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4-[(4-Hydroxybenzylidene)amino]-1,5-dimethyl-2-phenyl-2,3-dihydro-1*H*-pyrazol-3-one methanol solvate

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Key indicators

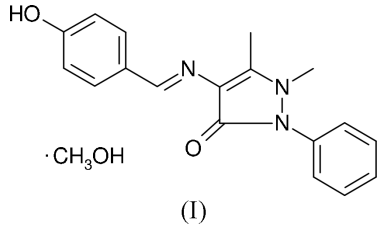
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.142
Data-to-parameter ratio = 13.7

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

The title compound, $C_{18}H_{17}N_3O_2\cdot CH_4O$, is a Schiff base compound, which is derived from the condensation of equimolar amounts of 4-aminoantipyrine and 4-hydroxybenzaldehyde. In the crystal structure, the MeOH molecules are linked to the Schiff base molecules through intermolecular O—H···O hydrogen bonds.

Comment

Antipyrine (2,3-dimethyl-1-phenyl-5-pyrazolone) and its derivatives exhibit a wide range of biological activities and applications (Ismail, 2000; Abd El Rehim *et al.*, 2001; 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). Estimates of half-life and systemic clearance of antipyrine have been used for the *in vivo* assessment of hepatic drug oxidation in different species (Koning & Cantilena, 1994). Owing to its low pK_a value and its small degree of plasma protein binding, antipyrine is distributed in total body water. Schiff base ligands have demonstrated significant biological activities and new examples are being tested for their antitumor, antimicrobial and antiviral activities (Tarafder *et al.*, 2002; Cukurovali *et al.*, 2002; Ali *et al.*, 2002). As an extension of work on the structural characterization of antipyrine derivatives, a new Schiff base compound, (I), is reported here.



(I)

All the bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and comparable to those observed in a similar antipyrine Schiff base (Liang *et al.*, 2002). The dihedral angle between the pyrazoline and C13–C18 phenyl ring is 123.3 (3)°. The C18—C13—N1—C9 and C14—C13—N1—N2 torsion angles are −106.7 (3) and −149.0 (3)°, respectively. Atom O1 deviates from the pyrazoline mean plane by 0.138 (3) Å, whereas atoms C11 and C12 deviate from it on the opposite side by 0.121 (7) and 0.576 (3) Å, respectively. The C7═N3 bond length of 1.271 (3) Å conforms to the value for a double bond. Because of conjugation through the imine double bond, C7═N3, the pyrazoline and C1–C6 substituted phenyl rings are approximately coplanar (mean deviation from the overall

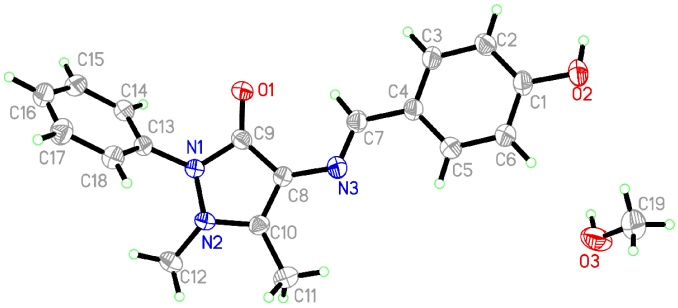


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

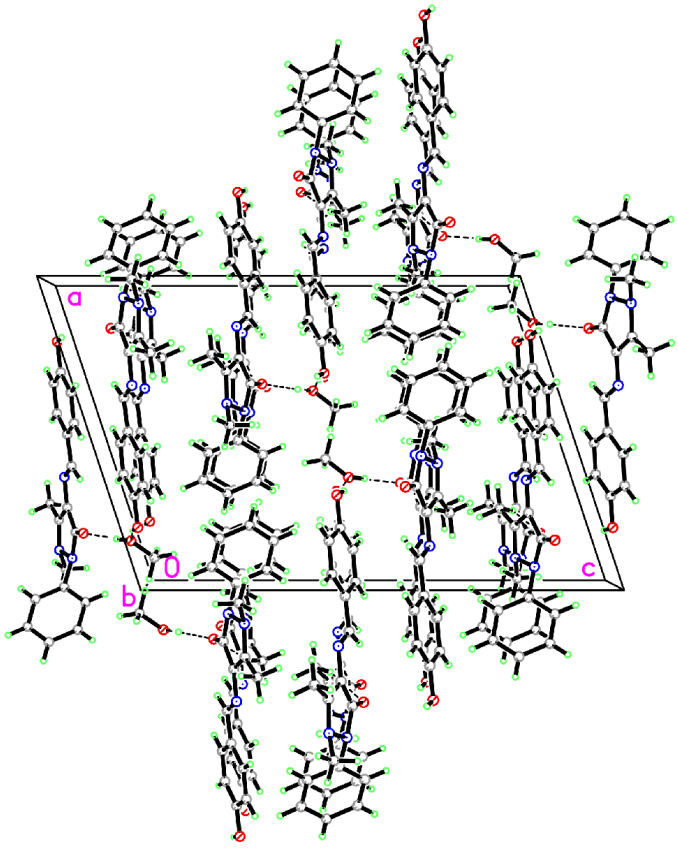


Figure 2
The crystal packing of (I), viewed along the *b* axis. Dashed lines indicate hydrogen bonds.

plane is 0.063 (3)°); the dihedral angle between the two rings is 8.2 (3)°. As expected, the molecular structure adopts a *trans* configuration about the C6═N3 bond.

In the crystal structure, the MeOH molecules are linked to the Schiff base molecules through intermolecular O—H···O hydrogen bonds.

Experimental

A mixture of 4-hydroxybenzaldehyde (0.1 mmol, 12.2 mg) and 4-aminoantipyrine (0.1 mmol, 20.3 mg) were dissolved in methanol (10 ml). The mixture was stirred for about 30 min at room temperature to give a clear yellow solution. After the solution had been kept in air for 7 d, yellow block-shaped crystals were formed at the bottom of the vessel on slow evaporation of the solvent. M.p. 445–446 K. Analysis found: C 67.17, H 6.31, N 12.32%; calculated for $C_{19}H_{21}N_3O_3$: C 67.24, H 6.24, N 12.38%.

Crystal data

$C_{18}H_{17}N_3O_2{\cdot}CH_4O$
M_r = 339.39
Monoclinic, $P2_1/n$
a = 13.680 (9) Å
b = 6.916 (5) Å
c = 19.968 (14) Å
β = 108.292 (11)°
V = 1794 (2) Å^3
Z = 4
D_x = 1.257 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1346 reflections
θ = 3.1–22.0°
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, yellow
0.43 × 0.24 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.964, T_{max} = 0.988
8910 measured reflections
3162 independent reflections
1498 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 25.0°
$h = -12 \rightarrow 16$
$k = -8 \rightarrow 8$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.142
S = 1.01
3162 reflections
231 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 0.0741P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3^{i}	0.82	1.86	2.668 (4)	166
O3—H3A···O1^{ii}	0.82	1.85	2.671 (3)	177

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

All H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H distances of 0.93–0.96 Å, O—H distances of 0.82 Å, and U_{iso}(H) = 1.2 or 1.5U_{eq}(C,O). The ratio of observed to unique reflections is low (47%), probably as a result of the poor diffraction quality of the crystal.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); 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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