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4-Amino-5-methyl-1,2,4-triazole-3-thione derivative of 4-[2-(2-hydroxyethyl)methylamino]benzaldehyde

Ji Wu,[a]* Yu-Peng Tian,[a] Fu-Xin Xie,[a] Suchada Chantrapromma,[b] Hoong-Kun Fun[c] and Ibrahim Abdul Razak[c]

[a]Department of Chemistry, Anhui University, Hefei 230039, People's Republic of China, [b]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: suchada@ratree.psu.ac.th

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.146
Data-to-parameter ratio = 13.0

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

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The title compound, 4-{4-[2-(2-hydroxyethyl)methylamino]-benzylideneamino}-5-methyl-1,2,4-triazole-3-thione, $C_{13}H_{17}N_5OS$, is present in a thioketone form and the triazole ring is almost planar. The asymmetric unit consists of two molecules which have different conformations. There is a weak C—H···S intramolecular interaction, a weak O—H···S intermolecular interaction and intermolecular N—H···O and O—H···N hydrogen bonds.

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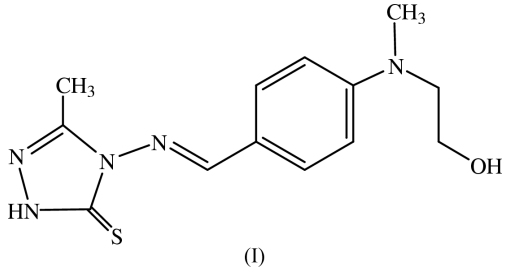
Comment

There has been continuous interest in the chemistry of Schiff base compounds because they possess antibacterial, anti-cancer and anti-inflammatory activities (Williams, 1972; Ren *et al.*, 1999). The sulfur-containing compounds are particularly effective (Dimmock *et al.*, 1997). Therefore, we synthesized a new sulfur-containing Schiff base compound, (I), that has a hydroxy group which helps to increase the solubility of the compound in water. The bioactivities of (I) are also being invesigated by our research group.



(I)

As shown in Fig. 1, there are two types of conformation of (I), molecules *A* and *B*. The dihedral angles of the mean planes of the triazole ring and the phenyl rings are 11.4 (2) and 69.3 (2)° for molecules *A* and *B*, respectively. The difference between these two molecules is further exhibited in that molecule A has a weak C—H···S intramolecular interaction, whereas molecule B has a weak O—H···S intermolecular interaction (Table 2).

The bond distances and angles in (I) are normal (Fun *et al.*, 1996; Liu *et al.*, 1999) and both molecules are in the thioketone form. There are also intermolecular N—H···O and O—H···N hydrogen bonds (Table 2).

Experimental

4-Amino-5-methyl-1,2,4-triazole-3-thione (0.05 mol in 20 ml ethanol), synthesized according to a reported method (Mohan, 1983), was added to a solution of 4-[(2-hydroxyethyl)methylamino]benzaldehyde (0.05 mol in 10 ml ethanol), also prepared according to the literature (Zhao *et al.*, 1995). Several drops of hydrochloric acid were

