

Yu-Xi Sun,<sup>a\*</sup> Xue-Hui Xie,<sup>b</sup> Ran Zhang,<sup>a</sup> Li-Feng Zhang<sup>a</sup> and Lai-Xiang Xu<sup>b</sup><sup>a</sup>Department of Chemistry, Qufu Normal University, Qufu 273165, People's Republic of China, and <sup>b</sup>College of Life Science, Qufu Normal University, Qufu 273165, People's Republic of China

Correspondence e-mail: yuxisun@163.com

## Key indicators

Single-crystal X-ray study  
 $T = 295\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
Disorder in solvent or counterion  
 $R$  factor = 0.059  
 $wR$  factor = 0.176  
Data-to-parameter ratio = 14.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

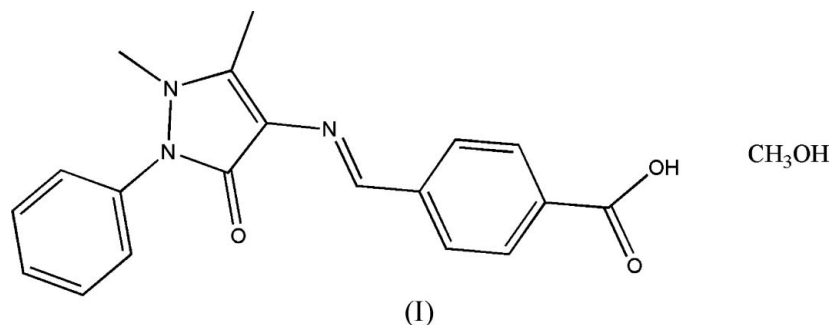
## 4-[(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)iminomethyl]benzoic acid methanol solvate

The title compound,  $\text{C}_{19}\text{H}_{17}\text{N}_3\text{O}_3 \cdot \text{CH}_3\text{OH}$ , was synthesized by the reaction of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one and 4-formylbenzoic acid in methanol solution. As expected, the antipyrine derivative adopts a *trans* configuration about the central  $\text{C}=\text{N}$  bond. In the crystal structure, the molecules are linked into chains along the  $[01\bar{1}]$  direction by  $\text{O}-\text{H} \cdots \text{O}$  hydrogen bonds.

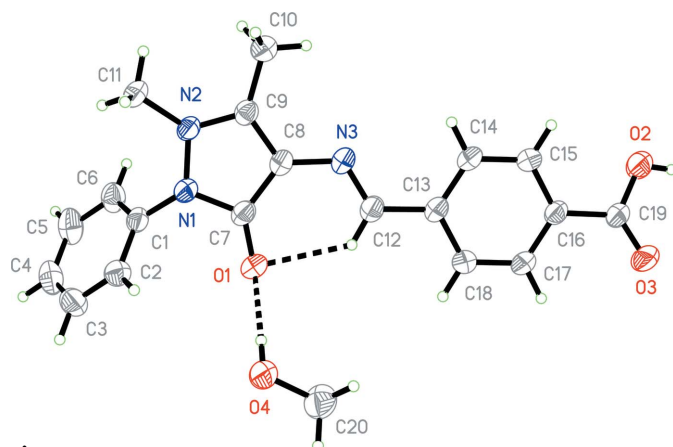
Received 18 September 2006  
Accepted 25 September 2006

## 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). The crystal structures of antipyrine derivatives have also been investigated (Liang *et al.*, 2002; Li & Zhang, 2004, 2005; Zhang & Li, 2005; You *et al.*, 2004, 2006; Wen, 2005). Schiff bases of salicylaldehyde 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 (Sun *et al.*, 2006) on the structural characterization of antipyrine derivatives, a new Schiff base compound, (I), is reported here.

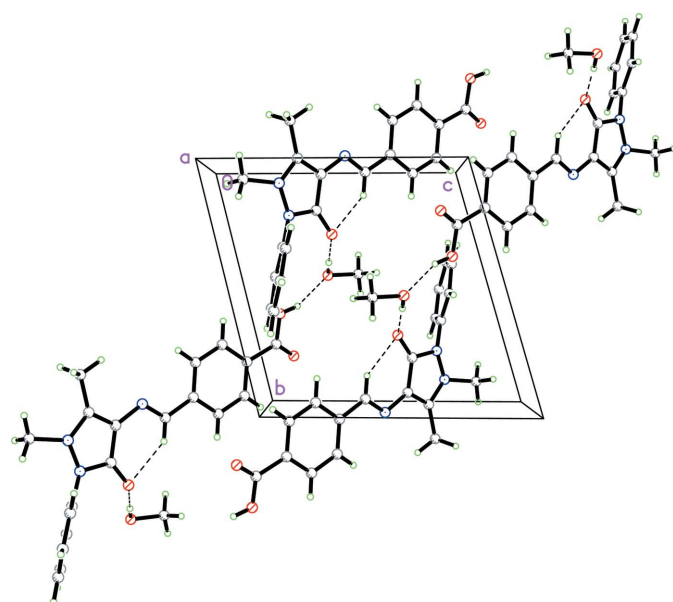


The asymmetric unit of (I) consists of a Schiff base molecule and a methanol solvent molecule (Fig. 1). The bond lengths and angles in the main molecule are in normal ranges, close to those observed in similar antipyrine Schiff bases cited above. Atom O1 deviates from the pyrazoline mean plane by 0.104 (2) Å, whereas atoms C10 and C11 deviate from it, on the opposite side, by 0.146 (2) and 0.502 (3) Å, respectively. The dihedral angle between the N1/N2/C7–C9 pyrazoline ring and the C1–C6 benzene ring is 52.19 (8)°. The C12=N3 bond length of 1.268 (2) Å conforms to the value for a double bond. As a result of conjugation through the imino double bond, the pyrazoline and C13–C18 benzene rings are nearly coplanar [mean deviation from the combined mean plane is 0.054 (4) Å]; the dihedral angle between the pyrazoline ring



**Figure 1**

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate hydrogen bonds. Only the major component of the disordered methanol molecule is shown.



