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(*E*)-4-(3-Ethoxy-4-hydroxybenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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Key indicators

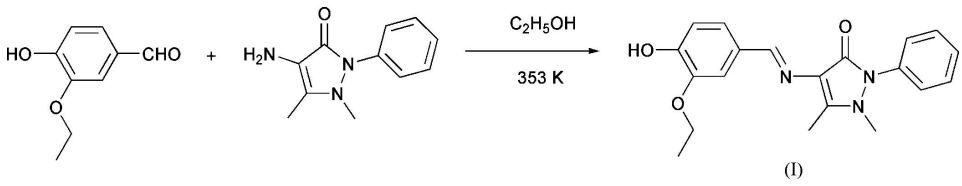
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.085
Data-to-parameter ratio = 7.7

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

The molecule of the title compound, $C_{20}H_{21}N_3O_3$, is non-planar. The central pyrazolone ring makes a dihedral angle of 49.85 (9)° with the phenyl ring and 11.10 (9)° with the vanillin moiety. The crystal structure shows the presence of intermolecular O—H···O hydrogen bonding in the solid state.

Comment

Metal complexes based on Schiff bases have been intensively studied as mimics of active centers in various proteins and enzymes (Kahwa *et al.*, 1986). Consequently, many of this species have been synthesized (Santos *et al.*, 2001). As part of this study, we report the synthesis and structure of the title compound, (I).



(I)

A view of the molecule is shown in Fig. 1. The central pyrazolone ring (C1–C4/N1/N2/O1) is planar, with an r.m.s. deviation of fitted atoms of 0.0393 Å, and this plane makes a dihedral angle of 49.85 (9)° with the phenyl ring (C5–C10). The vanillin moiety (C12–C19/O2/O3) is planar, with an r.m.s. deviation of fitted atoms of 0.0165 Å; the dihedral angle between the central pyrazolone ring and the vanillin moiety is 11.10 (9)°. Intermolecular O—H···O hydrogen bonds are observed (Table 1, Fig. 2), which stabilize the solid-state structure.

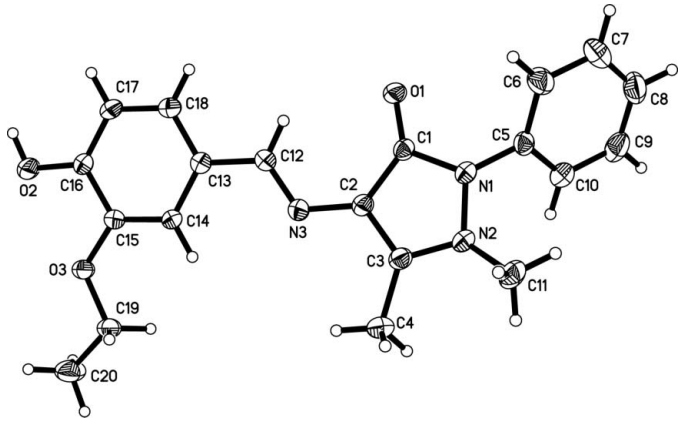


Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

Experimental

An anhydrous ethanol solution of 3-ethoxy-4-hydroxybenzaldehyde (1.66 g, 10 mmol) was added to an anhydrous ethanol solution of 4-amino-1,5-dimethyl-2-phenylpyrazolidin-3-one (2.03 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 ethanol, and then dried in a vacuum to give the pure compound in 81% yield. Bright yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{20}H_{21}N_3O_3$
M_r = 351.40
Monoclinic, *Cc*
a = 11.5991 (16) Å
b = 17.114 (2) Å
c = 9.2425 (13) Å
β = 96.883 (2)°
V = 1821.4 (4) Å^3
Z = 4
D_x = 1.281 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 3329 reflections
θ = 2.1–26.3°
μ = 0.09 mm^{-1}
T = 294 (2) K
Block, yellow
0.40 × 0.30 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.960, T_{max} = 0.979
5060 measured reflections
1854 independent reflections
1727 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 26.3°
h = −14 → 14
k = −21 → 21
l = −11 → 6

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.085
S = 1.05
1854 reflections
242 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2 + 0.3327P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.86 (4)	1.82 (4)	2.671 (2)	171 (4)

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

The hydroxy H atom was found in a difference Fourier map and refined with free coordinates and an isotropic *U* parameter. Other H atoms were included in calculated positions [C—H = 0.93 (aromatic) or 0.96 Å (methyl)] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) for aromatic CH and U_{iso}(H) = 1.5U_{eq}(C) for methyl CH_3. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

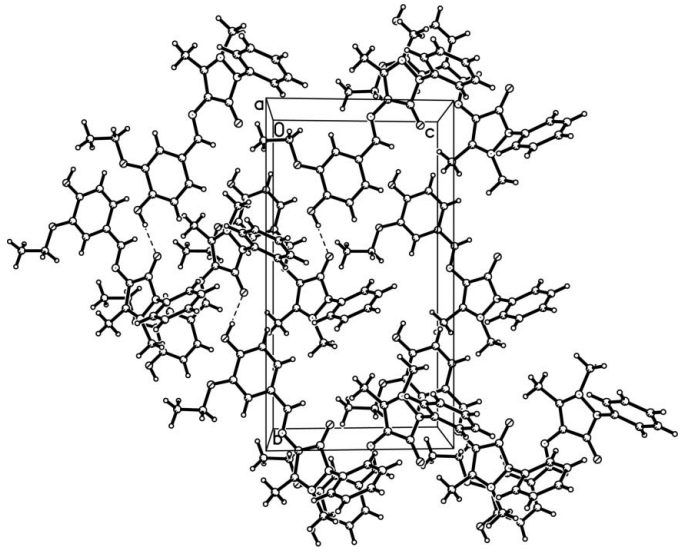


Figure 2
Intermolecular hydrogen-bonding interactions (dashed lines).

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