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(*E*)-4-[4-(4-Chlorobenzyloxy)benzylidene-amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Tuo-Ping Hu

College of Sciences, North University of China, Taiyuan 030051, People's Republic of China

Correspondence e-mail: hu_tuoping@163.com

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.091
Data-to-parameter ratio = 16.2

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

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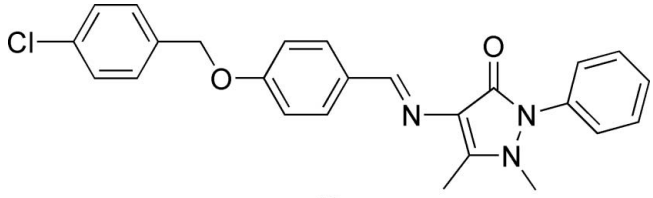
The title compound, $C_{25}H_{22}ClN_3O_2$, was prepared by the reaction of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one and 4-(4-chlorobenzyloxy)benzaldehyde. The substituted benzaldehyde group makes dihedral angles of 58.85 (5), 6.87 (9) and 47.36 (7)° with the chlorobenzyl ring, the pyrazolone ring and the terminal phenyl ring, respectively. Intermolecular C—H···O hydrogen bonds help to consolidate the crystal packing.

Comment

Transition metal complexes of pyrazolone derivatives are of great interest due to their biological activity. Consequently, many pyrazolone Schiff base derivatives have been synthesized which will provide useful information for the coordination properties functioning as ligands (Kuncheria & Indrasenan, 1988; Radhakrishnan *et al.*, 1984). In the present study, we report the synthesis and crystal structure of the 4-amino-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one Schiff base derivative (I).



(I)

In (I) (Fig. 1), the bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987). The substituted benzaldehyde group (C8–C14/O1) is essentially planar, with an r.m.s. deviation for fitted atoms of 0.0081Å, and it makes dihedral angles of 58.85 (5), 6.87 (9) and 47.36 (7)° with the chlorobenzyl ring (C1–C7/Cl1), the pyrazolone ring (C16–C18/N1–N3/O2) and the terminal phenyl ring (C20–C25), respectively.

The pyrazolone ring (C16–C18/N1–N3/O2) is also reasonably planar, with an r.m.s. deviation for fitted atoms of 0.0397Å. It makes a dihedral angle of 52.18 (6)° with the terminal phenyl ring (C16–C21).

The packing is stabilized by weak non-classical intermolecular C—H···O hydrogen bonds (Table 2), forming a supramolecular structure (Fig. 2).

Experimental

An anhydrous methanol solution (30 ml) of 4-amino-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (2.03 g, 10 mmol) was added to an anhydrous methanol solution (30 ml) of 4-(4-chlorobenzyloxy)benz-

