Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 1,5-Dimethyl-2-phenyl-4-[(*E*,*E*)-3-phenylprop-2-enylideneamino]-1*H*-pyrazol-3(2*H*)-one

**Zong-Xiao Li* and Xin-Li Zhang**

Department of Chemistry, Baoji College of Arts and Sciences, Baoji 721007, People's Republic of China

Correspondence e-mail: baojizhangxinli@163.com

**Key indicators**

Single-crystal X-ray study
*T* = 298 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.049
*wR* factor = 0.145
Data-to-parameter ratio = 14.5

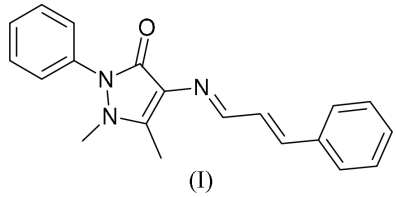
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 6 January 2005
Accepted 13 January 2005
Online 22 January 2005

The title compound, $C_{20}H_{19}N_3O$, is a Schiff base compound derived from 4-aminoantipyrine and cinnamaldehyde. As expected, the molecular structure adopts a *trans* configuration about the central C=N double bond.

## 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)

Compound (I) is an antipyrine derivative (Fig. 1). All the bond lengths and angles are in the normal ranges (Allen *et al.*,

