Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-4-[4-(Benzyloxy)-3-ethoxybenzylidene-amino]-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) = 0.004 Å
R factor = 0.046
wR factor = 0.127
Data-to-parameter ratio = 16.1

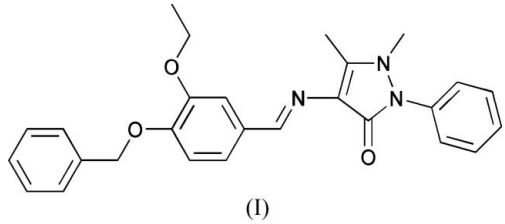
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 28 October 2005
Accepted 2 November 2005
Online 5 November 2005

The title compound, $C_{27}H_{27}N_3O_3$, was prepared by the reaction of 4-(benzyloxy)-3-ethoxybenzaldehyde and 4-amino-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one. The ethylvanillin group makes dihedral angles of 72.97 (8) and 76.75 (5)° with the planes of the two terminal phenyl rings, and an angle of 39.24 (5)° with the pyrazolone ring plane. Intermolecular C—H···N and C—H···O hydrogen bonds help to consolidate the crystal packing.

Comment

Metal complexes based on Schiff bases have attracted much attention because of their significant biological activity (Kahwa *et al.*, 1986). Consequently, a large number of Schiff base derivatives have been synthesized to develop protein and enzyme mimics (Santos *et al.*, 2001). We report here the synthesis and structure of the title pyrazalone Schiff base derivative, (I).



(I)

In (I), the ethylvanillin group (C8–C13/C16/O1/O2) is planar, with an r.m.s. deviation of fitted atoms of 0.0146 Å (Fig. 1). This group makes dihedral angles of 72.97 (8) and 76.75 (5), respectively, with the terminal C1–C6 and C22–C27 phenyl rings. The pyrazolone ring (C17–C20/N1–N3/O3) is also reasonably planar, with an r.m.s. deviation for fitted atoms of 0.0402 Å. It makes dihedral angles of 39.24 (5), 68.43 (8) and 46.41 (6), respectively, with the ethylvanillin group and the terminal C1–C6 and C22–C27 phenyl rings.

Packing is stabilized by weak non-classical intermolecular C26—H26···O3 hydrogen bonds, which form centrosymmetric dimers, together with an intermolecular C5—H5···N1 hydrogen bond (Fig. 2 and Table 2).

Experimental

An anhydrous ethanol solution of 4-(benzyloxy)-3-ethoxybenzaldehyde (2.66 g, 10 mmol) was added to an anhydrous ethanol solution of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one (2.03 g, 10 mmol) and the mixture stirred at 350 K for 5 h under nitrogen, whereupon a yellow precipitate appeared. The product was isolated, recrystallized from ethanol and dried in a vacuum to give the pure compound in 86% yield. Yellow single crystals of (I) suitable for X-

ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{27}H_{27}N_3O_3$
M_r = 441.52
Monoclinic, $P2_1/c$
a = 10.043 (3) Å
b = 24.317 (7) Å
c = 10.199 (3) Å
β = 108.275 (5)°
V = 2365.1 (12) Å^3
Z = 4
D_x = 1.240 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2344 reflections
θ = 2.3–22.3°
μ = 0.08 mm^{-1}
T = 294 (2) K
Block, yellow
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.969, T_{max} = 0.990
13268 measured reflections
4843 independent reflections
2442 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 26.4°
$h = -10 \rightarrow 12$
$k = -30 \rightarrow 22$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.127
S = 0.99
4843 reflections
301 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.366 (2)	N1—C17	1.407 (2)
O1—C7	1.432 (2)	N2—C19	1.406 (2)
O2—C9	1.371 (2)	N2—N3	1.410 (2)
O2—C14	1.416 (2)	N2—C22	1.426 (2)
O3—C19	1.235 (2)	N3—C18	1.363 (2)
N1—C16	1.274 (2)	N3—C21	1.463 (3)
C8—O1—C7	117.52 (16)	O1—C7—C6	107.28 (18)
C9—O2—C14	117.37 (15)	O1—C8—C13	124.96 (19)
C16—N1—C17	120.44 (17)	O1—C8—C9	115.41 (17)
C19—N2—N3	109.35 (16)	N3—C18—C20	121.17 (19)
C19—N2—C22	123.87 (17)	O3—C19—N2	123.94 (18)
N3—N2—C22	118.68 (15)	O3—C19—C17	131.63 (19)
C18—N3—N2	106.47 (15)	N2—C19—C17	104.42 (18)
C18—N3—C21	122.01 (18)	C27—C22—N2	118.77 (18)
N2—N3—C21	116.74 (17)	C23—C22—N2	121.1 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···N1^{i}	0.93	2.74	3.503 (3)	140
C26—H26···O3^{ii}	0.93	2.59	3.349 (3)	139

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z-1$.

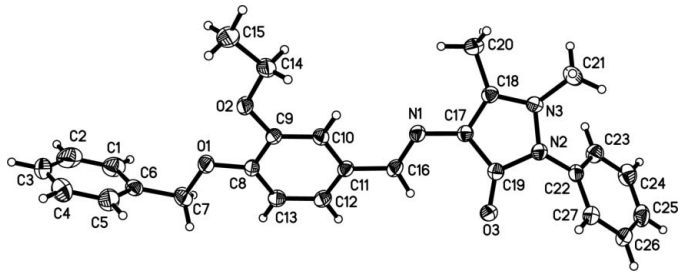


Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

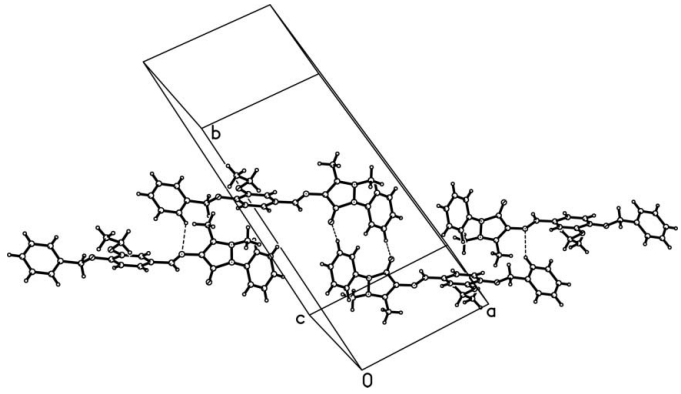


Figure 2
Packing diagram for (I) with hydrogen bonds drawn as dashed lines.

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