data

$C_{18}H_{15}Cl_2N_3O$
M_r = 360.23
Monoclinic, $P2_1/n$
a = 9.4236 (7) Å
b = 7.3711 (5) Å
c = 24.5011 (18) Å
β = 99.6930 (10)°
V = 1677.6 (2) Å^3
Z = 4
D_x = 1.426 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3316 reflections
θ = 2.9–25.3°
μ = 0.40 mm^{-1}
T = 294 (2) K
Block, yellow
0.44 × 0.32 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.802, T_{max} = 0.909
8814 measured reflections
2935 independent reflections
2235 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -11 \rightarrow 11$
$k = -8 \rightarrow 8$
$l = -26 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.094
S = 1.08
2935 reflections
219 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.3505P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C2	1.735 (2)	N3—C7	1.281 (2)
Cl2—C4	1.742 (2)	N3—C8	1.390 (2)
N1—N2	1.406 (2)		
N2—N1—C10	108.80 (14)	C9—N2—C12	125.05 (16)
N2—N1—C13	120.67 (14)	N1—N2—C12	119.41 (15)
C10—N1—C13	122.58 (15)	C7—N3—C8	119.45 (15)
C9—N2—N1	107.46 (13)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1	0.93	2.31	3.005 (2)	131
C6—H6···Cl1^{i}	0.93	2.79	3.703 (2)	168

Symmetry code: (i) $x, y+1, z$.

H atoms were included in calculated positions and refined using a riding-model approximation [C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic CH; C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl CH_3].

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); 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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