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

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Key indicators

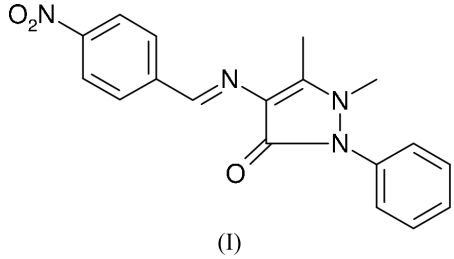
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.010 Å
R factor = 0.084
wR factor = 0.232
Data-to-parameter ratio = 11.9

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

In the title molecule, $C_{18}H_{16}N_4O_3$, the substituted *p*-nitrophenyl ring is almost coplanar with the pyrazoline ring [dihedral angle = 8.2 (4)°], whereas the phenyl ring directly attached to the pyrazoline ring forms a dihedral angle of 54.5 (2)°.

Comment

Antipyrine (2,3-dimethyl-1-phenylpyrazol-5-one) 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 activity and new examples are being tested for their antitumor, antimicrobial, and antiviral activity (Tarafder *et al.*, 2002; Cukurovali *et al.*, 2002; Ali *et al.*, 2002). As an extension of our work, a new antipyrine derivative, (I), is reported here.



(I)

The bond lengths and angles of the antipyrine moiety are in normal ranges (Allen *et al.*, 1987), comparable to those observed in a similar antipyrine Schiff base (Liang *et al.*, 2002). The dihedral angle between the pyrazoline ring and the C13–C18 phenyl ring is 54.5 (2)°. The torsion angles N2—N1—C13—C18 and C1—N1—C13—C14 are 146.5 (5) and 110.1 (7)°, respectively. Atom O1 deviates from the pyrazoline mean plane by 0.112 (9) Å, whereas C4 and C5 deviate from it, on the other side, by 0.133 (9) and 0.373 (10) Å, respectively. Because of conjugation through the imino double bond C6═N3, the pyrazoline ring and the *p*-nitrophenyl ring are nearly coplanar (r.m.s. deviation from the combined mean plane is 0.065 Å); the dihedral angle between the two rings is 8.2 (4)°. As expected, the molecule adopts a *trans* configuration about the C6═N3 bond.

In the crystal structure, the molecules are linked together by intermolecular C—H···O hydrogen bonds, forming two-dimensional sheets (Fig. 2 and Table 1).

Experimental

4-Nitrobenzaldehyde (0.1 mmol, 15.1 mg) and 4-aminoantipyrine (0.1 mmol, 20.3 mg) were dissolved in methanol (10 ml). The mixture was stirred for 30 min at room temperature to give a clear yellow solution. After allowing the resulting solution to stand in air for 7 d, yellow block-shaped crystals of (I) were formed at the bottom of the vessel by slow evaporation of the solvent.

Crystal data

$C_{18}H_{16}N_4O_3$
M_r = 336.35
Triclinic, $P\overline{1}$
a = 7.003 (2) Å
b = 9.537 (4) Å
c = 12.540 (3) Å
α = 100.397 (15)°
β = 98.808 (15)°
γ = 94.549 (15)°
V = 809.2 (4) Å^3
Z = 2
D_x = 1.380 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 569 reflections
θ = 2.5–22.4°
μ = 0.10 mm^{-1}
T = 298 (2) K
Block, yellow
0.45 × 0.32 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.958, T_{max} = 0.982
3931 measured reflections
2703 independent reflections
958 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 25.0°
$h = -8 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -10 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.084
$wR(F^2)$ = 0.232
S = 0.93
2703 reflections
228 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O1	0.93	2.38	3.046 (7)	128
C9—H9···O3^{i}	0.93	2.55	3.479 (9)	175
C11—H11···O1^{ii}	0.93	2.52	3.225 (8)	133

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

All H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.96 Å, and with U_{iso}(H) = 1.2 or 1.5U_{eq}(C). As a result of the large fraction of weak data at higher angles, the 2θ maximum was limited to 50°. Even with this limitation, the coverage is only 94.2% complete and the ratio of observed to unique reflections is low (35%), owing to 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: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

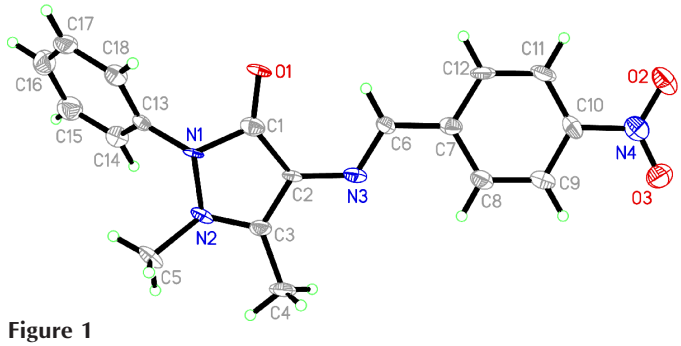


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

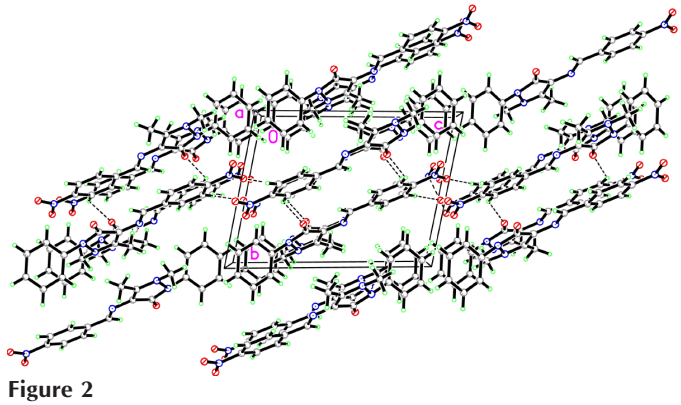


Figure 2
The crystal packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

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