

2-[5-(Cyclohexylamino)-1,3,4-thiadiazol-2-yl]phenol

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Key indicators

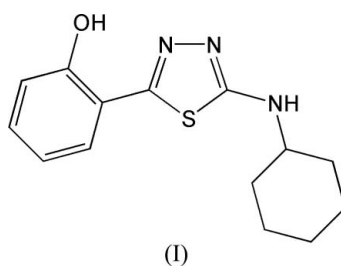
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.046
 wR factor = 0.112
Data-to-parameter ratio = 17.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{14}\text{H}_{17}\text{N}_3\text{OS}$, exists in the *E,E* configuration with respect to the $\text{C}=\text{N}$ bonds of the thiadiazole ring. In the crystal packing, molecules are linked into centrosymmetric dimers through $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds.

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Comment

Thiadiazoles and their derivatives represent a group of compounds possessing a wide spectrum of biological activities, such as hypoglycemic, antitubercular, antifungal and antibacterial properties (Bhat *et al.*, 1967). One route to obtain thiadiazolines is the heterocyclization of thiosemicarbazones (Somogyi, 1991; Kubota *et al.*, 1980). Thiosemicarbazones were subjected to ring closure by means of acetylating agents, to obtain the corresponding 1,3,4-thiadiazolines (Tarbell & Price, 1957). Some thiosemicarbazones and thiadiazolines showed interesting biological activity against *Bacillus subtilis*, *Candida albicans*, *Micrococcus luteus*, *Trichophyton mentagrophites* and *Aspergillus niger* (Turk *et al.*, 1986).



The molecular structure of (I) is shown in Fig. 1, and selected geometric parameters are listed in Table 1. The compound exists in an *E,E* configuration with respect to the double bonds $\text{C1}=\text{N2}$ and $\text{C2}=\text{N3}$. This is confirmed by the torsion angles $\text{C3}-\text{C2}=\text{N3}-\text{N2} = 179.73$ (16) $^\circ$ and $\text{N1}-\text{C1}=\text{N2}-\text{N3} = 178.19$ (18) $^\circ$ (Nagao *et al.*, 1998). Bond lengths for $\text{C}-\text{S}$, $\text{C}=\text{N}$ and $\text{N}-\text{N}$ functionalities are similar to those found in other thiadiazoline derivatives (*e.g.* Hu & Ying, 2005). As an example, the $\text{C}-\text{S}$ bond lengths are consistent with $\text{C}-\text{S}$ single bonds rather than with $\text{C}=\text{S}$ bonds (Suni *et al.*, 2006). Ring puckering analysis (Cremer & Pople, 1975) and least-squares calculations show that the cyclohexyl substituent has a chair conformation ($Q_T = 0.562$ Å) with equatorial substitution at C9 for N1.

When viewed along [100], the molecules form a herringbone arrangement (Fig. 2), similar to that reported in the structural arrangement of *trans*-[*N,N'*-bis(salicylidene)cyclohexane-1,2-diaminato]nickel(II) (de Castro *et al.*, 2001). The crystal packing involves one intra- and one intermolecular hydrogen-bonding interactions (Table 2). Intramolecular

hydrogen bonding leads to the formation of an $S(6)$ ring, while intermolecular $N-H \cdots N$ hydrogen bonds form centrosymmetric dimers in the crystal structure.

Experimental

Salicylaldehyde-*N*-cyclohexylthiosemicarbazone (1 mmol, 0.277 g) was refluxed with $Mn(OAc)_2 \cdot 4H_2O$ (1 mmol, 0.291 g) in methanol (25 ml) for 3 h, followed by evaporation to dryness under reduced pressure, resulting in the unexpected formation of (I). Intense gold block crystals suitable for single-crystal X-ray diffraction studies were obtained from a solution of (I) in a mixture of CH_3CN and DMF (1:1).

