

4,4,5,5-Tetramethyl-2-(thiazol-2-yl)imidazolidine-1,3-diol

Kai Jiang,^{a*} Jiu-Li Chang,^a Li-Ya Wang,^b Lu-Fang Ma^b and Yu-Fang Wang^b^aCollege of Chemistry and Environmental Science, Henan Normal University, Xixiang 453002, People's Republic of China, and^bDepartment of Chemistry, Luoyang Normal University, Luoyang 471022, People's Republic of China

Correspondence e-mail: wlyalynu.edu.cn

Key indicators

Single-crystal X-ray study

T = 273 K

Mean $\sigma(C-C)$ = 0.004 Å

R factor = 0.052

wR factor = 0.148

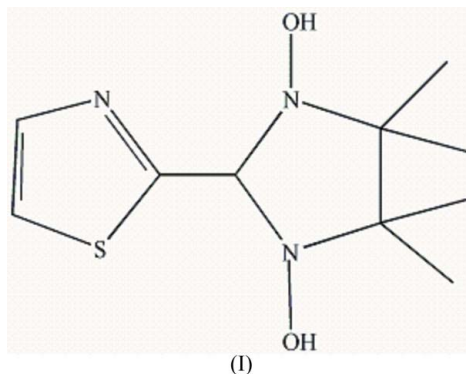
Data-to-parameter ratio = 19.1

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

In the crystal structure of the title compound, $C_{10}H_{17}N_3O_2S$, the packing of the molecules is mainly a result of two types of $O-H \cdots N$ hydrogen-bonding interactions, forming a two-dimensional network.

Comment

The design and synthesis of multidimensional molecule-based magnetic materials is one of the major challenges in molecular materials research (Kahn 1993; Laget *et al.*, 1998; Vaz *et al.*, 1999; Yamamoto *et al.*, 2001). Nitronyl nitroxide radicals, due to their intriguing structural diversity and potential applications, have played a prominent role in the design and construction of molecular magnetic materials (Vasilevsky *et al.*, 1998; Marvilliers *et al.*, 1999; Hirel *et al.*, 2001; Li *et al.*, 2003). However, problems associated with the preparation of key intermediates of nitronyl nitroxides are probably the reason for the limited number of developments reported so far on structural variations in nitronyl nitroxides (Lin *et al.*, 2003; Wang *et al.*, 2004). During the course of synthesizing a novel thiazolyl-substituted nitronyl nitroxide radical, we also obtained the title compound, (I), as an intermediate, and we now report its synthesis and crystal structure.



The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1.

There are two types of intermolecular $O-H \cdots N$ hydrogen-bonding interactions in the crystal structure of (I). One type of hydrogen bond occurs between the N atom of the thiazole ring and one O atom of a symmetry-related imidazolidine ring (Table 2). The other type of hydrogen bond exists between two adjacent imidazolidine rings: one O atom of one imidazolidine ring and one N atom of a neighbouring molecule form double hydrogen bonds (Table 2). In the crystal structure, single and double hydrogen bonds are alternately arranged in such a way that six adjacent molecules form a ring. Thus, the

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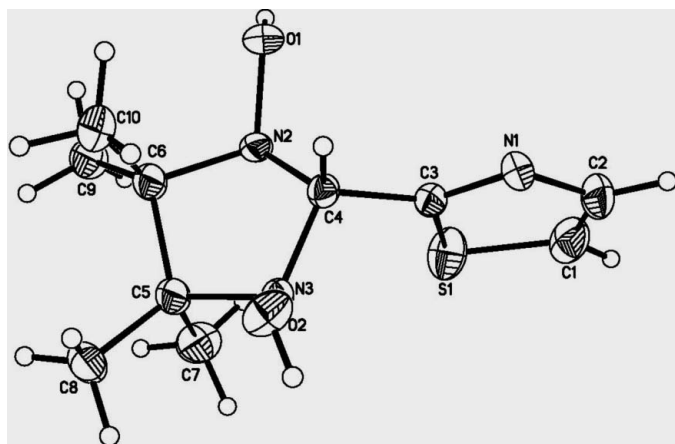


Figure 1
A drawing of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

molecules of (I) are linked *via* intermolecular O—H···N hydrogen bonds, resulting in a two-dimensional network arrangement (Fig. 2).

