

4-(*N*-Hydroxyethyl-*N*-methylamino)-benzaldehyde thiosemicarbazone

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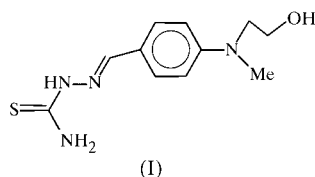
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In the crystal structure of the title compound, C₁₁H₁₆N₄OS, the phenyl ring and the thiosemicarbazone moiety from a dihedral angle of 7.7 (1)°. The crystal structure is governed by N—H···O and O—H···S hydrogen bonds leading to the formation of a two-dimensional network.

Comment

There is considerable interest in the chemistry of Schiff-base compounds containing N and S donors and their metal complexes. This is due to their non-linear optical properties (Tian *et al.*, 1996) and chelating ability with transition metal ions (Ali & Tarafdar, 1977; Ali & Bose, 1984; Davies *et al.*, 1990; Tian *et al.*, 1996). Thiosemicarbazones are derivatives of carbonyl compounds which exhibit a wide range of biological activities. The title compound, (I), is a Schiff-base derivative, more soluble in polar solvents, having potential second-order optical non-linearities. This work is a continuation of our studies on ligands with S and N as donor atoms and their metal complexes (Fun *et al.*, 1995, 1996; Tian, Wu *et al.*, 1999; Tian, Yu *et al.*, 1999).



The thiosemicarbazone moiety shows an *E* configuration about both the C2—N3 and C1—N2 bonds (Fig. 1) as found in dithiocarbazates (Fun *et al.*, 1995, 1996; Shanmuga Sundara Raj *et al.*, 2000) and thiosemicarbazones (Mathew & Palenik, 1971; Tian, Wu *et al.*, 1999; Tian, Yu *et al.*, 1999). The bond lengths, C1—S1 [1.680 (3) Å] and C1—N2 [1.337 (3) Å], respectively, confirm that the crystal contains the thione and thiol tautomers. The C=S distance, which lies intermediate between 1.82 Å for a C—S single bond and 1.56 Å for a C=S double bond (Sutton, 1965), agrees well with similar structures. The C—N distance is indicative of double-bond char-

acter. The phenyl and the thiosemicarbazone moiety makes a dihedral angle of 7.7 (1)° indicating the slight deviation from planarity of the molecule.

In the crystal lattice, the molecules of the title compound are arranged in two individual parallel layers connected through N—H···O (N1···O1) and O—H···S hydrogen bonds; these two layers are aligned nearly perpendicular to each other. It is extended to form a two-dimensional network through another N—H···O hydrogen bond (N2···O1) (Table 2). The molecular chain runs along the *c* axis. The molecules are stacked with a repeating distance of 4.400 (1) Å between the centroids of the phenyl ring. The packing of the molecules is also stabilized by the N—H···π interaction [N1···Cg(−*x*, ½ + *y*, ½ − *z*) = 3.225 (2); H1C···Cg = 2.698 (1) Å; N1—H1C···Cg = 120.9; Cg is the centroid of the phenyl ring].

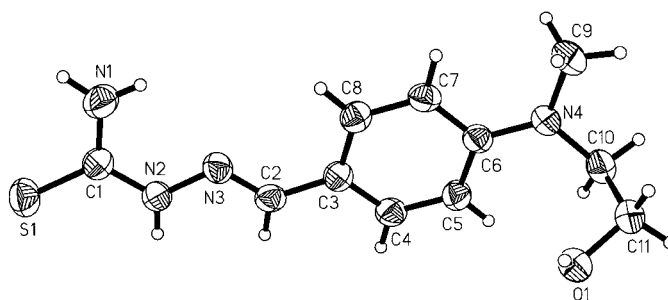


Figure 1
The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title compound was prepared by the reaction of 4-[(2-hydroxyethyl)methylamino]benzaldehyde and thiosemicarbazone under reflux in ethanol solution for 3 h. Single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of an acetone solution at room temperature.

Crystal data

C₁₁H₁₆N₄OS
M_r = 252.34
Monoclinic, *P*2₁/*c*
a = 14.7856 (3) Å
b = 7.7565 (1) Å
c = 11.9571 (2) Å
β = 112.517 (1)°
V = 1266.75 (4) Å³
Z = 4

D_x = 1.323 Mg m^{−3}
Mo *K*α radiation
Cell parameters from 4020 reflections
θ = 1.49–28.33°
μ = 0.246 mm^{−1}
T = 293 (2) K
Slab, yellow
0.44 × 0.32 × 0.14 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
T_{min} = 0.90, *T_{max}* = 0.97
8696 measured reflections

3071 independent reflections
1860 reflections with *I* > 2σ(*I*)
R_{int} = 0.065
θ_{max} = 28.23°
h = −19 → 19
k = −9 → 10
l = −15 → 14

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.061
wR (*F*²) = 0.117
S = 0.932
3071 reflections
154 parameters

H-atom parameters constrained
w = 1/[σ²(*F_o*²) + (0.1109*P*)²]
where *P* = (*F_o*² + 2*F_c*²)/3
(Δ/σ)_{max} < 0.001
Δρ_{max} = 0.69 e Å^{−3}
Δρ_{min} = −0.53 e Å^{−3}

Table 1Selected geometric parameters (\AA , $^\circ$).

O1—C11	1.428 (3)	N3—C2	1.275 (3)
N1—C1	1.342 (3)	N4—C6	1.369 (3)
N2—C1	1.337 (3)	N4—C9	1.452 (3)
N2—N3	1.391 (3)	N4—C10	1.457 (3)
C1—N2—N3	119.9 (2)	C6—N4—C10	122.7 (2)
C2—N3—N2	115.3 (2)	C9—N4—C10	115.3 (2)
C6—N4—C9	120.7 (2)		

Table 2Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1B \cdots N3	0.86	2.26	2.621 (3)	105
O1—H1A \cdots S1 ⁱ	0.82	2.39	3.187 (2)	163
N1—H1C \cdots O1 ⁱⁱ	0.86	2.60	3.244 (3)	133
N2—H2A \cdots O1 ⁱⁱⁱ	0.86	2.06	2.923 (3)	177

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

All H atoms were located from a difference Fourier map, fixed at calculated distances (C—H range 0.93–0.97 \AA) and allowed to ride on the parent atoms to which they were attached during refinement. The highest peak and deepest hole are located around the S atom.

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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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1408). Services for accessing these data are described at the back of the journal.

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