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4-[(4-Hydroxy-3-methoxybenzylidene)amino]-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 = 0.031
wR factor = 0.083
Data-to-parameter ratio = 8.9

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

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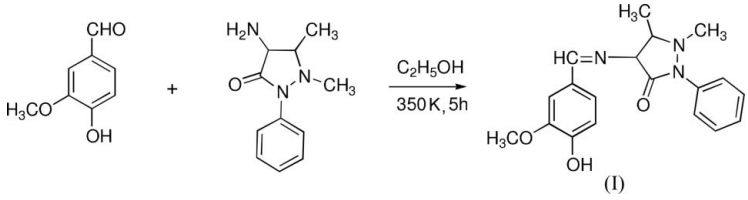
The title compound, $C_{19}H_{19}N_3O_3$, was prepared from 4-hydroxy-3-methoxybenzaldehyde and 4-amino-1,5-dimethyl-2-phenylpyrazolidin-3-one. The crystal structure shows that the molecules associate *via* intermolecular hydrogen bonds to form a supramolecular structure with a zigzag pattern.

Comment

The synthesis of new and designed crystal structures is part of a major strand of modern chemistry. One of the aims of crystal engineering is to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable (Tynan *et al.*, 2005; Parashar *et al.*, 1988). Here we report the synthesis and molecular structure of the title compound, (I).



(I)

The central system (N1/N2/C7/O1/C11/C9/C10/N3/C8) is planar, with an r.m.s. deviation of fitted atoms of 0.041 Å, and the dihedral angle with the phenyl ring (C1–C6) is 53.48 (8)°. The vanillin moiety (C12–C15/C17–C19/O2/O3/C16) is planar, with an r.m.s. deviation of fitted atoms of 0.023 Å; the dihedral angle between the central system and the vanillin moiety is 12.12 (7)°. Intermolecular hydrogen bonds (O2—H2···O1) are observed (Table 1), which stabilize the crystal structure. The molecules associate in a zigzag pattern *via* intermolecular hydrogen bonds to form a supramolecular structure.

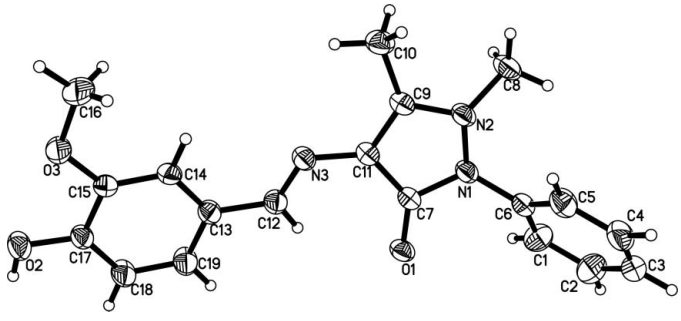


Figure 1
A view of the title compound, with 30% probability displacement ellipsoids.

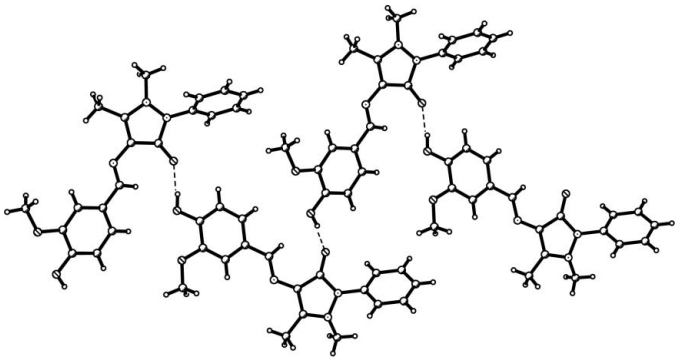


Figure 2
The intermolecular hydrogen-bonding interactions (dashed lines) in (I).

Experimental

An anhydrous ethanol solution of 4-hydroxy-3-methoxybenzaldehyde (1.52 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 was stirred at 350 K for 5 h under nitrogen, whereupon a yellow precipitate appeared. The product was isolated and recrystallized from ethanol, and then dried *in vacuo* 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

$C_{19}H_{19}N_3O_3$
M_r = 337.37
Monoclinic, *Cc*
a = 11.795 (2) Å
b = 16.976 (2) Å
c = 8.7656 (14) Å
β = 99.764 (3)°
V = 1729.7 (5) Å^3
Z = 4
D_x = 1.296 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2121 reflections
θ = 2.4–24.1°
μ = 0.09 mm^{-1}
T = 294 (2) K
Block, yellow
0.48 × 0.32 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.950, T_{max} = 0.981
5800 measured reflections
2083 independent reflections
1688 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 28.0°
$h = -15 \rightarrow 15$
$k = -22 \rightarrow 15$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.083
S = 1.02
2083 reflections
234 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0084 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.88 (3)	1.81 (3)	2.682 (2)	171 (2)

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

H atoms bonded to C atoms were included in calculated positions [C—H = 0.93–0.96 Å] and refined using a riding-model approximation, with $U_{iso} = 1.2U_{eq}$(C) or $1.5U_{eq}$(methyl C). The H atom attached to O2 was located in a difference Fourier map and refined freely.

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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