Experimental

The title compound was synthesized by dissolving 2,3-dimethyl-2,3-bis(hydroxylamino)butane (1.48 g, 10 mmol) in an ethanol solution (20 ml) of 2-thiazolecarboxaldehyde (1.07 g, 10 mmol), following the method of Ullman *et al.* (1970). The resulting solution was refluxed for 5 h and then stirred for 24 h at 298 K. The mixture was then filtered to remove a white precipitate and the clear filtrate was allowed to stand at 298 K in the dark for three weeks. Yellow crystals of (I) suitable for X-ray diffraction were obtained. Analysis, found: C 49.43, H 7.12, N 17.18%; calculated for C₁₀H₁₇N₃O₂S: C 49.36, H 7.04, N 17.27%.

Crystal data

C ₁₀ H ₁₇ N ₃ O ₂ S	$D_x = 1.281 \text{ Mg m}^{-3}$
$M_r = 243.33$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 4327 reflections
$a = 6.9785 (5) \text{ \AA}$	$\theta = 3.0\text{--}27.6^\circ$
$b = 18.7982 (13) \text{ \AA}$	$\theta_{\text{max}} = 27.5^\circ$
$c = 10.0764 (7) \text{ \AA}$	$\mu = 0.25 \text{ mm}^{-1}$
$\beta = 107.367 (1)^\circ$	$T = 273 (2) \text{ K}$
$V = 1261.59 (15) \text{ \AA}^3$	Prism, yellow
$Z = 4$	$0.39 \times 0.29 \times 0.28 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	2886 independent reflections
φ and ω scans	2326 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$R_{\text{int}} = 0.015$
$T_{\text{min}} = 0.909$, $T_{\text{max}} = 0.934$	$\theta_{\text{max}} = 27.5^\circ$
10799 measured reflections	$h = -8 \rightarrow 9$
	$k = -24 \rightarrow 24$
	$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 0.6505P]$
$R[F^2 > 2\sigma(F^2)] = 0.053$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.148$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.49 \text{ e \AA}^{-3}$
2886 reflections	$\Delta\rho_{\text{min}} = -0.33 \text{ e \AA}^{-3}$
151 parameters	
H-atom parameters constrained	

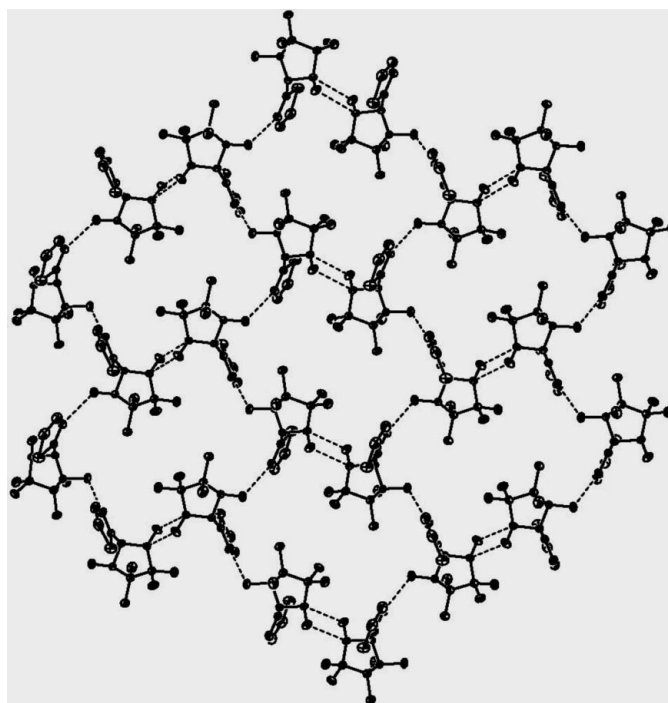


Figure 2
The two-dimensional network arrangement of the crystal structure of (I), viewed along the [100] axis. Dashed lines indicate hydrogen bonds.