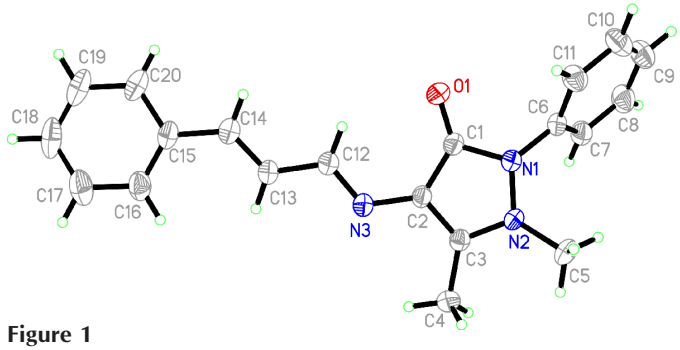



**Figure 1**
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

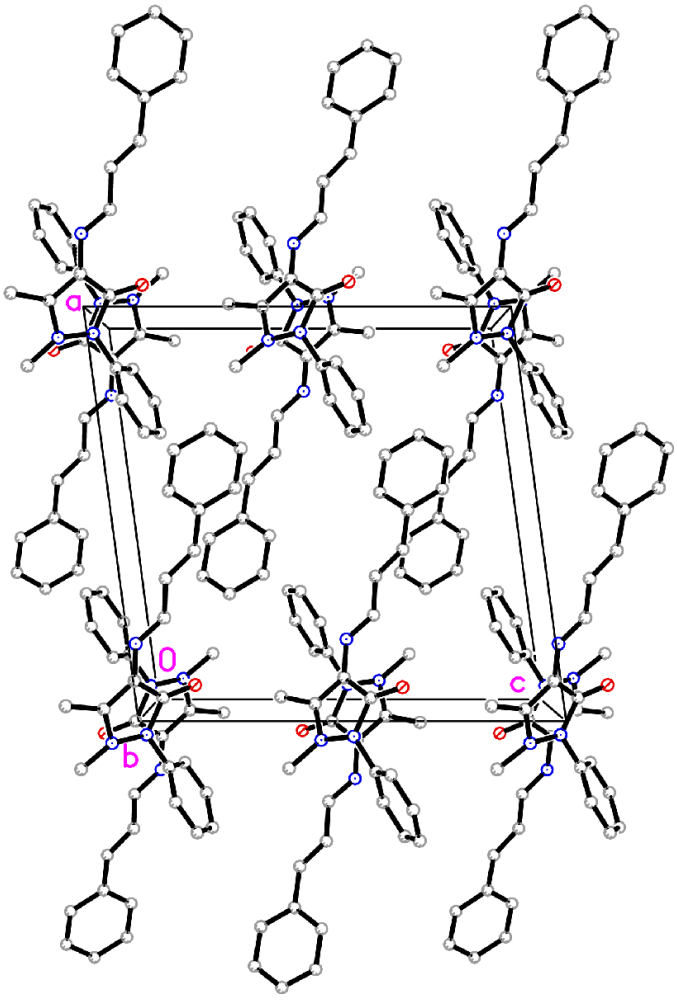



**Figure 2**
The crystal packing of (I), viewed along the *b* axis. H atoms have been omitted.

1987) and comparable to those observed in a similar antipyrine Schiff base, 1,5-dimethyl-4-{[(1-oxypyridin-2-yl)methylene]amino}-2-phenyl-1,2-dihydro-3*H*-pyrazol-3-one (Liang *et al.*, 2002). The dihedral angle between the pyrazoline and C6–C11 phenyl rings is 54.7 (2)°. The torsion angle N3—C12—C13—C14 is 179.6 (2)°. Atom O1 deviates from the pyrazoline mean plane by 0.120 (3) Å, whereas atoms C4 and C5 deviate from it on the opposite side by 0.098 (3) and 0.582 (3) Å, respectively. Because of conjugation through the C12═N3 and C13═C14 double bonds, the pyrazoline and C15—C20 phenyl rings are approximately coplanar [mean deviation from the overall plane is 0.026 (3) Å]; the dihedral angle between the two rings is 9.6 (3)°. As expected, the molecular structure adopts a *trans* configuration about the C12═N3 bond. In the crystal structure of (I) (Fig. 2), the molecules stack along the *b* axis and there are two rather short contacts (Table 1).

## Experimental

A mixture of cinnamaldehyde (0.1 mmol, 13.2 mg) and 4-aminoantipyrine (0.1 mmol, 20.3 mg) was dissolved in methanol (10 ml). The mixture was stirred for about 30 min at room temperature to give a clear yellow solution. After allowing the solution to stand in air for 5 d, yellow block-shaped crystals were formed at the bottom of the vessel on slow evaporation of the solvent (yield 71.8%). Analysis found: C 75.81, H 6.12, N 13.10%; calculated for $C_{20}H_{19}N_3O$: C 75.69, H 6.03, N 13.24%.

*Crystal data*

$C_{20}H_{19}N_3O$
$M_r$ = 317.38
Monoclinic, $P2_1/c$
$a$ = 13.480 (3) Å
$b$ = 9.796 (2) Å
$c$ = 13.757 (3) Å
$\beta$ = 97.556 (3)°
$V$ = 1800.8 (7) Å$^3$
$Z$ = 4
$D_x$ = 1.171 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 1749 reflections
$\theta$ = 2.6–22.6°
$\mu$ = 0.07 mm$^{-1}$
$T$ = 298 (2) K
Block, yellow
0.46 × 0.43 × 0.17 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.967, $T_{max}$ = 0.988
9060 measured reflections
3172 independent reflections
1740 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.044
$\theta_{max}$ = 25.0°
$h = -16 \rightarrow 15$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 15$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.145
$S$ = 1.01
3172 reflections
219 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.4155P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C4—H4$A$···O1$^{i}$ | 0.96 | 2.33 | 3.279 (3) | 168 |
| C12—H12···O1 | 0.93 | 2.36 | 3.038 (3) | 129 |

Symmetry code: (i) $x, \frac{1}{2}-y, \frac{1}{2}+z$.

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 and with $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$(C). The structure contains solvent-accessible voids of 57 Å$^3$, which might accommodate a disordered water molecule.

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*.

The authors are grateful for research grant (No. 02js40) from the Phytochemistry Key Laboratory of Shanxi Province.

## References

Abd El Rehim, S. S., Ibrahim, M. A. M. & Khalid, K. F. (2001). *Mater. Chem. Phys.* **70**, 268–273.
Ali, M. A., Mirza, A. H., Butcher, R. J., Tarafder, M. T. H., Keat, T. B. & Ali, A. M. (2002). *J. Inorg. Biochem.* **92**, 141–148.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Bruker (1998). *SMART* (Version 5.628) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
Cukurovali, A., Yilmaz, I., Özmen, H. & Ahmedzade, M. (2002). *Transition Met. Chem.* **27**, 171–176.
Ismail, K. Z. (2000). *Transition Met. Chem.* **25**, 522–528.
Koning, P. K. & Cantilena, L. (1994). *Ann. Int. Met.* **154**, 590–591.
Liang, H., Yu, Q. & Hu, R.-X. (2002). *Transition Met. Chem.* **27**, 454–457.
Poulsen, H. E. & Loft, S. (1988). *J. Hepatol.* **6**, 374–378.
Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXL97* and *SHELXS97.* University of Göttingen, Germany;
Sheldrick, G. M. (1997*b*). *SHELXTL.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Tarafder, M. T. H., Jin, K. T., Crouse, K. A., Ali, A. M., Yamin, B. M. & Fun, H.-K. (2002). *Polyhedron*, **21**, 2547–2554.
Yadav, P. N., Demertzis, M. A., Kovala-Demertzi, D., Skoulika, S. & West, D. X. (2003). *Inorg. Chim. Acta*, **349**, 30–36.