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trans-4,5-Dihydroxyimidazolidine-2-thione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.028
wR factor = 0.074
Data-to-parameter ratio = 12.8

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

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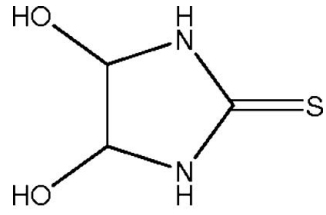
In the title compound, $C_3H_6N_2O_2S$, the five-membered ring has an envelope conformation and the two hydroxyl groups lie on opposite sides of the ring. The crystal structure is stablized by intermolecular O—H···S and N—-H···O hydrogen bonds, and also a close O···O intermolecular approach, to form a two-dimensional network parallel to *bc*.

Comment

It is known that both imidazolidine-2-thione and imidazolidin-2-one derivatives exhibit powerful bioactivities. For example, *N*,*N*′-disubstituted 4,5-dihydroxyimidazolidine-2-thiones have a powerful ectoparasiticidal action (Enders *et al.*, 1979) and *N*,*N*′-disubstituted 4,5-dihydroxyimidazolidin-2-ones possess good herbicidal activity (Perronnet & Teche, 1973). A number of molecules having this heterocyclic ring exhibit potent antidiabetic properties (Lenzen & Ahmad, 2001) and anti-HIV activity (Lam *et al.*, 1994). *N*,*N*′-disubstituted 4,5-dihydroxyimidazolidine-2-thiones (ones) can be readily prepared by condensation of disubstituted thioureas with glyoxal and they exhibit great stability (Enders *et al.*, 1979; Perronnet & Teche, 1973). In the course of our research on imidazolidinones, we recently needed to prepare *N*,*N*′-unsubstituted 4,5-dihydroxyimidazolidine-2-thione as a synthetic intermediate. However, initial synthetic efforts in our laboratory indicated that the title *N*,*N*′-unsubstituted 4,5-dihydroxyimidazolidine-2-thione, (I), is not easily accessible under the reported conditions, because of its instability. Thus, it seemed desirable to try to understand what structural factors affect its stability. Therefore, an X-ray crystallographic analysis of the compound has been completed and the results are presented here.



(I)

The molecular structure of (I) is shown in Fig. 1. The five-membered ring (N1/N2/C1–C3) has a flattened envelope conformation; C3 is the flap atom, displaced by 0.311 (2) Å from the plane of the other four atoms. The conformation of the five-membered ring and coplanarity of the S1/C1/N1/N2/C2 fragment are similar to the situation found in a complex of 1,3-imidazole-2-thione with mercury(II) chloride (Pavlović *et*

al., 2000). The two hydroxyl groups lie on opposite sides of the ring. The C1—N1 and C1—N2 bonds [1.336 (2) and 1.335 (2) Å, respectively] are shorter than the corresponding bonds in 1,3-dibenzoylimidazolidine-2-thione [1.377 (1) Å; Kazak *et al.*, 2005]. Conversely, the C1═S1 bond is longer [1.684 (2) *versus* 1.650 (1) Å].

The crystal structure is held together by a combination of two intermolecular N—H···O and two O—H···S hydrogen bonds. There is also a close O···O intermolecular approach [O2···O2^{v} = 2.895 (2) Å; symmetry code: (v) $1-x, 2-y, 1-z$]. These interactions result in the assembly of the molecules into layers parallel to *bc* (Fig. 2 and Table 1).

Experimental

Into a three-necked round-bottomed flask equipped with a mechanical stirrer were introduced thiourea (38 g, 0.5 mol) and water (50 ml). Glyoxal (75 g, 40%) was added in one portion and the mixure was then heated at about 343 K with stirring for 30 min under an inert atmosphere. Natural cooling of the reaction mixture overnight gave single crystals of (I) (yield 54 g, 80%).

