Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(3-Chlorobenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Ran Zhang and Yu-Xi Sun*

Department of Chemistry, Qufu Normal University, Qufu 273165, People's Republic of China

Correspondence e-mail: yuxisun@163.com

Received 9 November 2006
Accepted 20 November 2006

The title Schiff base compound, $C_{18}H_{16}ClN_3O$, was synthesized by the reaction of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one and 3-chlorobenzaldehyde in methanol solution. As expected, the compound adopts a *trans* configuration about the central C=N bond. The asymmetric unit contains two independent molecules. In the crystal structure, the molecules stack with no short contacts.

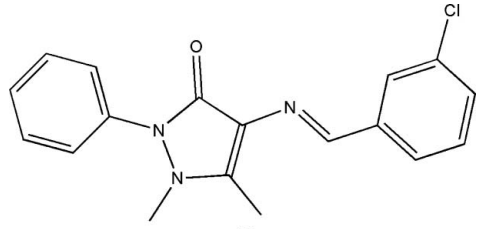
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.060
wR factor = 0.157
Data-to-parameter ratio = 16.3

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

Comment

Antipyrine and its derivatives exhibit a wide range of biological activities and applications (Yadav *et al.*, 2003; Ismail, 2000; Abd El Rehim *et al.*, 2001). A few crystal structures of antipyrine derivatives have been investigated (Liang *et al.*, 2002; Li & Zhang, 2004, 2005; Zhang & Li, 2005; You, *et al.*, 2004, 2006; Wen, 2005). Schiff bases of salicyladehyde have demonstrated significant biological activity and new examples are being tested for their antitumor, antimicrobial and antiviral activity (Tarafder *et al.*, 2002; Çukurovalı *et al.*, 2002; Ali *et al.*, 2002). As an extension of our work (Sun, Xie *et al.*, 2006; Sun, Zhang, Jin *et al.*, 2006; Sun, Zhang, Wang *et al.*, 2006) on the structural characterization of antipyrine derivatives, a new Schiff base compound, (I), is reported here.



(I)

The asymmetric unit of (I) consists of two independent molecules (Fig. 1). In both of these, all the bond distances and angles are in normal ranges, close to those observed in similar antipyrine Schiff bases cited above. The dihedral angle between the N1/N2/C9/C8/C7 pyrazoline ring and the C1–C6 phenyl ring planes is 55.3 (3)° and that between the N4/N5/C27/C26/C25 pyrazoline ring and the C19–C24 phenyl ring planes is 77.5 (3)°. Atom O1 deviates from the pyrazoline mean plane by 0.130 (2) Å, whereas atoms C10 and C11 deviate from it, on the opposite side, by 0.149 (2) and 0.514 (2) Å, respectively. Atom O2 deviates from the other pyrazoline mean plane by 0.127 (2) Å, whereas atoms C28 and C29 deviate from it, on the opposite side, by 0.162 (2) and 0.428 (2) Å, respectively. The N2—N1—C1—C2, C7—N1—C1—C6, N5—N4—C19—C20 and C25—N4—C19—C24 torsion angles are 148.1 (2), 109.5 (3), −146.3 (2) and