Table 1

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

S1—C1	1.696 (3)	N2—C4	1.460 (2)
S1—C3	1.718 (2)	N2—C6	1.500 (3)
O1—N2	1.4330 (19)	N3—C4	1.484 (2)
O2—N3	1.4415 (19)	N3—C5	1.526 (2)
N1—C3	1.298 (3)	C1—C2	1.326 (4)
N1—C2	1.387 (3)	C5—C6	1.536 (3)
C1—S1—C3	89.95 (13)	C4—N2—C6	105.36 (15)
C3—N1—C2	109.7 (2)	O2—N3—C4	106.26 (14)
O1—N2—C4	106.35 (13)	O2—N3—C5	112.83 (15)
O1—N2—C6	110.21 (15)	C4—N3—C5	106.57 (13)

Table 2

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

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1—H1···N1 ⁱ	0.82	2.00	2.821 (3)	174
O2—H2···N3 ⁱⁱ	0.82	2.10	2.848 (2)	151

Symmetry codes: (i) $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (ii) $-x + 1, -y + 1, -z + 1$.

H atoms were placed in calculated positions and refined using a riding-model approximation, with bond lengths and $U_{\text{iso}}(\text{H})$ values as follows: 0.93 \AA and $1.2U_{\text{eq}}(\text{C})$ for Csp^2H ; 0.96 \AA and $1.5U_{\text{eq}}(\text{C})$ for methyl CH_3 ; 0.98 \AA and $1.2U_{\text{eq}}(\text{C})$ for methine CH ; 0.82 \AA and $1.5U_{\text{eq}}(\text{O})$ for hydroxyl OH .

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); 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, 2002); software used to prepare material for publication: *SHELXL97*.

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References

- Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
- Hirel, C., Vostrikova, K. E., Pécaut, J., Ovcharenko, V. I. & Rey, P. (2001). *Chem. Eur. J.* **7**, 2007–2014.
- Kahn, O. (1993). *Molecular Magnetism*, pp. 251–332. New York: VCH.
- Laget, V., Hornick, C., Rabu, P., Drillon, M. & Ziessel, R. (1998). *Coord. Chem. Rev.* **178–180**, 1533–1553.
- Li, L. C., Liu, Z. L., Turner, S. T., Liao, D. Z., Jiang, Z. H. & Yan, S. P. (2003). *Eur. J. Inorg. Chem.* pp. 62–65.
- Lin, H.-H., Mohanta, S., Lee, C.-J. & Wei, H.-H. (2003). *Inorg. Chem.* **42**, 1584–1589.
- Marvilliers, A., Pei, Y., Cano Boquera, J., Rivière, E., Audière, J. P. & Mallah, T. (1999). *Chem. Commun.* pp. 1951–1952.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Ullman, E. F., Call, L. & Osiecki, J. H. (1970). *J. Org. Chem.* **35**, 3623–3631.
- Vasilevsky, S. F., Tretyakov, E. V., Usov, O. M., Molin, Y. N., Fokin, S. V., Shwedenkov, Y. G., Ikorskii, V. N., Romanenko, G. V., Sagdeev, R. Z. & Ovcharenko, V. I. (1998). *Mendeleev Commun.* pp. 216–218.
- Vaz, M. G. F., Pinheiro, L. M. M., Stumpf, H. O., Alcântara, A. F. C., Golhen, S., Ouahab, L., Cador, O., Mathonière, C. & Kahn, O. (1999). *Chem. Eur. J.* **5**, 1486–1495.
- Wang, L.-Y., Li, L.-C., Liao, D.-Z., Jiang, Z.-H. & Yan, S.-P. (2004). *Eur. J. Inorg. Chem.* pp. 2266–2271.
- Yamamoto, Y., Suzuki, T. & Kaizaki, S. (2001). *J. Chem. Soc. Dalton Trans.* pp. 1566–1572.