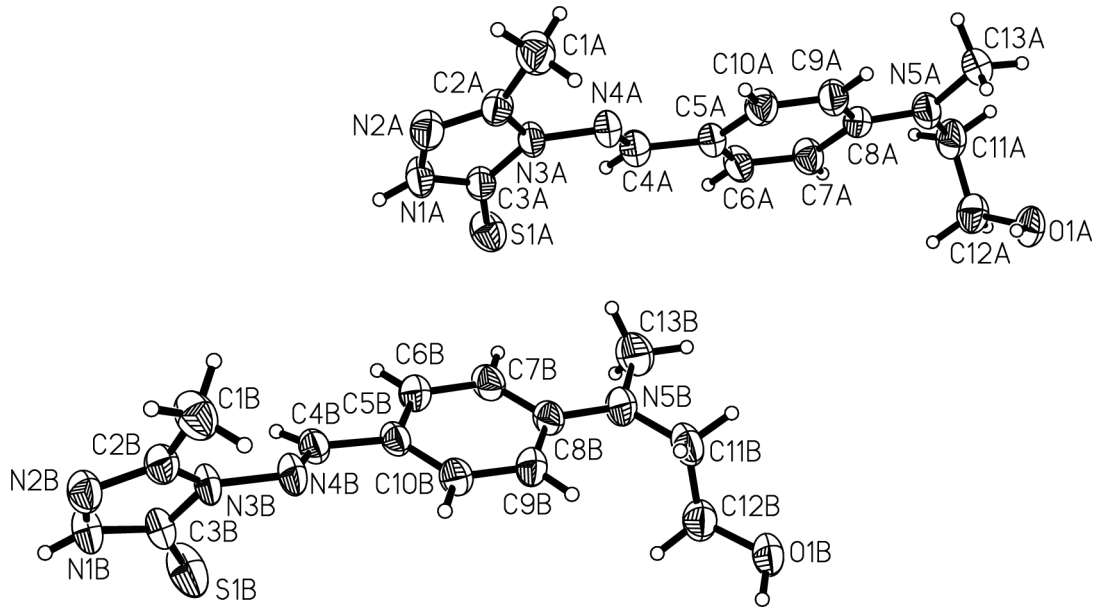


Figure 1
The structure of the title complex showing 50% probability displacement ellipsoids and the atom-numbering scheme.

added to the solution, which was then refluxed for 2 h. Yellow crystals of (I) were eventually obtained. The yield and the melting point of (I) are 70% and 448.6 K, respectively. The yellow crystals were recrystallized from ethanol by slow evaporation.

Crystal data

$C_{13}H_{17}N_5OS$	$Z = 4$
$M_r = 291.38$	$D_x = 1.352$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 10.7043$ (2) Å	Cell parameters from 4869 reflections
$b = 10.7230$ (2) Å	$\theta = 1.6$–$28.3°$
$c = 13.9487$ (3) Å	$\mu = 0.23$ mm^{-1}
$\alpha = 103.352$ (1)°	$T = 293$ (2) K
$\beta = 111.835$ (1)°	Plate, light yellow
$\gamma = 92.519$ (1)°	0.40 × 0.28 × 0.06 mm
$V = 1431.01$ (5) Å^3	

Data collection

Siemens SMART CCD area-detector diffractometer	4863 independent reflections
ω scans	3249 reflections with $I > 2\sigma(I)$
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.043$
$T_{min} = 0.914$, $T_{max} = 0.986$	$\theta_{max} = 25.0°$
7981 measured reflections	$h = -8 \rightarrow 12$
	$k = -12 \rightarrow 10$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.146$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.96$	$\Delta\rho_{max} = 0.30$ e Å^{-3}
4863 reflections	$\Delta\rho_{min} = -0.42$ e Å^{-3}
373 parameters	

Table 1
Selected bond lengths (Å).

S1*A*—C3*A*	1.663 (3)	S1*B*—C3*B*	1.666 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*AA*···O1*A*^{i}	0.86	2.00	2.845 (3)	167
N1*B*—H1*BA*···O1*B*^{i}	0.86	1.92	2.775 (3)	172
O1*A*—H1*AE*···N2*B*^{ii}	0.90 (5)	2.02 (4)	2.911 (3)	171 (4)
O1*B*—H1*BE*···S1*B*^{iii}	0.89 (7)	2.68 (7)	3.502 (2)	154 (6)
C4*A*—H4*AA*···S1*A*	0.93	2.53	3.255 (3)	135

Symmetry codes: (i) $1 + x, y, 1 + z$; (ii) $2 - x, 1 - y, 1 - z$; (iii) $2 - x, -y, 1 - z$.

After checking their presence in the difference maps, all H atoms, except the hydroxyl H atoms, were geometrically fixed and allowed to ride on their attached atoms with $U_{iso} = 1.5U_{eq}$ for methyl and $U_{iso} = 1.2U_{eq}$ for the other attached atoms. The methyl groups were refined as rigid rotors. The hydroxyl H atoms were located from the difference maps and refined isotropically.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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