Crystal data

$C_3H_6N_2O_2S$
M_r = 134.16
Triclinic, $P\overline{1}$
a = 5.8092 (13) Å
b = 6.5152 (13) Å
c = 7.9189 (17) Å
α = 98.595 (3)°
β = 106.740 (3)°
γ = 102.494 (3)°
V = 272.93 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 293 (2) K
0.50 × 0.40 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.644, T_{max} = 0.929
1137 measured reflections
938 independent reflections
913 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.074
S = 1.10
938 reflections
73 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···S1^{i}	0.82	2.44	3.245 (3)	167
O2—H2···S1^{ii}	0.82	2.42	3.238 (3)	175
N1—H3···O2^{iii}	0.86	2.35	3.004 (2)	133
N2—H4···O1^{iv}	0.86	2.00	2.826 (2)	162

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

H atoms were placed in idealized positions and allowed to ride on their respective parent atoms, with C—H = 0.98 Å, O—H = 0.82 Å and N—H = 0.86 Å, and with U_{iso}(H) = xU_{eq}(carrier atom), where x = 1.2 for C—H and N—H or 1.5 for O—H.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

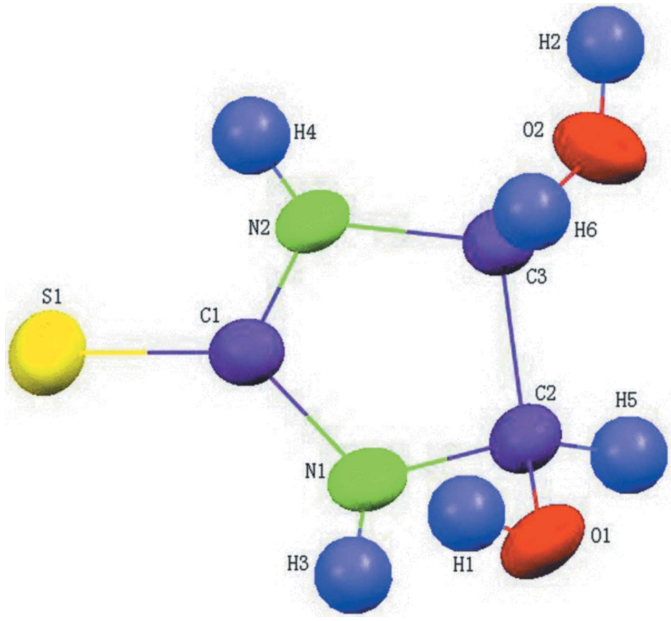


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

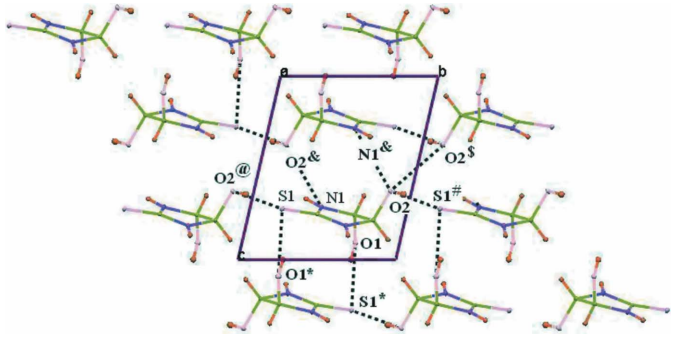


Figure 2
A packing diagram for (I), with intermolecular interactions represented by dashed lines. Non-H atoms are shown as 10% probability displacement ellipsoids and H atoms are drawn as small spheres of arbitrary radius. Atoms marked with an asterisk (*), hash (#), dollar sign ($), ampersand (&) or 'at' symbol (@) are at the symmetry positions $(1-x, 1-y, 2-z)$, $(x, 1+y, z)$, $(1-x, 2-y, 1-z)$, $(1-x, 1-y, 1-z)$ and $(x, -1+y, z)$, respectively.

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