organic papers

4-(3-Bromo-5-chloro-2-hydroxybenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Xin-Li Zhang* and Zong-Xiao Li

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

Correspondence e-mail: zhangxinli2006@tom.com

The title compound, $C_{18}H_{15}BrClN_3O_2$, was obtained by reacting 3-bromo-5-chlorosalicylic aldehyde and 4-aminoantipyrine. The structure displays a *trans* configuration about the imine C=N double bond. The N atom is also involved in an intramolecular O—H···N hydrogen bond, which stabilizes the configuration.

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.110
Data-to-parameter ratio = 13.5

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

Comment

Antipyrine (1-phenyl-2,3-dimethyl-5-pyrazolone, also called phenazone) is a pyrazolone-class analgesic agent in otic (relating to the ear) solutions in combination with other analgesics, such as benzocaine and phenylephrine. Antipyrine has been used as an antipyretic but has now been superseded, due to the possibility of a granulocytosis side effect. However, antipyrine itself is still available in some countries. Schiff base compounds have been of great interest for many years. These compounds play an important role in the development of coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures. As an extension of the work on the structural characterization of Schiff base compounds, the crystal structure of the title compound, (I), is reported.

(I)
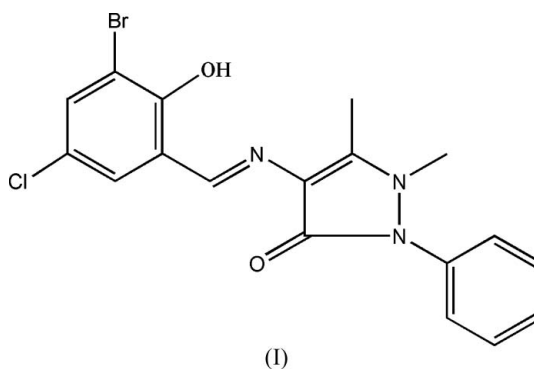
The molecular structure of (I) is illustrated in Fig. 1. All bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and are comparable with those observed in a similar antipyrine Schiff base (Liang *et al.*, 2002). The C7=N3 bond length of 1.288 (4) Å is as expected for a normal imine double bond. Because of conjugation through this double bond, the pyrazoline N1/N2/C8/C9/C10 ring and the C1–C6 benzene ring are approximately coplanar [dihedral angle 1.8 (2)°]. The dihedral angle between the pyrazoline ring and the C11–C16 phenyl ring is 36.8 (2)°. Atom O2 deviates from the pyrazoline mean plane by 0.123 (6) Å, whereas atoms C17 and C18 deviate from it on the opposite side by 0.737 (6) and 0.131 (7) Å, respectively.

In the crystal structure of (I), an intramolecular O—H···N hydrogen bond involving hydroxyl atom O1 and imine atom N3 (Table 1) stabilizes the *trans* configuration about the imine C═N bond.

Experimental

All reagents were of analytical grade from commercial sources and used without further purification. 3-Bromo-5-chlorosalicylaldehyde (0.1 mmol, 23.6 mg) and 4-aminoantipyrine (0.1 mmol, 20.3 mg) were dissolved in methanol (10 ml). The mixture was stirred for 30 min at 298 K to give a clear yellow solution. After allowing the resulting solution to evaporate slowly in air for 11 d, colourless block-shaped crystals formed at the bottom of the vessel.

Crystal data

$C_{18}H_{15}BrClN_3O_2$
$M_r = 420.69$
Monoclinic, $P2_1/n$
$a = 7.055$ (2) Å
$b = 8.058$ (3) Å
$c = 30.691$ (10) Å
$\beta = 91.716$ (4)°
$V = 1744.1$ (10) Å^3

$Z = 4$
$D_x = 1.602$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.53$ mm^{-1}
$T = 298$ (2) K
Plate, colourless
$0.50 \times 0.20 \times 0.05$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.683$, $T_{max} = 0.881$

6896 measured reflections
3070 independent reflections
2283 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.110$
$S = 1.02$
3070 reflections
228 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.3363P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N3	0.82	1.87	2.604 (4)	148
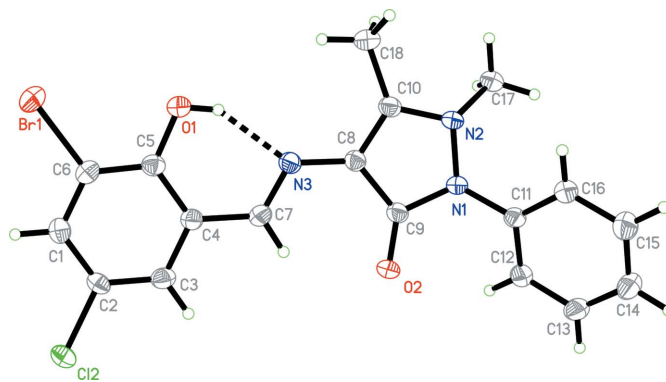
Figure 1
The molecular structure of compound (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radii. The dashed line represents a hydrogen bond.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with distances fixed at 0.93 (aromatic CH), 0.96 (methyl CH$_3$) and 0.82 Å (hydroxyl OH), and with $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic CH or $1.5U_{eq}(C,O)$ for other H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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, 2000); software used to prepare material for publication: *SHELXTL*.

References

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 (2000). *SMART* (Version 5.0) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
Liang, H., Yu, Q., Hu, R.-X., Zhou, Z.-Y. & Zhou, X.-G. (2002). *Transition Met. Chem.* **27**, 455–457.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.