Crystal data

$C_{14}H_{17}N_3OS$	$Z = 4$
$M_r = 275.37$	$D_x = 1.324 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 7.9402(16) \text{ \AA}$	$\mu = 0.23 \text{ mm}^{-1}$
$b = 10.5702(10) \text{ \AA}$	$T = 293(2) \text{ K}$
$c = 16.545(6) \text{ \AA}$	Block, intense gold
$\beta = 95.65(2)^\circ$	$0.33 \times 0.26 \times 0.21 \text{ mm}$
$V = 1381.9(6) \text{ \AA}^3$	

Data collection

Oxford Diffraction Xcalibur-S diffractometer	16447 measured reflections
ω scans	3173 independent reflections
Absorption correction: multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2006)	2483 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.928$, $T_{\max} = 0.962$	$R_{\text{int}} = 0.029$
	$\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 0.2525P]$
$R[F^2 > 2\sigma(F^2)] = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.112$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
3173 reflections	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
180 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

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

C1–N2	1.313 (2)	C4–O1	1.354 (2)
C1–N1	1.334 (2)	N1–H1N	0.85 (2)
C1–S1	1.7432 (18)	N2–N3	1.376 (2)
C2–N3	1.297 (2)	O1–H101	0.86 (3)
N2–C1–N1	122.95 (16)	C3–C2–S1	123.33 (13)
N2–C1–S1	113.91 (13)	N1–C9–C14	108.84 (16)
N1–C1–S1	123.13 (14)	N1–C9–C10	112.56 (16)
N3–C2–C3	123.56 (16)	C4–O1–H101	107 (2)
N3–C2–S1	113.12 (13)	C2–S1–C1	86.98 (9)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1–H101 \cdots N3	0.86 (3)	1.84 (3)	2.621 (2)	151 (3)
N1–H1N \cdots N2 ⁱ	0.85 (2)	2.13 (2)	2.968 (2)	175 (2)

Symmetry code: (i) $-x + 1, -y + 1, -z + 2$.

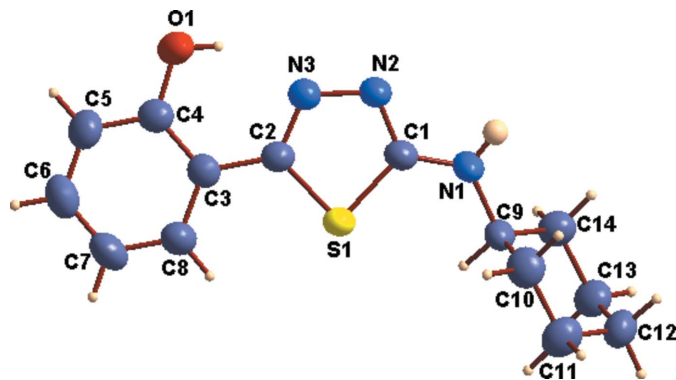


Figure 1

The molecular structure and labelling scheme for (I), with displacement ellipsoids drawn at the 50% probability level.

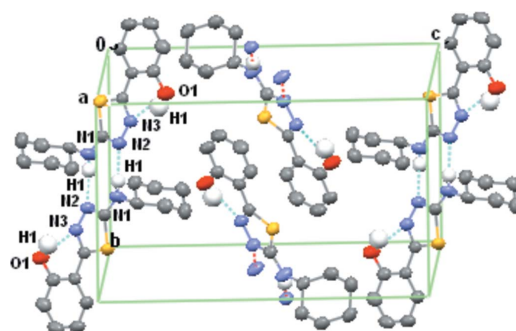


Figure 2

A packing diagram for (I), viewed approximately along the [100] axis. Dashed lines indicate intra- and intermolecular hydrogen-bonding interactions.

C-bound H atoms were placed in calculated positions and refined with C–H bond lengths constrained to 0.93 (aromatic CH), 0.97 (methylene CH_2) or 0.98 \AA (methine CH), and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(\text{carrier C atom})$. Heteroatom-bonded H atoms H1N and H101 were found in a difference map and refined with free coordinates and isotropic displacement parameters.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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