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

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Key indicators

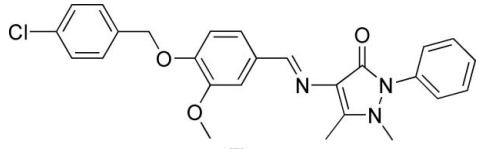
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.117
Data-to-parameter ratio = 16.0

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

The title compound, $C_{26}H_{24}ClN_3O_3$, was prepared by the reaction of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one and 4-(4-chlorobenzyloxy)-3-methoxybenzaldehyde. The vanillin group makes dihedral angles of 6.21 (10) and 48.05 (6)°, respectively, with the pyrazolone and chlorobenzene rings. The crystal packing is governed by C—H···O hydrogen bonds and C—H···π and π–π interactions.

Comment

Schiff base ligands have received a good deal of attention in the development of coordination chemistry for more than 100 years (Kahwa *et al.*, 1986). Among the large number of compounds, 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one forms a variety of Schiff bases with aldehydes, and the synthesis and crystal structures of some of them, such as (*E*)-(4)-(3-ethoxy-4-hydroxybenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Han & Zhen, 2005) and (*E*)-4-[4-(4-chlorobenzyloxy)-3-ethoxybenzylideneamino]-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (Zhang *et al.*, 2006) have been reported. We report here the synthesis and structure of the title compound, (I).



(I)

The bond lengths and angles in the title molecule (Fig. 1) are comparable to those found in previous similar studies (Han & Zhen, 2005; Zhang *et al.*, 2006). The pyrazolone ring (C16/C17/C20/N2/N3/O3) is almost planar, with an r.m.s. deviation for fitted atoms of 0.037 Å. It makes a dihedral angle of 53.79 (7)° with the terminal phenyl ring (C21–C26). The vanillin group (C8–C13/C15/O1/O2) is planar, with an r.m.s. deviation for fitted atoms of 0.016 Å. This group makes dihedral angles of 6.21 (10) and 48.05 (6)°, respectively, with the pyrazolone ring and the benzene ring (C1–C6).

The crystal packing is stabilized by weak, non-classical intermolecular C—H···O hydrogen bonds and C—H···π interactions (Table 1) involving the C8–C13 benzene ring (centroid *Cg*1). In addition, a π–π stacking interaction is observed between the pyrazolone ring and the C1–C6 benzene ring of the molecule at the symmetry position $(2-x, \frac{1}{2}+y, \frac{3}{2}-z)$; the centroid–centroid distance between the two rings is 3.624 (1) Å.

Experimental

An anhydrous ethanol solution (20 ml) of 3-(4-chlorobenzyloxy)-4-methoxybenzaldehyde (2.77 g, 10 mmol) was added to an anhydrous

ethanol solution (20 ml) of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one (2.03 g, 10 mmol) and the mixture was stirred at 350 K for 3 h under N_2, giving a yellow precipitate. The product was isolated, recrystallized from ethanol and then dried in a vacuum to give pure compound (I) in 89% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{26}H_{24}ClN_3O_3$	$Z = 4$
$M_r = 461.93$	$D_x = 1.303$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 16.831$ (3) Å	$\mu = 0.20$ mm^{-1}
$b = 7.4587$ (11) Å	$T = 294$ (2) K
$c = 18.993$ (3) Å	Block, yellow
$\beta = 99.099$ (3)°	0.36 × 0.32 × 0.26 mm
$V = 2354.3$ (7) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	12790 measured reflections
	4803 independent reflections
φ and ω scans	2892 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.043$
	$\theta_{max} = 26.4°$
$T_{min} = 0.923$, $T_{max} = 0.951$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.6873P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.043$	
$wR(F^2) = 0.117$	
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.002$
4803 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
301 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL97*
	Extinction coefficient: 0.0153 (11)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O3^{i}	0.93	2.48	3.218 (3)	136
C15—H15···O3	0.93	2.40	3.063 (3)	128
C13—H13···$Cg1^{ii}$	0.93	2.85	3.664 (2)	147
C18—H18A···$Cg1^{iii}$	0.96	2.85	3.568 (2)	133

Symmetry codes: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+2, -y+2, -z+1$. $Cg2$ is the centroid of the ring C8–C13.

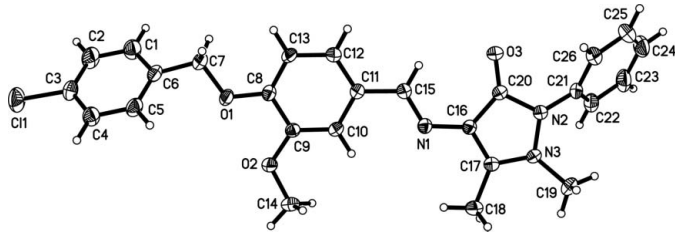


Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

H atoms were included at calculated positions and refined using a riding model. Constrained bond lengths and U_{iso}(H) parameters: 0.93 Å and $1.2U_{eq}$(C) for aromatic, 0.97 Å and $1.2U_{eq}$(C) for methylene, 0.96 Å and $1.5U_{eq}$(C) for methyl H atoms.

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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