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4-[(1*E*)-(2-Hydroxy-5-nitrophenyl)methyleneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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The title compound, $C_{18}H_{16}N_4O_4$, has been synthesized by condensation of 5-nitrosalicylaldehyde and 4-aminoantipyrine. There are two molecules in the asymmetric unit. The crystal structure shows a strong intramolecular O—H···N hydrogen bond, which leads to the existence of a phenol–imine tautomer.

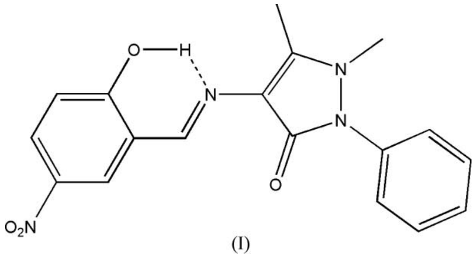
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.084
wR factor = 0.197
Data-to-parameter ratio = 12.3

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

Comment

Salicylaldehyde and its derivatives have attracted an intense interest because of their properties, such as tautomeric (Salman *et al.*, 1991), fluorescent (Morishige, 1980), and thermo- and photochromic properties (Barbara *et al.*, 1980; Cohen *et al.*, 1964). In a search for new analytical reagents, we report here the synthesis and crystal structure of the title compound, (I).



(I)

There are two molecules in the asymmetric unit. All the bond distances and angles are normal and agree with the corresponding values found in a similar compound, *viz.* 4-[(2-hydroxy-3-methoxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (Diao *et al.*, 2005). In each unique molecule of the title compound, the pyrazoline and the nitrophenyl ring are approximately coplanar as a

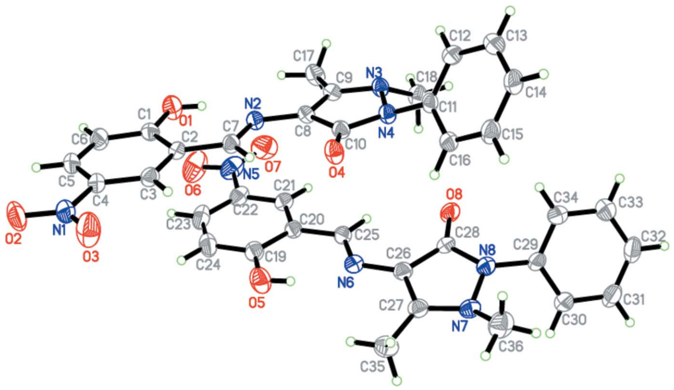


Figure 1
The asymmetric unit of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

result of conjugation *via* the imino C═N double bond, with dihedral angles of 3.6 (9) and 13.0 (2)°.

The imino N atoms are linked to the phenol O atoms and act as hydrogen-bond acceptors in O—H···N intramolecular interactions (Table 1), as in 4-{[(1*E*)-(2-hydroxyphenyl)methylidene]amino}-1,5-dimethyl-2-phenyl-2,3-dihydro-1*H*-pyrazol-3-one (Hökelek *et al.*, 2001).

Experimental

To 5-nitrosalicylaldehyde (10 mmol, 1.67 g) in MeOH (15 ml) was added 4-aminoantipyrine (10 mmol, 2.03 g) dissolved in MeOH (15 ml). The reaction mixture was refluxed on a water bath for 1 h. After cooling, the separated precipitate was filtered off, washed and recrystallized from methanol (yield 83%, m.p. 472.4–472.8 K).

Crystal data

$C_{18}H_{16}N_4O_4$
$M_r = 352.35$
Monoclinic, $P2_1/c$
$a = 8.4387\ (13)$ Å
$b = 36.870\ (5)$ Å
$c = 13.3937\ (16)$ Å
$\beta = 127.155\ (7)°$
$V = 3321.3\ (8)$ Å^3

$Z = 8$
$D_x = 1.409$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 298\ (2)$ K
Block, yellow
0.31 × 0.24 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.969$, $T_{\max} = 0.978$

17376 measured reflections
5837 independent reflections
4596 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.084$
$wR(F^2) = 0.197$
$S = 1.23$
5837 reflections
476 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 1.0761P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0041 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2	0.82	1.88	2.612 (3)	147
O5—H5···N6	0.82	1.86	2.586 (4)	148

All H atoms were positioned geometrically and were treated as riding on their parent C and O atoms, with aromatic C—H distances of 0.93 Å [$U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C)], other C—H distances of 0.96 Å [$U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C)] and O—H distances of 0.82 Å [$U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O)].

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXL97*.

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