aldehyde (2.47 g, 10 mmol) and the mixture refluxed for 3 h under nitrogen, whereupon a yellow precipitate appeared. The product was isolated and recrystallized from ethanol, and then dried *in vacuo* to give the pure compound in 91% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{25}H_{22}ClN_3O_2$	$Z = 4$
$M_r = 431.91$	$D_x = 1.289$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 17.038$ (6) Å	$\mu = 0.20$ mm^{-1}
$b = 6.916$ (2) Å	$T = 294$ (2) K
$c = 19.160$ (6) Å	Plate, yellow
$\beta = 99.597$ (6)°	0.40 × 0.30 × 0.12 mm
$V = 2226.0$ (13) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	12103 measured reflections
	4572 independent reflections
φ and ω scans	2209 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.056$
	$\theta_{max} = 26.6°$
$T_{min} = 0.920$, $T_{max} = 0.977$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0266P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$(\Delta/\sigma)_{max} = 0.003$
$wR(F^2) = 0.091$	$\Delta\rho_{max} = 0.21$ e Å^{-3}
$S = 1.03$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
4572 reflections	Extinction correction: *SHELXL97*
283 parameters	Extinction coefficient: 0.0116 (7)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cl1—C3	1.7428 (19)	N2—C16	1.364 (2)
O1—C8	1.379 (2)	N2—N3	1.411 (2)
O1—C7	1.423 (2)	N2—C19	1.462 (2)
O2—C18	1.233 (2)	N3—C18	1.405 (2)
N1—C14	1.279 (2)	N3—C20	1.426 (2)
N1—C17	1.396 (2)		
C8—O1—C7	116.73 (16)	O1—C8—C9	115.30 (19)
C14—N1—C17	120.30 (18)	N1—C14—C11	121.9 (2)
C16—N2—N3	106.62 (15)	N2—C16—C17	110.52 (18)
C16—N2—C19	124.11 (17)	N2—C16—C15	121.45 (18)
N3—N2—C19	117.03 (15)	C16—C17—N1	122.47 (19)
C18—N3—N2	109.23 (15)	N1—C17—C18	129.34 (19)
C18—N3—C20	123.76 (16)	O2—C18—N3	122.62 (18)
N2—N3—C20	120.37 (15)	O2—C18—C17	132.54 (18)
C2—C3—Cl1	119.29 (18)	N3—C18—C17	104.80 (17)
C4—C3—Cl1	119.68 (18)	C21—C20—N3	121.18 (19)
O1—C7—C6	109.42 (18)	C25—C20—N3	118.56 (18)
C13—C8—O1	124.67 (19)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C21—H21···O1^{i}	0.93	2.49	3.383 (3)	161
C4—H4···O2^{ii}	0.93	2.56	3.220 (3)	128
C15—H15*C*···O2^{iii}	0.96	2.46	3.413 (3)	173

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x, y-1, z$.

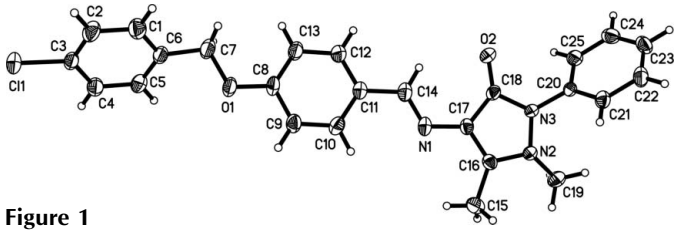


Figure 1
The structure of (I), with displacement ellipsoids drawn at the 30% probability level.

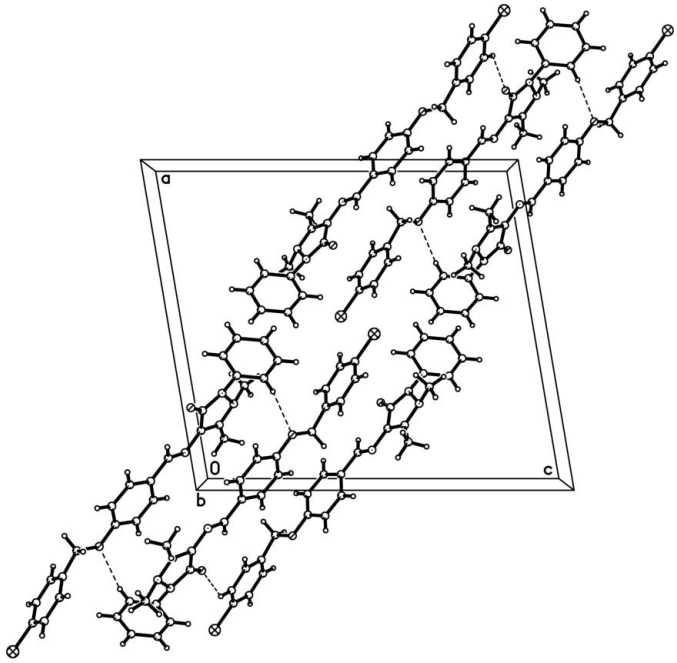


Figure 2
A view, down the *b* axis, of the packing arrangement in the crystal structure of (I). Dashed lines indicate hydrogen bonds. H atoms have been omitted.

H atoms were included in calculated positions and refined using a riding-model approximation. Constrained C—H and N—H bond lengths and isotropic U_{iso}(H) parameters: 0.93 Å and $1.2U_{eq}$(C) for aromatic CH; 0.97 Å and $1.2U_{eq}$(C) for methylene CH_2; 0.96 Å and $1.5U_{eq}$(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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