Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A new antipyrine Schiff base: 4-(5-chloro-2-hydroxybenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Zong-Xiao Li,* Xin-Li Zhang and Xiao-Ling Wang

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

Correspondence e-mail: lizongxiao1116@tom.com

Received 21 August 2006
Accepted 6 September 2006

Key indicators

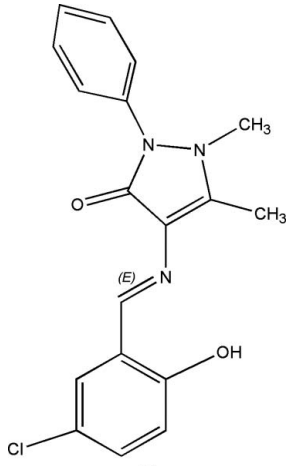
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.126
Data-to-parameter ratio = 13.4

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

The title compound, $C_{18}H_{16}ClN_3O_2$, has been synthesized by the reaction of 5-chlorosalicylaldehyde and 4-aminoantipyrine. The molecule adopts an *E* configuration about the central C=N double bond. Intramolecular hydrogen bonds and intra- and intermolecular C—H···O interactions are observed.

Comment

The roles of antipyrine and antipyridine derivatives in biological processes have become a topic of study in recent years (Carlton *et al.*, 1995; Coolen *et al.*, 1999; Jiang *et al.*, 2000). The title compound, (I), is a new antipyrine Schiff base compound prepared in our laboratory. We report its structure here.



(I)

All bond lengths and angles in (I) are in agreement with those in similar antipyrine Schiff bases. The pyrazoline ring is essentially planar. The dihedral angle between the pyrazoline ring and the C4–C9 phenyl ring is 125.9 (5)°. The N2—N1—C4—C5 and C1—N1—C4—C9 torsion angles are 149.7 (2) and 109.4 (3)°, respectively. Atom O1 at C1 and the methyl group at N2 deviate from the pyrazoline mean plane by 0.135 (3) and 0.652 (7) Å, respectively. The methyl group at C3 is essentially coplanar with the pyrazoline mean plane, with a deviation of 0.086 (8) Å. As a result of the conjugation through the imino C12=N3 double bond, the pyrazoline and the C13–C18 aryl ring are approximately coplanar, with a dihedral angle of 5.2 (5)°. Atom Cl1 and the hydroxyl group are essentially coplanar with their benzene ring.

Intramolecular hydrogen bonding between the hydroxyl group and the imine N atom, and intra- and intermolecular C—H···O interactions are observed (Table 1). The molecules stack along the *b* axis and there are two rather short contacts.

© 2006 International Union of Crystallography
All rights reserved

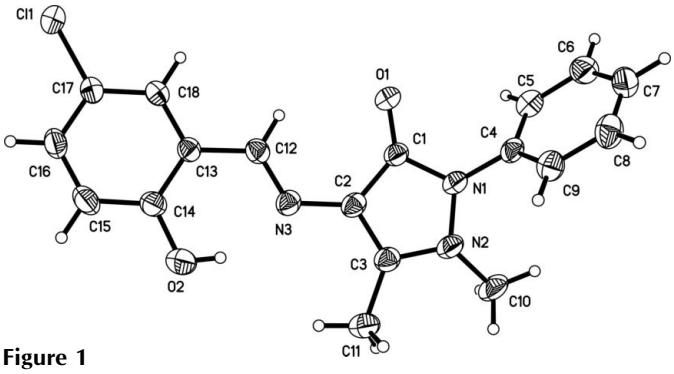


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

5-Chlorosalicylaldehyde (0.1 mmol, 15.7 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, giving a clear brown solution. After allowing this solution to stand in air for 7 d, brown block-shaped crystals of (I) were formed by slow evaporation of the solvent. The crystals were collected, washed with methanol and dried in a vacuum desiccator using anhydrous $CaCl_2$ (yield 54%). Analysis found: C 63.21, H 4.73%; calculated for $C_{18}H_{16}ClN_3O_2$: C 63.25, H 4.71%.

Crystal data

$C_{18}H_{16}ClN_3O_2$
M_r = 341.79
Monoclinic, $P2_1/n$
a = 14.426 (7) Å
b = 6.876 (3) Å
c = 17.555 (9) Å
β = 109.135 (6)°
V = 1645.1 (14) Å^3
Z = 4
D_x = 1.380 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 298 (2) K
Block, brown
0.53 × 0.38 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.880, T_{max} = 0.943
8128 measured reflections
2898 independent reflections
1814 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.126
S = 1.03
2898 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.465P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N3	0.82	1.86	2.589 (3)	147

All H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H = 0.93–0.96 Å, O—H = 0.82 Å; U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

The authors thank the Phytochemistry Key Laboratory of Shaanxi Province for a research grant (No. 02js40).

References

Bruker (1998). *SMART* (Version 5.0), *SAINT* Version 4.0) and *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.
Carlton, L. D., Schmith, V. D. & Brouwer, K. L. R. (1995). *Prostaglandins*, **50**, 341–347.
Coolen, S. A. J., Everaerts, F. M. & Huf, F. A. (1999). *J. Chromatogr. B*, **732**, 103–113.
Jiang, S. X., Bayon, J. E., Ferre, I., Mao, X. Z. & Gonzalez-Gallego, J. (2000). *Vet. Parasitol.* **88**, 177–186.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.