Crystal Structure of 4-(2-Aminothiazol-4-yl)-4-(hydroxyimino)acetic ester

Qian MIAO,* Zhi-Min JIN,** Jing XIONG,* and Mao-Lin HU*†

*Department of Chemistry and Material Science, Wenzhou Normal College,
Wenzhou, Zhejiang 325027, P. R. China

**College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou 310014, P. R. China

4-(2-Aminothiazol-4-yl)-4-(hydroxyimino)acetic ester crystallizes in monoclinic form, space group $P2_1/c$ with $a = 10.2822(17)$, $b = 10.9006(18)$, $c = 8.8072(15)$ Å, and $Z = 4$. The structure was solved by direct methods (SHELXS97) and refined by full-matrix least squares to a final value of $R1 = 0.0501$ for 2207 reflections [$I > 2\sigma(I)$]. The C5 atom and O1 atom are in *cis* conformation with regard to the C4–N3 double bond. There is an intra molecular hydrogen bond between O1–H1A...O2, and different molecules are associated to each other to form the whole by three kinds of intermolecular hydrogen bonds and van der Waals force.

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Hydroxyimino-carboxylic acid and their derivatives have been found important applications in medical and biological fields.¹⁻⁴ 4-(2-Aminothiazol-4-yl)-4-(hydroxyimino)acetic ester (Fig. 1) has a structure characterized by thiazolyl, hydroxyimino and amino groups which can coordinate to metal ions, so the compound acts as an efficient chelating agent for a variety of metal ions. In this work, an X-ray structural analysis of the title compound was carried out in order to understand the stereochemistry of hydroxyimino-carboxylic acid derivatives better.

Colorless plate-like crystals were obtained from a tetrahydrofuran solution at room temperature. One crystal with a size of $0.61 \times 0.47 \times 0.35$ mm was sealed in a glass tube and used for diffraction experiments. The crystal and experimental data are given in Table 1. The structure was solved by direct methods using SHELXS97⁵ and refined with anisotropic temperature factors for non-hydrogen atoms. All hydrogen atoms were located from difference Fourier maps and are not refined. The atomic coordinates for non-hydrogen atoms are listed in Table 2. An ORTEP-3⁶ drawing of the title compound together with the atomic numbering is shown in Fig. 2.

The bond lengths and angles are similar to those of other thiazolyloxime compounds.^{1,2} The O–N, S–C, N=C and N–C bond lengths [O(1)–N(3) 1.396(2) Å, S(1)–C(1) 1.741(2) Å, S(1)–C(2) 1.717(2) Å, N(2)–C(1) 1.307(3) Å, N(3)–C(4) 1.278(2) Å, N(2)–C(3) 1.386(2) Å, N(1)–C(1) 1.337(3) Å] are in the normal range for thiazolyloxime compounds. The C1, C2,

C3, N2 and S1 atoms form a plane with mean deviation 0.0045 Å, and S1 deviates by 0.0053 Å from this plane. The thiazolyloxime has a fully stretched skeleton with torsion angles of N3–C4–C5–O2, C3–C4–C5–O2, C3–C4–C5–O3, O1–N3–C4–C3, C6–O3–C5–C4, O1–N3–C4–C5, and C5–O3–C6–C7 being $-82.3(3)$, $97.7(2)$, $-82.0(2)$, $+178.65(17)$, $-174.7(2)$, $-1.4(3)$, and $79.2(3)^\circ$, respectively. The carbon C5 atom and oxygen O1 atom are in *cis* conformation with regard to the C4–N3 double bond, which can be corroborated by the O(1)–H(1A)···O(2) intra molecular hydrogen bond with the donor-acceptor distance 3.039(2) Å and the O(1)–H(1A)···O(2) angle 142.4° . The corresponding *trans* conformation would lead to a considerable repulsion between the hydroxyl group and the thiazolyl. Moreover, different molecules are packed close to each other by three kinds of intermolecular hydrogen bonds $[O(1)–H(1A)···N(1)(x,y,Z + 1) = 2.933(2)$ Å,

Table 1 Crystal and experimental data

Formula:	C ₇ H ₉ N ₃ O ₃ S
Formula weight:	215.23
Crystal system:	monoclinic
Space group:	$P2_1/c$ $Z = 4$
a :	10.2822(17) Å
b :	10.9006(18) Å
c :	8.8072(15) Å
V :	971.7(3) Å ³
D_{calc} :	1.471 g/cm ³
No. of reflections used:	=2207 ($I > 2\sigma(I)$)
$2\theta_{\text{max}}$:	56.46°
$R1$:	0.0501
$wR2$:	0.1357
Data/parameters:	2207/131
Goodness-of-fit:	0.974
$(\Delta/\sigma)_{\text{max}}$:	0.000
$(\Delta\rho)_{\text{max}}$:	0.564 e Å ⁻³
$(\Delta\rho)_{\text{min}}$:	-0.407 e Å ⁻³
Measurement:	Bruker SMART CCD system
Program system:	SHELXS97
Structure determination:	SHELXS97
Refinement:	full matrix least-squares SHELXL97

Fig. 1 Chemical structure.

† To whom correspondence should be addressed.
E-mail: maolin_hu@yahoo.com

Table 2 Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

Atom	x	y	z	U_{eq}
S(1)	3792(1)	8707(1)	1891(1)	51(1)
O(1)	2527(2)	11794(1)	7251(2)	52(1)
O(2)	3107(2)	9065(1)	7604(2)	49(1)
O(3)	1146(2)	9264(2)	6067(2)	64(1)
N(1)	3776(2)	10834(2)	350(2)	53(1)
N(2)	3315(2)	10863(2)	2858(2)	39(1)
N(3)	2933(2)	11589(2)	5842(2)	45(1)
C(1)	3613(2)	10288(2)	1664(2)	39(1)
C(2)	3414(3)	8851(2)	3705(2)	47(1)
C(3)	3206(2)	10036(2)	4024(2)	38(1)
C(4)	2856(2)	10453(2)	5475(2)	39(1)
C(5)	2403(2)	9512(2)	6524(2)	40(1)
C(6)	508(3)	8447(3)	7031(4)	78(1)
C(7)	179(4)	9110(4)	8342(5)	110(1)

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* (a_i \cdot a_j).$$

$\angle \text{O(1)-H(1A)} \cdots \text{N(1)} = 125.4^\circ$; $\text{N(1)-H(1B)} \cdots \text{N(3)}(x, -y + 5/2, z - 1/2) = 2.933(3) \text{\AA}$, $\angle \text{N(1)-H(1B)} \cdots \text{N(3)} = 153.5^\circ$; $\text{N(1)-H(1C)} \cdots \text{O(2)}(x, y, z - 1) = 3.076(2) \text{\AA}$, $\angle \text{N(1)-H(1C)} \cdots \text{O(2)} = 143.7^\circ$ and van der Waals force.

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Fig. 2 Molecular structure of ORTEP-3 with the atom numbering. Thermal ellipsoids of the non-hydrogen atoms are scaled to enclose 50% probability. The spheres of the hydrogen atoms are drawn in an arbitrary scale.

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