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4-(3,4-Dihydroxybenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Yu-Xi Sun

Department of Chemistry, Qufu Normal University, Qufu 273165, People's Republic of China

Correspondence e-mail: yuxisun@163.com

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The title Schiff base compound, $C_{18}H_{17}N_3O_3$, was synthesized by the reaction of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one and 3,4-dihydroxybenzaldehyde in methanol solution. As expected, the compound adopts a *trans* configuration about the central C=N bond. In the crystal structure, adjacent molecules are linked through a pair of O—H···O hydrogen-bonding interactions.

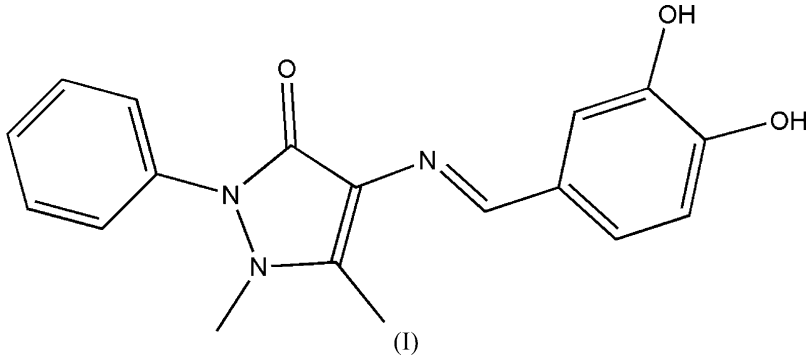
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.061
wR factor = 0.141
Data-to-parameter ratio = 14.8

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

Comment

The background to this study s described in the first paper of this series (Sun, 2006). As an extension of our work (Sun, Xie *et al.*, 2006; Sun, Zhang, Jin *et al.*, 2006; Sun, Zhang, Wang *et al.*, 2006) on the structural characterization of antipyrine derivatives, a new Shiff base compound, (I), is reported here.



(I)

The molecular structure is shown in Fig.1. All the bond distances and angles are in normal ranges, close to those observed in similar antipyrine Schiff bases. Atom O1 deviates from the pyrazoline mean plane by 0.109 (2) Å, whereas atom C10 and C11 deviate from it, on the opposite side, by 0.112 (2) and 0.399 (2) Å, respectively. The N2—N1—C1—C6 and C7—N1—C1—C2 torsion angles are −137.7 (2) and −97.5 (3)°, the pyrazoline ring and C1–C6 benzene ring planes are non-coplanar, the dihedral angle between them being 65.2 (3)°. The C12=N3 bond length of 1.281 (3) Å conforms to the value for a double bond. As a result of conjugation through the imino double bond, the pyrazoline and C13–C18 benzene rings are nearly coplanar [mean deviation from the overall combined mean plane is 0.090 (3) Å]; the dihedral angle between the pyrazoline ring and the C13–C18 benzene ring is 11.2 (3)°. As expected, the molecular structure of the Schiff base adopts a *trans* configuration about the central C12=N3 bond as do the other similar antipyrine derivatives that have been reported.

In the crystal structure, intramolecular hydrogen bonds exist between adjacent hydroxybenzene hydroxy groups and

intermolecular hydrogen bonds exist between adjacent molecules. The molecules stack along the *a* axis with no other short contacts apart from those described above (Table 1 and Fig. 2).

Experimental

All the chemicals were obtained from commercial sources and were used without purification. 4-Amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (0.2 mmol, 40.6 mg) and an equimolar quantity of 3,4-dihydroxybenzaldehyde (0.2 mmol, 27.6 mg) were dissolved in methanol (20 ml). The mixture was stirred for 30 min at room temperature to give a clear-yellow solution. This was kept in air for 10 d after which time yellow plate-shaped crystals of (I) were formed at the bottom of the vessel on slow evaporation of the methanol (yield 95.5%). Analysis calculated for $C_{18}H_{17}N_3O_3$: C 66.86, H 5.30, N 13.00%; found: C 66.81, H 5.31, N 12.96%.

Crystal data

$C_{18}H_{17}N_3O_3$
M_r = 323.35
Orthorhombic, *Pbca*
a = 6.8520 (5) Å
b = 16.8891 (13) Å
c = 27.204 (2) Å
V = 3148.1 (4) Å^3
Z = 8
D_x = 1.364 Mg m^{-3}
Mo *Kα* radiation
μ = 0.10 mm^{-1}
T = 295 (2) K
Thin plate, yellow
0.16 × 0.14 × 0.01 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.985, T_{max} = 0.999
23501 measured reflections
3265 independent reflections
2014 reflections with $I > 2\sigma(I)$
R_{int} = 0.078
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.141
S = 1.03
3265 reflections
221 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 1.5021P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 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}	0.82	1.83	2.640 (3)	171
O3—H3···O2	0.82	2.19	2.649 (3)	115

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

All H atoms were positioned geometrically (O—H = 0.82 Å and C—H = 0.93 or 0.96 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 for Csp^2 H atoms or 1.5U_{eq}(C) for methyl and hydroxy H atoms.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2002); data reduction: *SAINT-Plus*; 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*.

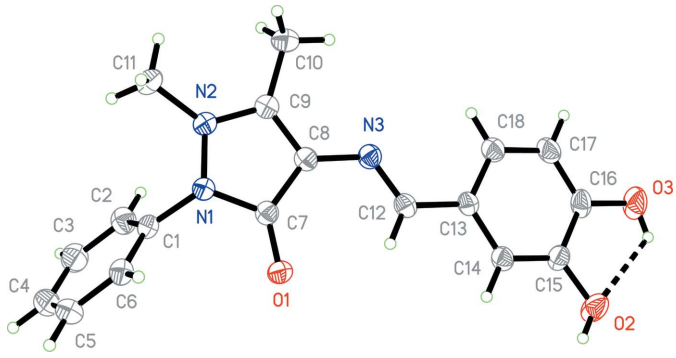


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. The O—H···O hydrogen bond is shown as a dashed line.

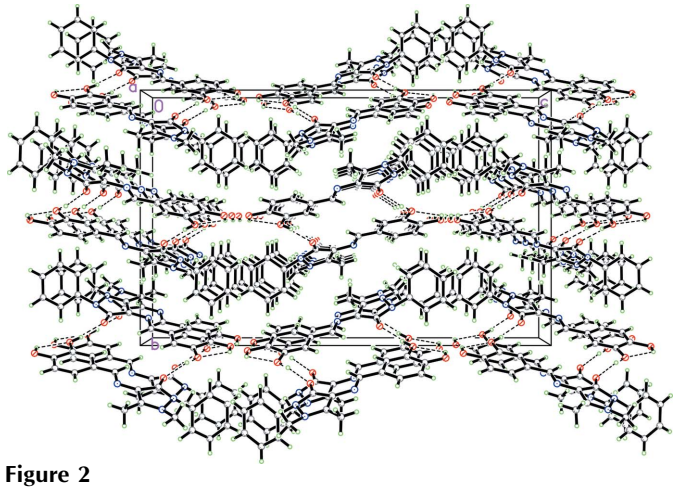


Figure 2
The packing of (I), viewed down the *a* axis. Intermolecular O—H···O hydrogen bonds are shown as dashed lines.

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