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4-[(4-Hydroxy-3-methoxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one

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Key indicators

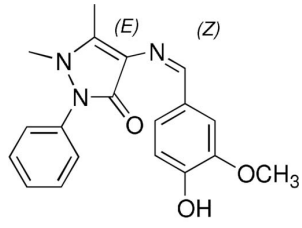
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.108
Data-to-parameter ratio = 9.7

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

In the crystal structure of the title compound, $C_{19}H_{19}N_3O_3$, the benzene ring substituted by hydroxy and methoxy groups is essentially coplanar with the pyrazoline ring [dihedral angle = 12.9 (2)°], whereas the phenyl ring bonded directly to the pyrazoline ring forms a dihedral angle of 50.4 (3)°.

Comment

Antipyrine (4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one) and its derivatives exhibit a wide range of biological activities and applications (Ismail, 2000; Yadav *et al.*, 2003). Antipyrine shows minimal protein binding and is rapidly and completely absorbed from the gastrointestinal tract and extensively metabolized by the cytochrome P450 liver enzymes (Poulsen & Loft, 1988). Owing to its low pK_a value and its small degree of plasma protein binding, antipyrine is distributed in total body water. On the other hand, related Schiff base ligands have demonstrated significant biological activity and new examples are being tested for their antitumor, antimicrobial and antiviral activity (Tarafder *et al.*, 2002; Çukurovali *et al.*, 2002; Ali *et al.*, 2002). As an extension of this work, a new Schiff base compound, (I), which was synthesized using antipyrine and vanillin (4-hydroxy-3-methoxybenzaldehyde) as starting materials, is reported here.



(I)

The molecular structure of (I) is shown in Fig. 1. In the structure, all bond lengths (Table 1) are in normal ranges (Montalvo-González & Ariza-Castolo, 2003). The dihedral angle between the C11–C16 phenyl and the pyrazoline ring is 50.4 (3)°, whereas the C1–C6 benzene ring substituted by hydroxy and methoxy groups is almost coplanar with the pyrazoline central ring [dihedral angle = 12.9 (2)°]. As expected, the molecule adopts a *trans* configuration about the C7═N3 bond. The C7═N3 bond length [1.273 (3) Å] conforms to the expected value for a normal C═N bond. A packing diagram of (I) is shown in Fig. 2. In the crystal structure, molecules interact through intermolecular O—H···O hydrogen bonds (Table 2).

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Experimental

All reagents used were of analytical grade from commercial sources and were used without further purification. Antipyrine (0.1 mmol, 20.3 mg) and vanillin (0.1 mmol, 15.2 mg) were dissolved in methanol (10 ml). The mixture was stirred for 30 min at 298 K to give a clear yellow solution. After allowing the resulting solution to evaporate in air for 10 d, yellow prismatic crystals of (I) were formed at the bottom of the vessel.

Crystal data

$C_{19}H_{19}N_3O_3$
M_r = 337.37
Orthorhombic, $P2_12_12_1$
a = 6.7467 (11) Å
b = 14.609 (2) Å
c = 17.785 (3) Å
V = 1752.9 (5) Å^3
Z = 4
D_x = 1.278 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1278 reflections
θ = 2.8–20.4°
μ = 0.09 mm^{-1}
T = 296 (2) K
Prism, yellow
0.48 × 0.17 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.959, T_{max} = 0.988
8650 measured reflections
2239 independent reflections
1549 reflections with $I > \sigma(I)$
R_{int} = 0.039
θ_{max} = 27.2°
$h = -8 \rightarrow 8$
$k = -18 \rightarrow 15$
$l = -22 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.108
S = 1.03
2239 reflections
230 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 0.076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C3—C7	1.460 (4)	C8—C10	1.426 (3)
C7—N3	1.273 (3)	C9—N2	1.356 (4)
C8—C9	1.361 (3)	C10—N1	1.382 (3)
C8—N3	1.398 (3)	N1—N2	1.391 (3)
O3—C6—C5	122.6 (2)	N1—C10—C8	105.5 (2)
N3—C7—C3	123.6 (2)	C10—N1—N2	109.3 (2)
C9—C8—N3	122.6 (2)	C10—N1—C12	123.5 (2)
C9—C8—C10	107.7 (2)	N2—N1—C12	120.6 (2)
N3—C8—C10	129.3 (2)	C9—N2—N1	107.0 (2)
N2—C9—C8	110.0 (2)	C9—N2—C17	124.8 (2)
N2—C9—C18	121.6 (2)	N1—N2—C17	119.2 (2)
C8—C9—C18	128.3 (3)	C7—N3—C8	120.1 (2)
O1—C10—N1	122.2 (2)	C6—O3—H3	109.5
O1—C10—C8	132.3 (2)		
C3—C7—N3—C8	175.2 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1^{i}	0.82	1.85	2.667 (3)	179

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

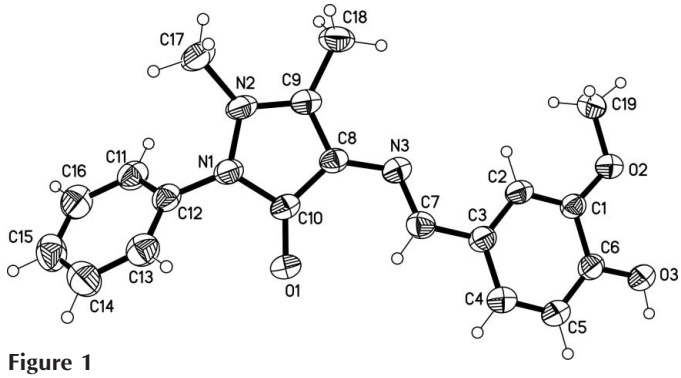

Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radii.

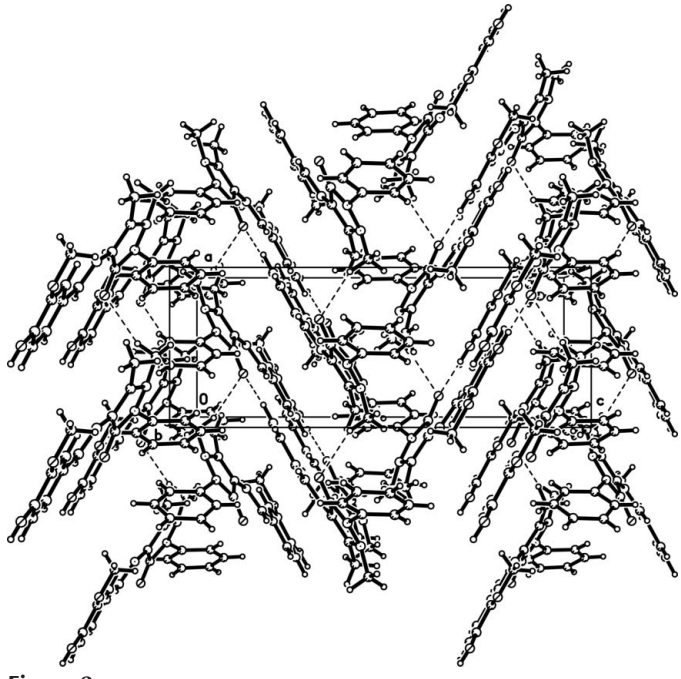

Figure 2
The crystal packing of (I), viewed along the *b* axis. Dashed lines indicate the hydrogen-bonding interactions.

H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms (O—H = 0.82 Å, aromatic C—H = 0.93 Å and methyl C—H = 0.96 Å), with $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic and $1.5U_{eq}(C,O)$ for OH and methyl H atoms.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SMART* **OR SAINT?**; data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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