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(*E*)-4-(4-Ethoxy-3-methoxybenzylideneamino)-1,5-dimethyl-2-phenylpyrazolidin-3-one

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.115
Data-to-parameter ratio = 15.8

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

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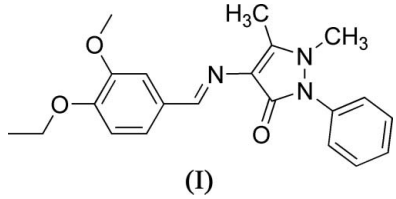
In the title compound, $C_{21}H_{23}N_3O_3$, the substituted vanillin group makes dihedral angles of 48.16 (5) and 4.01 (4)° with the terminal phenyl ring and the pyrazolone ring, respectively. An intermolecular C—H···O hydrogen bond helps to consolidate the crystal packing.

Comment

Schiff base ligands have played an important role in the development of coordination chemistry since the late 19th century. Consequently, a large number of these species have been reported to be superior reagents in biological, pharmacological, clinical and analytical applications (Wang *et al.*, 2005; Yang *et al.*, 2005). As part of an investigation of their crystal structures, which will provide useful information for the coordination properties of Schiff bases functioning as ligands, we report here the synthesis and molecular structure of the title compound, (I).



(I)

In (I), the bond lengths and angles (Table 1) are within normal ranges. The substituted vanillin group (C3–C8/C10/O1/O2) is planar, with an r.m.s. deviation of 0.0090 (17) Å (Fig. 1). The pyrazolone ring (C11–C13/N1–N3/O3) is also essentially planar, with an r.m.s. deviation of 0.0329 (19) Å. The vanillin group makes dihedral angles of 4.01 (4) and 48.16 (5)° with the pyrazolone ring and the terminal phenyl ring (C16–C21), respectively. The pyrazolone ring makes a dihedral angle of 44.43 (6)° with the phenyl ring.

The crystal packing is stabilized by a weak intermolecular C—H···O hydrogen bond (Table 2), forming an inversion-related dimer (Fig. 2).

Experimental

An anhydrous ethanol solution (50 ml) of 4-ethoxy-3-methoxybenzaldehyde (1.80 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) 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 then isolated and recrystallized from ethanol, and then dried *in vacuo* to give compound (I) in 83% yield. Yellow single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{21}H_{23}N_3O_3$
M_r = 365.42
Monoclinic, $P2_1/c$
a = 16.214 (4) Å
b = 8.4979 (18) Å
c = 14.888 (3) Å
β = 109.204 (4)°
V = 1937.1 (7) Å^3
Z = 4
D_x = 1.253 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
Block, yellow
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.962, T_{max} = 0.983
10562 measured reflections
3944 independent reflections
2373 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 26.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.115
S = 1.00
3944 reflections
249 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.3272P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0083 (10)

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.365 (2)	N1—C11	1.391 (2)
O1—C2	1.4330 (19)	N2—C12	1.368 (2)
O2—C8	1.3668 (18)	N2—N3	1.4047 (19)
O2—C9	1.414 (2)	N2—C15	1.462 (2)
O3—C13	1.235 (2)	N3—C13	1.392 (2)
N1—C10	1.276 (2)	N3—C16	1.422 (2)
C3—O1—C2	117.53 (13)	O1—C3—C8	115.12 (14)
C8—O2—C9	117.70 (13)	O2—C8—C7	125.38 (15)
C10—N1—C11	121.67 (16)	O2—C8—C3	114.41 (14)
C12—N2—N3	106.19 (14)	C12—C11—N1	122.30 (16)
C12—N2—C15	121.84 (15)	N1—C11—C13	129.79 (17)
N3—N2—C15	115.86 (13)	C11—C12—N2	110.76 (15)
C13—N3—N2	110.13 (13)	O3—C13—N3	124.20 (16)
C13—N3—C16	126.13 (15)	O3—C13—C11	131.17 (18)
N2—N3—C16	119.93 (14)	N3—C13—C11	104.61 (15)
O1—C3—C4	125.86 (15)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15C···O1^{i}	0.96	2.56	3.310 (2)	135

Symmetry code: (i) $-x+1, -y, -z$.

H atoms were included in calculated positions (C—H = 0.93–0.96 Å) and refined using the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

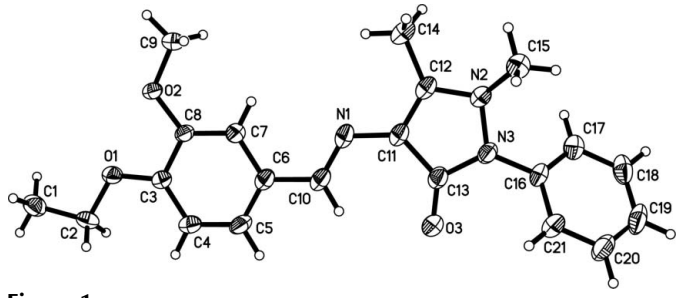


Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

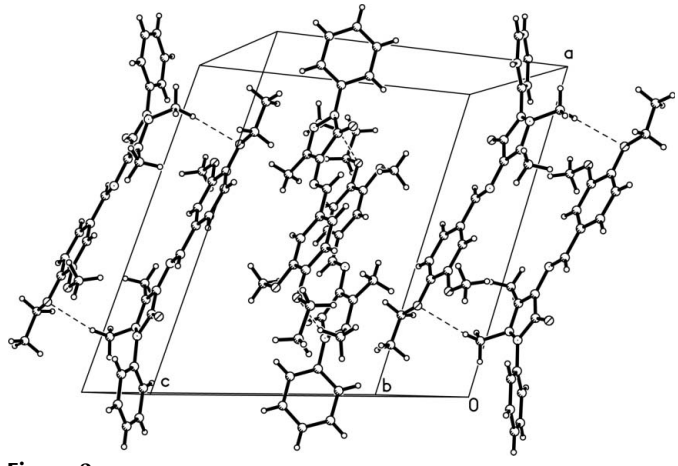


Figure 2
A packing view of (I), showing C—H···O hydrogen bonds as dashed lines.

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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References

Bruker (1999). *SMART* (Version 5.0) and *SAINT* (Version 4.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL97*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Wang, Y., Yang, Z.-Y. & Wang, B.-D. (2005). *Transition Met. Chem.* **7**, 879–883.
Yang, T.-L., Qin, W.-W. & Liu, W.-S. (2005). *J. Anal. Chem.* **4**, 325–329.