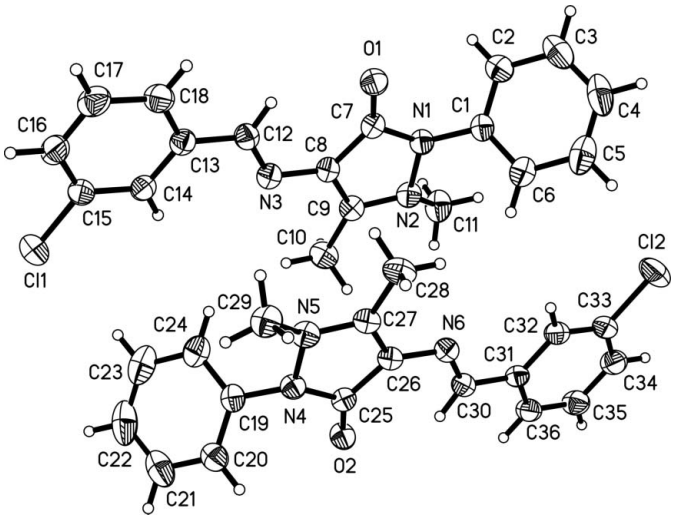


Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

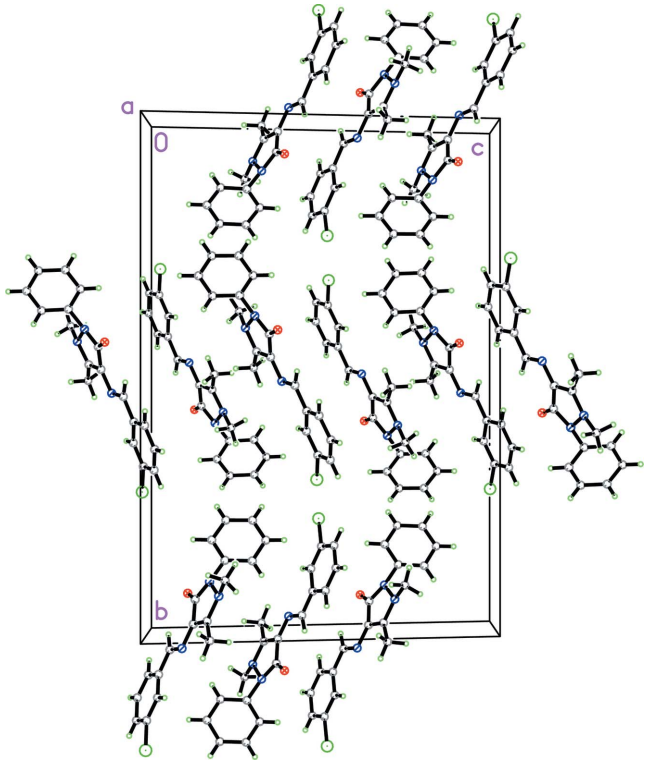


Figure 2
The crystal packing of (I), viewed down the *a* axis.

−107.3 (3)°. The C12═N3 and C30═N6 bond lengths of 1.280 (3) and 1.279 (3) conform to the value for double bonds. As a result of conjugation through the imino double bond, the pyrazoline and C13–C18 and C31–C36 benzene ring systems in both molecules are nearly coplanar, the dihedral angle between the N1/N2/C7/8/C9 pyrazoline ring and the C13–C18 benzene ring being 6.7 (4)° [mean deviation from the combined mean plane is 0.047 (4) Å] and that between the N4/N5/C25/C26/C27 pyrazoline ring and the C31–C36 benzene ring being 8.4 (4)° [mean deviation from the combined mean plane is 0.065 (4) Å]. As expected, the two molecules adopt the same *trans* configurations about the C12═N3 and C30═N6 bonds as in the other similar antipyrine derivatives that have reported.

In the crystal structure, the molecules stack along the *a* axis with no short contacts (Fig. 2).

Experimental

All the chemicals were obtained from commercial sources and used without purification. 4-Amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazole-3-one (0.2 mmol, 40.6 mg) and an equimolar quantity of 3-chlorobenzaldehyde (0.2 mmol, 28.1 mg) were dissolved in methanol (20 ml). The mixture was stirred for 30 min at room temperature to give a clear yellow solution. This solution was kept in air for 9 d, after which time yellow plate-shaped crystals of (I) were formed at the bottom of the vessel on slow evaporation of the methanol (yield 96.3%). Analysis calculated for $C_{18}H_{16}ClN_3O$: C 66.36, H 4.95, N 12.90%; found: C 66.23, H 4.94, N 12.93%.

Crystal data

$C_{18}H_{16}ClN_3O$
M_r = 325.79
Monoclinic, $P2_1/c$
a = 6.7580 (5) Å
b = 26.7540 (19) Å
c = 18.5240 (13) Å
β = 99.6200 (10)°
V = 3302.1 (4) Å^3
Z = 8
D_x = 1.311 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 298 (2) K
Plate, yellow
0.21 × 0.18 × 0.08 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.992, T_{max} = 0.995
25598 measured reflections
6822 independent reflections
4241 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.157
S = 1.02
6822 reflections
419 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.9648P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

All H atoms were positioned geometrically (C—H = 0.93 or 0.96 Å) and constrained to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms or U_{iso}(H) = 1.2U_{eq}(C) for other H atoms.

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

The work was supported by the National Natural Science Foundation of China (grant Nos. 30270245 and 30470247) and Qufu Normal University for Science and Technology.

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