**Figure 2**

The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines. Only the major component of the disordered methanol molecule is shown.

and the C13–C18 benzene ring is 8.0 (1)°. As expected, the Schiff base adopts a *trans* configuration about the central C12=N3 bond.

In the crystal structure, the methanol and Schiff base molecules are connected by strong intermolecular O4–H4···O1 and O2–H2···O4<sup>i</sup> [symmetry code is given in Table 1] hydrogen bonds to form one-dimensional chains along the [011] direction. In addition, weak C–H···O interactions are observed (Table 1).

## Experimental

All the chemicals were obtained from commercial sources and used without purification. 4-Amino-1,5-dimethyl-2-phenyl-1,2-dihydro-

pyrazol-3-one (2 mmol, 0.406 g) and an equimolar quantity of 4-formylbenzoic acid (2 mmol, 0.300 g) were dissolved in methanol (20 ml). The mixture was stirred for 30 min at room temperature to give a clear yellow solution. The resulting solution was kept in air for 15 d, during which time yellow block-shaped crystals of (I) were formed at the bottom of the vessel on slow evaporation of the methanol (yield 95.7%). Analysis found: C 65.29, H 5.73, N 11.39%; calculated for C<sub>20</sub>H<sub>21</sub>N<sub>3</sub>O<sub>4</sub>: C 65.38, H 5.76, N 11.44%.

## Crystal data

C<sub>19</sub>H<sub>17</sub>N<sub>3</sub>O<sub>3</sub>·CH<sub>4</sub>O  
*M<sub>r</sub>* = 367.40  
 Triclinic, *P* $\bar{1}$   
*a* = 9.4270 (7) Å  
*b* = 10.2516 (8) Å  
*c* = 10.3014 (8) Å  
 $\alpha$  = 74.450 (1)°  
 $\beta$  = 85.673 (1)°  
 $\gamma$  = 75.665 (1)°

*V* = 929.20 (12) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.313 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 $\mu$  = 0.09 mm<sup>-1</sup>  
*T* = 295 (2) K  
 Block, yellow  
 0.45 × 0.40 × 0.30 mm

## Data collection

Bruker APEX area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.959, *T<sub>max</sub>* = 0.973

6972 measured reflections  
 3760 independent reflections  
 2595 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.024  
 $\theta_{\max}$  = 26.5°

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.059  
*wR* (*F*<sup>2</sup>) = 0.176  
*S* = 1.09  
 3760 reflections  
 259 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0943P)^2 + 0.0586P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.38 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{Å}^{-3}$   
 Extinction correction: SHELXL97  
 Extinction coefficient: 0.029 (7)

**Table 1**

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O2–H2···O4 <sup>i</sup>	0.82	1.75	2.513 (3)	153
O2–H2···O4 <sup>ii</sup>	0.82	2.00	2.796 (6)	165
O4–H4···O1	0.82	1.89	2.712 (3)	177
C11–H11C···O3 <sup>ii</sup>	0.96	2.27	3.209 (3)	166
C12–H12···O1	0.93	2.26	2.966 (3)	132

Symmetry codes: (i) *x*, *y* + 1, *z* – 1; (ii) *x* – 1, *y*, *z* + 1.

The methanol molecule is disordered over two orientations with site-occupancy factors of 0.675 (3) and 0.325 (3), respectively. The same anisotropic displacement parameters were used for the atom pairs C20/C20' and O4/O4'. The C–O bond lengths were restrained to be 1.43 (1) Å. All H atoms were placed in geometrically idealized positions [O–H = 0.82 Å, and C–H = 0.93 or 0.96 Å] and constrained to ride on their parent atoms, with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C) for Csp<sup>2</sup>–H or 1.5*U*<sub>eq</sub>(C,O) 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: SHELXL97.

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

## 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.
- Bruker (2002). *SMART* (Version 5.628), *SAINT-Plus* (Version 6.22) and *SHELXTL* (Version 6.10). 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.
- Liang, H., Yu, Q. & Hu, R.-X. (2002). *Transition Met. Chem.* **27**, 454–457.
- Li, Z.-X. & Zhang, X.-L. (2004). *Acta Cryst. E* **60**, o2199–o2200.
- Li, Z.-X. & Zhang, X.-L. (2005). *Acta Cryst. E* **61**, o375–o377.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sun, Y.-X., Zhang, R., Jin, Q.-M., Zhi, X.-J. & Lü, X.-M. (2006). *Acta Cryst. C* **62**, o467–o469.
- Tarafder, M. T. H., Jin, K. T., Crouse, K. A., Ali, A. M., Yamin, B. M. & Fun, H.-K. (2002). *Polyhedron*, **21**, 2547–2554.
- Wen, P. (2005). *Acta Cryst. E* **61**, o2918–o2920.
- Yadav, P. N., Demertzis, M. A., Kovala-Demertzi, D., Skoulika, S. & West, D. X. (2003). *Inorg. Chim. Acta*, **349**, 30–36.
- You, Z.-L., Wang, J. & Chi, J.-Y. (2006). *Acta Cryst. E* **62**, o1652–o1653.
- You, Z.-L., Zhu, H.-L. & Liu, W.-S. (2004). *Acta Cryst. E* **60**, o801–o803.
- Zhang, X.-L. & Li, Z.-X. (2005). *Acta Cryst. E* **61**, o266–o268.