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1,5-Dimethyl-2-phenyl-4-[(1*E*)-(2,3,4-trihydroxybenzylidene)amino]-1*H*-pyrazol-3(2*H*)-one

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.004 Å
R factor = 0.060
wR factor = 0.138
Data-to-parameter ratio = 11.0

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

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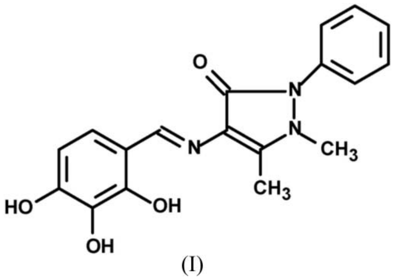
The title compound, $C_{18}H_{17}N_3O_4$, was synthesized by the condensation of 2,3,4-trihydroxybenzaldehyde with 4-aminoantipyrine. The molecule adopts a *trans* configuration about the central C=N double bond and exists in the phenol–imine form. Molecules are linked into a two-dimensional framework by intermolecular O—H···O hydrogen bonds.

Comment

Antipyrine (2,3-dimethyl-1-phenylpyrazol-5-one) and its derivatives are well known for their wide range of biological activities and applications (Yadav *et al.*, 2003). Moreover, antipyrine is also a multifunctional marker drug extensively used in studies on the capacity of hepatic oxidative metabolism (Marques *et al.*, 2002). As part of our continuing study of antipyrine derivatives, the title compound, (I), was synthesized and its crystal structure determined.



(I)

The molecule adopts a *trans* configuration about the central C=N double bond and exists in the phenol–imine form (Fig. 1). The bond distances and angles agree with the corresponding values found in similar compounds, namely, 4-salicylaldehydylaminoantipyrine, (II) (Liu *et al.*, 2002), and 4-[(1*E*)-(2,3-dihydroxybenzylidene)amino]-1,5-dimethyl-2-

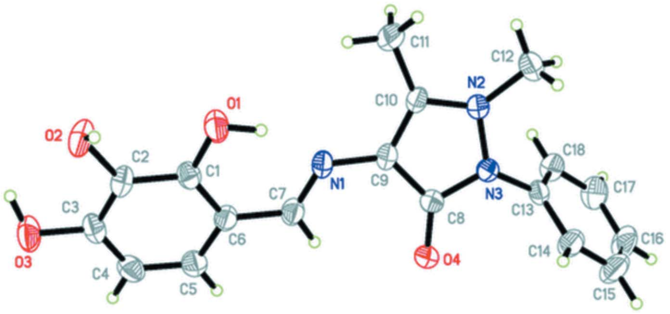


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radius.

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phenyl-2,3-dihydro-1*H*-pyrazol-3-one, (III) (Sun *et al.*, 2007). The dihedral angle between the pyrazolone ring and substituted benzene ring (atoms C1–C6) is 25.3 (2)°, close to the value of 26.9° found in (II) but different from the almost coplanar arrangement in (III).

A strong intramolecular O1—H1···N1 hydrogen bond is observed in the molecular structure of (I). In the crystal structure, molecules are linked by a pair of O3—H3···O2 hydrogen bonds into a centrosymmetric dimer (Fig. 2), and the dimers are cross-linked into a two-dimensional framework by intermolecular O2—H2···O4 hydrogen bonds (Fig. 3). In the crystal structure of (III) (Sun *et al.*, 2007), molecules are connected *via* intermolecular O—H···O hydrogen bonds into a zigzag chain structure.

Experimental

A mixture of 4-aminoantipyrine (2 mmol) and 2,3,4-trihydroxybenzaldehyde (2 mmol) in anhydrous ethanol (25 ml) was heated under reflux for 3 h. After cooling, the solvent was removed under reduced pressure and the solid residue was recrystallized from ethanol to yield the pure product. Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature. Analysis calculated for $C_{18}H_{17}N_3O_4$: C 63.71, H 5.05, N 12.38%; found: C 63.75, H 5.16, N 12.33%.

Crystal data

$C_{18}H_{17}N_3O_4$
M_r = 339.35
Monoclinic, $P2_1/n$
a = 12.916 (3) Å
b = 7.3123 (15) Å
c = 17.328 (4) Å
β = 101.85 (3)°
V = 1601.7 (6) $Å^3$
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 291 (2) K
0.20 × 0.17 × 0.17 mm

Data collection

Rigaku-R-AXIS-IV diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.980, T_{max} = 0.983
4561 measured reflections
2633 independent reflections
1994 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.138
S = 1.00
2633 reflections
240 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.26 e $Å^{-3}$

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{i}	0.92 (4)	2.13 (4)	2.931 (3)	144 (3)
O2—H2···O4^{ii}	0.93 (5)	1.76 (5)	2.673 (3)	170 (4)
O1—H1···N1	0.93 (4)	1.77 (4)	2.596 (3)	147 (3)

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

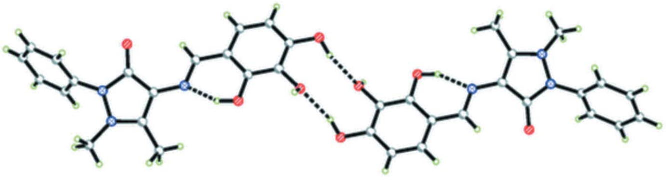

Figure 2
View of the O—H···O hydrogen-bonded (dashed lines) dimer in (I).

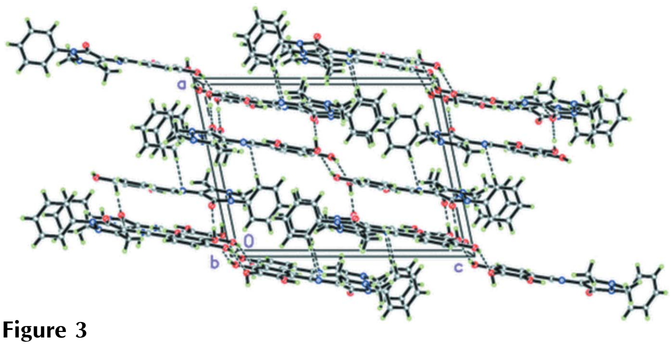

Figure 3
A packing diagram of (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$. The hydroxyl H atoms were refined freely along with an isotropic displacement parameter. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *R-AXIS* (Rigaku, 1996); cell refinement: *R-AXIS*; data reduction: *R-AXIS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1999); software used to prepare material for publication: *SHELXL97*'.

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