

(E)-4-[4-(2,4-Dichlorobenzoyloxy)-3-methoxybenzylideneamino]-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one**Xiao-Li Zhen,^a Jian-Rong Han,^{a*} Xia Tian,^a Zhen-Chao Li^a and Shou-Xin Liu^{b,‡}**^aCollege of Sciences, Hebei University of Science & Technology, Shijiazhuang 050018, People's Republic of China, and ^bCollege of Chemical & Pharmaceutical Engineering, Hebei University of Science & Technology, Shijiazhuang 050018, People's Republic of China

‡ Second contact author, email: liu_shouxin@163.com

Correspondence e-mail: han_jianrong@163.com

Key indicators

Single-crystal X-ray study

T = 294 K

Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$

R factor = 0.049

wR factor = 0.150

Data-to-parameter ratio = 13.9

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

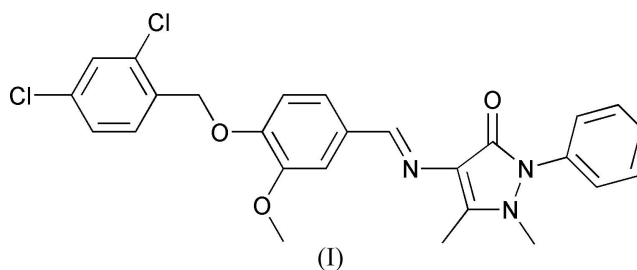
In the title compound, $\text{C}_{26}\text{H}_{23}\text{Cl}_2\text{N}_3\text{O}_3$, the central vanillin group makes dihedral angles of 51.19 (8), 7.51 (11) and 45.65 (11) $^\circ$ with the dichlorobenzene ring, the pyrazolone ring and the terminal phenyl ring, respectively. Intermolecular $\text{C}-\text{H} \cdots \text{O}$ interactions help to consolidate the crystal packing.

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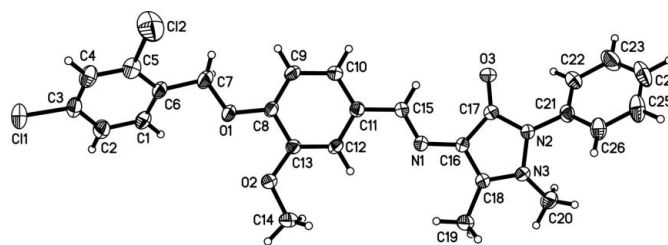
Comment

We report here the synthesis and crystal structure of the title compound, (I) (Fig. 1), which was investigated as part of our ongoing studies of Schiff base adducts with aldehydes (Han & Zhen, 2005).



The bond lengths and angles in (I) are within their normal ranges (Allen *et al.*, 1987). The pyrazolone ring (atoms C16–C18/N1–N3/O3) is close to being planar, with an r.m.s. deviation for the fitted atoms of 0.036 Å. It makes a dihedral angle of 52.12 (11) $^\circ$ with the attached phenyl ring (C21–C26). The central vanillin group (C8–C13/C15/O1/O2) is also planar, with an r.m.s. deviation for the fitted atoms of 0.015 Å, and it makes dihedral angles of 51.19 (8), 7.51 (11) and 45.65 (11) $^\circ$ with the benzene ring (C1–C6), the pyrazolone ring (C16/C17/C20/N1–N3/O3) and the terminal phenyl ring (C21–C26), respectively.

The crystal structure of (I) is stabilized by weak $\text{C}-\text{H} \cdots \text{O}$ interactions (Fig. 2 and Table 1).

**Figure 1**

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

Experimental

An anhydrous ethanol solution (50 ml) of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one (2.03 g, 10 mmol) was added to an anhydrous ethanol solution (100 ml) of 4-(2,4-dichlorobenzyloxy)-3-methoxybenzaldehyde (3.11 g, 10 mmol) and the mixture stirred at 350 K for 5 h under nitrogen, giving a yellow precipitate. The product was isolated, recrystallized from acetonitrile and then dried in a vacuum to give the pure compound in 78% yield. Yellow single crystals of (I) suitable for X-ray crystallographic analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{26}H_{23}Cl_2N_3O_3$ $Z = 4$
 $M_r = 496.37$ $D_x = 1.352 \text{ Mg m}^{-3}$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 16.952(3) \text{ \AA}$ $\mu = 0.30 \text{ mm}^{-1}$
 $b = 7.4311(12) \text{ \AA}$ $T = 294(2) \text{ K}$
 $c = 19.699(3) \text{ \AA}$ Block, yellow
 $\beta = 100.691(3)^\circ$ $0.22 \times 0.16 \times 0.12 \text{ mm}$
 $V = 2438.5(7) \text{ \AA}^3$

Data collection

Bruker SMART APEX CCD 11826 measured reflections
 diffractometer 4288 independent reflections
 φ and ω scans 2509 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{int} = 0.043$
 (SADABS; Sheldrick, 1996) $\theta_{max} = 25.0^\circ$
 $T_{min} = 0.928, T_{max} = 0.965$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 0.7898P]$
 $R[F^2 > 2\sigma(F^2)] = 0.050$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.150$ $(\Delta/\sigma)_{max} = 0.001$
 $S = 1.03$ $\Delta\rho_{max} = 0.47 \text{ e \AA}^{-3}$
 4288 reflections $\Delta\rho_{min} = -0.39 \text{ e \AA}^{-3}$
 309 parameters
 H-atom parameters constrained

Table 1

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C2-H2\cdots O3^i$	0.93	2.57	3.281 (4)	133

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

The H atoms were included in calculated positions ($C-H = 0.93-0.97 \text{ \AA}$) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(\text{methyl C})$.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve

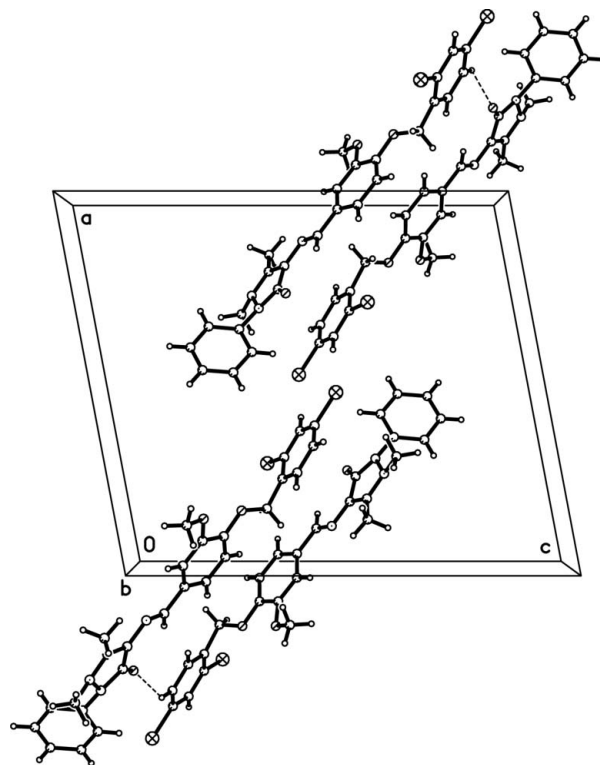


Figure 2 Partial packing diagram for (I), with hydrogen bonds shown as dashed lines.

structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bruker (1999). SMART (Version 5.0) and SAINT (Version 4.0) for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Han, J.-R. & Zhen, X.-L. (2005). *Acta Cryst.* E61, o3815–o3816.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997a). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). SHELXTL. Version 5.10 for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.